RADIOLOGICAL CHARACTERIZATION
Laboratory Procedures

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PURPOSE

- To measure activity concentration of materials taken from a shutdown nuclear research reactor

- To comply with regulatory requirements for clearance of materials for unrestricted or restricted reuse

RADIONUCLIDE INVENTORY

1. **Neutron activated materials**

- **Located in the reactor structure and have been irradiated by neutrons**
- **Major activation products – ^{3}H, ^{14}C, ^{22}Na, ^{36}Cl, ^{39}Ar, ^{41}Ca, ^{54}Mn, ^{55}Fe, ^{59}Ni, ^{63}Ni, ^{60}Co, ^{65}Zn, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{108m}Ag, ^{110m}Ag, ^{125}Sb, ^{133}Ba, ^{134}Cs, ^{152}Eu, ^{154}Eu, ^{155}Eu, ^{166m}Ho**

2. **Contaminated Materials**

- **typically found as surface contamination in reactor systems where fuel failure has occurred, on fuel handling systems, and in primary heat transport systems of any reactor**
- **Major fission products – ^{90}Sr, ^{99}Tc, ^{106}Ru, ^{129}I, ^{137}Cs, ^{144}Ce**
- **Major actinides - ^{238}Pu, ^{239}Pu, ^{241}Pu, ^{241}Am, ^{242}Cm, ^{244}Cm, ^{232}U, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U**

MATERIALS TO BE SAMPLED

- Material samples
  - -concrete, steel, resins, wood, tiles, paint chips, foam
  - - pipes, sludge, pool water, etc…
- Environmental samples
  - - soil/sediment, grass, water
- Biological
  - -urine, excrete

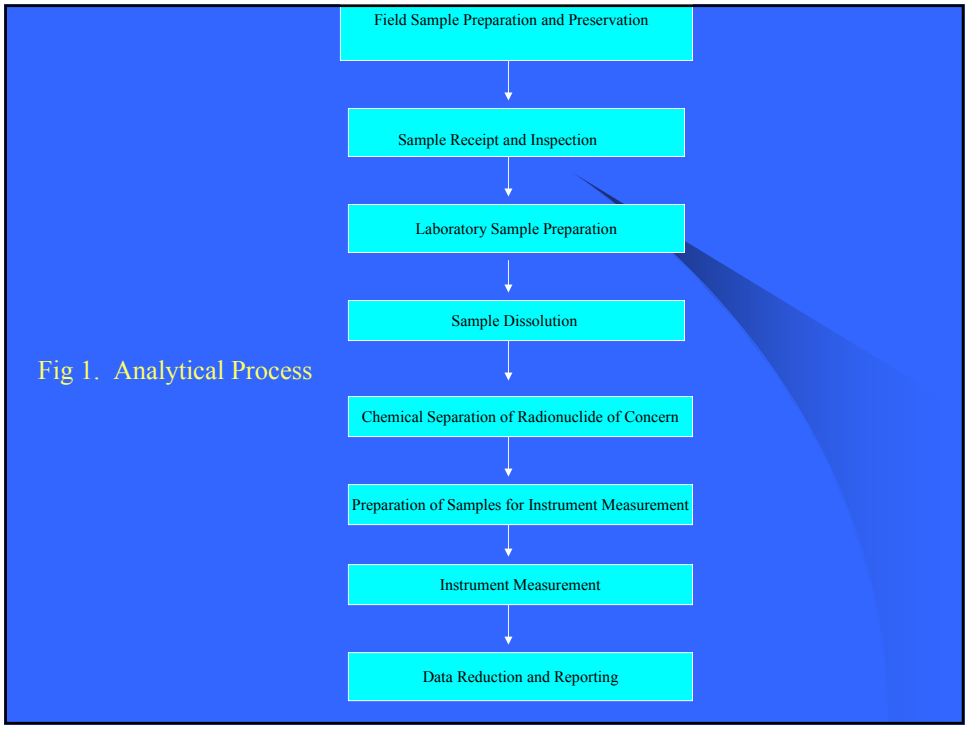


Fig 1. Analytical Process

Field Sampling

Sampling design should be carefully planned

Sampling is the source of biggest contribution to error in analysis

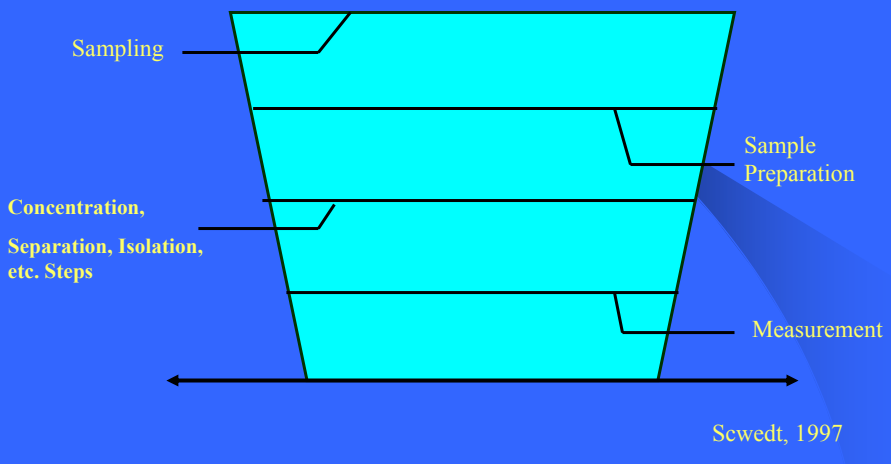


Figure 2. Degree of error in laboratory sample preparation relative to other activities

Sample Receipt and Inspection

Sample Receipt
- -receive in designated areas
- -Check accompanying documents
- -Conduct radiological survey

Sample Inspection
- -Check physical integrity of package and container
- -Confirm sample identity
- -Confirm field preservation, if any

Laboratory Sample Tracking
- -Sample log-in
- -Sample tracking during analysis
- -Storage of sample

Detection Methods, MDAs and Release Criteria for Radionuclides

Isotope	Half-life	Emission	Detection Methods	MDA, Bq/g	Clearance Level, Bq/g
H-3	1.2E+01	β^-	Liquid Scintillation	10	100
C-14	5.7E+03	β^-	Liquid Scintillation	1	1
Cl-36	3.0E+05	β^-	Liquid Scintillation	1	1
Ca-41	1.0E+05	EC	Liquid Scintillation	(1 to 10)	
Mn-54	8.6E-01	EC, γ	Gamma spectrometry	0.5	
Fe-55	2.7E+00	EC, X	Liquid Scintillation or X-ray spectrometry	10	
Co-60	5.3E+00	β^- , γ	Gamma spectrometry	0.5	0.1
Ni-59	7.5E+04	EC, X	X-ray spectrometry	10	100
Ce-144	8.0E-01	β^- , γ	Gamma spectrometry	0.5	
Ni-63	1.0E+02	β^-	Liquid Scintillation	1	100
Cs-134	2.1E+00	β^- , γ	Gamma spectrometry	0.5	0.1
Cs-137	3.0E+01	β^- , γ	Gamma spectrometry	0.5	0.1

Isotope	Half-life	Emission	Detection Methods	MDA, Bq/g	Clearance Level, Bq/g
I-129	1.6E+07	β^-	ICPMS (or X ray spectrometry)	0.007	0.01
Sr-90	2.9E+01	β^-	Beta counting or Liquid scintillation	1	1
Zr-93	1.5E+06	β^-	ICPMS	0.1	
Nb-93m	1.6E+01	IT, X	X ray spectrometry or liquid scintillation	10	
Nb-94	2.0E+04	β^- , γ	Gamma spectrometry (or ICPMS)	0.5 (7)	0.1
Mo-93	3.5E+03	EC,X	X ray spectrometry or liquid scintillation	10	10
Tc-99	2.1E+05	β^-	ICPMS	0.6	
Ru-106	1.0E+00	β^- , γ	Gamma spectrometry	0.5	
Ag108m	1.3E+02	EC, γ	Gamma spectrometry	0.5	
Ag-110m	7.0E-01	β^- , γ	Gamma spectrometry	0.5	
Sb-125	2.8E+00	β^- , γ	Gamma spectrometry	0.5	
Ba-133	1.1E+01	EC, X, γ	Gamma spectrometry	0.5	

Isotope	Half-life	Emission	Detection Methods	MDA, Bq/g	Clearance Level, Bq/g
Eu-152	1.3E+01	EC, β^-,X, γ	Gamma spectrometry	0.5	0.1
Eu-154	8.6E+00	β^-,X, γ	Gamma spectrometry	0.5	0.1
Eu-155	4.8E+00	β^-,X, γ	Gamma spectrometry	0.5	1
Ho-166m	1.2E+03	β^-,X, γ	Gamma spectrometry	0.5	
U-234	2.5E+05	α. X	Alpha spectrometry	0.02	
U-235	7.0E+08	α,, γ	ICPMS	0.0001	
U-238	4.5E+09	α	ICPMS	0.00001	
Pu-238	8.8E+01	α. X	Alpha spectrometry	0.02	
Pu-239	2.4E+04	α	Alpha spectrometry	0.02	0.1
Pu-241	1.4E+01	β^-	Liquid scintillation	1	
Am-241	4.3E+02	α. X, γ	Alpha spectrometry	0.02	
Cm-242	4.5E+01	α. X	Alpha spectrometry	0.02	
Cm-244	1.8E+01	α. X	Alpha spectrometry	0.02	

Gamma Spectrometry

- Nondestructive analysis of gamma emitting radionuclides
- Requires minimum sample preparation
- Measure gamma radiation using scintillation detectors (NaI (Tl)) crystals and semiconductor detectors (High purity germanium detectors)

I. Sample Preparation

1. Liquid Samples
   - Evaporation
   - Adsorption with AMP (Cs)/Precipitation with MnO_2 (other metals except Cr)
   - Ion-exchange resin
2. Solid Samples
   - Dry, grind, sieve, mix to homogenize, get sub sample
3. Air particulate filters,Swipe/Smear Samples
   - No pretreatment
   - Leach with dilute HNO_3 or 0.05M EDTA

II. Standard Calibration

III. Spectrometer Calibration

1. Energy Calibration
2. Efficiency Calibration
3. Quality Control Check

IV. Sample Measurement

1. Count a blank sample
2. Determine minimum detectable activity (MDA)
3. Count sample

V. Statistical Treatment of Data

1. Calculate Activity including correction factors
2. Calculate total uncertainty

Alpha Spectrometry

- Analysis of alpha emitting radionuclides
- Requires radiochemical separation
- Use of Si semiconductor detectors

I. Sample Treatment

1. Liquid Samples
   - Acidify and filter
   - May reduce volume by evaporation or ion exchange

2. Solid Samples
   - Drying, grinding, sieving, homogenize
   - May reduce volume by ashing
   - Sample decomposed with concentrated acids, e.g. HCl, HNO_3, HF; H_2O_2 or alkali fusion with K_2CO_3
   - Sample can be leached with dilute nitric acids or complexing agents

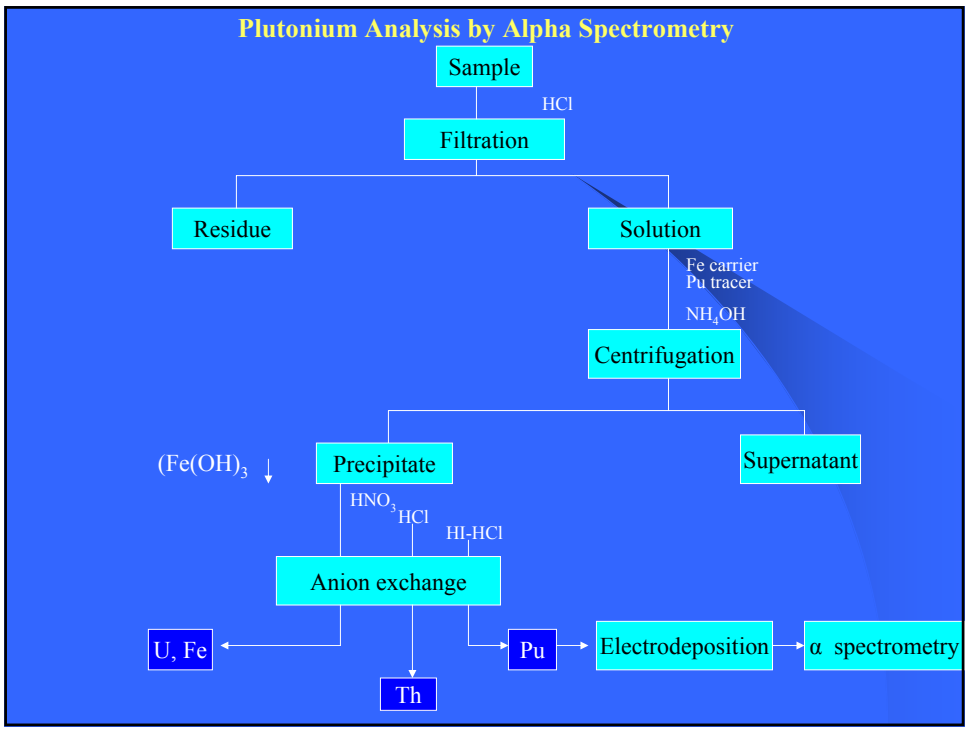
3. Air Particulate or Swipe/Smear Samples
   - Cellulose and membrane filters can be dissolved in mineral acids
   - Glass-fiber filters can be leached with dilute acids or chelating agents

I. Preconcentration
   - Precipitation

II. Chemical Separation
   - Anion exchange

III. Source Preparation
   - Electrodeposition
   - Precipitation with cerium or lanthanum

IV. Counting



Liquid Scintillation Counting

- Gross alpha/beta analysis of samples
- Analysis of beta emitters
- Analysis of alpha emitters
- May require radiochemical separation

I. Sample Preparation

1. Liquid Samples
   - Distillation/electrolytic enrichment (^{3}H)
   - Oxidation/CO_2 trapping (^{14}C)
   - Reduce volume by evaporation, ion exchange,precipitation
2. Solid Samples
   - Dissolution with concentrated mineral acids (for activated and geogenic samples)
   - Leaching with dilute mineral acids (for surface contaminated samples
   - Reduce volume by ashing at high tempeatures (except volatile radionuclides, e.g. ^{129}I, ^{99}Tc

3. Air Particulate/Swipe/Smear Samples
   - Cellulose and membrane filters can be dissolved in concentrated mineral acids
   - Glass fiber filters can be leached with dilute mineral acids or chelating agents
   - Filter paper can be placed directly in LSC vial with addition of liquid scintillant

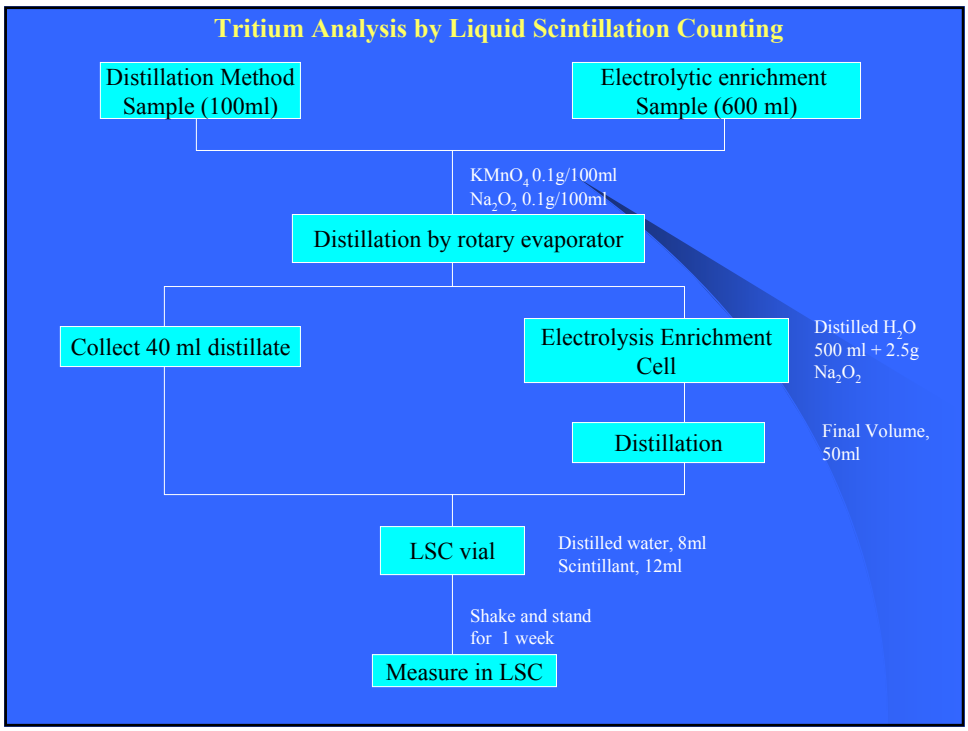
II. Setting of Measurement Conditions for Radionuclide
   - Determination of Figure of Merit (FOM)

III. Drawing of Quench Correction Curve
   - Preparation of quench standards
   - Draw quench correction curve
   - Efficiency determination

IV. Counting of Sample

V. Calculation of Activity

- Calculate Average, $X \pm 3\sigma$
- Correct activity using decay correction factor




THANK YOU