

# Zonal Refining and Bridgman Technique for CsI:Tl Scintillation Crystal Growth

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#### 1. Introduction

This work describes the development of the production of the crystal cesium iodide doped with thallium [CsI (Tl)] for use as a radiation detector. For salt purification, zonal refining in a horizontal oven at a constant temperature of 700°C was used. The high temperature region corresponds to approximately 10% of the bed containing the salt (26 centimeters), this region moves with a speed of 5cm/h. The Bridgman technique was used for crystal growth, using a vertical oven whose speed is 1mm/h.

#### 2. Methodology

The Cesium Iodide crystal, doped with 10<sup>-3</sup>M of Thallium was produced at IPEN/CNEN/SP. The cesium iodide salt was weighed and then placed in a properly cleaned quartz crucible. To clean the quartz crucible, 10% of Extran was used in relation to the crucible volume, which was soaked for 24 hours. Then, a solution containing 5% of hydrofluoric acid was prepared, which remained for 40 min and rinsed with ultra-pure water and mili-Q water. The quartz crucible was left in a vacuum and then sealed and taken to the horizontal furnace for zonal refining. The melting point of CsI is 626°C [1] and that is why the temperature of 700°C was chosen for the zonal refining procedure, as this would ensure the melting of the CsI salt. Zonal refining is an effective procedure to produce metals with high purity, in crystalline materials. Zonal refinement corresponds to the repeated movement between one or a series of zones caused to slowly melt the salt along a solid block [2]. In this process the impurities are carried out to the ends of the quartz crucible. According to Figure 1- (a), it is possible to observe the solid formed after the zonal refining. Upon removal from the tube disregarding the ends, the CsI solid was macerated and placed in another properly cleaned quartz crucible, as shown in Figure 1 (b).



Figure 1- (a) CsI solid after zonal refining and (b) after the mass of crystals are macerated.

After purification by zonal refinement of the CsI salt, Thallium Iodide was added and then the crucible was taken to a vacuum for the thermal treatment of the salt. Iodine was added to the crucible inside a glove box in an argon gas atmosphere, in order to prevent oxygen from entering the crucible. The addition of iodine is intended to compensate for any loss of saline iodine caused by evaporation.

After the quartz crucible was sealed and the CsI(Tl) salt previously purified by the zonal refining method was placed in a vertical oven, the CsI(Tl) crystal was grown by the Bridgman method [3,4], as shown in Figure 2 (a) . Subsequently, the thermal treatment was carried out on the obtained crystal in order to eliminate the coloration due to the addition of iodine, as shown in Figure 2 (b). The CsI(Tl) crystal was cut with a diamond wheel lubricated with ethylene glycol. Then the crystal was polished having the final geometry of a parallelepiped with dimensions of 12.22 x 12.22 x 18.80 mm<sup>3</sup> (base area x height), as shown in Figure 2 (c) and (d).





Figure 2 (a) CsI crystal (Tl) after the Bridgman method. (b) CsI crystal (Tl) after heat treatment. (c) CsI (Tl) crystal in cutting process. (d) Final geometry of the CsI (Tl) crystal.

#### 3. Results and Discussion

The Figure 3 shows the spectra obtained for the sources of <sup>22</sup>Na, <sup>133</sup>Ba, <sup>60</sup>Co and <sup>137</sup>Cs of the CsI(Tl) crystal whose source salt was previously purified by the zonal refinement method.

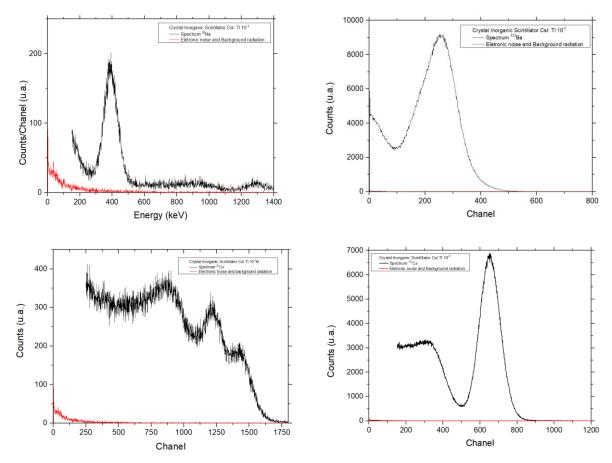


Figure 3 spectra obtained for the sources of (a)  $^{22}$ Na, (b)  $^{133}$ Ba, (c)  $^{60}$ Co and (d)  $^{137}$ Cs of the CsI(Tl) crystal.

### 4. Conclusions

The zonal refinement method is a powerful salt purification method used in crystal growth for scintillation detector purposes. By means of this method of purification it is possible to use salts with a low degree of purity and obtain the growth of scintillating crystals of adequate quality.

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