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# Structure of Sodium Hydrogen Succinate at 123 K

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## Abstract

The hydrogen succinate anion has $\bar{1}$ site symmetry and the residues are linked into infinite chains by very short crystallographically symmetric hydrogen bonds with an O···O separation of 2.4277(5) Å. The acidic H atom was best modelled by two equally populated sites on either side of the inversion centre.

## Comment

Acid salts ($MHY$) of dibasic carboxylic acids ($H_2Y$) have been the subject of numerous structural and physical investigations (Speakman, 1972; Hadzi, 1965). The salts classified as type $A_2$ are particularly interesting in view of their very short crystallographically symmetric hydrogen bonds which link carboxyl groups of different anions.

A recent structural study of some alkylammonium salts of the hydrogen succinate anion has suggested that the H atom in the short hydrogen bond is best described by two equally populated sites on either side of the centre of the bond, *i.e.* a double-minimum potential function rather than a single-minimum potential function with the H atom located on the site of $\bar{1}$ symmetry (Kalsbeek & Larson, 1991; Kalsbeek, 1991). This conclusion was reached following consideration of the more reasonable H-atom thermal parameters given by the double-potential-minimum model.

Structure analysis of the title compound (which was shown by IR spectroscopy to be a type $A_2$ salt) was undertaken to check its suitability for a high-resolution charge-density investigation of the symmetric hydrogen-bond environment.

A view of the hydrogen succinate anion with atom labelling is shown in Fig. 1. This anion has $\bar{1}$ symmetry and the sodium cation occupies a site of symmetry 2, as in many other acid salts where the molecular symmetry is dictated by crystallographic symmetry. The bond lengths and angles of the hydrogen succinate residue are in very good agreement with those recently reported for the alkylammonium salts (Kalsbeek & Larson, 1991; Kalsbeek, 1991), and also for the lithium, potassium and caesium salts (Küppers, 1982; McAdam, Currie & Speakman, 1971; McAdam & Speakman, 1971) with which the sodium salt is not isomorphous. Unlike the majority of the succinate salts, the anion deviates markedly from planarity (see Table 2) with C(2$^i$) displaced 1.28 Å from the mean plane defined by O(1), O(2), C(1) and C(2). The C(2$^i$)—C(2)—C(1)—O(1) and C(2$^i$)—C(2)—C(1)—O(2) torsion angles are $-116.3(1)$ and $62.4(1)°$ respectively.

The position of the acidic hydrogen, H(3), was initially fixed at the site of $\bar{1}$ symmetry (1/4, 1/4, 0). Refinement of $U_{iso}$ for this position gave a value of 0.062 (1) Å$^2$. This was considered unreasonable when compared with the
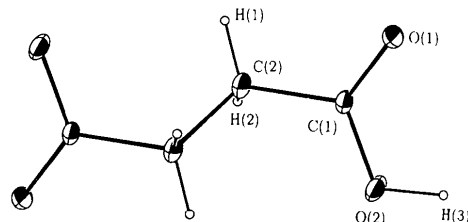


Fig. 1. The molecular structure of the anion showing the atom-labeling scheme. Thermal ellipsoids are shown at the 50% probability level except for the H atoms which are depicted as spheres of arbitrary size.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

|  | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Na(1) | 0.00000 | −0.07148 (2) | 0.25000 | 0.012 |
| O(1) | 0.13470 (4) | 0.08975 (3) | 0.14166 (5) | 0.014 |
| O(2) | 0.29371 (6) | 0.26527 (4) | 0.18122 (5) | 0.027 |
| C(1) | 0.23130 (5) | 0.17590 (4) | 0.25047 (6) | 0.014 |
| C(2) | 0.28231 (6) | 0.18594 (4) | 0.47648 (6) | 0.016 |
| H(1) | 0.2338 (9) | 0.1163 (7) | 0.5143 (11) | 0.014 (2) |
| H(2) | 0.4052 (11) | 0.1819 (8) | 0.5489 (12) | 0.024 (2) |
| H(3) | 0.288 (3) | 0.247 (2) | 0.053 (3) | 0.040 (5) |

Table 2. *Geometric parameters* (Å, °)

| O(1)—C(1) | 1.2353 (5) | C(2)—H(1) | 0.940 (7) |
|---|---|---|---|
| O(2)—C(1) | 1.2897 (6) | C(2)—H(2) | 0.970 (8) |
| O(2)—H(3) | 0.940 (20) | O(2)—O(2$^i$) | 2.4277 (5) |
| C(1)—C(2) | 1.5108 (6) | O(2)—H(3$^i$) | 1.53 (2) |
| C(2)—C(2$^i$) | 1.5349 (6) | H(3)—H(3$^i$) | 0.762 (29) |
| C(1)—O(2)—H(3) | 115.0 (14) | C(1)—C(2)—H(2) | 107.2 (5) |
| O(1)—C(1)—O(2) | 123.30 (4) | C(2$^i$)—C(2)—H(1) | 108.4 (5) |
| O(1)—C(1)—C(2) | 121.96 (4) | C(2$^i$)—C(2)—H(2) | 110.8 (5) |
| O(2)—C(1)—C(2) | 114.73 (4) | H(1)—C(2)—H(2) | 110.9 (7) |
| C(1)—C(2)—C(2$^i$) | 110.55 (3) | O(2)—H(3)—O(2$^i$) | 157.1 (20) |
| C(1)—C(2)—H(1) | 109.0 (5) | | |

| C(2$^i$)—C(2)—C(1)—O(1) | −116.3 (1) | C(2$^i$)—C(2)—C(1)—O(2) | 62.4 (1) |
|---|---|---|---|

Symmetry code: (i) $\tfrac{1}{2} - x, \tfrac{1}{2} - y, -z$.

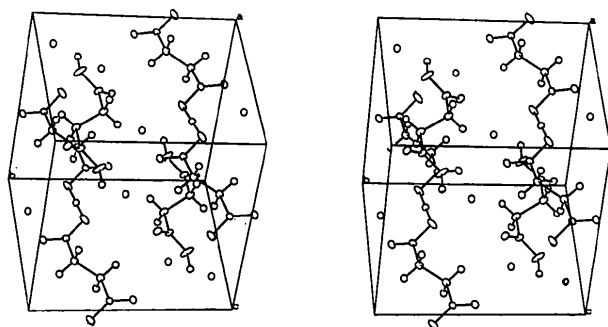

Fig. 2. Stereoview of the unit-cell contents.

mean value of 0.019 Å$^2$ obtained for the methylene protons H(1) and H(2). The model finally adopted for H(3) had two equally populated sites on either side of the inversion centre (see Table 1).

The crystal packing is shown in Fig. 2. The hydrogen succinate anions are linked into infinite chains by a very short crystallographically symmetric hydrogen bond. The sodium cation makes contact with three pairs of equivalent O atoms forming a distorted-octahedral environment.

## Experimental

### Crystal data

Na$^+$.C$_4$H$_5$O$_4^-$
$M_r = 140.07$
Monoclinic
$C2/c$
$a = 8.871$ (1) Å
$b = 10.256$ (1) Å
$c = 7.345$ (1) Å
$\beta = 117.27$ (1)°
$V = 593.9$ (2) Å$^3$
$Z = 4$
$D_x = 1.566$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.1$–$16.9$°
$\mu = 1.89$ cm$^{-1}$
$T = 123$ K
Prism
$0.25 \times 0.25 \times 0.22$ mm
Colourless

### Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega$–$2\theta$ scans
Absorption correction: none
6938 measured reflections
3113 independent reflections
2555 observed reflections
[$I > 2.5\sigma(I)$]

$R_{int} = 0.0119$
$\theta_{max} = 50°$
$h = -19 \rightarrow 19$
$k = 0 \rightarrow 22$
$l = -15 \rightarrow 15$
2 standard reflections
frequency: 60 min
intensity variation: 0.9%

### Refinement

Refinement on $F$
Final $R = 0.0276$
$wR = 0.0363$
$S = 2.29$
2555 reflections
54 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s,
$w = 1/[\sigma^2(F)]$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.49$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986). *GX* (Mallinson & Muir, 1985), which incorporates a locally modified version of *ORTEP* (Johnson, 1971), was used for all other calculations.

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