Acta Cryst E STRUCTURE REPORTS

ISSN 1600-5368

# Crystal structure of a four-layered [3.3](3,5)pyridinophane

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Received 14 October 2014
Accepted 27 October 2014

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

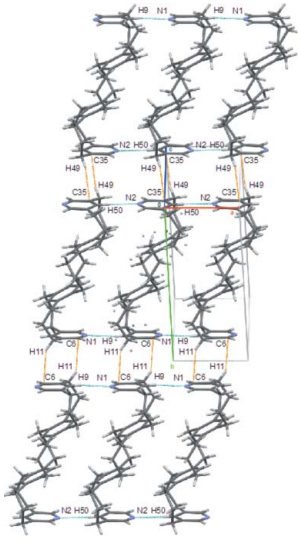
**Keywords**: crystal structure; metacyclophane; pyridinophane; transannular *p*-electronic interaction; C—H···N hydrogen bond; intermolecular short contact

**CCDC reference**: 1031274
**Supporting information**: this article has supporting information at journals.iucr.org/e

The title compound, $C_{40}H_{46}N_2$ {systematic name: 12,30-diazaheptacyclo-[21.13.1.1$^{5,19}$.1$^{6,18}$.1$^{10,14}$.1$^{24,36}$.1$^{28,32}$]dotetraconta-1(37),5(40),6(41),10(42),11,13,-18,23,28,30,32(39),36(38)-dodecaene}, has *syn–anti–syn* geometry wherein the two outer [3.3]metacyclophane (MCP) moieties have a *syn* geometry, and contain the facing benzene and pyridine rings at dihedral angles of 26.26 (10) and 26.46 (10)°, respectively. The rings of the central [3.3]MCP unit are not parallel, but orientated at a slight angle of 2.66 (9)°. Three bridging methylene groups are disordered over two sets of sites in a 0.60:0.40 ratio. In the crystal, the molecules are linked by C—H···N interactions and intermolecular C—H···$\pi$ short contacts, generating a three-dimensional network.

## 1. Chemical context

[3.3]Metapyridinophanes (MPyPs) have been used as ligands in transition metal complexes, and various kinds of metal complexes have been prepared using them (Muralidharan *et al.*, 1989; Fronczek *et al.*, 1989; Krüger, 1995). A variety of types of [3.3]MPyPs are possible, and the [3.3](2,6)PyPs have been studied in detail (Vögtle & Schunder, 1969; Shinmyozu *et al.*, 1986; Bottino *et al.*, 1988). Only a limited number of [3.3](3,5)PyPs have been produced up to now, mainly because of the instability of the coupling precursor, 3,5-bis(halomethyl)pyridine. We have previously used freshly prepared 3,5-bis(chloromethyl)pyridine as the coupling reaction to prepare 2,11-diaza[3.3](3,5)PyP (Satou & Shinmyozu, 2002). One of the major advantages of using [3.3](3,5)PyPs over using [3.3](2,6)PyPs is the potential for forming self-assembled supramolecules when [3.3](3,5)PyPs become coordinated. This occurs because the metacyclophanes (MCPs) have *syn* geometries and the nitrogen lone-pair electrons can readily coordinate with metals without steric hindrance being caused by the bridges. We have also described the synthesis of multilayered [3.3]cyclophanes using the (*p*-tolylsulfonyl)methyl isocyanide method (MCPs; Shibahara *et al.*, 2007) and the (*p*-ethylbenzenesulfonyl)methyl isocyanide method (paracyclophanes; Shibahara *et al.*, 2008). Multilayered [3.3]MCPs that have a pyridine ring at each end may, therefore, form larger supramolecules when they form complexes with transition metals. These new types of supramolecules could have uses as catalysts, inclusion hosts or nanometer-scale materials.




## 2. Structural commentary

The molecular structure of the title compound (at 123 K) is shown Fig.1. The trimethylene bridges are highly flexible and disordered even at this temperature. The molecule has a *syn–anti–syn* geometry, in which the two outer [3.3]MCP moieties have a *syn* geometry and contain opposing benzene and pyridine rings at angles of 26.26 (10)° (between the C4–C8/N1 and C13—C18 planes) and 26.46 (10)° (between the C26—C31 and C35–C39/N2 planes). These angles are comparable to the corresponding angle (24°) in the parent two-layered [3.3]MCP (Semmelhack *et al.*, 1985). The central [3.3]MCP unit is not parallel, but is at a slight angle of 2.66 (9)° between the C13–C18 and C26–C31 planes. There is a twist between the benzene rings of the parent two-layered [3.3]MCP of *ca* 15° about the axis through the centre of each ring, but the twists in the outer [3.3]MCP moieties are only 3.93° (between the N1–C8 and C15–C18 axes) and 2.49° (between the C28–C31 and N2–C36 axes), and the benzene rings overlap each other completely in this molecule. However, the twist in the benzene rings in the central [3.3]MCP unit is quite large, at 11.6° between the C15–C18 and C28–C31 axes. The transannular distances between C8 and C18 [2.968 (3) Å], C28 and C36 [2.955 (3) Å], N1 and C15 [4.168 (3) Å], and N2 and C31 [4.174 (3) Å] are comparable to the distances in the parent two-layered [3.3]MCP (2.995 and 4.171 Å) while the distance between C15 and C31 [2.910 (3) Å] is much shorter than that in the parent two-layered [3.3]MCP-2,11-dione (2.99 Å), which adopts an *anti* geometry (Isaji *et al.*, 2001).

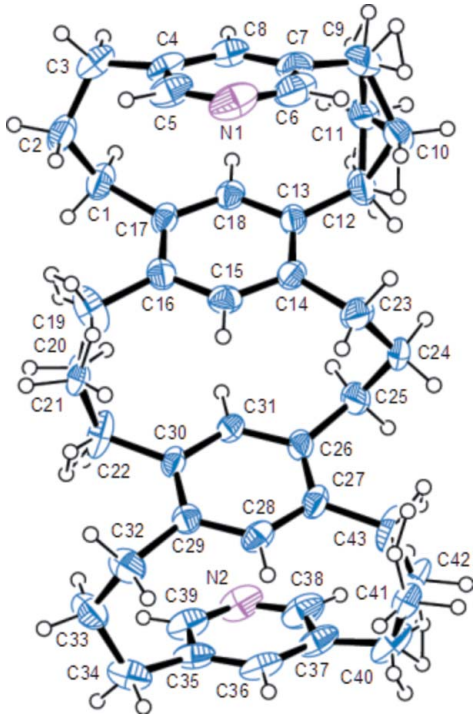



**Figure 1**
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

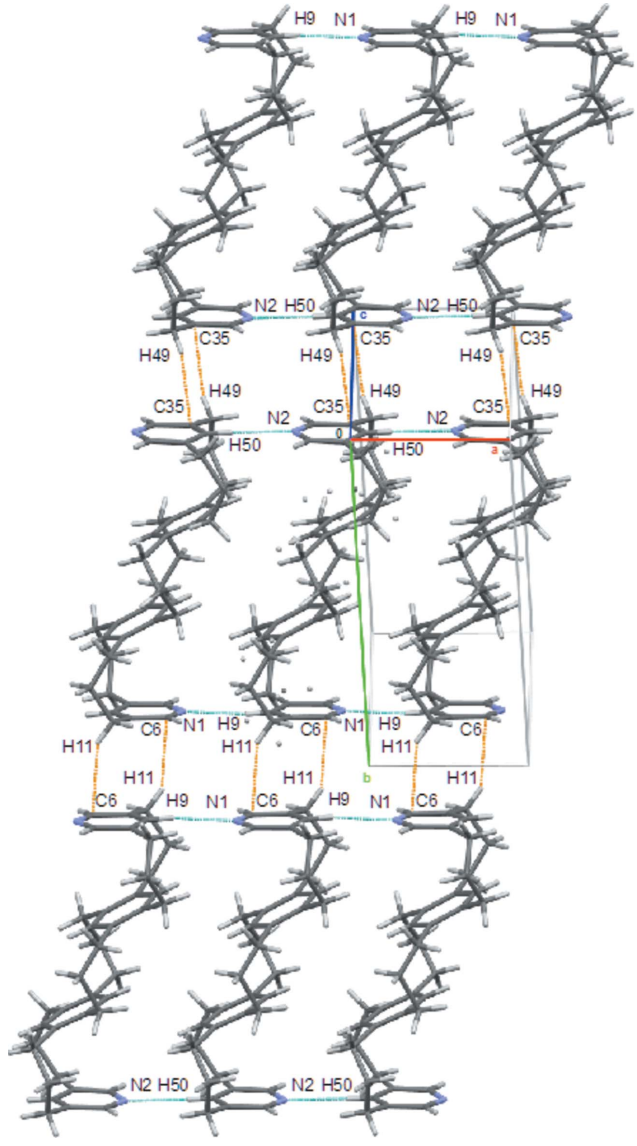



**Figure 2**
Short contacts of the title compound; C—H···$\pi$-type interactions between C6 and H11 and C35 and H49 (orange dashed lines) and short contacts between N1 and H9 and N2 and H50 (light-blue dashed lines).

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C8—H9···N1$^{i}$ | 0.95 | 2.43 | 3.373 (3) | 173 |
| C36—H50···N2$^{ii}$ | 0.95 | 2.47 | 3.394 (3) | 165 |

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

## 3. Supramolecular features

The crystal-packing diagram of the molecule (Fig. 2) shows that molecules are stacked alternately changing direction in the *bc* plane. Two types of intermolecular short contacts are observed. One is the C—H···$\pi$-type interactions between C6 and H11 (2.811 Å) and between C35 and H49 (2.868 Å) in the *bc* plane, while the other is between N1 and H9 (2.429 Å) and between N2 and H50 (2.468 Å) along the *a* axis (Table 1). Both instances of the second type of short contact were found to be shorter than the sum of the van der Waals radii of a nitrogen and hydrogen atom.

## 4. Database survey

The title compound is closely related to the four-layered [3.3]MCP, heptacyclo[21.13.1.1$^{5,19}$.1$^{6,18}$.1$^{10,14}$.1$^{24,36}$.1$^{28,32}$]dotetraconta-1(37),5(40),6(41),10(42),11,13,18,23,28,30,32(39),36(38)-dodecaene), which is the hydrocarbon-only parent molecule (Shibahara *et al.*, 2007), and its charge-transfer complex with tetracyanoethylene (Shibahara *et al.*, 2011, 2014). The four-layered [3.3]MCP changes conformation in the solid state depending on the environment its circumference is in, having a *syn–anti–syn* geometry like the letter '$\omega$' in a ligand-free environment and have a geometry like the letter 's' when it forms a complex.

## 5. Synthesis and crystallization

The title compound was prepared as described by Shibahara *et al.* (2008) by a coupling reaction of 5,7,14,16-tetrakis(bromomethyl)[3.3]metacyclophane with 3,5-bis[2-isocyano-2-(tolylsulfonyl)ethyl]pyridine, which afforded four-layered [3.3](3,5)pyridinophane tetraone, which was converted to the four-layered[3.3](3,5)pyridinophane Shibahara *et al.*, 2009) by a Wolff–Kishner reduction. Purification of the crude product by silica gel column chromatography with $CH_2Cl_2$/EtOH (9:1; *v*/*v*, $R_f$ = 0.53) gave the four-layered pyridinophane (12% isolated yield in two steps). Finally, the product was crystallized from $CH_2Cl_2$/acetone to give single crystals (colourless prisms), m.p. 518 K (decomposed).

$^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 1.8–2.0 (*m*, 12H, $CH_2CH_2CH_2$), 2.4–2.7 (*m*, 24H, $CH_2CH_2CH_2$), 5.97 (*s*, 2H, ArH), 6.21 (*s*, 2H, ArH), 6.91 (*s*, 2H, ArH), 7.84 (*d*, *J* = 1.5 Hz, 4H, ArH). $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$ 26.2, 27.7, 32.4, 32.7, 33.2, 134.0, 134.4, 134.8, 134.8, 135.8, 140.4, 146.8. HRMS (FAB): *m*/*z* [*M*+H]$^+$ calculated for $C_{40}H_{47}N_2$ 555.3739, found 555.3739. Analysis calculated for $C_{40}H_{46}N_2$: C, 86.59; H, 8.36; N, 5.05. found: C, 86.35; H, 8.34; N, 5.01.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{40}H_{46}N_2$ |
| $M_r$ | 554.79 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 123 |
| *a*, *b*, *c* (Å) | 6.1377 (15), 14.643 (4), 17.519 (4) |
| $\alpha$, $\beta$, $\gamma$ (°) | 75.619 (16), 88.369 (17), 86.755 (17) |
| *V* (Å$^3$) | 1522.6 (7) |
| *Z* | 2 |
| Radiation type | Cu *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.52 |
| Crystal size (mm) | 0.45 × 0.30 × 0.16 |
| | |
| Data collection | |
| Diffractometer | Rigaku R-AXIS RAPID |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 20167, 5396, 4455 |
| $R_{int}$ | 0.040 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.602 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.070, 0.204, 1.07 |
| No. of reflections | 5396 |
| No. of parameters | 410 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.42, −0.32 |

Computer programs: *RAPID-AUTO* (Rigaku, 1998), *SIR2011* (Camalli *et al.*, 2012), *SHELXL2014* (Sheldrick, 2008), *Yadokari-XG 2009* (Wakita, 2001; Kabuto *et al.*, 2009), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2006), *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically and refined using a riding model: C—H = 0.95–0.99 Å with $U_{iso}$(H) = 1.2$U_{eq}$(C).

## Acknowledgements

This work was partially supported by a Grant-in-Aid for Science Research (C 25410050) from the Japan Society for the Promotion of Science (JSPS), Japan, and was performed under the Cooperative Research Program of the Network Joint Research Center for Materials and Devices (IMCE, Kyushu University). MW thanks the World Premier International Research Center Initiative (WPI), Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT), Japan.

## References

Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
Bottino, F., Di Grazia, M., Finocchiaro, P., Fronczek, F. R., Mamo, A. & Pappalardo, S. (1988). *J. Org. Chem.* **53**, 3521–3529.
Camalli, M., Carrozzini, B., Cascarano, G. L. & Giacovazzo, C. (2012). *J. Appl. Cryst.* **45**, 351–356.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Fronczek, F. R., Mamo, A. & Pappalardo, S. (1989). *Inorg. Chem.* **28**, 1419–1422.

Isaji, H., Yasutake, M., Takemura, H., Sako, K., Tatemitsu, H., Inazu, T. & Shinmyozu, T. (2001). *Eur. J. Org. Chem.* pp. 2487–2499.
Kabuto, C., Akine, S., Nemoto, T. & Kwon, E. (2009). *J. Cryst. Soc. Jpn*, **51**, 218–224.
Krüger, H.-J. (1995). *Chem. Ber.* **128**, 531–539.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Muralidharan, S., Hojjatie, M., Firestone, M. & Freiser, H. (1989). *J. Org. Chem.* **54**, 393–399.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Satou, T. & Shinmyozu, T. (2002). *J. Chem. Soc. Perkin Trans. 2*, pp. 393–397.
Semmelhack, M. F., Harrison, J. J., Young, D. C., Gutierrez, A., Rafii, S. & Clardy, J. (1985). *J. Am. Chem. Soc.* **107**, 7508–7514.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shibahara, M., Watanabe, M., Aso, K. & Shinmyozu, T. (2008). *Synthesis*, pp. 3749–3754.
Shibahara, M., Watanabe, M., Goto, K. & Shinmyozu, T. (2014). *Acta Cryst.* E**70**, o625–o626.
Shibahara, M., Watanabe, M., Iwanaga, T., Ideta, K. & Shinmyozu, T. (2007). *J. Org. Chem.* **72**, 2865–2877.
Shibahara, M., Watanabe, M., Suenaga, M., Ideta, K., Matsumoto, T. & Shinmyozu, T. (2009). *Tetrahedron Lett.* **50**, 1340–1344.
Shibahara, M., Watanabe, M., Yuan, C. & Shinmyozu, T. (2011). *Tetrahedron Lett.* **52**, 5012–5015.
Shinmyozu, T., Hirai, Y. & Inazu, T. (1986). *J. Org. Chem.* **51**, 1551–1555.
Vögtle, F. & Schunder, L. (1969). *Chem. Ber.* **102**, 2677–2683.
Wakita, K. (2001). *Yadokari-XG*. http://www.hat.hi-ho.ne.jp/k-wakita/yadokari.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# supporting information

*Acta Cryst.* (2014). E**70**, 447-450 [doi:10.1107/S1600536814023691]

# Crystal structure of a four-layered [3.3](3,5)pyridinophane

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## Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *RAPID-AUTO* (Rigaku, 1998); program(s) used to solve structure: *SIR2011* (Camalli *et al.*, 2012); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *Yadokari-XG 2009* (Wakita, 2001; Kabuto *et al.*, 2009); software used to prepare material for publication: *Yadokari-XG 2009* (Wakita, 2001; Kabuto *et al.*, 2009), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2006), *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

## 12,30-Diazaheptacyclo[21.13.1.1$^{5,19}$.1$^{6,18}$.1$^{10,14}$.1$^{24,36}$.1$^{28,32}$]dotetraconta-1(37),5(40),6(41),10(42),11,13,18,23,28,30,32(39),36(38)-dodecaene

*Crystal data*

$C_{40}H_{46}N_2$
$M_r$ = 554.79
Triclinic, $P\overline{1}$
$a$ = 6.1377 (15) Å
$b$ = 14.643 (4) Å
$c$ = 17.519 (4) Å
$\alpha$ = 75.619 (16)°
$\beta$ = 88.369 (17)°
$\gamma$ = 86.755 (17)°
$V$ = 1522.6 (7) Å$^3$
$Z$ = 2
$F(000)$ = 600
$D_x$ = 1.210 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 20167 reflections
$\theta$ = 3.1–68.2°
$\mu$ = 0.52 mm$^{-1}$
$T$ = 123 K
Block, colorless
0.45 × 0.30 × 0.16 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
Radiation source: Rotating anode
Graphite monochromator
Detector resolution: 10.00 pixels mm$^{-1}$
$\omega$ scans
20167 measured reflections
5396 independent reflections
4455 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.040
$\theta_{max}$ = 68.2°, $\theta_{min}$ = 3.1°
$h$ = −7→7
$k$ = −17→17
$l$ = −21→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.204
$S$ = 1.07
5396 reflections
410 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0985P)^2 + 0.5512P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.42$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ 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 was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F2. *R*-factor (gt) are based on *F*. The threshold expression of F2 > 2.0 $\sigma$ (F2) is used only for calculating *R*-factor (gt).

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | −0.1497 (3) | 0.89284 (14) | 0.15093 (15) | 0.0710 (6) | |
| N2 | −0.3765 (3) | 0.11241 (14) | 0.34741 (14) | 0.0686 (6) | |
| C1 | −0.6871 (4) | 0.71516 (16) | 0.33718 (12) | 0.0548 (6) | |
| H1 | −0.6992 | 0.6659 | 0.3871 | 0.066* | |
| H2 | −0.8360 | 0.7317 | 0.3161 | 0.066* | |
| C2 | −0.5956 (4) | 0.80319 (16) | 0.35507 (13) | 0.0611 (6) | |
| H3 | −0.6656 | 0.8121 | 0.4045 | 0.073* | |
| H4 | −0.4377 | 0.7900 | 0.3652 | 0.073* | |
| C3 | −0.6234 (4) | 0.89604 (15) | 0.29260 (14) | 0.0568 (6) | |
| H5 | −0.7804 | 0.9087 | 0.2805 | 0.068* | |
| H6 | −0.5757 | 0.9476 | 0.3146 | 0.068* | |
| C4 | −0.4983 (3) | 0.89824 (13) | 0.21695 (14) | 0.0489 (5) | |
| C5 | −0.2702 (3) | 0.89796 (15) | 0.21396 (16) | 0.0593 (6) | |
| H7 | −0.1969 | 0.9017 | 0.2600 | 0.071* | |
| C6 | −0.2568 (4) | 0.88533 (16) | 0.08720 (17) | 0.0694 (7) | |
| H8 | −0.1726 | 0.8797 | 0.0421 | 0.083* | |
| C7 | −0.4813 (4) | 0.88527 (14) | 0.08282 (14) | 0.0585 (6) | |
| C8 | −0.6003 (3) | 0.89542 (13) | 0.14854 (13) | 0.0505 (5) | |
| H9 | −0.7551 | 0.9006 | 0.1466 | 0.061* | |
| C9 | −0.5889 (6) | 0.86779 (17) | 0.01185 (15) | 0.0813 (9) | |
| H10 | −0.4757 | 0.8638 | −0.0288 | 0.098* | 0.6 |
| H11 | −0.6909 | 0.9221 | −0.0105 | 0.098* | 0.6 |
| H12 | −0.5535 | 0.9178 | −0.0356 | 0.098* | 0.4 |
| H13 | −0.7493 | 0.8692 | 0.0196 | 0.098* | 0.4 |
| C10 | −0.5062 (11) | 0.7705 (4) | 0.0003 (3) | 0.0600 (14) | 0.4 |
| H14 | −0.5447 | 0.7669 | −0.0533 | 0.072* | 0.4 |
| H15 | −0.3450 | 0.7658 | 0.0034 | 0.072* | 0.4 |
| C11 | −0.7216 (7) | 0.7719 (3) | 0.0321 (2) | 0.0624 (10) | 0.6 |
| H16 | −0.8340 | 0.7766 | 0.0728 | 0.075* | 0.6 |
| H17 | −0.7987 | 0.7684 | −0.0159 | 0.075* | 0.6 |
| C12 | −0.5953 (5) | 0.68650 (16) | 0.05909 (12) | 0.0624 (6) | |
| H18 | −0.4697 | 0.6872 | 0.0223 | 0.075* | 0.6 |
| H19 | −0.6855 | 0.6342 | 0.0546 | 0.075* | 0.6 |

| | | | | | |
|---|---|---|---|---|---|
| H20 | −0.5686 | 0.6304 | 0.0375 | 0.075* | 0.4 |
| H21 | −0.7554 | 0.6976 | 0.0628 | 0.075* | 0.4 |
| C13 | −0.5062 (4) | 0.66231 (13) | 0.14222 (10) | 0.0439 (5) | |
| C14 | −0.3006 (4) | 0.61736 (13) | 0.15950 (11) | 0.0465 (5) | |
| C15 | −0.2239 (3) | 0.60393 (13) | 0.23604 (12) | 0.0458 (5) | |
| H22 | −0.0810 | 0.5762 | 0.2473 | 0.055* | |
| C16 | −0.3447 (3) | 0.62880 (13) | 0.29652 (11) | 0.0438 (5) | |
| C17 | −0.5504 (3) | 0.67436 (13) | 0.27882 (11) | 0.0410 (4) | |
| C18 | −0.6270 (3) | 0.68750 (13) | 0.20271 (11) | 0.0409 (4) | |
| H23 | −0.7698 | 0.7152 | 0.1915 | 0.049* | |
| C19 | −0.2635 (4) | 0.6006 (2) | 0.38046 (13) | 0.0730 (8) | |
| H24 | −0.1814 | 0.6546 | 0.3865 | 0.088* | 0.5 |
| H25 | −0.3961 | 0.6001 | 0.4141 | 0.088* | 0.5 |
| H26 | −0.3104 | 0.6488 | 0.4092 | 0.088* | 0.5 |
| H27 | −0.1022 | 0.5927 | 0.3812 | 0.088* | 0.5 |
| C20 | −0.3775 (7) | 0.4969 (3) | 0.4213 (2) | 0.0438 (9) | 0.5 |
| H28 | −0.4192 | 0.4961 | 0.4765 | 0.053* | 0.5 |
| H29 | −0.5135 | 0.4949 | 0.3929 | 0.053* | 0.5 |
| C21 | −0.1416 (7) | 0.5230 (3) | 0.4162 (2) | 0.0446 (9) | 0.5 |
| H30 | −0.1111 | 0.5271 | 0.4704 | 0.054* | 0.5 |
| H31 | 0.0001 | 0.5235 | 0.3876 | 0.054* | 0.5 |
| C22 | −0.2538 (4) | 0.4179 (2) | 0.42155 (14) | 0.0656 (7) | |
| H32 | −0.2123 | 0.3718 | 0.4713 | 0.088 (18)* | 0.5 |
| H33 | −0.4149 | 0.4262 | 0.4200 | 0.058 (13)* | 0.5 |
| H34 | −0.3369 | 0.3655 | 0.4537 | 0.060 (13)* | 0.5 |
| H35 | −0.1237 | 0.4216 | 0.4526 | 0.078 (15)* | 0.5 |
| C23 | −0.1580 (5) | 0.57678 (16) | 0.10252 (13) | 0.0699 (8) | |
| H36 | −0.0159 | 0.5545 | 0.1277 | 0.084* | |
| H37 | −0.1295 | 0.6282 | 0.0553 | 0.084* | |
| C24 | −0.2510 (5) | 0.49556 (16) | 0.07572 (12) | 0.0804 (9) | |
| H38 | −0.1332 | 0.4667 | 0.0483 | 0.097* | |
| H39 | −0.3658 | 0.5222 | 0.0367 | 0.097* | |
| C25 | −0.3485 (5) | 0.41715 (15) | 0.14020 (12) | 0.0644 (7) | |
| H40 | −0.4924 | 0.4407 | 0.1565 | 0.077* | |
| H41 | −0.3737 | 0.3632 | 0.1175 | 0.077* | |
| C26 | −0.2098 (3) | 0.38177 (13) | 0.21302 (10) | 0.0435 (5) | |
| C27 | −0.0039 (3) | 0.33628 (13) | 0.21292 (11) | 0.0448 (5) | |
| C28 | 0.1172 (3) | 0.31807 (13) | 0.28151 (12) | 0.0443 (5) | |
| H42 | 0.2606 | 0.2902 | 0.2807 | 0.053* | |
| C29 | 0.0400 (3) | 0.33839 (12) | 0.35121 (10) | 0.0392 (4) | |
| C30 | −0.1672 (3) | 0.38350 (12) | 0.35142 (10) | 0.0377 (4) | |
| C31 | −0.2876 (3) | 0.40213 (12) | 0.28279 (11) | 0.0396 (4) | |
| H43 | −0.4307 | 0.4303 | 0.2835 | 0.048* | |
| C32 | 0.1769 (4) | 0.30603 (15) | 0.42464 (13) | 0.0553 (6) | |
| H44 | 0.1901 | 0.3602 | 0.4484 | 0.066* | |
| H45 | 0.3255 | 0.2868 | 0.4090 | 0.066* | |
| C33 | 0.0864 (4) | 0.22411 (16) | 0.48715 (13) | 0.0651 (7) | |
| H46 | 0.1605 | 0.2208 | 0.5373 | 0.078* | |

| | | | | | |
|---|---|---|---|---|---|
| H47 | −0.0703 | 0.2395 | 0.4953 | 0.078* | |
| C34 | 0.1071 (4) | 0.12618 (16) | 0.47135 (15) | 0.0628 (6) | |
| H48 | 0.2628 | 0.1108 | 0.4614 | 0.075* | |
| H49 | 0.0597 | 0.0795 | 0.5193 | 0.075* | |
| C35 | −0.0238 (3) | 0.11599 (13) | 0.40259 (15) | 0.0532 (6) | |
| C36 | 0.0739 (3) | 0.11169 (14) | 0.33139 (15) | 0.0553 (6) | |
| H50 | 0.2286 | 0.1072 | 0.3271 | 0.066* | |
| C37 | −0.0508 (4) | 0.11380 (15) | 0.26629 (16) | 0.0595 (6) | |
| C38 | −0.2743 (4) | 0.11317 (17) | 0.27859 (18) | 0.0680 (7) | |
| H51 | −0.3623 | 0.1133 | 0.2348 | 0.082* | |
| C39 | −0.2509 (3) | 0.11535 (15) | 0.40745 (16) | 0.0597 (6) | |
| H52 | −0.3199 | 0.1171 | 0.4563 | 0.072* | |
| C40 | 0.0508 (5) | 0.12235 (19) | 0.18544 (18) | 0.0774 (8) | |
| H53 | −0.0669 | 0.1207 | 0.1484 | 0.093* | 0.6 |
| H54 | 0.1502 | 0.0661 | 0.1876 | 0.093* | 0.6 |
| H55 | 0.2112 | 0.1122 | 0.1902 | 0.093* | 0.4 |
| H56 | −0.0033 | 0.0728 | 0.1624 | 0.093* | 0.4 |
| C41 | 0.1840 (8) | 0.2138 (3) | 0.1500 (2) | 0.0668 (11) | 0.6 |
| H57 | 0.3133 | 0.2097 | 0.1834 | 0.080* | 0.6 |
| H58 | 0.2389 | 0.2097 | 0.0972 | 0.080* | 0.6 |
| C42 | 0.0035 (11) | 0.2064 (4) | 0.1380 (4) | 0.0637 (16) | 0.4 |
| H59 | −0.1577 | 0.2140 | 0.1391 | 0.076* | 0.4 |
| H60 | 0.0441 | 0.1999 | 0.0844 | 0.076* | 0.4 |
| C43 | 0.0848 (5) | 0.30389 (18) | 0.14163 (14) | 0.0708 (7) | |
| H61 | −0.0378 | 0.3097 | 0.1049 | 0.085* | 0.6 |
| H62 | 0.1923 | 0.3497 | 0.1150 | 0.085* | 0.6 |
| H63 | 0.2463 | 0.2999 | 0.1430 | 0.085* | 0.4 |
| H64 | 0.0403 | 0.3516 | 0.0932 | 0.085* | 0.4 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0479 (11) | 0.0539 (12) | 0.1144 (18) | −0.0067 (9) | 0.0227 (11) | −0.0285 (12) |
| N2 | 0.0409 (10) | 0.0638 (12) | 0.1137 (18) | −0.0058 (9) | −0.0037 (11) | −0.0448 (12) |
| C1 | 0.0666 (14) | 0.0559 (13) | 0.0480 (11) | −0.0095 (11) | 0.0150 (10) | −0.0248 (10) |
| C2 | 0.0778 (15) | 0.0628 (14) | 0.0530 (12) | −0.0038 (12) | 0.0063 (11) | −0.0346 (11) |
| C3 | 0.0511 (12) | 0.0500 (12) | 0.0799 (15) | −0.0006 (9) | 0.0029 (11) | −0.0369 (12) |
| C4 | 0.0444 (10) | 0.0324 (10) | 0.0750 (14) | −0.0021 (8) | 0.0053 (10) | −0.0234 (10) |
| C5 | 0.0450 (11) | 0.0454 (12) | 0.0942 (17) | −0.0045 (9) | −0.0016 (11) | −0.0292 (12) |
| C6 | 0.0770 (17) | 0.0441 (13) | 0.0855 (18) | −0.0014 (11) | 0.0254 (14) | −0.0165 (12) |
| C7 | 0.0746 (15) | 0.0308 (10) | 0.0663 (14) | 0.0007 (10) | 0.0092 (12) | −0.0069 (9) |
| C8 | 0.0471 (11) | 0.0335 (10) | 0.0720 (14) | −0.0005 (8) | −0.0029 (10) | −0.0155 (10) |
| C9 | 0.137 (3) | 0.0452 (13) | 0.0563 (14) | 0.0068 (15) | −0.0120 (15) | −0.0034 (11) |
| C10 | 0.091 (4) | 0.048 (3) | 0.037 (3) | −0.008 (3) | −0.003 (3) | −0.002 (2) |
| C11 | 0.093 (3) | 0.051 (2) | 0.0446 (19) | 0.011 (2) | −0.0237 (19) | −0.0144 (16) |
| C12 | 0.1004 (19) | 0.0522 (13) | 0.0364 (11) | −0.0181 (12) | −0.0042 (11) | −0.0109 (9) |
| C13 | 0.0676 (13) | 0.0327 (9) | 0.0324 (9) | −0.0112 (9) | 0.0030 (8) | −0.0089 (7) |
| C14 | 0.0672 (13) | 0.0309 (9) | 0.0396 (10) | −0.0050 (9) | 0.0170 (9) | −0.0068 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0492 (11) | 0.0328 (10) | 0.0515 (11) | −0.0034 (8) | 0.0065 (9) | −0.0036 (8) |
| C16 | 0.0559 (11) | 0.0379 (10) | 0.0379 (10) | −0.0118 (9) | −0.0004 (8) | −0.0080 (8) |
| C17 | 0.0536 (11) | 0.0360 (9) | 0.0370 (9) | −0.0121 (8) | 0.0084 (8) | −0.0147 (8) |
| C18 | 0.0478 (10) | 0.0337 (9) | 0.0434 (10) | −0.0084 (8) | 0.0026 (8) | −0.0123 (8) |
| C19 | 0.0617 (14) | 0.105 (2) | 0.0419 (12) | −0.0207 (14) | −0.0051 (10) | 0.0048 (12) |
| C20 | 0.065 (2) | 0.041 (2) | 0.0260 (16) | 0.0018 (18) | 0.0066 (16) | −0.0124 (15) |
| C21 | 0.062 (2) | 0.043 (2) | 0.0361 (19) | −0.0004 (18) | −0.0067 (17) | −0.0238 (16) |
| C22 | 0.0569 (13) | 0.101 (2) | 0.0585 (14) | −0.0181 (13) | 0.0111 (11) | −0.0554 (15) |
| C23 | 0.105 (2) | 0.0451 (12) | 0.0507 (13) | 0.0090 (12) | 0.0363 (13) | −0.0029 (10) |
| C24 | 0.159 (3) | 0.0534 (13) | 0.0282 (10) | 0.0263 (16) | 0.0051 (13) | −0.0161 (10) |
| C25 | 0.110 (2) | 0.0449 (12) | 0.0435 (12) | 0.0036 (12) | −0.0247 (12) | −0.0202 (10) |
| C26 | 0.0670 (13) | 0.0339 (9) | 0.0330 (9) | −0.0082 (9) | −0.0032 (8) | −0.0133 (7) |
| C27 | 0.0641 (12) | 0.0366 (10) | 0.0388 (10) | −0.0132 (9) | 0.0129 (9) | −0.0181 (8) |
| C28 | 0.0466 (10) | 0.0359 (10) | 0.0554 (12) | −0.0076 (8) | 0.0077 (9) | −0.0202 (9) |
| C29 | 0.0490 (10) | 0.0314 (9) | 0.0393 (10) | −0.0099 (8) | −0.0017 (8) | −0.0108 (8) |
| C30 | 0.0502 (10) | 0.0349 (9) | 0.0322 (9) | −0.0092 (8) | 0.0044 (7) | −0.0151 (7) |
| C31 | 0.0488 (10) | 0.0334 (9) | 0.0401 (10) | −0.0028 (8) | −0.0016 (8) | −0.0153 (8) |
| C32 | 0.0626 (13) | 0.0455 (11) | 0.0574 (13) | −0.0127 (10) | −0.0183 (10) | −0.0080 (10) |
| C33 | 0.0859 (17) | 0.0526 (13) | 0.0535 (13) | −0.0114 (12) | −0.0261 (12) | −0.0020 (10) |
| C34 | 0.0543 (13) | 0.0446 (12) | 0.0803 (16) | −0.0028 (10) | −0.0135 (11) | 0.0036 (11) |
| C35 | 0.0433 (11) | 0.0291 (9) | 0.0860 (16) | −0.0004 (8) | −0.0084 (10) | −0.0114 (10) |
| C36 | 0.0391 (10) | 0.0331 (10) | 0.0970 (17) | 0.0025 (8) | 0.0045 (11) | −0.0240 (11) |
| C37 | 0.0520 (12) | 0.0411 (11) | 0.0972 (18) | −0.0018 (9) | −0.0017 (12) | −0.0399 (12) |
| C38 | 0.0522 (13) | 0.0585 (14) | 0.108 (2) | −0.0025 (11) | −0.0110 (13) | −0.0475 (14) |
| C39 | 0.0447 (11) | 0.0446 (12) | 0.0938 (17) | −0.0024 (9) | 0.0033 (11) | −0.0253 (12) |
| C40 | 0.0780 (17) | 0.0647 (17) | 0.110 (2) | −0.0023 (13) | 0.0105 (15) | −0.0612 (17) |
| C41 | 0.084 (3) | 0.069 (3) | 0.050 (2) | 0.018 (2) | 0.014 (2) | −0.0262 (19) |
| C42 | 0.071 (4) | 0.064 (4) | 0.075 (4) | −0.011 (3) | 0.022 (3) | −0.053 (3) |
| C43 | 0.0954 (18) | 0.0707 (16) | 0.0608 (14) | −0.0262 (14) | 0.0334 (13) | −0.0428 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C5 | 1.327 (3) | C21—H30 | 0.9900 |
| N1—C6 | 1.342 (3) | C21—H31 | 0.9900 |
| N2—C39 | 1.332 (3) | C22—C30 | 1.512 (2) |
| N2—C38 | 1.341 (3) | C22—H32 | 0.9900 |
| C1—C17 | 1.518 (2) | C22—H33 | 0.9900 |
| C1—C2 | 1.539 (3) | C22—H34 | 0.9900 |
| C1—H1 | 0.9900 | C22—H35 | 0.9900 |
| C1—H2 | 0.9900 | C23—C24 | 1.527 (4) |
| C2—C3 | 1.524 (3) | C23—H36 | 0.9900 |
| C2—H3 | 0.9900 | C23—H37 | 0.9900 |
| C2—H4 | 0.9900 | C24—C25 | 1.532 (3) |
| C3—C4 | 1.507 (3) | C24—H38 | 0.9900 |
| C3—H5 | 0.9900 | C24—H39 | 0.9900 |
| C3—H6 | 0.9900 | C25—C26 | 1.518 (3) |
| C4—C8 | 1.378 (3) | C25—H40 | 0.9900 |
| C4—C5 | 1.399 (3) | C25—H41 | 0.9900 |

| | | | |
|---|---|---|---|
| C5—H7 | 0.9500 | C26—C31 | 1.394 (2) |
| C6—C7 | 1.383 (4) | C26—C27 | 1.395 (3) |
| C6—H8 | 0.9500 | C27—C28 | 1.392 (3) |
| C7—C8 | 1.381 (3) | C27—C43 | 1.519 (2) |
| C7—C9 | 1.507 (4) | C28—C29 | 1.391 (3) |
| C8—H9 | 0.9500 | C28—H42 | 0.9500 |
| C9—C10 | 1.544 (6) | C29—C30 | 1.399 (3) |
| C9—C11 | 1.621 (5) | C29—C32 | 1.516 (3) |
| C9—H10 | 0.9900 | C30—C31 | 1.390 (2) |
| C9—H11 | 0.9900 | C31—H43 | 0.9500 |
| C9—H12 | 0.9900 | C32—C33 | 1.528 (3) |
| C9—H13 | 0.9900 | C32—H44 | 0.9900 |
| C10—C12 | 1.512 (6) | C32—H45 | 0.9900 |
| C10—H14 | 0.9900 | C33—C34 | 1.524 (3) |
| C10—H15 | 0.9900 | C33—H46 | 0.9900 |
| C11—C12 | 1.416 (4) | C33—H47 | 0.9900 |
| C11—H16 | 0.9900 | C34—C35 | 1.509 (3) |
| C11—H17 | 0.9900 | C34—H48 | 0.9900 |
| C12—C13 | 1.521 (3) | C34—H49 | 0.9900 |
| C12—H18 | 0.9900 | C35—C36 | 1.383 (3) |
| C12—H19 | 0.9900 | C35—C39 | 1.395 (3) |
| C12—H20 | 0.9900 | C36—C37 | 1.385 (3) |
| C12—H21 | 0.9900 | C36—H50 | 0.9500 |
| C13—C18 | 1.388 (3) | C37—C38 | 1.383 (3) |
| C13—C14 | 1.395 (3) | C37—C40 | 1.510 (4) |
| C14—C15 | 1.397 (3) | C38—H51 | 0.9500 |
| C14—C23 | 1.517 (3) | C39—H52 | 0.9500 |
| C15—C16 | 1.386 (3) | C40—C42 | 1.325 (7) |
| C15—H22 | 0.9500 | C40—C41 | 1.591 (5) |
| C16—C17 | 1.399 (3) | C40—H53 | 0.9900 |
| C16—C19 | 1.516 (3) | C40—H54 | 0.9900 |
| C17—C18 | 1.391 (3) | C40—H55 | 0.9900 |
| C18—H23 | 0.9500 | C40—H56 | 0.9900 |
| C19—C21 | 1.350 (5) | C41—C43 | 1.396 (5) |
| C19—C20 | 1.691 (5) | C41—H57 | 0.9900 |
| C19—H24 | 0.9900 | C41—H58 | 0.9900 |
| C19—H25 | 0.9900 | C42—C43 | 1.555 (6) |
| C19—H26 | 0.9900 | C42—H59 | 0.9900 |
| C19—H27 | 0.9900 | C42—H60 | 0.9900 |
| C20—C22 | 1.347 (4) | C43—H61 | 0.9900 |
| C20—H28 | 0.9900 | C43—H62 | 0.9900 |
| C20—H29 | 0.9900 | C43—H63 | 0.9900 |
| C21—C22 | 1.701 (5) | C43—H64 | 0.9900 |
| | | | |
| C5—N1—C6 | 116.8 (2) | C30—C22—H32 | 110.3 |
| C39—N2—C38 | 116.6 (2) | C21—C22—H32 | 110.3 |
| C17—C1—C2 | 114.26 (18) | C30—C22—H33 | 110.3 |
| C17—C1—H1 | 108.7 | C21—C22—H33 | 110.3 |

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|---|---|---|---|
| C2—C1—H1 | 108.7 | H32—C22—H33 | 108.6 |
| C17—C1—H2 | 108.7 | C20—C22—H34 | 105.3 |
| C2—C1—H2 | 108.7 | C30—C22—H34 | 105.3 |
| H1—C1—H2 | 107.6 | C20—C22—H35 | 105.3 |
| C3—C2—C1 | 117.46 (19) | C30—C22—H35 | 105.3 |
| C3—C2—H3 | 107.9 | H34—C22—H35 | 106.0 |
| C1—C2—H3 | 107.9 | C14—C23—C24 | 115.7 (2) |
| C3—C2—H4 | 107.9 | C14—C23—H36 | 108.4 |
| C1—C2—H4 | 107.9 | C24—C23—H36 | 108.4 |
| H3—C2—H4 | 107.2 | C14—C23—H37 | 108.4 |
| C4—C3—C2 | 114.39 (17) | C24—C23—H37 | 108.4 |
| C4—C3—H5 | 108.7 | H36—C23—H37 | 107.4 |
| C2—C3—H5 | 108.7 | C23—C24—C25 | 116.52 (17) |
| C4—C3—H6 | 108.7 | C23—C24—H38 | 108.2 |
| C2—C3—H6 | 108.7 | C25—C24—H38 | 108.2 |
| H5—C3—H6 | 107.6 | C23—C24—H39 | 108.2 |
| C8—C4—C5 | 116.5 (2) | C25—C24—H39 | 108.2 |
| C8—C4—C3 | 122.13 (19) | H38—C24—H39 | 107.3 |
| C5—C4—C3 | 121.2 (2) | C26—C25—C24 | 115.1 (2) |
| N1—C5—C4 | 124.2 (2) | C26—C25—H40 | 108.5 |
| N1—C5—H7 | 117.9 | C24—C25—H40 | 108.5 |
| C4—C5—H7 | 117.9 | C26—C25—H41 | 108.5 |
| N1—C6—C7 | 124.4 (2) | C24—C25—H41 | 108.5 |
| N1—C6—H8 | 117.8 | H40—C25—H41 | 107.5 |
| C7—C6—H8 | 117.8 | C31—C26—C27 | 118.34 (17) |
| C8—C7—C6 | 116.8 (2) | C31—C26—C25 | 117.50 (19) |
| C8—C7—C9 | 121.8 (2) | C27—C26—C25 | 124.01 (18) |
| C6—C7—C9 | 121.3 (2) | C28—C27—C26 | 118.03 (16) |
| C4—C8—C7 | 121.1 (2) | C28—C27—C43 | 120.2 (2) |
| C4—C8—H9 | 119.4 | C26—C27—C43 | 121.71 (19) |
| C7—C8—H9 | 119.4 | C29—C28—C27 | 123.65 (18) |
| C7—C9—C10 | 109.2 (3) | C29—C28—H42 | 118.2 |
| C7—C9—C11 | 113.0 (2) | C27—C28—H42 | 118.2 |
| C7—C9—H10 | 109.0 | C28—C29—C30 | 118.22 (17) |
| C11—C9—H10 | 109.0 | C28—C29—C32 | 118.96 (18) |
| C7—C9—H11 | 109.0 | C30—C29—C32 | 122.72 (17) |
| C11—C9—H11 | 109.0 | C31—C30—C29 | 118.05 (16) |
| H10—C9—H11 | 107.8 | C31—C30—C22 | 120.01 (18) |
| C7—C9—H12 | 109.8 | C29—C30—C22 | 121.79 (17) |
| C10—C9—H12 | 109.8 | C30—C31—C26 | 123.58 (18) |
| C7—C9—H13 | 109.8 | C30—C31—H43 | 118.2 |
| C10—C9—H13 | 109.8 | C26—C31—H43 | 118.2 |
| H12—C9—H13 | 108.3 | C29—C32—C33 | 114.53 (17) |
| C12—C10—C9 | 115.1 (4) | C29—C32—H44 | 108.6 |
| C12—C10—H14 | 108.5 | C33—C32—H44 | 108.6 |
| C9—C10—H14 | 108.5 | C29—C32—H45 | 108.6 |
| C12—C10—H15 | 108.5 | C33—C32—H45 | 108.6 |
| C9—C10—H15 | 108.5 | H44—C32—H45 | 107.6 |

| | | | |
|---|---|---|---|
| H14—C10—H15 | 107.5 | C34—C33—C32 | 117.7 (2) |
| C12—C11—C9 | 116.1 (3) | C34—C33—H46 | 107.9 |
| C12—C11—H16 | 108.3 | C32—C33—H46 | 107.9 |
| C9—C11—H16 | 108.3 | C34—C33—H47 | 107.9 |
| C12—C11—H17 | 108.3 | C32—C33—H47 | 107.9 |
| C9—C11—H17 | 108.3 | H46—C33—H47 | 107.2 |
| H16—C11—H17 | 107.4 | C35—C34—C33 | 114.42 (17) |
| C11—C12—C13 | 119.1 (2) | C35—C34—H48 | 108.7 |
| C10—C12—C13 | 117.6 (3) | C33—C34—H48 | 108.7 |
| C11—C12—H18 | 107.5 | C35—C34—H49 | 108.7 |
| C13—C12—H18 | 107.5 | C33—C34—H49 | 108.7 |
| C11—C12—H19 | 107.5 | H48—C34—H49 | 107.6 |
| C13—C12—H19 | 107.5 | C36—C35—C39 | 117.3 (2) |
| H18—C12—H19 | 107.0 | C36—C35—C34 | 121.9 (2) |
| C10—C12—H20 | 107.9 | C39—C35—C34 | 120.8 (2) |
| C13—C12—H20 | 107.9 | C35—C36—C37 | 120.85 (19) |
| C10—C12—H21 | 107.9 | C35—C36—H50 | 119.6 |
| C13—C12—H21 | 107.9 | C37—C36—H50 | 119.6 |
| H20—C12—H21 | 107.2 | C38—C37—C36 | 116.2 (2) |
| C18—C13—C14 | 118.27 (17) | C38—C37—C40 | 121.9 (2) |
| C18—C13—C12 | 120.13 (19) | C36—C37—C40 | 121.8 (2) |
| C14—C13—C12 | 121.57 (18) | N2—C38—C37 | 125.2 (2) |
| C13—C14—C15 | 118.04 (17) | N2—C38—H51 | 117.4 |
| C13—C14—C23 | 124.8 (2) | C37—C38—H51 | 117.4 |
| C15—C14—C23 | 117.1 (2) | N2—C39—C35 | 123.7 (2) |
| C16—C15—C14 | 123.64 (19) | N2—C39—H52 | 118.1 |
| C16—C15—H22 | 118.2 | C35—C39—H52 | 118.1 |
| C14—C15—H22 | 118.2 | C42—C40—C37 | 111.4 (3) |
| C15—C16—C17 | 118.06 (17) | C37—C40—C41 | 116.6 (2) |
| C15—C16—C19 | 120.7 (2) | C37—C40—H53 | 108.1 |
| C17—C16—C19 | 121.05 (19) | C41—C40—H53 | 108.1 |
| C18—C17—C16 | 118.22 (17) | C37—C40—H54 | 108.1 |
| C18—C17—C1 | 118.57 (18) | C41—C40—H54 | 108.1 |
| C16—C17—C1 | 123.10 (17) | H53—C40—H54 | 107.3 |
| C13—C18—C17 | 123.63 (18) | C42—C40—H55 | 109.3 |
| C13—C18—H23 | 118.2 | C37—C40—H55 | 109.3 |
| C17—C18—H23 | 118.2 | C42—C40—H56 | 109.3 |
| C21—C19—C16 | 128.5 (3) | C37—C40—H56 | 109.3 |
| C16—C19—C20 | 104.8 (2) | H55—C40—H56 | 108.0 |
| C21—C19—H24 | 105.2 | C43—C41—C40 | 120.6 (3) |
| C16—C19—H24 | 105.2 | C43—C41—H57 | 107.2 |
| C21—C19—H25 | 105.2 | C40—C41—H57 | 107.2 |
| C16—C19—H25 | 105.2 | C43—C41—H58 | 107.2 |
| H24—C19—H25 | 105.9 | C40—C41—H58 | 107.2 |
| C16—C19—H26 | 110.8 | H57—C41—H58 | 106.8 |
| C20—C19—H26 | 110.8 | C40—C42—C43 | 128.5 (5) |
| C16—C19—H27 | 110.8 | C40—C42—H59 | 105.2 |
| C20—C19—H27 | 110.8 | C43—C42—H59 | 105.2 |

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| H26—C19—H27 | 108.9 | C40—C42—H60 | 105.2 |
| C22—C20—C19 | 116.5 (3) | C43—C42—H60 | 105.2 |
| C22—C20—H28 | 108.2 | H59—C42—H60 | 105.9 |
| C19—C20—H28 | 108.2 | C41—C43—C27 | 120.9 (3) |
| C22—C20—H29 | 108.2 | C27—C43—C42 | 113.0 (3) |
| C19—C20—H29 | 108.2 | C41—C43—H61 | 107.1 |
| H28—C20—H29 | 107.3 | C27—C43—H61 | 107.1 |
| C19—C21—C22 | 115.7 (3) | C41—C43—H62 | 107.1 |
| C19—C21—H30 | 108.4 | C27—C43—H62 | 107.1 |
| C22—C21—H30 | 108.4 | H61—C43—H62 | 106.8 |
| C19—C21—H31 | 108.4 | C27—C43—H63 | 109.0 |
| C22—C21—H31 | 108.4 | C42—C43—H63 | 109.0 |
| H30—C21—H31 | 107.4 | C27—C43—H64 | 109.0 |
| C20—C22—C30 | 127.9 (3) | C42—C43—H64 | 109.0 |
| C30—C22—C21 | 106.9 (2) | H63—C43—H64 | 107.8 |

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| C17—C1—C2—C3 | 75.5 (3) | C19—C21—C22—C30 | 94.6 (3) |
| C1—C2—C3—C4 | −66.0 (3) | C13—C14—C23—C24 | −63.4 (3) |
| C2—C3—C4—C8 | 107.4 (2) | C15—C14—C23—C24 | 113.0 (2) |
| C2—C3—C4—C5 | −69.0 (3) | C14—C23—C24—C25 | −46.4 (3) |
| C6—N1—C5—C4 | −1.6 (3) | C23—C24—C25—C26 | −46.4 (3) |
| C8—C4—C5—N1 | −1.8 (3) | C24—C25—C26—C31 | 111.4 (2) |
| C3—C4—C5—N1 | 174.8 (2) | C24—C25—C26—C27 | −63.9 (3) |
| C5—N1—C6—C7 | 1.9 (4) | C31—C26—C27—C28 | −3.2 (3) |
| N1—C6—C7—C8 | 1.2 (3) | C25—C26—C27—C28 | 172.08 (18) |
| N1—C6—C7—C9 | −174.4 (2) | C31—C26—C27—C43 | 174.58 (18) |
| C5—C4—C8—C7 | 5.0 (3) | C25—C26—C27—C43 | −10.1 (3) |
| C3—C4—C8—C7 | −171.58 (17) | C26—C27—C28—C29 | 3.4 (3) |
| C6—C7—C8—C4 | −4.8 (3) | C43—C27—C28—C29 | −174.48 (18) |
| C9—C7—C8—C4 | 170.89 (19) | C27—C28—C29—C30 | −3.0 (3) |
| C8—C7—C9—C10 | −117.0 (3) | C27—C28—C29—C32 | 173.57 (17) |
| C6—C7—C9—C10 | 58.5 (4) | C28—C29—C30—C31 | 2.5 (3) |
| C8—C7—C9—C11 | −59.7 (3) | C32—C29—C30—C31 | −173.94 (16) |
| C6—C7—C9—C11 | 115.8 (3) | C28—C29—C30—C22 | −173.11 (19) |
| C7—C9—C10—C12 | 73.5 (5) | C32—C29—C30—C22 | 10.5 (3) |
| C11—C9—C10—C12 | −31.4 (3) | C20—C22—C30—C31 | −32.6 (4) |
| C7—C9—C11—C12 | −63.3 (4) | C21—C22—C30—C31 | −95.1 (2) |
| C10—C9—C11—C12 | 34.1 (3) | C20—C22—C30—C29 | 142.9 (3) |
| C9—C11—C12—C10 | −32.8 (3) | C21—C22—C30—C29 | 80.4 (3) |
| C9—C11—C12—C13 | 73.3 (4) | C29—C30—C31—C26 | −2.7 (3) |
| C9—C10—C12—C11 | 34.3 (4) | C22—C30—C31—C26 | 173.01 (19) |
| C9—C10—C12—C13 | −74.3 (5) | C27—C26—C31—C30 | 3.1 (3) |
| C11—C12—C13—C18 | 35.0 (4) | C25—C26—C31—C30 | −172.56 (18) |
| C10—C12—C13—C18 | 101.7 (4) | C28—C29—C32—C33 | −108.0 (2) |
| C11—C12—C13—C14 | −143.1 (3) | C30—C29—C32—C33 | 68.4 (3) |
| C10—C12—C13—C14 | −76.3 (4) | C29—C32—C33—C34 | 73.9 (3) |
| C18—C13—C14—C15 | −2.7 (3) | C32—C33—C34—C35 | −65.6 (3) |
| C12—C13—C14—C15 | 175.31 (18) | C33—C34—C35—C36 | 105.7 (2) |

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| C18—C13—C14—C23 | 173.67 (19) | C33—C34—C35—C39 | −71.0 (3) |
| C12—C13—C14—C23 | −8.3 (3) | C39—C35—C36—C37 | 4.7 (3) |
| C13—C14—C15—C16 | 3.1 (3) | C34—C35—C36—C37 | −172.15 (18) |
| C23—C14—C15—C16 | −173.58 (18) | C35—C36—C37—C38 | −4.7 (3) |
| C14—C15—C16—C17 | −3.5 (3) | C35—C36—C37—C40 | 171.67 (19) |
| C14—C15—C16—C19 | 171.89 (19) | C39—N2—C38—C37 | 2.1 (4) |
| C15—C16—C17—C18 | 3.5 (3) | C36—C37—C38—N2 | 1.3 (3) |
| C19—C16—C17—C18 | −171.89 (19) | C40—C37—C38—N2 | −175.1 (2) |
| C15—C16—C17—C1 | −172.74 (18) | C38—N2—C39—C35 | −2.2 (3) |
| C19—C16—C17—C1 | 11.9 (3) | C36—C35—C39—N2 | −1.1 (3) |
| C2—C1—C17—C18 | −106.2 (2) | C34—C35—C39—N2 | 175.7 (2) |
| C2—C1—C17—C16 | 69.9 (3) | C38—C37—C40—C42 | 67.1 (4) |
| C14—C13—C18—C17 | 3.1 (3) | C36—C37—C40—C42 | −109.1 (4) |
| C12—C13—C18—C17 | −174.99 (17) | C38—C37—C40—C41 | 116.7 (3) |
| C16—C17—C18—C13 | −3.4 (3) | C36—C37—C40—C41 | −59.5 (4) |
| C1—C17—C18—C13 | 172.93 (17) | C42—C40—C41—C43 | 37.8 (4) |
| C15—C16—C19—C21 | −31.6 (4) | C37—C40—C41—C43 | −56.7 (5) |
| C17—C16—C19—C21 | 143.6 (3) | C37—C40—C42—C43 | 69.5 (6) |
| C15—C16—C19—C20 | −92.8 (2) | C41—C40—C42—C43 | −37.3 (4) |
| C17—C16—C19—C20 | 82.4 (3) | C40—C41—C43—C27 | 61.4 (5) |
| C21—C19—C20—C22 | −30.3 (3) | C40—C41—C43—C42 | −31.4 (4) |
| C16—C19—C20—C22 | 96.1 (3) | C28—C27—C43—C41 | 43.8 (4) |
| C16—C19—C21—C22 | −60.6 (4) | C26—C27—C43—C41 | −133.9 (3) |
| C20—C19—C21—C22 | 23.4 (2) | C28—C27—C43—C42 | 94.5 (4) |
| C19—C20—C22—C30 | −63.5 (4) | C26—C27—C43—C42 | −83.3 (4) |
| C19—C20—C22—C21 | 23.7 (2) | C40—C42—C43—C41 | 43.5 (5) |
| C19—C21—C22—C20 | −29.9 (3) | C40—C42—C43—C27 | −67.9 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C8—H9···N1$^{i}$ | 0.95 | 2.43 | 3.373 (3) | 173 |
| C36—H50···N2$^{ii}$ | 0.95 | 2.47 | 3.394 (3) | 165 |

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