ASTM E42 Surface Analysis Community Forum

Friday, November 6, 2020,

This PDF file:

- The announcement for the Community Forum which was held November 6, 2020
- The three presentations given as part of the Forum
    - Matthew R. Linford, An Assessment of the Frequency and Nature of Erroneous X-ray Photoelectron Spectroscopy (XPS) Analyses in the Scientific Literature
    - Mark H. Engelhard, The Structure of the Current Tool Kit, standards, *Historic drivers and creation of guides and reports prepared by ASTM E42 on Surface Analysis and ISO TC 201 on Surface Chemical Analysis*
    - Donald R. Baer, Issues affecting credibility in XPS analysis and interpretation, Strategies Going Forward
- Compiled version of Chat discussion of questions, comments and ideas from the Forum
- Copy of follow-up email to Forum Participants providing information, requesting input and talking about a path to move forward.

To navigate to the section of interest, load the PDF and click on the bookmark tab on the left side of the screen. You should be able to see an icon for each of the documents in the PDF. Clicking on that icon will expand for each page in a file or contract one image for each file, as indicated above.

Image of bookmark

ASTM E42 Surface Analysis Community Forum

Friday, November 6, 2020,
12:00 EST (18:00 Europe, 17:00 UK, 09:00 Pacific US)
Duration: 90 minutes

ASTM committee E42 on surface analysis invites you to attend our community forum discussion on **issues affecting credibility in XPS analysis and interpretation**.

Data in the literature that is poorly acquired, analyzed, or presented can have far reaching effects on the credibility of any technique. Ensuring that the community has the resources available to help the expanding user base will benefit us all!

Join us for a virtual discussion of this surface analysis research community topic and help determine the best solution path that addresses these pressing issues.

Leading experts will lead discussion with participants, laying out the specifics of the problem, explaining how this has progressed and the resources currently available, and then looking forward to how we can improve the resources available and their distribution, with a goal of providing tools to improve research results.

The Problem, as it affects the research community

Matt Linford will describe a multi-institutional and multi-country analysis of XPS reported in three scientific journals which demonstrates significant problems in the analysis of XPS data appearing in the literature. He will describe a quantitative assessment of the problems and report information learned about the most common issues that have been observed.

The Structure of the current toolkit

Mark Engelhard will then provide a short summary of early issues in XPS analysis which motivated the creation of the standards committees ASTM E42 and ISO TC201, and will summarize the types of standards and guides that have been created, and explain some of the inter-relationships among relevant surface analysis standards in E42 and TC201. Recent investigations questioning the adequacy of the reporting of analysis information specified in ISO and ASTM standards will be discussed, as an example of the limitations of the standards and guides in meeting the community data reporting challenges.

The Strategy Going Forward

Don Baer will relay the development of recent guides that are intended to help address the issues and explain what is still in the pipeline. He will explore other tools that might be useful, including the possibility of an XPS reporting guide that could indicate prescribed reporting for levels of confidence and the work towards normalization of these. One objective of this presentation is to seek community input on tools and/or other approaches on how to decrease the incorrect XPS data reports in the literature. Such discussion can guide development of ASTM 42, ISO TC201, AVS Recommended Practices and other activities.

Together, we can build a framework to ensure that resource is available for the research community to help improve the quality of surface analysis result reporting!

An Assessment of the Frequency and Nature of Erroneous X-ray Photoelectron Spectroscopy (XPS) Analyses in the Scientific Literature

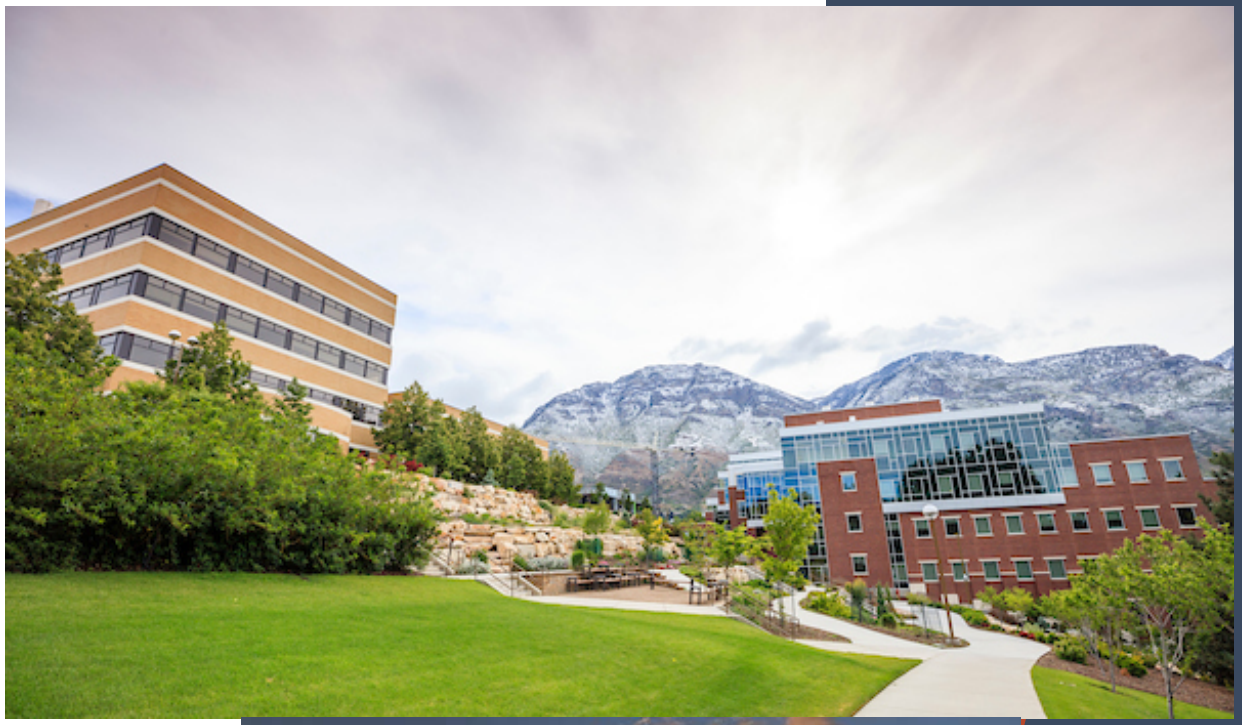
Matthew R. Linford

Department of Chemistry and Biochemistry

Brigham Young University

Provo, UT 84602

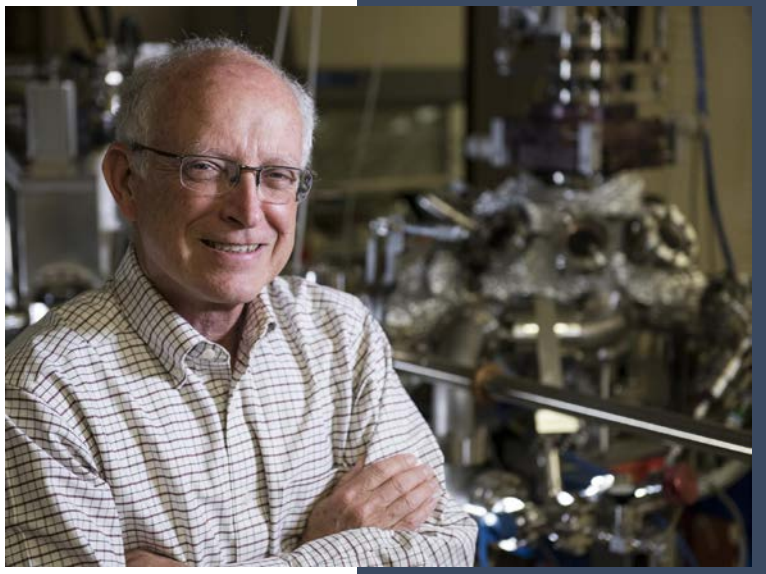
Nov. 6, 2020



https://www.chem.byu.edu/about/department-information/, uvu.edu

A problem with the XPS data analysis in the literature. Was there always a problem?

- Decades ago, surface analysis was mostly done by a very small community of experts
- However, as the importance of surface analysis has increased, many more people have been using XPS
- Most XPS is now being done by non-experts
  - About 150 instruments are sold each year
  - The world doesn't produce enough highly trained experts to go with those instruments
- A senior spokesman for XPS, Don Baer, retired from PNNL
- Don suggested that the community of experts write guides on XPS to help newer users do better
- Quite a few of us have been involved



https://www.pnnl.gov/people/donald-baer

Example of one of the guides

Practical guide for curve fitting in X-ray photoelectron spectroscopy

Running title: XPS curve fitting guide

Running Authors: George. H. Major et. al.

George H. Major [1], Neal Farley [2], Peter M.A. Sherwood [3], Matthew R. Linford [1], Jeff Terry [4], Vincent Fernandez [5], Kateryna Artyushkova [6a)]

[1]Department of Chemistry and Biochemistry, Brigham Young University, C100 BNSN, Provo, Utah 84602
[2]Casa Software Ltd., Bay House, Teignmouth, UK
[3]Department of Chemistry, University of Washington, Seattle, WA 98195
[4]Illinois Institute of Technology, 3101 S. Dearborn St., Chicago IL 60616
[5]Université de Nantes, CNRS, Institut des Matériaux Jean Rouxel, IMN, F-44000, Nantes, France
[6]Physical Electronics, 18725 Lake Drive East, Chanhassen, MN 55317

[a)] Electronic mail: kartyushkova@phi.com

http://www.unm.edu/~kartyush/

A Letter to Help Alert the Scientific Community to the Issue

Microscopy and Microanalysis (2020), 1–2
doi:10.1017/S1431927619015332

Microscopy AND Microanalysis

Letter to the Editor

Proliferation of Faulty Materials Data Analysis in the Literature

Matthew R. Linford[1], Vincent S. Smentkowski[2]*, John T. Grant[3], C. Richard Brundle[4], Peter M.A. Sherwood[5], Mark C. Biesinger[6], Jeff Terry[7], Kateryna Artyushkova[8], Alberto Herrera-Gómez[9], Sven Tougaard[10], William Skinner[11], Jean-Jacques Pireaux[12], Christopher F. McConville[13], Christopher D. Easton[14], Thomas R. Gengenbach[14], George H. Major[1], Paul Dietrich[15], Andreas Thissen[15], Mark Engelhard[16], Cedric J. Powell[17], Karen J. Gaskell[18] and Donald R. Baer[16]

[1]Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT 84602, USA; [2]General Electric Research, Niskayuna, NY 12309, USA; [3]Surface Analysis Consultant, Clearwater, FL 33767, USA; [4]C.R. Brundle & Associates, Soquel, CA 95073, USA; [5]University of Washington, Box 351700, Seattle, WA 98195, USA; [6]Surface Science Western, University of Western Ontario, London, Ontario N6G 0J3, Canada; [7]Department of Physics, Illinois Institute of Technology, Chicago, IL 60616, USA; [8]Physical Electronics, Chanhassen, MN 55317, USA; [9]CINVESTAV – Unidad Queretaro, Real de Juriquilla 76230, Mexico; [10]Department of Physics, University of Southern Denmark, Odense 5230, Denmark; [11]Future Industries Institute, University of South Australia, Mawson Lakes, SA 5095, Australia; [12]University of Namur, Namur Institute of Structured Matter, B-5000 Namur, Belgium; [13]College of Science, RMIT University, Melbourne, VIC 3001, Australia; [14]CSIRO Manufacturing, Ian Wark Laboratories, Clayton, VIC 3168, Australia; [15]SPECS Surface Nano Analysis GmbH, 13355 Berlin, Germany; [16]Pacific Northwest National Laboratory, Richland, WA 99354, USA; [17]National Institute of Standards and Technology, Gaithersburg, MD 20899, USA and [18]University of Maryland, College Park, MD 20742, USA

(Received 11 December 2019; revised 19 December 2019; accepted 19 December 2019)

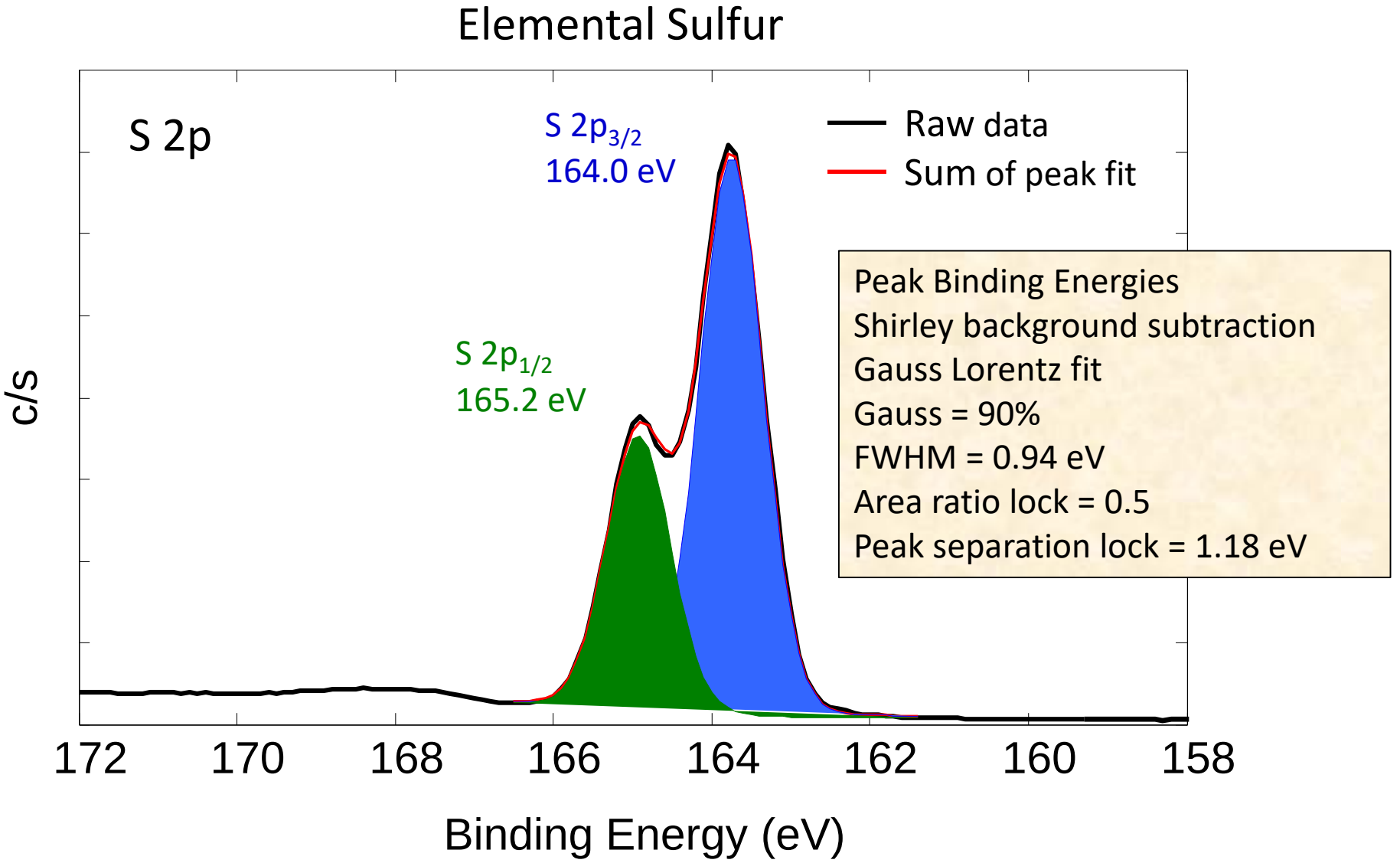
How bad are things really?

- Noise labeled as chemical states

"Aluminum Coating Influence on Nitride Layer Performance Deposited by MO-CVD in Fluidized Bed on Austenitic Stainless Steel Substrate". IOP Conf. Series: Materials Science and Engineering 374 (2018) 012020 doi:10.1088/1757-899X/374/1/012020.

The Proliferation of Bad XPS Peak Fitting in the Literature

s vs. *p*, *d*, and *f* orbitals



Peak Binding Energies
Shirley background subtraction
Gauss Lorentz fit
Gauss = 90%
FWHM = 0.94 eV
Area ratio lock = 0.5
Peak separation lock = 1.18 eV

https://www.linkedin.com/in/mark-engelhard-0b824739/?locale=de_DE

The Proliferation of Bad XPS Peak Fitting in the Literature

12.1 impact factor

The authors should have considered S 2p spin-orbit splitting
The S $2p_{1/2}$ S $2p_{3/2}$ peak area ratio should be set to 1:2
and peak separations should be 1.18 eV

Nature Communications 6:7436 **(2015)** DOI: 10.1038/ncomms8436

Figure from Mark Engelhard at EMSL

The Proliferation of Bad XPS Peak Fitting in the Literature

Widely varying peak widths in a fit here.

Disordering in
$Gd_2(Ti_{1-x}Zr_x)O_7$ Pyrochlores

"It can be fitted by two Gaussian functional curves, indicating the different anion migration mechanism from that in $Gd_2(Ti_{1-x}Zr_x)O_7$"

Phys Rev Letters,
V88, N10 **(2002)**
DOI:10.1103/PhysRevLett.88.015901

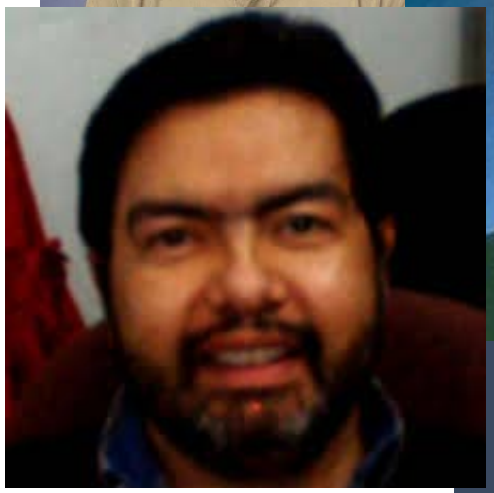
The Proliferation of Bad XPS Peak Fitting in the Literature

It is not uncommon to see spectra from similar materials compared. In these cases, authors sometimes do not constrain the fit components so that components that should represent the same chemical states in the spectra end up with very different positions and widths.

Measuring the Problem

- We were all seeing really bad XPS analysis in the literature, but no one had measured the problem. How bad was it really?
- We decided to measure it
- We formed an ad hoc committee with other experts: Don Baer (PNNL), Thomas Gegenbach and Chris Easton (CSIRO, Australia), Bill Skinner (Future Industries Institute, Australia), Alberto Herrera-Gomez (CINVESTAV, Mexico)
- We evaluated all the XPS spectra shown in three high-quality journals (A, B, and C) over a six-month time period: 407 of the papers we looked at showed XPS spectra, 63% of these were fitted
  - Journal A: Battery/energy journal IF ~ 25
  - Journal B: Surface and materials, IF ~ 4
  - Journal C: General science journal with a lot of materials content: IF ~ 4

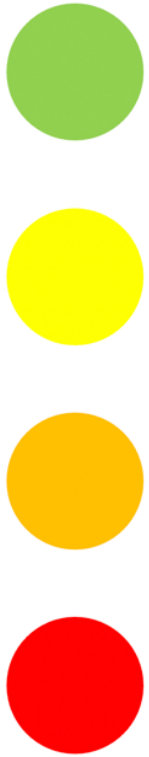


https://www.linkedin.com/in/thomas-gengenbach-4b1b5a53/, https://www.linkedin.com/in/bill-skinner-39198177/?originalSubdomain=au, https://www.researchgate.net/profile/Alberto_Herrera-Gomez, https://www.pnnl.gov/people/donald-baer

Measuring the Problem

- Our classification scheme



Green: No errors or very minor errors. This analysis should contribute to the message of the paper it is in.

Yellow: Minor issues. These may include deviations from standard practices, peak identification, or reporting. While these errors may indicate that the authors are inexperienced with XPS, they pose no major problems. This analysis should contribute to the message of the paper it is in.

Orange: Significant but perhaps not fatal, issues that may compromise the message of the paper, including noticeable errors in peak fitting and analysis.

Red: Significant errors that most likely compromise the validity of the work.

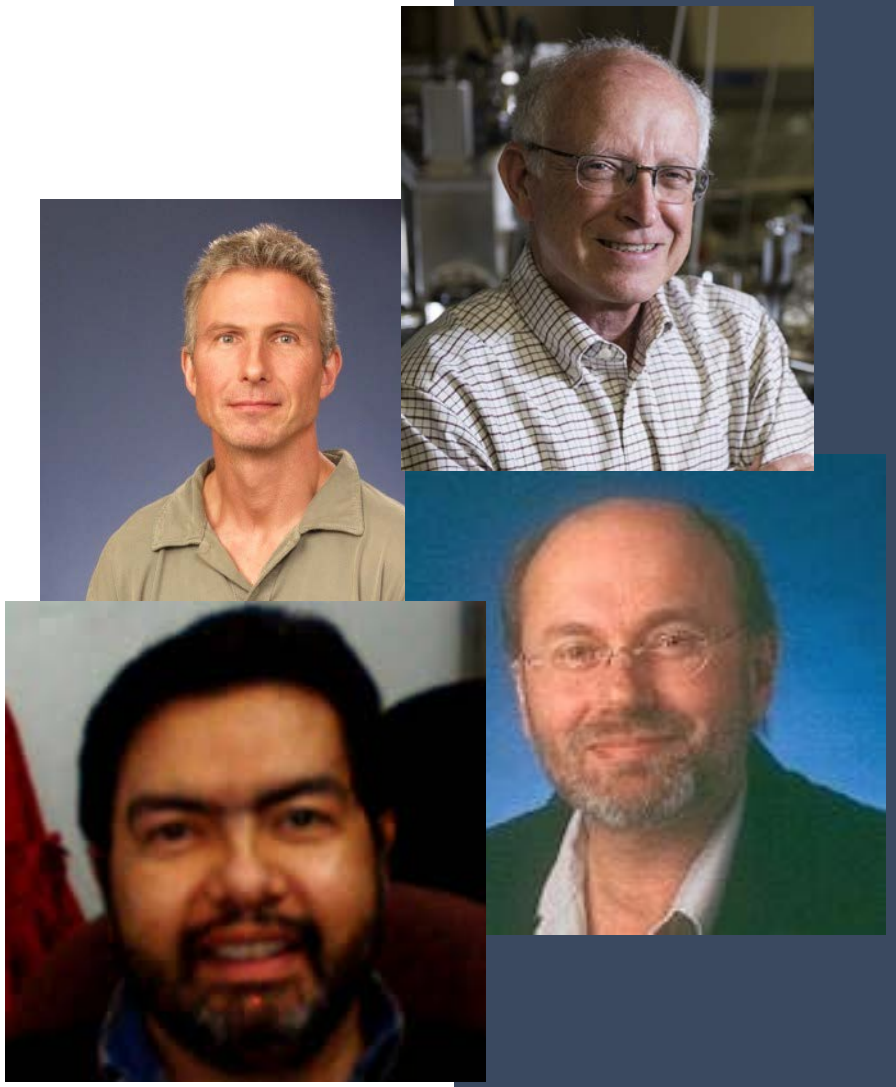
The Errors

Green Category

- No significant errors, although there may be a few minor issues

Yellow Category

- Modest truncation of the edges of a peak envelope
- Neglecting to include the sum of the fit components and/or the residuals to the fit (or some other figure of merit for peak fitting), but the fit components appeared to be a good approximation to the peak envelope.
- Not including/showing the background/baseline for the fit, but, again, the fit/data analysis otherwise seemed reasonably sound
- Some concerns about the selection of the baseline relative to the noise



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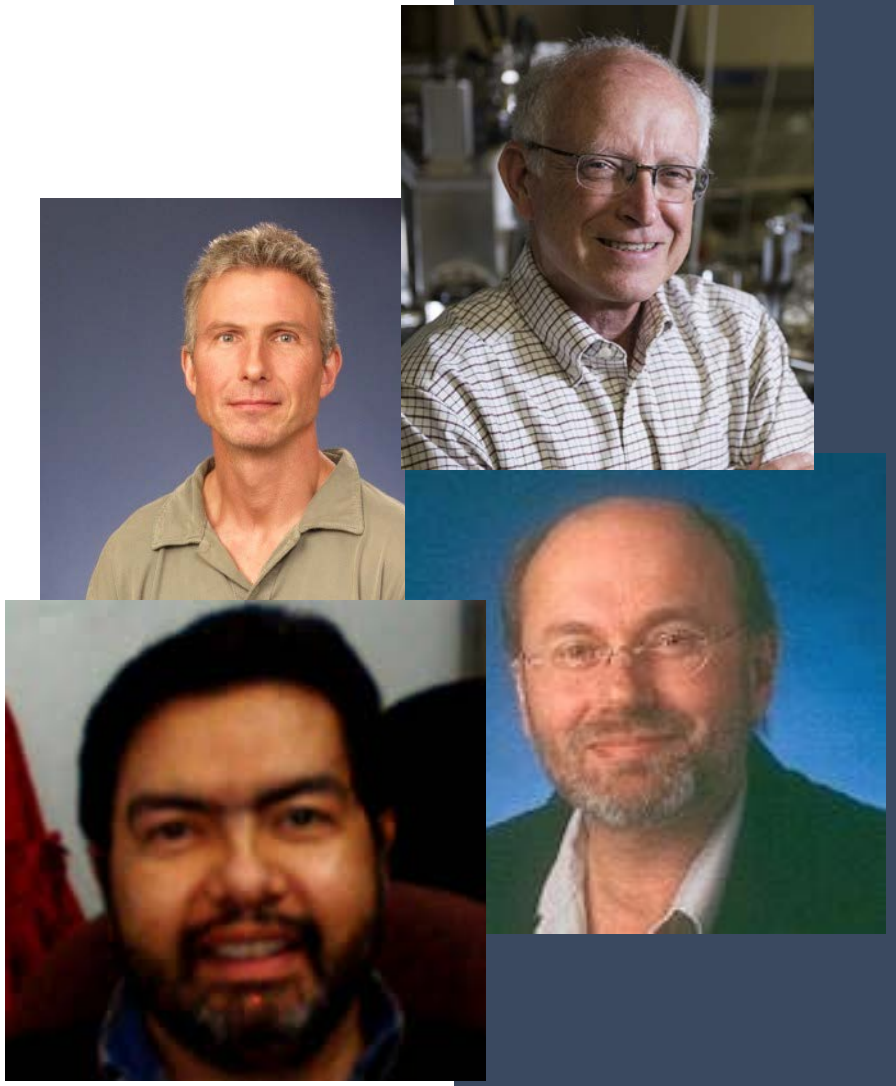
The Errors

Orange Category

- Significant truncation of the peak envelope in a narrow scan
- Not including the sum of the fit components and/or the residuals, where the sum of the fit components did not appear to be a good approximation of the peak envelope
- Using an incorrect background for a fit
- Failure to match background to the surrounding noise
- Employing varying peak widths in a fit when there was no good chemical reason for doing so
- Adding too many synthetic peaks to a fit, ignoring the sample physics and chemistry
- Attempting to fit and interpret noisy data when it was clear that little meaningful information could be extracted from the data



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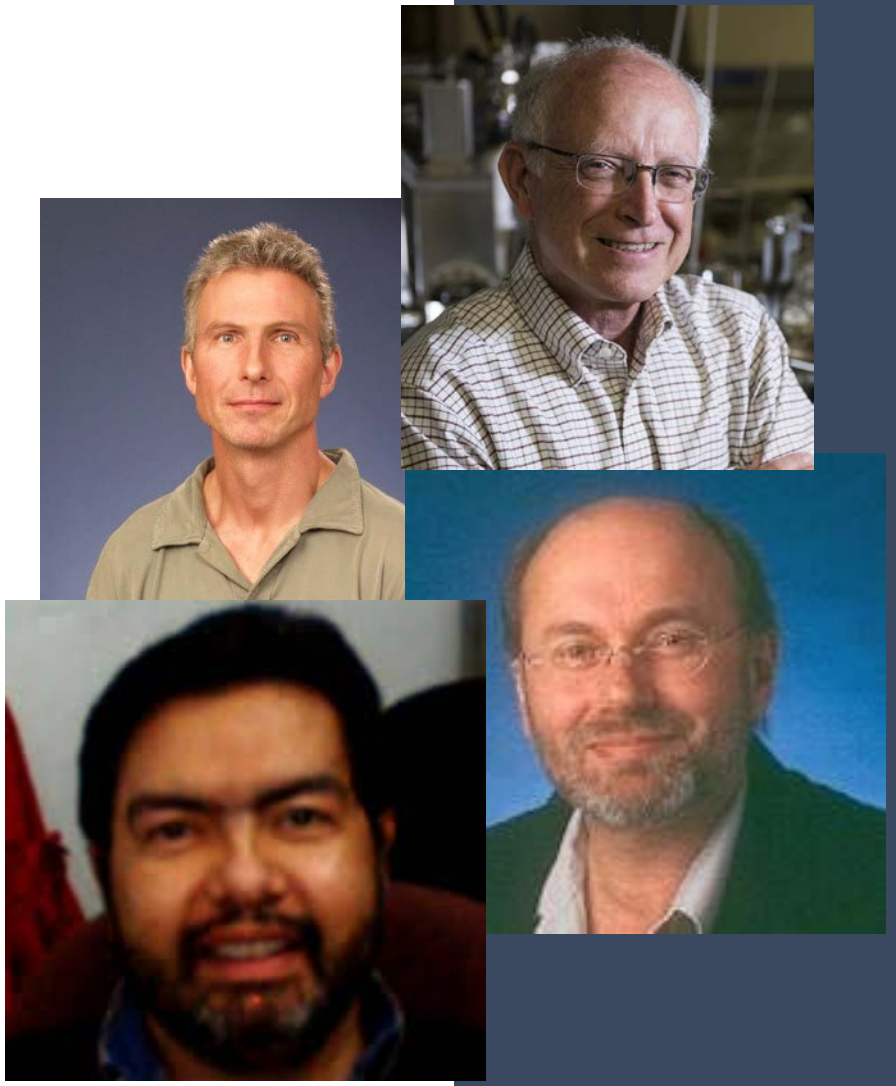
The Errors

- *Red Category*
- A paper could receive a Red rating if it contained a significant number of Orange errors or particularly egregious Orange errors
- Extreme truncation of the peak envelope in a narrow scan
- Gross failure to make the background match or be appropriately close to the noise surrounding the peak envelope such that the resulting peak areas/quantitation would be meaningless, e.g., the background line may cut through the spectral envelope
- Employing wildly varying peak widths in a fit when there was no good chemical or physical reason for doing so
- Adding far too many synthetic peaks to a fit
- Attempting to fit extremely noisy data



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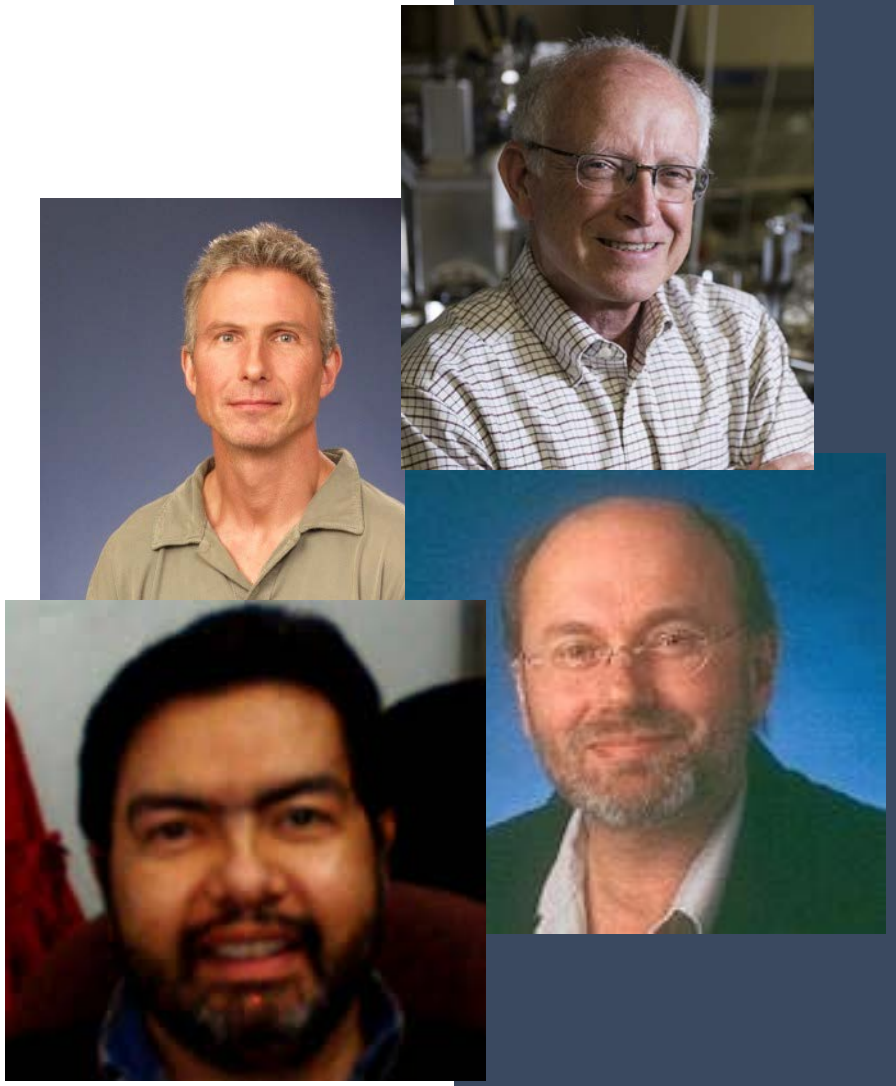
The Errors

- *Red Category, Cont.*
- Disregarding/neglecting spin-orbit splitting when it was present, not using proper spin-orbit splitting ratios, or labelling a pair of spin-orbit peaks as separate chemical states
- Failure to include the original data, e.g., showing only the synthetic peaks for a fit
- Gross mislabeling of chemical states, labeling noise as chemical states, omitting chemical states, or proposing impossible chemical states. For example, in their C 1s peak fitting, authors sometimes (i) mislabel (switch) the C-O and C=O chemical states fitting, (ii) omit the C=O state, (iii) try to fit the natural asymmetry (tailing) in the C 1s signal of sp^2-type carbon, e.g., from graphene or carbon nanotubes, as multiple carbon-oxygen type components, even when there is not enough oxygen in the material to justify these synthetic peaks, as indicated by a small or nonexistent O 1s peak from the sample – here, it might be better to first fit the C 1s spectrum from the unfunctionalized sp^2-containing material with an asymmetric line shape, and then use this line shape to fit the functionalized materials,[45] and (iv) try to fit (and label) the shake-up signal(s) from materials containing sp^2 carbon as carbon-oxygen type chemical states.
- There are obviously many more ways that XPS spectra can be inappropriately fit



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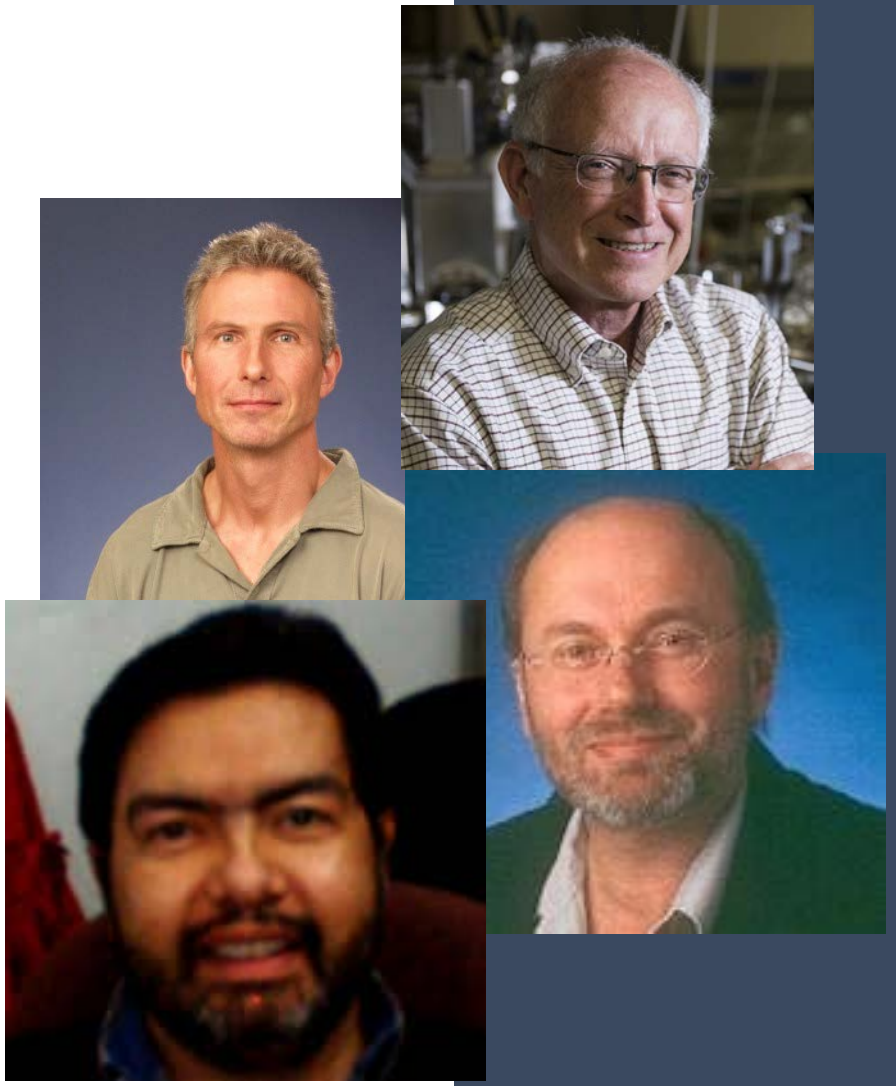
Measuring the Problem

- Five panel members evaluated these papers individually, and then met and came to a consensus on every paper
- There was good agreement in the initial ratings by the experts: there was no disagreement among the five panelists regarding ca. 60% of the initial rankings, in ca. 33% of all cases rankings fell into two neighboring color categories, and in only 7% of ranked papers did initial rankings differ by a greater amount.
- The sixth independent committee member then reviewed all the initial recommendations. He mostly agreed with the committee's evaluations, but also recommended that a few of the papers that had been classified as Orange should be recategorized as Red.



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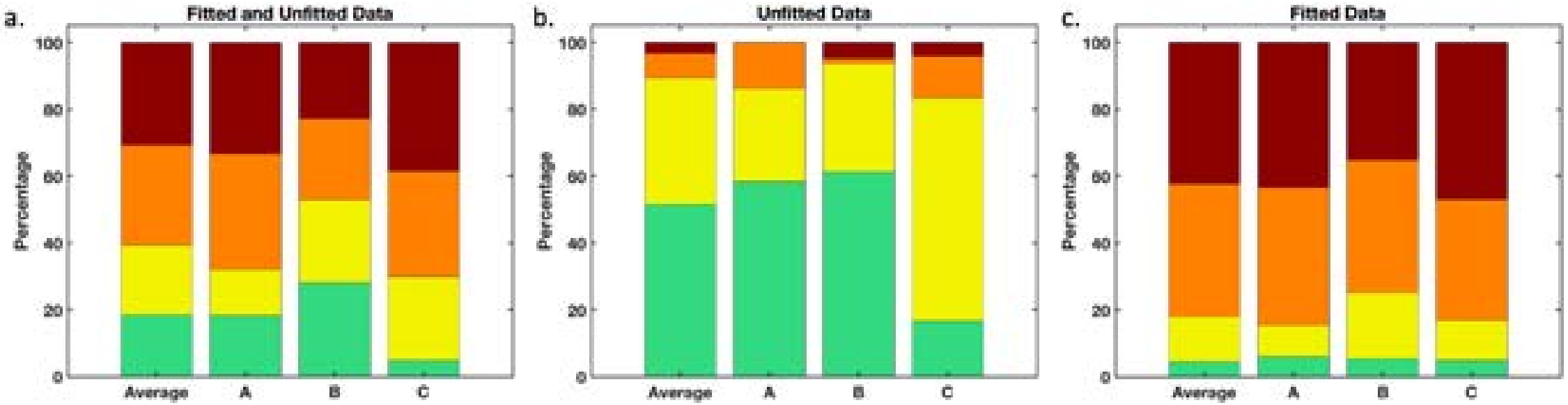
Measuring the Problem

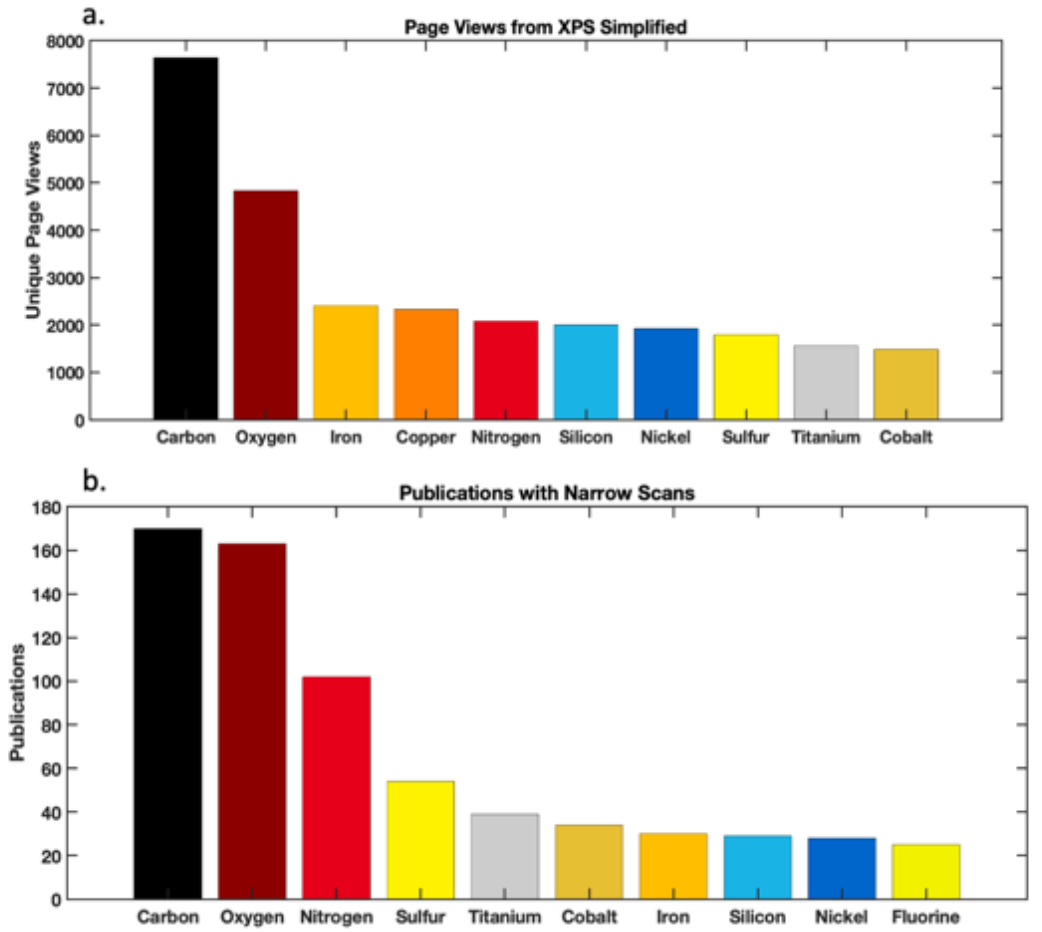
- Our results:



- Our study was just accepted to JVSTA

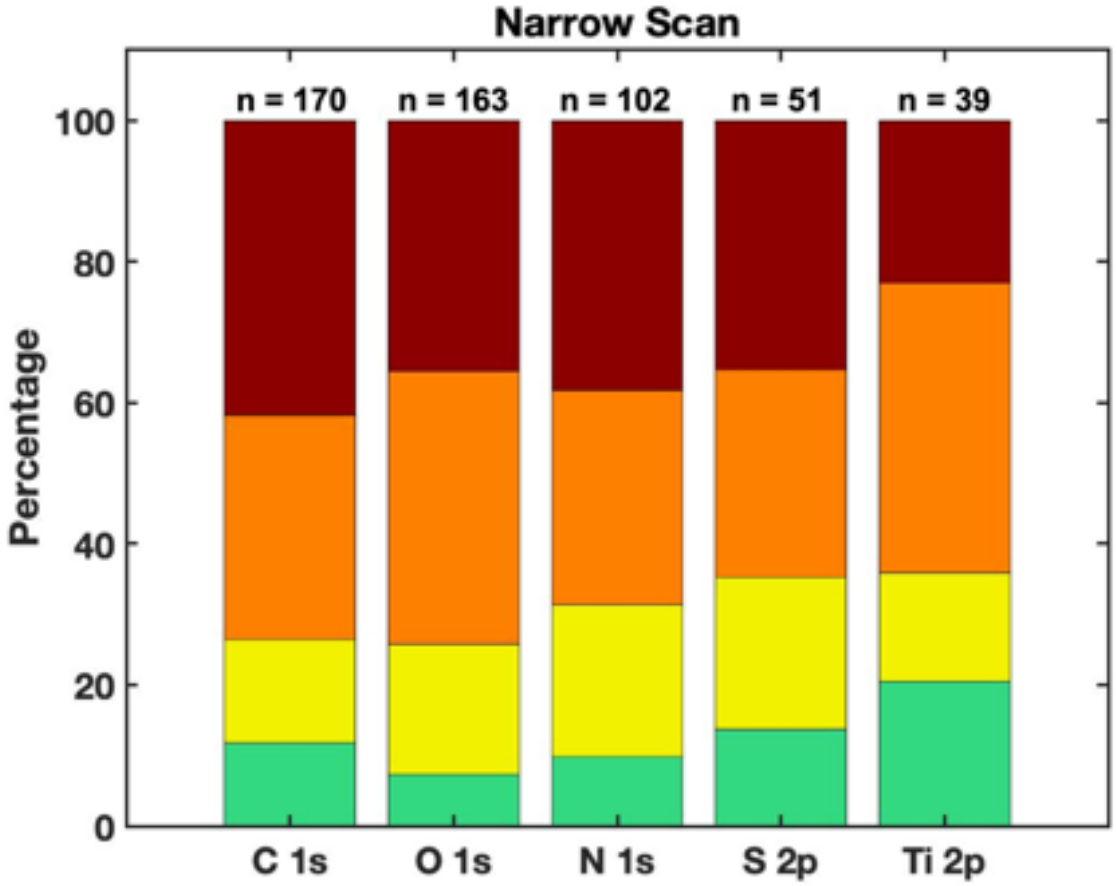
Which elements are shown in the literature?

- Tim Nunney from Thermo Fisher published an analysis on LinkedIn of the frequency with which the different elements are searched at their XPS Simplified web site
- He provided us an even more complete set of this information
  - Two months of results (a total of 48,996 unique page views)
- We compared his information to the frequency with which different narrow scans are shown in the literature
- 9 of the top 10 elements are the same on both lists
- C and O are the most researched and shown elements



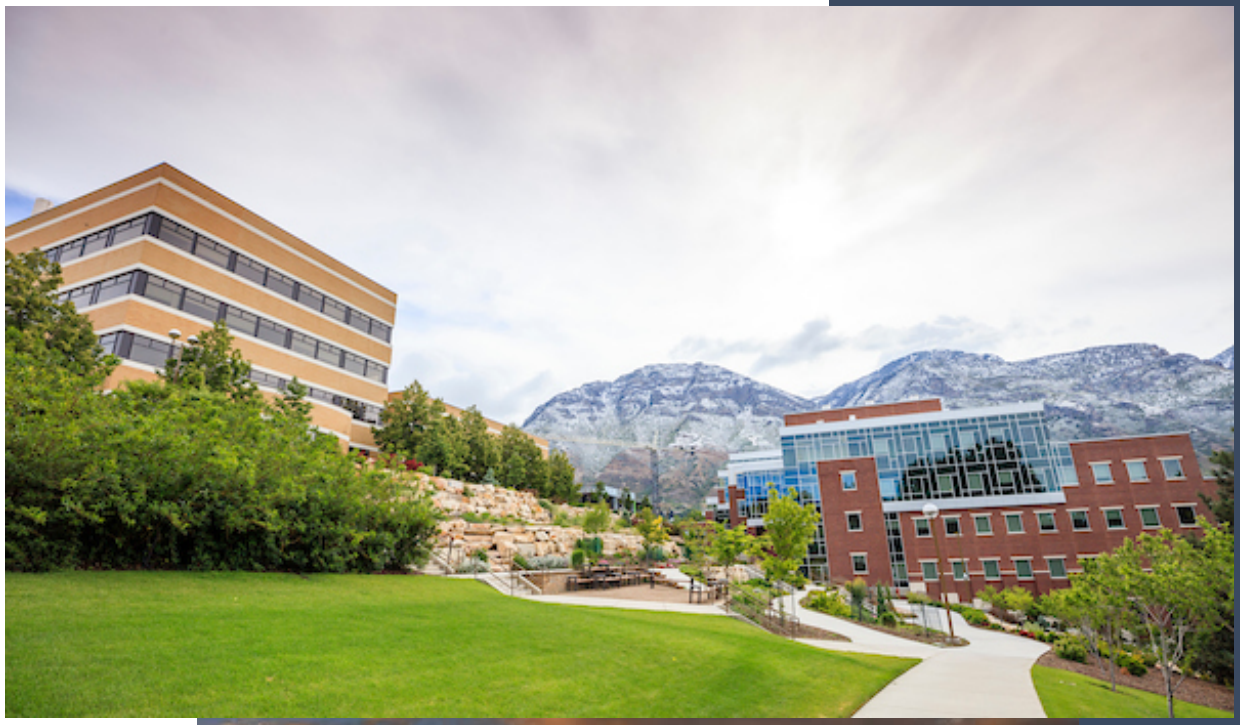
Which elements are shown in the literature?

- Errors in the top 5 elements shown in the literature
- Many errors in fitting the C, O, and N 1s spectra



What does this mean for me and you as scientists?

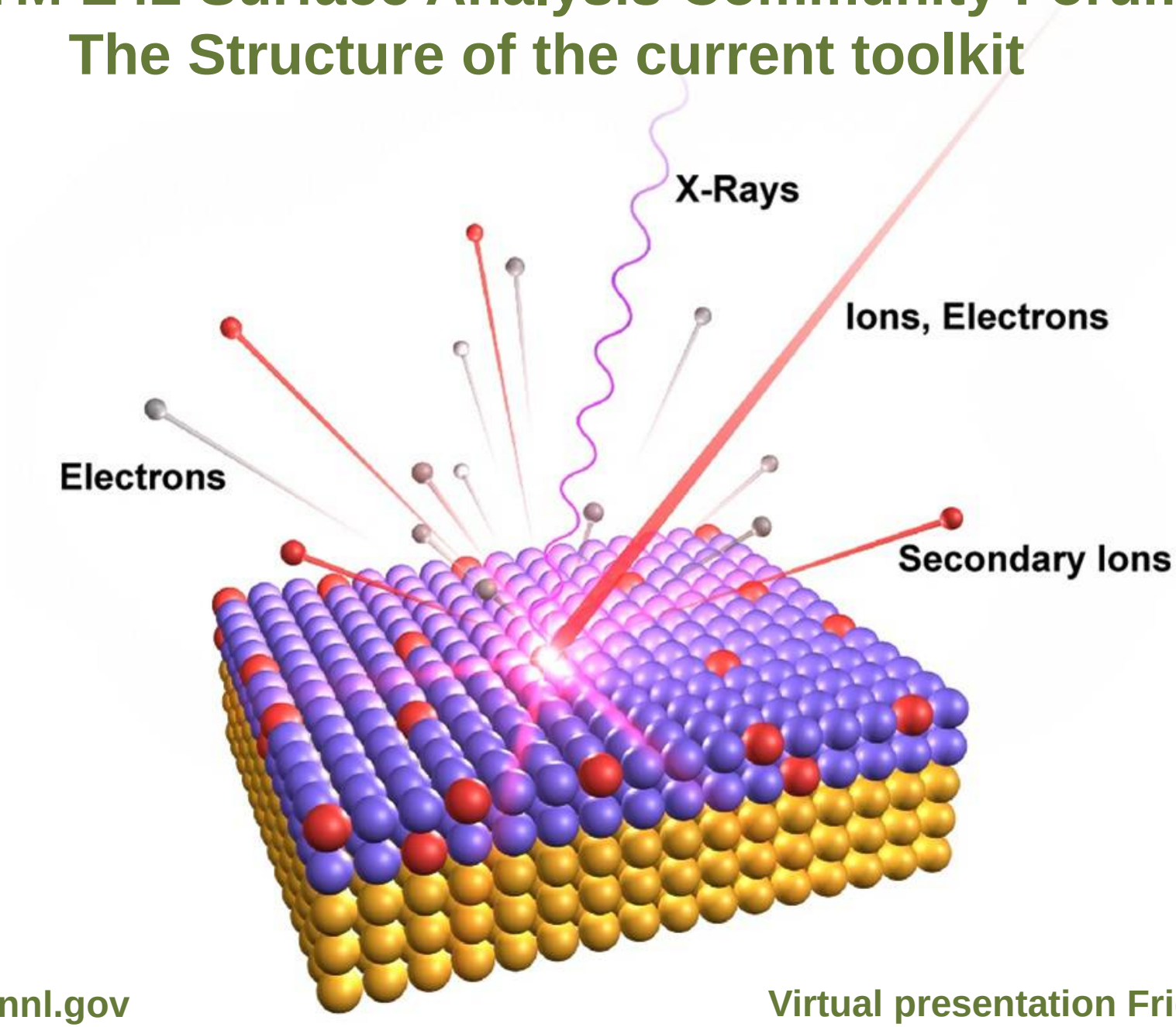
- People are reporting that other material characterization results in the literature have similarly high numbers of errors
- Another recent report[1] showed that data reliability in a paper *decreases* as the impact factor of the journal *increases*
- We as a community will have to think about and deal with this issue – how can individual scientists, journals, peer reviewers, and funding agencies do better?
- Happy to take questions on this presentation



[1]B. Brembs, Frontiers in Human Neuroscience **12**, 37 (2018).

https://www.chem.byu.edu/about/department-information/, uvu.edu

ASTM E42 Surface Analysis Community Forum
The Structure of the current toolkit



mark.engelhard@pnnl.gov

Virtual presentation Friday November 6, 2020

Outline

- Brief history of ASTM E42 and ISO TC201
- Some early ASTM Round Robins (i.e. 1977, 1979, and 1981)
- Summary of ASTM and E42 XPS “Practices” & “Guides”
- Summary of ISO TC201 XPS “Standards” & “Technical Reports”
- Examples of our experiences with early surface analysis instruments
- Selected E42 and ISO documents that help to educate our analysts and improved the quantity of results


Standards
Practices
Procedures
Guides
Technical Reports
Guidelines

ISO-ASTM-ANSI: History of ASTM E42 and ISO

- **ASTM E42** and the Applied Surface Science Division (**ASSD)** share a common history
  - ASTM E42 was established in 1976
  - ASSD was established in 1985 after 8 years of ASTM E42 co-sponsored sessions at the annual AVS International Symposium
  - http://avs.org/Divisions/assd/History
- **ISO** is the International Standards Organization

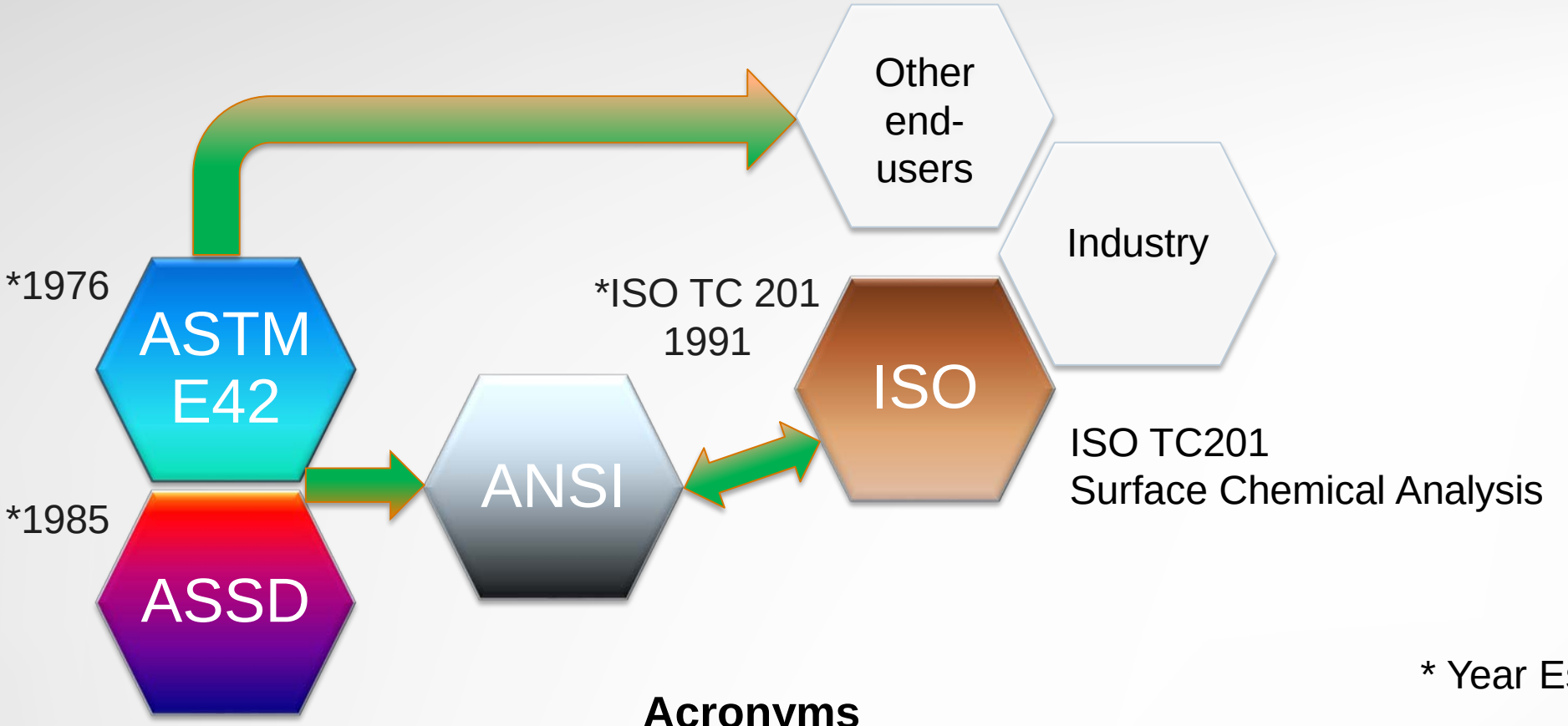
The need for reliable surface analyses together with quality-management requirements led ISO to form its Technical Committee (TC) 201 on Surface Chemical Analysis in 1991

ISO is organized by member **countries**

- Each country has an organization that is the liaison organization to ISO

For the US, this is ANSI (the American National Standards Institute)

Relationship between ASSD, ASTM, ANSI & ISO



* Year Established

Acronyms

ASSD = Applied Surface Science Division

ASTM = American Society Testing & Materials

ANSI = American National Standards Institute

ISO = International Organization for Standardization

SC = Sub Committee

TAG = Technical Advisory Group

TC = Technical Committee

HOD = Head of Delegation

ASTM E42 Surface Analysis

Chair: Dr. Chris Moffitt

ASTM E42 Surface Analysis *1976 * Year Established

Sub Committee		Sub Committee Chairs
E42.01	Terminology	Alberto Herrera-Gomez
E42.03	Auger Electron and X-Ray Photoelectron Spectroscopy	David Wieliczka
E42.06	SIMS	James Ohlhausen
E42.08	Ion Beam Sputtering	Arun Devaraj
E42.13	Vacuum Technology	Stuart Tison
E42.14	STM/AFM	Vacant
E42.15	Electron Probe Microanalysis/Electron	John Small
E42.92	US TAG ISO/TC 201 *Surface Chemical Analysis*	Mark Engelhard
E42.94	US TAG ISO/TC 112 *Vacuum Technology*	Stuart Tison
E42.96	US TAG ISO/TC 202 *Microbeam Analysis*	Scott Wight

1977 ASTM Round Robin Demonstrates the Need for Standard Calibration Procedures for AES & XPS

***Journal of Electron Spectroscopy and Related Phenomena*, 10 (1977) 359-388**

SURFACE CHARACTERIZATION OF CATALYSTS USING ELECTRON SPECTROSCOPIES: RESULTS OF A ROUND-ROBIN SPONSORED BY ASTM COMMITTEE D-32 ON CATALYSTS

THEODORE E. MADEY, CHARLES D. WAGNER, and A. JOSHI

Data on these samples (SiO_2, and Al_2O_3) were received from 12 laboratories using XPS and 8 laboratories using AES. The results indicate that the standard deviation in reported AES and XPS absolute line positions is much greater than the precision of any one measurement, indicating a great need for standardization of static charge referencing. In addition, there was a large spread in reported intensity ratios for instruments having nominally the same transmission characteristics and even of the same manufacture. The results demonstrate a need for standard calibration procedures.

1979 Round Robin: Researchers were asking "How reliable are the XPS and AES measurements?"

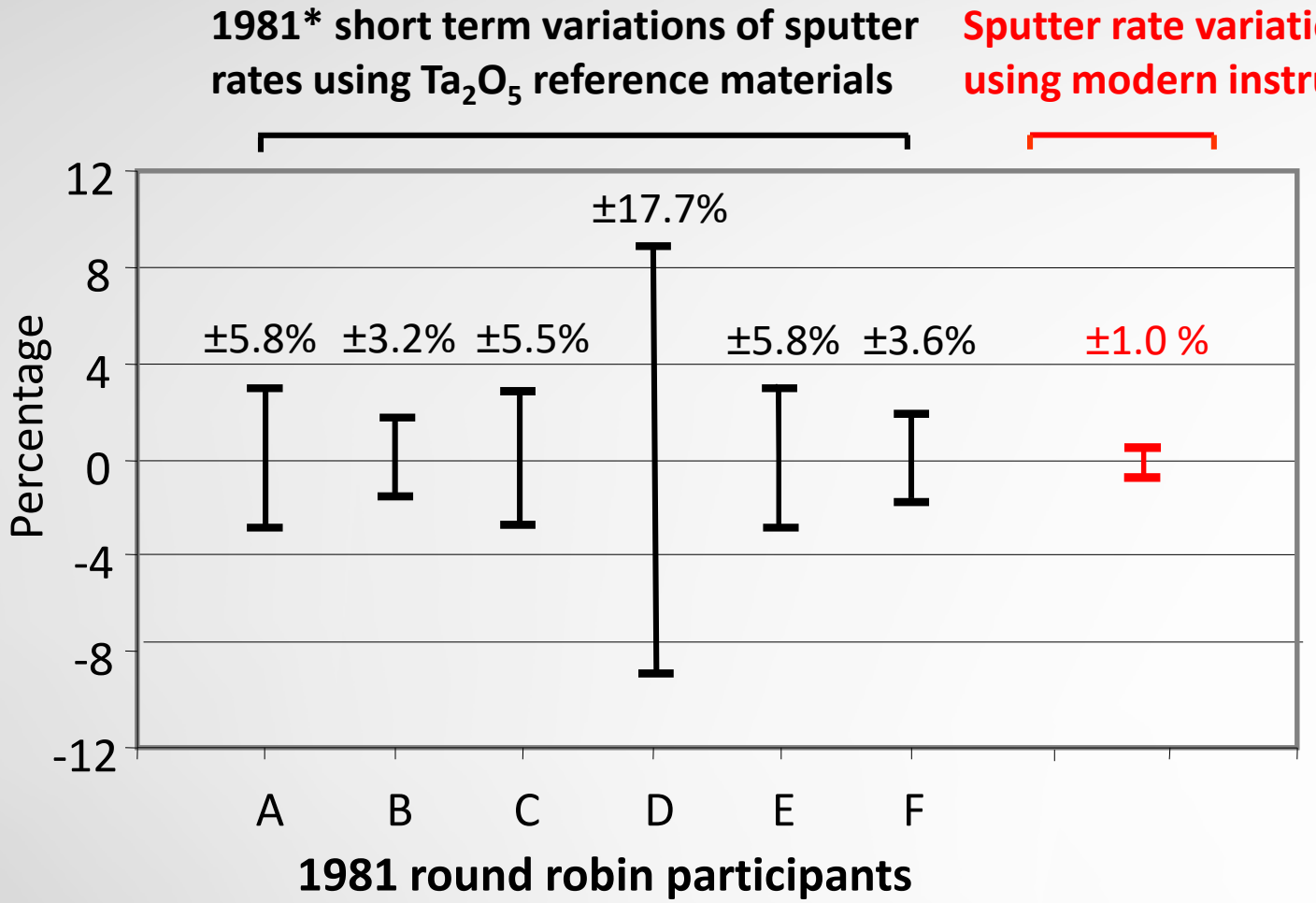
Journal of Electron Spectroscopy and Related Phenomena, 17 (1979) 361-403

RESULTS OF A JOINT AUGER/ESCA ROUND ROBIN SPONSORED BY ASTM COMMITTEE E-42 ON SURFACE ANALYSIS Part 1. ESCA results

C. J. POWELL, N. E. ERICKSON, and T. E. MADEY

We report results of a round robin involving binding-energy (BE) and relative-intensity measurements on high-purity gold and copper by X-ray photoelectron spectroscopy. These results were obtained on 38 different instruments manufactured by 8 companies. We found that the spread in reported BE values was typically greater than 2 eV while the spread in intensity ratios from cleaned samples was typically a factor of ten.

Comparison of short-term sputter rate variations of Ta_2O_5 (1981) with the short sputter rate repeatability of SiO_2 with a modern instrument



1981 E42.08 Six participants in the A. J. Bevolo, round robin study of sputter yields concluded that Ta_2O_5/Ta of known thickness can be used as a sputter rate standard with a precision of about **±5%**

2008 ILS- E42-08
"Consistence and Reproducibility of Sputter Rate Measurements"

Sputter rate measurements using SiO_2/Si using a modern instrument has demonstrated that the sputter rate reproducibility can be **±1%**

*Reference: A. J. Bevolo. "Results of a Ta_2O_5 Sputter Yield Round Robin," *Surf. Interface Analysis,* V3, N6 (1981) 240-242.

Contributions from researchers and vendors have helped solve many of these problems and make advances possible

ISO TC 201 Surface Chemical Analysis ‡1991

Committee Manager: Dr Satoshi Gonda

Chairperson: Dr Hidehiko Nonaka

‡ Year Established

TC 201 Sub-Committees (9 SC's, 3 WG's)		Sub-Committee Chairs
SC1	Terminology	Dr. Alexander Shard (2013-2021*)
SC2	General Procedures	Dr. Justin Gorham (2021-2023**)
SC3	Data Management and Treatment	Dr. Graham Cooke (2020-2022)
SC4	Depth Profiling	Dr. Takaharu Nagatomi (2021-2023*)
SC6	Secondary Ion Mass Spectrometry	Prof. Ian Gilmore (2021-2023*)
SC7	Electron Spectroscopies	Dr. Adam Bushell (2021-2023**)
SC8	Glow Discharge Spectroscopy	Dr. Peter Robinson (2021-2023**)
SC9	Scanning Probe Microscopy	Dr. Sang-Joon Cho (2018-2023**)
SC 10	X-ray Reflectometry and X-ray Fluorescence Analysis	Prof. Laura Depero (2016-2021**)
SG 1	Nano-materials characterization	
WG 4	Surface characterization of biological materials	
WG 5	Optical interface analysis	

The final year of *extended term/ **6-year limit

ASTM International Standards on X-Ray Photoelectron Spectroscopy

Practices for:

E2108-16 Calibration of the Electron Binding-Energy Scale of an XPS Spectrometer

E1217-19 Determination of the Specimen Area Contributing to the Detected Signal in AES and XPS Spectrometers

E996-19 Reporting Data in AES and XPS

Guides for:

E995-16 Standard Guide for Background Subtraction Techniques in AES and XPS

E1523-15 Charge Control and Charge Referencing Techniques in XPS

E1828-14 Handling of Specimens Prior to Surface Analysis

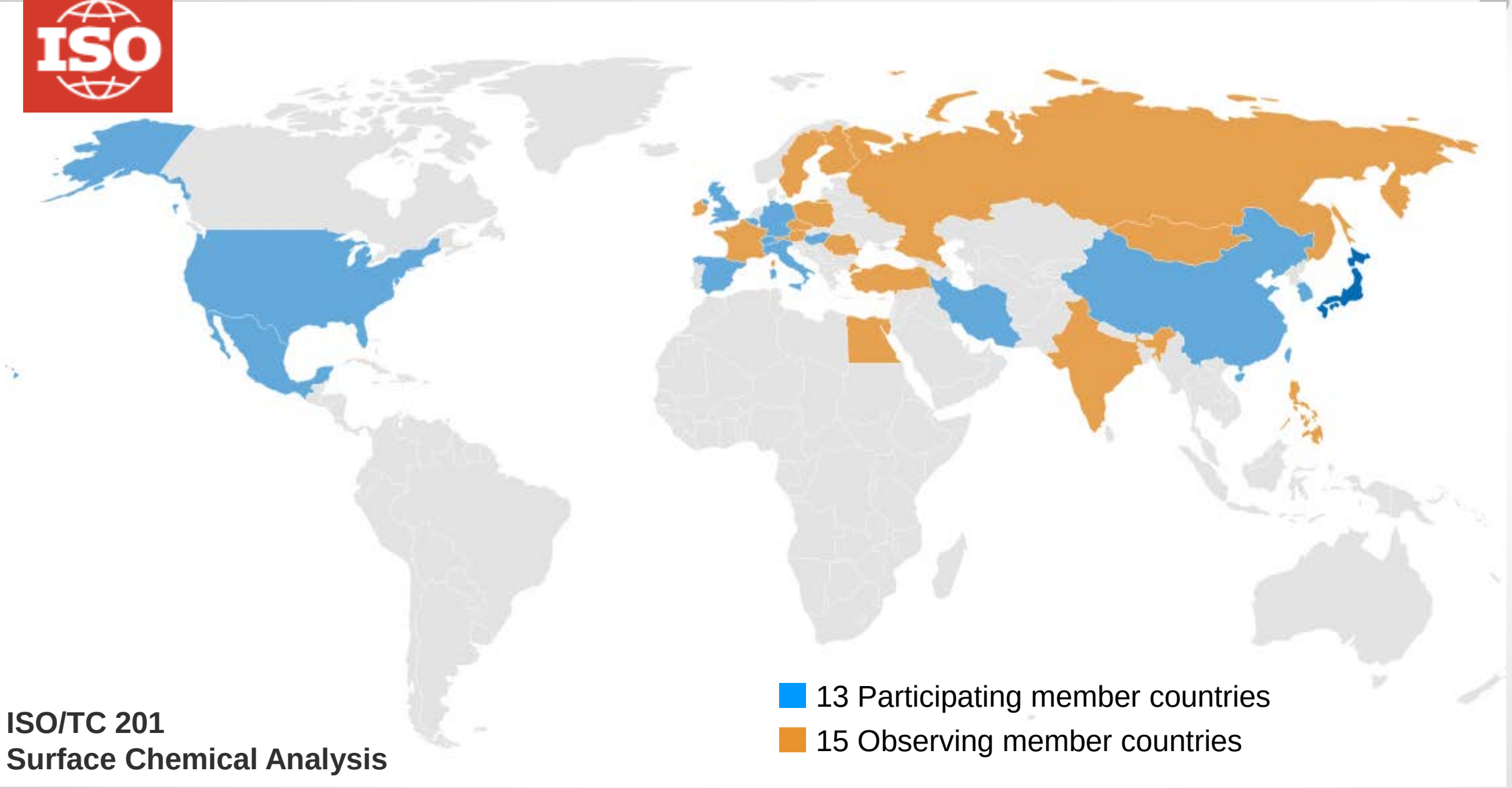
E1078-14 Specimen Preparation and Mounting in Surface Analysis

E1016-12 Literature Describing Properties of Electrostatic Electron Spectrometers

E2735-14 Standard Guide for Selection of Calibrations Needed for XPS Experiments

9 helpful ASTM documents related to X-ray Photoelectron Spectroscopy

Geographic distribution of ISO TC 201 member countries



ISO/TC 201
Surface Chemical Analysis

ISO TC 201 has 13 participating and 15 observing members

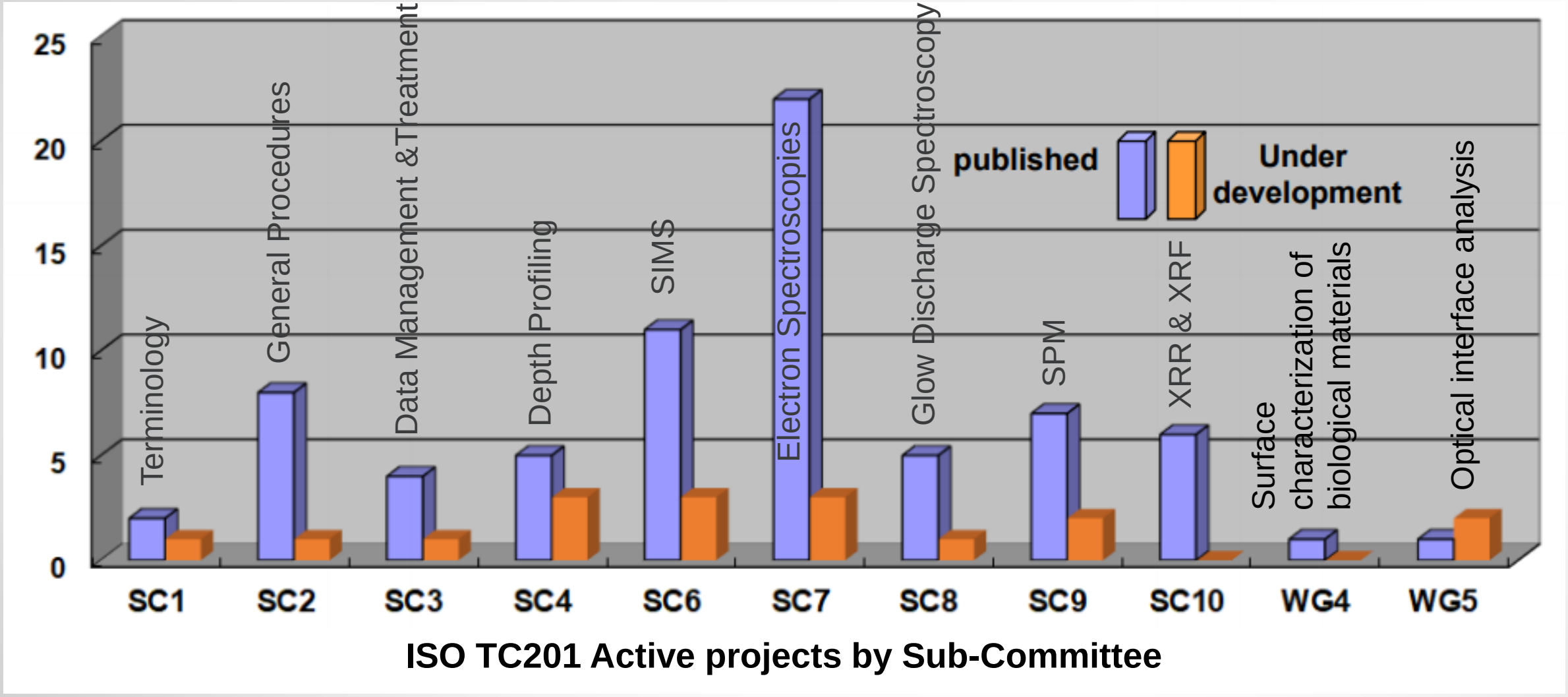
PARTICIPATING MEMBERS (13)

COUNTRY/TERRITORY	ACRONYM
Belgium	NBN
China	SAC
Germany	DIN
Hungary	MSZT
Iran, Islamic Republic of	ISIRI
Italy	UNI
Japan	JISC
Korea, Republic of	KATS
Mexico	DGN
Spain	UNE
Switzerland	SNV
United Kingdom	BSI
United States	ANSI

OBSERVING MEMBERS (15)

COUNTRY/TERRITORY	ACRONYM
Austria	ASI
Czech Republic	UNMZ
Egypt	EOS
Finland	SFS
France	AFNOR
Hong Kong Special Administrative Region of China	ITCHKSAR
India	BIS
Ireland	NSAI
Mongolia	MASM
Philippines	BPS
Poland	PKN
Romania	ASRO
Russian Federation	GOST R
Sweden	SIS
Turkey	TSE

72 active standards produced in 29-years of TC201
18 standards under development, 2 NP's, and 10 PWI's



ISO TC201 Active projects by Sub-Committee

Manuscript that covers the organization, operation, and output of TC 201 over the past 20 years

“Development of Standards for Reliable Surface Analyses by ISO Technical Committee 201 on Surface Chemical Analysis”

C. J. Powell, R. Shimizu, K. Yoshihara and S. Ichimura
Surface and Interface Analysis (wileyonlinelibrary.com) DOI 10.1002/sia.5684
October 2014

ISO Standards on X-Ray Photoelectron Spectroscopy

10810:2019	Guidelines for analysis
13424:2013	Reporting of results of thin-film analysis
14187:2020*	Characterization of nanostructured materials
14701:2018	Measurement of silicon oxide thickness
15470:2017	Description of selected instrumental performance parameters
15472:2010	Calibration of energy scales
16129:2018	Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer
18118:2015	Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials
18392:2005*	Procedures for determining backgrounds

* Technical Report

ISO Standards on X-Ray Photoelectron Spectroscopy “continued”

18554:2016	Procedures for identifying, estimating and correcting for unintended degradation by X-rays in a material undergoing analysis by X-ray photoelectron spectroscopy
19318:2004	Reporting of methods used for charge control and charge correction
19668:2017	Estimating and reporting detection limits for elements in homogeneous materials
19830:2015	Minimum reporting requirements for peak fitting in X-ray photoelectron spectroscopy
20903:2019	Methods used to determine peak intensities and information required when reporting results
21270:2004	Linearity of intensity scale
24237:2005	Repeatability and constancy of intensity scale
29081:2010	Reporting of methods used for charge control and charge correction

ASTM Standard Guide for Selection of Calibrations Needed for X-ray Photoelectron Spectroscopy

ASTM International

E2735−14 Standard Guide for Selection of Calibrations Needed for X-ray Photoelectron Spectroscopy (XPS) Experiments

X-ray photoelectron spectroscopy (XPS and ESCA)

The purpose of this guide is to assist users and analysts in selecting the standardization procedures relevant to a defined XPS experiment

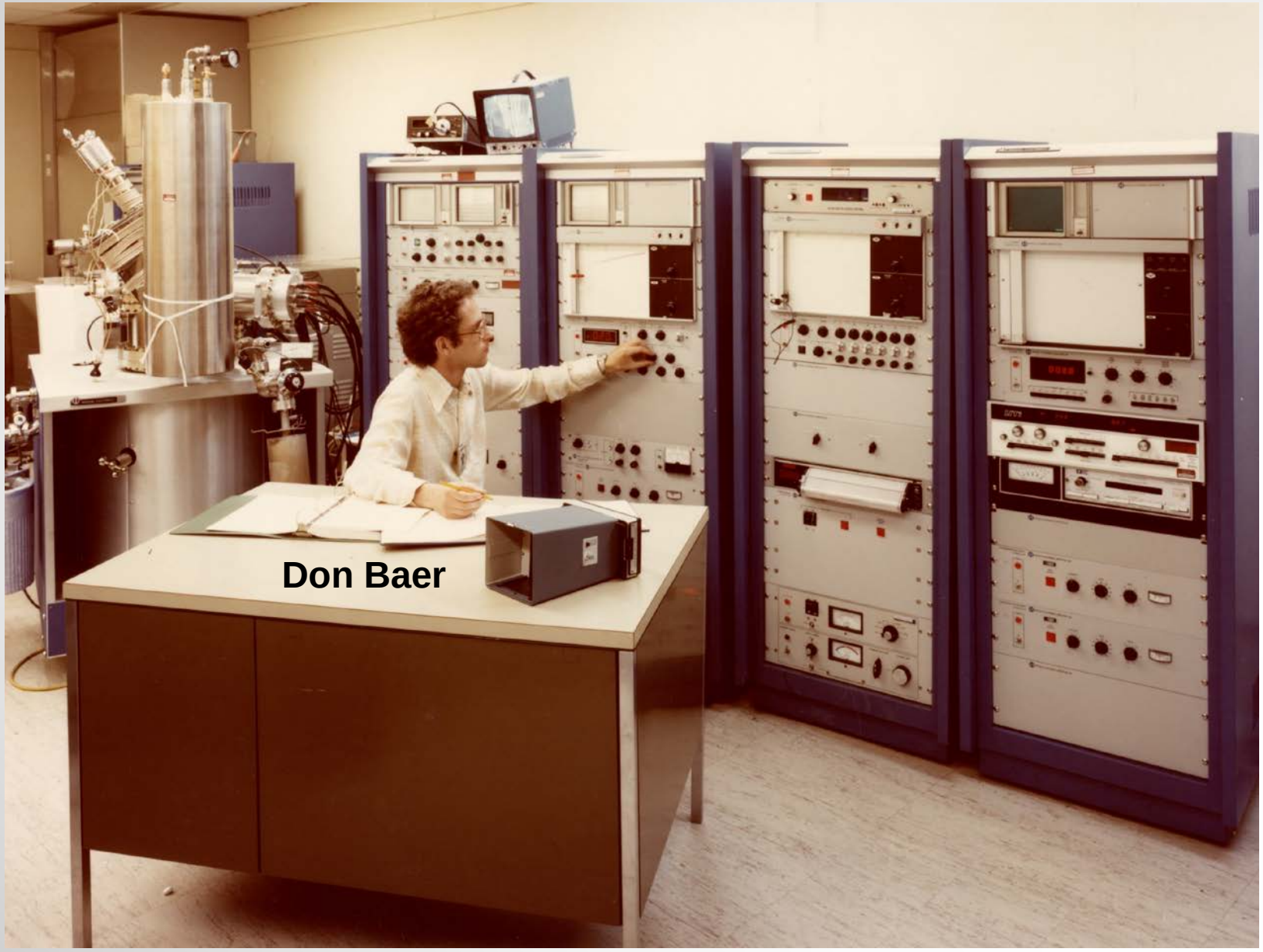
E2735-14 Standard Guide for Selection of Calibrations Needed for XPS Experiments

TABLE 1 Recommended Calibrations for Defined Experiments

Instrument Calibration and Checks	ASTM Standard	ISO Standard	Additional Sources	Elemental Compositior	Chemical States	Low Leve Detection	Quantifi-cation	Layer Thickness	Nano-structures
General System Check			Local Method	XX	XX	XX	XX	XX	XX
Sample Preparation	E1829 E1078	18116 18117		X	X	X	X	XX	X
Binding Energy	E2108 E1523	15472 19318		XX	XX		X		X
Intensity Repeatability & Consistency		24237		X	X	XX	XX	X	X
Intensity/Energy Response Function			NPL Software				XX	X	XX
Linearity of Intensity Scale		21270 18118		X	X		XX	X	XX
Peak Intensities	E995	18392 20903	Local Method	X	X	XX	X	X	X
Ion Gun and Sputter Rates	E1577 E1127 E1634	15969 22335 14606 14701	BCR 261					XX	XX
Depth Resolution	F1577 E1127 E1634 E1636	14606	BCR 261 NIST SRM 2135c					XX	XX
Analysis Area	E1217	19319		X			X	X	XX
Lateral Resolution		18516		X			X	X	X
Data Reporting	E996	14979		X	X	X	X	X	X

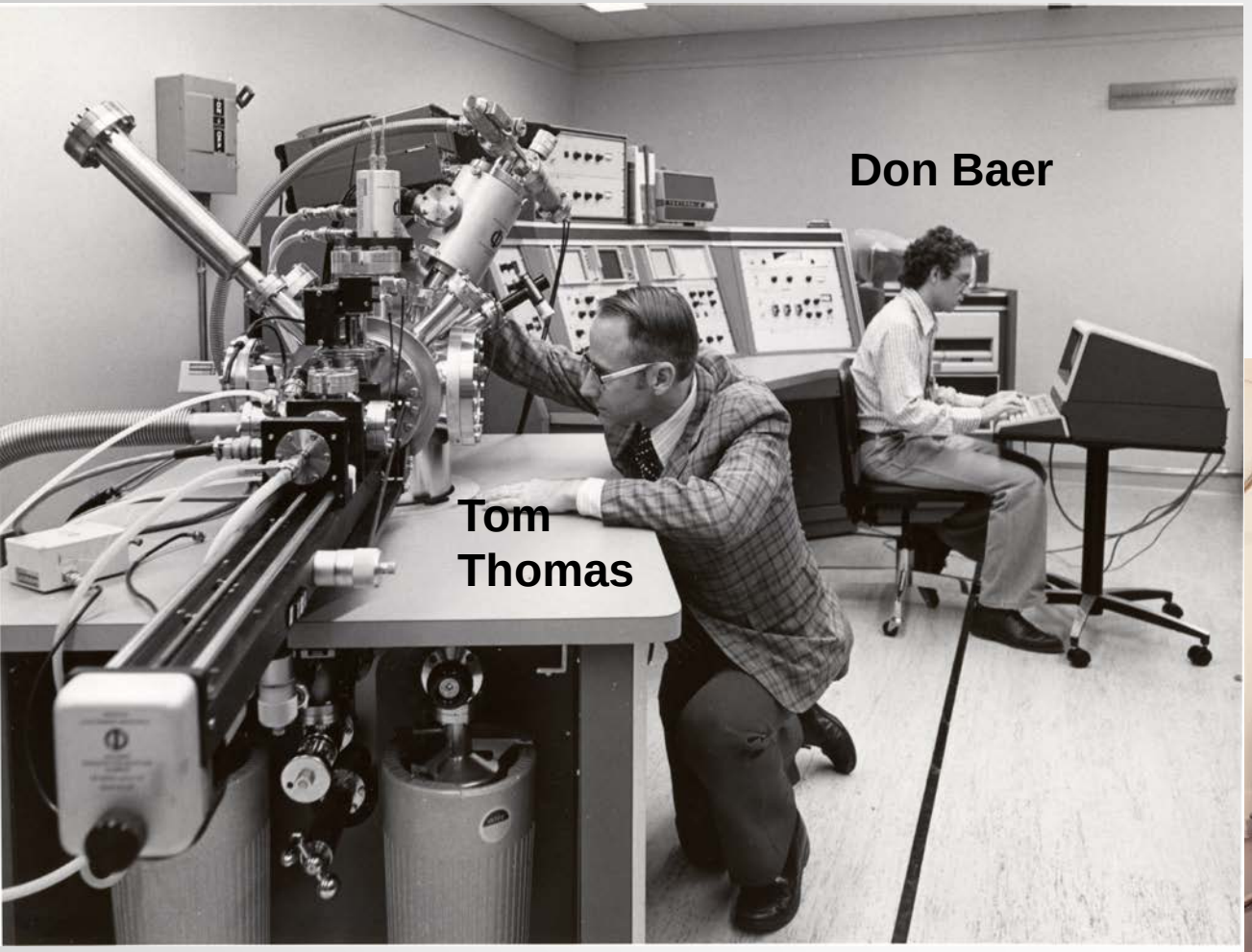
X = generally important XX = generally very important

Don Baer using a Physical Electronics Auger/SIMS 545 in 1977



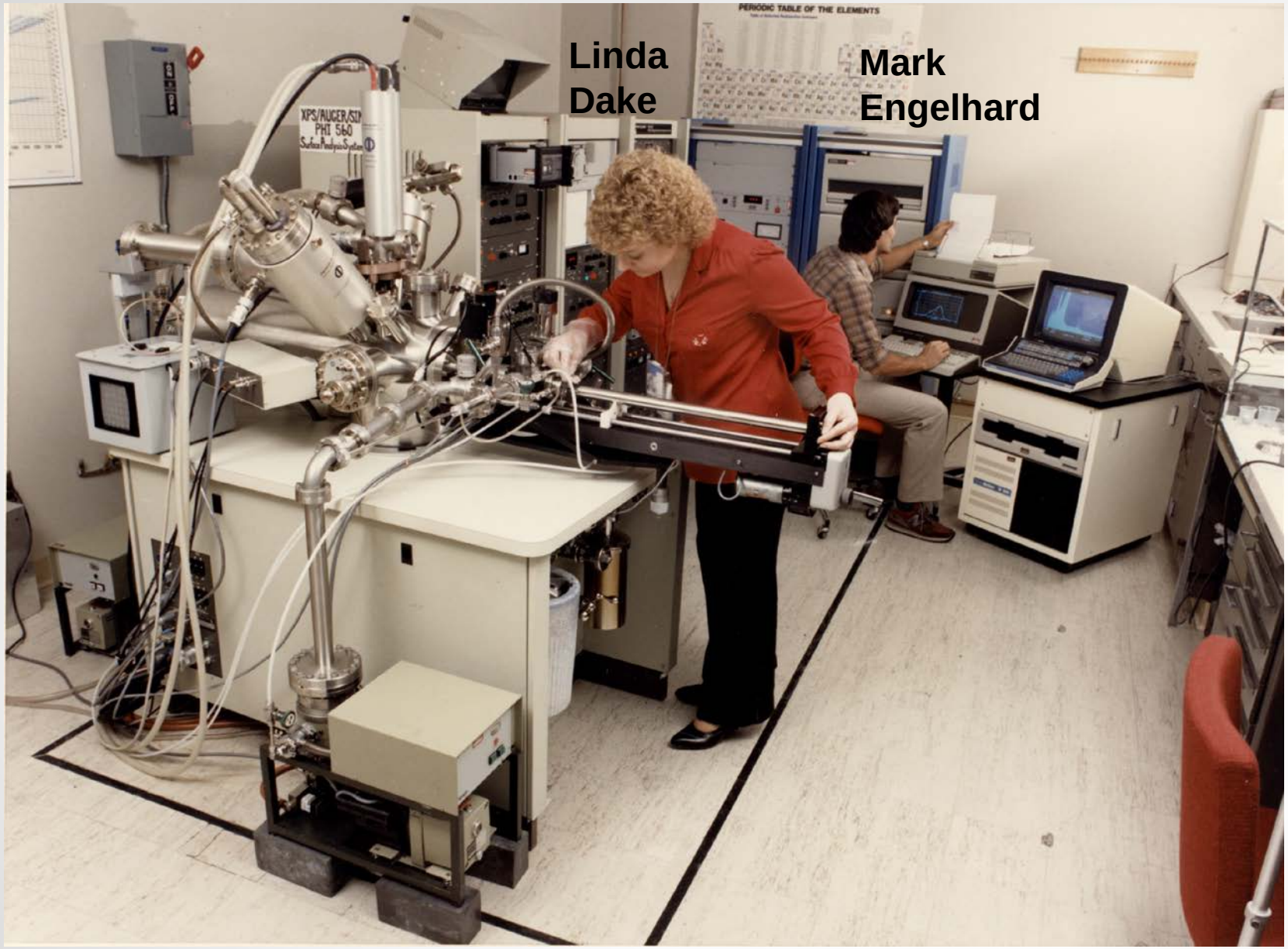
- Analog control with no computer.
- Top mount sample carousel.
- Required breaking vacuum to load a set of samples.
- Back fill with Ar to sputter
- Data plotted on chart paper.
- Was eventually upgraded to limited control using a Z80 computer.

Physical Electronics 550 XPS/AES/SIMS in early 80's



- Digital Equipment PDP 11/34 computer for data acquisition.
- Most of the system operation was analog control.

Physical Electronics 560 XPS/AES/SIMS in the late 80's



Problems with early XPS instruments

- Observed binding energy scale drifts

ASTM E2108-16 Calibration of the Electron Binding-Energy Scale of an XPS Spectrometer

ASTM E2735-14 Standard Guide for Selection of Calibrations Needed for XPS Experiments

ISO 15472:2010 Calibration of energy scales

ISO 16129:2018 Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer

Problems with early XPS instruments continued

- Quantification?

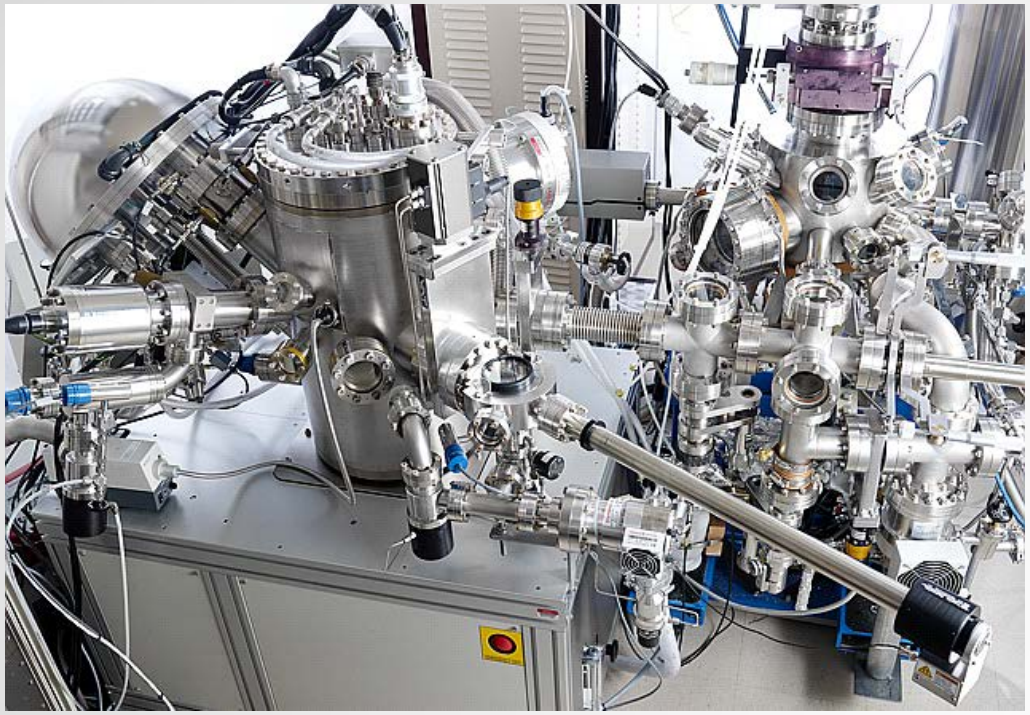
  ISO 18118:2015 Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

  ISO 16129:2018 Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer

- What is the area of analysis?

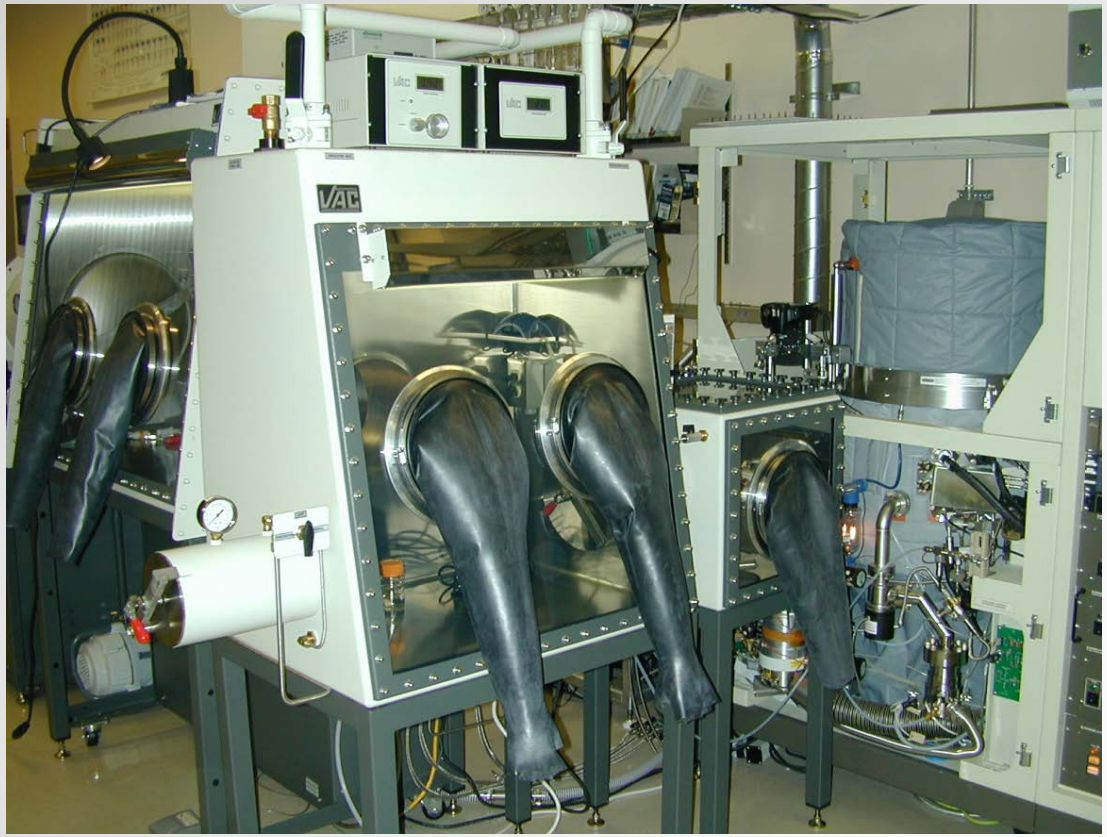
  ASTM E1217-19 Determination of the Specimen Area Contributing to the Detected Signal in AES and XPS Spectrometers

Physical Electronics VersaProbe



Kratos Analytical AXIS 165 XPS 1997

Physical Electronics Quantera XPS



Recently upgraded to a Quantera Hybrid

Kratos Analytical AXIS DLD XPS

Conclusions

- Surface analysis is in widespread use for the solution to a wide variety of scientific and technological problems
- Although the analytical techniques in common use are based on relatively simple concepts, the instrumentation is often complex and the analysts are required to make many choices. i.e. modes of instrument operation, data acquisition, and data analysis
- Analysts continue to face many challenges
- Analysts are often under pressure to increase productivity
- ASTM E42 and ISO TC201 produces many helpful documents that are educational and help the analysts to improve the quality and repeatability of data

ENVIRONMENTAL MOLECULAR SCIENCES LABORATORY

XPS Gone Bad!

Quantitative Surface Analysis Meeting (QSA-16)

Mark Engelhard mark.engelhard@pnnl.gov

ASTM E42 Surface Analysis Community Forum
Friday, November 6, 2020

Issues affecting credibility in XPS analysis and interpretation

Strategies Going Forward

D. R. Baer
don.baer@pnnl.gov
With input from many others

SCIENTIFIC INNOVATION THROUGH INTEGRATION

Summary So Far

- A high degree of badly collected, analyzed or reported XPS data in the literature
  - The issue is **much broader than XPS**
    - *Proliferation of faulty materials data analysis, Linford et al. Microsc. Microanal. 26 (2020))*
    - *Survey of >1500 scientists identified reproducibility as a significant problem*
- For roughly 40 years ASTM and ISO committees have been producing guides and standards that provide important information regarding quality XPS data
  - This information is either unknown or ignored by many XPS users
- To address solutions, need to identify the sources and causes. Many problems seems to come from different types of XPS users:
  - Rushed or **overwhelmed** analysts,
  - New and **inexperienced** users who want to do a good job
  - "**Casual**" XPS users who want results, but have no real interest in the method

Summary So Far

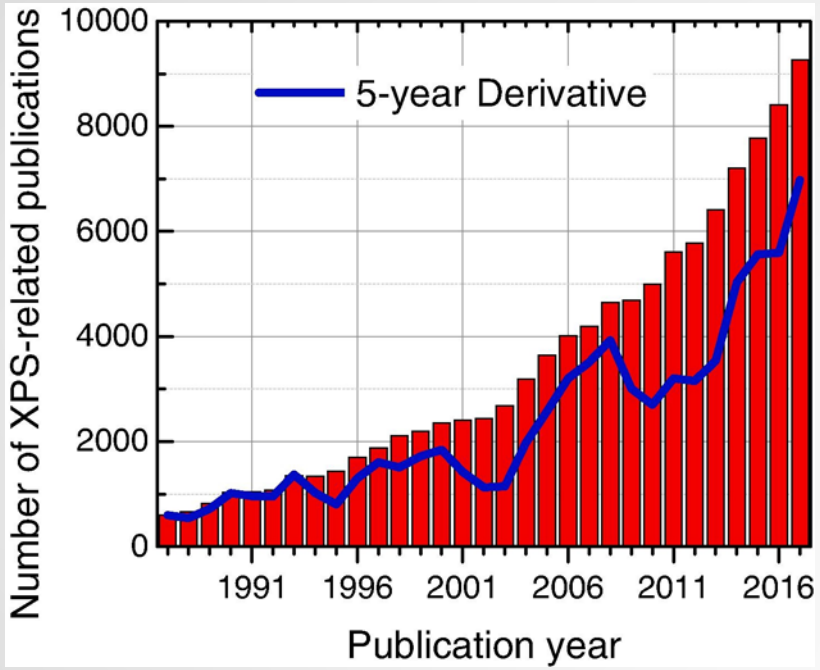
- A high degree of badly collected, analyzed or reported XPS data in the literature
  - The issue is **much broader than XPS**
    - *Proliferation of faulty materials data analysis, Linford et al. Microsc. Microanal. 26 (2020))*
- For
  - sta
- To
  - see
    - Rushed or **overwhelmed** analysts,
    - New and **inexperienced** users who want to do a good job
    - "**Casual**" XPS users who want results, but have no real interest in the method

This talk reports on some efforts to address this issue and seeks input on what more should/could be done?

We all have roles in addressing the issue which impacts the credibility of science and XPS

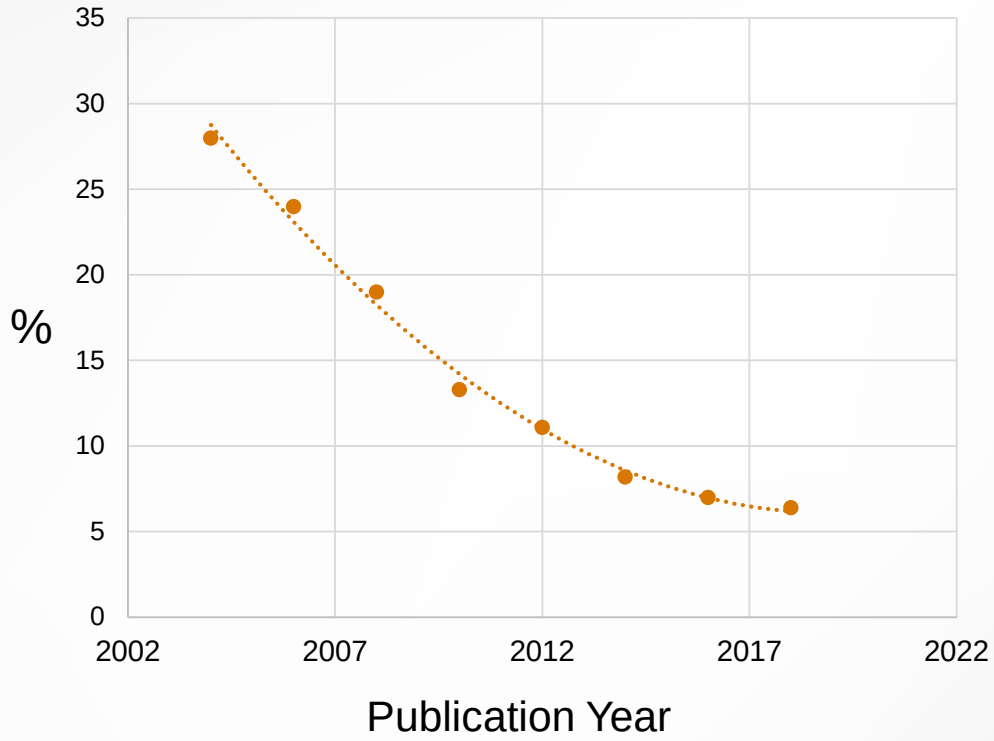
Usage – significant growth outside of the surface analysis community

Papers published with XPS as key word



G.Greczynski, L.Hultman, 2019
https://doi.org/10.1016/j.pmatsci.2019.100591

% Published in 17 “surface” Journals



J. Counsell ***Kratos Analytical Ltd.***

Graphs extracted from a talk Jonathan Counsell gave at the North American Kratos User meeting

Usage – significant growth outside of the surface analysis community

Number of XPS-related publications

From Jonathan Counsell [*with my inserts*]: I think the current issues can be summed up with these observations:

- XPS instruments are now easy to use. Data is abundant.
- Scientists [*especially those focused on applications*] are becoming increasing disinterested with the technique and the issues: They want a quick answer.
- Typically the XPS instrument is run as a facility meaning data is farmed out to specialists in applied areas who are not familiar with spectroscopy.
- There are not enough experts.
- Refereeing [*especially of XPS data in applied papers*] is poor and there is a lot of bad info (I'll use the term fake news!) out there especially in [*published papers and*] the q&a chats on Researchgate!

Graphs extracted from a talk Jonathan Counsell gave at the North American Kratos User meeting

Results of 2018 AVS survey on reproducibility
Recommended actions and useful tools

Important ways to help issue

- Informing AVS members of issue
- Providing processes and protocols
- Provide education opportunities

Useful Tools

- Experimental guides, standards & protocols (GPS))
- Sample preparation GPS
- Reporting computation and experimental details GPS
- Checklists for reviewing
- Tools for open science

- 65% responders indicated significant problem
- Incomplete reporting, reviewing and untrained staff were problems

- AVS needs to provide information and tools including:
  - Guides, protocols and standards **for conducting** experimental and computational research
  - Guides **to reporting** computational and experimental details
  - **Checklists** for reviewing

Two types of issues

1. Things we know, but that are frequently not done or reported
   - Instrument set up and operation
   - Good analysis practices
   - Good reporting practices
2. Things we know we do not fully understand or for which we don’t have full control.
   - Simple accurate method of BE referencing for insulators
   - Full understanding of background signals
   - How to measure all intrinsic peak of some simple and many complex photoelectron peak intensities for quantification

Two types of issues

1. Things we know, but that are frequently not done or repor
   - Instrument set up and operation
   - Good analysis practice
   - Good re
2. full

First focus is on what we know, but is not being done (most of this talk)

Need also to communicate that there are unknowns, limitations, and problem areas subject to ongoing or needed research

ic peak of some simple and many complex
n peak intensities for quantification

Addressing the issue - Many analysts working to provide tools for new and inexperienced XPS users, but we all have roles

- **Inform/Recognize** - Useful for all of us to **learn to recognize problem data and analysis**
  - *Last year we created a poster with more than 30 flaws as a "where's Waldo" type of test.*

Common Errors in XPS Analysis and Peak Fitting: *Can you identify the problems?*

George H. Major,[1] Christopher D. Easton,[2] Thomas R. Gengenbach,[2] Mark H. Engelhard,[4]
William Skinner,[3] Donald R. Baer,[4] Matthew R. Linford[1]
[1]Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT 84602, US A, [2]CSIRO Manufacturing, Ian Wark Laboratories, Clayton, Victoria, Australia
[3]Future Industries Institute, University of South Australia, Mawson Lakes 5095, Australia, [4]EMSL, Pacific Northwest National Laboratory, Richland WA, USA

X-ray photoelectron spectroscopy (XPS) is the most popular method for chemically analyzing surfaces. XPS spectra have layers of information that can be extracted with proper analysis. Information ranges from a rudimentary understanding of the components (elements) present in a material to advanced peak fitting and background analysis that reveal chemical states and sample morphologies. Although there are many examples of good analysis and XPS peak fitting with appropriate consideration of the physical and chemical nature of the relevant spectra, increasingly there are inadequate or significantly incorrect analyses of spectra in the literature.*

Use the answer sheets provided to indicate the issues you identify. Exchange your response sheet for a key to issues we have identified or send a request to georgemviolin@gmail.com

**This poster has both examples of XPS data extracted from the literature and examples artificially created to highlight types of issues commonly identified in published papers. We have attempted to obfuscate the origins of the erroneous spectra (and not distinguish our synthetic examples from those appearing in publications) in order to avoid the poster being about authors or publications but rather about data interpretation errors.*

Common Errors in XPS Found in the Literature

Examination of XPS data in hundreds of papers has been used to identify frequent errors in the literature (in the data, presentation, analysis, peak fitting, and data reporting). While some issues are superficial and do not drastically impact conclusions, others have significant potential to influence the results, and others show analysis that is fundamentally flawed. Disturbingly, some type of issues have been identified in more than 25% of the publications. Common errors include:

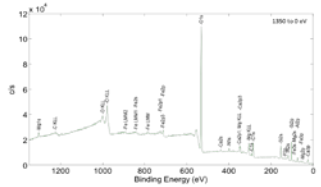
(i) No indication that instrument performance or appropriate calibration was completed before analysis.
(ii) No consideration of the relevant physics and chemistry of the spectra when doing analysis, peak identification (including satellite and multiplet splitting), or peak fitting
(iii) Not plotting the data according to the international convention, i.e., binding energy increasing to the left
(iv) Presenting and interpreting data that is far too noisy to be useful
(v) Labeling noise as chemical components
(vi) Not showing the original data -- only

Examples: Take a sheet and identify as many errors as you can in these examples.

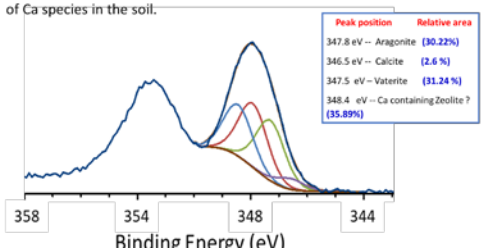
In general, the text below is a paraphrasing of the writing in the original documents.

A) The Chemistry of Ca in Soil - Soil chemistry plays an important role in agriculture and in carbon cycling. It can be useful to understand the nature of mineral phases and how they change with time and environmental conditions. We have applied XPS to determine the nature of Ca in the soil.

The survey spectrum shown in Fig 1 shows the general nature and complexity of the soil.



The high energy resolution spectrum of the Ca 2 p region is shown here. The Ca peaks were fit based on reference data published in the literature and peak ratios were used to identify the relative amounts of Ca species in the soil.



B) Stability of Solar Cell Materials - Although solar cells have significantly improved efficiency, long term stability remains a significant issue. Organic-inorganic halide perovskites have rapidly grown as favourable materials for photovoltaic applications, but accomplishing long-term stability is still a major research problem. This work attempts to demonstrate a new insight on instability by examining degradation factors in $CH_3NH_3PbI_3$ perovskite solar cells aging with time in open air.

XPS was used to investigate the compositional changes caused by device degradation over a period of 1000 h. XPS spectra confirm the migration of metallic (In) ions from the bottom electrode (ITO) as a key factor causing the chemical composition change in the perovskite layer besides the diffusion of oxygen.

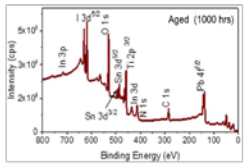


Figure 1. XPS survey spectrum of the perovskite film after the aging of (c) 1000 h in open air under the room temperature.

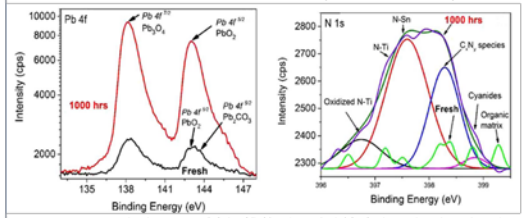
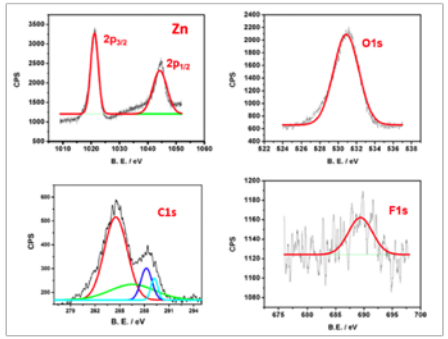


Figure 2. Core level XPS spectra (of Pb 4 f (left) and N 1 s (right) for fresh sample and 1000 h aged samples.

C) Designing materials with improved photocatalytic activity - Although ZnO has important photocatalytic properties, it is often modified to improve performance and to overcome the limitations of the pure oxide. This work seeks to obtain improved properties by encapsulating ZnO spheres with fluorinated reduced graphene oxide. XPS measurements were performed to verify the composition of the system and identify the chemical states of the elements present.



The strong peak in the C1s at 284.5 eV corresponds to C-C and C=C carbon in graphene as well as carbon bonded to hydrogen. The 288.3 eV and 288.3 eV peaks are assigned to carbon singly and doubly bonded to oxygen, respectively. The peak at 289 eV is labeled as the carbon – fluorine peak. The O 1s peak at 530.2 eV

Addressing the issue - Many analysts working to provide tools for new and inexperienced XPS users, but we all have roles

- **Inform/Recognize** - Useful for all of us to **learn to recognize problem data and analysis**
  - *Last year we created a poster with more than 30 flaws as a "where's Waldo" type of test.*
- **Tool development** -ISO TC 201 Surface Chemical Analysis and ASTM International Committee E42 on Surface Analysis are **busy developing standards and guides for surface analysis - *you can help develop and use them***
- **Journal guides** - A series of **guides for XPS has been developed for JVSTA** – call them to the attention of new users
  - https://avs.scitation.org/toc/jva/collection/10.1116/jva.2020.REPROD2020.issue-1
  - Practical guides for x-ray photoelectron spectroscopy: First steps in planning, conducting, and reporting XPS measurements JVSTA 37, 021401 (2019)
- **Self and Peer Review - Review more** papers**, but** feel free to **only** review the parts with your expertise
  - Looking to develop a guide for reviewers on evaluation of XPS data and reporting of data
  - You are your own peer reviewer – do the best work you can
- **Vendors** - Publish instrument characteristics and develop **smarter software**
  - Help with data reporting
  - Open discussion about limitations and development of Expert systems to help with analysis

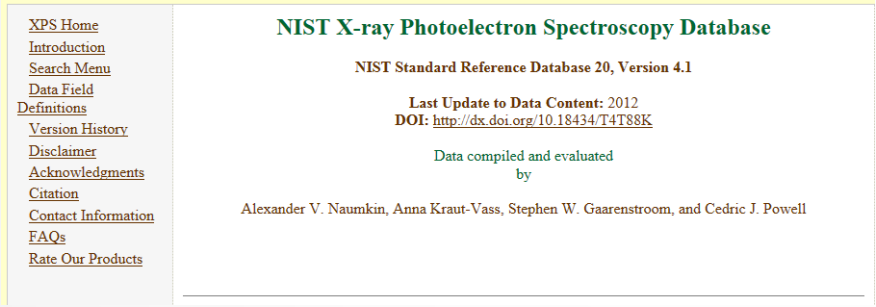
Slide from Jonathan Counsell

Many useful XPS - Resources

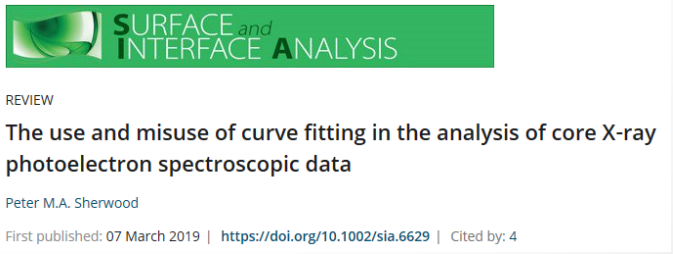
Resources to (new) users

Books

Websites



Publications



The onus should be on the members of the surface analysis community AND the instrument (tool) manufacturers to provide better direction.

The MAJOR MANUFACTURERS should work together to create a guide.

Tools - JVSTA 2020 Special Topic Collection: Reproducibility Challenges and Solutions with many XPS guides

- Topics covered: **generic XPS** and specific **application areas** (not all only XPS)

Information and Planning
- XPS Standards, XPS introduction
- Sample handling

Instrument set up and operation
- Instrument checks, spectrometer response
- Lateral resolution
- Charging and charge control

Peak identification, quantification and fitting
- Quantification and uncertainties
- XPS Backgrounds and Peak Fitting
- Carbon 1s information

Path Lengths and Depth Information
- Electron Path Lengths
- Depth information

Data and Reporting
- Data archiving and records
- Consistent terminology

Science and Technology Applications
- Atomic Layer Deposition
- Semiconductors
- Nanoparticles
- Catalysts
- Polymers
- Epitaxial Films and Heterostrutures

If you do not have access, sent me or the authors a request (don.baer@pnnl.gov)

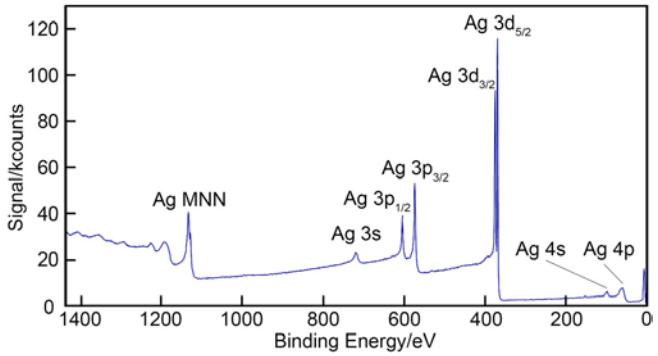
A soft bound version of the whole collection will be available for about $30 for a three-week period

https://avs.scitation.org/toc/jva/collection/10.1116/jva.2020.REPROD2020.issue-1

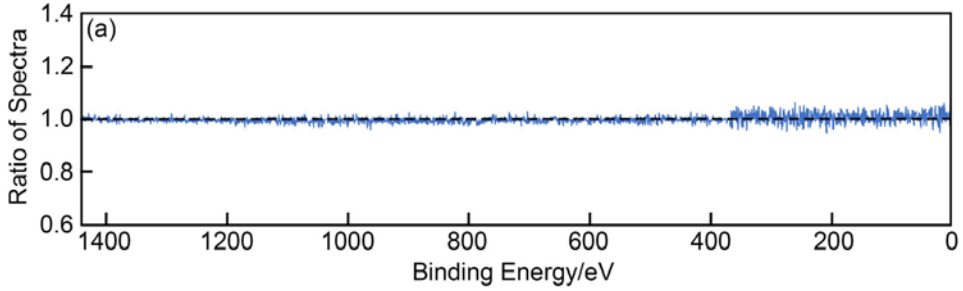
Highlight on just one of the collection guides: ***Procedure which allows the performance and calibration of an XPS instrument to be checked rapidly and frequently*** by John Wolstenholme

- The guide is based on comparison of instrument data collected on Ag and PET.
  - The method described enables rapid assessment of instrument status
  - A Microsoft Excel workbook is made available to assist in the comparison and highlight aspects of an instrument's performance

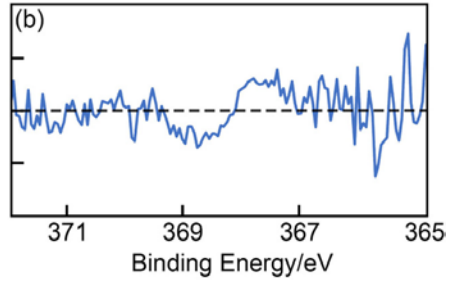
Ag survey spectrum



Ratio of two Ag survey spectra



Ratio of high-resolution spectra



Quality	Status
Spectrum Quality	
C contamination	OK
Detector noise	OK
Detector linearity	OK
Relative energy calibration	
Auger parameter	OK
Calibration gradients	OK
Peak shift	Check
Sensitivity	
Transmission function	OK
Ag $3d^{5/2}$	OK
Linearity	OK
Relative resolution	
Peak width (eV)	OK

J. Wolstenholme, JVSTA **38**, 043206 (2020)

Other actions that might be undertaken – these ideas come from multiple sources

- **Data and Analysis reporting**
  - Reporting Check List for users and reviewers: graded approach depending on use of data
  - Instrument information data files that can be referenced
- **Examples of common XPS errors**
  - Papers highlighting common errors by examples
  - Periodic posting of bad examples appearing on a website?
- **Enable users to validate their methods**
  - Create combinations of journal papers with data availability for user training and comparison
  - Interlaboratory comparisons studies to test new procedures and possibly ongoing data comparison studies to “calibrate” new users
- **Software and instrument operation advancements**
  - Highlight and record workflows and parameter settings
  - Expert systems as being developed by Jim Castle and SPECS

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  - Interlab ly ongoing data comparison studies to “calibrate” new users
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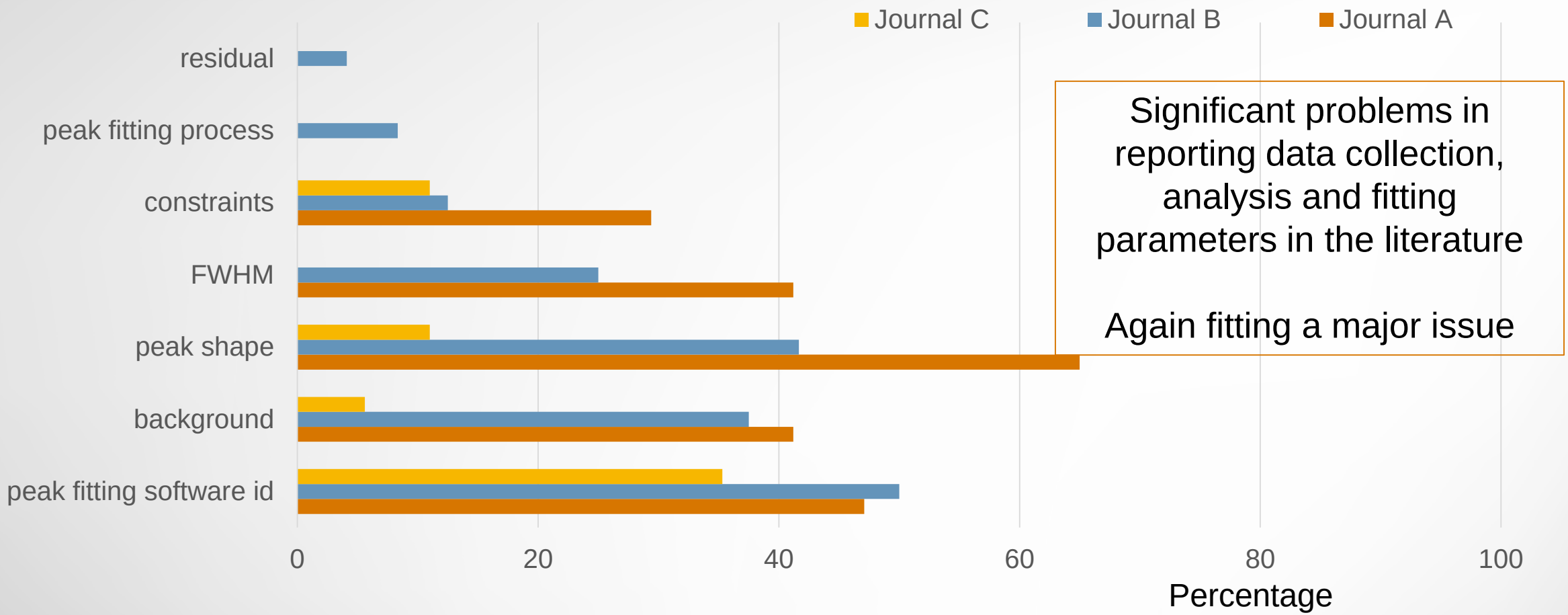
These will be expanded upon in the next few slides:

- Can we identify actions that might have significant impact?
- How might these be best done to optimize impact?
  - Standard, Guide, Publication, Reference, ASTM, ISO, AVS, VAMAS, other
- Are you interested and willing to help execute?

Status of data and reporting XPS measurements and results

Analysis by Karen Gaskell University of Maryland, AVS International Symposium 2019

Reporting Peak Fitting Parameters (data collected from 50 papers)



Significant problems in reporting data collection, analysis and fitting parameters in the literature

Again fitting a major issue

Minimum Reporting Requirements from ISO and ASTM Standards are **extensive**: Instrumental Parameters

- Manufacturer/Model of whole spectrometer or Parts
- Pass Energy
- X-ray source (x-ray energy / monochromator)
- *X-ray power*
- *X-ray radiation energy*
- *Acceleration voltage*
- *Electron emission current*

- *X-ray radiation energy*
- Energy step size
- *Sample anode distance (non-monochromated, if known)*
- *System pressure*
- *Acquisition time*
- *Angle of emission*
- *Start energy*

- *End energy or scan width*
- *Number of data points*
- Slit settings (if they affect instrument resolution)
- Area of analysis (if more than one size available, could be determined by x-ray beam size or analyzer collection area)
- Angular acceptance of analyzer (if this is changeable)
- Window material
- *Beam size at sample*
- *Stationary or scanned x-ray beam*
- Charge compensation (if used, type, approach for adjusting settings
- Lens mode

- *geometry of irradiation*
- Type of detector (Channeltrons/channel plates)
- Detector operation mode (scanned/snapshot)
- *Analyzer working radius (if hemispherical) and operating mode (ie FRR or FAT)*
- *geometry of irradiation*

List is too long to be used in many publications

Note that some of these are operator controlled and some more characteristic of an instrument should be known if instrument identified

Need to simplify and prioritize?

Helping with data and analysis reporting

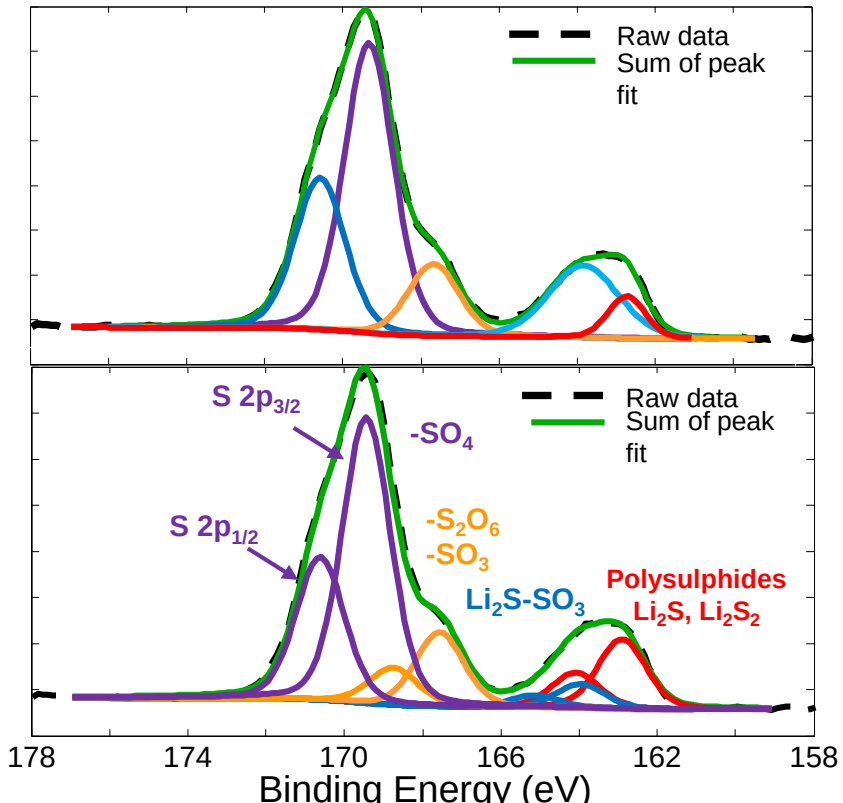
- Two ideas to simplify the reporting requirements and tailor them to the need and purpose
  - *Check List of parameters important for differing uses of XPS data*. Possible categories:
    - ***Credibility – appropriate use of XPS for measurement undertaken***
    - ***Replication – enough information that another researcher could conduct a similar experiment***
    - ***Repeatability – someone else could repeat the measurement in full detail***
  - *Publish basic instrument information that can be referenced to* shorten list of needed in a journal submission (Surface Science Spectra or as a data DOI).
    - ***Would need to identify what needed to be included in such a data record***
- How might these be implemented? How might we get journal editors and reviewers involved?

User education and assistance: i) Examples of common errors and ii) Operation and analysis software advances

- **Examples of common XPS errors**
  - Papers showing examples of both erroneous and appropriate analysis
  - Periodic web posting of example errors

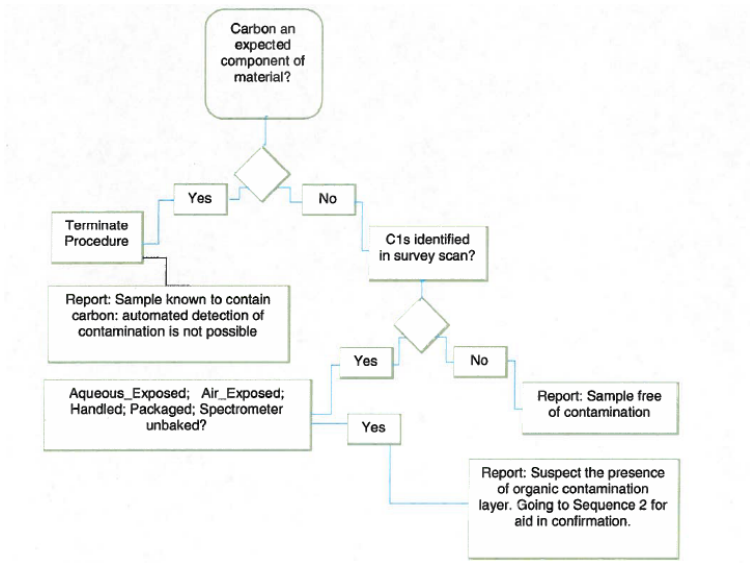
Erroneous and reasonable fits and peak identifications

Objective is to educate not point fingers



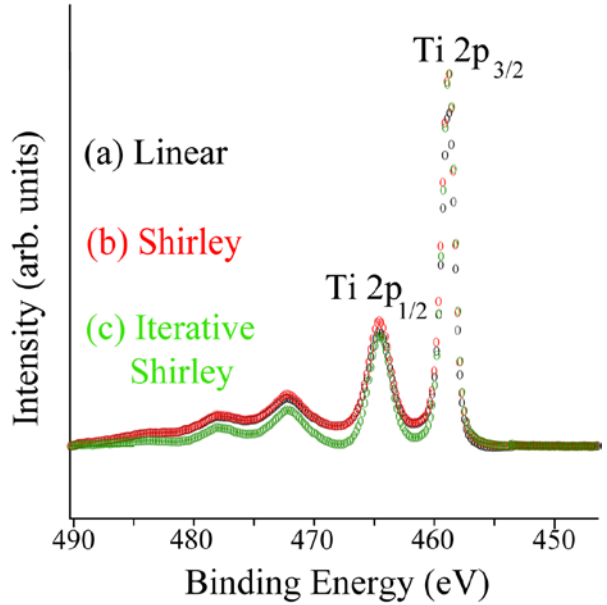
Operation and analysis software developments

- Highlight and record workflows and parameter settings
- Expert systems as being developed by Jim Castle, SPECS and ISO TC201: *Rules for Identification of, and Correction for, the Presence of Surface Contamination.*



Hands on experience: Enable users to compare and validate their methods and results

- **Allow user to compare their analysis to literature results** - Journal articles for which the data is available for user analysis

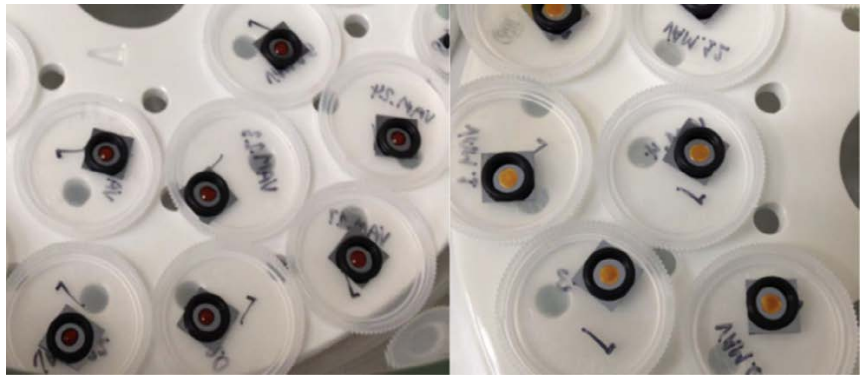


Ti data discussed in JVSTA background paper being published in SSS

Engelhard et al. JVSTA **38**, 063203 (2020) & SSS **27,** 024011 (2020)

Interlaboratory comparisons studies/samples

- ASTM and VAMAS can enable process/data comparison studies that enable technique advances and can "calibrate" new users



Nanoparticle deposition process from an NPL led interlaboratory comparison on reliable deposition for surface analysis

N.A. Belsey, et al. J. Phys. Chem. C 120 , 24070 (2016)**.**

Group discussion – Actions moving forward

- What are **your ideas to help address the problem**? **What tools or actions are needed?** What might have the **most impact?**
- **How could we do it?** ASTM, ISO, AVS, Manufacturer, Journal editors, ?????
- How can the information be **usefully distributed**?
- **Can you help?** What are you interested in working on?

Information and Announcements

- Feel free to send your ideas and suggestions at any time (don.baer@pnnl.gov and Chris Moffitt cmoffitt@kratos.com)
- To obtain information about obtaining a softbound copy of the reproducibility collection and the **three-week only** ordering period send me an email (don.baer@pnnl.gov).
- To get access to ASTM standards, join ASTM committee E42 on surface analysis and help review and develop them. (https://www.astm.org/COMMITTEE/E42.htm)
- To learn about ISO standards participate in your national ISO TC201 Surface Chemical Analysis - Technical Advisory Group/Mirror Committee. (mark.engelhard@pnnl.gov)

General Questions:

- from Jeff Terry to everyone: 11:15 AM
  Why will the journal collection only be availabe for a short time? [*This is somewhat answered in the email to which this note is attached. Because these are published papers a regular book is not an easy option. The AVS apparently has the option of requesting some collection copies to be printed and they will accept orders for that one printing. There are also limitations. Copies purchases are not to be purchased for resale.*]
- Will these slides be made available to attendees? [*They are or will be posted on the ASTM E42 Committee Web page.]*

Matt Linford – The Problem

- Jeff Fenton –
  The significant difference from unfitted to fitted seems to indicate a rapid drop off in knowledge in basic analysis to complex. Would you tend to agree with this and does this correlate with the growth in multiuser facility?
- ✓ from Manish Shinde to everyone: 11:48 AM
  is there any organization which can verify the individual analysis before submitting to journal
- from Uwe Scheithauer to everyone: 11:48 AM

  To much criticism of a referee produces to much work for the editor. Maybe therefore there exists a referee selection process, too.

- from Alberto Herrera-Gomez to everyone: 11:49 AM
  question for Matt: what would you think about a website dedicated to help people with their fittings?
- from Vince Crist to everyone: 11:49 AM
  JVSTA audience is too small. Need repeats in many different journals. Guide pubs have too few image examples. Professors are the main source of the problem. Editors are not being responsible. Need a book of actual spectra showing good and bad spectra
- from David Morgan to everyone: 11:49 AM
  I've often been called in by journal editors to review specifically suspect XPS data, do we feel there should be a lobby towards journal editors to specifically seek out a "characterisation expert"?
- from Robyn Goacher to everyone: 11:49 AM
  What recommendations would you make to journal publishers regarding review of the data? Is there a way that reviewers can be certified as expert in a topic?
- **from Stefan van Vliet to everyone: 11:50 AM**
  Is there a difference seen in the quality between primary use of XPS or supplementary use of XPS? [*This was not examined carefully, but the answer is likely yes. The surface and interface focused journal was better than the energy and general chemistry journals. The casual use of XPS seemed to be worse*]
- from Alexander Shard to everyone: 11:50 AM
  Did you assess the quality of elemental composition analysis as well as peak fits? [*No effort was made in that area. Focused only on published spectra – that was challenging enough. Clearly*

parameter reporting and quantitative analysis would have been appropriate, if challenging to analyze.]

- from william stickle to everyone: 11:51 AM
  Are most of the paers coming from the academy? If so, they authors are probably users of a tool, not the owners. Hence the folks that run the lab should beheld responsible as well. [*The operation model as many universities is to train people on instruments and allow them to do what they can/want with data. Often no resources for outside help.*]
- from Peter Cumpson to everyone: 11:52 AM
  This "red/yellow/orange/green" division looks really useful. Could some of the simpler "red" errors be highlighted by peak-fitting software - and "cut and paste" disabled?!?
- from william stickle to everyone: 11:53 AM
  people are using XPS like FTIR - put the samples in, get an answer and away you go!
- from Alberto Herrera-Gomez to everyone: 12:02 PM
  I believe that the issues with FTIR data analysis are even deeper. People conclude the presence of compounds with even less evidence
- from Alberto Herrera-Gomez to everyone: 12:04 PM
  in many institutions, the person in charge of the XPS equipment is a technician trained by the vendor
- from Vince Crist to everyone: 12:05 PM
  The XPS LIbrary website can be used to improve the issues, but it needs support and contributions. Currently getting 70-100 daily visitors from around the world.

Mark Engelhard – The Structure

- from Vince Crist to everyone: 11:56 AM
  Std Docs are only written in English. Where is French, Spanish, German, Russian, Australian?
- **from Vince Crist to everyone: 12:00 PM**
  Errors in Al2O3 and SiO2 and elements in Columns 1-4 suffer serious Surface Dipole Moment effects that move the C (1s) BE and even the metal oxides from those columns. No studies as yet.
- from Vince Crist to everyone: 12:08 PM
  Very few Professors will spent $100-$300 per copy that totals up to $5,000 to $10,000 for doc stds,
- from Vince Crist to everyone: 12:15 PM
  Many XPS systems around the world are old, or very old. Barely working, People still use old BEs and old SFs. They dont know and think their old calibrations are OK because Journal still accepts them.
- from John Grant to everyone: 12:19 PM
  Mark, the official English name for the acronym ISO is International Organization for Standardization, not International Standards Organization.
- from David Morgan to everyone: 12:19 PM
  There are still many laboratories which fail to check transmission functions, energy scale calibrations - despite some modern instruments having automated functions. This comes down in part to some academics/line managers/companies not understanding that a few hours (or a day) performing checks and calibrations is needed and not a waste of money

Don Baer – The Strategy

- from Vince Crist to everyone: 12:22 PM
  Editors and reviewers are not willing to be critical or to reject papers. They do not force professors to do due diligence.
- from Stefan van Vliet to everyone: 12:22 PM
  Are there summer-schools/winter-schools to train the new generation of XPS specialists?
- from Vince Crist to everyone: 12:23 PM
  Stefan, universities need to start normal grad courses dedicated to XPS, but universities will not
- from David Morgan to everyone: 12:24 PM
  Stefan, as the UK national facility for XPS this is something we are trying to organize in terms of experiment planning, system operation and data analysis. The latter we run many data analysis courses for example
- from Peter Cumpson to everyone: 12:24 PM
  Stefan and Vince: John Grant's courses fill a real need there
- from Jeff Fenton to everyone: 12:24 PM
  Stefan, Off the top of my mind AVS offers a number of short courses in XPS. Additionally John Grant offers XPS short courses. The latest AVS course list is available at: https://avs.org/education/short-courses/short-course-schedule/
- from Chris Moffitt to everyone: 12:25 PM
  AVS has a series of short courses that address XPS. There are also courses outside of AVS. John Grant is on this meeting, and does some direct courses.
- from Jeff Fenton to everyone: 12:25 PM
  John's course information is at: https://surfaceanalysis.org/
  from Stefan van Vliet to everyone: 12:26 PM
  Thank you very much.
- **from David Wieliczka to everyone: 12:27 PM**
  Can energy resolution be part of the curve fitting software to reduce the ability to place two peaks closer together than the resolution.
- from Vince Crist to everyone: 12:28 PM
- The XPS Library is designed to be a receptacle for all parameters for all instruments around the world. They can be shared https://xpslibrary.com/
- from Peter Cumpson to everyone: 12:29 PM
  There is some effort internationally to define "persistent instrument identifiers" so that publications can reference individual instruments like a DOI. The XPS community should integrate with that effort... [*The persistent identifiers seems to be a great place for analysts to list their instruments and reference them. Publication (by vendors?) in Surface Science Spectra might be another useful option because SSS already indicates some, but not all of the relevant parameters and instrument information could be in the same location as related data*]
- **from Robyn Goacher to everyone: 12:30 PM**
  Two ideas for reaching more analysts would be to generate short tutorial videos with good and bad examples, and to create and send out free posters listing the ASTM/ISO standards for each instrument, which could be posted at the instrument.
- from John Grant to everyone: 12:30 PM

Yes, I teach a 3-day course on XPS and Data Processing (last one was online in September 2020), 2-day CasaXPS (also was in September). I have a 2-day AES course online in March or April 2021, and I plan to repeat the XPS and CasaXPS online courses in May or June 2921. Email me if you would like to be added to my mailing list; j.grant@ieee.org

- from Uwe Scheithauer to everyone: 12:31 PM
  Drive licence system for intruments operators?
- from Peter Cumpson to everyone: 12:32 PM
  https://www.rd-alliance.org/persistent-identification-instruments
  from Alberto Herrera-Gomez to everyone: 12:33 PM
  what about a website puting together experts and users
- from william stickle to everyone: 12:34 PM
  you need to get the ediors of these (offending) journals to create an editorial about this general issue
- from Shohini Sen-Britain to everyone: 12:34 PM
  Perhaps working with instrumental vendors to prepare mandatory trainings for people purchasing new XPS instruments
- from Yung-Chen Wang to everyone: 12:34 PM
  Is ththis ere something the publishers can help with? provide information to authors?
  from Yung-Chen Wang to everyone: 12:34 PM
  is this*
- from John Grant to everyone: 12:37 PM
  Regarding instrument operation, when consulting with users in their lab operation, the best improvement in data acquisition time I was able to achieve with users in XPS was a factor of 10X! They were operating the instrument with incorrect settings for apertures and pass energies. Talk about seeing their mouths drop!
- **from David Morgan to everyone: 12:37 PM**
  re the repository, check out http://www.harwellxps.guru - this is not a "be all and end all" but put together as part of the UK National XPS facility to give ann overview of topics and link to ISO/ASTM documetns ro the relevant journal papers (jusch as these JVST A papers)
- **from william stickle to everyone: 12:37 PM**
  but you peak cannot be 0.3 ev wide like we saw in that one example
- from Jon Counsell to everyone: 12:37 PM
  There could be the option of providing the guides with all new instruments - as long as the main 3 vendors agree that would be simple to do. BUT most people who publish the data do not run the instruments..... and therefore wont see the guides.
- from Amal Cherian to everyone: 12:37 PM
  All the data originates from instrument. Developing awareness among operators/trainers to educate users (also the paper authors) about the pitfalls in analysis/processing will be the best shot
- from Vic Bermudez to everyone: 12:37 PM
  This would be a lot of work, but how about a series of You Tube videos, for example showing someone working through, step by step, the process of fitting a fairly complex XPS peak.
- from Jhonatan Rodriguez Pereira to everyone: 12:40 PM
  I think that each of the manufacturers should have at least one expert in data analysis to provide training and be consulted when the client requires it.
- from Manish Shinde to everyone: 12:41 PM
  How can I join the group for further conversation

- from Son Hoang to everyone: 12:41 PM
  Very helpful session. Thanks every one
- from Jeff Fenton to everyone: 12:43 PM
  Couple of comments on Youtube videos, but Vince Crist indicated that he has videos on Youtube. Additionally, Neil Fairley has videos on using CasaXPS.
- from Giacomo Ceccone to everyone: 12:43 PM
  Thanks for the interesting session. As Mark point out we need to invest on people. What about some common courses on XPS misuse in the Universities? This will avoid the excuse that ISO standards are expensive
- from Jeff Fenton to everyone: 12:43 PM
  Vince's Youtube: https://www.youtube.com/channel/UCHdLsIFIkJ5EaR5FC6aomkw
- from Vince Crist to everyone: 12:43 PM
  Reviewers dont care
- from Jeff Fenton to everyone: 12:44 PM
  CasaXPS: https://www.youtube.com/channel/UCHdLsIFIkJ5EaR5FC6aomkw
- from John Grant to everyone: 12:46 PM
  Some editors do not care. This is obvious when editors do not want to publish a letter on problems with XPS data published in their journals. Publishers are not involved with this and largely are mainly interested in making money.
- from Mark Engelhard to everyone: 12:46 PM
  CasaXPS and other textbooks are availbe and helpful for peak fitting
- to Manish Shinde (privately): 12:46 PM
  Greeting Manish: Do you want to get in contact with ASTM E42 in general?
- to Manish Shinde (privately): 12:47 PM
  You can find more information on ASTM E42 at https://www.astm.org/COMMITTEE/E42.htm
  from Manish Shinde (privately): 12:48 PM
  i wanted to get in touch and evolved if possible i accidentally saw this conference on linked in but could not find this info any where
- from Robyn Goacher to everyone: 12:48 PM
  Thank you for hosting this!
- to Manish Shinde (privately): 12:48 PM
  By becoming a member you can find out about upcoming meetings.
- from David Morgan to everyone: 12:49 PM
  Spectra and calibration data etc are also part of a drive by SpectrsocopyHub - https://spectroscopyhub.com/
- to Manish Shinde (privately): 12:49 PM
  Also AVS (https://avs.org/) is a good resource to get connected with.
- from Peter Cumpson to everyone: 12:50 PM
  XPS Software of 2020 is largely software of 1990 on steroids. It allows experts to process many more spectra efficiently, but if we were starting from here to provide software for new users to analyse spectra *safely*, it would look very different.

To: Participants in the November 6, 2020 ASTM E42 Surface Analysis Community Forum

Thank you for your participation in this Forum and for the many comments and suggestions! The purpose of this note is to answer some questions, summarize of the suggestions, and get your input on how we can move forward to address some of the issues raised and discussed during the Forum.

1. The **slides of the presentations** are available on the ASTM E42 Committee webpage: https://www.astm.org/COMMITTEE/E42.htm and can be found under the heading '**Surface Analysis Community Forum November 2020: Presentations and Discussion'** in the additional information section.
2. As **follow up** to this Forum we anticipate some **virtual "working group" meetings** focusing on specific topics. We will inform you of such meetings and the topic. You may participate in any topics as you wish and can. Below you will also be asked to identify any **areas or topics you specifically are interested in helping develop** and you would be added to a working group. **Other ASTM E42 Forums on surface analysis issues** are being planned.
3. You are welcome and encouraged to **join and participate in ASTM Committee E42** activities. Your membership enables you to get online or a hard copy of relevant ASTM E42 standards and you have formal participation in the ASTM E42 balloting process. Information can be found at the E42 link above. Dave Wieliczka is the chair of the electron spectroscopy subcommittee (dwieliczka@kcnsc.doe.gov) and more members would be very welcome.
4. As indicated, the **collection of XPS guides in the JVSTA Reproducibility Challenges and Solutions** collection of papers will be available for purchase as a soft bound book during a short period only. The time period is limited because this is a special onetime printing of the collection by AIPP for the AVS. Some instrument vendors have indicated that they may purchase many copies. We will send you information on when and how to order. We will send you information about obtaining a copy when it is available. A listing of the papers currently printed or online can be found at: https://avs.scitation.org/toc/jva/collection/10.1116/jva.2020.REPROD2020.issue-1.
5. Please indicate by email response to Chris Moffitt and Don Baer specific areas you would like to assist in addressing the XPS quality reproducibility issues.
    a. Development of a guide to reporting instrument and data parameters in publications and reports. Assist efforts to publish or establish DOI identifiers for specific instruments or instrument types to assist authors in reporting instrument relevant parameters. Explore ways to get attention of journal editors, reviewers and authors.
    b. Establishing a list of resources, short courses, websites and other information useful for those doing surface analysis and exploring ways to distribute or make available that information. May assist or complement current websites and other efforts.
    c. Identifying existing YouTube videos relevant to XPS/surface analysis and possibly creating more.
    d. Identifying possible software/operating system opportunities to address the issues and participating in discussions with instrument and other vendors. Exploring ways to engage vendors in developing solutions.

e. Exploring additional methods to distribute information about issues to the different types of XPS users including those who operate instruments and those who just use data.
f. Identifying important topics and participating in interlaboratory comparison studies to identify and solve specific problems or issues.

6. For your information a copy of the discussion questions from the Forum is attached.

We welcome your thoughts, ideas, and suggestions at any time! Feedback on your experience with the technical aspects of the WebEx presentation is also welcome. Please let us know if there were any specific issues, or you have any suggestions to improve this aspect of the forum. We also welcome input on any additional distribution channels for announcements regarding additional forums in the future.

Best healthy wishes for the coming holiday season and thank you again for your participation,

Chris Moffitt (cmoffitt@kratos.com)
Matt Linford (mrlinford@chem.byu.edu)
Mark Engelhard (mark.engelhard@pnnl.gov)
Don Baer (don.baer@pnnl.gov)
Jeff Fenton (jeffrey.l.fenton@medtronic.com)