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2-Amino-4-methylpyrimidinium dihydrogen phosphate

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Key indicators: single-crystal X-ray study; T = 295 K; mean σ (C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.088; data-to-parameter ratio = 13.7.

A charge-assisted hydrogen-bonding network involving N- $H \cdots O$ and $O - H \cdots O$ hydrogen bonds stabilizes the crystal of the title salt, $C_5H_8N_3^+$ · $H_2PO_4^-$. The dihydrogen phosphate anions form one-dimensional chains along [100], via O-H···O hydrogen bonds. The 2-amino-4-methylpyrimidinium cations are linked to these chains by means of two different kinds of N−H···O hydrogen bonds. Neighbouring chains are linked via $C-H \cdots N$ and $C-H \cdots O$ hydrogen bonds forming two-dimensional slab-like networks lying parallel to $(01\overline{1})$.

Related literature

Intriguing anion clusters formed by the supramolecular assembly of dihydrogen phosphates have been investigated recently (see: Hossain et al., 2012). Methylpyrimidine derivatives are known to be synthetic precursors to many bioactive pyrimidine derivatives (see: Xue et al., 1993). Metal complexes of pyrimidines (see: Zhu et al., 2008) and their proton transfer complexes with mineral acids are reported (see: Aakeroy et al., 2003). The infinite $O-H \cdots O$ hydrogen-bond chain present in this material is a structural feature suggestive of possible proton conducting behaviour (see: Haile et al., 2001).



Experimental

Crystal data C5H8N3+

| $C_5H_8N_3^+ \cdot H_2PO_4^-$ | c = 9.9216 (4) Å |
|-------------------------------|----------------------------------|
| $M_r = 207.13$ | $\alpha = 100.562 \ (3)^{\circ}$ |
| Triclinic, $P\overline{1}$ | $\beta = 99.821 \ (3)^{\circ}$ |
| a = 6.1720 (2) Å | $\gamma = 102.279 \ (4)^{\circ}$ |
| b = 7.5616 (3) Å | V = 434.07 (3) Å ³ |

Z = 2Mo $K\alpha$ radiation $\mu = 0.31 \text{ mm}^{-1}$

Data collection

Oxford Xcalibur Eos (Nova) CCD detector diffractometer Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006) $T_{\min} = 0.928, \ T_{\max} = 0.947$

Refinement

| $R[F^2 > 2\sigma(F^2)] = 0.031$ |
|---------------------------------|
| $wR(F^2) = 0.088$ |
| S = 1.08 |
| 1717 reflections |
| 125 parameters |

 $0.25 \times 0.20 \times 0.18 \; \mathrm{mm}$

T = 295 K

organic compounds

9718 measured reflections 1717 independent reflections 1546 reflections with $I > 2\sigma(I)$ $R_{\rm int} = 0.027$

H atoms treated by a mixture of independent and constrained refinement $\Delta \rho_{\rm max} = 0.21 \text{ e } \text{\AA}^{-3}$ $\Delta \rho_{\rm min} = -0.34 \text{ e } \text{\AA}^{-3}$

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

| $D - H \cdots A$ | D-H | $H \cdot \cdot \cdot A$ | $D \cdots A$ | $D - \mathbf{H} \cdots A$ |
|--------------------------------------|---------|-------------------------|--------------|---------------------------|
| O1-H1···O4 ⁱ | 0.82 | 1.80 | 2.6100 (18) | 168 |
| $N1 - H1N \cdot \cdot \cdot O2^{ii}$ | 0.86 | 2.14 | 3.000 (2) | 177 |
| O2−H2···O4 ⁱⁱⁱ | 0.82 | 1.80 | 2.5843 (17) | 161 |
| $N1 - H2N \cdot \cdot \cdot O3^{iv}$ | 0.86 | 2.01 | 2.845 (2) | 163 |
| $N3-H3N\cdots O3^{ii}$ | 0.90(2) | 1.73 (2) | 2.6276 (19) | 173 (2) |
| $C4-H4\cdots N2^{v}$ | 0.93 | 2.55 | 3.463 (2) | 166 |
| $C5-H5B\cdots O1^{vi}$ | 0.96 | 2.58 | 3.531 (3) | 171 |

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

Data collection: CrysAlis CCD (Oxford Diffraction, 2006); cell refinement: CrysAlis PRO (Oxford Diffraction, 2006); data reduction: CrysAlis RED (Oxford Diffraction, 2006); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and WinGX (Farrugia, 2012); molecular graphics: Mercury (Macrae et al., 2008); software used to prepare material for publication: PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2227).

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2-Amino-4-methylpyrimidinium dihydrogen phosphate

Sajesh P. Thomas and Jyothi Sunkari

S1. Comment

The title compound, a multicomponent crystal, AMHP, crystallizes in triclinic P-1, with a protonated 2-amino-4-methylpyrimidine molecule and a dihydrogenphosphate moiety in the asymmetric unit (Fig. 1). The dihydrogenphosphate residue forms a chain *via* O—H···O hydrogen bonds. 2-Amino-4-methylpyrimidinium cations are linked to these chains by means of two different kinds of N—H···O hydrogen bonds. The crystal packing is stabilized by N—H···O and O— H···O hydrogen bonds and the resulting supramolecular assembly is shown in Figure 2. The infinite hydrogen bond chains present in this structure are of special interest due to the anticipated proton conductivity of the material (see: Haile *et al.*2001).

S2. Experimental

The title compound was prepared by treating 2-amino 4-methyl pyramidine with phosphoric acid (H3PO4) in aqueous solution (Scheme 1) in 1:1 molar ratio. The crystals were harvested from the solution after 10 days and suitable crystal for single-crystal X-ray diffraction study were chosen using a polarizing microscope.

S3. Refinement

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(*s*) used to solve structure: *SHELXL97* (Sheldrick, 2008); program(*s*) used to refine structure: *SHELXL97* (Sheldrick, 2008), *WinGX*(Farrugia, 2012); molecular graphics: Mercury 2.3 (Macrae *et al.* 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).



Figure 1

ORTEP view of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.



Figure 2

A view of supramolecular chain showing the hydrogen bonding between dihydrogen phosphate residues and the interlinked 2-amino-4-methylpyrimidine molecules.

2-Amino-4-methylpyrimidinium dihydrogen phosphate

Crystal data

 $C_{5}H_{8}N_{3}^{+} \cdot H_{2}PO_{4}^{-}$ $M_{r} = 207.13$ Triclinic, *P*1 Hall symbol: -P 1 a = 6.1720 (2) Å b = 7.5616 (3) Å c = 9.9216 (4) Å a = 100.562 (3)° $\beta = 99.821$ (3)° $\gamma = 102.279$ (4)° V = 434.07 (3) Å³ Z = 2

Data collection

Oxford Xcalibur Eos (Nova) CCD detector diffractometer Radiation source: Enhance (Mo) X-ray Source Graphite monochromator ω scans Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006) $T_{\min} = 0.928, T_{\max} = 0.947$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier Least-squares matrix: full map $R[F^2 > 2\sigma(F^2)] = 0.031$ Hydrogen site location: inferred from $wR(F^2) = 0.088$ neighbouring sites S = 1.08H atoms treated by a mixture of independent 1717 reflections and constrained refinement $w = 1/[\sigma^2(F_0^2) + (0.0449P)^2 + 0.1805P]$ 125 parameters where $P = (F_0^2 + 2F_c^2)/3$ 0 restraints Primary atom site location: structure-invariant $(\Delta/\sigma)_{\rm max} < 0.001$ $\Delta \rho_{\rm max} = 0.21 \text{ e } \text{\AA}^{-3}$ direct methods $\Delta \rho_{\rm min} = -0.34 \text{ e} \text{ Å}^{-3}$

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating *-R*-factor-obs *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 $(Å^2)$

| | x | у | Ζ | $U_{\rm iso}$ */ $U_{\rm eq}$ |
|----|------------|------------|--------------|-------------------------------|
| N1 | 0.1136 (3) | 0.3869 (2) | 1.11795 (16) | 0.0405 (5) |

F(000) = 216Least Squares Treatment of 25 SET4 setting angles. $D_x = 1.585 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ Å}$ Cell parameters from 326 reflections $\theta = 2.8-26.0^{\circ}$ $\mu = 0.31 \text{ mm}^{-1}$ T = 295 KBlock, colourless $0.25 \times 0.20 \times 0.18 \text{ mm}$

9718 measured reflections 1717 independent reflections 1546 reflections with $I > 2\sigma(I)$ $R_{int} = 0.027$ $\theta_{max} = 26.0^{\circ}, \ \theta_{min} = 2.8^{\circ}$ $h = -7 \rightarrow 7$ $k = -9 \rightarrow 9$ $l = -12 \rightarrow 12$

| N2 | -0.0464 (2) | 0.1740 (2) | 0.90738 (15) | 0.0332 (4) |
|-----|--------------|--------------|--------------|------------|
| N3 | 0.3480 (2) | 0.2530 (2) | 0.99937 (15) | 0.0326 (4) |
| C1 | 0.1379 (3) | 0.2712 (2) | 1.00803 (17) | 0.0293 (5) |
| C2 | -0.0146 (3) | 0.0613 (2) | 0.79698 (18) | 0.0341 (5) |
| C3 | 0.2007 (3) | 0.0371 (3) | 0.7852 (2) | 0.0407 (6) |
| C4 | 0.3796 (3) | 0.1362 (3) | 0.88869 (19) | 0.0386 (6) |
| C5 | -0.2202 (4) | -0.0381 (3) | 0.6843 (2) | 0.0507 (7) |
| P1 | 0.28422 (7) | 0.48498 (6) | 0.64883 (4) | 0.0283 (1) |
| 01 | 0.0685 (2) | 0.31898 (17) | 0.59152 (14) | 0.0418 (4) |
| O2 | 0.4901 (2) | 0.39299 (18) | 0.64980 (12) | 0.0365 (4) |
| O3 | 0.2949 (2) | 0.5709 (2) | 0.79898 (13) | 0.0439 (4) |
| O4 | 0.29115 (19) | 0.61556 (16) | 0.55043 (13) | 0.0336 (4) |
| H1N | 0.23060 | 0.44920 | 1.18240 | 0.0490* |
| H2N | -0.01930 | 0.40020 | 1.12510 | 0.0490* |
| Н3 | 0.21960 | -0.04470 | 0.70830 | 0.0490* |
| H3N | 0.464 (4) | 0.319 (3) | 1.071 (2) | 0.048 (6)* |
| H4 | 0.52500 | 0.12390 | 0.88380 | 0.0460* |
| H5A | -0.31890 | -0.12620 | 0.71950 | 0.0760* |
| H5B | -0.17500 | -0.10210 | 0.60520 | 0.0760* |
| H5C | -0.29890 | 0.05020 | 0.65580 | 0.0760* |
| H1 | -0.03370 | 0.35420 | 0.54850 | 0.0630* |
| H2 | 0.53240 | 0.39280 | 0.57580 | 0.0550* |
| | | | | |

Atomic displacement parameters $(Å^2)$

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|-------------|-------------|-------------|
| N1 | 0.0258 (7) | 0.0530 (9) | 0.0348 (8) | 0.0121 (7) | 0.0011 (6) | -0.0069 (7) |
| N2 | 0.0252 (7) | 0.0384 (8) | 0.0319 (7) | 0.0049 (6) | 0.0049 (6) | 0.0030 (6) |
| N3 | 0.0238 (7) | 0.0423 (8) | 0.0309 (7) | 0.0087 (6) | 0.0047 (6) | 0.0069 (6) |
| C1 | 0.0241 (8) | 0.0338 (8) | 0.0303 (8) | 0.0081 (6) | 0.0052 (6) | 0.0081 (7) |
| C2 | 0.0327 (9) | 0.0334 (8) | 0.0331 (9) | 0.0033 (7) | 0.0079 (7) | 0.0048 (7) |
| C3 | 0.0410 (10) | 0.0435 (10) | 0.0373 (10) | 0.0132 (8) | 0.0140 (8) | 0.0003 (8) |
| C4 | 0.0315 (9) | 0.0494 (10) | 0.0403 (10) | 0.0173 (8) | 0.0134 (8) | 0.0103 (8) |
| C5 | 0.0393 (11) | 0.0546 (12) | 0.0424 (11) | -0.0023 (9) | 0.0047 (8) | -0.0077 (9) |
| P1 | 0.0201 (2) | 0.0388 (3) | 0.0242 (2) | 0.0076 (2) | 0.0030(2) | 0.0046 (2) |
| 01 | 0.0294 (7) | 0.0421 (7) | 0.0492 (8) | 0.0029 (5) | -0.0041 (6) | 0.0177 (6) |
| O2 | 0.0300 (6) | 0.0563 (8) | 0.0293 (6) | 0.0201 (6) | 0.0081 (5) | 0.0126 (6) |
| 03 | 0.0298 (7) | 0.0705 (9) | 0.0280 (7) | 0.0165 (6) | 0.0057 (5) | -0.0015 (6) |
| 04 | 0.0238 (6) | 0.0396 (6) | 0.0379 (7) | 0.0078 (5) | 0.0066 (5) | 0.0106 (5) |

Geometric parameters (Å, °)

| P1-03 | 1.4964 (13) | N1—H2N | 0.8600 | |
|-------|-------------|--------|-----------|--|
| P101 | 1.5623 (14) | N1—H1N | 0.8600 | |
| P1—O2 | 1.5725 (14) | N3—H3N | 0.90 (2) | |
| P1 | 1.5098 (13) | C2—C5 | 1.492 (3) | |
| 01—H1 | 0.8200 | C2—C3 | 1.401 (3) | |
| O2—H2 | 0.8200 | C3—C4 | 1.347 (3) | |
| | | | | |

| $\begin{array}{c c c c c c c c c c c c c c c c c c c $ | | | | |
|--|-----------------------|-------------|--------------------------|-------------|
| $N2-C2$ $1.329 (2)$ $C4-H4$ 0.9300 $N2-C1$ $1.348 (2)$ $C5-H5C$ 0.9600 $N3-C1$ $1.348 (2)$ $C5-H5B$ 0.9600 $N3-C4$ $1.347 (2)$ $C5-H5B$ 0.9600 $P1 \cdots H2N^{ii}$ 3.1000 $C2\cdots O1$ $3.099 (2)$ $P1 \cdots H2N^{ii}$ 3.000 $C3\cdots O1$ $3.257 (2)$ $P1 \cdots H2^{in}$ 2.8900 $C5\cdots O1$ $3.277 (3)$ $P1 \cdots H2^{in}$ 2.8800 $C5\cdots O1$ $3.277 (3)$ $O1 \cdots C3$ $3.257 (2)$ $C5\cdots H4^{inii}$ 2.8900 $O1 \cdots C4^{in}$ $2.6100 (18)$ $H1\cdots O4^{ini}$ 1.8000 $O1^{C4i}$ $2.6100 (18)$ $H1\cdots H1^{ini}$ 2.5200 $O2^{C4i}$ $3.395 (2)$ $H1\cdots H1^{ini}$ 2.5200 $O2^{O4i^{in}}$ $2.5843 (17)$ $H1\cdots P1^{ini}$ 2.600 $O3^{N1i^{in}}$ $2.6100 (18)$ $H2^{D4i^{in}}$ 1.8000 $O1^{C2i}$ 2.5800 $H2^{P1i^{in}}$ 2.4500 $O4^{D1i^{in}}$ $2.6100 (18)$ $H2^{P1i^{in}}$ 2.8000 | N1—C1 | 1.319 (2) | С3—Н3 | 0.9300 |
| N2-C1 $1.349(2)$ C5-H5C 0.9600 N3-C1 $1.348(2)$ C5-H5A 0.9600 N3-C4 $1.347(2)$ C5-H5B 0.9600 PI-H3N ⁱ $2.89(2)$ C^{C1ik} $3.481(2)$ PI-H2N ⁱⁱ 3.1000 C^{C1ik} $3.481(2)$ PI-H1N ⁱⁱ 2.8900 $C3 \cdots O1$ $3.257(2)$ PI-H2 ⁱⁱⁱ 2.8800 $C5 \cdots O1$ $3.277(2)$ O1-C5 $3.277(3)$ $H1 \cdots P1i^{iii}$ 2.8900 O1-C4 ⁱⁱⁱ $2.6100(18)$ $H1 \cdots O4^{dii}$ 1.8000 O1-C2 $3.099(2)$ $H1 \cdots H1^{iii}$ 2.5500 O2-··O4 ⁱⁱⁱ $2.6400(18)$ $H1 \cdots O4^{dii}$ 1.8000 O2-··O4 ⁱⁱⁱ $2.843(17)$ $H1 \cdots H2^{iii}$ 2.5200 O4-··O2 ⁱⁱⁱⁱ $2.845(2)$ $H1 N^{04^{iii}$ 1.8000 O3-··N1 ⁱⁱ $2.845(2)$ $H1 N^{04^{iiii}$ 1.8000 O4-··O2 ⁱⁱⁱⁱ 2.5800 $H2^{04^{iiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiii$ | N2—C2 | 1.329 (2) | C4—H4 | 0.9300 |
| N3-C1 1.348 (2) C5-H5A 0.9600 N3-C4 1.347 (2) C3-H5B 0.9600 P1-H2N ⁱⁱ 3.1000 C2···C1 ^{is} 3.481 (2) P1-H2N ⁱⁱ 3.000 C2···C1 ^{is} 3.481 (2) P1-H1N ⁱⁱ 2.8900 C3···C1 3.257 (3) P1-H2 ^{2s} 2.8800 C5···O1 3.277 (3) O1C5 3.277 (3) H1····P1 ⁱⁱⁱ 2.8900 O1C4 ⁱⁱⁱ 2.6100 (18) H1····O4 ⁱⁱⁱ 1.8000 O204 ⁱⁱⁱ 2.6100 (18) H1····P1 ⁱⁱⁱ 2.500 O204 ⁱⁱⁱ 2.6276 (19) H2····O4 ⁱⁱⁱ 1.8000 O4O1 ⁱⁱⁱ 2.6100 (18) H2····H1 ⁱⁱⁱ 2.500 O4O2 ⁱⁱⁱ 2.5800 H2···P1 ⁱⁱⁱ 2.800 O3H3 ⁱⁱⁱ 2.1400 H2···P1 ⁱⁱⁱ 2.8000 O3H3 ⁱⁱⁱⁱ < | N2—C1 | 1.349 (2) | C5—H5C | 0.9600 |
| N3-C4 1.347 (2) C5-H5B 0.9600 PI-H3N ⁴ 2.89 (2) C2-C1 ¹⁸ 3.481 (2) PI-H2N ¹⁶ 3.1000 C2-O1 3.099 (2) PI-H1 ¹⁸ 2.8900 C3-O1 3.257 (2) PI-H1 ¹⁸ 3.0600 C4-O2 3.395 (2) PI-H2 ¹⁷ 2.8800 C5-O1 3.277 (3) O1-C3 3.257 (2) C5-H4 ²⁶ 2.8900 O1-C4 3.099 (2) HI-H1 ¹⁶ 2.8900 O1-C4 3.099 (2) HI-H1 ¹⁶ 2.5200 O2-C4 3.395 (2) HIN-H3N 2.2400 O2-C4 3.395 (2) HIN-O2 ¹¹ 3.0600 O3-N1 ¹⁴ 2.845 (2) HIN-O2 ¹¹ 3.0600 O3-N1 ¹⁴ 2.845 (2) HIN-O2 ¹¹ 3.0600 O3-N3 ¹¹ 2.6100 (18) H2-H1N ¹⁶ 2.5200 O4-O2 ¹⁴ 2.845 (2) HIN-O2 ¹¹ 3.0600 O3-H3N ¹⁵ 2.676 (19) H2-O4 ⁴⁹ 1.8000 O4-O1 ¹⁴ 2.6100 (18) H2-H1N ¹⁶ | N3—C1 | 1.348 (2) | C5—H5A | 0.9600 |
| P1-H3N ¹ 2.89 (2) C2-C1 ^{is} 3.481 (2) P1-H2N ⁱⁱ 3.1000 C2-O1 3.099 (2) P1-H1N ⁱⁱ 3.8000 C3-O1 3.257 (2) P1-H1N ⁱⁱ 3.0600 C4-O2 3.395 (2) P1-H1N ⁱⁱ 3.0600 C4-O2 3.395 (2) P1-H2 ⁱⁱ 2.8800 C5-O1 3.277 (3) O1-O4 ⁱⁱ 2.6100 (18) H1-O4 ⁱⁱⁱ 2.9800 O1-O4 ⁱⁱ 2.6100 (18) H1-O4 ⁱⁱⁱ 2.8900 O1-O4 ⁱⁱⁱ 2.6100 (18) H1-O4 ⁱⁱⁱ 2.500 O2-O4 ⁱⁱⁱ 3.000 (2) H1N-H3N 2.2400 O2-O4 ⁱⁱⁱ 2.843 (17) H1N-O2 ⁱⁱ 3.0600 O3-N1 ⁱⁱ 2.6276 (19) H2-O4 ⁱⁱⁱ 1.8000 O4-O2 ⁱⁱⁱ 2.6410 (18) H2-H1 ⁱⁱⁱ 2.5200 O4-O2 ⁱⁱⁱ 2.6400 H2-O4 ⁱⁱⁱ 2.6400 O4-O2 ⁱⁱⁱ 2.6400 (18) H2-H1 ⁱⁱⁱ 2.8400 O3-H1N ⁱⁱ 2.7300 H2-H2 ⁱⁱⁱ 2.8400 O3-H1N ⁱⁱⁱ 2.9100 H3N-P1 ⁱⁱⁱ 2.8400 O3-H3N ⁱⁱⁱ 1.73 (2) | N3—C4 | 1.347 (2) | С5—Н5В | 0.9600 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | | | | 01,000 |
| P1····H2N ⁱⁱ 3.1000 C2···O1 3.099 (2) P1····H1 ⁱⁱⁱ 2.8900 C3···O1 3.257 (2) P1····H2 ^{iv} 2.8800 C5···O1 3.277 (3) O1···C3 3.257 (2) C5···H4 ⁱⁱⁱⁱ 2.9800 O1···C4 3.257 (2) C5···H4 ⁱⁱⁱⁱ 2.9800 O1···C5 3.277 (3) H1···P1 ⁱⁱⁱ 2.8900 O1···C4 3.059 (2) H1····H1 ⁱⁱⁱⁱ 2.8900 O1···C2 3.099 (2) H1····H1 ⁱⁱⁱⁱ 2.500 O2···O4 ⁱⁱⁱ 3.000 (2) H1····H1 ⁱⁱⁱⁱ 2.500 O2···O4 ^{ivi} 2.5843 (17) H1····O4 ⁱⁱⁱ 3.0600 O3···N1 ⁱⁱ 2.845 (2) H1····O2 ⁱⁱ 2.1400 O4···O1 ⁱⁱⁱ 2.6100 (18) H2····H1 ⁱⁱⁱ 3.000 O4···O2 ^{iv} 2.5843 (17) H2····P1 ⁱⁱⁱ 3.1000 O2···H1N ⁱⁱ 2.6100 (18) H2····P1 ⁱⁱⁱ 3.1000 O2···H1N ⁱⁱ 2.6100 (18) H2····P1 ⁱⁱⁱ 3.1000 O2···H1N ⁱⁱ 2.1400 H2····P1 ⁱⁱⁱ 3.1000 O2···H1N ⁱⁱⁱ 1.73 (2) H2····P1 ⁱⁱⁱ 3. | P1…H3N ⁱ | 2.89 (2) | C2····C1 ^{ix} | 3.481 (2) |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | P1…H2N ⁱⁱ | 3.1000 | C2…O1 | 3.099 (2) |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | P1…H1 ⁱⁱⁱ | 2.8900 | C3…O1 | 3.257 (2) |
| P1···H2 ^{ν} 2.8800 C5···01 3.277 (3) O1···C3 3.257 (2) C5···H4 ^{ν} ⁱⁱⁱ 2.9800 O1···C4 3.277 (3) H1···P1 ⁱⁱ 2.8900 O1···C5 3.277 (3) H1···O4 ⁱⁱⁱ 1.8000 O1···C2 3.099 (2) H1···H1 ⁱⁱⁱ 2.5500 O2···Ni ⁱ 3.000 (2) H1N···H2 ⁱ 2.5200 O2···O4 ^{iv} 2.5843 (17) H1N···P1 ⁱ 3.0600 O2···O4 ^{iv} 2.5843 (17) H1N···P1 ⁱⁱ 3.0600 O3···N1 ⁱⁱ 2.845 (2) H1N···D2 ⁱ 2.1400 O4···O1 ^{ivi} 2.6100 (18) H2···H1N ^{iv} 2.5200 O4···O2 ^{iv} 2.5843 (17) H2···P1 ^{iv} 2.8800 O2···H1N ⁱ 2.6100 (18) H2···H1N ^{iv} 2.500 O4···O2 ^{iv} 2.5800 H2···P1 ^{iv} 2.8800 O3···H2N ⁱⁱ 1.73 (2) H2N···P1 ⁱⁱ 3.1000 O3···H2N ⁱⁱ 2.9100 H3····H1N 2.2400 O4···H2 ^{iv} 1.8000 H3····H1 ⁱⁱ 2.8000 O3···H2N ⁱⁱ 2.9100 H3····H1N 2.4500 | P1…H1N ⁱ | 3.0600 | C4…O2 | 3.395 (2) |
| O1C3 3.257 (2) C5H4* ⁱⁱⁱ 2.980 O1C5 3.277 (3) $H1-P1^{iii}$ 2.8900 O1C4 ⁱⁱⁱ 2.6100 (18) $H1P1^{iii}$ 2.8900 O1C2 3.099 (2) $H1H1^{iii}$ 2.8900 O2C4 3.395 (2) $H1N-H2^{ii}$ 2.5200 O2C4 3.395 (2) $H1N-H2^{ii}$ 3.0600 O3N1 ⁱⁱ 2.843 (17) $H1N-P1^{ii}$ 3.0600 O3N3 ⁱⁱ 2.6276 (19) $H204^{ii}$ 1.8000 O4O1 ⁱⁱⁱ 2.6100 (18) $H2H1^{ii}$ 2.5200 O4O1 ⁱⁱⁱ 2.6100 (18) $H2H1^{ii}$ 2.5200 O4O2 ⁱⁱⁱ 2.6300 $H2P1^{iii}$ 2.8800 O2H1N ⁱⁱ 2.1400 $H2N-P1^{ii}$ 2.800 O3H2N ⁱⁱ 2.1000 $H3O4^{ii}$ 2.9100 O3H2N ⁱⁱ 2.9100 $H3NP1^{ii}$ 2.892 O4H1 ⁱⁱⁱ 1.8000 $H3NP1^{ii}$ 2.8900 O3H2N ⁱⁱⁱ 2.8000 | P1…H2 ^{iv} | 2.8800 | C5…O1 | 3.277 (3) |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 01···C3 | 3.257 (2) | C5····H4 ^{viii} | 2.9800 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 01…C5 | 3.277 (3) | $H1\cdots P1^{iii}$ | 2.8900 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 01…O4 ⁱⁱⁱ | 2.6100 (18) | H1····O4 ⁱⁱⁱ | 1.8000 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 01···C2 | 3.099 (2) | $H1\cdots H1^{iii}$ | 2.5500 |
| $02 \cdots C4$ 3.395 (2) $H1N \cdots H3N$ 2.2400 $02 \cdots O4^w$ 2.5843 (17) $H1N \cdots P1^i$ 3.0600 $03 \cdots N1^{ii}$ 2.845 (2) $H1N \cdots O2^i$ 2.1400 $03 \cdots N3^i$ 2.6276 (19) $H2 \cdots O4^w$ 1.8000 $04 \cdots O1^{ii}$ 2.6100 (18) $H2 \cdots H1^w$ 2.5200 $04 \cdots O2^w$ 2.5843 (17) $H2 \cdots H2^w$ 2.4500 $01 \cdots H5B^v$ 2.5843 (17) $H2 \cdots H2^w$ 2.4500 $01 \cdots H5B^v$ 2.5843 (17) $H2 \cdots P1^{iv}$ 2.8800 $02 \cdots H1N^i$ 2.1400 $H2N \cdots P1^{iv}$ 2.8800 $03 \cdots H3N^i$ 1.73 (2) $H2N \cdots O3^{ii}$ 2.0100 $03 \cdots H2N^{ii}$ 2.0100 $H3 \cdots O4^s$ 2.9100 $04 \cdots H2^{iv}$ 1.8000 $H3 \cdots O4^s$ 2.9100 $04 \cdots H2^{iv}$ 1.8000 $H3 \cdots H1N$ 2.2400 $04 \cdots H3^{ii}$ 2.9100 $H3 \cdots O3^i$ 1.73 (2) $N1 \cdots O2^i$ 3.000 (2) $H4 \cdots C5^{ii}$ 2.9800 $N1 \cdots O2^i$ 3.000 (2) $H4 \cdots C5^{ii}$ 2.9800 $N3 \cdots O3^i$ 2.6276 (19) $H5A \cdots O4^{iii}$ 2.8000 $N2 \cdots O3^i$ 2.6500 $H5B \cdots H3$ 2.3900 $O1 \cdots H2^{iii}$ 2.5500 $H5B \cdots O1^v$ 2.5800 $O3 \cdots P1 \cdots O4$ 109.55 (7) $N2 - C2 - C3$ 121.54 (15) $O1 - P1 - O4$ 109.55 (7) $N2 - C2 - C5$ 116.73 (7) $O1 - P1 - O4$ 109.55 (7) $N2 - C2 - C3$ 122.14 (16) $O1 - P1 - O4$ 109.55 (7) $N2 - C2 - C3$ </td <td>O2…N1ⁱ</td> <td>3.000 (2)</td> <td>$H1N\cdots H2^{i}$</td> <td>2.5200</td> | O2…N1 ⁱ | 3.000 (2) | $H1N\cdots H2^{i}$ | 2.5200 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O2…C4 | 3.395 (2) | H1N···H3N | 2.2400 |
| $O3 \cdots N1^{ii}$ 2.845 (2) $H1N \cdots O2^{i}$ 2.1400 $O3 \cdots N3^{i}$ 2.6276 (19) $H2 \cdots O4^{iv}$ 1.8000 $O4 \cdots O1^{ii}$ 2.6100 (18) $H2 \cdots H1N^{i}$ 2.5200 $O4 \cdots O2^{iv}$ 2.5843 (17) $H2 \cdots H2^{iv}$ 2.4500 $O1 \cdots H5B^{v}$ 2.5800 $H2 \cdots P1^{iv}$ 2.8800 $O2 \cdots H1N^{i}$ 2.1400 $H2 \cdots P1^{iv}$ 2.8800 $O2 \cdots H1N^{ii}$ 2.1400 $H2 \cdots P1^{iv}$ 2.8800 $O3 \cdots H2N^{ii}$ 2.0100 $H3 \cdots O4^{s}$ 2.9100 $O3 \cdots H2N^{ii}$ 2.0100 $H3 \cdots O4^{s}$ 2.9100 $O4 \cdots H2^{iv}$ 1.8000 $H3 \cdots H5B$ 2.3900 $O4 \cdots H3^{ivi}$ 2.9100 $H3N \cdots P1^{ii}$ 2.89 (2) $O4 \cdots H3^{ivi}$ 2.9100 $H3N \cdots O3^{i}$ 1.73 (2) $O4 \cdots H3^{ivi}$ 2.8000 $H3N \cdots O3^{i}$ 1.73 (2) $O4 \cdots H3^{ivi}$ 2.8000 $H3N \cdots O3^{i}$ 1.73 (2) $O4 \cdots H3^{ivi}$ 2.8000 $H3N \cdots O3^{i}$ 1.73 (2) $N \cdots O3^{ii}$ 2.845 (2) $H4 \cdots N2^{vi}$ 2.8900 $N \cdots O3^{ii}$ 2.845 (2) $H4 \cdots C5^{si}$ 2.9800 $N \cdots O3^{ii}$ 2.6276 (19) $H5A \cdots O4^{sii}$ 2.9000 $N \cdots O3^{ii}$ 2.6276 (19) $H5A \cdots O4^{sii}$ 2.3900 $O3 - P1 - O4$ 115.79 (8) $N2 - C1 - N3$ 121.54 (15) $O1 - P1 - O3$ 109.84 (8) $N2 - C2 - C5$ 116.73 (17) $O2 - P1 - O4$ 110.13 (7) $C2 - C3 - C4$ 117.84 (18) <t< td=""><td>O2···O4^{iv}</td><td>2.5843 (17)</td><td>$H1N\cdots P1^{i}$</td><td>3.0600</td></t<> | O2···O4 ^{iv} | 2.5843 (17) | $H1N\cdots P1^{i}$ | 3.0600 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 03…N1 ⁱⁱ | 2.845 (2) | H1N····O2 ⁱ | 2.1400 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O3…N3 ⁱ | 2.6276 (19) | H2…O4 ^{iv} | 1.8000 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O4…O1 ⁱⁱⁱ | 2.6100 (18) | H2…H1N ⁱ | 2.5200 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O4…O2 ^{iv} | 2.5843 (17) | H2…H2 ^{iv} | 2.4500 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O1···H5B ^v | 2.5800 | H2…P1 ^{iv} | 2.8800 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O2…H1N ⁱ | 2.1400 | H2N…P1 ⁱⁱ | 3.1000 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O3…H3N ⁱ | 1.73 (2) | H2N…O3 ⁱⁱ | 2.0100 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O3…H2N ⁱⁱ | 2.0100 | H3····O4 ^x | 2.9100 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O4…H2 ^{iv} | 1.8000 | H3…H5B | 2.3900 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O4…H1 ⁱⁱⁱ | 1.8000 | H3N…H1N | 2.2400 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O4···H3 ^{vi} | 2.9100 | H3N…P1 ⁱ | 2.89 (2) |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | O4…H5A ^{vii} | 2.8000 | H3N····O3 ⁱ | 1.73 (2) |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | N1…O2 ⁱ | 3.000 (2) | H4…N2 ^{xi} | 2.5500 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | N1…O3 ⁱⁱ | 2.845 (2) | H4····C5 ^{xi} | 2.9800 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | N3…O3 ⁱ | 2.6276 (19) | H5A····O4 ^{xii} | 2.8000 |
| $C1 \cdots C2^{ix}$ 115.79 (8) $N2-C1-N3$ 121.54 (15) $O3-P1-O4$ 115.79 (8) $N2-C2-C3$ 122.14 (16) $O1-P1-O3$ 109.84 (8) $N2-C2-C3$ 122.14 (16) $O1-P1-O4$ 109.55 (7) $N2-C2-C5$ 116.73 (17) $O1-P1-O2$ 104.78 (7) $C3-C2-C5$ 121.13 (17) $O2-P1-O4$ 110.13 (7) $C2-C3-C4$ 117.84 (18) $O2-P1-O3$ 106.13 (7) $N3-C4-C3$ 120.03 (18) $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $C1-N2-C2$ 117.90 (15) $C3-C4-H4$ 120.00 $C1-N3-C4$ 120.52 (15) $N3-C4-H4$ 120.00 $H1N-N1-H2N$ 120.00 $C2-C5-H5B$ 109.00 | N2…H4 ^{viii} | 2.5500 | H5B…H3 | 2,3900 |
| O3-P1-O4 $115.79 (8)$ $N2-C1-N3$ $121.54 (15)$ $O1-P1-O3$ $109.84 (8)$ $N2-C2-C3$ $122.14 (16)$ $O1-P1-O4$ $109.55 (7)$ $N2-C2-C5$ $116.73 (17)$ $O1-P1-O2$ $104.78 (7)$ $C3-C2-C5$ $121.13 (17)$ $O2-P1-O4$ $110.13 (7)$ $C2-C3-C4$ $117.84 (18)$ $O2-P1-O3$ $106.13 (7)$ $N3-C4-C3$ $120.03 (18)$ $P1-O1-H1$ 109.00 $C2-C3-H3$ 121.00 $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $C1-N2-C2$ $117.90 (15)$ $C3-C4-H4$ 120.00 $C1-N3-C4$ $120.52 (15)$ $N3-C4-H4$ 120.00 $H1N-N1-H2N$ 120.00 $C2-C5-H5B$ 109.00 | C1···C2 ^{ix} | 3481(2) | H5B···O1v | 2 5800 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 01 02 | 5.101 (2) | | 2.2000 |
| O1-P1-O3 109.84 (8) $N2-C2-C3$ 122.14 (16) $O1-P1-O4$ 109.55 (7) $N2-C2-C5$ 116.73 (17) $O1-P1-O2$ 104.78 (7) $C3-C2-C5$ 121.13 (17) $O2-P1-O4$ 110.13 (7) $C2-C3-C4$ 117.84 (18) $O2-P1-O3$ 106.13 (7) $N3-C4-C3$ 120.03 (18) $P1-O1-H1$ 109.00 $C2-C3-H3$ 121.00 $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $C1-N2-C2$ 117.90 (15) $C3-C4-H4$ 120.00 $C1-N3-C4$ 120.52 (15) $N3-C4-H4$ 120.00 $H1N-N1-H2N$ 120.00 $C2-C5-H5B$ 109.00 $C1-N1-H1N$ 120.00 $C2-C5-H5C$ 109.00 | O3—P1—O4 | 115.79 (8) | N2—C1—N3 | 121.54 (15) |
| 01-P1-O4 $109.55 (7)$ $N2-C2-C5$ $116.73 (17)$ $01-P1-O2$ $104.78 (7)$ $C3-C2-C5$ $121.13 (17)$ $02-P1-O4$ $110.13 (7)$ $C2-C3-C4$ $117.84 (18)$ $02-P1-O3$ $106.13 (7)$ $N3-C4-C3$ $120.03 (18)$ $P1-O1-H1$ 109.00 $C2-C3-H3$ 121.00 $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $C1-N2-C2$ $117.90 (15)$ $C3-C4-H4$ 120.00 $C1-N3-C4$ $120.52 (15)$ $N3-C4-H4$ 120.00 $H1N-N1-H2N$ 120.00 $C2-C5-H5B$ 109.00 | 01—P1—03 | 109.84 (8) | N2—C2—C3 | 122.14 (16) |
| O1-P1-O2 104.78 (7) $C3-C2-C5$ 121.13 (17) $O2-P1-O4$ 110.13 (7) $C2-C3-C4$ 117.84 (18) $O2-P1-O3$ 106.13 (7) $N3-C4-C3$ 120.03 (18) $P1-O1-H1$ 109.00 $C2-C3-H3$ 121.00 $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $C1-N2-C2$ 117.90 (15) $C3-C4-H4$ 120.00 $C1-N3-C4$ 120.52 (15) $N3-C4-H4$ 120.00 $H1N-N1-H2N$ 120.00 $C2-C5-H5B$ 109.00 | 01—P1—04 | 109.55 (7) | N2—C2—C5 | 116.73 (17) |
| O2-P1-O4 110.13 (7) $C2-C3-C4$ 117.84 (18) $O2-P1-O3$ 106.13 (7) $N3-C4-C3$ 120.03 (18) $P1-O1-H1$ 109.00 $C2-C3-H3$ 121.00 $P1-O2-H2$ 109.00 $C4-C3-H3$ 121.00 $C1-N2-C2$ 117.90 (15) $C3-C4-H4$ 120.00 $C1-N3-C4$ 120.52 (15) $N3-C4-H4$ 120.00 $H1N-N1-H2N$ 120.00 $C2-C5-H5B$ 109.00 $C1-N1-H1N$ 120.00 $C2-C5-H5C$ 109.00 | 01—P1—02 | 104.78 (7) | C3—C2—C5 | 121.13 (17) |
| O2—P1—O3 106.13 (7) N3—C4—C3 120.03 (18) P1—O1—H1 109.00 C2—C3—H3 121.00 P1—O2—H2 109.00 C4—C3—H3 121.00 C1—N2—C2 117.90 (15) C3—C4—H4 120.00 C1—N3—C4 120.52 (15) N3—C4—H4 120.00 H1N—N1—H2N 120.00 C2—C5—H5B 109.00 C1—N1—H1N 120.00 C2—C5—H5C 109.00 | O2—P1—O4 | 110.13 (7) | C2—C3—C4 | 117.84 (18) |
| P1—01—H1 109.00 C2—C3—H3 121.00 P1—02—H2 109.00 C4—C3—H3 121.00 C1—N2—C2 117.90 (15) C3—C4—H4 120.00 C1—N3—C4 120.52 (15) N3—C4—H4 120.00 H1N—N1—H2N 120.00 C2—C5—H5B 109.00 C1—N1—H1N 120.00 C2—C5—H5C 109.00 | 02—P1—03 | 106.13 (7) | N3—C4—C3 | 120.03 (18) |
| P1—O2—H2 109.00 C4—C3—H3 121.00 C1—N2—C2 117.90 (15) C3—C4—H4 120.00 C1—N3—C4 120.52 (15) N3—C4—H4 120.00 H1N—N1—H2N 120.00 C2—C5—H5B 109.00 C1—N1—H1N 120.00 C2—C5—H5C 109.00 | P1-01-H1 | 109.00 | С2—С3—Н3 | 121.00 |
| C1—N2—C2 117.90 (15) C3—C4—H4 120.00 C1—N3—C4 120.52 (15) N3—C4—H4 120.00 H1N—N1—H2N 120.00 C2—C5—H5B 109.00 C1—N1—H1N 120.00 C2—C5—H5C 109.00 | P1-02-H2 | 109.00 | C4—C3—H3 | 121.00 |
| C1—N3—C4 120.52 (15) N3—C4—H4 120.00 H1N—N1—H2N 120.00 C2—C5—H5B 109.00 C1—N1—H1N 120.00 C2—C5—H5C 109.00 | C1-N2-C2 | 117.90 (15) | C3—C4—H4 | 120.00 |
| H1N-N1-H2N 120.00 $C2-C5-H5B$ 109.00 $C1-N1-H1N$ 120.00 $C2-C5-H5C$ 109.00 | C1 - N3 - C4 | 120.52 (15) | N3—C4—H4 | 120.00 |
| C1—N1—H1N 120.00 C2—C5—H5C 109.00 | H1N—N1—H2N | 120.00 | C2—C5—H5B | 109.00 |
| | C1—N1—H1N | 120.00 | C2—C5—H5C | 109.00 |

supporting information

| C1—N1—H2N | 120.00 | С2—С5—Н5А | 109.00 |
|-------------|--------------|-------------|-------------|
| C1—N3—H3N | 117.5 (15) | H5A—C5—H5C | 109.00 |
| C4—N3—H3N | 121.9 (15) | H5B—C5—H5C | 109.00 |
| N1—C1—N3 | 118.80 (16) | H5A—C5—H5B | 110.00 |
| N1—C1—N2 | 119.66 (17) | | |
| C2—N2—C1—N1 | 178.81 (15) | C4—N3—C1—N2 | 0.1 (2) |
| C2—N2—C1—N3 | -1.3 (2) | C1—N3—C4—C3 | 0.3 (3) |
| C1—N2—C2—C3 | 2.1 (2) | N2-C2-C3-C4 | -1.7 (3) |
| C1—N2—C2—C5 | -177.38 (16) | C5—C2—C3—C4 | 177.75 (19) |
| C4—N3—C1—N1 | -179.98 (18) | C2—C3—C4—N3 | 0.4 (3) |
| | | | |

Symmetry codes: (i) -*x*+1, -*y*+1, -*z*+2; (ii) -*x*, -*y*+1, -*z*+2; (iii) -*x*, -*y*+1, -*z*+1; (iv) -*x*+1, -*y*+1, -*z*+1; (v) -*x*, -*y*, -*z*+1; (vi) *x*, *y*+1, *z*; (vii) *x*+1, *y*+1, *z*; (vii) *x*-1, *y*, *z*; (ix) -*x*, -*y*, -*z*+2; (x) *x*, *y*-1, *z*; (xi) *x*+1, *y*, *z*; (xii) *x*-1, *y*-1, *z*.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | Н…А | D····A | D—H···A |
|------------------------------------|----------|----------|-------------|---------|
| 01—H1…O4 ⁱⁱⁱ | 0.82 | 1.80 | 2.6100 (18) | 168 |
| N1— $H1N$ ···O2 ⁱ | 0.86 | 2.14 | 3.000 (2) | 177 |
| O2—H2···O4 ^{iv} | 0.82 | 1.80 | 2.5843 (17) | 161 |
| N1—H2 <i>N</i> ···O3 ⁱⁱ | 0.86 | 2.01 | 2.845 (2) | 163 |
| N3—H3 <i>N</i> ···O3 ⁱ | 0.90 (2) | 1.73 (2) | 2.6276 (19) | 173 (2) |
| C4—H4····N2 ^{xi} | 0.93 | 2.55 | 3.463 (2) | 166 |
| $C5-H5B\cdotsO1^{v}$ | 0.96 | 2.58 | 3.531 (3) | 171 |

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