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{2-[2-(Carboxymethoxy)phenoxy]acetato}-[2,2′-(*o*-phenylenedioxy)diacetic acid]-sodium 4,4′-bipyridine hemisolvate monohydrate

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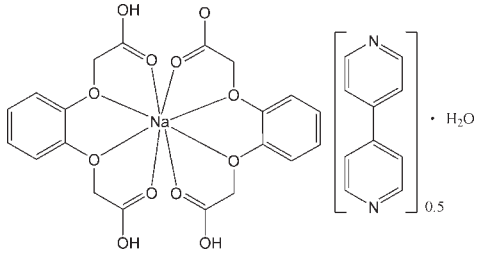
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.066; wR factor = 0.213; data-to-parameter ratio = 11.4.

In the title compound, $[Na(C_{10}H_9O_6)(C_{10}H_{10}O_6)]\cdot 0.5C_{10}H_8N_2\cdot H_2O$, the Na atom is eight-coordinated in an distorted dicapped-octahedral geometry by eight O atoms from a 2-(2-carboxymethoxyphenoxy)acetate (*o*-BDOAH) anion and a 2,2′-(*o*-phenylenedioxy)diacetic acid (*o*-$BDOAH_2$) molecule. The uncoordinated 4,4′-bipyridine molecule is arranged around an inversion center and exhibits rotational disorder. A three-dimensional supramolecular network is built up in the crystal through O—H···O and O—H···N hydrogen bonds between the uncoordinated water molecule, the uncoordinated 4,4′-bipyridine molecule and the sodium complex molecule.

Related literature

For a related structure with *o*-$BDOAH_2$ and 4,4′-bipyridine, see: Gao *et al.* (2006).



Experimental

Crystal data

$[Na(C_{10}H_9O_6)(C_{10}H_{10}O_6)]\cdot 0.5C_{10}H_8N_2\cdot H_2O$
M_r = 570.45
Triclinic, $P\overline{1}$
a = 8.427 (4) Å
b = 13.135 (7) Å
c = 13.411 (9) Å
α = 111.08 (2)°
β = 102.03 (2)°
γ = 103.17 (2)°
V = 1277.9 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 291 K
0.24 × 0.23 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.968, T_{max} = 0.972
9959 measured reflections
4457 independent reflections
3497 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.213
S = 1.14
4457 reflections
392 parameters
152 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H62···O8^{i}	0.83	1.70	2.517 (4)	173
O6—H61···N1B	0.82	1.76	2.568 (7)	168
O6—H61···N1A	0.82	1.76	2.573 (5)	171
O13—H132···O2^{ii}	0.85	2.16	2.958 (5)	156

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); 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: SI2266).

References

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supporting information

Acta Cryst. (2010). E**66**, m867 [https://doi.org/10.1107/S1600536810024797]

{2-[2-(Carboxymethoxy)phenoxy]acetato}[2,2′-(*o*-phenylenedioxy)diacetic acid]sodium 4,4′-bipyridine hemisolvate monohydrate

Liang Xu, Li-Yan Gu, Yan-Yun Yang, Bing Wang and Ting-Guo Kang

S1. Comment

1,2-Phenylenedioxydiacetic acid *o*-$BDOAH_2$ is a multidentate flexible ligand, which can be regarded as an excellent candidate for the construction of supramolecular architectures through the coordination or hydrogen bonding interaction (Gao *et al.* 2006). Herein, we report the title compound structure, synthesized by the reaction of *o*-$BDOAH_2$, 4,4′-bipyridine and NaOH in an aqueous solution.

The title compound consists of a Na(*o*-BDOAH)(*o*-$BDOAH_2$) coordinated complex, a water molecule and hemi-4,4′-bipyridine molecule. The sodium is eight-coordinated in an distorted dicapped octahedral geometry environment defined by eight oxygen atoms from a *o*-BDOAH molecule and a *o*-BDOAH~2~ molecule. The uncoordinated 4,4′-bipyridine molecule is arranged around an inversion center and exhibits a rotational disorder with occupancy of 0.682 (17) and 0.318 (17) for two splitting positions, respectively. (Figure 1, Table 1).

A three-dimensional supramolecular network is built up through O—H···O and O—H···N hydrogen bonds between the lattice water molecule, uncoordinated 4,4′-bipyridine molecule and the sodium complex (Table 2).

S2. Experimental

o-$BDOAH_2$ was prepared by the reaction of chloroacetic acid with *o*-dihydroxybenzene. *o*-$BDOAH_2$ (0.05 g, 2.5 mmol) and 4,4′-bipyridine (0.16 g, 1.0 mmol) were dissolved in 15 ml water, and then the pH was adjusted to about 5.5 using 0.5 *M* NaOH solution with stirring. The resulting solution was stand in the atmosphere for several days. Colorless block crystals of title compound were obtained.

S3. Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic C), C—H = 0.97 Å (methene C), and with U_{iso}(H) = 1.2U_{eq}(C). Water H atoms and carboxyl H atoms were initially located in a difference Fourier map but they were treated with O—H = 0.82 - 0.85 Å, and with U_{iso}(H) = 1.5U_{eq}(O). The pyridine ring of the centrosymmetric 4,4′-bipyridine molecule is rotational disordered (approximately along the N1A···C23A/N1B···C23B axis), which are splitted into two positions with free refined occupancy of 0.682 (17) and 0.318 (17), respectively.

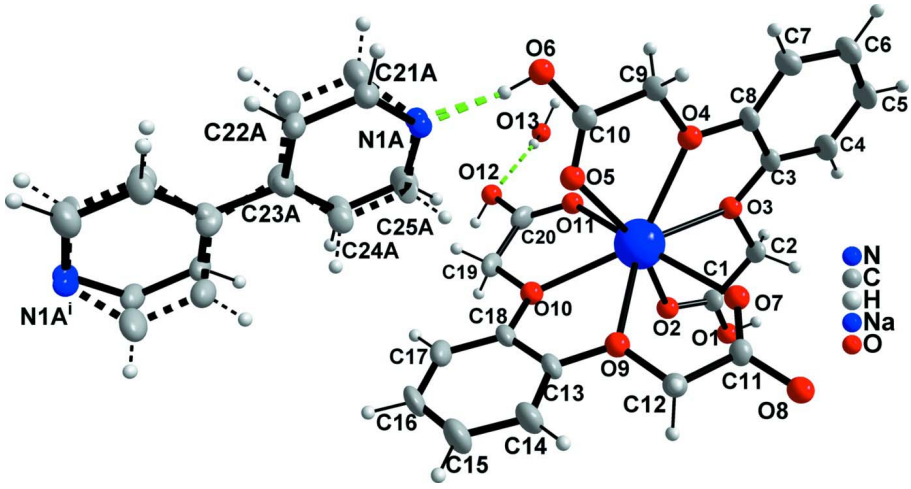

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids at the 30% probability level for non-H atoms. Dashed lines indicate the hydrogen bonds. Symmetry code i: -*x* + 1, -*y*, -*z*. Atoms that belong to the minor fraction of the disordered bipyridine molecule were omitted for clarity.

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Crystal data

$[Na(C_{10}H_9O_6)(C_{10}H_{10}O_6)]\cdot 0.5C_{10}H_8N_2\cdot H_2O$
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Triclinic, $P\overline{1}$
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a = 8.427 (4) Å
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c = 13.411 (9) Å
α = 111.08 (2)°
β = 102.03 (2)°
γ = 103.17 (2)°
V = 1277.9 (13) Å^3
Z = 2
$F(000)$ = 594
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9090 reflections
θ = 3.1–27.6°
μ = 0.14 mm^{-1}
T = 291 K
Block, colorless
0.24 × 0.23 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.968, T_{max} = 0.972
9959 measured reflections
4457 independent reflections
3497 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°, θ_{min} = 3.1°
h = −9→10
k = −15→15
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.213
S = 1.14
4457 reflections
392 parameters
152 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.1059P)^2 + 1.3876P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.79$ e Å^{-3}
	$\Delta\rho_{min} = -0.43$ e Å^{-3}

Special details

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

Refinement. 1. omit -3 50 2. eqiv $4 - *x* -1, -*y*, -*z* *DFIX* 1.485 0.015 C23A C23A_$4 C23B C23B_$4 3. SAME N1B C21B C22B C23B C24B C25B SAME N1A C21A C22A C23A C24A C25A SIMU C21A C22A C23A C24A C25A C21B C22B C23B C24B C25B SIMU N1A N1B

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}	Occ. (<1)
N1A	0.6188 (9)	0.2495 (3)	0.2549 (4)	0.057 (3)	0.682 (17)
C21A	0.5554 (11)	0.1402 (4)	0.2509 (3)	0.051 (2)	0.682 (17)
H21A	0.5436	0.1326	0.3156	0.062*	0.682 (17)
C22A	0.5096 (11)	0.0423 (3)	0.1503 (4)	0.0451 (19)	0.682 (17)
H22A	0.4672	−0.0309	0.1476	0.054*	0.682 (17)
C23A	0.5272 (8)	0.0536 (4)	0.0537 (3)	0.042 (3)	0.682 (17)
C24A	0.5907 (14)	0.1629 (6)	0.0577 (5)	0.050 (2)	0.682 (17)
H24A	0.6024	0.1706	−0.0069	0.060*	0.682 (17)
C25A	0.6365 (14)	0.2609 (4)	0.1583 (6)	0.061 (3)	0.682 (17)
H25A	0.6789	0.3340	0.1610	0.073*	0.682 (17)
N1B	0.591 (2)	0.2514 (7)	0.2486 (8)	0.062 (7)	0.318 (17)
C21B	0.461 (3)	0.1518 (9)	0.2270 (11)	0.067 (5)	0.318 (17)
H21B	0.4017	0.1518	0.2782	0.080*	0.318 (17)
C22B	0.420 (3)	0.0521 (7)	0.1287 (11)	0.056 (5)	0.318 (17)
H22B	0.3333	−0.0145	0.1142	0.067*	0.318 (17)
C23B	0.509 (2)	0.0521 (9)	0.0521 (7)	0.049 (8)	0.318 (17)
C24B	0.6381 (16)	0.1517 (12)	0.0738 (10)	0.036 (4)	0.318 (17)
H24B	0.6974	0.1517	0.0225	0.043*	0.318 (17)
C25B	0.6791 (16)	0.2514 (10)	0.1720 (11)	0.029 (3)	0.318 (17)
H25B	0.7658	0.3180	0.1865	0.034*	0.318 (17)
C1	1.2333 (5)	0.9344 (3)	0.3705 (3)	0.0449 (9)	
C2	1.2214 (5)	0.9812 (3)	0.4873 (3)	0.0427 (9)	
H2A	1.3347	1.0083	0.5419	0.051*	
H2B	1.1801	1.0465	0.4995	0.051*	
C3	1.1024 (5)	0.9213 (3)	0.6109 (3)	0.0393 (8)	
C4	1.1633 (5)	1.0313 (3)	0.6957 (3)	0.0496 (10)	
H4	1.2166	1.0933	0.6827	0.060*	
C5	1.1456 (6)	1.0503 (4)	0.8009 (4)	0.0616 (12)	

H5	1.1865	1.1251	0.8580	0.074*
C6	1.0677 (6)	0.9588 (4)	0.8206 (4)	0.0633 (12)
H6	1.0564	0.9718	0.8913	0.076*
C7	1.0057 (6)	0.8471 (4)	0.7356 (3)	0.0537 (10)
H7	0.9524	0.7854	0.7491	0.064*
C8	1.0231 (5)	0.8276 (3)	0.6309 (3)	0.0404 (8)
C9	0.8604 (5)	0.6264 (3)	0.5498 (3)	0.0432 (9)
H9A	0.7599	0.6441	0.5650	0.052*
H9B	0.9204	0.6126	0.6111	0.052*
C10	0.8062 (5)	0.5200 (3)	0.4395 (3)	0.0424 (9)
C11	1.4603 (5)	0.7310 (3)	0.4655 (3)	0.0405 (8)
C12	1.4237 (5)	0.6162 (3)	0.3699 (3)	0.0441 (9)
H12A	1.4988	0.6235	0.3251	0.053*
H12B	1.4432	0.5606	0.3987	0.053*
C13	1.1877 (5)	0.4758 (3)	0.2073 (3)	0.0453 (9)
C14	1.2722 (7)	0.3957 (4)	0.1820 (4)	0.0633 (12)
H14	1.3785	0.4095	0.2312	0.076*
C15	1.1982 (8)	0.2955 (4)	0.0837 (5)	0.0784 (16)
H15	1.2553	0.2419	0.0665	0.094*
C16	1.0408 (7)	0.2740 (4)	0.0108 (4)	0.0755 (16)
H16	0.9909	0.2051	−0.0544	0.091*
C17	0.9552 (6)	0.3544 (4)	0.0336 (4)	0.0629 (13)
H17	0.8508	0.3408	−0.0174	0.076*
C18	1.0267 (5)	0.4546 (3)	0.1329 (3)	0.0454 (9)
C19	0.7868 (5)	0.5188 (4)	0.0964 (3)	0.0524 (11)
H19A	0.7130	0.4413	0.0765	0.063*
H19B	0.7944	0.5247	0.0274	0.063*
C20	0.7118 (5)	0.6051 (4)	0.1570 (4)	0.0558 (11)
Na1	1.05417 (13)	0.69107 (9)	0.36791 (8)	0.0184 (3)
O1	1.3113 (4)	1.0102 (3)	0.3415 (3)	0.0670 (9)
H62	1.3358	1.0765	0.3903	0.100*
O2	1.1770 (4)	0.8307 (2)	0.3072 (2)	0.0532 (7)
O3	1.1079 (3)	0.8946 (2)	0.5032 (2)	0.0402 (6)
O4	0.9707 (3)	0.7210 (2)	0.5416 (2)	0.0451 (6)
O5	0.8786 (4)	0.5185 (2)	0.3693 (2)	0.0567 (8)
O6	0.6840 (5)	0.4372 (2)	0.4313 (3)	0.0691 (10)
H61	0.6665	0.3817	0.3714	0.104*
O7	1.3450 (4)	0.7679 (3)	0.4858 (2)	0.0601 (8)
O8	1.6201 (3)	0.7822 (2)	0.5237 (3)	0.0588 (8)
O9	1.2474 (3)	0.5785 (2)	0.3023 (2)	0.0503 (7)
O10	0.9534 (3)	0.5391 (2)	0.1663 (2)	0.0499 (7)
O11	0.7838 (4)	0.6794 (3)	0.2531 (3)	0.0636 (8)
O12	0.5598 (4)	0.5927 (4)	0.0985 (3)	0.0941 (14)
H121	0.5767	0.5753	0.0368	0.141*
O13	0.4114 (5)	0.7809 (3)	0.1738 (4)	0.0892 (12)
H131	0.4615	0.7313	0.1724	0.134*
H132	0.3730	0.8057	0.2284	0.134*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.066 (5)	0.043 (6)	0.052 (6)	0.014 (4)	0.018 (4)	0.014 (5)
C21A	0.058 (5)	0.042 (4)	0.041 (3)	0.001 (3)	0.020 (3)	0.012 (3)
C22A	0.051 (5)	0.034 (3)	0.036 (3)	−0.002 (3)	0.015 (3)	0.010 (2)
C23A	0.049 (5)	0.037 (6)	0.034 (6)	0.011 (4)	0.018 (4)	0.008 (5)
C24A	0.076 (6)	0.034 (3)	0.049 (4)	0.024 (4)	0.023 (4)	0.023 (3)
C25A	0.082 (7)	0.042 (4)	0.062 (5)	0.027 (4)	0.025 (4)	0.020 (4)
N1B	0.098 (13)	0.044 (12)	0.024 (9)	−0.010 (8)	0.029 (8)	0.008 (8)
C21B	0.097 (14)	0.044 (8)	0.052 (8)	0.013 (9)	0.034 (9)	0.014 (6)
C22B	0.061 (10)	0.044 (7)	0.047 (8)	0.001 (7)	0.020 (8)	0.012 (6)
C23B	0.061 (11)	0.040 (12)	0.043 (13)	0.005 (9)	0.014 (9)	0.023 (10)
C24B	0.045 (8)	0.034 (7)	0.036 (6)	0.021 (6)	0.010 (5)	0.020 (5)
C25B	0.044 (6)	0.020 (5)	0.030 (6)	0.012 (4)	0.009 (5)	0.020 (4)
C1	0.047 (2)	0.033 (2)	0.046 (2)	0.0057 (16)	0.0147 (17)	0.0109 (17)
C2	0.0401 (19)	0.0312 (19)	0.045 (2)	0.0077 (15)	0.0110 (16)	0.0084 (16)
C3	0.0376 (19)	0.0364 (19)	0.0345 (19)	0.0144 (15)	0.0075 (15)	0.0059 (15)
C4	0.057 (2)	0.033 (2)	0.046 (2)	0.0150 (17)	0.0147 (19)	0.0028 (17)
C5	0.075 (3)	0.045 (2)	0.038 (2)	0.016 (2)	0.015 (2)	−0.0074 (19)
C6	0.074 (3)	0.064 (3)	0.034 (2)	0.016 (2)	0.019 (2)	0.005 (2)
C7	0.063 (3)	0.054 (2)	0.040 (2)	0.019 (2)	0.0191 (19)	0.0151 (19)
C8	0.043 (2)	0.0342 (19)	0.0324 (18)	0.0142 (15)	0.0088 (15)	0.0032 (15)
C9	0.054 (2)	0.0333 (19)	0.041 (2)	0.0136 (16)	0.0191 (17)	0.0139 (16)
C10	0.052 (2)	0.034 (2)	0.042 (2)	0.0178 (17)	0.0177 (18)	0.0148 (17)
C11	0.040 (2)	0.0375 (19)	0.0386 (19)	0.0119 (16)	0.0063 (16)	0.0150 (16)
C12	0.038 (2)	0.045 (2)	0.043 (2)	0.0134 (16)	0.0134 (16)	0.0118 (17)
C13	0.053 (2)	0.039 (2)	0.0298 (18)	0.0097 (17)	0.0136 (17)	0.0017 (16)
C14	0.073 (3)	0.051 (3)	0.053 (3)	0.026 (2)	0.022 (2)	0.003 (2)
C15	0.088 (4)	0.056 (3)	0.066 (3)	0.029 (3)	0.025 (3)	−0.004 (2)
C16	0.082 (4)	0.050 (3)	0.057 (3)	0.013 (2)	0.028 (3)	−0.017 (2)
C17	0.055 (3)	0.063 (3)	0.040 (2)	−0.002 (2)	0.0141 (19)	0.003 (2)
C18	0.051 (2)	0.041 (2)	0.0325 (19)	0.0070 (17)	0.0195 (17)	0.0044 (16)
C19	0.040 (2)	0.065 (3)	0.033 (2)	0.0003 (19)	0.0008 (16)	0.0171 (19)
C20	0.033 (2)	0.078 (3)	0.049 (3)	0.010 (2)	0.0011 (18)	0.030 (2)
Na1	0.0191 (5)	0.0129 (5)	0.0135 (5)	0.0036 (4)	0.0030 (4)	−0.0026 (4)
O1	0.081 (2)	0.0409 (17)	0.063 (2)	−0.0012 (15)	0.0347 (17)	0.0113 (15)
O2	0.0685 (19)	0.0348 (15)	0.0458 (16)	0.0087 (13)	0.0225 (14)	0.0088 (13)
O3	0.0462 (14)	0.0321 (13)	0.0340 (13)	0.0111 (11)	0.0135 (11)	0.0059 (11)
O4	0.0557 (16)	0.0328 (13)	0.0371 (14)	0.0091 (11)	0.0181 (12)	0.0061 (11)
O5	0.075 (2)	0.0370 (15)	0.0499 (17)	0.0111 (14)	0.0299 (15)	0.0090 (13)
O6	0.094 (2)	0.0361 (16)	0.064 (2)	0.0022 (16)	0.0425 (18)	0.0086 (14)
O7	0.0496 (17)	0.0565 (18)	0.0506 (17)	0.0271 (14)	0.0010 (14)	−0.0001 (14)
O8	0.0388 (15)	0.0465 (16)	0.0674 (19)	0.0063 (12)	0.0076 (14)	0.0086 (14)
O9	0.0458 (15)	0.0449 (15)	0.0371 (14)	0.0177 (12)	0.0029 (12)	−0.0031 (12)
O10	0.0428 (15)	0.0551 (17)	0.0328 (14)	0.0081 (12)	0.0052 (11)	0.0069 (12)
O11	0.0488 (17)	0.064 (2)	0.063 (2)	0.0190 (15)	0.0049 (15)	0.0179 (17)
O12	0.051 (2)	0.139 (4)	0.069 (2)	0.035 (2)	0.0010 (18)	0.026 (2)

O13	0.086 (3)	0.084 (3)	0.109 (3)	0.034 (2)	0.039 (2)	0.046 (2)

Geometric parameters (Å, º)

N1A—C21A	1.3900	C9—H9A	0.9700
N1A—C25A	1.3900	C9—H9B	0.9700
C21A—C22A	1.3900	C10—O5	1.221 (5)
C21A—H21A	0.9300	C10—O6	1.269 (5)
C22A—C23A	1.3900	C11—O7	1.217 (5)
C22A—H22A	0.9300	C11—O8	1.283 (5)
C23A—C24A	1.3900	C11—C12	1.499 (5)
C23A—C23A^{i}	1.497 (7)	C12—O9	1.434 (5)
C24A—C25A	1.3900	C12—H12A	0.9700
C24A—H24A	0.9300	C12—H12B	0.9700
C25A—H25A	0.9300	C13—O9	1.372 (4)
N1B—C21B	1.3900	C13—C14	1.383 (6)
N1B—C25B	1.3900	C13—C18	1.407 (6)
C21B—C22B	1.3900	C14—C15	1.376 (6)
C21B—H21B	0.9300	C14—H14	0.9300
C22B—C23B	1.3900	C15—C16	1.373 (8)
C22B—H22B	0.9300	C15—H15	0.9300
C23B—C24B	1.3900	C16—C17	1.391 (7)
C23B—C23B^{i}	1.516 (12)	C16—H16	0.9300
C24B—C25B	1.3900	C17—C18	1.382 (6)
C24B—H24B	0.9300	C17—H17	0.9300
C25B—H25B	0.9300	C18—O10	1.373 (5)
C1—O2	1.227 (5)	C19—O10	1.421 (5)
C1—O1	1.289 (5)	C19—C20	1.491 (7)
C1—C2	1.497 (6)	C19—H19A	0.9700
C2—O3	1.421 (5)	C19—H19B	0.9700
C2—H2A	0.9700	C20—O11	1.218 (5)
C2—H2B	0.9700	C20—O12	1.294 (5)
C3—C4	1.372 (5)	Na1—O7	2.374 (3)
C3—O3	1.373 (5)	Na1—O2	2.379 (3)
C3—C8	1.403 (6)	Na1—O11	2.407 (3)
C4—C5	1.387 (6)	Na1—O5	2.416 (3)
C4—H4	0.9300	Na1—O4	2.495 (3)
C5—C6	1.375 (7)	Na1—O3	2.501 (3)
C5—H5	0.9300	Na1—O9	2.524 (3)
C6—C7	1.387 (6)	Na1—O10	2.531 (3)
C6—H6	0.9300	O1—H62	0.8261
C7—C8	1.379 (6)	O6—H61	0.8239
C7—H7	0.9300	O12—H121	0.8280
C8—O4	1.372 (4)	O13—H131	0.8502
C9—O4	1.426 (5)	O13—H132	0.8500
C9—C10	1.513 (5)		
C21A—N1A—C25A	120.0	H12A—C12—H12B	108.5

C22A—C21A—N1A	120.0	O9—C13—C14	125.3 (4)
C22A—C21A—H21A	120.0	O9—C13—C18	114.6 (3)
N1A—C21A—H21A	120.0	C14—C13—C18	120.1 (4)
C21A—C22A—C23A	120.0	C15—C14—C13	119.7 (5)
C21A—C22A—H22A	120.0	C15—C14—H14	120.2
C23A—C22A—H22A	120.0	C13—C14—H14	120.2
C24A—C23A—C22A	120.0	C16—C15—C14	120.6 (5)
C24A—C23A—C23A^{i}	121.5 (7)	C16—C15—H15	119.7
C22A—C23A—C23A^{i}	118.5 (8)	C14—C15—H15	119.7
C23A—C24A—C25A	120.0	C15—C16—C17	120.6 (4)
C23A—C24A—H24A	120.0	C15—C16—H16	119.7
C25A—C24A—H24A	120.0	C17—C16—H16	119.7
C24A—C25A—N1A	120.0	C18—C17—C16	119.5 (4)
C24A—C25A—H25A	120.0	C18—C17—H17	120.3
N1A—C25A—H25A	120.0	C16—C17—H17	120.3
C21B—N1B—C25B	120.0	O10—C18—C17	125.0 (4)
N1B—C21B—C22B	120.0	O10—C18—C13	115.4 (3)
N1B—C21B—H21B	120.0	C17—C18—C13	119.6 (4)
C22B—C21B—H21B	120.0	O10—C19—C20	109.9 (3)
C23B—C22B—C21B	120.0	O10—C19—H19A	109.7
C23B—C22B—H22B	120.0	C20—C19—H19A	109.7
C21B—C22B—H22B	120.0	O10—C19—H19B	109.7
C24B—C23B—C22B	120.0	C20—C19—H19B	109.7
C24B—C23B—C23B^{i}	113.6 (18)	H19A—C19—H19B	108.2
C22B—C23B—C23B^{i}	126.1 (18)	O11—C20—O12	122.4 (5)
C23B—C24B—C25B	120.0	O11—C20—C19	123.8 (4)
C23B—C24B—H24B	120.0	O12—C20—C19	113.8 (4)
C25B—C24B—H24B	120.0	O7—Na1—O2	78.09 (12)
C24B—C25B—N1B	120.0	O7—Na1—O11	160.33 (12)
C24B—C25B—H25B	120.0	O2—Na1—O11	84.79 (12)
N1B—C25B—H25B	120.0	O7—Na1—O5	116.02 (13)
O2—C1—O1	121.8 (4)	O2—Na1—O5	162.48 (11)
O2—C1—C2	122.7 (4)	O11—Na1—O5	82.69 (12)
O1—C1—C2	115.4 (3)	O7—Na1—O4	87.39 (12)
O3—C2—C1	110.6 (3)	O2—Na1—O4	129.15 (10)
O3—C2—H2A	109.5	O11—Na1—O4	96.11 (12)
C1—C2—H2A	109.5	O5—Na1—O4	64.66 (10)
O3—C2—H2B	109.5	O7—Na1—O3	72.13 (10)
C1—C2—H2B	109.5	O2—Na1—O3	66.79 (10)
H2A—C2—H2B	108.1	O11—Na1—O3	92.31 (11)
C4—C3—O3	124.4 (4)	O5—Na1—O3	125.85 (11)
C4—C3—C8	119.8 (4)	O4—Na1—O3	62.37 (9)
O3—C3—C8	115.8 (3)	O7—Na1—O9	63.92 (10)
C3—C4—C5	120.2 (4)	O2—Na1—O9	90.25 (11)
C3—C4—H4	119.9	O11—Na1—O9	126.36 (11)
C5—C4—H4	119.9	O5—Na1—O9	87.35 (11)
C6—C5—C4	120.1 (4)	O4—Na1—O9	126.16 (10)
C6—C5—H5	120.0	O3—Na1—O9	133.82 (10)

C4—C5—H5	120.0	O7—Na1—O10	123.12 (11)
C5—C6—C7	120.2 (4)	O2—Na1—O10	88.79 (11)
C5—C6—H6	119.9	O11—Na1—O10	65.49 (11)
C7—C6—H6	119.9	O5—Na1—O10	74.88 (11)
C8—C7—C6	119.9 (4)	O4—Na1—O10	137.55 (10)
C8—C7—H7	120.0	O3—Na1—O10	148.93 (10)
C6—C7—H7	120.0	O9—Na1—O10	61.03 (9)
O4—C8—C7	125.0 (4)	C1—O1—H62	111.3
O4—C8—C3	115.3 (3)	C1—O2—Na1	121.2 (3)
C7—C8—C3	119.7 (3)	C3—O3—C2	116.6 (3)
O4—C9—C10	108.7 (3)	C3—O3—Na1	121.5 (2)
O4—C9—H9A	109.9	C2—O3—Na1	115.1 (2)
C10—C9—H9A	109.9	C8—O4—C9	117.2 (3)
O4—C9—H9B	109.9	C8—O4—Na1	122.4 (2)
C10—C9—H9B	109.9	C9—O4—Na1	120.3 (2)
H9A—C9—H9B	108.3	C10—O5—Na1	123.5 (2)
O5—C10—O6	126.2 (4)	C10—O6—H61	107.1
O5—C10—C9	120.8 (3)	C11—O7—Na1	126.1 (3)
O6—C10—C9	113.0 (3)	C13—O9—C12	117.2 (3)
O7—C11—O8	124.7 (4)	C13—O9—Na1	122.5 (2)
O7—C11—C12	121.4 (3)	C12—O9—Na1	120.0 (2)
O8—C11—C12	113.8 (3)	C18—O10—C19	117.3 (3)
O9—C12—C11	107.5 (3)	C18—O10—Na1	121.9 (2)
O9—C12—H12A	110.2	C19—O10—Na1	117.8 (2)
C11—C12—H12A	110.2	C20—O11—Na1	122.4 (3)
O9—C12—H12B	110.2	C20—O12—H121	97.1
C11—C12—H12B	110.2	H131—O13—H132	117.5

Symmetry code: (i) $-x+1, -y, -z$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H62···O8^{ii}	0.83	1.70	2.517 (4)	173
O6—H61···N1*B*	0.82	1.76	2.568 (7)	168
O6—H61···N1*A*	0.82	1.76	2.573 (5)	171
O13—H132···O2^{iii}	0.85	2.16	2.958 (5)	156

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