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# Disodium dihydrogen pyridine-2,3,5,6-tetracarboxylate trihydrate

**Fang-Hong Hu and Jian-Li Lin***

State Key Laboratory Base of Novel Functional Materials and Preparation Science, Center of Applied Solid State Chemistry Research, Ningbo University, Ningbo, Zhejiang 315211, People's Republic of China
Correspondence e-mail: linjianli@nbu.edu.cn

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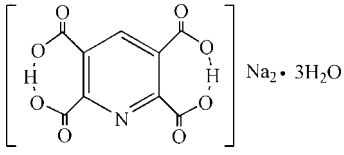
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.043; $wR$ factor = 0.139; data-to-parameter ratio = 10.8.

In the title compound, $2Na^+\cdot C_9H_3NO_8^{2-}\cdot 3H_2O$, the asymmetric unit consists of two $Na^+$ cations, one dihydrogen pyridine-2,3,5,6-tetracarboxylate dianion ($H_2pdtc^{2-}$) and three water molecules coordinated to the $Na^+$ cations. The configuration of the anion is stabilized by intramolecular O—H···O hydrogen bonding between vicinal carboxylate/carboxy groups. The $Na^+$ cations are bridged by the $H_2pdtc^{2-}$ dianions, generating layers extending infinitely in sheets parallel to (001), and further pillared by the water molecule linkers to build up a three-dimensional framework.

## Related literature

For related compounds involving the pyridine-2,3,5,6-tetracarboxylic acid ligand, see: Zhang *et al.* (2010); Yang *et al.* (2008); Sun, Zhou & An (2009); Sun, Zhou & Yan (2009).




## Experimental

### Crystal data

$2Na^+\cdot C_9H_3NO_8^{2-}\cdot 3H_2O$
$M_r$ = 353.15
Triclinic, $P\overline{1}$
$a$ = 5.5844 (11) Å
$b$ = 6.6770 (13) Å
$c$ = 18.631 (4) Å
$\alpha$ = 81.34 (3)°
$\beta$ = 86.77 (3)°
$\gamma$ = 68.36 (3)°
$V$ = 638.4 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.23 mm$^{-1}$
$T$ = 293 K
0.1 × 0.1 × 0.1 mm

### Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.97, $T_{max}$ = 0.98
5075 measured reflections
2247 independent reflections
1780 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.139
$S$ = 1.14
2247 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O2 | 0.86 | 1.55 | 2.410 | 175 |
| O7—H7···O6 | 0.86 | 1.55 | 2.402 | 175 |
| O9—H9$A$···O1$^{i}$ | 0.85 | 2.05 | 2.900 | 175 |
| O9—H9$B$···O1$^{ii}$ | 0.86 | 2.11 | 2.945 | 161 |
| O10—H10$A$···O2$^{iii}$ | 0.88 | 2.21 | 3.024 | 153 |
| O10—H10$B$···O8$^{iv}$ | 0.88 | 1.86 | 2.738 | 173 |
| O11—H11$A$···O4$^{v}$ | 0.89 | 2.05 | 2.917 | 167 |
| O11—H11$B$···O4$^{iv}$ | 0.88 | 2.08 | 2.949 | 170 |

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2024).

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# supplementary materials

*Acta Cryst.* (2010). E**66**, m524 [ **doi:10.1107/S1600536810012870** ]

# Disodium dihydrogen pyridine-2,3,5,6-tetracarboxylate trihydrate

**F.-H. Hu and J.-L. Lin**

## Comment

A great many metal-polycarboxylate compounds with benzene-1,2,4,5-tetracarboxylic acid ($H_4$btec) as ligands have been designed and characterized (Zhang *et al.*, 2010). Analogous in structure to $H_4$btec, only four complexes [Ni($H_2$pdtc)($H_2O$)$_2$].3$H_2O$ (Yang *et al.*, 2008), [$Zn_4$(pdtc)$_2$(phen)$_2$($H_2O$)$_2$].20$H_2O$ (Yang *et al.*, 2008), [Cd($H_2$pdtc)($H_2O$)$_3$].3$H_2O$ (Sun, Zhou & Yan, 2009) and [$Ni_2$(pdtc)($H_2O$)$_3$(2,2'-bpy)].4$H_2O$ (Sun, Zhou & An, 2009) about pyridine-2,3,5,6-tetracarboxylic acid ($H_4$pdtc) have been reported. The four coordination polymers are constructed by linking transition metal centres through the ligands. In this context, we represent a disodium salt of dihydrogen pyridine-2,3,5,6-tetracarboxylate dianion [$Na_2$($H_2$pdtc)($H_2O$)$_3$].

The asymmetric unit of the title compound consisits of two crystallographically independent Na cations, one dihydrogen pyridine-2,3,5,6-tetracarboxylate dianion ($H_2pdtc^{2-}$) and three water molecules (Fig. 1). The ligand is deprotonated at 2,5-positioned carboxylate groups. The values of the dihedral angle between the planes of the carboxylic groups and the planar pyridine ring are 3.7 (4)°, 2.0 (5)°, 10.5 (4)°, 2.4 (5)°, respectively. Both Na1 and Na2 ions are seven-coordinated with one N atom, two carboxylate O atoms and four water molecules at Na1 and five carboxylate O atoms and two water molecules at Na2, building highly distorted pentagonal–bipyramidal environment. The average value of the Na—O(water) length [2.494 (2) Å] is closer to the average Na—O (carboxylate) distance [2.498 (2) Å], and both are slightly smaller than the Na—N value [2.654 (2) Å]. The sodium cations are bridged by the ($H_2pdtc^{2-}$) to generate layers extending infinitely in sheets parallel to (001) (Fig. 2), and further pillared by the water molecule linkers (Fig. 3) to build up 3D framework, which is found to be stabilized by hydrogen bonds from water molecules to carboxylate O atoms (Table 1).

## Experimental

0.0766 g (0.3 mmol) pyridine-2,3,5,6-tetracarboxylic acid and 0.024 g (0.6 mmol) NaOH were successively added to 10.0 ml $H_2O$ and stirred at room temperature for 2 h, and the resulting colorless solution (pH = 3.48) was then transferred to a 50 ml beaker for slow evaporation at room temperature for several months, affording colorless block crystals (yield: 0.05 g).

## Refinement

H atoms bonded to C atoms were placed in geometrically calculated positions and were refined using a riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C). H atoms attached to O atoms were found in a difference Fourier synthesis and were refined using a riding model, with $U_{iso}$(H) values set at 1.2*U*eq(O).

**Figures**

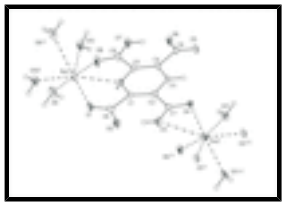

Fig. 1. ORTEP view of the title compound, Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) 2 - *x*, 1 - *y*, 1 - *z*; (ii) 3 - *x*, 1 - *y*, 1 - *z*; (iii) 2 - *x*, 1 - *y*, -*z*; (iv) *x* - 1, *y* - 1, *z*; (v) *x*, *y* - 1, *z*; (vi) 1 - *x*, -*y*, -*z*.]

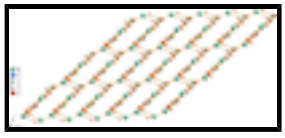

Fig. 2. 2D layer $_{\infty}[Na_2(H_2pdtc)]$ parallel to (001) generated from bridging sodium cations and ($H_2pdtc^{2-}$).

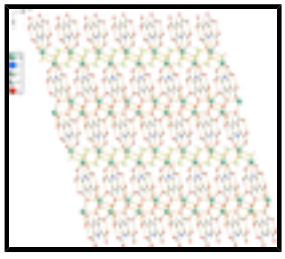

Fig. 3. 3D framework built from linking $_{\infty}[Na_2(H_2pdtc)]$ layers by the water molecules (the water molecule linkers are marked in green)

**Disodium dihydrogen pyridine-2,3,5,6-tetracarboxylate trihydrate**

*Crystal data*

| | |
|---|---|
| $2Na^{+}\cdot C_9H_3NO_8^{2-}\cdot 3H_2O$ | $Z = 2$ |
| $M_r = 353.15$ | $F(000) = 360$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.837$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 5.5844$ (11) Å | Cell parameters from 4730 reflections |
| $b = 6.6770$ (13) Å | $\theta = 3.3$–$27.5°$ |
| $c = 18.631$ (4) Å | $\mu = 0.23$ mm$^{-1}$ |
| $\alpha = 81.34$ (3)° | $T = 293$ K |
| $\beta = 86.77$ (3)° | Block, colourless |
| $\gamma = 68.36$ (3)° | 0.1 × 0.1 × 0.1 mm |
| $V = 638.4$ (2) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Rigaku R-AXIS RAPID diffractometer | 2247 independent reflections |
| Radiation source: fine-focus sealed tube | 1780 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.018$ |
| Detector resolution: 0 pixels mm$^{-1}$ | $\theta_{max} = 25.0°$, $\theta_{min} = 3.3°$ |
| ω scans | $h = -6\rightarrow6$ |
| Absorption correction: multi-scan (ABSCOR; Higashi, 1995) | $k = -7\rightarrow7$ |
| $T_{min} = 0.97$, $T_{max} = 0.98$ | $l = -22\rightarrow22$ |

5075 measured reflections

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.139$ | H-atom parameters constrained |
| $S = 1.14$ | $w = 1/[\sigma^2(F_o^2) + (0.0916P)^2 + 0.0526P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 2247 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 208 parameters | $\Delta\rho_{max} = 0.55$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.33$ 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.

**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 $F^2 > \sigma(F^2)$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Na1 | 1.28244 (19) | 0.41119 (18) | 0.44136 (5) | 0.0360 (3) |
| Na2 | 0.70103 (18) | 0.05082 (15) | 0.05556 (5) | 0.0275 (3) |
| N | 1.2379 (4) | 0.4267 (3) | 0.29938 (10) | 0.0234 (5) |
| C1 | 1.1039 (4) | 0.3240 (4) | 0.27451 (12) | 0.0222 (5) |
| C2 | 1.0250 (4) | 0.3696 (4) | 0.20111 (12) | 0.0227 (5) |
| C3 | 1.0921 (4) | 0.5287 (4) | 0.15822 (12) | 0.0225 (5) |
| H3A | 1.0392 | 0.5653 | 0.1100 | 0.027* |
| C4 | 1.2339 (4) | 0.6372 (4) | 0.18300 (12) | 0.0205 (5) |
| C5 | 1.3080 (4) | 0.5774 (4) | 0.25638 (12) | 0.0213 (5) |
| C6 | 1.0527 (5) | 0.1626 (4) | 0.33584 (13) | 0.0280 (6) |
| O1 | 1.1267 (4) | 0.1553 (3) | 0.39697 (9) | 0.0364 (5) |
| O2 | 0.9322 (4) | 0.0429 (3) | 0.32087 (10) | 0.0408 (5) |
| C7 | 0.8762 (5) | 0.2689 (4) | 0.16184 (13) | 0.0252 (5) |
| O3 | 0.7999 (4) | 0.1219 (3) | 0.19559 (9) | 0.0361 (5) |
| H3 | 0.8543 | 0.0954 | 0.2396 | 0.043* |
| O4 | 0.8325 (3) | 0.3309 (3) | 0.09639 (9) | 0.0299 (4) |
| C8 | 1.2832 (4) | 0.8074 (4) | 0.12553 (12) | 0.0231 (5) |

| | | | | |
|---|---|---|---|---|
| O5 | 1.1680 (3) | 0.8518 (3) | 0.06780 (9) | 0.0295 (4) |
| O6 | 1.4448 (3) | 0.8925 (3) | 0.13981 (9) | 0.0335 (5) |
| C9 | 1.4680 (5) | 0.6635 (4) | 0.29836 (13) | 0.0270 (6) |
| O7 | 1.5562 (4) | 0.8069 (3) | 0.26636 (10) | 0.0385 (5) |
| H7 | 1.5260 | 0.8359 | 0.2205 | 0.046* |
| O8 | 1.5129 (4) | 0.5911 (3) | 0.36238 (9) | 0.0437 (5) |
| O9 | 1.6891 (4) | 0.2384 (3) | 0.49492 (11) | 0.0466 (5) |
| H9A | 1.8156 | 0.2081 | 0.4655 | 0.056* |
| H9B | 1.7251 | 0.1117 | 0.5202 | 0.056* |
| O10 | 0.8582 (4) | 0.6942 (3) | 0.43369 (10) | 0.0363 (5) |
| H10A | 0.8616 | 0.8205 | 0.4131 | 0.044* |
| H10B | 0.7414 | 0.6720 | 0.4094 | 0.044* |
| O11 | 0.2943 (3) | 0.2647 (3) | 0.00399 (9) | 0.0304 (4) |
| H11A | 0.2605 | 0.3963 | −0.0208 | 0.037* |
| H11B | 0.1455 | 0.2949 | 0.0272 | 0.037* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Na1 | 0.0340 (6) | 0.0478 (6) | 0.0265 (6) | −0.0181 (5) | −0.0043 (4) | 0.0044 (4) |
| Na2 | 0.0285 (5) | 0.0318 (5) | 0.0256 (5) | −0.0155 (4) | −0.0043 (4) | −0.0008 (4) |
| N | 0.0272 (10) | 0.0255 (10) | 0.0180 (10) | −0.0103 (9) | −0.0006 (8) | −0.0027 (8) |
| C1 | 0.0233 (11) | 0.0233 (12) | 0.0200 (12) | −0.0090 (10) | −0.0002 (9) | −0.0012 (9) |
| C2 | 0.0238 (11) | 0.0224 (11) | 0.0227 (12) | −0.0092 (10) | −0.0015 (9) | −0.0036 (9) |
| C3 | 0.0277 (12) | 0.0245 (12) | 0.0150 (11) | −0.0092 (10) | −0.0040 (9) | −0.0018 (9) |
| C4 | 0.0223 (11) | 0.0206 (11) | 0.0190 (11) | −0.0087 (10) | 0.0001 (9) | −0.0020 (9) |
| C5 | 0.0239 (11) | 0.0210 (11) | 0.0189 (11) | −0.0087 (10) | 0.0008 (9) | −0.0012 (9) |
| C6 | 0.0314 (13) | 0.0300 (13) | 0.0237 (13) | −0.0146 (11) | 0.0001 (10) | 0.0013 (10) |
| O1 | 0.0510 (12) | 0.0438 (11) | 0.0195 (10) | −0.0264 (10) | −0.0049 (8) | 0.0057 (8) |
| O2 | 0.0608 (13) | 0.0453 (11) | 0.0292 (10) | −0.0379 (11) | −0.0054 (9) | 0.0056 (8) |
| C7 | 0.0299 (13) | 0.0243 (12) | 0.0228 (13) | −0.0113 (11) | −0.0018 (10) | −0.0031 (10) |
| O3 | 0.0517 (12) | 0.0412 (10) | 0.0275 (10) | −0.0324 (10) | −0.0060 (8) | 0.0008 (8) |
| O4 | 0.0415 (10) | 0.0320 (9) | 0.0227 (9) | −0.0210 (8) | −0.0068 (7) | −0.0009 (7) |
| C8 | 0.0273 (12) | 0.0261 (12) | 0.0169 (11) | −0.0116 (10) | 0.0006 (9) | −0.0017 (9) |
| O5 | 0.0327 (9) | 0.0376 (10) | 0.0201 (9) | −0.0176 (8) | −0.0049 (7) | 0.0046 (7) |
| O6 | 0.0455 (11) | 0.0461 (11) | 0.0213 (9) | −0.0337 (10) | −0.0057 (8) | 0.0040 (8) |
| C9 | 0.0331 (13) | 0.0292 (13) | 0.0226 (13) | −0.0171 (11) | −0.0036 (10) | 0.0007 (10) |
| O7 | 0.0557 (12) | 0.0519 (12) | 0.0234 (9) | −0.0394 (11) | −0.0088 (8) | 0.0027 (8) |
| O8 | 0.0642 (14) | 0.0651 (13) | 0.0189 (10) | −0.0469 (12) | −0.0146 (9) | 0.0090 (9) |
| O9 | 0.0342 (11) | 0.0582 (13) | 0.0355 (11) | −0.0105 (10) | 0.0001 (9) | 0.0132 (10) |
| O10 | 0.0387 (10) | 0.0452 (11) | 0.0304 (10) | −0.0236 (9) | −0.0077 (8) | 0.0033 (8) |
| O11 | 0.0286 (9) | 0.0302 (9) | 0.0296 (9) | −0.0098 (8) | 0.0007 (7) | 0.0019 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Na1—O9 | 2.337 (2) | C3—H3A | 0.9300 |
| Na1—O8 | 2.379 (2) | C4—C5 | 1.406 (3) |
| Na1—O10 | 2.420 (2) | C4—C8 | 1.531 (3) |
| Na1—O1 | 2.442 (2) | C5—C9 | 1.528 (3) |

| | | | |
|---|---|---|---|
| Na1—O10$^{i}$ | 2.498 (2) | C6—O1 | 1.221 (3) |
| Na1—N | 2.654 (2) | C6—O2 | 1.287 (3) |
| Na1—O9$^{ii}$ | 2.835 (3) | C7—O4 | 1.235 (3) |
| Na1—Na1$^{i}$ | 3.657 (2) | C7—O3 | 1.278 (3) |
| Na1—Na1$^{ii}$ | 3.948 (2) | O3—H3 | 0.8603 |
| Na2—O11 | 2.358 (2) | C8—O5 | 1.225 (3) |
| Na2—O5$^{iii}$ | 2.4395 (18) | C8—O6 | 1.288 (3) |
| Na2—O6$^{iv}$ | 2.4477 (19) | O5—Na2$^{iii}$ | 2.4395 (18) |
| Na2—O5$^{v}$ | 2.457 (2) | O5—Na2$^{viii}$ | 2.457 (2) |
| Na2—O4 | 2.4738 (18) | O6—Na2$^{ix}$ | 2.4477 (19) |
| Na2—O11$^{vi}$ | 2.516 (2) | C9—O8 | 1.221 (3) |
| Na2—O3 | 2.840 (2) | C9—O7 | 1.287 (3) |
| Na2—C7 | 3.022 (3) | O7—H7 | 0.8596 |
| Na2—Na2$^{vi}$ | 3.444 (2) | O9—Na1$^{ii}$ | 2.835 (3) |
| Na2—Na2$^{vii}$ | 3.724 (2) | O9—H9A | 0.8516 |
| N—C1 | 1.325 (3) | O9—H9B | 0.8625 |
| N—C5 | 1.349 (3) | O10—Na1$^{i}$ | 2.498 (2) |
| C1—C2 | 1.414 (3) | O10—H10A | 0.8790 |
| C1—C6 | 1.539 (3) | O10—H10B | 0.8816 |
| C2—C3 | 1.382 (3) | O11—Na2$^{vi}$ | 2.516 (2) |
| C2—C7 | 1.519 (3) | O11—H11A | 0.8872 |
| C3—C4 | 1.389 (3) | O11—H11B | 0.8833 |
| O9—Na1—O8 | 81.23 (8) | O11—Na2—Na2$^{vii}$ | 120.83 (6) |
| O9—Na1—O10 | 151.17 (9) | O5$^{iii}$—Na2—Na2$^{vii}$ | 40.67 (5) |
| O8—Na1—O10 | 99.80 (8) | O6$^{iv}$—Na2—Na2$^{vii}$ | 146.74 (7) |
| O9—Na1—O1 | 112.23 (9) | O5$^{v}$—Na2—Na2$^{vii}$ | 40.31 (4) |
| O8—Na1—O1 | 120.07 (7) | O4—Na2—Na2$^{vii}$ | 83.00 (6) |
| O10—Na1—O1 | 92.37 (7) | O11$^{vi}$—Na2—Na2$^{vii}$ | 79.10 (6) |
| O9—Na1—O10$^{i}$ | 81.74 (8) | O3—Na2—Na2$^{vii}$ | 108.88 (6) |
| O8—Na1—O10$^{i}$ | 150.12 (7) | C7—Na2—Na2$^{vii}$ | 93.42 (6) |
| O10—Na1—O10$^{i}$ | 83.92 (8) | Na2$^{vi}$—Na2—Na2$^{vii}$ | 102.29 (5) |
| O1—Na1—O10$^{i}$ | 89.14 (7) | C1—N—C5 | 122.15 (19) |
| O9—Na1—N | 119.02 (8) | C1—N—Na1 | 120.01 (14) |
| O8—Na1—N | 62.02 (6) | C5—N—Na1 | 116.33 (15) |
| O10—Na1—N | 85.55 (8) | N—C1—C2 | 121.20 (19) |
| O1—Na1—N | 60.81 (6) | N—C1—C6 | 110.34 (19) |
| O10$^{i}$—Na1—N | 147.64 (7) | C2—C1—C6 | 128.5 (2) |
| O9—Na1—O9$^{ii}$ | 80.93 (8) | C3—C2—C1 | 116.0 (2) |
| O8—Na1—O9$^{ii}$ | 70.80 (7) | C3—C2—C7 | 114.6 (2) |
| O10—Na1—O9$^{ii}$ | 72.45 (7) | C1—C2—C7 | 129.44 (19) |
| O1—Na1—O9$^{ii}$ | 163.26 (7) | C2—C3—C4 | 123.8 (2) |
| O10$^{i}$—Na1—O9$^{ii}$ | 82.40 (7) | C2—C3—H3A | 118.1 |
| N—Na1—O9$^{ii}$ | 123.02 (7) | C4—C3—H3A | 118.1 |

| | | | |
|---|---|---|---|
| O9—Na1—Na1$^{i}$ | 118.79 (7) | C3—C4—C5 | 116.01 (19) |
| O8—Na1—Na1$^{i}$ | 134.81 (8) | C3—C4—C8 | 114.27 (19) |
| O10—Na1—Na1$^{i}$ | 42.77 (5) | C5—C4—C8 | 129.72 (19) |
| O1—Na1—Na1$^{i}$ | 90.98 (6) | N—C5—C4 | 120.8 (2) |
| O10$^{i}$—Na1—Na1$^{i}$ | 41.15 (5) | N—C5—C9 | 111.11 (19) |
| N—Na1—Na1$^{i}$ | 121.70 (7) | C4—C5—C9 | 128.05 (19) |
| O9$^{ii}$—Na1—Na1$^{i}$ | 73.15 (6) | O1—C6—O2 | 122.8 (2) |
| O9—Na1—Na1$^{ii}$ | 45.17 (6) | O1—C6—C1 | 118.3 (2) |
| O8—Na1—Na1$^{ii}$ | 70.94 (5) | O2—C6—C1 | 118.9 (2) |
| O10—Na1—Na1$^{ii}$ | 107.58 (7) | C6—O1—Na1 | 127.72 (16) |
| O1—Na1—Na1$^{ii}$ | 155.73 (7) | O4—C7—O3 | 120.7 (2) |
| O10$^{i}$—Na1—Na1$^{ii}$ | 79.63 (5) | O4—C7—C2 | 118.50 (19) |
| N—Na1—Na1$^{ii}$ | 132.72 (6) | O3—C7—C2 | 120.8 (2) |
| O9$^{ii}$—Na1—Na1$^{ii}$ | 35.76 (5) | O4—C7—Na2 | 52.54 (11) |
| Na1$^{i}$—Na1—Na1$^{ii}$ | 94.41 (5) | O3—C7—Na2 | 69.52 (13) |
| O11—Na2—O5$^{iii}$ | 80.18 (7) | C2—C7—Na2 | 164.48 (15) |
| O11—Na2—O6$^{iv}$ | 82.71 (7) | C7—O3—Na2 | 85.55 (14) |
| O5$^{iii}$—Na2—O6$^{iv}$ | 150.56 (7) | C7—O3—H3 | 107.5 |
| O11—Na2—O5$^{v}$ | 161.10 (7) | Na2—O3—H3 | 160.1 |
| O5$^{iii}$—Na2—O5$^{v}$ | 80.98 (7) | C7—O4—Na2 | 104.12 (14) |
| O6$^{iv}$—Na2—O5$^{v}$ | 113.75 (7) | O5—C8—O6 | 123.9 (2) |
| O11—Na2—O4 | 101.38 (7) | O5—C8—C4 | 117.35 (19) |
| O5$^{iii}$—Na2—O4 | 89.54 (6) | O6—C8—C4 | 118.8 (2) |
| O6$^{iv}$—Na2—O4 | 117.34 (7) | C8—O5—Na2$^{iii}$ | 130.76 (15) |
| O5$^{v}$—Na2—O4 | 79.82 (7) | C8—O5—Na2$^{viii}$ | 123.43 (15) |
| O11—Na2—O11$^{vi}$ | 90.16 (7) | Na2$^{iii}$—O5—Na2$^{viii}$ | 99.02 (7) |
| O5$^{iii}$—Na2—O11$^{vi}$ | 78.64 (7) | C8—O6—Na2$^{ix}$ | 128.80 (14) |
| O6$^{iv}$—Na2—O11$^{vi}$ | 77.63 (6) | O8—C9—O7 | 121.0 (2) |
| O5$^{v}$—Na2—O11$^{vi}$ | 84.77 (7) | O8—C9—C5 | 118.5 (2) |
| O4—Na2—O11$^{vi}$ | 161.90 (7) | O7—C9—C5 | 120.5 (2) |
| O11—Na2—O3 | 117.19 (7) | C9—O7—H7 | 112.8 |
| O5$^{iii}$—Na2—O3 | 134.97 (6) | C9—O8—Na1 | 126.87 (16) |
| O6$^{iv}$—Na2—O3 | 74.36 (6) | Na1—O9—Na1$^{ii}$ | 99.07 (8) |
| O5$^{v}$—Na2—O3 | 77.80 (7) | Na1—O9—H9A | 115.5 |
| O4—Na2—O3 | 47.92 (6) | Na1$^{ii}$—O9—H9A | 104.4 |
| O11$^{vi}$—Na2—O3 | 137.20 (7) | Na1—O9—H9B | 118.8 |
| O11—Na2—C7 | 113.48 (7) | Na1$^{ii}$—O9—H9B | 120.3 |
| O5$^{iii}$—Na2—C7 | 110.79 (7) | H9A—O9—H9B | 98.8 |
| O6$^{iv}$—Na2—C7 | 97.96 (7) | Na1—O10—Na1$^{i}$ | 96.08 (8) |
| O5$^{v}$—Na2—C7 | 74.82 (7) | Na1—O10—H10A | 112.2 |
| O4—Na2—C7 | 23.34 (6) | Na1$^{i}$—O10—H10A | 129.4 |

| | | | |
|---|---|---|---|
| O11$^{vi}$—Na2—C7 | 155.41 (7) | Na1—O10—H10B | 116.3 |
| O3—Na2—C7 | 24.93 (6) | Na1$^{i}$—O10—H10B | 97.7 |
| O11—Na2—Na2$^{vi}$ | 46.94 (5) | H10A—O10—H10B | 105.2 |
| O5$^{iii}$—Na2—Na2$^{vi}$ | 74.89 (5) | Na2—O11—Na2$^{vi}$ | 89.84 (7) |
| O6$^{iv}$—Na2—Na2$^{vi}$ | 75.92 (5) | Na2—O11—H11A | 122.9 |
| O5$^{v}$—Na2—Na2$^{vi}$ | 125.55 (7) | Na2$^{vi}$—O11—H11A | 121.8 |
| O4—Na2—Na2$^{vi}$ | 146.06 (7) | Na2—O11—H11B | 125.4 |
| O11$^{vi}$—Na2—Na2$^{vi}$ | 43.23 (5) | Na2$^{vi}$—O11—H11B | 100.3 |
| O3—Na2—Na2$^{vi}$ | 148.10 (6) | H11A—O11—H11B | 96.5 |
| C7—Na2—Na2$^{vi}$ | 159.59 (7) | | |

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*+3, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*+1, −*z*; (iv) *x*−1, *y*−1, *z*; (v) *x*, *y*−1, *z*; (vi) −*x*+1, −*y*, −*z*; (vii) −*x*+2, −*y*, −*z*; (viii) *x*, *y*+1, *z*; (ix) *x*+1, *y*+1, *z*.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O2 | 0.86 | 1.55 | 2.410 | 175 |
| O7—H7···O6 | 0.86 | 1.55 | 2.402 | 175 |
| O9—H9A···O1$^{x}$ | 0.85 | 2.05 | 2.900 | 175 |
| O9—H9B···O1$^{xi}$ | 0.86 | 2.11 | 2.945 | 161 |
| O10—H10A···O2$^{viii}$ | 0.88 | 2.21 | 3.024 | 153 |
| O10—H10B···O8$^{xii}$ | 0.88 | 1.86 | 2.738 | 173 |
| O11—H11A···O4$^{xiii}$ | 0.89 | 2.05 | 2.917 | 167 |
| O11—H11B···O4$^{xii}$ | 0.88 | 2.08 | 2.949 | 170 |

Symmetry codes: (x) *x*+1, *y*, *z*; (xi) −*x*+3, −*y*, −*z*+1; (viii) *x*, *y*+1, *z*; (xii) *x*−1, *y*, *z*; (xiii) −*x*+1, −*y*+1, −*z*.

**Fig. 1**

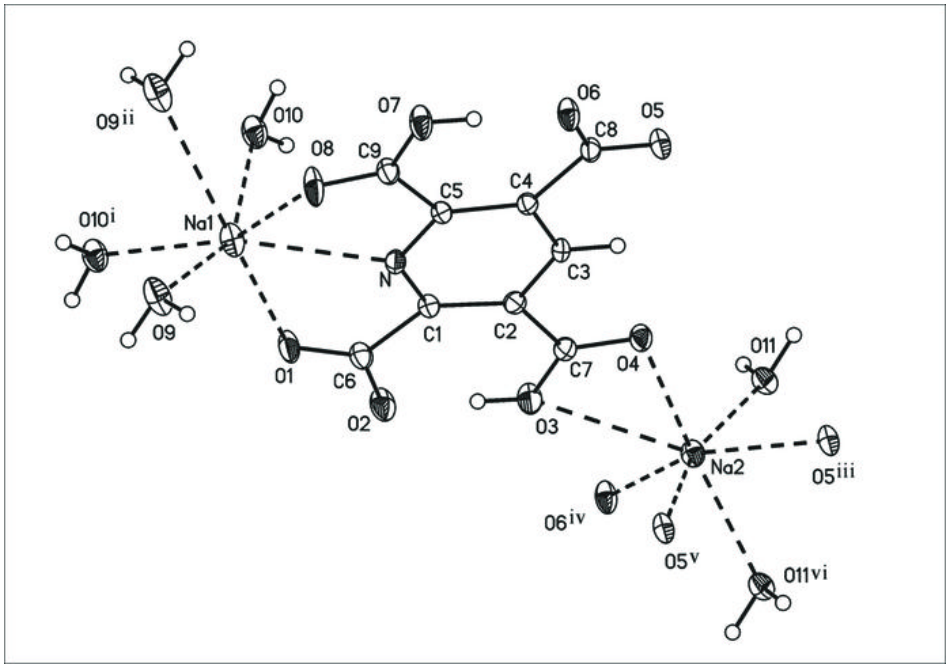
O9ii
O10
O7
O6
O5
O8
C9
C8
C5
C4
O10i
Na1
N
C3
O9
C1
C2
O1
C6
C7
O4
O11
O2
O3
Na2
O5iii
O6iv
O5v
O11vi

**Fig. 2**

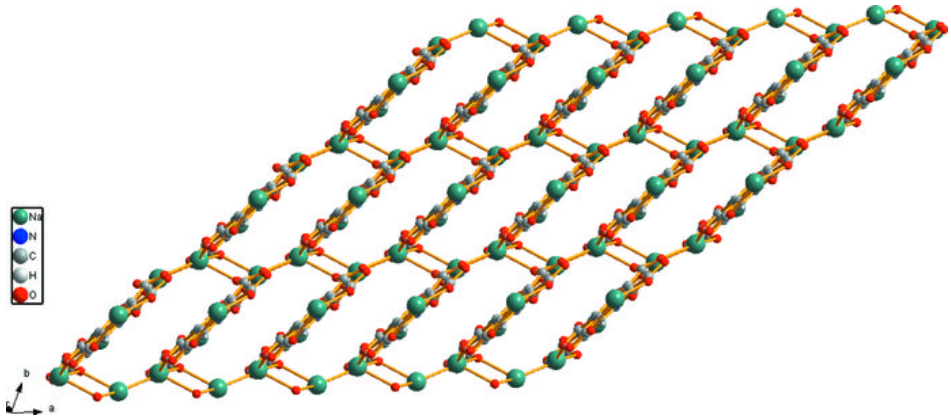

**Fig. 3**