

Bioassay Analyses

Theresa Davis

EQO-Analytical Services

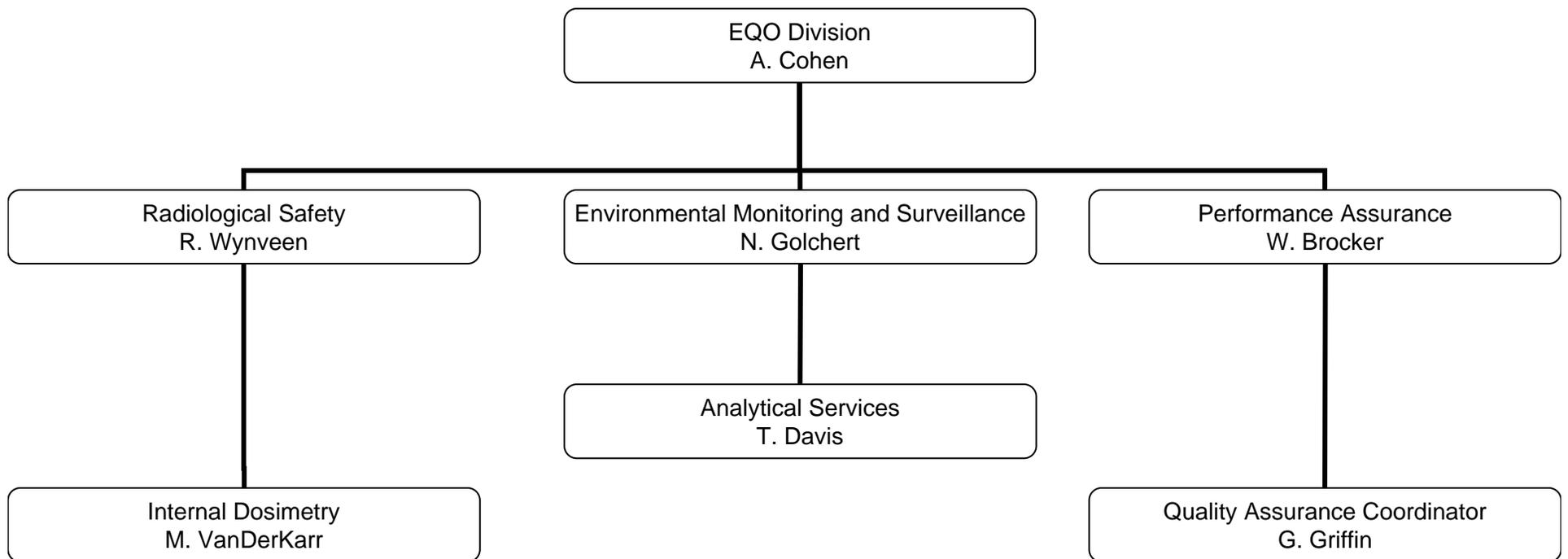
7/23/2004



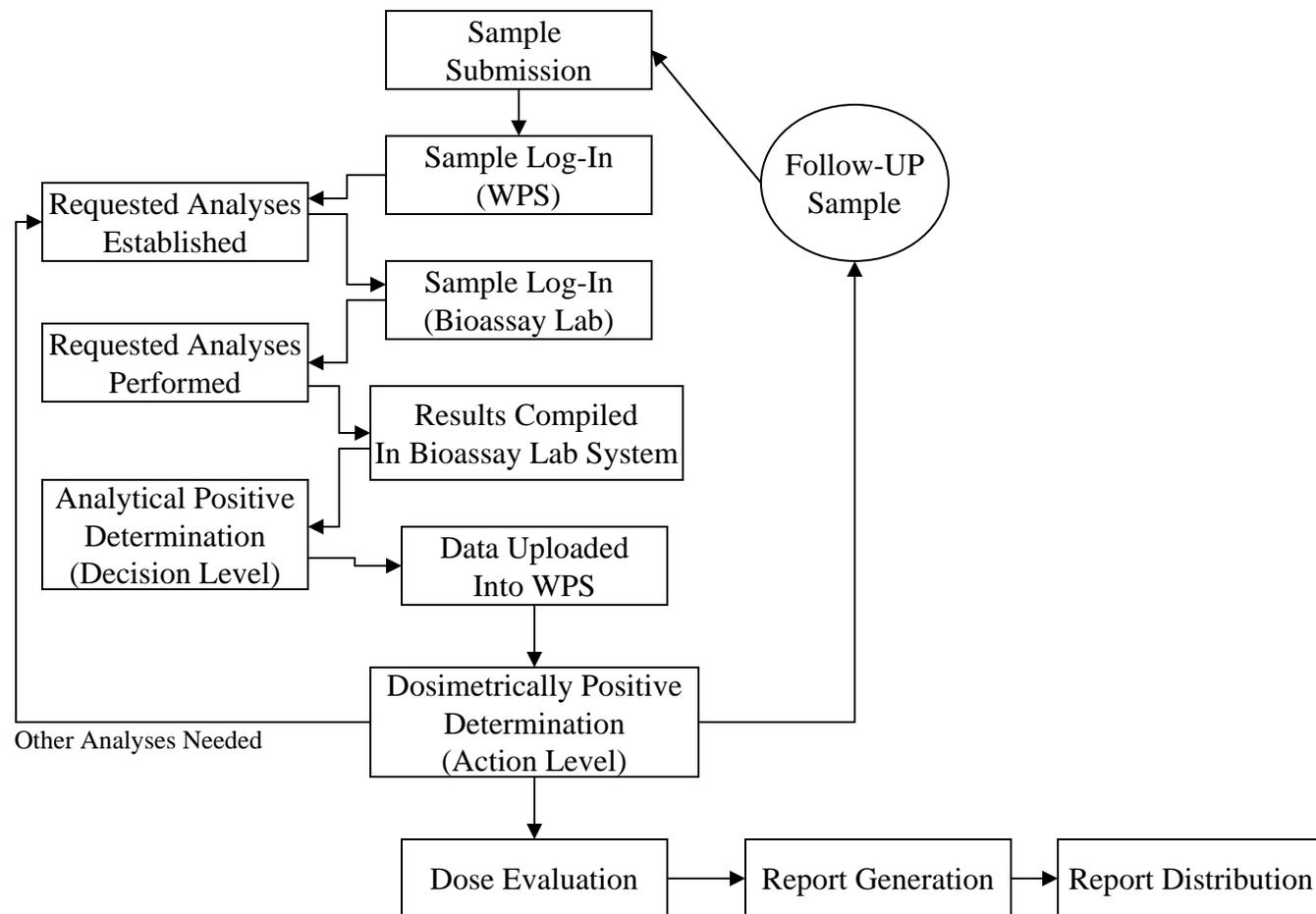
Program Elements

- Strong organization
- Strong client relationship
- Accreditation (DOELAP, DOE STD1112-98)
- Work Process Controls
- Experienced and Dedicated Workers
- Proficiency Samples
- Flexibility

Partial Organization Chart



Internal Dosimetry and Analytical Services Work Flow





Client Relationships

- Emergency Situations
- Ad-Hoc Analyses
- Changing priorities
- Shortened Turn-around Times
- All analytical work performed in-house



Accreditations



- DOE Laboratory Accreditation Program
 - Urine and fecal
 - Granted March 1999 and July 2002
 - Next visit 2005

- American Industrial Hygiene Assoc.
 - Solvents, Asbestos (fibers and bulk), metals
 - Granted September 1999 and May 2003
 - Next visit 2006



DOELAP Accreditation Process

Timeframe = approx 1 year

Cycle every three years

- Accreditation Application
- Proficiency Samples
- On-site Review
- DOELAP Oversight Board Review
- Accreditation Received



Work Process Controls

- Quality Assurance/Quality Control Plans
 - Conduct of Work
- Analytical Procedures (94 total/32 bioassay)
 - Analytical Process Steps
 - Safety Concerns
 - Training
- Training Matrix
 - List of Qualified Analysts
- Controlled Environment

Analytical Processes Training



■ Discussion Period

- Manager, Technical Lead, Qualified Analyst, and Trainee

■ Observation Period

- Qualified Analyst → Trainee
- Trainee → Qualified Analyst

■ Independent Performance

- Manager and Technical Lead -- Quality Check

■ Qualified Analyst

- Continued Proficiency
- Annual Skill Assessment Performed



Experienced Workers

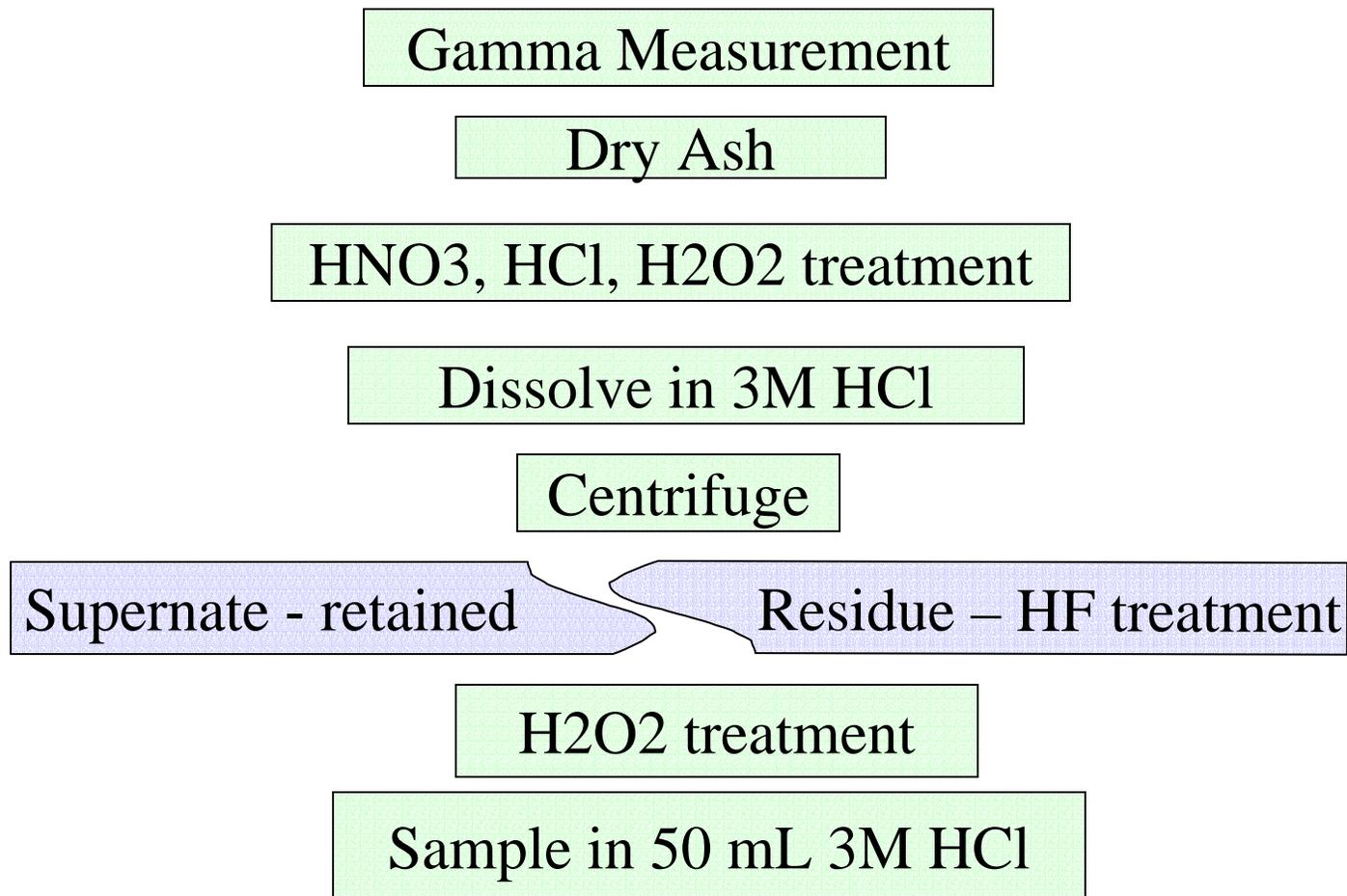
# of workers	# of years in bioassay laboratory
2	16
1	14
1	5
1	4



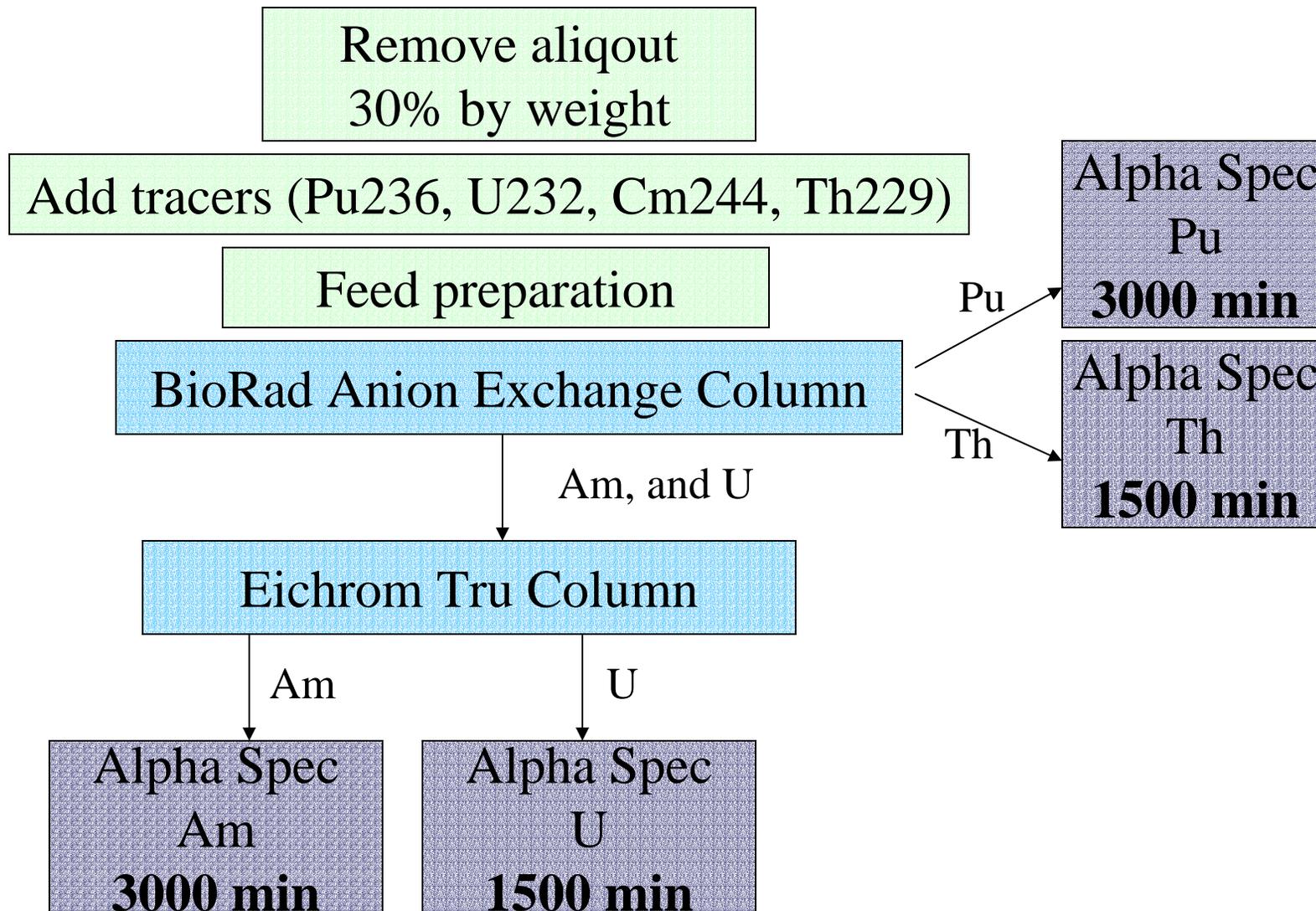
What happens to a Bioassay Sample?



Fecal Sample Analysis – 1



Fecal Sample Analysis – 2



Fecal Analysis

Listing of steps – page 1

Fecal - Pu/Am/U

TRACERS:
 Pu ²³⁶ 2003 AG 100 λ each
 Cm ²⁴⁴ 2004A4 50 λ each

Date: 6-23-04
 Analyst: 24891 / 35399

1. Remove Aliquot	
SAMPLE # / Size (g) Used:	
1. <u>04-0511</u> 18.75	<input checked="" type="checkbox"/> Enter information into fecal logbook
2. <u>-0515</u> 17.88	<input checked="" type="checkbox"/> Add appropriate tracers
3. <u>-0518</u> 17.37	<input checked="" type="checkbox"/> Add 10mg Fe ²⁺
4. <u>-0523</u> 16.02	<input checked="" type="checkbox"/> Add 1ml 30% H ₂ O ₂
5. <u>-0528</u> 16.5	<input checked="" type="checkbox"/> Add 30ml CONC. HNO ₃
6. <u>-0532</u> 16.65	<input checked="" type="checkbox"/> Reduce volume to 15ml
7. <u>-0534</u> 17.25	<input checked="" type="checkbox"/> Add 5ml CONC. HNO ₃ volume to 20ml
8. <u>-0541</u> 16.29	<input checked="" type="checkbox"/> Add 10ml deionized H ₂ O
9. <u>-0543</u> 19.53	<input checked="" type="checkbox"/> Add 10ml 2m AL(NO ₃) ₃
10. <u>-0545</u> 17.16	<input checked="" type="checkbox"/> Add 50mg NaNO ₂
11. <u>-0548</u> 16.62	<input checked="" type="checkbox"/> HEAT TO BOIL, COVERED
12. <u>-0550</u> 16.44	<input checked="" type="checkbox"/> Transfer to 50ml GL centrifuge tube
2. Centrifuge Samples	

Shake first/keep suspended. Transfer from centrifuge tube (30%) to 400 ml beaker (label) using disposable pipet.

Prepare as described below. Add 1ml. Wait 10 min. .7g Fe(NH₄)₂(SO₄)₂·6H₂O in 10 ml H₂O (deionized)

About 20 min. Covered (speedy vac). Prepare/number centrifuge tubes. (med. on Chromalox or 2.5 on Corning).

Flat covers. Does NOT take long. Evap 5 ml. Cool.

Rinse beaker with small amount deionized H₂O and also add. Volume should be about 40 ml. Cool sample then centrifuge for 1 hour.

#2 popped when evap feed from 4. Am/U step.



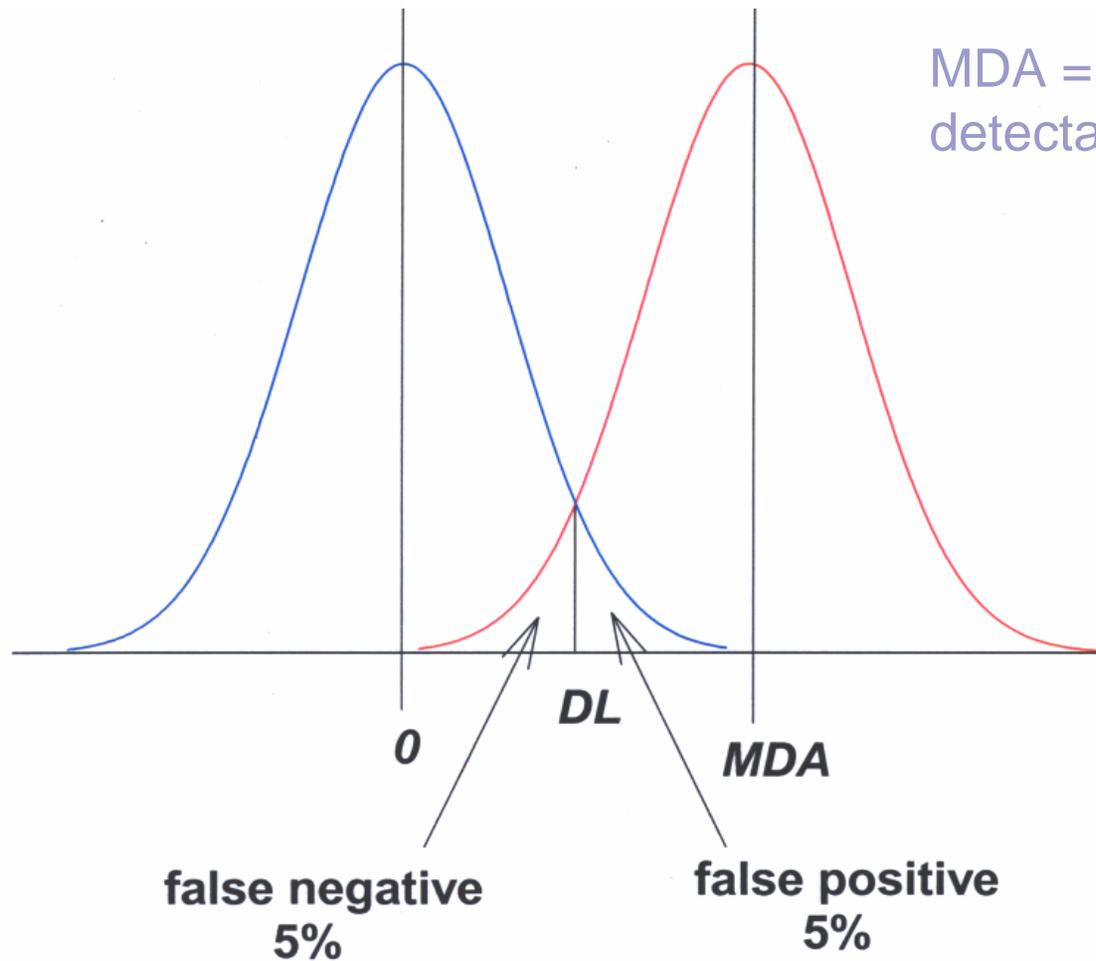
An positive result

- A result is analytically positive if it is above the decision level.
- A result can be analytically positive but not dosimetrically positive.

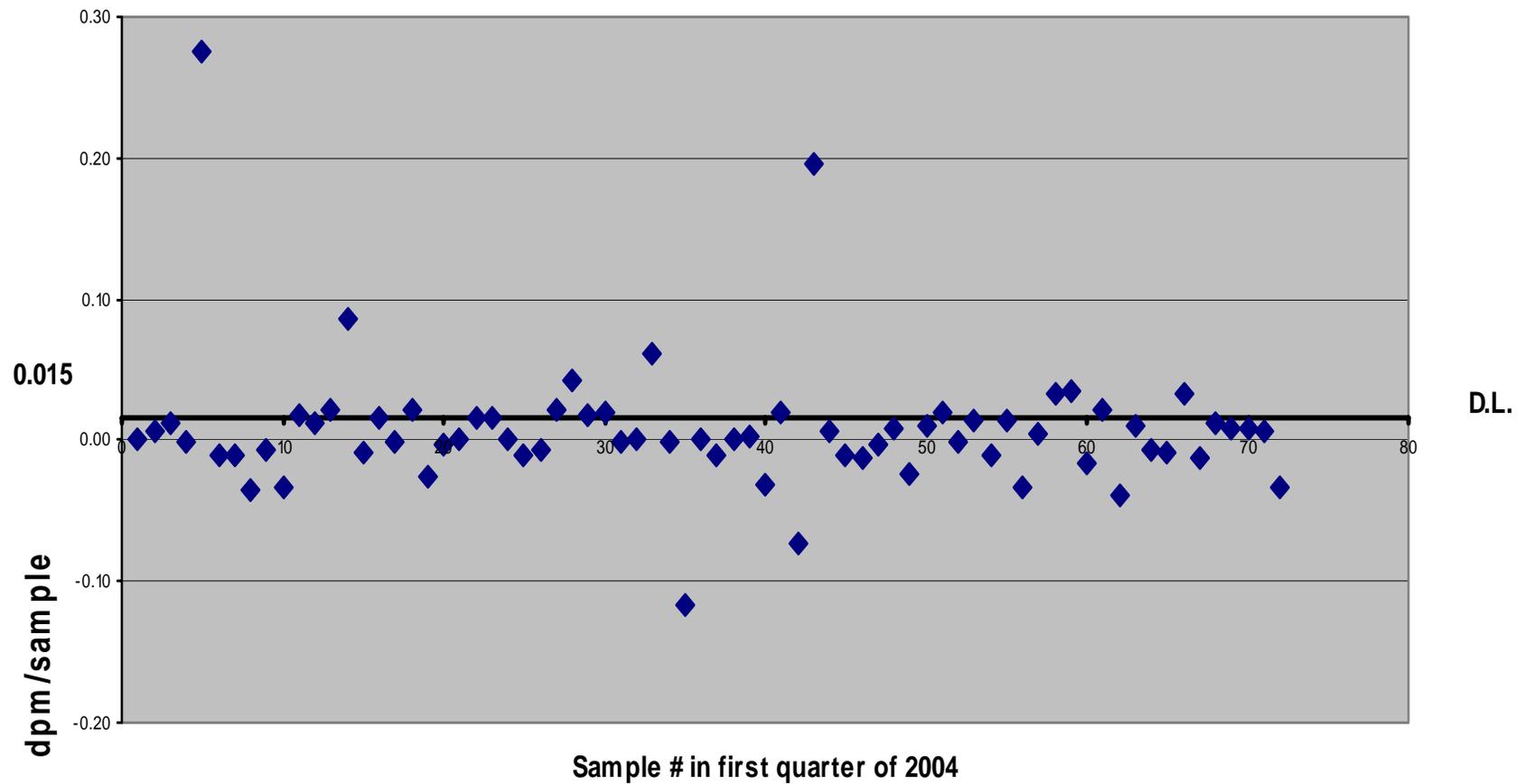
MDA and DL

DL = decision level

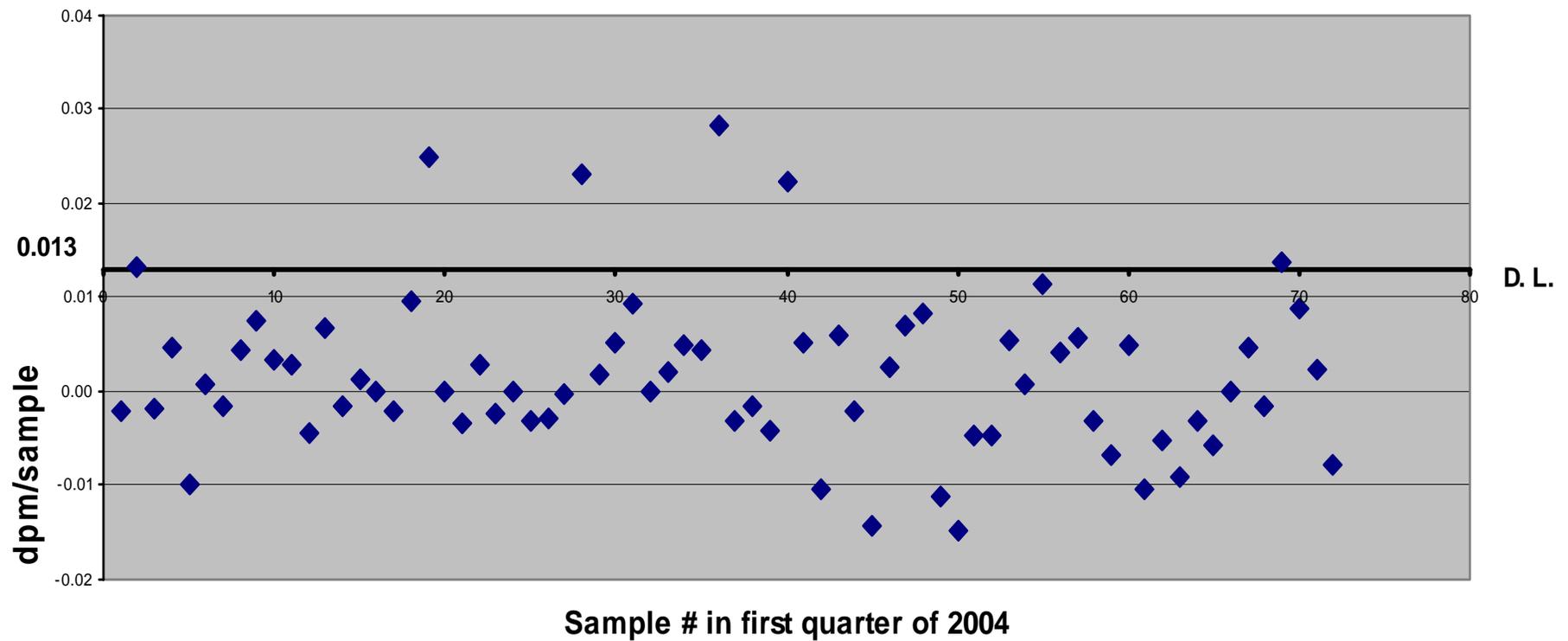
MDA = minimum detectable activity



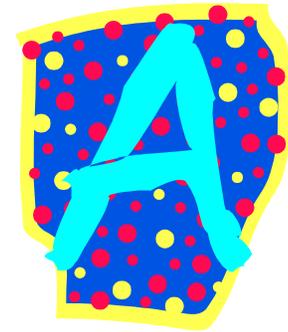
Pu-239/240 in Fecal



Pu-242 in Fecal



Proficiency Testing



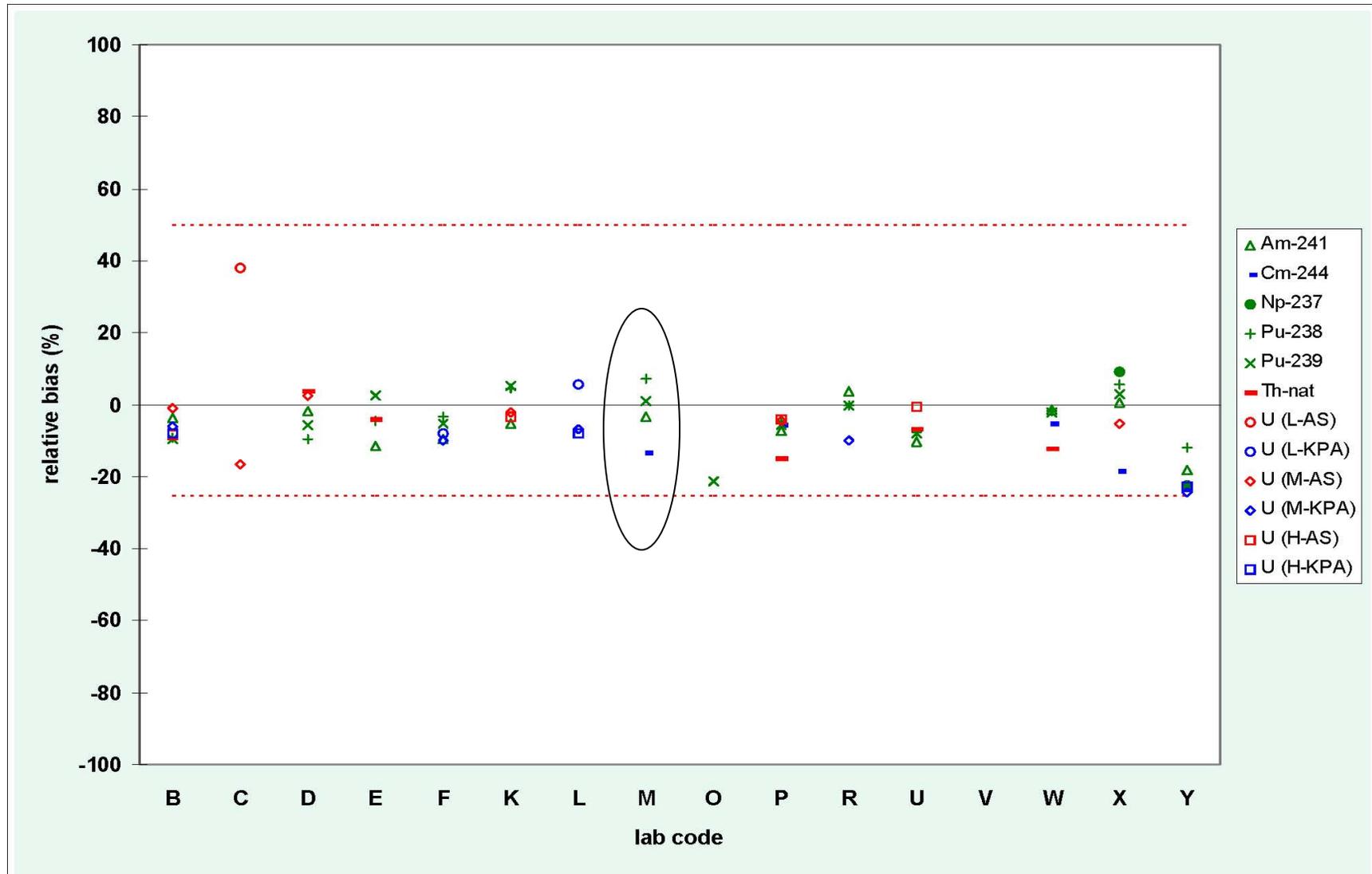
- Discharge Monitoring Report
 - Aqueous annual
- AIHA proficiency samples
 - Filters, charcoal, paint chips quarterly
- DOE-EML-QAP
 - Water, air filters, soil semiannual
- **DOELAP**
 - Urine and fecal triennial
- **Oak Ridge National Laboratory**
 - Urine and Fecal semiannual

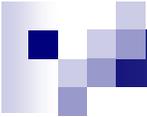
Proficiency Sample Results ORNL Urine Q2 2003

Nuclide	Known	Result	%Bias
Am-241	0.70 ± 0.04	0.77 ± 0.20	9.44
Am-241	4.28 ± 0.21	4.50 ± 0.68	5.21
Cm-244	0.57 ± 0.03	0.57 ± 0.15	-0.18
Co-60	375 ± 19	339 ± 34	-9.52
Cs-137	293 ± 15	279 ± 38	-4.73
H-3	116 ± 6	109 ± 4	-6.38
Pu-238	0.41 ± 0.02	0.48 ± 0.10	16.05
Pu-239	0.28 ± 0.01	0.29 ± 0.07	2.33
Pu-239	3.75 ± 0.19	3.80 ± 0.56	1.21
Sr-90	33.4 ± 1.7	35.0 ± 3.0	4.72
Sr-90	102 ± 5	106 ± 9	3.86
U-natural	0.21 ± 0.01	0.16 ± 0.06	-24.11
U-natural	0.92 ± 0.05	0.97 ± 0.18	5.20

-25% to +50% Bias is termed as acceptable

Proficiency Sample Results ORNL Urine Jan 01 – Dec 03





Proficiency Sample Results

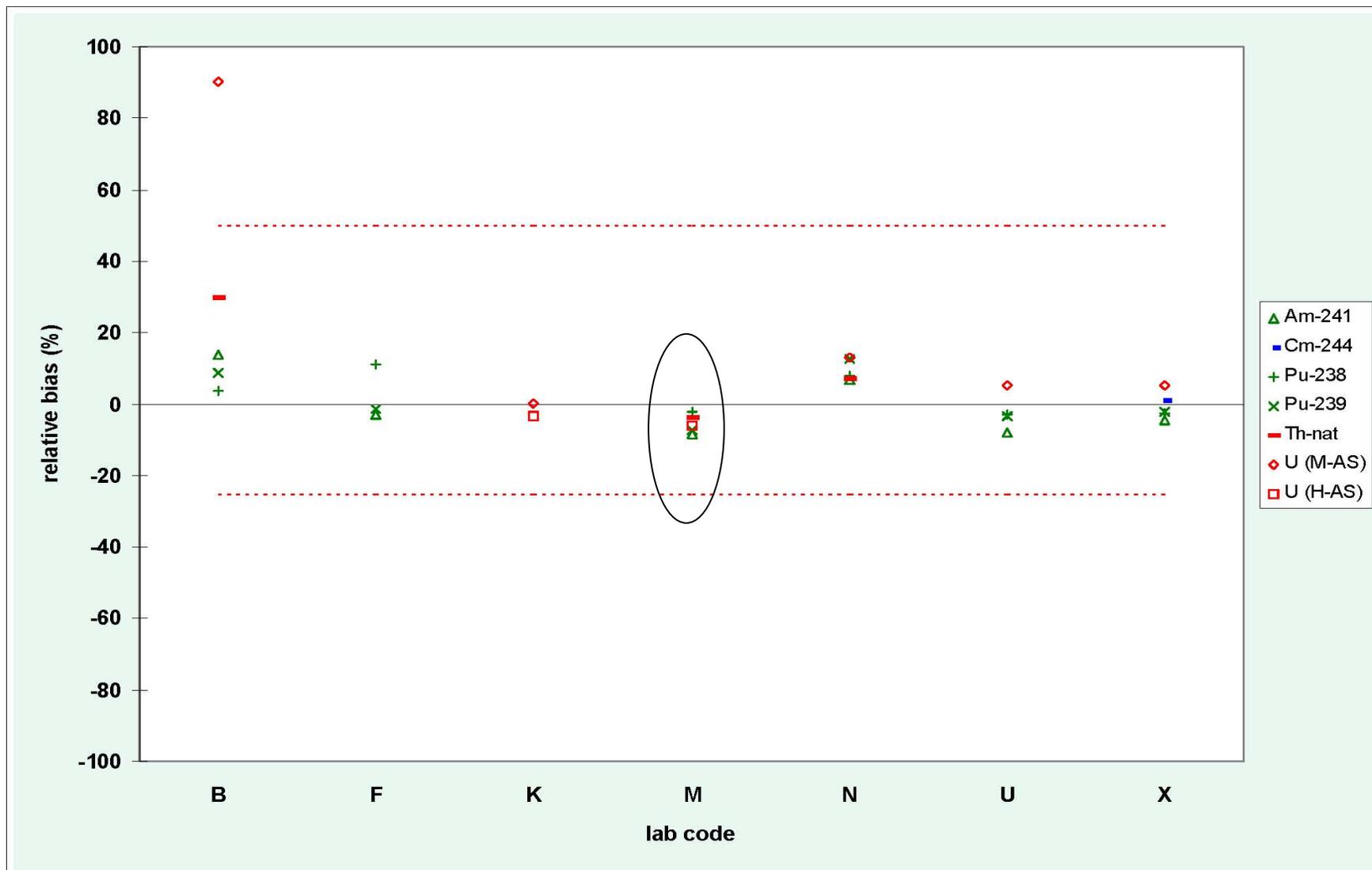
ORNL Fecal Q2 2003

Nuclide	Known	Result	%Bias
Am-241	BLANK	0.00 ± 0.01	na
Am-241	0.92 ± 0.05	1.01 ± 0.15	9.94
Pu-238	BLANK	0.01 ± 0.01	na
Pu-238	0.65 ± 0.03	0.62 ± 0.10	-5.78
Pu-239	BLANK	0.00 ± 0.01	na
Pu-239	0.80 ± 0.04	0.75 ± 0.11	-5.19
Th-natural	10.5 ± 0.5	9.39 ± 1.11	-10.55
U-natural	14.4 ± 0.7	13.2 ± 1.4	-7.91

-25% to +50% Bias is termed as acceptable

Proficiency Sample Results

ORNL Fecal Jan 01 – Dec 03





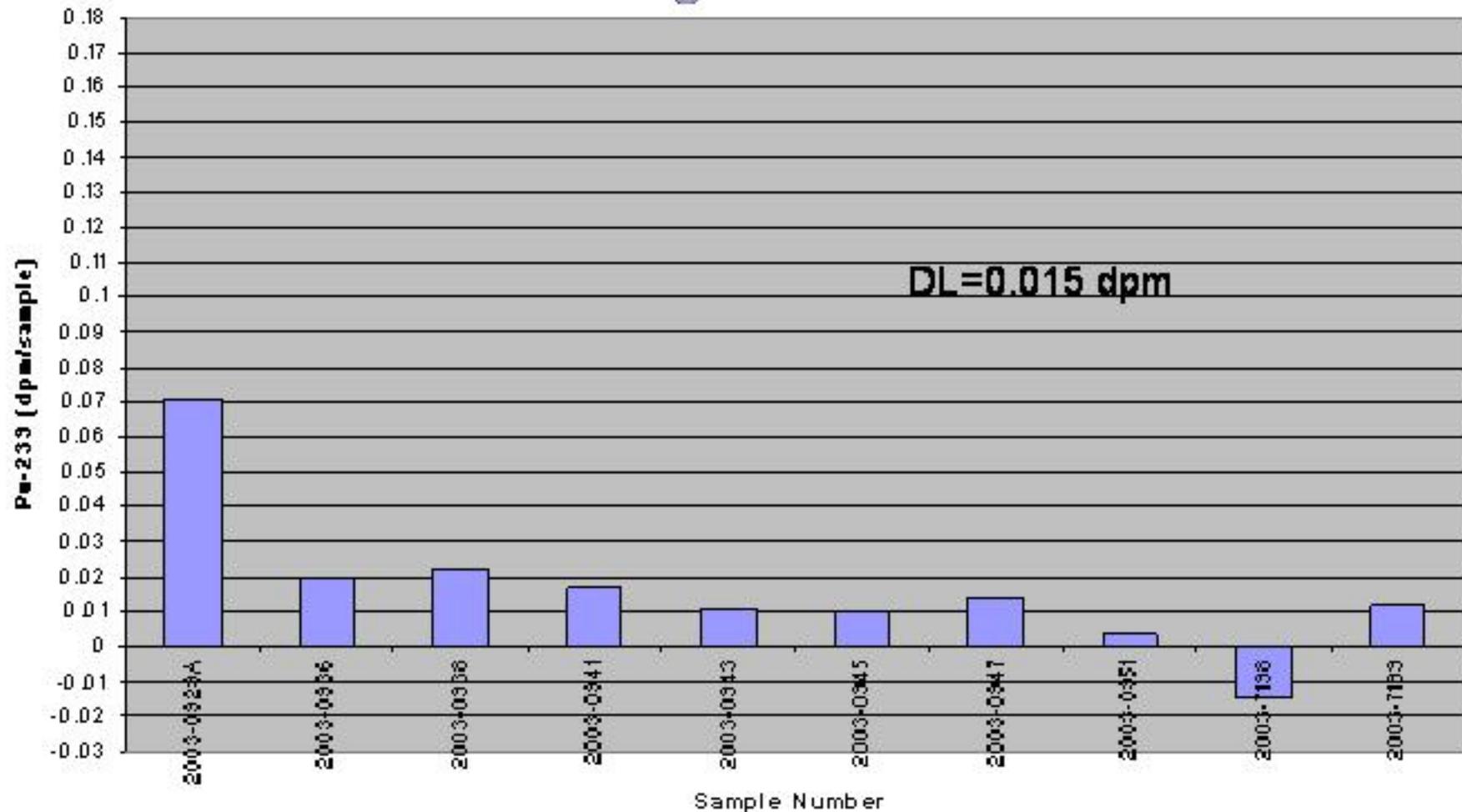
Typical Requests from Internal Dosimetry to Validate Results

- Count again
- Count longer
- Count on a different detector
- Strip plate, further separate, recount
- Analyze another aliquot
- Use a different spike
- Request another sample from employee
- Etc...

Pu-239/240 Results

Group 10022003

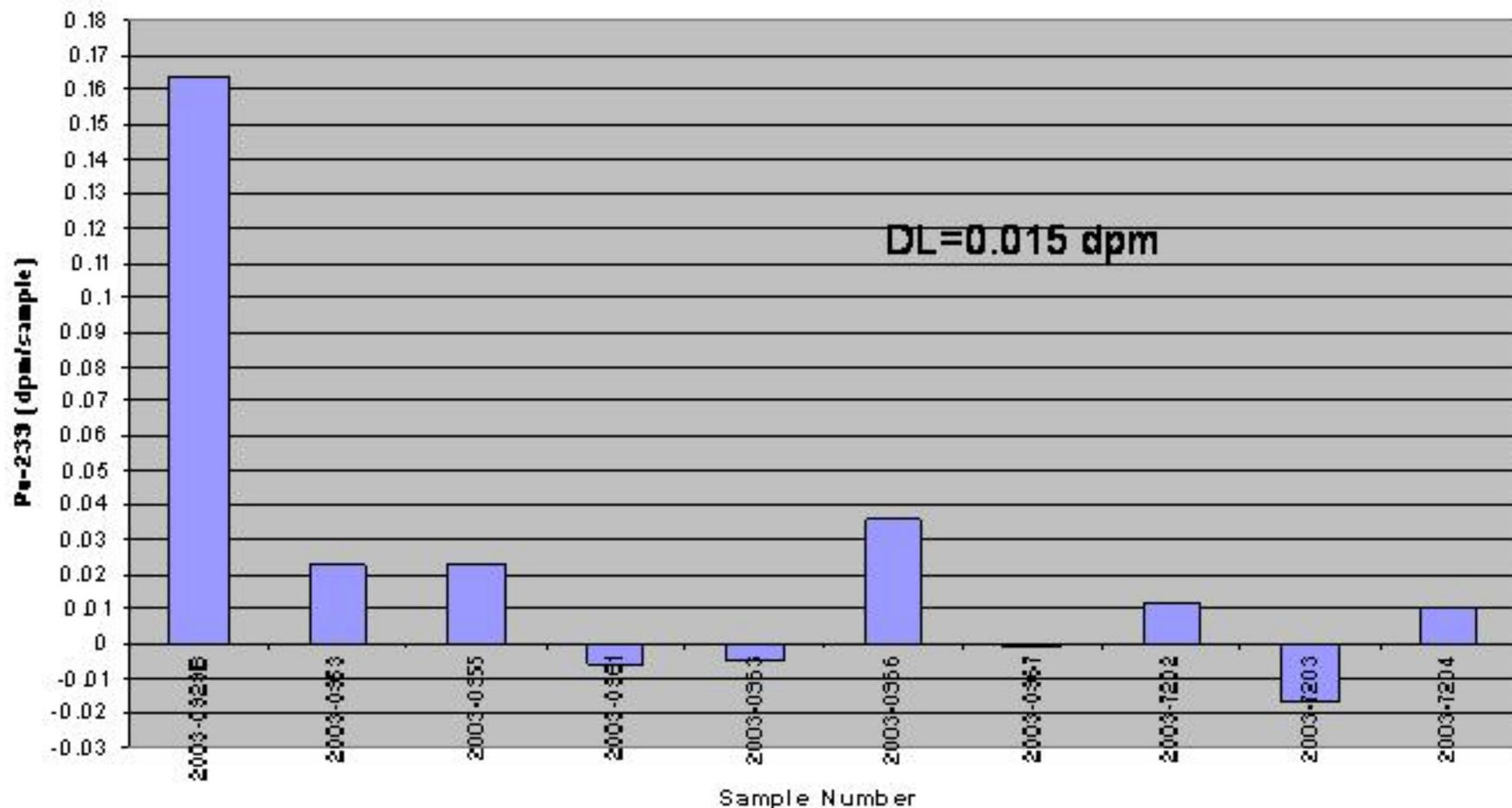
showing 2003-0929A



Pu-239/240 Results

Group 10082003

showing 2003-0929B





Internal Dosimetry and Bioassay

Theresa Davis	Building 200	2-6077
Mike VanDerKarr	Building 202	2-3915
Al Keane	Building 202	2-8546

Our doors are always open.

Stop in for a tour.