Radiochemical Analysis of ^{63}Ni, ^{55}Fe, and radiostronium in waste and environmental samples

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Production of ^{63}Ni and ^{55}Fe in Nuclear Reactor (neutron activation)

- ^{63}Ni:
  - $^{62}Ni(n, \gamma)^{63}Ni$ (σ=14.5 b; η_{62Ni}=3.63%)
  - $^{63}Cu(n, p)^{63}Ni$, (η_{63Cu}=69.17%)
- ^{55}Fe:
  - $^{54}Fe(n, \gamma)^{55}Fe$ (σ=2.3 b; η_{54Fe}=5.85%)
  - $^{56}Fe(n, 2n)^{55}Fe$, (η_{56Fe}=91.75%)

Type of waste samples for decommissioning

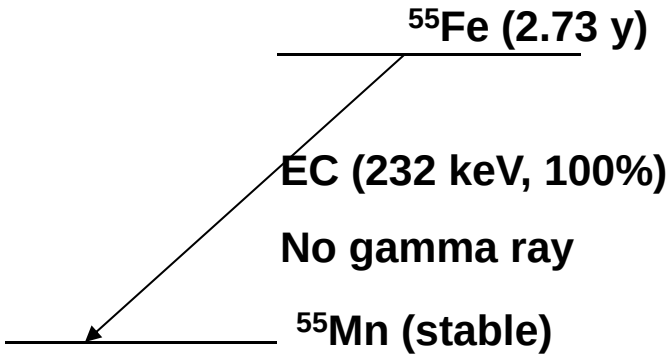
- Graphite (reactor)
  - ^{3}H, ^{14}C, ^{55}Fe, $^{63,59}Ni$, ^{60}Co, ^{152}Eu
- Concrete (normal or heavy)
  - ^{41}Ca, ^{60}Co, ^{55}Fe, $^{63,59}Ni$, ^{133}Ba, ^{152}Eu
- Steel/stainless steel
  - ^{55}Fe, $^{63,59}Ni$, ^{36}Cl, ^{93}Zr, ^{93}Mo, ^{94}Nb, ^{60}Co, ^{152}Eu, transuranics
- Aluminium
  - ^{60}Co, ^{63}Ni, ^{55}Fe, ^{36}Cl
- Lead
  - , ^{60}Co, ^{63}Ni, ^{55}Fe
- Water
  - ^{3}H, ^{14}C, ^{63}Ni,^{55}Fe, ^{99}Tc, ^{129}I, ^{90}Sr, ^{60}Co, ^{137}Cs, transuranics
- Ion exchange resin
  - ^{55}Fe, $^{63,59}Ni$, ^{14}C, ^{99}Tc, ^{36}Cl, ^{93}Zr, ^{93}Mo, ^{94}Nb ^{90}Sr, ^{129}I, ^{137}Cs, ^{60}Co, ^{135}Cs, transuranics

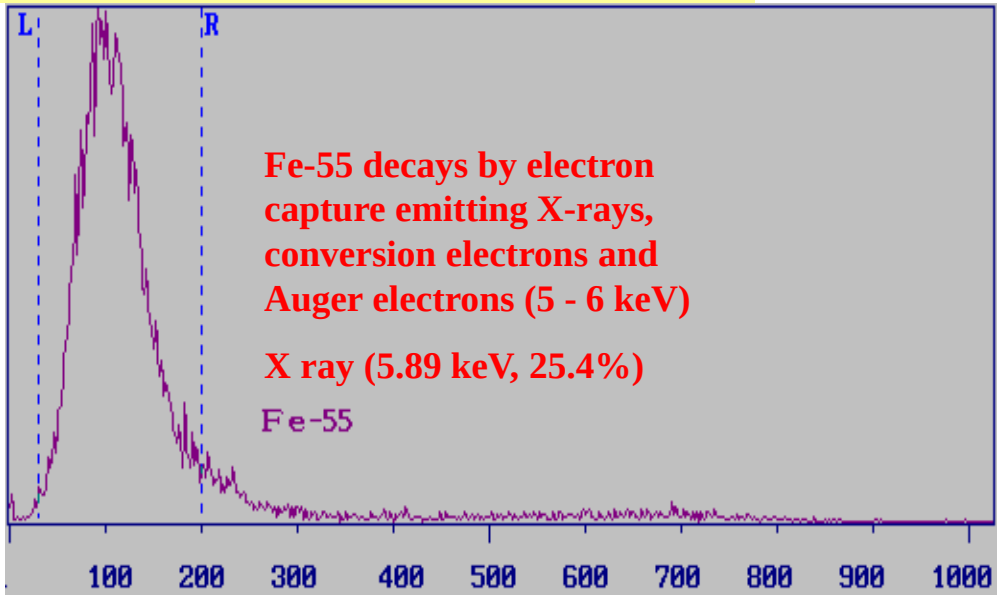
^{55}Fe and ^{63}Ni in the Environment

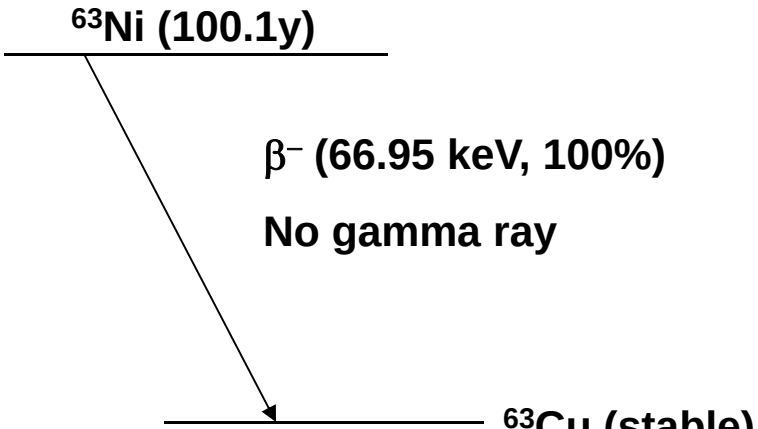
- Sorce:
  - Nuclear weapons testing in 1960's
  - Discharge fron reprocessing plants
  - **Discharge from nuclear power plants**

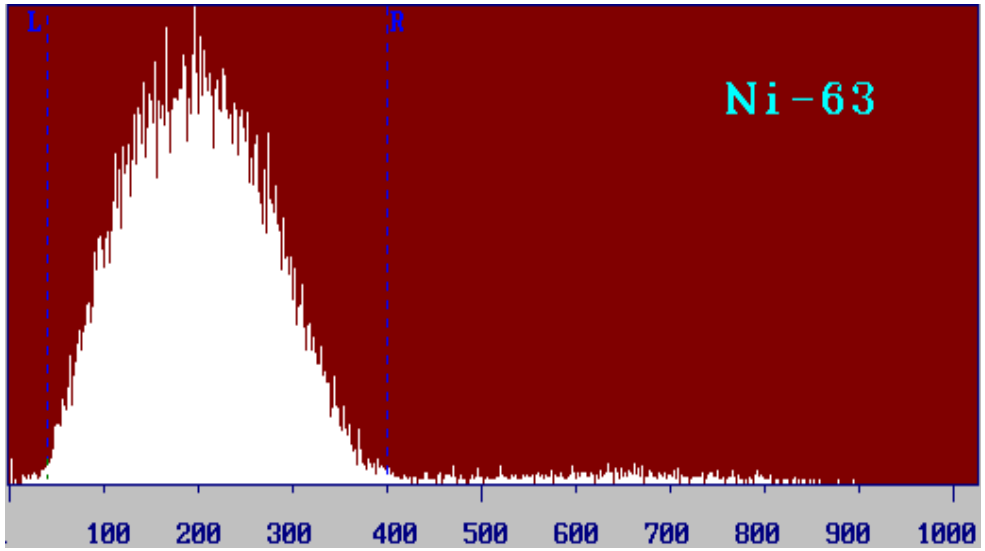
- Investigation of ^{63}Ni and ^{55}Fe in the environment
  - Measurement of the contamination level from nuclear facility
  - Using ^{55}Fe and ^{63}Ni released from nuclear facility as a tracer to study their environmental and chemical behaviours
- Environmental Samples
  - Water
  - plants (seaweed, grass, lichen, fish.)
  - soil, sediment

Decay of ^{63}Ni and ^{55}Fe









Aneutron activation products of Fe and Ni

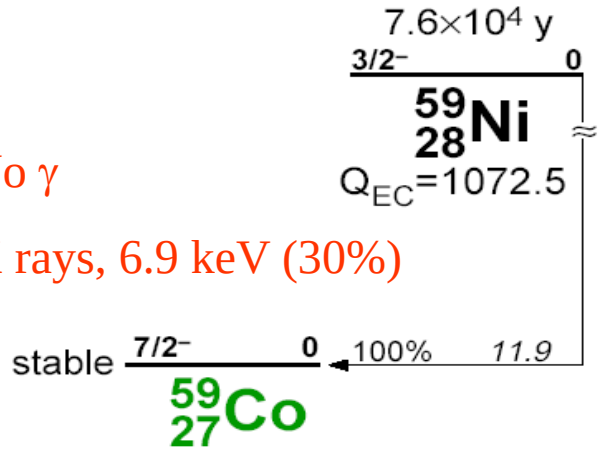
Target nuclide	Abundance %	(n, γ) cross section	Activation product	Half life	Decay
^{58}Ni	68.3	4.64	^{59}Ni	7.6×10^4 y	EC (Kx=6.9 keV)
^{60}Ni	26.1	2.82	^{61}Ni	Stable	
^{61}Ni	1.13	2.51	^{62}Ni	Stable	
^{62}Ni	3.59	14.25	^{63}Ni	100 y	β-, 66.9 keV
^{54}Fe	5.85	2.3	^{55}Fe	2.73 y	EC
^{56}Fe	91.75	2.6	^{57}Fe	stable	
^{58}Fe	0.28	1.31	^{59}Fe	44.5 d	β-, γ

Atomic ratio: ^{59}Ni/^{63}Ni=6.5:1

Activity ratio: ^{59}Ni/^{63}Ni=1:133

Other isotopes of Ni and Fe produced in Nuclear reactor

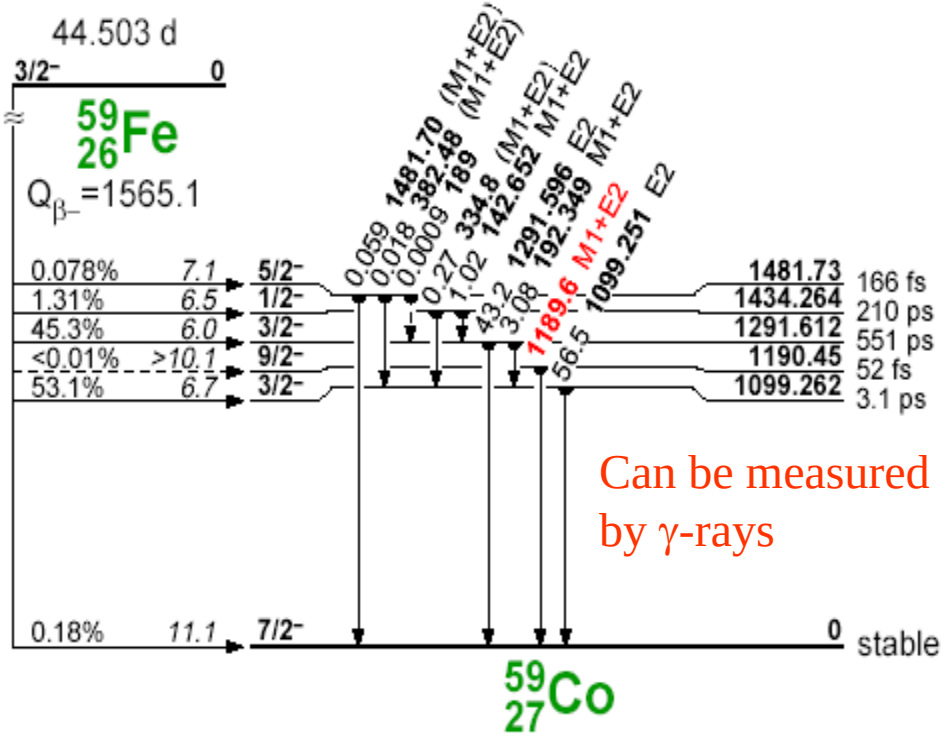
- Ni isotopes: ^{59}Ni,
  - ^{58}Ni(n, γ)^{59}Ni; ^{60}Ni(n,2n)^{59}Ni



No γ

X rays, 6.9 keV (30%)

- Fe isotopes: ^{59}Fe
  - ^{58}Fe(n, γ)^{59}Fe; ^{59}Co(n, p)^{59}Fe



Analytical method for ^{63}Ni and ^{55}Fe

- Due to their low energy of beta particle and measurable electrons, LSC is the most suitable method for their measurement.
- Due to their pure beta and EC decay, they have to be separated from matrix elements and all other radionuclides.

Mearement methods

^{55}Fe: X-ray spectrometry (<1%);

LSC (30-45%)

^{63}Ni: gass flow counting(anti-coincidence, <10-50%)

Ion implanted silicon detector (1-6%)

LSC (60-80%)

- Analytical procedure:
  - Decomposition of sample
  - Separation of Ni or Fe from matrix elements and all other radionuclides
  - Preparation of a suitable solution for LSC measurement.

Decomposition of samples

- Metals (steel, Ni-Cr-X alloy, Copper, Lead, Al alloy)
  - Acid degestion
- Concrete, soil, sediment
  - Alkali fusion followed by water leaching
  - Acid digestion
- Plants, organic materials, resin
  - Ashing followed by acid digestion
- Graphite
  - Ashing (800 ^{o}C) followed by acid leaching
  - Degestion with mixed acids (HNO_3+$HClO_4$+H_2SO_4) (it takes 3-4 hours)

Decomposition of graphite by ashing for determination of ^{63}Ni and ^{55}Fe

Temp ^{o}C	Ashing time	Carrier,mg		Degestion	Recovery, %	
		Fe	Ni		^{55}Fe	^{63}Ni
1100	3 min.	---	---	---	---	45%
800	2-3 h	…	…	HCl	96-98 %	97-99%
800	2-3 h	10	…	HCl	95-98%	90-95%
800	2-3 h	…	10	HCl	90-95%	95-98%
800	3 h	4	2	HCl	40-75%	30-65%
800	24 h	4	2	HCl+ $HClO_4$		30-45%
800	6 h	….	2	HCl+ $HClO_4$	80-85%	75-80%
800	3.5 h	4	2	HCl + $HClO_4$	92-95%	90-95%
800	3	10	5	HCl + $HClO_4$	91-95%	90-95%

- Ni and Fe is lost during ashing at:
  - Higher ashing temperature (>900 ^{o}C)
  - Longer ashing time
  - Add both Fe and Ni carrie and leaching just with HCl (this is due to a Fe-Ni compound formed which is difficult to be dissolved by HCl)
- Optimal ashing conditions:
  - 750-850 ^{o}C
  - Less than 3.5 hours
  - Leaching with HCl + $HClO_4$ or add Fe and Ni carrier individually or after ashing.

Interfering Radionuclides

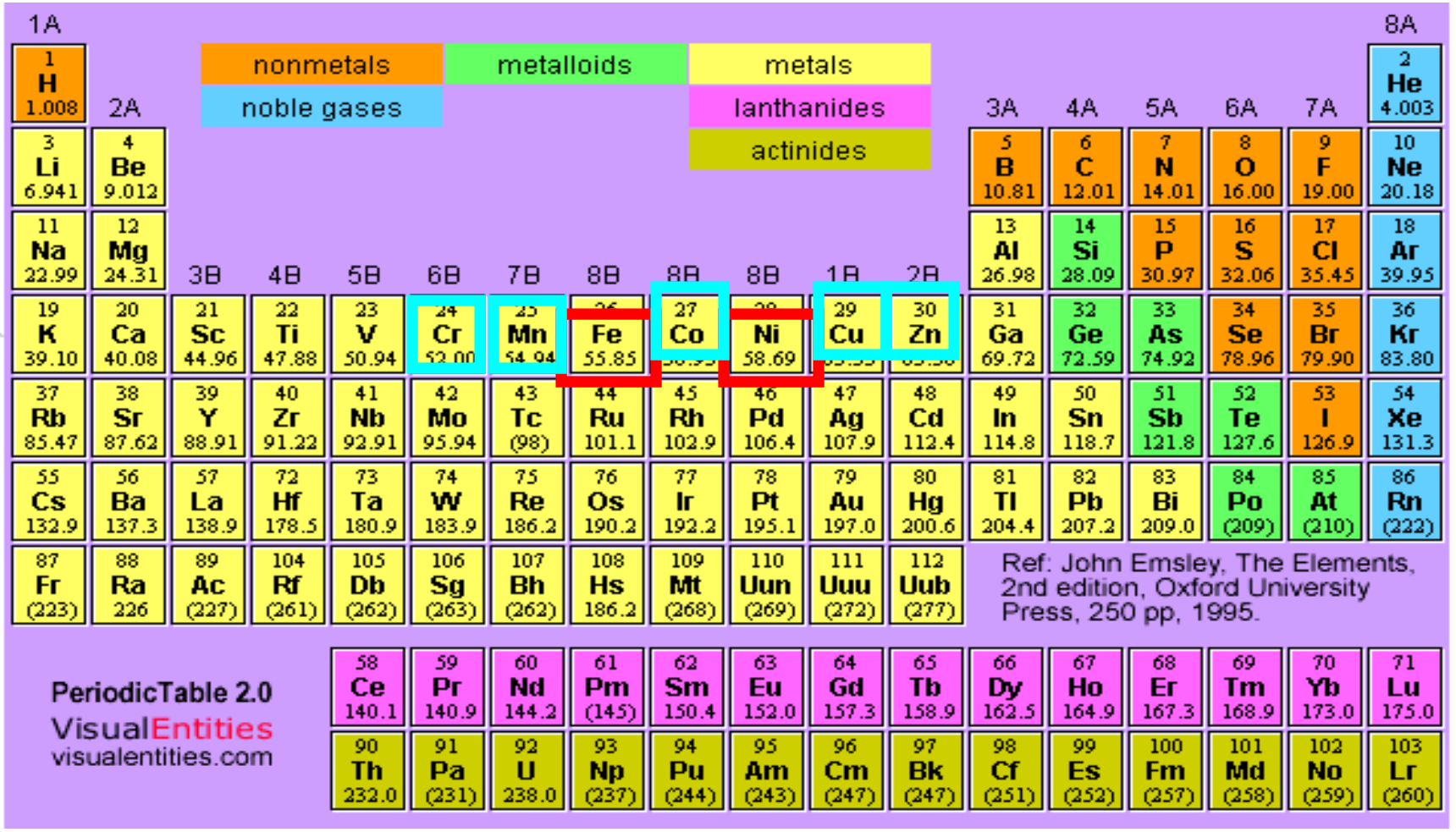
Nuclide	Half-life	Decay	Nuclide	Half-life	Decay
^{60}Co	5.27 y	β^-,γ	^{3}H	12.33 y	β^-
^{58}Co	70.86 d	β^+, γ	^{14}C	5730 y	β^-
^{152}Eu	13.54 y	$\varepsilon, \beta^-,\gamma$	^{133}Ba	10.51 y	ε
^{154}Eu	8.59 y	β^-,γ	^{41}Ca	1.03E5 y	ε
^{51}Cr	27.7 d	ε,γ	^{36}Cl	3.01E5 y	β^-, ε
^{65}Zn	244.3 d	$\varepsilon, \beta^+, \gamma$	^{137}Cs	30.7 y	β^-
^{54}Mn	312.3 d	$\varepsilon, \beta^+, \gamma$	^{134}Cs	2.06 y	β^-,ε
^{151}Sm	90 y	β^-	^{90}Y	64 h	β^-
^{90}Sr	28.79 y	β^-			

Chemical Separation of Ni

1. From main matrix elements
2. From, Ba, Cl, Cs, Ca, Sr, etc.
3. From actinides
4. From Fe, Co, Cu, Mn, Zn etc.
5. From Cr, Eu, Cu, Co.

Chemical Separation of Fe

1. From main matrix elements
2. From, Ba, Cl, Cs, Ca, Sr, etc.
3. From actinides
4. from Ni, Co, Cu, Mn,Zn, Cr, Eu, etc.



Fe and Ni are transite metels

Traditional methods for separation of Ni

- Precipitation as $Ni(OH)_2$, separation from Sr, Cs, ^{3}H, ^{14}C, Ba, Ca, Cl.
- Precipition by ammonium, separate Ni from Fe, Mn, Eu, Pb, Al, Cr.
  - Low recovery of Ni in this method (Ni can be also partly precipitate in ammonium solution)
  - Cannot separate Cu, Co, etc.
- Ion exchange to separate Ni from Co, Cu, Zn, Fe, and transuranics.
- Precipitation or extraction of complex of Ni with dimethylglyoxime (DMG).
  - Co and Cu can also form a complex with DMG and extracted
- Evaporation of $Ni(CO)_6$

Separation of Fe and Ni by hydroxides precipitation

Element	Precipitation, %		Solution, %
	NaOH (pH9)	NH_4OH	NH_4OH
Fe^{3+}	>99.8	>99.8	<0.2
Ni^{2+}	>99.8	>20	< 80
Co^{2+}	>99.5	<20	< 80
Ba^{2+}	<30.5	<30.0	>70
Eu^{3+}	>99.8	>99.8	<0.2
Cs^{+}	<0.2	<0.2	>99.8
Sr^{2+}	<37.5	<35.0	>60

- Most of matrix in concrete and environmental samples, such as C, S, Ca, Si, Na will be separated.
- The recovery of Ni is not satisfied using ammonium to separate Ni from other matals by hydroxides precipitation
- Other metals such as Mn, Cr, V, Al, Pb, and transuranics will also be precipitated by NaOH, and cannot be separated from Ni and Fe.

Separation of Ni and Fe by anion exchange

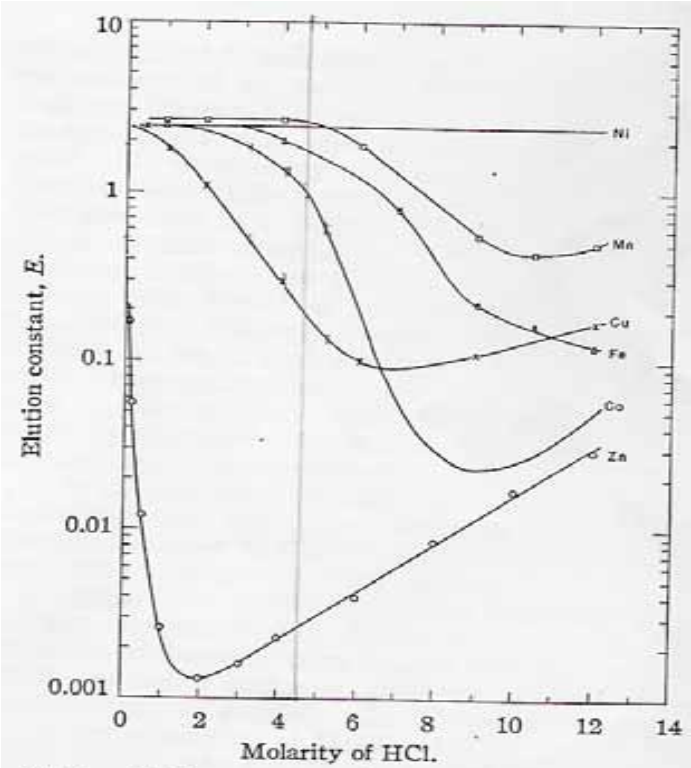


Fig. 1.—Elution constants of some divalent transition elements in hydrochloric acid (data for Zn(II) from unpublished results of F. Nelson).

Many metals can form a anion complex with Cl- in HCl solution (MCl_x^-), so can adsorbed on anion exchange column

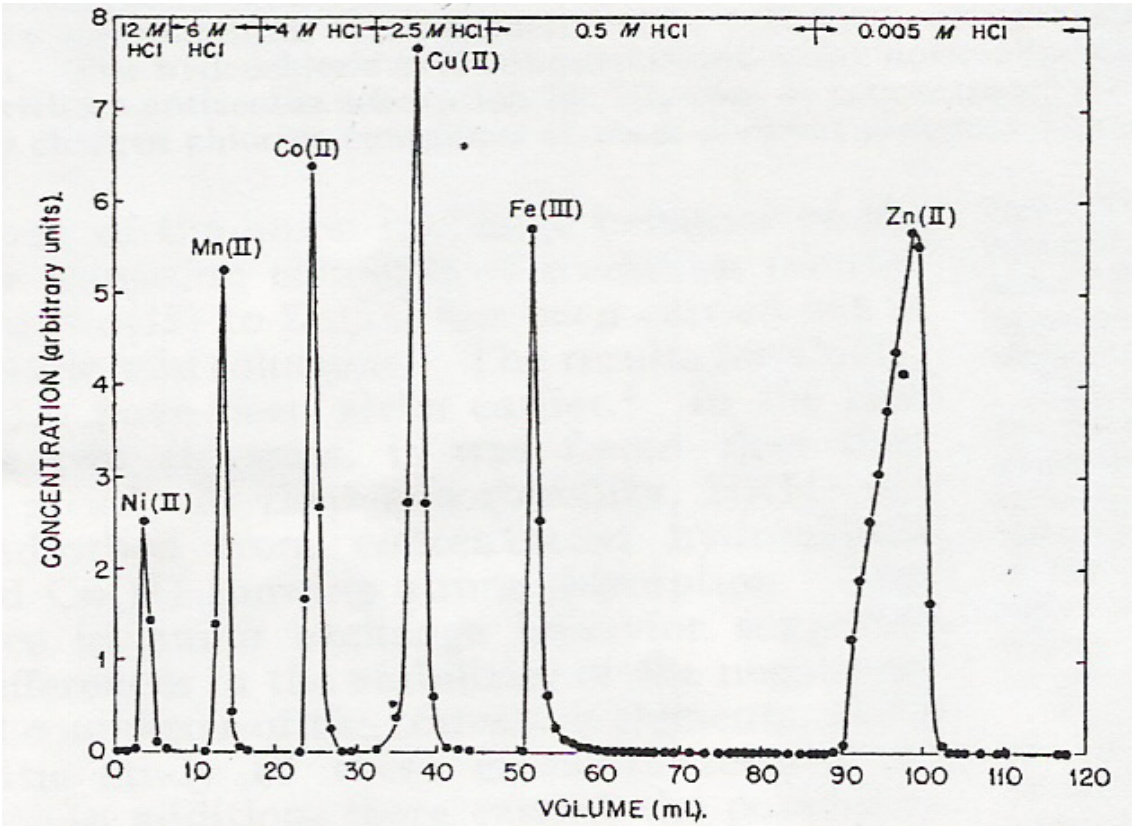
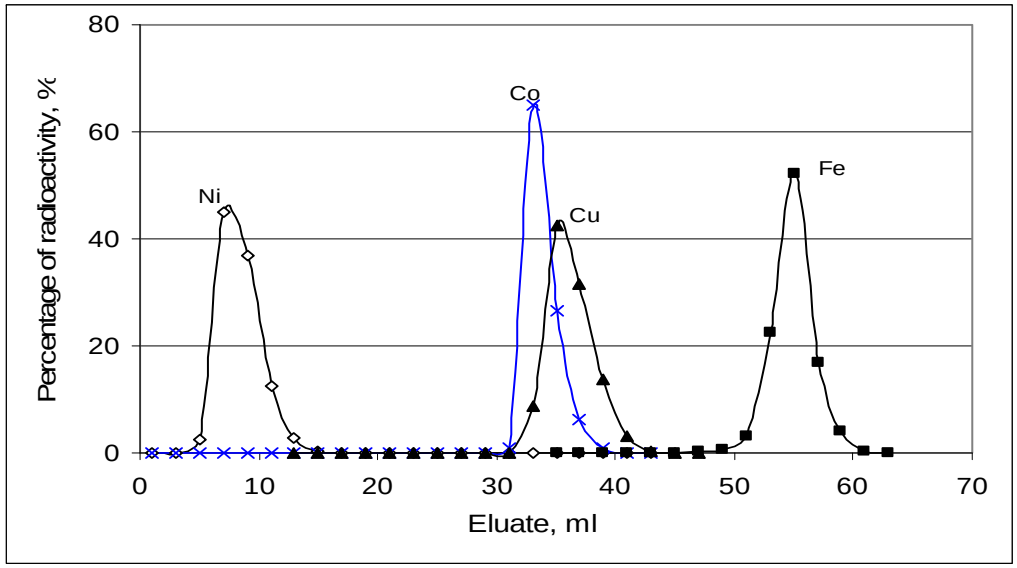
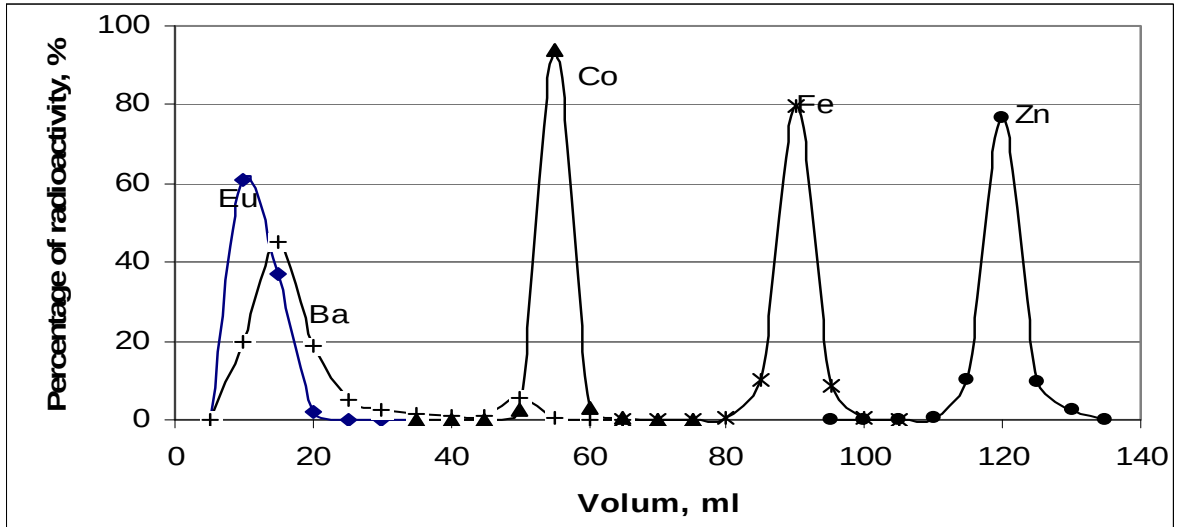


Fig. 2.—Separation of transition elements Mn to Zn (Dowex-1 column; 26 cm. × 0.29 cm.; flowrate = 0.5 cm./min.).

Separation of Fe, Ni, Co, Cu, Eu, Ba by anion exchange chromatography





Separation of Eu, Ba, Co by anion exchange chromatography, Bio-Rad AG1x4, 1x15 cm, 0-40ml:9M HCl, 40-70ml:4M HCl, 70-90ml, 0.05M HCl

- **loading at 9 mol/l HCl, Ni is not absorbed on column, while others are absorbed.**
- **Removing Co, Cu by washing with 4 mol/l HCl**
- **Eluting Fe with 0.5 mol/l HCl**

Separation of Ni and Fe by anion exchange chromatography (conclusion)

Element	Content, %	
	Ni fraction	Fe fraction
Fe^{3+}	<0.001	>98.5
Ni^{2+}	>99.5	<0.001
Co^{2+}	<0.01	<0.1
Ba^{2+}	<7.5	<0.001
Eu^{3+}	>99.8	<0.001
Cs^{+}	>99.5	<0.001
Sr^{2+}	>99.5	<0.001

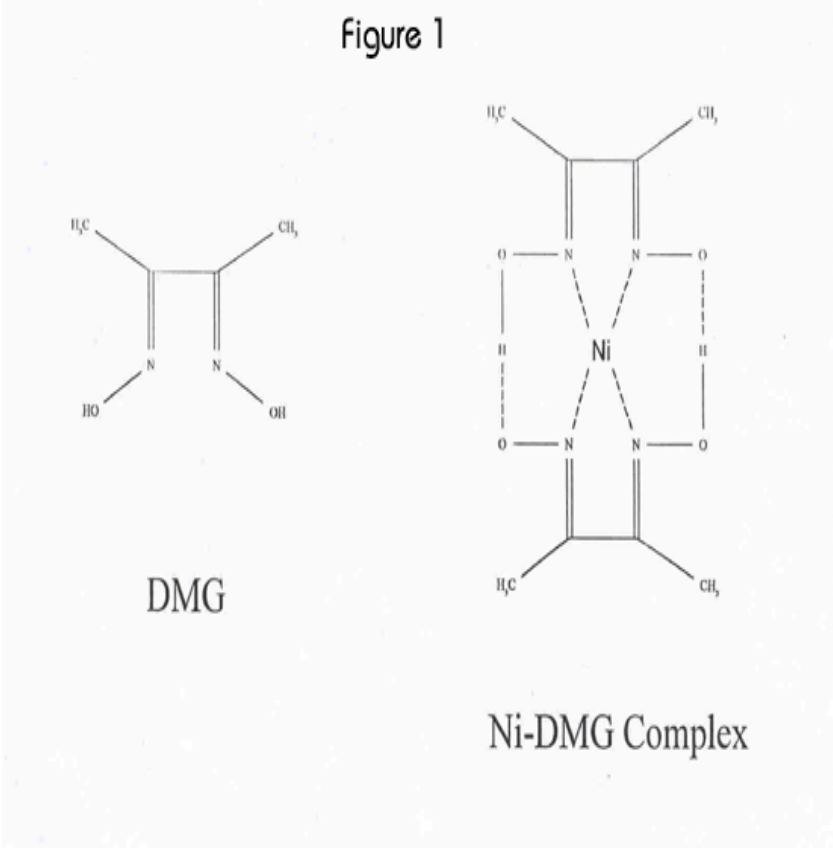
- Ni can be completely separated from Fe, Co, Cu, Zn, U, Pu, etc.
- Fe can be separated from Ni, Cr, Mn, Th, etc.

- Ni cannot be efficiently separated from Cr, Eu, Sm, Mn, V, Sc, Ti, Zr, Ba, Th, Am. Of them, the radioisotopes of Eu, Sm, Ba, Zr, Mn, Cr and matrix elements of Cr, Mn V in metal and alloy seriously interfer the determination of Ni-63.

- Fe cannot be completely separated from Zn, Co, Cu, Pu, Np, especially when a large amount of Fe (>10 mg) is loaded on the column.

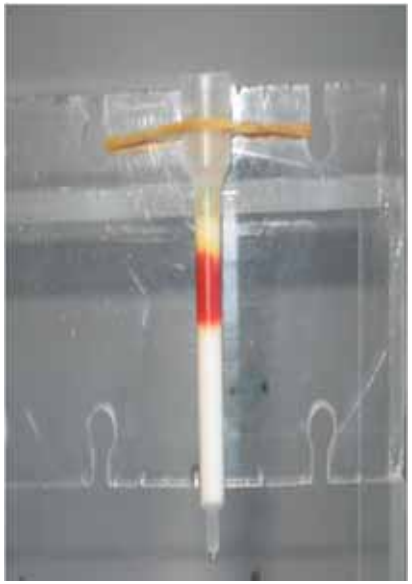
Thus: a further purification for both Ni and Fe is needed.

Separation of Ni using Ni-DMG complex



- Ni can form a stable specific complex with dimethylglyoxime. By Ni-DMG precipitation or organic solvent extraction of Ni-DMG complex at low concentration, Ni can be separated from many other elements.
- While, some other metals, such as Co, Cu can also form a complex with DMG and interferring the separation of Ni.

Formation of M-DMG complex



2mg Ni^{2+}

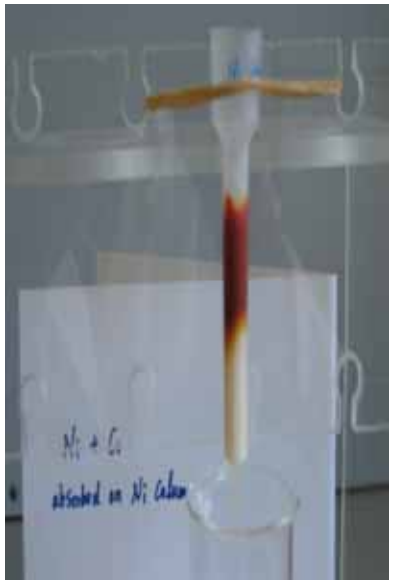
2mg Ni^{2+} +
2mg Co^{2+}

2mg Ni^{2+} +
2mg Cu^{2+}

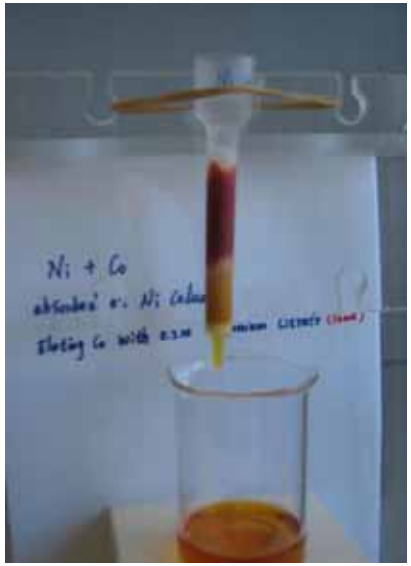
2mg Ni^{2+} +
8mg Fe^{3+}

Purification of Ni by specific Ni-extraction chromatography

The Nickel Resin contains the DMG inside the pores of a polymethacrylate resin. The nickel-DMG precipitate occurs on the resin , where it is held and readily separated from other elements in the supernatant.



1. Loading of solution



2. Washing with 0.2 M ammonium citrate to remove other elements

3. Eluting Ni using HNO3

4. Evaporte eluted Ni-DMG solution to 0.1-0.2 ml for LSC

Purification of Ni by specific Ni-extraction chromatography (conclusion)

Element	Recovery or decontamination factor
Ni^{2+}	> 98.5%
Fe^{3+}	10^4
Co^{2+}	10^3
Ba^{2+}	10^4
Eu^{3+}	10^4
Cs^{+}	10^4
Sr^{2+}	10^4

Ni specific extraction chromatography has a higher decontamination to most of elements, such as Fe, Co, Cu, Cr. Mn, Ba, Eu, transuranics, etc.

•A higher recovery of Ni can be obtained in the procedure.

Separation of Fe by solvent extraction

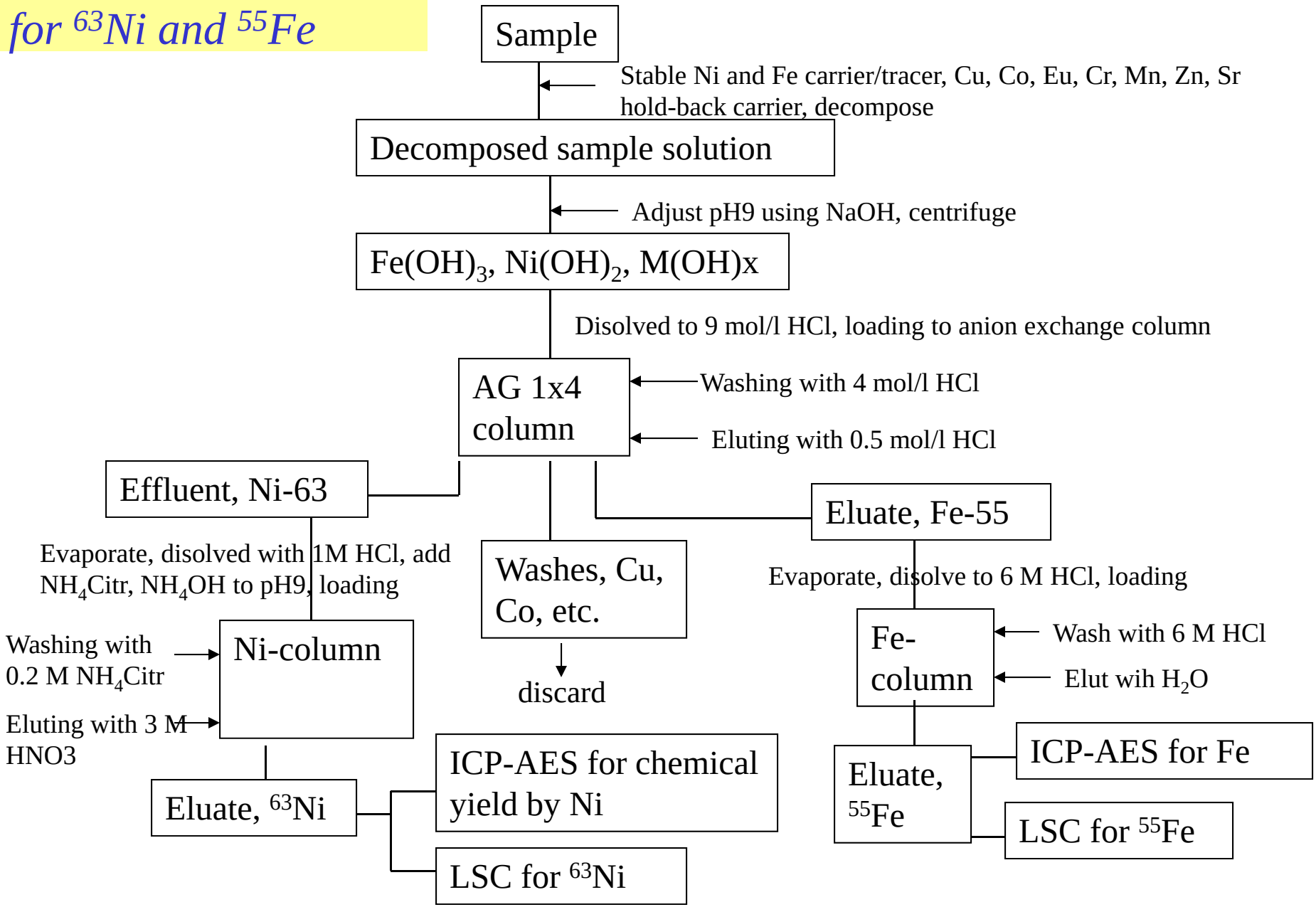
- Di-isopropyl ether (DIPE)
- Methyl-isobutyle (MIBK)
- Ethyl acetate
- Iso-pentanol
- 8-hydroxyquinoline
- Triisooctylamine(TIOA)
- …

Solvent	Medium	Distribution factor
Ethyl acetate	6 mol/l	65
DIBK	6 mol/l	38
DIPE	6 mol/l	4

Purification of Fe by extraction chromatography

Column	capacity	Fe-specific
TRU	3 mg	no
Silica-immobilised formylsalic acid	54 mg	no
XAD-7/DIBK	1700 mg	yes

Analytical procedure for ^{63}Ni and ^{55}Fe



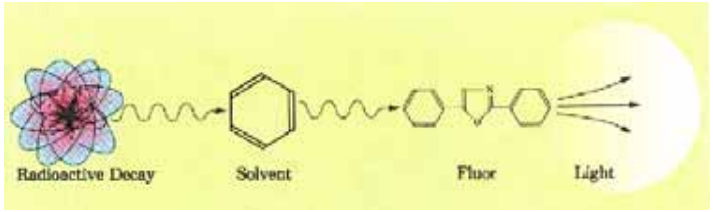
The recovery of Fe and Ni and decomtamination factors for main interferring radionuclides

Interference	Recovery/decontamination factor		Interference	Recovery/decontamination factor	
	Fe fraction	Ni fraction		Fe fraction	Ni fraction
^{55}Fe	85-95%	$>10^5$	^{133}Ba	$>10^6$	$>10^5$
^{63}Ni	$>10^5$	80-95%	$^{134,137}Cs$	$>10^6$	$>10^6$
$^{58,60}Co$	$>10^5$	$>10^5$	$^{89,90}Sr$	$>10^6$	$>10^6$
$^{152,154}Eu$	$>10^6$	$>10^5$	$^{41,45}Ca$	$>10^6$	$>10^6$
^{151}Sm	$>10^6$	$>10^5$	^{36}Cl	$>10^6$	$>10^6$
^{54}Mn	$>10^5$	$>10^6$	^{3}H	$>10^6$	$>10^6$
^{51}Cr	$>10^6$	$>10^5$	^{14}C	$>10^6$	$>10^6$

For all interferring radionuclides, the decontamination factors higher than 10^5.

Meaurement of ^{63}Ni and ^{55}Fe by LSC

• In LSC, the radionuclide is mixed with a cocktail, the decay energy will be transferred to the cocktail, and converted to photons, by counting the photons using a PMT(photomultoplier tube), the activity of radionuclides is measured.



• A single photon striking the photocathode has at most a 30% probability of producing an electrical pulse. There are always losses associated with the full light generation process. The factors which effect the energy transfer process is called quench.

Chemical Quenching

Any compound that does not have an aromatic structure will produce some quenching effect.

Physical Quenching

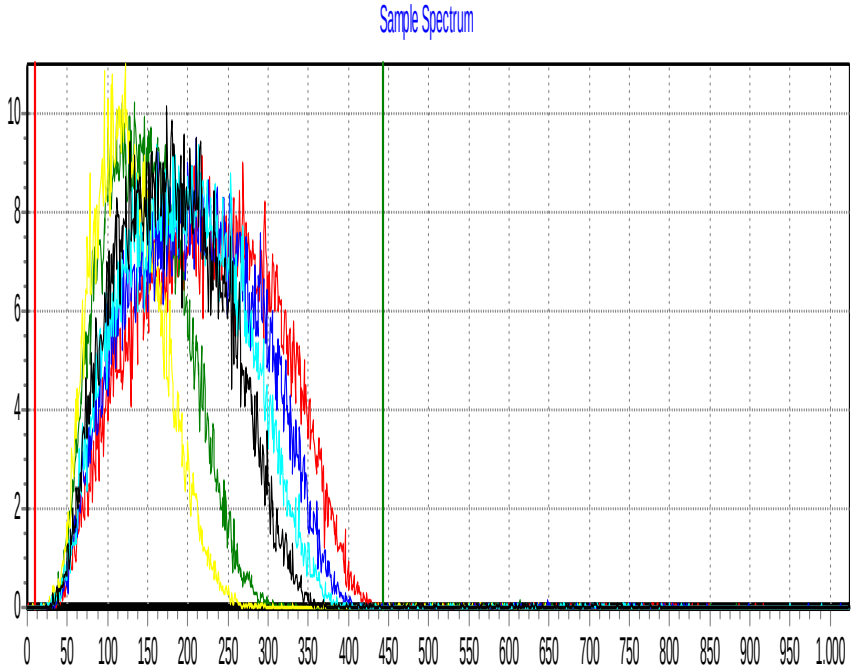
It occurs when a barrier impairs contact between the radioactive particle and the scintillator solution, or when the photons of light generated are absorbed by some solid within the vial.

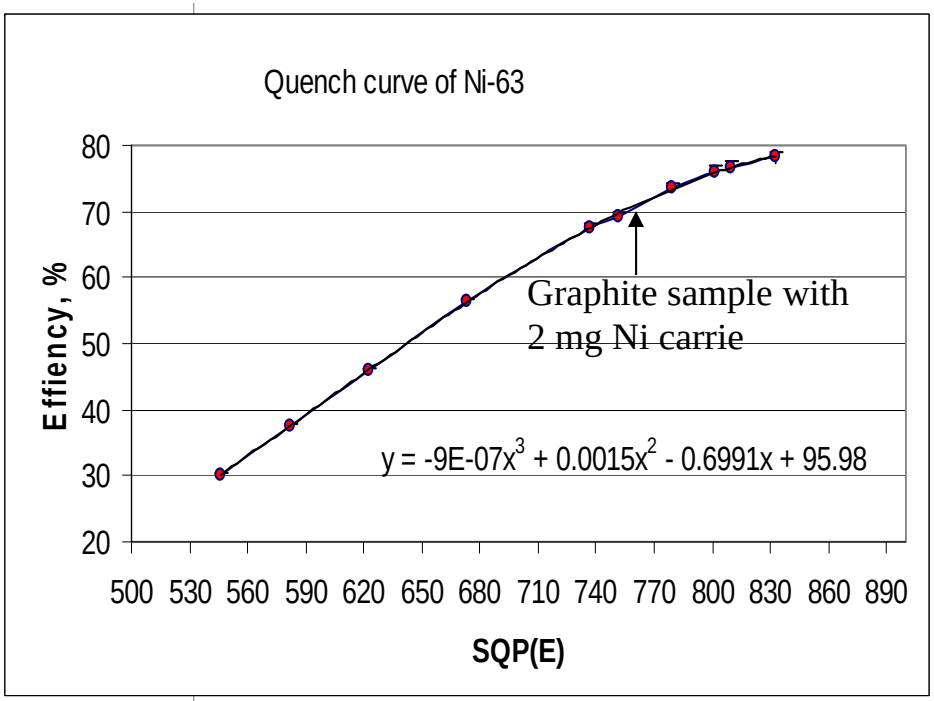
Colour Quenching

light absorbing compounds interpose and lessen the number of photons leaving the scintillation vial. Fluorescence emission takes place in the blue region of the spectrum, therefore the order of severity of colour quenching is:

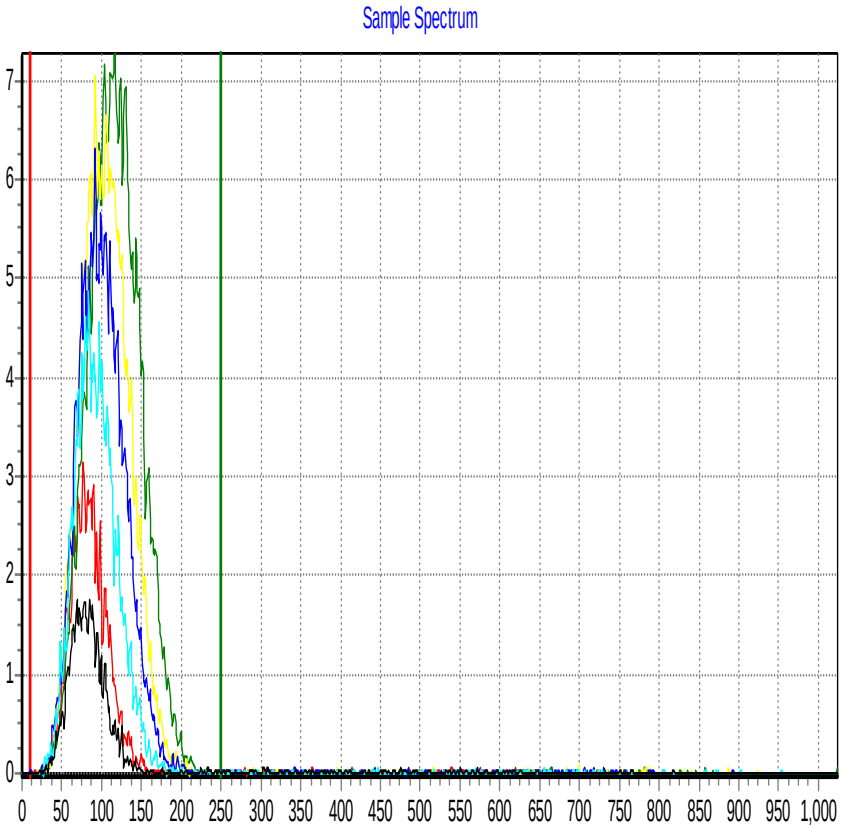
Red > orange > yellow > green > blue

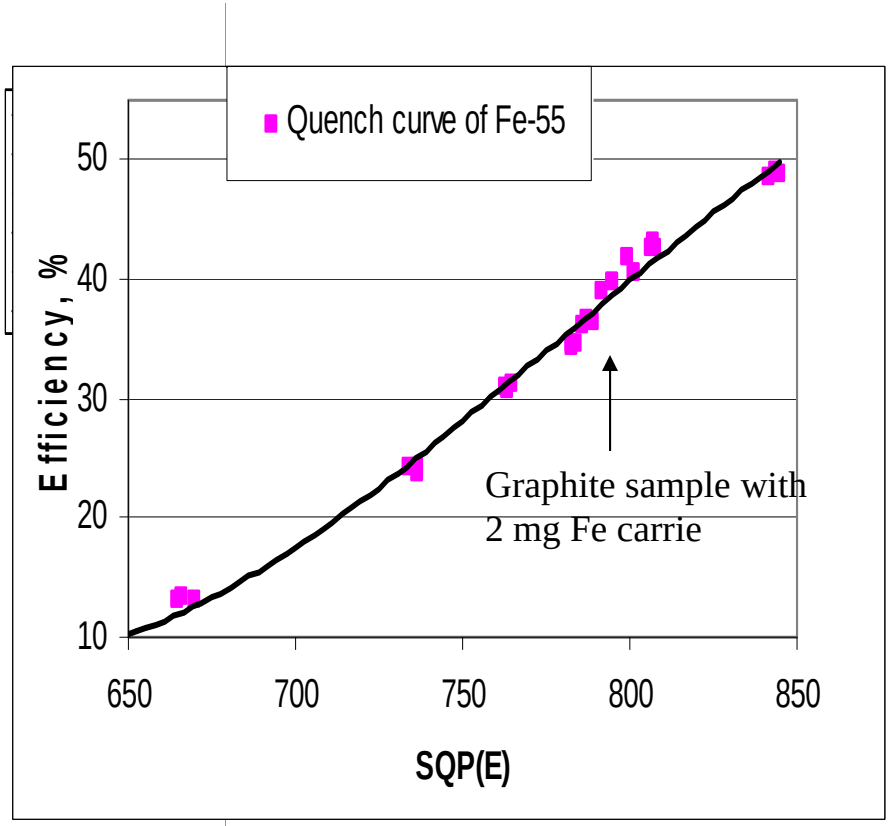
Quench correction for Ni-63





Quench correction for Fe-55

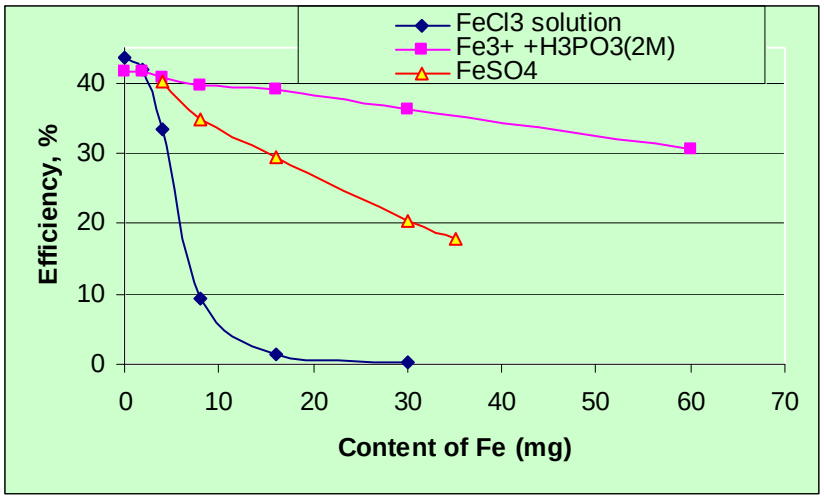




Preparation of separated ^{63}Ni for LSC

- ^{63}Ni was separated in 10-15 ml of 3 mol/l HNO_3 eluate, it cannot be used for LSC, due to large volume and high acid concentration. Because:
  - 20 ml counting vial with high capacity scintillation cocktail can only mix with less than 1.5 ml of 3 mol/l HNO_3 eluate.
  - Acid solution is a high quench regent, it seriously reduce the counting efficiency of LSC. In 1.5 ml HNO_3 eluate, the counting efficiency for ^{63}Ni is only 10%
  - The Energy of beta particle emitted from ^{63}Ni is low (66.95 keV).
- The eluate can be evaporated to small volumn (<0.2 ml) at low temperature (<150 ^{o}C) and transfer to counting vial with 0.5 ml water. In this case, the counting efficiency for ^{63}Ni can be increased to 70%.
- It is very important, do not evaporate eluate to dryness.
  - Low boiling point of $Ni(NO_3)_2$ (137 ^{o}C) makes >95% loss of ^{63}Ni when evaporating sample to dryness.
  - Burned Ni-DMG complex is difficult to be decomposed to obtain a clear solution.

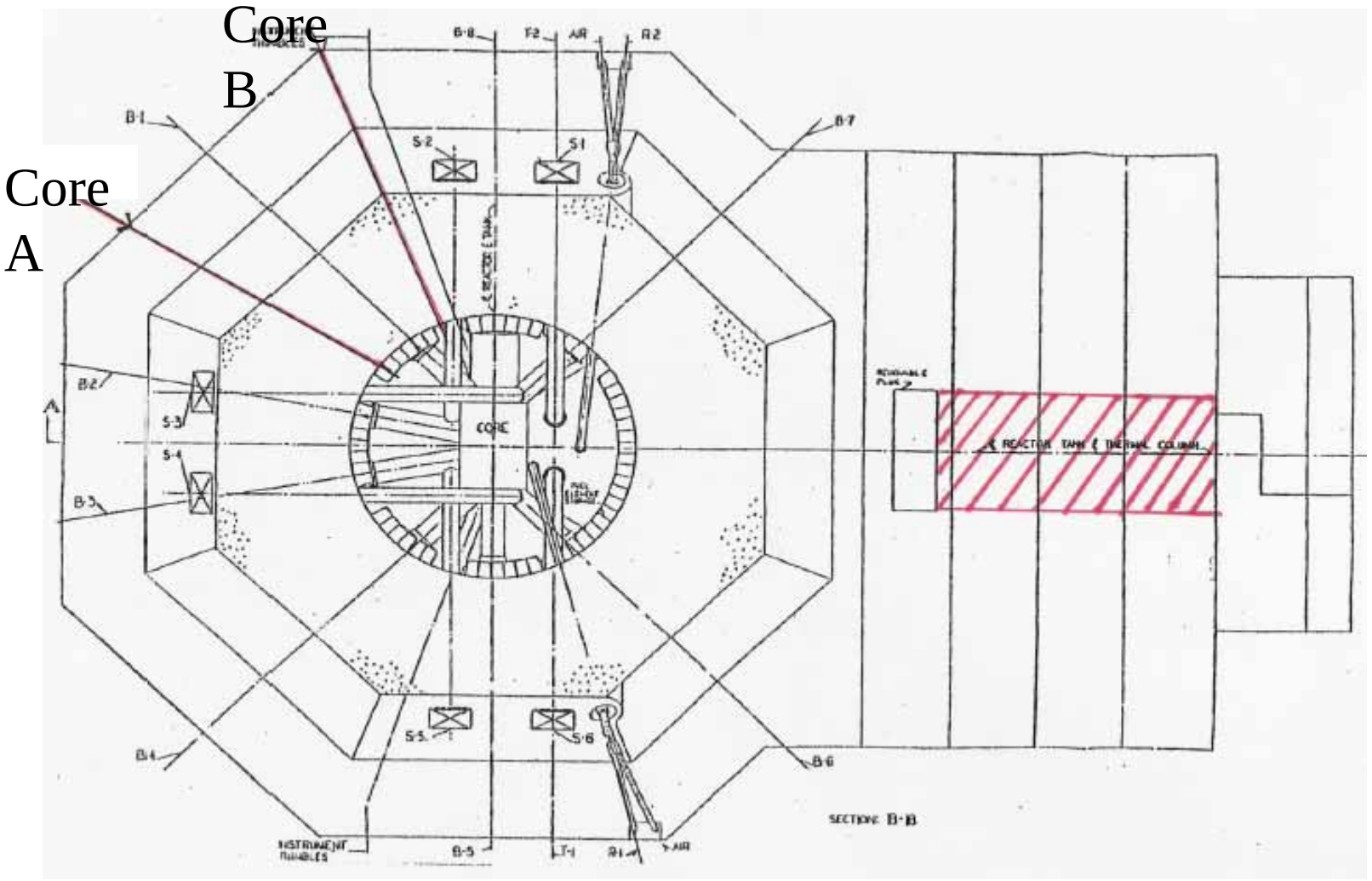
Preparation of separated ^{55}Fe for LSC



- Yellow colour Fe^{3+} is a very effective quenching agent
- Reduction of Fe^{3+} to Fe^{2+} using suitable reductant, such as ascorbic acid can reduce the quench, but Fe^{2+} is not stable and can be oxidize to Fe^{3+} again, and Fe^{2+} also has some colour quench.
- Solvent extraction of Fe^{3+} using some organic compounds such as di-2-ethylhexyl phosporic acid can reduce the Fe^{3+} colour quench, but not effective for large Fe content sample.
- In H_3PO_3 solution, a stable and colourless Fe- H_3PO_3 complex can be formed, therefore can significantly reduce the Fe^{3+} colour quench.
- H_3PO_3 as an acid has less quench and high solubility with scintillation cocktail, therefore can significantly improve the counting efficiency.
- As high as 40% counting efficiency of ^{63}Ni in 1.5 ml of 2 mol/l H_3PO_3 solution.
- The separated Fe was evaporated to dryness and then dissolved in 2 M H_3PO_3 solution in this work, for less than 10 mg Fe, 40% counting efficiency was obtained, and for 200 mg Fe, the efficiency is still as high as 15%.

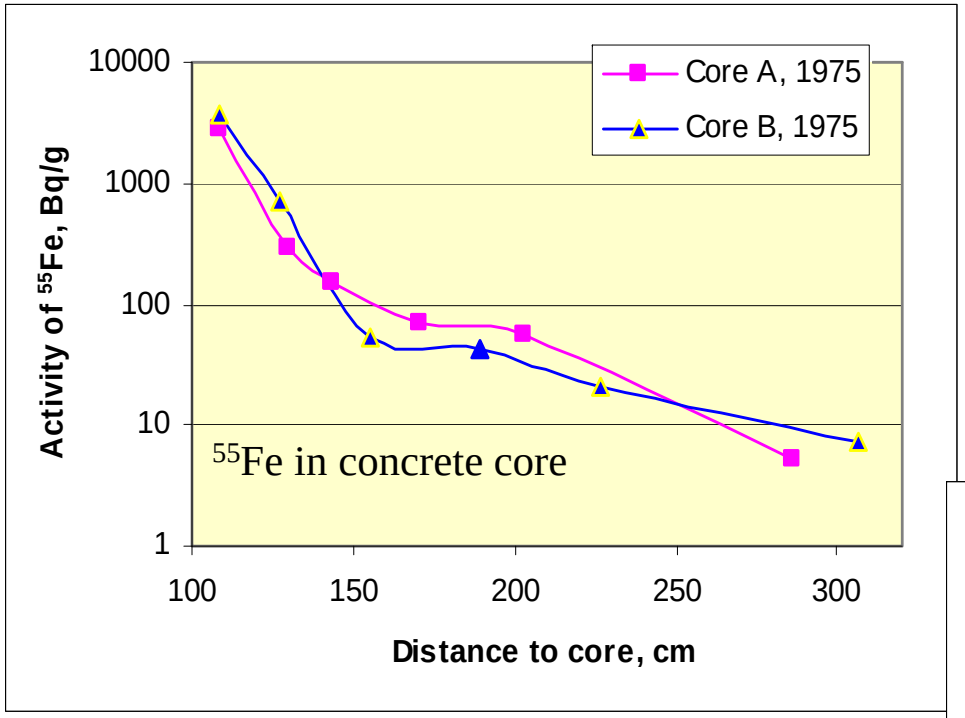
Determination of ^{63}Ni and ^{55}Fe in graphite samples

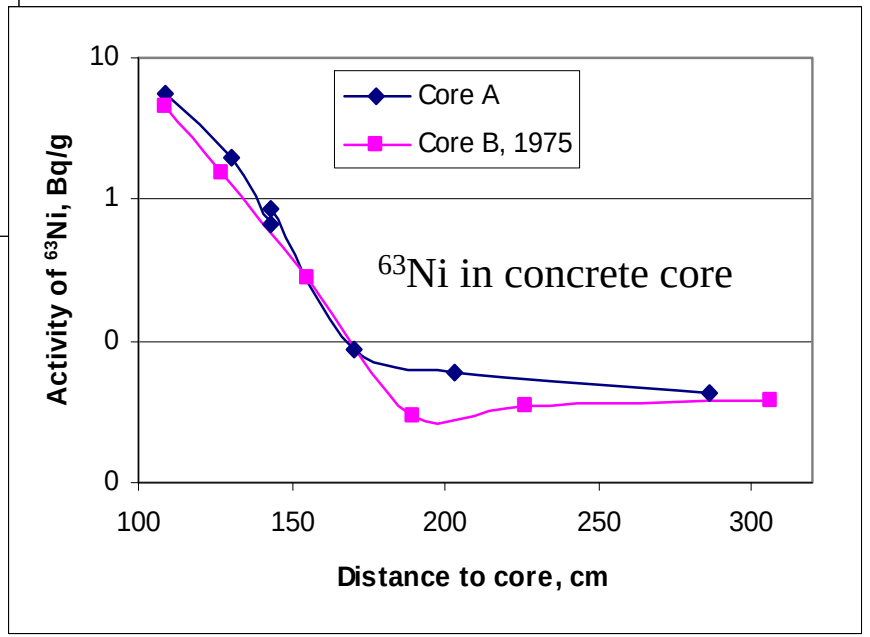
Sample No	Sample and Source	^{55}Fe		^{63}Ni	
		Recovery,%	Bq/g	Recovery,%	Bq/g
DR-3-T	Graphite from DR3	92.2	545000	94.63	5552
Ly7.5	Graphite from DR-2	90.4	0.53	93.89	92.5
Ly5.5	Graphite from DR-2	90.6	1.05	93.74	22.3
Yi7.5	Graphite from DR-2	92.5	1.92	93.35	7.71
Yi5.5	Graphite from DR-2	91.3	9.21	91.56	43.1
B-6	Heavy concrete from DR-2	92.3	0.015	90.34	0.022



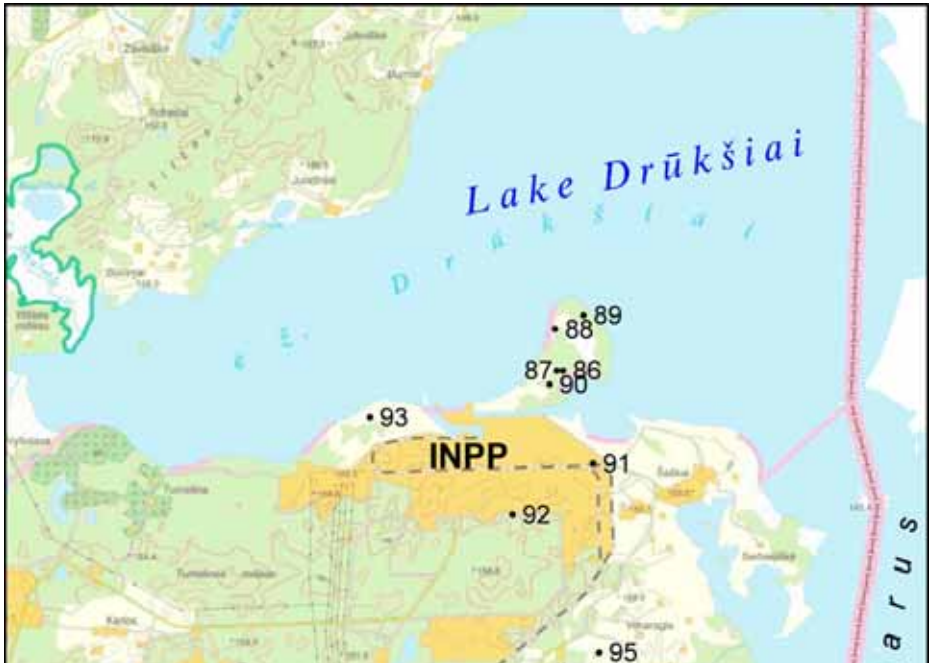
Sampling of concrete and graphite from Danish reactor, DR-2

Results of ^{55}Fe, ^{63}Ni in concrete from Danish reactor, DR-2





^{55}Fe and ^{63}Ni in the environment surrounding Ignalina NPP

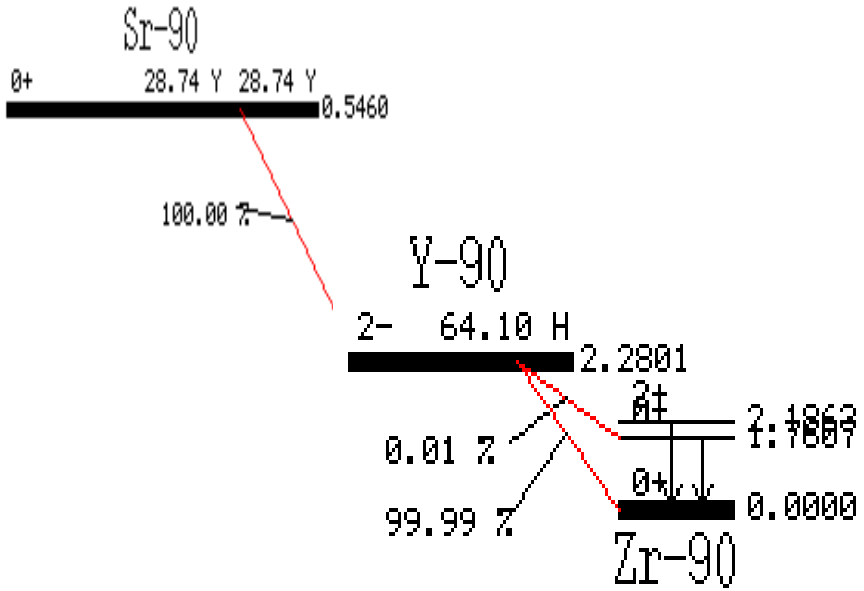


Surface deposition density levels of radionuclides

Moss sample code	Surface deposition density, Bq/m^2 (1σ)				
	^{137}Cs	^{60}Co	^{40}K	^{55}Fe	^{63}Ni
86	31 ± 3	1.4 ± 0.2	47 ± 4	36 ± 6	–
87	46 ± 4	5.2 ± 0.4	76 ± 6	17.3 ± 2.6	–
88	4.7 ± 0.4	2.5 ± 0.2	114 ± 9	70 ± 11	–
89	8.6 ± 0.7	0.9 ± 0.1	44 ± 4	13.5 ± 2.1	–
90	13.6 ± 1.1	3.4 ± 0.3	77 ± 6	106 ± 16	2.0 ± 0.3
91	1.6 ± 0.2	0.8 ± 0.1	55 ± 4	110 ± 17	–
92	1.9 ± 0.2	0.7 ± 0.1	47 ± 4	222 ± 34	–
93	1.7 ± 0.2	43 ± 4	53 ± 5	940 ± 140	–
95	13.4 ± 1.1	0.7 ± 0.1	80 ± 7	200 ± 30	–
9	20.8 ± 1.3	–	55 ± 4	–	–

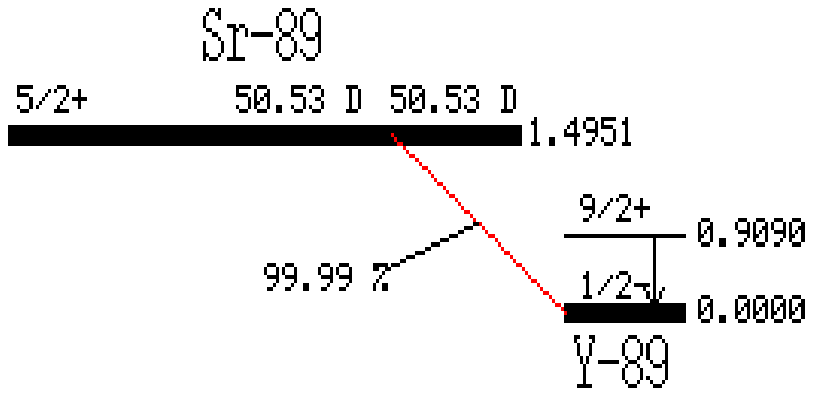
^{90}Sr Analysis

- **One of main fission products (Y=5.8%)**
- **Beta emitter, measured by GM detector or LSC.**
- **It has to be separated from other radionuclides before measurement.**
- **For some waste, it may contain ^{89}Sr (50.5 d), it can be also measured by LSC with ^{90}Sr.**



^{89}Sr properties

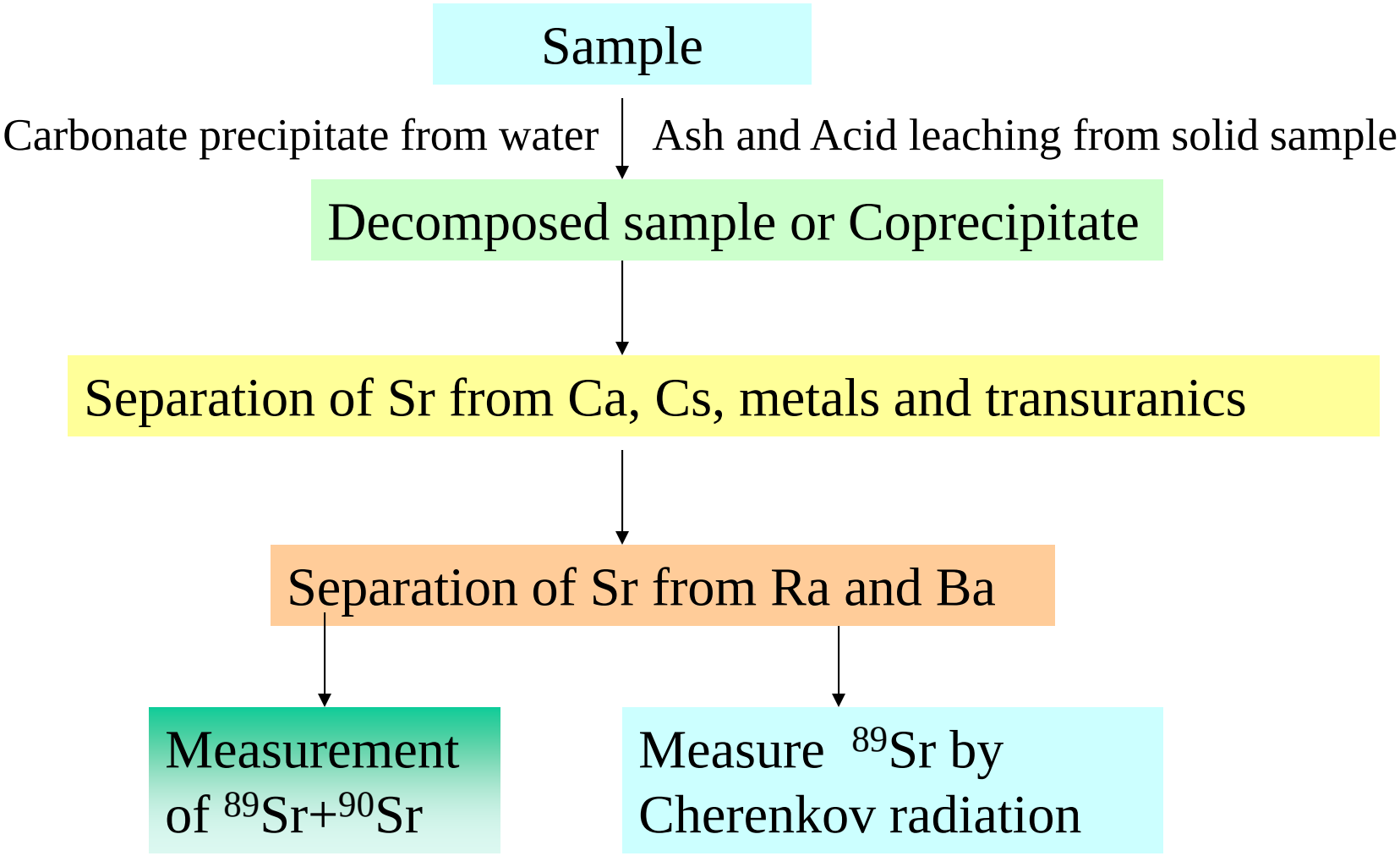
- **^{89}Sr: fision product, Y=4.73%, Beta emitter**



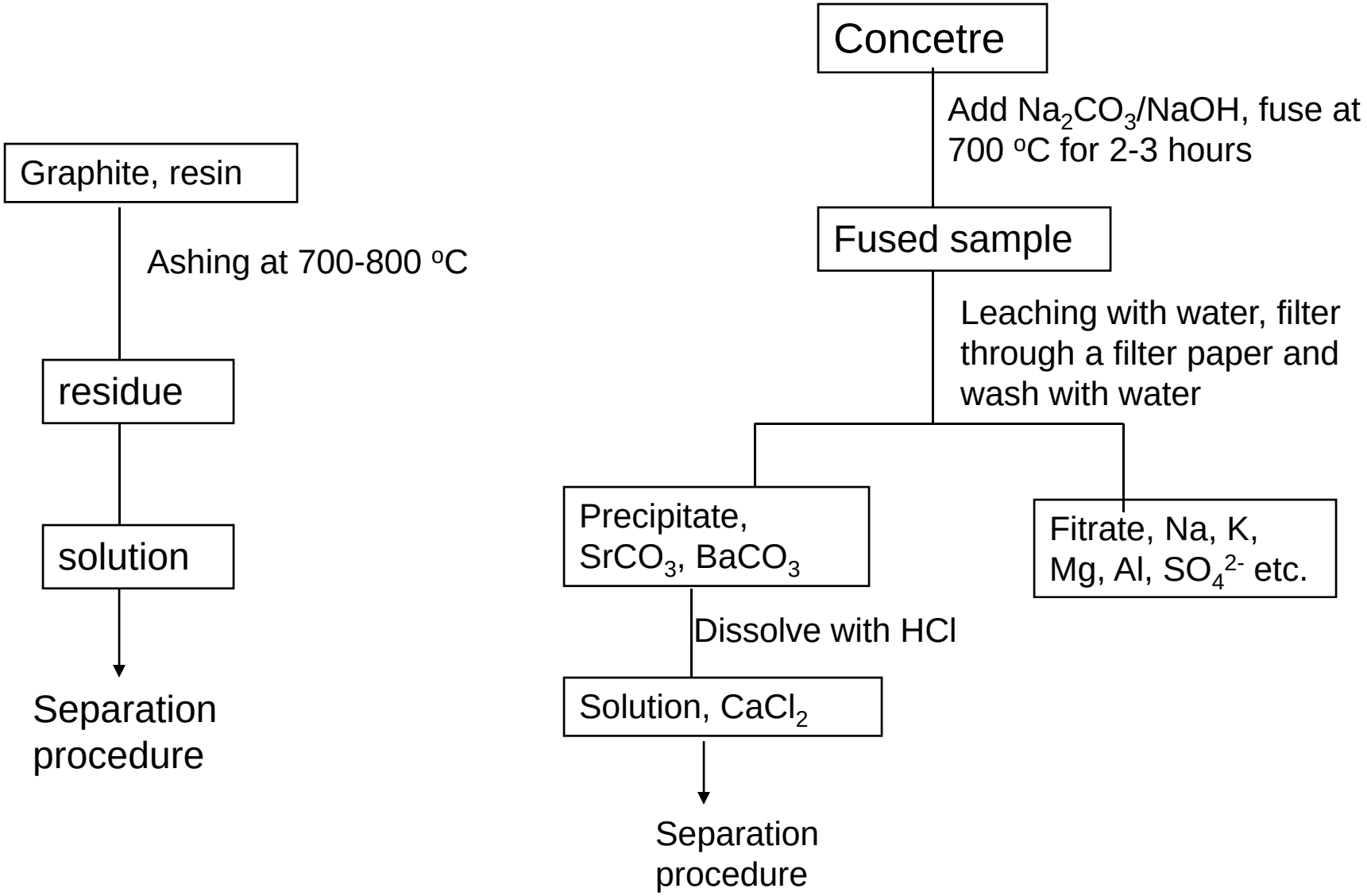
Gammas from ^{89}Sr (50.53 d)

Eγ (keV)	Iγ (%)	Decay mode
908.96	0.010	β^-

Analytical Procedure for ^{90}Sr and ^{89}Sr



Decomposition of solid sample



Methods for the Separation of Sr

- Solvent extraction
- Liquid membrane extraction
- Extraction chromatography (Sr-Spec resin)
- Ion-exchange chromatography
- Strontium rhodizonate precipitation
- $Sr(NO_3)_2$ precipitation in 70% HNO_3

Note: These procedures cannot be used for separation of Sr from a large amount of Ca)

Solubilities of Ca, Sr,Y, Ba and Ra compounds

$Ca(OH)_2$: insoluble, Ksp = 5.2×10^{-6}

$Sr(OH)_2$: Soluble in alkine solution

- $SrCl_2$, $BaCl_2$ and $RaCl_2$: soluble in water
- $SrCl_2$: soluble in HCl<9.5 mol/L solution
- $BaCl_2$ and $RaCl_2$: insoluble in HCl >9 mol/l solution

$Y_2(SO_4)_3$: soluble in water

$Sr(Ba, Ra)SO_4$: insoluble in water

A new method for the separation of Sr

Separation of Sr from Ca:

$Ca(OH)_2$ precipitation

Separation of Sr from Ba and Ra

$Ba(Ra)Cl_2$ precipitation in concentrated HCl solution

Separation of ^{90}Y from Sr, Ra and Ba

$Sr(Ra, Ba)SO_4$ precipiatation

Sepation of Sr from Ca by $Ca(OH)_2$

^{85}Sr added (Bq)	Ca added (g)	^{85}Sr (Bq)		Recovery of Sr (%)	Ca in supernatant (g)	Ca decontamination (%)
		Precip.	Supern.			
1050	50.00	31.6	1058.4	97.0	0.18	99.5
1050	31.00	38.1	1037.7	96.4	0.14	99.5
1050	10.00	30.6	1029.6	97.1	0.07	99.3

* Concentration of NaOH in the solution: 0.5 mol/L

Separation of Sr from Ba by $BaCl_2$ and $BaCrO_4$

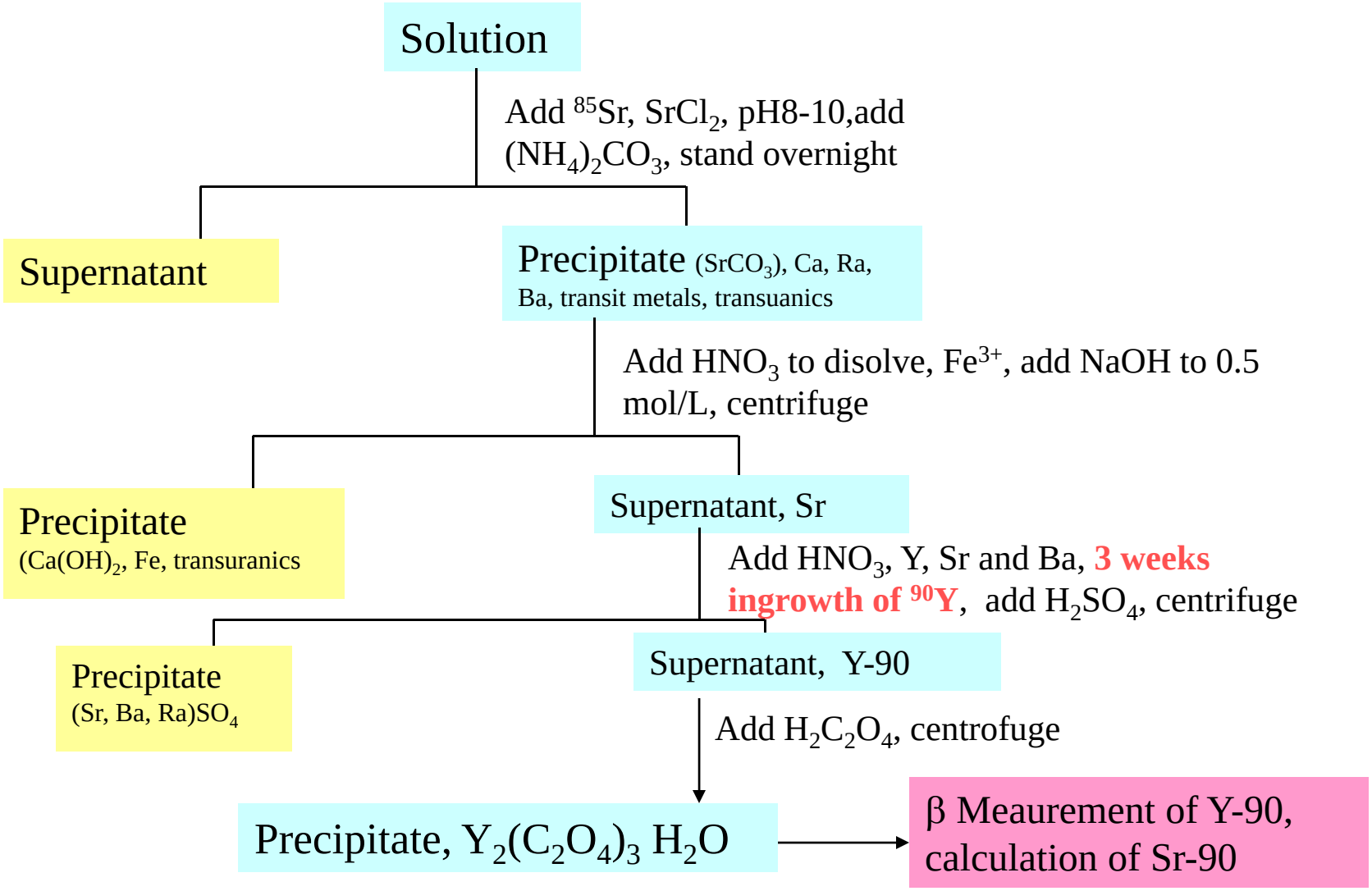
[HCl], mol/l	Tracers added (Bq)		Supernat. ^{133}Ba (Bq) *	Precipitate ^{85}Sr (Bq) *	Recovery of Sr, %	Decontam. of Ba, %
	^{85}Sr	^{133}Ba				
8.5	**1050**	**1510**	**918.8±14.2**	**2.0±3.4**	**99.8±0.3**	**40.0±1.3**
9.0	**1050**	**1510**	**484.7±18.4**	**2.1±2.4**	**98.8±0.3**	**67.9±1.8**
9.5	**1050**	**1510**	**43.8±9.4**	**62.4±5.9**	**94.1±0.6**	**97.1±1.4**
10.0	**1050**	**1510**	**40.5±7.2**	**331.2±11.1**	**68.5±1.5**	**97.3±1.3**
10.5	**1050**	**1510**	**28.6±4.2**	**743.4±18.4**	**29.5±1.3**	**98.1±2.7**
11.0	**1050**	**1510**	**7.9±4.8**	**846.3±21.2**	**21.1±1.1**	**99.5±1.3**
$BaCrO_4$	**1050**	**1510**	**15.3±5.7**	**49.0±7.8**	**95.3±0.7**	**99.0±0.4**

* Average and standard deviation of two determinations

Separation of Y from Ra, Sr, and Ba by sulphate precipitation

Tracer	Added	Supernat.	Precipitate	Recovery of Y, %	Decontam.of Sr, Ba, Ra %
Y, mg	11.10	10.95±0.22		98.6±2.0	
^{85}Sr, Bq	1050	8.7±2.4	1042±38		99.2±0.2
^{133}Ba, Bq	1510	2.2±1.8	1511±21		99.9±0.1
^{226}Ra, Bq	33.0	0.14±0.26	32.87±0.47		99.6±1.4

Separation procedure of ^{90}Sr

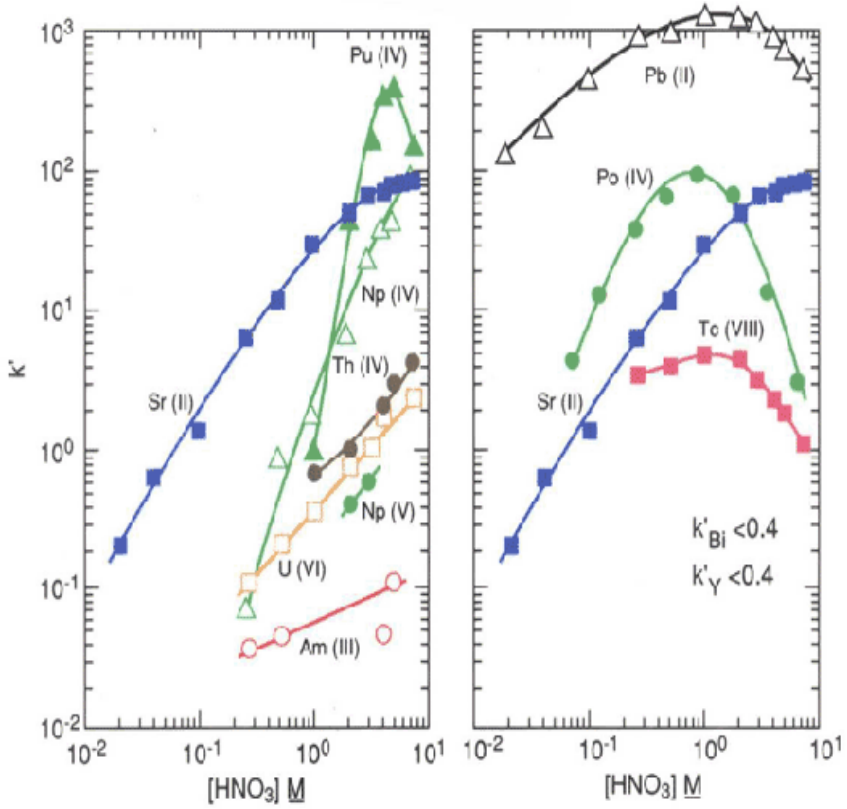


Chen, Hou, Nielsen, Dahlgaard, Yu, Anal. Chim. Actc, 466 (1): 109-116, 2002

Sr-Resin

Figures 4 and 5

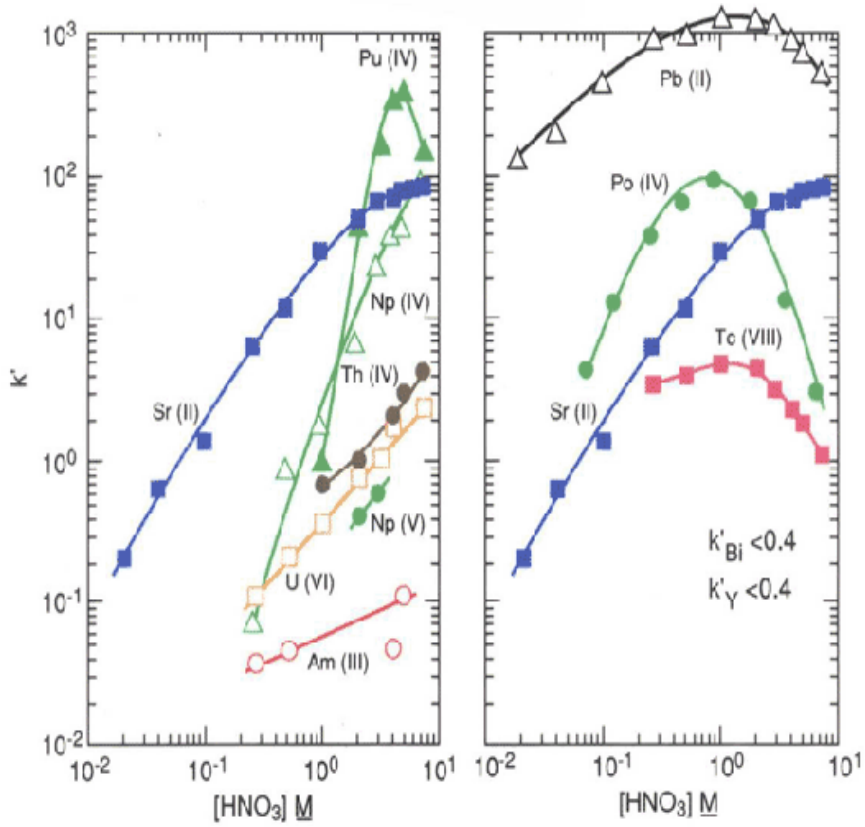
Acid dependency of k' for various ions at 23-25°C.
Sr Resin



Horwitz (HP199)

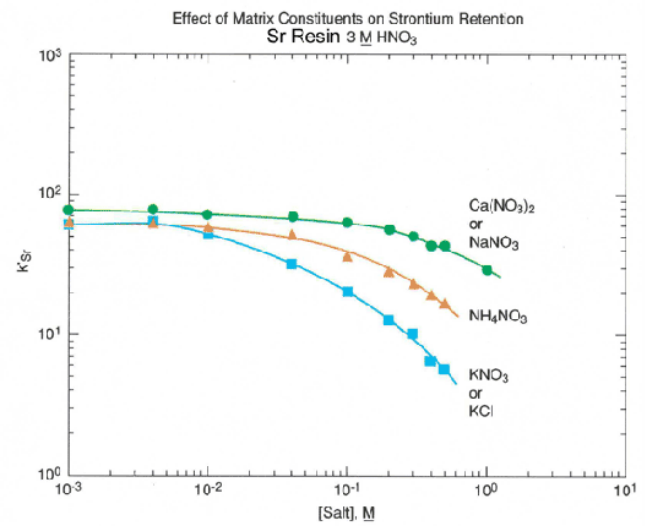
Figures 4 and 5

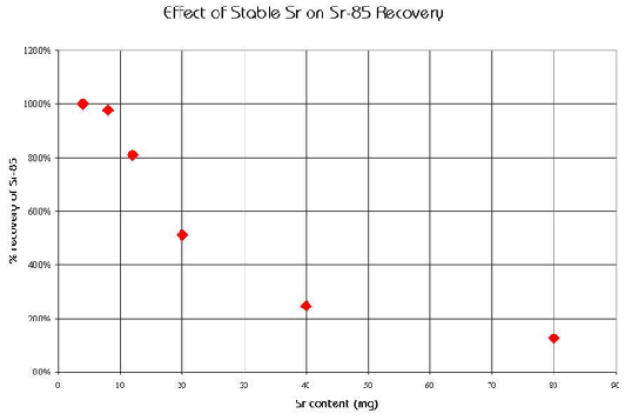
Acid dependency of k' for various ions at 23-25°C.
Sr Resin



Horwitz (HP199)

Separation of Sr using Sr-resin





Solution

Add Sr, Y, Ba, pH8-10, add Na_2CO_3, heat, cool to room temperature

Precipitate
($SrCO_3$), Ra, Ba, transit metals, transuanics

Supernatant

Add HNO_3 to 8M

Sr-column

Wash with 8M HNO_3

Elute with 0.05M HNO_3

Effluent

Add Scintillation cocktail

LSC for ^{90}Sr (no ^{89}Sr)

Measure immediately

LSC for Cherenkov radiation of ^{89}Sr

Measure after >10 days for ingrowth of ^{90}Y

LSC for Cherenkov radiation of $^{89}Sr+^{90}Sr/^{90}Y$

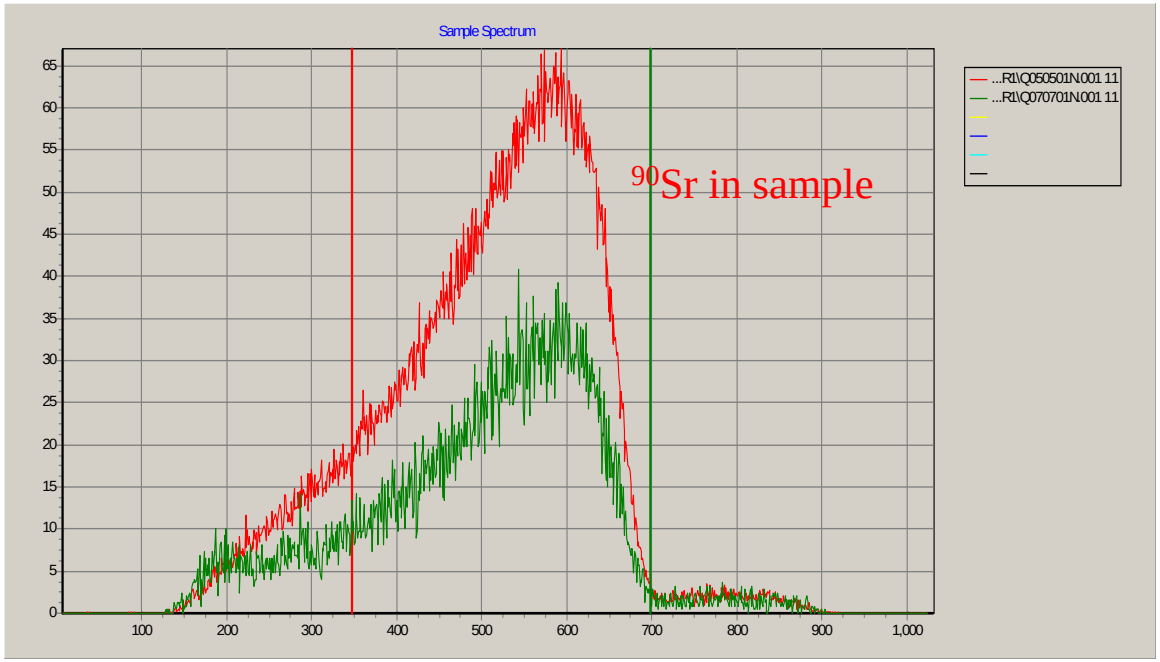


Fig.1 Beta spectrum of Sr sample separated from DR1 core solution.

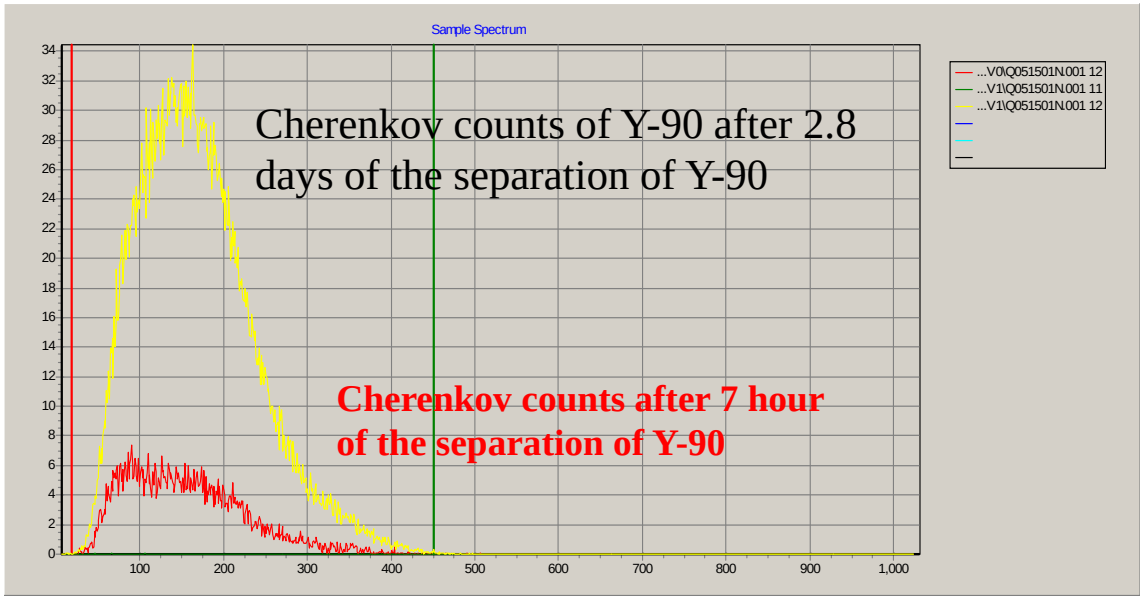
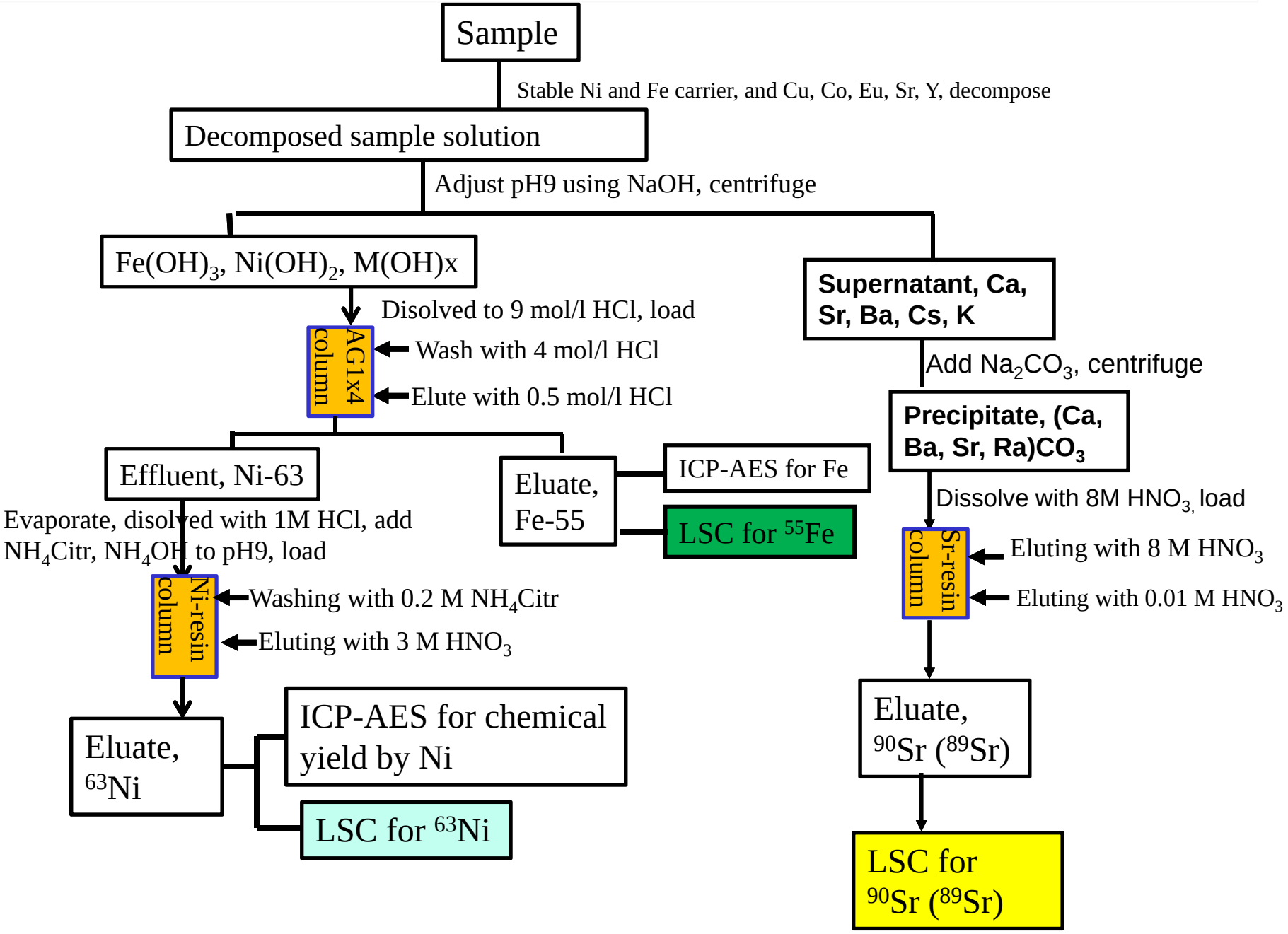


Fig.2 Cherenkov radiation of Sr sample separated from one DR1 core solution sample

Procedure for simultaneous determination of ^{55}Fe, ^{63}Ni, and ^{90}Sr



Summary

- A analytical method based on radiochemical separation and liquid scintilation counting was developed for the determination of ^{63}Ni and ^{55}Fe in nuclear waste and environmental samples.
- Nickel and iron are separated from matrix and interferring nuclides by a procedure combining hydroxidesprecipitation, anion exchange and extraction chromatography.
- The recoveries of Fe and Ni are 80-95%. The decontamination factors for most of interferring nuclides are higher than 10^5 . The detection limit for ^{63}Ni and ^{55}Fe are 0.015 Bq and 0.035 Bq respectively.
- Some graphite , concrete and sediment have been analysed for the ^{63}Ni and ^{55}Fe.
- By combining with a $SrCO_3$ preciptiation and Sr-column extraction chromatographic separation, ^{89}Sr and ^{90}Sr can be also determined using the same sample.