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## Configuration of Polymorphic Single Crystal $\text{Er}_2\text{Si}_2\text{O}_7$ and Its Comparison with C-type & D-type Single Crystals

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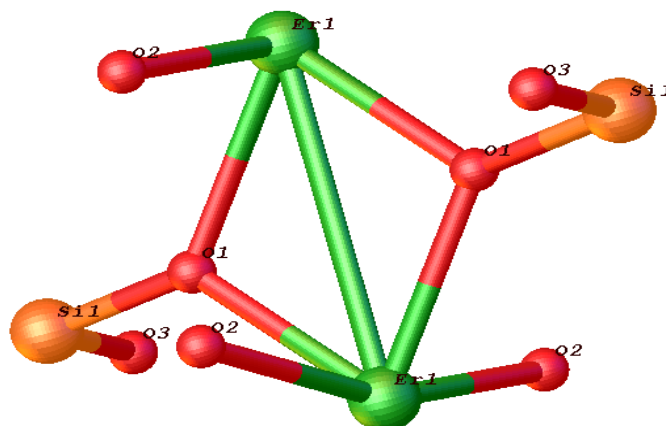
### Abstract

Structure of polymorphic  $\text{Er}_2\text{Si}_2\text{O}_7$  single crystal grain is determined using X-Ray diffractometer (Bruker Kappa ApexII). Crystal structure is refined through the use of software Shelxl. Findings declared also three dimensional view of the compound under study. The crystal crystallizes in space group (C2/m) with monoclinic symmetry. Bond length between erbium and oxygen is  $2.24(\text{Å})^0$ , bond length of oxygen and silicon is  $1.62(\text{Å})^0$ . The bond angle between Er-O-Er is  $101^0$ , O -Er-O is  $85.13^0$  and Er -O1-Si is  $128.94^0$  respectively with  $Z=2$ . The data is further compared with the previously reported powder x-ray diffraction results of C- and D-type crystal structures. The comparison showed a brief description of all structures and position of atoms in crystal.

**Keywords:**  $\text{Er}_2\text{Si}_2\text{O}_7$ ; Flux method; Single crystals; X-Ray diffraction; SheIxl.

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**Graphical Abstract:****1. Introduction**

With the development of new technology, it has become easy to study the internal structure and configuration of any particular compound. Indirect band gap and the absence of the linear electro-optic effect in silicon are incompatible for optoelectronic practices. Advance researches showed that fixing of rare earth ions into the Si structure is advantageous for Si optoelectronic characteristics, especially when using erbium Er [1].  $\text{Er}_2\text{Si}_2\text{O}_7$  is a compound that exists in three polymorphs e.g. a triclinic low-temperature phase (type B), a monoclinic modification (type C) and a high-temperature monoclinic modification (type D). Single crystals preparation of  $\text{R}_2\text{Si}_2\text{O}_7$ , (R = Tm, Er, Ho, Dy) by a flux technique has been reported [2]. Magnetic characteristics of D- $\text{Er}_2\text{Si}_2\text{O}_7$  at low temperatures were also measured [3]. The high Er content, relatively small thermal quenching of Er emission intensity and narrow emission line width suggest that erbium silicate represents a very interesting material for future application in Si-based amplifiers, light-emitting diodes and lasers [4]. Si-rich silicon oxide (SRO)/Er-Si-O/SRO multi layers on p-Si substrates were prepared by magnetron sputtering [5]. The structures and phase transitions of the rare earth disilicates was investigated and prepared by sintering the oxide components [6]. The description on the efficiency of thermal forging was attained in  $\text{N}_2$  or  $\text{O}_2$  ambient at  $1200^\circ\text{C}$  on the physical and optical properties of Er silicate films having different structural compositions [7]. The dispersal, arrangement and nucleation of erbium surrounded in  $\text{SiO}_2$ -Si layer was achieved with high resolution transmission electron microscopy (TEM) [8]. The structural characteristics of  $\text{SiO}_2$  thin films grown on Si substrates was also determined [9]. In this paper, we are interested in growth of single crystals and study the crystallographic structure of polymorphic  $\text{Er}_2\text{Si}_2\text{O}_7$  single grain crystal. The obtained results are also compared with the previous reports and results for above structure.

**2. Experimental Work****2.1. Preparation of crystal**

The polymorphic crystal was prepared [10] by a flux growth method. The composition of the  $\text{Er}_2\text{Si}_2\text{O}_7$  includes  $\text{Er}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{MoO}_3$ ,  $\text{PbF}_2$ ,  $\text{PbO}$  and experimental conditions for the growth of the given crystal were crucible

volume ( $20 \text{ cm}^3$ ), initial temperature ( $1270^\circ\text{C}$ ), soak period (24h). Further detail is tabulated in Table 1 and Table 2 respectively.

**Table 1:** Composition and experimental conditions for the growth of C- $\text{Er}_2\text{Si}_2\text{O}_7$  & D- $\text{Er}_2\text{Si}_2\text{O}_7$

No	Starting composition (mole %)					Crucible Volume ( $\text{cm}^3$ )	Initial temp ( $^\circ\text{C}$ )	Soak period (h)	Cooling rate ( $\text{kh}^{-1}$ )	Final temp ( $^\circ\text{C}$ )	Comments on the crystals
	$\text{Er}_2\text{O}_3$	$\text{SiO}_2$	$\text{MoO}_3$	$\text{PbF}_2$	$\text{PbO}$						
1	5.65	17.66	26.38	20.37	30.03	20	1270	24	2.4	1020	A few crystal up to 7 mm x 5mm x 1mm
2	5.81	17.49	26.40	20.25	30.04	20	1270	24	1.0	800	Optically clear faceted crystal up to 2mm x 3mm x 2mm
3	2.86	5.99	22.18	35.00	39.97	20	1270	24	1.0	800	Small crystal of C- $\text{Er}_2\text{Si}_2\text{O}_7$ or larger equidimensional $\text{Er}_2\text{SiO}_5$

When we used  $\text{K}_2\text{O}+\text{KF}$  in place of  $\text{PbO}+\text{PbF}_2$  instead  $\text{PbF}_2$ ,  $\text{PbO}$  then;

**Table 2:** Composition and experimental condition for the growth of C- $\text{Er}_2\text{Si}_2\text{O}_7$  & D- $\text{Er}_2\text{Si}_2\text{O}_7$

No.	Starting composition (mole %)					Crucible Volume ( $\text{cm}^3$ )	Initial temp ( $^\circ\text{C}$ )	Soak period (h)	Cooling rate ( $\text{kh}^{-1}$ )	Final temp ( $^\circ\text{C}$ )	comments on the crystals
	$\text{Er}_2\text{O}_3$	$\text{SiO}_2$	$\text{MoO}_3$	$\text{K}_2\text{O}$	$\text{KF}$						
1	5.75	15.17	25.31	0	14.55	20	1270	24	2.4	850	Six faceted crystals upto 4mm x 3mm x 2mm
2	5.71	16.87	23.45	5.01	10.96	20	1260	24	2.4	900	Thick nearly equidimensional crystal of C- type only

The material was characterized with the single-grain x-ray diffraction. For the crystallographic structure determination, the experimental work was carried out at Department of Physics, University of Sargodha. Single crystal x ray diffractometer, Bruker Kappa Apex II was used for the study of the structure of  $\text{Er}_2\text{Si}_2\text{O}_7$ . Bond lengths in Table 8, bond angles in Table 9 and torsion angles in Table 10 were determined by X-Ray diffractometer. Three dimensional view of the crystal is also illustrated in Figure 1, Figure 2 (a&b) and Figure 3.

### 3. Results and Discussion

Data was collected, refined and produced with the help of software's that are installed, in a master computer attached to Kappa APEX II Diffractometer. Using software's packages connected with Kappa APEX II Diffractometer crystal data and detail of structure refinement for  $\text{Er}_2\text{Si}_2\text{O}_7$ , are explained below.

**Table 3:** Crystal data

i	Identification code	Er <sub>2</sub> Si <sub>2</sub> O <sub>7</sub>
ii	Empirical formula	Er <sub>2</sub> Si <sub>2</sub> O <sub>7</sub>
iii	Formula weight/a.m.u	502.70
iv	Temperature/K	296.15
v	Crystal system	monoclinic
vi	Space group	C2/m

**Table 4:** Cell Parameters

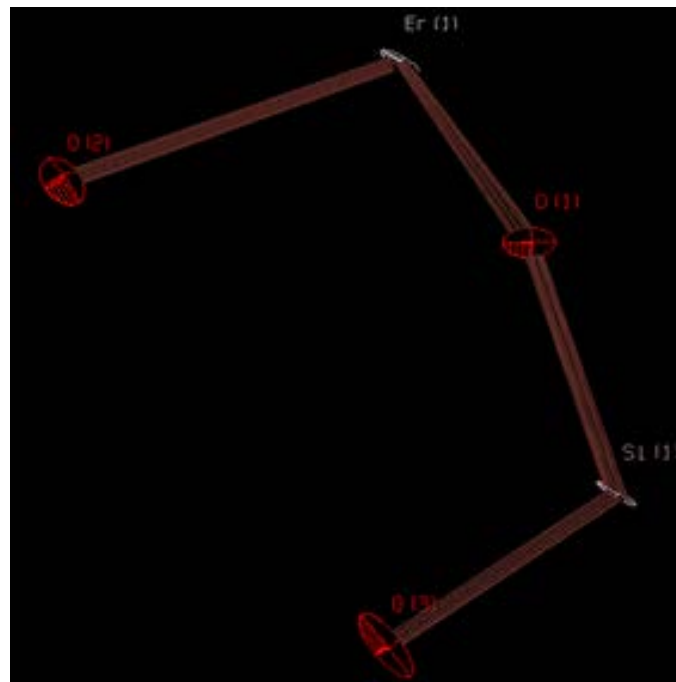
i	a/Å	6.8537(5)
ii	b/Å	8.9508(7)
iii	c/Å	4.7170(4)
iv	$\alpha$ /°	90.00
v	$\beta$ /°	101.655(4)
vi	$\gamma$ /°	90.00
vii	Volume/Å <sup>3</sup>	283.40(4)
viii	Z	2
ix	$\rho_{\text{calc}}$ g/cm <sup>3</sup>	5.891
xii	Crystal size/mm <sup>3</sup>	0.3 × 0.25 × 0.18
xiii	Radiation/A <sup>0</sup>	Mo K $\alpha$ ( $\lambda = 0.71073$ )

**Table 5:** Data collection and Refinement

i	2 $\theta$ range for data collection	7.58 to 72.4°
ii	Index ranges	-11 ≤ h ≤ 11, -14 ≤ k ≤ 13, -6 ≤ l ≤ 7
iii	Reflections collected	2479
iv	Independent reflections	689 [R <sub>int</sub> = 0.0442, R <sub>sigma</sub> = 0.0386]
v	Data/restraints/parameters	689/0/32
vi	Goodness-of-fit on F <sup>2</sup>	1.204
vii	Final R reliability indexes [I >= 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0233, wR <sub>2</sub> = 0.0641
viii	Final R reliability indexes [all data]	R <sub>1</sub> = 0.0236, wR <sub>2</sub> = 0.0643
ix	Largest diff. peak/hole / e Å <sup>-3</sup>	3.24/-3.55

### 3.1. Three Dimensional view of the crystal

Different Three Dimensional structures are given beneath for  $\text{Er}_2\text{Si}_2\text{O}_7$ .



**Figure 1:** View of  $\text{Er}_2\text{Si}_2\text{O}_7$  in simplest form having angle  $101^\circ$  between Er and O

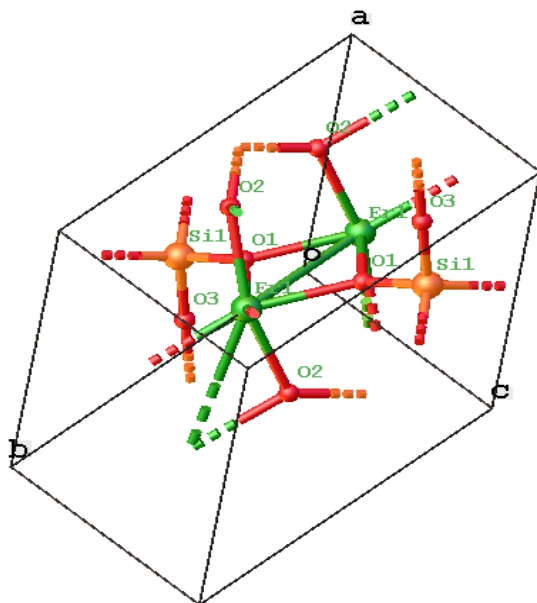


Figure 2a

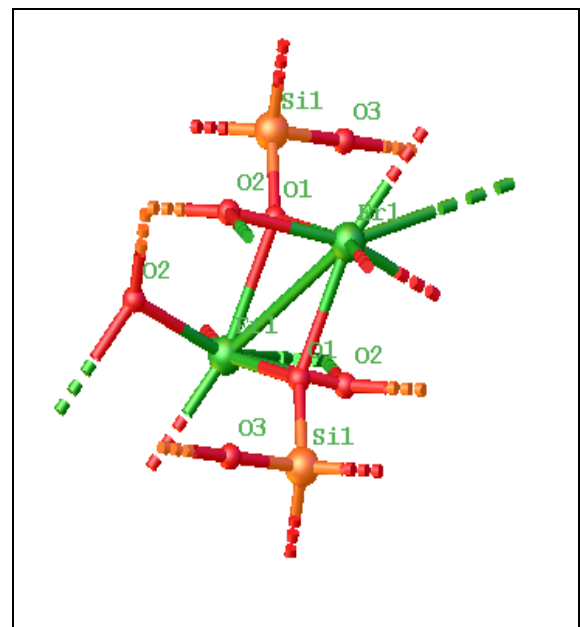
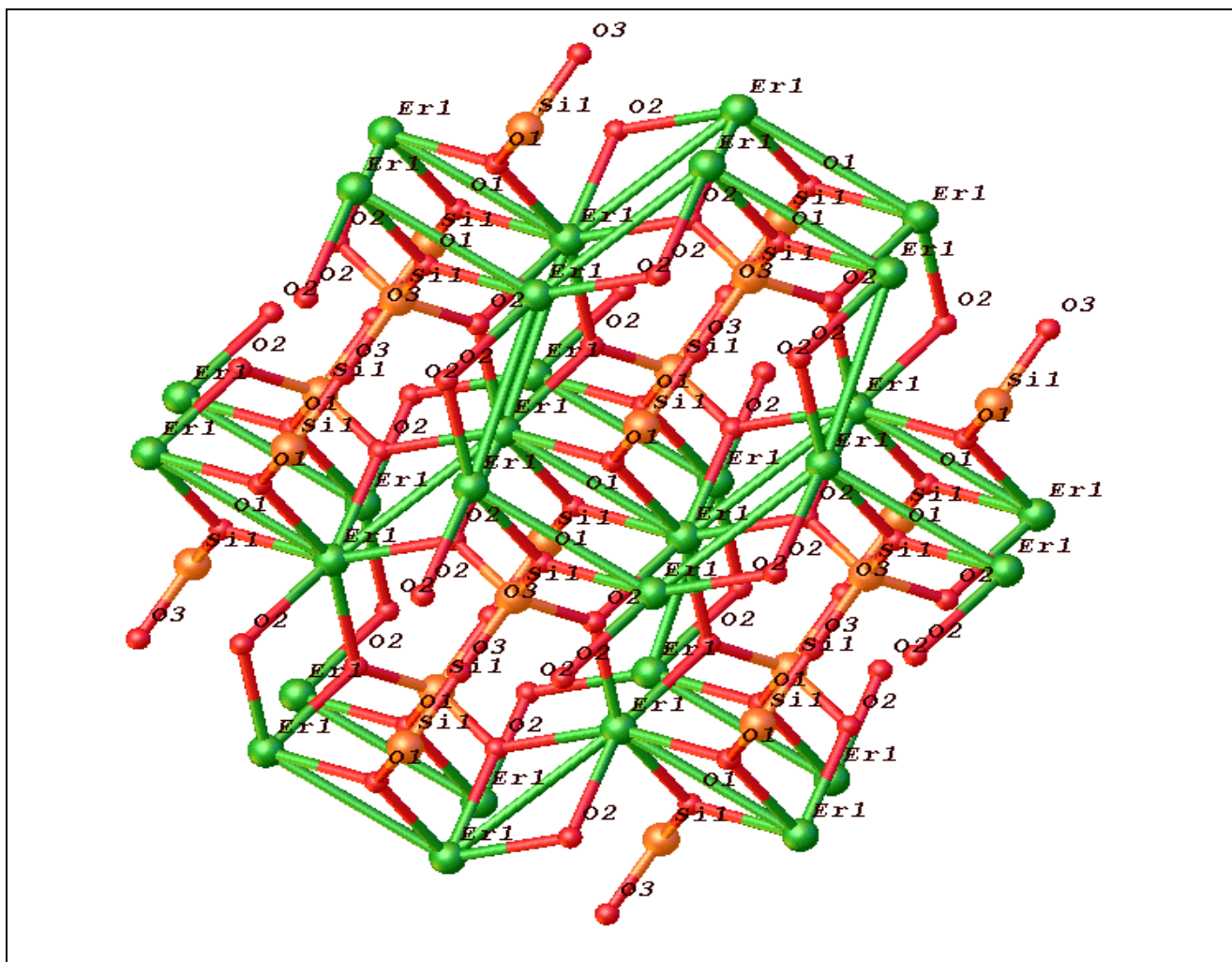


Figure 2b

**Figure 2 (a&b):** Three dimensional view of  $\text{Er}_2\text{Si}_2\text{O}_7$  in the unit cell and three dimensional view of packing diagram of erbium disilicate.



**Figure 3:** Three dimensional view of complex packing structure of  $Er_2Si_2O_7$

**Table 6:** Final Coordinates and Equivalent Isotropic Displacement Parameters of the non-Hydrogen atoms

Atom	X	Y	Z	Ueq [Å <sup>3</sup> ] <sup>2</sup>
Er1	0	0.19376	0	0.0038
Si 1	0.28196	0	0.58652	0.0034
O1	0.11972	0	0.28413	0.0067
O2	0.26320	0.14966	0.77651	0.0076
O3	1/2	0	1/2	0.0164

Where  $U_{eq} = 1/3$  of the trace of the orthogonalised  $U_{ij}$  Tensor

$$U_{eq} = \frac{1}{3} \sum_i \sum_j a_i^* a_j^* a_i a_j$$

**Table 7:** Anisotropic Displacement parameter For Er<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>

Atom	U(1,1)	U(2,2)	U(3,3)	U(2,3)	U(1,3)	U(1,2)
Er1	0.0005	0.0029	0.0078	0	0.0002	0
Si1	0.0002	0.0024	0.0074	0	0.0004	0
O1	0.0052	0.0069	0.0068	0	0.0016	0
O2	0.0066	0.0048	0.0124	0.0029	0.0041	-0.0021
O3	0.0062	0.0231	0.0223	0	0.0087	0

The Temperature Factor has the Form of Exp (-T) Where

$$T = 2\pi^2 [ h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12} ]$$

For an isotropic atoms. a\*,b\*,... are reciprocal axial lengths and h,k,l are the reflection indices.

**Table 8:** Bond Distances (Angstrom) for Er<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>

(Here Index a, c, d, i, j, h describes translation of symmetry code to equivalent positions given in Table 3.7)

Atoms	Bond length	Atoms	Bond length
Er1-O1	2.2434(2)	Er1-O2_j	2.2402(2)
Er1-O2_a	2.2999(2)	Si1-O1	1.6218(1)
Er1-O1_c	2.2434(2)	Si1-O2	1.6312(1)
Er1-O2_d	2.2999(2)	Si1-O3	1.6267(1)
Er1-O2_i	2.2402(2)	Si1-O2_h	1.6312(1)

**Table 9:** Bond Angles (Degrees) for Er<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>

(Here Index a, b, c, d, e... describes Translation of Symmetry Code to Equivalent positions given in Table 9)

Atoms	Bond angle	Atom	Bond angle
O1 -Er1 -O2_a	85.13(1)	O2_i -Er1 -O2_j	102.54(1)
O1 -Er1 -O1_c	78.74(1)	O1 -Si1 -O2	111.54(1)
O1 -Er1 -O2_d	79.61(1)	O1 -Si1 -O3	106.30(1)
O1 -Er1 -O2_i	93.43(1)	O1 -Si1 -O2_h	111.54(1)
O1 -Er1 -O2_j	155.17(1)	O2 -Si1 -O3	108.43(1)
O1_c -Er1 -O2_a	79.61(1)	O2 -Si1 -O2_h	110.42(1)
O2_a -Er1 -O2_d	160.24(1)	O2_h -Si1 -O3	108.43(1)
O2_a -Er1 -O2_i	76.25(1)	Er1 -O1 -Si1	128.94(1)

O2_a -Er1 -O2_j	116.90(1)	Er1 -O1 -Er1_f	101.26(1)
O1_c -Er1 -O2_d	85.13(1)	Er1_f -O1 -Si1	128.94(1)
O1_c -Er1 -O2_i	155.17(1)	Er1_b -O2 -Si1	123.30(1)
O1_c -Er1 -O2_j	93.43(1)	Er1_i -O2 -Si1	129.97(1)
O2_d -Er1 -O2_i	116.90(1)	Er1_b -O2 -Er1_i	103.75(1)

**Table 10:** Torsion Angles (Degrees) for Er<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>

(In crystallography it refers to the angle between two planes)

Planes	Angle
O2 -Si1 -O1 -Er1	34.37(1)
O3 -Si1 -O1 -Er1	-83.64(1)

**Table 11:** Translation of Symmetry Code to Equivalent Positions

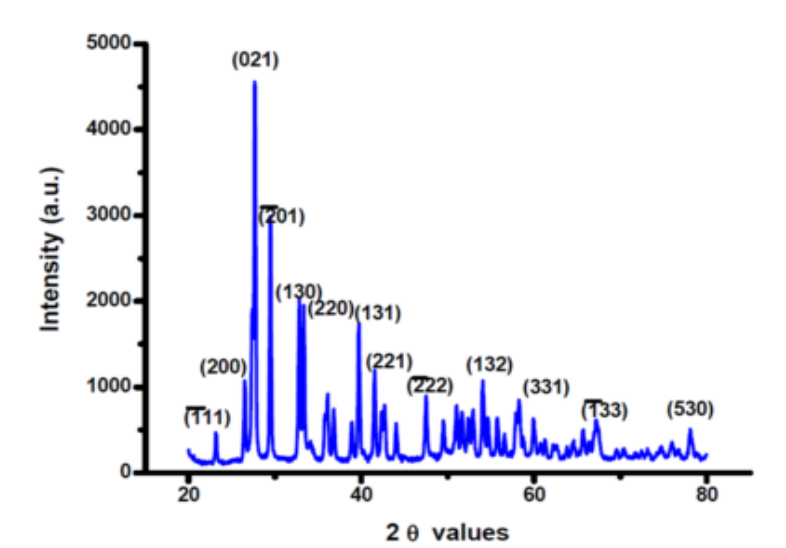
(Here Index a, b, c, d, e... describes Translation of Symmetry in three dimensions)

Symmetry code	Equivalent position	Symmetry code	Equivalent position
a = [ 1554.00]	x,y,-1+z	q = [ 5454.00]	-1+x,-y,-1+z
b = [ 1556.00]	x,y,1+z	r = [ 5455.00]	-1/2+x,1/2+y,-1+z
c = [ 2555.00]	-x,y,-z	s = [ 6555.00]	-1/2+x,1/2+y,z
d = [ 2556.00]	-x,y,1-z	t = [ 6556.00]	1/2-x,1/2+y,-z
e = [ 2656.00]	1-x,y,1-z	u = [ 7555.00]	1/2-x,1/2+y,1-z
f = [ 3555.00]	-x,-y,-z	v = [ 8455.00]	1/2-x,1/2-y,-z
g = [ 3656.00]	1-x,-y,1-z	w = [ 4556.00]	x,-y,1+z
h = [ 4555.00]	x,-y,z	x = [ 5545.00]	1/2+x,-1/2+y,z
i = [ 7556.00]	1/2-x,1/2-y,1-z	y = [ 5546.00]	1/2+x,-1/2+y,1+z
j = [ 8454.00]	-1/2+x,1/2-y,z	z = [ 6545.00]	1/2-x,-1/2+y,-z
k = [ 8556.00]	-1/2+x,1/2-y,-1+z	* = [ 6546.00]	1/2-x,-1/2+y,1-z
l = [ 1454.00]	1/2+x,1/2-y,1+z	* = [ 8555.00]	1/2+x,1/2-y,z
m = [ 2554.00]	-1+x,y,-1+z	* = [ 7557.00]	1/2-x,1/2-y,2-z
n = [ 3556.00]	-x,y,-1-z	* = [ 1656.00]	1+x,y,1+z
o = [ 4554.00]	-x,-y,1-z	* = [ 4656.00]	1+x,-y,1+z
p = [ 4454.00]	x,-y,-1+z		



#### 4. Conclusion

The structure of the above mentioned compound was solved using Single Crystal X ray diffractometer Bruker Kappa Apex II. Data was refined and collected using Shelxl programs, respectively, down to  $R_1 = 0.02$  and  $R_{2w} = 0.06$ . [11]. The entire unit of the single crystal is recognized. On the basis of peaks obtained (Figure 4) and atomic elements suggested in chemical formula, the atomic labeling of the molecule was carried out. The structure of compound  $\text{Er}_2\text{Si}_2\text{O}_7$  is monoclinic (type C). The results of compound was the verification of previous measurements of type C rare earth disilicates taken by powder X-ray technique that the high temperature polymorph forms a monoclinic structure with lattice constants,  $a = 6.849 \text{ \AA}$ ,  $b = 8.939 \text{ \AA}$  and  $c = 4.722 \text{ \AA}$ ,  $\beta = 101.8$  taken by Debye Sherer camera [10]. Present results also contain the additional information and values that enables us to know the detailed description of the compound. This study is carried out by latest single crystal X-ray diffractometer Bruker Kappa Apex II in grain form. The formula weight of compound is 502.70 a.m.u and have Space group  $C2/m$  with  $a/\text{\AA}=6.8537$ ,  $b/\text{\AA}=8.9508$ ,  $c/\text{\AA}=4.7170$  containing angles  $\alpha^\circ=90.00$ ,  $\beta^\circ=101.655(4)$ ,  $\gamma^\circ=90.00$ . It verifies the results with a goof factor less than one. Fractional atomic coordinates of non-hydrogen atoms in Table 6, Anisotropic displacement parameters in Table 7, Bond distances in Table 8 and bond angles in Table 9, and torsion angles in Table 10.



**Figure 4:** X-ray diffraction pattern of C-  $\text{Er}_2\text{Si}_2\text{O}_7$  (SEM)

In the whole process translation of symmetry is according to Table 11. Peaks in the diffraction pattern also show exact index value which justifies crystal structure [12].

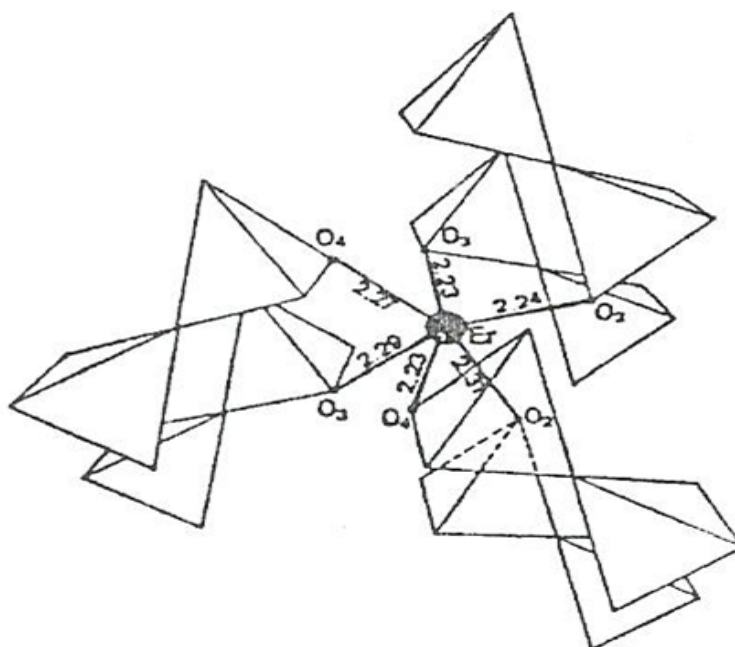
##### 4.1. Contrast of the single crystal grain structure with powder XRD structure of C-type & D-type Single Crystals

The crystal structure of type D-disilicates was first designated for  $\text{Y}_2(\text{Si}_2\text{O}_7)$  which is iso-structure with (Ho-Er) ( $\text{Si}_2\text{O}_7$ ). The configuration has lately been experienced from 1860 autonomous observations for D- $\text{Er}_2\text{Si}_2\text{O}_7$  which provided a final R-value of 6.2% for the 3 dimensional data and for C- $\text{Er}_2\text{Si}_2\text{O}_7$  the final R-value

determined is 5.4% [13].

The remarkable unit in the structure is  $\text{Si}_2\text{O}_7$  double tetrahedral group. Its centro-symmetry and  $180^\circ$  Si-O-Si angle charted right from the space group  $P2_1/b$ . Four fold common positions and two fold unusual positions at the center of symmetry are probable in this space group. Since the unit cell with  $Z=2$  contains only 2 pyro-silicates groups, they certainly conquer the exceptional positions. Proposition for the linearity of the Si-O-Si bond, consequently does not depend on the precision of the intensity data useful during the assembly modification as was the case with C type structure. The structure of D type crystal seems to provide additional evidence for the possible presence of  $\text{Si}_2\text{O}_7$  groups. Using powdered X-rays diffraction cell dimensions of polymorphic rare earth disilicates C-  $\text{Er}_2\text{Si}_2\text{O}_7$  were observed with values  $a=6.841 \text{ \AA}$ ,  $b=9.135 \text{ \AA}$  and  $c=6.94 \text{ \AA}$  with angle  $\beta=101.70^\circ$  and density of the same structure were  $\rho=5.80 \text{ g/cm}^3$ , in comparison dimension [14] of the D-  $\text{Er}_2\text{Si}_2\text{O}_7$   $a=5.588 \text{ \AA}$ ,  $b=10.793 \text{ \AA}$  and  $c=4.689 \text{ \AA}$  with angles  $\alpha=90^\circ$ ,  $\beta=95.82^\circ$  and  $\gamma=90^\circ$  and density was  $5.85 \text{ g/cm}^3$ . In current studies using latest grain X-ray Diffractometer the cell dimension for C-  $\text{Er}_2\text{Si}_2\text{O}_7$  is determined with  $a=6.853 \text{ \AA}$ ,  $b=8.850 \text{ \AA}$  and  $c=4.7170 \text{ \AA}$  with angles  $\alpha=90^\circ$ ,  $\beta=101.65^\circ$  and  $\gamma=90^\circ$  and calculated density  $\rho=5.891 \text{ mg/mm}^3$  which justify the previous aggregated data and updated the recent values for the same crystal.

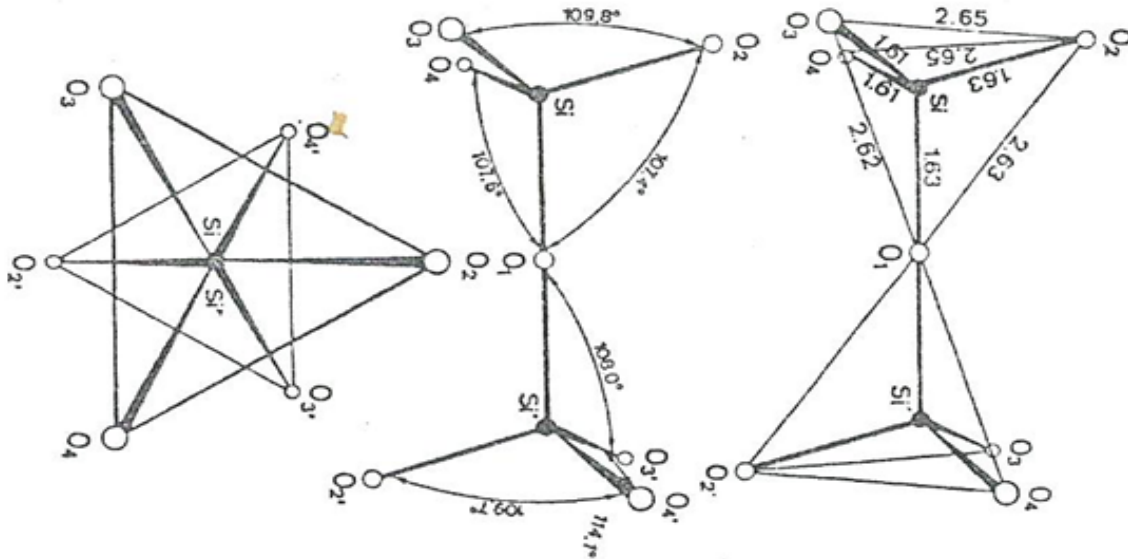
The stability of structure C-type is expected to be described by nearly closest hexagonal packing of oxygen holding rare earth cations in octahedral holes and silicones in the tetrahedral holes in the alternating parallel layers (001) in Figure 4. The  $\text{SiO}_4$  tetrahedra demonstrate a very little degree of falsification related to other disilicates configuration in Figure 6. The mean value of Si-O bond length is  $1.63 \text{ \AA}$ .



**Figure 5:** crystal structure of D- $\text{Er}_2\text{Si}_2\text{O}_7$  by powder X-ray diffraction

The mean Si-O terminal O length is  $1.62 \text{ \AA}$  Figure 6. These terminal oxygen atoms of the  $\text{Si}_2\text{O}_7$  group form intensely partial octahedral around the Er atoms like to those around the C- $\text{Er}_2\text{Si}_2\text{O}_7$  the associating oxygen atom

O (1) is not bonded to the Er cation. The mean Er-O space in the octahedron is 2.27 angstrom. With the sub group relation of the space group  $P2_1/b$  to space group  $C2/m$  of structure type C, some organizational topographies are correlated, namely, the staggered orientation of the double tetrahedral with the  $180^\circ$  Si-O-Si linking angle and the six fold oxygen synchronization around the dense atoms.



**Figure 6:** Cation-oxygen matching in D- $\text{Er}_2\text{Si}_2\text{O}_7$  by powder X-ray diffraction

Though, in structure type D in Figure 6. The orientation of the group ( $\text{Si}_2\text{O}_7$ ) groups relative to each other and the connection of the Er-O octahedral are contradictory. The  $\text{Si}_2\text{O}_7$  groups in structure type D are reciprocally directed along  $[0\ 1\ 1]$  and  $[0\ \bar{1}\ 1]$ . Afterward the synchronization of the heavy atoms does not product in a network forming topographies as in structure type C, but in a strip like arrange crystal along the B axis.

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