$^{3}H_2O$

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+ today's silent partner,

L. Laureano-Perez

Radioactivity Group Seminar
13 May 2015

NIST

$^{3}H_2O$

What, Why & How

SRM 4927g

relative LS measurements

against 4927f

4926e

F1994

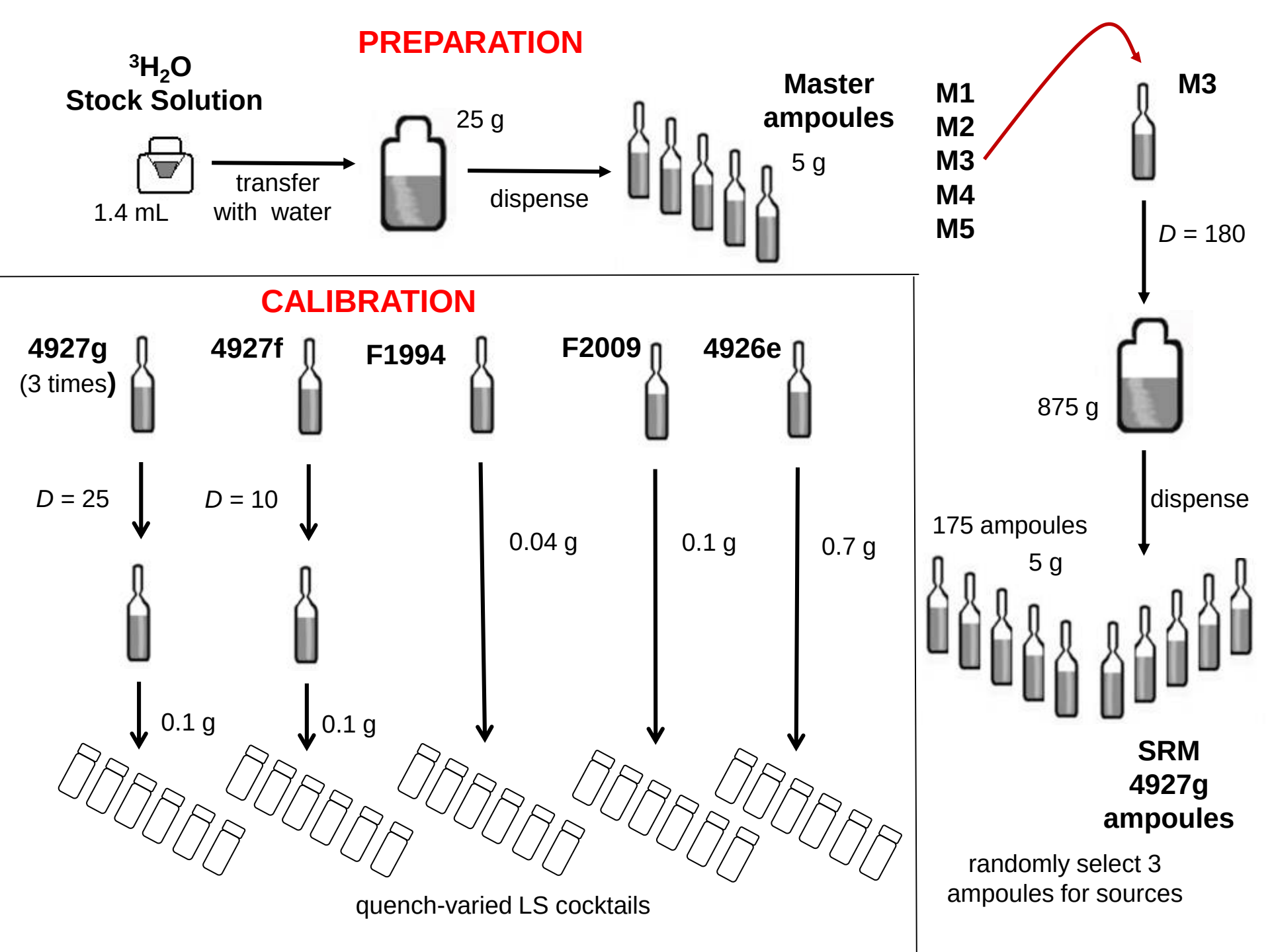
F2009

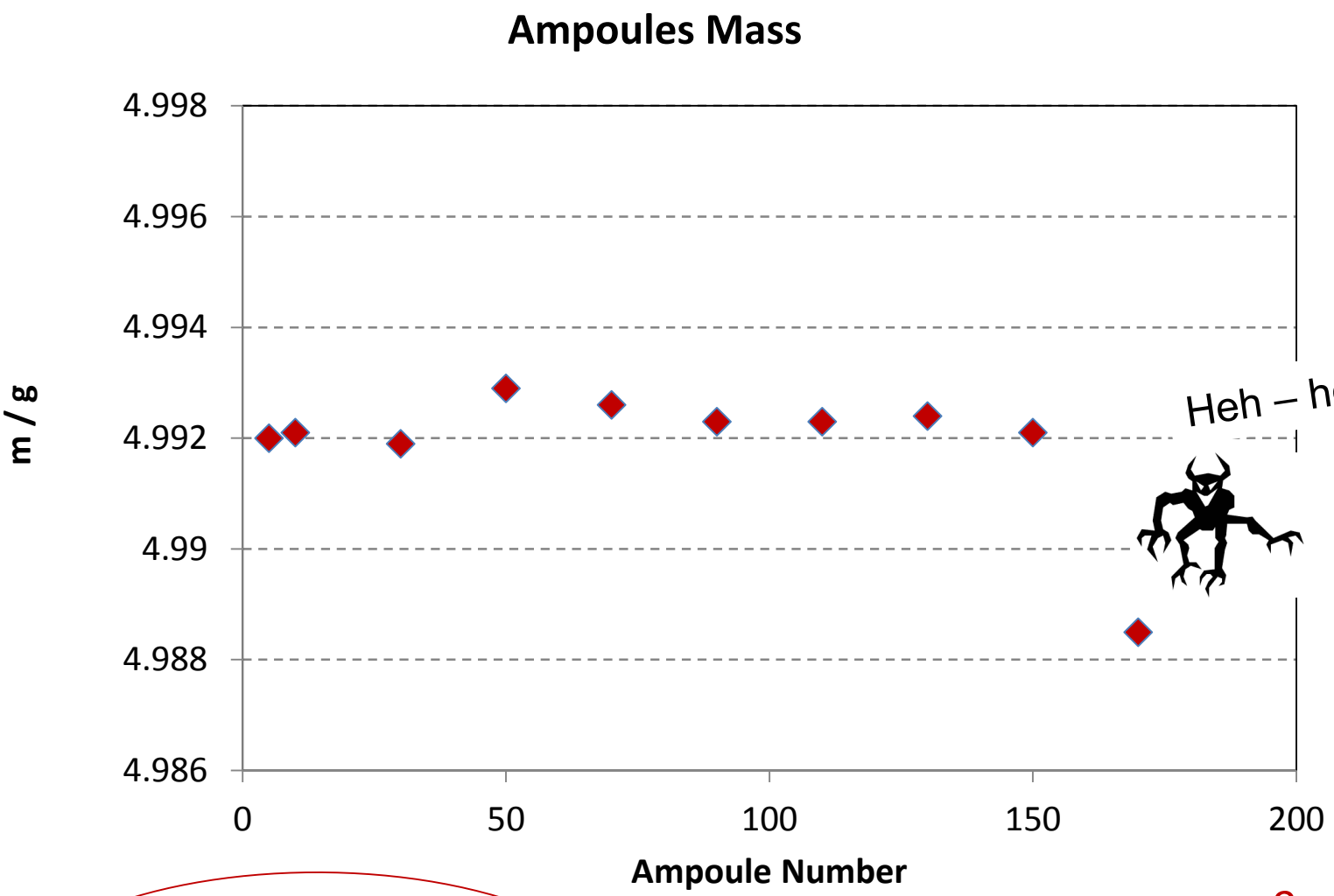
massic activity at T_0 = 1200 EST, 1 May 2015

uncertainty for **k = 1** including propagation for ^{3}H half-life uncertainty

^{3}H half-life = 12.312 (25) a

4927F	3 sept 1998	248.48 kBq g^{-1}	0.407 %
4926E	3 sept 1998	1.9723 kBq g^{-1}	0.407 % (Lucas has great linkage with 4927F) (0.06 % agree between DF & LS)
F1994	27 jan 1994	63.397 kBq g^{-1}	0.573 % (-0.37 % agree with 4927e in 1995)
F2009	31 may 2009	26.246 kBq g^{-1}	0.603 % (differs from “club” ave by – 1.4 %)
4927G	1 may 2015	≈ 540 kBq g^{-1}	≈ 0.5 % (at best a 1 % std on certificate)


PREPARATION
^3H_2O
Stock Solution
1.4 mL
transfer
with water
25 g
dispense
Master
ampoules
5 g
M1
M2
M3
M4
M5
M3
D = 180
875 g
dispense
175 ampoules
5 g
SRM
4927g
ampoules
randomly select 3
ampoules for sources
CALIBRATION
4927g
(3 times)
4927f
F1994
F2009
4926e
D = 25
D = 10
0.04 g
0.1 g
0.7 g
0.1 g
0.1 g
quench-varied LS cocktails



n	10	9
mean	4.9919	4.9923
sd	0.0012	0.00031
%sd	0.025	0.0063
%sdm	0.0078	0.0021

interesting issue on what to report

Is this okay ?

4.992 ± 0.002 ($k = 2$)

0.05 %

Some considerations on mass determinations

Recall example from ^{209}Po

26 april 2015

JUPITOR balace calibration for 3H SRM master

basket		35.26610			
		35.26669	35.26693		
		35.26800			
				apparent mass	
basket +	603.2	638.54610		638.46801	1.000124
		638.54651	638.54689	603.27888	1.000131
		638.54807			
basket +	603.4	638.74774		638.66801	1.000125
		638.74908	638.74792	603.47991	1.000132
		638.74694			
basket +	603.9	639.16772			
		639.16938	639.16907	603.90106	1.000002
		639.17010			
basket +	1483	1518.27100		1518.26801	1.000002
		1518.27232	1518.27178	1483.00377	1.000003
		1518.27202			
basket +	1485	1520.26782		1520.26801	1.000001
		1520.27007	1520.26907	1485.00106	1.000001
		1520.26932			
basket		35.26741			
		35.27032	35.26908		
		35.26952			
basket	ave		35.26801		

0.006% from initial.

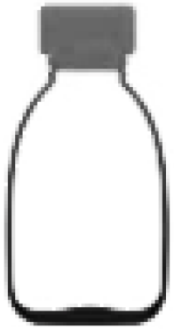
basket	35.265
basket+ bottle	638.537
bottle	603.272
basket + bottle + solution	1518.748
solution	880.211

0.013% at bottle

0.0003% at bottle+soln

Voland Jupiter 3000 (mechanical)
balance calibration error
< 0.0002 % at 600. to 605.125 g
< 0.0003 % at 1680. to 1685.225 g

Mettler AT20 (electronic)
balance calibration error
< 0.0009 % at 2.252 g
< 0.0004 % at 7.152 g



605.69543
(empty)

1075.90762
(total contained)

SRM
solution

master
amp # 2

4.919196

master
amp # 4

4.858217

1671.82726
(with HCl)

1681.60305
(filled)

Σ = 9.777413
(dispensed)

Δ = 9.77579
(contained)

0.017 %

LS mean values

$$\frac{\text{master}}{\text{SRM}} = \frac{4292.1 \pm 0.13\ \%}{39.011 \pm 0.23\ \%}$$

= 110.0235
± 0.26 %
(highly correlated overestimated uncertainty)

0.015 %

$$DF = \frac{1075.90762}{9.777413}$$

DF = 110.04011
± 0.025 %
(uncorrelated)

estimate

FORM NBS-569 (10-8-69)

COMPUTATION SHEET

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

TEST NO. _____ 3H DATE April 16, 2015

A	B	C	D	E	F	G	H	I	J	K

RUN NUMBER

Preparation of Master Solution for SRM 4927g

T= 21.6
P= 29.91
RH= 26.0%

ρ_{air} = 1.194716678
$\rho_{a,w}$ = 8.00
ρ_{H_2O} = 0.9976
corr = 1.001049147

(1) Jupiter

MT bottle + basket = 638.53759
638.53720 } 638.53701
638.53625

bottle + H_2O = 1513.75038
875g 1513.74910 } 1513.74986 876.13108
1513.75009

(2)

bottle + H_2O + ^3H = 1518.74830
1518.74843 } 1518.74855 5.00393
1518.74893

(3)

T= 21.6
P= 29.91
RH= 29%

ρ_{air} = 1.186315036
$\rho_{a,w}$ = 8.00
ρ_{H_2O} = 0.9973
corr = 1.001042477

T= 24.0
P= 29.92
RH= 17%

M3 Opened

TARE = 2.323726 2.323756
pyc + ^3H = 8.831306
pyc - ^3H = 3.827600 5.008722

T= 24.0
P= 29.92
RH= 17%

Total in bottle = 876.13108 + 5.008722 = 881.139802

basket = 35.26401 35.26169 35.26020
35.26575 35.26641 35.25992
35.26541 35.26499 35.26151

USCOMM-NBS-DC

5/2/2015

RE-CALCULATED DF FOR SRM 4927G from master campoule

baskets tare

35.26401 + .26575 + .26541 = 35.26506
35.26169 + .26641 + .26499 = 35.26436
35.26020 + .25992 + .26151 = 35.26054

(1) − 0.00070
(2) (3) − 0.00382

USE THIS TO GET ACTIVITY OF MASTER AMPOULES FROM 4927G CERTIFICATION

638.53759, 720, 625 } 638.53701

1513.75038, 74910, 75009 } 1513.74986

1518.74830, 843, 893 } 1518.74855

875.21285 + 0.00070 (1.001049147) = 876.13178

4.99869 + 0.00382 (1.001049147) = 5.007758

w/ tares

^3H_2O dispensed/contained

$\frac{5.008722}{5.007758} = 1.0001925$

0.019%

NOT $\frac{5.008722}{5.00393} = 1.0009576$

0.096%

TOTAL CONTAINED = 881.139538

^3H_2O DISPENSED = 5.008722

DF = $\frac{881.139538}{5.008722}$ = 175.92103 ☆

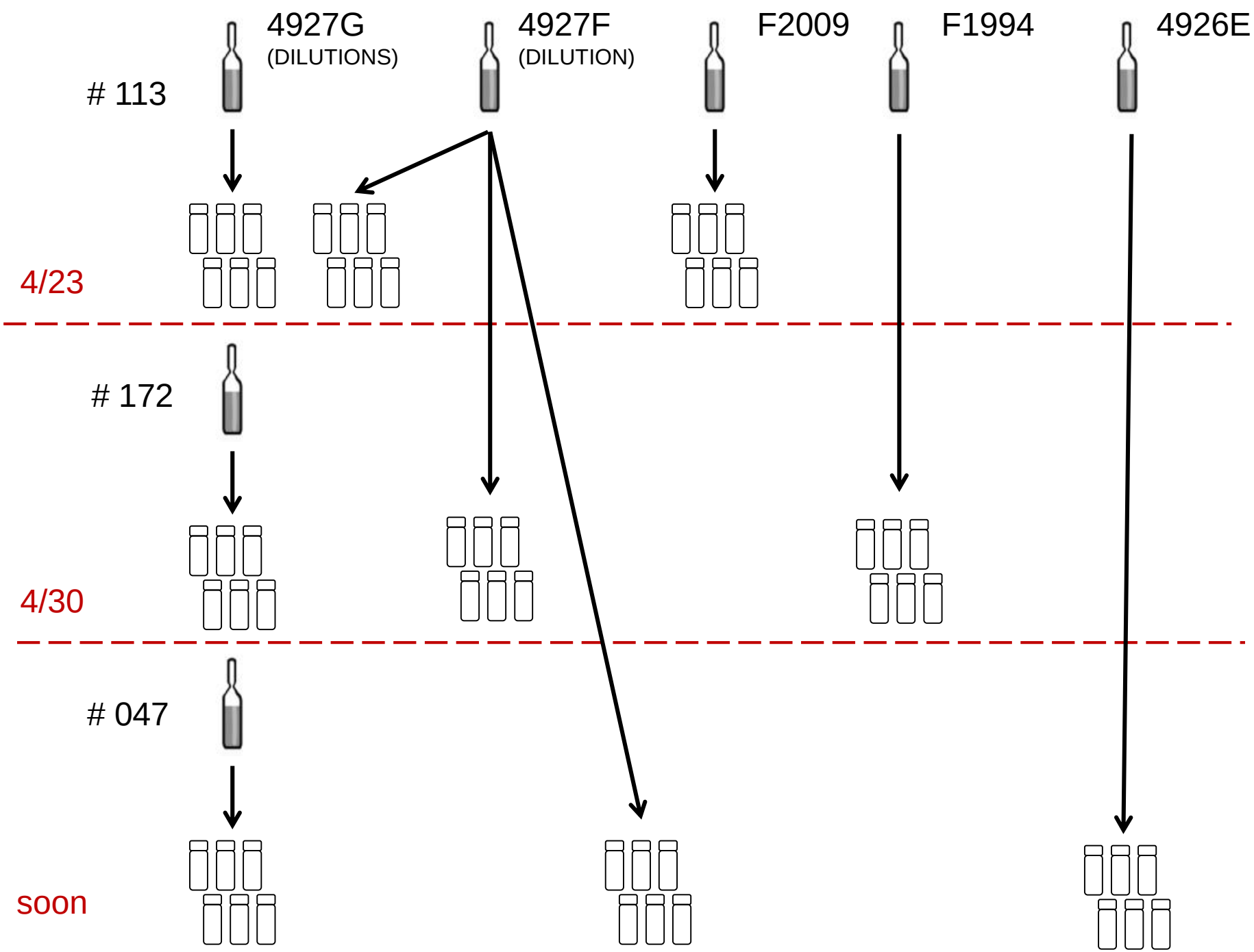
DILUTIONS OF SRM AMPOULES

TO MAKE SOLUTIONS FOR LS SOURCES

ampoule	stuff	contained mass	dispensed mass	% difference	DF
# 113	diluent	4.670206	4.670482	0.006	24.3648
	^{3}H	0.199740	0.199876	0.068	
# 172	diluent	4.820733	4.820920	0.004	22.8051
	^{3}H	0.220922	0.221056	0.061	

N.B. generally,

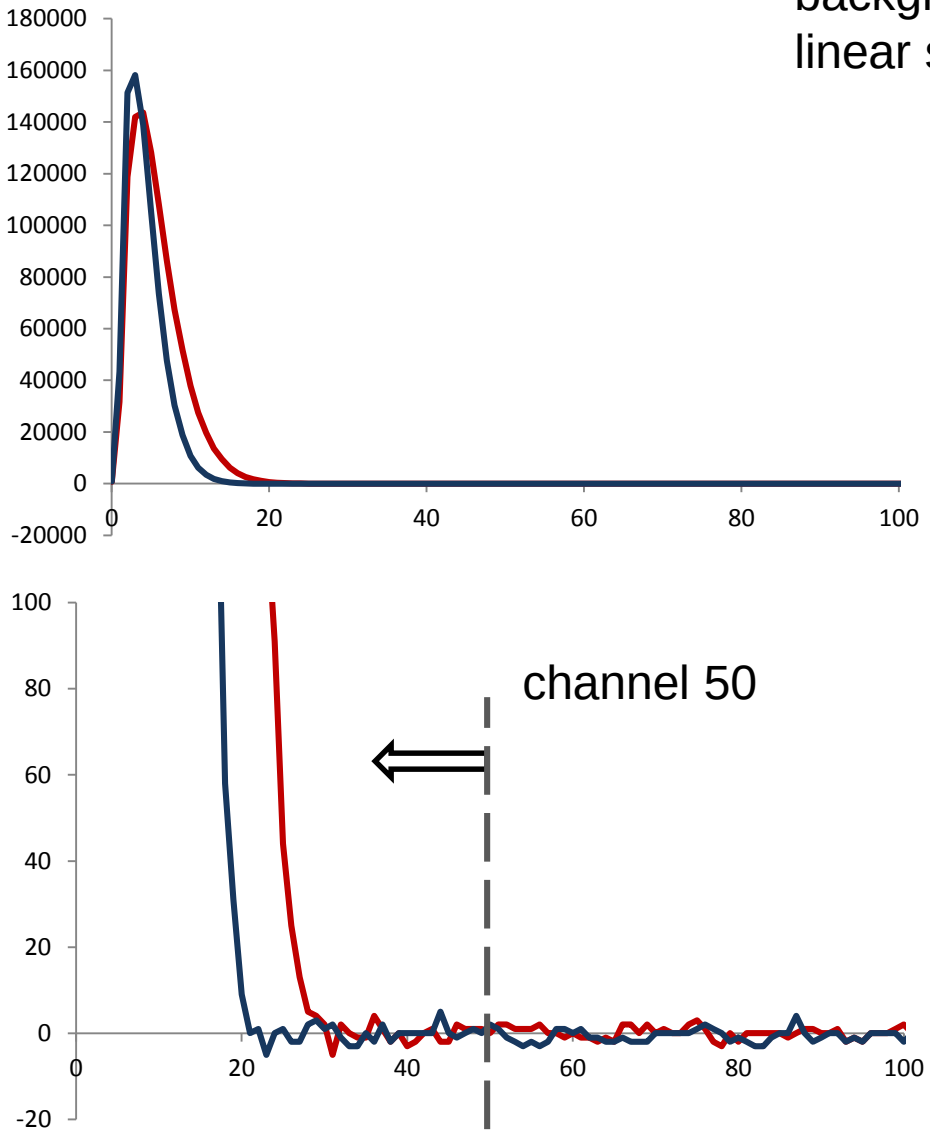
$$DF = \frac{total\ contained}{dispensed\ aliquant}$$


4927G
(DILUTIONS)
4927F
(DILUTION)
F2009
F1994
4926E
113
4/23
172
4/30
047
soon

Beckman spectra

background subtracted

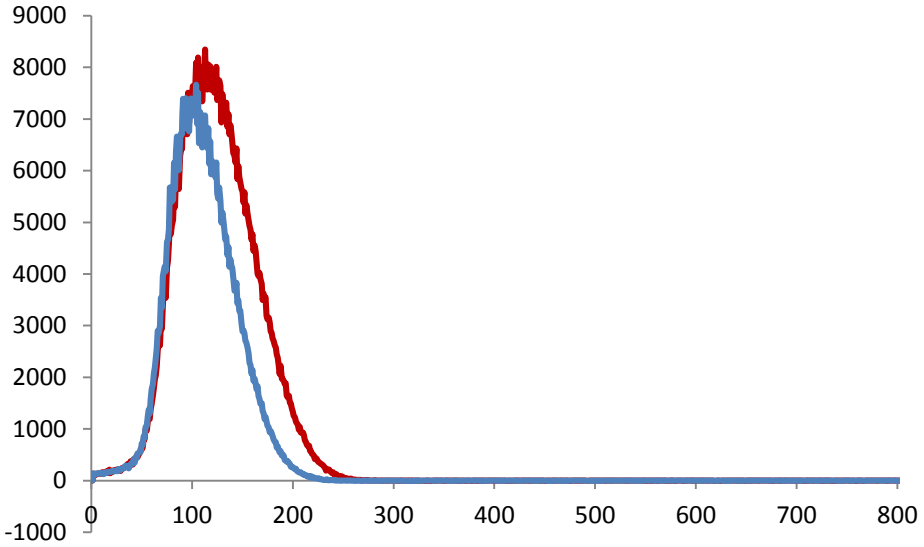
linear scale

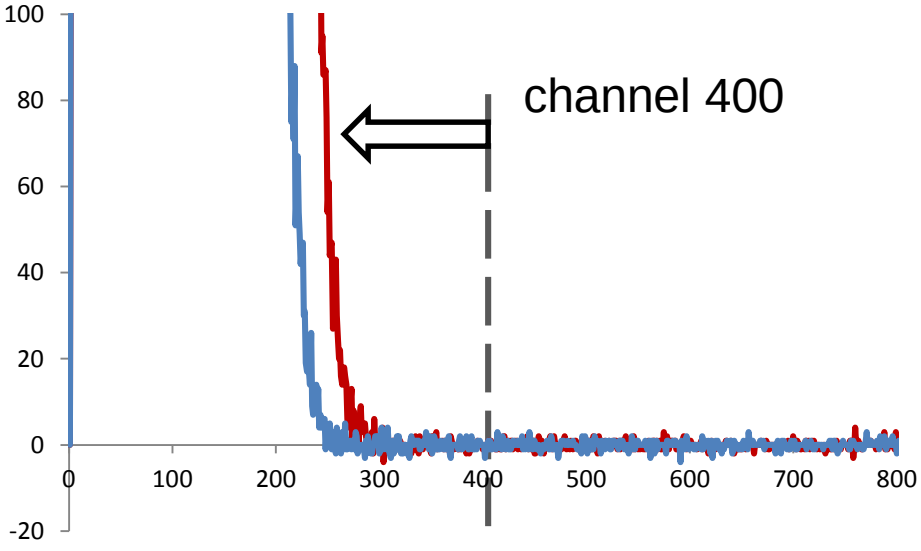


Wallac spectra

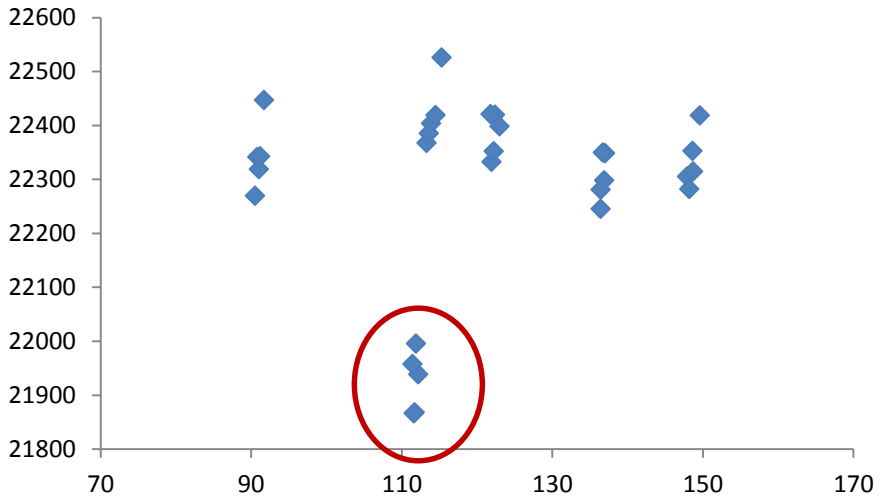
background subtracted

log scale





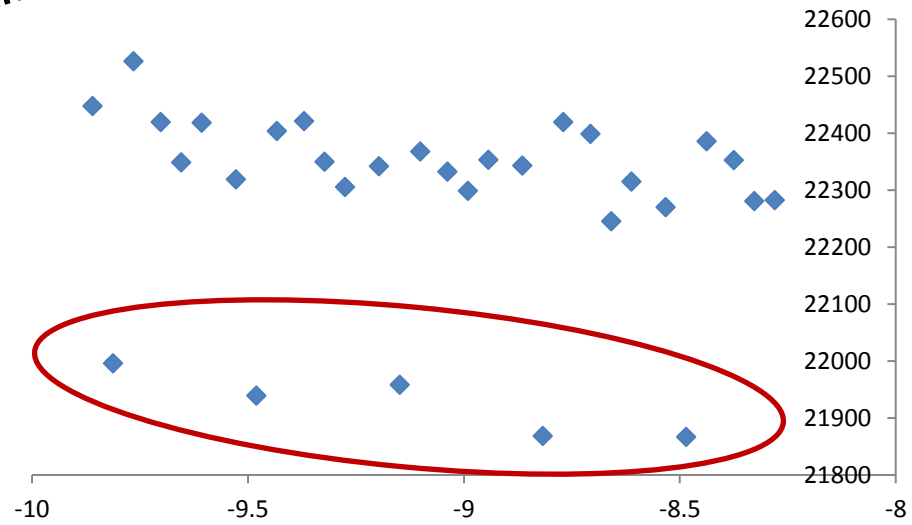
against H#



one screwy cocktail

in 4927G series

against time



Same in all 3 runs

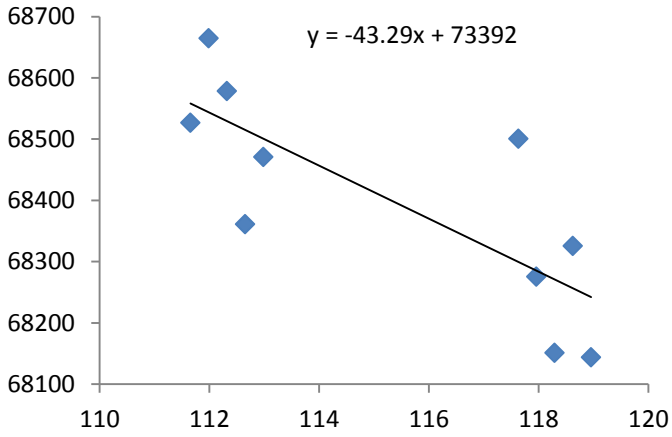
4/21 Beckman

4/23 Wallac

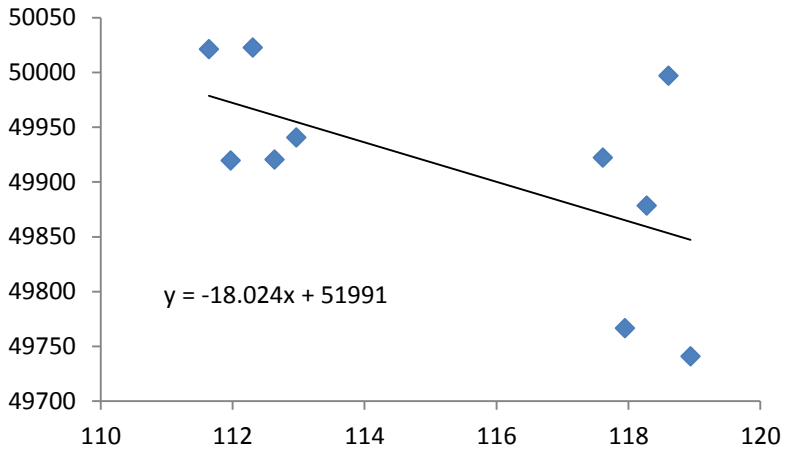
4/27 Beckman

slight cocktail instability

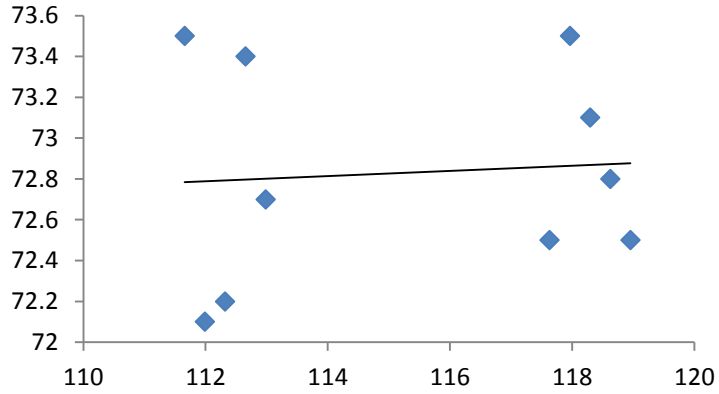
4927F



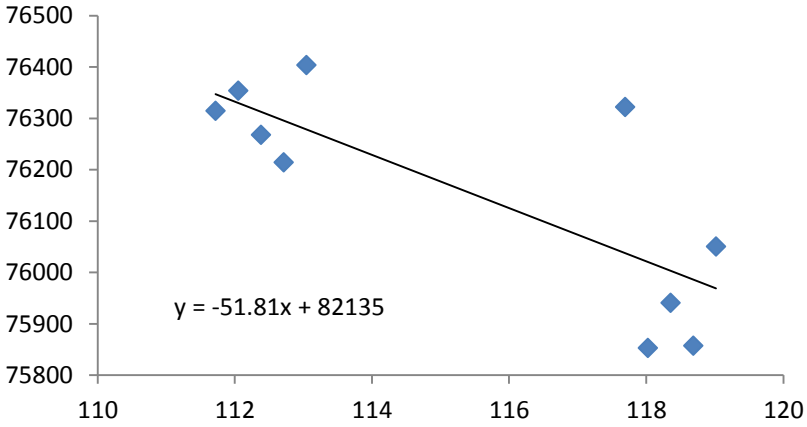
4927G



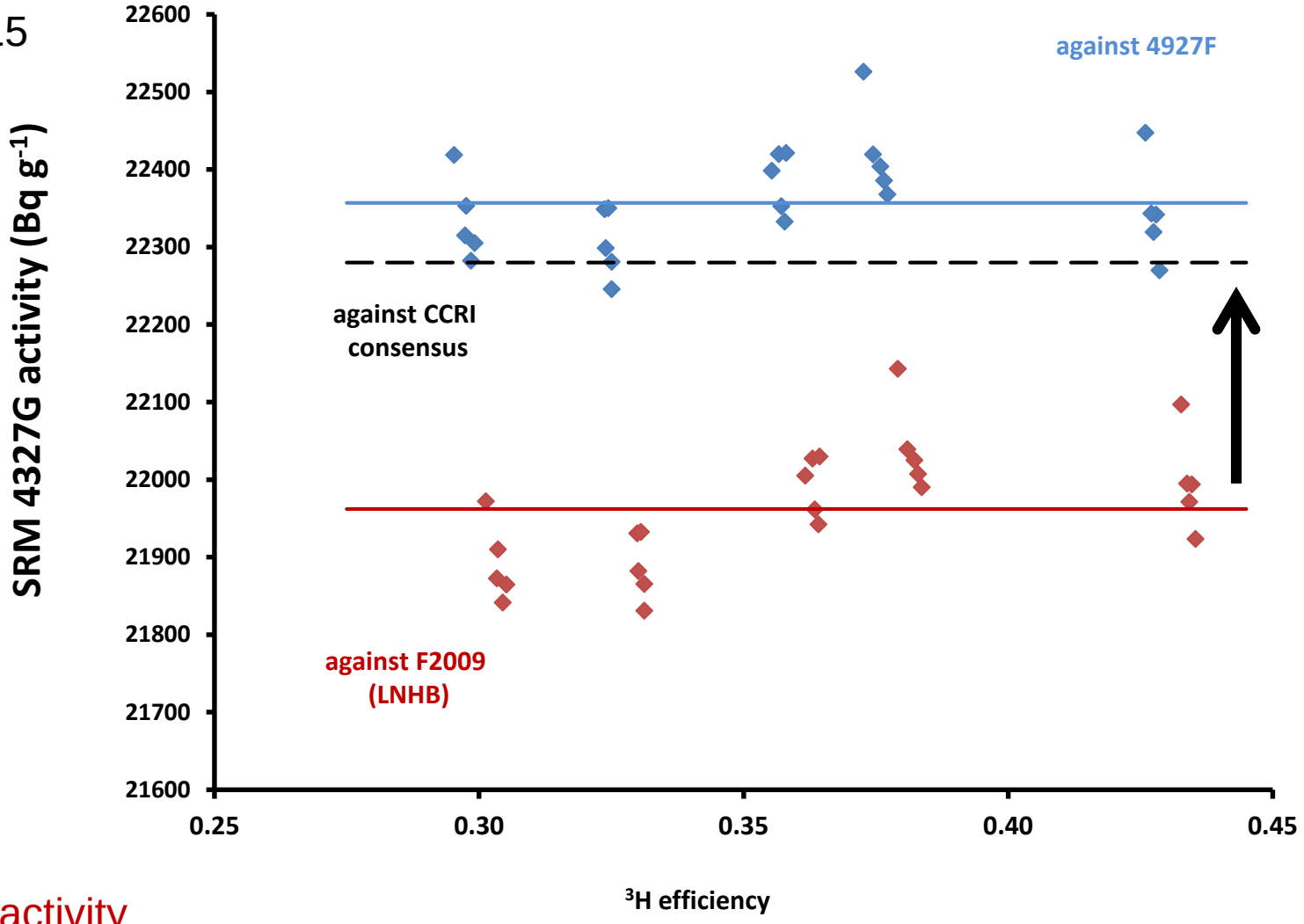
4927F



F2009



BECKMAN
21 april 2015



just dilution activity
for # 113

needs DF = 24.3624

^{3}H results

So far …

		vs 4927F		vs F2009		
	2015 date	Bq/g	s (%)	Bq/g	s (%)	ratio
Beckman	21-Apr	22316	0.20	21918	0.26	1.018
Wallac	23-Apr	22334	0.16	22001	0.29	1.015
Beackamn	27-Apr	22371	0.19	21977	0.28	1.018
mean		22340		21965		
sd between		28.042		42.712		
%sd between		0.126		0.194		
%s within typcal		0.187		0.278		
big S %		0.225		0.340		
difference %			1.71%			

n = 5 within cycles
(typical sdm)
+
n = 5 between sources
(full sd)

n = 3 between sources
(full sd, but equivalent to sdm)

n = 5 x 5 typical s

will continue to nest similarly with data from more trials

Uncertainty = [$(\text{sdm within})^2 = (\text{sd between})^2$] $^{1/2}$

Where are we ....

And where are we going ...

counted & analyzed

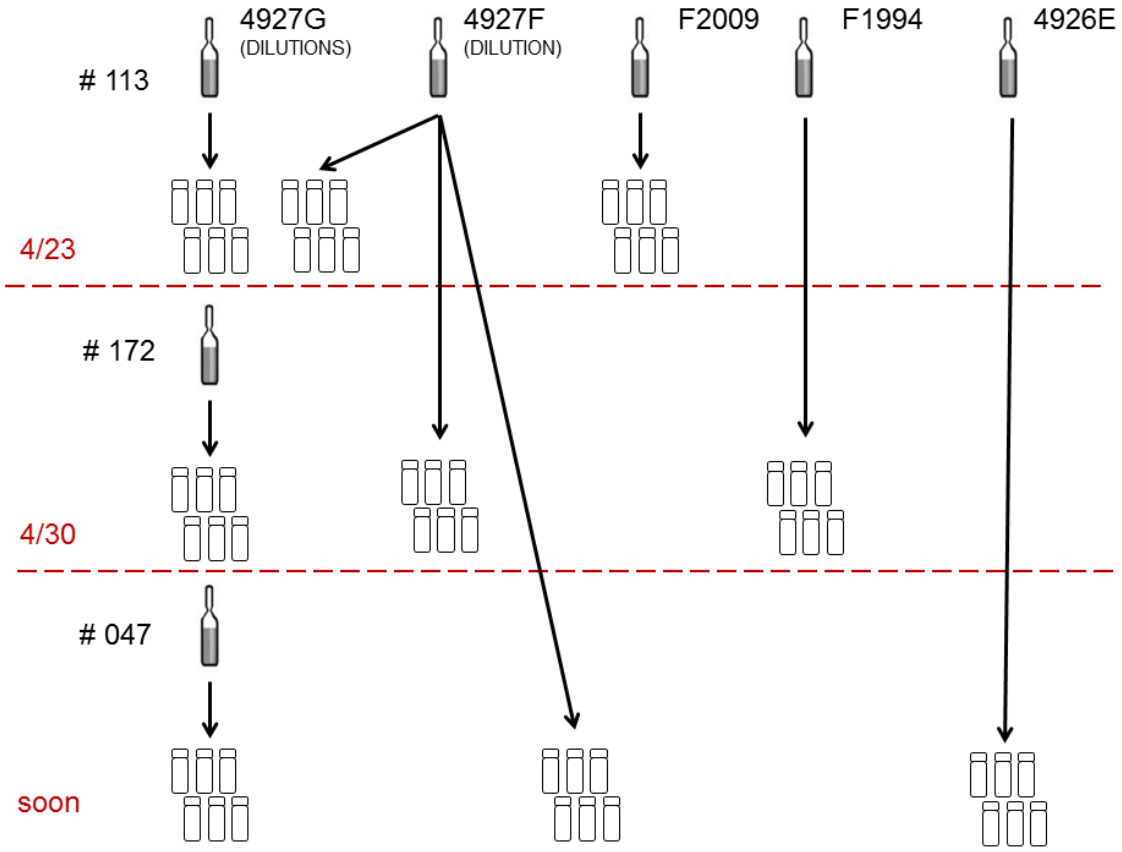
Beckman
Wallac
Beckman

counting

Beckman
Wallac
Beckman

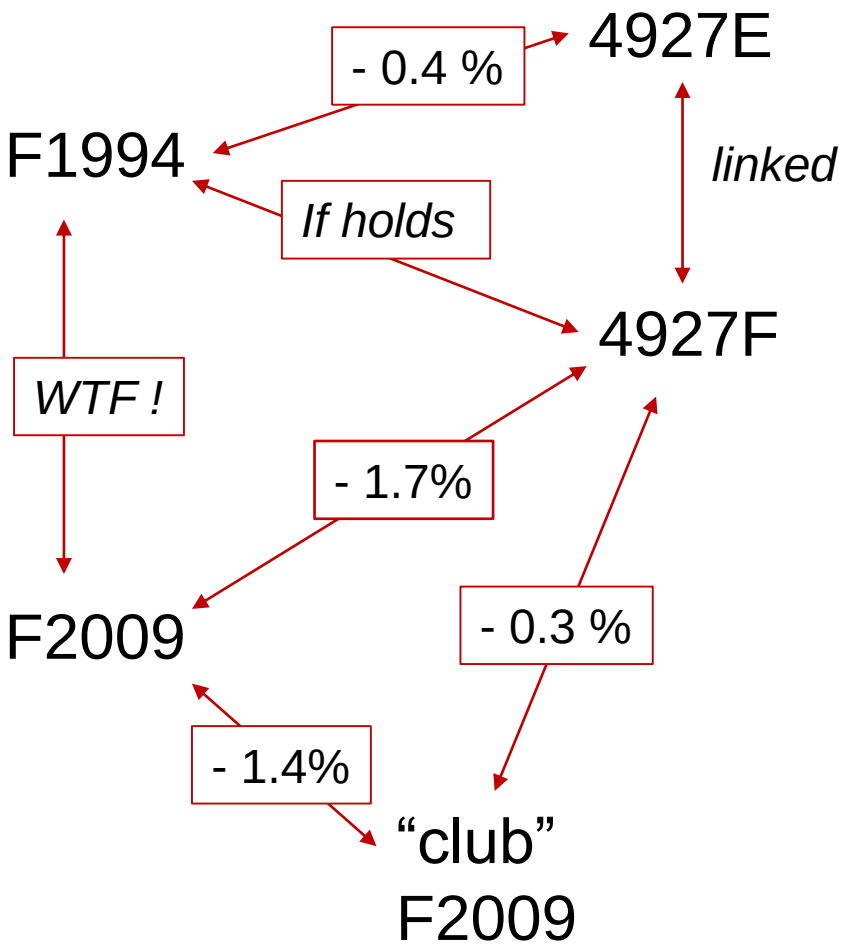
tbd

Wallac
Beckman
Wallac
Wallac



All cocktails with UGAB at 7 % water

Will do some part of same scheme with another cocktail composition



WHAT MAY

TURN OUT TO

BE GREAT

FUN

Voila – you got it so wrong ....

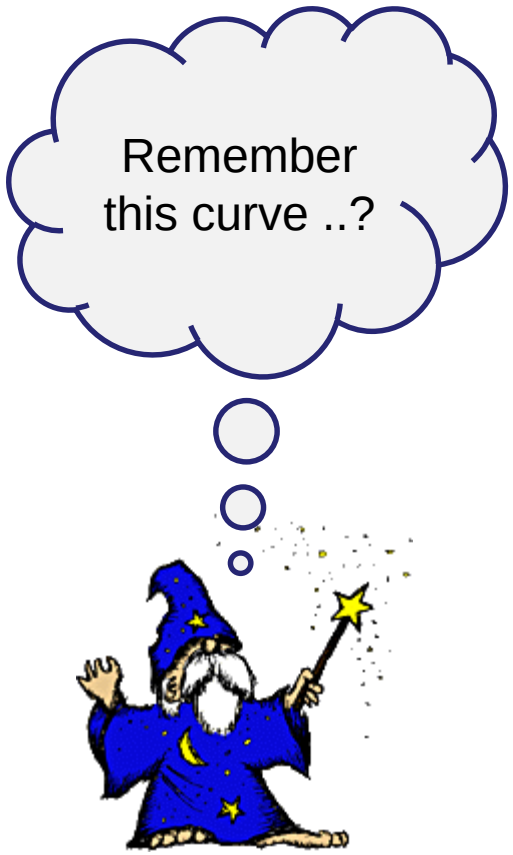
mais pourquoi

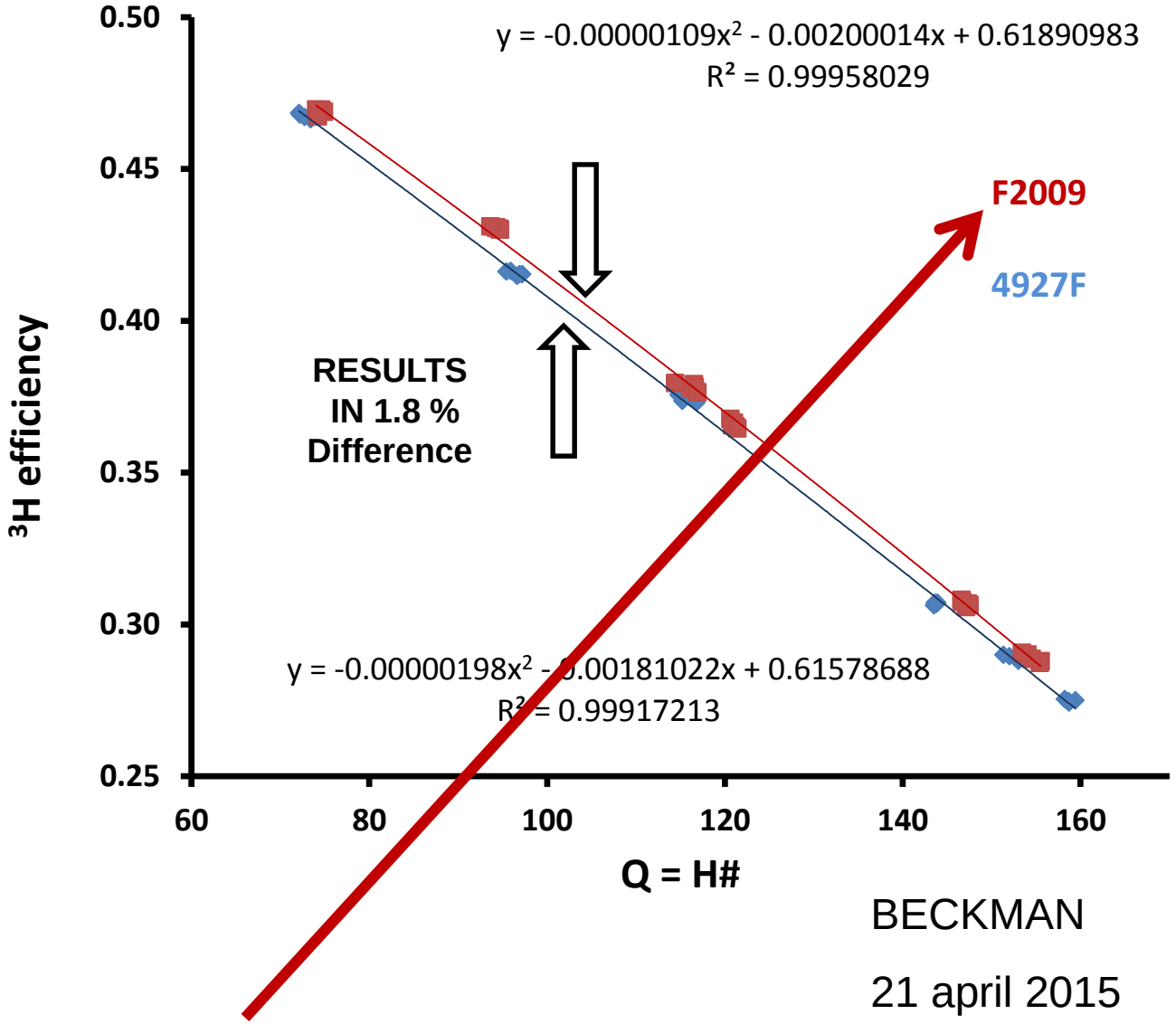
THAT WAS THE STORY

I HAD LAST FRIDAY

BEFORE BEING OUT

FOR 4 DAYS





BECKMAN

21 april 2015

For the 1st French solution

This is for the 2nd French solution
against the same 4927F