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Sandia National Laboratories

Statistics and Metrology: A Collaboration

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Statistical Sciences

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Topics

- Overview of Primary Standards Lab (PSL)
- Case Study: Measurement of Neutron Yield
- Opportunities for Collaboration
- Resources

Primary Standards Lab for the Nuclear Security Enterprise (NSE)

- Located at Sandia National Labs, Albuquerque
- Maintains primary standards traceable to national standards
- Calibrates and certifies customer reference standards
- Statistical Sciences Department has collaborated with the PSL for over 15 years
  - Project Collaboration (Uncertainty Analyses)
  - Training Classes developed and taught
  - Publications, both internal and external
  - Developing a metrology handbook for the NSE

PSL Overview

Critical Measurements within NSE

- Design
  - Dimensional and weight requirements, performance in harsh environments
- Qualification
  - Demonstrate margin, lifetime requirements, reliability requirements
- Manufacturing
  - Process, product performance measurements
- Reliability Assessment
  - Assure reliability of aging stockpile in dormant storage environments

Flow, Acceleration, Shock and Humidity Laboratory

The FASH Lab capabilities include acceleration measurements for a wide variety of accelerometers and shock pulses to 100,000 g. Gas flow measurements can be performed over a wide range of flow rates from a few milliliters/minute to 3000 liters/minute for a variety of flow standards and devices. Frost/dew point can be measured to a few hundred parts per billion moisture and relative humidity from a few percent to 95 percent.

Temperature Lab

Capabilities include resistance thermometry (Standard Platinum Resistance Thermometers Platinum Resistance Thermometers, thermistors, and Resistance Thermometer Devices), liquid-in-glass thermometry, thermocouple thermometry, temperature simulator/readout calibrations, on-site calibrations of temperature chambers, and associated recorders and controllers.

Electrical Calibration Lab

The PSL's Electrical Calibration Laboratory standards include a resistance bridge system; shunts; high-accuracy multifunction calibration systems; picoamp source and measurement systems; synthesizer/function generators; counters; oscilloscope calibrators; audio analyzers; spectrum analyzers; microwave and frequency counters; capacitance bridges; and Inductance, Capacitance, Resistance meters.

Pressure Lab

Primary pressure standards consist of pneumatic and hydraulic Deadweight Piston Gauges. Secondary transfer standards consist of pressure controller/calibrators with varying ranges of Quartz Reference Pressure Transducers. Capabilities include hydrostatic pressure comparisons from 0.2 psi to 100,000 psi, and dynamic pressure comparisons from 300 psi to 80,000 psi, with rise times as low as 3 milliseconds.

Mechanical Calibration Lab

The Mechanical Calibration Laboratory performs a wide variety of specialized calibrations (e.g. production gages and fixtures) in addition to calibrations of various types of dimensional measuring and test equipment, along with torque and mass. The team provides consultation and guidance on drawing definition (i.e. GD&T) and measurement practices.

AC Lab

Metrology resources include high-accuracy AC current and voltage measurement systems to quantify component AC-DC difference, unique pulsed high-voltage generation and measurement capability to certify resistive and capacitive voltage dividers, and time and frequency measurements using a NIST-designed system. In addition, electrical impedance of inductors and capacitors over a wide frequency range can be measured and certified for the customer.

DC Lab

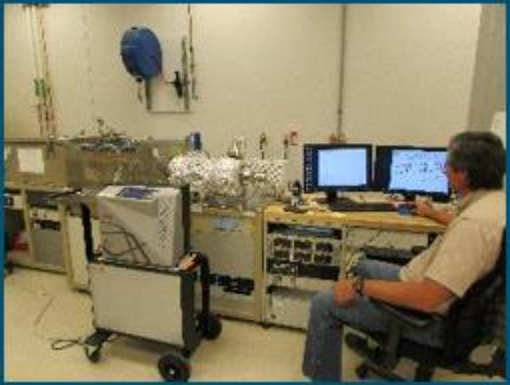
The DC Lab's capabilities include voltage, current, resistance, and ratio devices. The primary standards include laboratory and portable Josephson Array Voltage Standards, a set of Thomas 1-ohm resistors, high-voltage dividers, Hamon transfer standards, and various ratio devices (current comparators, potentiometers, ratio sets, and cryogenic current comparator).

Microwave Lab

PSL microwave standards capabilities include power (both CW and pulse), group delay, and scattering parameters (which include attenuation and reflection coefficients or Voltage Standing-Wave Ratio). The primary standards include 50 ohm terminations, various coaxial air lines, and coaxial and waveguide thermistor mounts. These standards support a variety of measurement systems, including four Vector Network Analyzers, a power standard calibration system, and a power meter calibration system.

Leaks Lab

The Leaks Lab capabilities include both fundamental and direct comparison leak measurements for any nonreactive, nontoxic, nonradioactive gas from 2 to 128 atomic mass units (range of mass spectrometers). The range covered is 1×10-7 mol/s to 1×10-14 mol/s. In addition, the Leaks Laboratory measures temperature coefficients from 5°C to 50°C for permeation leaks and measures either open or closed reservoir leaks.



Length, Mass, Force (LMF) Lab

LMF Submicrometer capabilities include gage blocks, roundness, thread wires, gaging balls, surface roughness, step gages, line standards, and three-dimensional measurements. Mass certifications are available from 1 milligram to 64 kilograms.

Vacuum Lab

Transfer vacuum standards consist of Capacitance Diaphragm Gages (CDGs), Spinning Rotor Gages, and Ionization Gages with direct traceability to the Systeme International through NIST. The transfer standards cover a vacuum range from 1×10^{-7} Pa to 133 kPa. Primary calibrations are performed using an automated Forced-Balance Piston Gauge to calibrate transducers such as CDGs in differential or absolute mode from -15 to 15 kPa.

Radiation/Optics Lab

The PSL Radiation/Optics Lab has the responsibility of calibrating lead probe neutron detectors for the NSE. The lead probe is a unique detector vital for neutron generator and neutron tube characterization and testing throughout the NSE. Primary optical power and energy standards are based on heat-flow calorimetry, while spectral responsivity is based both on NIST-certified and "absolute" Si detectors.

NVLAP Accreditation

United States Department of Commerce
National Institute of Standards and Technology

NVLAP®

Certificate of Accreditation to ISO/IEC 17025:2005

NVLAP LAB CODE: 105002-0

Sandia National Laboratories
Albuquerque, NM

is accredited by the National Voluntary Laboratory Accreditation Program for specific services, listed on the Scope of Accreditation, for:

Calibration Laboratories

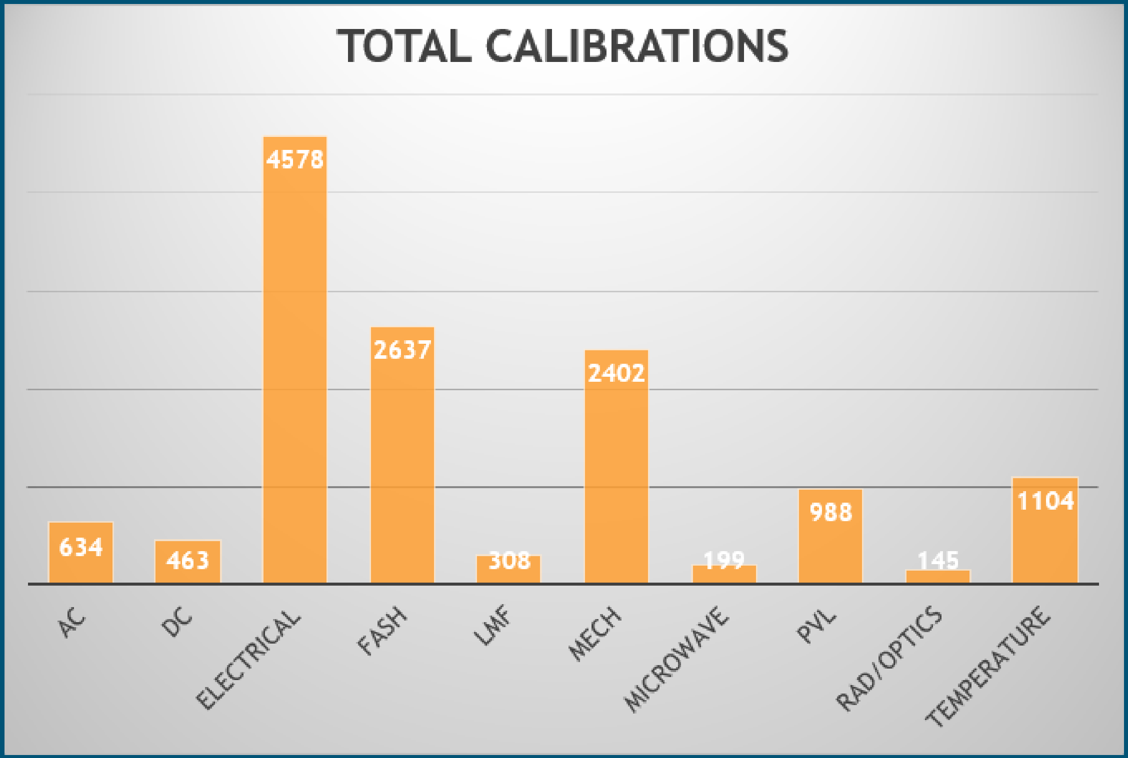
This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communique dated January 2009).

DEPARTMENT OF COMMERCE • UNITED STATES OF AMERICA

2018-03-23 through 2019-03-31
Effective Dates

For the National Voluntary Laboratory Accreditation Program

PSL Metrics: Number of Calibrations



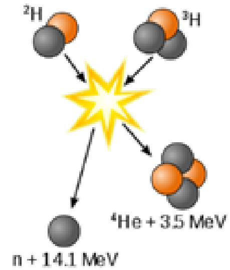
Includes all calibrations: Primary and Secondary;
In-House and Outsourced

Number of Calibrations Data for 2/1/2017 through 1/31/2018: **13458** calibrations

Case Study: Measurement of Neutron Yield

- Calibration Chain for measuring neutron yield of a neutron generator (critical component of a nuclear weapon)
- Uncertainty analysis of secondary lead probe standard
- Results and implications for improving the measurement

Calibration Chain – Neutron Yield Measurements



Associated Particle Technique
Ion Beam Laboratory

Primary Lead Probe Standard
IBL/PSL

Secondary Lead Probe Standard
Primary Standards Laboratory

Lead Probe Neutron Detectors

Neutron Tube/Generator Product Testers

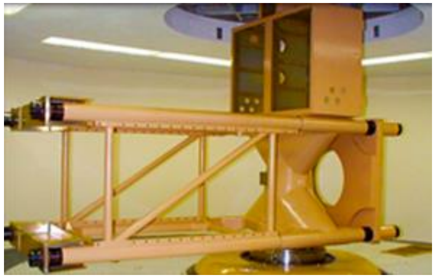
NGM Calibration Tester

Static WETL Testers

NGMs

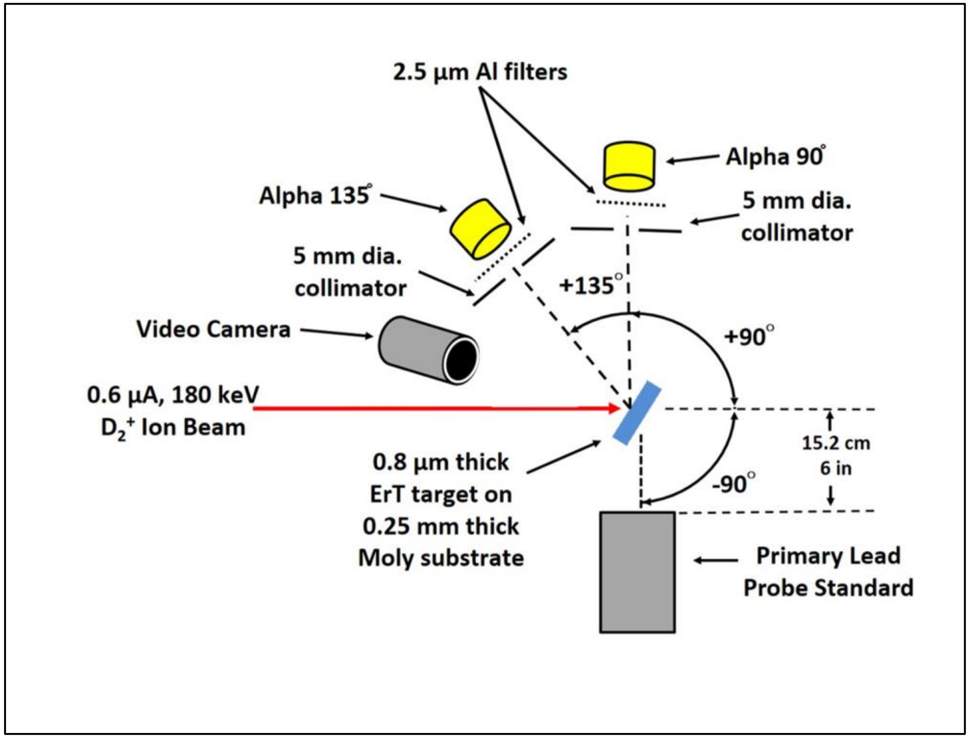
JTAs

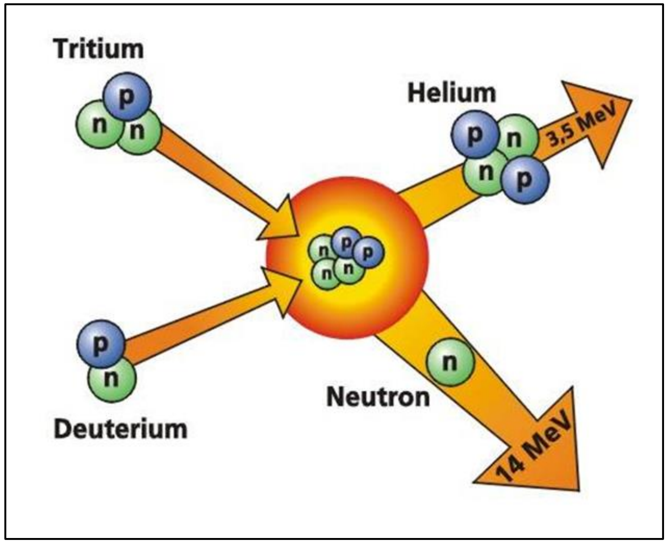
Dynamic WETL Testers



Calibration of Primary Lead Probe

Ion Beam Accelerator



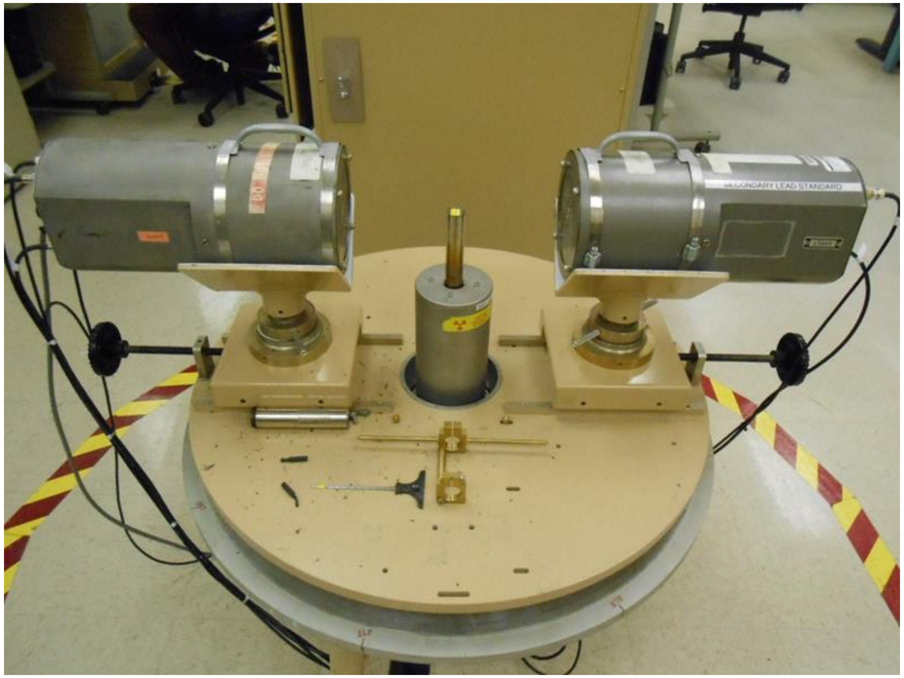


Observed neutron count (alpha particle count) from ion beam accelerator used to calibrate primary lead probe standard

Calibration of Secondary Lead Probe

1. Laboratory neutron source is measured simultaneously by both primary and secondary lead probes at the PSL.
2. Calibration factor (F) results from this comparison.
3. All subsequent lead probe detectors are calibrated to this secondary lead probe at the PSL.

Calibration of Lead Probe Detectors



1. Laboratory neutron source is measured simultaneously by both secondary lead probe standard and lead probe detector at the PSL.
2. Calibration factor for lead probe detector results from this comparison.
3. All subsequent lead probe detectors are calibrated on the same secondary lead probe standard at the PSL.

Calibration of Neutron Generator Monitor (NGM)

1. NGMs are calibrated at the manufacturing facility against a traceable lead probe detector.
2. These NGMs are used for dynamic surveillance measurements (flight tests and centrifuge tests).

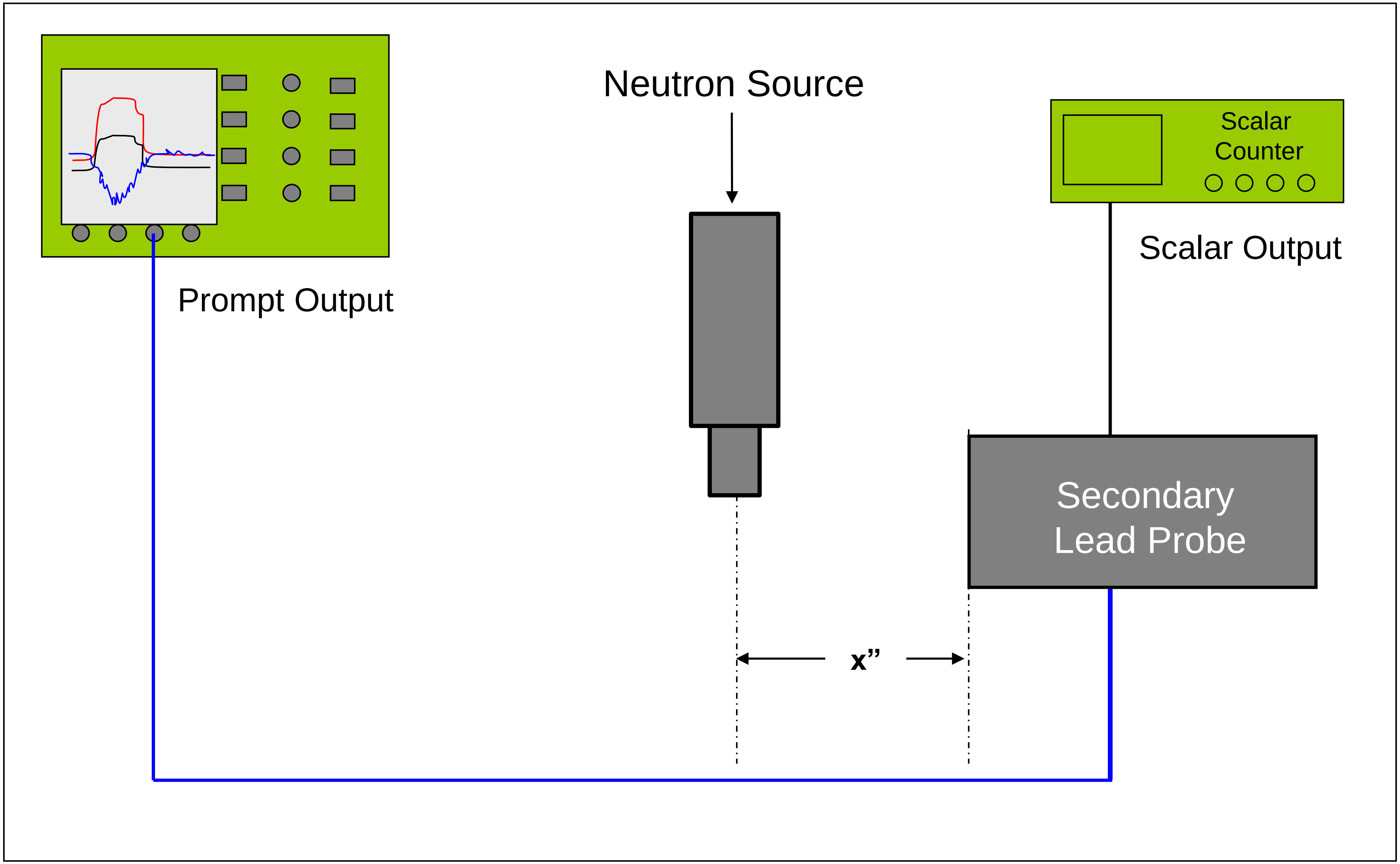
Uncertainty Analysis of Secondary Lead Probe Standard

- All neutron measurements made by SNL are referenced to a secondary lead probe standard via calibration

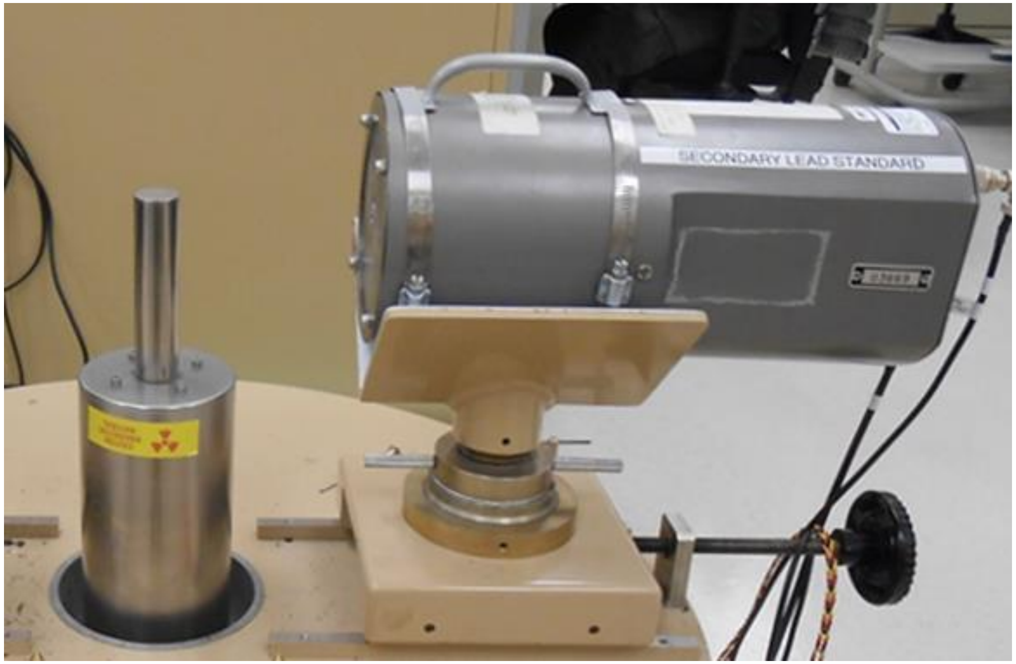
- Therefore, the uncertainty of any neutron measurement is linked through the calibration chain to the uncertainty of the secondary lead probe standard

- Estimating the uncertainty of the secondary lead probe is thus vital to making good estimates of the uncertainty of any neutron measurement made in the NSE
  - Product acceptance tests (lead probe detector)
  - Surveillance tests (lead probe detector)
  - Flight tests (NGM)
  - Dynamic Lab tests (NGM)

Neutron Yield Laboratory Measurement Setup



Neutron Yield Measurement Setup



1. The Type A uncertainty of the secondary lead probe is determined by repeated measurements of the laboratory neutron source.
2. All subsequent lead probe detectors are calibrated with the same secondary lead probe.
3. These lead probe detectors are used in product testers and static surveillance testers.

Experimental Test Plan

- Forty scalar measurements (40 neutron source runs) at multiple levels
- Ten background measurements taken immediately before the forty scalar shots

Steps to Determine Uncertainty (GUM Reference)

1. List the sources of uncertainty
2. Formulate the measurement equation, $\eta = AfF\,(S\text{-}B)$
3. Determine the distributions for the input quantities, $A, f, F, S, B,$ and evaluate the standard uncertainties for each input quantity
   - Type A evaluations are used for S and B
   - Type B evaluations are used for A and F
   - f is treated as a conversion factor with no uncertainty
4. Determine the combined standard uncertainty (Taylor Series)
5. Estimate the measurand, η, from the measurement equation
6. Compute the expanded uncertainty using a coverage factor k based on the degrees of freedom and the level of confidence required
7. Report the uncertainty interval

Measurement Equation

$\eta = AfF\,(S\text{-}B)$

η = Total number of neutrons produced

A = Attenuation factor, determined by geometry of test setup
f = Nominal conversion factor, treated as known
F = Calibration factor, determined by the PSL
S = Total scalar counts from the lead probe
B = Background counts measured by Lead Probe

A, F, S, and B are the sources of uncertainty

Type A and Type B Uncertainties

- Type A evaluation of uncertainty
  - "Method of evaluation of uncertainty by the statistical analysis of series of observations." (Captures the variation in the current test data.)

- Type B evaluation of uncertainty
  - "Method of evaluation of uncertainty by means other than the statistical analysis of series of observations." (Captures the variation that would NOT be seen in the current test data)
    - Calibration certificates
    - Manufacturer's specifications
    - Experience (expert knowledge of instrument)
    - Previous measurement data

Reference is: JCGM 100:2008. "Guide to the Expression of Uncertainty in Measurement." (GUM)

Distributions for Input Quantities and Standard Uncertainties

Input Quantity	Mean (μ)	Standard Uncertainty Type A	PDF Type A	Standard Uncertainty Type B	PDF Type B
f	3333				
A	1.00			0.020	Rectangular
F	1.15			0.032	Rectangular
S	4800	69.3	Poisson		
B	200	4.47	Normal		

$$\eta = AfF\,(S\text{-}B)$$

Combined Standard Uncertainty via Taylor Series Approximation

With the input quantities ($x_i's$), and measurement equation

$$y = g(x_1, x_2, \dots, x_N)$$

the combined standard uncertainty, $u_c(y)$, is the positive square root of the combined variance, given by

$$u_c^2(y) = \sum_{i=1}^{N} \left(\frac{\partial g}{\partial x_i}\right)^2 u^2(x_i)\,.$$

Each $u(xi)$ is a standard uncertainty evaluated using either Type A or Type B evaluation.

Calculation of Type A Combined Standard Uncertainty

- Compute the sensitivity coefficients

$$\frac{\partial \eta}{\partial S} = AfF \qquad \frac{\partial \eta}{\partial B} = -AfF$$

- Compute the combined standard uncertainty from the standard uncertainties and sensitivity coefficients

$$u_A(\eta) = \sqrt{\left(AfF\right)^2 u^2(S) + \left(-AfF\right)^2 u^2(B)}$$

$$= \sqrt{(265{,}555)^2 + (17{,}141)^2} = 266{,}100$$

Calculation of Type B Combined Standard Uncertainty

- Compute the sensitivity coefficients

$$\frac{\partial \eta}{\partial A} = fF(S-B) \qquad \frac{\partial \eta}{\partial F} = Af(S-B)$$

- Compute the combined standard uncertainty from the standard uncertainties and sensitivity coefficients

$$u_B(\eta) = \sqrt{\left(fF\left(S-B\right)\right)^2 u^2(A) + \left(Af\left(S-B\right)\right)^2 u^2(F)}$$

$$= \sqrt{\left(367{,}963\right)^2 + \left(490{,}618\right)^2} \quad = 613{,}272$$

Combined Standard Uncertainty

- Root-Sum-Square the Type A and Type B Combined Standard Uncertainties

$$u_c(\eta) = \sqrt{u_A^2(\eta) + u_B^2(\eta)}$$

$$= \sqrt{(266{,}100)^2 + (613{,}272)^2}$$

$$= 6.685 \times 10^5$$

Estimate the Total Neutron Yield

$$\eta = AfF\,(S\text{-}B)$$

$$= (1.00)(3333)(1.15)(4800\text{-}200)$$

$$= 1.76 \text{ x } 10^7$$

Determine the Expanded Uncertainty

- Calculate the effective degrees of freedom using the Welch-Satterthwaite formula

- The $t_{95}(v_{eff})$ values obtained from this calculation ranged from 1.98 to 2.02 for the application discussed here
  - 40 scalar calibration points
  - 10 background points
  - This is the coverage factor k that gives a 95% CI

Determine the Expanded Uncertainty

- Calculate the expanded uncertainty using the coverage factor determined from the t-distribution for a 95% level of confidence

$$U_{\eta}^{95} = t_{95}(\nu_{eff})\, u_C(\eta)$$

$$= (1.98) \times (6.685 \times 10^{5})$$

$$= 1.32 \times 10^{6}$$

Report the Uncertainty Interval

- The uncertainty interval at a 95% level of confidence (k = 1.98) for total neutron count is:

$$\eta \pm U_{\eta}^{95} = 1.76 \times 10^{7} \pm 1.32 \times 10^{6}$$

$$= (1.63 \times 10^{7}, 1.89 \times 10^{7})$$

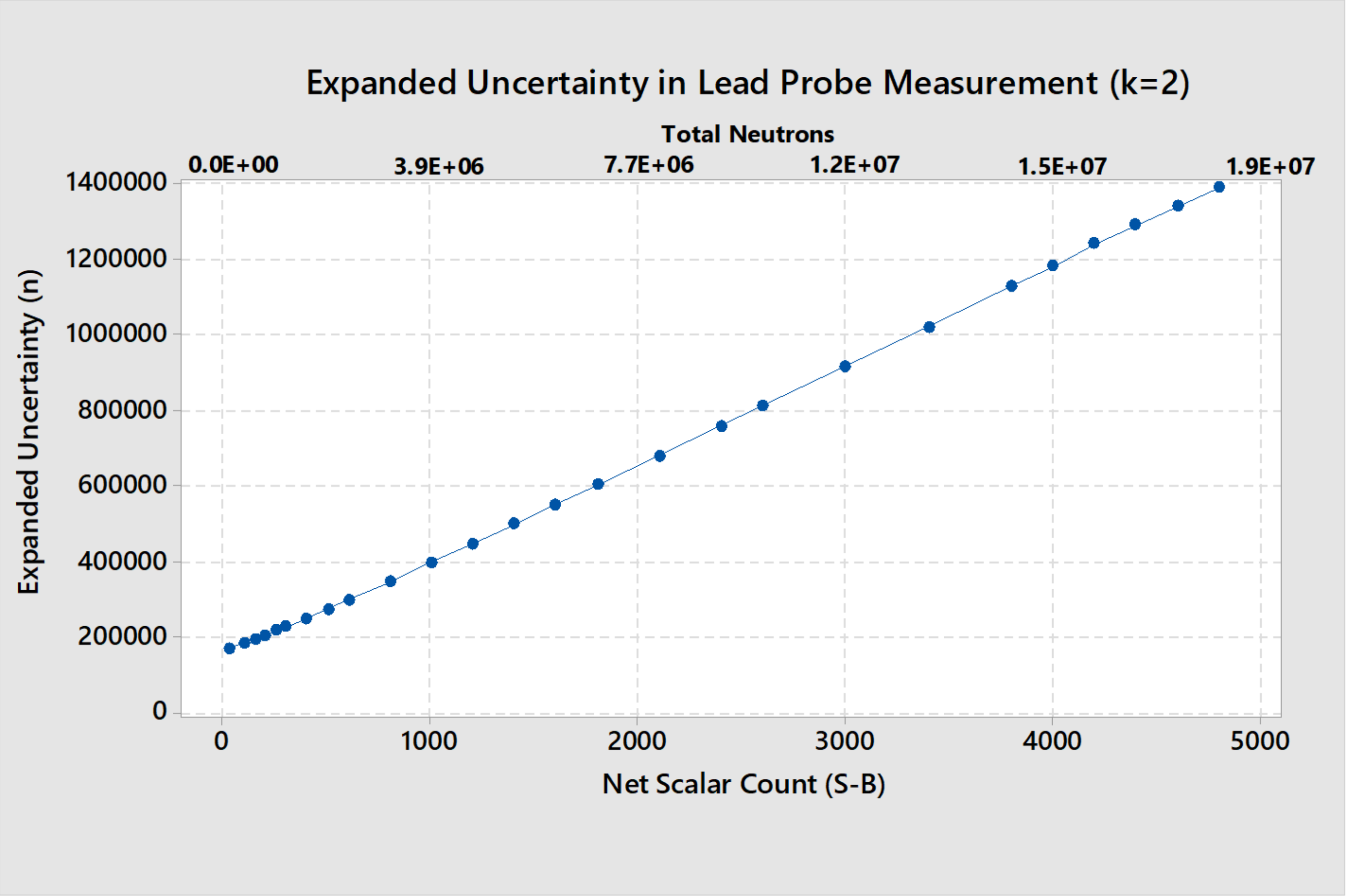
$$= 1.76 \times 10^{7} \pm 7.5\%$$

Expanded and Relative Expanded Uncertainties in Lead Probe

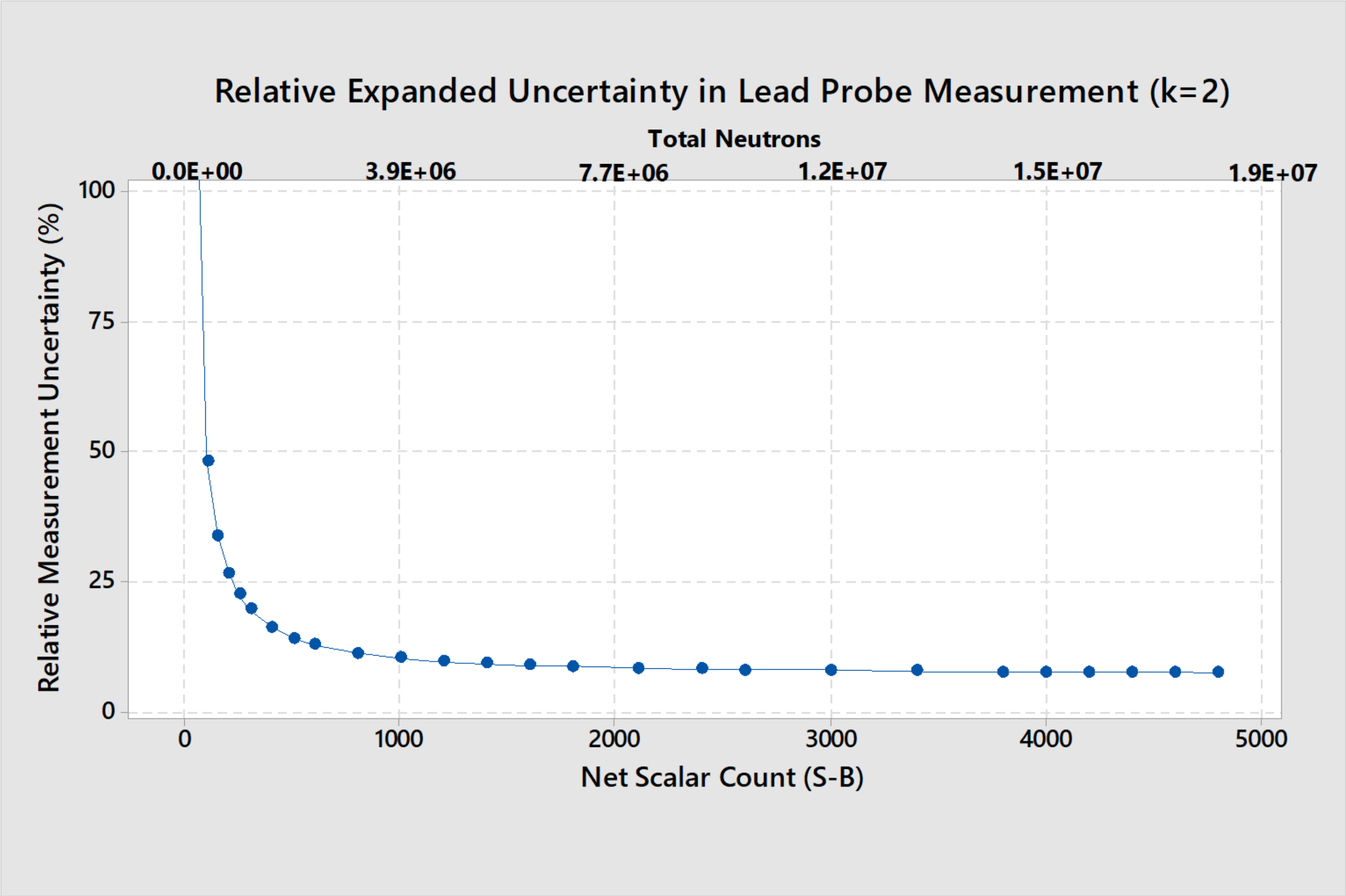
(*S*-*B*)	n_{Pb}	***U***	U / n_{Pb}
4800	1.84 E+07	1.39 E+06	7.5%
4600	1.76 E+07	1.34 E+06	7.6%
4400	1.69 E+07	1.29 E+06	7.6%
4200	1.61 E+07	1.24 E+06	7.7%
4000	1.53 E+07	1.18 E+06	7.7%
3800	1.46 E+07	1.13 E+06	7.8%
3400	1.30 E+07	1.02 E+06	7.9%
3000	1.15 E+07	9.17 E+05	8.0%
2600	9.97 E+06	8.12 E+05	8.1%
2400	9.20 E+06	7.59 E+05	8.3%
2100	8.05 E+06	6.80 E+05	8.5%
1800	6.90 E+06	6.02 E+05	8.7%
1600	6.13 E+06	5.50 E+05	9.0%
1400	5.37 E+06	4.98 E+05	9.3%
1200	4.60 E+06	4.47 E+05	9.7%
1000	3.83 E+06	3.96 E+05	10.3%
800	3.07 E+06	3.46 E+05	11.3%
600	2.30 E+06	2.97 E+05	12.9%
500	1.92 E+06	2.73 E+05	14.2%
400	1.53 E+06	2.49 E+05	16.3%
300	1.15 E+06	2.27 E+05	19.7%
350	9.58 E+05	2.16 E+05	22.5%
200	7.67 E+05	2.05 E+05	26.8%
150	5.75 E+05	1.95 E+05	33.9%
100	3.83 E+05	1.85 E+05	48.2%
25	9.58 E+04	1.70 E+05	177.7%

Results were confirmed with Monte Carlo analysis.

Expanded Uncertainties in Secondary Lead Probe



Relative Expanded Uncertainties in Secondary Lead Probe



Sensitivity Analysis

$$u_c = \sqrt{(AfF)^2 u^2(S) + (-AfF)^2 u^2(B) + \big(fF(S-B)\big)^2 u^2(A) + \big(Af(S-B)\big)^2 u^2(F)}$$

$$= (AfF)\sqrt{\left(S + \frac{B}{N}\right) + (S-B)^2\left(\frac{u^2(A)}{A} + \frac{u^2(F)}{F}\right)}$$

$$\cong f\sqrt{\left(S + \frac{B}{N}\right) + (S-B)^2\big(u^2(A) + u^2(F)\big)}$$

f = Nominal conversion value (neutrons per output count) = 3333
A= Adjustment factor associated with measurement geometries ≈ 1.0
F= Calibration lead-probe conversion factor ≈ 1.15
S= Total scalar counts ≈ 4800
B= Background counts ≈ 200

Steps to Reduce Uncertainty

- Reduce $u(A), u(F)$
  - Calibration factors based on measurement geometry

- Reduce background counts (B)

- Decrease f
  - Increase the net scalar count per neutron

Opportunities for Collaboration

- Design of Experiments
  - Factorial designs, mixed-models, variance component estimation

- Failures in the Assumptions
  - GUM-Propagation of Error Model limitations
  - Non-linearity, Non-independence of input quantities

- Degrees of Freedom
  - Proper use of Welch-Satterthwaite Approximation

- Type A and Type B Uncertainties
  - Type A: Variation in the data at time of experimentation
  - Type B: Variation not seen in the data at time of experimentation
  - Avoid double counting

- Interpretation of Gage R&R results

Resources

- GUM, NIST Guide (Technical Note 1297), AIAG Guide
  - GUM Supplement 1 for Monte Carlo approach
- NIST On-line handbook (www.itl.nist.gov/div898/handbook/)
- *Metrologia*, NCSL *Measure,* NIST Publications
- Metrology conferences (NCSLI), Short courses

Backup Slides

Case Study: Gage R&R of a Coordinate Measuring Machine (CMM)

- 4 parts
- 3 operators
- 3 repeats

Typical recommended design (AIAG Guide) has 10 parts, 3 operators, 3 repeats

Gage R&R Results

Source	DF	SS	MS	F-Value	P-Value
Part	3	70256	23419	171	0.00
Operator	2	1910	955	7.0	0.03
Part*Op	6	821	137	1.3	0.31
Error	24	2604	109		
Total	35	75591			

Using the ANOVA approach, equating Mean Squares to their expectations,

$$\hat{\sigma}^2_{Part} = 2586.9$$

$$\hat{\sigma}^2_{Oper} = 68.2$$

$$\hat{\sigma}^2_{Part\ x\ Oper} = 9.4$$

$$\hat{\sigma}^2_{Error} = 108.5$$

Gage R&R – Reproducibility Term

$$\hat{\sigma}^2_{Repr} = \hat{\sigma}^2_{Oper} + \hat{\sigma}^2_{Part\ x\ Oper}$$

$$= \frac{1}{12}\left(MS_{Oper}\ -\ MS_{Part\ x\ Oper}\right)\ + \frac{1}{3}\left(MS_{Part\ x\ Oper} - MS_{Error}\right)$$

$$= \frac{1}{12}\left(MS_{Oper}\right) + \frac{1}{4}\left(MS_{Part\ x\ Oper}\right) - \frac{1}{3}\left(MS_{Error}\right)$$

$$= 79.6 + 34.2 - 36.2$$

$= 77.6$ with $\upsilon_{Repr} \cong 2$ (via the Welch-Satterthwaite approximation)

Gage R&R

Reproducibility and R&R variance terms:

$$\hat{\sigma}^2_{Repr} = \hat{\sigma}^2_{Oper} + \hat{\sigma}^2_{Part\ x\ Oper} = 77.6 \qquad \upsilon_{Repr} \cong 2$$

$$\hat{\sigma}^2_{R\&R} = \hat{\sigma}^2_{Oper} + \hat{\sigma}^2_{Part\ x\ Oper} + \hat{\sigma}^2_{Error} = 186.1 \qquad \upsilon_{R\&R} \cong 9.7$$

(Effective degrees of freedom via W-S approximation)

Observations

- Reproducibility term σ^2_{Repr} poorly estimated
- Many more operators would be needed to obtain a useful estimate of σ^2_{Repr}
- Recommend not using σ^2_{Repr} from Gage R&R study alone
- Recommend collecting data over time (control chart methodology) to estimate reproducibility term
  - Repeated measurements of a check standard over time
  - Repeated measurements of manufactured units (4-5) set aside

Metrology Handbook

- Developed for practitioners of metrology within the NSE
- “How to” guide of metrology best practices
- Ideas illustrated with numerous case studies from the NSE
- Includes basics, some advanced concepts, and special topics

Topics

- Basic Measurement Terminology

- International System of Units (SI), Traceability and Calibration
  - Base Units and Derived Units of Measure

- Introduction to Probability and Statistics

- Measurement Uncertainty in Decision Making

Topics (Cont'd)

- The Measurement Model and Uncertainty
- Analytical Methods for the Propagation of Uncertainties
- Monte Carlo Methods for the Propagation of Uncertainties
- Determining Uncertainties in Fitted Curves

Topics (Cont'd)

- Design of Experiments in Metrology
  - Factorial Designs
  - ANOVA with mixed models
  - Gage R&R Studies
  - Tester Qualification

- Special Topics in Metrology
  - SPC for a Measurement Process
  - Binary Measurement Systems
    - Measure of Correctness
    - Measure of Agreement
  - One-Shot Devices
  - Sample Size Determination