

4'-(3-Bromophenyl)-1'-methylspiro-[indan-2,2'-pyrrolidine-3',2''-indan]-1,3,1''-trione

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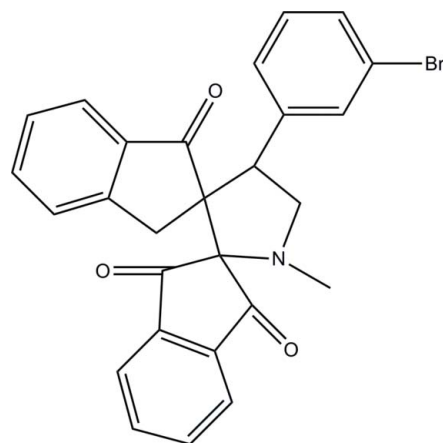
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.028; wR factor = 0.075; data-to-parameter ratio = 21.8.

In the title compound, $\text{C}_{27}\text{H}_{20}\text{BrNO}_3$, two intramolecular C—H...O hydrogen bonds both form $S(6)$ rings. The pyrrolidine ring adopts a twisted conformation about the C—C bond bearing the indane ring systems. The other two five-membered rings within the indane systems are in shallow envelope conformations, with the spiro C atoms as the flap atoms. The mean plane of the pyrrolidine ring [maximum deviation = 0.275 (1) Å] makes dihedral angles of 65.25 (7), 78.33 (6) and 75.25 (6)° with the bromo-substituted benzene ring and the mean planes of the mono- and dioxo-substituted indane rings, respectively. In the crystal, molecules are linked by C—H...O and C—H...N hydrogen bonds into a three-dimensional network. In addition, C—H... π interactions are observed.

Related literature

For related structures and medicinal background, see: Wei *et al.* (2011, 2012*a,b*). For ring conformations, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{27}\text{H}_{20}\text{BrNO}_3$

$M_r = 486.35$

Triclinic, $P\bar{1}$

$a = 8.3998$ (1) Å

$b = 11.2082$ (2) Å

$c = 12.4816$ (2) Å

$\alpha = 112.004$ (1)°

$\beta = 96.850$ (1)°

$\gamma = 93.191$ (1)°

$V = 1075.28$ (3) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 1.94$ mm⁻¹

$T = 100$ K

$0.51 \times 0.31 \times 0.29$ mm

Data collection

Bruker SMART APEXII CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2009)

$T_{\min} = 0.436$, $T_{\max} = 0.599$

23814 measured reflections

6318 independent reflections

5810 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.075$

$S = 1.04$

6318 reflections

290 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.52$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.53$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C21–C26 ring.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------|-------|-------------|-------------|---------------|
| C18—H18B...O2 | 0.99 | 2.39 | 3.0787 (17) | 126 |
| C19—H19A...O1 | 1.00 | 2.53 | 3.1649 (17) | 121 |
| C4—H4A...O3 ⁱ | 0.95 | 2.53 | 3.4056 (18) | 153 |
| C16—H16A...N1 ⁱⁱ | 0.95 | 2.58 | 3.4912 (16) | 161 |
| C20—H20B...O1 ⁱⁱⁱ | 0.99 | 2.44 | 3.3586 (18) | 153 |
| C23—H23A...O1 ^{iv} | 0.95 | 2.48 | 3.3847 (18) | 160 |
| C5—H5A...Cg1 ^v | 0.95 | 2.65 | 3.3386 (17) | 130 |
| C15—H15A...Cg1 ^{vi} | 0.95 | 2.82 | 3.6623 (15) | 149 |

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x, -y+1, -z+1$; (iii) $-x, -y+1, -z+2$; (iv) $-x+1, -y+1, -z+2$; (v) $x, y+1, z$; (vi) $-x+1, -y+1, -z+1$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

[‡] Thomson Reuters ResearcherID: F-9119-2012.

[§] Thomson Reuters ResearcherID: A-5599-2009.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6949).

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supporting information

Acta Cryst. (2012). E68, o2907–o2908 [https://doi.org/10.1107/S1600536812037993]

4'-(3-Bromophenyl)-1'-methyldispiro[indan-2,2'-pyrrolidine-3',2''-indan]-1,3,1''-trione

Ang Chee Wei, Mohamed Ashraf Ali, Tan Soo Choon, Suhana Arshad and Ibrahim Abdul Razak

S1. Comment

Tuberculosis (TB) remains a global health problem and has infected about one third of the world population. No new drugs have been discovered for the past 40 years and therefore new anti-TB agents are desperately needed. As part of our ongoing search for novel heterocyclic compounds with antitubercular activity (Wei *et al.*, 2011; 2012*a,b*), our group has synthesized the title compound as described below.

The molecular structure of the title compound is shown in Fig. 1. The intramolecular C18—H18B \cdots O2 and C19—H19A \cdots O1 hydrogen bonds (Table 1) form two *S*(6) ring motifs (Bernstein *et al.*, 1995). The pyrrolidine ring (N1/C9/C10/C19/C20) is twisted about C9—C10 bond [puckering parameters, $Q = 0.4492$ (14) Å and $\varphi = 242.64$ (18) $^\circ$], thereby adopting half-chair conformation (Cremer & Pople, 1975). Meanwhile, the other two five-membered rings within the indane moiety (C1/C2/C7—C9 & C10—C12/C17/C18) are in envelope conformation with puckering parameters, $Q = 0.1825$ (14) Å and $\varphi = 148.5$ (4) $^\circ$ in which C9 at the flap and $Q = 0.1694$ (14) Å and $\varphi = 172.8$ (5) $^\circ$ in which C10 at the flap, respectively. The mean plane of the pyrrolidine ring [N1/C9/C10/C19/C20, with maximum deviation of 0.275 (1) Å at atom C10] makes dihedral angles of 65.25 (7), 78.33 (6) and 75.25 (6) $^\circ$ with the bromo-substituted benzene ring (C21—C26) and the mean planes of the mono-oxo substituted [C10—C18; maximum deviation of 0.145 (1) Å at atom C10] and di-oxo substituted [C1—C9; maximum deviation of 0.180 (1) Å at atom C9] indane rings, respectively. The bond lengths and angles are within normal ranges and comparable to the related structure (Wei, Ali, Yoon *et al.*, 2011; Wei, Ali, Choon *et al.*, 2012*a,b*).

In the crystal (Fig. 2), C4—H4A \cdots O3, C20—H20B \cdots O1, C16—H16A \cdots N1 and C23—H23A \cdots O1 hydrogen bonds (Table 1) connect the molecules into a three-dimensional network. The crystal structure are further stabilized by the intermolecular C5—H5A \cdots Cg1 and C15—H15A \cdots Cg1 (Table 1) interactions (Cg1 is the centroid of C21—C26).

S2. Experimental

A mixture of (*E*)-2-(3-bromobenzylidene)-2,3-dihydro-1*H*-indene-1-one (0.001 mol), ninhydrin (0.001 mol) and sarcosine (0.002 mol) (1:1:2) were dissolved in methanol (10 ml) and refluxed for 4 h. After completion of the reaction as evident from TLC, the excess solvent was evaporated slowly and the product was separated and recrystallized from methanol to reveal the title compound as yellow blocks.

S3. Refinement

All H atoms were positioned geometrically [C—H = 0.95 and 1.00 Å] and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2$ and 1.5 $U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl group. One outlier -9 -1 2 was omitted in the final refinement.

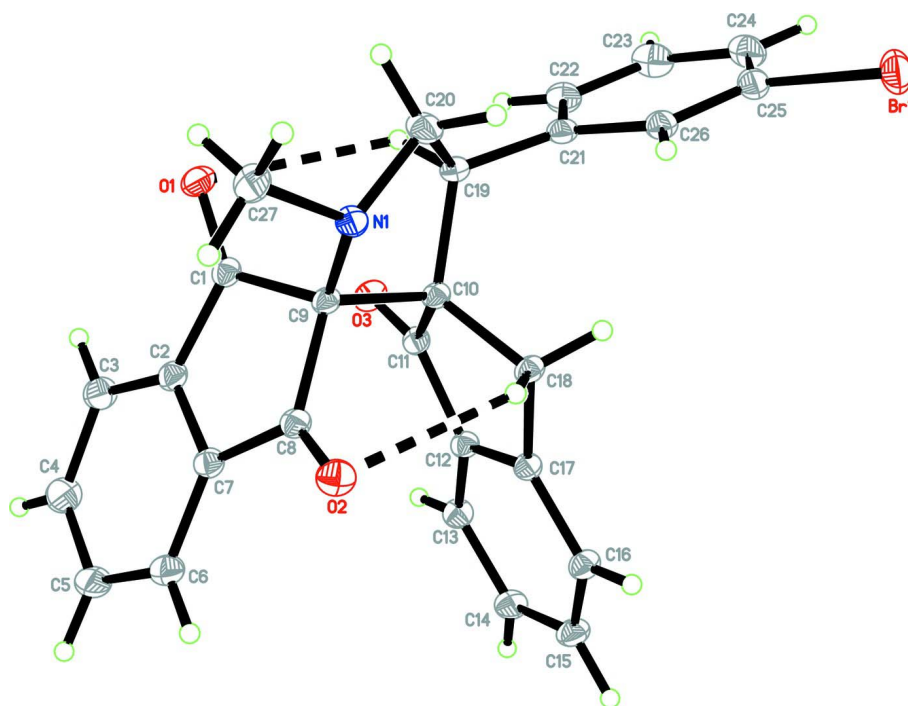


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

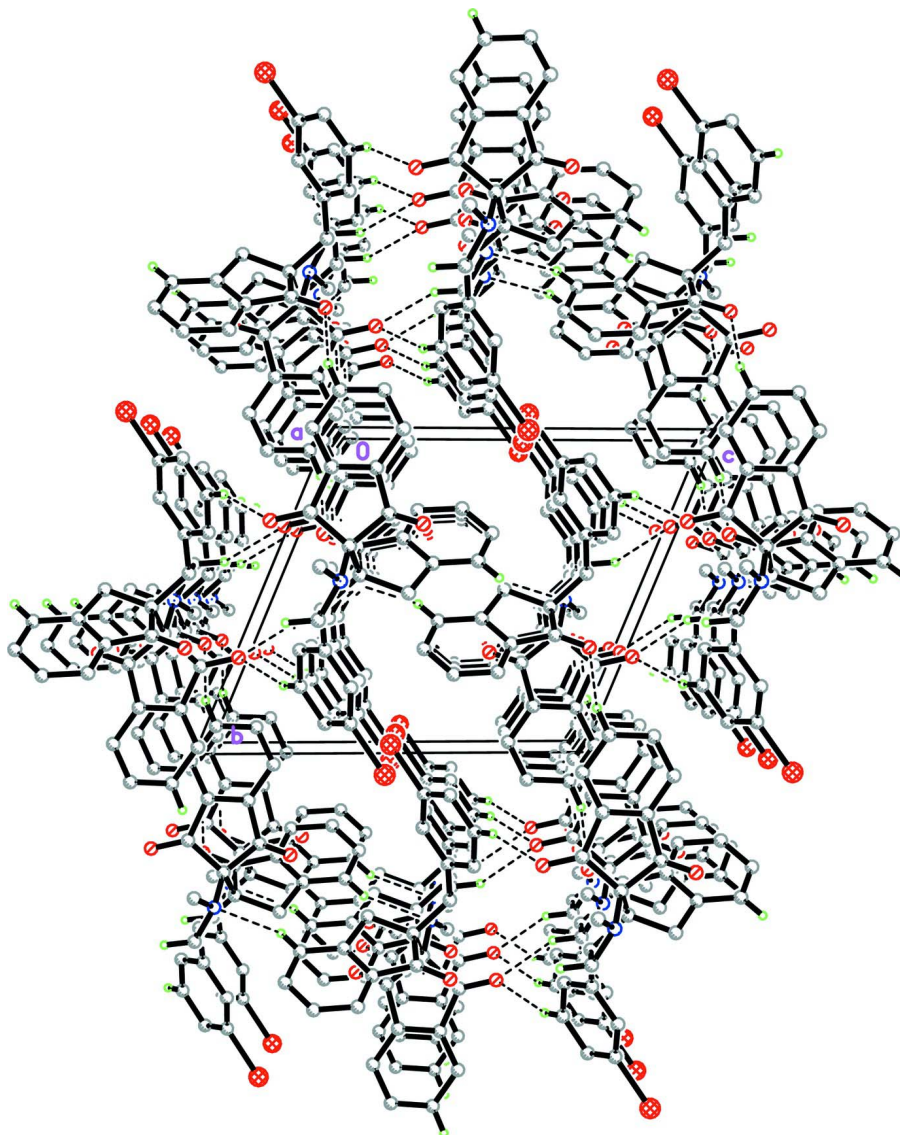


Figure 2

The crystal packing of the title compound. The H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

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Crystal data

$C_{27}H_{20}BrNO_3$

$M_r = 486.35$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.3998$ (1) Å

$b = 11.2082$ (2) Å

$c = 12.4816$ (2) Å

$\alpha = 112.004$ (1)°

$\beta = 96.850$ (1)°

$\gamma = 93.191$ (1)°

$V = 1075.28$ (3) Å³

$Z = 2$

$F(000) = 496$

$D_x = 1.502$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9917 reflections

$\theta = 3.0$ – 30.1 °

$\mu = 1.94$ mm⁻¹

$T = 100$ K

Block, yellow

$0.51 \times 0.31 \times 0.29$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.436$, $T_{\max} = 0.599$

23814 measured reflections
6318 independent reflections
5810 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 30.1^\circ$, $\theta_{\text{min}} = 1.8^\circ$
 $h = -11 \rightarrow 11$
 $k = -15 \rightarrow 15$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.075$
 $S = 1.04$
6318 reflections
290 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.5548P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.52 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.53 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|----------------|---------------|----------------------------------|
| Br1 | 0.225327 (19) | -0.034338 (13) | 0.491671 (13) | 0.02629 (5) |
| O1 | 0.13721 (12) | 0.70139 (9) | 1.01601 (8) | 0.01768 (18) |
| O2 | -0.06941 (12) | 0.68997 (9) | 0.64765 (8) | 0.01841 (18) |
| O3 | 0.45101 (11) | 0.66808 (9) | 0.88616 (8) | 0.01675 (18) |
| N1 | -0.05884 (13) | 0.52853 (10) | 0.78612 (9) | 0.01380 (19) |
| C1 | 0.12730 (14) | 0.72671 (12) | 0.92921 (10) | 0.0133 (2) |
| C2 | 0.15991 (15) | 0.85606 (12) | 0.92485 (11) | 0.0147 (2) |
| C3 | 0.24222 (17) | 0.96921 (13) | 1.01069 (12) | 0.0197 (3) |
| H3A | 0.2879 | 0.9714 | 1.0849 | 0.024* |
| C4 | 0.25502 (19) | 1.07871 (13) | 0.98385 (12) | 0.0233 (3) |
| H4A | 0.3133 | 1.1565 | 1.0398 | 0.028* |
| C5 | 0.18338 (19) | 1.07629 (13) | 0.87559 (13) | 0.0226 (3) |
| H5A | 0.1907 | 1.1534 | 0.8606 | 0.027* |
| C6 | 0.10196 (17) | 0.96343 (13) | 0.78980 (12) | 0.0188 (2) |

| | | | | |
|------|---------------|--------------|--------------|------------|
| H6A | 0.0534 | 0.9617 | 0.7164 | 0.023* |
| C7 | 0.09426 (15) | 0.85257 (12) | 0.81561 (11) | 0.0144 (2) |
| C8 | 0.01860 (14) | 0.72063 (12) | 0.73996 (11) | 0.0134 (2) |
| C9 | 0.06749 (14) | 0.62884 (11) | 0.80214 (10) | 0.0118 (2) |
| C10 | 0.20794 (14) | 0.54710 (11) | 0.75111 (10) | 0.0117 (2) |
| C11 | 0.37225 (14) | 0.63118 (11) | 0.78907 (11) | 0.0128 (2) |
| C12 | 0.41207 (14) | 0.65657 (12) | 0.68743 (10) | 0.0128 (2) |
| C13 | 0.53931 (15) | 0.73898 (12) | 0.68183 (11) | 0.0152 (2) |
| H13A | 0.6138 | 0.7891 | 0.7497 | 0.018* |
| C14 | 0.55375 (16) | 0.74547 (13) | 0.57472 (12) | 0.0172 (2) |
| H14A | 0.6395 | 0.8002 | 0.5683 | 0.021* |
| C15 | 0.44190 (16) | 0.67141 (13) | 0.47562 (11) | 0.0172 (2) |
| H15A | 0.4525 | 0.6778 | 0.4030 | 0.021* |
| C16 | 0.31575 (15) | 0.58883 (12) | 0.48106 (11) | 0.0152 (2) |
| H16A | 0.2412 | 0.5388 | 0.4132 | 0.018* |
| C17 | 0.30179 (14) | 0.58155 (11) | 0.58869 (10) | 0.0126 (2) |
| C18 | 0.18261 (15) | 0.49507 (12) | 0.61636 (10) | 0.0129 (2) |
| H18A | 0.2052 | 0.4037 | 0.5829 | 0.016* |
| H18B | 0.0707 | 0.5013 | 0.5852 | 0.016* |
| C19 | 0.19356 (15) | 0.44614 (12) | 0.80828 (11) | 0.0137 (2) |
| H19A | 0.2382 | 0.4924 | 0.8930 | 0.016* |
| C20 | 0.01072 (16) | 0.41788 (13) | 0.80165 (13) | 0.0186 (2) |
| H20A | -0.0317 | 0.3363 | 0.7348 | 0.022* |
| H20B | -0.0147 | 0.4104 | 0.8745 | 0.022* |
| C21 | 0.28456 (15) | 0.32899 (12) | 0.76277 (11) | 0.0141 (2) |
| C22 | 0.43223 (16) | 0.32640 (13) | 0.82660 (12) | 0.0183 (2) |
| H22A | 0.4753 | 0.3995 | 0.8952 | 0.022* |
| C23 | 0.51708 (17) | 0.21825 (15) | 0.79110 (14) | 0.0230 (3) |
| H23A | 0.6175 | 0.2186 | 0.8353 | 0.028* |
| C24 | 0.45598 (17) | 0.11009 (14) | 0.69174 (14) | 0.0224 (3) |
| H24A | 0.5124 | 0.0356 | 0.6678 | 0.027* |
| C25 | 0.31020 (17) | 0.11346 (12) | 0.62810 (12) | 0.0182 (2) |
| C26 | 0.22394 (15) | 0.22029 (12) | 0.66123 (11) | 0.0156 (2) |
| H26A | 0.1248 | 0.2198 | 0.6156 | 0.019* |
| C27 | -0.19926 (16) | 0.56775 (13) | 0.84332 (12) | 0.0185 (2) |
| H27A | -0.2416 | 0.6379 | 0.8236 | 0.028* |
| H27B | -0.1682 | 0.5980 | 0.9282 | 0.028* |
| H27C | -0.2825 | 0.4938 | 0.8167 | 0.028* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| Br1 | 0.03101 (9) | 0.01684 (7) | 0.02711 (8) | 0.00156 (5) | 0.01300 (6) | 0.00162 (5) |
| O1 | 0.0203 (4) | 0.0199 (4) | 0.0145 (4) | 0.0010 (4) | 0.0044 (3) | 0.0082 (4) |
| O2 | 0.0171 (4) | 0.0200 (4) | 0.0177 (4) | 0.0007 (3) | -0.0005 (3) | 0.0080 (4) |
| O3 | 0.0142 (4) | 0.0212 (4) | 0.0140 (4) | -0.0012 (3) | 0.0008 (3) | 0.0067 (3) |
| N1 | 0.0116 (4) | 0.0132 (4) | 0.0174 (5) | -0.0003 (4) | 0.0051 (4) | 0.0062 (4) |
| C1 | 0.0120 (5) | 0.0145 (5) | 0.0135 (5) | 0.0006 (4) | 0.0039 (4) | 0.0052 (4) |

| | | | | | | |
|-----|------------|------------|------------|-------------|------------|------------|
| C2 | 0.0161 (5) | 0.0141 (5) | 0.0140 (5) | 0.0001 (4) | 0.0048 (4) | 0.0051 (4) |
| C3 | 0.0250 (7) | 0.0161 (6) | 0.0157 (6) | -0.0022 (5) | 0.0023 (5) | 0.0045 (5) |
| C4 | 0.0316 (7) | 0.0152 (6) | 0.0200 (6) | -0.0049 (5) | 0.0047 (5) | 0.0041 (5) |
| C5 | 0.0326 (7) | 0.0151 (6) | 0.0222 (6) | -0.0016 (5) | 0.0081 (5) | 0.0088 (5) |
| C6 | 0.0236 (6) | 0.0172 (6) | 0.0179 (6) | 0.0008 (5) | 0.0059 (5) | 0.0088 (5) |
| C7 | 0.0145 (5) | 0.0141 (5) | 0.0150 (5) | 0.0004 (4) | 0.0044 (4) | 0.0057 (4) |
| C8 | 0.0121 (5) | 0.0149 (5) | 0.0148 (5) | 0.0016 (4) | 0.0044 (4) | 0.0069 (4) |
| C9 | 0.0119 (5) | 0.0124 (5) | 0.0117 (5) | -0.0003 (4) | 0.0030 (4) | 0.0051 (4) |
| C10 | 0.0110 (5) | 0.0126 (5) | 0.0118 (5) | -0.0006 (4) | 0.0029 (4) | 0.0050 (4) |
| C11 | 0.0115 (5) | 0.0129 (5) | 0.0143 (5) | 0.0011 (4) | 0.0039 (4) | 0.0050 (4) |
| C12 | 0.0120 (5) | 0.0141 (5) | 0.0135 (5) | 0.0014 (4) | 0.0034 (4) | 0.0062 (4) |
| C13 | 0.0130 (5) | 0.0160 (5) | 0.0168 (5) | -0.0006 (4) | 0.0026 (4) | 0.0066 (4) |
| C14 | 0.0160 (6) | 0.0177 (5) | 0.0203 (6) | -0.0007 (4) | 0.0060 (5) | 0.0094 (5) |
| C15 | 0.0186 (6) | 0.0201 (6) | 0.0166 (5) | 0.0023 (5) | 0.0065 (4) | 0.0102 (5) |
| C16 | 0.0154 (5) | 0.0169 (5) | 0.0138 (5) | 0.0003 (4) | 0.0035 (4) | 0.0063 (4) |
| C17 | 0.0125 (5) | 0.0129 (5) | 0.0137 (5) | 0.0014 (4) | 0.0042 (4) | 0.0058 (4) |
| C18 | 0.0133 (5) | 0.0140 (5) | 0.0112 (5) | -0.0018 (4) | 0.0026 (4) | 0.0048 (4) |
| C19 | 0.0157 (5) | 0.0136 (5) | 0.0140 (5) | 0.0011 (4) | 0.0048 (4) | 0.0069 (4) |
| C20 | 0.0171 (6) | 0.0155 (5) | 0.0280 (7) | 0.0027 (4) | 0.0109 (5) | 0.0114 (5) |
| C21 | 0.0150 (5) | 0.0147 (5) | 0.0157 (5) | 0.0014 (4) | 0.0053 (4) | 0.0084 (4) |
| C22 | 0.0161 (6) | 0.0201 (6) | 0.0205 (6) | 0.0007 (5) | 0.0022 (5) | 0.0101 (5) |
| C23 | 0.0160 (6) | 0.0273 (7) | 0.0309 (7) | 0.0049 (5) | 0.0034 (5) | 0.0167 (6) |
| C24 | 0.0211 (6) | 0.0215 (6) | 0.0313 (7) | 0.0083 (5) | 0.0116 (5) | 0.0148 (6) |
| C25 | 0.0204 (6) | 0.0148 (5) | 0.0208 (6) | 0.0015 (5) | 0.0092 (5) | 0.0067 (5) |
| C26 | 0.0163 (5) | 0.0160 (5) | 0.0168 (5) | 0.0020 (4) | 0.0052 (4) | 0.0081 (5) |
| C27 | 0.0138 (5) | 0.0205 (6) | 0.0226 (6) | 0.0018 (4) | 0.0074 (5) | 0.0084 (5) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|----------|-------------|
| Br1—C25 | 1.9019 (14) | C13—H13A | 0.9500 |
| O1—C1 | 1.2135 (15) | C14—C15 | 1.4030 (18) |
| O2—C8 | 1.2100 (15) | C14—H14A | 0.9500 |
| O3—C11 | 1.2165 (15) | C15—C16 | 1.3925 (17) |
| N1—C9 | 1.4416 (15) | C15—H15A | 0.9500 |
| N1—C27 | 1.4553 (16) | C16—C17 | 1.3943 (16) |
| N1—C20 | 1.4665 (16) | C16—H16A | 0.9500 |
| C1—C2 | 1.4827 (17) | C17—C18 | 1.5120 (16) |
| C1—C9 | 1.5544 (17) | C18—H18A | 0.9900 |
| C2—C3 | 1.3935 (17) | C18—H18B | 0.9900 |
| C2—C7 | 1.3939 (17) | C19—C21 | 1.5123 (17) |
| C3—C4 | 1.3900 (19) | C19—C20 | 1.5369 (18) |
| C3—H3A | 0.9500 | C19—H19A | 1.0000 |
| C4—C5 | 1.402 (2) | C20—H20A | 0.9900 |
| C4—H4A | 0.9500 | C20—H20B | 0.9900 |
| C5—C6 | 1.3887 (19) | C21—C22 | 1.4000 (18) |
| C5—H5A | 0.9500 | C21—C26 | 1.4035 (18) |
| C6—C7 | 1.3956 (17) | C22—C23 | 1.3938 (19) |
| C6—H6A | 0.9500 | C22—H22A | 0.9500 |

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|-------------|-------------|---------------|-------------|
| C7—C8 | 1.4793 (17) | C23—C24 | 1.387 (2) |
| C8—C9 | 1.5488 (16) | C23—H23A | 0.9500 |
| C9—C10 | 1.5809 (17) | C24—C25 | 1.388 (2) |
| C10—C18 | 1.5427 (16) | C24—H24A | 0.9500 |
| C10—C11 | 1.5434 (16) | C25—C26 | 1.3881 (18) |
| C10—C19 | 1.5540 (16) | C26—H26A | 0.9500 |
| C11—C12 | 1.4708 (16) | C27—H27A | 0.9800 |
| C12—C17 | 1.3959 (17) | C27—H27B | 0.9800 |
| C12—C13 | 1.3988 (16) | C27—H27C | 0.9800 |
| C13—C14 | 1.3849 (17) | | |
| | | | |
| C9—N1—C27 | 117.31 (10) | C16—C15—C14 | 121.67 (11) |
| C9—N1—C20 | 110.09 (10) | C16—C15—H15A | 119.2 |
| C27—N1—C20 | 115.37 (10) | C14—C15—H15A | 119.2 |
| O1—C1—C2 | 126.85 (11) | C15—C16—C17 | 118.25 (11) |
| O1—C1—C9 | 125.46 (11) | C15—C16—H16A | 120.9 |
| C2—C1—C9 | 107.59 (10) | C17—C16—H16A | 120.9 |
| C3—C2—C7 | 121.15 (11) | C16—C17—C12 | 119.94 (11) |
| C3—C2—C1 | 129.27 (11) | C16—C17—C18 | 128.53 (11) |
| C7—C2—C1 | 109.57 (11) | C12—C17—C18 | 111.46 (10) |
| C4—C3—C2 | 117.60 (13) | C17—C18—C10 | 104.24 (9) |
| C4—C3—H3A | 121.2 | C17—C18—H18A | 110.9 |
| C2—C3—H3A | 121.2 | C10—C18—H18A | 110.9 |
| C3—C4—C5 | 121.12 (13) | C17—C18—H18B | 110.9 |
| C3—C4—H4A | 119.4 | C10—C18—H18B | 110.9 |
| C5—C4—H4A | 119.4 | H18A—C18—H18B | 108.9 |
| C6—C5—C4 | 121.28 (12) | C21—C19—C20 | 115.88 (10) |
| C6—C5—H5A | 119.4 | C21—C19—C10 | 116.93 (10) |
| C4—C5—H5A | 119.4 | C20—C19—C10 | 103.69 (10) |
| C5—C6—C7 | 117.38 (12) | C21—C19—H19A | 106.5 |
| C5—C6—H6A | 121.3 | C20—C19—H19A | 106.5 |
| C7—C6—H6A | 121.3 | C10—C19—H19A | 106.5 |
| C2—C7—C6 | 121.37 (12) | N1—C20—C19 | 105.19 (10) |
| C2—C7—C8 | 110.26 (10) | N1—C20—H20A | 110.7 |
| C6—C7—C8 | 128.36 (11) | C19—C20—H20A | 110.7 |
| O2—C8—C7 | 126.41 (11) | N1—C20—H20B | 110.7 |
| O2—C8—C9 | 125.88 (11) | C19—C20—H20B | 110.7 |
| C7—C8—C9 | 107.69 (10) | H20A—C20—H20B | 108.8 |
| N1—C9—C8 | 113.45 (10) | C22—C21—C26 | 118.48 (12) |
| N1—C9—C1 | 117.20 (10) | C22—C21—C19 | 118.89 (11) |
| C8—C9—C1 | 101.52 (9) | C26—C21—C19 | 122.59 (11) |
| N1—C9—C10 | 101.75 (9) | C23—C22—C21 | 121.04 (13) |
| C8—C9—C10 | 113.18 (9) | C23—C22—H22A | 119.5 |
| C1—C9—C10 | 110.18 (9) | C21—C22—H22A | 119.5 |
| C18—C10—C11 | 104.54 (9) | C24—C23—C22 | 120.48 (13) |
| C18—C10—C19 | 117.18 (10) | C24—C23—H23A | 119.8 |
| C11—C10—C19 | 113.68 (10) | C22—C23—H23A | 119.8 |
| C18—C10—C9 | 111.30 (9) | C23—C24—C25 | 118.31 (13) |

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|---------------|--------------|-----------------|--------------|
| C11—C10—C9 | 111.58 (9) | C23—C24—H24A | 120.8 |
| C19—C10—C9 | 98.74 (9) | C25—C24—H24A | 120.8 |
| O3—C11—C12 | 127.83 (11) | C26—C25—C24 | 122.29 (13) |
| O3—C11—C10 | 124.83 (11) | C26—C25—Br1 | 119.05 (10) |
| C12—C11—C10 | 107.34 (10) | C24—C25—Br1 | 118.66 (10) |
| C17—C12—C13 | 121.81 (11) | C25—C26—C21 | 119.40 (12) |
| C17—C12—C11 | 109.50 (10) | C25—C26—H26A | 120.3 |
| C13—C12—C11 | 128.68 (11) | C21—C26—H26A | 120.3 |
| C14—C13—C12 | 118.21 (11) | N1—C27—H27A | 109.5 |
| C14—C13—H13A | 120.9 | N1—C27—H27B | 109.5 |
| C12—C13—H13A | 120.9 | H27A—C27—H27B | 109.5 |
| C13—C14—C15 | 120.12 (12) | N1—C27—H27C | 109.5 |
| C13—C14—H14A | 119.9 | H27A—C27—H27C | 109.5 |
| C15—C14—H14A | 119.9 | H27B—C27—H27C | 109.5 |
| O1—C1—C2—C3 | 16.5 (2) | C9—C10—C11—O3 | 75.27 (15) |
| C9—C1—C2—C3 | -166.90 (13) | C18—C10—C11—C12 | 15.76 (12) |
| O1—C1—C2—C7 | -163.84 (13) | C19—C10—C11—C12 | 144.75 (10) |
| C9—C1—C2—C7 | 12.81 (14) | C9—C10—C11—C12 | -104.65 (11) |
| C7—C2—C3—C4 | 0.6 (2) | O3—C11—C12—C17 | 171.44 (12) |
| C1—C2—C3—C4 | -179.67 (13) | C10—C11—C12—C17 | -8.65 (13) |
| C2—C3—C4—C5 | 1.9 (2) | O3—C11—C12—C13 | -7.4 (2) |
| C3—C4—C5—C6 | -2.3 (2) | C10—C11—C12—C13 | 172.53 (12) |
| C4—C5—C6—C7 | 0.0 (2) | C17—C12—C13—C14 | 0.34 (19) |
| C3—C2—C7—C6 | -3.0 (2) | C11—C12—C13—C14 | 179.03 (12) |
| C1—C2—C7—C6 | 177.30 (12) | C12—C13—C14—C15 | 0.47 (19) |
| C3—C2—C7—C8 | 178.12 (12) | C13—C14—C15—C16 | -0.9 (2) |
| C1—C2—C7—C8 | -1.62 (14) | C14—C15—C16—C17 | 0.40 (19) |
| C5—C6—C7—C2 | 2.6 (2) | C15—C16—C17—C12 | 0.41 (18) |
| C5—C6—C7—C8 | -178.72 (13) | C15—C16—C17—C18 | -176.36 (12) |
| C2—C7—C8—O2 | 167.84 (13) | C13—C12—C17—C16 | -0.79 (18) |
| C6—C7—C8—O2 | -11.0 (2) | C11—C12—C17—C16 | -179.71 (11) |
| C2—C7—C8—C9 | -10.25 (14) | C13—C12—C17—C18 | 176.50 (11) |
| C6—C7—C8—C9 | 170.92 (13) | C11—C12—C17—C18 | -2.42 (14) |
| C27—N1—C9—C8 | -69.28 (14) | C16—C17—C18—C10 | -170.69 (12) |
| C20—N1—C9—C8 | 156.11 (10) | C12—C17—C18—C10 | 12.31 (13) |
| C27—N1—C9—C1 | 48.61 (15) | C11—C10—C18—C17 | -16.54 (12) |
| C20—N1—C9—C1 | -86.00 (13) | C19—C10—C18—C17 | -143.39 (10) |
| C27—N1—C9—C10 | 168.82 (10) | C9—C10—C18—C17 | 104.06 (11) |
| C20—N1—C9—C10 | 34.20 (12) | C18—C10—C19—C21 | 48.01 (15) |
| O2—C8—C9—N1 | -34.63 (17) | C11—C10—C19—C21 | -74.23 (13) |
| C7—C8—C9—N1 | 143.47 (10) | C9—C10—C19—C21 | 167.49 (10) |
| O2—C8—C9—C1 | -161.28 (12) | C18—C10—C19—C20 | -80.86 (12) |
| C7—C8—C9—C1 | 16.82 (12) | C11—C10—C19—C20 | 156.90 (10) |
| O2—C8—C9—C10 | 80.66 (15) | C9—C10—C19—C20 | 38.62 (11) |
| C7—C8—C9—C10 | -101.24 (11) | C9—N1—C20—C19 | -9.38 (14) |
| O1—C1—C9—N1 | 34.79 (18) | C27—N1—C20—C19 | -144.95 (11) |
| C2—C1—C9—N1 | -141.92 (11) | C21—C19—C20—N1 | -149.54 (10) |

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| O1—C1—C9—C8 | 158.94 (12) | C10—C19—C20—N1 | -20.02 (13) |
| C2—C1—C9—C8 | -17.76 (12) | C20—C19—C21—C22 | -136.87 (12) |
| O1—C1—C9—C10 | -80.86 (15) | C10—C19—C21—C22 | 100.35 (13) |
| C2—C1—C9—C10 | 102.43 (11) | C20—C19—C21—C26 | 40.61 (16) |
| N1—C9—C10—C18 | 79.65 (11) | C10—C19—C21—C26 | -82.17 (15) |
| C8—C9—C10—C18 | -42.44 (13) | C26—C21—C22—C23 | -0.50 (19) |
| C1—C9—C10—C18 | -155.32 (9) | C19—C21—C22—C23 | 177.08 (12) |
| N1—C9—C10—C11 | -163.99 (9) | C21—C22—C23—C24 | -0.5 (2) |
| C8—C9—C10—C11 | 73.92 (12) | C22—C23—C24—C25 | 1.0 (2) |
| C1—C9—C10—C11 | -38.96 (12) | C23—C24—C25—C26 | -0.6 (2) |
| N1—C9—C10—C19 | -44.13 (10) | C23—C24—C25—Br1 | 179.99 (10) |
| C8—C9—C10—C19 | -166.22 (10) | C24—C25—C26—C21 | -0.34 (19) |
| C1—C9—C10—C19 | 80.89 (11) | Br1—C25—C26—C21 | 179.05 (9) |
| C18—C10—C11—O3 | -164.32 (12) | C22—C21—C26—C25 | 0.89 (18) |
| C19—C10—C11—O3 | -35.33 (17) | C19—C21—C26—C25 | -176.60 (11) |

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the C21–C26 ring.

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---|-------------|---------------------|----------------------------|-------------------------------|
| C18—H18 <i>B</i> \cdots O2 | 0.99 | 2.39 | 3.0787 (17) | 126 |
| C19—H19 <i>A</i> \cdots O1 | 1.00 | 2.53 | 3.1649 (17) | 121 |
| C4—H4 <i>A</i> \cdots O3 ⁱ | 0.95 | 2.53 | 3.4056 (18) | 153 |
| C16—H16 <i>A</i> \cdots N1 ⁱⁱ | 0.95 | 2.58 | 3.4912 (16) | 161 |
| C20—H20 <i>B</i> \cdots O1 ⁱⁱⁱ | 0.99 | 2.44 | 3.3586 (18) | 153 |
| C23—H23 <i>A</i> \cdots O1 ^{iv} | 0.95 | 2.48 | 3.3847 (18) | 160 |
| C5—H5 <i>A</i> \cdots Cg1 ^v | 0.95 | 2.65 | 3.3386 (17) | 130 |
| C15—H15 <i>A</i> \cdots Cg1 ^{vi} | 0.95 | 2.82 | 3.6623 (15) | 149 |

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x, -y+1, -z+1$; (iii) $-x, -y+1, -z+2$; (iv) $-x+1, -y+1, -z+2$; (v) $x, y+1, z$; (vi) $-x+1, -y+1, -z+1$.