Trace Evidence
Measurement Science & Standards
Research at NIST

Eric Steel (eric.steel@nist.gov)
Greg Gillen (j.gillen@nist.gov)

Material Measurement Laboratory
What is this talk about?

• Why are we here?
• Why NIST is working in this field?
  – What NIST can do for the forensic science community (and does not do)
• The Standards process and NIST
• Analytical uncertainty
  – One of NIST’s strengths
• NIST and trace evidence
Why we are here? NRC Study

Strengthening Forensic Science in the United States: A Path Forward (2009)

• Long Term, Broad Issues Highlighted by NAS
  – Trust
  – Validity
  – Reliability

• Commented on
  – Need for Standardization, Certification, and Accreditation
    • NIST: Conformity Assessment
  – Problems Relating to the Interpretation of Forensic Evidence
    • NIST: Measurement services – reference materials & data
  – Need for Research to Establish Limits and Measures of Performance
    • NIST: Measurement Science and Uncertainty Analysis
  – Problems Relating to the Broad Range of Forensic Science Disciplines
    • Breadth of NIST measurement expertise
Why NIST?

- to develop a fundamental basis and methods for testing materials, ...
- to assure the compatibility of United States national measurement standards with those of other nations
- to advise government and industry on scientific and technical problems
- to compare standards used in scientific investigations, ... and to coordinate the use by Federal agencies of private sector standards
- to coordinate Federal, State, and local technical standards activities and conformity assessment activities ...

Article I, Section 8: “The Congress shall have the power to... *fix the standard of weights and measures*”

For Commerce, Regulations, and Forensics... places where quantity or quality of a technology is critical to the nation
What does NIST offer the Forensic Community?

✓ Independence
  ✓ Outside of the U.S. adversarial legal system
  ◯ case work
  ◯ court testimony
  ✓ Nonregulatory
✓ Interagency coordination of standards
✓ Broad and Deep Expertise
  ✓ Measurement Science
    ✓ Validity testing, Traceability, Uncertainty Characterization, Measurement Innovation
  ✓ Quality systems
  ✓ Standards
✓ Facilities
  ✓ Chemical, Materials, and Physical Properties Measurement
✓ Services
  ✓ Standards
    ✓ Key Reference Materials
    ✓ Key Calibrations
    ✓ Key Reference Data
    ◯ ALL reference materials/data/calibrations
  ✓ Measurement & Standards Research
  ✓ Documentary Standards, Research Publications, Guides to Practice, ...
NIST: Basic Stats and Facts

- **Major assets**
  - ~3,000 employees
  - ~2,800 associates and facilities users
  - ~1,600 field staff in partner organizations (Manufacturing Extension Partnership)
  - Locations: Gaithersburg, Md. and Boulder, Co.
  - Four external collaborative institutes: basic physics, biotech, quantum, and marine science

FY 2012 Appropriations $750.8 M
Forensic Science Agencies and NIST have a symbol in common
Standards

- Flag
- Ethical/Moral
- Level of performance
- Regulatory
- Written rules of practice
  - Documentary
- Authoritative basis for comparison or use
  - Physical reference
  - Known information

- NIST:
  - Documentary Standards
    - Via Standards Developing Organizations (ASTM Int., ISO, IEC, IEEE, ...)
  - Physical Standards
    - Reference Materials
    - Reference Data
    - Calibration Services
Examples of NIST Knowledge Transfer & Traceability

- Collaborations
  - ~2800 Associates and Facility Users
  - ~100 CRADAs/year
- Measurement Research
  - ~2,200 publications per year
  - ~8,000 attendees at >60 technical workshops/conferences
- Training symposia
- Standard Reference Data
  - ~100 different types
  - ~6,000 units sold per year
  - ~25 million data downloads per year
- Standard Reference Materials
  - ~1,300 products available
  - ~30,000 units sold per year
- Patents and Inventions
  - ~10-50/year
- Baldrige National Quality Program
  - 99 Award recipients
- Manufacturing Extension Partnership
  - ~28,000 Clients
- Calibration Tests
  - ~18,000 tests per year
- Laboratory Accreditation
  - ~800 accreditations of testing and calibrations laboratories per year
- Standards Committees
  - ~400 NIST staff serving on 1,000 national and international standards committees
- Other Agency R&D
  - Agreements with ~80 Fed. Agencies
  - ~$100M/year
NIST Forensic Activities

• DNA Profiling
  – NIST Human Identity Project Team
  – Standard Materials for DNA profiling
  – Short Tandem Repeat Database and Research

• Fingerprint Analysis
  – Fingerprint Research and Standards

• Firearms, Toolmarks, and Impressions
  – Standard Bullets and Casings

• Digital and multimedia analysis
  – Computer Forensics Tools Testing (CFTT)
  – National Software Reference Library (NSRL)
  – Real Time Forensics Analysis for Analog and Digital Video Tapes
  – Mugshots and Facial Recognition
  – Forensics for Mobile Phones and Devices

• Forensic Toxicology
  – Standard material for alcohol for blood testing
  – Standard material for alcohol for breath testing
  – Toxic metals in blood
  – Drugs of Abuse

• Homeland Security Applications of Forensic Science
  – Biodefense
  – Detecting trace explosives
  – Finding dirty bombs and other radiation threats

• Questioned Documents
  – Northern Softwood Pulp Paper
  – Eucalyptus Hardwood Pulp Paper

• Trace Evidence
  – Glass, Paint, and Coatings
    • Commercial glass elemental composition standards
    • Trace elements in glass
    • Refractive index of glass
    • Lead in paint
  – Explosives/Fire Debris Evidence
    • Standard Material for Additives in Smokeless Powder
    • Standard Material for Arson Investigation
    • Standard Material for Trace Particulate Explosives
  – Trace narcotics detection
  – Arson Investigation Research
  – Chemical Characterization Powders and Particulate Matter

• Forensic Engineering
  – Building and Fire Safety Investigation

• Chemical Analysis
  – Review of Advances in Analytical Chemical Methods for Forensics 2009
## Our Measurement System: Timelines for Standards

### Measurement Methods
- Research
- Draft protocol
- Interlaboratory Comparisons
- Guidelines
- Draft method
- Standard method
- Applied method
- Rewrite
- Accreditation, Certification, Conformity Assessment
- International Traceability, Interoperability, Recognition

### Materials/Data/Calib
- Whatever you can get
- In-house stds
- Consensus stds
- Reference Materials/Data
- Certified materials/data
  - Calibration (pure/simple)
- Certified materials/data/calib
  - Quality Assurance (complex/matrix)
- Proficiency testing materials, data, algorithms, artifacts
- NMI comparisons

---

*Increasing difficulty, decreasing uncertainty*

*New or Low Use Technology*

*Mature or High Use Technology*

*Time*
Measurement Science

- How sure are you of your measurement result?
- Is it biased? ... how close to “true” is it?
- How do you figure this out?

- Uncertainty Determination
  - NIST is the most uncertain place in the US!
The Uncertainty Triangle

Reference Materials & Data

Uncertainty

Empirical Measurement

Modeling & Simulation
The Uncertainty Triangle

• SEM imaging and elemental analysis
  – Ref. Mat.: mineral-glass spheres of known composition
  – Ref. Data: x-ray generation physics, characteristic x-ray line energies
  – Simulation: Monte Carlo of electron, x-ray interactions in solids
  – SEM x-ray spectrum image measurement

• Understand and quantify estimate of uncertainty for elemental analysis in inorganic particle, thin film, and bulk specimens

Nicholas W.M. Ritchie, Microsc Microanal, 16, pp 248-258, 2010
The Uncertainty Triangle

Collected and calculated spectrum from sphere

Nicholas W.M. Ritchie, Microsc Microanal, 16, pp 248-258, 2010
The Uncertainty Triangle

Reference Materials & Data

Uncertainty

Empirical Measurement

Modeling & Simulation
Trace Evidence

• Broadest area in Forensic Science
  – Encyclopedic
• Often do not know what type of clue to look for or what you will find
• Makes sampling, analysis, and interpretation very difficult
• But, sometimes it is the only physical evidence
Roles of NIST in Trace Evidence

- Measurement Science
- Standards
- Technology

**BUT** NIST is a small agency with a large and broad measurement and standards mandate

- What does the forensic community need?
  - How important? How useful? How common a need?

- Trace Sampling
- Standard Test Materials
  - Reference Materials
  - Reference Data
  - Calibration

- Operational Protocols
  - Documentary Standards

- Technique Development for Trace Evidence
  - Advanced technologies

- Data analysis
  - Statistical analysis
  - Uncertainty determination
Trace Analysis: Three Distinct Functions

• Crime Scene Investigation
  – Sampling, evidence collection & tracking, documentation

• Materials Analysis:
  – Uncertainty, accuracy, precision, sensitivity, selectivity, ... of technique or analytical approach
  – Reliability, trust, quality control, chain of custody, ... of an individual result

• Interpretation:
  – Significance of results
    • How well does the analytical result identify a perpetrator or explain a crime
    • Uncertainty of this interpretation
Sampling

• Crime Scene/Suspect/Reference
  – Searching for/collecting one analyte
    • Is there Anthrax? Drugs? Blood?...
  – Sampling for unknown component(s)/analyte(s)
    • Is there something added to the scene by the criminal, the victim, or the criminal activity?
  – Sampling for normal (background/reference)
    • Becomes more important as techniques become more sensitive
  – Sampling the sampler/process (blanks)
Materials Analysis

• Measurement Expertise:
  – Morphologic, Molecular, Elemental, Isotopic, Crystallographic, Physical properties, ...
  – Comparison techniques
    • One or more of above
  – Sample state:
    • Liquid, Vapor, Solid
    • Particles: single to populations
  – Imaging by composition or physical property
    • Spatial resolution from cm to nm
    • 1D, 2D, 3D
  – Most chromatographies, spectroscopies and microscopies:
    • GC, LC, UV-Vis, Mass, Raman, IR, X-ray, XPS, Electron, Auger, Ion Mobility, Gamma, Alpha, Beta, Neutron...

• Characterization of Accuracy, Sensitivity, Specificity, Precision, Repeatability, Reproducibility, Validation

• Statistical Analysis and Uncertainty Determination
Validating Analytical Approaches

• Accuracy & Precision
  – Qualitative:
    • Sensitivity
      – Sufficient analyte sample and concentration
    • Selectivity
      – Matrix and interference issues
  – Quantitative
    • All Qualitative items
    • Calibration
    • Matrix Effects
  – Repeatability & Reproducibility

• Quantifying comparison between crime scene and suspect evidence

• Quality Control
  – Blind repeats, inter-operator, inter-lab, proficiency test
Interpretation

The hardest part for trace evidence?

• Significance of results
  – How well does the analytical result identify a perpetrator(s) or explain a crime
  – Uncertainty of this interpretation

• Unique/Ubiquitous nature of single or combinations of components and characteristics
  – Bullet lead, hair, ...
  – Change over time
    • Glass: refractive index, elemental composition, other

• Existence of data on frequency and locations of occurrence
Roles of NIST in Trace Evidence

- Measurement Science
- Standards
- Technology

- Trace Sampling
- Standard Test Materials
  - Reference Materials
  - Reference Data
- Operational Protocols
  - Documentary Standards & Guides
- Technique Development for Trace Evidence
  - Advanced technologies
- Data analysis
  - Statistical analysis
  - Uncertainty determination
Trace Evidence

• Very Broad Application Space
  – Can use hundreds of our products

• NIST is always prioritizing, replacing and creating new products
  – What products do you need for trace applications?

• Will ask later ... keep question in mind all day.
Forensic Science: A NIST Priority

- Develop, critically evaluate, and publish **new reference methods and technologies** for understanding crime scenes and identifying criminals.
- **Test and measure the uncertainty**, including such factors as bias, precision, and human errors, in existing forensic methods.
- **Improve the accuracy, reliability, and interoperability of forensic methods and data** through research in underlying science, rigorous testing, and methods for assessing conformance to standards.
- Develop calibration systems, reference materials and databases, and **technology test beds** for reliable and accurate forensic practice.
- Work with national and international standards developing organizations, academia, instrument manufacturers, database creators and disseminators, and the forensic science user communities to **encourage adoption of scientifically rigorous and well-characterized methods and practices**.
- **Create rigorous training programs** to facilitate basic understanding of underlying metrology in **applied forensic procedures and methods**
Trace evidence talks

Trace Sampling
9:30-9:50 Enabling Forensics Investigations of Biothreat Incidents through Sampling Standards – Jayne Morrow, Biochemical Science Division, MML
9:50-10:10 Surface Wipe Sampling for Trace Narcotics and Explosives Collection – Jennifer R. Verkouteren, Surface and Microanalysis Science Division, MML
10:10-10:30 Aerodynamic Sampling – Matthew E. Staymates, Surface & Microanalysis Science Division, MML
10:30-10:50 Break and Poster Viewing/Exhibitor Displays

Standard Test Materials/Operational Protocols
10:50-11:10 Production of Seized Drug Analysis Standards by Inkjet Printing– Jeanita S. Pritchett, Analytical Chemistry Division, MML
11:10-11:30 Nuclear Forensics Reference Materials – Kenneth G. W. Inn, Radiation & Biomolecular Physics Division, PML
11:30-11:50 Following the Scent – Development of Canine Training Aids – William A. MacCrehan, Analytical Chemistry Division, MML
11:50-12:10 Performance Validation for Trace – R. Mike Verkouteren, PhD, Microanalysis Science Division, MML
12:10-12:30 NIST Trace Explosives Test Bed – Marcela Najarro, Materials Measurement Science Division, MML
12:50-2:20 Lunch and Poster Viewing/Exhibitor Displays/Deployable Laboratory/Mobile Decontamination Vehicle (Hands-on Biothreat Response and Sample Collection Demo)

Technique Development for Trace Evidence
2:20-2:40 Automated Particle Analysis – Nicholas W. M. Ritchie, Materials Measurement Science Division, MML
2:40-3:00 Combined IMS and Biometrics – Jessica L. Staymates, Surface and Microanalysis Science Division, MML
3:00-3:20 Atmospheric Pressure MS – Tim M. Brewer, Surface and Microanalysis Science Division, MML
3:20-3:40 Improvements in Trace Involatile Vapor Analysis – Tom J. Bruno, Applied Chemicals and Materials Division, MML
3:40-4:00 Unified Organic, Inorganic, and Morphological Analysis of Forensic Samples via SEM-based, High-Resolution X-ray Spectroscopy – W. B. Doriese, Quantum Electronics and Photonics Division, PML
4:00-5:00 Optional Open House and Trace Analysis Lab Tours of NIST Facilities
4:00-5:00 Poster Viewing/Exhibitor Displays
• Ethanol in Water Standard Reference Materials to Support Forensic Testing, Michele M. Schantz, Analytical Chemistry Division
• Towards Improvement of Trace Detector Screening for the Analysis of Illicit Drugs, L.T. Demoranville, J.R. Verkouteren, G. Gillen
• Nano Particle Generation from Heated Explosives, Robert Fletcher, Marcela Najarro, Tim Brewer, Matthew Staymates and Greg Gillen
• Techniques for the Production of Standard Explosive Test Particles, Matthew Staymates, Michael Verkouteren, Jessica Staymates, Robert Fletcher, Tim M. Brewer, and Greg Gillen
• Analysis of Trace Quantities of Explosive Materials Using Laser Diode Thermal Desorption-Atmospheric Pressure Chemical Ionization-Tandem Mass Spectrometry, Eric Windsor
• Forensic Applications of DART MS, Ed Sisco, USACIL/NIST
• Electrostatic Effects in Swipe Sampling, R. Fletcher, NIST
• Age Dating of Fingerprints, Ed Sisco
• Forensic Analysis Methodology and Database of Statistically Combined HME Thermal, Mass, and Spectral Signatures, Ashot Nazarian
• Surrogate Controls for Confidence in Field Measurements, Vang
• HPLC for Quant of Explosives and Narcotics Standards, Tim Brewer
• SRMS for Trace Explosives, Bill MacCrehan
• Inkjet Printing for Trace Detection Standards, Greg Gillen
• Micro CT Scanning of Explosives, Greg Gillen
• Confocal Raman of Single Particles, Chris Michaels
• Fundamental Measurements for Trace Detection of Energetic Materials and Fire Debris, Tara M. Lovestead, Jason A. Widegren, Samuel Allen, and Thomas J. Bruno
• Reproducible Dynamic Vapor-Time Profiles of Explosives and Nonexplosive Canine Training Aids, Bill MacCrehan, Michele Schantz, Stephanie Moore; Chemical Sciences Division
• Optimization of Negative Chemical Ionization Mass Spectrometry for Explosives for Two Gas Chromatographic/Mass Spectrometric Instruments from Different Manufacturers, Bruce A, Benner, Jr. and Marcela Najarro
NIST Research Example Focus Areas

- Energy
- Healthcare
- Environment
- Information Technology and Cybersecurity
- Manufacturing
- Physical Infrastructure

All photos courtesy Shutterstock.com