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(1*E*,2*E*)-1,2-Bis[(1-benzyloxymethyl-1*H*-indol-3-yl)methylidene]hydrazine

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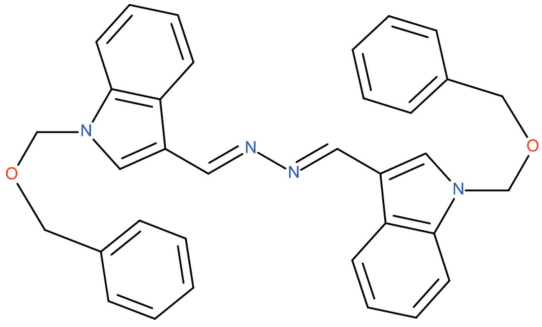
Key indicators: single-crystal X-ray study; T = 90 K, P = 0.0 kPa; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.126; data-to-parameter ratio = 14.7.

The title compound, $C_{34}H_{30}N_4O_2$, lies on an inversion center and consists of two 3-substituted-1*H*-indole units linked by a 1,2-dimethylenehydrazine bridge. It is one of numerous examples in which two aromatic ring systems are joined by this 4-atom bridge. The geometry of the centrosymmetric bridge is: C(arom)—C = 1.444 (3), C=N = 1.284 (3), N—N = 1.414 (4) Å, C(arom)—C=N = 122.6 (2) and C=N—N = 111.9 (2)°. The nine non-H atoms of the indole unit lie in a plane ($\delta_{r.m.s.}$ = 0.0089 Å) which is twisted 6.0 (2)° with respect to the hydrazine bridge plane. The benzyloxymethyl substituents do not lie in the plane of the rest of the molecule and are in a folded rather than an extended conformation. This is described by the three torsion angles in the middle of the C=N—C—O—C_{Bz} group, *viz.* 98.5 (3), −62.1 (3), and −66.3 (2)°.

Related literature

For the synthesis, see: Shui (1994). For related structures, see: Burke-Laing & Laing (1976); Mom & de With (1978); Biswas *et al.* (1999); Rizal *et al.* (2008). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$C_{34}H_{30}N_4O_2$
M_r = 526.62
Monoclinic, $P2_1/n$
a = 12.8518 (10) Å
b = 7.9663 (9) Å
c = 13.6810 (12) Å
β = 103.672 (5)°
V = 1361.0 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 90 K
0.18 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor 1997)
T_{min} = 0.986, T_{max} = 0.996
4340 measured reflections
2678 independent reflections
1481 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.126
S = 0.99
2678 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor 1997); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The purchase of the diffractometer was made possible by grant No. LEQSF(1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents. We thank Dr Lee Shui for kindly providing the sample.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2449).

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(1*E*,2*E*)-1,2-Bis[(1-benzyloxymethyl-1*H*-indol-3-yl)methylidene]hydrazine

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S1. Comment

Title compound **I** was synthesized as part of a project targeting the synthesis of tryptophan and other tyrosine derivatives (Shui, 1994). The molecule occupies a crystallographic inversion center and consists of two 3-substituted 1*H*-indole moieties linked by a (1E,2E)-1,2-dimethylenehydrazine bridge, one of numerous examples in which two aromatic ring systems are joined by this 4-atom bridge. The bridge geometry is consistent with that found, for example, in Ph—CH═N—N═CH—Ph [CAS:1048662–10-7, Mom & de With (1978); Burke-Laing & Laing (1976); CSD:BZAZIN02, BZAZIN11, respectively, Allen (2002)]. The geometry of the indole is also consistent with that found in other 1,3-disubstituted indoles [*e.g.*, CSD:OBAVIW, CAS:256391–53-4, Biswas *et al.* (1999)].

S2. Experimental

The synthesis of **I** is detailed by Shui (1994), who prepared a suitable single-crystal by recrystallization from dichloromethane.

S3. Refinement

All H atoms were placed in calculated positions, guided by difference maps, with C—H bond distances 0.95 (aromatic C) and 0.99 (alkyl C) Å, and U_{iso}=1.2U_{eq}, thereafter refined as riding.

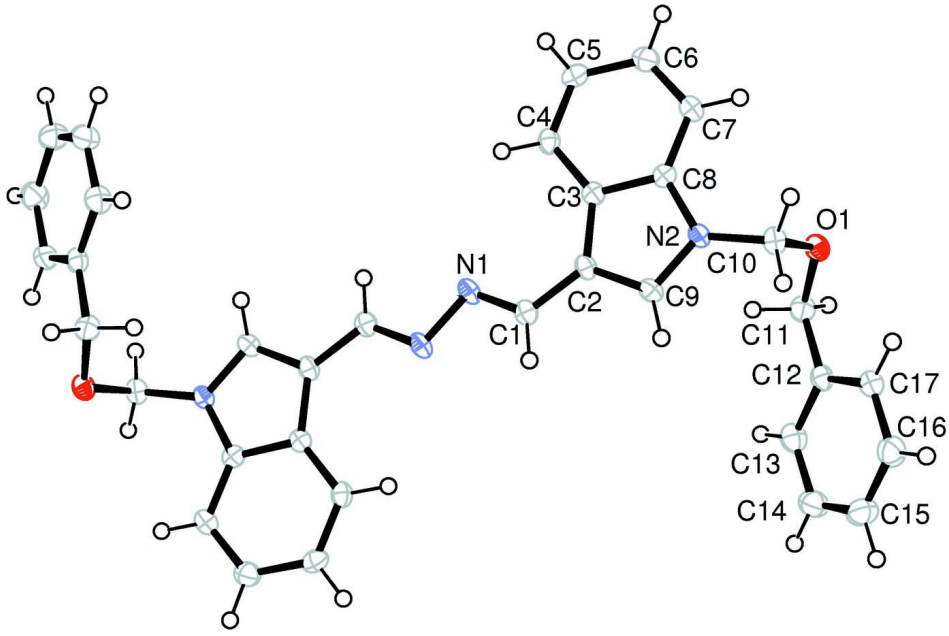

Figure 1

View of (I) (50% probability displacement ellipsoids)

(1*E*,2*E*)-1,2-Bis[(1-benzyloxymethyl-1*H*-indol- 3-yl)methylidene]hydrazine

Crystal data

$C_{34}H_{30}N_4O_2$
$M_r = 526.62$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.8518$ (10) Å
$b = 7.9663$ (9) Å
$c = 13.6810$ (12) Å
$\beta = 103.672$ (5)°
$V = 1361.0$ (2) Å^3
$Z = 2$

$F(000) = 556$
$D_x = 1.285$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2472 reflections
$\theta = 2.6$–$26.0°$
$\mu = 0.08$ mm^{-1}
$T = 90$ K
Fragment, colorless
0.18 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: sealed tube
Horizonally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor 1997)

$T_{min} = 0.986$, $T_{max} = 0.996$
4340 measured reflections
2678 independent reflections
1481 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.0°$
$h = -15 \rightarrow 15$
$k = -8 \rightarrow 9$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.126$
$S = 0.99$
2678 reflections
182 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0062 (13)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.02086 (15)	0.0030 (3)	0.55263 (14)	0.0211 (5)
C1	0.11765 (18)	−0.0511 (3)	0.57786 (17)	0.0206 (6)
H1	0.15	−0.0917	0.5268	0.025*
C2	0.17927 (18)	−0.0526 (3)	0.68083 (16)	0.0191 (6)
C3	0.14858 (18)	−0.0067 (3)	0.77188 (17)	0.0180 (6)
C4	0.05355 (19)	0.0445 (3)	0.79619 (18)	0.0207 (6)
H4	−0.0108	0.0538	0.7452	0.025*
C5	0.05426 (19)	0.0813 (3)	0.89494 (18)	0.0236 (6)
H5	−0.0098	0.1178	0.9115	0.028*
C6	0.14857 (19)	0.0654 (3)	0.97152 (19)	0.0258 (7)
H6	0.147	0.0915	1.0389	0.031*
C7	0.24299 (19)	0.0127 (3)	0.95068 (17)	0.0226 (6)
H7	0.3066	0.0011	1.0023	0.027*
C8	0.24147 (18)	−0.0229 (3)	0.85072 (17)	0.0182 (6)
N2	0.32454 (15)	−0.0785 (3)	0.81007 (14)	0.0190 (5)
C9	0.28592 (19)	−0.0961 (3)	0.70805 (17)	0.0205 (6)
H9	0.3269	−0.133	0.6628	0.025*
C10	0.43362 (18)	−0.1094 (3)	0.86494 (18)	0.0218 (6)
H10A	0.4327	−0.1657	0.9293	0.026*
H10B	0.468	−0.1866	0.8254	0.026*
O1	0.49589 (12)	0.0398 (2)	0.88566 (11)	0.0225 (4)
C11	0.50844 (19)	0.1279 (3)	0.79804 (18)	0.0241 (6)
H11A	0.5435	0.237	0.8194	0.029*
H11B	0.4365	0.152	0.7552	0.029*
C12	0.57299 (18)	0.0365 (3)	0.73523 (18)	0.0219 (6)
C13	0.5658 (2)	0.0890 (3)	0.63721 (18)	0.0276 (7)

H13	0.5168	0.1754	0.6092	0.033*
C14	0.6293 (2)	0.0168 (4)	0.57959 (19)	0.0329 (7)
H14	0.6248	0.0562	0.5132	0.039*
C15	0.6992 (2)	−0.1122 (4)	0.6180 (2)	0.0352 (7)
H15	0.7423	−0.1619	0.5782	0.042*
C16	0.7056 (2)	−0.1679 (4)	0.71520 (19)	0.0328 (7)
H16	0.7525	−0.2576	0.7419	0.039*
C17	0.64369 (19)	−0.0929 (3)	0.77379 (19)	0.0263 (7)
H17	0.6497	−0.1304	0.8408	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0247 (12)	0.0193 (12)	0.0164 (10)	0.0004 (11)	−0.0011 (9)	−0.0027 (10)
C1	0.0221 (14)	0.0174 (15)	0.0212 (13)	−0.0012 (12)	0.0030 (11)	−0.0005 (11)
C2	0.0203 (12)	0.0162 (14)	0.0186 (13)	0.0005 (11)	0.0004 (10)	−0.0009 (11)
C3	0.0209 (12)	0.0141 (14)	0.0185 (13)	−0.0019 (12)	0.0038 (10)	0.0015 (11)
C4	0.0190 (12)	0.0189 (14)	0.0225 (14)	−0.0022 (12)	0.0017 (10)	0.0011 (12)
C5	0.0235 (13)	0.0229 (16)	0.0272 (14)	−0.0017 (12)	0.0116 (11)	0.0002 (12)
C6	0.0292 (15)	0.0261 (17)	0.0228 (14)	−0.0024 (13)	0.0074 (12)	−0.0007 (12)
C7	0.0253 (13)	0.0217 (15)	0.0189 (13)	0.0000 (13)	0.0016 (11)	−0.0007 (12)
C8	0.0196 (12)	0.0136 (14)	0.0218 (13)	−0.0007 (12)	0.0059 (10)	0.0000 (11)
N2	0.0198 (11)	0.0198 (13)	0.0161 (11)	0.0003 (9)	0.0013 (8)	−0.0012 (9)
C9	0.0237 (13)	0.0197 (15)	0.0172 (13)	0.0002 (12)	0.0031 (10)	−0.0017 (11)
C10	0.0214 (13)	0.0179 (15)	0.0242 (14)	0.0003 (12)	0.0016 (11)	0.0000 (12)
O1	0.0240 (9)	0.0201 (10)	0.0220 (9)	−0.0030 (8)	0.0025 (7)	−0.0021 (8)
C11	0.0241 (13)	0.0227 (15)	0.0252 (14)	−0.0006 (12)	0.0052 (11)	0.0048 (12)
C12	0.0209 (13)	0.0204 (14)	0.0224 (13)	−0.0035 (12)	0.0016 (11)	−0.0009 (12)
C13	0.0292 (14)	0.0246 (16)	0.0274 (15)	−0.0002 (13)	0.0031 (12)	0.0045 (13)
C14	0.0357 (15)	0.0386 (19)	0.0251 (15)	−0.0055 (15)	0.0088 (12)	−0.0007 (14)
C15	0.0326 (16)	0.0410 (19)	0.0348 (17)	0.0004 (15)	0.0136 (13)	−0.0079 (15)
C16	0.0295 (15)	0.0346 (18)	0.0324 (16)	0.0066 (14)	0.0036 (12)	−0.0025 (14)
C17	0.0229 (14)	0.0330 (17)	0.0218 (14)	0.0017 (13)	0.0030 (11)	−0.0008 (13)

Geometric parameters (Å, º)

N1—C1	1.284 (3)	C9—H9	0.95
N1—N1^{i}	1.414 (4)	C10—O1	1.424 (3)
C1—C2	1.444 (3)	C10—H10A	0.99
C1—H1	0.95	C10—H10B	0.99
C2—C9	1.377 (3)	O1—C11	1.431 (3)
C2—C3	1.440 (3)	C11—C12	1.514 (3)
C3—C4	1.400 (3)	C11—H11A	0.99
C3—C8	1.413 (3)	C11—H11B	0.99
C4—C5	1.380 (3)	C12—C13	1.387 (3)
C4—H4	0.95	C12—C17	1.393 (4)
C5—C6	1.408 (3)	C13—C14	1.386 (3)
C5—H5	0.95	C13—H13	0.95

C6—C7	1.376 (3)	C14—C15	1.384 (4)
C6—H6	0.95	C14—H14	0.95
C7—C8	1.392 (3)	C15—C16	1.385 (4)
C7—H7	0.95	C15—H15	0.95
C8—N2	1.388 (3)	C16—C17	1.391 (3)
N2—C9	1.373 (3)	C16—H16	0.95
N2—C10	1.446 (3)	C17—H17	0.95

C1—N1—N1^{i}	111.9 (2)	O1—C10—N2	113.05 (19)
N1—C1—C2	122.6 (2)	O1—C10—H10A	109
N1—C1—H1	118.7	N2—C10—H10A	109
C2—C1—H1	118.7	O1—C10—H10B	109
C9—C2—C3	106.77 (19)	N2—C10—H10B	109
C9—C2—C1	123.2 (2)	H10A—C10—H10B	107.8
C3—C2—C1	130.0 (2)	C10—O1—C11	114.34 (17)
C4—C3—C8	118.2 (2)	O1—C11—C12	115.2 (2)
C4—C3—C2	135.4 (2)	O1—C11—H11A	108.5
C8—C3—C2	106.41 (19)	C12—C11—H11A	108.5
C5—C4—C3	119.3 (2)	O1—C11—H11B	108.5
C5—C4—H4	120.3	C12—C11—H11B	108.5
C3—C4—H4	120.3	H11A—C11—H11B	107.5
C4—C5—C6	121.0 (2)	C13—C12—C17	118.5 (2)
C4—C5—H5	119.5	C13—C12—C11	118.9 (2)
C6—C5—H5	119.5	C17—C12—C11	122.5 (2)
C7—C6—C5	121.3 (2)	C12—C13—C14	120.8 (2)
C7—C6—H6	119.4	C12—C13—H13	119.6
C5—C6—H6	119.4	C14—C13—H13	119.6
C6—C7—C8	117.2 (2)	C15—C14—C13	120.5 (2)
C6—C7—H7	121.4	C15—C14—H14	119.7
C8—C7—H7	121.4	C13—C14—H14	119.7
N2—C8—C7	128.7 (2)	C16—C15—C14	119.3 (3)
N2—C8—C3	108.3 (2)	C16—C15—H15	120.4
C7—C8—C3	123.0 (2)	C14—C15—H15	120.4
C9—N2—C8	108.20 (19)	C15—C16—C17	120.2 (3)
C9—N2—C10	125.7 (2)	C15—C16—H16	119.9
C8—N2—C10	126.11 (19)	C17—C16—H16	119.9
N2—C9—C2	110.3 (2)	C16—C17—C12	120.7 (2)
N2—C9—H9	124.9	C16—C17—H17	119.6
C2—C9—H9	124.9	C12—C17—H17	119.6

C1^{i}—N1^{i}—N1—C1	180	C7—C8—N2—C10	−1.0 (4)
N1^{i}—N1—C1—C2	177.9 (2)	C3—C8—N2—C10	179.2 (2)
N1—C1—C2—C9	−174.1 (2)	C8—N2—C9—C2	0.2 (3)
N1—C1—C2—C3	3.4 (4)	C10—N2—C9—C2	−178.6 (2)
C9—C2—C3—C4	−178.3 (3)	C3—C2—C9—N2	−0.8 (3)
C1—C2—C3—C4	4.0 (5)	C1—C2—C9—N2	177.2 (2)
C9—C2—C3—C8	1.0 (3)	C9—N2—C10—O1	98.5 (3)
C1—C2—C3—C8	−176.8 (2)	C8—N2—C10—O1	−80.1 (3)

C8—C3—C4—C5	1.6 (4)	N2—C10—O1—C11	−62.1 (3)
C2—C3—C4—C5	−179.2 (3)	C10—O1—C11—C12	−66.3 (2)
C3—C4—C5—C6	−1.0 (4)	O1—C11—C12—C13	163.0 (2)
C4—C5—C6—C7	0.0 (4)	O1—C11—C12—C17	−20.4 (3)
C5—C6—C7—C8	0.5 (4)	C17—C12—C13—C14	−1.4 (4)
C6—C7—C8—N2	−179.5 (2)	C11—C12—C13—C14	175.3 (2)
C6—C7—C8—C3	0.2 (4)	C12—C13—C14—C15	1.7 (4)
C4—C3—C8—N2	178.5 (2)	C13—C14—C15—C16	−0.4 (4)
C2—C3—C8—N2	−0.9 (3)	C14—C15—C16—C17	−1.0 (4)
C4—C3—C8—C7	−1.2 (4)	C15—C16—C17—C12	1.3 (4)
C2—C3—C8—C7	179.4 (2)	C13—C12—C17—C16	0.0 (4)
C7—C8—N2—C9	−179.8 (3)	C11—C12—C17—C16	−176.7 (2)
C3—C8—N2—C9	0.4 (3)		

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