Nutshells of Powder X-ray Diffraction (PXRD)

You don't want to line up. Then we will catch your family !



3

Solid

<u>Gas, Liquid</u> No structure of ordering or stacking Structure in molecule or atom

Solid All solids in the universe have a certain number of ways of packing – **crystalline solid**

> 비정형 고체 (Amorphous solid) 결정형 고체 (Crystalline solid) 준결정 (Quasicrystal)



Solid Structure (Crystal Structure)

Unit Cell

CRYSTAL STRUCTURE

The periodic arrangement of atoms in the crystal.

LATTICE

An infinite array of points in space, in which each point has identical surroundings to all others.

(= Lattice points are all equivalent.)

UNIT CELL

A component of the crystal, which when stacked together with pure translational repetition reproduces the whole crystal.

PRIMITIVE (P) UNIT CELLS contain only a *single lattice point*



(a) (a) (b) (b) (a) (a) (b) (b)

Unit Cell

Unit cell?

(a) (a) (b) (b) (a) (a) (b) (b)

Unit Cell

Unit cell?

Requirement of Unit Cell (How to fill the space)

Crystallographic restriction theorem:

Must have 2-, or 3- fold axes => C_2 , C_3 , C_4 , C_6 axes (except for quasicrystal)





7 Crystal Systems (14 Bravais Lattices)





32 Point Groups, 230 Space groups



A point group is defined by the symmetry operations of

Symmetry Operations	Symmetry Elements
Rotation (C_n) Reflection (σ)	Rotational axis Mirror plane
Inversion (<i>i</i>)	Center of symmetry
Improper rotation (S_n)	Improper rotational axis

Theoretically, there are infinite number of point groups, but the crystallographic restriction theorem gives only **32 point groups** in the crystal systems.

Point group symmetry defines the symmetry of an isolated object or group of objects.



If **only lattice points** are considered, the point group of hexagonal lattice is D_{6h} .

Depending on the motif, it can have C_6 , C_{3h} , C_{6h} , D_6 , C_{6v} , D_{3h} .

Hexagonal lattice

32 Point Groups

(n) means \overline{n}

Counted Sustame	Hermann	-Mauguin	Chubaikay	Cohoonfling	Orbifold	Coveter	Order	
Crystal Systems	(full)	(short)	Shubhikov	Schoentlies	Orbitold	Coxeter		
Triclinic	1	1	1	C1	11	[]+	1	
Themae	1	1	2	$C_i = S_2$	×	[2+,2+]	2	
	2	2	2	C ₂	22	[2]+	2	
Monoclinic	m	m	m	$C_s = C_{1h}$	*	[]	2	
	<u>2</u> m	2/m	2:m	C _{2h}	2*	[2,2 ⁺]	4	
	222	222	2:2	$D_2 = V$	222	[2,2]+	4	
Orthorhombic	mm2	mm2	$2 \cdot m$	C _{2v}	*22	[2]	4	
	$\frac{2}{m}\frac{2}{m}\frac{2}{m}\frac{2}{m}$	mmm	$m \cdot 2 : m$	$D_{2h} = V_h$	*222	[2,2]	8	
	4	4	4	C4	44	[4]+	4	
	4	4	ã	S4	2×	[2+,4+]	4	
	<u>4</u> m	4/m	4:m	C _{4h}	4*	[2,4+]	8	
Tetragonal	422	422	4:2	D4	422	[4,2] ⁺	8	
	4mm	4mm	$4 \cdot m$	C _{4v}	*44	[4]	8	
	@ <mark>2m</mark>	<mark>⊛2m</mark>	$\tilde{4} \cdot m$	$D_{2d} = V_d$	2*2	[2+,4]	8	
	$\frac{4}{m}\frac{2}{m}\frac{2}{m}$	4/mmm	$m \cdot 4 : m$	D _{4h}	*422	[4,2]	16	
	3	3	3	C3	33	[3]+	3	
	3	3	õ	$S_6 = C_{3i}$	3x	[2+,6+]	6	
Trigonal	32	32	3:2	D ₃	322	[3,2]+	6	
	3m	3m	$3 \cdot m$	C _{3v}	*33	[3]	6	
	$3\frac{2}{m}$	③m	$\tilde{6} \cdot m$	D _{3d}	2*3	[2+,6]	12	
	6	6	6	C ₆	66	[6]+	6	
	۲	6	3:m	C _{3h}	3*	[2,3 ⁺]	6	
	<u>6</u> m	6/m	6:m	C _{6h}	6*	[2,6 ⁺]	12	
Hexagonal	622	622	6:2	D ₆	622	[6,2]+	12	
	6mm	6mm	$6 \cdot m$	C _{6v}	*66	[6]	12	
	©m2	©m2	$m \cdot 3 : m$	D _{3h}	*322	[3,2]	12	
	$\frac{6}{m} \frac{2}{m} \frac{2}{m}$	6/mmm	$m \cdot 6 : m$	D _{6h}	*622	[6,2]	24	
	23	23	3/2	Т	332	[3,3]+	12	
Cubic	$\frac{2}{m}$ ③	m③	$\tilde{6}/2$	Τ _h	3*2	[3+,4]	24	
	432	432	3/4	0	432	[4,3]+	24	
	@3m	<mark>⊛3m</mark>	$3/\tilde{4}$	T _d	*332	[3,3]	24	
	$\frac{4}{m} \Im \frac{2}{m}$	m③m	$\tilde{6}/4$	Oh	*432	[4,3]	48	

32 Point Groups, 230 Space groups



To generate an infinite 3D lattice from an object it is necessary to add translational symmetry to point group symmetry.

Symmetry Operations	Defined
Rotation (C_n) Reflection (σ) Inversion (i) Improper rotation (S_n)	Point group
+ Glide plane (= translation + reflection) Screw axis (= translation + rotation)	Space group

Glide plane is a symmetry operation describing how a reflection in a plane, followed by a translation (**less than a unit cell vector**) parallel with that plane, may leave the crystal unchanged.

- a: reflection followed by $\frac{1}{2}$ **a** translation
- b: reflection followed by $\frac{1}{2}$ b translation
- c: reflection followed by $\frac{1}{2}$ c translation

n : reflection followed by $\frac{1}{2}$ **a** + $\frac{1}{2}$ **b** or $\frac{1}{2}$ **a** + $\frac{1}{2}$ **c** or $\frac{1}{2}$ **b** + $\frac{1}{2}$ **c** translation (diagonal glide) d : reflection followed by $\frac{1}{4}$ **a** + $\frac{1}{4}$ **b** or $\frac{1}{4}$ **a** + $\frac{1}{4}$ **c** or $\frac{1}{4}$ **b** + $\frac{1}{4}$ **c** translation (diagonal glide)



Screw axis is a symmetry operation describing how a rotation about an axis, followed by a translation (**less than a unit cell vector**) parallel with that axis, may leave the crystal unchanged.

11 unique screw axes: $2_1, 3_1, 3_2, 4_1, 4_2, 4_3, 6_1, 6_2, 6_3, 6_4, 6_5$ (n_m: counter clockwise $C_n + 1/n$ translation)



Crystal systems, Bravais lattices Point Groups, Space groups





Crystal systems, Bravais lattices Point Groups, Space groups

ш	Crystal system			Point group							
# В	Bravais lattice	Intl	Schön,	Orbifold notation	Сох	Ord.	Space groups (international short symbol)				
1	Triclinic (2) α, β, γ ≠ 90°	1	C ₁	11	[]+	1	P1				
2		Ţ	Ci	1×	[2+,2+]	2	PT				
3-5	Monoclinic	2	C2	22	[2]+	2	P2, P2 ₁ C2				
6-9	(13) $\beta \neq 90^{\circ} \beta \neq 90^{\circ} \\ a, \gamma = 90^{\circ} a, \gamma = 90^{\circ}$	m	Cs	*11	[]	2	Pm, Pc Cm, Cc				
10-15		2/m	C _{2h}	2*	[2,2*]	4	P2/m, P21/m C2/m, P2/c, P21/c C2/c				
16-24		222	D2	222	[2,2]+	4	P222, P2221, P21212, P212121, C2221, C222, F222, I222, I2121				
25-46	Orthorhombic (59)	mm2	C _{2v}	*22	[2]	4	Pmm2, Pmc2 ₁ , Pcc2, Pma2, Pca2 ₁ , Pnc2, Pmn2 ₁ , Pba2, Pna2 ₁ , Pnn2 Cmm2, Cmc2 ₁ , Ccc2, Amm2, Aem2, Ama2, Aea2 Fmm2, Fdd2 Imm2, Iba2, Ima2				
47-74	$ \begin{array}{c} \begin{array}{c} \begin{array}{c} \\ \\ \\ \\ \\ \end{array} \end{array} \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \\ \end{array} \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \end{array} \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \end{array} \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} $	mmm	D _{2h}	*222	[2,2]	8	Pmmm, Pnnn, Pccm, Fban, Pmma, Pnna, Pmna, Pcca, Pbam, Pccn, Pbcm, Pnnm, Pmmn, Pbcn, Pbca, Pnma Cmcm, Cmce, Cmmm, Cccm, Cmme, Ccce Fmmm, Fddd Immm, Ibam, Ibca, Imma				
75-80		4	C4	44	[4]+	4	P4, P4 ₁ , P4 ₂ , P4 ₃ , I4, I4 ₁				
81-82		4	S4	2×	[2+,4+]	4	P4, I4				
83-88	Tetragonal (68)	4/m	C _{4h}	4*	[2,4*]	8	P4/m, P4 ₂ /m, P4/n, P4 ₂ /n I4/m, I4 ₁ /a				
89-98	c	422	D4	224	[2,4]+	8	P422, P42 ₁ 2, P4 ₁ 22, P4 ₁ 212, P4 ₂ 22, P4 ₂ 212, P4 ₃ 22, P4 ₃ 212 I422, I4 ₁ 22				
99-110		4mm	C _{4v}	*44	[4]	8	P4mm, P4bm, P4 ₂ cm, P4 ₂ nm, P4cc, P4nc, P4 ₂ mc, P4 ₂ bc I4mm, I4cm, I4 ₁ md, I4 ₁ cd				
111-122	a c	4 2m	D _{2d}	2*2	[2+,4]	8	P42m, P42c, P421m, P421c, P4m2, P4c2, P4b2, P4n2 I4m2, I4c2, I42m, I42d				
123-142		4/mmm	D _{4h}	*224	[2,4]	16	P4/mmm, P4/mcc, P4/nbm, P4/nnc, P4/mbm, P4/mnc, P4/nmm, P4/ncc, P4 ₂ /mmc, P4 ₂ /mcm, P4 ₂ /nbc, P4 ₂ /nnm, P4 ₂ /mbc, P4 ₂ /mmm, P4 ₂ /nmc, P4 ₂ /ncm I4/mmm, I4/mcm, I4 ₁ /amd, I4 ₁ /acd				

Crystal systems, Bravais lattices Point Groups, Space groups

	Crystal system (count) Bravais lattice			Point group			Space groups (international short symbol)
#		Inti	Schön,	Orbifold notation	Cox	Ord.	
143-146	Trigonal	3	Сз	33	[3]+	3	P3, P3 ₁ , P3 ₂ R3
147-148	(25)	3	S ₆	3×	[2+,6+]	6	P3, R3
149-155		32	D3	223	[2,3]+	6	P312, P321, P3 ₁ 12, P3 ₁ 21, P3 ₂ 12, P3 ₂ 21 R32
156-161	y = 120*	Зm	С _{зv}	*33	[3]	6	P3m1, P31m, P3c1, P31c R3m, R3c
162-167	a a	Зm	D _{3d}	2*3	[2+,6]	12	P31m, P31c, P3m1, P3c1 R3m, R3c
168-173		6	C ₆	66	[6]+	6	P6, P6 ₁ , P6 ₅ , P6 ₂ , P6 ₄ , P6 ₃
174	Hexagonal	6	С _{зһ}	3*	[2,3*]	6	Põ
175-176	(27) 6/m C _{6h} 6* [2,6*] 12		12	P6/m, P6 ₃ /m			
177-182		622	D ₆	226	[2,6]*	12	P622, P6 ₁ 22, P6 ₅ 22, P6 ₂ 22, P6 ₄ 22, P6 ₃ 22
183-186	·	6mm	C _{6v}	*66	[6]	12	P6mm, P6cc, P6gcm, P6gmc
187-190		ī6m2	D _{3h}	*223	[2,3]	12	Põm2, Põc2, Põ2m, Põ2c
191-194		6/mmm	D _{6h}	*226	[2,6]	24	P6/mmm, P6/mcc, P63/mcm, P63/mmc
195-199		23	Т	332	[3,3]+	12	P23, F23, I23 P2 ₁ 3, I2 ₁ 3
200-206	Cubic (36)	mЗ	Th	3*2	[3*,4]	24	Pm3, Pn3, Fm3, Fd3, Im3, Pa3, Ia3
207-214		432	0	432	[3,4]*	24	P432, P4 ₂ 32 F432, F4 ₁ 32 I432 P4 ₃ 32, P4 ₁ 32, I4 ₁ 32
215-220		43m	Td	*332	[3,3]	24	P43m, F43m, I43m P43n, F43c, I43d
221-230		mЗm	O _h	*432	[3,4]	48	Pm3m, Pn3n, Pm3n, Pn3m Fm3m, Fm3c, Fd3m, Fd3c Im3m, Ia3d





(1/Real) World



Miller Indices



Miller index of the crystal plane: (1/n 1/m 1/p) => smallest integer (*h k l*)











incident reflected beam beam θ В Α X 60 UNIT (1,0,0) а CELL m n Ð θ D С У incident reflected beam beam A A' B X 00 d m ์ท UNIT (2,0,0) — -E Э, ∕₽ CELL а У F Ρ d O D С Ζ

Constructive interference

$$2d\sin\theta = n\lambda$$

Bragg's Law

Reciprocal Lattice



Reciprocal Lattice

Due to the linear relationship between planes (ex, $d_{200} = (1/2)d_{100}$), a periodic lattice is generated. In general, the periodicity in the reciprocal lattice is given by

$$\rho *_{hkl} = 1/d_{hkl}$$

In vector form, the general reciprocal lattice vector for the (*hkl*) plane is given by

 $g_{hkl} = \mathbf{n}_{hkl}/d_{hkl}$

 \mathbf{n}_{hkl} : unit vector normal to the (*hkl*) planes.



Reciprocal Lattice

Direct and Reciprocal Cell Relationships



Cubic a = b = c, $\alpha = \beta = \gamma = \alpha * = \beta * = \gamma * = 90^{\circ}$ **Tetragonal** $a = b \neq c$, $\alpha = \beta = \gamma = \alpha * = \beta * = \gamma * = 90^{\circ}$

Reciprocal Lattice

Direct and Reciprocal Cell Relationships



Hexagonal $a = b \neq c$, $\alpha = \beta = \alpha * = \beta * = 90^{\circ}$, $\gamma = 120^{\circ}$, $\gamma * = 60^{\circ}$,

Reciprocal Lattice

Direct and Reciprocal Cell Relationships



Trigonal a = b = c, $\alpha = \beta = \gamma \neq 90^{\circ}$

Reciprocal Lattice

Due to the linear relationship between planes (ex, $d_{200} = (1/2)d_{100}$), a periodic lattice is generated. In general, the periodicity in the reciprocal lattice is given by

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 \mathbf{n}_{hkl} : unit vector normal to the (*hkl*) planes.



Reciprocal Lattice



Absence in reciprocal lattice

High symmetry can lead to reflections being systematically absent from the data set.

For non-primitive lattices, such as a Ccentred lattice, systematic absences can occur in the reciprocal lattice (and in diffraction pattern) due to the construction of the lattices.



Reciprocal Lattice

Absence in reciprocal lattice

General absences determine the lattice type; Primitive (**P**) has no general absences. End Centered (**C**) h+k=2n+1 are all absent. Face Centered (**F**) only h, k, l, all even or all odd are observed.

Body Cantered (I) h+k+l=2n+1 are all absent.

Special absences refer to specific sets of reflections and are used to detect the presence of glide planes and screw axes.

Give information for determining space groups



Ewald Sphere



If this geometry is constructed **in reciprocal space** with

 $r = 1/\lambda$ Y is 000 point X is a general *hkl* point

 $=> XY = 1/d_{hkl}$

 $\frac{2}{\lambda}\sin\theta = 1/d_{hkl}$ $2d_{hkl}\sin\theta = \lambda \quad \text{Bragg's Law}$

 $2r\sin\theta = XY$

In 3D, it's a sphere, called **Ewald Sphere**.

Even though the Ewald sphere is in reciprocal space (inverse distance) and we are in real space, we can use the predicted angles of diffraction (2θ) to predict the diffraction pattern.

Ewald Sphere



Ewald Sphere

Even though the Ewald sphere is in reciprocal space (inverse distance) and we are in real space, we can use the predicted angles of diffraction (2θ) to predict the diffraction pattern.

→ Diffraction occurs when a reciprocal lattice point intersects Ewald sphere.



 $2d_{hkl}\sin\theta = \lambda$ **Bragg's Law**

Ewald Sphere



Limiting Sphere



If one rotates the Ewald sphere completely about the (000) reciprocal lattice point in all three dimensions, the larger sphere (of radius $2/\lambda$) contains all of the reflections that it is possible to collect using that wavelength of X-rays. This construction is known as the "Limiting sphere" and it defines the complete data set. Any reciprocal lattice points outside of this sphere can not be observed.

We have to collect diffraction patterns in all possible angles. Then **how many diffraction spots can we get theoretically**?



Limiting Sphere



Complete Data Set: total # of reciprocal lattice points within the limiting sphere

= theoretically possible total # of reflection points

$$\approx \frac{\frac{4}{3}\pi r^{3}}{V^{*}} = \frac{4}{3}\pi (2/\lambda)^{3}V \approx 33.5 \ V/\lambda^{3} = 33.5/V^{*}\lambda^{3}$$

(V*: volume of reciprocal unit cell

V: volume of unit cell. $\lambda = X$ -ray wavelength



Resolution:

Theoretical maximum resolution: when $\sin \theta = 1$, $\mathbf{d}_{res} = \lambda/2$ Real maximum resolution: $\mathbf{d}_{res} = (\lambda/2) \sin \theta_{max}$

$$2d \sin \theta = \lambda$$
$$\sin \theta_{max} = \lambda/2d_{min}$$

By reducing the wavelength, Bragg's Law indicates that the diffraction angles (θ) will decrease; the spectrum shrinks, but on the other hand, more diffraction data will be obtained, and therefore a better structural resolution will be achieved.







(120) planes Intensity 560-1120 Х 280-559 0 140-279 70-139 35-69 18-34 9-17 4-8 2-3 \checkmark у 1-1 h + <1 $2d\sin\theta = n\lambda$ Different sets of planes Circles of constant in the crystal give rise to theta (resolution) different diffraction spots. ľ₹ (120)(240)(360





- For every set of planes, there will be a small percentage of crystallites that are properly oriented to diffract (the plane perpendicular bisects the incident and diffracted beams).
- Basic assumptions of powder diffraction are that for every set of planes there is an equal number of crystallites that will diffract and that there is a statistically relevant number of crystallites, not just one or two.





Diffractometer



Bragg-Brentano Geometry Parallel Beam Geometry generated by Göbel Mirrors

Ewald Sphere and PXRD Spectrum



Information obtained from PXRD

- **peak position** \rightarrow dimension of the elementary cell
- **peak intensity** \rightarrow content of the elementary cell
- **peak broadening** → strain/crystallite size
- scaling factor \rightarrow quantitative phase amount (phase identity, Phase purity, % phase composition) Crystallinity
- Crystal structure (till now very small molecules)
- diffuse background \rightarrow false order
- modulated background \rightarrow close order

d-spacing

Cubic **Rhombohedral** $\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$ Tetragonal a $\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$ **Monoclinic** Hexagonal $\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$ **c** $\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$ $\frac{1}{12} = \frac{1}{120} \left(S_{11}h^2 + S_{22}k^2 + S_{33}l^2 + 2S_{12}hk + 2S_{23}kl + 2S_{13}hl \right)$ S

$$\frac{1}{l^2} = \frac{(h^2 + k^2 + l^2)\sin^2\alpha + 2(hk + kl + hl)(\cos^2\alpha - \cos\alpha)}{a^2(1 - 3\cos^2\alpha + 2\cos^3\alpha)}$$

$$\frac{1}{d^2} = \frac{1}{\sin^2 \beta} \left(\frac{h^2}{a^2} + \frac{k^2 \sin^2 \beta}{b^2} + \frac{l^2}{c^2} - \frac{2hl \cos \beta}{ac} \right)$$

Triclinic

where
$$S_{11} = b^2 c^2 \sin^2 \alpha$$
 $S_{12} = abc^2 (\cos \alpha \cos \beta - \cos \gamma)$
 $S_{22} = a^2 c^2 \sin^2 \beta$ $S_{23} = a^2 bc (\cos \beta \cos \gamma - \cos \alpha)$

$$S_{33} = a^2 b^2 \sin^2 \gamma$$
 $S_{13} = a b^2 c (\cos \gamma \cos \alpha - \cos \beta)$

 $2d_{hkl}\sin\theta = n\lambda$

Indexing is the process of determining the unit cell dimensions from the peak positions Manual indexing (time consuming...but still useful)

Pattern matching/auto indexing (JADE or other computer based indexing software)

Cubic $\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$ $\sin^2 \theta = \frac{\lambda^2}{4} (\frac{h^2 + k^2 + l^2}{a^2})$

How many d-spacings can be are detected ? Infinte But Bragg's equation limits the smallest d-spacing possible = $\lambda/2$. Also the first peak is arising from the largest d-spacing in the unit cell (if not absent).



Try to get *a* with $\lambda = 1.54$ Å

Indexing XRD Patterns



Peak Width

Peak Width-Full Width at Half Maximum



Effect of lattice strain



Scherrer's Formula



$$t = \frac{K * \lambda}{B * \cos \theta_{\rm B}}$$

t = thickness of crystallite (< 1000 Å) K = constant dependent on crystallite shape (~0.9) λ = x-ray wavelength B = FWHM = $\theta_{high} - \theta_{low}$ (or integral breadth) θ_{B} = Bragg Angle (radian)

It is used in the determination of average size of particles of crystals in the form of powder (for less than 1000 Å) (if there is no other broaden effect)

Preferred Orientation



Preferred Orientation



Sample Preparation for PXRD

Roughly

It needs to be a powder

It needs to be a pure powder

Its nice to have about 1/2 g of sample, but one can work with less

The powder needs to be packed tightly in the sample holder. Lose powders will give poor intensities.

Sample Preparation for PXRD

Details

Many crystallites in random orientations

If the crystallites in a sample are very large, there will not be a smooth distribution of crystal orientations. You will not get a powder average diffraction pattern. (crystallites should be $<10\mu$ m in size to get good powder statistics)

Large crystallite sizes and non-random crystallite orientations both lead to peak intensity variation

Characteristics of sample

A flat plate sample for XRPD should have a smooth flat surface

- If the surface is not smooth and flat, X-ray absorption may reduce the intensity of low angle peaks

- parallel-beam optics can be used to analyze samples with odd shapes or rough surfaces

Densely packed

Randomly oriented grains/crystallites

Grain size less than 10 microns

'Infinitely' thick

Data Collection

The scattering intensity drops as $1/2(1+\cos^22\theta)$

This means that you don't get much intensity over 70° (2 θ).

A good range is $10-70^{\circ}$ (2 θ).

How long should you collect (time per step)? Depending on what you want.

- Routine analysis may only take 30-60 min.
- Data for Rietveld analysis may take 12-18 hours to collect

JCPDS Cards

JCPDS - Joint Committee on Powder Diffraction Standards (or ICDD - International Centre for Diffraction Data)

PXRD database over 100,000 materials.

Want to confirm your spectrum is arising from a know material?

- search JCPDS database

If it is new, index the pattern with JADE and report.



1.file number 2.three strongest lines 3.lowest-angle line 4.chemical formula and name 5.data on diffraction method used 6.crystallographic data 7.optical and other data 8.data on specimen 9.data on diffraction pattern







Examples

SUMOF-3



PXRD

Examples

SUMOF-4





This difference indicates that when the guest species are removed, symmetry of SUMOF-4 changes.



J. Mater. Chem., 2012, 22, 10345

PXRD

Examples

Reaction of SO₂ with Ni(BME-DACH)*



Inorg. Chem. 2004, 43, 4702-4707