Standards and Certifications

2019 EPA REGION 4 QUALITY ASSURANCE TRAINING

Standards

- For NAAQS monitoring, calibration standards will need to meet NIST-traceability specifications found in 40 CFR Part 58, Appendix A
- When purchasing standards, the intended use of the standards should be considered when determining the accuracy specifications needed
  - For example, audit standards should be of higher accuracy than working standards

Types of Standards

- Photometers
- Calibration gases (cylinders)
- Calibrator Mass Flow Controllers (MFCs)
- Other flow rate devices
- Thermometers
- Barometers
- Mass reference standards (i.e., check weights)
- Others

Standard Certifications

- Traceability protocols are available for certifying a working (field or lab) standard against a primary standard
- At the national level, two performance evaluation programs exist to further ensure viability of standards
  - For gaseous standards – Protocol Gas Verification Program
  - For photometers – Standard Reference Photometer (SRP) Program

Standard Certifications

- In general, standards should be certified in-house against an agency-maintained primary standard, or returned to the vendor
- Annual (i.e., 365 days) certification for most standards; some may require more frequent certifications
  - Use QA Handbook, Appendix D (Data Validation Templates) as a guide
  - If not listed, use manufacturer recommendations
- Certificate (**record**) documenting NIST-traceability needed for **each standard**

Standard Certifications

- Original copies of traceability certificates and other vendor-supplied records should be maintained at the central office
- As a best practices, copies can be maintained at the field sites for quick reference

Tracking Certifications

Equipment Calibration and Certification Schedule
National $PM_{2.5}$ Weighing Lab
10/3/2014

Instrument or Device	Serial number	INT	Last Certification Date or Purchase Date	Needs to be Calibrated or replaced by:	Submitted for calibration by and date
Secondary Sartorius Microbalance	81204851	6 Mon.	07/21/14	01/19/15	
Tertiary Sartorius Microbalance	81107171	6 Mon.	07/21/14	01/19/15	
FH625 Main UNIT	13149009	N/A	06/14/13	N/A	
R200 Sensor Probe	13144066	1yr	09/30/14	09/29/15	
R200 Sensor Probe	14237105	1yr	08/28/14	08/27/15	
Thomas Temp and % & RH	9904590	1yr	02/12/14	02/11/15	
Dickson FH525	11234248	1yr	09/01/13	08/31/14	May not be using in the future.
Working Std Weights (set1)	SN-476C/3TP4	1yr	02/10/14	02/09/15	
Working Std Weights (set2)	SN-476D/1I16	1yr	04/08/14	04/07/15	
Primary STD Weights (set 3)	SB-49WX/49WY	1yr	02/10/14	02/09/15	
Dickson WT240	11027130	1yr	08/12/14	08/11/15	
Humidity canister	154016P1	5 Mon.	09/11/14	02/08/15	Liebert #154016P1/ Checked 10/25/13
Lab Filter	N/A	1yr	09/11/14	09/10/15	Puracell PUGB162043AX 16X20X4
Staticmaster Polonium Strips	2U500	6 Mon.	06/01/14	11/30/14	
Staticmaster Polonium Brush	1C200	6 Mon.	06/01/14	11/30/14	
Control Comp. IR Temp Guns	122622280	1yr	01/03/14	01/02/15	
Control Comp. IR Temp Guns	101883781	1yr	09/19/13	09/18/14	Sent off 09/24/13

* Not required under SOP for room monitoring, only used for quick comparisons. These will be recalibrated in-house using calibration software.

Order polonium from Rice Lake 800-472-6703

Do not use standards that have expired!

Maintain a spreadsheet, database, or other mechanism to keep track of the certification and/or expiration dates of all standards utilized within the network

Calibration Standards

- To complete calibrations, the organization must procure calibration standards (equipment and reagents)
- Quality / sensitivity of the standards procured depends on the monitoring objectives (intended use). Examples:
  - NCore vs SLAMS vs source-oriented
  - Audit standards versus calibration standards
- If monitoring for NAAQS, the calibration standards must meet the requirements specified in 40 CFR Parts 50 and 58

Gaseous & Flow Rate Audit Standards Requirements Recap

TABLE 2-2. NIST and VSL[a] Primary Gas Mixture Suites that are declared to be Equivalent

Certified component	Balance gas	Concentration[b] range for gas mixture suite	Maximum allowable difference[c]	Stability period (years)
Carbon dioxide	Nitrogen	10 ppm to 20 %	0.3 % relative	3
Carbon dioxide	Air	100 to 500 ppm	0.5 % relative	3
Carbon monoxide	Nitrogen or Air	1 ppm to 10 %	0.3 % relative	3
Ethanol	Nitrogen or Air	75 to 500 ppm	0.5 % relative	3
Hydrogen sulfide	Nitrogen	10 to 1000 ppm	1.0 % relative	2 or 3[f]
Natural gas[d]	Nitrogen	Typical	0.5 % relative (0.3 % for CH_4)	3
Nitric oxide	Nitrogen	0.5 ppm to 1 %	0.5 % relative	2 or 3[f]
Nitrogen dioxide	Nitrogen or Air	10 ppm to 1 %	0.5 % relative	2
Oxygen	Nitrogen	10 ppm to 25 %	0.2 % relative	3
Propane	Nitrogen or Air	1 ppm to 1 %	0.3 % relative	1, 2 or 3[f]
Sulfur dioxide	Nitrogen	1 ppm to 1 %	0.5 % relative	2 or 3[f]
Volatile organic compounds[e]	Nitrogen	1 ppb to 1 ppm	2 % relative	2

[a] Information about Reference Gas Mixtures can be obtained from:
VSL [i.e., the Van Swinden Laboratorium, the National Metrology Institute of the Netherlands]
Thijsseweg 11, 2629 JA Delft, NL — P.O. Box 654, 2600 AR Delft NL
Telephone: 31 (0) 15 269 1550 — FAX: 31 (0) 15 261 2971
E-mail: vsl@vsl.nl — Website: www.vsl.nl
[b] Within the listed ranges, any concentration is available. PRMs are prepared individually in 5-L cylinders according to ISO Standard 6142 (Gas Analysis–Preparation of calibration gas mixtures-weighing methods). After preparation, the composition is verified against VSL Primary Standard Gas Mixtures. CRMs are available in larger size cylinders and are gravimetrically prepared by an accredited supplier. They are certified by VSL against VSL Primary Standard Gas Mixtures.
[c] Maximum allowable difference between NIST and VSL primary standard gas mixture suites.
[d] Methane, ethane, propane, n-butane, iso-butane, n-pentane, iso-pentane, 2,2-dimethylpropane (neopentane), n-hexane, carbon dioxide, and helium.
[e] Ethane, ethene, propane, propene, iso-butane, iso-butene, 1-butene, n-butane, 2-methyl butane, iso-pentane, n-pentane, 1-pentene, 1,3-butadiene, trans-2-pentene, 2-methyl pentane, 2,2,4-trimethyl pentane, n-hexane, n-heptane, benzene, toluene, n-octane, and o-xylene.
[f] Stability period is dependent on the concentration of the PRM/CRM.

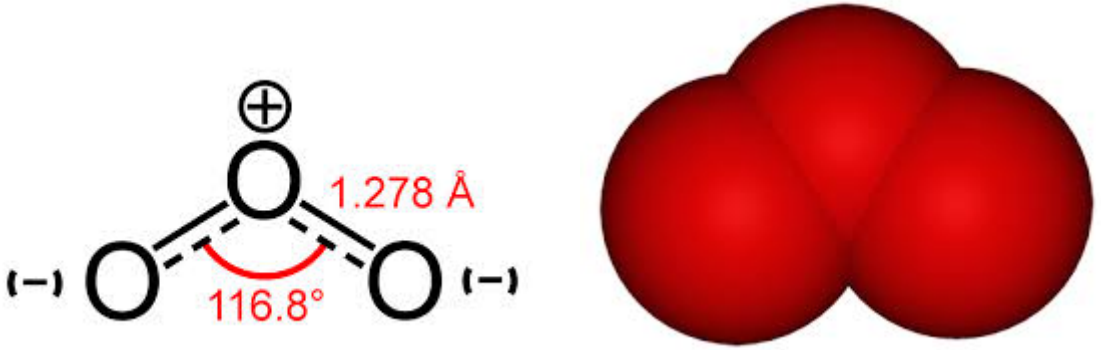
40 CFR Part 58, Appendix A, §2.6.1

Gaseous pollutant concentration standards used to obtain test concentrations for CO, SO_2, NO, NO_2 **must be traceable** to either a NIST-Traceable Reference Material or a NIST-Certified Gas Manufacturer's Internal Standard

Gaseous & Flow Rate Audit Standards Recap, Continued

40 CFR Part 58, Appendix A, §2.6.2

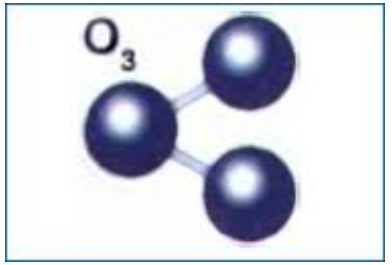
Test concentrations for ozone (O_3) must be obtained in accordance with the ultra violet photometric calibration procedure specified in Appendix D to Part 50, and by means of a **certified O_3 transfer standard**



Ozone is unlike most of the gaseous pollutants in that there are no gaseous calibration standards available

Ozone is unstable, which makes it impossible to produce gas cylinders of standardized ozone concentrations

The only means available for the calibration of ozone monitors is to produce stable, known amounts of ozone at the site of calibration



Generating Ozone

- Ozone is most commonly produced by irradiating oxygen with an ultraviolet light source in an ozone generator
- Varying concentrations of ozone can be obtained by the dilution of a stable ozone source (from an ozone generator) with varying amounts of zero air
- The ozone concentration is read by a **photometer**, which is the certifiable transfer standard

Gaseous & Flow Rate Audit Standards Recap, Continued

40 CFR Part 58, Appendix A, §2.6.3

Flow rate measurements **must** be made by a flow measuring instrument that is **traceable** to an authoritative volume or other applicable standard

What is traceability?

Traceability

- Is the property of a measurement result whereby the result can be related to a stated reference through a documented **unbroken chain of calibrations / comparisons**, each contributing to the measurement uncertainty
- Is the ability to verify the history, location, or application of an item by means of documented recorded identification

Calibration to a traceable standard can be used to determine an instrument's bias, precision, & accuracy

Traceability – 40 CFR Part 50

40 CFR 50.1(h):

"**Traceable** means that a **local standard** has been compared and certified either directly or via not more than one **intermediate standard**, to a **primary standard** such as a National Bureau of Standards Standard Reference Material (NBS SRM), or a USEPA/NBS-approved Certified Reference Material (CRM)."

Traceability – 40 CFR Part 58*

40 CFR 58.1:

"**Traceable** means that a **local standard** has been compared and certified, either directly or via not more than one **intermediate standard**, to a **NIST-Certified primary standard** such as a NIST-Traceable Reference Material or a NIST-Certified Gas Manufacturer's Internal Standard."

*Revised in 2016

Standard traceability is the process of transferring the accuracy or authority of a primary standard to a field-useable standard

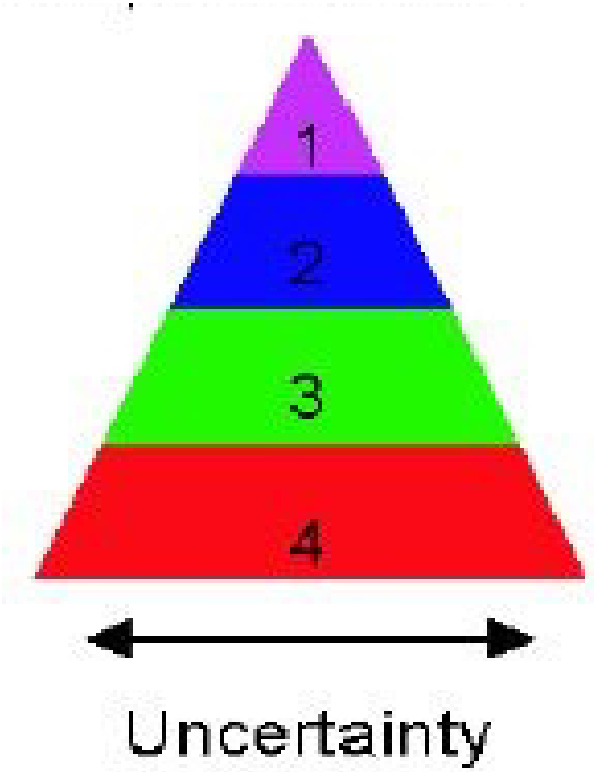
The slides that follow will shed light on the terminology in 40 CFR Parts 50 & 58, as well as various EPA guidance documents

National Institute of Standards and Technology (NIST)

- Originally founded in 1901 as the National Bureau of Standards (NBS)
- The highest authority / accuracy lies with NIST
- The NIST keeps a set of standards that is referenced by all manufacturers of glassware, standard equipment, and electronic primary standards
- Called a “Level 1” standard in some EPA guidance documents

National Institute of Standards and Technology

Measurement uncertainty grows with each step away from the authoritative standard (NIST)

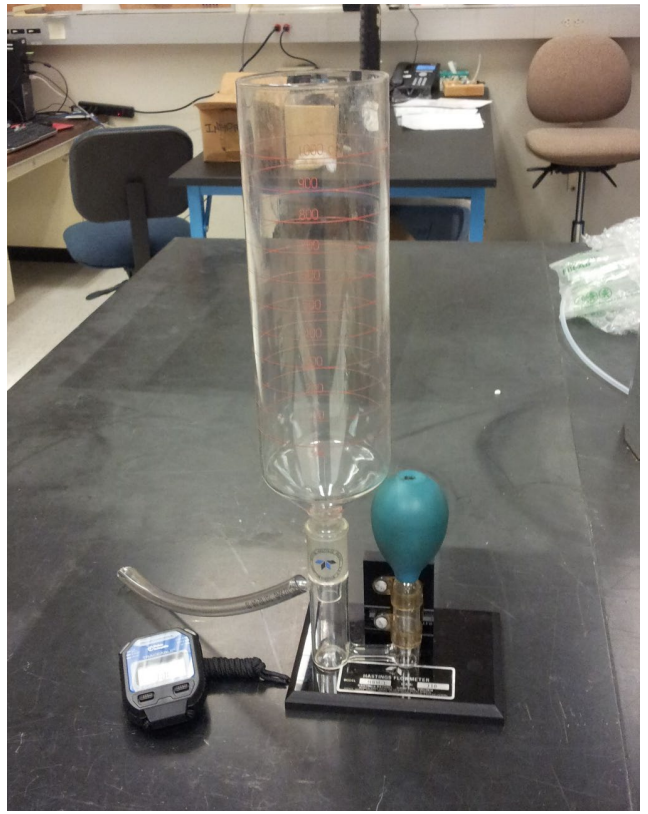


Primary Standard

- By definition, a primary standard is an accurate standard that is not calibrated by or subordinate to other standards
- Primary standard meters are those whose volumes can be determined by measurement of internal physical dimensions alone

Primary Standard

- Usually expensive
- Should be maintained, stored, and handled in a manner that maintains its integrity
- Should be kept under secure conditions
- Often used to calibrate, develop, or assay working of subordinate standards



Glass bubble meter

Transfer Standards

- Term used to describe where traceability to a higher standard has been "**transferred**" to a subordinate standard
- Also described as a **transportable device** or apparatus which, together with associated operational procedures, is capable of accurately reproducing pollutant concentration standards (see O_3 TAD)
- Transfer standards are certified against a NIST / primary standard
- Term refers to a variety of different devices (e.g., ozone transfer standard, flow transfer standard (FTS), among others)
- May also be referred to as Levels 2, 3, or 4 standards in other EPA guidance documents

Transfer standards are often used to perform audits or verifications, which means they travel to field sites across the monitoring network. In many cases, an auditor must carry the transfer standard into a monitoring shelter or atop a platform.

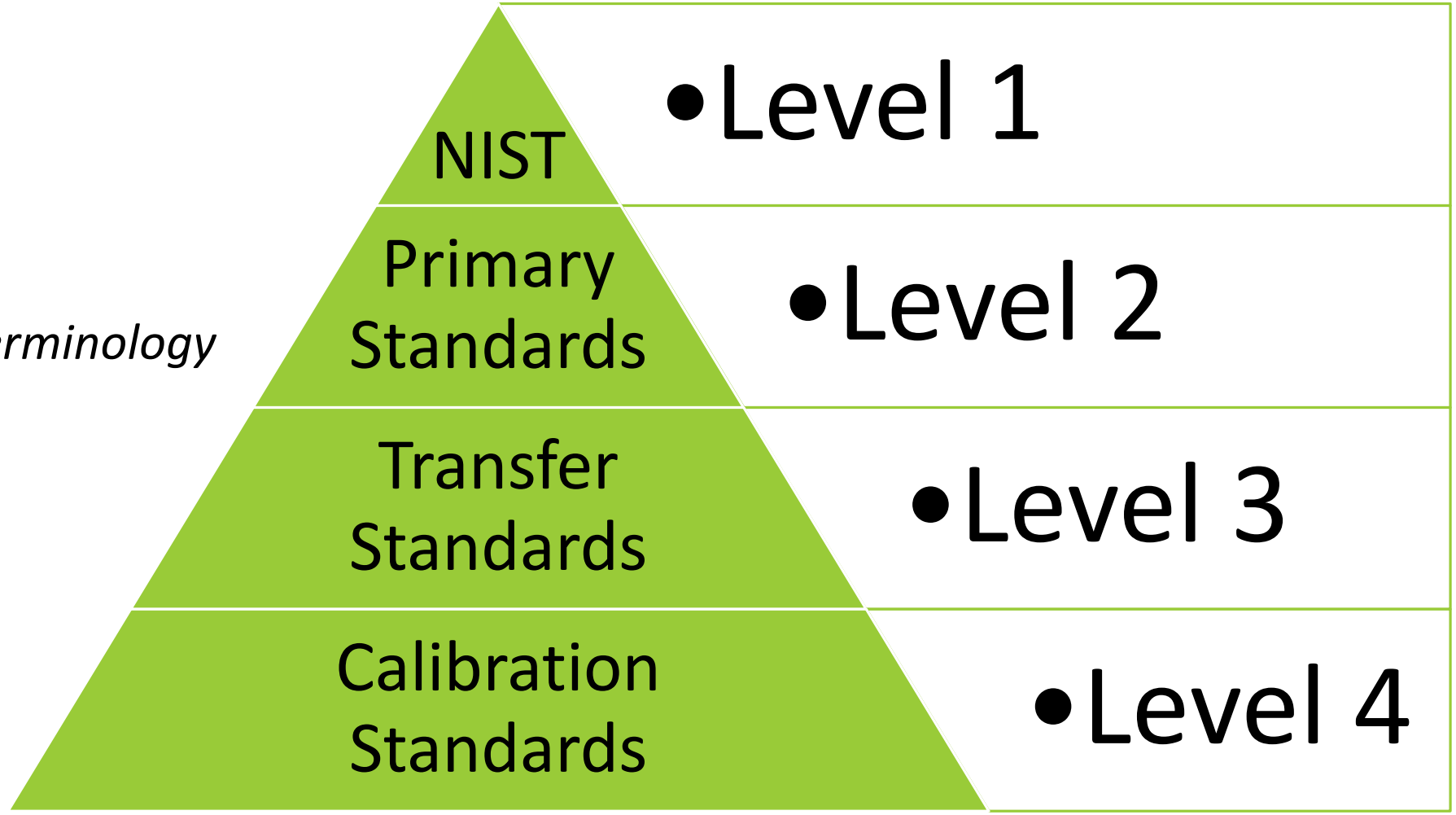
Calibration Standards

- General term used to describe the field or laboratory standards used for conducting calibrations (includes equipment and reagents)
- May also be referred to as “working standards” in some EPA documents
- Some calibration standards are transfer standards, and may be 2 or more steps away from NIST in the traceability chain
- As a best practice, monitoring organizations should maintain two sets of calibration standards – one for calibrations and the other for QC checks (verifications)

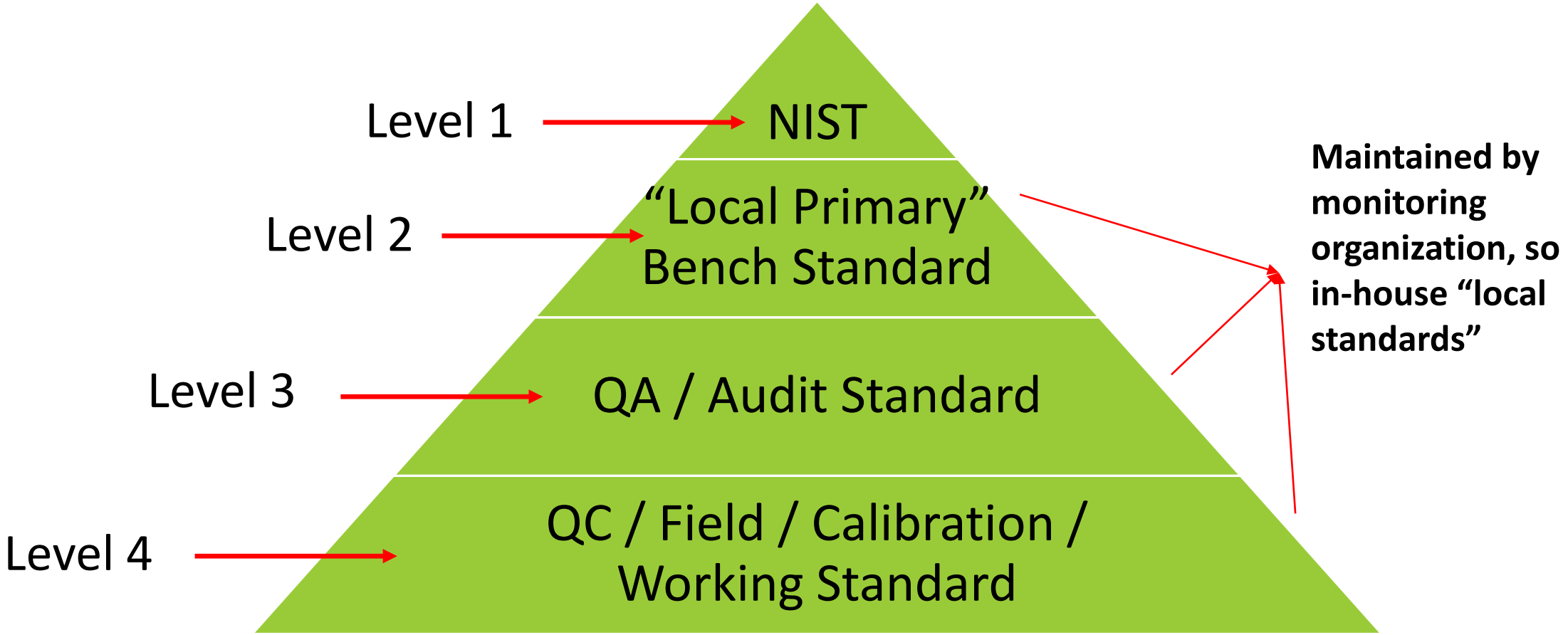
Hierarchy of Standards

Newer Terminology

Historic Terminology



Your agency may call standards by a variety of names
EPA encourages the use of the Levels 1 – 4 terminology



Example: Hi-Volume Flow Standards Hierarchy

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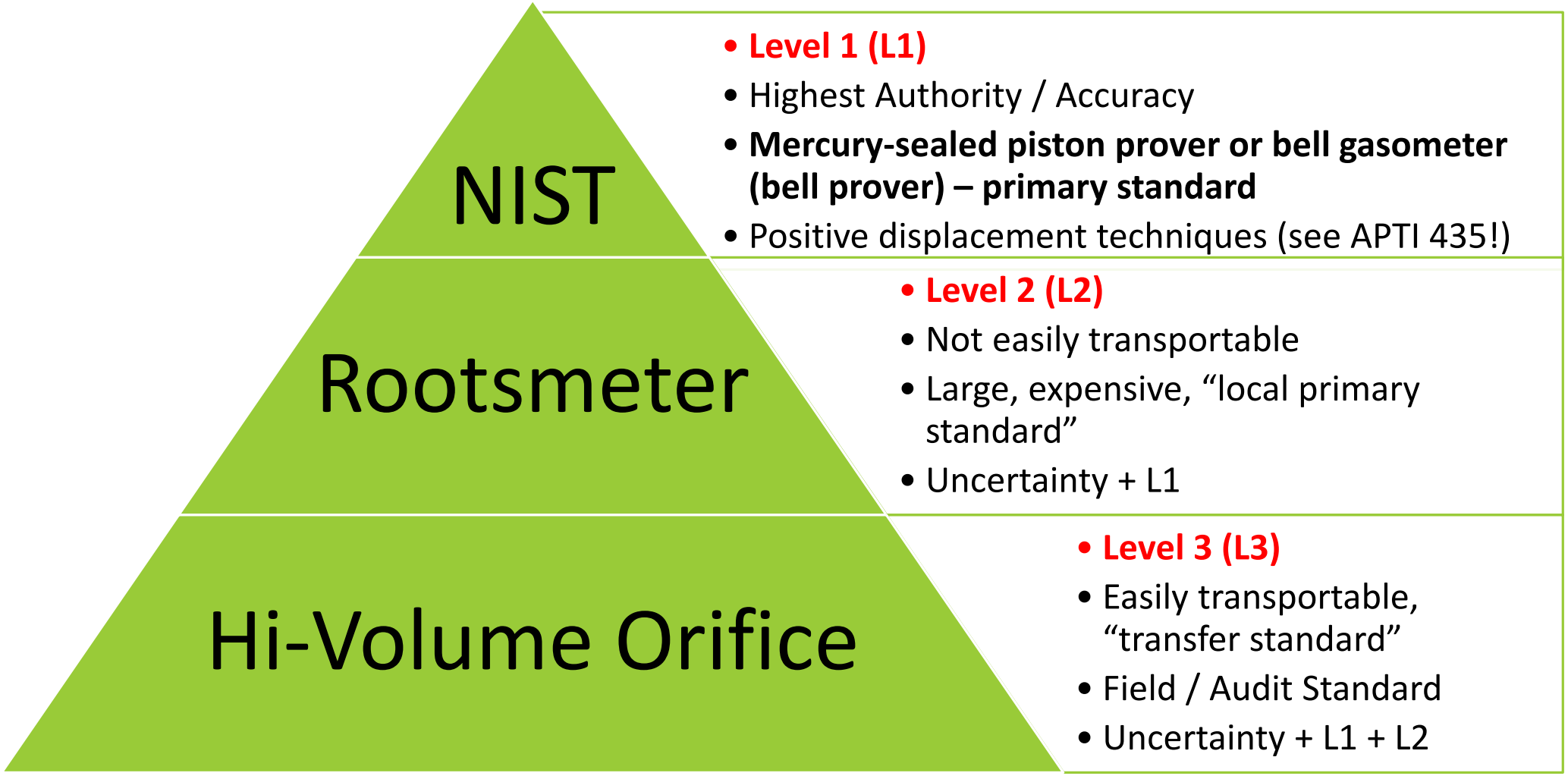
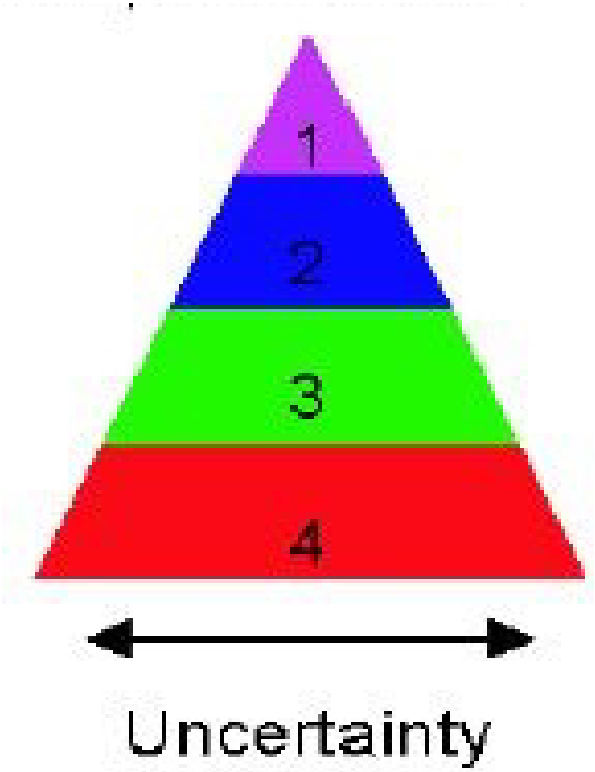


- **Level 1 (L1)**
- Highest Authority / Accuracy
- **Mercury-sealed piston prover or bell gasometer (bell prover) – primary standard**
- Positive displacement techniques (see APTI 435!)

- **Level 2 (L2)**
- Not easily transportable
- Large, expensive, “local primary standard”
- Uncertainty + L1

- **Level 3 (L3)**
- Easily transportable, “transfer standard”
- Field / Audit Standard
- Uncertainty + L1 + L2

Monitoring organizations should **avoid** using standards that are **greater than Level 4**



Standard Certification Process

Generally, the certification procedure should:

- Establish the concentration of the local standard relative to the primary standard over the appropriate range identified by the user
- Certify that the primary standard (and hence the local standard) is traceable to NIST
- Include a test of stability of the local standard over several days
- Specify a recertification period for the local standard

Standard Certification Process

- A goal of a **QA Program** is to assess and control measurement uncertainty
- Recertification of standards is a process that helps achieve that goal
- The QA Handbook, Appendix D provides guidelines for recertification schedules for many types of standards

The QAM and/or QA staff may be responsible for tracking the annual certification of standards, as well as filing the certification records

In some cases, the QAM or QA staff may perform certification procedures directly

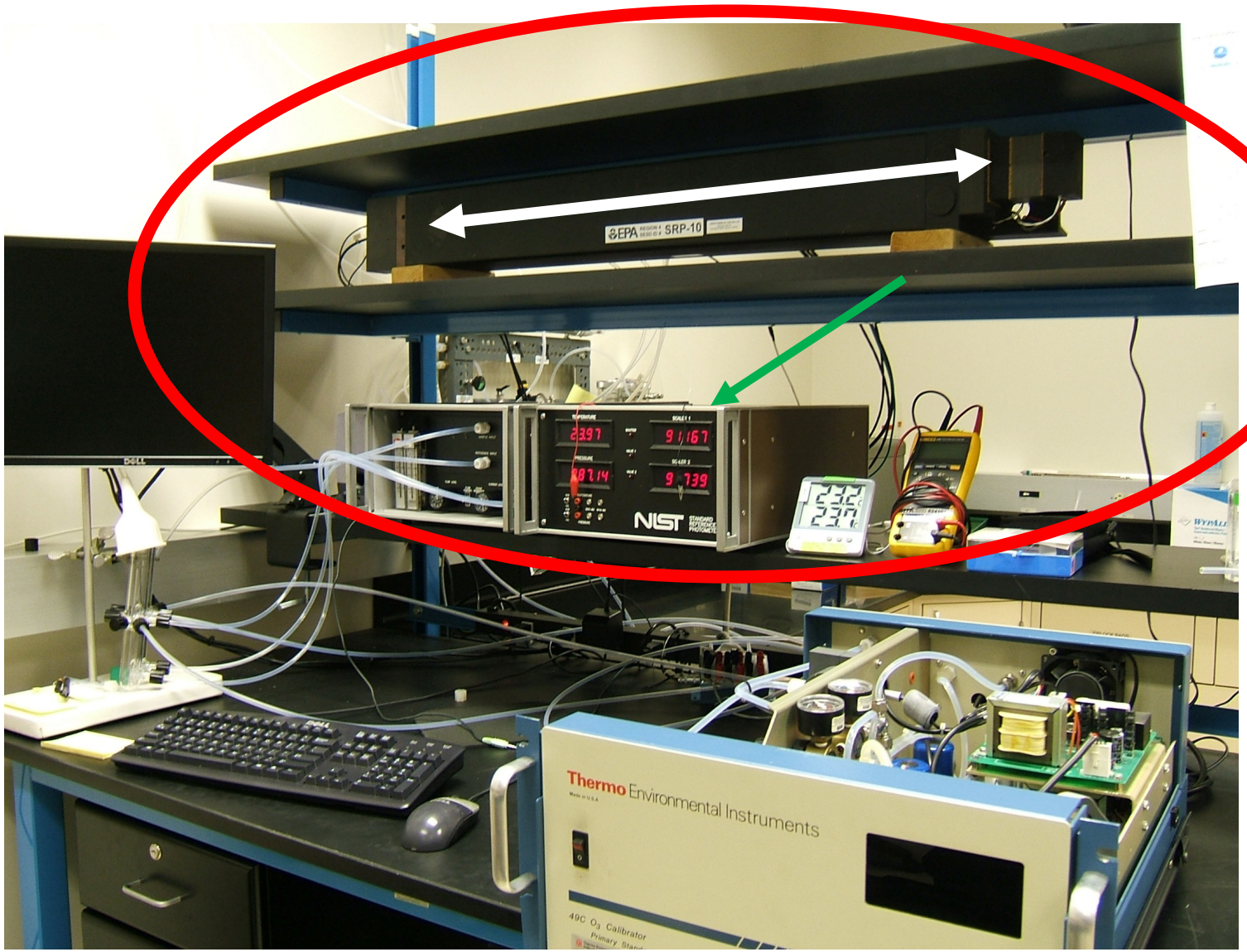
Such procedures should be included in the QAPP and detailed in a standards certification SOP

Standard Reference Photometer (SRP) Program

- Ultraviolet (UV) photometry is the most accepted technique for assaying ozone calibration atmospheres in the sub-ppm concentration range to obtain primary ozone standards
- EPA has adopted UV photometry as the prescribed procedure for calibration of reference methods to measure ozone
- The ozone calibration procedure specifically allows the use of **transfer standards** for calibrating ozone monitors
- Transfer standards must be suitably referenced to a UV standard of higher authority and traceability – i.e., the SRP

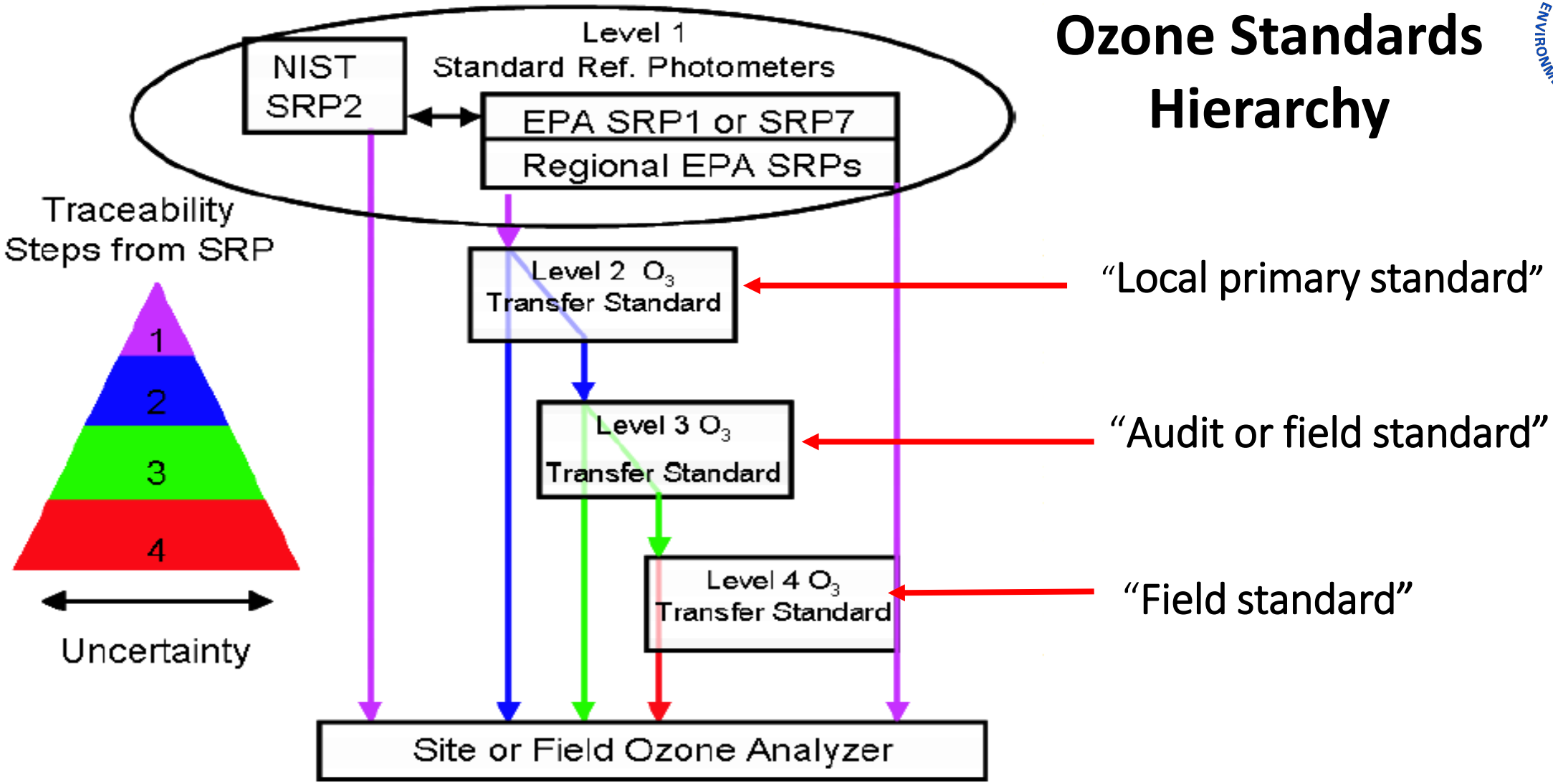
SRP Program

- NIST has produced multiple SRP
- All are equal primary standards
- EPA owns several SRP
- Authority maintained by comparison to other SRPs
- SLTs level 2 standards transfer the SRP authority to local standards
- This process is described in more detail in the EPA Ozone TAD



The Standard Reference Photometer (SRP) is a NIST-equivalent primary standard

Ozone Standards Hierarchy



Levels 2 – 4 ozone standards are all transportable devices, but the Level 2 standard should remain stationary

Standards and Certifications

Questions? Comments? Concerns?

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