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EICHROM TECHNOLOGIES

New Applications and Services

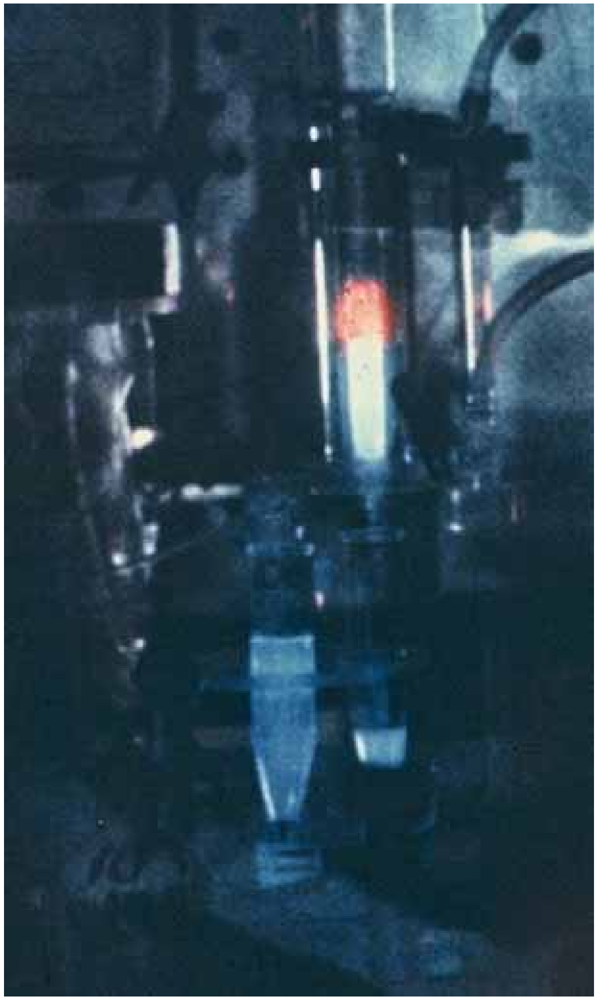
Terence O’Brien
7 February 2017
Eichrom Workshop / User’s Group Meeting
At the 62 Radiobioassay and Radiochemical Measurements Conference

Topics to be covered our New Applications and Services

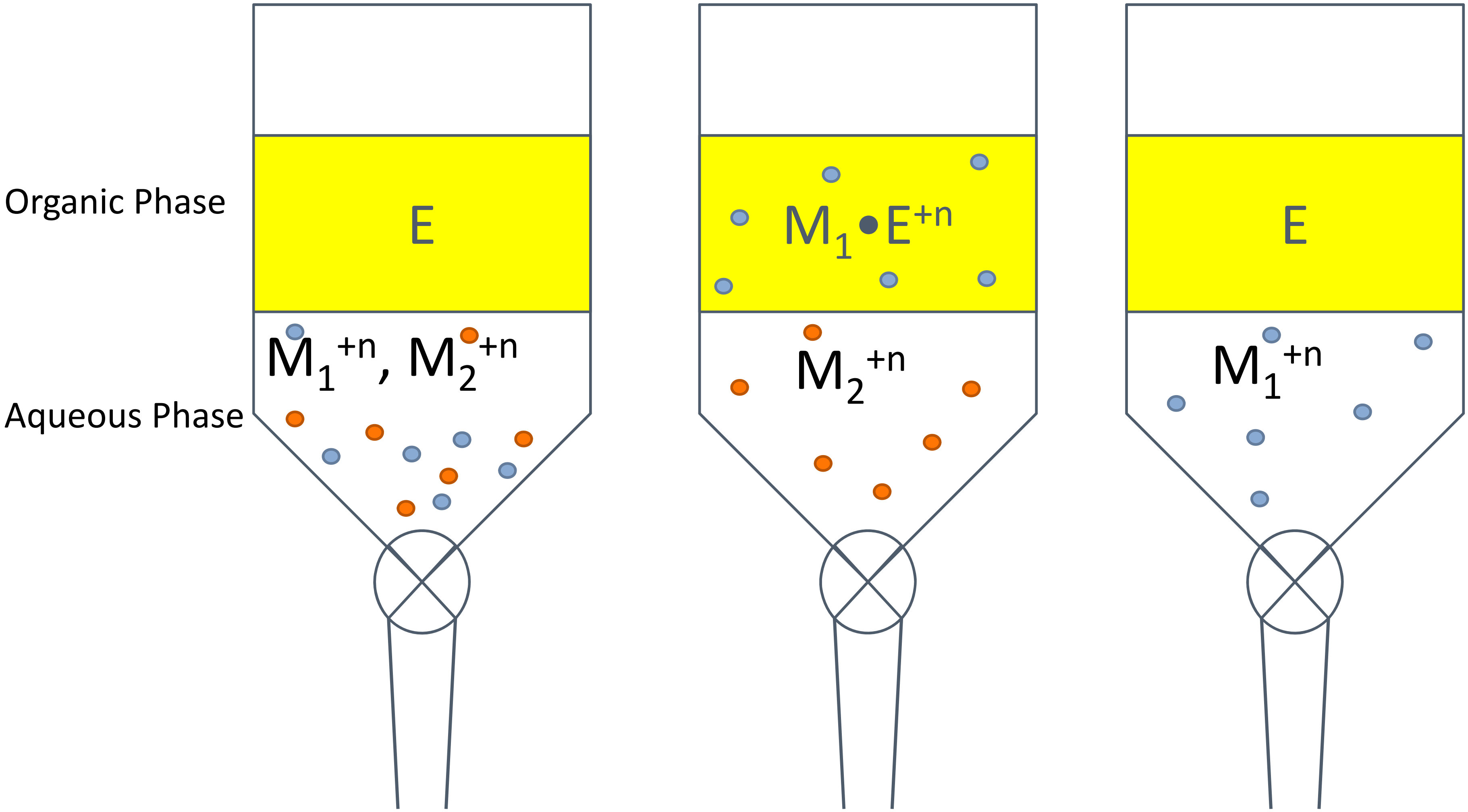
- Overview of Currently Published
  - 2014 Eichrom Methods Revisions
  - 2014 Application Notes
  - 2016 Application Notes
- Training Services
- RadVision$^{3D®}$

Eichrom Technologies

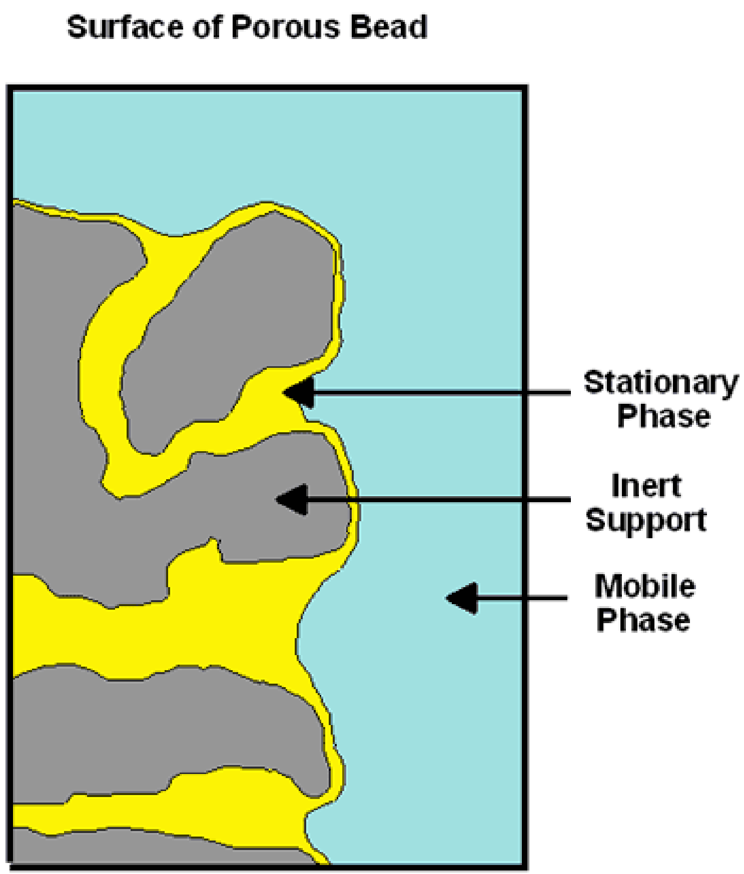
- Extraction Chromatographic Resins were developed at Argonne National Laboratory.
- Eichrom was founded in 1990 to commercially develop these resins.
- In 2015 Eichrom Celebrated our 25th Anniversary



Basis of Resin Technology – Solvent Extraction



Extraction Chromatographic Resin



Inert support =

Macroporous Acrylic Resin

Example Stationary Phases

- **Crown Ether (Sr)**
- **CMPO (TRU)**
- **DAAP (UTEVA)**

Eichrom Methods:

- First Introduced in the 1990s
- Separation of radioactive elements from water samples

Title	Reference
Strontium in Water	SRW 01
Nickel in Water	NIW 01
Iron in Water	FEW 01
Radium in Water	RAW 01
Technetium in Water	TCW 02
Radium in Water	RAW 01
Uranium in Urine	ACU 01
Am, Pu and U in Urine	ACU 02
Lead in Soil	SRS 01

Original Eichrom Method (1990s – 2000s)

1. Scope
2. Summary of Method
3. Significance of Use
4. Interferences
5. Apparatus
6. Reagents
7. Procedure (7.1, 7.2, 7.3, 7.4 …)
8. Calculations
9. Validation Data
10. References

Advantages:

Procedures are very detailed and multi-stepped

Calculations and validations included.

Disadvantages:

Many pages 10-20+

Slight changes in chemistry between versions.

Many Column Only:

Updated Eichrom Method 2014

1. Scope
2. Summary of Method
3. Significance of Use
4. Interferences
5. Apparatus
6. Reagents
7. Procedure (7.1, 7.2, 7.3, 7.4 …)
8. Calculations
9. Validation Data
10. References

Advantages:
Procedures are very detailed and multi-stepped

Calculations and validations included.

Flow Charts Included

Chemistry harmonized

Cartridge and Column options

Eichrom Methods

Analytical Procedure

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AMERICIUM, NEPTUNIUM, PLUTONIUM, THORIUM, CURIUM, AND URANIUM IN WATER

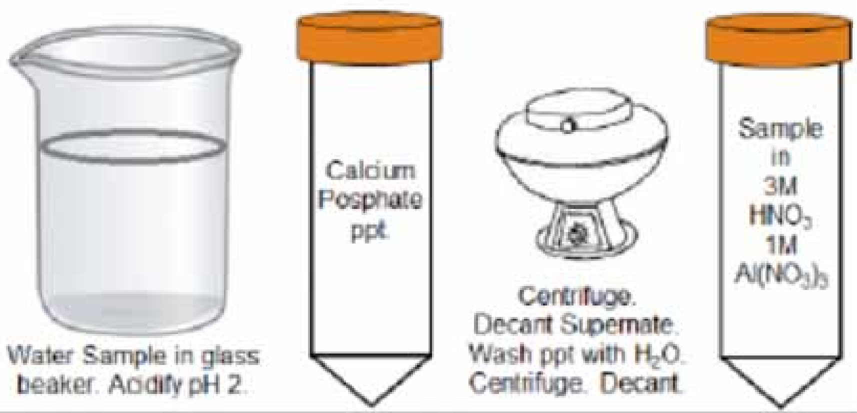
(WITH VACUUM BOX SYSTEM)

1. SCOPE

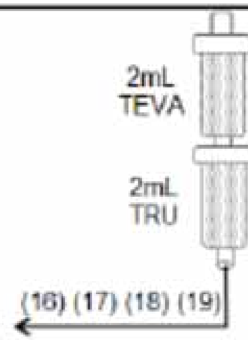
1.1. This is a method for the separation of americium, neptunium, plutonium, thorium, curium and uranium in water. After completing this method, source preparation for measurement of actinides by alpha spectrometry is performed by electrolytic deposition onto stainless steel planchets (Eichrom Method SPA02) or by rare earth fluoride microprecipitation onto polypropylene filters (Eichrom Method SPA01).

Flow charts for easy method application

1) Aliquot up to 1000mL of water into glass beaker.
2) Add 5mL concentrated HNO_3 and add yield tracers.
3) Add 1mL of 1.25M $Ca(NO_3)_2$.
4) Heat samples at medium setting for 30-60 minutes.
5) Remove samples from heat.
6) Add 0.75mL of phenolphthalein and 3mL of 3.2M $(NH_4)_2HPO_4$.
7) While stirring sample, slowly add conc. NH_4OH until reaching pH 9.
8) Cool to room temperature. Allow precipitate to settle or centrifuge.
9) Decant supernate and discard as waste.
10) Transfer precipitate to centrifuge tube with DI water.
11) Centrifuge ~10minutes at 2000rpm. Decant supernate.
12) Add 10mL DI water to ppt. Mix well. Centrifuge. Decant supernate. Dissolve ppt with 5mL conc. HNO_3. Transfer to 100mL beaker.
13) Rinse centrifuge tube with 2-3mL conc. HNO_3. Transfer to 100mL beaker. Evaporate to dryness.
14) Dissolve reside in 16mL 3M HNO_3-1M $Al(NO_3)_3$. Add 1mL 1.5M Sulfamic Acid, 0.5 mL Fe, and 1mL 1M Ascorbic Acid. Swirl to mix. Wait 3-5 minutes.
15) Add 1mL 3.5M $NaNO_2$. Swirl to mix.

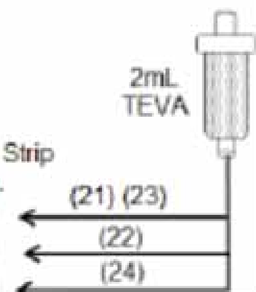


16) Precondition TEVA-TRU with 5mL 3M HNO_3.
17) Load sample onto TEVA-TRU. Allow liquid to drain. TEVA retaines U. TRU retains Am and Pu.
18) Rinse sample tube with 5mL 3M HNO_3. Add rinse to TEVA-TRU. Allow liquid to drain.
19) Rinse TEVA-TRU with 5mL 3M HNO_3. Allow liquid to drain.
20) Separate TEVA and TRU cartridges.



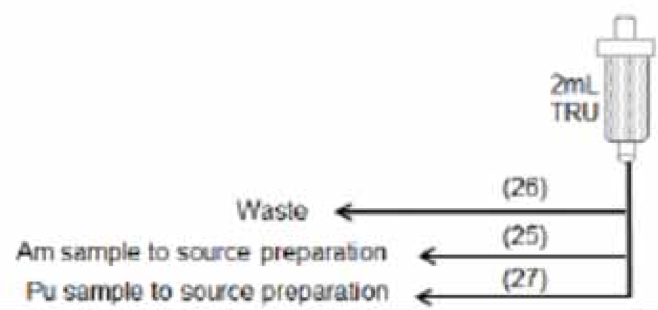
Waste

21) Rinse TEVA column with 10mL 3M HNO_3.
22) Place clean centrifuge tube below TEVA. Strip Th with 15mL 9M HCl.
23) Rinse TEVA column with 20mL 5M HCl-0.05M oxalic acid. Discard to Waste.
24) Place clean centrifuge tube below each TEVA. Strip Pu-Np with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.



Waste
Th sample to source preparation
Pu-Np sample to source preparation

25) Place clean centrifuge tubes below TRU. Strip Am with 15mL of 4M HCl.
26) Rinse TRU with 12mL 4M HCl-0.1M HF. Discard as waste.
27) Place a clean centrifgue tube below each cartridge. Strip U with 15mL 0.1M ammonium bioxalate.



Some of the 32 Published Methods at eichrom.com

Reference	Title	Column	VBS
ACS 07	U in Soil	X	X
ACW 03	Am-Pu-U in Water	X	X
ACW 08	Th-Np in Water	X	X
ACW 11	Alpha Total Water	X	
ACW 17	Am-Pu-U-Np-Th-Sr in Water		X
PBW 01	Pb -Po in Water	X	X
H3W 02	H3 in Water	X	
SRW 01	Sr in Water	X	X
TCS 01	Tc in Soil	X	X
TCU 01	Tc in Urine (disc)	X	
TCW 01	Tc in Water	X	X
TCW 02	Tc in Water (disc)	X	

However there is a problem.

2014 Eichrom Application Notes

The goal of Eichrom application notes is to:

- Add matrices
- Add rugged sample preparation
- Add rapid techniques
- Add cutting edge separation techniques
- Shorten what the analyst needed to have on hand

Example of Application Note Layout

AN-1413-10

Rapid Determination of Actinides in Emergency Water Samples

Summary of Method Uranium, Plutonium and Americium-Curium are separated and concentrated from up to 400mL water samples using calcium phosphate precipitation. The precipitate is dissolved in nitric acid and aluminum nitrate. Actinides are separated from matrix impurities and potentially interfering radionuclides in the sample using 2mL cartridges of Eichrom TEVA and TRU Resins. Actinides are measured by alpha spectrometry following source preparation by cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical yields are determined by recovery of ^{232}U, ^{243}Am, and ^{242}Pu (or ^{236}Pu, if measuring ^{237}Np) tracers. Typical chemical recoveries are >90%. A single operator can complete the separation method for batches of 12-24 samples in as little as 4-5 hours.

Example of Application Note Layout (Reagents)

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Deionized Water
Iron Carrier (50mg/mL)
Cerium Carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
10% (w:w) $TiCl_3$
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Sulfamic Acid
Ascorbic Acid
Sodium Nitrite
Denatured Ethanol

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Example of Application Note Layout (Equipment)

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)
Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)
50mL and 250mL Centrifuge Tubes
Centrifuge
Analytical Balance
Alpha Spectrometry System
Vacuum Pump

Sample Preparation

400mL water + Tracers,
2mL 1.25M $Ca(NO_3)_3$,
5mL 3.2 M $(NH_4)_2HPO_4$.
Mix Well

↓

Adjust to pH 10 with NH_4OH.
Mix Well.

↓

Centrifuge 3500 rpm, 10 min. → Decant Supernate To Waste

↓

Dissolve precipitate in 8mL 6M HNO_3 and 8mL 2M $Al(NO_3)_3$.
Cool to room temperature.

↓

Adjust valence states of actinides by adding the following reagents in the order listed (mix between additions):

0.5mL 1.5M Sulfamic acid, 10uL 50mg/mL Fe carrier, 1.25mL 1M Ascorbic acid, 1mL 3.5M $NaNO_2$, and 1.5mL 70% HNO_3.

↓

Load Solution to Resin Separation

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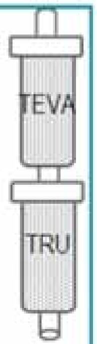
Example of Application Note (Separation)

(1) Precondition stacked 2mL TEVA-TRU with 10mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 6M HNO_3. Add tube rinse to cartridges.**



(4) Rinse cartridges with 5mL 3M HNO_3.

(5) Separate TEVA and TRU cartridges.

(6) Rinse TEVA cartridge with:

- -15mL 3M HNO_3
- -20mL 9M HCl(remove Th)
- -5mL 3M HNO_3

(7) Strip Pu(Np) from TEVA with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Strip Am/Cm from TRU with 15mL 4M HCl. Dilute to 30mL prior to CeF_3 ppt.

(9) Rinse TRU with 15mL 4M HCl-0.2M HF. (Th removal)

(10) Strip U from TRU with 15mL 0.1M ammonium bioxalate.

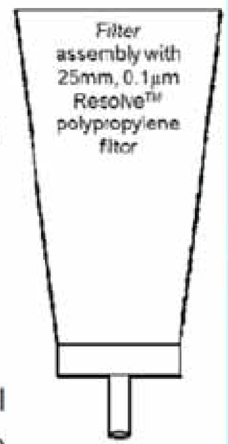
(11) Add 0.5mL 30% H_2O_2 to Pu and 0.2mL 30% H_2O_2 to Am/Cm samples for additional U decon. during CeF_3 ppt.

(12) Add 0.5mL of 10% $TiCl_3$ to each U sample for CeF_3 ppt.

(13) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.



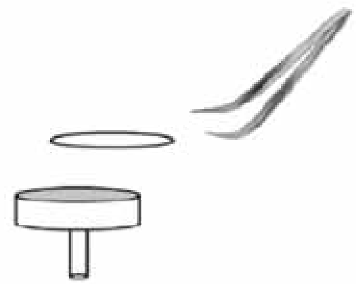
(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.

(18) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

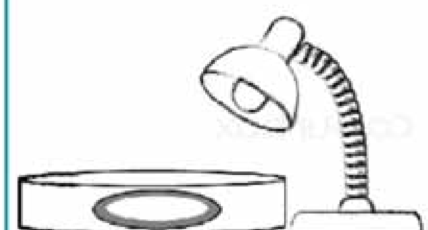
(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure actinides by alpha spectrometry.



Example of Application Note (Results)

Method Performance 10-40L Spike Seawater Samples

Sample Volume, L	% Recovery Y carrier	^{90}Sr (mBq/L) Reference	^{90}Sr (mBq/L) Measured	% Bias
10	85.5	296	310	4.7
20	89.2	28.2	28.1	-0.4
30	72.3	18.8	18.5	-1.6
40	87.6	14.1	13.7	-2.8
40	86.5	14.1	13.9	-1.4

MDA for 40L sample = 0.35 mBq/L for 2 hour count time

MDA for 40L sample = 0.20 mBq/L for 8 hour count time

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey, Daniel R. McAlister, "Rapid determination of ^{90}Sr in seawater samples," *J. Radioanal. Nucl. Chem.*, 303, 709-717 (2015).

Eichrom Method Application Notes

Number	Title
AN-1401	Rapid Determination of ^{226}Ra in Emergency Urine and Water
AN-1402	Rapid Determination of Sr in Emergency Milk Samples
AN-1403	Rapid Determination of Sr in 50g Soil Samples
AN-1404	Rapid Determination of Sr in 1-2 Liter Seawater Samples
AN-1405	Rapid Determination of Sr in Vegetation Samples
AN-1406	Rapid Determination of Actinides in Vegetation Samples
AN-1407	Rapid Determination of Sr in Animal Tissue Samples
AN-1408	Rapid Determination of Actinides in Animal Tissue Samples
AN-1409	Rapid Determination of Sr in Building Materials
AN-1410	Rapid Determination of Sr in Emergency Urine Samples
AN-1411	Rapid Determination of Sr in Emergency Water Samples
AN-1412	Rapid Determination of Actinides in Emergency Urine Samples
AN-1413	Rapid Determination of Actinides in Emergency Water Samples
AN-1414	Rapid Determination of ^{90}Sr in Up to 40 Liter Seawater Samples
AN-1415	Rapid Determination of ^{210}Po in Water Samples
AN-1416	Rapid Determination of Actinides and ^{210}Po in Water
AN-1417	Rapid Determination of $^{226/228}Ra$ in Water Samples
AN-1418	Rapid Determination of ^{226}Ra in Water Samples
AN-1419	Rapid Determination of ^{226}Ra in Concrete and Brick

Application Notes (AN-2014-####)

	Actinide(s)	Radium	Strontium
Animal Tissue	1408	--	1407
Building Materials	1429, 1432	1419	1409
Food/Vegetation	1406, 1425, 1426, 1427	1422	1402, 1405
Glass Fiber Air Samples	1433	1420	1434
Seawater	1423, 1424	--	1404, 1414
Soils	1430, 1431, 1435, 1436	1421	1403
Urine	1412, 1437, 1438	1401	1410
Water	1413, 1416	1401, 1417, 1418	1411

Others: Po – 1415, 1416

Rapid Determination of Pu, Np, and U in 1-8L Seawater AN-1423-10

Method Performance Pu, Np and U from Seawater

Analyte	Volume, L	Replicates	Tracer	% Tracer Recovery	Analyte(mBq/L) Reference	Analyte(mBq/L) Measured	% Bias
^{239}Pu	2	5	^{236}Pu	91 ± 9	33.8	32.6 ± 1.4	-3.6
^{239}Pu	4	1	^{236}Pu	86	16.9	16.2	-4.1
^{239}Pu	8	2	^{236}Pu	87 ± 3	27.8	27.6 ± 0.5	-0.7
^{237}Np	2	5	^{236}Pu	91 ± 9	17.4	17.7 ± 1.5	1.7
^{237}Np	4	1	^{236}Pu	86	8.7	7.2	-17
^{237}Np	8	2	^{236}Pu	87 ± 3	4.4	4.2 ± 0.4	-4.5
^{238}U	2	5	^{232}U	99 ± 2	51.8	49.3 ± 1.5	-4.8
^{238}U	4	1	^{232}U	86	25.9	25.0	-3.6
^{238}U	8	2	^{232}U	92 ± 5	96.3	94 ± 3	-2.4

16 hour count times

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey, Daniel R. McAlister, "Rapid determination of actinides in seawater samples," *J. Radioanal. Nucl. Chem., 300(3), 1175-1189* (2014).

Rapid Determination of $^{226/228}Ra$ in Water Samples AN-1417-10

Method Performance $^{226/228}Ra$ in Water

Sample	Volume Liters	Replicates	^{133}Ba Tracer % Recovery	% Recovery ^{226}Ra	% Recovery ^{228}Ra
River Water	1.5	3	101 $\pm$ 5	103 $\pm$ 1	103 $\pm$ 7
Ground Water	1.0	5	95 $\pm$ 4	104 $\pm$ 1	102 $\pm$ 8

1040pCi ^{133}Ba, 5.0pCi ^{226}Ra, 20pCi ^{228}Ra

Rapid Determination of Sr in Animal Tissue Samples AN-1407-10

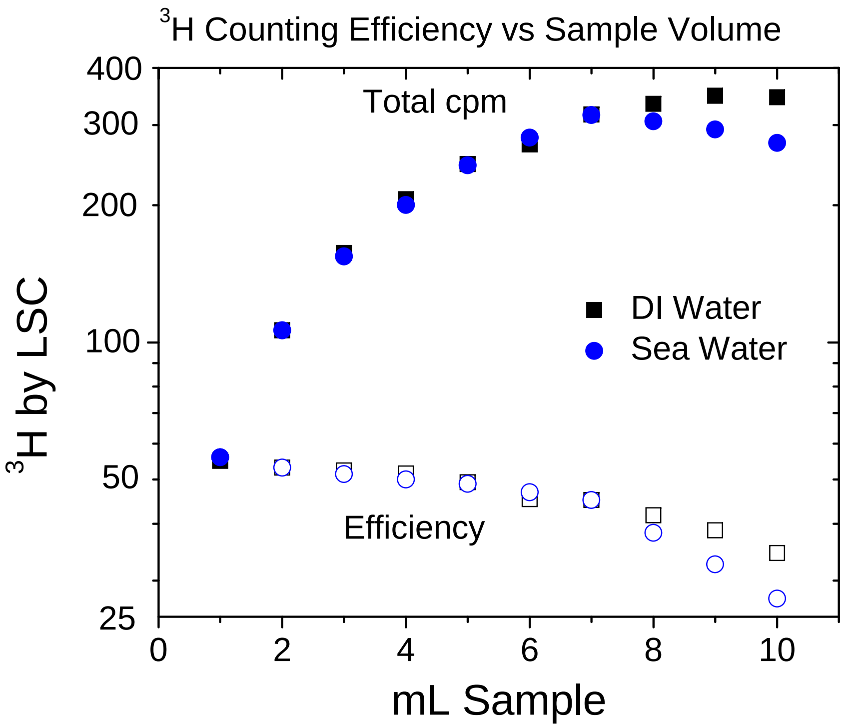
Sample	grams	replicate	% Recovery Sr carrier
Beef	100	6	96.3 ± 0.5
Deer	100	59	83.4 ± 3.5
Fish-Bass	200	72	89.0 ± 16
Fish-Bream	100	57	91.7 ± 10
Fish-Catfish	200	69	89.4 ± 17
Fish-Mullet	200	6	85.6 ± 17
Fish-Red Fish	200	6	77.7 ± 21
Fish-Sea Trout	200	6	74.4 ± 25
Hog	100	17	86.0 ± 7.1
Shellfish	100	5	97.5 ± 0.9

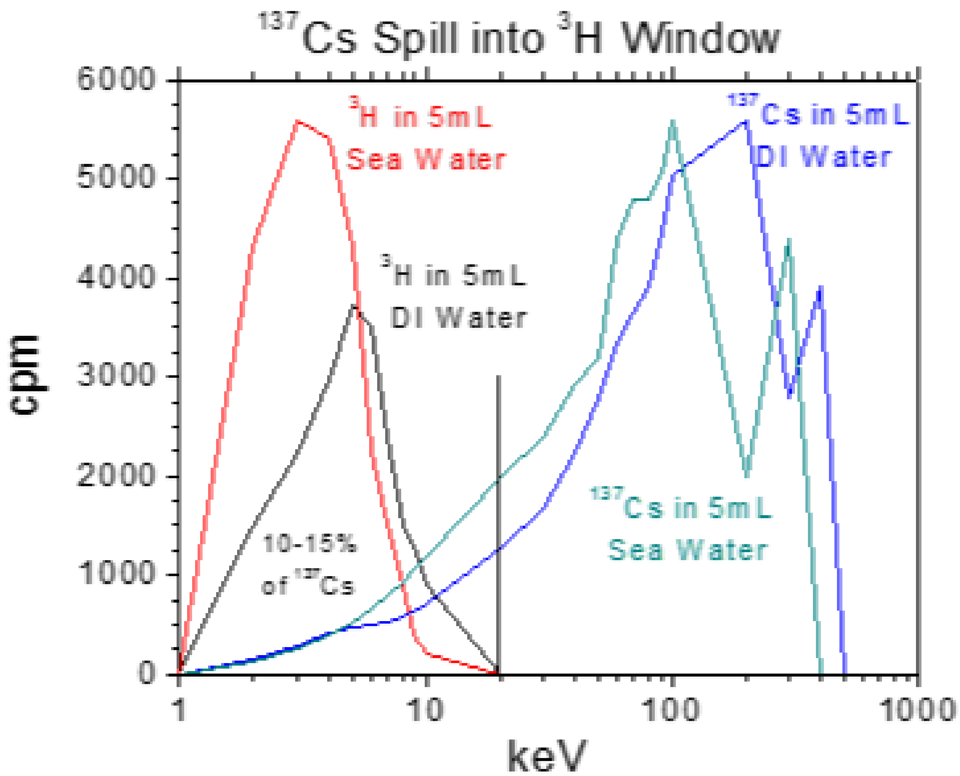
2016 More Application Notes

AN-1601	Method for ^{227}Ac in Geological Samples
AN-1602	Method for ^{227}Ac in Water Samples
AN-1603	Rapid Method for Actinides in Limestone and Marble
AN-1604	Rapid Method for $^{89/90}Sr$ in Limestone and Marble
AN-1605	Rapid Method for $^{89/90}Sr$ in 5g Large Concrete Samples
AN-1606	Rapid Method for ^{90}Sr in 10g Concrete Samples
AN-1607	Rapid Method for Pu, Np, Am in Large Soil Samples
AN-1608	Rapid Method for U and Th in soil
AN-1609	Rapid Method for ^{3}H in water
AN-1610	Rapid Method for $^{59/63}Ni$ in Water
AN-1611	Rapid Method for ^{55}Fe in Water (TEVA)
AN-1612	Rapid Method for ^{55}Fe in Water (TRU)
AN-1613	^{68}Ga Generator
AN-1614	$^{225}Ac/^{225}Ra$ Generator
AN-1615	^{90}Y Generator
AN-1616	$^{210}Po/^{210}Bi$ Generator
AN-1617	$^{227}Th/^{223}Ra$ Generator
AN-1618	$^{228}Th/^{231}Th$ Generator
AN-1619	^{239}Np Generator
AN-1620	$^{224}Ra/^{212}Pb$ Generator
AN-1621	^{234}Th Generator
AN-1622	Separation of ^{89}Zr from Y Target
AN-1623	Separation of ^{86}Y From Sr Target

Examples of New Information in 2016 Applications

- Tritium Columns





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Iron – TRU or TEVA ?

Method Performance

Method	**Replicate**	**%Rec 2mg Fe tracer**	**Fe-55 raw %rec**	**Fe-55 Tracer corrected**	**Bias**	**Impurity***
TRU	1	90.6	93.1	102.8	2.8	<0.5%
	2	90.0	92.3	102.5	2.5	
	3	94.8	92.4	97.5	-2.5	
	4	89.5	94.0	105.0	5.0	
	5	95.8	94.3	98.5	-1.5	
	6	95.8	92.8	96.9	-3.1	
	AVG	92.8	93.2	100.5		
	SD	3.0	0.8	3.3		

Method Performance

Method	**Replicate**	**%Rec 2mg Fe tracer**	**Fe-55 raw %rec**	**Fe-55 Tracer corrected**	**Bias**	**Impurity***
TEVA	1	95.8	89.2	93.1	-6.9	<0.5%
	2	94.4	89.7	95.0	5.0	
	3	97.6	87.2	89.4	10.6	
	4	95.3	88.2	92.6	7.4	
	5	83.9	79.8	95.1	4.9	
	6	89.1	89.6	100.5	-0.5	
	7	80.6	86.4	107.2	-7.2	
	AVG	91.0	87.2	96.1		
	SD	6.6	3.5	5.9		

Where can you find these applications ?

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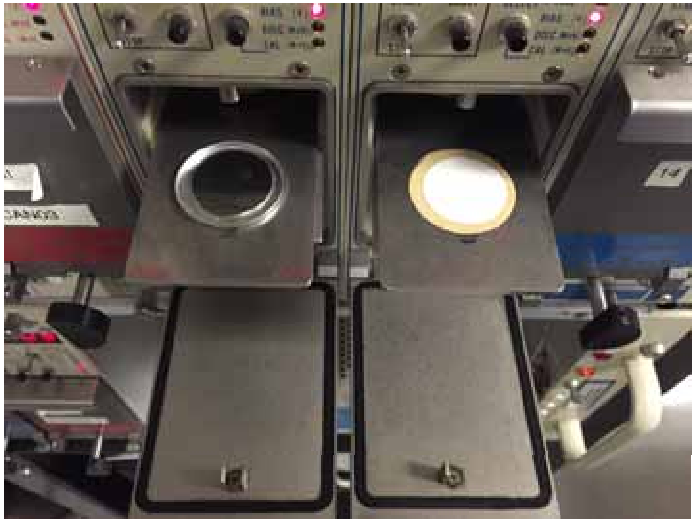
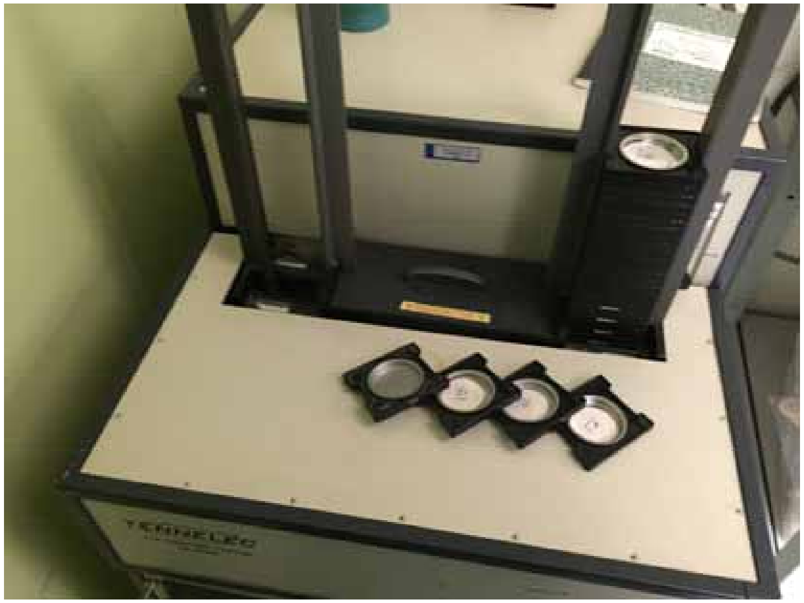
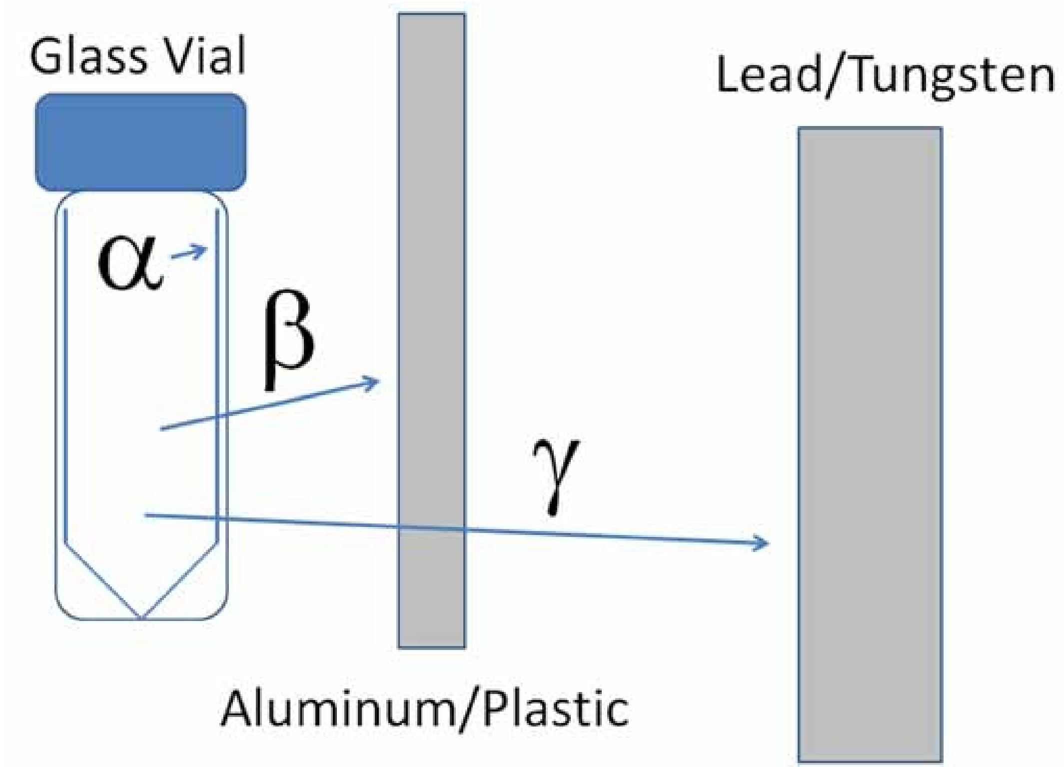
- Custom Separations Training
  - Training is tailored to your needs and goals.
  - Training can incorporate a methods review and modifications recommendation
  - On site training can better equip key staff in trouble avoidance and lead to laboratories savings.





Types of Radiation, in the Lab and Counted by the Lab



T-Flex® in the Lab

- Beaker Shields
- Single Vial Shields
- Multi Vial Shields

State Health Laboratory

RadVision^{3D} Services

- **RadVision^{3D} Services is an innovative, TURNKEY approach to radiation identification and mitigation.**

- **We utilize 3D scanning technology & world-class analysis tools to design work planning improvements and shielding solutions.**

- **Our solutions result in:**

Enhanced Planning & Performance	Improved Worker Safety & Reduced Dose Exposure	More efficient shielding placement and procurement

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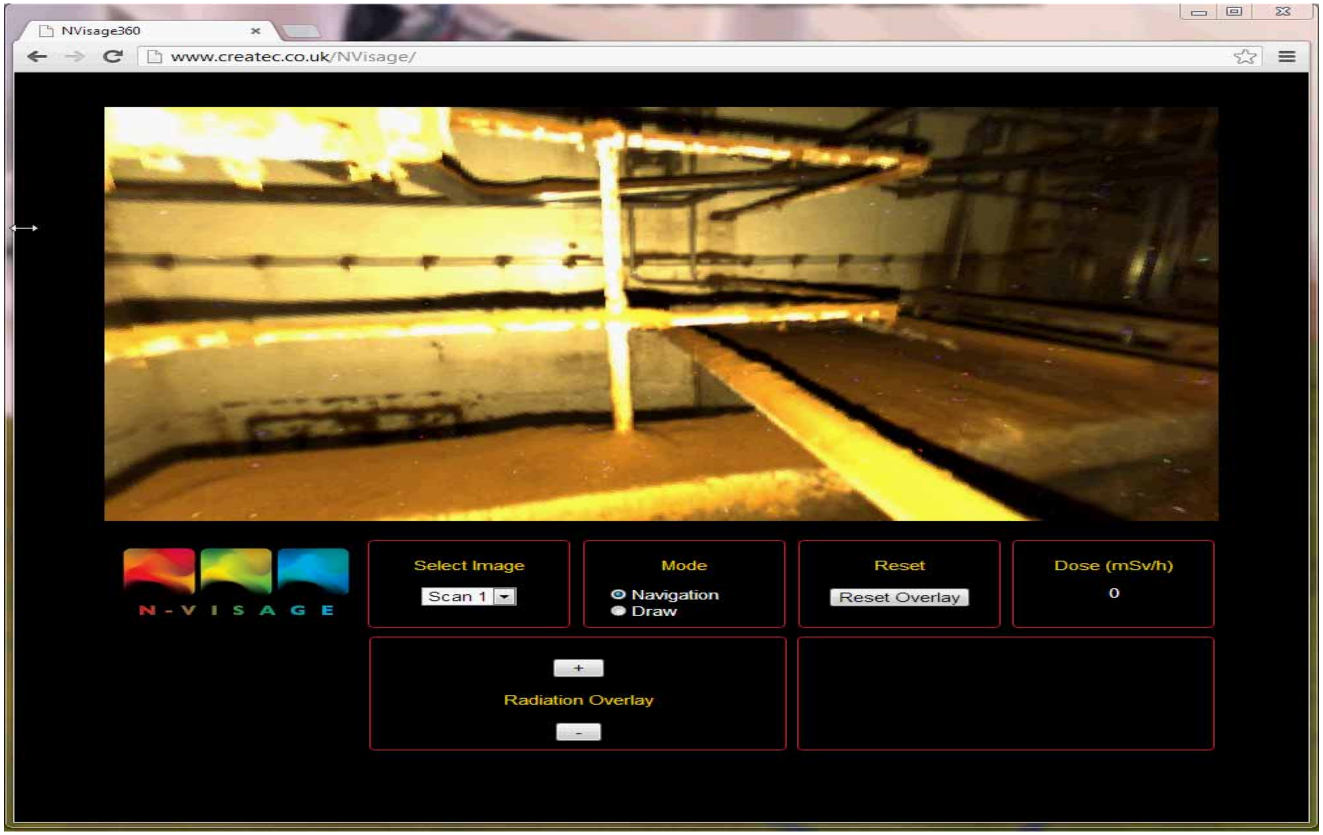
RadVision^{3D} Services

RadVision^{3D} Services is NOT…

- 2-D Snapshots
- Point & Shoot
- Limited Field of View
- Fixed Location
- Dedicated Expert
- Software Purchases

RadVision^{3D} Services

RadVision^{3D} Services takes a Gamma Camera to a third dimension!

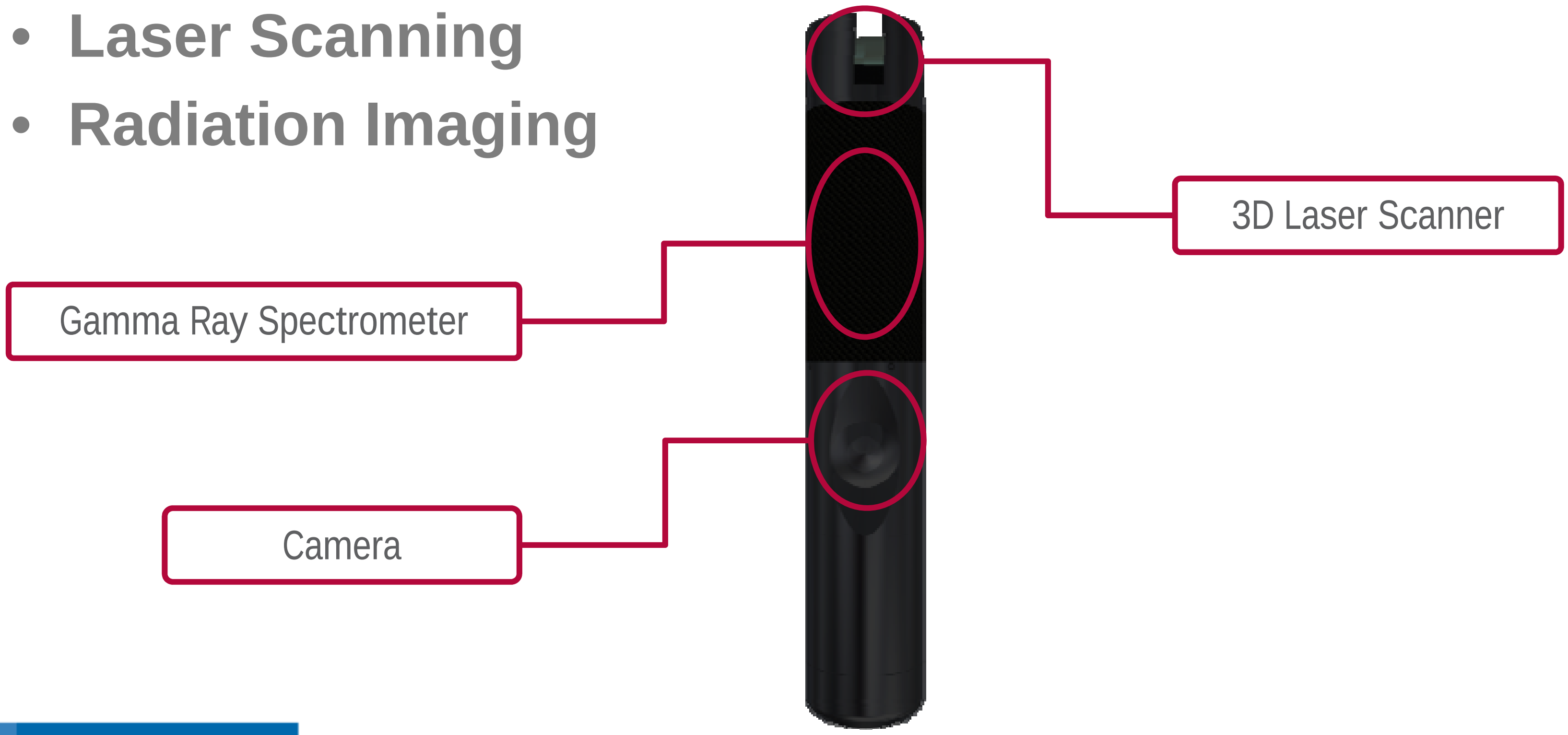


RadVision^{3D} Services

A service	• No personnel requirements • No capital investment • No expensive software upgrades
3-D & 360° x 360°	Data is captured in all directions, not just what is limited to your pointed field of vision
A camera	Provides instant optical imagery of the scanned area in a fisheye view
A laser scanner	Assigns an X,Y,Z geometrical coordinate to all surfaces and creates a 3-D point cloud model
A Gamma Ray Spectrometer	Identifies of individual isotopes, measures dose rate intensity and reveals hot spot locations

Data Collection

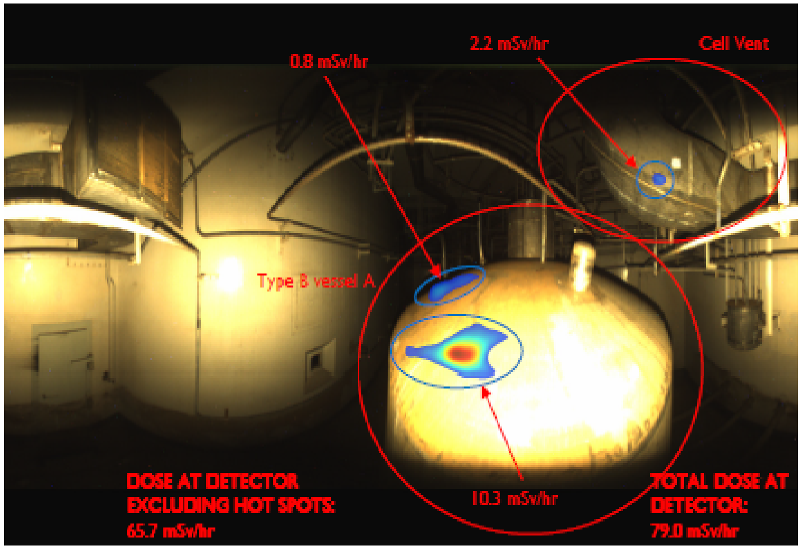
- **Revolutionary 3–in–1 data collection hardware integrates:**
  - **Optical Imaging**
  - **Laser Scanning**
  - **Radiation Imaging**

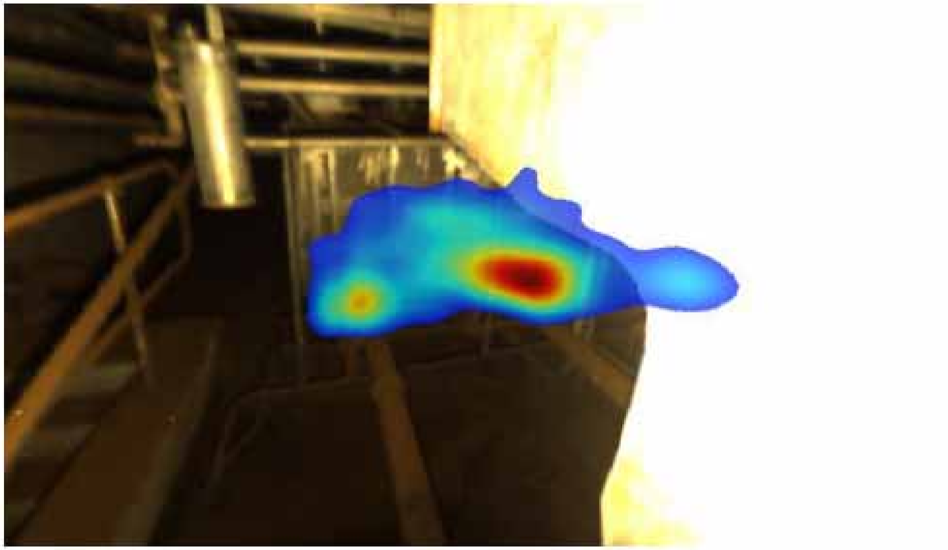


Data Collection – Process Overview

Instant Output

Gamma radiation profile overlaid on optical images





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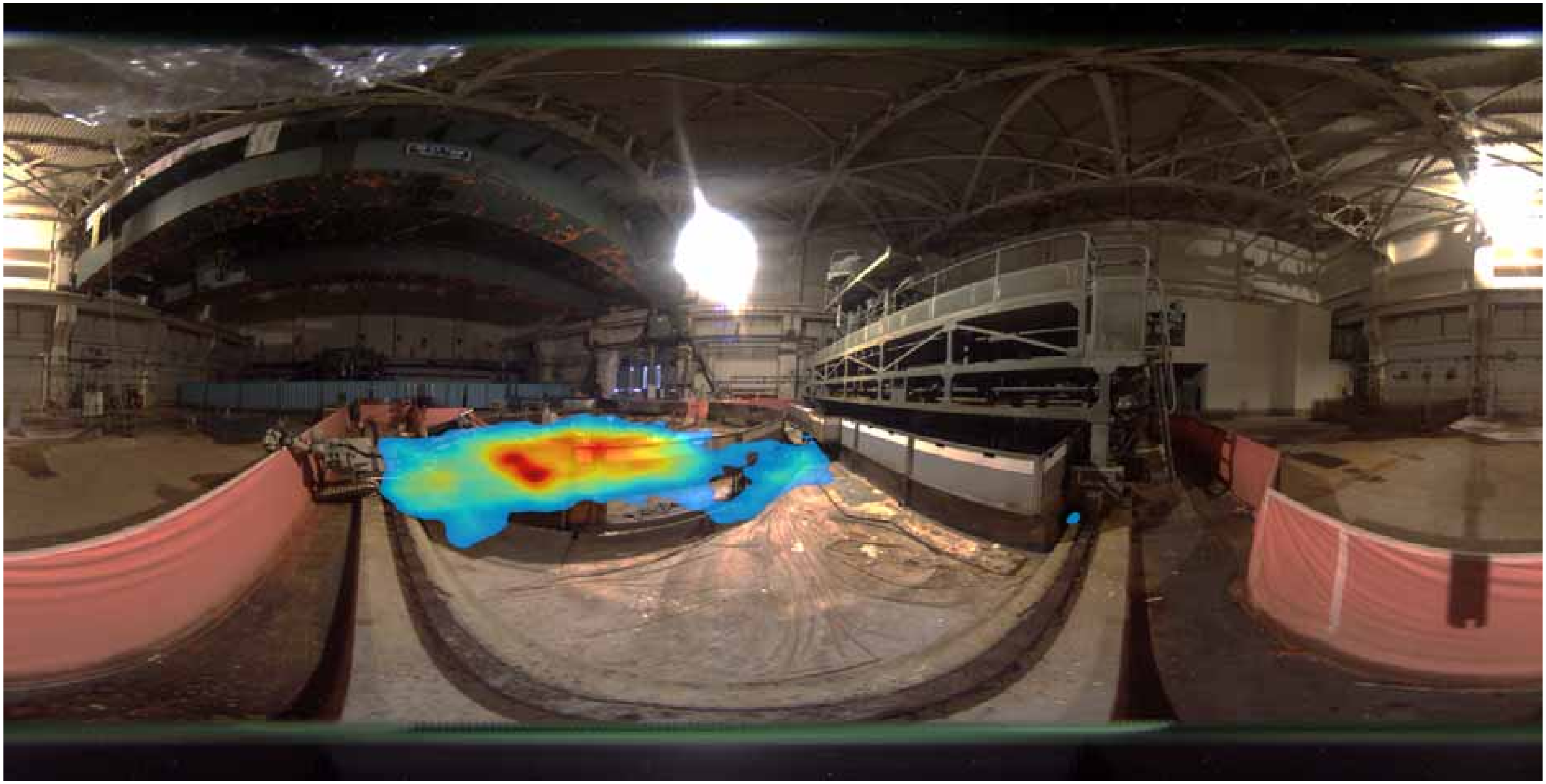
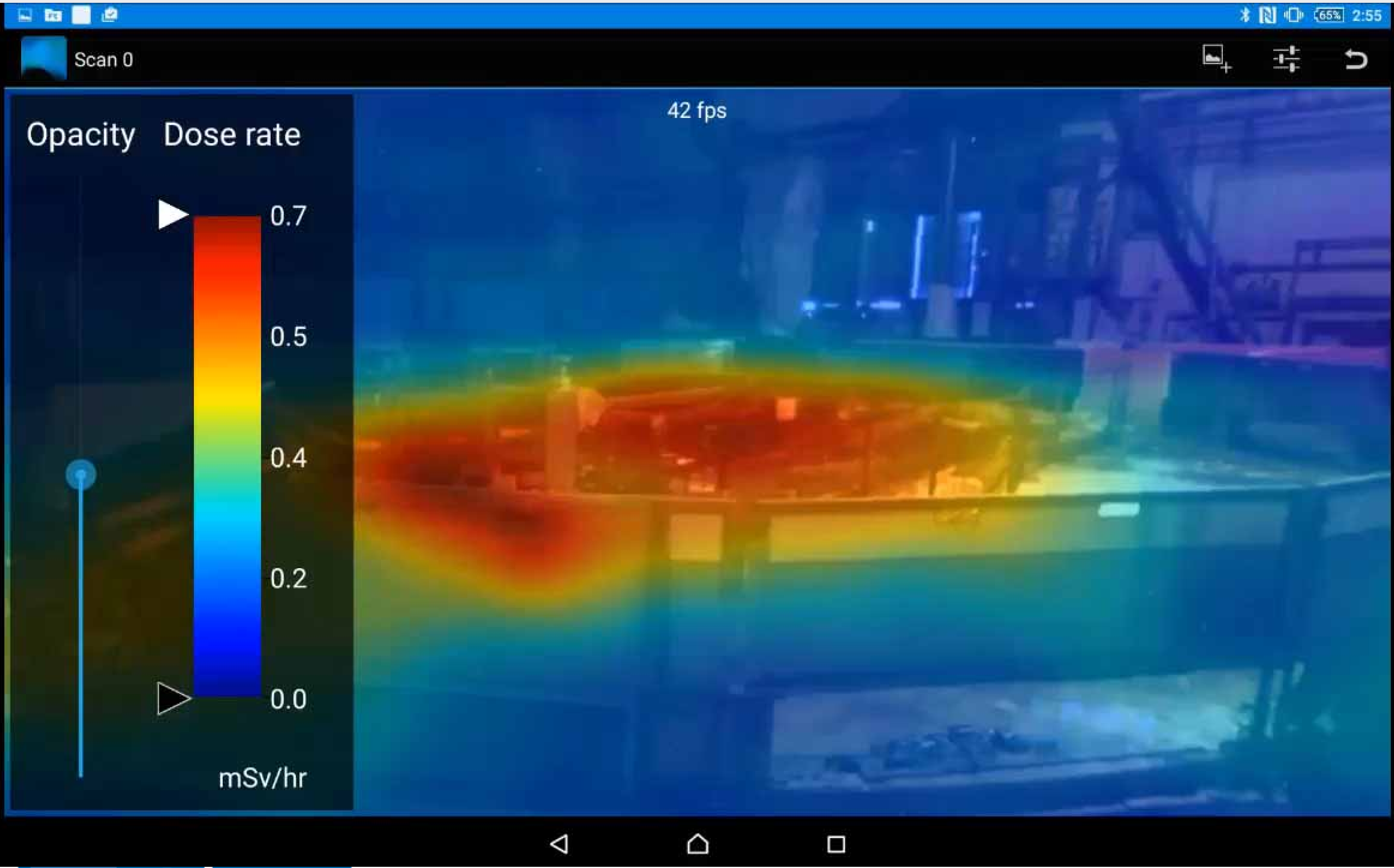
See Facility Conditions

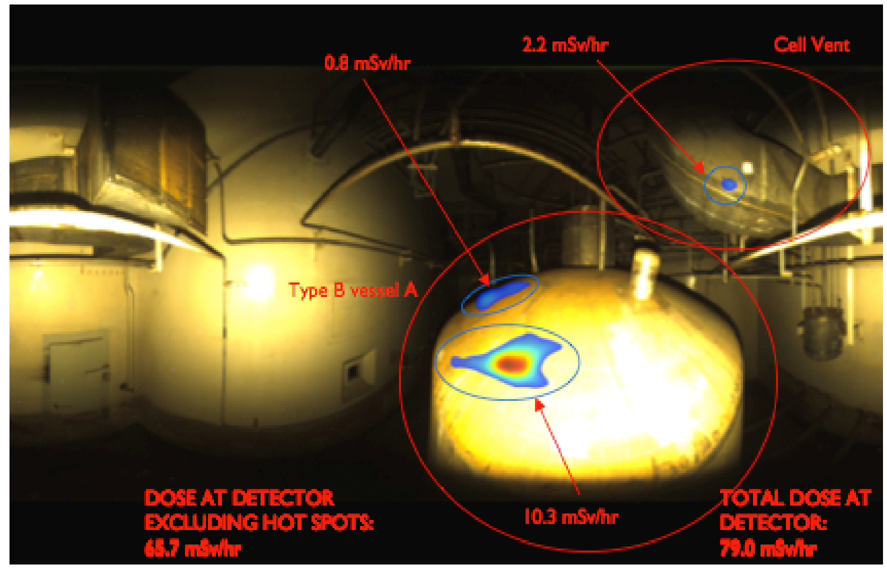


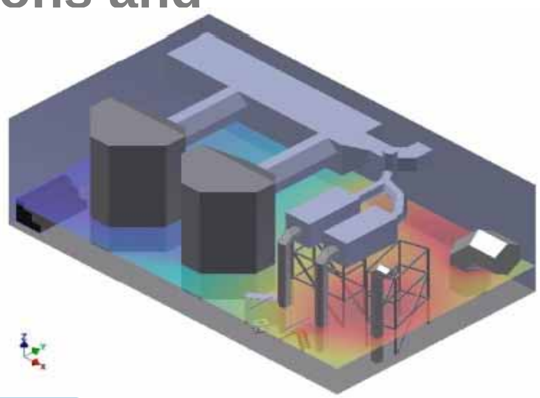
See Faclity Conditions

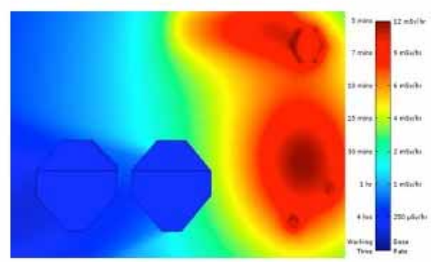


Data Analysis – Knowledge is Power

After the data collection, RadVision^{3D} uses proprietary software to analyze the plant radiation conditions and develop specific plant recommendations. The analyses provide custom work planning, estimated dose reductions and optimized sh utilization wi







Deliverables

- **Instant Snapshots of radiation profile for hot spot identification**
- **Instant quantification of hot spot dose readings**
- **3-D Model Creation**
  - **Dose Plane Analysis**
  - **Accumulated dose estimates**
  - **Shielding effectiveness evaluations**
- **Individual Isotope ID**

Shielding System Configurations

- Present visual evidence of radiation field to upper management
- Optimize Shielding placement and amount of shielding
- 3D laser scan data valuable to all plant departments
- 3D laser scan data + gamma field can be used to optimize shielding design
  - More accurate attenuation estimates
  - Minimized shielding weight = easier engineering/labor
  - 3D measurements for attaching hardware, custom molds, etc.
  - Superior presentation material for PHC meetings
- Optional post processing for dose plane style dose estimation

RadVision^{3D} Services

Better Data Collection | **Sophisticated Analysis & Conclusions** | **Targeted Shielding Solutions**

See the Full Picture	Understand the Impact	Optimize Your Shielding
360° Gamma Imaging & Laser Scanning	Powerful Proprietary Software Differentiates from a Standard Gamma-cam	Customized, Engineered Shielding Solutions
Precise and Complete Radiation Profile of Area	2D Radiation Overlay & 3D Dose Plane Analyses	Reduced Barriers in Containment
Visualize Plant Conditions in a 3D Virtual Reality Environment	Sophisticated Work Planning Tools	Optimized Balance Between Permanent and Temporary Shielding Applications

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Thank you for your attention.

Questions?