

# 50-я Международная Тулиновская конференция по физике взаимодействия заряженных частиц с кристаллами (ФВЗЧК-2021)

25 – 26 мая 2021, МГУ, Москва

## Модификация метода протон-индуцированной рентгеновской эмиссии применением плоских рентгеновских волноводов-резонаторов



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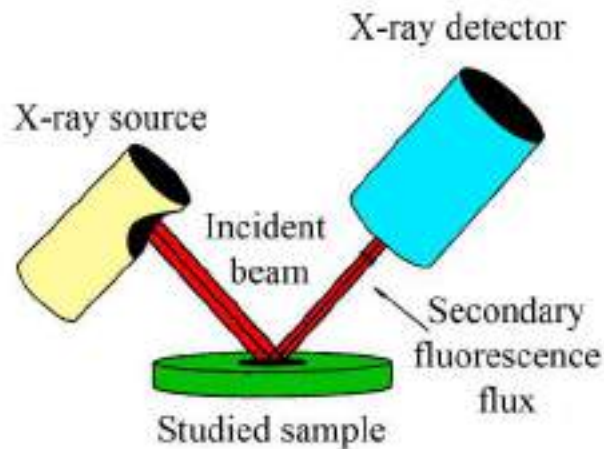
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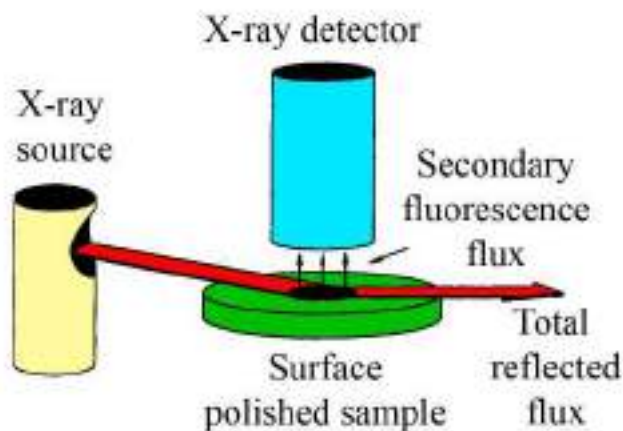
a

XRF



b

TXRF



Discovery of TXRF method:

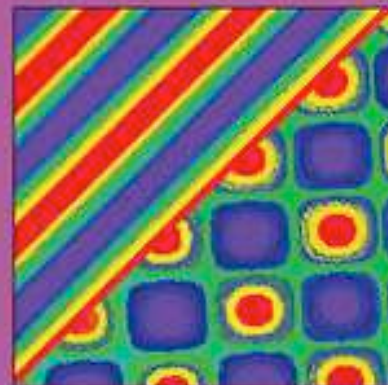
Y. Yoneda, T. Horiuchi, Optical flats for use in X-ray spectrochemical microanalysis // Rev. Sci. Instr., v42, 1971, pp. 1069-1070.

Chemical Analysis: A Series of Monographs on Analytical Chemistry and Its Applications

Mark F. Vitka, Series Editor

# Total-Reflection X-ray Fluorescence Analysis and Related Methods

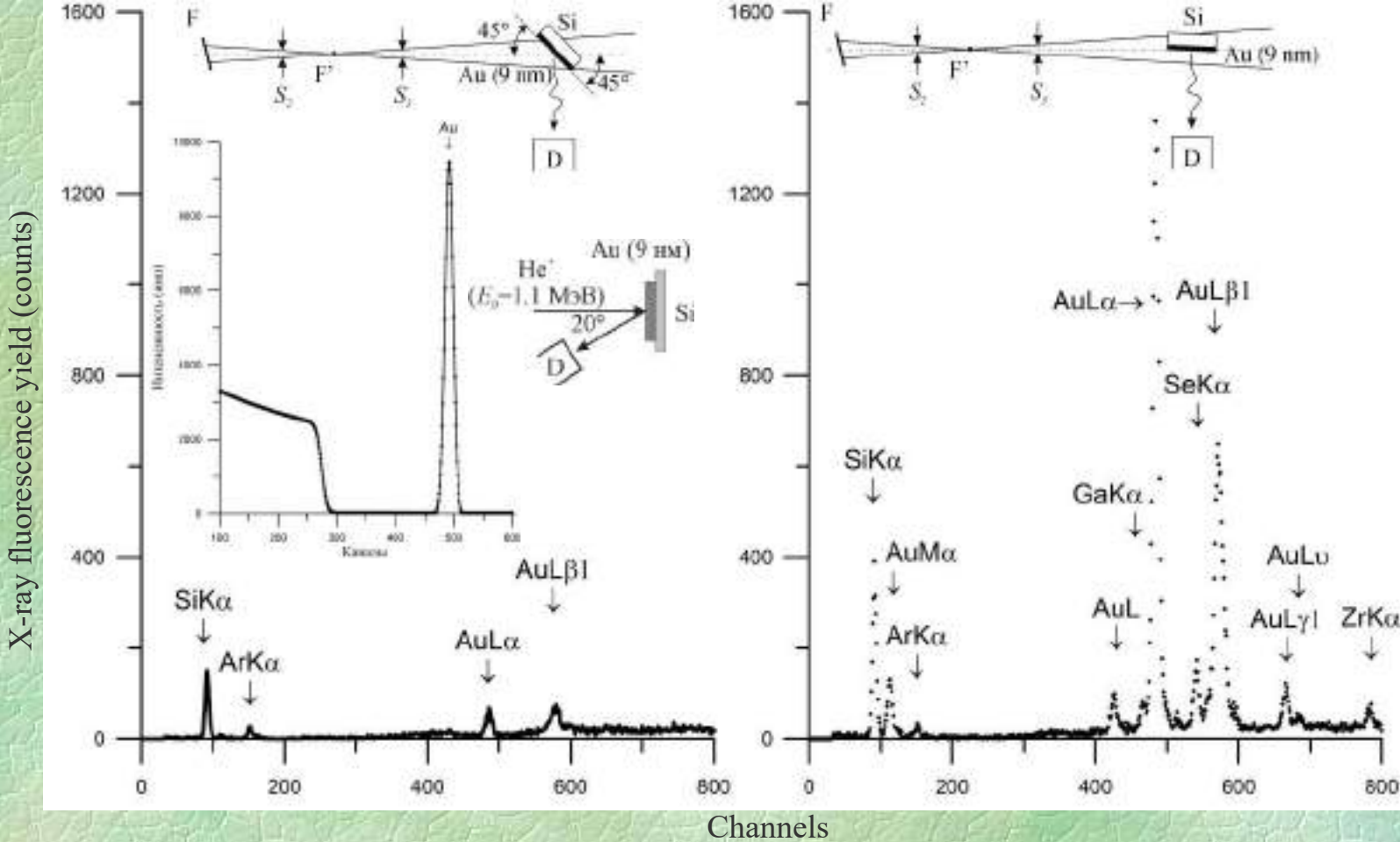
SECOND EDITION



REINHOLD KLOCKENKÄMPER  
ALEX VON BOHLEN

WILEY

# Spectra of X-ray fluorescence yield for Au (9 nm)/Si films structure collected in conditions of XRF standard geometry and in case of total external reflection of X-ray exciting flux

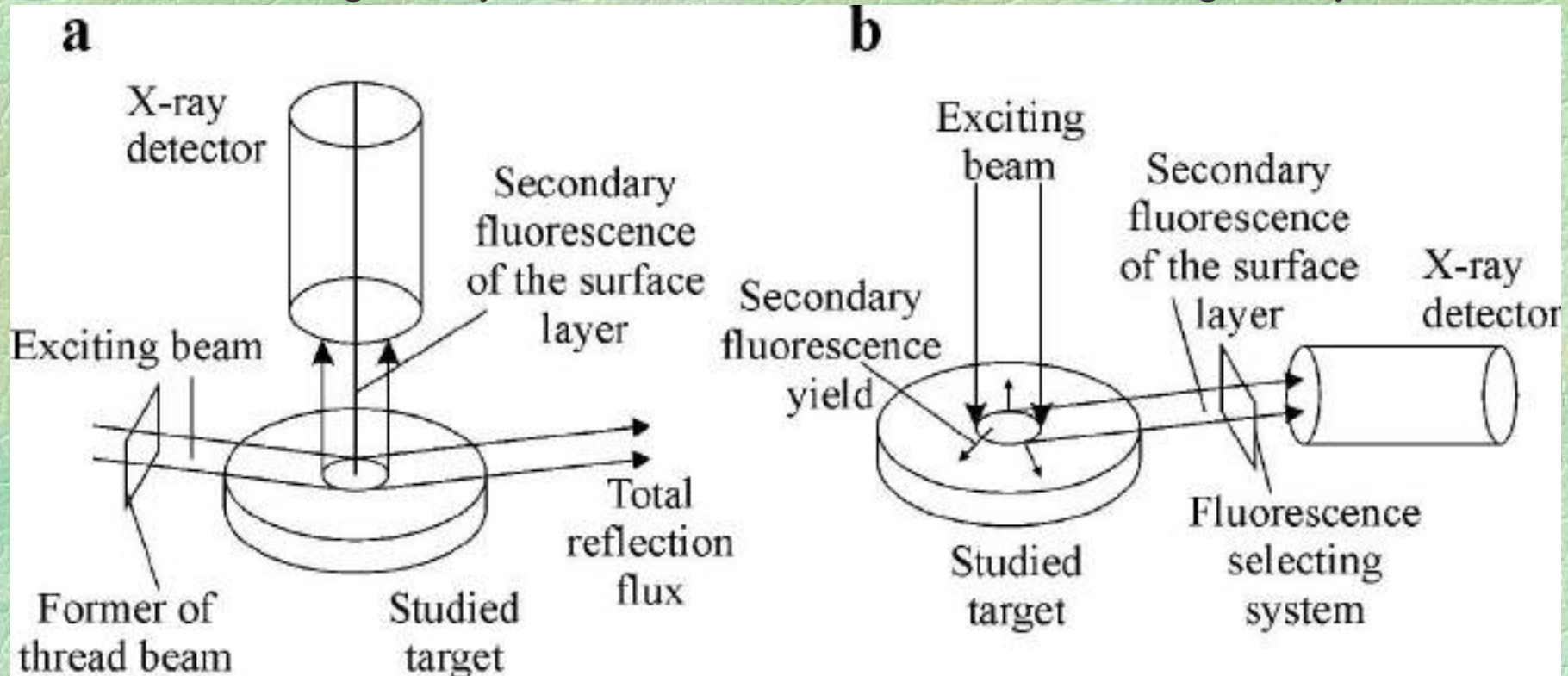


Measurement geometries and shown in the upper positions. RBS spectrum of He<sup>+</sup> ions for the target is shown on insertion. XRF spectra were collected at BSW (Mo) source conditions U=25 keV, I=10 mA, 300 sec. S<sub>1</sub>=S<sub>2</sub>=6 μm. Energy step 20 eV/channel.

# Schemes of X-ray fluorescence spectrometry at the total reflection exciting beam (TXRF) in direct and inversional geometries

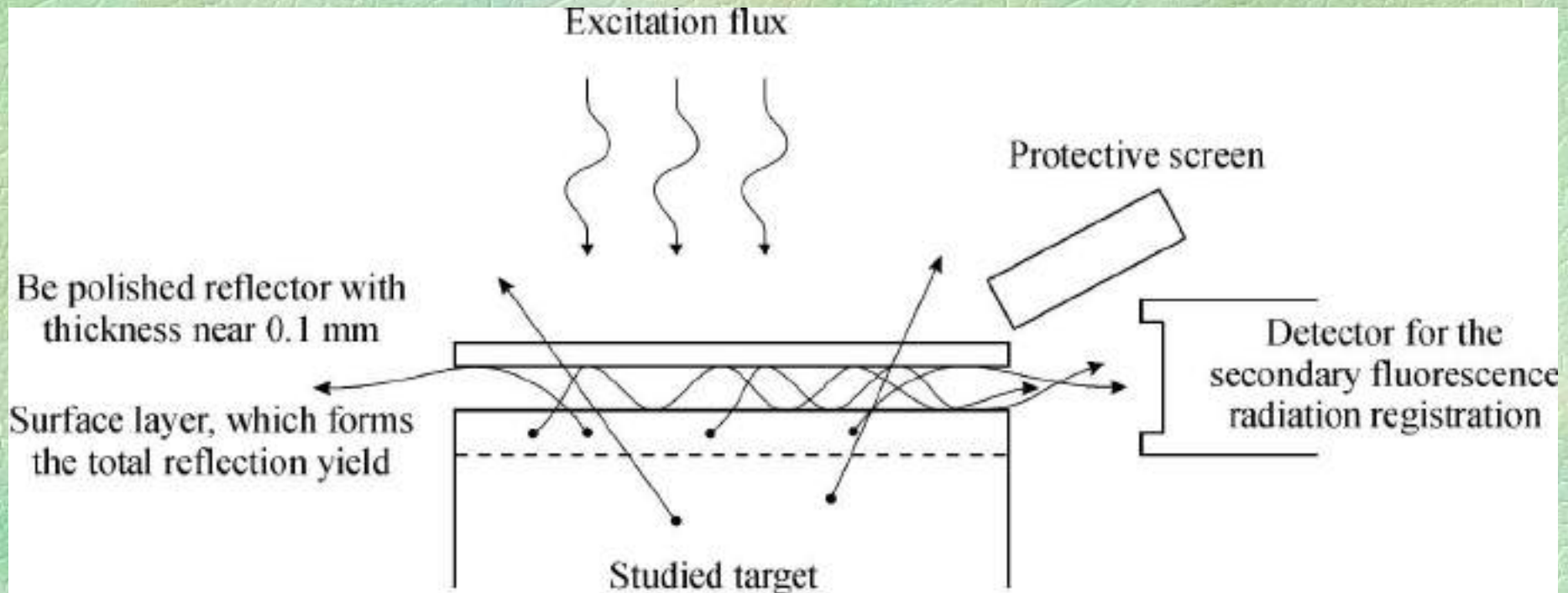
Direct geometry

Inversional geometry



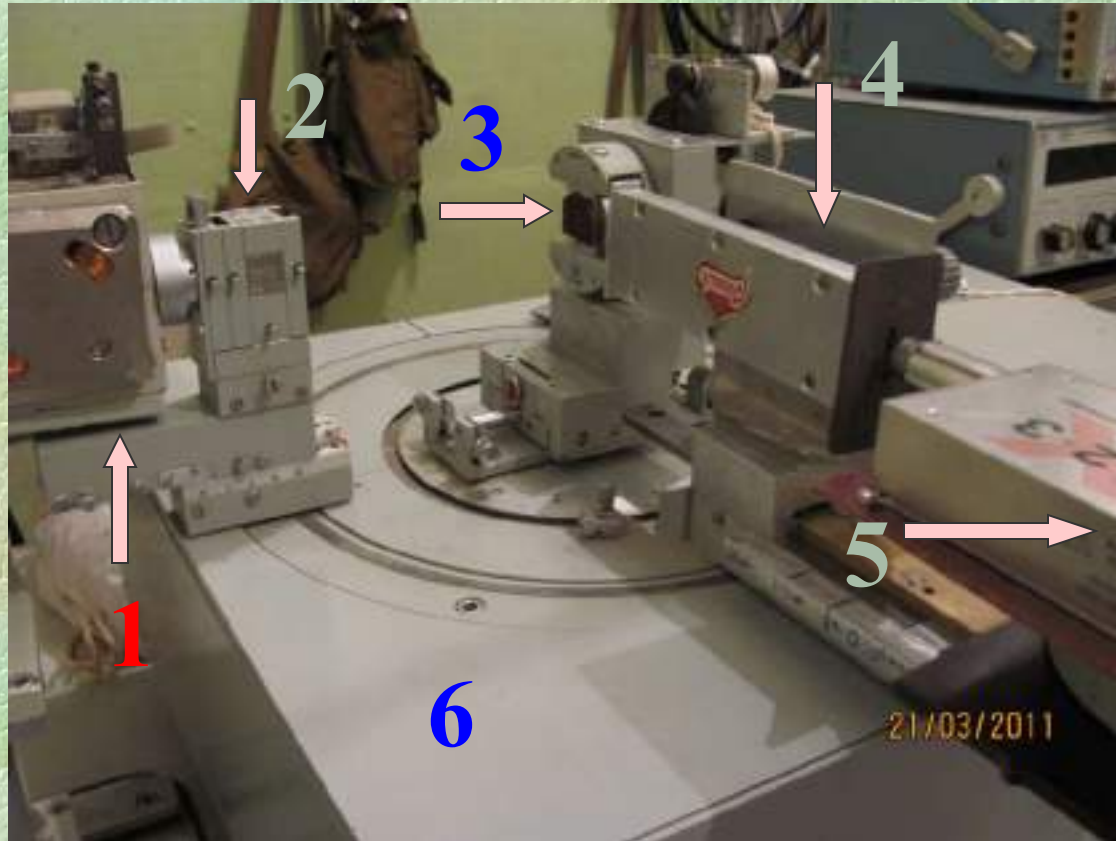
Direct and inversional geometries are suitable for the elemental analysis of thin film (3-5 nm) of target surface layer. The inversional geometry is characterized by enhanced sensitivity but is need in introduction of matrix correction. This geometry is very effective in case of the secondary fluorescence excitation by ions and electrons beams.

# Modified scheme of inversional TXRF spectrometry by planar X-ray waveguide-resonator application



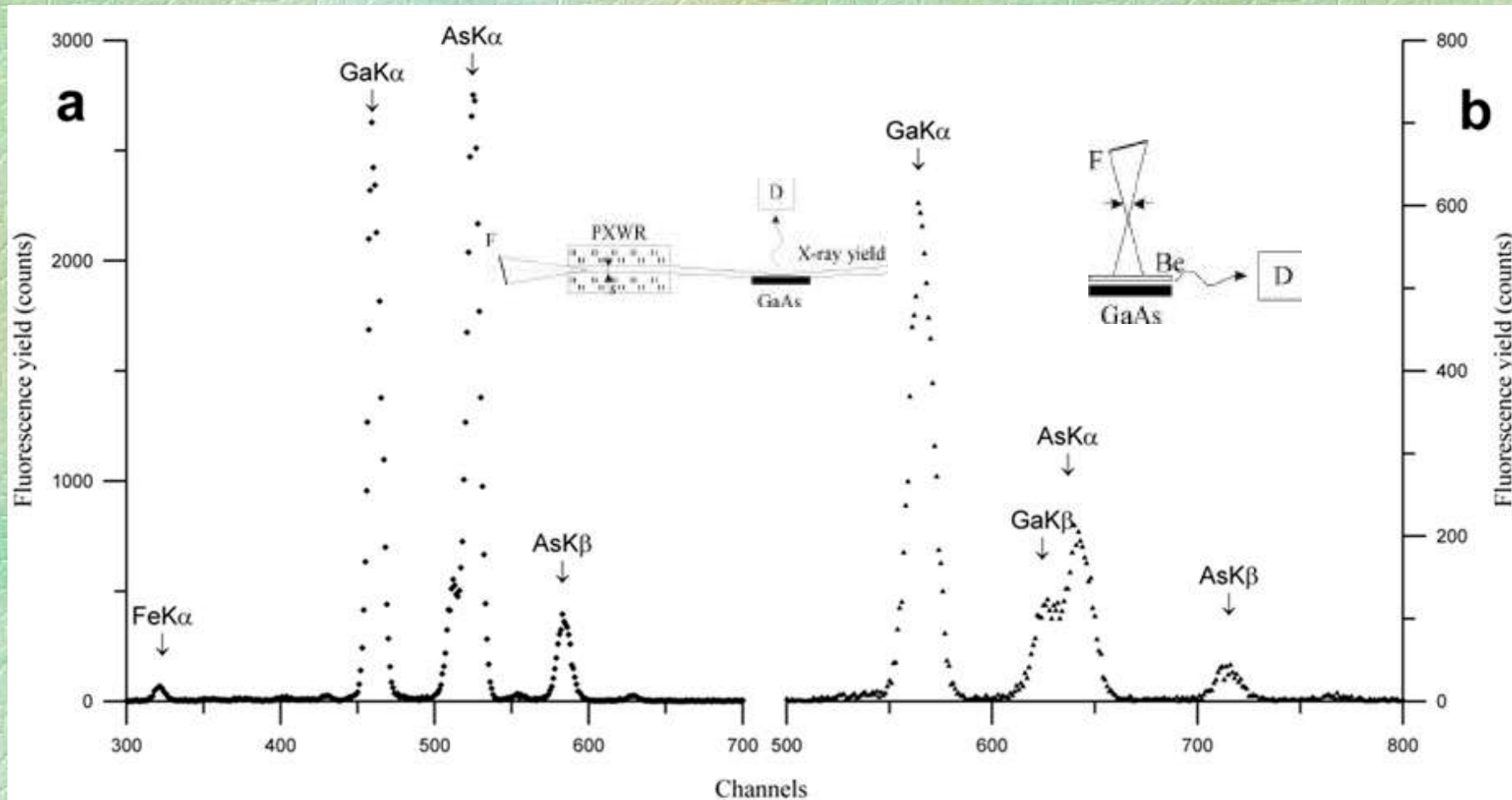
PXWR application allows collection of the total reflection yield in angular interval equal to the double critical angle of total reflection fluorescence radiation.

# Experimental stand for study of the inversional TXRF spectrometer at X-ray flux excitation of characteristic fluorescence



1. Radiation source BSW-24 (Mo); 2. Slit collimator of the exciting flux; 3. Studied target – GaAs crystal on diffractometrical attachment; 4. Double slit-cut collimator ( $S_1=S_2=1$  mm,  $l_{S_1S_2}=100$  mm) for X-ray fluorescence selection in conditions of standard inversional geometry; 5. X-ray fluorescence spectrometer 6. HZG-4 goniometer. At using of Be waveguide-resonator the double slit collimator (4) changes on the protective screen.

# TXRF spectra of GaAs stoichiometric target collected in direct (a) and inversion geometries (b)



Spectrum **a** – energy step 20 eV/channel. Spectrum **b** – energy step 16 eV/channel. Direct TXRF measurements characterize by absence of the matrix effect influence. Inversion geometry is not free from the effect.

# Monographs about PIXE application

IAEA-TECDOC-1190

## Instrumentation for PIXE and RBS

INTERNATIONAL ATOMIC ENERGY AGENCY



IAEA

December 2000

## PIXE: *A Novel Technique for Elemental Analysis*

SVEN A. E. JOHANSSON

*Department of Nuclear Physics, Lund Institute of Technology, Lund, Sweden*

and

JOHN L. CAMPBELL

*Department of Physics, The University of Guelph, Guelph, Ontario, Canada*

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1988

В.М.КОЛДА · А.Н.ЗАЙНЕНКО · Р.В.ДМИТРЕНКО

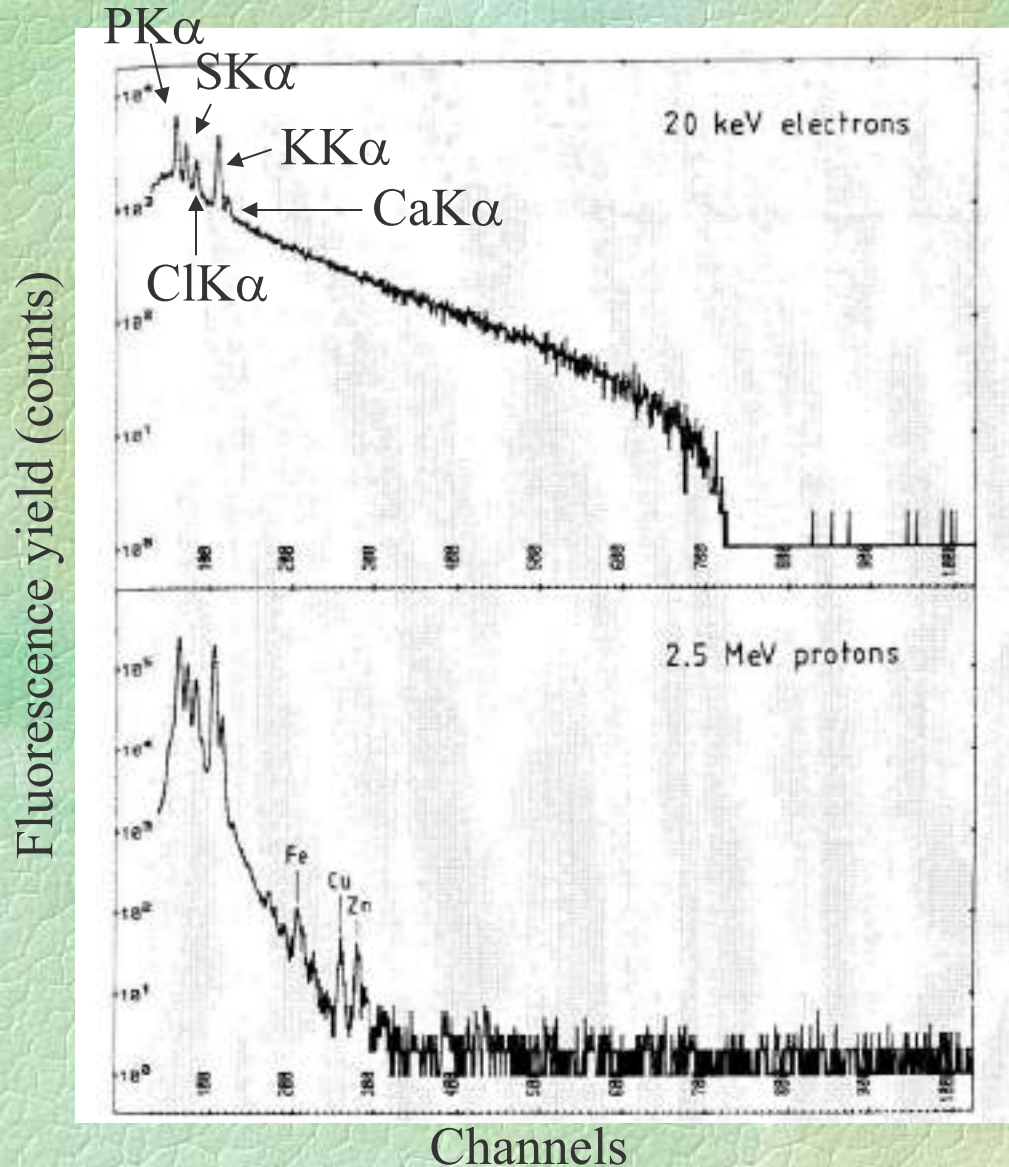
## РЕНТГЕНО- СПЕКТРАЛЬНЫЙ АНАЛИЗ С ИОННЫМ ВОЗБУЖДЕНИЕМ



МОСКВА · АТОМИЗДАТ · 1978



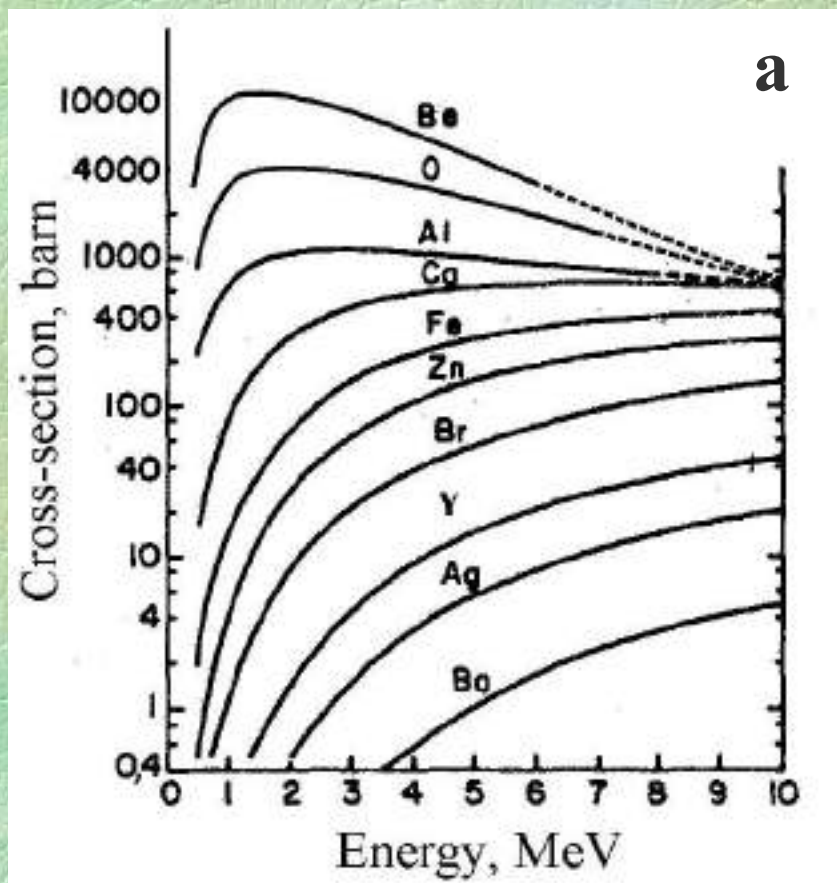
# X-ray fluorescence spectra of human brain tissue collected at electron and ion excitation



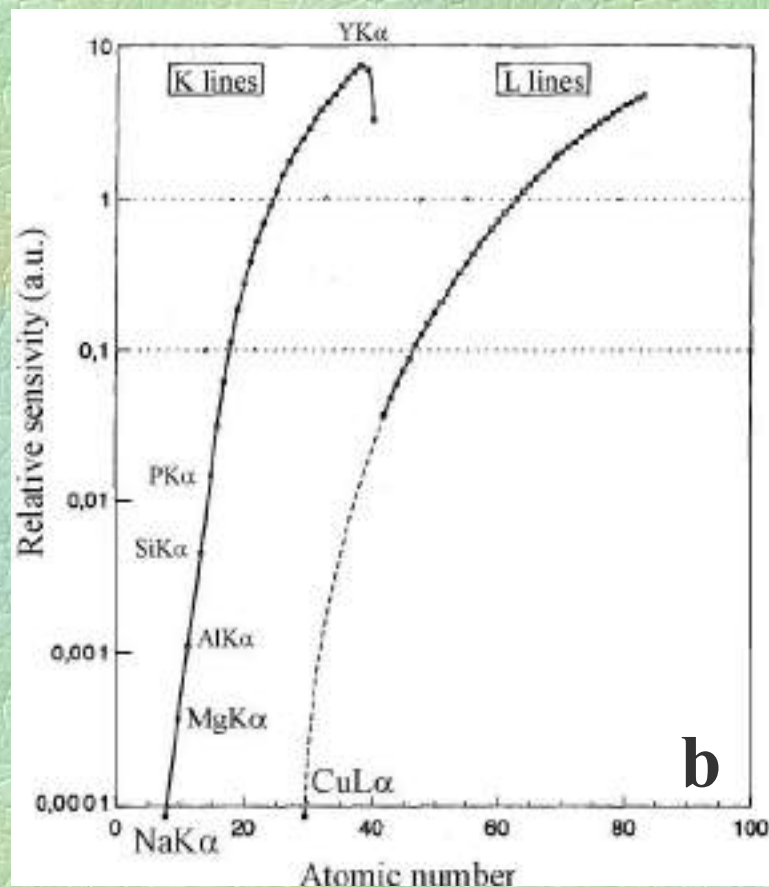
X-ray fluorescence spectrum collected in conditions of ion beam excitation shows decreasing of the background intensity. In result of it we can registrate additional elements (Fe, Cu, Zn), which are concealed by background initiated by bremsstrahlung of electron beam.

The results were received by prof. S.A.E. Johanson, J.L. Campbell.

# Cross-section of X-ray fluorescence excitation by proton beam and MoK $\alpha$ radiation flux



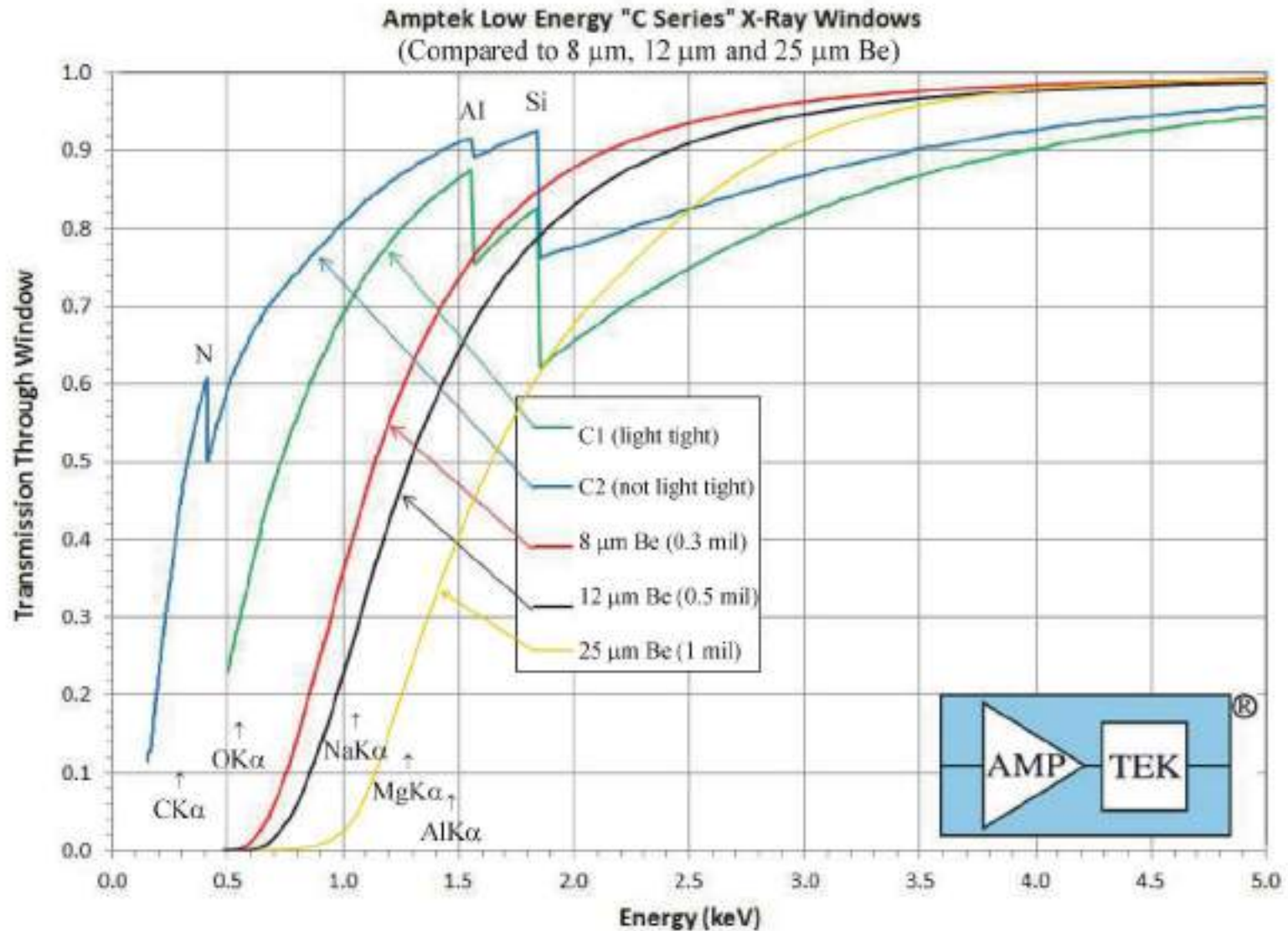
Excitation by H<sup>+</sup> beam with different energy.



Excitation by MoK $\alpha$  radiation ( $E_0=17.4$  keV).

Comparison of AlK $\alpha$  and YK $\alpha$  fluorescence excitation factors shows that Al detection is more effective by PIXE method in  $10^8$  times!

# Registration efficiency of X-ray radiation with different energy by detectors with Be and specific material entrance windows



# The experimental hall of ion beam analytical complex Sokol-3 built on base of electrostatic Van de Graaff accelerator ESU-2



## Complex parameters:

1. Ion beams:  $H^+$ ,  $D^+$ ,  $He^+$ ;
2. Energy range:  $0.05 \div 2.0$  MeV;
3. Current range:  
 $0.1 \div 50000$  nA  
( $6.25 \cdot 10^8 \div 3 \cdot 10^{14}$  ion/sec);
4. Beam spot:  $0.1 \div 5$  mm;
5. Vacuum:  $(1 \div 5) \cdot 10^{-6}$  torr;
6. Energy stability:  
 $0.03 \div 0.1\%$ ;
7. Current stability:  $3 \div 5\%$ ;
8. Radiation background:  
 $0.1 \div 3$  roentgen/hour.

# Control desk of ion beam analytical complex Sokol-3

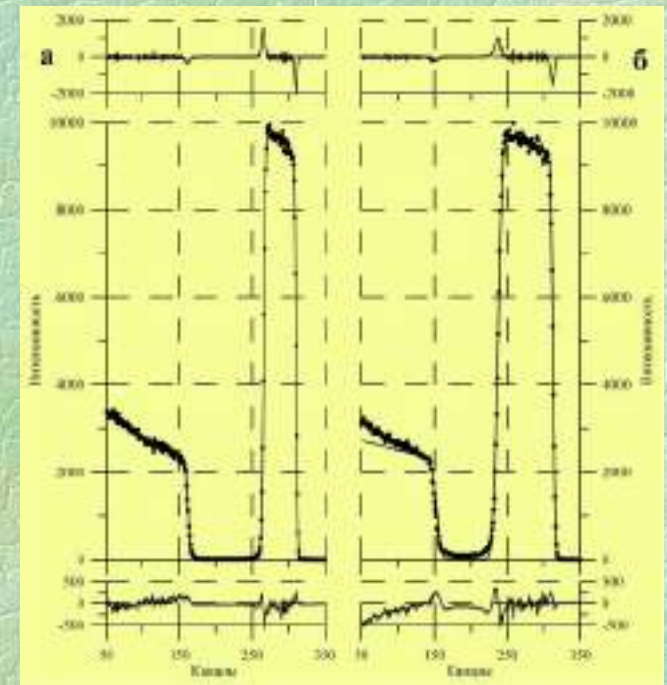
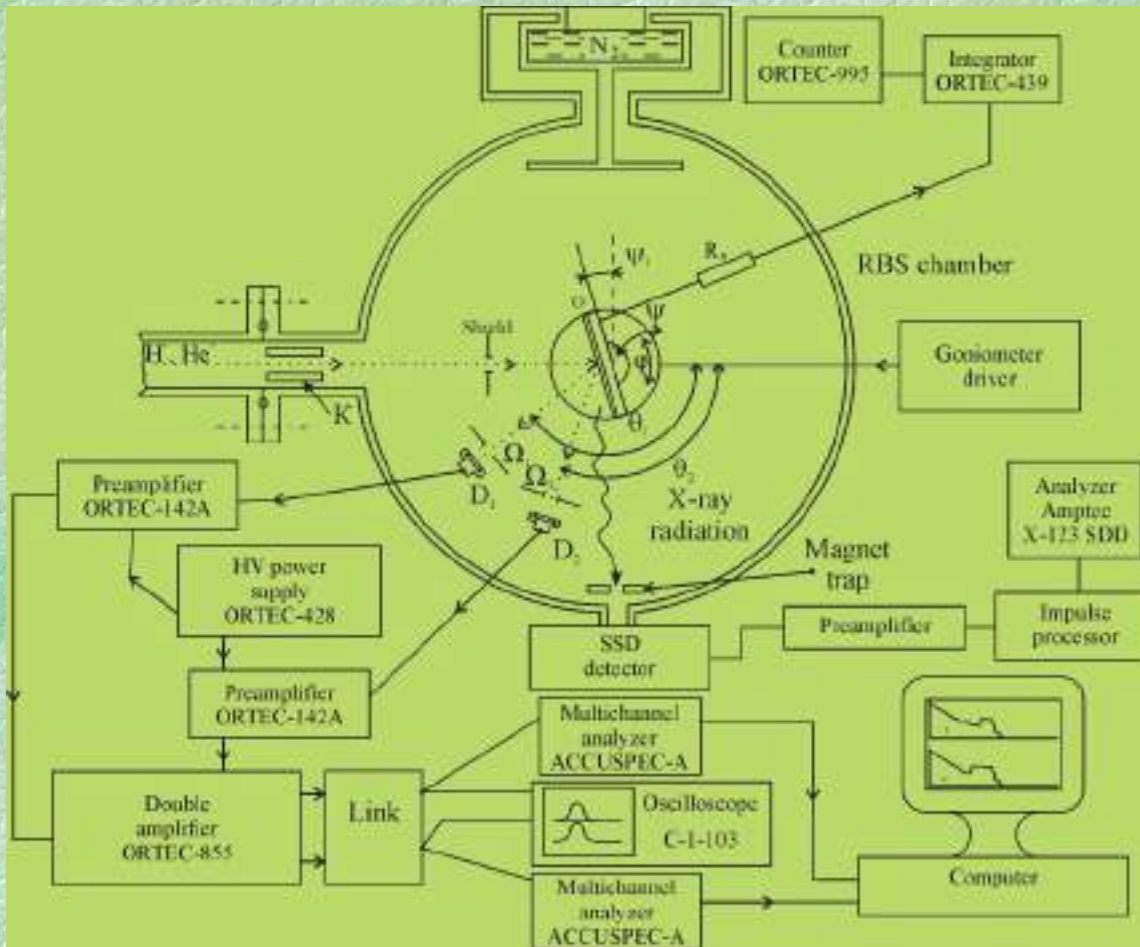


## Ion beam diagnostical method realized on the complex:

1. Rutherford backscattering spectrometry (RBS);
2. X-ray fluorescence analysis at ion excitation (PIXE);
3. Nuclear reaction analytical spectrometry (NRA);
4. Optical luminescence analysis (IBLA);
5. Method of nuclear elastic recoil detection (ERD);
6. Ion channeling method for study of monocrystal and epitaxial structures.

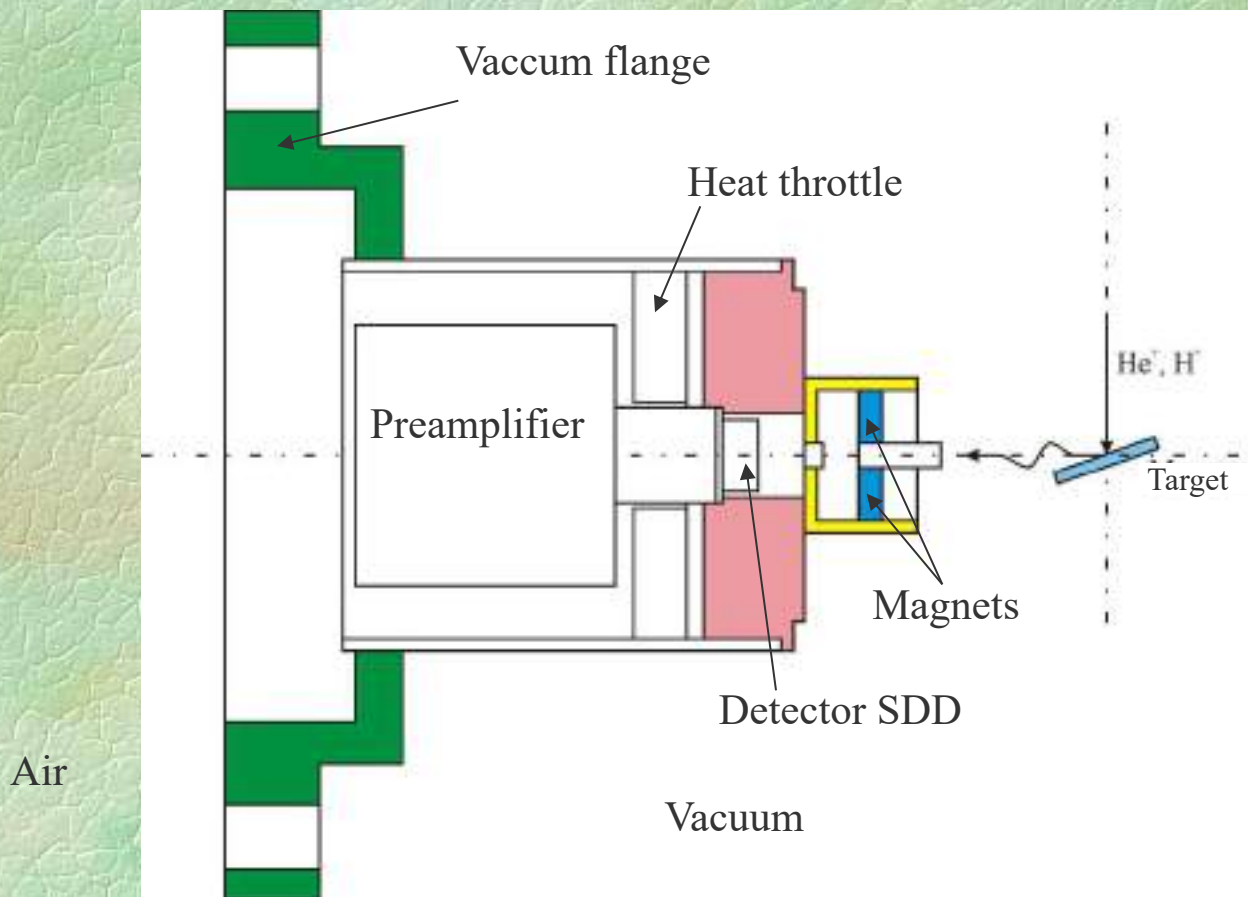
# Analytical chamber with two scattering ions registration channels and X-ray collector

Pattern of double angular RBS  $\text{He}^+$  ( $E_0=1.5 \text{ MeV}$ ) registration for Nb thin film on Si



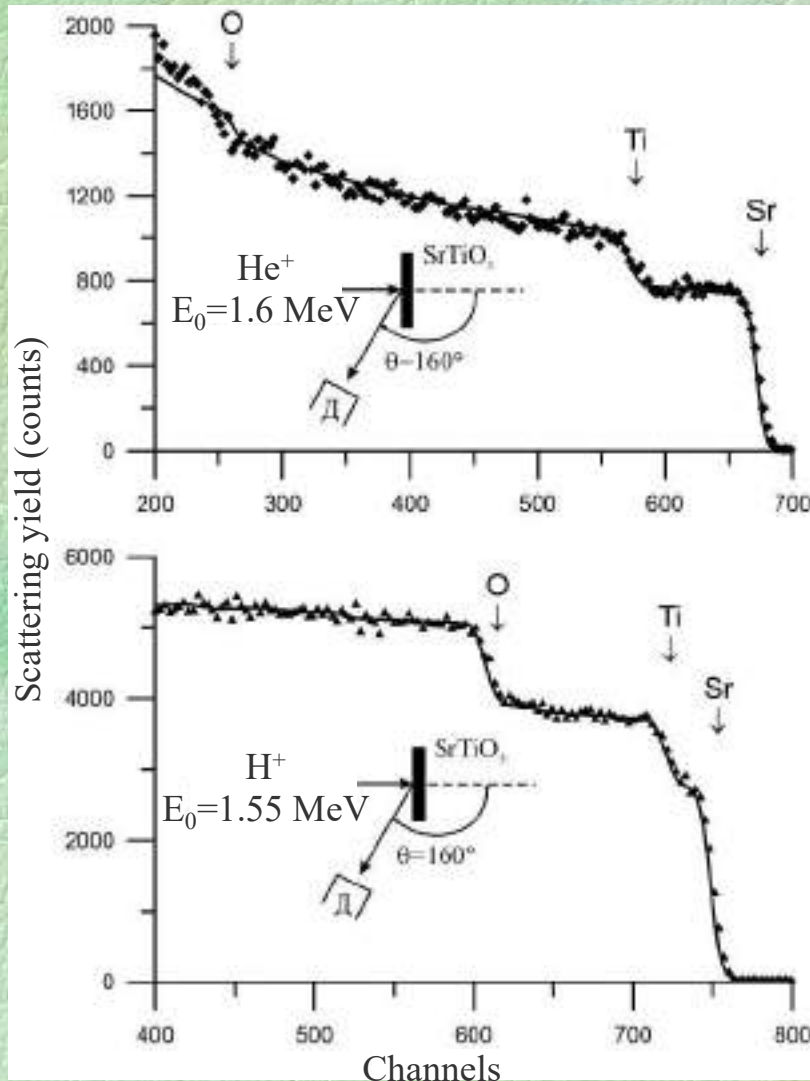
Film thickness  
 $t=135.2 \pm 0.7 \text{ nm}$

# Vacuum holder of X-123 Amptec SDD in the experimental chamber of Sokol-3 ion beam analytical complex



The construction supplies vacuum consolidation of the detector body the heat throttling and protection from electrons and ions

# Theoretical and experimental RBS spectra of $H^+$ and $He^+$ for $SrTiO_3$ monocrystal target (for nonoriented state)



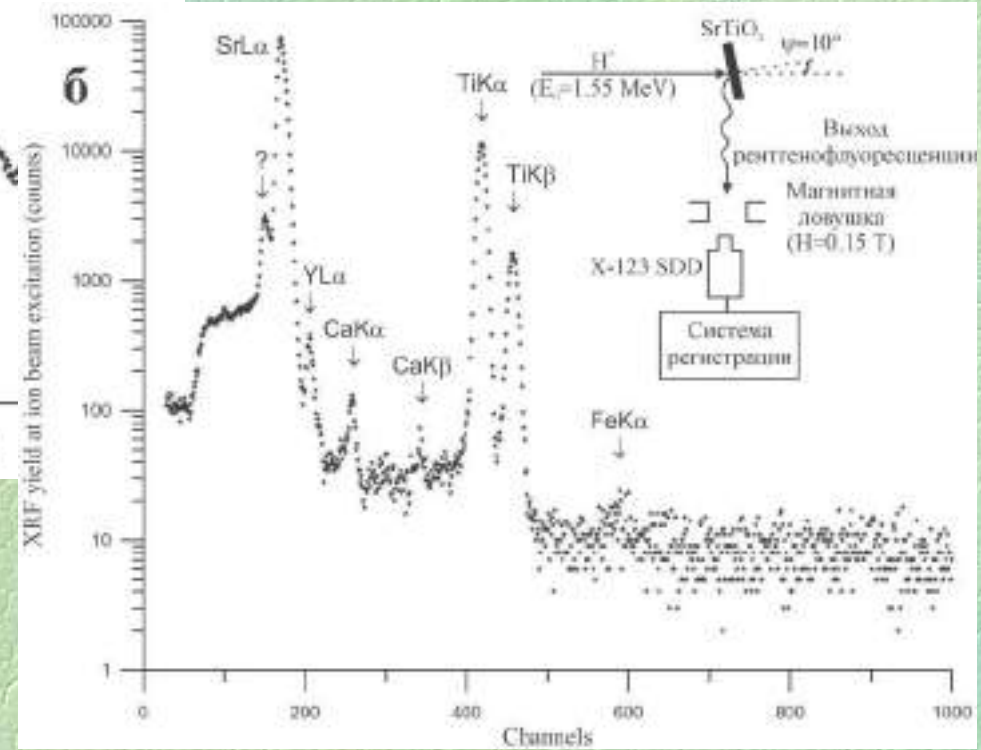
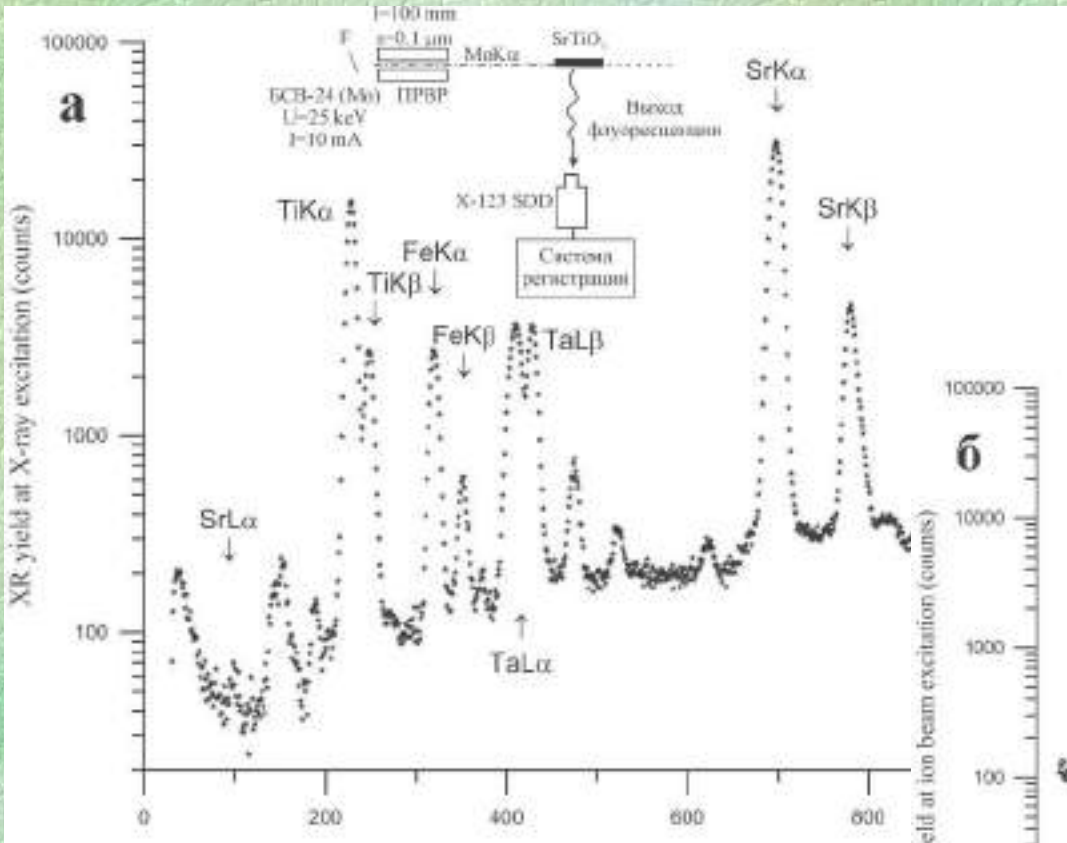
a

RBS spectra of helium and hydrogen for  $SrTiO_3$  monocrystal. Arrows show the scattering energies of ions on atoms located on the target's surface. Geometries of measurements are shown on inserts. There are presented every third channel. Energy cost of the channel is 1.9 keV/channel.

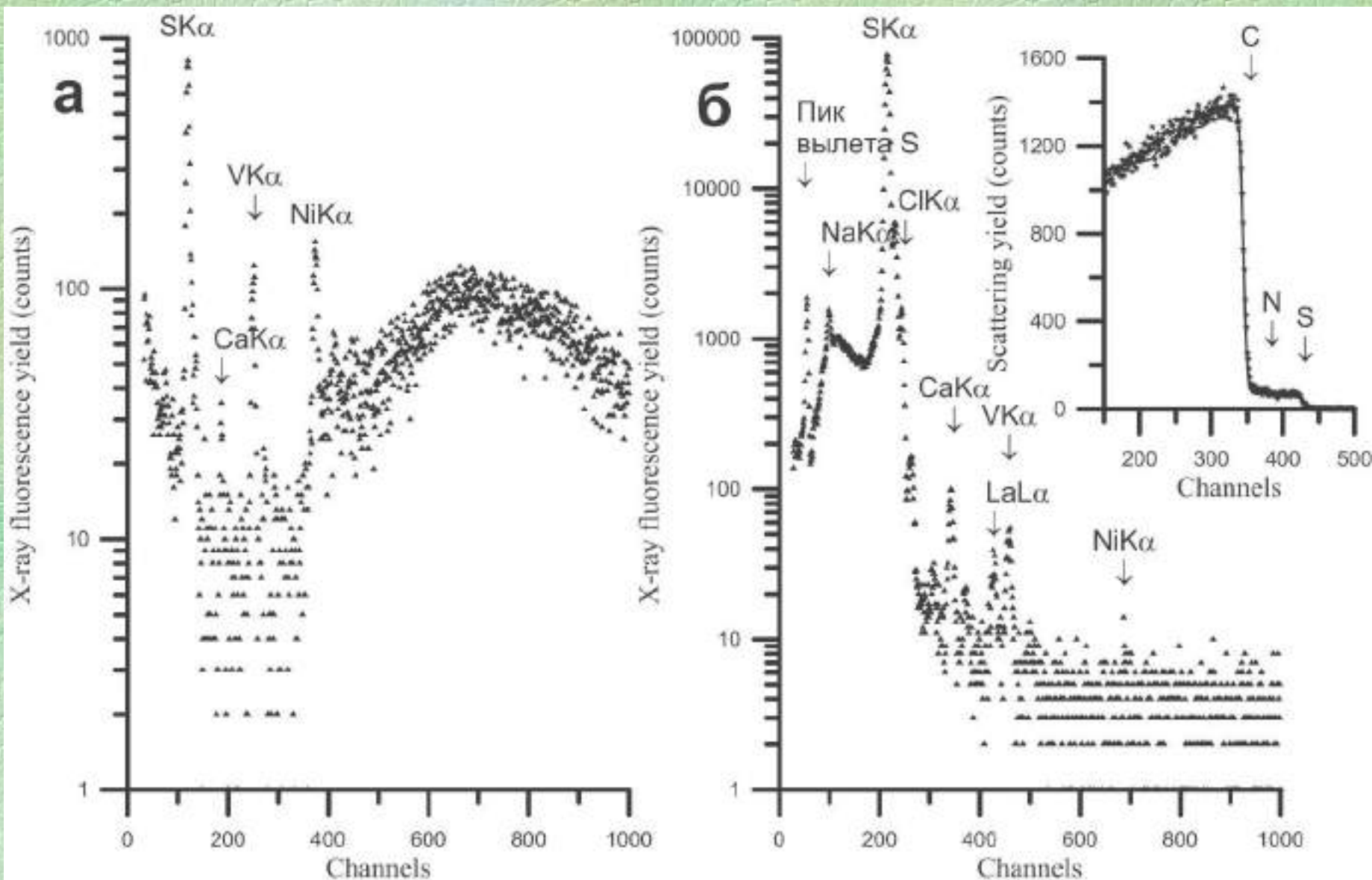
b



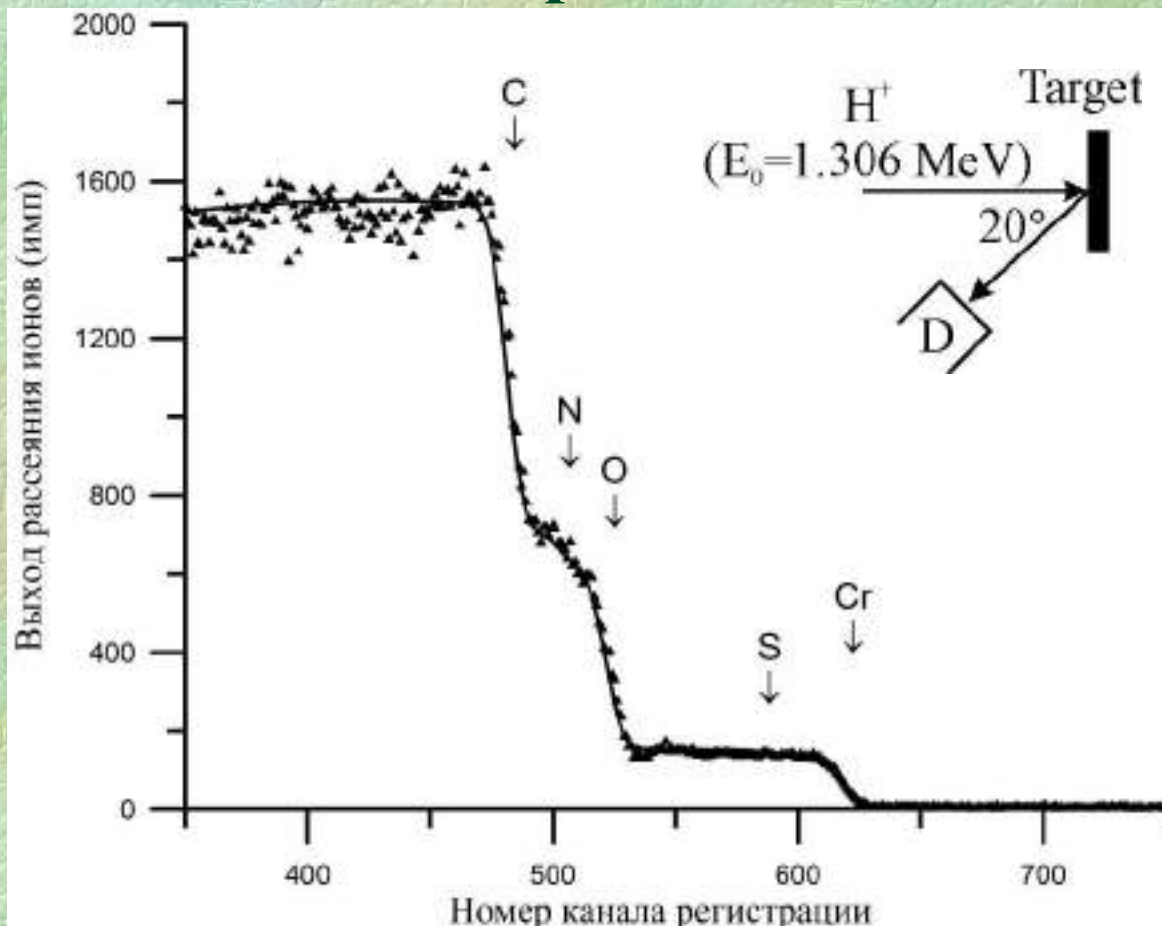
# TXRF (a) and PIXE (b) spectra obtained for the SrTiO<sub>3</sub> single crystal.



# Спектры TXRF (а), PIXE (б) и RBS (в) пленки нефти, осажденной на Ве подложку

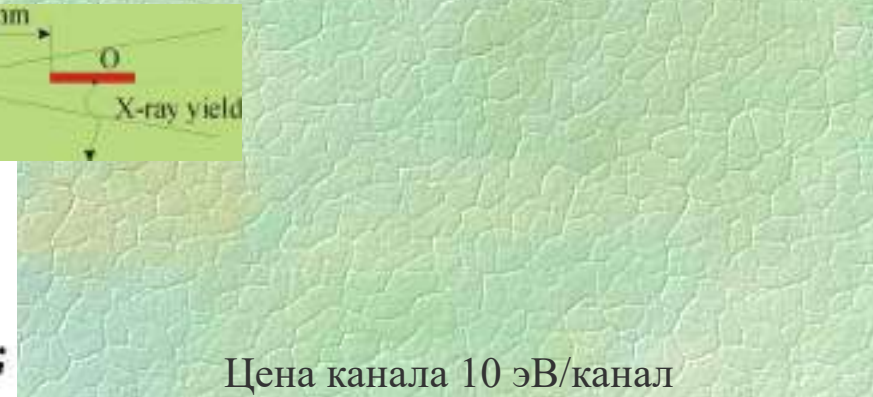
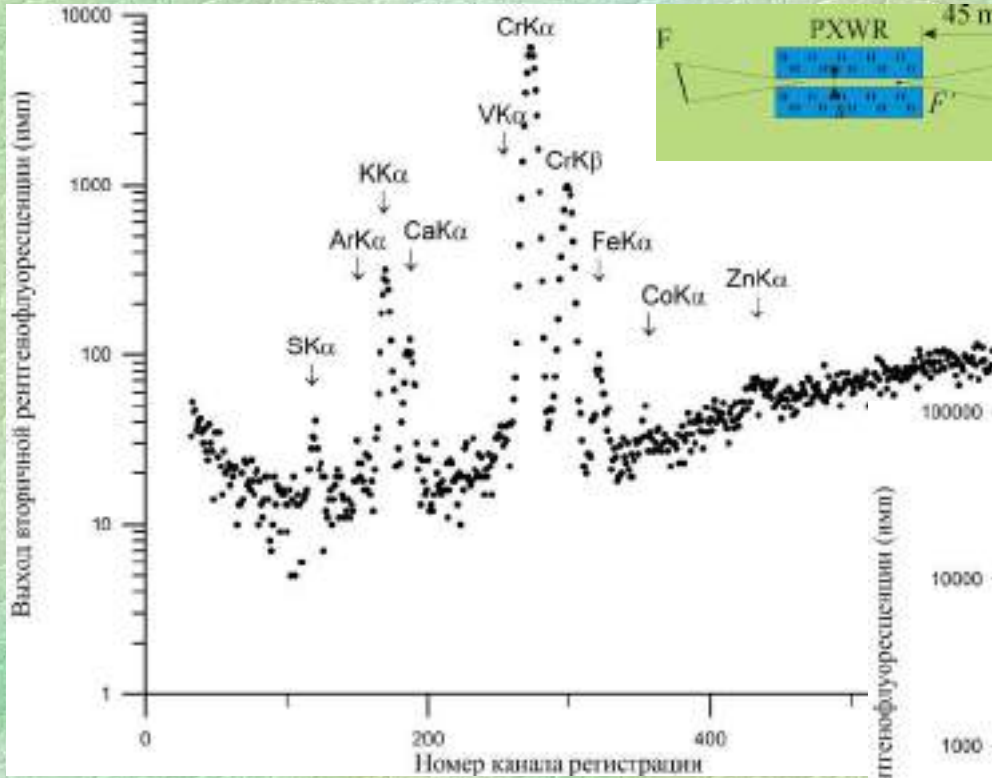


# Экспериментальный и теоретический спектры РОР ионов $H^+$ ( $E_0=1.306$ МэВ) для образца кожевенного материала



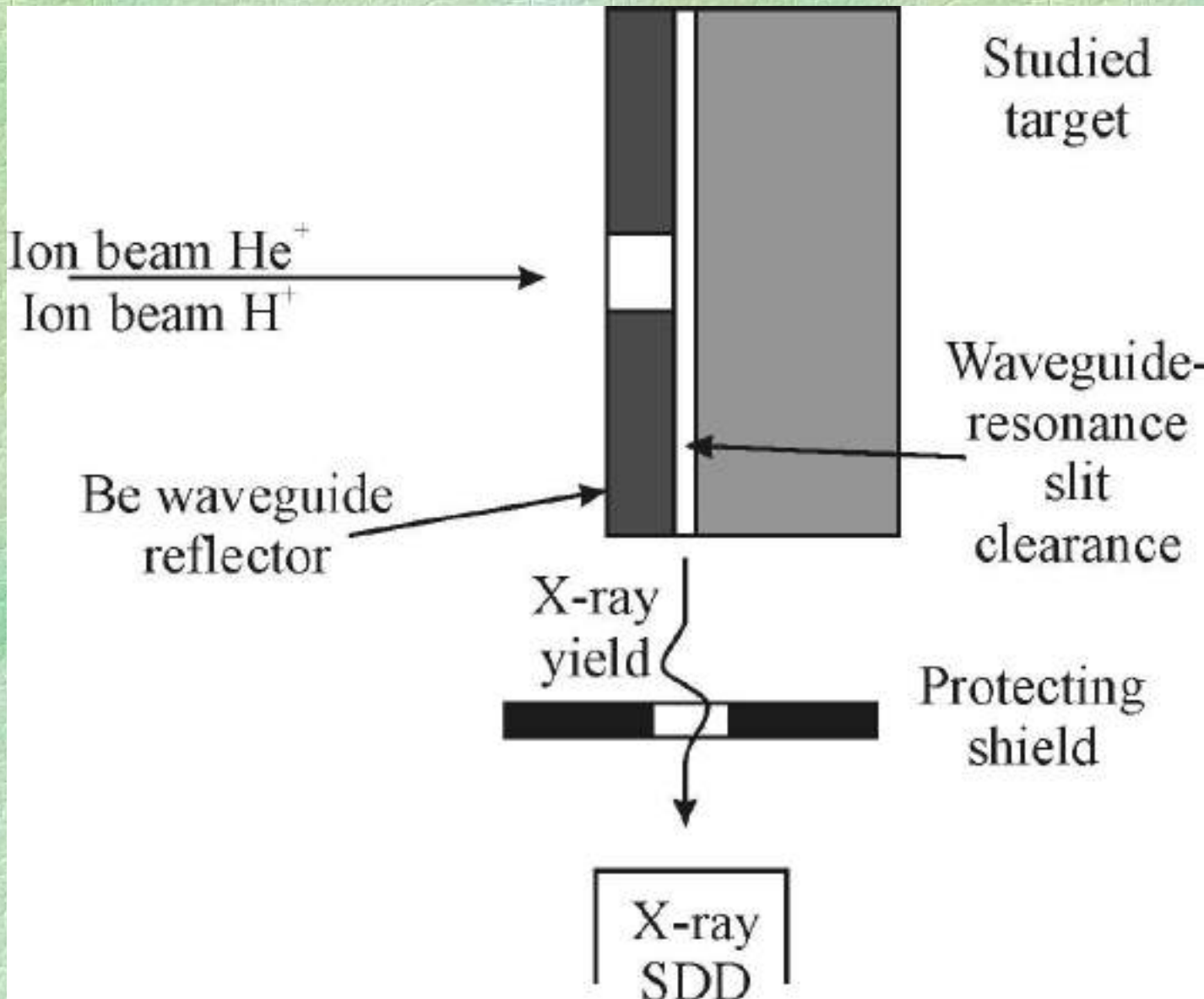
Стрелками указаны энергии, соответствующие рассеянию ионов водорода на ядрах атомов Cr, S, O, N и C, находящихся на поверхности образца кожи. Цена канала 1.9 кэВ/канал.

# Спектры рентгеновской флуоресценции образца кожевенного материала, полученные в условиях РФА ПВО при возбуждении потоком $\text{MoK}\alpha$ (а) и пучком ионов $\text{H}^+$ ( $E_0=1.3 \text{ МэВ}$ ) (б)



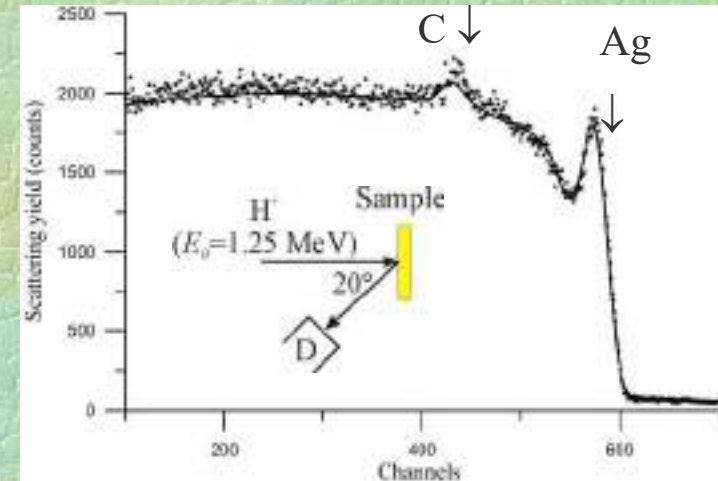
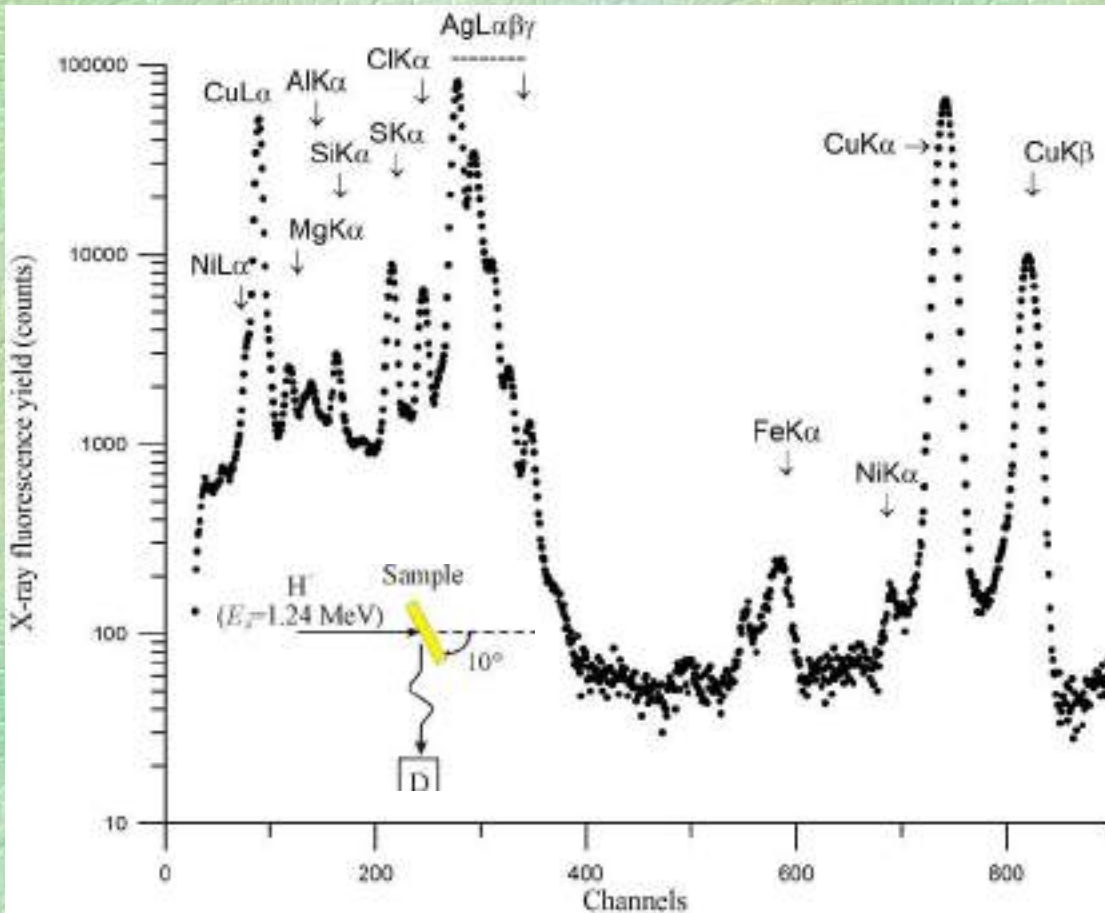
Сравнение спектров показывает, что РИХЕ измерения более чувствительны, чем РФА ПВО при  $\text{Mo}$  возбуждении

# Scheme of TXRF measurements at ion beam excitation in conditions of the waveguide-resonator application (TXRF-PE)



The scheme realizes X-ray fluorescence yield by waveguide-resonator formed by Be polished reflector and surface of studied target. The waveguide-resonance slit clearance transports fluorescence radiation undergoing the total external reflection on Be reflector. The transportation takes place without attenuation. In result, we have the radiation yield with great intensity owing to radiation concentration by waveguide-resonator and element characterization of surface layer with thickness  $t=5$  nm.

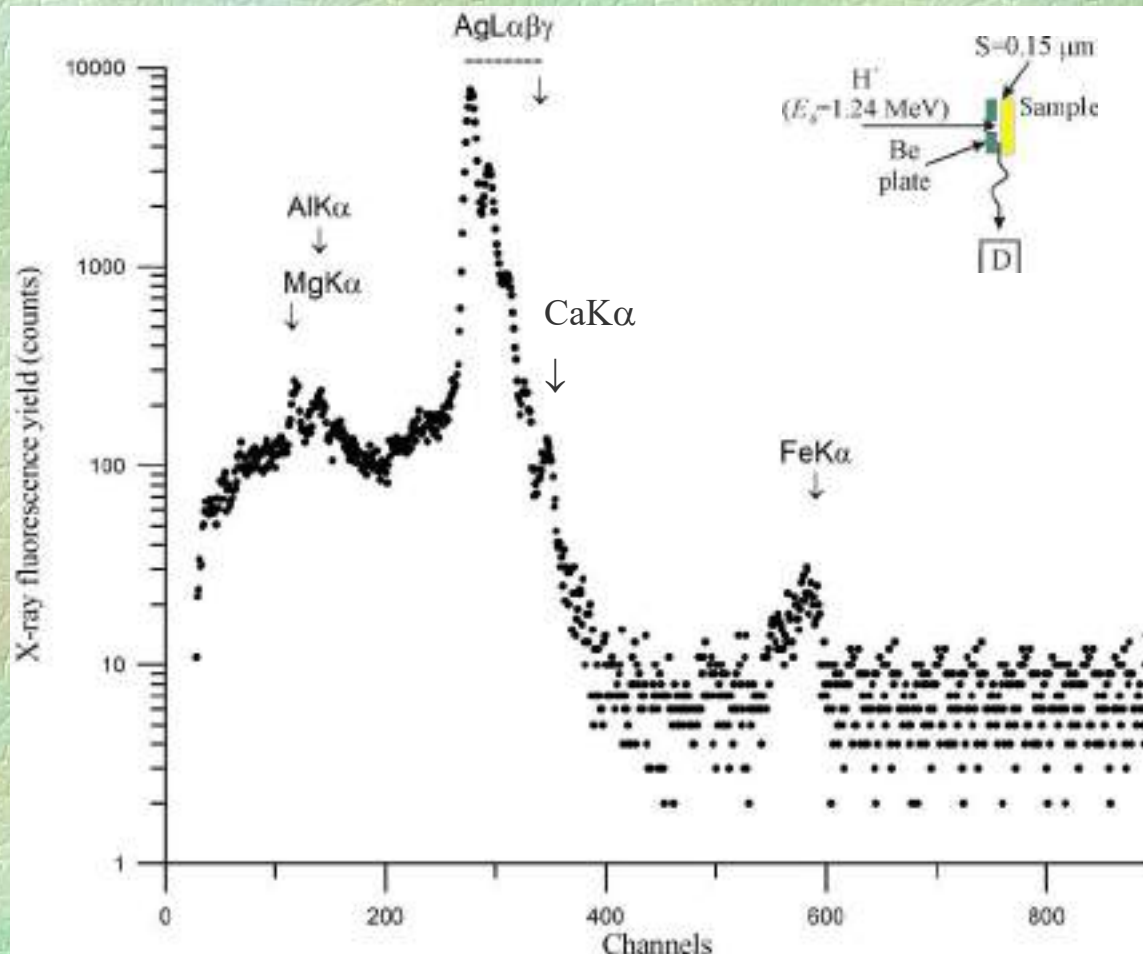
# PIXE spectrum at ion beam excitation and RBS spectrum of old Soviet coin “half copeek” of 1925 year fabrication



Ag is the main alloying addition for host (Cu) material and demonstrate some its excess in the surface in comparison with concentration in the coin volume. Energy step 1.9 keV/channel

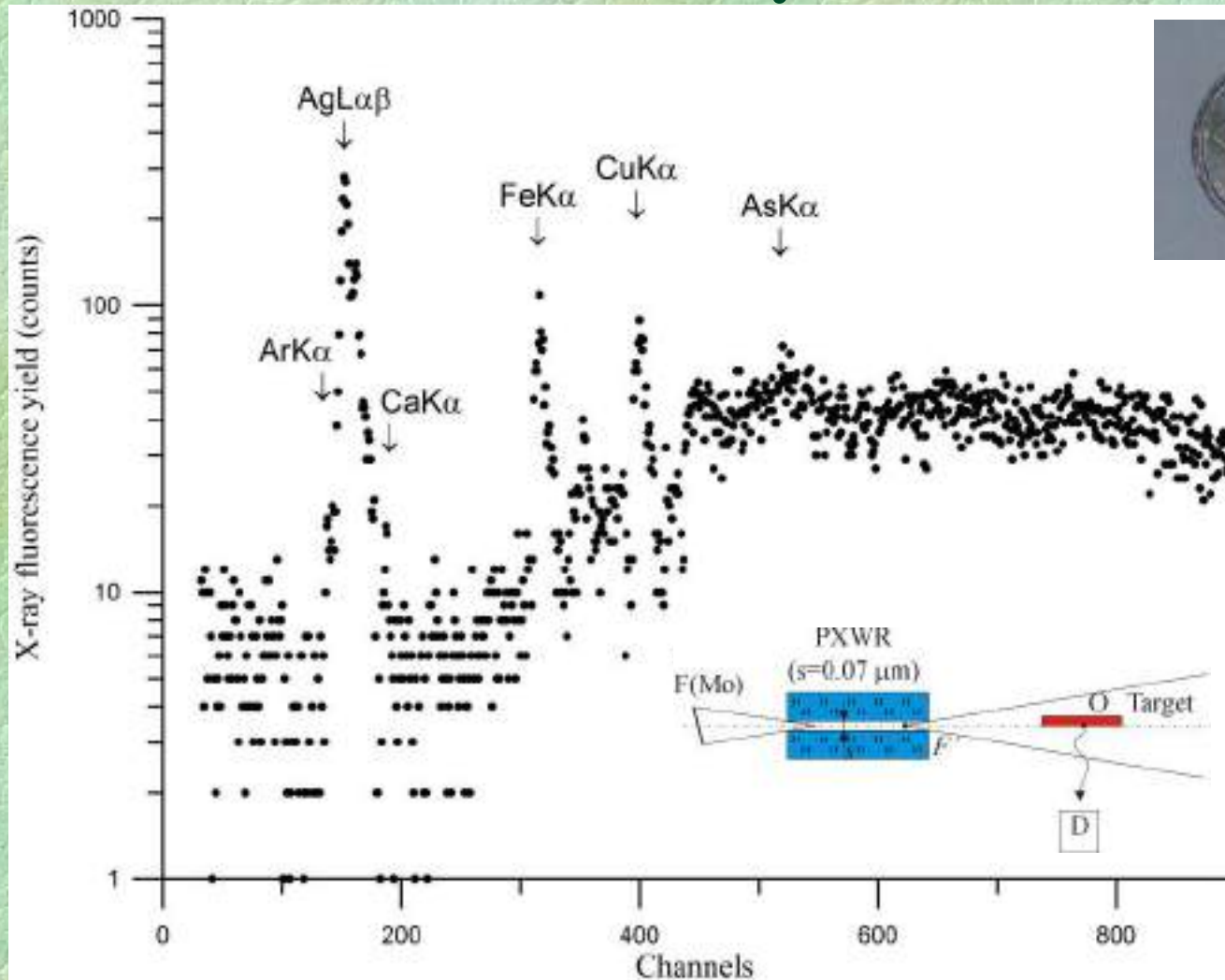
X-ray fluorescence spectrum is the illustration of XRF yield registration at ion beam excitation. Energy step 10.5 eV/channel.

# TXRF-PE spectrum at ion beam excitation and RBS spectrum of old Soviet coin “half copeek” of 1925 year fabrication



X-ray fluorescence spectrum is the illustration of TXRF yield registration at ion beam excitation. Spectrum characterizes element content in surface layer of the coin one. Energy step 10.5 eV/channel.

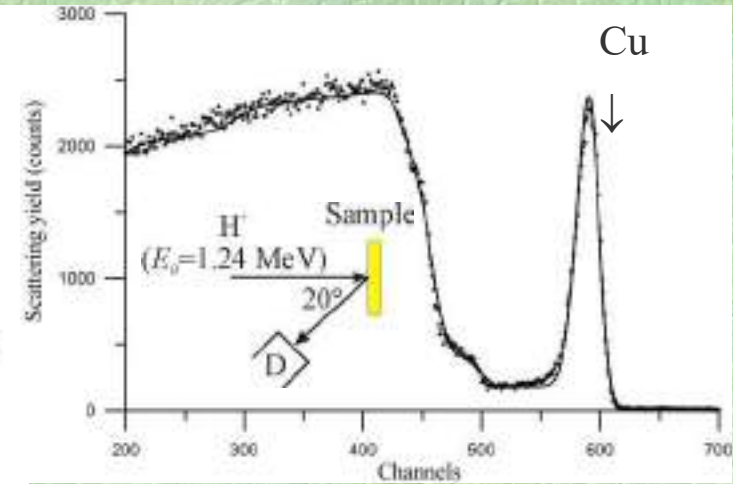
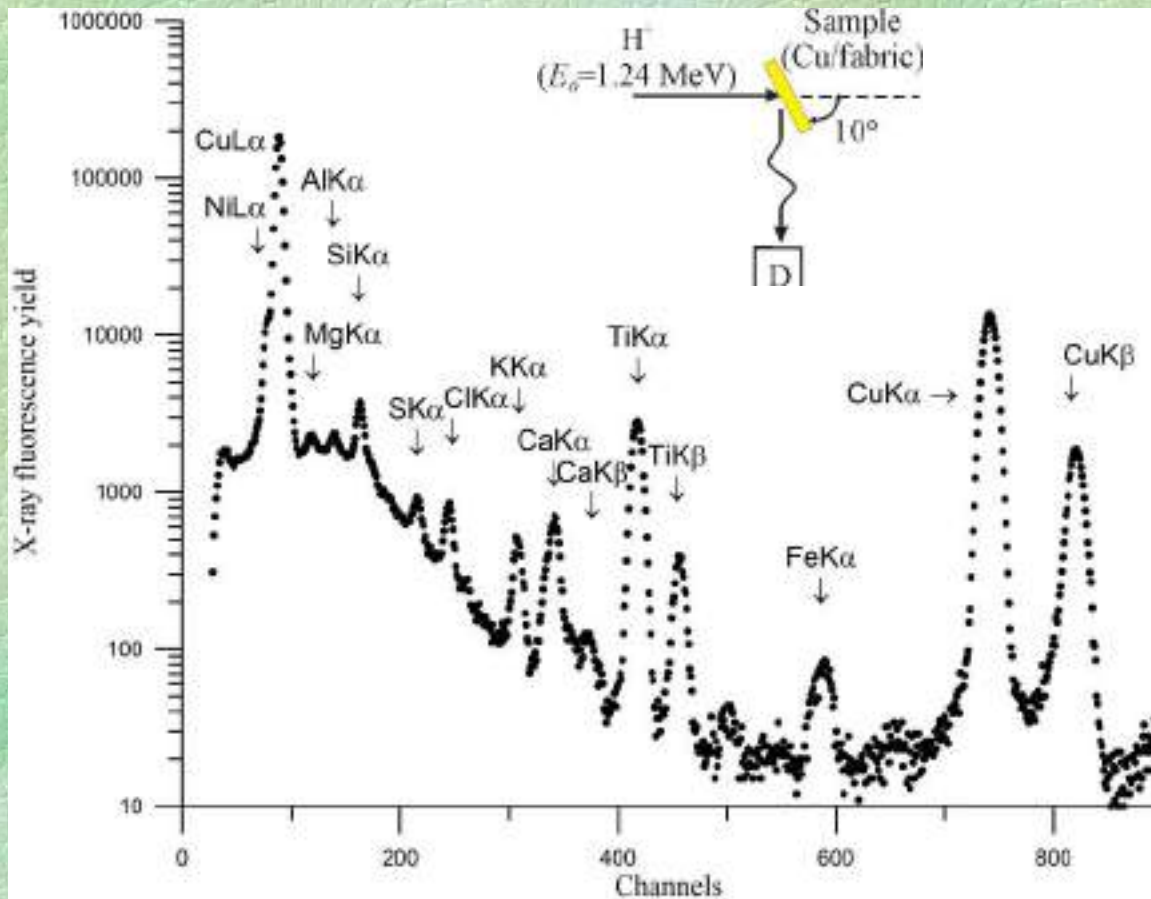
# TXRF spectrum of Soviet coin “half copeek” fabricated at 1925 year



Spectrum shows silver as the main element of the coin surface. Energy step 20 eV/channel.



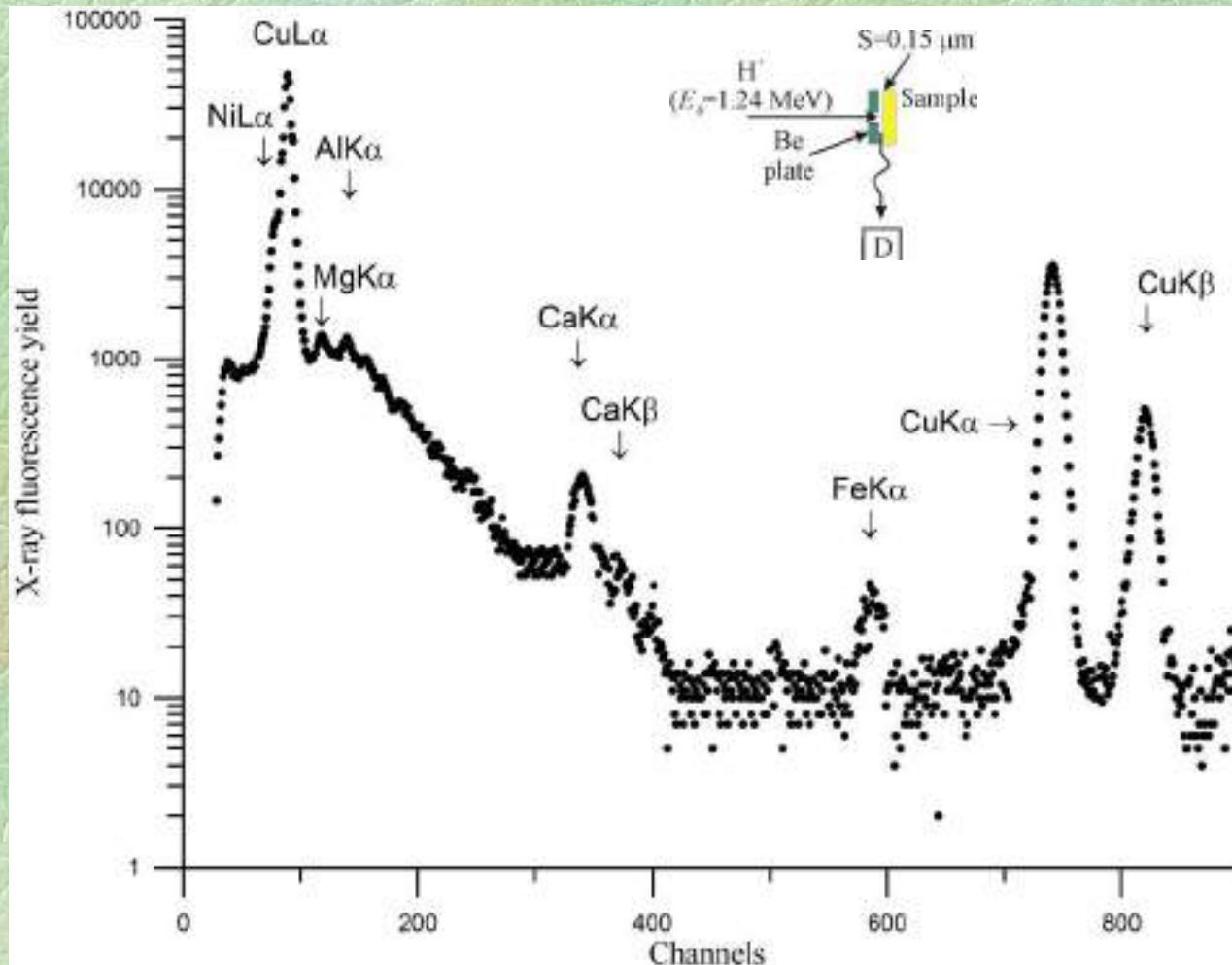
# PIXE spectrum at ion beam excitation and RBS spectrum of the fabric covered by Cu coating.



RBS spectrum show intensive peak connected with Cu-film ( $t=52$  nm). The fabric contains C and O atoms in response to  $C_{1.5}O_1$ . Hydrogen concentration in fabric structure did not define. Energy step 1.9 keV/channel

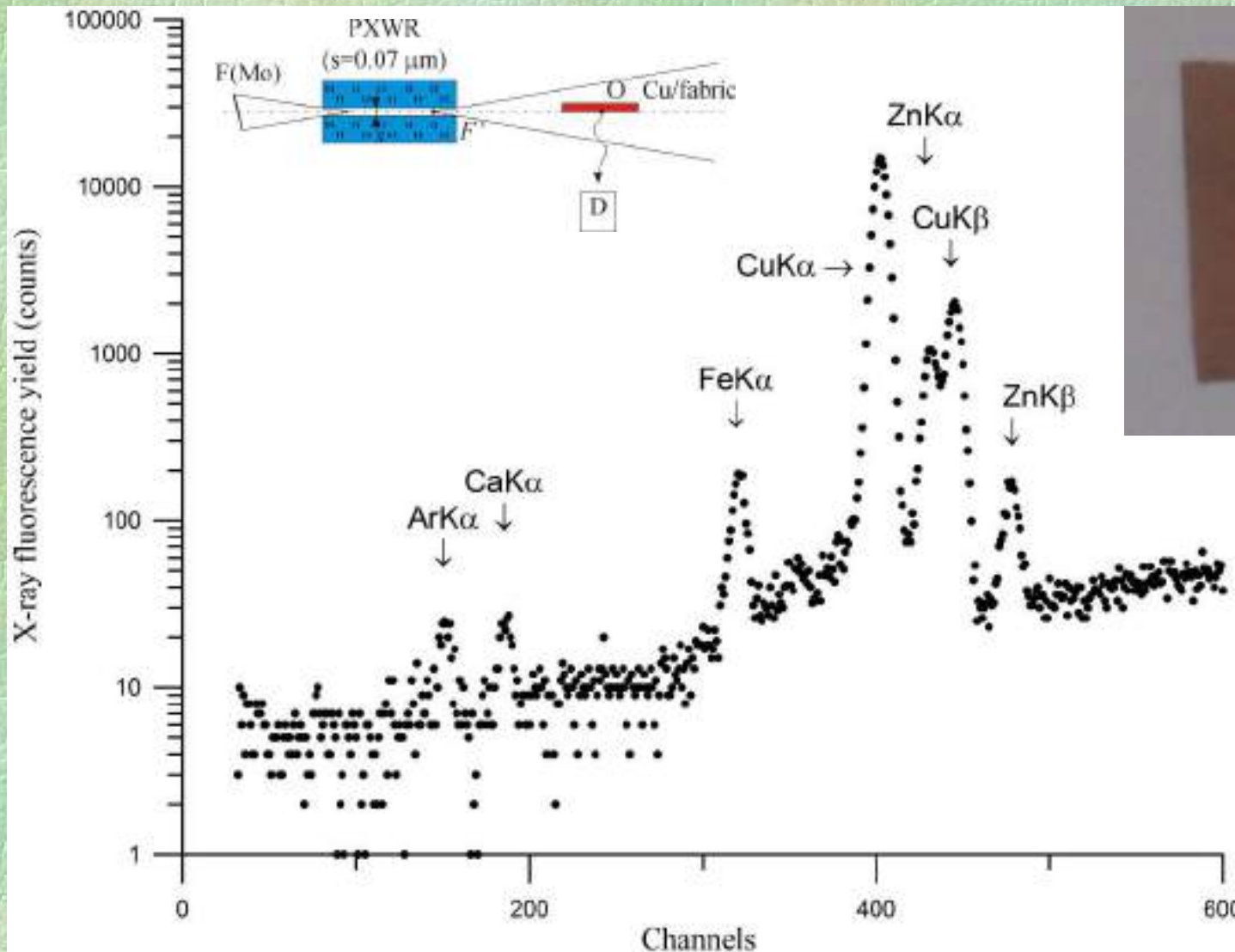
PIXE spectrum allows to registration low concentrations of elements copper coating and fabric body. Energy step 10.5 eV/channel.

# TXRF-PE spectrum at ion beam excitation of the fabric covered by Cu coating



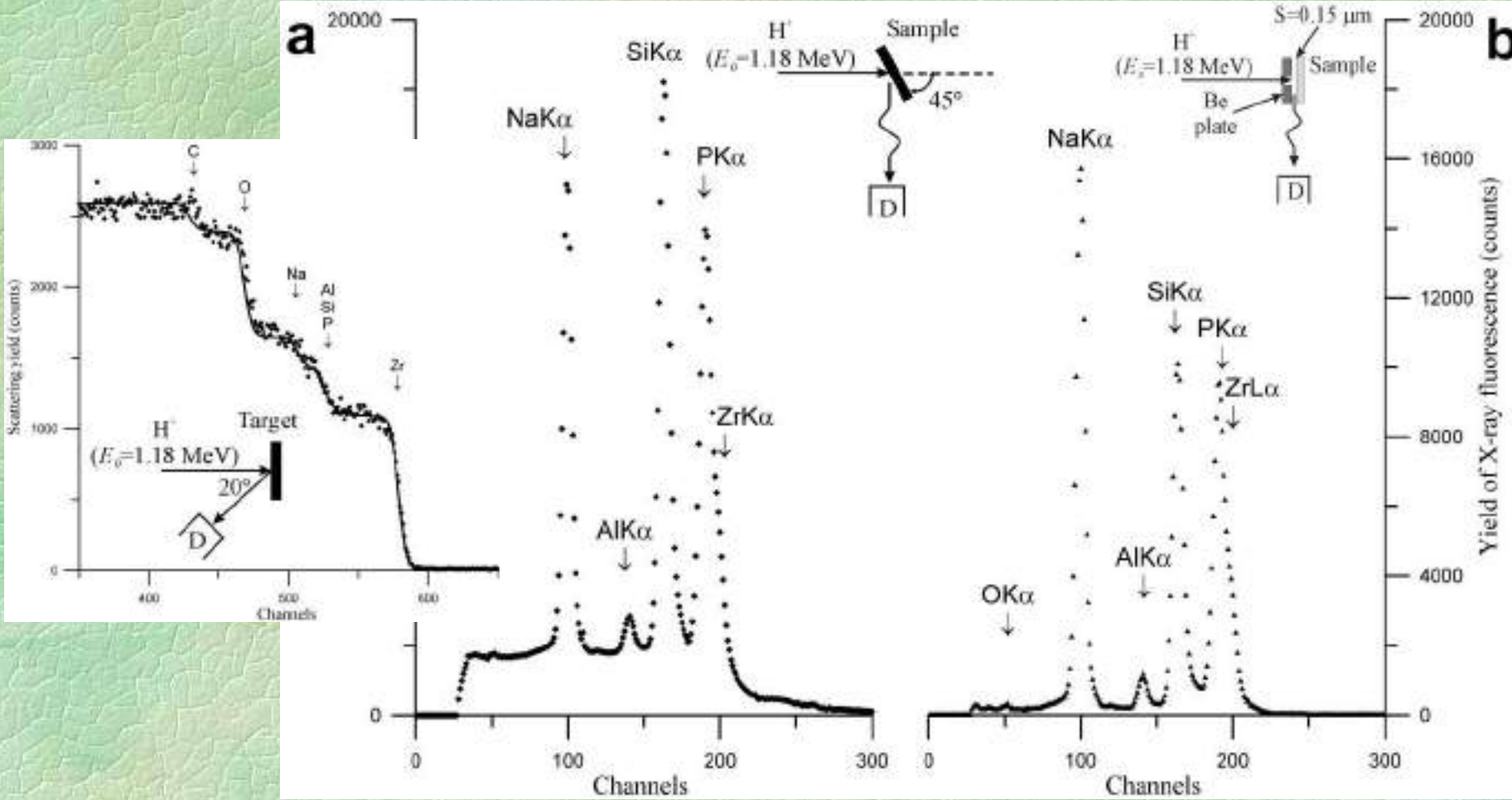
TXRF-PE spectrum allows to registration low concentrations of elements copper coating. Energy step 10.5 eV/channel.

# TXRF spectrum of the fabric coated by Cu film



Spectrum characterizes set pollutions existence in the Cu film. Energy step 20 eV/channel.

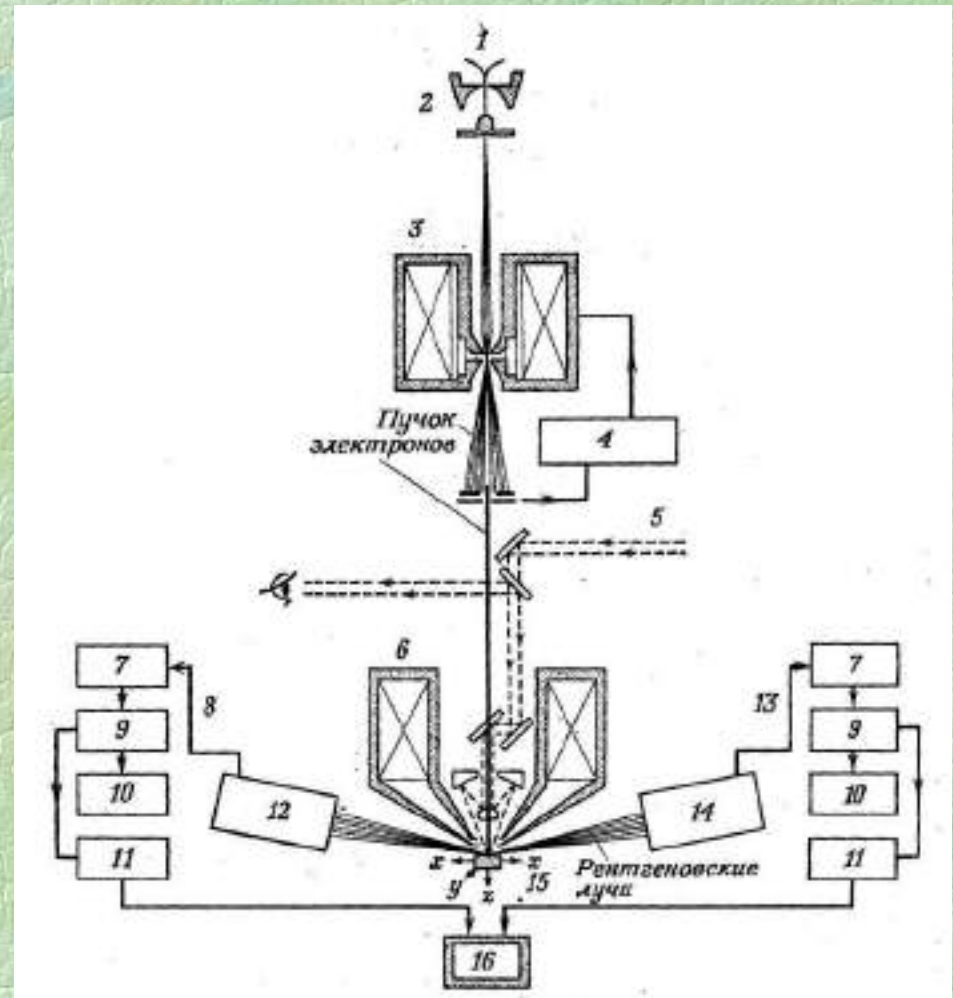
# X-ray fluorescence spectra collected in the conventional (a) and modified (b) geometries for $\text{Na}_3\text{Zr}_{1.3}\text{Si}_{1.9}\text{Al}_{0.1}\text{P}_{1.0}\text{O}_{12}\text{C}_2$ natural target



Energy step for X-ray fluorescence spectra 10.6 eV/channel, for RBS spectrum 1.9 keV/channel. Modified geometry of the PIXE shows the background deposit decreasing.

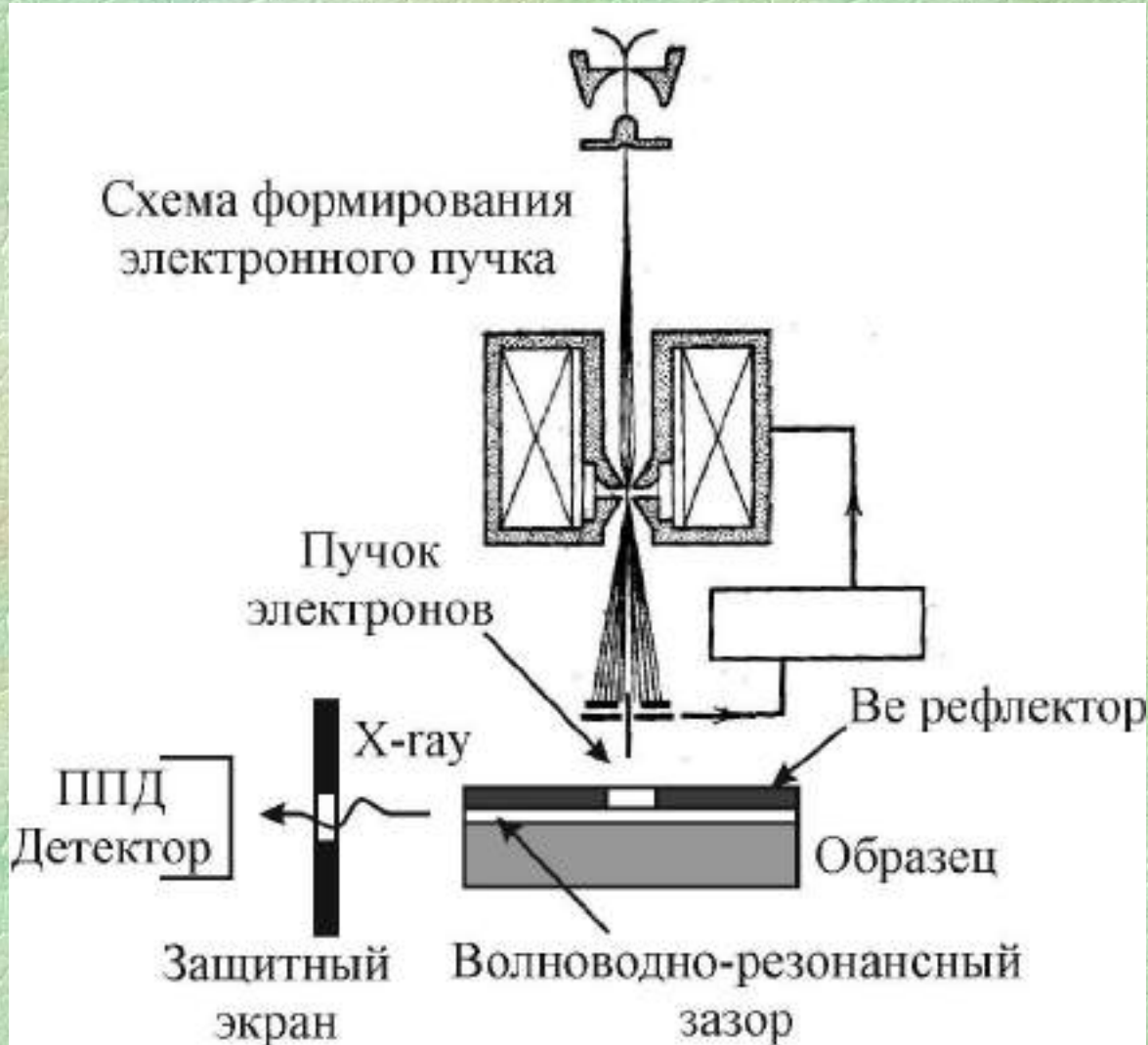
# Схема рентгенофлуоресцентного анализа материалов в условиях электронного микронзондового возбуждения

1. Нить накала;
2. электронная пушка (8-34 кВ);
3. конденсаторная линза;
4. стабилизация тока зонда;
5. освещение образца;
6. объектная линза;
7. усилитель;
8. выход пропорционального счетчика;
9. амплитудный анализатор импульсов;
10. пересчетная схема;
11. интенсиметр;
12. спектрометр для мягкого рентгеновского излучения;
13. выход сцинтиляционного счетчика;
14. спектрометр для жесткого рентгеновского излучения;
15. образец;
16. 2х перьевой самописец.



Микронзонд Самеса

# Схема электронного микронзондирования концентрации элементов в тонком поверхностном слое с применением волноводно-резонансной технологии



Thank You for attention!

