

## METAL-ORGANIC COMPOUNDS

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**Sodium 3-(2-Chlorophenyl)propenoate Dihydrate and Tetraaquamagnesium Bis[3-(2-hydroxyphenyl)propenoate]**

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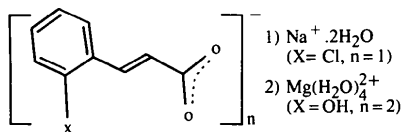
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**Abstract**

In the crystal structures of both  $\text{Na}^+[\text{Cl}-\text{C}_6\text{H}_4-(\text{CH}_2\text{CO}_2)^- \cdot 2\text{H}_2\text{O}]$  and  $\text{Mg}(\text{H}_2\text{O})_4^{2+} \cdot 2[\text{HO}-\text{C}_6\text{H}_4-(\text{CH}_2\text{CO}_2)^-]$ , the coordinated cations are sandwiched between organic layers. Each anion–cation–anion triple layer is held together by electrostatic forces and hydrogen bonds and interacts with others through van der Waals forces.

**Comment**

The structures of (1) and (2) were determined during an investigation of the packing and reactivity of substituted cinnamate anions intercalated in layered double hydroxides (Valim, Kariuki, King & Jones, 1992; Kariuki, Valim, Jones & King, 1994).



The asymmetric unit of the sodium salt (1) consists of an anion, a cation and two molecules of water of crystallization (Fig. 1). Each cation is coordinated to six O atoms (three from water molecules and three from carboxylate groups) which form a distorted octahedron. Ribbons made of octahedra linked by edge sharing run parallel to the *b* axis and are connected through

hydrogen bonding (Fig. 2). In the anion the  $-(\text{CH}_2)-\text{CO}_2$  group is planar and the angle between its plane and the phenyl ring is  $12.19(7)^\circ$ .

The asymmetric unit of the magnesium salt (2) contains one anion, one cation and two molecules of water of hydration. The cations occupy centres of

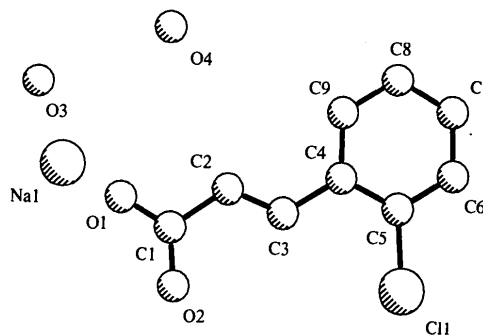


Fig. 1. The asymmetric unit of the sodium salt showing the numbering scheme.

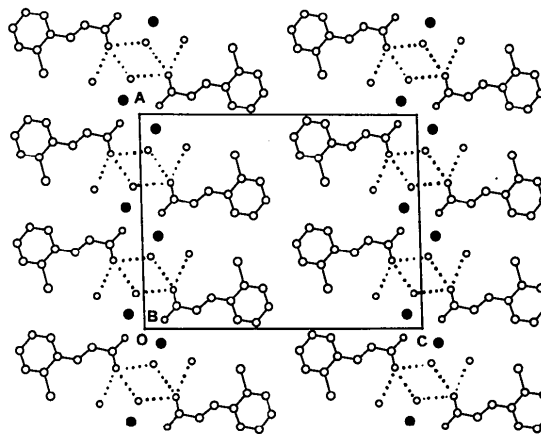


Fig. 2. The structure of the sodium salt viewed down the *b* axis. H atoms have been omitted for clarity and the filled circles represent  $\text{Na}^+$ . The dotted lines represent hydrogen bonds.

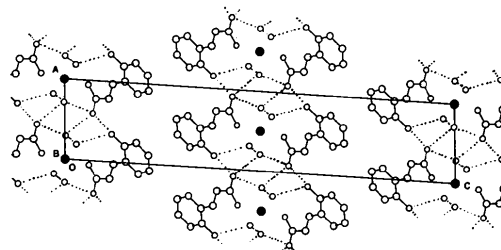


Fig. 3. The structure of the magnesium salt viewed along the *b* axis.

symmetry and each has an octahedral environment composed of four water and two carboxylate O atoms. The octahedra are linked by a network of hydrogen bonds (Fig. 3). The dihedral angle between the planar —(CH<sub>2</sub>)—CO<sub>2</sub> group and the phenyl ring is 13.21 (15)°.

## Experimental

The crystals of both materials were obtained by slow evaporation of aqueous solutions at 295 K.

### Compound (1)

#### Crystal data

Na<sup>+</sup>.C<sub>9</sub>H<sub>6</sub>ClO<sub>2</sub><sup>-</sup>.2H<sub>2</sub>O     Mo *K*α radiation  
*M<sub>r</sub>* = 240.61     λ = 0.71069 Å  
 Monoclinic     Cell parameters from 25  
 P2<sub>1</sub>/a     reflections  
*a* = 14.506 (4) Å     θ = 9–14°  
*b* = 3.7329 (10) Å     μ = 0.397 mm<sup>-1</sup>  
*c* = 19.245 (3) Å     *T* = 293 (2) K  
 β = 91.45 (2)°     Transparent plate  
*V* = 1041.8 (4) Å<sup>3</sup>     0.42 × 0.35 × 0.10 mm  
*Z* = 4     Colourless  
*D<sub>x</sub>* = 1.534 Mg m<sup>-3</sup>

#### Data collection

Enraf–Nonius CAD-4     θ<sub>max</sub> = 26.97°  
 diffractometer     *h* = -18 → 18  
 2θ/ω scans     *k* = 0 → 4  
 Absorption correction:     *l* = 0 → 24  
 none     3 standard reflections  
 2341 measured reflections     monitored every 100  
 2274 independent reflections     reflections  
 1827 observed reflections     frequency: 120 min  
 [*I* > 2σ(*I*)]     intensity decay: none  
*R*<sub>int</sub> = 0.0093

#### Refinement

Refinement on *F*<sup>2</sup>     (Δ/σ)<sub>max</sub> = 0.006  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.0340     Δρ<sub>max</sub> = 0.311 e Å<sup>-3</sup>  
*wR*(*F*<sup>2</sup>) = 0.0943     Δρ<sub>min</sub> = -0.255 e Å<sup>-3</sup>  
*S* = 1.156     Extinction correction: none  
 2272 reflections     Atomic scattering factors  
 160 parameters     from *International Tables*  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0653*P*)<sup>2</sup>     for *Crystallography* (1992),  
 + 0.0713*P*]     Vol. C, Tables 4.2.6.8 and  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3     6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>) for (1)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Na1	0.56684 (4)	0.76807 (15)	0.94412 (3)	0.0269 (2)
Cl1	0.18735 (3)	0.15556 (15)	0.66109 (2)	0.0487 (2)
O1	0.45799 (7)	0.2610 (3)	0.92918 (5)	0.0279 (2)
O2	0.31786 (6)	0.4412 (3)	0.89495 (5)	0.0308 (3)
O3	0.66380 (7)	0.2631 (3)	0.97238 (6)	0.0298 (3)
O4	0.65062 (12)	-0.2016 (7)	0.83462 (8)	0.0630 (5)
C1	0.39427 (9)	0.2972 (4)	0.88415 (7)	0.0229 (3)
C2	0.41375 (10)	0.1610 (4)	0.81293 (7)	0.0314 (3)

C3	0.35600 (10)	0.1888 (4)	0.75961 (7)	0.0273 (3)
C4	0.37091 (10)	0.0568 (4)	0.68890 (7)	0.0270 (3)
C5	0.29868 (11)	0.0284 (4)	0.64000 (7)	0.0315 (3)
C6	0.31178 (13)	-0.0987 (5)	0.57304 (8)	0.0412 (4)
C7	0.3981 (2)	-0.1973 (5)	0.55350 (8)	0.0452 (4)
C8	0.47207 (13)	-0.1732 (5)	0.60029 (9)	0.0422 (4)
C9	0.45814 (11)	-0.0497 (5)	0.66698 (8)	0.0349 (3)

Table 2. Selected geometric parameters (Å, °) for (1)

Na1—O3 <sup>i</sup>	2.3772 (13)	O2—C1	1.254 (2)
Na1—O3	2.4059 (13)	C1—C2	1.496 (2)
Na1—O1 <sup>i</sup>	2.4370 (12)	C2—C3	1.312 (2)
Na1—O4 <sup>i</sup>	2.462 (2)	C3—C4	1.469 (2)
Na1—O1 <sup>ii</sup>	2.4762 (12)	O2...O3 <sup>ii</sup>	2.788 (2)
Na1—O1	2.4770 (13)	O2...O3 <sup>iii</sup>	2.822 (2)
Cl1—C5	1.741 (2)	O2...O4 <sup>iii</sup>	2.835 (2)
O1—C1	1.258 (2)		
O3 <sup>i</sup> —Na1—O3	102.60 (5)	O1 <sup>ii</sup> —Na1—O1	88.43 (4)
O3 <sup>i</sup> —Na1—O1 <sup>i</sup>	79.50 (5)	O2—C1—O1	124.68 (12)
O3—Na1—O1 <sup>i</sup>	172.55 (4)	O2—C1—C2	119.07 (12)
O3 <sup>i</sup> —Na1—O4 <sup>i</sup>	81.84 (7)	O1—C1—C2	116.24 (12)
O3—Na1—O4 <sup>i</sup>	86.05 (6)	C3—C2—C1	123.92 (14)
O3 <sup>i</sup> —Na1—O1 <sup>ii</sup>	84.76 (4)	C2—C3—C4	126.30 (14)
O3—Na1—O1 <sup>ii</sup>	80.89 (4)	C5—C4—C3	121.68 (13)
O1 <sup>i</sup> —Na1—O1 <sup>ii</sup>	92.25 (4)	C9—C4—C3	121.96 (13)
O3 <sup>i</sup> —Na1—O1	172.92 (4)	C6—C5—Cl1	117.27 (12)
O3—Na1—O1	78.17 (5)	C4—C5—Cl1	120.50 (12)
O1 <sup>i</sup> —Na1—O1	98.86 (4)		

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

### Compound (2)

#### Crystal data

Mg(H<sub>2</sub>O)<sub>4</sub><sup>2+</sup>.2C<sub>9</sub>H<sub>7</sub>O<sub>3</sub><sup>-</sup>     Mo *K*α radiation  
*M<sub>r</sub>* = 211.33     λ = 0.71069 Å  
 Monoclinic     Cell parameters from 25  
 P2<sub>1</sub>/*n*     reflections  
*a* = 6.025 (3) Å     θ = 9–12.5°  
*b* = 5.385 (3) Å     μ = 0.147 mm<sup>-1</sup>  
*c* = 29.991 (14) Å     *T* = 293 (2) K  
 β = 94.46 (4)°     Transparent plate  
*V* = 969.9 (9) Å<sup>3</sup>     0.4 × 0.3 × 0.2 mm  
*Z* = 4     Colourless  
*D<sub>x</sub>* = 1.447 Mg m<sup>-3</sup>

#### Data collection

Enraf–Nonius CAD-4     θ<sub>max</sub> = 24.98°  
 diffractometer     *h* = -7 → 7  
 ω scans     *k* = 0 → 6  
 Absorption correction:     *l* = 0 → 35  
 none     2 standard reflections  
 1745 measured reflections     monitored every 100  
 1712 independent reflections     reflections  
 1470 observed reflections     frequency: 60 min  
 [*I* > 2σ(*I*)]     intensity decay: none  
*R*<sub>int</sub> = 0.0647

#### Refinement

Refinement on *F*<sup>2</sup>     (Δ/σ)<sub>max</sub> = 0.004  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.0425     Δρ<sub>max</sub> = 0.321 e Å<sup>-3</sup>  
*wR*(*F*<sup>2</sup>) = 0.1138     Δρ<sub>min</sub> = -0.281 e Å<sup>-3</sup>  
*S* = 1.177     Extinction correction: none  
 1711 reflections     Atomic scattering factors  
 159 parameters     from *International Tables*  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0500*P*)<sup>2</sup>     for *Crystallography* (1992),  
 + 0.9521*P*]     Vol. C, Tables 4.2.6.8 and  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3     6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (2)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^*$$

	x	y	z	$U_{eq}$
Mg1	0	0	0	0.0224 (3)
O1	-0.0551 (3)	-0.1321 (3)	0.06030 (5)	0.0306 (4)
O2	-0.4100 (3)	-0.0685 (3)	0.07154 (6)	0.0354 (4)
O3	0.3435 (3)	-0.7882 (3)	0.11978 (6)	0.0366 (4)
O4	-0.1922 (3)	-0.2839 (3)	-0.02889 (6)	0.0336 (4)
O5	-0.2916 (3)	0.2246 (3)	-0.00191 (7)	0.0287 (4)
C1	-0.2264 (3)	-0.1805 (4)	0.07987 (7)	0.0247 (5)
C2	-0.2154 (4)	-0.3834 (5)	0.11325 (8)	0.0311 (5)
C3	-0.0287 (4)	-0.5042 (4)	0.12352 (7)	0.0259 (5)
C4	0.0128 (4)	-0.7119 (4)	0.15430 (7)	0.0271 (5)
C5	0.2057 (4)	-0.8527 (4)	0.15210 (7)	0.0281 (5)
C6	0.2544 (4)	-1.0472 (5)	0.18146 (9)	0.0375 (6)
C7	0.1084 (5)	-1.1059 (5)	0.21307 (9)	0.0436 (7)
C8	-0.0852 (5)	-0.9716 (5)	0.21513 (9)	0.0460 (7)
C9	-0.1329 (5)	-0.7774 (5)	0.18624 (8)	0.0379 (6)

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (2)

Mg1—O1	1.995 (2)	C2—C3	1.315 (3)
Mg1—O4	2.067 (2)	C3—C4	1.460 (3)
Mg1—O5	2.130 (2)	O2...O5	2.846 (3)
O1—C1	1.253 (3)	O2...O5 <sup>i</sup>	2.777 (3)
O2—C1	1.268 (3)	O2...O3 <sup>ii</sup>	2.628 (3)
O3—C5	1.369 (3)	O3...O4 <sup>iii</sup>	2.833 (3)
C1—C2	1.480 (3)	O4...O5 <sup>iv</sup>	2.845 (3)
O1—Mg1—O4	89.37 (8)	O2—C1—C2	118.5 (2)
O1—Mg1—O5	91.81 (8)	C3—C2—C1	121.0 (2)
O4—Mg1—O5	88.54 (8)	C2—C3—C4	128.9 (2)
O1—C1—O2	123.1 (2)	C9—C4—C3	122.9 (2)
O1—C1—C2	118.4 (2)		

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

All non-H atoms were assigned anisotropic displacement parameters. Water and hydroxyl H atoms were located in difference Fourier synthesis maps while the remainder were placed in calculated positions.

For both compounds, data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *SHELXL92* (Sheldrick, 1992); molecular graphics: *DTMM* (Crabbe & Appleyard, 1991); software used to prepare material for publication: *SHELXL92*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Monosodium L-Glutamate Pentahydrate

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## Abstract

The title compound,  $\text{Na}^+ \cdot \text{C}_5\text{H}_8\text{NO}_4^- \cdot 5\text{H}_2\text{O}$ , crystallizes with two independent chemical units, composed of two L-glutamate anions (Glu A and Glu B), two  $\text{Na}^+$  ions and ten water molecules, per asymmetric unit. Each  $\text{Na}^+$  ion is coordinated by six water molecules in an octahedral arrangement. Strands of coordination octahedra sharing edges with neighbouring octahedra along the *a* axis are linked to one another through hydrogen bonds, forming extended layers parallel to the *ac* plane. Each of the Glu A and Glu B anions also forms hydrogen-bonded layers parallel to the *ac* plane on either side of the  $\text{Na}^+$ -water coordination layer. The crystal packing is dominated by stacking of this triple-layer structure.

## Comment

Monosodium L-glutamate (MSG) pentahydrate was found in 1936 and reported by Ogawa (1949). It crystallizes from a concentrated aqueous solution at a temperature between 264.5 and 272.2 K. At room temperature it transforms immediately to the MSG monohydrate crystal. Compared with the monohydrate crystal, the pentahydrate crystal has a higher selectivity for impurities such as other L-amino acids and colouring substances, *i.e.* these impurities are incorporated less in the pentahydrate crystal than in the monohydrate crystal when crystals are grown from an impure solution. The structure analysis of the pentahydrate crystal (I) was undertaken as a part of an investigation of these phenomena.

