5. LATTICE VARIATION

In this chapter we deal with the determination of lattice parameters of the grown crystals and checking for lattice distortion using the x-ray diffraction data collected on powdered samples of all the crystals grown.

5.1 X-ray Diffraction Measurements

X-ray diffraction has served to establish detailed features of molecular structures of all kinds of stable chemical species, from the simplest to those containing thousands of atoms. X-ray intensity measurements made by means of fully automated diffractometer has greatly contributed to the growth of successful crystal structure analysis due to its precision and easy to handle. A typical x-ray powder diffraction data will provide us the angle of scattering (2θ) and the corresponding intensities of diffracted beams for each reflection. Lattice parameters can be determined using this data. Using this data we can also check for any serious lattice distortion.

Using Bruker AXS D8 Advance X-ray diffractometer with CuKα radiation at a temperature of 25±1°C the x-ray diffraction data were collected from powdered samples of best crystals (for all the seven crystals grown). The data collection parameters are given in Table 5.1.

5.2 Indexing the Data

Indexing of a diffraction pattern is done by assigning the three numbers h,k,l (the Miller indices) to each reflection. The data were indexed following the procedures of Lipson and Steeple [1]. Calcium tartrate tetrahydrate belongs to the orthorhombic system [2]. Hence the following relation for the orthorhombic system was used for indexing the data for pure calcium tartrate tetrahydrate.
### Table 5.1: X-ray powder diffraction data collection parameters

<table>
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<tr>
<th>Make/Model</th>
<th>Bruker AXS D8 Advance</th>
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<tr>
<td>Configuration</td>
<td>Vertical, Theta/2 Theta geometry</td>
</tr>
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<td>435, 500, and 600 mm predefined</td>
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<td>360°</td>
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<tr>
<td>Max. Usable angular range:</td>
<td>3 to 135°</td>
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<tr>
<td>Smallest addressable increment</td>
<td>0.001°</td>
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<tr>
<td>Max. angular speed</td>
<td>30°/s</td>
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<tr>
<td>X-ray source</td>
<td>Cu, Wavelength 1.5406 Å</td>
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<tr>
<td>Detector</td>
<td>Si(Li) PSD</td>
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</table>
\[ 4\sin^2\theta = \{\lambda^2/a^2\} h^2 + \{\lambda^2/b^2\} k^2 + \{\lambda^2/c^2\} l^2 \quad (5.1) \]

where \( \theta \) (hkl) is the Bragg angle, \( \lambda \) is the wavelength of X-radiation used, a, b and c are the lattice parameters and h, k and l are the reflection indices. The indexed X-ray powder diffraction intensity data for pure and strontium added calcium tartrate tetrahydrate single crystals obtained in the present study are given in Tables 5.2 to 5.8. Also, data for the pure crystal are compared with that from JCPDS file in Table 5.9.

### 5.3 Lattice Parameters

From the indexed data, unit cell parameters were calculated from the high angle reflections using the relation (5.1). The calculated lattice parameters are presented in Table 5.10.

The lattice parameters for pure calcium tartrate tetrahydrate crystals obtained in the present study are \( a = 9.234 \), \( b = 10.635 \) and \( c = 9.667 \) Å. The values reported in the literature are \( a = 9.24 \), \( b = 10.63 \) and \( c = 9.66 \) Å [2]. The observed increase in volume of calcium tartrate tetrahydrate crystal caused by the impurities indicates that the impurities have entered into the lattice of calcium tartrate tetrahydrate crystals.
Table 5.2: Indexed intensity data for pure calcium tartrate tetrahydrate

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Table 5.3: Indexed intensity data for impurity added calcium tartrate tetrahydrate (0.4 mole%)

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Table 5.4: Indexed intensity data for impurity added calcium tartrate tetrahydrate (0.8 mole%)

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Table 5.5: Indexed intensity data for impurity added calcium tartrate tetrahydrate (1.2 mole%)

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Table 5.8: Indexed intensity data for impurity added calcium tartrate tetrahydrate (10 mole%)

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Table 5.9:
XRD data for pure calcium tartrate tetrahydrate crystals compared with JCPDS file

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<th>20(°)</th>
<th>I/I₀</th>
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<td>21</td>
<td>21.446</td>
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Table 5.10: Lattice parameters of the crystals grown in the present study

<table>
<thead>
<tr>
<th>System</th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>V (Å³)</th>
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<td>Pure</td>
<td>9.234(8)</td>
<td>10.635(3)</td>
<td>9.667(5)</td>
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<tr>
<td>Impurity added</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>(concentration in mole %)</td>
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<tr>
<td>0.4</td>
<td>9.231(32)</td>
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<td>9.677(12)</td>
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<td>9.648(41)</td>
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<td>9.318(43)</td>
<td>10.639(59)</td>
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<td>9.335(84)</td>
<td>10.648(19)</td>
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<td>9.412(38)</td>
<td>10.654(102)</td>
<td>9.657(98)</td>
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<td>9.541(26)</td>
<td>10.688(112)</td>
<td>9.641(87)</td>
<td>983.13</td>
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</table>

# - esd’s are given in parentheses.
5.4 Check for Lattice Distortion

Calculation of $\Delta 2\theta$ values ($\Delta 2\theta = 2\theta_{\text{exp}} - 2\theta_{\text{calc}}$; $2\theta_{\text{exp}}$ is the experimentally observed $2\theta$ value and $2\theta_{\text{calc}}$ is the $2\theta$ calculated using the lattice parameters for pure calcium tartrate tetrahydrate crystal available in the literature [2]) was done to check for any serious lattice distortion.

The $\Delta 2\theta$ values vary from $-0.34$ to $+0.27$ suggesting that there was no serious lattice distortion in any of the systems studied in the present study. This slight variation may be attributed to strains caused by powdering the sample crystals as well as the addition of impurities. As no early works are available on this for calcium tartrate tetrahydrate we are unable to compare our results.
References


2. G.K. Ambady *Acta Cryst.* **B24** (1968) 1548