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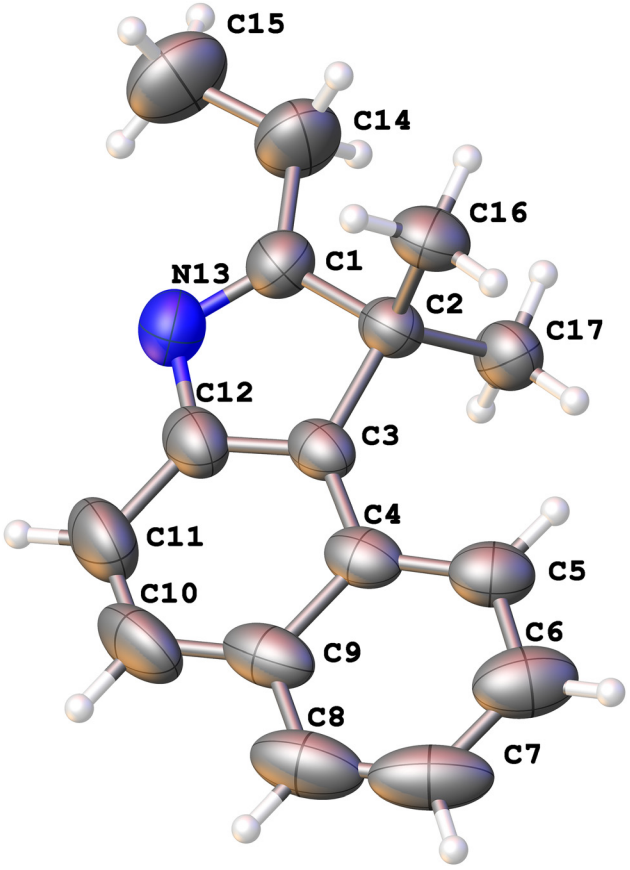
The crystal structure of 2-ethyl-1,1-dimethyl-1*H*-benzo[*e*]indole, $C_{16}H_{17}N$



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Abstract

$C_{16}H_{17}N$, orthorhombic, *Pbca* (no. 61), a = 10.3706(10) Å, b = 8.3879(7) Å, c = 29.555(3) Å, V = 2,570.9(4) Å^3, Z = 8, $R_{gt}(F)$ = 0.0579, w$R_{ref}(F^2)$ = 0.1327, T = 293(2) K.

CCDC no.: 2364731

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.15 × 0.14 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.1°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9,570, 3,001, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1987
$N(param)_{refined}$:	157
Programs:	CrysAlis^{PRO},[1] Olex2,[2] SHELX[3,4]

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

The chemical reagents are all commercially available and treated by standard methods. Under the nitrogen atmosphere, 4.18 g of 1,1,2-trimethyl-3*H*-benzo[*e*]indole was dispersed into 500 mL of tetrahydrofuran and then cooled to −70 °C. 7 mL of 2.5 mol/L *n*-butyl lithium *n*-hexane solution was cautiously added dropwise into the above solutions and refluxed for about 1 h. Subsequently, 3.5 g of iodomethane was added and reacted for another 3 h. After rising to room temperature, this reaction solution was charged into 70 mL of water, and then subjected to extraction by adding CH_2Cl_2. After concentrating the oil layer and drying with anhydrous sodium sulfate, a light yellow powder of 2-ethyl-1,1-dimethyl-1*H*-benzo[*e*]indole was obtained.

The single-crystal of 2-ethyl-3, 3-trimethyl-3*H*-benzo[*e*] indole, suitable for structural determination, was obtained by a solvent evaporation method. A certain amount of title compound was dissolved in ethyl acetate, slowly heated to form a saturated solution, then left standing at room temperature. The yellow block crystal can be harvested after two weeks.

2 Experimental details

The C–H bond distances were constrained to be between 0.90 and 0.97 Å; All H atoms in the ring and methylene group were

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.48707 (17)	0.6189 (2)	0.56929 (6)	0.0569 (5)
C2	0.56891 (15)	0.59777 (18)	0.61168 (6)	0.0485 (4)
C3	0.51569 (15)	0.44013 (17)	0.62826 (6)	0.0481 (4)
C4	0.54663 (16)	0.34352 (18)	0.66606 (6)	0.0523 (4)
C5	0.64513 (19)	0.3771 (2)	0.69743 (6)	0.0615 (5)
H5	0.694165	0.469165	0.694012	0.074*
C6	0.6700 (2)	0.2764 (2)	0.73296 (7)	0.0804 (6)
H6	0.736099	0.299928	0.753161	0.096*
C7	0.5960 (3)	0.1383 (3)	0.73883 (8)	0.0916 (8)
H7	0.612169	0.071175	0.763223	0.110*
C8	0.5012 (3)	0.1022 (2)	0.70924 (9)	0.0874 (8)
H8	0.453242	0.009721	0.713582	0.105*
C9	0.4731 (2)	0.2010 (2)	0.67191 (7)	0.0671 (6)
C10	0.3757 (2)	0.1620 (2)	0.64037 (9)	0.0865 (7)
H10	0.328342	0.068984	0.644525	0.104*
C11	0.34913 (19)	0.2557 (3)	0.60432 (9)	0.0840 (7)
H11	0.285404	0.227307	0.583696	0.101*
C12	0.42015 (16)	0.3969 (2)	0.59877 (7)	0.0607 (5)
C14	0.5033 (2)	0.7576 (3)	0.53812 (8)	0.0866 (7)
H14A	0.503921	0.854127	0.556194	0.104*
H14B	0.587175	0.748856	0.523798	0.104*
C15	0.4043 (2)	0.7766 (3)	0.50183 (8)	0.0993 (8)
H15A	0.438377	0.842543	0.478129	0.149*
H15B	0.328647	0.825723	0.514299	0.149*
H15C	0.382237	0.673893	0.489739	0.149*
C16	0.54012 (19)	0.73327 (19)	0.64531 (6)	0.0633 (5)
H16A	0.449027	0.738040	0.650932	0.095*
H16B	0.568606	0.832809	0.632779	0.095*
H16C	0.584772	0.713482	0.673207	0.095*
C17	0.71228 (15)	0.5919 (2)	0.59964 (6)	0.0598 (5)
H17A	0.761758	0.571143	0.626454	0.090*
H17B	0.738045	0.692264	0.586923	0.090*
H17C	0.727015	0.508545	0.577984	0.090*
N13	0.40370 (14)	0.50797 (18)	0.56280 (6)	0.0667 (4)

refined using riding coordinates with U_{iso}(H) = 1.2U_{eq}(C), whereas the H atoms of methyl groups were placed in the idealized position with U_{iso}(H) = 1.5U_{eq}(C).

3 Comment

Nitrogen-containing heterocyclic rings are one important source of new pharmaceuticals,[5] and functional materials.[6] Amongst them, benzindole and its derivatives are the most commonly rigid aromatic compounds for various electroluminescent or photoelectric materials[4] and pharmaceutical intermediates[5] and also structural motifs in biologically active molecules.[5] In our synthesis of raw materials for dye-sensitized solar cells, we unexpectedly obtained the single-crystal of 2-ethyl-1,1-dimethyl-1*H*-benzo[*e*]indole.

2–Ethyl-1,1-dimethyl-1*H*-benzo[*e*]indole crystallizes in the orthorhombic space group *Pbca* and its asymmetric unit contains only one title compound molecule without any solvent attachment as shown in figure. In the structure, three aromatic rings are approximately coplanar, which is similar to the reported 1,1-dimethyl-1*H*-benzo[*e*]indole-2-carbonitrile[7] and 3-ethyl-1,1,2-trimethyl-1*H*-benzo[*e*]indolium iodide.[8] The rigid planar structure is beneficial for electron delocalization. The lengths of C12–N13, C1–N13, C1–C2 and C2–N3 bonds are 1.424(2), 1.284 (2), 1.523(2) and 1.514(2) Å, respectively, and are all in the expected ranges of neutral molecule.[9] Two methyl groups are located on each side of the indole, and the plane formed by two methyl groups (C16–C2–C17) is closely perpendicular to the indole ring plane with dihedral angles of 89.64°. The ethyl group tends to the side of the N atom in the indole ring, probably influenced by the steric hindrance of the two methyl groups, and the dihedral angle between the plane of the ethyl group (C15–C14–C1) and the indole plane is only 8.90°. The molecule in the asymmetric unit is linked by weak C8–H8···C6 short contact with the shortest C···C distance of 2.82 Å within two adjacent molecules, respectively. In addition, the C–H···π interaction is a stabilizing feature with the shortest distance (2.55 and 2.62 Å) between the two H atoms of two methyl groups and two benzene rings in indole. Surprisingly, no π–π interactions are found for this crystal.

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