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Crystal structure of sodium dihydrogen arsenate

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Single crystals of the title compound, Na(H₂AsO₄), were obtained by partial neutralization of arsenic acid with sodium hydroxide in aqueous solution. The crystal structure of Na(H₂AsO₄) is isotypic with the phosphate analogue and the asymmetric unit consists of two sodium cations and two tetrahedral H₂AsO₄⁻ anions. Each of the sodium cations is surrounded by six O atoms of five H₂AsO₄⁻ groups, defining distorted octahedral coordination spheres. In the extended structure, the sodium cations and dihydrogen arsenate anions are arranged in the form of layers lying parallel to (010). Strong hydrogen bonds [range of O··O distances 2.500 (3)–2.643 (3) Å] between adjacent H₂AsO₄⁻ anions are observed within and perpendicular to the layers. The isotypic structure of Na(H₂PO₄) is comparatively discussed.

1. Chemical context

Arsenic acid is triprotic and thus can form various salts, depending on the degree of deprotonation (H2AsO4-, $HAsO_4^{2-}$, AsO_4^{3-}), the condensation grade of the anion (mono-, di-, tri-, polyarsenate, etc) and the amount of water incorporated in the crystal. With respect to sodium arsenates, numerous crystal structures have been determined so far, including arsenic in tetrahedral and/or in octahedral coordination by oxygen atoms. Arsenate structures with arsenic exclusively in tetrahedral coordination resemble those of the related phosphates and in some cases show isotypism with them (marked by an asterisk): $Na_{3,25}(AsO_4)(OH)_{0,25}(H_2O)_{1,2}*$ (Tillmanns & Baur, 1971), Na₄(AsO₄)OH (zur Love et al., 2015), Na₂(HAsO₄)(H₂O)₇* (Baur & Khan, 1970; Ferraris et al., 1971), Na(H₂AsO₄)(H₂O) (Ferraris et al., 1974), $Na_3(H_2As_3O_{10})$ (Driss & Jouini, 1990), $Na_4As_2O_7$ (Leung & Calvo, 1973), Na(AsO₃) (Liebau, 1956) and Na₅(AsO₅) (Haas & Jansen, 2001). Arsenate structures with arsenic in (complete or partial) octahedral coordination include $Na(H_2As_3O_9)$ (Driss, Jouini, Durif et al., 1988), Na₃(H₅As₄O₁₄) (Driss & Jouini, 1989), Na(HAs₂O₆) (Dung & Tahar, 1978), Na₂As₄O₁₁ (Driss, Jouini & Omezzine, 1988) and Na7As11O31 (Guesmi et al., 2006). A detailed discussion of the structural principles and crystal chemical characteristics of arsenates with arsenic in octahedral coordination was given some time ago by Schwendtner & Kolitsch (2007).

Besides the Na:As 1:1 phase Na(H_2AsO_4)(H_2O) another 1:1 phase, Na(H_2AsO_4), has been reported but without an additional water molecule (Fehér & Morgenstern, 1937). To our surprise, a detailed structural investigation of this salt has not yet been reported. Therefore, we started crystal growth experiments and determined its structure and report here on the results.



Figure 1

The crystal structure of Na(H₂AsO₄) in a projection along [100]. All atoms are depicted with displacement ellipsoids at the 97% probability level. Dihydrogen arsenate tetrahedra are given in polyhedral representation, Na⁺ cations as single ellipsoids without bonds to surrounding O atoms. H. $\cdot \cdot O$ hydrogen bonds are illustrated with green lines.

2. Structural commentary

The crystal structure of $Na(H_2AsO_4)$ is isotypic with that of Na(H₂PO₄) (Catti & Ferraris, 1974). The asymmetric unit of Na(H₂AsO₄) comprises two Na⁺ cations and two tetrahedral $AsO_2(OH)_2^-$ groups. The Na1⁺ cation shows a narrow Na-Obond-length distribution in the range 2.337 (2) to 2.498 (2) Å with a distorted octahedron as the corresponding coordination polyhedron. The bond-valence sum (Brown, 2002) for the Na1⁺ cation amounts to 1.15 valence units. The surrounding of the Na2⁺ cation is much more distorted, with a bond-length range from 2.338 (2) to 2.769 (3) Å under consideration of a sixfold coordination (bond-valence sum 0.92 valence units). There is an additional remote oxygen atom at a distance of 3.000 (3) Å from Na2⁺. Its contribution of 0.04 valence units to the bond-valence sum might be considered as too low for a significant interaction, and therefore the first coordination sphere of Na2⁺ is discussed as that of a considerably distorted octahedron. The two dihydrogen arsenate groups show the usual differences (Weil, 2000, 2016) between As-O and As-(OH) bonds, with two significantly shorter As-O bonds [mean 1.659 (8) Å] and two longer As-(OH) bonds [1.723 (12) Å].

In the crystal structure of $Na(H_2AsO_4)$ the $AsO_2(OH)_2$ tetrahedra are arranged in layers lying parallel to (010) with the Na^+ cations approximately on the same level (Fig. 1).

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

D_H4 D_H H4 D4 D_H	[4
	1 1
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	5) 4) 5) 4)

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

Strong, asymmetric hydrogen bonds $[O \cdots O]$ distances between 2.500 (3) and 2.643 (3) Å, Table 1] between each of the OH groups of the two dihydrogen arsenate tetrahedra and O atoms of adjacent tetrahedra significantly contribute to the crystal packing. These hydrogen bonds are both within a layer and towards adjacent layers (Fig. 1).

The differences between the isotypic arsenate and phosphate structures can mainly be seen in the X-O bond lengths of the anions (X = As, mean of 1.69 Å; X = P, mean of 1.55 Å), with $\Delta_{\max}(X-O)$ of 0.15 Å between arsenate and phosphate tetrahedra. The difference with respect to the Na-O distances in the two structures is less pronounced, with $\Delta_{\text{max}}(\text{Na-O}) = 0.10$ Å. Relevant bond lengths of the isotypic crystal structures of Na(H₂AsO₄) and Na(H₂PO₄) (Catti & Ferraris, 1974) are compiled in Table 2. A more quantitative comparison of the two crystal structures with the help of the COMPSTRU routine (de la Flor et al., 2016) revealed the following values: The degree of lattice distortion (S), i.e. the spontaneous strain (sum of the squared eigenvalues of the strain tensor divided by 3), is 0.0159; the maximum distance $(d_{\text{max.}})$, *i.e.* the maximal displacement between the atomic positions of paired atoms, is 0.1920 Å for atom pair O1; the arithmetic mean (d_{av}) of the distances of all atom pairs is

Table 2

Comparison of bond lengths (Å) in the title compound and the isotypic phosphate analogue (Catti & Ferraris, 1974).

Bond	Na(H ₂ AsO ₄)	Na(H ₂ PO ₄)
Na1-O3 ⁱ	2.337 (2)	2.355 (1)
Na1-O5 ⁱⁱ	2.376 (2)	2.406 (1)
Na1-O3 ⁱⁱⁱ	2.382 (2)	2.371 (1)
Na1-O7	2.456 (2)	2.501 (1)
Na1-O2 ^{iv}	2.459 (2)	2.436 (1)
Na1-O6 ^{iv}	2.498 (2)	2.564 (1)
Na2-O8	2.338 (2)	2.334 (1)
Na2-O3 ^{iv}	2.371 (2)	2.369 (1)
Na2-O1 ⁱⁱ	2.419 (2)	2.433 (1)
Na2-O6 ⁱ	2.586 (2)	2.601 (1)
Na2-O2 ^{iv}	2.703 (3)	2.600(1)
Na2-O4 ⁱ	2.769 (3)	2.730 (1)
Na2-O7 ^v	3.000 (3)	2.930 (1)
As/P1-O3	1.6484 (19)	1.499 (1)
As/P1 - O1	1.657 (2)	1.508 (1)
As/P1 - O4	1.730 (2)	1.592 (1)
As/P1 - O2	1.736 (2)	1.597 (1)
As/P2 - O6	1.663 (2)	1.523 (1)
As/P2 - O8	1.668 (2)	1.519(1)
As/P2 - O7	1.711 (2)	1.562 (1)
As/P2-O5	1.713 (2)	1.572 (1)

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

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0.1108 Å; the measure of similarity (Δ) (Bergerhoff *et al.*, 1999) is a function of the differences in atomic positions (weighted by the multiplicities of the sites) and the ratios of the corresponding lattice parameters of the structures and amounts to 0.049.

3. Synthesis and crystallization

The title compound was prepared following a procedure by Fehér & Morgenstern (1937). An arsenic acid solution (*ca* $65\%_{wt}$) was partly neutralized with diluted NaOH solution using methyl red as indicator. The resulting solution was concentrated by heating. Standing of the solution overnight on a warm plate (*ca* 313 K) afforded colourless crystals with a lath-like form and maximal edge lengths of 0.5 mm.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Starting coordinates and labelling of atoms were taken from the isotypic Na(H₂PO₄) structure (Catti & Ferraris, 1974). Hydrogen atoms were clearly discernible from difference maps and were refined with distance restraints d(O-H) = 0.85 (1) Å.

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Table 3	
Experimental	details.

Crystal data	
Chemical formula	Na(H ₂ AsO ₄)
M _r	163.93
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	7.0528 (14), 13.798 (3), 7.4792 (15)
β(°)	93.02 (3)
$V(Å^3)$	726.8 (3)
Z	8
Radiation type	Μο Κα
$\mu \text{ (mm}^{-1})$	9.32
Crystal size (mm)	$0.12\times0.08\times0.01$
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Krause et al., 2015)
T_{\min}, T_{\max}	0.534, 0.746
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	11092, 2651, 1890
R _{int}	0.052
$(\sin \theta / \lambda)_{\rm max} ({\rm \AA}^{-1})$	0.758
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.030, 0.059, 1.04
No. of reflections	2651
No. of parameters	125
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta \rho_{\rm max}, \Delta \rho_{\rm min} \ ({ m e} \ { m \AA}^{-3})$	0.87, -0.86

Computer programs: APEX3 and SAINT (Bruker, 2016), SHELXL2016 (Sheldrick, 2015), ATOMS (Dowty, 2006) and publCIF (Westrip, 2010).

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Crystal structure of sodium dihydrogen arsenate

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Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: coordinates taken from isotypic structure; program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *ATOMS* (Dowty, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Sodium dihydrogen arsenate

Crystal data

Na(H₂AsO₄) $M_r = 163.93$ Monoclinic, $P2_1/c$ a = 7.0528 (14) Å b = 13.798 (3) Å c = 7.4792 (15) Å $\beta = 93.02$ (3)° V = 726.8 (3) Å³ Z = 8

Data collection

Bruker APEXII CCD diffractometer ω and φ scans Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015) $T_{\min} = 0.534$, $T_{\max} = 0.746$ 11092 measured reflections

Refinement

Refinement on F^2 Least-squares matrix: full $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.059$ S = 1.042651 reflections 125 parameters 4 restraints F(000) = 624 $D_x = 2.996 \text{ Mg m}^{-3}$ Mo Ka radiation, $\lambda = 0.71073 \text{ Å}$ Cell parameters from 2433 reflections $\theta = 3.1-31.8^{\circ}$ $\mu = 9.32 \text{ mm}^{-1}$ T = 100 KLath, colourless $0.12 \times 0.08 \times 0.01 \text{ mm}$

2651 independent reflections 1890 reflections with $I > 2\sigma(I)$ $R_{int} = 0.052$ $\theta_{max} = 32.6^\circ, \ \theta_{min} = 2.9^\circ$ $h = -10 \rightarrow 10$ $k = -20 \rightarrow 20$ $l = -11 \rightarrow 11$

Primary atom site location: isomorphous structure methods Hydrogen site location: difference Fourier map H atoms treated by a mixture of independent and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.020P)^2 + 0.0156P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{max} = 0.001$ $\Delta\rho_{max} = 0.87$ e Å⁻³ $\Delta\rho_{min} = -0.86$ 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.

	x	у	Ζ	$U_{ m iso}$ */ $U_{ m eq}$
As1	0.32467 (4)	0.36827 (2)	0.84669 (4)	0.00611 (7)
As2	0.82170 (4)	0.37010 (2)	0.50756 (4)	0.00595 (7)
Na1	0.85834 (16)	0.40338 (8)	-0.00598 (15)	0.0091 (2)
Na2	0.34819 (17)	0.39825 (9)	0.26282 (16)	0.0134 (3)
01	0.2417 (3)	0.26833 (14)	0.7478 (3)	0.0108 (4)
O2	0.5315 (3)	0.34069 (15)	0.9726 (3)	0.0099 (4)
O3	0.1910 (3)	0.42983 (14)	0.9809 (3)	0.0078 (4)
O4	0.4015 (3)	0.44833 (15)	0.6878 (3)	0.0124 (4)
05	0.9202 (3)	0.26110 (14)	0.5709 (3)	0.0113 (4)
O6	0.8540 (3)	0.45079 (14)	0.6715 (3)	0.0098 (4)
07	0.9303 (3)	0.40531 (15)	0.3188 (3)	0.0093 (4)
08	0.5931 (3)	0.34459 (14)	0.4613 (3)	0.0082 (4)
H1	0.545 (7)	0.2797 (14)	0.970 (6)	0.055 (15)*
H2	0.462 (5)	0.415 (3)	0.619 (5)	0.038 (13)*
H3	1.026 (4)	0.270 (3)	0.627 (6)	0.071 (18)*
H4	0.995 (5)	0.457 (2)	0.332 (6)	0.045 (13)*

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\hat{A}^2)

Atomic displacement parameters $(Å^2)$

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.00551 (13)	0.00631 (14)	0.00645 (13)	0.00067 (11)	-0.00029 (10)	-0.00017 (11)
As2	0.00609 (13)	0.00599 (14)	0.00564 (13)	-0.00119 (11)	-0.00081 (10)	0.00025 (11)
Na1	0.0087 (6)	0.0086 (5)	0.0099 (6)	0.0004 (4)	-0.0002 (4)	0.0003 (4)
Na2	0.0161 (6)	0.0132 (6)	0.0106 (6)	0.0027 (5)	-0.0038 (5)	-0.0001 (5)
O1	0.0101 (10)	0.0078 (10)	0.0141 (11)	-0.0005 (8)	-0.0031 (8)	-0.0037 (8)
02	0.0077 (10)	0.0068 (9)	0.0147 (11)	0.0005 (8)	-0.0044 (8)	0.0006 (8)
O3	0.0062 (10)	0.0094 (9)	0.0078 (10)	0.0016 (7)	0.0007 (7)	-0.0011 (7)
04	0.0150 (11)	0.0112 (10)	0.0114 (11)	0.0040 (9)	0.0059 (9)	0.0033 (8)
O5	0.0105 (10)	0.0064 (10)	0.0163 (11)	-0.0006 (8)	-0.0054 (9)	0.0012 (8)
06	0.0101 (10)	0.0110 (10)	0.0083 (10)	-0.0033 (8)	0.0012 (8)	-0.0034 (8)
O7	0.0122 (10)	0.0091 (9)	0.0070 (9)	-0.0034 (9)	0.0038 (7)	-0.0008 (8)
08	0.0058 (9)	0.0078 (9)	0.0108 (10)	-0.0011 (7)	-0.0013 (7)	0.0011 (8)

Geometric parameters (Å, °)

As1—O3	1.6484 (19)	Na1—O6 ^{iv}	2.498 (2)
As1—01	1.657 (2)	Na2—O8	2.338 (2)
As1—O4	1.730 (2)	Na2—O3 ^{iv}	2.371 (2)
As1—O2	1.736 (2)	Na2—O1 ⁱⁱ	2.419 (2)

supporting information

As2—O6	1.663 (2)	Na2—O6 ⁱ	2.586 (2)
As2—08	1.668 (2)	Na2—O2 ^{iv}	2.703 (3)
As2—07	1.711 (2)	Na2—O4 ⁱ	2.769 (3)
As2—O5	1.713 (2)	Na2—O7 ^v	3.000 (3)
Na1—O3 ⁱ	2.337 (2)	O2—H1	0.847 (19)
Na1—O5 ⁱⁱ	2.376 (2)	O4—H2	0.828 (18)
Na1—O3 ⁱⁱⁱ	2.382 (2)	О5—Н3	0.847 (19)
Na1—O7	2.456 (2)	O7—H4	0.849 (19)
Na1—O2 ^{iv}	2.459 (2)		
O3—As1—O1	120.05 (10)	O1 ⁱⁱ —Na2—O4 ⁱ	157.97 (8)
O3—As1—O4	107.40 (10)	$O6^{i}$ —Na2—O4 i	73.32 (7)
O1—As1—O4	109.92 (11)	$O2^{iv}$ —Na2—O4 ⁱ	90.18 (7)
O3—As1—O2	105.89 (10)	08—Na2—O7 ^v	128.32 (8)
O1—As1—O2	109.06 (10)	O3 ^{iv} —Na2—O7 ^v	72.63 (7)
O4—As1—O2	103.17 (10)	O1 ⁱⁱ —Na2—O7 ^v	74.48 (7)
O6—As2—O8	112.80 (10)	O6 ⁱ —Na2—O7 ^v	52.53 (6)
O6—As2—O7	111.60 (10)	$O2^{iv}$ —Na2—O7 ^v	129.53 (8)
O8—As2—O7	111.00 (10)	O4 ⁱ —Na2—O7 ^v	125.50 (7)
O6—As2—O5	110.24 (10)	As1—O1—Na2 ^{vi}	131.96 (12)
O8—As2—O5	104.21 (10)	As1—O2—Na1 ^{vii}	135.72 (11)
O7—As2—O5	106.56 (10)	As1—O2—Na2 ^{vii}	86.99 (8)
O3 ⁱ —Na1—O5 ⁱⁱ	161.36 (9)	Na1 ^{vii} —O2—Na2 ^{vii}	109.31 (9)
O3 ⁱ —Na1—O3 ⁱⁱⁱ	90.20 (7)	As1—O2—H1	107 (3)
O5 ⁱⁱ —Na1—O3 ⁱⁱⁱ	89.30 (8)	Na1 ^{vii} —O2—H1	104 (3)
O3 ⁱ —Na1—O7	86.19 (8)	Na2 ^{vii} —O2—H1	111 (3)
O5 ⁱⁱ —Na1—O7	75.23 (8)	As1—O3—Na1 ⁱ	130.46 (11)
O3 ⁱⁱⁱ —Na1—O7	83.49 (8)	As1—O3—Na2 ^{vii}	101.02 (9)
O3 ⁱ —Na1—O2 ^{iv}	102.04 (8)	Na1 ⁱ —O3—Na2 ^{vii}	100.03 (8)
O5 ⁱⁱ —Na1—O2 ^{iv}	80.75 (8)	As1—O3—Na1 ^{viii}	122.85 (11)
O3 ⁱⁱⁱ —Na1—O2 ^{iv}	166.71 (9)	Na1 ⁱ —O3—Na1 ^{viii}	89.80 (7)
O7—Na1—O2 ^{iv}	102.26 (9)	Na2 ^{vii} —O3—Na1 ^{viii}	110.53 (9)
O3 ⁱ —Na1—O6 ^{iv}	79.94 (8)	As1—O4—Na2 ⁱ	128.10 (12)
O5 ⁱⁱ —Na1—O6 ^{iv}	118.47 (8)	As1—O4—H2	105 (3)
O3 ⁱⁱⁱ —Na1—O6 ^{iv}	83.21 (8)	Na2 ⁱ —O4—H2	99 (3)
$O7$ —Na1— $O6^{iv}$	160.71 (8)	As2—O5—Na1 ^{vi}	134.83 (12)
O2 ^{iv} —Na1—O6 ^{iv}	93.76 (8)	As2—O5—H3	111 (3)
O8—Na2—O3 ^{iv}	156.69 (9)	Na1 ^{vi} —O5—H3	113 (3)
O8—Na2—O1 ⁱⁱ	86.89 (8)	As2—O6—Na1 ^{vii}	122.07 (11)
O3 ^{iv} —Na2—O1 ⁱⁱ	90.22 (8)	As2—O6—Na2 ⁱ	128.58 (11)
O8—Na2—O6 ⁱ	122.07 (8)	Na1 ^{vii} —O6—Na2 ⁱ	90.36 (7)
O3 ^{iv} —Na2—O6 ⁱ	77.53 (7)	As2—O7—Na1	137.29 (12)
O1 ⁱⁱ —Na2—O6 ⁱ	126.96 (8)	As2—O7—Na2 ^{ix}	126.26 (11)
O8—Na2—O2 ^{iv}	92.76 (8)	Na1—O7—Na2 ^{ix}	90.85 (7)
O3 ^{iv} —Na2—O2 ^{iv}	63.95 (7)	As2—07—H4	114 (3)
O1 ⁱⁱ —Na2—O2 ^{iv}	81.03 (8)	Na1—O7—H4	102 (3)
O6 ⁱ —Na2—O2 ^{iv}	132.91 (8)	Na2 ^{ix} —O7—H4	61 (3)

supporting information

08—Na2—O4 ⁱ	73.31 (7)	As2—O8—Na2	137.60 (11)
$O3^{iv}$ —Na2—O4 ⁱ	104.07 (8)		

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

Hydrogen-bond geometry (Å, °)

D—H···A	<i>D</i> —Н	H···A	$D \cdots A$	D—H···A
02—H1…O8 ^{vi}	0.85 (2)	1.75 (2)	2.595 (3)	175 (5)
O4—H2…O8	0.83 (2)	1.82 (2)	2.643 (3)	178 (4)
O5—H3···O1 ^{ix}	0.85 (2)	1.73 (2)	2.566 (3)	171 (5)
O7—H4···O6 ^x	0.85 (2)	1.66 (2)	2.500 (3)	169 (4)

Symmetry codes: (vi) *x*, -*y*+1/2, *z*+1/2; (ix) *x*+1, *y*, *z*; (x) -*x*+2, -*y*+1, -*z*+1.