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Crystal structure of 1-(2-(4-bromophenyl)-2,3-dihydro-1*H*-benzo[*e*]indol-1-yl)-naphthalen-2-ol – dichloromethane – dimethyl sulfoxide (1/1/1), $C_{28}H_{18}BrNO \cdot CH_2Cl_2 \cdot C_2H_6SO$

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Abstract

$C_{28}H_{18}BrNO$, triclinic, $P\bar{1}$ (no. 2), $a = 11.7627(7)$ Å, $b = 11.8731(9)$ Å, $c = 11.9240(7)$ Å, $\alpha = 76.952(3)^\circ$, $\beta = 85.718(6)^\circ$, $\gamma = 60.534(3)^\circ$, $V = 1411.16(16)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0497$, $wR_{ref}(F^2) = 0.1365$, $T = 273$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.10 \times 0.08 \times 0.08$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.75 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	28.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	29548, 7018, 0.050
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6502
$N(\text{param})_{\text{refined}}$:	345
Programs:	Bruker [1], SHELX [2], Olex2 [3]

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.

Source of material

In an argon atmosphere, with constant magnetic stirring, the reaction mixture of 3-(4-bromophenyl)-2*H*-azirine (33 g,

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.16574 (4)	0.96114 (3)	−0.20009 (2)	0.05149 (12)
O1	−0.06193 (14)	0.58713 (16)	0.24543 (13)	0.0314 (3)
H1	−0.119251	0.654832	0.263737	0.047*
N1	0.16185 (16)	0.36401 (17)	−0.00787 (14)	0.0228 (3)
H1A	0.158270	0.376514	−0.081713	0.027*
C1	0.16119 (17)	0.45067 (19)	0.05295 (16)	0.0215 (3)
C2	0.16866 (17)	0.39531 (18)	0.16987 (15)	0.0203 (3)
C3	0.17351 (17)	0.27056 (18)	0.17996 (16)	0.0210 (3)
C4	0.16832 (18)	0.2555 (2)	0.06781 (17)	0.0237 (4)
C5	0.15999 (19)	0.5727 (2)	−0.00815 (16)	0.0245 (4)
C6	0.1707 (3)	0.6558 (3)	0.0512 (2)	0.0458 (7)
H6	0.176651	0.634042	0.131318	0.055*
C7	0.1726 (4)	0.7695 (3)	−0.0060 (2)	0.0554 (8)
H7	0.180460	0.823055	0.035491	0.066*
C8	0.1629 (3)	0.8040 (3)	−0.1246 (2)	0.0375 (5)
C9	0.1514 (2)	0.7252 (2)	−0.18709 (18)	0.0323 (4)
H9	0.144432	0.748686	−0.267157	0.039*
C10	0.1503 (2)	0.6101 (2)	−0.12873 (17)	0.0275 (4)
H10	0.142885	0.556717	−0.170794	0.033*
C11	0.1671 (2)	0.1454 (2)	0.0432 (2)	0.0350 (5)
H11	0.164990	0.137222	−0.032325	0.042*
C12	0.1690 (3)	0.0517 (3)	0.1339 (2)	0.0427 (6)
H12	0.168879	−0.021967	0.119398	0.051*
C13	0.1713 (2)	0.0619 (2)	0.2509 (2)	0.0325 (4)
C14	0.17453 (18)	0.17214 (19)	0.27576 (17)	0.0235 (4)
C15	0.1736 (2)	0.1801 (2)	0.39254 (18)	0.0277 (4)
H15	0.176842	0.250784	0.410530	0.033*
C16	0.1681 (2)	0.0853 (2)	0.4797 (2)	0.0341 (5)
H16	0.167641	0.092440	0.555818	0.041*
C17	0.1632 (3)	−0.0221 (2)	0.4550 (2)	0.0389 (5)
H17	0.158625	−0.085502	0.514435	0.047*
C18	0.1653 (3)	−0.0333 (2)	0.3428 (2)	0.0420 (6)
H18	0.162594	−0.105189	0.326959	0.050*
C19	0.16789 (18)	0.45365 (19)	0.26792 (16)	0.0210 (3)
C20	0.0518 (2)	0.5467 (2)	0.30344 (16)	0.0244 (4)
C21	0.0492 (2)	0.5992 (2)	0.39974 (19)	0.0316 (4)
H21	−0.030050	0.661940	0.422208	0.038*
C22	0.1619 (3)	0.5585 (2)	0.45970 (19)	0.0344 (5)
H22	0.158653	0.593216	0.523162	0.041*
C23	0.2839 (2)	0.4640 (2)	0.42657 (18)	0.0290 (4)
C24	0.28696 (19)	0.41168 (19)	0.32951 (16)	0.0237 (4)
C25	0.4097 (2)	0.3191 (2)	0.29589 (19)	0.0300 (4)
H25	0.413667	0.283295	0.232959	0.036*
C26	0.5235 (2)	0.2812 (3)	0.3552 (2)	0.0384 (5)
H26	0.603372	0.221116	0.331439	0.046*
C27	0.5197 (3)	0.3328 (3)	0.4516 (2)	0.0431 (6)
H27	0.596831	0.306529	0.491455	0.052*
C28	0.4029 (3)	0.4214 (3)	0.4863 (2)	0.0387 (5)
H28	0.401134	0.454491	0.550364	0.046*
Cl1	0.60361 (10)	0.32222 (11)	−0.24122 (7)	0.0686 (3)
Cl2	0.49334 (13)	0.31539 (16)	−0.01698 (13)	0.1131 (5)
C31	0.5110 (5)	0.4146 (5)	−0.1412 (5)	0.103 (2)
H31A	0.552827	0.460514	−0.120760	0.124*
H31B	0.425249	0.480794	−0.175791	0.124*
S1	−0.38850 (6)	0.82992 (6)	0.28085 (5)	0.03470 (14)

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O2	−0.25120 (16)	0.80849 (17)	0.28534 (17)	0.0392 (4)
C29	−0.4708 (3)	0.9238 (3)	0.3857 (3)	0.0480 (6)
H29A	−0.460784	1.001257	0.371429	0.072*
H29B	−0.434304	0.871026	0.460934	0.072*
H29C	−0.561955	0.950058	0.381476	0.072*
C30	−0.4656 (3)	0.9563 (4)	0.1554 (3)	0.0656 (10)
H30A	−0.428230	0.922117	0.088076	0.098*
H30B	−0.452667	1.029875	0.156417	0.098*
H30C	−0.557472	0.985192	0.154022	0.098*

0.17 mol) and 2-naphthol (48 g, 0.34 mol) was heated in dichloromethane solvent at reflux overnight in the presence of a copper catalyst. The product was purified by chromatography to afford the title product as a white solid in 35% yield. Crystals of the title compound were obtained by slow evaporation from a dichloromethane/DMSO/hexanes solvent mixture in a freezer.

Experimental details

The raw area detector data frames were reduced and corrected for absorption effects using the SAINT+ and SADABS programs [1]. Hydrogen atoms were placed in geometrically idealized positions and refined as standard riding atoms.

Comment

Nitrogen containing heterocyclic scaffolds are important molecules which may act as bioactive substances [4] and functional materials [5–7]. The construction of such molecules has become a long-term concern of synthetic chemists. In order to develop new methods to obtain important heterocyclic compounds, new transition metal catalyzed reactions with multifunctional synthetic intermediates have attracted much attention.

In the structure of the title compound, the asymmetric unit consists of one of the target molecule, one dichloromethane and one dimethyl sulfoxide solvent molecules. As the ORTEP-type figure of the structure shows, the bromophenyl group resides in the same plane as the benzindole ring, but is almost perpendicular to the naphthol ring. There is a hydrogen bond between O2 and H1 with the distance of 1.78 Å and O2–H2–O1 of 172.9°, indicating a strong intermolecular interaction. The angles and bond

lengths in all three molecules are in line with previous studies [8–10].

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