

lengths, 2.026 (2) and 1.944 (2) Å, indicate the probable presence of multiple bonding in both cases. Other distances of this moiety are as expected (Lawton, 1970).

The crystal contains no significantly short intermolecular distances and it is built only by van der Waals forces. The packing in the unit cell, (010) projection, is shown in Fig. 2.

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## Structure of Sodium Succinate Hexahydrate

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**Abstract.**  $\text{Na}_2[\text{C}_4\text{H}_4\text{O}_4] \cdot 6\text{H}_2\text{O}$   $M_r = 270.14$ , triclinic,  $P\bar{1}$ ,  $a = 5.545$  (1),  $b = 6.588$  (4),  $c = 9.020$  (4) Å,  $\alpha = 96.87$  (4),  $\beta = 102.19$  (3),  $\gamma = 109.61$  (3)°,  $V = 296.8$  (3) Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.511$  Mg m<sup>-3</sup>,  $D_m$  not measured,  $\text{Mo } K\alpha$ ,  $\lambda = 0.7108$  Å,  $\mu = 0.196$  mm<sup>-1</sup>, room temperature,  $F(000) = 142$ ,  $R = 0.054$  for 1376 reflections [ $I > 3\sigma(I)$ ] of 2611 total unique data. The succinate ion lies on a centre of symmetry. The  $\text{Na}^+$  is coordinated to six O atoms with distances ranging from 2.346 (4) to 2.563 (4) Å. The succinate ions are linked through the oxygens of water molecules by hydrogen bonds. The crystals are highly hygroscopic and very unstable when exposed to air.

**Introduction.** We have performed a study of some alkaline succinates. Previously, we have reported the structure of potassium succinate trihydrate (Fonseca, Martínez-Carrera & García-Blanco, 1985).

Now, the crystal structure of sodium succinate hexahydrate has been solved.

**Experimental.** Recrystallized from water, a prismatic, colourless, transparent crystal of  $0.17 \times 0.23 \times 0.33$  mm was sealed in a Lindemann capillary tube to inhibit alteration, since the compound is highly hygroscopic. CAD-4 diffractometer; room temperature,  $\text{Mo } K\alpha$  radiation, graphite monochromator; unit-cell dimensions from a least-squares fit of 24 reflections with  $2 < \theta < 35^\circ$ , 1376 observed with  $I > 3\sigma(I)$ ; maximum value of  $2\theta$   $70^\circ$ ; no significant intensity variation observed for two standard reflections ( $3\bar{2}\bar{1}$ ,  $\bar{3}21$ ). Corrections for Lorentz and polarization, absorption ignored. The succinate ion lies on a centre of symmetry. The structure was solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); an *E* map

calculated with the best figure of merit showed six non-hydrogen atoms; two O atoms of water molecules were located in a difference electron density map calculated after a few cycles of isotropic refinement for the atoms already determined. After several cycles of isotropic refinement [ $R(F) = 0.098$ ], anisotropic, full-matrix refinement reduced  $R$  to 0.075; in this step all the H atoms were located from the resulting difference electron density map and introduced in the refinement with isotropic temperature factors [ $B = B(\text{parent carbon})$ ]; subsequent mixed refinement, H fixed, led to a final  $R = 0.054$  ( $wR = 0.059$ ); best agreement obtained with unit weights;  $S = 4.6$ . Largest ratio of shift to e.s.d. 0.003; average shift/e.s.d. ratio 0.0009; final difference electron density map showed the largest positive and negative peaks to be 0.36 and  $-0.57 \text{ e } \text{Å}^{-3}$  respectively. Atomic scattering factors and correction factors for anomalous dispersion from *International Tables for X-ray Crystallography* (1974). No correction for secondary extinction. Computations carried out with programs of the *XRAY* system (Stewart, Kundell & Baldwin, 1970) and *PARST* (Nardelli, 1983) running on the UNIVAC 1100 computer.

Table 1. Atomic parameters for  $\text{Na}[\text{C}_4\text{H}_4\text{O}_4] \cdot 6\text{H}_2\text{O}$

$$U_{\text{eq}} = \left[ \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \cos(a_i, a_j) \right] \times 10^4.$$

	$x$	$y$	$z$	$U_{\text{eq}}(\text{Å}^2)$
Na	0.1675 (3)	0.3610 (3)	0.9003 (2)	213 (6)
O(1)	0.7419 (6)	-0.0506 (5)	0.1977 (3)	244 (11)
O(2)	0.7054 (7)	0.2092 (5)	0.3630 (3)	261 (11)
C(1)	0.8475 (7)	-0.0592 (7)	0.4668 (4)	187 (12)
C(2)	0.7554 (7)	0.0375 (6)	0.3329 (4)	171 (11)
O(3)	0.2085 (6)	0.7539 (5)	1.0047 (3)	238 (10)
O(4)	0.8734 (7)	0.4295 (5)	0.6738 (3)	281 (12)
O(5)	0.4329 (6)	0.4124 (5)	0.1636 (3)	256 (11)

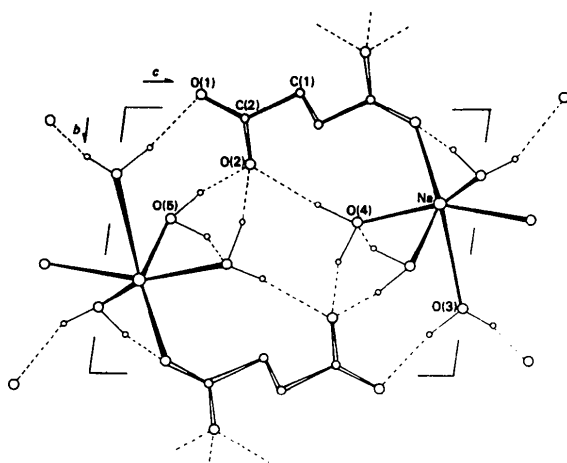


Fig. 1. View of the cell showing the atomic numbering, the  $\text{Na}^+$  coordination and the hydrogen bonding (dashed lines).

**Discussion.** Final coordinates and equivalent isotropic values ( $U_{\text{eq}}$ ) of the anisotropic temperature coefficients are given in Table 1.\* Atomic numbering is shown in Fig. 1. The molecule lies on a centre of symmetry; the carbon chain in the succinate ion is planar and the two O atoms of the carboxylic group deviate out of this plane by 1.10 and  $-1.11 \text{ Å}$  respectively. Table 2 shows the bond lengths and bond angles: the  $\text{C}(1)\text{--}\text{C}(1')$  distance of  $1.549(5) \text{ Å}$  is similar to that found in  $\text{Mn}^{\text{II}}$  succinate tetrahydrate,  $1.54(2) \text{ Å}$  (Gupta, Sahu, Rajaram & Maulik, 1983) but significantly longer than those of potassium succinate trihydrate,  $1.505(8) \text{ Å}$  (Fonseca, Martínez-Carrera & García-Blanco, 1985),  $\text{Cu}^{\text{II}}$  succinate dihydrate,  $1.508(13) \text{ Å}$  (O'Connor & Maslen, 1966), potassium trihydrogen disuccinate,  $1.513(8) \text{ Å}$  (Dunlop & Speakman, 1973)

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42919 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Interatomic distances (Å) and angles ( $^\circ$ ) in the title compound (e.s.d.'s in parentheses)

Succinate ion					
$\text{C}(2)\text{--}\text{O}(1)$	1.262 (5)	$\text{O}(1)\text{--}\text{C}(2)\text{--}\text{O}(2)$	123.3 (4)		
$\text{C}(2)\text{--}\text{O}(2)$	1.265 (5)	$\text{O}(1)\text{--}\text{C}(2)\text{--}\text{C}(1)$	118.8 (4)		
$\text{C}(1)\text{--}\text{C}(2)$	1.506 (6)	$\text{O}(2)\text{--}\text{C}(2)\text{--}\text{C}(1)$	117.8 (3)		
$\text{C}(1)\text{--}\text{C}(1')$	1.549 (5)	$\text{C}(2)\text{--}\text{C}(1)\text{--}\text{C}(1')$	108.9 (3)		
$\text{C}(1)\text{--}\text{H}(11)$	1.10				
$\text{C}(1)\text{--}\text{H}(12)$	0.99				
Environment of the sodium ion					
$\text{Na}\text{--}\text{O}(3^{\text{II}})$	2.563 (4)	$\text{Na}\text{--}\text{O}(5^{\text{III}})$	2.449 (4)		
$\text{Na}\text{--}\text{O}(5^{\text{I}})$	2.432 (3)	$\text{Na}\text{--}\text{O}(1^{\text{IV}})$	2.380 (4)		
$\text{Na}\text{--}\text{O}(4^{\text{II}})$	2.519 (4)	$\text{Na}\text{--}\text{O}(3^{\text{V}})$	2.346 (4)		
$\text{O}(3^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(5^{\text{I}})$	81.1 (1)	$\text{O}(5^{\text{I}})\text{--}\text{Na}\text{--}\text{O}(3^{\text{V}})$	87.2 (1)		
$\text{O}(3^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(4^{\text{II}})$	78.7 (1)	$\text{O}(4^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(5^{\text{III}})$	91.2 (1)		
$\text{O}(3^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(5^{\text{III}})$	77.2 (1)	$\text{O}(4^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(1^{\text{IV}})$	106.2 (1)		
$\text{O}(3^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(1^{\text{IV}})$	163.5 (1)	$\text{O}(4^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(3^{\text{V}})$	86.4 (1)		
$\text{O}(3^{\text{II}})\text{--}\text{Na}\text{--}\text{O}(3^{\text{V}})$	85.9 (1)	$\text{O}(5^{\text{III}})\text{--}\text{Na}\text{--}\text{O}(1^{\text{IV}})$	86.8 (1)		
$\text{O}(5^{\text{I}})\text{--}\text{Na}\text{--}\text{O}(4^{\text{II}})$	159.2 (2)	$\text{O}(5^{\text{III}})\text{--}\text{Na}\text{--}\text{O}(3^{\text{V}})$	163.1 (2)		
$\text{O}(5^{\text{I}})\text{--}\text{Na}\text{--}\text{O}(5^{\text{III}})$	89.3 (1)	$\text{O}(1^{\text{IV}})\text{--}\text{Na}\text{--}\text{O}(3^{\text{V}})$	110.0 (1)		
$\text{O}(5^{\text{I}})\text{--}\text{Na}\text{--}\text{O}(1^{\text{IV}})$	94.6 (1)				
Hydrogen bonds					
$\text{O}(3)\cdots\text{O}(1^{\text{I}})$	2.817 (5)	$\text{O}(3)\text{--}\text{H}(31)$	1.04	$\text{H}(31)\cdots\text{O}(1^{\text{I}})$	1.80
$\text{O}(3)\cdots\text{O}(1^{\text{II}})$	2.858 (4)	$\text{O}(3)\text{--}\text{H}(32)$	1.01	$\text{H}(32)\cdots\text{O}(1^{\text{II}})$	1.89
$\text{O}(4)\cdots\text{O}(2^{\text{III}})$	2.831 (4)	$\text{O}(4)\text{--}\text{H}(41)$	1.07	$\text{H}(41)\cdots\text{O}(2^{\text{III}})$	1.80
$\text{O}(4)\cdots\text{O}(2^{\text{IV}})$	2.801 (4)	$\text{O}(4)\text{--}\text{H}(42)$	1.02	$\text{H}(42)\cdots\text{O}(2^{\text{IV}})$	1.82
$\text{O}(5)\cdots\text{O}(2^{\text{V}})$	2.846 (5)	$\text{O}(5)\text{--}\text{H}(51)$	1.10	$\text{H}(51)\cdots\text{O}(2^{\text{V}})$	1.78
$\text{O}(5)\cdots\text{O}(4^{\text{I}})$	2.827 (6)	$\text{O}(5)\text{--}\text{H}(52)$	1.19	$\text{H}(52)\cdots\text{O}(4^{\text{I}})$	1.79
$\text{O}(3)\text{--}\text{H}(31)\cdots\text{O}(1^{\text{I}})$	165	$\text{O}(4)\text{--}\text{H}(42)\cdots\text{O}(2^{\text{IV}})$	159		
$\text{O}(3)\text{--}\text{H}(32)\cdots\text{O}(1^{\text{II}})$	160	$\text{O}(5)\text{--}\text{H}(51)\cdots\text{O}(2^{\text{V}})$	160		
$\text{O}(4)\text{--}\text{H}(41)\cdots\text{O}(2^{\text{III}})$	161	$\text{O}(5)\text{--}\text{H}(52)\cdots\text{O}(4^{\text{I}})$	143		

Symmetry code: (0)  $x, y, z$ ; (i)  $-x+1, -y+1, -z+1$ ; (ii)  $x, y+1, z+1$ ; (iii)  $-x+2, -y+1, -z+1$ .

and lithium succinate, 1.525 (2) Å (Klapper & Küppers, 1973). The angle O(1)–C(2)–O(2) of 123.3 (4)° and the distances O(1)–C(2), 1.262 (5), and C(2)–O(2), 1.265 (5) Å, show that the two oxygen atoms are both ionized. The crystal structure determination shows that Na<sup>+</sup> is coordinated by six O atoms (Fig. 1), five from the water molecules and one from the succinate ion, with distances ranging from 2.346 (4) to 2.563 (4) Å; the bond distances and angles given in Table 2 show that there is a significant distortion from an ideal octahedron. An extended hydrogen-bond system (Fig. 1, Table 2) is present in the crystal. The succinate ions are linked through the water molecules and the Na<sup>+</sup> ion, giving rise to a strong three-dimensional network.

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## Structure of a Mononuclear Gold(I) Complex Containing a Covalently Bound Ylide Ligand

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**Abstract.** Bromo[methyl(methylene)diphenylphosphoranyl-C]gold(I), [AuBr{P(CH<sub>3</sub>)(CH<sub>2</sub>)(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>}], *M*<sub>r</sub> = 491.1, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 15.746 (4), *b* = 11.930 (4), *c* = 16.564 (5) Å, β = 105.10 (2)°, *V* = 3004 (2) Å<sup>3</sup>, *Z* = 8, *D*<sub>x</sub> = 2.17 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 12.496 mm<sup>-1</sup>, *F*(000) = 1824, *T* = 298 K, *R* = 0.0477 and *wR* = 0.0489 for 3081 reflections with *F*<sub>o</sub><sup>2</sup> > 3σ(*F*<sub>o</sub><sup>2</sup>). The structure of a mononuclear gold(I) complex containing a covalently bound phosphonium ylide ligand is reported. The asymmetric unit contains two crystallographically independent molecules, each consisting of a diphenyldimethylphosphonium ylide linked by a methylene group to an Au<sup>I</sup> center. The Au atoms are two-coordinate linear, with a Br ligand *trans* to the ylide group.

**Introduction.** Ylides have long been regarded as highly versatile and useful synthetic reagents. However, only relatively recently has the coordination chemistry of ylides become well established. The dipolar nature of

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ylides enables these compounds to form exceptionally strong metal–carbon bonds. As a result, organometallic species containing ylide ligands are often quite stable, and examples can currently be found for many of the transition-metal series of elements (Schmidbaur, 1978, 1983; Kaska, 1983).

The gold(I) ylide dimer first reported by Schmidbaur & Franke (1975) has been shown to have an extensive reaction chemistry. In many instances the chemistry parallels that observed in other dimeric complexes containing bridging bidentate phosphine ligands. Examples include two-center two-electron oxidative-addition reactions leading to Au<sup>III</sup> products with discrete metal–metal bonds (Murray, Fackler & Mazany, 1984), as well as the formation of a molecular Au<sup>III</sup> *A*-frame species containing bridging methylene groups (Murray, Mazany & Fackler, 1985). In a limited number of instances isomerization reactions have been observed (Dudis & Fackler, 1985). The predominant reaction products obtained, however, are in general those which do not involve any changes in the basic binuclear configuration.

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