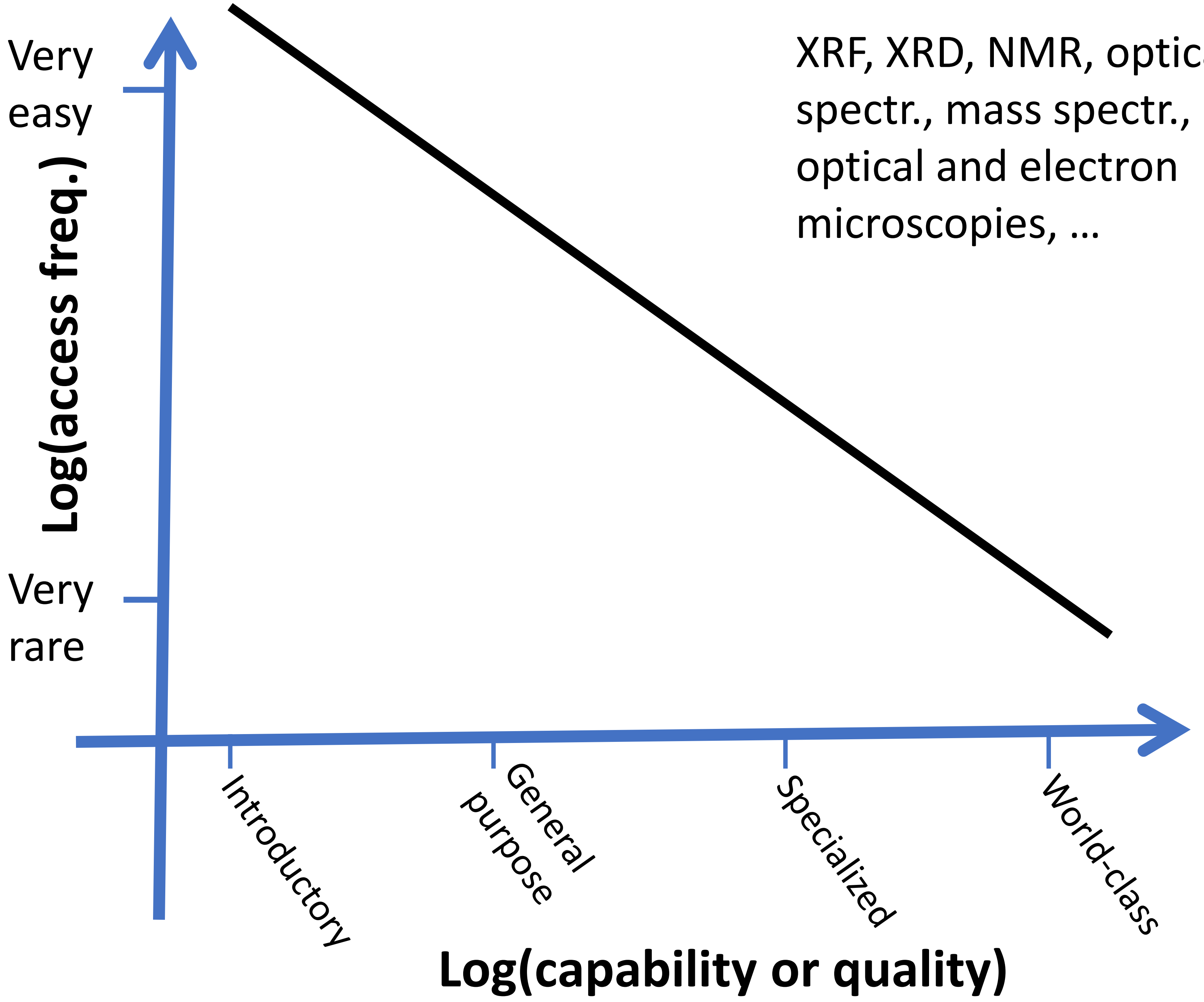
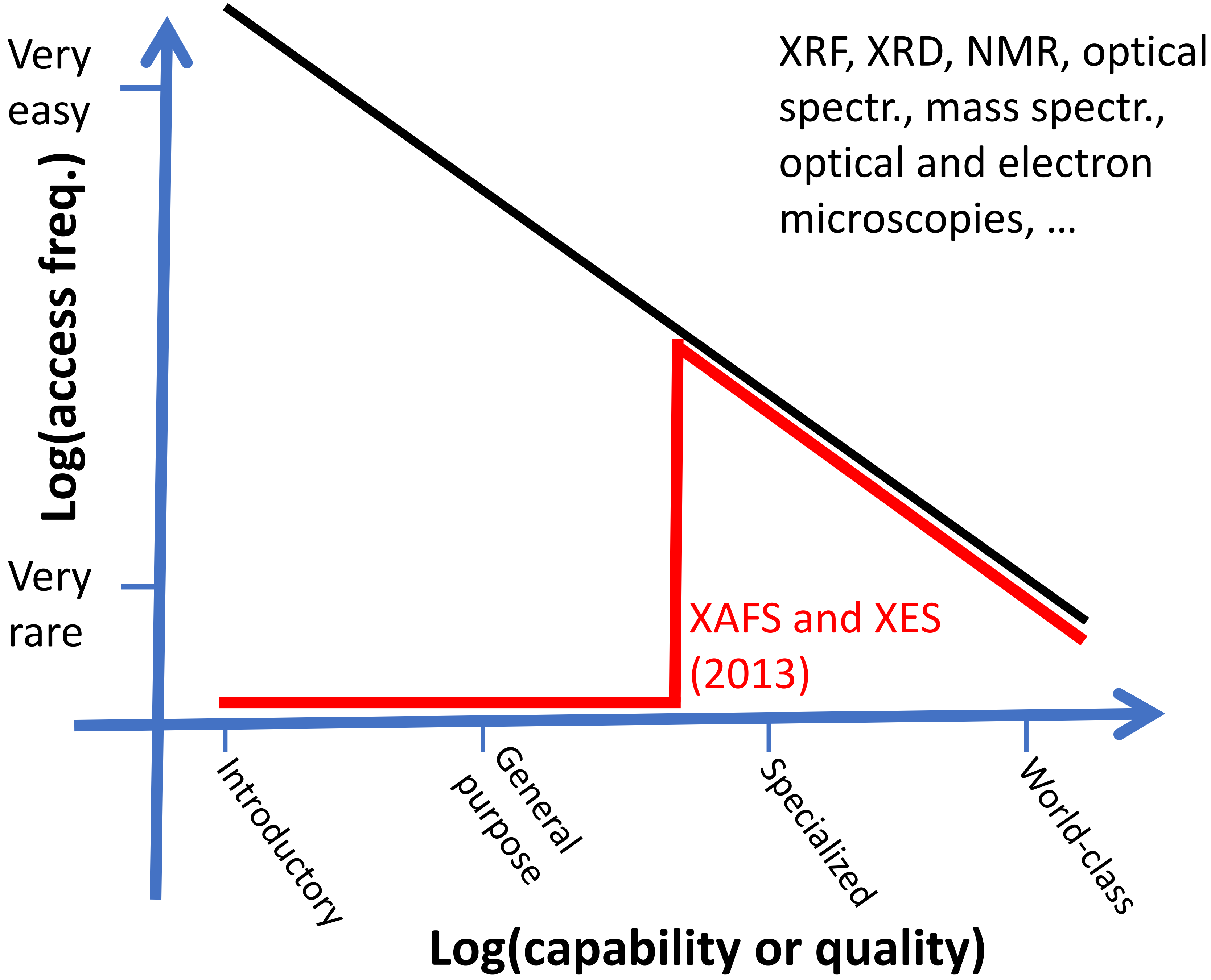
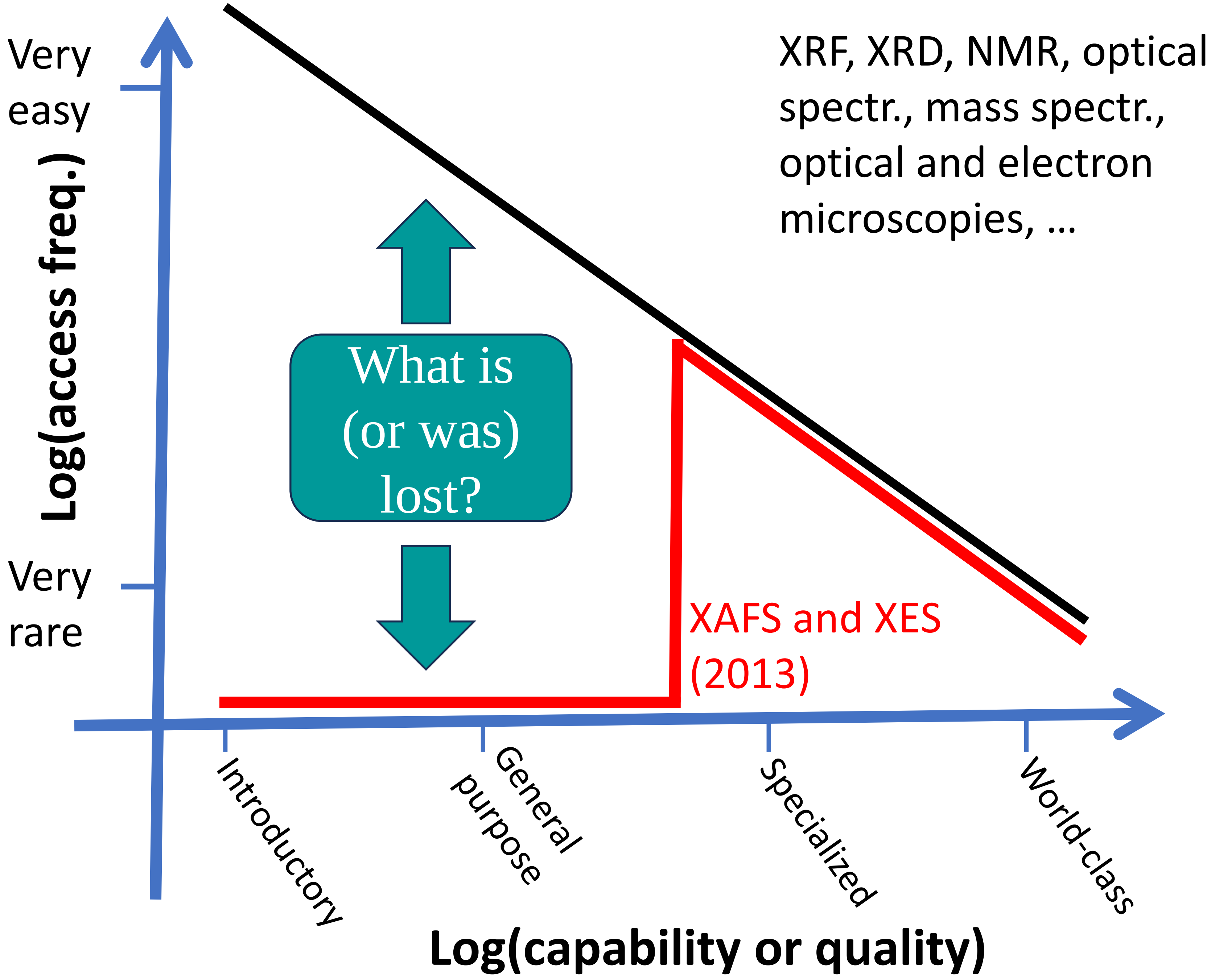

Very easy
Log(access freq.)
Very rare
XRF, XRD, NMR, optical spectr., mass spectr., optical and electron microscopies, …
Introductory
General purpose
Specialized
World-class
Log(capability or quality)


XRF, XRD, NMR, optical spectr., mass spectr., optical and electron microscopies, …
Very easy
Very rare
Log(access freq.)
XAFS and XES (2013)
Introductory
General purpose
Specialized
World-class
Log(capability or quality)


Very easy
Very rare
Log(access freq.)
XRF, XRD, NMR, optical spectr., mass spectr., optical and electron microscopies, …
What is (or was) lost?
XAFS and XES (2013)
Introductory
General purpose
Specialized
World-class
Log(capability or quality)

Prep for synchrotron
Routine analytical chemistry:
Inorg. Materials, Metalorganics
(Academic) Research projects
(Academic) Research programs
Education

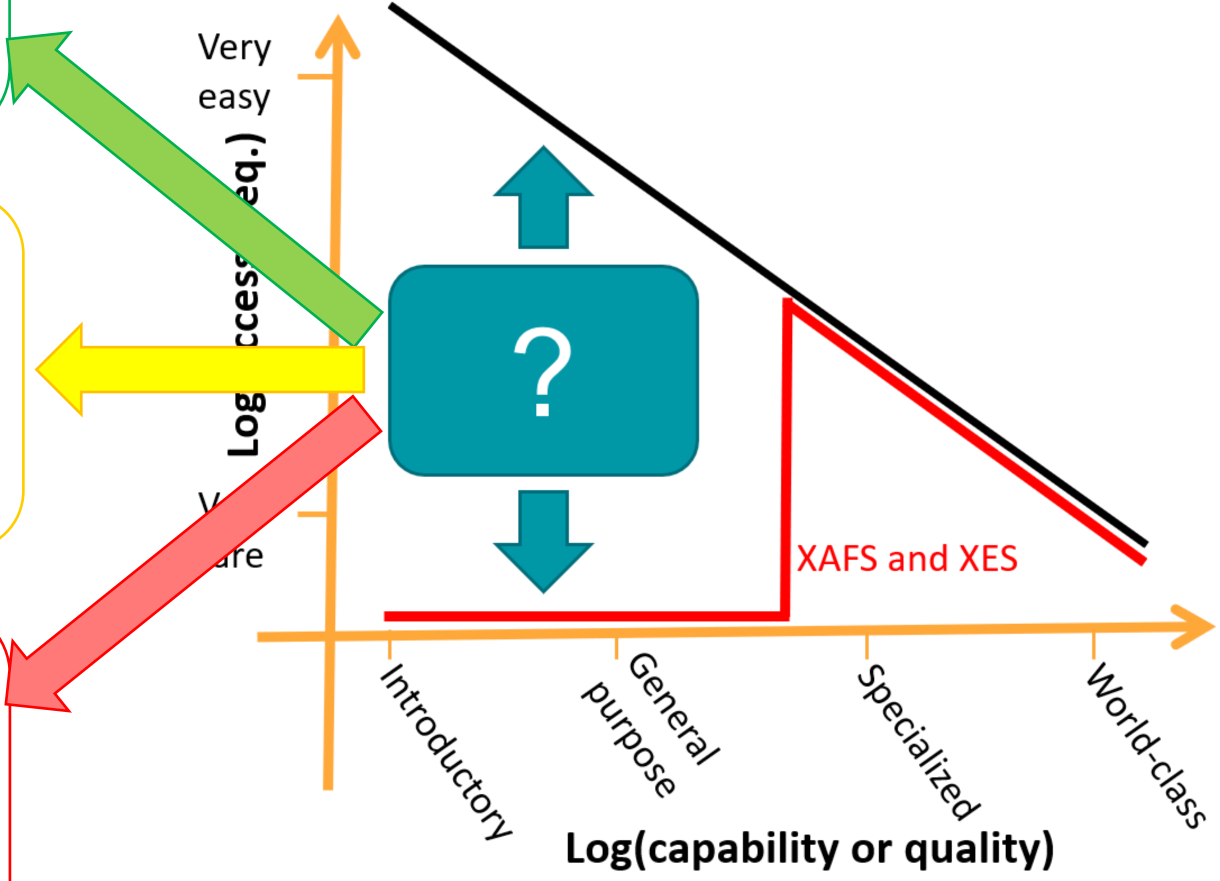
Proven

Industrial R&D:
Mining, battery components, catalysts, …
Routine analytical chemistry:
Environment

Likely

Routine analytical chemistry:
Regulatory compliance:
Cr(VI), …
Industrial quality control:
Mining, battery, catalyst, pharma, nanophases,…

Possible



Prep for synchrotron
Routine analytical chemistry:
    Inorg. Materials, Metalorganics
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    Environment

Likely

Routine analytical chemistry:
    Regulatory compliance:
        Cr(VI), …
    Industrial quality control:
        Mining, battery, catalyst,
        pharma, nanophases,…

Possible

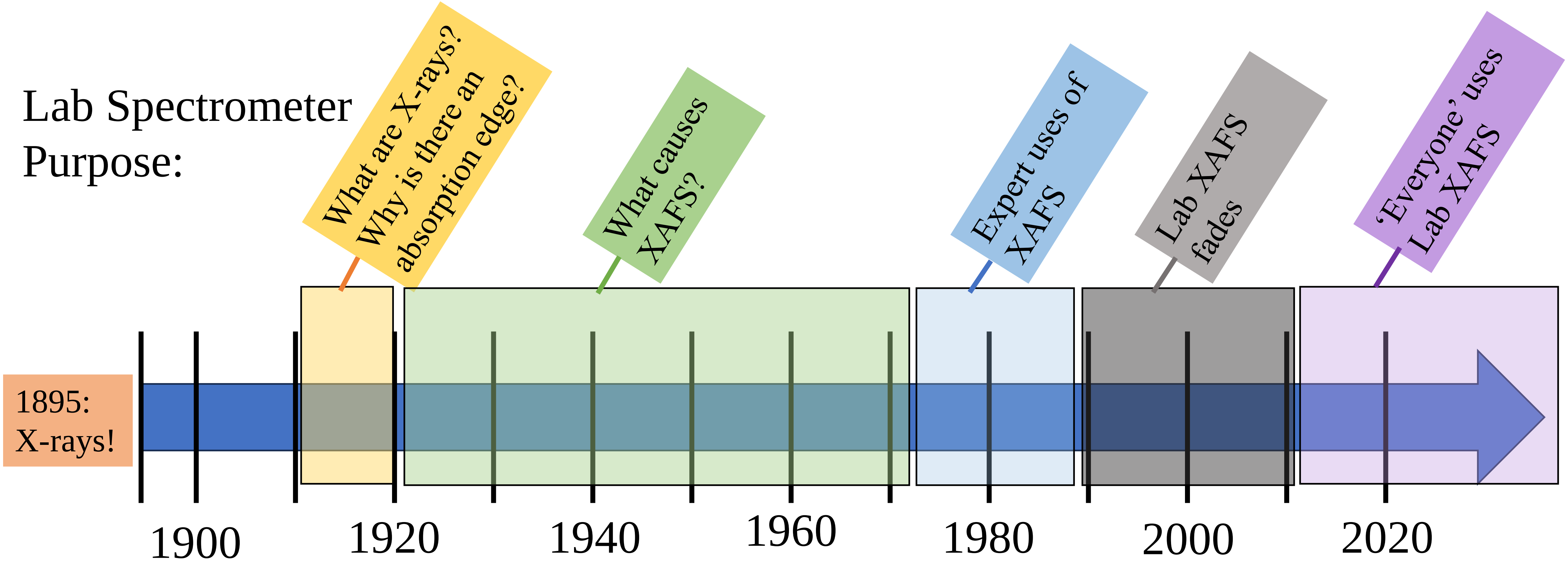
There are modern roles for lab XAFS, some complement synchrotron usage and some are completely independent.

But, how did we get here?

Lab Based XAFS:
A History of Spectrometers and Spectroscopy

Prof. Jerry Seidler (seidler@uw.edu)
University of Washington, Seattle WA
Co-founder of easy*XAFS* LLC

OUTLINE


Lab Spectrometer Purpose:
What are X-rays? Why is there an absorption edge?
What causes XAFS?
Expert uses of XAFS
Lab XAFS fades
'Everyone' uses Lab XAFS
1895: X-rays!
1900
1920
1940
1960
1980
2000
2020

Ann. Phys. Fr. **14** (1989) 377-466 AOUT **1989,** PAGE 377

A History of X-ray absorption fine structure (*)

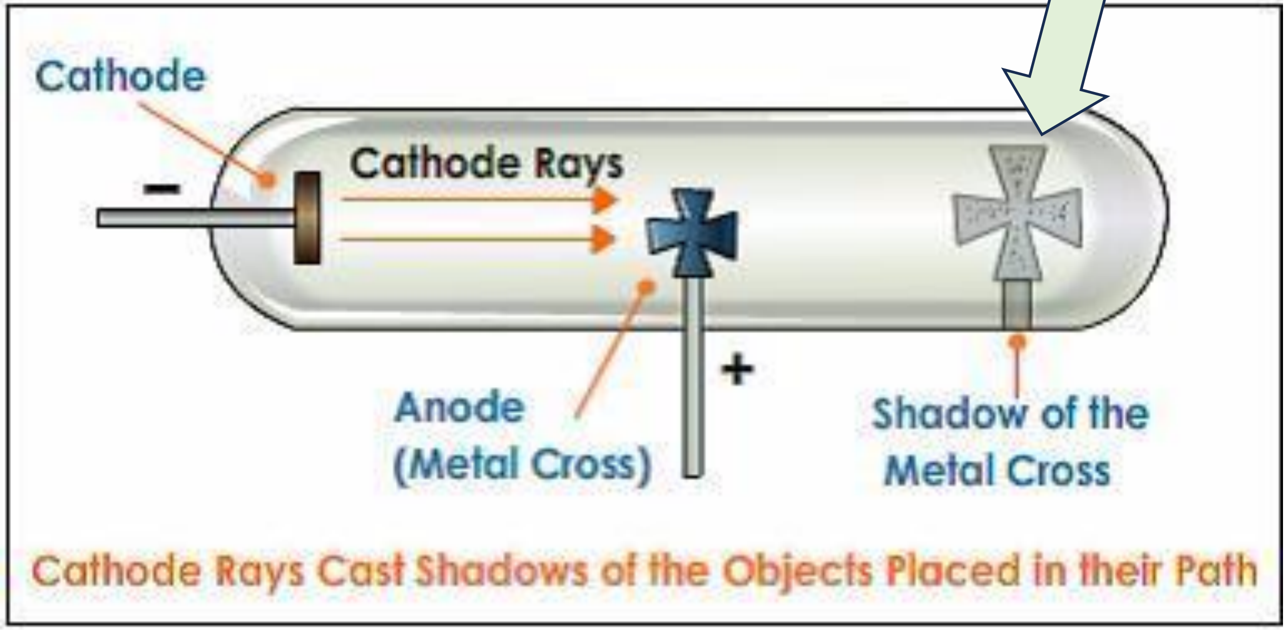
R. Stumm von Bordwehr [Christian Brouder?]

Laboratoire de Physique du Solide, Université de Nancy I, B.P. 239, F-54506 Vandoeuvre- lès-Nancy Cedex, France

Abstract. — This historical account of X-ray absorption fine structure (XAFS) spectroscopy from the origin to 1975 begins with the first observations of X-ray absorption edges and the experimental setups used at the turn of the century. Then, the discovery of XAFS and Kossel's early interpretation are discussed. A close look is taken at the three outstanding papers written by Kronig to explain XAFS in solids and molecules. Petersen's development of XAFS in molecules and Smoluchowski's investigation of XAFS in crystals during the thirties are reviewed. Then, the Japanese and Soviet contributions to X-ray absorption spectroscopy up to the sixties are described. We conclude with the advent of the present understanding of XAFS developed in the early seventies. Although many experiments are presented, we emphasize the conceptual evolution of the interpretation of XAFS, including false steps and overlooked works.

Wilhelm Roentgen
(1845-1923)

$Ba[Pt(CN)_4]$ screen
Inside the tube.
(cathodoluminescent)

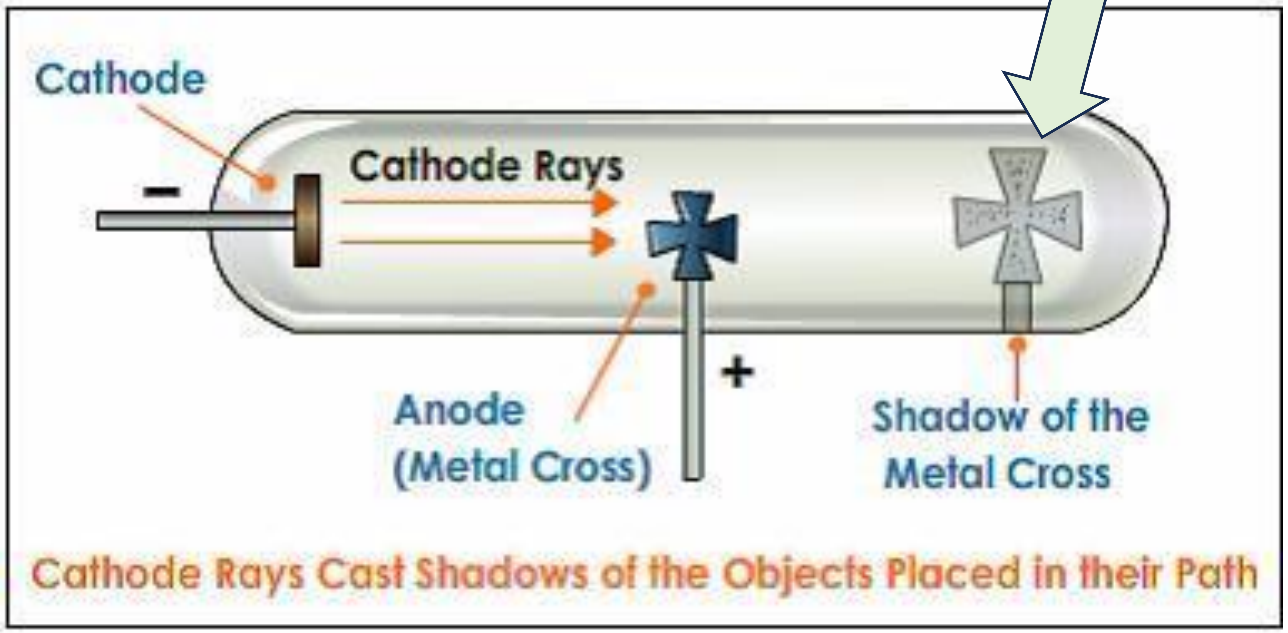


8 November 1895

A spare $Ba[Pt(CN)_4]$ screen outside the tube.

Wilhelm Roentgen
(1845-1923)

$Ba[Pt(CN)_4]$ screen
Inside the tube.
(cathodoluminescent)



8 November 1895

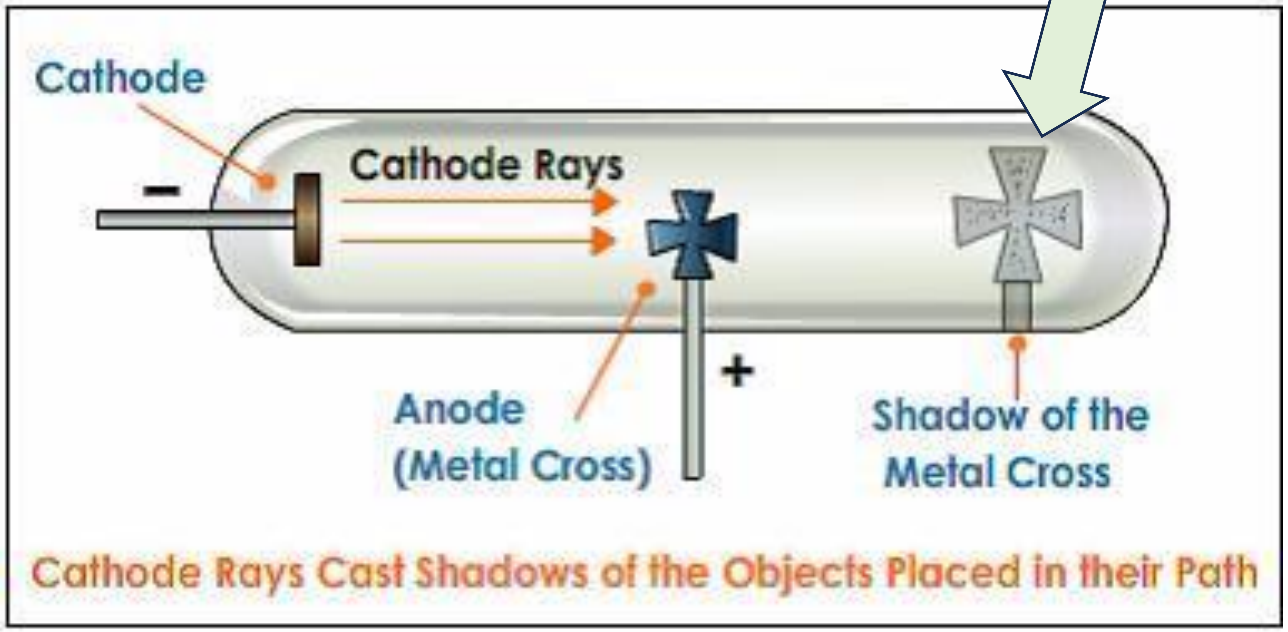
Wilhelm Roentgen
(1845-1923)

A spare $Ba[Pt(CN)_4]$ screen outside the tube.

Roentgen: Cathode rays don't go through air → there must be some other ray being generated.

"X-ray" where "X" is the unknown variable from mathematics.

$Ba[Pt(CN)_4]$ screen Inside the tube. (cathodoluminescent)



8 November 1895

X = ‘unknown variable’

Wilhelm Roentgen
(1845-1923)

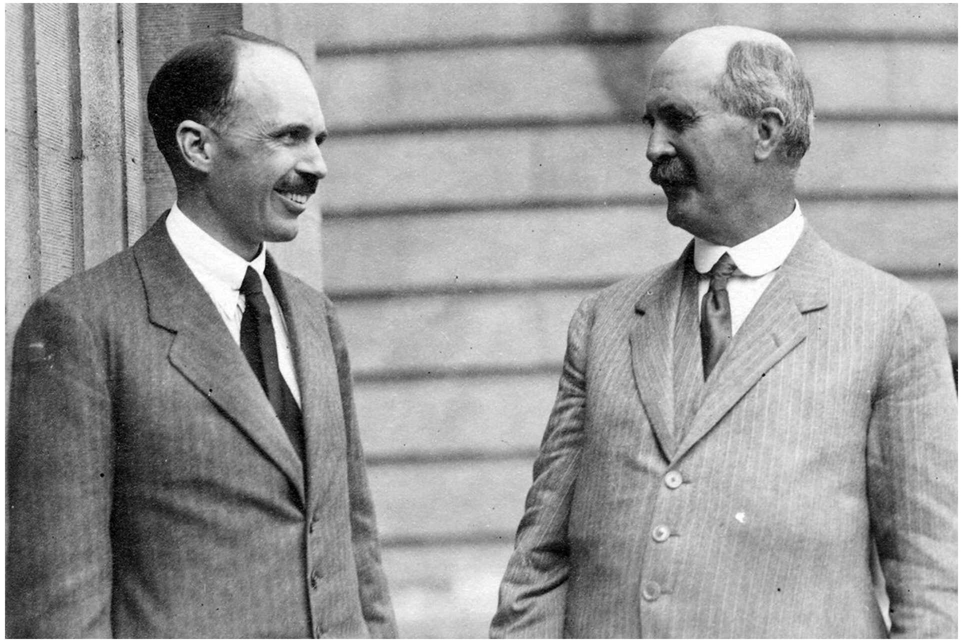
The medical applications of X-rays were apparent, and research started to learn **“what is X”.**

Nobel Prize in Physics 1901

X = ‘unknown variable’

If X-rays are waves. Then they will diffract.

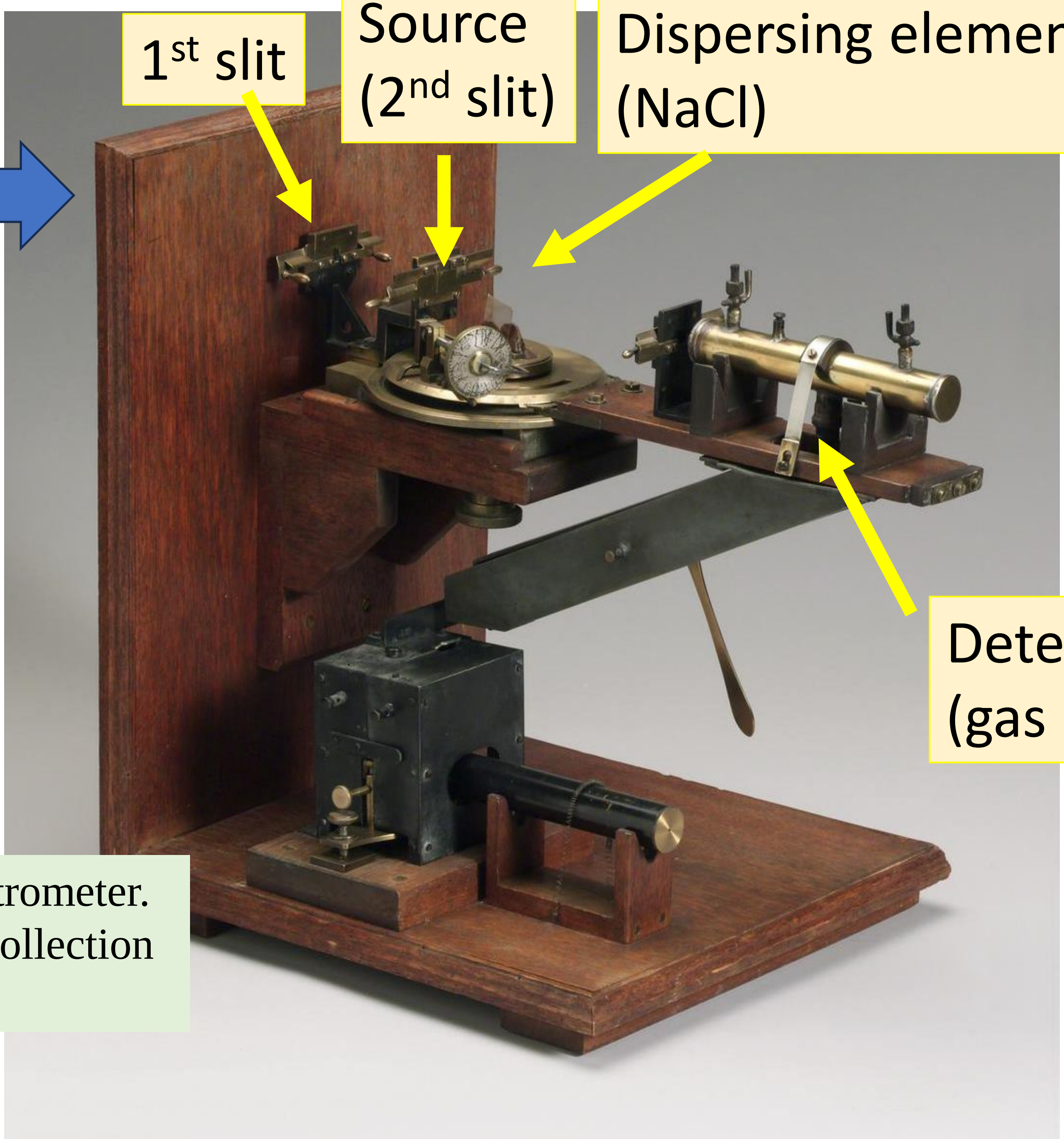
Max von Laue (1879-1960)
1914 Nobel Prize



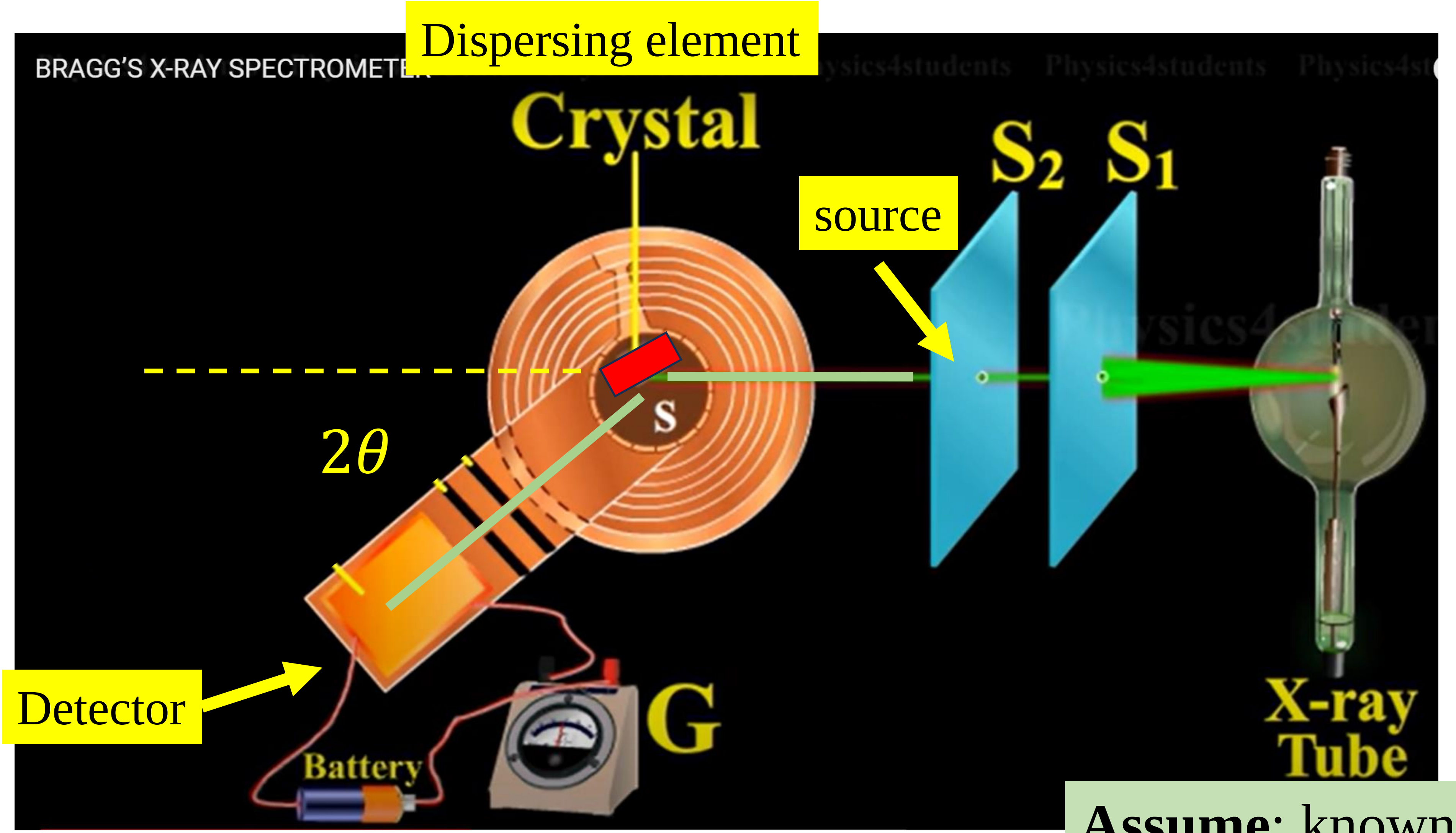
William Henry Bragg (1862-1942)
William Laurence Bragg (1890-1971)

1915 Nobel Prize

X-rays are waves with wavelength comparable to the spacing between atoms in solids.

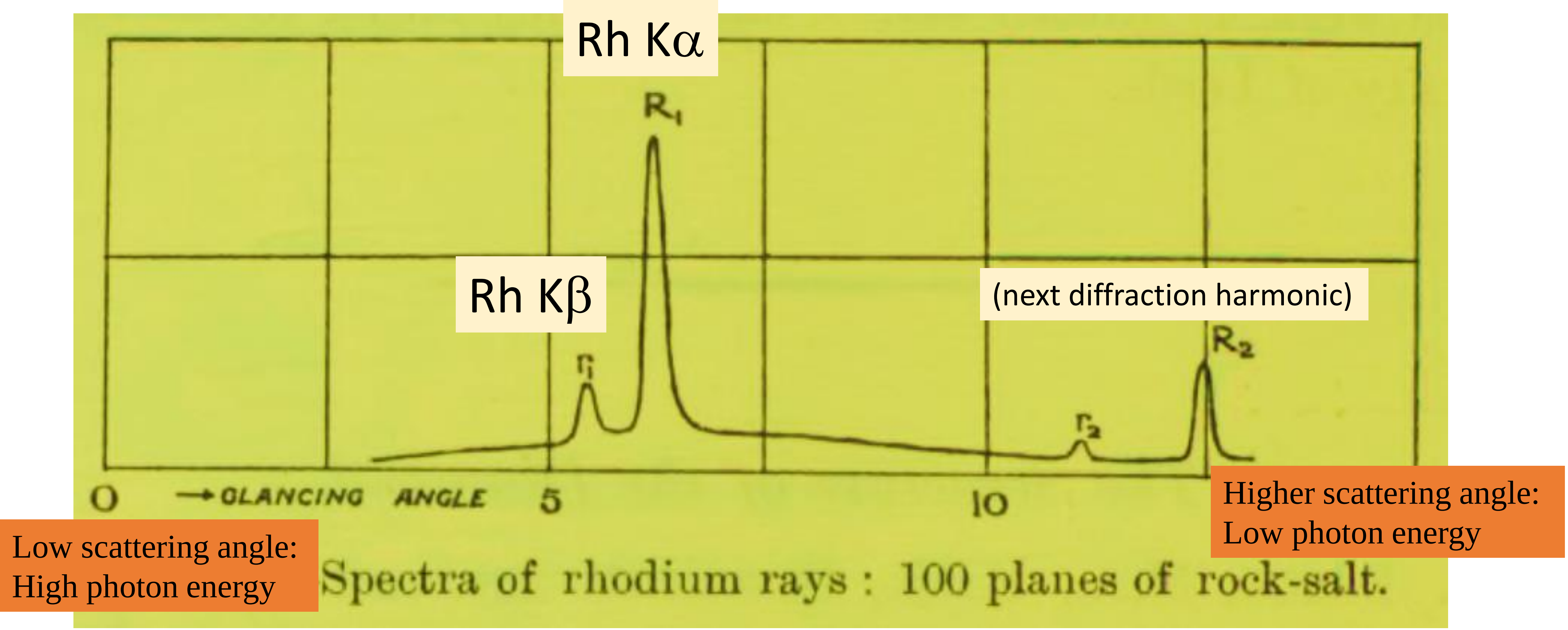


Original Bragg x-ray spectrometer. Science Museum Group Collection (UK)



Assume: known *d*-spacing, then this is a spectrometer to study the spectrum of the tube.

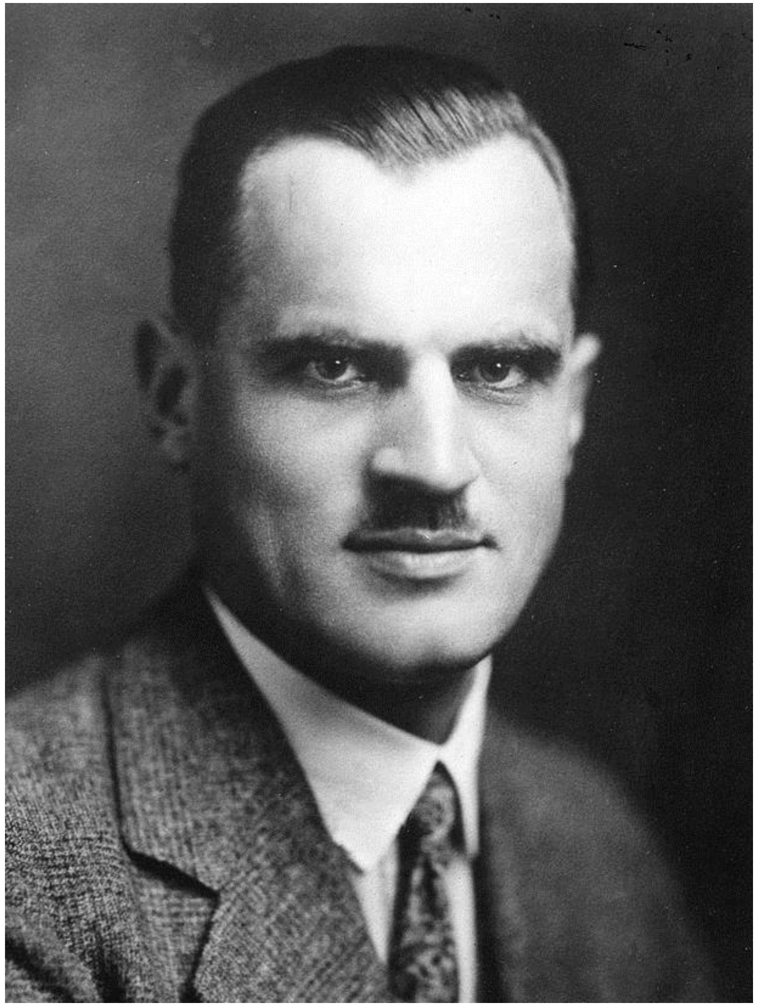
From Physics4Students
https://www.youtube.com/watch?v=yFI9xb2uLsU



Spectra of rhodium rays : 100 planes of rock-salt.

"The Structure of Diamond," Bragg & Bragg, Proc. Royal. Soc. 1913

X-rays are electromagnetic radiation and are photons.



Arthur H. Compton
(1892-1962)

ARTHUR H. COMPTON

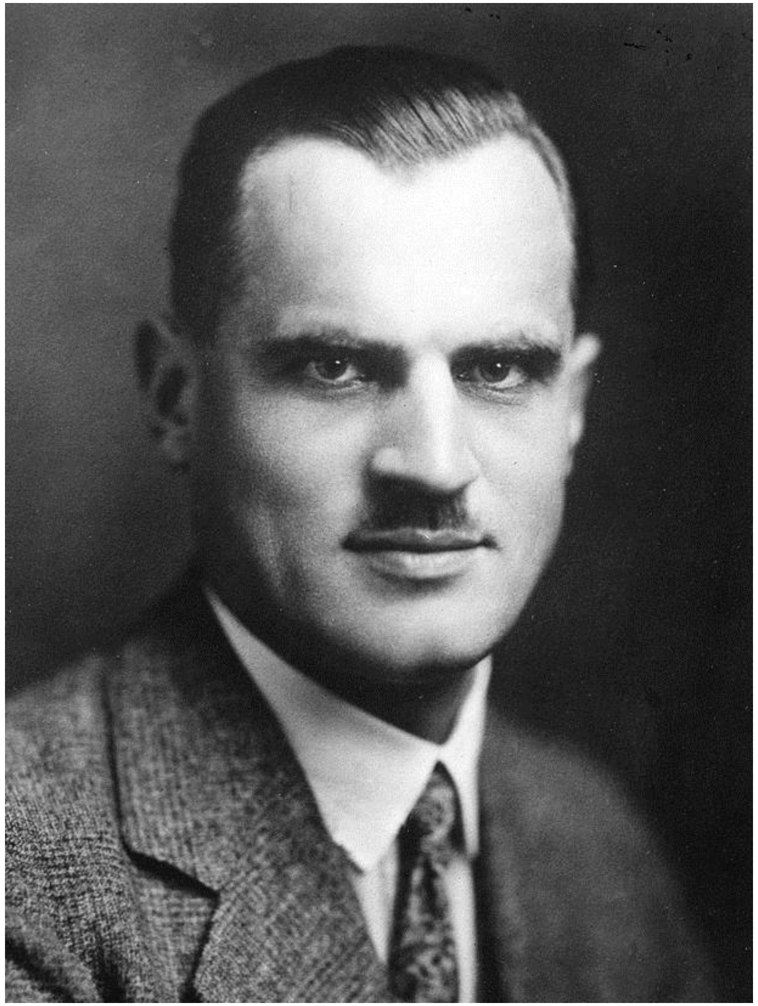
X-rays as a branch of optics

Nobel Lecture, December 12, 1927

First part of lecture: Summary of work by Compton and others that proves that X-rays are part of the electromagnetic spectrum: successive patchwork studies using higher and higher order reflection from gratings.

Second part of lecture: The Compton effect, ***photon quantization***, and relativistic kinematics of photon-electron scattering.

X-rays are electromagnetic radiation and are photons.



Arthur H. Compton
(1892-1962)

ARTHUR H. COMPTON

X-rays as a branch of optics

Nobel Lecture, December 12, 1927

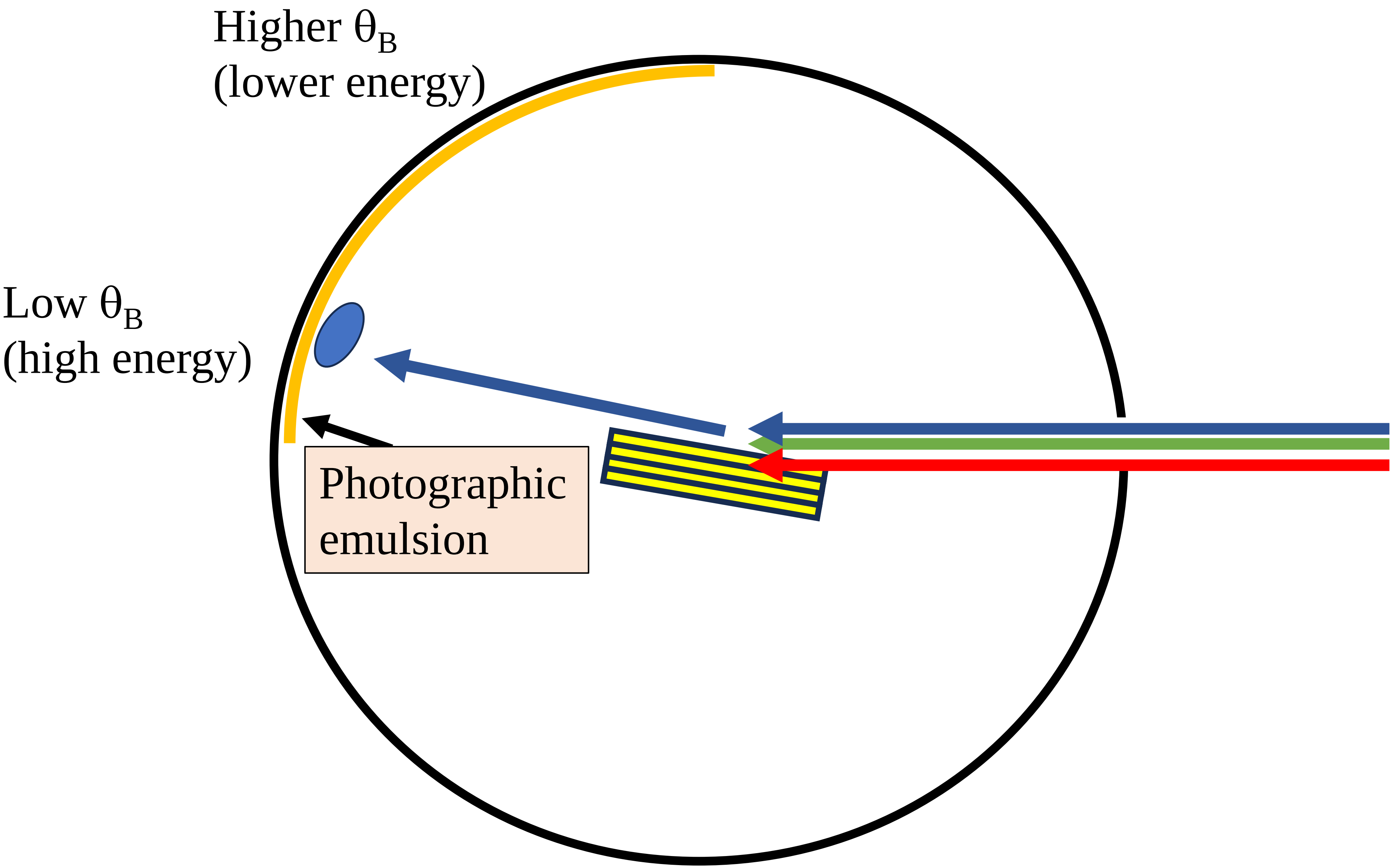
One of the most fascinating aspects of recent physics research has been the gradual extension of familiar laws of optics to the very high frequencies of X-rays, until at the present there is hardly a phenomenon in the realm of light whose parallel is not found in the realm of X-rays. Reflection, refraction, diffuse scattering, polarization, diffraction, emission and absorption spectra, photoelectric effect, all of the essential characteristics of light have been found also to be characteristic of X-rays. At the same time it has been found that some of these phenomena undergo a gradual change as we proceed to the extreme frequencies of X-rays, and as a result of these interesting changes in the laws of optics we have gained new information regarding the nature of light.

Manne Siegbahn (1886-1978)

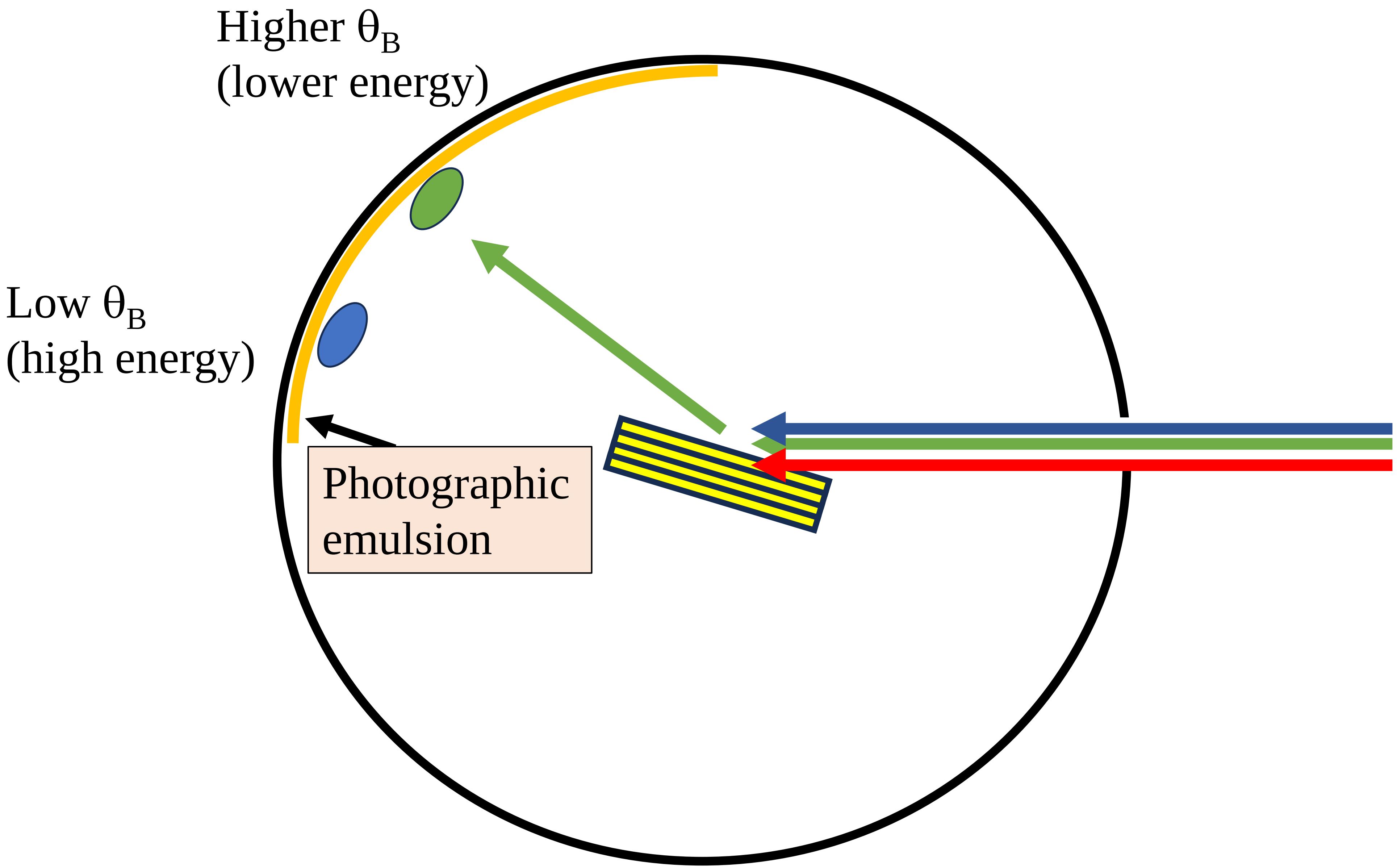
Led the development of high-resolution x-ray spectrometers using Bragg diffraction from perfect crystals to study x-ray fluorescence.

1924 Nobel Prize for “The X-rays Spectra and the Structure of Atoms”

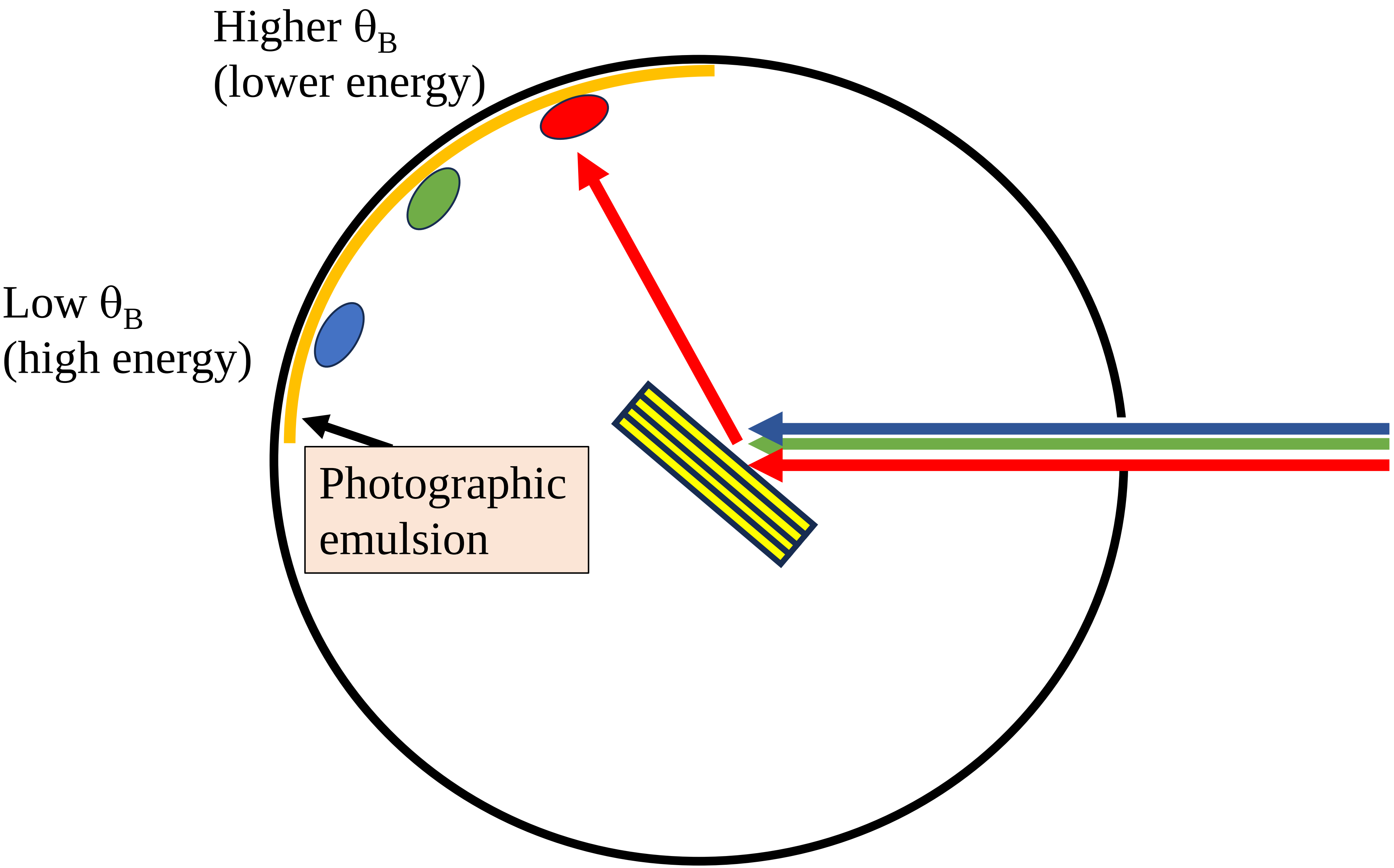
‘Rotating Crystal’ Spectrometer (sort of Bragg-Brantano…)

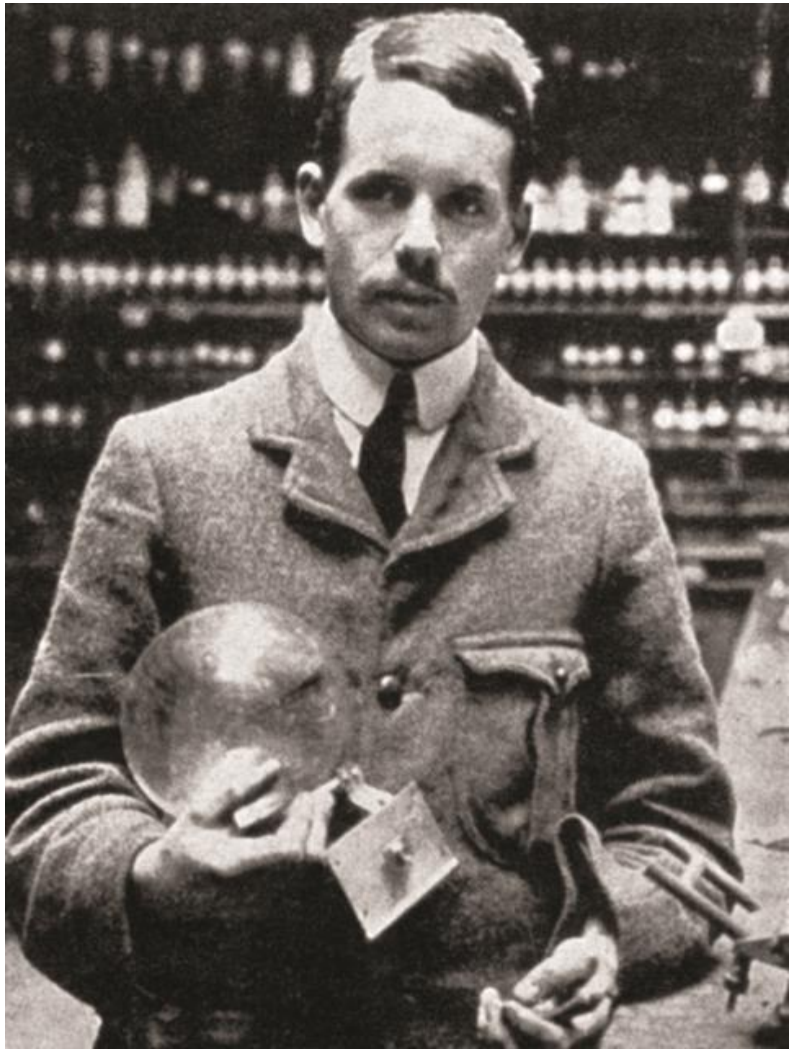


'Rotating Crystal' Spectrometer (sort of Bragg-Brantano…)

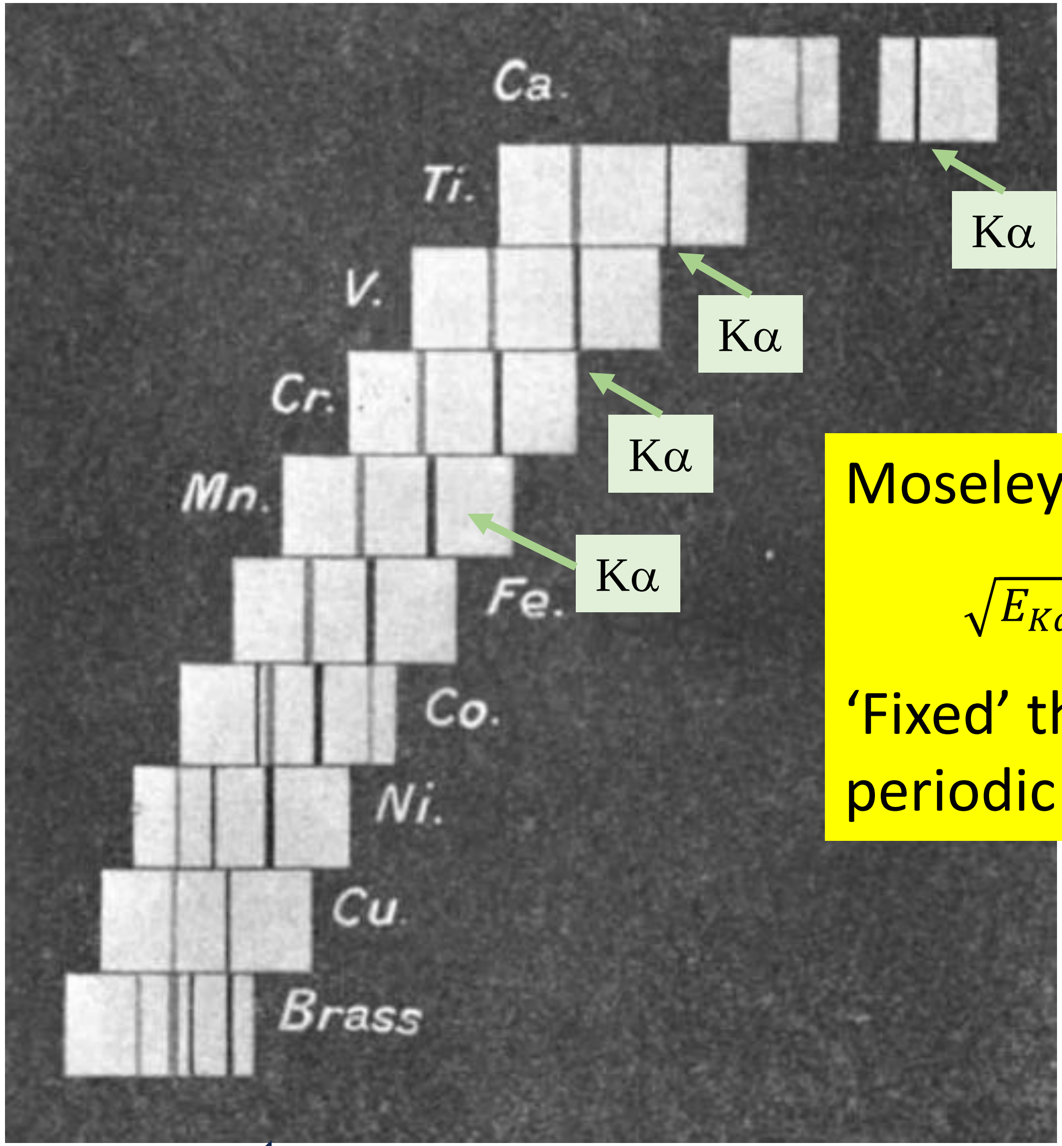


‘Rotating Crystal’ Spectrometer (sort of Bragg-Brantano…)





Henry Moseley
(1887 – 1915)



Moseley's Law:

$$\sqrt{E_{K\alpha}} \propto Z$$

'Fixed' the periodic table.

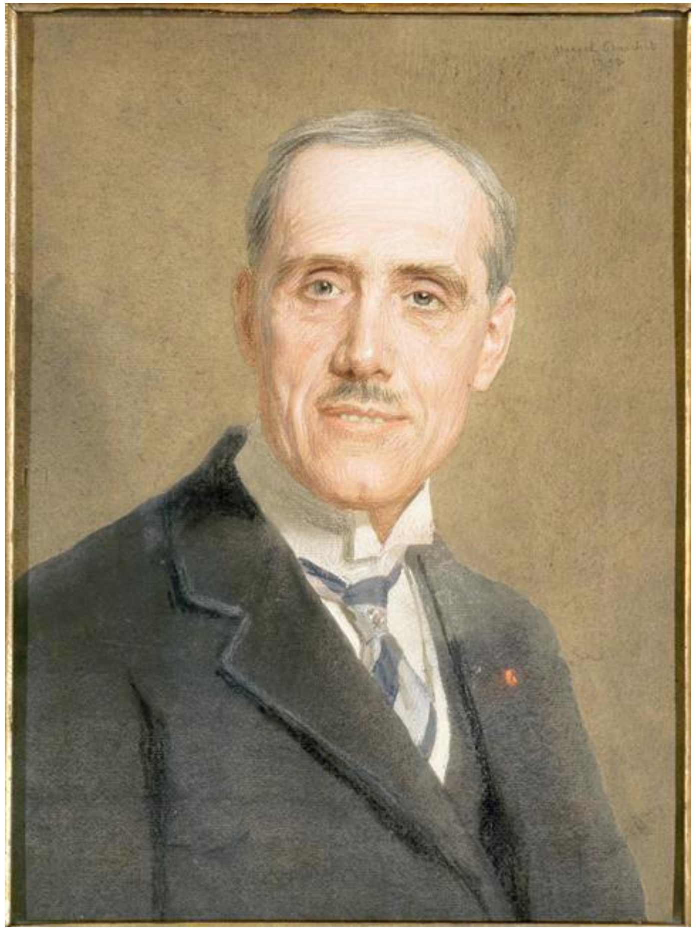
Increasing frequency

H.G.J. Moseley, Phil. Mag. **26**, 1024 (1913)

Siegbahn, Moseley and others performed exacting studies of x-ray fluorescence.

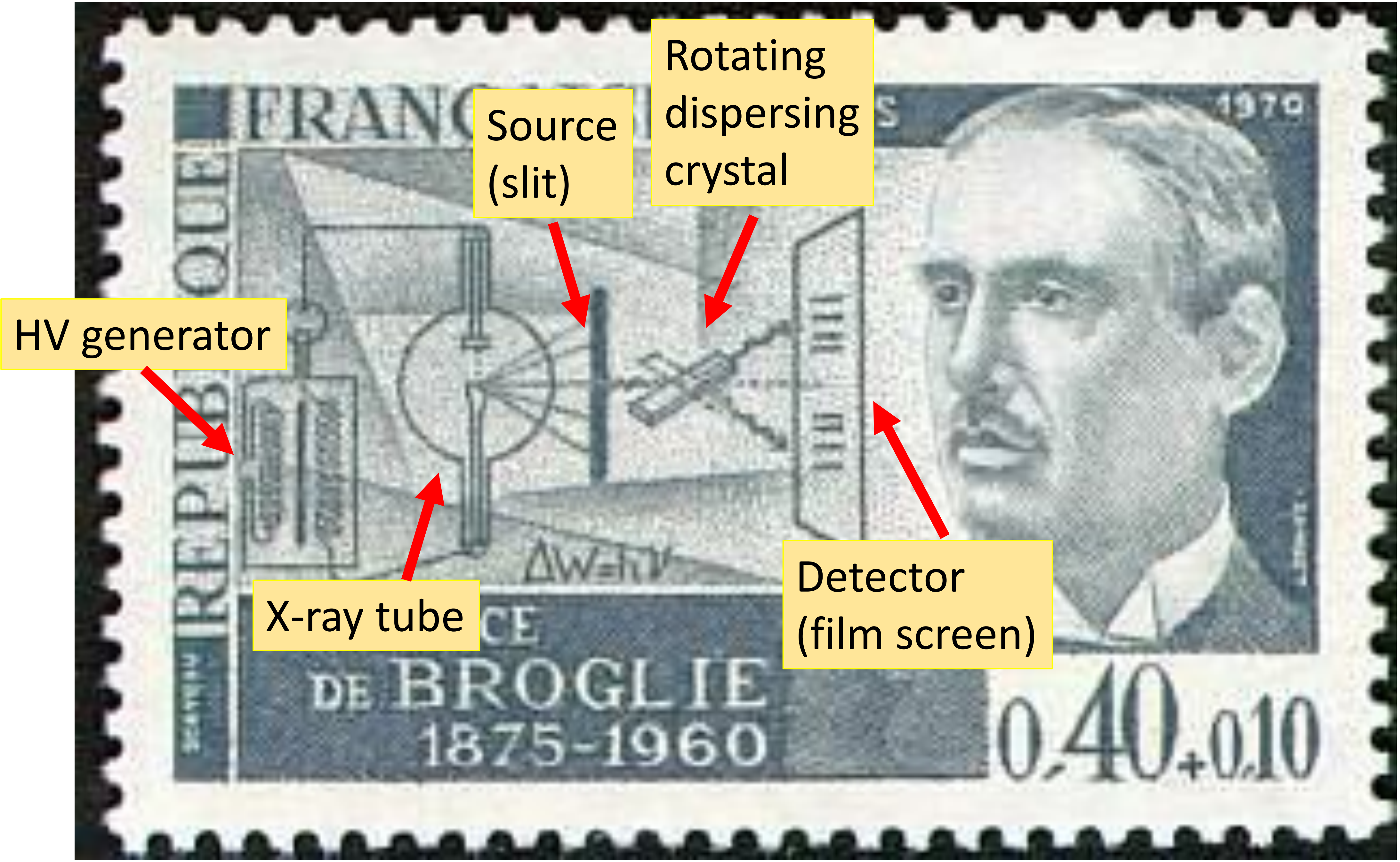
What about absorption?

The First Evidence for an Absorption Edge at X-ray Energies



M. deBroglie and F.A. Lindemann, Vehr.Dtsch.Phys.Ges. **16** 195 (1914)

Maurice deBroglie
(1875-1960)


Rotating dispersing crystal
Source (slit)
HV generator
X-ray tube
Detector (film screen)
FRANÇAIS
1970
REPUBLIQUE
ΔW=hν
DE BROGLIE
1875-1960
0,40+0,10

The First Evidence for an Absorption Edge at X-ray Energies

E. Wagner, Ann. Phys. **46**, 868-892 (1915)

Ernst Wagner (?) (1876-1928)
[Ph.D. student of Roentgen]

… also recognized by von Laue in his Nobel Address for early precise measurements of Planck’s constant via the Bremsstrahlung endpoint.

https://commons.wikimedia.org/wiki/File:Ernst_Wagner_CIPB0723.jpg

The First Evidence for an Absorption Edge at X-ray Energies

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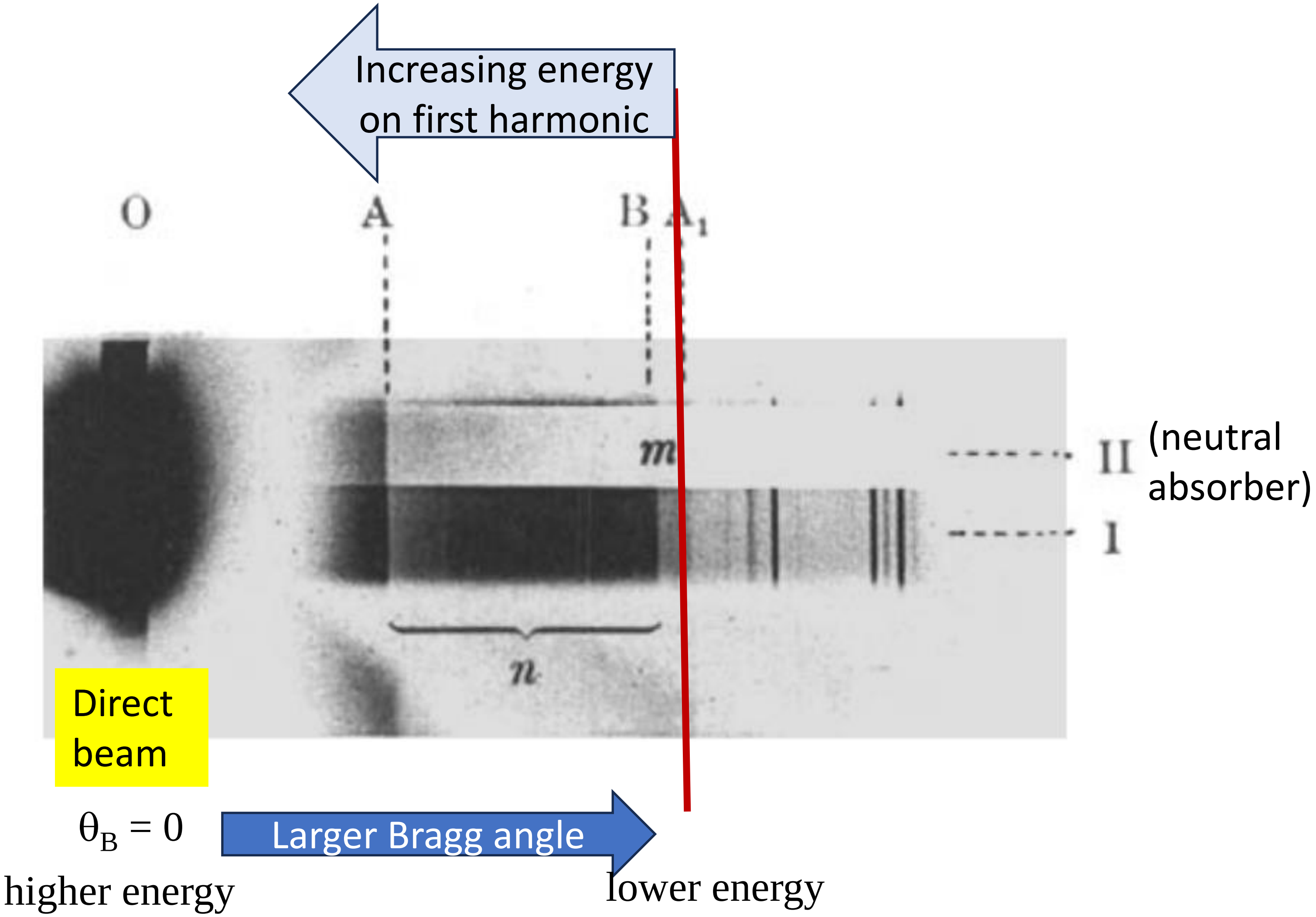
[Ph.D. student of Roentgen]

https://commons.wikimedia.org/wiki/File:Ernst_Wagner_CIPB0723.jpg

AgBr film spanning a range of Bragg angles in a rotating crystal spectrometer.



The First Evidence for an Absorption Edge at X-ray Energies

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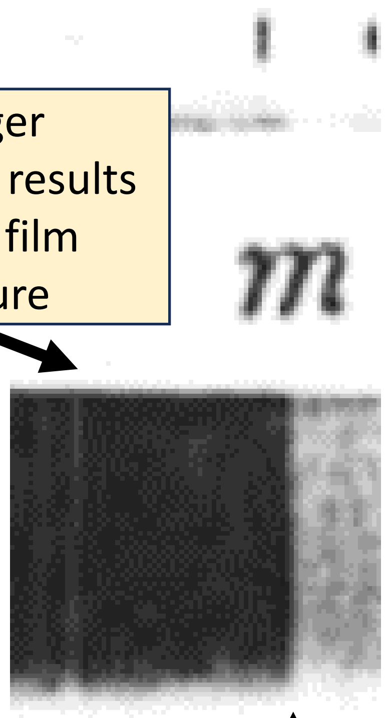
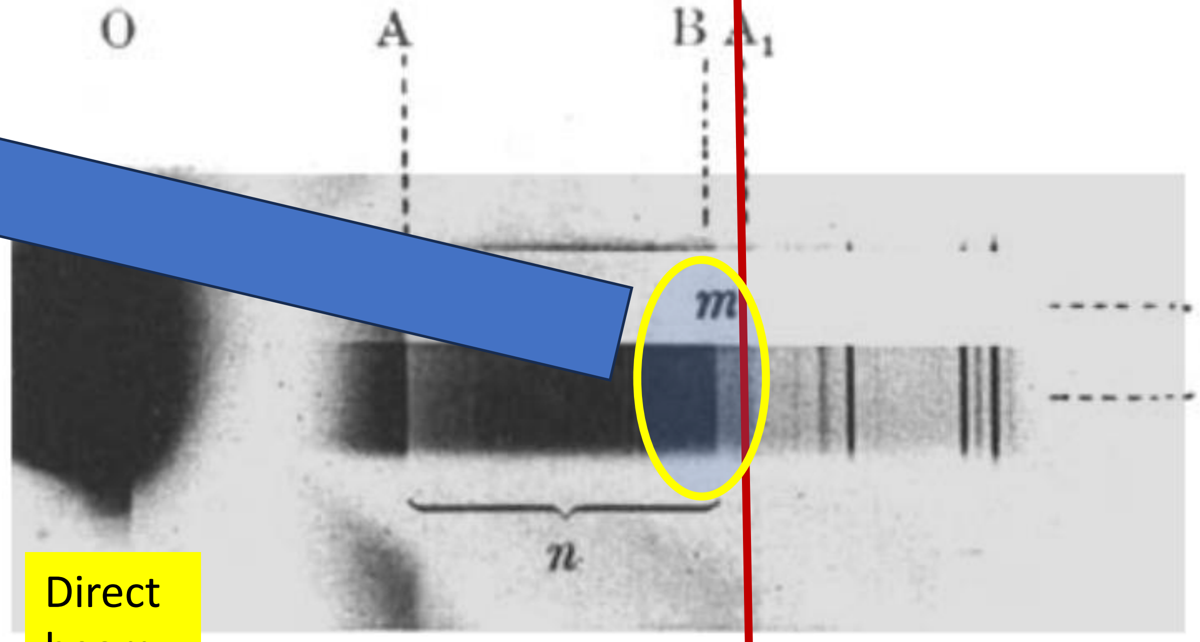


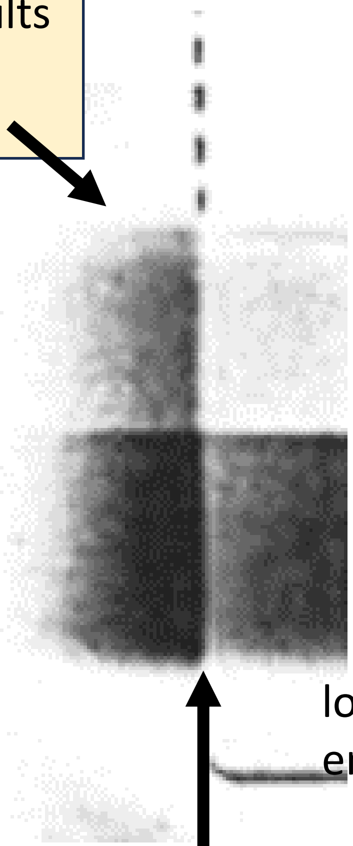


The First Evidence for an Absorption Edge at X-ray Energies

E. Wagner, Ann. Phys. **46**, 868-892 (1915)

AgBr film spanning a range of Bragg angles in a rotating crystal spectrometer.

Stronger absorption results in more film exposure

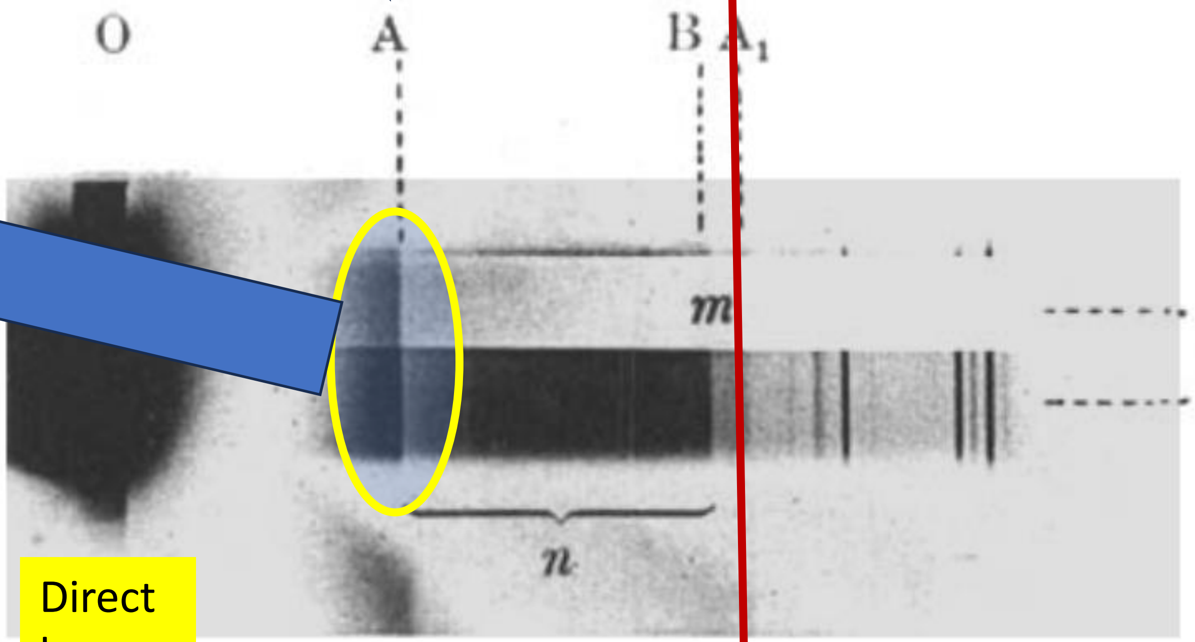


higher energy

lower energy

Ag K-edge
25.5 keV

Increasing energy on first harmonic



(neutral absorber)

Direct beam

$\theta_B = 0$

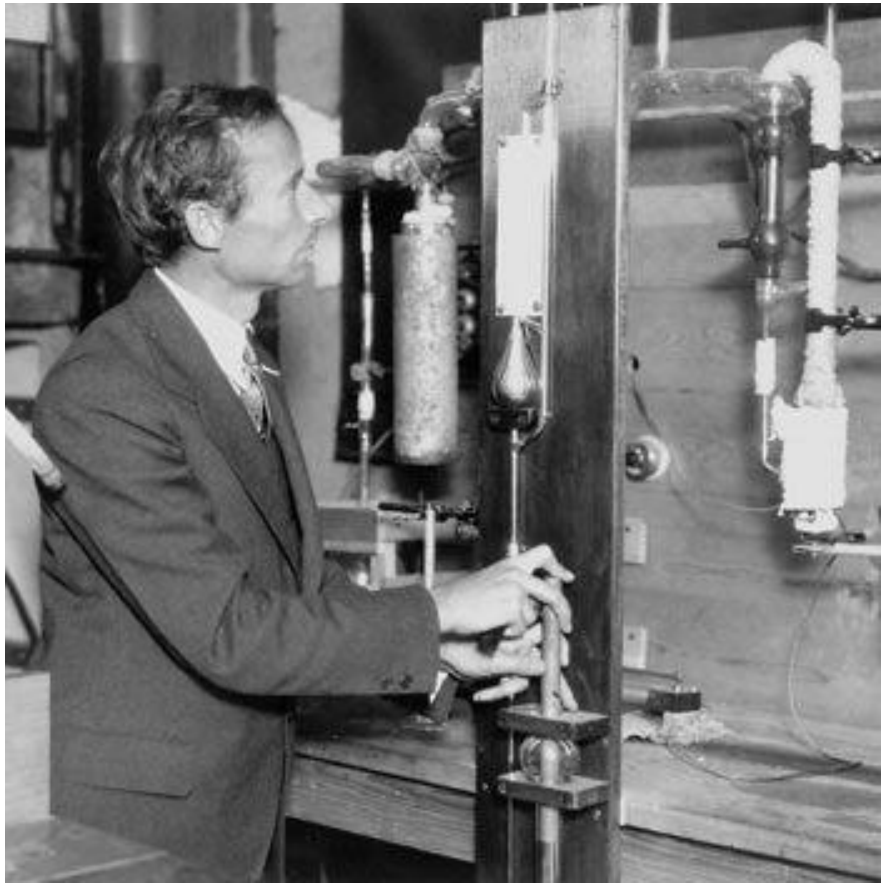
higher energy

Larger Bragg angle

lower energy

The First Evidence for Fine Structure

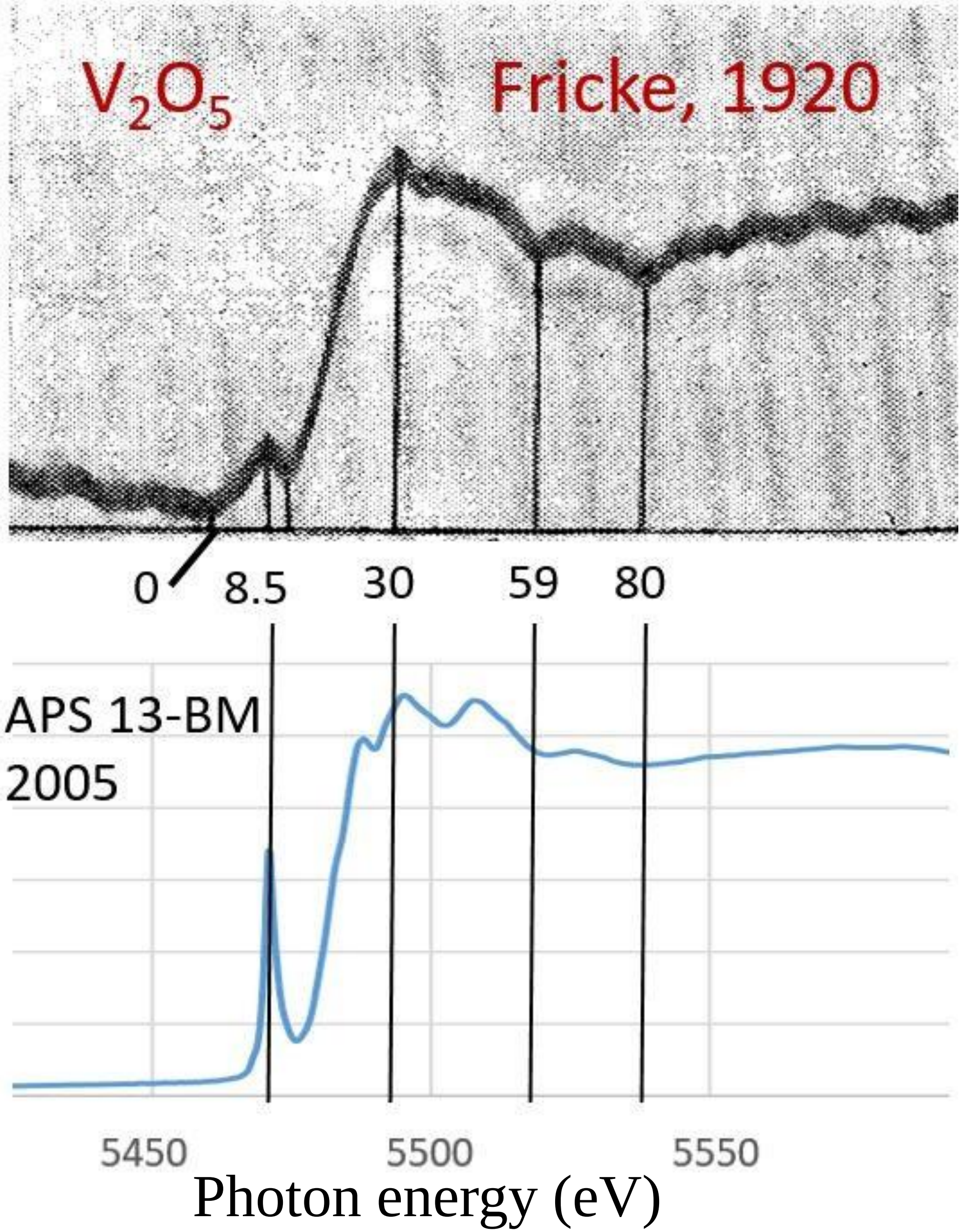
Hugo Fricke, Phys. Rev. **16**, 202 (1920)



Hugo Fricke
(1892 – 1972)

Student and postdoc: Worked with Bohr, Seigbahn, and Lyman

Fricke was a prominent early researcher on x-ray dosimetry and the biological effects of x-ray exposure.



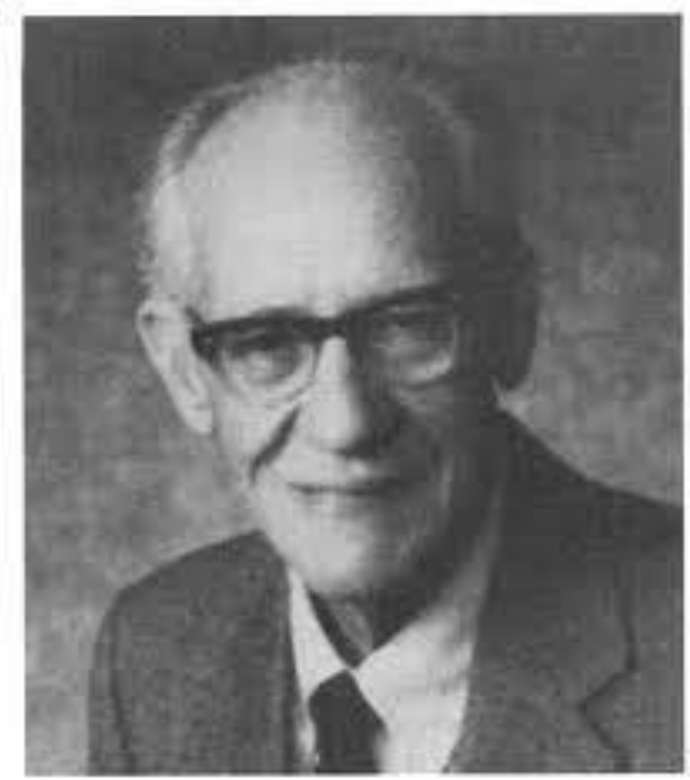
Prior to 1920, spectrometers were built for three reasons:

- To understand "what are x-rays"
- To understand atoms:
  - measure fluorescence energies
  - measure absorption energies

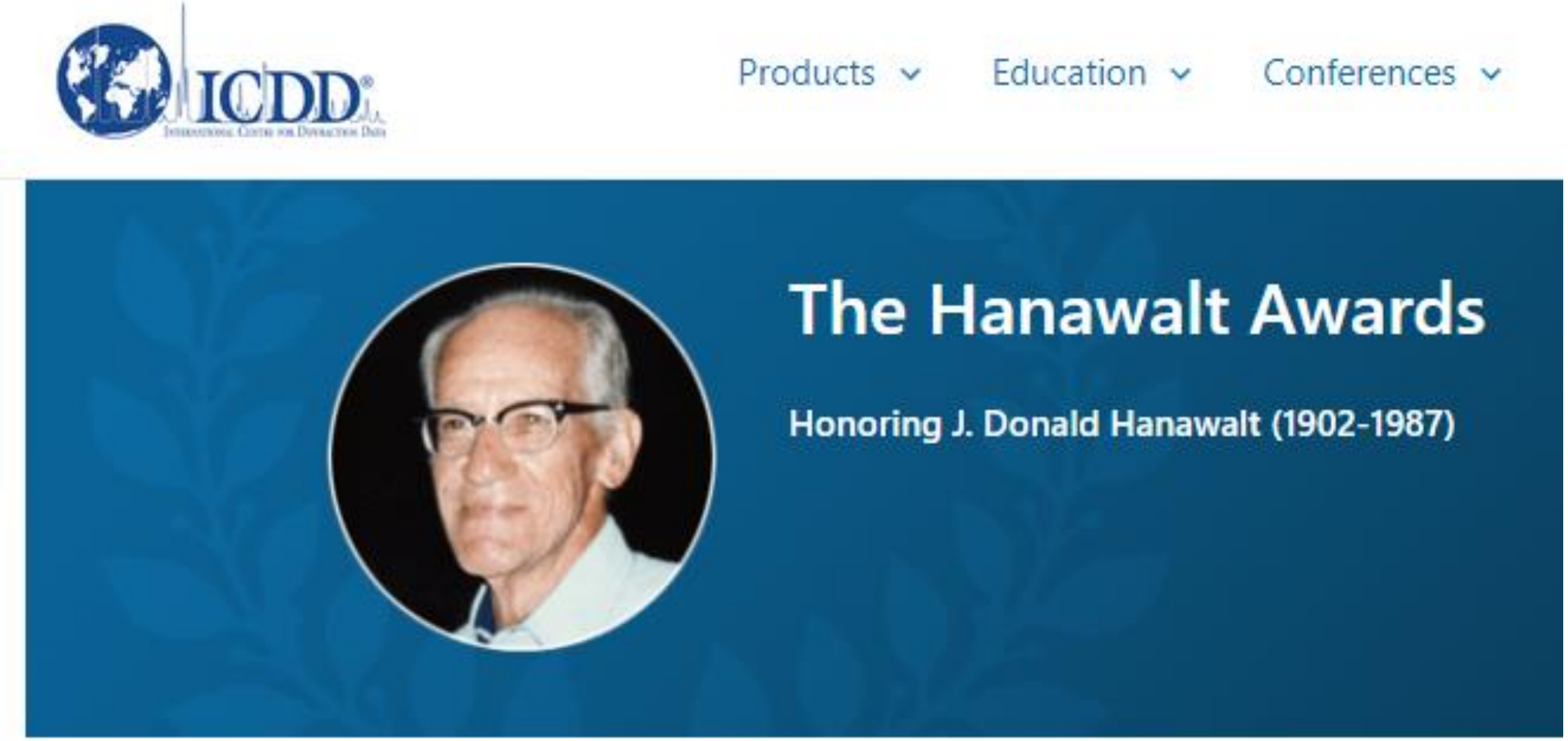
The community realized that the fine structure had something to do with crystal structure, and the success of diffraction and emergence of quantum theory made XAFS a hot research topic.

Scientists started building spectrometers to learn what caused the fine structure.

Is the ‘fine structure’ due to purely atomic processes, or does the local environment play a role?

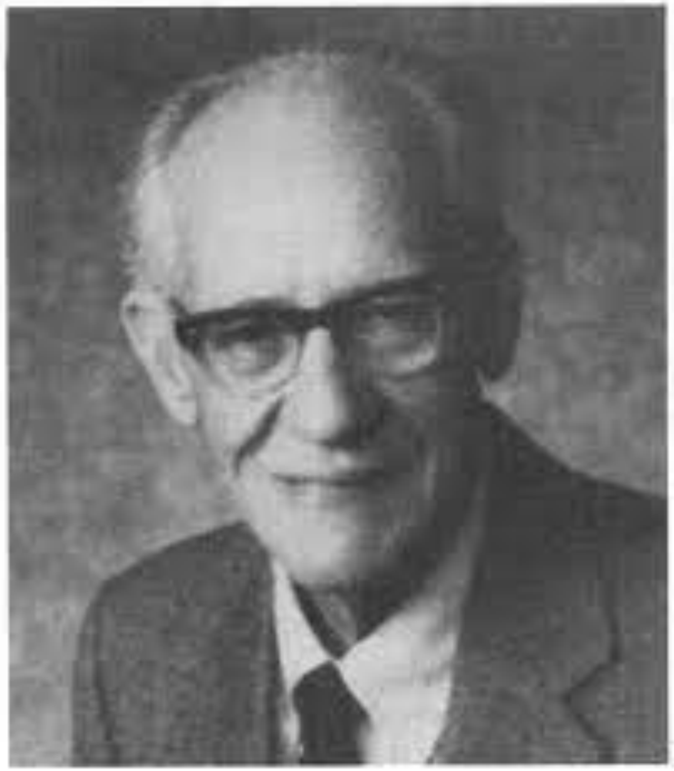


J. Donald Hanawalt
(1902-1987)



One of the founders of analytical powder XRD.

Is the ‘fine structure’ due to purely atomic processes, or does the local environment play a role?



J. Donald Hanawalt
(1902-1987)

MARCH 15, 1931 *PHYSICAL REVIEW* *VOLUME 37*

THE DEPENDENCE OF X-RAY ABSORPTION SPECTRA UPON CHEMICAL AND PHYSICAL STATE

BY J. D. HANAWALT*
UNIVERSITY OF MICHIGAN

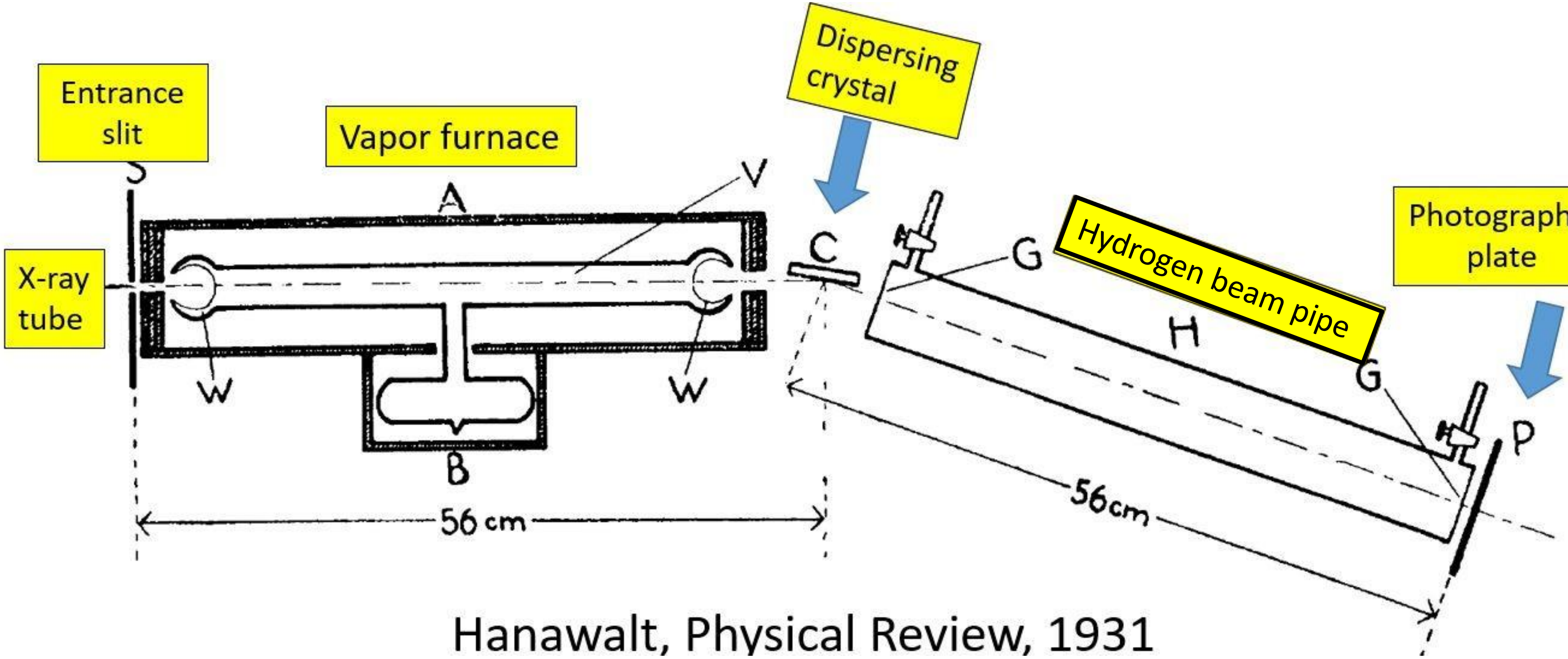
Der Einfluß der Temperatur auf die *K*-Absorption des Eisens.

Von **J. D. Hanawalt** [1]), zurzeit in Groningen.

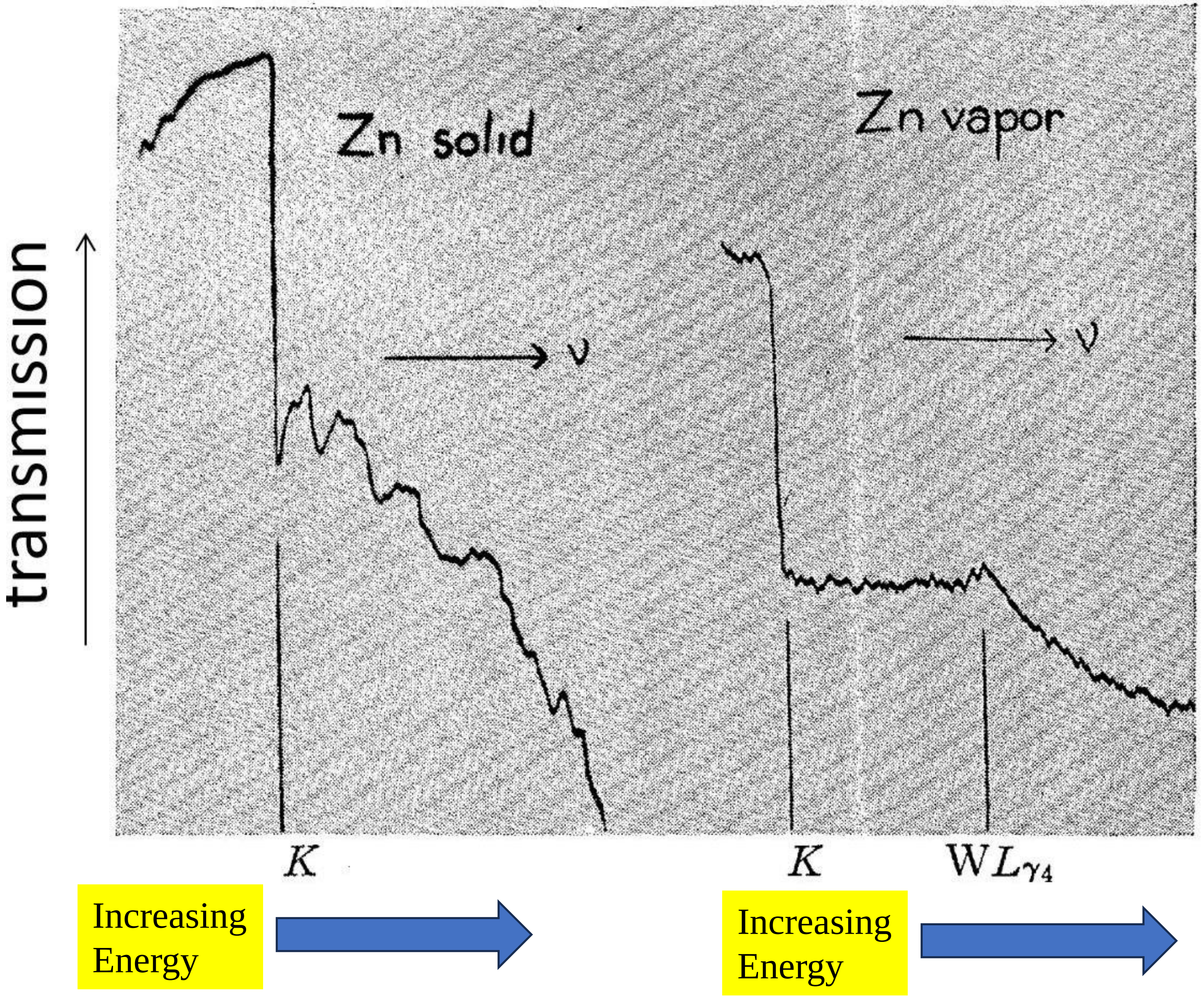
Mit 4 Abbildungen. (Eingegangen am 7. Mai 1931.)

[J.D. Hanawalt, “The Influence of Temperature on the K-absorption of Iron”, Z. Phys. 1931]

Is the ‘fine structure’ due to purely atomic processes, or does the local environment play a role?

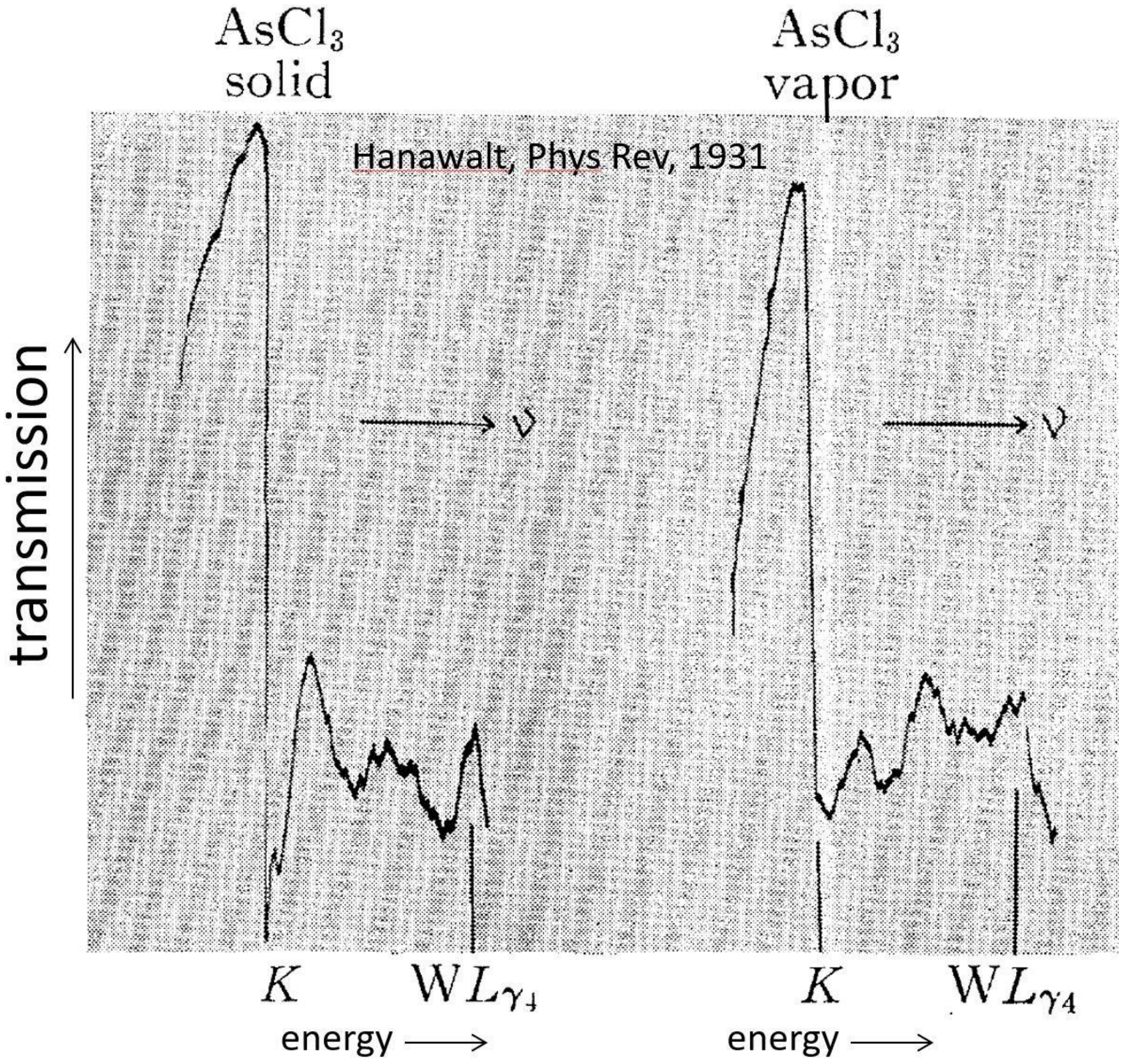


Hanawalt, Physical Review, 1931



Hanawalt, Phys. Rev. 1931

Solids show fine structure, but isolated atoms do not.



Hanawalt, Phys. Rev. 1931

Molecular solids/liquids show fine structure.

And a gas of molecules *also* shows fine structure, **which is different than for the solid.**

The strong effect of temperature on x-ray diffraction was already known.   What about XAFS?

Temperature dependence of fine structure: Hanawalt, Z.Phys. 1931

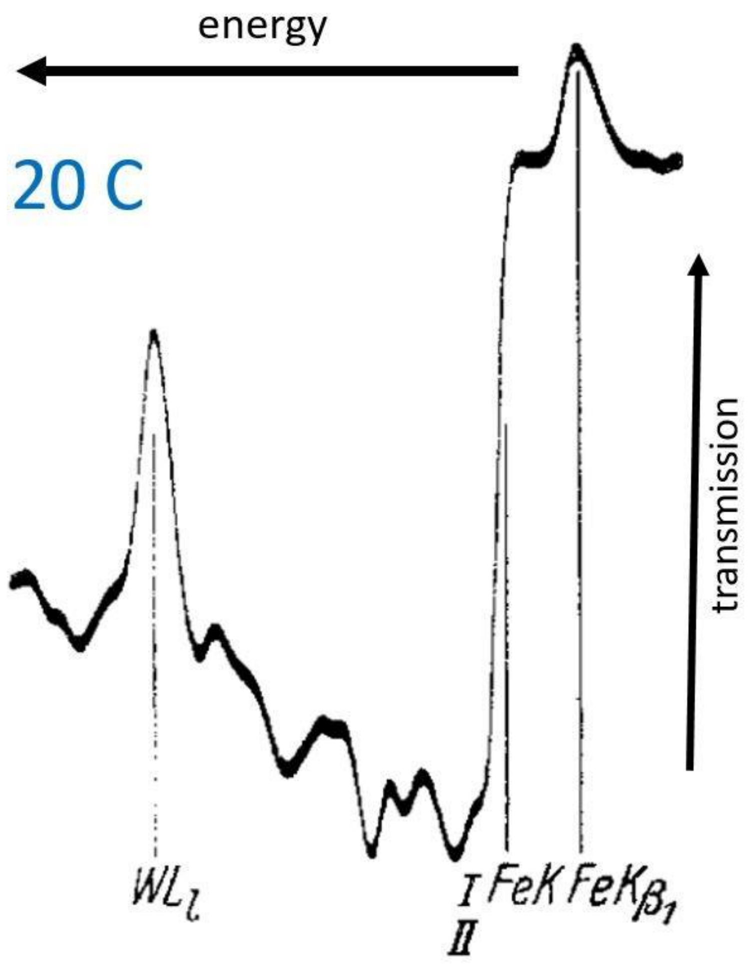


Fig. 2a.
Absorptionskante für Eisen bei 20⁰ C.

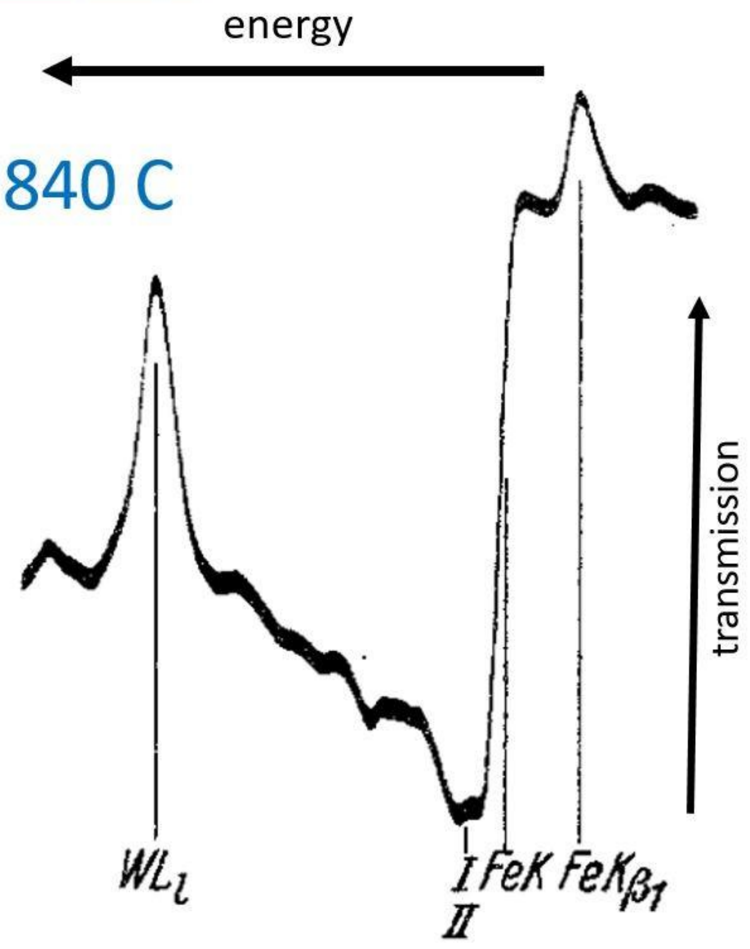


Fig. 2b.
Absorptionskante für Eisen bei 840⁰ C.

The strong effect of temperature on x-ray diffraction was already known. What about XAFS?

Temperature dependence of fine structure: Hanawalt, Z.Phys. 1931

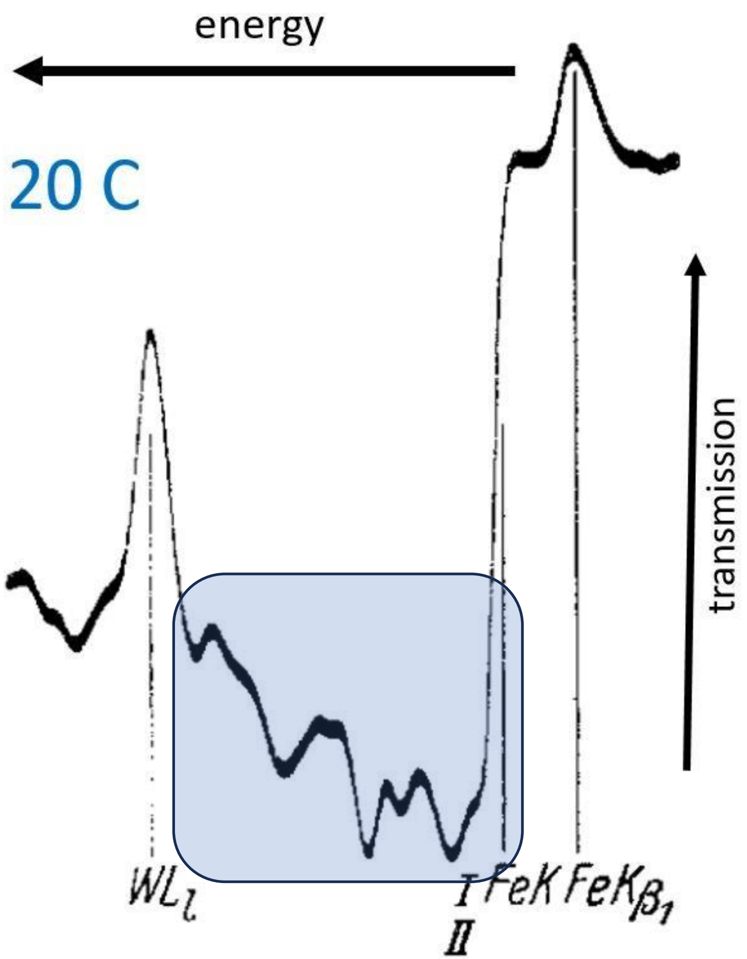


Fig. 2a.
Absorptionskante für Eisen bei 20⁰ C.

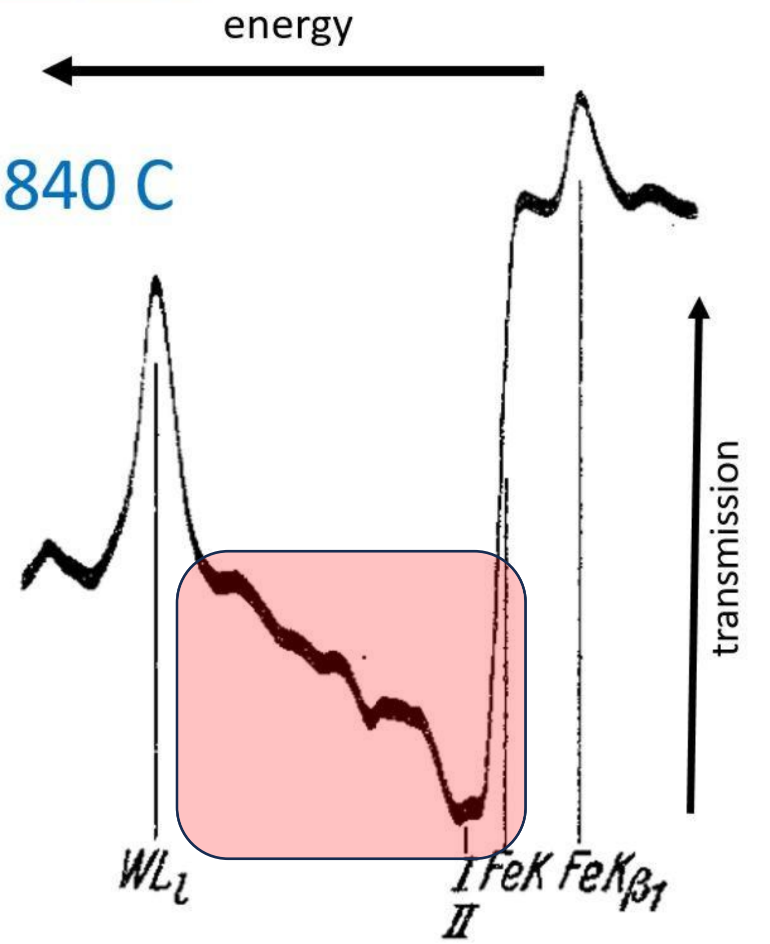
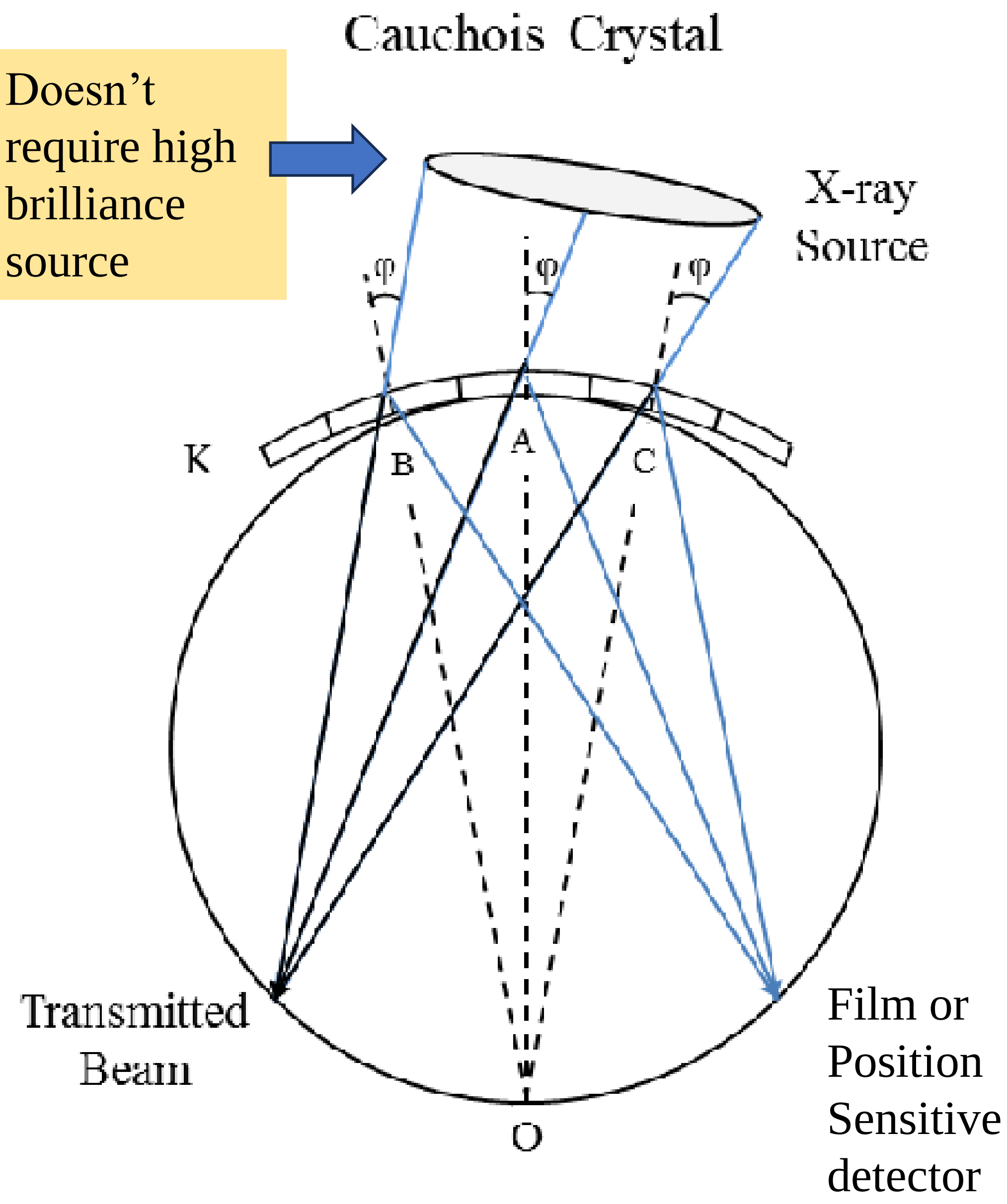


Fig. 2b.
Absorptionskante für Eisen bei 840⁰ C.

Doesn’t require high brilliance source



A. Goodsell, U Texas dissertation (2012)

Yvette Cauchois
(1908-1999)

F. Lytle, J Synch Rad (1999):

Early in a long career devoted to the study of the emission and absorption of X-rays, Yvette Cauchois (Cauchois, 1932, 1933), Fig. 2, developed the transmission bent-crystal X-ray spectrometer. This instrument allowed rapid accurate measurement of absorption edges. For 40 years it was used for short-wavelength spectroscopy in many laboratories of the world. The modern equivalent is

→1971: Many More Studies aimed at Understanding EXAFS

Common: Use lots of slits and a flat crystal on a diffractometer goniometer.

Advantage: Very high resolution, excellent mechanical aspects are already fully engineered.

Disadvantage: Very low flux. Harmonic contamination (detector limitation)

→1971: Many More Studies aimed at Understanding EXAFS

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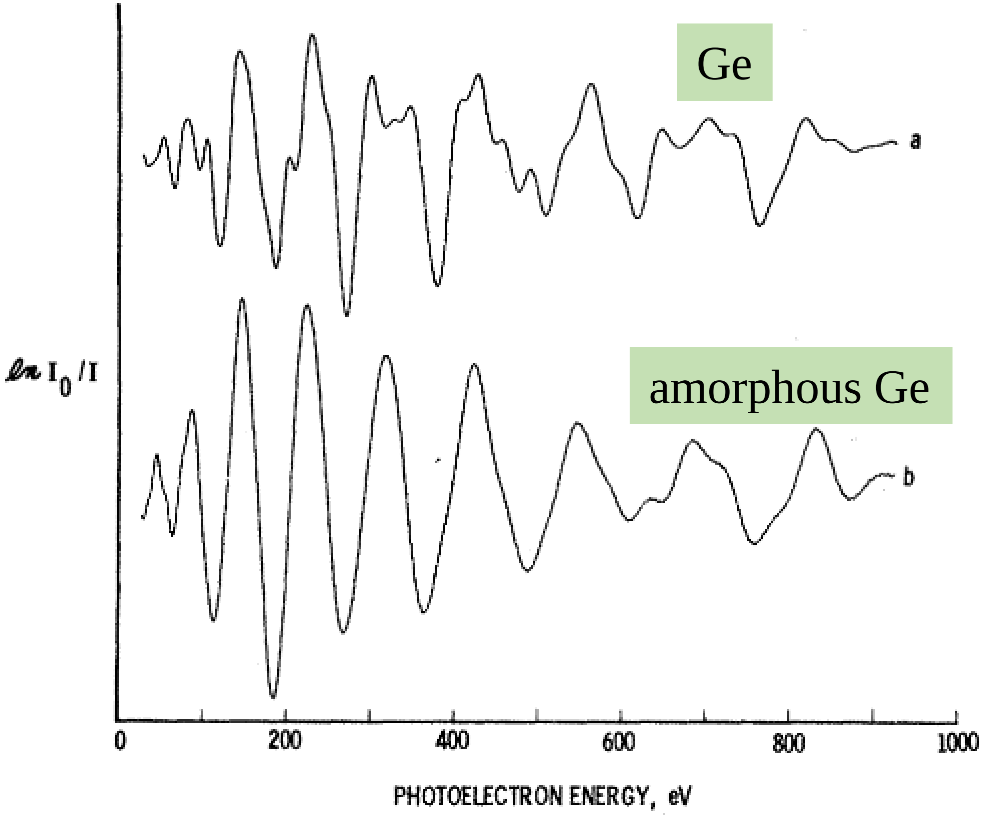


FIG. 1. Smoothed experimental EXAFS data for (a) crystalline and (b) amorphous Ge. Only the oscillatory part χ of the absorption edge is shown.

VOLUME 27, NUMBER 18 PHYSICAL REVIEW LETTERS 1 NOVEMBER 1971

New Technique for Investigating Noncrystalline Structures: Fourier Analysis of the Extended X-Ray—Absorption Fine Structure*

Dale E. Sayers† and Edward A. Stern†‡
Department of Physics, University of Washington, Seattle, Washington 98105

and

Farrel W. Lytle
Boeing Scientific Research Laboratories, Seattle, Washington 98124

(Received 16 July 1971)

Ed Stern Dale Sayers Farrel Lytle

1950’s to 1990’s: Better X-rays Sources and Better Optics

1980’s Dominant approach: Use a curved cylindrical analyzer on the Rowland circle.

Advantage: Much higher flux.

Disadvantage: Optic quality, Rowland spectrometer construction needed, harmonic contamination (detector limitation)

Discussed in detail in “Laboratory EXAFS facilities, 1980 : University of Washington workshop”, edited E.A. Stern

EVALUATION OF FOCUSING
MONOCHROMATORS FOR AN EXAFS SPECTROMETER*

S. M. Heald
Brookhaven National Laboratory, Upton, NY 11973

ABSTRACT

Various focusing monochromators are compared and evaluated for use in an EXAFS spectrometer. For a line focus the Johann and Johansson cases are found to be most suitable. Obtaining a point focus is more difficult, with the best case appearing to be a singly bent mirror used in conjunction with the Johann or Johansson crystals.

1950’s to 1990’s: Better X-rays Sources and Better Optics

1980’s Dominant approach: Use a curved cylindrical analyzer on the Rowland circle.

Advantage: Much higher flux.

Disadvantage: Optic quality, Rowland spectrometer construction needed, harmonic contamination (detector limitation)

Discussed in detail in “Laboratory EXAFS facilities, 1980 : University of Washington workshop”, edited E.A. Stern

EVALUATION OF FOCUSING
MONOCHROMATORS FOR AN EXAFS SPECTROMETER*

S. M. Heald
Brookhaven National Laboratory, Upton, NY 11973

ABSTRACT

Various focusing monochromators are compared and evaluated for use in an EXAFS spectrometer. For a line focus the Johann and Johansson cases are found to be most suitable. Obtaining a point focus is more difficult, with the best case appearing to be a singly bent mirror used in conjunction with the Johann or Johansson crystals.

1970's-90's: Make laboratory instruments to **use** EXAFS

1980's Dominant approach: Use a curved cylindrical analyzer on the Rowland circle.

Advantage: Much higher flux.

Disadvantage: Optic quality, Rowland spectrometer construction needed, harmonic contamination (detector limitation)

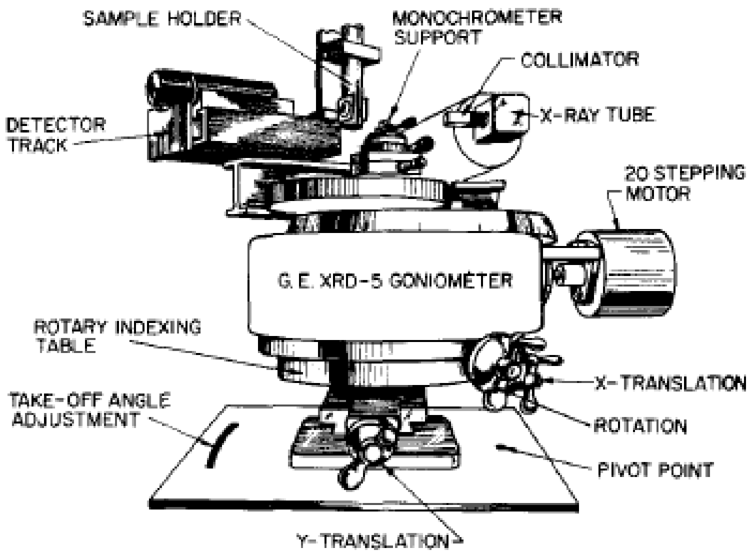
Rev Sci Instrum 1978

Development of a laboratory EXAFS facility

G. S. Knapp, Haydn Chen, and T. E. Klippert

Argonne National Laboratory, Argonne, Illinois 60439

(Received 14 March 1978; in final form, 22 May 1978)



Physica B 1989

A New In-Laboratory XAFS Facility

G.S. Knapp, A.G. McKale,
Surface Science Instruments, 465 National Avenue, Mtn. View, CA 94043

J. Quintana and T. Georgopoulos
Northwestern University, Evanston, IL

Commercialized instrument with multiple cylindrical analyzers for different energy ranges.

"energy resolution 10eV or less…"

Design: **Linear Spectrometer**

physica status solidi (a)

Original Paper

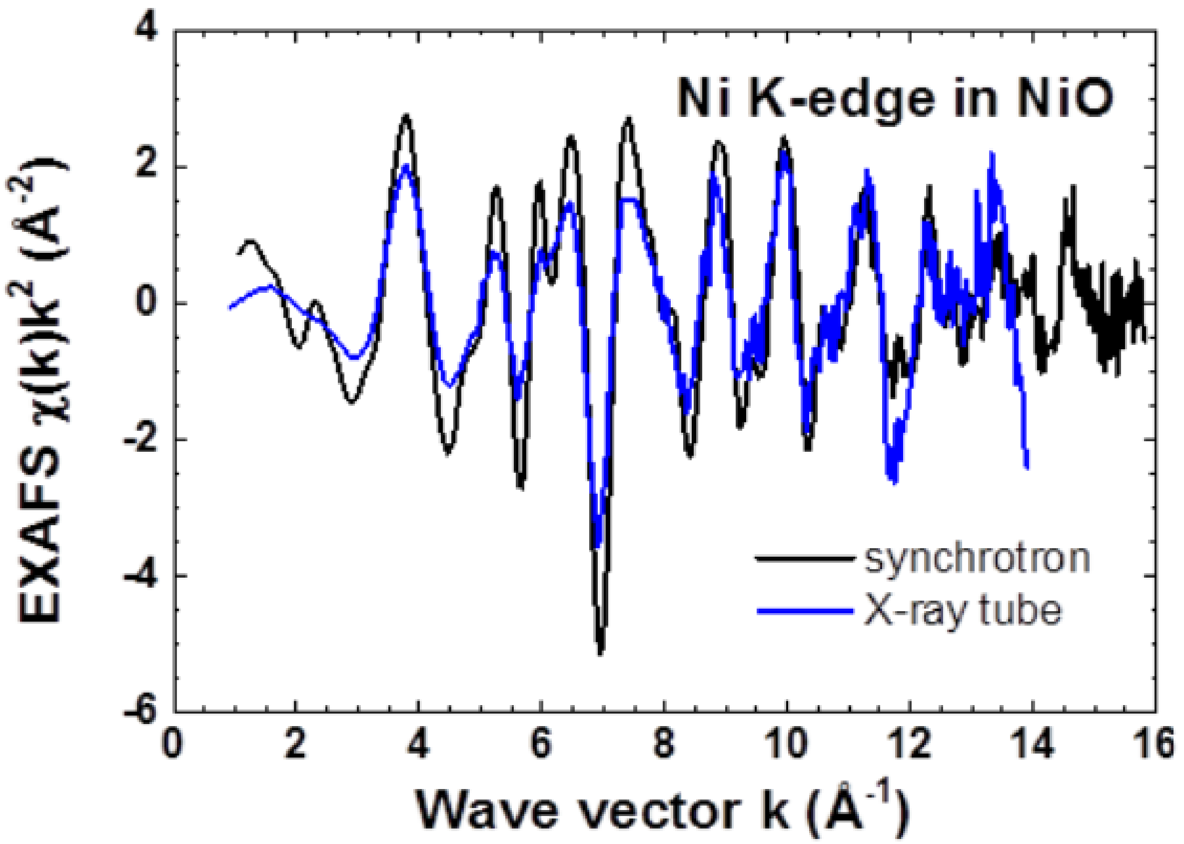
EXAFS and XANES Studies of $Co_xMg_{1-x}O$ Solid Solutions Using a Laboratory EXAFS Spectrometer

A. Kuzmin, N. Mironova, J. Purans, A. Sazonov

First published: 16 January 1993 | https://doi.org/10.1

Kengaraga 8, 226063 Riga, Latvia.
229021 Salaspils, Latvia.

(1989-1997)



Dynamically curved quartz analyzer, energy resolution 5 - 10 eV.

Among the best of the home-built laboratory systems of this era.

1970's-90's: Make laboratory instruments to <u>**use**</u> EXAFS

1980's Dominant approach: Use a curved cylindrical analyzer on the Rowland circle.

Advantage: Much higher flux.

Disadvantage: Optic quality, Rowland spectrometer construction needed, harmonic contamination (detector limitation)

1980's (and beyond) ***extensive and very careful*** work by academics in Japan and Rigaku Corp to refine and perfect the linear spectrometer with cylindrical analyzers, but generally still limited by optics performance and detector limitations.

Physica Scripta. Vol. T115, 1017–1018, 2005

Customization of an In-House XAFS Spectrometer for Sulfur Measurement

T. Taguchi[1*], K. Shinoda[2] and K. Tohji[2]

[1]XRD-division, Rigaku Corporation, 3-9-12 Matsubara-cho, Akishima-shi, Tokyo 196-8666, Japan
[2]Graduate School of Environmental Studies, TOHOKU University, 01 Aza-Aoba, Aramaki, Aoba-ku, Sendai-shi, Mi

Kazuyuki Tohji

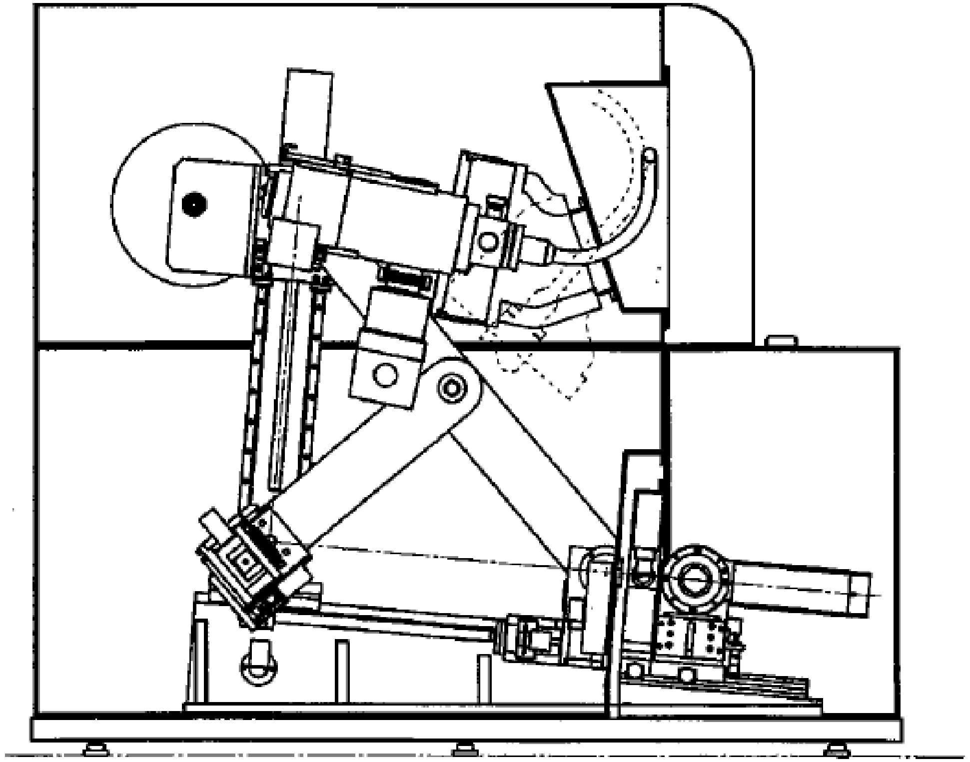
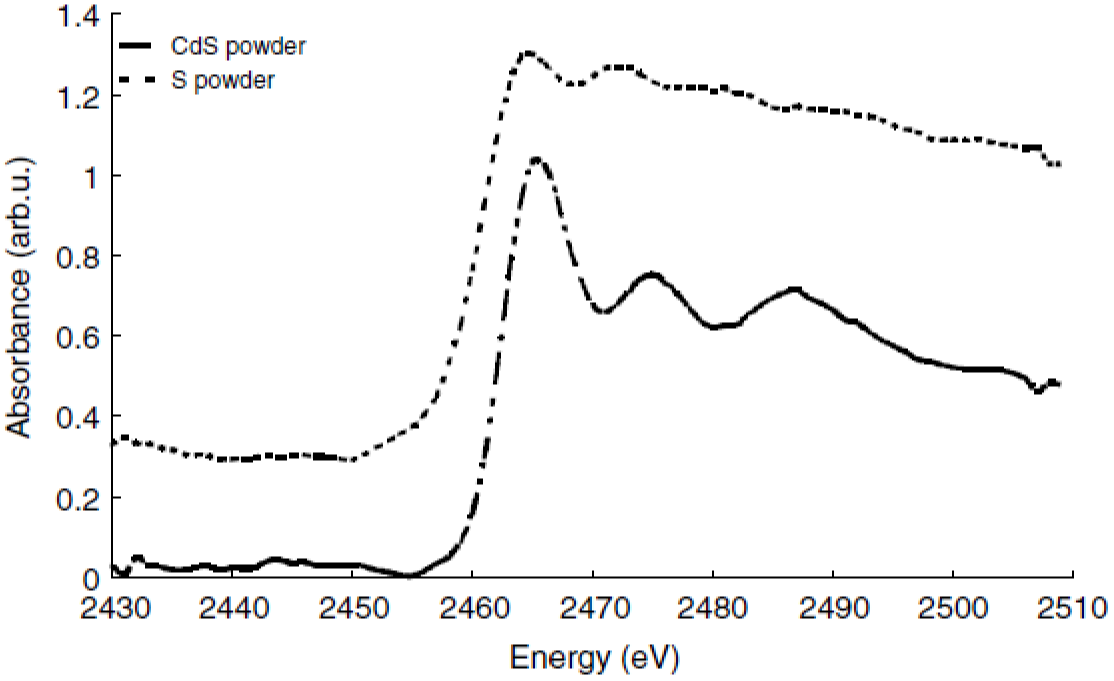


Fig. 1. Schematic diagram of the in-house spectrometer and helium chamber.



And then the use of lab XAFS faded in the 1980's-90's-00's.

J. Synchrotron Rad. (1999). **6**, 123–134

The EXAFS family tree: a personal history of the development of extended X-ray absorption fine structure

Farrel W. Lytle

The EXAFS Company, Pioche, NV 89043, USA. E-mail: fwlytle@pioche.igate.com

Then the Stanford Radiation Research Project started and you know the rest of that story. In one trip to the synchrotron we collected more and better data in three days than in the previous ten years. I shut down all three X-ray spectrometers in the Boeing laboratory. A new era had arrived!

Mid-1980’s

2013

The reasons:

1) **Detector problems**: Often impossible to remove harmonics in lab XAFS and solid-state detectors were still in their early stages in 1980’s. Harmonic contamination kills data quality.
2) **Synchrotron access** (for a while) grew, throughput went up, many new EXAFS applications required the higher flux, XANES hadn’t gained its present importance.
3) The XAFS community now knew what *good data* looked like and recognized a need for high quality control. ***The lower energy resolution of lab XAFS data wasn’t considered trustworthy in the new synchrotron era.***

Mid-1980's

2013

The reasons:

1) **Detector problems**: Often impossible to remove harmonics in lab XAFS and solid-state detectors were still in their early stages in 1980's. Harmonic contamination kills data quality.

Silicon drift detectors with high saturation rates are a mass-produced commodity, easily able to reject harmonics and background scatter.

2) **Synchrotron access** (for a while) grew, throughput went up, many new EXAFS applications required the higher flux, XANES hadn't gained its present importance.

XAFS community outgrew access, 300% to 1000% oversubscription globally.

3) The XAFS community now knew what *good data* looked like and recognized a need for high quality control. ***The lower energy resolution of lab XAFS data wasn't considered trustworthy in the new synchrotron era.***

1-m spherically bent crystal analyzers are commercially available with all orientations. (and later with 0.5-m bend!)

Elimination of the Inner-Shell Lifetime Broadening in X-Ray-Absorption Spectroscopy

K. Hämäläinen,[a] D. P. Siddons, J. B. Hastings, and L. E. Berman
National Synchrotron Light Source, Brookhaven National Laboratory, Upton, New York 11973

A novel method to measure x-ray-absorption spectra without the inner-shell lifetime broadening is presented. It is based on a high-resolution spectrometer which can be used to analyze the fluorescence photon energy with better resolution than the natural linewidth. The dramatic improvement in resolution and detailed structure for the x-ray-absorption near-edge structure (XANES) at the Dy L_{III} edge is presented. This new technique reveals structure which is totally invisible using conventional XANES.

The spectrometer used to measure the fluorescence is based on a spherically bent perfect Si(440) crystal and a position-sensitive detector (PSD) sitting on the same Rowland circle as the sample. The fluorescence radiation

VOLUME 67, NUMBER 20 PHYSICAL REVIEW LETTERS 11 NOVEMBER 1991

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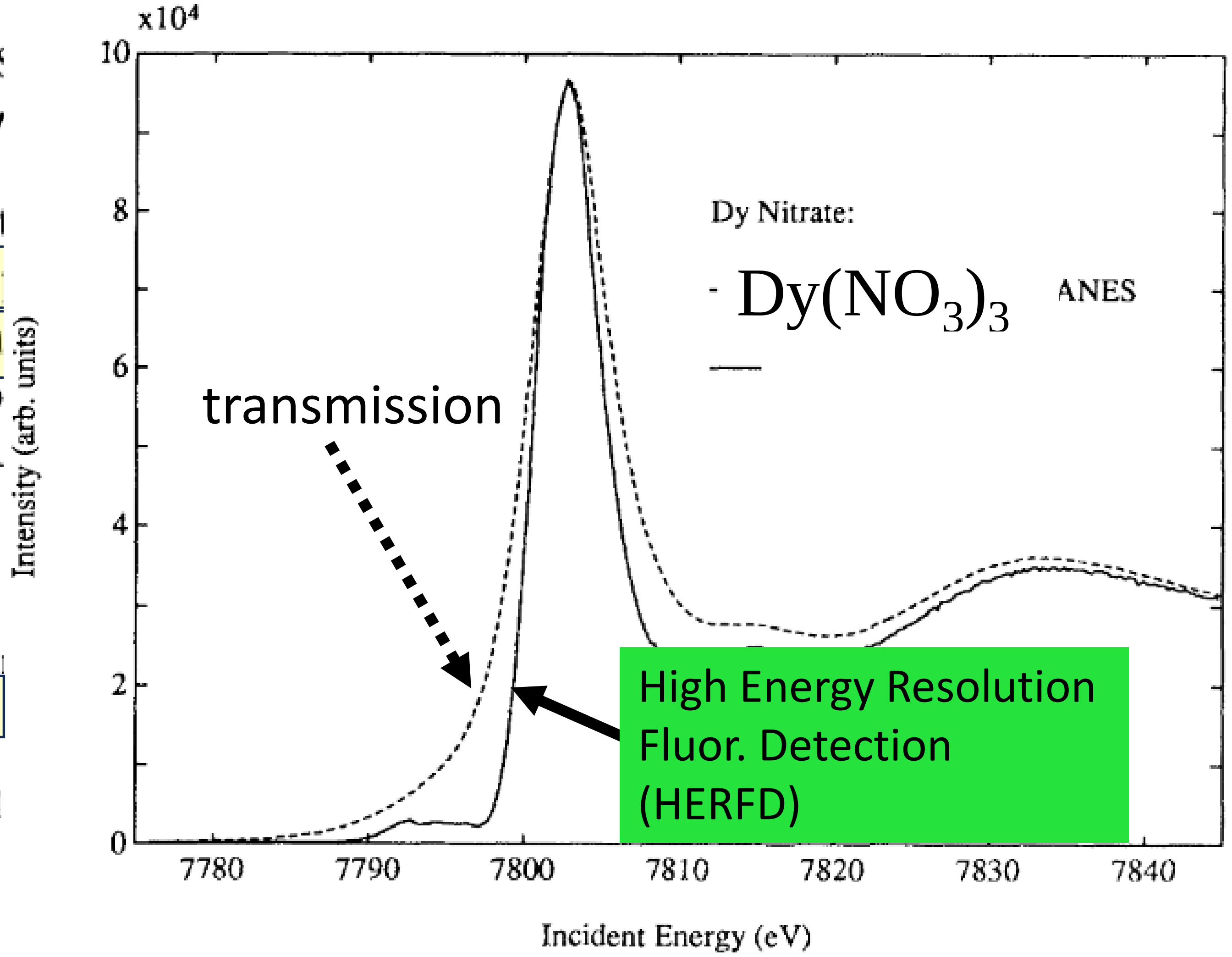

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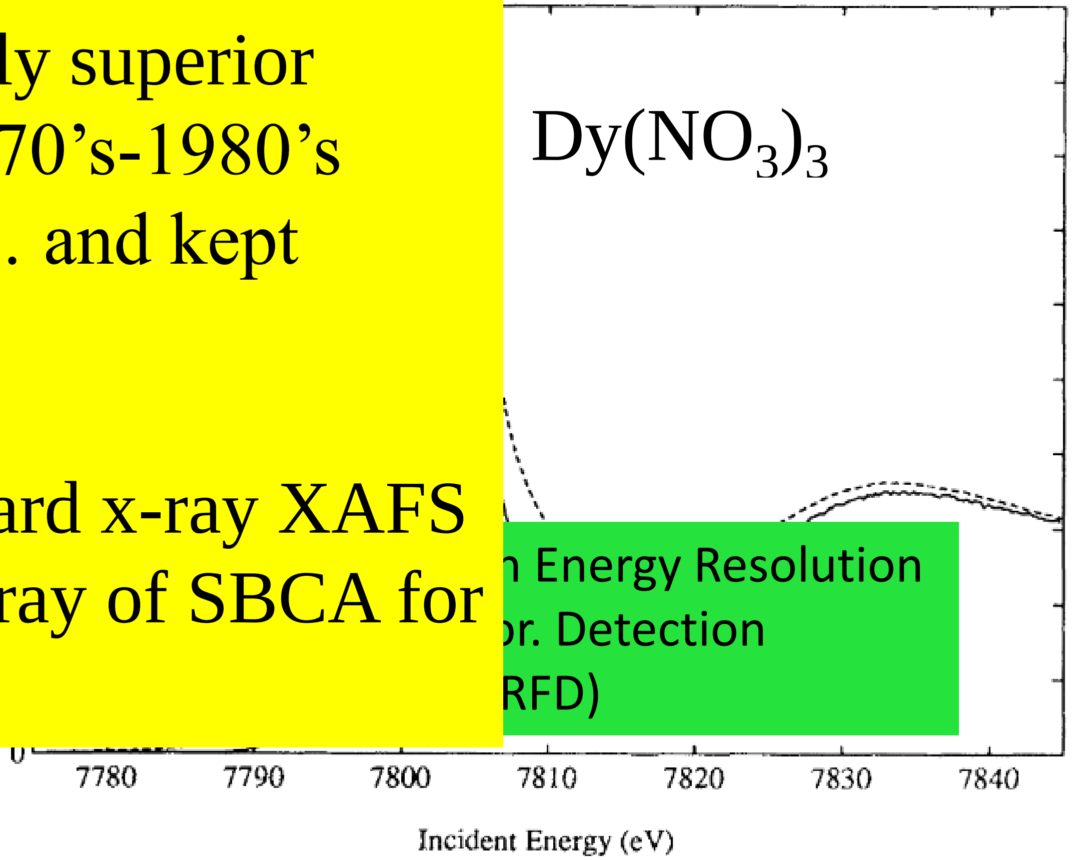
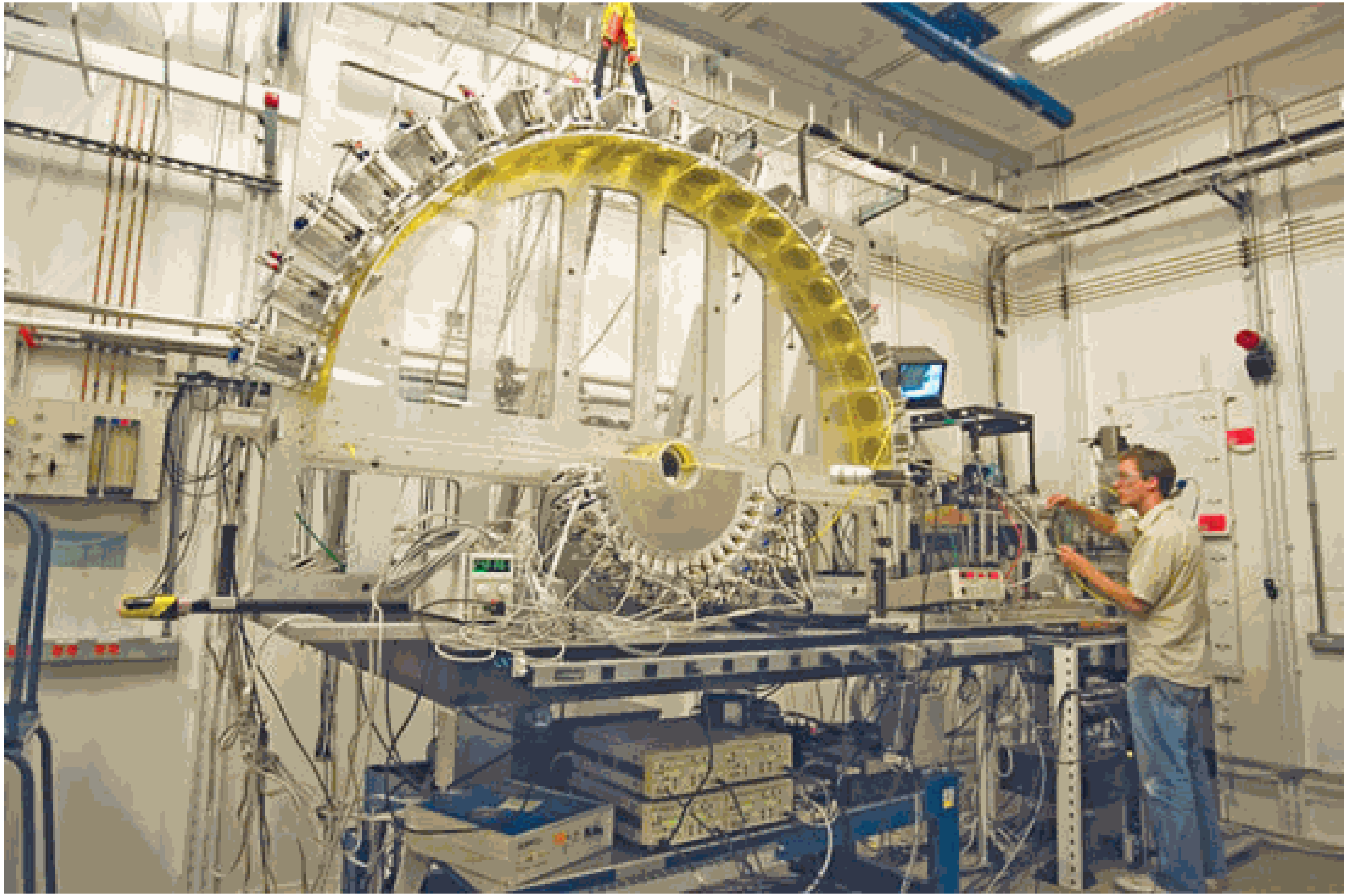
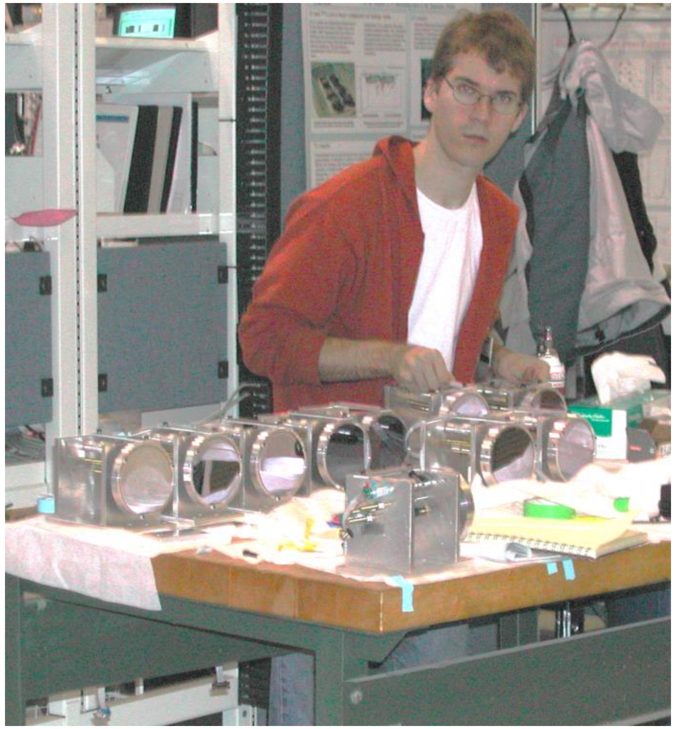
position-sensitive detector (P
Rowland circle as the sample.

Bottom line:
2010 SBCA were vastly superior optics compared to 1970's-1980's cylindrical analyzers… and kept improving.

Probably 50% of all hard x-ray XAFS endstations have an array of SBCA for XES/RIXS or XRS.



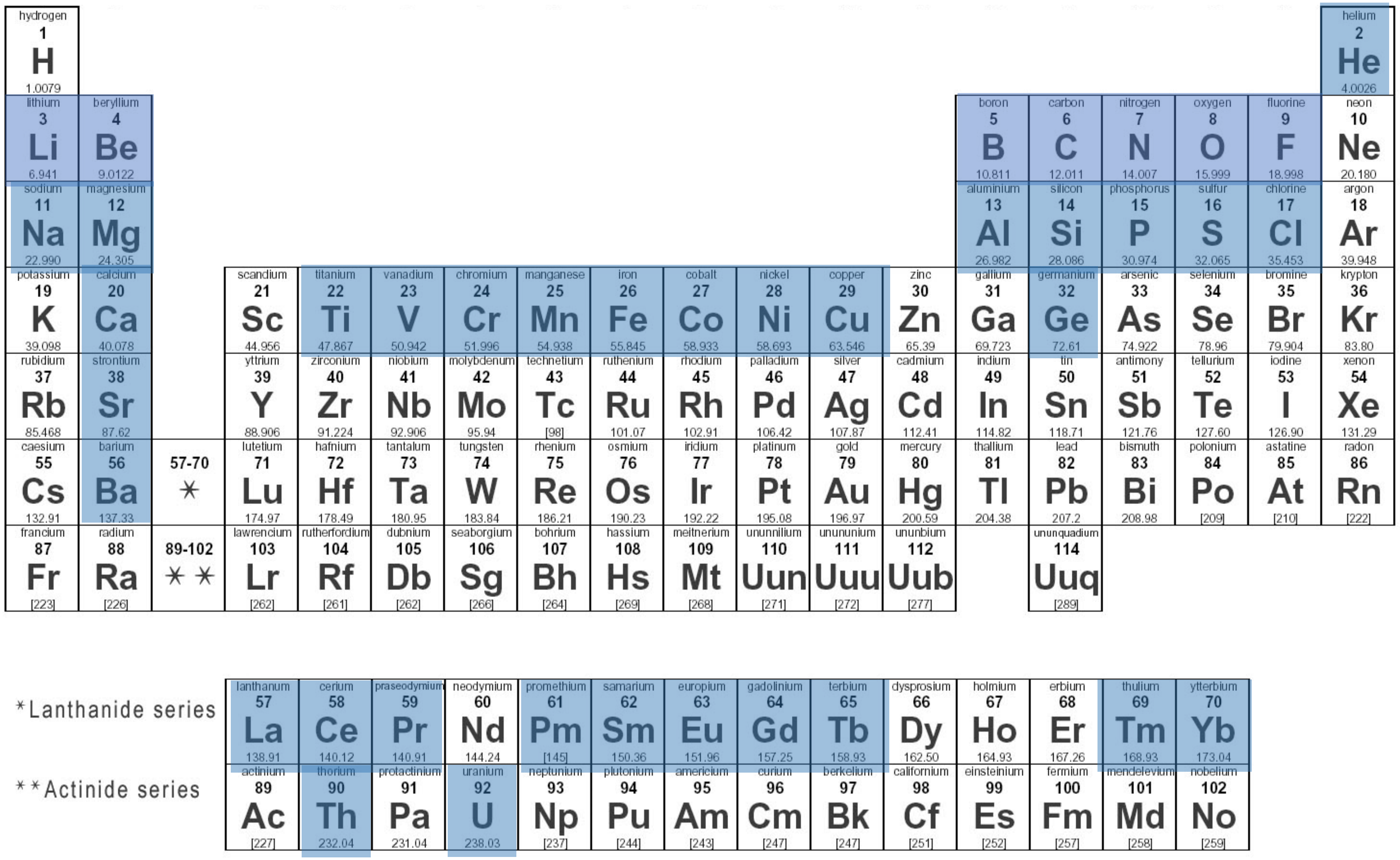




Tim Fister

LERIX spectrometer: Fister, et al., Rev. Sci. Instrum. (2006)

LERIX operations 2006-2022 (APS 20-ID)

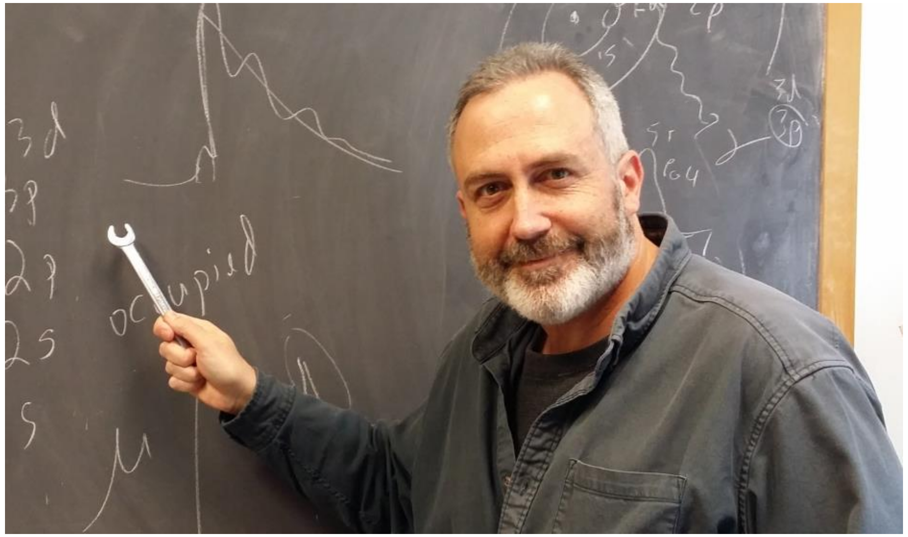


LERIX:

- >50 papers.
- Expanded XRS much farther in the periodic table.
- Made XRS accessible to a broad community of users.

August 2013: There was possible funding to make LERIX larger.

Steve Heald: “There isn’t enough money to both build the body of spectrometer + detector *and* to buy the ~100 optics.”



Jerry: “Well, maybe we can learn to make the optics.”

“But we need to make a testing facility at UW that we can use every day.”

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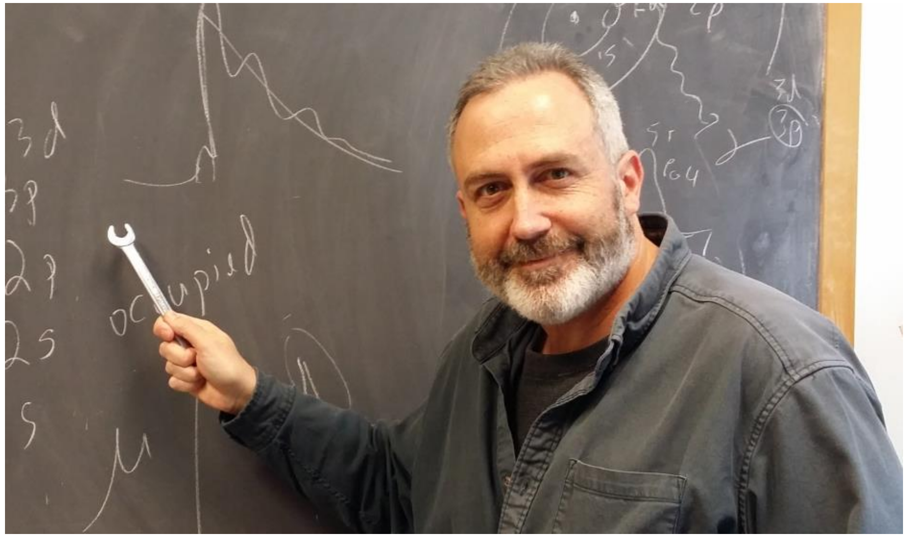

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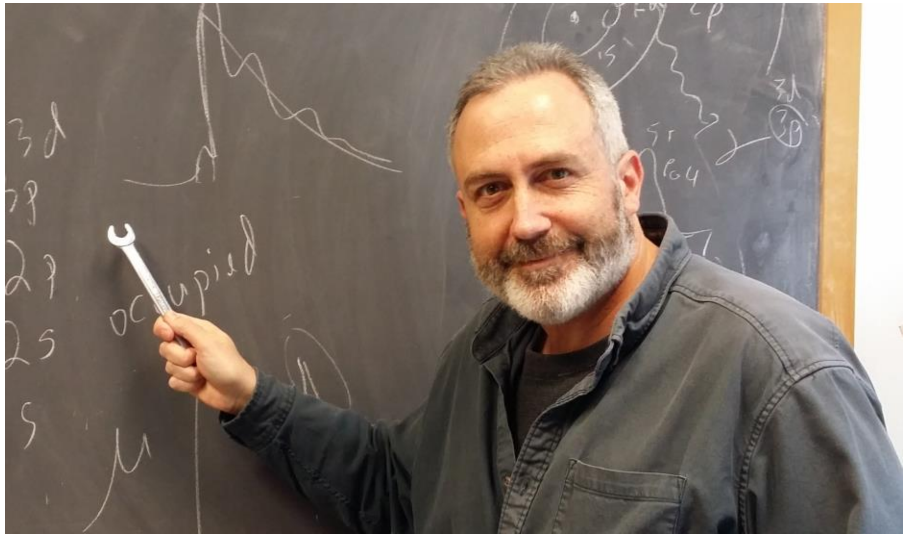
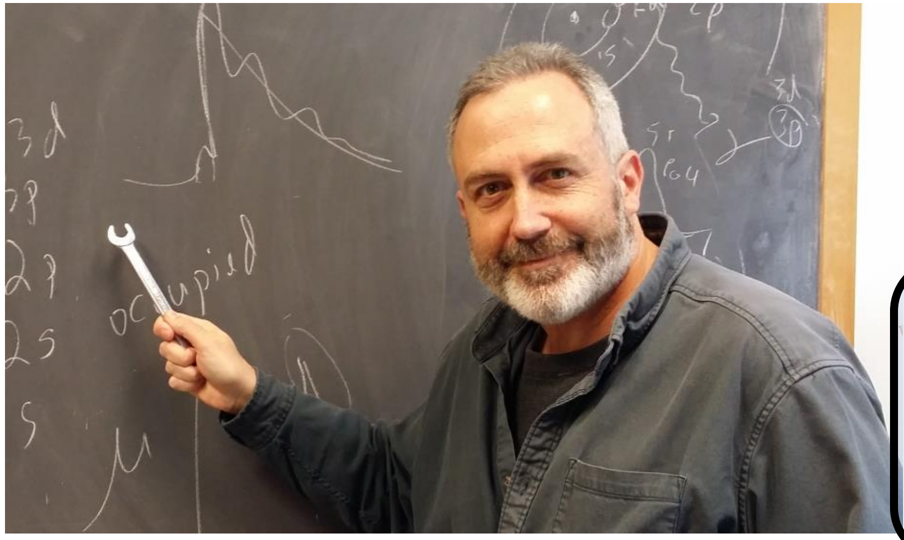


Jerry: “Well, maybe we can learn to make the optics.”

“But we need to make a testing facility at UW that we can use every day.”

August 2013: There was possible funding to make LERIX larger.

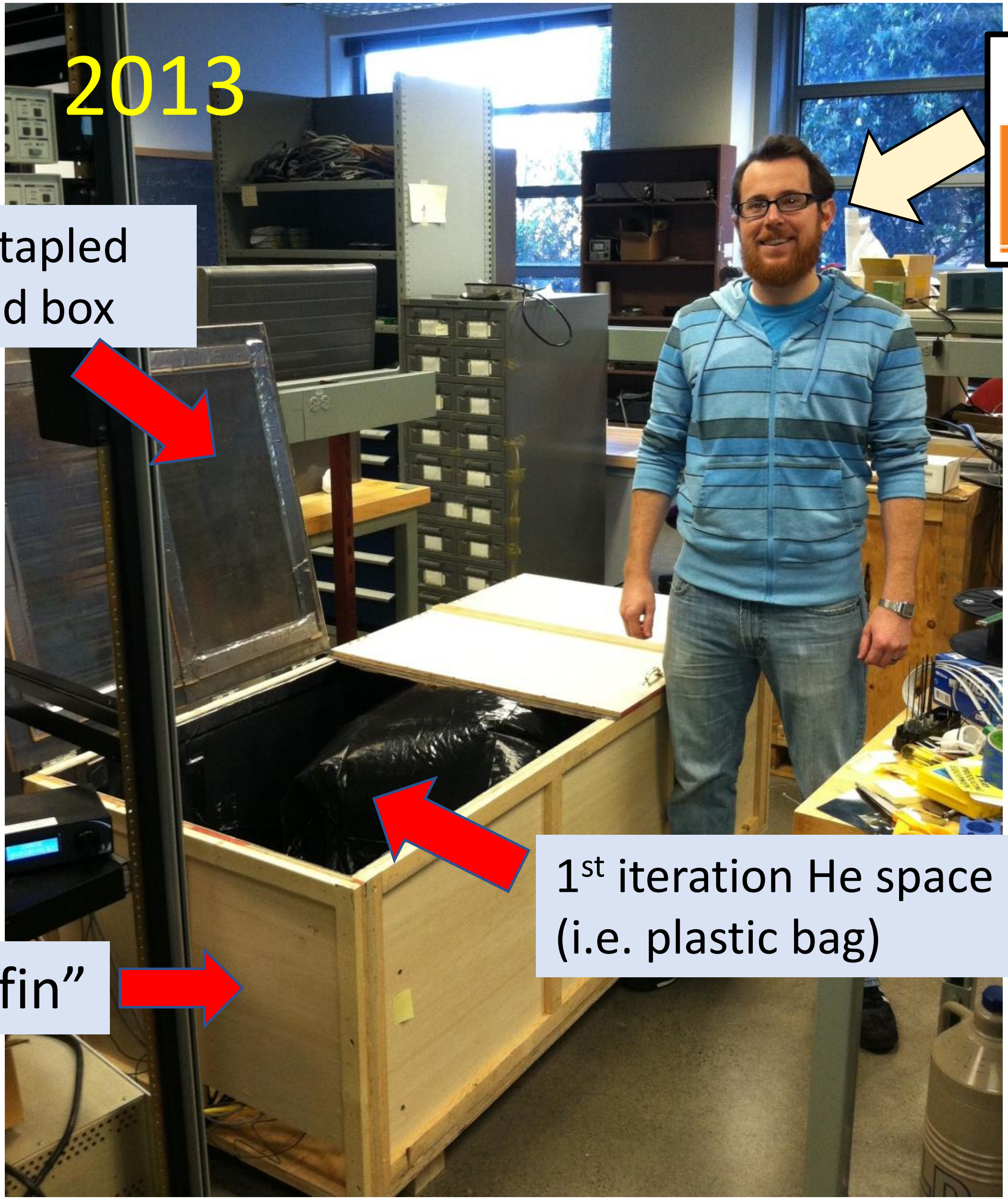
Back-of-envelope calculation showed:
1) useful monochromatic flux for XAFS even low powered x-ray tube.
2) Lab XES should would have count rates intermediate between BM and 3rd-generation ID.
3) We could build a prototype for 30k$ in components, and I had the funds.



Jerry: “Well, maybe we can learn to make the optics

“But we need to make a testing facility at UW that we can use every day.”

The “coffin” prototype spectrometer at UW



Devon Mortensen

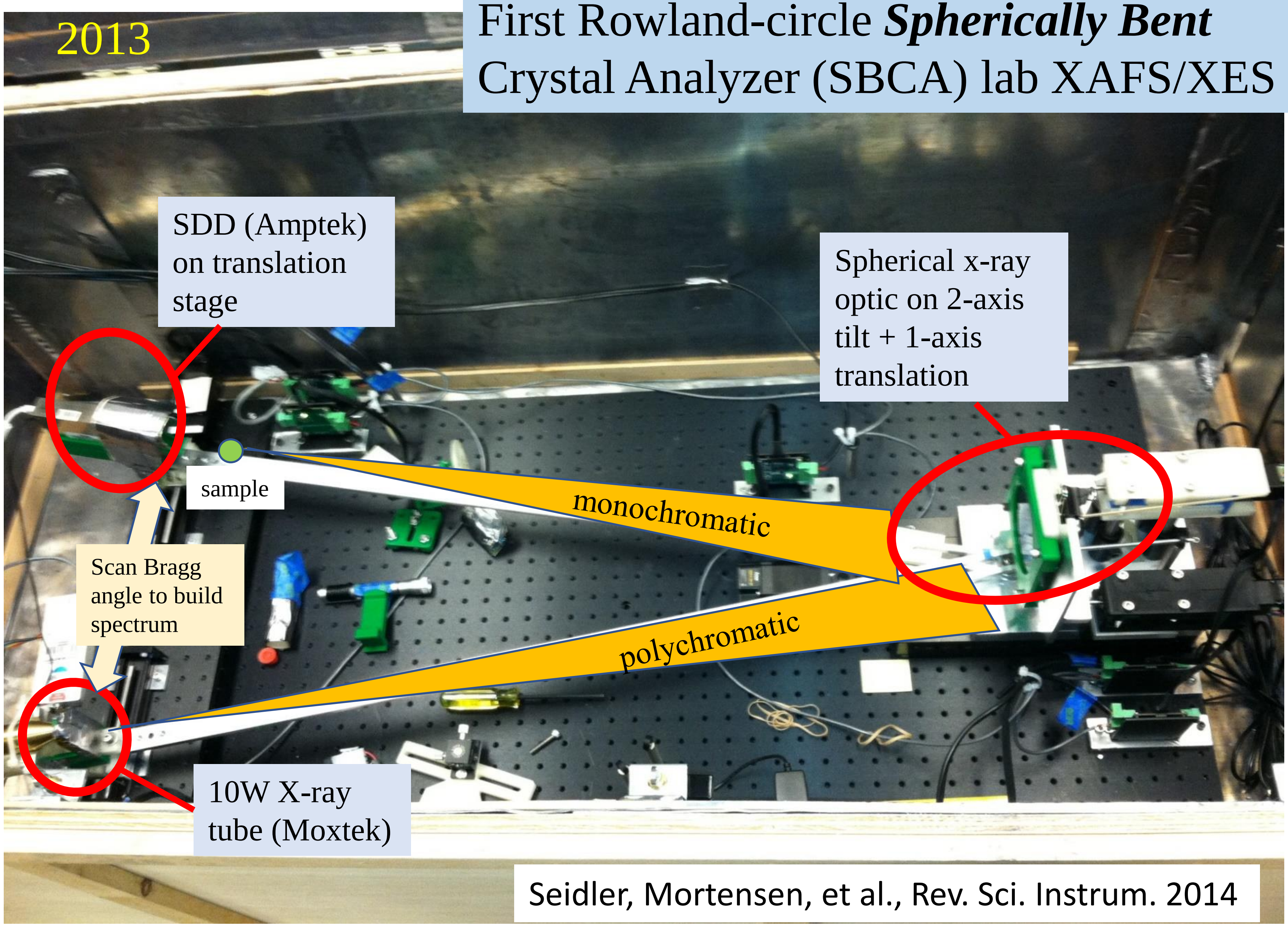
easyXAFS

Lead sheet stapled onto plywood box

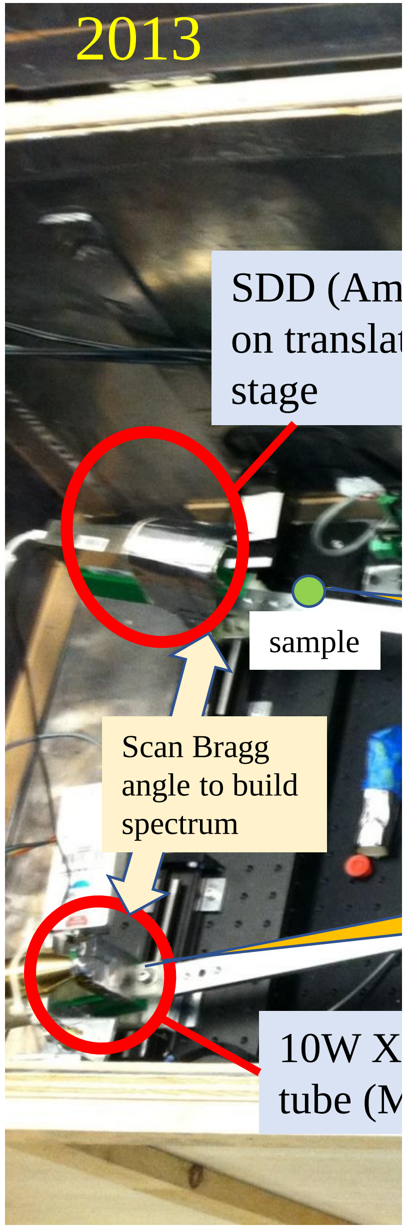
1^{st} iteration He space (i.e. plastic bag)

“X-ray coffin”

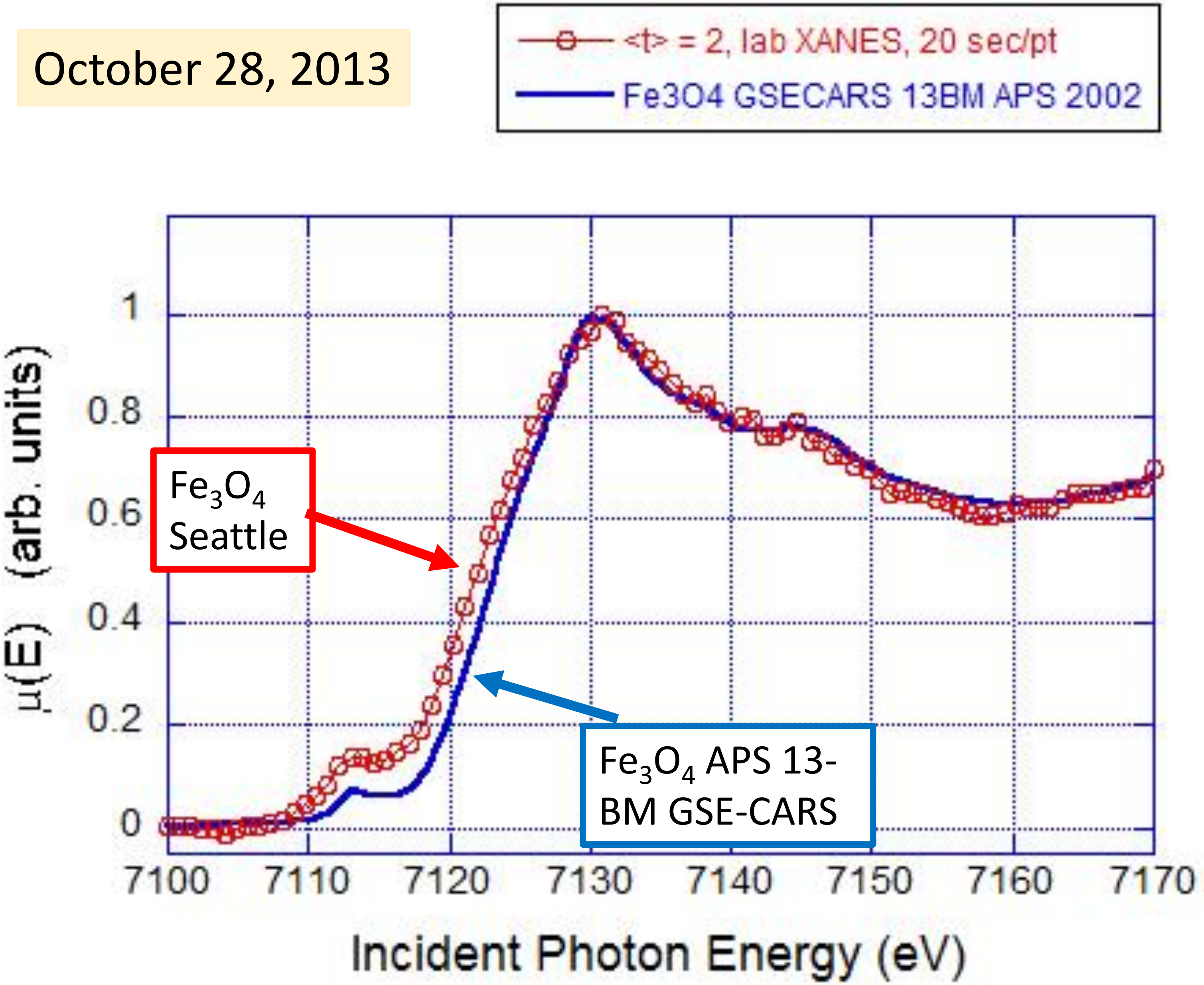
First Rowland-circle ***Spherically Bent*** Crystal Analyzer (SBCA) lab XAFS/XES



Seidler, Mortensen, et al., Rev. Sci. Instrum. 2014

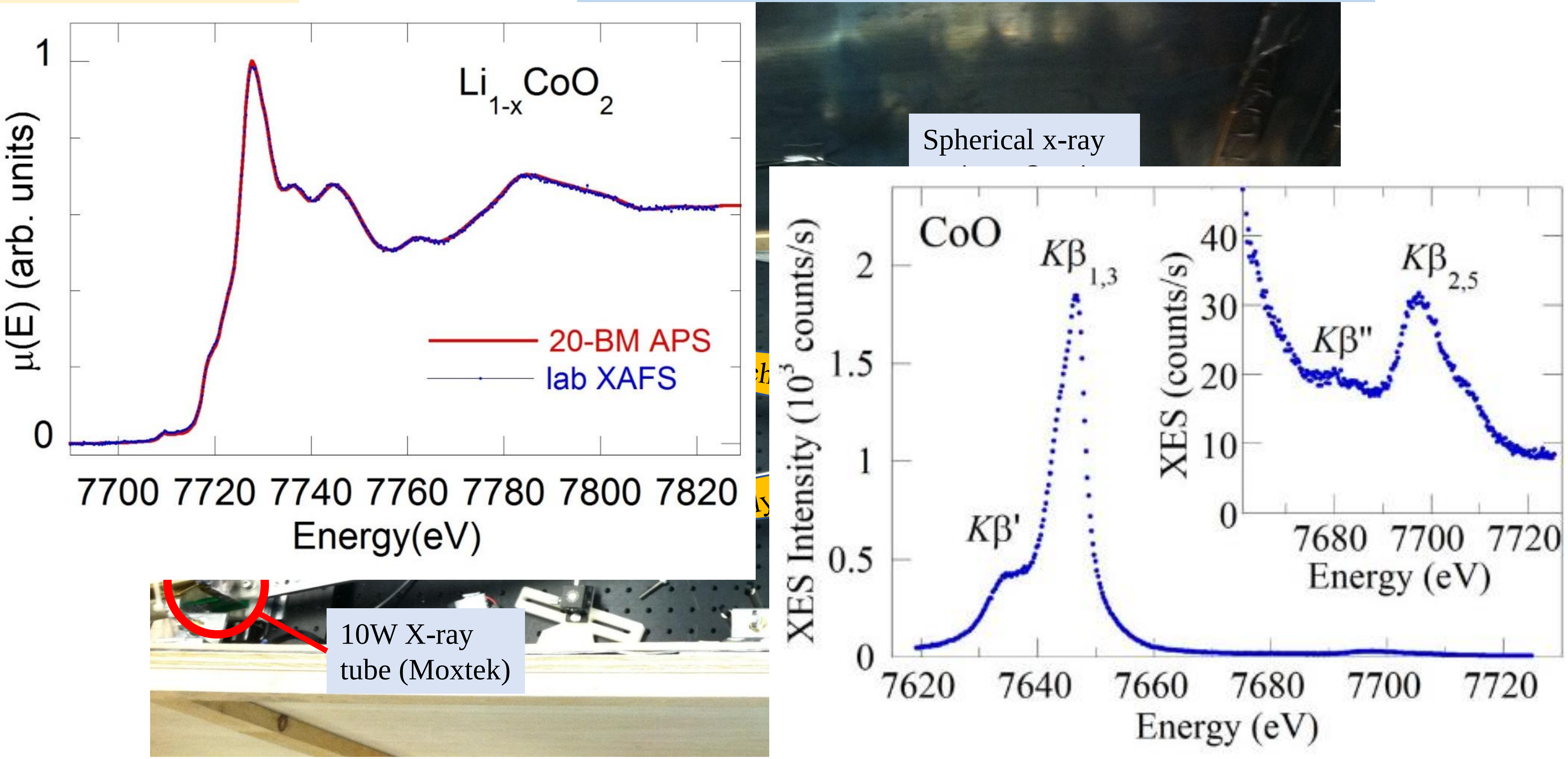


October 28, 2013



A few months later…

First Rowland-circle ***Spherically Bent*** Crystal Analyzer (SBCA) lab XAFS/XES



Seidler, Mortensen, et al., Rev. Sci. Instrum. 2014

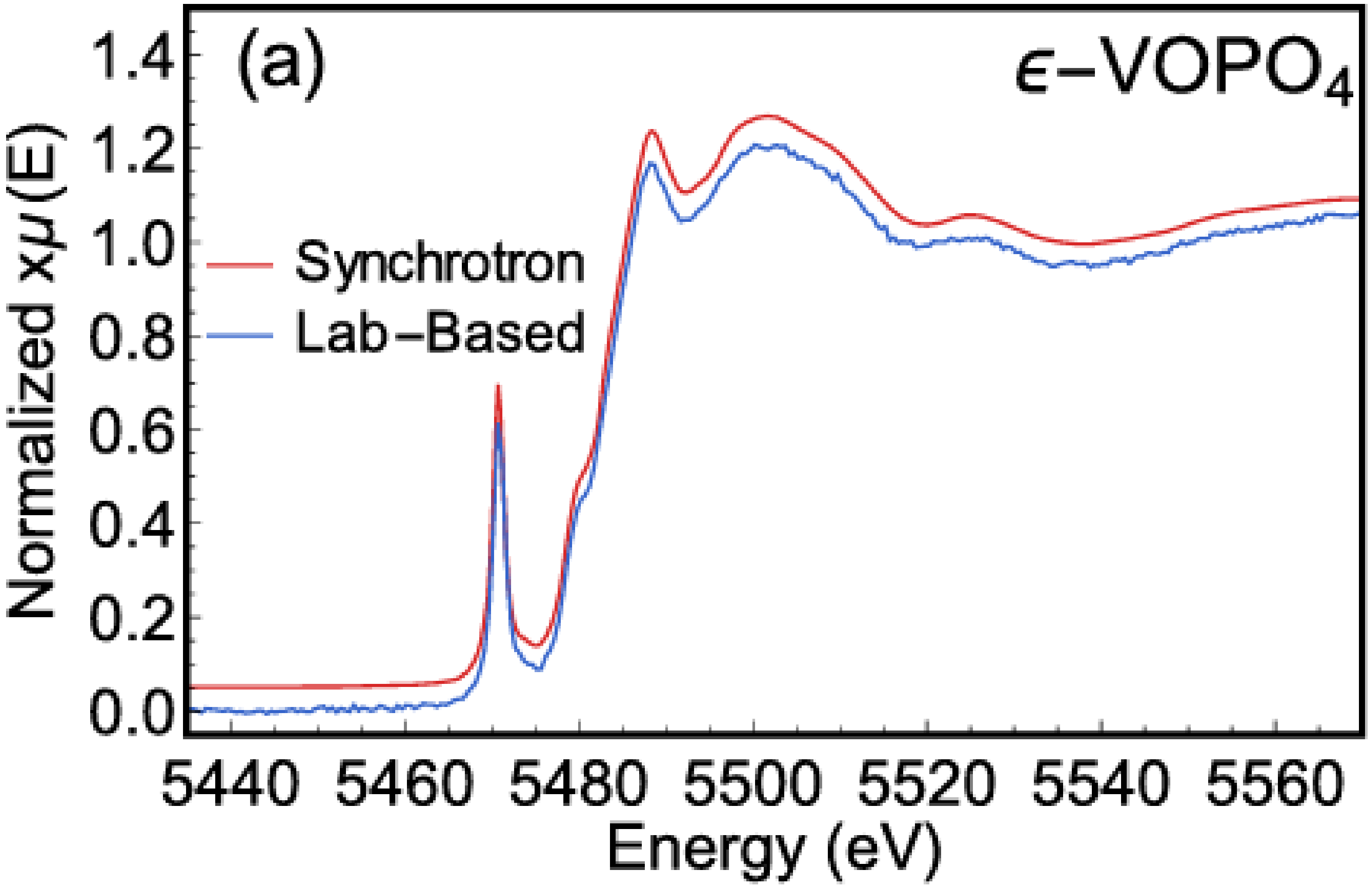
Seidler, Mortensen, et al., Rev. Sci. Instrum. 2014
Holden, et al., Rev. Sci. Instrum. 2017
Jahrman, et al., Rev Sci. Instrum. 2019
TU-Berlin (Kangiessen, Malzer)
Budapest (Nemeth, Vanko)
Warsaw/Krakow (Szlachetko)
Helsinki (Huotari, Bes)
NIST (O'Neil, Ullom)

(commercialization)

Hypothesis:
The world needs lab XAFS and XES again, *but for more reasons than in the pre-synchrotron era* **and** *for reasons that are often incompatible with synchrotron access.*

2017 Many upgrades of prototype, workhorse lab XAFS still running, >30 papers.
E. Jahrman, et al., Rev. Sci. Instrum. (2019)

Many examples of perfect transmission-mode XANES



Evan Jahrman

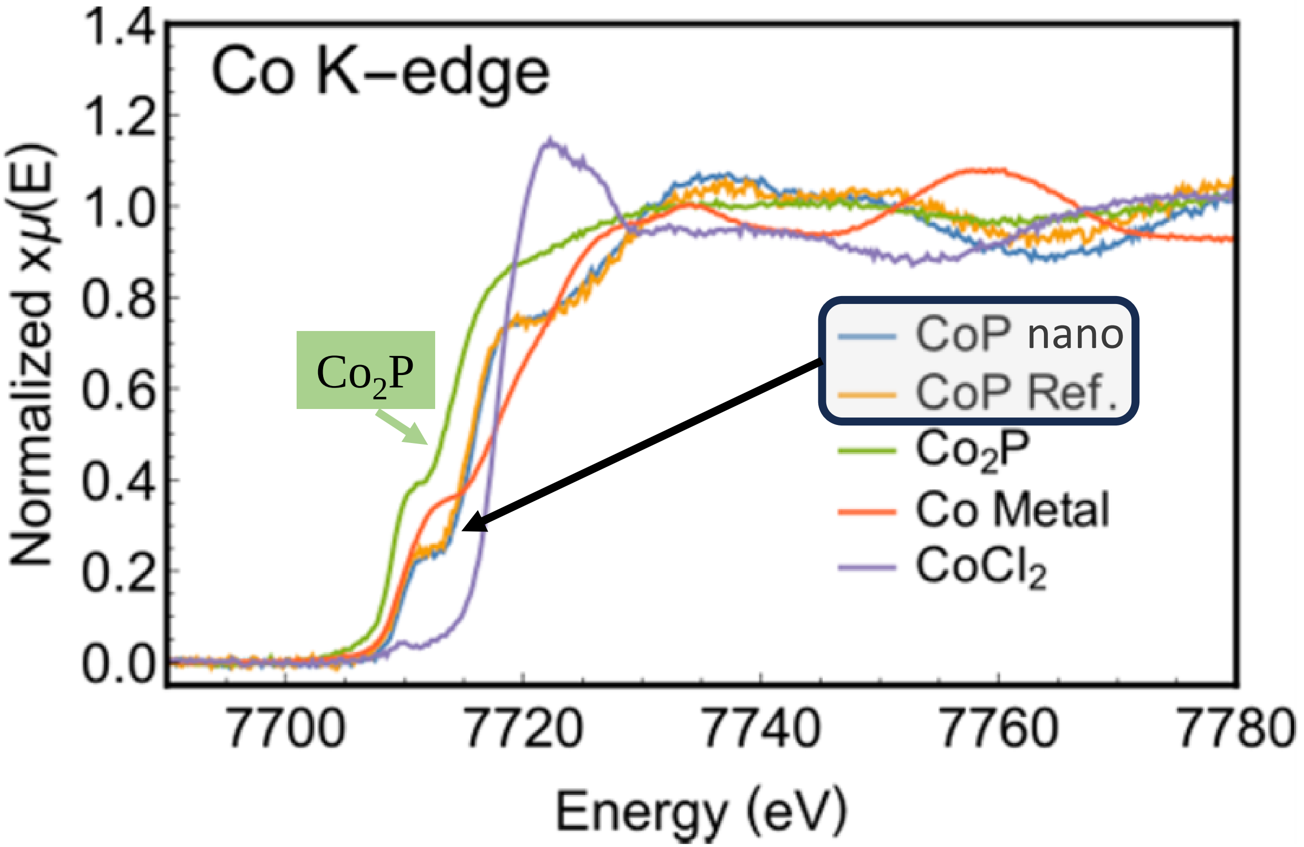
CHEMISTRY OF MATERIALS
Article
Cite This: *Chem. Mater.* 2018, 30, 5373–5379
pubs.acs.org/cm

Aminophosphines as Versatile Precursors for the Synthesis of Metal Phosphide Nanocrystals

M. Elizabeth Mundy,† David Ung,† Nathan L. Lai,† Evan P. Jahrman,‡ Gerald T. Seidler,‡ and Brandi M. Cossairt*,†

XRD can't distinguish CoP and Co_2P nanoparticles (5 nm). But the XANES is clear!

Purely analytical, non-expert application.



E. Jahrman, et al., Rev. Sci. Instrum. (2019)

First favorable demonstration of modern lab EXAFS.

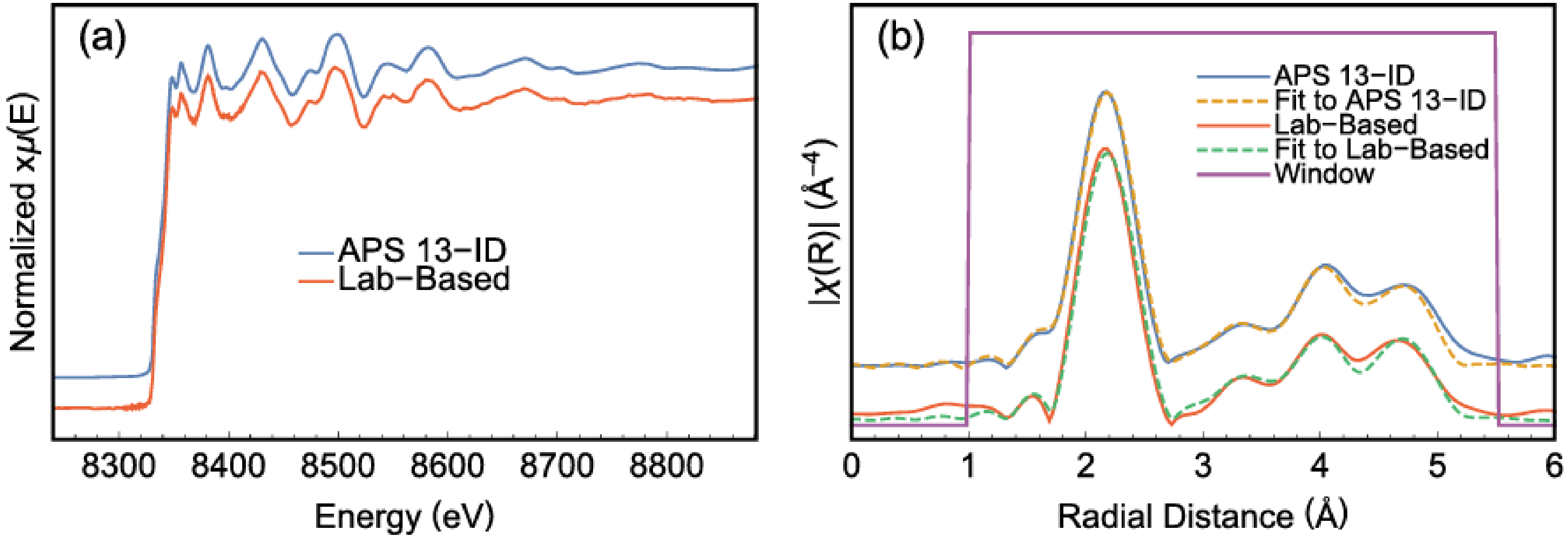
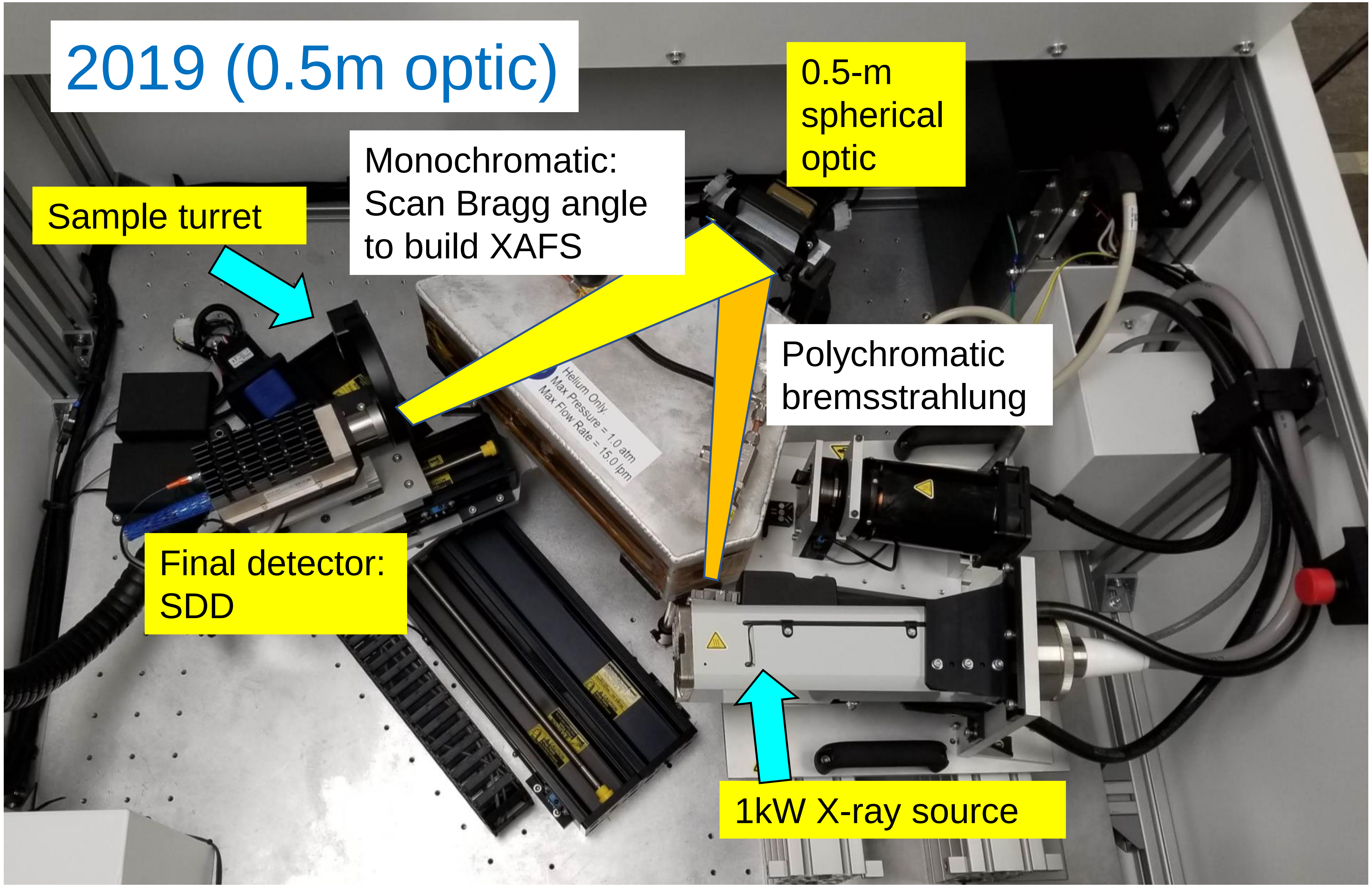


TABLE II. Selected EXAFS fitting parameters for the Ni foil measured at APS and at UW

			Shell1		Shell2		Shell3		Shell4	
	S_0^2	R-factor	Ni-Ni (Å)	σ^2 (10^{-4} Å^2)	Ni-Ni (Å)	σ^2 (10^{-4} Å^2)	Ni-Ni (Å)	σ^2 (10^{-4} Å^2)	Ni-Ni (Å)	σ^2 (10^{-4} Å^2)
APS 13-ID[47]	0.90 (6)	0.015	2.493 (4)	67 ± 6	3.525 (5)	96 ± 19	4.317 (6)	91 ± 10	4.985 (7)	79 ± 8
UW	0.81 (6)	0.016	2.490 (4)	61 ± 6	3.522 (5)	76 ± 16	4.314 (7)	92 ± 11	4.981 (8)	79 ± 9

*easy*XAFS spectrometers globally!

Linear Spectrometer (1-3 kW tube): enables *in operando* studies and some fluorescence mode.



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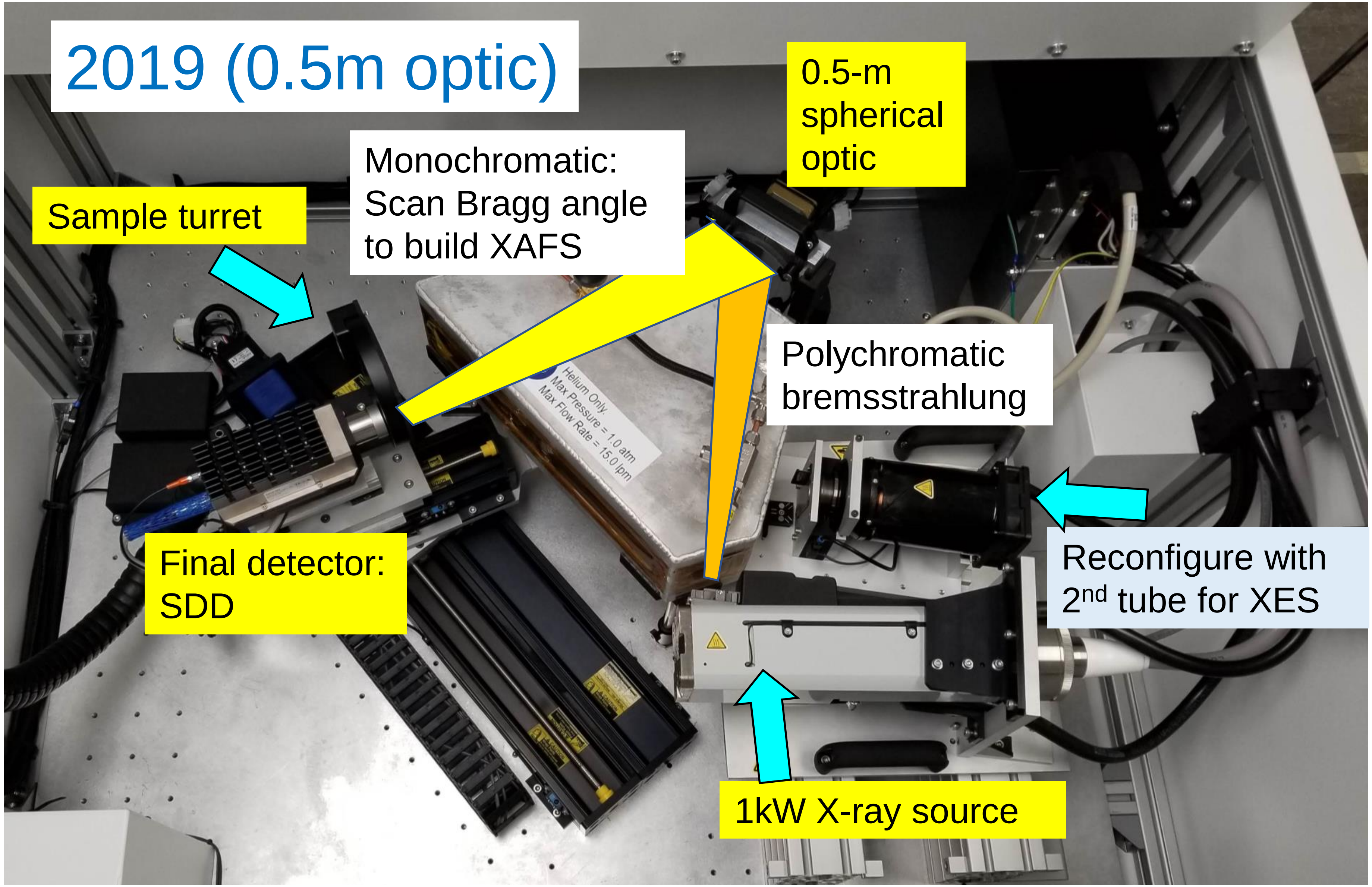
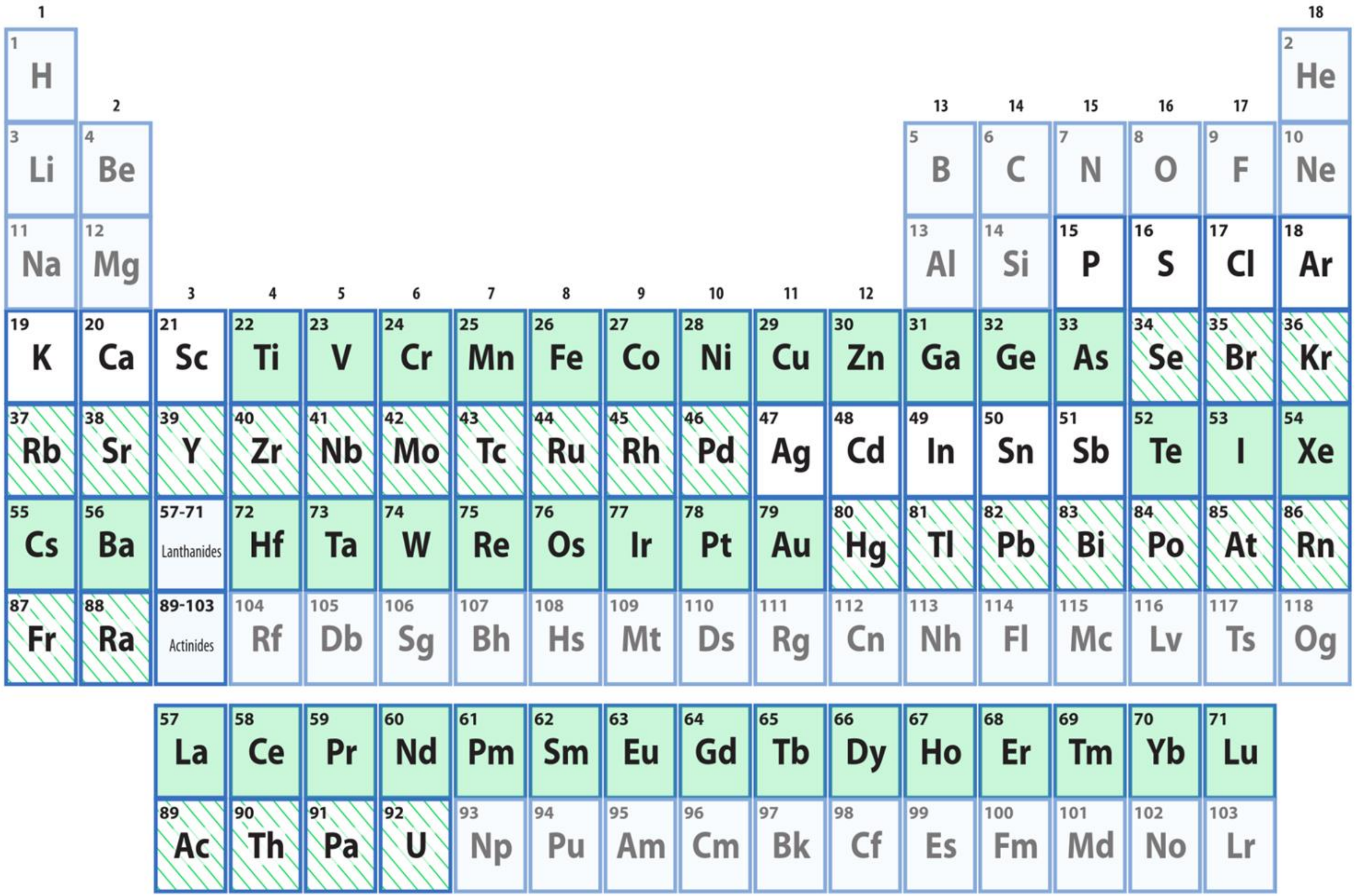


Accessible elements with high-powered line focus tube and 0.5-m SBCA

"Optimal" energy range: 4.5-12 keV

"Expanded" energy range: 12-25 keV



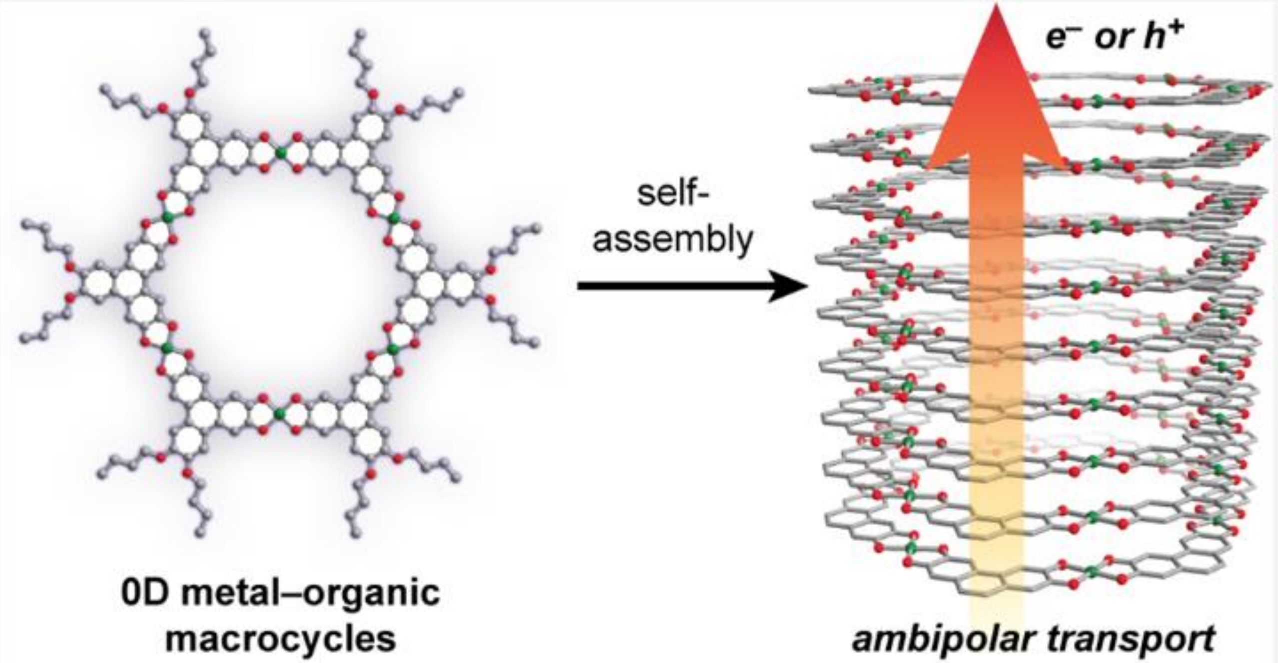
easyXAFS

JACS
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
pubs.acs.org/JACS
Article

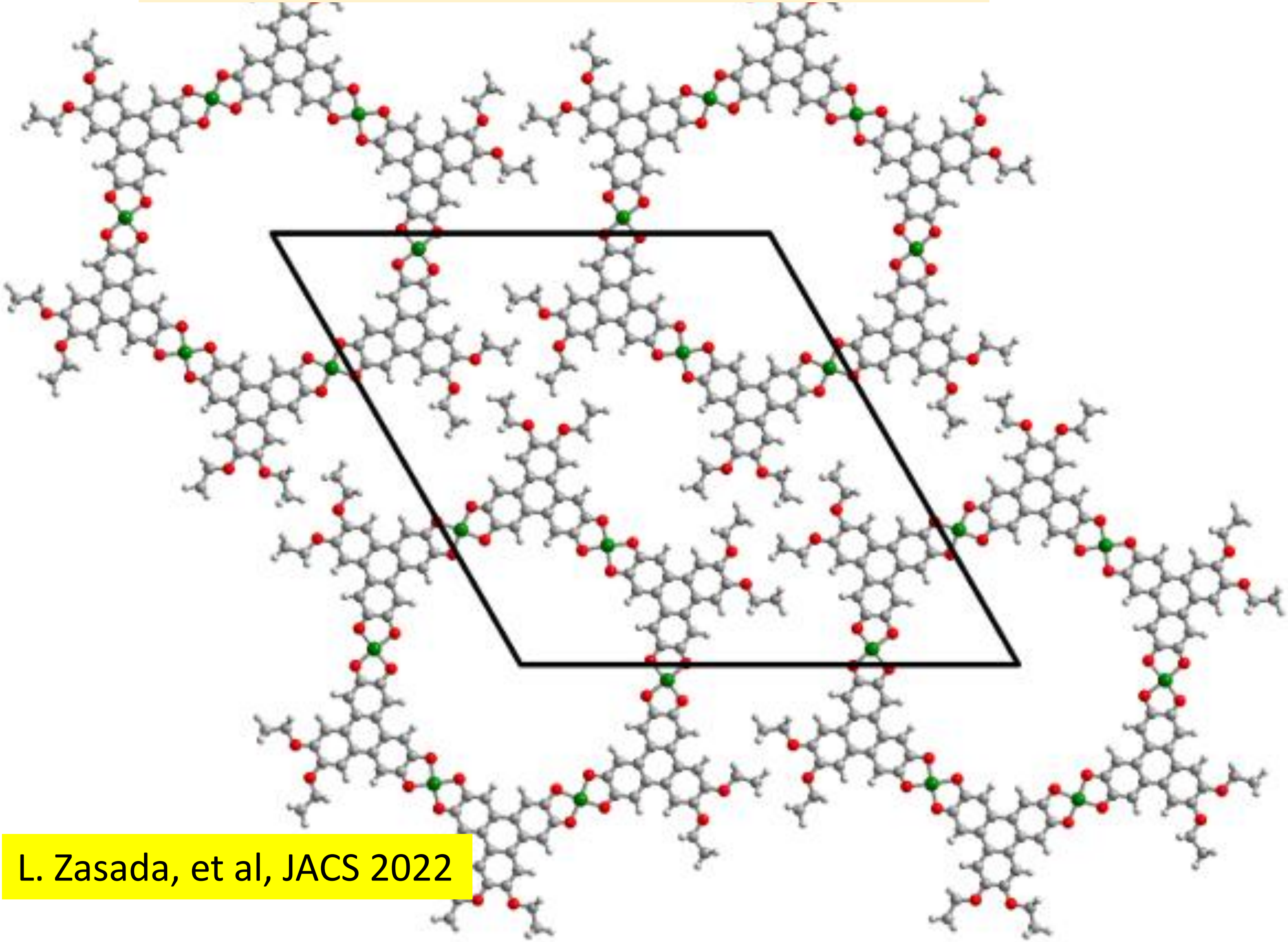
Conjugated Metal–Organic Macrocycles: Synthesis, Characterization, and Electrical Conductivity

Leo B. Zasada, Lorenzo Guio, Ashlyn A. Kamin, Diwash Dhakal, Madison Monahan, Gerald T. Seidler, Christine K. Luscombe, and Dianne J. Xiao*

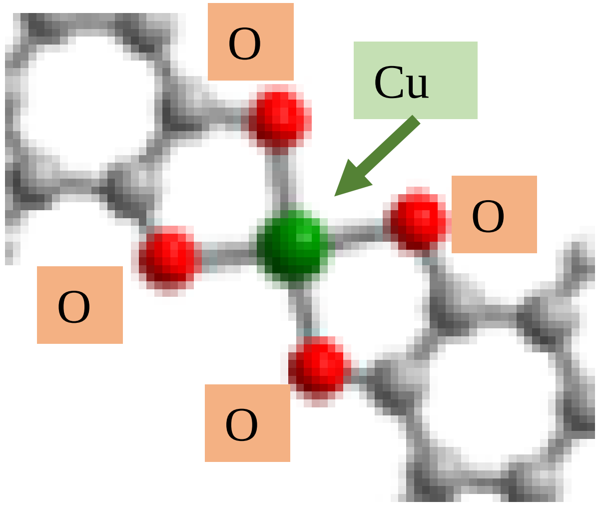
Cite This: J. Am. Chem. Soc. 2022, 144, 4515–4521
Read Online



XRD (based on some chemical intuition)



Hypothesis:
Cu(II) in square planar configuration with four O ligands.



L. Zasada, et al, JACS 2022

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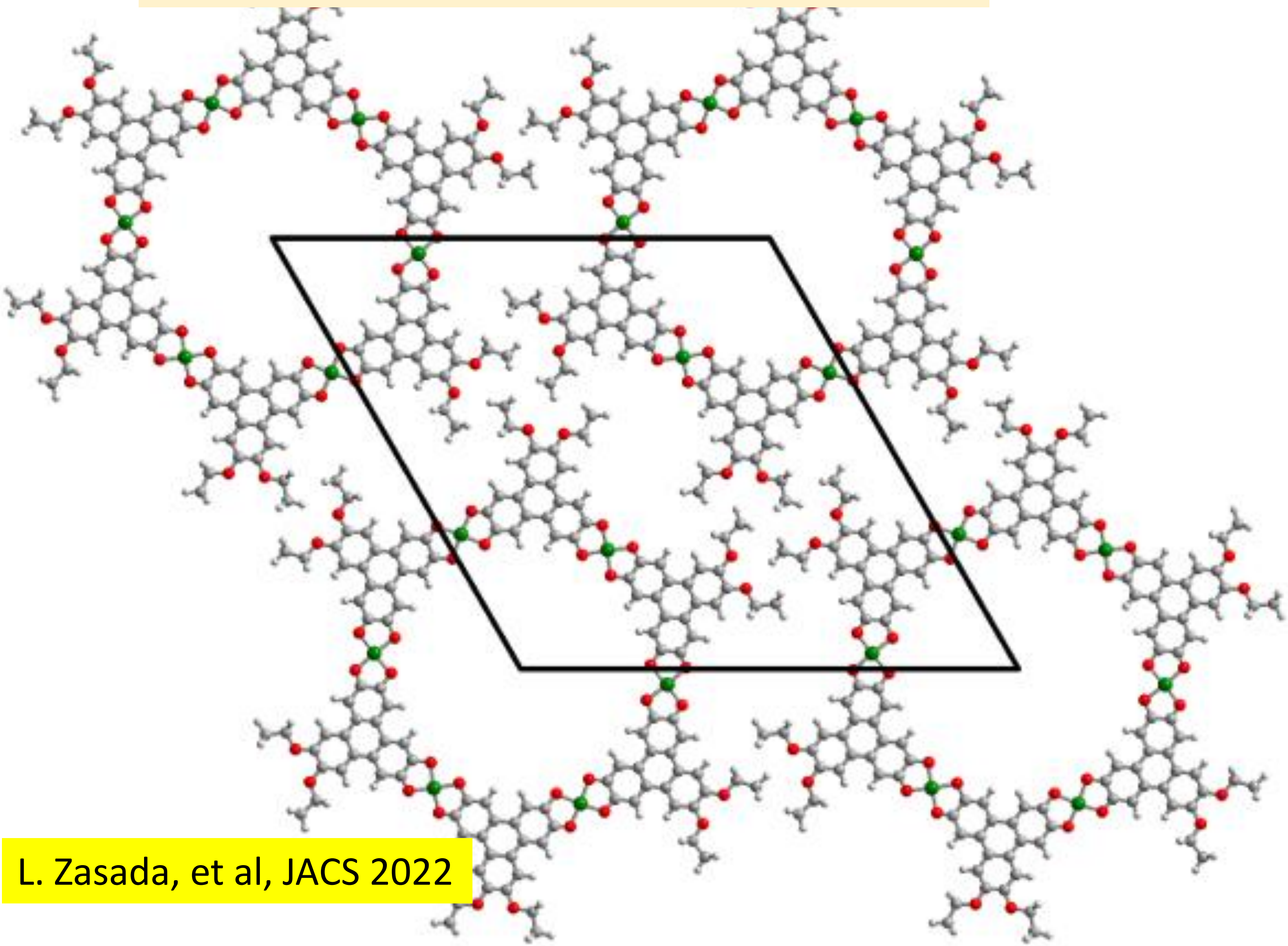

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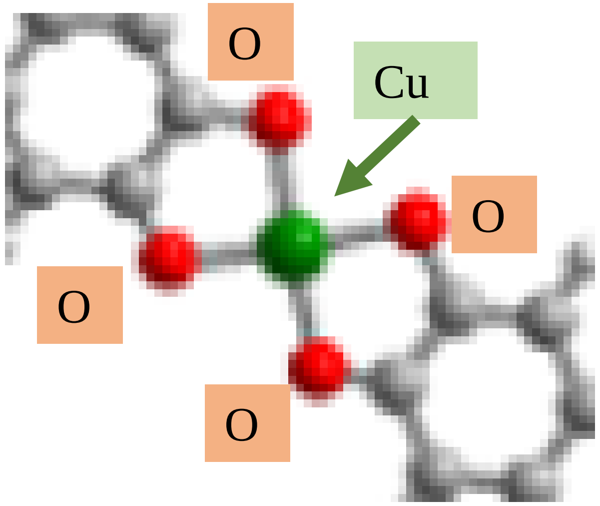
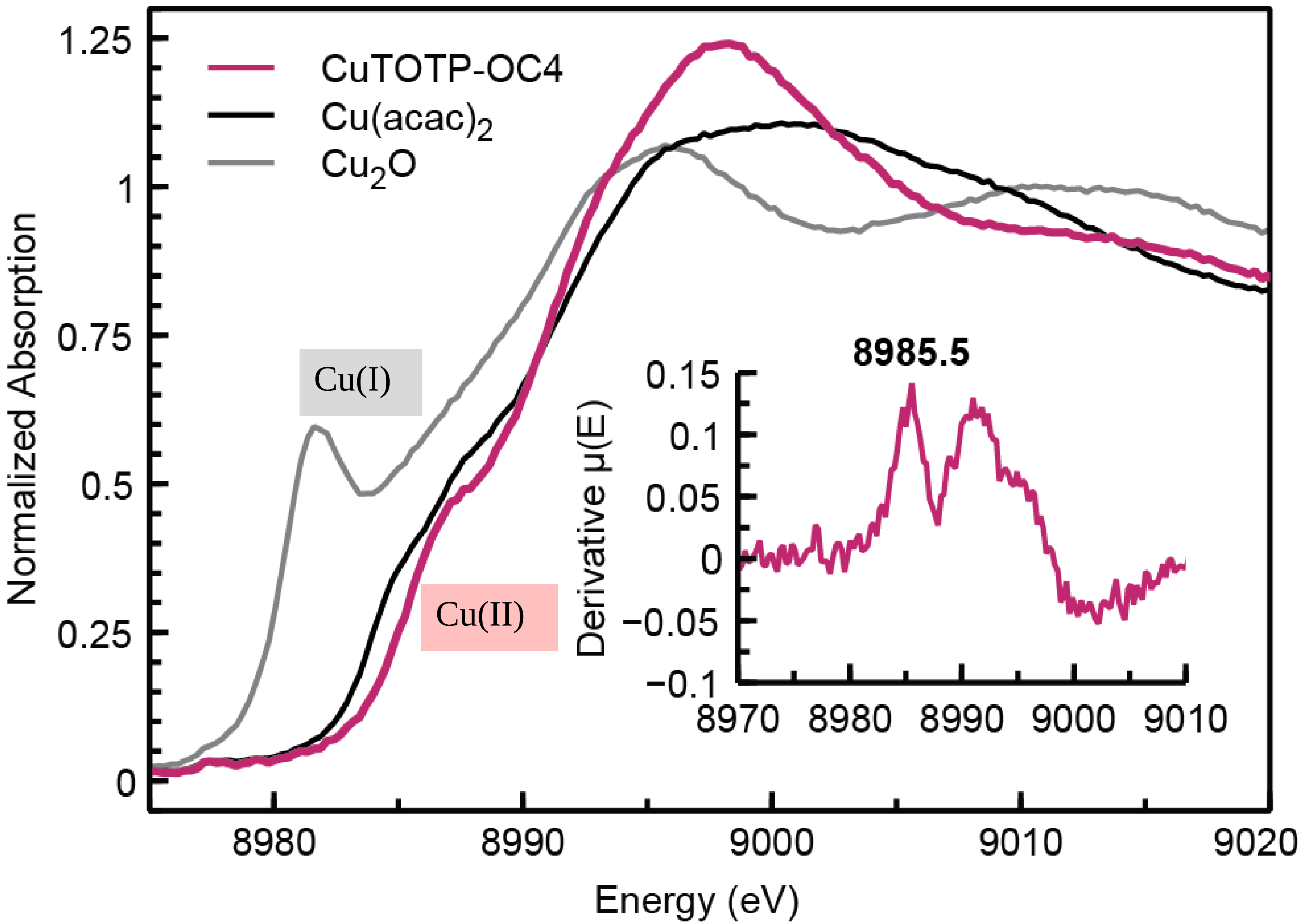


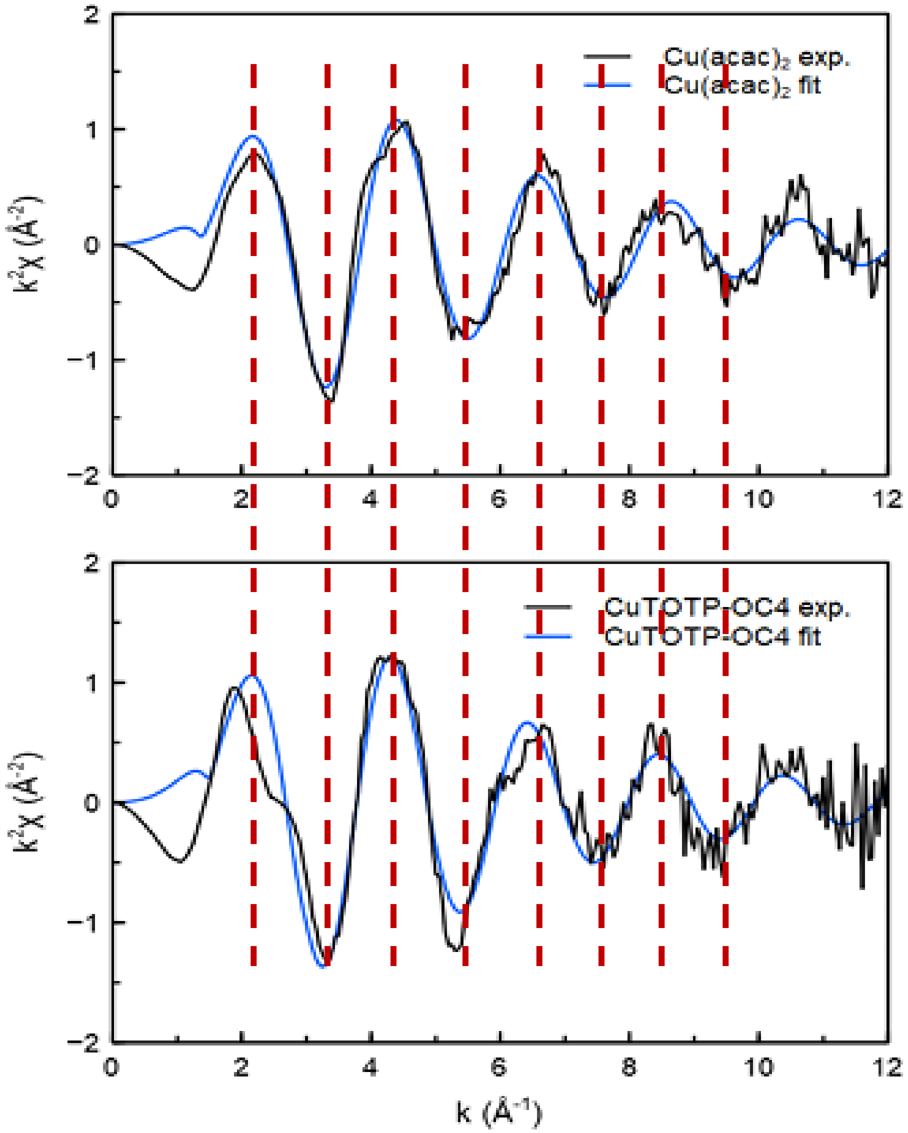
Compare to: $Cu(acac)_2$ is Cu(II) with square planar coordination to four O.

Zasada, et al, JACS 2022



Oxidation state is correct (edge shift)

Zasada, et al, JACS 2022

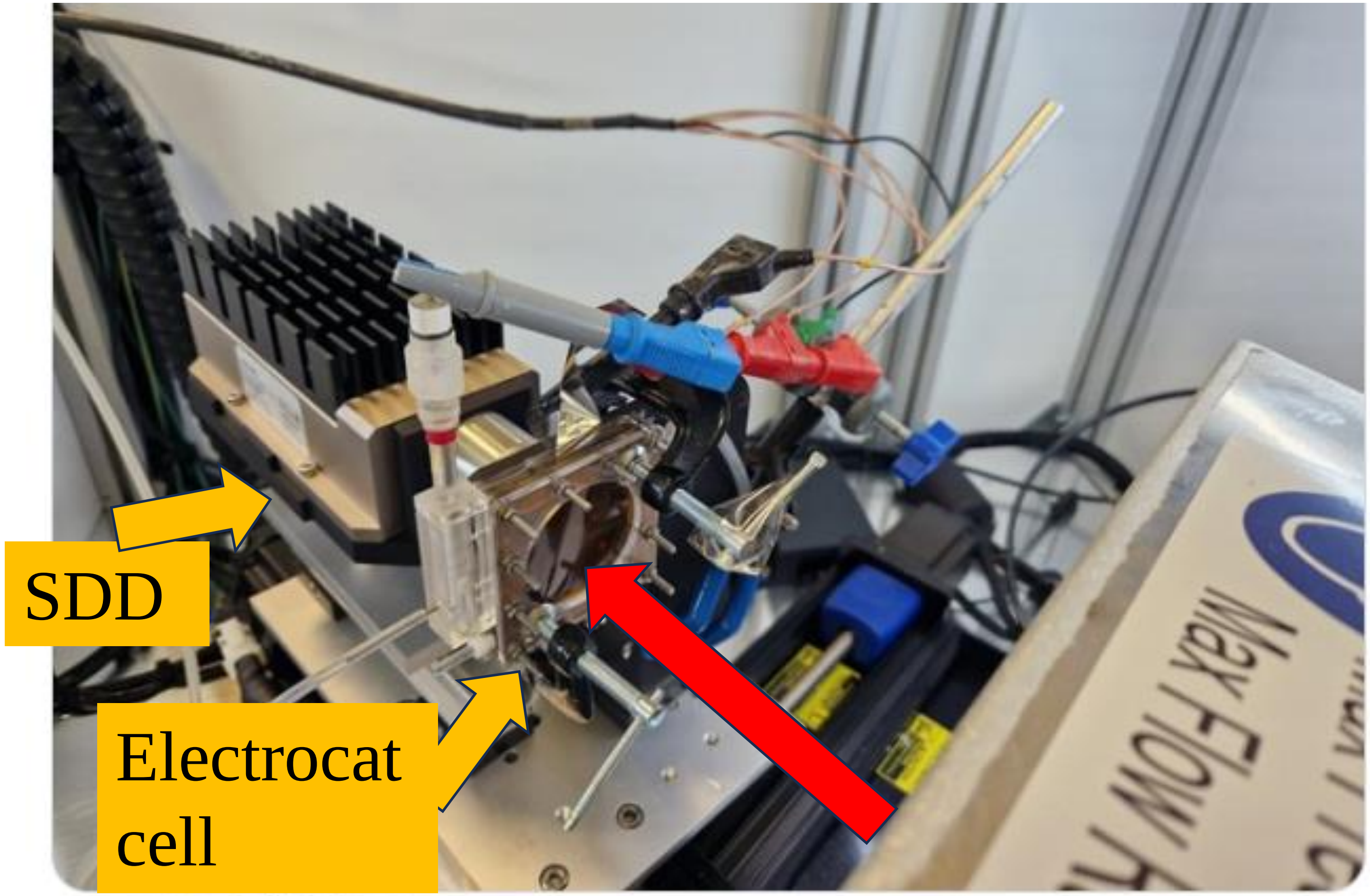


Fits give very similar Cu-O distance and first-shell N.

Cu (II) in a square planar configuration with oxygen ligands.

Harwell XPS
@HarwellXPS

Setting up our insitu electrocatalytic XAS measurements on our @easyXAFS spectrometer. @gsl1996 @MarkIsaacsChem bringing in some great data to go with our insitu XPS and Raman data!!

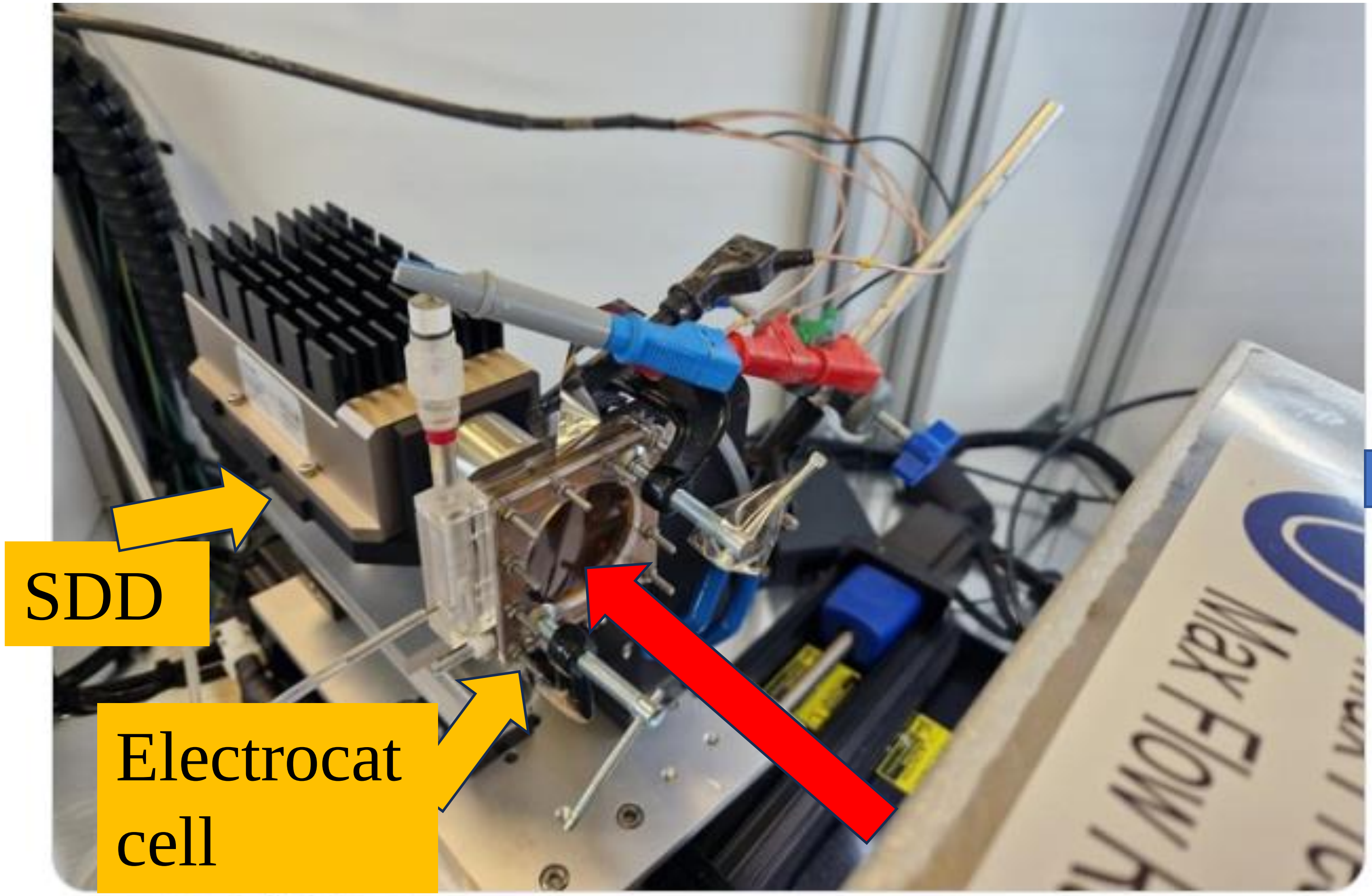


7:40 AM · Mar 16, 2023 · **3,608** Views

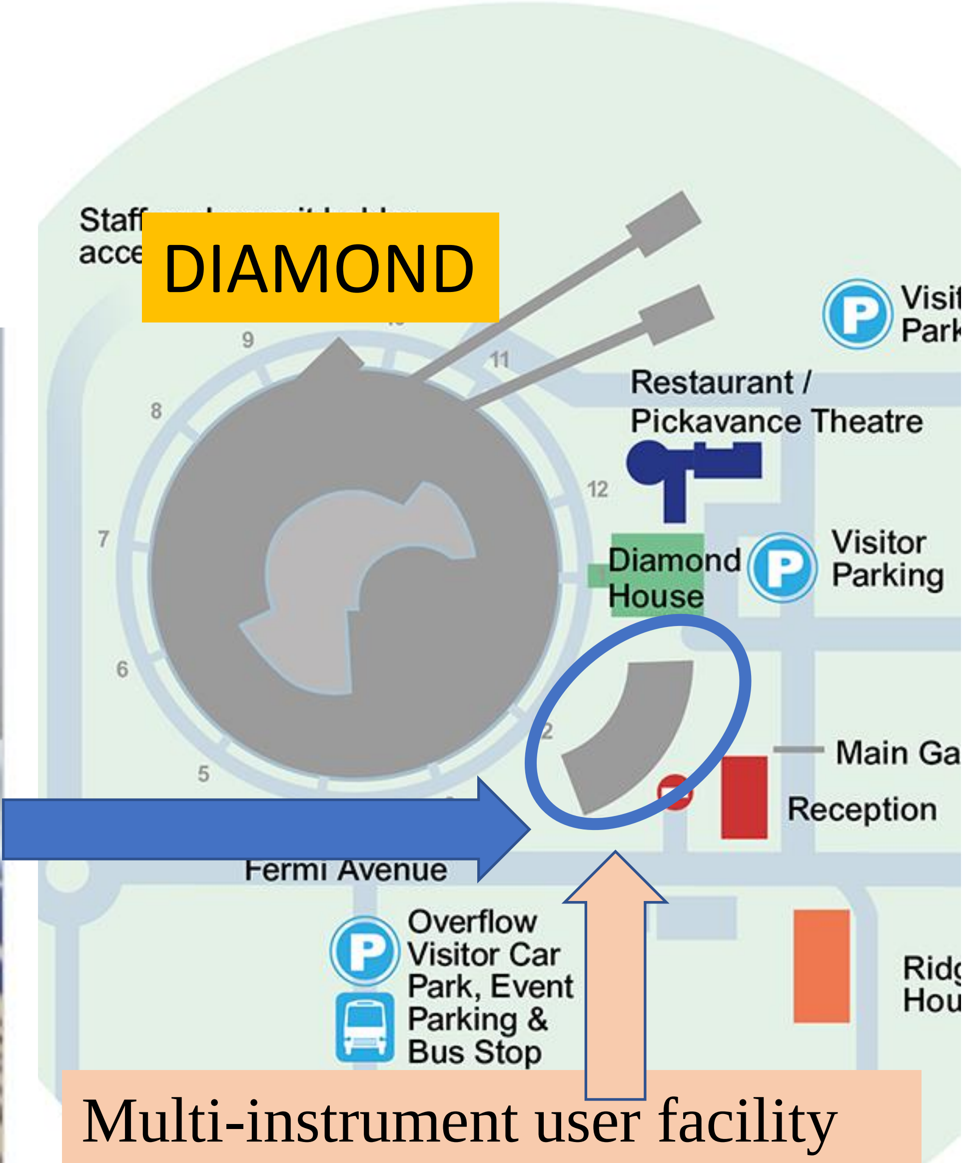
Growing use of special sample environments in modern lab XAFS instruments.

Harwell XPS
@HarwellXPS

Setting up our insitu electrocatalytic XAS measurements on our @easyXAFS spectrometer. @gsl1996 @MarkIsaacsChem bringing in some great data to go with our insitu XPS and Raman data!!



7:40 AM · Mar 16, 2023 · 3,608 Views



Multi-instrument user facility for research and industry in Harwell Science Campus in UK

IN PARTNERSHIP WITH easyXAFS,
C&EN PRESENTS

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Industrial Applications for Laboratory XAFS

BROUGHT TO YOU BY

easyXAFS

Two weeks ago:
October 16, 2023

2023: The XAFS Community is at the cusp of immense changes.

- Ambient transmission-mode measurements can usually be done in the lab with synchrotron quality results. (5 keV – 15keV+)
- Great preparation for synchrotron beamtime.
- Almost all bulk nonresonant XES can be done in the lab.
- Growing capability for *operando*, special environments, fluor mode for expert usage.
- *Student education, new user training, reach new research groups and disciplines, …*
- *Industrial special-purpose applications are coming.*

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- *Industrial special-purpose applications are coming.*

Many of these users are not in XAFS-experienced groups and *many will never go to a synchrotron.*

- What do new lab XAS users need?
  - Data quality: Need instrument-specific, comprehensive training and resources.
  - Experiment planning
  - Fitting or Inferences
  - Task-specific *Standard Test Methods*

2023: The XAFS Community is at the cusp of immense changes.

NOT JUST A LAB XAFS PROBLEM!
At the recent IUCR Quality and Quantity in XAFS workshop *beamline scientists were expressing the same concerns.*

- **In ~2000**, XAFS users were in expert groups and they did studies that were 'XAFS papers'.
- **Now**: The typical user needs XAFS to complement other analytical methods to set the scope of a more complex argument. The typical user is often a first-time XAFS users.

- *Student education, new user training, reach new research groups and disciplines, …*
- *Industrial special-purpose applications are coming.*

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At the recent IUCR Quality and Quantity in XAFS workshop *beamline scientists were expressing the same concerns.*

- **In ~2000**, XAFS users were in expert groups and they did studies that were 'XAFS papers'.
- **Now**: The typical user needs XAFS to complement other analytical methods to set the scope of a more complex argument. The typical user is often a first-time XAFS users.

What can we learn from XPS?

- **In ~2000**: most XPS studies are done by expert groups using in-group equipment.
- **Now**: most XPS studies are done by non-expert users in shared University facilities.

- *Student education, new user training, reach new research groups and disciplines, …*
- *Industrial special-purpose applications are coming.*

Many of these users are not in XAFS-experienced groups and *many will never go to a synchrotron.*

- What do new lab XAS users need?
  - Data quality: Need instrument-specific, comprehensive training and resources.
  - Experiment planning
  - Fitting or Inferences
  - Task-specific *Standard Test Methods*

What can we learn from XPS?

J Vac. Sci. Tech. A (2020)

Assessment of the frequency and nature of erroneous x-ray photoelectron spectroscopy analyses in the scientific literature FREE

Special Collection: Special Topic Collection: Reproducibility Challenges and Solutions

George H. Major ; Tahereh G. Avval; Behnam Moeini; Gabriele Pinto; Dhruv Shah; Varun Jain; Victoria Carver; William Skinner ; Thomas R. Gengenbach ; Christopher D. Easton ; Alberto Herrera-Gomez ; Tim S. Nunney; Donald R. Baer ; Matthew R. Linford

30% of XPS publications have *major* errors in execution or fitting. Several XPS-heavy journals are now desk-rejecting a large portion of submitted manuscripts.

J Appl. Physics (2022)

A step-by-step guide to perform x-ray photoelectron spectroscopy F SCI

Grzegorz Greczynski[a)] and Lars Hultman

Outstanding tutorial paper, combines instrument principles with examples of failures and guidance for good practice.

Lab XAFS needs this kind of technical tutorial.

J Appl. Physics (2022)

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Will be posted on chemrxiv and submitted *very soon*!

Best Practice for High Data Quality in Laboratory-Based X-ray Absorption Spectroscopy

Gerald T. Seidler[1,*], Anthony Gironda[2], Jared Abramson[1], Charles Cardot[1], Yuen Chen[1], Diwash Dhakal[2]

1 Physics Department, University of Washington, Seattle, United States 98195
2 Department of Materials Science and Engineering, University of Washington, Seattle, United States 98195

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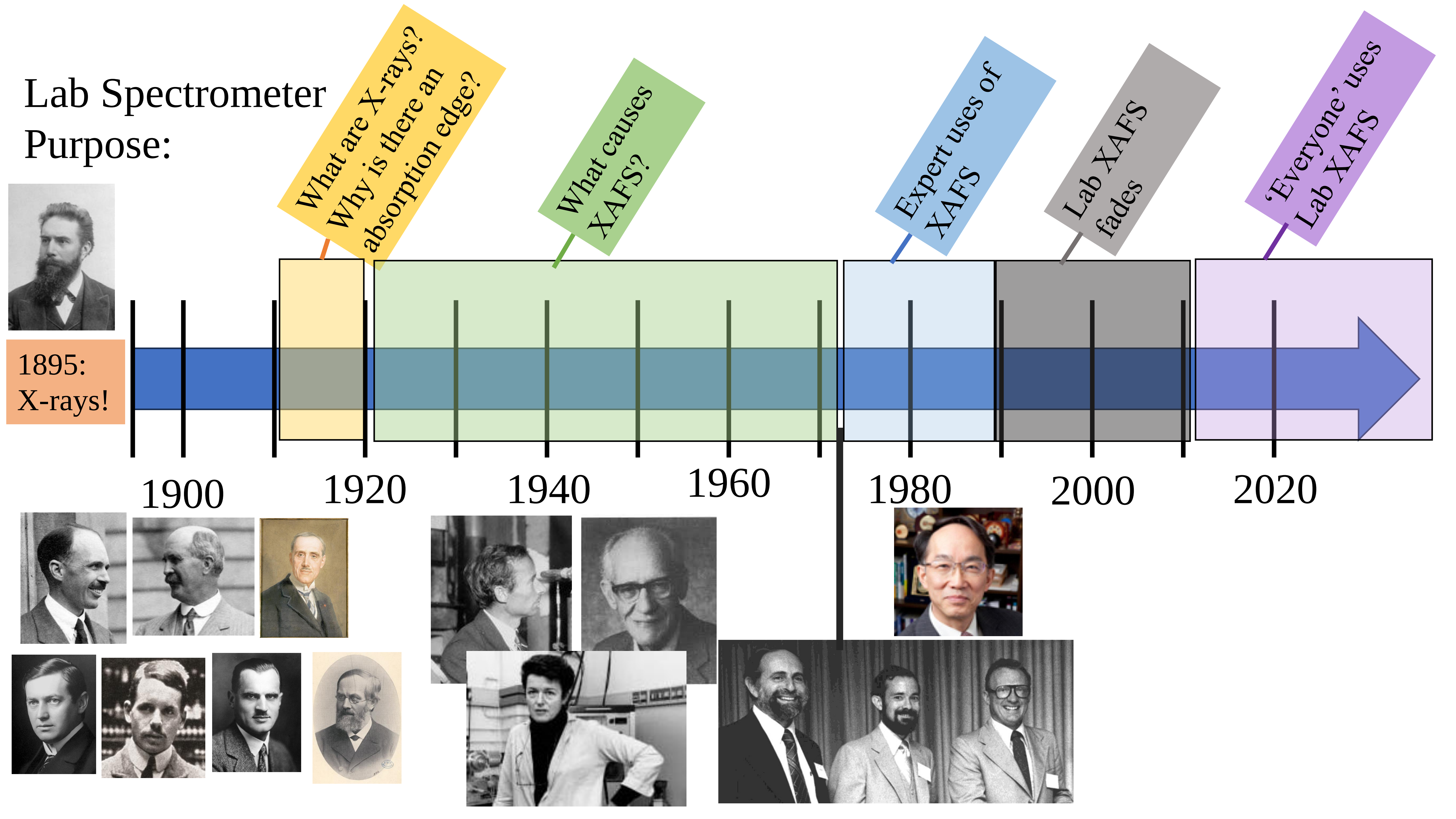
Goal: 'Best practice' means that you reduce instrument-specific systematic errors until your analysis issues are the same as at a synchrotron.

There cannot be 'lab XAFS' and 'synchrotron XAFS' as distinct fields, there can only be XAFS!

Contents



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Why is there an
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What causes
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Expert uses of
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fades
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2000
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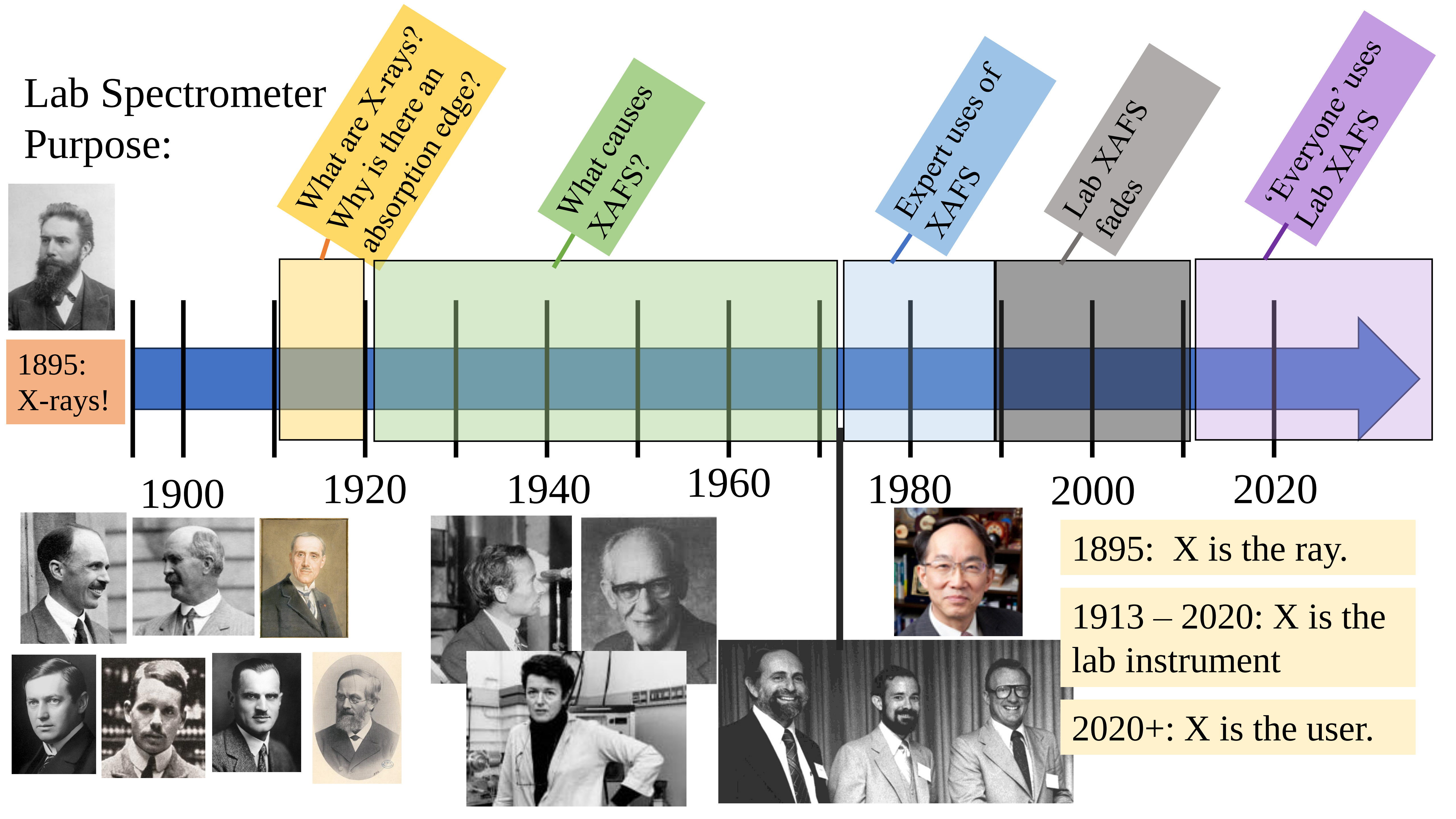
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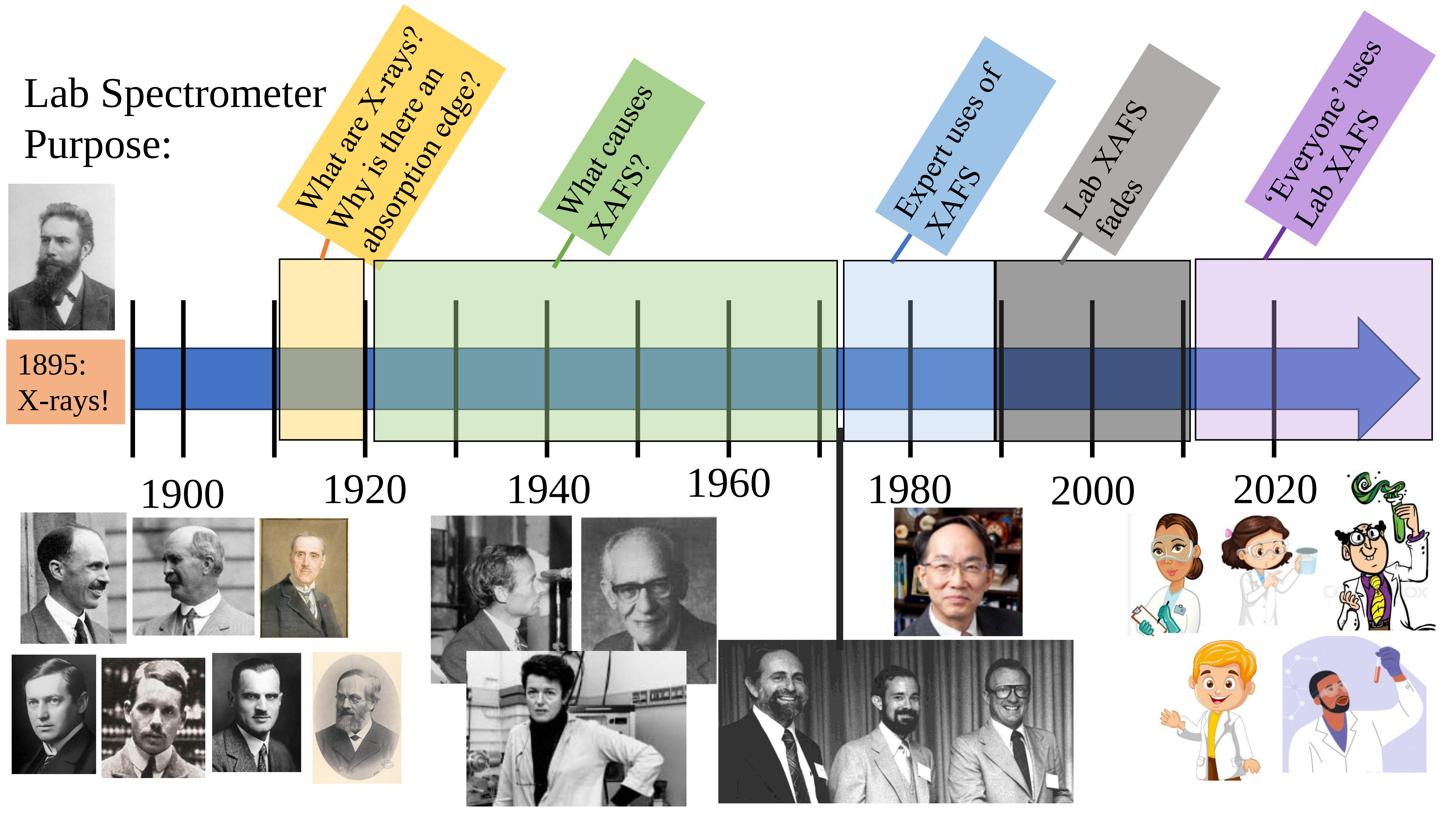
1895: X is the ray.
1913 – 2020: X is the lab instrument
2020+: X is the user.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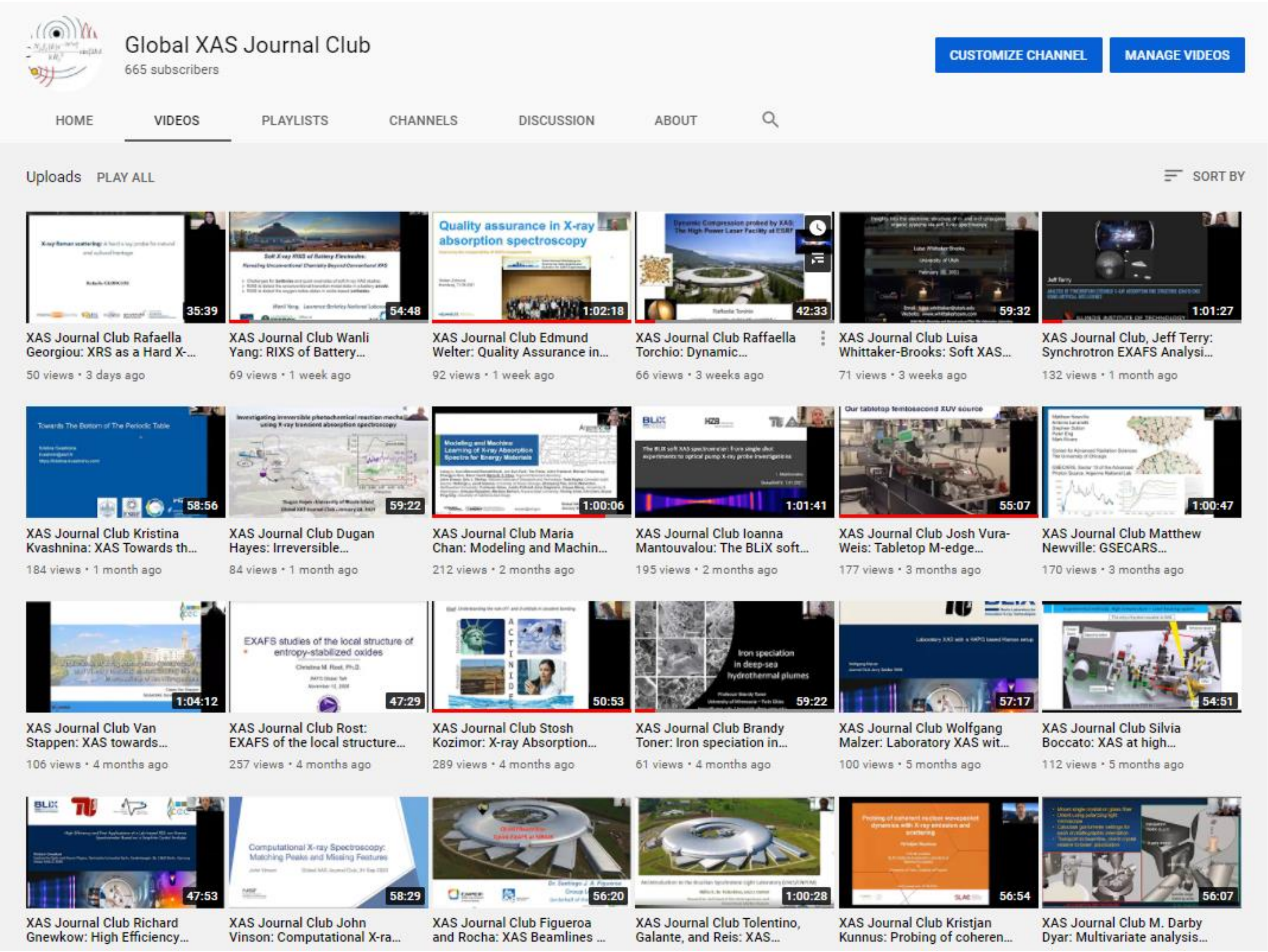

YouTube channel: tinyurl.com/XASvideos

Email me to be added to the mailing list: seidler@uw.edu