Transactions of the Korean Nuclear Society Autumn Meeting
Gyeongju, Korea, October 26-27, 2017

Determination of ^{90}Sr and ^{99}Tc from concrete and soil

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1. Introduction

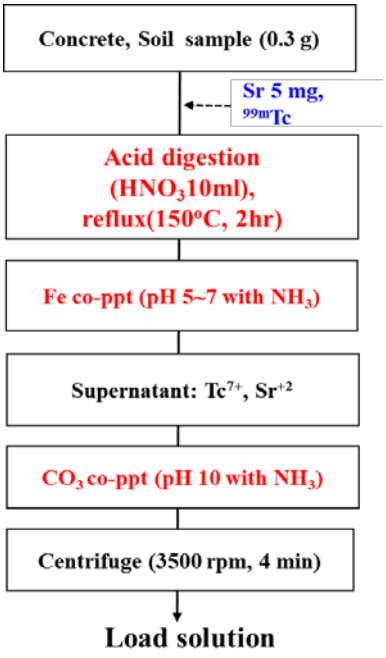
The fate of radioactive waste produced from nuclear facilities depends on its radioactive concentration level. Now in Korea, the low/intermediate level of radioactive waste should be transferred to the radioactive waste repository placed in Gyung-Ju. According to the Nuclear Safety Act, the part operating the nuclear facility have to characterize its radioactive waste, for example ^{3}H, ^{14}C, ^{55}Fe, ^{90}Sr, ^{99}Tc, ^{129}I, gamma isotopes and gross alpha. The fast and reliable analytical technique is a big issue in the characterization of radioactive waste, because the waste handling cost depends on its analytical results. The sequential determination of several radionuclides from one sample could be the economic alternative in processing wastes. Except the volatile radionuclides, such as ^{3}H and ^{14}C and gamma emitters, alpha and beta emitters should be chemically separated from the sample. This study presented the sequential separation of ^{90}Sr and ^{99}Tc from soil and concrete sample based on extraction chromatography.

2. Methods and Results

We digested the concrete and soil samples with nitric acid to extract ^{90}Sr and ^{99}Tc, and chemically separated them from the sample using TEVA resin for ^{99}Tc and Sr resin for ^{90}Sr, respectively. ICP-MS and LSC were used to determine the level of ^{99}Tc and ^{90}Sr. The recovery of each target nuclides was estimated by ^{99m}TC using HPGe and stable Sr using ICP-OES. The method standard sample was used to test of the performance of the suggested method.

2.1 Pretreatment

In considering the minimum detectable activity to meet NSCC notice (2014-3), 0.3 g of concrete and soil samples were transferred into Teflon beaker. About 40 kBq of ^{99m}Tc and 0.5 mg of Sr carrier (10 mg of Sr mL-1) were added with the sample. To evaluate the performance of this method, the method standard samples were prepared with a mixture of 3-5 Bq of ^{90}Sr and ^{99}Tc, respectively. Ten mL of 14 M nitric acid and 0.5 mL of 1 M NaNO2 was mixed with the sample, and it was refluxed at 150oC for 2 hr. Tc existed as Tc^{7+} under oxidant phase. Then it was filtered through GF/F (Whatman), and 1 mL of Fe carrier (10 mg mL^{-1}) was added. Its pH was increased to 7 using NH3 solution. It was centrifuged, and then ^{99}Tc in the supernatant was loaded on TEVA resin. The precipitate having ^{90}Sr was dissolved in 8 M HNO_3, and then loaded on Sr resin.

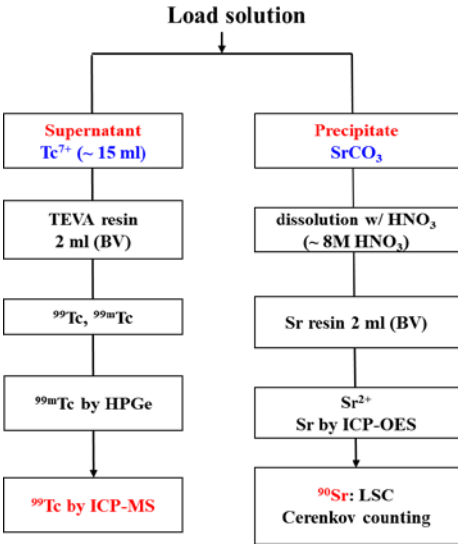


2.2 chemical separation

TEVA resin (2 ml, BV) was conditioned with 10 mL of 0.5 M HNO_3. 10 mL of 0.1 M HCl and 10 mL of 2 M HNO_3 were used to remove interferences from TEVA resin, and 8 mL of 8 M HNO_3 was used to elute ^{99}Tc. Then the recovery of ^{99}Tc was estimated by determining ^{99m}Tc by HPGe.

The precipitate having 90Sr was dissolved in 10 mL of 8 M HNO_3, and loaded on Sr resin conditioned with 10 mL of 8 M HNO_3. Additional 10 mL of 8 M HNO_3 was used to remove interferences, and then 10 mL of de-ionized water was used to elute ^{90}Sr from Sr resin. ^{90}Sr of the sample eluted from Sr resin was precipitated as $SrCO_3$, then dissolved with 10 mL of 0.1 M HNO_3.

The recovery of Sr was determined by ICP-OES to estimate the recovery of Sr.



2.3 Determination of ^{90}Sr and ^{99}Tc

The sample eluted from TEVA resin was diluted to desirable concentration for determining ^{99}Tc by ICP-MS. ^{90}Sr of the sample was measured with Cerenkov mode by LSC.

Table 1 Analytical Results of method standard for ^{90}Sr

Sample	known A[1] (Bq)	Recovery (%)	meas. A[2] (Bq)	R.E.[3] (%)
STD-1	3.10	78.3	3.07	-1.0
STD-2	5.37	46.7	5.62	4.7
STD-3	5.32	61.5	5.16	-3.0
STD-4	3.21	87.6	3.12	-2.8
STD-5	3.23	82.9	3.19	-1.2
STD-6	3.18	79.1	3.21	0.9
STD-7	3.18	84.8	2.98	-6.3
STD-8	3.16	84.4	2.93	-7.3

[1]: known activity, [2]: measured activity, [3]: relative error

Table 2 Analytical Results of method standard for ^{99}Tc

Sample	known A (Bq)	Recovery (%)	means. A (Bq)	R.E. (%)
STD-1	4.01	80.5	4.09	2.0
STD-2	4.05	91.5	4.28	5.7
STD-3	3.86	94.0	3.63	-6.0
STD-4	4.00	92.6	3.64	-8.9
STD-5	2.87	93.7	2.82	-1.9
STD-6	2.90	92.6	2.66	-8.4
STD-7	2.95	92.2	2.87	-2.7
STD-8	2.93	93.6	2.71	-7.3

For ^{90}Sr, the recovery of it was ranged from 60 to 88 %, except STD-2 with 46 %, which lost part of $SrCO_3$ at precipitation process. For ^{99}Tc, the recovery of it was over 90 %, except STD-1 with 80 %.

In regards to the performance test of the analytical method, the measured value agrees with known activity, well. These method employed for determining ^{90}Sr and ^{99}Tc in real soil and concrete samples (n=31) produced in decommissioning of the research reactor. The results show that the level of ^{90}Sr and ^{99}Tc was less than the minimum detectable activity.

3. Conclusions

We collected soil and concrete samples produced from decommissioning of research reactor in Seoul. We analyzed the level of ^{90}Sr and ^{99}Tc in soil and concrete samples. The acid digestion was employed for the pretreatment and TEVA resin/Sr resin was used for chemical separation. The recovery of each radionuclide was estimated by determining stable Sr by ICP-OES and ^{99m}Tc by HPGe. ^{99}Tc and ^{90}Sr were determined by ICP-MS and LSC, respectively. Based on the performance test using the method standard sample, this method shows the reliable result, having from -8 to 2 % of relative error. The level of ^{99}Tc and ^{90}Sr in real soil and concrete samples were less than MDA.

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