$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 1.9123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters and hydrogen-bonding geometry (Å, °)*

Er—O7	2.040 (2)	Er···Er^i	3.5918 (7)
Er—O1	2.241 (2)	Al—O6	1.697 (2)
Er—O2	2.346 (2)	Al—O5	1.740 (2)
Er—O3	2.355 (2)	Al—O2	1.774 (2)
Er—O4	2.406 (2)	Al—O3	1.785 (2)
Er···Al	3.2041 (10)		
O7—Er—O1	105.16 (8)	O6—Al—O2	120.38 (11)
O7—Er—O2	156.93 (8)	O5—Al—O2	106.79 (10)
O1—Er—O2	94.94 (7)	O6—Al—O3	119.00 (11)
O1^i—Er—O2	92.06 (7)	O5—Al—O3	107.40 (10)
O7—Er—O3	94.82 (8)	O2—Al—O3	89.29 (9)
O1—Er—O3	159.11 (7)	O3—Al—Er	46.27 (7)
O1^i—Er—O3	107.65 (7)	C2—O1—Er	123.8 (2)
O2—Er—O3	64.30 (7)	C2—O1—Er^i	129.5 (2)
O7—Er—O4	90.89 (9)	Er—O1—Er^i	106.36 (7)
O1—Er—O4	93.07 (7)	C1—O2—Er	126.1 (2)
O1^i—Er—O4	161.92 (8)	Al—O2—Er	101.16 (8)
O2—Er—O4	76.62 (7)	C3—O3—Al	125.5 (2)
O3—Er—O4	80.38 (7)	C3—O3—Er	124.7 (2)
O7—Er—Al	124.32 (7)	Al—O3—Er	100.50 (9)
O1—Er—Al	126.04 (5)	C4—O4—Er	131.2 (2)
O6—Al—O5	111.48 (11)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H100···O5	0.84 (2)	1.88 (3)	2.710 (3)	167 (3)

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

The position of the H atom participating in hydrogen bonding was determined from the final difference Fourier map and refined isotropically. All other H atoms were refined with fixed isotropic displacement parameters using a riding model. The largest peak and deepest hole in the final difference Fourier map were located 1.15 and 0.83 Å from C7*B* and Er, respectively.

Data collection: *DIF*4 (Stoe, 1990). Cell refinement: *DIF*4. Data reduction: *X-RED* (Stoe, 1996*a*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1990). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1001). Services for accessing these data are described at the back of the journal.

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A New Phase of Sodium Succinate Hexahydrate

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Abstract

The title compound, $2Na^+.C_4H_4O_4^{2-}.6H_2O$, is made up of chains parallel to the *b* axis, in which Na–$[OH_2]_2$–Na–$[C_4H_4O_2]$–Na–$[OH_2]_2$–Na units are mono-bridged by water molecules. The two central C atoms of the succinate ion are disordered over two positions; the dihedral angles between the carboxylate group and the C—C—C—C backbone in each of its disorder positions are 5.7 (3) and 64.0 (2)°.

Comment

Simple α,ω-dicarboxylate ligands such as oxalate, malonate and succinate are known to exhibit extraordinary versatility in binding metal ions. Many structures of succinates have already been reported. The following univalent cations form non-isostructural salts: Li^+ (Klapper & Küppers, 1973), K^+ (Fonseca *et al.*, 1985), Na^+ (Fonseca

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et al., 1986) and NH_4^+ (Kutzke *et al.*, 1994). The following divalent cations form non-isostructural salts: Ca^{2+} (Karipides & Reed, 1980), Mn^{2+} (Gupta *et al.*, 1983), Ni^{2+} (Gupta & Devi, 1978), Cu^{2+} (O'Connor & Maslen, 1966), Cd^{2+} (Griffith *et al.*, 1982) and Sr^{2+} (Gupta, 1981). In most cases, the succinate moiety binds the particular metal ions in different monodentate modes. Bidentate chelation and four-membered-ring formation by succinate were observed for the Ca^{2+} complex.

Structures of succinic acid have also been reported (Leviel *et al.*, 1981; Petropavlov & Yarantsev, 1983); there are two crystalline forms, α and β, with triclinic and monoclinic symmetry, respectively.

The previously reported crystal structure of sodium succinate hexahydrate, (I), henceforth called the α phase (Fonseca *et al.*, 1986), has triclinic symmetry [cell parameters: $a = 5.545\,(1)$, $b = 6.588\,(4)$, $c = 9.020\,(4)$ Å, $\alpha = 96.87\,(4)$, $\beta = 102.19\,(3)$ and $\gamma = 109.61\,(3)°$]; the monoclinic form (β) is described in this study.

$$2Na^+.^-O(O)C—CH_2—CH_2—C(O)O^-.6H_2O$$

(I)

The α phase (Fonseca *et al.*, 1986) can be described in terms of chains of Na^+ ions parallel to the x axis, linked by a double bridge of H_2O; the intra-chain Na· · ·Na distance is 3.598 (2) Å. The succinate ion links two such chains. The β phase can be described as a chain of $Na–[H_2O]_2–Na$ units, linked by a single bridge of H_2O, parallel to the y axis. The intrachain Na· · ·Na distance is 3.5377 (9) Å between doubly-bridged sodium and 4.1430 (13) Å between singly-bridged sodium. Two double chains are linked by the succinate ion, forming a quadruple chain. The $H_2O—Na—OH_2$ bond angle for water molecules involved in the double bridge is 89.92 (3)° in the β phase and 88 (1)° (average) in the α phase, whereas the corresponding angle for water molecules in single bridges is 102.06 (2)°.

The second main difference between the two phases is a consequence of the first mentioned difference. The two carboxylate groups in the succinate are *trans* (the C1—C2—C2*—C1* torsion angle is 180°, as a consequence of the inversion centre in the middle of the C2—C2* bond), as in all previously determined structures of succinate salts, with the exception of the Cu^{2+} complex, but the orientation of the carboxylate group and the C—C—C—C backbone plane is different between the two phases: dihedral angles are 5.7 (3)° for C2 and 64.0 (2)° for C2′ in the β phase, and 86.6 (4)° for the α phase. This dihedral angle is close to 0° in the NH_4^+ [2.0 (1)°], Mn^{2+} [7.7 (2)°] and Ca^{2+} [18.4 (2)°] complexes, and also in succinic acid [10.0 (2)°], or close to 90° in the Li^+ [87.0 (1)°] and Ca^{2+} [85.4 (2)° for the second succinate ion] complexes, and in the α phase of the Na^+ complex [86.6 (4)°]. Thus, the title compound is the first salt of succinic acid with a synclinal value (*gauche*) for this angle, albeit only for one of the disorder components.

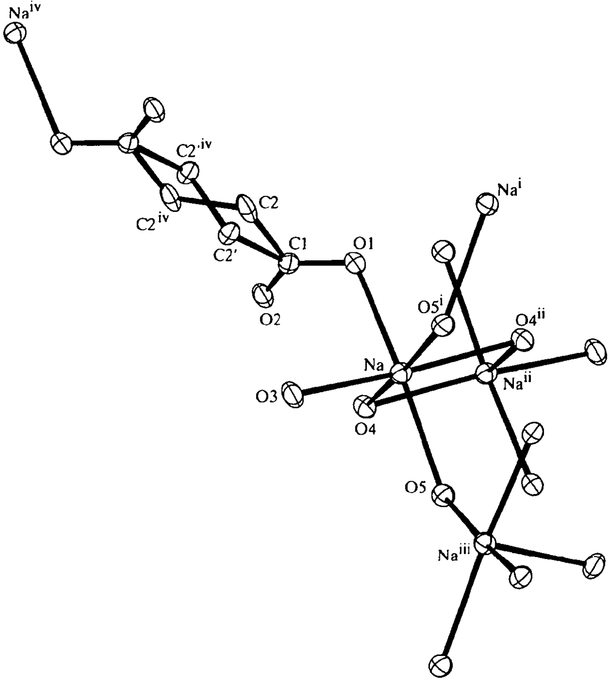
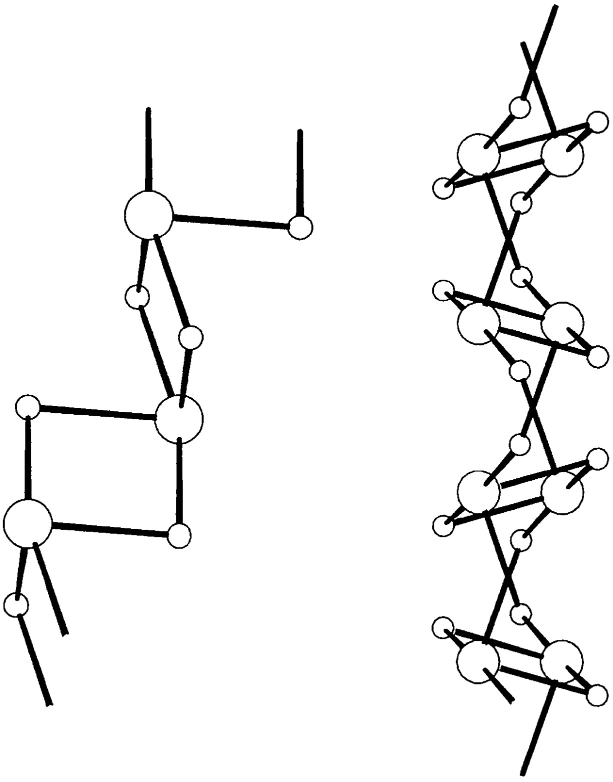


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids and the disorder of the C2 atom. Symmetry codes are given in Table 1.



α phase β phase

Fig. 2. A chain scheme for the two phases of sodium succinate hexahydrate.

Experimental

Attempts to prepare a coordination complex of tungstate with succinic acid yielded the title compound. A solution (10 ml) of sodium tungstate dihydrate (0.5 mol dm^{-3}) and succinic acid (0.5 mol dm^{-3}) was adjusted to pH 5.56 with NaOH. Then dimethyl sulfoxide (6 ml) was added and the mixture was left at room temperature with magnetic stirring. The title compound was isolated as a by-product when the mother liquor was left at low temperature (297 K). The crystals were dried in air and analysed by IR spectroscopy (KBr pellet).

Crystal data

$2Na^+.C_4H_4O_4^{2-}.6H_2O$
$M_r = 270.15$
Monoclinic
$P2_1/c$
$a = 8.9033\,(8)$ Å
$b = 6.300\,(3)$ Å
$c = 10.717\,(2)$ Å
$\beta = 102.614\,(13)°$
$V = 586.6\,(3)$ Å^3
$Z = 2$
$D_x = 1.529$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12$–25°
$\mu = 0.209$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
1812 measured reflections
1716 independent reflections
1467 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$
$\theta_{\text{max}} = 29.95°$
$h = -12 \rightarrow 12$
$k = -2 \rightarrow 8$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0290$
$wR(F^2) = 0.075$
$S = 1.080$
1716 reflections
112 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.0332P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.239$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.193$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 1.30 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Na—O5	2.4022 (12)	Na···Na^{iii}	4.1430 (13)
Na—O3	2.4054 (8)	O1—C1	1.2540 (10)
Na—O1	2.4063 (11)	O2—C1	1.2576 (11)
Na—O5^i	2.4066 (8)	C1—C2	1.513 (3)
Na—O4^{ii}	2.4397 (8)	C1—C2′	1.542 (2)
Na—O4	2.5584 (9)	C2—C2^{iv}	1.504 (5)
Na···Na^{ii}	3.5377 (9)	C2′—C2′^{iv}	1.533 (5)
Na···Na^i	4.1430 (13)		
O5—Na—O3	95.51 (3)	O1—Na—O4^{ii}	89.27 (3)
O5—Na—O1	159.45 (3)	O5^i—Na—O4^{ii}	83.81 (3)
O3—Na—O1	92.87 (3)	O5—Na—O4	83.66 (3)
O5—Na—O5^i	102.06 (2)	O3—Na—O4	103.98 (3)
O3—Na—O5^i	82.24 (3)	O1—Na—O4	76.12 (3)
O1—Na—O5^i	97.63 (3)	O5^i—Na—O4	171.24 (3)
O5—Na—O4^{ii}	87.10 (3)	O4^{ii}—Na—O4	89.92 (3)
O3—Na—O4^{ii}	166.05 (3)		

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

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^i	0.827 (16)	1.960 (16)	2.7808 (16)	171.7 (16)
O3—H3A···O2^{ii}	0.846 (18)	2.082 (18)	2.9078 (17)	165.1 (16)
O4—H4···O2	0.873 (14)	1.940 (14)	2.7983 (16)	167.3 (15)
O4—H4A···O3^{iii}	0.808 (17)	2.217 (17)	3.0229 (18)	174.8 (16)
O5—H5···O1^{iv}	0.845 (15)	1.937 (14)	2.7703 (16)	168.5 (15)
O5—H5A···O1^v	0.815 (13)	2.051 (13)	2.8627 (16)	174.0 (13)

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

The C2 atom was disordered over two positions, denoted C2 and C2′; an occupancy factor of 0.5 was assigned according to the heights of the difference peaks. The six water H atoms were located from a difference map and refined freely; the positions of the two (half) H atoms of the disordered C2 sites were computed and refined isotropically using a riding model.

Data collection: *CAD-4/PC* (Kretschmar, 1996). Cell refinement: *CAD-4/PC*. Data reduction: *CFEO* (Solans, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1976; Brueggemann & Schmid, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1231). Services for accessing these data are described at the back of the journal.

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