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6-Bromo-2,4-bis(3-methoxyphenyl)-3,4-dihydro-2*H*-1,3-naphthoxazine

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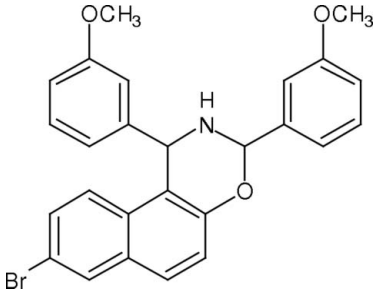
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.086; data-to-parameter ratio = 17.0.

The oxazine ring in the title compound, $C_{26}H_{22}BrNO_3$, adopts a half-chair conformation. The amino H atom is not involved in a classical hydrogen bond, but there is a weak N—H$\cdots\pi$ interaction to the centre of an aromatic ring.

Related literature

For related literature, see: Allingham *et al.* (1968); Armstrong *et al.* (1996); Baert *et al.* (1989); Bienayme *et al.* (2000); Chaudhuri *et al.* (2001); Domling & Ugi (2000); Huang & Sun (2005); Kappe (2000); Simon *et al.* (2004); Tailor & Hall (2000); Tietze (1996); Tietze & Rachkelmann (2004).



Experimental

Crystal data

$C_{26}H_{22}BrNO_3$
M_r = 476.36
Triclinic, $P\overline{1}$
a = 5.9753 (4) Å
b = 9.9177 (7) Å
c = 18.1925 (13) Å
α = 84.632 (6)°
β = 88.618 (6)°
γ = 81.088 (5)°
V = 1060.37 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.97 mm^{-1}
T = 173 (2) K
0.33 × 0.27 × 0.25 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.563, T_{max} = 0.639
23965 measured reflections
4875 independent reflections
4449 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.04
4875 reflections
287 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

*Cg*1 is the centroid of the C5/C6/C11–C14 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N8—H8$\cdots$*Cg*1^{i}	0.86 (2)	2.709 (s.u.?)	3.496 (s.u.?)	153.3 (s.u.?)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2044).

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supplementary materials

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6-Bromo-2,4-bis(3-methoxyphenyl)-3,4-dihydro-2*H*-1,3-naphthoxazine

B. K. Sarojini, B. Narayana, A. N. Mayekar, H. S. Yathirajan and M. Bolte

Comment

Domino (Tietze, 1996; Armstrong *et al.*, 1996) and multicomponent (Domling & Ugi, 2000; Kappe, 2000; Bienayme *et al.*, 2000; Simon *et al.*, 2004) reactions (MCRs) are powerful strategies in current organic synthesis. Consistently, several reported MCRs feature Diels-Alder chemistry with heterodiene (Tailor & Hall, 2000; Tietze & Rachkelmann, 2004;) building blocks for the synthesis of six-membered heterocyclic rings. In 1950's, oxazinic derivatives have drawn the attention because of their antitumoraly antituberculostatic activities and assigned a conformation with an axial N—*R* group when *R*= methyl and ethyl whereas a conformation with an equatorial N—*R* group when *R*= cyclohexyl and *tert*-butyl. The same series of compounds with n-propyl and iso-propyl derivatives have been studied by Allingham *et al.* (1968) using the 1*H*-NMR. Some related structures *viz*, (*Z*)-7-chloro-3-[(3-chlorophenyl)methylidene]-4-*p*-tosyl-3,4-dihydro-2*H*-1,4- benzoxazine (Chaudhuri *et al.*, 2001), 2-amino-4,6-diphenyl-4*H*-1,3-oxazinium trifluoroacetate (Huang & Sun, 2005) and spirocyclohexane oxazines, thiazines and selenazines (Baert *et al.*, 1989) have been reported. A new compound, (I), $C_{26}H_{22}BrNO_3$ was synthesized and its crystal structure is reported.

Geometric parameters in (I) are in the usual ranges. The amino H atom is not involved in a classical hydrogen bond but there is a weak N—H···π interaction to the centre of the ring composed of C5, C6, C11, C12, C13 and C14 [N—H 0.86 (2) Å, N···cog 2.709 Å, N···cog 3.496 Å, N—H···cog 153.3°; symmetry operator $x-1,y,z$].

Experimental

To 6-bromo-2-naphthol (2.23 g, 0.01 mol) in methanol (10 ml) was added *m*-anisaldehyde (2.7 g, 0.02 mol) and 10 ml of 30% methanolic ammonia and stirred at room temperature for 48 h. The solid separated was collected by filtration and crystallized in ethyl acetate and good quality crystals were obtained by evaporation of the solution prepared in acetone (m.p:361–363 K).

Refinement

All H atoms were found in a difference map, but those bonded to C were geometrically positioned at C—H = 0.95, 0.98 and 1.00 ° for the aromatic, methyl and tertiary H-atoms, respectively, with fixed individual displacement parameters [U(H) = 1.2 $U_{eq}(C_{non\text{-}methyl})$ or U(H) = 1.5 $U_{eq}(C_{methyl})$] during the refinements; the methyl groups were allowed to rotate but not to tip. The amino H atom was allowed to refine freely.

Figures

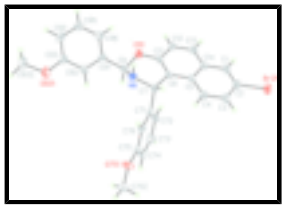


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

6-Bromo-2,4-bis(3-methoxyphenyl)-3,4-dihydro-2*H*-1,3-naphthoxazine

Crystal data

$C_{26}H_{22}BrNO_3$	$Z = 2$
$M_r = 476.36$	$F_{000} = 488$
Triclinic, $P\overline{1}$	$D_x = 1.492$ Mg m^{-3}
$a = 5.9753$ (4) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.9177$ (7) Å	Cell parameters from 29479 reflections
$c = 18.1925$ (13) Å	$\theta = 3.5$–27.9º
$\alpha = 84.632$ (6)º	$\mu = 1.97$ mm^{-1}
$\beta = 88.618$ (6)º	$T = 173$ (2) K
$\gamma = 81.088$ (5)º	Block, colourless
$V = 1060.37$ (13) Å^3	0.33 × 0.27 × 0.25 mm

Data collection

Stoe IPDSII two-circle diffractometer	4875 independent reflections
Radiation source: fine-focus sealed tube	4449 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.044$
$T = 173$(2) K	$\theta_{max} = 27.6$º
ω scans	$\theta_{min} = 3.5$º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$h = -7 \rightarrow 7$
$T_{min} = 0.563$, $T_{max} = 0.639$	$k = -12 \rightarrow 12$
23965 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.321P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.086$	$(\Delta/\sigma)_{max} = 0.001$

S = 1.04	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
4875 reflections	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
287 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0214 (18)
Secondary atom site location: difference Fourier map	

Special details

Experimental. Analysis found: C, H, N %; 65.48, 4.61, 2.90% for $C_{26}H_{22}BrNO_3$; required: C 65.55, H 4.66, N 2.94%.

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness 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 threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *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}
C1	1.2244 (3)	1.10888 (19)	0.05325 (9)	0.0293 (3)
H1	1.3472	1.0830	0.0208	0.035*
C2	1.1620 (3)	1.24339 (19)	0.06485 (9)	0.0291 (3)
Br21	1.31702 (3)	1.37957 (2)	0.014156 (11)	0.04071 (9)
C3	0.9805 (3)	1.28612 (19)	0.11293 (10)	0.0309 (3)
H3	0.9389	1.3803	0.1202	0.037*
C4	0.8654 (3)	1.18928 (18)	0.14888 (9)	0.0279 (3)
H4	0.7446	1.2177	0.1816	0.034*
C5	0.9223 (3)	1.04756 (17)	0.13832 (8)	0.0228 (3)
C6	0.8016 (3)	0.94569 (16)	0.17455 (8)	0.0225 (3)
C7	0.5995 (3)	0.98412 (16)	0.22469 (8)	0.0212 (3)
H7	0.5004	1.0640	0.1987	0.032*
C71	0.6622 (3)	1.02780 (15)	0.29929 (8)	0.0207 (3)
C72	0.8827 (3)	0.99958 (18)	0.32690 (9)	0.0269 (3)
H72	1.0019	0.9528	0.2991	0.032*
C73	0.9255 (3)	1.0409 (2)	0.39569 (10)	0.0321 (4)
H73	1.0754	1.0223	0.4143	0.038*
C74	0.7538 (3)	1.10898 (18)	0.43773 (9)	0.0295 (3)
H74	0.7860	1.1365	0.4845	0.035*
C75	0.5339 (3)	1.13628 (17)	0.41039 (9)	0.0263 (3)
O751	0.3513 (2)	1.20077 (15)	0.44689 (7)	0.0371 (3)
C752	0.3873 (4)	1.2390 (2)	0.51912 (11)	0.0410 (4)
H75A	0.4524	1.1577	0.5508	0.061*
H75B	0.2424	1.2791	0.5402	0.061*

H75C	0.4918	1.3064	0.5158	0.061*
C76	0.4895 (3)	1.09611 (16)	0.34104 (9)	0.0236 (3)
H76	0.3398	1.1157	0.3223	0.028*
N8	0.4640 (2)	0.87111 (14)	0.23740 (8)	0.0231 (3)
H8	0.383 (4)	0.870 (2)	0.1992 (12)	0.026 (5)*
C9	0.6035 (3)	0.74024 (17)	0.25247 (9)	0.0240 (3)
H9	0.6894	0.7416	0.2988	0.036*
C91	0.4583 (3)	0.62705 (16)	0.26350 (9)	0.0246 (3)
C92	0.2967 (3)	0.63350 (17)	0.32011 (9)	0.0280 (3)
H92	0.2815	0.7069	0.3508	0.034*
C93	0.1567 (3)	0.53274 (18)	0.33211 (10)	0.0297 (3)
O931	0.0050 (3)	0.54830 (14)	0.38976 (9)	0.0454 (4)
C932	−0.1263 (4)	0.4403 (2)	0.40788 (14)	0.0440 (5)
H93A	−0.2307	0.4373	0.3676	0.066*
H93B	−0.2129	0.4571	0.4534	0.066*
H93C	−0.0249	0.3525	0.4151	0.066*
C94	0.1746 (3)	0.42578 (18)	0.28676 (11)	0.0324 (4)
H94	0.0785	0.3577	0.2942	0.039*
C95	0.3360 (3)	0.42067 (19)	0.23030 (11)	0.0351 (4)
H95	0.3492	0.3481	0.1991	0.042*
C96	0.4787 (3)	0.51908 (19)	0.21830 (10)	0.0316 (4)
H96	0.5890	0.5130	0.1798	0.038*
O10	0.7651 (2)	0.70462 (12)	0.19325 (7)	0.0286 (3)
C11	0.8681 (3)	0.80986 (17)	0.16211 (9)	0.0237 (3)
C12	1.0515 (3)	0.76955 (18)	0.11308 (9)	0.0272 (3)
H12	1.0938	0.6757	0.1051	0.033*
C13	1.1661 (3)	0.86601 (19)	0.07776 (9)	0.0283 (3)
H13	1.2871	0.8386	0.0449	0.034*
C14	1.1069 (3)	1.00730 (17)	0.08938 (8)	0.0248 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0260 (8)	0.0370 (9)	0.0253 (8)	−0.0079 (7)	0.0056 (6)	−0.0017 (6)
C2	0.0277 (8)	0.0333 (9)	0.0274 (8)	−0.0110 (7)	0.0030 (6)	0.0013 (6)
Br21	0.04119 (13)	0.03886 (12)	0.04419 (13)	−0.01807 (8)	0.01209 (8)	0.00198 (8)
C3	0.0316 (8)	0.0278 (8)	0.0339 (9)	−0.0083 (7)	0.0051 (7)	−0.0012 (7)
C4	0.0262 (8)	0.0285 (8)	0.0291 (8)	−0.0046 (6)	0.0066 (6)	−0.0033 (6)
C5	0.0221 (7)	0.0266 (8)	0.0197 (7)	−0.0044 (6)	0.0012 (5)	−0.0010 (6)
C6	0.0218 (7)	0.0265 (8)	0.0193 (7)	−0.0043 (6)	0.0020 (5)	−0.0021 (6)
C7	0.0204 (7)	0.0218 (7)	0.0214 (7)	−0.0038 (6)	0.0016 (5)	−0.0019 (5)
C71	0.0220 (7)	0.0197 (7)	0.0208 (7)	−0.0054 (6)	0.0017 (5)	−0.0008 (5)
C72	0.0212 (7)	0.0331 (8)	0.0260 (8)	−0.0041 (6)	0.0014 (6)	−0.0010 (6)
C73	0.0254 (8)	0.0437 (10)	0.0282 (8)	−0.0099 (7)	−0.0043 (6)	−0.0002 (7)
C74	0.0367 (9)	0.0326 (9)	0.0220 (7)	−0.0138 (7)	−0.0020 (6)	−0.0019 (6)
C75	0.0319 (8)	0.0221 (7)	0.0253 (7)	−0.0053 (6)	0.0044 (6)	−0.0029 (6)
O751	0.0401 (7)	0.0416 (7)	0.0288 (6)	0.0012 (6)	0.0067 (5)	−0.0140 (5)
C752	0.0582 (13)	0.0375 (10)	0.0297 (9)	−0.0112 (9)	0.0113 (8)	−0.0131 (8)

C76	0.0225 (7)	0.0250 (7)	0.0236 (7)	−0.0041 (6)	0.0010 (6)	−0.0024 (6)
N8	0.0211 (6)	0.0233 (6)	0.0257 (6)	−0.0052 (5)	−0.0006 (5)	−0.0038 (5)
C9	0.0232 (7)	0.0247 (7)	0.0240 (7)	−0.0039 (6)	0.0020 (6)	−0.0015 (6)
C91	0.0255 (7)	0.0227 (7)	0.0255 (7)	−0.0037 (6)	0.0002 (6)	−0.0009 (6)
C92	0.0317 (8)	0.0236 (8)	0.0297 (8)	−0.0065 (7)	0.0058 (6)	−0.0050 (6)
C93	0.0289 (8)	0.0237 (8)	0.0360 (9)	−0.0038 (6)	0.0071 (7)	−0.0013 (6)
O931	0.0507 (9)	0.0302 (7)	0.0576 (9)	−0.0153 (6)	0.0303 (7)	−0.0090 (6)
C932	0.0391 (10)	0.0299 (9)	0.0623 (13)	−0.0095 (8)	0.0189 (9)	0.0016 (9)
C94	0.0310 (9)	0.0233 (8)	0.0444 (10)	−0.0081 (7)	0.0030 (7)	−0.0045 (7)
C95	0.0407 (10)	0.0269 (8)	0.0403 (10)	−0.0089 (7)	0.0043 (8)	−0.0115 (7)
C96	0.0339 (9)	0.0290 (8)	0.0327 (8)	−0.0060 (7)	0.0067 (7)	−0.0068 (7)
O10	0.0282 (6)	0.0218 (5)	0.0351 (6)	−0.0037 (5)	0.0097 (5)	−0.0027 (5)
C11	0.0224 (7)	0.0265 (8)	0.0223 (7)	−0.0047 (6)	0.0015 (6)	−0.0009 (6)
C12	0.0257 (8)	0.0279 (8)	0.0277 (8)	−0.0010 (6)	0.0023 (6)	−0.0071 (6)
C13	0.0262 (8)	0.0345 (9)	0.0243 (7)	−0.0035 (7)	0.0051 (6)	−0.0059 (6)
C14	0.0235 (7)	0.0306 (8)	0.0206 (7)	−0.0057 (6)	0.0017 (6)	−0.0020 (6)

Geometric parameters (Å, °)

C1—C2	1.363 (3)	C752—H75C	0.9800
C1—C14	1.419 (2)	C76—H76	0.9500
C1—H1	0.9500	N8—C9	1.437 (2)
C2—C3	1.415 (2)	N8—H8	0.86 (2)
C2—Br21	1.9093 (17)	C9—O10	1.4603 (19)
C3—C4	1.375 (2)	C9—C91	1.518 (2)
C3—H3	0.9500	C9—H9	1.0000
C4—C5	1.424 (2)	C91—C92	1.394 (2)
C4—H4	0.9500	C91—C96	1.399 (2)
C5—C14	1.432 (2)	C92—C93	1.399 (2)
C5—C6	1.433 (2)	C92—H92	0.9500
C6—C11	1.382 (2)	C93—O931	1.373 (2)
C6—C7	1.518 (2)	C93—C94	1.394 (2)
C7—N8	1.4796 (19)	O931—C932	1.433 (2)
C7—C71	1.534 (2)	C932—H93A	0.9800
C7—H7	1.0000	C932—H93B	0.9800
C71—C76	1.394 (2)	C932—H93C	0.9800
C71—C72	1.400 (2)	C94—C95	1.391 (3)
C72—C73	1.394 (2)	C94—H94	0.9500
C72—H72	0.9500	C95—C96	1.392 (3)
C73—C74	1.392 (3)	C95—H95	0.9500
C73—H73	0.9500	C96—H96	0.9500
C74—C75	1.394 (2)	O10—C11	1.3644 (19)
C74—H74	0.9500	C11—C12	1.428 (2)
C75—O751	1.367 (2)	C12—C13	1.365 (2)
C75—C76	1.401 (2)	C12—H12	0.9500
O751—C752	1.431 (2)	C13—C14	1.425 (2)
C752—H75A	0.9800	C13—H13	0.9500
C752—H75B	0.9800		
C2—C1—C14	120.37 (16)	C75—C76—H76	119.6

C2—C1—H1	119.8	C9—N8—C7	112.20 (12)
C14—C1—H1	119.8	C9—N8—H8	109.6 (14)
C1—C2—C3	121.56 (16)	C7—N8—H8	109.1 (14)
C1—C2—Br21	119.97 (13)	N8—C9—O10	113.44 (13)
C3—C2—Br21	118.45 (13)	N8—C9—C91	110.47 (13)
C4—C3—C2	119.02 (16)	O10—C9—C91	106.88 (13)
C4—C3—H3	120.5	N8—C9—H9	108.6
C2—C3—H3	120.5	O10—C9—H9	108.6
C3—C4—C5	121.73 (15)	C91—C9—H9	108.6
C3—C4—H4	119.1	C92—C91—C96	119.41 (16)
C5—C4—H4	119.1	C92—C91—C9	118.17 (14)
C4—C5—C14	118.02 (15)	C96—C91—C9	122.40 (15)
C4—C5—C6	122.37 (14)	C91—C92—C93	120.51 (15)
C14—C5—C6	119.62 (15)	C91—C92—H92	119.7
C11—C6—C5	119.06 (14)	C93—C92—H92	119.7
C11—C6—C7	119.46 (14)	O931—C93—C94	123.98 (16)
C5—C6—C7	121.47 (14)	O931—C93—C92	115.70 (15)
N8—C7—C6	111.18 (13)	C94—C93—C92	120.32 (16)
N8—C7—C71	109.34 (12)	C93—O931—C932	117.07 (15)
C6—C7—C71	113.95 (12)	O931—C932—H93A	109.5
N8—C7—H7	107.4	O931—C932—H93B	109.5
C6—C7—H7	107.4	H93A—C932—H93B	109.5
C71—C7—H7	107.4	O931—C932—H93C	109.5
C76—C71—C72	119.53 (14)	H93A—C932—H93C	109.5
C76—C71—C7	117.80 (13)	H93B—C932—H93C	109.5
C72—C71—C7	122.66 (14)	C95—C94—C93	118.64 (16)
C73—C72—C71	119.22 (15)	C95—C94—H94	120.7
C73—C72—H72	120.4	C93—C94—H94	120.7
C71—C72—H72	120.4	C94—C95—C96	121.71 (16)
C74—C73—C72	121.55 (15)	C94—C95—H95	119.1
C74—C73—H73	119.2	C96—C95—H95	119.1
C72—C73—H73	119.2	C95—C96—C91	119.39 (16)
C73—C74—C75	119.18 (15)	C95—C96—H96	120.3
C73—C74—H74	120.4	C91—C96—H96	120.3
C75—C74—H74	120.4	C11—O10—C9	115.03 (12)
O751—C75—C74	124.50 (15)	O10—C11—C6	124.07 (14)
O751—C75—C76	115.79 (15)	O10—C11—C12	114.49 (14)
C74—C75—C76	119.71 (15)	C6—C11—C12	121.42 (15)
C75—O751—C752	117.67 (16)	C13—C12—C11	119.94 (16)
O751—C752—H75A	109.5	C13—C12—H12	120.0
O751—C752—H75B	109.5	C11—C12—H12	120.0
H75A—C752—H75B	109.5	C12—C13—C14	121.01 (15)
O751—C752—H75C	109.5	C12—C13—H13	119.5
H75A—C752—H75C	109.5	C14—C13—H13	119.5
H75B—C752—H75C	109.5	C1—C14—C13	121.75 (15)
C71—C76—C75	120.80 (15)	C1—C14—C5	119.30 (15)
C71—C76—H76	119.6	C13—C14—C5	118.94 (15)
C14—C1—C2—C3	0.2 (3)	N8—C9—C91—C92	−59.34 (19)
C14—C1—C2—Br21	−178.56 (12)	O10—C9—C91—C92	176.81 (14)

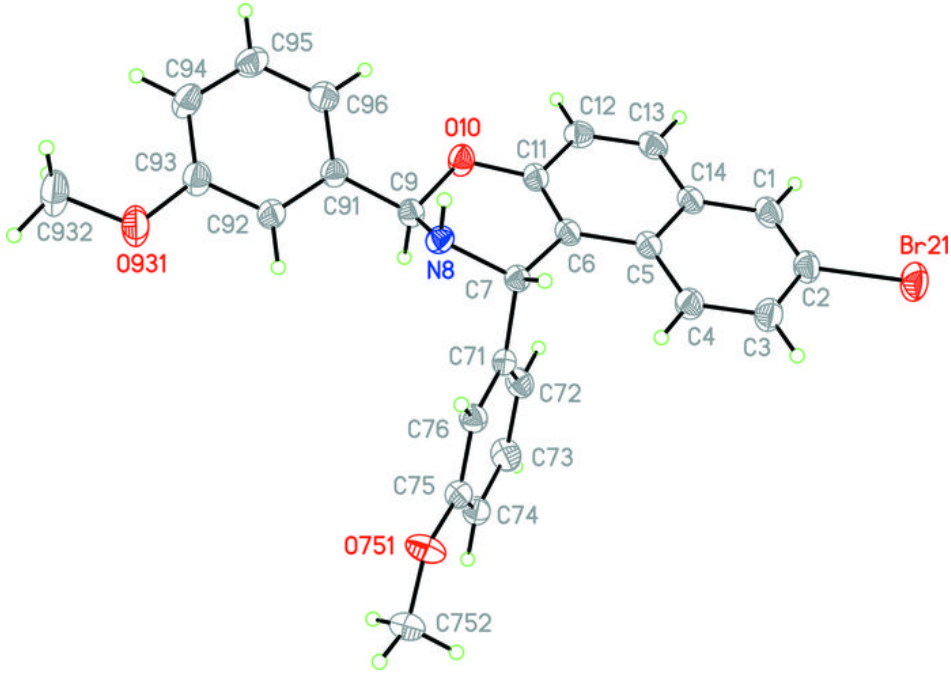
C1—C2—C3—C4	0.2 (3)	N8—C9—C91—C96	119.51 (17)
Br21—C2—C3—C4	179.02 (13)	O10—C9—C91—C96	−4.3 (2)
C2—C3—C4—C5	−0.8 (3)	C96—C91—C92—C93	0.4 (3)
C3—C4—C5—C14	0.8 (2)	C9—C91—C92—C93	179.25 (15)
C3—C4—C5—C6	−179.05 (16)	C91—C92—C93—O931	179.39 (16)
C4—C5—C6—C11	−179.36 (15)	C91—C92—C93—C94	−1.1 (3)
C14—C5—C6—C11	0.8 (2)	C94—C93—O931—C932	6.1 (3)
C4—C5—C6—C7	2.1 (2)	C92—C93—O931—C932	−174.35 (18)
C14—C5—C6—C7	−177.75 (14)	O931—C93—C94—C95	−179.65 (19)
C11—C6—C7—N8	−15.49 (19)	C92—C93—C94—C95	0.9 (3)
C5—C6—C7—N8	163.04 (13)	C93—C94—C95—C96	0.1 (3)
C11—C6—C7—C71	108.62 (16)	C94—C95—C96—C91	−0.7 (3)
C5—C6—C7—C71	−72.85 (18)	C92—C91—C96—C95	0.5 (3)
N8—C7—C71—C76	−70.34 (17)	C9—C91—C96—C95	−178.31 (16)
C6—C7—C71—C76	164.57 (14)	N8—C9—O10—C11	41.16 (18)
N8—C7—C71—C72	108.51 (16)	C91—C9—O10—C11	163.16 (13)
C6—C7—C71—C72	−16.6 (2)	C9—O10—C11—C6	−11.2 (2)
C76—C71—C72—C73	−0.2 (2)	C9—O10—C11—C12	170.21 (13)
C7—C71—C72—C73	−179.07 (15)	C5—C6—C11—O10	−179.62 (14)
C71—C72—C73—C74	0.4 (3)	C7—C6—C11—O10	−1.1 (2)
C72—C73—C74—C75	0.0 (3)	C5—C6—C11—C12	−1.1 (2)
C73—C74—C75—O751	179.20 (16)	C7—C6—C11—C12	177.48 (14)
C73—C74—C75—C76	−0.6 (3)	O10—C11—C12—C13	179.12 (14)
C74—C75—O751—C752	−2.0 (3)	C6—C11—C12—C13	0.5 (2)
C76—C75—O751—C752	177.72 (15)	C11—C12—C13—C14	0.5 (2)
C72—C71—C76—C75	−0.3 (2)	C2—C1—C14—C13	179.23 (16)
C7—C71—C76—C75	178.61 (14)	C2—C1—C14—C5	−0.1 (2)
O751—C75—C76—C71	−179.09 (15)	C12—C13—C14—C1	179.86 (16)
C74—C75—C76—C71	0.7 (2)	C12—C13—C14—C5	−0.8 (2)
C6—C7—N8—C9	44.30 (17)	C4—C5—C14—C1	−0.3 (2)
C71—C7—N8—C9	−82.39 (15)	C6—C5—C14—C1	179.50 (14)
C7—N8—C9—O10	−58.80 (17)	C4—C5—C14—C13	−179.74 (15)
C7—N8—C9—C91	−178.77 (12)	C6—C5—C14—C13	0.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8···Cg1^{i}	0.86 (2)	2.709(s.u.?)	3.496(s.u.?)	153.3(s.u.?)

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1


C95
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C7
C6
C5
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Br21
C4
C3
C71
C72
C76
C73
C75
C74
0751
C752