Estrutura de materiais

Difratometria de raios X

Prof. Paulo A. Suzuki

(DEMAR - EEL - USP)

Difração: λ ~ d

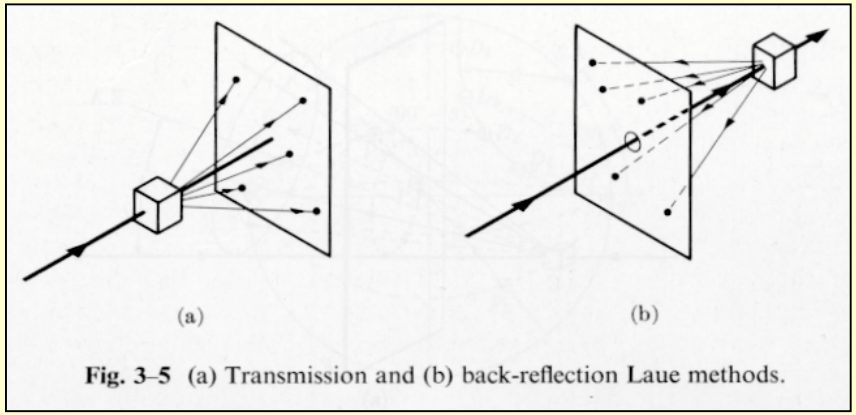


Fig. 3–5 (a) Transmission and (b) back-reflection Laue methods.

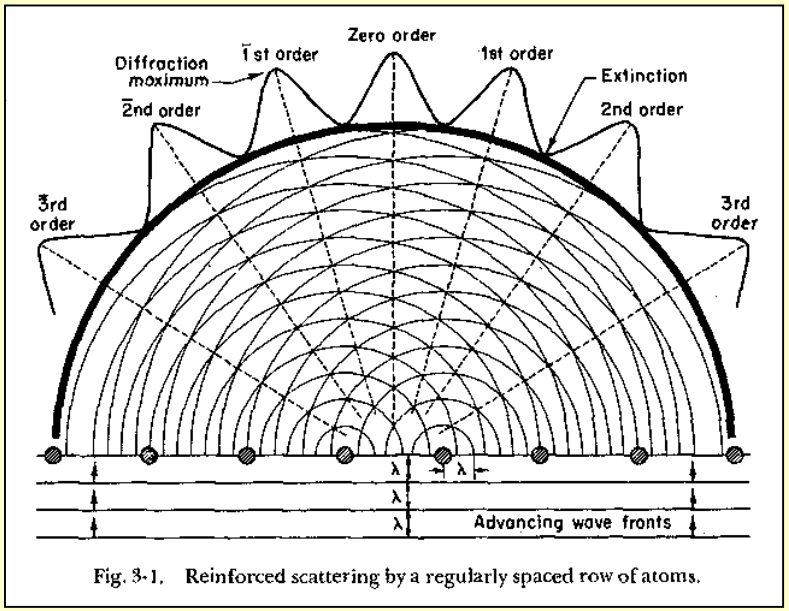
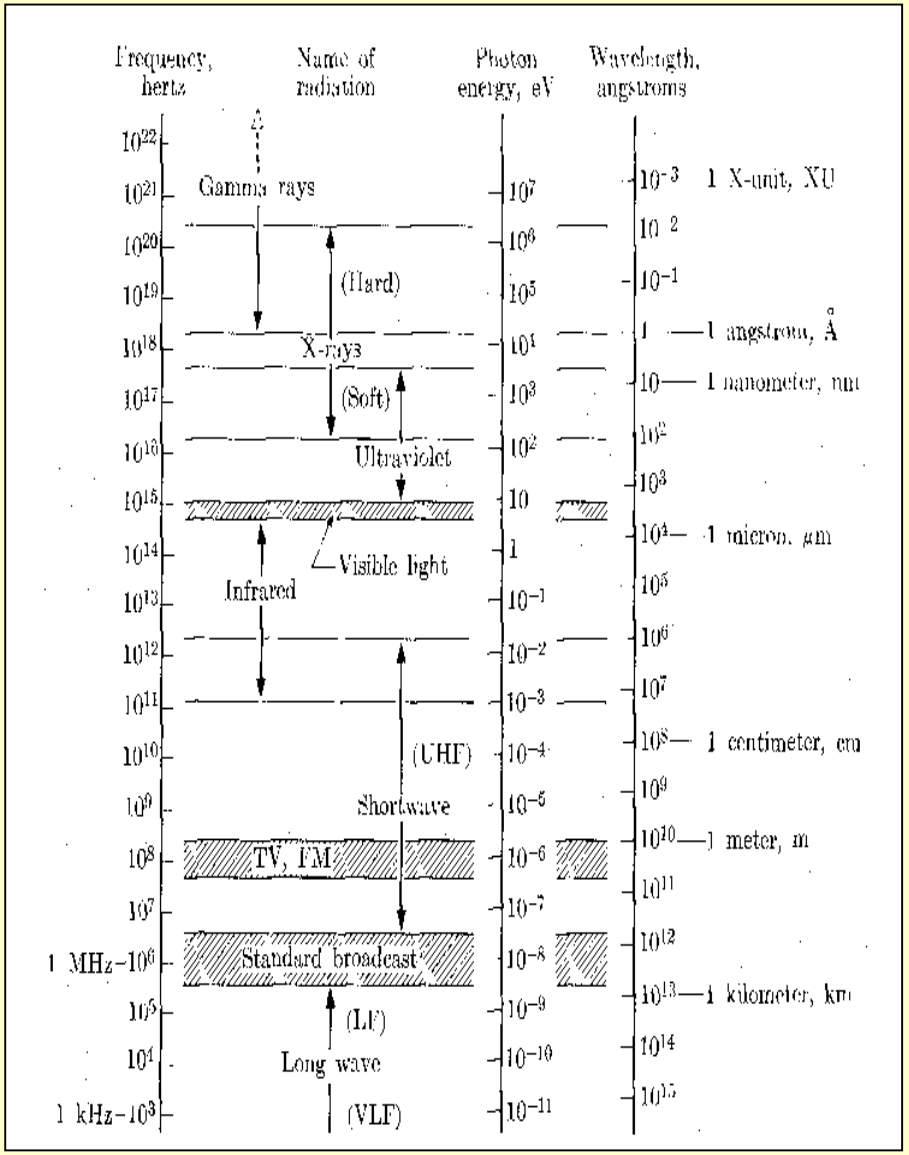
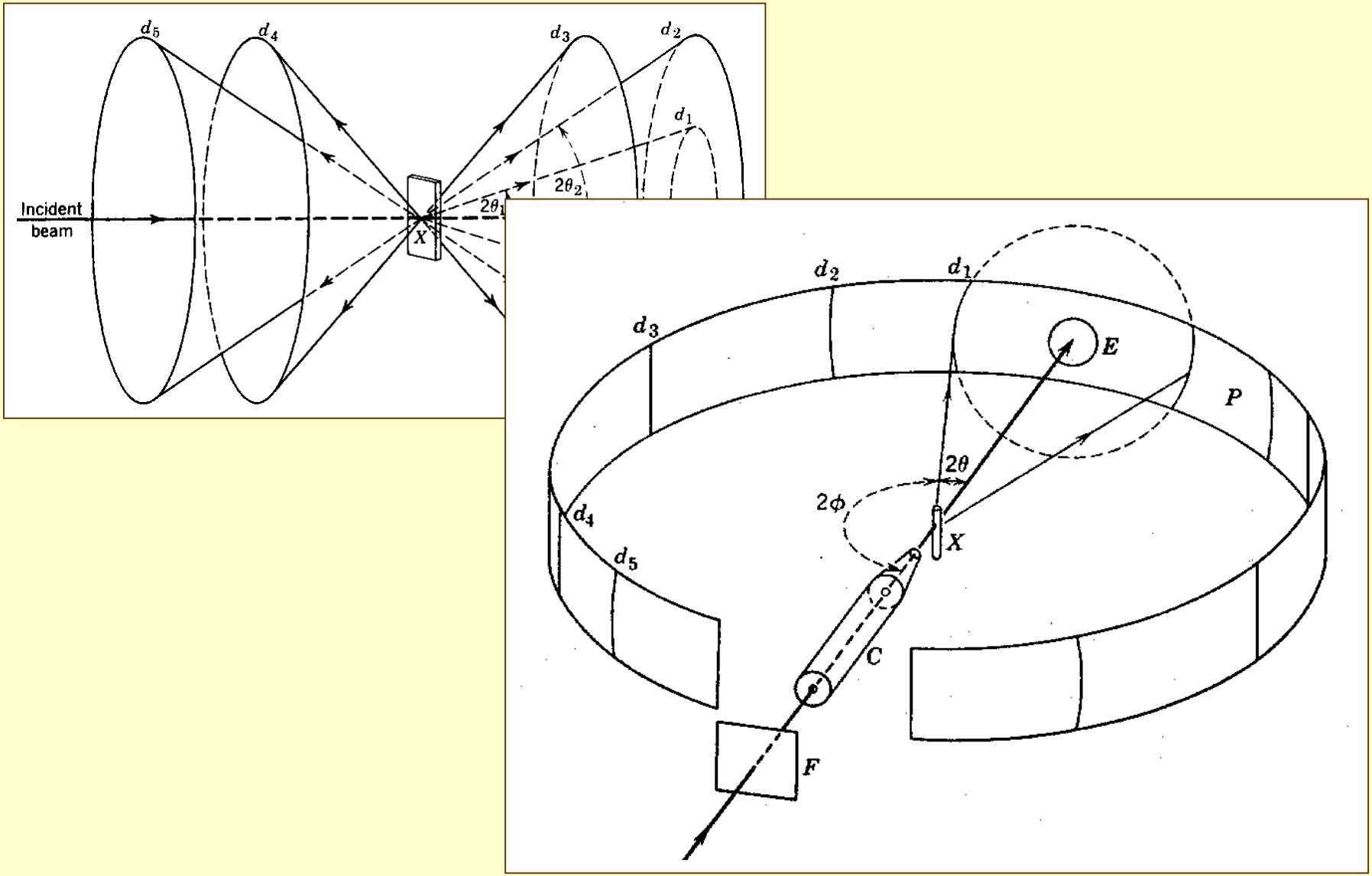


Fig. 3-1. Reinforced scattering by a regularly spaced row of atoms.

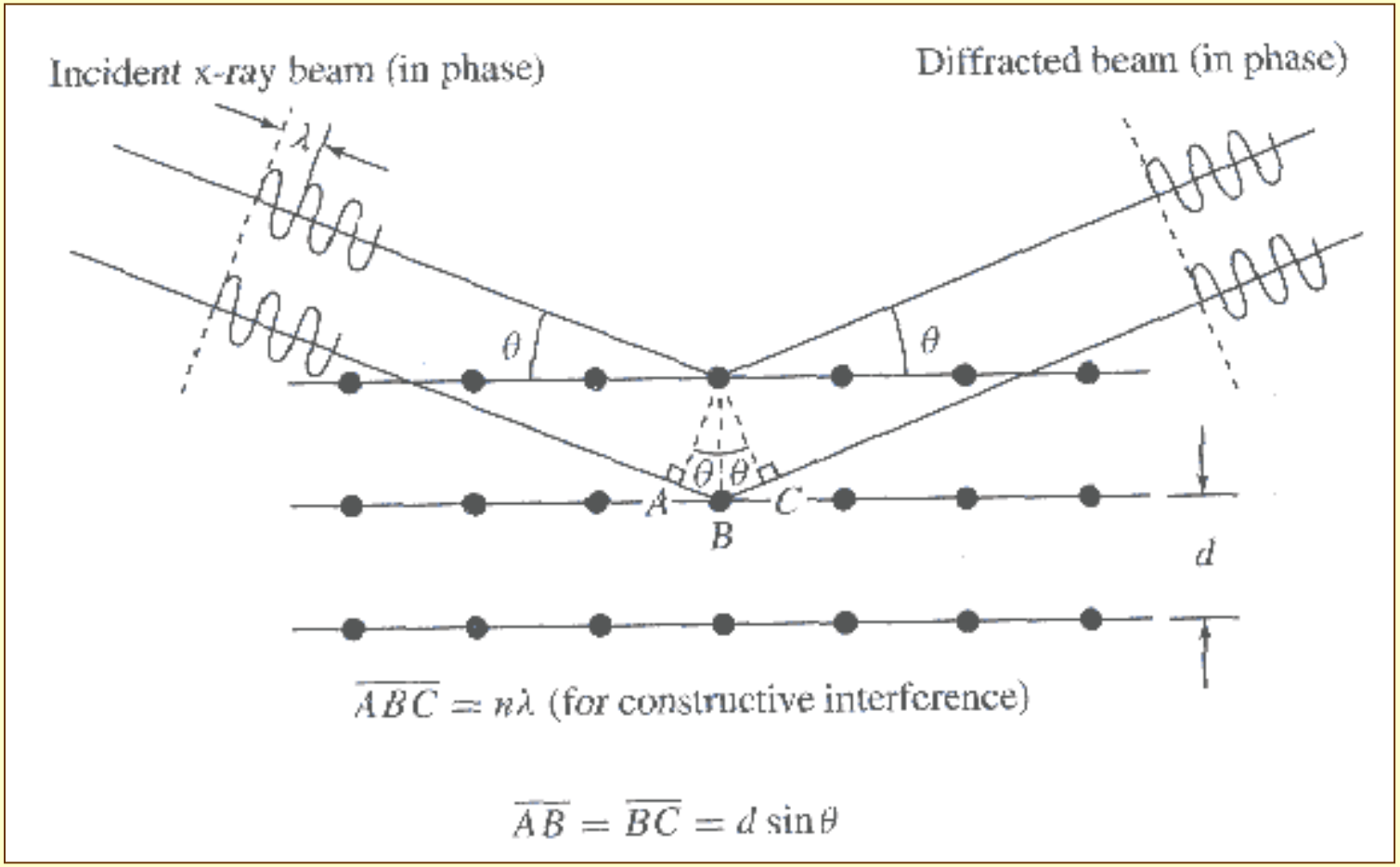


Câmara de Debye-Scherrer

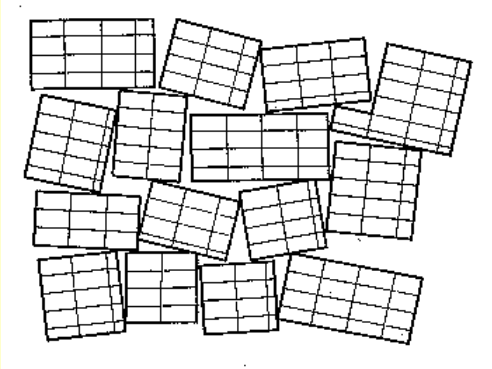


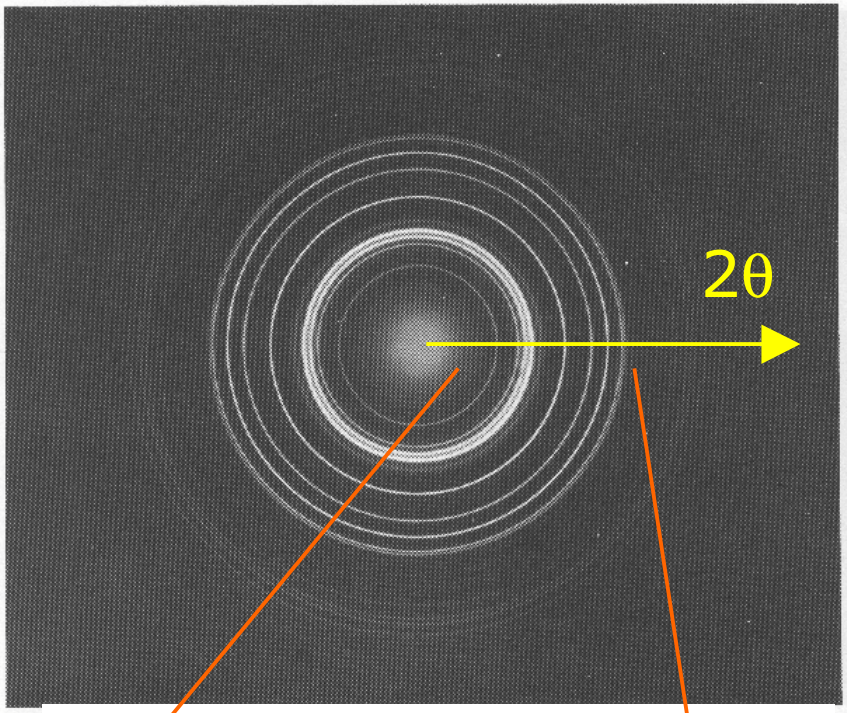
Lei de Bragg

$n.\lambda = 2\ d.\text{sen}\ \theta$



policristal





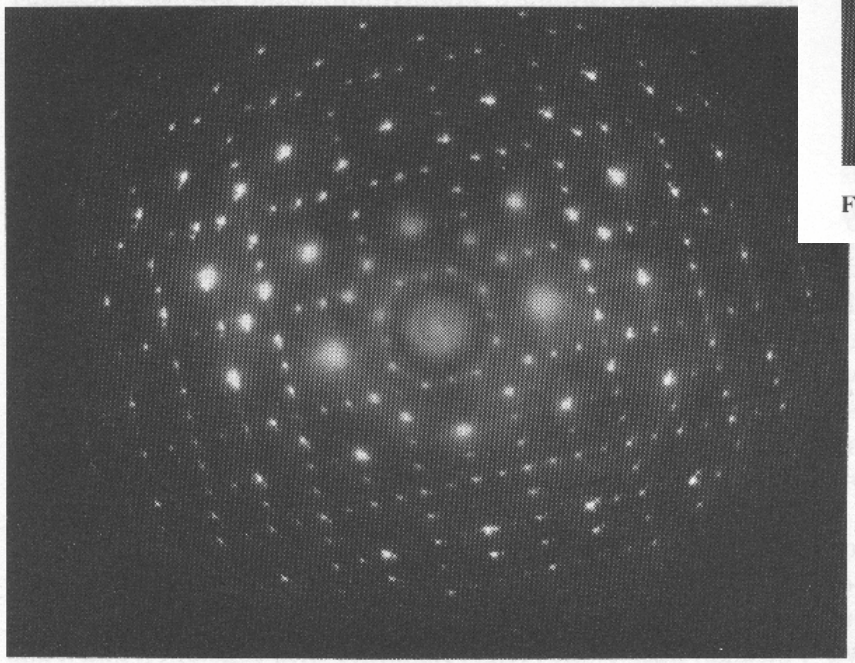
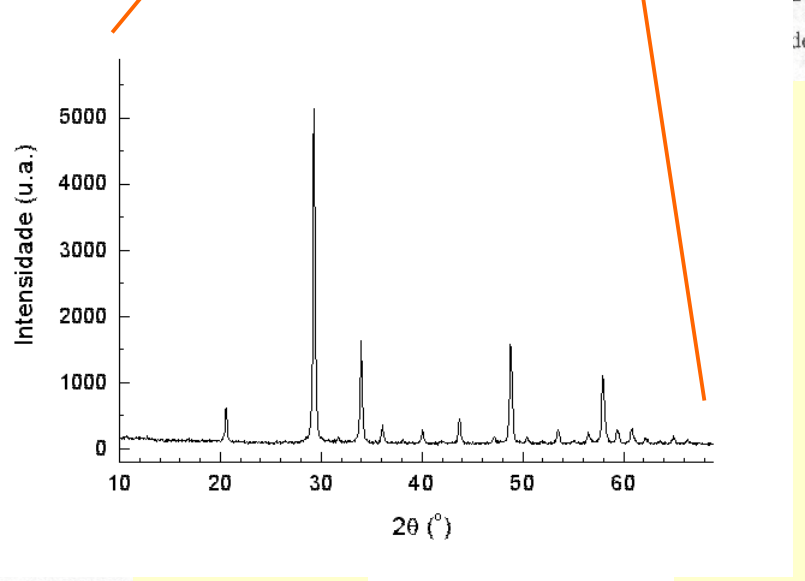
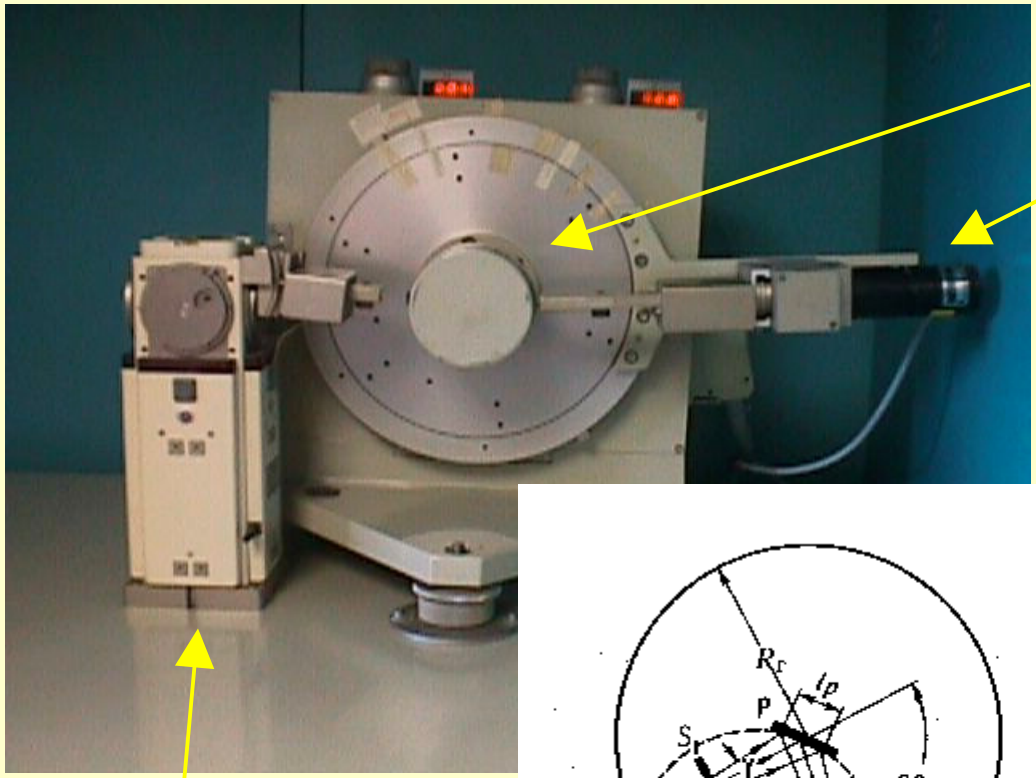


Fig. 4.81. Electron diffraction spot pattern from a mosaic crystal of $BaCl_2 \cdot H_2O$ (a
voltage 60 kV, $L = 700$ mm) [4.81]



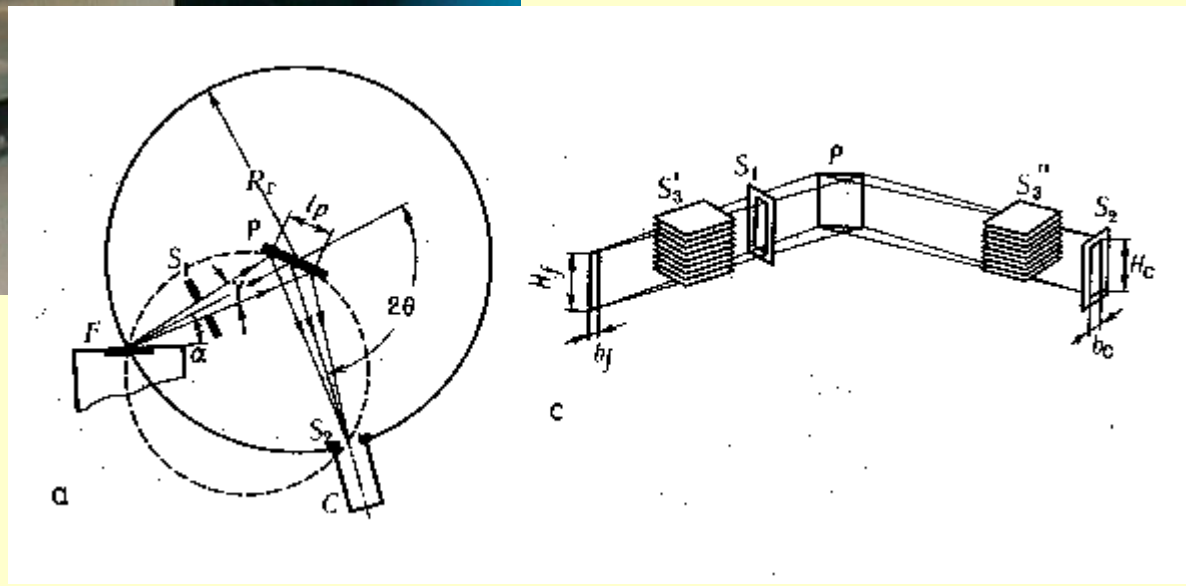
Difratômetro de raios X



Suporte de amostras

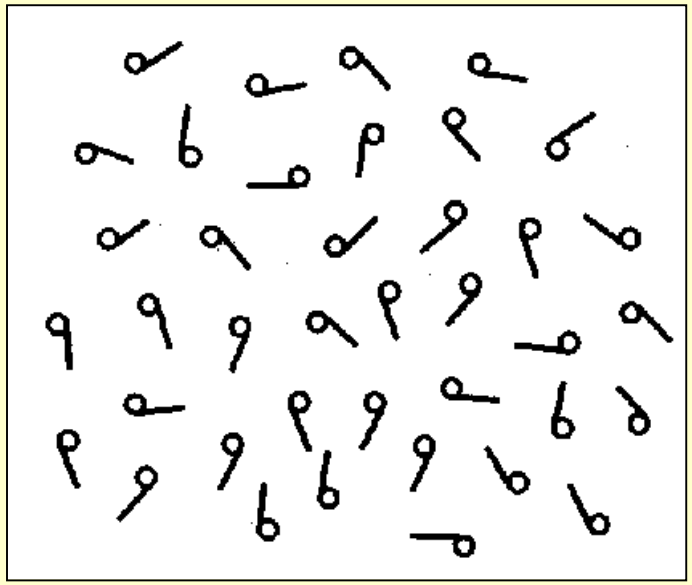
detetor de raios X

tubo de raios X

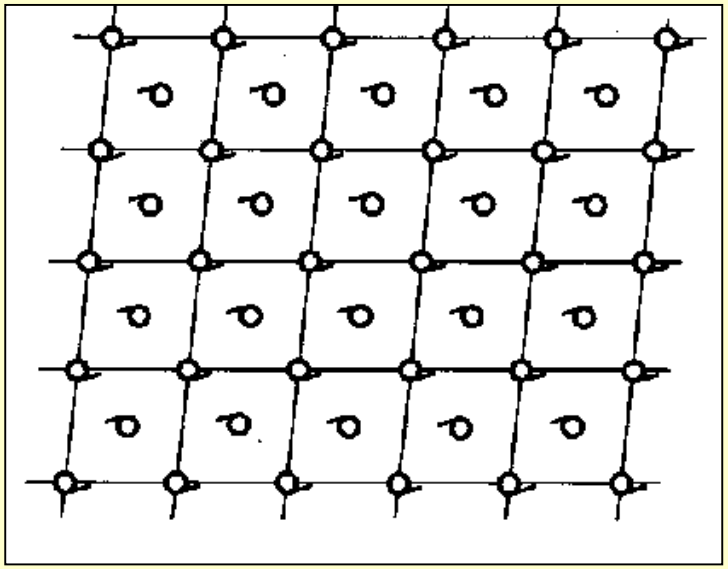


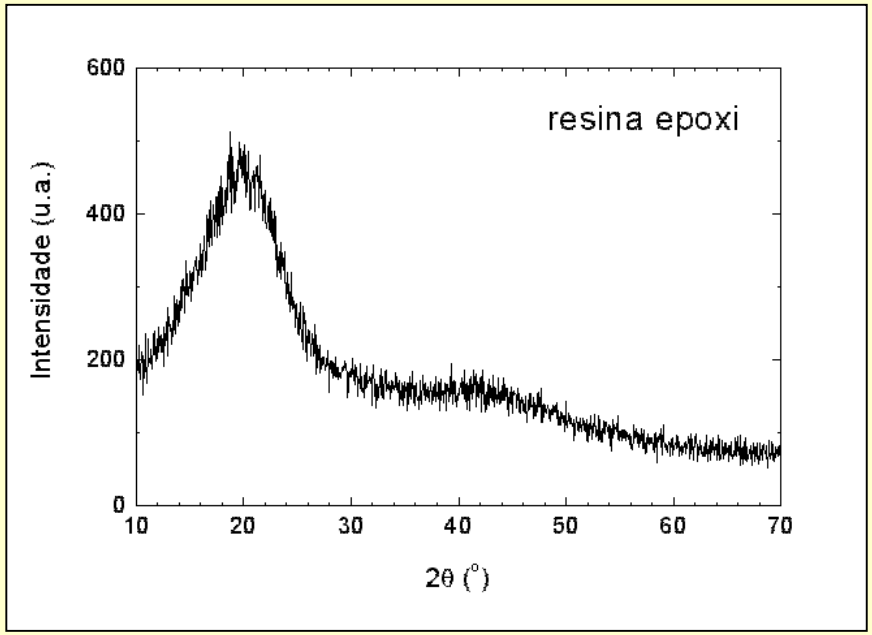
Geometria de Bragg-Bretano

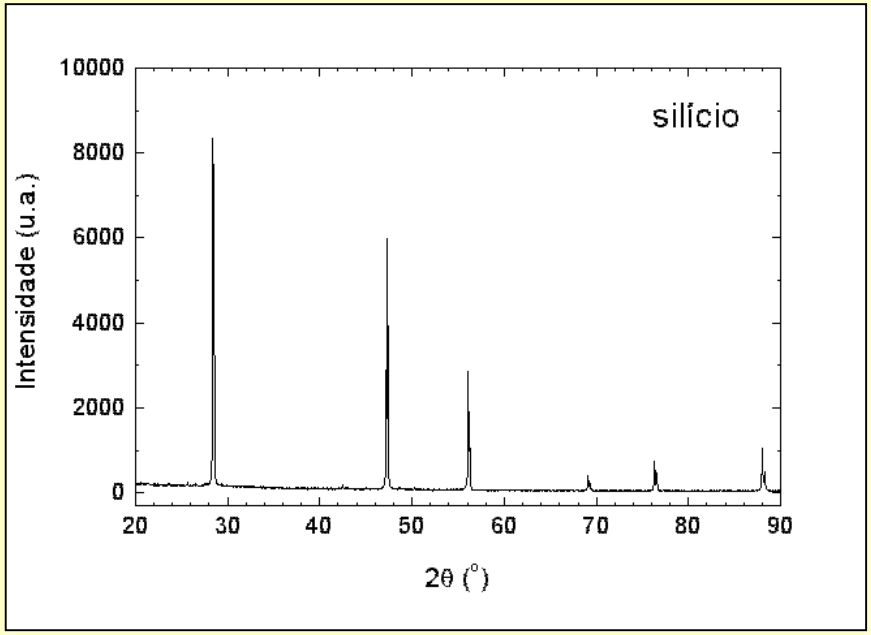
amorfo



cristalino

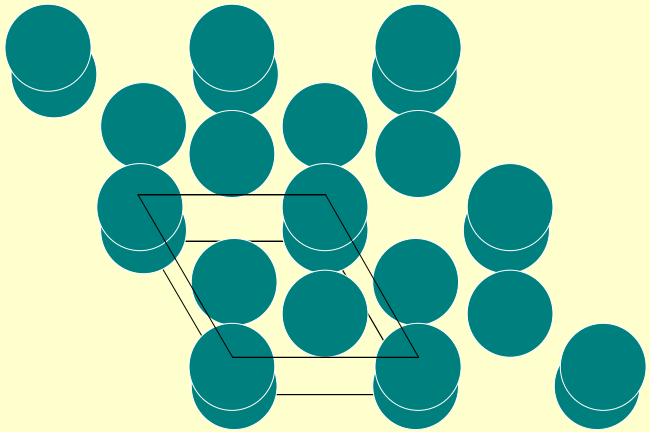


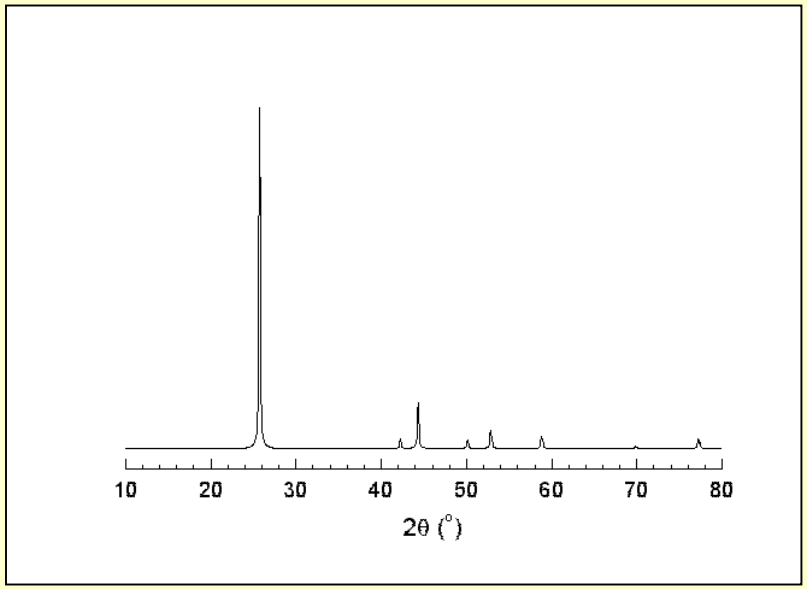




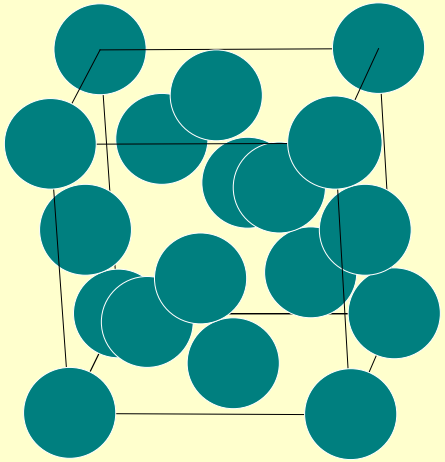
Carbono

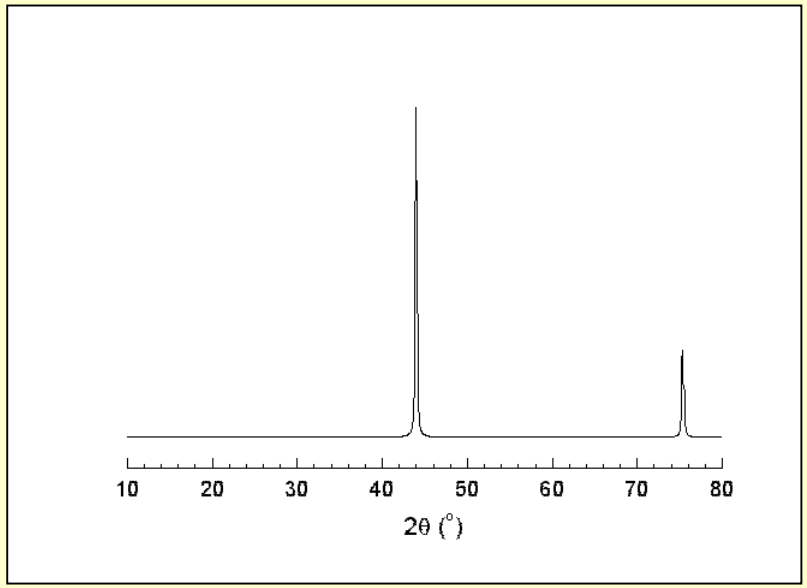
grafite





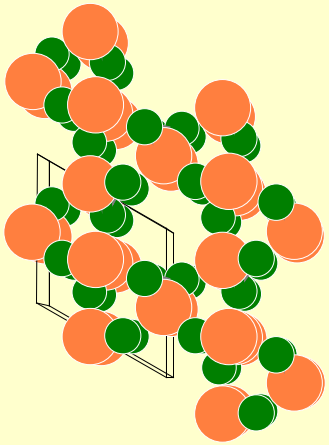
diamante



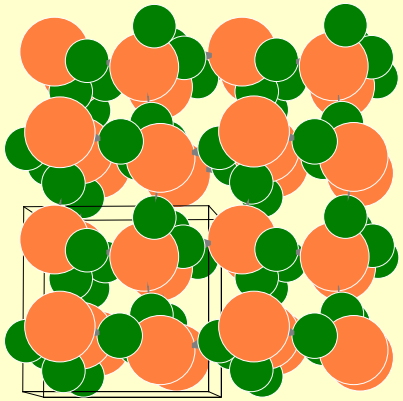


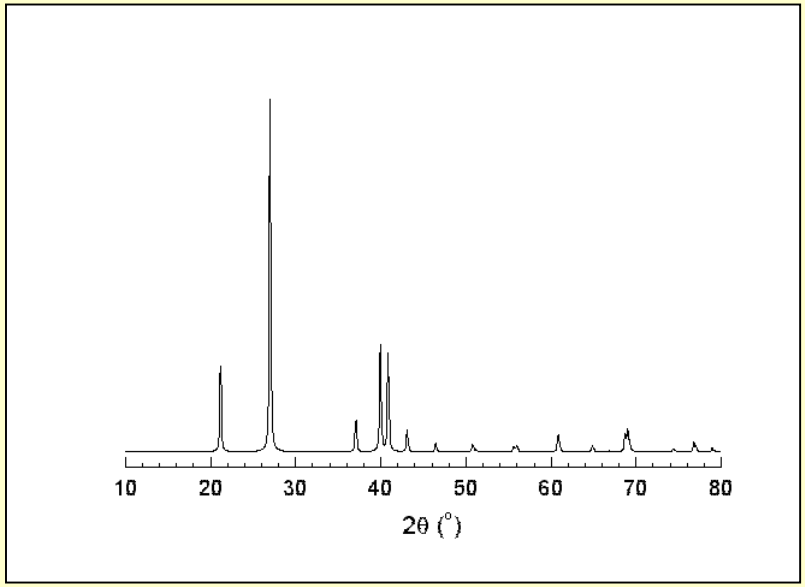
SiO_2

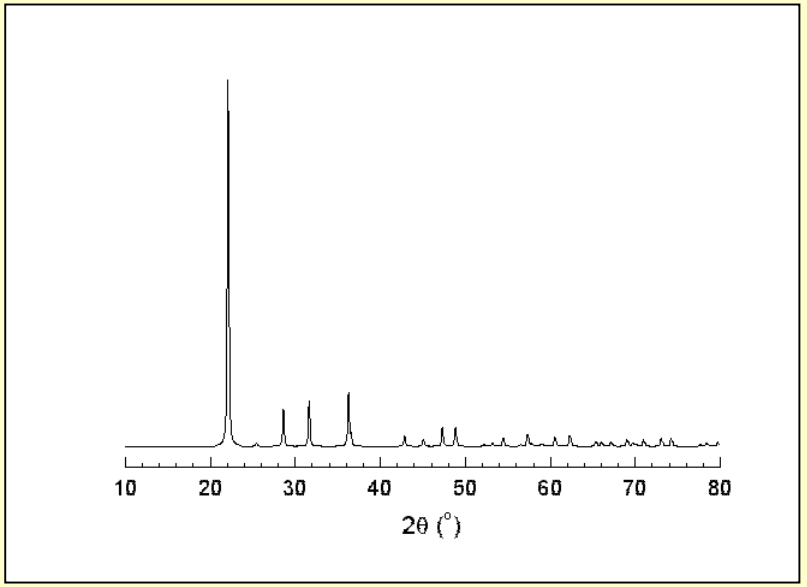
quartzo

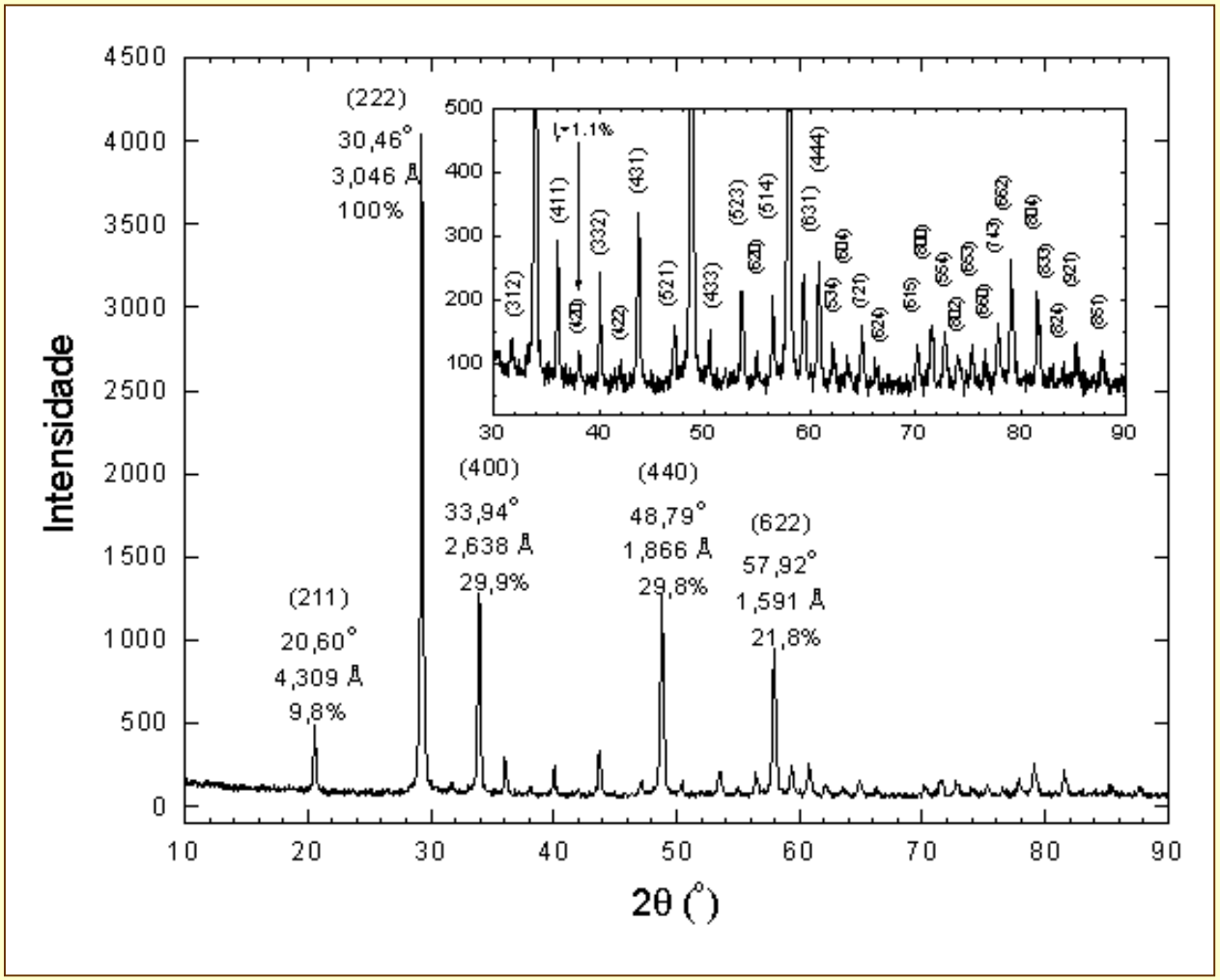


cristobalita









d (Å)	I (%)
3,046	100
2,638	30
1,866	30
1,591	22
4,309	10

Método Hanawalt

d (Å)	I (%)
3,046	100
2,638	30
1,866	30
1,591	22
4,309	10

3.04 – 3.00 (±.01)

										File No.
	3.04_X	1.87_X	1.59_X	1.22_9	1.08_9	1.19_7	2.64_6	1.02_6	Dy_6MoO_{12}	28–1284
o	3.04_X	1.87_X	1.59_5	1.08_3	2.77_1	1.82_1	1.66_1	1.32_1	Cu_3AsS_4/Arsenosulvanite	25– 265
c	3.03_X	1.87_3	3.85_3	2.09_3	2.28_2	1.91_2	1.60_2	2.50_1	$CaCO_3$/Calcite	24– 27
*	3.03_X	1.87_3	2.58_2	1.56_2	2.72_1	1.83_1	1.62_1	2.18_1	T'-$YNbO_4$	38– 187
	3.03_X	1.87_9	1.59_7	2.63_4	1.56_3	1.22_3	2.08_2	1.19_2	GeY_8O_{14}	23– 271
o	3.02_X	1.87_X	3.87_8	2.55_8	2.13_8	1.95_8	1.64_8	1.55_8	$Ag_{0.8}Na_{0.2}NO_3$	32–1021
i	3.02_X	1.87_5	3.67_3	2.75_3	2.00_3	1.67_2	1.91_2	1.74_2	Cu_4SnS_6	36– 53
	3.02_X	1.87_X	1.60_X	2.63_8	1.21_8	1.19_8	1.08_8	1.02_8	$PbSmZrNbO_7$	30– 722
	3.01_X	1.87_3	4.61_2	2.51_2	1.53_2	2.10_2	1.66_2	2.80_1	$HoVO_4$	16– 482
i	3.01_X	1.87_6	3.66_4	2.00_3	2.75_2	1.67_2	1.59_2	2.91_2	$Cu_{9.67}Sn_{2.33}S_{13}$	33– 502
*	3.01_X	1.87_7	1.84_7	2.62_5	3.09_4	1.58_2	3.12_2	1.57_1	Er_6WO_{12}	23–1068
*	3.05_X	1.86_6	2.64_4	1.59_3	4.31_2	2.07_1	2.49_1	1.71_1	Er_2O_3	8– 50
	3.05_X	1.86_7	2.64_5	1.59_5	3.73_4	2.07_4	1.71_4	1.08_4	$(Cu,Ag,Zn)_{12}Sb_{4.4}S_{12.6}$/Freibergite, syn	27– 190
i	3.05_X	1.86_2	1.87_2	2.63_2	1.59_1	2.65_1	1.60_1	1.21_1	$Zr_{0.5}Ce_{0.5}O_2$	38–1436
	3.05_X	1.86_9	1.59_7	1.58_6	1.20_6	1.08_6	1.32_5	1.07_5	Cu_3AsS_4/Luzonite, syn	10– 450
i	3.04_X	1.86_3	2.65_3	3.54_2	1.87_2	1.59_2	2.61_1	7.71_1	α-Tb_2TiO_5	34–1309
	3.04_X	1.86_2	2.63_2	1.59_1	1.37_1	6.07_1	3.16_1	2.41_1	$Pb_2Nb_2TiO_9$	19– 692
i	3.04_X	1.86_4	2.63_3	1.59_3	1.21_1	1.52_1	1.18_1	1.32_1	$Eu_2Zr_2O_7$	24– 418
o	3.04_X	1.86_5	1.87_4	1.59_4	2.63_2	1.52_1	1.21_1	1.18_1	$Ho_{10}Mo_2O_{21}$	24– 475
*	3.04_X	1.86_3	1.87_2	1.59_1	1.58_1	2.65_1	2.61_1	1.21_1	$CuFeS_2$/Chalcopyrite	37– 471
	3.04_X	1.86_X	1.60_6	1.88_5	1.57_3	1.08_3	1.21_3	1.33_2	$CuAlS_2$	25– 14
	3.04_X	1.86_6	1.59_6	2.63_4	1.21_2	1.18_2	1.01_2	1.51_1	$Eu_2Hf_2O_7$	24– 410
i	3.04_X	1.86_5	1.59_4	2.63_4	1.21_1	1.52_1	1.18_1	1.07_1	$Gd_2Hf_2O_7$	24– 425
i	3.04_X	1.86_5	1.59_5	2.63_4	1.21_2	1.18_2	0.89_2	1.52_1	$(Cd_{0.5}Pb_{0.5})_2Nb_2O_7$	33– 240
i	3.04_X	1.86_6	1.59_4	2.63_3	1.21_1	1.52_1	1.31_1	6.05_1	Y_3TaO_7	38–1403
*	3.04_X	1.86_4	1.59_3	1.87_2	1.58_1	2.64_1	1.21_1	1.08_1	$CuFeS_2$/Chalcopyrite	35– 752
i	3.04_X	1.86_7	1.59_5	1.32_2	2.63_1	4.30_1	5.25_1	1.52_1	$Cu_3(As,V)S_4$/Arsenosulvanite, syn	35–1017
o	3.04_X	1.86_X	1.59_X	1.01_X	3.17_9	1.37_9	1.02_9	2.02_8	$(Cs,Na)SbTa_4O_{12}$/Cesstibtantite	35– 672
	3.04_X	1.86_5	1.58_4	2.63_4	1.21_2	0.89_1	1.18_1	1.07_1	$Sm_2Sn_2O_7$	13– 181
	3.03_X	1.86_X	2.62_8	2.41_6	6.09_4	2.02_4	3.17_2	1.78_2	$Gd_2Zr_2O_7$	16– 799

Fichas JCPDS - ICDD

8-50 MINOR CORRECTION

d	3.05	1.86	2.64	4.31	$(Er_2O_3)80\underline{B}$
I/I_1	100	45	40	16	ERBIUM OXIDE

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni Dia.
Cut off I/I_1 DIFFRACTOMETER
Ref. NBS CIRCULAR 539, 8 25 (1959)

Sys. CUBIC S.G. Ia3 (206)
a_o 10.548 b_o c_o A C
α β γ Z 16 Dx8.658
Ref. IBID.

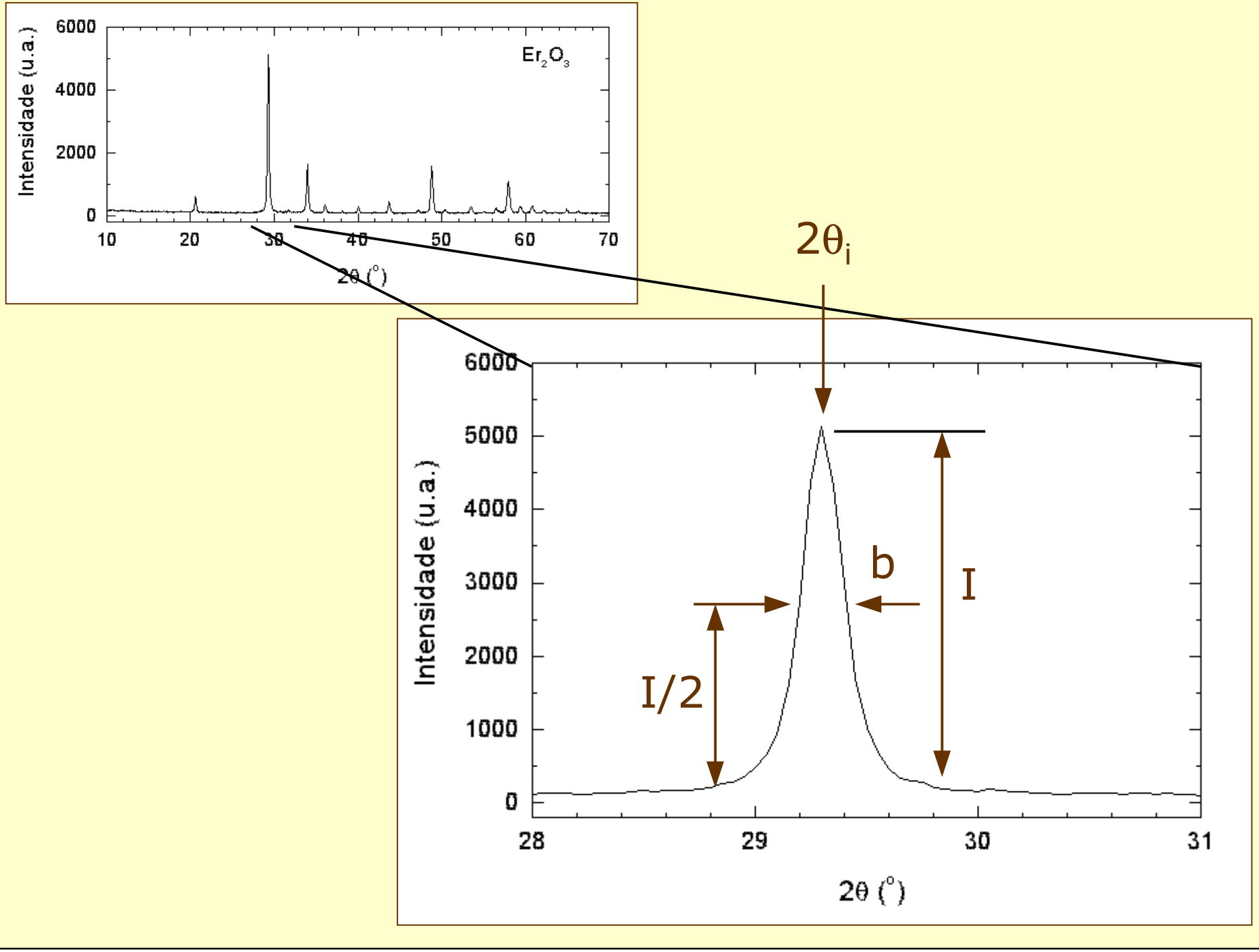
ξ α 1.932 n ω β ξ γ Sign
2V D mp Color PINK
Ref. IBID.

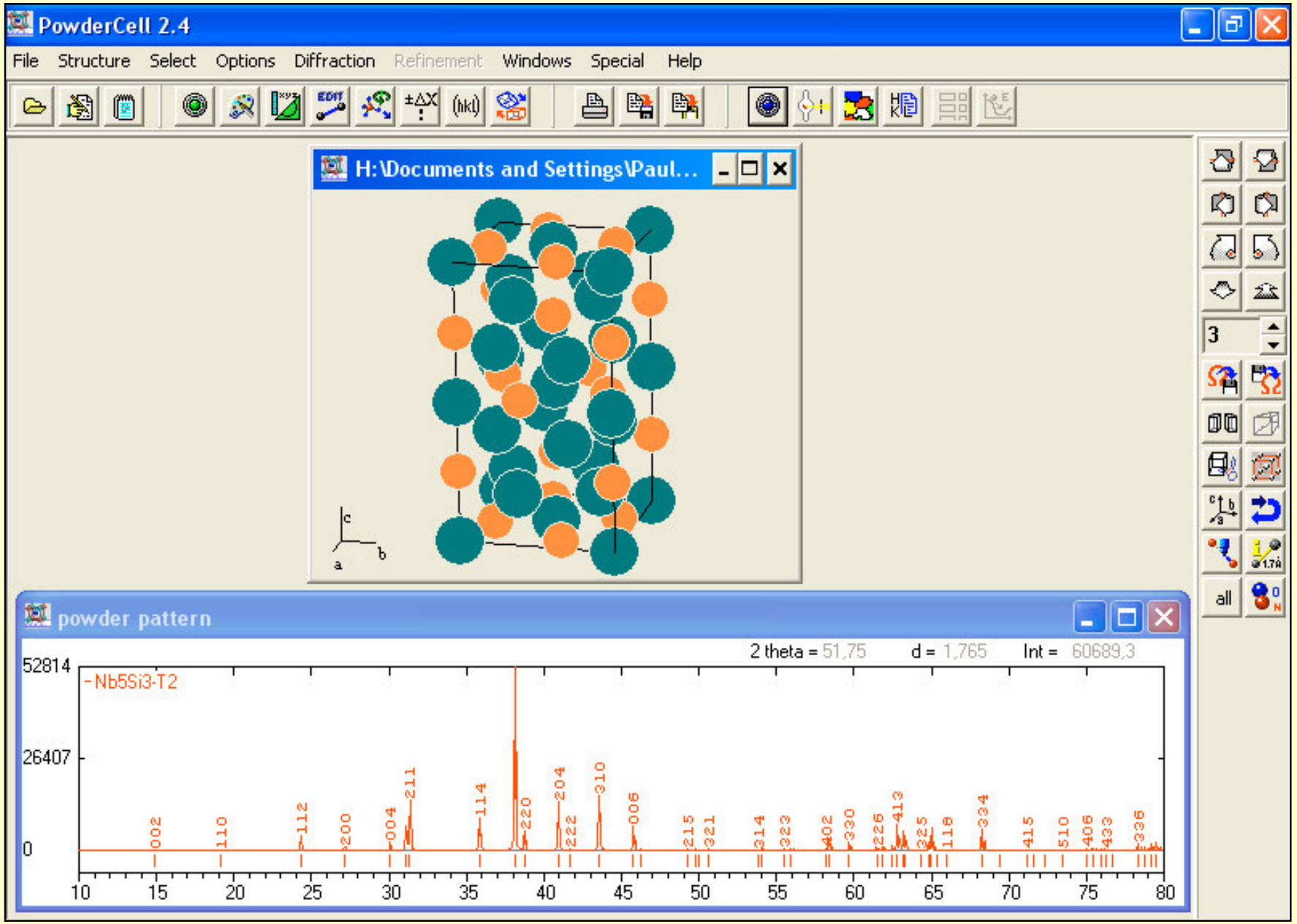
SAMPLE PREPARED AT THE NBS AND ANNEALED AT 1000°C FOR TWO HOURS. SPECT. ANAL. SHOWED IMPURITIES <0.01% . Tl_2O_3 STRUCTURE TYPE. PATTERN MADE AT 25°C. MERCK INDEX, 8TH ED., P. 413.

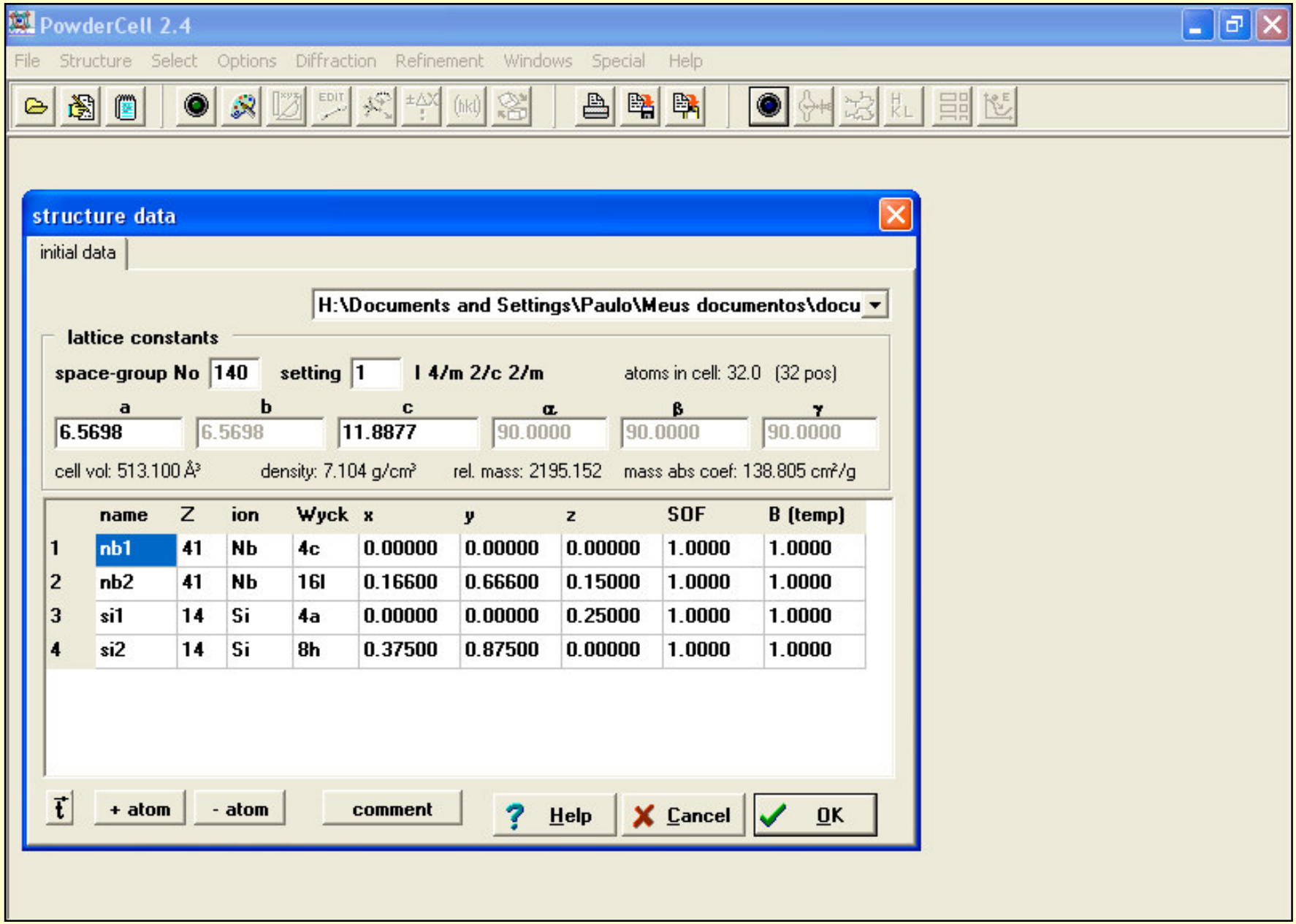
SEE FOLLOWING CARD

d Å	I/I_1	hkl	d Å	I/I_1	hkl
4.308	16	211	1.4629	2	640
3.045	100	222	1.4359	4	721
2.818	4	321	1.4099	2	642
2.637	40	400	1.3398	4	732
2.486	8	411	1.3189	6	800
2.3575	2	420	1.2988	4	811
2.2488	6	332	1.2795	2	820
2.1532	2	422	1.2608	4	653
2.0687	10	431	1.2436	2	822
1.9258	4	521	1.2266	4	831
1.8645	45	440	1.2102	8	662
1.8094	4	433	1.1794	6	840
1.7590	2	600	1.1650	2	833
1.7113	8	611	1.1511	2	842
1.6681	2	620	1.1377	4	921
1.6276	6	541	1.1119	4	851
1.5903	30	622	1.0883	4	932
1.5558	8	631	1.0768	4	844
1.5228	8	444	1.0656	4	941
1.4918	4	543	1.0547	2	1000

						File No.
i	Erbium Nickel : Boron	$B_2Er_3Ni_7$	2.38_x	2.06_9	2.16_5	34– 438
i	Erbium Niobium Oxide :	$ErNbO_4$	3.12_x	2.95_9	1.89_5	22–1095†
	Erbium Niobium Oxide :	$ErNbO_4$	3.11_x	2.94_x	1.89_x	24– 408
i	Erbium Niobium Oxide :	Er_3NbO_7	3.03_x	1.85_6	1.58_4	24–1080†
*	Erbium Niobium Oxide : Barium	Ba_2ErNbO_6	2.98_x	1.72_5	2.11_4	24–1051†
o	Erbium Nitrate Hydrate : Potassium	$K_2Er(NO_3)_5{\cdot}2H_2O$	6.42_x	5.13_9	4.44_6	32– 777
c	Erbium Nitride :	ErN	2.79_x	2.42_7	1.71_4	15– 885
	Erbium Osmium :	$ErOs_2$	2.26_x	2.21_x	2.45_8	19– 451
	Erbium Oxalate Hydrate :	$C_6Er_2O_{12}{\cdot}18H_2O$	5.10_x	8.84_9	4.82_4	28– 411
*	Erbium Oxide :	Er_2O_3	3.05_x	1.86_5	2.64_4	8– 50
i	Erbium Oxide :	Er_2O_3	2.98_x	1.82_6	1.56_6	26– 604
	Erbium Oxide :	Er_2O_3	2.88_x	3.02_3	1.51_2	19– 452
i	Erbium Oxide : Aluminum	$Er_4Al_2O_9$	2.90_x	3.00_x	4.67_9	32– 13
i	Erbium Oxide : Aluminum	$Al_{10}Er_6O_{24}$	2.68_x	3.21_5	4.90_5	32– 12
*	Erbium Oxide : Aluminum	$ErAlO_3$	2.61_x	3.71_5	2.58_3	24– 396
i	Erbium Oxide : Antimony	$SbErO_3$	3.09_x	1.90_5	2.68_5	26–1021
*	Erbium Oxide : Bismuth	$0.8Bi_2O_3{\cdot}0.2Er_2O_3$	3.18_x	2.75_4	1.66_4	34– 377†
o	Erbium Oxide : Bismuth	$Bi_6Er_2O_{12}$	3.16_x	2.74_6	1.93_6	30– 182
	Erbium Oxide : Bismuth	$BiErO_3$	3.10_x	1.91_5	2.70_5	27–1041
	Erbium Oxide Bromide :	$ErOBr$	2.80_x	2.70_7	8.27_4	18– 488
	Erbium Oxide : Calcium	$CaEr_2O_4$	2.89_x	2.83_5	4.96_3	28– 709
i	Erbium Oxide : Calcium Aluminum	$CaErAlO_4$	2.67_x	2.58_8	3.48_5	24– 189
i	Erbium Oxide Chloride :	Er_3O_4Cl	3.20_x	2.70_8	5.79_6	31– 502
i	Erbium Oxide : Chromium	$ErCrO_3$	2.67_x	2.61_3	2.75_2	25–1052†
*	Erbium Oxide : Copper	$Cu_2Er_2O_5$	2.84_x	2.69_8	2.66_6	33– 456


Intensidade (u.a.)
6000
4000
2000
0
10
20
30
40
50
60
70
2θ (°)
Er2O3
2θi
5000
3000
1000
28
29
30
31
b
I
I/2


PowderCell 2.4
File Structure Select Options Diffraction Refinement Windows Special Help
H:\Documents and Settings\Paul...
c
a
b
powder pattern
2 theta = 51,75 d = 1,765 Int = 60689,3
52814
26407
0
-Nb5Si3-T2
002 110 112 200 004 211 114 220 204 222 310 006 215 321 314 323 402 330 226 413 325 118 334 415 510 406 433 336
10 15 20 25 30 35 40 45 50 55 60 65 70 75 80
all


PowderCell 2.4
File Structure Select Options Diffraction Refinement Windows Special Help
structure data
initial data
H:\Documents and Settings\Paulo\Meus documentos\docu
lattice constants
space-group No 140 setting 1 I 4/m 2/c 2/m atoms in cell: 32.0 (32 pos)
a b c α β γ
6.5698 6.5698 11.8877 90.0000 90.0000 90.0000
cell vol: 513.100 Å³ density: 7.104 g/cm³ rel. mass: 2195.152 mass abs coef: 138.805 cm²/g
name Z ion Wyck x y z SOF B (temp)
1 nb1 41 Nb 4c 0.00000 0.00000 0.00000 1.0000 1.0000
2 nb2 41 Nb 16l 0.16600 0.66600 0.15000 1.0000 1.0000
3 si1 14 Si 4a 0.00000 0.00000 0.25000 1.0000 1.0000
4 si2 14 Si 8h 0.37500 0.87500 0.00000 1.0000 1.0000
+ atom
- atom
comment
Help
Cancel
OK

Sites interessantes:

http://www.iucr.org - International Union of Crystallography

http://www.fisica.ufc.br/raiox/ - Cristalografia/Difração de raios X

http://labcacc.iq.unesp.br - Método de Rietveld

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- H. P. Klug e L. E. Alexander, X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials, John Wiley, New York (1974).
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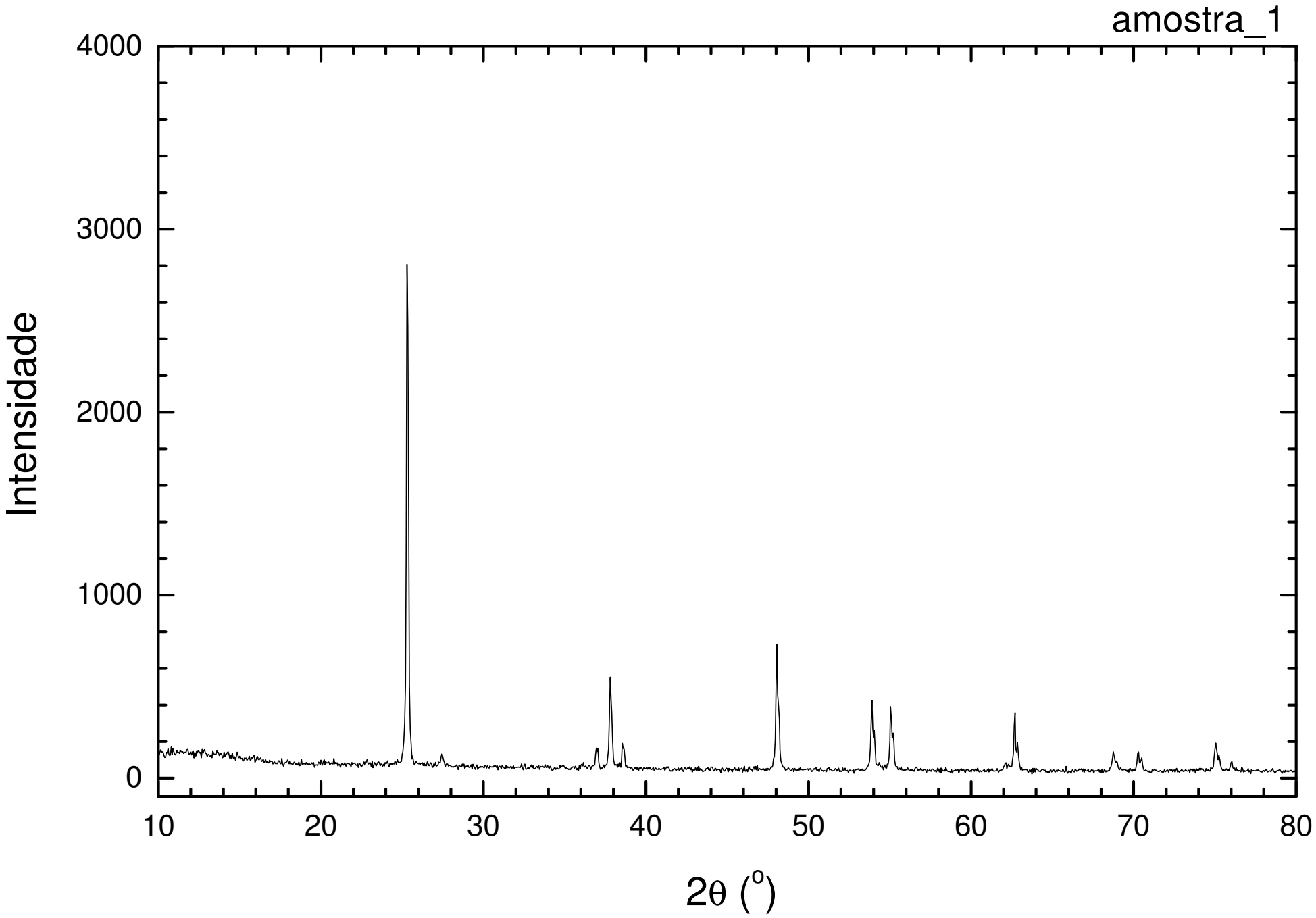
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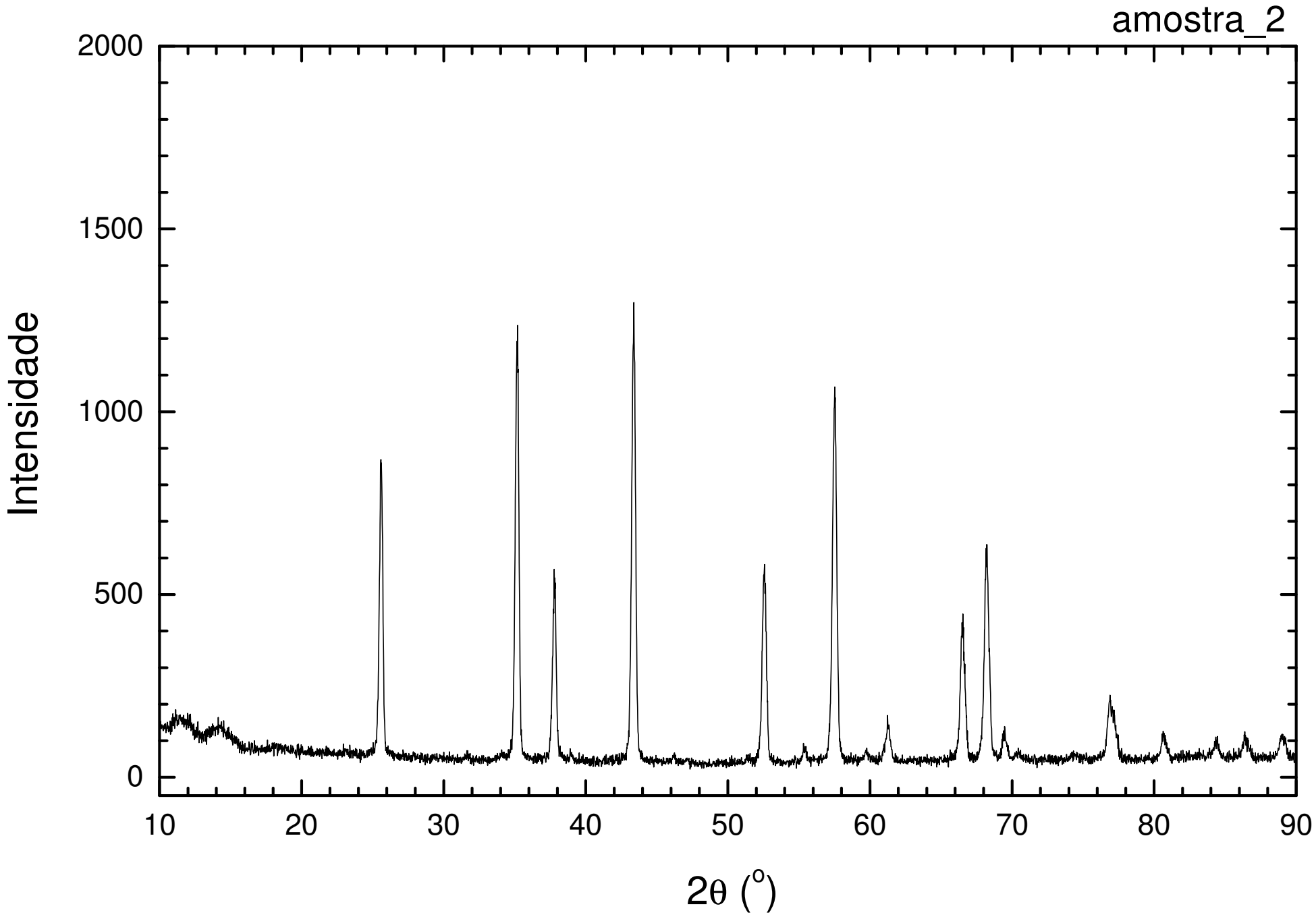
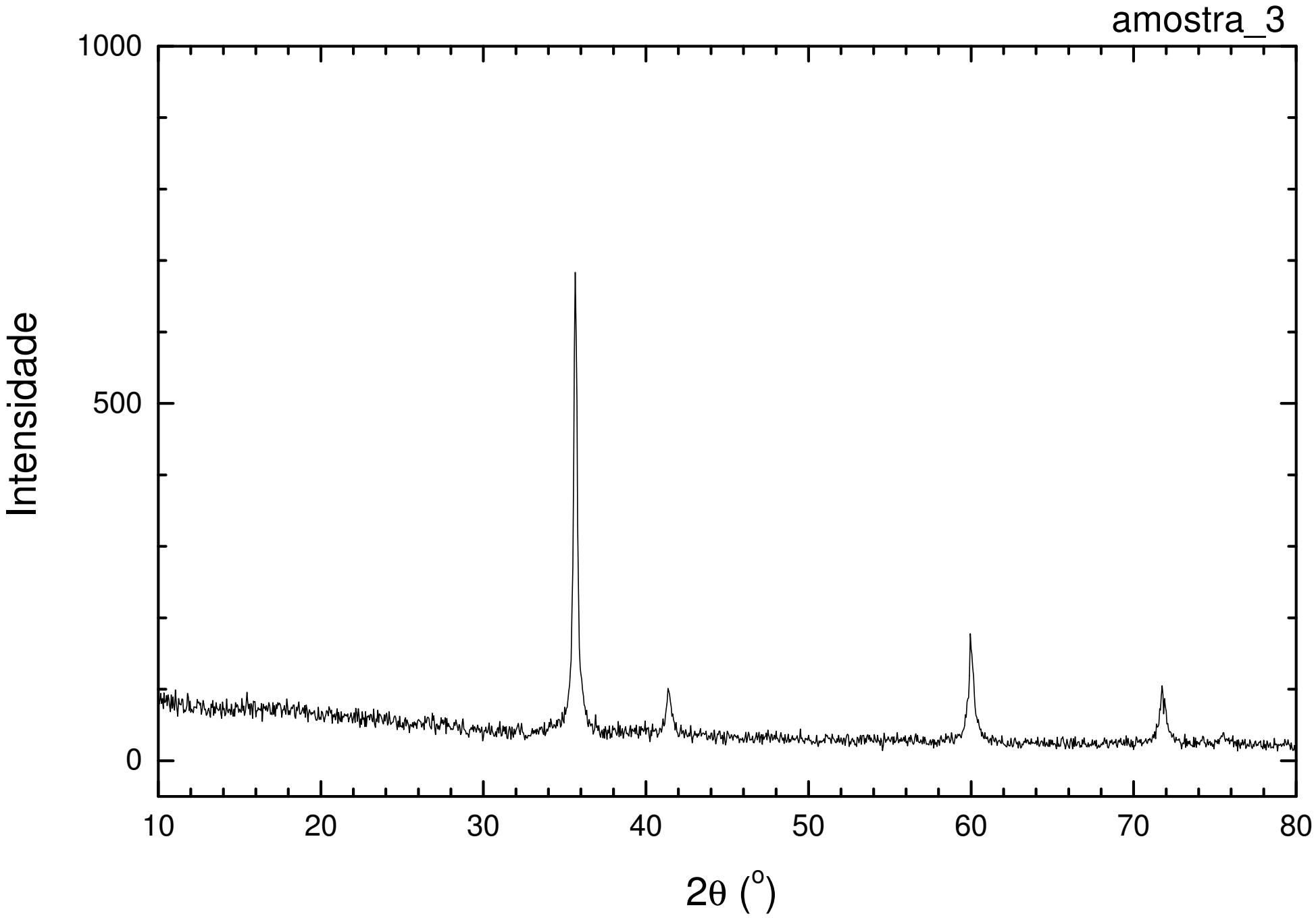
4000
3000
2000
1000
0
10
20
30
40
50
60
70
80
2θ (°)


amostra_2
Intensidade
2000
1500
1000
500
0
10
20
30
40
50
60
70
80
90
2θ (°)


amostra_3
1000
500
0
Intensidade
10
20
30
40
50
60
70
80
2θ (°)