Elements of a “Good” Radionuclidic Standardization Program

An Overview

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Given title of "Certification of Reference Materials"

EASY TALK FOR ME / pristine, single nuclide SRMs

- i decide what std. to do
- ii make it
- iii standardize it
- iv write certificate based on data

Instead I'll talk about

Elements of a "Good" Radionuclidic Standardization Program

I'll give talk backwards

.... summary & conclusions first

then show you examples

Characteristic Elements

1. **C**hoice of nuclides / standards based on identified **<u>user needs</u>**

2. **S**tandardization based on at least one **<u>primary method</u>**

3. **V**alidity of #2 supported & confirmed by one or more **<u>independent confirmatory methods</u>**

4. **S**tandardizations typically utilize **<u>many trials</u>**, with widely varying experimental conditions, minimizing type-B uncertainty assessments

5. **A**ny new standardization **<u>linked back</u>** to all previous ones (when possible) through stored solutions or calibration factors for 2ndary instruments

6. **<u>Disseminated standards</u>** from primary methods used as SRM transfer standards and/or employed as sources for quality assurance, & proficiency testing programs

7. Primary standardization **<u>uncertainties</u>** (k = 2) are typically < 1 % (few tenths at k = 1)

8. **<u>Comparisons</u>** with others metrology labs to demonstrate & ensure international consistency

1896	←	radioactivity
1898	←	polonium / radium
1901	←	NBS (NIST)
1914	←	1^{st} 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

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)

Radioactivity Group Roles & Major Program Elements

i.e., primary standardizations

BASIC RADIONUCLIDIC METROLOGY

Standards (SRMs) & calibrations

proficiency testing (MQA) programs

NUCLEAR MEDICINE / RADIOPHARMACEUTICAL APPLICATIONS

LOW-LEVEL ENVIRONMENTAL / BIOLOGICAL MEASUREMENTS

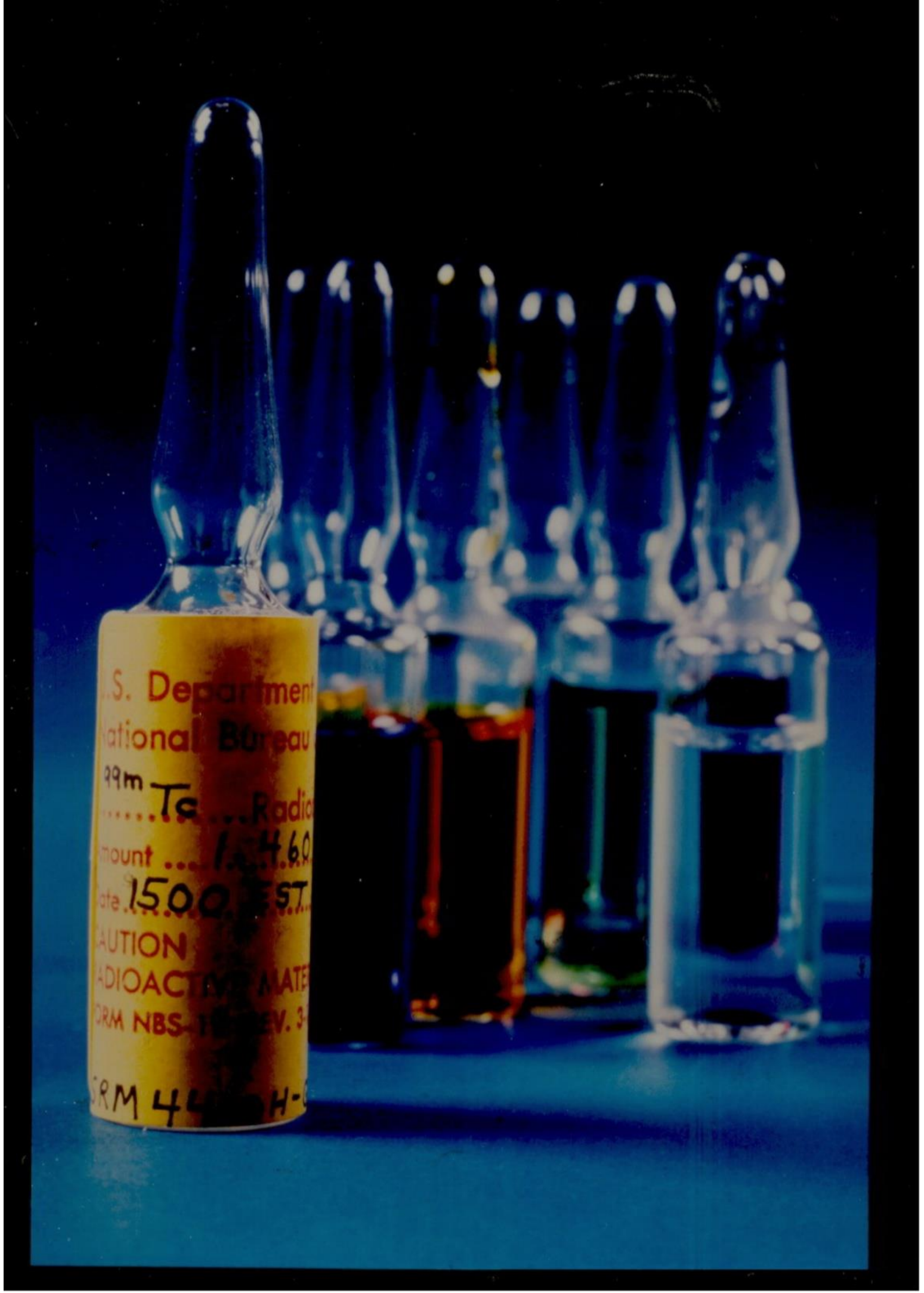
HOMELAND SECURITY

Our principal product...

solution standards of
radionuclides

60⁺ nuclides

calibrations in many other geometries and states too



Natural Matrix SRMs

for Environmental Radioactivity Measurement

- Rocky Flat Soil I (1981)
- River Sediment (1981)
- Peruvian Soil (1982)
- Human Lung (1982)
- Human Liver (1982)
- Lake Sediment (1986)
- Ocean Sediment (1997)
- Bone Ash (2000)
- Seaweed (2005)
- Rocky Flats Soil II (2007)
- Shell Fish (underway)
- Peruvian Soil II (underway)

Radionuclides standardized at NIST

130^{+} nuclides

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	

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

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

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

many measurement systems used at NIST

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

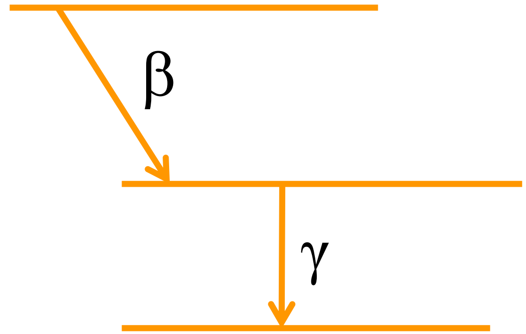
•use strictly controlled geometric constructions
incorporating a large variety of detectors with known detection efficiencies

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)

β−γ coincidence

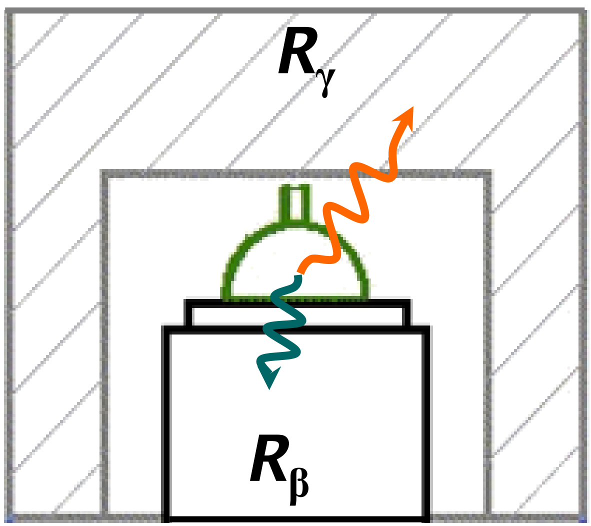


$$\boxed{R_\gamma = \varepsilon_\gamma A}$$

$$\boxed{R_\beta = \varepsilon_\beta A}$$

$$\boxed{R_c = \varepsilon_\beta \, \varepsilon_\gamma \, A}$$

A decays per second

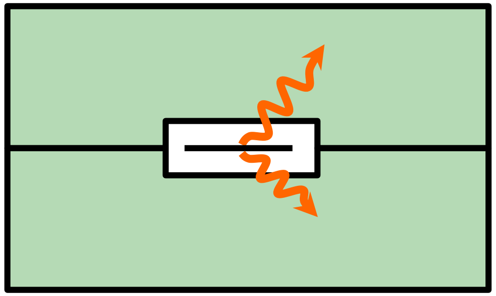


$$\frac{R_\beta R_\gamma}{R_c} = \frac{\varepsilon_b A \, \varepsilon_\gamma A}{\varepsilon_b \varepsilon_\gamma A}$$

$$= A$$

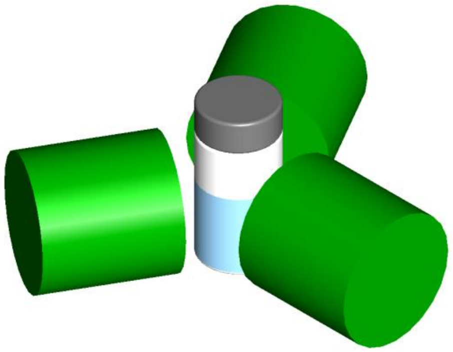
β-*γ* *β*-*x* *e*-*x* α-*γ*

γ-γ γ-X



NaI sandwich detector
coincidence & near total absorption

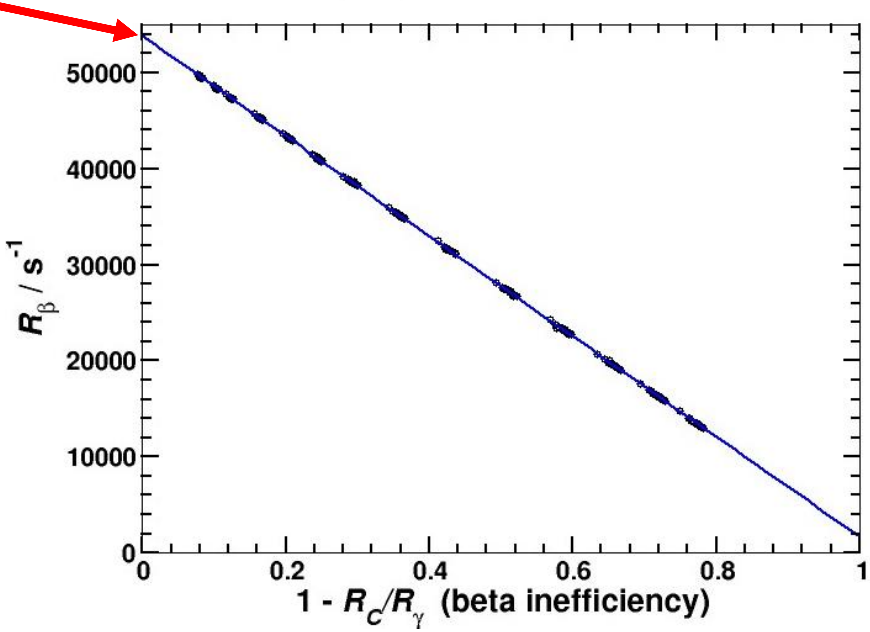
β e^-



TDCR
3-phototubes

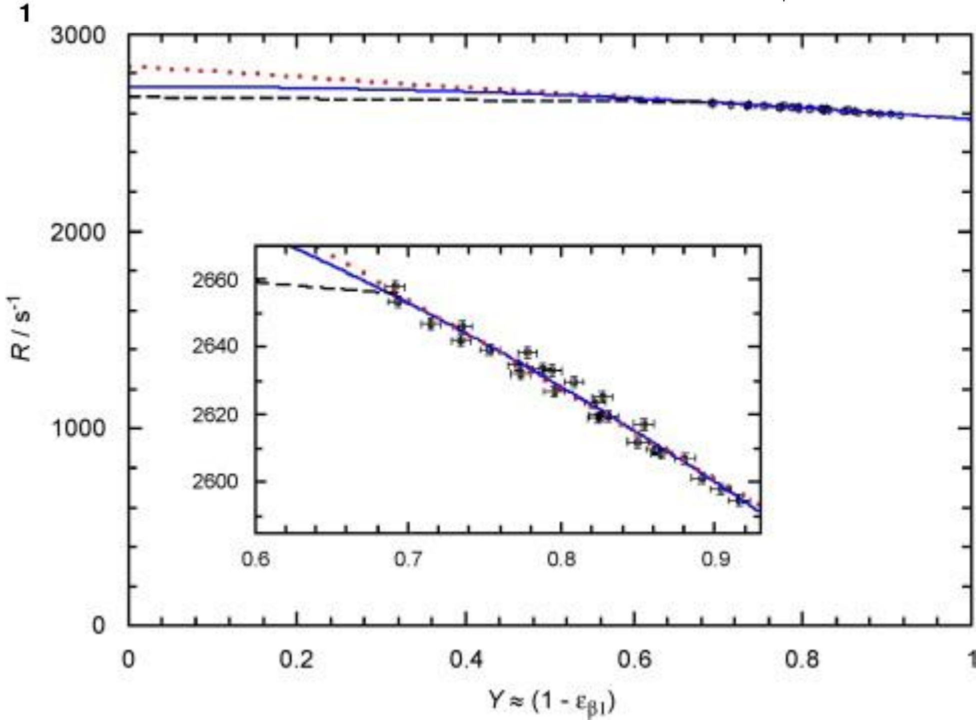
A

^{60}Co β–γ coincidence



some are easy

some are not (^{210}Pb)



$$R_\beta = A\frac{R_c}{R_\gamma}$$

$$R_\beta = A\left(1 - k\left(1 - \frac{R_c}{R_\gamma}\right)\right)$$

Primary method ?

Two exceptions ....

- Simultaneously measure 2 nuclides in coin. system

  trace pure β with β-γ case

  (e.g., ^{99}Tc with ^{60}Co)

- LS based ^{3}H-standard efficiency tracing (CNET)

  CIEMAT /NIST method

  same model as LS

Now some examples

Standardizations from period 2005-2009

^{63}Ni

^{55}Fe

^{210}Pb

^{209}Po

^{241}Pu

^{90}Sr

^{241}Am

^{229}Th

^{239}Pu

My many collaborators on these will be acknowledged & thanked later ...

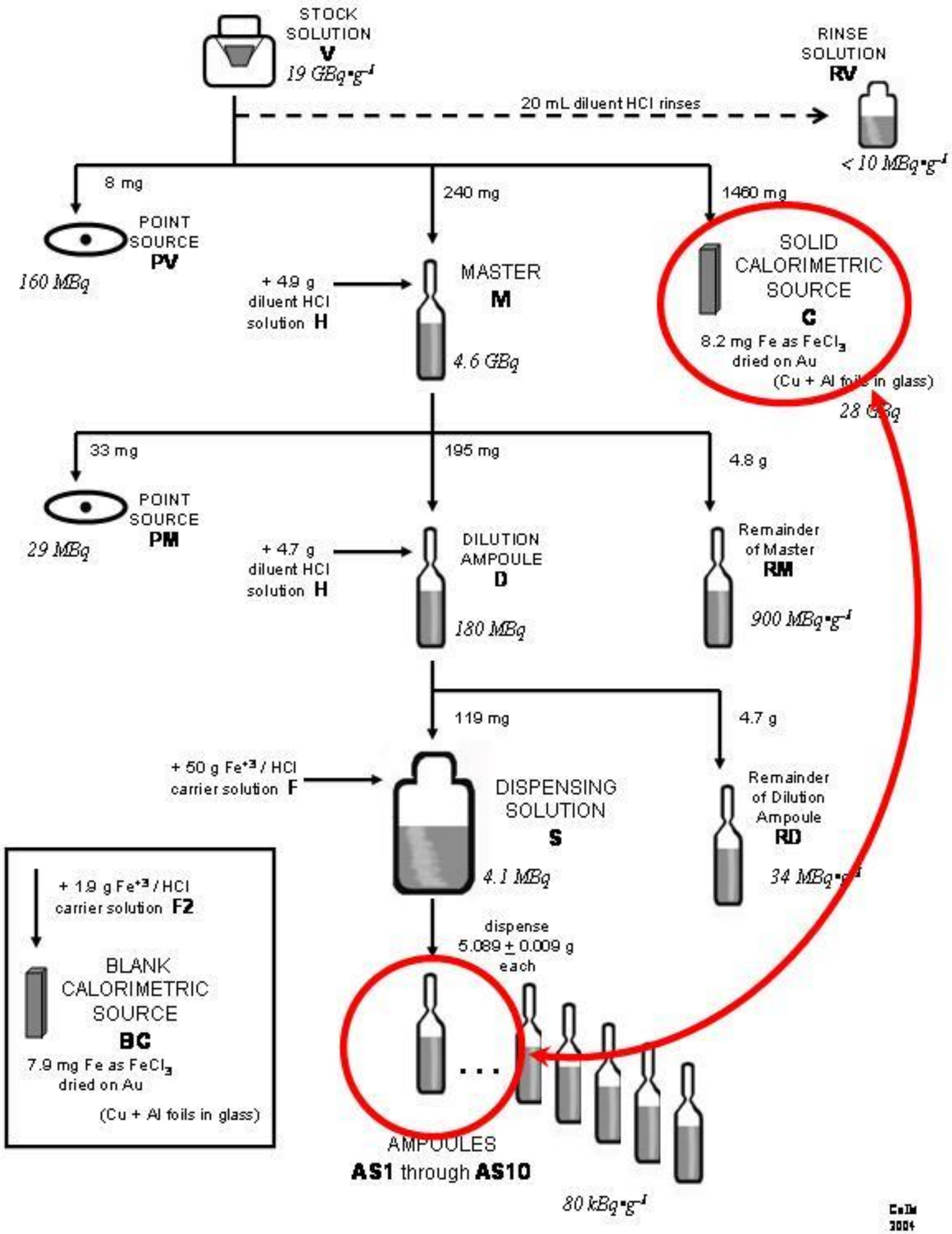
^{55}Fe

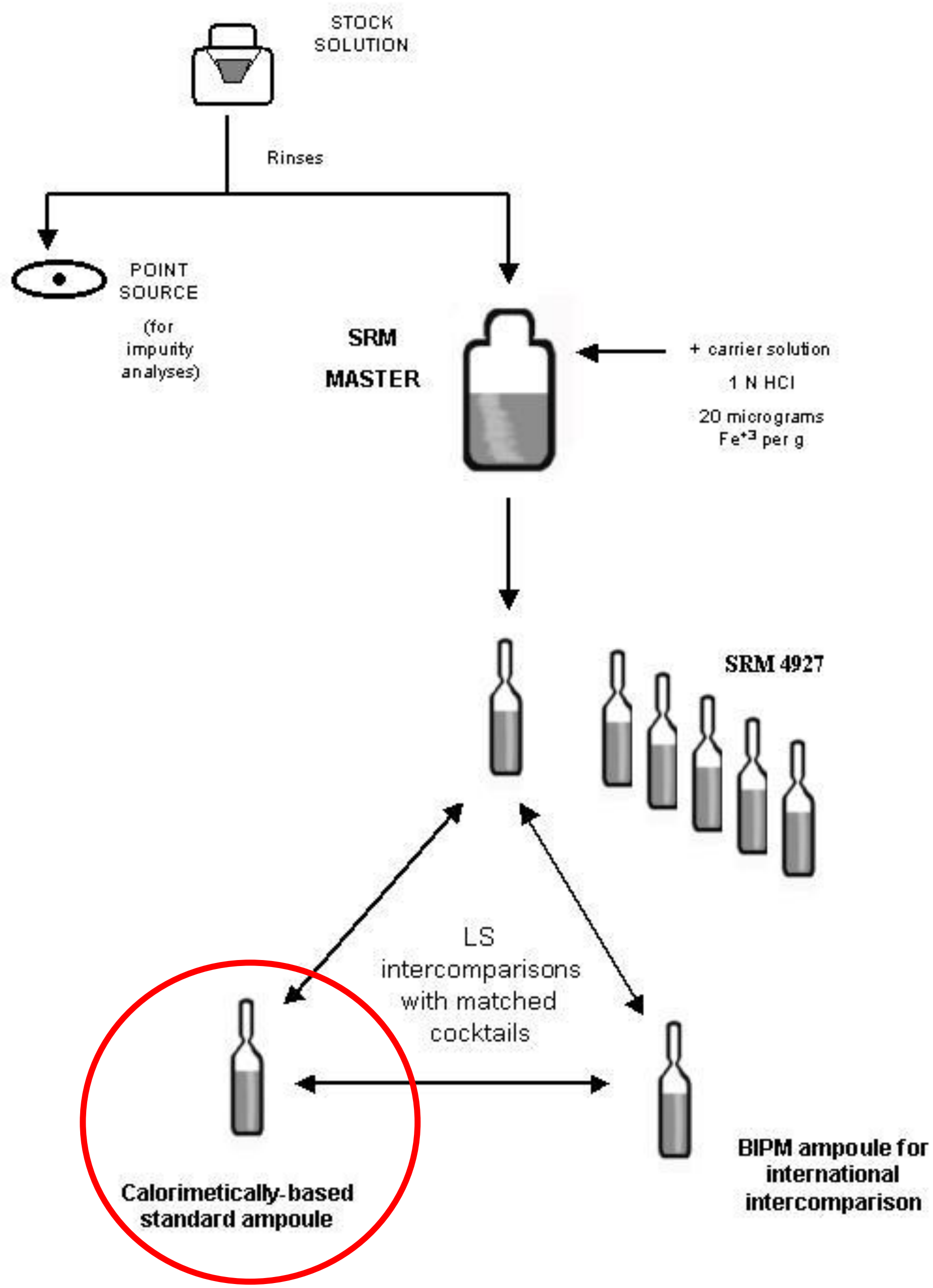
Pure EC – low *Z* – low *E* (really hard)

primary standardization by microcalorimetry

linked to SRM

international intercomparison


STOCK SOLUTION
V
19 GBq•g-1
RINSE SOLUTION
RV
20 mL diluent HCl rinses
< 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

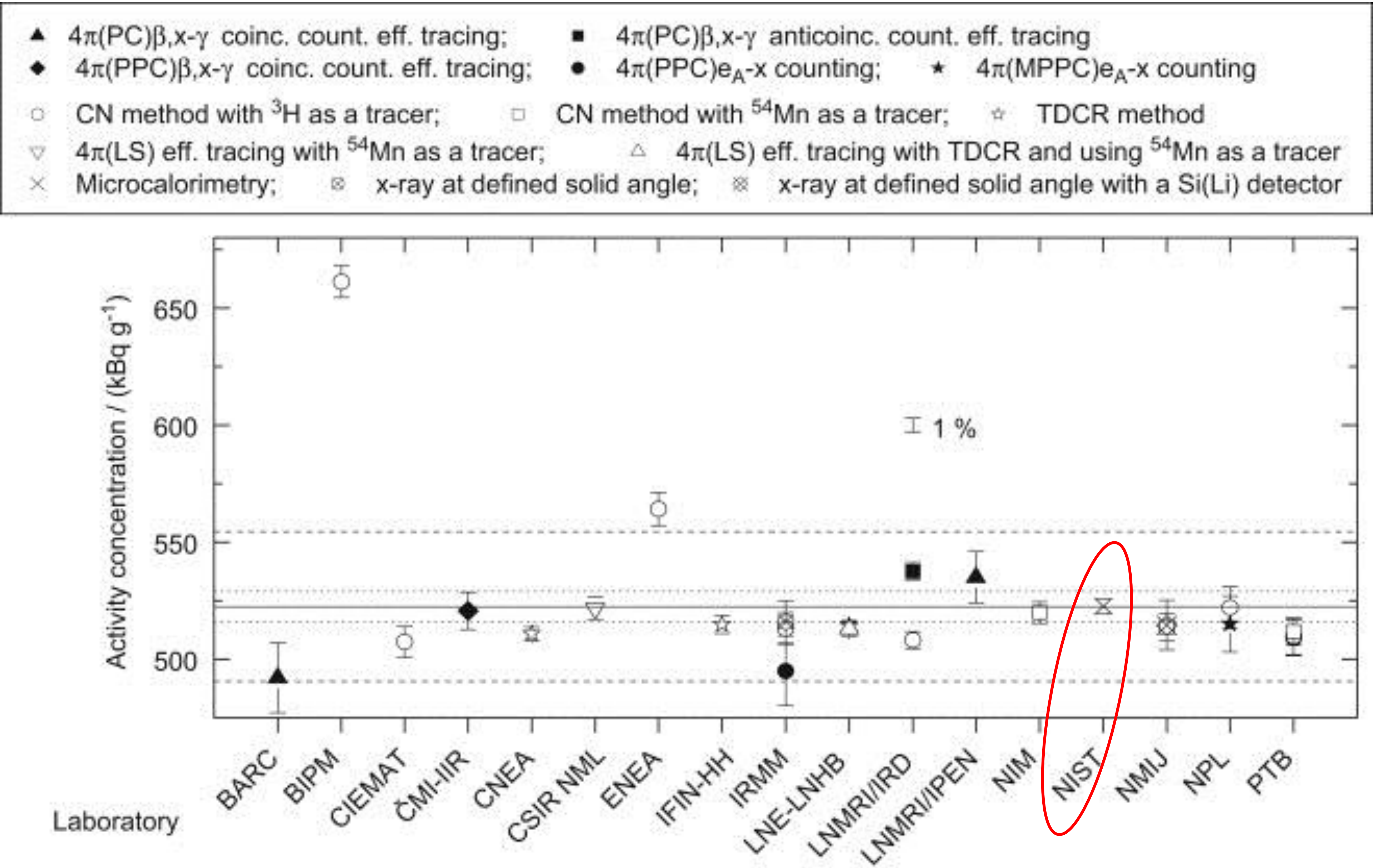
LS intercomparisons

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

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 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.

^{55}Fe

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 %	4παβ(LS)-γ(NaI) ACC 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

^{63}Ni

medium energy pure β

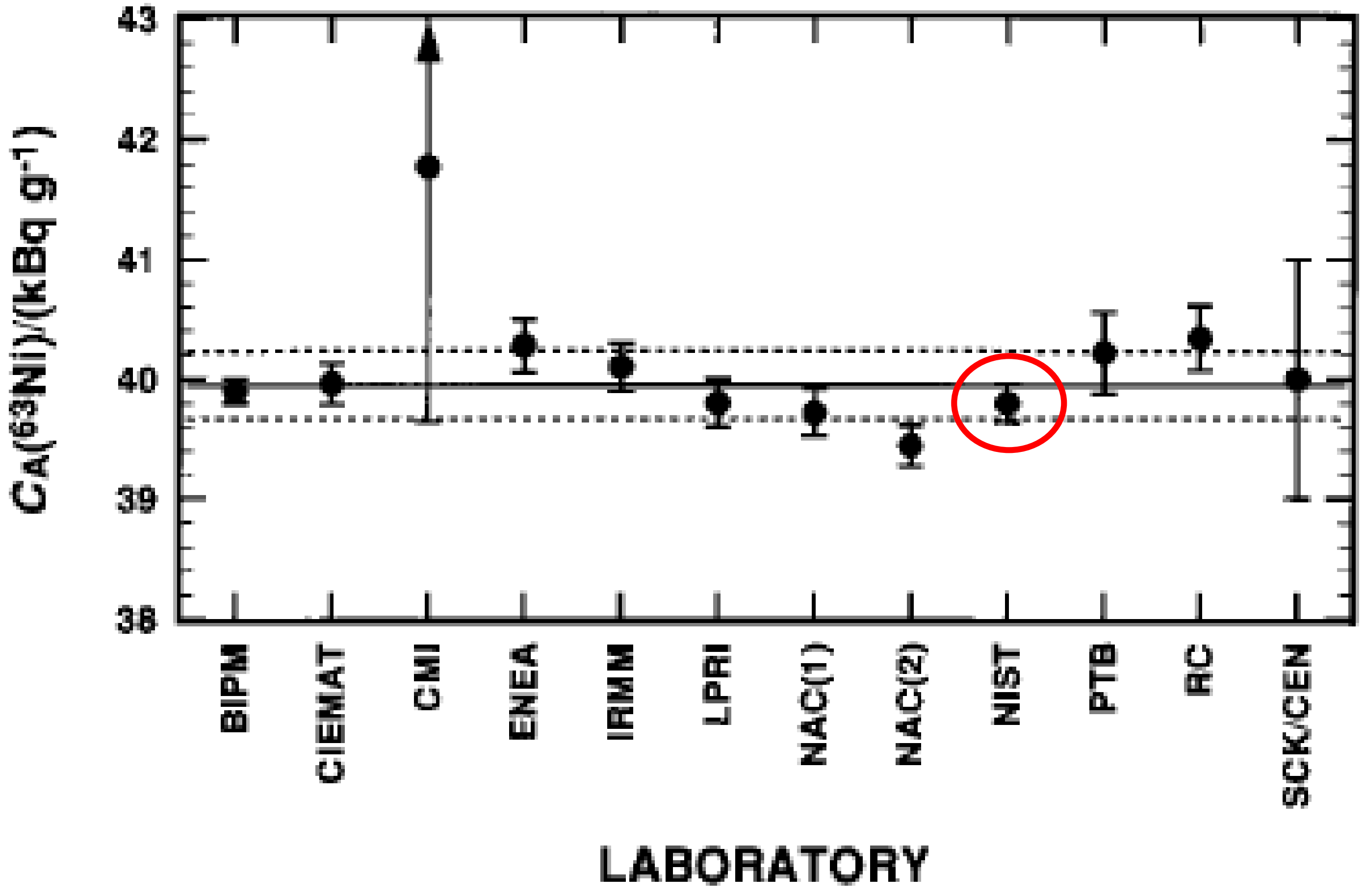
often used for international comparisons

Test LS methods

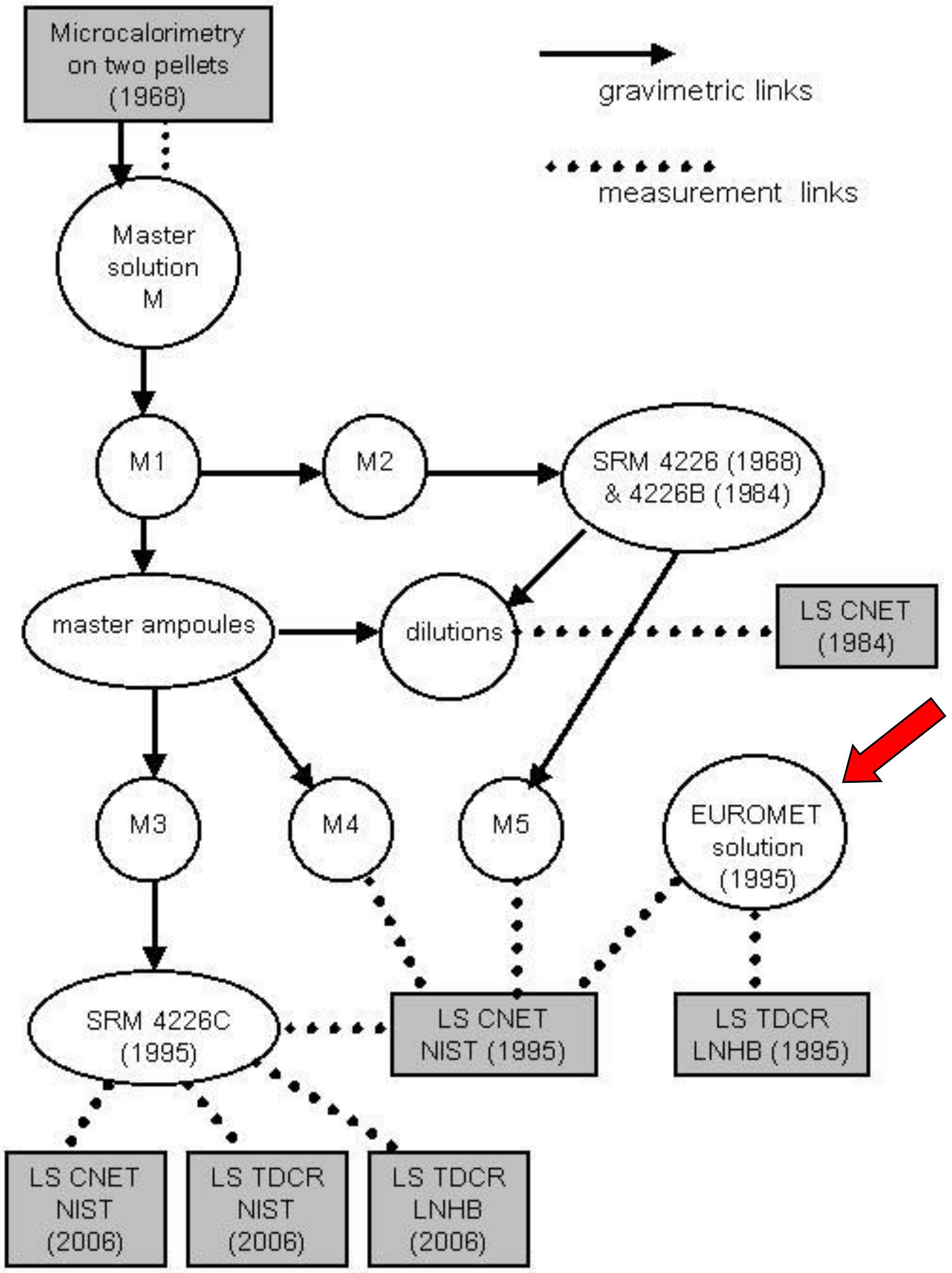
$T_{1/2}$ by decay 40+ years

linked to all ^{63}Ni SRM

^{63}Ni international intercomparison 1995



38 years of ^{63}Ni results



most recent

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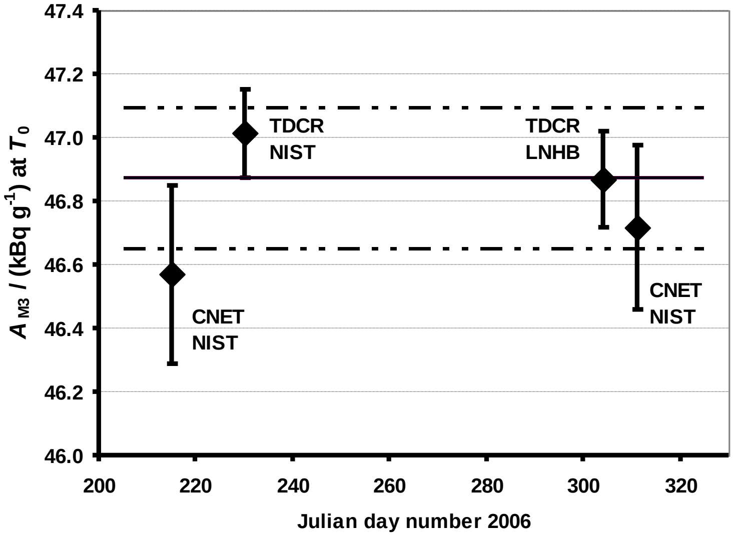
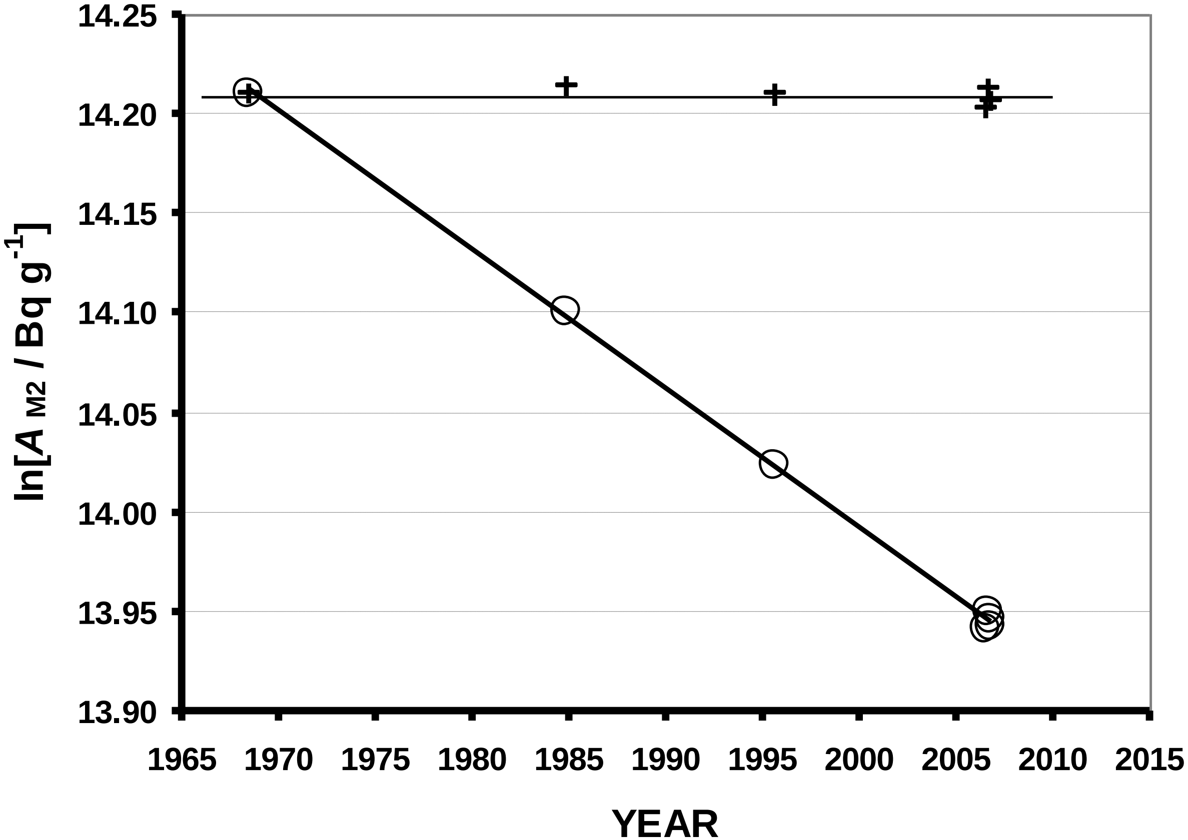


with 2006 result



Precise value of 100 a half-life **by decay** over 38 years

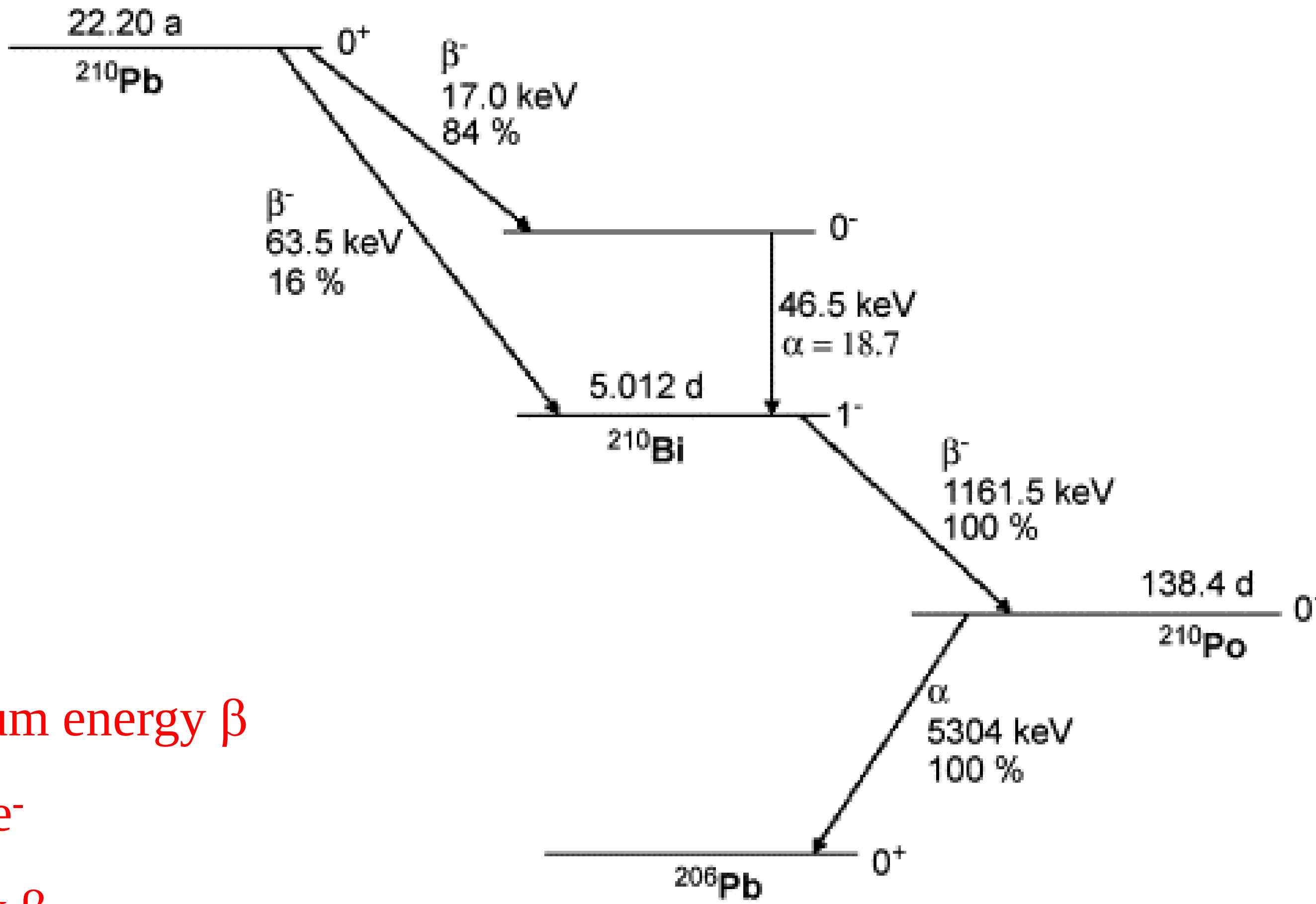
^{210}Pb

Difficult case

rarely done by metrology labs

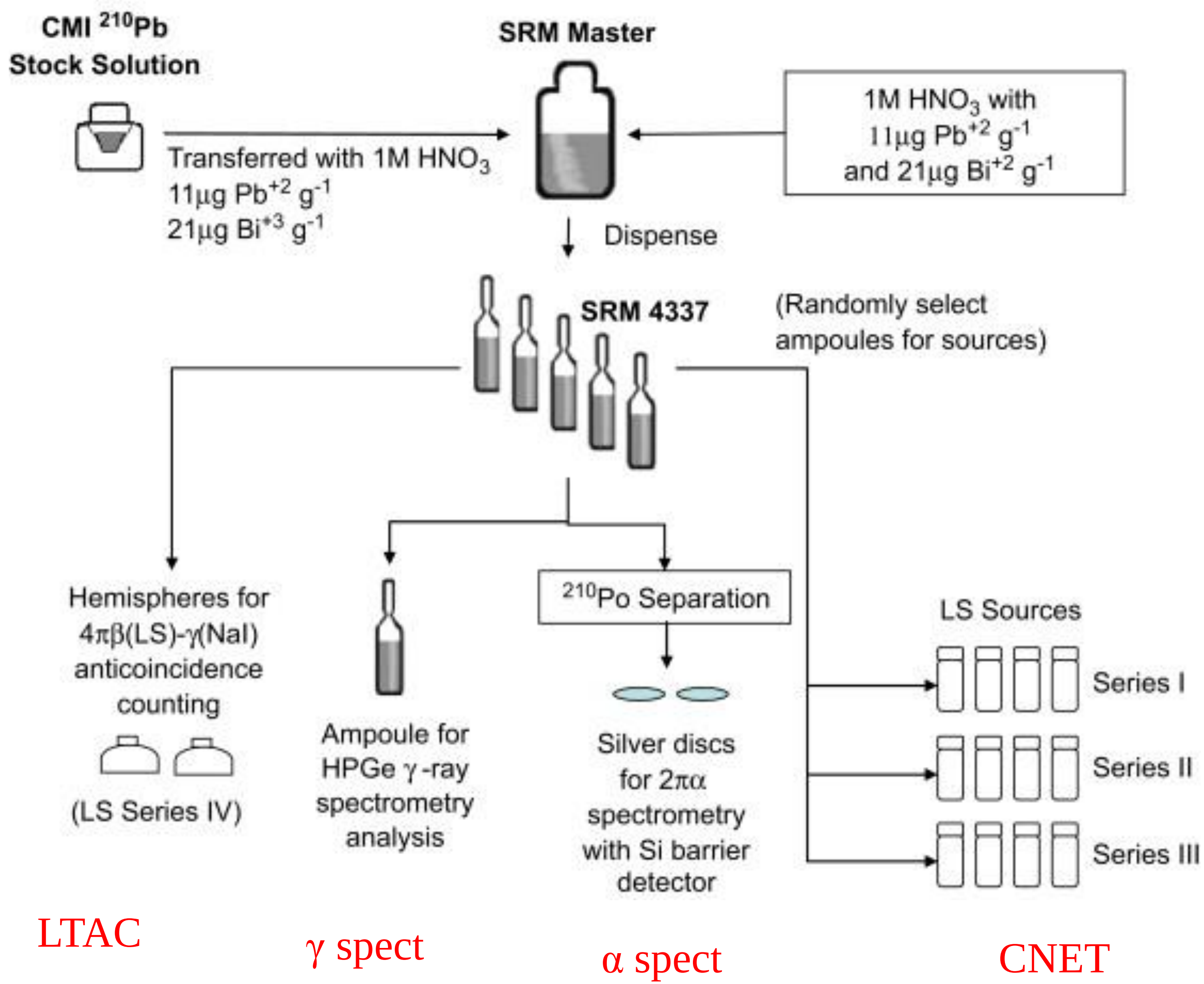
different methods used

Comparisons with NPL standard (via PTB too)



Need to detect

- low – medium energy β
- conversion e^-
- High energy β
- α


CMI ^{210}Pb Stock Solution
Transferred with 1M HNO_3
11µg Pb^{+2} g^{-1}
21µg Bi^{+3} g^{-1}
SRM Master
1M HNO_3 with
11µg Pb^{+2} g^{-1}
and 21µg Bi^{+2} g^{-1}
Dispense
SRM 4337
(Randomly select ampoules for sources)
Hemispheres for 4πβ(LS)-γ(NaI) anticoincidence counting
(LS Series IV)
Ampoule for HPGe γ-ray spectrometry analysis
^{210}Po Separation
Silver discs for 2πα spectrometry with Si barrier detector
LS Sources
Series I
Series II
Series III
LTAC
γ spect
α spect
CNET

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^{210}Pb

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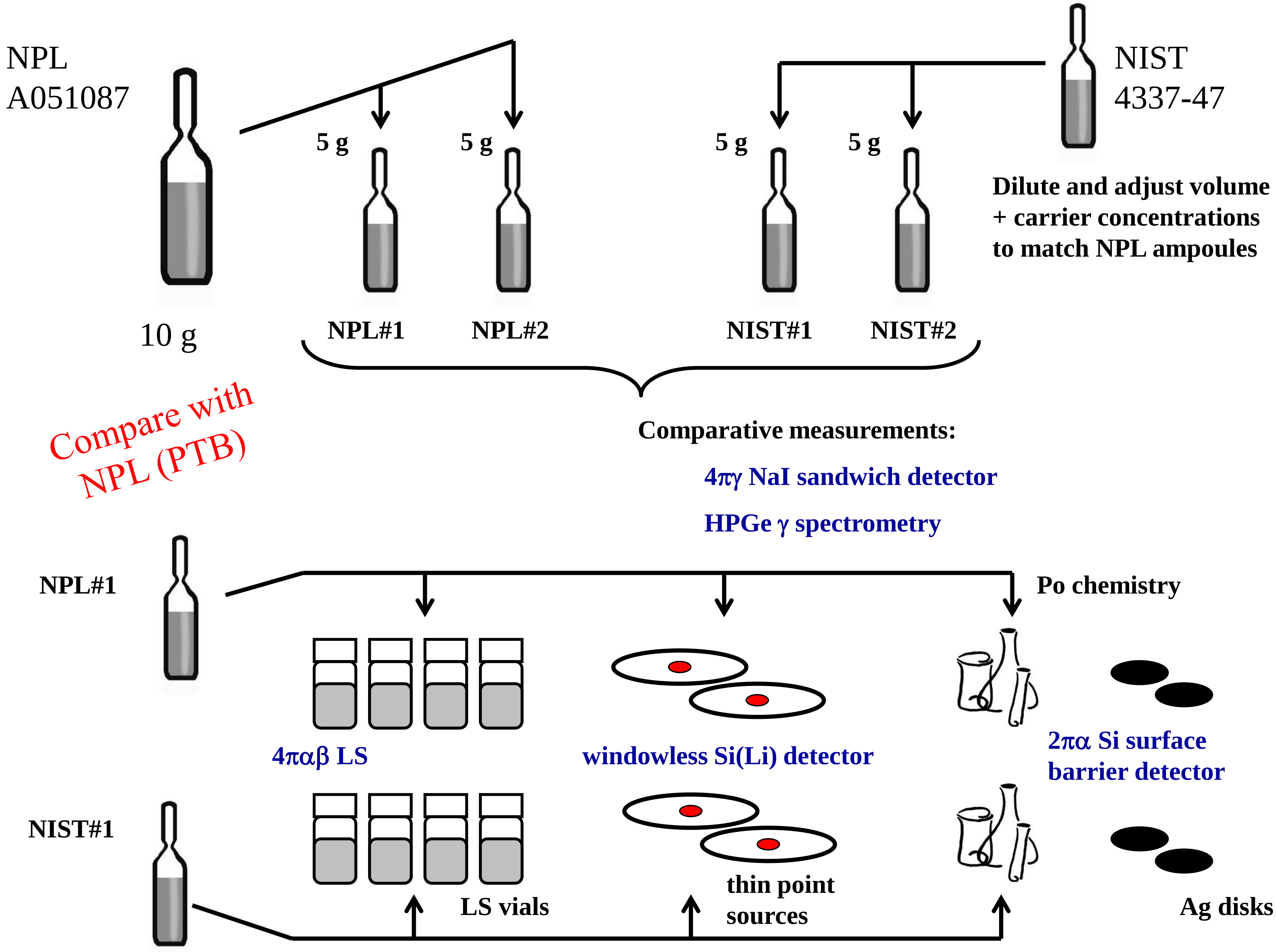
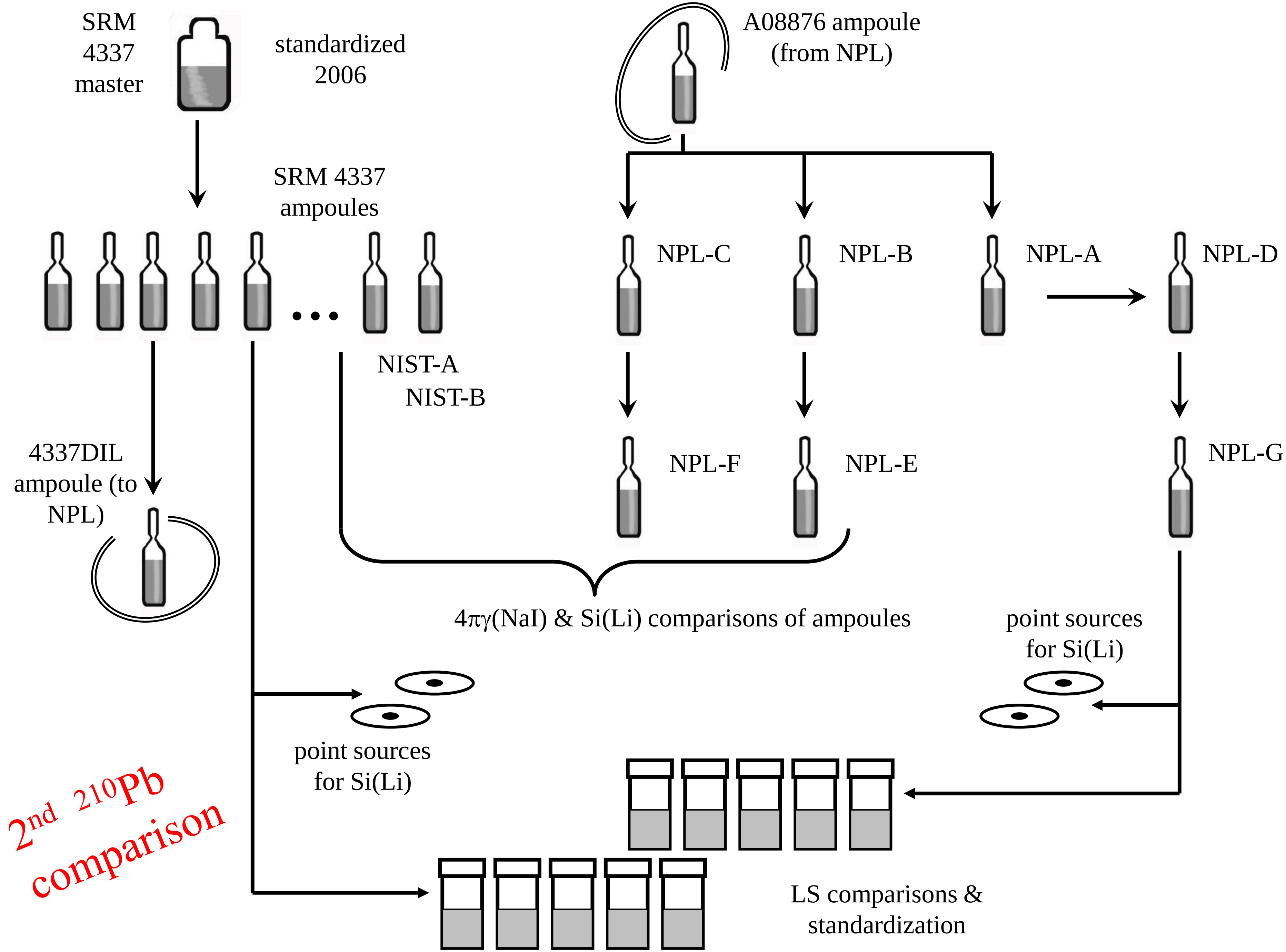

NPL
A051087
10 g
5 g
5 g
NPL#1
NPL#2
5 g
5 g
NIST#1
NIST#2
NIST
4337-47
Dilute and adjust volume
+ carrier concentrations
to match NPL ampoules
Compare with
NPL (PTB)
Comparative measurements:
4πγ NaI sandwich detector
HPGe γ spectrometry
NPL#1
Po chemistry
4παβ LS
windowless Si(Li) detector
2πα Si surface
barrier detector
NIST#1
LS vials
thin point
sources
Ag disks

Comparison of the NIST and NPL ^{210}Pb standards by five measurement methods.

method	NPL / NIST ratio	relative standard uncertainty	difference
NPL and NIST certified values from primary standardizations	0.037484	1.5 %	--
$4\pi\gamma$(NaI)	0.037373	0.56 %	- 0.30 %
HPGe spectrometry.	0.036542	0.71 %	- 2.6 %
$4\pi\alpha\beta$(LS)	0.037249	0.17 %	- 0.63 %
^{210}Po assay ($2\pi\alpha$ spect.)	0.03736	0.75 %	- 0.33 %
Si(Li) low-energy spectrometry	0.0381	1.9 %	+ 1.6 %


SRM 4337 master
standardized 2006
A08876 ampoule (from NPL)
SRM 4337 ampoules
NPL-C
NPL-B
NPL-A
NPL-D
NIST-A
NIST-B
4337DIL ampoule (to NPL)
NPL-F
NPL-E
NPL-G
4πγ(NaI) & Si(Li) comparisons of ampoules
point sources for Si(Li)
point sources for Si(Li)
2nd 210Pb comparison
LS comparisons & standardization

Comparison of the NIST and NPL ^{210}Pb standards by five measurement methods.

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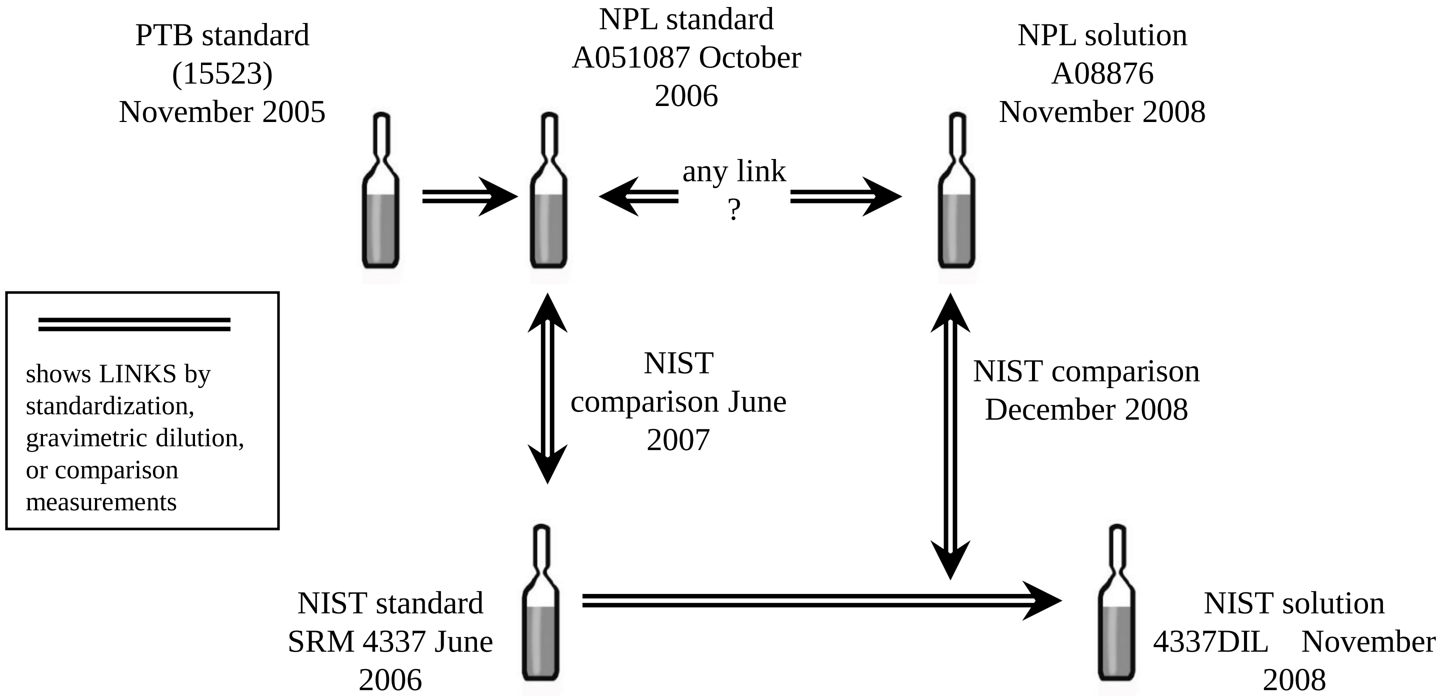
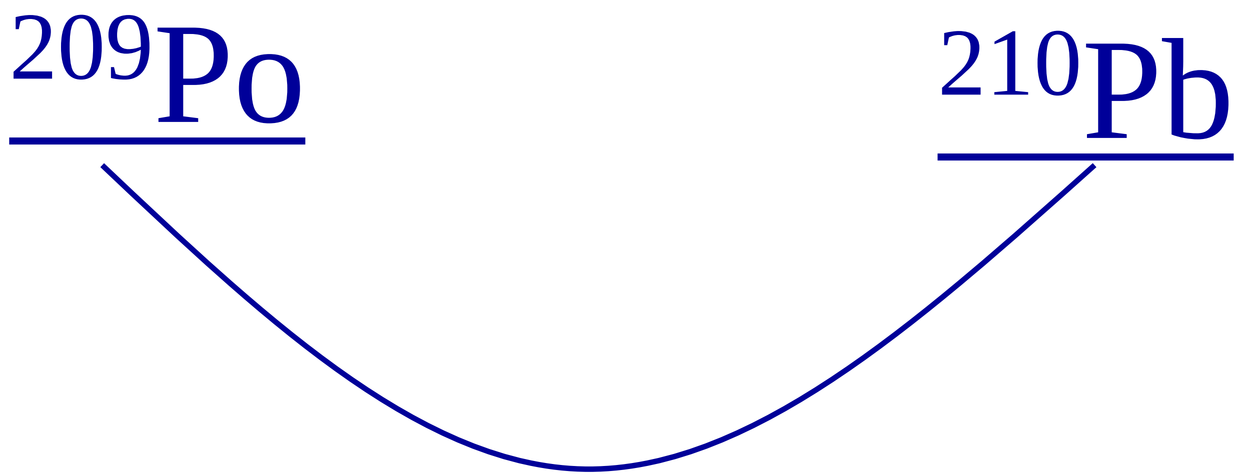

PTB standard
(15523)
November 2005
NPL standard
A051087 October
2006
NPL solution
A08876
November 2008
any link
?
shows LINKS by
standardization,
gravimetric dilution,
or comparison
measurements
NIST
comparison June
2007
NIST comparison
December 2008
NIST standard
SRM 4337 June
2006
NIST solution
4337DIL November
2008

^{209}Po



LINKED

In applications

– use of ^{210}Po tracer for ^{210}Pb assays

In our standardization measurements

^{209}Po half-life in error by 25 % !!

Result supported by work on ^{210}Pb – previous story

15 march 1994

R_{α} = (85.42 ± 0.18) $s^{-1}g^{-1}$

15 November 2005

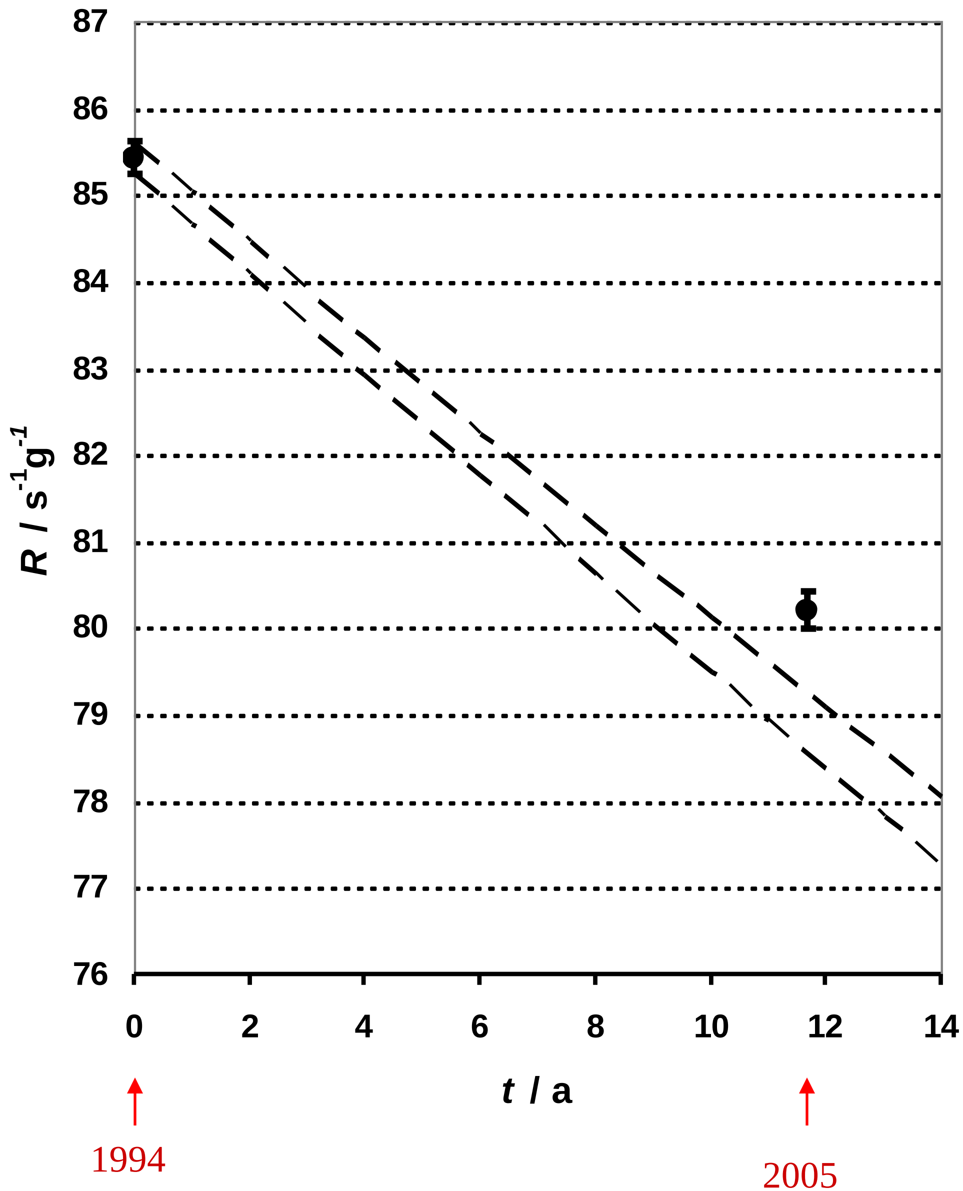
R_{α} = (80.20 ± 0.22) s-1g-1

2 point fit gives

$T_{1/2}$ = 128 a

U = 5.5 % (7 a)

<u>Not</u> considered a new determination



Collé, et al., *Appl. Radiat. Isot.* **65**, 728 (2007)

^{241}Pu

very low energy β (like ^3H)

thought would be easy by LS methods

Wasn't !

Extant problem with method discrepancy

Work planned

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

^{241}Pu

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 %	4παβ(LS)-γ(NaI) ACC 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

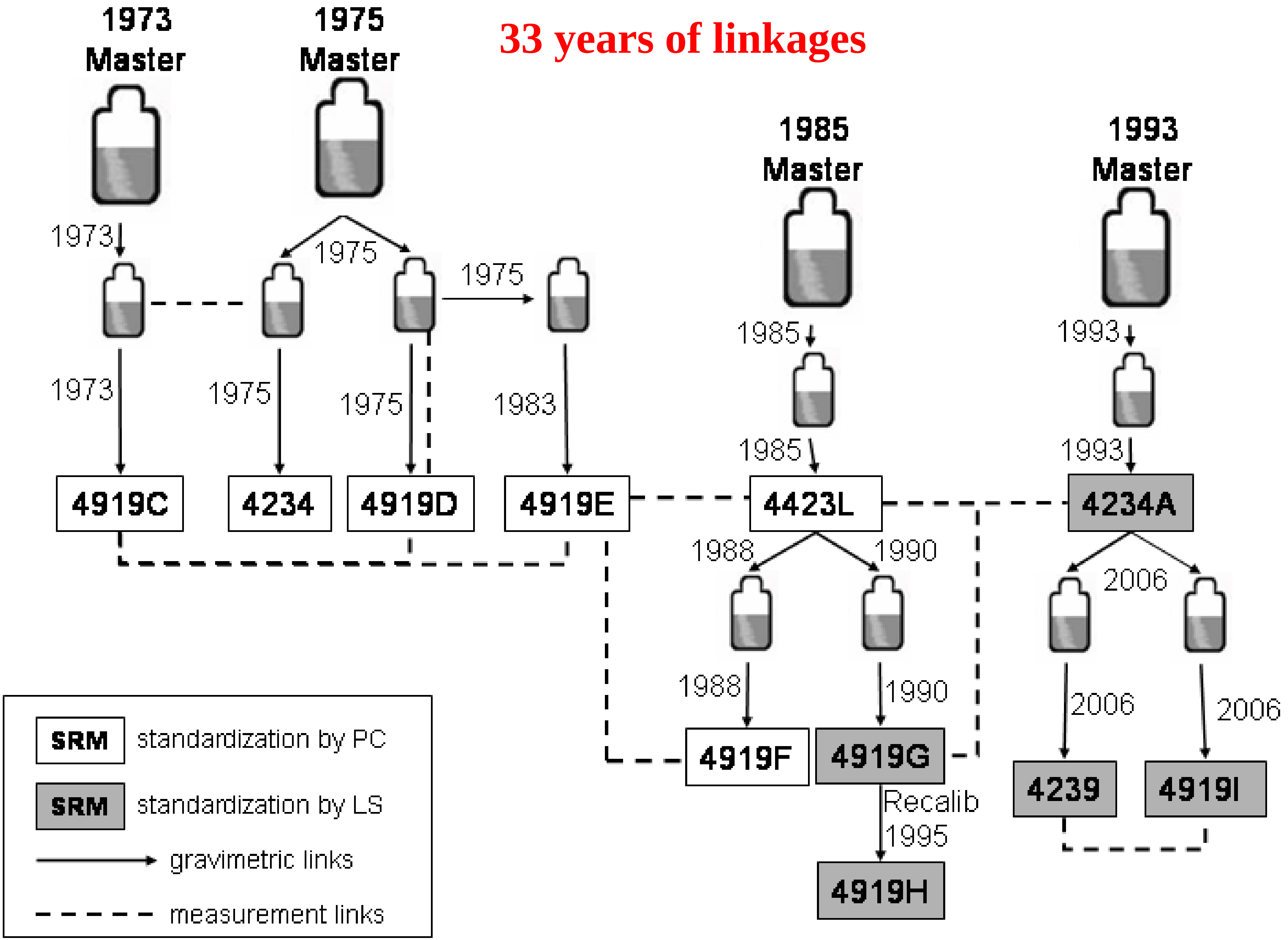
very recent LTAC
agrees with
^{241}Am ingrowth
to ≅ 0.2 %

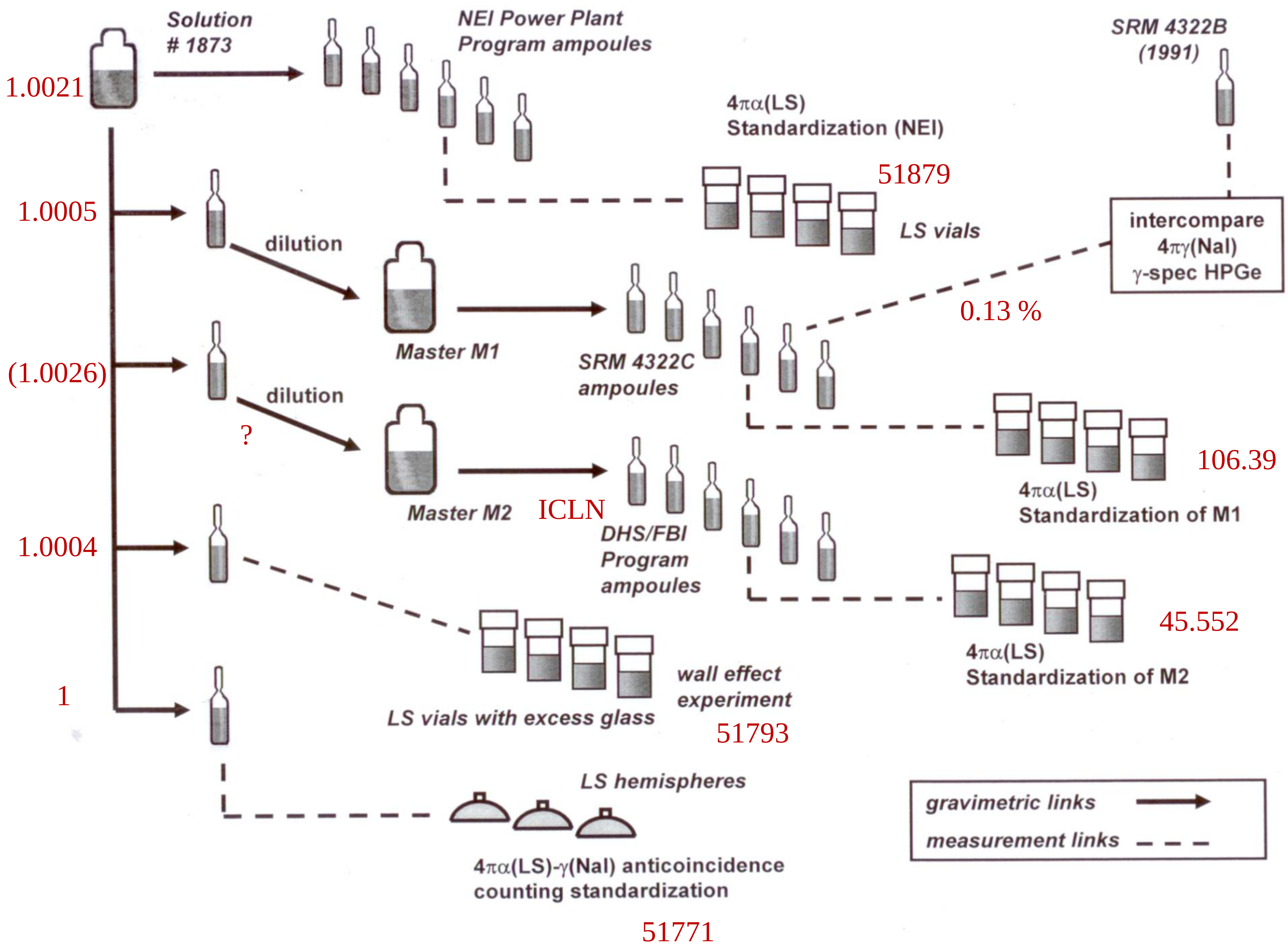
* Values are discrepant, and not considered to have confirmed

^{90}Sr & ^{241}Am

Both really easy β & pure α cases (near 100% efficiency)

33 years of linkages




Solution
1873
NEI Power Plant
Program ampoules
SRM 4322B
(1991)
1.0021
4πα(LS)
Standardization (NEI)
51879
1.0005
LS vials
dilution
intercompare
4πγ(NaI)
γ-spec HPGe
0.13 %
Master M1
(1.0026)
SRM 4322C
ampoules
dilution
?
106.39
Master M2
ICLN
4πα(LS)
Standardization of M1
DHS/FBI
Program
ampoules
1.0004
45.552
wall effect
experiment
4πα(LS)
Standardization of M2
1
LS vials with excess glass
51793
LS hemispheres
gravimetric links
measurement links
4πα(LS)-γ(NaI) anticoincidence
counting standardization
51771

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

^{90}Sr

^{241}Am

Nuclide	Method	relative standard uncertainty	Confirmatory Measurement	Difference (%)
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* Values are discrepant, and not considered to have confirmed

^{229}Th

hard case with 9 member decay chain

two nuclides (4 μs ^{213}Po & 32 ms ^{217}At) – resolving time issues

^{228}Th impurity (8 member chain) requires correction

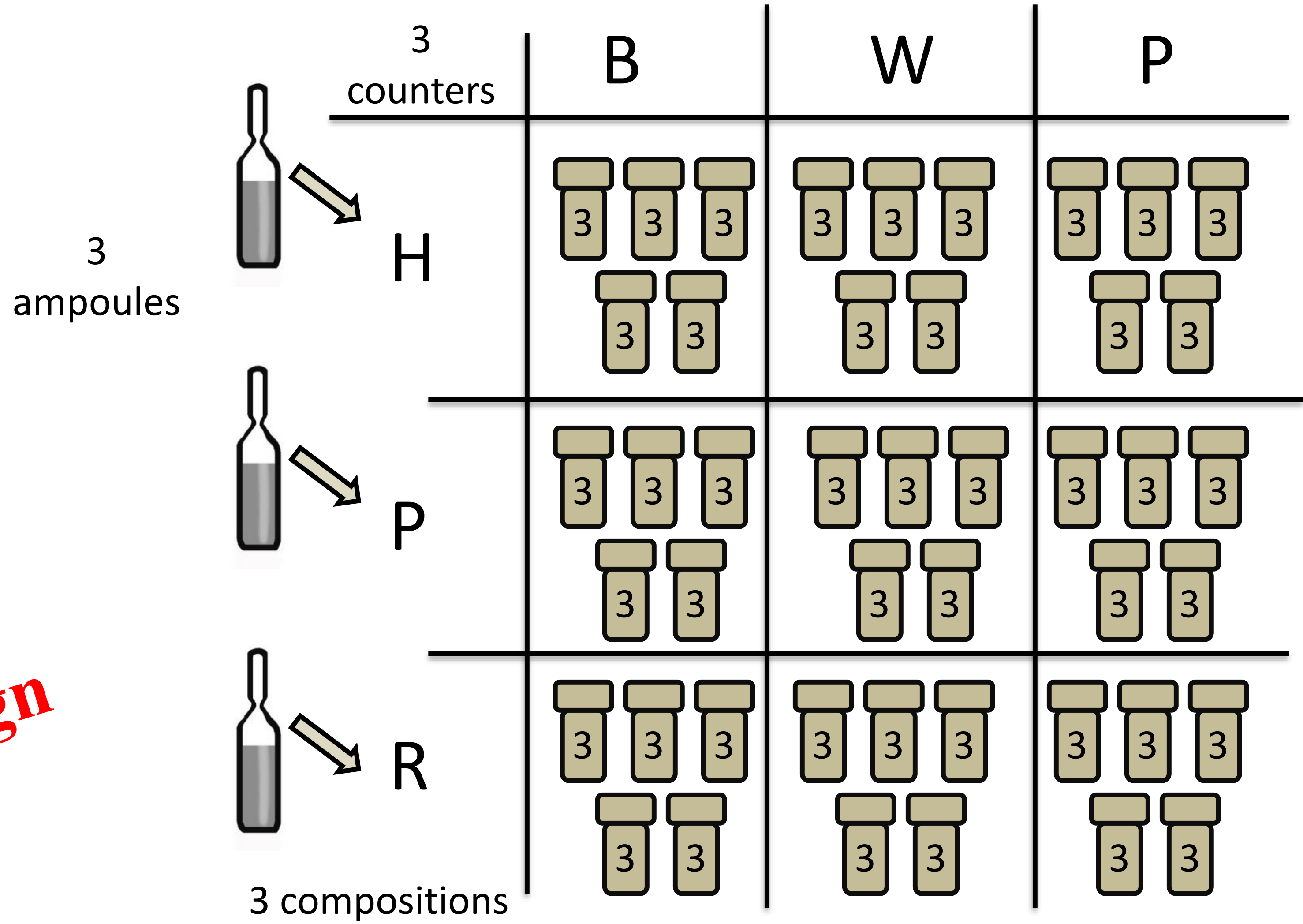
many methods

u_c = 0.30 %

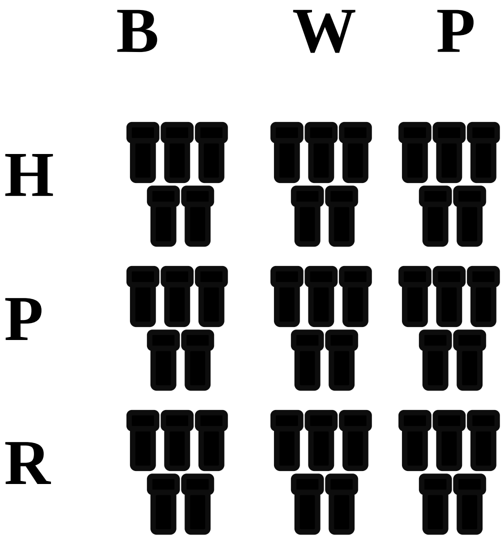
- extrapolation (0.2 %)
- ^{228}Th impurity (0.2 %)

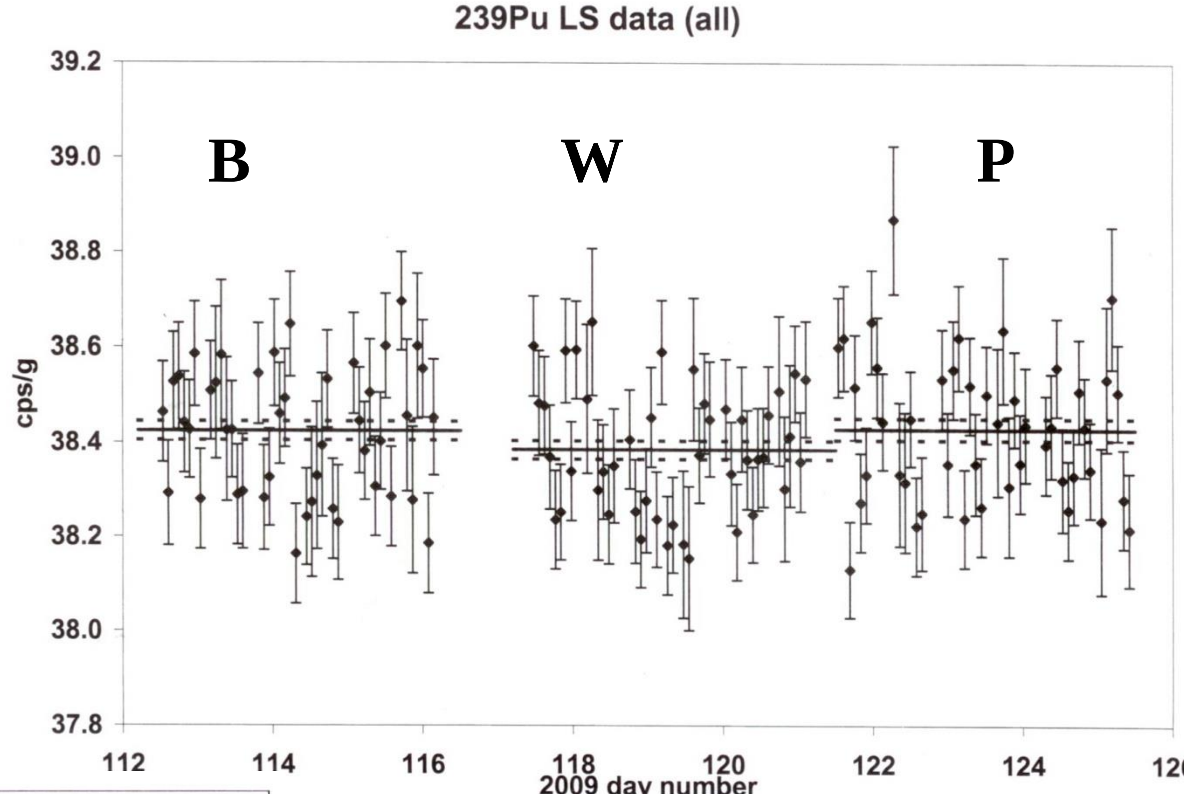
method	**Δ (%)**	**u_c (%)**
anticoincidence	---	0.3
CNET	-0.1	0.7
TDCR	-2.6	0.3
2πα	0.0	1.4
α-spec.	0.0	0.9
1986 SRM	+ 0.3	0.6

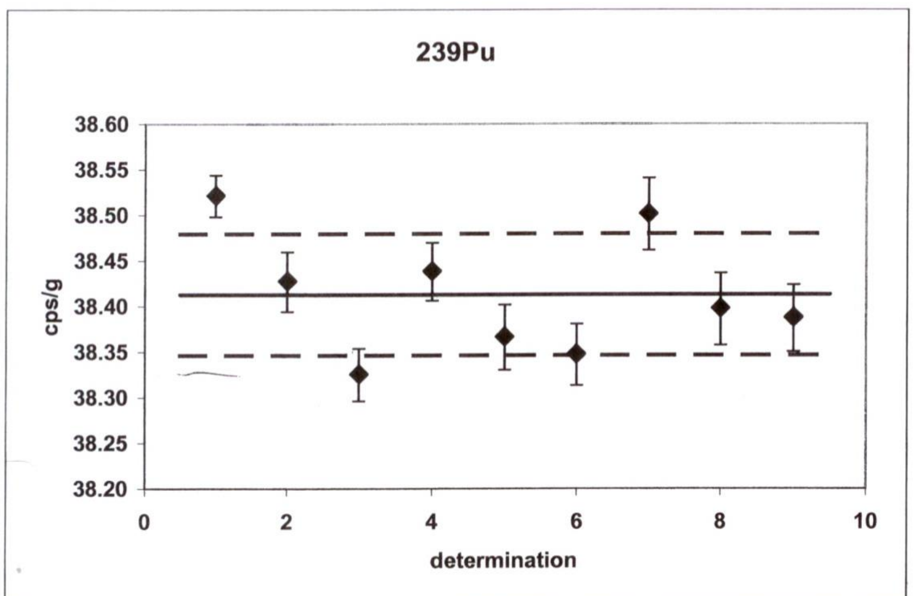
^{239}Pu

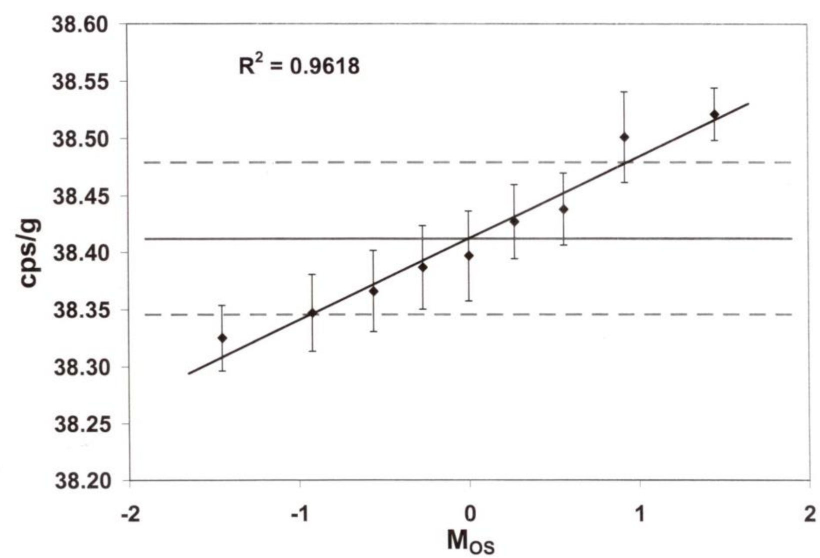


expt. design


B
W
P
H
P
R


239Pu LS data (all)
B
W
P
cps/g
2009 day number


239Pu
cps/g
determination


R2 = 0.9618
cps/g
MOS

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...*

→ *There are **NO"International Standards"** – censuses of equivalent labs doing primary standardizations*

FIN

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Brian Zimmerman (NIST)

Philippe Cassette (LNHB, France)

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Peter Volkovitsky (NIST)

Lynne King (NIST)

Denis Bergeron (NIST)

Lonnie Cumberland (NIST)

Michelle Hammond (NIST)

Evan Crawford (NIST)

Jerry LaRos (NIST)

Ken Inn (NIST)

Svetlana Nour (NIST)

Bruce Norman (NIST)

……others (world))

Backup slides only follow