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# Diethyl 3,3′-(phenylmethylene)bis(1*H*-indole-2-carboxylate)

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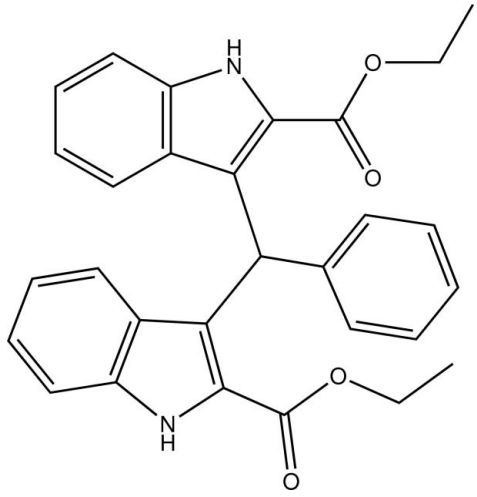
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.059; $wR$ factor = 0.144; data-to-parameter ratio = 14.4.

In the title compound, $C_{29}H_{26}N_2O_4$, the benzene ring is twisted by 73.5 (5) and 84.9 (3)° with respect to the mean planes of the two indole ring systems; the mean planes of the indole ring systems are oriented at a dihedral angle of 82.0 (5)°. In the crystal, molecules are linked by pairs of N—H···O hydrogen bonds into chains.

## Related literature

For applications of indole derivatives, see: Poter *et al.* (1977); Sundberg (1996); Chang *et al.* (1999); Ge *et al.* (1999); Ni (2008).



## Experimental

### Crystal data

$C_{29}H_{26}N_2O_4$
$M_r$ = 466.52
Triclinic, $P\overline{1}$
$a$ = 8.7340 (17) Å
$b$ = 9.871 (2) Å
$c$ = 15.000 (3) Å
$\alpha$ = 76.14 (3)°
$\beta$ = 83.91 (3)°
$\gamma$ = 83.09 (3)°
$V$ = 1242.4 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
4879 measured reflections
4557 independent reflections
2813 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
3 standard reflections every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.144
$S$ = 1.00
4557 reflections
316 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···O2$^{i}$ | 0.86 | 2.21 | 2.955 (3) | 144 |
| N2—H2$A$···O4$^{ii}$ | 0.86 | 2.07 | 2.880 (3) | 157 |

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

The authors thank the Center of Testing and Analysis, Nanjing University, for assistance with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5612).

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# supporting information

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# Diethyl 3,3′-(phenylmethylene)bis(1*H*-indole-2-carboxylate)

**Hong-Shun Sun, Yu-Long Li, Ning Xu, Hong Xu and Ji-Dong Zhang**

## S1. Comment

Indole derivatives are found abundantly in a variety of natural plants and exhibit various physiological properties (Poter *et al.*, 1977; Sundberg, 1996). Among them, bis-indolymethane derivatives are found to be kinds of potentially bioactive compounds (Chang *et al.*, 1999; Ge *et al.*, 1999). In recent years, the synthesis and application of bis-indolymethane derivatives have been widely studied. The title compound is one of bis-indolymethane derivatives as a precursor for MRI Contrast Agents (Ni, 2008). We report here its crystal structure.

The molecular structure of the title compound is shown in Fig. 1. The benzene ring is twisted to the two indole rings with the dihedral angles of 73.5 (5) and 84.9 (3)°, respectively. Two indole rings make a dihedral angle of 82.0 (5)° to each other.

As shown in Figure 2, the molecules are linked by paired N—H···O hydrogen bonds into dimers in the crystal lattice. The structural parameters for the intermolecular hydrogen bonds resulting in the formation of dimers are given in Table 1.

## S2. Experimental

Ethyl indole-2-carboxylate (18.9 g, 100 mmol) was dissolved in 200 ml ethanol; commercially available benzaldehyde (5.3 g, 50 mmol) was added and the mixture was heated to reflux temperature. Concentrated HCl (3.7 ml) was added and the reaction was left for 1 h. After cooling the white product was filtered off and washed thoroughly with ethanol. The reaction can be followed by TLC ($CHCl_3$:hexane = 1:1). Yield was 90%. Crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

## S3. Refinement

H atoms were positioned geometrically, with N— H = 0.86 Å and C—H = 0.93, 0.96, 0.97 and 0.98 Å for aromatic, methyl, methylene and methine H, respectively, and constrained to ride on their parent atoms, with $U_{iso}$(H) = x$U_{eq}$(C,N), where $x$ = 1.5 for methyl H, and $x$ = 1.2 for all other H atoms.

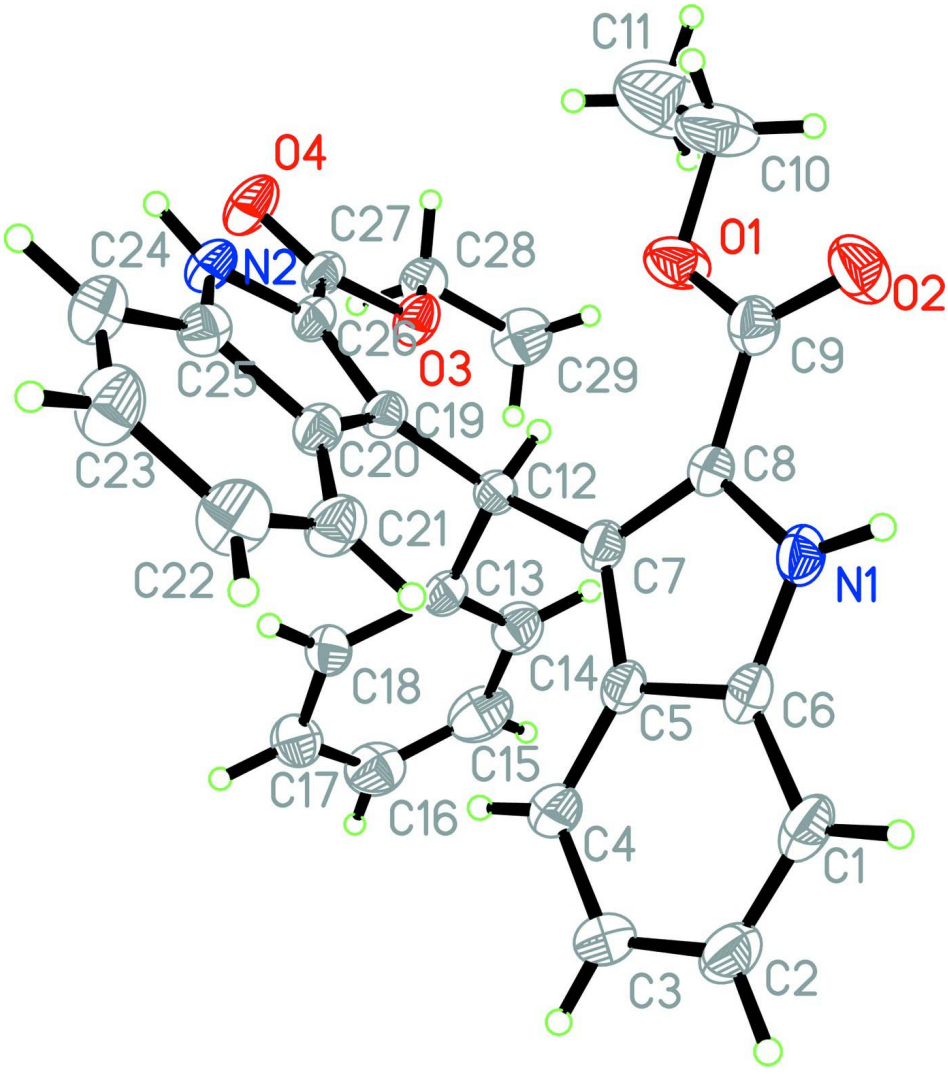


**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

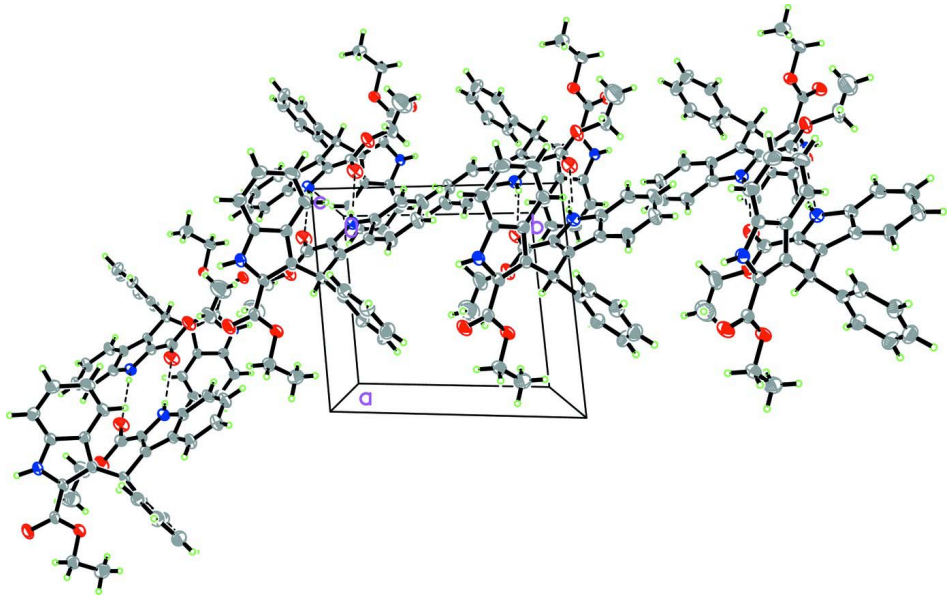


**Figure 2**

A packing diagram of (I). Intermolecular hydrogen bonds are shown as dashed lines.

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*Crystal data*

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$\gamma = 83.09\ (3)°$
$V = 1242.4\ (4)$ Å$^3$
$Z = 2$
$F(000) = 492$
$D_x = 1.247$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–13°$
$\mu = 0.08$ mm$^{-1}$
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
4879 measured reflections
4557 independent reflections
2813 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.4°$
$h = 0\rightarrow10$
$k = -11\rightarrow11$
$l = -17\rightarrow18$
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.144$
$S = 1.00$
4557 reflections
316 parameters
2 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

$w = 1/[\sigma^2(F_o^2) + (0.068P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.1091 (2) | 1.1102 (2) | 0.57403 (13) | 0.0464 (5) |
| H1A | 0.0562 | 1.1030 | 0.5305 | 0.056* |
| O1 | 0.3652 (2) | 0.79594 (18) | 0.61548 (14) | 0.0671 (6) |
| C1 | 0.0036 (3) | 1.3484 (3) | 0.59245 (18) | 0.0543 (7) |
| H1B | −0.0653 | 1.3654 | 0.5469 | 0.065* |
| N2 | 0.3177 (2) | 0.6564 (2) | 0.93548 (13) | 0.0455 (5) |
| H2A | 0.3453 | 0.5760 | 0.9700 | 0.055* |
| O2 | 0.1783 (2) | 0.8636 (2) | 0.51907 (13) | 0.0684 (6) |
| C2 | 0.0155 (3) | 1.4450 (3) | 0.64135 (19) | 0.0622 (8) |
| H2B | −0.0449 | 1.5305 | 0.6283 | 0.075* |
| O3 | 0.65528 (18) | 0.75379 (17) | 0.78745 (11) | 0.0501 (5) |
| C3 | 0.1166 (3) | 1.4189 (3) | 0.71086 (19) | 0.0617 (8) |
| H3A | 0.1214 | 1.4872 | 0.7435 | 0.074* |
| O4 | 0.6289 (2) | 0.57275 (18) | 0.90776 (13) | 0.0650 (6) |
| C4 | 0.2091 (3) | 1.2949 (3) | 0.73230 (18) | 0.0556 (7) |
| H4A | 0.2753 | 1.2789 | 0.7791 | 0.067* |
| C5 | 0.2020 (3) | 1.1927 (2) | 0.68248 (15) | 0.0408 (6) |
| C6 | 0.0979 (3) | 1.2226 (3) | 0.61280 (16) | 0.0451 (6) |
| C7 | 0.2770 (3) | 1.0539 (2) | 0.68458 (15) | 0.0384 (6) |
| C8 | 0.2186 (3) | 1.0099 (2) | 0.61581 (15) | 0.0402 (6) |
| C9 | 0.2502 (3) | 0.8841 (3) | 0.57918 (18) | 0.0491 (6) |
| C10 | 0.4101 (4) | 0.6726 (3) | 0.5781 (3) | 0.0934 (12) |
| H10A | 0.3348 | 0.6054 | 0.6016 | 0.112* |
| H10B | 0.4081 | 0.6996 | 0.5116 | 0.112* |
| C11 | 0.5551 (5) | 0.6089 (5) | 0.5996 (4) | 0.1460 (19) |
| H11A | 0.5781 | 0.5285 | 0.5733 | 0.219* |
| H11B | 0.5575 | 0.5800 | 0.6653 | 0.219* |
| H11C | 0.6308 | 0.6740 | 0.5752 | 0.219* |
| C12 | 0.3962 (3) | 0.9705 (2) | 0.74867 (15) | 0.0373 (6) |
| H12A | 0.4708 | 0.9217 | 0.7106 | 0.045* |

| | | | | |
|---|---|---|---|---|
| C13 | 0.4886 (3) | 1.0644 (2) | 0.78505 (16) | 0.0395 (6) |
| C14 | 0.6008 (3) | 1.1351 (3) | 0.72766 (19) | 0.0568 (7) |
| H14A | 0.6244 | 1.1202 | 0.6686 | 0.068* |
| C15 | 0.6790 (4) | 1.2282 (3) | 0.7569 (3) | 0.0764 (9) |
| H15A | 0.7541 | 1.2759 | 0.7170 | 0.092* |
| C16 | 0.6473 (4) | 1.2511 (3) | 0.8438 (2) | 0.0710 (9) |
| H16A | 0.6991 | 1.3154 | 0.8624 | 0.085* |
| C17 | 0.5397 (3) | 1.1794 (3) | 0.9029 (2) | 0.0611 (8) |
| H17A | 0.5190 | 1.1934 | 0.9623 | 0.073* |
| C18 | 0.4605 (3) | 1.0849 (3) | 0.87424 (17) | 0.0478 (6) |
| H18A | 0.3882 | 1.0350 | 0.9151 | 0.057* |
| C19 | 0.3294 (3) | 0.8562 (2) | 0.82476 (15) | 0.0374 (6) |
| C20 | 0.1766 (3) | 0.8539 (2) | 0.87173 (15) | 0.0395 (6) |
| C21 | 0.0399 (3) | 0.9474 (3) | 0.86615 (18) | 0.0511 (7) |
| H21A | 0.0363 | 1.0322 | 0.8226 | 0.061* |
| C22 | −0.0862 (3) | 0.9119 (3) | 0.92517 (19) | 0.0619 (8) |
| H22A | −0.1757 | 0.9738 | 0.9214 | 0.074* |
| C23 | −0.0847 (3) | 0.7852 (3) | 0.9912 (2) | 0.0632 (8) |
| H23A | −0.1734 | 0.7642 | 1.0298 | 0.076* |
| C24 | 0.0448 (3) | 0.6912 (3) | 1.00012 (17) | 0.0527 (7) |
| H24A | 0.0464 | 0.6071 | 1.0443 | 0.063* |
| C25 | 0.1743 (3) | 0.7277 (2) | 0.93981 (16) | 0.0426 (6) |
| C26 | 0.4107 (3) | 0.7330 (2) | 0.86766 (15) | 0.0388 (6) |
| C27 | 0.5734 (3) | 0.6772 (2) | 0.85723 (16) | 0.0402 (6) |
| C28 | 0.8177 (3) | 0.7041 (3) | 0.77442 (18) | 0.0553 (7) |
| H28A | 0.8282 | 0.6102 | 0.7638 | 0.066* |
| H28B | 0.8701 | 0.7019 | 0.8287 | 0.066* |
| C29 | 0.8863 (3) | 0.8028 (3) | 0.6932 (2) | 0.0802 (10) |
| H29A | 0.9940 | 0.7727 | 0.6825 | 0.120* |
| H29B | 0.8759 | 0.8952 | 0.7048 | 0.120* |
| H29C | 0.8335 | 0.8044 | 0.6400 | 0.120* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0488 (13) | 0.0511 (13) | 0.0378 (11) | 0.0003 (10) | −0.0143 (10) | −0.0049 (10) |
| O1 | 0.0723 (14) | 0.0525 (12) | 0.0859 (14) | 0.0166 (10) | −0.0364 (11) | −0.0322 (10) |
| C1 | 0.0510 (16) | 0.0538 (17) | 0.0462 (15) | 0.0093 (13) | −0.0099 (13) | 0.0071 (13) |
| N2 | 0.0469 (13) | 0.0359 (11) | 0.0461 (12) | 0.0014 (10) | −0.0069 (10) | 0.0039 (9) |
| O2 | 0.0705 (13) | 0.0795 (14) | 0.0662 (13) | 0.0077 (11) | −0.0280 (11) | −0.0355 (11) |
| C2 | 0.067 (2) | 0.0520 (17) | 0.0555 (18) | 0.0173 (15) | −0.0037 (15) | −0.0005 (15) |
| O3 | 0.0440 (10) | 0.0501 (10) | 0.0468 (10) | 0.0059 (8) | −0.0009 (8) | 0.0007 (8) |
| C3 | 0.076 (2) | 0.0451 (16) | 0.0604 (18) | 0.0116 (15) | −0.0099 (16) | −0.0114 (13) |
| O4 | 0.0580 (12) | 0.0507 (11) | 0.0675 (12) | 0.0134 (9) | −0.0037 (10) | 0.0132 (10) |
| C4 | 0.0693 (19) | 0.0456 (16) | 0.0508 (16) | 0.0055 (14) | −0.0184 (14) | −0.0086 (13) |
| C5 | 0.0446 (14) | 0.0399 (14) | 0.0320 (13) | 0.0002 (11) | −0.0026 (11) | 0.0008 (11) |
| C6 | 0.0468 (15) | 0.0456 (15) | 0.0356 (14) | −0.0011 (12) | −0.0037 (11) | 0.0032 (11) |
| C7 | 0.0397 (13) | 0.0400 (13) | 0.0317 (12) | 0.0015 (11) | −0.0049 (11) | −0.0030 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8 | 0.0442 (14) | 0.0384 (13) | 0.0361 (13) | 0.0003 (11) | −0.0100 (11) | −0.0040 (11) |
| C9 | 0.0489 (16) | 0.0536 (16) | 0.0442 (15) | −0.0016 (13) | −0.0104 (13) | −0.0088 (13) |
| C10 | 0.098 (3) | 0.060 (2) | 0.138 (3) | 0.0167 (19) | −0.046 (2) | −0.050 (2) |
| C11 | 0.123 (4) | 0.135 (4) | 0.200 (5) | 0.021 (3) | −0.042 (4) | −0.082 (4) |
| C12 | 0.0407 (14) | 0.0348 (13) | 0.0353 (13) | 0.0057 (11) | −0.0102 (11) | −0.0073 (10) |
| C13 | 0.0398 (14) | 0.0362 (13) | 0.0390 (14) | 0.0037 (11) | −0.0102 (11) | −0.0026 (11) |
| C14 | 0.0574 (17) | 0.0537 (17) | 0.0561 (17) | −0.0123 (14) | 0.0014 (14) | −0.0055 (14) |
| C15 | 0.069 (2) | 0.0589 (19) | 0.097 (3) | −0.0266 (17) | 0.0001 (19) | −0.0023 (18) |
| C16 | 0.074 (2) | 0.0601 (19) | 0.085 (2) | −0.0156 (17) | −0.0234 (19) | −0.0172 (18) |
| C17 | 0.074 (2) | 0.0598 (18) | 0.0549 (17) | −0.0035 (16) | −0.0240 (16) | −0.0178 (15) |
| C18 | 0.0524 (16) | 0.0494 (15) | 0.0412 (15) | −0.0021 (13) | −0.0084 (12) | −0.0090 (12) |
| C19 | 0.0405 (14) | 0.0338 (13) | 0.0363 (13) | 0.0004 (11) | −0.0105 (11) | −0.0036 (10) |
| C20 | 0.0413 (14) | 0.0376 (13) | 0.0379 (13) | −0.0032 (11) | −0.0069 (11) | −0.0042 (10) |
| C21 | 0.0479 (16) | 0.0469 (15) | 0.0527 (16) | 0.0056 (13) | −0.0080 (13) | −0.0034 (12) |
| C22 | 0.0407 (16) | 0.0669 (19) | 0.070 (2) | 0.0074 (14) | 0.0033 (15) | −0.0103 (16) |
| C23 | 0.0467 (17) | 0.071 (2) | 0.0633 (19) | −0.0068 (15) | 0.0103 (14) | −0.0054 (16) |
| C24 | 0.0485 (16) | 0.0557 (17) | 0.0483 (16) | −0.0087 (14) | −0.0017 (13) | −0.0004 (13) |
| C25 | 0.0413 (14) | 0.0420 (14) | 0.0442 (14) | −0.0024 (12) | −0.0085 (12) | −0.0080 (11) |
| C26 | 0.0444 (14) | 0.0343 (13) | 0.0343 (13) | 0.0017 (11) | −0.0066 (11) | −0.0023 (10) |
| C27 | 0.0447 (15) | 0.0333 (13) | 0.0382 (13) | 0.0012 (11) | −0.0054 (12) | −0.0014 (11) |
| C28 | 0.0501 (17) | 0.0554 (17) | 0.0543 (17) | 0.0047 (13) | 0.0025 (13) | −0.0084 (13) |
| C29 | 0.065 (2) | 0.082 (2) | 0.088 (2) | −0.0115 (18) | 0.0150 (18) | −0.0142 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C6 | 1.361 (3) | C12—C13 | 1.524 (3) |
| N1—C8 | 1.375 (3) | C12—H12A | 0.9800 |
| N1—H1A | 0.8600 | C13—C14 | 1.374 (3) |
| O1—C9 | 1.320 (3) | C13—C18 | 1.392 (3) |
| O1—C10 | 1.455 (3) | C14—C15 | 1.382 (4) |
| C1—C2 | 1.354 (4) | C14—H14A | 0.9300 |
| C1—C6 | 1.393 (3) | C15—C16 | 1.370 (4) |
| C1—H1B | 0.9300 | C15—H15A | 0.9300 |
| N2—C25 | 1.364 (3) | C16—C17 | 1.362 (4) |
| N2—C26 | 1.368 (3) | C16—H16A | 0.9300 |
| N2—H2A | 0.8600 | C17—C18 | 1.394 (3) |
| O2—C9 | 1.219 (3) | C17—H17A | 0.9300 |
| C2—C3 | 1.394 (4) | C18—H18A | 0.9300 |
| C2—H2B | 0.9300 | C19—C26 | 1.382 (3) |
| O3—C27 | 1.331 (3) | C19—C20 | 1.442 (3) |
| O3—C28 | 1.453 (3) | C20—C25 | 1.410 (3) |
| C3—C4 | 1.372 (3) | C20—C21 | 1.415 (3) |
| C3—H3A | 0.9300 | C21—C22 | 1.363 (3) |
| O4—C27 | 1.207 (3) | C21—H21A | 0.9300 |
| C4—C5 | 1.402 (3) | C22—C23 | 1.397 (4) |
| C4—H4A | 0.9300 | C22—H22A | 0.9300 |
| C5—C6 | 1.413 (3) | C23—C24 | 1.371 (4) |
| C5—C7 | 1.440 (3) | C23—H23A | 0.9300 |

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|---|---|---|---|
| C7—C8 | 1.375 (3) | C24—C25 | 1.397 (3) |
| C7—C12 | 1.520 (3) | C24—H24A | 0.9300 |
| C8—C9 | 1.460 (3) | C26—C27 | 1.467 (3) |
| C10—C11 | 1.379 (4) | C28—C29 | 1.486 (4) |
| C10—H10A | 0.9700 | C28—H28A | 0.9700 |
| C10—H10B | 0.9700 | C28—H28B | 0.9700 |
| C11—H11A | 0.9600 | C29—H29A | 0.9600 |
| C11—H11B | 0.9600 | C29—H29B | 0.9600 |
| C11—H11C | 0.9600 | C29—H29C | 0.9600 |
| C12—C19 | 1.522 (3) | | |

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| C6—N1—C8 | 108.91 (19) | C18—C13—C12 | 122.3 (2) |
| C6—N1—H1A | 125.5 | C13—C14—C15 | 120.6 (3) |
| C8—N1—H1A | 125.5 | C13—C14—H14A | 119.7 |
| C9—O1—C10 | 117.3 (2) | C15—C14—H14A | 119.7 |
| C2—C1—C6 | 117.7 (3) | C16—C15—C14 | 120.8 (3) |
| C2—C1—H1B | 121.2 | C16—C15—H15A | 119.6 |
| C6—C1—H1B | 121.2 | C14—C15—H15A | 119.6 |
| C25—N2—C26 | 109.28 (19) | C17—C16—C15 | 119.7 (3) |
| C25—N2—H2A | 125.4 | C17—C16—H16A | 120.2 |
| C26—N2—H2A | 125.4 | C15—C16—H16A | 120.2 |
| C1—C2—C3 | 121.4 (3) | C16—C17—C18 | 120.0 (3) |
| C1—C2—H2B | 119.3 | C16—C17—H17A | 120.0 |
| C3—C2—H2B | 119.3 | C18—C17—H17A | 120.0 |
| C27—O3—C28 | 116.10 (19) | C13—C18—C17 | 120.6 (3) |
| C4—C3—C2 | 121.6 (3) | C13—C18—H18A | 119.7 |
| C4—C3—H3A | 119.2 | C17—C18—H18A | 119.7 |
| C2—C3—H3A | 119.2 | C26—C19—C20 | 105.12 (19) |
| C3—C4—C5 | 119.0 (2) | C26—C19—C12 | 125.5 (2) |
| C3—C4—H4A | 120.5 | C20—C19—C12 | 129.27 (19) |
| C5—C4—H4A | 120.5 | C25—C20—C21 | 117.3 (2) |
| C4—C5—C6 | 117.8 (2) | C25—C20—C19 | 107.5 (2) |
| C4—C5—C7 | 135.3 (2) | C21—C20—C19 | 135.2 (2) |
| C6—C5—C7 | 106.9 (2) | C22—C21—C20 | 119.4 (2) |
| N1—C6—C1 | 129.3 (2) | C22—C21—H21A | 120.3 |
| N1—C6—C5 | 108.1 (2) | C20—C21—H21A | 120.3 |
| C1—C6—C5 | 122.6 (2) | C21—C22—C23 | 121.8 (3) |
| C8—C7—C5 | 105.8 (2) | C21—C22—H22A | 119.1 |
| C8—C7—C12 | 126.1 (2) | C23—C22—H22A | 119.1 |
| C5—C7—C12 | 128.1 (2) | C24—C23—C22 | 121.3 (3) |
| N1—C8—C7 | 110.2 (2) | C24—C23—H23A | 119.3 |
| N1—C8—C9 | 116.0 (2) | C22—C23—H23A | 119.3 |
| C7—C8—C9 | 133.8 (2) | C23—C24—C25 | 116.9 (2) |
| O2—C9—O1 | 122.9 (3) | C23—C24—H24A | 121.5 |
| O2—C9—C8 | 122.9 (2) | C25—C24—H24A | 121.5 |
| O1—C9—C8 | 114.2 (2) | N2—C25—C24 | 129.0 (2) |
| C11—C10—O1 | 113.3 (3) | N2—C25—C20 | 107.6 (2) |
| C11—C10—H10A | 108.9 | C24—C25—C20 | 123.3 (2) |

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| O1—C10—H10A | 108.9 | N2—C26—C19 | 110.4 (2) |
| C11—C10—H10B | 108.9 | N2—C26—C27 | 117.0 (2) |
| O1—C10—H10B | 108.9 | C19—C26—C27 | 132.5 (2) |
| H10A—C10—H10B | 107.7 | O4—C27—O3 | 122.8 (2) |
| C10—C11—H11A | 109.5 | O4—C27—C26 | 123.4 (2) |
| C10—C11—H11B | 109.5 | O3—C27—C26 | 113.7 (2) |
| H11A—C11—H11B | 109.5 | O3—C28—C29 | 107.3 (2) |
| C10—C11—H11C | 109.5 | O3—C28—H28A | 110.2 |
| H11A—C11—H11C | 109.5 | C29—C28—H28A | 110.2 |
| H11B—C11—H11C | 109.5 | O3—C28—H28B | 110.2 |
| C7—C12—C19 | 113.36 (18) | C29—C28—H28B | 110.2 |
| C7—C12—C13 | 112.51 (18) | H28A—C28—H28B | 108.5 |
| C19—C12—C13 | 112.78 (18) | C28—C29—H29A | 109.5 |
| C7—C12—H12A | 105.8 | C28—C29—H29B | 109.5 |
| C19—C12—H12A | 105.8 | H29A—C29—H29B | 109.5 |
| C13—C12—H12A | 105.8 | C28—C29—H29C | 109.5 |
| C14—C13—C18 | 118.2 (2) | H29A—C29—H29C | 109.5 |
| C14—C13—C12 | 119.4 (2) | H29B—C29—H29C | 109.5 |

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| C6—C1—C2—C3 | −1.2 (4) | C13—C14—C15—C16 | 0.5 (5) |
| C1—C2—C3—C4 | 0.5 (4) | C14—C15—C16—C17 | 1.3 (5) |
| C2—C3—C4—C5 | 0.4 (4) | C15—C16—C17—C18 | −1.1 (4) |
| C3—C4—C5—C6 | −0.6 (4) | C14—C13—C18—C17 | 2.8 (4) |
| C3—C4—C5—C7 | −179.5 (3) | C12—C13—C18—C17 | −175.3 (2) |
| C8—N1—C6—C1 | 179.6 (2) | C16—C17—C18—C13 | −1.0 (4) |
| C8—N1—C6—C5 | −0.8 (3) | C7—C12—C19—C26 | 152.1 (2) |
| C2—C1—C6—N1 | −179.6 (2) | C13—C12—C19—C26 | −78.6 (3) |
| C2—C1—C6—C5 | 0.9 (4) | C7—C12—C19—C20 | −32.3 (3) |
| C4—C5—C6—N1 | −179.6 (2) | C13—C12—C19—C20 | 97.0 (3) |
| C7—C5—C6—N1 | −0.5 (3) | C26—C19—C20—C25 | −0.8 (2) |
| C4—C5—C6—C1 | 0.0 (4) | C12—C19—C20—C25 | −177.0 (2) |
| C7—C5—C6—C1 | 179.1 (2) | C26—C19—C20—C21 | 176.8 (3) |
| C4—C5—C7—C8 | −179.5 (3) | C12—C19—C20—C21 | 0.5 (4) |
| C6—C5—C7—C8 | 1.6 (2) | C25—C20—C21—C22 | −0.5 (4) |
| C4—C5—C7—C12 | 0.6 (4) | C19—C20—C21—C22 | −177.8 (3) |
| C6—C5—C7—C12 | −178.3 (2) | C20—C21—C22—C23 | −0.2 (4) |
| C6—N1—C8—C7 | 1.9 (3) | C21—C22—C23—C24 | 0.7 (5) |
| C6—N1—C8—C9 | −177.3 (2) | C22—C23—C24—C25 | −0.5 (4) |
| C5—C7—C8—N1 | −2.2 (3) | C26—N2—C25—C24 | −178.0 (2) |
| C12—C7—C8—N1 | 177.8 (2) | C26—N2—C25—C20 | 0.3 (3) |
| C5—C7—C8—C9 | 176.9 (3) | C23—C24—C25—N2 | 177.9 (2) |
| C12—C7—C8—C9 | −3.2 (4) | C23—C24—C25—C20 | −0.2 (4) |
| C10—O1—C9—O2 | 2.7 (4) | C21—C20—C25—N2 | −177.8 (2) |
| C10—O1—C9—C8 | −176.1 (2) | C19—C20—C25—N2 | 0.3 (3) |
| N1—C8—C9—O2 | −4.4 (4) | C21—C20—C25—C24 | 0.7 (4) |
| C7—C8—C9—O2 | 176.7 (3) | C19—C20—C25—C24 | 178.7 (2) |
| N1—C8—C9—O1 | 174.4 (2) | C25—N2—C26—C19 | −0.8 (3) |
| C7—C8—C9—O1 | −4.6 (4) | C25—N2—C26—C27 | 175.8 (2) |

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|---|---|---|---|
| C9—O1—C10—C11 | 162.2 (4) | C20—C19—C26—N2 | 1.0 (3) |
| C8—C7—C12—C19 | −76.6 (3) | C12—C19—C26—N2 | 177.4 (2) |
| C5—C7—C12—C19 | 103.4 (3) | C20—C19—C26—C27 | −175.0 (2) |
| C8—C7—C12—C13 | 154.0 (2) | C12—C19—C26—C27 | 1.5 (4) |
| C5—C7—C12—C13 | −26.1 (3) | C28—O3—C27—O4 | 0.4 (3) |
| C7—C12—C13—C14 | −74.5 (3) | C28—O3—C27—C26 | 179.4 (2) |
| C19—C12—C13—C14 | 155.7 (2) | N2—C26—C27—O4 | −3.8 (4) |
| C7—C12—C13—C18 | 103.5 (2) | C19—C26—C27—O4 | 171.9 (3) |
| C19—C12—C13—C18 | −26.2 (3) | N2—C26—C27—O3 | 177.19 (19) |
| C18—C13—C14—C15 | −2.5 (4) | C19—C26—C27—O3 | −7.1 (4) |
| C12—C13—C14—C15 | 175.6 (2) | C27—O3—C28—C29 | 179.4 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···O2$^{i}$ | 0.86 | 2.21 | 2.955 (3) | 144 |
| N2—H2*A*···O4$^{ii}$ | 0.86 | 2.07 | 2.880 (3) | 157 |

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+2.