Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(3-Bromophenyl)-3,4,5-trimethoxybenzamide

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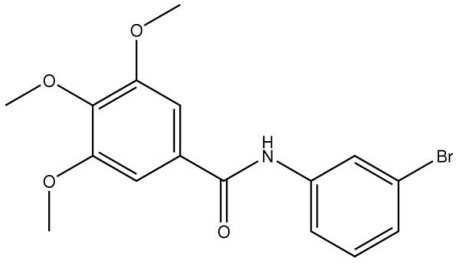
Received 21 May 2009; accepted 29 May 2009

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.033; wR factor = 0.076; data-to-parameter ratio = 14.6.

In the title compound, $C_{16}H_{16}BrNO_4$, the dihedral between the planes of the aromatic rings is 7.74 (18)°. The amide group is tilted with respect to the bromo- and methoxy-substituted aromatic rings by 36.3 (8) and 35.2 (8)°, respectively. The *meta*-methoxy groups are essentially in-plane with the aromatic ring [dihedral angles $CH_3-O-C-C$ = −4.6 (4) and −2.5 (4)°]. The *para*-methoxy group is markedly displaced from the ring plane [dihedral angle $CH_3-O-C-C$ = −72.5 (4)°]. The crystal packing is stabilized by N—H···O hydrogen bonds linking the molecules into chains running along the *b* axis.

Related literature

For related structures and general background, see: Saeed *et al.* (2009). For conformations of aromatic methoxy groups, see: Vande Velde *et al.* (2006).



Experimental

Crystal data

$C_{16}H_{16}BrNO_4$ M_r = 366.21
Orthorhombic, $Pna2_1$
a = 13.3085 (8) Å
b = 4.9953 (3) Å
c = 23.4061 (12) Å
V = 1556.04 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.66 mm^{-1}
T = 173 K
0.37 × 0.34 × 0.19 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan [*MULABS* (Spek, 2009; Blessing, 1995)]
T_{min} = 0.418, T_{max} = 0.600
11994 measured reflections
3028 independent reflections
2748 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.076
S = 0.99
3028 reflections
207 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983), with 1405 Friedel pairs
Flack parameter: 0.001 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.85 (4)	2.06 (4)	2.821 (3)	149 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

AI gratefully acknowledges a research scholarship from the HEC, Islamabad, Pakistan, under the HEC Indigenous PhD Scholarship 5000 Scheme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2213).

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supporting information

Acta Cryst. (2009). E**65**, o1470 [doi:10.1107/S1600536809020509]

N-(3-Bromophenyl)-3,4,5-trimethoxybenzamide

Aamer Saeed, Shahid Hussain, Aliya Ibrar and Michael Bolte

S1. Comment

The background to this study has been described in an earlier paper on *N*-(2-chlorophenyl)-4-chlorobenzamide (Saeed *et al.*, 2009). As part of our work on the structure of benzanilides and related compounds, we report here the structure of the title compound, Fig. 1.

In the title compound, $C_{16}H_{16}BrNO_4$, the dihedral angle between the aromatic rings is 7.74 (18)°. The amide moiety is tilted against the bromo and methoxy substituted aromatic rings by 36.3 (8)° and 35.2 (8)°, respectively. The *meta* methoxy groups are essentially in plane with the aromatic ring [dihedral angles CH_3—O—C—C = -4.6 (4)° and -2.5 (4)°], the methoxy group in *para* position is markedly displaced from the ring plane [dihedral angle CH_3—O—C—C = -72.5 (4)°]. This can be attributed to a combination of resonance effects, which lead for aromatic methoxy groups to being coplanar with an aromatic ring, and steric interactions, which prohibit a coplanar arrangement when more than two methoxy groups are present per benzene moiety (Vande Velde *et al.*, 2006). The crystal packing is stabilized by N—H···O hydrogen bonds linking the molecules to chains running along the *b* axis (Fig. 2).

S2. Experimental

3,4,5-Trimethoxybenzoyl chloride (5.4 mmol) in $CHCl_3$ was treated with 3-bromoaniline (21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with $CHCl_3$ and washed consecutively with aq 1 *M* HCl and saturated aq $NaHCO_3$. The organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Crystallization of the residue in $CHCl_3$ afforded the title compound (81%) as colourless needles. Anal. calcd. for $C_{16}H_{16}BrNO_4$: C, 52.48; H, 4.40; N, 3.82%; found: C, 52.51; H, 4.36; N, 3.87

S3. Refinement

H atoms were located in a difference map but those bonded to C were geometrically positioned and refined using a riding model with fixed individual displacement parameters [$U(H_{iso}) = 1.2U_{eq}(C)$ or $U(H_{iso}) = 1.5U_{eq}(C_{methyl})$] using a riding model with $C_{aromatic}$—H = 0.95 Å or C_{methyl}—H = 0.98 Å. The H atom bonded to N was freely refined.

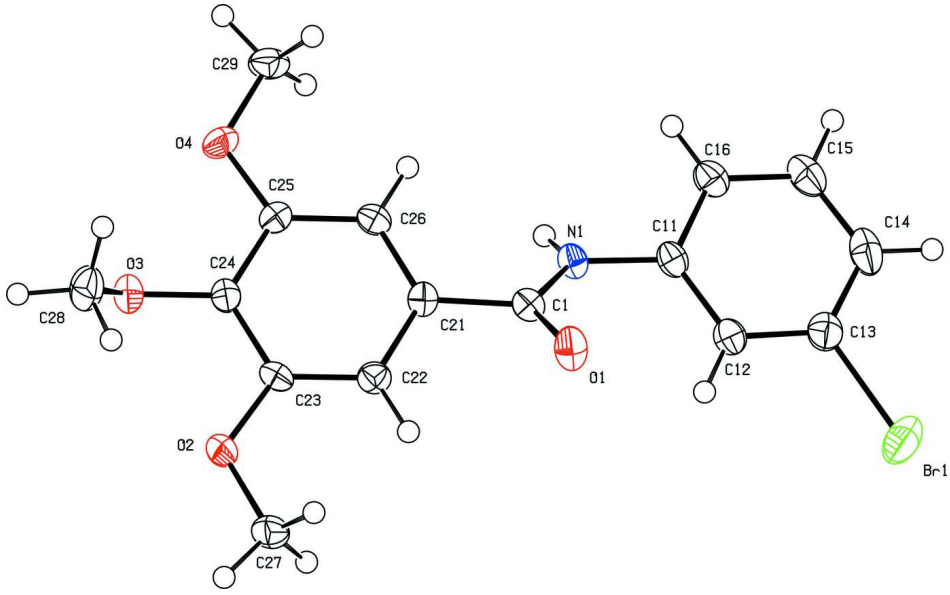

Figure 1

The title compound with the atom numbering scheme; displacement ellipsoids are at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii.

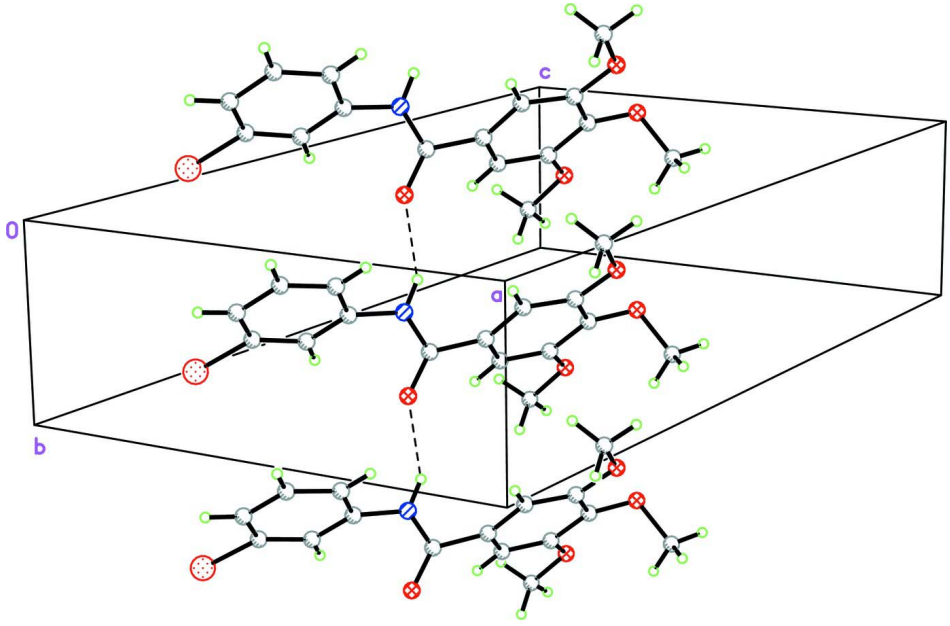

Figure 2

Partial packing diagram of the title compound. Hydrogen bonds are drawn as dashed lines.

N-(3-Bromophenyl)-3,4,5-trimethoxybenzamide

Crystal data

$C_{16}H_{16}BrNO_4$
$M_r = 366.21$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n

$a = 13.3085\ (8)$ Å
$b = 4.9953\ (3)$ Å
$c = 23.4061\ (12)$ Å
$V = 1556.04\ (15)$ Å^3

$Z = 4$
$F(000) = 744$
$D_x = 1.563$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11796 reflections

$\theta = 3.5$–$26.7°$
$\mu = 2.66$ mm^{-1}
$T = 173$ K
Plate, colourless
0.37 × 0.34 × 0.19 mm

Data collection

Stoe IPDS II two-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan [*MULABS* (Spek, 2009; Blessing, 1995)]
$T_{min} = 0.418$, $T_{max} = 0.600$

11994 measured reflections
3028 independent reflections
2748 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 26.3°$, $\theta_{min} = 3.5°$
$h = -16 \rightarrow 16$
$k = -6 \rightarrow 6$
$l = -29 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.076$
$S = 0.99$
3028 reflections
207 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0168 (10)
Absolute structure: Flack (1983), with 1405 Friedel pairs
Absolute structure parameter: 0.001 (8)

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.

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.19897 (2)	0.79946 (7)	0.12371 (2)	0.04356 (13)
N1	0.52764 (18)	0.5080 (5)	0.23414 (11)	0.0203 (5)
H1	0.546 (3)	0.351 (8)	0.2441 (17)	0.023 (9)*
O1	0.51118 (18)	0.9515 (4)	0.25246 (10)	0.0285 (5)
O2	0.62891 (16)	0.9862 (4)	0.46087 (9)	0.0276 (4)
O3	0.78413 (15)	0.6364 (4)	0.46308 (10)	0.0259 (4)
O4	0.82971 (16)	0.3369 (4)	0.37342 (10)	0.0276 (5)
C1	0.5457 (2)	0.7303 (5)	0.26493 (13)	0.0204 (6)

C11	0.4666 (2)	0.4924 (5)	0.18432 (13)	0.0220 (6)
C12	0.3799 (2)	0.6447 (6)	0.17818 (13)	0.0246 (6)
H12	0.3613	0.7724	0.2063	0.030*
C13	0.3216 (2)	0.6057 (6)	0.13014 (16)	0.0279 (6)
C14	0.3467 (3)	0.4244 (7)	0.08727 (14)	0.0334 (7)
H14	0.3060	0.4043	0.0542	0.040*
C15	0.4334 (3)	0.2740 (7)	0.09471 (15)	0.0332 (7)
H15	0.4520	0.1466	0.0665	0.040*
C16	0.4931 (3)	0.3066 (6)	0.14241 (13)	0.0270 (6)
H16	0.5523	0.2022	0.1467	0.032*
C21	0.6099 (2)	0.6961 (5)	0.31655 (12)	0.0201 (5)
C22	0.5883 (2)	0.8619 (5)	0.36327 (13)	0.0220 (6)
H22	0.5343	0.9859	0.3614	0.026*
C23	0.6460 (2)	0.8435 (5)	0.41199 (12)	0.0206 (6)
C24	0.7280 (2)	0.6655 (5)	0.41433 (13)	0.0210 (6)
C25	0.7487 (2)	0.5016 (5)	0.36721 (13)	0.0201 (5)
C26	0.6890 (2)	0.5141 (5)	0.31853 (13)	0.0213 (6)
H26	0.7021	0.3997	0.2870	0.026*
C27	0.5500 (2)	1.1804 (6)	0.45934 (14)	0.0279 (6)
H27A	0.5610	1.3033	0.4273	0.042*
H27B	0.5495	1.2817	0.4952	0.042*
H27C	0.4853	1.0895	0.4545	0.042*
C28	0.8483 (3)	0.8582 (7)	0.47549 (15)	0.0337 (7)
H28A	0.9001	0.8727	0.4459	0.051*
H28B	0.8805	0.8308	0.5127	0.051*
H28C	0.8086	1.0232	0.4764	0.051*
C29	0.8502 (2)	0.1607 (6)	0.32691 (15)	0.0287 (7)
H29A	0.7910	0.0496	0.3194	0.043*
H29B	0.9074	0.0456	0.3366	0.043*
H29C	0.8664	0.2659	0.2928	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02770 (15)	0.0536 (2)	0.0494 (2)	0.00300 (13)	−0.0092 (2)	0.0107 (3)
N1	0.0287 (12)	0.0138 (10)	0.0184 (12)	−0.0002 (9)	−0.0028 (10)	−0.0013 (9)
O1	0.0429 (12)	0.0152 (9)	0.0273 (11)	0.0026 (8)	−0.0104 (10)	−0.0011 (8)
O2	0.0303 (11)	0.0332 (10)	0.0192 (10)	0.0084 (8)	−0.0025 (9)	−0.0069 (9)
O3	0.0274 (10)	0.0289 (10)	0.0214 (11)	−0.0008 (8)	−0.0053 (8)	0.0023 (8)
O4	0.0232 (10)	0.0286 (11)	0.0311 (12)	0.0097 (8)	−0.0021 (9)	−0.0039 (9)
C1	0.0237 (13)	0.0175 (12)	0.0199 (14)	−0.0026 (9)	0.0012 (11)	0.0009 (10)
C11	0.0280 (14)	0.0206 (11)	0.0176 (14)	−0.0031 (11)	0.0002 (11)	0.0026 (11)
C12	0.0277 (15)	0.0248 (13)	0.0212 (14)	−0.0013 (11)	−0.0001 (12)	0.0019 (11)
C13	0.0250 (12)	0.0337 (13)	0.0251 (18)	−0.0052 (10)	0.0008 (13)	0.0053 (14)
C14	0.0352 (17)	0.0428 (17)	0.0223 (16)	−0.0146 (14)	−0.0052 (13)	0.0034 (14)
C15	0.0402 (19)	0.0357 (17)	0.0235 (17)	−0.0072 (14)	0.0011 (14)	−0.0058 (13)
C16	0.0326 (16)	0.0253 (14)	0.0232 (16)	−0.0008 (12)	0.0003 (12)	−0.0039 (11)
C21	0.0225 (14)	0.0169 (11)	0.0208 (14)	−0.0018 (10)	−0.0025 (10)	0.0014 (11)

C22	0.0244 (14)	0.0184 (12)	0.0232 (15)	0.0018 (10)	0.0007 (12)	−0.0005 (10)
C23	0.0242 (14)	0.0197 (13)	0.0178 (14)	−0.0015 (10)	0.0040 (11)	−0.0035 (11)
C24	0.0215 (13)	0.0229 (13)	0.0186 (14)	−0.0018 (10)	−0.0016 (11)	0.0009 (11)
C25	0.0181 (12)	0.0185 (13)	0.0238 (15)	−0.0001 (10)	0.0005 (11)	0.0021 (10)
C26	0.0243 (14)	0.0191 (12)	0.0205 (14)	−0.0010 (10)	0.0029 (11)	−0.0019 (10)
C27	0.0324 (16)	0.0244 (14)	0.0269 (16)	0.0064 (12)	0.0015 (13)	−0.0055 (12)
C28	0.0318 (17)	0.0352 (17)	0.0341 (19)	−0.0032 (14)	−0.0116 (14)	−0.0036 (14)
C29	0.0296 (16)	0.0257 (15)	0.0308 (17)	0.0070 (12)	0.0062 (13)	−0.0021 (13)

Geometric parameters (Å, º)

Br1—C13	1.903 (3)	C15—H15	0.9500
N1—C1	1.346 (4)	C16—H16	0.9500
N1—C11	1.423 (4)	C21—C26	1.392 (4)
N1—H1	0.85 (4)	C21—C22	1.401 (4)
O1—C1	1.232 (3)	C22—C23	1.378 (4)
O2—C23	1.367 (3)	C22—H22	0.9500
O2—C27	1.431 (3)	C23—C24	1.409 (4)
O3—C24	1.371 (4)	C24—C25	1.401 (4)
O3—C28	1.429 (4)	C25—C26	1.391 (4)
O4—C25	1.363 (3)	C26—H26	0.9500
O4—C29	1.426 (4)	C27—H27A	0.9800
C1—C21	1.490 (4)	C27—H27B	0.9800
C11—C12	1.389 (4)	C27—H27C	0.9800
C11—C16	1.396 (4)	C28—H28A	0.9800
C12—C13	1.380 (5)	C28—H28B	0.9800
C12—H12	0.9500	C28—H28C	0.9800
C13—C14	1.393 (5)	C29—H29A	0.9800
C14—C15	1.388 (5)	C29—H29B	0.9800
C14—H14	0.9500	C29—H29C	0.9800
C15—C16	1.379 (4)		
C1—N1—C11	125.9 (2)	C21—C22—H22	120.3
C1—N1—H1	124 (2)	O2—C23—C22	124.4 (3)
C11—N1—H1	110 (2)	O2—C23—C24	115.2 (2)
C23—O2—C27	117.1 (2)	C22—C23—C24	120.4 (3)
C24—O3—C28	114.4 (2)	O3—C24—C25	119.1 (3)
C25—O4—C29	116.3 (2)	O3—C24—C23	121.4 (2)
O1—C1—N1	123.2 (3)	C25—C24—C23	119.4 (3)
O1—C1—C21	120.6 (2)	O4—C25—C26	124.5 (3)
N1—C1—C21	116.2 (2)	O4—C25—C24	115.1 (3)
C12—C11—C16	120.1 (3)	C26—C25—C24	120.4 (2)
C12—C11—N1	121.9 (3)	C25—C26—C21	119.3 (3)
C16—C11—N1	117.9 (3)	C25—C26—H26	120.4
C13—C12—C11	118.3 (3)	C21—C26—H26	120.4
C13—C12—H12	120.8	O2—C27—H27A	109.5
C11—C12—H12	120.8	O2—C27—H27B	109.5
C12—C13—C14	122.9 (3)	H27A—C27—H27B	109.5

C12—C13—Br1	118.4 (3)	O2—C27—H27C	109.5
C14—C13—Br1	118.7 (2)	H27A—C27—H27C	109.5
C15—C14—C13	117.5 (3)	H27B—C27—H27C	109.5
C15—C14—H14	121.3	O3—C28—H28A	109.5
C13—C14—H14	121.3	O3—C28—H28B	109.5
C16—C15—C14	121.1 (3)	H28A—C28—H28B	109.5
C16—C15—H15	119.5	O3—C28—H28C	109.5
C14—C15—H15	119.5	H28A—C28—H28C	109.5
C15—C16—C11	120.1 (3)	H28B—C28—H28C	109.5
C15—C16—H16	119.9	O4—C29—H29A	109.5
C11—C16—H16	119.9	O4—C29—H29B	109.5
C26—C21—C22	121.0 (3)	H29A—C29—H29B	109.5
C26—C21—C1	122.4 (2)	O4—C29—H29C	109.5
C22—C21—C1	116.6 (2)	H29A—C29—H29C	109.5
C23—C22—C21	119.5 (3)	H29B—C29—H29C	109.5
C23—C22—H22	120.3		

C11—N1—C1—O1	−0.9 (5)	C27—O2—C23—C22	−4.6 (4)
C11—N1—C1—C21	178.3 (3)	C27—O2—C23—C24	176.5 (2)
C1—N1—C11—C12	−35.8 (4)	C21—C22—C23—O2	−176.9 (3)
C1—N1—C11—C16	147.8 (3)	C21—C22—C23—C24	1.9 (4)
C16—C11—C12—C13	0.3 (4)	C28—O3—C24—C25	111.7 (3)
N1—C11—C12—C13	−176.0 (3)	C28—O3—C24—C23	−72.5 (4)
C11—C12—C13—C14	−1.2 (4)	O2—C23—C24—O3	1.4 (4)
C11—C12—C13—Br1	176.5 (2)	C22—C23—C24—O3	−177.5 (3)
C12—C13—C14—C15	1.5 (5)	O2—C23—C24—C25	177.1 (2)
Br1—C13—C14—C15	−176.2 (2)	C22—C23—C24—C25	−1.8 (4)
C13—C14—C15—C16	−1.0 (5)	C29—O4—C25—C26	−2.5 (4)
C14—C15—C16—C11	0.2 (5)	C29—O4—C25—C24	177.7 (3)
C12—C11—C16—C15	0.2 (4)	O3—C24—C25—O4	−4.4 (4)
N1—C11—C16—C15	176.6 (3)	C23—C24—C25—O4	179.8 (2)
O1—C1—C21—C26	−146.1 (3)	O3—C24—C25—C26	175.8 (3)
N1—C1—C21—C26	34.6 (4)	C23—C24—C25—C26	0.0 (4)
O1—C1—C21—C22	32.5 (4)	O4—C25—C26—C21	−178.2 (2)
N1—C1—C21—C22	−146.8 (3)	C24—C25—C26—C21	1.6 (4)
C26—C21—C22—C23	−0.3 (4)	C22—C21—C26—C25	−1.5 (4)
C1—C21—C22—C23	−178.9 (2)	C1—C21—C26—C25	177.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.85 (4)	2.06 (4)	2.821 (3)	149 (3)

Symmetry code: (i) *x*, *y*−1, *z*.