

UNITED STATES TRANSURANIUM AND URANIUM REGISTRIES  
ANALYTICAL PROCEDURE MANUAL

**USTUR 820: Radioactive Liquid Waste Disposal**

<b>Purpose</b>	Prepare radioactive liquid waste for disposal via the Radiation Safety Office	<b>Method Number</b>	USTUR 820
<b>Original Date</b>	6/10/03	<b>Author</b>	USTUR Dorothy Stuit
<b>Revision Number</b>	2	<b>Approved By</b>	Jim Elliston
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**1. Principle of Method**

- 1.1. Using liquid scintillation counting the activity of the radioactive liquid waste is determined. The radioactive liquid waste pH is adjusted to be in the range of 5-8 using NaOH.

**2. Apparatus**

- 2.1. Packard Tri-Carb 1900 CA Liquid Scintillation Analyzer.
- 2.2. 4.25 cm filter paper (Whatman No. 1).
- 2.3. Squeeze-bulb siphon
- 2.4. Radioactive waste log notebook
- 2.5. 20 mL glass scintillation vials

**3. Reagents**

- 3.1. OPTI-FLUOR liquid scintillation cocktail (Packard).
- 3.2. Concentrated HNO<sub>3</sub>
- 3.3. 1M HNO<sub>3</sub>
- 3.4. NaOH pellets

**4. Radioactive Liquid Waste Activity Determination**

- 4.1. The radioactive liquid waste container should be no more than half full. If necessary, transfer enough of the radioactive acid waste such that the container for disposal is no more than half full using a squeeze-bulb siphon. If a container has only electrodeposition waste, it can be approximately 2/3 full.
- 4.2. List each radioactive waste container in the Radioactive Waste Log notebook (NRC 114). Mark each container with the next available number from the Radioactive Waste Log notebook. (Remove all previous labeling from the radioactive liquid waste containers). Note the approximate volume of the radioactive liquid waste in each container.

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- 4.3. Take a 2.0 mL aliquot from each container, using a pipet, and place the aliquot in a clean 20 mL glass scintillation vial. Mark each vial with the appropriate container number.
- 4.4. Add 2-3 mL of concentrated HNO<sub>3</sub> to each vial and bring to dryness on a hot plate at 140°C -150°C.
- 4.5. The residue in the vial should be a white color. If not, add an additional 2-3 mL of concentrated HNO<sub>3</sub> and warm on a hot plate at 120°C. Add dropwise, 30% H<sub>2</sub>O<sub>2</sub>, to oxidize any color-causing compounds. Bring the aliquot to dryness on a hot plate at 140°C -150°C. Repeat this step as necessary until the residue is white in color.
- 4.6. Add 2 mL of 1M HNO<sub>3</sub> to each vial and place a cap loosely on top of each vial. Warm the vials on a hotplate at 120°C to completely dissolve the residue.
- 4.7. Cool the vial to room temperature, then add 18 mL of Opti-Fluor scintillation cocktail.
- 4.8. Cap the vial tightly and shake vigorously. Mark the top of the vial cap with the appropriate container number.
- 4.9. Remove any markings and/or fingerprints from the external surface of the glass scintillation vial using 70% ethanol.
- 4.10. Prepare a blank solution by adding 2 mL of 1M HNO<sub>3</sub> and 18 mL of Opti-Fluor scintillation cocktail into a 20 mL glass scintillation vial. Mark the cap of the blank vial with a "B".
- 4.11. Load the glass scintillation vials into a cassette or rack for the Tri-Carb 1900 CA Liquid Scintillation Analyzer, make sure that the "blank" is in the first position.
- 4.12. Allow the samples to "dark adapt" for at least 1 hour in the analyzer and then count the samples using protocol "25". Start the counting cycle near the end of the day to prevent tying up the instrument during normal working hours. The Regions with channel numbers are shown in Table 1.

**Table 1: Liquid Scintillation Counter Setup**

	<b>Region A (Beta Region)</b>	<b>Region B (Alpha Region)</b>	<b>Region C (Total)</b>
Channel Numbers	2-50	51-500	0-2000

- 4.14. Using the Microsoft Excel program "USTUR 820 Radwaste Calc" (on a disk in the inside flap of the Waste Receipt looseleaf binder in NRC 114) and the printout from the Tri-Carb 1900 CA Liquid Scintillation Analyzer, calculate the activity of the original volume of radioactive liquid waste in the container. Record the

activity in the Radioactive Waste Log notebook and file the liquid scintillation printout in the Waste Receipt looseleaf binder.

## 5. Neutralization of Radioactive Liquid Waste

- 5.1. Radioactive liquid waste must be in the pH range of 5-8 to be accepted by the Radiation Safety Office for disposal.
- 5.2. The majority of the USTUR liquid waste is highly acidic, except for electrodeposition waste. When adding sodium hydroxide (NaOH) or any other base, additions need to be done slowly to prevent major spattering or foaming.
- 5.3. Prepare a slurry of approximately 50% NaOH and 50% water in a 1 L plastic beaker.  
**Note:** The mixture will be very hot! Wear acid/base protective clothing (lab coat, acid/base apron, acid/base gloves, goggles, face shield, etc.).
- 5.4. Very slowly add the NaOH solution to a waste container with constant stirring.  
**Note:** The reaction can be quite violent, so keep the fumehood sash as low as possible.
- 5.5. Continue adding the NaOH solution until the pH is in the range of 5-8. When the vigorous reactions begin to subside with additions of NaOH solution, begin checking the pH of the container solution with wide range pH paper (1-14). If the pH rises above 8 add non-radioactive acid waste until the pH is in the acceptable range (5-8).
- 5.6. When the pH is in the acceptable range allow the solution to cool over night and verify that the pH is still in the acceptable range. If the pH is not within the acceptable range (5-8) repeat steps 5.4-5.6 as necessary.
- 5.7. Once the solution is cool and the pH is stable in the acceptable range cap the container tightly and place tape over the cap. Record the pH of the solution on the tape.
- 5.8. Obtain a Radioactive Waste Receipt (form RSO-08-01) from the Radiation Safety Office. Fill out the form completely and call the Radioactive Waste Pick-Up recording at 335-7383.

## 6. References

- 6.1. ANSI N13.30
- 6.2. L.A. Currie, "Limits for Qualitative Detection and Quantitative Determination," *Anal. Chem.* 40, 586-593 (1968).
- 6.3. L.A. Currie, "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements," U.S. Nuclear Regulatory Commission report NUREG/CR-4007 (September 1984).