Crystal structures of monohydrate and methanol solvate compounds of {1-[(3,5-bis[(4,6-dimethylpyridin-2-yl)amino]methyl]-2,4,6-triethylbenzyl]-amino)cyclopentyl]methanol

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In the title monohydrate compound, 1a, and the methanol solvate compound, 1b, the triethylbenzene derivative, C_{35}H_{51}N_{5}O, has three functionalized side arms and three ethyl groups, the former being located on one side of the central benzene ring, while the latter are directed to the opposite side. Both the crystals are constructed of structurally similar dimers of 1:1 host–guest complexes held together by N—H···O and O—H···N hydrogen bonds, and in 1a additionally by O—H···O hydrogen bonds. The structure of 1b contains additional highly disordered solvent molecules. Thus, the SQUEEZE routine [Spek (2015). Acta Cryst. C71, 9–18] in PLATON was used to generate a modified data set, in which the contribution of the disordered molecules to the structure amplitudes is eliminated. These solvent molecules are not considered in the reported chemical formula.

1. Chemical context

Representatives of the class of 1,3,5-trisubstituted 2,4,6-tri-alkylbenzenes have been shown to have the ability to act as artificial carbohydrate receptors. Depending on the nature of their building blocks, these compounds display different, interesting binding efficiencies and selectivities towards carbohydrates (Mazik, 2009, 2012; Stapf et al., 2020). Our systematic studies have shown the enormous potential of this acyclic receptor architecture for versatile structural modifications, which enable the identification of interesting structure–activity relationships. For example, we have observed that the combination of two 2-aminopyridine units with another recognition group provides receptors having a binding preference for β-glucoside vs β-galactoside (Mazik & Kuschel, 2008; Mazik & Geffert, 2011; Stapf et al., 2020).

[1-[(3,5-Bis[(4,6-dimethylpyridin-2-yl)amino]methyl]-2,4,6-triethylbenzyl]amino)cyclopentyl]methanol, I, represents a
triethylbenzene derivative bearing, in addition to the above-mentioned pyridinyl units, a 1-hydroxymethyl-cyclopentyl-amino group. The crystal structures of the monohydrate, 1a, and the methanol solvate, 1b, are described here.

2. Structural commentary
Compounds 1a and 1b crystallize in the space groups $P2_1/c$ and $P1$, respectively. The molecular structures depicted in Figs. 1 and 2 reveal similar host geometries with a fully alternating arrangement of the substituents above and below the plane of the central arene ring $[ab'ab'ab']$ pattern, $a =$ above, $b =$ below ($a'/b' = $ Et above/below); for a discussion on the conformations of triethylbenzene-based compounds, see: Koch et al., 2017; Schulze et al., 2017]. In other words, the three functionalized side arms point to one face of the central benzene ring and participate in the formation of hydrogen bonds with the guest solvent molecule, while the ethyl groups are directed to the opposite side. The heterocyclic units are inclined by 62.4 (1) and 73.0 (1)$^\circ$ for 1a [78.9 (1) and 85.1 (1)$^\circ$ for 1b] with respect to the benzene ring. The cyclopentane rings adopt a slightly distorted envelope conformation with C33 (1a) and C31 (1b) as the flap.

3. Supramolecular features
The crystal structures of 1a and 1b are composed of inversion-symmetric dimers of 1:1 host–guest complexes (Fig. 3). The donor/acceptor properties of the solvent species have, however, a marked influence on the patterns of hydrogen-bonding interactions. In the crystal of 1a, the dimeric structural unit is held together by classical hydrogen bonds ($N5—H5/W, O1W—H2/W$; symmetry code as given in Table 1) that contribute to the formation of a cyclic supramolecular synthon with a graph-set motif $R_4^2(8)$. Within this dimeric unit, the oxygen atom of the water molecules acts as a trifurcated acceptor, as it is involved in the formation of an $O—H· · ·O$ bond [$d(H· · ·O) = 1.83 (1)$ Å] and two $N_{amine}—H· · ·O$ interactions [$d(H· · ·O) = 2.50 (1)$ and 2.52 (1) Å]. The H atoms of the water molecule participate in an asymmetric fashion in $O—H· · ·N$ bonding [$d(H· · ·N) = 2.03 (1)$ and 1.93 (1) Å] with pyridine atom N2 and amine atom N5,
respectively, of different host molecules. The interactions between the host molecules are confined to only one N—H⋯O hydrogen bond [$d(H⋯O) = 2.05 (1)$ Å] per molecule. In the crystal of 1a, the complexes are connected via C—H⋯π interactions [$d(H⋯Cg) = 2.69$ and $2.84$ Å], forming a three-dimensional network. An portion of the crystal structure is displayed in Fig. 4. The presence of the alcohol solvent in 1b reduces the number of hydrogen bonds within the dimeric structural unit (Table 2). In this case, the complex components create a continuous pattern of hydrogen bonds in the structure: N—H⋯O$_{\text{solvent}}$—H⋯N—H⋯O$_{\text{host}}$—H⋯N$_{\text{pyr}}$ [$d(H⋯O) = 2.01 (3)$ and $2.36 (3)$ Å; $d(H⋯N) = 1.90$ and $1.97$ Å]. While one of the amine hydrogen atoms is excluded from hydrogen bonding, a second one contributes by the formation of an intramolecular N—H⋯O bond. Cross-linking of the complexes via C—H⋯π and π—π interactions [Cg2⋯Cg2$''$ = 4.076 (2) Å; Cg2 is the centroid of the C14–C18/N2 ring; symmetry code: (iv) $-x + 2$, $-y + 1$, $-z + 1$] results in a three-dimensional supramolecular architecture. A view of the crystal structure along the $b$ axis reveals channel-like lattice voids in which the disordered solvent molecules are accommodated (Fig. 5).
4. Database survey

A search in the Cambridge Structural Database (CSD, Version 5.40, update of February 2019; Groom et al., 2016) for 1,3,5-trisubstituted 2,4,6-triarylbenzene derivatives containing the 4,6-dimethylpyridin-2-yl-aminomethyl-2,4,6-trimethylbenzene (refcode OAPVAF; Mazik et al., 2005), which has proven to be an effective receptor for the studied azines, was performed. In the CSD, five hits were found, which include 1,3,5-tris[(4,6-dimethylpyridin-2-yl)aminomethyl]-2,4,6-triethylbenzene (RAJZAE; Mazik et al., 2005), 1,3,5-tris[(4,6-dimethylpyridin-2-yl)aminomethyl]-3,5-trimethyl-2,4,6-triethylbenzene (RAJYUX; Mazik et al., 2004), and of 1,3,5-tris[(4,6-dimethylpyridin-2-yl)aminomethyl]-2,4,6-triethylbenzene (Mazik et al., 2004). In the crystals of the ethanol solvates (Mazik et al., 2004), the functionalized side arms of the corresponding host are arranged in an *aa*b fashion with respect to the benzene plane. In addition to the symmetrical trisubstituted trimethyl- and triethylbenzene derivatives, the crystal structures of the solvates of two triethylbenzene-based compounds containing one or two phenanthroline groups (ROKJEH, ROKJEH01; Mazik & Hartmann, 2008; Mazik et al., 2009) in addition to the 2-amino pyridine unit(s) have also been reported. In the case of 1-[N-(1,10-phenanthroline-2-yl-carbonyl)aminomethyl]-3-bis[4,6-dimethylpyridin-2-yl]aminomethyl]-2,4,6-triethylbenzene, three water molecules are located in the binding pocket created by the heterocyclic units of the host (Mazik & Hartmann, 2008), whereas the binding pocket of 1-bis[N-(1,10-phenanthroline-2-yl-carbonyl)aminomethyl]-5-[4,6-dimethylpyridin-2-yl]aminomethyl]-2,4,6-triethylbenzene is filled with one ethanol and two water molecules (Mazik et al., 2009). The above-mentioned aggregates are stabilized by eight and ten hydrogen bonds, respectively.

5. Synthesis and crystallization

To a solution of 1-amino-1-cyclopentylmethanol (1.48 mmol) in acetonitrile (20 ml) was added 1-bromomethyl-3,5-bis[4,6-dimethylpyridin-2-yl]aminomethyl]-2,4,6-triethylbenzene (0.71 mmol) dissolved in tetrahydrofuran/acetonitrile (20 ml, 1:1 v/v). The reaction mixture was stirred at room temperature and under exclusion of light. The completion of the reaction was monitored by TLC. After evaporation of the solvents in vacuo and purification of the yellowish crude product via column chromatography (SiO2; chloroform/methanol 7:1 v/v), compound II was obtained as a white solid. Yield: 33%; Rf = 0.26 (chloroform/methanol 7:1 v/v); m.p. 375 K. Crystals of 1a and 1b suitable for single crystal X-ray diffraction were grown by slow evaporation of the respective solvent (acetone in case of 1a) at ambient temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. N- and O-bound H atoms in 1a were located in a difference-Fourier map and refined freely with distance restraints of N—H = 0.89 (1) Å and O—H = 0.85 (1) Å. For 1b, N-bound H atoms were refined freely, while O-bound H atoms were treated as riding with O—H = 0.84 Å. All other H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.99 Å, and with C—H = 0.95 Å for methyl groups and C—H = 0.97 Å for aromatic H atoms. The crystal structure 1b contains highly disordered solvent molecules that could not be refined to an acceptable level. Thus, the SQUEEZE routine (Spek, 2015) in the PLATON (Spek, 2020) program was used to generate a modified data set in which the contribution of the disordered molecules to the structure amplitudes is eliminated. These solvent molecules are not considered in the given chemical formula. The void volume of 267.9 Å3 occupied by the disordered solvent represents 14.3% of the cell volume, and the calculated electron count was 65 per void.

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### Table 3
Experimental details.

|          | Ib                                                                 | lb                                                                 |
|----------|--------------------------------------------------------------------|--------------------------------------------------------------------|
| **Crystal data** |                                                      |                                                                   |
| Chemical formula | C_{35}H_{51}N_{5}O_{2}H_{2}O                                       | C_{35}H_{51}N_{5}O_{2}CH_{2}O                                      |
| $M_r$     | 575.82                                                             | 589.85                                                             |
| Crystal system, space group | Monoclinic, $P_{2}1/c$                                              | Triclinic, $P\bar{1}$                                             |
| Temperature (K) | 100                                                                | 100                                                                |
| $a$, $b$, $c$ (Å) | 13.2239 (7), 15.3576 (8), 17.1061 (8)                          | 12.1169 (4), 13.2380 (5), 13.6258 (5)                            |
| $\alpha$, $\beta$, $\gamma$ (°) | 90, 107.0289 (17), 90                                            | 68.373 (2), 79.379 (2), 67.392 (2)                               |
| $V$ (Å³)  | 3321.7 (3)                                                         | 1873.32 (12)                                                       |
| $Z$       | 4                                                                 | 2                                                                  |
| Radiation type | Mo $K\alpha$                                                      | Mo $K\alpha$                                                      |
| $\mu$ (mm$^{-1}$) | 0.07                                                               | 0.07                                                               |
| Crystal size (mm) | 0.45 x 0.32 x 0.10                                                | 0.56 x 0.34 x 0.32                                               |
| **Data collection** |                                                      |                                                                   |
| Diffractometer | Bruker X8 APEXII CCD                                              | Bruker X8 APEXII CCD                                              |
| No. of measured, independent and observed $| F > 2\sigma(F)|$ reflections | 28794, 7467, 5791                                                | 32191, 7654, 5160                                                |
| $R_{	ext{int}}$ | 0.034                                                             | 0.022                                                             |
| (sin $\theta$/\lambda)$_{\text{max}}$ (Å$^{-1}$) | 0.647                                                             | 0.626                                                             |
| **Refinement** |                                                      |                                                                   |
| $R[F^2 > 2\sigma(F^2)]$, w$R(F^2)$, $S$ | 0.043, 0.114, 1.03                                                | 0.065, 0.184, 1.21                                               |
| No. of reflections | 7467                                                               | 7654                                                              |
| No. of parameters | 410                                                                | 410                                                               |
| No. of restraints | 6                                                                 | 0                                                                  |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta$ρ$_\text{max}$, $\Delta$ρ$_\text{min}$ (e Å$^{-3}$) | 0.27, −0.20                                                       | 0.64, −0.39                                                      |

Computer programs: APEX2 and SAINT (Bruker, 2014), SHELX97 and SHELXTL (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015) and ORTEP-3 for Windows (Farrugia, 2012).

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Crystal structures of monohydrate and methanol solvate compounds of \(\text{1-[(3,5-bis][(4,6-dimethylpyridin-2-yl)amino]methyl]-2,4,6-triethylbenzyl)amino]cyclopentyl}methanol\)

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Computing details
For both structures, data collection: \textit{APEX2} (Bruker, 2014); cell refinement: \textit{SAINT} (Bruker, 2014); data reduction: \textit{SAINT} (Bruker, 2014); program(s) used to solve structure: \textit{SHELXS97} (Sheldrick, 2008); program(s) used to refine structure: \textit{SHELXL2014} (Sheldrick, 2015); molecular graphics: \textit{ORTEP-3 for Windows} (Farrugia, 2012); software used to prepare material for publication: \textit{SHELXTL} (Sheldrick, 2008).

\{1-[(3,5-Bis[(4,6-dimethylpyridin-2-yl)amino]methyl]-2,4,6-triethylbenzyl)amino]cyclopentyl}methanol monohydrate (1a)

| Crystal data | \(F(000) = 1256\) |
|--------------|------------------|
| \(C_{35}H_{51}N_{5}O\cdot H_{2}O\) | \(D_{s} = 1.151 \text{ Mg m}^{-3}\) |
| \(M_r = 575.82\) | Mo \(K\alpha\) radiation, \(\lambda = 0.71073 \text{ Å}\) |
| Monoclinic, \(P_{2_1}/c\) | Cell parameters from 9674 reflections |
| \(a = 13.2239\) (7) Å | \(\theta = 2.2-28.2^\circ\) |
| \(b = 15.3576\) (8) Å | \(\mu = 0.07 \text{ mm}^{-1}\) |
| \(c = 17.1061\) (8) Å | \(T = 100 \text{ K}\) |
| \(\beta = 107.0289\) (17)° | Irregular, colourless |
| \(V = 3321.7\) (3) Å\(^3\) | 0.45 × 0.32 × 0.10 mm |
| \(Z = 4\) |

| Data collection | \(R_{int} = 0.034\) |
|----------------|------------------|
| Bruker X8 APEXII CCD | \(\theta_{\text{max}} = 27.4^\circ, \theta_{\text{min}} = 2.1^\circ\) |
| diffractometer | \(h = -17 \rightarrow 17\) |
| phi and \(\omega\) scans | \(k = -19 \rightarrow 17\) |
| 28794 measured reflections | \(l = -22 \rightarrow 21\) |
| 7467 independent reflections | |
| 5791 reflections with \(I > 2\sigma(I)\) |

| Refinement | Hydrogen site location: mixed |
|------------|-----------------------------|
| Refinement on \(F^2\) | H atoms treated by a mixture of independent |
| Least-squares matrix: full | and constrained refinement |
| \(R[F^2 > 2\sigma(F^2)] = 0.043\) | \(w = 1/[\sigma(F_c^2) + (0.0551P)^2 + 0.8757P]\) |
| \(wR(F^2) = 0.114\) | where \(P = (F_c^2 + 2F_c^2)/3\) |
| \(S = 1.02\) | \((\Delta/\sigma)_{\text{max}} < 0.001\) |
| 7467 reflections | \(\Delta\rho_{\text{max}} = 0.27 \text{ e Å}^{-3}\) |
| 410 parameters | \(\Delta\rho_{\text{min}} = -0.20 \text{ e Å}^{-3}\) |
| 6 restraints | |
**Special details**

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

| Atom | X           | Y           | Z           | Uiso*/Ueq |
|------|-------------|-------------|-------------|-----------|
| O1   | -0.21807 (7)| 0.54810 (6) | 0.00959 (6) | 0.0238 (2)|
| N1   | -0.11044 (9)| 0.24557 (8) | -0.12255 (7)| 0.0218 (2)|
| H1   | -0.0834 (13)| 0.2969 (7)  | -0.1039 (10)| 0.036 (5)*|
| N2   | -0.23912 (9)| 0.33295 (7) | -0.20586 (6)| 0.0198 (2)|
| N3   | 0.33881 (9) | 0.29584 (8) | 0.05481 (7) | 0.0223 (3)|
| H3   | 0.3046 (12) | 0.4346 (7)  | 0.0466 (10) | 0.036 (5)*|
| N4   | 0.44662 (8) | 0.20100 (7) | 0.00817 (7) | 0.0201 (2)|
| N5   | -0.03784 (8)| 0.45806 (7) | 0.12196 (6) | 0.0176 (2)|
| H5   | -0.0752 (11)| 0.4402 (10)| 0.0720 (6)  | 0.024 (4)*|
| C1   | -0.03521 (10)| 0.26699 (8)| 0.06516 (7) | 0.0163 (3)|
| C2   | -0.00019 (10)| 0.21235 (8)| 0.01228 (7) | 0.0161 (3)|
| C3   | 0.10826 (10)| 0.20103 (8) | 0.02240 (7) | 0.0167 (3)|
| C4   | 0.18206 (10)| 0.24315 (8) | 0.08766 (7) | 0.0169 (3)|
| C5   | 0.14833 (10)| 0.30214 (8) | 0.13754 (7) | 0.0172 (3)|
| C6   | 0.03906 (10)| 0.31377 (8) | 0.12622 (7) | 0.0166 (3)|
| C7   | -0.15304 (10)| 0.27362 (9)| 0.05636 (8) | 0.0197 (3)|
| H7A  | -0.1936     | 0.2693      | -0.0022     | 0.024*    |
| H7B  | -0.1682     | 0.3311      | 0.0766      | 0.024*    |
| C8   | -0.18900 (11)| 0.20182 (10)| 0.10409 (9) | 0.0264 (3)|
| H8A  | -0.1753     | 0.1448      | 0.0835      | 0.040*    |
| H8B  | -0.2649     | 0.2080      | 0.0971      | 0.040*    |
| H8C  | -0.1499     | 0.2066      | 0.1622      | 0.040*    |
| C9   | 0.14599 (11)| 0.14569 (9) | -0.03707 (8)| 0.0223 (3)|
| H9A  | 0.2149      | 0.1684      | -0.0399     | 0.027*    |
| H9B  | 0.0949      | 0.1512      | -0.0922     | 0.027*    |
| C10  | 0.15839 (13)| 0.04917 (10)| -0.01346 (10)| 0.0335 (4)|
| H10A | 0.2087      | 0.0431      | 0.0412      | 0.050*    |
| H10B | 0.1847      | 0.0173      | -0.0532     | 0.050*    |
| H10C | 0.0897      | 0.0254      | -0.0133     | 0.050*    |
| C11  | 0.22863 (10)| 0.35482 (9) | 0.20189 (8) | 0.0215 (3)|
| H11A | 0.1992      | 0.4135      | 0.2052      | 0.026*    |
| H11B | 0.2932      | 0.3620      | 0.1845      | 0.026*    |
| C12  | 0.25915 (12)| 0.31327 (10)| 0.28712 (8) | 0.0288 (3)|
| H12A | 0.1950      | 0.3008      | 0.3028      | 0.043*    |
| H12B | 0.3042      | 0.3535      | 0.3268      | 0.043*    |
| H12C | 0.2977      | 0.2590      | 0.2863      | 0.043*    |
| C13  | -0.08201 (10)| 0.17532 (9)| -0.06179 (8)| 0.0190 (3)|
| H13A | -0.0524     | 0.1251      | -0.0840     | 0.023*    |
| H13B | -0.1451     | 0.1556      | -0.0468     | 0.023*    |
| Atom  | u1   | u2   | u3   | u4   |
|-------|------|------|------|------|
| C14   | -0.21009 (10) | 0.25244 (9) | -0.17714 (7) | 0.0181 (3) |
| C15   | -0.27565 (10) | 0.17971 (9) | -0.20134 (8) | 0.0206 (3) |
| H15   | -0.2528 | 0.1238 | -0.1796 | 0.025* |
| C16   | -0.37413 (11) | 0.19050 (9) | -0.25737 (9) | 0.0246 (3) |
| C17   | -0.40303 (11) | 0.27384 (9) | -0.28877 (8) | 0.0237 (3) |
| H17   | -0.4696 | 0.2832 | -0.3282 | 0.028* |
| C18   | -0.33386 (10) | 0.34256 (9) | -0.26202 (8) | 0.0205 (3) |
| C19   | -0.44869 (13) | 0.11508 (11) | -0.28255 (12) | 0.0432 (4) |
| H19A  | -0.4083 | 0.0607 | -0.2750 | 0.065* |
| H19B  | -0.4893 | 0.1213 | -0.3402 | 0.065* |
| H19C  | -0.4972 | 0.1140 | -0.2489 | 0.065* |
| C20   | -0.36075 (12) | 0.43342 (10) | -0.29491 (9) | 0.0282 (3) |
| H20A  | -0.3585 | 0.4732 | -0.2497 | 0.042* |
| H20B  | -0.4319 | 0.4338 | -0.3338 | 0.042* |
| H20C  | -0.3094 | 0.4523 | -0.3227 | 0.042* |
| C21   | 0.29964 (10) | 0.22967 (9) | 0.09957 (8) | 0.0201 (3) |
| H21A  | 0.3387 | 0.2332 | 0.1585 | 0.024* |
| H21B  | 0.3115 | 0.1711 | 0.0797 | 0.024* |
| C22   | 0.40895 (10) | 0.28164 (9) | 0.01095 (8) | 0.0191 (3) |
| C23   | 0.44048 (11) | 0.35362 (9) | -0.02740 (8) | 0.0227 (3) |
| H23   | 0.4112 | 0.4096 | -0.0245 | 0.027* |
| C24   | 0.51400 (11) | 0.34216 (9) | -0.06903 (9) | 0.0249 (3) |
| C25   | 0.55286 (11) | 0.25789 (9) | -0.07272 (9) | 0.0241 (3) |
| H25   | 0.6034 | 0.2474 | -0.1013 | 0.029* |
| C26   | 0.51739 (10) | 0.19036 (9) | -0.03474 (8) | 0.0217 (3) |
| C27   | 0.55221 (14) | 0.41751 (11) | -0.10917 (11) | 0.0386 (4) |
| H27A  | 0.5240 | 0.4122 | -0.1687 | 0.058* |
| H27B  | 0.6297 | 0.4171 | -0.0935 | 0.058* |
| H27C  | 0.5278 | 0.4723 | -0.0914 | 0.058* |
| C28   | 0.55646 (13) | 0.09887 (10) | -0.03838 (10) | 0.0306 (3) |
| H28A  | 0.5931 | 0.0787 | 0.0170 | 0.046* |
| H28B  | 0.6054 | 0.0978 | -0.0717 | 0.046* |
| H28C  | 0.4963 | 0.0605 | -0.0629 | 0.046* |
| C29   | -0.00203 (10) | 0.38089 (8) | 0.17448 (8) | 0.0190 (3) |
| H29A  | -0.0616 | 0.3563 | 0.1914 | 0.023* |
| H29B  | 0.0547 | 0.3976 | 0.2243 | 0.023* |
| C30   | -0.10008 (10) | 0.52576 (8) | 0.14971 (8) | 0.0192 (3) |
| C31   | -0.02821 (11) | 0.57698 (9) | 0.22365 (8) | 0.0239 (3) |
| H31A  | 0.0443 | 0.5526 | 0.2400 | 0.029* |
| H31B  | -0.0245 | 0.6391 | 0.2094 | 0.029* |
| C32   | -0.07871 (14) | 0.56730 (11) | 0.29311 (9) | 0.0360 (4) |
| H32A  | -0.0488 | 0.5167 | 0.3281 | 0.043* |
| H32B  | -0.0677 | 0.6204 | 0.3274 | 0.043* |
| C33   | -0.19561 (13) | 0.55366 (10) | 0.24928 (10) | 0.0341 (4) |
| H33A  | -0.2308 | 0.6094 | 0.2284 | 0.041* |
| H33B  | -0.2323 | 0.5265 | 0.2859 | 0.041* |
| C34   | -0.19394 (11) | 0.49275 (9) | 0.17924 (8) | 0.0230 (3) |
| H34A  | -0.2613 | 0.4962 | 0.1346 | 0.028* |
### Atomic displacement parameters (Å²)

|   | \(U_{11}^2\) | \(U_{22}^2\) | \(U_{33}^2\) | \(U_{12}^2\) | \(U_{13}^2\) | \(U_{23}^2\) |
|---|---|---|---|---|---|---|
| O1 | 0.0248 (5) | 0.0247 (5) | 0.0211 (5) | 0.0028 (4) | 0.0057 (4) | −0.0022 (4) |
| N1 | 0.0216 (6) | 0.0227 (6) | 0.0174 (5) | −0.0049 (5) | 0.0001 (5) | 0.0022 (5) |
| N2 | 0.0228 (6) | 0.0218 (6) | 0.0145 (5) | −0.0014 (4) | 0.0050 (4) | −0.0001 (5) |
| N3 | 0.0219 (6) | 0.0215 (6) | 0.0262 (6) | 0.0011 (5) | 0.0114 (5) | 0.0022 (5) |
| N4 | 0.0202 (5) | 0.0211 (6) | 0.0181 (5) | −0.0012 (4) | 0.0044 (4) | 0.0001 (5) |
| N5 | 0.0225 (5) | 0.0153 (5) | 0.0154 (5) | 0.0005 (4) | 0.0063 (4) | −0.0017 (4) |
| C1 | 0.0194 (6) | 0.0137 (6) | 0.0158 (6) | 0.0003 (5) | 0.0052 (5) | 0.0049 (5) |
| C2 | 0.0203 (6) | 0.0213 (6) | 0.0143 (6) | −0.0016 (5) | 0.0031 (5) | 0.0026 (5) |
| C3 | 0.0225 (6) | 0.0141 (6) | 0.0145 (6) | 0.0010 (5) | 0.0069 (5) | 0.0029 (5) |
| C4 | 0.0181 (6) | 0.0172 (6) | 0.0150 (6) | 0.0002 (5) | 0.0045 (5) | 0.0045 (5) |
| C5 | 0.0207 (6) | 0.0165 (6) | 0.0135 (6) | −0.0013 (5) | 0.0036 (5) | 0.0025 (5) |
| C6 | 0.0213 (6) | 0.0145 (6) | 0.0145 (6) | 0.0000 (5) | 0.0060 (5) | 0.0016 (5) |
| C7 | 0.0196 (6) | 0.0179 (6) | 0.0214 (7) | 0.0000 (5) | 0.0058 (5) | −0.0004 (5) |
| C8 | 0.0254 (7) | 0.0261 (8) | 0.0303 (8) | −0.0017 (6) | 0.0121 (6) | 0.0027 (6) |
| C9 | 0.0238 (7) | 0.0241 (7) | 0.0199 (7) | 0.0007 (5) | 0.0079 (5) | −0.0034 (6) |
| C10 | 0.0371 (8) | 0.0227 (8) | 0.0425 (9) | 0.0041 (6) | 0.0144 (7) | −0.0069 (7) |
| C11 | 0.0207 (6) | 0.0236 (7) | 0.0189 (6) | −0.0018 (5) | 0.0037 (5) | −0.0033 (6) |
| C12 | 0.0301 (7) | 0.0345 (8) | 0.0177 (7) | −0.0014 (6) | 0.0004 (6) | −0.0021 (6) |
| C13 | 0.0215 (6) | 0.0177 (6) | 0.0160 (6) | −0.0013 (5) | 0.0029 (5) | −0.0004 (5) |
| C14 | 0.0196 (6) | 0.0229 (7) | 0.0126 (6) | 0.0000 (5) | 0.0059 (5) | −0.0015 (5) |
| C15 | 0.0232 (7) | 0.0178 (7) | 0.0196 (8) | 0.0012 (5) | 0.0045 (5) | −0.0040 (5) |
| C16 | 0.0238 (7) | 0.0239 (7) | 0.0235 (7) | −0.0014 (6) | 0.0028 (6) | −0.0058 (6) |
| C17 | 0.0199 (6) | 0.0283 (8) | 0.0197 (7) | 0.0018 (5) | 0.0009 (5) | −0.0020 (6) |
| C18 | 0.0229 (6) | 0.0239 (7) | 0.0153 (6) | 0.0018 (5) | 0.0068 (5) | −0.0001 (5) |
| C19 | 0.0332 (8) | 0.0283 (9) | 0.0527 (10) | −0.0068 (7) | −0.0115 (8) | −0.0018 (8) |
| C20 | 0.0313 (8) | 0.0271 (8) | 0.0244 (7) | 0.0014 (6) | 0.0052 (6) | 0.0053 (6) |
| C21 | 0.0192 (6) | 0.0227 (7) | 0.0180 (6) | 0.0015 (5) | 0.0048 (5) | 0.0018 (5) |
| C22 | 0.0169 (6) | 0.0230 (7) | 0.0160 (6) | −0.0008 (5) | 0.0024 (5) | −0.0014 (5) |
| C23 | 0.0255 (7) | 0.0195 (7) | 0.0238 (7) | 0.0014 (5) | 0.0084 (6) | 0.0003 (6) |
| C24 | 0.0278 (7) | 0.0245 (7) | 0.0241 (7) | −0.0009 (6) | 0.0101 (6) | 0.0014 (6) |
| C25 | 0.0244 (7) | 0.0266 (7) | 0.0239 (7) | 0.0020 (6) | 0.0113 (6) | −0.0018 (6) |
| C26 | 0.0232 (7) | 0.0229 (7) | 0.0183 (6) | 0.0009 (5) | 0.0048 (5) | −0.0025 (6) |
| C27 | 0.0504 (10) | 0.0276 (8) | 0.0482 (10) | 0.0014 (7) | 0.0308 (9) | 0.0057 (7) |
| C28 | 0.0370 (8) | 0.0240 (8) | 0.0341 (8) | 0.0038 (6) | 0.0158 (7) | 0.0005 (6) |
| C29 | 0.0222 (6) | 0.0186 (7) | 0.0161 (6) | 0.0013 (5) | 0.0056 (5) | 0.0007 (5) |
| C30 | 0.0229 (6) | 0.0160 (6) | 0.0200 (6) | 0.0002 (5) | 0.0081 (5) | −0.0019 (5) |
Geometric parameters (Å, °)

|       |       |       |
|-------|-------|-------|
| O1—C35 | 1.4275 (16) | C15—H15 | 0.9500 |
| O1—H1A  | 0.874 (9) | C16—C17 | 1.397 (2) |
| N1—C14  | 1.3779 (16) | C16—C19 | 1.500 (2) |
| N1—C13  | 1.4685 (17) | C17—C18 | 1.3837 (19) |
| N1—H1   | 0.885 (9) | C17—H17 | 0.9500 |
| N2—C14  | 1.3443 (17) | C18—C20 | 1.5076 (19) |
| N2—C18  | 1.3451 (17) | C19—H19A | 0.9800 |
| N3—C22  | 1.3705 (17) | C19—H19B | 0.9800 |
| N3—C21  | 1.4558 (17) | C19—H19C | 0.9800 |
| N3—H3   | 0.889 (9) | C20—H20A | 0.9800 |
| N4—C22  | 1.3410 (17) | C20—H20B | 0.9800 |
| N4—C26  | 1.3578 (17) | C20—H20C | 0.9800 |
| N5—C29  | 1.4787 (16) | C21—H21A | 0.9900 |
| N5—C30  | 1.4876 (16) | C21—H21B | 0.9900 |
| N5—H5   | 0.896 (9) | C22—C23 | 1.4093 (19) |
| C1—C6   | 1.4039 (17) | C23—C24 | 1.3744 (19) |
| C1—C2   | 1.4080 (18) | C23—H23 | 0.9500 |
| C1—C7   | 1.5243 (17) | C24—C25 | 1.401 (2) |
| C2—C3   | 1.4041 (17) | C24—C27 | 1.506 (2) |
| C2—C13  | 1.5155 (17) | C25—C26 | 1.377 (2) |
| C3—C4   | 1.4067 (18) | C25—H25 | 0.9500 |
| C3—C9   | 1.5177 (18) | C26—C28 | 1.5046 (19) |
| C4—C5   | 1.4037 (18) | C27—H27A | 0.9800 |
| C4—C21  | 1.5219 (17) | C27—H27B | 0.9800 |
| C5—C6   | 1.4120 (17) | C27—H27C | 0.9800 |
| C5—C11  | 1.5197 (17) | C28—H28A | 0.9800 |
| C6—C29  | 1.5172 (18) | C28—H28B | 0.9800 |
| C7—C8   | 1.5284 (19) | C28—H28C | 0.9800 |
| C7—H7A  | 0.9900 | C29—H29A | 0.9900 |
| C7—H7B  | 0.9900 | C29—H29B | 0.9900 |
| C8—H8A  | 0.9800 | C30—C35 | 1.5310 (18) |
| C8—H8B  | 0.9800 | C30—C31 | 1.5548 (18) |
| C8—H8C  | 0.9800 | C30—C34 | 1.5556 (18) |
| C9—C10  | 1.532 (2) | C31—C32 | 1.531 (2) |
| C9—H9A  | 0.9900 | C31—H31A | 0.9900 |
| C9—H9B  | 0.9900 | C31—H31B | 0.9900 |
| C10—H10A | 0.9800 | C32—C33 | 1.522 (2) |
| C10—H10B | 0.9800 | C32—H32A | 0.9900 |
| C10—H10C | 0.9800 | C32—H32B | 0.9900 |
C11—C12 1.5333 (19)  C33—C34 1.525 (2)
C11—H11A 0.9900  C33—H33A 0.9900
C11—H11B 0.9900  C33—H33B 0.9900
C12—H12A 0.9800  C34—H34A 0.9900
C12—H12B 0.9800  C34—H34B 0.9900
C12—H12C 0.9800  C35—H35A 0.9900
C13—H13A 0.9900  C35—H35B 0.9900
C13—H13B 0.9900  O1W—H1W 0.861 (9)
C14—C15 1.4001 (18)  O1W—H2W 0.871 (9)
C15—C16 1.3824 (18)

C35—O1—H1A 112.5 (13)  C16—C19—H19B 109.5
C14—N1—C13 122.12 (11)  H19A—C19—H19B 109.5
C14—N1—H1 112.4 (11)  C16—C19—H19C 109.5
C13—N1—H1 114.0 (11)  H19A—C19—H19C 109.5
C14—N2—C18 117.94 (11)  C16—C19—H19B 109.5
C22—N3—C21 116.6 (11)  C18—C20—H20A 109.5
C22—N3—H3 116.7 (11)  C18—C20—H20B 109.5
C21—N3—H3 116.7 (11)  C18—C20—H20C 109.5
C22—N4—C26 116.90 (11)  H20A—C20—H20B 109.5
C29—N5—C30 118.35 (10)  H20A—C20—H20C 109.5
C29—N5—H5 108.8 (10)  H20B—C20—H20C 109.5
C30—N5—H5 108.4 (10)  N3—C21—C4 109.97 (10)
C6—C1—C2 119.53 (11)  N3—C21—H21A 109.7
C6—C1—C7 120.77 (11)  C4—C21—H21A 109.7
C2—C1—C7 119.70 (11)  N3—C21—H21B 109.7
C3—C2—C1 120.72 (11)  C4—C21—H21B 109.7
C3—C2—C13 120.66 (11)  H21A—C21—H21B 108.2
C1—C2—C13 118.17 (11)  N4—C22—N3 118.97 (12)
C2—C3—C4 119.15 (11)  N4—C22—C23 122.97 (12)
C2—C3—C9 120.74 (11)  N3—C22—C23 118.03 (12)
C4—C3—C9 120.10 (11)  C24—C23—C22 119.43 (13)
C5—C4—C3 120.60 (11)  C24—C23—H23 120.3
C5—C4—C21 120.03 (11)  C22—C23—H23 120.3
C3—C4—C21 119.19 (11)  C22—C23—C25 117.79 (13)
C4—C5—C6 119.53 (11)  C23—C24—C25 121.36 (13)
C4—C5—C11 120.33 (11)  C25—C24—C27 120.85 (13)
C6—C5—C11 120.12 (11)  C26—C25—C24 119.63 (12)
C1—C6—C5 120.14 (11)  C26—C25—H25 120.2
C1—C6—C29 117.93 (11)  C24—C25—H25 120.2
C5—C6—C29 121.78 (11)  N4—C26—C25 123.26 (12)
C1—C7—C8 111.59 (11)  N4—C26—C28 115.73 (12)
C1—C7—H7A 109.3  C25—C26—C28 121.01 (12)
C8—C7—H7A 109.3  C24—C27—H27A 109.5
C1—C7—H7B 109.3  C24—C27—H27B 109.5
C8—C7—H7B 109.3  H27A—C27—H27B 109.5
H7A—C7—H7B 108.0  C24—C27—H27C 109.5
C7—C8—H8A 109.5  H27A—C27—H27C 109.5
| Bond                  | Angle   | Bond                  | Angle   | Bond                  | Angle   |
|----------------------|---------|----------------------|---------|----------------------|---------|
| C7—C8—H8B           | 109.5   | H27B—C27—H27C       | 109.5   |
| H8A—C8—H8B          | 109.5   | C26—C28—H28A        | 109.5   |
| C7—C8—H8C           | 109.5   | C26—C28—H28B        | 109.5   |
| H8A—C8—H8C          | 109.5   | H28A—C28—H28B       | 109.5   |
| H8B—C8—H8C          | 109.5   | C26—C28—H28C        | 109.5   |
| C3—C9—C10           | 113.32 (11) | H28A—C28—H28C       | 109.5   |
| C3—C9—H9A           | 108.9   | H28B—C28—H28C       | 109.5   |
| C10—C9—H9A          | 108.9   | N5—C29—C6           | 108.41 (10) |
| C3—C9—H9B           | 108.9   | N5—C29—H29A         | 110.0   |
| C10—C9—H9B          | 108.9   | C6—C29—H29A         | 110.0   |
| H9A—C9—H9B          | 107.7   | N5—C29—H29B         | 110.0   |
| C9—C10—H10A         | 109.5   | C6—C29—H29B         | 110.0   |
| C9—C10—H10B         | 109.5   | H29A—C29—H29B       | 108.4   |
| H10A—C10—H10B       | 109.5   | N5—C30—C35          | 106.05 (10) |
| C9—C10—H10C         | 109.5   | N5—C30—C31          | 110.47 (11) |
| H10A—C10—H10C       | 109.5   | C35—C30—C31         | 109.58 (11) |
| H10B—C10—H10C       | 109.5   | N5—C30—C34          | 116.39 (11) |
| C5—C11—C12          | 113.62 (11) | C35—C30—C34         | 109.43 (11) |
| C5—C11—H11A         | 108.8   | C31—C30—C34         | 104.86 (10) |
| C12—C11—H11A        | 108.8   | C32—C31—C30         | 106.29 (11) |
| C5—C11—H11B         | 108.8   | C32—C31—H31A        | 110.5   |
| C12—C11—H11B        | 108.8   | C30—C31—H31A        | 110.5   |
| H11A—C11—H11B       | 107.7   | C32—C31—H31B        | 110.5   |
| C11—C12—H12A        | 109.5   | C30—C31—H31B        | 110.5   |
| C11—C12—H12B        | 109.5   | H31A—C31—H31B       | 108.7   |
| H12A—C12—H12B       | 109.5   | C33—C32—C31         | 104.00 (12) |
| C11—C12—H12C        | 109.5   | C33—C32—H32A        | 111.0   |
| H12A—C12—H12C       | 109.5   | C31—C32—H32A        | 111.0   |
| H12B—C12—H12C       | 109.5   | C33—C32—H32B        | 111.0   |
| N1—C13—C2           | 106.75 (10) | C31—C32—H32B        | 111.0   |
| N1—C13—H13A         | 110.4   | H32A—C32—H32B       | 109.0   |
| C2—C13—H13A         | 110.4   | C32—C33—C34         | 103.01 (12) |
| N1—C13—H13B         | 110.4   | C32—C33—H33A        | 111.2   |
| C2—C13—H13B         | 110.4   | C34—C33—H33A        | 111.2   |
| H13A—C13—H13B       | 108.6   | C32—C33—H33B        | 111.2   |
| N2—C14—N1           | 115.60 (11) | C34—C33—H33B        | 111.2   |
| N2—C14—C15          | 122.67 (12) | H33A—C33—H33B       | 109.1   |
| N1—C14—C15          | 121.72 (12) | C33—C34—C30         | 104.58 (11) |
| C16—C15—C14         | 119.04 (13) | C33—C34—H34A        | 110.8   |
| C16—C15—H15         | 120.5   | C30—C34—H34A        | 110.8   |
| C14—C15—H15         | 120.5   | C33—C34—H34B        | 110.8   |
| C15—C16—C17         | 118.19 (12) | C30—C34—H34B        | 110.8   |
| C15—C16—C19         | 120.87 (13) | H34A—C34—H34B       | 108.9   |
| C17—C16—C19         | 120.93 (13) | O1—C35—C30          | 113.25 (11) |
| C18—C17—C16         | 119.44 (12) | O1—C35—H35A         | 108.9   |
| C18—C17—H17         | 120.3   | C30—C35—H35A        | 108.9   |
| C16—C17—H17         | 120.3   | O1—C35—H35B         | 108.9   |
| N2—C18—C17          | 122.67 (13) | C30—C35—H35B        | 108.9   |
| Bond                  | Dihedral Angle | Bond                  | Dihedral Angle |
|----------------------|---------------|----------------------|---------------|
| N2—C18—C20          | 115.89 (12)   | H35A—C35—H35B        | 107.7         |
| C17—C18—C20         | 121.45 (12)   | H1W—O1W—H2W          | 107.2 (18)    |
| C16—C19—H19A        | 109.5         |                      |               |
| C6—C1—C2—C3         | 3.01 (18)     | C15—C16—C17—C18     | −1.2 (2)      |
| C7—C1—C2—C3         | −176.05 (11)  | C19—C16—C17—C18     | 177.68 (14)   |
| C6—C1—C2—C13        | −169.44 (11)  | C14—N2—C18—C17      | 2.26 (18)     |
| C7—C1—C2—C13        | 11.50 (17)    | C14—N2—C18—C20      | −177.50 (11)  |
| C1—C2—C3—C4         | 1.77 (18)     | C16—C17—C18—N2      | −0.6 (2)      |
| C13—C2—C3—C4        | 174.02 (11)   | C16—C17—C18—C20     | 179.14 (13)   |
| C1—C2—C3—C9         | −4.72 (18)    | C22—N3—C21—C4       | −139.10 (13)  |
| C13—C2—C3—C9        | −5.90 (18)    | C5—C4—C21—N3        | −84.62 (14)   |
| C2—C3—C4—C5         | 172.84 (11)   | N3—C22—C23—C24      | 90.57 (14)    |
| C9—C3—C4—C5         | −178.80 (11)  | C26—N4—C22—C23      | −0.6 (18)     |
| C2—C3—C4—C21        | 178.94 (11)   | C26—N4—C22—C23      | −0.42 (19)    |
| C9—C3—C4—C21        | −2.32 (18)    | C21—N3—C22—N4       | −178.64 (12)  |
| C3—C4—C5—C6         | 5.22 (18)     | C21—N3—C22—C23      |               |
| C21—C4—C5—C6        | −179.67 (11)  | N4—C22—C23—C24      | −0.8 (2)      |
| C3—C4—C5—C11        | −173.26 (11)  | N3—C22—C23—C24      | 177.36 (12)   |
| C21—C4—C5—C11       | 1.85 (18)     | C22—C23—C24—C25     | 1.3 (2)       |
| C2—C1—C6—C5         | −3.71 (18)    | C22—C23—C24—C27     | −178.42 (14)  |
| C7—C1—C6—C5         | 175.34 (11)   | C23—C24—C25—C26     | −0.4 (2)      |
| C2—C1—C6—C29        | 171.98 (11)   | C27—C24—C25—C26     | 179.31 (14)   |
| C7—C1—C6—C29        | −8.97 (17)    | C22—N4—C26—C25      | 1.63 (19)     |
| C4—C5—C6—C1         | −0.36 (18)    | C22—N4—C26—C28      | −178.68 (12)  |
| C11—C5—C6—C1        | 178.12 (11)   | C24—C25—C26—N4      | −1.1 (2)      |
| C4—C5—C6—C29        | −175.88 (11)  | C24—C25—C26—C28     | 179.23 (13)   |
| C11—C5—C6—C29       | 2.60 (18)     | C30—N5—C29—C6       | 168.54 (10)   |
| C6—C1—C7—C8         | −93.07 (14)   | C1—C6—C29—N5        | −74.81 (14)   |
| C2—C1—C7—C8         | 85.97 (14)    | C5—C6—C29—N5        | 100.80 (13)   |
| C2—C3—C9—C10        | −88.30 (15)   | C29—N5—C30—C35      | −170.13 (10)  |
| C4—C3—C9—C10        | 92.98 (15)    | C29—N5—C30—C31      | 71.21 (14)    |
| C4—C5—C11—C12       | −94.72 (14)   | C29—N5—C30—C34      | −48.19 (15)   |
| C6—C5—C11—C12       | 86.81 (15)    | N5—C30—C31—C32      | −122.98 (12)  |
| C14—N1—C13—C2       | −148.51 (12)  | C35—C30—C31—C32     | 120.53 (13)   |
| C3—C2—C13—N1        | −95.00 (13)   | C34—C30—C31—C32     | 3.17 (15)     |
| C1—C2—C13—N1        | 77.45 (14)    | C30—C31—C32—C33     | −27.13 (16)   |
| C18—N2—C14—N1       | 177.18 (11)   | C31—C32—C33—C34     | 40.92 (15)    |
| C18—N2—C14—C15      | −2.14 (18)    | C32—C33—C34—C30     | −38.95 (15)   |
| C13—N1—C14—N2       | 154.81 (12)   | N5—C30—C34—C33      | 144.32 (12)   |
| C13—N1—C14—C15      | −25.87 (18)   | C35—C30—C34—C33     | −95.53 (13)   |
| N2—C14—C15—C16      | 0.36 (19)     | C31—C30—C34—C33     | 21.93 (14)    |
| N1—C14—C15—C16      | −178.91 (12)  | N5—C30—C35—O1       | 67.44 (13)    |
| C14—C15—C16—C17     | 1.31 (19)     | C31—C30—C35—O1      | −173.32 (11)  |
| C14—C15—C16—C19     | −177.57 (14)  | C34—C30—C35—O1      | −58.86 (14)   |
Hydrogen-bond geometry (Å, °)

Cg1 and Cg2 are the centroids of the C1–C6 and C14–C18/N2 rings, respectively.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|-------|---------|
| O1W—H2W···N5 | 0.87 (1) | 1.93 (1) | 2.8018 (14) | 175 (2) |
| O1W—H1W···N2i | 0.86 (1) | 2.03 (1) | 2.8780 (15) | 166 (2) |
| O1—H1A···O1W | 0.87 (1) | 1.83 (1) | 2.6993 (14) | 176 (2) |
| N5—H5···O1W | 0.90 (1) | 2.52 (1) | 3.3302 (14) | 151 (1) |
| N3—H3···O1 | 0.89 (1) | 2.05 (1) | 2.9115 (15) | 162 (2) |
| N1—H1···O1W | 0.89 (1) | 2.50 (1) | 3.2618 (16) | 145 (1) |
| C32—H32B···Cg1ii | 0.99 | 2.69 | 3.666 (2) | 169 |
| C25—H25···Cg2iii | 0.95 | 2.84 | 3.728 (2) | 156 |

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

{1-[(3,5-Bis[(4,6-dimethylpyridin-2-yl)amino]methyl)-2,4,6-triethylbenzyl]amino[cyclopentyl)methanol methanol monosolvate (1b)}

Crystal data

C_{35}H_{51}N_{5}O · CH_{4}O  
M_r = 589.85  
Triclinic, P  
a = 12.1169 (4) Å  
b = 13.2380 (5) Å  
c = 13.6258 (5) Å  
α = 68.373 (2)°  
β = 79.379 (2)°  
γ = 67.392 (2)°  
V = 1873.32 (12) Å³  
Z = 2  
F(000) = 644  
D_x = 1.046 Mg m⁻³  
Mo Kα radiation, λ = 0.71073 Å  
Cell parameters from 9915 reflections  
θ = 2.8–30.5°  
µ = 0.07 mm⁻¹  
T = 100 K  
Irregular, colourless  
0.56 × 0.34 × 0.32 mm

Data collection

Bruker X8 APEXII CCD  
diffraclacter  
ϕ and ω scans  
32191 measured reflections  
7654 independent reflections  
5160 reflections with I > 2σ(I)

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.065  
wR(F²) = 0.184  
S = 1.21  
7654 reflections  
410 parameters  
0 restraints

H atoms treated by a mixture of independent and constrained refinement

Hydrogen site location: mixed

Δρ_{max} = 0.64 e Å⁻³  
Δρ_{min} = −0.39 e Å⁻³
**Special details**

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

|    | x     | y     | z     | Uiso*/Ueq |
|----|-------|-------|-------|-----------|
| O1 | 1.27014 (18) | 0.57360 (19) | 0.78975 (18) | 0.0337 (5) |
| H1A| 1.3308 | 0.5159 | 0.7868 | 0.051* |
| N1 | 0.8131 (3) | 0.6398 (2) | 0.68505 (19) | 0.0298 (6) |
| H1 | 0.848 (3) | 0.588 (3) | 0.750 (3) | 0.030 (8)* |
| N2 | 0.7667 (3) | 0.6725 (3) | 0.5157 (2) | 0.0382 (7) |
| N3 | 0.6133 (2) | 0.73074 (19) | 1.09127 (18) | 0.0208 (5) |
| H3 | 0.656 (3) | 0.667 (3) | 1.081 (2) | 0.023 (8)* |
| N4 | 0.53696 (19) | 0.61277 (18) | 1.23326 (17) | 0.0182 (4) |
| N5 | 1.12190 (19) | 0.67304 (18) | 0.94010 (18) | 0.0190 (4) |
| H5 | 1.110 (3) | 0.628 (3) | 0.910 (3) | 0.027 (8)* |
| C1 | 0.9042 (2) | 0.7808 (2) | 0.78008 (19) | 0.0159 (5) |
| C2 | 0.7918 (2) | 0.7816 (2) | 0.76743 (19) | 0.0179 (5) |
| C3 | 0.6984 (2) | 0.7982 (2) | 0.8449 (2) | 0.0186 (5) |
| C4 | 0.7184 (2) | 0.8192 (2) | 0.93271 (19) | 0.0173 (5) |
| C5 | 0.8303 (2) | 0.8189 (2) | 0.94606 (19) | 0.0164 (5) |
| C6 | 0.9235 (2) | 0.7990 (2) | 0.86997 (19) | 0.0160 (5) |
| C7 | 1.0021 (2) | 0.7657 (2) | 0.6940 (2) | 0.0207 (5) |
| H7A| 0.9917 | 0.7167 | 0.6589 | 0.025* |
| C8 | 1.0809 | 0.7252 | 0.7264 | 0.025* |
| H8A| 1.0008 (3) | 0.8814 (2) | 0.6114 (2) | 0.0287 (6) |
| H9A| 0.9235 | 0.9212 | 0.5781 | 0.043* |
| H8B| 1.0652 | 0.8677 | 0.5575 | 0.043* |
| H9B| 1.0127 | 0.9296 | 0.6456 | 0.043* |
| H8C| 0.5782 (2) | 0.7944 (2) | 0.8331 (2) | 0.0249 (6) |
| C9 | 0.5417 | 0.7656 | 0.9043 | 0.030* |
| H9C| 0.5909 | 0.7389 | 0.7960 | 0.030* |
| C10| 0.4912 (3) | 0.9125 (3) | 0.7722 (3) | 0.0349 (7) |
| C10A| 0.4798 | 0.9686 | 0.8075 | 0.052* |
| H10B| 0.4141 | 0.9056 | 0.7701 | 0.052* |
| H10C| 0.5241 | 0.9391 | 0.6999 | 0.052* |
| C11| 0.8503 (2) | 0.8411 (2) | 1.0417 (2) | 0.0195 (5) |
| H11A| 0.9340 | 0.7959 | 1.0622 | 0.023* |
| C12| 0.7971 | 0.8138 | 1.1016 | 0.023* |
| H12A| 0.8259 (3) | 0.9694 (2) | 1.0213 (2) | 0.0283 (6) |
| H12B| 0.8792 | 0.9967 | 0.9628 | 0.042* |
| H12C| 0.8406 | 0.9791 | 1.0851 | 0.042* |
| C13| 0.7709 (3) | 0.7627 (2) | 0.6698 (2) | 0.0240 (6) |
| H13A| 0.6844 | 0.7971 | 0.6565 | 0.029* |
H13B 0.8140 0.8016 0.6074 0.029*
C14 0.8157 (3) 0.5977 (3) 0.6068 (2) 0.0301 (7)
C15 0.8695 (3) 0.4779 (3) 0.6252 (3) 0.0351 (7)
H15 0.9032 0.4270 0.6910 0.042*
C16 0.8722 (3) 0.4360 (4) 0.5462 (3) 0.0480 (10)
C17 0.8216 (4) 0.5129 (4) 0.4515 (3) 0.0548 (12)
H17 0.8222 0.4854 0.3960 0.066*
C18 0.7710 (3) 0.6284 (4) 0.4385 (3) 0.0516 (11)
C19 0.9332 (4) 0.3088 (4) 0.5622 (4) 0.0662 (14)
H19A 0.8931 0.2853 0.5225 0.099*
H19B 0.9289 0.2646 0.6375 0.099*
H19C 1.0172 0.2936 0.5368 0.099*
C20 0.7149 (5) 0.7156 (5) 0.3377 (3) 0.0755 (16)
H20A 0.6293 0.7531 0.3520 0.113*
H20B 0.7252 0.6763 0.2863 0.113*
H20C 0.7535 0.7741 0.3089 0.113*
C21 0.6162 (2) 0.8401 (2) 1.0146 (2) 0.0200 (5)
H21A 0.6267 0.8879 1.0510 0.024*
H21B 0.5393 0.8828 0.9791 0.024*
C22 0.5286 (2) 0.7214 (2) 1.1721 (19) 0.0168 (5)
C23 0.4398 (2) 0.8186 (2) 1.1916 (2) 0.0183 (5)
H23 0.4344 0.8945 1.1460 0.022*
C24 0.3604 (2) 0.8028 (2) 1.2778 (2) 0.0206 (5)
C25 0.3712 (2) 0.6901 (2) 1.3418 (2) 0.0226 (5)
H25 0.3185 0.6767 1.4022 0.027*
C26 0.4592 (2) 0.5973 (2) 1.3170 (2) 0.0211 (5)
C27 0.2644 (3) 0.9050 (2) 1.3014 (2) 0.0293 (6)
H27A 0.2900 0.9726 1.2716 0.044*
H27B 0.2505 0.8873 1.3781 0.044*
H27C 0.1902 0.9218 1.2698 0.044*
C28 0.4714 (3) 0.4746 (2) 1.3806 (2) 0.0282 (6)
H28A 0.4429 0.4419 1.3406 0.042*
H28B 0.4236 0.4724 1.4472 0.042*
H28C 0.5556 0.4291 1.3956 0.042*
C29 1.0471 (2) 0.7940 (2) 0.8843 (2) 0.0191 (5)
H29A 1.0858 0.8231 0.8144 0.023*
H29B 1.0398 0.8442 0.9257 0.023*
C30 1.2540 (2) 0.6488 (2) 0.9314 (2) 0.0212 (5)
C31 1.2823 (2) 0.7329 (2) 0.9663 (2) 0.0277 (6)
H31A 1.3654 0.7311 0.9441 0.033*
H31B 1.2264 0.8130 0.9367 0.033*
C32 1.2660 (3) 0.6884 (3) 1.0860 (3) 0.0313 (7)
H32A 1.1801 0.7099 1.1090 0.038*
H32B 1.3061 0.7192 1.1194 0.038*
C33 1.3256 (3) 0.5566 (3) 1.1131 (2) 0.0308 (6)
H33A 1.2857 0.5159 1.1770 0.037*
H33B 1.4113 0.5318 1.1261 0.037*
C34 1.3113 (2) 0.5300 (2) 1.0158 (2) 0.0265 (6)
|     |  
|-----|-----|-----|-----|-----|-----|-----|-----|
|     | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
| O1  | 0.0203 (10) | 0.0337 (12) | 0.0483 (13) | 0.0011 (9) | 0.0023 (9) | $-0.0275$ (11) |
| N1  | 0.0507 (16) | 0.0266 (13) | 0.0192 (12) | $-0.0167$ (12) | $-0.0054$ (11) | $-0.0102$ (10) |
| N2  | 0.0466 (16) | 0.0630 (19) | 0.0195 (12) | $-0.0329$ (15) | $-0.0007$ (11) | $-0.0154$ (12) |
| N3  | 0.0201 (11) | 0.0157 (11) | 0.0223 (11) | $-0.0041$ (9) | 0.0068 (9) | $-0.0074$ (9) |
| N4  | 0.0167 (10) | 0.0180 (10) | 0.0200 (11) | $-0.0060$ (8) | 0.0019 (8) | $-0.0075$ (9) |
| N5  | 0.0153 (10) | 0.0168 (10) | 0.0252 (11) | $-0.0034$ (8) | $-0.0012$ (8) | $-0.0094$ (9) |
| C1  | 0.0184 (12) | 0.0112 (11) | 0.0143 (11) | $-0.0024$ (9) | 0.0019 (9) | $-0.0041$ (9) |
| C2  | 0.0222 (13) | 0.0144 (11) | 0.0149 (12) | $-0.0045$ (10) | $-0.0027$ (10) | $-0.0035$ (9) |
| C3  | 0.0196 (12) | 0.0133 (11) | 0.0198 (12) | $-0.0042$ (9) | $-0.0008$ (10) | $-0.0036$ (10) |
| C4  | 0.0175 (12) | 0.0149 (11) | 0.0170 (12) | $-0.0053$ (9) | 0.0017 (9) | $-0.0039$ (9) |
| C5  | 0.0186 (12) | 0.0140 (11) | 0.0151 (11) | $-0.0034$ (9) | 0.0004 (9) | $-0.0061$ (9) |
| C6  | 0.0150 (12) | 0.0119 (11) | 0.0194 (12) | $-0.0028$ (9) | $-0.0002$ (9) | $-0.0058$ (9) |
| C7  | 0.0214 (13) | 0.0223 (13) | 0.0177 (12) | $-0.0066$ (10) | 0.0038 (10) | $-0.0090$ (10) |
| C8  | 0.0338 (16) | 0.0219 (14) | 0.0242 (14) | $-0.0081$ (12) | 0.0097 (12) | $-0.0076$ (11) |
| C9  | 0.0213 (13) | 0.0315 (15) | 0.0254 (14) | $-0.0139$ (11) | $-0.0010$ (11) | $-0.0082$ (12) |
| C10 | 0.0247 (15) | 0.0402 (18) | 0.0346 (17) | $-0.0095$ (13) | $-0.0074$ (12) | $-0.0057$ (14) |
| C11 | 0.0220 (13) | 0.0199 (13) | 0.0175 (12) | $-0.0054$ (10) | 0.0007 (10) | $-0.0097$ (10) |
| C12 | 0.0347 (16) | 0.0196 (13) | 0.0317 (15) | $-0.0048$ (12) | $-0.0030$ (12) | $-0.0140$ (12) |
| C13 | 0.0304 (15) | 0.0230 (13) | 0.0194 (13) | $-0.0114$ (11) | $-0.0015$ (11) | $-0.0056$ (11) |
| C14 | 0.0360 (16) | 0.0473 (18) | 0.0230 (14) | $-0.0286$ (14) | 0.0079 (12) | $-0.0184$ (13) |
| C15 | 0.0389 (17) | 0.0447 (18) | 0.0386 (17) | $-0.0292$ (15) | 0.0183 (14) | $-0.0265$ (15) |
| C16 | 0.046 (2)  | 0.077 (3)  | 0.056 (2)  | $-0.045$ (2)  | 0.0341 (18) | $-0.053$ (2)  |
| C17 | 0.060 (2)  | 0.110 (4)  | 0.044 (2)  | $-0.065$ (3)  | 0.0349 (18) | $-0.060$ (2)  |
| C18 | 0.051 (2)  | 0.110 (4)  | 0.0274 (17) | $-0.058$ (2)  | 0.0143 (15) | $-0.037$ (2)  |
| C19 | 0.066 (3)  | 0.088 (3)  | 0.089 (3)  | $-0.052$ (3)  | 0.052 (2)  | $-0.078$ (3)  |
| C20 | 0.083 (3)  | 0.142 (5)  | 0.030 (2)  | $-0.066$ (3)  | 0.001 (2)  | $-0.033$ (3)  |
| C21 | 0.0188 (12) | 0.0170 (12) | 0.0203 (13) | $-0.0051$ (10) | 0.0046 (10) | $-0.0059$ (10) |
| C22 | 0.0153 (12) | 0.0199 (12) | 0.0182 (12) | $-0.0060$ (10) | $-0.0002$ (9) | $-0.0098$ (10) |
| C23 | 0.0166 (12) | 0.0162 (12) | 0.0192 (12) | $-0.0034$ (9) | 0.0013 (9) | $-0.0061$ (10) |
| C24 | 0.0185 (12) | 0.0226 (13) | 0.0210 (13) | $-0.0063$ (10) | 0.0041 (10) | $-0.0110$ (11) |
| C25 | 0.0180 (12) | 0.0250 (14) | 0.0232 (13) | $-0.0073$ (11) | 0.0057 (10) | $-0.0092$ (11) |
| C26 | 0.0188 (13) | 0.0207 (13) | 0.0228 (13) | $-0.0074$ (10) | 0.0020 (10) | $-0.0069$ (11) |
| C27 | 0.0268 (14) | 0.0202 (13) | 0.0314 (15) | $-0.0023$ (11) | 0.0117 (12) | $-0.0106$ (12) |
### Geometric parameters (Å, °)

| Bond            | Length (Å) | Bond Angle (°) |
|-----------------|------------|----------------|
| O1—C35          | 1.423 (4)  | C16—C17        | 1.386 (6) |
| O1—H1A          | 0.8400     | C16—C19        | 1.503 (6) |
| N1—C14          | 1.364 (4)  | C17—C18        | 1.366 (6) |
| N1—C13          | 1.451 (4)  | 1.451 (4)      | 0.9500    |
| N1—H1           | 0.94 (3)   | C18—C20        | 1.501 (6) |
| N2—C14          | 1.337 (4)  | C19—H19A       | 0.9800    |
| N2—C18          | 1.363 (4)  | C19—H19B       | 0.9800    |
| N3—C22          | 1.365 (3)  | C19—H19C       | 0.9800    |
| N3—C21          | 1.448 (3)  | C20—H20A       | 0.9800    |
| N3—H3           | 0.85 (3)   | C20—H20B       | 0.9800    |
| N4—C22          | 1.345 (3)  | C20—H20C       | 0.9800    |
| N4—C26          | 1.346 (3)  | C21—H21A       | 0.9900    |
| N5—C29          | 1.485 (3)  | C21—H21B       | 0.9900    |
| N5—C30          | 1.497 (3)  | C22—C23        | 1.402 (3) |
| N5—H5           | 0.90 (3)   | C23—C24        | 1.381 (3) |
| C1—C2           | 1.400 (4)  | C23—H23        | 0.9500    |
| C1—C6           | 1.407 (3)  | C24—C25        | 1.391 (4) |
| C1—C7           | 1.517 (3)  | C24—C27        | 1.503 (3) |
| C2—C3           | 1.408 (3)  | C25—C26        | 1.388 (4) |
| C2—C13          | 1.522 (3)  | C25—H25        | 0.9500    |
| C3—C4           | 1.404 (4)  | C26—C28        | 1.495 (4) |
| C3—C9           | 1.517 (4)  | C27—H27A       | 0.9800    |
| C4—C5           | 1.398 (3)  | C27—H27B       | 0.9800    |
| C4—C21          | 1.520 (3)  | C27—H27C       | 0.9800    |
| C5—C6           | 1.401 (3)  | C28—H28A       | 0.9800    |
| C5—C11          | 1.515 (3)  | C28—H28B       | 0.9800    |
| C6—C29          | 1.518 (3)  | C28—H28C       | 0.9800    |
| C7—C8           | 1.526 (4)  | C29—H29A       | 0.9900    |
| C7—H7A          | 0.9900     | C29—H29B       | 0.9900    |
| C7—H7B          | 0.9900     | C30—C31        | 1.525 (4) |
| C8—H8A          | 0.9800     | C30—C35        | 1.527 (4) |
| C8—H8B          | 0.9800     | C30—C34        | 1.549 (4) |
| C8—H8C          | 0.9800     | C31—C32        | 1.519 (4) |
| C9—C10          | 1.531 (4)  | C31—H31A       | 0.9900    |
| C9—H9A          | 0.9900     | C31—H31B       | 0.9900    |

**C28 0.0314 (15) 0.0211 (14) 0.0270 (14) −0.0098 (12) 0.0087 (12) −0.0064 (11)**

**C29 0.0200 (12) 0.0168 (12) 0.0214 (13) −0.0070 (10) 0.0031 (10) −0.0085 (10)**

**C30 0.0147 (12) 0.0198 (13) 0.0309 (14) −0.0070 (10) 0.0028 (10) −0.0111 (11)**

**C31 0.0189 (13) 0.0237 (14) 0.0447 (17) −0.0077 (11) −0.0020 (12) −0.0152 (13)**

**C32 0.0240 (14) 0.0360 (16) 0.0426 (18) −0.0095 (12) −0.0047 (13) −0.0219 (14)**

**C33 0.0168 (13) 0.0198 (13) 0.0309 (14) −0.0070 (10) 0.0028 (10) −0.0111 (11)**

**C34 0.0147 (12) 0.0214 (13) 0.0309 (14) −0.0070 (10) 0.0028 (10) −0.0111 (11)**

**C35 0.0189 (13) 0.0237 (14) 0.0447 (17) −0.0077 (11) −0.0020 (12) −0.0152 (13)**

**O1A 0.0292 (11) 0.0375 (12) 0.0312 (11) −0.0152 (9) −0.0019 (9) 0.0033 (9)**

**C1A 0.0297 (15) 0.0257 (14) 0.0330 (16) −0.0124 (12) 0.0041 (12) −0.0089 (12)**

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| Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|
| C9—H9B               | 0.9900       | C32—C33              | 1.536 (4)    |
| C10—H10A             | 0.9800       | C32—H32A             | 0.9900       |
| C10—H10B             | 0.9800       | C32—H32B             | 0.9900       |
| C10—H10C             | 0.9800       | C33—C34              | 1.545 (4)    |
| C11—C12              | 1.531 (4)    | C33—H33A             | 0.9900       |
| C11—H11A             | 0.9900       | C33—H33B             | 0.9900       |
| C11—H11B             | 0.9900       | C34—H34A             | 0.9900       |
| C12—H12A             | 0.9800       | C34—H34B             | 0.9900       |
| C12—H12B             | 0.9800       | C35—H35A             | 0.9900       |
| C12—H12C             | 0.9800       | C35—H35B             | 0.9900       |
| C13—H13A             | 0.9900       | O1A—C1A              | 1.399 (4)    |
| C13—H13B             | 0.9900       | O1A—H1AA             | 0.8400       |
| C14—C15              | 1.406 (5)    | C1A—H1A1             | 0.9800       |
| C15—C16              | 1.371 (4)    | C1A—H1A2             | 0.9800       |
| C15—H15              | 0.9500       | C1A—H1A3             | 0.9800       |

C35—O1—H1A 109.5  H19A—C19—H19C 109.5
C14—N1—C13 122.7 (3) H19B—C19—H19C 109.5
C14—N1—H1  119 (2)  C18—C20—H20A 109.5
C13—N1—H1  118 (2)  C18—C20—H20B 109.5
C14—N2—C18 117.3 (3) H20A—C20—H20B 109.5
C22—N3—C21 123.8 (2) C18—C20—H20C 109.5
C22—N3—H3  114 (2)  H20A—C20—H20C 109.5
C21—N3—H3  121 (2)  H20B—C20—H20C 109.5
C22—N4—C26 118.8 (2) N3—C21—C4 110.2 (2)
C29—N5—C30 115.6 (2) N3—C21—H21A 109.6
C29—N5—H5  106 (2)  C4—C21—H21A 109.6
C30—N5—H5  106 (2)  C4—C21—H21B 109.6
C2—C1—C6  119.8 (2)  C4—C21—H21B 109.6
C2—C1—C7  119.4 (2)  C4—C21—C27 120.6 (2)
C6—C1—C7  120.7 (2)  H21A—C21—H21B 108.1
C1—C2—C3  120.5 (2)  N4—C22—N3 115.6 (2)
C1—C2—C13 119.7 (2)  N4—C22—C23 122.0 (2)
C3—C2—C13 119.8 (2)  N3—C22—C23 122.3 (2)
C4—C3—C2  118.8 (2)  C24—C23—C22 119.2 (2)
C4—C3—C9  120.7 (2)  C24—C23—H23 120.4
C2—C3—C9  120.5 (2)  C22—C23—H23 120.4
C5—C4—C3  121.1 (2)  C23—C24—C25 118.4 (2)
C5—C4—C21 120.5 (2)  C23—C24—C27 120.6 (2)
C3—C4—C21 118.4 (2)  C25—C24—C27 121.0 (2)
C4—C5—C6  119.6 (2)  C26—C25—C24 119.8 (2)
C4—C5—C11 120.3 (2)  C26—C25—H25 120.1
C6—C5—C11 120.1 (2)  C24—C25—H25 120.1
C5—C6—C29 120.7 (2)  N4—C26—C25 121.9 (2)
C1—C6—C29 119.2 (2)  N4—C26—C28 116.3 (2)
C1—C7—C8  112.1 (2)  C25—C26—C28 121.8 (2)
C1—C7—H7A 109.2  H27A—C27—H27B 109.5
H27B—C27—H27B 109.5
| Bond                  | Angle   | Bond                  | Angle   |
|-----------------------|---------|-----------------------|---------|
| C8—C7—H7A            | 109.2   | C24—C27—H27C         | 109.5   |
| C1—C7—H7B            | 109.2   | H27A—C27—H27C        | 109.5   |
| C8—C7—H7B            | 109.2   | H27B—C27—H27C        | 109.5   |
| H7A—C7—H7B           | 107.9   | C26—C28—H28A         | 109.5   |
| C7—C8—H8A            | 109.5   | C26—C28—H28B         | 109.5   |
| C7—C8—H8B            | 109.5   | H28A—C28—H28B        | 109.5   |
| H8A—C8—H8B           | 109.5   | C26—C28—H28C         | 109.5   |
| C7—C8—H8C            | 109.5   | H28A—C28—H28C        | 109.5   |
| H8A—C8—H8C           | 109.5   | H28B—C28—H28C        | 109.5   |
| H8B—C8—H8C           | 109.5   | N5—C29—C6            | 110.1 (2) |
| C3—C9—C10            | 113.1 (2)| N5—C29—H29A          | 109.6   |
| C3—C9—H9A            | 109.0   | C6—C29—H29A          | 109.6   |
| C10—C9—H9A           | 109.0   | N5—C29—H29B          | 109.6   |
| C3—C9—H9B            | 109.0   | C6—C29—H29B          | 109.6   |
| C10—C9—H9B           | 109.0   | H29A—C29—H29B        | 108.1   |
| C9—C10—H10A          | 109.5   | N5—C30—C31           | 110.6 (2) |
| C9—C10—H10B          | 109.5   | N5—C30—C35           | 110.3 (2) |
| H10A—C10—H10B        | 109.5   | C31—C30—C35          | 113.0 (2) |
| C9—C10—H10C          | 109.5   | N5—C30—C34           | 107.1 (2) |
| C10—C10—H10C         | 109.5   | C31—C30—C34          | 103.3 (2) |
| H10B—C10—H10C        | 109.5   | C35—C30—C34          | 112.1 (2) |
| C5—C11—C12           | 112.6 (2)| C32—C31—C30          | 103.5 (2) |
| C5—C11—H11A          | 109.1   | C32—C31—H31A         | 111.1   |
| C12—C11—H11A         | 109.1   | C32—C31—H31B         | 111.1   |
| C5—C11—H11B          | 109.1   | C30—C31—H31B         | 111.1   |
| C12—C11—H11B         | 109.1   | H31A—C31—H31B        | 109.0   |
| H11A—C11—H11B        | 107.8   | C31—C32—C33          | 103.2 (2) |
| C11—C12—H12A         | 109.5   | C31—C32—H32A         | 111.1   |
| C11—C12—H12B         | 109.5   | C33—C32—H32A         | 111.1   |
| H12A—C12—H12B        | 109.5   | C31—C32—H32B         | 111.1   |
| C11—C12—H12C         | 109.5   | C33—C32—H32B         | 111.1   |
| H12A—C12—H12C        | 109.5   | H32A—C32—H32B        | 109.1   |
| H12B—C12—H12C        | 109.5   | C32—C33—C34          | 105.5 (2) |
| N1—C13—C2            | 109.9 (2)| C32—C33—H33A         | 110.6   |
| N1—C13—H13A          | 109.7   | C34—C33—H33A         | 110.6   |
| C2—C13—H13A          | 109.7   | C32—C33—H33B         | 110.6   |
| N1—C13—H13B          | 109.7   | C34—C33—H33B         | 110.6   |
| C2—C13—H13B          | 109.7   | H33A—C33—H33B        | 108.8   |
| H13A—C13—H13B        | 108.2   | C33—C34—C30          | 106.2 (2) |
| N2—C14—N1            | 118.4 (3)| C33—C34—H34A         | 110.5   |
| N2—C14—C15           | 122.8 (3)| C30—C34—H34A         | 110.5   |
| N1—C14—C15           | 118.8 (3)| C33—C34—H34B         | 110.5   |
| C16—C15—C14          | 118.6 (4)| C30—C34—H34B         | 110.5   |
| C16—C15—H15          | 120.7   | H34A—C34—H34B        | 108.7   |
| C14—C15—H15          | 120.7   | O1—C35—C30           | 110.5 (2) |
| C15—C16—C17          | 119.0 (4)| O1—C35—H35A          | 109.5   |
| C15—C16—C19          | 119.8 (4)| C30—C35—H35A         | 109.5   |
| Bond                  | Distance  | Bond                  | Distance  |
|----------------------|-----------|----------------------|-----------|
| C17—C16—C19         | 121.2 (3) | O1—C35—H35B         | 109.5     |
| C18—C17—C16         | 119.6 (3) | C30—C35—H35B        | 109.5     |
| C18—C17—H17         | 120.2     | H35A—C35—H35B       | 108.1     |
| C16—C17—H17         | 120.2     | C1A—O1A—H1AA        | 109.5     |
| N2—C18—C17          | 122.7 (4) | O1A—C1A—H1A1        | 109.5     |
| N2—C18—C20          | 115.5 (4) | O1A—C1A—H1A2        | 109.5     |
| C17—C18—C20         | 121.8 (4) | H1A1—C1A—H1A2       | 109.5     |
| C16—C19—H19A        | 109.5     | O1A—C1A—H1A3        | 109.5     |
| C16—C19—H19B        | 109.5     | H1A1—C1A—H1A3       | 109.5     |
| H19A—C19—H19B       | 109.5     | H1A2—C1A—H1A3       | 109.5     |
| C16—C19—H19C        | 109.5     |                       |           |
| C6—C1—C2—C3         | −1.2 (4)  | C15—C16—C17—C18     | 0.4 (5)   |
| C7—C1—C2—C3         | −178.5 (2)| C19—C16—C17—C18     | −177.6 (3)|
| C6—C1—C2—C13        | 179.5 (2) | C14—N2—C18—C17      | 0.6 (5)   |
| C7—C1—C2—C13        | 2.3 (3)   | C14—N2—C18—C20      | 180.0 (3) |
| C1—C2—C3—C4         | 2.8 (4)   | C16—C17—C18—N2      | −0.6 (5)  |
| C13—C2—C3—C4        | −177.9 (2)| C16—C17—C18—C20     | −180.0 (3)|
| C1—C2—C3—C9         | −177.7 (2)| C22—N3—C21—C4       | −177.5 (2)|
| C13—C2—C3—C9        | 1.6 (4)   | C5—C4—C21—N3        | −93.1 (3) |
| C2—C3—C4—C5         | −2.7 (4)  | C3—C4—C21—N3        | 85.7 (3)  |
| C9—C3—C4—C5         | 177.8 (2) | C26—N4—C22—N3       | 177.4 (2) |
| C2—C3—C4—C21        | 178.5 (2) | C26—N4—C22—C23      | −1.5 (4)  |
| C9—C3—C4—C21        | −1.0 (3)  | C21—N3—C22—N4       | 177.4 (2) |
| C3—C4—C5—C6         | 1.0 (4)   | C21—N3—C22—C23      | −3.7 (4)  |
| C21—C4—C5—C6        | 179.7 (2) | N4—C22—C23—C24      | 1.8 (4)   |
| C3—C4—C5—C11        | −179.6 (2)| N3—C22—C23—C24      | −177.0 (2)|
| C21—C4—C5—C11       | −0.9 (3)  | C22—C23—C24—C25     | −0.5 (4)  |
| C4—C5—C6—C1         | 0.7 (4)   | C22—C23—C24—C27     | 179.7 (3) |
| C11—C5—C6—C1        | −178.7 (2)| C23—C24—C25—C26     | −0.9 (4)  |
| C4—C5—C6—C29        | −177.8 (2)| C27—C24—C25—C26     | 178.8 (3) |
| C11—C5—C6—C29       | 2.8 (3)   | C22—N4—C26—C25      | 0.0 (4)   |
| C2—C1—C6—C5         | −0.6 (3)  | C22—N4—C26—C28      | 178.8 (2) |
| C7—C1—C6—C5         | 176.6 (2) | C24—C25—C26—N4      | 1.3 (4)   |
| C2—C1—C6—C29        | 178.0 (2) | C24—C25—C26—C28     | −177.5 (3)|
| C7—C1—C6—C29        | −4.8 (3)  | C30—N5—C29—C6       | 162.4 (2) |
| C2—C1—C7—C8         | 89.8 (3)  | C5—C6—C29—N5        | 90.9 (3)  |
| C6—C1—C7—C8         | −87.4 (3) | C1—C6—C29—N5        | −87.6 (3) |
| C4—C3—C9—C10        | 90.5 (3)  | C29—N5—C30—C31      | 54.1 (3)  |
| C2—C3—C9—C10        | −89.0 (3) | C29—N5—C30—C35      | −71.7 (3) |
| C4—C5—C11—C12       | −91.6 (3) | C29—N5—C30—C34      | 166.1 (2) |
| C6—C5—C11—C12       | 87.8 (3)  | N5—C30—C31—C32      | 75.2 (3)  |
| C14—N1—C13—C2       | −173.9 (3)| C35—C30—C31—C32     | −160.5 (2)|
| C1—C2—C13—N1        | 83.0 (3)  | C34—C30—C31—C32     | −39.2 (3) |
| C3—C2—C13—N1        | −96.2 (3) | C30—C31—C32—C33     | 42.6 (3)  |
| C18—N2—C14—N1       | 179.7 (3) | C31—C32—C33—C34     | −29.1 (3) |
| C18—N2—C14—C15      | −0.4 (4)  | C32—C33—C34—C30     | 5.1 (3)   |
| C13—N1—C14—N2       | −6.6 (4)  | N5—C30—C34—C33      | −96.1 (2) |
C13—N1—C14—C15 173.5 (3)  C31—C30—C34—C33 20.7 (3)
N2—C14—C15—C16 0.3 (4)  C35—C30—C34—C33 142.8 (2)
N1—C14—C15—C16 −179.8 (3)  N5—C30—C35—O1 −52.9 (3)
C14—C15—C16—C17 −0.3 (4)  C31—C30—C35—O1 −177.4 (2)
C14—C15—C16—C19 177.8 (3)  C34—C30—C35—O1 66.3 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A     | D—H   | H···A   | D···A   | D—H···A |
|-------------|-------|--------|--------|---------|
| N1—H1···O1A | 0.94  (3) | 2.01 (3) | 2.930 (3) | 162 (2) |
| C15—H15···O1A | 0.95 | 2.56 | 3.318 (3) | 137 |
| N5—H5···O1 | 0.90 (3) | 2.36 (3) | 2.823 (3) | 112 (2) |
| O1—H1A···N4i | 0.84 | 1.90 | 2.741 (3) | 174 |
| O1A—H1A···N5i | 0.84 | 1.97 | 2.798 (3) | 170 |
| C27—H27A···Cg1ii | 0.98 | 2.67 | 3.541 (3) | 148 |
| C32—H32B···Cg3iii | 0.98 | 2.69 | 3.614 (3) | 156 |

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