

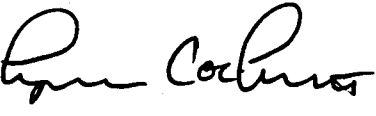

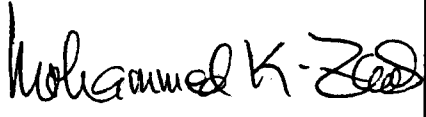



RESL TECHNICAL PROCEDURE

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CHEM-TP-SP.2

SAMPLE RECONSTITUTION

Preparer: 	Date 5-10-99	Independent Review: 	Date 5/10/99
ES&H Review: 	Date 5/17/99	Radiological Controls Review: 	Date 5/11/99
QA Review: 	Date 5/17/99	Supervisor Approval: 	Date 5-11-99

RESL TECHNICAL PROCEDURE

TITLE: CHEM-TP- SP.2, SAMPLE RECONSTITUTION

PURPOSE

The purpose of this procedure is to provide instructions for the recovery of nuclides from sample containers.

APPLICABILITY

This procedure is applicable to the recovery and dissolution of the insoluble fractions present in liquid samples from nuclear power facilities and to which acid and paper pulp were added at the time of collection. This procedure normally is not used with environmental water samples.

RESPONSIBILITIES

RESL staff responsible for implementing this procedure are:

Radiochemist(s)

DEFINITIONS

None.

PROCEDURE

- 1 **ABSTRACT** The samples, as received, contain paper pulp, and are 2% in concentrated hydrochloric acid or nitric acid. Samples containing hydrochloric acid also might contain a holding reductant if nuclides of iodine are of interest. The paper pulp and other particulate materials are filtered, wet ashed in a Teflon beaker with nitric and perchloric acids, and the solution is evaporated to fumes of perchloric acid. The hydrochloric and hydrofluoric acid wash of the container is added to this perchloric acid fraction and the solution is evaporated until a milliliter of perchloric acid remains. The perchloric acid solution is mixed with the original filtrate and transferred back into the original container, thus providing a stable, homogeneous sample.

- 2 **LIMITATIONS AND INTERFERENCES** Volatile elements present in the filterable fraction or container wash can be lost (Reference).

- 3 **SAFETY PRECAUTIONS**
 - 3.1 Follow laboratory safety addressed in RESL-TP-IH.2 and RESL-TP-IH.4. Also review any applicable Radiation Work Permit prior to beginning this procedure.

 - 3.2 Wear proper eye protection (RESL-TP-IH.1).

 - 3.3 Wear the appropriate protective gloves while performing work associated with this TP to prevent contamination and acid burns to the hands (RESL-TP-IH.1).

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- 3.4 Refer to RESL-TP-IH.6 when working with perchloric acid which is a powerful oxidizing agent.
- 3.5 Wear nitrile or natural rubber gloves when handling hydrofluoric acid. Concentrated hydrofluoric acid can be absorbed through the skin. If you suspect contact with your skin, wash immediately with large amounts of water for 1 min, gently rub Hydrofluoric Acid Burn Gel on the affected area, and contact your supervisor immediately. Concentrated HF (28.9 N) shall not be used outside a fume hood (RESL-TP-IH.9).
- 3.6 Perform a personal frisk (hands, front of lab coat) after performance of this procedure to verify that there is no contamination.

4 EQUIPMENT

- 4.1 Filter holder: 47 mm, glass
- 4.2 Suction flask: 1 L, glass
- 4.3 Filter paper: 47-mm filter, resistant to 2% HCl and HNO₃, pore size 0.45 Nm, easily wet ashed
- 4.4 Shaking machine
- 4.5 Hotplate, 3600-W, 46 x 61 cm
- 4.6 Fiberglass mat, 1.6 mm thick, to cover hotplate.

5 SAMPLE INSPECTION

- 5.1 Examine the sample container and/or Sample Record Sheet submitted with the sample for written statements concerning the addition of paper pulp, HCl plus reductant (NaHSO₃ or Na₂S₂O₅), HCl, or HNO₃.
- 5.2 Check the container visually for the presence of paper pulp.
- 5.3 Check the pH of the sample with pH paper; the pH should be .1. Add the appropriate acid (HCl or HNO₃), if necessary, to make the pH .1. Record on the container and in the Sample Reconstitution Logbook the volume of acid added to lower the pH. Shake the sample vigorously and allow it to set overnight before proceeding.
- 5.4 If NaHSO₃ or Na₂S₂O₅ was supposed to be present and/or a gamma scan was requested, check the sample for the odor of SO₂.
- 5.5 Record any discrepancies observed in the Sample Reconstitution Logbook.

NOTE: THE ABSENCE OF PAPER PULP OR A pH > 1 COULD PREVENT COMPLETE

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RECOVERY OF NUCLIDES SORBED ONTO THE WALL OF THE CONTAINER. THE ABSENCE OF SO₂ COULD INDICATE A LOSS OF, e.g., ¹³¹I, WHICH WOULD MAKE SUSPECT A GAMMA SPECTROMETRIC RESULT, IF ANY, FOR THAT NUCLIDE.

- 5.6 Observe the level of the gross gamma activity written on the sample container. Select a separate filtering apparatus and Teflon beaker designated for use with the activity levels listed below:
- 5.6.1 Low level $>0 \leq 10^4$ cpm/sample
- 5.6.2 Medium level $>10^4 \leq 10^6$ cpm/sample
- 5.6.3 High level $>10^6$ cpm/sample

6 SAMPLE PREPARATION

- 6.1 Filter the sample by suction through a 47-mm filter with a pore size of 0.45 μ m (the filter should be acid-resistant but easily wet ashed). Replace the filter with a new one whenever plugging occurs and place each filter used in the 250-mL Teflon beaker.
- 6.2 Rinse the container with about 20 mL of filtrate and pour the rinse through the final filter.
- 6.3 Add 50 mL of 16 M HNO₃ and 10 mL of concentrated (11.7 M) HClO₄ to the filter(s) and insoluble material in the 250-mL Teflon beaker.
- 6.4 Heat the sample on a covered hotplate to the first fumes of perchloric acid.

NOTE: THE SAMPLE SHOULD BE GIVEN CONSTANT ATTENTION AT THIS POINT TO AVOID SPATTERING OR POSSIBLE FIRING. IF THE SOLUTION TURNS BLACK, COOL SLIGHTLY, ADD A FEW mL OF 16 M HNO₃, AND CONTINUE HEATING TO FUMES OF PERCHLORIC ACID. REPEAT THE HNO₃ ADDITIONS UNTIL THE FUMING PERCHLORIC CEASES TURNING BLACK.

- 6.5 Add 40 mL of distilled or demineralized H₂O, 20 mL 12 M HCl, and 50 mL 28.9 M HF to the original sample container. Place the container in a boiling water bath for 1 min. Place the container in a plastic bag and shake the sample on a mechanical shaker for 15 min. Transfer the wash solution to the Teflon beaker containing the perchloric acid from Step 6.4. Use a few mL of distilled or demineralized H₂O to rinse the container and add the rinse to the Teflon beaker.

NOTE: THIS TREATMENT IS APPLICABLE TO MOST PLASTIC BOTTLES. GLASS BOTTLES ARE WASHED WITH 100 mL OF 1% HF.

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- 6.6 Set the beaker on the hottest portion of the covered hotplate and evaporate the solution to fumes of perchloric acid. Add 5 mL of concentrated HNO₃, cover the beaker with a Teflon watch glass, and heat gently on a cooler portion of the hotplate for 10 min. The refluxing action should wash down the walls of the beaker and can be repeated if carbonaceous residue still remains on the walls. Uncover the beaker and evaporate the solution to about 1 mL of perchloric acid in the beaker but do not allow the sample to go to dryness.
- 6.7 Add some of the original filtrate to the beaker and transfer this and the remaining filtrate to the original, washed container. Write the pH of the sample, reconstitution date, and "RECONSTITUTED" on the container and in the Sample Reconstitution Logbook.

REFERENCES

R. P. Bernabee, D. R. Percival, D. B. Martin, "Fractionation of Radionuclides in Liquid Samples from Nuclear Power Facilities." *Health Physics*, 39, 1980, pp. 57-67.

RESL-TP-IH 1, 2, 4, 6, 9.

QUALITY RECORDS

Entry in RESL database.
Sample Reconstitution Logbook.