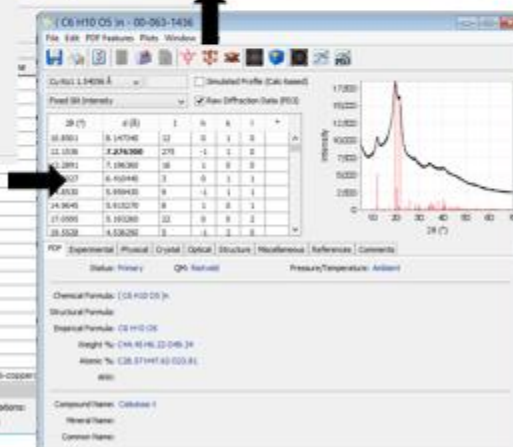
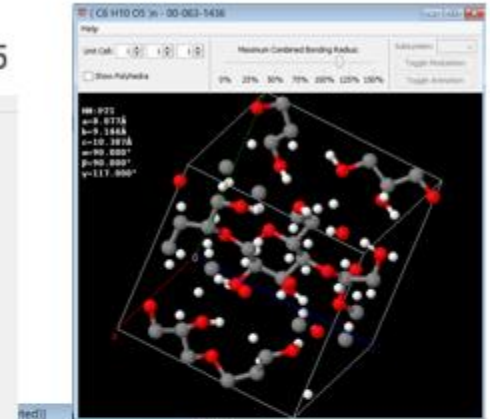
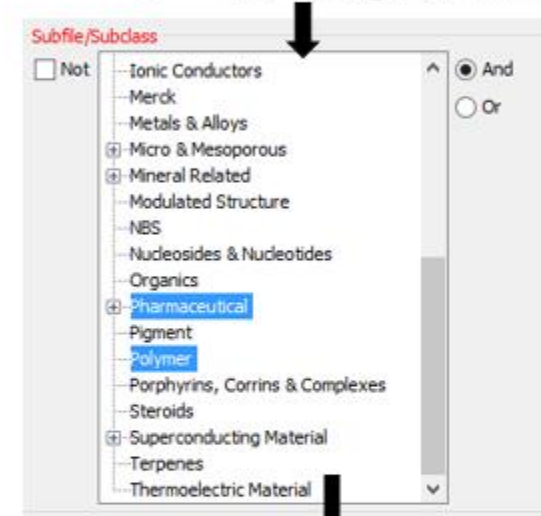


# ICDD Powder Diffraction File® Coverage of Polymers Used in Pharmaceutical and Biomedical Applications



TOM BLANTON AND STACY GATES, ICDD

# This document was presented at PPXRD - Pharmaceutical Powder X-ray Diffraction Symposium

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PPXRD Website – [www.icdd.com/ppxrd](http://www.icdd.com/ppxrd)

ICDD Website - [www.icdd.com](http://www.icdd.com)

# Co-presenters

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Tim Fawcett

Suri Kabekkodu

Amy Gindhart

Vesna Bosnic

Diane Sagnella

Emily Foster

Megan Rost

Jackie Hollencamp

Justin Blanton

Kai Zhong

Rose Vithayathil

Mike Carr

Janet Grande

# What is a polymer?

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The word polymer is derived from the Greek words *polus*, meaning many, and *meros*, meaning parts.

A long-chained molecule that is composed of individual units, called monomers.

A compound typically of high molecular weight derived either by the **addition** of many smaller molecules, such as polyethylene, or by the **condensation** of many smaller molecules with the elimination of water, alcohol, or the like, such as nylon.

Polymers can be natural or synthetic

There are inorganic polymer and coordination polymer materials, we will focus on carbon/organic based polymers

# Polymer analysis using XRD

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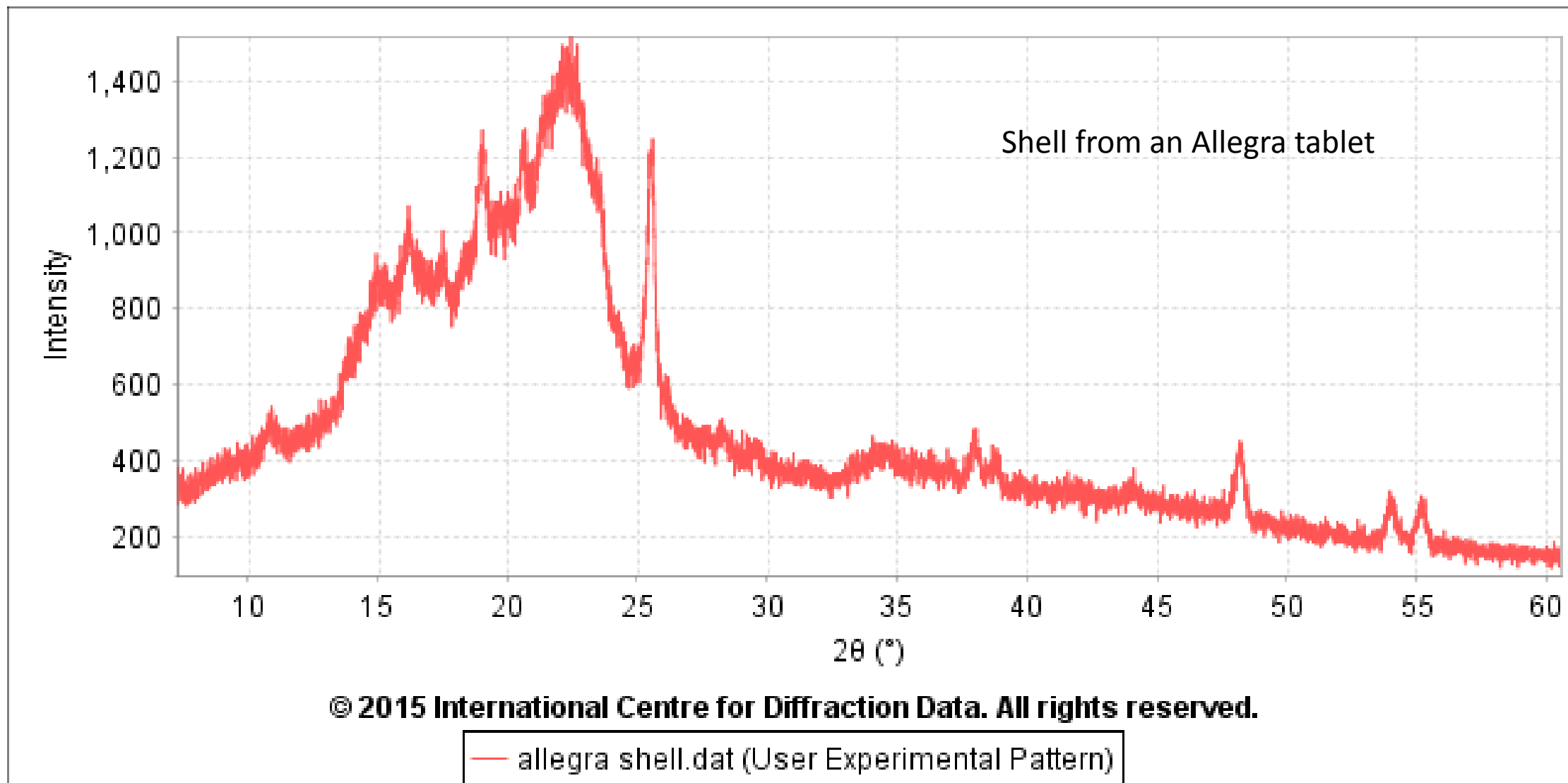
Many techniques are used for polymer characterization

- IR, NMR, GC/MS, etc.

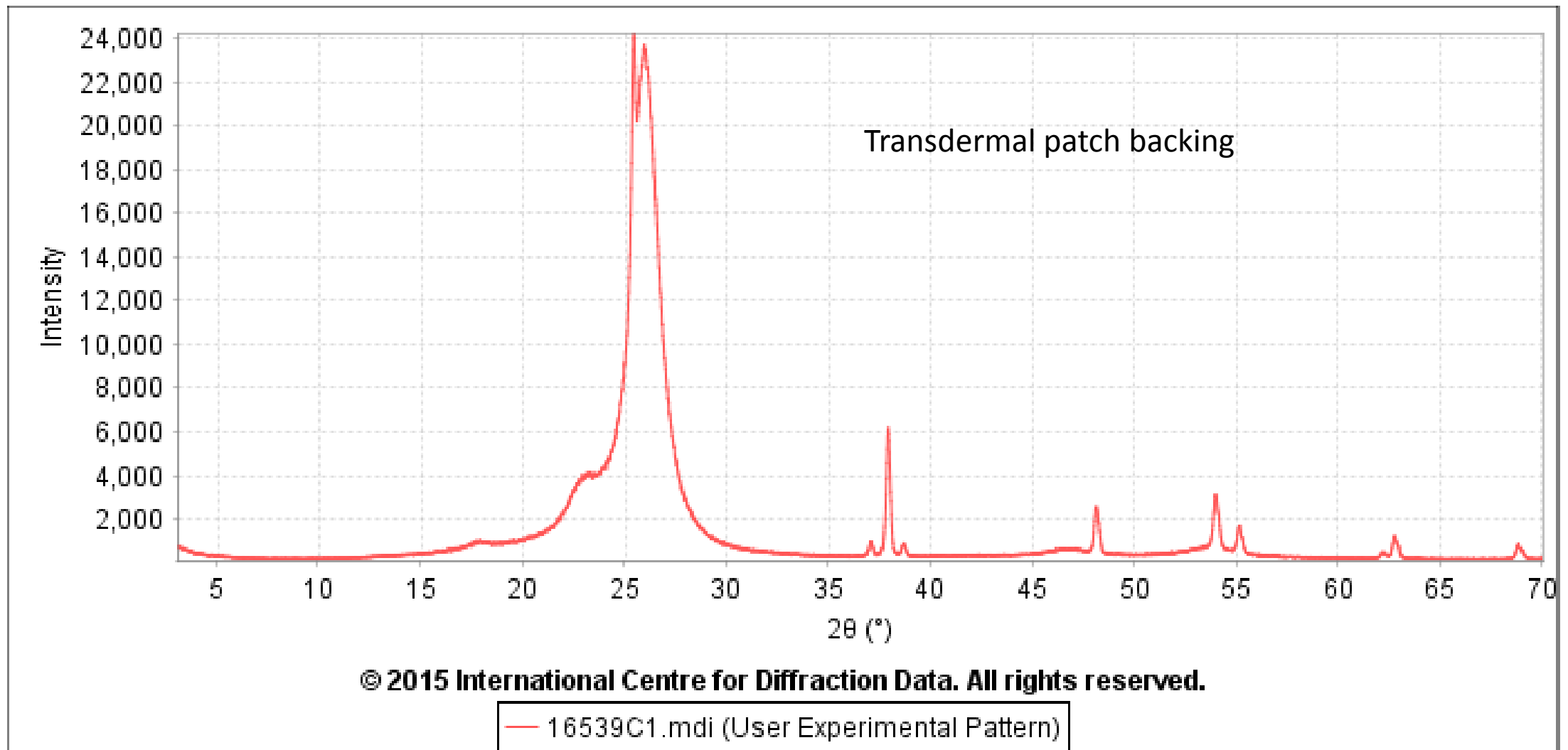
Why use XRD?

- Polymers can have amorphous and crystalline regions
- Crystallite size is small and measurable by XRD
- Polymers are often used as the base component in pharmaceutical and biomedical composite materials

# Pharmaceuticals

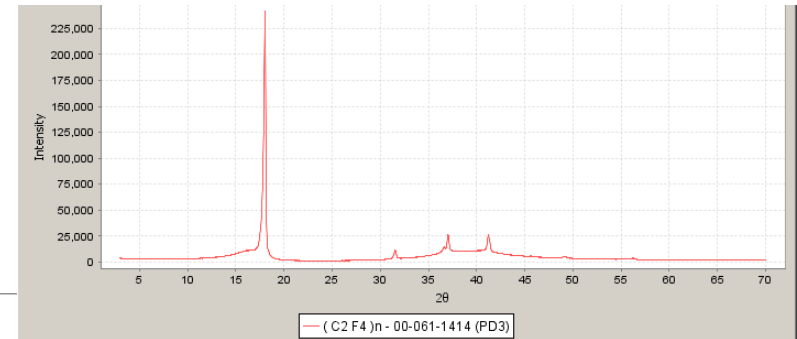


# Biomedical devices

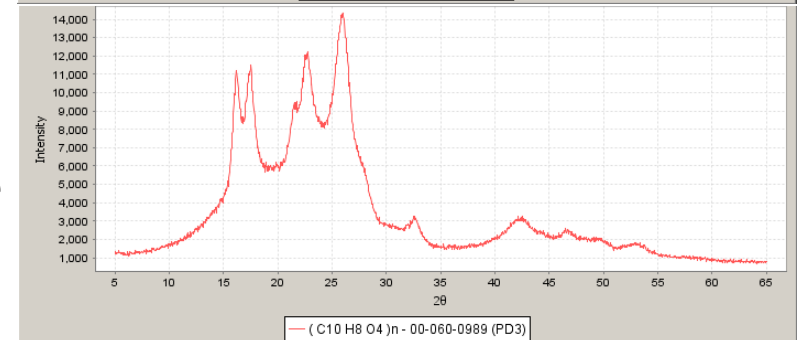


# Polymer order: chemistry matters

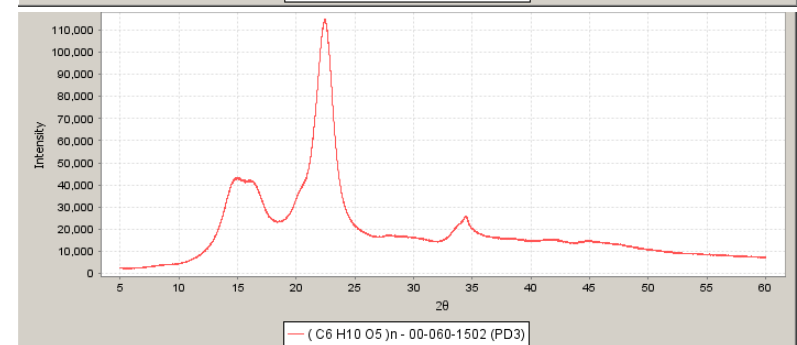
Highly crystalline - Teflon



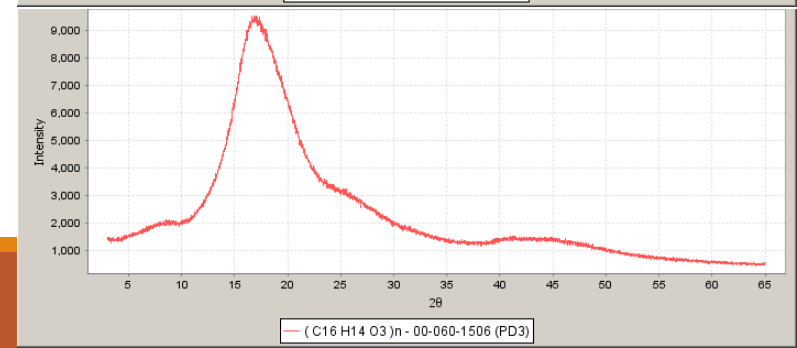
Semi crystalline – polyethylene terephthalate



Micro crystalline – cellulose 1 $\beta$

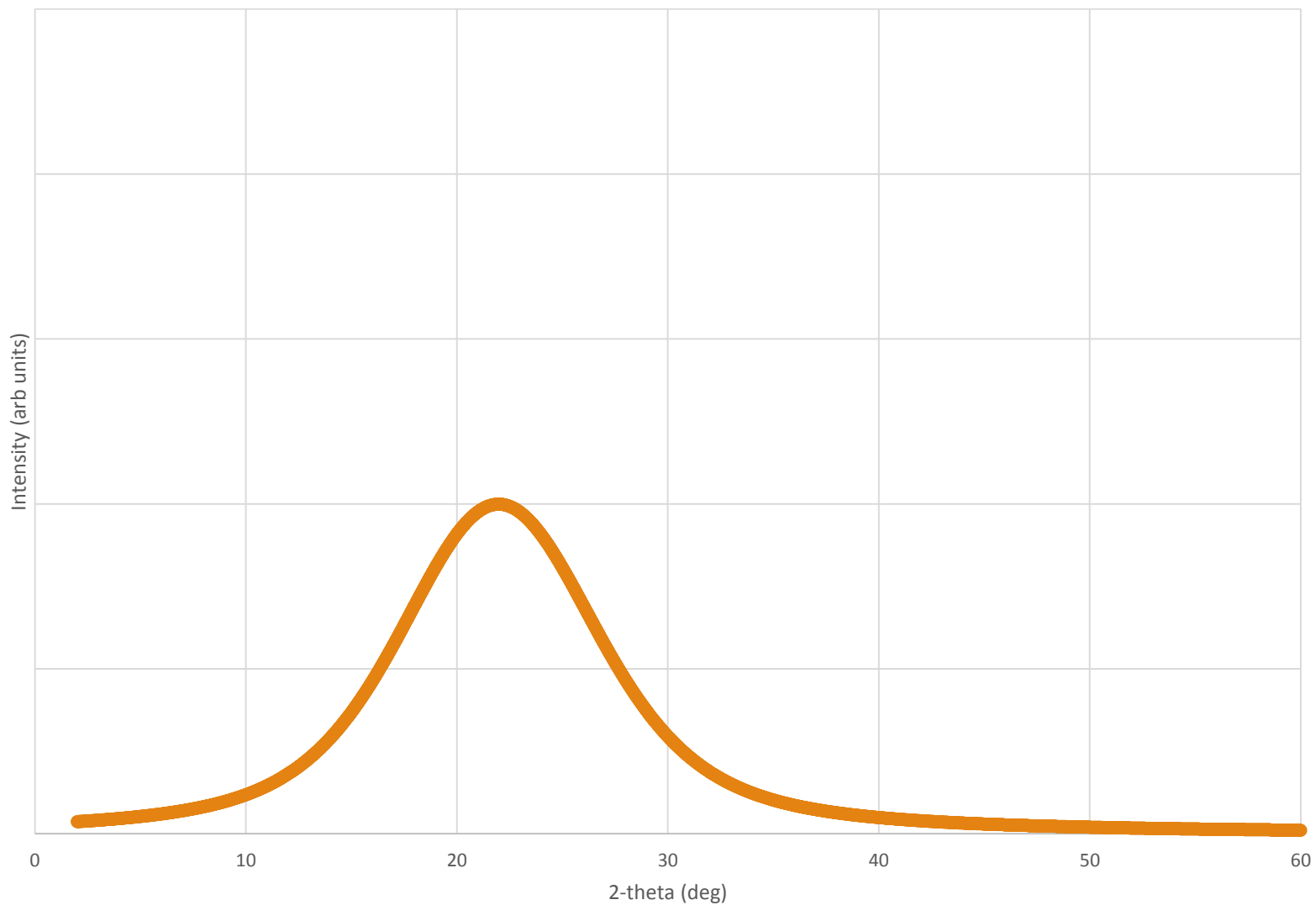


Amorphous - polycarbonate

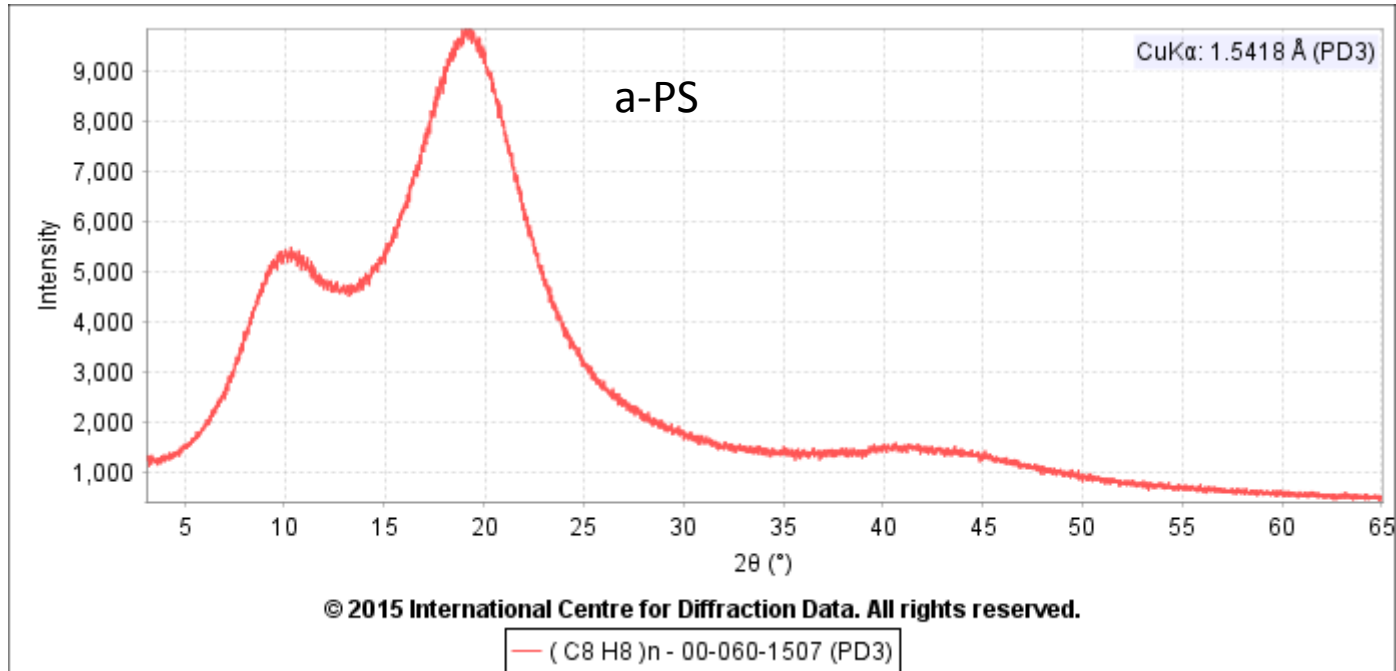




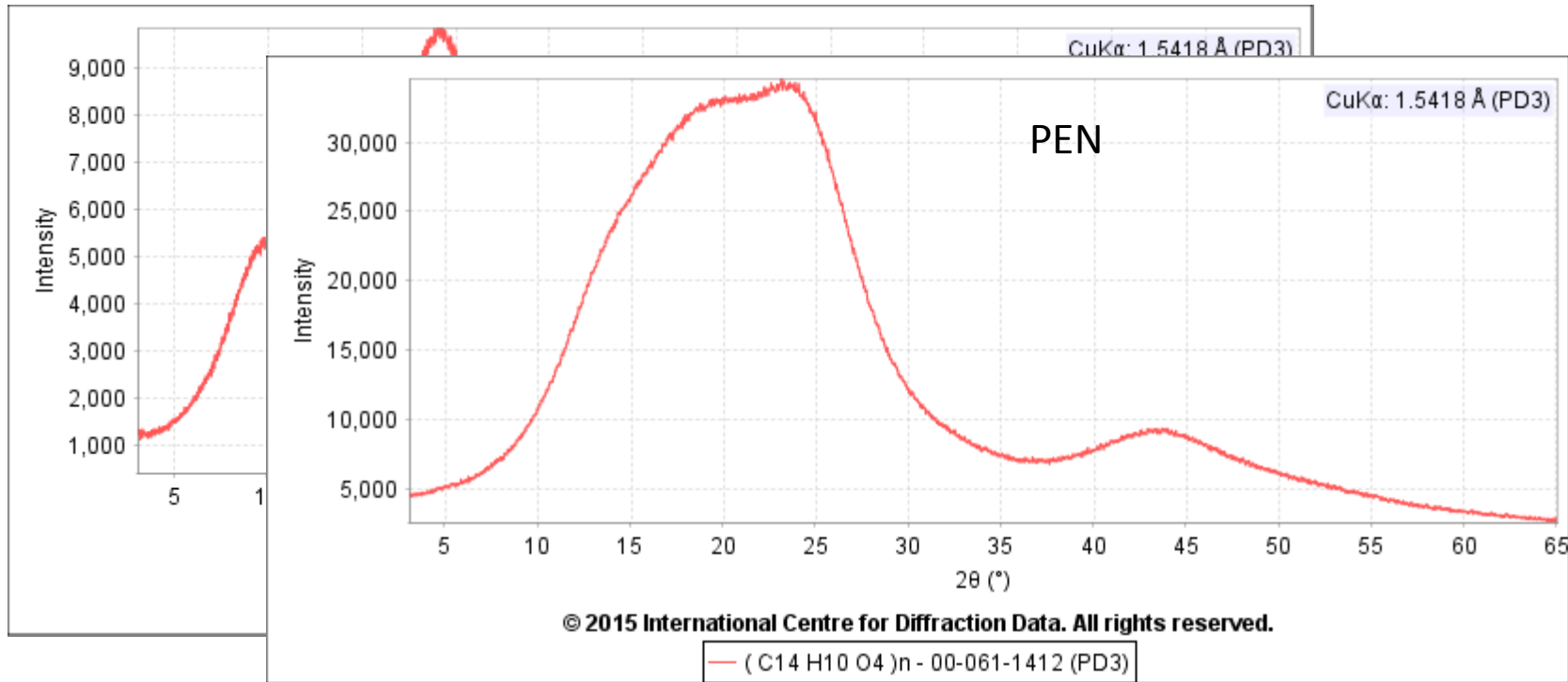
Can I just model the polymer amorphous component as a single peak?



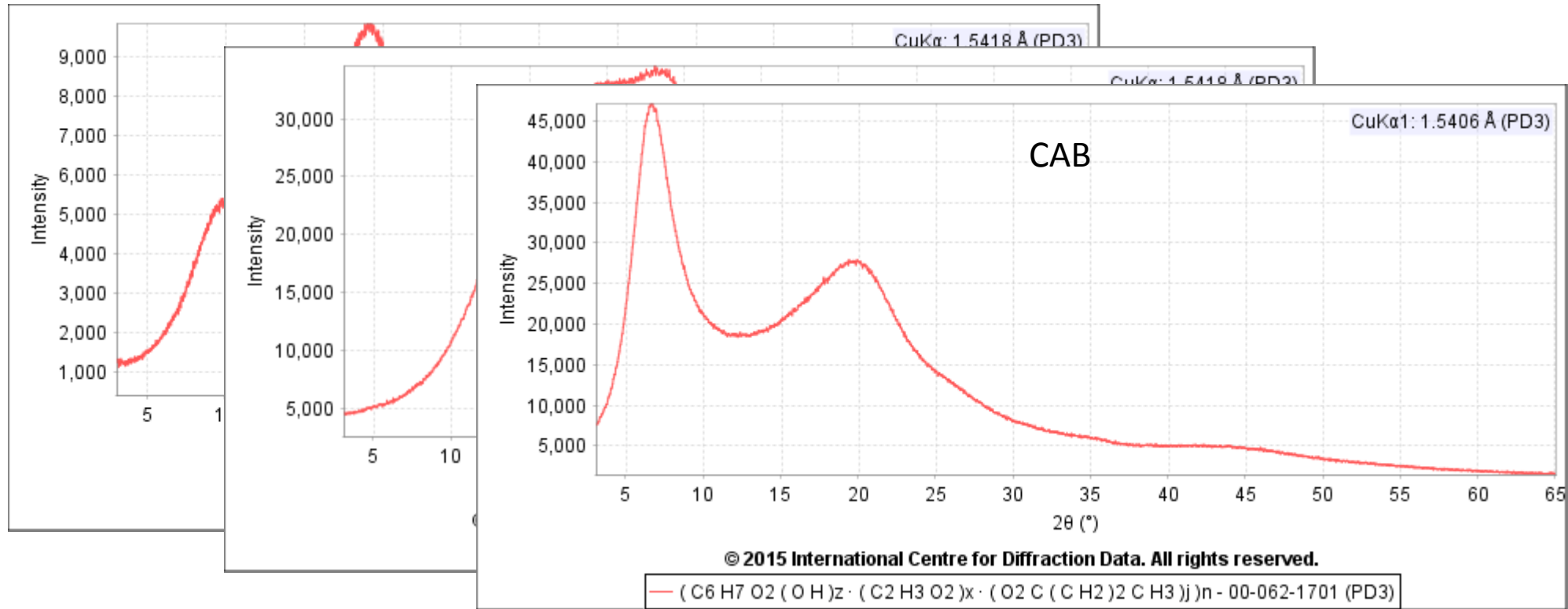
# Amorphous patterns – usually not a single peak



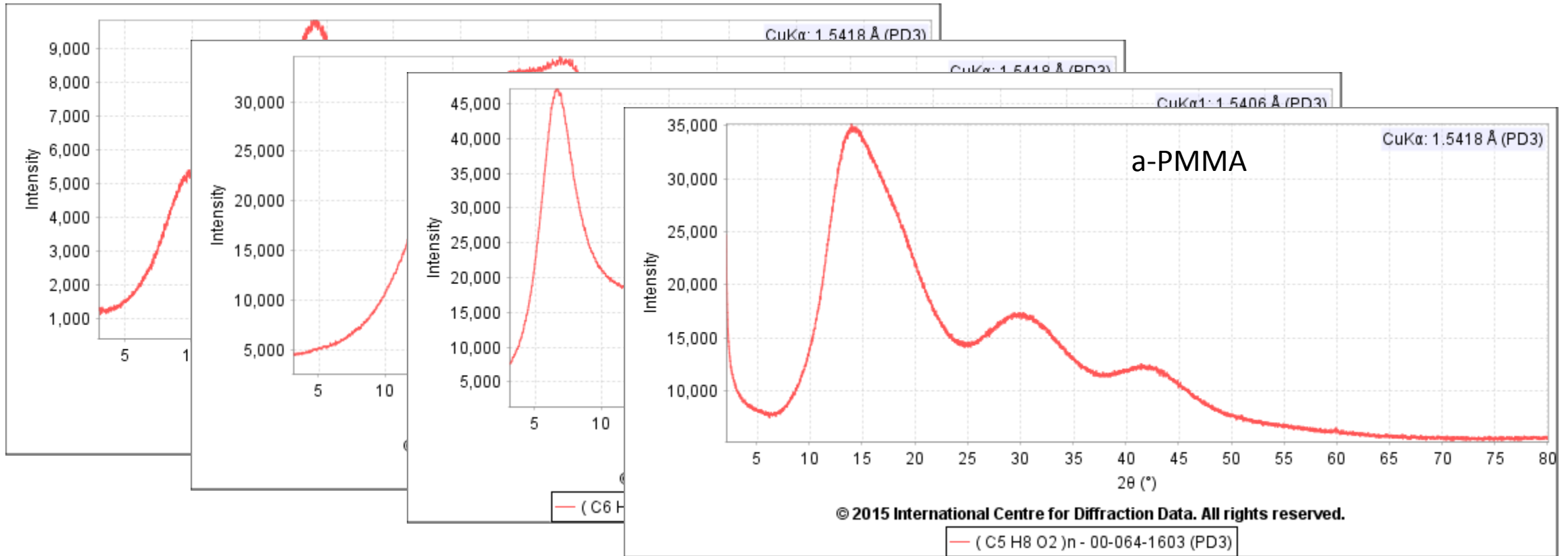
# Amorphous patterns – usually not a single peak



# Amorphous patterns – usually not a single peak

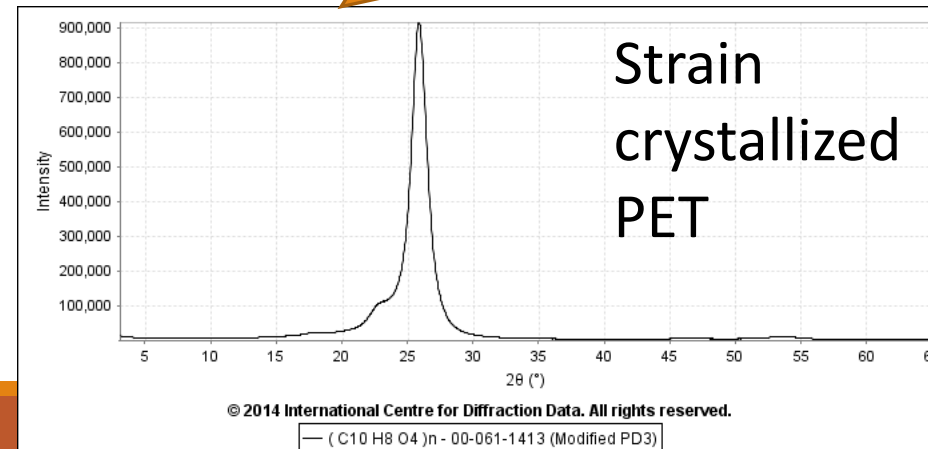
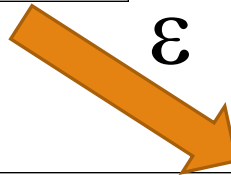
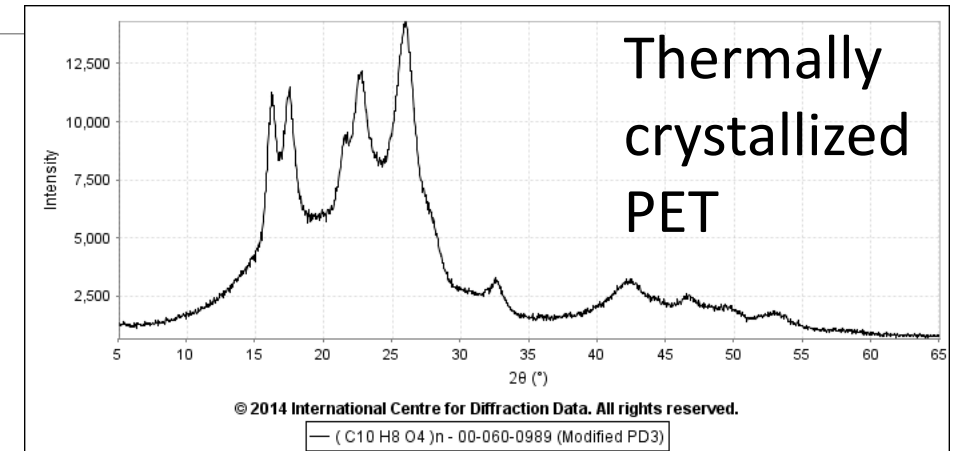
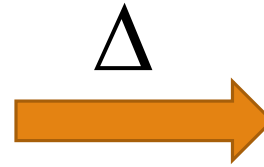
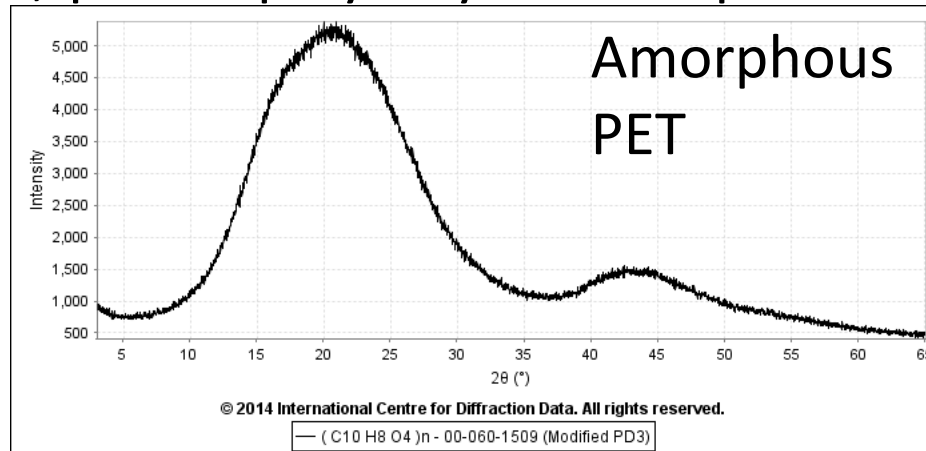


# Amorphous patterns – usually not a single peak



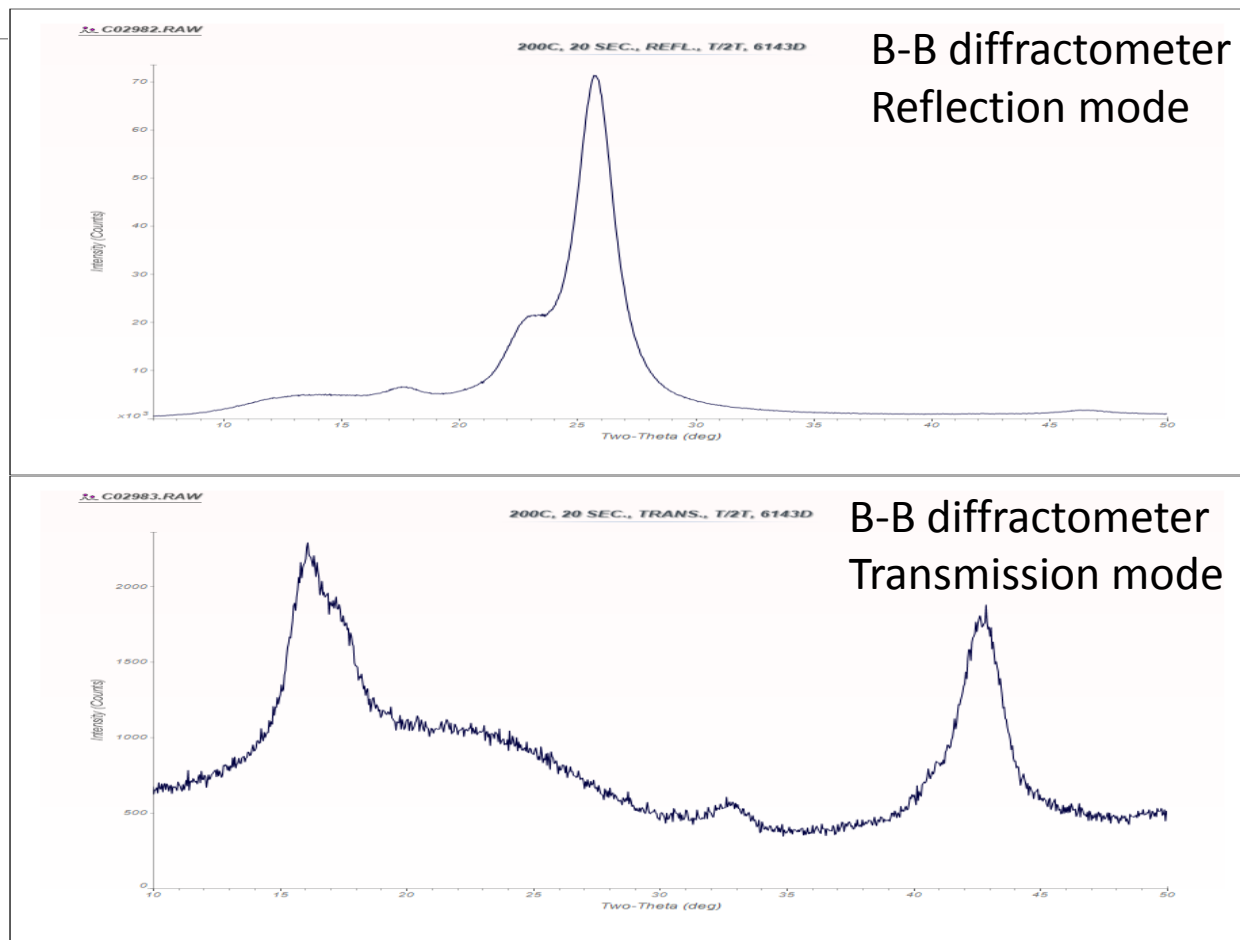
# Polymer microstructure: processing matters

Melt,quench polyethylene terephthalate (PET)

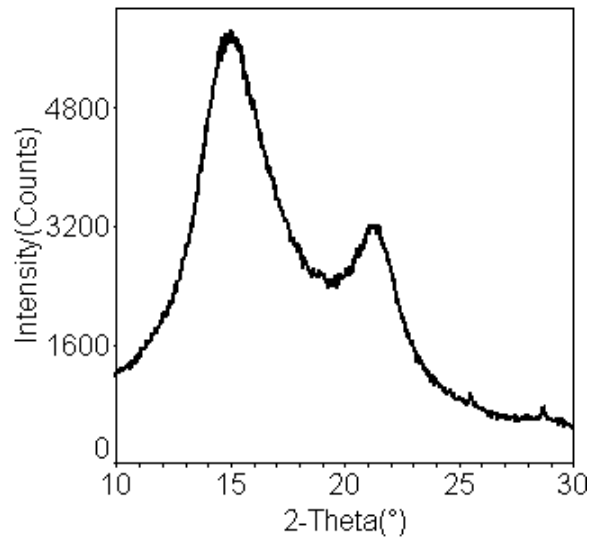


# Polymer diffraction pattern: sample orientation matters

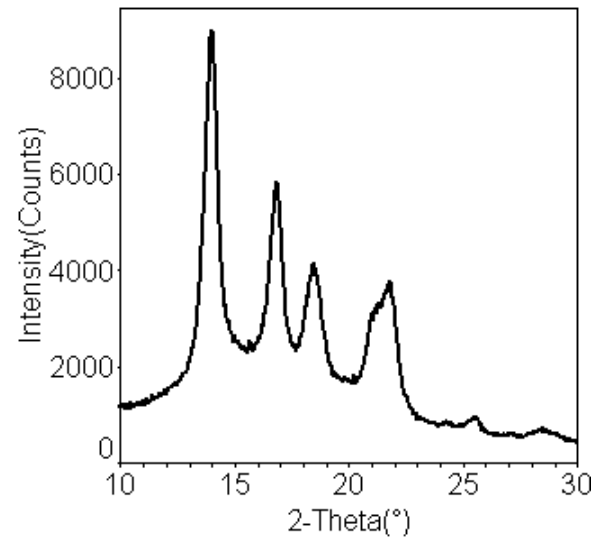
Strain crystallized PET



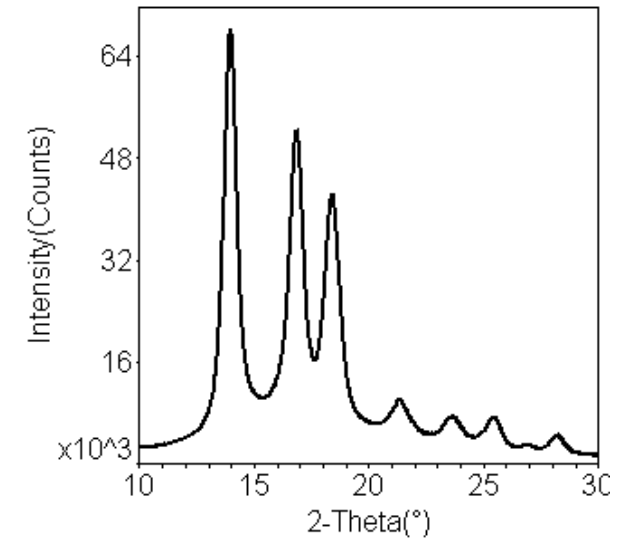
# Bragg Brentano reflection mode XRD patterns - Effect of processing and sample orientation on polypropylene XRD patterns



Cast  
No heatset  
No stretch



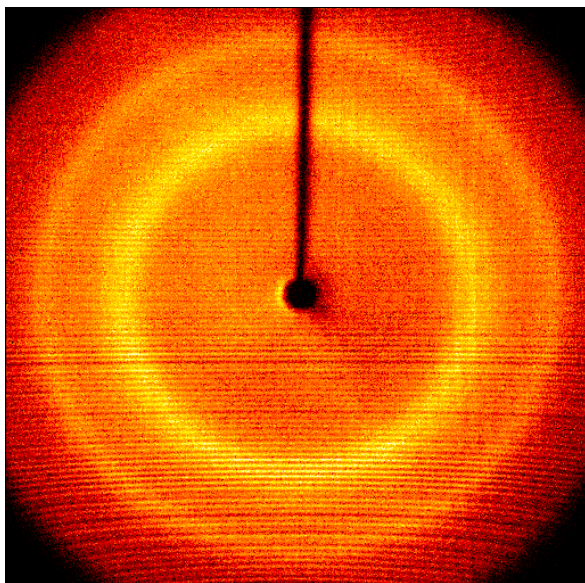
Heatset  
120 °C, 10s  
No stretch



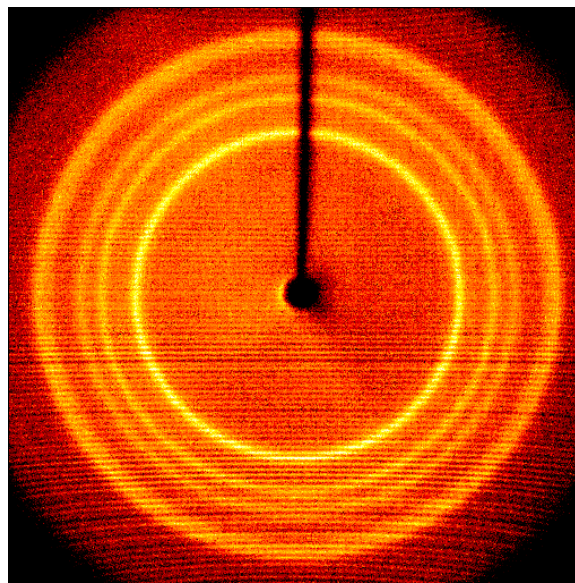
Heatset  
6X  
Uniax stretch



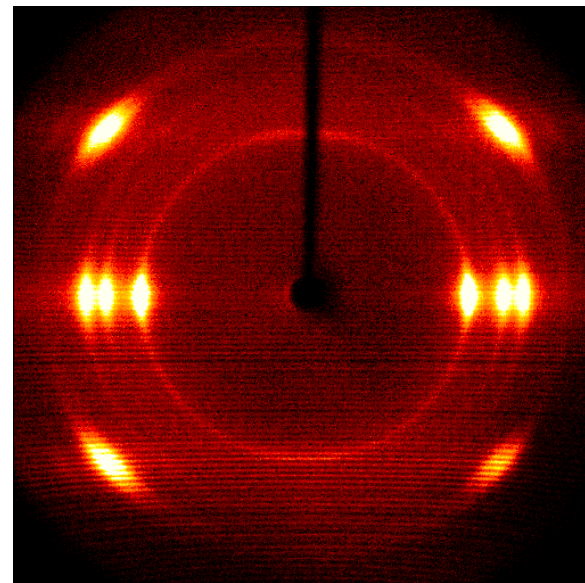
## 2-D transmission XRD patterns - Effect of processing and sample orientation on polypropylene XRD patterns



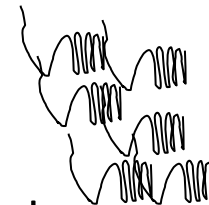
Cast  
No heatset  
No stretch



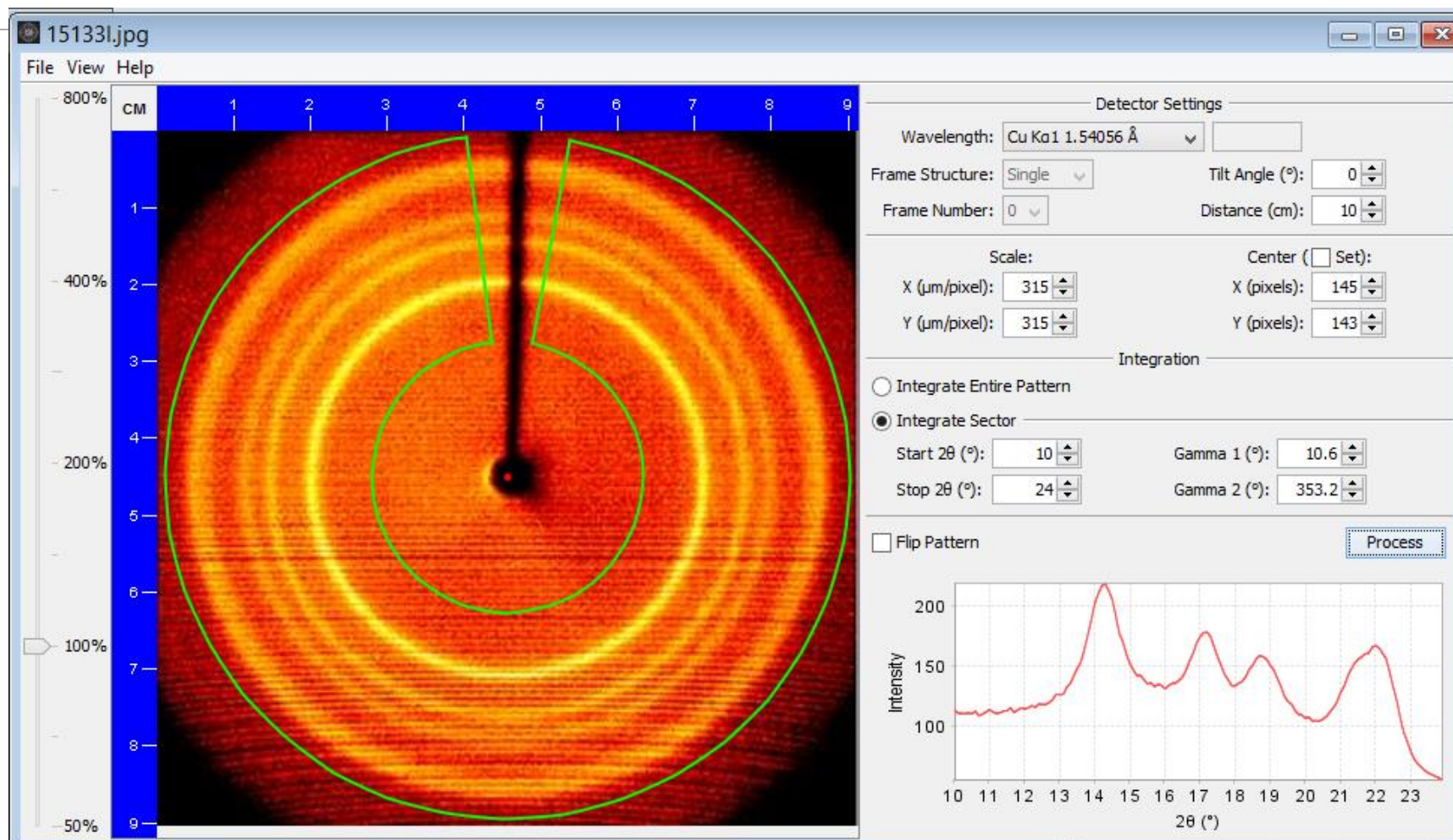
Heatset  
120 °C, 10s  
No stretch



Heatset  
6X  
Uniax stretch



# Import 2D diffraction image files for phase ID using ICDD Sieve – New feature for PDF 2015 databases



# Import 2D diffraction image files for phase ID using ICDD Sieve – New feature for PDF 2015 databases

Sieve+ - 151331.xml

File Edit Matches Phases Help

Import (1D) Import (2D) Open Session Save Session Print Preferences Accept Phase Remove Last Phase

Matches (39 of 1,302)

GOM	PDF #	QM	Status	Amb.	Coords	Compound Name	Mineral Name	Chemical Formula	I/Ic	D1 (Å)	D2 (Å)	D3 (Å)
4917	00-059-1501	B	P	A		Polypropylene		( C3 H6 )n		5.169320	6.201790	4.754090
3627	00-059-1502	I	P	A		Polypropylene		( C3 H6 )n	*1.06	6.149950	5.179810	5.427950
3031	00-050-2397	C	P	A		$\alpha$ -Polypropylene		( C3 H6 )n	1.06	6.237530	4.754940	4.059330
3021	00-054-2323	I	P	A		Nylon 1,6		( C7 H12 N2 O2 )n		4.110000	4.020000	2.400000
2967	00-046-1807	O	P	A		Poly(amidothioetheramine)		( C19 H38 N4 O2 S2 )n		4.030000	5.080000	4.600000
2934	00-048-2032	O	P	A		Poly(terephthalic acid-methylhydroxy...		( C35 H25 O10 )n		4.740000	4.600000	6.190000

Change Filter... [Subfile (Polymer)] And [Status (Primary, Alternate)]

Search Lines: 6.205... Experiment D1 Range: 6.076 Å - 6.336 Å Rotation: All

Preferences

Radiation: X-ray

Wavelength: Cu K $\alpha$ 1 1.54056 Å

Search Method: Hanawalt

Search Window: 0.3°

Match Window: 0.3°

☒ Use Residual Intensities

☐ 2nd Pass Filter

☒ Weight d-Spacings

Lowest Allowable GOM: 2000

Phases (1)

#	PDF #	QM	Compound Name	I Ratio	I %	I/Ic	Est Wt %
1	00-050-2397	C	$\alpha$ -Polypropylene	1.030	100	1.06	

Diffraction Patterns Lines (4 of 5) Estimated Weight %

# XRD analysis of polyvinyl chloride (PVC) polymer

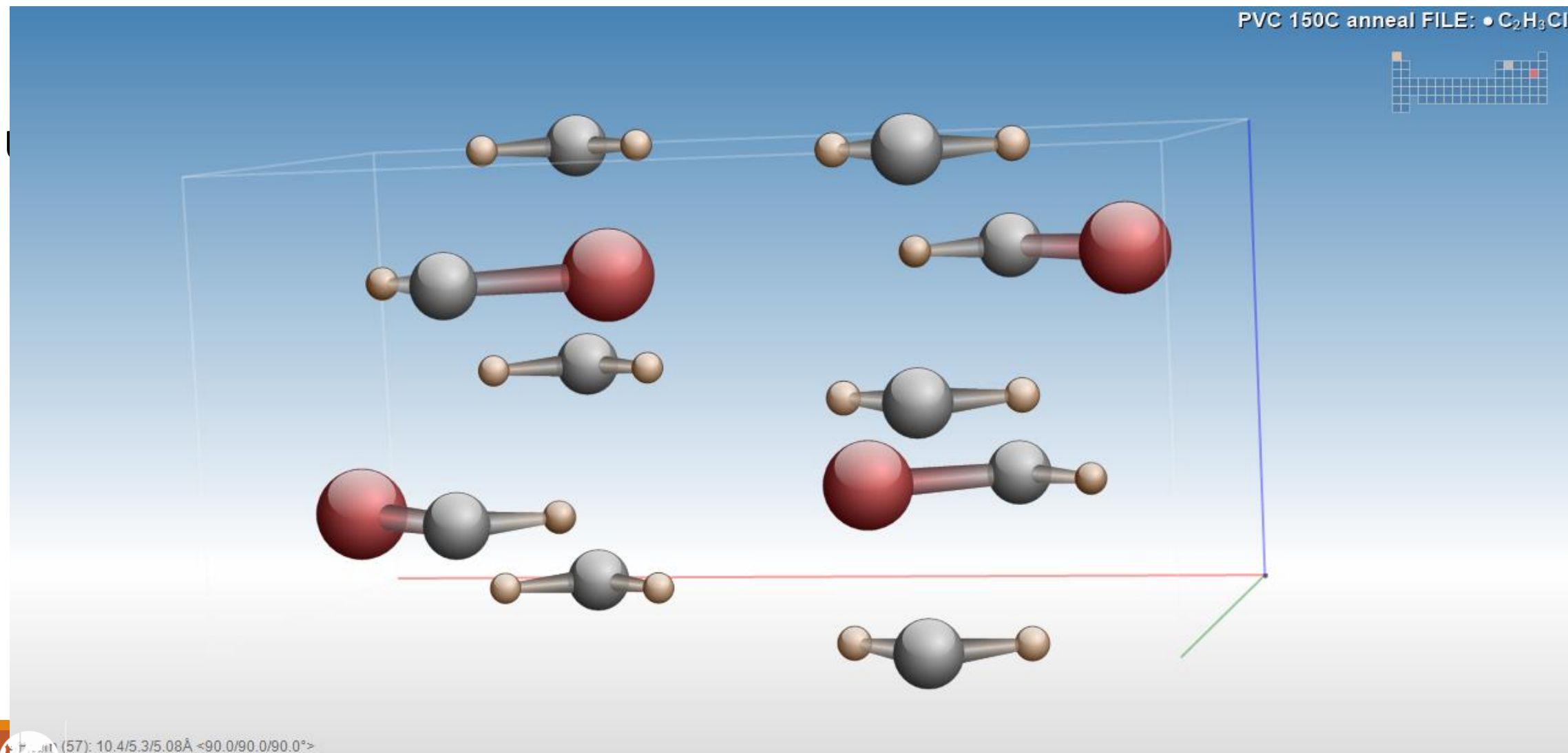
Unit cell from fiber patterns



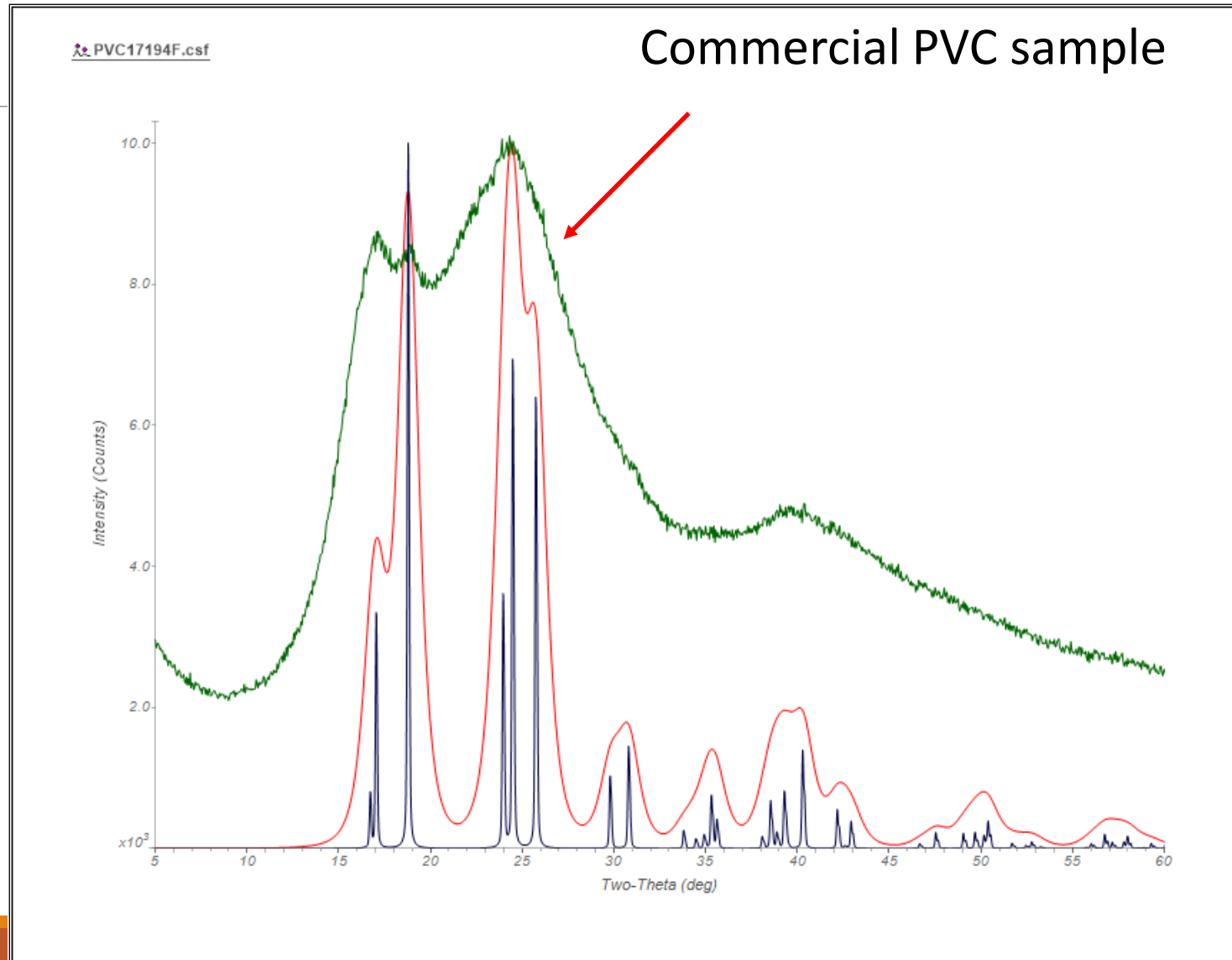
PVC 150C anneal FILE:							
C2H3Cl							
1.4824	139.5	oP12	Orthorhombic: Pcam (57)				
10.4	5.3	5.08	90.0	90.0	90.0		
(esd)	(esd)	(esd)	(esd)	(esd)	(esd)		
[PS oP12]							
6	ID	S.F.	Site	Fill	x	y	z
1	C1	C	4	1.0	0.25	0.965	0.0
2	C2	C	4	1.0	0.25	0.126	0.25
3	Cl	Cl	4	1.0	0.393	0.313	0.25
4	H1	H	8	0.5	0.16	0.85	0.0
5	H2	H	8	0.5	0.34	0.85	0.0
6	H3	H	4	1.0	0.16	0.241	0.25



# XRD analysis of polyvinyl chloride (PVC) polymer

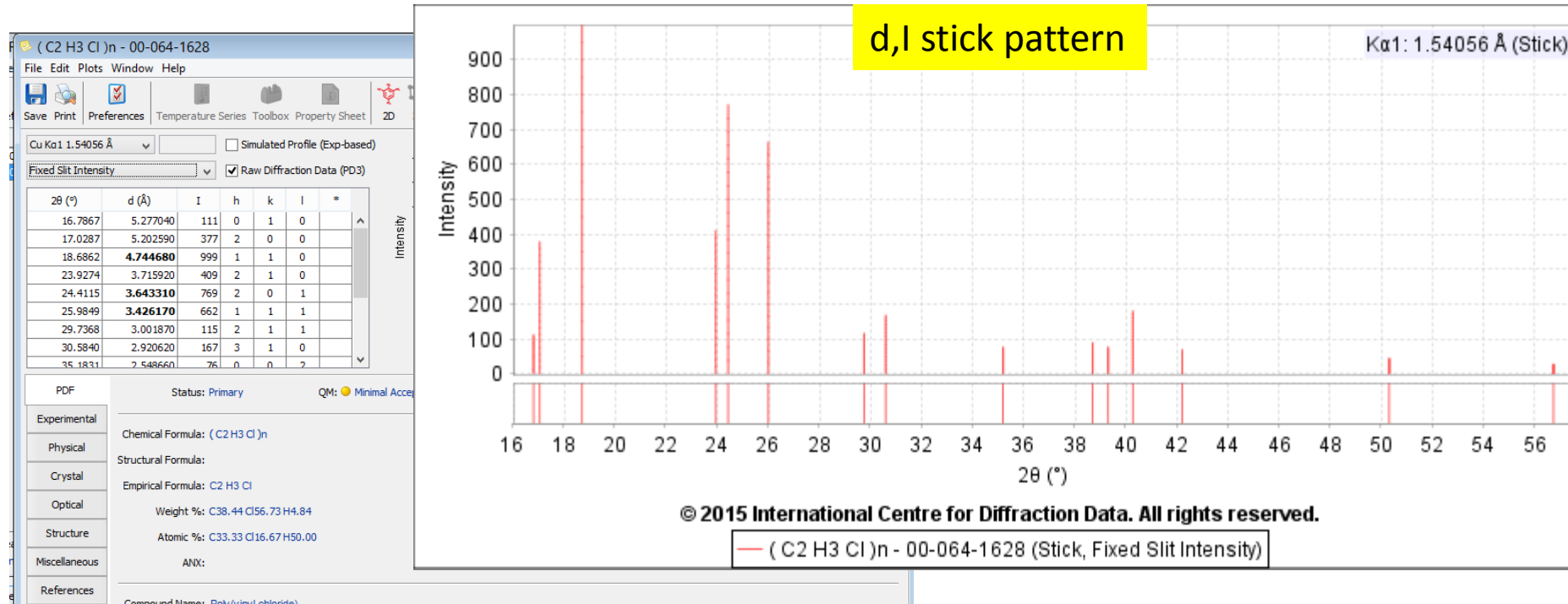


# PVC – XRD powder patterns calculated vs. observed



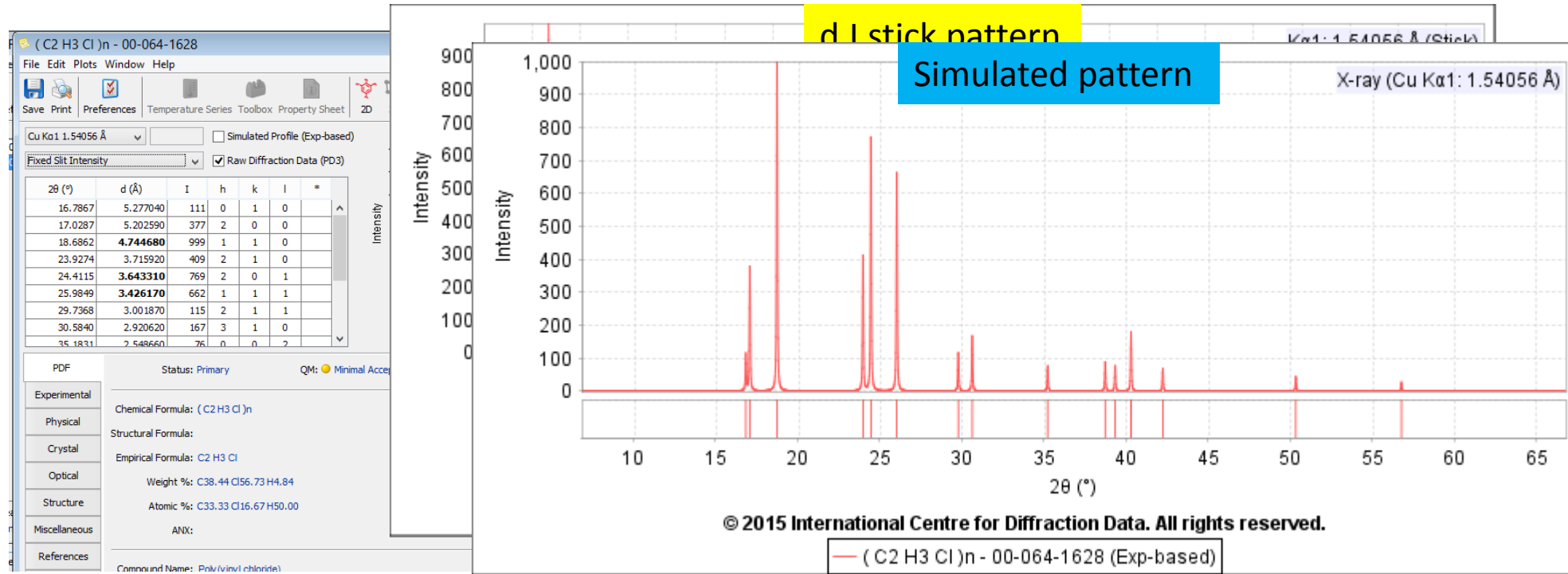
# Diffraction polymer entries in ICDD PDF databases

## Polyvinyl chloride



# Diffraction polymer entries in ICDD PDF databases

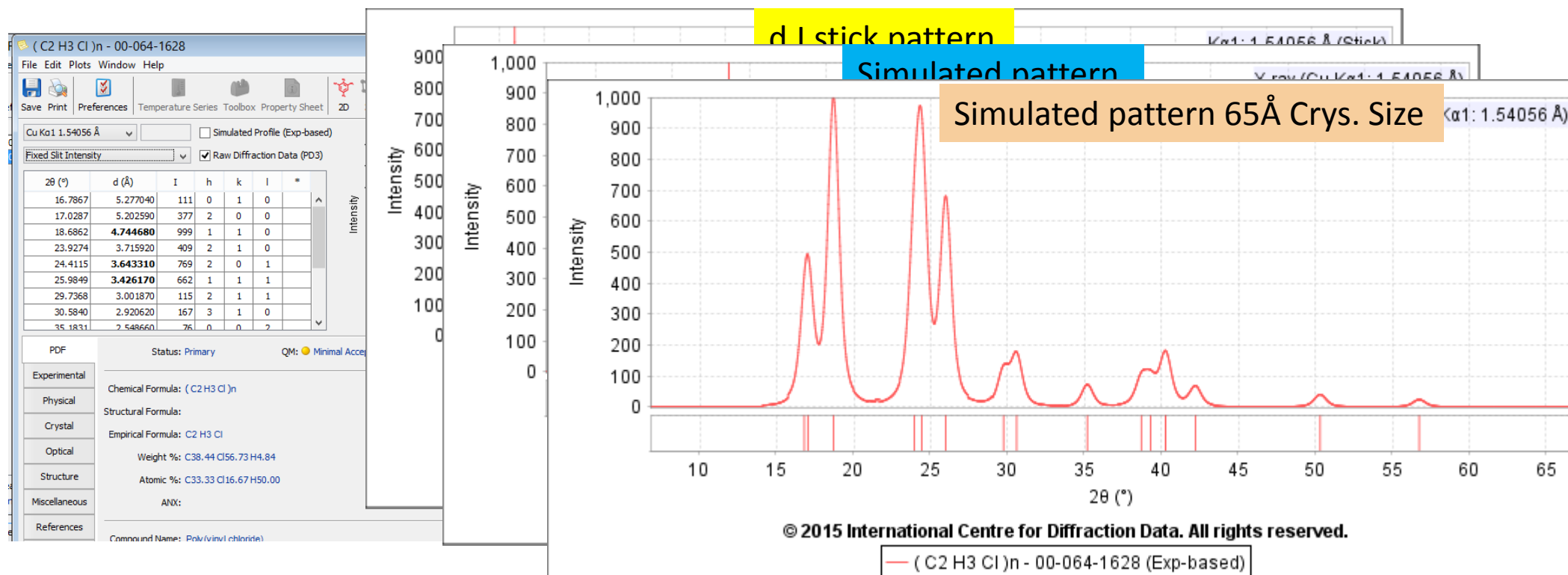
## Polyvinyl chloride





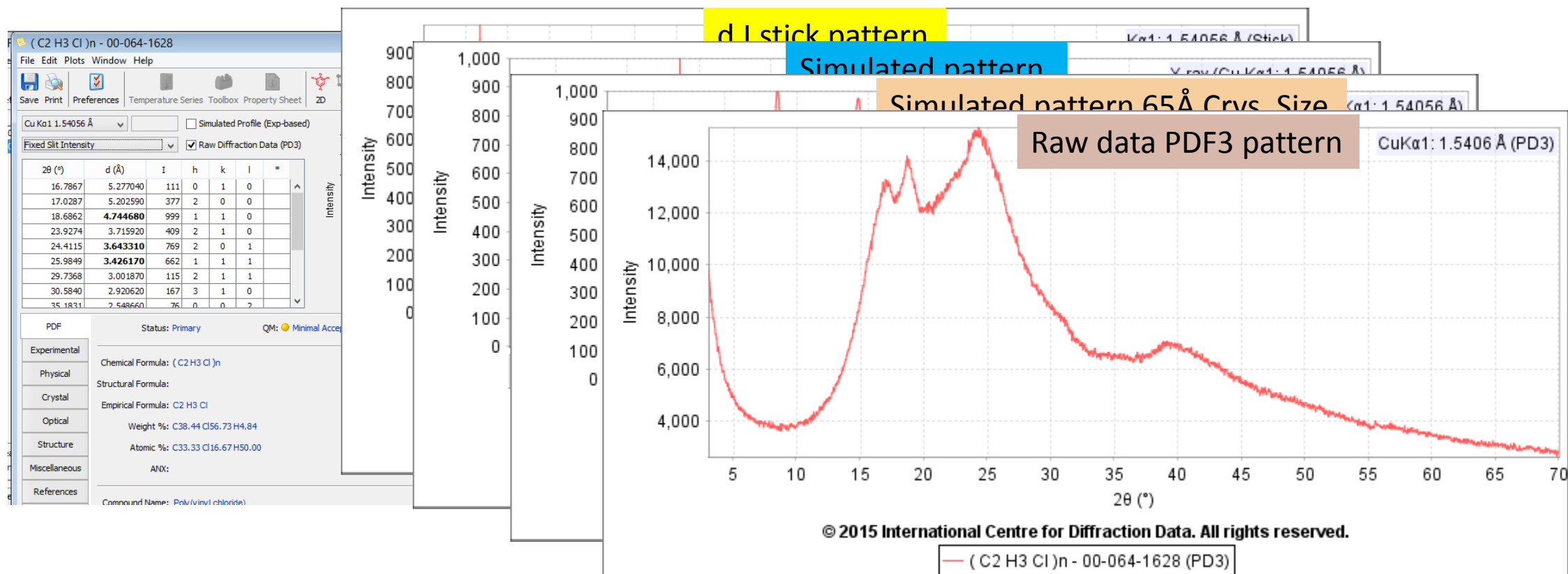
# Diffraction polymer entries in ICDD PDF databases

## Polyvinyl chloride



# Diffraction polymer entries in ICDD PDF databases

## Polyvinyl chloride



# Polymer entries in PDF 2015

Subfile search

Entries with raw data

Quality mark

PDF-4+ 2015

File Edit Tools Window Help

Open PDF Cards Preferences Search History Results Sieve+

Search

Subfile

Environment

- ☐ Ambient
- ☐ Press. (Non-ambient)
- ☐ Temp. (Non-ambient)
- ☐ Press. & Temp. (Non-ambient)
- ☐ Atomic Coordinates
- ☐ Raw Diffraction Data

Status

- ☒ Primary
- ☒ Alternate
- ☒ Deleted

Quality Mark

- ☒ Star
- ☐ Rietveld
- ☐ Good
- ☐ Indexed
- ☐ Calculated
- ☐ Prototyping
- ☐ Minimal Acceptable
- ☐ Blank

Database

- ☐ ICDD (00)
- ☐ ICSD (01)
- ☐ CSD (02)
- ☐ NIST (03)
- ☐ LPF (04)
- ☐ ICDD Crystal Data (05)

Modulated Structure

- NBS
- Nucleosides & Nucleotides
- Organics
- Pharmaceutical
- Pigment & Dye
- Polymer
- Porphyrins, Corrins & Complexes
- Steroids

Periodic Table

Chemistry

Crystallography

Modulated

Diffraction

Physical Properties

Reference

Comments

Search

Reset Tab

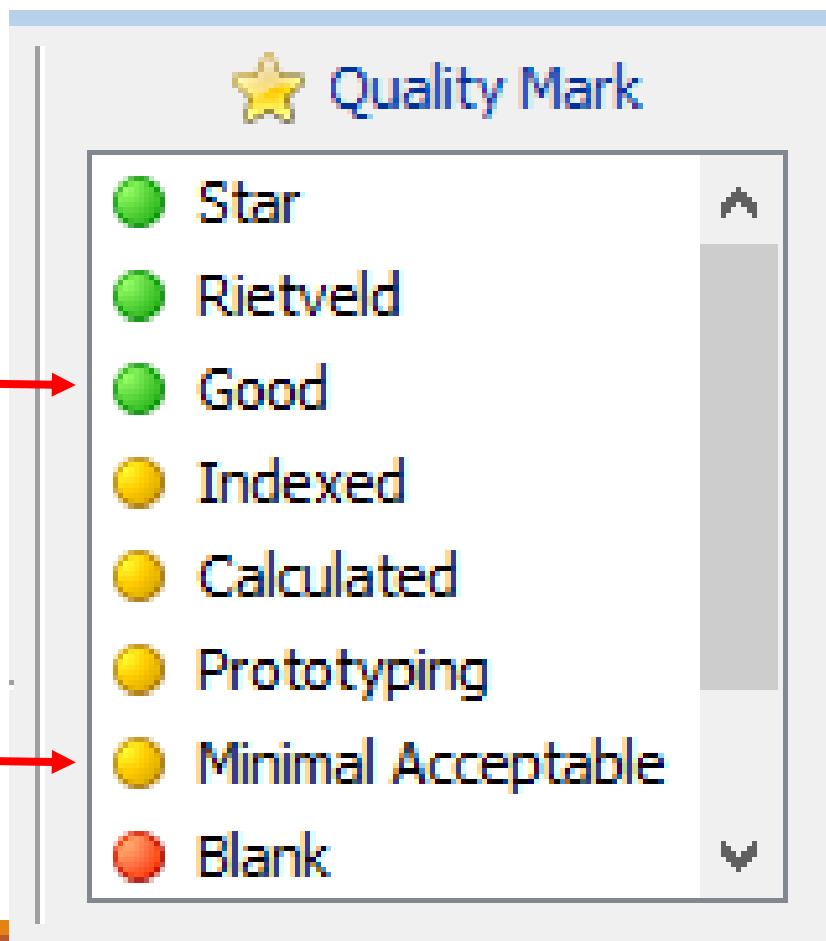
Reset All

Help

Numeric Input Global Operator

[Subfile (Polymer)] And [Status (Primary, Alternate, Deleted)]

# Quality Marks



**Good (G).** Indicates the material has significant amorphous content, and has a digital diffraction pattern with a good signal-to-noise ratio. Additionally, some chemical analysis information has been provided to support the specified composition of the material (i.e. spectroscopy, pair distribution functions, commercial source, etc.).

**Minimal Acceptable (M).** Indicates the material has significant amorphous content, and has a digital diffraction pattern with a good signal-to-noise ratio. However, no chemical analysis data to support the materials composition was provided.

# Polymer entries in PDF 2015

Results - [Subfile (Polymer)] And [Status (Primary, Alternate, Deleted)]

File Edit Fields Similarity Index Help

☒ Preferences
 ☐ Open PDF Card
 ☐ Simulated Profile

Results: 1,325 of 365,877

ICDD Defaults

PDF #	QM	Chemical Formula	Compound Name	D1 (Å)	D2 (Å)	D3 (Å)	SYS
00-003-0193	● O	( C4 H5 Cl )x	Polychloroprene	4.450000	4.070000	3.360000	O
00-003-0203	● B	( C12 H24 O12 · H Cl O4 )x	Cellulose perchloric acid	4.420000	4.650000	3.560000	M
00-003-0215	● B	C12 H22 O11	Cellobiose	4.370000	8.380000	4.740000	M
00-003-0226	● B	( C6 H10 O5 )x	Cellulose	4.300000	5.140000	7.550000	M
00-003-0254	● O	( C H2 )x	Paraffin wax	4.150000	3.730000	3.880000	X
00-003-0259	● O	( C H2 )x	Paraffin wax	4.130000	3.730000	3.480000	X
00-003-0289	● O	( C6 H12 O6 )x	Native cellulose	3.890000	5.940000	2.570000	M
00-004-0419	● O	( C H2 O )x	Paraformaldehyde	3.760000	2.590000	1.870000	X
00-007-0506	● O	C30 H57 N5 O6	ε-Polyaminocaproic acid	3.690000	4.470000	3.600000	X
00-007-0511	● O	C24 H46 N4 O5	ε-Polyaminocaproic acid	4.430000	3.690000	4.470000	X
00-008-0689	● O	C57 H108 O6	β-2-Oleyl-1,3-distearyltriglycerol	4.600000	5.420000	5.030000	M
00-009-0853	● O	( C4 H6 )n	1,4-cis-Polybutadiene	3.980000	4.750000	4.070000	X
00-011-0834	● O	( C2 H4 )n	β-Polyethylene	4.100000	3.600000	2.490000	X
00-012-0876	● O	( C2 H3 Cl O )n	Poly monochloroacetaldehyde	7.760000	3.850000	3.450000	X
00-012-0877	● O	( C4 H8 O )n	Butyraldehyde polymer	12.000000	9.000000	4.800000	X
00-012-0878	● O	( C4 H8 O )n	Polyisobutyraldehyde	9.300000	7.900000	4.600000	X
00-012-0879	● O	( C2 H2 Cl2 O )n	Poly dichloroacetaldehyde	8.320000	4.000000	3.130000	X
00-012-0880	● O	( C7 H14 O )n	Polyheptylaldehyde	13.100000	11.500000	4.180000	X
00-012-0896	● B	( C3 H6 O )n	d,l-Poly(propylene oxide)	4.210000	5.180000	2.072000	O
00-013-0675	● I	( C H2 )n	Paraffin	4.180000	3.740000	2.250000	M
00-013-0684	● I	( C8 H8 )n	α-Poly-p-xylylene	5.380000	3.970000	5.060000	M
00-013-0686	● B	( C2 H4 O )n	Metalddehyde	7.500000	3.880000	3.350000	T
00-013-0743	● O	C5 H9 N O2	Poly-L-proline	5.800000	4.900000	3.650000	X
00-013-0744	● O	( C5 H9 N O2 )n	Poly-L-proline	8.400000	4.940000	3.220000	X

Search Description: [Subfile (Polymer)] And [Status (Primary, Alternate, Deleted)]

Calculations: Mean: Median: ESD:

# Polymer entries in PDF 2015

Results - [Subfile (Polymer)] And [Has Raw Diffraction Data] And [Status (Primary, Alternate, Deleted)]

File Edit Fields Similarity Index Help

Preferences Open PDF Card Simulated Profile

With PDF3 raw data

Results: 125 of 365,877

ICDD Defaults

PDF #	QM	Chemical Formula	Compound Name	D1 (Å)	D2 (Å)	D3 (Å)	SYS
00-061-1416	B	(C <sub>3</sub> H <sub>6</sub> ) <sub>n</sub>	α-Polypropylene	6.244550	5.226510	4.189310	M
00-062-0923	R	C <sub>45</sub> H <sub>86</sub> O <sub>6</sub>	β-1,2,3-tris Tetradecanoyl glycerol	4.589920	4.549930	3.684620	A
00-062-0924	R	C <sub>57</sub> H <sub>110</sub> O <sub>6</sub>	β-1,2,3-Trioctadecanoyl-glycerol	4.582690	4.556430	3.832850	A
00-062-1286	R	(C <sub>6</sub> H <sub>10</sub> O <sub>2</sub> ) <sub>n</sub>	Poly-ε-caprolactone	4.140580	3.735750	4.026870	O
00-062-1287	R	C <sub>2.12</sub> H <sub>4.12</sub> O <sub>0.12</sub>	Ethylene vinyl acetate	4.132970	3.747230	2.477160	O
00-062-1288	M	C <sub>2.22</sub> H <sub>4.22</sub> O <sub>0.22</sub>	Ethylene vinyl acetate	4.449160	2.217140	14.018100	X
00-062-1289	M	C <sub>2.36</sub> H <sub>4.36</sub> O <sub>0.36</sub>	Ethylene vinyl acetate	4.564780	16.413000	2.229220	X
00-062-1290	M	(C <sub>8.45</sub> H <sub>14.9</sub> O <sub>5</sub> ) <sub>n</sub>	Methyl cellulose, amorphous	9.814620	4.536000	3.364680	X
00-062-1291	I	(C <sub>8.45</sub> H <sub>14.9</sub> O <sub>5</sub> ) <sub>n</sub>	Methyl cellulose	10.369800	4.464680	4.107080	O
00-062-1292	B	(C <sub>22</sub> H <sub>10</sub> N <sub>2</sub> O <sub>5</sub> ) <sub>n</sub>	Kapton	6.062230	4.073580	5.674000	O
00-062-1293	B	(C <sub>22</sub> H <sub>10</sub> N <sub>2</sub> O <sub>5</sub> ) <sub>n</sub>	Kapton	16.322000	6.074650	5.443550	O
00-062-1701	G	(C <sub>6</sub> H <sub>7</sub> O <sub>2</sub> (O H)z · (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )x...	Cellulose acetate butyrate	13.450500	4.414020		X
00-062-1702	G	(C <sub>6</sub> H <sub>7</sub> O <sub>2</sub> (O H)z · (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )x...	Cellulose acetate butyrate	13.268800	4.506810		X
00-062-1703	M	(C <sub>6</sub> H <sub>7</sub> O <sub>2</sub> (O H)z · (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )x...	Cellulose acetate butyrate	13.417800	4.435870	4.328790	X
00-062-1704	M	(C <sub>6</sub> H <sub>7</sub> O <sub>2</sub> (O H)z · (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )x...	Cellulose acetate propionate	11.570400	4.185460	7.787560	X
00-062-1705	M	(C <sub>4</sub> H <sub>8</sub> ) <sub>n</sub>	Poly(butene-1)	8.845650	4.356160	4.466800	X
00-062-1706	M	(O C H <sub>2</sub> ) <sub>n</sub>	Poly(oxyethylene)	3.930910	2.630020	1.899100	X
00-062-1707	M	(O C <sub>5</sub> H <sub>6</sub> Cl <sub>4</sub> ) <sub>n</sub>	Poly (3,3-bis(chloromethyl)oxetane)	5.888960	4.036160	3.997390	X
00-062-1708	M	(C <sub>4</sub> H <sub>8</sub> O) <sub>n</sub>	Poly(tetrahydrofuran)	4.492410	4.561160	3.688190	X
00-062-1709	M	(C <sub>2</sub> H <sub>2</sub> C H (C <sub>6</sub> H <sub>4</sub> ) C H <sub>3</sub> ) <sub>n</sub>	Poly(o-vinyl toluene)	5.933620	5.760570	6.142820	X
00-062-1710	M	(C <sub>2</sub> H <sub>2</sub> C Cl <sub>2</sub> ) <sub>n</sub>	Poly(vinylidene chloride)	5.643990	13.657800	10.203600	X
00-062-1711	M	(C <sub>2</sub> H <sub>2</sub> C H (C <sub>6</sub> H <sub>5</sub> ) <sub>n</sub> (C <sub>2</sub> H <sub>2</sub> C H ...	Poly(styrene-acrylic acid)	4.785400	8.293680	9.837960	X
00-062-1712	G	(C <sub>4</sub> H <sub>8</sub> O <sub>2</sub> ) <sub>n</sub> · (C <sub>2</sub> H <sub>4</sub> O <sub>2</sub> ) <sub>n</sub>	Cellulose acetate butyrate	11.097800	4.970540	10.695400	X

Search Description: [Subfile (Polymer)] And [Has Raw Diffraction Data] And [Status (Primary, Alternate, Deleted)]

Calculations: Mean: Median: ESD:

# 2015 – ICDD pharmaceutical polymers project

Polymer	Mol. Formula	PDF Entry	PD3
poly(acrylic acid), PAA	(C <sub>3</sub> H <sub>4</sub> O <sub>2</sub> ) <sub>n</sub>	N	N
poly(ethylene oxide), PEO	(C <sub>2</sub> H <sub>4</sub> O) <sub>n</sub>	Y	N
poly(ethylene glycol), PEG	(C <sub>2</sub> H <sub>4</sub> O) <sub>n</sub>	Y	N
poly(vinyl pyrrolidone), PVP	(C <sub>6</sub> H <sub>9</sub> NO) <sub>n</sub>	N	N
poly(vinyl alcohol), PVOH, PVA	(C <sub>2</sub> H <sub>4</sub> O) <sub>n</sub>	Y	Y
polyacrylamide, PAM	(C <sub>3</sub> H <sub>5</sub> NO) <sub>n</sub>	N	N
poly(N-isopropylacrylamide)	(C <sub>6</sub> H <sub>11</sub> NO) <sub>n</sub>	N	N
cellulose	(C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> ) <sub>n</sub>	Y	Y
methyl cellulose	(C <sub>6</sub> H <sub>7</sub> O <sub>5</sub> R <sub>1, 2, 3</sub> ) <sub>n</sub> R=CH <sub>3</sub>	Y	Y
ethyl cellulose	((C <sub>6</sub> H <sub>8</sub> O <sub>5</sub> (C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> ) <sub>n</sub>	N	N
carboxymethyl cellulose	(C <sub>6</sub> H <sub>7</sub> O <sub>5</sub> R <sub>1, 2, or 3</sub> ) <sub>n</sub> R=H or CH <sub>2</sub> CO <sub>2</sub> H	N	N
hydroxyethyl cellulose	(C <sub>6</sub> H <sub>7</sub> O <sub>5</sub> R <sub>1, 2, or 3</sub> ) <sub>n</sub> R=H or CH <sub>2</sub> CH <sub>2</sub> OH	N	N
hydroxypropyl cellulose	(C <sub>6</sub> H <sub>7</sub> O <sub>5</sub> R <sub>1,2, or 3</sub> ) <sub>n</sub> R=H or CH <sub>2</sub> CH(OH)CH <sub>3</sub>	N	N
hydroxypropyl methyl cellulose, HPMC	(C <sub>6</sub> H <sub>7</sub> O <sub>5</sub> R <sub>1,2, or 3</sub> ) <sub>n</sub> R=H or CH <sub>3</sub> or CH <sub>2</sub> CH(OH)CH <sub>3</sub>	N	N
cellulose acetate phthalate	(C <sub>6</sub> H <sub>7</sub> O <sub>5</sub> R <sub>1,2, or 3</sub> ) <sub>n</sub> R=H or CH <sub>3</sub> CO or C <sub>6</sub> H <sub>4</sub> COCOOH	Y	Y
alginic acid	(C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> ) <sub>n</sub>	N	N
chitosan	(C <sub>6</sub> H <sub>11</sub> O <sub>4</sub> N) <sub>n</sub>	Y	N
hyaluronic acid	(C <sub>14</sub> H <sub>21</sub> NO <sub>11</sub> ) <sub>n</sub>	N	N
pectinic acid	(C <sub>13</sub> H <sub>14</sub> O <sub>13</sub> ) <sub>n</sub>	N	N
poly(lactide-co-glycolic acid, PLGA)	(C <sub>3</sub> H <sub>4</sub> O <sub>2</sub> ) <sub>m</sub> (C <sub>2</sub> H <sub>2</sub> O <sub>2</sub> ) <sub>n</sub>	N	N
starch	(C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> ) <sub>n</sub>	Y	N
sodium starch glycolate	(C <sub>2</sub> H <sub>4</sub> O <sub>3</sub> Na) <sub>n</sub>	N	N
dextran	H(C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> ) <sub>n</sub> OH	Y	Y
xanthum Gum	C <sub>35</sub> H <sub>49</sub> O <sub>29</sub> (monomer)	N	N
gelatin	(C <sub>35</sub> H <sub>55</sub> N <sub>12</sub> O <sub>12</sub> ) <sub>n</sub>	Y	Y



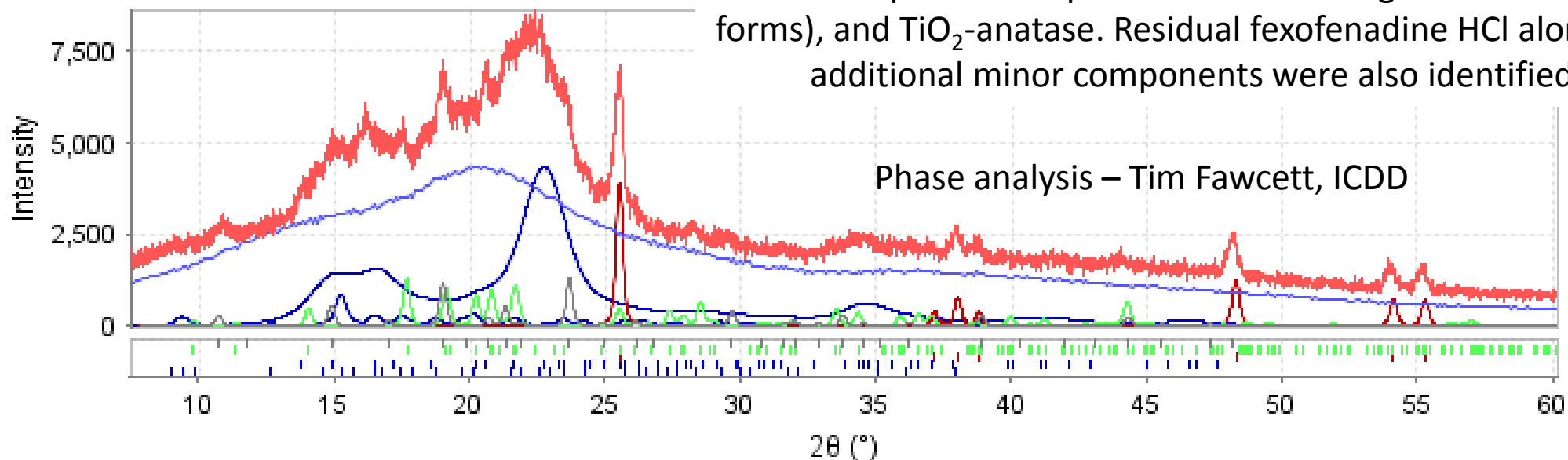
# 2015 – ICDD biomedical polymers

Polymer	Mol. Formula	PDF Entry	PD3
polyurethane, PU	(R-NHCO <sub>2</sub> ) <sub>n</sub>	Y	N
silicone	(OSiR <sub>2</sub> ) <sub>n</sub>	Y	N
polycarbonate, PC	(ROCO <sub>2</sub> ) <sub>n</sub>	Y	Y
polychloroprene	(C <sub>4</sub> H <sub>5</sub> Cl) <sub>n</sub>	Y	N
polyisobutylene, PIB	(CH <sub>2</sub> C(CH <sub>3</sub> ) <sub>2</sub> ) <sub>n</sub>	Y	N
polycyanoacrylate	(C <sub>5</sub> H <sub>5</sub> O <sub>2</sub> N) <sub>n</sub>	N	N
poly(vinyl acetate), PVAc	(C <sub>4</sub> H <sub>6</sub> O <sub>2</sub> ) <sub>n</sub>	N	N
polystyrene, PS atactic	(C <sub>8</sub> H <sub>8</sub> ) <sub>n</sub>	Y	Y
polystyrene, PS isotactic	(C <sub>8</sub> H <sub>8</sub> ) <sub>n</sub>	Y	N
polypropylene, PP	(C <sub>3</sub> H <sub>6</sub> ) <sub>n</sub>	Y	Y
poly(vinyl chloride), PVC	(C <sub>2</sub> H <sub>3</sub> Cl) <sub>n</sub>	Y	Y
polyethylene	(C <sub>2</sub> H <sub>4</sub> ) <sub>n</sub>	Y	Y
poly (methyl methacrylate)	(C <sub>5</sub> H <sub>8</sub> O <sub>2</sub> ) <sub>n</sub>	Y	N
poly(hydroxyethyl methacrylate)	(C <sub>6</sub> H <sub>10</sub> O <sub>3</sub> ) <sub>n</sub>	N	N
Ethylene vinyl acetate, EVA	(C <sub>2</sub> H <sub>4</sub> ) <sub>m</sub> (CC <sub>4</sub> H <sub>6</sub> O <sub>2</sub> ) <sub>n</sub>	Y	Y
poly(ethylene terephthalate, PET	(C <sub>10</sub> H <sub>8</sub> O <sub>4</sub> ) <sub>n</sub>	Y	Y
polyether ether ketone	(OC <sub>6</sub> H <sub>4</sub> OC <sub>6</sub> H <sub>4</sub> COC <sub>6</sub> H <sub>4</sub> ) <sub>n</sub>	N	N



# Phase ID – Allegra Shell

Allegra® (fexofenadine HCl) is an antihistamine used for the treatment of hay fever symptoms. XRD analysis of the shell covering reveals the major components include polymers cellulose1 $\beta$  and amorphous cellulose along with mannitol (2 forms), and TiO<sub>2</sub>-anatase. Residual fexofenadine HCl along with additional minor components were also identified.

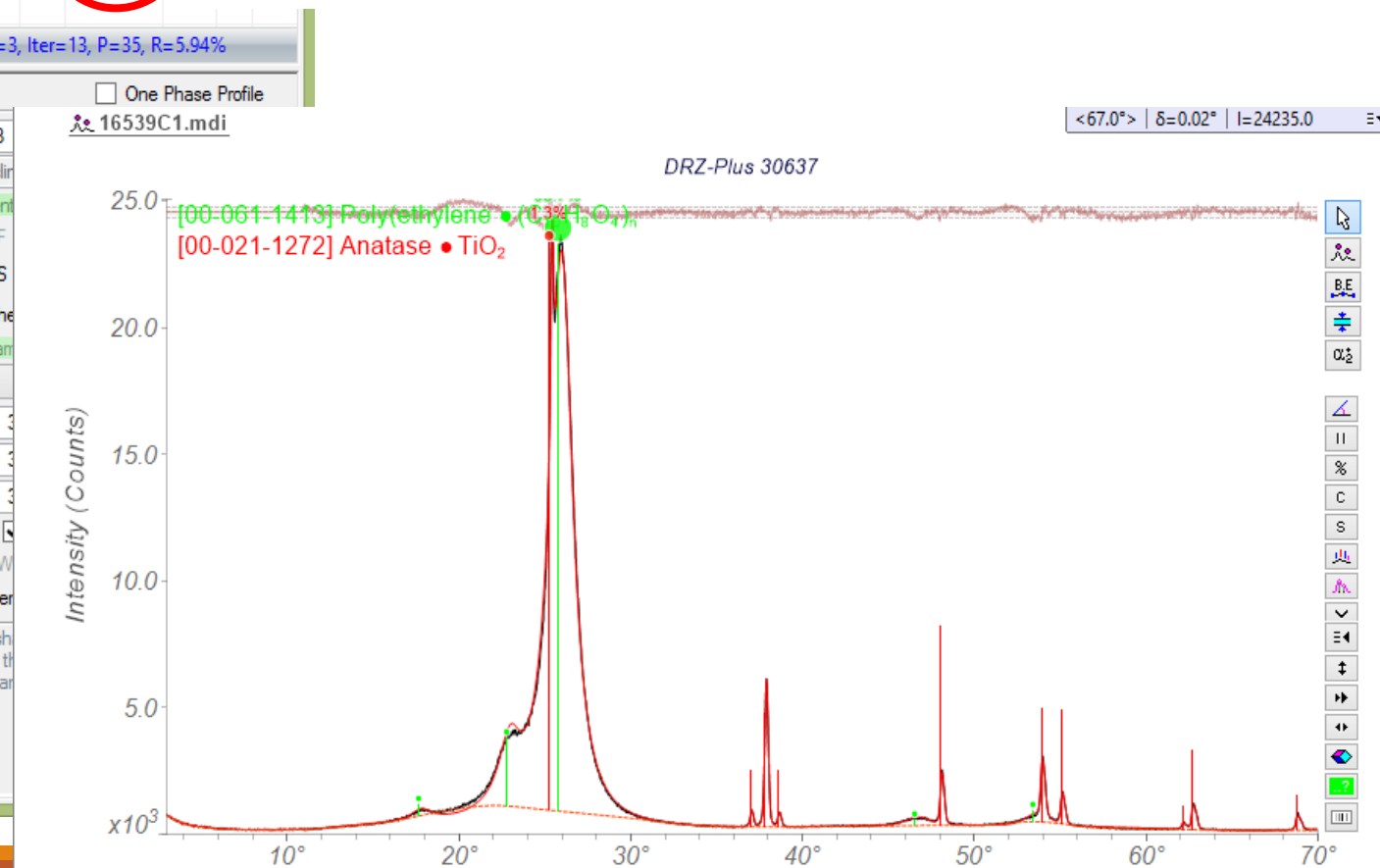
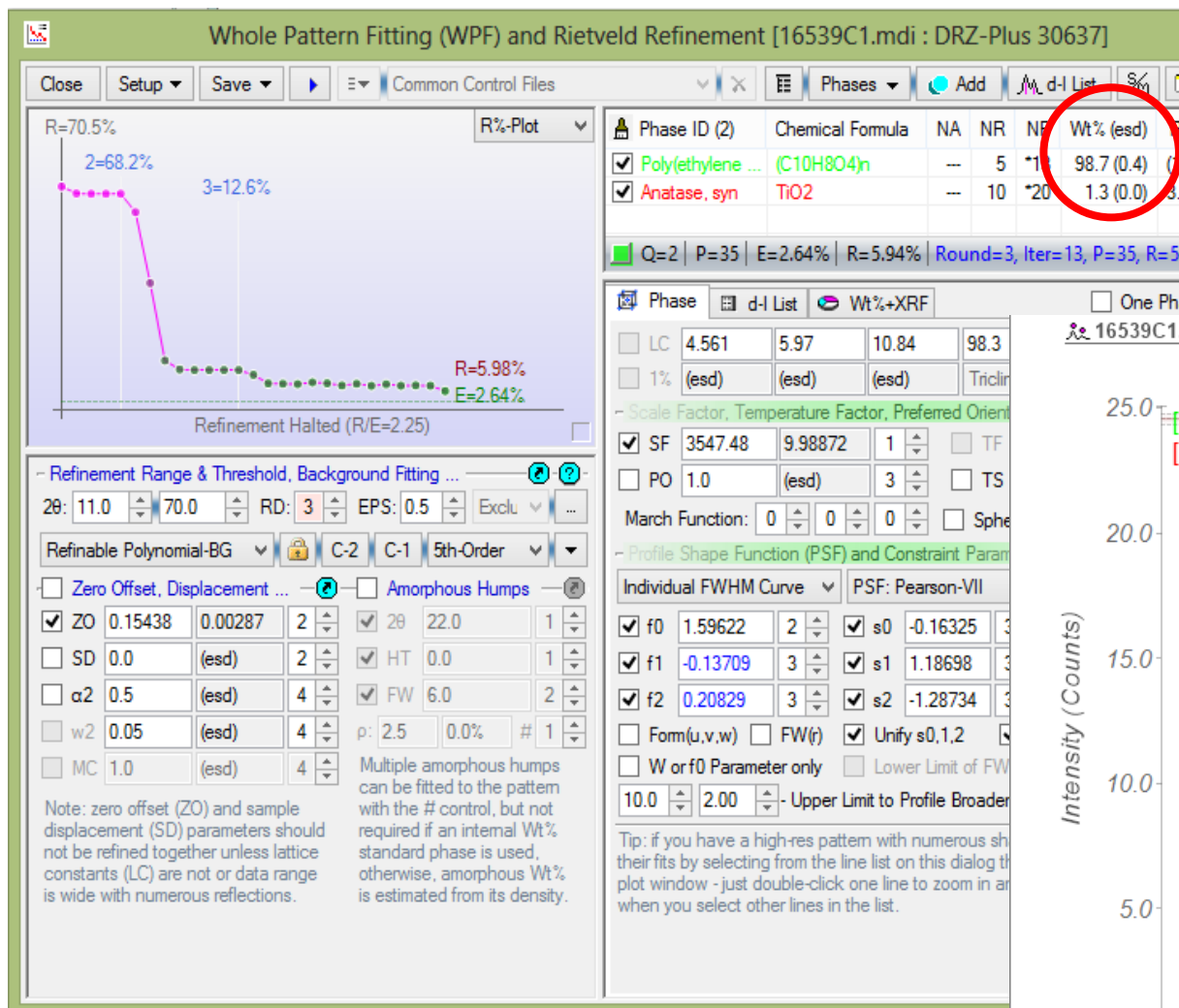


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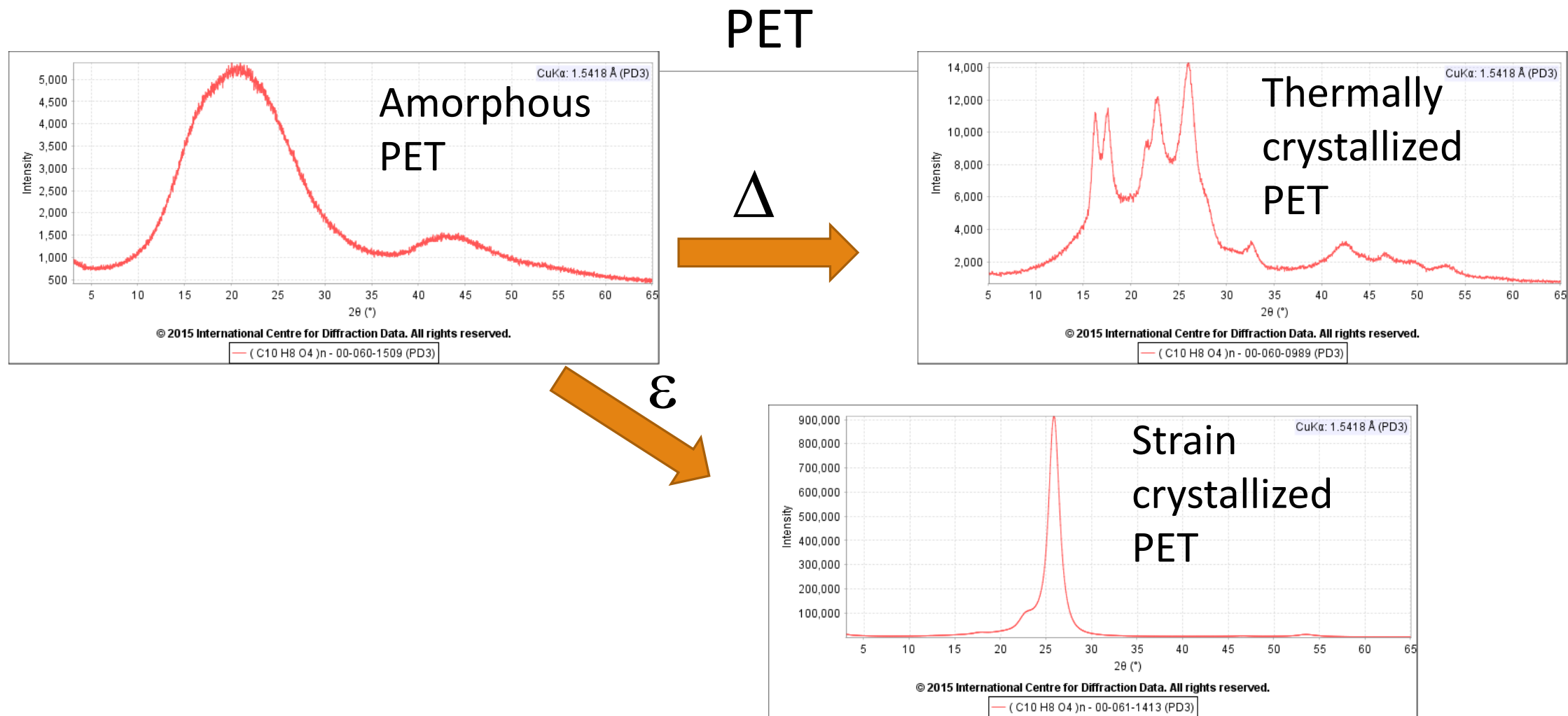
- allegra shell.dat (User Experimental Pattern) — C6 H14 O6 - 00-047-2052 (Exp-based, Intensity: 15.0%)
- C6 H14 O6 - 02-062-0119 (Calc, Intensity: 15.0%) — ( C6 H10 O5 )n - 00-060-1501 (PD3, Intensity: 50.0%)
- Ti O2 - 00-021-1272 (Exp-based, Intensity: 45.0%) — ( C6 H10 O5 )n - 00-060-1502 (Exp-based, Intensity: 50.0%)
- C32 H39 N O4 · H Cl - 00-058-1149 (Exp-based, Intensity: 10.0%)

# Quantitative Phase Analysis PET Composite Film

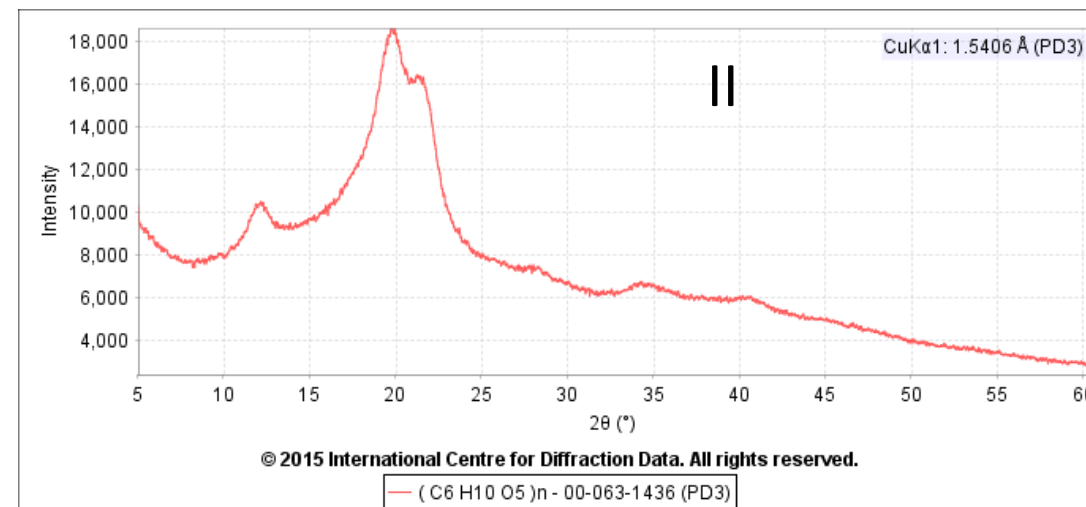
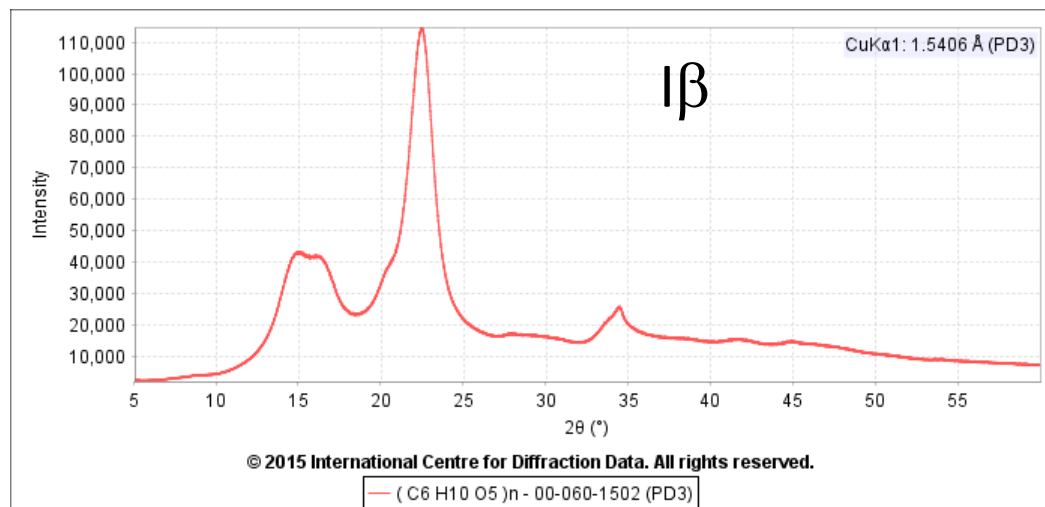
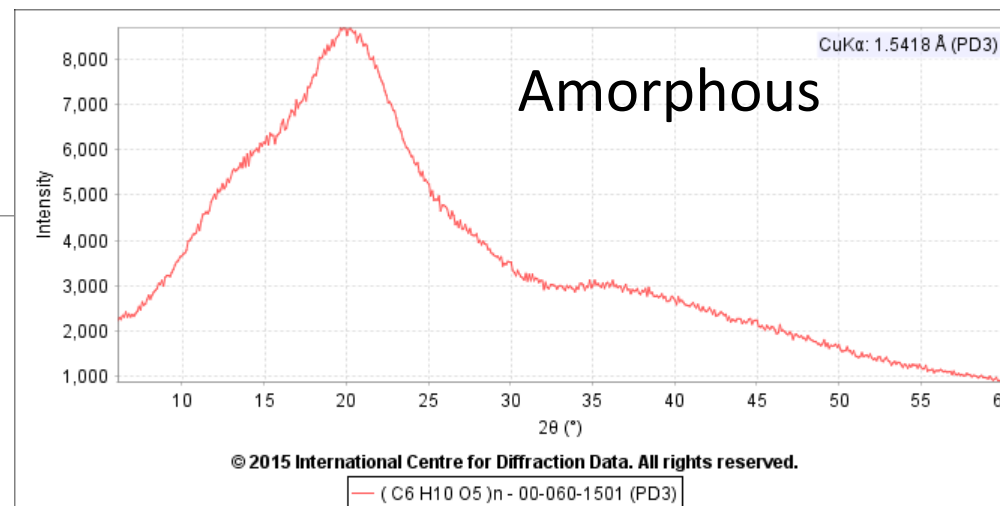
Composite used for transdermal patch backing. XRD phase analysis identified biaxially oriented poly(ethylene terephthalate) (PDF 00-061-1413) and  $\text{TiO}_2$ -anatase (00-021-1272). Since the biaxially oriented PET raw data pattern is stored in the PDF, whole pattern fitting can be used for quantitative analysis. WPF finds 1.3wt%  $\text{TiO}_2$  present, in line with the formulation calling for 1.5wt%  $\text{TiO}_2$ .



# Do you need multiple Entries in the PDF Database for a polymer?

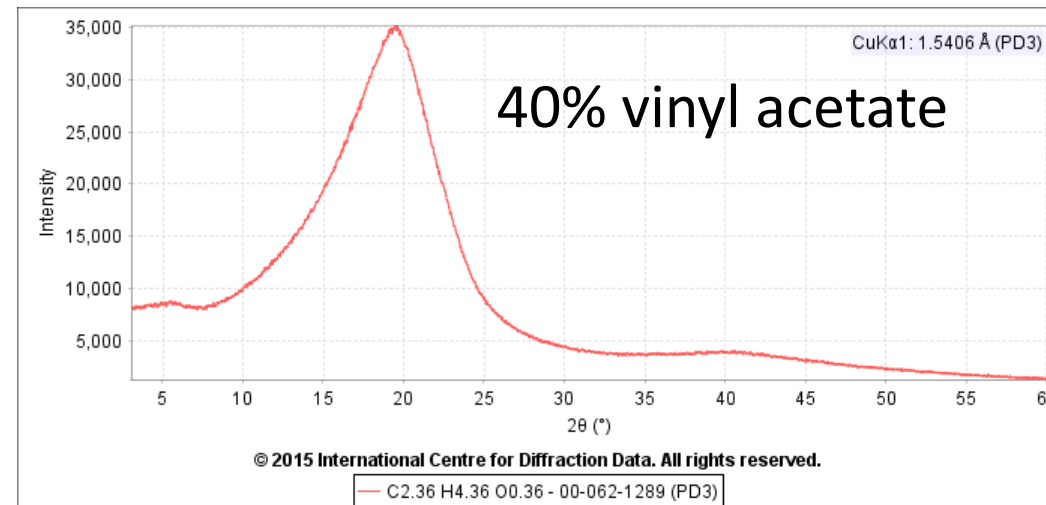
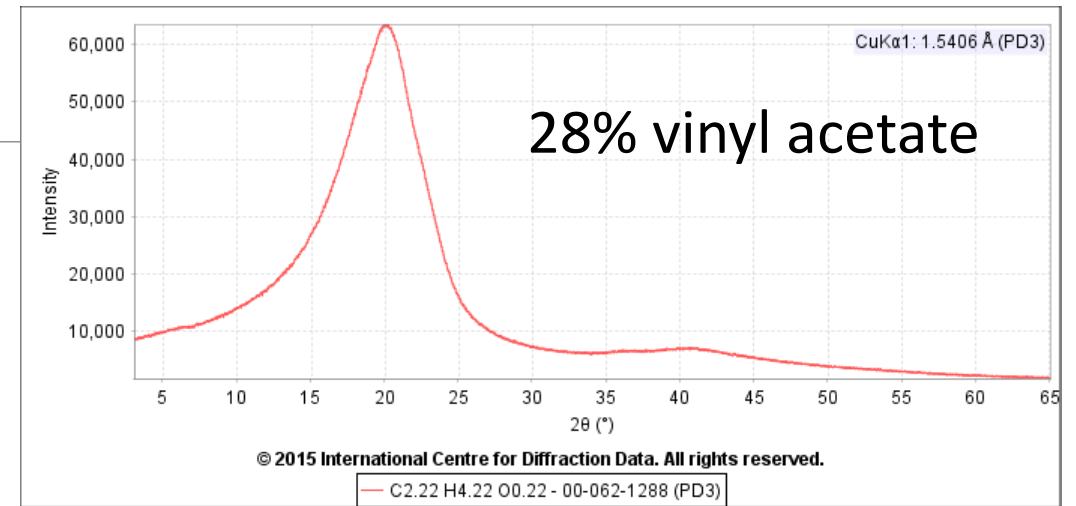
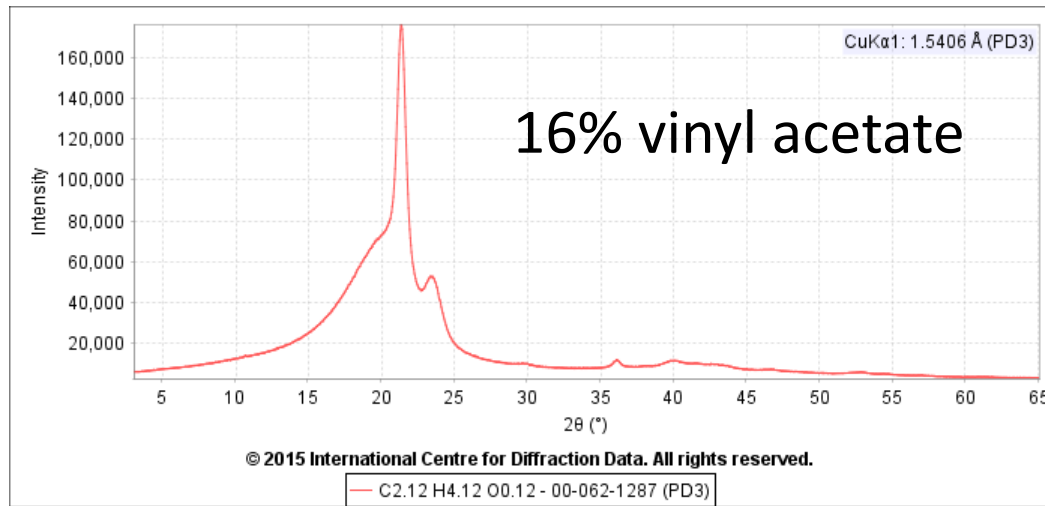


# Cellulose

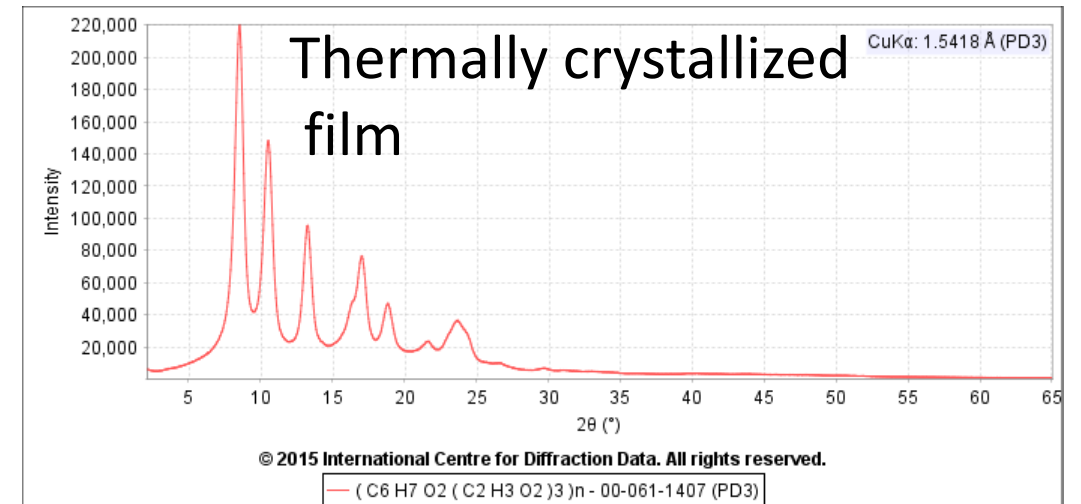
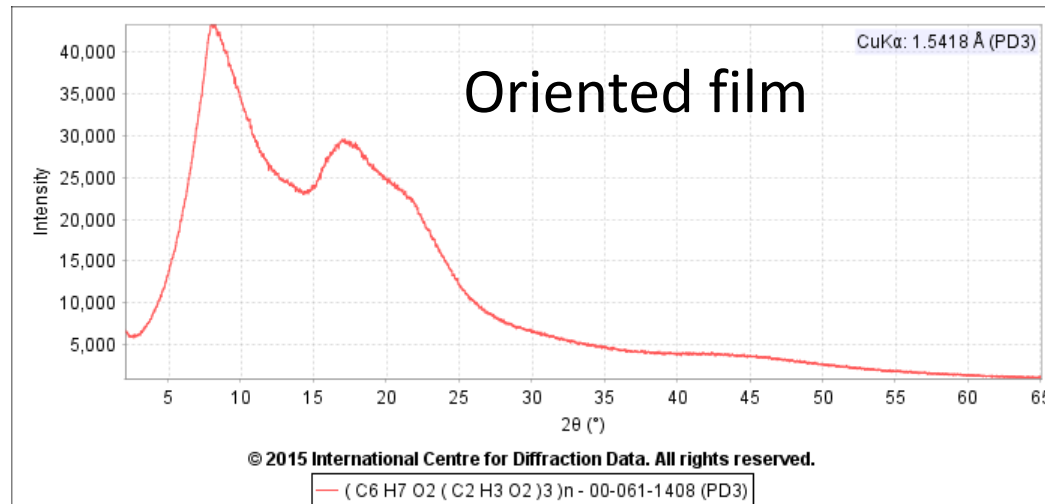
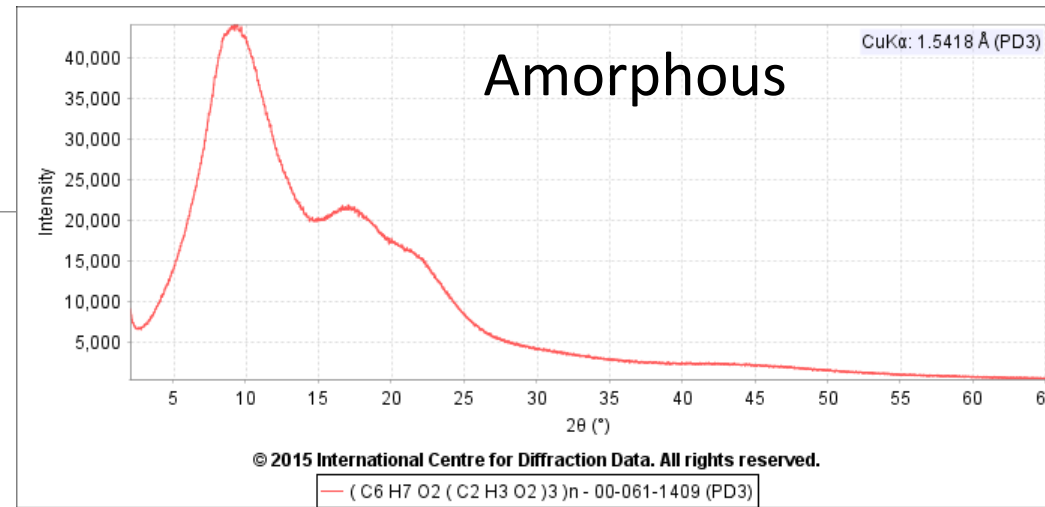


Kaduk and Blanton, Powd. Diffr., 28(3), 2013. pp194-199.

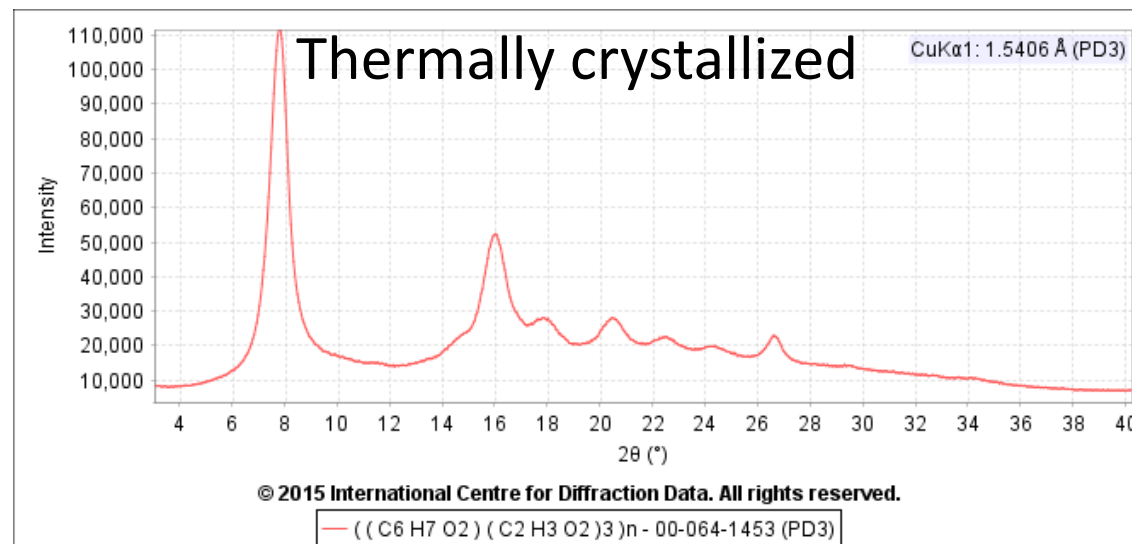
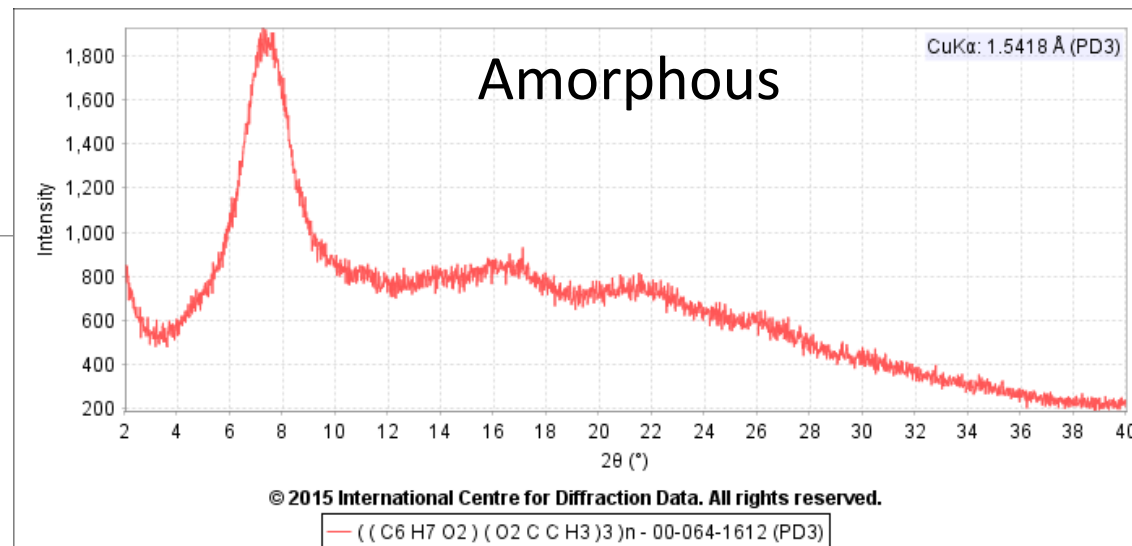
# Ethylene vinyl acetate



# Cellulose triacetate II

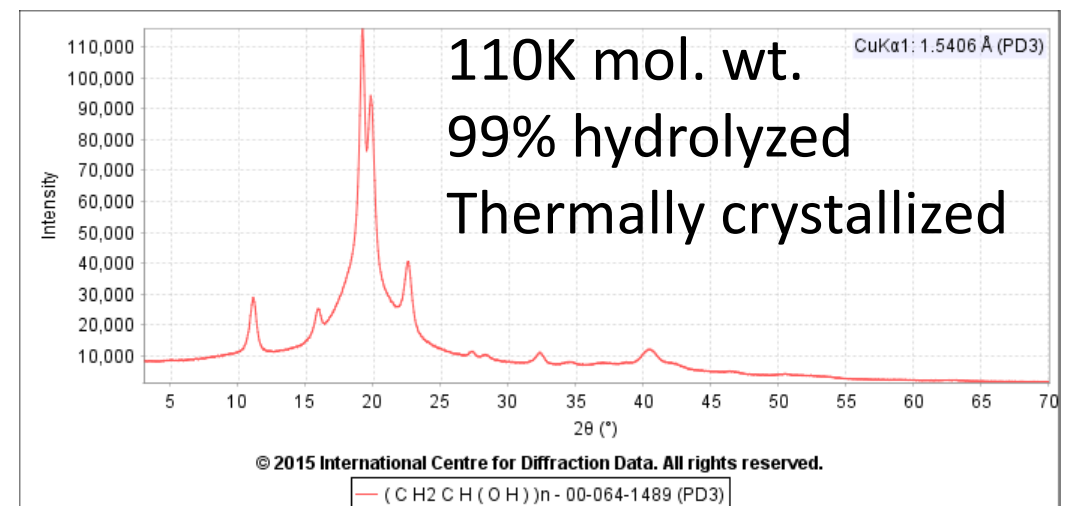
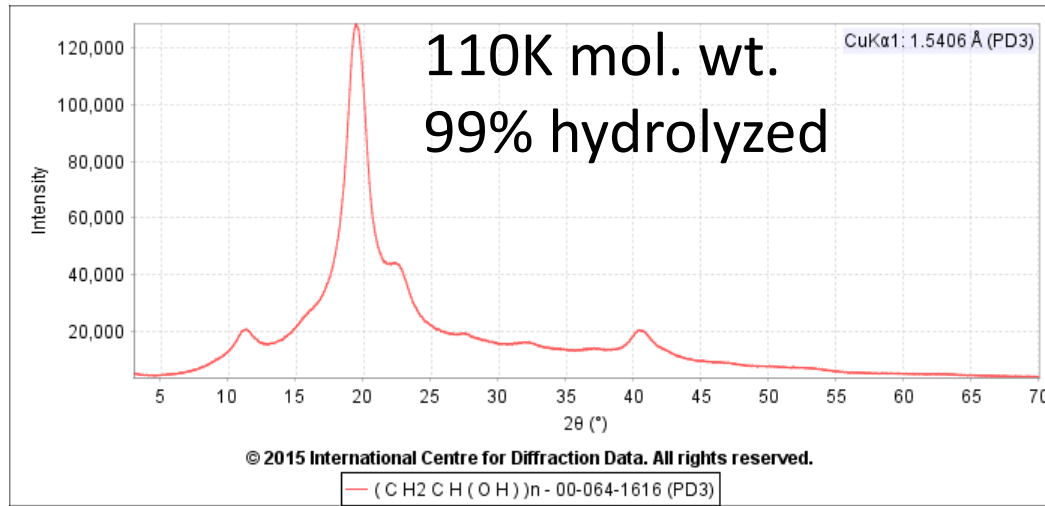
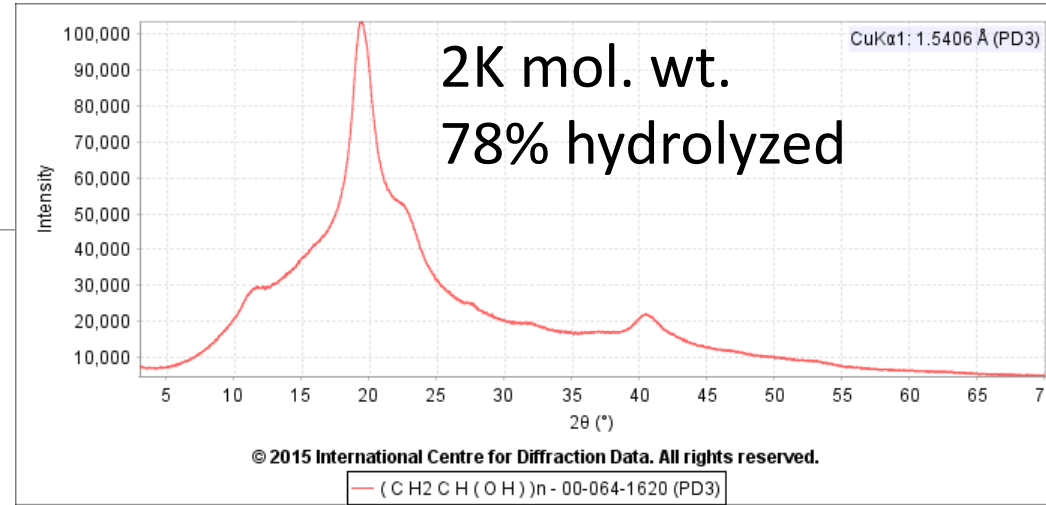


# Cellulose triacetate I



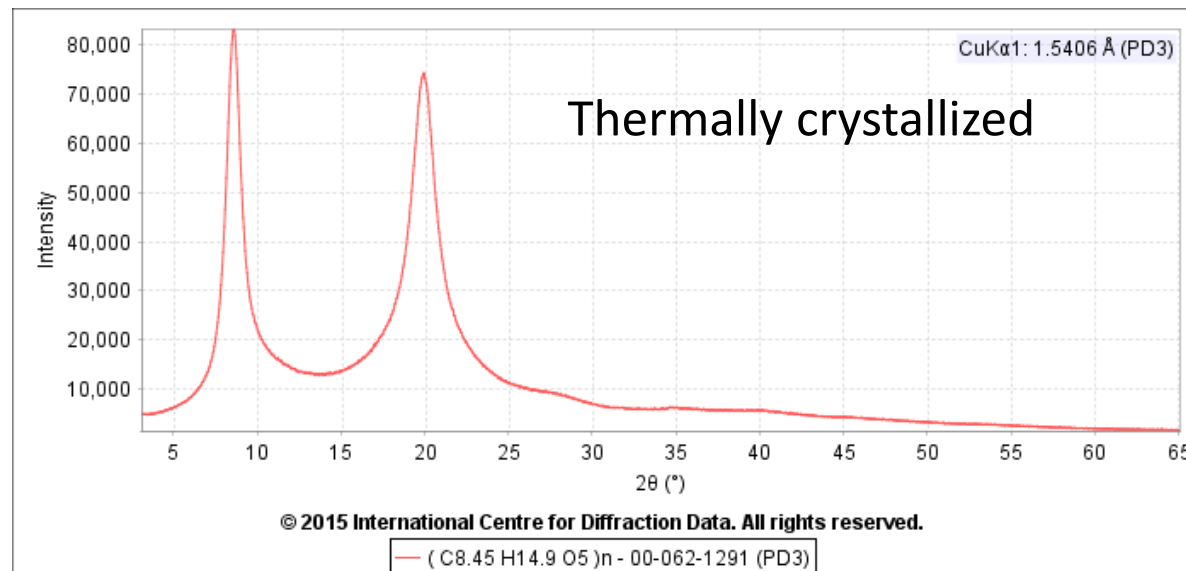
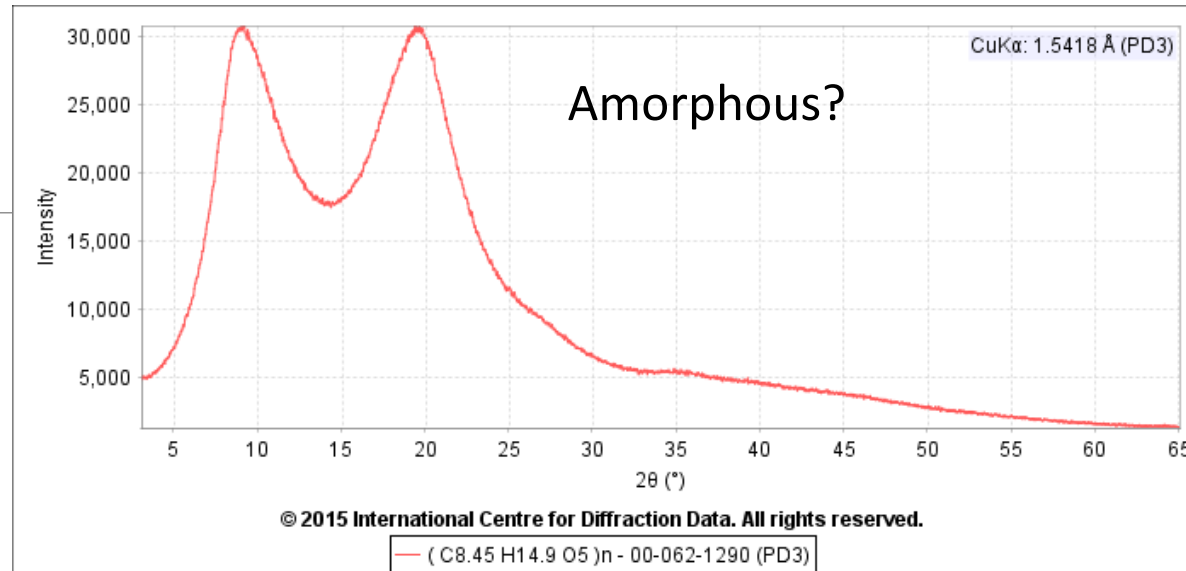
Kaduk, Gates, Blanton  
PDF Entry 00-064-1453, 2014.

# Polyvinyl alcohol





# Methyl cellulose



# Summary

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- Raw data diffraction patterns generated from analysis of polymer samples are being added to ICDD PDF-4 databases
- Important considerations:
  - Polymer chemistry
  - Polymer processing
  - Sample orientation in diffractometer - reflection vs. transmission
  - Sample type (powder, film, fiber)
- Use caution when using a single peak amorphous pattern to define the amorphous phase
- Pay attention to sample prep

# 2015 PDF Data Entry

