Radionuclidic Standardization by Primary Methods

An Overview

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Primary Standardizations

means

Realization of the SI unit Becquerel

- Direct measure number of nuclear transformations per unit time
- Only in terms of base SI units of frequency, time, mass (sometimes length)
- No use of other radioactivity calibration or standard
- Sometimes called “direct” or “absolute” (sic)

NIST

Who are we ?

And what do we do ?

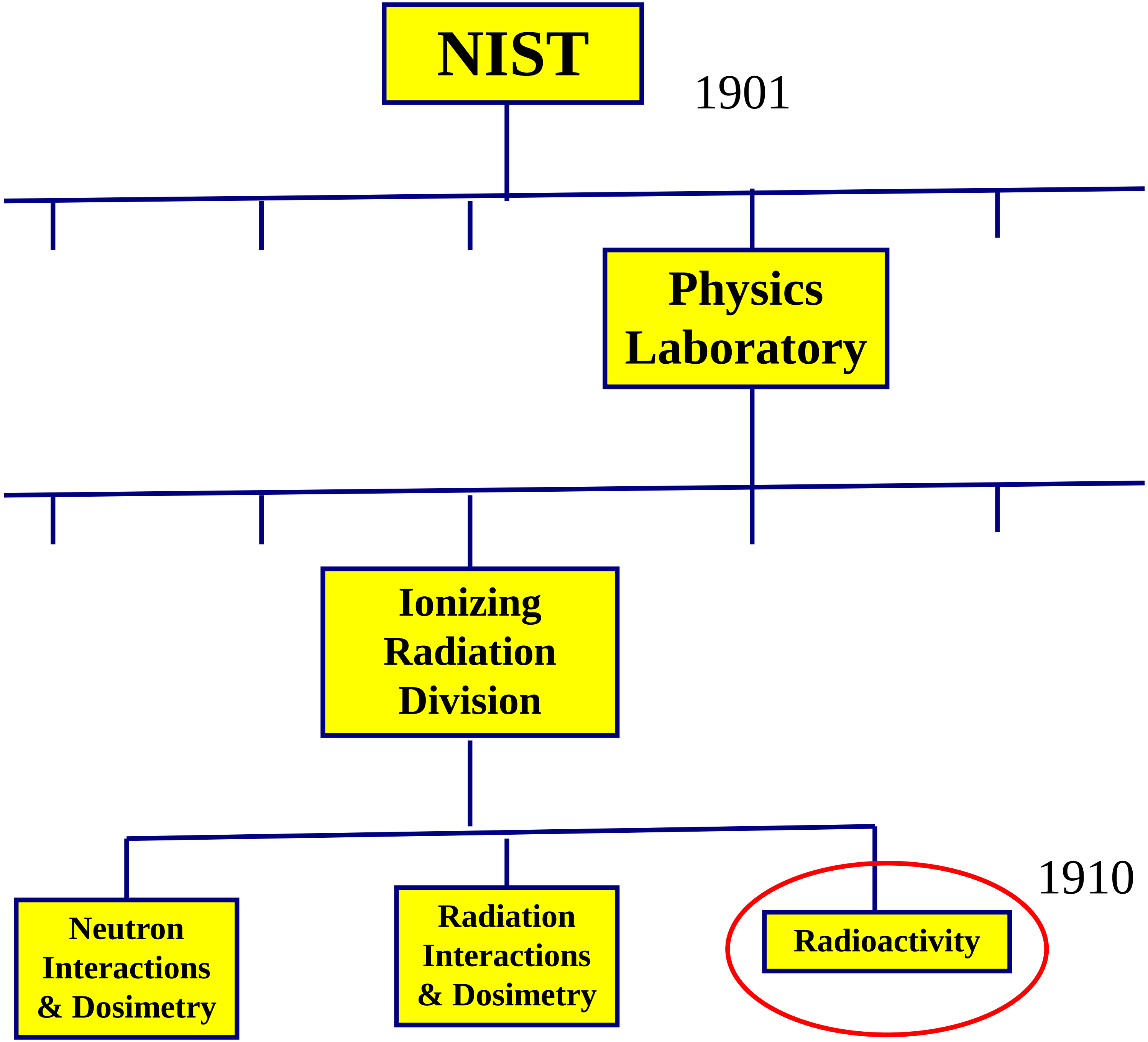
NIST (NBS)

1901 - 1998

> 1998

Highest authority in USA

- for setting physical measurement standards
- and ensuring accurate measurements


NIST
1901
Physics Laboratory
Ionizing Radiation Division
Neutron Interactions & Dosimetry
Radiation Interactions & Dosimetry
Radioactivity
1910

OUR ROLES

primary objectives

- develop and maintain national standards through primary standardizations
- develop secondary standards and transfer standards;
- disseminate standards;

    and ***provide mechanisms*** for insuring the quality of measurements

principal elements of what we do

- basic & applied research

    includes technology & method development

- standards (SRMs) and calibrations
- proficiency testing (MQA)
- international intercomparisons & collaborations

Radioactivity Group

Major Program Elements

BASIC RADIONUCLIDIC METROLOGY

Standards (SRMs) & calibrations

proficiency testing (MQA) programs

NUCLEAR MEDICINE / RADIOPHARMACEUTICAL APPLICATIONS

LOW-LEVEL ENVIRONMENTAL / BIOLOGICAL MEASUREMENTS

HOMELAND SECURITY

Metology

Metrology

- 130^{+} nuclides
- Many geometries
- > 30 systems

Standards

- 60^{+} nuclides
- 9 natural matrix (multi-nuclide)
- 500 – 1000 units per year
- by over 200 users
- $ 300K - $ 600K USD in sales per year (depends on availability)
- 20 users buy 10 or more SRMs per year

Calibrations

- > 100 routine per year
- 20 – 30 nuclides; many geometries
- $ 100K – 200K USD per year
- few special $ 30 - $ 100K USD **EACH**

MQA programs

- 17 unique
- from monthly to 2x per year

Our measurement methods and instruments include:

- Basic (primary) standardizations
- Secondary standardization methods
- Routine measurements
  (e.g., for monitoring, QC, etc.)
- Ancillary measurements
  (e.g., for impurity analyses, research, etc.)

high-pressure re-entrant ion chamber

our SRM work horse -- calibration factors for 40^{+} nuclides for 30+ years

LS counters (Beckam, Wallac, Packard) – our other SRM work horses!

3-phototube TDCR system (under development)

isothermal microcalorimeter (a low-temp 8K calorimeter is inactive)

high-resolution γ spectrometry with HPGe & Si (4 systems)

defined-geometry $0.8\pi\alpha$ counter

NaI(Tl) x-ray counting system

$4\pi\beta$(LS)-γ(NaI) anticoincidence counting system

$4\pi\beta$-coincidence counting system (inactive)

sum-peak -- 2 NaI(Tl) -- counting system

NaI(Tl) pin-well detector

coupled large (8”) NaI(Tl) detectors in 4π geometry for total efficiency counting and γ-γ coincidence counting

length-compensated proportional counters for gas counting

RIMS + TIMS

primary radon measurement system (pulse ionization chambers + gas handling)

secondary NaI(Tl)-based radon counting system

α-table with PIPS detector

large-area proportional counter & the α “pancake” counter

Various $2\pi\alpha$ surface-barrier detectors, gas-flow β proportional counters; and γ NaI(Tl) well counters

Fuji phosphor-imaging system

commercial “dose calibrator” ion chambers (6+)

windowless Si(Li) x-ray detector

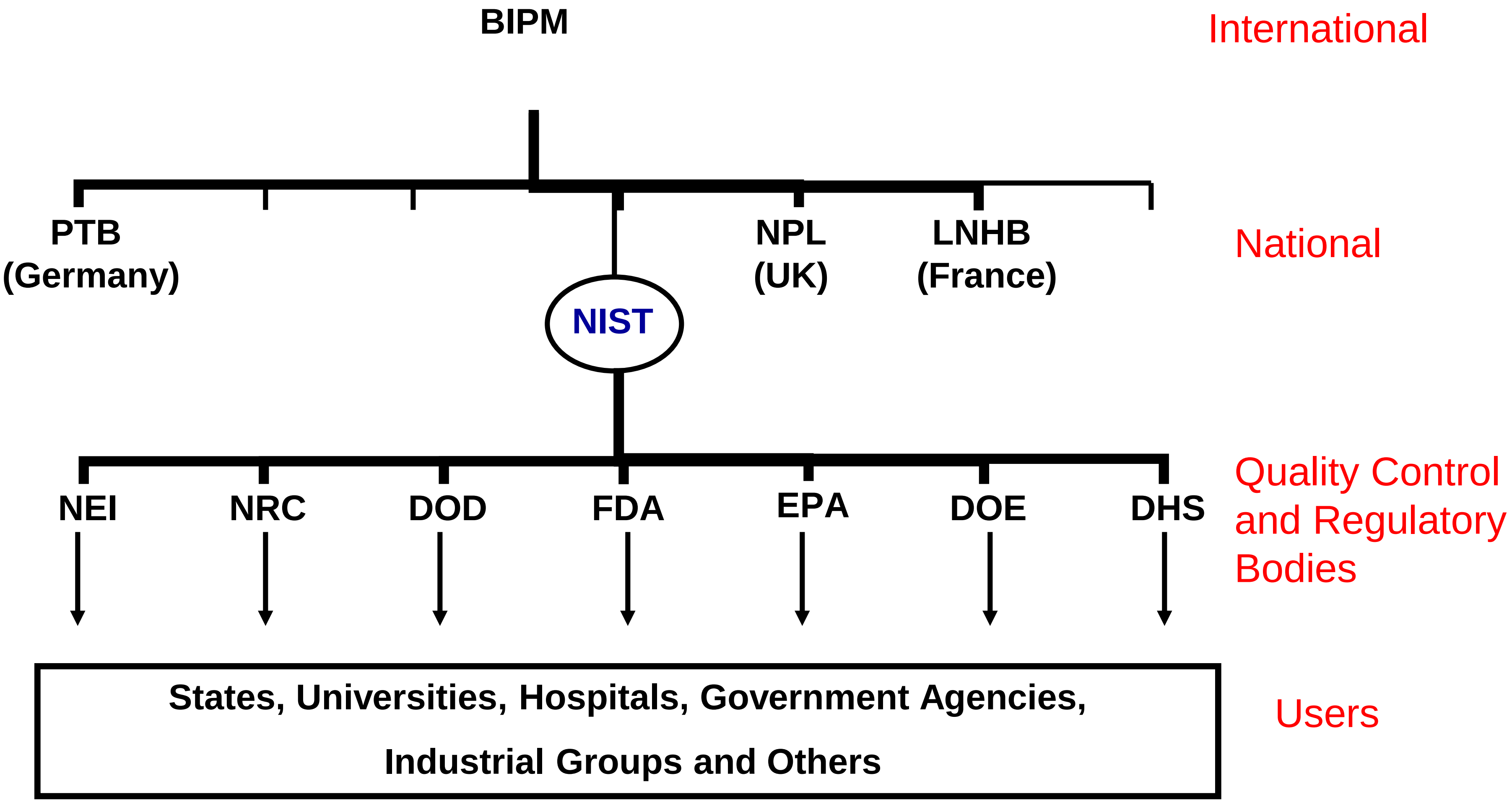
NIST measurement systems

Who are our users ?

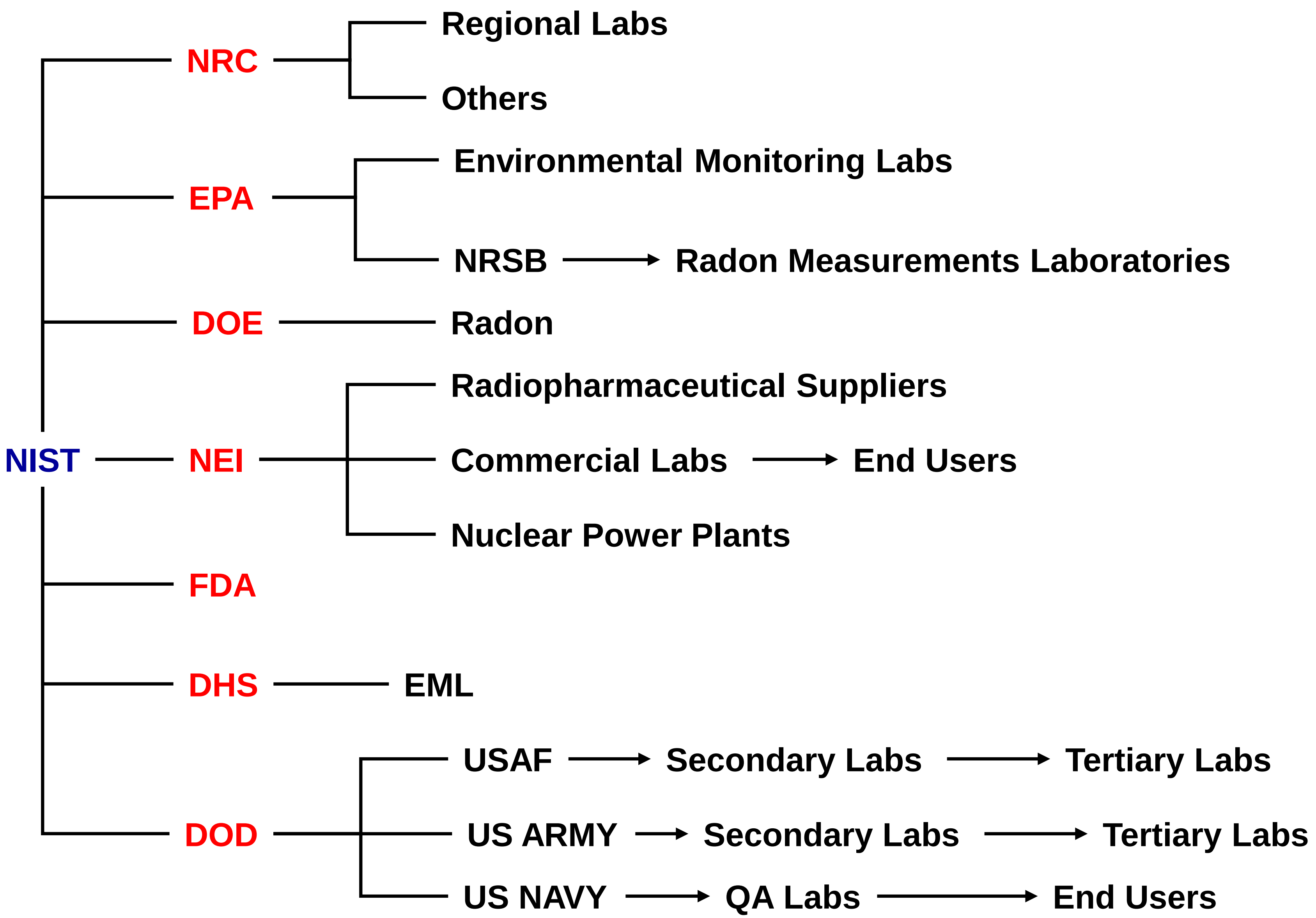
And how do we know their needs?

Many ..... domestically & other countries

International and National Radioactivity Measurements "Traceability Tree"



Traceability of Radioactivity Measurements in the U.S



1896 ← radioactivity

1898 ← polonium / radium

1901 ← NBS (NIST)

1914 ← 1st calibrations of radium

- radium / radon
- Natural series
- Ores/ minerals

c. 1950 ← Anthropogenic nuclides

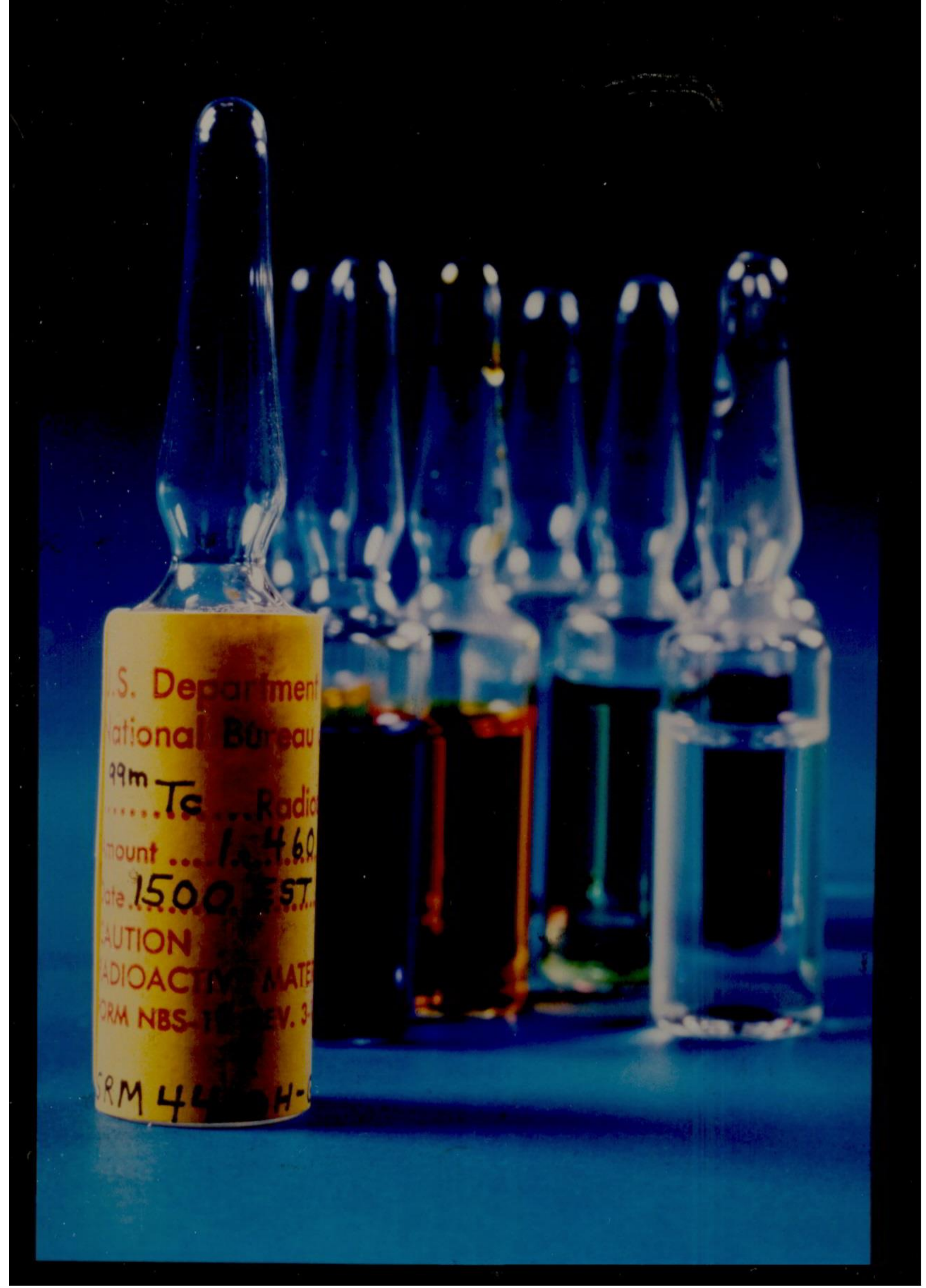
- nuclear power
- international comparisons
- golden age radionuclidic metrology

1970's - ← Specialization ("needs" studies)

- radiopharmaceuticals
- environment --- Natural Matrix Stds --
- power plant
- nuclear medicine – Therapy
- forensics -- homeland security

Our principal product...

solution standards of radionuclides



Radionuclides standardized at NIST

H-3	Fe-55	Sr-90	Sb-124	Ce-141	Hg-197	Th-229
Be-10	Co-56	Nb-93	I-124	Ce-144	Au-198	Th-230
C-14	Mn-56	Nb-94	I-125	Pm-147	Tl-201	Th-232
F-18	Co-57	Nb-95	Sb-125	Gd-148	Hg-203	U-232
Na-22	Co-58	Zr-95	Te-125m	Eu-152	Pb-203	U-234
Na-24	Fe-59	Mo-99	I-126	Gd-153	Tl-204	U-235
Al-26	Co-60	Tc-99	Xe-127	Sm-153	Bi-207	Np-237
P-32	Cu-62	Tc-99m	I-129	Eu-154	Po-208	U-238
P-33	Ni-63	Pd-103	I-130	Eu-155	Po-209	Pu-238
S-35	Zn-65	Ru-106	I-131	Eu-156	Bi-210	Pu-239
Cl-36	Ga-67	Ag-108m	Ba-131	Ho-166	Po-210	Pu-240
Ar-37	Se-75	Cd-109	Xe-131m	Ho-166m	Pb-210	Pu-241
Ar-39	Kr-79	Ag-110m	Ba-133	Yb-169	At-211	Am-241
K-40	Sr-82	In-111	Xe-133	Lu-177	Bi-214	Pu-242
K-42	Kr-85	In-113m	Xe-133m	Re-184	Pb-214	Am-243
Ca-45	Sr-85	Sn-113	Cs-134	Re-186	Rn-222	Cm-243
Sc-46	Rb-86	Sn-117m	Cs-137	Re-188	Ra-223	Cm-244
V-49	Y-88	Sn-121m	Ce-139	W-188	Ra-226	
Cr-51	Sr-89	I-123	Ba-140	Ir-192	Ra-228	
Mn-54	Y-90	Te-123m	La-140	Au-195	Th-228	

2007 Schedule for NEI Program

January	I-131	4401, lot 32	0.8 GBq	30 MBq
February	Mo-99	4412, lot 31	3.0 GBq	75 MBq
March	*open*			
April	Ga-67	4416, lot 27	0.4 GBq	20 MBq
May	Tc-99m	4410, lot 32	7.4 GBq	
June	Tl-201	4404, lot 29	0.4 GBq	33 MBq
July	*open*			
August	In-111	4417, lot 26	0.4 GBq	19 MBq
September	Xe-133	4415, lot 30	7.4 GBq	740 MBq
October	Y-90	4427, lot 10	0.2 GBq	19 MBq
November	*open*			
December	I-125	4407, lot 31	0.8 GBq	6 MBq

NIST Standard Reference Materials (SRMs) for Radionuclides Used in Nuclear Medicine and Biology

Radionuclide	SRM ID	Last Issued	Radionuclide	SRM ID	Last Issued
Chromium-51	4400N	July 1992	**Xenon-133**	4415, lot 29	September 2005
Iodine-131	4401, lot 32	January 2006	**Gallium-67**	4416, lot 27	April 2006
Tin-113-indium-113m	4402C	October 1980	**Indium-111**	4417, lot 25	August 2005
Strontium-85	4403B	April 1977	**Mercury-203**	4418A	November 1976
Thallium-201	4404, lot 28	June 2005	**Ytterbium-169**	4419C	October 1986
Gold-198	4405B	August 1978	**Lead-203**	4420B	November 1984
Phosphorus-32	4406O	October 1997	**Gold-195**	4421A	December 1979
Iodine-125	4407, lot 31	December 2005	**Chlorine-36**	4422A	April 1980
Cobalt-57	4408F	July 1995	**Strontium-90**	4423A	November 1985
Selenium-75	4409D	August 1981	**Sulfur-35**	4424A	October 1988
Technetium-99m	4410, lot 32	May 2006	**Samarium-153**	4425D	July 2002
Iron-59	4411B	January 1979	**Strontium-89**	4426A	April 1995
Molybdenum-99	4412, lot 31	February 2006	**Yttrium-90**	4427, lot 9	October 2005
Mercury-197	4413A	May 1976	**Gadolinium-153**	4428A	October 1998
Iodine-123	4414C	June 1980			

Natural Matrix SRMs

for Environmental Radioactivity Measurement

- **Rocky Flat Soil I**
- **River Sediment**
- **Peruvian Soil**
- **Human Lung**
- **Human Liver**
- **Lake Sediment**
- **Ocean Sediment**
- **Bone Ash**
- **Shell Fish**

Many other geometries for standards & calibrations

- Spiked sources (soils, synthetic urine, water)
- Filter sources
- Point Sources
- Marinelli beakers
- Radioactive gas ampoules
- Polyethlene encapsulated radon emanation sources
- Large area sources
- Dose vials
- Brachytherapy sources

Alpha-Particle Emitters Including Mixed Alpha
Beta-Particle Emitters
X- and γ-ray Emitters

Customers include: Department of Defense, Department of Energy, Environmental Protection Agency, Nuclear Regulatory Agency, National Aeronautics and Space Agency, Department of Homeland Security, Nuclear power industry, Source & instrument manufacturers, Radiopharmacies, etc..

Radioactivity Measurement Assurance Programs (MQAs)

Nuclear Energy Institute – Radiopharmaceuticals
(33 years)

Nuclear Energy Institute – Power Station Radiochemistry
(19 years)

NIST Radiochemistry Intercomparison Program (NRIP)
(8 years)

Primary methods (Pommé Classification)

realization of the SI unit Becquerel

not based or referenced to other standards or calibrations) *

high-geometry methods

•4π or 2π proportional counting of particles
•internal gas counting with length-compensated tubes
•$4\pi\gamma$ counting with large NaI(Tl) or CsI(Tl) sandwich detectors
•liquid scintillation (LS) counting
•and both classical and cryogenic calorimetry

defined-solid-angle methods

•use strictly controlled geometric constructions
incorporating a large variety of detectors with known detection efficiencies

classical *coincidence counting methods*

• including the variants of anticoincidence, sum-peak, and correlation counting,
• LS-based triple-to-double-coincidence ratio (TDCR) method.

* Exceptions: efficiency tracing by ^{3}H-standard CNET & coincidence counting (e.g. ^{99}Tc w/ ^{60}Co)coinicidence)

^{55}Fe

primary standardization by microcalorimetry

linked to SRM

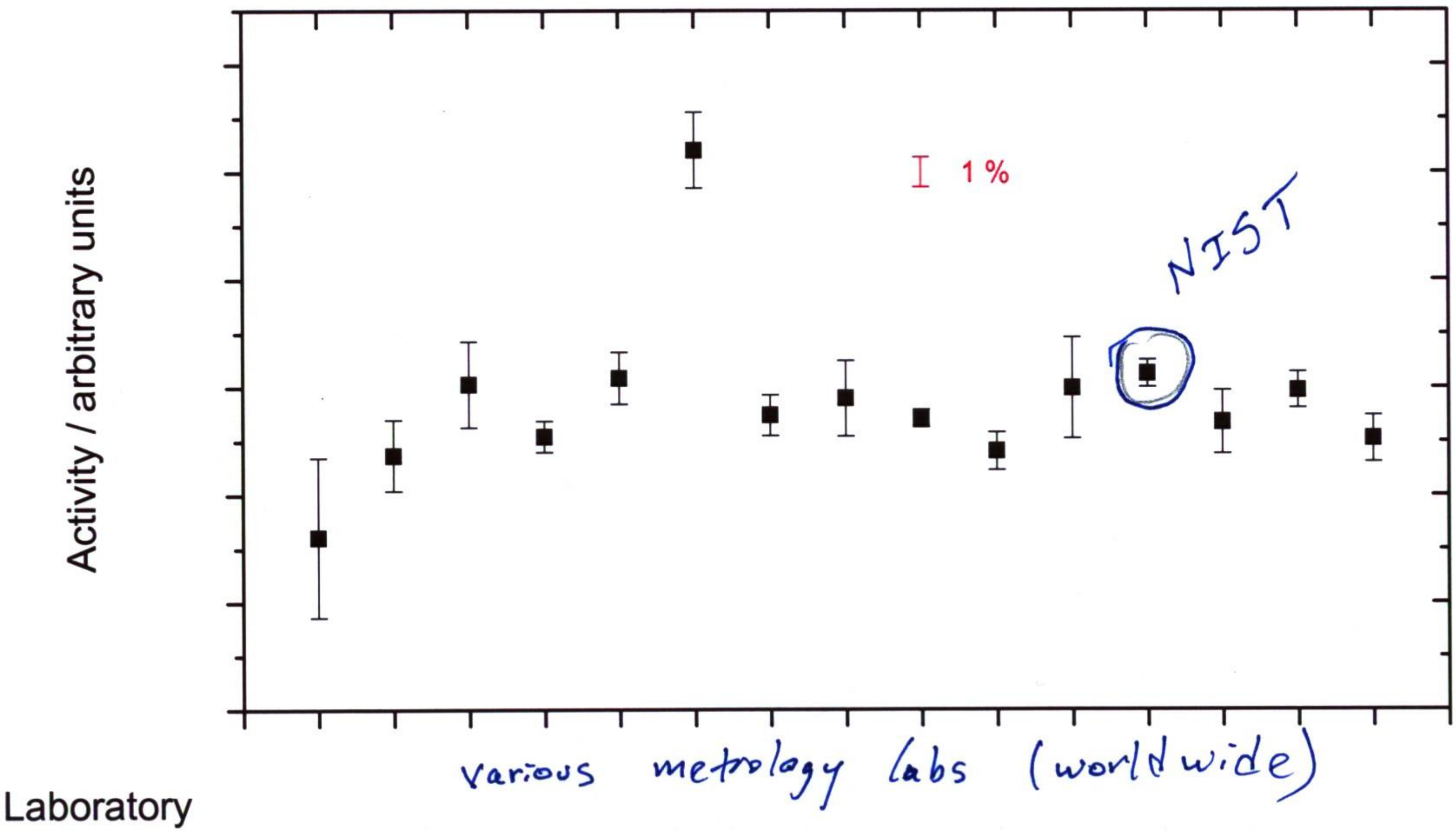
international intercomparison

NIST Uncertainty Analysis for ^{55}Fe Microcalorimetric Standardization of NIST Solution Standards

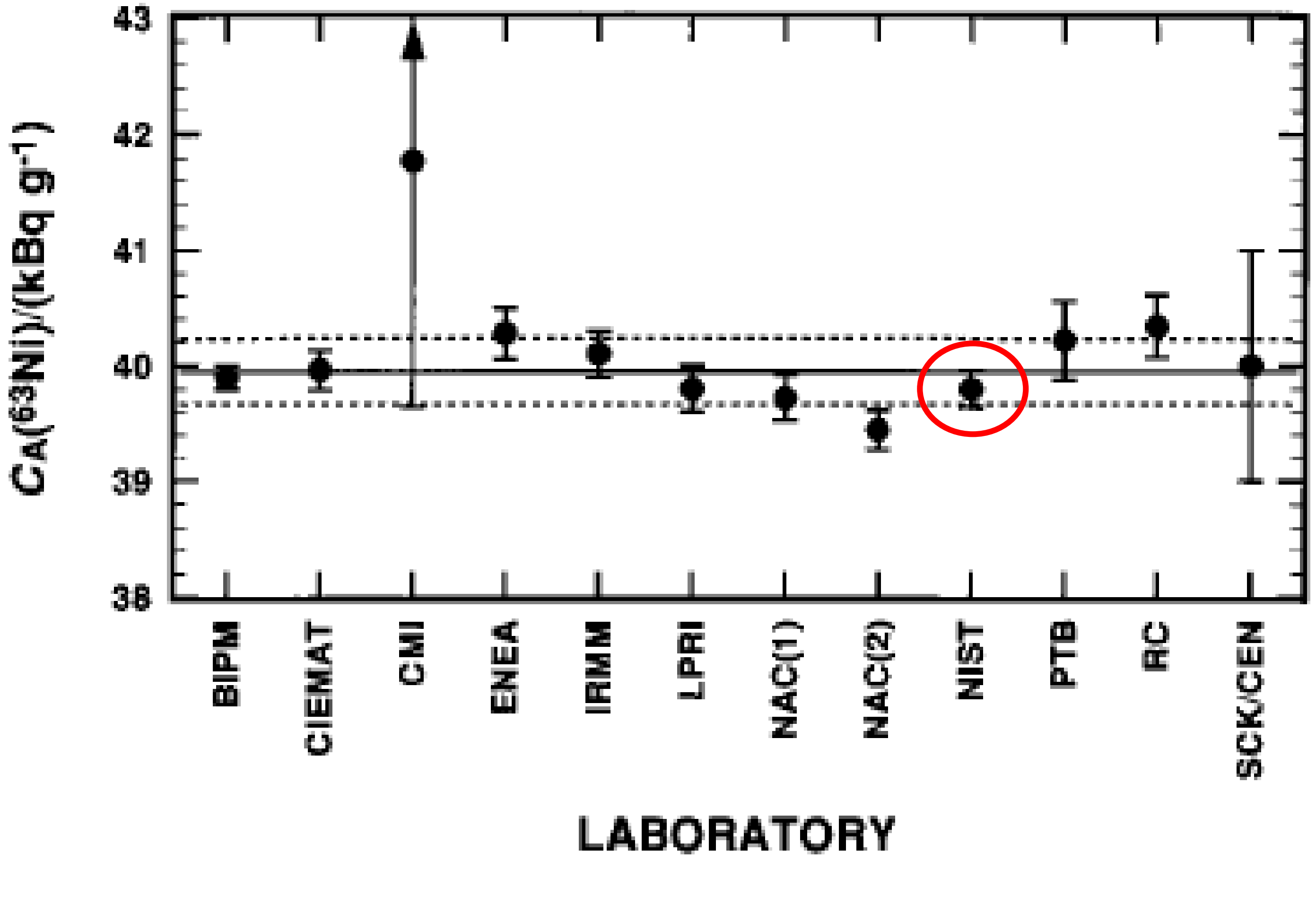
Item	Uncertainty component	Assessment Type	Relative standard uncertainty contribution on massic activity of ^{55}Fe (%)
1	Measurement precision for 13 independent calorimetric determinations of the power of solid source C; includes precision in the calibrations & baseline measurements for each determination; std. dev mean for ν=12 degrees freedom (passes Normal test)	A	0.25
2	Gravimetric (mass) linkage of source C to NIST standard solutions	B	0.07
3	Activity loss in source C preparation	B	0.15
4	Power calibration of calorimeter, includes any systemic heat losses	B	0.05
5	Possible heat defect / excess effects	B	0.1
6	^{55}Fe decay corrections during calorimetric measurements	B	0.02
7	^{55}Fe decay corrections from calorimetric reference time to BIPM reference time.	B	0.08
8	Average energy per decay for ^{55}Fe (to convert calorimetric power to activity)	B	0.17
	COMBINED STANDARD UNCERTAINTY		0.39

Results (without the outlier value)

International comparison of activity measurements of a solution of ^{55}Fe
Preliminary results; January 2007



^{63}Ni international intercomparison



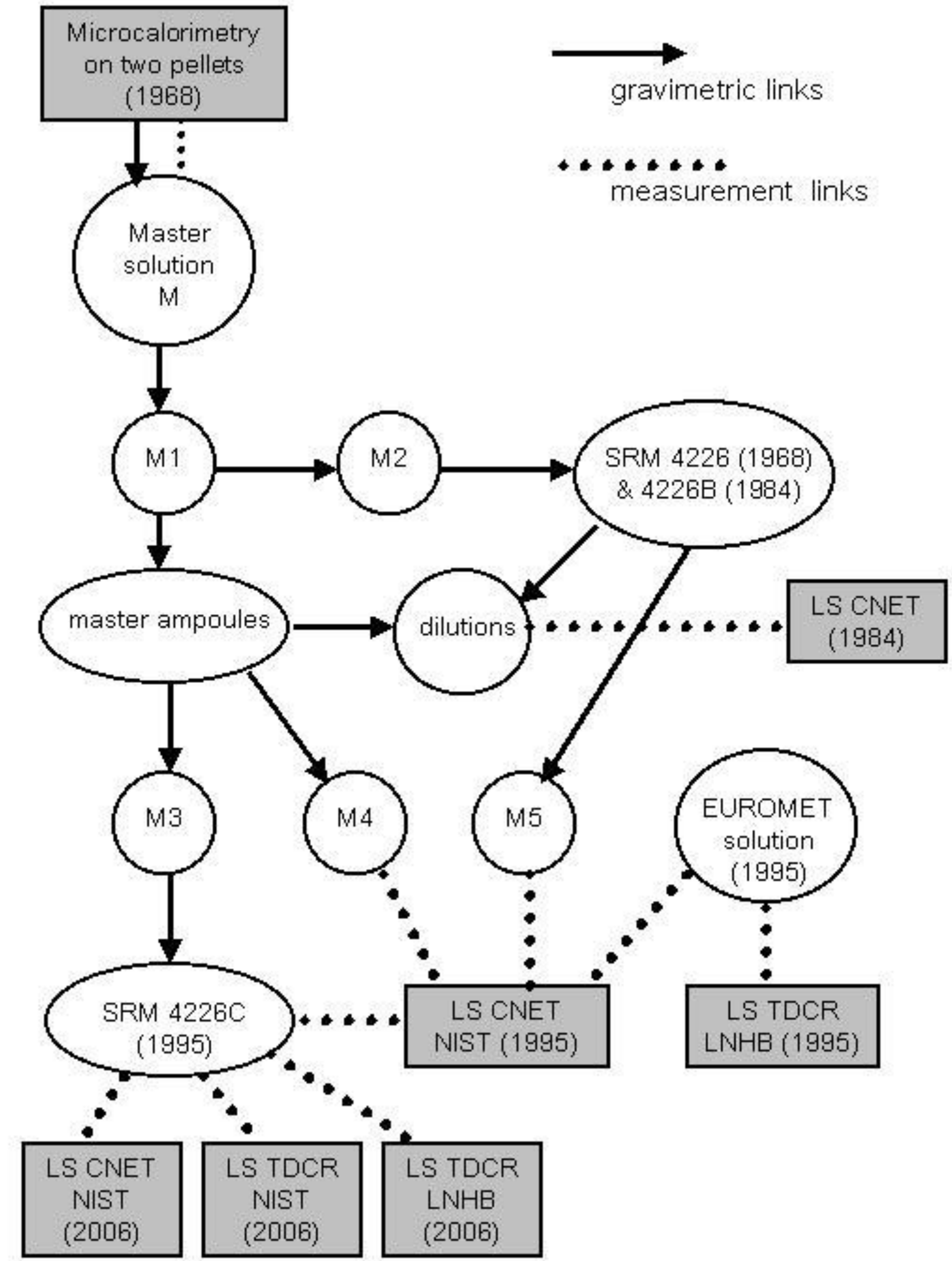
Summary of some recent NIST primary standardizations and comparison to confirmatory measurements.

^{63}Ni

Nuclide	Method	relative standard uncertainty	Confirmatory Measurement	Difference (%)
^{63}Ni	4π LS TDCR (NIST)	0.16 %	4πβ LS TDCR (LNHB) 4πβ LS CNET (NIST)	-0.31 -0.77
^{55}Fe (NIST)	4π calorimetry (linked by LS)	0.39 %	4π LS TDCR (Polatom) 4π LS TDCR (LNHB)	-0.87 -0.43
^{55}Fe (BIPM)	4π calorimetry (linked by LS)	0.39 %	weighted mean value of 15 NMI labs	-0.37
^{210}Pb	4παβ LS CNET	1.2 %	4παβ(LS)-γ(NaI) anticoin. counting ^{210}Po α spect. (102 a ^{209}Po tracer) ^{210}Po α spect. (128 a ^{209}Po tracer) HPGe photon spect.	+0.7 -3.0 -1.3 +4.7
^{241}Pu	4πβ LS CNET	1.9 %	LS (^{241}Am ingrowth) 4πβ LS TDCR (NIST) 4πβ LS TDCR (LNHB)	+1.2 -7.9 * -7.7 *
^{210}Pb	4παβ LS CNET	1.2 %	compare to NPL standard (5 methods) see Table2	-0.3
^{90}Sr	4πβ LS TDCR	0.51 %	4πβ LS CNET	+ 0.09
^{241}Am	4πα LS	0.22 %	4πα LS (independent) 4πα LS (independent)	-0.05 -0.01 -0.15
^{229}Th	4παβ(LS)-γ(NaI) anticoincidence counting	0.28 %	4παβ LS CNET 4παβ LS TDCR 2π α proportional counting HPGe photon spectrometry	-0.09 -1.7 -0.09 +2.1

* Values are discrepant, and not considered to have confirmed

38 years of ^{63}Ni results



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^{55}Fe

Nuclide	Method	relative standard uncertainty	Confirmatory Measurement	Difference (%)
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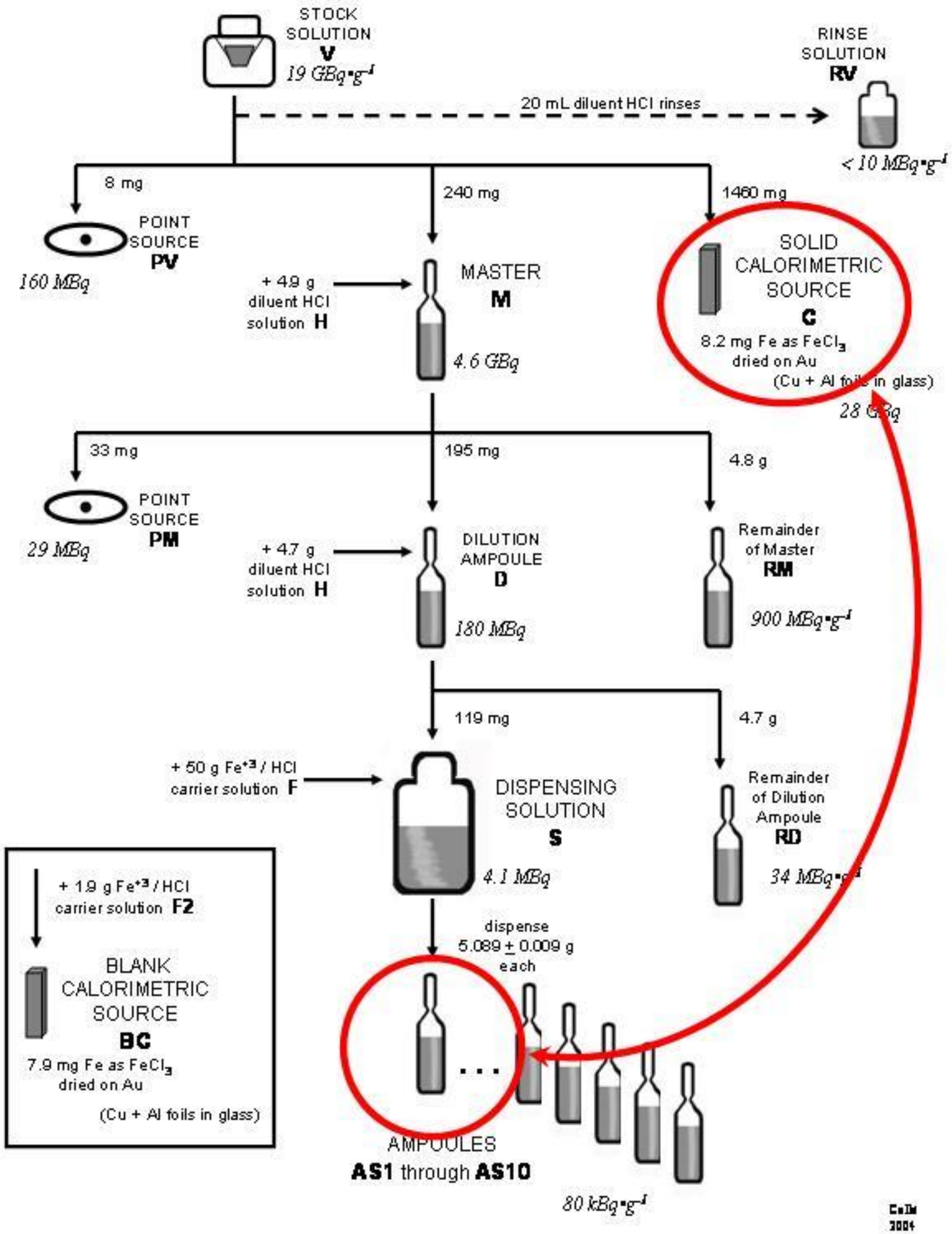
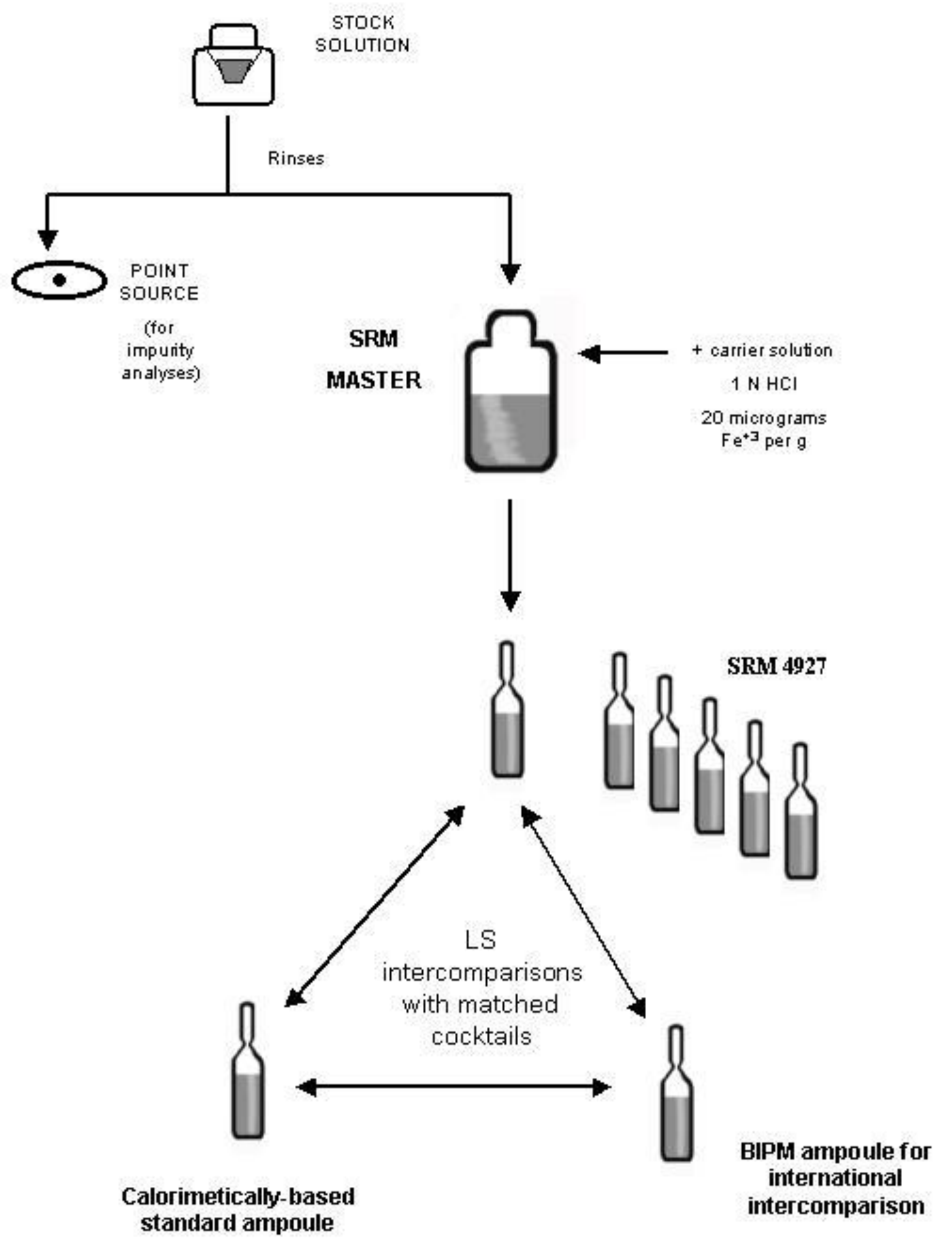


STOCK SOLUTION
V
19 GBq•g⁻¹
20 mL diluent HCl rinses
RINSE SOLUTION
RV
< 10 MBq•g⁻¹
8 mg
240 mg
1460 mg
POINT SOURCE
PV
160 MBq
+ 4.9 g diluent HCl solution H
MASTER
M
4.6 GBq
SOLID CALORIMETRIC SOURCE
C
8.2 mg Fe as FeCl3 dried on Au
(Cu + Al foils in glass)
28 GBq
33 mg
195 mg
4.8 g
POINT SOURCE
PM
29 MBq
+ 4.7 g diluent HCl solution H
DILUTION AMPOULE
D
180 MBq
Remainder of Master
RM
900 MBq•g⁻¹
119 mg
4.7 g
+ 50 g Fe+3 / HCl carrier solution F
DISPENSING SOLUTION
S
4.1 MBq
Remainder of Dilution Ampoule
RD
34 MBq•g⁻¹
+ 1.9 g Fe+3 / HCl carrier solution F2
BLANK CALORIMETRIC SOURCE
BC
7.9 mg Fe as FeCl3 dried on Au
(Cu + Al foils in glass)
dispense 5.089 ± 0.009 g each
AMPOULES
AS1 through AS10
80 kBq•g⁻¹



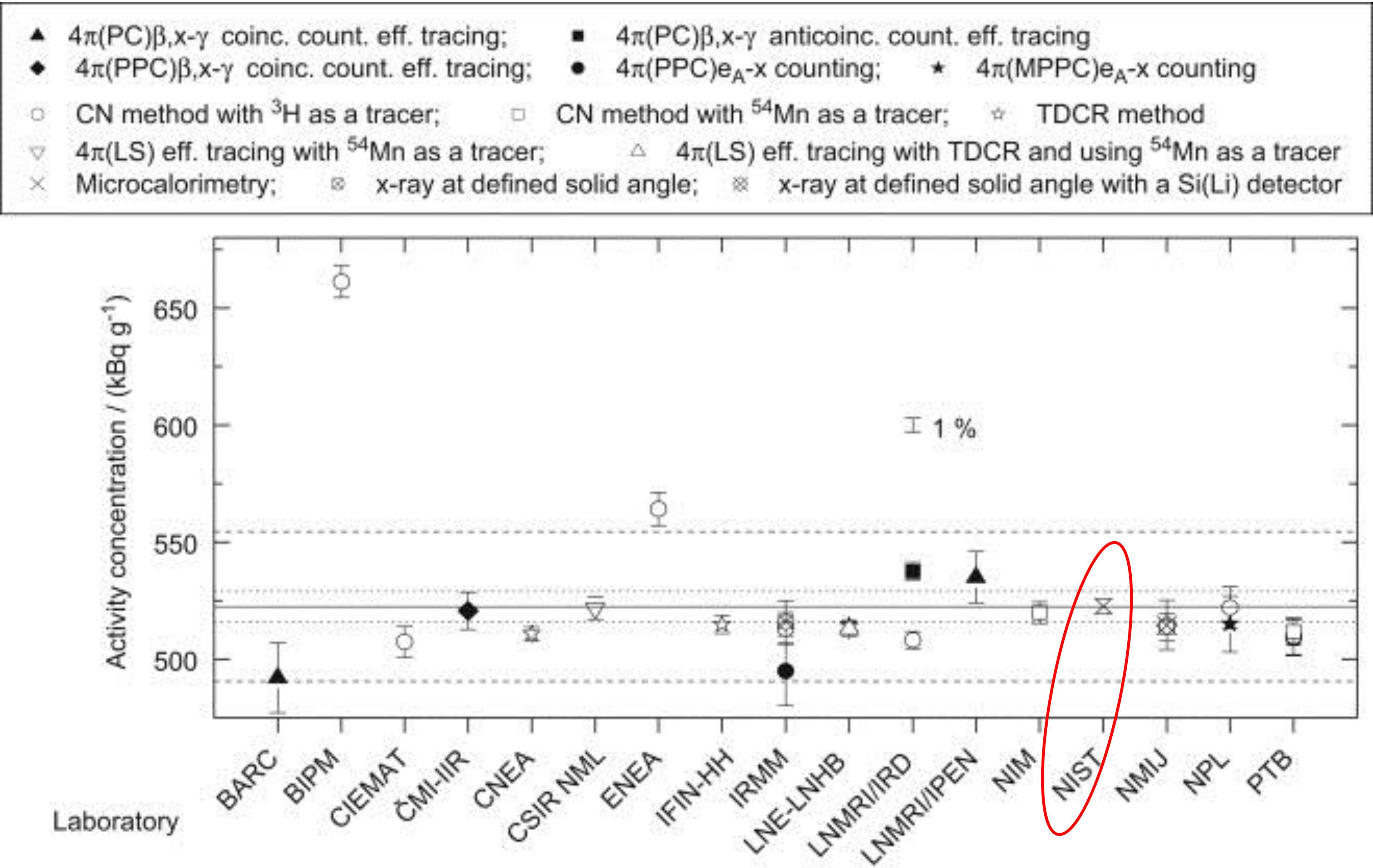
Calorimetry

13 independent determinations

LS intercomparisons

776 activity ratios; variables include:

- 3 counters
- 3 scintillators
- 44 matched cocktails
- 4 distinct aq. fraction (+Fe) compositions
- 2 NIST solution dilutions
- 97 days of aging



Results of the international comparison of activity concentration of a solution of 55Fe by a participant for each method. The arithmetic mean value (—), the sample standard deviation (- -) and the standard deviation of the mean (· · ·) are also drawn.

Summary of some recent NIST primary standardizations and comparison to confirmatory measurements.

^{210}Pb

Nuclide	Method	relative standard uncertainty	Confirmatory Measurement	Difference (%)
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Comparison of the NIST and NPL ^{210}Pb standards by five measurement methods.

method	**NPL / NIST ratio**	**relative standard uncertainty**
NPL and NIST certified values from primary standardizations	0.037484	1.5 %
$4\pi\gamma$(NaI)	0.037373	0.56 %
HPGe spectrometry.	0.036542	0.71 %
$4\pi\alpha\beta$(LS)	0.037249	0.17 %
^{210}Po assay ($2\pi\alpha$ spect.)	0.03736	0.75 %
Si(Li) low-energy spectrometry	0.0381	1.9 %

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^{90}Sr

^{241}Am

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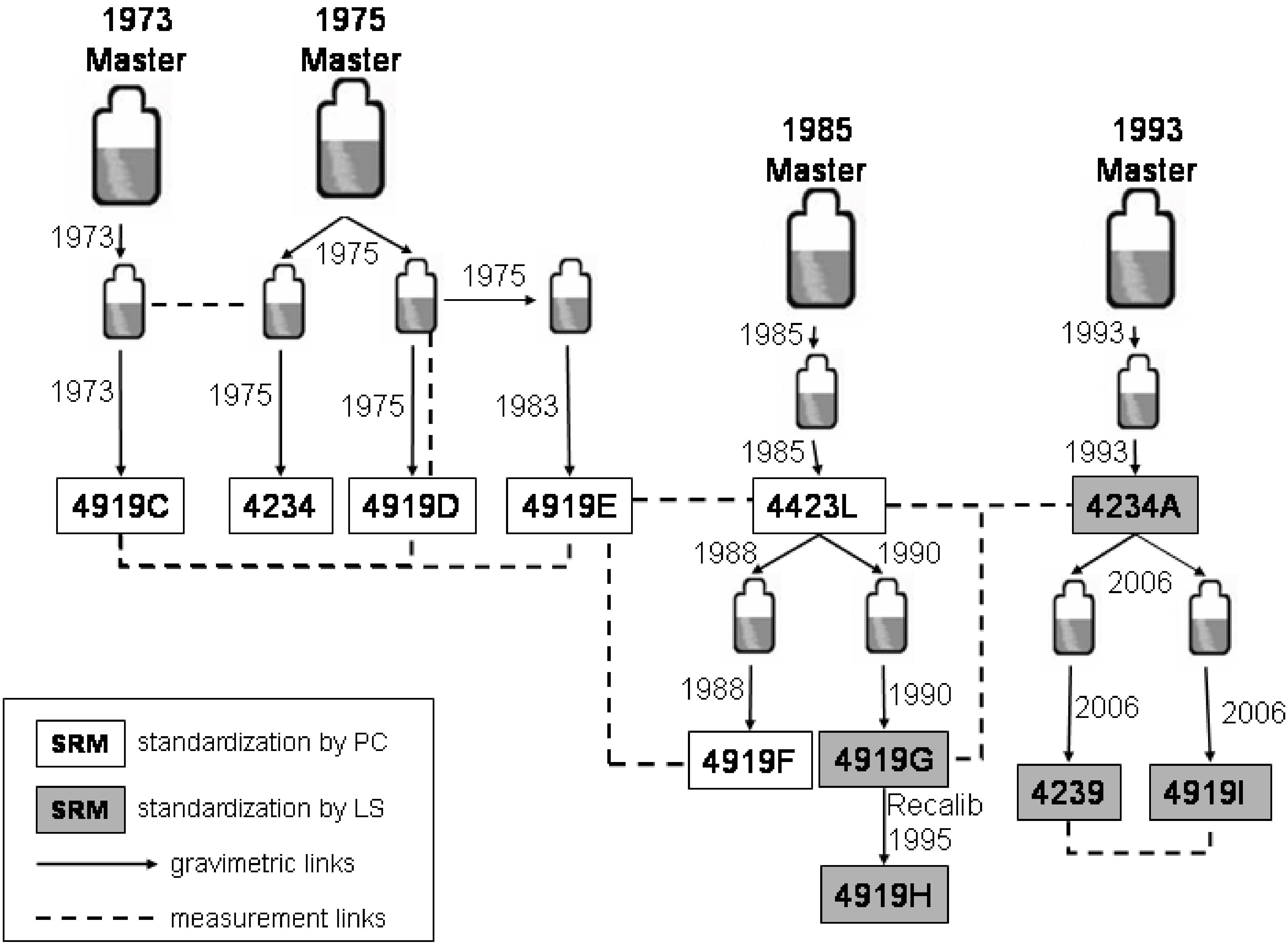
1973
Master
1975
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1985
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1993
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1985
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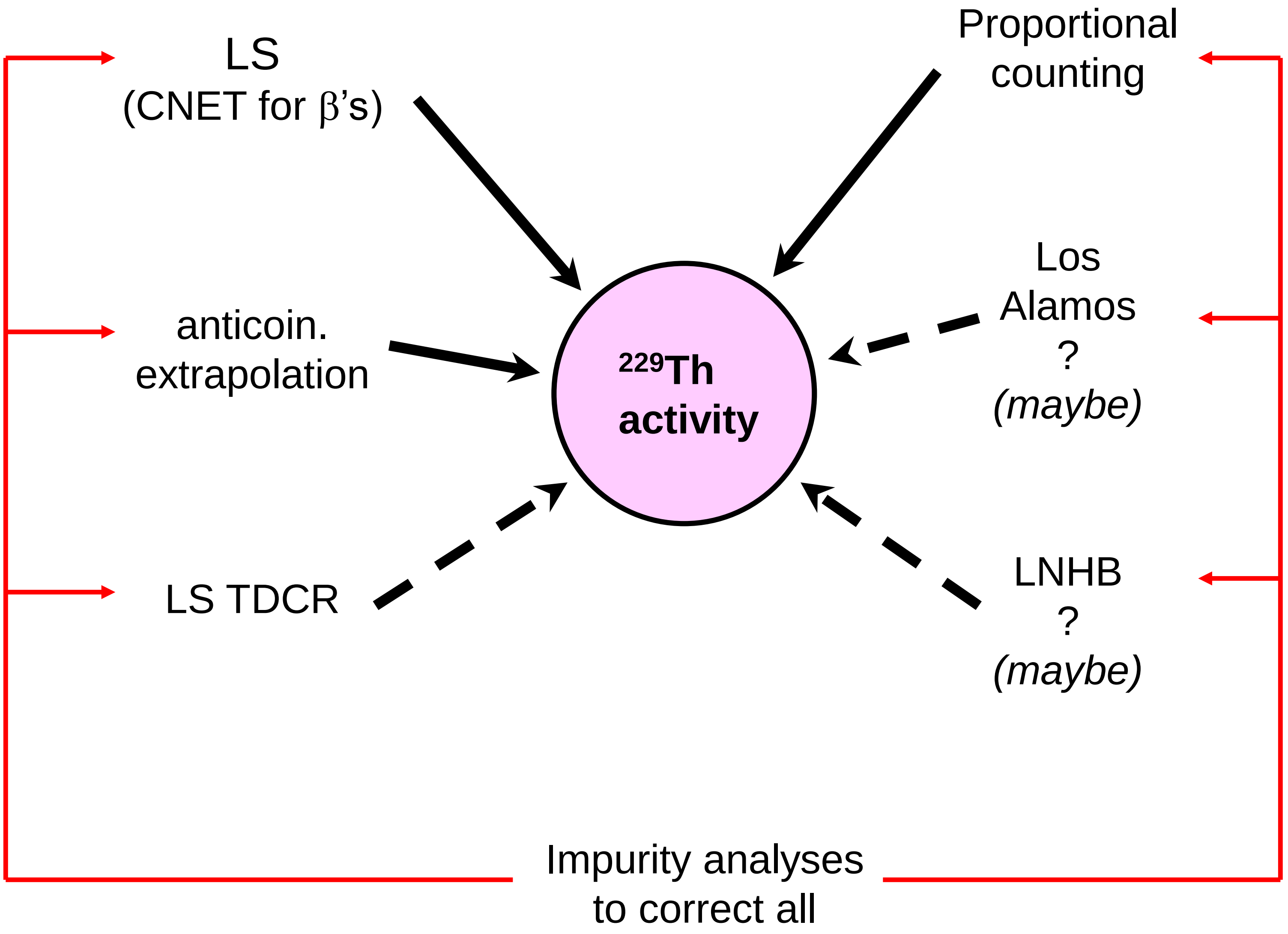
1993
1993
4919C
4234
4919D
4919E
4423L
4234A
1988
1990
2006
1988
1990
2006
2006
4919F
4919G
Recalib
1995
4919H
4239
4919I
SRM
standardization by PC
SRM
standardization by LS
gravimetric links
measurement links

Summary of some recent NIST primary standardizations and comparison to confirmatory measurements.

^{229}Th

Nuclide	Method	relative standard uncertainty	Confirmatory Measurement	Difference (%)
^{63}Ni	4π LS TDCR (NIST)	0.16 %	4πβ LS TDCR (LNHB) 4πβ LS CNET (NIST)	-0.31 -0.77
^{55}Fe (NIST)	4π calorimetry (linked by LS)	0.39 %	4π LS TDCR (Polatom) 4π LS TDCR (LNHB)	-0.87 -0.43
^{55}Fe (BIPM)	4π calorimetry (linked by LS)	0.39 %	weighted mean value of 15 NMI labs	-0.37
^{210}Pb	4παβ LS CNET	1.2 %	4παβ(LS)-γ(NaI) anticoin. counting ^{210}Po α spect. (102 a ^{209}Po tracer) ^{210}Po α spect. (128 a ^{209}Po tracer) HPGe photon spect.	+0.7 -3.0 -1.3 +4.7
^{241}Pu	4πβ LS CNET	1.9 %	LS (^{241}Am ingrowth) 4πβ LS TDCR (NIST) 4πβ LS TDCR (LNHB)	+1.2 -7.9 * -7.7 *
^{210}Pb	4παβ LS CNET	1.2 %	compare to NPL standard (5 methods) see Table2	-0.3
^{90}Sr	4πβ LS TDCR	0.51 %	4πβ LS CNET	+ 0.09
^{241}Am	4πα LS	0.22 %	4πα LS (independent) 4πα LS (independent)	-0.05 -0.01 -0.15
^{229}Th	4παβ(LS)-γ(NaI) anticoincidence counting	0.28 %	4παβ LS CNET 4παβ LS TDCR 2π α proportional counting HPGe photon spectrometry	-0.09 -1.7 -0.09 +2.1

* Values are discrepant, and not considered to have confirmed


LS
(CNET for β's)
Proportional counting
anticoin. extrapolation
^{229}Th activity
Los Alamos
?
(maybe)
LS TDCR
LNHB
?
(maybe)
Impurity analyses to correct all

Features of our standardization work

Available "<u>transfer standards</u>" (SRMs, etc.) are based on identified "needs"

Standardized by primary method

Usually at least one confirmatory determination

Establish links to previous calibrations, if possible

Develop & maintain secondary calibrations

Uncertainties (k = 2) typically < 1 %

u (k = 1) few tenths of %

Comparisons with other metrology labs

N.B. → *"**National Standards**" are <u>not artifacts</u> but are our (NIST's) ability to perform primary standardizations with our instruments, procedures, people, etc...*