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# Methods for determination of strontium-90 in food and environmental samples by Cerenkov counting

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**Sammanfattning / Abstract:**

The procedure for determination of strontuim-90 (=yttrium-90) by HDEHP-extraction of yttrium-90 and counting the Cerenkov radiation in a liquid scintillation counter, is applicable for all kind of samples. Prior to analyses liquid samples are evaporated to dryness and ashed.
Environmental and food samples are oven dried and ashed.

The ash is dissolved in 1 molar hydrochloric acid and, at pH 1.0-1.2 the yttrium-90 is extracted from the solution with 10% HDEHP. All mono and divalent ions will stay in the acid phase. Yttrium-90 is backextracted into 3 molar nitric acid and precipitated as hydroxide. The hydroxide precipitation is dissolved in 1 ml conc nitric acid, transferred to a liquid scintillation vial and the Cerenkov radiation from yttrium-90 is counted in a liquid counter.

**Nyckelord** (valda av författaren) / **Key words** (chosen by the author):

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Division of Radiochemistry
and
Radioecology

# Methods for determination of strontium-90 in food and environmental samples by Cerenkov counting

by

Jorma Suomela, Lena Wallberg and Judith Melin

# DETERMINATION OF STRONTIUM-90 IN FOOD AND ENVIRONMENTAL SAMPLES

Two methods are described in this paper. The first is a standard method to be used in the absence of shortlived activation and fission products. The second a complementing procedure to be used in the presence of shortlived activation and fission products.

## Principle

The determination of strontium -90 in equilibrium with yttrium -90, is accomplished by monitoring the Cerenkov radiation of high energetic beta particles (2.27 MeV ) from yttrium -90 in a liquid scintillation counter. Yttrium -90 is the decay product of strontium -90. The chemical yield of yttrium -90 is determined by adding a known amount of inactive yttrium carrier. The amount of yttrium recovered is determined by acidimetric titration of the sample in the scintillation vial with "Titriplex III."

According to the standard procedure, interfering nuclides such as uranium, thorium, radium and their decay products as well as isotopes of cesium, potassium and strontium are separated from the sample by an extraction with HDEHP .

In cases of a nuclear accident or a nuclear weapon explosion a release of short lived fission and activation products to the environment can be expected. Some of these radionuclides , mainly lanthanides and actinedes, will form complexes with HDEHP and consequently interfere with the subsequent counting of the Cerenkov radiation from yttrium -90. The interference of short lived fission and activation products can be overcome by one additional extraction with HDEHP (20 % ) before counting. The presence of interfering short lived nuclides can be identified by gamma spectrometric analyses.

By using the following procedures and a low level LSC a lower limit of detection of 10 mBq / sample can be reached.

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METHOD II ( presence of short lived activation and fission products)
Determination of Strontium-90 in food and environmental samples in the presence of short lived activation and fission products

# DETERMINATION OF STRONTIUM-90 IN FOOD AND ENVIRONMENTAL SAMPLES IN THE ABSENCE OF SHORT LIVED ACTIVATION AND FISSION PRODUCTS

## 1. Summary

The procedure for determination of strontium-90 (=yttrium-90 ) by HDEHP-extraction of yttrium-90 and counting the Cerenkov radiation in a liquid scintillation counter, is applicable for all kind of samples. Prior to analyses liquid samples are evaporated to dryness and ashed. Environmental and food samples are oven dried and ashed.

The ash is dissolved in 1 molar hydrochloric acid and, at pH 1.0-1.2 the yttrium-90 is extracted from the solution with 10% HDEHP. All mono and divalent ions will stay in the acid phase. Yttrium-90 is backextracted into 3 molar nitric acid and precipitated as hydroxide. The hydroxide precipitation is dissolved in 1 ml conc nitric acid, transferred to a liquid scintillation vial and the Cerenkov radiation from yttrium -90 is counted in a liquid scintillation counter.

## 2. Apparatus

Chemical separation and counting
Liquid scintillation counter
Liquid scintillation vials in polyethylene with a capacity of at least 20 ml
Electronic balance, resolution 0.01g
Drying oven
Muffle furnace
Hot plate
Infrared lamp
pH-meter
Centrifuge, 3000 rpm
Centrifuge tube, 100ml, with stopper
Burette
Magnetic stirrer + magnet
Porcelain evaporating bowl, 200 ml and 500 ml
Watch glass
Separatory funnel, 250 ml
Stand + ring support with clamp
Beaker, 200 ml , 500 ml, 1000ml and 2000ml
Pipettes different size
Pasteur pipette
Buchner funnel
Filtring flask with hose connection 500ml
Rubber sleeves conical
Rubber tubing for vakuum
Filter paper, grade OOH
Filter pump
Erlenmyer flask, 100 ml
Measuring cylinder, 25 ml,50ml, 100ml and 1000ml
pH-paper range 1-14

Sample preparation
Drying cabinet
Food mixer
Sieve 2 mm and bottom

## 3. Reagents

All reagents are to be analytical grade
Certified Sr-90 standard solution
Distilled or de-ionized water
Hydrochloric acid, HCl : 1M
Hydrochloric acid, HCl : 0.08M
Nitric acid, $HNO_3$ : 3M
Nitric acid, $HNO_3$ : conc. ( 14.4 M)
Ammonia solution, 25% : $NH_3$
Ammonium hydroxide, $NH_4OH$ : 6M
Sodium hydroxide, NaOH : 6M
Citric acid , $C_6H_8O_7 \times H_2O$
Phenolphtalein 1% in ethanol
Toluene,$C_7H_8$
HDEHP : 10% in Toluene v/v =(volume/volume)
HDEHP= di(2-etyl-hexyl)phosphoric acid $(CH_3(CH_2)_3CH(CH_2\,CH_3)CH_2O)_2PO_2H$
Titriplex III solution for metal titration : 0.1M
Sodiumacetate-Trihydrate, $CH_3COONa \times 3H_2O$
Xylenolorange in $KNO_3$ , 1g xylenolorange is mixed with 99g potassiumnitrate
Potassiumnitrate , $KNO_3$
Xylenolorange
Buffert solutions pH1, pH4 and pH7
Yttrium carrier: 10mg $Y^{3+}$ per ml. Heat (avoid boiling), 12.7g of $Y_2O_3$ in a minimum volume of 14.4M $HNO_3$ until the oxide is dissolved. Dilute to 1000 ml with distilled water.

## 4. Procedure

### 4.1 Sample

Food (except milk and water) and environmental samples

1a. Weigh a portion of 0.5-20 g of dried, grind and thoroughly homogenized sample Transfer the weighted sample to a 200 ml porcelain evaporating bowl. Place the sample in a cold muffle furnace and slowly raise the temperature to 310 °C. Ash the sample at 310 °C for 3 hours, and finish the ashing at 610 °C for 15 hours.

1b Add 50 ml 1M HCl, cover the bowl with a watch glass, and boil the sample for a few minutes until all the ash is dissolved.

1c Filter with suction (Buchner funnel) through filter paper OOH and wash the filter with 2x10 ml 1M HCl. Transfer the solution to a 200 ml beaker.

Continue from step 2.

Water

1a. For a water sample, take a quantity of 0.5-2 liters depending upon the activity concentration in the sample. Evaporate on a hot plate to a volume of about 100 ml. Transfer the solution to a porcelain evaporating bowl, and evaporate to dryness. Place the sample in a cold muffle furnace and slowly raise the temperature to 310 °C. Ash the sample at 310 °C for 3 hours, and finish the ashing at 610 °C for 15 hours.

1b Add 50 ml 1M HCl, cover the bowl with a watch glass, and boil the sample for a few minutes until all the ash is dissolved.

1c Filter with suction (Buchner funnel ) through filter paper OOH and wash the filter with 2x10 ml 1M HCl. Transfer the solution to a 200 ml beaker.

Continue from step 2.

Milk

1a. Transfer 1000ml fresh milk in a two litre beaker and add 4ml conc acetic acid $CH_3COOH$ and mix thoroughly (acidic milk is easier to homogenize). Transfer 500 ml of the acidic milk to two 500 ml porcelain evaporation bowls (250 ml in each). Put the evaporation bowls on a hot plate. Place an infrared lamp at a stand over the evaporation bowls. Use low temperatture, avoid boiling, evaporate the milk to dryness. Place the bowls in a cold muffle furnace and slowly raise the temperature to 310 °C. Ash the sample at 310 °C for 3 hours, and finish the ashing at 610 °C for 15 hours.

1b. Add 25 ml 1M HCL to each bowl. Cover the bowls with a watch glass, and boil the sample for a few minutes until all the ash is dissolved.

1c. Combine the two solutions (point 1b) and filter with suction (Buchner funnel) through filter paper OOH and wash the filter with 2 x10 ml 1M HCl. Transfer the solution to a 200 ml beaker.

Continue from step 2.

All samples, step 2 and onward

2. Add 1ml yttrium carrier and 2g citric acid and adjust pH to 1.0 -1.2 with 6M $NH_4OH$

Note: step 3 to 14 must be accomplished at the same day.

3. Transfer the solution to a separatory funnel and extract the yttrium into 50 ml 10% HDEHP by shaking for 1 minute.

4. Record the time and date for the yttrium separation, t=t1

5. Allow the phases to separate. Drain the aqueous phase and discard it. Phase separation should be as clean as possible and it is better to leave a few drops of the aqueous phase in the organic rather than vice versa.

6. Wash the organic HDEHP phase by shaking for 1 minute with 50 ml 0.08M HCl. Allow the phases to separate. Drain the aqueous phase and discard it.

7. Extract the yttrium from the organic phase into 50 ml 3M $HNO_3$ by shaking for 1 minute. Allow the phases to separate.

8. Drain the aqueous phase into a 100 ml centrifuge tube. Discard the organic phase.

9. Add a few drops of phenolphtalein (1% in ethanol) to the solution. Precipitate the yttrium-hydroxide by adjusting pH to 10 with conc. ammonia $NH_3$. Colour changes to red at pH 9 -10

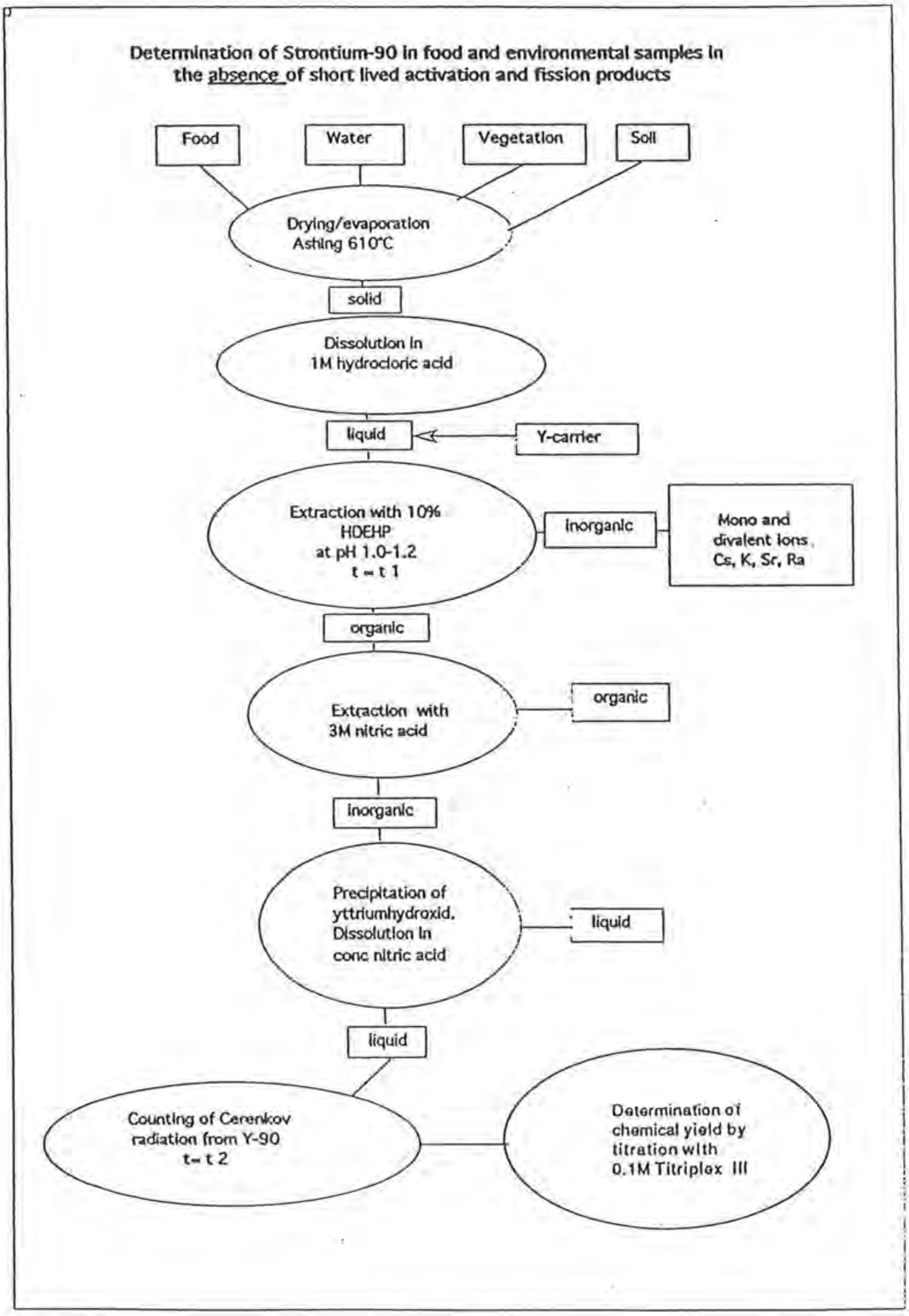
10. Let the solution cool and centrifuge at 3000 rpm for 5 minutes.

11. Discard the supernate and dissolve the precipitation in 1 ml conc. $HNO_3$

12. Add 14 ml distilled water to the centrifuge tube and mix thoroughly.

13. Transfer the solution to a 20 ml plastic scintillation vial.

14. The sample is now ready for measurement of Cerenkov radiation. t = t 2



**Determination of Strontium-90 in food and environmental samples in the <u>absence</u> of short lived activation and fission products**

## 4.2 Determination of efficiency of the LSC

Prepare a set of calibration solutions in liquid scintillation vials (15 ml 0.1M HCl ) to cover the anticipated activity range of the samples to be analyzed, by accurate dilution of a Sr-90 standard solution , (Sr-90 and Y-90 in equilibrium e.i. $A_{Sr\text{-}90} = A_{Y\text{-}90}$).

Prepare blank vials containing 15 ml 0.1M HCl for determination of the backround radiation for the counter .

Place the counting vials in the liquid scintillation counter and count each vial for a preset period of time.

For a single calibration solution the efficiency can be calculated using the expression :

$$E \quad = \quad \frac{R^{*} - Ro}{A_{Sr\text{-}90}} \quad F(I)$$

where

E is the counting efficiency for the system, in counts per second per becquerel of activity.

$R^{*}$ is the gross count rate of the Sr-90 calibration solution, in counts per second:

R o is the count rate of the blank counting vials, in counts per second:

$A_{Sr\text{-}90}$ is the activity of Sr-90 in the calibration solution, in becquerel.

A set of calibration solutions containing different amounts of Sr-90 are measured in order to check the constancy of E.

## 4.3 Determination of chemical yield for yttrium

Procedure

1. Transfer the solution from the scintillation vial, after measurement of Cerenkov radiation, into a 100 ml Erlenmyer flask and dilute the solution to 50 ml with dest. $H_2O$.

2. Add 1.5 g sodiumacetate.

3. Add 50-100 mg xylenolorange in $KNO_3$ and adjust the pH to 5-6 with 6M NaOH (1-2 ml is required use pH paper). The colour of the solution at pH 5-6 is violette . If the pH should become to high a precipitate appears and the pH has to be adjusted by adding a few drops of 3M $HNO_3$, until the precipitate disappears. If the pH becomes to low, add a few drops of 6M NaOH.

4. Titrate the sample with 0.1M Titriplex III to a yellow colour . The colour changes from violette to yellow or red-yellow . Record the consumption (ml) of Titriplex III (= $T_{sample}$ )

5. Quality of yttrium carrier

The quality of the yttrium carrier should be checked frequently by Titreplex III titration, at least two samples for each batch of strontium analyses. The procedure is as follows. Take the same volume yttrium carrier that was added to the sample (1-2 ml). Dilute the sample with 50 ml dest. $H_2O$, add 1.5 g sodiumacetate and 50 -100 mg xylenolorange in $KNO_3$. Titrate the standard with 0.1M Titriplex III to a yellow colour . The colour changes from violette to yellow or red-yellow . Record the consumption (ml) of Titriplex III (=$T_{standard}$ )

Determination of Chemical yield ( f )

$$f = \frac{T_{sample}}{T_{standard}}$$

## 5. Calculations

### 5.1 Determination of strontium-90 in the sample

To determine the activity of Sr-90 in a sample, use the following equation

$$C_{Sr} = \frac{(R - Ro)\exp(\lambda \Delta t)}{E x f} \qquad F(II)$$

where

$C_{Sr}$ is the activity of strontium-90 (=Y-90) , in becquerel (counts per second)
R is the gross counting rate for the sample, in counts per second
R o is the counting rate for the blank counting vials in counts per second
$\lambda$ is the decay constant for Y-90,in hours$^{-1}$,i.e. 0.0108 $h^{-1}$
$\Delta t$ is the time interval , in hours, between the separation of Y-90 and the counting : $\Delta t = t_2 - t_1$
E is as previously defined in ( F I )
f is the chemical yield

### 5.2 Standard deviation in activity

Calculate the standard deviation in the activity of the sample due to the statistical nature of radioactive decay and background radiation from the following equation :

$$Sc = \frac{\sqrt{\frac{R}{t} + \frac{Ro}{to}}}{E \times \exp(-\lambda \Delta t)} \qquad F(III)$$

where

| | |
|---|---|
| S c | is the standard deviation of Cs in becquerel per sample |
| E | is as defined in F ( I ) |
| R, Ro ,$\lambda$and $\Delta t$ | are defined in F ( II ) |
| t | is the counting time for the sample, in seconds |
| $t_o$ | is the counting time for the blank counting vial, in seconds |

## 5.3 Lower limit of detection:

The estimate of the lower limit of detection, Cmin becquerel per sample, can be calculated according to the following equation:

$$\text{Cmin} = \frac{2k\sqrt{\dfrac{\text{Ro} \times (t + t_o)}{t \times t_o}}}{E \times f} \qquad \text{F (V)}$$

where

| | |
|---|---|
| k | is the confidence coefficient for type 1 errors. It is recommended to set k = 1.64, which is equivalent to 95% confidence level for detection of activity. |
| t | is the counting time for the sample, in seconds; |
| $t_o$ | is the counting time for blank vials, in seconds; |
| Ro | is defined in F(II) |
| E | is defined in F(I) |
| f | is the chemical yield |

With $k = 1.64$ and $t = t_o$, this equation reduces to:

$$\text{Cmin} = \frac{4.65\sqrt{\text{Ro}/t}}{E \times f} \qquad \text{F (VI)}$$

As an example: With Ro = 0.012 c.s-1;
t = 10 000 seconds;
E = 0.6
f = 0.95

then

$$\text{Cmin} = \frac{4.65\sqrt{0.012 / 10000}}{0.6 \times 0.95}$$

$$= 10 \text{ mBq per sample}$$

## 6. Interference by other radionuclides

Radionuclides present in milk, water and environmental samples, not contaminated with fresh fallout, are mainly decay products from the naturally occurring uranium and thorium isotopes, potassium-40 and the longlived fission products cesium and strontium-90. Of these nuclides, which emit beta particle energies high enough to produce Cerenkov radiation, only yttrium-90 (daughter of strontium-90) will form complexes with HDEHP. This means that in the absence of short lived activation and fission products, the procedure will separate all interfering nuclides prior to counting.

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(1) Butler, F.E. Strontium-90 monitoring at the Savannah River plant. Health Physics 8(1962), pp. 833-836

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# DETERMINATION OF STRONTIUM -90 IN FOOD AND ENVIRONMENTAL SAMPLES IN THE PRESENCE OF SHORT LIVED ACTIVATION AND FISSION PRODUCTS

## 1. Summary

The procedure for determination of strontium-90 (=yttrium-90 ) by HDEHP-extraction of yttrium-90 and counting the Cerenkov radiation in a liquid scintillation counter, is applicable for all kind of samples. Prior to analyses liquid samples are evaporated to dryness and ashed. Environmental and food samples are oven dried and ashed.

The ash is dissolved in 1 molar hydrochloric acid and at pH 1.0-1.2 extracted with 20% HDEHP in order to remove interfering lanthanides and actinides.

The sample is stored for two weeks, for the ingrowth of yttrium-90. Yttrium is extracted from the stored sample into 5% HDEHP. Yttrium-90 is backextracted into 3 molar nitric acid and precipitated as hydroxide. The hydroxide precipitation is dissolved in 1ml conc nitric acid, transferred to a liquid scintillation vial and the Cerenkov radiation from yttrium -90 is counted in a liquid scintillation counter.

## 2. Apparatus

Chemical separation and counting
Liquid scintillation counter
Liquid scintillation vials in polyethylene with a capacity of at least 20 ml
Electronic balance, resolution 0.01g
Drying oven
Muffle furnace
Hot plate
Infrared lamp
pH-meter
Centrifuge, 3000 rpm
Centrifuge tube, 100ml, with stopper
Burette
Magnetic stirrer + magnet
Porcelain evaporating bowl, 200 ml and 500 ml
Watch glass
Separatory funnel, 250 ml
Stand + ring support with clamp
Beaker, 200 ml , 500 ml, 1000ml and 2000ml
Pipettes different size
Pasteur pipette
Buchner funnel
Filtring flask with hose connection 500ml
Rubber sleeves conical
Rubber tubing for vakuum
Filter paper, grade OOH
Filter pump
Erlenmyer flask, 100 ml
pH-paper range 1-14
Measuring cylinder, 25 ml,50ml, 100ml and 1000ml

Sample preparation
Drying cabinet
Food mixer
Sieve 2 mm and bottom

## 3. Reagents

All reagents are to be analytical grade
Certified Sr-90 standard solution
Distilled or de-ionized water
Hydrochloric acid, HCl : 1M
Hydrochloric acid, HCl : 0.08M
Nitric acid, $HNO_3$ : 3M
Nitric acid, $HNO_3$ : conc. (14.4 M)
Ammonia solution, 25% : $NH_3$
Ammonium hydroxide, $NH_4$ OH : 6M
Sodium hydroxide, NaOH
Citric acid , $C_6 H_8 O_7$ x $H_2O$
Phenolphtalein 1% in ethanol
Toluene , $C_7 H_8$
HDEHP : 20% in Toluene v/v=(volume/volume)
HDEHP : 5% in Toluene v/v =(volume/volume)
HDEHP di(2-ethyl-hexyl)phosphoric acid $(CH_3(CH_2)_3\ CH(CH_3\ CH_3)CH_2\ O)_2PO_2\ H$
Titriplex III solution for metal titration : 0.1M
Sodiumacetate-Trihydrate, $CH_3COO$ Na x $3H_2$ O
Xylenolorange in $KNO_3$ , 1g xylenolorange is mixed with 99g potassiumnitrate
Potassiumnitrate , $KNO_3$
Xylenolorange
Buffert solutions pH1, pH4, and pH7
Yttrium carrier: 10mg $Y^{3+}$ per ml. Heat (avoid boiling) 12.7g of $Y_2\ O_3$ in a minimum volume of 14.4M $HNO_3$ until the oxide is dissolved. Dilute to 1000 ml with distilled water.

## 4. Procedure

### 4.1 Sample

Food (except milk and water) and environmental samples

1a. Weigh a portion of 0.5-20 g of dried, grind and thoroughly homogenized sample.Transfer the weighted sample to a 200 ml porcelain evaporation bowl. Place the sample in a cold muffle furnace and slowly raise the temperature to 310 °C. Ash the sample at 310 °C for 3 hours, and finish the ashing at 610 °C for 15 hours.

1b Add 50 ml 1M HCl, cover the bowl with a watch glass, and boil the sample for a few minutes until all the ash is dissolved.

1c Filter with suction (Buchner funnel) through filter paper OOH and wash the filter with 2x10 ml 1M HCl .Transfer the solution to a 200 ml beaker.

Continue from step 2.

Water

1a. For a water sample, take a quantity of 0.5-2 liters depending upon the activity concentration in the sample. Evaporate the sample on a hot plate to a volume of about 100 ml. Transfer the solution to a porcelain evaporating bowl, and evaporate to dryness. Place the sample in a cold muffle furnace and slowly raise the temperature to 310 °C. Ash the sample at 310 °C for 3 hours, and finish the ashing at 610 °C for 15 hours.

1b. Add 50 ml 1M HCl, cover the bowl with a watch glass, and boil the sample for a few minutes until all the ash is dissolved.

1c. Filter with suction (Buchner funnel ) through filter paper OOH and wash the filter with 2x10 ml 1M HCl. Transfer the solution to a 200 ml beaker.

Continue from step 2.

Milk

1a. Transfer 1000 ml fresh milk into a 2000 ml beaker and add 4ml conc acetic acid $CH_3COOH$ and mix thoroughly (acidic milk is easier to homogenize). Transfer 500 ml of the acidic milk to two 500 ml porcelain evaporation bowls (250 ml in each). Put the evaporation bowls on a hot plate. Place an infrared lamp at a stand over the evaporation bowls. Use low temperature, avoid boiling, evaporate the milk to dryness. Place the bowls in a cold muffle furnace and slowly raise the temperature to 310 °C. Ash the sample at 310 °C for 3 hours, and finish the ashing at 610 °C for 15 hours.

1b. Add 25 ml 1M HCl to each bowl. Cover the bowls with a watch glass and boil the sample for a few minutes until all the ash is dissolved.

1c. Combine the two solutions (point 1b) and filter with suction (Buchner funnel) through filter paper OOH and wash the filter with 2 x10 ml 1M HCl. Transfer the solution to a 200 ml beaker.

Continue from step 2.

All samples step 2 and onward

2. Add 2gram citric acid and adjust pH to 1.0 -1.2 with 6M $NH_4$ OH

3. Transfer the solution to a separatory funnel and extract the sample by shaking for 1 minute with 50 ml 20% HDEHP.

4. Record the time and date as the beginning of the yttrium -90 ingrowth.

5. Allow the phases to separate. Drain the aqueous phase into a suitable glass bottle, and add 1 ml yttrium carrier. Discard the organic phase. The separation of phases should be as clean as possible but it is better to leave a few drops of water in the organic phase rather than vice versa.

6. Store the aqueous solution for 2 weeks for ingrowth of yttrium-90.

Note: steps 7 to 18 must be accomplished at the same day.

7. Transfer the sample from step 6 to a separatory funnel.

8. Extract yttrium -90 from the solution by adding 50 ml of 5% HDEHP and shaking for 1 minute. Allow the phases to separate. Discard the aqueous phase.

9. Record the time and date of the yttrium -90 separation. t=t1

10. Wash the organic HDEHP phase by shaking for 1 minute with 50 ml 0.08M HCl . Allow the phases to separate. Discard the aqueous phase .

11 .Extract the yttrium from the organic phase into 50 ml 3M $HNO_3$ by shaking for 1 minute. Allow the phases to separate.

12. Drain the aqueous phase into a 100 ml centrifuge tube. Discard the organic phase.

13. Add a few drops of phenolphtalein (1% in alcohol) to the solution. Precipitate the yttrium-hydroxide by adjusting pH to 10 with conc. ammonia $NH_3$. Colour will change to red at pH 9-10.

14. Let the solution cool and centrifuge at 3000 rpm for 5 minutes.

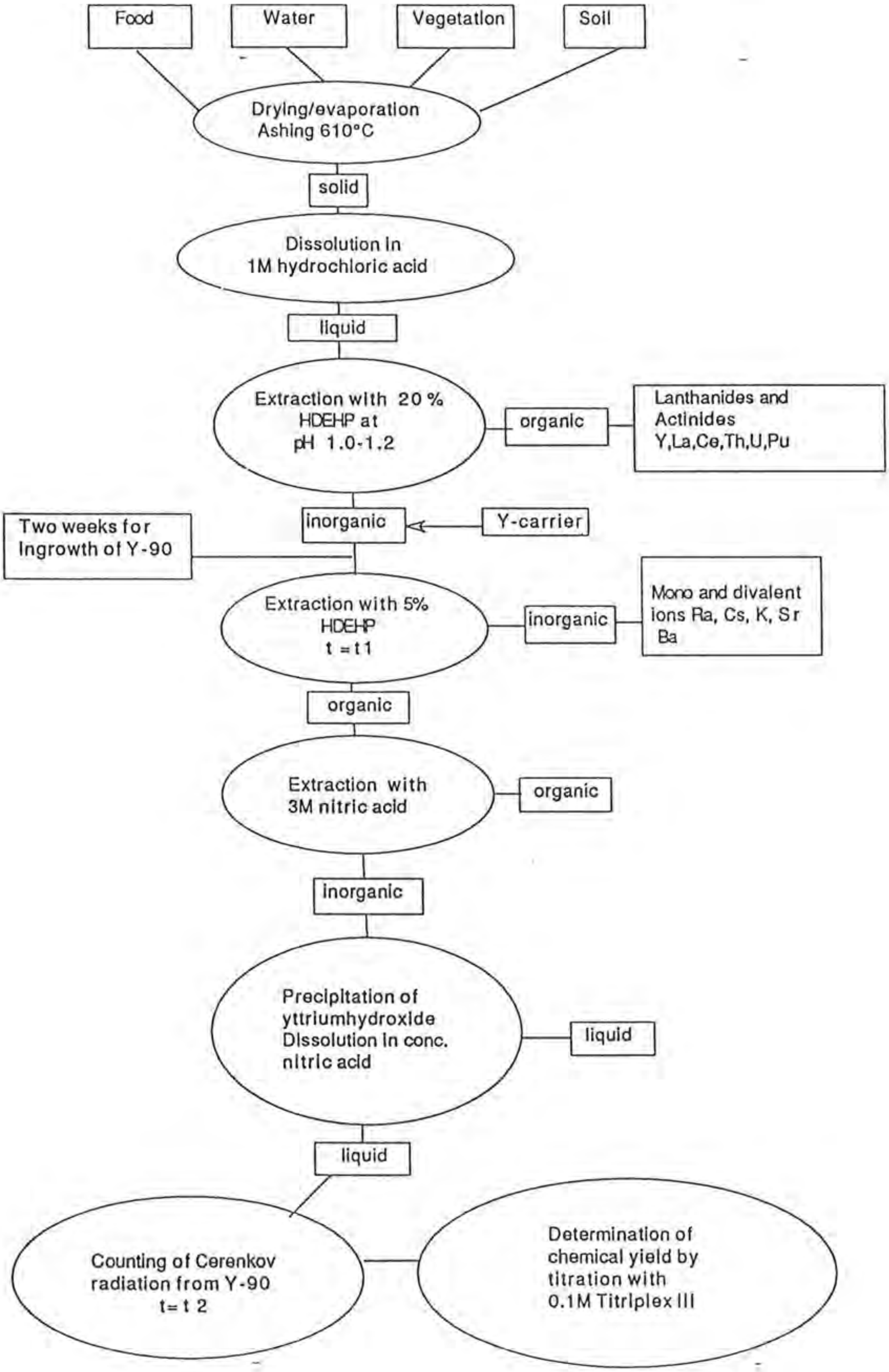
15. Discard the supernate and dissolve the precipitation in 1 ml conc. $HNO_3$

16. Add 14 ml distilled water to the centrifuge tube and mix thoroughly.

17. Transfer the solution to a 20 ml plastic scintillation vial.

18. The sample is now ready for measurement of Cerenkov radiation. t= $t_2$

**Determination of Sr-90 in food and environmental samples in presence of short lived activation and fission products**



## 4.2 Determination of efficiency of the LSC

Prepare a set of calibration solutions in liquid scintillation vials (15 ml 0.1M HCl ) to cover the anticipated activity range of the samples to be analyzed, by accurate dilution of a Sr-90 standard solution , (Sr-90 and Y-90 in equilibrium e.i. $A_{Sr-90} = A_{Y-90}$)
Prepare blank vials containing 15 ml 0.1M HCl for counting the Cerenkov radiation from Y-90.

Place the counting vials in the liquid scintillation counter and count each vial for a preset period of time.

For a single calibration solution the efficiency can be calculated using the expression :

$$E = \frac{R^* - Ro}{A_{sr-90}} \qquad F(I)$$

where

E is the counting efficiency for the system, in counts per second per becquerel of activity.

$R^*$ is the gross count rate of the Sr-90 calibration solution, in counts per second:

R o is the count rate of the blank counting vials, in counts per second:

$A_{sr-90}$ is the activity of Sr-90 in the calibration solution, in becquerel.

A set of calibration solutions containing different amounts of Sr-90 are measured in order to check the constancy of E.

## 4.3 Determination of chemical yield for yttrium

Procedure

1. Transfer the solution from the scintillation vial, after measurement of Cherenkov radiation, into a 100 ml Erlenmyer flask and dilute the solution to 50 ml with dest. $H_2O$.

2. Add 1.5 g sodiumacetate.

3. Add 50-100 mg xylenolorange in $KNO_3$ and adjust the pH to 5-6 with 6M NaOH (1-2 ml is required use pH paper). The colour of the solution at pH 5-6 is violette . If the pH should become to high a precipitate appears and the pH has to be adjusted by adding a few drops of 3M $HNO_3$, until the precipitate disappears. If the pH becomes to low, add a few drops of 6M NaOH.

4. Titrate the sample with 0.1M Titriplex III to a yellow colour . The colour changes from violette to yellow or red-yellow . Record the consumption (ml) of Titriplex III (= T sample )

5. Quality of yttrium carrier

The quality of the yttrium carrier should be checked frequently by Titreplex III titration, at least two samples for each batch of strontium analyses. The procedure is as follows. Take the same volume yttrium carrier that was added to the sample (1-2 ml). Dilute the sample with 50 ml dest. $H_2O$, add 1.5 g sodiumacetate and 50 -100 mg xylenolorange in $KNO_3$. Titrate the standard with 0.1M Titriplex III to a yellow colour . The colour changes from violette to yellow or red-yellow . Record the consumption (ml) of Titriplex III (=$T_{standard}$ )

Determination of Chemical yield ( f )

$$f = \frac{T_{sample}}{T_{standard}}$$

## 5. Calculations

### 5.1 Determination of strontium-90 in the sample

To determine the activity of Sr-90 in a sample, use the following equation

$$C_{Sr} = \frac{(R - R_o) \exp(\lambda \Delta t)}{E \times f} \qquad F(II)$$

where

$C_{Sr}$ is the activity of strontium-90 (=Y-90) , in becquerel (counts per second)
R is the gross counting rate for the sample, in counts per second
R o is the counting rate for the blank counting vials in counts per second
$\lambda$ is the decay constant for Y-90,in hours$^{-1}$,i.e. 0.0108 $h^{-1}$
$\Delta t$ is the time interval , in hours, between the separation of Y-90 and the counting : $\Delta t = t_2 - t_1$
E is as previously defined in ( F I )
f is the chemical yield

### 5.2 Standard deviation in activity

Calculate the standard deviation in the activity of the sample due to the statistical nature of radioactive decay and background radiation from the following equation :

$$S_c = \frac{\sqrt{\frac{R}{t} + \frac{R_o}{t_o}}}{E \times \exp(-\lambda \Delta t)} \qquad F(III)$$

where

| | |
|---|---|
| S c | is the standard deviation of Cs in becquerel per sample |
| E | is as defined in F ( I ) |
| R, Ro , $\lambda$ and $\Delta t$ | are defined in F ( II ) |
| t | is the counting time for the sample, in seconds |
| $t_o$ | is the counting time for the blank counting vial, in seconds |

## 5.3 Lower limit of detection:

The estimate of the lower limit of detection, Cmin becquerel per sample, can be calculated accoording to the following equation:

$$\mathrm{Cmin} = \frac{2k\sqrt{\dfrac{\mathrm{Ro} \times (t + to)}{t \times to}}}{E \times f} \qquad \mathrm{F\,(V)}$$

where

| | |
|---|---|
| k | is the confidence coefficient for type 1 errors. It is recommended to set k = 1.64, which is equivalent to 95% confidence level for detection of activity. |
| t | is the counting time for the sample, in seconds; |
| $t_o$ | is the counting time for blank vials, in seconds; |
| Ro | is defined in F(II) |
| E | is defined in F(I) |
| f | is the chemical yield |

With k = 1.64 and $t = t_o$, this equation reduces to:

$$\mathrm{Cmin} = \frac{4.65\sqrt{\mathrm{Ro} / t}}{E \times f} \qquad \mathrm{F\,(VI)}$$

As an exampel: With Ro = 0.012 c.s-1;
t = 10 000 seconds;
E = 0.6
f = 0.95

then

$$\mathrm{Cmin} = \frac{4.65\sqrt{0.012 / 10000}}{0.6 \times 0.95} = 10 \text{ mBq per sample}$$

## 6. Interference by other radionuclides

The chemical separation described above will effectively remove radioactive elements other than yttrium-90. The first extraction with 20% HDEHP will remove all the lanthanides and actinides present in the sample.

Di valent elements like barium and strontium will remain in the aqueous phase. The radionuclides strontium-90 and barium-140 will, if they are present in the sample, produce the beta-emitting nuclides yttrium-90 and lanthanum-140 respectively during two weeks storage of the sample.

The second extraction with 5% HDEHP will, however separate yttrium from lanthanum together with monovalent ions. This means that the contribution fom interfering nuclides will be neglectable in the determination of strontium-90 by Cerencov counting..

# Utgivna SSI-rapporter 1993

## SSI-reports published in 1993

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| 09 | Isotopkommittérapporter 1991 | Mauricio Alvarez |
| 10 | Radon 1993<br>En rapport över läget | Statens strålskyddsinstitut |
| 11 | Methods for determination of strontium-90 in food and environmental samples by Cerenkov counting | Lena Wallberg<br>J. Suomela, J. Melin |