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HIGH RESOLUTION ELECTRON DENSITY DISTRIBUTION OF POWDER X – RAY DATA OF CSCL AND CSI

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Introduction

The powder sample of CsCl and CsI used in this work was sieved using a mesh. The mesh was a nylon cloth and the powder X – ray data were collected by X – ray diffractometer. The X – ray intensity data was collected using X- ray monochromatic beam of wavelength 1.54046 Å.

Data Collection:

Parameters	CsCl	CsI
Radiation used	CuKα	CuKα
2 range	$10 – 120^0$	$10 – 120^0$
Step size	0.08356	0.08356
No. of unique bragg peaks	19	25
Refined cell parameters	4.1228Å	4.5784Å
Space group	Pm 3 m	Pm 3 m
Diffractometer	X – pert pro alpha - I (Philips)	

Indexing procedure

The cell constant obtained from JCPDS for both CsCl and CsI is used in the indexing procedure. By knowing the cell constant and Θ values for various intensities the value N is determined.

Hence by finding the $\sin^2\Theta/\lambda^2$ values for various intensities one can identify and index the reflections. In this manner, the prominent peaks in the powder profiles have been identified and indexed for CsCl and CsI.

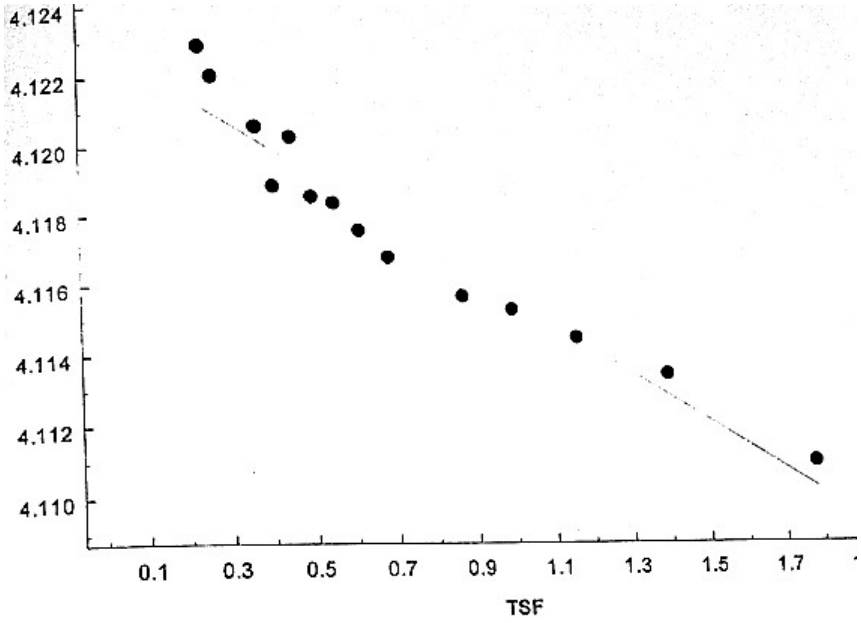
Nelson – Riley method

Using this Taylor-Synclair function, the accurate value of the cell parameter was determined by plotting TSF vs. cell constant. The cell parameters are fitted to a straight line, the y-intercept gives

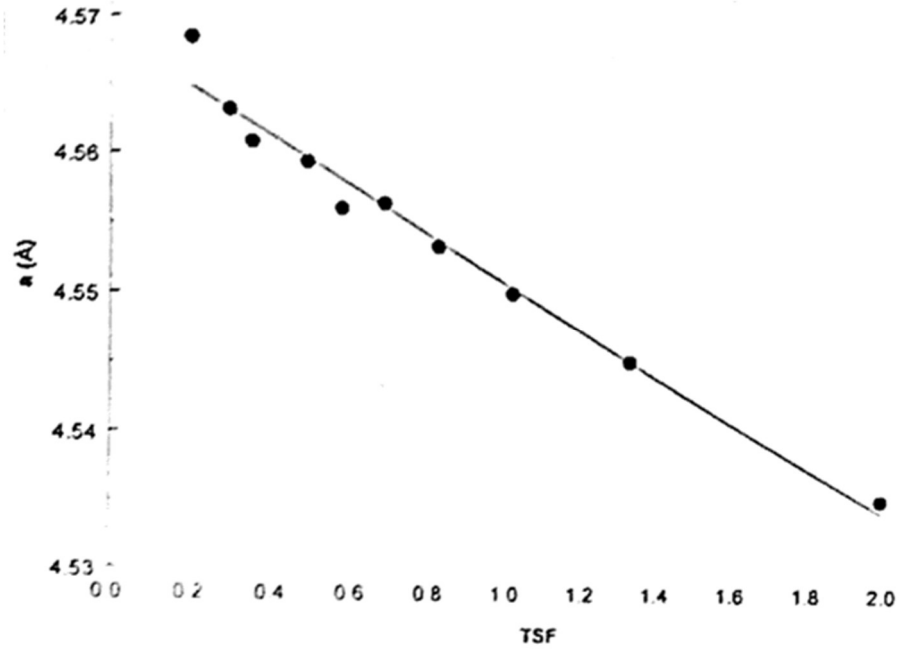
the exact value of the cell constant.

The cell constant determined by Nelson-Riley method for CsCl and CsI is in excellent agreement with those of reported values.

Nelson – Riley plot for CsCl



Nelson – Riley plot for CsI



Comparison of the value of cell constant obtained from Nelson – Riley method with the reported JCPDS value

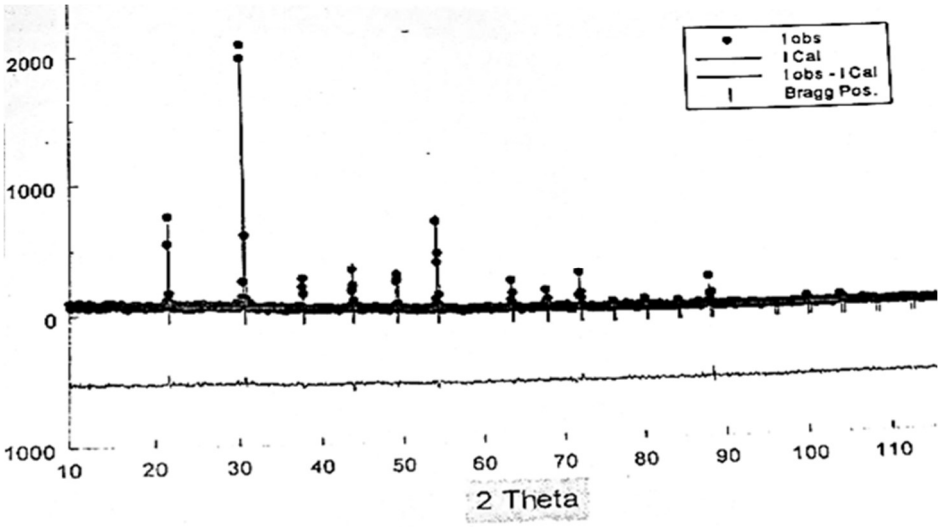
	CsCl	CsI

Nelson – Riley	4.1229(5)	4,5684(9)

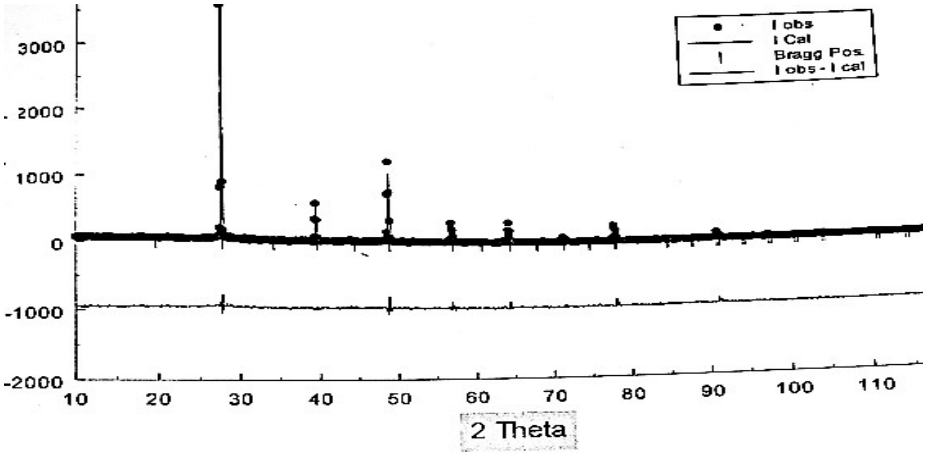
Fullprof Refinment

The raw powder X-ray intensity data of CsCl and CsI has been subjected to the well-known powder profile fitting technique known as Rietveld refinement. The background parameters of the profile will be fitted to suitable polynomials. There are various parameters models available in Rietveld profile fitting methodology. The cell parameters and other structural parameters can be refined using this technique. The peak width can be adjusted and refined using the peak shape parameters. The asymmetric peak widths can also be refined. The profile fitting models such as Voigt, pseudo-Voigt, Pearson II etc., can be used for the profile fitting. For the present work of both the samples, the asymmetry correction as reported elsewhere and the background was refined by a polynomial function. The Pseudo-Voigt function (ETA variable) Profile fitting model was used.

Fullprof Refinment profile for CsCl



Fullprof Refinment profile for CsI



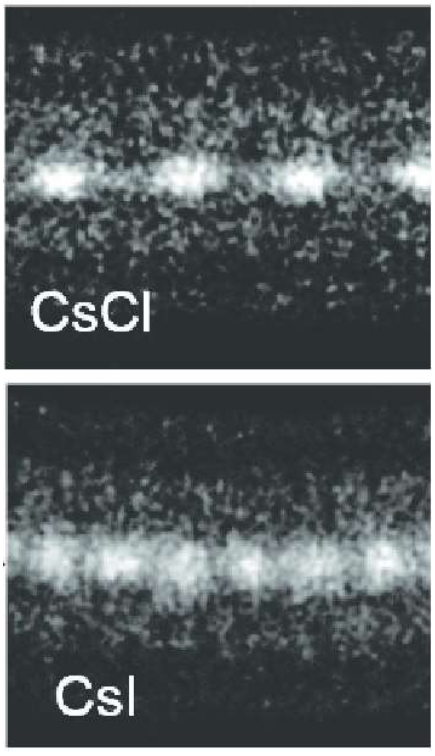
Final R-factor for both CsCl and CsI after the completion of fullprof refinement

Parameter	CsCl	CsI
R	4.78	5.84

Maximum Entropy Method

An investigation on the bonding in CsCl and CsI was carried out from the high resolution electron density mapping plotted using maximum entropy method. In the present work, the data was refined using Rietveld technique and the refined data was used to obtain the high resolution electron density by maximum entropy method. The softwares Fullprof and JANA2000 were used for profile refinements. The main advantage of using powder intensity data over single crystal data is very obvious as it avoids the extinction effects. If the extinction correction was not correctly applied then the data will not be able to elucidate on bonding between atoms. Thus making the data collected on single crystal redundant. The program used to calculate the electron density using maximum entropy method is reported elsewhere. All the calculations were carried out by using the formulations adopted to determine accurate structure by maximum entropy.

Electron density distribution in CsCl and CsI



Conclusion

A systematic approach X-ray powder data of CSCI and CsI has been used to elucidate accurate cell

constant using Nelson-Riley technique. The X- ray powder data was refined using Rietveld Technique by Fullprof refinement. The low R-factor values for both the samples reveal the quality of the data and the sample. Highly resolved electron density maps for both CsCl and Csl using powder data have been elucidated for the first time. The electron density map drawn clearly revealed qualitative as well as quantitative and versatility of MEM techniques.

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