

# Bis(pyridin-2-ylmethyl)ammonium nitrate

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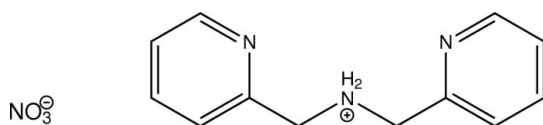
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Key indicators: single-crystal X-ray study;  $T = 200$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.122; data-to-parameter ratio = 17.1.

In the title compound,  $\text{C}_{12}\text{H}_{14}\text{N}_3^+\cdot\text{NO}_3^-$ , the mononitrate of protonated bis(pyridin-2-ylmethyl)amine, the least-squares planes defined by the non-H atoms of the two aromatic moieties intersect at an angle of  $7.91$  ( $6^\circ$ ). In the crystal,  $\text{N}-\text{H}\cdots\text{N}$ ,  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds, as well as  $\text{C}-\text{H}\cdots\text{O}$  contacts, connect the entities into a three-dimensional network. The shortest centroid-centroid distance between two aromatic systems is  $3.7255$  ( $8$ ) Å and is apparent between the two different aromatic moieties.

## Related literature

For the crystal structure of the trinitrate of bis(pyridin-2-ylmethyl)amine, see: Junk *et al.* (2006). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



## Experimental

### Crystal data

$\text{C}_{12}\text{H}_{14}\text{N}_3^+\cdot\text{NO}_3^-$

$M_r = 262.27$

Orthorhombic, *Pbca*

$a = 11.2236$  (2) Å

$b = 13.4714$  (4) Å

$c = 16.5303$  (4) Å

$V = 2499.34$  (11) Å<sup>3</sup>

$Z = 8$

Mo  $K\alpha$  radiation

$\mu = 0.10$  mm<sup>-1</sup>

$T = 200$  K

$0.50 \times 0.35 \times 0.24$  mm

### Data collection

Bruker APEXII CCD

diffractometer

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.923$ ,  $T_{\max} = 1.000$

12816 measured reflections

3082 independent reflections

2568 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.122$

$S = 1.04$

3082 reflections

180 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.29$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                                       | $D-H$      | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|------------|-------------|-------------|---------------|
| $\text{C13}-\text{H13}\cdots\text{N2}$              | 0.95       | 2.61        | 3.5339 (18) | 164           |
| $\text{N1}-\text{H71}\cdots\text{O3}^{\text{I}}$    | 0.893 (18) | 2.349 (18)  | 3.0721 (15) | 138.2 (14)    |
| $\text{N1}-\text{H72}\cdots\text{O2}^{\text{II}}$   | 0.902 (19) | 1.982 (19)  | 2.8831 (15) | 176.1 (15)    |
| $\text{N1}-\text{H72}\cdots\text{O1}^{\text{II}}$   | 0.902 (19) | 2.584 (18)  | 3.2201 (16) | 128.1 (14)    |
| $\text{N1}-\text{H72}\cdots\text{N2}^{\text{II}}$   | 0.902 (19) | 2.651 (18)  | 3.4905 (15) | 155.1 (14)    |
| $\text{C15}-\text{H15}\cdots\text{O2}^{\text{III}}$ | 0.95       | 2.39        | 3.1858 (19) | 141           |
| $\text{C1}-\text{H1A}\cdots\text{O1}^{\text{IV}}$   | 0.99       | 2.54        | 3.5132 (18) | 167           |
| $\text{C25}-\text{H25}\cdots\text{O1}^{\text{V}}$   | 0.95       | 2.53        | 3.3959 (16) | 151           |
| $\text{C24}-\text{H24}\cdots\text{O2}^{\text{VI}}$  | 0.95       | 2.55        | 3.4620 (16) | 161           |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VN2068).

## References

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## supporting information

*Acta Cryst.* (2013). E69, o661 [https://doi.org/10.1107/S1600536813008593]

**Bis(pyridin-2-ylmethyl)ammonium nitrate****Abubak'r Abrahams, Bernardus van Brecht and Richard Betz****S1. Comment**

Upon the attempted synthesis of a rare-earth metal coordination compound applying bis(pyridin-2-ylmethyl)amine as an auxiliary ligand, the title compound was unintentionally obtained as the only crystalline reaction product. The crystal structure of the trinitrate salt of bis(pyridin-2-ylmethyl)amine has been reported earlier (Junk *et al.*, 2006).

The twofold-protonated amine-type nitrogen atom is present in a tetrahedral coordination environment. The angles set up by the atoms connected to it cover a range of 107.7 (11)–111.81 (9) °. The least-squares planes defined by the respective non-hydrogen atoms of the two aromatic moieties enclose an angle of 7.91 (6) ° (Fig. 1).

In the crystal, classical hydrogen bonds of the N–H···O type are observed next to C–H···O contacts whose range falls by more than 0.1 Å below the sum of van-der-Waals radii of the atoms participating. In addition, a C–H···N contact involving the nitrate anion is apparent. One of the N–H···O hydrogen bonds shows bifurcation. All the aforementioned contacts are exclusively established between atoms on the cation as well as the anion. Metrical parameters as well as information about the symmetry of these contacts are summarized in Table 1. In total, the entities of the crystal structure are connected to a three-dimensional network. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for the C–H···O contacts is *DDDD* on the unary level while the classical hydrogen bonds necessitate a *DDD* descriptor on the same level if the bifurcated hydrogen bond is counted as two separate hydrogen bonds. The shortest intercentroid distance between two aromatic systems is measured at 3.7255 (8) Å and is apparent between the two different aromatic moieties (Fig. 2).

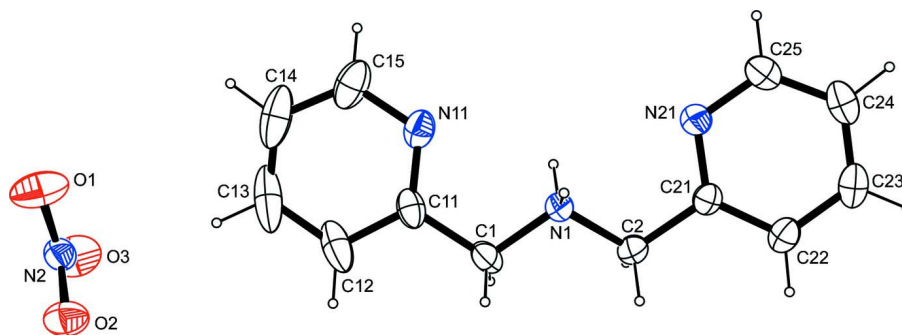
The packing of the title compound in the crystal structure is shown in Figure 3.

**S2. Experimental**

Lanthanum nitrate was reacted with bis(pyridin-2-ylmethyl)amine in water. Upon free evaporation of the solvent, a crystalline solid was obtained from which irregular-shaped white crystals could be selected.

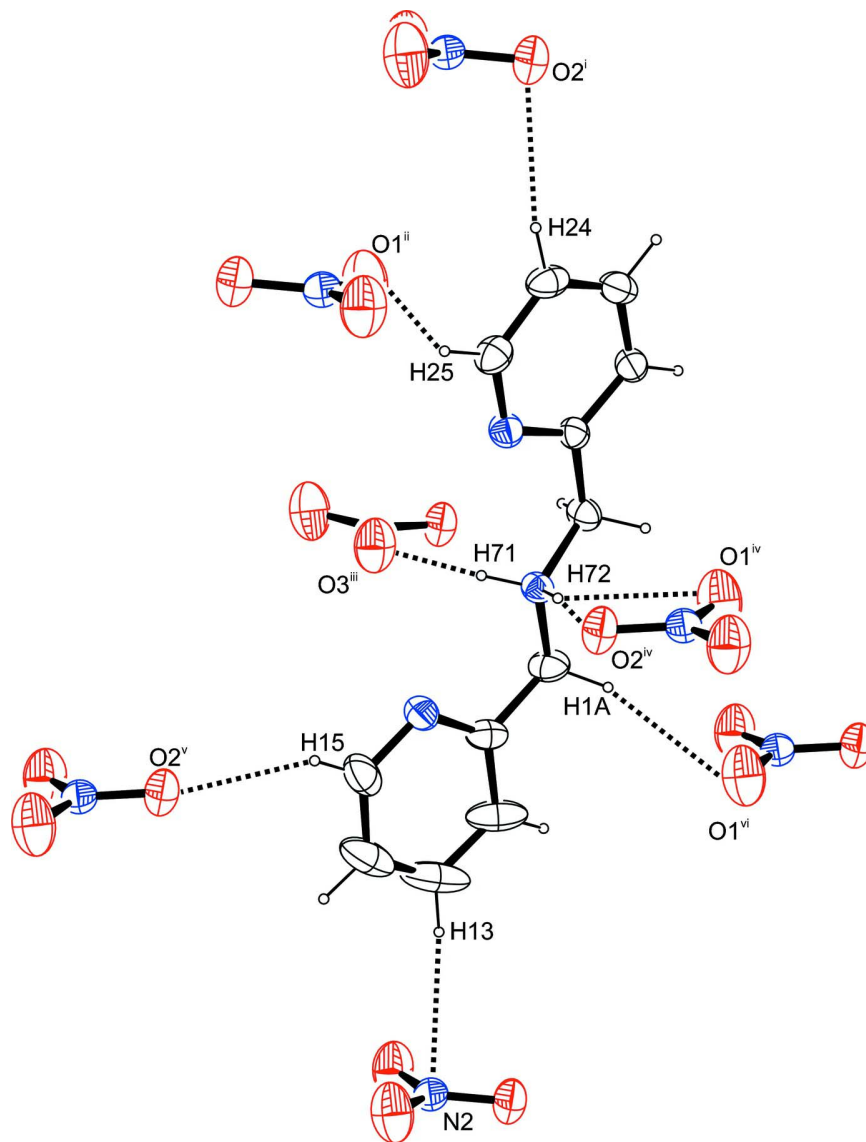
**S3. Refinement**

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms and C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to  $1.2U_{\text{eq}}(\text{C})$ . Both nitrogen-bound H atoms were located on a difference Fourier map and refined freely.



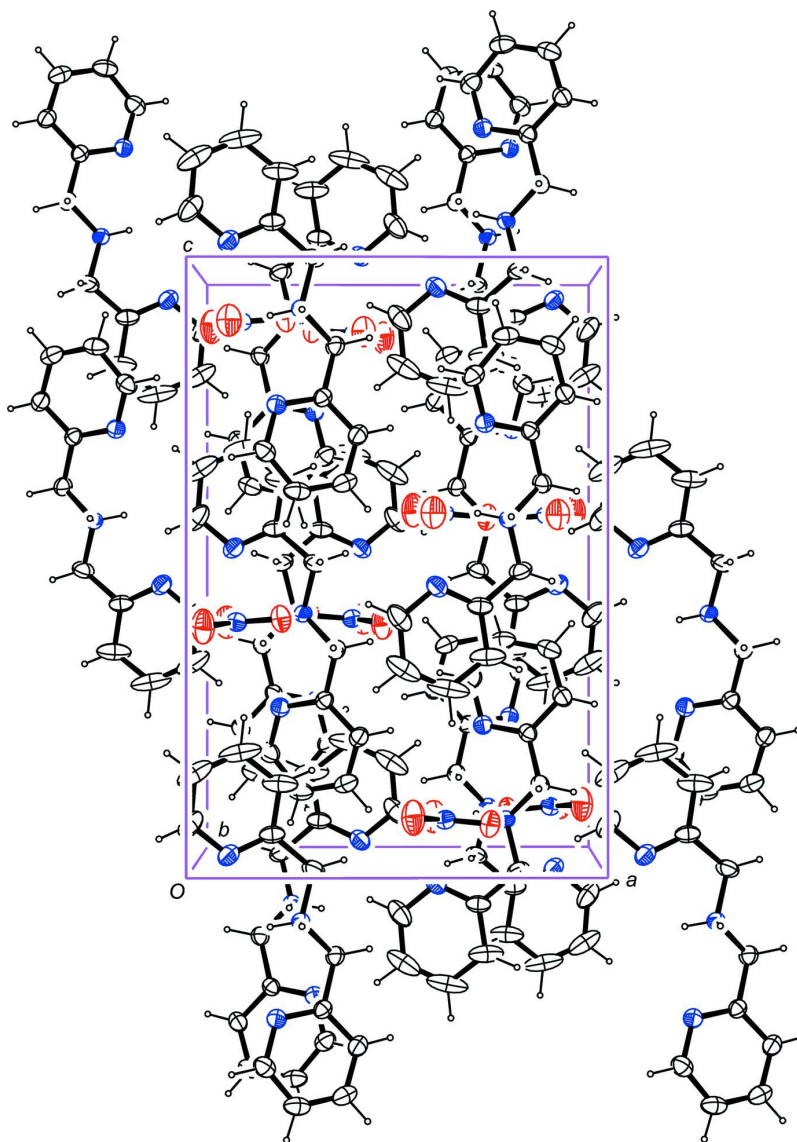
**Figure 1**

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).



**Figure 2**

Intermolecular contacts, viewed approximately along  $[1\ 1\ 0]$ . Symmetry operators: <sup>i</sup>  $x, y, z + 1$ ; <sup>ii</sup>  $-x + 1, -y, -z + 1$ ; <sup>iii</sup>  $x, -y + 1/2, z + 1/2$ ; <sup>iv</sup>  $-x + 3/2, -y, z + 1/2$ ; <sup>v</sup>  $x - 1/2, y, -z + 1/2$ ; <sup>vi</sup>  $x + 1/2, y, -z + 1/2$ .



**Figure 3**

Molecular packing of the title compound, viewed along [0 1 0] (anisotropic displacement ellipsoids drawn at 50% probability level).

### Bis(pyridin-2-ylmethyl)ammonium nitrate

#### Crystal data

$C_{12}H_{14}N_3^+ \cdot NO_3^-$

$M_r = 262.27$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 11.2236$  (2) Å

$b = 13.4714$  (4) Å

$c = 16.5303$  (4) Å

$V = 2499.34$  (11) Å<sup>3</sup>

$Z = 8$

$F(000) = 1104$

$D_x = 1.394$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 5628 reflections

$\theta = 2.5$ – $28.2^\circ$

$\mu = 0.10$  mm<sup>-1</sup>

$T = 200$  K

Irregular, white

$0.50 \times 0.35 \times 0.24$  mm

*Data collection*

Bruker APEXII CCD  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2001)  
 $T_{\min} = 0.923$ ,  $T_{\max} = 1.000$

12816 measured reflections  
3082 independent reflections  
2568 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.014$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.7^\circ$   
 $h = -8 \rightarrow 14$   
 $k = -17 \rightarrow 17$   
 $l = -22 \rightarrow 17$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.122$   
 $S = 1.04$   
3082 reflections  
180 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 atoms treated by a mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 0.627P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| O1  | 0.53822 (10) | 0.03915 (9)  | 0.09542 (8)  | 0.0595 (3)                       |
| O2  | 0.72195 (8)  | 0.07826 (8)  | 0.08342 (6)  | 0.0438 (3)                       |
| O3  | 0.58787 (10) | 0.19315 (8)  | 0.09035 (8)  | 0.0540 (3)                       |
| N1  | 0.76754 (9)  | 0.13548 (8)  | 0.57810 (6)  | 0.0257 (2)                       |
| H71 | 0.6936 (16)  | 0.1564 (13)  | 0.5883 (10)  | 0.044 (4)*                       |
| H72 | 0.7725 (15)  | 0.0688 (14)  | 0.5820 (10)  | 0.044 (4)*                       |
| N2  | 0.61491 (9)  | 0.10403 (8)  | 0.08911 (6)  | 0.0300 (2)                       |
| N11 | 0.59551 (10) | 0.11038 (8)  | 0.46858 (6)  | 0.0345 (2)                       |
| N21 | 0.71063 (9)  | 0.11508 (7)  | 0.73540 (6)  | 0.0297 (2)                       |
| C1  | 0.79740 (12) | 0.16440 (10) | 0.49410 (7)  | 0.0351 (3)                       |
| H1A | 0.8717       | 0.1305       | 0.4774       | 0.042*                           |
| H1B | 0.8116       | 0.2369       | 0.4919       | 0.042*                           |
| C2  | 0.84972 (10) | 0.18126 (9)  | 0.63756 (7)  | 0.0295 (2)                       |
| H2A | 0.8442       | 0.2544       | 0.6335       | 0.035*                           |
| H2B | 0.9326       | 0.1617       | 0.6246       | 0.035*                           |
| C11 | 0.69911 (12) | 0.13770 (8)  | 0.43630 (7)  | 0.0313 (3)                       |
| C12 | 0.71806 (18) | 0.14422 (10) | 0.35348 (8)  | 0.0507 (4)                       |
| H12 | 0.7937       | 0.1623       | 0.3324       | 0.061*                           |
| C13 | 0.6227 (2)   | 0.12345 (11) | 0.30224 (9)  | 0.0657 (6)                       |
| H13 | 0.6315       | 0.1291       | 0.2453       | 0.079*                           |
| C14 | 0.51552 (19) | 0.09468 (12) | 0.33522 (11) | 0.0598 (5)                       |
| H14 | 0.4496       | 0.0794       | 0.3014       | 0.072*                           |
| C15 | 0.50553 (14) | 0.08849 (11) | 0.41766 (10) | 0.0472 (4)                       |
| H15 | 0.4316       | 0.0678       | 0.4400       | 0.057*                           |

|     |              |             |             |            |
|-----|--------------|-------------|-------------|------------|
| C21 | 0.82056 (10) | 0.14965 (8) | 0.72268 (7) | 0.0255 (2) |
| C22 | 0.90516 (11) | 0.16031 (9) | 0.78369 (7) | 0.0309 (3) |
| H22 | 0.9828       | 0.1843      | 0.7719      | 0.037*     |
| C23 | 0.87333 (12) | 0.13503 (9) | 0.86208 (7) | 0.0359 (3) |
| H23 | 0.9287       | 0.1417      | 0.9052      | 0.043*     |
| C24 | 0.75986 (13) | 0.10001 (9) | 0.87633 (7) | 0.0366 (3) |
| H24 | 0.7353       | 0.0829      | 0.9295      | 0.044*     |
| C25 | 0.68233 (11) | 0.09025 (9) | 0.81153 (7) | 0.0346 (3) |
| H25 | 0.6049       | 0.0646      | 0.8216      | 0.041*     |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|-----|-------------|------------|------------|-------------|-------------|-------------|
| O1  | 0.0397 (6)  | 0.0467 (6) | 0.0920 (9) | -0.0119 (5) | -0.0001 (5) | 0.0126 (6)  |
| O2  | 0.0298 (5)  | 0.0473 (6) | 0.0542 (6) | 0.0045 (4)  | 0.0046 (4)  | -0.0013 (4) |
| O3  | 0.0484 (6)  | 0.0330 (5) | 0.0805 (8) | 0.0060 (4)  | -0.0001 (5) | 0.0027 (5)  |
| N1  | 0.0254 (5)  | 0.0290 (5) | 0.0229 (4) | -0.0029 (4) | -0.0008 (3) | 0.0030 (3)  |
| N2  | 0.0309 (5)  | 0.0333 (5) | 0.0260 (5) | 0.0003 (4)  | 0.0013 (4)  | 0.0017 (4)  |
| N11 | 0.0339 (5)  | 0.0374 (5) | 0.0322 (5) | 0.0069 (4)  | -0.0073 (4) | -0.0051 (4) |
| N21 | 0.0281 (5)  | 0.0325 (5) | 0.0284 (5) | -0.0005 (4) | 0.0000 (4)  | 0.0034 (4)  |
| C1  | 0.0395 (6)  | 0.0421 (6) | 0.0236 (5) | -0.0092 (5) | 0.0062 (5)  | 0.0033 (5)  |
| C2  | 0.0270 (5)  | 0.0327 (6) | 0.0287 (6) | -0.0068 (4) | -0.0022 (4) | 0.0014 (4)  |
| C11 | 0.0494 (7)  | 0.0219 (5) | 0.0225 (5) | 0.0018 (5)  | -0.0017 (5) | 0.0013 (4)  |
| C12 | 0.0989 (13) | 0.0300 (6) | 0.0233 (6) | -0.0127 (7) | 0.0039 (7)  | 0.0010 (5)  |
| C13 | 0.138 (2)   | 0.0328 (7) | 0.0259 (7) | 0.0027 (9)  | -0.0235 (9) | -0.0003 (5) |
| C14 | 0.0905 (14) | 0.0376 (7) | 0.0513 (9) | 0.0206 (8)  | -0.0415 (9) | -0.0113 (7) |
| C15 | 0.0444 (8)  | 0.0432 (7) | 0.0542 (9) | 0.0160 (6)  | -0.0214 (6) | -0.0127 (6) |
| C21 | 0.0259 (5)  | 0.0244 (5) | 0.0263 (5) | 0.0028 (4)  | -0.0025 (4) | -0.0014 (4) |
| C22 | 0.0289 (5)  | 0.0323 (6) | 0.0316 (6) | 0.0044 (4)  | -0.0048 (4) | -0.0051 (4) |
| C23 | 0.0464 (7)  | 0.0334 (6) | 0.0278 (6) | 0.0122 (5)  | -0.0096 (5) | -0.0052 (5) |
| C24 | 0.0546 (8)  | 0.0303 (6) | 0.0248 (5) | 0.0119 (5)  | 0.0031 (5)  | 0.0020 (4)  |
| C25 | 0.0367 (7)  | 0.0339 (6) | 0.0332 (6) | 0.0018 (5)  | 0.0063 (5)  | 0.0047 (5)  |

*Geometric parameters (Å, °)*

|         |             |         |             |
|---------|-------------|---------|-------------|
| O1—N2   | 1.2311 (15) | C11—C12 | 1.3883 (17) |
| O2—N2   | 1.2541 (14) | C12—C13 | 1.393 (3)   |
| O3—N2   | 1.2385 (14) | C12—H12 | 0.9500      |
| N1—C1   | 1.4806 (14) | C13—C14 | 1.376 (3)   |
| N1—C2   | 1.4822 (14) | C13—H13 | 0.9500      |
| N1—H71  | 0.893 (18)  | C14—C15 | 1.370 (2)   |
| N1—H72  | 0.902 (19)  | C14—H14 | 0.9500      |
| N11—C11 | 1.3313 (17) | C15—H15 | 0.9500      |
| N11—C15 | 1.3473 (17) | C21—C22 | 1.3926 (15) |
| N21—C21 | 1.3354 (15) | C22—C23 | 1.3866 (18) |
| N21—C25 | 1.3403 (15) | C22—H22 | 0.9500      |
| C1—C11  | 1.5030 (18) | C23—C24 | 1.378 (2)   |
| C1—H1A  | 0.9900      | C23—H23 | 0.9500      |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C1—H1B          | 0.9900       | C24—C25         | 1.3864 (19)  |
| C2—C21          | 1.5061 (16)  | C24—H24         | 0.9500       |
| C2—H2A          | 0.9900       | C25—H25         | 0.9500       |
| C2—H2B          | 0.9900       |                 |              |
| C1—N1—C2        | 111.81 (9)   | C11—C12—H12     | 121.0        |
| C1—N1—H71       | 107.7 (11)   | C13—C12—H12     | 121.0        |
| C2—N1—H71       | 108.8 (11)   | C14—C13—C12     | 119.15 (15)  |
| C1—N1—H72       | 108.4 (10)   | C14—C13—H13     | 120.4        |
| C2—N1—H72       | 109.1 (10)   | C12—C13—H13     | 120.4        |
| H71—N1—H72      | 111.0 (15)   | C15—C14—C13     | 118.86 (15)  |
| O1—N2—O3        | 121.03 (11)  | C15—C14—H14     | 120.6        |
| O1—N2—O2        | 118.66 (11)  | C13—C14—H14     | 120.6        |
| O3—N2—O2        | 120.29 (11)  | N11—C15—C14     | 123.15 (17)  |
| C11—N11—C15     | 117.70 (13)  | N11—C15—H15     | 118.4        |
| C21—N21—C25     | 116.99 (10)  | C14—C15—H15     | 118.4        |
| N1—C1—C11       | 111.53 (10)  | N21—C21—C22     | 123.50 (11)  |
| N1—C1—H1A       | 109.3        | N21—C21—C2      | 116.51 (10)  |
| C11—C1—H1A      | 109.3        | C22—C21—C2      | 119.95 (10)  |
| N1—C1—H1B       | 109.3        | C23—C22—C21     | 118.41 (12)  |
| C11—C1—H1B      | 109.3        | C23—C22—H22     | 120.8        |
| H1A—C1—H1B      | 108.0        | C21—C22—H22     | 120.8        |
| N1—C2—C21       | 111.51 (9)   | C24—C23—C22     | 118.81 (11)  |
| N1—C2—H2A       | 109.3        | C24—C23—H23     | 120.6        |
| C21—C2—H2A      | 109.3        | C22—C23—H23     | 120.6        |
| N1—C2—H2B       | 109.3        | C23—C24—C25     | 118.70 (11)  |
| C21—C2—H2B      | 109.3        | C23—C24—H24     | 120.6        |
| H2A—C2—H2B      | 108.0        | C25—C24—H24     | 120.6        |
| N11—C11—C12     | 123.13 (13)  | N21—C25—C24     | 123.58 (12)  |
| N11—C11—C1      | 116.90 (10)  | N21—C25—H25     | 118.2        |
| C12—C11—C1      | 119.95 (13)  | C24—C25—H25     | 118.2        |
| C11—C12—C13     | 117.98 (17)  |                 |              |
| C2—N1—C1—C11    | -166.97 (10) | C13—C14—C15—N11 | 0.8 (2)      |
| C1—N1—C2—C21    | -178.69 (10) | C25—N21—C21—C22 | 0.28 (17)    |
| C15—N11—C11—C12 | -0.18 (18)   | C25—N21—C21—C2  | -177.56 (10) |
| C15—N11—C11—C1  | 178.44 (11)  | N1—C2—C21—N21   | -20.60 (14)  |
| N1—C1—C11—N11   | 11.67 (16)   | N1—C2—C21—C22   | 161.48 (10)  |
| N1—C1—C11—C12   | -169.68 (11) | N21—C21—C22—C23 | -0.91 (17)   |
| N11—C11—C12—C13 | 1.7 (2)      | C2—C21—C22—C23  | 176.87 (10)  |
| C1—C11—C12—C13  | -176.91 (12) | C21—C22—C23—C24 | 0.33 (17)    |
| C11—C12—C13—C14 | -1.9 (2)     | C22—C23—C24—C25 | 0.79 (17)    |
| C12—C13—C14—C15 | 0.8 (2)      | C21—N21—C25—C24 | 0.94 (18)    |
| C11—N11—C15—C14 | -1.1 (2)     | C23—C24—C25—N21 | -1.49 (19)   |



*Hydrogen-bond geometry (Å, °)*

| <i>D</i> —H··· <i>A</i>     | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C13—H13···N2                | 0.95        | 2.61          | 3.5339 (18)           | 164                     |
| N1—H71···O3 <sup>i</sup>    | 0.893 (18)  | 2.349 (18)    | 3.0721 (15)           | 138.2 (14)              |
| N1—H72···O2 <sup>ii</sup>   | 0.902 (19)  | 1.982 (19)    | 2.8831 (15)           | 176.1 (15)              |
| N1—H72···O1 <sup>ii</sup>   | 0.902 (19)  | 2.584 (18)    | 3.2201 (16)           | 128.1 (14)              |
| N1—H72···N2 <sup>ii</sup>   | 0.902 (19)  | 2.651 (18)    | 3.4905 (15)           | 155.1 (14)              |
| C15—H15···O2 <sup>iii</sup> | 0.95        | 2.39          | 3.1858 (19)           | 141                     |
| C1—H1A···O1 <sup>iv</sup>   | 0.99        | 2.54          | 3.5132 (18)           | 167                     |
| C25—H25···O1 <sup>v</sup>   | 0.95        | 2.53          | 3.3959 (16)           | 151                     |
| C24—H24···O2 <sup>vi</sup>  | 0.95        | 2.55          | 3.4620 (16)           | 161                     |

Symmetry codes: (i)  $x, -y+1/2, z+1/2$ ; (ii)  $-x+3/2, -y, z+1/2$ ; (iii)  $x-1/2, y, -z+1/2$ ; (iv)  $x+1/2, y, -z+1/2$ ; (v)  $-x+1, -y, -z+1$ ; (vi)  $x, y, z+1$ .