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2,3-Diaminopyridinium (2*E*,4*E*)-hexa-2,4-dienoate

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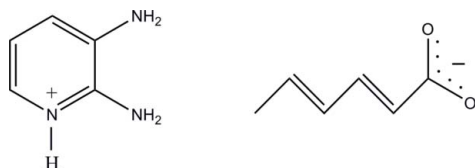
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.049; wR factor = 0.111; data-to-parameter ratio = 13.3.

In the title salt, $\text{C}_5\text{H}_8\text{N}_3^+\cdot\text{C}_6\text{H}_7\text{O}_2^-$, the pyridine N atom of the 2,3-diaminopyridine molecule is protonated. The 2,3-diaminopyridinium cation is essentially planar, with a maximum deviation of 0.068 (2) Å for one of the amino N atoms. The sorbate anion adopts an extended conformation. In the crystal structure, the protonated N atom and one of the two amino-group H atoms are hydrogen bonded to the sorbate anion through a pair of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming an $R_2^2(8)$ ring motif. The ion pairs are further connected via intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming two-dimensional networks parallel to (100).

Related literature

For details of non-covalent interactions, see: Desiraju (1995); Moulton & Zaworotko (2001); Biradha & Fujita (2002). For applications of sorbic acid, see: Martindale (1996); Richards (1972). For related structures, see: Cox (1994); Thanigaimani *et al.* (2007); Raj *et al.* (2003). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_5\text{H}_8\text{N}_3^+\cdot\text{C}_6\text{H}_7\text{O}_2^-$
 $M_r = 221.26$
 Monoclinic, $P2_1/c$
 $a = 9.0440$ (3) Å
 $b = 10.6964$ (3) Å

$c = 12.4632$ (4) Å
 $\beta = 94.947$ (2)°
 $V = 1201.18$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 296$ K

0.15 × 0.15 × 0.09 mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.987$, $T_{\max} = 0.993$

8260 measured reflections
 2736 independent reflections
 1794 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.111$
 $S = 1.02$
 2736 reflections

205 parameters
 All H-atom parameters refined
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1N1}\cdots\text{O2}$ | 1.00 (2) | 1.66 (2) | 2.6591 (19) | 174.7 (18) |
| $\text{N2}-\text{H1N2}\cdots\text{O2}$ | 0.86 (2) | 2.05 (2) | 2.9065 (18) | 173.1 (17) |
| $\text{N2}-\text{H2N2}\cdots\text{O1}$ | 0.94 (2) | 1.90 (2) | 2.8432 (19) | 176.2 (18) |
| $\text{N3}-\text{H1N3}\cdots\text{O2}$ | 0.89 (2) | 2.04 (2) | 2.930 (2) | 174 (2) |
| $\text{N3}-\text{H2N3}\cdots\text{O1}^{\text{i}}$ | 0.91 (2) | 2.11 (2) | 2.913 (2) | 146.9 (16) |
| $\text{C10}-\text{H10}\cdots\text{O1}^{\text{ii}}$ | 0.983 (19) | 2.531 (18) | 3.330 (2) | 138.2 (14) |

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5118).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Biradha, K. & Fujita, M. (2002). *Angew. Chem. Int. Ed.* **41**, 3392–3395.
 Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Cox, P. J. (1994). *Acta Cryst.* **C50**, 1620–1622.
 Desiraju, G. R. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 2311–2327.
 Martindale, W. (1996). *The Extra Pharmacopoeia*, 30th ed. London: Pharmaceutical Press.
 Moulton, B. & Zaworotko, M. (2001). *Chem. Rev.* **101**, 1629–1658.
 Raj, S. B., Sethuraman, V., Francis, S., Hemamalini, M., Muthiah, P. T., Bocelli, G., Cantoni, A., Rychlewska, U. & Warzajtis, B. (2003). *CrystEngComm*, **5**, 70–76.
 Richards, R. M. E. (1972). *Pharm. J.* **2**, 91.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Thanigaimani, K., Muthiah, P. T. & Lynch, D. E. (2007). *Acta Cryst.* **E63**, o4450–o4451.

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supplementary materials

Acta Cryst. (2010). E66, o2369 [doi:10.1107/S1600536810032617]

2,3-Diaminopyridinium (2*E*,4*E*)-hexa-2,4-dienoate

M. Hemamalini and H.-K. Fun

Comment

Non-covalent interactions, such as hydrogen bonds, $\pi\cdots\pi$ staking and the C—H $\cdots\pi$ interactions, play important roles in determining the conformation of molecules, crystal packing and molecular assembly in an organized supramolecular structure (Desiraju, 1995; Moulton & Zaworotko, 2001; Biradha & Fujita, 2002). Sorbic acid (2,4-hexadienoic acid) exhibits antibacterial and antifungal properties (Martindale, 1996) and has been used to prevent spoilage of syrup by moulds (Richards, 1972). The crystal structure of sorbic acid (Cox, 1994), 2,4-diamino-6-phenyl-1,3,5-triazine-sorbic acid (Thanigaimani *et al.*, 2007) and trimethoprim sorbate (Raj *et al.*, 2003) have been reported in the literature. Since our aim is to study some interesting hydrogen-bonding interactions, the crystal structure of the title compound is presented herein.

The asymmetric unit of the title compound, (Fig. 1), contains one 2,3-diaminopyridinium cation and one sorbate anion. The bond lengths (Allen *et al.*, 1987) and angles are normal. The 2,3-diaminopyridinium cation is planar with a maximum deviation of 0.068 (2) Å for atom N3. Protonation of atom N1 has resulted in a slight increase in the magnitude of angle C1—N1—C5 [122.86 (16)°]. The sorbic acid moiety is in the *EE* configuration. The extended conformation of the sorbic acid can be described by the torsion angles C6—C7—C8—C9 = -173.77 (16)°, C7—C8—C9—C10 = 176.15 (18)°, C8—C9—C10—C11 = -176.92 (18)° and O2—C6—C7—C8 = 171.14 (16)°. This conformation is similar to that found in the trimethoprim sorbate dihydrate (Raj *et al.*, 2003).

In the crystal structure, (Fig. 2), the protonated N1 atom and the 2-amino group N atom (N2) is hydrogen-bonded to the carboxylate oxygen atoms (O1 and O2) via a pair of N1—H1N1 \cdots O2 and N2—H2N2 \cdots O1 hydrogen bonds forming a ring motif $R_2^2(8)$ (Bernstein *et al.*, 1995). The ion pairs are further connected via N2—H1N2 \cdots O2ⁱ; N3—H1N3 \cdots O2ⁱ; N3—H2N3 \cdots O1ⁱⁱ and C10—H10 \cdots O1ⁱⁱⁱ hydrogen bonds (see Table 1 for symmetry codes) forming two-dimensional networks parallel to (100).

Experimental

A hot methanol solution (20 ml) of 2,3-diaminopyridine (59 mg, Aldrich) and sorbic acid (56 mg, Merck) were mixed and warmed over a heating magnetic stirrer hotplate for a few minutes. The resulting solution was allowed to cool slowly at room temperature and crystals of the title compound appeared after a few days.

Refinement

All H atoms were located in a difference Fourier map and refined freely [C—H = 0.94 (2)–1.04 (2) Å and N—H = 0.86 (2)–1.00 (2) Å].

Figures

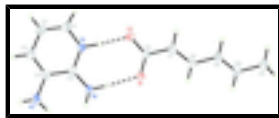


Fig. 1. The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

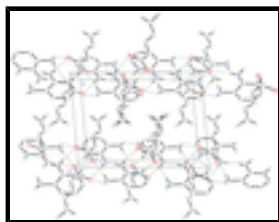
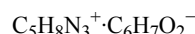


Fig. 2. The crystal packing of (I) showing hydrogen bonds as dashed lines. H atoms not involved in the intermolecular interactions have been omitted for clarity.

2,3-Diaminopyridinium (2*E*,4*E*)-hexa-2,4-dienoate

Crystal data



$M_r = 221.26$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.0440$ (3) Å

$b = 10.6964$ (3) Å

$c = 12.4632$ (4) Å

$\beta = 94.947$ (2)°

$V = 1201.18$ (6) Å³

$Z = 4$

$F(000) = 472$

$D_x = 1.224$ Mg m⁻³

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

Cell parameters from 1444 reflections

$\theta = 3.3$ – 28.9 °

$\mu = 0.09$ mm⁻¹

$T = 296$ K

Block, red

$0.15 \times 0.15 \times 0.09$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

$T_{\min} = 0.987$, $T_{\max} = 0.993$

8260 measured reflections

2736 independent reflections

1794 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.053$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.5$ °

$h = -11 \rightarrow 8$

$k = -13 \rightarrow 11$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

| | |
|-------------------|--|
| $wR(F^2) = 0.111$ | All H-atom parameters refined |
| $S = 1.02$ | $w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.1735P]$ |
| 2736 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 205 parameters | $(\Delta/\sigma)_{\max} < 0.001$ |
| 0 restraints | $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$ |
| | $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$ |

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|---------------|--------------|----------------------------------|
| O1 | -0.19667 (13) | 0.14636 (12) | 0.53361 (8) | 0.0233 (3) |
| O2 | -0.04642 (13) | 0.18916 (12) | 0.40526 (8) | 0.0241 (3) |
| C6 | -0.14500 (18) | 0.12200 (17) | 0.44625 (11) | 0.0180 (4) |
| H7 | -0.148 (2) | -0.0069 (18) | 0.3182 (13) | 0.026 (5)* |
| H8 | -0.364 (2) | -0.0391 (18) | 0.4703 (14) | 0.030 (5)* |
| H9 | -0.317 (2) | -0.1811 (18) | 0.2736 (14) | 0.028 (5)* |
| H10 | -0.545 (2) | -0.2085 (19) | 0.4233 (15) | 0.037 (6)* |
| H11A | -0.672 (3) | -0.307 (2) | 0.2669 (16) | 0.050 (6)* |
| H11B | -0.556 (2) | -0.407 (2) | 0.3260 (14) | 0.031 (5)* |
| H11C | -0.507 (2) | -0.3389 (19) | 0.2230 (15) | 0.034 (5)* |
| C7 | -0.1975 (2) | 0.01064 (18) | 0.38280 (12) | 0.0224 (4) |
| C8 | -0.3120 (2) | -0.06038 (18) | 0.40571 (12) | 0.0209 (4) |
| C9 | -0.3730 (2) | -0.16160 (17) | 0.33880 (13) | 0.0219 (4) |
| C10 | -0.4924 (2) | -0.22749 (18) | 0.35940 (14) | 0.0252 (4) |
| C11 | -0.5606 (2) | -0.3280 (2) | 0.28818 (17) | 0.0309 (5) |
| H2N3 | 0.224 (2) | 0.516 (2) | 0.8425 (15) | 0.035 (6)* |
| N1 | 0.09874 (16) | 0.36086 (15) | 0.52745 (10) | 0.0212 (4) |
| N2 | 0.00087 (17) | 0.28443 (16) | 0.67865 (12) | 0.0236 (4) |
| N3 | 0.1886 (2) | 0.44680 (19) | 0.80698 (11) | 0.0286 (4) |
| C1 | 0.1867 (2) | 0.43933 (19) | 0.47441 (14) | 0.0278 (5) |
| C2 | 0.2698 (2) | 0.5265 (2) | 0.52935 (15) | 0.0324 (5) |
| C3 | 0.2682 (2) | 0.53273 (19) | 0.64186 (14) | 0.0276 (5) |
| C4 | 0.18421 (19) | 0.45140 (18) | 0.69625 (12) | 0.0222 (4) |
| C5 | 0.09207 (18) | 0.36429 (17) | 0.63493 (12) | 0.0193 (4) |
| H1 | 0.178 (2) | 0.4288 (18) | 0.3983 (14) | 0.027 (5)* |

supplementary materials

| | | | | |
|------|------------|-----------|-------------|------------|
| H2 | 0.324 (2) | 0.586 (2) | 0.4933 (15) | 0.038 (6)* |
| H3 | 0.326 (2) | 0.599 (2) | 0.6825 (14) | 0.032 (5)* |
| H1N1 | 0.039 (2) | 0.297 (2) | 0.4839 (15) | 0.037 (6)* |
| H1N2 | -0.021 (2) | 0.291 (2) | 0.7442 (17) | 0.038 (6)* |
| H2N2 | -0.065 (2) | 0.236 (2) | 0.6330 (16) | 0.036 (6)* |
| H1N3 | 0.113 (3) | 0.407 (2) | 0.8330 (16) | 0.049 (7)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | 0.0270 (7) | 0.0241 (8) | 0.0191 (5) | -0.0026 (6) | 0.0040 (5) | -0.0010 (5) |
| O2 | 0.0274 (7) | 0.0258 (8) | 0.0193 (5) | -0.0058 (6) | 0.0023 (5) | 0.0002 (5) |
| C6 | 0.0193 (9) | 0.0172 (11) | 0.0170 (7) | 0.0011 (8) | -0.0020 (6) | 0.0033 (7) |
| C7 | 0.0238 (10) | 0.0241 (12) | 0.0190 (7) | 0.0022 (9) | 0.0005 (7) | -0.0010 (7) |
| C8 | 0.0206 (9) | 0.0205 (12) | 0.0211 (7) | 0.0050 (8) | -0.0006 (7) | 0.0010 (7) |
| C9 | 0.0237 (10) | 0.0191 (12) | 0.0225 (8) | 0.0016 (8) | 0.0002 (7) | -0.0009 (7) |
| C10 | 0.0243 (10) | 0.0218 (12) | 0.0295 (9) | 0.0013 (9) | 0.0023 (7) | 0.0001 (8) |
| C11 | 0.0272 (11) | 0.0276 (14) | 0.0373 (10) | -0.0041 (10) | -0.0007 (9) | -0.0009 (10) |
| N1 | 0.0237 (8) | 0.0201 (9) | 0.0197 (6) | -0.0022 (7) | 0.0010 (6) | -0.0009 (6) |
| N2 | 0.0252 (9) | 0.0272 (11) | 0.0187 (7) | -0.0052 (8) | 0.0034 (6) | -0.0046 (7) |
| N3 | 0.0309 (10) | 0.0300 (11) | 0.0246 (7) | -0.0088 (8) | 0.0012 (7) | -0.0094 (7) |
| C1 | 0.0320 (11) | 0.0280 (13) | 0.0235 (8) | -0.0025 (9) | 0.0033 (8) | 0.0042 (8) |
| C2 | 0.0346 (12) | 0.0280 (14) | 0.0351 (9) | -0.0075 (10) | 0.0053 (9) | 0.0068 (9) |
| C3 | 0.0293 (11) | 0.0183 (12) | 0.0346 (9) | -0.0039 (9) | -0.0001 (8) | -0.0032 (8) |
| C4 | 0.0207 (9) | 0.0206 (11) | 0.0249 (8) | 0.0029 (8) | 0.0003 (7) | -0.0029 (8) |
| C5 | 0.0185 (9) | 0.0167 (11) | 0.0223 (7) | 0.0030 (8) | 0.0006 (6) | -0.0012 (7) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|-------------|--------------|------------|
| O1—C6 | 1.2488 (18) | N1—C1 | 1.366 (2) |
| O2—C6 | 1.285 (2) | N1—H1N1 | 1.00 (2) |
| C6—C7 | 1.485 (2) | N2—C5 | 1.336 (2) |
| C7—C8 | 1.335 (2) | N2—H1N2 | 0.86 (2) |
| C7—H7 | 0.974 (17) | N2—H2N2 | 0.95 (2) |
| C8—C9 | 1.446 (2) | N3—C4 | 1.378 (2) |
| C8—H8 | 0.993 (18) | N3—H2N3 | 0.91 (2) |
| C9—C10 | 1.333 (3) | N3—H1N3 | 0.89 (3) |
| C9—H9 | 1.017 (18) | C1—C2 | 1.347 (3) |
| C10—C11 | 1.493 (3) | C1—H1 | 0.952 (16) |
| C10—H10 | 0.982 (19) | C2—C3 | 1.405 (2) |
| C11—H11A | 1.04 (2) | C2—H2 | 0.94 (2) |
| C11—H11B | 0.97 (2) | C3—C4 | 1.372 (3) |
| C11—H11C | 0.99 (2) | C3—H3 | 0.99 (2) |
| N1—C5 | 1.3466 (19) | C4—C5 | 1.427 (2) |
| O1—C6—O2 | 123.69 (16) | C1—N1—H1N1 | 117.8 (11) |
| O1—C6—C7 | 120.34 (15) | C5—N2—H1N2 | 122.4 (14) |
| O2—C6—C7 | 115.97 (14) | C5—N2—H2N2 | 119.2 (12) |
| C8—C7—C6 | 124.12 (15) | H1N2—N2—H2N2 | 115.5 (18) |

| | | | |
|---------------|--------------|--------------|--------------|
| C8—C7—H7 | 119.4 (11) | C4—N3—H2N3 | 116.1 (12) |
| C6—C7—H7 | 116.4 (11) | C4—N3—H1N3 | 115.1 (14) |
| C7—C8—C9 | 124.52 (16) | H2N3—N3—H1N3 | 117.3 (19) |
| C7—C8—H8 | 118.2 (11) | C2—C1—N1 | 120.15 (16) |
| C9—C8—H8 | 117.1 (11) | C2—C1—H1 | 125.5 (12) |
| C10—C9—C8 | 124.02 (16) | N1—C1—H1 | 114.3 (12) |
| C10—C9—H9 | 121.0 (11) | C1—C2—C3 | 119.12 (19) |
| C8—C9—H9 | 114.9 (11) | C1—C2—H2 | 121.0 (12) |
| C9—C10—C11 | 124.47 (17) | C3—C2—H2 | 119.8 (12) |
| C9—C10—H10 | 120.1 (12) | C4—C3—C2 | 121.12 (19) |
| C11—C10—H10 | 115.3 (12) | C4—C3—H3 | 119.4 (11) |
| C10—C11—H11A | 109.4 (13) | C2—C3—H3 | 119.5 (11) |
| C10—C11—H11B | 110.0 (11) | C3—C4—N3 | 123.23 (17) |
| H11A—C11—H11B | 108.2 (17) | C3—C4—C5 | 118.16 (15) |
| C10—C11—H11C | 111.7 (11) | N3—C4—C5 | 118.57 (17) |
| H11A—C11—H11C | 110.2 (16) | N2—C5—N1 | 118.07 (16) |
| H11B—C11—H11C | 107.3 (16) | N2—C5—C4 | 123.47 (15) |
| C5—N1—C1 | 122.86 (16) | N1—C5—C4 | 118.45 (16) |
| C5—N1—H1N1 | 119.3 (11) | | |
| O1—C6—C7—C8 | 8.5 (3) | C2—C3—C4—N3 | 174.29 (19) |
| O2—C6—C7—C8 | -171.14 (16) | C2—C3—C4—C5 | -3.3 (3) |
| C6—C7—C8—C9 | 173.77 (16) | C1—N1—C5—N2 | 179.13 (17) |
| C7—C8—C9—C10 | -176.15 (18) | C1—N1—C5—C4 | -1.8 (3) |
| C8—C9—C10—C11 | 176.92 (18) | C3—C4—C5—N2 | -177.03 (18) |
| C5—N1—C1—C2 | -1.2 (3) | N3—C4—C5—N2 | 5.3 (3) |
| N1—C1—C2—C3 | 2.0 (3) | C3—C4—C5—N1 | 4.0 (3) |
| C1—C2—C3—C4 | 0.3 (3) | N3—C4—C5—N1 | -173.69 (16) |

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> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1N1...O2 | 1.00 (2) | 1.66 (2) | 2.6591 (19) | 174.7 (18) |
| N2—H1N2...O2 ⁱ | 0.86 (2) | 2.05 (2) | 2.9065 (18) | 173.1 (17) |
| N2—H2N2...O1 | 0.94 (2) | 1.90 (2) | 2.8432 (19) | 176.2 (18) |
| N3—H1N3...O2 ⁱ | 0.89 (2) | 2.04 (2) | 2.930 (2) | 174 (2) |
| N3—H2N3...O1 ⁱⁱ | 0.91 (2) | 2.11 (2) | 2.913 (2) | 146.9 (16) |
| C10—H10...O1 ⁱⁱⁱ | 0.983 (19) | 2.531 (18) | 3.330 (2) | 138.2 (14) |

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

Fig. 1

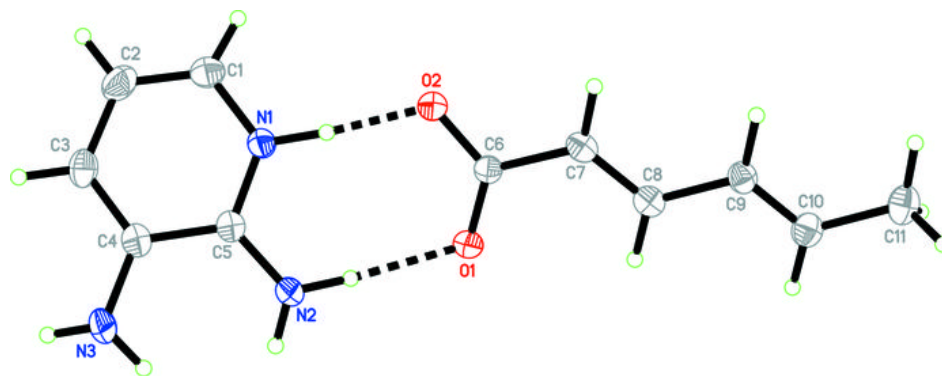


Fig. 2

