

$S = 1.021$   
 5104 reflections  
 208 parameters  
 H atoms: see below  
 $w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 0.0057P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)  
 Absolute structure:  
 Flack (1983)  
 Flack parameter = 0.040 (12)

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Table 3. Selected geometric parameters (Å, °) for (2)

Pt—N1	2.124 (5)	C11—C12	1.487 (10)
Pt—N2	2.019 (5)	C12—C13	1.467 (9)
Pt—C12	2.108 (5)	O21—C21	1.290 (6)
Pt—O21	2.058 (4)	O23—C23	1.243 (7)
O11—C11	1.222 (8)	C21—C22	1.375 (8)
O13—C13	1.220 (8)	C22—C23	1.436 (8)
C12—Pt—N1	173.7 (2)	Pt—O21—C21	120.5 (3)
C12—Pt—N2	89.8 (2)	O11—C11—C12	122.5 (7)
C12—Pt—O21	94.6 (2)	O13—C13—C12	122.3 (7)
O21—Pt—N1	91.5 (2)	C11—C12—C13	115.1 (6)
O21—Pt—N2	175.4 (2)	O21—C21—C22	124.2 (5)
N1—Pt—N2	84.1 (2)	O23—C23—C22	123.1 (6)
Pt—C12—C13	109.5 (4)	C21—C22—C23	123.2 (6)
Pt—C12—C11	104.5 (4)		
Pt—O21—C21—C22	−6.2 (8)		

Table 4. Hydrogen-bonding geometry (Å, °) for (2)

D—H...A	D—H	H...A	D...A	D—H...A
N2—H5...O23 <sup>i</sup>	0.90	2.16	2.985 (7)	152
N2—H6...O23 <sup>ii</sup>	0.90	2.20	3.046 (7)	157

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

For both compounds, all H atoms other than those of water were included as riding and the positions of the water H atoms in (1) were refined with O—H constrained to 0.82 (3) Å;  $U_{iso}(H) = 1.2U_{eq}(\text{parent atom})$ . A disordered conformation was observed in (1) for the enolate chdo<sup>−</sup> and the site-occupancy factors of C25 and C251 were refined to 0.65 (2) and 0.35 (2), respectively. Each member of the Friedel pairs in (2) was treated as an independent observation in the least-squares refinement.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993a); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993b); program(s) used to solve structures: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Mr Takahiro Kishi for his help in the preparative work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1013). Services for accessing these data are described at the back of the journal.

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## Sodium hydrogen *trans*-glutaconate and ammonium hydrogen *trans*-glutaconate

JUN-ICHIRO TAKA† AND SETSUO KASHINO

*Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700-8530, Japan. E-mail: kashinos@cc.okayama-u.ac.jp*

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## Abstract

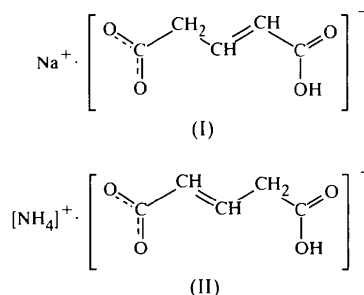
Sodium hydrogen *trans*-glutaconate (sodium hydrogen *trans*-pent-2-ene-1,5-dioate, Na<sup>+</sup>·C<sub>5</sub>H<sub>5</sub>O<sub>4</sub><sup>−</sup>), (I), crystallizes in the monoclinic space group *P2<sub>1</sub>/n* with  $Z = 4$ , and ammonium hydrogen *trans*-glutaconate (ammonium hydrogen *trans*-pent-2-ene-1,5-dioate, NH<sub>4</sub><sup>+</sup>·C<sub>5</sub>H<sub>5</sub>O<sub>4</sub><sup>−</sup>), (II), crystallizes in the orthorhombic space group *Ibam* with  $Z = 8$ . In both crystals, a non-centrosymmetric O—H...O hydrogen bond is formed between the carboxyl and carboxylate groups of neighboring anions. The Na<sup>+</sup> cation in (I) is coordinated by six O atoms. There are two symmetry-independent NH<sub>4</sub><sup>+</sup> cations in (II). The number of hydrogen bonds around each NH<sub>4</sub><sup>+</sup> cation is four.

## Comment

The crystal structures of the title compounds, sodium hydrogen *trans*-glutaconate, (I), and ammonium hydrogen *trans*-glutaconate, (II), have been determined as part of a detailed investigation of H-atom bonding behavior in short asymmetric O—H...O hydrogen bonds in

† Present address: Institute for Protein Research, Osaka University, 3-2 Yamadaoka, Suita, Osaka 565-0871, Japan.

the acid salts of carboxylic acids (Taka *et al.*, 1998). Because a molecule of *trans*-glutaconic acid has only mirror symmetry, it was expected that the acid salts of *trans*-glutaconic acid might form asymmetric O—H...O hydrogen bonds.



In (I), the ionized carboxylate group is attached to C3, while in (II), it is attached to C1 (Fig. 1). Although the structure of (II), built up only from the non-H atoms, has

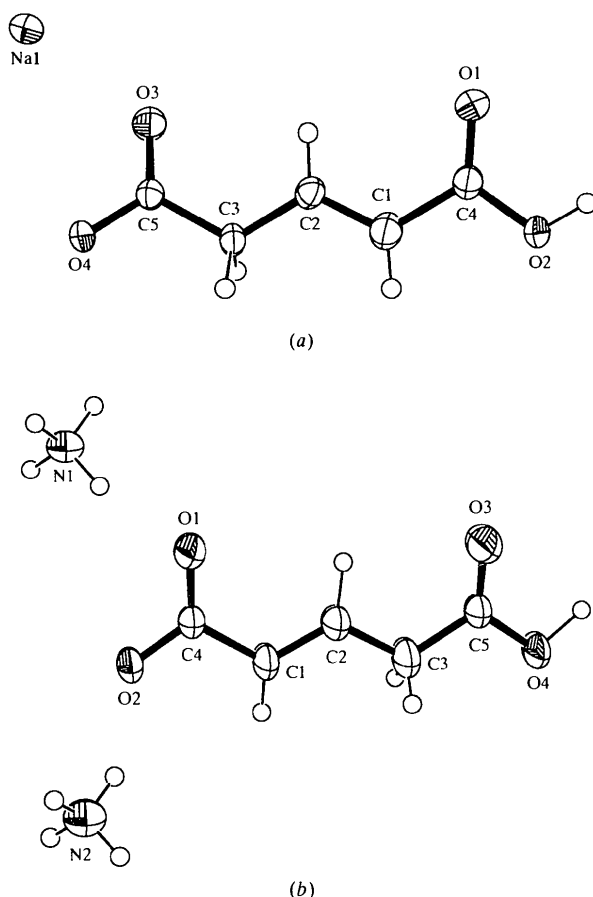


Fig. 1. ORTEP-3 (Farrugia, 1997) representations of (a) (I) and (b) (II), with the atomic numbering of non-H atoms. Displacement ellipsoids are shown at the 50% probability level for non-H atoms and H atoms are drawn as spheres of arbitrary radii.

non-space-group translation  $(\frac{1}{2}, 0, 0)$ , space group *Ibam* and the setting of the crystal axes are correct when the H atoms are taken into consideration. The hydrogen *trans*-glutaconate anion in (II) has mirror symmetry. In this anion, only the H atom attached to C3 occupies the general position 16k, and the other atoms of the anion occupy special position 8j. Two symmetrically independent N atoms occupy special positions 4a and 4b, and H atoms of the ammonium cations occupy a general position (Fig. 2).

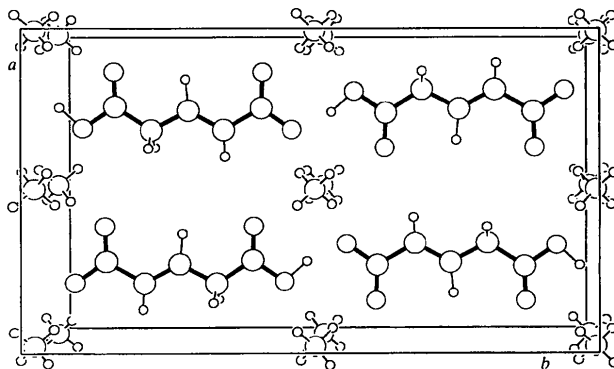


Fig. 2. The crystal structure of (II) viewed down the *c* axis. The anions at  $z = \frac{1}{2}$  are drawn for clarity.

In both crystals, a chain of anions is formed along [010] through O—H...O hydrogen bonds. In the chains, the anions are arranged head-to-tail. Thus, the hydrogen bonds are not centrosymmetric. The O...O distances [2.485 (3) Å in (I) and 2.513 (3) Å in (II)] are longer than the distance of 2.443 (2) Å found in ammonium hydrogen tartronate (Taka *et al.*, 1998). Disorder was not found for the H atoms involved in the O—H...O hydrogen bonds in (I) and (II) in contrast with the case of ammonium hydrogen tartronate. The Na<sup>+</sup> cation in (I) is coordinated by six O atoms. Each NH<sub>4</sub><sup>+</sup> cation in (II) forms four hydrogen bonds, one of which is symmetrically independent.

## Experimental

Crystals of (I) were grown by slow evaporation from an aqueous solution of *trans*-glutaconic acid (Aldrich) and sodium carbonate in a 2:1 molar ratio. Crystals of (II) were grown by slow evaporation from an aqueous solution of *trans*-glutaconic acid and ammonia in a 1:1 molar ratio.

## Compound (I)

### Crystal data

Na<sup>+</sup>·C<sub>5</sub>H<sub>5</sub>O<sub>4</sub><sup>-</sup>  
M<sub>r</sub> = 152.082

Mo Kα radiation  
λ = 0.71073 Å

Monoclinic  
*P*<sub>2</sub><sub>1</sub>/*n*  
*a* = 5.290 (4) Å  
*b* = 18.214 (6) Å  
*c* = 6.781 (3) Å  
 $\beta$  = 111.66 (4)°  
*V* = 607.2 (1) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.664 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

#### Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 1543 measured reflections  
 1402 independent reflections  
 993 reflections with  
 $I > \sigma(I)$

#### Refinement

Refinement on *F*  
*R* = 0.069  
 $\omega R$  = 0.060  
*S* = 1.14  
 993 reflections  
 111 parameters  
 All H-atom parameters refined

Cell parameters from 25 reflections  
 $\theta$  = 9.5–11.0°  
 $\mu$  = 0.202 mm<sup>-1</sup>  
*T* = 295 K  
 Plate  
 0.50 × 0.18 × 0.08 mm  
 Colorless

*R*<sub>int</sub> = 0.041  
 $\theta_{\max}$  = 27.5°  
 $h = 0 \rightarrow 6$   
 $k = 0 \rightarrow 23$   
 $l = -8 \rightarrow 8$   
 3 standard reflections every 97 reflections  
 intensity decay: none

$w = 1/\sigma^2(F)$   
 $(\Delta/\sigma)_{\max} = 0.014$   
 $\Delta\rho_{\max} = 0.35 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.35 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Na1—O2 <sup>i</sup>	2.326 (3)	O2—C4	1.303 (4)
Na1—O4 <sup>ii</sup>	2.370 (3)	O3—C5	1.234 (4)
Na1—O3 <sup>iii</sup>	2.462 (3)	O4—C5	1.286 (4)
Na1—O3	2.474 (3)	C1—C2	1.360 (5)
Na1—O1 <sup>iv</sup>	2.507 (3)	C1—C4	1.479 (5)
Na1—O4 <sup>v</sup>	2.736 (3)	C2—C3	1.450 (5)
O1—C4	1.210 (4)	C3—C5	1.495 (5)
C2—C1—C4	122.0 (4)	O2—C4—C1	111.5 (3)
C1—C2—C3	121.7 (3)	O3—C5—O4	123.9 (3)
C2—C3—C5	118.8 (3)	O3—C5—C3	121.7 (3)
O1—C4—O2	123.1 (3)	O4—C5—C3	114.4 (3)
O1—C4—C1	125.4 (3)		

Symmetry codes: (i)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (ii)  $1 + x, y, z$ ; (iii)  $1 - x, 1 - y, -z$ ; (iv)  $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (v)  $1 - x, 1 - y, 1 - z$ .

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

D—H...A	D—H	H...A	D...A	D—H...A
O2—H2O...O4 <sup>i</sup>	0.88 (4)	1.61 (4)	2.485 (3)	173 (4)

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

#### Compound (II)

##### Crystal data

NH<sub>4</sub><sup>+</sup>·C<sub>5</sub>H<sub>5</sub>O<sub>4</sub><sup>-</sup>  
*M<sub>r</sub>* = 147.13

Mo *K*α radiation  
 $\lambda = 0.71073 \text{ Å}$

Orthorhombic  
*Ibam*  
*a* = 10.669 (5) Å  
*b* = 18.479 (6) Å  
*c* = 7.280 (6) Å  
*V* = 1435 (2) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.362 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

#### Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 1662 measured reflections  
 1476 independent reflections  
 888 reflections with  
 $I > \sigma(I)$

#### Refinement

Refinement on *F*  
*R* = 0.077  
 $\omega R$  = 0.067  
*S* = 1.51  
 888 reflections  
 82 parameters  
 All H-atom parameters refined

Cell parameters from 25 reflections  
 $\theta$  = 9.5–12.0°  
 $\mu$  = 0.119 mm<sup>-1</sup>  
*T* = 295 K  
 Prismatic  
 0.50 × 0.30 × 0.18 mm  
 Colorless

*R*<sub>int</sub> = 0.012  
 $\theta_{\max}$  = 27.5°  
 $h = 0 \rightarrow 16$   
 $k = 0 \rightarrow 27$   
 $l = -1 \rightarrow 10$   
 3 standard reflections every 97 reflections  
 intensity decay: none

$w = 1/\sigma^2(F)$   
 $(\Delta/\sigma)_{\max} = 0.008$   
 $\Delta\rho_{\max} = 0.33 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.36 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 3. Selected geometric parameters (Å, °) for (II)

O1—C4	1.240 (3)	C1—C2	1.374 (4)
O2—C4	1.280 (3)	C1—C4	1.496 (4)
O3—C5	1.202 (4)	C2—C3	1.430 (4)
O4—C5	1.314 (3)	C3—C5	1.479 (4)
C2—C1—C4	120.5 (3)	O2—C4—C1	114.0 (3)
C1—C2—C3	122.6 (3)	O3—C5—O4	122.8 (3)
C2—C3—C5	118.2 (3)	O3—C5—C3	125.4 (3)
O1—C4—O2	123.4 (2)	O4—C5—C3	111.8 (3)
O1—C4—C1	122.6 (2)		

Table 4. Hydrogen-bonding geometry (Å, °) for (II)

D—H...A	D—H	H...A	D...A	D—H...A
O4—H4O...O2 <sup>i</sup>	1.05 (4)	1.50 (4)	2.513 (3)	161 (3)
N1—H1N...O1	0.98 (2)	2.02 (2)	2.986 (2)	168 (2)
N2—H2N...O2	0.88 (2)	2.04 (3)	2.848 (2)	153 (2)

Symmetry code: (i)  $\frac{3}{2} - x, \frac{1}{2} + y, 1 - z$ .

For both compounds, data collection: RASAIL (Rigaku Corporation, 1990); cell refinement: RASAIL; program(s) used to solve structures: SIR (Burla *et al.*, 1989); program(s) used to refine structures: TEXSAN (Molecular Structure Corporation, 1985); molecular graphics: ORTEP-3 (Farrugia, 1997).

This work was supported in part by a Grant-in-Aid for Scientific Research (No. 09640609) from the Ministry of Education, Science, Sports and Culture, Japan.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1012). Services for accessing these data are described at the back of the journal.

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### A linear-chain silver(I) coordination polymer with tetrakis(isopropylthio)-*p*-benzoquinone

YUSAKU SUENAGA,<sup>a</sup> TAKAYOSHI KURODA-SOWA,<sup>a</sup>  
MASAHIKO MAEKAWA<sup>b</sup> AND MEGUMU MUNAKATA<sup>a</sup>

<sup>a</sup>Department of Chemistry, Kinki University 3-4-1, Kowakae, Higashi-Osaka, Osaka 577-8502, Japan, and <sup>b</sup>Research Institute for Science and Technology, Kinki University, 3-4-1 Kowakae, Higashi-Osaka, Osaka 577-8502, Japan. E-mail: suenagay@chem.kindai.ac.jp

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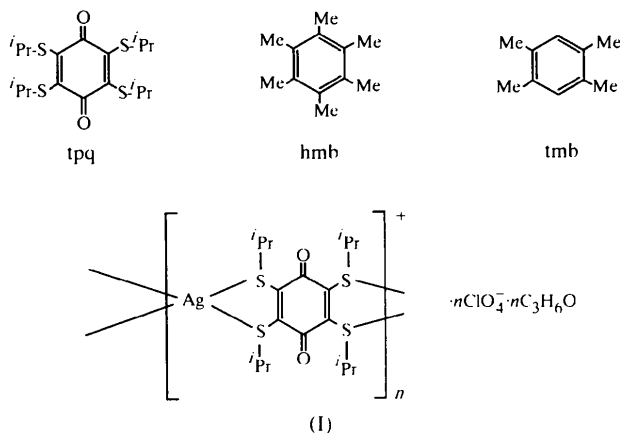
## Abstract

The silver(I) complex of tetrakis(isopropylthio)-*p*-benzoquinone (tpq), *catena*-poly[silver(I)- $\mu$ -[2,3,5,6-tetrakis(isopropylthio)-*p*-benzoquinone - S<sup>2</sup>,S<sup>3</sup>:S<sup>5</sup>,S<sup>6</sup>]] perchlorate acetone solvate, {[Ag(C<sub>18</sub>H<sub>28</sub>S<sub>4</sub>O<sub>2</sub>)]ClO<sub>4</sub>·nC<sub>3</sub>H<sub>6</sub>O}<sub>n</sub>, has been prepared and the molecular structure determined. The silver ion prefers a tetrahedral coordination geometry comprising four S atoms from two different tpq molecules, giving a linear-chain structure of alternating metal cations and organic ligands. Also, the four isopropyl groups of the tpq molecule lie in the perpendicular direction of the plane of the benzoquinone ring.

## Comment

Many metal–dithiolene complexes have been synthesized and studied (McCleverty, 1968; Eisenberg, 1970; Rivera & Engler, 1979; Munakata *et al.*, 1995). We synthesized aromatic multi-alkyl thioether ligands and have recently reported the formation of a metal–complex polymer with aromatic multi-alkyl thioether ligands. We have been able to use the metal–complex polymer to construct linear chain [Cu(hmb)]PF<sub>6</sub> [hmb is hexakis(methylthio)benzene; Suenaga *et al.*, 1998], zigzag

chain [(CuI)<sub>2</sub>(tmb)] [tmb is 1,2,4,5-tetrakis(methylthio)benzene; Ohruai *et al.*, 1996], two-dimensional [(CuBr)<sub>2</sub>(tmb)] sheets (Suenaga *et al.*, 1997) and a two-dimensional [Ag(hmb)]PF<sub>6</sub> network with inter-chain S··S contacts (Suenaga *et al.*, 1999). We prepared tetrakis(isopropylthio)-*p*-benzoquinone (tpq) for use in place of hmb as an acceptor organic compound. We report here the polymeric structure of [Ag(tpq)](ClO<sub>4</sub>)-acetone, (I), as part of our studies on two and three-dimensional polymers of metal ions interconnected by aromatic ligands with novel stereochemistry and special physical properties.



The Ag<sup>I</sup> atom lies on the twofold axis along **a**. The C11, O3, and O4 atoms of the perchlorate ion, the O1, C2, O2 and C4 atoms of tpq, and the O6 and C11 atoms of acetone lie on mirror planes perpendicular to **c**. Each Ag<sup>I</sup> atom is coordinated by four S atoms from two tpq ligands in a distorted tetrahedral geometry, with bond angles around silver ranging from 81.25 (7) to 151.5 (1)°. The Ag—S distances of 2.521 (2) and 2.601 (2) Å are comparable to those in the complexes of hmb with silver(I) hexafluorophosphate [2.565 (2) Å]. Four isopropyl groups are perpendicular to the benzoquinone ring plane. Interestingly, all the isopropyl groups are directed to the same side of the S—Ag<sup>I</sup>—S plane. The suggested classification for the description of possible ‘leg’ orientations in spider host molecules comprises 14 types (MacNicol *et al.*, 1985); descriptors *a* and *b* denote side-chain hydrocarbon moieties projecting, respectively, above and below the mean plane of the benzene core, *e.g.* the conformation of hexakis(phenyloxy)benzene and hexakis(phenylthio)benzene, with regular alternation of pairs of legs, is *ababab*. In contrast, complex (I) exhibits the previously unknown type *aaaa* conformation. In this complex, the O atom of the acetone solvent molecule is positioned on the same side of the four isopropyl groups. This stereospecific orientation may occur because of dipole–dipole interactions. Thus, one tpq bridges two Ag atoms