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# *N*-(*n*-Decyl)-4-nitroaniline

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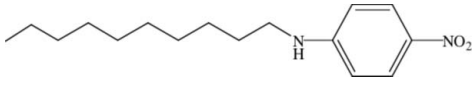
Key indicators: single-crystal X-ray study; $T$ = 140 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.054; $wR$ factor = 0.140; data-to-parameter ratio = 11.1.

*N*-(*n*-Decyl)-4-nitroaniline, $C_{16}H_{26}N_2O_2$, crystallizes with two essentially planar molecules in the asymmetric unit. The decyl chains are fully extended in an anti conformation. The molecules pack in planar layers, within which molecules are linked into chains by two approximately linear N—H···O hydrogen bonds between the amine N atom and one O atom of the nitro group of an adjacent molecule. These molecular chains propagate *via* interleaving of the decyl chains to form the two dimensional sheets. The sheets are associated exclusively *via* non-bonded contacts. The structure has features in common with those of other *N*-alkyl-4-nitroanilines, but also subtle differences in packing.

## Related literature

For the structures of other *N*-alkyl-4-nitroanilines, see: Panunto *et al.* (1987); Gangopadhyay *et al.* (1999); Teng *et al.* (2006).




## Experimental

### *Crystal data*

$C_{16}H_{26}N_2O_2$
$M_r$ = 278.39
Monoclinic, $P2_1/c$
$a$ = 13.291 (6) Å
$b$ = 29.117 (12) Å
$c$ = 8.279 (4) Å
$\beta$ = 91.457 (7)°
$V$ = 3203 (2) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 140 (2) K
0.40 × 0.35 × 0.28 mm

### *Data collection*

Bruker SMART 6000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)
$T_{min}$ = 0.94, $T_{max}$ = 0.98
28685 measured reflections
6310 independent reflections
4359 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.140
$S$ = 1.03
6310 reflections
569 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2*N*···O3$^{i}$ | 0.873 (18) | 2.234 (19) | 3.101 (2) | 171.7 (16) |
| N4—H4*N*···O1$^{ii}$ | 0.869 (19) | 2.265 (19) | 3.121 (2) | 168.3 (17) |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenberg & Berndt, 2006) and *Crystal Maker* (Crystal Maker, 2006); software used to prepare material for publication: *SHELTXL* and local programs.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2595).

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# supporting information

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## *N*-(*n*-Decyl)-4-nitroaniline

**Matthew M. Yonkey, Christopher P. Walczak, Philip J. Squattrito, Dillip K. Mohanty and Kristin Kirschbaum**

### S1. Comment

Recently we have reported the preparation and characteristics of highly solvent-resistant polyamines containing two aromatic nitro groups in the polymer repeat units (Teng *et al.*, 2006). These polymers were prepared by the nucleophilic displacement of fluorine atoms from 1,5-difluoro-2,4-dinitrobenzene, using aliphatic diamines. The aforementioned solvent resistance of these polymers is a direct consequence of inter- and intrachain hydrogen bonding interactions between the secondary amine and nitro groups in the polymer repeat units. In order to further understand the importance of these interactions, we have undertaken the preparation of polyamines containing one aromatic nitro group in contrast to two such groups. The syntheses and properties of these polymers using both 2,6-difluoronitrobenzene and isomeric 2,4-difluoronitrobenzene, and a series of aliphatic diamines, will be reported elsewhere. In the course of these studies, it was necessary to prepare model compounds prior to the preparation of the polymers. This was accomplished by the reactions of either mono- or difluoro-substituted nitrobenzene with a homologous series of n-alkyl amines. The X-ray crystal structures of *N*-methyl-4-nitroaniline (Panunto *et al.*, 1987) and *N*-alkyl-4-nitroanilines with alkyl groups ranging from n-propyl to n-pentyl (Gangopadhyay *et al.*, 1999) have been reported. The *N*-decyl-4-nitroaniline (I) reported herein represents the longest chain *N*-alkyl-4-nitroaniline derivative thus far characterized by single-crystal X-ray methods.

The asymmetric unit of the title compound (I) contains two independent molecules which do not differ significantly in conformation (Figure 1). Both molecules are essentially planar and have the decyl tails in the characteristic zigzag anti conformations. The molecules are packed into layers that are parallel to and at the same intervals as the $(20\bar{2})$ lattice planes (Figure 2). Within each of these layers, the molecules are linked by two N—H···O hydrogen bonds between the amine N atom of one molecule and one of the nitro O atoms of another (Figure 3). As a consequence of these interactions, the N—O bond that participates in the hydrogen bond is about 0.01 Å longer than the other N—O bond on each nitro group. Also, the relative shortness of the H···O interactions and the linearity of the N—H···O bonds (Table 1) clearly demonstrate that these are classic single-acceptor hydrogen bonds, unlike the three-center interactions found in many nitroaniline derivatives including *N*-methyl-4-nitroaniline (Panunto *et al.*, 1987). The hydrogen bonding links the molecules into chains that run along the $10\bar{1}$ direction with the decyl chains on adjacent molecules oriented up and down, with an angle between the chains of about 77°. To complete the two-dimensional layers, these molecular chains then stack along the *b* axis with the decyl tails interleaved in parallel fashion so as to maximize favorable non-bonded contacts.

The series of *N*-alkyl-4-nitroanilines where alkyl = propyl-octyl has been examined for potential optical second harmonic generation (SHG) behavior (Gangopadhyay *et al.*, 1999). SHG effects require the absence of a center of inversion, although this condition alone does not guarantee activity. Physical measurements of this series showed that only the butyl compound is active, and single-crystal structure analyses of the propyl, butyl and pentyl derivatives

confirmed that only the butyl crystallizes in an noncentrosymmetric space group. The authors report that poor crystal quality prevented structure determinations of the longer chain derivatives, however, our successful crystallization of the decyl compound demonstrates that at least some of these may be characterized. The N—H···O hydrogen bonding in (I) is very similar to that found in the shorter chain analogs in terms of involving only one of the nitro O atoms. The packing in (I) is subtly different. In the propyl and pentyl compounds, the molecules stack in one direction in identical orientation so that both the rings and chains are parallel and in close contact. By contrast, in (I), the layers are staggered so that rings in adjacent layers are not directly over one another but rather have an alkyl chain in between. This indicates that the fully interleaved packing of the decyl chains within the layer, which is unique to (I), is more important than the $\pi$-$\pi$ interactions between the rings in determining the overall packing.

## S2. Experimental

Anhydrous potassium carbonate (2.0811 g, 0.015 mol) and a solution of 4-nitrofluorobenzene (0.9993 g, 0.007 mol) in 8 ml of dimethyacetamide (DMAC) were combined in a three-necked 100 ml round-bottomed flask, fitted with a nitrogen inlet, a thermometer, a magnetic stirring bar, and a Dean-Stark trap fitted with a condenser. To the clear yellow solution, n-decylamine (1.1767 g, 0.0075 mol) dissolved in DMAC (5 ml) was added with stirring. Additional DMAC (8 ml) was used to wash the transfer container and this was added to the reaction mixture, followed by the addition of 20 ml of toluene. The temperature of the reaction mixture was raised to 403 K, and the reaction was allowed to continue at this temperature for one hour. Water, the by-product of the reaction, was removed *via* azeotropic distillation with toluene. After the removal of water, toluene was removed *via* the Dean-Stark trap, and the temperature of the reaction mixture was allowed to rise to 433 K. The reaction was allowed to continue at this temperature for three hours, after which it was allowed to cool to room temperature and then diluted with 20 ml of dichloromethane. The resulting heterogeneous mixture was then filtered through celite at reduced pressure, and the solvents from the filtrate were removed under high vacuum to yield a bright orange liquid residue. This crude product was dissolved in trichloromethane (15 ml), transferred to a separatory funnel, and washed repeatedly with deionized water. The organic layer was collected, dried over anhydrous magnesium sulfate, filtered, and the filtrate was evaporated using a rotary evaporator to yield a bright yellow solid. Crystals suitable for X-ray diffraction were obtained by recrystallization from hexane. Yield 56%, m.p. 332–333 K. IR (KBr, $\nu > 1400$ cm$^{-1}$): 3353, 3064, 2952, 2925, 2850, 1602, 1541, 1475, 1466. $^1$H NMR [400 MHz, $\delta$ p.p.m., $CDCl_3$], 8.12 (m, 2H), 6.53 (m, 2H), 4.44 (s, 1H), 1.65 (m, 2H), 1.30 (m, 14H), 0.92 (t, 3H). $^{13}$C NMR [$\delta$, $CDCl_3$], 153.63, 137.74, 129.69.111.11, 43.66, 32.09, 29.75, 29.54, 29.51, 29.36, 27.21, 22.89, 14.33. MS (*M*/*Z*) (% base peak), 278 (11.6), 151 (100), 105 (19.7).

## S3. Refinement

Upon evaluation of systematic absences and weaknesses, the space group was determined to be $P2_1/c$ with an additional pseudo-a glide perpendicular to the *b* axis. A partial structure solution was obtained by direct methods and revealed two crystallographically independent molecules in the asymmetric unit. The remaining non-hydrogen atoms were located with difference Fourier techniques and refined with anisotropic atomic displacement parameters. All hydrogen atoms could be located in the difference Fourier maps and refined isotropically.

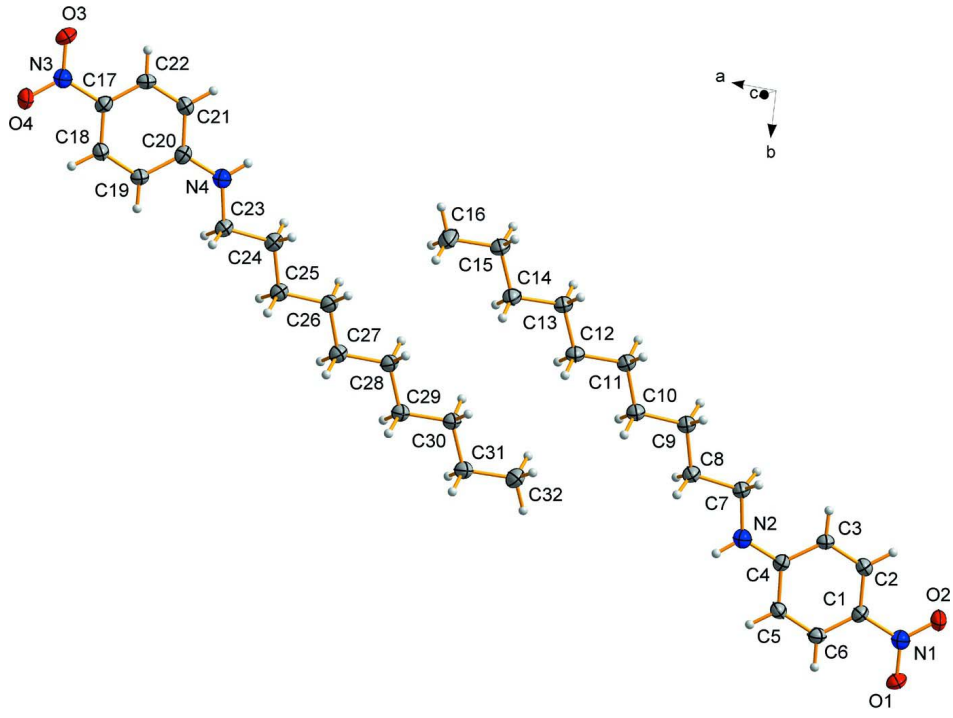


**Figure 1**

The asymmetric unit of (I) showing two independent molecules with atom labels and 50% probability displacement ellipsoids for non-hydrogen atoms.

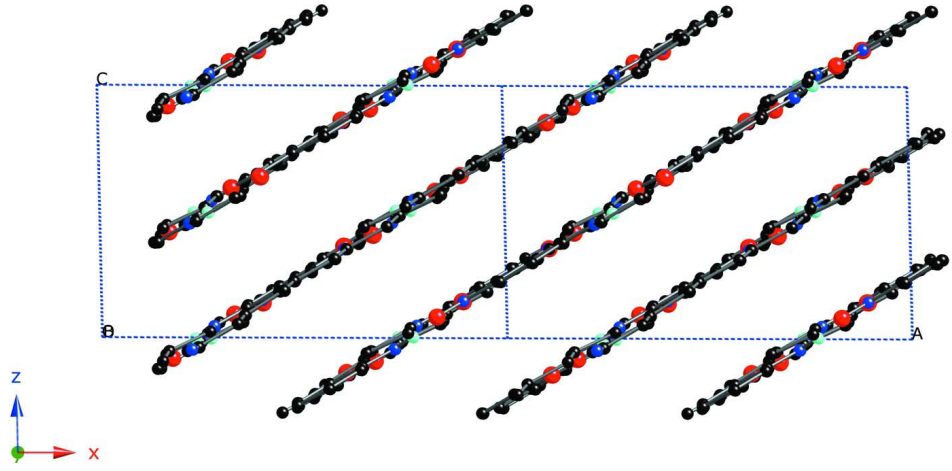


**Figure 2**

Packing diagram of (I) as viewed along the *b* axis. Note the planar layers parallel to (20$\bar{2}$).

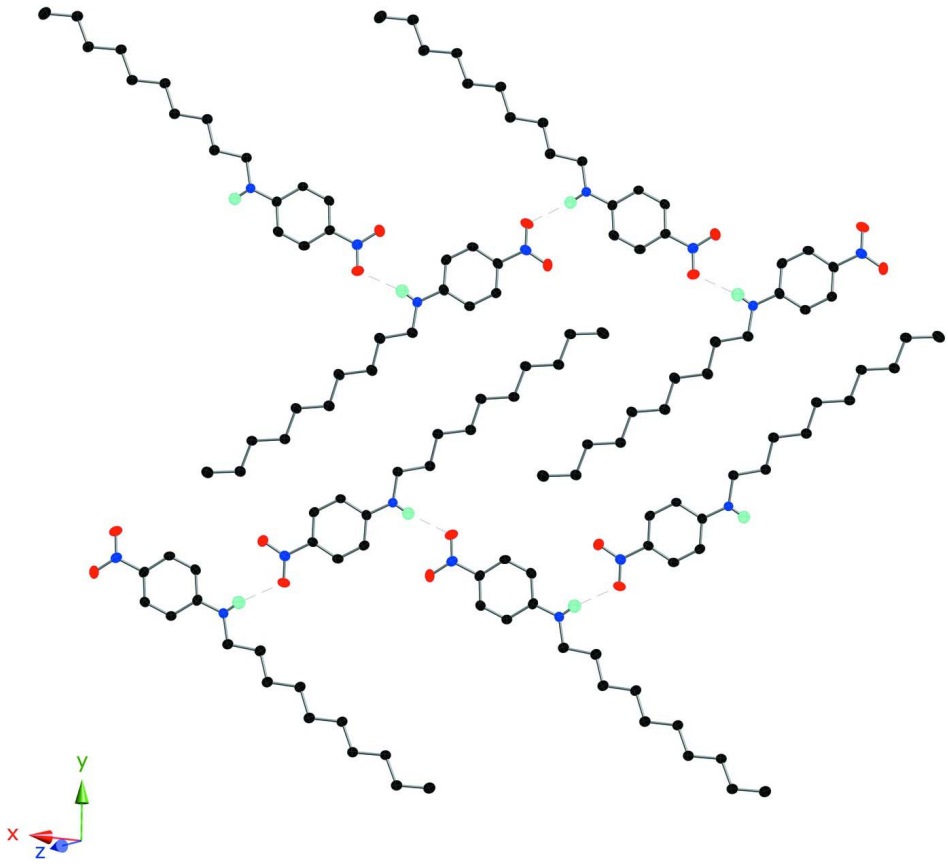


**Figure 3**

View onto a single layer of molecules with hydrogen bonds shown as dashed lines.

### *N*-(n-Decyl)-4-nitroaniline

*Crystal data*

$C_{16}H_{26}N_2O_2$
$M_r = 278.39$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.291\ (6)$ Å
$b = 29.117\ (12)$ Å
$c = 8.279\ (4)$ Å
$\beta = 91.457\ (7)°$
$V = 3203\ (2)$ Å$^3$
$Z = 8$

$F(000) = 1216$
$D_x = 1.155$ Mg m$^{-3}$
Melting point = 332–333 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 950 reflections
$\theta = 2.6–25.8°$
$\mu = 0.08$ mm$^{-1}$
$T = 140$ K
Pyramidal, yellow
0.40 × 0.35 × 0.28 mm

*Data collection*

Bruker SMART 6000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)
$T_{min} = 0.94$, $T_{max} = 0.98$

28685 measured reflections
6310 independent reflections
4359 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 26.1°$, $\theta_{min} = 1.5°$
$h = -16\rightarrow16$
$k = -32\rightarrow35$
$l = -10\rightarrow10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.140$
$S = 1.03$
6310 reflections
569 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0796P)^2 + 0.231P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | −0.60160 (9) | 0.67377 (4) | −0.86284 (15) | 0.0412 (3) |
| O2 | −0.67258 (9) | 0.60818 (4) | −0.90706 (16) | 0.0453 (4) |
| O3 | 1.10779 (9) | 0.09460 (4) | 0.85523 (15) | 0.0406 (3) |
| O4 | 1.17105 (10) | 0.16069 (4) | 0.91774 (17) | 0.0484 (4) |
| N1 | −0.60284 (10) | 0.63137 (5) | −0.85126 (16) | 0.0302 (3) |
| N2 | −0.27827 (10) | 0.54358 (5) | −0.54454 (16) | 0.0295 (3) |
| N3 | 1.10435 (10) | 0.13712 (5) | 0.85407 (16) | 0.0320 (3) |
| N4 | 0.77760 (11) | 0.22343 (5) | 0.54846 (17) | 0.0312 (3) |
| C1 | −0.51989 (11) | 0.60874 (6) | −0.77006 (18) | 0.0256 (4) |
| C2 | −0.51950 (12) | 0.56106 (6) | −0.75980 (19) | 0.0276 (4) |
| C3 | −0.44015 (12) | 0.53898 (6) | −0.68323 (19) | 0.0265 (4) |
| C4 | −0.35872 (11) | 0.56424 (5) | −0.61648 (18) | 0.0246 (4) |
| C5 | −0.36234 (12) | 0.61265 (6) | −0.62760 (19) | 0.0274 (4) |
| C6 | −0.44100 (12) | 0.63459 (6) | −0.70329 (19) | 0.0273 (4) |
| C7 | −0.26796 (13) | 0.49425 (6) | −0.5227 (2) | 0.0283 (4) |
| C8 | −0.16565 (13) | 0.48362 (6) | −0.4446 (2) | 0.0300 (4) |
| C9 | −0.14962 (13) | 0.43315 (6) | −0.4047 (2) | 0.0295 (4) |
| C10 | −0.04873 (13) | 0.42560 (6) | −0.3167 (2) | 0.0317 (4) |
| C11 | −0.02331 (13) | 0.37619 (6) | −0.2754 (2) | 0.0292 (4) |
| C12 | 0.07786 (13) | 0.37202 (6) | −0.1859 (2) | 0.0296 (4) |
| C13 | 0.10972 (13) | 0.32300 (6) | −0.1464 (2) | 0.0290 (4) |
| C14 | 0.20989 (13) | 0.31956 (6) | −0.0544 (2) | 0.0310 (4) |
| C15 | 0.24269 (14) | 0.27065 (6) | −0.0180 (2) | 0.0354 (4) |
| C16 | 0.34653 (16) | 0.26753 (8) | 0.0615 (3) | 0.0499 (6) |
| C17 | 1.01992 (12) | 0.15945 (6) | 0.77582 (18) | 0.0271 (4) |

| | | | | |
|---|---|---|---|---|
| C18 | 1.01434 (12) | 0.20703 (6) | 0.77712 (19) | 0.0289 (4) |
| C19 | 0.93436 (12) | 0.22876 (6) | 0.70194 (19) | 0.0289 (4) |
| C20 | 0.85730 (11) | 0.20307 (6) | 0.62333 (18) | 0.0264 (4) |
| C21 | 0.86534 (12) | 0.15453 (6) | 0.62463 (19) | 0.0283 (4) |
| C22 | 0.94510 (12) | 0.13317 (6) | 0.69935 (19) | 0.0275 (4) |
| C23 | 0.76177 (13) | 0.27270 (6) | 0.5393 (2) | 0.0295 (4) |
| C24 | 0.66360 (13) | 0.28244 (6) | 0.4467 (2) | 0.0312 (4) |
| C25 | 0.64562 (13) | 0.33306 (6) | 0.4121 (2) | 0.0297 (4) |
| C26 | 0.54838 (13) | 0.34037 (6) | 0.3138 (2) | 0.0310 (4) |
| C27 | 0.52284 (13) | 0.39032 (6) | 0.2770 (2) | 0.0307 (4) |
| C28 | 0.42175 (13) | 0.39529 (6) | 0.1880 (2) | 0.0294 (4) |
| C29 | 0.39176 (13) | 0.44438 (6) | 0.1484 (2) | 0.0286 (4) |
| C30 | 0.29031 (13) | 0.44809 (6) | 0.0593 (2) | 0.0292 (4) |
| C31 | 0.26026 (14) | 0.49683 (6) | 0.0147 (2) | 0.0323 (4) |
| C32 | 0.15712 (16) | 0.49976 (7) | −0.0678 (3) | 0.0420 (5) |
| H2N | −0.2325 (13) | 0.5606 (6) | −0.496 (2) | 0.032 (5)* |
| H2 | −0.5723 (13) | 0.5437 (6) | −0.8041 (19) | 0.027 (4)* |
| H3 | −0.4412 (13) | 0.5071 (7) | −0.679 (2) | 0.035 (5)* |
| H4N | 0.7319 (14) | 0.2059 (7) | 0.504 (2) | 0.037 (5)* |
| H5 | −0.3087 (13) | 0.6296 (6) | −0.5867 (19) | 0.030 (5)* |
| H6 | −0.4425 (13) | 0.6666 (7) | −0.7100 (19) | 0.031 (5)* |
| H7B | −0.3219 (13) | 0.4826 (6) | −0.453 (2) | 0.031 (4)* |
| H7A | −0.2746 (12) | 0.4790 (6) | −0.630 (2) | 0.027 (4)* |
| H8B | −0.1144 (13) | 0.4933 (6) | −0.519 (2) | 0.034 (5)* |
| H8A | −0.1593 (13) | 0.5016 (6) | −0.347 (2) | 0.039 (5)* |
| H9B | −0.2026 (13) | 0.4221 (6) | −0.334 (2) | 0.035 (5)* |
| H9A | −0.1528 (12) | 0.4146 (6) | −0.507 (2) | 0.033 (5)* |
| H10B | 0.0036 (13) | 0.4388 (6) | −0.385 (2) | 0.034 (5)* |
| H10A | −0.0496 (13) | 0.4451 (6) | −0.220 (2) | 0.038 (5)* |
| H11B | −0.0757 (13) | 0.3637 (6) | −0.205 (2) | 0.030 (4)* |
| H11A | −0.0216 (12) | 0.3566 (6) | −0.376 (2) | 0.033 (5)* |
| H12B | 0.1303 (13) | 0.3863 (6) | −0.251 (2) | 0.035 (5)* |
| H12A | 0.0726 (12) | 0.3896 (6) | −0.083 (2) | 0.031 (4)* |
| H13B | 0.0577 (13) | 0.3094 (6) | −0.083 (2) | 0.033 (5)* |
| H13A | 0.1142 (12) | 0.3066 (6) | −0.246 (2) | 0.036 (5)* |
| H14B | 0.2036 (12) | 0.3372 (6) | 0.047 (2) | 0.031 (4)* |
| H14A | 0.2602 (14) | 0.3339 (6) | −0.121 (2) | 0.044 (5)* |
| H15B | 0.2401 (15) | 0.2530 (7) | −0.116 (3) | 0.054 (6)* |
| H15A | 0.1955 (14) | 0.2567 (6) | 0.052 (2) | 0.039 (5)* |
| H16C | 0.3993 (16) | 0.2807 (8) | −0.007 (3) | 0.064 (7)* |
| H16B | 0.3464 (18) | 0.2856 (9) | 0.166 (3) | 0.079 (8)* |
| H16A | 0.3665 (16) | 0.2361 (9) | 0.085 (3) | 0.063 (7)* |
| H18 | 1.0663 (14) | 0.2236 (6) | 0.829 (2) | 0.037 (5)* |
| H19 | 0.9313 (13) | 0.2604 (7) | 0.705 (2) | 0.038 (5)* |
| H21 | 0.8145 (13) | 0.1366 (6) | 0.578 (2) | 0.036 (5)* |
| H22 | 0.9501 (12) | 0.1018 (6) | 0.6989 (18) | 0.021 (4)* |
| H23A | 0.7597 (13) | 0.2854 (6) | 0.650 (2) | 0.035 (5)* |
| H23B | 0.8176 (12) | 0.2866 (6) | 0.4814 (19) | 0.026 (4)* |

| | | | | |
|---|---|---|---|---|
| H24A | 0.6678 (13) | 0.2668 (6) | 0.346 (2) | 0.035 (5)* |
| H24B | 0.6091 (13) | 0.2712 (6) | 0.511 (2) | 0.037 (5)* |
| H25A | 0.6444 (12) | 0.3504 (6) | 0.512 (2) | 0.029 (4)* |
| H25B | 0.7028 (14) | 0.3458 (6) | 0.351 (2) | 0.041 (5)* |
| H26B | 0.5542 (13) | 0.3223 (6) | 0.212 (2) | 0.038 (5)* |
| H26A | 0.4937 (14) | 0.3258 (6) | 0.375 (2) | 0.039 (5)* |
| H27A | 0.5216 (13) | 0.4080 (6) | 0.380 (2) | 0.035 (5)* |
| H27B | 0.5763 (14) | 0.4048 (6) | 0.210 (2) | 0.035 (5)* |
| H28B | 0.3706 (13) | 0.3813 (6) | 0.257 (2) | 0.032 (5)* |
| H28A | 0.4228 (13) | 0.3761 (6) | 0.087 (2) | 0.037 (5)* |
| H29B | 0.4433 (13) | 0.4581 (6) | 0.0850 (19) | 0.034 (5)* |
| H29A | 0.3906 (12) | 0.4617 (6) | 0.252 (2) | 0.030 (4)* |
| H30B | 0.2386 (13) | 0.4343 (6) | 0.131 (2) | 0.033 (5)* |
| H30A | 0.2948 (13) | 0.4291 (6) | −0.039 (2) | 0.034 (5)* |
| H31B | 0.2590 (13) | 0.5154 (6) | 0.110 (2) | 0.031 (4)* |
| H31A | 0.3098 (14) | 0.5093 (7) | −0.057 (2) | 0.045 (5)* |
| H32C | 0.1556 (15) | 0.4806 (7) | −0.168 (2) | 0.054 (6)* |
| H32B | 0.1019 (17) | 0.4875 (8) | 0.003 (3) | 0.066 (7)* |
| H32A | 0.1373 (15) | 0.5321 (8) | −0.101 (2) | 0.057 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0429 (8) | 0.0236 (7) | 0.0564 (8) | 0.0058 (5) | −0.0149 (6) | 0.0077 (6) |
| O2 | 0.0334 (7) | 0.0367 (8) | 0.0647 (9) | −0.0033 (6) | −0.0236 (6) | 0.0049 (6) |
| O3 | 0.0417 (7) | 0.0266 (8) | 0.0528 (8) | 0.0096 (6) | −0.0097 (6) | 0.0047 (6) |
| O4 | 0.0381 (8) | 0.0369 (8) | 0.0688 (9) | −0.0005 (6) | −0.0288 (7) | 0.0033 (6) |
| N1 | 0.0289 (8) | 0.0277 (9) | 0.0339 (7) | 0.0015 (6) | −0.0054 (6) | 0.0025 (6) |
| N2 | 0.0261 (8) | 0.0215 (8) | 0.0402 (8) | −0.0022 (6) | −0.0109 (6) | 0.0010 (6) |
| N3 | 0.0307 (8) | 0.0276 (9) | 0.0373 (8) | 0.0045 (7) | −0.0060 (6) | 0.0035 (6) |
| N4 | 0.0268 (8) | 0.0247 (8) | 0.0416 (8) | 0.0013 (6) | −0.0113 (6) | −0.0014 (6) |
| C1 | 0.0257 (8) | 0.0236 (9) | 0.0273 (8) | 0.0026 (7) | −0.0036 (7) | 0.0023 (6) |
| C2 | 0.0243 (9) | 0.0242 (10) | 0.0340 (9) | −0.0036 (7) | −0.0044 (7) | −0.0016 (7) |
| C3 | 0.0274 (9) | 0.0175 (9) | 0.0345 (9) | 0.0004 (7) | −0.0048 (7) | 0.0010 (7) |
| C4 | 0.0229 (8) | 0.0241 (9) | 0.0265 (8) | 0.0007 (7) | −0.0023 (6) | 0.0011 (6) |
| C5 | 0.0258 (9) | 0.0252 (10) | 0.0308 (9) | −0.0049 (7) | −0.0039 (7) | −0.0005 (7) |
| C6 | 0.0318 (9) | 0.0195 (9) | 0.0304 (8) | −0.0006 (7) | −0.0015 (7) | 0.0011 (7) |
| C7 | 0.0263 (9) | 0.0216 (9) | 0.0367 (9) | 0.0006 (7) | −0.0049 (7) | 0.0015 (7) |
| C8 | 0.0287 (9) | 0.0241 (9) | 0.0369 (10) | 0.0006 (7) | −0.0060 (8) | 0.0031 (8) |
| C9 | 0.0285 (9) | 0.0231 (9) | 0.0366 (10) | 0.0006 (7) | −0.0039 (8) | 0.0009 (7) |
| C10 | 0.0305 (9) | 0.0235 (10) | 0.0408 (10) | 0.0002 (7) | −0.0062 (8) | 0.0019 (8) |
| C11 | 0.0294 (9) | 0.0237 (10) | 0.0343 (9) | 0.0020 (7) | −0.0033 (8) | 0.0017 (7) |
| C12 | 0.0300 (9) | 0.0216 (9) | 0.0371 (9) | −0.0001 (7) | −0.0032 (8) | 0.0031 (7) |
| C13 | 0.0293 (9) | 0.0222 (9) | 0.0352 (9) | 0.0014 (7) | −0.0028 (7) | 0.0016 (7) |
| C14 | 0.0286 (9) | 0.0254 (10) | 0.0388 (10) | 0.0008 (7) | −0.0028 (8) | 0.0050 (8) |
| C15 | 0.0352 (10) | 0.0241 (10) | 0.0466 (11) | 0.0016 (8) | −0.0081 (9) | 0.0049 (8) |
| C16 | 0.0400 (12) | 0.0368 (13) | 0.0719 (16) | 0.0049 (10) | −0.0178 (11) | 0.0130 (11) |
| C17 | 0.0253 (9) | 0.0258 (10) | 0.0301 (8) | 0.0034 (7) | −0.0029 (7) | 0.0031 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C18 | 0.0263 (9) | 0.0258 (10) | 0.0343 (9) | −0.0030 (7) | −0.0076 (7) | 0.0007 (7) |
| C19 | 0.0300 (9) | 0.0204 (10) | 0.0361 (9) | 0.0009 (7) | −0.0056 (7) | 0.0008 (7) |
| C20 | 0.0238 (8) | 0.0276 (10) | 0.0277 (8) | 0.0030 (7) | −0.0014 (6) | 0.0017 (6) |
| C21 | 0.0260 (9) | 0.0261 (10) | 0.0325 (9) | −0.0031 (7) | −0.0041 (7) | −0.0024 (7) |
| C22 | 0.0312 (9) | 0.0179 (10) | 0.0334 (9) | 0.0018 (7) | −0.0011 (7) | −0.0002 (7) |
| C23 | 0.0292 (9) | 0.0250 (10) | 0.0339 (9) | 0.0028 (7) | −0.0046 (7) | 0.0013 (7) |
| C24 | 0.0285 (9) | 0.0277 (10) | 0.0372 (10) | 0.0030 (7) | −0.0039 (8) | −0.0004 (8) |
| C25 | 0.0291 (9) | 0.0269 (10) | 0.0330 (9) | 0.0042 (7) | −0.0027 (7) | −0.0012 (7) |
| C26 | 0.0330 (10) | 0.0255 (10) | 0.0342 (9) | 0.0049 (8) | −0.0056 (8) | −0.0015 (7) |
| C27 | 0.0315 (10) | 0.0262 (10) | 0.0342 (9) | 0.0030 (7) | −0.0042 (8) | 0.0003 (7) |
| C28 | 0.0325 (9) | 0.0231 (9) | 0.0323 (9) | 0.0025 (7) | −0.0053 (7) | −0.0004 (7) |
| C29 | 0.0299 (9) | 0.0234 (10) | 0.0322 (9) | 0.0001 (7) | −0.0041 (7) | −0.0003 (7) |
| C30 | 0.0315 (9) | 0.0212 (9) | 0.0344 (9) | 0.0002 (7) | −0.0056 (8) | 0.0004 (7) |
| C31 | 0.0352 (10) | 0.0239 (10) | 0.0374 (10) | 0.0016 (8) | −0.0067 (8) | −0.0018 (8) |
| C32 | 0.0398 (11) | 0.0317 (12) | 0.0536 (12) | 0.0067 (9) | −0.0155 (9) | −0.0001 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—N1 | 1.2383 (18) | C15—C16 | 1.516 (3) |
| O2—N1 | 1.2275 (17) | C15—H15B | 0.96 (2) |
| O3—N3 | 1.2389 (19) | C15—H15A | 0.954 (19) |
| O4—N3 | 1.2293 (18) | C16—H16C | 0.99 (2) |
| N1—C1 | 1.437 (2) | C16—H16B | 1.01 (2) |
| N2—C4 | 1.352 (2) | C16—H16A | 0.97 (2) |
| N2—C7 | 1.454 (2) | C17—C18 | 1.387 (2) |
| N2—H2N | 0.873 (18) | C17—C22 | 1.394 (2) |
| N3—C17 | 1.437 (2) | C18—C19 | 1.373 (2) |
| N4—C20 | 1.351 (2) | C18—H18 | 0.938 (19) |
| N4—C23 | 1.452 (2) | C19—C20 | 1.414 (2) |
| N4—H4N | 0.869 (19) | C19—H19 | 0.92 (2) |
| C1—C2 | 1.391 (2) | C20—C21 | 1.417 (2) |
| C1—C6 | 1.394 (2) | C21—C22 | 1.364 (2) |
| C2—C3 | 1.376 (2) | C21—H21 | 0.930 (18) |
| C2—H2 | 0.933 (17) | C22—H22 | 0.916 (18) |
| C3—C4 | 1.410 (2) | C23—C24 | 1.523 (2) |
| C3—H3 | 0.930 (19) | C23—H23A | 0.987 (17) |
| C4—C5 | 1.413 (2) | C23—H23B | 0.982 (17) |
| C5—C6 | 1.364 (2) | C24—C25 | 1.519 (2) |
| C5—H5 | 0.924 (17) | C24—H24A | 0.957 (18) |
| C6—H6 | 0.935 (19) | C24—H24B | 0.966 (18) |
| C7—C8 | 1.523 (2) | C25—C26 | 1.524 (2) |
| C7—H7B | 0.989 (17) | C25—H25A | 0.970 (17) |
| C7—H7A | 0.997 (16) | C25—H25B | 0.995 (19) |
| C8—C9 | 1.520 (2) | C26—C27 | 1.522 (2) |
| C8—H8B | 0.973 (18) | C26—H26B | 1.000 (18) |
| C8—H8A | 0.965 (18) | C26—H26A | 0.992 (18) |
| C9—C10 | 1.526 (2) | C27—C28 | 1.523 (2) |
| C9—H9B | 0.982 (18) | C27—H27A | 0.999 (18) |

| | | | |
|---|---|---|---|
| C9—H9A | 1.002 (17) | C27—H27B | 1.003 (18) |
| C10—C11 | 1.515 (2) | C28—C29 | 1.518 (2) |
| C10—H10B | 0.986 (17) | C28—H28B | 0.988 (17) |
| C10—H10A | 0.984 (18) | C28—H28A | 1.008 (17) |
| C11—C12 | 1.524 (2) | C29—C30 | 1.524 (2) |
| C11—H11B | 0.989 (17) | C29—H29B | 0.961 (18) |
| C11—H11A | 1.012 (17) | C29—H29A | 0.995 (17) |
| C12—C13 | 1.522 (2) | C30—C31 | 1.518 (2) |
| C12—H12B | 0.985 (18) | C30—H30B | 1.003 (17) |
| C12—H12A | 1.000 (17) | C30—H30A | 0.983 (18) |
| C13—C14 | 1.520 (2) | C31—C32 | 1.518 (3) |
| C13—H13B | 0.964 (17) | C31—H31B | 0.957 (17) |
| C13—H13A | 0.959 (18) | C31—H31A | 0.968 (19) |
| C14—C15 | 1.517 (3) | C32—H32C | 1.00 (2) |
| C14—H14B | 0.988 (17) | C32—H32B | 1.02 (2) |
| C14—H14A | 0.973 (19) | C32—H32A | 1.01 (2) |

| | | | |
|---|---|---|---|
| O2—N1—O1 | 122.05 (13) | C15—C16—H16C | 112.1 (12) |
| O2—N1—C1 | 119.14 (14) | C15—C16—H16B | 108.6 (14) |
| O1—N1—C1 | 118.81 (13) | H16C—C16—H16B | 107.8 (19) |
| C4—N2—C7 | 124.44 (14) | C15—C16—H16A | 112.5 (13) |
| C4—N2—H2N | 119.0 (12) | H16C—C16—H16A | 106.6 (18) |
| C7—N2—H2N | 116.1 (12) | H16B—C16—H16A | 109.1 (19) |
| O4—N3—O3 | 121.91 (14) | C18—C17—C22 | 120.94 (14) |
| O4—N3—C17 | 119.14 (15) | C18—C17—N3 | 119.32 (14) |
| O3—N3—C17 | 118.95 (14) | C22—C17—N3 | 119.74 (15) |
| C20—N4—C23 | 124.66 (15) | C19—C18—C17 | 119.84 (15) |
| C20—N4—H4N | 118.1 (12) | C19—C18—H18 | 121.6 (11) |
| C23—N4—H4N | 117.3 (12) | C17—C18—H18 | 118.6 (11) |
| C2—C1—C6 | 120.88 (14) | C18—C19—C20 | 120.58 (16) |
| C2—C1—N1 | 119.20 (14) | C18—C19—H19 | 118.8 (11) |
| C6—C1—N1 | 119.91 (15) | C20—C19—H19 | 120.6 (11) |
| C3—C2—C1 | 119.74 (15) | N4—C20—C19 | 121.98 (16) |
| C3—C2—H2 | 119.2 (11) | N4—C20—C21 | 119.95 (15) |
| C1—C2—H2 | 121.1 (10) | C19—C20—C21 | 118.07 (14) |
| C2—C3—C4 | 120.57 (16) | C22—C21—C20 | 121.05 (15) |
| C2—C3—H3 | 118.2 (11) | C22—C21—H21 | 118.6 (11) |
| C4—C3—H3 | 121.2 (11) | C20—C21—H21 | 120.3 (11) |
| N2—C4—C3 | 122.09 (15) | C21—C22—C17 | 119.53 (16) |
| N2—C4—C5 | 119.85 (14) | C21—C22—H22 | 120.5 (10) |
| C3—C4—C5 | 118.06 (14) | C17—C22—H22 | 119.9 (10) |
| C6—C5—C4 | 121.42 (15) | N4—C23—C24 | 109.36 (14) |
| C6—C5—H5 | 119.6 (11) | N4—C23—H23A | 109.2 (10) |
| C4—C5—H5 | 119.0 (11) | C24—C23—H23A | 110.7 (10) |
| C5—C6—C1 | 119.31 (16) | N4—C23—H23B | 108.9 (10) |
| C5—C6—H6 | 120.6 (11) | C24—C23—H23B | 109.0 (9) |
| C1—C6—H6 | 120.0 (11) | H23A—C23—H23B | 109.7 (14) |
| N2—C7—C8 | 109.51 (13) | C25—C24—C23 | 113.85 (14) |

| | | | |
|---|---|---|---|
| N2—C7—H7B | 110.0 (10) | C25—C24—H24A | 108.0 (11) |
| C8—C7—H7B | 109.6 (10) | C23—C24—H24A | 106.4 (11) |
| N2—C7—H7A | 108.8 (9) | C25—C24—H24B | 108.5 (10) |
| C8—C7—H7A | 110.3 (9) | C23—C24—H24B | 107.9 (10) |
| H7B—C7—H7A | 108.5 (13) | H24A—C24—H24B | 112.3 (15) |
| C9—C8—C7 | 114.04 (14) | C24—C25—C26 | 111.31 (14) |
| C9—C8—H8B | 108.7 (10) | C24—C25—H25A | 110.4 (10) |
| C7—C8—H8B | 107.6 (10) | C26—C25—H25A | 110.5 (10) |
| C9—C8—H8A | 109.4 (11) | C24—C25—H25B | 109.9 (11) |
| C7—C8—H8A | 107.7 (11) | C26—C25—H25B | 108.9 (10) |
| H8B—C8—H8A | 109.2 (15) | H25A—C25—H25B | 105.6 (14) |
| C8—C9—C10 | 111.07 (14) | C27—C26—C25 | 114.96 (14) |
| C8—C9—H9B | 110.3 (10) | C27—C26—H26B | 110.8 (10) |
| C10—C9—H9B | 107.6 (10) | C25—C26—H26B | 107.1 (10) |
| C8—C9—H9A | 109.6 (10) | C27—C26—H26A | 110.3 (11) |
| C10—C9—H9A | 110.0 (10) | C25—C26—H26A | 106.8 (10) |
| H9B—C9—H9A | 108.1 (14) | H26B—C26—H26A | 106.4 (14) |
| C11—C10—C9 | 115.65 (14) | C26—C27—C28 | 112.15 (14) |
| C11—C10—H10B | 110.1 (10) | C26—C27—H27A | 109.2 (10) |
| C9—C10—H10B | 107.1 (10) | C28—C27—H27A | 109.4 (10) |
| C11—C10—H10A | 111.7 (11) | C26—C27—H27B | 110.7 (10) |
| C9—C10—H10A | 106.2 (10) | C28—C27—H27B | 108.9 (10) |
| H10B—C10—H10A | 105.6 (14) | H27A—C27—H27B | 106.4 (14) |
| C10—C11—C12 | 111.98 (14) | C29—C28—C27 | 114.68 (14) |
| C10—C11—H11B | 109.0 (10) | C29—C28—H28B | 109.4 (10) |
| C12—C11—H11B | 108.0 (9) | C27—C28—H28B | 106.9 (9) |
| C10—C11—H11A | 111.0 (10) | C29—C28—H28A | 110.6 (10) |
| C12—C11—H11A | 108.6 (9) | C27—C28—H28A | 108.6 (10) |
| H11B—C11—H11A | 108.1 (14) | H28B—C28—H28A | 106.2 (14) |
| C13—C12—C11 | 114.60 (14) | C28—C29—C30 | 113.29 (14) |
| C13—C12—H12B | 108.4 (10) | C28—C29—H29B | 108.8 (11) |
| C11—C12—H12B | 109.0 (10) | C30—C29—H29B | 109.9 (10) |
| C13—C12—H12A | 108.7 (10) | C28—C29—H29A | 107.5 (9) |
| C11—C12—H12A | 107.2 (10) | C30—C29—H29A | 110.4 (9) |
| H12B—C12—H12A | 108.7 (14) | H29B—C29—H29A | 106.7 (14) |
| C14—C13—C12 | 113.87 (14) | C31—C30—C29 | 114.08 (14) |
| C14—C13—H13B | 109.3 (10) | C31—C30—H30B | 109.7 (10) |
| C12—C13—H13B | 107.6 (10) | C29—C30—H30B | 107.2 (9) |
| C14—C13—H13A | 109.1 (10) | C31—C30—H30A | 110.2 (10) |
| C12—C13—H13A | 107.7 (11) | C29—C30—H30A | 106.7 (10) |
| H13B—C13—H13A | 109.2 (14) | H30B—C30—H30A | 108.7 (14) |
| C15—C14—C13 | 113.89 (15) | C30—C31—C32 | 113.01 (15) |
| C15—C14—H14B | 110.5 (10) | C30—C31—H31B | 109.7 (10) |
| C13—C14—H14B | 107.4 (10) | C32—C31—H31B | 107.8 (10) |
| C15—C14—H14A | 108.6 (11) | C30—C31—H31A | 108.8 (12) |
| C13—C14—H14A | 107.0 (11) | C32—C31—H31A | 108.9 (11) |
| H14B—C14—H14A | 109.4 (15) | H31B—C31—H31A | 108.6 (16) |
| C16—C15—C14 | 113.34 (16) | C31—C32—H32C | 110.0 (12) |

| | | | |
|---|---|---|---|
| C16—C15—H15B | 110.3 (12) | C31—C32—H32B | 112.1 (12) |
| C14—C15—H15B | 109.3 (12) | H32C—C32—H32B | 106.6 (17) |
| C16—C15—H15A | 108.5 (11) | C31—C32—H32A | 113.5 (12) |
| C14—C15—H15A | 109.2 (11) | H32C—C32—H32A | 106.9 (16) |
| H15B—C15—H15A | 106.0 (17) | H32B—C32—H32A | 107.3 (17) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*N*···O3$^{i}$ | 0.873 (18) | 2.234 (19) | 3.101 (2) | 171.7 (16) |
| N4—H4*N*···O1$^{ii}$ | 0.869 (19) | 2.265 (19) | 3.121 (2) | 168.3 (17) |

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