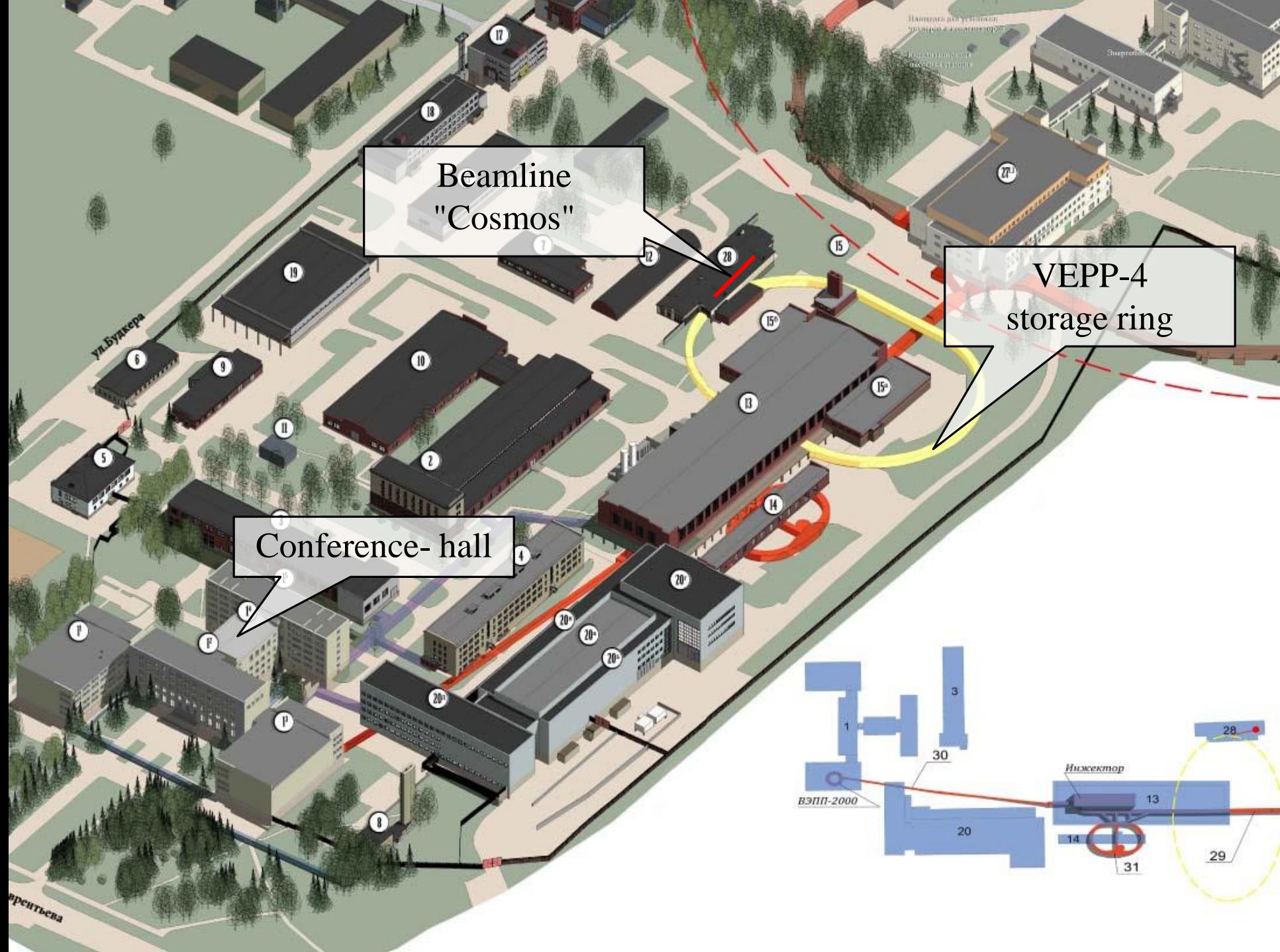


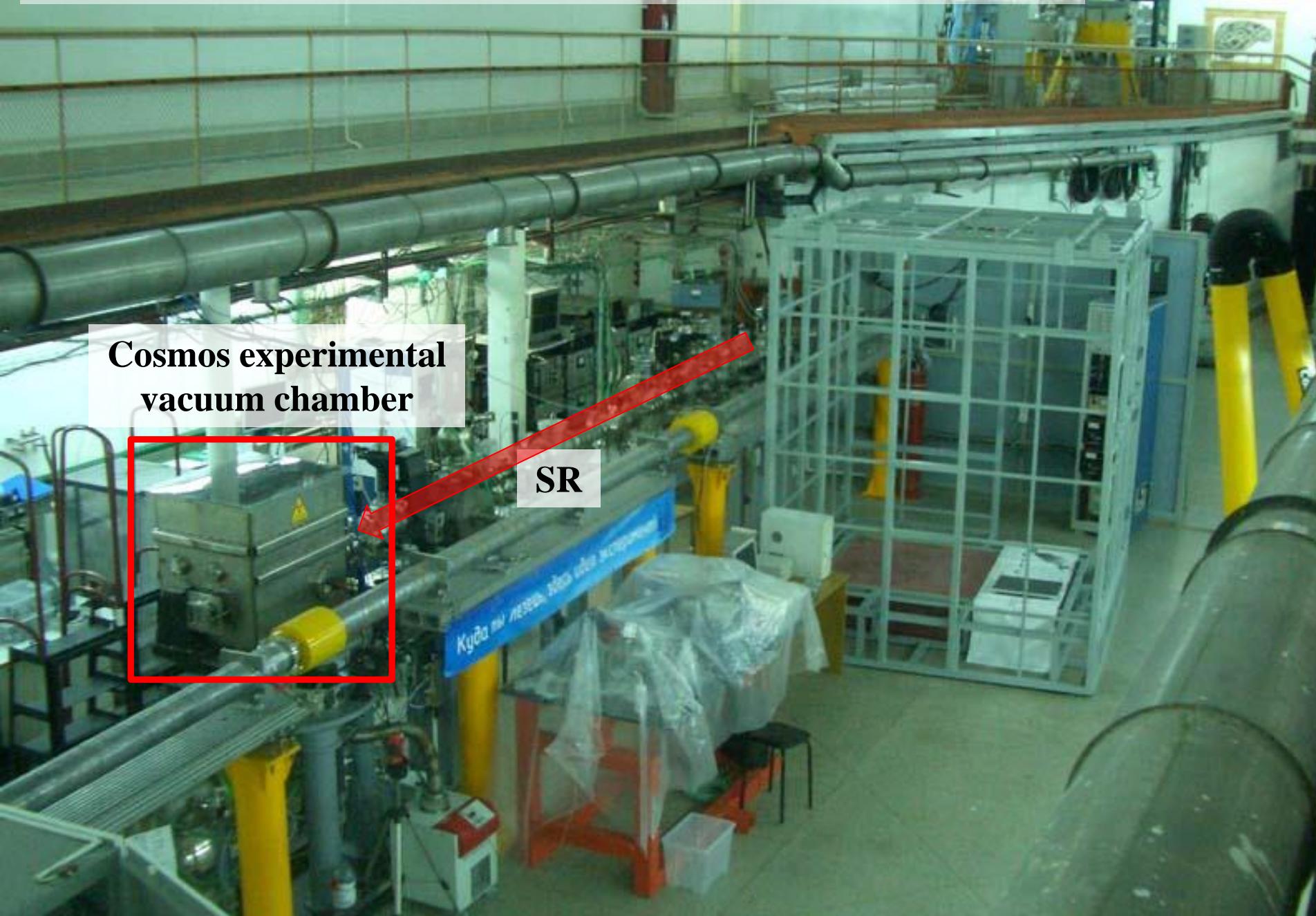
# The first results of measuring XAFS spectra in the soft X-ray range at KOSMOS station

A.D. Nikolenko, P.S. Zavertkin, D.V. Ivlyushkin, A.D. Fedorenko, E.V. Korotaev, M.M. Syrokvashin





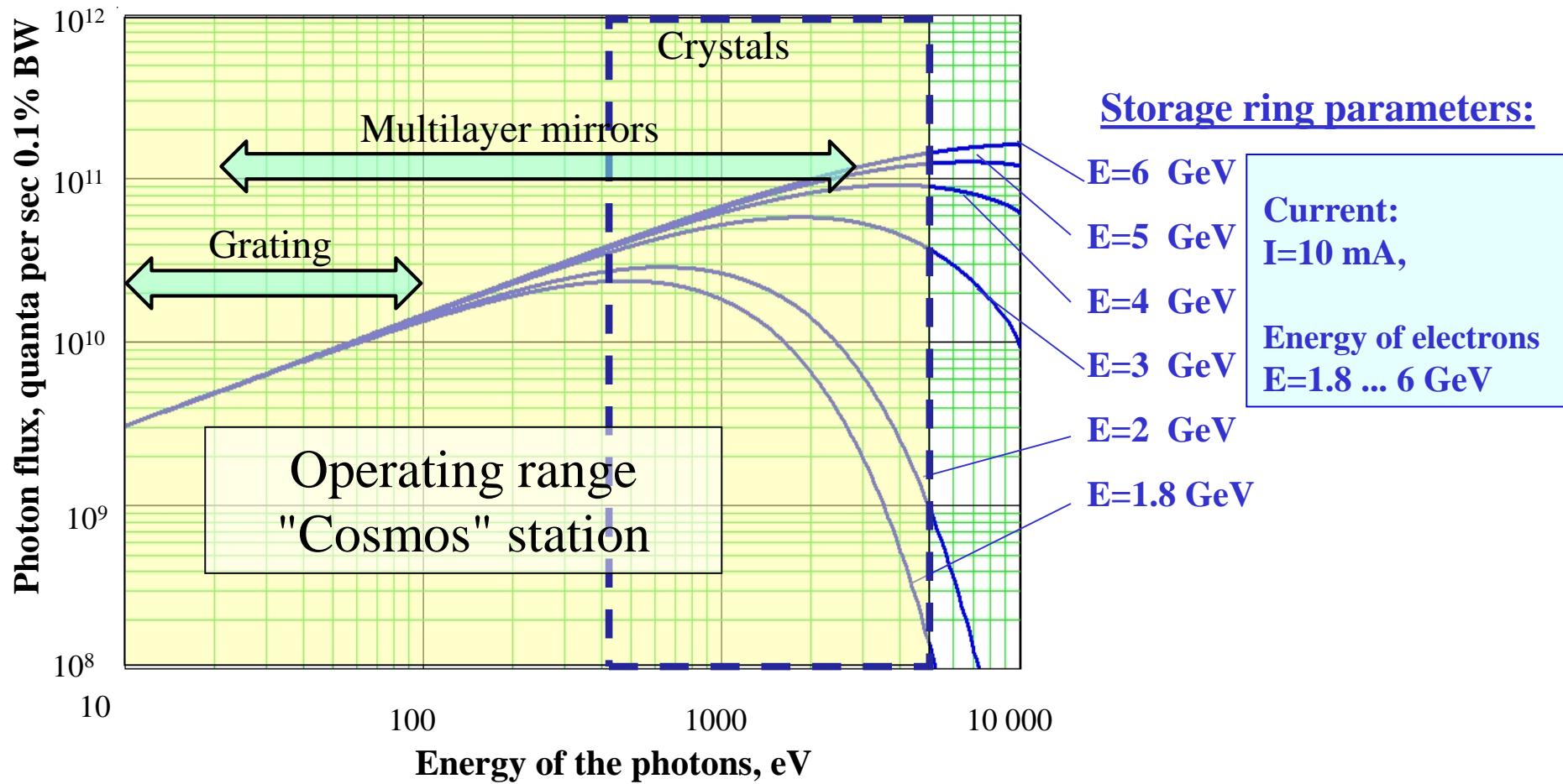
# "Cosmos" Beamline at the VEPP-4 SR experimental hall



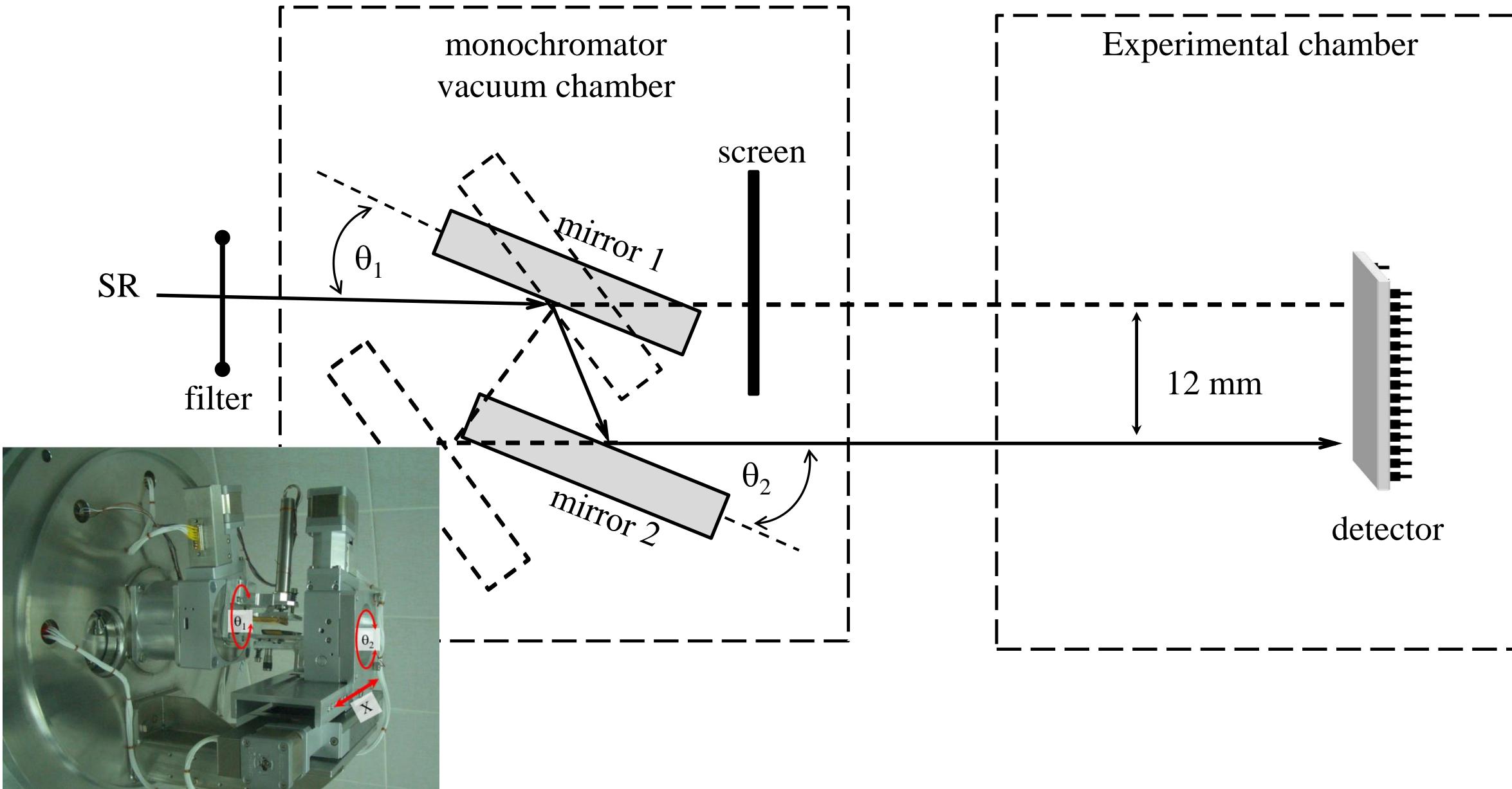
# Photon flux at the «Cosmos» station for different energy of electrons in the VEPP-4 storage ring.

## 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: **7°-80°**

Mirrors: **30x40 mm<sup>2</sup>**

Spectral range - **20-3000 eV**

Spectral resolution: **0.1-10%**

Fixed position of monochromatic beam

Vacuum - **10<sup>-4</sup>Pa**

## Two-mirror monochromator (multilayer mirrors & crystal)

### Parameters:

Angle range: **7°-80°**

Crystals: **Si (111)**

Spectral range - **2000-6000 eV**

Spectral resolution: **0.01%**

Fixed position of monochromatic beam

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

## Two-mirror monochromator (multilayer mirrors & crystal)

### Parameters:

Angle range: **7°-80°**

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

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

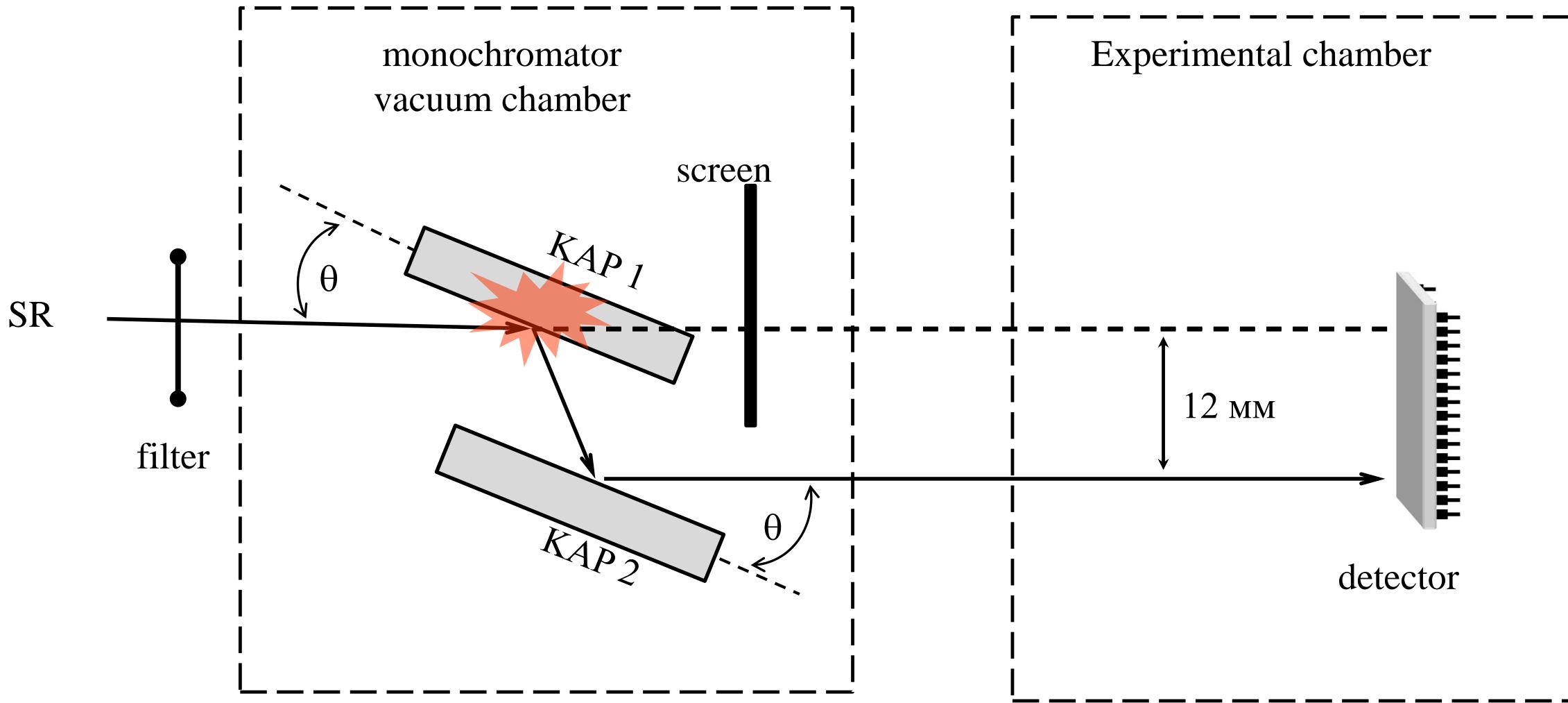
Spectral resolution: **0.01%-0.004% (?)**

Fixed position of monochromatic beam

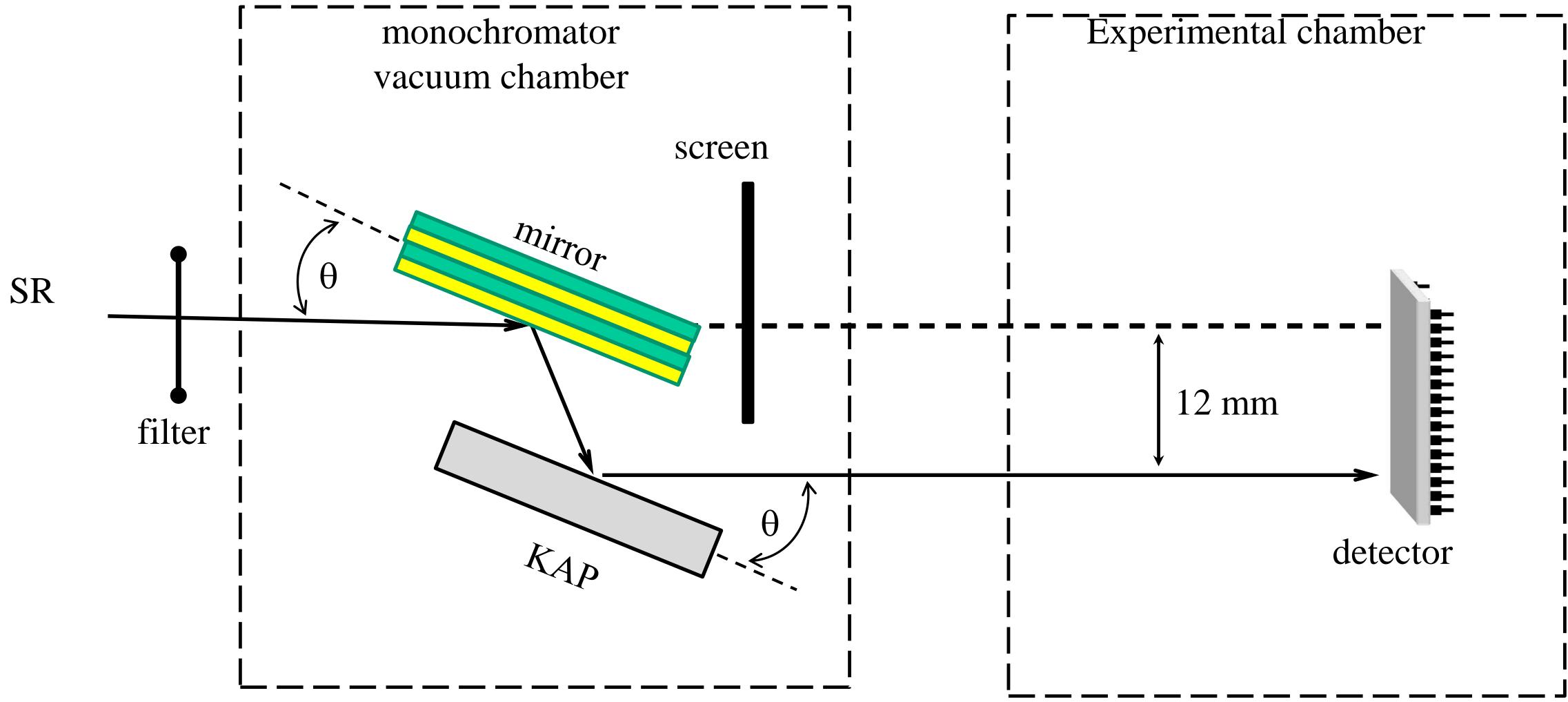
Vacuum - **10<sup>-4</sup>Pa**

# 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 ( $2d = 26.6 \text{ \AA}$ ) + KAP crystal  
 $E = 500 - 2\,000 \text{ eV}$

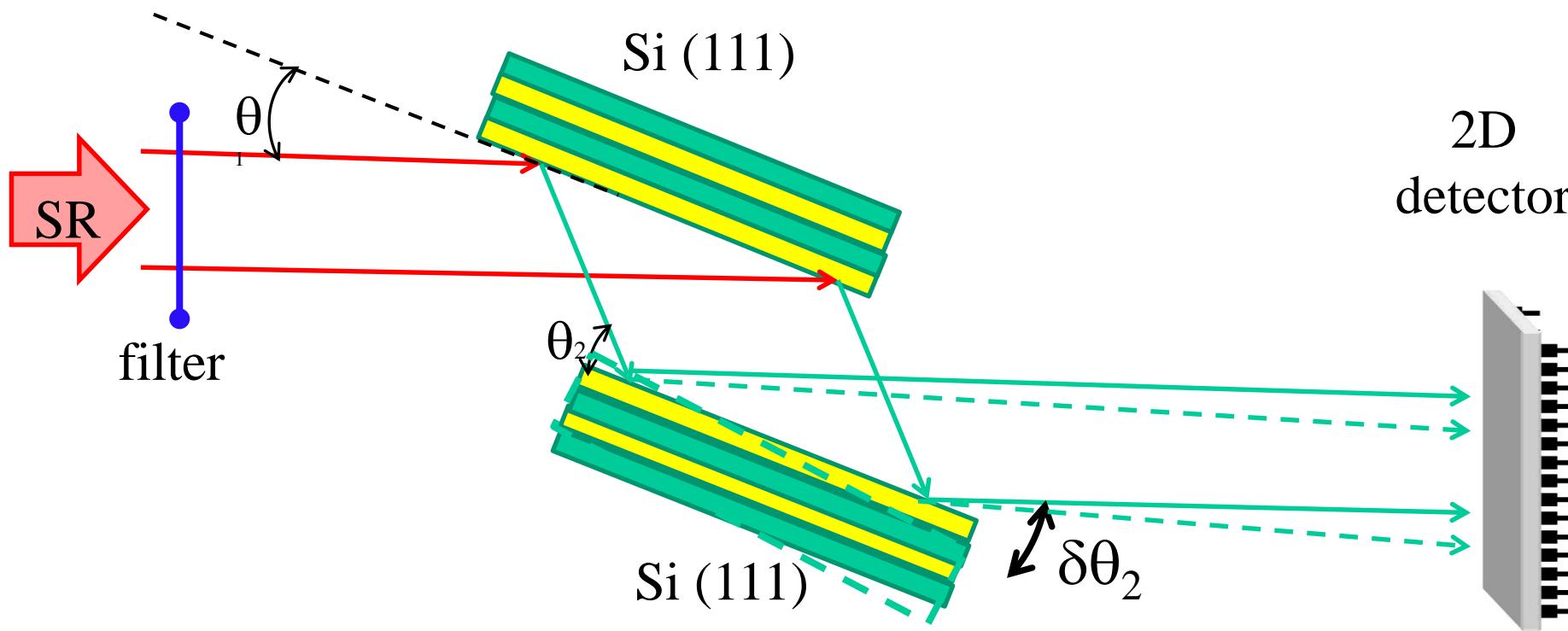


# ПЕРИОДИЧЕСКАЯ СИСТЕМА ЭЛЕМЕНТОВ Д. И. МЕНДЕЛЕЕВА

ПЕРИОДЫ	РЯДЫ	ГРУППЫ ЭЛЕМЕНТОВ																											
		A I В	A II В	A III В	A IV В	A V В	A VI В	A VII В	В	VIII	A																		
1	1	H водород 1,01							(H)									He гелий 2 4,00											
2	2	Li литий 3 6,94	Be бериллий 4 9,01	B бор 5 10,81	C углерод 6 12,01	N азот 7 14,01	O кислород 8 16,00	F фтор 9 19,00										Ne неон 10 20,18											
3	3	Na натрий 11 22,99	Mg магний 12 24,31	Al алюминий 13 26,98	Si кремний 14 28,0	P фосфор 15 30,97	S сера 16 32,06	Cl хлор 17 35,45										Ar аргон 18 39,95											
4	4	K калий 19 39,10	Ca кальций 20 40,08	Sc скандий 21 44,96	Ti титан 23 47,90	V ванадий 24 50,94	Cr хром 25 54,94	Mn марганец 26 55,05	Fe железо 27 56,93	Co cobальт 28 58,70	Ni никель 29 58,69																		
	5	Cu медь 29 63,55	Zn цинк 30 63,93	Ga галлий 31 69,72	Ge германий 32 72,59	As мышьяк 33 74,92	Se себен 34 78,96	Br брон 35 79,90										Kr криpton 36 83,80											
5	6	Rb лучистый 37 85,47	Sr стеклянnyй 38 87,62	Y иттрий 39 88,91	Zr цирконий 40 91,22	Nb ниобий 41 92,91	Mo молибден 42 95,94	Tc технеций 43 98,91	Ru рутений 44 101,07	Rh родий 45 102,91	Pd палладий 46 106,42																		
	7	Ag серебро 47 107,87	Cd кальций 48 112,41	In индий 49 114,82	Sn олово 50 118,71	Sb сульфур 51 121,75	Te теллур 52 127,60	I йод 53 126,90										Xe ксенон 54 131,30											
6	8	Cs цезий 55 132,91	Ba барий 56 137,33	La* лантан 57 138,91	Hf гафний 72 178,49	Ta таантал 73 180,95	W вольфрам 74 183,84	Re рений 75 186,21	Os осмий 76 190,23	Ir иридий 77 192,22	Pt платина 78 195,09							Rn радон 86 [222]											
	9																												
7	10	высшие оксиды		летучие водородные соединения		лантаноиды		актиноиды		K – edges available using Si(111)		L – edges available using Si (111)		K – edges available using KAP (001)		RO <sub>3</sub>		RO <sub>4</sub>											
		Fr франций 79 [223]	Au руй 80 196,97	Hg ртуть 81 200,59	Tl таллий 82 204,38	Pb ракурс 83 207,20	Bi биген 84 208,99	Po полоний 85 [209]	At атомий 86 [210]	Bh богородия 87 [211]	Hs гассия 88 [212]	108 [213]	Hs гассия 109 [214]	Mt мейтнерий 110 [215]	Ds дормштадтий 111 [216]														
		Ce цирконий 88 [224]	Pr празеодим 89 [225]	Nd нейодим 90 [226]	Прометий 91 [227]	Самарий 92 [228]	Европий 93 [229]	Дисперзий 94 [230]	Тербий 95 [231]	Диспрозий 96 [232]	Больмий 97 [233]	Лютий 98 [234]	Эрбий 99 [235]	Ттулий 100 [236]	Тембрий 101 [237]	Людерсий 102 [238]	Лоуренсий 103 [239]												
		Th торий 90 232,03	Pa протактиний 91 231,04	U уран 92 238,03	Np нейптоний 93 [237]	Pu плутоний 94 [244]	Am америций 95 [243]	Cm курций 96 [247]	Bk берклий 97 [247]	Cf калифорний 98 [251]	Es зинштейний 99 [252]	Fm фермий 100 [257]	Md менделевий 101 [258]	No нобелий 102 [259]	Lr лоуренсий 103 [262]														
		ряд активности металлов		Li	Cs	Rb	K	Ba	Sr	Ca	Na	Mg	Be	Al	Mn	Zn	Cr	Fe	Cd	Co	Ni	Sn	Pb	H <sub>2</sub>	Cu	Hg	Ag	Pt	Au
		ряд напряжений металлов		Li	Rb	K	Ba	Sr	Ca	Na	Mg	Al	Mn	Zn	Cr	Fe	Cd	Co	Ni	Sn	Pb	H <sub>2</sub>	Sb	Cu	Hg	Ag	Pt	Au	

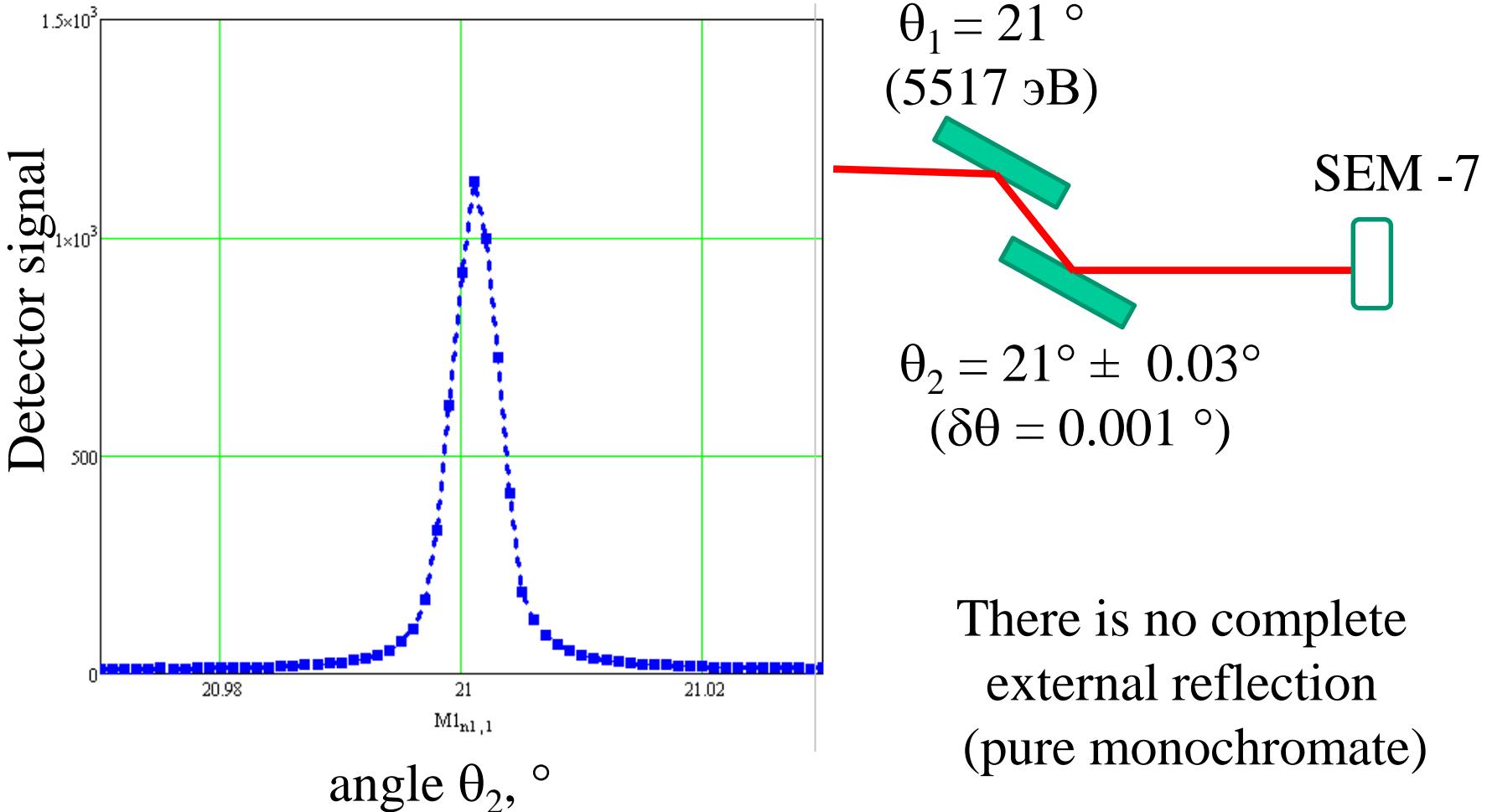
# Commissioning of the two-crystal monochromator

*measurement of the rocking curve*

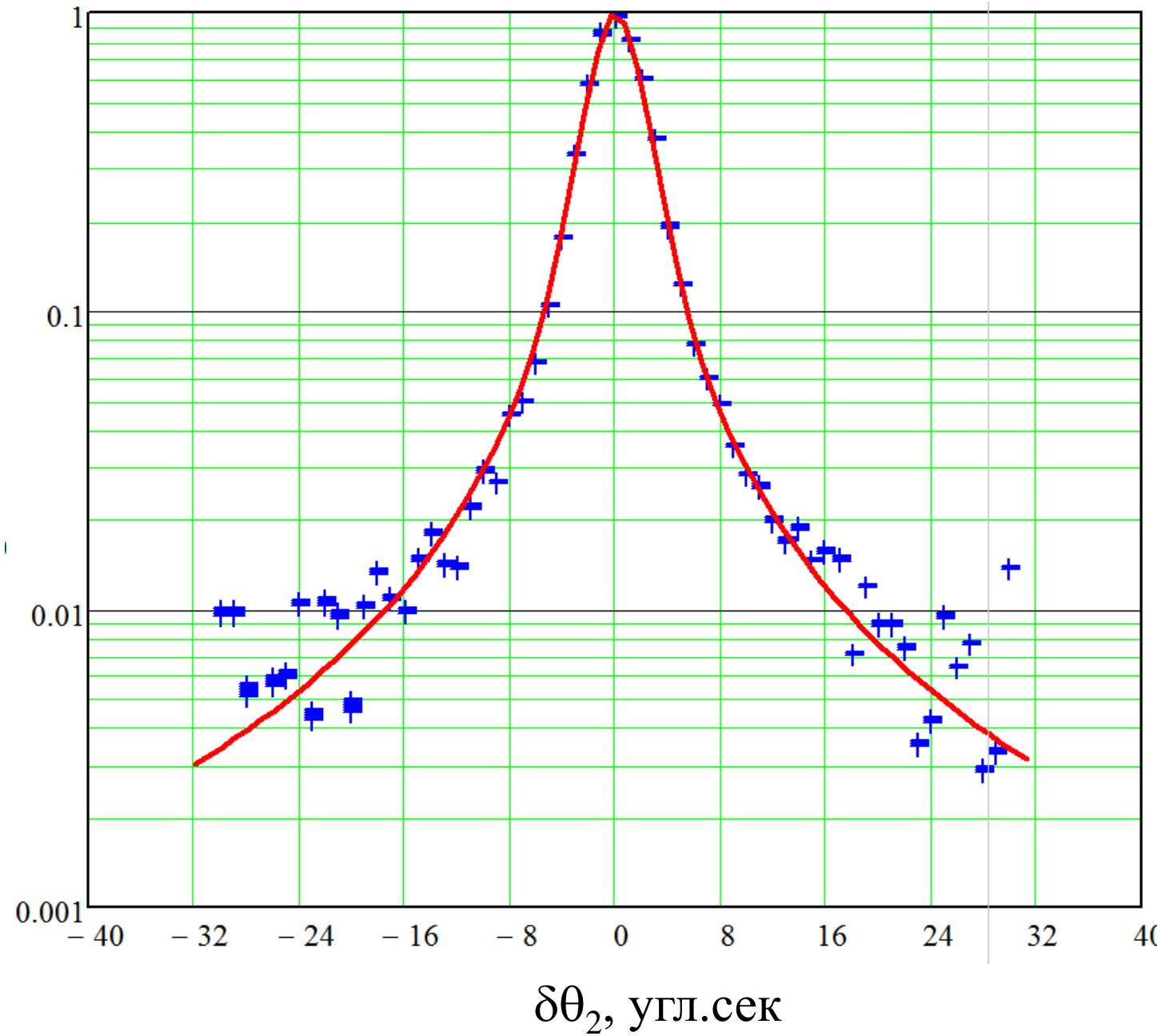


# Commission of the two-crystal monochromator

*measurement of the rocking curve*



# Approximation of the rocking curve by the pseudo-Voigt function



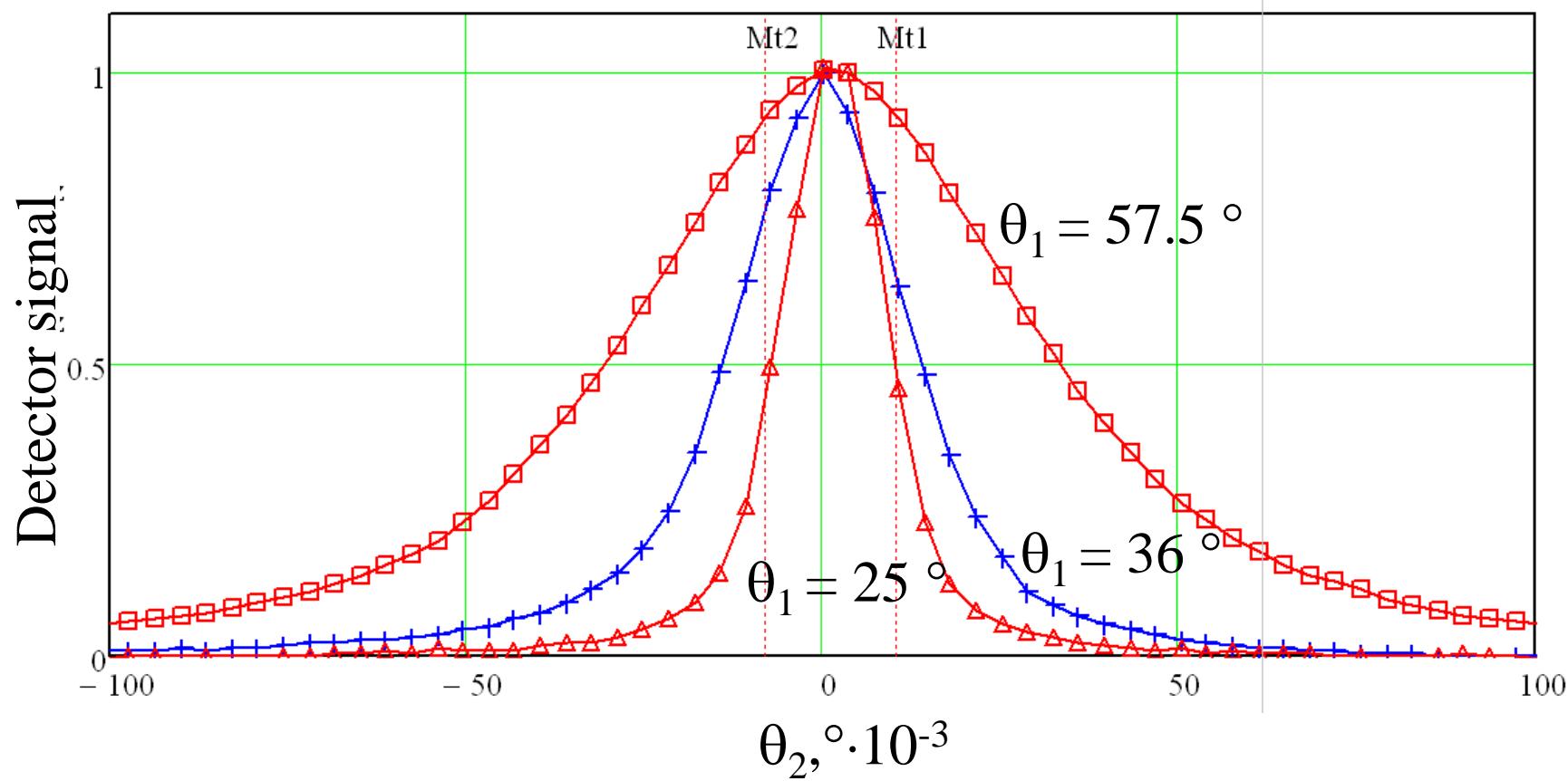
$$E_{ph} = 5\ 167 \text{ eV}$$

$$\theta_1 = 22.5^\circ$$

$$\text{FWHM } \delta\theta_2 = 16''$$

# Commission of the two-crystal monochromator

*measurement of the rocking curve Si(111)*



# Commission of the two-crystal monochromator

*measurement of the rocking curve*

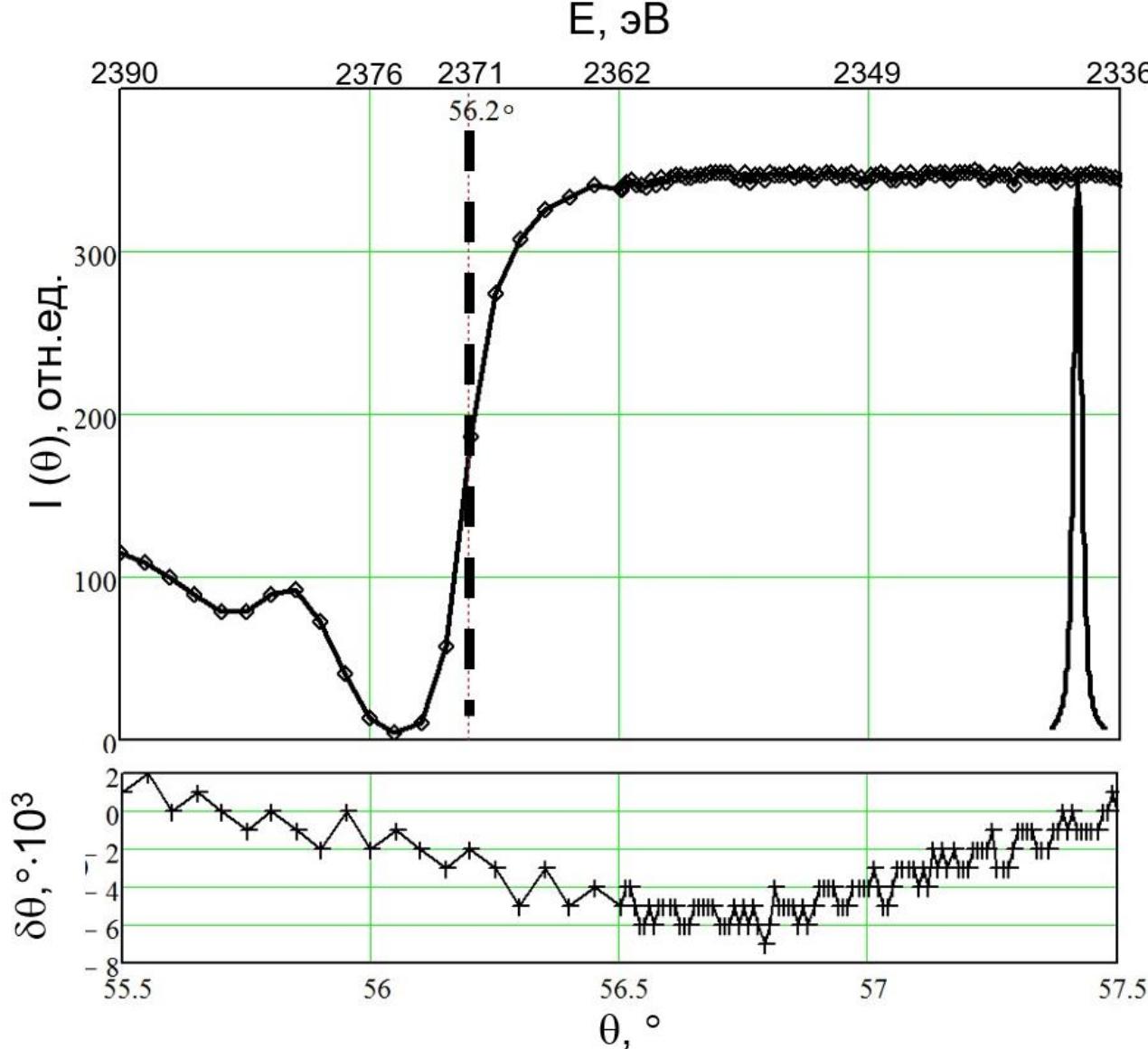
	<i>Width of the rocking curve, arc. sec.</i>	<i>Width of the rocking curve, eV</i>	<i>Photon energy, eV</i>	<i>rocking curve width</i>
$\theta_1$	$\Delta\theta_2$	$\Delta E_{rc}$	E	$\Delta E_{rc} / E$
24°	16,89"	0,926	4 843	$1,912 * 10^{-4}$
36°	29,93"	0,647	3 364	$1,922 * 10^{-4}$
57.5°	64,98"	0,459	2 345	$1,957 * 10^{-4}$

good  
concurrence

Measured spectral resolution of the double-crystal scheme :

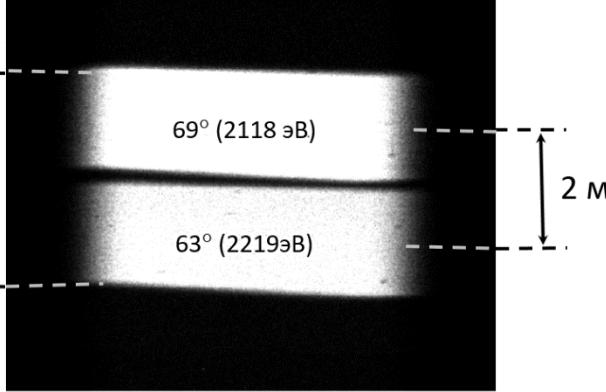
$$\frac{\Delta E_m}{E} = \frac{1}{\sqrt{2}} \cdot \frac{1}{\sqrt{2}} \cdot \frac{\Delta E_{rc}}{E} \approx 10^{-4}$$

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

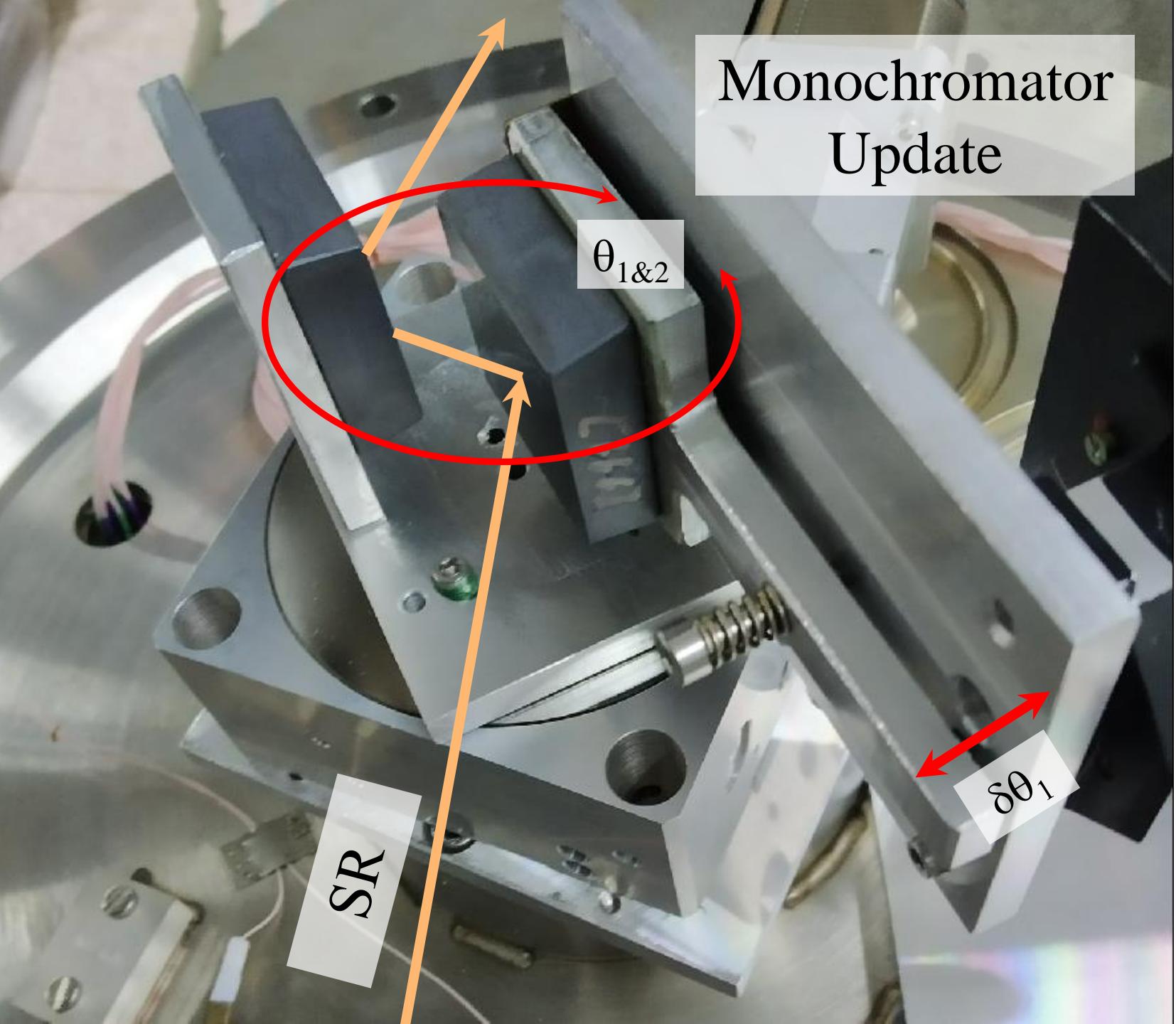


Since each crystal is driven by its own goniometer, a positioning error  $\delta\theta$  accumulates, leading to a violation of the setting for the Bragg condition. An attempt is made to automatically fine-tune the second crystal according by the signal amplitude.

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

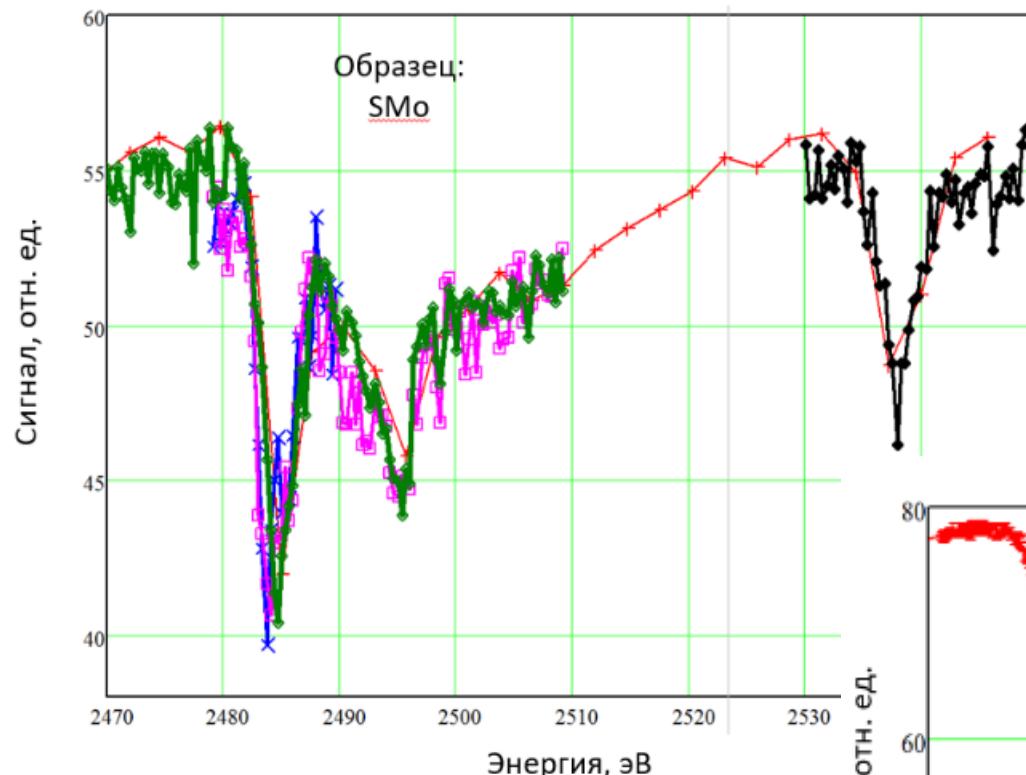


Vertical beam shift during scanning



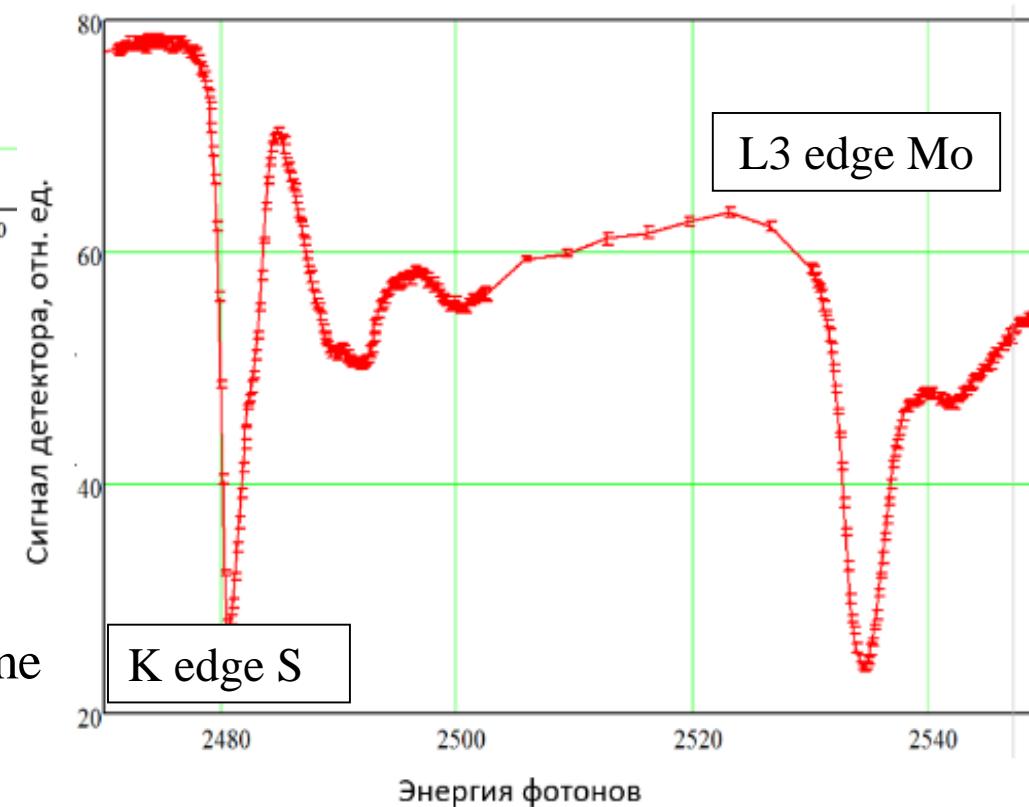
## Monochromator Update

# Monochromator Update Results:



scanning using the same sample

~ 20 seconds per point.



~4.5 seconds per point.

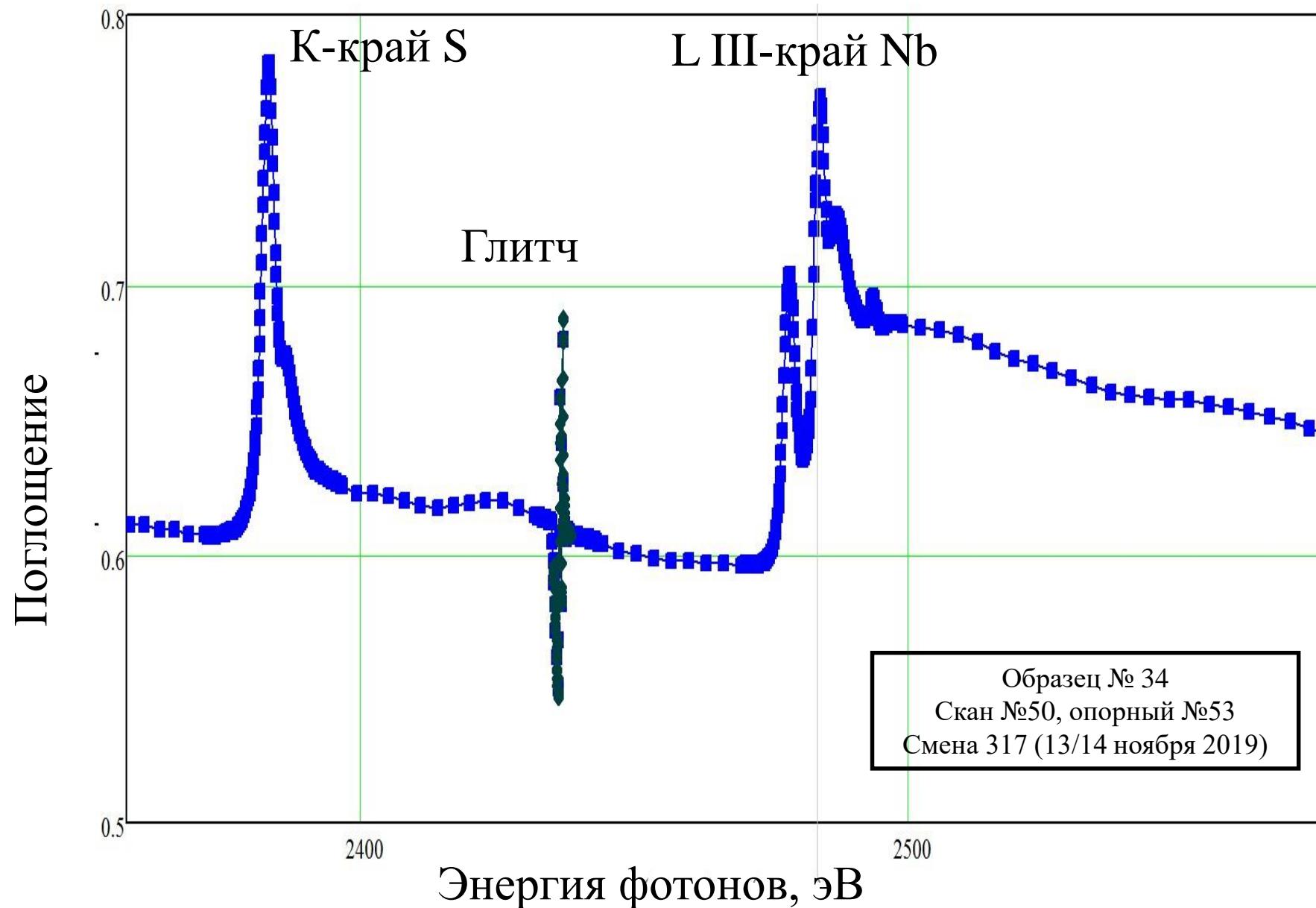
Spectrum acquisition time ~ 27 min.

significantly cleaner spectrum in less time

# Sample holder



2020/3/3 17:34

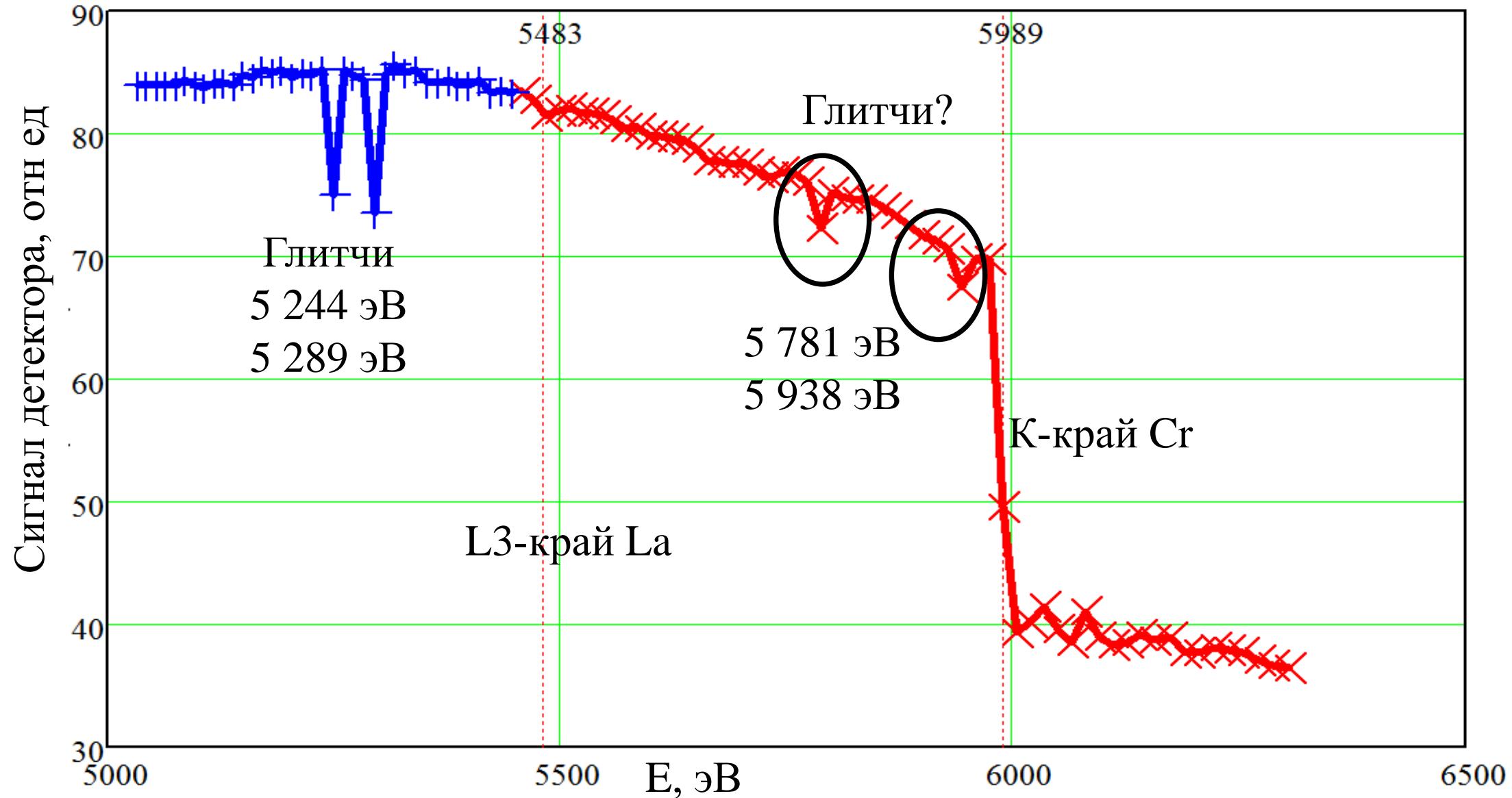


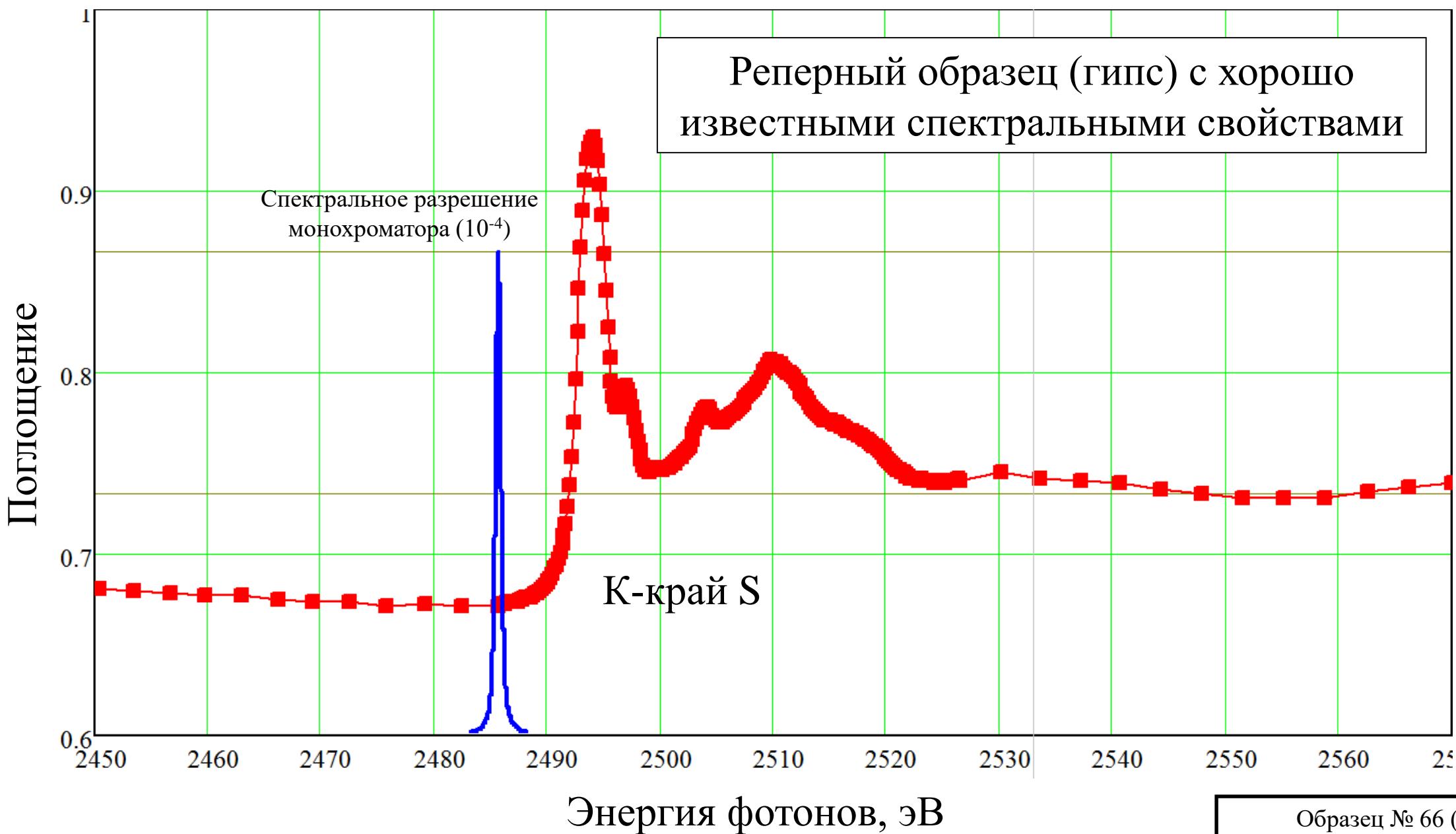
Биядерный кластер  $\{Nb_2S_4\}$  с мостиковыми дисульфидными лигандами  $S_2^{2-}$

# Попытка измерения спектра вблизи L3 края лантана

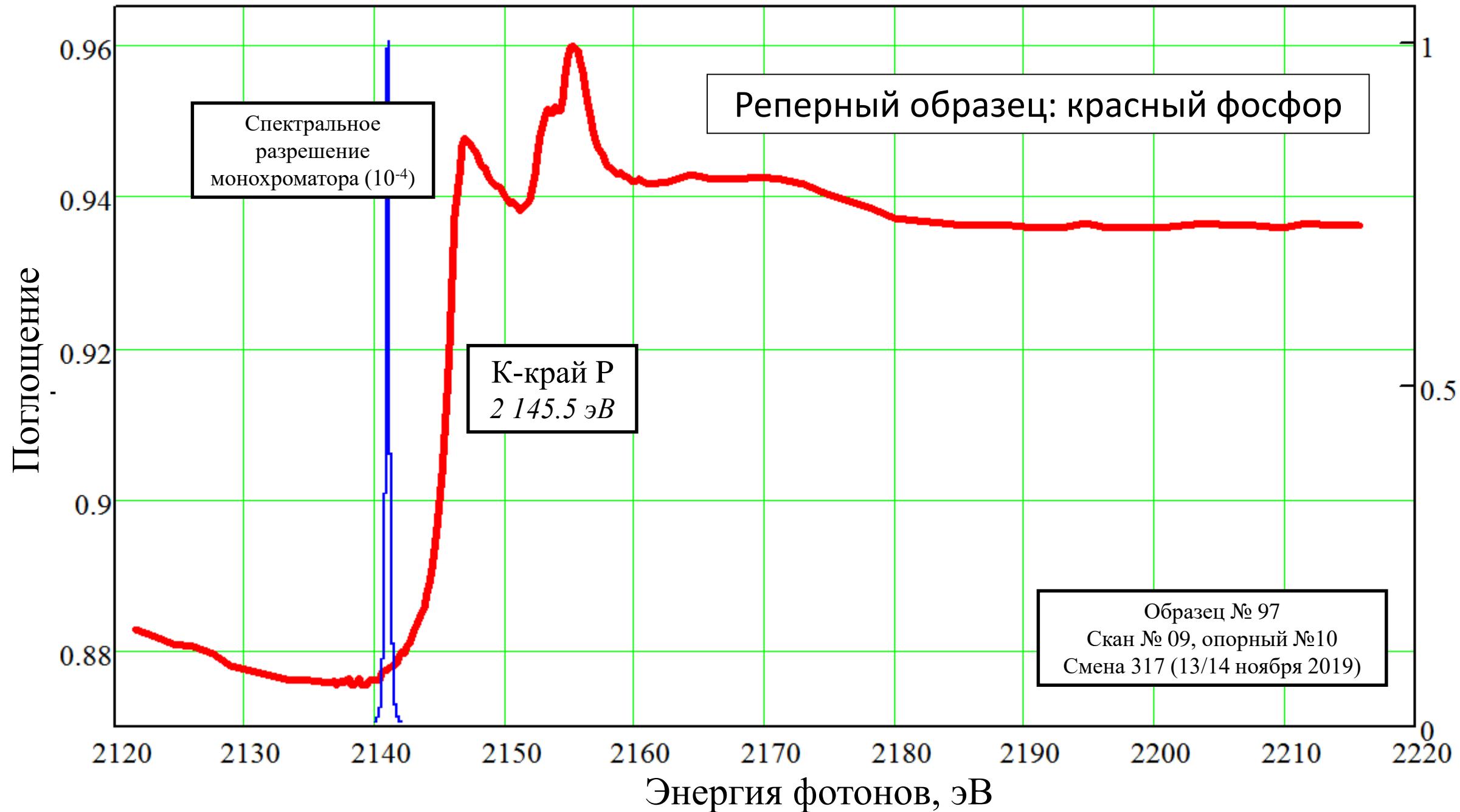
Образец CuCr<sub>0.99</sub>La<sub>0.01</sub>S<sub>2</sub>

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





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



Поглощение



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 L<sub>3</sub> 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