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# 2-Dimethylamino-1-(2-ethoxy-2-oxoethyl)-3-methyl-4,5-dihydroimidazolium tetraphenylborate

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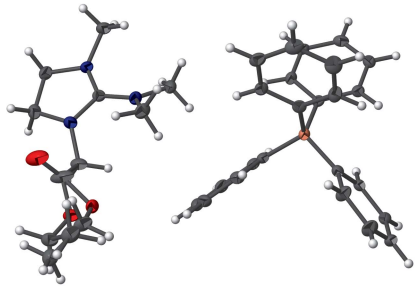
**Keywords:** crystal structure; dihydroimidazolium; tetraphenylborate; salt; C—H$\cdots\pi$ interactions.

**CCDC reference**: 1448192

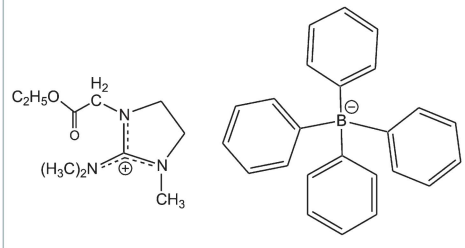
**Structural data**: full structural data are available from iucrdata.iucr.org

In the crystal structure of the title salt, $C_{10}H_{20}N_3O_2^+\cdot C_{24}H_{20}B^-$, the C—N bond lengths in the cation are 1.327 (3), 1.339 (3) and 1.342 (3) Å, indicating partial double-bond character. The central C atom is bonded to the three N atoms, indicating only a slight deviation from a trigonal–planar geometry. The positive charge is delocalized in the $CN_3$ plane. The ethoxy group is disordered over two orientations, with an occupancy ratio of 0.60 (1):0.40 (1). C—H$\cdots\pi$ interactions are present between the guanidinium H atoms and the phenyl C atoms of the tetraphenylborate ions. The phenyl rings form aromatic pockets, in which the cations are embedded. This leads to the formation of a two-dimensional supramolecular pattern along the *ac* plane.

**3D view**



**Chemical scheme**




## Structure description

By the reaction of *N*,*N*,*N*′,*N*′-tetramethylchloroformamidinium chloride (Tiritiris & Kantlehner, 2008) with *N*-methyl-1,2-ethanediamine, a mixture consisting of two guanidinium dichlorides and one bisguanidinium dichloride was obtained. After treating the salt mixture with an aqueous sodium hydroxide solution, the cyclic guanidine 1-methyl-2-dimethylamino-1*H*-4,5-dihydroimidazole emerged as one of the products (Tiritiris & Kantlehner, 2013). By alkylation of the free nitrogen atom of the resulting guanidine base, various cyclic guanidinium salts can be obtained. The title salt is the first dihydroimidazole derivative in our series; it has been structurally characterized after anion exchange with sodium tetraphenylborate.

The bond lengths in the cation of the title salt are in very good agreement with those in a similar compound, 2-dimethylamino-1-(2-ethoxy-2-oxoethyl)-3-methyl-3,4,5,6-tetrahydropyrimidin-1-ium tetraphenylborate (Tiritiris & Kantlehner, 2012). Prominent bond parameters in the dihydroimidazolium ion are: C1—N1 = 1.327 (3) Å, C1—N2 =

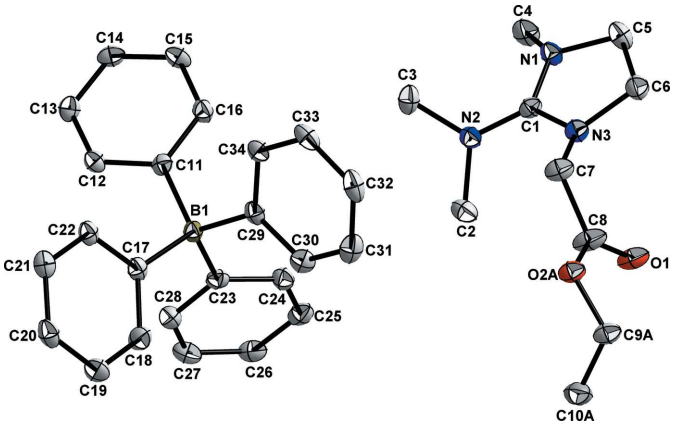



**Figure 1**
The structure of the title compound, with displacement ellipsoids at the 50% probability level. All H atoms have been omitted for clarity. Only the major orientation of the disordered ethoxy group is shown.

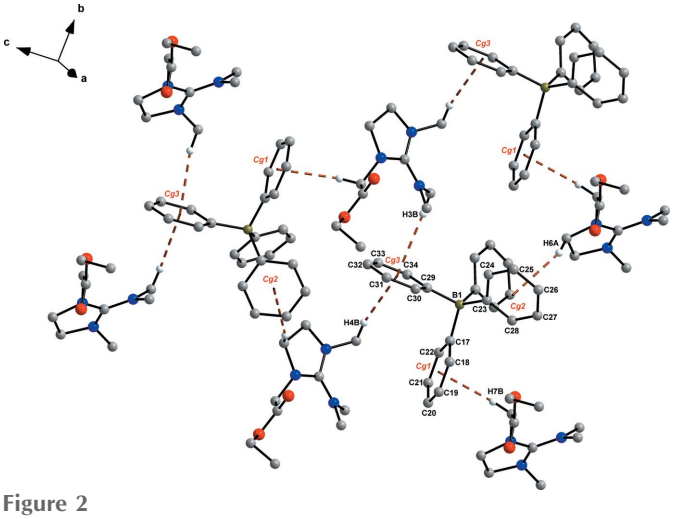



**Figure 2**
C—H···$\pi$ interactions (brown dashed lines) between the H atoms of the guanidinium ion and the phenyl C atoms (centroids) of the tetraphenylborate ion.

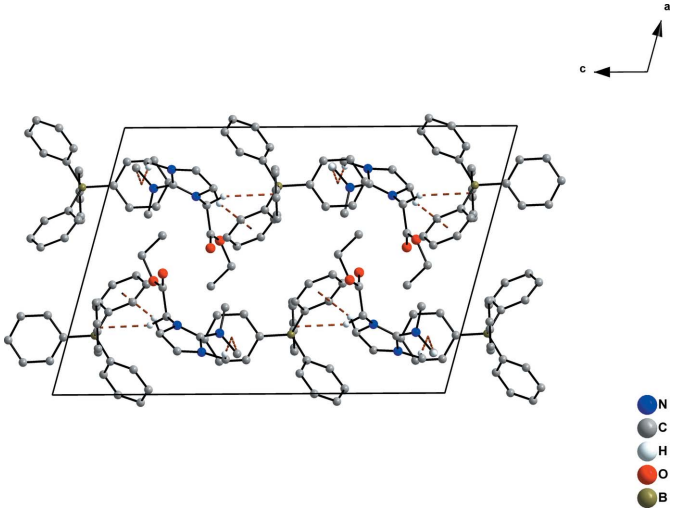



**Figure 3**
C—H···$\pi$ interactions (brown dashed lines) showing the two-dimensional supramolecular architecture in the *ac* plane.

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

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the C17–C22, C23–C28 and C29–C34 rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7*B*···*Cg*1$^{i}$ | 0.99 | 2.77 | 3.694 (3) | 155 |
| C6—H6*A*···*Cg*2$^{ii}$ | 0.99 | 2.96 | 3.870 (3) | 153 |
| C4—H4*B*···*Cg*3$^{iii}$ | 0.98 | 2.79 | 3.758 (3) | 169 |
| C3—H3*B*···*Cg*3 | 0.98 | 2.94 | 3.868 (3) | 159 |

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $x, y+1, z$.

1.339 (3) Å and C1—N3 = 1.342 (3) Å, indicating partial double-bond character (Fig. 1). The N—C1—N angles are: 124.52 (18)° (N1—C1—N2), 123.02 (19)° (N2—C1—N3) and 112.45 (18)° (N1—C1—N3), indicating only a slight deviation from an ideal trigonal–planar surrounding of the carbon centre by the three nitrogen atoms. The positive charge is completely delocalized in the $CN_3$ plane. The ethoxy group is disordered over two orientations, with an occupancy ratio of 0.60 (1):0.40 (1).

The bond lengths and angles in the tetraphenylborate ions are in good agreement with the data from the crystal structure analysis of the alkali metal tetraphenylborates (Behrens *et al.*, 2012). C—H···$\pi$ interactions between the guanidinium hydrogen atoms of the –N(CH$_3$)$_2$ and –CH$_2$ groups and the phenyl carbon atoms of the tetraphenylborate ion are also present (Fig. 2), ranging from 2.77 to 2.96 Å (Table 1). The

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{10}H_{20}N_3O_2^+\cdot C_{24}H_{20}B^-$ |
| $M_r$ | 533.50 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 100 |
| $a, b, c$ (Å) | 14.3033 (6), 10.3598 (4), 20.2825 (9) |
| $\beta$ (°) | 105.468 (2) |
| $V$ (Å$^3$) | 2896.6 (2) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.08 |
| Crystal size (mm) | 0.30 × 0.23 × 0.12 |
| Data collection | |
| Diffractometer | Bruker–Nonius KappaCCD |
| Absorption correction | – |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 12960, 7012, 4502 |
| $R_{int}$ | 0.071 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.665 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.065, 0.148, 1.04 |
| No. of reflections | 7012 |
| No. of parameters | 397 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.39, −0.26 |

Computer programs: *COLLECT* (Hooft, 2004), *DENZO-SMN* (Otwinowski & Minor, 1997), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 2005).

phenyl rings form aromatic pockets, in which the guanidinium ions are embedded. This leads to the formation of a two-dimensional supramolecular pattern in the *ac* plane (Fig. 3).

## Synthesis and crystallization

The title compound was obtained by reaction of 1-methyl-2-dimethylamino-1*H*-4,5-dihydroimidazole (Tiritiris & Kantlehner, 2013) with bromoacetic acid ethyl ester in acetonitrile at room temperature. After evaporation of the solvent the crude 2-dimethylamino-1-(2-ethoxy-2-oxoethyl)-3-methyl-4,5-dihydroimidazolium bromide (I) was washed with diethyl ether and dried *in vacuo*. 1.0 g (3.4 mmol) of (I) was dissolved in 20 ml acetonitrile and 1.16 g (3.4 mmol) of sodium tetraphenylborate in 20 ml acetonitrile was added. After stirring for one hour at room temperature, the precipitated sodium bromide was filtered off. The title compound crystallized from a saturated acetonitrile solution after several days at 273 K, forming colorless single crystals. Yield: 1.44 g (79%).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The atoms O2, C9 and C10 of the ethoxy group are disordered over two sets of sites (O2*A*, C9*A* and C10*A*; O2*B*, C9*B* and C10*B*) with refined occupancies of 0.60 (1):0.40 (1), 0.58 (1):0.42 (1) and 0.59 (1):0.41 (1).

## Acknowledgements

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# full crystallographic data

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## 2-Dimethylamino-1-(2-ethoxy-2-oxoethyl)-3-methyl-4,5-dihydroimidazolium tetraphenylborate

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### 2-Dimethylamino-1-(2-ethoxy-2-oxoethyl)-3-methyl-4,5-dihydroimidazolium tetraphenylborate

*Crystal data*

$C_{10}H_{20}N_3O_2^+ \cdot C_{24}H_{20}B^-$
$M_r$ = 533.50
Monoclinic, $P2_1/c$
$a$ = 14.3033 (6) Å
$b$ = 10.3598 (4) Å
$c$ = 20.2825 (9) Å
$\beta$ = 105.468 (2)°
$V$ = 2896.6 (2) Å$^3$
$Z$ = 4
$F(000)$ = 1144
$D_x$ = 1.223 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 7075 reflections
$\theta$ = 0.4–28.3°
$\mu$ = 0.08 mm$^{-1}$
$T$ = 100 K
Block, colorless
0.30 × 0.23 × 0.12 mm

*Data collection*

Bruker–Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ scans, and $\omega$ scans
12960 measured reflections
7012 independent reflections
4502 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.071
$\theta_{max}$ = 28.2°, $\theta_{min}$ = 3.0°
$h$ = −18→18
$k$ = −13→13
$l$ = −26→26

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.148
$S$ = 1.04
7012 reflections
397 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 2.4901P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.15751 (13) | 0.85761 (17) | 0.14856 (9) | 0.0214 (4) | |
| N2 | 0.22801 (13) | 0.67062 (17) | 0.11579 (9) | 0.0218 (4) | |
| N3 | 0.25859 (13) | 0.74356 (18) | 0.22959 (9) | 0.0224 (4) | |
| C1 | 0.21433 (14) | 0.7544 (2) | 0.16263 (10) | 0.0195 (4) | |
| C2 | 0.32486 (16) | 0.6191 (2) | 0.11954 (12) | 0.0276 (5) | |
| H2A | 0.3723 | 0.6579 | 0.1585 | 0.041* | |
| H2B | 0.3424 | 0.6398 | 0.0772 | 0.041* | |
| H2C | 0.3246 | 0.5252 | 0.1254 | 0.041* | |
| C3 | 0.15055 (17) | 0.6305 (2) | 0.05694 (11) | 0.0261 (5) | |
| H3A | 0.0883 | 0.6628 | 0.0618 | 0.039* | |
| H3B | 0.1486 | 0.5360 | 0.0543 | 0.039* | |
| H3C | 0.1626 | 0.6656 | 0.0151 | 0.039* | |
| C4 | 0.12980 (18) | 0.9223 (2) | 0.08231 (11) | 0.0284 (5) | |
| H4A | 0.1623 | 0.8807 | 0.0511 | 0.043* | |
| H4B | 0.1491 | 1.0133 | 0.0881 | 0.043* | |
| H4C | 0.0594 | 0.9165 | 0.0632 | 0.043* | |
| C5 | 0.16176 (18) | 0.9309 (2) | 0.21168 (11) | 0.0271 (5) | |
| H5A | 0.0965 | 0.9391 | 0.2194 | 0.032* | |
| H5B | 0.1889 | 1.0182 | 0.2096 | 0.032* | |
| C6 | 0.22867 (16) | 0.8499 (2) | 0.26790 (11) | 0.0259 (5) | |
| H6A | 0.2854 | 0.9008 | 0.2934 | 0.031* | |
| H6B | 0.1937 | 0.8167 | 0.3004 | 0.031* | |
| C7 | 0.29159 (16) | 0.6205 (2) | 0.26243 (11) | 0.0250 (5) | |
| H7A | 0.2727 | 0.5503 | 0.2284 | 0.030* | |
| H7B | 0.2593 | 0.6047 | 0.2992 | 0.030* | |
| C8 | 0.40090 (18) | 0.6179 (2) | 0.29273 (14) | 0.0376 (6) | |
| O1 | 0.45299 (12) | 0.70977 (17) | 0.30205 (9) | 0.0374 (4) | |
| O2A | 0.42497 (19) | 0.5050 (3) | 0.3209 (3) | 0.0239 (13) | 0.60 (1) |
| O2B | 0.4324 (3) | 0.4853 (4) | 0.2830 (4) | 0.0219 (18) | 0.40 (1) |
| C9A | 0.5210 (4) | 0.4881 (5) | 0.3677 (2) | 0.0252 (14) | 0.58 (1) |
| H9A | 0.5192 | 0.4163 | 0.3996 | 0.030* | 0.578 (13) |
| H9B | 0.5397 | 0.5676 | 0.3951 | 0.030* | 0.578 (13) |
| C9B | 0.5365 (7) | 0.4603 (6) | 0.3073 (4) | 0.029 (2) | 0.42 (1) |
| H9C | 0.5727 | 0.5331 | 0.2943 | 0.035* | 0.422 (13) |
| H9D | 0.5526 | 0.3810 | 0.2854 | 0.035* | 0.422 (13) |
| C10A | 0.5954 (5) | 0.4591 (4) | 0.3294 (3) | 0.0309 (15) | 0.59 (1) |
| H10A | 0.5733 | 0.3863 | 0.2984 | 0.046* | 0.589 (12) |
| H10B | 0.6573 | 0.4372 | 0.3620 | 0.046* | 0.589 (12) |
| H10C | 0.6041 | 0.5352 | 0.3029 | 0.046* | 0.589 (12) |
| C10B | 0.5659 (7) | 0.4445 (8) | 0.3820 (4) | 0.029 (2) | 0.41 (1) |

| | | | | | |
|---|---|---|---|---|---|
| H10D | 0.5509 | 0.5236 | 0.4037 | 0.043* | 0.411 (12) |
| H10E | 0.6357 | 0.4276 | 0.3973 | 0.043* | 0.411 (12) |
| H10F | 0.5305 | 0.3718 | 0.3948 | 0.043* | 0.411 (12) |
| B1 | 0.22231 (17) | 0.2365 (2) | −0.06717 (11) | 0.0184 (5) | |
| C11 | 0.12514 (14) | 0.2878 (2) | −0.12493 (10) | 0.0180 (4) | |
| C12 | 0.09364 (15) | 0.2303 (2) | −0.18964 (10) | 0.0202 (4) | |
| H12 | 0.1254 | 0.1542 | −0.1984 | 0.024* | |
| C13 | 0.01801 (15) | 0.2796 (2) | −0.24152 (10) | 0.0224 (5) | |
| H13 | −0.0010 | 0.2373 | −0.2846 | 0.027* | |
| C14 | −0.02973 (15) | 0.3908 (2) | −0.23034 (11) | 0.0233 (5) | |
| H14 | −0.0817 | 0.4248 | −0.2654 | 0.028* | |
| C15 | −0.00054 (16) | 0.4511 (2) | −0.16742 (11) | 0.0235 (5) | |
| H15 | −0.0328 | 0.5270 | −0.1590 | 0.028* | |
| C16 | 0.07592 (16) | 0.4010 (2) | −0.11641 (11) | 0.0219 (4) | |
| H16 | 0.0957 | 0.4454 | −0.0740 | 0.026* | |
| C17 | 0.23750 (15) | 0.0801 (2) | −0.07090 (10) | 0.0198 (4) | |
| C18 | 0.32987 (17) | 0.0230 (2) | −0.05127 (12) | 0.0276 (5) | |
| H18 | 0.3852 | 0.0777 | −0.0395 | 0.033* | |
| C19 | 0.34389 (18) | −0.1098 (2) | −0.04832 (12) | 0.0301 (5) | |
| H19 | 0.4078 | −0.1438 | −0.0346 | 0.036* | |
| C20 | 0.26550 (17) | −0.1927 (2) | −0.06516 (11) | 0.0254 (5) | |
| H20 | 0.2748 | −0.2835 | −0.0634 | 0.030* | |
| C21 | 0.17262 (16) | −0.1403 (2) | −0.08472 (10) | 0.0225 (5) | |
| H21 | 0.1178 | −0.1958 | −0.0964 | 0.027* | |
| C22 | 0.15955 (16) | −0.0067 (2) | −0.08722 (10) | 0.0204 (4) | |
| H22 | 0.0954 | 0.0267 | −0.1005 | 0.024* | |
| C23 | 0.31026 (14) | 0.3182 (2) | −0.08575 (10) | 0.0200 (4) | |
| C24 | 0.34530 (15) | 0.4359 (2) | −0.05523 (11) | 0.0216 (4) | |
| H24 | 0.3225 | 0.4660 | −0.0181 | 0.026* | |
| C25 | 0.41212 (16) | 0.5108 (2) | −0.07703 (11) | 0.0250 (5) | |
| H25 | 0.4346 | 0.5897 | −0.0544 | 0.030* | |
| C26 | 0.44602 (16) | 0.4705 (2) | −0.13184 (12) | 0.0269 (5) | |
| H26 | 0.4909 | 0.5218 | −0.1474 | 0.032* | |
| C27 | 0.41315 (16) | 0.3543 (2) | −0.16324 (12) | 0.0300 (5) | |
| H27 | 0.4361 | 0.3251 | −0.2005 | 0.036* | |
| C28 | 0.34649 (15) | 0.2798 (2) | −0.14064 (11) | 0.0243 (5) | |
| H28 | 0.3249 | 0.2005 | −0.1631 | 0.029* | |
| C29 | 0.21625 (15) | 0.25991 (19) | 0.01178 (10) | 0.0195 (4) | |
| C30 | 0.29935 (16) | 0.2660 (2) | 0.06704 (11) | 0.0238 (5) | |
| H30 | 0.3611 | 0.2650 | 0.0578 | 0.029* | |
| C31 | 0.29566 (17) | 0.2735 (2) | 0.13480 (11) | 0.0277 (5) | |
| H31 | 0.3541 | 0.2776 | 0.1706 | 0.033* | |
| C32 | 0.20657 (18) | 0.2749 (2) | 0.15029 (11) | 0.0281 (5) | |
| H32 | 0.2035 | 0.2805 | 0.1964 | 0.034* | |
| C33 | 0.12275 (17) | 0.2679 (2) | 0.09746 (11) | 0.0265 (5) | |
| H33 | 0.0614 | 0.2684 | 0.1072 | 0.032* | |
| C34 | 0.12770 (15) | 0.2601 (2) | 0.02969 (11) | 0.0212 (4) | |
| H34 | 0.0690 | 0.2547 | −0.0057 | 0.025* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0257 (9) | 0.0168 (9) | 0.0217 (9) | 0.0042 (7) | 0.0062 (7) | 0.0017 (7) |
| N2 | 0.0230 (9) | 0.0196 (9) | 0.0220 (9) | 0.0037 (7) | 0.0048 (7) | −0.0014 (7) |
| N3 | 0.0246 (9) | 0.0214 (10) | 0.0202 (8) | 0.0028 (7) | 0.0044 (7) | 0.0022 (7) |
| C1 | 0.0184 (10) | 0.0191 (11) | 0.0201 (10) | −0.0011 (8) | 0.0035 (8) | 0.0019 (8) |
| C2 | 0.0287 (12) | 0.0219 (12) | 0.0345 (12) | 0.0047 (9) | 0.0128 (10) | −0.0012 (10) |
| C3 | 0.0342 (12) | 0.0200 (11) | 0.0221 (11) | −0.0033 (9) | 0.0039 (9) | −0.0017 (9) |
| C4 | 0.0361 (13) | 0.0215 (12) | 0.0261 (11) | 0.0076 (10) | 0.0057 (10) | 0.0052 (9) |
| C5 | 0.0359 (13) | 0.0202 (11) | 0.0278 (11) | 0.0028 (10) | 0.0131 (10) | −0.0013 (9) |
| C6 | 0.0279 (12) | 0.0258 (12) | 0.0245 (11) | −0.0006 (10) | 0.0080 (9) | −0.0034 (9) |
| C7 | 0.0220 (11) | 0.0247 (12) | 0.0273 (11) | 0.0009 (9) | 0.0046 (9) | 0.0093 (9) |
| C8 | 0.0242 (12) | 0.0264 (13) | 0.0568 (16) | −0.0009 (10) | 0.0013 (11) | 0.0206 (12) |
| O1 | 0.0228 (8) | 0.0303 (10) | 0.0548 (11) | −0.0017 (7) | 0.0028 (8) | 0.0138 (8) |
| O2A | 0.0213 (14) | 0.0220 (15) | 0.027 (3) | 0.0022 (10) | 0.0039 (12) | 0.0065 (15) |
| O2B | 0.024 (2) | 0.019 (2) | 0.021 (4) | 0.0051 (16) | 0.0040 (18) | −0.0016 (19) |
| C9A | 0.021 (3) | 0.026 (3) | 0.027 (2) | 0.004 (2) | 0.0025 (18) | 0.0058 (18) |
| C9B | 0.023 (5) | 0.021 (3) | 0.041 (4) | 0.007 (3) | 0.007 (3) | 0.005 (3) |
| C10A | 0.025 (3) | 0.027 (2) | 0.041 (3) | 0.0048 (19) | 0.009 (2) | 0.0016 (19) |
| C10B | 0.027 (4) | 0.028 (4) | 0.031 (4) | 0.000 (3) | 0.007 (3) | −0.003 (3) |
| B1 | 0.0215 (11) | 0.0156 (11) | 0.0172 (11) | 0.0015 (9) | 0.0034 (9) | −0.0010 (9) |
| C11 | 0.0179 (10) | 0.0158 (10) | 0.0202 (10) | −0.0019 (8) | 0.0051 (8) | 0.0011 (8) |
| C12 | 0.0230 (10) | 0.0182 (10) | 0.0205 (10) | 0.0017 (8) | 0.0076 (8) | 0.0009 (8) |
| C13 | 0.0256 (11) | 0.0225 (11) | 0.0189 (10) | −0.0018 (9) | 0.0054 (8) | 0.0006 (9) |
| C14 | 0.0196 (11) | 0.0243 (11) | 0.0251 (11) | 0.0024 (9) | 0.0043 (8) | 0.0075 (9) |
| C15 | 0.0273 (11) | 0.0163 (10) | 0.0276 (11) | 0.0066 (9) | 0.0088 (9) | 0.0046 (9) |
| C16 | 0.0263 (11) | 0.0167 (10) | 0.0219 (10) | 0.0024 (9) | 0.0048 (8) | −0.0023 (8) |
| C17 | 0.0247 (11) | 0.0183 (11) | 0.0157 (9) | 0.0030 (8) | 0.0042 (8) | 0.0007 (8) |
| C18 | 0.0239 (11) | 0.0184 (11) | 0.0357 (13) | 0.0016 (9) | −0.0005 (9) | −0.0013 (10) |
| C19 | 0.0286 (12) | 0.0212 (12) | 0.0370 (13) | 0.0072 (10) | 0.0025 (10) | −0.0012 (10) |
| C20 | 0.0387 (13) | 0.0156 (11) | 0.0209 (10) | 0.0053 (9) | 0.0062 (9) | 0.0008 (8) |
| C21 | 0.0305 (11) | 0.0190 (11) | 0.0187 (10) | −0.0022 (9) | 0.0077 (9) | 0.0004 (8) |
| C22 | 0.0263 (11) | 0.0194 (11) | 0.0151 (9) | 0.0022 (9) | 0.0051 (8) | 0.0002 (8) |
| C23 | 0.0170 (10) | 0.0200 (11) | 0.0209 (10) | 0.0038 (8) | 0.0013 (8) | 0.0024 (8) |
| C24 | 0.0233 (11) | 0.0181 (10) | 0.0218 (10) | 0.0027 (9) | 0.0033 (8) | 0.0001 (8) |
| C25 | 0.0216 (11) | 0.0190 (11) | 0.0303 (12) | 0.0009 (9) | −0.0004 (9) | 0.0035 (9) |
| C26 | 0.0193 (11) | 0.0252 (12) | 0.0363 (13) | 0.0022 (9) | 0.0077 (9) | 0.0048 (10) |
| C27 | 0.0254 (12) | 0.0335 (13) | 0.0344 (12) | 0.0048 (10) | 0.0139 (10) | −0.0011 (11) |
| C28 | 0.0224 (11) | 0.0229 (11) | 0.0276 (11) | 0.0009 (9) | 0.0067 (9) | −0.0042 (9) |
| C29 | 0.0254 (10) | 0.0134 (10) | 0.0196 (10) | 0.0021 (8) | 0.0059 (8) | 0.0013 (8) |
| C30 | 0.0262 (11) | 0.0202 (11) | 0.0243 (11) | 0.0016 (9) | 0.0055 (9) | 0.0034 (9) |
| C31 | 0.0351 (13) | 0.0216 (11) | 0.0216 (10) | −0.0007 (10) | −0.0009 (9) | 0.0036 (9) |
| C32 | 0.0471 (14) | 0.0176 (11) | 0.0215 (11) | −0.0006 (10) | 0.0124 (10) | −0.0012 (9) |
| C33 | 0.0350 (12) | 0.0179 (11) | 0.0308 (11) | 0.0037 (10) | 0.0161 (10) | −0.0007 (9) |
| C34 | 0.0236 (10) | 0.0147 (10) | 0.0247 (10) | 0.0025 (8) | 0.0057 (8) | 0.0005 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.327 (3) | B1—C11 | 1.649 (3) |
| N1—C4 | 1.459 (3) | C11—C12 | 1.402 (3) |
| N1—C5 | 1.475 (3) | C11—C16 | 1.402 (3) |
| N2—C1 | 1.339 (3) | C12—C13 | 1.390 (3) |
| N2—C3 | 1.456 (3) | C12—H12 | 0.9500 |
| N2—C2 | 1.467 (3) | C13—C14 | 1.388 (3) |
| N3—C1 | 1.342 (3) | C13—H13 | 0.9500 |
| N3—C7 | 1.457 (3) | C14—C15 | 1.382 (3) |
| N3—C6 | 1.476 (3) | C14—H14 | 0.9500 |
| C2—H2A | 0.9800 | C15—C16 | 1.390 (3) |
| C2—H2B | 0.9800 | C15—H15 | 0.9500 |
| C2—H2C | 0.9800 | C16—H16 | 0.9500 |
| C3—H3A | 0.9800 | C17—C22 | 1.401 (3) |
| C3—H3B | 0.9800 | C17—C18 | 1.405 (3) |
| C3—H3C | 0.9800 | C18—C19 | 1.390 (3) |
| C4—H4A | 0.9800 | C18—H18 | 0.9500 |
| C4—H4B | 0.9800 | C19—C20 | 1.381 (3) |
| C4—H4C | 0.9800 | C19—H19 | 0.9500 |
| C5—C6 | 1.529 (3) | C20—C21 | 1.391 (3) |
| C5—H5A | 0.9900 | C20—H20 | 0.9500 |
| C5—H5B | 0.9900 | C21—C22 | 1.396 (3) |
| C6—H6A | 0.9900 | C21—H21 | 0.9500 |
| C6—H6B | 0.9900 | C22—H22 | 0.9500 |
| C7—C8 | 1.520 (3) | C23—C24 | 1.399 (3) |
| C7—H7A | 0.9900 | C23—C28 | 1.405 (3) |
| C7—H7B | 0.9900 | C24—C25 | 1.392 (3) |
| C8—O1 | 1.192 (3) | C24—H24 | 0.9500 |
| C8—O2A | 1.307 (4) | C25—C26 | 1.390 (3) |
| C8—O2B | 1.475 (6) | C25—H25 | 0.9500 |
| O2A—C9A | 1.456 (7) | C26—C27 | 1.385 (3) |
| O2B—C9B | 1.461 (10) | C26—H26 | 0.9500 |
| C9A—C10A | 1.506 (8) | C27—C28 | 1.396 (3) |
| C9A—H9A | 0.9900 | C27—H27 | 0.9500 |
| C9A—H9B | 0.9900 | C28—H28 | 0.9500 |
| C9B—C10B | 1.470 (12) | C29—C30 | 1.401 (3) |
| C9B—H9C | 0.9900 | C29—C34 | 1.408 (3) |
| C9B—H9D | 0.9900 | C30—C31 | 1.392 (3) |
| C10A—H10A | 0.9800 | C30—H30 | 0.9500 |
| C10A—H10B | 0.9800 | C31—C32 | 1.391 (3) |
| C10A—H10C | 0.9800 | C31—H31 | 0.9500 |
| C10B—H10D | 0.9800 | C32—C33 | 1.380 (3) |
| C10B—H10E | 0.9800 | C32—H32 | 0.9500 |
| C10B—H10F | 0.9800 | C33—C34 | 1.398 (3) |
| B1—C17 | 1.639 (3) | C33—H33 | 0.9500 |
| B1—C23 | 1.641 (3) | C34—H34 | 0.9500 |
| B1—C29 | 1.645 (3) | | |

| | | | |
|---|---|---|---|
| C1—N1—C4 | 124.65 (18) | C17—B1—C23 | 112.33 (17) |
| C1—N1—C5 | 110.29 (17) | C17—B1—C29 | 103.45 (16) |
| C4—N1—C5 | 120.06 (18) | C23—B1—C29 | 112.97 (17) |
| C1—N2—C3 | 122.92 (18) | C17—B1—C11 | 112.52 (17) |
| C1—N2—C2 | 120.90 (18) | C23—B1—C11 | 102.89 (16) |
| C3—N2—C2 | 116.11 (17) | C29—B1—C11 | 113.02 (17) |
| C1—N3—C7 | 122.96 (18) | C12—C11—C16 | 115.12 (18) |
| C1—N3—C6 | 110.22 (17) | C12—C11—B1 | 121.85 (18) |
| C7—N3—C6 | 121.10 (17) | C16—C11—B1 | 122.54 (18) |
| N1—C1—N2 | 124.52 (18) | C13—C12—C11 | 122.92 (19) |
| N1—C1—N3 | 112.45 (18) | C13—C12—H12 | 118.5 |
| N2—C1—N3 | 123.02 (19) | C11—C12—H12 | 118.5 |
| N2—C2—H2A | 109.5 | C14—C13—C12 | 119.9 (2) |
| N2—C2—H2B | 109.5 | C14—C13—H13 | 120.0 |
| H2A—C2—H2B | 109.5 | C12—C13—H13 | 120.0 |
| N2—C2—H2C | 109.5 | C15—C14—C13 | 119.06 (19) |
| H2A—C2—H2C | 109.5 | C15—C14—H14 | 120.5 |
| H2B—C2—H2C | 109.5 | C13—C14—H14 | 120.5 |
| N2—C3—H3A | 109.5 | C14—C15—C16 | 120.2 (2) |
| N2—C3—H3B | 109.5 | C14—C15—H15 | 119.9 |
| H3A—C3—H3B | 109.5 | C16—C15—H15 | 119.9 |
| N2—C3—H3C | 109.5 | C15—C16—C11 | 122.8 (2) |
| H3A—C3—H3C | 109.5 | C15—C16—H16 | 118.6 |
| H3B—C3—H3C | 109.5 | C11—C16—H16 | 118.6 |
| N1—C4—H4A | 109.5 | C22—C17—C18 | 115.21 (19) |
| N1—C4—H4B | 109.5 | C22—C17—B1 | 122.55 (18) |
| H4A—C4—H4B | 109.5 | C18—C17—B1 | 121.95 (19) |
| N1—C4—H4C | 109.5 | C19—C18—C17 | 122.9 (2) |
| H4A—C4—H4C | 109.5 | C19—C18—H18 | 118.6 |
| H4B—C4—H4C | 109.5 | C17—C18—H18 | 118.6 |
| N1—C5—C6 | 103.74 (17) | C20—C19—C18 | 120.4 (2) |
| N1—C5—H5A | 111.0 | C20—C19—H19 | 119.8 |
| C6—C5—H5A | 111.0 | C18—C19—H19 | 119.8 |
| N1—C5—H5B | 111.0 | C19—C20—C21 | 118.6 (2) |
| C6—C5—H5B | 111.0 | C19—C20—H20 | 120.7 |
| H5A—C5—H5B | 109.0 | C21—C20—H20 | 120.7 |
| N3—C6—C5 | 103.22 (17) | C20—C21—C22 | 120.4 (2) |
| N3—C6—H6A | 111.1 | C20—C21—H21 | 119.8 |
| C5—C6—H6A | 111.1 | C22—C21—H21 | 119.8 |
| N3—C6—H6B | 111.1 | C21—C22—C17 | 122.5 (2) |
| C5—C6—H6B | 111.1 | C21—C22—H22 | 118.8 |
| H6A—C6—H6B | 109.1 | C17—C22—H22 | 118.8 |
| N3—C7—C8 | 111.94 (18) | C24—C23—C28 | 115.68 (19) |
| N3—C7—H7A | 109.2 | C24—C23—B1 | 123.65 (18) |
| C8—C7—H7A | 109.2 | C28—C23—B1 | 120.21 (19) |
| N3—C7—H7B | 109.2 | C25—C24—C23 | 122.8 (2) |
| C8—C7—H7B | 109.2 | C25—C24—H24 | 118.6 |

| | | | |
|---|---|---|---|
| H7A—C7—H7B | 107.9 | C23—C24—H24 | 118.6 |
| O1—C8—O2A | 124.4 (3) | C26—C25—C24 | 120.1 (2) |
| O1—C8—O2B | 124.4 (3) | C26—C25—H25 | 119.9 |
| O1—C8—C7 | 125.6 (2) | C24—C25—H25 | 119.9 |
| O2A—C8—C7 | 108.4 (2) | C27—C26—C25 | 118.7 (2) |
| O2B—C8—C7 | 106.3 (2) | C27—C26—H26 | 120.6 |
| C8—O2A—C9A | 119.0 (3) | C25—C26—H26 | 120.6 |
| C9B—O2B—C8 | 116.1 (4) | C26—C27—C28 | 120.5 (2) |
| O2A—C9A—C10A | 111.2 (5) | C26—C27—H27 | 119.7 |
| O2A—C9A—H9A | 109.4 | C28—C27—H27 | 119.7 |
| C10A—C9A—H9A | 109.4 | C27—C28—C23 | 122.1 (2) |
| O2A—C9A—H9B | 109.4 | C27—C28—H28 | 118.9 |
| C10A—C9A—H9B | 109.4 | C23—C28—H28 | 118.9 |
| H9A—C9A—H9B | 108.0 | C30—C29—C34 | 115.05 (18) |
| O2B—C9B—C10B | 110.6 (7) | C30—C29—B1 | 122.13 (18) |
| O2B—C9B—H9C | 109.5 | C34—C29—B1 | 122.42 (18) |
| C10B—C9B—H9C | 109.5 | C31—C30—C29 | 123.0 (2) |
| O2B—C9B—H9D | 109.5 | C31—C30—H30 | 118.5 |
| C10B—C9B—H9D | 109.5 | C29—C30—H30 | 118.5 |
| H9C—C9B—H9D | 108.1 | C32—C31—C30 | 120.1 (2) |
| C9A—C10A—H10A | 109.5 | C32—C31—H31 | 119.9 |
| C9A—C10A—H10B | 109.5 | C30—C31—H31 | 119.9 |
| H10A—C10A—H10B | 109.5 | C33—C32—C31 | 118.8 (2) |
| C9A—C10A—H10C | 109.5 | C33—C32—H32 | 120.6 |
| H10A—C10A—H10C | 109.5 | C31—C32—H32 | 120.6 |
| H10B—C10A—H10C | 109.5 | C32—C33—C34 | 120.3 (2) |
| C9B—C10B—H10D | 109.5 | C32—C33—H33 | 119.8 |
| C9B—C10B—H10E | 109.5 | C34—C33—H33 | 119.8 |
| H10D—C10B—H10E | 109.5 | C33—C34—C29 | 122.6 (2) |
| C9B—C10B—H10F | 109.5 | C33—C34—H34 | 118.7 |
| H10D—C10B—H10F | 109.5 | C29—C34—H34 | 118.7 |
| H10E—C10B—H10F | 109.5 | | |

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|---|---|---|---|
| C4—N1—C1—N2 | −23.3 (3) | C23—B1—C17—C22 | −151.06 (18) |
| C5—N1—C1—N2 | −178.10 (19) | C29—B1—C17—C22 | 86.8 (2) |
| C4—N1—C1—N3 | 155.7 (2) | C11—B1—C17—C22 | −35.5 (3) |
| C5—N1—C1—N3 | 0.9 (2) | C23—B1—C17—C18 | 35.4 (3) |
| C3—N2—C1—N1 | −38.3 (3) | C29—B1—C17—C18 | −86.7 (2) |
| C2—N2—C1—N1 | 138.4 (2) | C11—B1—C17—C18 | 150.98 (19) |
| C3—N2—C1—N3 | 142.8 (2) | C22—C17—C18—C19 | 0.3 (3) |
| C2—N2—C1—N3 | −40.5 (3) | B1—C17—C18—C19 | 174.2 (2) |
| C7—N3—C1—N1 | 154.57 (19) | C17—C18—C19—C20 | 0.1 (4) |
| C6—N3—C1—N1 | 1.1 (2) | C18—C19—C20—C21 | −0.3 (3) |
| C7—N3—C1—N2 | −26.4 (3) | C19—C20—C21—C22 | 0.1 (3) |
| C6—N3—C1—N2 | −179.87 (19) | C20—C21—C22—C17 | 0.3 (3) |
| C1—N1—C5—C6 | −2.4 (2) | C18—C17—C22—C21 | −0.5 (3) |
| C4—N1—C5—C6 | −158.52 (19) | B1—C17—C22—C21 | −174.41 (19) |
| C1—N3—C6—C5 | −2.5 (2) | C17—B1—C23—C24 | −145.63 (19) |

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|---|---|---|---|
| C7—N3—C6—C5 | −156.55 (19) | C29—B1—C23—C24 | −29.1 (3) |
| N1—C5—C6—N3 | 2.8 (2) | C11—B1—C23—C24 | 93.1 (2) |
| C1—N3—C7—C8 | 117.9 (2) | C17—B1—C23—C28 | 42.5 (3) |
| C6—N3—C7—C8 | −91.4 (2) | C29—B1—C23—C28 | 159.10 (19) |
| N3—C7—C8—O1 | 14.5 (4) | C11—B1—C23—C28 | −78.7 (2) |
| N3—C7—C8—O2A | −179.8 (4) | C28—C23—C24—C25 | −0.4 (3) |
| N3—C7—C8—O2B | −144.2 (4) | B1—C23—C24—C25 | −172.53 (19) |
| O1—C8—O2A—C9A | 0.8 (8) | C23—C24—C25—C26 | 0.8 (3) |
| C7—C8—O2A—C9A | −165.1 (4) | C24—C25—C26—C27 | −1.0 (3) |
| O1—C8—O2B—C9B | 19.8 (9) | C25—C26—C27—C28 | 0.6 (3) |
| C7—C8—O2B—C9B | 178.8 (5) | C26—C27—C28—C23 | −0.2 (3) |
| C8—O2A—C9A—C10A | −84.2 (6) | C24—C23—C28—C27 | 0.0 (3) |
| C8—O2B—C9B—C10B | 77.9 (7) | B1—C23—C28—C27 | 172.5 (2) |
| C17—B1—C11—C12 | −33.8 (3) | C17—B1—C29—C30 | 80.4 (2) |
| C23—B1—C11—C12 | 87.3 (2) | C23—B1—C29—C30 | −41.3 (3) |
| C29—B1—C11—C12 | −150.53 (19) | C11—B1—C29—C30 | −157.59 (19) |
| C17—B1—C11—C16 | 154.62 (19) | C17—B1—C29—C34 | −91.9 (2) |
| C23—B1—C11—C16 | −84.3 (2) | C23—B1—C29—C34 | 146.4 (2) |
| C29—B1—C11—C16 | 37.9 (3) | C11—B1—C29—C34 | 30.0 (3) |
| C16—C11—C12—C13 | −1.2 (3) | C34—C29—C30—C31 | −0.8 (3) |
| B1—C11—C12—C13 | −173.33 (19) | B1—C29—C30—C31 | −173.7 (2) |
| C11—C12—C13—C14 | 0.1 (3) | C29—C30—C31—C32 | 0.1 (4) |
| C12—C13—C14—C15 | 0.4 (3) | C30—C31—C32—C33 | 0.4 (3) |
| C13—C14—C15—C16 | 0.3 (3) | C31—C32—C33—C34 | −0.3 (3) |
| C14—C15—C16—C11 | −1.5 (3) | C32—C33—C34—C29 | −0.5 (3) |
| C12—C11—C16—C15 | 1.9 (3) | C30—C29—C34—C33 | 1.0 (3) |
| B1—C11—C16—C15 | 174.0 (2) | B1—C29—C34—C33 | 173.88 (19) |

*Hydrogen-bond geometry (Å, º)*

Cg1, Cg2 and Cg3 are the centroids of the C17–C22, C23–C28 and C29–C34 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7*B*···*Cg*1$^{i}$ | 0.99 | 2.77 | 3.694 (3) | 155 |
| C6—H6*A*···*Cg*2$^{ii}$ | 0.99 | 2.96 | 3.870 (3) | 153 |
| C4—H4*B*···*Cg*3$^{iii}$ | 0.98 | 2.79 | 3.758 (3) | 169 |
| C3—H3*B*···*Cg*3 | 0.98 | 2.94 | 3.868 (3) | 159 |

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) *x*, *y*+1, *z*.