X-ray diffraction

Examples

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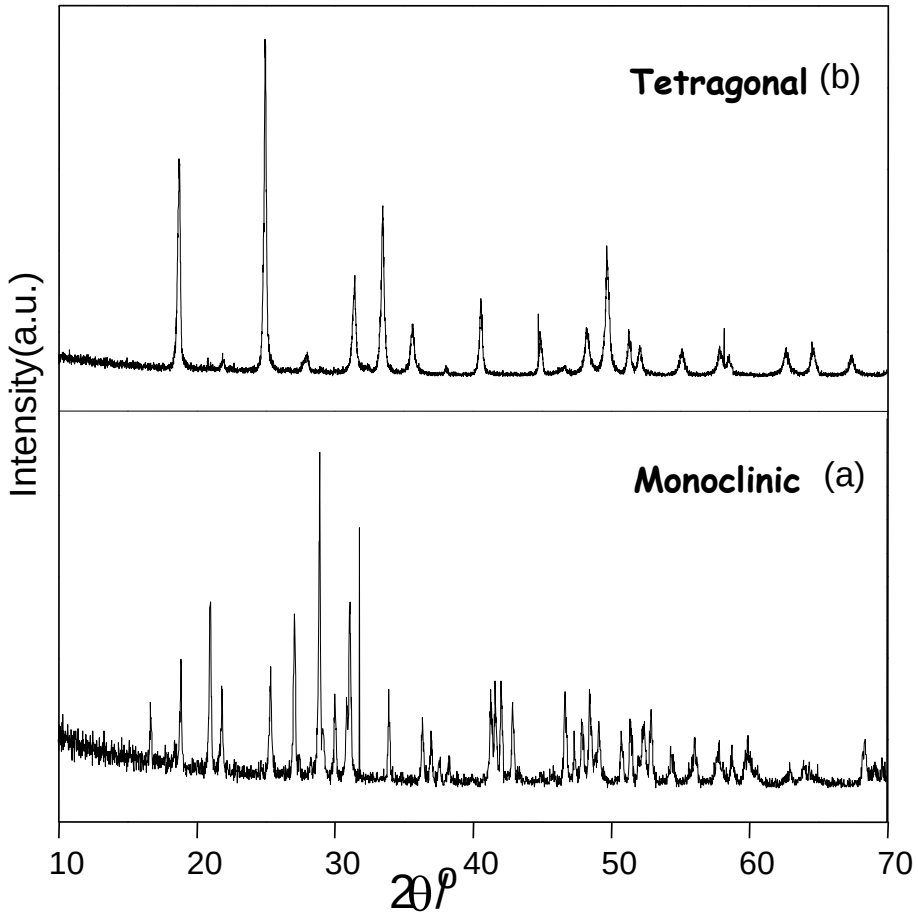
DATE:- 23/08/2018

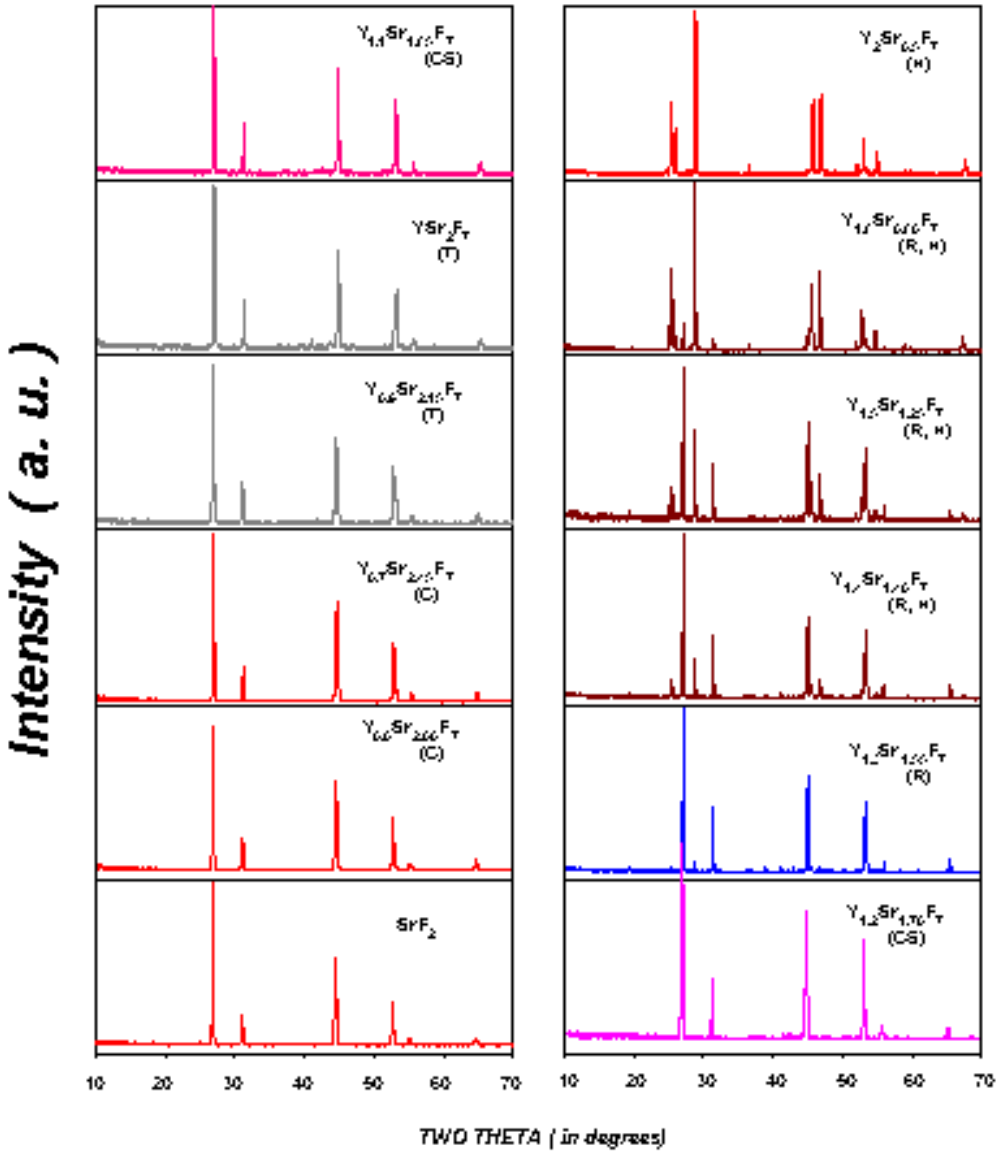
Effect of substitution on unit cell

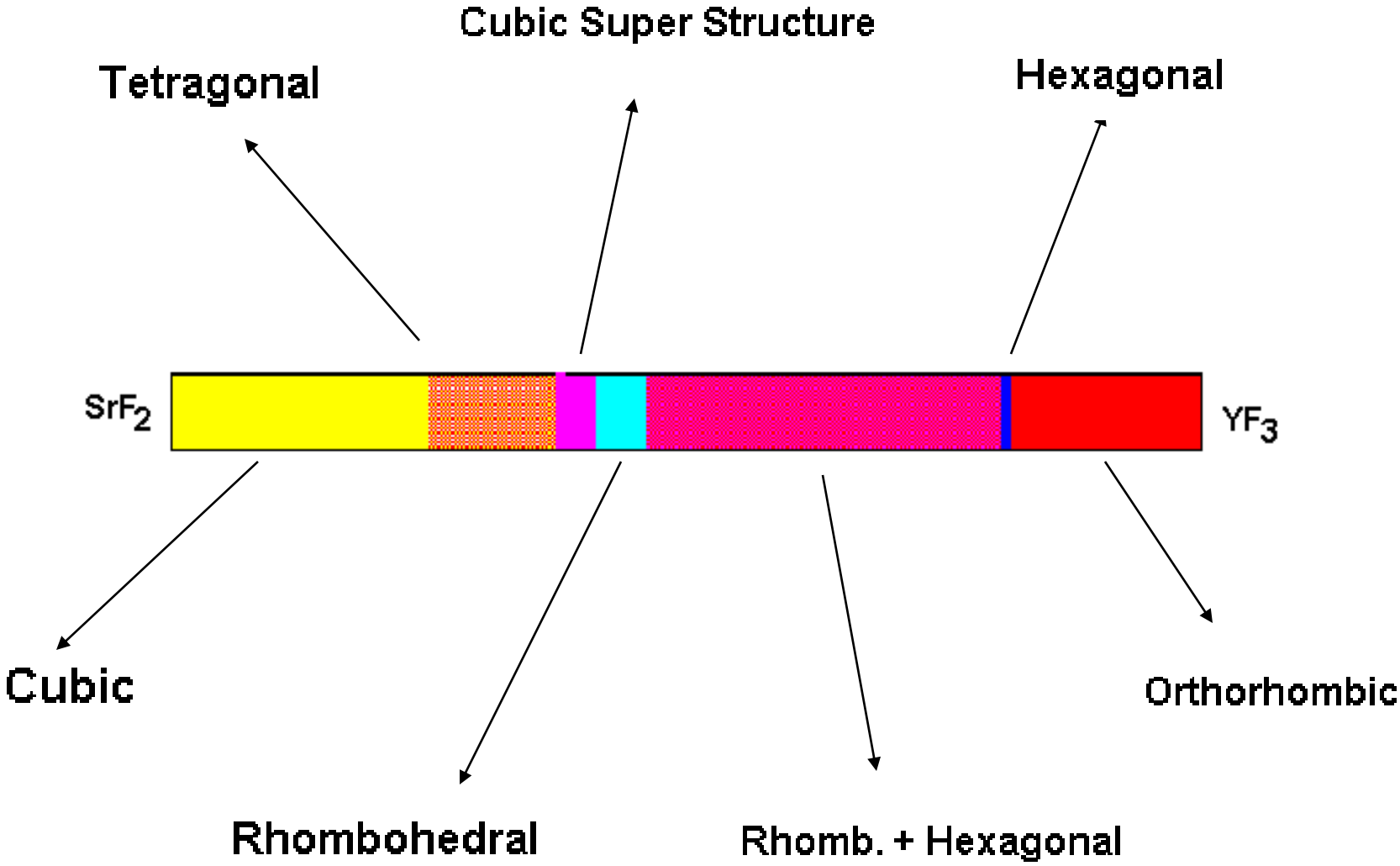


$Th_{0.9}Ce_{0.1}SiO_4$

$ThSiO_4$

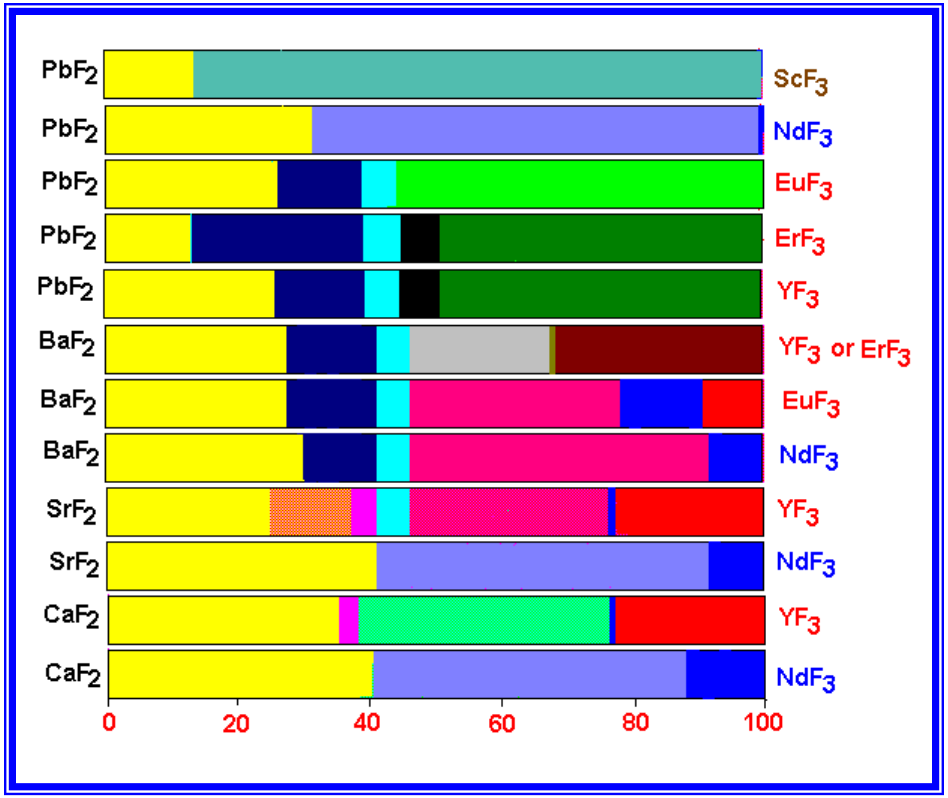
SrF_2-YF_3 SYSTEM





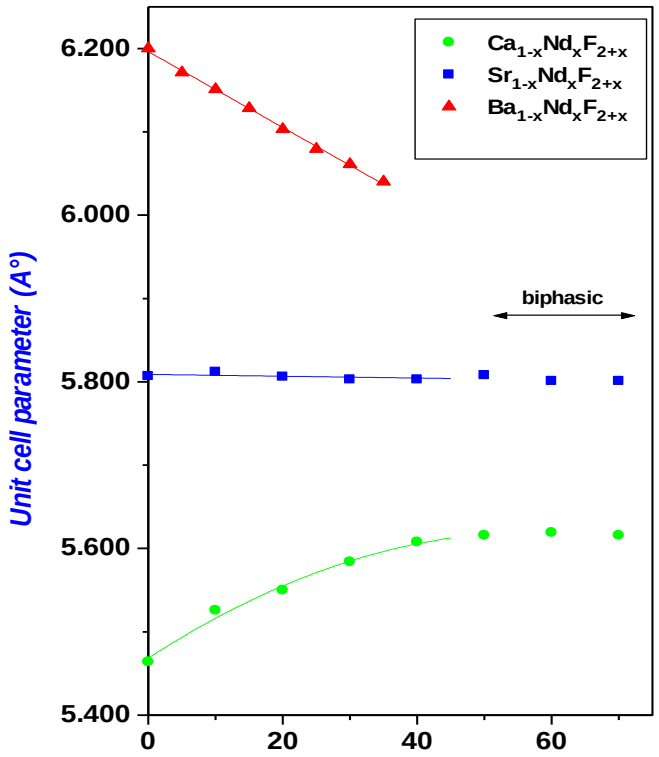
Phase fields present in SrF_2-YF_3 system

MF_2-$M'F_3$ Phase Relation



- Fluorite type S.S. (Cubic - I)
- Fluorite + Tysonite
- C.S. + Tysonite
- Tetragonal - I
- Cubic - I + Rhomb.
- M_2BaF_8 (Mono.)
- Rhomb. + M_2BaF_8
- Rhomb. + MF_3 (Ortho.)
- Tysonite or Tysonite type S.S. (Hexa.)
- Cubic - II (C.S.)
- Tysonite + MF_3 (Ortho.)
- Rhombohedral
- Rhomb. + Tysonite
- M_2BaF_8 + MF_3 (Ortho.)
- Tetragonal - II
- Fluorite + ScF_3

Solubility limit of a guest ion in a host lattice

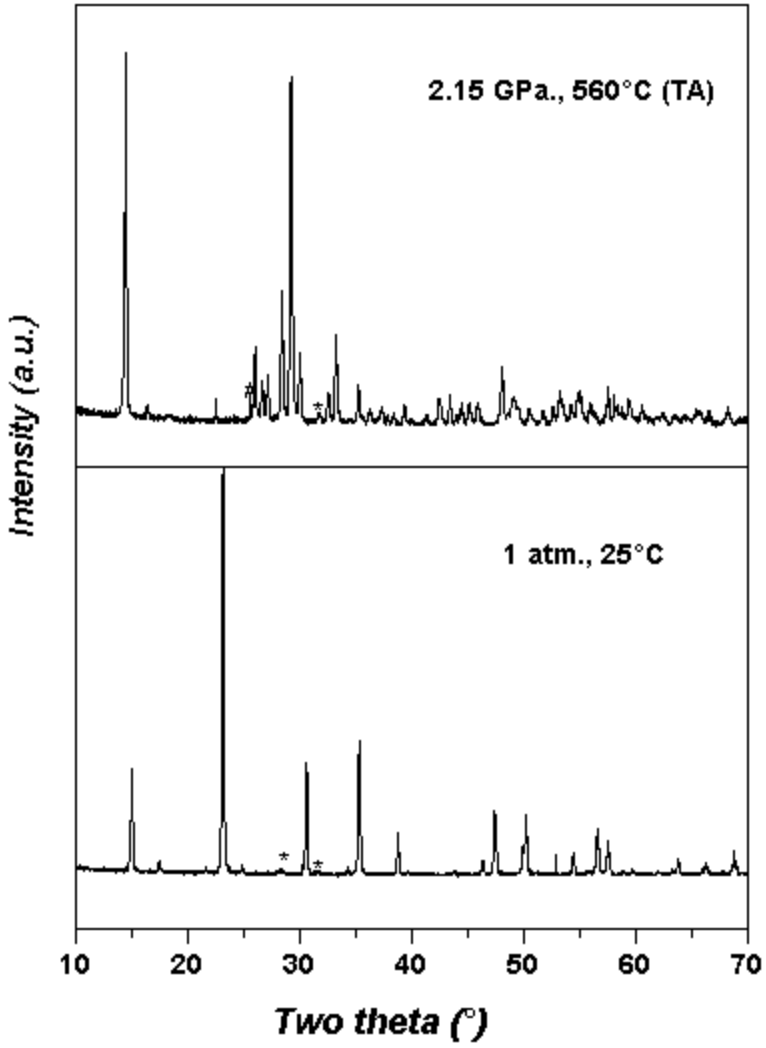


MF_2 **NdF_3**

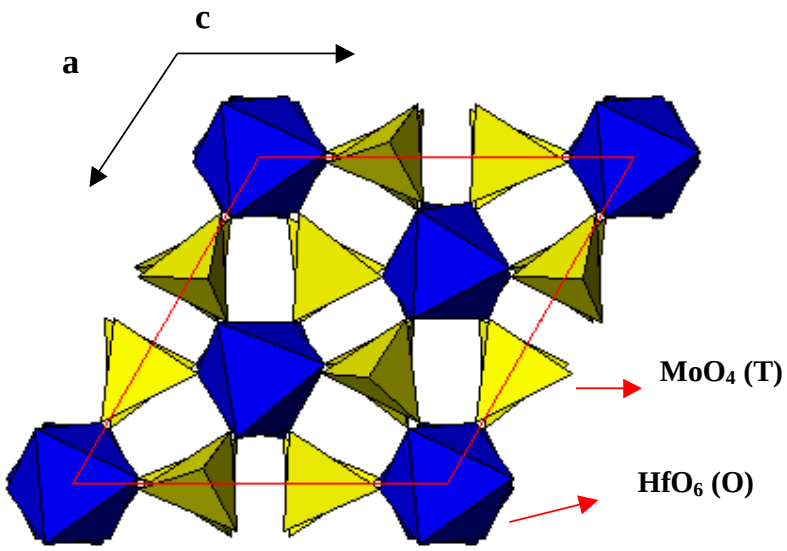
Powder XRD pattern of $HfMo_2O_8$

α (below)

β (upper)

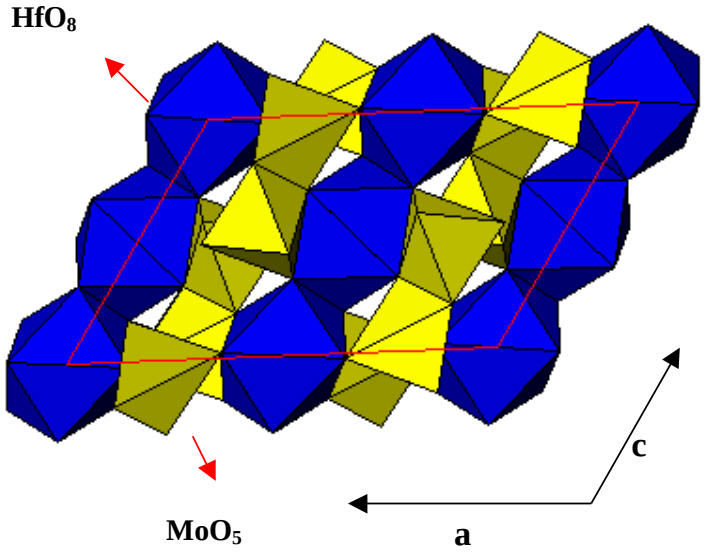


α-$HfMo_2O_8$

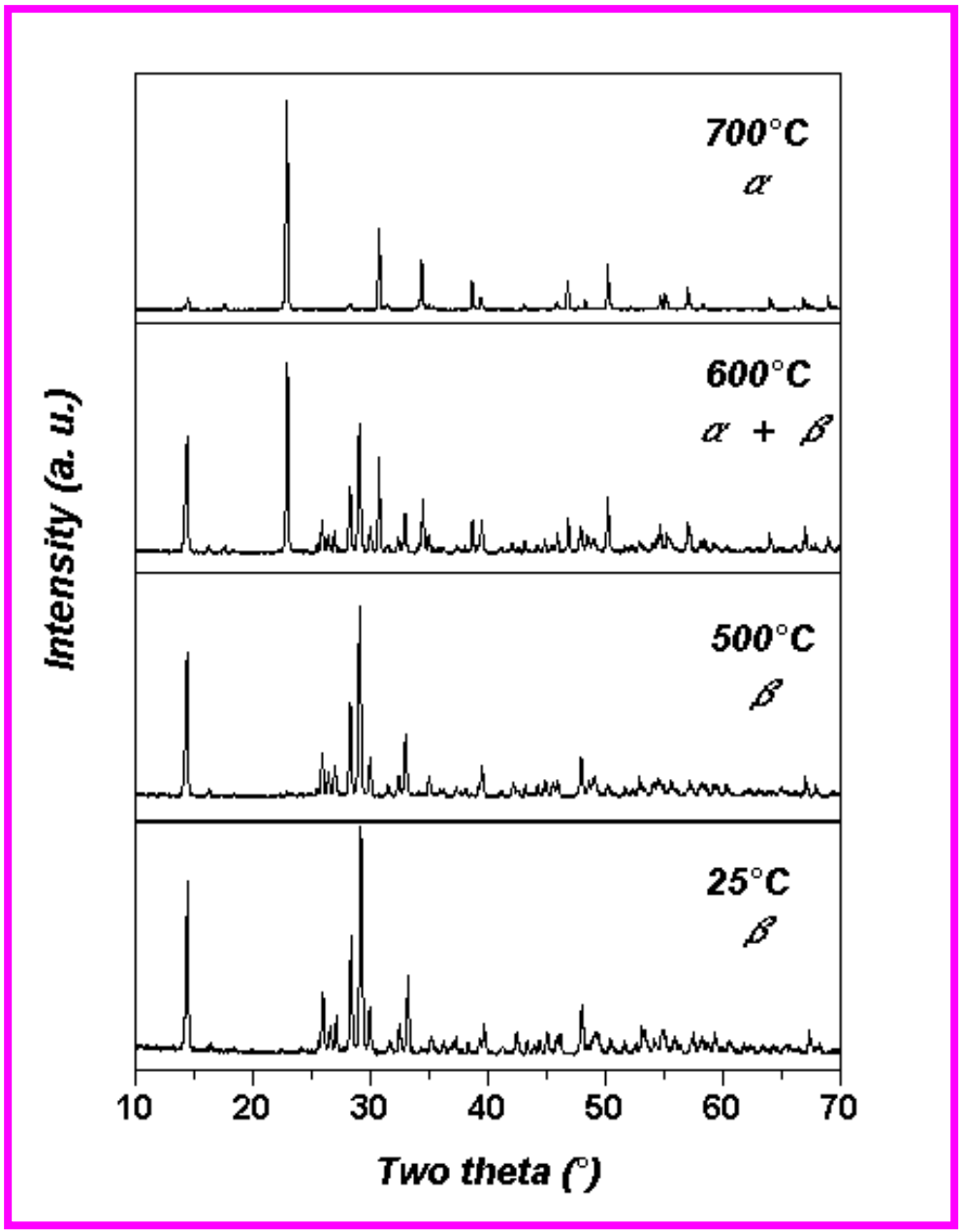


Sp. Gr. P–3 1 C
a = 10.1058(2) & c = 11.7492(3) Å
V = 1039.15(4) $Å^3$, Z = 6

β-$HfMo_2O_8$



Sp. Gr. C 2/c
a = 11.4138(6), b = 7.9105(4), c = 7.4395(3) Å
and β = 122.35(0)°, V = 567.45(5) $Å^3$, Z = 4



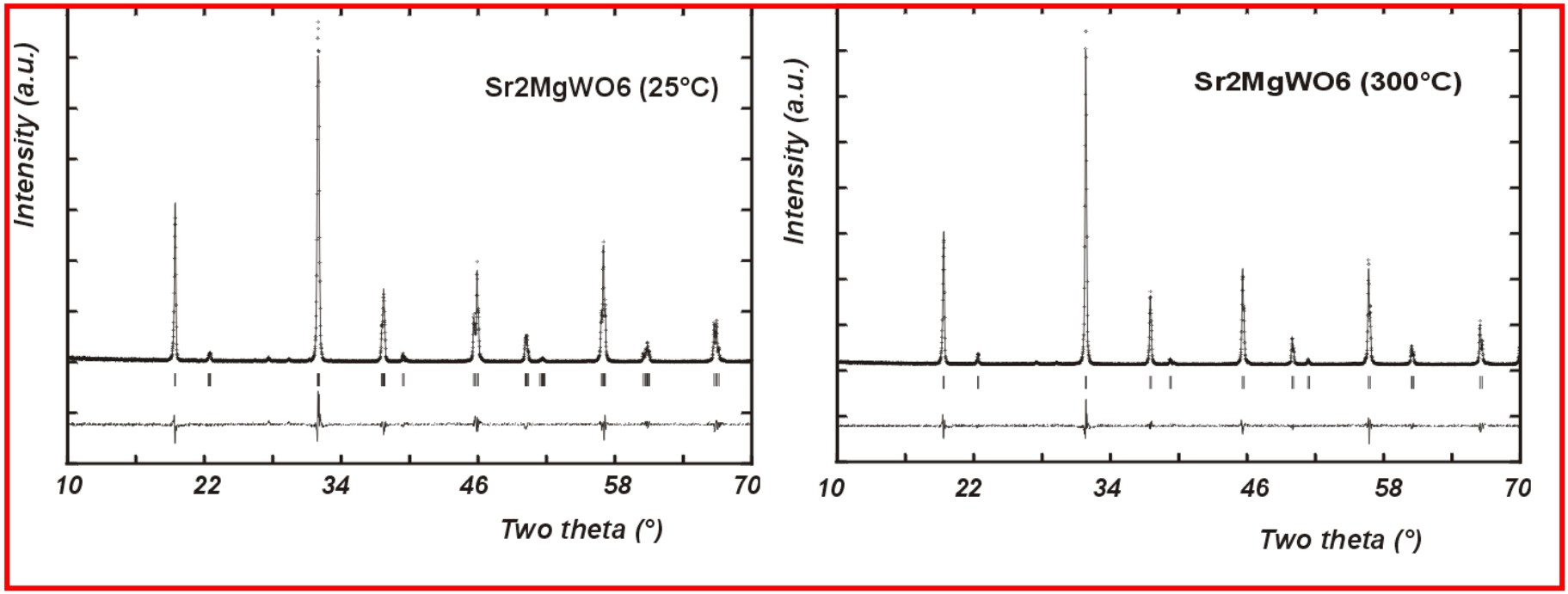
HT-XRD Studies on β-$HfMo_2O_8$

STUDIES IN ThO_2-UO_2 SYSTEM

Temp(K)	ThO_2	ThO_2-2 % UO_2	ThO_2-4 % UO_2	ThO_2-6 % UO_2
293	5.599	5.591	5.589	5.585
423	5.605	5.597	5.595	5.593
573	5.612	5.605	5.603	5.601
723	5.620	5.613	5.611	5.608
873	5.628	5.620	5.618	5.615
1023	5.636	5.628	5.626	5.624
1173	5.644	5.637	5.635	5.634
1323	5.654	5.646	5.644	5.642
1473	5.662	5.653	5.653	5.652
1623	5.671	5.664	5.664	5.662
α_a x 10^6* (293-1623 K)	**9.67**	**9.82**	**10.09**	**10.37**

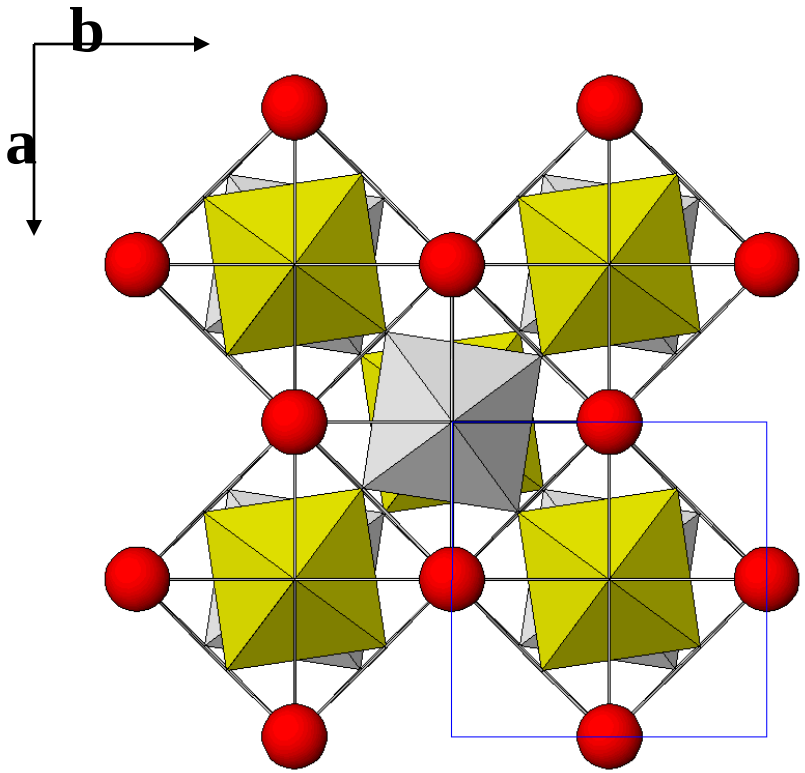
*Unit:K^{-1}

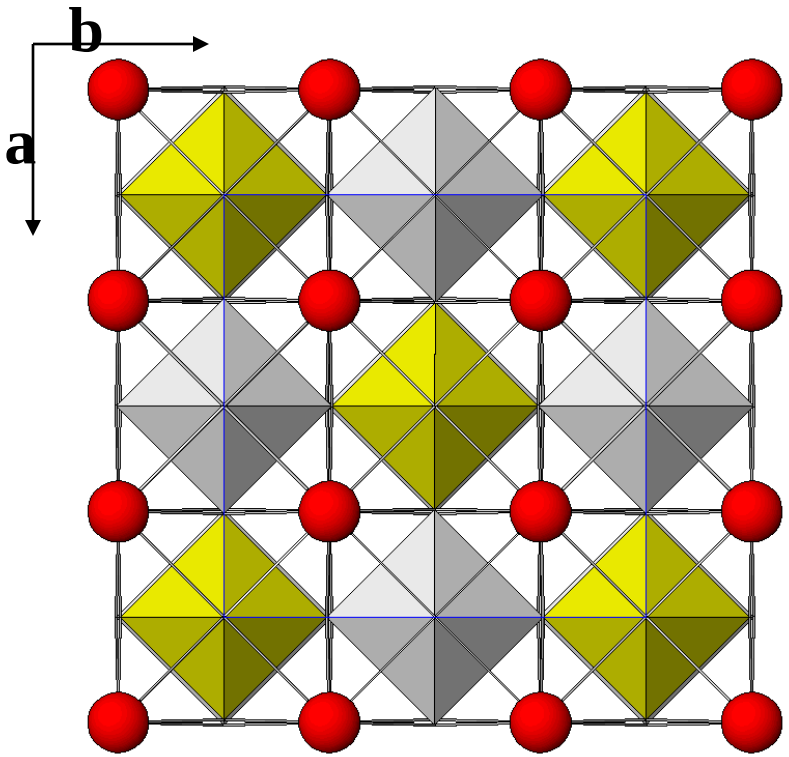
High temperature structural studies on Sr_2MgWO_6



	Sr_2MgWO_6			Ba_2MgWO_6	
Temp (°C)	$\alpha_a \times 10^6$ (/°C)	$\alpha_c \times 10^6$ (/°C)	$\alpha_V \times 10^6$ (/°C)	$\alpha_a \times 10^6$ (/°C)	$\alpha_V \times 10^6$ (/°C)
25-200	16.5	-0.58	32.5	10.7	32.2
25-300	-	-	48.1	11.1	33.2
300-1200	12.1	-	36.5	12.2	37.1
25-1200	-	-	39.6	12.0	36.4

Crystal structure of Sr_2MgWO_6

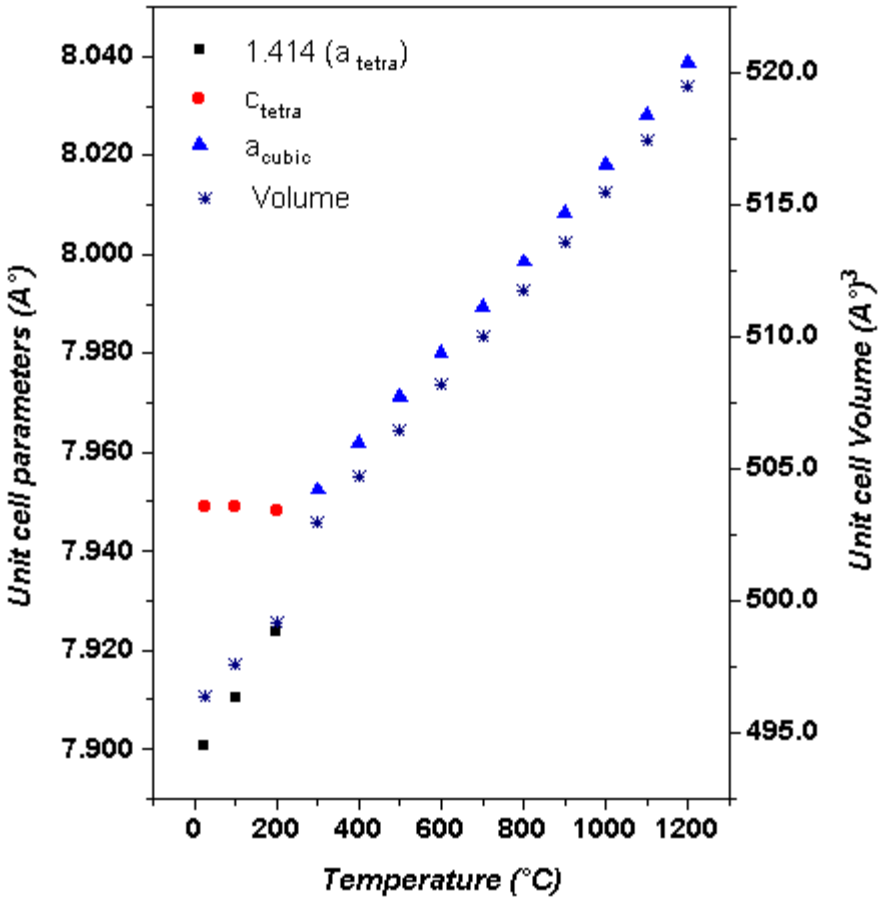




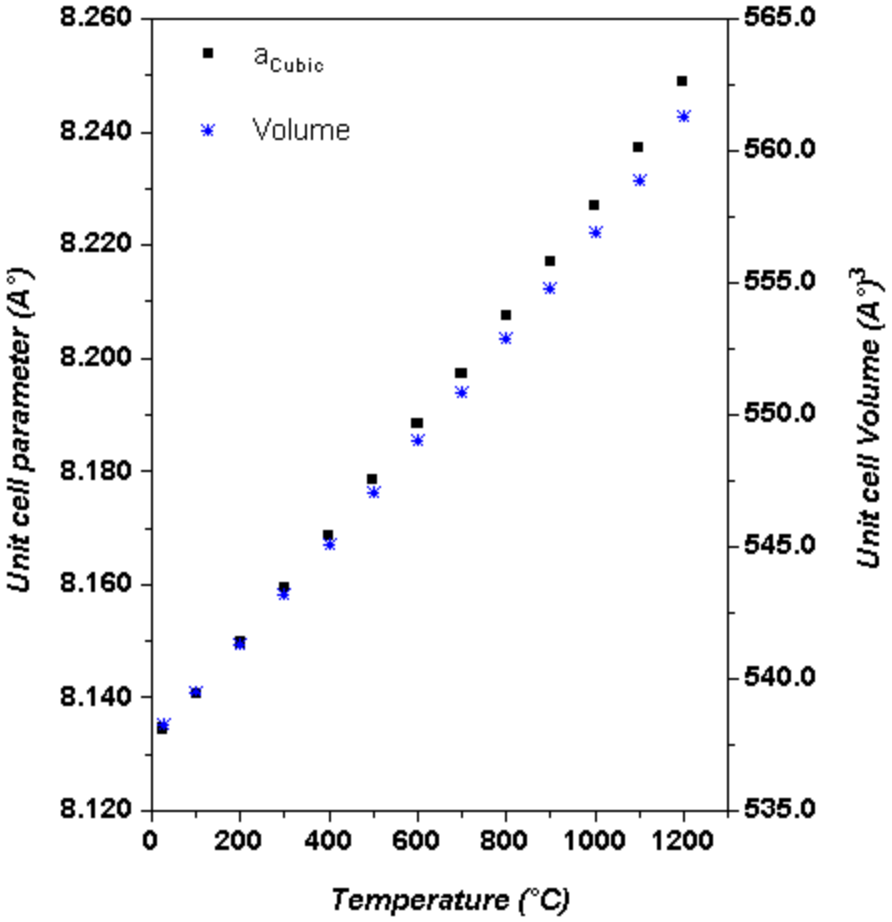
T = 25°C, P = 1 atm.
Tetragonal, Sp.Gr. = I4/m (No. 87),
a = 5.5876(1), c = 9.9490(3) Å
V = 248.18(1) Å^3, Z = 2

T = 300°C, P = 1 atm.
Cubic, Sp.Gr. = Fm3m (No. 225)
a = 5.5876(1) Å V
= 248.18(1) Å^3, Z = 2

Variation of unit cell parameters with Temperature



Sr_2MgWO_6



Ba_2MgWO_6

Crystal strcture of Er_2BaF_8 (Monoclinic)

(900°C/18 hrs)

M'_2BaF_8 (0.68 mol % of $M'F_3$)

Atom	site	x	y	Z	U_{iso} (Å^2)	Occ.
Ba	2a	0	0	0	0.0302(11	1.0
		0	0	0	0.0301(11)	
Er	4h	0	0.67630(8)	0.5	0.0258(8)	1.0
		0	0.67635(8)	0.5	0.0259(8)	
F1	8j	0.1904(6)	0.1399(3)	0.5645(9)	0.029(4)	1.0
		0.1906(6)	0.1399(3)	0.5644(9)	0.027(4)	
F2	4i	0.3859(8)	0	0.2201(12)	0.020(2)	1.0
		0.3859(8)	0	0.2200(12)	0.020(2)	
F3	4g	0	0.7452(5)	0	0.043(6	1.0
	8j	0.0353(18)	0.7454(6)	0	0.021(3)	0.5

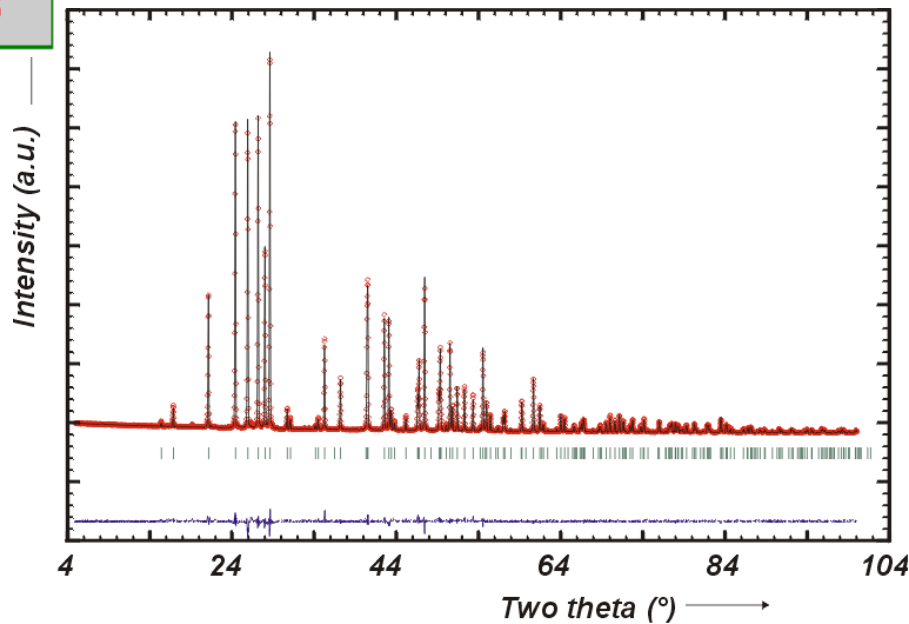
Orthorhombic: Pnma

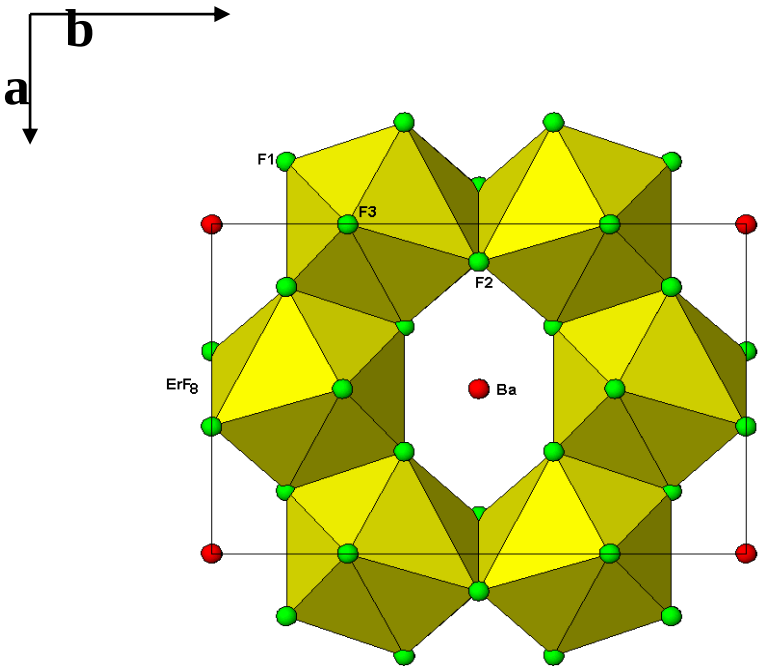
Monoclinic : C2/m

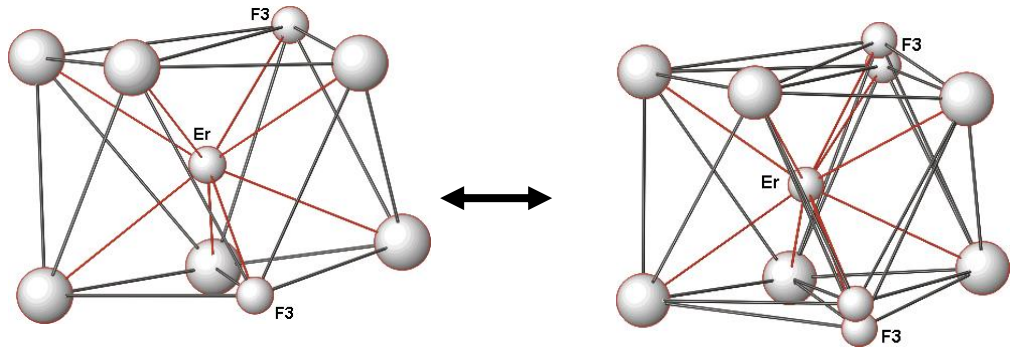
Sp. Gr. C2/m (No. 12)

a = 6.9620(2), b = 10.4860(3)
c = 4.2541(1) Å and β = 99.6846(5)°
V = 306.14(1) Å^3, Z = 2

	R_B	R_F
Ord.	3.9 %	3.6 %
Disord.	4.0 %	3.6 %

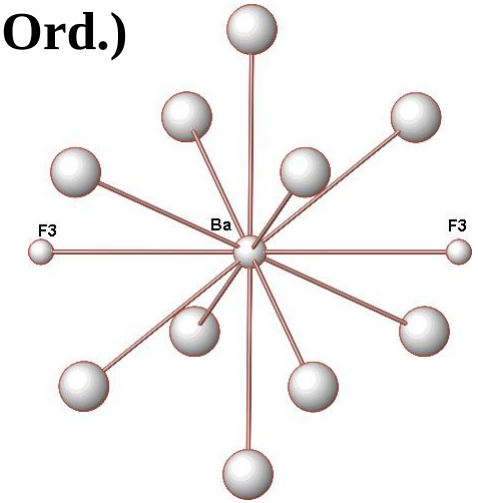




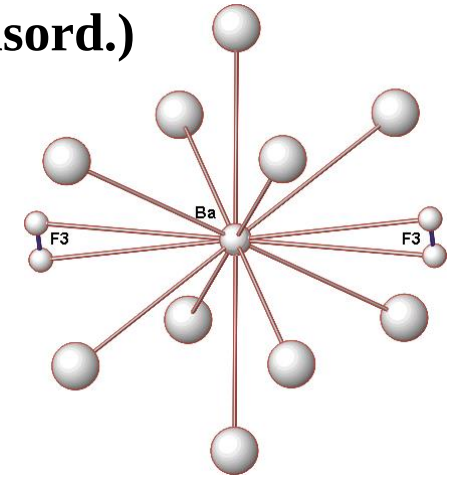


(Ord.) ErF_8 (Disord.)

(Ord.)



(Disord.)



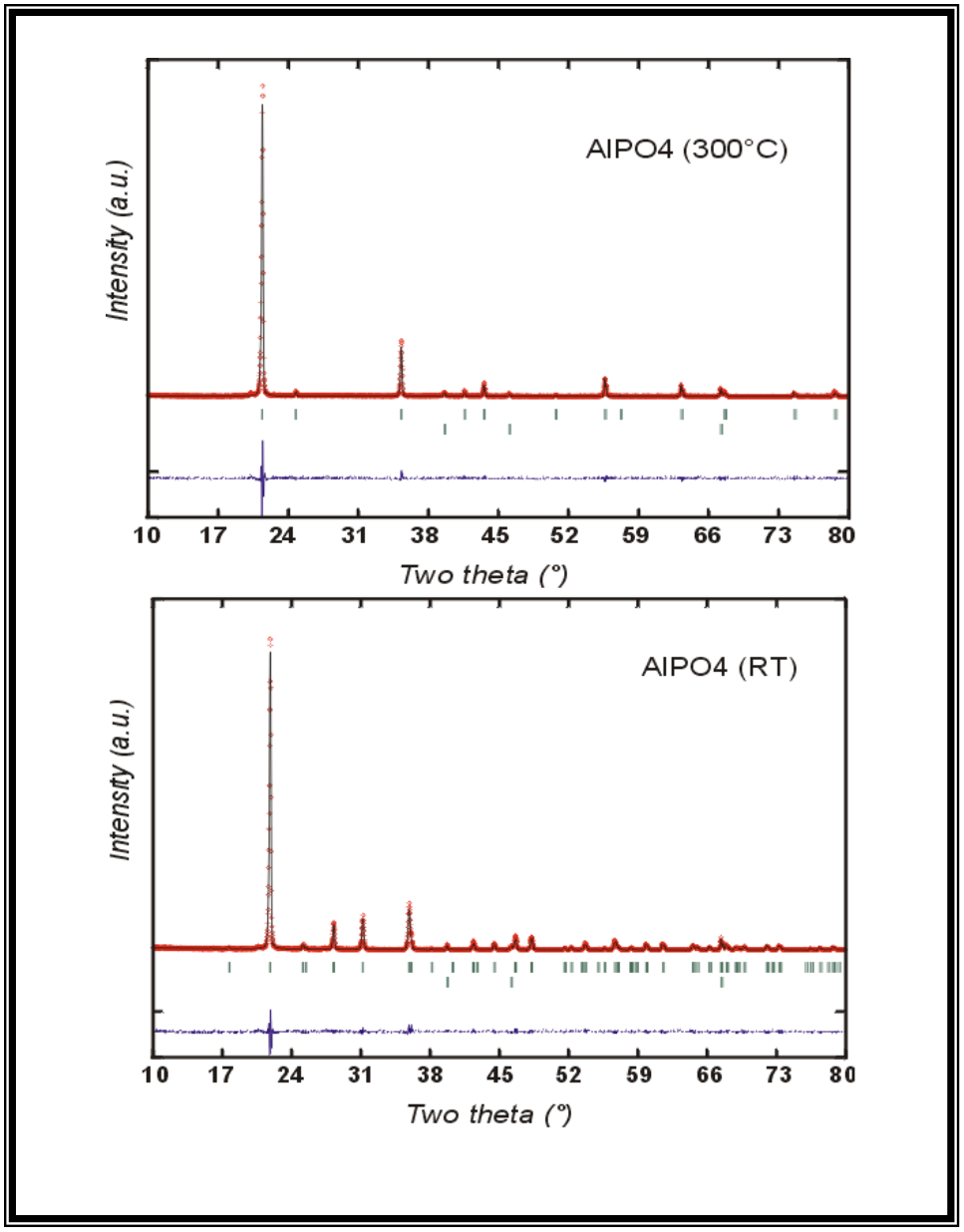
BaF_{12}

HT-XRD STUDIES ON $Al_{1-x}Ga_xPO_4$

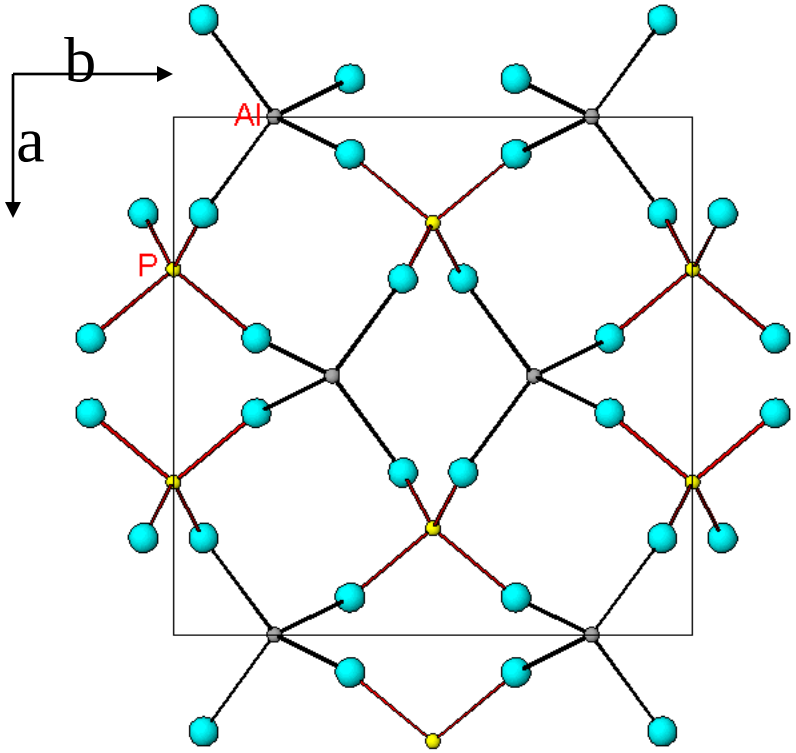
β-$AlPO_4$ (Cubic)

Powder XRD pattern of cristobalite type $AlPO_4$

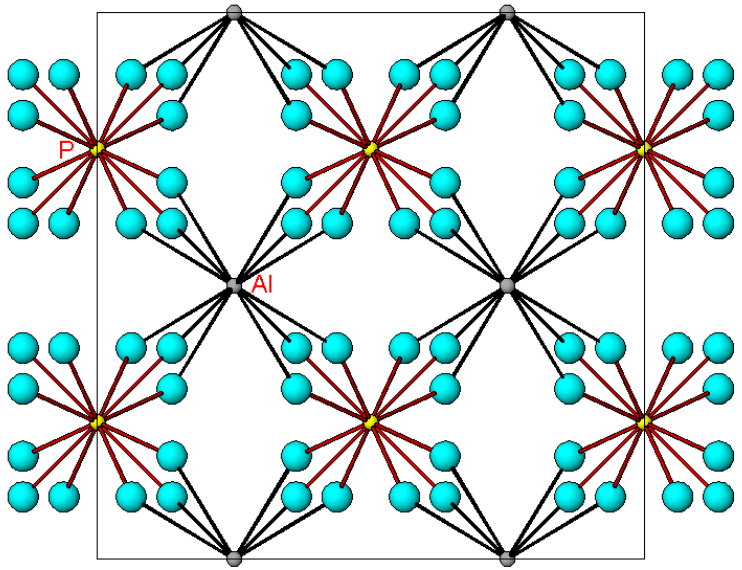
α-$AlPO_4$ (Ortho.,)



Crystal structure of cristobalite type $AlPO_4$



T= 25°C, 1Atm.



T= 300°C, 1Atm.

Low Cristobalite type

S.Gr. $C222_1$

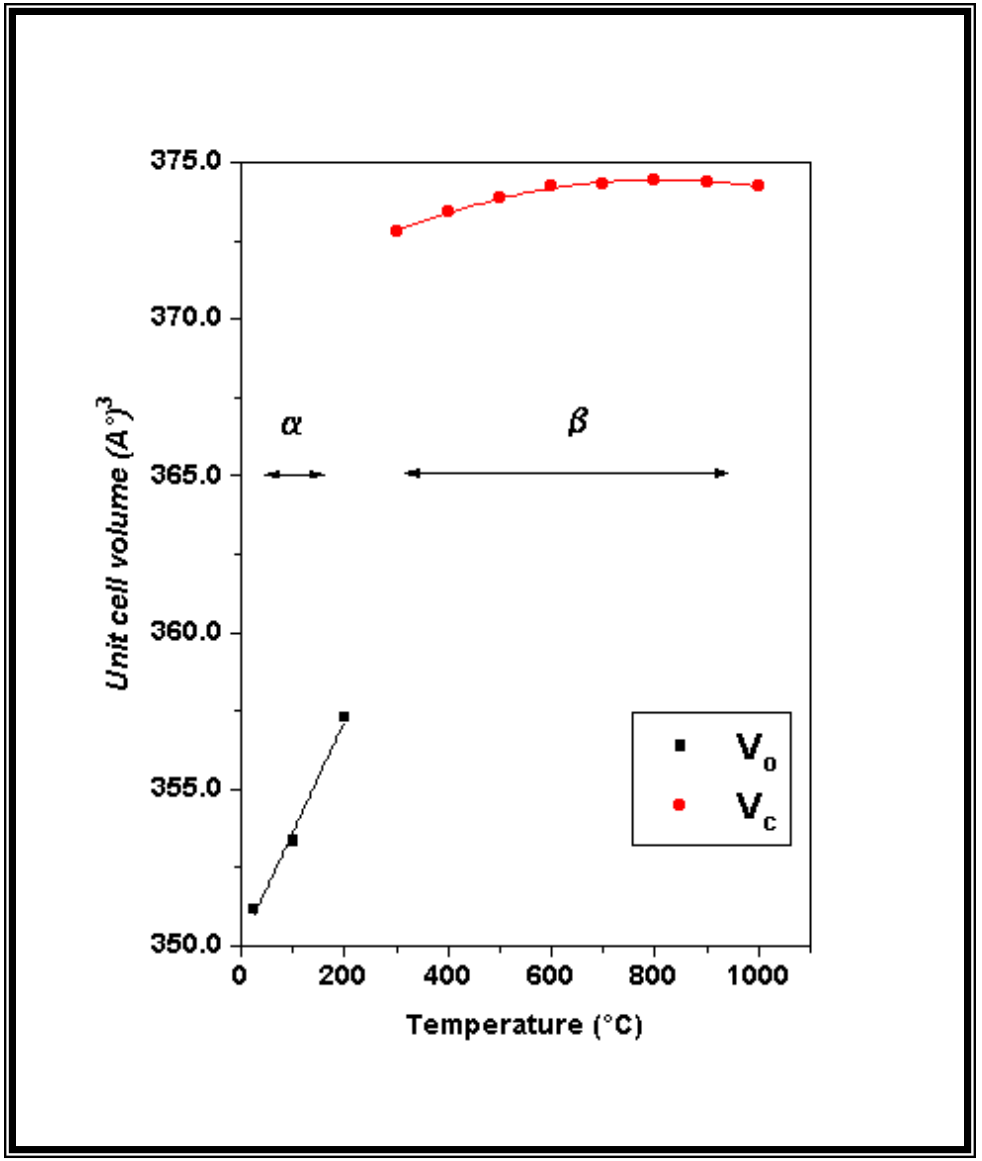
7.0843(14), 7.0823(13), 6.9989(4)Å
V = 351.22(1) $Å^3$, Z = 4

High Cristobalite type

S.Gr. F4-3m

a = 7.1969(2) Å, V = 372.77(1) $Å^3$
Z = 4

Variation of unit cell Volume of $AlPO_4$ with Temperature



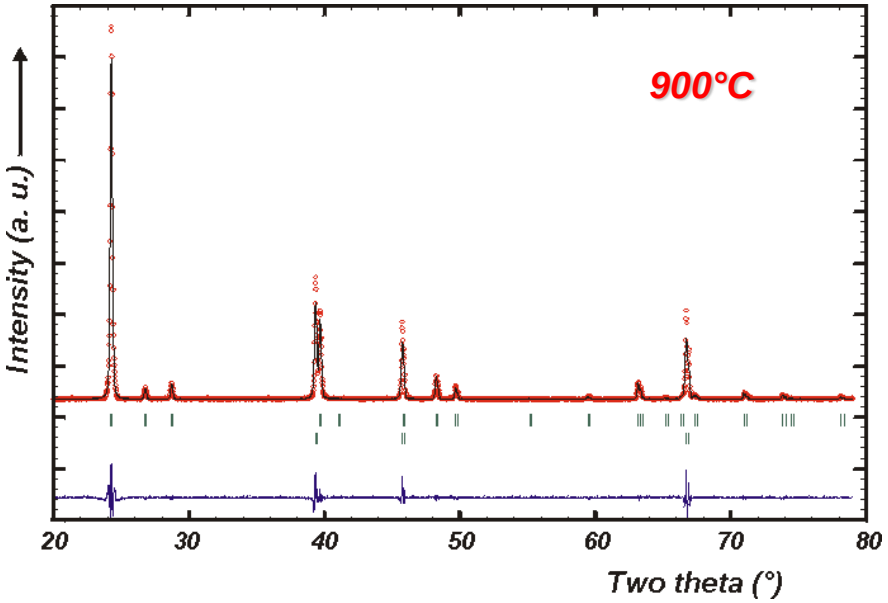
HT-XRD STUDIES ON BPO_4 (Tetragonal, cristobalite type)

a = 4.3447(2) Å

c = 6.6415(5) Å

V = 125.37(1) Å^3

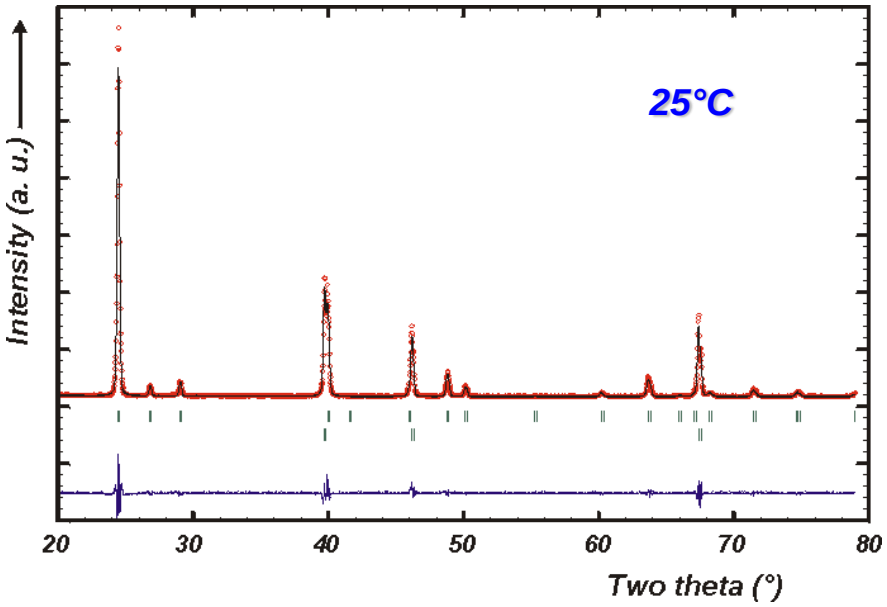
Rietveld refinement plot of powder XRD



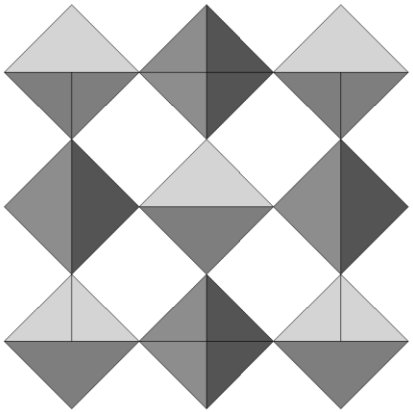
a = 4.3939(2) Å

c = 6.6539(6) Å

V = 128.46(1) Å^3



Ideal C9

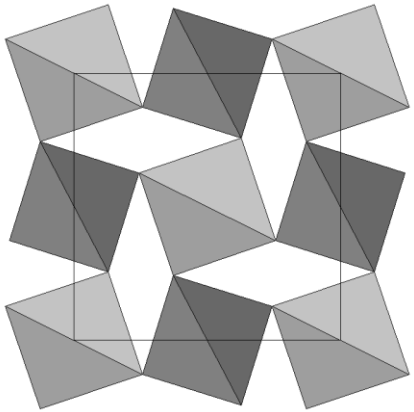


T

P

$\phi = 0°$

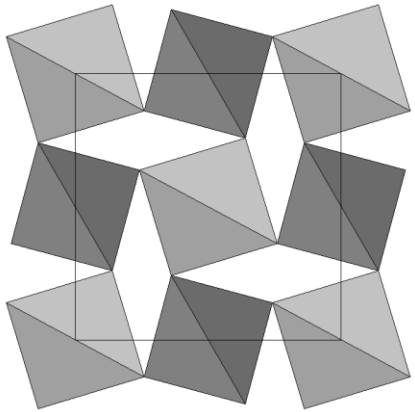
BPO_4 (900°C)



T

P

BPO_4 (25°C)

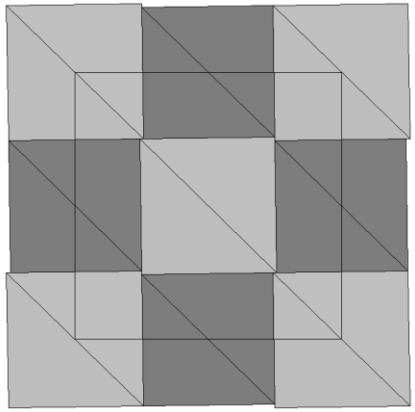


$\phi = 27°$

$\phi = 29°$

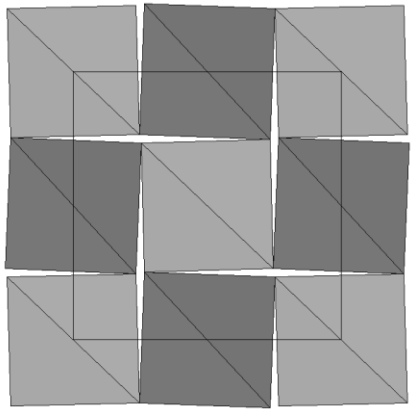
P

T



P

T



$\phi = 45°$

$ZnCl_2$

BPO_4 (50 GPa)

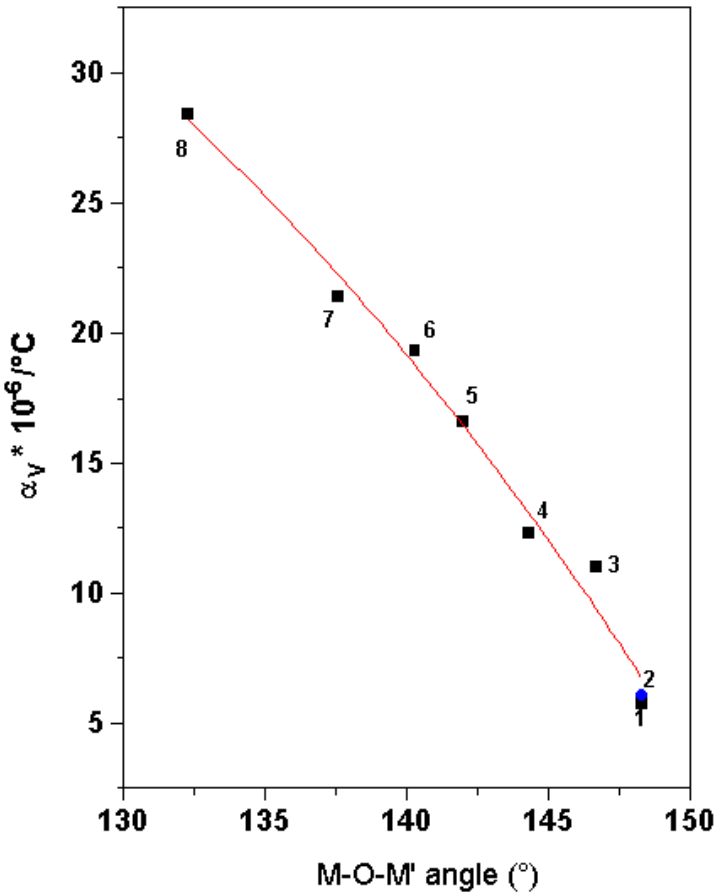
$\phi = 43°$

Transformation topology for cristobalite frame with Temp. /Press.

Variation thermal expansion coefficients with inter-polyhedral angle of *Cristobalite* type compounds

$$\alpha_V\ (/°C) = -191.32 + 4.33 \times [\theta] - 0.02 \times [\theta]^2$$

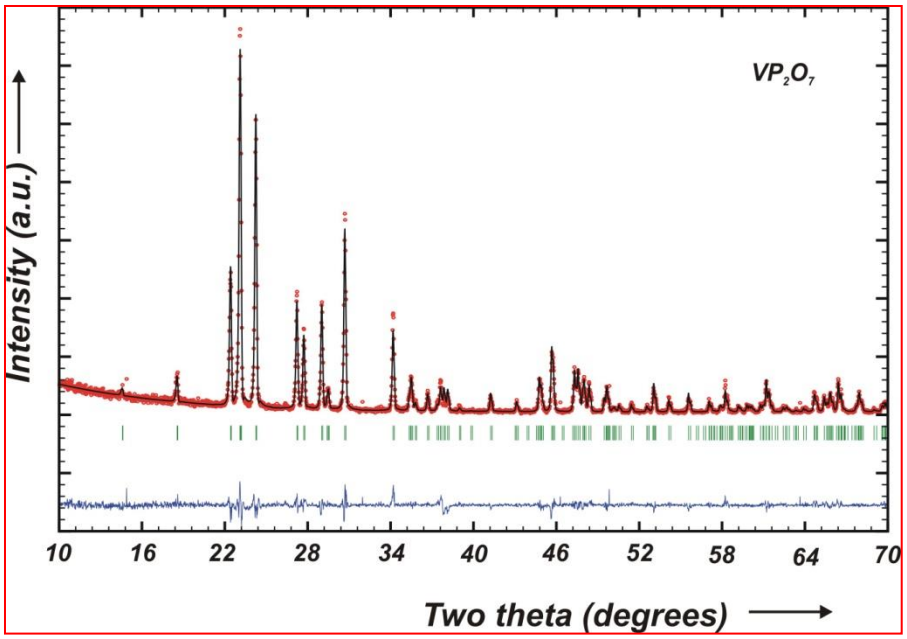
1. $AlPO_4$ (at 300°C)
2. $AlPO_4$ (at 300°C) *(lit. data)*
3. SiO_2 (at 300°C) *(lit. data)*
4. $Al_{0.8}Ga_{0.2}PO_4$ (300°C)
5. $Al_{0.5}Ga_{0.5}PO_4$ (400°C)
6. $Al_{0.2}Ga_{0.8}PO_4$ (600°C)
7. $GaPO_4$ (700°C)
8. BPO_4 (25°C)



Crystal structure of α-$VO(PO_3)_2$

Space Group: C2/c (No. 15)

a = 15.0924(4)
b = 4.1919(1)
c = 9.5706(3) Å
β = 126.464(2)°
V = 486.95(3) Å^3



Atom	site	x	y	z
V	4e	0	0.2551(4)	0.25
P	8f	0.3290(1)	0.0067(4)	0.8468(2)
O1	8f	0.4105(4)	0.159(1)	0.8307(8)
O2	8f	0.2376(4)	0.253(1)	0.8061(5)
O3	8f	0.3674(5)	-0.147(1)	0.0125(5)
O4	4e	0	-0.1342(4)	0.25

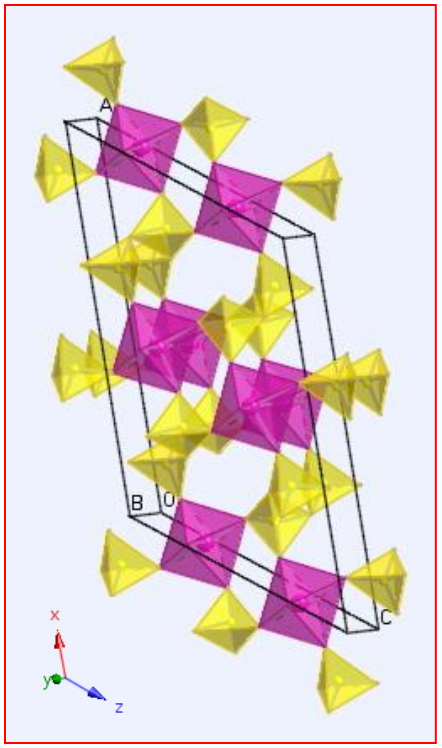
R_p: 10.8 %, R_{wp}: 14.9 %, R_{exp}: 11.5 %

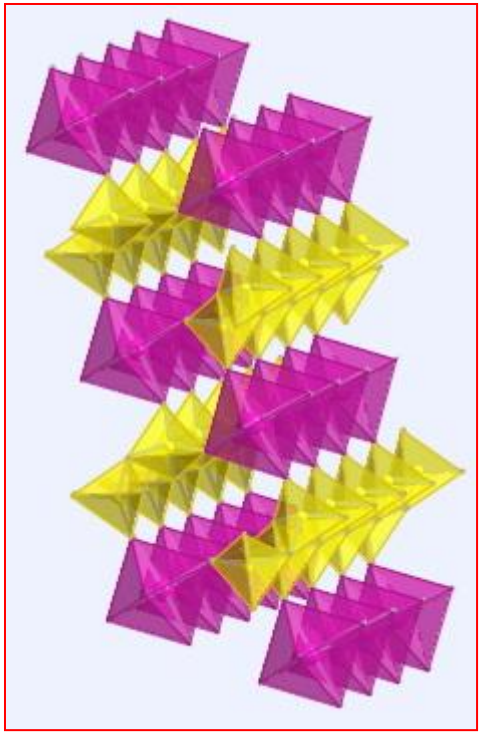
χ^2: 1.68

R_B: 6.04 %, R_F: 5.19 %

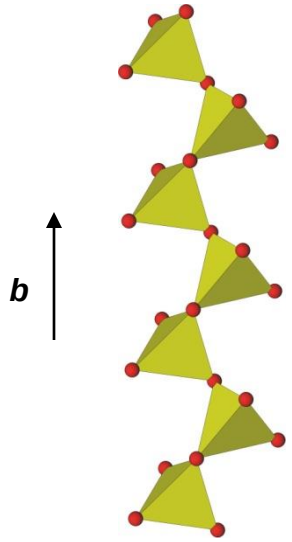
$B_{overall}$ = 1.83(7) Å^2

Crystal structure of α-$VO(PO_3)_2$



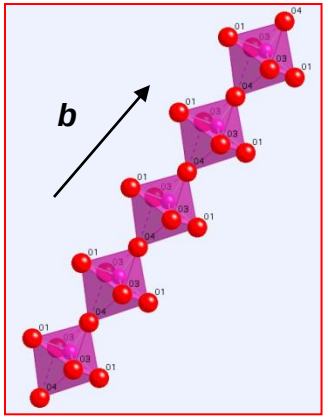


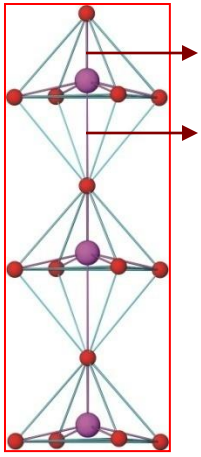
PO_3 chains



Structural features of $VO(PO_3)_2$

VO_5 chains

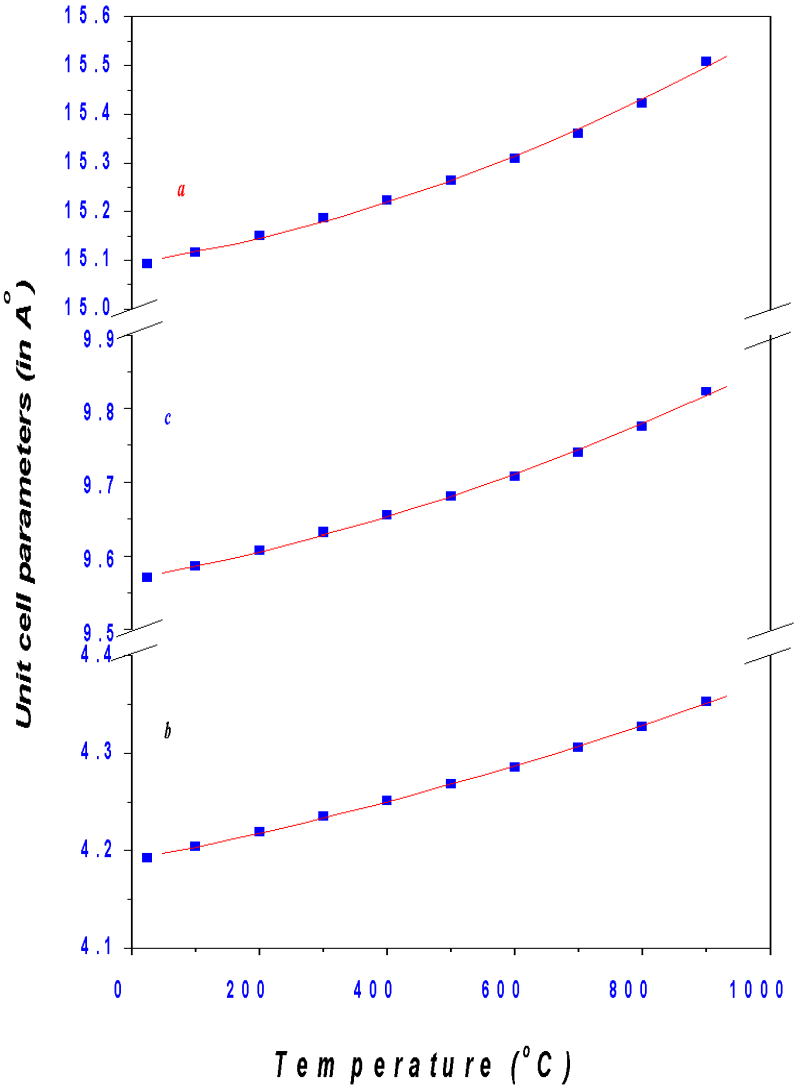




Variation of unit cell parameters with temperature

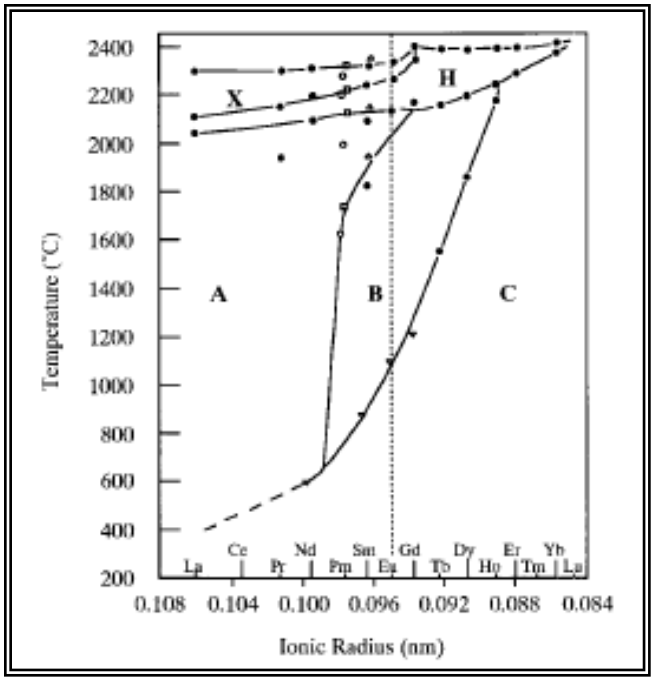
Coefficients of thermal expansion (/°C)

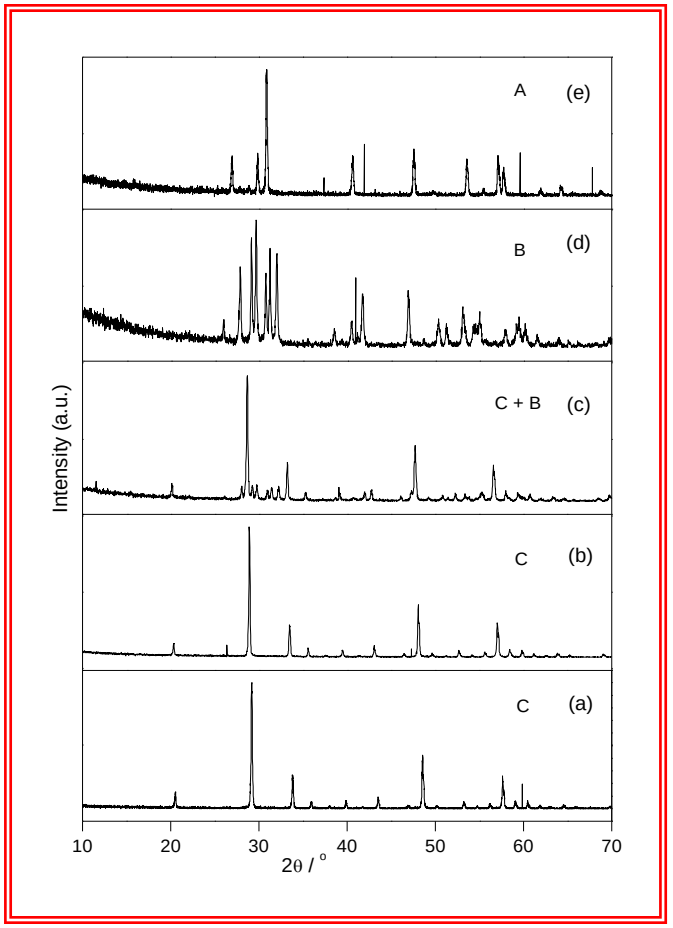
$\alpha_a = 31.4 \times 10^{-6}$
$\alpha_b = 43.8 \times 10^{-6}$
$\alpha_c = 30.2 \times 10^{-6}$
$\alpha_V = 87.5 \times 10^{-6}$



Crystal structure of stabilized monoclinic phase of $Nd_{2-x}Y_xO_3$

Most common crystal structure of Rare-earth sesquioxides are hexagonal monoclinic and cubic

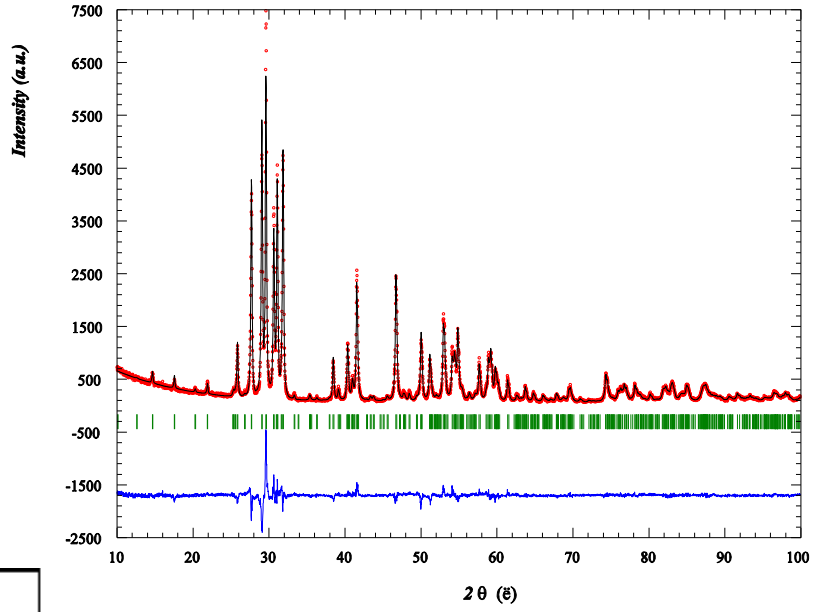




C = C-type cubic, B = Monoclinic, A = Hexagonal

Crystal structure of stabilized monoclinic phase of $Nd_{2-x}Y_xO_3$

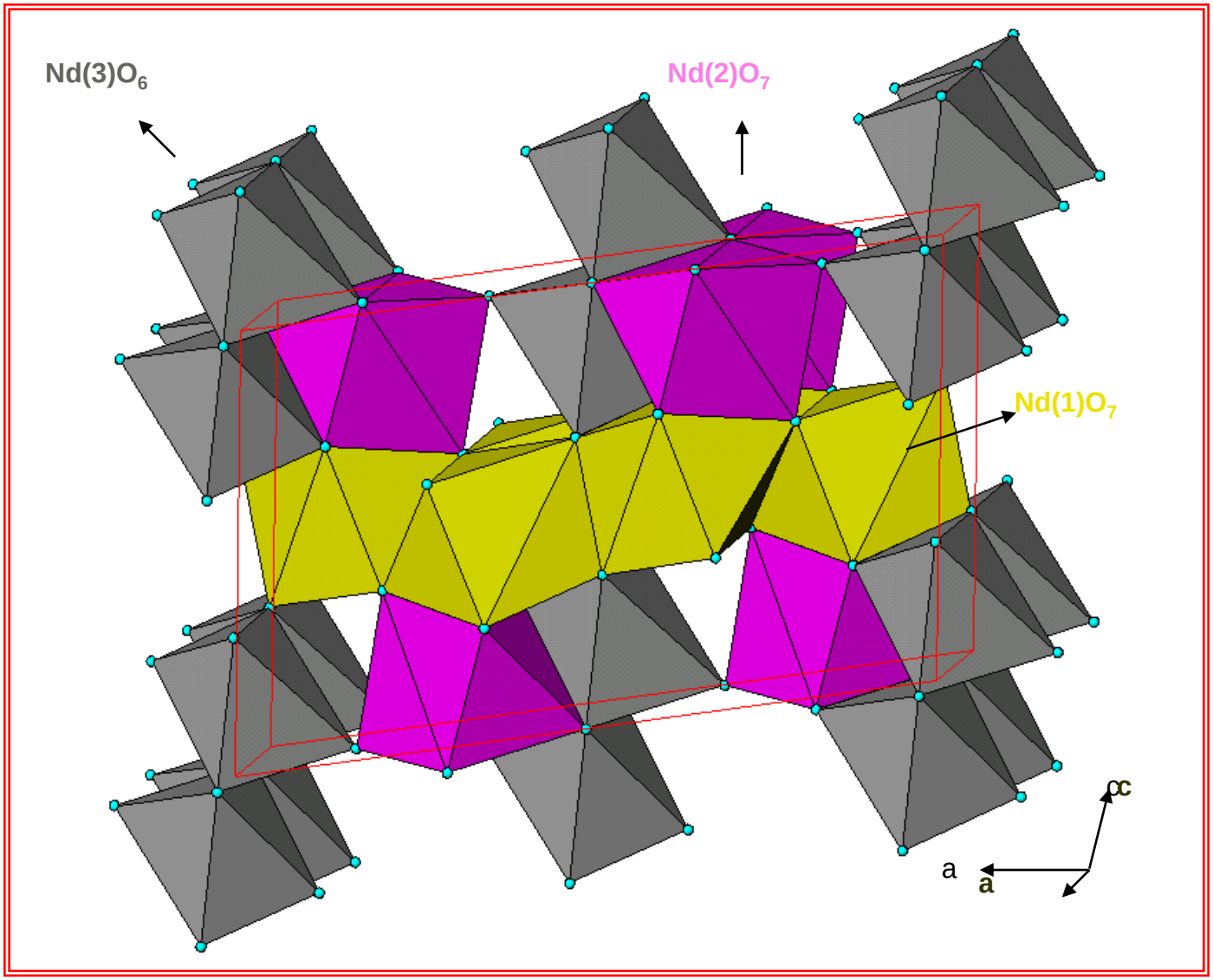
Space Group: C2/m (12)
a = 14.2761(3), b = 3.6449(1), c = 8.9022(2) Å, β = 100.391(2)°
V = 455.62(2) Å³, Z = 6



Atoms	site	x	y	z	B_{iso} (Å)^2	occ.
Nd1	4i	0.1336(2)	0.5	0.4922(2)	0.75(7)	1
Nd2	4i	0.1885(2)	0.5	0.1379(2)	0.24(8)	1
Nd3	4i	0.4651(2)	0.5	0.1884(3)	0.72(6)	0.83(2)
Y	4i	0.4651(2)	0.5	0.1884(3)	0.72(6)	0.17(2)
O1	4i	0.1220(6)	0	0.2857(14)	0.6(2)	1
O2	4i	0.3279(5)	0.5	0.0270(10)	0.6(2)	1
O3	4i	0.2932(7)	0.5	0.3729(10)	0.6(2)	1
O4	4i	0.4795(7)	0.0	0.3416(11)	0.6(2)	1
O2	2b	0.0	0.5	0.0	0.6(2)	1

Y selectively occupy the six coordinated site of B-type rare-earth oxide lattice

Crystal structure of stabilized monoclinic phase of $Nd_{2-x}Y_xO_3$



Crystal structure thorium metavanadate

Crystal system: Tetragonal

Space Group: I 4_1/a (No. 88)

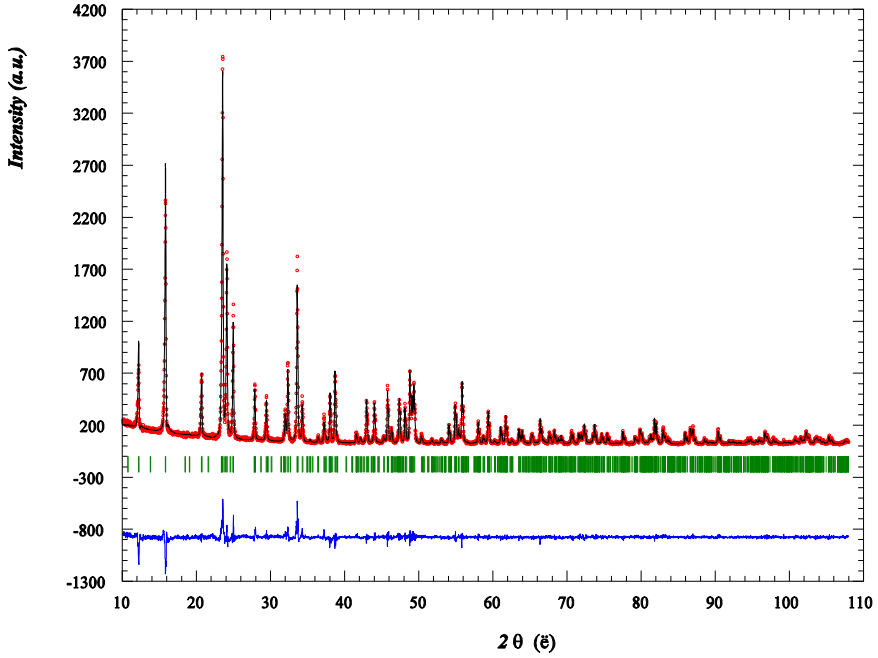
a = 8.5771(1) Å

c = 28.9739(4) Å

V = 2131.51(4) Å3

Z = 8

Density (x-ray) : 3.91 g/cc

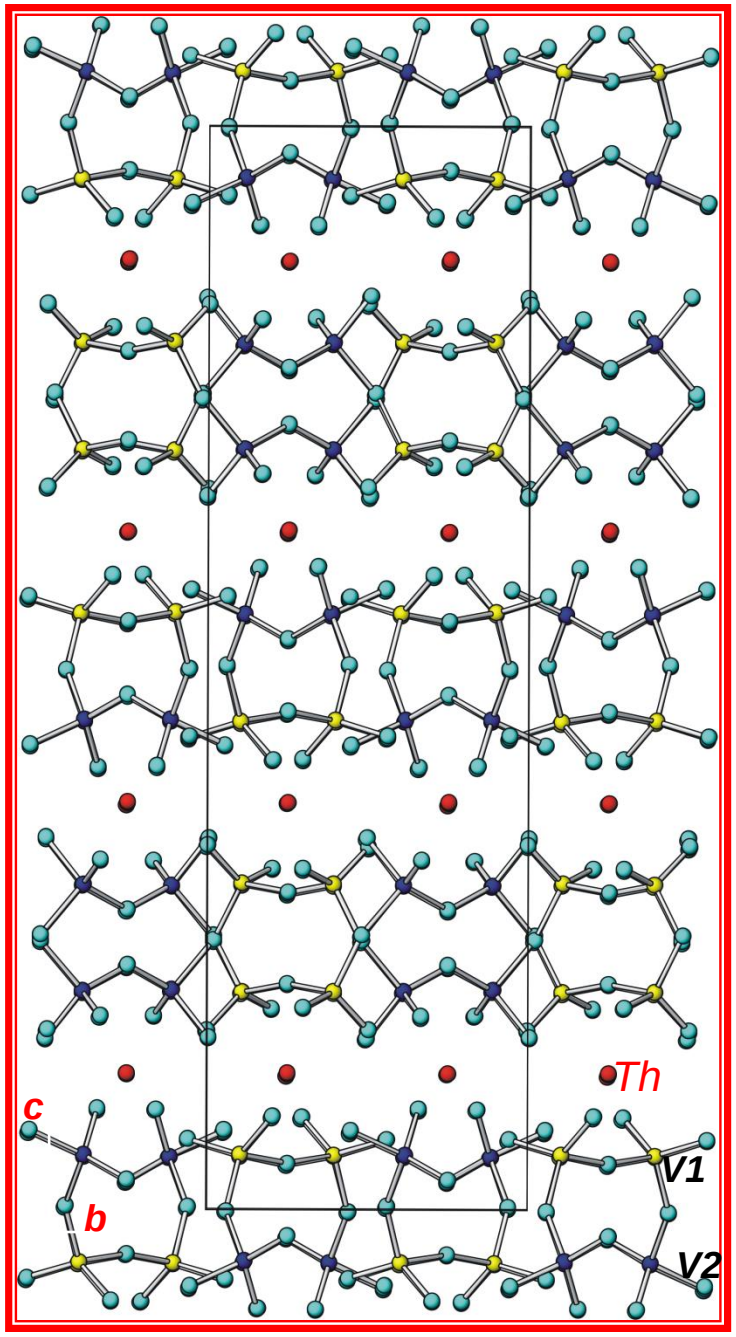


Rp = 10.1 %, Rwp = 12.9 %, (χ2) = 1.8 %
RB = 7.04 %, RF = 4.62 %

Structural formula

ThV(1)2V(2)2O(1)O(2)O(3)2O(4)2O(5)2O(6)2(O7)2

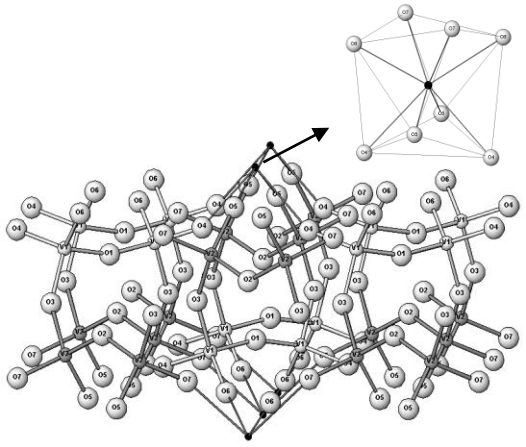
Thorium have eight coordinated polyhedra and Vanadium have tetrahedral coordination

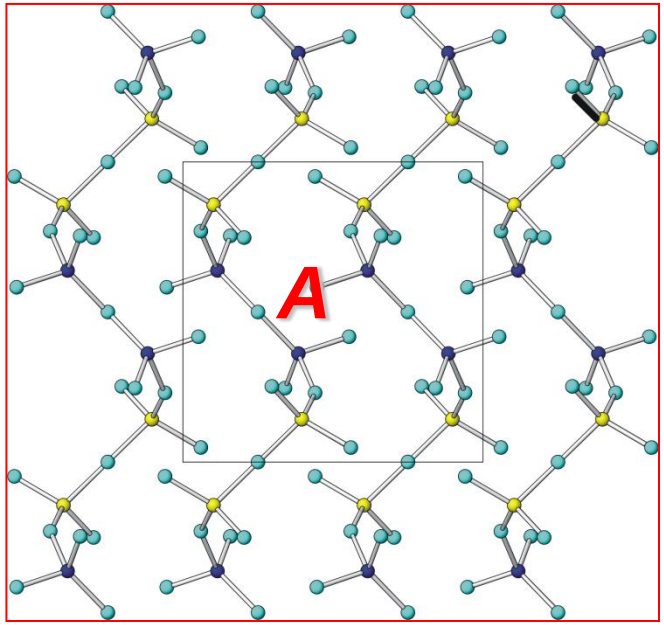


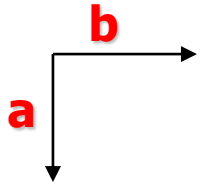
Crystal structure of $Th(VO_3)_4$

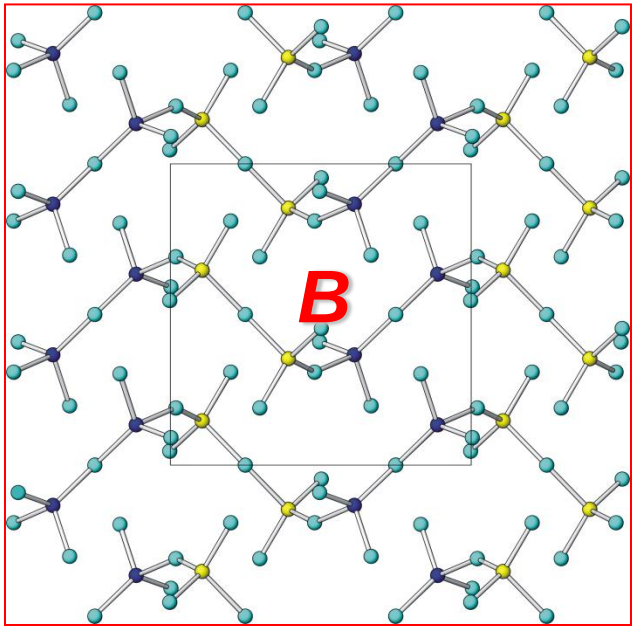
(projected along 001 direction)

Thorium atoms are sandwiched between the layers of VO_3 chains

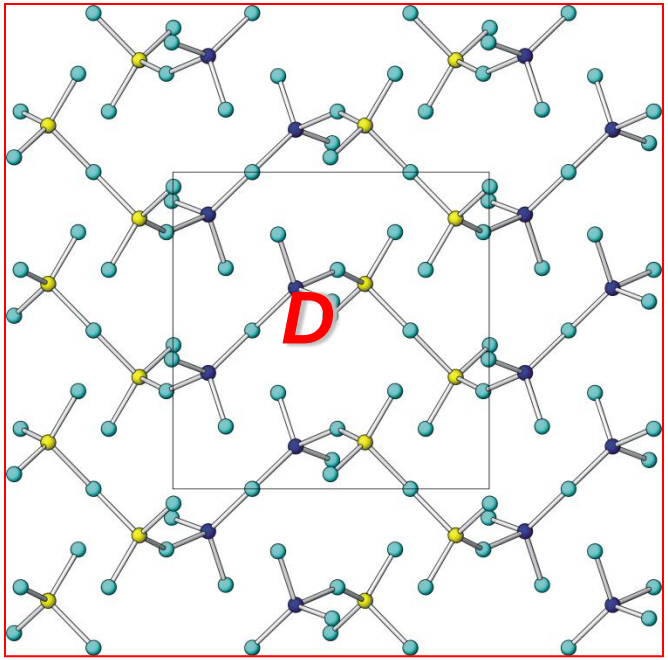


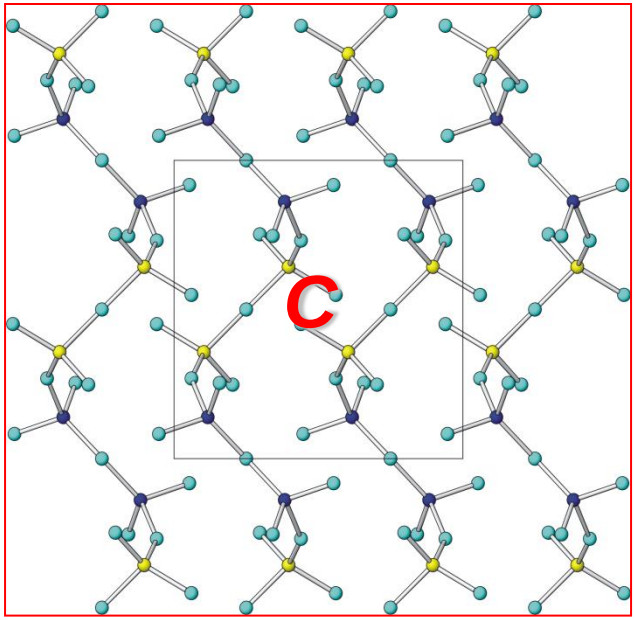




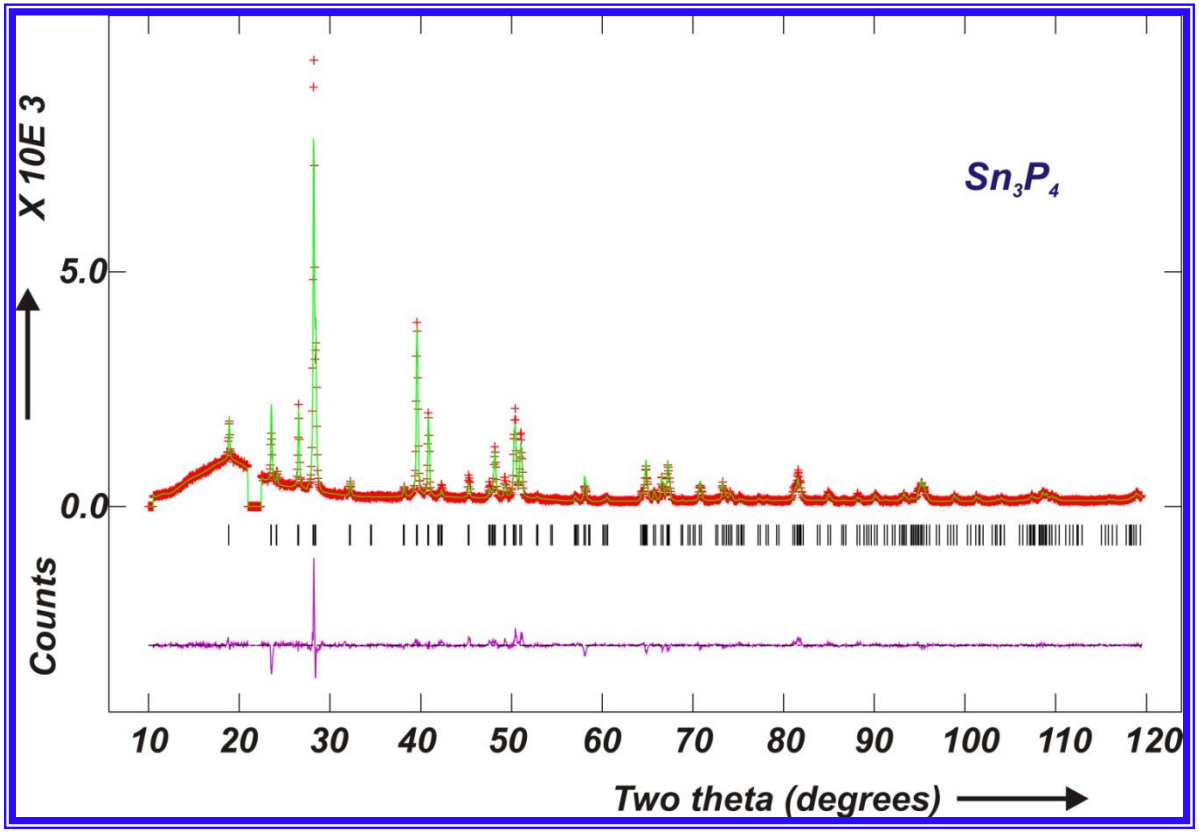


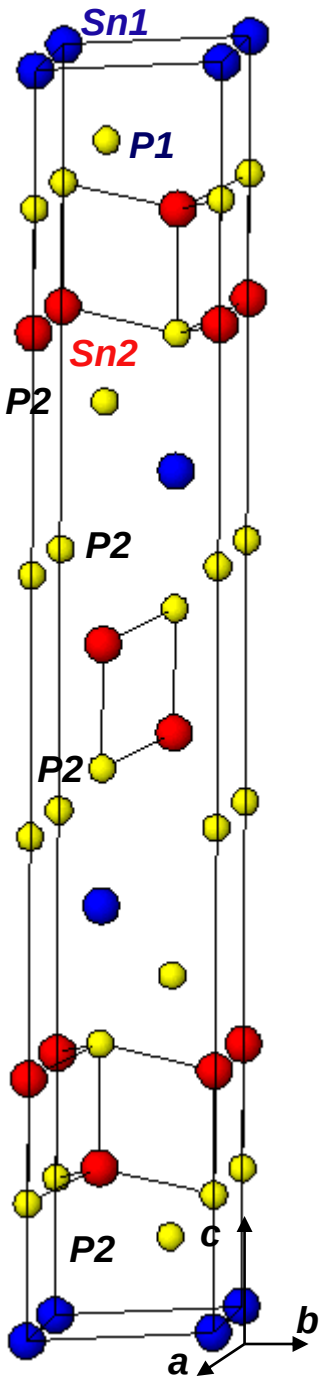
Propagation of VO_3 chains in different layers of $Th(VO_3)_4$ crystal structure





Crystal structure of Sn_3P_4

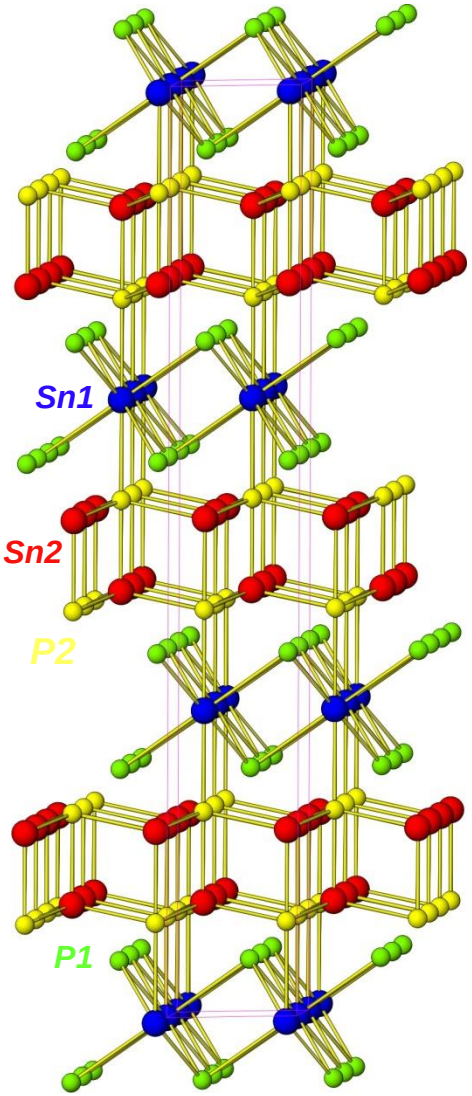


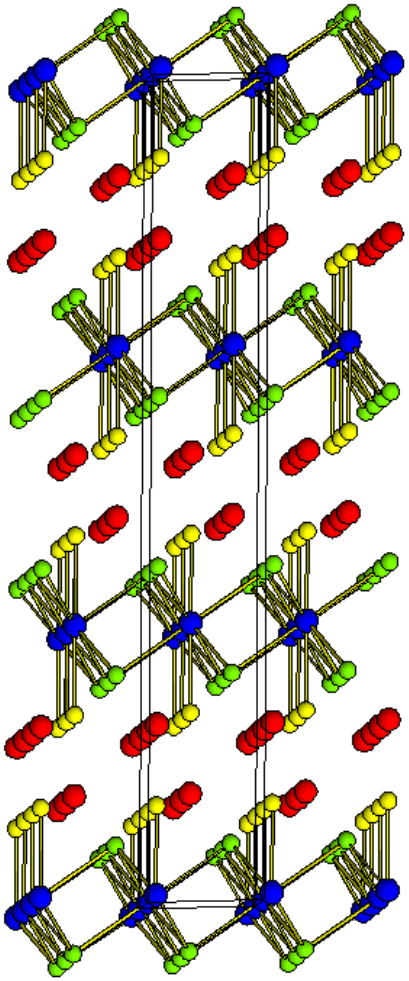


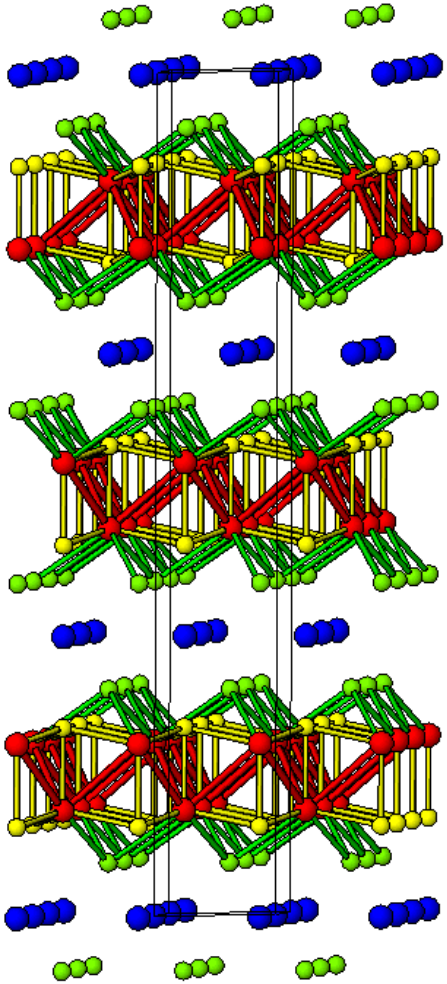
Rhombohedral (R-3m), No. 166
a= 4.4362(1) and c = 28.4220(5) Å

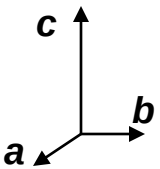
Atom	Wyck.	z	U11	U22	U33	U12
Sn1	3a	0	0.0079(10)	0.0079(10)	0.0153(4)	0.0040(5)
Sn2	6c	0.79371(6)	0.0258(8)	0.0258(8)	0.0215(7)	0.0129(4)
P1	6c	0.8918(14)	0.80(4)	0.80(4)	0.80(5)	0.40(2)
P2	6c	0.6030(6)	0.24(1)	0.24(1)	0.24(1)	0.120(6)

Crystal structure of Sn_3P_4

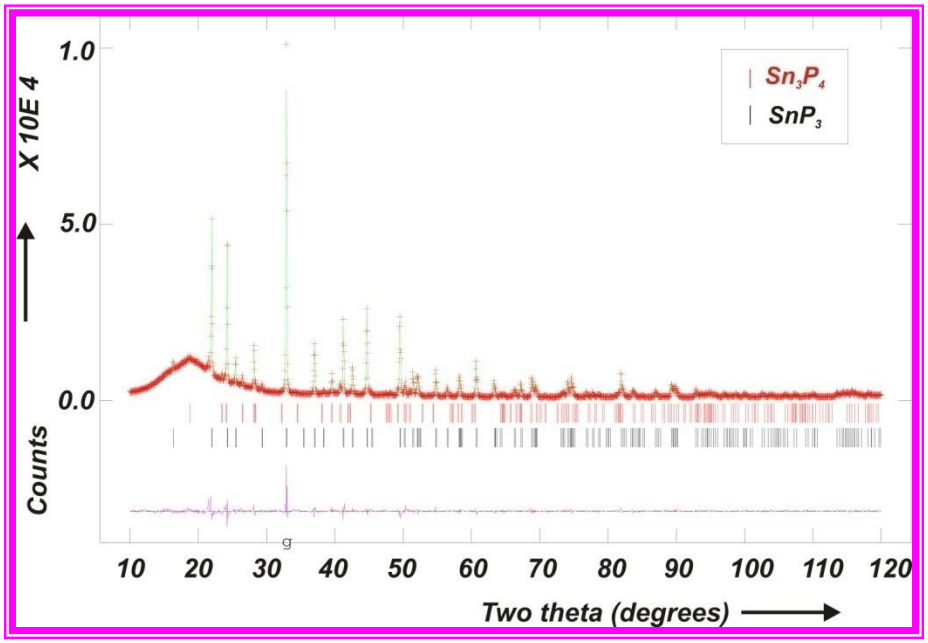


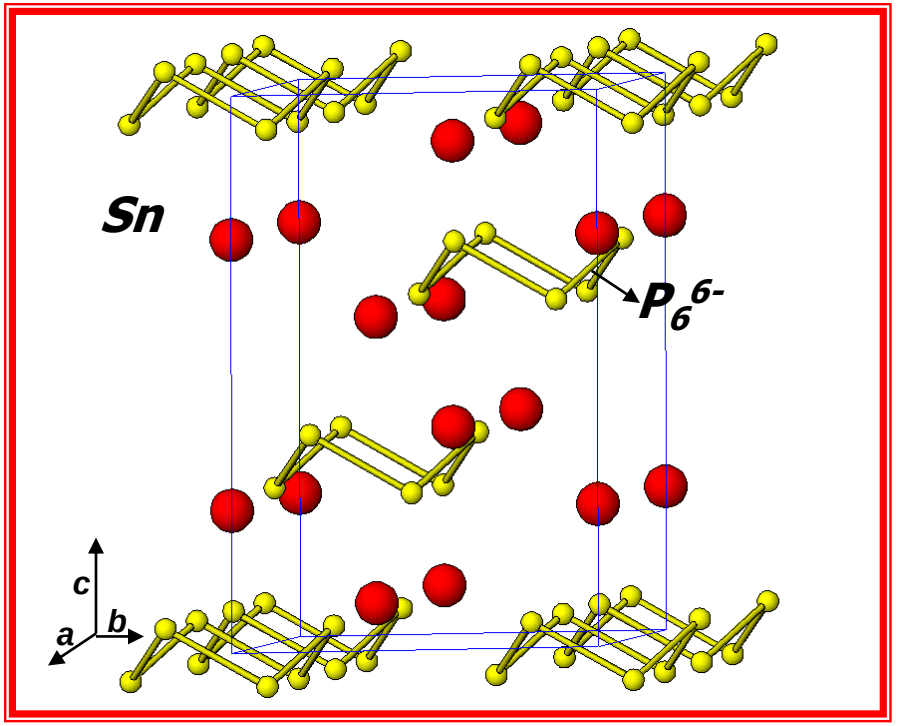






Crystal structure of SnP_3



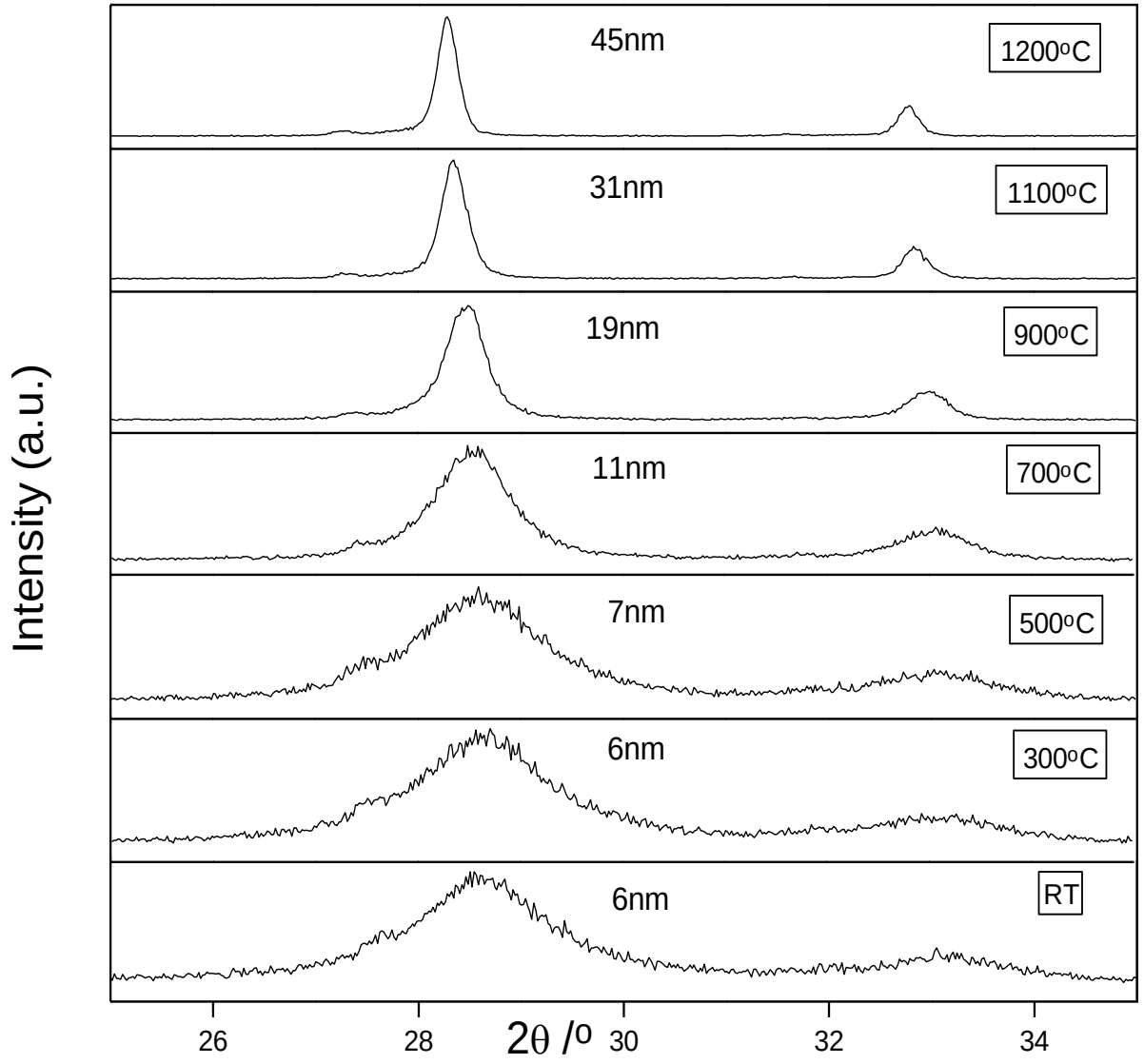


Rhombohedral (R-3m), No. 166
a= 7.3730(1) and c = 10.5361(4)

Atom	Wyck.	x	y	z
Sn1	6c (0,0,z)	0	0	0.2569(3)
P1	18h (x-x,z)	0.5135(2)	0.4865(2)	0.2820(3)

U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
0.0152(6)	0.0152(6)	0.022(2)	0.0076(3)	0	0
0.016(1)	0.016(1)	0.017(2)	0.009(2)	-0.0011(8)	0.0011(8)

High Temperature XRD data for nc-$Ce_{0.55}Y_{0.45}O_{1.775}$



- Noticeable grain growth begins at 700°C

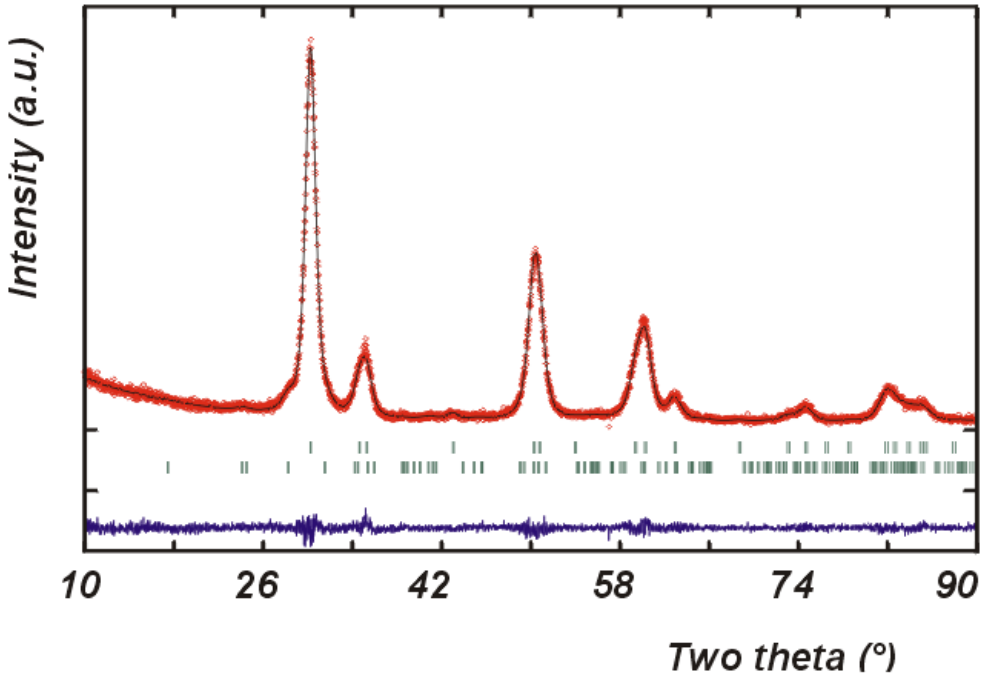
Crystallite size and strain in nano ZrO_2 powders

Tetragonal ZrO_2
(P42/nmc, Z = 2)
a = 3.5970(3), c = 5.1804(9) Å
V = 67.03(1) Å^3
RB = 1.04 an RF = 0.90 %

Monoclinic ZrO_2
(P21/c, Z = 4)
a = 5.1420, b = 5.2010, c = 5.3111 Å
and β = 99.2°, V = 140.2 Å^3
RB = 4.10 an RF = 2.1 %

Rp = 6.2, Rwp = 8.7, Rexp = 7.9 %
χ2 = 1.21

Tetragonal ZrO_2 = 93.0(7)
Monoclinic ZrO_2 = 7.0(3)



$$Vs = K\frac{S_S \times V_S}{S_A}$$

$$Wj = \frac{S_j Z_j M_j V_j / t_j}{\sum (S_i Z_i M_i V_i / t_i)}$$

$$\frac{H \times \cos\theta}{\lambda} = \frac{K}{V} + 2\varepsilon \frac{\sin\theta}{\lambda}$$

Sin(θ)/λ

Tetragonal ZrO_2: Crystallite sizes 86 Å and strain 0.4 %

Property	X-ray diffraction	Neutron diffraction
Location of low Z elements	Not favourable	Favourable
Neighbouring elements	Difficult to distinguish	Easy to distinguish
Penetration	10-100 μm	Few cm
Sample size	Few mg	5-10 gm
Resolution	Good	Very good
Single crystal work	Easy to do	Not so easy
Monochromatization	Easy	Difficult
Isotopes	Cannot be differentiated	Can be differentiated
Magnetic structure	Cannot be obtained	Can be obtained
Study of lattice vibrations (dynamic)	Not possible	Possible
Preferred orientation	Problem	No effect
Intensity	Drops as angle increases	No correlation with angle

Thank you very much