

the dimer $[81.8(1)^\circ]$. Consequently, the bent structure of the Ni_2S_2 unit in $[\text{Ni}(\text{SET})_2]_6$ is contracted and collapsed to its inside hole. The resulting short $\text{Ni}\cdots\text{Ni}$ distances, 2.931 (1) (triclinic) and 2.923 (1) Å (monoclinic), should be compared with the distance of 3.356 (2) Å in $[\text{Ni}_2(\text{SET})_6]^{2-}$.

The packing modes of the hexamers in the crystals are depicted in Fig. 2. The hexamer rings stack parallel in the triclinic form while they are packed so that the pseudo-hexagonal axis of the molecule is almost perpendicular with each in the monoclinic form. The molecular planes incline at *ca* 40° with respect to the 2_1 axis.

One of the axial methyl groups in the triclinic form, which is directed towards the center of the adjacent ring, is allowed to be situated in two sites (Fig. 2*a*). The methyl group restraint by the adjacent hexamer in the triclinic crystal does not seem to be enough to fix the alkyl configuration, presumably owing to the large vacancy of the tiara ring. Nevertheless, the density of the triclinic crystal is slightly larger than that of the monoclinic one.

We gratefully thank Professor Yukiyoshi Sasaki of The University of Tokyo for his kind support.

Acta Cryst. (1988). C44, 609–611

Structure of Potassium (Rubidium) Trihydrogen Diglutarate

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(Received 29 September 1987; accepted 2 December 1987)

Abstract. $\text{KH}_3(\text{C}_5\text{H}_6\text{O}_4)_2$, $M_r = 302.3$, monoclinic, $P2_1/a$, $a = 9.392(2)$, $b = 12.782(1)$, $c = 11.147(2)$ Å, $\beta = 93.34(2)^\circ$, $V = 1335.9$ Å³, $Z = 4$, $D_m = 1.46$, $D_x = 1.50$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 38.4$ cm⁻¹, $F(000) = 632$, $T = 291$ K, final $R = 0.049$ for 1866 independent observed reflections. $\text{RbH}_3(\text{C}_5\text{H}_6\text{O}_4)_2$, $M_r = 348.7$, monoclinic, $P2_1/a$, $a = 9.232$, $b = 13.306$, $c = 11.134$ Å, $\beta = 94.4^\circ$, $V = 1363.7$ Å³, $Z = 4$, $D_m = 1.64$, $D_x = 1.698$ g cm⁻³, $F(000) = 704$. The potassium and rubidium salts are isomorphous. Isomorphism of the two salts was established photographically and, as the structure of the K salt was easily determined, that of the Rb salt was not investigated.

HY^- anions ($\text{H}_2\text{Y} = \text{glutaric acid}$) are linked into infinite spiral chains by short asymmetric hydrogen bonds [$\text{O}\cdots\text{O} = 2.506(4)$ Å]. These chains are cross-linked through longer hydrogen bonds [$\text{O}\cdots\text{O} = 2.553(3)$ and $2.641(4)$ Å] to glutaric acid molecules. K^+ is eight-coordinate with $\text{K}^+\cdots\text{O}$ in the range $2.727\text{--}3.010(3)$ Å.

Introduction. In addition to their normal acid salts (MHY), dicarboxylic acids (H_2Y) form 'super-acid' salts of general formula (MHYH_2Y) where M is a monovalent cation (Marshall & Cameron, 1907). The crystal structures of two such salts of formula $\text{KH}_3(\text{CH}_2)_n(\text{CO}_2)_2$, $n = 1, 2$ have been determined using neutron diffraction data (Currie, 1972; Dunlop &

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Speakman, 1973). We report here the results of an X-ray analysis of the salt with $n = 3$. Brief details of the structure have been reported elsewhere (Speakman, 1972).

Table 1. Final positional parameters and equivalent isotropic thermal parameters

$$U_{eq} = \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
K	0.43446 (8)	0.41853 (6)	0.12108 (6)	0.042
O(1)	0.8264 (3)	0.3000 (2)	0.0726 (2)	0.061
O(2)	0.6405 (3)	0.2954 (2)	0.2307 (2)	0.046
O(3)	0.7095 (3)	0.1317 (2)	0.2592 (2)	0.049
O(4)	0.7873 (3)	0.4780 (2)	0.6011 (2)	0.053
O(5)	0.5718 (2)	0.4496 (2)	0.6607 (2)	0.050
O(6)	0.3011 (3)	0.3983 (2)	0.6760 (3)	0.077
O(7)	0.3196 (3)	0.4316 (3)	0.8700 (3)	0.072
O(8)	-0.3418 (3)	0.3848 (3)	0.9660 (2)	0.059
C(1)	0.6362 (3)	0.2100 (3)	0.2861 (3)	0.037
C(2)	0.5368 (4)	0.2017 (3)	0.3873 (3)	0.047
C(3)	0.5254 (4)	0.2994 (3)	0.4630 (3)	0.042
C(4)	0.6683 (4)	0.3347 (3)	0.5152 (3)	0.046
C(5)	0.6686 (3)	0.4274 (3)	0.5987 (3)	0.037
C(6)	0.2500 (4)	0.4060 (3)	0.7821 (4)	0.052
C(7)	0.0974 (4)	0.3728 (5)	0.7773 (4)	0.082
C(8)	0.0193 (4)	0.3798 (4)	0.8912 (4)	0.057
C(9)	-0.1275 (5)	0.3515 (6)	0.8757 (4)	0.098
C(10)	-0.2250 (4)	0.3489 (3)	0.9756 (3)	0.049

Table 2. Selected interatomic distances (\AA) and angles ($^\circ$)

K—O(2)	2.727 (3)	K—O(1')	3.010 (3)
K—O(5 ⁱⁱ)	2.963 (3)	K—O(3)	2.762 (3)
K—O(7 ⁱⁱⁱ)	2.999 (4)	K—O(7 ⁱⁱⁱ)	2.945 (3)
K—O(8 ^{iv})	2.814 (3)	K—O(8 ^{iv})	2.831 (3)
O(2)—C(1)	1.255 (5)	O(1')—C(10)	1.316 (5)
O(4)—C(5)	1.288 (5)	O(3)—C(1)	1.261 (5)
O(6)—C(6)	1.306 (5)	O(5)—C(5)	1.208 (4)
O(8)—C(10)	1.188 (5)	O(7)—C(6)	1.191 (5)
C(2)—C(3)	1.514 (5)	C(1)—C(2)	1.510 (5)
C(4)—C(5)	1.506 (5)	C(3)—C(4)	1.500 (5)
C(7)—C(8)	1.506 (6)	C(6)—C(7)	1.493 (6)
C(9)—C(10)	1.482 (6)	C(8)—C(9)	1.427 (7)

O(1)—K—O(2)	76.6 (1)	O(1)—K—O(3')	67.9 (1)
O(1)—K—O(5 ⁱⁱ)	130.8 (1)	O(1)—K—O(7 ⁱⁱⁱ)	77.3 (1)
O(1)—K—O(7 ⁱⁱⁱ)	148.2 (1)	O(1)—K—O(8 ^{iv})	90.2 (1)
O(1)—K—O(8 ^{iv})	132.3 (1)	O(2)—K—O(3')	99.3 (1)
O(2)—K—O(5 ⁱⁱ)	90.5 (1)	O(2)—K—O(7 ⁱⁱⁱ)	132.0 (1)
O(2)—K—O(7 ⁱⁱⁱ)	80.1 (1)	O(2)—K—O(8 ^{iv})	69.7 (1)
O(2)—K—O(8 ^{iv})	150.1 (1)	O(3)—K—O(5 ⁱⁱ)	67.8 (1)
O(3)—K—O(7 ⁱⁱⁱ)	107.1 (1)	O(3)—K—O(7 ⁱⁱⁱ)	138.0 (1)
O(3)—K—O(8 ^{iv})	157.6 (1)	O(3)—K—O(8 ^{iv})	99.7 (1)
O(5 ⁱⁱ)—K—O(7 ⁱⁱⁱ)	136.4 (1)	O(5 ⁱⁱ)—K—O(7 ⁱⁱⁱ)	70.2 (1)
O(5 ⁱⁱ)—K—O(8 ^{iv})	129.7 (1)	O(5 ⁱⁱ)—K—O(8 ^{iv})	75.8 (1)
O(7 ⁱⁱⁱ)—K—O(7 ⁱⁱⁱ)	103.6 (1)	O(7 ⁱⁱⁱ)—K—O(8 ^{iv})	70.9 (1)
O(7 ⁱⁱⁱ)—K—O(8 ^{iv})	62.2 (1)	O(7 ⁱⁱⁱ)—K—O(8 ^{iv})	61.3 (1)
O(7 ⁱⁱⁱ)—K—O(8 ^{iv})	70.3 (1)	O(8 ^{iv})—K—O(8 ^{iv})	98.7 (1)
O(2)—C(1)—O(3)	122.7 (3)	O(2)—C(1)—C(2)	117.9 (3)
O(3)—C(1)—C(2)	119.4 (3)	C(1)—C(2)—C(3)	115.1 (3)
C(2)—C(3)—C(4)	111.9 (3)	C(3)—C(4)—C(5)	116.5 (3)
O(4)—C(5)—O(5)	123.4 (4)	O(4)—C(5)—C(4)	112.2 (3)
O(5)—C(5)—C(4)	124.3 (3)	O(6)—C(6)—O(7)	123.6 (4)
O(6)—C(6)—C(7)	110.4 (4)	O(7)—C(6)—C(7)	126.0 (4)
C(6)—C(7)—C(8)	117.8 (4)	C(7)—C(8)—C(9)	113.4 (4)
C(8)—C(9)—C(10)	123.4 (4)	O(1)—C(10)—O(8)	123.3 (4)
O(1')—C(10)—C(9)	114.5 (4)	O(8)—C(10)—C(9)	122.1 (4)

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

Experimental. Prepared by dissolving glutaric acid and potassium hydroxide (rubidium carbonate) in hot water, 2:1 (2:0.5) mol. Large colourless plates separated on slow evaporation elongated along a with (010) and (0 $\bar{1}$ 0) prominent. For the rubidium salt, crystals of the normal acid salt obtained initially redissolve on standing to afford super-acid salt crystals. Crystal ca $0.5 \times 0.4 \times 0.2$ mm used in data collection, CAD-4 diffractometer, Weissenberg photographs indicated monoclinic, $P2_1/a$. Density by flotation in methyl iodide/chlorobenzene, 2529 independent intensities, θ limit 70° , $\omega/2\theta$ scan. Two standard intensities used to monitor variations in intensity data: $<2\%$ variation observed. Least-squares technique based on 16 reflections, $\theta > 20^\circ$, used to refine lattice parameters. h 0 to 11, k 0 to 15, l -13 to 13. Structure solved by Patterson and Fourier methods. Empirical absorption corrections applied (Walker & Stuart, 1983) with $T_{max}/T_{min} = 1.71/0.69$. Full-matrix least-squares refinement on F of coordinates and anisotropic thermal

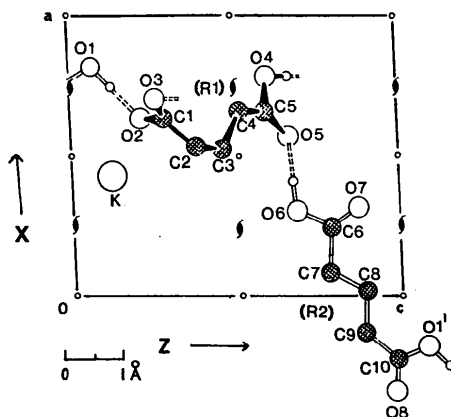


Fig. 1. The atom-numbering scheme in $\text{KH}_3(\text{C}_5\text{O}_4\text{H}_6)_2$. (O1' is at the equivalent position $-1+x, y, 1+z$.)

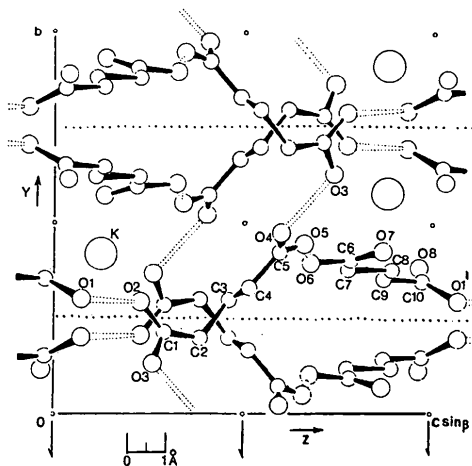


Fig. 2. The contents of the unit cell viewed along a (methylene hydrogens omitted for clarity).

parameters for all non-H atoms converged to R and wR of 0.049 and 0.067 with $w = 1/\sigma^2(F_o)$. H-atom coordinates, located from difference Fourier maps, included in structure-factor calculations, but not refined. 1866 reflections, $I > 3.0\sigma_p$, used. $\Delta_{\max}/\sigma = 0.09$; max. and min. heights in final difference Fourier synthesis = 0.34 and $-0.47 e \text{ \AA}^{-3}$. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using the *GX* package (Mallinson & Muir, 1985).

Discussion. Final positional and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths, bond angles and pertinent intermolecular contacts are given in Table 2. Fig. 1 shows the atom-numbering scheme and Fig. 2 the contents of the unit cell along a .

The crystal contains two different types of glutarate residue (Fig. 1) – half-acid anions (R_1) and glutaric acid molecules (R_2). These can be differentiated by the location of the H atoms attached to O(1), O(4) and O(6) and by the dimensions of their CO_2^- groups (Table 2). The carbon skeleton of the glutaric acid molecule is substantially planar (r.m.s. deviation 0.014 \AA) with the carboxyl groups inclined at 3.4° [C(6), O(6), O(7)] and 34.5° [C(10), O(1), O(8)] to the plane of the five carbon atoms. Atoms C(2), C(3), C(4), C(5) also approach coplanarity. The structure contains pairs of centrosymmetrically-related antiparallel helices formed from the end-to-end linking of R_1 residues into infinite

chains by short asymmetric hydrogen bonds, O(4)···O(3) $2.506(4) \text{ \AA}$. These chains spiral around the twofold screw axes at $\frac{1}{2}y\frac{1}{2}$ and $\frac{3}{2}y\frac{1}{2}$. In this respect the structure is that of a type B_2 acid salt (Currie & Speakman, 1970). Cross-linking of helices which are related by the translation ($\bar{1}01$) occurs through hydrogen bonding [O(5)···O(6) $2.641(4)$ and O(1)···O(2) $2.553(3) \text{ \AA}$] to the glutaric acid molecules which are arranged in ribbons along the edges of the unit cells (Fig. 2). By contrast $\text{KH}_3(\text{CH}_2)(\text{CO}_2)_2$ (Currie, 1972) has planar HY^- residues linked into rings interconnected by H_2Y molecules while $\text{KH}_3(\text{CH}_2)_2(\text{CO}_2)_2$ (Dunlop & Speakman, 1973) has succinic acid molecules 'festooned' along the B_2 chains. The pattern in $\text{LiH}_3(\text{CH}_2)(\text{CO}_2)_2$ (Soriano-Garcia & Parthasarathy, 1978) is again different, with chains of alternating HY^- and H_2Y residues. The K ion is coordinated by eight O atoms at distances less than 3.1 \AA (Table 2). There are no further contacts less than 3.5 \AA ; O(4) and O(6) do not coordinate to the cation – both are of the O(H) type.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44590 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). **C44**, 611–614

trans-Diamminebis(1-methylcytosine- N^3)platinum(II) Diperchlorate

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(Received 8 October 1987; accepted 23 November 1987)

Abstract. $[\text{Pt}(\text{NH}_3)_2(\text{C}_5\text{H}_7\text{N}_3\text{O})_2](\text{ClO}_4)_2$, $M_r = 1015.8(4) \text{ \AA}^3$, $Z = 2$, $D_m = 2.23(2)$, $D_x = 678.31$, monoclinic, $P2_1/n$,* $a = 14.569(3)$, $b = 2.22 \text{ g cm}^{-3}$, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$, $F(000) = 652$, $\mu = 10.388(3)$, $c = 6.753(1) \text{ \AA}$, $\beta = 96.35(1)^\circ$, $V = 76.0 \text{ cm}^{-1}$, $T = 297 \text{ K}$, $R = 0.0529$ and $wR = 0.0483$ for 1794 unique reflections and 183 parameters. The Pt atom resides on an inversion centre and is coordinated to the N atom of each ammonia and the N(3) of each

* This non-standard cell may be transformed to the $P2_1/c$ cell, $a = 6.753(1)$, $b = 10.388(3)$, $c = 15.365(3) \text{ \AA}$, $\beta = 109.55(1)^\circ$ by the matrix $(00 -1/010/101)$.