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# 1,3-Bis(3-phenylpropyl)benzimidazolium bromide monohydrate

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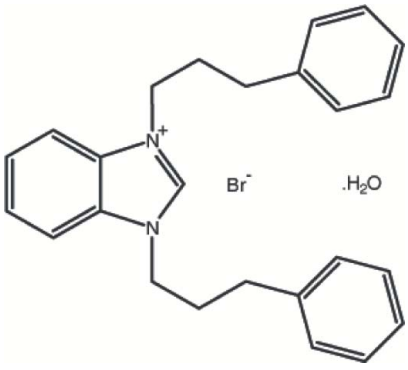
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.010 Å; $R$ factor = 0.065; $wR$ factor = 0.172; data-to-parameter ratio = 19.7.

In the title compound, $C_{25}H_{27}N_2^+\cdot Br^-\cdot H_2O$, the benzimidazole unit is essentially planar, with a maximum deviation of 0.020 (6) Å. The benzimidazole unit makes dihedral angles of 83.6 (3) and 81.0 (3)° with the two terminal phenyl rings. The dihedral angle between the phenyl rings is 58.5 (4)°. In the crystal structure, there are C—H···O hydrogen bonds, a C—H···$\pi$ interaction between a phenyl H atom and the phenyl ring of a neighbouring molecule, and a $\pi$–$\pi$ interaction [3.512 (3) Å] between the centroids of the five-membered ring and the benzene ring of the benzimidazole unit of an adjacent molecule.

## Related literature

For general background, see: Sakai *et al.* (1989); Küçükbay *et al.* (2001, 2003, 2004). For a similar structure, see: Akkurt *et al.* (2005). For related structures, see: Akkurt *et al.* (2004, 2007); Karaca *et al.* (2005); Pınar *et al.* (2006); Yıldırım *et al.* (2005).




## Experimental

### Crystal data

$C_{25}H_{27}N_2^+\cdot Br^-\cdot H_2O$
$M_r$ = 453.40
Monoclinic, $P2_1/c$
$a$ = 14.1933 (8) Å
$b$ = 11.4594 (3) Å
$c$ = 18.3014 (10) Å
$\beta$ = 128.916 (3)°
$V$ = 2316.1 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.79 mm$^{-1}$
$T$ = 295 (2) K
0.71 × 0.63 × 0.54 mm

### Data collection

Stoe IPDS II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.363, $T_{max}$ = 0.444
18735 measured reflections
5283 independent reflections
2688 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.071

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.172
$S$ = 0.99
5283 reflections
268 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7···O1 | 0.93 | 2.50 | 3.257 (10) | 139 |
| C17—H17$A$···O1 | 0.97 | 2.38 | 3.236 (13) | 148 |
| C24—H24···$Cg1^{i}$ | 0.93 | 2.84 | 3.771 (14) | 176 |

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$. $Cg1$ is the centriod of the C11–C16 phenyl ring.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); 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* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS II diffractometer (purchased under grant F.279 of the University Research Fund). HK and ÜY thank İnönü University Research Fund (Directed project BAPB-2008/60) for financial support ofthis study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2337).

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# supplementary materials

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# 1,3-Bis(3-phenylpropyl)benzimidazolium bromide monohydrate

**M. Akkurt, S. Karaca, Ü. Yilmaz, H. Küçükbay and O. Büyükgüngör**

## Comment

Benzimidazole and related heterocyclic compounds have been extensively investigated because of their versatile pharmacological activity. They are also present in various naturally occurring drugs such as omeprazole, astemizole andemedastine difumarate (Sakai *et al.*, 1989). Substituted benzimidazole moieties are estabilished pharmacophores in parasitic chemotheraphy. In some previous papers (Küçükbay *et al.*, 2001, 2003, 2004), we reported the synthesis and antimicrobial activity of some benzimidazole derivatives. The objective of this study was to elucidate the crystal structure of the title compound, (I).

In the title molecule (I) (Fig. 1), the values of the geometric parameters are comparable with those of previously reported structures (Akkurt *et al.*, 2004, 2005, 2007; Pınar *et al.*, 2006; Yıldırım *et al.*, 2005; Karaca *et al.*, 2005). The benzimidazole unit (N1/N2/C1–C7) is essentially planar, with a maximum deviation of 0.020 (6) Å for C7 from the least-squares plane defined by the nine constituent atoms. The benzimidazole unit makes the dihedral angles of 83.6 (3) and 81.0 (3)° with the two terminal phenyl rings (C11–C16) and (C20–C25), respectively. The dihedral angle between the phenyl rings is 58.5 (4)°.

Molecular conformation is stabilized by intramolecular C—H···O hydrogen bonding interactions. The molecular packing (Fig. 2) is stabilized by a C—H···π interaction between a phenyl H atom and the phenyl ring of neighbouring molecules, with a C24—H24···*Cg*1$^{ii}$ separation of 2.84 Å [Table 1; *Cg*1 is the C11–C16 phenyl ring, symmetry code: (ii) *x*, -*y* + 1/2, *z* + 1/2]. In the crystal packing, there is also a π–π interaction with a distance of 3.512 (3) Å between the centroids of the five-membered ring (N1/N2/C1/C6/C7) (centroid *Cg*2) and the benzene ring (C1–C6) (centroid *Cg*3) of the benzimidazole unit of the adjacent molecule.

## Experimental

A solution of 1-(3-phenylpropyl)benzimidazole (4.20 g,17.80 mmol) and $Ph(CH_2)_3Br$ (2.70 ml, 17.85 mmol) was refluxed in DMF for 4 h. The mixture was then cooled and the volatiles were removed from the filtrate *in vacuo*. The residue obtained was then crytallized from EtOH/$Et_2O$(1:5) (yield 6.50 g, 84%; m.p. 376–377 K). $^1$H-NMR (DMSO-$d_6$): δ (p.p.m.) 2.26 (*p*, $CH_2$, 4H), 2.73 (*t*, $CH_2$—Ar, 4H), 4.55 (t, $CH_2$—N, 4H), 7.14–8.13 (*m*, Ar—H, 14H), 9.99 (s, >CH, 1H). $^{13}$C-NMR (DMSO-$d_6$): δ 30.5, 32.4, 47.0, 114.2, 126.5, 126.9, 128.7, 131.6, 141.4, 142.7. Analysis calculated for $C_{25}H_{29}N_2OBr$: C 66.23, H 6.40, N 6.18%; found: C 65.99, H 6.13, N 6.10%.

## Refinement

Water H atoms were found in a difference Fourier map and distance restraints [O—H = 0.84 (9) and H···H = 1.37 (9) Å] were used to obtain reasonable values for O–H distances and H—O—H angles, with $U_{iso}$(H) = 1.5$U_{eq}$(O). Other H atoms were positioned to the ideal geometric positions and refined with a riding model, with C—H = 0.93 and 0.97 Å, and with $U_{iso}$ = 1.2$U_{eq}$(C).

## Figures

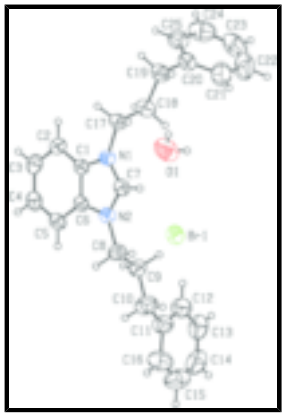

Fig. 1. The title molecule (I), with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

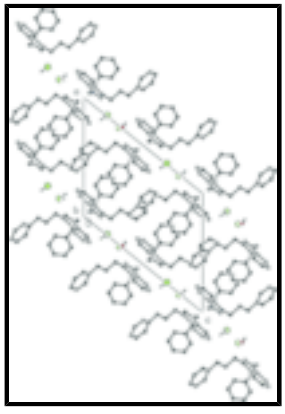

Fig. 2. The crystal packing diagram of (I), down the *b* axis. Dashed lines indicate hydrogen contacts. H atoms not involved in hydrogen bonding have been omitted for clarity.

### 1,3-Bis(3-phenylpropyl)benzimidazolium bromide monohydrate

*Crystal data*

| | |
|---|---|
| $C_{25}H_{27}N_2^+ \cdot Br^- \cdot H_2O$ | $F_{000}$ = 944 |
| $M_r$ = 453.40 | $D_x$ = 1.300 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 13886 reflections |
| $a$ = 14.1933 (8) Å | θ = 1.4–28.0º |
| $b$ = 11.4594 (3) Å | μ = 1.79 mm$^{-1}$ |
| $c$ = 18.3014 (10) Å | $T$ = 295 (2) K |
| β = 128.916 (3)º | Block, colourless |
| $V$ = 2316.1 (2) Å$^3$ | 0.71 × 0.63 × 0.54 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Stoe IPDS II diffractometer | 5283 independent reflections |
| Monochromator: plane graphite | 2688 reflections with $I > 2\sigma(I)$ |

| | |
|---|---|
| Detector resolution: 6.67 pixels $mm^{-1}$ | $R_{int}$ = 0.071 |
| $T$ = 295(2) K | $\theta_{max}$ = 27.6º |
| ω scans | $\theta_{min}$ = 1.8º |
| Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002) | $h$ = −18→18 |
| $T_{min}$ = 0.363, $T_{max}$ = 0.444 | $k$ = −13→14 |
| 18735 measured reflections | $l$ = −23→23 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.065 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.173 | $w = 1/[\sigma^2(F_o^2) + (0.0827P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 0.99 | $(\Delta/\sigma)_{max} < 0.001$ |
| 5283 reflections | $\Delta\rho_{max}$ = 0.47 e $Å^{-3}$ |
| 268 parameters | $\Delta\rho_{min}$ = −0.23 e $Å^{-3}$ |
| 3 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | −0.1093 (3) | 0.1691 (3) | 0.9193 (2) | 0.0541 (10) |
| N2 | −0.1505 (3) | 0.0103 (3) | 0.8391 (2) | 0.0539 (11) |
| C1 | −0.1415 (3) | 0.0860 (3) | 0.9554 (3) | 0.0514 (11) |
| C2 | −0.1469 (4) | 0.0912 (4) | 1.0285 (3) | 0.0622 (14) |
| C3 | −0.1784 (4) | −0.0099 (4) | 1.0477 (3) | 0.0753 (17) |
| C4 | −0.2034 (4) | −0.1121 (4) | 0.9973 (3) | 0.0742 (17) |
| C5 | −0.1995 (4) | −0.1173 (3) | 0.9243 (3) | 0.0645 (16) |
| C6 | −0.1673 (3) | −0.0157 (3) | 0.9049 (3) | 0.0505 (11) |
| C7 | −0.1159 (4) | 0.1196 (3) | 0.8509 (3) | 0.0587 (12) |

| | | | | |
|---|---|---|---|---|
| C8 | −0.1677 (4) | −0.0701 (4) | 0.7691 (3) | 0.0700 (17) |
| C9 | −0.3031 (5) | −0.0933 (4) | 0.6894 (3) | 0.0853 (19) |
| C10 | −0.3233 (6) | −0.1767 (6) | 0.6209 (4) | 0.105 (2) |
| C11 | −0.4522 (5) | −0.2161 (5) | 0.5455 (4) | 0.0905 (19) |
| C12 | −0.5522 (5) | −0.1513 (5) | 0.5160 (4) | 0.102 (2) |
| C13 | −0.6656 (6) | −0.1874 (7) | 0.4419 (4) | 0.107 (3) |
| C14 | −0.6809 (8) | −0.2866 (8) | 0.3982 (5) | 0.121 (3) |
| C15 | −0.5849 (10) | −0.3547 (7) | 0.4275 (6) | 0.128 (4) |
| C16 | −0.4716 (7) | −0.3209 (6) | 0.5006 (6) | 0.113 (3) |
| C17 | −0.0775 (4) | 0.2925 (3) | 0.9491 (3) | 0.0652 (16) |
| C18 | −0.1947 (5) | 0.3644 (4) | 0.8951 (4) | 0.0844 (19) |
| C19 | −0.1744 (5) | 0.4916 (4) | 0.9136 (4) | 0.085 (2) |
| C20 | −0.2942 (5) | 0.5556 (4) | 0.8583 (4) | 0.079 (2) |
| C21 | −0.3563 (8) | 0.5820 (7) | 0.7649 (5) | 0.126 (3) |
| C22 | −0.4712 (9) | 0.6297 (8) | 0.7129 (6) | 0.166 (4) |
| C23 | −0.5191 (7) | 0.6575 (6) | 0.7529 (8) | 0.134 (4) |
| C24 | −0.4537 (8) | 0.6398 (7) | 0.8464 (8) | 0.132 (4) |
| C25 | −0.3441 (6) | 0.5865 (5) | 0.8976 (5) | 0.093 (2) |
| O1 | 0.0138 (8) | 0.3343 (4) | 0.8276 (5) | 0.145 (3) |
| Br1 | 0.01170 (5) | 0.12751 (4) | 0.69968 (4) | 0.0869 (2) |
| H2 | −0.13010 | 0.15940 | 1.06230 | 0.0750* |
| H3 | −0.18330 | −0.01050 | 1.09600 | 0.0900* |
| H4 | −0.22340 | −0.17910 | 1.01360 | 0.0890* |
| H5 | −0.21740 | −0.18530 | 0.89000 | 0.0780* |
| H7 | −0.09830 | 0.15740 | 0.81580 | 0.0700* |
| H8A | −0.12700 | −0.14330 | 0.79930 | 0.0850* |
| H8B | −0.13160 | −0.03690 | 0.74310 | 0.0850* |
| H9A | −0.33960 | −0.12290 | 0.71610 | 0.1020* |
| H9B | −0.34280 | −0.02040 | 0.65790 | 0.1020* |
| H10A | −0.27450 | −0.24520 | 0.65450 | 0.1260* |
| H10B | −0.29360 | −0.14250 | 0.59040 | 0.1260* |
| H12 | −0.54230 | −0.08240 | 0.54700 | 0.1230* |
| H13 | −0.73240 | −0.14220 | 0.42180 | 0.1270* |
| H14 | −0.75850 | −0.30960 | 0.34680 | 0.1460* |
| H15 | −0.59700 | −0.42500 | 0.39730 | 0.1540* |
| H16 | −0.40610 | −0.36840 | 0.52080 | 0.1360* |
| H17A | −0.02540 | 0.32160 | 0.93590 | 0.0780* |
| H17B | −0.03440 | 0.29890 | 1.01610 | 0.0780* |
| H18A | −0.24030 | 0.35110 | 0.82840 | 0.1010* |
| H18B | −0.24350 | 0.33690 | 0.91190 | 0.1010* |
| H19A | −0.12700 | 0.52050 | 0.89590 | 0.1020* |
| H19B | −0.12920 | 0.50610 | 0.98010 | 0.1020* |
| H21 | −0.32100 | 0.56780 | 0.73680 | 0.1510* |
| H22 | −0.51500 | 0.64220 | 0.64880 | 0.2000* |
| H23 | −0.59660 | 0.68890 | 0.71770 | 0.1610* |
| H24 | −0.48460 | 0.66450 | 0.87600 | 0.1580* |
| H25 | −0.30310 | 0.57120 | 0.96090 | 0.1110* |
| HW1 | −0.042 (7) | 0.364 (8) | 0.775 (4) | 0.2170* |
| HW2 | 0.071 (6) | 0.383 (7) | 0.854 (7) | 0.2170* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0533 (19) | 0.0500 (15) | 0.0617 (19) | −0.0075 (14) | 0.0374 (17) | −0.0040 (15) |
| N2 | 0.0536 (19) | 0.0543 (17) | 0.0612 (19) | −0.0002 (14) | 0.0396 (17) | −0.0013 (14) |
| C1 | 0.038 (2) | 0.0523 (19) | 0.054 (2) | 0.0024 (16) | 0.0242 (18) | 0.0012 (17) |
| C2 | 0.059 (3) | 0.070 (2) | 0.058 (2) | −0.002 (2) | 0.037 (2) | −0.0056 (19) |
| C3 | 0.079 (3) | 0.094 (3) | 0.065 (3) | 0.001 (3) | 0.051 (3) | 0.010 (2) |
| C4 | 0.079 (3) | 0.070 (3) | 0.080 (3) | −0.003 (2) | 0.053 (3) | 0.015 (2) |
| C5 | 0.067 (3) | 0.052 (2) | 0.074 (3) | −0.004 (2) | 0.044 (2) | 0.001 (2) |
| C6 | 0.043 (2) | 0.0499 (19) | 0.057 (2) | 0.0042 (16) | 0.0307 (19) | 0.0045 (17) |
| C7 | 0.054 (2) | 0.056 (2) | 0.069 (2) | −0.0047 (19) | 0.040 (2) | 0.004 (2) |
| C8 | 0.078 (3) | 0.071 (3) | 0.074 (3) | −0.004 (2) | 0.054 (3) | −0.010 (2) |
| C9 | 0.087 (4) | 0.080 (3) | 0.066 (3) | 0.013 (3) | 0.037 (3) | −0.004 (2) |
| C10 | 0.100 (4) | 0.116 (4) | 0.101 (4) | 0.011 (4) | 0.064 (4) | −0.021 (4) |
| C11 | 0.071 (3) | 0.088 (3) | 0.089 (4) | −0.002 (3) | 0.039 (3) | −0.016 (3) |
| C12 | 0.078 (4) | 0.099 (4) | 0.089 (4) | 0.008 (3) | 0.033 (3) | −0.010 (3) |
| C13 | 0.075 (4) | 0.133 (5) | 0.091 (4) | −0.012 (4) | 0.042 (4) | 0.015 (4) |
| C14 | 0.116 (6) | 0.141 (6) | 0.084 (4) | −0.059 (5) | 0.052 (4) | −0.009 (4) |
| C15 | 0.158 (8) | 0.116 (5) | 0.138 (6) | −0.047 (6) | 0.106 (7) | −0.041 (5) |
| C16 | 0.108 (5) | 0.099 (4) | 0.152 (6) | −0.006 (4) | 0.091 (5) | −0.020 (4) |
| C17 | 0.068 (3) | 0.050 (2) | 0.078 (3) | −0.0074 (19) | 0.046 (3) | −0.0058 (19) |
| C18 | 0.082 (3) | 0.067 (3) | 0.101 (4) | −0.002 (2) | 0.056 (3) | −0.009 (3) |
| C19 | 0.093 (4) | 0.059 (3) | 0.100 (4) | −0.008 (2) | 0.059 (3) | −0.006 (2) |
| C20 | 0.087 (4) | 0.064 (3) | 0.090 (4) | 0.001 (2) | 0.058 (3) | 0.006 (2) |
| C21 | 0.143 (6) | 0.133 (5) | 0.111 (5) | 0.037 (5) | 0.084 (5) | 0.026 (4) |
| C22 | 0.161 (8) | 0.177 (9) | 0.109 (6) | 0.082 (7) | 0.060 (6) | 0.045 (5) |
| C23 | 0.084 (5) | 0.103 (5) | 0.174 (8) | 0.030 (4) | 0.061 (6) | 0.019 (5) |
| C24 | 0.129 (6) | 0.121 (6) | 0.174 (8) | 0.014 (5) | 0.109 (6) | −0.024 (6) |
| C25 | 0.104 (4) | 0.086 (3) | 0.107 (4) | 0.002 (3) | 0.075 (4) | −0.005 (3) |
| O1 | 0.248 (6) | 0.096 (3) | 0.196 (5) | 0.007 (4) | 0.190 (5) | 0.018 (3) |
| Br1 | 0.1042 (4) | 0.0715 (3) | 0.0851 (4) | −0.0021 (3) | 0.0595 (3) | 0.0094 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—HW1 | 0.84 (7) | C23—C24 | 1.355 (16) |
| O1—HW2 | 0.84 (11) | C24—C25 | 1.356 (15) |
| N1—C1 | 1.390 (6) | C2—H2 | 0.9300 |
| N1—C7 | 1.323 (6) | C3—H3 | 0.9300 |
| N1—C17 | 1.480 (5) | C4—H4 | 0.9300 |
| N2—C6 | 1.399 (6) | C5—H5 | 0.9300 |
| N2—C8 | 1.469 (6) | C7—H7 | 0.9300 |
| N2—C7 | 1.313 (5) | C8—H8B | 0.9700 |
| C1—C6 | 1.386 (5) | C8—H8A | 0.9700 |
| C1—C2 | 1.389 (7) | C9—H9B | 0.9700 |
| C2—C3 | 1.365 (7) | C9—H9A | 0.9700 |
| C3—C4 | 1.392 (6) | C10—H10A | 0.9700 |
| C4—C5 | 1.373 (8) | C10—H10B | 0.9700 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.376 (6) | C12—H12 | 0.9300 |
| C8—C9 | 1.539 (8) | C13—H13 | 0.9300 |
| C9—C10 | 1.455 (8) | C14—H14 | 0.9300 |
| C10—C11 | 1.511 (10) | C15—H15 | 0.9300 |
| C11—C12 | 1.376 (11) | C16—H16 | 0.9300 |
| C11—C16 | 1.382 (9) | C17—H17A | 0.9700 |
| C12—C13 | 1.363 (10) | C17—H17B | 0.9700 |
| C13—C14 | 1.327 (12) | C18—H18B | 0.9700 |
| C14—C15 | 1.355 (17) | C18—H18A | 0.9700 |
| C15—C16 | 1.350 (15) | C19—H19B | 0.9700 |
| C17—C18 | 1.535 (9) | C19—H19A | 0.9700 |
| C18—C19 | 1.483 (7) | C21—H21 | 0.9300 |
| C19—C20 | 1.514 (10) | C22—H22 | 0.9300 |
| C20—C25 | 1.336 (12) | C23—H23 | 0.9300 |
| C20—C21 | 1.377 (9) | C24—H24 | 0.9300 |
| C21—C22 | 1.384 (16) | C25—H25 | 0.9300 |
| C22—C23 | 1.313 (19) | | |
| Br1···O1 | 3.318 (8) | HW2···Br1$^{iii}$ | 2.96 (8) |
| Br1···O1$^{i}$ | 3.383 (5) | HW2···H8B$^{iii}$ | 2.5800 |
| Br1···HW1$^{i}$ | 3.04 (9) | H3···Br1$^{iv}$ | 3.2000 |
| Br1···H17B$^{ii}$ | 3.1100 | H5···C8 | 3.0200 |
| Br1···H8A$^{iii}$ | 3.0900 | H5···C20$^{vi}$ | 3.0900 |
| Br1···H3$^{iv}$ | 3.2000 | H5···H9A | 2.5900 |
| Br1···HW2$^{i}$ | 2.96 (8) | H7···O1 | 2.5000 |
| Br1···H8B | 3.2200 | H7···H17A | 2.5700 |
| Br1···H2$^{ii}$ | 3.1600 | H7···H8B | 2.4800 |
| Br1···H13$^{v}$ | 3.1000 | H8A···H10A | 2.4100 |
| O1···C17 | 3.236 (13) | H8A···Br1$^{i}$ | 3.0900 |
| O1···Br1$^{iii}$ | 3.383 (5) | H8A···C5 | 3.0600 |
| O1···C7 | 3.257 (10) | H8B···H10B | 2.5500 |
| O1···Br1 | 3.318 (8) | H8B···HW2$^{i}$ | 2.5800 |
| O1···H17A | 2.3800 | H8B···H7 | 2.4800 |
| O1···H7 | 2.5000 | H8B···Br1 | 3.2200 |
| N1···N2 | 2.175 (5) | H9A···C5 | 2.9900 |
| N2···N1 | 2.175 (5) | H9A···C12 | 2.9600 |
| C1···C6$^{iv}$ | 3.509 (7) | H9A···H5 | 2.5900 |
| C2···C8$^{iv}$ | 3.597 (7) | H9A···C6 | 2.9700 |
| C3···C7$^{iv}$ | 3.568 (9) | H9B···C13$^{v}$ | 3.0500 |
| C4···C7$^{iv}$ | 3.528 (8) | H9B···C12 | 2.8500 |
| C5···C9 | 3.574 (7) | H9B···H12 | 2.3300 |
| C6···C1$^{iv}$ | 3.509 (7) | H10A···H8A | 2.4100 |
| C7···O1 | 3.257 (10) | H10A···H16 | 2.3900 |
| C7···C3$^{iv}$ | 3.568 (9) | H10B···H25$^{ii}$ | 2.4300 |
| C7···C4$^{iv}$ | 3.528 (8) | H10B···H8B | 2.5500 |
| C8···C2$^{iv}$ | 3.597 (7) | H12···H9B | 2.3300 |

| | | | |
|---|---|---|---|
| C9···C5 | 3.574 (7) | H12···C25$^{vii}$ | 3.0700 |
| C17···O1 | 3.236 (13) | H12···C9 | 2.6900 |
| C1···H18B | 3.0900 | H13···Br1$^{v}$ | 3.1000 |
| C2···H17B | 2.9500 | H14···HW1$^{v}$ | 2.3300 |
| C5···H9A | 2.9900 | H15···H21$^{v}$ | 2.5400 |
| C5···H8A | 3.0600 | H16···H10A | 2.3900 |
| C6···H9A | 2.9700 | H17A···O1 | 2.3800 |
| C7···H18A | 3.0700 | H17A···H19A | 2.5500 |
| C8···H5 | 3.0200 | H17A···H7 | 2.5700 |
| C9···H12 | 2.6900 | H17B···C2 | 2.9500 |
| C11···H22$^{vi}$ | 3.0200 | H17B···H2 | 2.5600 |
| C11···H24$^{ii}$ | 2.9000 | H17B···Br1$^{x}$ | 3.1100 |
| C12···H9A | 2.9600 | H18A···C21 | 2.9400 |
| C12···H9B | 2.8500 | H18A···C7 | 3.0700 |
| C13···H9B$^{v}$ | 3.0500 | H18B···C1 | 3.0900 |
| C15···H24$^{ii}$ | 3.0600 | H19A···H17A | 2.5500 |
| C16···H24$^{ii}$ | 2.8100 | H19A···H21 | 2.5100 |
| C17···H2 | 3.0200 | H19B···H25 | 2.3800 |
| C18···H23$^{vii}$ | 3.0700 | H21···H15$^{v}$ | 2.5400 |
| C20···H5$^{viii}$ | 3.0900 | H21···H19A | 2.5100 |
| C21···H18A | 2.9400 | H22···C11$^{viii}$ | 3.0200 |
| C25···H12$^{ix}$ | 3.0700 | H23···C18$^{ix}$ | 3.0700 |
| HW1···H14$^{v}$ | 2.3300 | H24···C11$^{x}$ | 2.9000 |
| HW1···Br1$^{iii}$ | 3.04 (9) | H24···C16$^{x}$ | 2.8100 |
| H2···Br1$^{x}$ | 3.1600 | H24···C15$^{x}$ | 3.0600 |
| H2···H17B | 2.5600 | H25···H10B$^{x}$ | 2.4300 |
| H2···C17 | 3.0200 | H25···H19B | 2.3800 |
| HW1—O1—HW2 | 105 (9) | N2—C8—H8A | 109.00 |
| C1—N1—C7 | 107.9 (3) | C9—C8—H8A | 109.00 |
| C1—N1—C17 | 126.5 (4) | C9—C8—H8B | 109.00 |
| C7—N1—C17 | 125.6 (4) | N2—C8—H8B | 109.00 |
| C6—N2—C7 | 108.1 (4) | H8A—C8—H8B | 108.00 |
| C6—N2—C8 | 126.3 (4) | C8—C9—H9B | 109.00 |
| C7—N2—C8 | 125.6 (4) | C10—C9—H9A | 109.00 |
| N1—C1—C6 | 106.7 (4) | C10—C9—H9B | 109.00 |
| C2—C1—C6 | 121.8 (4) | H9A—C9—H9B | 108.00 |
| N1—C1—C2 | 131.5 (4) | C8—C9—H9A | 109.00 |
| C1—C2—C3 | 115.9 (4) | C9—C10—H10A | 108.00 |
| C2—C3—C4 | 122.1 (5) | C9—C10—H10B | 108.00 |
| C3—C4—C5 | 122.2 (5) | C11—C10—H10B | 108.00 |
| C4—C5—C6 | 115.9 (4) | H10A—C10—H10B | 107.00 |
| N2—C6—C1 | 106.2 (4) | C11—C10—H10A | 108.00 |
| C1—C6—C5 | 122.1 (4) | C11—C12—H12 | 120.00 |
| N2—C6—C5 | 131.8 (4) | C13—C12—H12 | 120.00 |
| N1—C7—N2 | 111.2 (5) | C12—C13—H13 | 120.00 |

| | | | |
|---|---|---|---|
| N2—C8—C9 | 111.1 (5) | C14—C13—H13 | 120.00 |
| C8—C9—C10 | 112.5 (6) | C15—C14—H14 | 120.00 |
| C9—C10—C11 | 117.0 (7) | C13—C14—H14 | 120.00 |
| C10—C11—C12 | 123.8 (6) | C16—C15—H15 | 120.00 |
| C12—C11—C16 | 117.7 (7) | C14—C15—H15 | 120.00 |
| C10—C11—C16 | 118.5 (8) | C11—C16—H16 | 120.00 |
| C11—C12—C13 | 120.6 (6) | C15—C16—H16 | 120.00 |
| C12—C13—C14 | 120.2 (9) | N1—C17—H17B | 110.00 |
| C13—C14—C15 | 120.8 (9) | C18—C17—H17A | 110.00 |
| C14—C15—C16 | 120.2 (8) | N1—C17—H17A | 110.00 |
| C11—C16—C15 | 120.5 (9) | H17A—C17—H17B | 108.00 |
| N1—C17—C18 | 108.6 (4) | C18—C17—H17B | 110.00 |
| C17—C18—C19 | 113.8 (5) | C17—C18—H18A | 109.00 |
| C18—C19—C20 | 110.4 (6) | C17—C18—H18B | 109.00 |
| C19—C20—C21 | 120.7 (8) | C19—C18—H18B | 109.00 |
| C19—C20—C25 | 121.4 (6) | H18A—C18—H18B | 108.00 |
| C21—C20—C25 | 117.9 (8) | C19—C18—H18A | 109.00 |
| C20—C21—C22 | 120.0 (10) | C18—C19—H19B | 110.00 |
| C21—C22—C23 | 120.8 (9) | C20—C19—H19A | 110.00 |
| C22—C23—C24 | 118.9 (12) | C20—C19—H19B | 110.00 |
| C23—C24—C25 | 121.2 (12) | H19A—C19—H19B | 108.00 |
| C20—C25—C24 | 120.9 (8) | C18—C19—H19A | 110.00 |
| C3—C2—H2 | 122.00 | C22—C21—H21 | 120.00 |
| C1—C2—H2 | 122.00 | C20—C21—H21 | 120.00 |
| C2—C3—H3 | 119.00 | C21—C22—H22 | 120.00 |
| C4—C3—H3 | 119.00 | C23—C22—H22 | 120.00 |
| C5—C4—H4 | 119.00 | C24—C23—H23 | 120.00 |
| C3—C4—H4 | 119.00 | C22—C23—H23 | 121.00 |
| C4—C5—H5 | 122.00 | C23—C24—H24 | 119.00 |
| C6—C5—H5 | 122.00 | C25—C24—H24 | 119.00 |
| N2—C7—H7 | 124.00 | C20—C25—H25 | 119.00 |
| N1—C7—H7 | 124.00 | C24—C25—H25 | 120.00 |
| C7—N1—C1—C2 | 178.3 (6) | C4—C5—C6—C1 | −0.6 (8) |
| C17—N1—C1—C2 | −3.8 (8) | N2—C8—C9—C10 | −177.5 (5) |
| C7—N1—C1—C6 | 0.2 (5) | C8—C9—C10—C11 | 173.4 (5) |
| C17—N1—C1—C6 | 178.1 (4) | C9—C10—C11—C12 | 24.9 (9) |
| C1—N1—C7—N2 | 0.3 (6) | C9—C10—C11—C16 | −157.8 (7) |
| C17—N1—C7—N2 | −177.7 (4) | C10—C11—C12—C13 | 174.1 (7) |
| C1—N1—C17—C18 | −85.3 (5) | C12—C11—C16—C15 | 3.1 (13) |
| C7—N1—C17—C18 | 92.3 (7) | C16—C11—C12—C13 | −3.2 (11) |
| C6—N2—C8—C9 | 72.9 (5) | C10—C11—C16—C15 | −174.4 (9) |
| C7—N2—C8—C9 | −107.4 (6) | C11—C12—C13—C14 | 1.0 (12) |
| C7—N2—C6—C5 | −177.9 (6) | C12—C13—C14—C15 | 1.4 (14) |
| C8—N2—C6—C5 | 1.8 (9) | C13—C14—C15—C16 | −1.5 (17) |
| C7—N2—C6—C1 | 0.6 (5) | C14—C15—C16—C11 | −0.8 (16) |
| C8—N2—C6—C1 | −179.7 (4) | N1—C17—C18—C19 | −175.7 (5) |
| C6—N2—C7—N1 | −0.6 (6) | C17—C18—C19—C20 | −179.6 (5) |
| C8—N2—C7—N1 | 179.7 (4) | C18—C19—C20—C21 | −81.0 (8) |
| N1—C1—C2—C3 | −177.6 (5) | C18—C19—C20—C25 | 97.9 (7) |

| | | | |
|---|---|---|---|
| C2—C1—C6—C5 | −0.1 (8) | C19—C20—C21—C22 | 173.4 (7) |
| N1—C1—C6—C5 | 178.3 (5) | C25—C20—C21—C22 | −5.5 (11) |
| C6—C1—C2—C3 | 0.4 (8) | C19—C20—C25—C24 | −177.4 (7) |
| C2—C1—C6—N2 | −178.8 (5) | C21—C20—C25—C24 | 1.5 (10) |
| N1—C1—C6—N2 | −0.5 (5) | C20—C21—C22—C23 | 4.6 (14) |
| C1—C2—C3—C4 | 0.1 (8) | C21—C22—C23—C24 | 0.6 (14) |
| C2—C3—C4—C5 | −0.9 (9) | C22—C23—C24—C25 | −4.8 (14) |
| C3—C4—C5—C6 | 1.1 (8) | C23—C24—C25—C20 | 3.8 (13) |
| C4—C5—C6—N2 | 177.8 (5) | | |

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+3/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) −*x*, *y*+1/2, −*z*+3/2; (iv) −*x*, −*y*, −*z*+2; (v) −*x*−1, −*y*, −*z*+1; (vi) *x*, *y*−1, *z*; (vii) −*x*−1, *y*−1/2, −*z*+3/2; (viii) *x*, *y*+1, *z*; (ix) −*x*−1, *y*+1/2, −*z*+3/2; (x) *x*, −*y*+1/2, *z*+1/2.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7···O1 | 0.93 | 2.50 | 3.257 (10) | 139 |
| C17—H17A···O1 | 0.97 | 2.38 | 3.236 (13) | 148 |
| C24—H24···Cg1$^{x}$ | 0.93 | 2.84 | 3.771 (14) | 176 |

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

**Fig. 1**

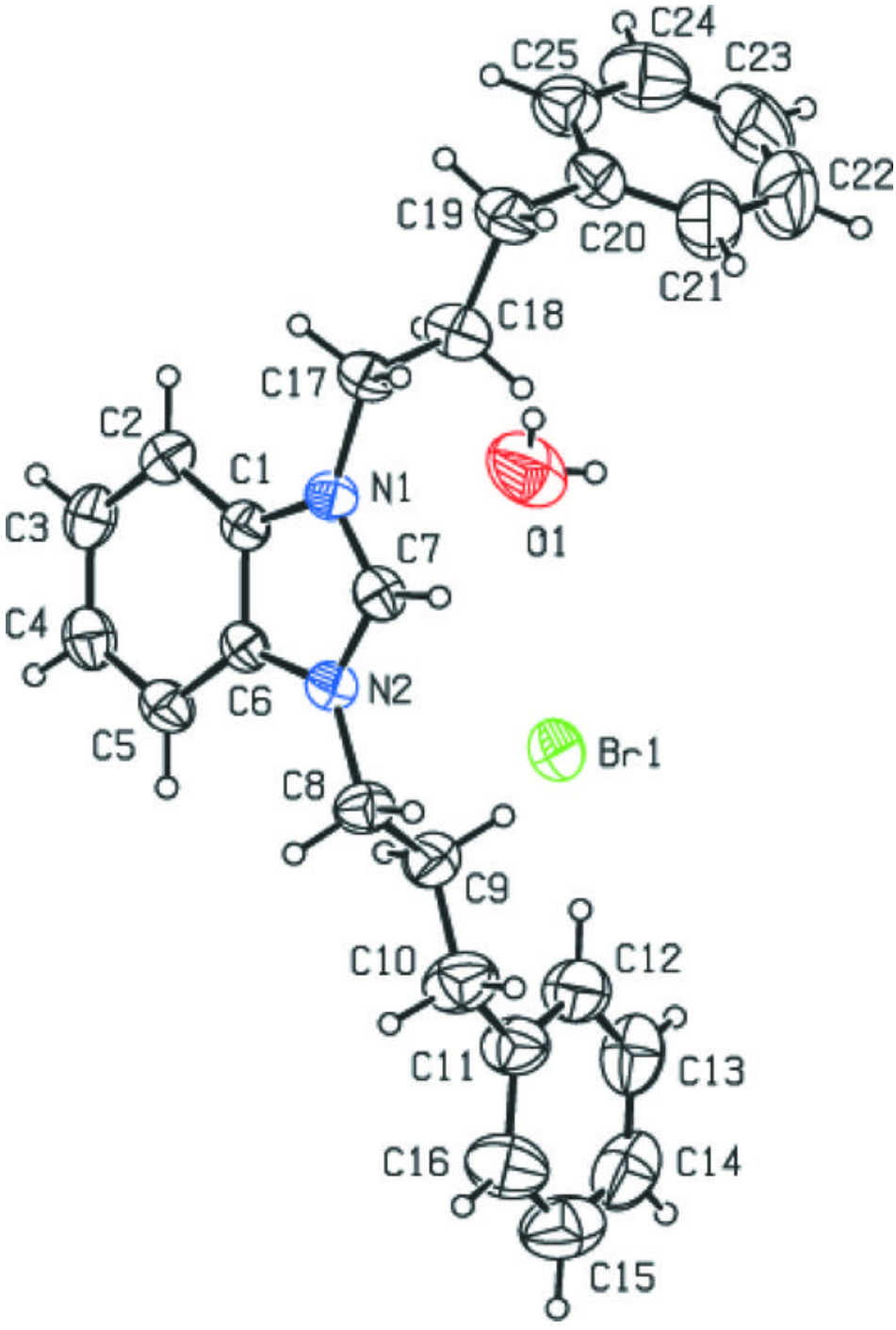
C24
C25
C23
C22
C19
C20
C21
C18
C17
C2
C1
N1
O1
C3
C7
C4
N2
C6
C5
Br1
C8
C9
C10
C12
C11
C13
C16
C14
C15

**Fig. 2**