



# POWDER DIFFRACTION

Possibilities – Problems

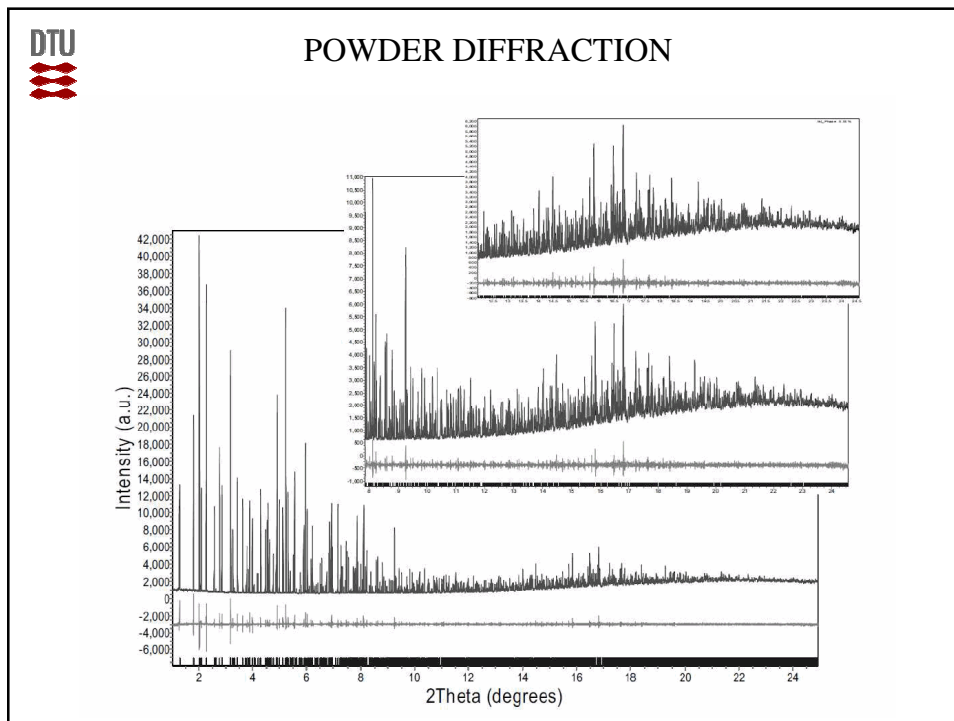
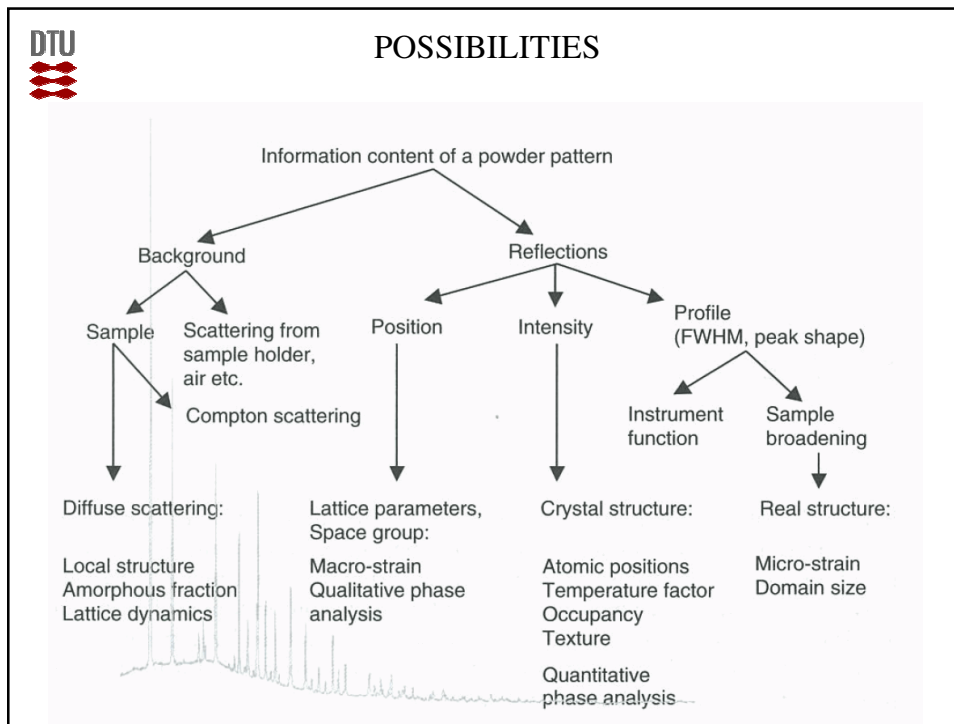
Kenny Ståhl

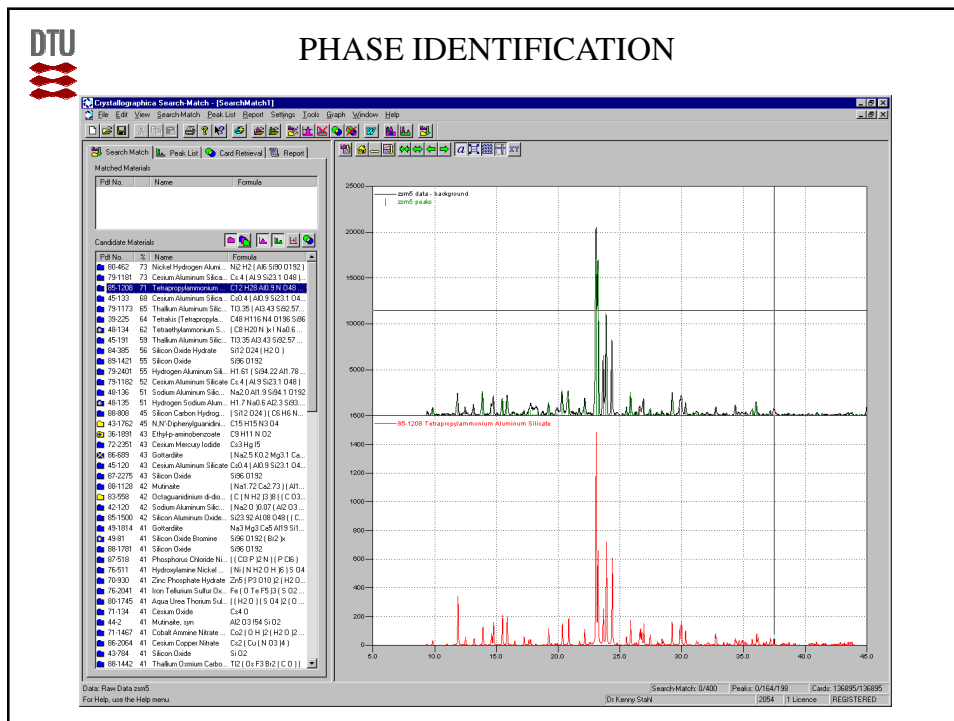
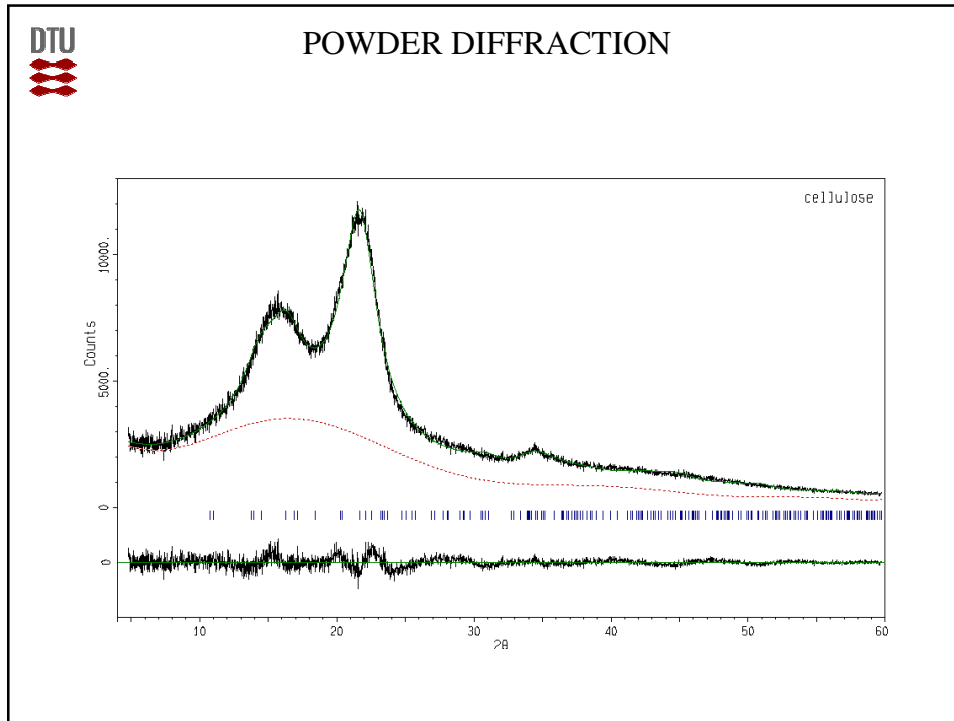
DTU Chemistry

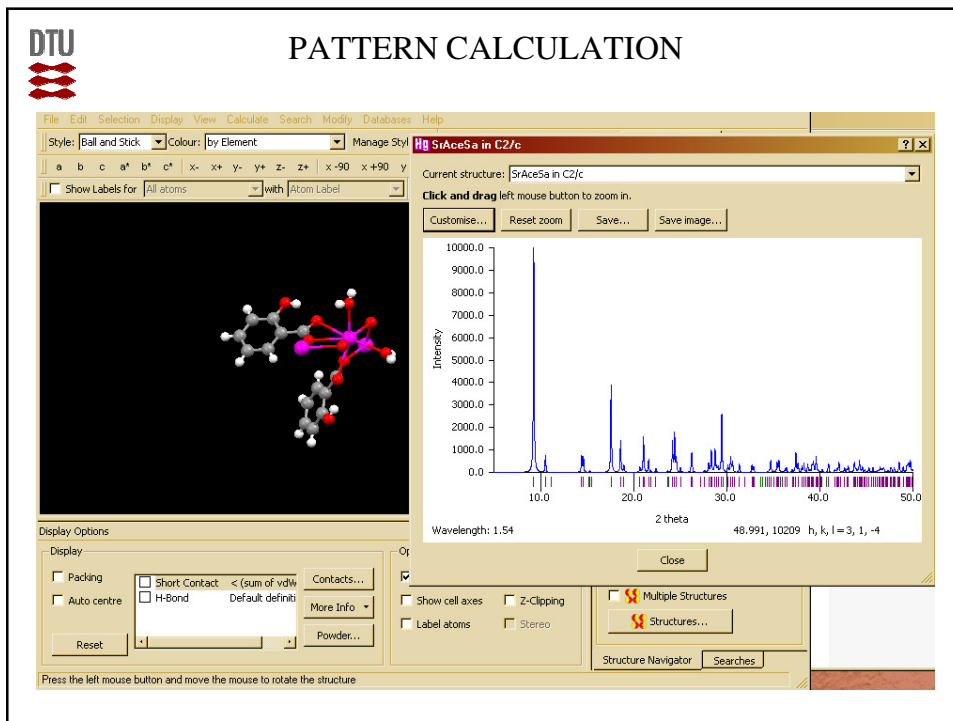
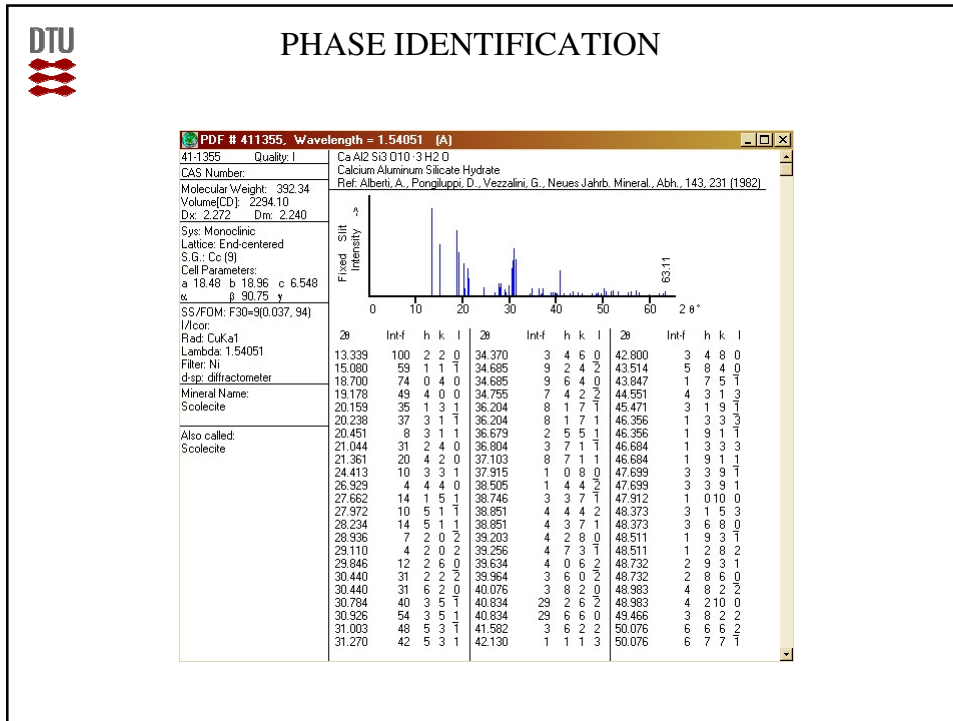



## CONTENTS

- Information from a powder diffraction pattern
- Appearances
- Phase identification
- Indexing
- Stress/strain
- Rietveld refinements
- Background, counting statistics
- Preferred orientation
- Absorption
- Axial divergence










## INDEXING

	2θ	sin <sup>2</sup> θ	(h <sup>2</sup> + k <sup>2</sup> + l <sup>2</sup> )	h k l
	25.96	0.05043		
	30.01	0.06704		
Cubic:	43.06	0.13468		
$(1 / d_{hkl})^2 = (h^2 + k^2 + l^2) / a^2$	50.97	0.18517		
$2 d_{hkl} \sin\theta_{hkl} = \lambda$	53.41	0.20196		
$\sin^2 \theta_{hkl} = (h^2 + k^2 + l^2) \lambda^2 / (4a^2)$	62.54	0.26941		
	68.88	0.31984		
	70.97	0.33693		
	78.92	0.40391		

$$\sin^2 \theta_{hkl} = h^2 X_1 + k^2 X_2 + l^2 X_3 + hk X_4 + hl X_5 + kl X_6$$

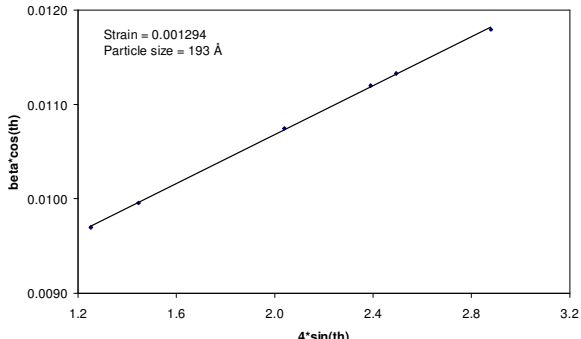


## PARTICLE SIZE - STRESS / STRAIN (DEFECTS)

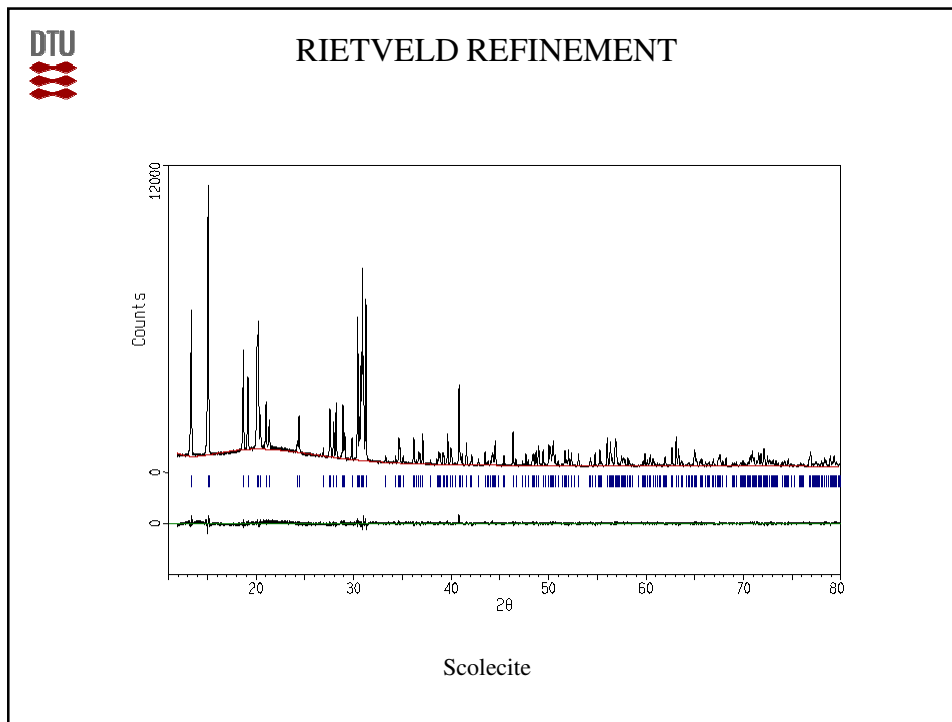
**Size (τ):**  $\beta = k \lambda / \tau \cos(\theta)$  (Sherrer equation)  
 $\beta^2 = FWHM_{obs}^2 - FWHM_{ref}^2$  (rad)

**Stress/strain (ε):**  $\beta = 4 \epsilon \tan(\theta)$

**Williamson-Hall:**  $\beta = k \lambda / \tau \cos(\theta) + 4 \epsilon \tan(\theta)$   
 $\beta \cos(\theta) = k \lambda / \tau + 4 \epsilon \sin(\theta)$



Strain = 0.001294  
Particle size = 193 Å



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### RIETVELD REFINEMENT

Least-squares:  $D = \sum_j w_j (Y_{oi} - Y_{ci})^2$

$$Y_{ci} = B_i + S \sum_{hkl} A(2\theta) P_{hkl} L_p(2\theta) \Phi(2\theta - 2\theta_{Bragg}) |F_{hkl}|^2$$

$Y_{ci}$  = Calculated intensity  
 $B_i$  = Background intensity  
 $S$  = Scale factor  
 $A(2\theta)$  = Absorption correction  
 $P_{hkl}$  = Preferred orientation correction  
 $L_p(2\theta)$  = Lorentz and polarization correction  
 $\Phi(2\theta - 2\theta_{Bragg})$  = Profile function  
 $|F_{hkl}|^2$  = Diffracted (single-crystal) intensity



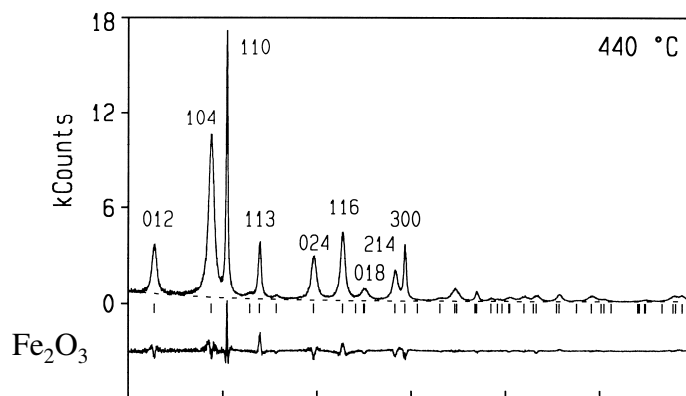
## RIETVELD REFINEMENT

Scolecite, Huber data

Si(1)	- O(1)	1.632(7)			
	- O(2)	1.593(6)	Al(1)	- O(1)	1.694(8)
	- O(9)	1.597(6)		- O(3)	1.691(7)
	- O(10)	1.609(7)		- O(5)	1.786(8)
Si(2)	- O(3)	1.676(8)		- O(7)	1.748(8)
	- O(6)	1.642(7)	Al(2)	- O(2)	1.775(8)
	- O(7)	1.610(7)		- O(4)	1.768(8)
	- O(9)	1.636(8)		- O(6)	1.739(7)
Si(3)	- O(4)	1.586(8)		- O(8)	1.730(8)
	- O(5)	1.621(8)			<u>1.75 Å</u>
	- O(8)	1.622(7)			
	- O(10)	1.641(8)			<u>1.62 Å</u>



## ANISOTROPIC SIZE EFFECTS



$a = 5.0364(8)$ ,  $c = 13.750(2)$  Å      $D(a) = 399(3)$  Å,  $D(c) = 87(2)$  Å

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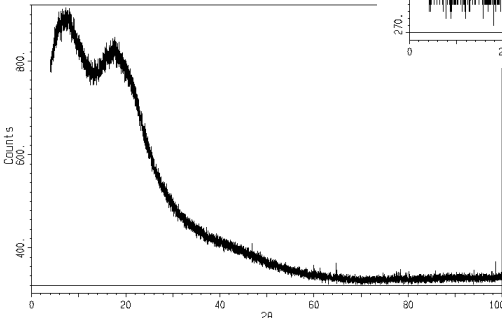
## PROBLEMS - ERRORS

<p>Background</p> <p>Preferred orientation</p> <p>Systematic errors</p>	<p>General</p> <p>Fluorescence/incoherent scattering</p> <p>Counting statistics</p> <p>Poor sample</p> <p>Texture</p> <p>Absorption – intensities</p> <p>Absorption – peak positions</p> <p>Axial divergence</p>
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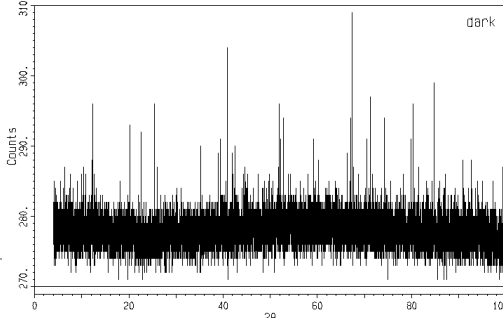
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## BACKGROUND

Read-out-noise

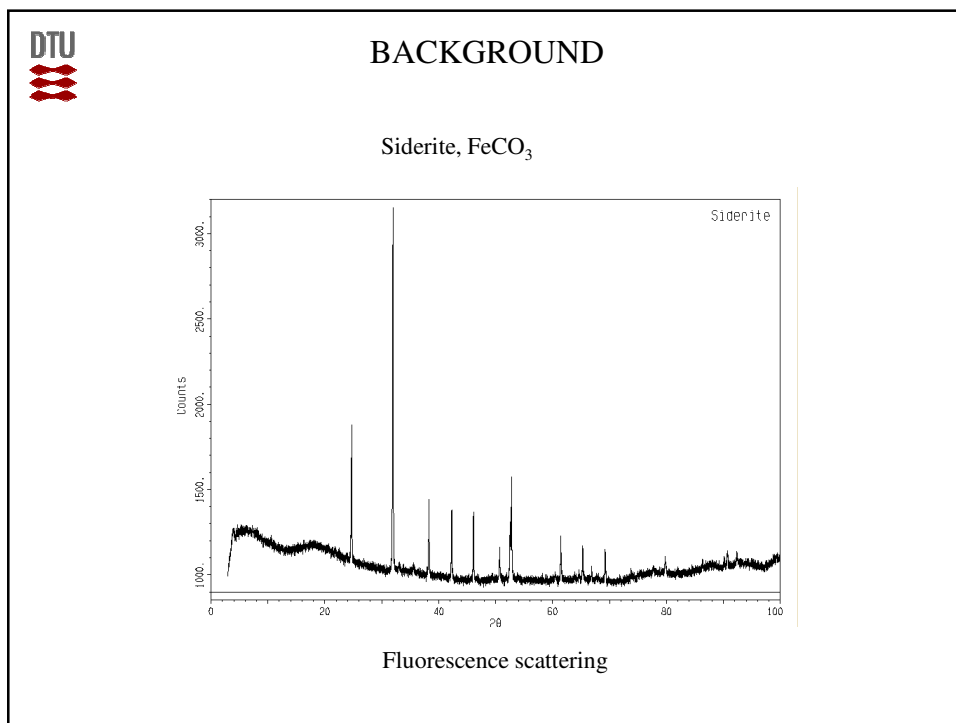
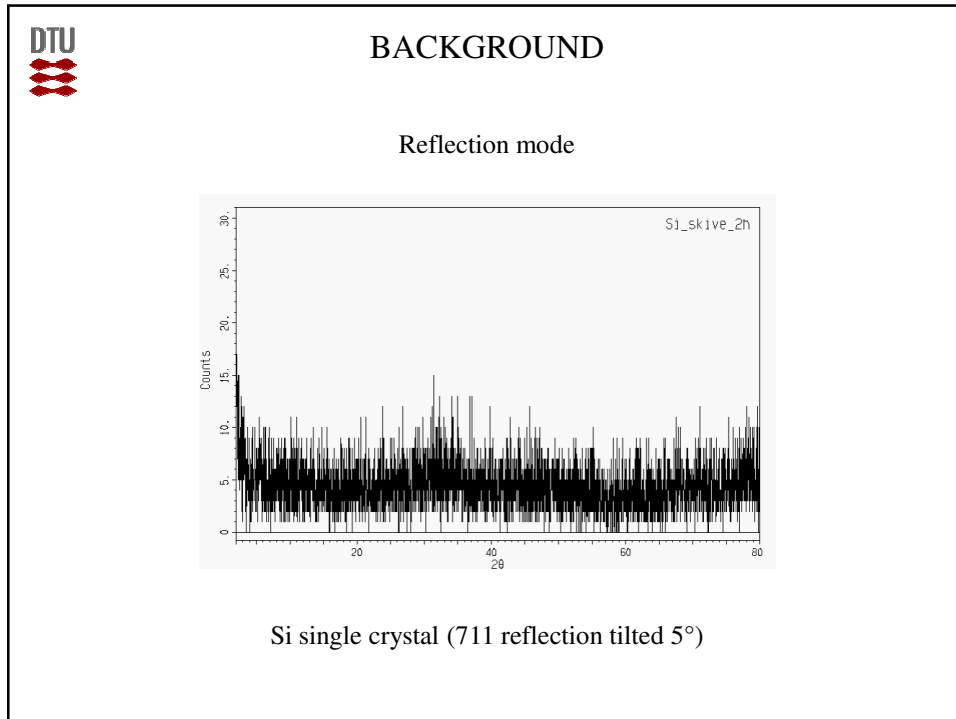


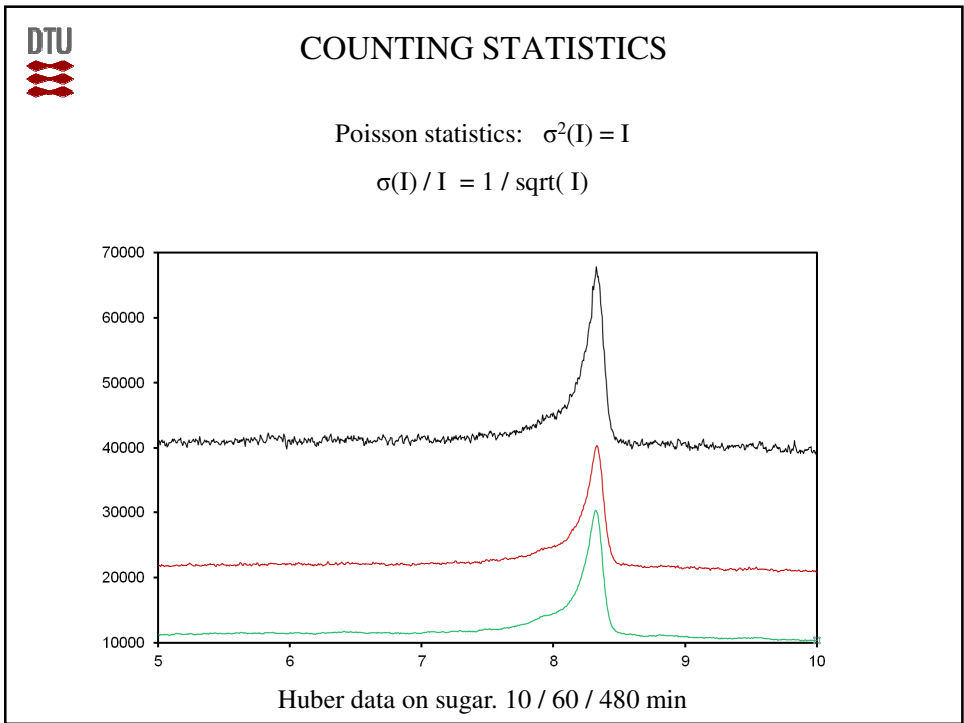
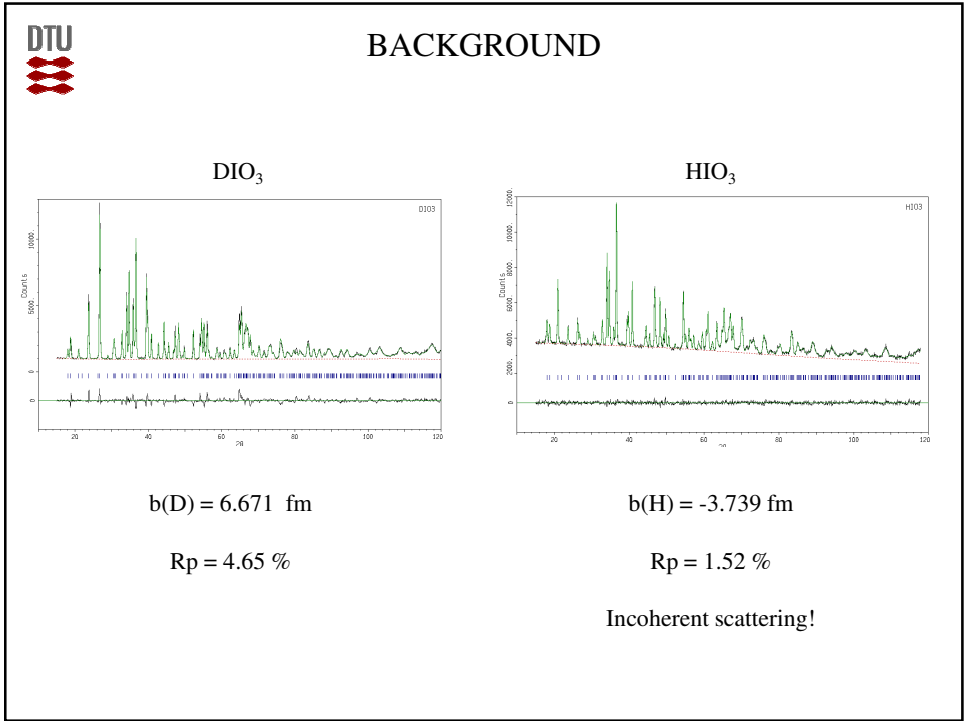
dark

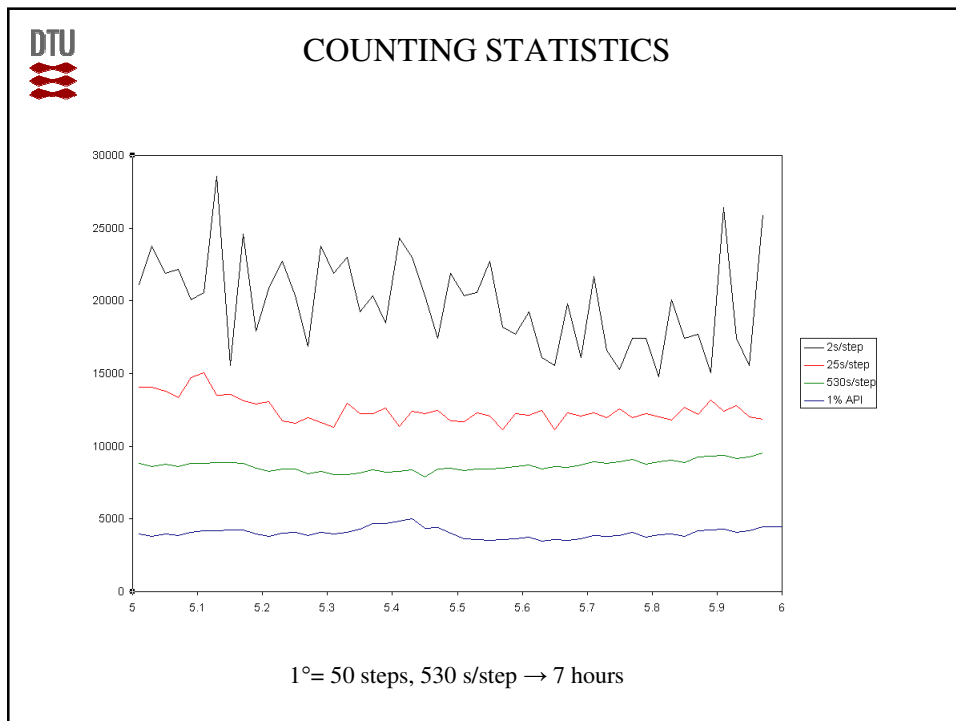
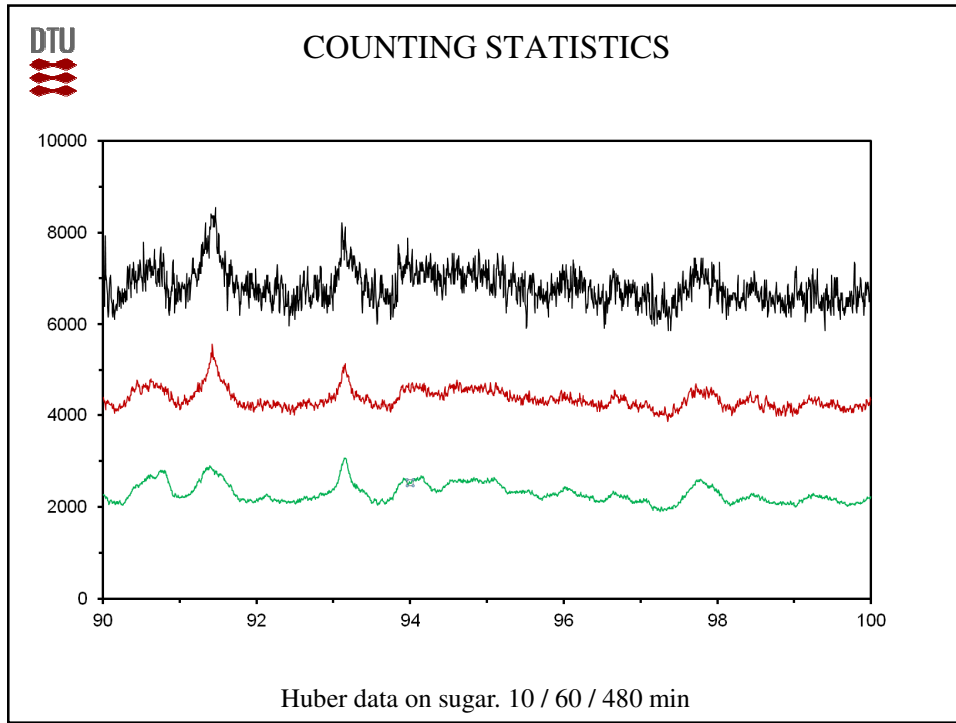


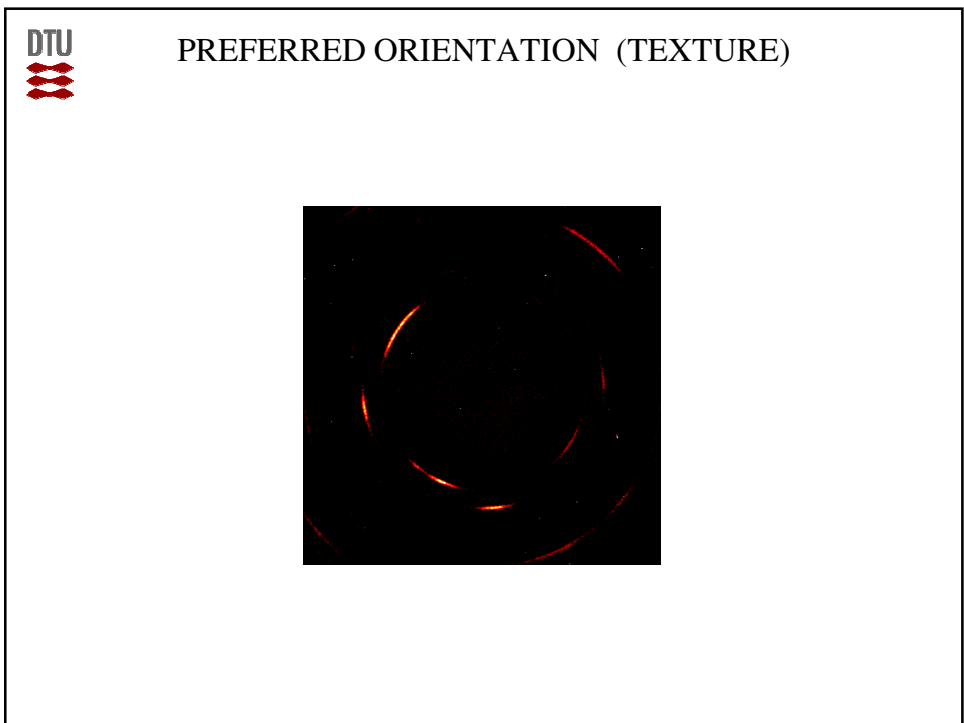
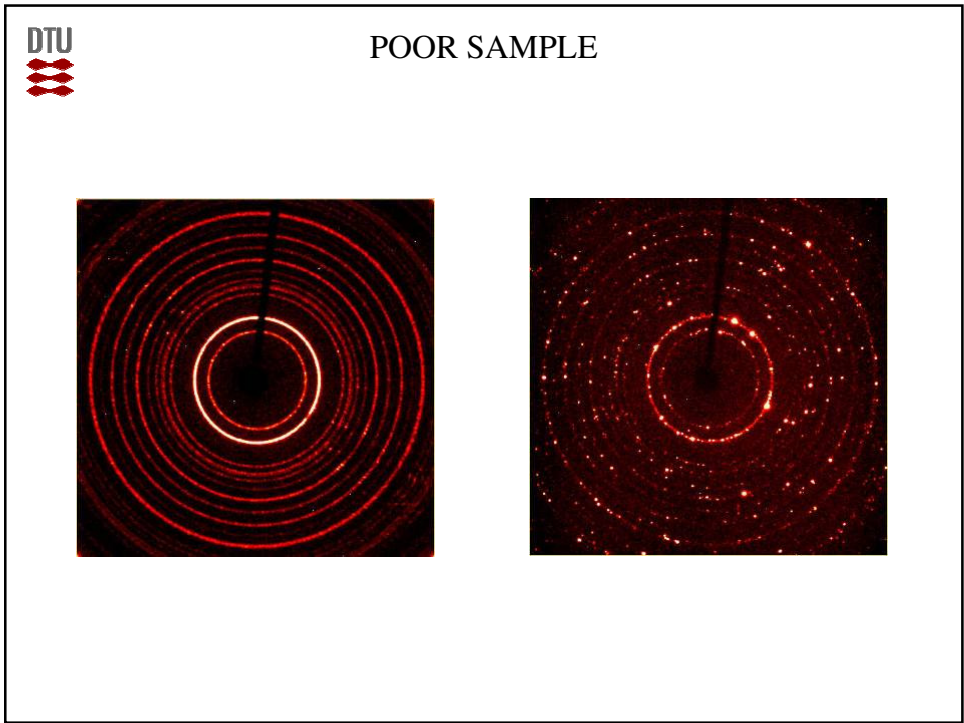
Sample holder (transmission mode)

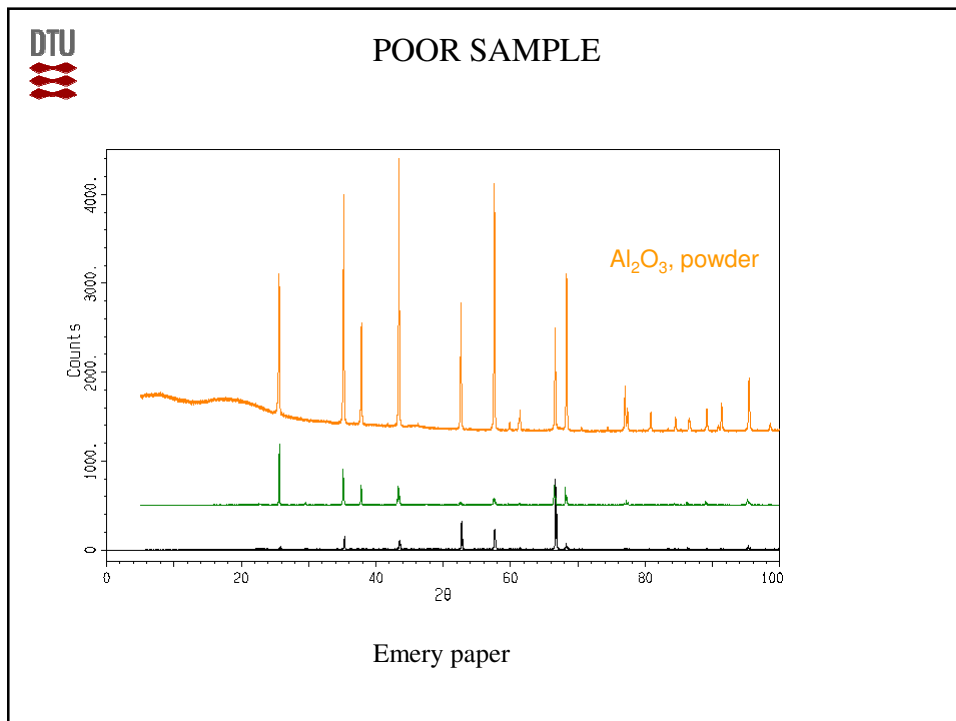
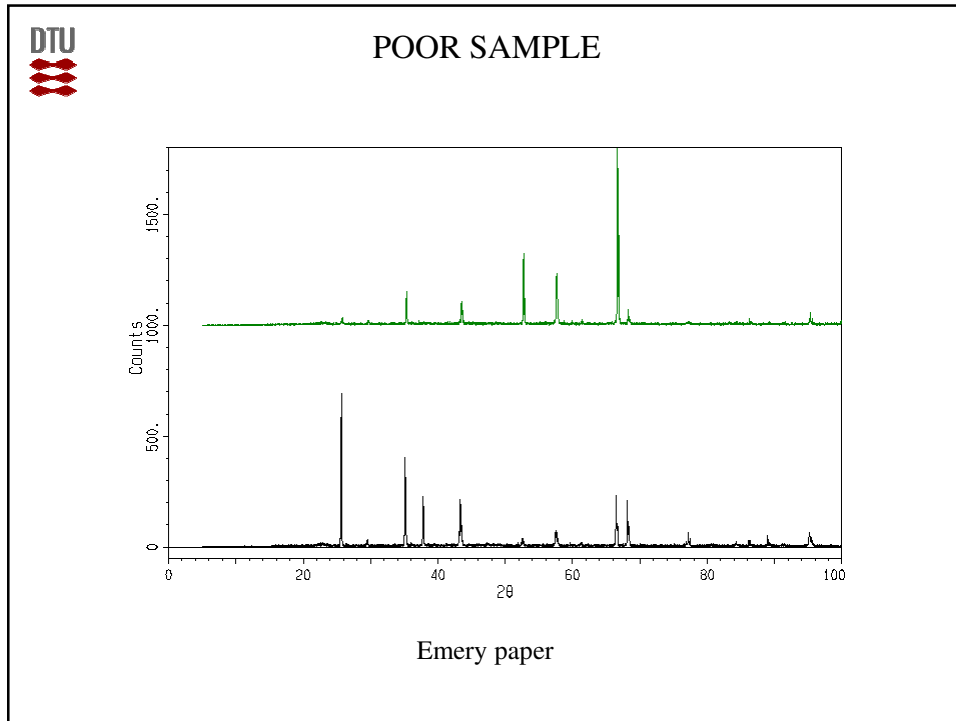


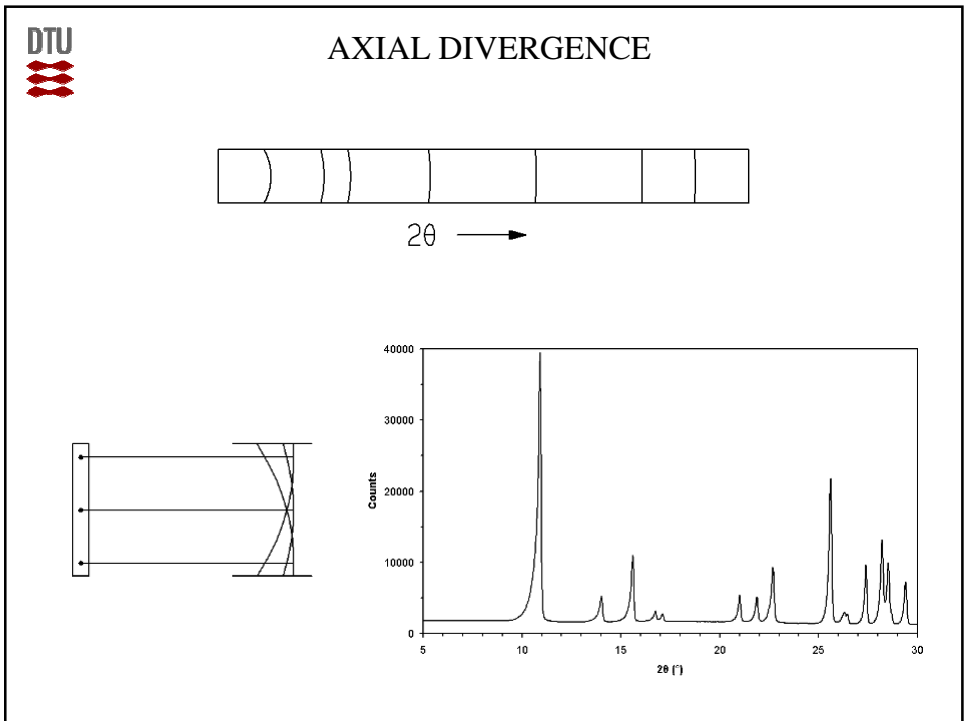
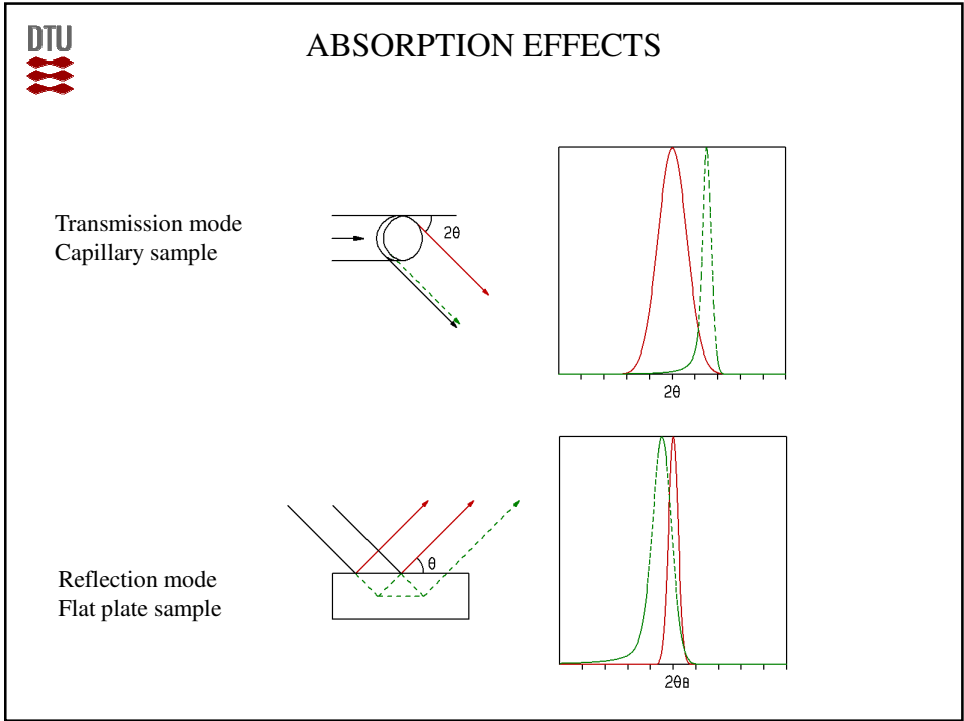














## AXIAL DIVERGENCE

Capillary sample: 1 mm beam height vs. 8 mm beam height

