

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



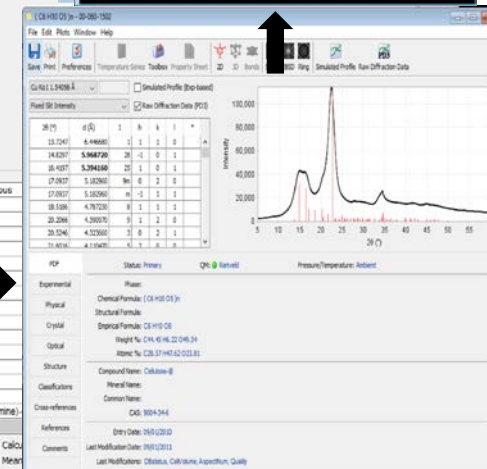
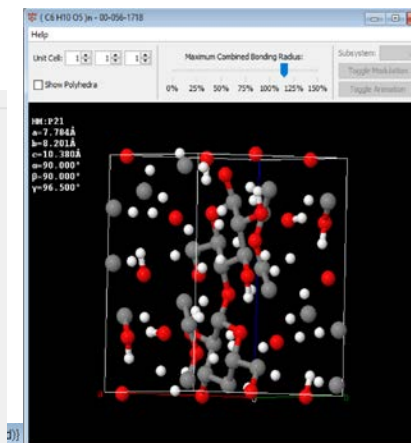
PDF-4/Organics 2016

Subfile/Subclass

- Not
- Ionic Conductors
- Merck
- Metals & Alloys
- Micro & Mesoporous
- Mineral Related
- Modulated Structure
- NBS
- Nucleosides & Nucleotides
- Organics
- Pharmaceutical
- Pigment
- Polymer
- Porphyrins, Corrins & Complexes
- Steroids
- Superconducting Material
- Terpenes
- Thermoelectric Material

And  
Or

00-082-1714	G	C118 H116 O64	Cellulose acetate phthalate, amorphous
00-083-1436	R	(C6 H10 O5)n	Cellulose II
00-083-1601	G	H (C6 H10 O6)n O H	Dextran-4, amorphous
00-083-1602	G	(C6 H10 O6)n	Dextran-250, amorphous
00-083-1603	G	(C6 H8 N O)n	Polystyrene, amorphous
00-083-1604	G	(C6 H8 N O)n (C4 H8 O2)n	Copolystyrene, amorphous
00-083-1806	G	(C6 H8 N O)n	Copolystyrene, amorphous
00-081-0661	B	C12 H22 O11	beta-Cellobiose
00-081-0662	I	C12 H22 O11	beta-Cellobiose
00-081-0663	S	C12 H22 O11	beta-Cellobiose
00-070-0904	B	(C12 H22 O)n	poly (poly methylene)
00-070-2866	B	(C8 H13 N O5)n	Poly (N-acetyl-D-glucosamine)
00-070-2067	B	(C16 H32)n	Polyisobutylene
00-074-7680	I	(C16 H26 N2 O10)n	Poly (N-acetyl-D-glucosamine)
00-085-2907	S	C8 H14 N2 O2	2-(2-Oxopropion-1-yl)butylamine
00-088-6911	I	C20 H20 Cu2 N6 O2 2 (C F3 O3 S)	bis(mu 2 Hydroxo) bis(di 2 pyridyl)amine



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S. MISTURE, ALFRED UNIVERSITY

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

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

# 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 or silicone based polymers

# Polymer analysis using XRD

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Many techniques are used for polymer characterization

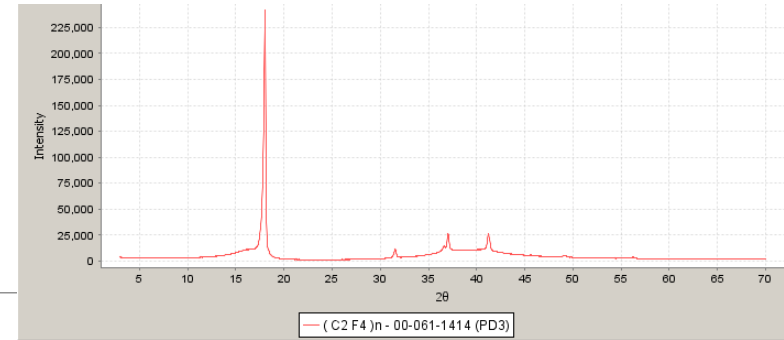
- IR, NMR, GC/MS, etc.

Why use XRD?

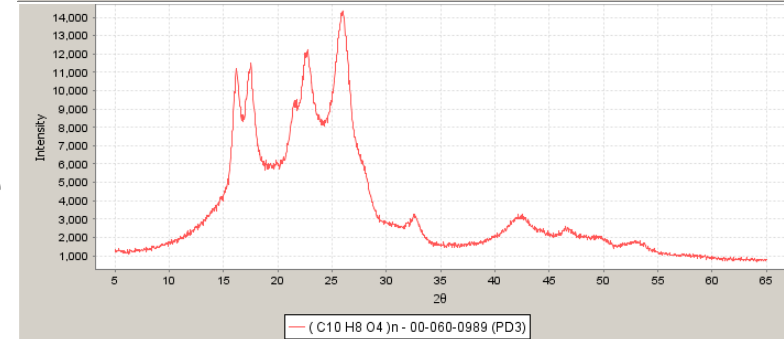
- Polymers can have amorphous and crystalline regions
- Crystallite size is small and measurable by XRD
- Polymers are often used as the base component in pharmaceutical and biomedical composite materials

# Polymer order:

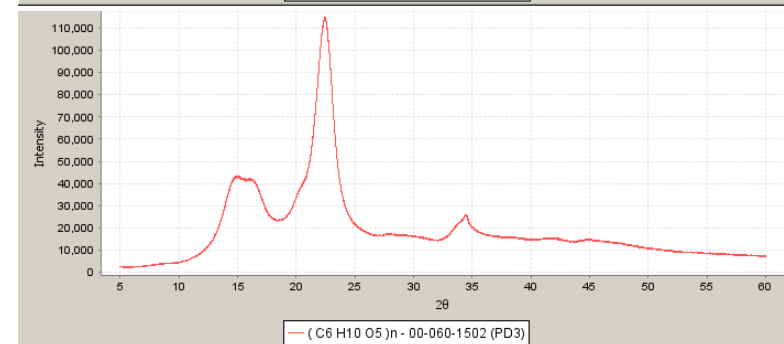
Highly crystalline - Teflon



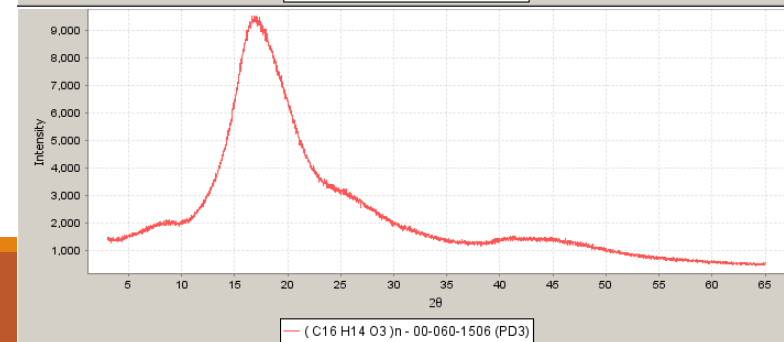
Semi crystalline – polyethylene terephthalate



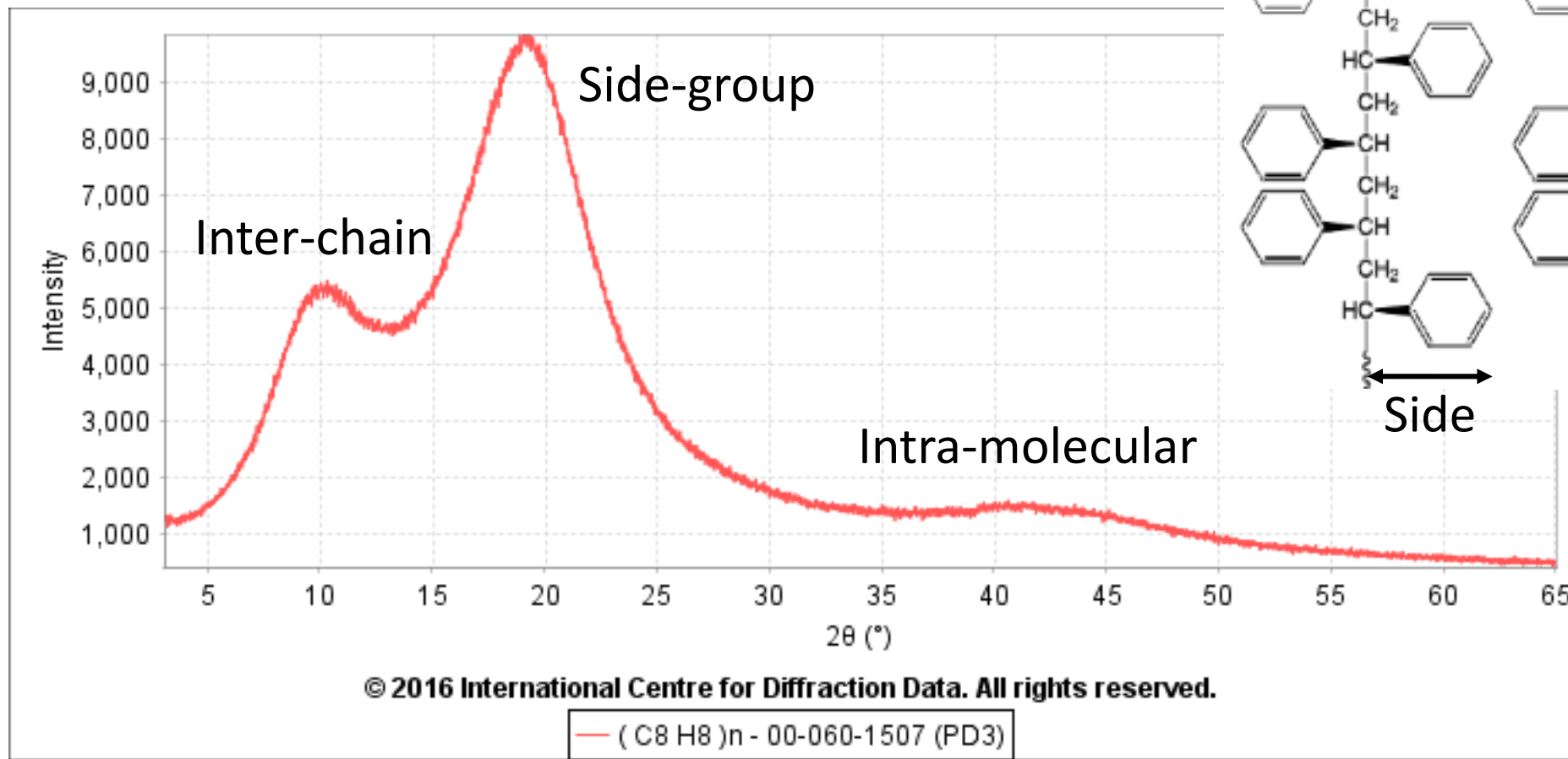
Micro crystalline – cellulose 1β



Amorphous - polycarbonate



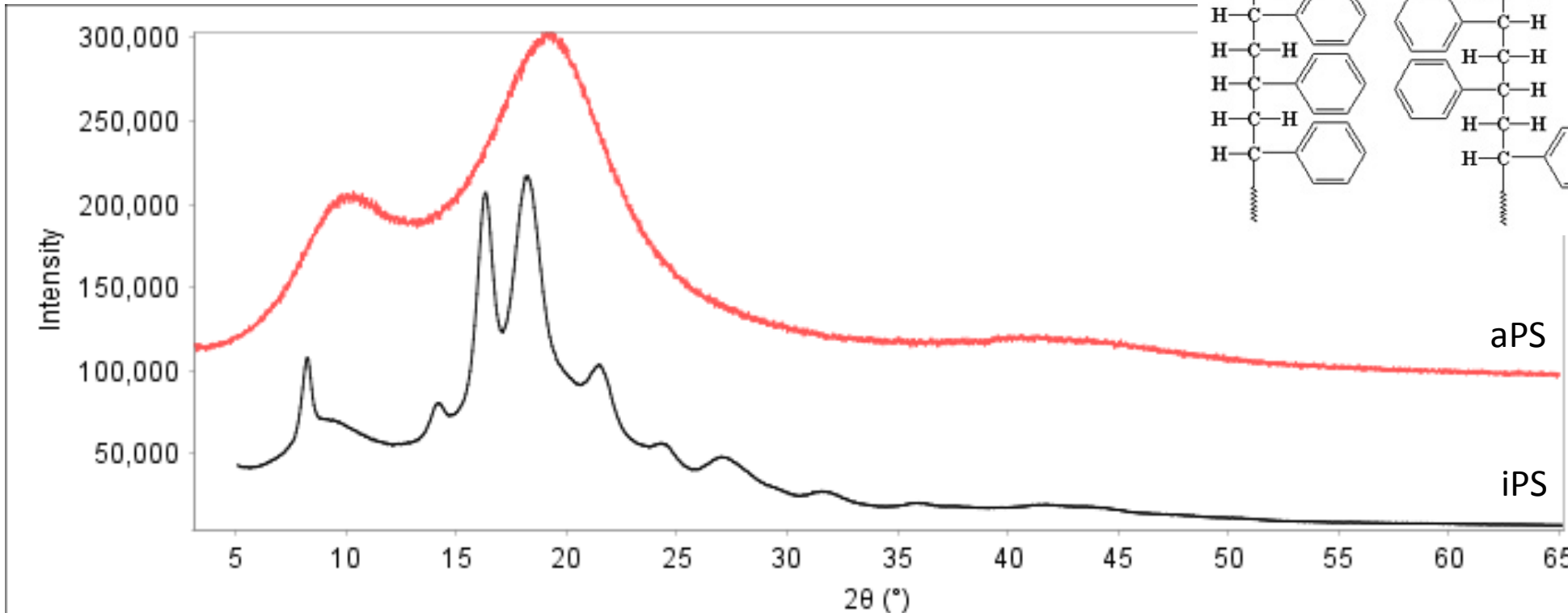
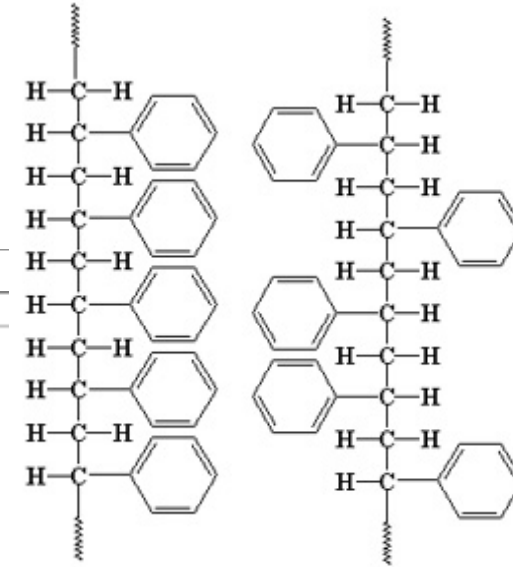
# XRD pattern for atactic polystyrene – effect of the polymer chain structure



# Effect of tacticity on polymer order

Isotactic PS

Atactic PS



aPS

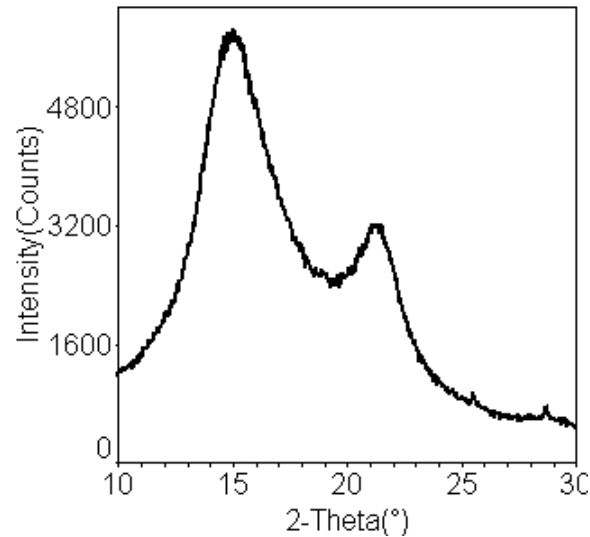
iPS

Rhombohedral R-3c  
 $a=22.049\text{\AA}$   
 $c=6.723\text{\AA}$

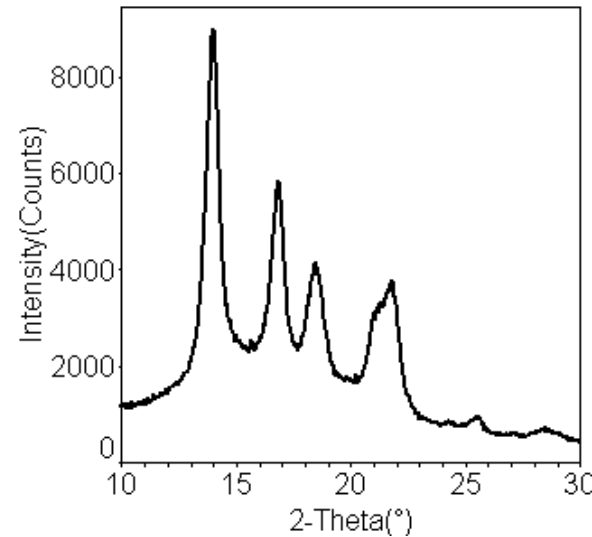
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—  $*(C_8H_8)_n$  - 00-060-1507 (PD3) —  $((C_6H_5)CHCH_2)_n$  - 00-066-1667 (PD3)

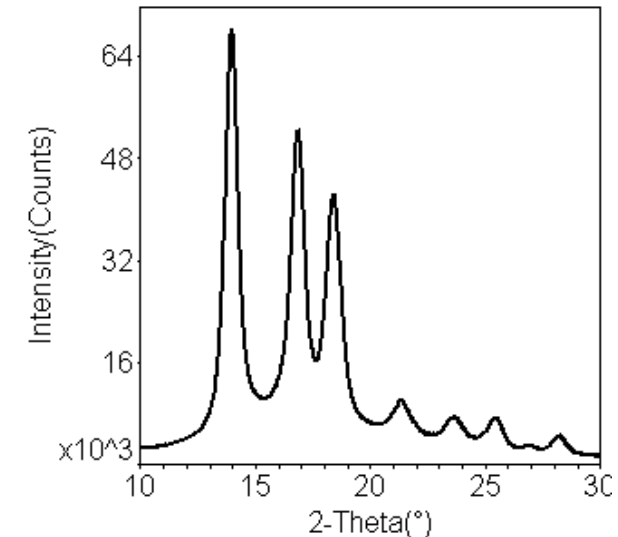
# Bragg Brentano Reflection Mode XRD Patterns - Effect of Processing on Polypropylene



Cast  
No heatset  
No stretch



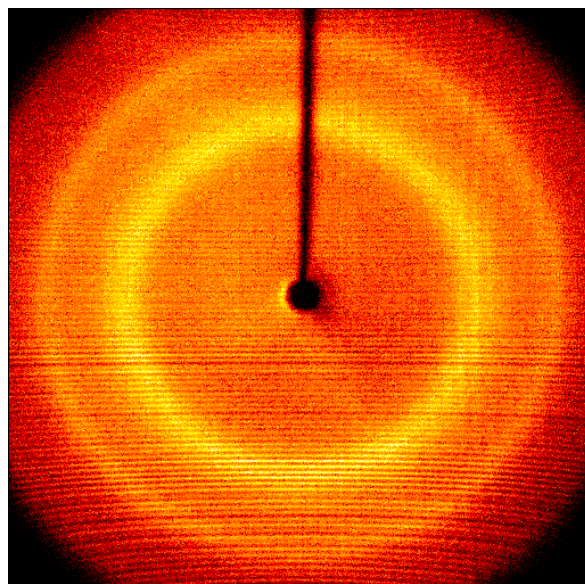
Heatset  
120 °C, 10s  
No stretch



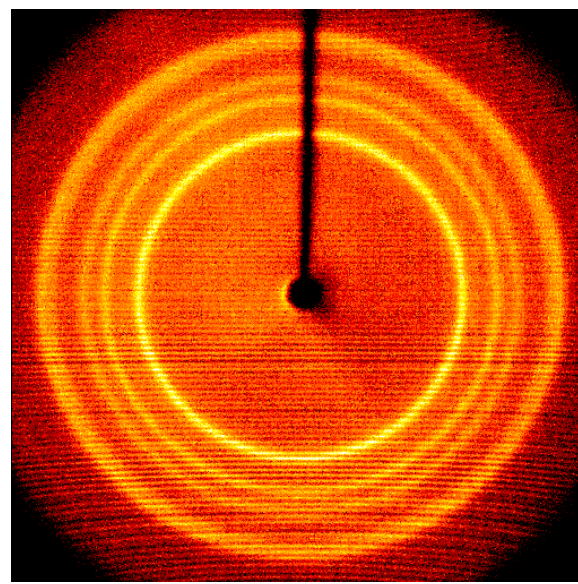
Heatset  
6X  
Uniax stretch



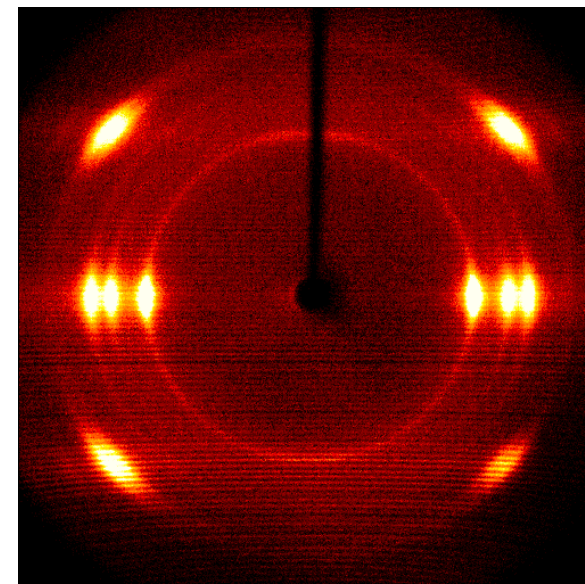
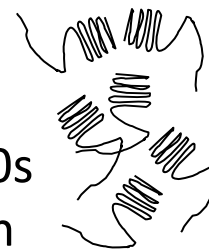
# 2-D transmission XRD patterns - Effect of processing and sample orientation on polypropylene XRD patterns



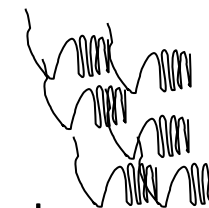
Cast  
No heatset  
No stretch



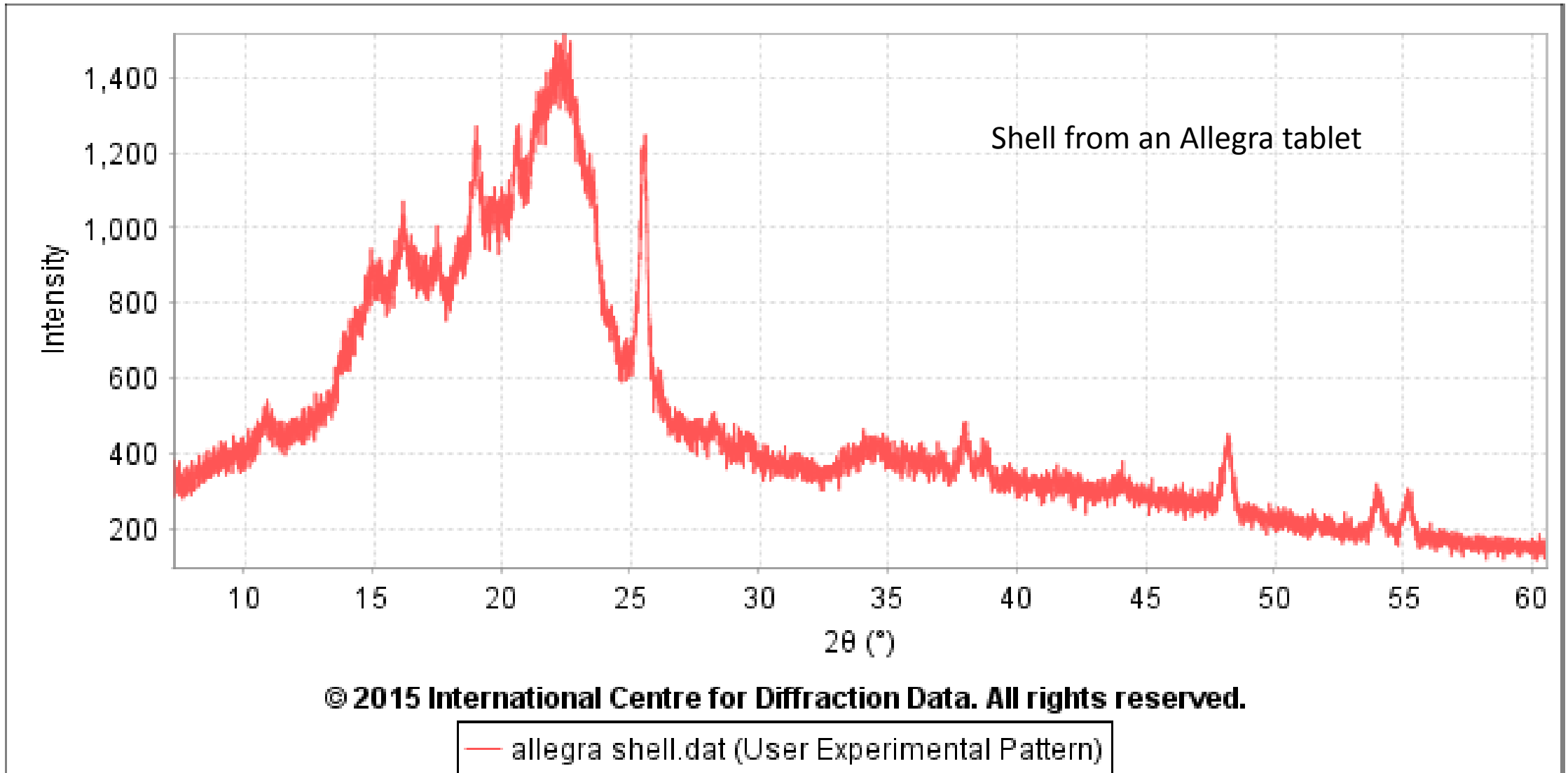
Heatset  
120 °C, 10s  
No stretch



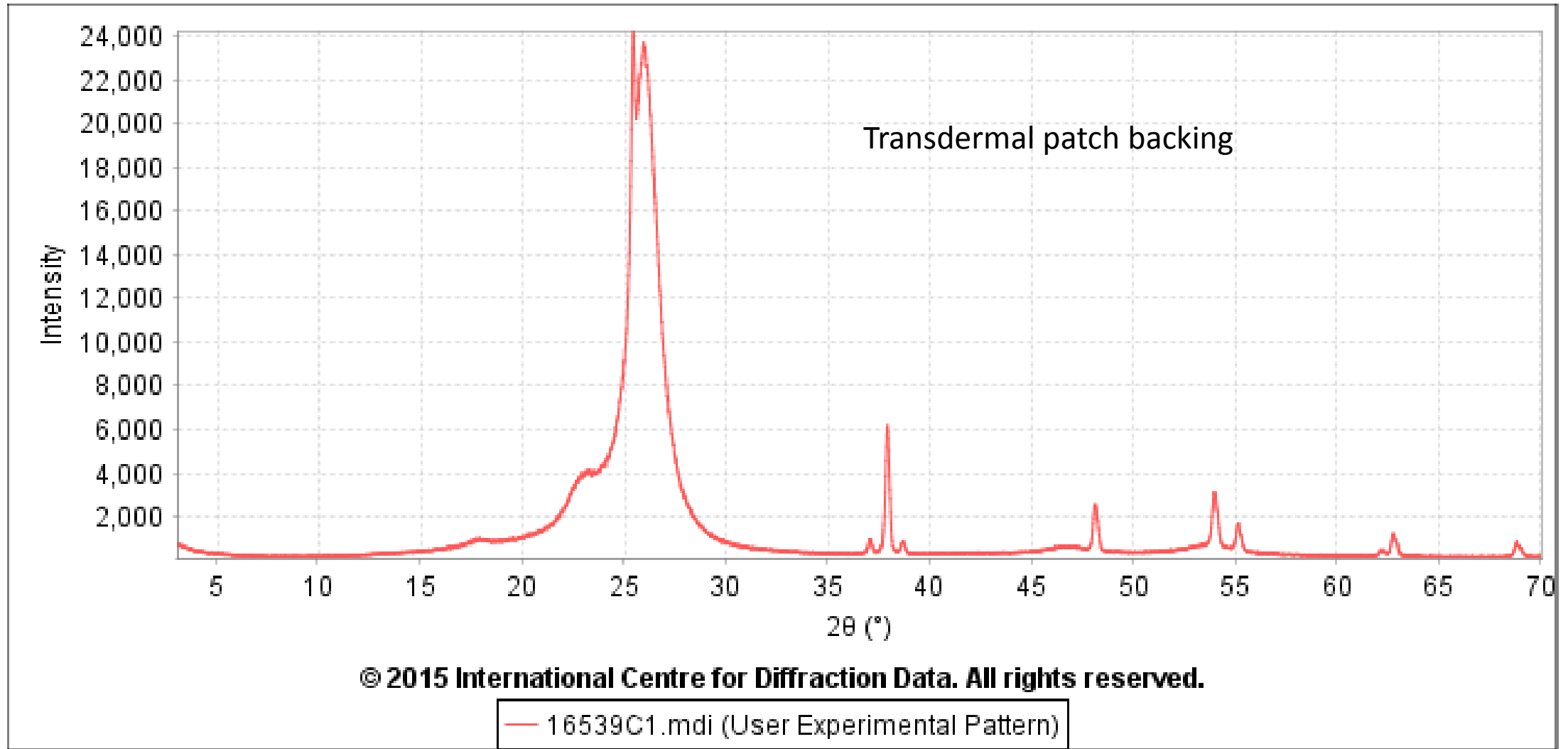
Heatset  
6X  
Uniax stretch



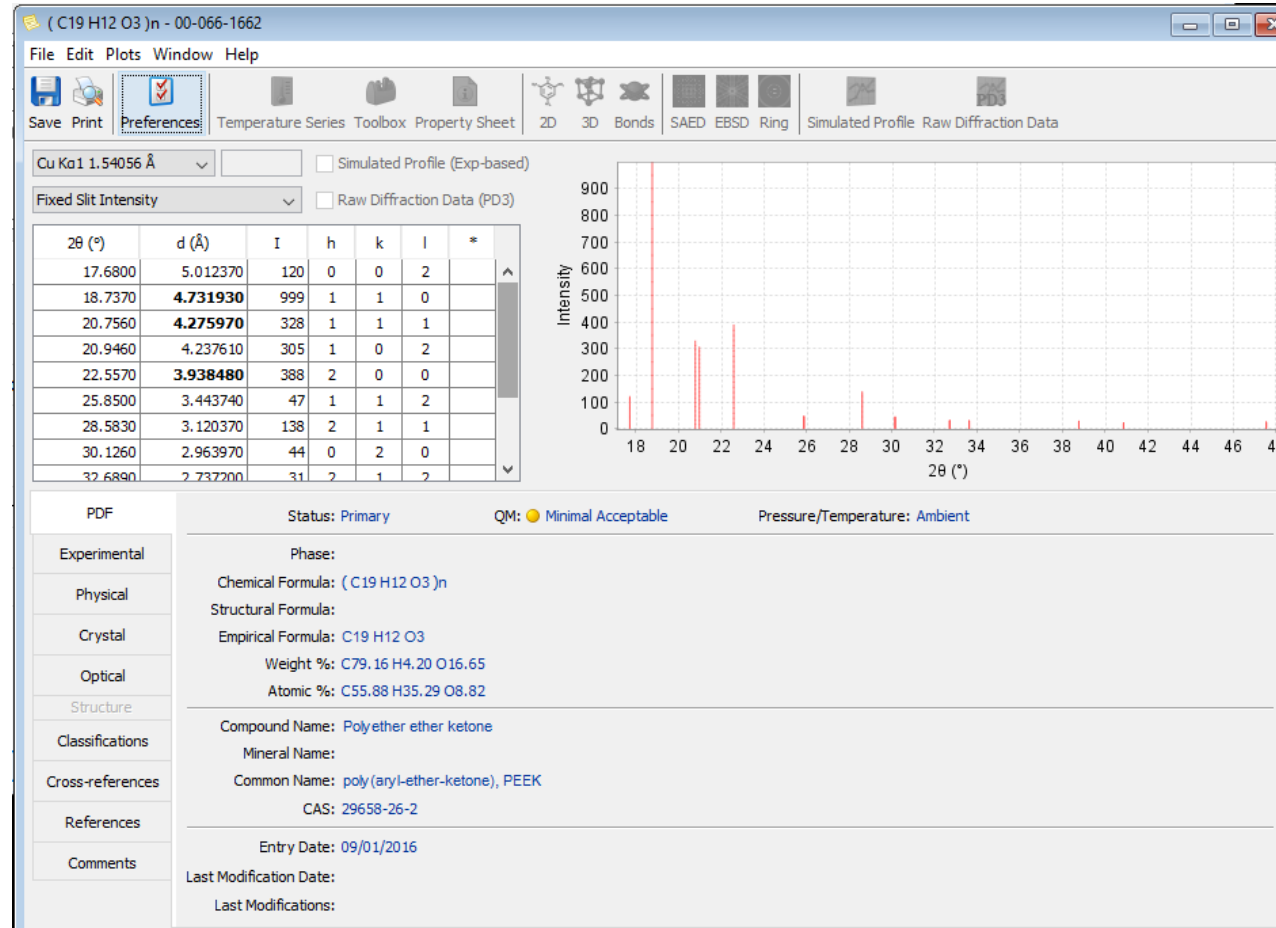
# Pharmaceuticals



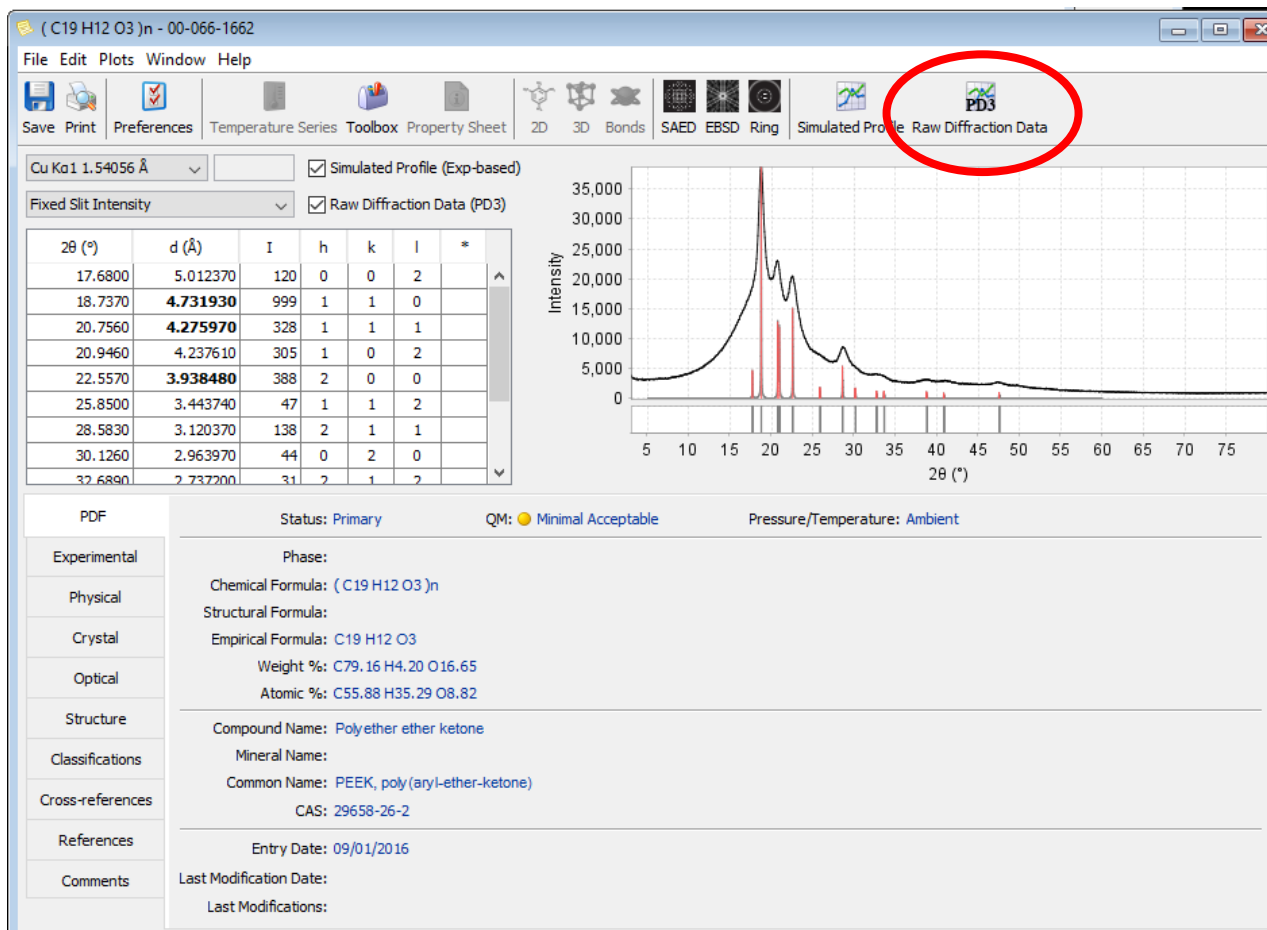
# Biomedical devices



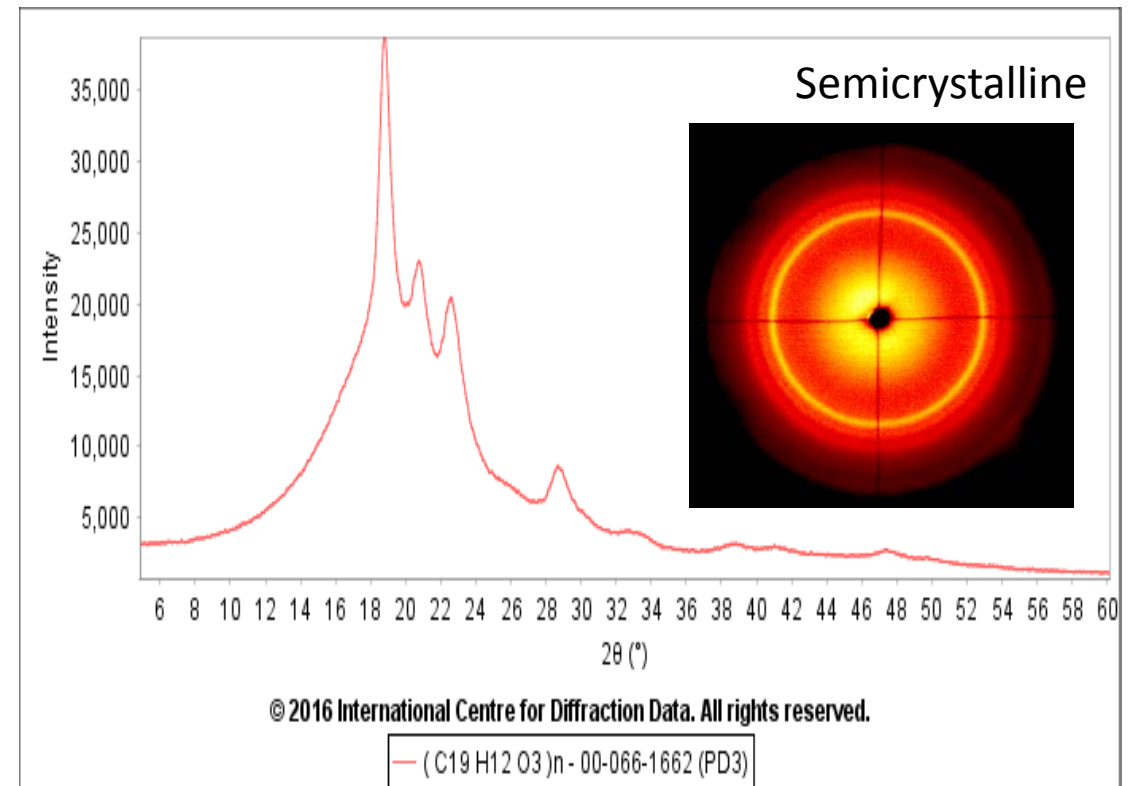
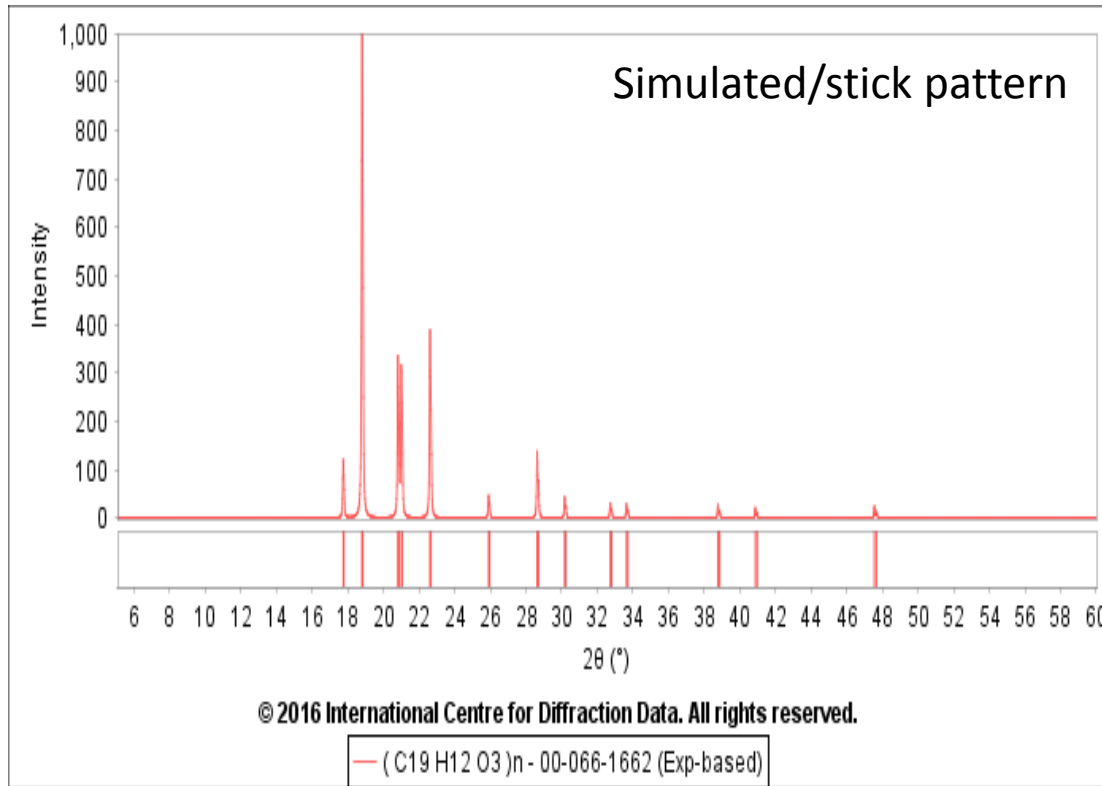
# Powder Diffraction File PDF Polymer Entry – PEEK Polymer



# Powder Diffraction File PDF Polymer Entry – PEEK Polymer



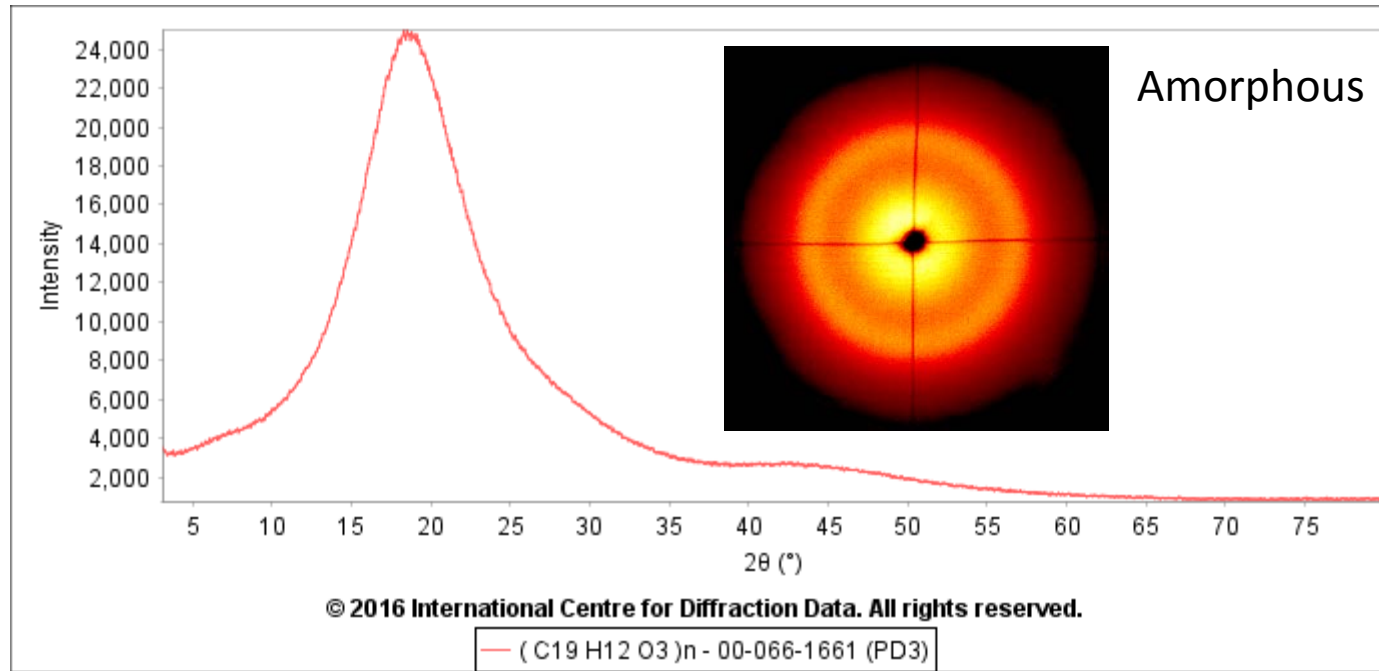
# PD3 Raw Data Patterns in the Powder Diffraction File – PEEK Polymer



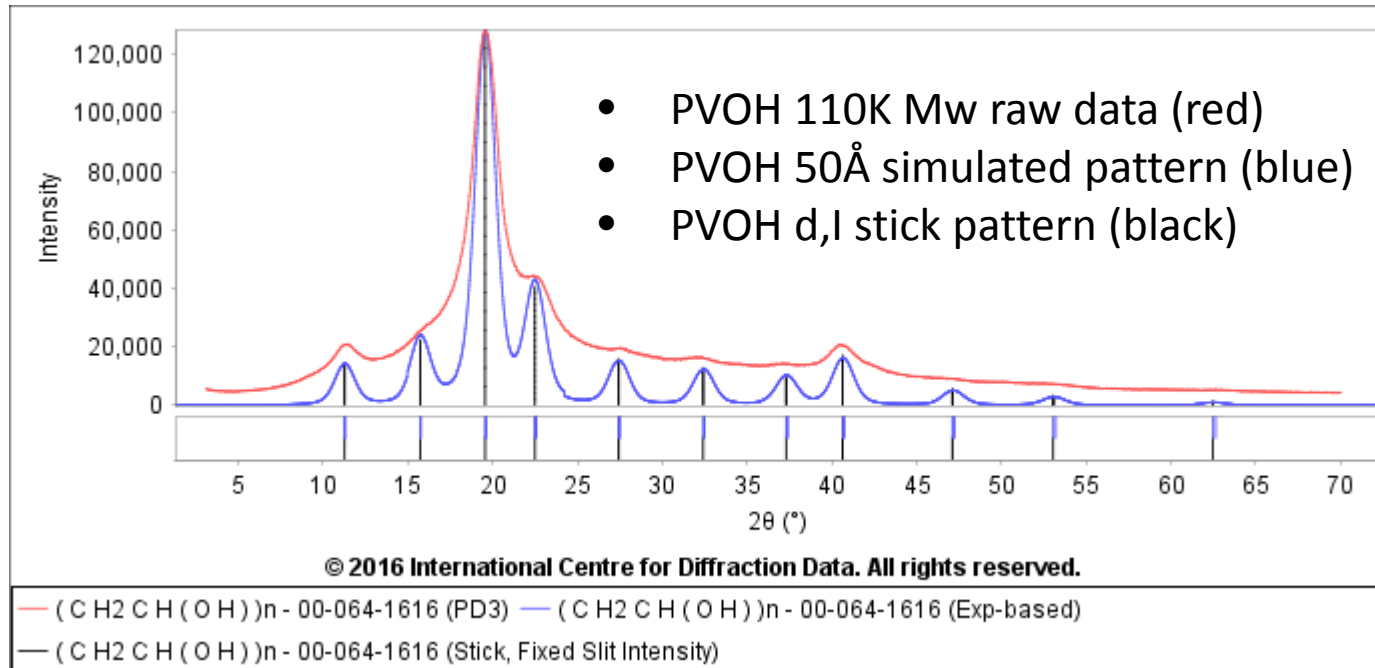
Orthorhombic Pbcn  
 $a=7.831\text{Å}$   $b=5.891\text{Å}$   $c=9.958\text{Å}$

# PD3 Raw Data Patterns in the Powder Diffraction File – PEEK Polymer

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# Stick vs. Simulated vs. PD3 (PVOH – tablet packaging)





# 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	● ○	( C4 H5 Cl )x	Polychloroprene	4.450000	4.070000	3.360000	○
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	● ○	( C H2 )x	Paraffin wax	4.150000	3.730000	3.880000	X
00-003-0259	● ○	( C H2 )x	Paraffin wax	4.130000	3.730000	3.480000	X
00-003-0289	● ○	( C6 H12 O6 )x	Native cellulose	3.890000	5.940000	2.570000	M
00-004-0419	● ○	( C H2 O )x	Paraformaldehyde	3.760000	2.590000	1.870000	X
00-007-0506	● ○	C30 H57 N5 O6	ε-Polyaminocaproic acid	3.690000	4.470000	3.600000	X
00-007-0511	● ○	C24 H46 N4 O5	ε-Polyaminocaproic acid	4.430000	3.690000	4.470000	X
00-008-0689	● ○	C57 H108 O6	β-2-Oleyl-1,3-distearyltriglycerol	4.600000	5.420000	5.030000	M
00-009-0853	● ○	( C4 H6 )n	1,4-cis-Polybutadiene	3.980000	4.750000	4.070000	X
00-011-0834	● ○	( C2 H4 )n	β-Polyethylene	4.100000	3.600000	2.490000	X
00-012-0876	● ○	( C2 H3 Cl O )n	Polymonochloroacetaldehyde	7.760000	3.850000	3.450000	X
00-012-0877	● ○	( C4 H8 O )n	Butyraldehyde polymer	12.000000	9.000000	4.800000	X
00-012-0878	● ○	( C4 H8 O )n	Polyisobutyraldehyde	9.300000	7.900000	4.600000	X
00-012-0879	● ○	( C2 H2 Cl2 O )n	Polydichloroacetaldehyde	8.320000	4.000000	3.130000	X
00-012-0880	● ○	( 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	○
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	Metlaldehyde	7.500000	3.880000	3.350000	T
00-013-0743	● ○	C5 H9 N O2	Poly-L-proline	5.800000	4.900000	3.650000	X
00-013-0744	● ○	( 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:

# PD3 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 PD3 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	( C3 H6 )n	α-Polypropylene	6.244550	5.226510	4.189310	M
00-062-0923	R	C45 H86 O6	β-1,2,3-tris Tetradecanoyl glycerol	4.589920	4.549930	3.684620	A
00-062-0924	R	C57 H110 O6	β-1,2,3-Trioctadecanoyl-glycerol	4.582690	4.556430	3.832850	A
00-062-1286	R	( C6 H10 O2 )n	Poly-ε-caprolactone	4.140580	3.735750	4.026870	O
00-062-1287	R	C2.12 H4.12 O0.12	Ethylene vinyl acetate	4.132970	3.747230	2.477160	O
00-062-1288	M	C2.22 H4.22 O0.22	Ethylene vinyl acetate	4.449160	2.217140	14.018100	X
00-062-1289	M	C2.36 H4.36 O0.36	Ethylene vinyl acetate	4.564780	16.413000	2.229220	X
00-062-1290	M	( C8.45 H14.9 O5 )n	Methyl cellulose, amorphous	9.814620	4.536000	3.364680	X
00-062-1291	I	( C8.45 H14.9 O5 )n	Methyl cellulose	10.369800	4.464680	4.107080	O
00-062-1292	B	( C22 H10 N2 O5 )n	Kapton	6.062230	4.073580	5.674000	O
00-062-1293	B	( C22 H10 N2 O5 )n	Kapton	16.322000	6.074650	5.443550	O
00-062-1701	G	( C6 H7 O2 ( O H )z · ( C2 H3 O2 )x...	Cellulose acetate butyrate	13.450500	4.414020		X
00-062-1702	G	( C6 H7 O2 ( O H )z · ( C2 H3 O2 )x...	Cellulose acetate butyrate	13.268800	4.506810		X
00-062-1703	M	( C6 H7 O2 ( O H )z · ( C2 H3 O2 )x...	Cellulose acetate butyrate	13.417800	4.435870	4.328790	X
00-062-1704	M	( C6 H7 O2 ( O H )z · ( C2 H3 O2 )x...	Cellulose acetate propionate	11.570400	4.185460	7.787560	X
00-062-1705	M	( C4 H8 )n	Poly(butene-1)	8.845650	4.356160	4.466800	X
00-062-1706	M	( O C H2 )n	Poly(oxyethylene)	3.930910	2.630020	1.899100	X
00-062-1707	M	( O C5 H6 Cl4 )n	Poly (3,3-bis(chloromethyl)oxetane)	5.888960	4.036160	3.997390	X
00-062-1708	M	( C4 H8 O )n	Poly(tetrahydrofuran)	4.492410	4.561160	3.688190	X
00-062-1709	M	( C H2 C H ( ( C6 H4 ) C H3 ) )n	Poly(o-vinyl toluene)	5.933620	5.760570	6.142820	X
00-062-1710	M	( C H2 C Cl2 )n	Poly(vinylidene chloride)	5.643990	13.657800	10.203600	X
00-062-1711	M	( C H2 C H ( C6 H5 ) )n ( C H2 C H ...	Poly(styrene-acrylic acid)	4.785400	8.293680	9.837960	X
00-062-1712	G	( C4 H8 O2 )n · ( C2 H4 O2 )n	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:

# 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
alginate 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	

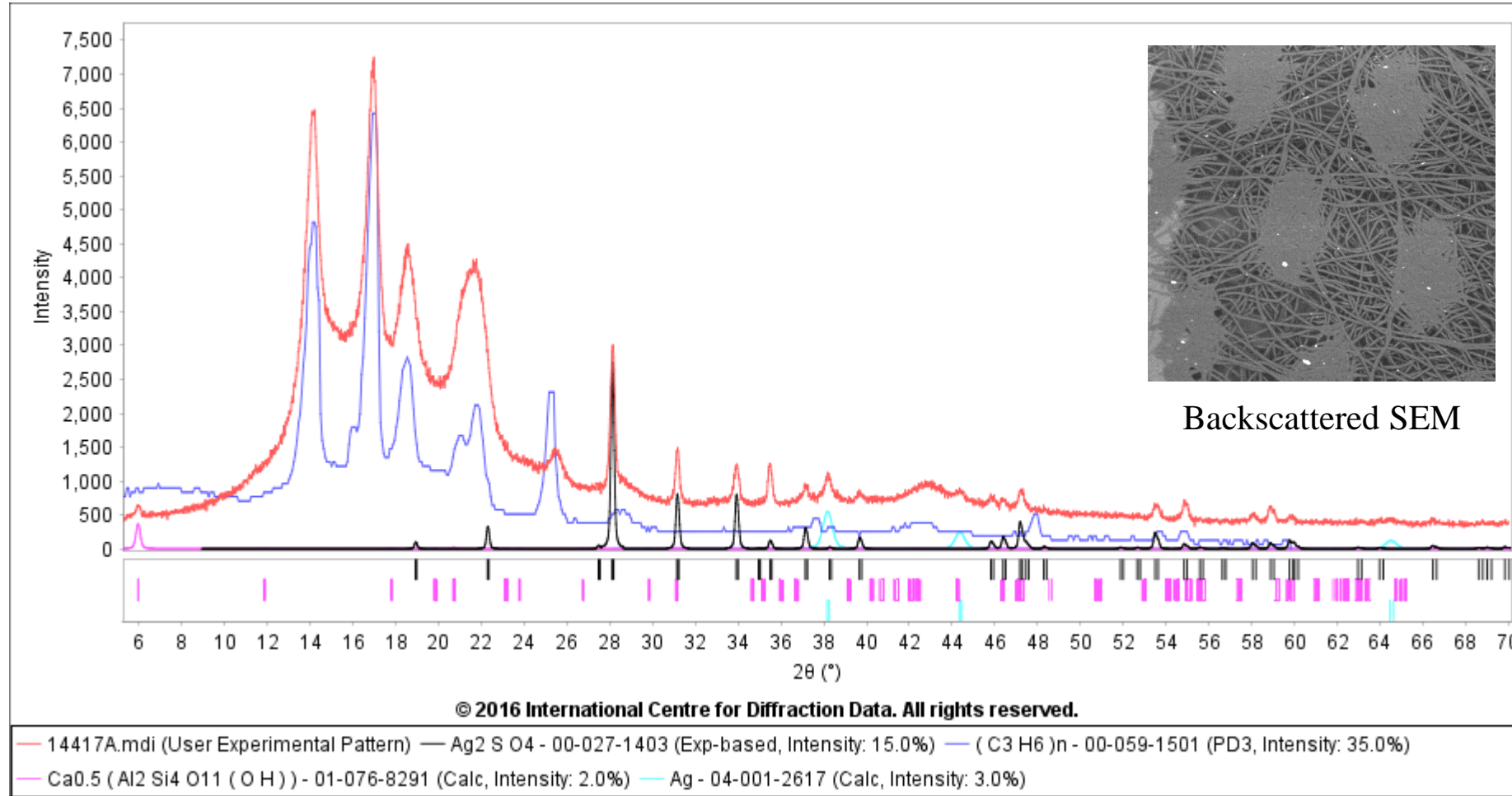
# ICDD biomedical polymers

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

## 2016 PDF-4 new polymer entries (PHR, BIO subfiles)

PDFID	Name	Chemical Formula	Common Name
00-66-1656	Ethylene vinyl alcohol	$(\text{C}_2\text{H}_4)_n (\text{C}_2\text{H}_3\text{O})_n$	Clarene
00-66-1657	Nylon 11	$(\text{NH}(\text{C}_9\text{H}_{17})\text{CO})_n$	Rilsan
00-66-1658	Polysulfone	$(\text{C}_6\text{H}_4\text{C}(\text{CH}_3)_2\text{C}_6\text{H}_4\text{OC}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{O})_n$	Udel
00-66-1659	Ethyl cellulose	$(\text{C}_6\text{H}_8\text{O}_5(\text{C}_2\text{H}_5)_2)_n$	Ethocel 45
00-66-1660	Ethyl cellulose	$(\text{C}_6\text{H}_8\text{O}_5(\text{C}_2\text{H}_5)_2)_n$	Ethocel 100
00-66-1661	Polyether ether ketone	$(\text{C}_{19}\text{H}_{12}\text{O}_3)_n$	PEEK - Amor
00-66-1662	Polyether ether ketone	$(\text{C}_{19}\text{H}_{12}\text{O}_3)_n$	PEEK - Crys
00-66-1663	Hydroxypropyl cellulose	$(\text{C}_{36}\text{H}_{70}\text{O}_{19})_n$	Methocal K15M
00-66-1666	Acetal	$(\text{C}_3\text{H}_4\text{O})_n$	Delrin
00-66-1667	Polystyrene, isotactic	$(\text{C}_8\text{H}_8)_n$	i-PS
00-66-1668	Polyethoxazoline	$(\text{N}(\text{COCH}_2\text{CH}_3)\text{CH}_2\text{CH}_2)_n$	PEOx
00-66-1669	Polyarylate	$(\text{CO}(\text{C}_6\text{H}_4)\text{CO}_2(\text{C}_6\text{H}_4)\text{C}(\text{CH}_3)_2(\text{C}_6\text{H}_4)\text{O})_n$	Ardel

# Phase ID – Ag based antimicrobial hospital gown



$\text{Ag}_2\text{SO}_4$  compounded in polypropylene was analyzed for phase composition. Using PDF-4 tools, phases were identified. Phases present:  $\alpha$ -polypropylene (base polymer),  $\text{Ag}_2\text{SO}_4$  (antimicrobial agent),  $\text{Ag}^0$  ( $\text{Ag}_2\text{SO}_4$  reduced during compounding), and montmorillonite (clay for improving polymer physical properties)



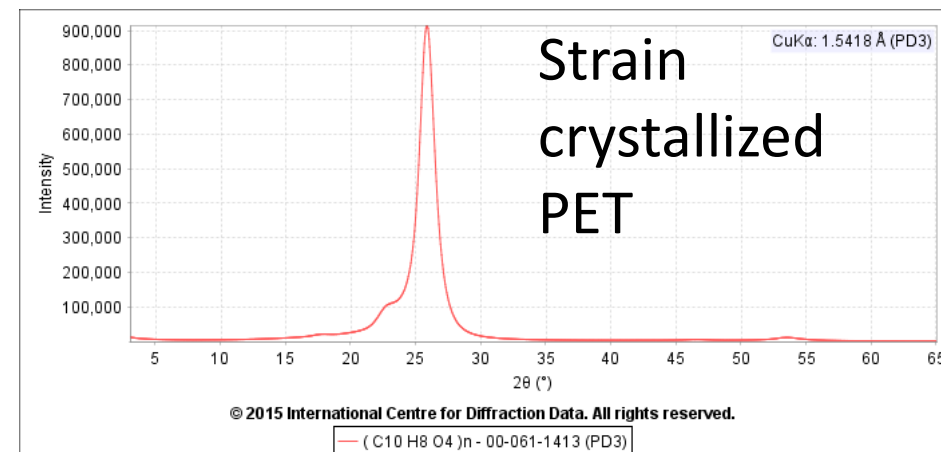
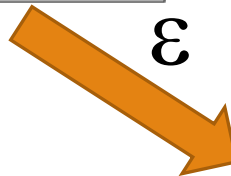
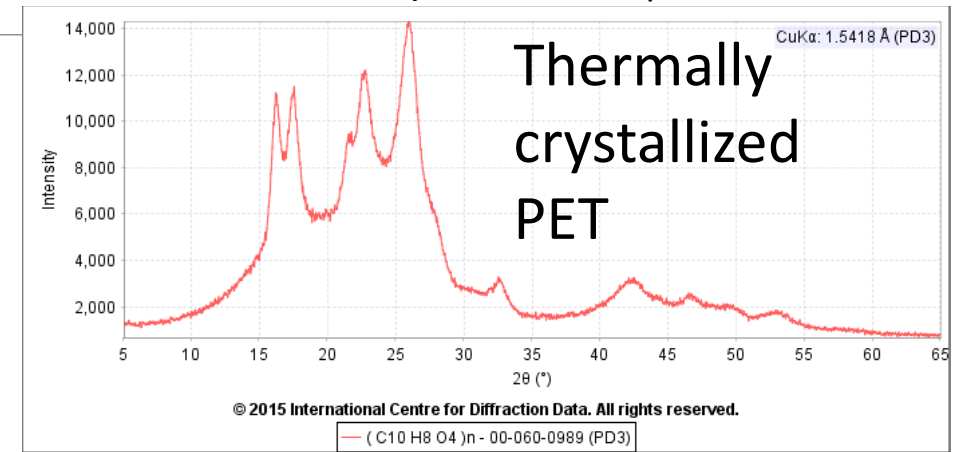
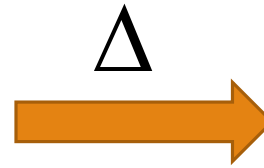
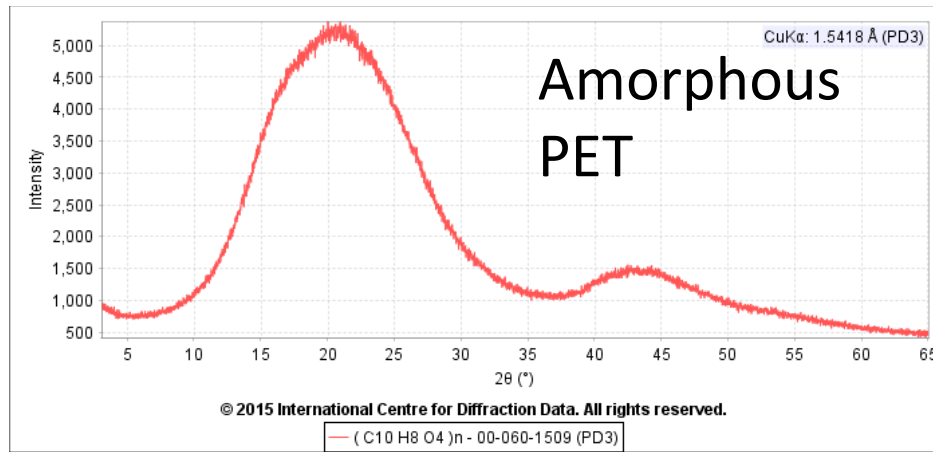
# Do you need multiple Entries in the PDF Database for a polymer?

PET

Triclinic P-1

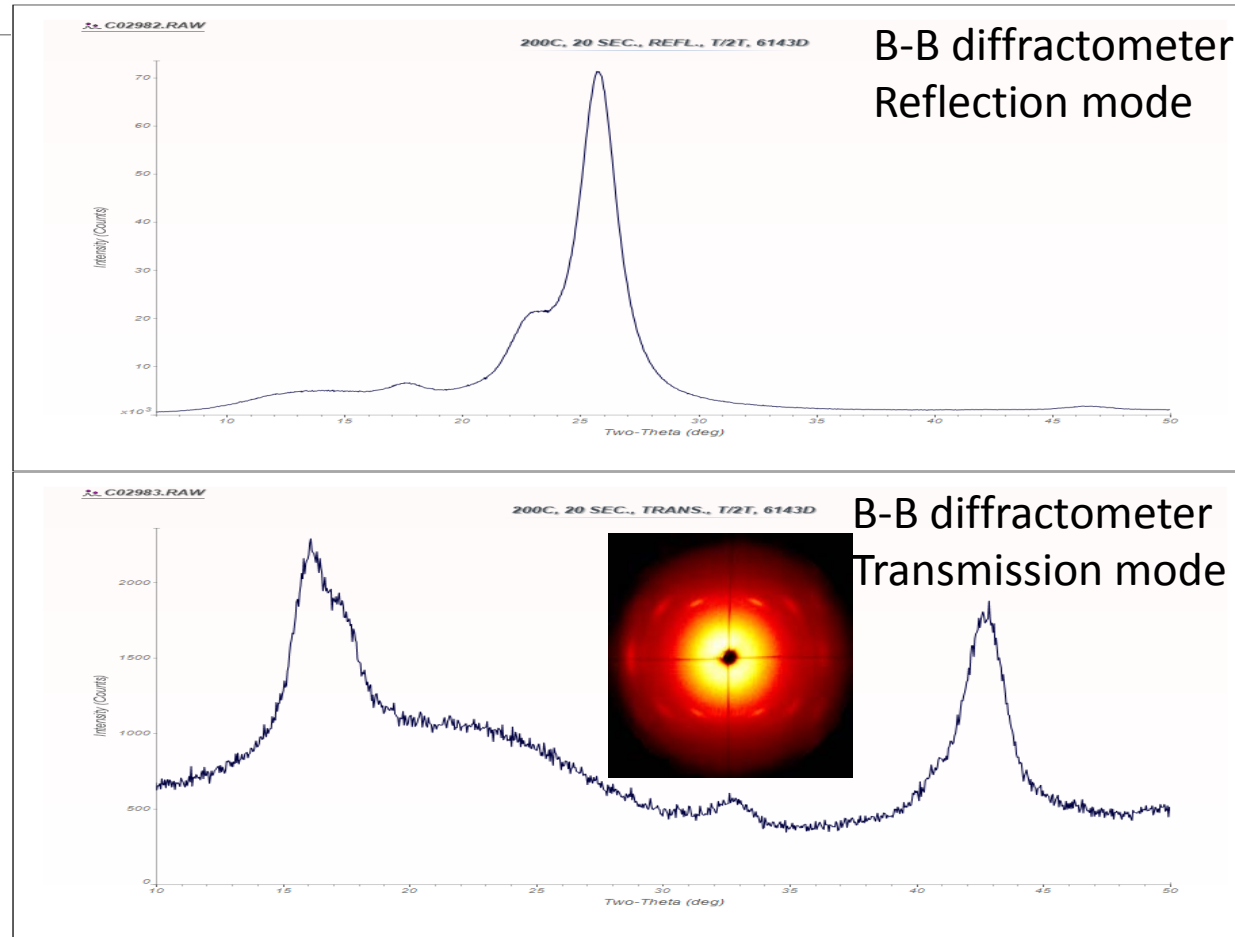
$a=5.791\text{\AA}$   $b=9.040\text{\AA}$   $c=4.561\text{\AA}$

$\alpha=100.64^\circ$   $\beta=112.46^\circ$   $\gamma=75.93^\circ$



# Polymer diffraction pattern: sample orientation matters

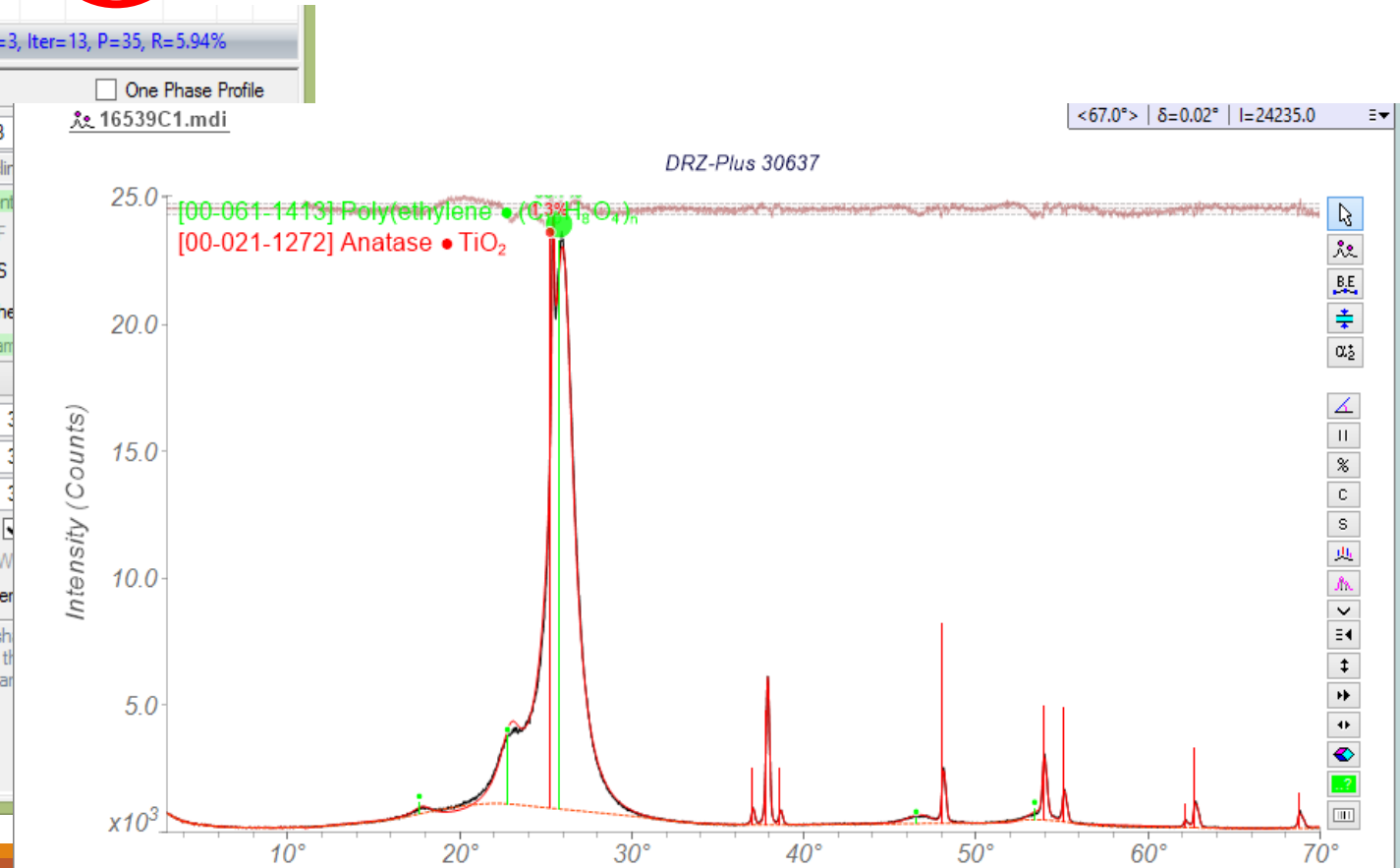
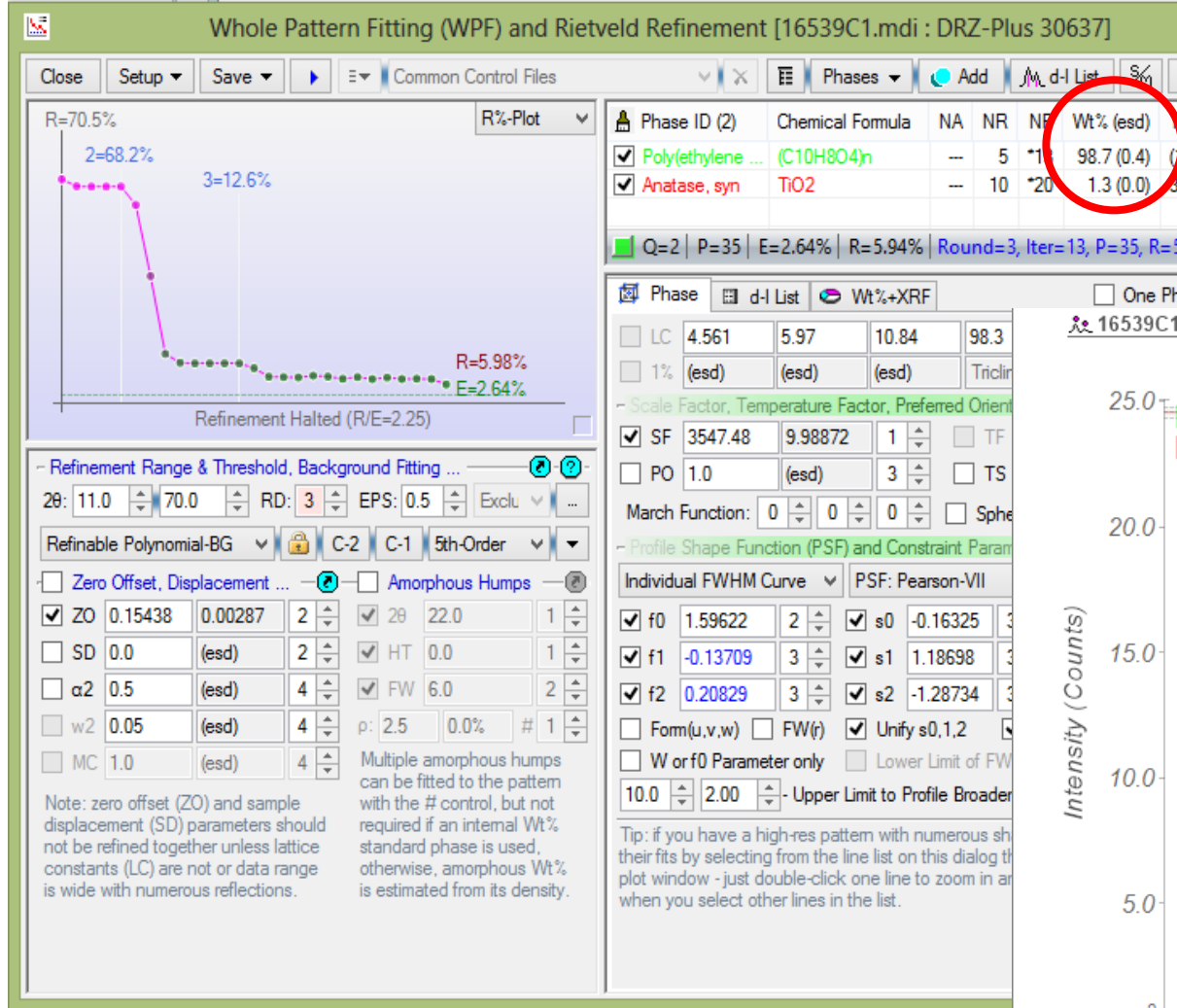
Strain crystallized PET



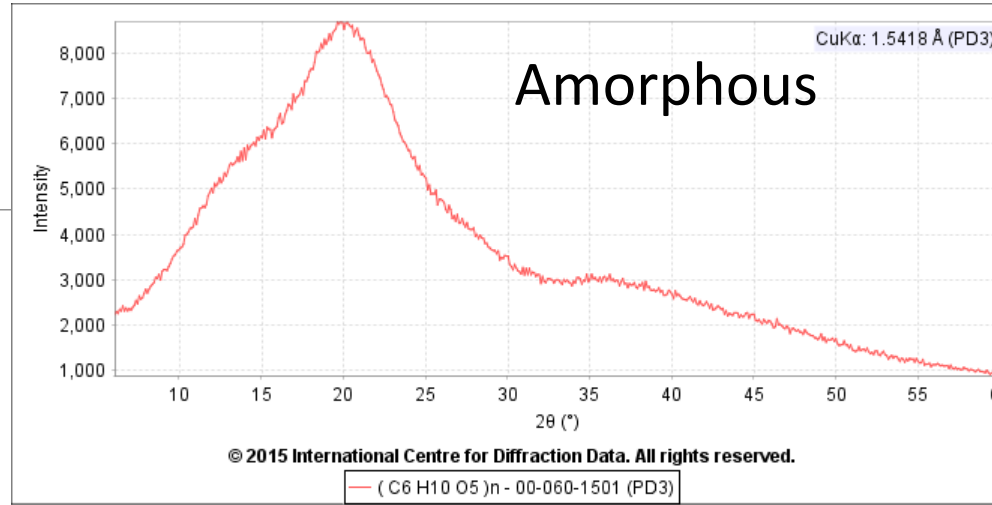


# Quantitative Phase Analysis PET Composite Film

Composite used for transdermal patch backing. XRD phase analysis identified biaxially oriented poly(ethylene terephthalate) (PDF 00-061-1413) and TiO<sub>2</sub>-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% TiO<sub>2</sub> present, in line with the formulation calling for 1.5wt% TiO<sub>2</sub>.

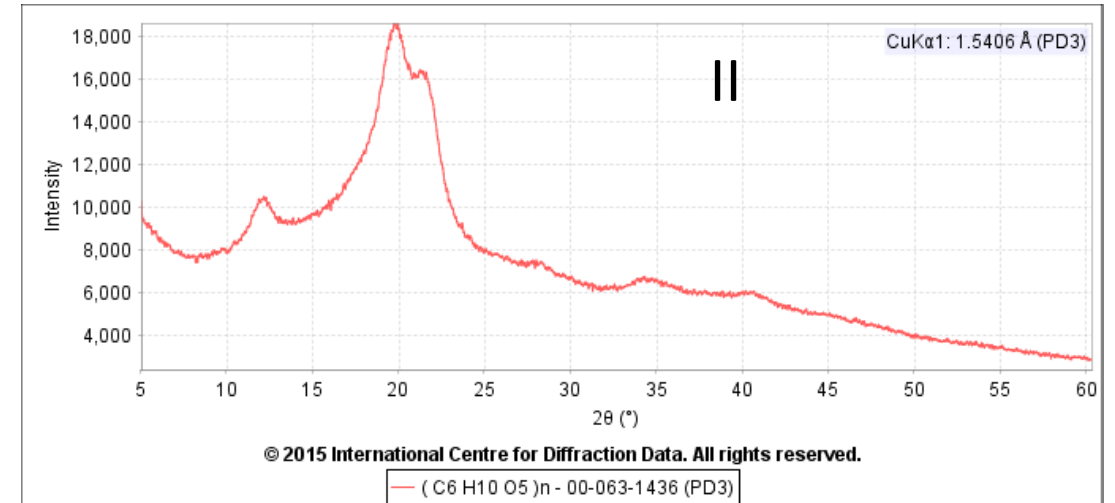
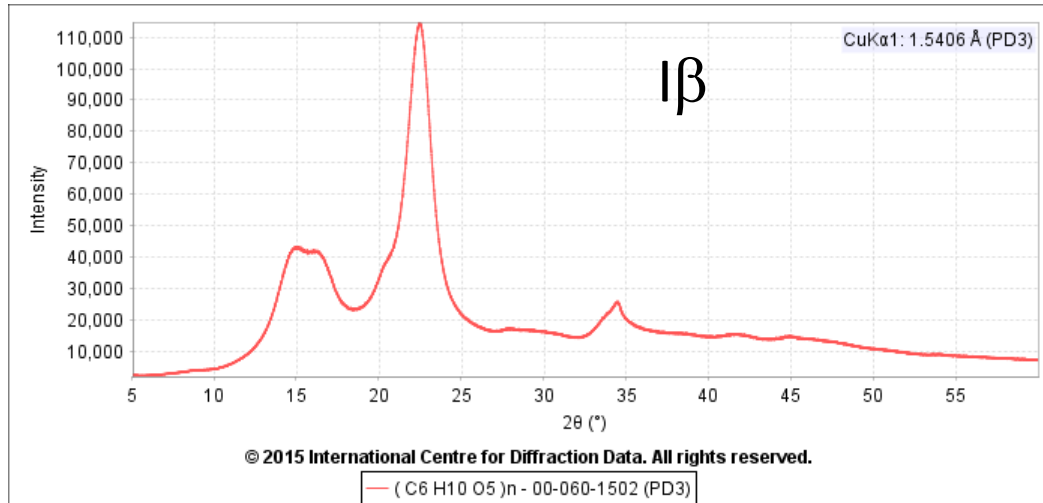


# Cellulose

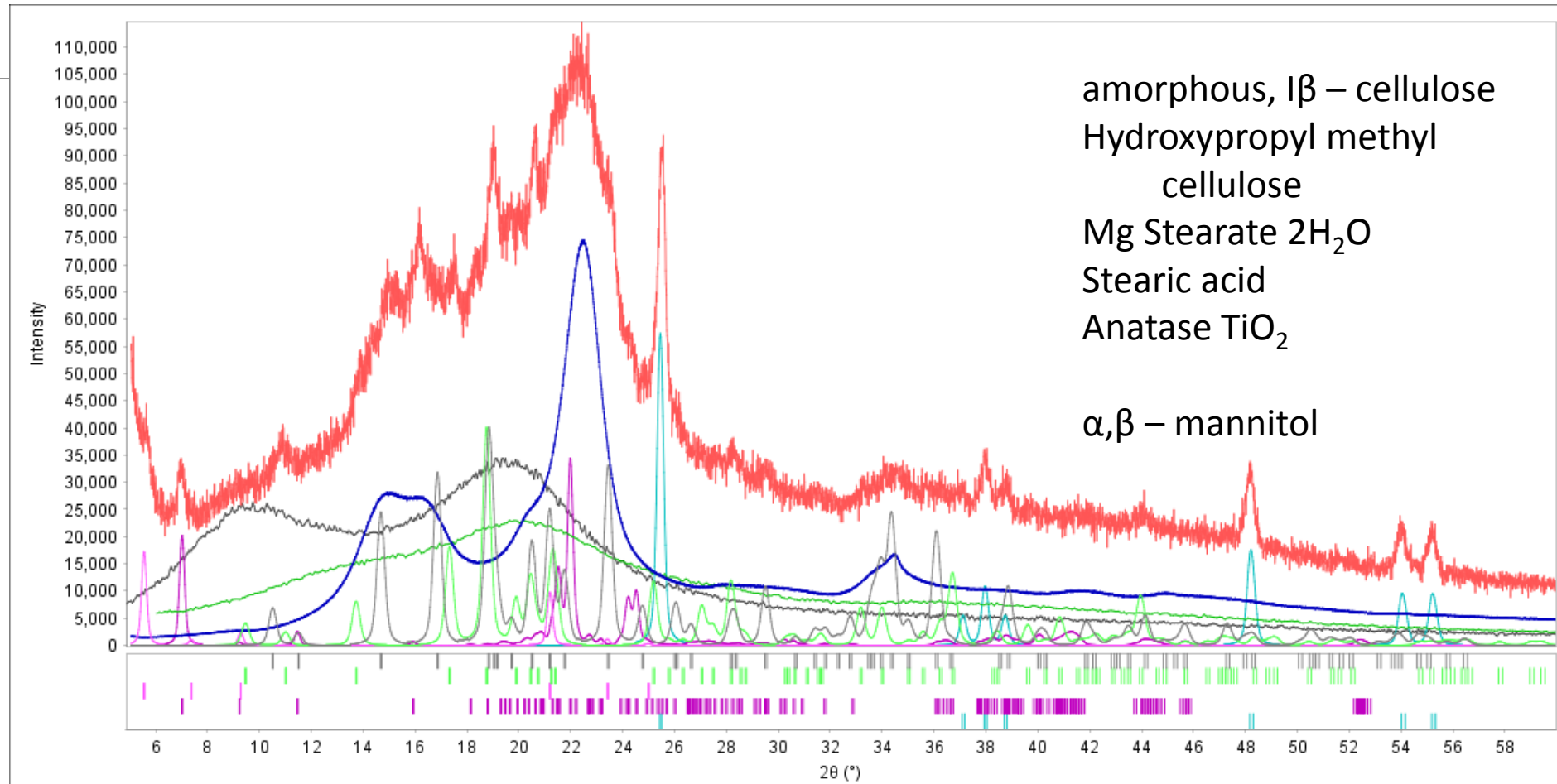


Monoclinic P<sub>2</sub><sub>1</sub>  
a=8.260Å b=10.388Å c=7.740Å  
 $\beta$ =95.80°

Monoclinic P<sub>2</sub><sub>1</sub>  
a=9.044Å b=10.387Å c=8.077Å  
 $\beta$ =115.73°



# Phase identification – Allegra<sup>®</sup> shell

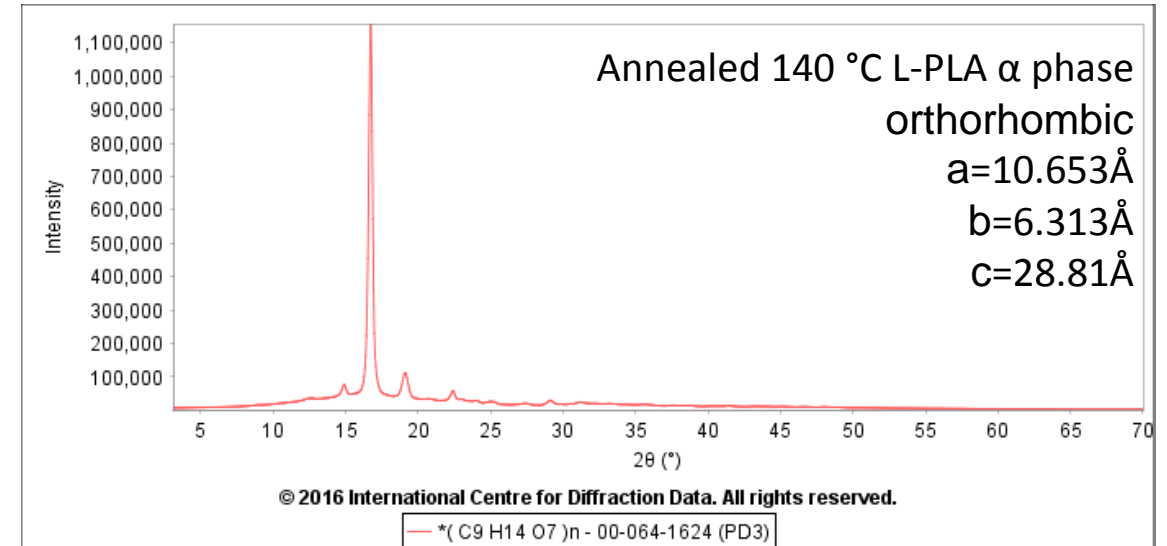
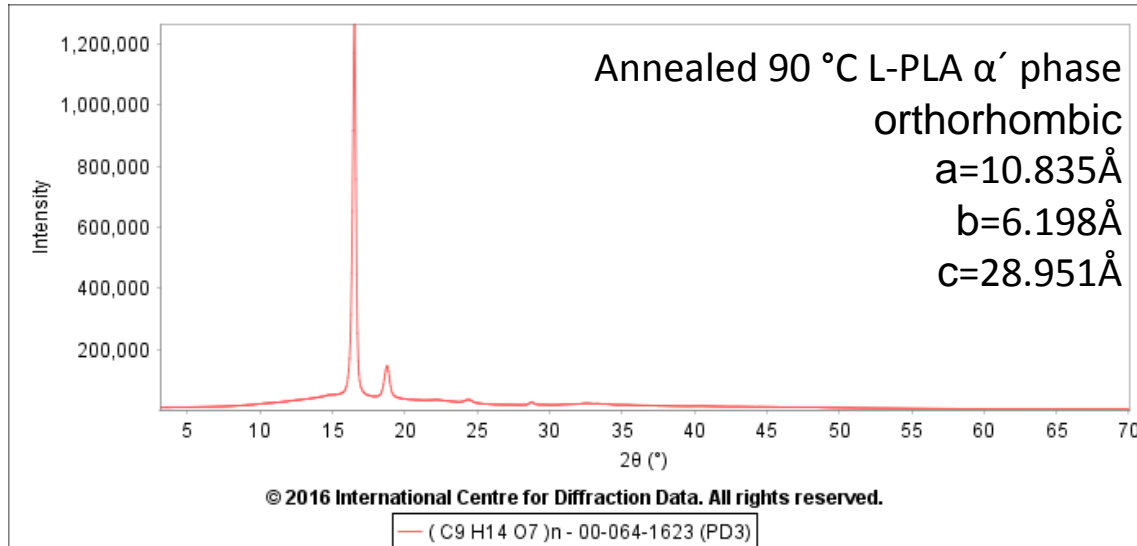
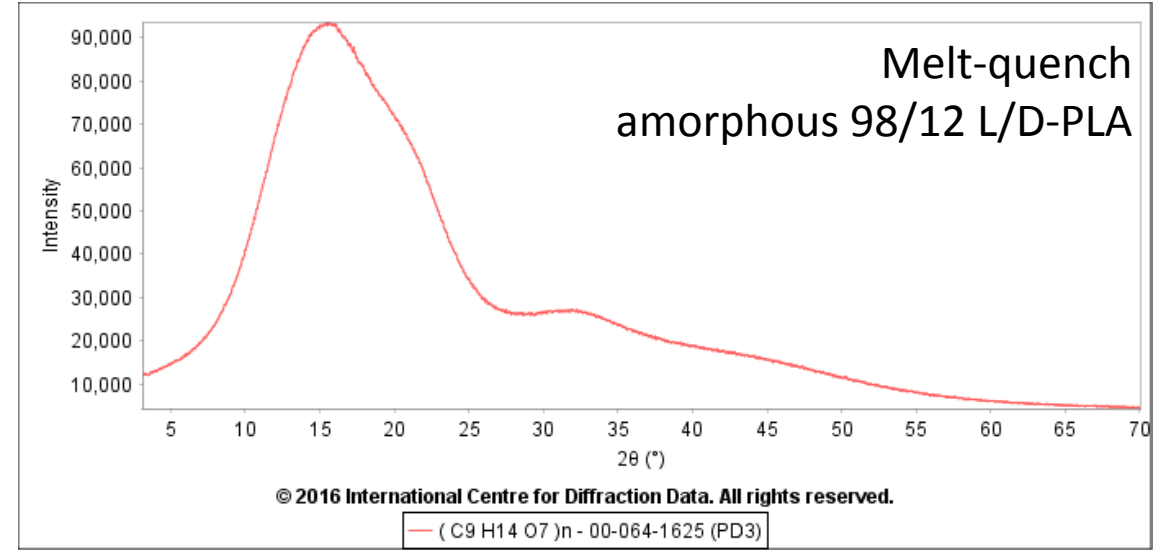
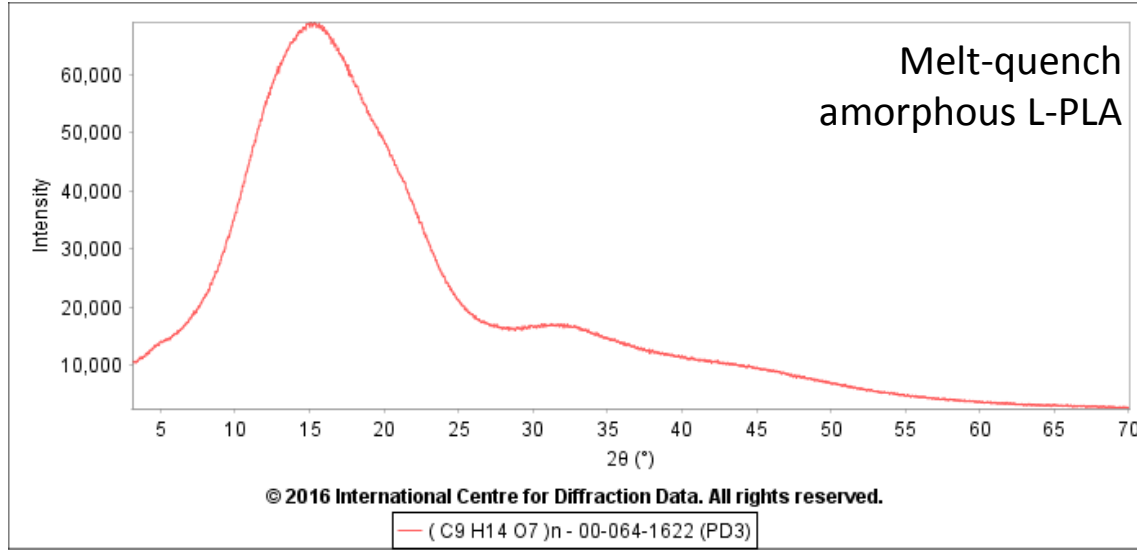


amorphous, β – cellulose  
 Hydroxypropyl methyl  
 cellulose  
 Mg Stearate 2H<sub>2</sub>O  
 Stearic acid  
 Anatase TiO<sub>2</sub>  
  
 α,β – mannitol

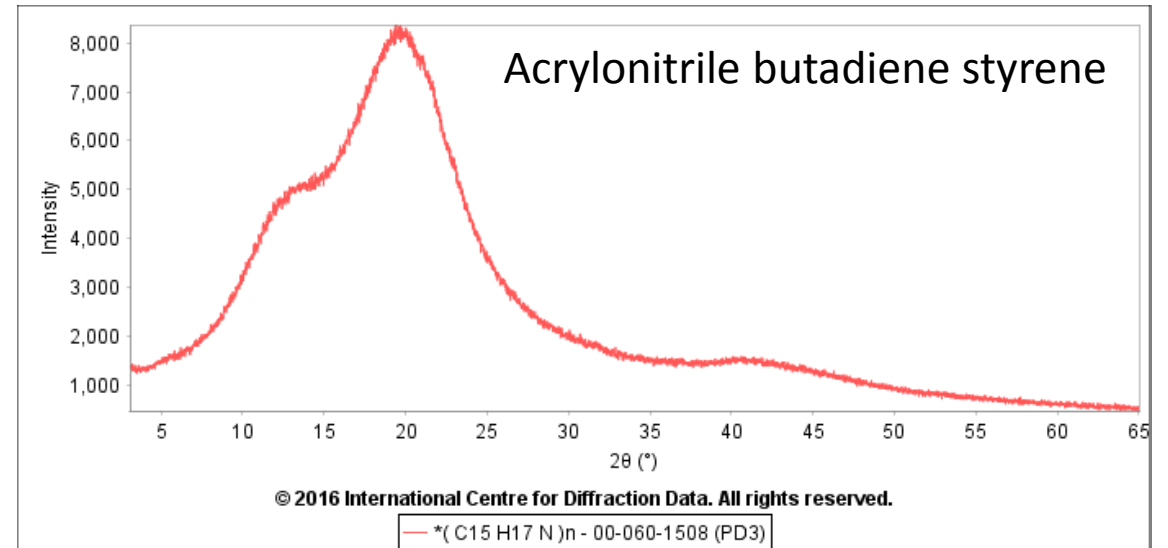
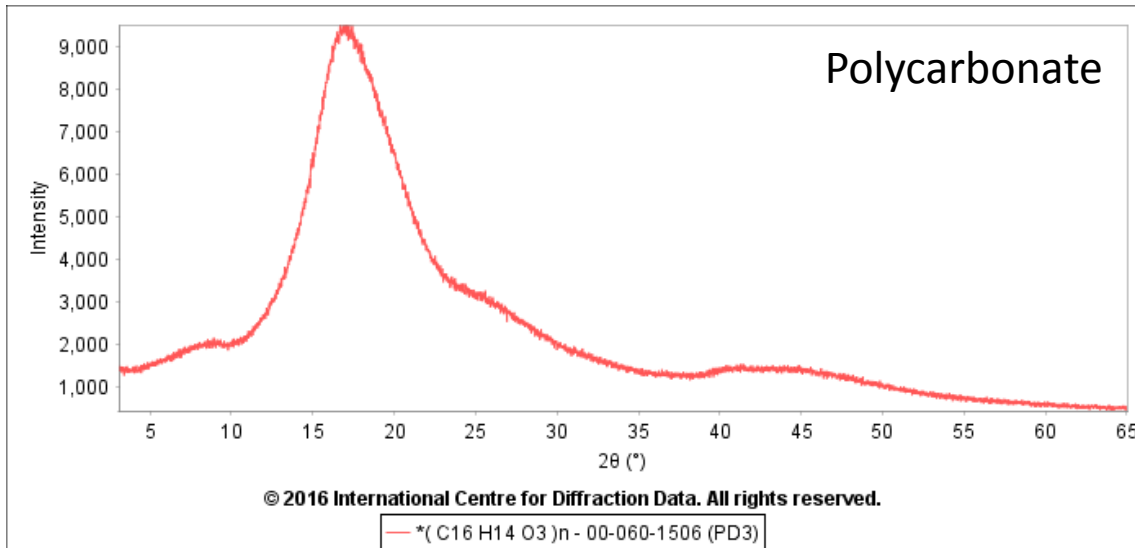
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- C6 H14 O6 - 00-022-1797 (Exp-based, Intensity: 35.0%)
- ( C6 H10 O5 )n - 00-060-1502 (PD3, Intensity: 65.0%)
- allegra shell.dat (User Experimental Pattern)
- C6 H14 O6 - 00-022-1793 (Exp-based, Intensity: 35.0%)
- \*( C6 H10 O5 )n - 00-060-1501 (PD3, Intensity: 20.0%)
- Ti O2 - 00-021-1272 (Exp-based, Intensity: 50.0%)
- C36 H70 Mg O4 · 2 H2 O - 00-054-1973 (Exp-based, Intensity: 15.0%)
- C18 H36 O2 - 00-038-1923 (Exp-based, Intensity: 30.0%)
- ( C36 H70 O19 )n - 00-066-1663 (PD3, Intensity: 30.0%)

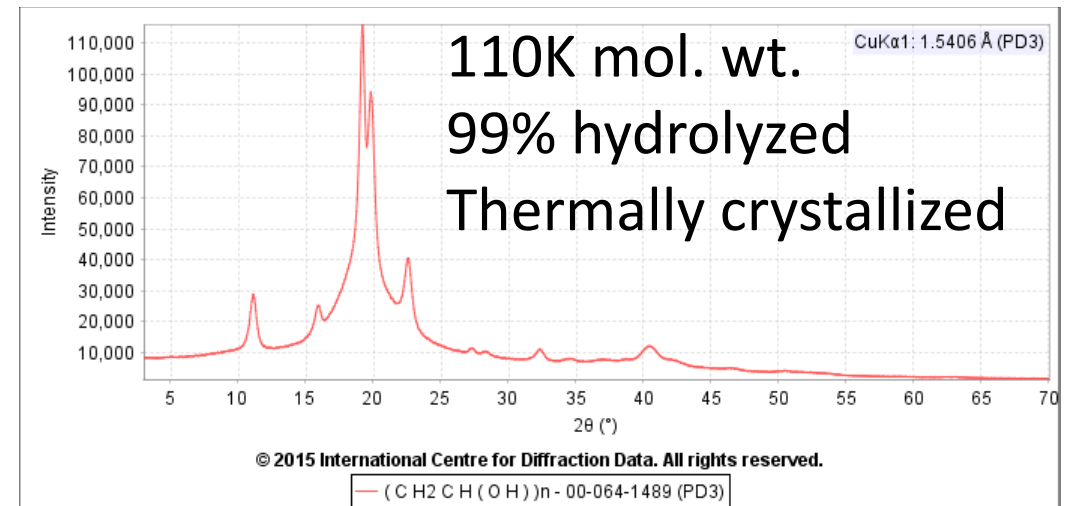
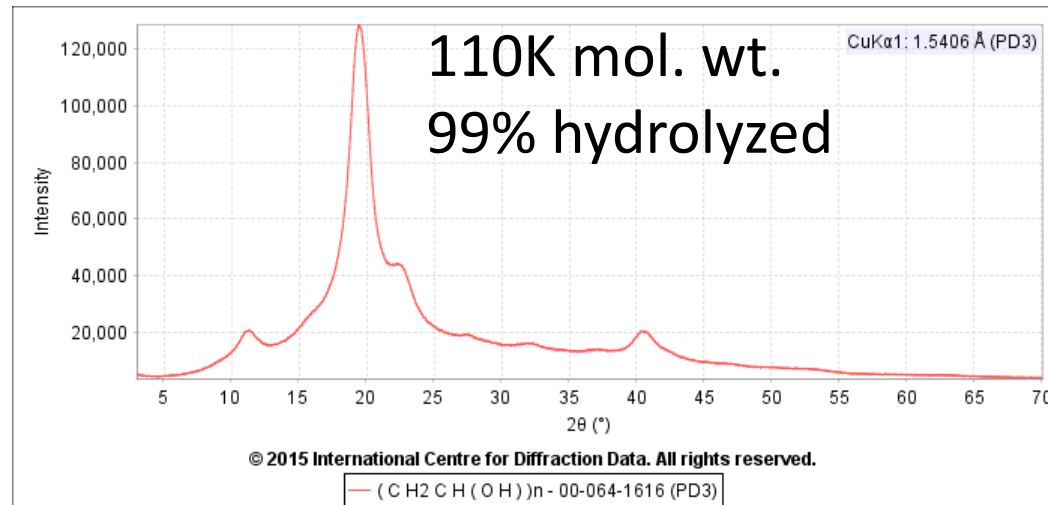
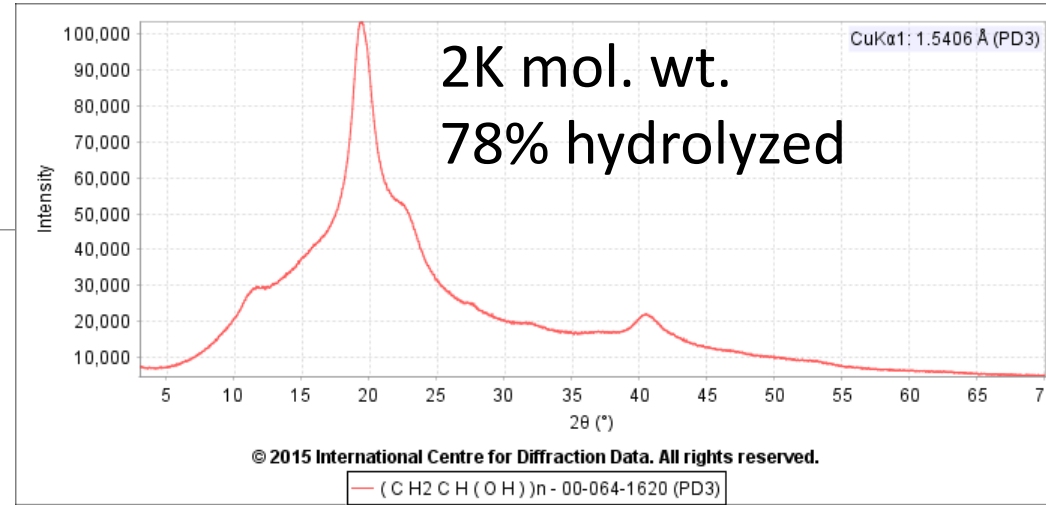
# Poly Lactic Acid PLA



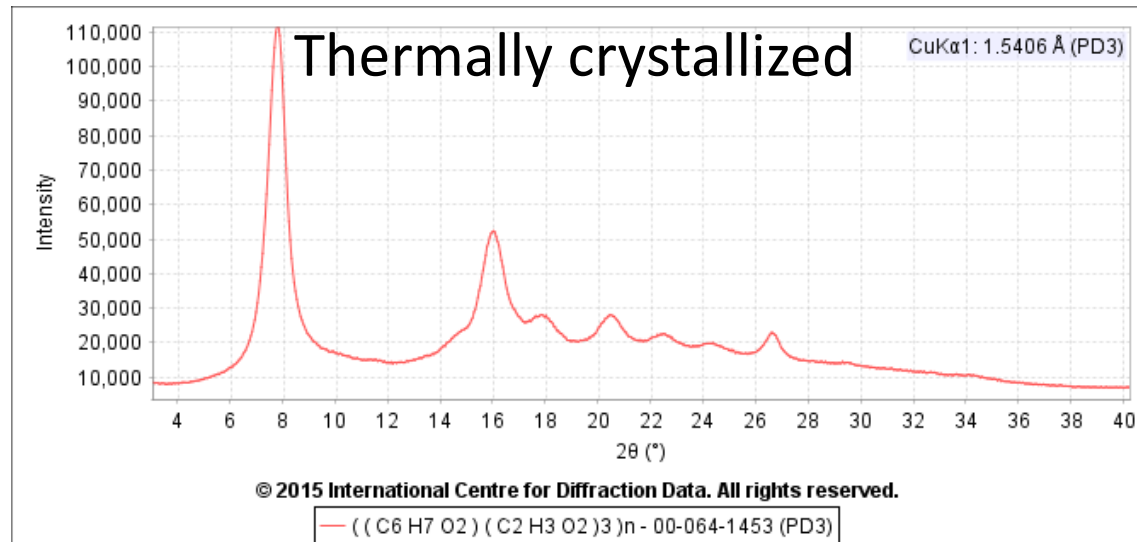
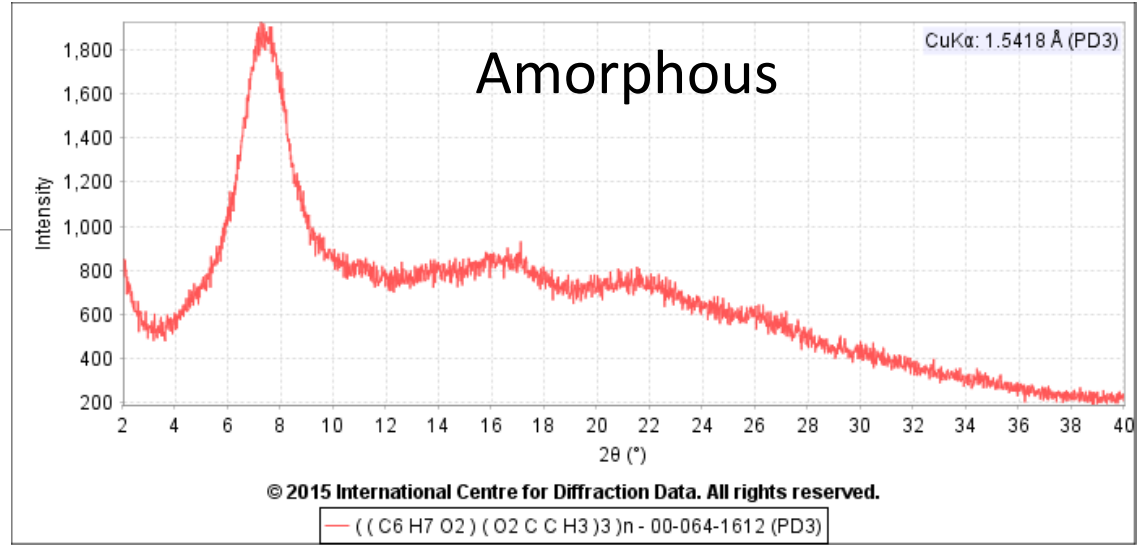
# 3d printing polymers, continued



# Polyvinyl alcohol

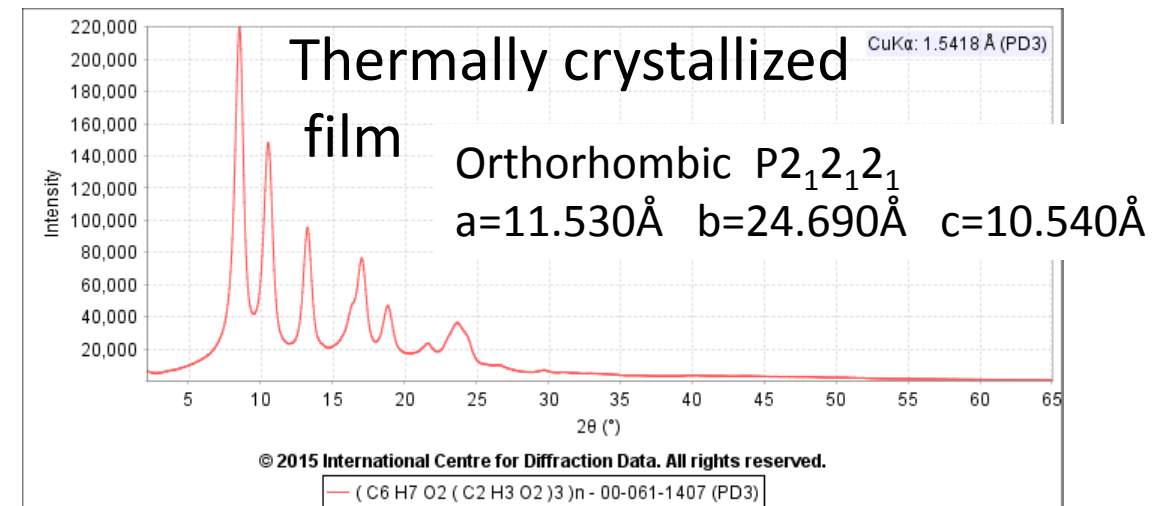
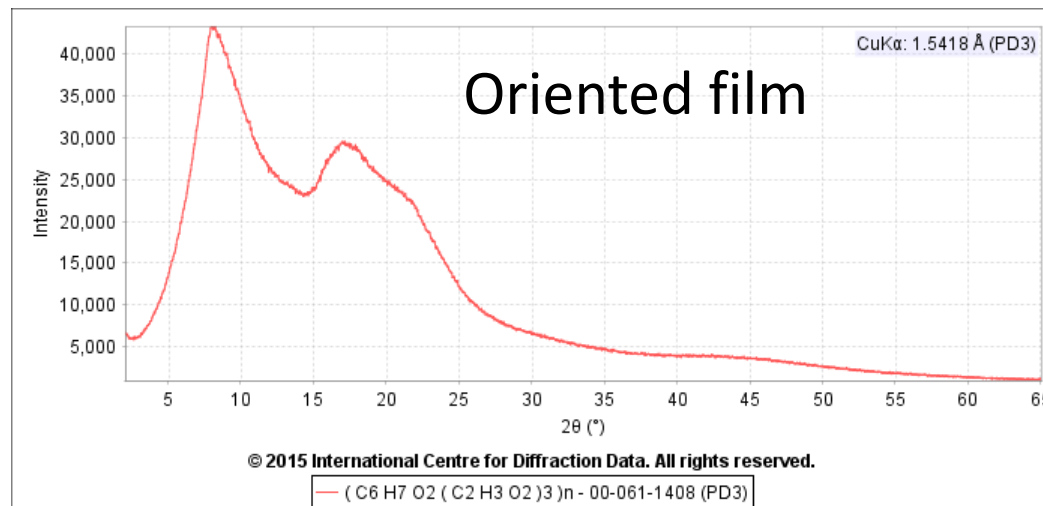
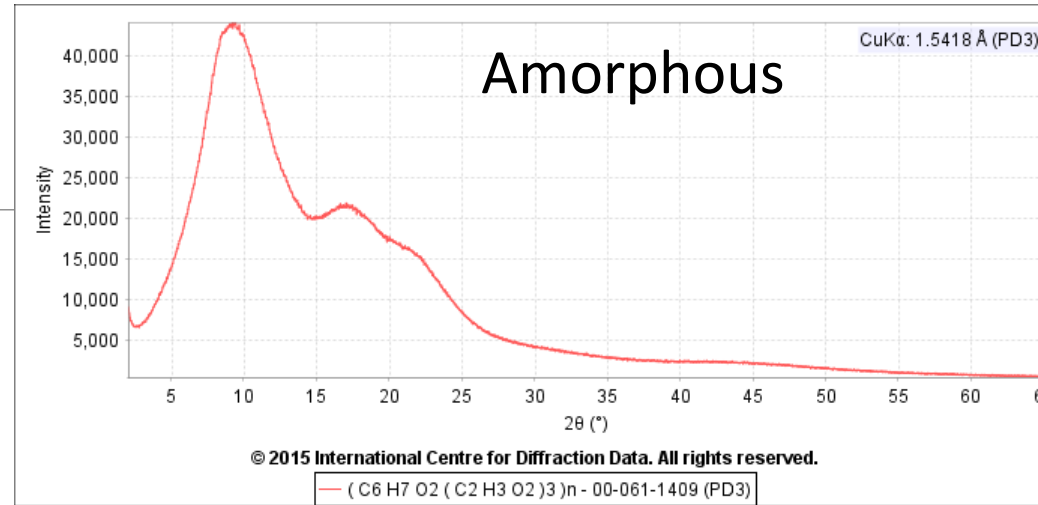


# Cellulose triacetate I



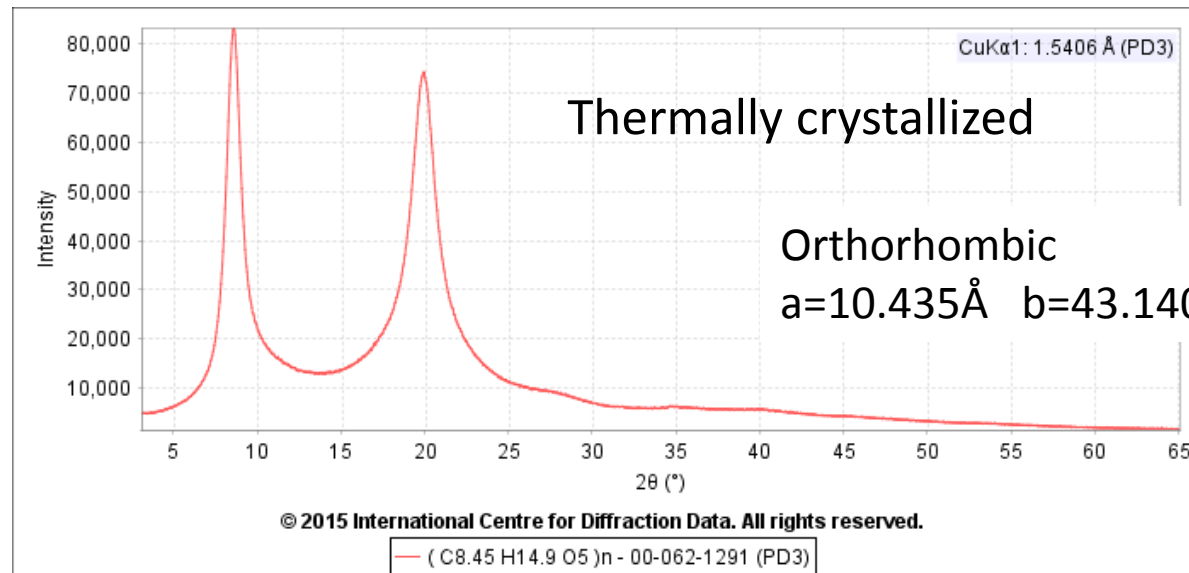
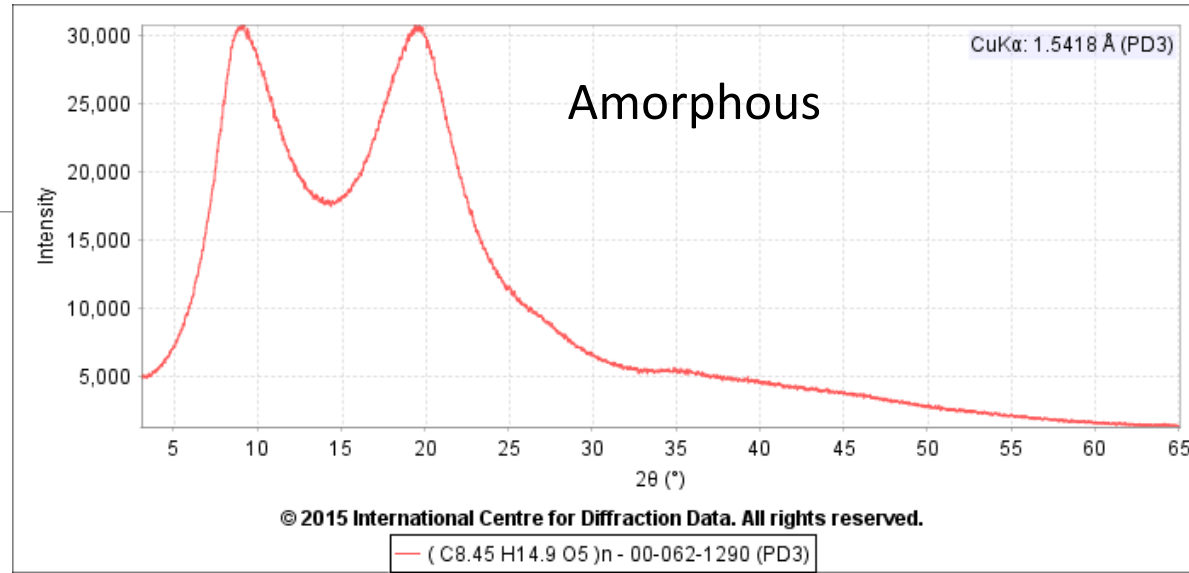
Monoclinic  $P2_1$   
 $a=11.348\text{\AA}$   $b=24.690\text{\AA}$   $c=6.060\text{\AA}$   
 $\beta=95.60^\circ$

# Cellulose triacetate II



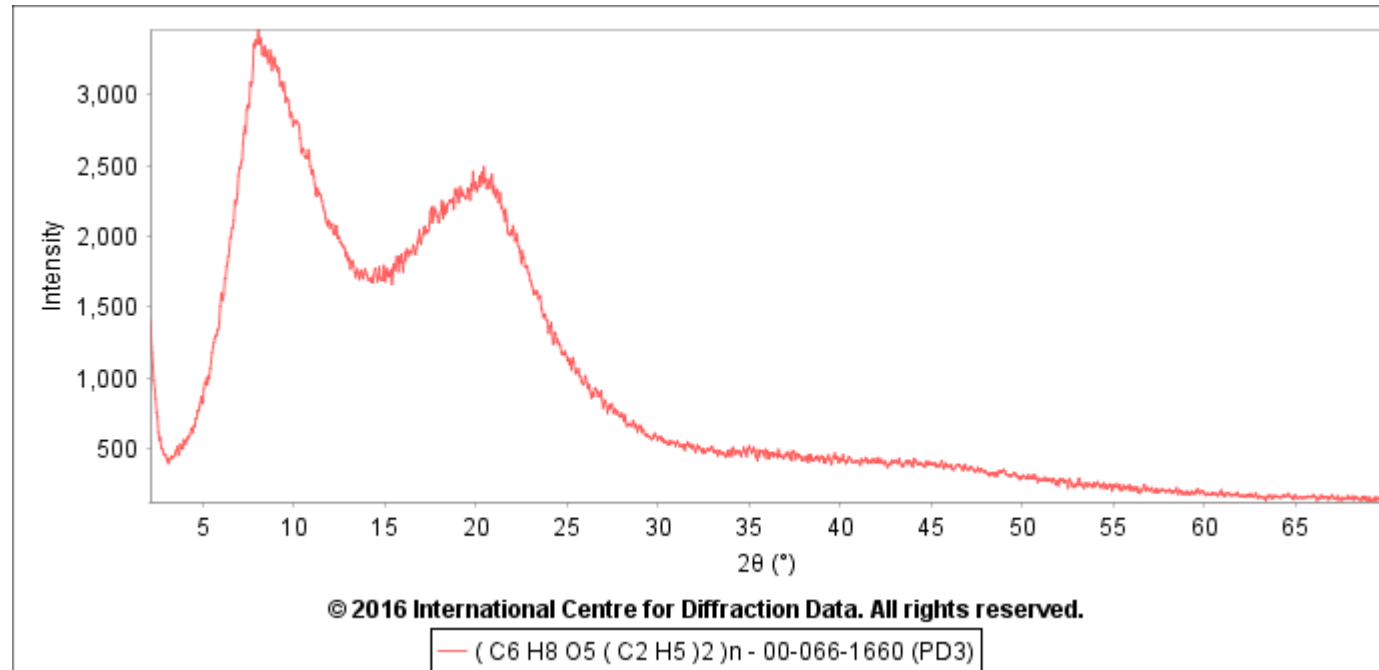


# Methyl cellulose

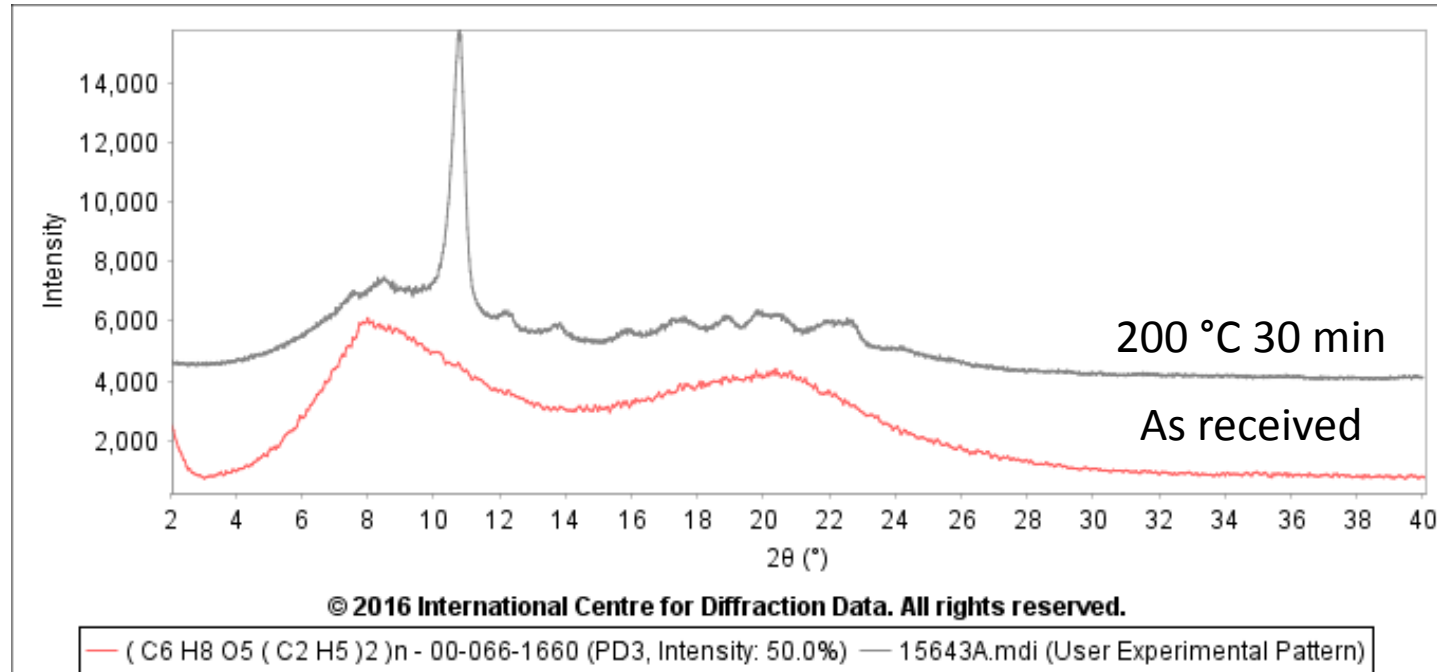


# Ethyl cellulose – new entry for 2016

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# Ethyl cellulose



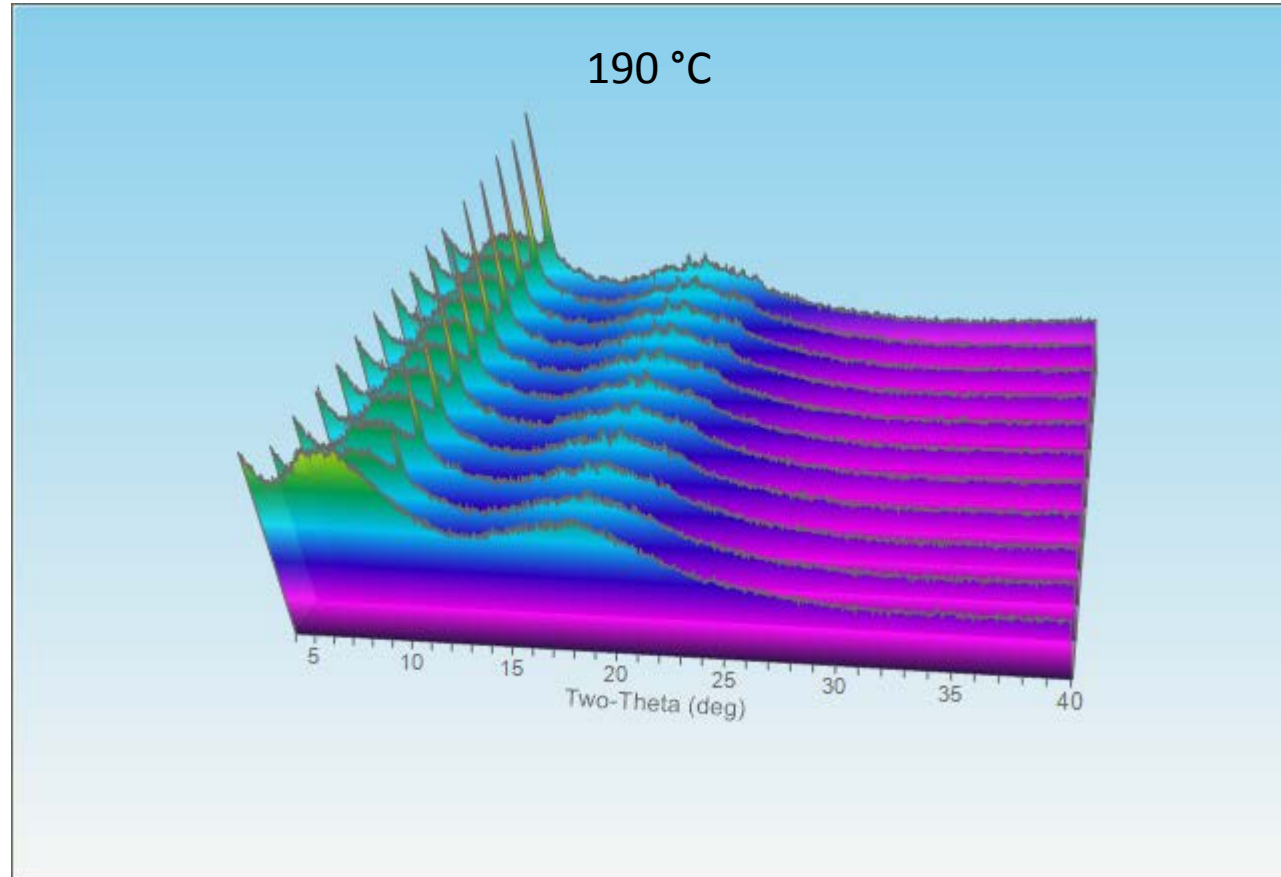
# Indexing/WPF of crystalline “ethyl cellulose” phase



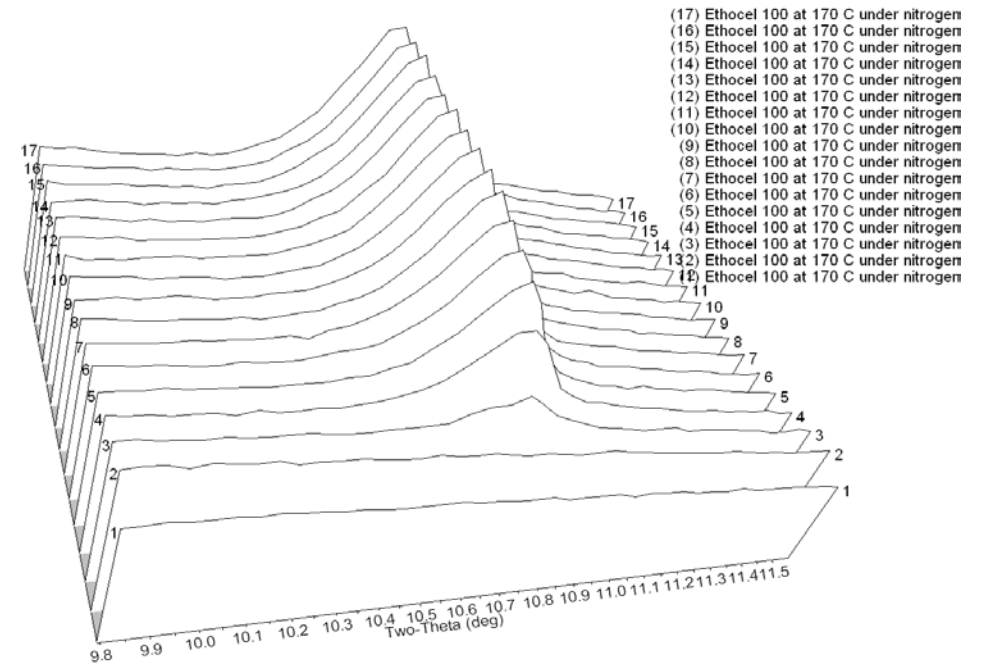
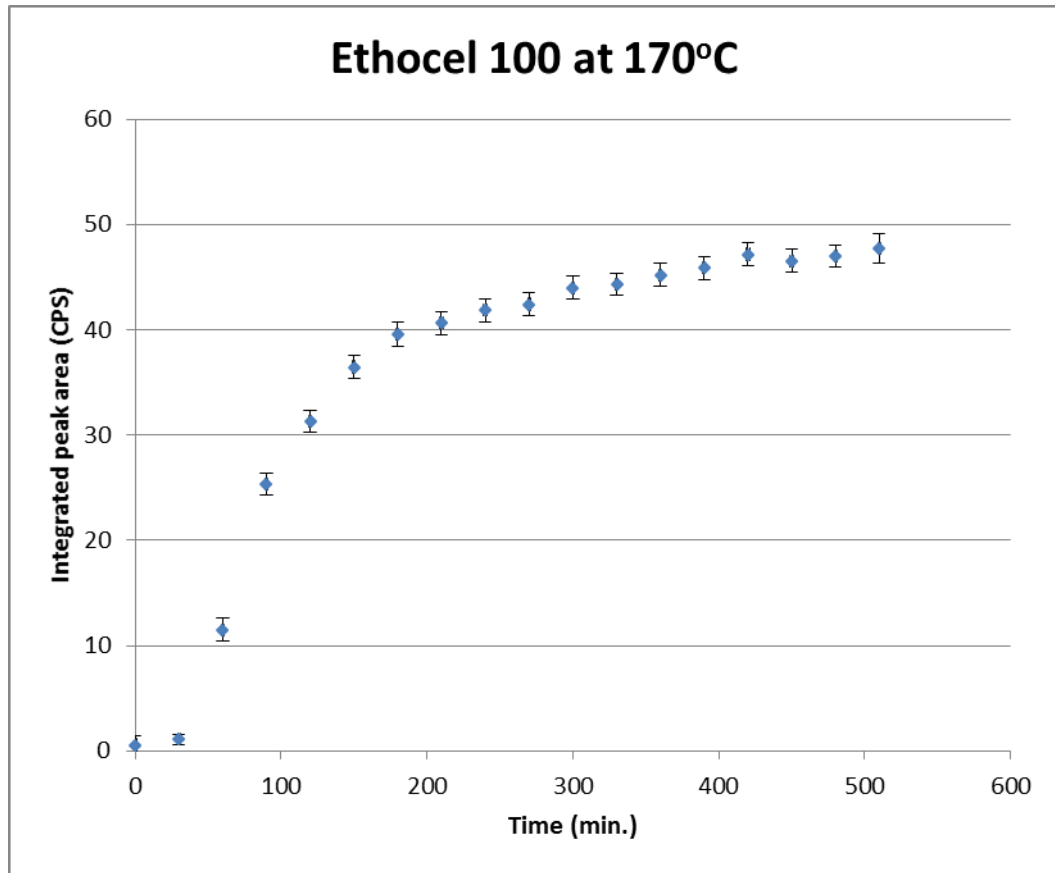
Possible cell:  
 Monoclinic  
 SG  $P2_1/c$   
 $a=11.74\text{\AA}$   
 $b=23.50\text{\AA}$   
 $c=7.65\text{\AA}$   
 $\beta=95.2^\circ$

Collecting supporting data:  
 TGA/DSC, NMR, IR, XRF, SEM

# Ethyl cellulose – isothermal anneal studies



# Ethyl cellulose – isothermal anneal studies



# 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

