The molecular and crystal structures of 2-(3-hydroxypropyl)benzimidazole and its nitrate salt

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2-(3-Hydroxypropyl)-1H-benzimidazole, C_{10}H_{12}N_{2}O, which has potential biological activity, can be used as a ligand for complexation with metals. This compound is an electron donor, due to the lone pair of the nitrogen atom in the imidazole ring. This nitrogen atom also acts as a proton acceptor. In the crystalline phase, the nitrate salt, namely, 2-(3-hydroxypropyl)-1H-benzimidazol-3-ium nitrate, C_{10}H_{13}N_{2}O\cdot\text{NO}_3, has been studied. The protonation of the 2-(3-hydroxypropyl)benzimidazole unit results in significant delocalization of the electron density within the imidazole ring. The salt formation leads to variations in the intermolecular interactions, which were studied by analysis of the Hirshfeld surfaces and two-dimensional fingerprint plots.

1. Chemical context

Benzimidazole derivatives and their complex compounds possess a wide spectrum of biological activity (Salahuddin et al., 2012), including antibacterial (Chkirate et al., 2020), antifungal (Khabnadideh et al., 2012), antiviral (Kharitonova et al., 2017), antiparasitic (Katti et al., 2019), anti-inflammatory and analgesic (Gaba et al., 2014) activities.

Nitrogen-containing heterocycles can be lone-pair donors, forming complex compounds with a metal; in some, the nitrogen heterocycle binds to the metal atom (Mottillo et al., 2015). The lone pair of the cyclic nitrogen atom can be protonated, forming an organic cation (Yan et al., 2009; Yu et al., 2007, Bayar et al., 2018; Chen et al., 2010). It has been shown (Pilipenko & Tananaiko, 1983) that compounds containing a protonated cation are formed as a result of the combination with counter-ions. Such compounds, also called ionic associates, are intermediate compounds between simple salts and complex (coordination) compounds. They have properties similar to those of mixed-ligand complexes, although the properties of the compound as a whole depends on many factors.

In the present paper we report the molecular and crystal structures of 2-(3-hydroxypropyl)benzimidazole (BIZ) and its
nitrile salt (BIZHNO3), which were determined to study the influence of protonation.

2. Structural commentary

Analysis of the molecular structures of the title compounds revealed that the C7–N1 and C7–N2 bonds have different lengths [N1–C7 = 1.322 (4) Å and N2–C7 = 1.352 (4) Å] in the neutral BIZ molecule (Fig. 1) but are equal within standard uncertainties [N1–C7 = 1.329 (2) Å and N2–C7 = 1.331 (2) Å] in its protonated form in BIZHNO3 (Fig. 2). Such a delocalization of the electron density during protonation allows the structure of protonated BIZ molecule to be described as a superposition of two resonance structures, as shown in the scheme below.

The neutral and protonated BIZ molecules differ in the conformation of the hydroxyalkyl substituent (Figs. 1 and 2).

In the neutral BIZ molecule, the hydroxyalkyl substituent is almost coplanar to the benzimidazole fragment [the N2–C7–C8–C9 torsion angle is 15.3 (4)°]. The hydroxyalkyl substituent has an all-trans conformation [C7–C8–C9–C10 = 179.8 (3)° and 178.7 (3)°, respectively]. In the protonated BIZ molecule, the hydroxyalkyl substituent is rotated orthogonally to the benzimidazole fragments [N2–C7–C8–C9 = 103.1 (2)°] and has an ap–sc conformation [C7–C8–C9–C10 and C8–C9–C10–O1 = −179.3 (2)° and −62.3 (2)°, respectively].

3. Supramolecular features

In the crystal, BIZ molecules are linked by O–H···N and N–H···O hydrogen bonds (Table 1). The zigzag chains formed by the N–H···O hydrogen bonds propagate in the [100] direction (Fig. 3, on the left). These chains are connected by O–H···N hydrogen bonds in the [010] and [001] directions (Fig. 3, on the right; the chains are highlighted in blue). In addition, weak C3–H···C3 (π) interactions are observed between the BIZ molecules.

In the crystal of the nitrate salt, the protonated BIZ molecules are connected by N–H···O hydrogen bonds (Table 2),
forming centrosymmetric dimers (Fig. 4). These dimers are linked by the bridging nitrate anions in the [001] direction via N—H···O, O—H···O and C—H···O hydrogen bonds (Fig. 5). Stacking interactions of the head-to-tail type between the imidazole rings of BIZH+ molecules are observed in the [010] direction, the distance between π-systems being 3.502 (2) Å.

4. Hirshfeld surface analysis

Hirshfeld surface analysis (Turner et al., 2017) is one of the modern methods allowing intermolecular interactions to be studied in a more analytical way. This method appears to be effective for comparing the capability of the neutral BIZ molecule and its protonated form to participate in intermolecular interactions of different types. The Hirshfeld surfaces were calculated for the BIZ and BIZH+ molecules using a standard high surface resolution, mapped over $d_{\text{norm}}$ (Fig. 6). Bright-red spots are observed for all the donors and acceptors of strong hydrogen bonds in the two structures under study, indicating their participation in intermolecular interactions. It should be noted that the bright-red spot on the N1 atom in the BIZ molecule indicates its capability to be protonated or participate in complexation with a metal.

The two-dimensional fingerprint plots constructed for the BIZ and BIZH+ molecules show that the hydrogen bonds are stronger in the structure of the nitrate salt (see the sharp spikes in Fig. 6). To compare intermolecular interactions of different types in the structures under study, we have analysed their contributions to the total Hirshfeld surfaces (Fig. 7). As can be seen from the histogram, the protonation of the BIZ molecule and presence of the nitrate anion results in a significant increase of the contribution of O—H···O interactions associated with X—H···O hydrogen bonds. In addition, the contributions of N—C/C—N and C—C interactions indicate that stacking between imidazole rings also increases in the BIZHNO3 structure (Fig. 7). A significant decrease in the contribution of N—H/H···N interactions (X—H···N bonding) in the BIZHNO3 structure can be explained by the protonation of the N1 atom, which participates as proton acceptor of hydrogen bonds in the BIZ structure. The different contributions of C—C/H···C interactions associated with X—H···C (π) hydrogen bonds coin-
cide with the presence of a C—H···C(π) hydrogen bond in the BIZ structure (Table 1) and the absence of similar interactions in the BIZHNO3 structure (Table 2). The nitrate anions act as bridging moieties in the BIZHNO3 structure, which results in an increase in the distances between BIZH+ molecules. This fact can explain the decrease in the contribution of H···H interactions in the BIZHNO3 structure (Fig. 7).

5. Database survey

A search of the Cambridge Structural Database (CSD, version 5.42, update of November 2020; Groom et al., 2016) revealed three structures containing the BIZ molecule [refcodes FIYXAN and FIYXER (Elmalı et al., 2005) and RIYNUL (Zhao et al., 2019)]. Two of these structures (FIYXAN and FIYXER) contain protonated BIZ molecules, which form salts with PtCl42− or PtCl63− anions. In the RIYNUL structure, the BIZ molecule forms a coordination bond with the Cd atom.

In addition, three structures with a close analogue of the BIZ molecule containing a carboxylic group instead of a hydroxyl group were found in the CSD [refcodes JOOOROZ (Fu et al., 2016), NOVCEI (Liu et al., 2015) and TILGOL (Zeng et al., 2007)]. In all of these structures, the organic ligand forms an N—M+ coordination bond with participation of the N2 atom of the imidazole ring.

6. Synthesis and crystallization

All chemicals were obtained from commercial sources and used directly without further purification. 1,2-Phenylene diamine (2.16 g, 0.02 mol) was dissolved in hydrochloric acid (25 mL, 4 M) at 373 K, and γ-hydroxybutyric acid (2.82 g, 0.02 mol) was added to the solution. The mixture was heated with reflux for 6 h at 398 K. After cooling to room temperature, the mixture was neutralized using NaOH (pH 7–9). The product was dissolved in aqueous ethanol and treated with activated carbon for purification. The 2-hydroxypropyl-benzimidazole precipitate was filtered off and dried in air. Pale-beige single crystals of the title compound suitable for X-ray diffraction analysis were recrystallized from ethanol solution by slow evaporation, yield 80%, m.p. 437 K.

Synthesis of the \([\text{BIZH}^+\text{NO}_3^-]\) salt:

A weighed portion of copper nitrate \((3 \times 10^{-3} \text{ mol})\) was dissolved in a minimum amount of water and mixed with an alcoholic saturated solution of the ligand \((6 \times 10^{-3} \text{ mol})\) while heating in a water bath. The solution turned green. The solution was then acidified with nitric acid to pH 5 to prevent
the precipitation of hydroxides. The reaction was carried out for 40 minutes while heating in a water bath, after which the reaction mixture was allowed to crystallize. After three days, the precipitated light-yellow crystals were separated, washed with ethanol, and dried in air. The product yield was 62%, m.p. 371–373 K.

7. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 3. All hydrogen atoms were located in difference-Fourier maps. All of the hydrogen atoms in the BIZ structure and H atoms participating in strong hydrogen bonds in the BIZHNO3 structure were refined using an isotropic approximation. Other hydrogen atoms in the BIZHNO3 structure were refined as riding with C

\[
U_{	ext{iso}}(H) = 1.2U_{eq}(C) \quad \text{for the methylene fragments or} \\
U_{	ext{iso}}(H) = 1.2U_{eq}(C) \quad \text{for the aromatic rings.}
\]

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Computing details
For both structures, data collection: CrysAlis PRO (Oxford Diffraction, 2009); cell refinement: CrysAlis PRO (Oxford Diffraction, 2009); data reduction: CrysAlis PRO (Oxford Diffraction, 2009); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL2016/6 (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

2-(3-Hydroxypropyl)-1H-benzimidazole (BIZ)

Crystal data

\[ \text{C}_{10}\text{H}_{12}\text{N}_{2}\text{O} \quad M_r = 176.22 \]
Orthorhombic, \( P2_12_12_1 \)
a = 5.852 (2) Å
b = 12.437 (3) Å
c = 12.444 (3) Å
\( V = 905.7 (4) \text{ Å}^3 \)
Z = 4
\( F(000) = 376 \)

\( D_\text{c} = 1.292 \text{ Mg m}^{-3} \)
Cu \( K\alpha \) radiation, \( \lambda = 1.54178 \text{ Å} \)
Cell parameters from 1839 reflections
\( \theta = 5.2\text{--}32.4^\circ \)
\( \mu = 0.69 \text{ mm}^{-1} \)
\( T = 293 \text{ K} \)
Block, colorless
\( 0.12 \times 0.10 \times 0.08 \text{ mm} \)

Data collection

Oxford Diffraction Xcalfibur, Ruby diffractometer
Radiation source: Enhance (Cu) X-ray Source
Detector resolution: 10.2576 pixels mm\(^{-1} \)
\( \omega \) scans
Absorption correction: multi-scan
(CrysAlisPro; Oxford Diffraction, 2009)
3945 measured reflections
1371 independent reflections
1191 reflections with \( I > 2\sigma(I) \)
\( R_{\text{int}} = 0.029 \)
\( \theta_{\text{max}} = 62.0^\circ, \theta_{\text{min}} = 5.0^\circ \)
h = −6→6
\( k = −13→11 \)
l = −12→14

Refinement

Refinement on \( F^2 \)
Least-squares matrix: full
\( R(F^2) = 0.039 \)
\( wR(F^2) = 0.091 \)
\( S = 1.06 \)
1371 reflections
166 parameters
0 restraints

Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
\[ w = 1/[\sigma(F^2) + (0.0573P)^2] \]
where \( P = (F^2 + 2F^2)/3 \)
\( \Delta\sigma_{\text{max}} < 0.001 \)
Δρ_{\text{max}} = 0.11 \text{ e Å}^{-3}
Δρ_{\text{min}} = -0.19 \text{ e Å}^{-3}

Absolute structure: Flack x determined using 428 quotients \([I^{-}] / [I^{+}]\) (Parsons et al., 2013)
Absolute structure parameter: 0.1 (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)

| Atom | x     | y     | z     | U_{iso}/U_{eq} |
|------|-------|-------|-------|----------------|
| O1   | -0.0261 (5) | 0.38855 (16) | 0.5824 (2) | 0.0635 (7) |
| H1   | -0.080 (6)   | 0.439 (3)   | 0.621 (3)   | 0.083 (14)* |
| N1   | 0.6471 (4)   | 0.43719 (19) | 0.2047 (2) | 0.0470 (6) |
| N2   | 0.6271 (4)   | 0.2901 (2)  | 0.3056 (2)  | 0.0457 (7) |
| H2N  | 0.587 (5)    | 0.243 (2)   | 0.350 (3)   | 0.045 (9)*  |
| C1   | 0.8204 (5)   | 0.3668 (2)  | 0.1742 (2)  | 0.0454 (7) |
| C2   | 0.9878 (6)   | 0.3766 (3)  | 0.0962 (3)  | 0.0559 (9) |
| H2   | 0.994 (6)    | 0.444 (3)   | 0.051 (3)   | 0.068 (10)* |
| C3   | 1.1430 (6)   | 0.2942 (3)  | 0.0847 (3)  | 0.0631 (9) |
| H3   | 1.268 (7)    | 0.297 (3)   | 0.034 (3)   | 0.076 (11)* |
| C4   | 1.1335 (6)   | 0.2037 (3)  | 0.1499 (3)  | 0.0616 (10) |
| H4   | 1.232 (6)    | 0.148 (3)   | 0.144 (3)   | 0.081 (12)* |
| C5   | 0.9683 (6)   | 0.1915 (3)  | 0.2277 (3)  | 0.0555 (9) |
| H5   | 0.961 (6)    | 0.128 (3)   | 0.274 (3)   | 0.075 (11)* |
| C6   | 0.8096 (5)   | 0.2745 (2)  | 0.2377 (2)  | 0.0446 (7) |
| C7   | 0.5365 (5)   | 0.3875 (2)  | 0.2830 (2)  | 0.0427 (7) |
| C8   | 0.3317 (6)   | 0.4331 (3)  | 0.3370 (3)  | 0.0505 (8) |
| H8A  | 0.369 (6)    | 0.511 (3)   | 0.353 (3)   | 0.076 (11)* |
| H8B  | 0.206 (6)    | 0.431 (3)   | 0.286 (3)   | 0.059 (9)*  |
| C9   | 0.2555 (6)   | 0.3800 (3)  | 0.4403 (3)  | 0.0485 (8) |
| H9A  | 0.376 (6)    | 0.382 (3)   | 0.493 (3)   | 0.055 (9)*  |
| H9B  | 0.220 (5)    | 0.307 (3)   | 0.428 (3)   | 0.049 (8)*  |
| C10  | 0.0467 (6)   | 0.4349 (3)  | 0.4844 (3)  | 0.0507 (8) |
| H10A | 0.073 (5)    | 0.512 (3)   | 0.495 (3)   | 0.051 (8)*  |
| H10B | -0.086 (6)   | 0.432 (3)   | 0.432 (3)   | 0.069 (10)* |

Atomic displacement parameters (Å²)

| Atom | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|------|--------|--------|--------|--------|--------|--------|
| O1   | 0.0956 (18) | 0.0401 (12) | 0.0547 (14) | 0.0140 (12) | 0.0202 (13) | 0.0041 (11) |
| N1   | 0.0527 (15) | 0.0425 (12) | 0.0457 (15) | -0.0018 (11) | -0.0032 (13) | 0.0034 (11) |
| N2   | 0.0518 (15) | 0.0398 (13) | 0.0454 (16) | -0.0046 (11) | -0.0032 (12) | 0.0091 (12) |
| C1   | 0.0476 (17) | 0.0427 (15) | 0.0459 (18) | -0.0053 (14) | -0.0035 (14) | -0.0040 (13) |
| C2   | 0.064 (2)   | 0.0517 (18) | 0.052 (2)   | -0.0131 (17) | 0.0045 (16)  | -0.0032 (16) |
| C3   | 0.060 (2)   | 0.066 (2)  | 0.064 (2)  | -0.0104 (18) | 0.0101 (19)  | -0.016 (2)  |
Geometric parameters (Å, °)

| Bond/Distance                      | Value 1 (units) | Value 2 (units) | Value 3 (units) | Value 4 (units) | Value 5 (units) | Value 6 (units) |
|------------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| C4—O1                              | 1.414 (4)       | C4—C5           | 1.376 (5)       |                  |                  |                  |
| C4—C5                              | 1.376 (5)       | C4—H4           | 0.91 (4)        |                  |                  |                  |
| N1—C7                              | 1.322 (4)       | C5—C6           | 1.394 (4)       |                  |                  |                  |
| N1—C1                              | 1.393 (4)       | C5—H5           | 0.98 (4)        |                  |                  |                  |
| N2—C7                              | 1.352 (4)       | C7—C8           | 1.486 (5)       |                  |                  |                  |
| N2—C6                              | 1.376 (4)       | C8—C9           | 1.513 (4)       |                  |                  |                  |
| N2—H2N                             | 0.84 (3)        | C8—H8A          | 1.02 (4)        |                  |                  |                  |
| C1—C2                              | 1.385 (4)       | C8—H8B          | 0.97 (3)        |                  |                  |                  |
| C1—C6                              | 1.395 (4)       | C9—C10          | 1.503 (5)       |                  |                  |                  |
| C2—C3                              | 1.377 (5)       | C9—H9A          | 0.96 (4)        |                  |                  |                  |
| C2—H2                              | 1.01 (4)        | C9—H9B          | 0.94 (3)        |                  |                  |                  |
| C3—C4                              | 1.389 (5)       | C10—H10A        | 0.98 (3)        |                  |                  |                  |
| C3—H3                              | 0.97 (4)        | C10—H10B        | 1.01 (4)        |                  |                  |                  |
| C10—O1—H1                          | 107 (3)         | C5—C6—C1        | 121.9 (3)       |                  |                  |                  |
| C7—N1—C1                           | 105.3 (2)       | N1—C7—N2        | 112.3 (3)       |                  |                  |                  |
| C7—N2—C6                           | 107.6 (3)       | N1—C7—C8        | 123.4 (3)       |                  |                  |                  |
| C7—N2—H2N                         | 131 (2)         | N2—C7—C8        | 124.3 (3)       |                  |                  |                  |
| C6—N2—H2N                         | 121 (2)         | C7—C8—C9        | 117.1 (3)       |                  |                  |                  |
| C2—C1—N1                           | 130.6 (3)       | C7—C8—H8A      | 106 (2)         |                  |                  |                  |
| C2—C1—C6                           | 120.1 (3)       | C9—C8—H8A      | 109 (2)         |                  |                  |                  |
| N1—C1—C6                           | 109.3 (3)       | C7—C8—H8B      | 107 (2)         |                  |                  |                  |
| C3—C2—C1                           | 118.3 (3)       | C9—C8—H8B      | 109 (2)         |                  |                  |                  |
| C3—C2—H2                           | 123 (2)         | H8A—C8—H8B     | 109 (3)         |                  |                  |                  |
| C1—C2—H2                           | 119 (2)         | C10—C9—C8      | 110.6 (3)       |                  |                  |                  |
| C2—C3—C4                           | 121.1 (4)       | C10—C9—H9A     | 109.7 (19)      |                  |                  |                  |
| C2—C3—H3                           | 123 (2)         | C8—C9—H9A      | 111 (2)         |                  |                  |                  |
| C4—C3—H3                           | 116 (2)         | C10—C9—H9B     | 108.7 (19)      |                  |                  |                  |
| C5—C4—C3                           | 121.9 (4)       | C8—C9—H9B      | 110.1 (19)      |                  |                  |                  |
| C5—C4—H4                           | 115 (2)         | H9A—C9—H9B     | 107 (3)         |                  |                  |                  |
| C3—C4—H4                           | 123 (2)         | O1—C10—C9      | 112.0 (3)       |                  |                  |                  |
| C4—C5—C6                           | 116.7 (3)       | O1—C10—H10A    | 109.1 (19)      |                  |                  |                  |
| C4—C5—H5                           | 122 (2)         | C9—C10—H10A    | 111.5 (19)      |                  |                  |                  |
| C6—C5—H5                           | 121 (2)         | O1—C10—H10B    | 108 (2)         |                  |                  |                  |
| N2—C6—C5                           | 132.6 (3)       | C9—C10—H10B    | 112 (2)         |                  |                  |                  |
| N2—C6—C1                           | 105.5 (3)       | H10A—C10—H10B  | 104 (3)         |                  |                  |                  |
| C7—N1—C1—C2                        | 179.9 (3)       | N1—C1—C6—N2    | 0.5 (3)         |                  |                  |                  |
C7—N1—C1—C6  −0.5 (3) C2—C1—C6—C5  2.1 (4)
N1—C1—C2—C3  178.5 (3) N1—C1—C6—C5  −177.5 (3)
C6—C1—C2—C3  −1.0 (4) C1—N1—C7—N2  0.3 (3)
C1—C2—C3—C4  −0.4 (5) C1—N1—C7—C8  −177.5 (3)
C2—C3—C4—C5  0.8 (5) C6—N2—C7—C8  0.0 (3)
C3—C4—C5—C6  0.2 (5) C6—N2—C7—C8  178.2 (3)
C6—C1—C2—C3  −1.0 (4) C1—N1—C7—C8  0.0 (3)
C2—C1—C6—N2  −179.9 (3) C7—N2—C6—C1  0.3 (3)
C4—C5—C6—C1  −1.7 (5) N2—C7—C8—C9  15.3 (4)
C4—C5—C6—C1  −1.7 (5) N2—C7—C8—C9  178.2 (3)
C7—N2—C6—C1  0.0 (3) N1—C7—C8—C9  −166.7 (3)
C7—N2—C6—C1  0.0 (3) N1—C7—C8—C9  −166.7 (3)
C7—N2—C6—C1  0.0 (3) C6—N2—C7—C8  178.2 (3)
C4—C5—C6—C1  −1.7 (5) C7—N2—C6—C1  0.0 (3)
C2—C1—C6—N2  −179.9 (3) C7—N2—C6—C1  0.0 (3)
C2—C1—C6—N2  −179.9 (3) C7—N2—C6—C1  0.0 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H   | H···A | D···A   | D—H···A |
|-----------|-------|-------|---------|---------|
| N2—H2N···O1i | 0.84 (3) | 1.95 (3) | 2.772 (3) | 165 (3) |
| O1—H1···N1ii | 0.85 (4) | 1.90 (4) | 2.741 (3) | 169 (4) |
| C3—H3···C3iii | 0.97 (4) | 2.87 (4) | 3.770 (4) | 154 (3) |

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

2-(3-Hydroxypropyl)-1H-benzimidazol-3-ium nitrate (BIZHNO3)

Crystal data

C_{10}H_{13}N_{2}O^{+}·NO_{3}^{-}  
F(000) = 504  
M_r = 239.23  
Monoclinic, P2_1/n  
a = 8.5100 (3) Å  
b = 8.2525 (4) Å  
c = 16.5130 (7) Å  
β = 93.760 (4)°  
V = 1157.19 (9) Å^3  
Z = 4  

Data collection

Oxford Diffraction Xcalibur, Ruby diffractometer  
Radiation source: Enhance (Cu) X-ray Source  
Detector resolution: 10.2576 pixels mm^{-1}  
Cell parameters from 1670 reflections  
θ = 5.2–75.5°  
µ = 0.91 mm^{-1}  
T = 293 K  
Block, colorless  
0.12 × 0.10 × 0.08 mm  

Refinement

Refinement on F^2  
Least-squares matrix: full  
R[F^2 > 2σ(F^2)] = 0.044  
wR(F^2) = 0.128  
S = 1.04  
2345 reflections  
167 parameters  
0 restraints  
Primary atom site location: difference Fourier map  
Secondary atom site location: difference Fourier map  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement

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\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0715P)^2 + 0.0026P} \]

where \( P = (F_o^2 + 2F_c^2)/3 \)

\( (\Delta/\sigma)_{\text{max}} < 0.001 \)

\[ \Delta \rho_{\text{max}} = 0.16 \text{ e Å}^{-3} \]

\[ \Delta \rho_{\text{min}} = -0.15 \text{ e Å}^{-3} \]

**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     | U_iso*/U_eq |
|----|-------|-------|-------|-------------|
|   |       |       |       |             |
| O1 | 1.1212 (17) | 0.57042 (19) | 0.61912 (9) | 0.0691 (5) |
| H1O | 1.102 (4) | 0.662 (3) | 0.6461 (18) | 0.112 (10)* |
| O2 | 0.4689 (2) | 0.27088 (18) | 0.74998 (8) | 0.0732 (5) |
| O3 | 0.4922 (3) | 0.5257 (2) | 0.74234 (10) | 0.1045 (7) |
| O4 | 0.3822 (2) | 0.4195 (2) | 0.84232 (10) | 0.1011 (6) |
| N1 | 0.65129 (19) | 0.34198 (19) | 0.47832 (10) | 0.0519 (4) |
| H1N | 0.726 (3) | 0.374 (3) | 0.4442 (14) | 0.075 (7)* |
| N2 | 0.54243 (17) | 0.29223 (18) | 0.59032 (9) | 0.0477 (4) |
| H2N | 0.527 (3) | 0.290 (3) | 0.6412 (13) | 0.065 (6)* |
| C1 | 0.5164 (2) | 0.2529 (2) | 0.45731 (11) | 0.0464 (4) |
| C2 | 0.4513 (3) | 0.1965 (2) | 0.38345 (12) | 0.0591 (5) |
| H2 | 0.498209 | 0.216950 | 0.335175 | 0.071* |
| N3 | 0.4465 (2) | 0.4091 (2) | 0.77860 (10) | 0.0616 (4) |
| C3 | 0.3151 (3) | 0.1095 (2) | 0.38500 (13) | 0.0663 (6) |
| H3 | 0.268665 | 0.069383 | 0.336521 | 0.080* |
| C4 | 0.2433 (2) | 0.0789 (2) | 0.45704 (14) | 0.0641 (6) |
| H4 | 0.149566 | 0.020639 | 0.455243 | 0.077* |
| C5 | 0.3081 (2) | 0.1330 (2) | 0.53068 (12) | 0.0541 (5) |
| H5 | 0.261444 | 0.111399 | 0.578915 | 0.065* |
| C6 | 0.44674 (19) | 0.22143 (19) | 0.52923 (10) | 0.0444 (4) |
| C7 | 0.6645 (2) | 0.3629 (2) | 0.55826 (11) | 0.0486 (4) |
| C8 | 0.7967 (2) | 0.4460 (2) | 0.60431 (12) | 0.0576 (5) |
| H8A | 0.765574 | 0.471662 | 0.658317 | 0.069* |
| H8B | 0.819160 | 0.547104 | 0.577420 | 0.069* |
| C9 | 0.9449 (2) | 0.3426 (2) | 0.61118 (13) | 0.0567 (5) |
| H9A | 0.921976 | 0.240986 | 0.637422 | 0.068* |
| H9B | 0.976665 | 0.318146 | 0.557167 | 0.068* |
| C10 | 1.0784 (2) | 0.4255 (3) | 0.65864 (12) | 0.0618 (5) |
| H10A | 1.047114 | 0.450854 | 0.712636 | 0.074* |
| H10B | 1.168414 | 0.353286 | 0.664051 | 0.074* |
### Atomic displacement parameters (Å²)

| Atom | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|------|-----------|-----------|-----------|-----------|-----------|-----------|
| O1   | 0.0677 (10) | 0.0726 (10) | 0.0693 (9) | −0.0224 (8) | 0.0217 (7) | −0.0162 (8) |
| O2   | 0.1068 (13) | 0.0561 (8) | 0.0580 (8) | 0.0013 (8) | 0.0154 (8) | −0.0021 (7) |
| O3   | 0.1610 (19) | 0.0591 (10) | 0.0956 (13) | −0.0156 (11) | 0.0260 (13) | 0.0054 (9) |
| O4   | 0.1175 (15) | 0.1153 (15) | 0.0749 (11) | 0.0069 (12) | 0.0408 (10) | −0.0192 (10) |
| N1   | 0.0507 (9)  | 0.0494 (8) | 0.0569 (9) | −0.0031 (7) | 0.0136 (7) | 0.0017 (7)  |
| N2   | 0.0479 (8)  | 0.0489 (8) | 0.0468 (8) | −0.0003 (6) | 0.0071 (7) | −0.0006 (7) |
| C1   | 0.0469 (9)  | 0.0403 (8) | 0.0521 (9) | 0.0048 (7) | 0.0040 (7) | 0.0032 (7)  |
| C2   | 0.0714 (13) | 0.0556 (11) | 0.0498 (10) | 0.0078 (10) | −0.0003 (9) | 0.0008 (9)  |
| N3   | 0.0658 (11) | 0.0633 (10) | 0.0556 (10) | 0.0006 (8) | 0.0026 (8) | −0.0041 (8) |
| C3   | 0.0695 (14) | 0.0583 (12) | 0.0681 (13) | 0.0051 (10) | −0.0170 (11) | −0.0093 (10) |
| C4   | 0.0478 (11) | 0.0521 (11) | 0.0911 (16) | −0.0028 (9) | −0.0055 (10) | −0.0061 (10) |
| C5   | 0.0473 (10) | 0.0466 (10) | 0.0691 (12) | −0.0012 (8) | 0.0096 (9) | 0.0020 (9)  |
| C6   | 0.0433 (9)  | 0.0393 (8) | 0.0509 (9) | 0.0033 (7) | 0.0041 (7) | 0.0011 (7)  |
| C7   | 0.0453 (9)  | 0.0427 (9) | 0.0582 (10) | 0.0022 (7) | 0.0057 (8) | −0.0029 (8) |
| C8   | 0.0489 (11) | 0.0524 (10) | 0.0715 (13) | −0.0021 (8) | 0.0041 (9) | −0.0132 (9) |
| C9   | 0.0548 (11) | 0.0457 (9) | 0.0685 (11) | 0.0003 (8) | −0.0027 (9) | −0.0031 (9) |
| C10  | 0.0558 (11) | 0.0662 (12) | 0.0622 (12) | 0.0003 (10) | −0.0053 (9) | −0.0056 (10) |

### Geometric parameters (Å, °)

| Bond/Angle | Length/Distance | Angle |
|------------|-----------------|-------|
| O1—C10     | 1.421 (2)       |       |
| O1—H1O     | 0.90 (3)        |       |
| O2—N3      | 1.254 (2)       |       |
| O3—N3      | 1.211 (2)       |       |
| O4—N3      | 1.220 (2)       |       |
| N1—C7      | 1.329 (2)       |       |
| N1—C1      | 1.388 (2)       |       |
| N1—H1N     | 0.91 (2)        |       |
| N2—C7      | 1.331 (2)       |       |
| N2—C6      | 1.384 (2)       |       |
| N2—H2N     | 0.86 (2)        |       |
| C1—C6      | 1.386 (2)       |       |
| C1—C2      | 1.387 (3)       |       |
| C2—C3      | 1.365 (3)       |       |
| C2—H2      | 0.9300          |       |
| C10—O1—H1O| 114.7 (19)      |       |
| C7—N1—C1  | 109.37 (16)     |       |
| C7—N1—H1N | 124.0 (14)      |       |
| C1—N1—H1N | 126.4 (14)      |       |
| C7—N2—C6  | 109.41 (15)     |       |
| C7—N2—H2N | 125.0 (15)      |       |
| C6—N2—H2N | 125.6 (15)      |       |
| C6—C1—C2  | 121.46 (17)     |       |
| C6—C1—N1  | 106.16 (15)     |       |

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C2—C1—N1 132.37 (18) C7—C8—H8B 109.2
C3—C2—C1 116.80 (19) C9—C8—H8B 109.2
C3—C2—H2 121.6 H8A—C8—H8B 107.9
C1—C2—H2 121.6 C10—C9—C8 112.27 (16)
O3—N3—O4 123.2 (2) C10—C9—H9A 109.2
O3—N3—O2 118.34 (18) C8—C9—H9A 109.2
O4—N3—O2 118.47 (18) C10—C9—H9B 109.2
C2—C3—C4 122.06 (19) C8—C9—H9B 109.2
C2—C3—H3 119.0 H9A—C9—H9B 107.9
C4—C3—C4 119.0 O1—C10—C9 110.57 (17)
C5—C4—C3 121.47 (19) O1—C10—H10A 109.5
C5—C4—H4 119.3 C9—C10—H10A 109.5
C3—C4—H4 119.3 O1—C10—H10B 109.5
C4—C5—C6 119.3 C9—C10—H10B 109.5
C4—C5—H5 119.0 H10A—C10—H10B 108.1
C6—C5—H5 119.0

Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H  | H···A | D···A  | D—H···A |
|-----------|------|------|--------|---------|
| N2—H2N···O2 | 0.86 (2) | 1.90 (2) | 2.755 (2) | 173 (2) |
| N1—H1N···O1i | 0.91 (2) | 1.78 (2) | 2.696 (2) | 177 (2) |
| O1—H1O···O2ii | 0.90 (3) | 2.06 (3) | 2.866 (2) | 149 (3) |
| O1—H1O···O4i | 0.90 (3) | 2.14 (3) | 2.951 (3) | 150 (3) |
| C5—H5···O4iii | 0.93 | 2.43 | 3.251 (3) | 147 |

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