



Review

Determination of radiostrontium isotopes: A review of analytical methodology

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ARTICLE INFO

Article history:

Received 18 January 2010

Received in revised form

6 May 2010

Accepted 14 May 2010

Keywords:

Sr-90

Y-90

Sr-89

β counting

β spectrometry

Mass spectrometry

ABSTRACT

A great number of analytical methods have been developed and applied for the determination of ⁹⁰Sr and ⁸⁹Sr in environmental and nuclear samples using various measuring techniques, i.e. beta counting, liquid scintillation spectrometry and mass spectrometry. This paper is intended to give an overview about the development of the radiochemical procedures for the separation of strontium and/or yttrium including the classical procedure based on a series of semi-selective precipitations, the ion exchange and solvent extraction procedures and the recently developed extraction chromatographic ones offering high selectivity. An outlook to the procedures under development aiming the technological separation of strontium from radioactive wastes and process solutions is also given. Nuclear measuring techniques and mass spectrometric techniques will be comparatively evaluated.

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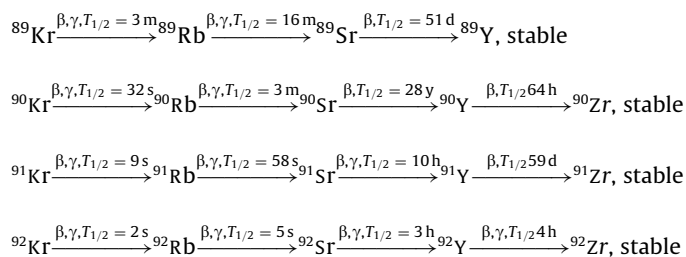
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1. Introduction

Radiostromium isotopes are present in the environment on a global scale from atmospheric nuclear weapons testing and on a local scale from authorized or unauthorized releases from nuclear facilities and nuclear accidents. Analytical results are used to determine the contamination levels in the environment, biota, foodstuffs including milk, human organisms to calculate transfer factors, follow the migration and uptake processes and estimate the dose impact to man. ^{90}Sr is one of the most hazardous radionuclides due to its long physical and biological half-life, accumulation in bone tissue and the high-energy beta particles from its daughter nuclide ^{90}Y causing damage to bone marrow. Detailed radioecological investigations carried out after the Chernobyl accident revealed an unexpectedly high mobility of radiostromium in the environment compared to many other radionuclides, e.g. to the Cs isotopes that are usually well retained by clay minerals (IAEA, 1992). The present levels of contamination are typically low, thus sensitive analytical techniques are needed to obtain accurate and reliable data.

The term ‘radiostromium’ is used for two strontium isotopes, i.e. ^{90}Sr that is a long-lived nuclide of a half-life of 28.80 years that decays by emission of beta particles (maximum beta energy of 545.96 keV) to radioactive ^{90}Y , and ^{89}Sr that has a half-life of 50.57 days and decays by emission of beta particles (maximum beta energy of 1495.1 keV) to stable ^{89}Y (NUCLÉIDE, 2004). There are other radioactive strontium isotopes of short half-lives, e.g. ^{91}Sr , ^{92}Sr , that emit both beta particles and gamma radiation. Radiostromium isotopes are produced at relatively high yields in nuclear fission, e.g. the cumulative fission yield of ^{90}Sr in the thermal neutron reaction of ^{235}U is 5.88% and ^{239}Pu is 2.10%. The major production and decay processes are summarized in the following equations:



In the decay series of the mass numbers 89–92 there are 4 radionuclides of relatively long half-life, i.e. ^{89}Sr , ^{90}Sr , ^{90}Y and ^{91}Y , all of them are pure beta emitters. The end-point energies of ^{90}Y and ^{91}Y are high, 2279.8 and 1544.8 keV, respectively.

Although the longest-lived radiostromium isotope, ^{90}Sr is of major concern in environmental contamination and nuclear processes, all strontium and yttrium isotopes are released together to the environment in nuclear explosions or reactor accidents. Activities of the shorter-lived Sr and Y nuclides can be 1–2 orders of magnitude higher than that of ^{90}Sr . This is the reason why analysts are faced with the demanding task of accurate and rapid determination of ^{90}Sr during emergency situations. Couple of years after a nuclear incident ^{89}Sr , ^{90}Sr , ^{90}Y and ^{91}Y will be present together in the environment, and only after several year-long cooling time will ^{89}Sr and ^{91}Y decay to levels below the detection limits, and “undisturbed” samples will contain only ^{90}Sr in secular equilibrium with its daughter ^{90}Y . In the following we will use the term radiostromium for ^{89}Sr and ^{90}Sr not taking into account the short-lived Sr isotopes that will decay after a couple of days following a release scenario.

The purpose of this paper is to review the radiochemical procedures that have been developed and used for the

determination of radiostromium in various matrices focusing on the up-to-date methods. An outlook to future developments in the separation chemistry of Sr and Y will be given. Nuclear measuring techniques, e.g. beta counting, liquid scintillation spectrometry and calculation methods will be briefly discussed. Capabilities of these techniques will be compared with those of various mass spectrometric (MS) techniques. The literature survey is based on the publications of the last 20 years referred in the International Nuclear Information System (INIS) database.

2. Measurement methods

The determination of ^{89}Sr and ^{90}Sr can be performed by beta counting techniques using gas ionization detectors (proportional counters, GM counters), solid or liquid scintillators (LS), Cerenkov counters, surface barrier detectors. Owing to the continuous nature of the energy distribution of beta radiation beta emitting radionuclides have significant spectrum interferences that cannot be resolved by beta counters and the chances for instrumental resolution in beta spectrometers are also limited. Thus, for the determination of radiostromium isotopes from complex matrices Sr or Y has to be separated from other beta emitting nuclides, most likely from all sample components. Radiochemical procedures aiming at the separation of strontium provide beta sources in which the sum of the activities of ^{89}Sr and ^{90}Sr can be detected by beta counting techniques. Counting of the source with a delay relative to the time of Sr–Y separation (e.g. repeated or long counting) will result in the in-growth of ^{90}Y daughter according to the following equation that can be taken into account while calculating the activities:

$$A_{\text{Y-90}} = A_{\text{Sr-90}}(1 - e^{-\lambda_{\text{Y-90}}t})$$

where $A_{\text{Y-90}}$ is the activity of ^{90}Y , $A_{\text{Sr-90}}$ is the activity of ^{90}Sr , $\lambda_{\text{Y-90}}$ is the decay constant of ^{90}Y , t is the time between Sr and Y separation and counting.

If the secular equilibrium between ^{90}Sr and ^{90}Y exists the activity of ^{90}Sr can be determined by measuring ^{90}Y after a properly chosen radiochemical procedure for the separation of Y. In the latter case measurements will not provide information about the activity of ^{89}Sr , and the method can only be used if it has been proven that the sample was free from ^{91}Y (not in emergency situation).

The beta radiation of ^{89}Sr , ^{90}Sr and ^{90}Y can be detected by beta counters. Gas proportional counters have acceptably high efficiency (around 50%) and relatively low background offering a sensitive analytical tool. Detection limits around 10 mBq/sample can be achieved (see in details in Table 4). Geiger–Müller counters can also be used to detect the relatively high-energy beta radiation. Nevertheless, these counting techniques are unable to discriminate the beta particles of ^{89}Sr , ^{90}Sr and ^{90}Y . Repeated counting of the source and checking the ^{90}Y in-growth in certain time intervals as well as the repeated separation of Y after attaining the equilibrium followed by counting the ^{90}Y source can improve the reliability of the method.

Although the background of liquid scintillation spectrometers is higher than that of proportional counters, liquid scintillation is favored due to the much better spectral resolution that enables differentiation of ^{89}Sr , ^{90}Sr and ^{90}Y and the contribution of the three nuclides can be calculated theoretically from a single measurement using the 2- or 3-window method. Nonetheless repeated counting is recommended. The counting efficiencies in LSC are high, close to 100% for all three nuclides. Detection limits are comparable with those of proportional counters (see in details in Table 4). Solid scintillators have also been used for the

measurement of ^{89}Sr , ^{90}Sr and ^{90}Y . Detection limits of about 10 mBq/sample can be achieved (see in details in Table 4).

Silicon surface barrier detectors have also been reported to detect beta particle spectra. The resolution of the ^{89}Sr , ^{90}Sr and ^{90}Y spectrum is poor due to the continuous character of the beta particle energy distribution. To detect the high-energy beta particles thick, consequently expensive Si detectors are needed, the counting efficiency depends on the detector thickness. The background is a few counts per day, so the limit of detection is significantly lower than that of proportional counters or scintillation counters.

^{89}Sr and ^{90}Sr can be detected using Cerenkov detectors. The high-energy beta particles emitted by ^{89}Sr or the ^{90}Y daughter of ^{90}Sr produce Cerenkov light in a medium where the speed of the beta particle is higher than that of light. Owing to the high Cerenkov counting efficiency ($> 40\%$) for ^{89}Sr and ^{90}Y and the relatively low background a sensitive analytical technique is obtained for both isotopes, but they will significantly interfere. If Sr is separated by a radiochemical method from Y (and from other sample components) the Cerenkov light detected in the freshly separated source originates from ^{89}Sr . Cerenkov counting can be performed in commercial liquid scintillation counters without scintillation cocktail, typical detection limits are higher (> 100 mBq/sample) than in case of proportional counters or LS spectrometers (see in details in Table 4).

Recently, radiometric methods have been reported to be used for the flow scintillation analysis of ^{90}Sr (Roane et al., 2003; Plionis et al., 2009). In this technique, also called radiochromatography, an on-line scintillation counter is attached to an ion chromatograph or a high performance extraction chromatography system that separates Sr.

Radiometric methods have been limited by slow analysis time and low sample throughput. Mass spectrometry offers the advantage of short analysis time, but only high sensitivity mass spectrometric techniques are capable to detect the extremely low masses of the “short-lived” isotopes of radiostrontium. A few papers (Taylor et al., 2007; Feuerstein et al., 2008; Tumey et al., 2008; Lantzsich et al., 1995; Trautmann, 1995) have been published about the detection of ^{90}Sr , the longest-lived strontium isotope, the detection of ^{89}Sr has been even more problematic. Mass spectrometric methods are often sensitive to isobaric interferences, and/or they require special source preparation techniques, thus time-consuming chemical processing cannot be avoided.

Among the high sensitivity mass spectrometric methods, inductively coupled plasma mass spectrometry (ICP-MS), accelerator mass spectrometry (AMS) and resonance ionization mass spectrometry (RIMS) have been applied recently for the determination of ^{90}Sr with more or less success. The instrumental detection limits of high sensitivity ICP-MS, AMS and RIMS are in the range 10^6 – 10^8 atoms (about 1–100 mBq), 10^5 atoms (about 0.1 mBq) and 10^4 atoms (0.01 mBq) (Taylor et al., 2007; Feuerstein et al., 2008; Tumey et al., 2008; Lantzsich et al., 1995; Trautmann, 1995), respectively, but there are several limitations. Determination of ^{90}Sr in environmental matrices by ICP-MS requires sufficient abundance sensitivity to resolve the peak tail of the natural stable Sr, i.e. ^{88}Sr isotope, complete removal of the isobar interference of stable ^{90}Zr , and elimination of various molecular interferences. Applying radiochemical separation and dynamic reaction cell ICP-MS, detection limits of 0.02–0.2 Bq/g ^{90}Sr were reported (Taylor et al., 2007; Feuerstein et al., 2008) what is still far too high to detect environmental levels of ^{90}Sr . AMS is insensitive to molecular interferences, but ^{90}Zr causes a pervasive interference in the determination of ^{90}Sr . After instrumental developments and a special source preparation technique to produce negative ion sources, a detection limit of 75 mBq ^{90}Sr per sample has been reported (Tumey et al., 2008). In RIMS, Laser beams are used to

excite and ionize the analyte atoms selectively/resonantly, thus isobaric interferences due to other elements are eliminated. By far, the most sensitive analytical technique for the determination of ^{90}Sr and ^{89}Sr is collinear RIMS, a detection limit for ^{90}Sr of 10^7 atoms (about 8 mBq) in the presence of 10^{17} atoms of stable Sr has been reported and the applicability of the method for ^{90}Sr determination was demonstrated by the analysis of air filter samples (Lantzsich et al., 1995; Trautmann, 1995). This detection limit is comparable with those of radiometric methods. The major limitation of RIMS is the high cost of the sophisticated instrument.

3. The radiochemistry of Sr

A few monographs and review papers have been published about the physical, chemical and nuclear properties of Sr isotopes including the early review of Sunderman and Townley from (1960), followed by the works of Wilken (1988), Wilken and Joshi (1991), and Brun et al. (2003).

Strontium is an alkaline earth metal. Although fission product radiostrontium isotopes have very high specific activity, consequently low Sr content, samples of environmental origin, e.g. soil, bone, milk, often contain natural Sr and typically other alkaline earth metals in amounts exceeding that of radiostrontium by orders of magnitude. The major issue of radiochemical processing is the separation of Sr from alkaline earth matrix components, alkaline earth radionuclides, e.g. ^{133}Ba , ^{140}Ba , ^{226}Ra , ^{228}Ra and several other possible interferences, the most important being Pb, Y nuclides. In mass spectrometry the most important isobaric interference of ^{90}Sr is stable ^{90}Zr .

Strontium similarly to other alkaline earth elements forms divalent cations in aqueous solutions, and has many insoluble compounds and stable chelate complexes. Radiochemical separation procedures are often based on precipitation and complex formation. Below we are shortly summarizing the most important insoluble compounds and complexes of Sr compared to those of Ca and Ba. The common insoluble salts of Ca, Sr and Ba are listed in Table 1.

Nitrate salts precipitated from concentrated nitric acid are conventionally used for the separation of Sr from big amounts of Ca. Solubilities of Ca, Sr and Ba nitrates in nitric acid are given in Table 2.

The formation of complex ions in aqueous solutions with inorganic ions or organic compounds is an important property of Sr.

Some stable complexes of Sr with inorganic and simple organic complexants and their stability constants are given in Table 3.

Yttrium is a transition metal belonging to the 3rd subgroup of the periodic table. In general, environmental samples contain relatively small amounts of yttrium and elements chemically similar to Y, e.g. La, Sc, thus the separation of Y is usually a less

Table 1
Solubilities of common Ca, Sr, Ba salts according to CRC Handbook of Chemistry and Physics (1987–1988).

Anion	Solubility in water g/100 mL cold water		
	Ca	Sr	Ba
Fluoride	0.0016	0.011	0.12
Carbonate	0.0015	0.0011	0.002
Oxalate	0.00067	0.0051	0.0093
Sulfate	0.209	0.0113	0.00025
Chromate	16.3	0.12	0.00034
Phosphate (ortho)	0.032	i	0.01
Nitrate	121.2	70.9	8.7

Table 2

Solubilities of Ca, Sr and Ba nitrates in nitric acid according to Bojanowski (mg/L) at 25 °C.

Metal ion	67% HNO ₃	82% HNO ₃
Ca	25,200	1750
Sr	58	3
Ba	16.8	0.6
Pb	280	10

Table 3

Ca, Sr and Ba complexes of some complexing agents according to Sunderman and Townley.

Chelating agent	Stability constant lg K		
	Ca	Sr	Ba
EDTA	10.59	8.63	7.76
Oxalic acid	3.00	2.54	2.31
Citric acid	3.4	2.7	2.3
Lactic acid	1.07	0.70	0.55

demanding job than the separation of Sr from big amounts of Ca or Ba. There are separation procedures of high selectivity for Y that make Y processing simpler. Y as a trivalent cation forms strong complexes with chelates, e.g. HDEHP and is strongly retained by cation exchangers. As several rare earth elements, it can also form neutral ion association complexes in concentrated nitric acid solution with organo-phosphorous extractants. The stability of the Y nitrate tributyl phosphate complex is extremely high (see later).

4. Development of separation procedures for the determination of radiostrontium isotopes

4.1. Sample pre-treatment

The objective of sample pre-treatment is to obtain a homogeneous sample solution, that contains the analytes in dissolved form without significant losses, (i) free of insoluble residues that would interfere during the subsequent chemical processing by retaining the analyte, (ii) free of any organic material that could form complexes with the analyte or the reagents, and that assures the isotope exchange among the analyte and the stable Sr (both from sample and carrier). Several good monographs have been published about the various methods of sample attack (Bock, 1970; Sill and Sill, 1995; Chiu et al., 1984; Claisse, 1995). In the HASL-300 Procedures Manual of the Environmental Measurements Laboratory (1997) detailed ashing, complete solution and leaching methods are described to determine radiostrontium in various matrices.

The following standard procedures are available about sample decomposition: the ASTM C1342-96 standard deals with fusion decomposition of solid materials, the ASTM D1971-95 standard describes hotplate and convection-oven digestion of water and solid samples with nitric and hydrochloric acids, the ASTM D4309-91 standard is about microwave digestion with hydrochloric and nitric acids for determination of metals and groundwater and the U.S. EPA Method 3052 (U.S. EPA, 1991) discusses microwave-heated, closed-vessel decomposition of ashes, biological tissues, oils, oil-contaminated soils, sediments, sludges and soils.

For **soil, sediment and mineral samples**, pre-treatment means drying, homogenizing, sieving, ashing at moderately high temperature (500–600 °C) followed by wet chemical destruction. Higher temperatures can cause losses of Sr or turn leachable Sr compounds into refractory ones. For sample dissolution leaching with mineral acids favorably by nitric or hydrochloric acid, and complete destruction with a mixture of mineral acids including nitric, hydrochloric and hydrofluoric acids and hydrogen peroxide proved to be efficient. It has to be noted that leaching of Sr from soil and sediment samples is usually an adequate procedure to solubilize radiostrontium originating from fallout what is not always the case for actinides because radiostrontium can be produced by condensation during accidental releases, while actinides are more often embedded in refractory particles. According to the HASL-300 Procedures Manual (1997) the HCl leaching method yielded ⁹⁰Sr values which averaged 91% of those obtained by the complete dissolution method. Wet acid decomposition can be carried out in open or closed systems using microwave or electric ovens. Samples are fast and completely decomposed by fusion with various bases or salts, e.g. with sodium carbonate fusion in the HASL-300 Procedures Manual (1997). A NaOH–HCl leaching method has also been recommended. Comparative soil analyses showed that the tracer/carrier could be completely equilibrated with the radiostrontium in the soils using the latter method.

In case of **biological samples** complete destruction is carried out usually by the combination of dry and wet chemical ashing. Microwave oven is used to decompose small samples (Solatie et al., 2002). An efficient way of decomposition of organic materials is the Fenton's reaction using hydrogen peroxide and Fe²⁺ catalyst. Tavcar and Benedik (2005) applied this procedure to destroy spent organic resin. According to the HASL-300 Procedures Manual (1997) dry ashing is followed by sodium carbonate fusion to destroy organic residues of the resin.

Milk is of special interest for radiostrontium determination in emergency situations because Sr transfer from soil–plant to milk is efficient and milk ingestion is a substantial pathway of radiostrontium incorporation in human body. The classical pretreatment of milk samples comprises preservation, e.g. using formaldehyde, sodium azide, drying and incineration followed by dissolution of the ash with nitric or hydrochloric acid. This method has been recommended and used by many authors for long time (HASL-300, 1997; IAEA, 1989a, 1989b; Kimura et al., 1979; Wilken and Diehl, 1987; Lopes and Madruga, 2008). Although calcinations pretreatment is efficient, it takes generally long-time even for small sample sizes. Abbadi et al. (1997) introduced a fast incineration technique, liquid milk was ashed in an electrically heated, fluidized bed reactor at 600 °C. Two hours were required to ash 500 mL sample.

For emergency situation rapid procedures had to be developed. Recently an excellent review paper summarized rapid methods of radiostrontium analysis in milk where the methods were compared with each other in terms of analysis time (Brun et al., 2003). Unfortunately, leaching is not efficient because of the high protein and fat content of milk. Strontium like calcium is bound basically to the phosphate groups of casein, consequently is located about 80% in the casein micelles. By lowering the pH to 5.3, a major part of Sr is transferred to the milk soluble phase. This part of Sr can be analysed in a non-destructive way after direct concentration of Sr from acidified milk. Gjerci (1996) used “leaching” by repeated evaporation of the sample with concentrated nitric acid. Mikulaj and Svec (1993) precipitated and removed proteins and part of fat using also nitric acid. Strong acid cation exchange resins were used for pre-concentration of alkaline earth metals from milk samples by many authors, e.g. by Nervik et al. (1954) and by Migikowsky (1960) both reported

by Brun et al. (2003). In the procedure of Gillard-Baruh (1973) 150 mL of cation exchange resin is mixed with 500 mL milk, and 600 mL 4 M HNO₃ is used to strip the alkaline earths.

After the Chernobyl accident to meet the multiplied demands towards rapid analytical procedures new procedures were proposed by Oravec and Navarcik (1987), Vaney et al. (1989), Suomela and Wallberg (1989), Jeter and Grob (1994), and Baratta (2000) that incorporated the use of cation exchangers in batch or column techniques for preconcentration purposes. Although the procedure has unambiguous advantages regarding rapidity it has major drawbacks as the consumption of big amount of resin. Fat and protein can cause the clogging of the columns, prevent the reuse of the resin and reduce the Sr recovery.

Acidified **water** samples are often concentrated by evaporation and the residue is decomposed by ashing. According to the HASL-300 procedure (1997), ashes are further treated by sodium carbonate fusion to complete decomposition of organic residues. According to the recommended procedure of the IAEA (1989a, 1989b), acidified water samples up to 100 L are evaporated using a rotation evaporator. Chmielewska et al. (2008) pointed at the possibility of Sr losses during evaporation due to sorption of radiostrontium on the walls of the container. In total 100–1000 mg of Ca were added to 10 L of water to suppress adsorption. The Sr content of the water samples can be pre-concentrated by precipitation (see next paragraph), what is recommended for big volumes of water samples especially of high salt content, e.g. sea water, and by cation exchange. According to the ASTM Standard Test Method (2008) for the determination of ⁹⁰Sr in water, and the recommended Eichrom method (2003), 1 L water sample is passed through a cation resin column of 10 mL volume. Cation exchange resin was used by Taylor et al. (2007) for pre-concentration of alkaline earths from 1 L of water. The procedure is easier to perform than in case of milk samples, and small columns can be advantageously applied.

Cation exchangers are selective neither for Sr nor for alkaline earth elements, selectivities are higher for ions of higher charges. This selectivity can be influenced by the addition of complexing agents or using chelating resins instead of highly acidic cation exchangers. Juznic and Fedina (1986) increased the selectivity of the resin for alkaline earths by complexing trivalent cations with citric acid (pH 3.5), Y forms an anionic citrate complex. Noshkin and Mott (1967) eluted first Ca then Sr complexes with cyclohexanediaminetetra-acetic acid (CyDTA) from cation exchange resin column using 0.03 M CyDTA+0.4 M NH₃ of pH 5.1 and 6.7, respectively. Carmon (1977) retained Ca and Sr from soil solutions on cation exchange resin, and removed Ca selectively with 1.5 M ammonium lactate of pH 7, finally stripped Sr with 4 M HCl. Tait and Wiechen (1990) used a chelating resin containing aminomethyl phosphonate group to retain divalent cations including Sr directly from milk. Cations were later quantitatively eluted with 2 M HCl.

4.2. Sr pre-concentration by precipitation

In water samples and in sample solutions obtained after destruction of solid materials, Sr is often pre-concentrated by precipitation or co-precipitation together with other alkaline earth metals as the insoluble carbonates, phosphates, oxalates, sulfates, etc. (see Table 1) or their mixtures. Hydroxides have relatively high solubility, and are not adequate for pre-concentration of Sr, but many insoluble precipitates are formed in alkaline solutions.

In case of **water samples** carbonate precipitates that are formed in slightly alkaline solution are most frequently used. According to the HASL-300 procedure (1997), the recommended

IAEA procedure (IAEA, 1989a, 1989b), the methods of Borcherding and Nies (1986), Noshkin and Mott (1967), Vogl and Gesewsky (1993), and Chobola et al. (2006) alkaline earths are precipitated with carbonates from water samples. Carbonates are simply decomposed by mineral acids. Common calcium–strontium–barium oxalate precipitates are formed at lower pH (5) offering some selectivity against many metal ions. Bojanowski and Knapinska-Skiba (1990) used oxalate precipitate that was easily decomposed by ashing or wet chemical oxidation. Sulfate precipitation is less frequently chosen because of the difficulties to dissolve the precipitate (Vogl and Gesewsky, 1993). Calcium–strontium phosphate is precipitated from alkaline solutions in the procedures of Eichrom (2006) and Maxwell and Culligan (2009a, 2009b).

In case of **sample solutions** the same precipitates can be formed to pre-concentrate Sr. In case of bone, milk and urine samples, phosphate precipitates (pH 10) are frequently used, in case of biological materials, vegetation and tissues carbonates are often precipitated, while in case of soil samples oxalates are often preferred due to the lower pH. If samples are decomposed by sodium carbonate fusion than carbonate precipitates are the best choice for alkaline earths pre-concentration (HASL-300 procedures, 1997). In the most recently issued ISO standard (2009) on measurement of ⁹⁰Sr in soil three procedures are described, one is based on a series of precipitations, the other one applies extraction without preliminary precipitation and the third one uses extraction chromatography after phosphate pre-concentration.

Yttrium forms also a great variety of insoluble compounds under similar conditions as Sr, e.g. carbonates, oxalates, phosphates, sulfates, fluorides. On the contrary to Sr, Y has insoluble hydroxide that is often used to separate Y from Sr.

4.3. Chemical separation of Sr

Strontium is amenable to a variety of chemical separation procedures including precipitation/co-precipitation, liquid–liquid extraction, ion-exchange chromatography, extraction chromatography and combination of two or more of these methods.

4.3.1. Selective precipitation

A series of more or less selective precipitation reactions have been traditionally used for the separation of Sr from other alkali earth metals, yttrium and other interferences, e.g. lead. Because of the limited selectivity precipitation steps have been repeated many times. The crucial procedures are the separation of Sr from the usually big amounts of Ca, and from Ba. A final separation and purification of Sr from Y is usually also included in the methods:

- (i) *Separation of Sr from Ca*: The oldest and still popular method is based on the difference in solubilities of Ca and Sr nitrates in concentrated (fuming) nitric acid. It is simple, cheap, robust and has the great advantage that it can handle big amounts of samples. Because of the limited selectivity the precipitation steps are often repeated – according to the HASL-300 procedures (1997) – for difficult matrices sometimes 4 or 5 times. The fuming nitric acid procedure is widely used for Ca–Sr separations. It is part of the recent ISO standard (2009), the IAEA recommended procedure (IAEA, 1989a, 1989b) and many old and new methods (Dehos, 1985; Juznic and Fedina, 1986; Wilken and Diehl, 1987; Stadlbauer et al., 1988; Chu et al., 1998; Gillard-Baruh, 1973). It has one serious disadvantage that the corrosive hazardous fuming nitric acid is required. Bojanowski and Knapinska-Skiba (1990) proposed a method in which fuming nitric acid was

replaced by concentrated nitric acid. The sample in the form of mixed carbonates or oxides is stirred into concentrated nitric acid in the proportion of 1:7 w/v. Sr forms insoluble nitrate that is washed with acetone while Ca remains in solution. After two re-precipitation steps Sr recoveries were reported to be >90%. To replace fuming nitric acid Popov et al. (2009) recommended the use of NaOH. The method utilizes the difference in solubility of Sr and Ca hydroxides. From 0.4 to 0.6 M NaOH solution at 90 °C $\text{Ca}(\text{OH})_2$ precipitates while Sr remains in solution. The presence of carbonates and high salt concentrations interfere with the separation. Weiss and Shipman (1997) have shown that Sr can be separated from Ca with the organic precipitant, potassium rhodizonate but the method has not been adopted in routine analysis probably due to the need for the special reagent.

- (ii) *Separation of Sr from Ba*: ^{140}Ba is a radioactive fission product that can interfere with radiostromium determination. Furthermore the presence of stable Ba in the Sr source can cause the overestimation of the Sr chemical yield and the underestimation of the activity if recovery is determined by gravimetry. Ba is most often separated from Sr as the insoluble Ba chromate precipitate that is formed from a slightly acidic solution (pH 5.5) (HASL-300, 1997; IAEA, 1989a, 1989b). The chromate precipitation is well suited to remove Pb including the radioactive ^{210}Pb from the Sr source.
- (iii) *Separation of Sr from Y*: The Sr–Y separation can be carried out by co-precipitation of Y with ferric hydroxide. This is a typical scavenging, purification step in Sr chemistry. Y sources for beta counting are often prepared as the hydroxide or oxalate precipitates (HASL-300, 1997; IAEA, 1989a, 1989b). If the Y chemical recovery is determined by gravimetry precipitates of known stoichiometric composition are required, or more accurate relative measurements can be performed. Neither hydroxides nor oxalates meet these demands because of the varying water content of the precipitates.

4.3.1.1. The classical procedure for ^{90}Sr and ^{89}Sr determination. The most recently issued international standard (ISO 18589-5:2009) on “Measurement of radioactivity in the environment – Soil – Part 5: Measurement of strontium 90” describes three radiochemical procedures that are adequate for the determination of ^{90}Sr and ^{89}Sr in soil. The 1st procedure, called classical one, is based on precipitations. It is suited for the separation of Sr from samples of high mineral salt content. The technique is efficient but not selective, laborious and large quantities of chemicals are needed. Below we are summarizing the major steps of the procedure. Several other variants of the methods of basically the same principle are in routine use (Bojanowski and Knapinska-Skiba, 1990; HASL-300, 1997; IAEA, 1989a, 1989b; Wilken and Diehl, 1987; Lopes and Madruga, 2008; Juznic and Fedina, 1986; Dehos, 1985; Stadlbauer et al., 1988; Chu et al., 1998).

In total 1–50 g of soil can be treated. Sr carrier is added. One of the sample decomposition methods is used. The Sr is precipitated by adding 65% nitric acid (not fuming). Yttrium and other interfering elements are eliminated by precipitating the hydroxides followed by precipitation with Ba chromate. The final product is a Sr carbonate precipitate that can be measured by proportional counting (PC) or by liquid scintillation counting (LSC) after being dissolved. Another possibility is a separation of the Y after it has reached equilibrium from the Sr by precipitation in the form of oxalate and subsequent measurement by PC or LSC. The flow chart of the procedure is shown in Fig. 1.

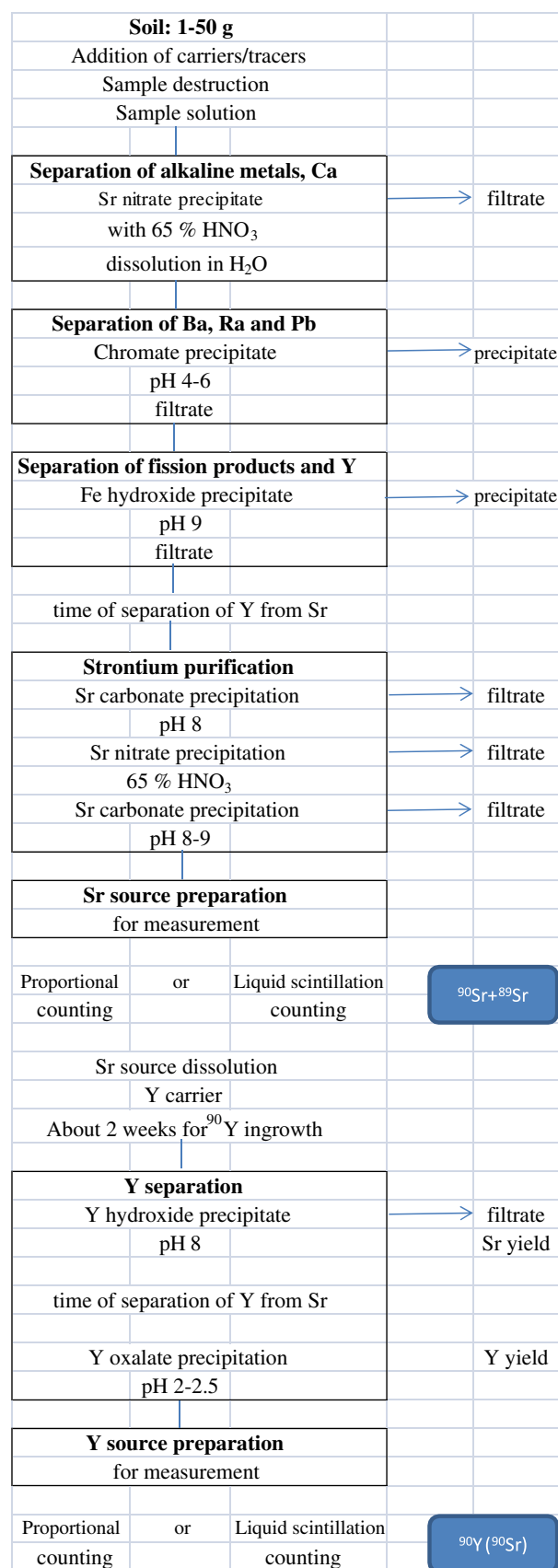


Fig. 1. The flow-chart of the classical procedure for the determination of ^{90}Sr and ^{89}Sr in soil according to ISO 18589-5:2009 standard.

4.3.2. Liquid–liquid extraction (LLE)

In LLE (also called solvent extraction) three major types of extractants can be distinguished:

- neutral extractants form neutral (ion association) complexes, also called solvating extraction systems with the analytes involving the counterions and the bonding site,
- acidic extractants form chelate complexes with the analyte cations replacing the H^+ in the organic compound in a manner similar to cation exchange, and
- basic amine extractants or ion pair formation systems contain organic tertiary or quaternary amines that form cationic groups and anions as counterions, where the anions can be replaced by anionic actinide complexes in a manner analogous to anion exchange.

The formation of ion association complexes and ion pairs in neutral and basic extractants, respectively is favored by high acid concentrations, and chelates are used in low acidity media because H^+ ions are competitors of the cations for the binding sites. Trivalent Y^{3+} cations can be well extracted/retained by chelates and they form ion association complexes with simple monofunctional organic compounds, e.g. with tributyl phosphate (TBP), but they do not have the affinity to form anions. The extractability of Y has been exploited to obtain a “fast” method for ^{90}Sr determination compared to the classical precipitation procedure. On the contrary, divalent Sr^{2+} cations are not selectively retained by chelates, do not form stable ion association complexes with monofunctional extractants and do not form anions. Recently new polyfunctional extractants have been discovered that form complexes with Sr.

4.3.2.1. Chelate extractants. Bis-2-ethylhexyl-phosphoric acid (HDEHP) is a chelate used for Y separation from slightly acidic solutions. It was introduced in the analytical practice in the sixties by Mason et al. and Fukai et al. referred by [Borcherding and Nies \(1986\)](#) to eliminate the fuming nitric acid procedure. [Borcherding and Nies \(1986\)](#) extracted Y from water with 0.45 M HDEHP in n-heptane after pre-concentration of Sr as carbonate from 30 to 100 L of seawater and a 14-day-long ingrowth time. Y was back-extracted with 9 M HCl. In case of large sea water samples a ^{234}Th contamination was discovered that could be eliminated either by a cation exchange procedure or by a co-precipitation with Zr phosphate. The procedure is often used for the final separation of Y when ^{90}Sr activity is measured via ^{90}Y . [Clark \(1995\)](#) extracted Y by HDEHP after separation of Sr using a crown ether. HDEHP extraction forms the basis of one of the three ISO standard procedures (2009) that is shown in [Fig. 2](#). Yttrium is extracted with HDEHP from the sample solution, at a pH of 1.4 and re-extracted from the organic phase with 9 M HCl. The solution is purified by extraction with t-octylmethyl ammonium chloride (TOM). Finally, Y is precipitated as an oxalate.

Ion association complexes/solvating extraction systems: The well-known organophosphorous extractant of tetra and hexavalent actinides, i.e. tributyl phosphate (TBP), is also adequate for the quantitative extraction of Y from concentrated nitric acid solutions. Already in (1967), Baratta and Ferri extracted Y from concentrated nitric acid solution (14 M) with 100% TBP followed by Y oxalate precipitation in order to analyze bone samples. [Maier and Scholl \(1982\)](#) used a similar method to determine ^{90}Sr in soil and vegetation. [Martin \(1987\)](#) determined ^{90}Sr in reactor waste by repeating the TBP extraction three times and detecting the Cerenkov radiation of ^{90}Y and got 60% recovery and a detection limit of 55 mBq/g while treating 10 g of sample. According to the basically identical method of [Zhu et al. \(1990\)](#) Y was back-

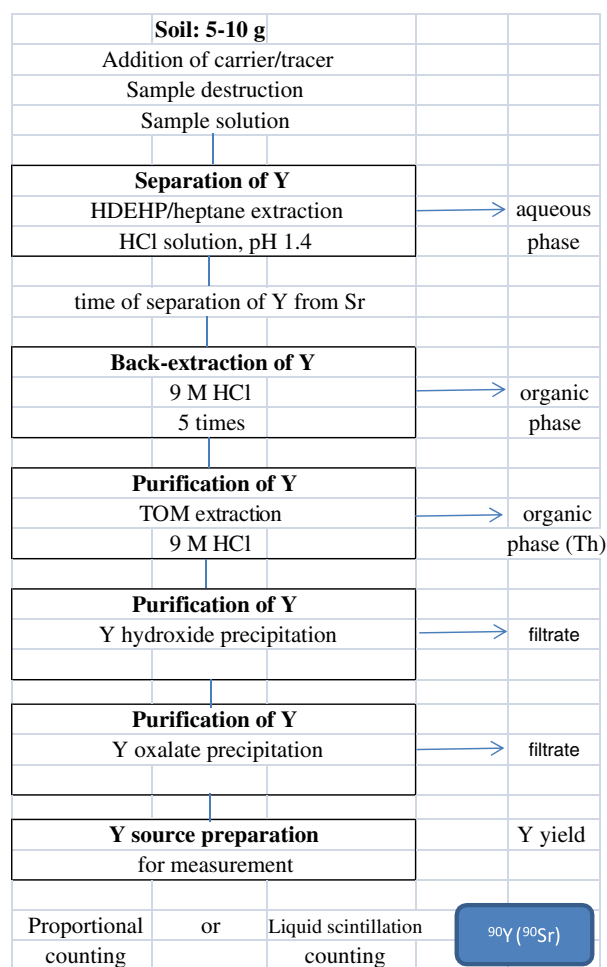


Fig. 2. Measurement of ^{90}Sr from its daughter product ^{90}Y at equilibrium by organic extraction according to ISO 18589-5:2009 standard.

extracted and precipitated as hydroxide directly from the organic phase by the addition of ethanol. The procedure of [Zhu et al.](#) was adopted by [Mietelski et al. \(1993\)](#) for the analysis of mushrooms and forest soil. To improve the separation of Y from Th, [Mikulaj and Svec \(1993\)](#) inserted a solvent extraction step in the procedure. Before the TBP extraction, Th was extracted from 8 M HNO_3 with Aliquat 336, a liquid anion exchanger. The TBP extraction and Cerenkov counting method has been used successfully till the recent times, e.g. [Jabbar et al. \(2009\)](#) applied the method for the determination of ^{90}Sr in various environmental samples from Pakistan. Other extractants and mixtures, e.g. tenoyl-tri-fluoroacetone (TTA) and t-octyl phosphine oxid (TOPO) ([Lapid et al., 1984](#)) have also been reported as efficient extractants of Y but were not superior to the HDEHP or TBP.

A new class of size selective extractants, called **crown ethers** has been discovered, developed and tested in the seventies and eighties of the last century. In (1967), Pedersen was the first to synthesize the new macrocyclic compounds that form strong complexes with alkali or alkaline earth metals. He called them “crown ethers”. Crown compounds are cyclic ethers where methylene groups are bound together with ether bridges. They are relatively soluble in water, addition of hydrophobic side chains, e.g. alkyl or cyclohexyl substituents reduces the solubility and increases the complexing power. [Kinard and McDowell \(1981\)](#) observed a correlation between the hole size of the crown compounds and the ionic size of the retained species. The most efficient crown ether for Sr retention was found to be the dicyclohexano-18-crown-6 (DC18C6) where the 18-membered

polyether ring contained 6 oxygen atoms (ether binding). The hole size is about 0.26–0.32 nm. The early experiments were focused on finding the optimal conditions for the extraction, selection of the crown molecule with the proper substituents, the solvent, the concentration of the extractant, the composition of the aqueous phase, etc. Sekine et al. (1979) extracted Sr from a picrate solution with dibenzo-18-crown-6 in to chloroform. Blasius et al. (1985) extracted Sr from waste solution containing nitric acid with DC18C6 into tetrachloroethane.

Applications of the solvent extraction procedure using crown ethers started from the end of the seventies. Kimura et al. (1977) proposed a method for the separation of Sr from large amounts of Ca from picrate solution (pH 2.5–7) into chloroform with DC18C6. The average recovery of Sr was 97% and the decontamination factor for Ca was higher than 10^4 , but Ba was not separated. The method was later improved by removing Ba with the chromate precipitation (Kimura et al., 1979). Recovery of Sr in this procedure was about 75%. Tait and Wiechen (1990) separated Ba by solvent extraction with 21-crown-7 from a solution containing picrate (pH 3–5), then Sr was extracted into chloroform with DC18C6. The recovery from 2 L milk for the whole procedure was higher than 90%. Vaney et al. (1989) determined radiostrontium in milk and water also using DC18C6 for separation of Sr from Ca. The procedure was facilitated as a transport of Sr picrate with DC18C6 through a dichlorobenzene liquid membrane (Mikulaj et al., 1993).

All these methods using selective crown ethers for Sr-Ca separation proved to be simpler and faster than the traditional precipitation methods, but the use of large organophilic counterions (picrate) and slightly acidic or alkaline conditions, and the lack of Ba separation made the procedures still complicated for the analyses of environmental samples. After systematic studies on the crown ether extraction, Horwitz et al. (1990) found that the di-*t*-butyl derivative of DC18C6 dissolved in octanol extracts Sr also from nitric acid solutions. They found a 1st order dependence of the Sr distribution ratio on nitric acid concentration. Horwitz et al. (1991a) have developed the SREX process using the *t*-butyl derivative of DC18C6 in octanol which efficiently extracts Sr from 1 to 6 M HNO_3 and became a promising method for Sr recovery from high-level wastes. The factor mainly responsible for the better extraction ability of the SREX solvent is the higher water uptake of octanol which in turn causes the effective solvation of the Sr nitrate complex. Extraction of Sr by the di-*t*-butyl DC18C6 was studied later by Kumar et al. (1999) who obtained an improved extraction constant for Sr by replacing octanol with butanol–octanol (80:20%) mixture. The extraction is optimum at 4 M HNO_3 concentration and the presence of salting-out agents enhances the extraction. Researches to develop extraction processes for the separation of Fission Products especially cesium and strontium are under investigations. In the Fission Product Extraction (FPEX) process studied at the Idaho National Laboratory by Law et al. (2007) bis-(*t*-butyl) DC18C6 and calix[4]arene-bis-(*t*-butylbenzo-crown-6) are utilized for the extraction of Sr and Cs, respectively. Parallel with the development of the SREX system Horwitz et al. prepared the supported crown ether for extraction chromatographic purposes that became a widely used technique in radiostrontium analysis (see it in the next chapter).

New extractants with higher distribution coefficients and/or special selectivity for Sr are under investigations. As candidate molecules for the selective extraction of Sr, cobalt dicarbollide and synergistic mixtures have been studied. Hydrogen dicarbollylcobaltate is the polyhedral complex of a boron containing organic molecule, i.e. $\text{H}^+[(\pi\text{-}3/1\text{-}2\text{-C}_2\text{B}_9\text{H}_{11})_2\text{Co}^-]$ in nitrobenzene. Koprda and Scasnar (1982) studied the extraction of Sr using a mixture of dicarbollide of cobalt and polyethyleneglycol (PEG).

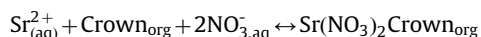
After repeated extraction they reported a Sr recovery of 99%. Kyrs and Selucky (1993) improved the method using a chlorinated cobalt dicarbollide acid and another PEG (Slovafo 909) in a CCl_4 –nitrobenzene mixture. Makrlík et al. (2008) used also hydrogen dicarbollylcobaltate in nitrobenzene in the presence of dibenzo-18-crown-6 for the extraction of Ca, Sr and Ba. Neither of these dicarbollyl mixtures proved to be superior to DC18C6 derivatives regarding the distribution coefficients and the selectivity for Sr. A rapid method for determination of radiostrontium using cation exchange resin associated with Cryptand 222 (4,7,13,16,21,24-Hexaoxa-1,10-diazabicyclo[8.8.8] hexacosane) was proposed by Tait et al. (1997).

4.3.3. Extraction chromatography (EC)

In EC, also called solid state extraction and reversed-phase partition chromatography, liquid extractants are sorbed on the surface of an inert solid support material. The theory of EC was described already more than 30 years ago in the book of Braun and Ghersemi (1975). EC has the following advantages compared to solvent extraction and ion exchange (IE):

- the extraction process takes place in the thin surface layer allowing good contact of the reagents and fast exchange kinetics compared to ion exchangers,
- as a result of the chromatographic technique more effective separation is achieved than in batch technique in which LLE is performed,
- less reagents and chemicals are used than in IE or LLC;
- less hazardous waste is produced, and
- the whole process is more economic.

Attempts have been made to replace solvent extraction by extraction chromatography from the end of the 1960s. A novel Sr-selective extraction chromatographic material was reported by Horwitz et al. (1991a, 1991b, 1992) in 1991–1992 that opened a new period in radiostrontium analysis. It was demonstrated that EC using a solution of 4,4'(5')-bis(tert-butylcyclohexano)-18-crown-6 in 1-octanol sorbed on an inert polymeric substrate provides a simple and effective method for the separation of Sr from nitric acid media. The EC material was prepared by impregnating the support material, i.e. Amberlite XAD-7, Amberchrom CG-71md or Amberchrom CG-71 cd resin. The resultant chromatographic resin is now commercially available under the name Sr Resin (it was also called Sr^*Spec) from EiChrom Industries, Inc. and TRISKEM Int. The extraction equilibrium can be described by the following equation:



The characteristics of the Sr Resin as bulk material and packed columns, the chromatographic parameters in various acids and the effect of possible interferences have been systematically studied by Horwitz et al. The log–log plot of distribution ratio of Sr (D) (defined as the ratio of the organic phase concentration of Sr to its aqueous phase concentration) versus nitric acid concentration yielded a straight line up to 3 M HNO_3 concentration with a maximum D value close to 100 what is close to or more than 2 orders of magnitude higher than the values for other alkali and alkaline earth metal ions with the exception of Ba. This means that excellent separation can be achieved from alkali metals and Ca what has been of major concern in classical radiostrontium determination. The separation of Sr from Ba can also be realized on a chromatographic column that was not possible in LLE with crown ethers. Although the separation factor for K is close to 100 Vajda et al. (1992) pointed out that interferences can occur in case of large amounts of K present,

e.g. in environmental samples. They recommended to pre-concentrate and separate Sr from alkali metals by precipitation with oxalate before EC separation using Sr Resin. Under appropriate conditions the resin can function as a Pb-selective material, the distribution ratio of Pb from a wide concentration range of nitric acid (0.01–8 M) is higher than 100. The Sr Resin can well retain tetravalent actinides from 3 to 8 M HNO_3 . Using Sr Resin, a simple and efficient separation method was obtained.

Application of the Sr Resin for the analyses of nuclear, environmental and bioassay samples is widespread, it replaced many of the older techniques. The first methods to determine radiostrontium in nuclear waste (Horwitz et al., 1991b) and urine (Dietz et al., 1991) samples were developed by the research group of Horwitz at Argonne National Laboratory. According to the method of Dietz et al. (1991), 0.6 L of urine sample was treated. After digestion Sr was preconcentrated with Ca phosphate coprecipitation. Sample solution in 2 M HNO_3 after the addition of $\text{Al}(\text{NO}_3)_3$ salting-out agent (0.5 M) was loaded on a 2 mL Sr Resin column, Ba was eluted with 8 M HNO_3 and Sr was stripped with water. Measuring the Sr source with LSC a detection limit of 0.12 Bq/L was achieved while the Sr chemical yield was 95%.

This separation procedure became the basis of one of the ISO 18589-5:2009 standard methods on 'measurement of strontium after separation on a "crown ether"-specific resin', the flowchart of the method is summarized in Fig. 3. The alkaline earth elements are precipitated in the form of phosphates in a basic medium. The barium, if present in large quantities, is removed as the nitrate. The strontium is selectively adsorbed on a Sr Resin column in a HNO_3 medium (between 3 and 8 M) then eluted using a 0.5 M HNO_3 . The beta activity is measured by LSC or using a proportional counter.

There have been other approaches to prepare supported crown ethers or to bond the crown molecules covalently to inert support. Wai and Du (1990) impregnated papers with ionizable crown ethers, i.e. dibenzo-16-crown-5-oxyacetic acid and dibenzo-16-crown-5-oxysebacic acid and demonstrated its applicability to

separate Y from Sr by varying the pH of the solution, eluting Y at pH 6–7. Kremliaikova et al. (1993) used a porous styrene-divinylbenzene copolymer as an inert support for DC18C6 in tetrachloroethane and demonstrated the possibility to separate Cs, Ba and Sr with 2 M HNO_3 eluent. Wood et al. (1993) prepared condensation polymers containing subunits of dibenzo crown ether carboxylic acid monomers. Using Na acetate eluent (pH 6.5) Y is better retained than Sr. Ermakov et al. (2008) used polymeric fibres that contained the crown ether by "flexible links" how it was defined by the authors. The advantage of the use of polymeric crown ether resins is that they can be regenerated and reused without crown ether losses. Altogether these approaches did not prove to be superior to Sr Resin.

The Sr Resin got a wide spread application in almost all fields of radiostrontium determination. There was a long way of development work till the new EC procedure became the basis of national and international standards.

Vajda et al. (1992) described a simple method for Sr separation for soil samples that comprised a pre-concentration with oxalate and an EC separation on a big (3 g) Sr Resin column. In the method of Vincze et al. (1998) for the analysis of NPP samples, Ag_2S scavenging is included in a similar procedure to improve decontamination from the radionuclides present in "fresh" samples of reactor coolant origin. The rapid method of Baron et al. (2004) for radiostrontium determination in bone consists of oxalate co-precipitation and separation on Sr Resin column and takes 24 hours. To achieve better separation from Pb in bone samples, Mietelski and Gaca (2002) made a Pb iodide precipitation before counting. Jeter and Grob (1994) developed a method for milk samples in which 1 L milk was either ashed or passed through a cation resin column followed by co-precipitation of alkaline earths, and separation of Sr on a big Sr Resin column (3 g). In the rapid method of Brun et al. (2002) the pre-concentration of Sr from milk by cation exchange was also followed by precipitation and EC on Sr Resin. To improve the separation of Sr from Ba, Ba chromate was precipitated in the conventional manner. The method of Spasova et al. (2008) for milk analysis is simple, consists of oxalate precipitation and separation on Sr Resin, but milk samples are ashed before chemical processing what makes the procedure long. The rapid method of Maxwell and Culligan (2009a, 2009b) takes only 8 h including the 20 min long counting. Calcium phosphate is precipitated directly from 100 mL milk sample, the precipitate is ashed and Sr is separated on Sr Resin column. The method has an acceptable low detection limit (0.5 Bq/L) for analysis in emergency situation. Clark (1995) developed a method for the determination of radiostrontium in Ca carbonate matrices of shells, molluscs, etc. that consist of a simple EC separation on Sr Resin.

Before the publication of the ISO 18589-5 standard in 2009, the Eichrom methods SRW-01 for the determination of strontium-89, 90 in water from 2003, and ACW17 VBS for the analysis of americium, neptunium, plutonium, thorium, curium, uranium, and strontium in water with Vacuum Box System from 2006, the new ASTM standard test methods for radiochemical determination of strontium-90 in soil (C 1507-07) and in water (D 5811-08) from 2007 and 2008, respectively, were published. In all these procedures small (2 mL) Sr Resin columns are used for Sr separation. According to the C 1507-07 method Sr is separated from 5 g of soil solution without pre-concentration, according to the D 5811-08 and SRW 01 procedures Sr is pre-concentrated on a cation resin from 1 L water followed by EC separation on Sr Resin, and according to the combined ACW17 VBS procedure Sr is pre-concentrated together with actinides using Ca phosphate from 1 L water, actinides are separated using selective EC materials and finally Sr is retained on a Sr Resin. Many other authors use Sr Resin for the separation or purification of Sr for

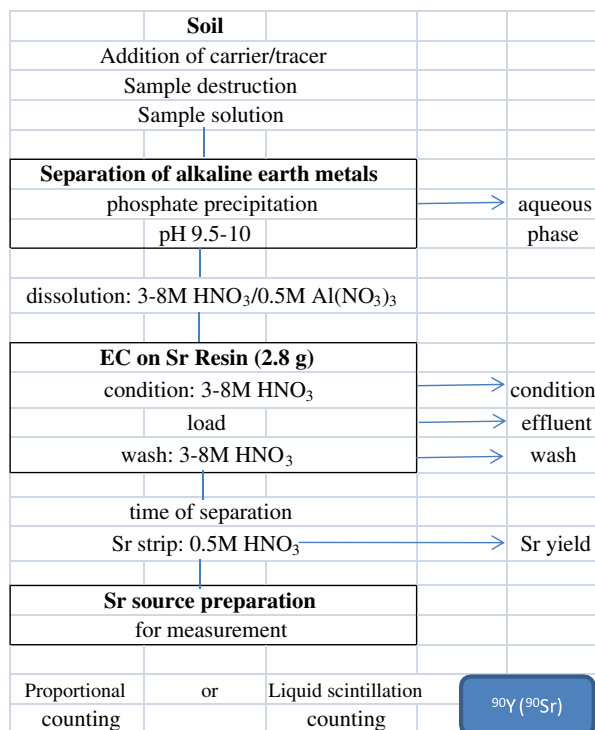


Fig. 3. Measurement of ^{90}Sr by extraction chromatography using Sr Resin according to ISO 18589-5:2009 standard.

radiostrontium determination purposes (Lee et al., 2002; Saxen, 2002; Eikenberg et al., 2006; Gaca et al., 2006; Landstetter et al., 2008; Minne et al., 2008; Asgharizadeh et al., 2008; Wallova and Wallner, 2008; Chmielewska et al., 2008; Lopes and Madruga, 2008; Muynck et al., 2009; Chobola et al., 2006, etc.).

The new EC techniques perfectly suit for the purposes of inductively coupled plasma mass spectrometry (ICP-MS) or on-line flow scintillation analysis where sample and reagents are directed through chromatographic columns to the ICP-MS or the LS counter, respectively.

Taylor et al. (2007) determined ^{90}Sr in contaminated environmental samples by dynamic reaction cell (DRC) ICP-MS. Water samples (up to 1 L) were pre-concentrated on cation exchange resin. The Sr content of the samples (water, plant, sediment) was separated twice using 4 mL Sr Resin cartridges. Additionally, Fe and Ni responsible for molecular isobaric interferences were removed from the samples using a chelating resin. Chemical recoveries varied between 68 and 98%. Method detection limits (0.2–0.5 Bq/g for 2 g of plant or sediment; 5 Bq/L for 1 L water) do not rival with those of radiometric methods. Feuerstein et al. (2008) determined ^{90}Sr in soil samples contaminated by the Chernobyl accident by DRC ICP-MS also using Sr Resin column for Sr separation. Separation from the isobaric ^{90}Zr interference was eliminated by reaction with O_2 in the DRC unit. A detection limit of about 1 Bq/g was achieved when 1 g soil containing Zr was analyzed. Plionis et al. (2009) determined ^{90}Sr in bioassay samples by high performance EC and flow scintillation analysis (FSA). For the separation 30 or 150 mm long Sr Resin columns (4.6 mm I.D.) were loaded with 8 M HNO_3 sample solution and the strip solution was counted both with scintillation detector on-line and with LSC off-line. No loss of Sr recovery was observed. Detection limit of about 1–2 Bq/sample was achieved for simulated and diluted urine samples. Roane et al. (2003) investigated the possibility to determine radiostrontium and actinides by on-line radiochromatography using both EC and IC. About 0.1 g soil sample was destroyed, Sr and actinides were pre-concentrated using Sr Resin and TRU resin, respectively, followed by their ion chromatographic separation using cation exchange pre-concentration and mixed bed separation columns. For sequential elution oxalic, diglycolic and nitric acids were applied. The eluate was counted with a scintillation detector in near real time. Although interferences in the separation were identified the method represents an approach to analyze several radionuclides in a rapid and automated manner.

A Sr selective disk, Empore™ Strontium Rad Disk (L. Smith et al., 1996) has been developed for the analysis of radiostrontium in environmental water samples. The disk is commercially available from 3 M Company, US. The extraction disk contains a proprietary multicyclic macrocycle covalently bonded to a silica solid support embedded in a stable, inert matrix of polytetrafluoroethylene fibrils. The exact molecular structure of the extractant has not been given. In the simple and rapid procedure sample solution acidified to 2–4 M with HNO_3 or HCl is pulled through the disk with vacuum that is subsequently assayed for beta counting. A one-liter aqueous sample containing up to 3 mg Sr can be treated in about 20 minutes. Alternatively, ^{90}Y daughter can be stripped from the disk after a given in-growth period with 2 M HNO_3 . If sample contains ^{228}Ra – ^{228}Ac , ^{140}Ba – ^{140}La or ^{210}Pb – ^{210}Bi than Y is stripped with a 95% methanol–5% 10 M HNO_3 mixture and is subsequently purified using an anion exchange chromatographic procedure. In the simple and fast radiostrontium assay procedure high recoveries (close to 100%) are attained for water sample, but interferences due to Ba, Ra and Pb isotopes cannot be excluded. Empore™ Strontium Rad Disks are expensive, this might be the reason for the limited use of this material.

4.3.4. Ion exchange/ion chromatography

Ion exchange resins are high-molecular weight organic polymers, e.g. styrene and di-vinyl-benzene co-polymers containing various functional groups covalently bound to the polymer network. The most frequently used *cation exchangers* are the strongly acidic sulfonated resins containing $-\text{SO}_3^-$ groups, and less acidic phosphorylated and carboxylated resins are also produced. The positively charged counterions can be replaced by any other cations. The ion exchange affinity of the cations increases with increasing charge and decreasing hydrated ion radius. The cation exchange behavior of Sr and Y were studied in various solutions of acids, i.e. HClO_4 , HBr, HCl, HNO_3 , etc. Trivalent Y^{3+} and divalent Sr^{2+} cations are retained well (high D) at low acidities and they are removed from the resin by concentrated acids (Lederer, 1992).

Cation exchangers have been used for the pre-concentration of Sr from high volume water and milk samples (see in detail in the chapter on pre-concentration of Sr) and for the chromatographic separation of Sr (see below) in Ion Chromatographs (IC).

Lamb et al. (1989) separated Sr from nuclear reprocessing solutions up to 500 mL using automated analytical ion chromatography that contained an acid suppression system to reduce the acidity of the load solution to near neutral ($\text{pH} > 2.3$), four cation concentrator columns and a cation separator column. In the acid suppressor anions are replaced by the OH^- ions of t-butylammonium hydroxide through a liquid anion exchange membrane. Cations together with Sr are removed from the concentrator column with diaminopropionic acid (DAP) or histidine and are chromatographically separated on the analytical column. Strontium fraction detected by a conductivity detector is collected for subsequent radiostrontium analysis by liquid scintillation counting. Results with simulated reprocessing solutions have shown quantitative Sr separation and recovery. Lazare et al. (2009) determined ^{90}Sr in primary coolant of pressurized water reactors using high pressure ion chromatography. Water samples up to 25 mL spiked with Sr carrier were passed through a CG12A and a TCC pre-concentration column and cations were separated on a CS12A column using methane sulfonic acid (MSA) eluent. Strontium fraction detected by the conductivity detector was collected and subsequently analysed by LSC. The global Sr yield was 90%, the detection limit was 2.8 Bq/L using 25 mL sample solution. Fjeld et al. (2005) used cation resin column for the pre-concentration of actinides and Sr from groundwater and liquid radioactive wastes. Sr, U, Pu and Am were separated by ion chromatography and detected off-line with scintillation detector. Detection sensitivity of the method was low due to small sample masses.

IC separation fits well to the purposes of radiostrontium determination from high activity samples, e.g. reactor coolant, reprocessing solutions or high level wastes, but is less adequate for the determination of radiostrontium in low level environmental samples because of the presence of relatively big amounts of stable Sr.

4.4. Source preparation for beta particle detection

For the accurate determination of ^{90}Sr and ^{89}Sr sources for beta counting, liquid scintillation spectrometry or Cerenkov counting have to be prepared. Beta counting techniques require solid sources that can be prepared simply by evaporation or precipitation from the separated and purified analyte. Sr is usually precipitated as carbonate or oxalate, Y is precipitated as oxalate. Sources for LS counting or spectrometry are prepared by simply mixing the analyte solution with a liquid scintillator. Sources for Cerenkov counting are simple colorless sample solutions.

5. Combined procedures for the determination of Sr and actinides

The demand for the determination of radiostrontium isotopes and actinides from single sample aliquot can be met by the development of sequential separation methods. These procedures also have that great advantage that the usually tedious sample pre-treatment is performed only once for the determination of the various analytes. In the combined procedures, actinides are concentrated and removed by co-precipitation, anion exchange and/or extraction chromatography and Sr is removed usually by EC with Sr Resin. Methods used for the separation of Pu have been reviewed recently by Vajda and Kim (2009). Almost parallel to the development of crown ether based procedures a major breakthrough occurred in the analysis of transuranium nuclides by developing various transuranium element-specific EC materials, i.e. the UTEVA, TRU, TEVA resins. These are the registered trade names of materials produced by Eichrom Co. EC techniques can be advantageously combined in to procedures for the simultaneous determination of actinides and radiostrontium isotopes, where the EC materials are used either separately or also in the form of stacked columns.

Anion exchange chromatography often forms the basic part of a combined procedure. Pu and Th can be directly separated by anion exchange resins from concentrated HNO_3 solution while Sr and Am together with other sample components are collected in the effluent. After preconcentration e.g. with Ca oxalate, Am can be separated using a TRU column and further purified from lanthanide impurities either with an anion exchange resin or a TEVA resin. Finally, Sr is separated on a Sr Resin column. This flow-chart forms the basis of several procedures to determine Pu, Am and Sr isotopes in environmental samples (Moreno et al., 1997, 1998; Michel et al., 2008a, 2008b; Wang et al., 2004; Tavcar and Benedik, 2005; Martin and Odell, 1998, and Srnec et al., 2008).

Berlitz et al. (2005) combined the cation exchange separation of Am with the separation of Pu and Sr using anion exchange and Sr Resin chromatography, respectively. Ageyev et al. (2005a, 2005b) applied the classical separation scheme for the determination of radiostrontium attached to a similar combined procedure.

Recently, a great variety of combinations of EC procedures have been reported that seem to be adequate for the determination of Sr and various actinides. Sr Resin is used in all of these procedures for the separation of Sr usually with acceptably high Sr chemical recoveries (> 50%). Horwitz et al. (1995) and Thakkar (2001/2002) recommended to use UTEVA and TRU tandem columns for the separation of actinides where U and Th are retained on the UTEVA column while Am and Pu are retained on the TRU column from 2 to 3 M HNO_3 solution. Sr can be recovered from the effluent and can be separated on a Sr Resin column. This procedure has been applied by LaRosa et al. (2001), Mellado et al. (2001). For the rapid determination of actinides and radiostrontium Maxwell (2006, 2009) recommended the use of TEVA, TRU and Sr Resin columns where Pu and Th are separated on TEVA, Am and U on TRU and Sr on Sr Resin column. All actinides can be separated on a single TRU column and Sr can be retained and separated from the effluent on Sr Resin column. This procedure was applied by Spry et al. (2000); Olahova et al. (2005).

In the combined procedures actinides are usually separated first followed by the separation of Sr using the Sr Resin. This separation sequence is advantageous for radiostrontium determination because the actinides are not loaded on the Sr Resin column that could retain the tetravalent species, thus the decontamination of actinides in the Sr source is improved.

6. Comparative evaluation of different separation procedures

There are only a limited number of publications that compare radiochemical procedures experimentally.

Three methods for the determination of radiostrontium isotopes, i.e. the classical one using fuming nitric acid, the method using ion exchangers, and the extraction chromatographic method using the Sr Resin were compared by Chu et al. (1998). Three counting systems, i.e. GM counters, proportional counters and Cerenkov counters were also comparatively evaluated. The classical procedure contained oxalate precipitation, fuming nitric acid separation, chromate precipitation, followed by carbonate, hydroxide and repeated carbonate precipitations. In the ion exchange procedure alkaline earth elements were pre-concentrated as oxalates followed by their separation from ammonium acetate solution on a cation exchange resin. In the 3rd procedure after the oxalate pre-concentration Sr was separated by EC on Sr Resin column. All results obtained by the analyses of soil, rice, tea leaves and milk by the three methods agreed well. The time needed for chemical separation with the Sr Resin is much shorter than for the other two methods. The detection limit for the Cerenkov counter (due to the high background of the LSC) is about twice as high as for low background beta counters. Three extraction chromatographic materials, i.e. the Empore™ Strontium Rad Disk, the AnaLig Sr-01 (“molecular recognition ligand covalently bond to inert support”, produced by IBC Advanced Technologies, the exact composition is not known) and Sr Resin were tested by Dulanska et al. (2008). It was reported that all three materials were suitable for the determination of ^{90}Sr in water samples. Slightly higher Sr recoveries were reported for the 1st two methods.

Although comparison of experimental results are very useful to estimate the performance of the radiochemical method, it cannot replace individual method validation for the given sample type and measurement conditions.

The performance parameters of selected radiochemical procedures are summarized in Table 4.

7. Beta particle detection and calculation of results

As it was mentioned in the 1st chapter, beta particles can be detected by a variety of nuclear detectors including gas ionization detectors (proportional and Geiger Müller counters), solid and liquid scintillation detectors, semiconductor detectors and Cerenkov detectors. A comparison of proportional counters, Cerenkov detectors and LS counters was made by Poletiko et al. (1994), Wilken and Joshi (1991). The type of detector basically determines the measurement method and the way of calculation of the results. To calculate the activities of ^{90}Sr and ^{89}Sr , in general, the measurement of two count rates are required. The activity of ^{90}Y can always be calculated from the activity of the ^{90}Sr parent. To determine the two unknown parameters either two independent measurements of the same source in different time intervals are needed, or after measuring the Sr source the ^{90}Y daughter can be separated and counted, or two independent data can be obtained by simply measuring the Sr-Y source by beta spectrometry using at least two windows.

If ^{89}Sr is absent and only ^{90}Sr is present in the sample than the activity of ^{90}Sr is determined by the following ways:

- Sr is separated radiochemically, and counted immediately for a relatively short counting time, ^{90}Y ingrowth is neglected. This is a typical method for rapid determination of ^{90}Sr that has an inherent source of error.
- Sr is separated radiochemically, and after a two week-long ingrowth period ^{90}Sr in secular equilibrium with ^{90}Y is

Table 4

Performance parameters of selected radiochemical procedures.

Title	Authors	Year	Analyte	Measurement	Sample		Digestion	Pre-concentration	Main separation	Sr yield	Sr DF (carry-over)	Sr LD
					Matrix	Amounts (g, L)						
Determination of ^{90}Sr in contaminated samples by tuneable bandpass dynamic reaction cell ICP-MS	V. F. Taylor, R. D. Evans, R. J. Cornett	2007	^{90}Sr	DRC-ICP-MS, Cerenkov	Sed., plant, water	2 g, 2 g, 1 L	MW	Water: cation resin (50 mL)	Sr Resin, 2x, chelating resin (Fe, Ni)	68–98%		Sed. 0.5 Bq/g, plant 0.2 Bq/g, water 5 Bq/L
Determination of ^{90}Sr in soil samples using inductively coupled plasma mass spectrometry equipped with dynamic reaction cell (ICP-DRC-MS)	J. Feuerstein, S. F. Boulyga, P. Galler, G. Stinger, T. Prohaska	2008	^{90}Sr	ICP-DRC-MS, multicoll. ICP-MS	Soil	1 g	HF/HClO ₄ in Teflon bomb		Sr Resin, Zr removal by DRC & cold plasma			20–1000 Bq/kg
Accelerator mass spectrometry of strontium-90 for homeland security, environmental monitoring, and human health	S. J. Tumey, T. A. Brown, T. F. Hamilton, D. J. Hillegonds	2008	^{90}Sr	AMS								Planned LD 10 ⁶ atoms
Trace determination of ^{90}Sr and ^{89}Sr in environmental samples by collinear resonance ionization spectroscopy	J. Lantzs, B. A. Bushaw, V. A. Bystrow, G. Herrmann, H.-J. Kluge, S. Niess, E. W. Otten, G. Passler, R. Schwalbach, M. Schwarz, J. Stenner, N. Trautmann, K. Wendt, Y. V. Yushkevich, K. Zimmer	1995	^{90}Sr , ^{89}Sr	Collinear RIMS	Air filter				Chemical separation			^{90}Sr : 10 ⁷ atom (8 mBq) in the presence of 10 ¹⁷ atoms Sr (1.2 mBq/m ³); ^{89}Sr : 10 ⁸ atoms
Ultratrace analysis of long-lived radioisotopes in the environment	N. Trautmann	1995	^{90}Sr , ^{89}Sr	Radiometric, MS (AMS, RIMS)								AMS: > 10 ⁵ atoms, RIMS: 10 ⁶ atoms of ^{90}Sr
Determination of low-level ^{90}Sr in environmental materials: a novel approach to the classical method	R. Bojanowski, D. Knapinska-Skiba	1990	$^{90}\text{SrO}_3$		Large samples (sea water, soil)			Oxalate, carbonate	Nitrate: cc. HNO ₃ (3 cycles), chromate, carbonate	86–90% (nitrate ppt.)		
HASL-300, Sr-01-RC, Sr-02-RC	M. D. Erickson (director)	1997	^{90}Sr , ^{89}Sr	Beta scint.	Envir.				Classical			^{90}Sr : 3–5 mBq/sample; ^{89}Sr : 5 mBq/sample (in mixture higher)
Determination of actinides and ^{90}Sr in spent ion exchange resins	P. Tavcar, L. Benedik	2005	Ac, Np, Sr	Alpha	Spent resin	30 g	Fenton's reagent (H ₂ O ₂ +F-e ²⁺)		Dowex-1 TRU Sr Resin	77–85%		
Separation of strontium-89 and strontium-90 from calcium in milk with a macrocyclic ether	T. Kimura, K. Iwashima, T. Ishimori, T. Hamada	1979	^{90}Sr , ^{89}Sr	Low bkg GM counter	Milk	1.5 L		Oxalate, hydroxide scavage	LLE: 0.012M DCH18C6, chromate, carbonate, Y hydroxide	69–81%	DF > 10 ⁴ for mg, Ca, Ru, I, Cs, Ba, La	
Strontium-90 in environmental samples from Northern Germany before and after the chernobyl accident	R. D. Wilken, R. Diehl	1987	^{90}Sr	Proport. ^{90}Y or double counting of Sr and Y source	Soil, milk, plants, grass	5 kg, 5 L, 2 kg, 2 kg	Leaching 1:1 HCl	Sulphate, carbonate	Fuming HNO ₃ (Ca/Sr), chromate (Ba/Sr), hydroxide scavage, Y ppt.			Few mBq/kg

Table 4 (continued)

Title	Authors	Year	Analyte	Measurement	Sample		Digestion	Pre-concentration	Main separation	Sr yield	Sr DF (carry-over)	Sr LD
					Matrix	Amounts (g, L)						
Application of liquid scintillation counting technique to the determination of ^{90}Sr in milk samples	I. Lopes, M. J. Madruga	2008	^{90}Sr	LSC: TR3170	Milk	1 L (raw)		Oxalate, carbonate	Sr Resin (3 g) (reuse)	77		0.012 Bq/L
Determination of ^{137}Cs and ^{90}Sr in milk by the static method with an ion exchanger	J. Oravec, I. Navarcik	1987	^{90}Sr , ^{137}Cs	Propor. (Y)	Milk	10 L		250 mL formaldehyde	Cation resin (275 mL) to concentrate Sr, Cs, hydroxide, chromate, Y oxalate	87.5% (Y: 92.5%)		
Rapid trace determination of radiostromium in milk and drinking water	B. Vaney, C. Friedli, J. J. Geering, P. Lerch	1989	^{90}Sr	GM counter with active shield	Milk, water	500 mL		Milk: Cation (65 mL), pH 5.3, carbonate	LLE: DC18CE6/CH2Cl2, carbonate, hydroxide, chromate	Milk: 74%, water: 91%	$< 10^{-4}$	0.03 Bq/L
Determination of radiostromium in milk using an extraction chromatography column	H. W. Jeter, B. Grob	1994	^{90}Sr , ^{89}Sr	Propor. (sr & Y source counted once)	Milk	1 L		(1) Ca phosphate (2) cation (170 mL)-batch, column, Ca CO3	Sr Resin (3g), repeated use (10x), Y hydroxide	Ash: 65%, ion-exchange 80%, classical 50%		
Application of calcium carrier to avoid losses of ^{90}Sr during the chemical preparation of liquid samples for liquid scintillation spectrometry	I. Chmielewska, S. Chalupnik, E. Bokori	2008	^{90}Sr	LSC	Water	10 L		Evaporation	Sr Resin			0.01 Bq/L
ASTM Standard Test Method for strontium-90 in water		2008	^{90}Sr	Propor.	Water	1L		Cation resin (10 mL)	Sr Resin (2 mL)			0.037 Bq/L
Separation of strontium from large amounts of calcium, with application to radiostromium analysis	V. E. Noshkin, N. S. Mott	1967	Sr-Ca		Sea water	500 mL-55 L		Carbonate pH 7 (EDTA)	Cation exchange, DCYTA	75% (500 mL sea water)		
Use of liquid scintillation counting for fast determination of ^{89}Sr and ^{90}Sr in milk	D. Tait, A. Wiechen	1990	^{90}Sr , ^{89}Sr	LSC: 3-windows, spectrum stripping technique	milk	2 L		Chelating resin carbonate	LLE: 21-crown-7 (Ba), DCH18crown6 (Sr), carbonate	90%	DF: for Ba 79, for Ca > 620, Y 16000	
A rapid method for the determining ^{89}Sr and ^{90}Sr from nuclear accidents	K. Vogl, P. Gesewsky	1993	^{90}Sr , ^{89}Sr	LSC: counting immediately 2 windows	Air filter, vegetation soil, water		Leaching	Sulfate (pH 7), carbonate, nitrate, hydroxide, chromate	Sr carbonate			0.1 Bq/sample
Rapid determination of radiostromium isotopes in samples of NPP origin	R. Chobola, P. Mell, L. Daróczy, A. Vincze	2006	^{90}Sr , ^{89}Sr	LSC: 3 counting window, decay low correction	NPP samples: water, air filter	5 L	Leaching	Carbonate	Cation (pH 1) with Cu ring indicator, Sr Resin	60%		^{89}Sr : 30 mBq/sample, ^{90}Sr : 18 mBq/sample (600 min)
Americium, neptunium, plutonium, thorium, curium, uranium, and strontium in water (with Vacuum Box System)	EiChrom procedure	2006	^{90}Sr	propor.	water	1L		phosphate	TEVA(Pu, Th, Np), TRU (U, Am), Sr Resin, (DGA: Ac, Fr)	74–89%		

Rapid separation method for emergency water and urine samples	S. L. Maxwell, B. K. Culligan	2009	Am, Pu, U, Sr	Alpha (30–45 min), propor. (10 min)	Water urine	400 mL 100 mL		Ca phosphate pH 10	TEVA (Th,Pu)-TRU (Am,U) Sr Resin (Sr)			Sr: 0.26 Bq/L (100 mL, 10 min counting)
Feinreinigung von Radiostrontiumproben mittels Ionenchromatographie	E. A. Stadlbauer, C. Trieu, H. Hingmann, H. Rohatzsch, J. Weiss, R. Maushart	1988	⁹⁰ Sr, ⁸⁹ Sr		Envir.			Carbonate, nitrate or cation resin	IC: surface sulfonated cation resin HPIC-CS2	80% (with nitrate pre-conc.)		5 Bq/L or 5 Bq/kg
Separation and determination of radiostrontium in calcium carbonate matrices of biological origin	S. B. Clark	1995	⁹⁰ Sr, ⁸⁹ Sr	Cerenkov (check ⁸⁹ Sr), LSC (2 ROIs)	Shells (high Ca:Sr=500)		Wet destruction	None	Sr Resin, Y HDEHP	> 90%		
Measurement of ⁹⁰ Sr in reactor Wastes by Cerenkov counting of ⁹⁰ Y	J. E. Martin	1987	⁹⁰ Sr	Cerenkov, decay curve to control purity	Waste	10 g	cc. HNO ₃		LLE: TBP 3x	0.6		55 Bq/g
⁹⁰ Sr and ²³⁹⁺²⁴⁰ Pu, ²³⁸ Pu and ²⁴¹ Am in some samples of mushroom and forest soil from Poland	J. W. Mietelski, J. LaRosa, A. Ghods	1993	⁹⁰ Sr, ²³⁹⁺²⁴⁰ Pu, ²³⁸ Pu, ²⁴¹ Am	Cerenkov (2M HCl), alpha	Mushroom, soil		Leaching: 65% HNO ₃		LLE: TBP(Y), Y oxalate			
Determination of ⁹⁰ Sr in environment of district Swat, Pakistan	T. Jabbar, K. Khan, M. S. Subhani, P. Akhter	2009	⁹⁰ Sr	Cerenkov	Envir. (soil, vegetation, water)				Y—TBP extr.	75–90% (Y)		0.03 Bq
Separation of strontium ion from a large amount of calcium ion by the use of a macrocyclic ether	T. Kimura, K. Iwashima, T. Ishimori, H. Hamaguchi	1977	Sr		model				LLE: DCH18C6	97%		
Extraction of radiostrontium from the mixture of radionuclides in milk using dicarbollide of cobalt	V. Koprda, V. Scasnar	1982	⁹⁰ Sr		milk, water				LLE: (1) Cs, Rb—0.1 M dicarbollide-H (2) Sr, Ba—0.01 M dicarbollide-H+0.25% PEG	99% (double extr.)		
Rapid concentrating of radiostrontium from model fallout, drinking and technological water using solvent extraction with cobalt dicarbollide and Slovafo 909	M. Kyrs, P. Selucky	1993	Sr		Water	1 L				80–97%		
The application of novel extraction chromatographic materials to the characterization of radioactive waste solutions	E. P. Horwitz, M. L. Dietz, R. Chiarizia							Actinides removed by TRU	Sr Resin (10 cm, d=1.4 mm), 0.6 mL	99%	DF > 100 (Al), 10 ⁵ (Ca)	
Determination of radiostrontium in soil samples using a crown ether	N. Vajda, A. Ghods, P. Danesi	1992	⁹⁰ Sr	LSC	Soil, sediment	10 g		Oxalate	Sr Resin	60–80%		
An improved method for determining ⁸⁹ Sr and ⁹⁰ Sr in urine	M. L. Dietz, E. P. Horwitz, D. M. Nelson, M. Wahlgren	1991	⁹⁰ Sr, ⁸⁹ Sr	LSC: double window (⁹⁰ Sr, ⁸⁹ Sr) immediately	Urine	0.6 L	Nitric acid digestion (3 h)	Ca phosphate	Sr Resin (2 mL)	95%	> 76 (smallest for Po, Th, Hg)	0.12 Bq/L
Determination of radiostrontium in environmental objects by extraction chromatography using TVEX-DCH16C6	N. Yu. Kremlyakova, A. P. Novikov, B. F. Myasoedov	1993	⁹⁰ Sr	Beta counter (Tesla)	Envir.		Leaching: 7.5 M HNO ₃	None	tvex-DCH18C6, Sr carbonate	> 95% (no need for yield measurement!)	For Cs, Ce, Ru, Y: 100–1000	

Table 4 (continued)

Title	Authors	Year	Analyte	Measurement	Sample		Digestion	Pre-concentration	Main separation	Sr yield	Sr DF (carry-over)	Sr LD
					Matrix	Amounts (g, L)						
A new method of radiostromtium content determination using synthetic selective sorbents	A. I. Ermakov, I. A. Kashirin, A. I. Bobolev, V. A. Tikhomirov, A. V. Kovalev, V. B. Ribalka, V. V. Serbinovich		⁹⁰ Sr	Low bkg alpha/beta counter-LSC	envir. E.g. soil	100 g		No need for coppt.	CE bound to polymeric fibers, sulfate	50–60%		
Determination of radiostromtium in samples of NPP origin	A. Vincze, E. Erdős, J. Solymosi, I. Gresits, P. Mell, L. Török, R. Chobola, P. Ormai, A. Fritz, T. Pintér	1998	⁹⁰ Sr, ⁸⁹ Sr	propor.	NPP waste concentrate	100 mL (500 mL)	Leaching/destruction	Oxalate, Ag ₂ S scavage	Sr Resin, Y oxalate	51–67%		
Microwave digestion for rapid radiostromtium analyses in salmon fishbone	G. Baron, S. Brun, A. S. Griere, S. Metz, B. Boursier	2004	⁹⁰ Sr	Propor. LB1500 (α/β)	Fishbone	1.5 g	Microwave: 7.8M HNO ₃	Ca oxalate	Brun procedure: Sr Resin	73		24 Bq/kg
Measurement of ⁹⁰ Sr and ²⁴¹ Pu in various matrices	J. W. Mieltski, P. Gaca	2002	⁹⁰ Sr, ²⁴¹ Pu	LSC	Bone, dust			With or without oxalate	Sr Resin	71–91%		
Rapid method for the determination of radiostromtium in milk	S. Brun, S. Bessac, D. Uridat, B. Boursier	2002	⁹⁰ Sr, ⁸⁹ Sr	Propor.	milk	0.5 L	None	Cation resin (100 mL) pH 5.3, oxalate	Sr Resin, chromate	48–74%		0.09 Bq/L (LT 3600 s)
Rapid method for determination of radiostromtium in emergency milk samples	S. L. Maxwell, B. K. Culligan	2009	⁹⁰ Sr, ⁸⁹ Sr	Propor. (20 min)	milk	100 mL (500 mL)		Ca phosphate pH 10, ashing	Sr Resin 3 mL cartridge	93%		0.5 Bq/L
Sequential radiochemical separation of ⁹⁰ Sr and ⁹⁹ Tc in aqueous samples and measurement by liquid scintillation counting	M-Ho Lee, K-Ho Chung, Y. -H. Cho, C.-W. Lee	2002	⁹⁰ Sr, ⁹⁹ Tc	LSC: Full Spectrum DPM, spectrum unfolding; gas propor.	Aqueous samples, waste	1–50 L		Anion exchange, pH 6	Sr: Sr Resin, Tc: TEVA Sr carbonate	70%		
Determination of ⁹⁰ Sr by extraction chromatography in the presence of ²¹⁰ Pb in environmental samples	R. Saxen	2002	⁹⁰ Sr	LSC or prop.	envir.: water,		Na ₂ CO ₃ fusion	Fe hydroxide scavage, Pb-Ba chromate, carbonate	Sr Resin (3g)	71–88%		0.0017 Bq/sample
Precise determination of ⁸⁹ Sr and ⁹⁰ Sr/ ⁹⁰ Y in various matrices: the LSC 3-window approach	J. Eikenberg, H. Beer, M. Rüthi, I. Zumsteg, A. Vetter	2006	⁹⁰ Sr, ⁸⁹ Sr	LSC: 3-window, Cerenkov	Soil, grass, urine, waste	0,2 L		Oxalate or carbonate	Sr Resin			< 0.1 Bq/L (5 × 100 min, 0.2 L water); < 0.5 Bq/kg (2 h, 30 g soil)
Determination of ⁹⁰ Sr in milk in Austria	C. Landstetter, N. Cernohlawek, C. Katzlberger	2008	⁹⁰ Sr	LSC(Quantulus - HiSafe cocktail)	Milk	30 mL (raw)	Leaching: 65% HNO ₃ , 3 h	Oxalate	Sr Resin (2 mL)			0.12 Bq/L (2 mL resin, 30 mL milk); 0.01 Bq/L (5.4 g resin, 300 mL milk)
Determination of ⁹⁰ Sr concentration in soil and sediment samples from Southern shores of Iran using a Sr Resin and LSC method	F. Asgharizadeh, B. Salimi, M. Ghannadi Maragheh, M. Khayatzaeh Mahani, M. Aliabadi	2008	⁹⁰ Sr	LSC	Soil, sed. (10 g ash)			Oxalate	Sr Resin (10 cm), oxalate	50–95%		0.33 Bq/kg (5 h)

Evaluation of flow scintillation analysis for the determination of Sr-90 in bioassay samples	A. A. Plionis, E. R. Gonzales, S. Landsberger, D. S. Peterson	2009	⁹⁰ Sr	Flow scin. Analyzer	Water, urine			Sample dilution for urine	Sr Resin—HPEC (high performance extr. Chrom.)	About 100%	About 1 Bq
The use of extraction chromatography resins to concentrate actinides and strontium from soil for radiochromatographic analyses	J. E. Roane, T. A. DeVol, J. D. Leyba, R. A. Fjeld	2003	Sr, Pu, Am, U	On-line scint. glass (Ce activated)	Soil	< 0.5 g	Leaching or acid destr.	Sr Resin+TRU Resin	Radiochromatogr.: 2 cation columns, mixed bed column	0.69–0.96	
Application of Empore™ strontium Rad disks to the analysis of radiostromium in environmental water samples	L. L. Smith, K. A. Orlandini, J. S. Alvarado, K. M. Hoffmann, D. C. Seely, R. T. Shannon	1996	⁹⁰ Sr, ⁸⁹ Sr	LSC (Sr or Y), propor.	Water	1 L			Empore Sr Rad Disk, Empore Anion Exchange- SR Disk	Depending on salt conc.	
Ion chromatographic separation for analysis of radiostromium in nuclear reprocessing solutions of high ionic strength	J. D. Lamb, F. R. Nordmeyer, P. A. Drake, M. P. Elder, R. W. Miles, R. P. Lash	1989	⁹⁰ Sr	LSC of the collected Sr fraction	Reprocessing solutions	1 uL–500 mL		Acid suppression, cation concentration	IC: cation column	100%	
Measurment of ⁹⁰ Sr in primary coolant of pressurized water reactor	L. Lazare, C. Crestey, C. Bleistein	2009	⁹⁰ Sr, ⁸⁹ Sr	LSC	Coolant	25 mL		Sr pre-concentrated on cation column	Ion chrom: cation (CS12, CG12)	90%	3 Bq/L
Measurement of radionuclides using ion chromatography and on-line radiation detection	R. A. Fjeld, T. A. DeVol, J. D. Leyba, A. Paulenova	2005	Sr, U, Pu, Am	Csl scint. Alpha/beta discr.	Water, waste water		None	None	Ion CHROM.: cation, mixed bed ion exch.	100%	0.4 Bq/L or 200 Bq/kg with off-line counting
Combined procedure for determination of ⁹⁰ Sr, ²⁴¹ Am and Pu radionuclides in soil samples	J. Moreno, N. Vajda, P. R. Danesi, J. J. LaRosa, E. Zeiller, M. Sinojmeri	1997	Pu, Am, Sr	Alpha, LSC	Envir.		HNO ₃ , HF, HCl		Anion-Pu, oxalate, TRU-resin-Am, Sr Resin	42–91%	
Sequential determination of natural (²³² Th, ²³⁸ U) and anthropogenic (¹³⁷ Cs, ⁹⁰ Sr, ²⁴¹ Am, ²³⁹ + ²⁴⁰ Pu) radionuclides in environmental matrix	H. Michel, D. Levent, V. Barci, G. Barci-Funel, C. Hurel	2008	Sr, Cs, Th, U, Pu, Am	Alpha, gamma, LSC	Soil, sed.		(1) leaching: HNO ₃ +HCl (2) acid destr. HF, HNO ₃ (3) microwave		Sr Resin, UTEVA+anion (U, Th), anion (Pu), anion+cation, TRU, TEVA (Am)	50–75	
Sequential isotopic determination of plutonium, thorium, americium, strontium and uranium in environmental and bioassay samples	J.-J. Wang, I.-J. Chen, J.-H. Chiu	2004	Pu, Th, Am, Sr, U	Alpha	Envir, bioassay			-	Anion (8 M HNO ₃)—Th, Pu, Ca oxalate-Sr Resin—Sr, oxalate-TRU- Am, Chelex 100 - U	58–83%	54–66 mBq/sample
The development of emergency radioanalytical techniques for the determination of radiostromium and transuranic radioisotopes in environmental materials	J. P. Martin, K. J. Odell	1998	Pu, Am, Sr	Sr: Cerenkov (⁸⁹ Sr), LSC (⁸⁹ Sr+ ⁹⁰ Sr)Pu, Am: alpha	Biol. (grass, foodstuff, milk)	10 g	MW, leaching with H ₂ O ₂		Anion—Pu, Sr Resin—Sr, TRU Resin—Am	62%	

Table 4 (continued)

Title	Authors	Year	Analyte	Measurement	Sample		Digestion	Pre-concentration	Main separation	Sr yield	Sr DF (carry-over)	Sr LD
					Matrix	Amounts (g, L)						
Routine radiochemical method for the determination of ^{90}Sr , ^{238}Pu , $^{239+240}\text{Pu}$, ^{241}Am , and ^{244}Cm in environmental samples	V. A. Ageyev, O. O. Odintsov, A. D. Sajeniouk	2005	Sr, Pu, Am, Cm	Alpha, beta counting	Soil, sed., aerosols, water, vegetation	Soil:100–200 g	Leaching	Ca oxalate+ $\text{Fe}(\text{OH})_3$ + LaF_3	Anion—Pu, cation/ α HIBA-Am, ppt.-Sr	50–70		
Determination of Pu, Am, U, Th and Sr in marine sediment by extraction chromatography	J. Mellado, M. Llaurado, G. Rauret	2001	Th, U, Am, Pu, Sr	Alpha	Synthetic	2–5	Microwave: HNO_3 , HF, HClO_4		UTEVA, TRU, Sr Resin	> 75%		
The development of a sequential method for the determination of actinides and ^{90}Sr in power station effluent using extraction chromatography	N. Spry, S. Parry, S. Jerome	2000	Pu, Am, Sr	Alpha	NPP effluent		HNO_3 evaporation		TRU–Sr Resin	23–54%		0.9 Bq/L
Measurement of Sr-90 by Cerenkov counting technique	C. Poletiko, L. Lemercier, B. Leiba, P. Llug	1994	^{90}Sr	Cerenkov (propor., Cerenkov, LSC compared)					Chemically processed			About 10 mBq/sample
Determination of $^{89}\text{Sr}/^{90}\text{Sr}$ in food and environmental samples	M. Pimpl	1991	^{90}Sr , ^{89}Sr	Beta-counting $2 \times$ (total Sr, Y)			Leaching:HCl	Sulfate (pH 4.5), EDTA, hydroxide scavage	Chromate, sulfate, Y oxalate	80%		6–9 mBq/sample
The use of Cerenkov radiation for the assay of radiostrontium in aqueous solutions	B. Carmon	1979	^{90}Sr , ^{89}Sr	Prompt Cerenkov counting— ^{89}Sr , Y Cerenkov								About 15 Bq
ASTM Standard Test Method for radiochemical determination of strontium-90 in soil		2007	^{90}Sr	Propor. counter (or LSC)	Soil	5 g	Leaching (HNO_3 +HCl), destruction of filter paper	None	Sr Resin (2 mL)	95%		4 Bq/kg

counted, counting efficiency is determined by an equilibrium ^{90}Sr – ^{90}Y standard. This method is long, but accurate. This is a recommended calculation method according to the ISO 18589-5:2009 standard.

- (c) Y is separated and ^{90}Y measured immediately after its separation from ^{90}Sr that was in secular equilibrium with its daughter at the time of separation. This is a recommended calculation method according to the ISO 18589-5:2009 standard.

If ^{89}Sr and ^{90}Sr are also present in the sample than the activities are determined by different ways depending on the type of the detector and the counter.

Gas ionization detectors and solid scintillators have limited or no energy resolution thus the beta spectra of the radiostrontium isotopes cannot be recorded. Activities can be calculated in the following ways:

- (d) Sr is separated radiochemically, the Sr source is counted at two different times, usually immediately and after a two week-long ingrowth period again. The count rates are expressed as the sum of the count rates of ^{90}Sr , ^{90}Y and ^{89}Sr corrected for ingrowth and decay. This is a recommended calculation method according to the ISO 18589-5:2009 standard.
- (e) Sr is separated radiochemically, the Sr source is counted immediately for a relatively short counting time, and after a two week-long ingrowth period ^{90}Y is chemically separated and counted, the measured count rate is corrected by the decay factor taking into account the time interval between separation of Y and counting the Y source. The 1st measurement provides the sum of the count rates of ^{90}Sr and ^{89}Sr , the 2nd measurement provides the count rates of ^{90}Y . From the latter the activity of ^{90}Sr can be calculated. This method was used, e.g., by Pimpl (1991).
- (f) Sr is separated radiochemically, the Sr source is counted twice after each other, with and without an absorber that absorbs weak beta radiation but does not absorb significantly the hard beta radiation of ^{90}Y . Activities of ^{89}Sr and ^{90}Sr are calculated based on the calibration using the absorber. The method was applied by Gillard-Baruh (1973).

Cerenkov counters are sensitive only to the high-energy beta radiation of ^{89}Sr and ^{90}Y . Activities of the radiostrontium isotopes can be calculated in the following ways:

- (g) Sr is separated radiochemically, the Sr source is counted immediately for a relatively short counting time (^{90}Y ingrowth is neglected) and after a given ingrowth period the Sr source is re-counted again, the 1st measurement provides the count rate of ^{89}Sr , the 2nd measurement provides the sum of the count rates of ^{90}Y (with ingrowth correction) and ^{89}Sr (with decay correction).
- (h) Sr is separated radiochemically, the Sr source is counted immediately for a relatively short counting time, and after a two week-long ingrowth period ^{90}Y is chemically separated and counted, the measured count rate is corrected by the decay factor taking into account the time interval between separation of Y and counting the Y source. The 1st measurement provides the count rates of ^{89}Sr , the 2nd measurement provides the count rate of ^{90}Y . From the latter the activity of ^{90}Sr can be calculated. This measurement and calculation method was applied, e.g., by Carmon (1979) with the modification that the Sr source was counted 4–6 times to follow the ^{90}Y ingrowth and extrapolate the curve to time zero.

Liquid scintillation spectrometry provides information about the shape of the beta spectra that are overlapping in case of ^{90}Sr , ^{90}Y and ^{89}Sr , but because of the big differences in the end-point beta energies the spectra can be well interpreted as a sum of the individual components, especially if other beta emitting nuclides are missing from the sample. Data evaluation can be performed in the following ways:

- (i) The separated Sr source is counted in a full-spectrum count mode by an LS spectrometer, after a certain decay-ingrowth period the measurement is repeated. Results are calculated by mathematical decomposition similar to method d). Benzi et al. (1988) used this method, in emergency situation the 2nd measurement was performed after a short ingrowth time.
- (j) The separated Sr source is counted in two properly chosen energy windows by the LS spectrometer, typically a high-energy pure ^{90}Y window and a low energy mixed $^{90}\text{Sr} + ^{89}\text{Sr} + ^{90}\text{Y}$ window are selected. Results are calculated by mathematical decomposition. Vogl and Gesewsky (1992) used the double window method. Kim et al. (2009) reported a rapid method of ^{89}Sr and ^{90}Sr in milk by combined separation technique using cation exchange resin and Sr Resin and two window counting method. Eikenberg et al. (2006) used the triple window method to calculate the activities of ^{90}Sr (^{90}Y), ^{89}Sr and ^{85}Sr tracer. A window optimization method is described by Rauret et al. (1990).
- (k) The separated Sr source is counted in a full-spectrum count mode by an LS spectrometer. Spectrum deconvolution is performed comparing the measured composite spectrum with the reference spectra of the individual components. Shape factors can also be calculated (Grau Carles et al., 2008). Quench corrections and efficiency calculations can be performed by the CIEMAT/NIST efficiency tracing method. This method was applied, e.g., by Altitoglou (2008) who compared it with the window based method and found that spectra deconvolution is rapid and reliable method of determining radiostrontium even at low activity levels. The spectrum-unfolding method was used by Hong et al. (2001) to measure ^{89}Sr and ^{90}Sr directly, and other full spectrum methods were applied by Heilgeist (2000) and Chobola et al. (2006).
- (l) Cerenkov counting technique can also be combined with liquid scintillation detection thus obtaining a fast calculation method. Sr is separated radiochemically, Cerenkov radiation of the Sr source is counted immediately for a relatively short counting time (^{90}Y ingrowth is neglected) and after addition of a cocktail the sample is re-counted by an LS counter. The 1st measurement provides the count rate of ^{89}Sr , the 2nd measurement provides the sum of the count rates of ^{90}Sr and ^{89}Sr .

8. Conclusions

The radiochemical separation procedures developed and applied for sample decomposition, pre-concentration and selective separation of radiostrontium using precipitation, co-precipitation, ion exchange, solvent extraction and extraction chromatographic techniques have been reviewed. The classical method of radiostrontium determination is based on a series of precipitations including the fuming nitric acid separation of Ca from Sr. Although the method is tedious, time-consuming, uses hazardous chemical, this is still the method of choice if big amounts of samples containing a lot of stable Sr are processed. Indirect methods for ^{90}Sr determination based on the separation of Y by solvent extraction are relatively fast, and simple, but recently have been replaced by the more selective, simple and fast

extraction chromatographic method using Sr Resin that has been successfully used for almost all types of samples.

Nuclear measuring techniques applied for the detection of radiostrontium isotopes have been comparatively evaluated. It was pointed out that gas proportional counter due to the low-cost instrumentation and the high sensitivity is still an adequate technique in the routine determination of ^{90}Sr and ^{89}Sr . Liquid scintillation spectrometry provides information about the energy distribution of the beta radiation, it has proven to be the most reliable analytical technique for the identification of radiostrontium isotopes. Cerenkov counters can also be used for the rapid determination of ^{90}Sr via its ^{90}Y daughter. Radiometric measurement methods are superior to mass spectrometric techniques regarding sensitivity and the low cost instrumentation. At present, only RIMS is comparable with the radiometric methods for the determination of ^{90}Sr . Future developments in mass spectrometric techniques are expected to increase the sensitivity, thus providing ultrasensitive, fast and high through-put analytical techniques for the determination of ^{90}Sr in environmental samples.

Acknowledgement

This work was financially supported under the IAEA sub-programme 'Supporting Quality in the Analysis of Terrestrial Environmental Samples'.

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