SDPDRR-3

Sample 1 – Calcium Tartrate Tetrahydrate

CIF including the details about the crystal structure determination

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data_global
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# 3. TITLE AND AUTHOR LIST


_publ_section_title
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Calcium tartrate tetrahydrate second form in rat urinary stones
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    _publ_author_name
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4. TEXT

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The title compound, [Ca(C~'4~H~4~O~6~)].4H~2~O is a new triclinic
centrosymmetric polymorph from rat kidney calculus. The four water
molecules belong to the calcium atom coordination, a square antiprism.
;
_publ_section_comment
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Large crystals (100-200 microns) were extracted from rat kydney.
The chemical analysis suggested a hydrated calcium tartrate. The
crystal structure determination revealed a new tetrahydrated form.
This is unusual since calcium in human urinary stones is mainly
found included in oxalates (70% of the cases) or phosphates (15%)
(Bazin et al., 2007)

The crystal structure of the previously known form of calcium tartrate
tetrahydrate (frequently growing in bottles of wine) has been
reported four times (Ambady, 1968; Hawthorne at al., 1982; Boese and
Heinemann, 1993, Kaduk, 2007) in the P2~1~2~1~2~1~ space group. The
present second form is 2.4% less dense and centrosymmetrical. The calcium
environment is an almost perfect square antiprism (Fig. 1), the four water
molecules forming one of the squares. By the other square of oxygen atoms,
calcium links the tartrate molecules into infinite chains parallel to
the a axis. The two remaining oxygen atoms (O(5), O(6)) of the tartrate
molecule are involved in inter-chain linkage through hydrogen bonding
in the ac and ab planes together with the water molecules (Fig. 2).
The two polymorphs are quite different. In the first form, the calcium atom
is in a distorted Siamese dodecahedron formed by 2 water molecules and the
six oxygen atoms of the tartrate molecule, the two remaining water
molecules
participating only in intermolecular linkage.
;
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;
Crystals were found systematically twinned, and first resisted to the
characterization attempts. Indexing in a triclinic cell was realized
from powder diffraction data by using the McMaille software (Le Bail,
2004).
The structure was solved from the powder data by direct space
methods using the ESPOIR software (Le Bail, 2001) applied to intensities
extracted by iterating the Rietveld decomposition formula (Le Bail et al.,
1988). The tartrate molecule was rotated and translated together with the
calcium and remaining water oxygen atoms up to find an optimum by a Monte
Carlo process. From these convincing results, more efforts were done with
the data recorded from twinned crystals, producing a final refinement of
much
higher quality than from the powder data, this in spite of the relatively

high R factor due to peak overlapping from several twin domains. Refinement applied full-matrix least-squares methods (Sheldrick, 1997). Hydrogen atoms were taken from a difference Fourier map and refined isotropically with geometrical constraints and two common isotropic displacement factor (0.07 A^2^
for Hw and 0.04 A^2^ for the others). A few (35) reflections severely affected
by the twinning were omitted.
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;

Allen, F.H., Johnson, O., Shields, G.P., Smith, B.R. & Towler, M. (2004). J. Appl. Cryst. 37, 335-338.

Ambady, G.K. (1968). Acta Cryst. B58, 380-388.

Bazin, D., Chevallier, P., Matzen, G., Jungers, P. & Daudon, M (2007) Urol. Res. 35, 179-184.

Boese, R. & Heinemann, O. (1993). Z. Kristallogr. 205, 348-349.

Brandenburg, K. (2001) DIAMOND. Version 2.1e. Crystal Impact GbR, Bonn, Germany.

Farrugia, L.J. (1997). J. Appl. Cryst. 30, 565.

Hawthorne, F. C., Borys, I. & Ferguson, R. B. (1982). Acta Cryst. B38, 2461-2463.

Kaduk, J. A. (2007). Powder Diffraction 22, 74-82.

Le Bail, A. (2001). Mat. Sci. Forum, 378-381, 65-70.

Le Bail, A. (2004). Powder Diffraction 19, 249-254.

Le Bail, A., Duroy, H. & Fourquet, J. L. (1988). Mat. Res. Bull. 23, 447-452.

Sheldrick, G. M. (1997). SHELXL97. University of Gottingen, Germany.

Stoe & Cie (1998). Stadi4 (Version 1.07) and XRED32 (Version 1.10). Stoe & Cie, Darmstadt, Germany.
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Figure 1.
ORTEP (Farrugia, 1997) views of tartrate molecule and of the calcium square antiprism.

Figure 2.
Packing diagram with hydrogen bonds.
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OW1 H1W1 OW2 2_666 0.89(4) 1.84(5) 2.729(6) 178
OW1 H2W1 O4 2_677 0.89(8) 2.26(8) 2.919(5) 131
OW2 H1W2 O5 1_554 0.90(7) 1.86(6) 2.728(6) 163
OW2 H2W2 OW3 1_655 0.89(6) 2.11(6) 2.933(5) 152
OW3 H1W3 O6 1.555 0.89(5) 1.91(5) 2.798(6) 173
OW3 H2W3 O5 2_667 0.91(4) 1.87(5) 2.738(6) 157
OW4 H1W4 O6 1_655 0.90(5) 1.96(6) 2.853(5) 170
OW4 H2W4 O6 2_667 0.90(5) 2.10(5) 2.947(5) 155
O4 HO4 O2 2_778 0.88(3) 1.94(3) 2.783(5) 160
O3 HO3 O1 2_677 0.87(3) 1.82(3) 2.683(5) 169
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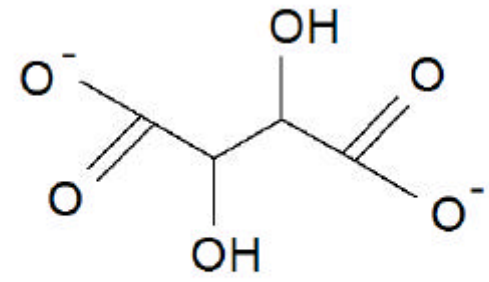
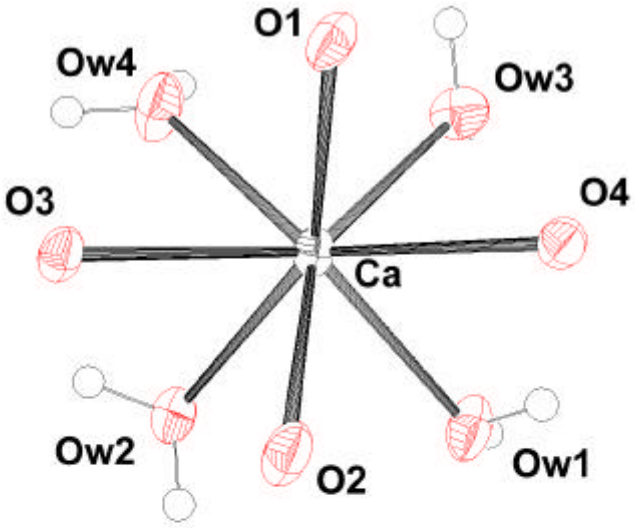
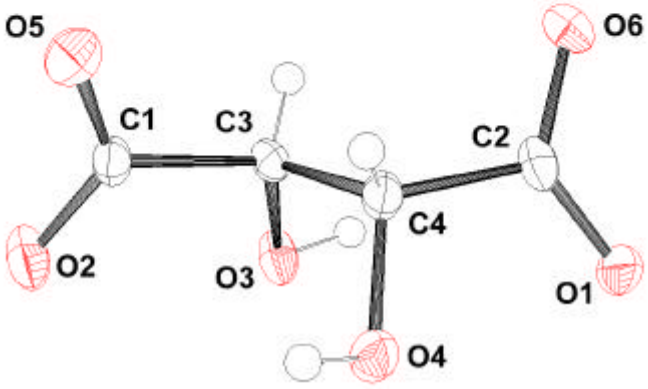
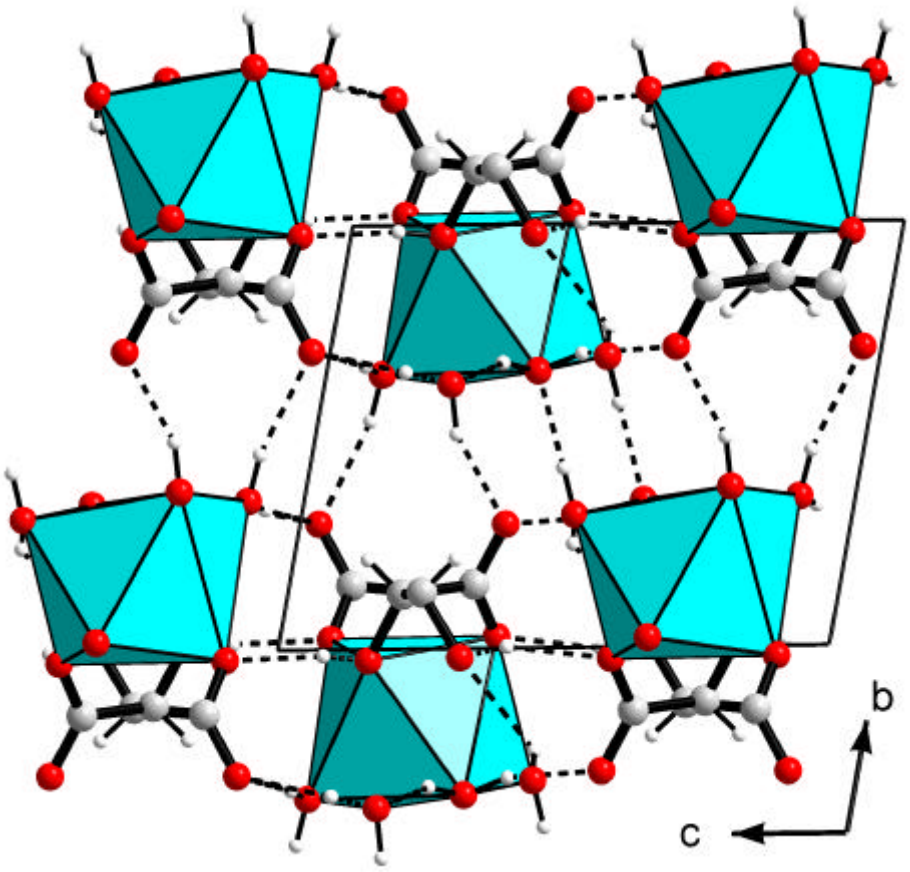
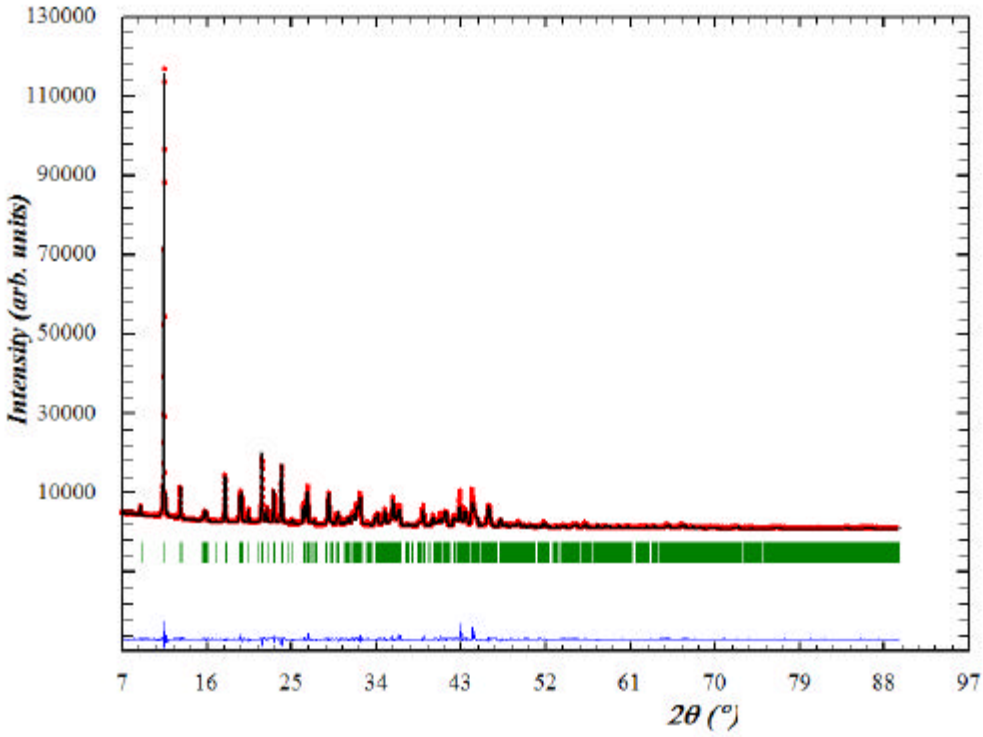

OH
O-
O
O
O-
OH

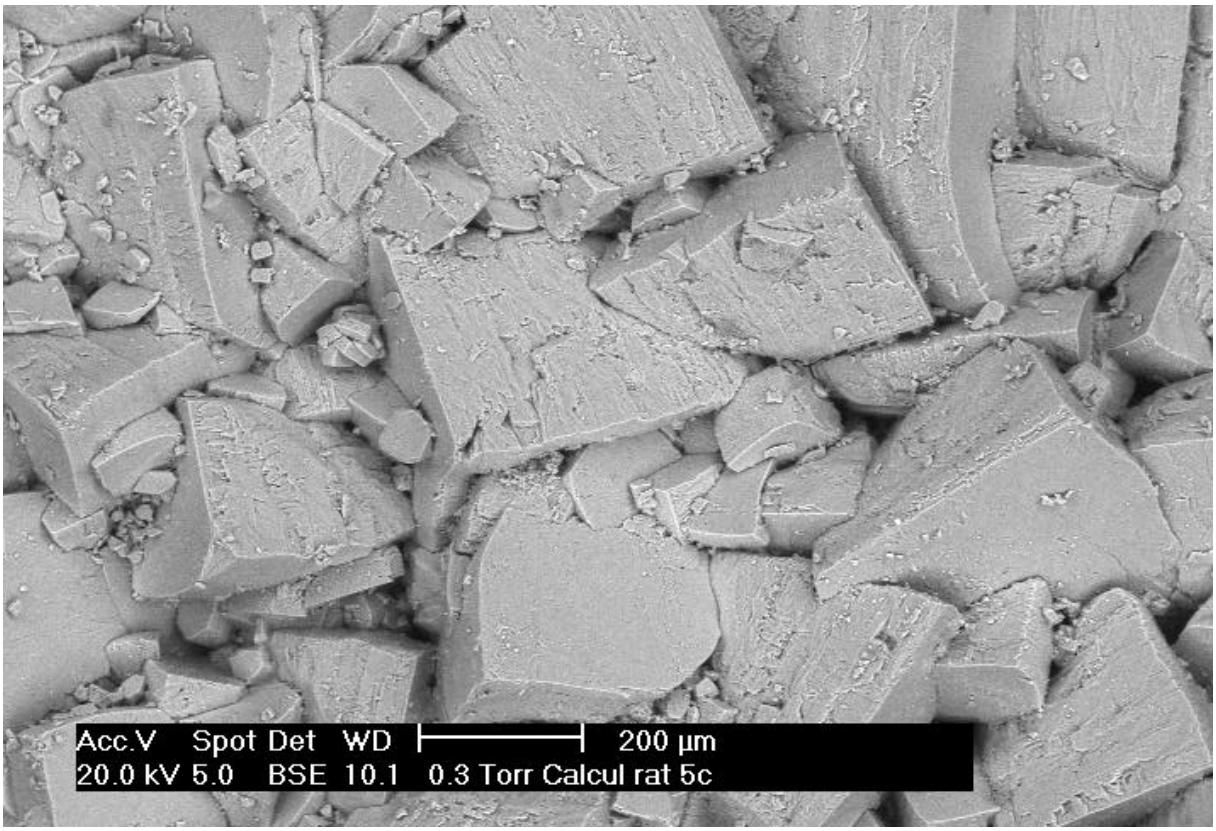
Ca^{2+} $4H_2O$


O1
Ow4
Ow3
O3
O4
Ca
Ow2
O2
Ow1


O5
O6
C1
C3
C2
C4
O2
O3
O1
O4


b
c


130000
110000
90000
70000
50000
30000
10000
Intensity (arb. units)
7
16
25
34
43
52
61
70
79
88
97
2θ (°)


Acc.V Spot Det WD 200 µm
20.0 kV 5.0 BSE 10.1 0.3 Torr Calcul rat 5c