
USP – Universidade de São Paulo

Física Experimental C - Laboratório

# Difração de Raio X

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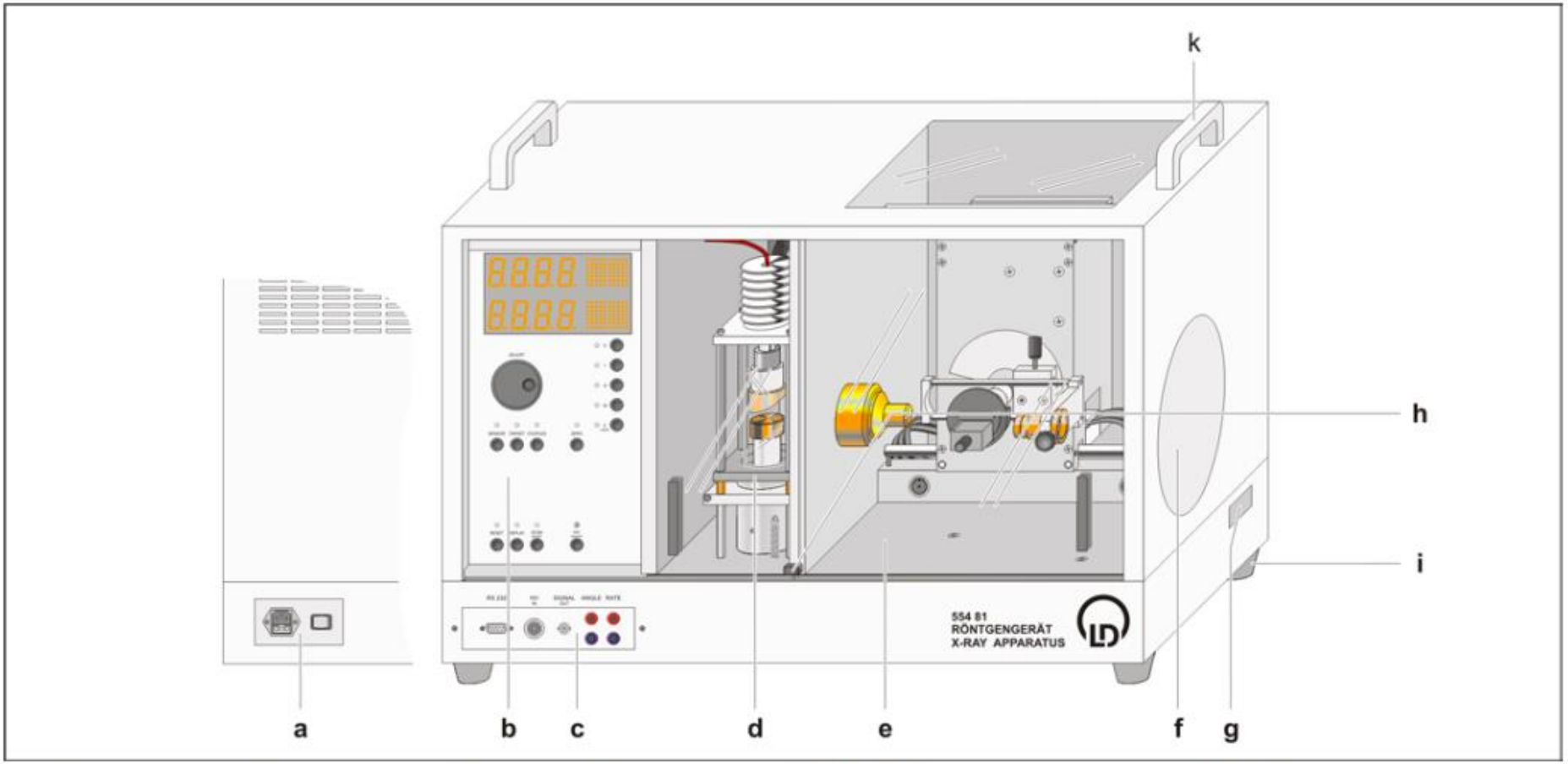
PSI / EP USP

Versão 2022

# Duas sessões simultâneas

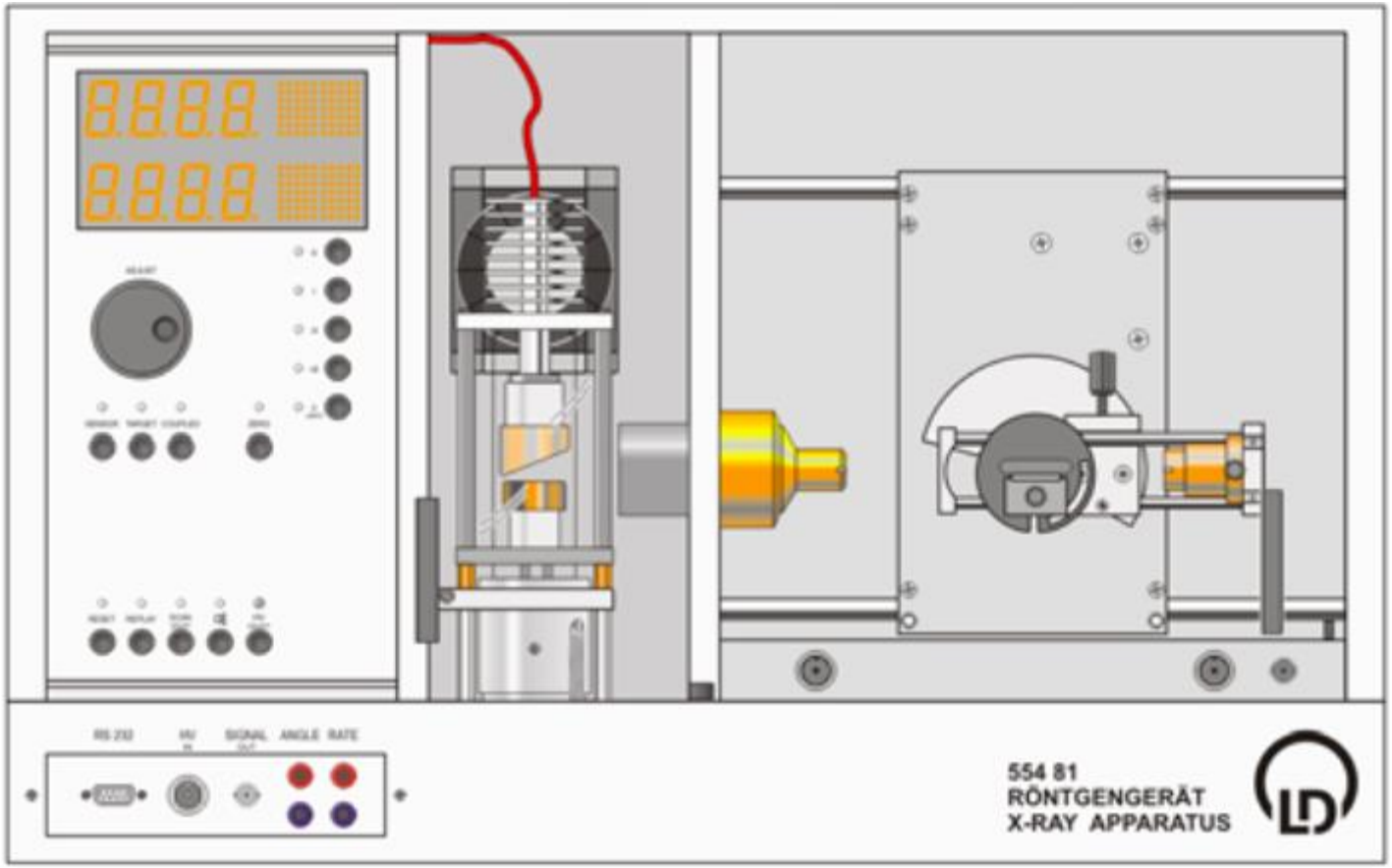
- **Sessão no laboratório (presencial)**
  - *1 aluno* da equipe
  - Adquirir dados reais
    - *só para* o experimento de Bragg a 35 kV
  - Calcular e analisar resultados reais
- **Sessão à distância**
  - *2 alunos* da equipe
  - Adquirir dados simulados
    - Para o experiment de Bragg a 35, 30 e 22 kV
    - Para o experiment de Duane-Hunt
  - Calcular e analisar resultados simulados
- **Relatório**
  - Parte do laboratório
  - Parte à distância
  - Comparação dos resultados reais com os simulados
    - Para o experiment de Bragg a 35 kV (*feito no lab e à distância*)

# Difratômetro de Raio X – no Laboratório (presencial)

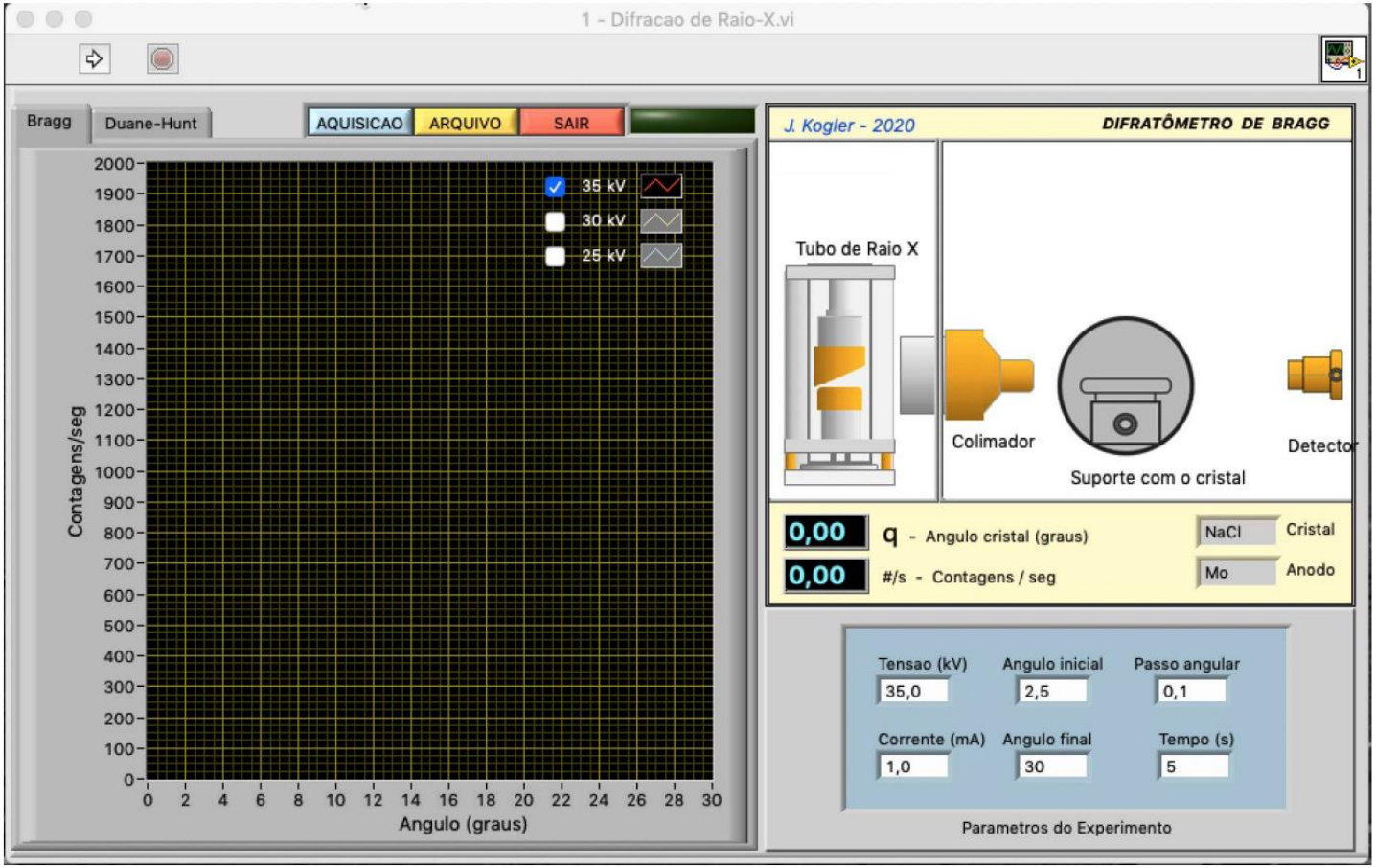



**a** Campo de conexão à rede
**b** Campo de comando
**c** Campo de conexão
**d** Câmara do tubo (com tubo de raios X)
**e** Câmara de experiências (aqui com goniômetro)
**f** Tela fluorescente
**g** Canal livre
**h** Tecla de bloqueio
**i** Pés
**k** Alças de transporte

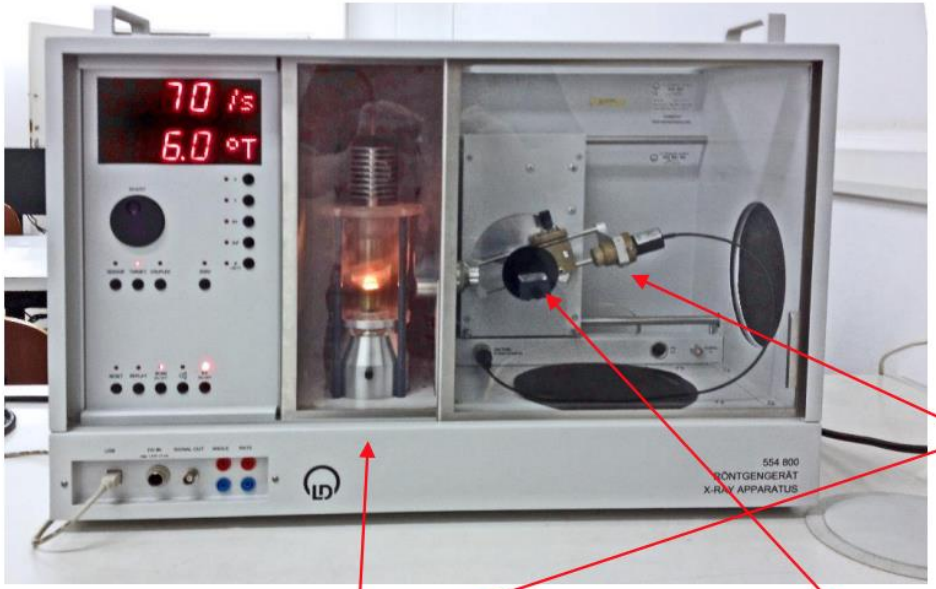
# Difratômetro de Raio X – no Laboratório (presencial)

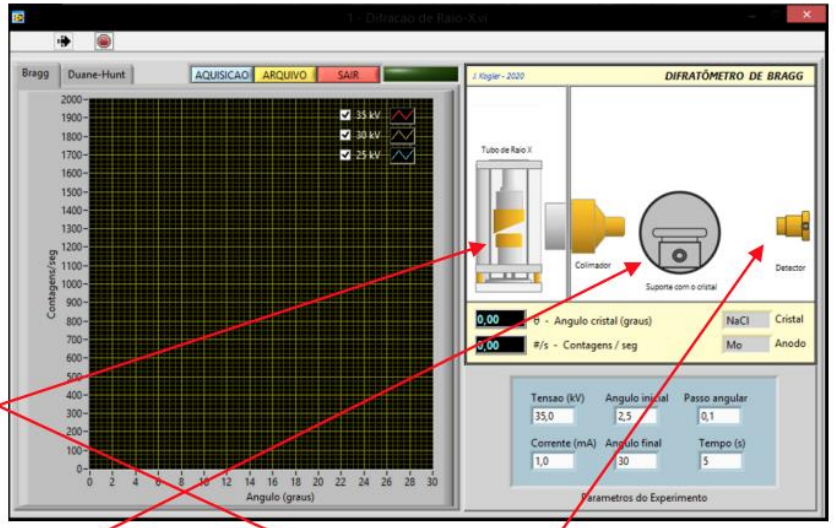


# Difratômetro de Raio X – Simulador em software (EAD)



# Comparação do equipamento físico com o simulador em software



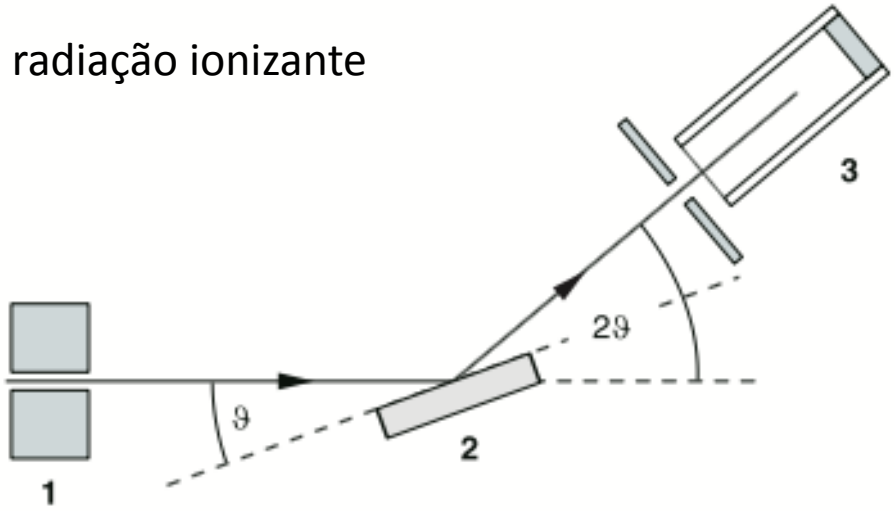




Tubo de raio-x

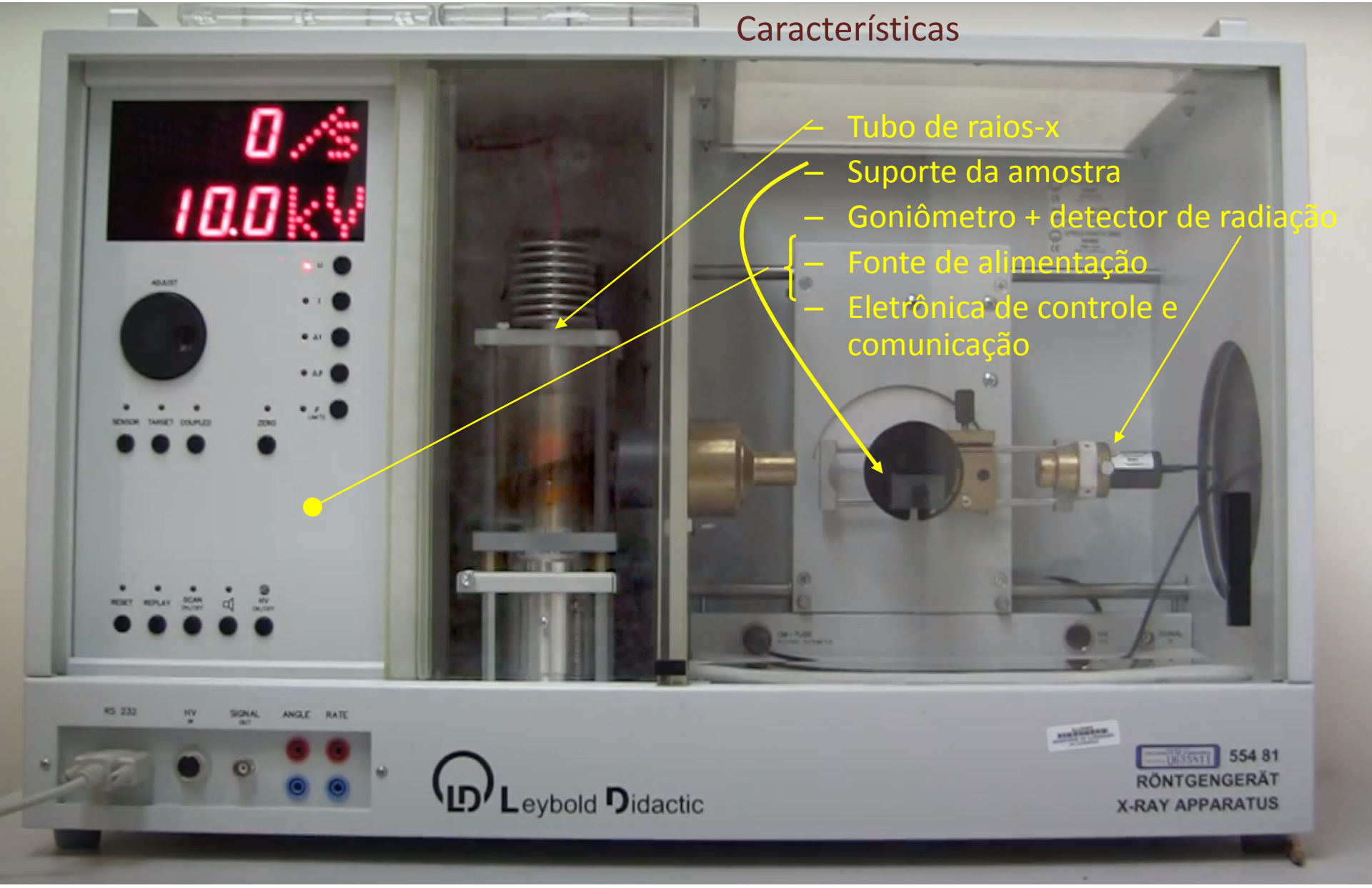
Cristal e suporte

Goniômetro e detector

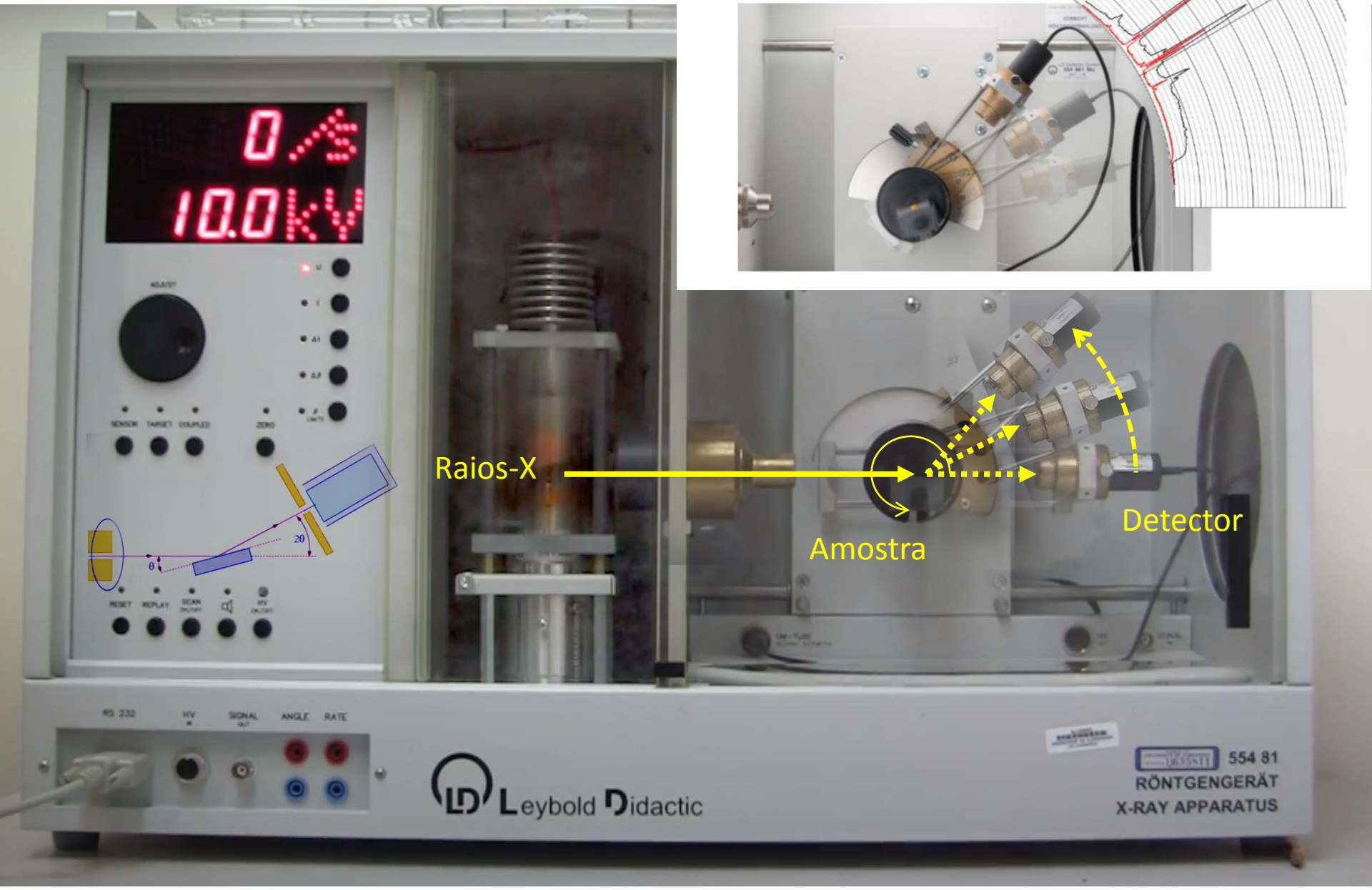
1. Fenda colimadora
2. Cristal
3. Detector de radiação ionizante



# O Difratômetro de Raio-X



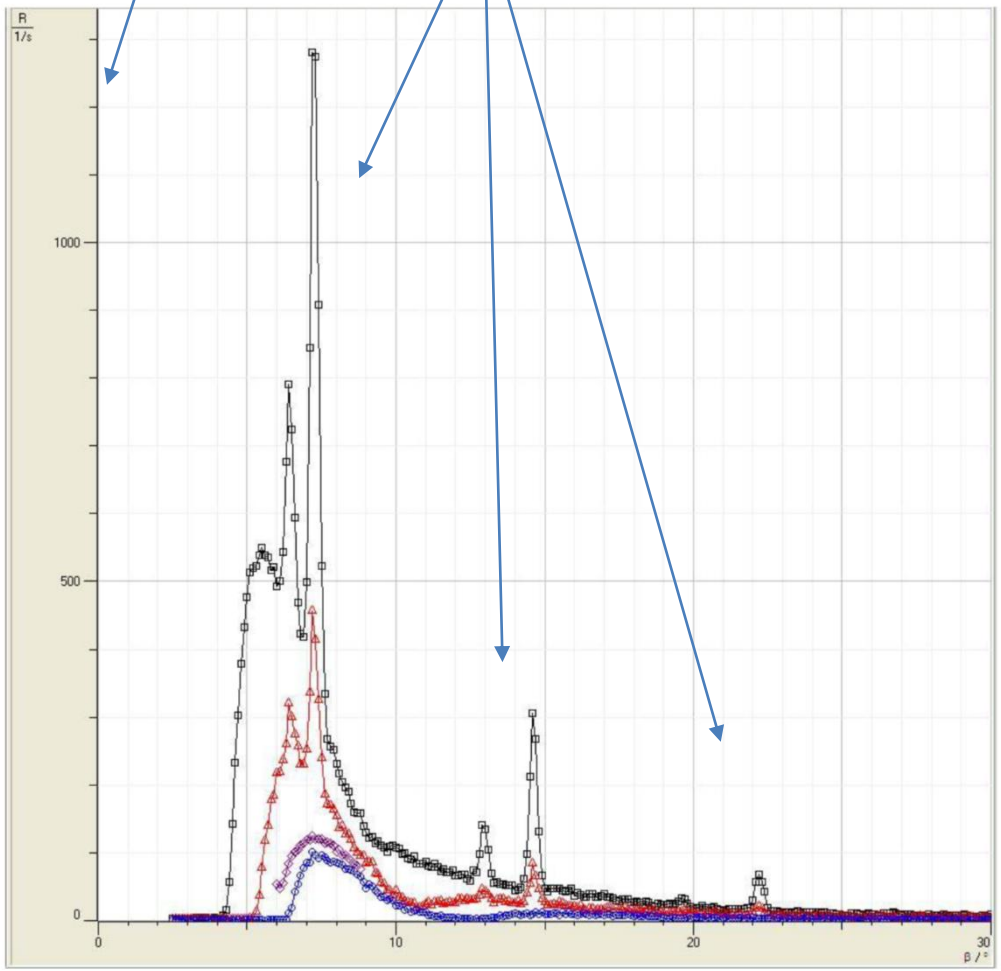
# O Difratômetro de Raio-X

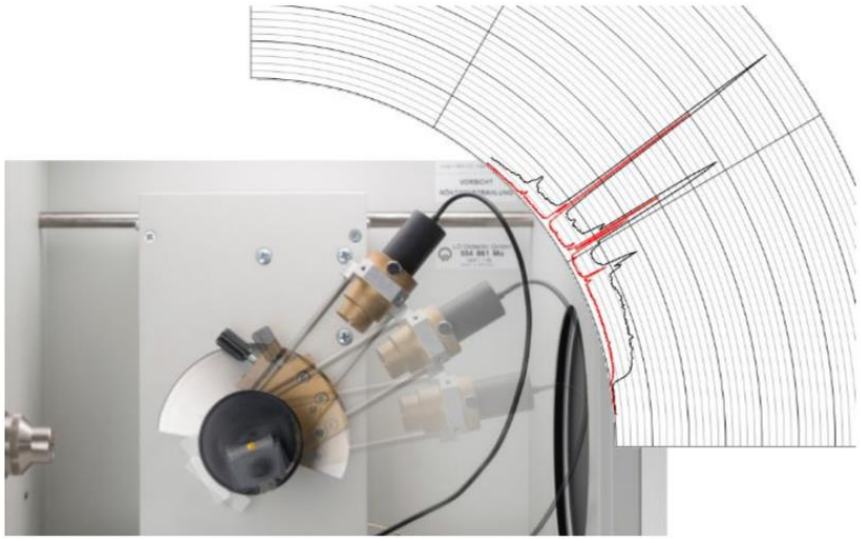


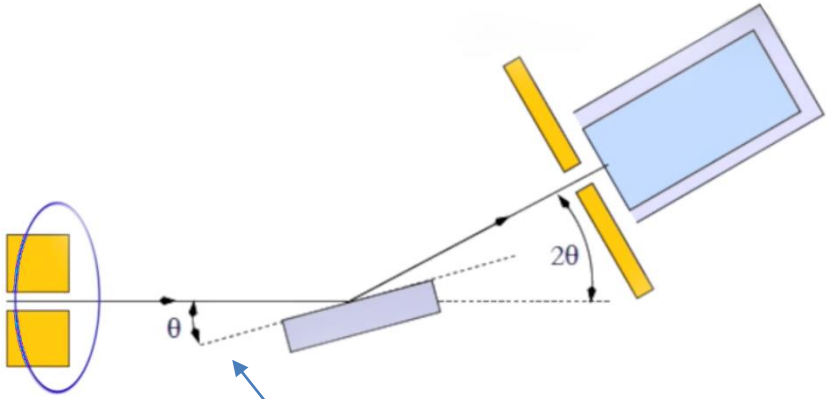
# O Difratômetro de Raio-X

# contagens / unidade te tempo

Picos de radiação característica

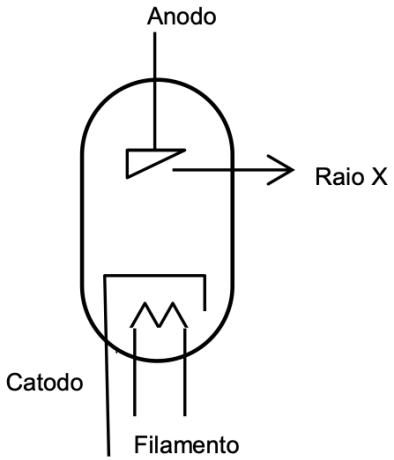


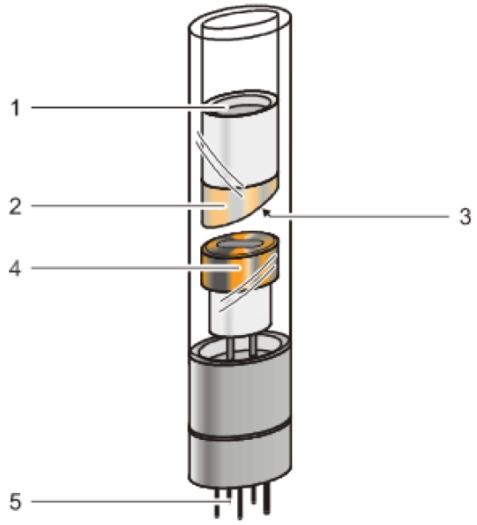






Ângulo de incidência

# Tubo de raio X




1 – Conexão com o dissipador de calor

2 – Bloco de cobre de suporte

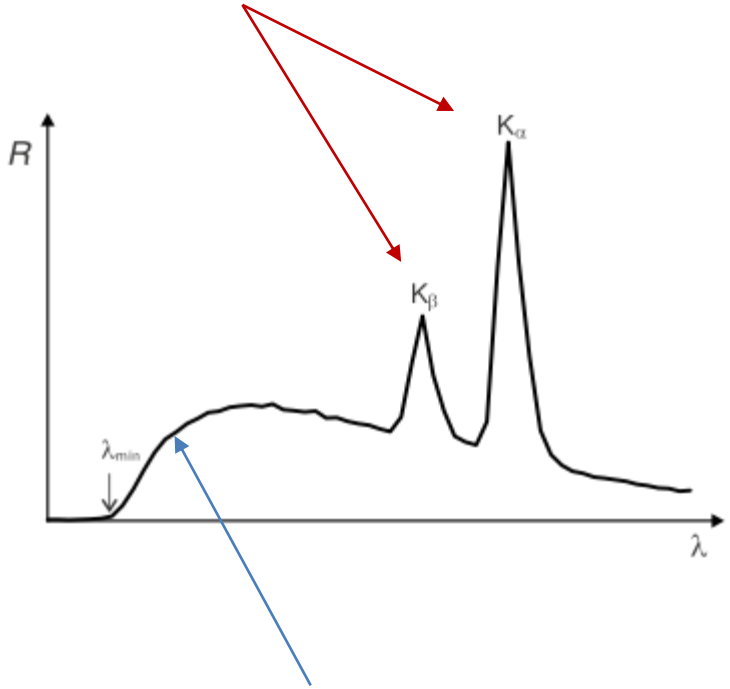
3 – Anodo de Molibdênio

4 – Catodo aquecido

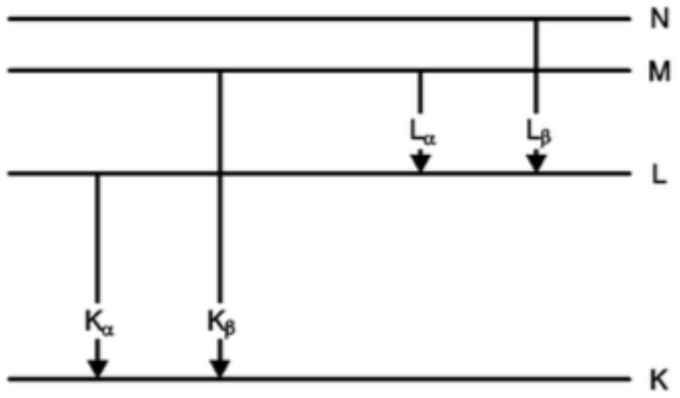
5 – Pinos de conexão ao soquete

# Emissões de raio X

Emissões de pico (‘fluorescências‘)
Radiação de decaimento

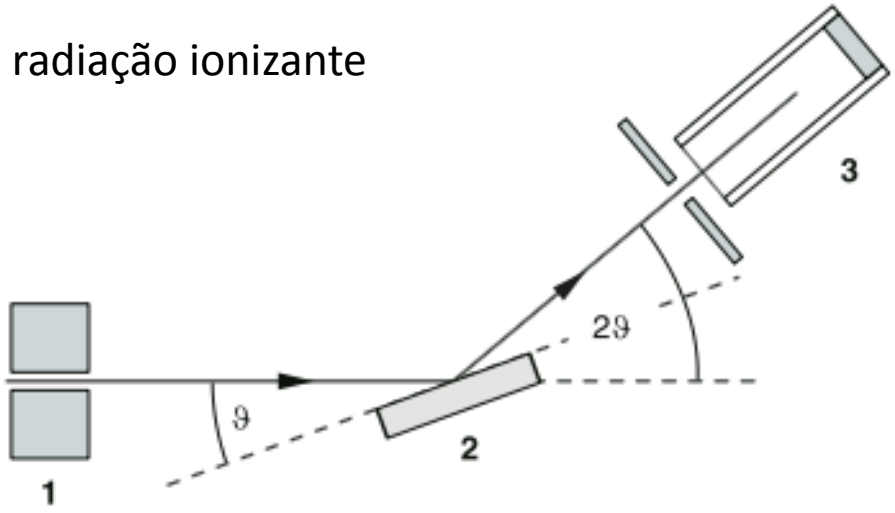


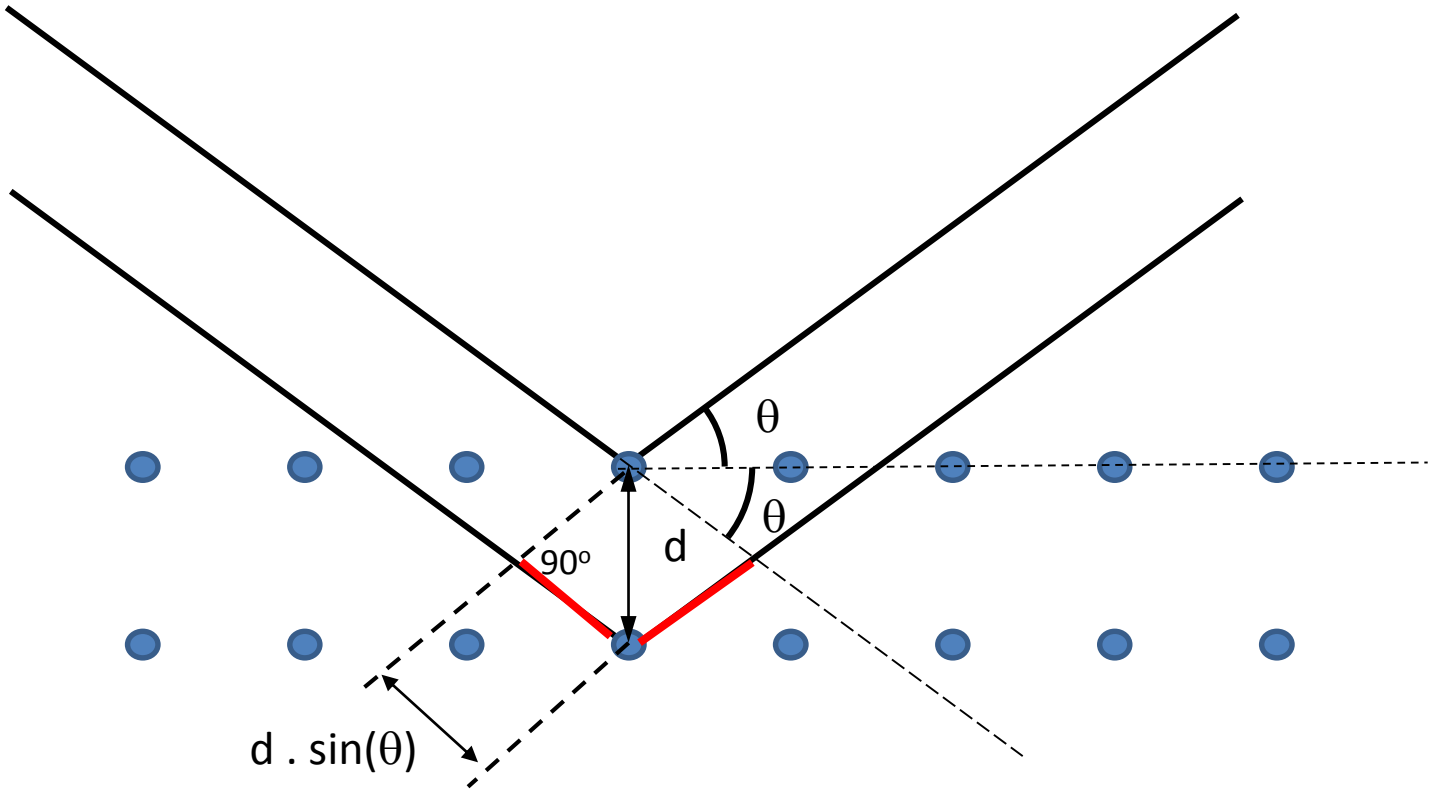

Emissão de fundo (background)
‘Bremsstrahlung’
(radiação de freamento)



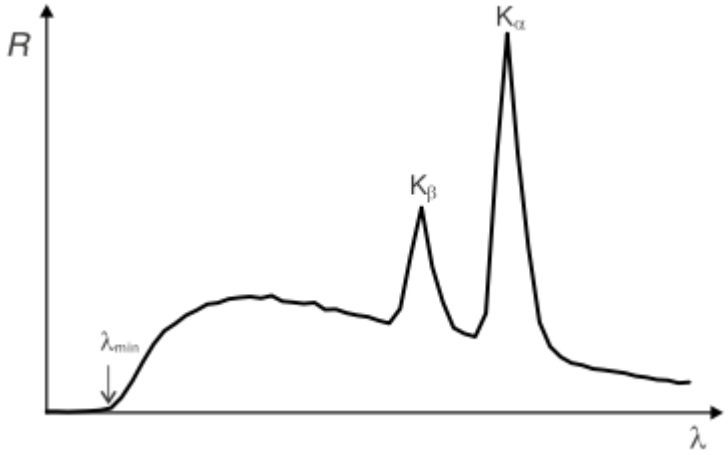

Emissões de pico (‘fluorescências‘)
Radiação de decaimento

1. Fenda colimadora
2. Cristal
3. Detector de radiação ionizante

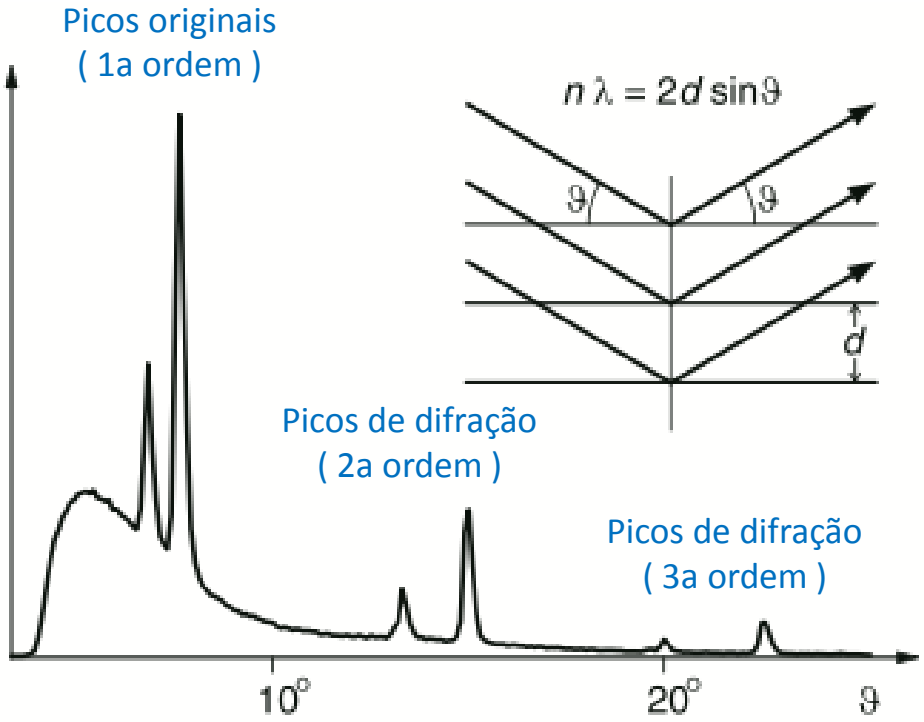





Lei de Bragg $n\lambda = 2 . d \sin(\theta)$




Sem a difração




Com a difração

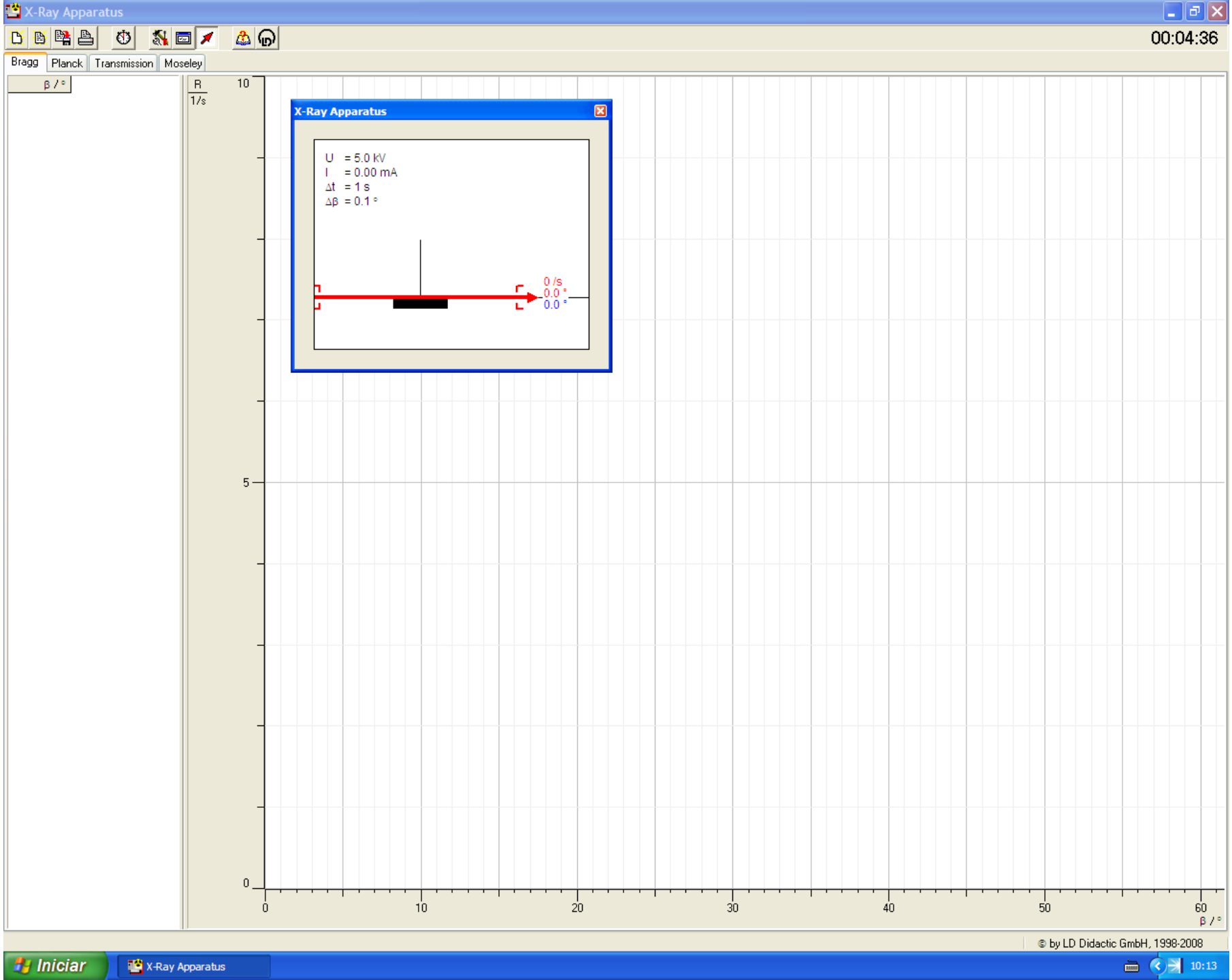
# Software *X ray apparatus*

- **Para quem estiver em casa**
  - Siga o link EAD no experiment de Difração de Raio X no web site da disciplina de Fisica C


- **Para quem estiver no lab**
  - Procure no desktop do computador o icone do software X_ray_app e clique nele e abra o programa

# Aquisição dos dados no LAB

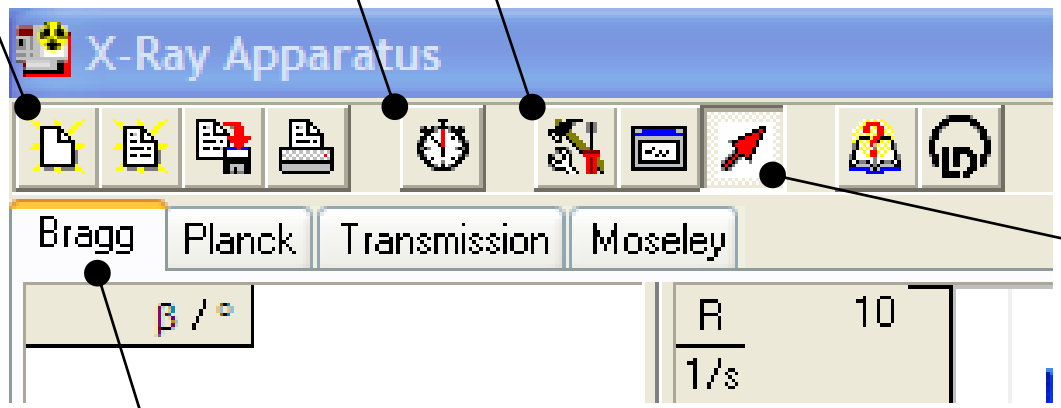
Primeiramente, abra o programa X ray apparatus no computador


X-Ray Apparatus
00:04:36
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
5
0
0
10
20
30
40
50
60
β / °
X-Ray Apparatus
U = 5.0 kV
I = 0.00 mA
Δt = 1 s
Δβ = 0.1 °
0 /s
0.0 °
0.0 °
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
10:13

Salvar / apagar area de trabalho

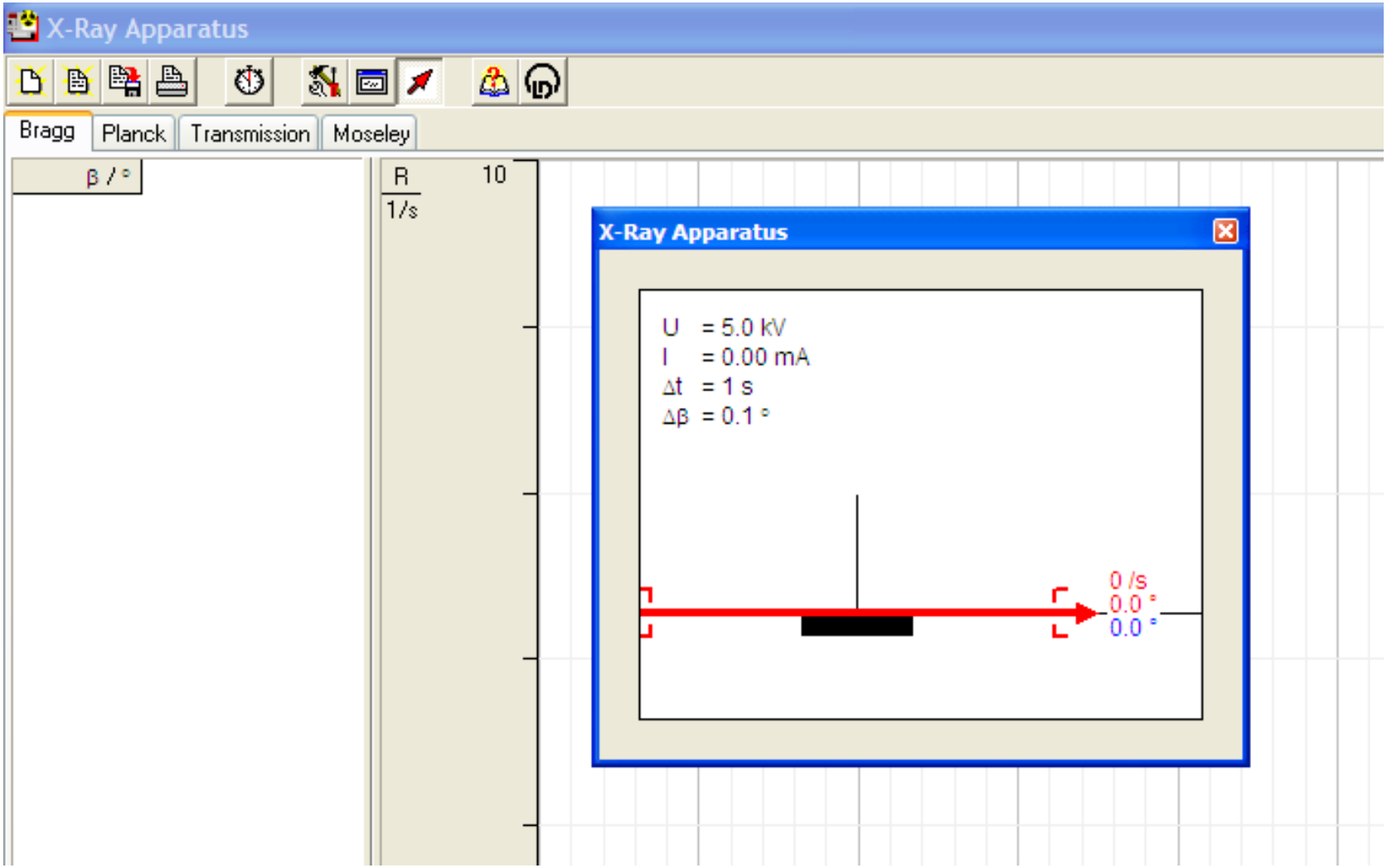
Parar / reiniciar medições

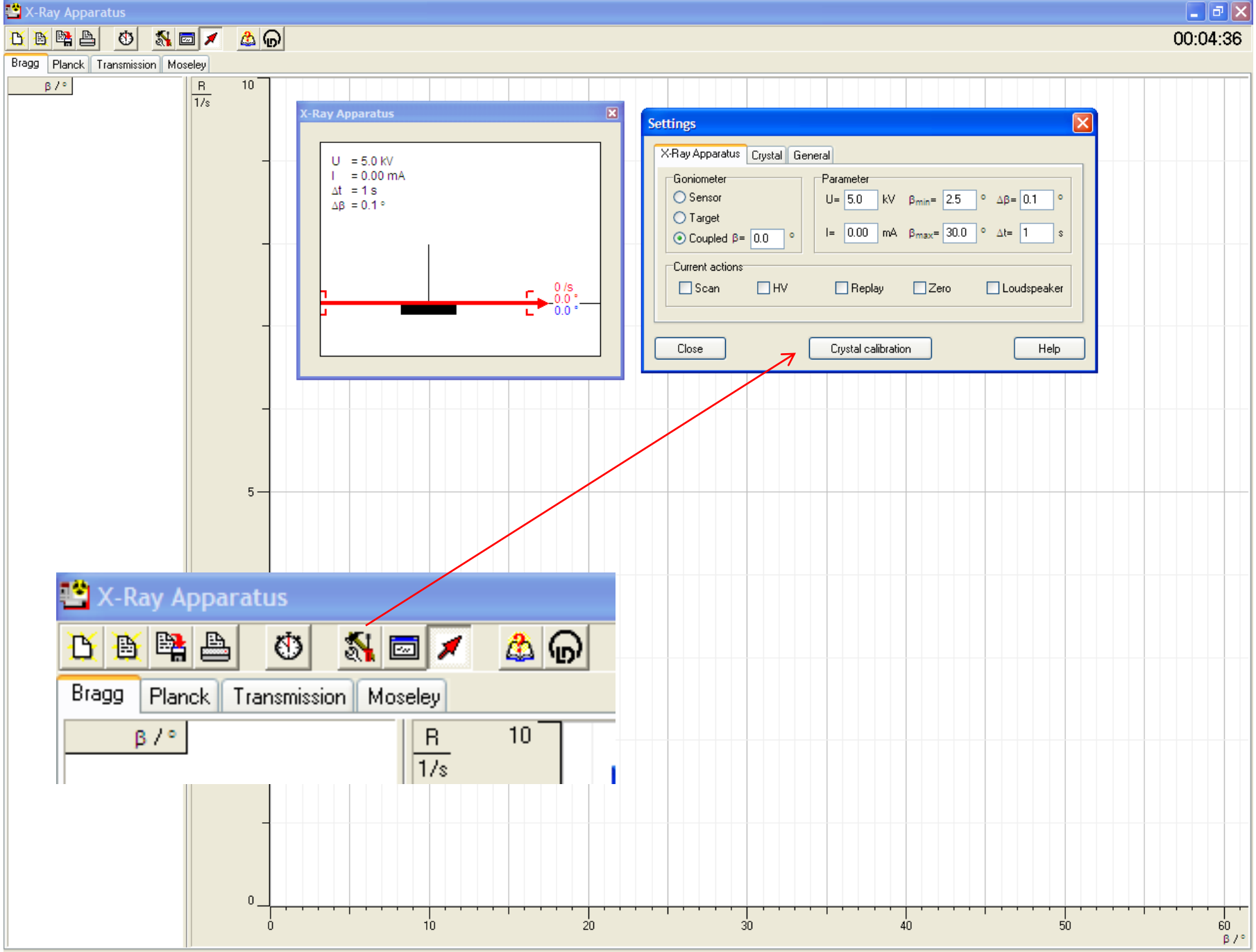
Ajustes dos parâmetros experimentais

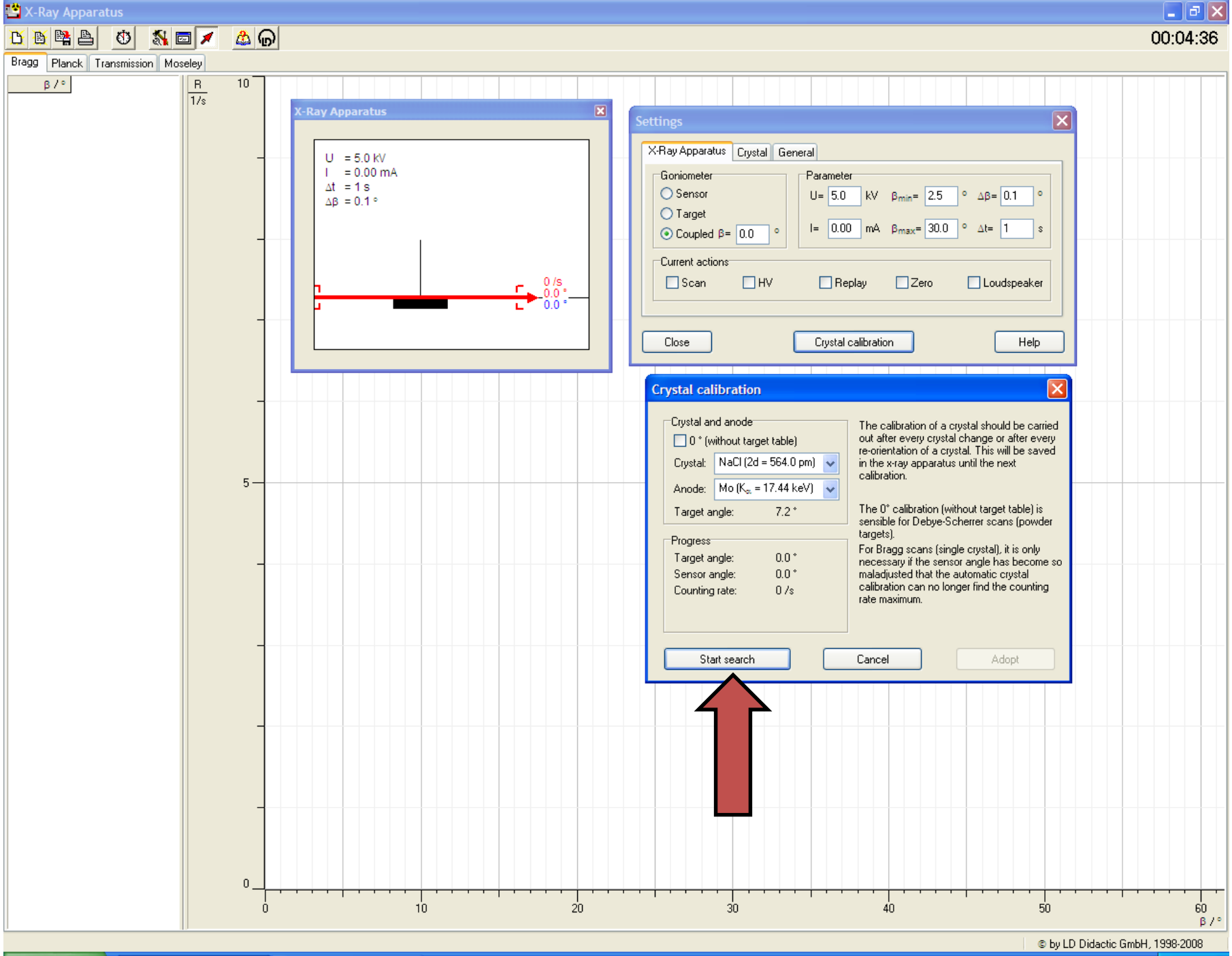


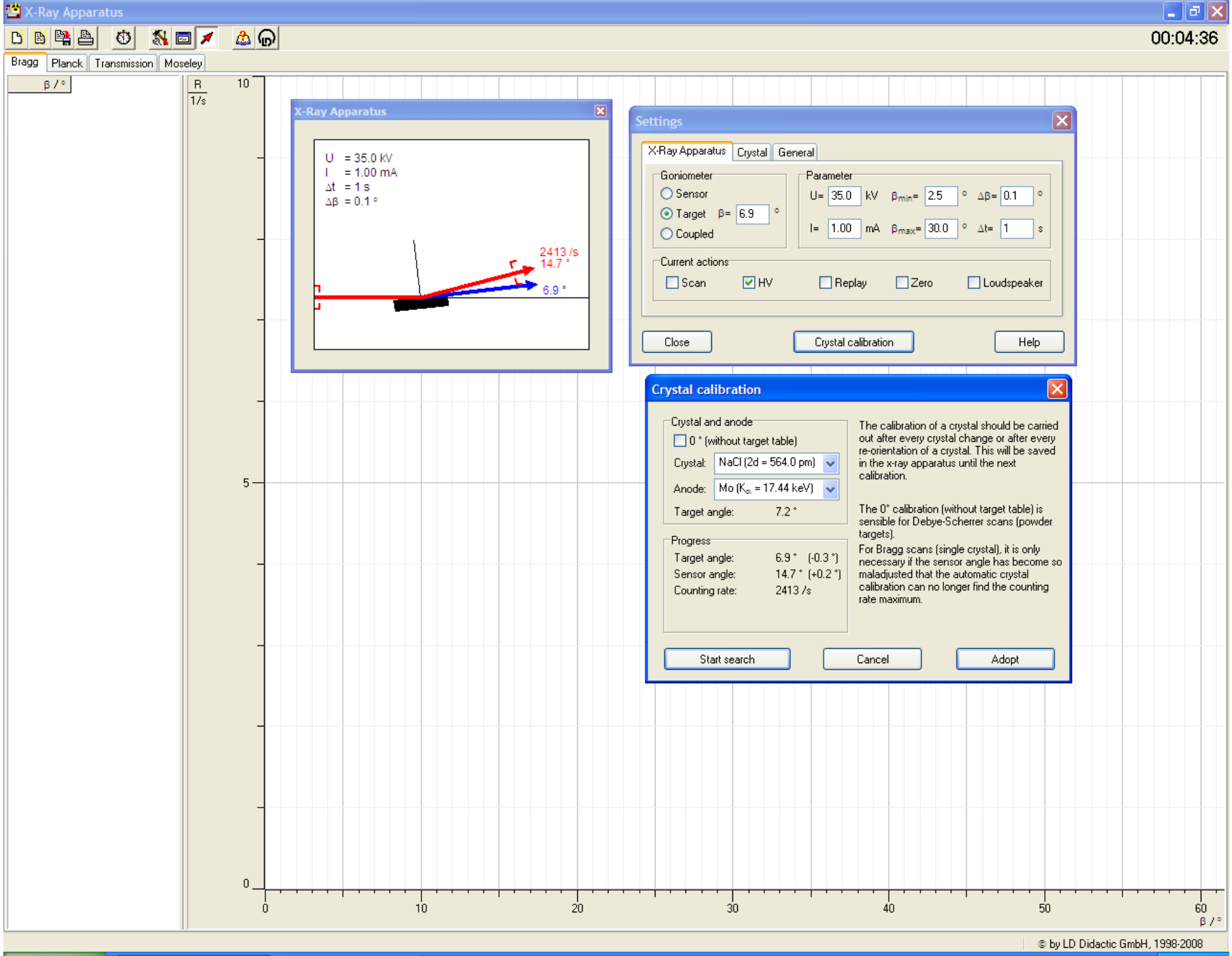

Visualização dos ângulos

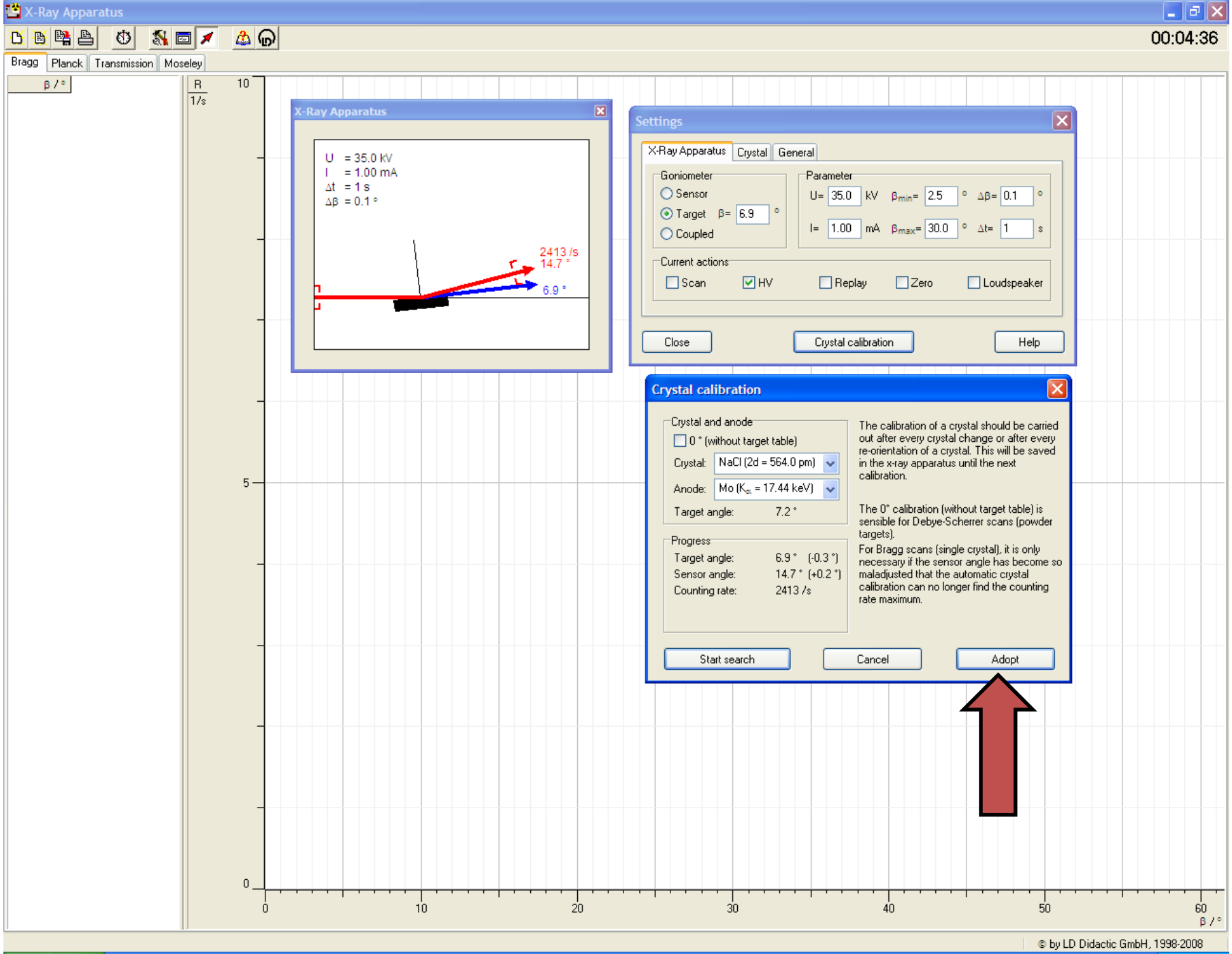
1ª parte – difratometria de Bragg p/medir "***d***"

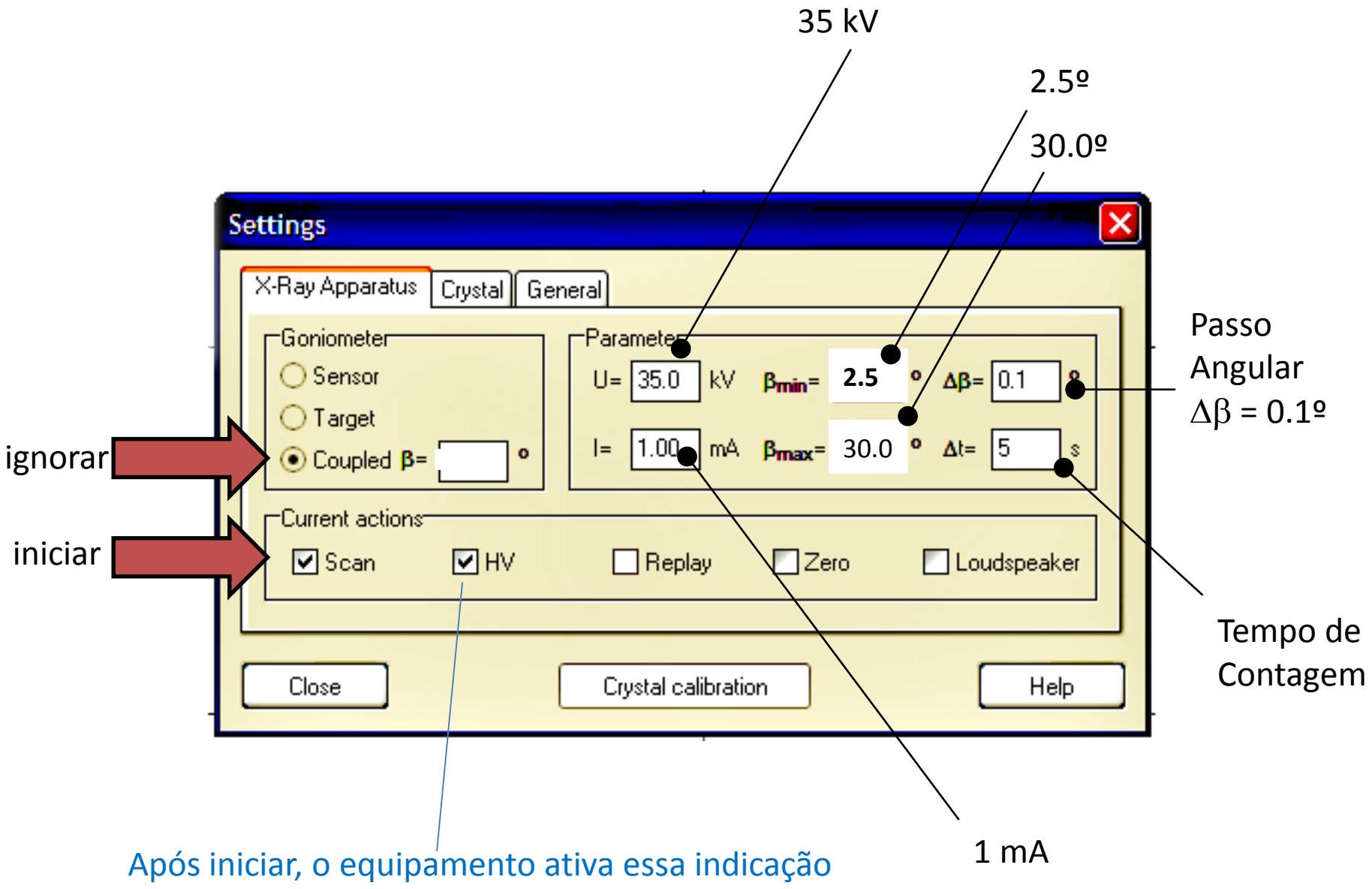

X-Ray Apparatus
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
X-Ray Apparatus
U = 5.0 kV
I = 0.00 mA
Δt = 1 s
Δβ = 0.1 °
0 /s
0.0 °
0.0 °


X-Ray Apparatus
00:04:36
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
5
0
0
10
20
30
40
50
60
β / °
X-Ray Apparatus
U = 5.0 kV
I = 0.00 mA
Δt = 1 s
Δβ = 0.1 °
0 /s
0.0 °
0.0 °
Settings
X-Ray Apparatus
Crystal
General
Goniometer
Sensor
Target
Coupled β= 0.0 °
Parameter
U= 5.0 kV
I= 0.00 mA
βmin= 2.5 °
βmax= 30.0 °
Δβ= 0.1 °
Δt= 1 s
Current actions
Scan
HV
Replay
Zero
Loudspeaker
Close
Crystal calibration
Help
X-Ray Apparatus
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx1 - Paint
10:16


X-Ray Apparatus
00:04:36
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
5
0
0
10
20
30
40
50
60
β / °
X-Ray Apparatus
U = 5.0 kV
I = 0.00 mA
Δt = 1 s
Δβ = 0.1 °
0 /s
0.0 °
0.0 °
Settings
X-Ray Apparatus
Crystal
General
Goniometer
Sensor
Target
Coupled β= 0.0 °
Parameter
U= 5.0 kV
βmin= 2.5 °
Δβ= 0.1 °
I= 0.00 mA
βmax= 30.0 °
Δt= 1 s
Current actions
Scan
HV
Replay
Zero
Loudspeaker
Close
Crystal calibration
Help
Crystal calibration
Crystal and anode
0 ° (without target table)
Crystal: NaCl (2d = 564.0 pm)
Anode: Mo (Kα = 17.44 keV)
Target angle: 7.2 °
Progress
Target angle: 0.0 °
Sensor angle: 0.0 °
Counting rate: 0 /s
The calibration of a crystal should be carried out after every crystal change or after every re-orientation of a crystal. This will be saved in the x-ray apparatus until the next calibration.
The 0° calibration (without target table) is sensible for Debye-Scherrer scans (powder targets).
For Bragg scans (single crystal), it is only necessary if the sensor angle has become so maladjusted that the automatic crystal calibration can no longer find the counting rate maximum.
Start search
Cancel
Adopt
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx2 - Paint
10:16


X-Ray Apparatus
00:04:36
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
5
0
0
10
20
30
40
50
60
β / °
X-Ray Apparatus
U = 35.0 kV
I = 1.00 mA
Δt = 1 s
Δβ = 0.1 °
2413 /s
14.7 °
6.9 °
Settings
X-Ray Apparatus
Crystal
General
Goniometer
Sensor
Target β= 6.9 °
Coupled
Parameter
U= 35.0 kV βmin= 2.5 ° Δβ= 0.1 °
I= 1.00 mA βmax= 30.0 ° Δt= 1 s
Current actions
Scan
HV
Replay
Zero
Loudspeaker
Close
Crystal calibration
Help
Crystal calibration
Crystal and anode
0 ° (without target table)
Crystal: NaCl (2d = 564.0 pm)
Anode: Mo (Kα = 17.44 keV)
Target angle: 7.2 °
Progress
Target angle: 6.9 ° (-0.3 °)
Sensor angle: 14.7 ° (+0.2 °)
Counting rate: 2413 /s
The calibration of a crystal should be carried out after every crystal change or after every re-orientation of a crystal. This will be saved in the x-ray apparatus until the next calibration.
The 0° calibration (without target table) is sensible for Debye-Scherrer scans (powder targets).
For Bragg scans (single crystal), it is only necessary if the sensor angle has become so maladjusted that the automatic crystal calibration can no longer find the counting rate maximum.
Start search
Cancel
Adopt
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx3 - Paint
10:19


X-Ray Apparatus
00:04:36
Bragg
Planck
Transmission
Moseley
β / °
R
1/s
10
5
0
0
10
20
30
40
50
60
β / °
X-Ray Apparatus
U = 35.0 kV
I = 1.00 mA
Δt = 1 s
Δβ = 0.1 °
2413 /s
14.7 °
6.9 °
Settings
X-Ray Apparatus
Crystal
General
Goniometer
Sensor
Target β= 6.9 °
Coupled
Parameter
U= 35.0 kV βmin= 2.5 ° Δβ= 0.1 °
I= 1.00 mA βmax= 30.0 ° Δt= 1 s
Current actions
Scan
HV
Replay
Zero
Loudspeaker
Close
Crystal calibration
Help
Crystal calibration
Crystal and anode
0 ° (without target table)
Crystal: NaCl (2d = 564.0 pm)
Anode: Mo (Kα = 17.44 keV)
Target angle: 7.2 °
Progress
Target angle: 6.9 ° (-0.3 °)
Sensor angle: 14.7 ° (+0.2 °)
Counting rate: 2413 /s
The calibration of a crystal should be carried out after every crystal change or after every re-orientation of a crystal. This will be saved in the x-ray apparatus until the next calibration.
The 0° calibration (without target table) is sensible for Debye-Scherrer scans (powder targets).
For Bragg scans (single crystal), it is only necessary if the sensor angle has become so maladjusted that the automatic crystal calibration can no longer find the counting rate maximum.
Start search
Cancel
Adopt
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx3 - Paint
10:19

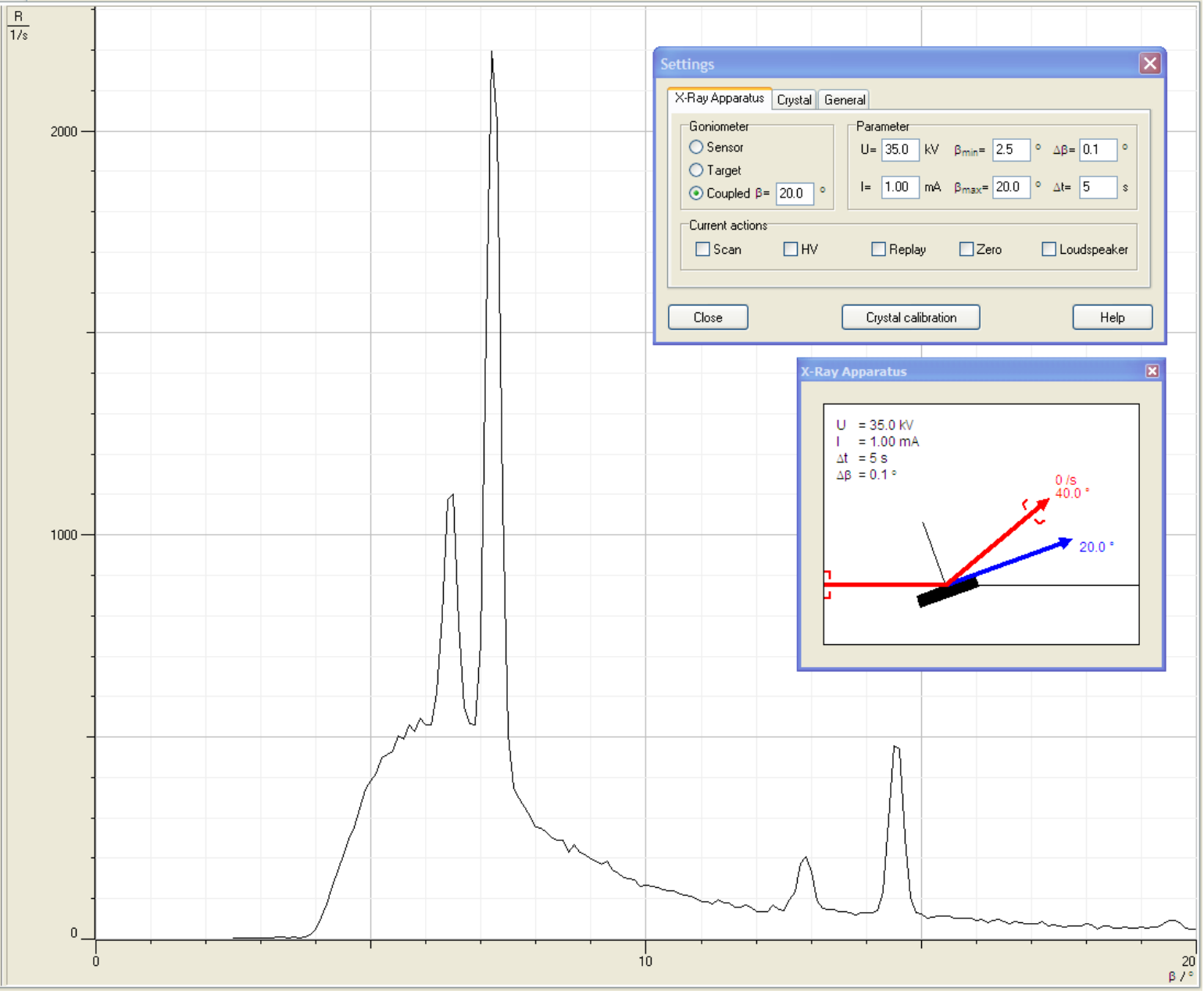

35 kV
2.5º
30.0º
Settings
X-Ray Apparatus
Crystal
General
Goniometer
Sensor
Target
Coupled β=
o
Parameter
U= 35.0 kV
βmin= 2.5 o
Δβ= 0.1 o
I= 1.00 mA
βmax= 30.0 o
Δt= 5 s
Current actions
Scan
HV
Replay
Zero
Loudspeaker
Close
Crystal calibration
Help
ignorar
iniciar
Passo Angular Δβ = 0.1º
Tempo de Contagem
Após iniciar, o equipamento ativa essa indicação
1 mA

X-Ray Apparatus

00:14:40

Bragg | Planck | Transmission | Moseley

| β / ° | $R_0$ / 1/s |
|---|---|
| 15.3 | 56.4 |
| 15.4 | 56.0 |
| 15.5 | 56.6 |
| 15.6 | 52.4 |
| 15.7 | 52.8 |
| 15.8 | 51.2 |
| 15.9 | 51.0 |
| 16.0 | 45.2 |
| 16.1 | 48.6 |
| 16.2 | 40.2 |
| 16.3 | 45.0 |
| 16.4 | 49.0 |
| 16.5 | 44.2 |
| 16.6 | 39.2 |
| 16.7 | 44.4 |
| 16.8 | 40.4 |
| 16.9 | 38.4 |
| 17.0 | 38.8 |
| 17.1 | 37.6 |
| 17.2 | 43.4 |
| 17.3 | 33.0 |
| 17.4 | 34.0 |
| 17.5 | 32.8 |
| 17.6 | 29.6 |
| 17.7 | 33.0 |
| 17.8 | 32.6 |
| 17.9 | 31.6 |
| 18.0 | 37.4 |
| 18.1 | 35.0 |
| 18.2 | 25.4 |
| 18.3 | 32.0 |
| 18.4 | 33.2 |
| 18.5 | 26.8 |
| 18.6 | 27.0 |
| 18.7 | 28.6 |
| 18.8 | 27.4 |
| 18.9 | 27.4 |
| 19.0 | 29.0 |
| 19.1 | 26.6 |
| 19.2 | 30.0 |
| 19.3 | 30.0 |
| 19.4 | 38.2 |
| 19.5 | 47.0 |
| 19.6 | 47.2 |
| 19.7 | 40.4 |
| 19.8 | 28.2 |
| 19.9 | 24.6 |
| 20.0 | 24.8 |




Settings

X-Ray Apparatus | Crystal | General

Goniometer
- Sensor
- Target
- Coupled β= 20.0 °

Parameter
U= 35.0 kV  $\beta_{min}$= 2.5 °  Δβ= 0.1 °
I= 1.00 mA  $\beta_{max}$= 20.0 °  Δt= 5 s

Current actions
Scan | HV | Replay | Zero | Loudspeaker

Close | Crystal calibration | Help

X-Ray Apparatus



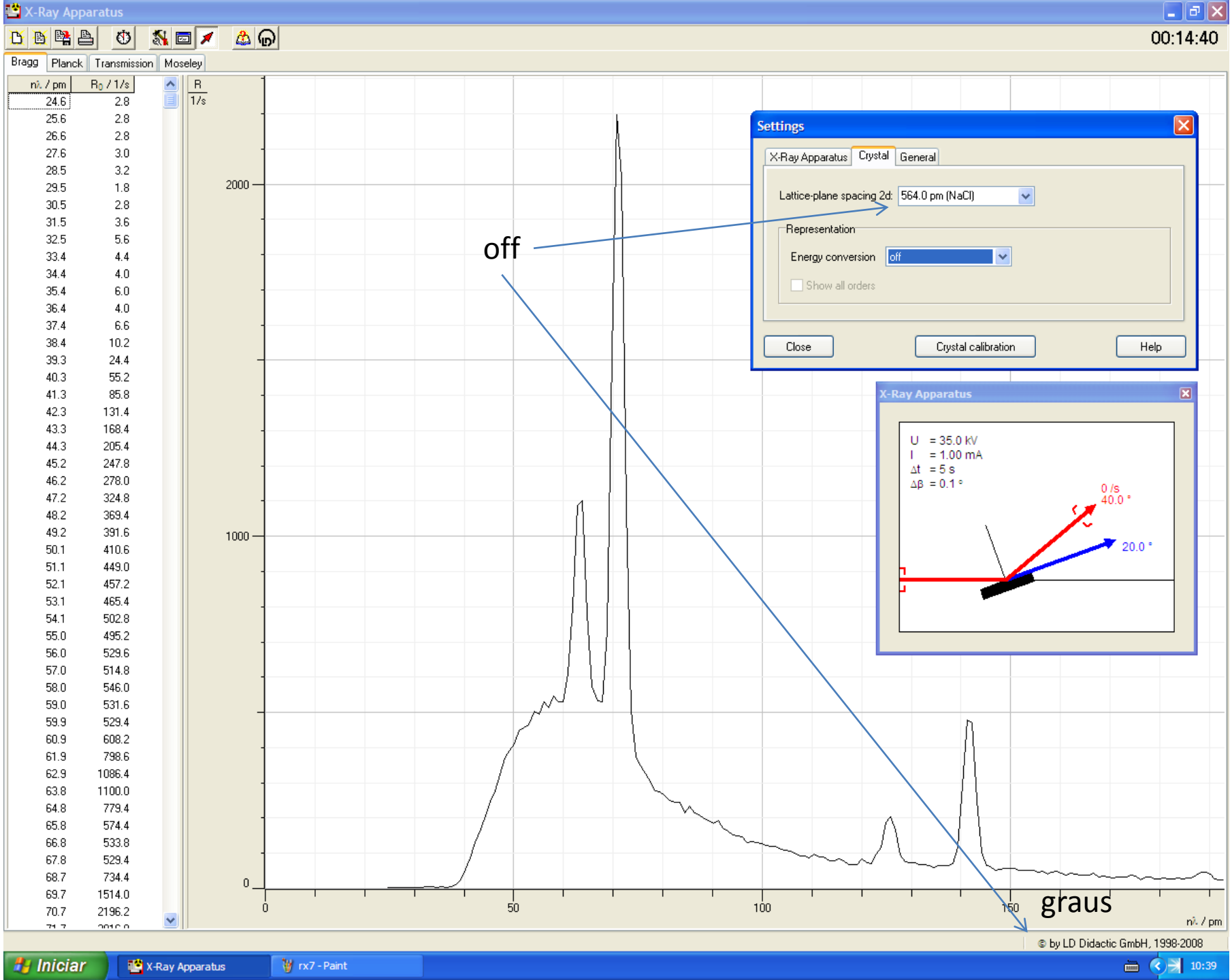


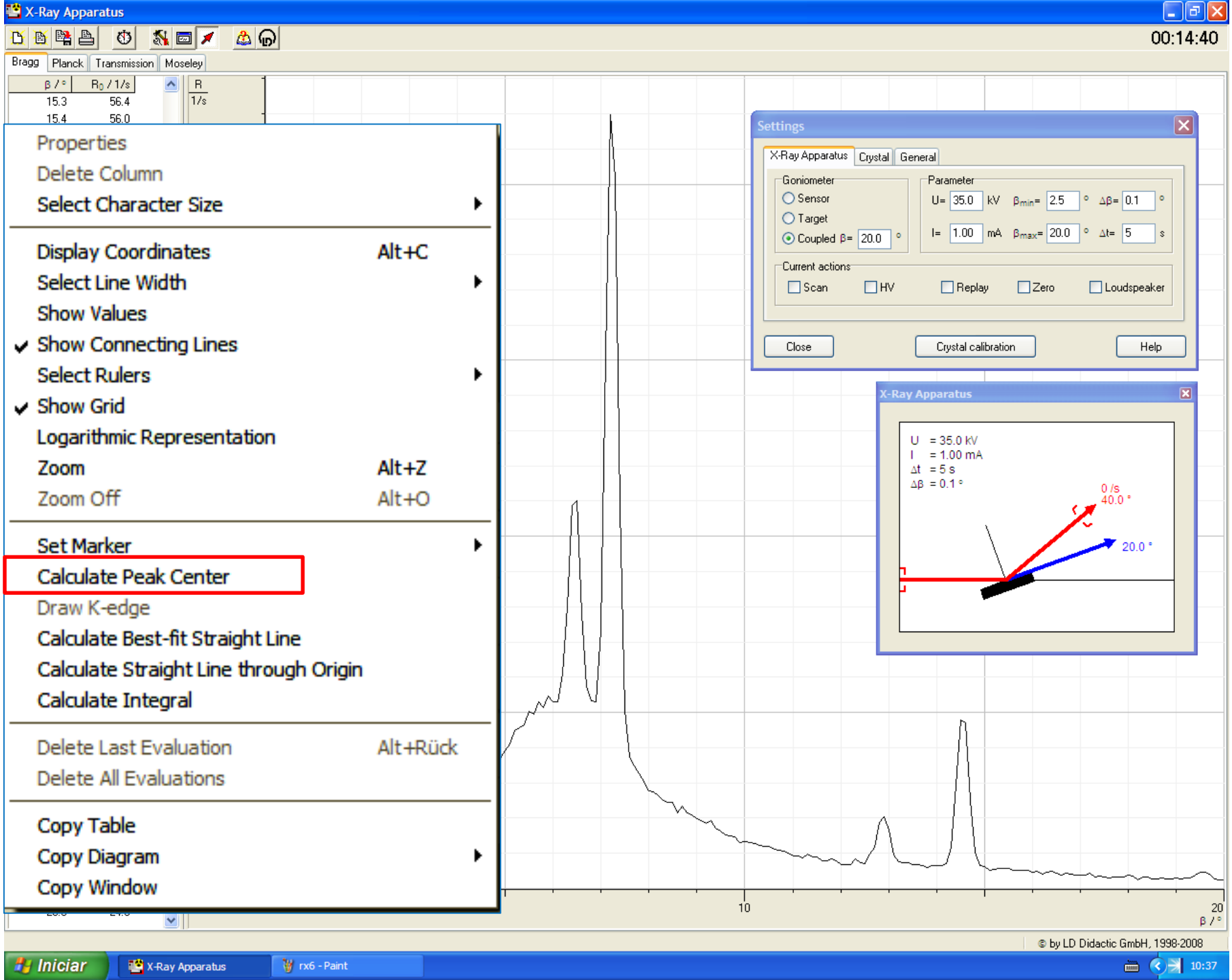

Iniciar | X-Ray Apparatus | rx6 - Paint | 10:37

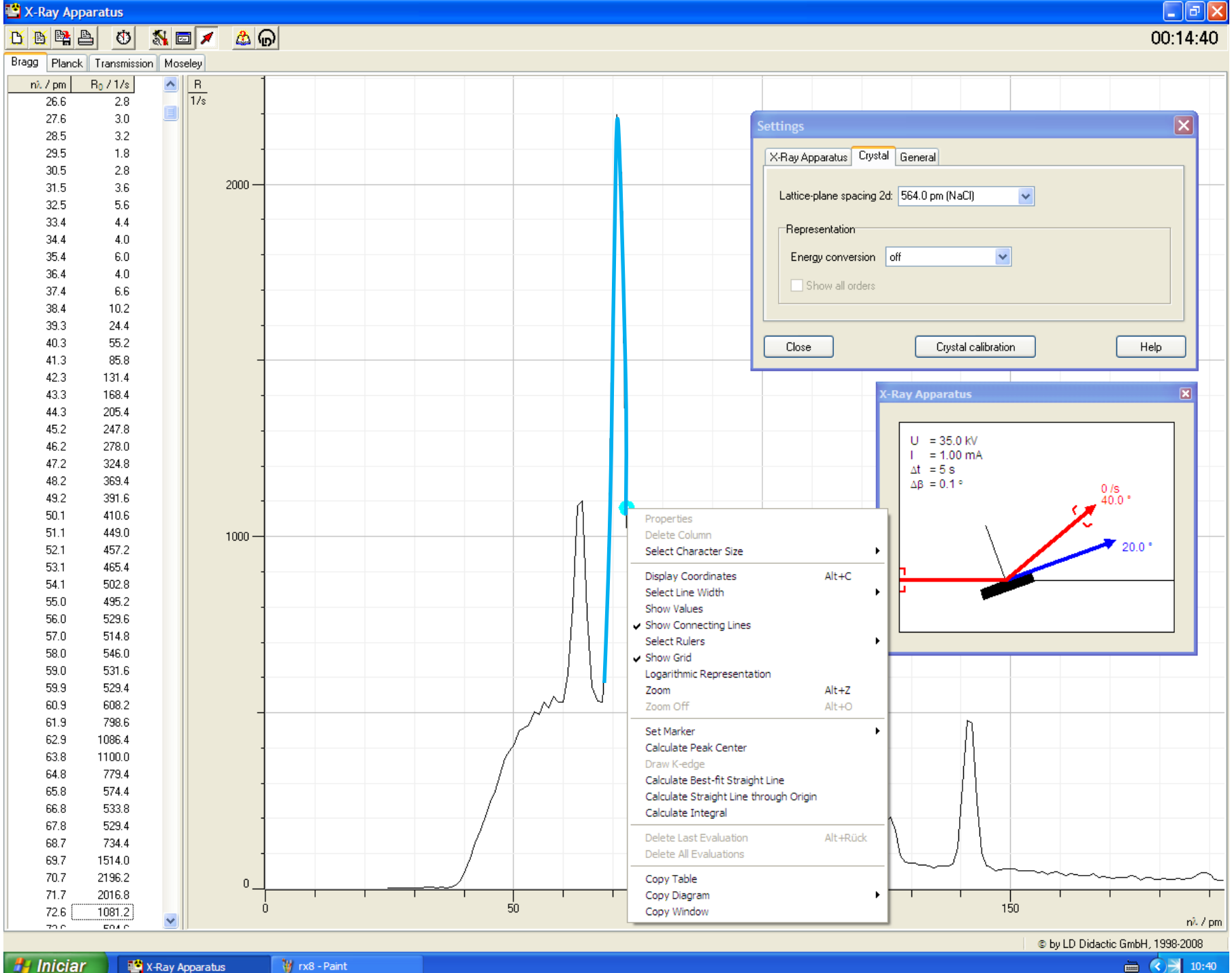
# Análise dos dados no LAB

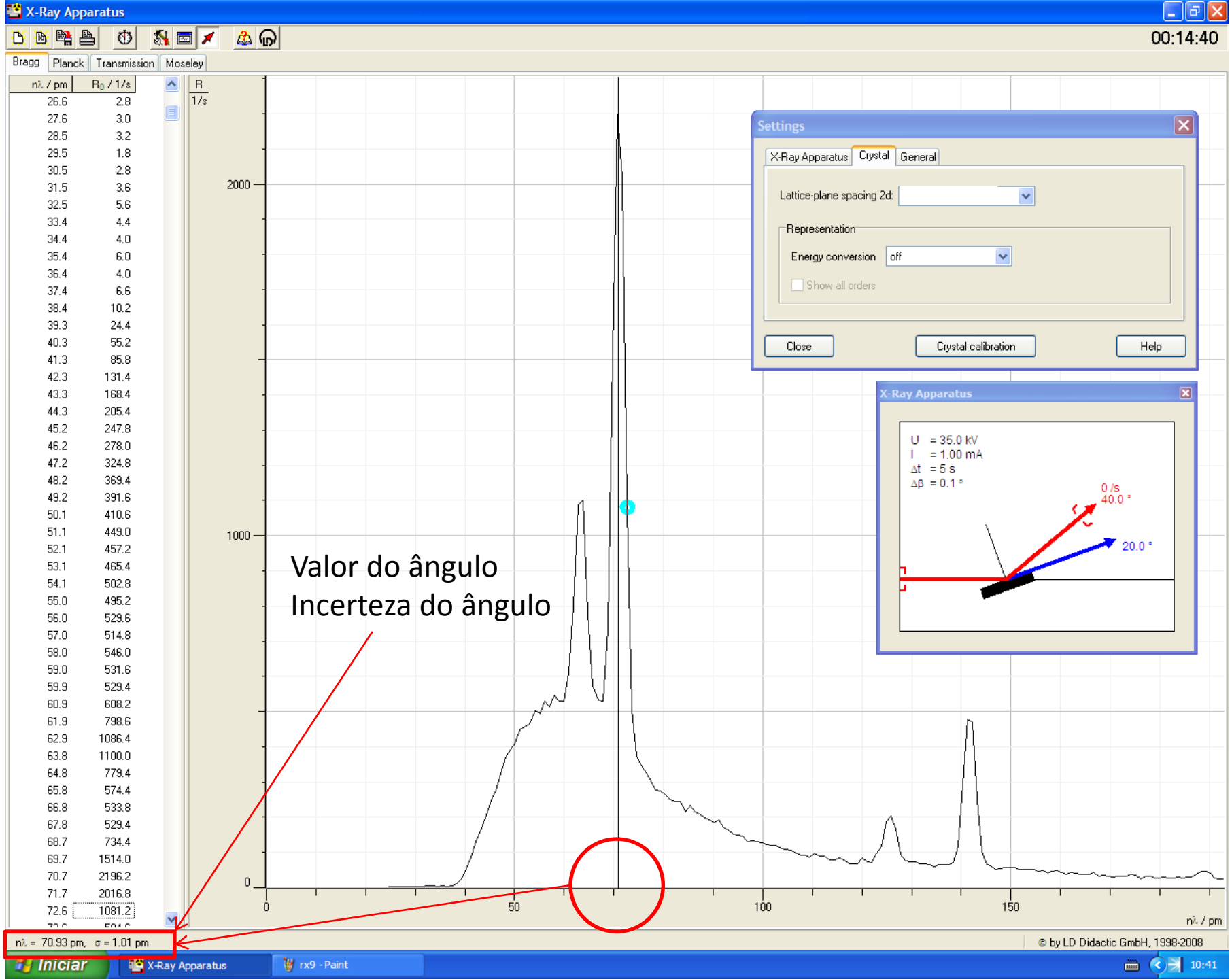
Feita com auxílio do programa
X ray apparatus

1. Determinar os centros dos picos
2. Determinar as incertezas dos centros


X-Ray Apparatus
00:14:40
Bragg
Planck
Transmission
Moseley
nλ / pm
R0 / 1/s
24.6 2.8
25.6 2.8
26.6 2.8
27.6 3.0
28.5 3.2
29.5 1.8
30.5 2.8
31.5 3.6
32.5 5.6
33.4 4.4
34.4 4.0
35.4 6.0
36.4 4.0
37.4 6.6
38.4 10.2
39.3 24.4
40.3 55.2
41.3 85.8
42.3 131.4
43.3 168.4
44.3 205.4
45.2 247.8
46.2 278.0
47.2 324.8
48.2 369.4
49.2 391.6
50.1 410.6
51.1 449.0
52.1 457.2
53.1 465.4
54.1 502.8
55.0 495.2
56.0 529.6
57.0 514.8
58.0 546.0
59.0 531.6
59.9 529.4
60.9 608.2
61.9 798.6
62.9 1086.4
63.8 1100.0
64.8 779.4
65.8 574.4
66.8 533.8
67.8 529.4
68.7 734.4
69.7 1514.0
70.7 2196.2
R
1/s
2000
1000
0
0
50
100
150
nλ / pm
off
graus
Settings
X-Ray Apparatus
Crystal
General
Lattice-plane spacing 2d:
564.0 pm (NaCl)
Representation
Energy conversion
off
Show all orders
Close
Crystal calibration
Help
X-Ray Apparatus
U = 35.0 kV
I = 1.00 mA
Δt = 5 s
Δβ = 0.1 °
0 /s
40.0 °
20.0 °
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx7 - Paint
10:39


X-Ray Apparatus
00:14:40
Bragg
Planck
Transmission
Moseley
β / °
R0 / 1/s
R
1/s
15.3
56.4
15.4
56.0
Properties
Delete Column
Select Character Size
Display Coordinates Alt+C
Select Line Width
Show Values
Show Connecting Lines
Select Rulers
Show Grid
Logarithmic Representation
Zoom Alt+Z
Zoom Off Alt+O
Set Marker
Calculate Peak Center
Draw K-edge
Calculate Best-fit Straight Line
Calculate Straight Line through Origin
Calculate Integral
Delete Last Evaluation Alt+Rück
Delete All Evaluations
Copy Table
Copy Diagram
Copy Window
Settings
X-Ray Apparatus
Crystal
General
Goniometer
Sensor
Target
Coupled β= 20.0 °
Parameter
U= 35.0 kV
βmin= 2.5 °
Δβ= 0.1 °
I= 1.00 mA
βmax= 20.0 °
Δt= 5 s
Current actions
Scan
HV
Replay
Zero
Loudspeaker
Close
Crystal calibration
Help
X-Ray Apparatus
U = 35.0 kV
I = 1.00 mA
Δt = 5 s
Δβ = 0.1 °
0 /s
40.0 °
20.0 °
10
20
β / °
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx6 - Paint
10:37


X-Ray Apparatus
00:14:40
Bragg
Planck
Transmission
Moseley
nλ / pm
R0 / 1/s
R
1/s
26.6 2.8
27.6 3.0
28.5 3.2
29.5 1.8
30.5 2.8
31.5 3.6
32.5 5.6
33.4 4.4
34.4 4.0
35.4 6.0
36.4 4.0
37.4 6.6
38.4 10.2
39.3 24.4
40.3 55.2
41.3 85.8
42.3 131.4
43.3 168.4
44.3 205.4
45.2 247.8
46.2 278.0
47.2 324.8
48.2 369.4
49.2 391.6
50.1 410.6
51.1 449.0
52.1 457.2
53.1 465.4
54.1 502.8
55.0 495.2
56.0 529.6
57.0 514.8
58.0 546.0
59.0 531.6
59.9 529.4
60.9 608.2
61.9 798.6
62.9 1086.4
63.8 1100.0
64.8 779.4
65.8 574.4
66.8 533.8
67.8 529.4
68.7 734.4
69.7 1514.0
70.7 2196.2
71.7 2016.8
72.6 1081.2
2000
1000
0
0
50
150
nλ / pm
Settings
X-Ray Apparatus
Crystal
General
Lattice-plane spacing 2d: 564.0 pm (NaCl)
Representation
Energy conversion off
Show all orders
Close
Crystal calibration
Help
X-Ray Apparatus
U = 35.0 kV
I = 1.00 mA
Δt = 5 s
Δβ = 0.1 °
0 /s
40.0 °
20.0 °
Properties
Delete Column
Select Character Size
Display Coordinates Alt+C
Select Line Width
Show Values
Show Connecting Lines
Select Rulers
Show Grid
Logarithmic Representation
Zoom Alt+Z
Zoom Off Alt+O
Set Marker
Calculate Peak Center
Draw K-edge
Calculate Best-fit Straight Line
Calculate Straight Line through Origin
Calculate Integral
Delete Last Evaluation Alt+Rück
Delete All Evaluations
Copy Table
Copy Diagram
Copy Window
© by LD Didactic GmbH, 1998-2008
Iniciar
X-Ray Apparatus
rx8 - Paint
10:40


X-Ray Apparatus
00:14:40
Bragg
Planck
Transmission
Moseley
nλ / pm
R0 / 1/s
26.6 2.8
27.6 3.0
28.5 3.2
29.5 1.8
30.5 2.8
31.5 3.6
32.5 5.6
33.4 4.4
34.4 4.0
35.4 6.0
36.4 4.0
37.4 6.6
38.4 10.2
39.3 24.4
40.3 55.2
41.3 85.8
42.3 131.4
43.3 168.4
44.3 205.4
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51.1 449.0
52.1 457.2
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54.1 502.8
55.0 495.2
56.0 529.6
57.0 514.8
58.0 546.0
59.0 531.6
59.9 529.4
60.9 608.2
61.9 798.6
62.9 1086.4
63.8 1100.0
64.8 779.4
65.8 574.4
66.8 533.8
67.8 529.4
68.7 734.4
69.7 1514.0
70.7 2196.2
71.7 2016.8
72.6 1081.2
R
1/s
2000
1000
0
0
50
100
150
nλ / pm
Settings
X-Ray Apparatus
Crystal
General
Lattice-plane spacing 2d:
Representation
Energy conversion
off
Show all orders
Close
Crystal calibration
Help
X-Ray Apparatus
U = 35.0 kV
I = 1.00 mA
Δt = 5 s
Δβ = 0.1 °
0 /s
40.0 °
20.0 °
Valor do ângulo
Incerteza do ângulo
nλ = 70.93 pm, σ = 1.01 pm
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Iniciar
X-Ray Apparatus
rx9 - Paint
10:41

# Cálculos

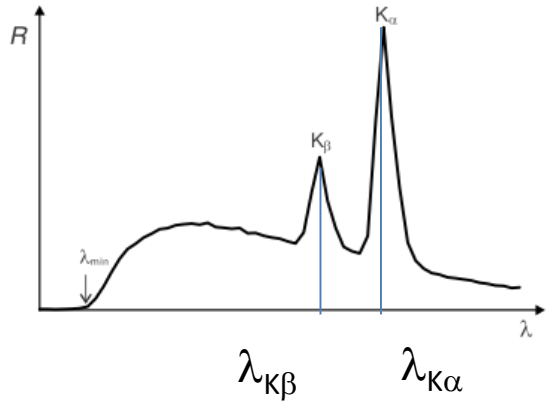
- **Cálculo da distância interatômica d**
  - Para cada valor do ângulo q determinado (respectivamente, para $K_\alpha$ e $K_\beta$) pode ser calculado o valor de d , usando a lei de Bragg
    - Lei de Bragg: $n\lambda = 2 . d \sin(\theta)$

- Os *dois valores* de d precisam ser comparados
  - Para isso, vai precisar das incertezas nos valores dos ângulos $\theta$
  - Elas *precisam ser propagadas* para os valores de d
  - Usar o método de propagação de intertezad apresentado no link
    - Propagação de erros
  - Você tem de *deduzir* a expressão de $\Delta d$ a partir da de $\Delta\theta$
    - Deve apresentar sua dedução em seu relatório
  - De posse da formula que assim deduzir, poderá calcular os respectivos valores de d + $\Delta d$
    - Cuidado: precisa converter a incerteza em $\theta$ para radianos !!

## Valores dos comprimentos de onda das transições K do Mo

| | $\frac{E}{\text{keV}}$ | $\frac{\nu}{\text{EHz}}$ | $\frac{\lambda}{\text{pm}}$ |
|---|---|---|---|
| $K_\alpha$ | 17.443 | 4.2264 | 71.080 |
| $K_\beta$ | 19.651 | 4.8287 | 63.095 |

$\text{keV} = 10^3\ \text{eV}$, $\text{EHz} = 10^{18}\ \text{Hz}$, $\text{pm} = 10^{-12}\ \text{m}$



A duração aproximada da tomada de dados do primeiro experimento é de 90 minutos ( 3 sessões de 30 minutos, uma para cada valor de U ).

Após a tomada de dados, realizar as etapas de determinação dos ângulos $\theta$ para os quais ocorrem os picos $K_\alpha$ e $K_\beta$, com suas respectivas incertezas.

O cálculo do valor da distância interatômica d empregando a lei de Bragg

$n\lambda = 2 \cdot d \sin(\theta)$

para $K_\alpha$ e $K_\beta$, usando os respectivos valores de $\theta$ encontrados para cada caso podem ser feitos aqui no Lab

Os cálculos das incertezas em d devem ser feitos em casa (aula de sábado)


Universidade de São Paulo
Instituto de Física
Laboratório de Física Moderna

Disciplina: Física Experimental C - 4323301
Coordenador: José Helder Facundo Severo

# Experimento: Difração de Raio-X

Edição 2020 – EAD

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