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Examples of different X-ray diffraction optical configurations to Clay Minerals analysis

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Aim

- The aim of this work is to present results of data collection of clay minerals by XRD obtained using different configurations

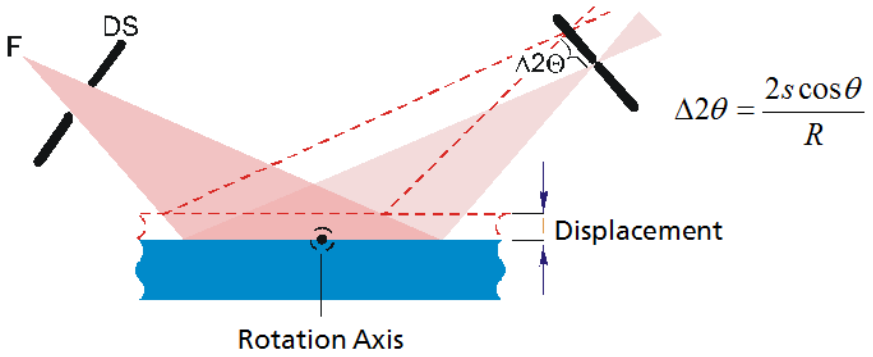
Materials vs Configuration

- Different configuration are suggested by different materials; examples:
  - Mesoporous silica – peaks at around 1˚ 2θ
  - Pharmaceutical – 1-40˚ 2θ
  - Rock forming minerals – 3-75˚ 2θ
  - **Clays – mainly 3-75˚ 2θ**
  - Metals – from 15˚-130˚ 2θ

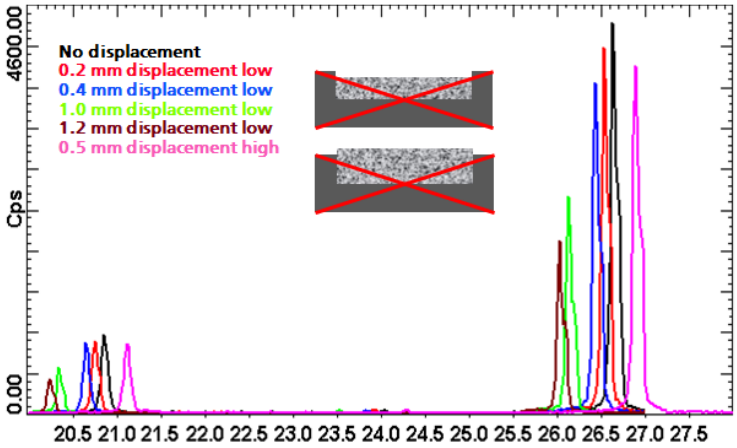
- Sample preparation is important to obtain a good data quality

Sample preparation – (1) Sample height

Sample height

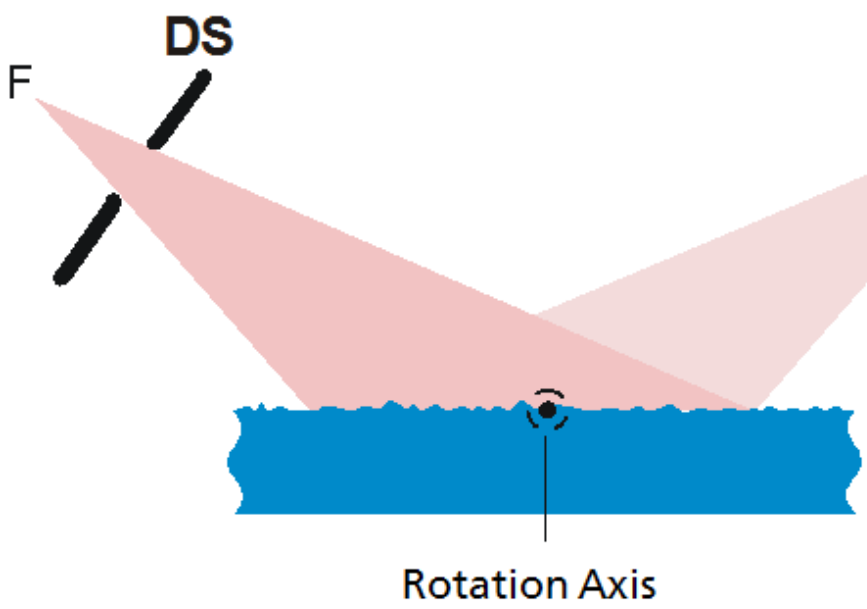


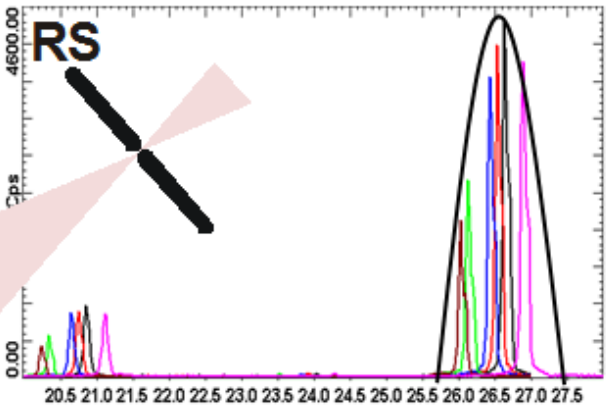
$$\Delta 2\theta = \frac{2s\cos\theta}{R}$$



Sample preparation – (2) Flat surface

- Sample plain surface

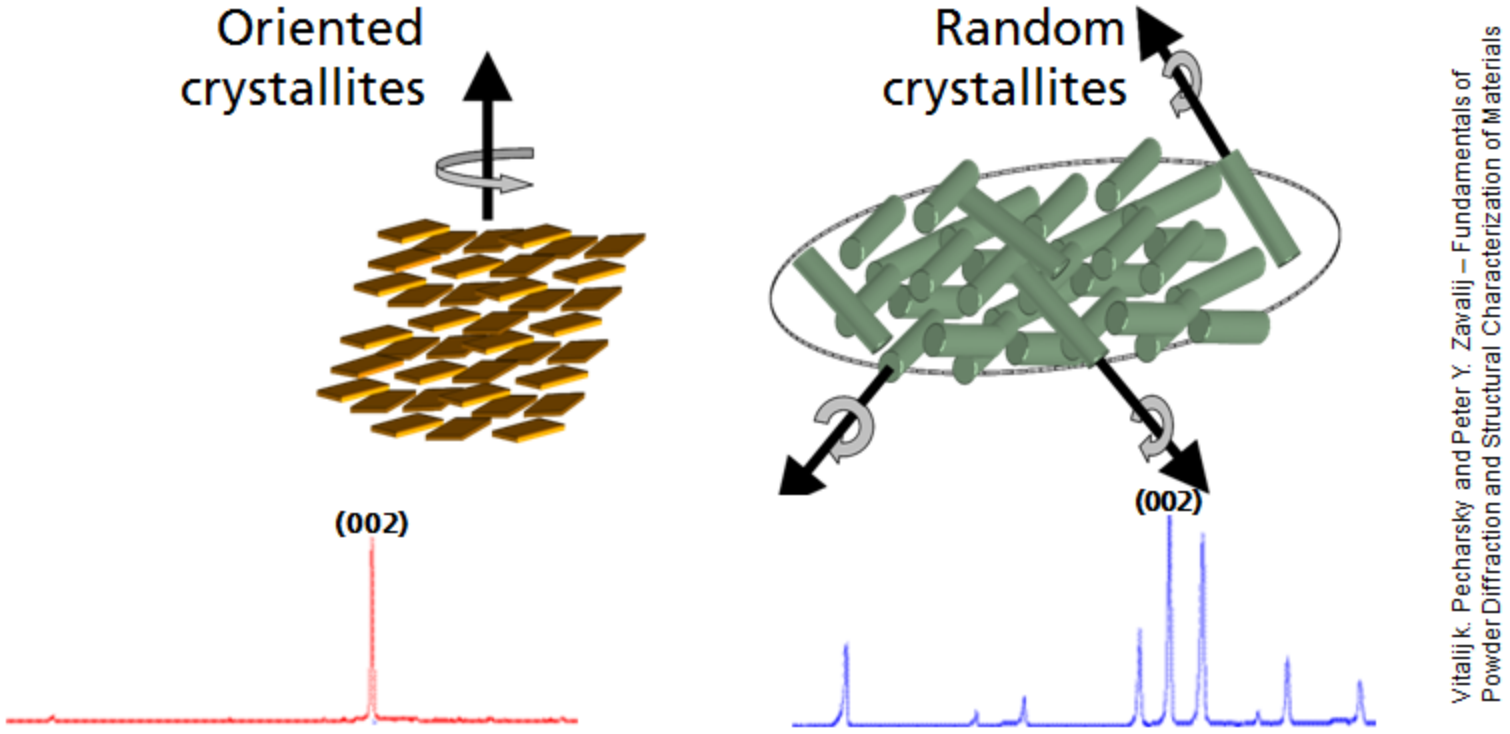




EFECTS:

- **Peak displacement**
- **Peak broadening**

Sample preparation – (3) Preferred orientation



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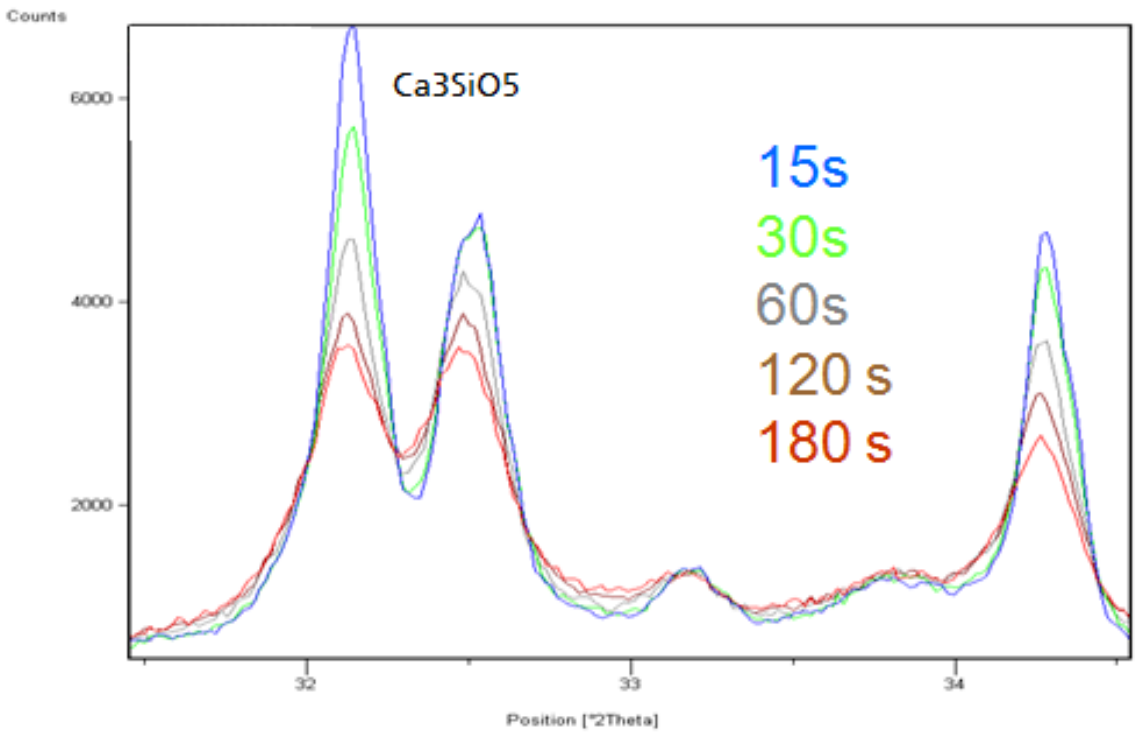
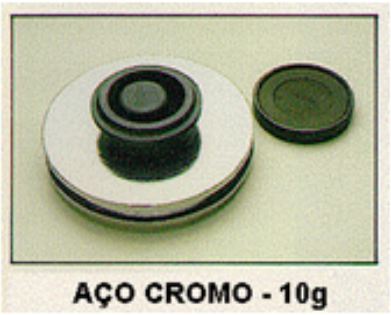
Sample preparation – (4) Grain size

Differences in peak profiles of same sample after different time of milling





AÇO CROMO - 10g

Configurations

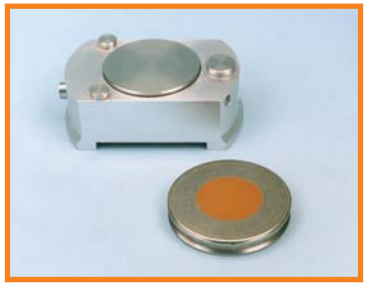
- (1) use of fixed divergent slit settings (1°; ½°; ¼°; 1/8°; 1/16°; 1/32°)
- (2) beam knife
- (3) Soller slits – 0.02 and 0.04 radians
- (4) Active lengh (detector)
- (5) goniometer radius of 240 and145mm radius in a benchtop
- (6) capillary and transmission mode

Sample preparation

- Samples enriched in Illite, Sepiolite, Nacrite, Montmorillonite, Attapulgite, Kaolinite, Beidellite, Halloysite and Dickite were used

Sample preparation

- Samples were all prepared in a backloading kit

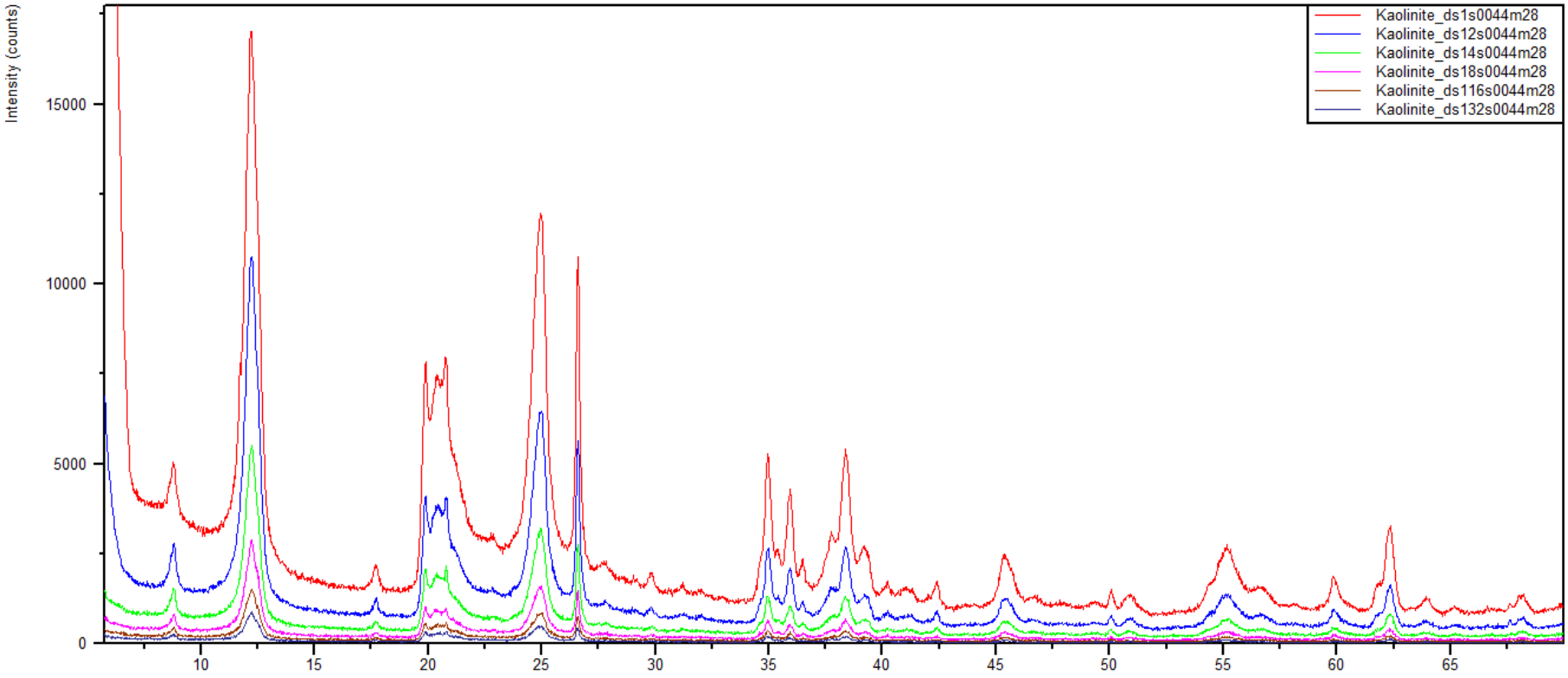

- Or in a capillary

(1) Different divergent slit sizes

- Fixed divergent slits => 1°; ½°; ¼°; 1/8°; 1/16°; 1/32°
- Soller slit => 0.04 rad
- Beam knife
- Total time => 4’28’’

1°and ½° => high direct beam effect => INTENSITY

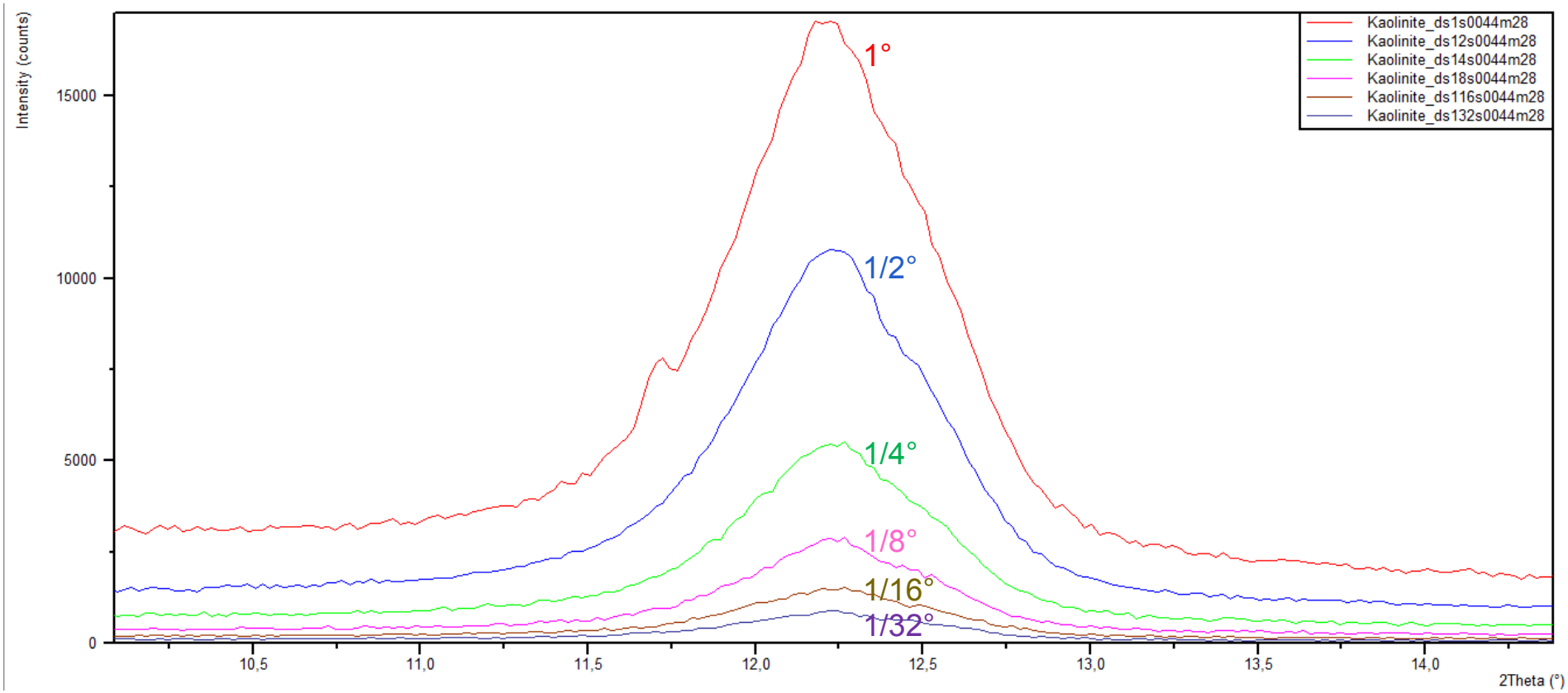
1/32°and 1/16° => much lower intensities => RESOLUTION



• By using a bigger divergence slit opening, the irradiated area is increased as well as the number of grain orientations that can participate to the diffracted signal.

• However, below 20°, the 1° divergence slit irradiate a larger area, more than the sample size, leading to an increase of the background intensity.

• As a consequence, the gain factor in intensity at low angle is reduced compared to the one at higher angle.


Intensity (counts)
15000
10000
5000
0
10,5
11,0
11,5
12,0
12,5
13,0
13,5
14,0
2Theta (°)
Kaolinite_ds1s0044m28
Kaolinite_ds12s0044m28
Kaolinite_ds14s0044m28
Kaolinite_ds18s0044m28
Kaolinite_ds116s0044m28
Kaolinite_ds132s0044m28
1°
1/2°
1/4°
1/8°
1/16°
1/32°

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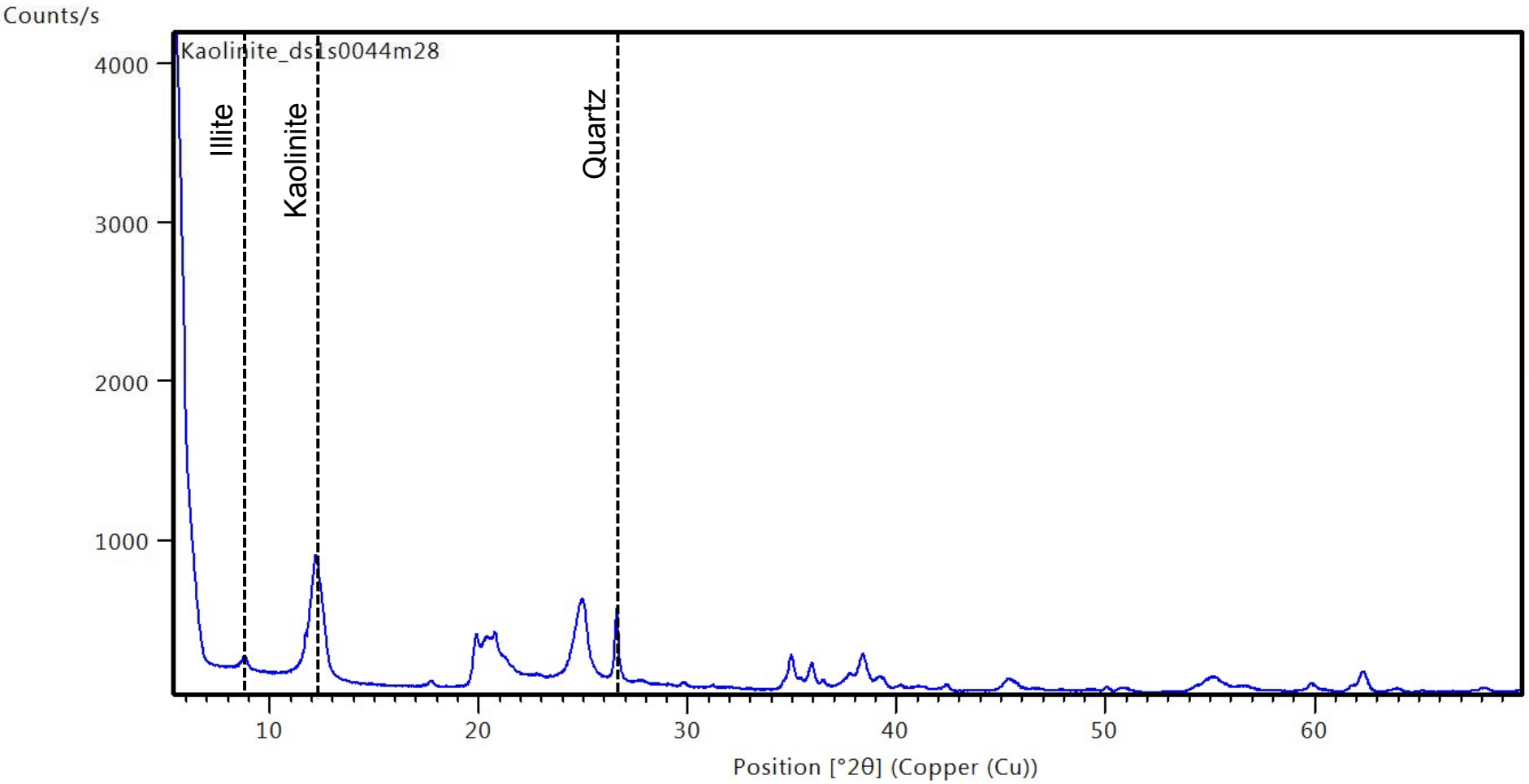

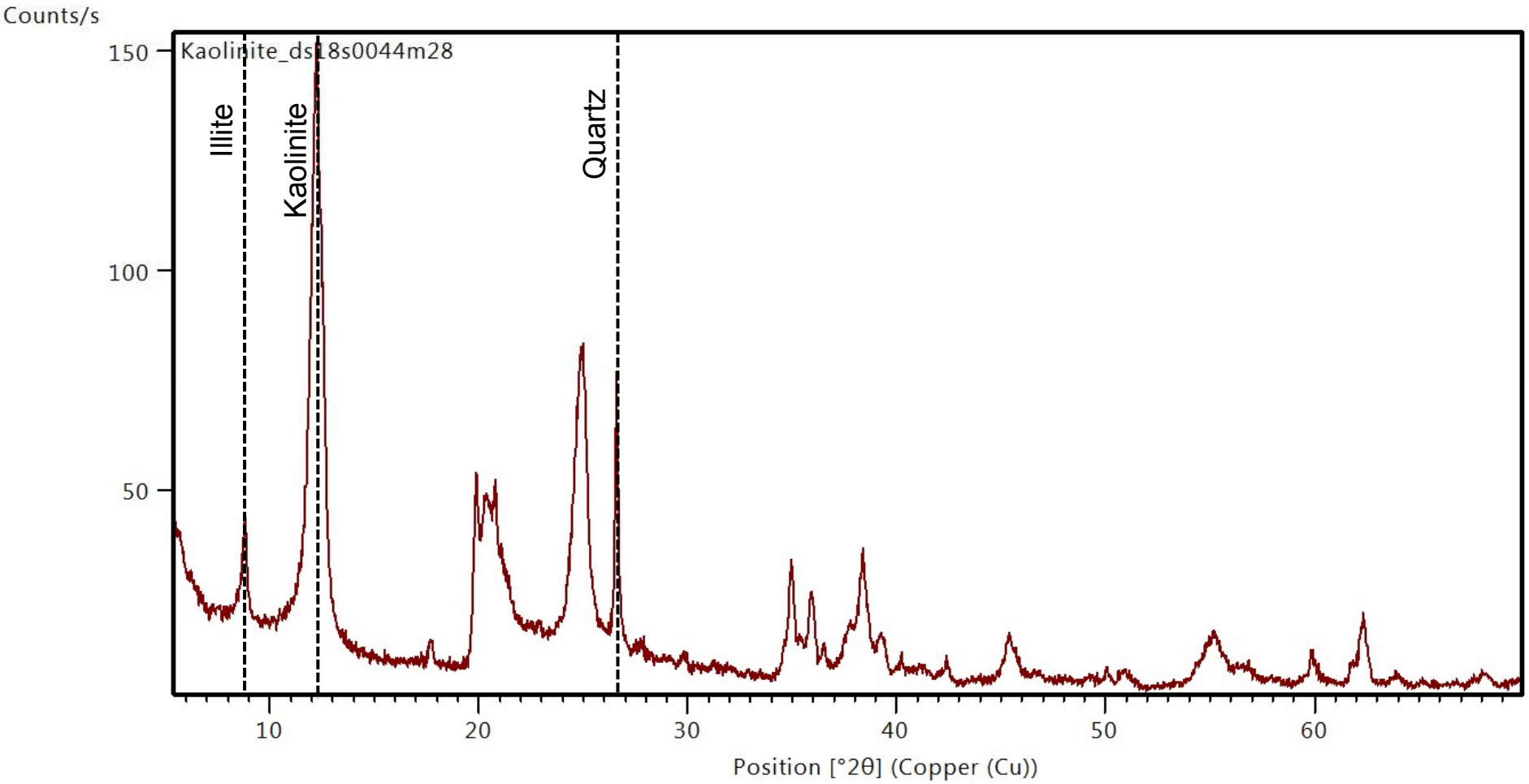
Counts/s
150
100
50
Kaolinite_ds18s0044m28
Illite
Kaolinite
Quartz
10
20
30
40
50
60
Position [°2θ] (Copper (Cu))

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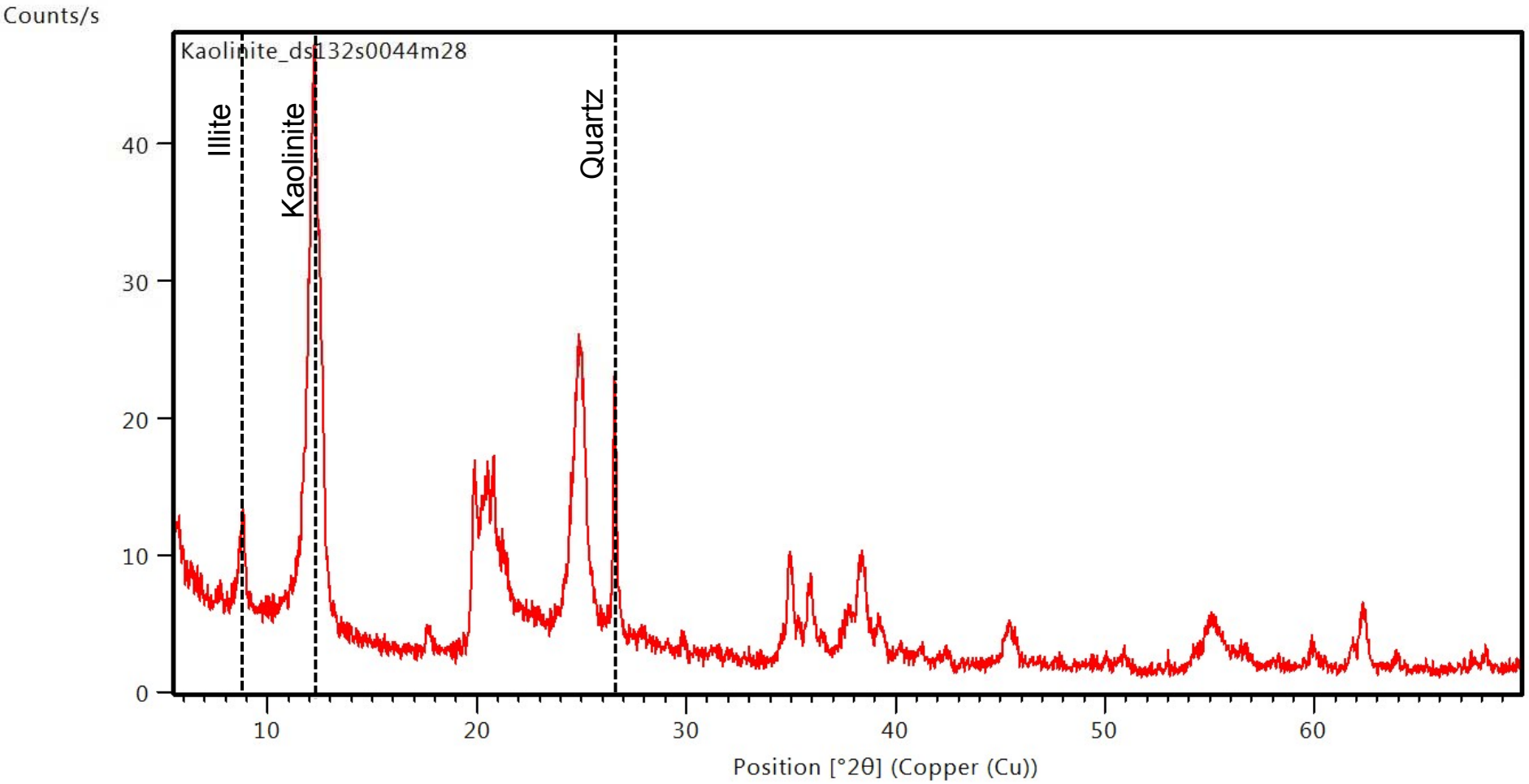
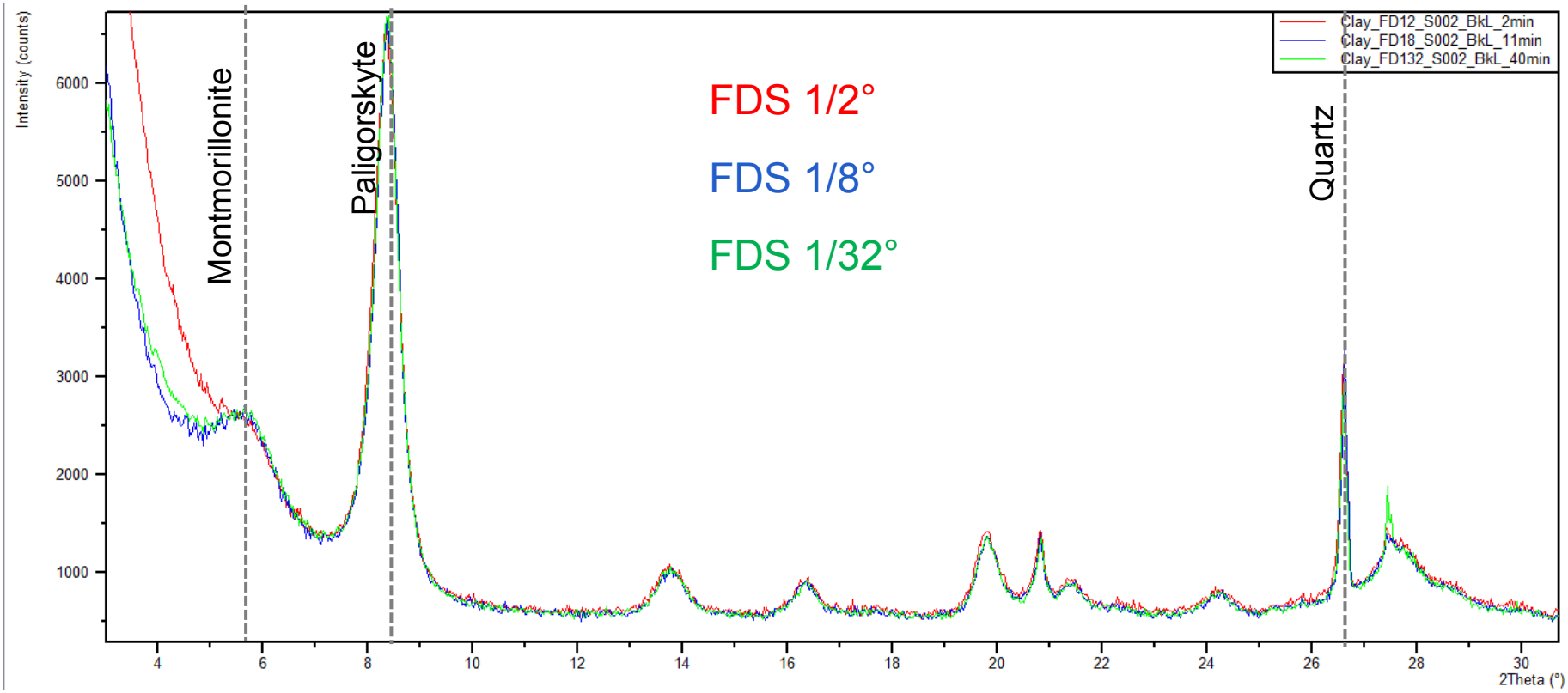
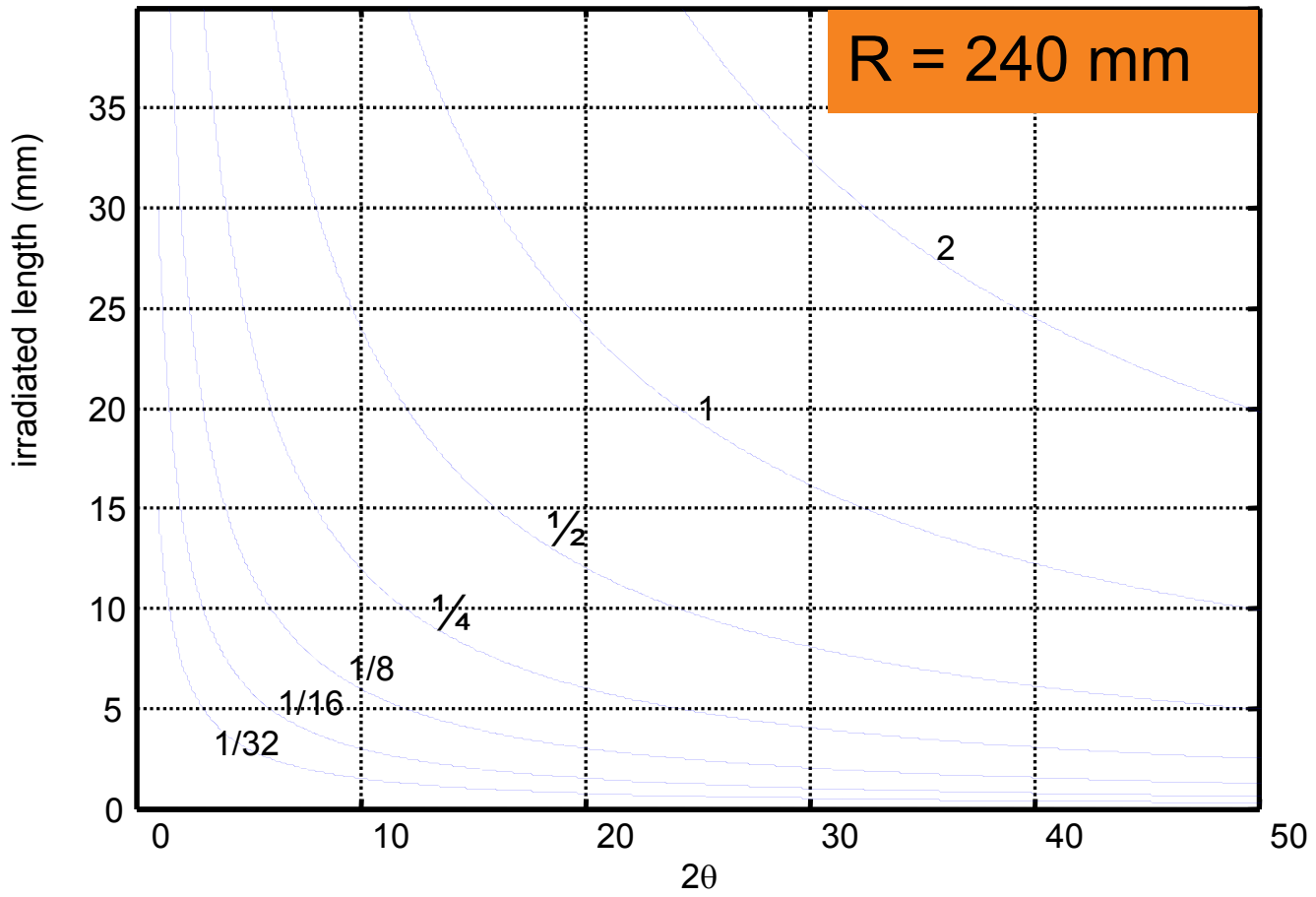


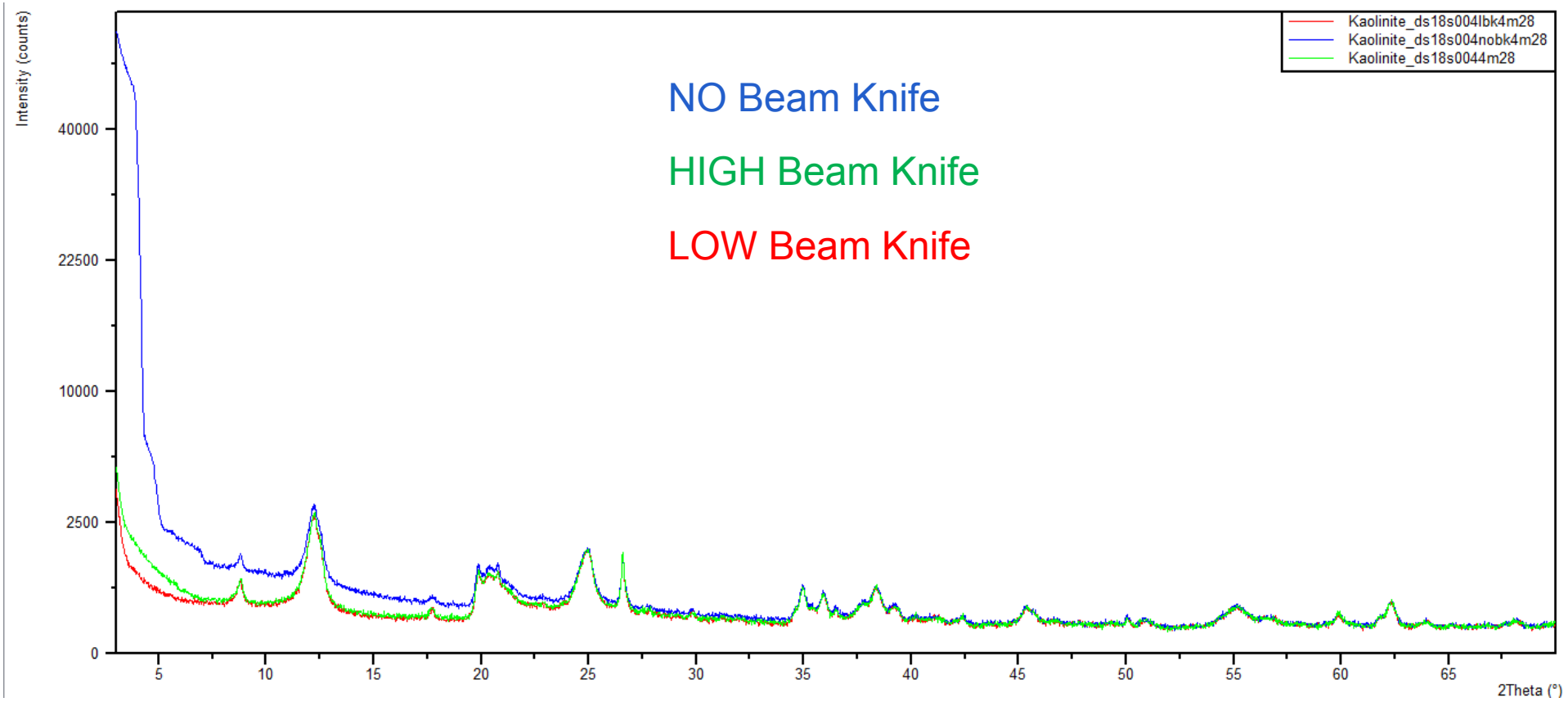
Goniometer radius => 145 mm

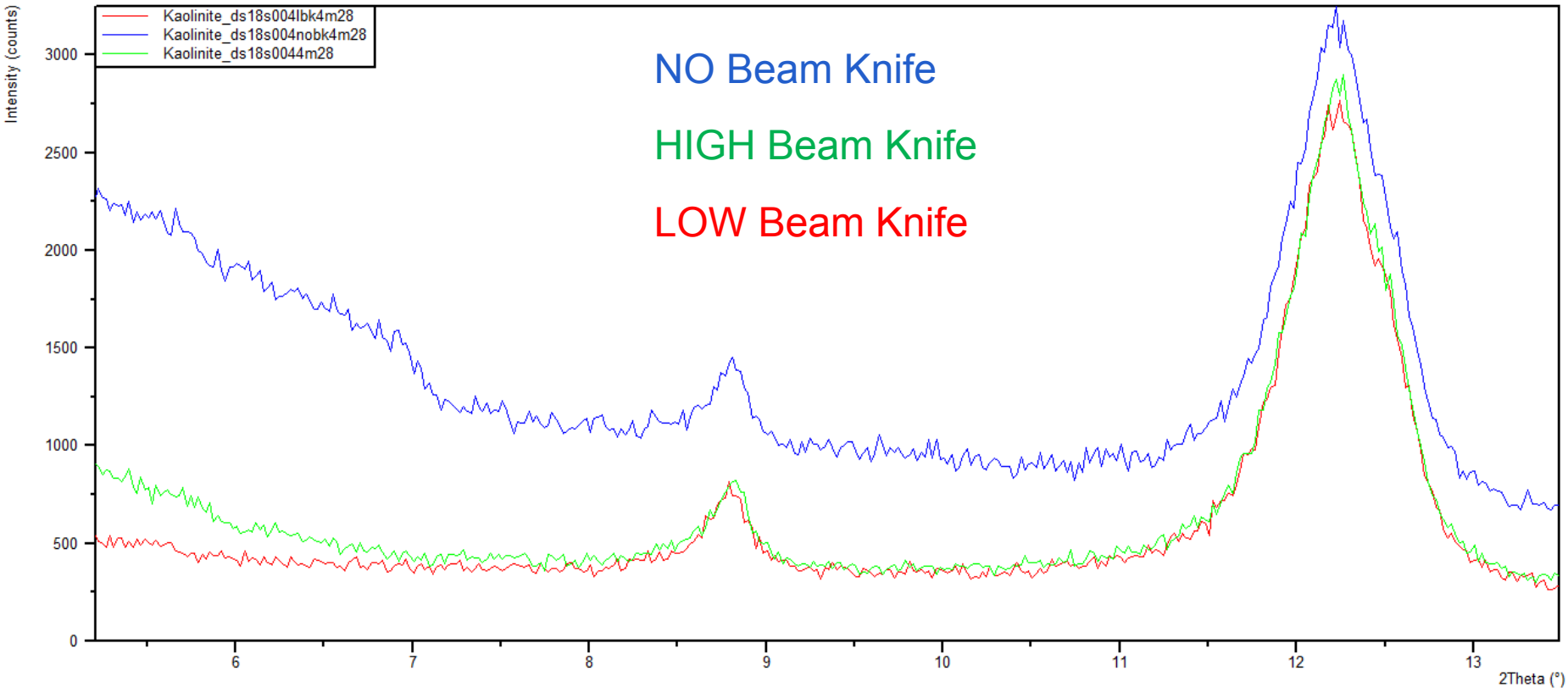


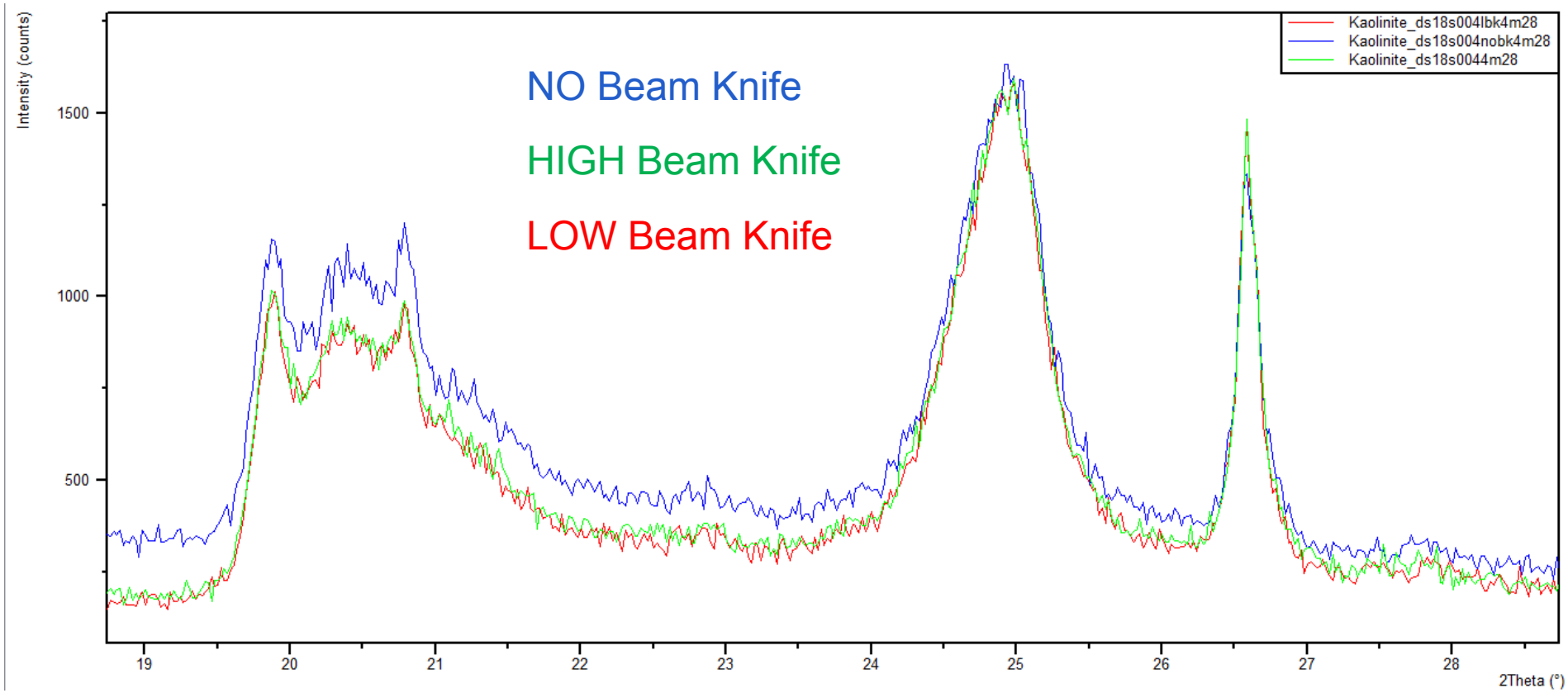

R = 240 mm
irradiated length (mm)
35
30
25
20
15
10
5
0
0
10
20
30
40
50
2θ
2
1
1/2
1/4
1/8
1/16
1/32

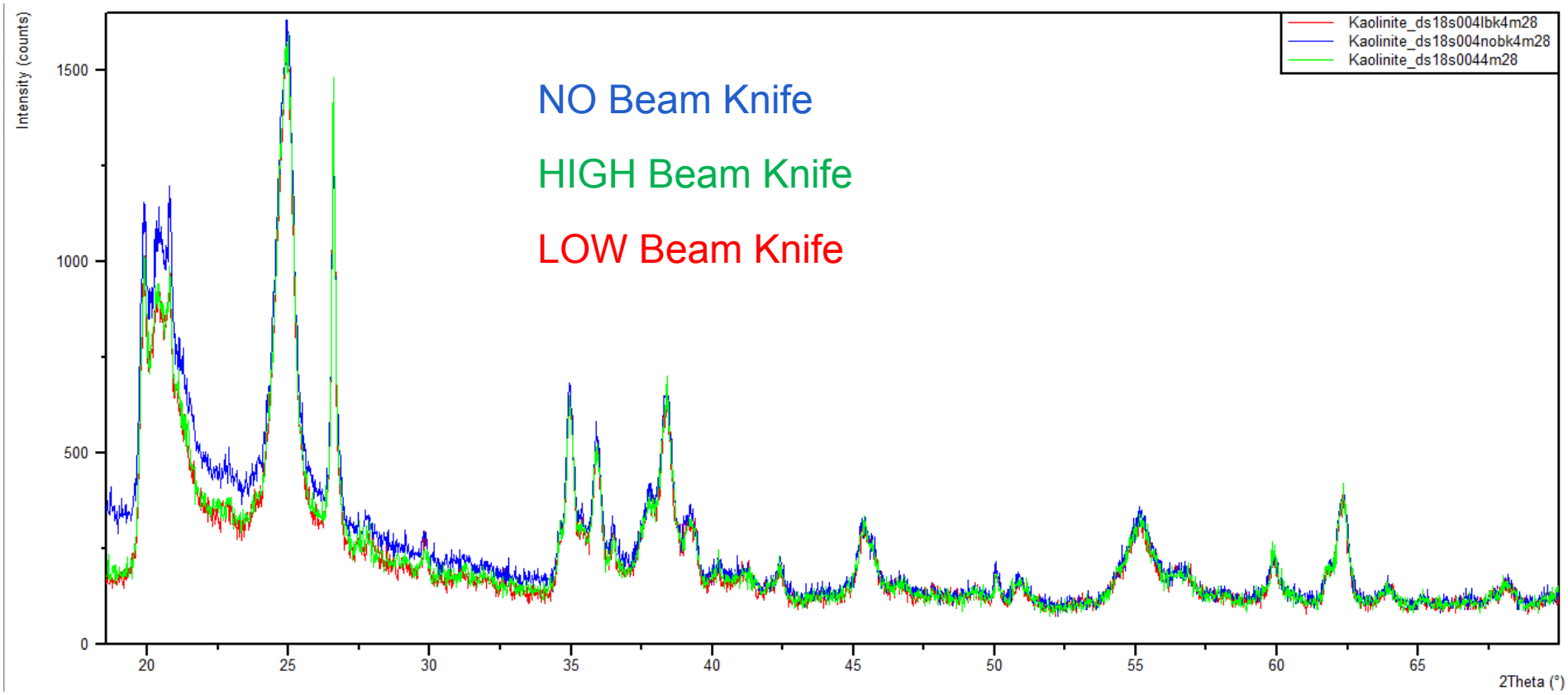
(2) Use of beam knife

- Fixed divergent slit => 1/8°
- Soller slit => 0.04 rad
- Total time => 4’28’’


Kaolinite_ds18s004lbk4m28
Kaolinite_ds18s004nobk4m28
Kaolinite_ds18s0044m28
NO Beam Knife
HIGH Beam Knife
LOW Beam Knife
Intensity (counts)
40000
22500
10000
2500
0
5
10
15
20
25
30
35
40
45
50
55
60
65
2Theta (°)


Kaolinite_ds18s004lbk4m28
Kaolinite_ds18s004nobk4m28
Kaolinite_ds18s0044m28
NO Beam Knife
HIGH Beam Knife
LOW Beam Knife
Intensity (counts)
3000
2500
2000
1500
1000
500
0
6
7
8
9
10
11
12
13
2Theta (°)


Intensity (counts)
1500
1000
500
19
20
21
22
23
24
25
26
27
28
2Theta (°)
Kaolinite_ds18s004lbk4m28
Kaolinite_ds18s004nobk4m28
Kaolinite_ds18s0044m28
NO Beam Knife
HIGH Beam Knife
LOW Beam Knife

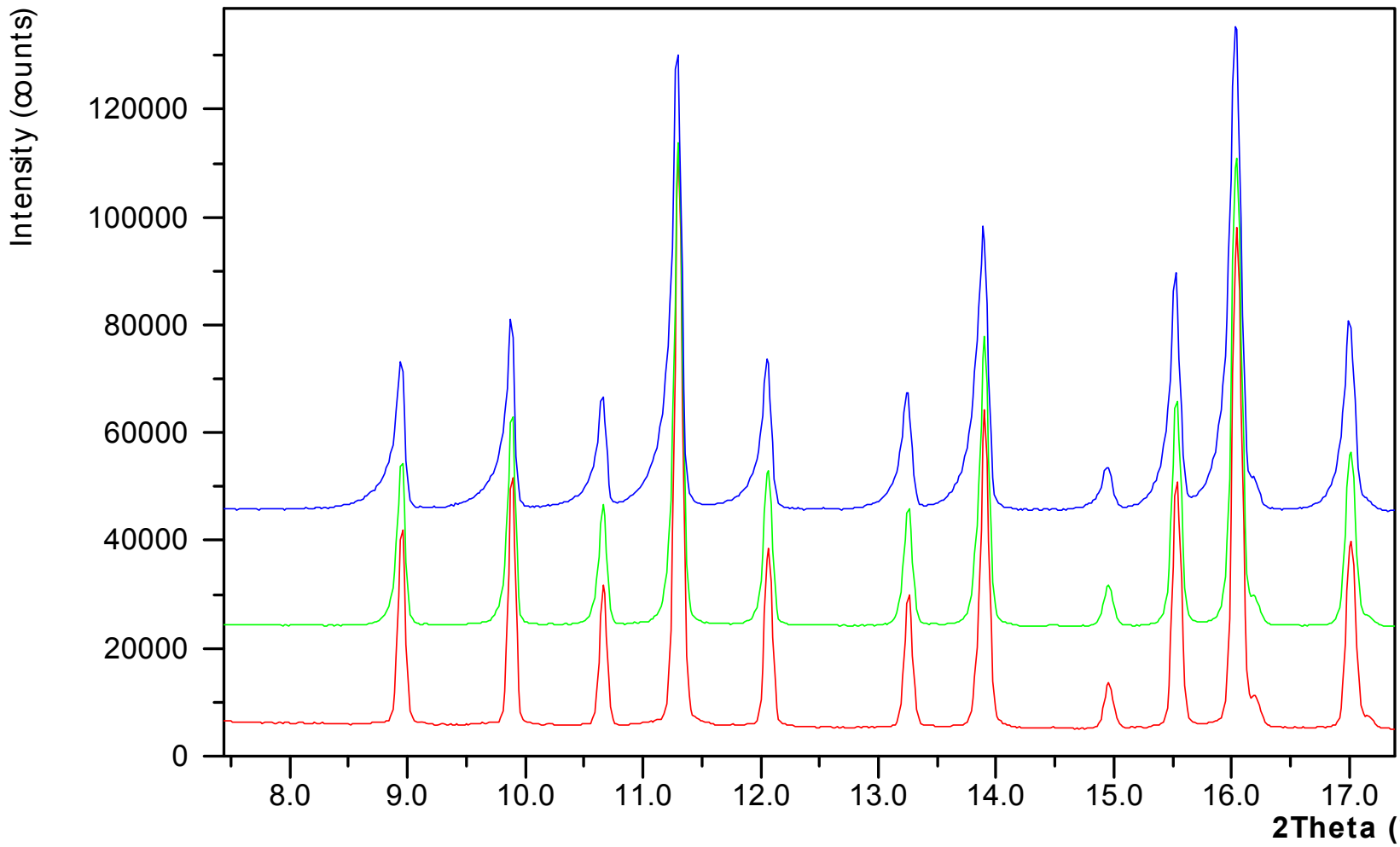

Kaolinite_ds18s004lbk4m28
Kaolinite_ds18s004nobk4m28
Kaolinite_ds18s0044m28
NO Beam Knife
HIGH Beam Knife
LOW Beam Knife
Intensity (counts)
1500
1000
500
0
20
25
30
35
40
45
50
55
60
65
2Theta (°)

(3) Soller slits

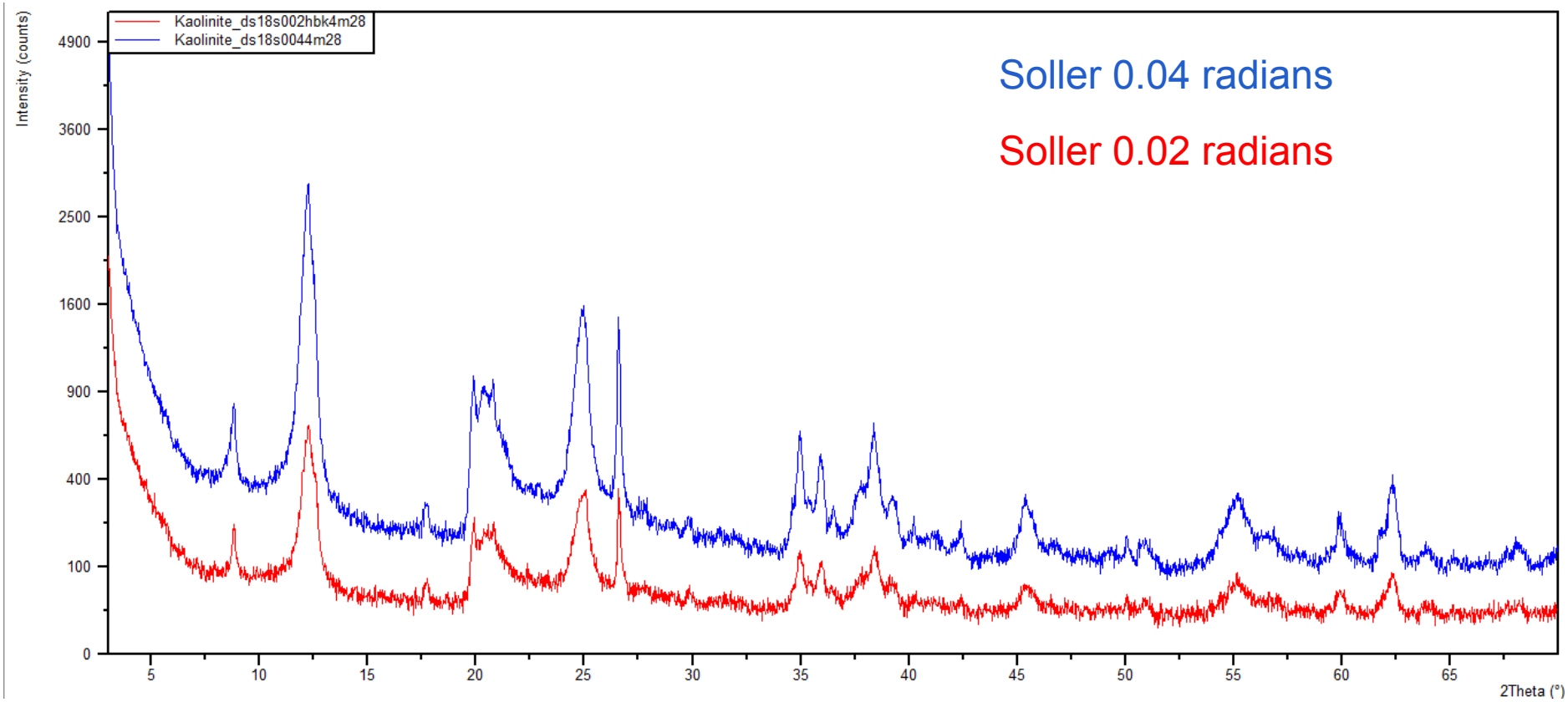
- 0.04 radians
- 0.02 radians
- 0.01 radians

Relationship between mask's marking and irradiated width in millimeters for the fixed divergence slit

Mask marking	Soller Slits		
	0.01 rad	0.02 rad	0.04 rad
20	18.4	19.9	22.8
15	13.4	14.9	17.8
10	8.4	9.9	12.8
5	3.4	4.9	7.8


Intensity (counts)
120000
100000
80000
60000
40000
20000
0
8.0
9.0
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
2Theta (°)

Goniometer radius => 145 mm



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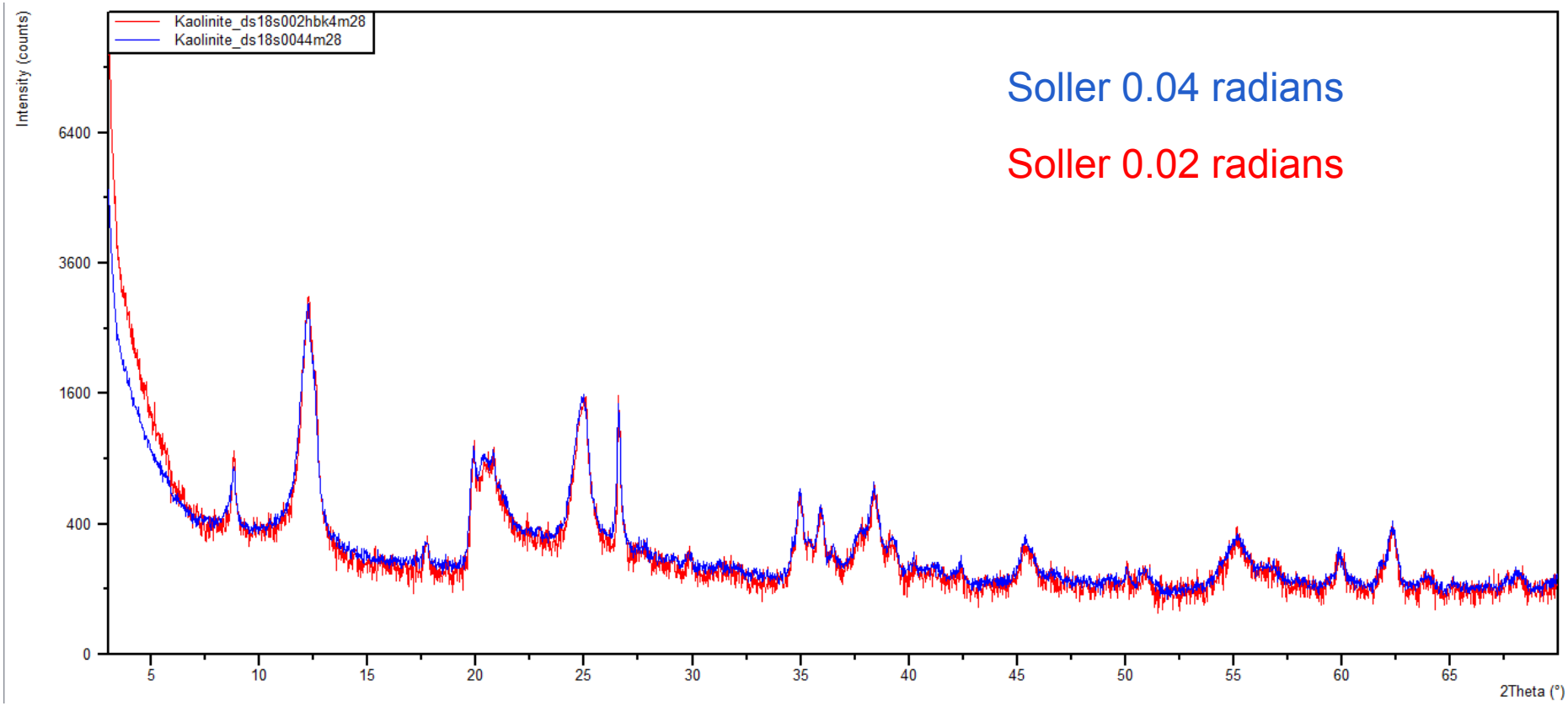
Goniometer radius => 145 mm



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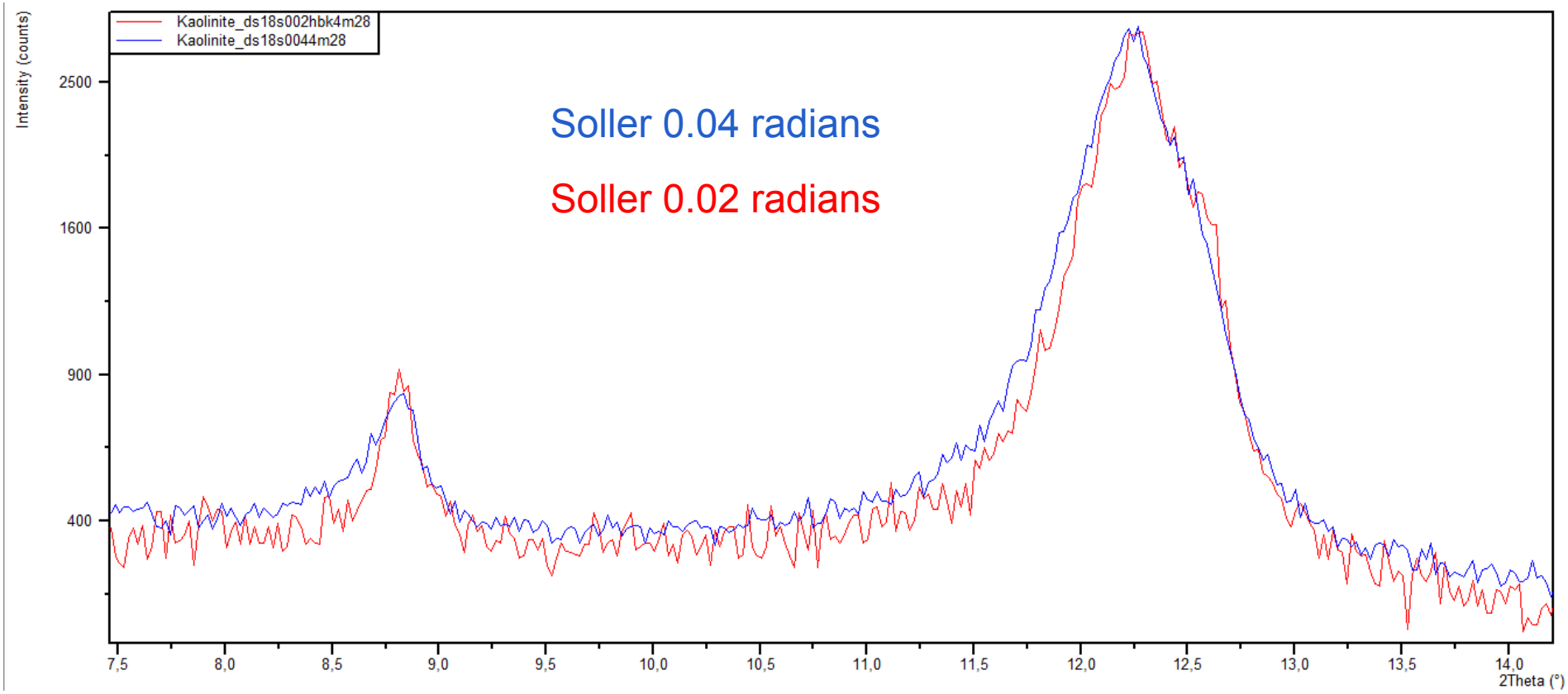
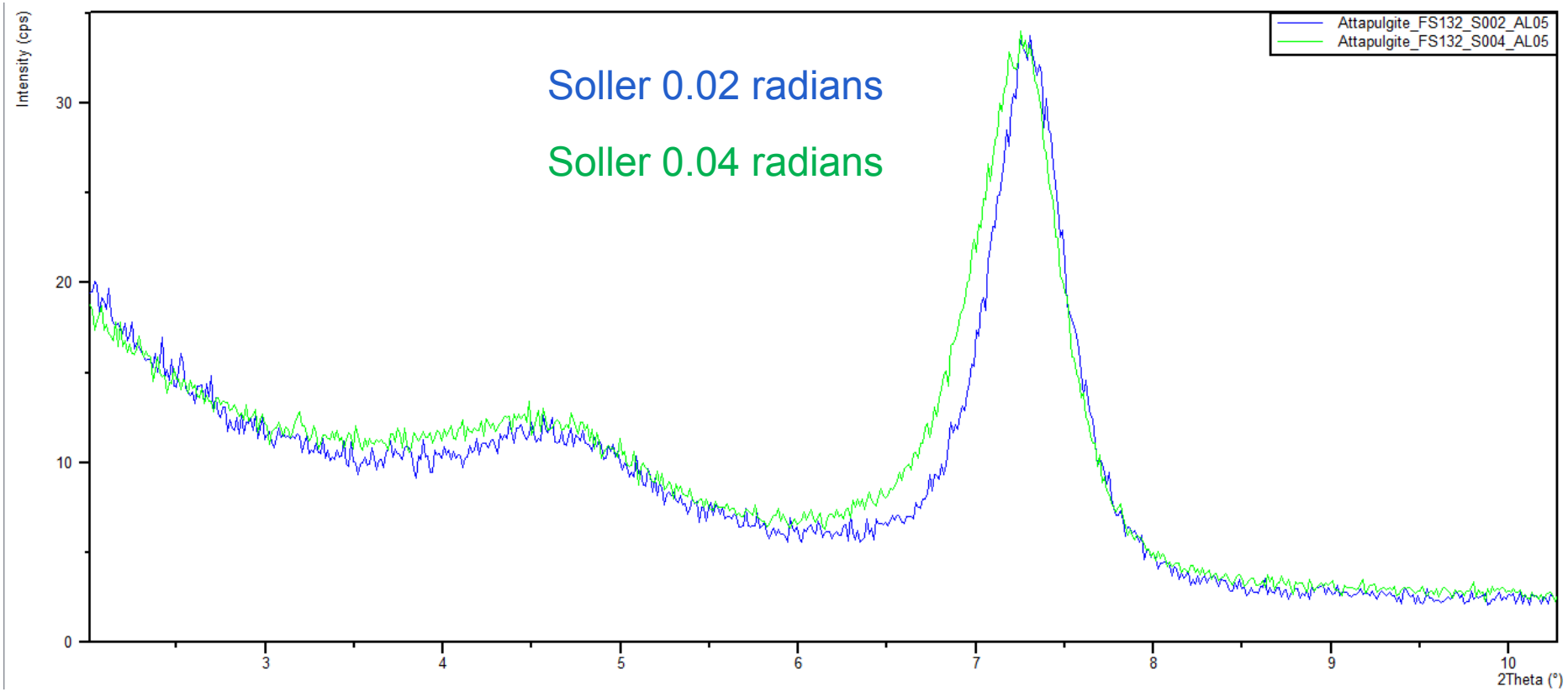
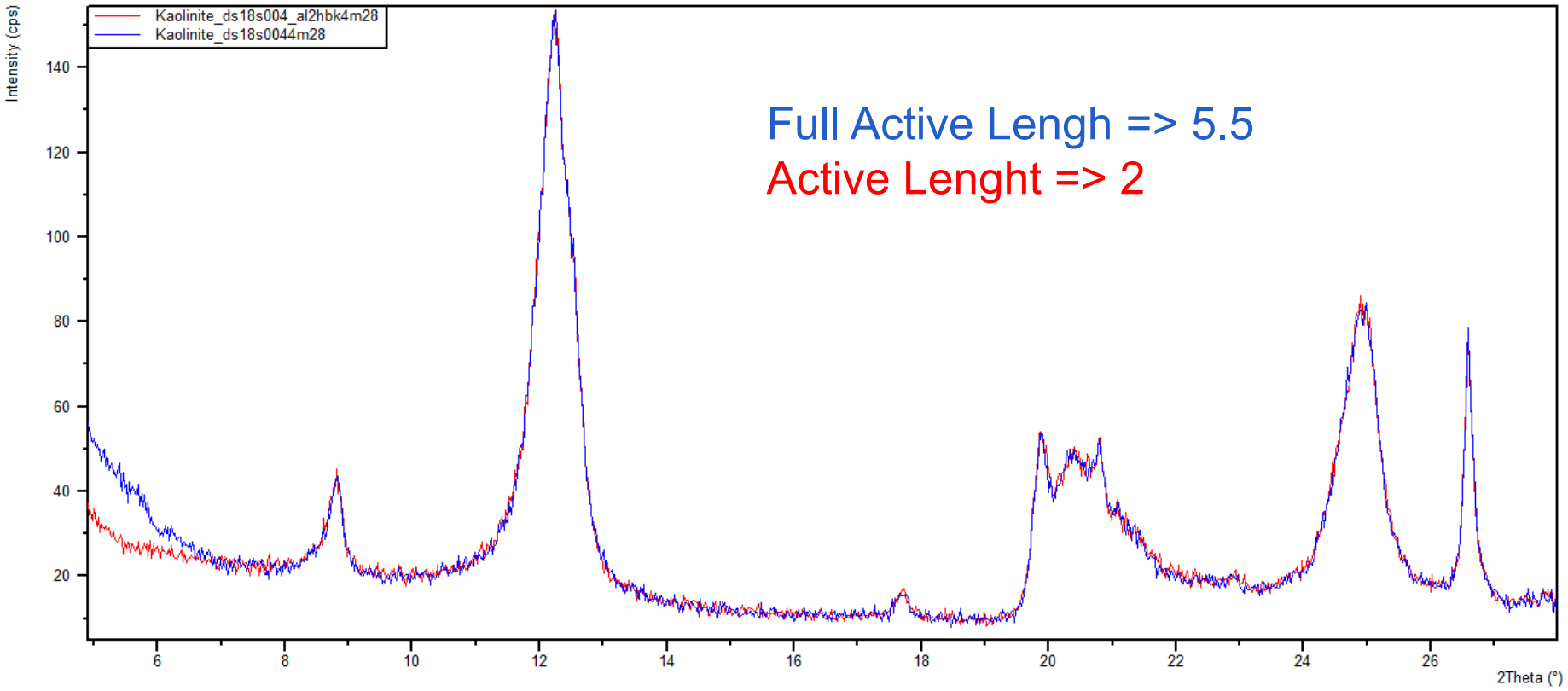
Goniometer radius => 145 mm



Goniometer radius => 240 mm

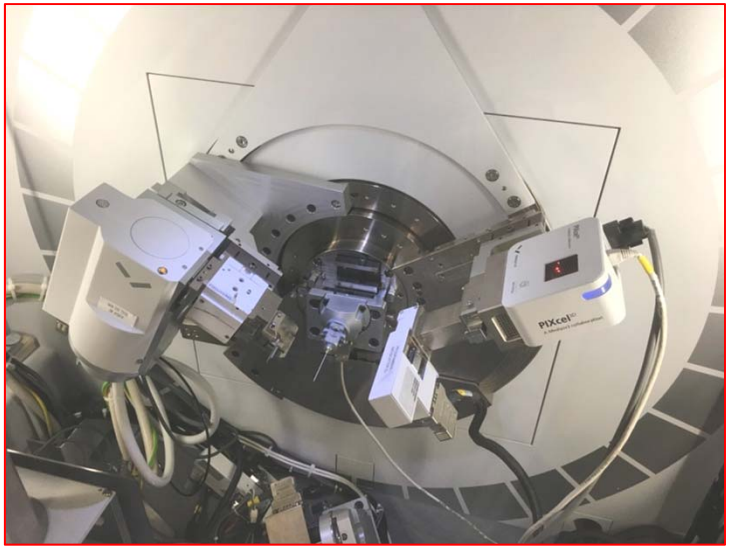


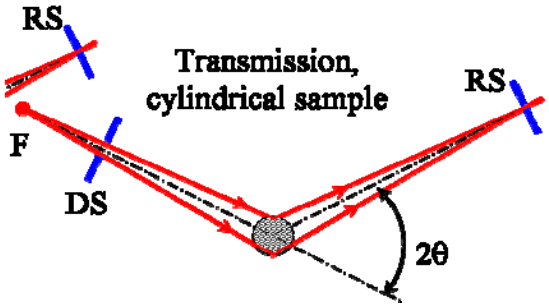
(4) Active Lenght



(5) Cappilary spinner and transmission mode

Glass capillary => ø 0.7 mm





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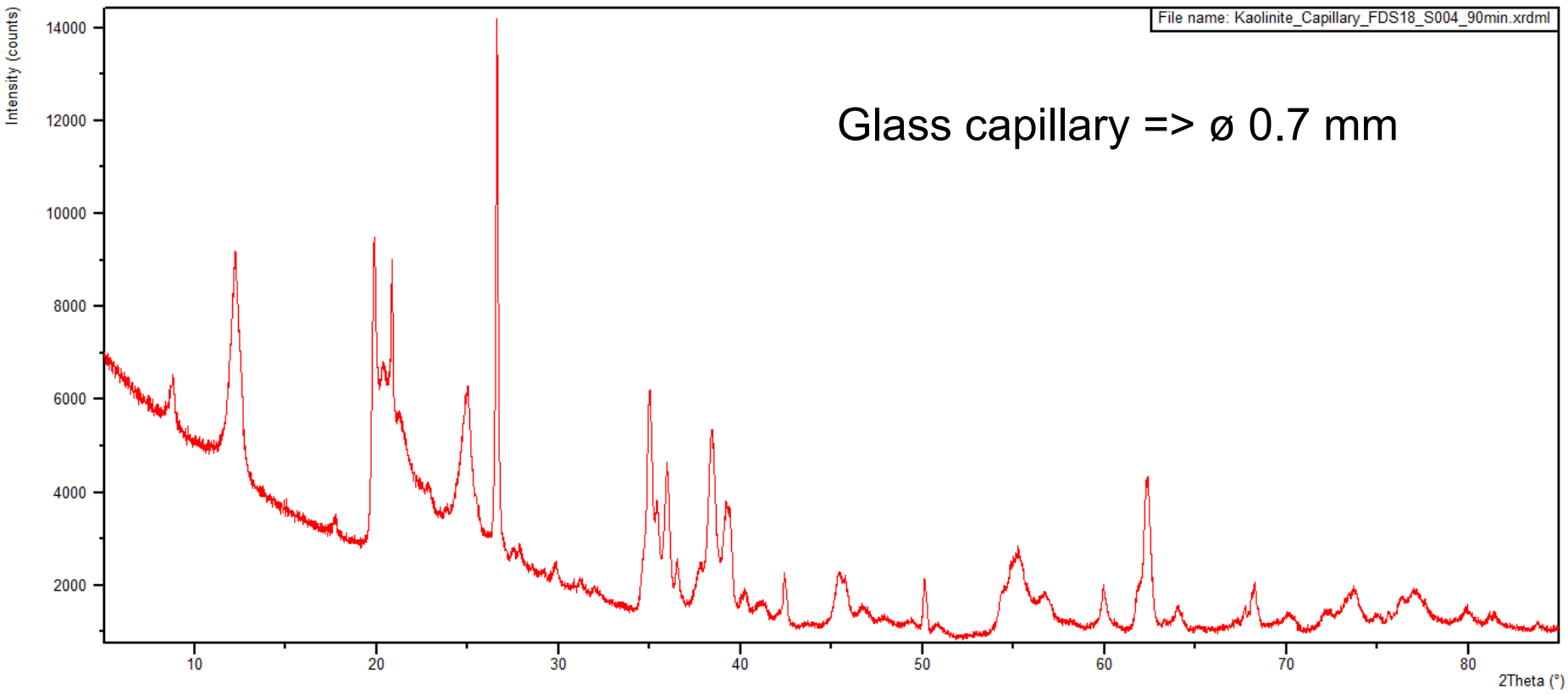
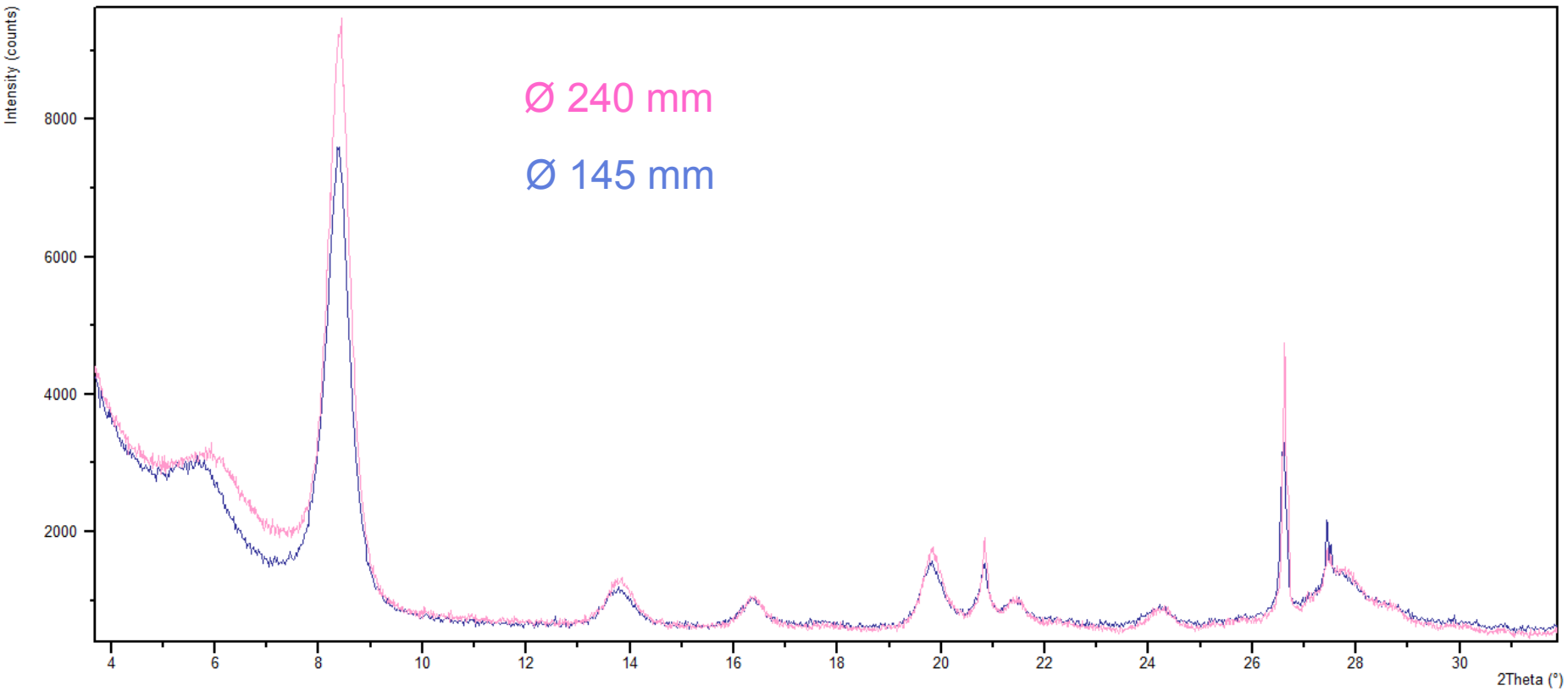

File name: Kaolinite_Capillary_FDS18_S004_90min.xrdml
Glass capillary => ø 0.7 mm
Intensity (counts)
14000
12000
10000
8000
6000
4000
2000
10
20
30
40
50
60
70
80
2Theta (°)

(6) Goniometer Radius

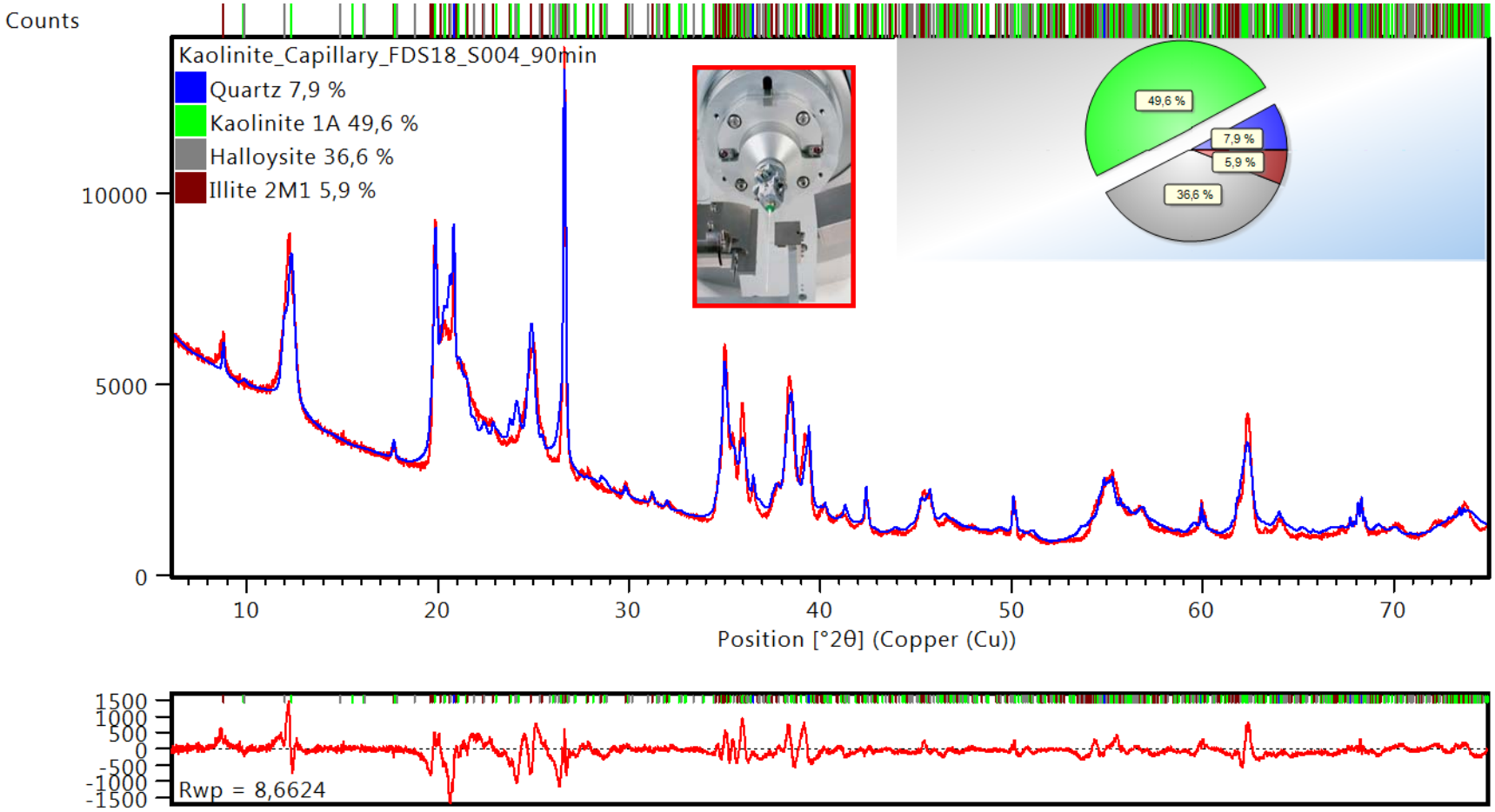
Ø 240 mm

A	Configuration	B
240 mm	**Goniometer radius**	145 mm
Empyrean	**Tube**	Empyrean
1/32°	**Fixed divergent slit**	1/32°
0.02 rad	**Soller slit**	0.02 rad
Pixcel 3D	**Detector**	Pixcel 3D
3.3°	**Active Lenght**	5.5°
1800 W	**Power**	600 W

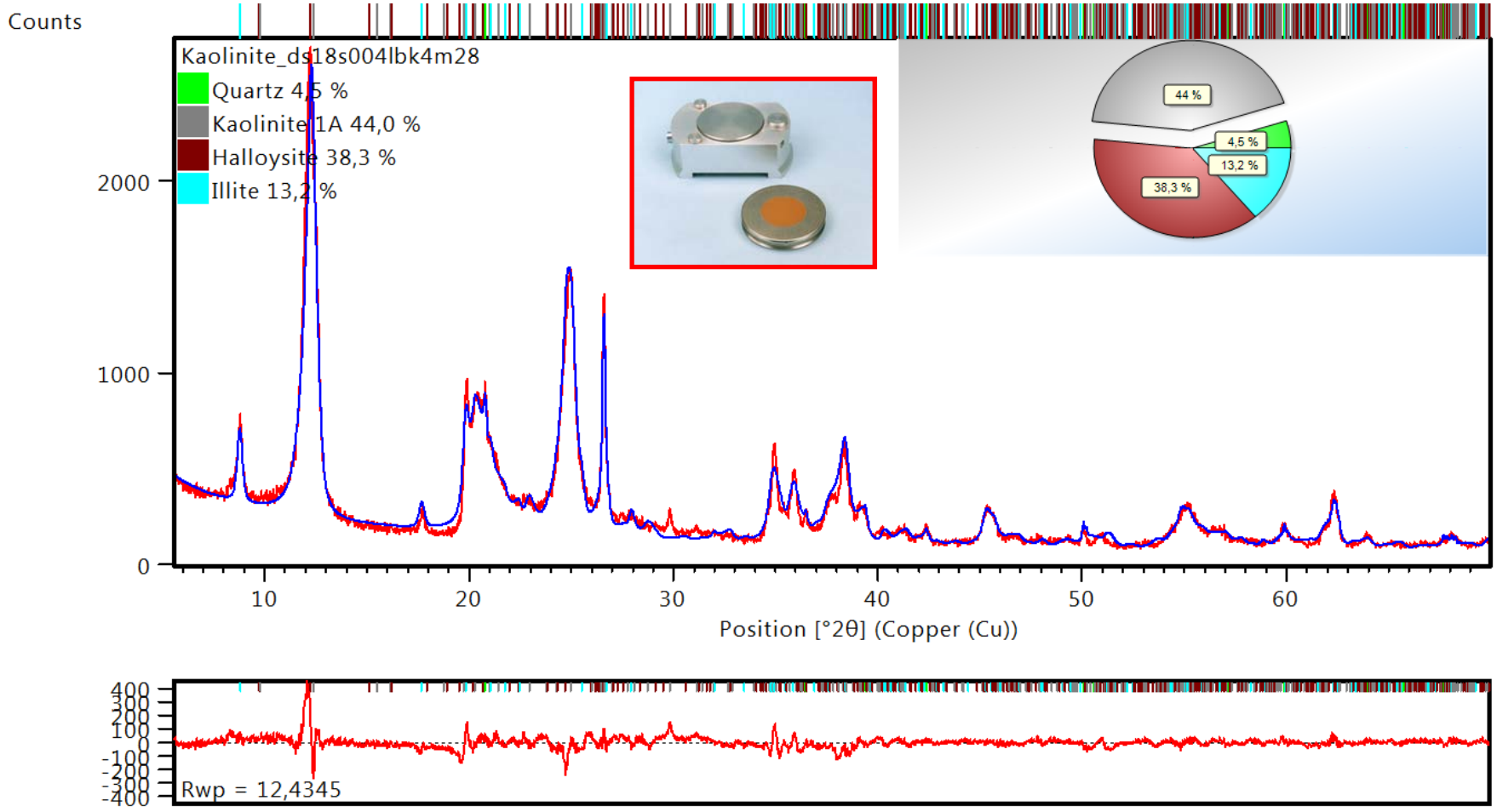
Ø 145 mm


Intensity (counts)
8000
6000
4000
2000
4
6
8
10
12
14
16
18
20
22
24
26
28
30
2Theta (°)
Ø 240 mm
Ø 145 mm

Capillary Sample



Pressed powder – backload sample preparation



Considerations

- It is very important to choose the proper system configuration on clay analysis – (1) peaks at low angles and (2) good resolution
- Quartz main peak can be helpful on verifying the appropriate sample height
- It is important to have between 5-11 points in a peak measured over the FWHM – resolution and low background noise
- Small divergent slits will improve the peak resolution but can decrease the intensities – always play with slit size and time/step
- In samples with multiple phases and with peak overlaps, the use of reduced Soller slits can be very helpful, specially at low angles – and also gives symmetry to the peaks
- The use of beam knife can eliminate the direct beam effect at low angles – therefore it is always important to select the proper height of the knife not to cut the intensity of the main peaks
- Short goniometer radius (145 mm) can give high intensity – in this way the low power (600 watts) does not affect the quality)

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Thank you!