



Emergency Method for Radiochemical Separations in Bioassay and Environmental Samples

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An AECOM-led partnership with B&W and AREVA

WIPP LABORATORIES
Carlsbad, NM

WIPP LABORATORIES

CAPABILITIES:

- ALPHA SPECTROSCOPY
- GAS PROPORTIONAL COUNTING
- GAMMA SPECTROSCOPY
- LIQUID SCINTILLATION COUNTING
- ICP-MASS SPECTROSCOPY

MATRICES:

- URINE
- FECES
- AIR FILTERS
- WATERS
- SOILS/SEDIMENTS
- ANIMAL TISSUE
- VEGETATION
- ROCK SALT
- BRINE
- OTHERS, AS REQUESTED

PERFORMANCE EVALUATION:

- DOELAP
- NRIP ROUTINE/EMERGENCY
- MAPEP
- EPA

History of NRIP Emergency Samples at WIPP Labs

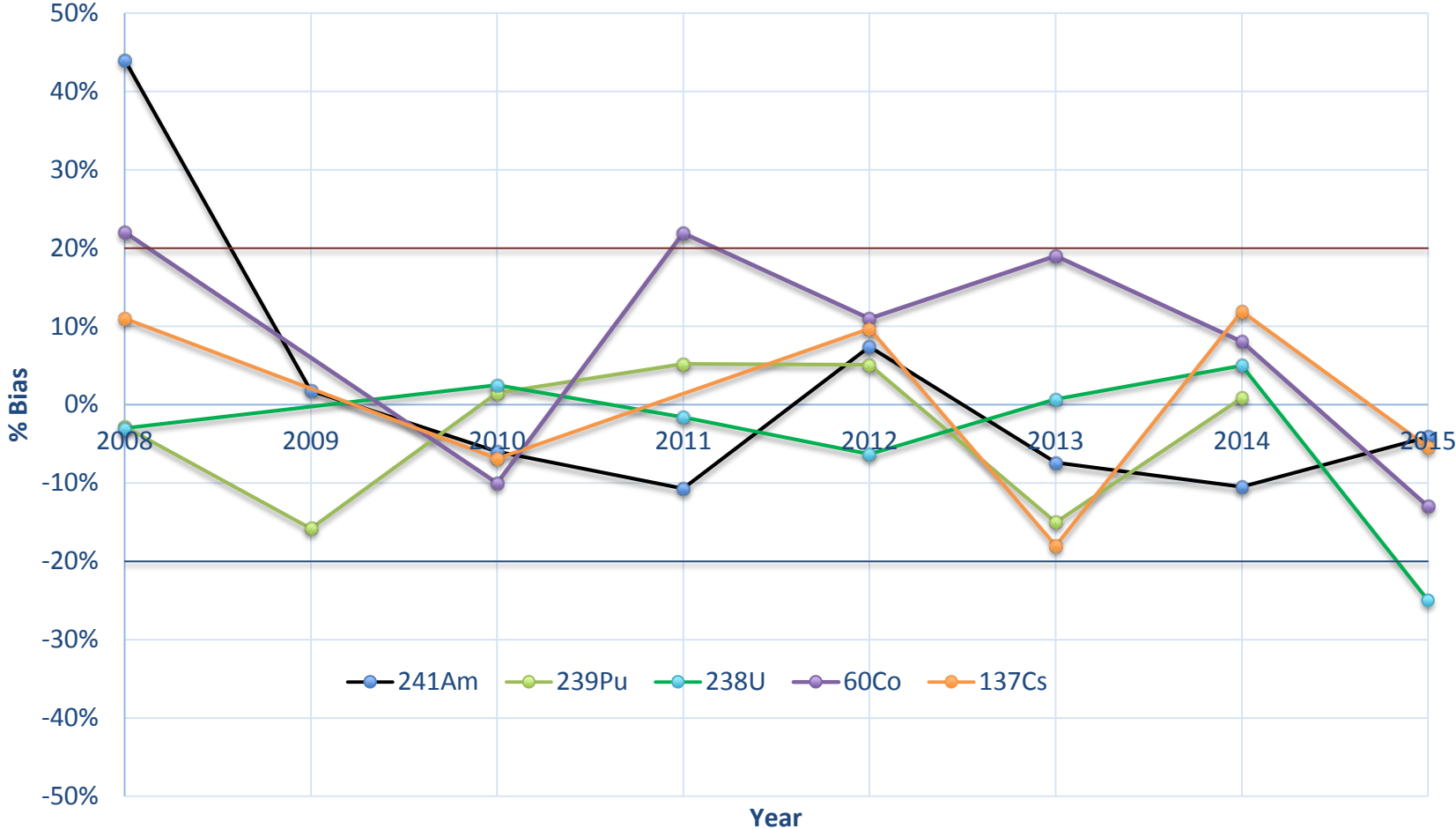
Year	Matrix	Measured Analyses	TAT (Destructive)	TAT (Non-Destructive)
2004	Water/Filters	Co-60, Ba-133, Cs-137, Eu-152	NA	<8 hours
2005	Filters	Mn-54, Zn-65, Cs-137, Gross Alpha & Beta	NA	<8 hours
2006	Soil	U-238, U-234, Pu-238, Pu-239+240, Am-241, Cs-137, Zn-65, Mn-54, Pu-238, Pu-239, Am-241, U-238, U-234, Th-230, Sr-90, Co-60, Ba-133, Cs-237, Eu-152	24 hours	NA
2007	Synthetic Urine	Am-241, Cm-243, Pu-238, Pu-240, U-238, U-234, Sr-90, Co-60, Co-57, Cs-137	9.8 hours	4 hours
2008	Synthetic Urine/ Synthetic Feces	Am-241, Pu-238, Pu-240, Sr-90, Co-60, Co-57, Cs-137	8.7 hours (urine) 10.5 hours (Feces)	4.2 hours
2009	Synthetic Urine/ Synthetic Feces	Am-241, Pu-238, Pu-240, Sr-90, Co-60, Co-57, Cs-137	7.5 hours (urine) 11.9 hours (Feces)	3.7 hours

History of Emergency Samples at WIPP Labs Continued

Year	Matrix	Measured Analyses	TAT (Destructive)	TAT (Non-Destructive)
2010	Synthetic Urine/ Synthetic Feces	Cm-243, Am-241, Pu-238, Pu-240, U-238, U-235, U-234, Sr-90, Co-60, Co-57, Cs-237	7.9 hours (urine) 9.8 hours (feces)	4.9 hours
2011	Synthetic Feces	Am-241, Pu-238, Pu-240, U-238, U-235, U-234, Sr-90, Mn-54, Co-60, Cs-134	8 hours	8 hours
2012	Synthetic Feces	Cm-243, Am-241, Pu-238, Pu-240, U-238, U-235, U-234, Sr-90, Co-60, Cs-137	8 hours	3.8 hours
2013	Synthetic Feces	Am-241, Pu-240, Pu-238, U-238, U-234, Sr-90, Cs-134, Co-60	7.7 hours	6.0 hours
2014	Synthetic Feces	Am-241, Pu-240, Pu-238, U-238, U-234, Sr-90, Cs-137, Co-60	8.1 hours	5.1 hours
2015	Synthetic Feces/ Synthetic Urine	Am-241, Pu-240, Pu-238, U-238, U-234, Sr-90, Cs-137, Co-60	7.8 hours (urine)	2.8 hours (urine)
			7.9 hours (feces)	4.0 hours (Feces)

History of Emergency Samples at WIPP Labs

Emergency Exercise Bias (Feces)

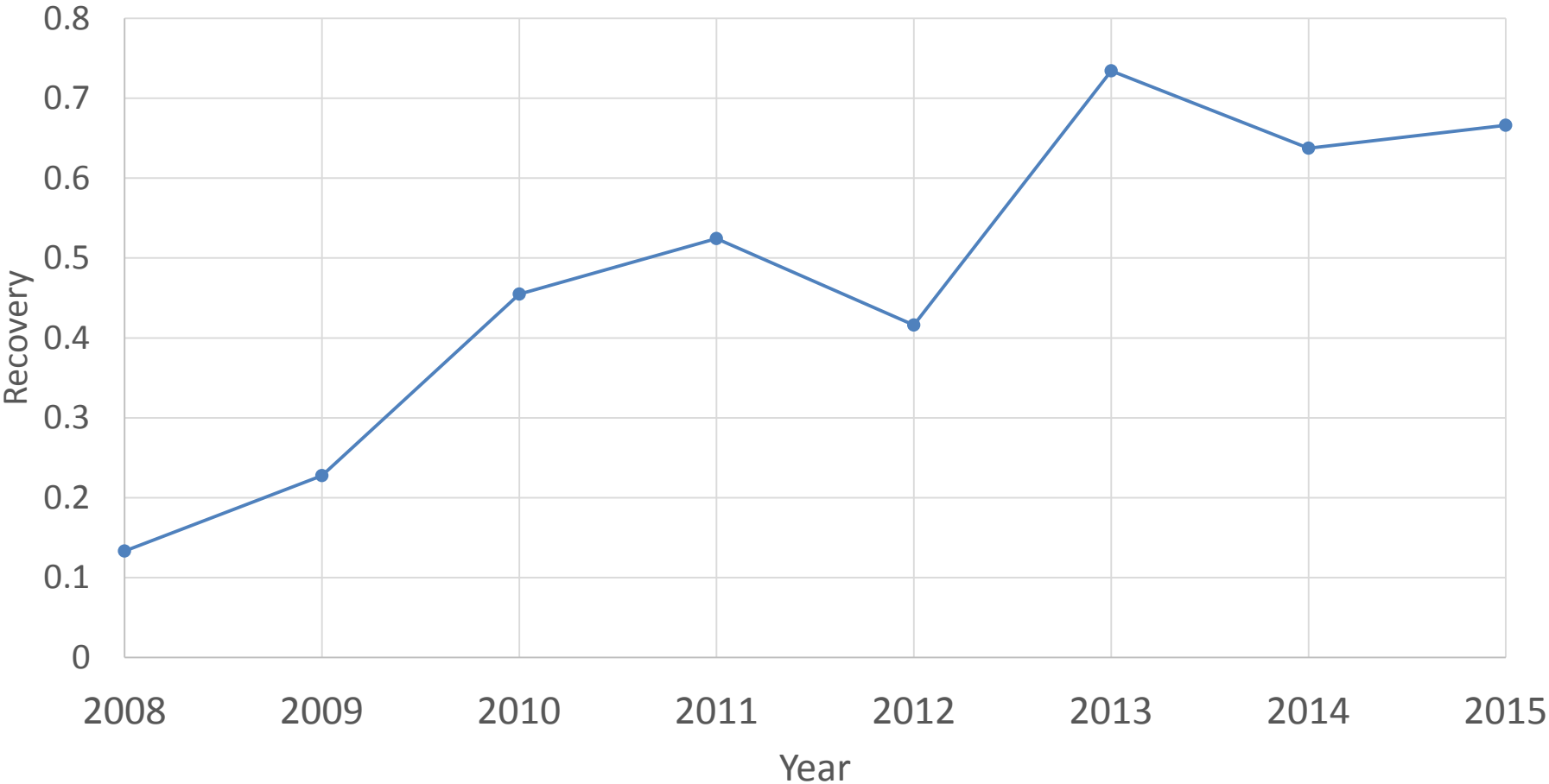


Standard Procedure for Emergency Samples at WIPP Labs

1. Aliquot tracers prior to arrival
2. Rapid digestion methods
3. Standard Eichrom Vacuum Box Methods
 - A. TEVA/TRU/SR column cartridge stack
 - B. Double SR resin to increase Sr recoveries
4. NdF₃ micro-precipitation, utilizing vacuum box to filter multiple samples simultaneously
5. Improved back end data V+V with implementation of automated LIMS

History of Emergency Samples at WIPP Labs

Sr Recoveries For Emergency Feces Samples



Assembly Line Processing Sample Loading



- OXIDATION STATE
ADJUSTMENT

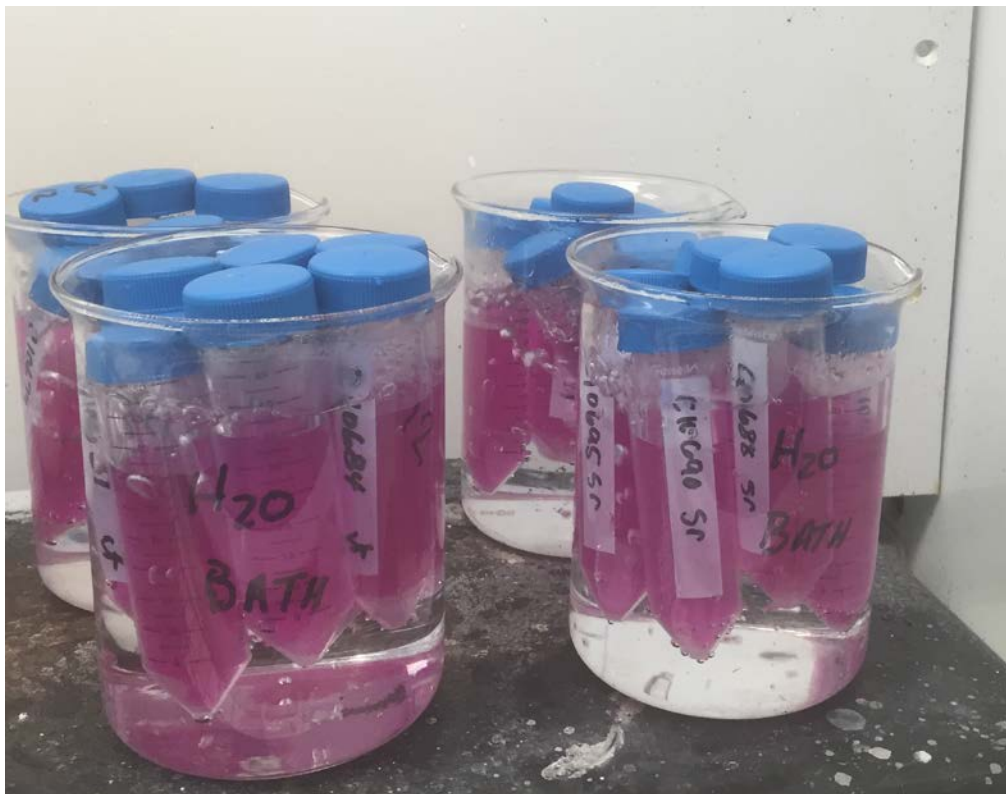
- 1.5M SULFAMIC ACID
- 1.5M ASCORBIC ACID
- 5mg/mL FERRIC NITRATE
- 3.5M SODIUM NITRITE

- LOAD COLUMNS

Assembly Line Processing Split Columns



Assembly Line Processing SrCO₃ Precipitation



- Double SR resin cartridge for improved Sr recoveries

Assembly Line Processing Rapid Micro-Filtration

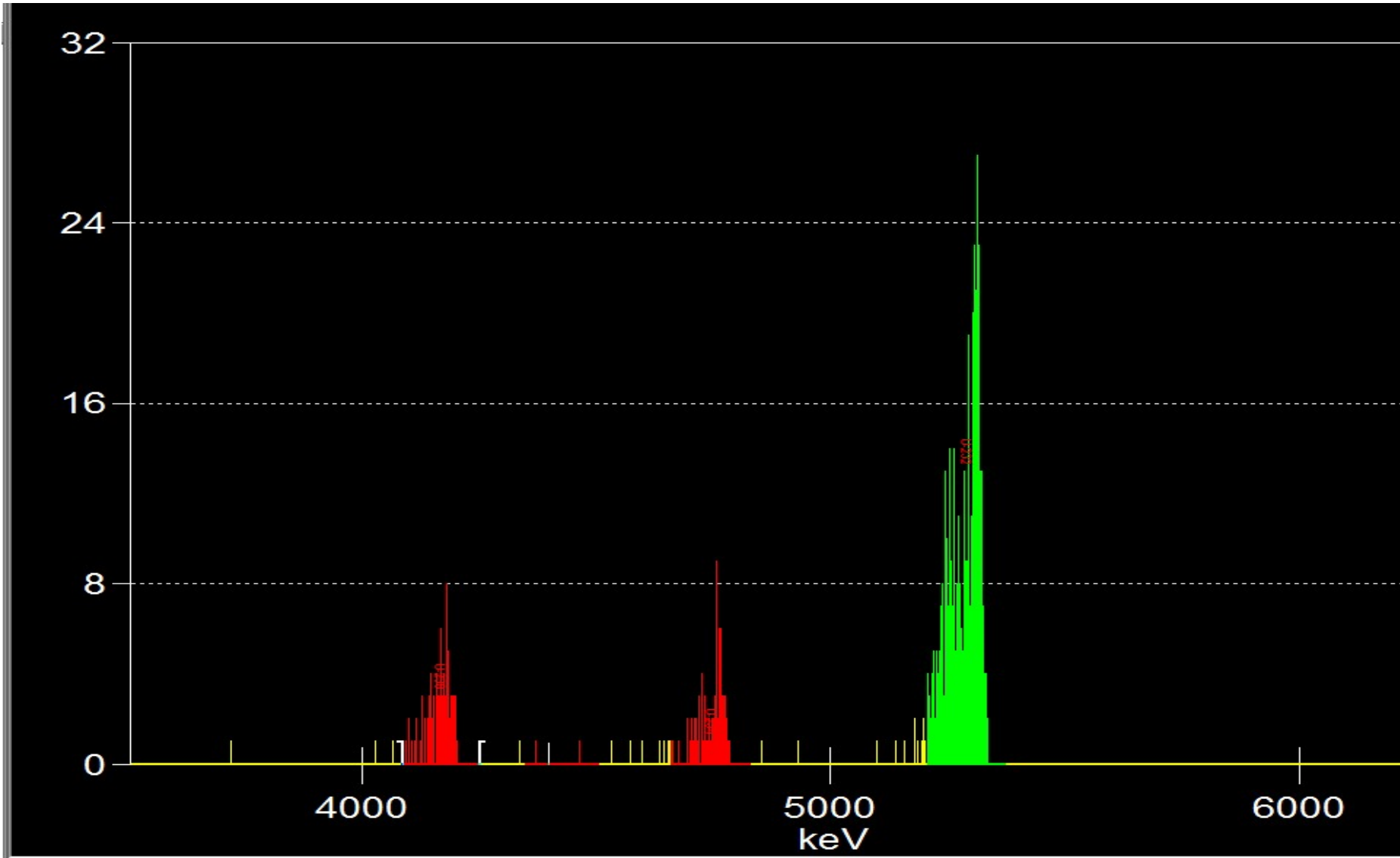


TIME BREAKDOWN

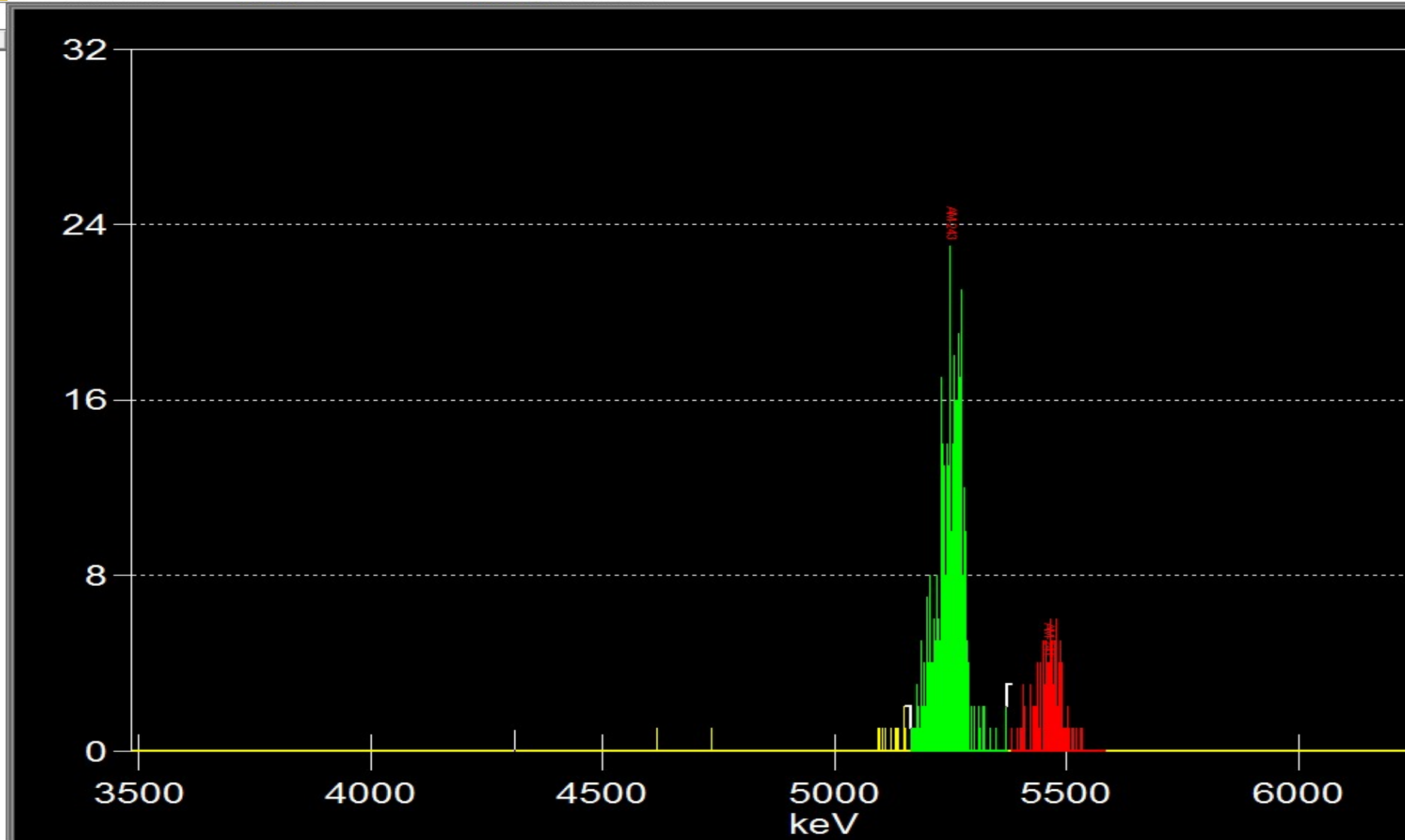
Fecal

	Pu	Am	U	Sr	Gamma
Column Prep	150	150	150	150	150
Separations	120	100	115	100	NA
Post Columning	10	10	10	60	NA
Mounting	20	20	20	30	NA
Count Time	60	60	60	30	30
Processing	60	60	60	40	20
V+V / Reporting	46	55	45	45	38
Total (min)	466	455	455	455	238
Total (Hours)	7.92	7.58	7.58	7.58	3.98
NIST Reporting Time (hrs)	7.92	7.58	7.58	7.58	3.98

URANIUM SPECTRUM



AMERICIUM SPECTRUM



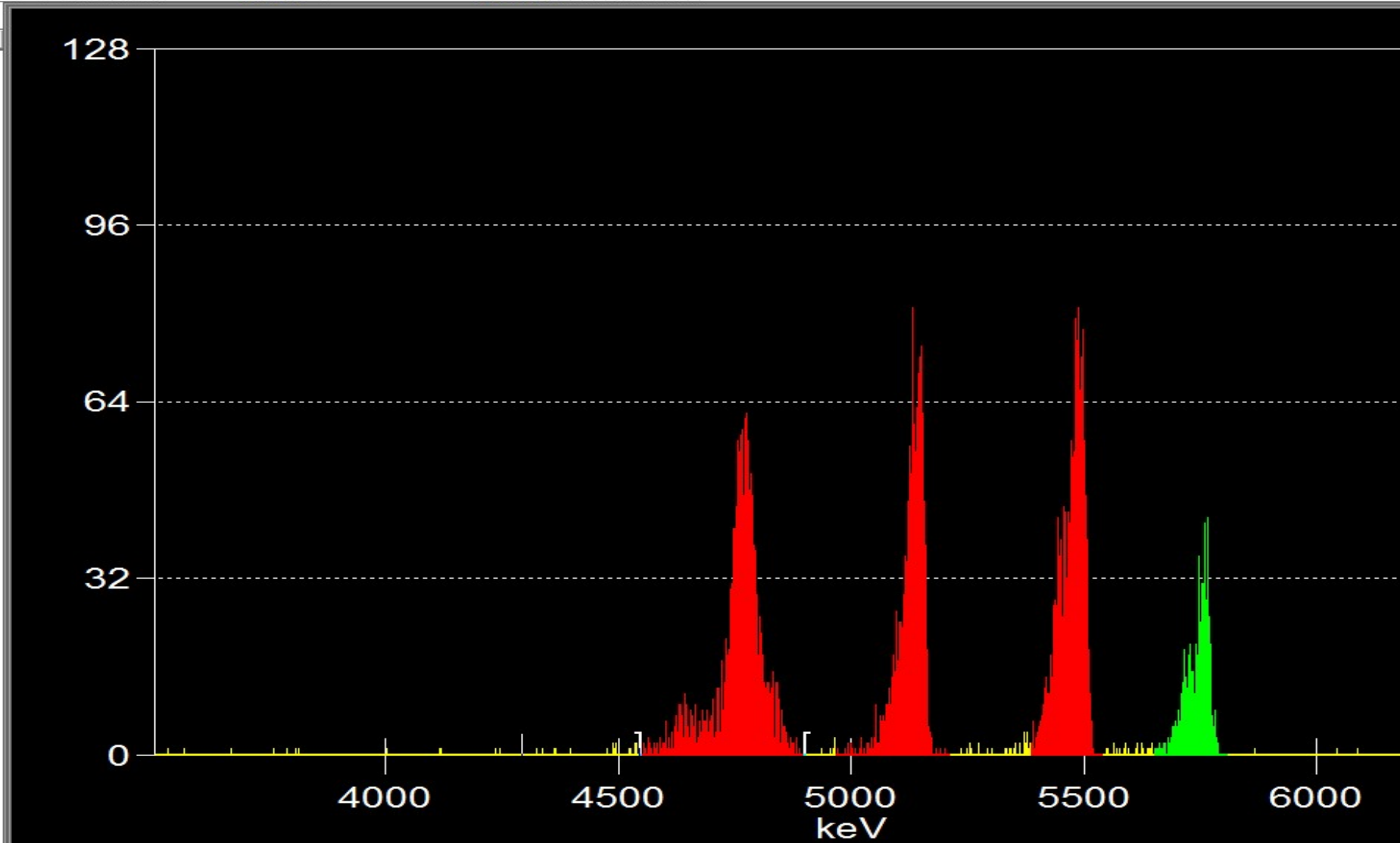
PLUTONIUM SPECTRUM

LESSONS LEARNED!



PLUTONIUM SPECTRUM

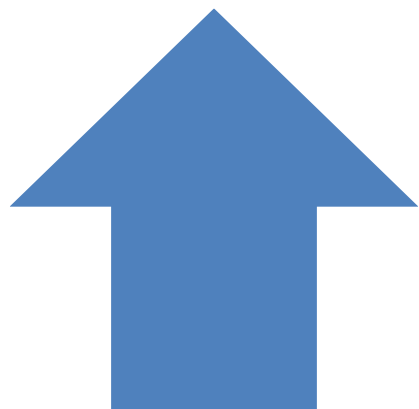
Normal TAT Urine



CURRENT & FUTURE IMPROVEMENTS

1. INCREASE (D)COUNT TIME TO DECREASE MDA 30 MINUTES TO 60 MINUTES
2. INCREASE COLUMN FLOW RATES, CUT SEPERATION TIME. PLAN TO CONDUCT FRACTIONATION STUDIES OF ELUTION PATTERNS
3. USING PU 236 TRACER FOR PU EMERGENCY SAMPLES
4. INCREASE (D) Sr RECOVERIES FOR MORE ACCURATE MEASUREMENTS DOUBLING CARTRIDGE
5. UTILIZATION OF LSA TO REDUCE INITIAL BACKGROUND
6. INCORPORATE LIMS SYSTEM TO STREAMLINE DATA PROCESSING

Application of Emergency Methods to Routine samples



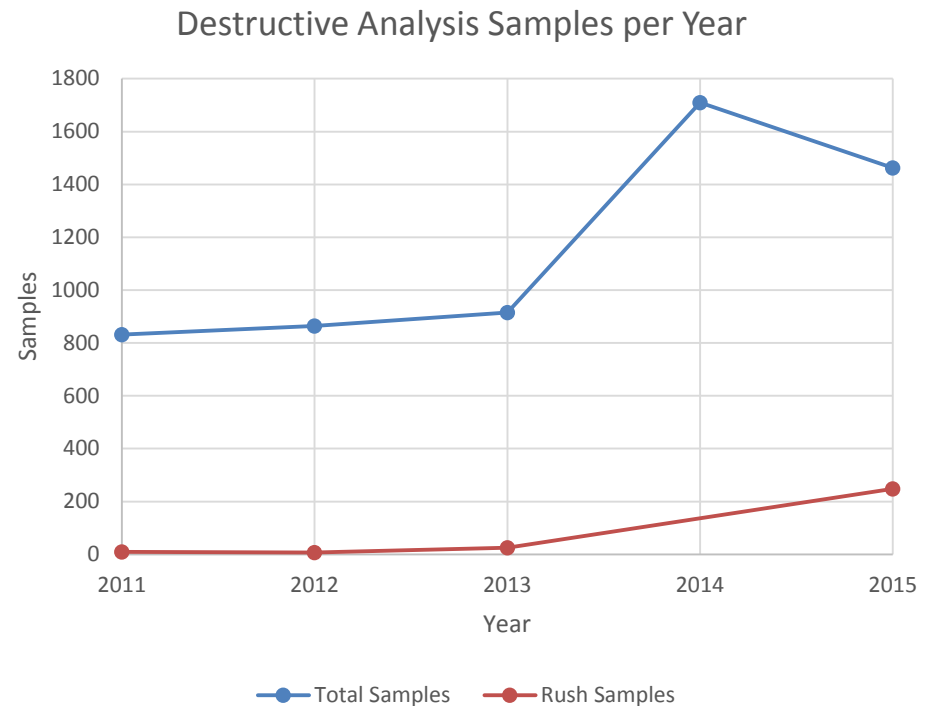
More
Samples



Less
Time

APPLICATION TO WIPP LABORATORIES

Year	Destructive Analysis Samples	Rush Sample Analyses
2011	832	9
2012	864	7
2013	915	25
2014	1710	N/A
2015	1170*	198*



*thru 10-1-15

- “New Normal” at WIPP Laboratories
- Apply Emergency methods to “every day” samples

New Normal

•Bioassay Monitoring Program

- ✓ 30 samples/quarter every year prior to February 2014
- ✓ 197 bioassay samples analyzed in 2014 for Pu and Am
- ✓ 213 samples analyzed in 2014 for Pu, Am, Sr, and Cs-137
- ✓ >120 samples/quarter since initial recovery requiring Pu, Am, Sr and Cs-137



New Normal

Standard Procedure for Rush Brine Samples at WIPP Labs

Communicate with Client
(midday)



Aliquot tracers prior to arrival



Upon arrival (3:30PM) - Sample
aliquot, precipitate, spin, prep
for columns

New Normal

Standard Procedure for Rush Brine Samples at WIPP Labs

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graph TD; A[Separate through TEVA/TRU Stack, NdF3 micro-precipitation (completed by 12:00PM day after arrival)] --> B[1000 minute overnight alpha count]; B --> C[Data reported to client (12:00 PM 2nd day) <48 hours after receipt];
```

Separate through TEVA/TRU Stack,
NdF₃ micro-precipitation (completed
by 12:00PM day after arrival)

1000 minute overnight alpha count

Data reported to client (12:00 PM
2nd day) <48 hours after receipt

APPLICATION TO WIPP LABORATORIES

Rapid Sample Processing = Quality Data Consistently!

Analyte	Average Bioassay Tracer Recovery (2015)
Pu	95%
Am	80%
Sr	70%
U	85%

APPLICATION TO WIPP LABORATORIES

Rapid Sample Processing = Quality Data Consistently!

MAPEP Series 32 Biases				
Analyte	Soil (%)	Water (%)	Vegetation (%)	Air Filter (%)
^{238}Pu	-0.7	Sensitivity (A)	-0.9	False Pos. (A)
^{239}Pu	-2.0	-4.9	-3.4	-0.7
^{241}Am	-1.3	-10.4	-6.5	-7.5
^{90}Sr	-9.2	-2.2	0.0	-0.7
^{234}U	1.0	-3.4	6.0	-8.4
^{238}U	-5.0	-2.9	-10.2	-10.2

Conclusions

1. Trust the Data
 - Years of NRIP emergency exercise data show that the process works
 - Value in performance evaluations
2. Increased Quantity \neq Decreased Quality
3. Adapt to the situation

ACKNOWLEDGEMENTS

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