The first results of measuring XAFS spectra in the soft X-ray range at KOSMOS station

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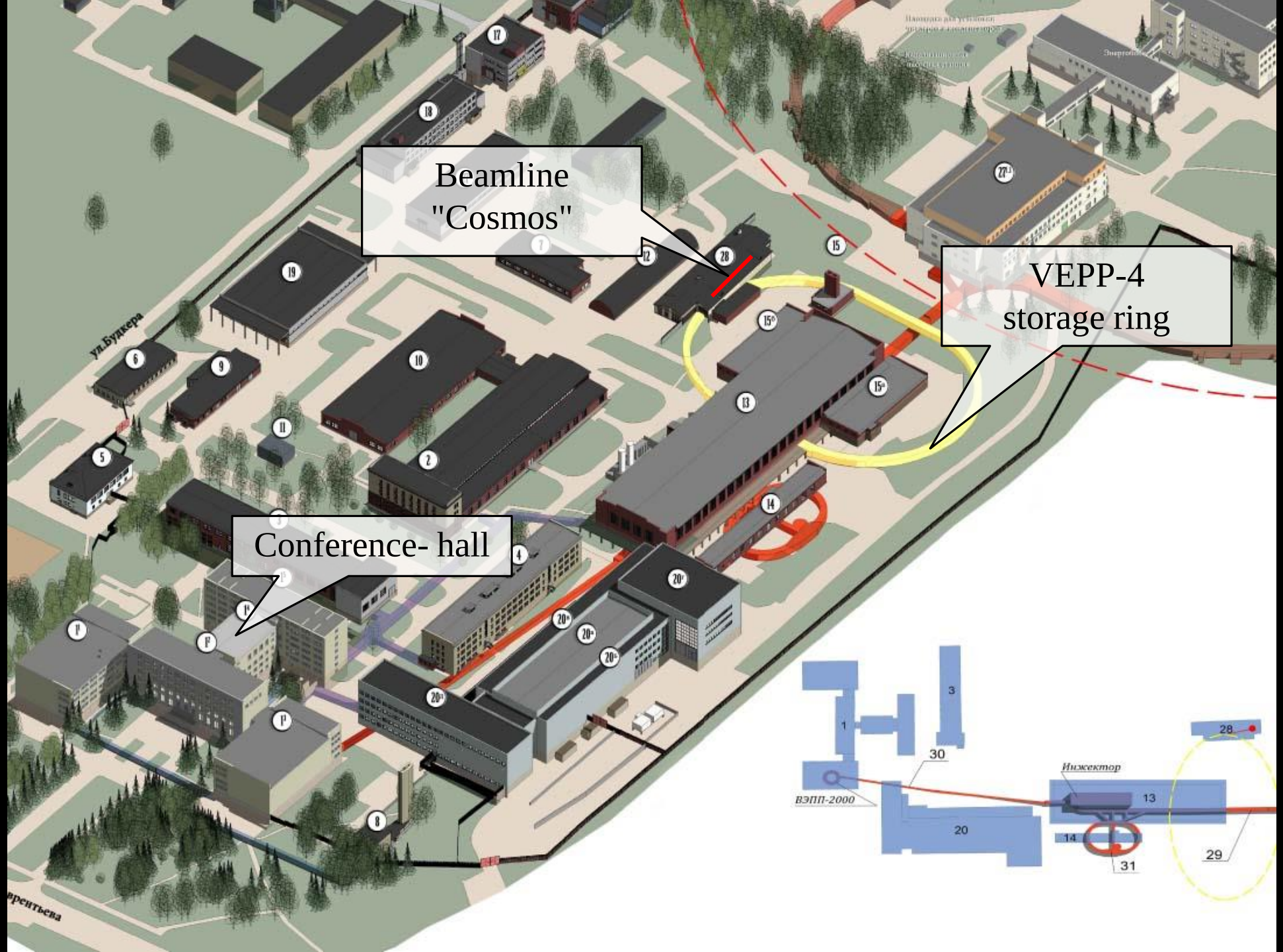
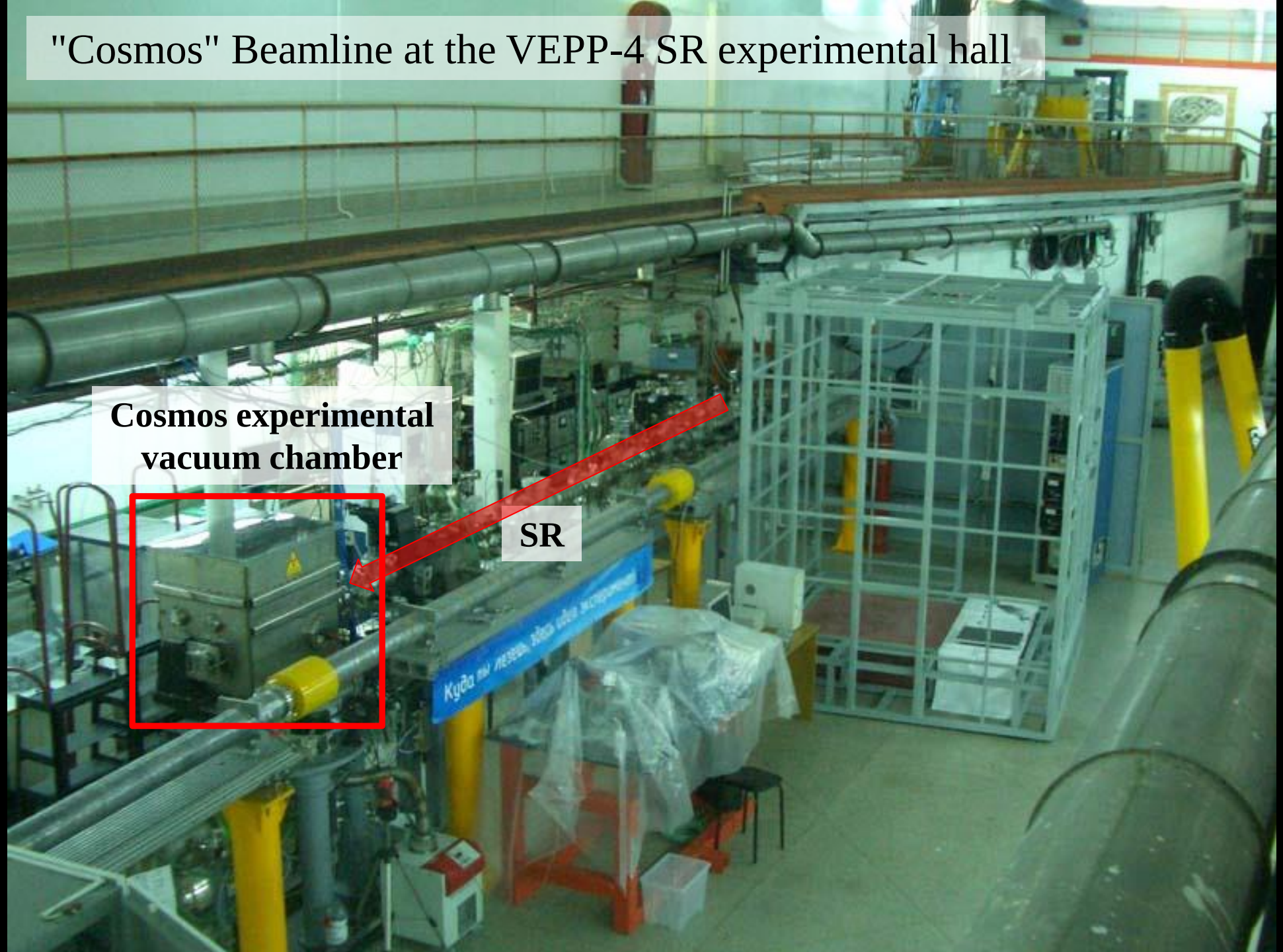
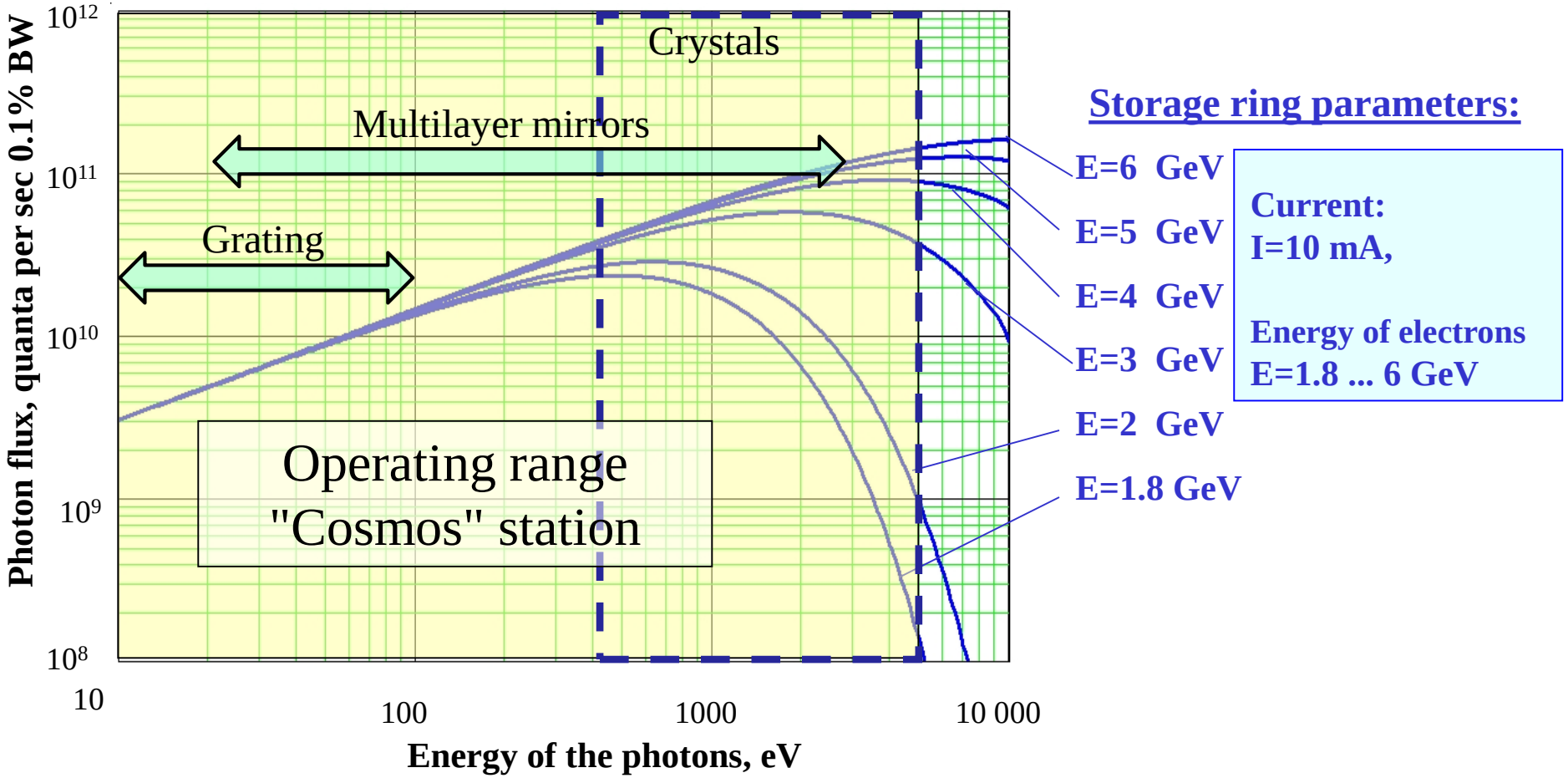

Beamline
"Cosmos"
VEPP-4
storage ring
Conference- hall
ул.Будкера
ВЭПП-2000
Инжектор
1
3
30
20
13
14
28
29
31


"Cosmos" Beamline at the VEPP-4 SR experimental hall
Cosmos experimental vacuum chamber
SR

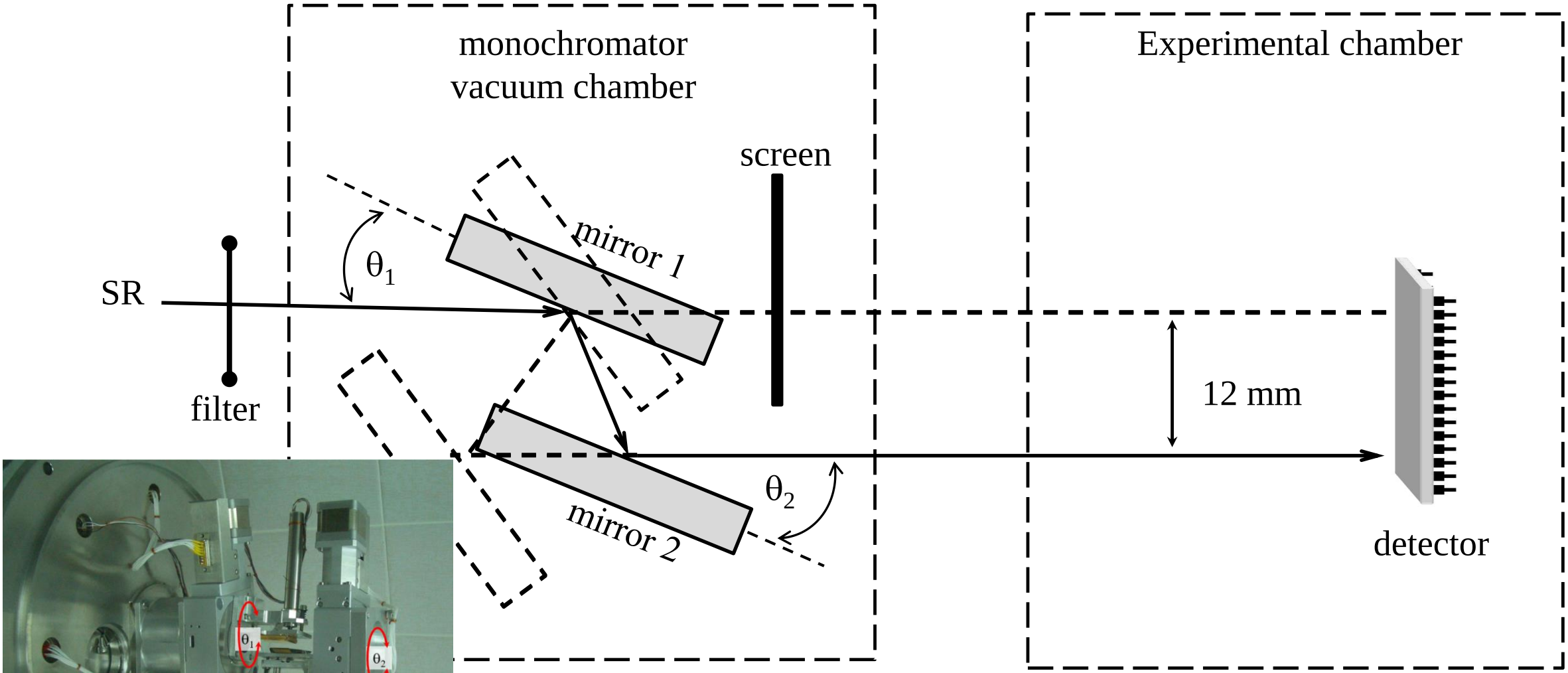
Photon flux at the «Cosmos» station for different energy of electrons in the VEPP-4 storage ring.

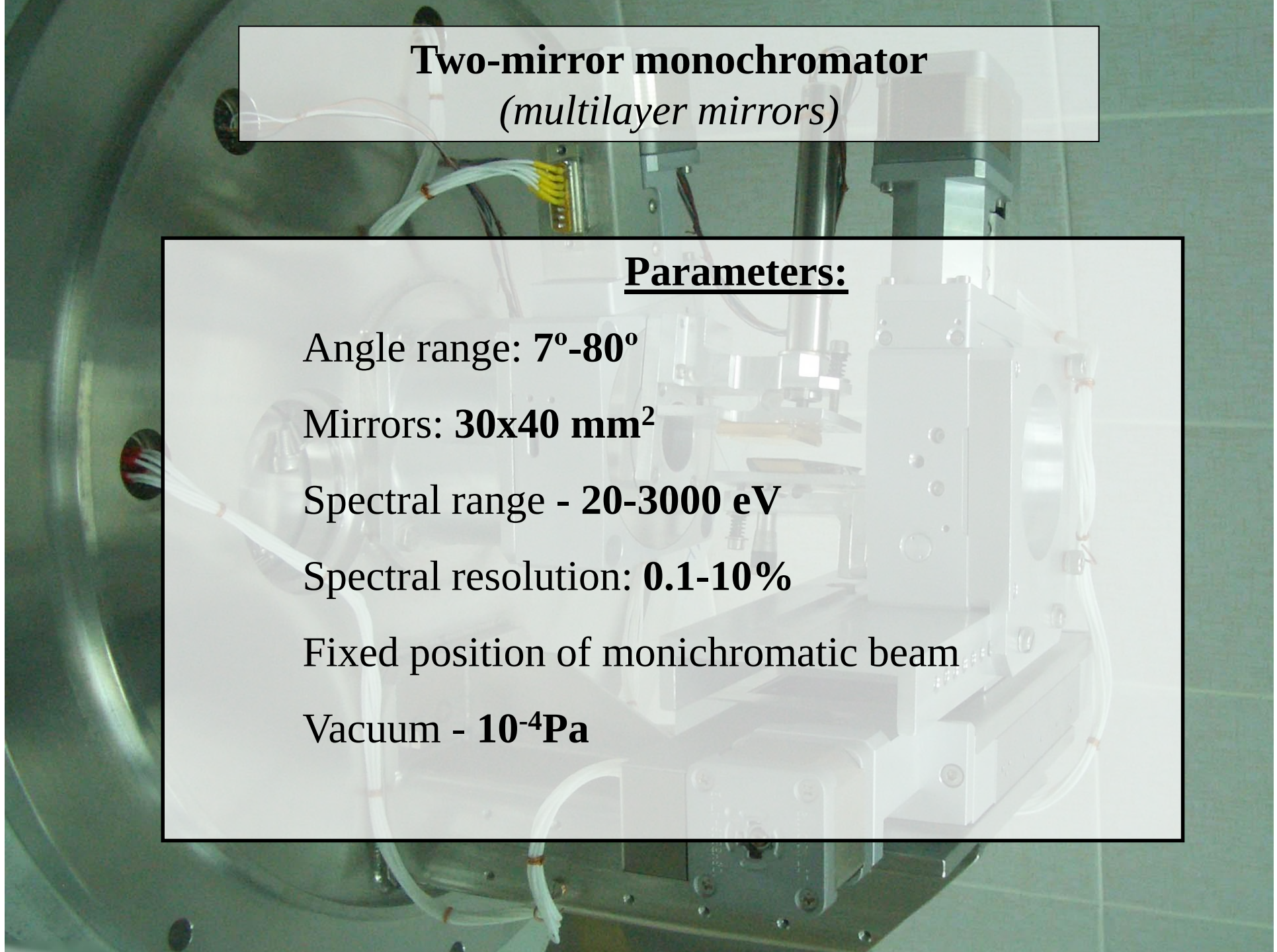
Beamline parameters:

aperture - 5X5 mm, distance to the source - 30



Optical scheme of a monochromator with multilayer mirrors





Two-mirror monochromator

(multilayer mirrors)

Parameters:

Angle range: **7º-80º**

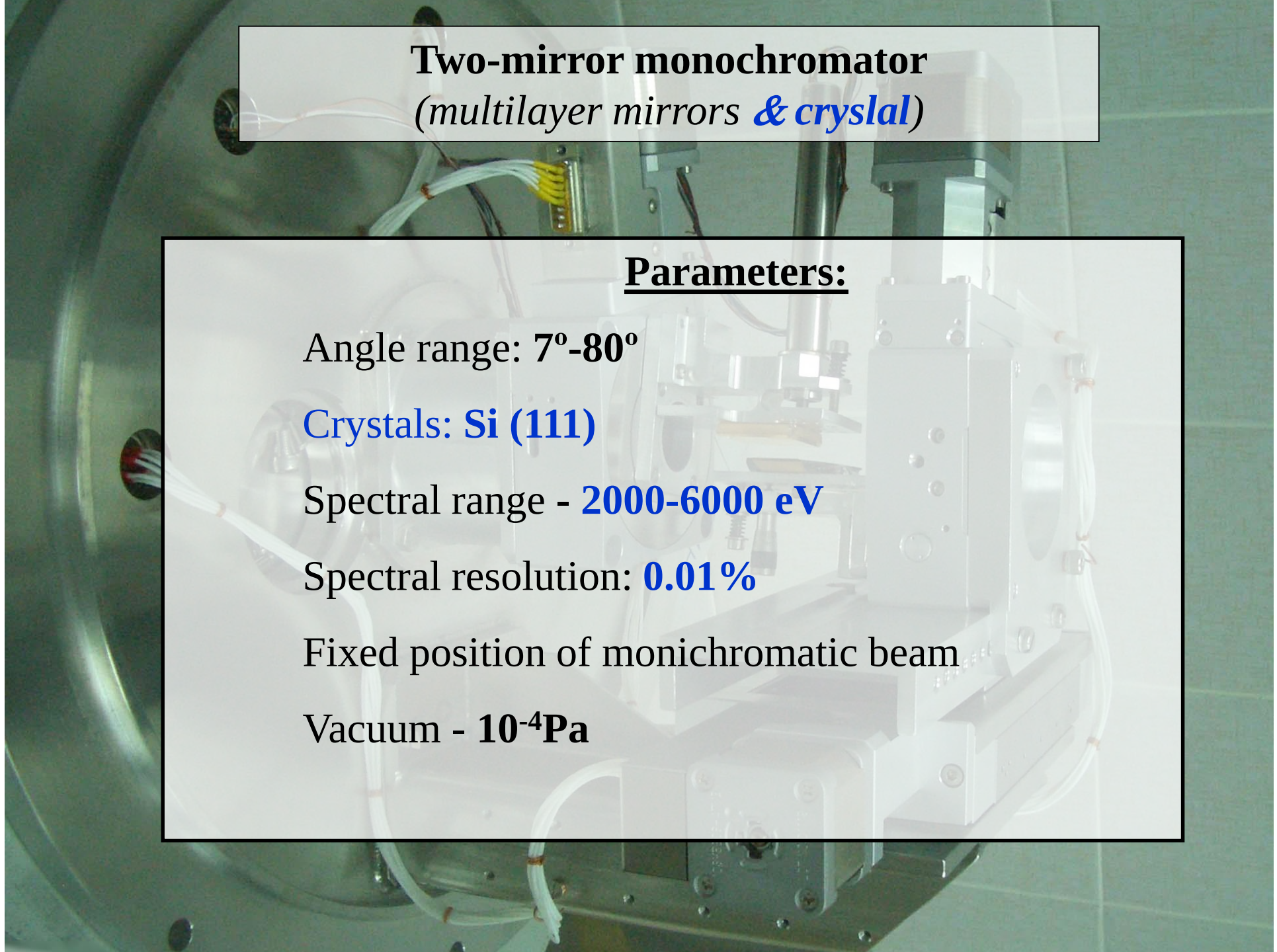
Mirrors: **30x40 mm^2**

Spectral range **- 20-3000 eV**

Spectral resolution: **0.1-10%**

Fixed position of monichromatic beam

Vacuum - **10^{-4}Pa**



Two-mirror monochromator
(multilayer mirrors & cryslal)

Parameters:

Angle range: **7º-80º**

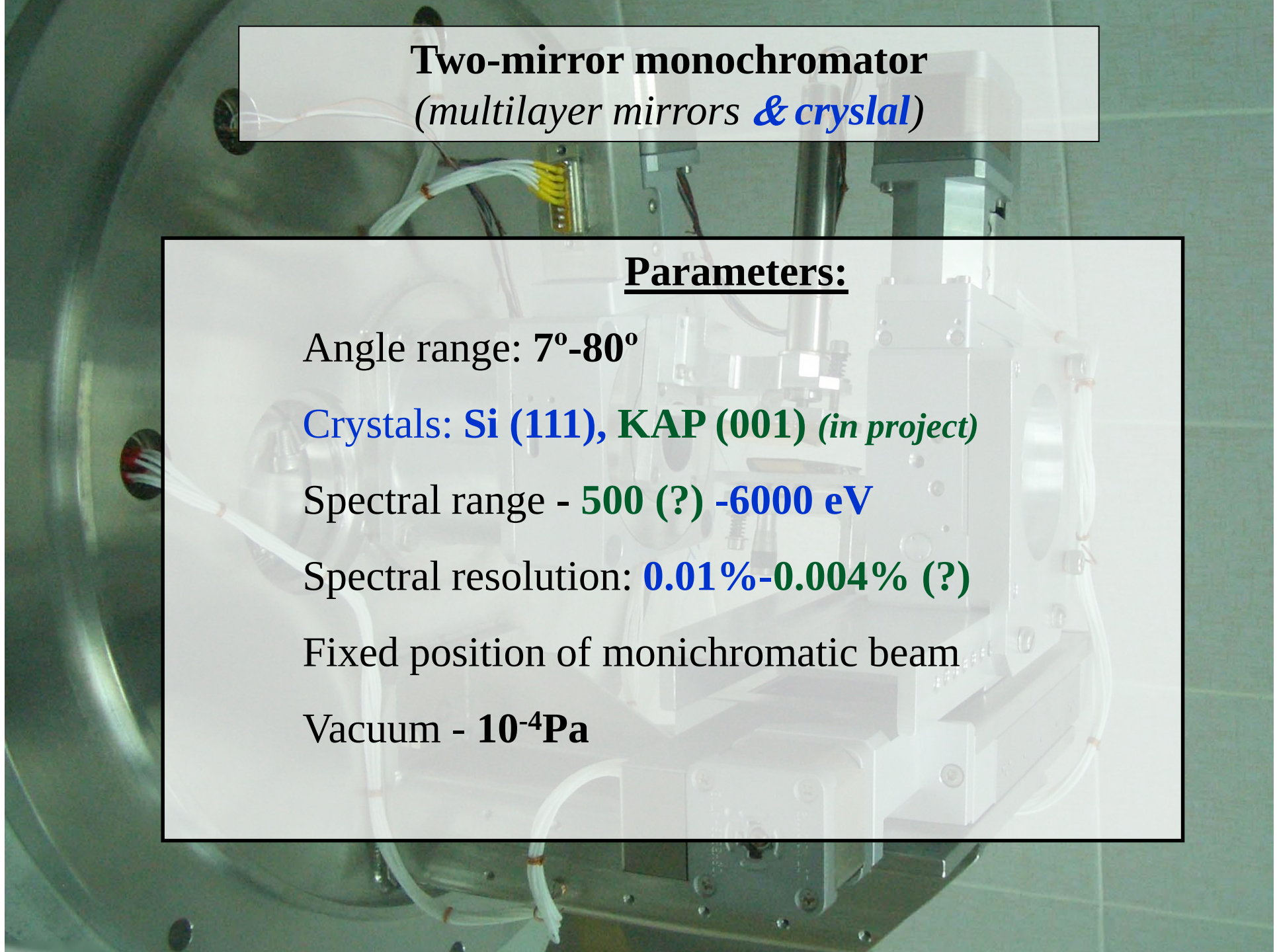
Crystals: **Si (111)**

Spectral range **- 2000-6000 eV**

Spectral resolution: **0.01%**

Fixed position of monichromatic beam

Vacuum - **10^{-4}Pa**



Two-mirror monochromator
(multilayer mirrors & cryslal)

Parameters:

Angle range: **7º-80º**

Crystals: **Si (111), KAP (001)** ***(in project)***

Spectral range - **500 (?) -6000 eV**

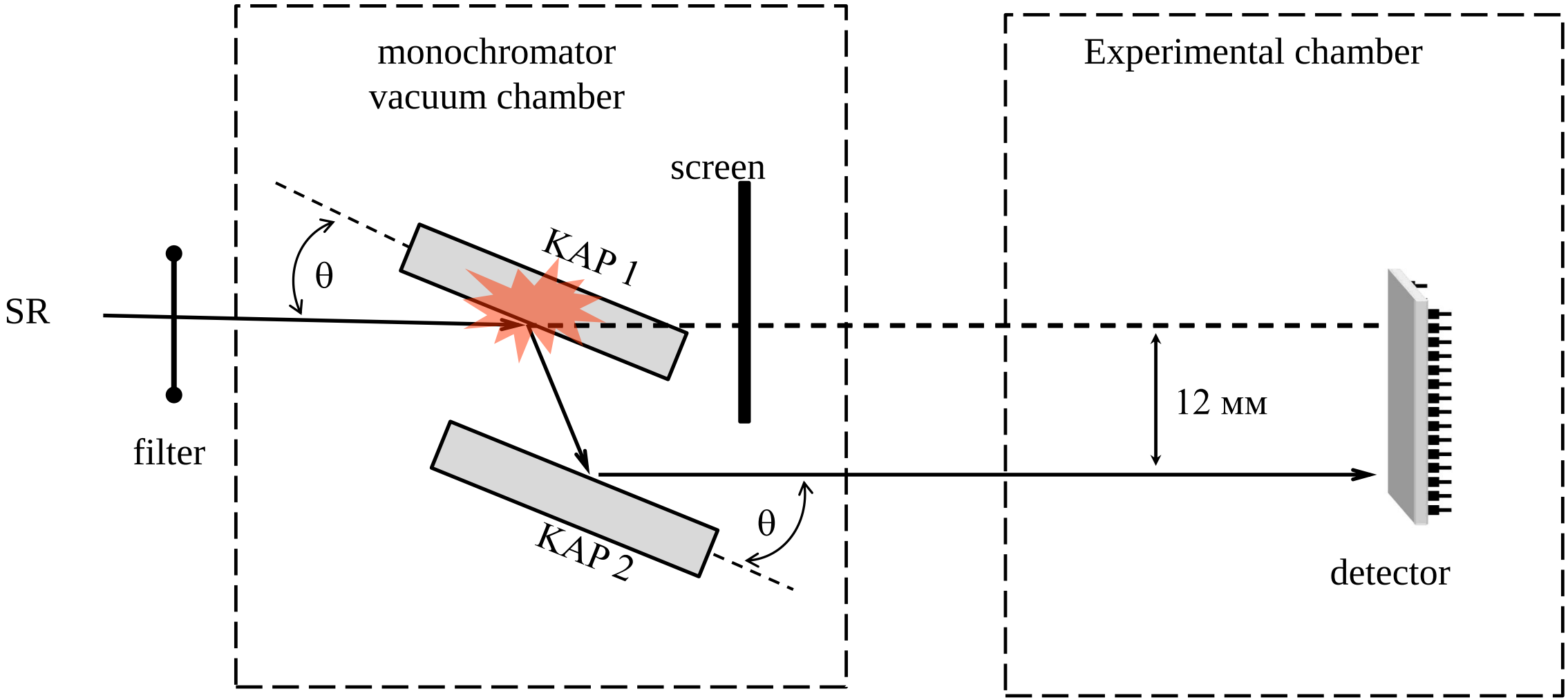
Spectral resolution: **0.01%-0.004% (?)**

Fixed position of monichromatic beam

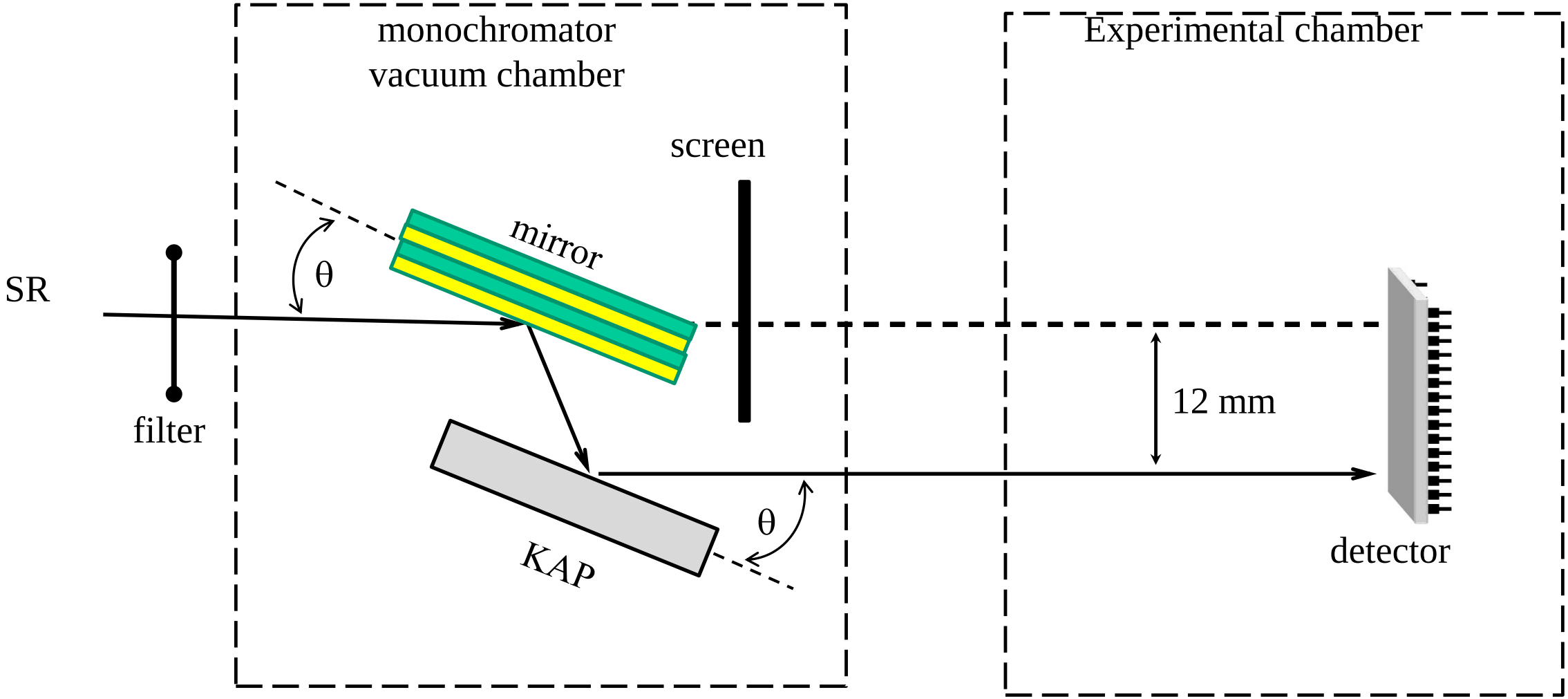
Vacuum - **10^{-4}Pa**

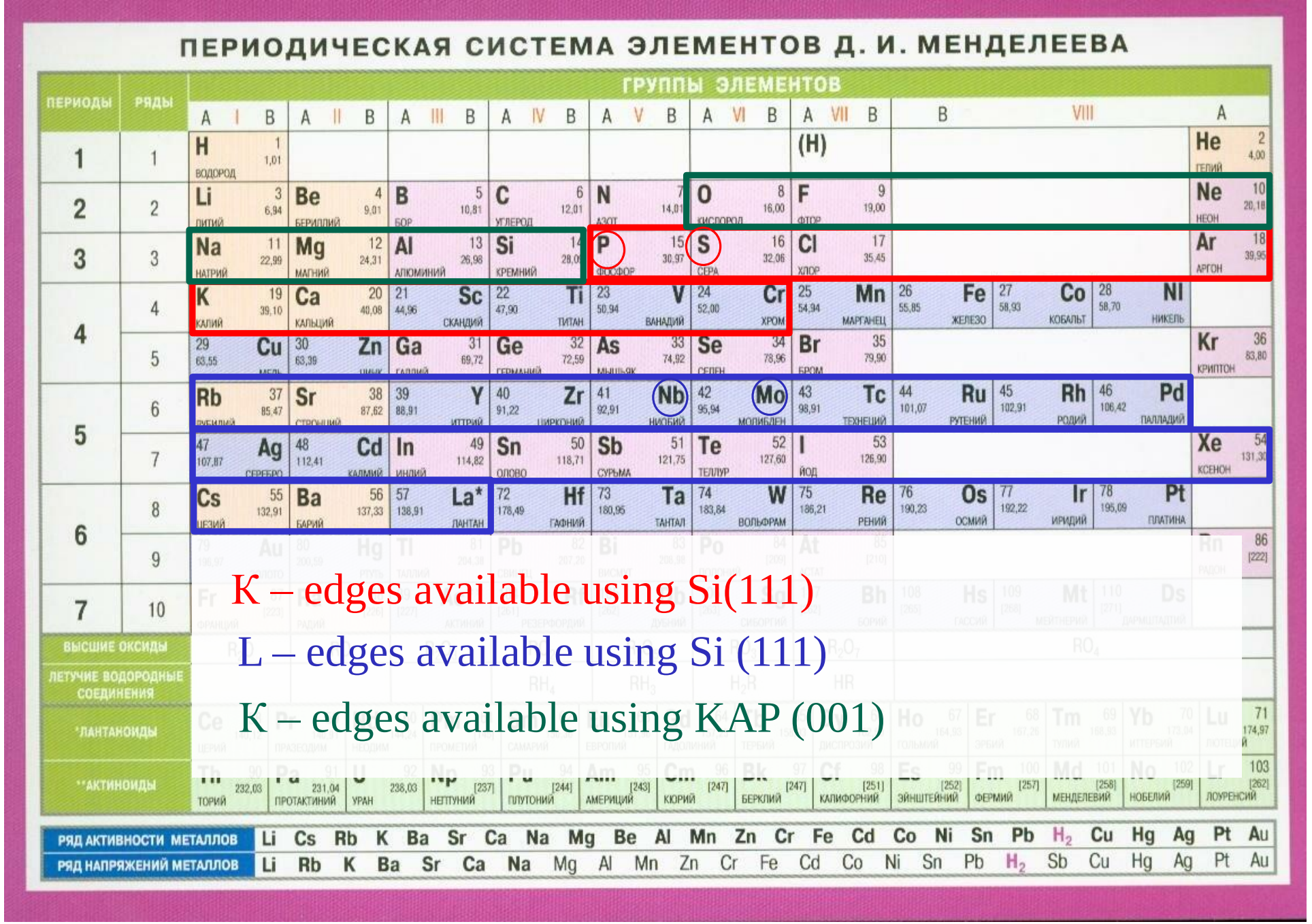
Optical design of the monochromator using KAP crystals

Problem: low radiation resistance of a KAP under a white SR beam



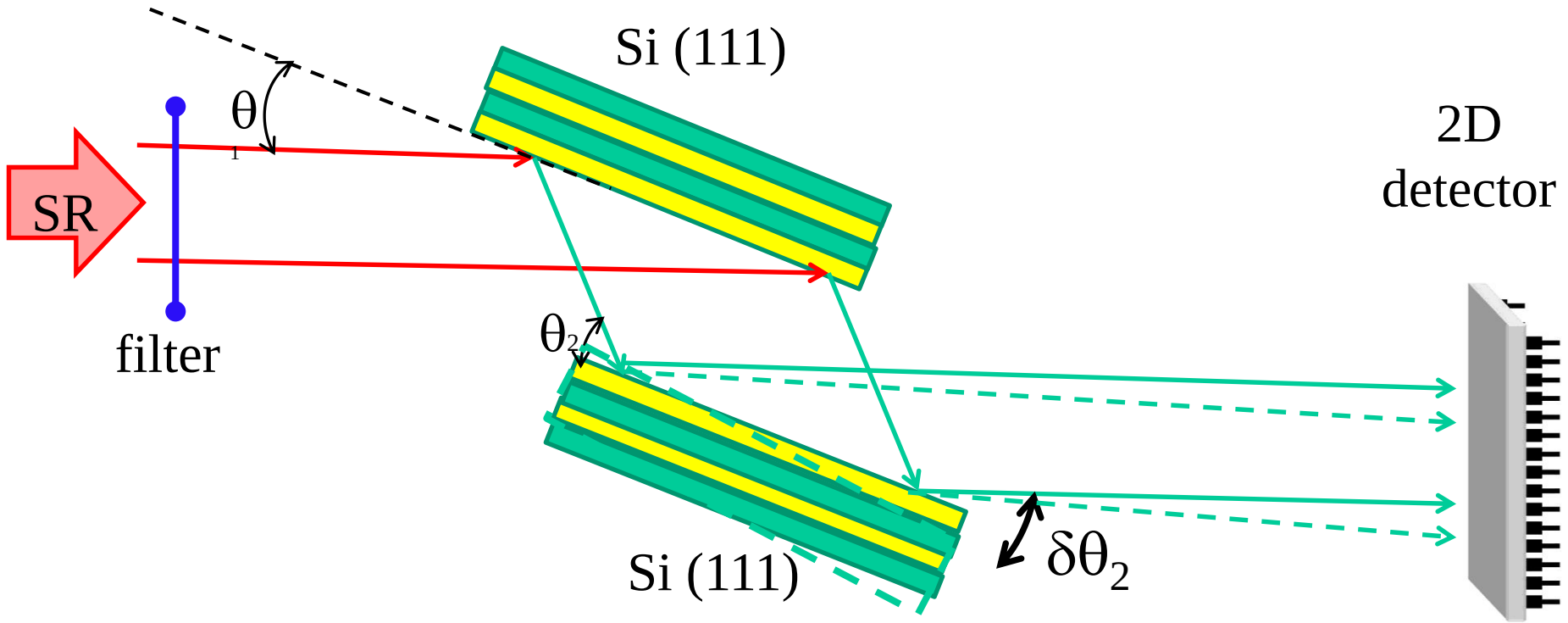
Possible hybrid optical design: multilayer mirror (2d = 26.6 A) + KAP crystal
E = 500 – 2 000 eV




ПЕРИОДИЧЕСКАЯ СИСТЕМА ЭЛЕМЕНТОВ Д. И. МЕНДЕЛЕЕВА
ГРУППЫ ЭЛЕМЕНТОВ
ПЕРИОДЫ
РЯДЫ
K – edges available using Si(111)
L – edges available using Si (111)
K – edges available using KAP (001)
РЯД АКТИВНОСТИ МЕТАЛЛОВ
РЯД НАПРЯЖЕНИЙ МЕТАЛЛОВ

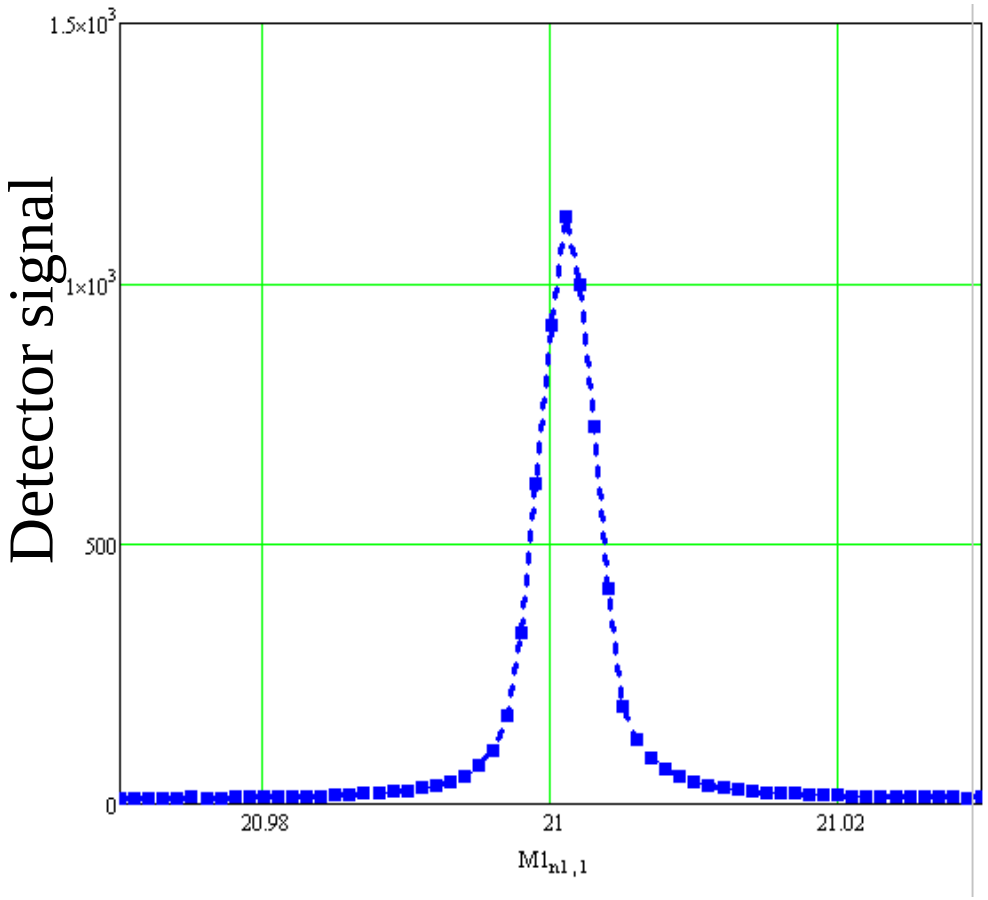
Commission of the two-crystal monochromator

measurement of the rocking curve

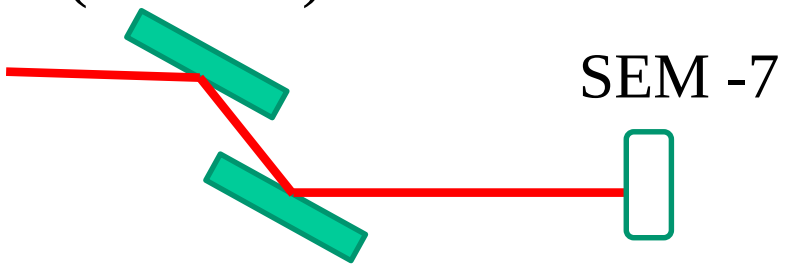


Commission of the two-crystal monochromator

measurement of the rocking curve



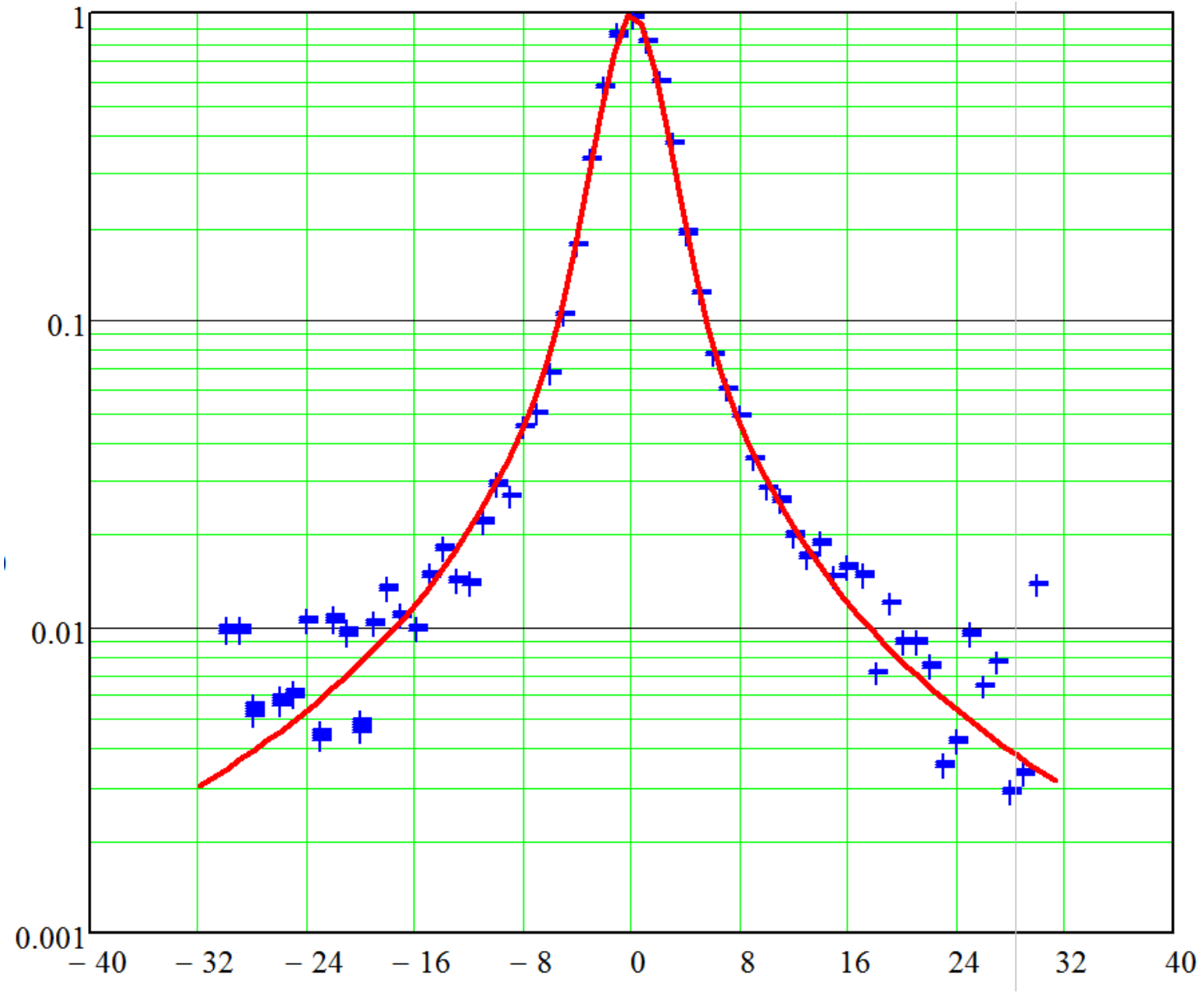
$\theta_1 = 21\ °$
(5517 эВ)



$\theta_2 = 21° \pm 0.03°$
$(\delta\theta = 0.001\ °)$

There is no complete external reflection (pure monochromate)

Approximation of the rocking curve by the pseudo-Voigt function



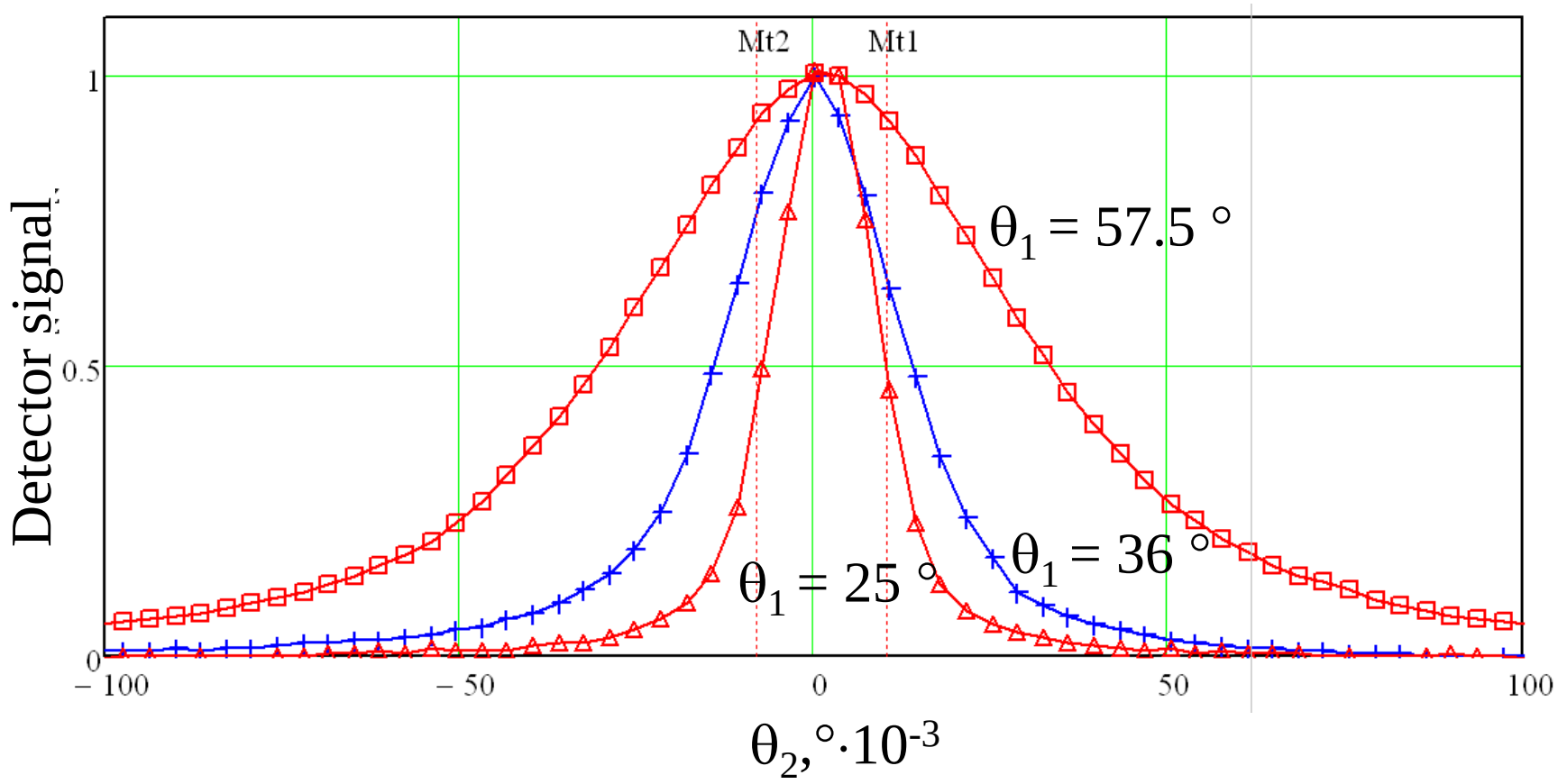
E_{ph}= 5 167 eV

$\theta_1 = 22.5°$

FWHM $\delta\theta_2 = 16$ ”

Commission of the two-crystal monochromator

measurement of the rocking curve Si(111)



Commission of the two-crystal monochromator

measurement of the rocking curve

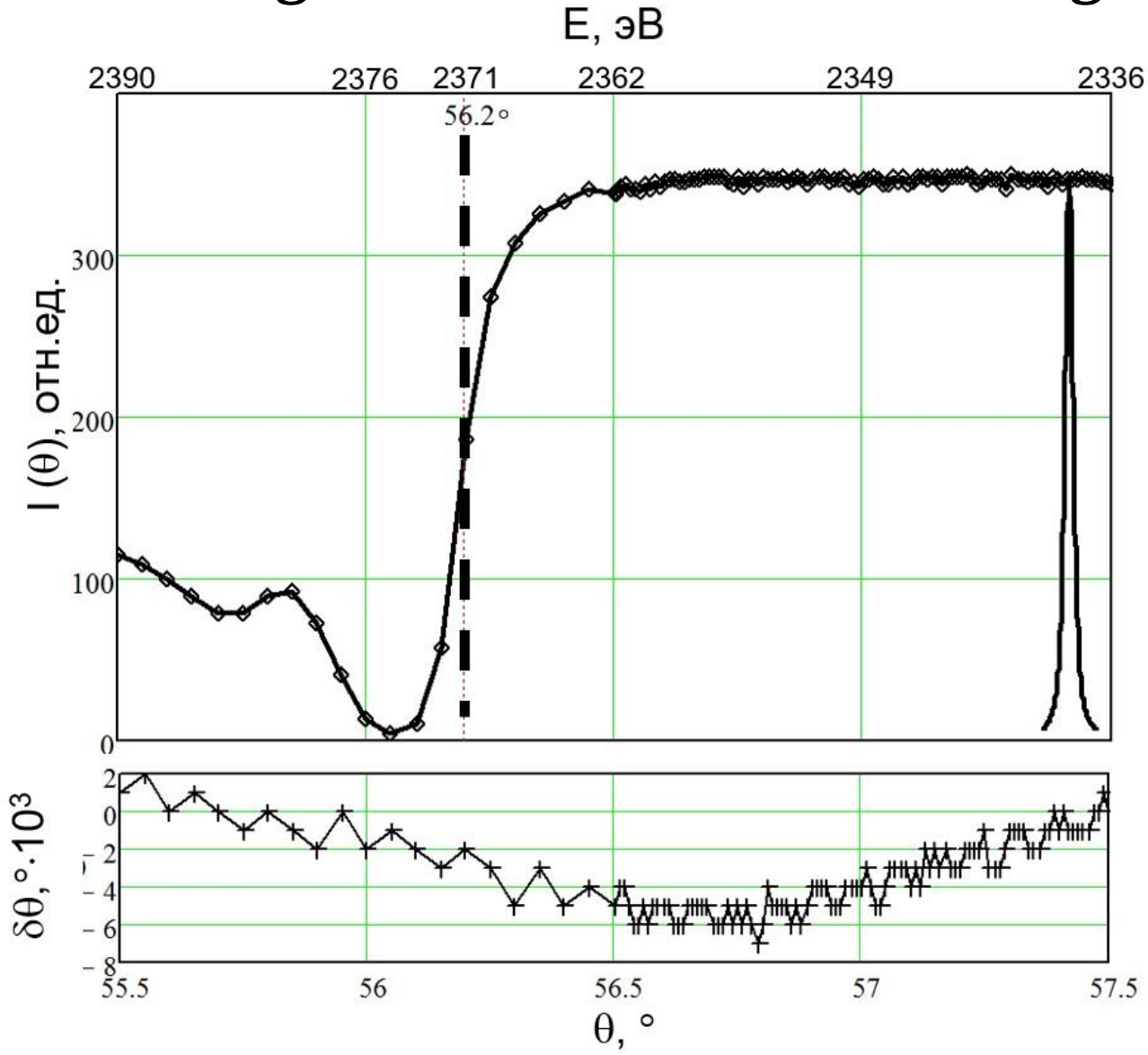
	Width of the rocking curve, arc. sec.	***Width of the rocking curve, eV***	***Photon energy, eV***	***rocking curve width***
θ_1	$\Delta\theta_2$	ΔE_{rc}	E	ΔE_{rc} /E
24°	16,89"	0,926	4 843	$1{,}912 * 10^{-4}$
36°	29,93"	0,647	3 364	$1{,}922 * 10^{-4}$
57.5°	64,98"	0,459	2 345	$1{,}957 * 10^{-4}$

good concurrence

Measured spectral resolution of the double-crystal scheme :

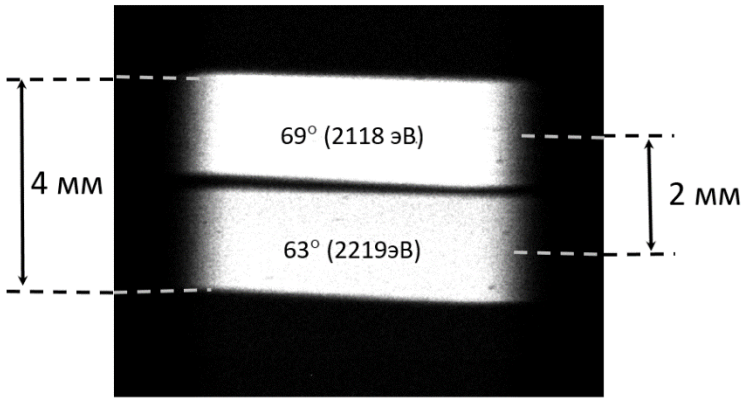
$$\frac{\Delta E_m}{E} = \frac{1}{\sqrt{2}} \cdot \frac{1}{\sqrt{2}} \cdot \frac{\Delta E_{rc}}{E} \approx 10^{-4}$$

L-3 edge of niobium obtained using second crystal fine tuning



Since each crystal is driven by its own goniometer, a positioning error $\delta\theta$ accumulates, leading to a violation of the setting for the Bragg condition. An attempt is made to automatically fine-tune the second crystal according by the signal amplitude.

Using the fine-tuning algorithm increases the scan time (20 seconds per point. The total scan time is 40 minutes).



Vertical beam shift during scanning

Monochromator Update

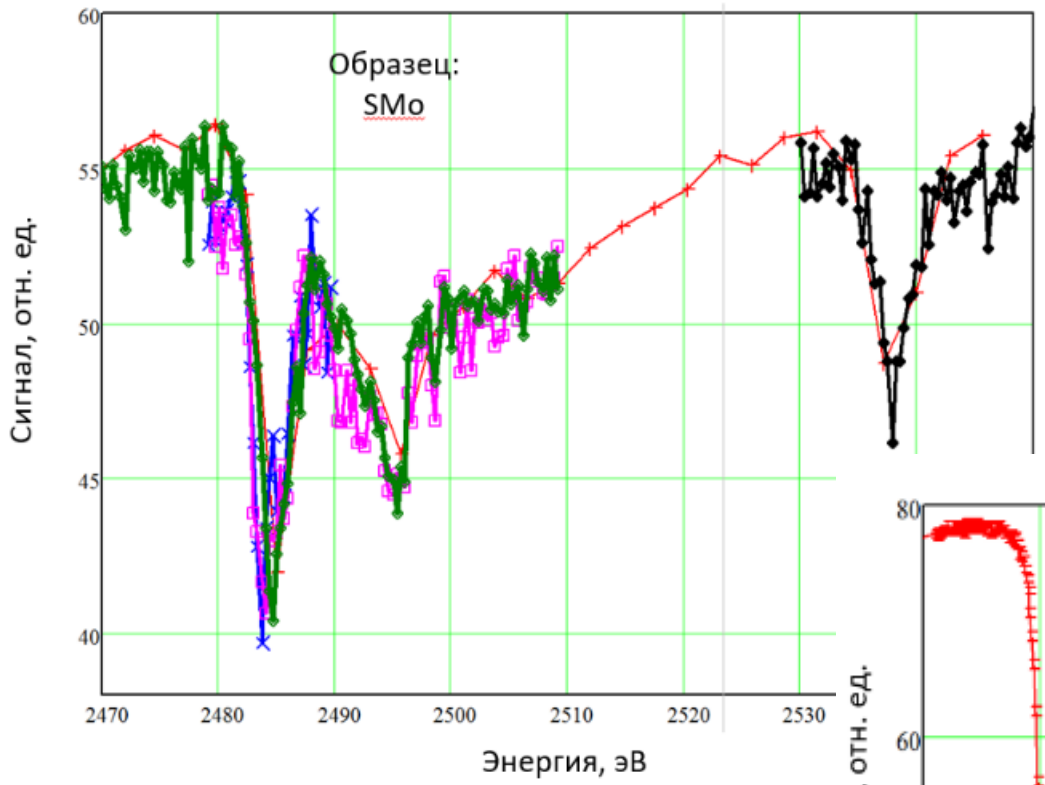
$\theta_{1\&2}$

$\delta\theta_1$

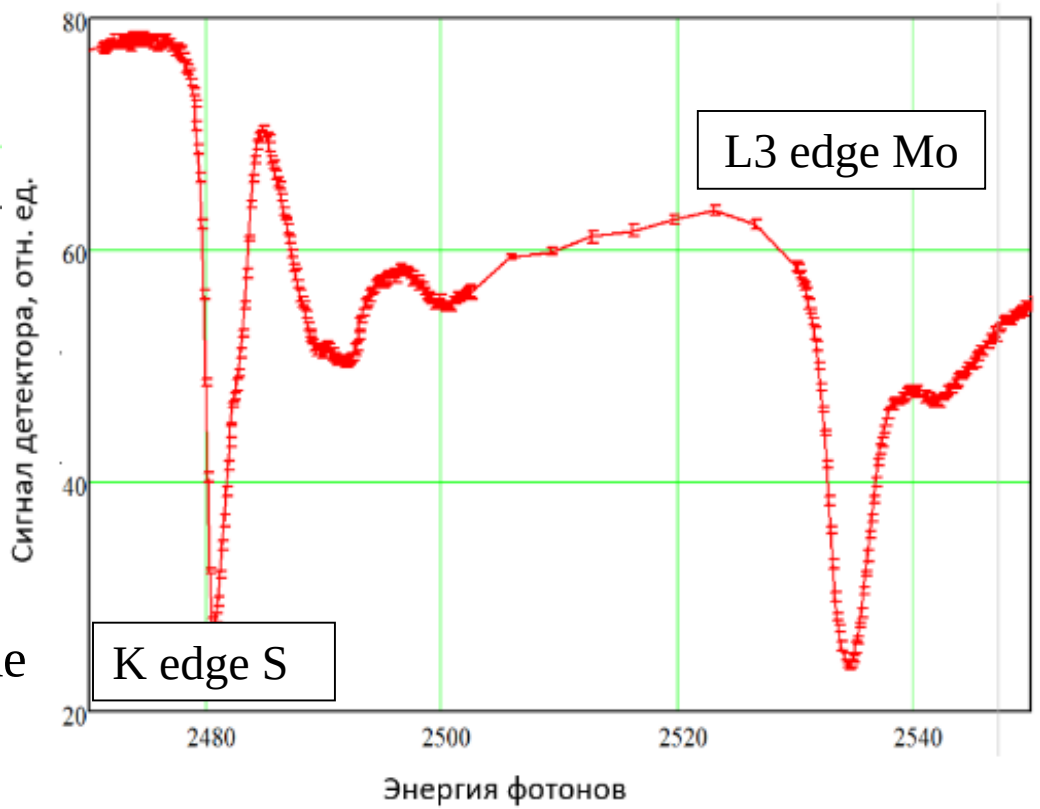
SR

Monochromator Update Results:

scanning using the same sample

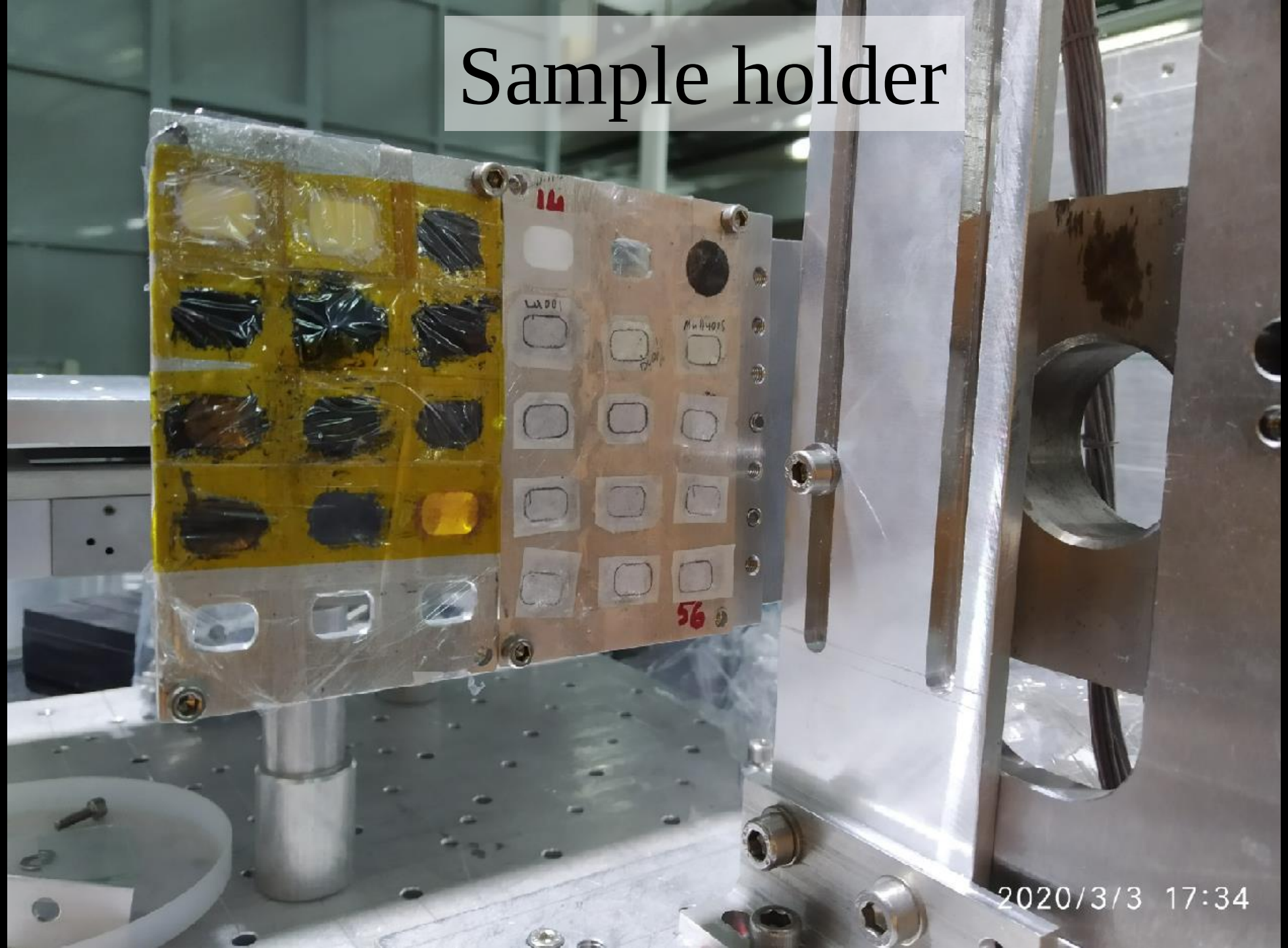


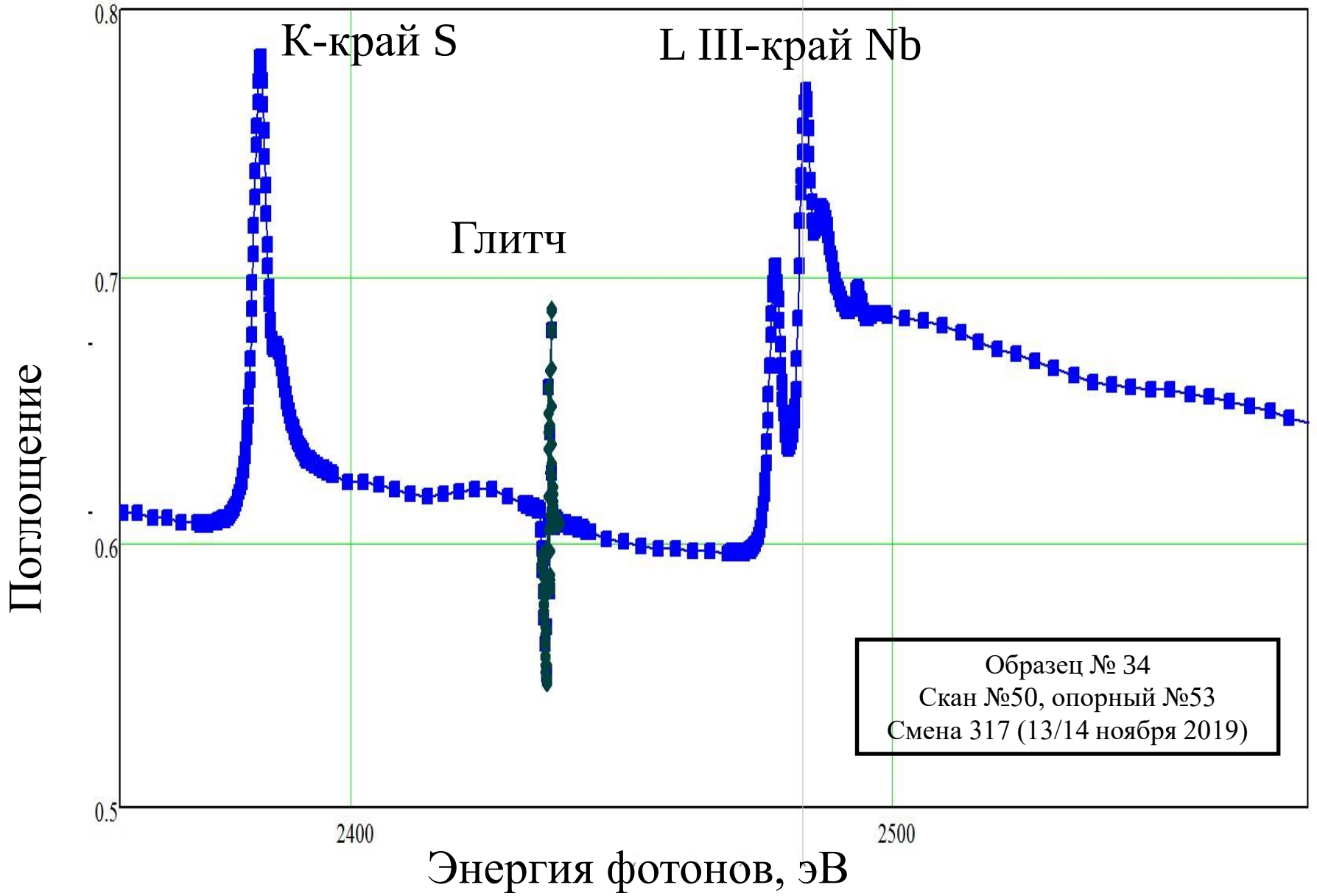
~ 20 seconds per point.



~4.5 seconds per point.
Spectrum acquisition time ~ 27 min.
significantly cleaner spectrum in less time

Sample holder



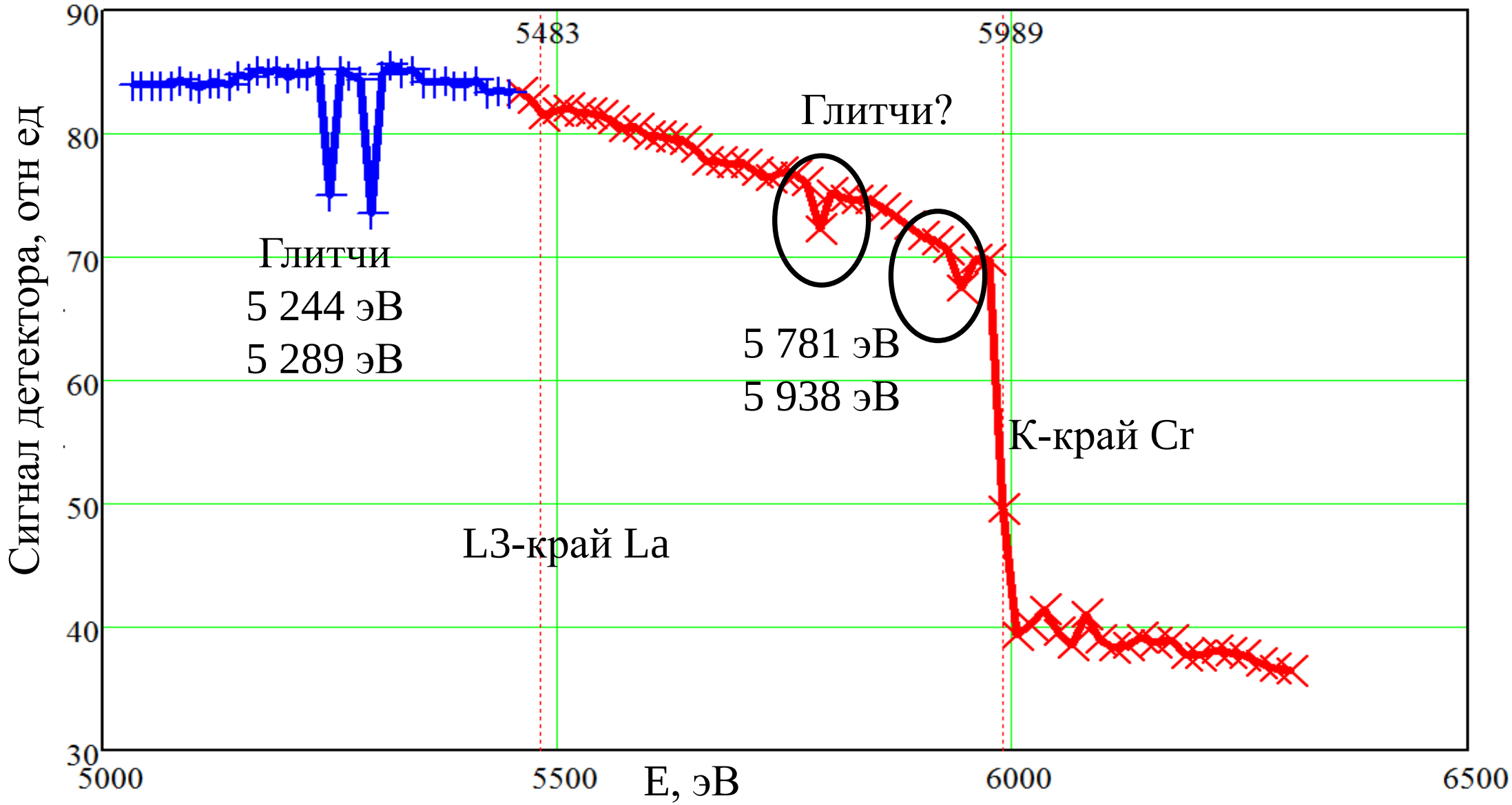


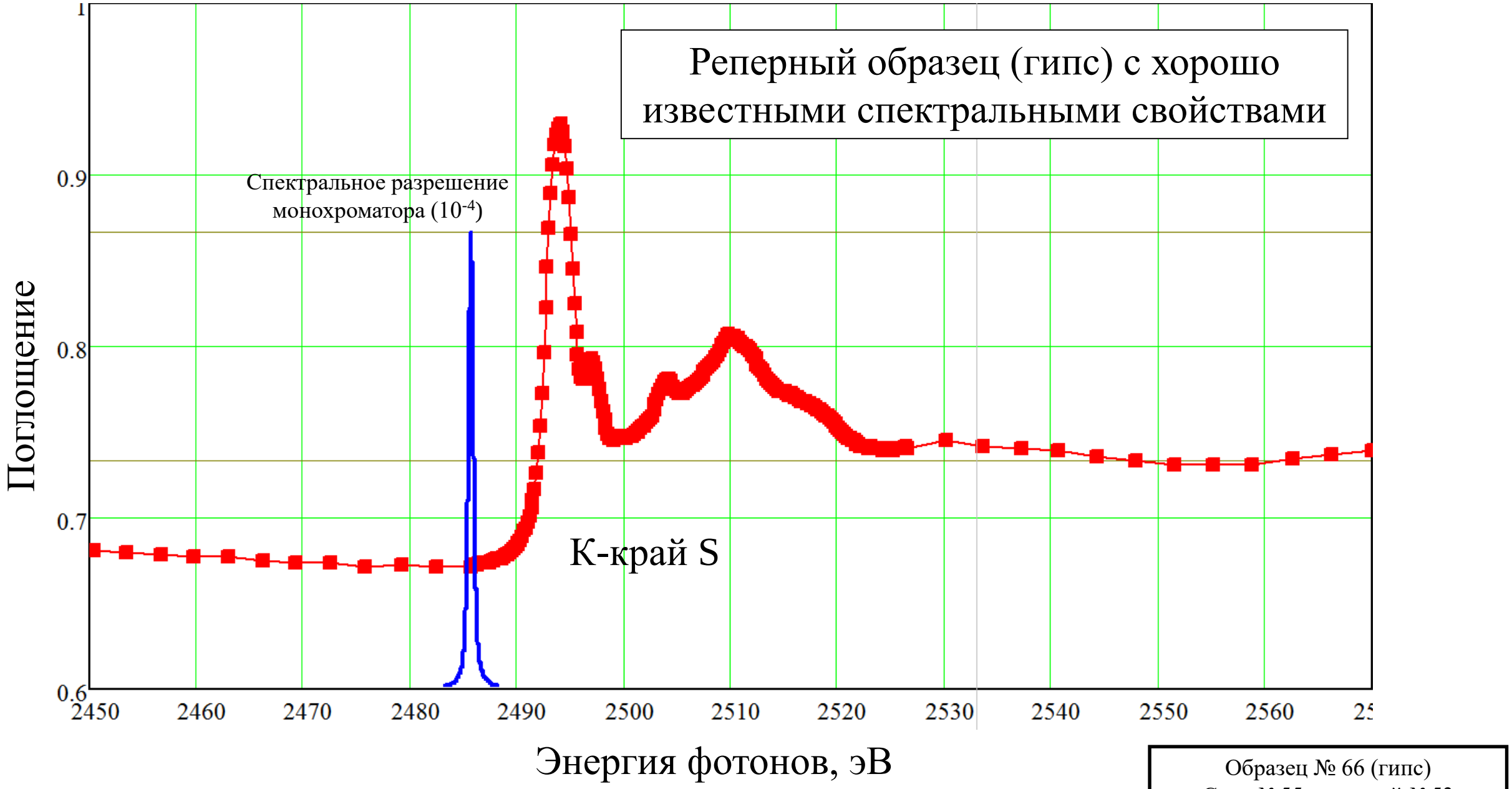
Биядерный кластер $\{Nb_2S_4\}$ с мостиковыми дисульфидными лигандами S_2^2

Попытка измерения спектра вблизи L3 края лантана

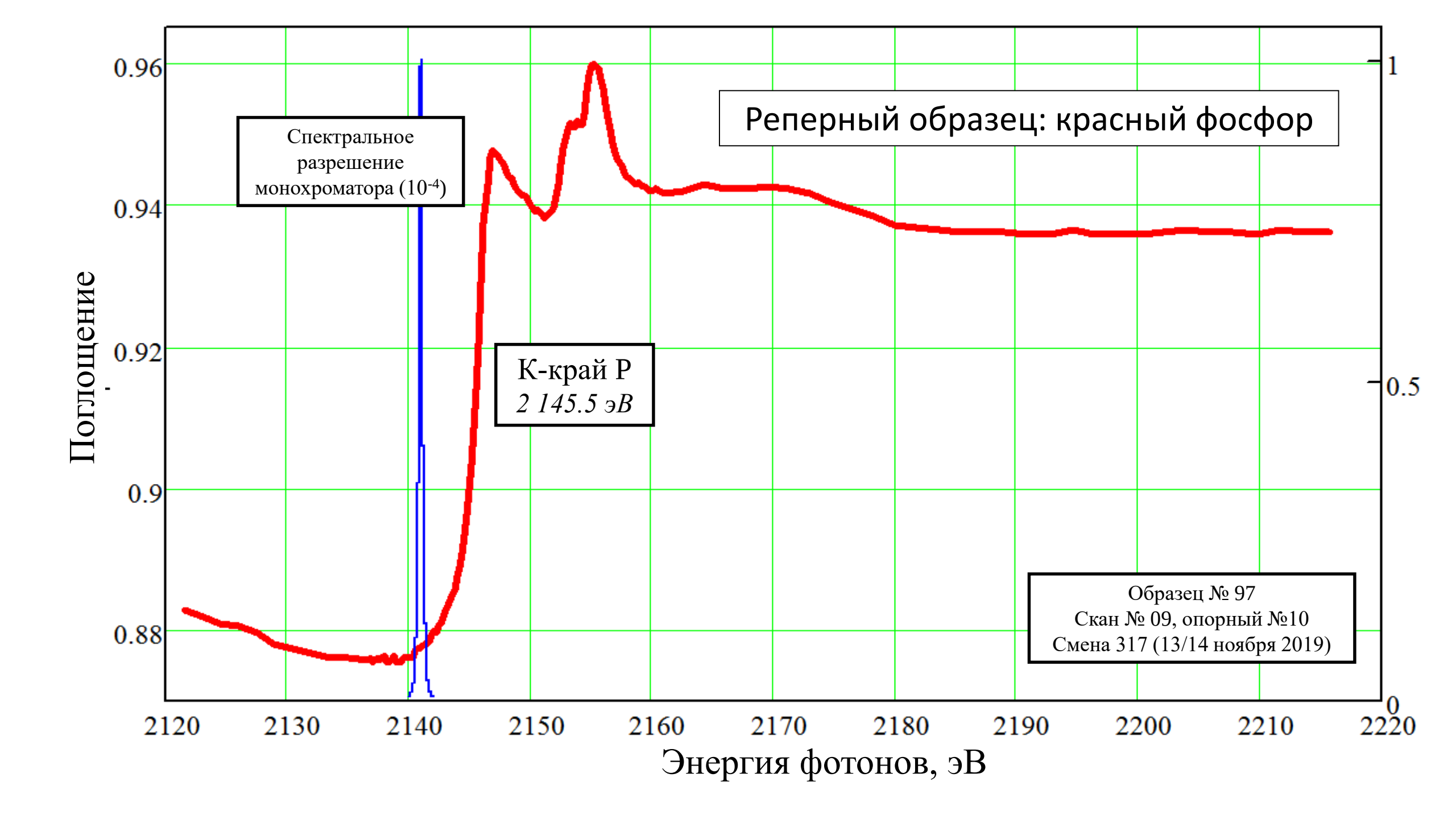
Образец $CuCr_{0.99}La_{0.01}S_2$

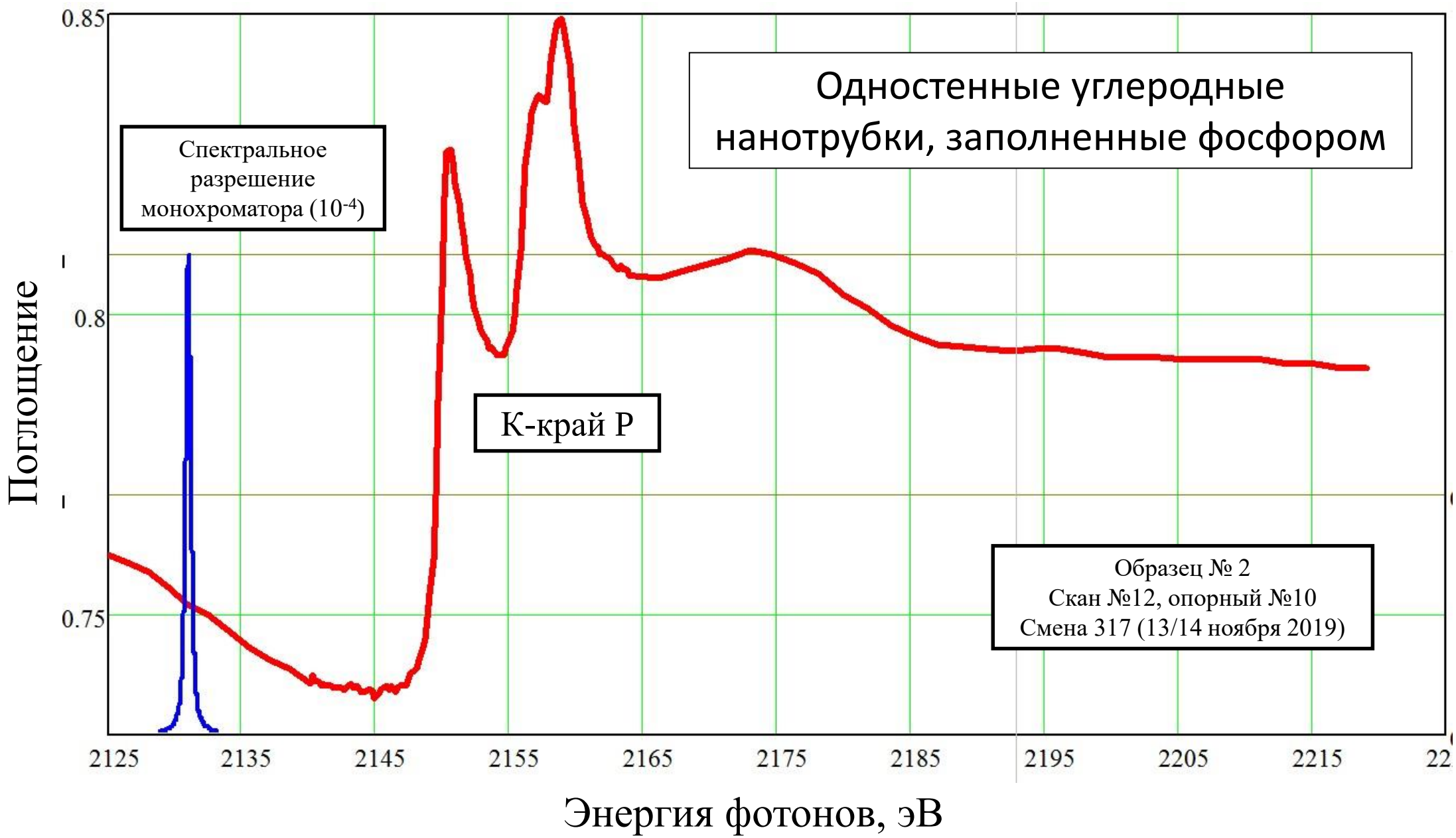
Образец № 16
Скан №14,15,
Смена 323 (15 декабря 2019)





Образец № 66 (гипс)
Скан №55, опорный №53
Смена 317 (13/14 ноября 2019)


Реперный образец: красный фосфор
Спектральное разрешение монохроматора (10^{-4})
К-край P
2 145.5 эВ
Образец № 97
Скан № 09, опорный №10
Смена 317 (13/14 ноября 2019)
Поглощение
0.96
0.94
0.92
0.9
0.88
1
0.5
0
2120
2130
2140
2150
2160
2170
2180
2190
2200
2210
2220
Энергия фотонов, эВ


Односттенные углеродные нанотрубки, заполненные фосфором
Спектральное разрешение монохроматора (10^{-4})
К-край P
Образец № 2
Скан №12, опорный №10
Смена 317 (13/14 ноября 2019)
Поглощение
0.85
0.8
0.75
2125
2135
2145
2155
2165
2175
2185
2195
2205
2215
Энергия фотонов, эВ

Conclusions

- At Cosmos station, it became possible to measure XAFS spectra in the range of 2–6 keV, which corresponds to the K - edges of elements from P to Cr and L - edges from Rb to La. The measurement rate is about 20 samples per shift.

- Spectra were measured near the K - edges P and S and L3 of the Mo and Nb edges (about 50 spectra on different samples)

- It seems appropriate to switch to the hybrid MRZ + KAP scheme, which will expand the range of applicability to 500 eV, (K is the edge from O to Si)

- The metrological capabilities of the Cosmos station for the certification of optical elements have been expanded