Calibration Standards and Proficiency Testing Samples in NORM Analysis.

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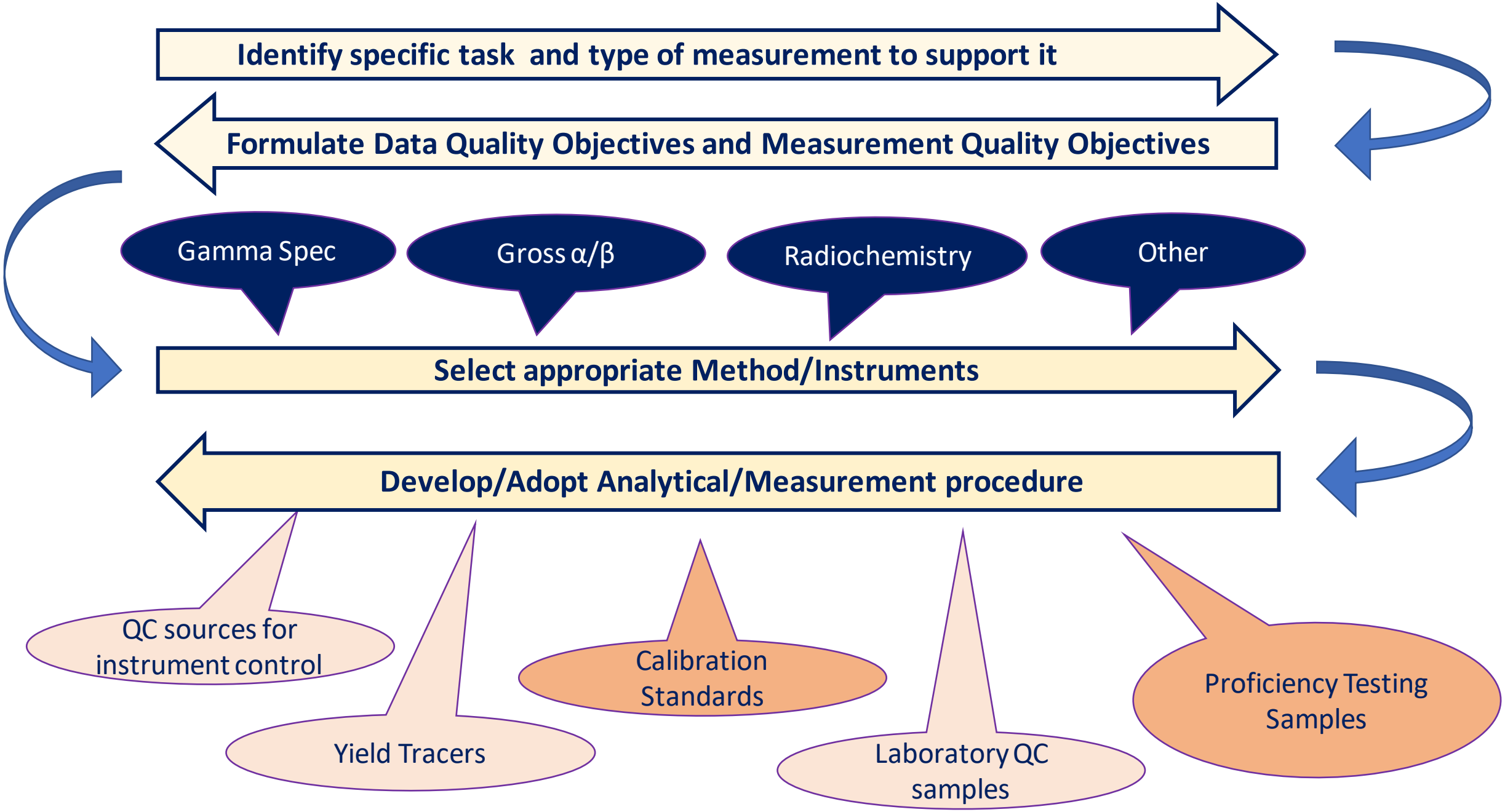
Identify specific task and type of measurement to support it
Formulate Data Quality Objectives and Measurement Quality Objectives
Gamma Spec
Gross α/β
Radiochemistry
Other
Select appropriate Method/Instruments
Develop/Adopt Analytical/Measurement procedure
QC sources for instrument control
Yield Tracers
Calibration Standards
Laboratory QC samples
Proficiency Testing Samples

Calibration and calibration standard

- Joint Committee for Guides in Metrology (JCGM) 200:2012 International vocabulary of metrology – Basic and general concepts and associated terms (VIM) 3rd edition
- 2.39 calibration:

  Operation that, under specified conditions, in a first step,

  establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step,

  uses this information to establish a relation for obtaining a measurement result from an indication
- 5.1 measurement standard, etalon:

  Realization of the definition of a given quantity, with stated quantity value and associated measurement uncertainty, used as a refer
- 5.7 working measurement standard:

  Measurement standard that is used routinely to calibrate or verify measuring instruments or measuring systems

- ISO 19017- 2017, Note 1 to entry:

The calibration standard should be physically, radiologically, and chemically similar to the items to be assayed, for which the activity of the radionuclide(s) of interest and all relevant properties to which the measurement technique is sensitive are known with sufficient accuracy.

- ASTM D7902-20:

Calibration source, CS, a known quantity of radioactive material, traceable to the SI via a national metrology institute, prepared for the purpose of calibrating nuclear instruments.

- In NORM analysis, calibration standards are radioactive sources used to calibrate measurement system. Depending on the measurement type, system used, and nature of the samples to be measured, one or several sources need be used for calibration. Most likely radioactive sources are prepared as a Certified Reference Materials. Commutability of the calibration standards is one of the most important their parameter.

Proficiency Testing plays a highly valuable role as it provides an objective evidence of the competence of the participant. This evidence can be used to improve the performance of the participant and/or provide confidence in the participant's ability to perform a specific measurement.

- Proficiency tests can validate the participating laboratory' s measurement method, technical training, traceability of standards, and uncertainty budgets.
- Successful participation in PT program will satisfy regulatory and accreditation bodies
- For some applications PT sample can be design to be used as whole (no subsampling).
- Annex C, ISO 17043 – "… it is the responsibility of the participants themselves to select the appropriate proficiency testing scheme and to evaluate their results correctly".

PT sample

Proficiency test item (PT item) - Sample, product, artefact, **reference material**, piece of equipment, measurement standard, data set or other information used for proficiency testing *

In NORM analysis, PT sample most likely is a radioactive source prepared as a Reference Material/Certified Reference Material and fit for certain purpose in the measurement process. Manufacturing of these sources is driven by five major parameters of the final products:

- Uncertainty of the Activity Value,
- Homogeneity,
- Stability,
- Quantity prepared,
- Commutability

* B. Brookman and I. Mann (eds.) Eurachem Guide: Selection, Use and Interpretation of Proficiency Testing (PT) Schemes (3rd ed. 2021).

PT sample – “used as a whole” option

One of the reasons for the shortage of RM on the market is the cost of manufacturing. To drive costs down, EZA have adopted “used a whole” concept for some of PT sample preparation (no subsampling or division). Sample is prepared per customer specifications regarding isotopic composition, activity level, matrix and quantity. Samples must be quantitively transfer from the shipment container for further analysis. This option provides exceptional commutability and significant manufacturing cost reduction, allowing:

- Less restrictive (if any at all) homogeneity requirements
- Flexibility in matrix choice, including perishable materials
- Flexibility in spike choice (including refractory spices, specific complexes etc.)
- Overall lower uncertainty for the activity value of isotopes used for spiked
- Manufacturing of PT within regulatory requirements (ISO 17025, ISO 17043 etc.)

Provider of PT samples must ensure that spikes are contained within the matrix and not absorbed by container material.

User must ensure quantitative sample transferred from the container for further analysis.

Reference material

- Nominal ($$) or certified value ($$$$)
- Solid, Liquid or Gas.
- Naturally existing matrices-soil, vegetation, water etc.
- Artificial matrices - filters, resins, engineered materials etc.

Spiked, induced, or natural activity

PT sample
Can be liquid, solid or gas with similar parameters as real samples and define purpose in the measurement process
( ) $-$$$

QC source for Instrument Control
$

Calibration Standard
Can be liquid, solid or gas with similar parameters as real samples. Activity value, uncertainty for the activity value and traceability must be stated
$-$$$

Laboratory QC sample
Can be liquid, solid or gas with similar parameters as real samples.
( ) $-$$

Yield Tracer
(mostly liquids)
$-$$

Commutability (fitness for intended use)..
Uncertainty of the Activity Value.....
Homogeneity, and.....
Stability.....

Common Measurements methods for NORM analysis

- Gamma spectrometry – most versatile, nondestructive, quantitative
  - Liquids, solid, gases
    - Natural or engineered matrices
    - Olivine – low background mineral with low NORM content, Σ< 12 mBq/g.
  - Sample volume – several cm^3 to 250 dm^3 in custom geometry
  - Sample densities – 0.02- 5 g/cm^3.
  - In-situ measurements – up to 1m x 1m flat sources for single source.
  - Isotopes – generic mixture for 40-2000 keV range counting efficiency calibration or project specific isotopes, including pitchblende based.
  - KUTh – sources in any geometry.
- Gross alpha/beta counting most likely to be used as screening method for solids, liquids (after evaporation) and filters.

Common Measurements methods for NORM analysis

- Liquid scintillation counting most likely to be used as screening method for liquids and filters.
- Radiochemical separation can be used with any measurement method
- Radon counting by Lucas cells and other detectors

Thank you for your attention!

Questions?