N2-CSP-14

SOLID RADWASTE CHEMICAL SURVEILLANCE

AT UNIT 2

1.0 PURPOSE

To provide directions for the Chemical Surveillance of solid radioactive waste at Unit 2.

NOTE: This procedure is not applicable for the Surveillance of Class B and C waste for product stability.

2.0 REFERENCES

2.1 Nine Mile Point Nuclear Station - Unit 2 Technical Specifications

Section 3/4.11.3 - Solid Radioactive Waste

2.2 Nine Mile Point Nuclear Station - Unit 2 Final Safety Analysis Report:

Question F460.19

2.3 Niagara Mohawk Procedures, NMPNS

N2-CRP-4 Isotopic Analysis

2.4 Installation, Operation & Maintenance Manual, WPC VRS. Volume 2, Section 10 & 2 Auxiliary Steam System.

2.5 ASTM D312-78 Standard Specification for Asphalt Used in Roofing.

2.6 Proposed Process Control Program, Consumers Power Company.

3.0 PREREQUISITES AND PRECAUTIONS

3.1 Observe all standard radiation protection rules while handling radioactive materials.

3.2 All persons performing this procedure should be trained on this procedure.

3.3 Radwaste Auxiliary Boiler piping is hot. Use caution when working near system.

4.0 LIMITATIONS AND ACTIONS

4.1 Evaporator

4.1.1 Limits:

a.	pH at 25°C	8.0 - 10.0
b.	Specific Gravity at 25°C	As specified by supervision
c.	Fluid Behavior at	<25°C

4.1.2 Actions:

a. Adjust pH to within range by addition of acid or caustic.

b. Notify Radwaste operations to pump evaporator to bottoms tank when specific gravity is high.

c. Notify supervision if non-fluid behavior is observed.

4.2 Radwaste Boiler

4.2.1 Limits:

a. Boiler Water

Conductivity at 25°C	<4,500 μmho/cm
pH at 25°C	7.5 - 10.5
Total suspended solids	<10ppm

b. Feedwater

Conductivity at 25°C	<20 μmho/cm
pH at 25°C	6.0 - 9.0

4.2.2 Actions

a. Boiler Water

Blowdown to control conductivity and total suspended solids.

Feed sodium hydroxide or phosphoric acid to control pH.

b. Feedwater

Notify supervision if parameters are out of specification.

4.3 Extruder Evaporator

4.3.1 Limits:

Incoming asphalt shipment must have a softening point between 85-96°C (185-205°F).

Radwaste Feed must have a pH at 25°C between 6.0 - 10.0, Oil Grease <1% and demonstrate solidification with asphalt.

The product of solidified radwaste must demonstrate a compressive strength greater than 50 psi.

4.3.2 Actions:

Notify supervision if incoming asphalt shipment is out of specification.

Notify radwaste operations and supervision if extruder feed is out of specification (feed acid or caustic to correct pH).

Notify supervision if a product of solidified radwaste is out of specification. The batch may not be shipped offsite without further study.

5.0 PROCEDURE

5.1 AS REQUESTED, (at least weekly) sample the radwaste auxiliary boiler, take corrective action and complete figure 1-V.

5.1.1 Blowdown the solid radwaste auxiliary boiler by opening 2WSS-V161 and 162 one at a time for 1 minute each.

5.1.2 Obtain a sample of feedwater from 2WSS-V172.

CAUTION: Sample may be hot. Wear heavy gloves.

5.1.3 Obtain a sample of the boiler water from 2WSS-V(LTR)

NOTE: Place ice in cooling coil bucket and open valve slowly to avoid blowing steam.

5.1.4 Analyze each sample for pH, total suspended solids, gross activity, conductivity and chemical requirements. See appendix A.

5.1.5 If conductivity or total suspended solids are over limit, repeat blowdown and sampling. Notify supervision is activity is detected.

5.1.6 Measure out the amount of acid or caustic and 50 gm of sodium sulfite (Na_2SO_3).

5.1.7 Use 2WSS-V178 to drain the chemical feed tank.

5.1.8 Use 2WSS-V184 to add water to the chemical feed tank up to the mark.

5.1.9 Add the chemicals and use 2WSS-M1X49 to aid in dissolution.

5.1.10 Notify Radwaste operators to pump chemicals.

5.2 AS REQUESTED sample the evaporator and complete figure 2-V.

5.2.1 Contact radiation protection for a RWP.

NOTE: Radiation Protection should survey and make protective action recommendations for sampling, handling and transportation unless radiation levels are within self monitoring limits.

5.2.2 Inspect sample valve (AOV 201 or 202) to ensure that there is no water in it.

5.2.3 Open the sample loop isolation valves (AOV 161 & 162 or AOV 163 and 164).

5.2.4 Position a sample bottle under the sample spigot.

5.2.5 Operate the sample valve (AOV 201 or 202) to obtain approximately 30 ml of sample. Do not use dilution water.

NOTE: If only a pH measurement is needed, collect just 10 ml.

5.2.6 Remove sample bottle, cap and rinse outside with water from water valve (V126 or 128).

5.2.7 Close sample supply valve (AOV 162 or 164).

5.2.8 Open flush water valve (V127 or 130) to flush sample loop.

5.2.9 Close sample return valve (AOV 161 or 163).

5.2.10 Operate sample valve (AOV 201 or 202) to drain sample recirc loop of water.

5.2.11 Close flush water valve (V127 or 130).

5.2.12 Rinse sample valve with water (V118 or 125).

5.2.13 Rinse inside of sample cubicle from water valve (126 or 128).

5.2.14 Transport sample to lab.

5.2.15 Cool sample to 25° (22.5 - 27.5).

5.2.16 Pour sample into a 25ml graduated cylinder. Record weight (A).

5.2.17 Verify that sample has not solidified or thickened excessively.

5.2.18 Record sample volume. (B).

5.2.19 Weigh graduate with sample (C).

5.2.20 Calculate specific gravity.

5.2.21 Specific Gravity = (C - A)/B

Where: A = Weight of graduate (gm)
B = Volume of sample (ml)
C = Weight of graduate and sample (gm)

5.2.22 Measure the pH of sample.

5.2.23 Review completed sheet with respect to limits and actions. Sign and submit to supervision for review. Distribute copies as requested and store orginal in log book.

NOTE: If pH adjustment is necessary, notify operators and ensure that not too much acid/caustic is pumped. Resample approximately 5 min. after addition.

5.3 AS REQUESTED, sample the radwaste feed to the extruder. Complete figure 3-V.

5.3.1 Ensure that the tank (waste sludge or evaporator bottoms) has been isolated and recirculated for at least 1 hour by conversing with radwaste operators.

NOTE: Also obtain radwaste form and batch number from radwaste operators. Record.

5.3.2 Contact radiation protection for a RWP.

NOTE: Radiation protection should survey and make protective action recommendations for sampling, transport and handling unless radiation levels are within self monitoring limits.

5.3.3 Inspect sample valve (AOV 352 or 356) to ensure that there is no water in it.

5.3.4 Open the sample recirc loop valves (AOV 351 or 354) for at least 2 minutes.

5.3.5 Position a sample bottle under the sample spigot.

5.3.6 Close the recirc loop valve (AOV 351 or 354).

5.3.7 Operate the sample valve (AOV 352 or 356) until approximately 80ml have been collected. Do not use dilution water.

NOTE: It may become necessary to refill the recirc loop. Close sample valve and open recirc loop valve. Then repeat last two steps.

5.3.8 Remove sample bottle and cap.

5.3.9 Wash down outside of bottle and inside of cubicle by opening water valve (AOV 353 or 358).

5.3.10 Operate sample valve (AOV 352 or 356) to flush internals.

5.3.11 Close water valve and leave sample valve open.

5.3.12 Transport sample to lab.

5.3.13 Cool sample to 25°C (22.5 - 27.5) in a water bath.

5.3.14 Place 10.0 ml of sample into a centrifuge tube. Spin for 10 minutes and visually exam. Report any visible oil and grease as greater than 1%.

5.3.15 Measure the pH of the liquid portion of sample from the centrifuge operation.

5.3.16 Determine the specific gravity, solids & water fraction and the waste and asphalt feed rates in accordance with Appendix B.

5.3.17 Use the planchet from solids determination to verify solidification in accordance with Appendix I.

5.3.18 If solidification is verified, notify radwaste operators of proper waste and asphalt feed rates.

5.3.19 Determine the Alpha content of the sample in accordance with Appendix C.

5.3.20 Isotopically analyze a portion of the sample in accordance with N2-CRP-4. Attach printout.

5.3.21 Save the remaining sample (∿ 50 ml) in a shielded area. Label sample appropriately and ensure that storage area is properly posted for radiation protection.

5.3.22 Calculate the activity of nuclides not directly measured by using the appropriate scaling factors supplied by supervision.

5.3.23 Review completed sheet with respect to limits and actions. Sign and submit to supervision for review. Distribute copies as requested and store original in log book.

5.4 MONTHLY ACTIONS

5.4.1 Service the Great Lakes pH monitors in the solid radwaste system in accordance with appendix F.

5.4.2 Obtain a specific RWP and mark-up for the job and complete figure F.1-M. Notify radwaste operations.

5.4.3 Submit completed form to supervision for review and store original in log book.

5.5 QUARTERLY ACTIONS

5.5.1 Complete Figure 4-Q for each waste form.

NOTE: Frequency may vary according to supervisory direction.

5.5.2 Contact radiation protection for a suitable RWP.

5.5.3 Gather the samples from each batch of each type of waste fed to the extruder.

5.5.4 Transfer an equal volume of each into a suitable container.

NOTE: If the batch volume represented by a particular sample is significantly different from the norm, transfer a proportionally different amount.

5.5.5 Mix the composite thoroughly.

5.5.6 Ship approximately 5 μCi of the composite to a vendor specified by supervision for 10CFR61 analysis, including tritium.

5.5.7 Retain remaining composite for gross alpha and an isotopic analysis on date directed by supervision. Attach printout.

5.5.8 Upon receipt of vendors results, determine new scaling factors in accordance with Appendix D and attach figure D.1-V.

5.5.9 Attach vendors results and submit completed form to supervision for review and signature. Store original form in log book.

5.6 AS REQUESTED

5.6.1 Sample an incoming shipment of asphalt in accordance with Appendix G and check the softening point in accordance with Appendix H.

5.6.2 Complete Figure H.1-V.

5.6.3 Verify that the softening point temperature is between 85 and 96°C.

5.6.4 Sign completed form and submit to supervision for signature. Store original in log book.

5.7 AS REQUESTED

5.7.1 Sample the solidified radwaste/asphalt product from the extruder in accordance with Appendix J and check the compressive strength.

5.7.2 Complete figure J.1-V.

5.7.3 Verify that the compressive strength is greater than 50 psi.

5.7.4 Sign completed form and submit to supervision for signature. Store original in Log Book.

5.8 RADWASTE/ASPHALT SHIPMENT

5.8.1 Calculate the isotopic activity of a radwaste/asphalt shipment in accordance with Appendix K and classify the waste in accordance with RP-6.

5.8.2 Complete Figure K.1-V.

5.8.3 Complete Attachment 2 and 3 of RP-6 as appropriate.

5.8.4 Sign completed forms and submit to supervision for signature. Distribute copies as requested and store original in Log Book.

5.9 AS REQUESTED, sample resins to be processed by cask dewatering.

Complete Figure 5-V.

5.9.1 Collect sample following section 5.3.1 to 5.3.13 but collect 150 ml.

5.9.2 Place sample in a ml graduate cylinder and allow resins to settle.

5.9.3 Decant water until no free liquid remains. (Invert cylinder using a screen to hold back the resins).

5.9.4 Isotopically analyze a portion in accordance wth N2-CRP-4.

5.9.5 Determine solid and water content in accordance with Appendix B.

5.9.6 Determine the Alpha content of the sample in accordance with Appendix C.

5.9.7 Save the remaining portion of the sample in a shielded area. Label appropriately and ensure that the storage area is properly posted for radiation protection.

5.9.8 Calculate the activity of nuclides not directly measured by using the appropriate scaling factors.

5.9.10 Calculate the isotopic activity of the dewatered resin shipment in accordance with Appendix L and classify the waste in accordance with RP-G.

5.9.10.1 Attach completed Figure L.1-V

5.9.10.2 Attach completed Attachment 2 and/or 3 of RP-6 as appropriate.

5.9.11 Sign completed forms and submit to supervision for signature. Distribute copies as requested and store original in Log Book

FIGURE 1-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

RADWASTE AUX. BOILER CHEMISTRY SURVEILLANCE

Date: ______________________

Boiler Blowndown?: ______________________

Feedwater: pH: ______________________ (6.0 - 9.0)

TSS: ______________________ ppm

Conductivity: ______________________ μ mho/cm (20)

Boilerwater: pH: ______________________ (7.5 - 10.5)

TSS: ______________________ ppm (10)

Conductivity: ______________________ μ mho/cm (4,500)

Gross Activity: ______________________ cpm/ml

NaOH needed: ______________________ gm.

85% H_3PO_4: ______________________ ml.

Radwaste Operators Notified To Pump?: ______________________

Technician: ______________________ Supervisor: ______________________

FIGURE 2-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

EVAPORATOR SAMPLE

Date:______________________ Evaporator:____________________

Cylinder Weight: ______________________ gm
Fluid Behavior?: ______________________ at 25°C
______________________ at:____°C

Sample Volume: ____________________ ml
Sample & Cylinder Weight: ____________________ gm
Sample Specific Gravity: ____________________

Sample pH: ______________________ (8.0 - 10.0)

pH Adjustment Necessary?: ____________________

pH After Chemical Addition: ____________________

Comments:

Technician:____________________ Supervisor:________________________

NINE MILE POINT NUCLEAR STATION - UNIT 2

EXTRUDER FEED SAMPLE

Date: ______________________ BATCH #: ____________________

Waste Form: ________________________ Volume: ____________________

Isolation & Recirculation 1/2 hour?: ______________________

Oil and Grease: __________________% (< 1.0)
pH: __________________ (6 -10)

Planchet Weight: ______________ gm (A)
Planchet and Sample Weight: ______________ gm (B)
Bone Dry Weight: ______________ gm.(C)

Specific Gravity: ______________ (B-A)/5
Solids Fraction: ______________ (C-A)/(B-A)
Water Fraction ______________ 1 - Solid Fraction

Maximum Waste Feed Rate:
Sludge: ______________ gpm (0.352/FW.SG)
Bottoms: ______________ gpm (0.528/FW.SG)

Minimum Asphalt Rate: ______________ gpm (0.528.FS/FW)

Solidification Verified: ______________

Tenth Batch: ______________

COMMENTS:

Technician: ______________________ Supervisor: ____________________________

NINE MILE POINT NUCLEAR STATION - UNIT 2

EXTRUDER FEED SAMPLE

Date: ______________________ BATCH #: ______________________

Crucible Weight: ____________________ gm(F)
Crucible & Sample Weight: _______________ gm (G)
Sample Specific Gravity: ________________ gm/ml.(SG)

ALPHA COUNTING:

PC-5 used: _____________ Calib. Date: ______________________
Efficiency: ______________________ cpm/dpm (E)

Label(L)	Alpha (cpm)	Net(dpm/gm) = [L(A-B)/(G-F)E]
20	____________ (A)	____________
50	____________ (A)	____________
100	____________ (A)	____________
200	____________ (A)	____________
500	____________ (A)	____________
1000	____________ (A)	____________
Blank	____________ (B)	

Alpha at infinite dilution: ______________________ dpm/gm (C)

Alpha Activity: __________________ nCi/ml (C SG/2200)

Technician: ______________________________ Supervisor: ______________________

NINE MILE POINT NUCLEAR STATION - UNIT 2

EXTRUDER FEED SAMPLE

Date:________________ Batch:____________________

GAMMA EMMITING NUCLIDES

Nuclide	Activity (μ Ci/ml)	Nuclide	Activity (μ Ci/ml)
Mn-54			
Co-58			
Co-60			
Cs-134			
Cs-137			
		Total	

WEAK GAMMA EMMITERS

Nuclide	Scale	Scale(μ Ci/ml)	Scaler	Activity(μ Ci/ml)
Ni-63	(Co-60)			
Sr-90	(Cs-137)			
Tc-99	(Cs-137)			
I-129	(Cs-137)			
C-14	(Cs-137)			

ALPHA EMMITERS: ALPHA ACTIVITY:____________(nCi/ml)

Nuclide	Scaler	Activity (nCi/ml)
Pu-238		
Pu-239		
Pu-241		
Am-241		
Am-243		
Am-244		
Cm-242		
TRU		

Technician:____________________ Supervisor:____________________

FIGURE 4-Q

NINE MILE POINT NUCLEAR STATION - UNIT 2

10CFR61 Scaling Factors

Date:____________________ Waste Form:____________________________

GROSS ALPHA ANALYSIS DATE:____________________________

Crucible Weight: ______________________gm(F)
Crucible & Sample Weight: __________________gm(G)
Sample Specific Gravity: __________________gm/ml(SG)

PC-5 used: _______________ Calib. Date:_____________________

Efficiency:___________________cpm/dpm(E)

LABEL(L)	Alpha (cpm)		Net (dpm/gm) = [L(A-B)/(G-F)E]
20	______________	(A)	______________
50	______________	(A)	______________
100	______________	(A)	______________
200	______________	(A)	______________
500	______________	(A)	______________
1000	______________	(A)	______________
Blank	______________	(B)	

Alpha at infinite dilution: ______________dpm/gm (C)

Alpha Activity:______________________nCi/ml (C SG/2200)

COMMENTS:

Technician:_______________________ Supervisor:____________________________

FIGURE 5-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

DEWATERED RESIN SAMPLE

DATE:______________________ BATCH #:______________________

Isolated and Recirculation 1/2 hour?: ______________________

Sample Volume Collected:	______________	ml.
Planchet Weight:	______________	gm (A)
Planchet & Sample Weight:	______________	gm (B)
Bone Dry Weight:	______________	gm (C)
Specific Gravity:	______________	(B-A)/5
Solids Fraction:	______________	(C-A)/(B-A)
Water Fraction:	______________	1-Solid Fraction
Crucible Weight:	______________	gm (F)
Crucible & Sample Weight:	______________	gm (G)
Sample Specific Gravity:	______________	gm/ml (SG)

COMMENTS:

TECHNICIAN:______________________ SUPERVISOR:______________________

FIGURE 5-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

DEWATERED RESIN SAMPLE

ALPHA COUNTING

PC-5 used: ________________ Calib. Date: ________________

Efficency: ________________ cpm/dpm(E)

Label (L)	Alpha (cpm)		Net (dpm/gm)
20	________	(A)	________
50	________	(A)	________
100	________	(A)	________
200	________	(A)	________
500	________	(A)	________
1000	________	(A)	________
Blank	________	(B)	

Net (dpm/gm) = [L(A-B)/(G-F)E]

Alpha at infinite dilution: ________________ dpm/gm(C)

Alpha Activity: ________________ nCi/ml (CSG/2200)

Technician: ________________ Supervisor: ________________

FIGURE 5-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

DEWATERED RESIN SAMPLE

Date:________________________ Batch:____________________________

GAMMA EMMITING NUCLIDES

Nuclide	Activity (mCi/ml)	Nuclide	Activity (mCi/ml)
Mn-54	______	______	______
Co-58	______	______	______
Co-60	______	______	______
Cs-134	______	______	______
Cs-137	______	______	______
______	______	______	______
______	______	______	______
______	______	______	______
		Total	______

WEAK GAMMA EMMITERS

Nuclide	Scale	Scale (mCi/ml)	Scaler	Activity (mCi/ml)
Ni-63	(Co-60)	______	______	______
Sr-90	(Cs-137)	______	______	______
Tc-99	(Cs-137)	______	______	______
I-129	(Cs-137)	______	______	______
C-14	(Cs-137)	______	______	______
______	______	______	______	______

ALPHA EMMITERS: ALPHA ACTIVITY:____________________(nCi/ml)

Nuclide	Scaler	Activity (nCi/ml)
Pu-238	______	______
Pu-239	______	______
Pu-241	______	______
Am-241	______	______
Am-243	______	______
Am-244	______	______
Cm-242	______	______
TRU	______	______

Technician:__________________________ Supervisor:______________________

APPENDIX A

SOLID RADWASTE BOILER CHEMICAL NEEDS

A.1.0 PURPOSE

To determine the amount of acid or caustic needed by the solid radwaste auxiliary boiler.

A.2.0 EQUIPMENT:

A.2.1 pH meter
A.2.2 Buret, 50 ml
A.2.3 0.1N NaOH
A.2.4 0.1N H_3PO_4

A.3.0 PROCEDURE

A.3.1 Cool sample to ambient temperature. Measure 100ml of a representative sample aliquot into a 250ml beaker.

A.3.2 If sample pH is less than 8.0, titrate to pH 9.0 using 0.1N NaOH.

Calculate the amount of solid caustic needed:

NaOH (gm) = (ml 0.1N NaOH)(17gm/ml)

A.3.3 If sample pH is between 8.0 and 10.0, no caustic or acid is needed.

A.3.4 If sample pH is greater than 10.0, titrate to pH 9.0.

Calculate the amount of 85% phosphoric acid needed.

85% H_3PO_4(ml) = (ml 0.1N H_3PO_4)(11.5 ml/ml)

APPENDIX B

SOLIDS CONTENT

B.1.0 PURPOSE

To provide directions for the determination of the specific gravity and fraction solids of a liquid radwaste sample. A method to calculate the waste and asphalt feed rates to the extruder is also presented.

B.2.0 EQUIPMENT

B.2.1 Stainless Steel Planchet
B.2.2 Heat Lamp
B.2.3 Oven

B.3.0 PROCEDURE

B.3.1 Weigh a clean planchet to constant weight. (A)

B.3.2 Pour exactly 5.0 ml of sample into planchet.

NOTE: Ensure that cation and anion resins if present are in proper ratios.

B.3.3 Weigh planchet and sample. (B)

B.3.4 Ensure that sample is evenly distributed over bottom of planchet. Add demin water and stir if necessary.

B.3.5 Evaporate planchet to dryness under a heat lamp.

B.3.6 Bake planchet in oven at 125°C (120-130) for 10 minutes.

B.3.7 Cool and desicate.

B.3.8 Weigh to constant weight. (C)

B.4.0 <u>CALCULATIONS</u>

B.4.1 Specific Gravity = (B-A)/5

B.4.2 Fraction Solids = (C-A)/(B-A)

B.4.3 Fraction Water = 1 - Fraction Solids

B.4.4 Evaporator Bottoms <u>maximum</u> feed rate (gpm):

= 0.528/(Fraction Water x Specific Gravity)

B.4.5 Waste Sludge <u>maximum</u> feed rate (gpm):

= 0.352/(Fraction Water x Specific Gravity)

B.4.6 Asphalt <u>minimum</u> feed rate (gpm):

= 0.528 (Fraction Solid)/(Fraction Water)

NOTES:

1. Absolute minimum asphalt rate is 0.065gpm for lubrication of extruder.
2. Waste to Asphalt ratio, by weight, is $\leq$1.0.

APPENDIX C

GROSS ALPHA ANALYSIS

C.1.0 PURPOSE:

To provide direction for the gross alpha analysis of waste samples.

C.2.0 EQUIPMENT

Porcelain Crucible, <100 ml capacity.

C.3.0 PROCEDURE

C.3.1 Clean a porcelain crucible.

C.3.2 Ignite crucible in muffle furnace at 1400°F for 20 min.

C.3.3 Cool in desicattor and weigh to constant weight. (F)

C.3.4 Transfer approximately 2 ml of sample to crucible.

NOTE: Ensure that cation and anion resins if present are in proper ratios.

C.3.5 Weigh sample and crucible. (G)

C.3.6 Cover crucible and dry in an oven at 105°C. Then ash crucible in muffle furnace, at 1400°F for 30 min.

C.3.7 Remove from furnace, cool and add 5ml concentrated nitric acid to the ash. USE CAUTION!

C.3.8 Break up any large particles with a stirring rod.

C.3.9 Quantitatively transfer the dissolved ash to a 100 ml volumetric.

C.3.10 Dilute ash to 100ml with demin water. USE CAUTION! This is solution A.

C.3.11 Pipet 10.0ml into a second 100ml volumetric.

C.3.12 Dilute to 100 ml with demin water. This is solution B.

C.3.13 Pipet 1.0, 2.0 and 5.0 ml of each solution into a label 2" SS planchet. Containing 9.0, 8.0 and 5.0 ml respectively of demin. water to bring total volume to 10.0 ml.

Solution	(ml)	Label
A	1.0	100
A	2.0	50
A	5.0	20
B	1.0	1000
B	2.0	500
B	5.0	200

C.3.14 Dry the planchets with a heat lamp.

C.3.15 Alpha count each on a PC-5 for 5-30 min. Obtain a minimum of 10 net counts.

C.3.16 Alpha count a blank for the maximum time duration used above.

C.3.17 Calculate the net alpha count:

Net dpm/gm = L(A-B)/(G-F)E

Where:

L = planchet label
A = samples alpha count (cpm)
B = blanks alpha count (cpm)
G = sample and crucible weight (gm)
F = crucible weight (gm)
E = PC-5 efficiency (cpm/dpm)

C.3.18 Graph the data. Net dpm/gm on vertical axis and planchet label on the horizontal.

C.3.19 Extrapolate and determine the net dpm/gm which would correspond to a planchet label of infinity (infinite dilution).

C.3.20 Calculate the alpha activity (nCi/ml):

nCi/ml = C SG/2200

where:

C = Alpha activity at infinite dilution (dpm/gm)

SG= specific gravity of sample (gm/ml)
2200 = dpm/nCi

APPENDIX D

SCALING FACTOR CALCULATION

D.1.0 PURPOSE

To provide the methodology to be used in the calculation of scaling factors necessary to determine the activity of nuclides in radwaste shipments not directly measured.

D.2.0 PROCEDURE

NOTE: Scaling factor determinations should be performed within one week of receiving primary sample composite analysis data from vendor.

Determine the scaling factors associated with each waste form by referring to and filling out figure D.1-V.

D.2.1 Obtain a copy of the most recent primary sample composite analysis report from supervision. Also obtain the primary sample composite gross alpha analysis report.

D.2.2 Find the scaling factors for the five high intensity transuranic alpha emitters (ie., Cm-242, Pu239, Pu238, Cm243, 244, Am241) using the formula:

$$(SF)_i = \frac{(A_i)}{A_{Cm242} + A_{Cm243} + A_{Pu238} + A_{Pu239} + A_{Am241}}$$

Where: A_i = specific activity of a respective high intensity alpha emitter, i.

$(SF)_i$ = scaling factor for isotope, i.

D.2.3 Note that this process amounts to finding the fractional distribution of each of the high intensity alpha emitters and that Pu-241 (which is a low intensity alpha emitter) is not included.

D.2.4 Set the scaling factor for TRU with half-line greater than five years equal to (1- scaling factor for Cm-242).

D.2.5 Find the scaling factor for Pu-241 by ratioing the Pu-241 specific activity found on the primary sample composite analysis report to the gross alpha specific activity.

NOTE: Ensure units are consistent (ie., normally nCi/g). If not, make appropriate conversions.

D.2.6 Find the scaling factors for Tc-99, I-129, and Sr-90 by ratioing the specific activity of each respective isotope to Cs-137 found on the primary sample composite analysis report.

D.2.7 Find the scaling factor for Ni-63 by ratioing the specific activity found on the primary sample composite analysis report of Ni-63 to Co-60.

D.2.8 Insert the date of the Primary Sample Analysis Report at the bottom of the Record Sheet.

FIGURE D.1-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

SCALING FACTOR DETERMINATION RECORD SHEET

Waste Form: ______________________ Date: ______________________

Radionuclide	Scaling Factor Calculation			Scaling Factor
Cm-242	A_i/Sum	/	=	________
Cm-243, 244	A_i/Sum	/	=	________
Am-241	A_i/Sum	/	=	________
Pu-238	A_i/Sum	/	=	________
Pu-239	A_i/Sum	/	=	________
TRU	$1-SF_{Cm}-242$	-	=	________
Pu-241	A_i/Gross	/	=	________
Tc-99	Tc-99/Cs-137	/	=	________
I-129	I-129/Cs-137	/	=	________
Sr-90	Sr-90/Cs-137	/	=	________
Ni-63	Ni-63/Co-60	/	=	________
C-14	C-14/Cs-137	/	=	________

DATE OF PRIMARY SAMPLE ANALYSIS REPORT ______________________

Technician: ______________________ Supervisor: ______________________

APPENDIX F

GREAT LAKES pH METER

F.1.0 PURPOSE

To provide directions for the maintenance, calibration and trouble shooting of the pH meter in the solid radwaste system at Unit 2.

F.2.0 PRECAUTIONS

Obtain a specific RWP to work on the probe.

F.3.0 MAINTENANCE

F.3.1 Check the glass electrode monthly to see that it is not fouled. If it is fouled, wipe with a soft cloth, taking care not to break glass electrode.

F.3.2 If the glass electrode cannot be wiped clean, try soaking a few minutes in hydrochloric acid(10%). Tar or greases can usually be removed with acetone. Oil is best removed with dishwashing detergent.

F.3.3 The other electrodes on the probe need not be as clean as the glass electrode, but should not be grossly fouled.

F.3.4 Never use abrasive cleaners on the probe or glass electrode.

F.3.5 Removable salt bridge on Derakane probes.

F.3.5.1 Derakane probes are manufactured so that the outer chamber on the standard cell (this is the component with the white ceramic in the center) may be removed. This enables the user to flush out the standard cell and renew the buffer solution.

F.3.5.2 To remove the outer chamber, turn it counterclockwise. It may be necessary to use a pair of pliers to loosen the chamber. If pliers are used, take care not to damage the glass electrode.

F.3.5.3 After removing the outer chamber, pour out the contents of the standard cell, flush the cell cavity with distilled water, place new buffer in the cavity and replace the outer chamber (salt bridge) by turning it clockwise until finger tight. Then tighten a 1/4 turn more with a pair of pliers.

F.4.0 CALIBRATION

F.4.1 Place the RUN/TEST switch to RUN, put the pH probe in a pH 7 buffer and allow the reading to settle. Use the CAL adjust to make the meter read pH 7 (or the pH of the buffer at the ambient temperature).

F.4.2 Rinse the probe in tap water or distilled water and then place in a second buffer whose value is other than pH 7 (4, 7 and 10 are readily available from GLI). Allow the reading to stabilize and then use the SPAN control to make the meter read the pH of the second buffer.

F.4.3 If the pH probe used with the system has a buffer solution other than pH 7 in the standard cell, calibrate as follows:

F.4.3.1 Place the RUN/TEST switch to RUN, put the pH probe in a buffer that has the same pH as the pH of the standard cell solution. Allow the reading to settle. Use the CAL adjust to make the meter read the pH of this buffer.

F.4.3.2 Rinse the probe in tap water or distilled water and then place in a pH 7 buffer. Allow the reading to stabilize and then use the SPAN control to make the meter read pH 7.

F.4.4 If steps F.4.2 and F.4.3 are followed calibration will be easily accomplished. If, on the other hand, a pH 7 buffer is not used as described, then it is necessary to repeat the calibration steps until the meter reading agrees at both check points.

F.4.5 An alternate method of calibration is to determine the pH of the process on a separate pH meter and then set the CAL adjustment so that the two readings agree. This method is not recommended if accurate readings are required.

F.5.0 TROUBLESHOOTING

Contact I&C for assistance, if the meter will not calibrate.

F.5.1 A few simple measurements can determine if a probe is operational or not. Two 9-volt batteries (or similiar DC power sources), a multimeter and two buffer solutions are needed for the test.

F.5.2 Disconnect the probe leads from the rest of the system. Place the probe in pH 7 buffer at room temperature.

F.5.3 The resistance between the yellow and black probe leads should be near 320 ohms.

F.5.4 Connect one of the batteries as follows: + terminal to the blue lead, - terminal to the black lead. Connect the other battery as follows: + terminal to the black lead, - terminal to the white lead. The voltage between the red lead and the green lead should be less than $\pm$ 50 millivolts.

F.5.5 With the connections as in F.5.4, but with the probe in a pH 4 buffer, the voltage measured from the green lead to the red lead should be in the range of +100 to +230 millivolts with +165 millivolts a typical reading.

<u>NOTE:</u> If the batteries required for steps F.5.4 and F.5.5 are not available, these steps can be accomplished using the analyzer power supply. Disconnect only the red and green leads at the junction box (or in the analyzer if there is no junction box), turn on the power to the analyzer and make the measurements across the red and green leads as described in F.5.4 and F.5.5.

FIGURE F-1-M

NINE MILE POINT NUCLEAR STATION - UNIT 2

SOLID RADWASTE pH MONITORS

Date:______________________

2WWS - A1T105: Waste Sludge

Initial Buffer 7 value: ________________
Final Buffer 7 value: ________________
Initial Buffer 10 value: ________________
Final Buffer 10 value: ________________

Comments:

2WWS - A1T110: Waste Concentrator

Initial Buffer 7 value: ________________
Final Buffer 7 value: ________________
Initial Buffer 10 value: ________________
Final Buffer 10 value: ________________

Comments:

Technician:____________________________ Supervisor:________________________

APPENDIX G

SAMPLING OF ASPHALT TANKERS

G.1.0 PURPOSE

To provide directions for the sampling of incoming tanker trucks shipments of asphalt to be used in the solid radwaste system.

G.2.0 REFERENCES

ASTM D140-70 Standard Methods of Sampling Bituminous Materials

G.3.0 PRECAUTIONS

Asphalt shipments are hot. Wear glasses with eyeshields, heavy gloves and heavy clothing while working with or near hot asphalt.

G.4.0 EQUIPMENT

G.4.1 Sample containers shall be wide-mouth cans with lined screw caps or Triple-Seal friction-top cans. They shall be new and not rinsed or wiped with oil or solvent.

G.4.2 Sampling device (if used). A clean wide-mouth can securely fastened to a handle of suitable length.

G.5.0 PROCEDURE

G.5.1 Sample Tap: If a sample tap is available on the tanker truck, it may be used.

G.5.1.1 Purge the sample tap with a minimum of one gallon of asphalt. Discard.

G.5.1.2 Fill the sample container, seal and label.

G.5.2 No sample Tap: If no tap is available, the dip method must be used.

G.5.2.1 Using the sampling device, obtain a sample. Be certain that the device is fully submerged before withdrawing.

G.5.2.2 Immediately fill the sample contianer, seal and label.

APPENDIX H

SOFTENING POINT OF ASPHALT

H.1.0 PURPOSE

To provide directions for the determination of the softening point of asphalt in the range of 80-200°C by the ring and ball method.

H.2.0 REFERENCES

H.2.1 ASTM D-36-76 Standard Test Method for Softening point of Bitumen (Ring-and-Ball Apparatus)

H.2.2 LIP-33 Niagara Mohawk Procedure, Calibration Verification of Thermometers

H.3.0 PRECAUTIONS

Wear glasses with eyeshields, heavy gloves and heavy clothing while working with or near hot asphalt.

H.4.0 EQUIPMENT

H.4.1 Softening Point Apparatus. Fisher Scientific 01-551 or equivalent as described in ASTM D36-76. Fill with USP glycerin.

H.4.2 Pouring Plate. A smooth plate treated to prevent asphalt from adhering to it. Suitable treatment includes a thin layer of a glycerin/dextrin mixture, talc or china clay.

H.4.3 Thermometer - ASTM 16 or equivalent. Calibraton checked yearly in accordance with LIP-33.

H.5.0 SAMPLE PREPARATION

H.5.1 Heat the sample with care to prevent local overheating, with constant stirring until it has become sufficiently fluid to pour.

NOTE: Do not heat sample above 140°C (280°F). Avoid incorporating air bubbles, and complete heating process in 2 hours.

H.5.2 Pour a slight excess of the heated sample into two rings, preheated to approximately the pouring temperature. While being filled, the rings shall rest on the pouring plate, previously treated.

H.5.3 Cool the speciman for a minimum of 30 min.

H.5.4 Cut the excess material off cleanly with a slightly heated knife or spatula.

H.6.0 <u>PROCEDURE</u>

Complete figure H.1-V

H.6.1 The glycerin bath should be preheated to 32 ± 1°C (90 ± 2°F) with the depth adjusted to between 4.0 and 4.25 inches.

H.6.2 Place the asphalt filled rings in assembled apparatus bath and allow to equilibrate for 15 min.

H.6.3 Using forceps, place a ball, previously adjusted to the bath temperature, in each ball centering guide.

H.6.4 Apply heat in such a manner that the temperature is raised 5 ± 0.5°C (9 ± 1°F) per minute.

<u>NOTE:</u> Variations outside this range are permissible for the first 3 minutes.

H.6.5 Record the temperature at which the speciman touches the bottom plate.

H.6.6 If the difference between the two specimans exceed 1°C (2°F), repeat the test.

FIGURE H.1-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

INCOMING ASPHALT QUALITY CHECK

DATE:____________________

Thermometer Calibration:____________________________(within 1 yr)

Glycerin Bath Temperature:

Time (min)	Temp (°F)	Time (min)	Temp (°F)
Start	________	10	________
1	________	11	________
2	________	12	________
3	________	13	________
4	________	14	________
5	________	15	________
6	________	16	________
7	________	17	________
8	________	18	________
9	________	19	________

Softening Temp. #1: ______________________________

Softening Temp. #2: ______________________________

Within 2°F?: ______________________________

Temp. Rise Rate Within Limit:_________________________

Comments:

Technician:____________________________ Supervisor:______________________

APPENDIX I

SOLIDIFICATION VERIFICATION

I.1.0 PURPOSE

To provide directions for the verification that a radioactive waste sample is solidified by the asphalt extruder evaporator.

I.2.0 PRECAUTIONS

Wear glasses with eyeshields, heavy gloves and heavy clothing while working with or near hot asphalt.

I.3.0 EQUIPMENT

I.3.1 Oven

I.3.2 Stirring implement. A small wooden split.

I.4.0 PROCEDURE

I.4.1 Obtain a planchet prepared as in Appendix B for solids content.

I.4.2 Determine the weight of the solid waste contained in the planchet. (C-A).

I.4.3 Add asphalt to the planchet. For evaporator bottoms use an equal amount, for waste sludge use 1 1/2 times as much. Weigh asphalt to within ± 10% of the required amount.

I.4.4 Heat to 150°C in the oven. Periodically withdraw planchet and stir to incorporate the solids into the asphalt.

I.4.5 When the solids are throughly incorporated in the asphalt, cool the planchet. It is permissible to place the planchet on a wet surface to speed cooling.

I.4.6 Observe the product to verify solidification.

I.4.6.1 Check that it is not soupy (does not flow).

I.4.6.2 Check that it is cohesive (there is enough asphalt so that all the solids are stuck together and covered).

APPLENDIX J

ASPHALT SOLID RADWASTE COMPRESSIVE STRENGTH

J.1.0 PURPOSE

To provide directions for the sampling of the radwaste extruder evaporator asphalt product and subsequent compressive strength test.

J.2.0 REFERENCES

ASTM D1074-83 Standard Test Method for Compressive Strength of Bituminous Mixtures.

J.3.0 PRECAUTIONS

Asplant and the extruder evaporator are hot. Wear glasses with eyeshields, heavy gloves and heavy clothing while working with or near the extruder or hot asphalt.

J.4.0 EQUIPMENT

J.4.1 Testing Machine

J.4.2 Molds - Cylindrical, thin wall aluminum nominally 2 inches in diameter by 5 1/2 inches high. Form the mold be wrapping a 2" diameter cylinder with a thin aluminum sheet 5 1/2" x 8". Fold and press the excess metal along the side of the cylinder to form a tight seal. Cover the bottom of mold with an aluminum cap. Seal the seam along the cap so that the molten asphalt will not leak out.

J.5.0 SAMPLING AND MOLDING

J.5.1 Obtain an appropriate RWP for sampling and molding.

J.5.2 Attach the mold rigidly to a long stick with the mold bottom resting on a plate.

J.5.3 Fill the mold halfway with solid radwaste being extruded from the 2WSS System.

J.5.4 Spade the asphalt in the mold with a heated spatula to reduce voids.

J.5.5 Fill the mold and repeat spading action.

J.5.6 Allow speciman to cool and harden.

J.5.7 Remove the mold from the asphalt. Cut and trim the asphalt to a cylindrical shape 2" x 4" nominal dimensions.

J.5.8 Examine the asphalt closely to verify that no voids exist. Repeat molding if voids are found.

J.5.9 Measure the specimans perimeter at the narrowest point.

J.5.10 Calculate the cross sectional area and record

Area = $P^2/12.57$

Where: P = Perimeter (inches)
12.57 = 4π

J.6.0 <u>CONDITIONING AND TESTING</u>

J.6.1 Store the speciman in air for at least 24 hours at 55°F (52.5 - 57.5).

J.6.2 Measure Compressive Strength

<u>NOTE:</u> Time and Temperature is essential. Room must be cool (less than 75°F) and compression test must be within 10 min. of removal from conditioning. Asphalt will lose considerable compressive strength as it warms.

J.6.2.1 Record room temperature. Verify that it is less than 75°F.

J.6.2.2 Load speciman squarely in machine.

J.6.2.3 Advance machine to fit snugly against speciman.

J.6.2.4 Compress at 0.05 in/min.

J.6.2.5 Record the force exerted in 5 lb_f increments starting at 50 lb_f.

J.6.3 Determine the maximum force exerted and calculate the compressive strength.

CS = F/A

Where: F = Maximum force exerted (lb_f)
A = Orginal nominal area ($inch^2$)
CS = Compressive Strength (psi)

FIGURE J.1-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

ASPHALT COMPRESSIVE STRENGTH

Waste Form:______________________ Sample Date:________________

Batch ID: ______________________

Were Voids evident?:______________________

Narrowest Perimeter: ______________________ inches (P)

Cross Sectional Area: ______________________ sqaure inch ($P^2/12.57$)

Room Temperature: ______________________ °F (< 75°F)

Compress Force Exerted: (lb_f)

Maximum Force:____________________lb_f

Compressive Strength: ____________psi (>50 psi)

Comments:

Technician:__________________________ Supervisor:______________________

APPENDIX K

ASPHALT WASTE SHIPMENT ISOTOPIC SUMMARY

K.1.0 PURPOSE

To provide directions for the calculation of the activity level and concentration in solidified asphalt containing radioactive waste at Unit 2.

K.2.0 PROCEDURE

K.2.1 Record the liner I.D. and the date.

K.2.2 Check radwaste operators log to determine the volume and batch # of the waste actually fed to the liner.

K.2.3 Also record the volume of solidified waste/asphalt in the liner.

K.2.4 Review chemistry data sheet and record the date of the isotopic analysis for the waste batch, days since the analysis, nuclides quantified (direct and indirect) and activity concentration.

NOTE 1: The alpha and gamma spectrum should have been performed on the same date.

NOTE 2: Be certain units are all in μCi/ml.

K.2.5 Decay correct the activity concentration of each nuclide with a short half-life.

NOTE: This step is optional at supervisory discretion

DA = FA exp (DYF)

Where:

DA = Decayed Activity (μCi/ml)
FA = Feed Activity (μCi/ml)
DY = Days since analysis
F = Decay Factor from table K.1 $(days)^{-1}$

K.2.6 Calculate the activity in the liner.

LA = 3785 DAFV

Where:

LA = Activity in Liner (μCi)
3785 = ml/gal
DA = Decayed Activity (μCi/ml)
FV = Volume of waste fed to liner (gal)

K.2.7 Calculate the activity concentration in the liner.

$$LC = LA/(LV\ 3785)$$

Where:

LC = Activity Concentration in Liner (μCi/ml)
LA = Activity in Liner (μCi)
LV = Solid Volume in Liner (gal)
3785 = ml/gal

K.2.8 Tritum

K.2.8.1 Record the activity concentration as equal to the highest concentration recently detected at Unit 2. Consult supervision if necessary and record an entry in comment area explaining source.

K.2.8.2 Decay correction is not necessary.

K.2.8.3 For solidified sludge estimate the liner activity concentration at 10% of the feed concentration.

K.2.8.4 For solidified evaporator bottoms estimate the liner activity concentration at 1% of the feed concentration.

K.2.8.5 Calculate the total tritium activity in the liner.

$$LA = LC\ LV\ 3785$$

Where:

LA = Activity in Liner (μCi)
LC = Activity concentration in liner (μCi/ml)
LV = Solid Volume in Liner (gal)
3785 = ml/gal

FIGURE K.1-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

ASPHALT WASTE SHIPMENT ISOTOPIC SUMMARY

Liner #: ______________________ Date: ________________

Batch #: ______________________ Volume Fed: ________________ gal. (FV)

Solid Volume in Liner: ____________________ gal. (LV)

Isotopic Analysis Date: ____________________

Days Since Analysis: ____________________ days ((DY)

NUCLIDE	FEED (µ Ci/ml)	DECAYED (µ Ci/ml)	LINER (µ Ci/ml)	LINER (µ Ci/ml)
H3				
Total				

Comments:

Technician: ______________________ Supervisor: ______________________

FIGURE K.1

NINE MILE POINT NUCLEAR STATION - UNIT 2

NUCLIDE DECAY FACTORS

NUCLIDE	FACTOR $(Days)^{-1}$
Cr 51	-2.50E-2
Co 58	-9.79E-3
Nb 95	-1.97E-2
Cs 134	-9.20E-4
Ba 140	-5.42E-2
Ce 141	-2.13E-2
Cm 242	-4.25E-3

REFERENCE:

ORNL/NUREG/TM-102

APPENDIX L

DEWATERED RESIN SHIPMENT ISOTOPIC SUMMARY

L.1.0 PURPOSE

To provide directions for the calculation of the activity level and concentration in dewatered resin shipments at Unit 2.

L.2.0 PROCEDURE

L.2.1 Record the I.D. and date

L.2.2 Check radwaste operators log to determine the batch # of resin fed to cask and the dewatered volume of resin in cubic feet.

CF = 7.48 gallons

L.2.3 Review chemistry data sheets and record the nuclides quantified (direct and indirect) and activity concentration.

NOTE: Be certain that all units are in μCi/ml.

L.2.4 Calculate the activity in the cask

$$CA = 28{,}317\ AV$$

Where:

CA	=	Activity in cask (μCi)
28,317	=	ml/cubic foot
A	=	Activity Concentration (μCi/ml)
V	=	Volume of dewatered resin in cask (CF)

L.2.5 Calculate the activity of tritium

Record the activity concentration as equal to the highest concentration recently detected at Unit 2. Consult supervision if necessary and record an entry in comment area explaining source.

Correct tritium concentration by multiplying by the fraction water in cask.

$$AT = FWA$$

Where:

AT	=	Actual tritium concentration (μCi/ml)
FW	=	Fraction of cask which is water
A	=	Recorded activity of Tritium (μCi/ml)

FIGURE L.1-V

NINE MILE POINT NUCLEAR STATION - UNIT 2

ASPHALT WASTE SHIPMENT ISOTOPIC SUMMARY

Cask #:____________________ Date:________________

Batch #: ____________________ Resin Volume:________________CF

NUCLIDE	CONCENTRATION (μCi/ml)	CASK (μCi)
Total		

Comments:

Technician:____________________ Supervisor:____________________