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# Test Procedures and Instructions for Hanford Tank Waste Supernatant Cesium Removal

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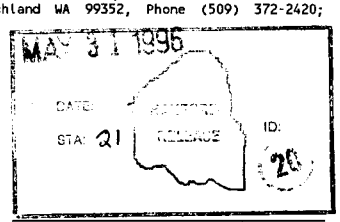
Abstract: This document provides specific test procedures and instructions to implement the test plan for the preparation and conduct of a cesium removal test using Hanford Double-Shell Slurry Feed supernatant liquor from tank 241-AW-101 in a bench-scale column. Cesium sorbents to be tested include resorcinol-formaldehyde resin and crystalline silicotitanate. The test plan for which this provides instructions is WHC-SD-RE-TP-022, *Hanford Tank Waste Supernatant Cesium Removal Test Plan.*



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# Test Procedures and Instructions for Hanford Tank Waste Supernatant Cesium Removal

## 1.0 INTRODUCTION

Cesium-137 ($^{137}Cs$) is a primary radiation source in the dissolved tank waste at the Hanford Site. $^{137}Cs$ removal from the waste can reduce the hazard and waste classification of the low level waste and reduce treatment and disposal costs.

The object of these test procedures is to conduct a test of cesium sorption of actual Hanford tank waste with materials which have been proposed for development and potential deployment in Hanford Site waste treatment. Treated effluent from these tests is proposed to be received by Battelle Pacific Northwest National Laboratory to conduct technetium sorption studies in further support of site waste treatment needs.

These procedures are written to directly meet the procedural needs of the *Hanford Tank Waste Supernatant Cesium Removal Test Plan* (Hendrickson and Duncan 1996) to ensure adequacy of conduct and capture of appropriate samples and data.

This work is funded by the U.S. Department of Energy's (DOE) Office of Science and Technology under FY1996 technical task plan RL46WT41 *Cesium Removal Tests Using Hanford DSSF Supernatants.*

## 2.0 DESCRIPTION OF TEST

For this cesium ion exchange test, a test apparatus will be constructed, functionally tested, then placed in a hot cell within the 222-S Laboratory. Approximately four and one-half liters of drainable Double-Shell Slurry Feed (DSSF) supernatant liquor from Hanford Tank 241-AW-101 will be acquired through the sampling efforts of the TWRS Characterization Project at Hanford (Benar 1996) and will be placed in the hot cell with the test apparatus. The DSSF supernatant liquor will be diluted with water to a concentration target of five molar sodium, mixed, settled, decanted, and centrifuged to remove particles, and then placed in the feed tank(s) to be used as the feed for the column flow tests.

The test apparatus will contain two test columns and two scavenging (guard) columns. One test column will be packed with crystalline silicotitanate (CST) and the other with Resorcinol-Formaldehyde (R-F). The scavenging columns, packed with the same materials, will be used to reduce any cesium remaining in the effluent. The effluent is intended to be provided to subsequent Tanks Focus Area (TFA) tasks which will require the material for

technetium (Tc) removal studies. A sketch of the process flows and instrumentation is

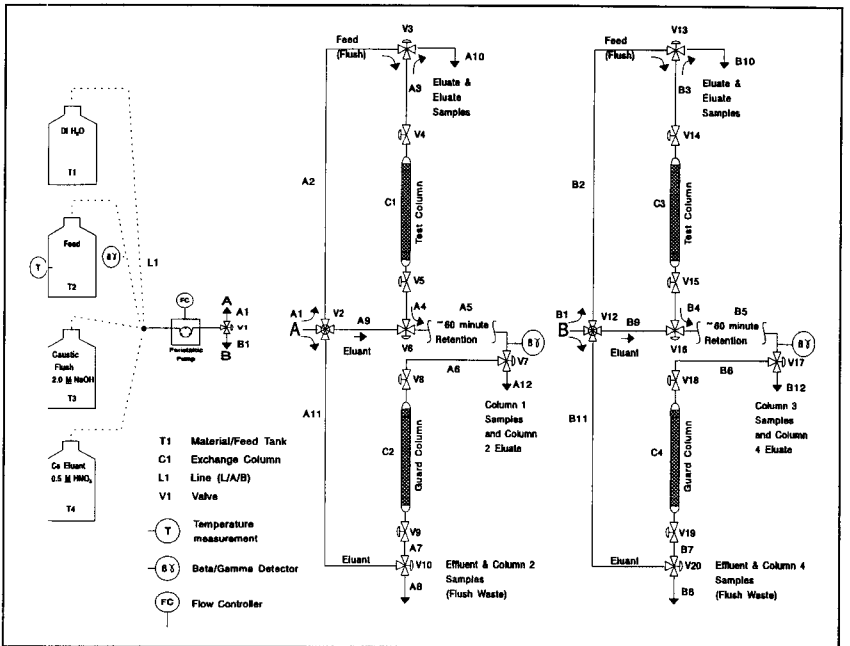


**Figure 1:** Bench-Scale Cesium Exchange Flows and Instrumentation

provided in Figure 1. Physical layout of the test apparatus is provided in Figure 2.

During the loading phase of the column test, the feed solution will be pumped from the feed tank downward through a test column of cesium sorbent. The effluent from the test column will be monitored for cesium breakthrough and will be sampled periodically. Samples will also be taken of the feed, guard column effluent, digested resin, and the eluate as necessary. Sample analysis, as available, will be performed to determine the concentration of analytes to which the cesium ion exchange and vitrification processes are sensitive. These analytes include: cesium-137, total cesium, strontium, sodium, potassium, rubidium, aluminum, phosphorus (for phosphate), chromium, and iron as well as the pH of the sample. The feed temperatures will be recorded at the beginning and during the test.

After cesium breakthrough is detected in the R-F test column, the R-F column will be flushed and the cesium will be eluted from the R-F. After cesium breakthrough is detected in the CST test column, the CST column will be flushed and drained, removed from the system, digested, sampled, and disposed. Initial plans for the cesium sorbent materials are in-cell digestion and disposal due to the high shielding requirements and limited capacity of

manipulators entailed with removal from the hot cell (Hendrickson and Duncan 1996).

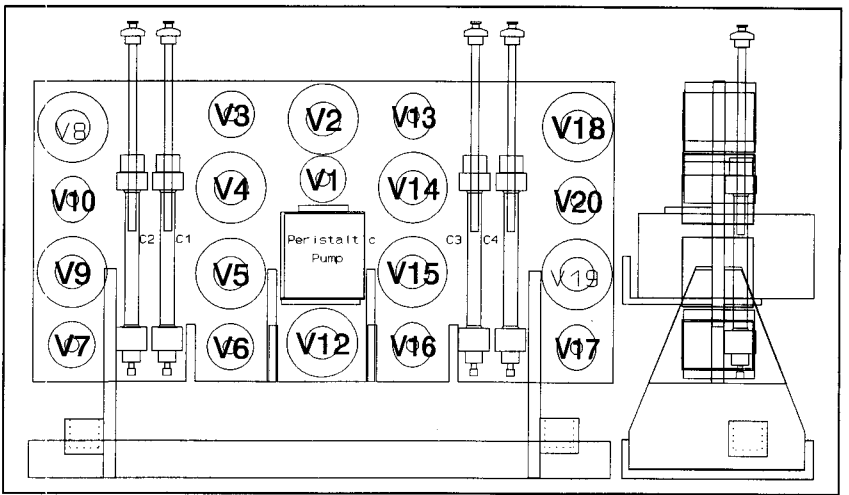


**Figure 2:** Physical Test Assembly Valve and Column Arrangement

The performance of the sorbents in the removal of cesium from DSSF, and process considerations of such removal, will be evaluated and reported in a final report.

## 2.1 TEST ENVIRONMENT

The test environment is primarily that of the WHC operated 222-S analytical laboratories.

During assembly and acceptance testing of the exchange equipment, assembly is largely anticipated to occur in the 306E laboratories (300 Area) and acceptance testing in non-radioactive portions of the 222-S laboratories (building 222-SA). The exchange equipment will be placed into a high-radiation hot cell (1F) of the 222-S laboratory where the process tests will occur.

Waste material core samples, retrieved from tank farms will be brought into the 222-S laboratory and extruded in-cell 11A. Smaller sample bottles (polyethylene) of the waste supernatant liquor will be transferred from cell 11A to cell 1F for the test work.

With the exception of sample analyses at the 222-S laboratory or the Battelle Pacific Northwest National Laboratory 325 laboratory, the remainder of work will be done within the 1F hot cell at 222-S.

## 2.2 EQUIPMENT AND FACILITIES

Equipment needed for the apparatus assembly includes:

| | |
|---|---|
| 4 | Exchange Columns, 1 cm internal diameter (ID), packing height 10 cm, glass walled |
| 8 | column clamps for extraction column retention |
| 1 | Peristaltic pump, flow range from 15 to 80 mL/hr |
| 8 | gate valves, ~3 mm ID, Teflon[1] core |
| 9 | three way valves, ~3 mm ID, Teflon core |
| 2 | four way valves, ~3 mm ID, Teflon core |
| 5m | Tygon[2] tubing, ~0.89, 1.59 mm, 3.175 mm, and 4.76 mm ID |
| 1 | Feed Reagent Tanks, polyethylene, 1 L (0.5 M $HNO_3$) |
| 2 | Feed Reagent Tank, polyethylene, 2 L (waste feed and $H_2O$) |
| 1 | Feed Reagent Tank, polyethylene, 500 mL (2.5 M NaOH) |
| 9 | Effluent Receivers, polyethylene, 2 L |
| 1 | Flush receiver, polyethylene, 250 mL |
| 1 | Eluant receiver, polyethylene, 250 mL |
| 1 | Shears for cutting large feed reagent tanks |
| 75 | Glass/polyethylene sample vials with stoppers, 15 mL |
| 12 | Centrifuge cones, glass, 50 mL |
| 2 | sample vial holders |
| 1 | Continuous $\beta/\gamma$ detector and recorder |
| 1 | thermometer |
| 1 | Test/sample clock |
| 25 | R-F resin, grams (MSDS in Appendix B) |
| 40 | CST engineered exchange material, grams (MSDS in Appendix B) |

Reagents required (for material safety data sheets, see Appendix B), and anticipated volumes include:

| | | |
|---|---|---|
| Deionized Water | 5.5 -7.5 | Liters |
| Sodium Hydroxide (NaOH), 2.5 M | 1.0 | Liters |
| Nitric Acid ($HNO_3$), 0.5 M | 0.15 | Liters |
| Nitric Acid ($HNO_3$), 3.0 M | 0.2 | Liters |
| Hydrofluoric Acid (HF), 1 M | 0.1 | Liters |
| Hydrochloric Acid (HCl), 2 M | 0.2 | Liters |
| Tank Waste, 241-AW-101 | 4.5 | Liters |

Facilities required are those of the 222-S laboratory 1F hot cell, and nonradioactive portions of 222-S and 306E for equipment assembly and acceptance testing.

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[1] Teflon is a trademark of E. I. duPont de Nemours, Co., Wilmington, DE.

[2] Tygon is a trademark of Norton Performance Plastics, Akron, OH.

**Table 1:** Reagent Densities at Experimental Temperature

| Reagent | mol/L | Density (g/mL) 27°C |
|---|---|---|
| HF | 1 | 0.998118 |
| HCl | 2 | 1.030655 |
| $H_2O$ | 55.51 | 0.996513 |
| $HNO_3$ | 3 | 1.09902 |
| $HNO_3$ | 0.5 | 1.017293 |
| NaOH | 2.5 | 1.042449 |

Source: *Perry's Chemical Engineer's Handbook*, Sixth Edition, R.H.Perry, D. W. Green, J. O. Maloney, McGraw-Hill Book Company, New York, New York, 1984.

Additional or differing equipment and reagents may be applied at the discretion of operating personnel with concurrence of the lead chemist or engineer. Change procedures are provided in § 5.0. A detail of equipment and reagents used in conduct will be provided in the test report.

### 2.3 DATA

Parameters to be measured, and the precision required is, in large part, defined by the test subtask. In general, the parameters of measurement, when not in chemical assay, are those of interval, volume, mass, and beta/gamma decay. The subtasks to be described and executed herein are waste feed preparation exchange column packing, activation, flush, operation, sampling, elution, and waste material handling. Chemical assay quality assurance is described by Meznarich (1995). Data acquisition and handling particular to this test will be defined within the Quality Assurance project Plan written to support this test. Data Sheets and checksheets for operation are provided in Appendix A. Completed datasheets and checksheets will be entered into appropriate logbooks. 222-S chemical assay analyses will be stored in the Laboratory Information Management System.

### 2.4 CRITERIA/CONSTRAINTS

The nature of this test is that of a treatability test. As such, the activities encompassed within this test are governed by WAC 173-303-071(3)(r) [*Treatability Study Samples*] and WAC 173-303-071(3)(s) [*Samples undergoing treatability studies at laboratories and testing facilities*] thereby generating a requirement that the Washington State Department of Ecology (WDOE) be notified, in writing, of the intent to conduct treatability studies no less than 45 days prior to conducting the studies. Treatment of actual waste in the test apparatus shall not proceed unless such a notice compliant with WAC 173-303-071(s)(i) is submitted. (WDOE 1994) As described by the test plan (Hendrickson and Duncan 1996), compliance with these provisions has been met with 1988 notification.

ALARA principles must guide all actions in this test.

### 2.5 WASTE MANAGEMENT AND DISPOSAL

Wastes from the conduct of this test scope will include extraction media, extraction columns, sample bottles, feed bottles (tanks), tygon tubing, valves, waste liquor, treatment effluent and eluant, and sampling wastes. All materials having contacted the tank waste liquor must be considered mixed wastes as the tank wastes have been designated to contain F-listed solvents (EPA 1986). To the greatest extent possible, bottles containing wastes brought into the hot cell will be of polyethylene so that they may be melted down within the cell following use. The extraction media will be dissolved, sampled, and slurped (poured down hot cell drain). Eluants, excess and spent samples, and undispositioned effluent will be slurped. Glass vessels will be decontaminated and removed from the cell for appropriate waste disposal.

Although there has been some consideration for further application of the extraction media, preliminary dose models (Hendrickson and Duncan 1996) indicate that shielding sufficient to reduce dose to the 222-S laboratory administrative requirement of 10 mR/hr would exceed the 18 kg (40 lb) capacity of the cell manipulators. As such, it cannot be considered safe to remove these materials from the hot cell intact. Further, preliminary calculations would indicate that achievable removals within the manipulator capacity would not significantly exceed one percent of the loaded CST column ($\sim$60 mg) with each loadout. These minute removal capabilities are not considered economically or technically justifiable.

## 3.0 EXPECTED RESULTS

This test will demonstrate the cesium selectivity and load capacity of CST and R-F using actual Hanford DSSF waste. This information will be compared to similar data gathered using simulants and will be used to validate the simulant data's use in the designing of a cesium removal pretreatment process.

Success of these tests will be in the form of chemical analysis and automatic beta/gamma sample counts demonstrating rise in concentration of the effluent cesium beyond 50% of inlet concentration (breakthrough, $\lambda_{50}$) such that the number of column volumes processed at breakthrough be determinable on a constant slope.

Prior development of these sorption materials has indicated that treatment expectations for the R-F resin may vary from 30 to 65 column volumes of waste feed to $\lambda_{50}$ while the CST treatment may vary from 300 to 800 column volumes of waste feed to $\lambda_{50}$. Due to the wide range of these expectations, on-line beta-gamma detection capability will be required to ensure adequate sampling to describe the effluent profile.

## 4.0 TEST PROCEDURE

This test procedure is partitioned into four primary subdivisions: system acceptance testing and calibration; Resorcinol-Formaldehyde test run preparation, load and elution; crystalline silicotitanate test run preparation, load and elution; system flush, decontamination, and waste management. Sampling procedures are a component of each of these subdivisions of the test and are provided as a fifth component of the test.

Laboratory instruction, as necessary will be developed to implement these laboratory 222-S procedures.

Initial pump calibration and subassembly resin conditioning will be conducted in non-radiologically contaminated conditions. Thus portions of test conduct through conditioning (§§ 4.2.2 and 4.3.2) will be conducted serially prior to hot cell entry of the assembly. Subsequent work will be conducted in the sequence specified by this document unless modified through change procedure (§ 5.0). All checksheets and data sheets are provided in Appendix A.

### 4.1 SYSTEM ACCEPTANCE TESTING AND CALIBRATIONS

The flow system apparatus (apparatus is defined as the entire system to include pumps, valving, plumbing, and columns without the exchange material) will be assembled in the nonradiological WHC laboratories in the 300 Area.

The valving, piping, tubing, columns, and exchange material will be the same that will be used in the hot cell in 222-S laboratory and shall follow the schematic as shown in Figure 1. Physical layout is depicted in Figure 2. The assembly of the apparatus in a cold area will also entail test run preparations of loading and conditioning of the exchange material in the respective columns as per manufacturer's specifications. With the exception of § 4.1.5, these acceptance testing and calibration activities will be nonradiological activities.

#### 4.1.1 Equipment Assembly

Equipment assembly consists of assembling the tubing, fittings, valves, pump and columns to meet the test needs. Specification of assembly is composed of feed assembly, RF test assembly, and CST test assembly. Fabrication of the substrate and basin of the test apparatus is conducted separately and prior to this assembly. Checksheets and assembly instructions are provided in Appendix A.

##### 4.1.1.1 Feed Assembly

The ion exchange columns will be loaded and the RF and CST resins conditioned before tank waste supernatant liquor challenge. The conditioning will be accomplished as outlined in sections 4.2.2 (RF) and 4.3.2 (CST).

The feed bottles, composed of polyethylene, for deionized water (T1), tank supernatant

liquor (T2), 2.5 M NaOH (caustic flush, T3), and 0.5 M $HNO_3$ (Cs eluant, T4) will share one line (L1) to the peristaltic pump (Figure 1). The discharge line from the peristaltic pump will connect to a three connection valve (V1). The line diameter selected for the pump mid-range has an ID of 0.89 mm. There will be two lines connected from V1 to the test columns, the A1 line to the RF columns and the B1 line to the CST columns.

No assembly is required for the feed tanks, thus feed assembly shall consist of assembling line L1 and mounting it through the pump and to valve V1. Use Sheet 1 for feed assembly directions, imagery, and checksheet.

#### 4.1.1.2 RF Test Assembly

The R-F test subassembly shall be completed using Sheet 2. The function of all valve configurations are detailed in Table 2.

The object of the subassembly is to provide feed liquors from the primary distribution valve V1 through distribution and isolation valves in configurations allowing flow and flush in each direction through the test and guard columns to sample and effluent points.

#### 4.1.1.3 CST Test Assembly

The CST test subassembly shall be completed using Sheet 3. The function of all valve configurations are detailed in Table 3.

The object of the subassembly is to provide feed liquors from the primary distribution valve V1 through distribution and isolation valves in configurations allowing flow and flush in each direction through the test and guard columns to sample and effluent points.

### 4.1.2 Leak Testing in Cold Environment

Using Figure 1 as a reference for flow streams and Figure 2 as reference for physical layout, the following steps will be carried out to cold test the system in Building 306E. Care should be taken to try and anticipate problems that may arise during the operation in a hot cell. This will allow a "fix it" mode to be undertaken in a non-radiation area. The resin will not be loaded in the columns for this portion of the test. Record conduct through indicated checksheets and datasheets.

The system will be challenged with deionized (DI) water for this phase. The connecting lines are the required ID and length and the valving is connected as indicated in Table 2 and Table 3. An additional objective of this portion of the test is system displacement volume measurement; for this reason, collected liquids and flow times are recorded.

#### 4.1.2.1 RF Subassembly Leak Testing

##### 4.1.2.1.1 Pump Set

**Table 2:** Valve Connection/Function for R-F Resin Columns

| Valve | After | Before | Function |
|---|---|---|---|
| V1 | Peristaltic Pump | V2 | Connects pump to Test bed A |
| | | V12 | Connects pump to Test bed B |
| V2 | V1 | V3 | Directs Feed to C1 |
| | | V6 | Directs eluant to C1 |
| | | V10 | Directs eluant to C2 |
| V3 | V2 | V4 | Directs Feed to C1 |
| | V4 | NA | Eluate from C2 and eluate samples |
| V4 | V3 | C1 | Column Isolation, Forward |
| | C1 | V3 | Column Isolation, Reverse |
| V5 | C1 | V6 | Column Isolation, Forward |
| | V6 | C1 | Column Isolation, Reverse |
| V6 | V5 | 60 min retention line w/ beta/gamma and V7 | Directs C1 effluent to V7 |
| | V2 | C1 | Directs eluant into C1 |
| V7 | V6 | V8 | C1 Flow to C2 |
| | | NA | C1 effluent samples |
| | V8 | NA | C2 eluate flow |
| V8 | V7 | C2 | Column Isolation, Forward |
| | C2 | V7 | Column Isolation, Reverse |
| V9 | C2 | V10 | Column Isolation, Forward |
| | V10 | C2 | Column Isolation, Reverse |
| V10 | V9 | NA | Directs effluent from C2 to receiver |
| | V2 | V9 | Directs eluant to C2 |
| | | NA | Line flush to waste |

NA = not applicable, flow to receiver.

The peristaltic pump does not have a digital readout or record. Calibration will be conducted with each flow rate change and recorded on Sheet 4 as required by each procedure. The flow path (Config. 1) for calibration will be:

**Config. 1**: R-F Pump Flow Rate Calibration Valve Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A2 |
| V3 | Open | A2 → V3 → A10 |

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED**

Use Sheet 4 to record and conduct this procedure.

Objective: 4 CV/hr = 31.4 mL/hr = 31.4 g/hr (DI $H_2O$)

- Tare receiver bottle (~ 100 mL capacity)
- Place line L1 into T1 (DI $H_2O$). Place Line A10 into tared receiver.
- Set valve positions to: Config. 1
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.
- Shut down the pump.
- Close and confirm closure of all valves.

4.1.2.1.2 Column Forward Tests

Use Sheet 5 to record and conduct this procedure.

- Tare receiver bottle (~ 100 mL capacity)
- Place line L1 into feed vessel T1, place line A8 into tared receiver
- Set valve positions to: Config. 2.
- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For the RF exchanger, 4 CV/hr are targeted which is approximately 0.5 mL/min or 31 mL/hr. Run in this configuration for 3 hours.
- If leaks are observed, adjust fittings as necessary to eliminate leaks. Record observations and leak repairs.
- After the appropriate time period for leak testing in a forward flow mode, shut down the pump. Record masses recovered and time interval on Sheet 5.

4.1.2.1.3 Column Reverse Flow Tests

**Table 3**: Valve Connection/Function for CST Resin Columns

| Valve | After | Before | Function |
|---|---|---|---|
| V1 | Peristaltic Pump | V2 | Connects pump to Test bed A |
| | | V12 | Connects pump to Test bed B |
| V12 | V1 | V13 | Directs Feed to C3 |
| | | V16 | Directs eluant to C3 |
| | | V20 | Directs eluant to C4 |
| V13 | V12 | V14 | Directs Feed to C3 |
| | V14 | NA | Eluate from C4 and eluate samples |
| V14 | V13 | C3 | Column Isolation, Forward |
| | C3 | V13 | Column Isolation, Reverse |
| V15 | C3 | V16 | Column Isolation, Forward |
| | V16 | C3 | Column Isolation, Reverse |
| V16 | V15 | 60 min retention line w/ beta/gamma and V17 | Directs C3 effluent to V17 |
| | V12 | C3 | Directs eluant into C1 |
| V17 | V16 | V18 | C3 Flow to C4 |
| | | NA | C3 effluent samples |
| | V18 | NA | C4 eluate flow |
| V18 | V17 | C4 | Column Isolation, Forward |
| | C4 | V7 | Column Isolation, Reverse |
| V19 | C4 | V10 | Column Isolation, Forward |
| | V20 | C4 | Column Isolation, Reverse |
| V20 | V19 | NA | Directs effluent from C4 to receiver |
| | V12 | V9 | Directs eluant to C4 |
| | | NA | Line flush to waste |

NA = Not Applicable, flow to receiver vessel

4.1.2.1.3.1 Column 1 Elution Leak Test

Use Sheet 6 to record and conduct this procedure.

- Tare receiver bottle ( ~ 100 mL capacity)

**Config. 2:** R-F Subassembly Forward Feed Valving Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A2 |
| V3 | Open | A2 → V3 → A3 |
| V4 | Open | A3 → V4 → C1 |
| V5 | Open | C1 → V5 → A4 |
| V6 | Open | A4 → V6 → A5 |
| V7 | Open | A5 → V7 → A6 |
| V8 | Open | A6 → V8 → C2 |
| V9 | Open | C2 → V9 → A7 |
| V10 | Open | A7 → V10 → A8 |

- Place line L1 into feed vessel T1, place line A10 into tared receiver
- Set valve positions to: Config. 3:

**Config. 3:** R-F Subassembly Column 1 Reverse Feed Valving Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A9 |
| V6 | Open | A9 → V6 → A4 |
| V5 | Open | A4 → V5 → C1 |
| V4 | Open | C1 → V4 → A3 |
| V3 | Open | A3 → V3 → A10 |

- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For the RF exchanger, 4 CV/hr are targeted which is approximately 0.5 mL/min or 31 mL/hr. Run in this configuration for 1 hour.
- If leaks are observed, adjust fittings as necessary to eliminate leaks. Record observations and leak repairs.
- After the appropriate time period for leak testing in a reverse flow mode, shut down

the pump. Record masses recovered and time interval on Sheet 6.

4.1.2.1.3.2 Column 2 Elution Leak Test

Use Sheet 7 to record and conduct this procedure.

- Tare receiver bottle (~ 100 mL capacity)
- Place line L1 in feed vessel T1, place line A12 into tared receiver
- Set valves positions to: Config. 4:

**Config. 4**: R-F Subassembly Column 2 Reverse Feed Valving Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A11 |
| V10 | Open | A11 → V10 → A7 |
| V9 | Open | A7 → V9 → C2 |
| V8 | Open | C2 → V8 → A6 |
| V7 | Open | A6 → V7 → A12 |

- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For the RF exchanger, 4 CV/hr are targeted which is approximately 0.5 mL/min or 31 mL/hr. Run in this configuration for 1 hour.
- If leaks are observed, adjust fittings as necessary to eliminate leaks. Record observations and leak repairs.
- After the appropriate time period for leak testing in a reverse flow mode, shut down the pump. Record masses recovered and time interval on Sheet 7.

#### 4.1.2.2 CST Subassembly Leak Test

##### 4.1.2.2.1 Pump Set

The peristaltic pump does not have a digital readout or record. Calibration will be conducted with each flow rate change and recorded on Sheet 11 as required by each procedure. The flow path (Config. 5) for calibration will be:

**Config. 5**: CST Pump Flow Rate Calibration Valve Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B2 |
| V13 | Open | B2 → V13 → B10 |

Use Sheet 11 to record and conduct this procedure.

Objective: 6 CV/hr = 47.1 mL/hr = 47.1 g/hr (DI $H_2O$)

- Tare receiver bottle (~ 100 mL capacity)
- Place line L1 into T1. Place Line B10 into tared receiver.
- Set valve positions to: Config. 5
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.
- Shut down the pump.
- Close and confirm closure of all valves.

##### 4.1.2.2.2 Column Forward Tests

Use Sheet 12 to record and conduct this procedure.

- Tare receiver bottle (100 mL < receiver < 250 mL)
- Place L1 into feed vessel T1, place line B8 into tared receiver
- Set valve positions to: Config. 6.
- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For the CST exchanger, 6 CV/hr are targeted which is approximately 0.8 mL/min or 47.1 mL/hr. Run in this configuration for 3 hours.
- If leaks are observed, adjust fittings as necessary to eliminate leaks. Record observations and leak repairs.

**Config. 6**: CST Subassembly Forward Feed Valving Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B2 |
| V13 | Open | B2 → V13 → B3 |
| V14 | Open | B3 → V14 → C3 |
| V15 | Open | C3 → V15 → B4 |
| V16 | Open | B4 → V16 → B5 |
| V17 | Open | B5 → V17 → B6 |
| V18 | Open | B6 → V18 → C4 |
| V19 | Open | C4 → V19 → B7 |
| V20 | Open | B7 → V20 → B8 |

- After the appropriate time period for leak testing in a reverse flow mode, shut down the pump. Record masses recovered and time interval on Sheet 12.

#### 4.1.2.2.3 Column Reverse Flow Tests

##### 4.1.2.2.3.1 Column 3 Elution Leak Test

Use Sheet 13 to record and conduct this procedure.

- Tare receiver bottle (~100 mL)
- Place line L1 in feed vessel T1, place line B10 into tared receiver
- Set valve positions to: Config. 7:

**Config. 7**: CST Subassembly Column 3 Reverse Feed Valving Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B9 |
| V16 | Open | B9 → V16 → B4 |
| V15 | Open | B4 → V15 → C3 |
| V14 | Open | C3 → V14 → B3 |
| V13 | Open | B3 → V13 → B10 |

- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For the CST exchanger, 6 CV/hr are targeted which is approximately 0.8 mL/min or 47.1 mL/hr. Run in this configuration for 1 hour.
- If leaks are observed, adjust fittings as necessary to eliminate leaks. Record observations and leak repairs.
- After the appropriate time period for leak testing in a reverse flow mode, shut down the pump. Record masses recovered and time interval on Sheet 13.

##### 4.1.2.2.3.2 Column 4 Elution Leak Test

Use Sheet 14 to record and conduct this procedure.

- Tare receiver bottle (~100 mL)
- Place line L1 into feed vessel T1, place line B12 into tared receiver
- Set valve positions to: Config. 8:
- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For the CST exchanger, 6 CV/hr are targeted which is approximately 0.8 mL/min or 47.1 mL/hr. Run in this configuration for 1 hour.
- If leaks are observed, adjust fittings as necessary to eliminate leaks. Record

**Config. 8**: CST Subassembly Column 4 Reverse Feed Valving Configuration

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B11 |
| V20 | Open | B11 → V20 → B7 |
| V19 | Open | B7 → V19 → C4 |
| V18 | Open | C4 → V18 → B6 |
| V17 | Open | B6 → V17 → B12 |

observations and leak repairs.

- After the appropriate time period for leak testing in a reverse flow mode, shut down the pump. Record masses recovered and time interval on Sheet 14.

#### 4.1.3 Reagent Preparation

The following reagents shall be prepared by the standards laboratory and delivered to 222-S for hot cell entry as needed:

| | | |
|---|---|---|
| NaOH, *aq.* | 2.5 M | 1.0 L |
| HF, *aq.* | 1.0 M | 100 mL |
| HCl, *aq.* | 2.0 M | 200 mL |
| $HNO_3$, *aq.* | 3.0 M | 200 mL |
| $HNO_3$, *aq.* | 0.5 M | 750 mL |

Specifications and contaminants of the reagents shall be identified for inclusion in test data analysis.

#### 4.1.4 Exchange Material Dissolution

Prior to loading the columns with resin, a qualitative evaluation of the dissolution of the resin must be carried out. These dissolutions will be conducted in 222-S nonradiological facilities in a hood with appropriate personal protective equipment. MSDS review is required.

##### 4.1.4.1 R-F Resin

Use Sheet 18 to record and conduct this procedure.

- In an evaporating dish, weigh out 1g of R-F resin
- Add 2 mL of 3 M $HNO_3$ acid.
- Allow ~1 hour for digestion, do not heat above 60°C

- Transfer to 100 mL volumetric flask and dilute with DI water to the mark.
- Subsample ~5 mL and retain, label the subsample as SRF1.
- Record all observations on Sheet 18.

#### 4.1.4.2 CST Resin

Use Sheet 19 to record and conduct this procedure.

- In an evaporating dish, weigh out 0.1 g of CST resin
- Add 3 mL (+/- 0.5 mL) DI water
- Add about 1 mL of HCl (2 M)
- Add 0.5 mL of HF (1 M)
- Heat gently to dissolve.
- Transfer to 100 mL volumetric flask and dilute with DI water to the mark.
- Subsample ~5 mL and retain, label the subsample as SCST1.
- Record all observations on Sheet 19.

**NOTE: Due to the proprietary nature of the resin substrate and binder, analytical information from sample analyses of this material will not be publicly released.**

### 4.1.5 Beta-Gamma Probe Calibration

The calibration of the beta/gamma probe will be carried out using 222-S laboratory sources, for a range of 9.25 E9 Bq/L to 9.25 E5 Bq/L (0.25 Ci/L $^{137}Cs$ to 25 $\mu$Ci/L). The source configuration will be Tygon tubing, 4.76 mm ID, 9.52 mm OD. The tubing standard should be about 10 cm in length. The length of the tubing need only be measured approximately, since previous work (Beck *et al.* 1996) has shown that the gamma detector is sensitive to cesium (barium) gammas only in a small cone (6 cm diameter at sample) below the probe. The lower level standard may not be detectable on the time scale used during the tests, as it represents about 1650 disintegrations per minute for the entire standard. Calibration responses from prepared standards will be entered into an appropriate laboratory notebook.

## 4.2 R-F TEST PREPARATION AND RUN

This portion of the test plan is intended to provide explicit instructions and expectations of the conduct of ion exchange with the apparatus using Resorcinol-Formaldehyde. The expectations are that the 50% breakthrough will be at approximately 62-65 column volumes ($\lambda_{50\%}$ = 62-65 CV), and that the run will be conducted for 80 column volumes, sampled on the first and every 5 column volumes.

### 4.2.1 Pump Flow Set

Use Sheet 4 to record and conduct this procedure.

Object 10 CV/hr = 79.6 mL/hr = 79.6 g/hr DI $H_2O$

- Tare receiver bottle ( ~ 100 mL capacity)
- Set valve positions to: Config. 1
- Place line L1 into T1 (DI $H_2O$). Place Line A10 into tared receiver.
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.
- Shut down the pump.
- Close and confirm closure of all valves.

#### 4.2.2 Exchange Column Preparation

Both the test column, C1, and the guard column, C2, will be charged with R-F resin and conditioned outside of the hot cell. After conditioning and pump calibration, the columns and system will be transferred into the hot cell (**NOTE: SIMILAR SERIAL WORK IS REQUIRED FOR THE CST SUBASSEMBLY PRIOR TO HOT CELL PLACEMENT**).

The resin received is in the potassium form, it must be conditioned by:

- 10 CV/h of 0.5 M $HNO_3$, 1 hour, followed by
- 10 CV/h of DI water, 1 hour followed by
- 10 CV/h of 2.5 M NaOH, 1 hour.

Use Sheet 24 to prepare and measure the bed density of the resin.

Use Sheet 5(s) to record and conduct these column procedural activities.

- Place Line L1 into T4 (0.5 M $HNO_3$).
- Place Line A8 into flush container
- Place System Valving in Config. 2. Confirm and record valving alignment.
- Turn on pump and run for one hour. Record time on/off, flowrate and feed source.
- Rinse feed end of L1 with $H_2O$, place Line L1 into T1 (DI Water)
- Turn on pump and run for one hour. Record time on/off, flowrate and feed source.
- Place Line L1 into T3 (2.5 M NaOH)
- Turn on pump and run for one hour. Record time on/off, flowrate and feed source.
- Close and confirm closure of all valves.

The total time to condition the resin will be 3 to 4 hours.

#### 4.2.3 R-F Apparatus Pump Curve Acquisition

Because of the varying pressure drops across the apparatus dependent upon sampling location, pump curves will be developed with the loaded columns while feeding 2.5 M NaOH. This fluid, used in resin conditioning, is also expected to well represent the density and viscosity of waste feed. Following the pump curve acquisition, the flow settings configuration (Config. 1) may be used to ensure adequate flow at the apparatus terminus (line

A8).

Use Sheet 20 to record and conduct this procedure

- Tare receiver bottle (~250 mL)
- Place line L1 into T3. Place line A10 into tared receiver
- Set valve positions to: Config. 2
- Close Valve V10
- Open Valve V3 to Line A10
- Set pump knob flow (low ~15%)
- Turn on pump for 20 minutes, record time on.
- Turn off pump, record time off, weigh receiver.

- Reset Valve V3 to line A3.
- Place A12 into tared receiver.
- Open Valve V7 to Line A12
- Turn on pump for 20 minutes, record time on.
- Turn off pump, record time off, weigh receiver.

- Reset Valve V7 to Line A6
- Place A8 into tared receiver.
- Open Valve V10 to line A8
- Turn on pump for 20 minutes, record time on.
- Turn off pump, close all valves, record time off, weigh receiver.

Repeat this procedure, for a total of at least five point calibration, at flow settings of approximately 40%, 50%, 60% and 75% of pump knob range.

#### 4.2.4 R-F Waste Feed Preparation and Analysis

The waste feed will be composed of a composite of drainable supernatant liquor from samples taken from Hanford Double-Shell tank 241-AW-101 during the 1996 core drilling operations which commenced in February 1996 (Benar 1996). The waste feed is of approximately 10 M $Na^+$ and 5 M $OH^-$. The waste feed will be diluted to a target of approximately 5 M Na with water. Analysis of the waste composition for the feed will be carried out by thermal ionization mass spectrometry and inductively coupled plasma at Pacific Northwest National Laboratory (PNNL) under separate work order.

It is anticipated that the supernatant liquor will be delivered to the 1F Hot Cell in 125 mL bottles, any undiluted material will remain stoppered. The waste material may be delivered to the hotcell at unspecified times. No time constraint for this work applies with the exception that test run (§ 4.2.6.4) cannot proceed unless the material is prepared.

Use Sheet 8 to conduct and record this procedure.

- Take an aliquot of the waste material and submit it for density analysis.

- By mass, add 350 mL equivalent of waste liquor from waste sample bottles (550 g at SPG = 1.56) of waste liquor into 1L mixing vessel. Record source material history (core, segment, bottle) on Sheet 8.
- Add DI water to mixing vessel in aliquots to equal waste mass divided by density (~350 g). Record actual mass added.
- Mix on magnetic stirring plate.
- Allow to settle, and decant clear liquor into feed tank T2.
- Tare centrifuge cones.
- Centrifuge the remaining liquor, and decant into feed tank T2. Record observations of liquor clarity, color and of any suspended solids.
- Upon completion of waste material addition to T2, remove an aliquot (~5 mL) from T2 into prelabeled sample vial F1.
- Take an additional aliquot of the waste feed from T2 and submit it for density analysis.
- Weigh all centrifuge cones for summation of solids mass.
- Place a sample (~1 g) of centrifuge solids in prelabeled sample vial FS1

#### 4.2.5 System Flush

After the system is assembled in the hot cell, begin the test by setting pump rates and flushing the system.

Use Sheet 4 to set pump and Sheet 5 to record and conduct the system flush.

Object 4 CV/hr = 31.4 mL/hr = 31.4 g/hr (DI $H_2O$) =

Set Pump:

- Tare receiver bottle (~100 mL capacity)
- Place line L1 into T1 (DI Water)
- Place Line A10 into receiver
- Set valve positions to: Config. 1. Confirm and record valving alignment.
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.
- Shut down pump

Flush System:

- Place Line A8 into tared collection vessel
- Place Line L1 into T3 (2.5 M NaOH)
- Place System Valving in Config. 2. Confirm and record valving alignment.
- Turn on pump and run for 100 minutes. Record time on/off, flowrate and feed source.
- Close and confirm closure of all valves.

The 2.5 M NaOH from the conditioning phase (§ 4.2.2) will have been flushed through the column and testing for $^{137}Cs$ removal may begin.

#### 4.2.6 Cesium Removal Test Run

Although it is anticipated that breakthrough ($^{137}Cs$ $C/C_0$ = 0.5) will occur at 62 to 65 CV, the test run will schedule use of 80 CV to ensure data past breakthrough will be obtained. The test will proceed until $C/C_0$ = 0.6 - 0.75 and will be discontinued by direction of the lead engineer or chemist.

##### 4.2.6.1 Beta-Gamma Detector Data Logging

During the test run, the beta-gamma detector will be continuously monitoring the effluent line and recording data on an IBM[3] compatible computer using the GammaVision[4] program. The breakthrough of $^{137}Cs$ will be indicated by the increase in gamma response over time for the window of ~610 - 680 KeV. The detector logging should commence with initial feed startup (§ 4.2.6.4) and discontinue with final sample acquisition from the flow stream following the test column.

##### 4.2.6.2 Primary Column Sampling

All samples will be recorded with column number, date time/group, sample sequence number (C1E-1, C1E-2, ...C1E-n). The leading front of the treated effluent stream is anticipated at 1 hr 32 min after pump start. Samples will be acquired for a period of 20 hours following this front passage of the sampling point.

Samples will be acquired as detailed in Table 4.

Sampling shall be conducted using Sheet 9 for sample acquisition data and Sheet 10 for process parameters of the sample.

- Record sample number, sampling time, and beta-gamma probe response on Sheets 9 and 10 as required.
- Tare flush vial
- Tare sample vial and stopper
- Place line A12 into flush vial in base holder.
- Redirect valve V7 to allow line A12 effluent to enter vial to flush the line (~2 mL)
- Return valve V7 to flow configuration (Config. 2)
- Remove line A12 from flush vial, move flush vial aside and place sample vial in base holder.
- Place line A12 into sample vial.
- Redirect valve V7 to allow effluent to enter sample vial to volume of approximately 5 mL. Time sample acquisition time for pump calibration confirmation.
- Return valve V7 to flow configuration (Config. 2)

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[3] IBM is a trademark of International Business Machines, Inc., White Plains, NY.

[4] GammaVision is a trademark of EG&G ORTEC, Inc., Oak Ridge, TN.

**Table 4:** R-F Test Run Primary Column Sampling

| Sample | Projected Run Time |
|---|---|
| C1E-1 | 1 hr 32 min |
| C1E-2 | 2 hr 47 min |
| C1E-3 | 4 hr 2 min |
| C1E-4 | 5 hr 17 min |
| C1E-5 | 6 hr 32 min |
| C1E-6 | 7 hr 47 min |
| C1E-7 | 9 hr 2 min |
| C1E-8 | 10 hr 17 min |
| C1E-9 | 11 hr 32 min |
| C1E-10 | 12 hr 47 min |
| C1E-11 | 14 hr 2 min |
| C1E-12 | 15 hr 17 min |
| C1E-13 | 16 hr 32 min |
| C1E-14 | 17 hr 47 min |
| C1E-15 | 19 hr 2 min |
| C1E-16 | 20 hr 17 min |
| C1E-17 | 21 hr 32 min |

- Remove line A12 from vial and stopper sample vial.
- Close valve V7 and allow residual fluid in line A12 to fall into flush vial.
- Weigh stoppered sample vial and record.
- Move sample vial to sample retention/rack pending transfer out of cell for analysis.
- Weigh flush vial and record.
- Calculate flow rate from net sample mass and flow time. Read system flow from pump curve.

---

**DURING THE TEST RUN, ALL SAMPLE POINTS (LINES A8, A10, A12) WILL HAVE A CATCH CONTAINER TO CONTAIN SPILLS.**

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED.**

If the system flow rate varies from the target flow by > ±10%, reset the pump through change procedures in § 5.0. It must be recalled that system target flow rates are established as rates of effluent following the guard column. With the guard column out of the flow path during sampling, it is expected that flows from the sample point will be slightly higher than they would be with the added pressure drop across the guard column. Hence, the pump curves of Sheet 20 are required to appropriately monitor flow.

#### 4.2.6.3 Effluent Sampling

The sampling from the guard column (C2), will be a total composite sample. At test initiation place a 2L container under V10/A8. This volume should be sufficient to contain all effluent. At the end of the run, take an aliquot of approximately 5 mL for analysis. Identify the sample as C2E-1.

#### 4.2.6.4 Run Execution

Use Sheet 5 to record and conduct this procedure.

Confirm or do:
Objective: 4 CV/hr = 31.4 mL/hr

- Tare receiver bottle (~2 L)
- Place Line A8 into tared receiver
- Rinse line L1 at feed end with $H_2O$
- Place line L1 into T2 (Waste Feed)
- Establish valving for Config. 2. Confirm and record valving alignment on Sheet 5.
- Measure and record feed temperature (°C)
- Turn on pump and initiate test run clock. Record test initiation on Sheet 5.
- Acquire data and samples as directed above.
- Continue flow until $\beta$-$\gamma$ probe response is in range of ~60-75% $C/C_0$.
- Immediately following last primary column sample, shut down pump, and take the composite effluent sample from the receiver below A8.
- Close and confirm closure of all valves.

### 4.2.7 System Flush

After the feed solution has run through the system, the system will be flushed with 2.5 M NaOH for 10 CV at 4 CV/h (2.5 hours). Allow the flush to run to a suitable container (1L).

Use Sheet 5 to record and conduct this procedure.

---

**DURING THE TEST RUN, ALL SAMPLE POINTS (LINES A8, A10, A12) WILL HAVE A CATCH CONTAINER TO CONTAIN SPILLS.**

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED.**

- Wash exterior of Line L1 with $H_2O$, place L1 in T3 (2.5 M NaOH)
- Tare Flush receiver.
- Place A8 into flush waste receiver (1L)
- Establish valving for Config. 2. Confirm and record valving alignment.
- Turn on pump for 2.5 hours. Record start time.
- Turn off pump. Record stop time
- Close and confirm closure of all valves.

No samples will be taken of this waste. Proceed to column elution.

#### 4.2.8 Column Elution

##### 4.2.8.1 Pump Flow Set

Use Sheet 4 to record and conduct this procedure.

Objective: 2 CV/hr = 15.8 mL/hr

- Place line L1 into T1 (DI Water)
- Place Line A10 into graduated cylinder
- Set valve positions to: Config. 1. Confirm and record valving alignment.
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.
- Shut down pump
- Close and confirm closure of all valves.

##### 4.2.8.2 Primary Column Elution

After the columns (C1 and C2) are flushed with caustic, the columns will be eluted using 5CV $H_2O$ then 10CV 0.5 M $HNO_3$.

Use Sheet(s) 6 to record and conduct this procedure.
Column rinse:

- Rinse line L1 (feed end) with $H_2O$
- Place line L1 in T1
- Place line A10 into eluate receiver (~250 mL)
- Set valve positions to: Config. 3
- Turn on pump for 2½ hours. Record time on.
- Turn off pump. Record time off.

Column elution:

- Place line L1 in T4
- Place line A10 into eluate receiver
- Set valve positions to: Config. 3. Confirm and record valving alignment.
- Turn on pump for 5 hours. Record time on.
- Turn off pump. Record time off.

- Pipette two 5 mL aliquots of the composite eluate into sample vials labeled C1W-1 and C1W-2.

Following this activity, prepare to conduct the elution of C2.

#### 4.2.8.3 Secondary Column Elution

The discharge line will terminate in a container (eluate receiver) with a suitable volume to collect the entire eluate. No samples are to be taken from the Column C2 eluate.

Use Sheet(s) 7 to record and conduct this procedure.

Column rinse:

- Rinse line L1 (feed end) with $H_2O$
- Place line L1 in T1
- Place line A12 into eluate receiver (~250 mL)
- Set valve positions to: Config. 4
- Turn on pump for 2½ hours. Record time on.
- Turn off pump. Record time off.

Column elution:

- Place line L1 in T4
- Place line A12 into eluate receiver
- Set valve positions to: Config. 4. Confirm and record valving alignment.
- Turn on pump for 5 hours. Record time on.
- Turn off pump. Record time off.

### 4.3 CST TEST PREPARATION AND RUN

This portion of the test plan is intended to provide explicit instructions and expectations of the conduct of ion exchange with the apparatus using crystalline silicotitanate. The expectations are that the 50% breakthrough will be at approximately 750 column volumes ($\lambda_{50\%}$ = 750 CV). The run is expected to be conducted for up to 1000 column volumes, sampled every 100 column volumes for the first 500 CV (5 samples) and every 25 CV thereafter (20 samples).

#### 4.3.1 Pump Flow Set

Objective: 10 CV/hr = 78.5 mL/hr

- Place line L1 into T1 (DI Water)
- Place Line A10 into graduated cylinder
- Set valve positions to: Config. 5. Confirm and record valving alignment.
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.

- Shut down pump
- Close and confirm closure of all valves.

#### 4.3.2 Exchange Column Preparation

Both the test column, C3, and the guard column, C4, will be charged with CST resin and conditioned outside of the hot cell. After conditioning and pump calibration, the columns and system will be transferred into the hot cell (**NOTE: SIMILAR SERIAL WORK IS REQUIRED FOR THE R-F SUBASSEMBLY PRIOR TO HOT CELL PLACEMENT**). To prepare the CST resin, manufacturer's specifications will be met with fines removal and sodium hydroxide conditioning. These specifications are not incorporated herein as they are business confidential instructions, but are provided to test personnel.

Use Sheet 25 to prepare and measure the bed density of the resin.

Following this conditioning and the following pump calibration, and that of 4.2.2, the test assembly will be prepared for hot cell placement.

#### 4.3.3 CST Apparatus Pump Curve Acquisition

Because of the varying pressure drops across the apparatus dependent upon sampling location, pump curves will be developed with the loaded columns while feeding 2.5 M NaOH. This fluid, used in resin conditioning, is also expected to well represent the density and viscosity of waste feed. Following the pump curve acquisition, the flow settings configuration (Config. 5) may be used to ensure adequate flow at the apparatus terminus (line B8).

Use Sheet 21 to record and conduct this procedure

- Tare receiver bottle (~250 mL)
- Place line L1 into T3. Place line B10 into tared receiver
- Set valve positions to: Config. 6
- Close Valve V20
- Open Valve V13 to Line B10
- Set pump knob flow (low ~15%)
- Turn on pump for 20 minutes, record time on.
- Turn off pump, record time off, weigh receiver.

- Reset Valve V13 to line B3.
- Place B12 into tared receiver.
- Open Valve V17 to Line B12
- Turn on pump for 20 minutes, record time on.
- Turn off pump, record time off, weigh receiver.

- Reset Valve V17 to Line B6
- Place B8 into tared receiver.

- Open Valve V10 to line A8
- Turn on pump for 20 minutes, record time on.
- Turn off pump, close all valves, record time off, weigh receiver.

Repeat this procedure, for a total of at least five point calibration, at flow settings of approximately 40%, 50%, 60% and 75% of pump knob range.

#### 4.3.4 CST Waste Feed Preparation and Analysis

The waste feed will be composed of a composite of drainable supernatant liquor from samples taken from Hanford Double-Shell tank 241-AW-101 during the 1996 core drilling operations which commenced in February 1996 (Benar 1996). The waste feed is of approximately 10 M $Na^+$ and 5 M $OH^-$. The waste feed will be diluted to a target of approximately 5 M Na with water. Analysis of the waste composition for the feed will be carried out by thermal ionization mass spectrometry and inductively coupled plasma at PNNL laboratories under separate work order.

It is anticipated that the supernatant liquor will be delivered to the 1F Hot Cell in 125 mL bottles, any undiluted material will remain stoppered. The waste material may be delivered to the hotcell at unspecified times. No time constraint for this work applies with the exception that test run (§ 4.3.6.4) cannot proceed unless the material is prepared.

This feed stock will be prepared batchwise and will initially require one or two batches, with additional batches to follow as the test proceeds. A total of approximately eight batches will be prepared in this manner.

Use Sheet(s) 15 to record and conduct this procedure.

For each batch:

- By mass, add 500 mL equivalent of waste liquor from waste sample bottles (780 g at SPG = 1.56) into 2L mixing vessel. Density is acquired in procedure § 4.2.4. Record source material history (core, segment, bottle) on Sheet 15.
- Add DI water to mixing vessel in aliquots to equal waste mass divided by density (~500 g). Record actual mass added.
- Mix on magnetic stirring plate.
- Allow to settle, and decant clear liquor into feed tank T2.
- Tare centrifuge cones
- Centrifuge the remaining liquor, and decant into feed tank T2. Record observations of liquor clarity, color and of any suspended solids.
- Upon completion of waste material addition to T2, remove an aliquot (~5 mL) from T2 into prelabeled sample vial F2 (F3..F8 for additional batches).
- Record time of each new transfer into T2.
- Weigh all centrifuge cones for summation of solids mass.

To minimize waste with sequential batches continue use of centrifuge cones through no more than ⅓ height of cone.

- Composite centrifuge solids in 250-500 mL vial when cones exceed ⅓ fill height.

- Following last batch of feed preparation take two composite samples of centrifuged solids (~ 1g) and place them in sample vials FS2 and FS3.

#### 4.3.5 System Flush

After the system is assembled in the hot cell, begin the test by setting pump rates and flushing the system.

Use Sheet 11 to set pump and Sheet 12 to record and conduct the system flush.

Objective: 6 CV/hr = 47.1 mL/hr

Set Pump:

- Tare receiver bottle (~ 100 mL capacity)
- Place line L1 into T1 (DI Water)
- Place Line A10 into tared receiver
- Set valve positions to: Config. 5. Confirm and record valving alignment.
- Set flow rate - turn on pump, measure for 15 min. Check against objective, record results.
- Reset to get to objective as necessary, repeat.
- When at objective, confirm flow with two more test periods.
- Shut down pump

Flush:

- Place Line B8 into tared receiver
- Place Line L1 into T3 (2.5 M NaOH)
- Place System Valving in Config. 6. Confirm and record valving alignment.
- Turn on pump and run for 100 minutes. Record time on/off, flowrate and feed source.
- Close and confirm closure of all valves.

The 2.5 M NaOH from the conditioning phase (§ 4.3.2) will have been flushed through the column and testing for $^{137}Cs$ removal may begin.

#### 4.3.6 Cesium Removal Test Run

Although it is anticipated that breakthrough ($^{137}Cs$ $C/C_0 = 0.5$) will occur at 750 to 800 CV, the test run will schedule use of 1000 CV to ensure data past breakthrough will be obtained. The test will proceed until $C/C_0 = 0.6 - 0.75$ and will be discontinued by direction of the lead engineer or chemist.

#### 4.3.6.1 Beta-Gamma Detector Data Logging

During the test run, the beta-gamma detector will be continuously monitoring the effluent line and recording data on an IBM compatible computer using the GammaVision program. The breakthrough of $^{137}Cs$ will be indicated by the increase in gamma response over time for the window of ~610 - 680 KeV. The detector logging should commence with initial feed startup (§ 4.3.6.4) and discontinue with final sample acquisition from the flow stream following the test column.

The beta-gamma probe must provide an additional function for this component of the test. If the response from the beta-gamma probe indicates $C/C_0 < 0.10$ prior to 500 CV, the sample regime will accelerate to one sample per 25 CV.

#### 4.3.6.2 Primary Column Sampling

All samples will be recorded with column number, date time/group, sample sequence number (C3E-1, C3E-2, ...C3E-n).

It is anticipated that the CST resin will be exposed to 1000 CV before the test is stopped. Samples of 5 mL each will be taken at Valve 17. At 1000 CV with a flow rate of 6 CV/h, the test will run for 166 hours. The first sample will be taken at 1 hr 30 min, representing the effluent stream front. Additional samples will be taken every 16 hours and 40 minutes (100 CV) through 500 CV. Subsequent samples will be taken every 4 hours and 10 minutes (25 CV) through the remainder of the test. Expected samples total 27 and are detailed on Table 5. Should the on-line beta gamma detector indicate that the cesium $C/C_0$ exceeds 10%, the sample regime shall immediately (nearest 25 CV increment) switch to the accelerated sampling structure of Table 5. Change procedures of § 5.0 will be implemented to generate new sampling runtimes in the event of accelerated sampling needs.

Samples shall be acquired as detailed in Table 5.

Sampling shall be conducted using Sheet 16 for sample acquisition data and Sheet 17 for process parameters of the sample.

- Record sample number, sampling time, and beta-gamma probe response on Sheets 16 and 17 as required.
- Tare flush vial
- Tare sample vial and stopper
- Place line B12 into flush vial in base holder.

---

**DURING THE TEST RUN, ALL SAMPLE POINTS (LINES B8, B10, B12) WILL HAVE A CATCH CONTAINER TO CONTAIN SPILLS.**

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED**

**Table 5:** CST Test Run Primary Column Sampling

| Sample | Projected Run Time |
|---|---|
| C3E-1 | 1 hr 30 min |
| C3E-2 | 68 hr 10 min |
| C3E-3 | 72 hr 20 min |
| C3E-4 | 76 hr 30 min |
| C3E-5 | 80 hr 40 min |
| C3E-6 | 84 hr 50 min |
| C3E-7 | 89 hr 0 min |
| C3E-8 | 93 hr 10 min |
| C3E-9 | 97 hr 20 min |
| C3E-10 | 101 hr 30 min |
| C3E-11 | 105 hr 40 min |
| C3E-12 | 109 hr 50 min |
| C3E-13 | 114 hr 0 min |
| C3E-14 | 118 hr 10 min |
| C3E-15 | 122 hr 20 min |
| C3E-16 | 126 hr 30 min |
| C3E-17 | 130 hr 40 min |
| C3E-18 | 134 hr 50 min |
| C3E-19 | 139 hr 0 min |
| C3E-20 | 143 hr 10 min |
| C3E-21 | 147 hr 20 min |
| C3E-22 | 151 hr 30 min |
| C3E-23 | 155 hr 40 min |
| C3E-24 | 159 hr 50 min |
| C3E-25 | 164 hr 0 min |
| C3E-26 | 168 hr 10 min |
| C3E-27 | 172 hr 20 min |

- Place line B12 into flush vial in base holder.
- Redirect valve V17 to allow line B12 effluent to enter vial to flush the line (~2 mL)
- Return valve V17 to flow configuration (Config. 6)
- Remove line B12 from flush vial, move flush vial aside and place sample vial in base

---

**DURING THE TEST RUN, ALL SAMPLE POINTS (LINES B8, B10, B12) WILL HAVE A CATCH CONTAINER TO CONTAIN SPILLS.**

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED**

holder.

- Place line B12 into sample vial.
- Redirect valve V17 to allow effluent to enter sample vial to volume of approximately 5 mL. Time sample acquisition time for pump calibration confirmation.
- Return valve V17 to flow configuration (Config. 6)
- Remove line B12 from vial and stopper sample vial.
- Close valve V17 and allow residual fluid in line A12 to fall into flush vial.
- Weigh stoppered sample vial and record.
- Move sample vial to sample retention/rack pending transfer out of cell for analysis.
- Weigh flush vial and record.
- Calculate flow rate from net sample mass and flow time. Read system flow from pump curve.

If the system flow rate varies from the target flow by $> \pm 10\%$, reset the pump through change procedures in § 5.0. It must be recalled that system target flow rates are established as rates of effluent following the guard column. With the guard column out of the flow path during sampling, it is expected that flows from the sample point will be slightly higher than they would be with the added pressure drop across the guard column. Hence, the pump curves of Sheet 21 are required to appropriately monitor flow.

#### 4.3.6.3 Effluent Sampling

The sampling from the guard column (C4), will be a total composite sample. At test initiation place a 2L container under V20/B8. This volume should be sufficient for approximately 40 hours of operation. Replace this container when it has reached 75% of capacity or will do so by the next sampling effort. At the end of the run, take an aliquot of approximately 5 mL from each container. Identify the samples as C4E-1 to C4E-n, where n is the container number (should not exceed 6).

#### 4.3.6.4 Run Execution

Use Sheet 12 to record and conduct this procedure

Confirm or do:
Objective: 6 CV/hr = 47.1 mL/hr

- Tare receiver bottle ($\sim$2 L)
- Rinse line L1 at feed end with $H_2O$
- Place Line B8 into 2L collection vessel
- Place line L1 into T2 (Waste Feed)
- Confirm valving for Config. 6. Confirm and record valving alignment on Sheet 12.

---

**DURING THE TEST RUN, ALL SAMPLE POINTS (LINES B8, B10, B12) WILL HAVE A CATCH CONTAINER TO CONTAIN SPILLS.**

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED**

- Measure and record feed temperature (°C)
- Turn on pump and initiate test run clock. Record test initiation on Sheet 12.
- Acquire data and samples as directed above.
- Continue flow until $\beta$-$\gamma$ probe response is in range of ~60-75% $C/C_0$.
- Immediately following last sample primary column sample, shut down pump, and take the composite effluent sample from the received below B8.
- Close and confirm closure of all valves.

#### 4.3.7 System Flush

After the feed solution has run through the system at 1000 CV, the system will be flushed with 2.5 M NaOH for 10 CV at 6 CV/h (1.66 hours). Allow the flush to run to a suitable container (1L).

Use Sheet 12 to record and conduct this procedure.

- Wash exterior of Line L1 with $H_2O$, place L1 in T3 (2.5 M NaOH)
- Place B8 into flush waste receiver (1L)
- Establish valving for Config. 6. Confirm and record valving alignment.
- Turn on pump for 2.5 hours. Record start time.
- Turn off pump. Record stop time
- Close and confirm closure of all valves.

No samples will be taken of this waste. Proceed to column elution.

#### 4.3.8 Column Elution

This material will not be eluted under present planning. If elution becomes necessary, elution procedures similar to those above will be herein amended.

### 4.4 SYSTEM CLEANOUT AND WASTE MANAGEMENT

This section is applicable to both R-F and CST resin columns. The application of this section is to minimize the volume and form of waste to be disposed from the hot cells. Wastes shall be handled in accordance with WAC 173-303 (Ecology 1994).

---

**DURING THE TEST RUN, ALL SAMPLE POINTS (LINES B8, B10, B12) WILL HAVE A CATCH CONTAINER TO CONTAIN SPILLS.**

**ALL VALVING CONFIGURATIONS REQUIRE THAT ALL VALVES NOT SPECIFIED AS OPEN SHALL BE CLOSED**

4.4.1 Thermolabile Wastes

All poly tubing, thermoplastics, etc. that will melt are to be placed in a metal container. The container will then be placed on a hot plate and the material melted in accordance with appropriate procedure (LO-100-106, Marshall 1994), the can cooled and sealed and disposed in accordance with WAC 173-303. (WDOE 1994)

4.4.2 Resin Columns

Use Sheet 22 for R-F resin dissolution and Sheet 23 for CST dissolution record and procedural conduct.

Prior to removing the columns, confirm the following valve positions:

| | | |
|---|---|---|
| C1 | V5 | off |
| C2 | V9 | off |
| C3 | V15 | off |
| C4 | V19 | off |

These valves are the lower isolation valves for the columns. When removing the columns, the tubing should be crimped to inhibit leaks. The material in the columns should, be removed from the columns by removing the top of the column and rinsing and scraping the resin into a receiver.

From the determination of the amount of acid ($HNO_3$ for R-F and HCl and HF for CST) under section 4.1.4, remove the resin from C1, and digest proportionately, sampling as SRF2, SRF3, and SRF4. Digest C2 in the same manner, no samples are to be taken. Each column should be dissolved separately. Remove the resin from C3 and digest proportionately to Sheet 19 observations, sampling as SCST2, SCST3, and SCST4. Digest C4 in the same manner, no samples are to be taken. Each column should be dissolved separately. The samples will be analyzed by AEA, ICP and GEA.

**NOTE: Due to the proprietary nature of the CST resin substrate and binder, analytical information from sample analyses of this material will not be publicly released.**

Remaining dissolved exchange material solutions are to be poured down the hot cell drain.

4.4.3 Other Liquors

Effluent materials from test conduct, acquired at A8 and B8, are to be retained, in separate, labeled, 1 liter bottles. These materials will be shipped to PNNL for additional anionic exchange studies.

Waste flushes and washes are to be poured down the hot cell drain. Unanalyzed samples, unless otherwise specified at test close out, are to be poured down the hot cell drain. Unused diluted waste feed, unless otherwise specified at test close out, are to be poured

down the hot cell drain. Unused and undiluted waste feed is to be returned to archive.

#### 4.4.4 Other Solid Wastes

Other solid wastes will include the glass exchange columns, valves, fittings, and glassware for material handling. This material should be triple rinsed and packaged for disposal. Pour rinse liquors down hot cell drain.

### 4.5 SAMPLING AND SAMPLE ANALYSES

Analytical sample selection for analyses of feed wastes, effluents, digest, and eluates are presented in Table 6. Rubidium, potassium, strontium, and cesium isotopic analyses are to be conducted by thermal ionization mass spectroscopy (TIMS); hydroxide by potentiometric titration or specific ion electrode, other metals (Na, Al, K, Cr, Fe, P) are to by analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES), transuranic metals (Am, Pu, Np) are to be analyzed by alpha emission spectroscopy, and $^{137}Cs$ and $^{134}Cs$ by gamma energy analysis (GEA). Selected waste feeds and effluents will be analyzed for total organic carbon by TOC, total inorganic carbon, and for nitrate, nitrite, fluoride, chloride, and sulfate by ion chromatography. Test conduct will include on-line beta-gamma analysis.

Table 6: Cesium Ion Exchange Summary Sample Analysis Plan

| Sample | Method Description | TIMS | ICP-AES | GEA | AEA | IC | TIC | TOC | OH- | Density | On-line $\beta$-$\gamma$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Core | | X | X | | | | | | | | |
| R-F Cesium Ion Exchange Test | | | | | | | | | | | |
| SRF1 | cold RF digest | | X | | | | | | | | |
| SRF2 | hot RF digest | | X | X | X | | | | | | |
| SRF3 | hot RF digest | | X | X | X | | | | | | |
| SRF4 | hot RF digest | | X | X | X | | | | | | |
| W1D | Raw Waste | | | | | | | | | X | |
| F1D | Waste Feed | | | | | | | | | X | |
| F1 | Waste Feed | | X | X | X | X | X | X | X | | |
| FS1 | Centrifuge slds | | X | X | X | | | | | | |
| C1E-1 | C1 Effluent | | X | X | | | | | | | X |
| C1E-2 | C1 Effluent | | X | X | | | | | | | X |
| C1E-3 | C1 Effluent | | X | X | | X | X | | X | | X |
| C1E-4 | C1 Effluent | | X | X | | | | | | | X |
| C1E-5 | C1 Effluent | | X | X | | | | | | | X |
| C1E-6 | C1 Effluent | | X | X | | | | | | | X |
| C1E-7 | C1 Effluent | | X | X | | X | X | | X | | X |
| C1E-8 | C1 Effluent | | X | X | | | | | | | X |
| C1E-9 | C1 Effluent | | X | X | | | | | | | X |
| C1E-10 | C1 Effluent | | X | X | | | | | | | X |
| C1E-11 | C1 Effluent | | X | X | | X | X | | X | | X |
| C1E-12 | C1 Effluent | | X | X | | | | | | | X |

| Table 6: Cesium Ion Exchange Summary Sample Analysis Plan | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample | Method Description | TIMS | ICP-AES | GEA | AEA | IC | TIC | TOC | OH- | Density | On-line $\beta$-$\gamma$ |
| C1E-13 | C1 Effluent | | X | X | | | | | | | X |
| C1E-14 | C1 Effluent | | X | X | | | | | | | X |
| C1E-15 | C1 Effluent | | X | X | | X | X | | X | | X |
| C1E-16 | C1 Effluent | | X | X | | | | | | | X |
| C1E-17 | C1 Effluent | | X | X | | | | | | | X |
| C2E-1 | Comp. C2 Effluent | X | X | X | | X | X | X | | | |
| C1W-1 | Comp. C1 Eluate | | X | X | X | | | | | | |
| C1W-2 | Comp. C1 Eluate | | X | X | X | | | | | | |
| CST Cesium Ion Exchange Test | | | | | | | | | | | |
| SCST1 | cold CST digest | | X | | | | | | | | |
| SCST2 | hot CST digest | | X | X | X | | | | | | |
| SCST3 | hot CST digest | | X | X | X | | | | | | |
| SCST4 | hot CST digest | | X | X | X | | | | | | |
| F2 | Waste Feed | | X | X | X | X | X | | X | | |
| F3 | Waste Feed | | X | X | X | X | X | X | X | | |
| F4 | Waste Feed | | X | X | X | X | X | | X | | |
| F5 | Waste Feed | | X | X | X | X | X | | X | | |
| F6 | Waste Feed | | X | X | X | X | X | X | X | | |
| F7 | Waste Feed | | X | X | X | X | X | | X | | |
| F8 | Waste Feed | | X | X | X | X | X | | X | | |
| F9 | Waste Feed | | X | X | X | X | X | X | X | | |
| FS2 | Centrifuge slds | | X | X | X | | | | | | |
| FS3 | Centrifuge slds | | X | X | X | | | | | | |
| C3E-1 | C3 Effluent | | X | X | | | | | | | X |
| C3E-2 | C3 Effluent | | X | X | | | | | | | X |
| C3E-3 | C3 Effluent | | X | X | | X | X | | X | | X |
| C3E-4 | C3 Effluent | | X | X | | | | | | | X |
| C3E-5 | C3 Effluent | | X | X | | | | | | | X |
| C3E-6 | C3 Effluent | | X | X | | | | | | | X |
| C3E-7 | C3 Effluent | | X | X | | X | X | | X | | X |
| C3E-8 | C3 Effluent | | X | X | | | | | | | X |
| C3E-9 | C3 Effluent | | X | X | | | | | | | X |
| C3E-10 | C3 Effluent | | X | X | | | | | | | X |
| C3E-11 | C3 Effluent | | X | X | | X | X | | X | | X |
| C3E-12 | C3 Effluent | | X | X | | | | | | | X |
| C3E-13 | C3 Effluent | | X | X | | | | | | | X |
| C3E-14 | C3 Effluent | | X | X | | | | | | | X |
| C3E-15 | C3 Effluent | | X | X | | X | X | | X | | X |
| C3E-16 | C3 Effluent | | X | X | | | | | | | X |
| C3E-17 | C3 Effluent | | X | X | | | | | | | X |
| C3E-18 | C3 Effluent | | X | X | | | | | | | X |

Table 6: Cesium Ion Exchange Summary Sample Analysis Plan

| Sample | Method Description | TIMS | ICP-AES | GEA | AEA | IC | TIC | TOC | OH- | Density | On-line $\beta$-$\gamma$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| C3E-19 | C3 Effluent | | X | X | | X | X | | X | | X |
| C3E-20 | C3 Effluent | | X | X | | | | | | | X |
| C3E-21 | C3 Effluent | | X | X | | | | | | | X |
| C3E-22 | C3 Effluent | | X | X | | | | | | | X |
| C3E-23 | C3 Effluent | | X | X | | X | X | | X | | X |
| C3E-24 | C3 Effluent | | X | X | | | | | | | X |
| C3E-25 | C3 Effluent | | X | X | | | | | | | X |
| C3E-26 | C3 Effluent | | X | X | | | | | | | X |
| C3E-27 | C3 Effluent | | X | X | | X | X | | X | | X |
| C4E-1 | Comp. C4 Effluent | X | X | X | | X | X | X | | X | |
| C4E-2 | Comp. C4 Effluent | | X | X | X | | | | X | | |
| C4E-3 | Comp. C4 Effluent | X | X | X | | X | X | X | | X | |
| C4E-4 | Comp. C4 Effluent | | X | X | X | | | | X | | |
| C4E-5 | Comp. C4 Effluent | X | X | X | | X | X | X | | X | |
| C4E-6 | Comp. C4 Effluent | | X | X | X | | | | X | | |
| Total Samples/Method | | TIMS | ICP-AES | GEA | AEA | IC | TIC | TOC | OH- | Density | On-line $\beta$-$\gamma$ |
| 76 | | 5 | 74 | 72 | 24 | 25 | 25 | 9 | 24 | 6 | 45 |

**NOTE: Due to the proprietary nature of the resin substrate and binder, analytical information from sample analyses of this material (SCST1, SCST2, SCST3, SCST4) will not be publicly released.**

## 5.0 CHANGE PROCEDURE

### 5.1 General Changes

As in any experimental test conduct, the possibility of procedural change revision requirement exists. Should procedural change be mandated to ensure adequate performance of the work and safe conduct of operations, the operating personnel may institute the required change with approval of the lead engineer or chemist. Such change will be incorporated in an engineering change notice at test completion for appropriate configuration control.

### 5.2 Sampling Frequency Change

Due to the potential of early breakthrough of analytes of interest (i.e. $^{137}Cs$) in test columns, the on-line $\beta$-$\gamma$ detector will be operated to report effluent activities. Should the activity exceed 10% of breakthrough prior to sampling frequency shift stated in sampling sections of

4.2.6.4 and 4.3.6.4, then the operating personnel should contact the lead engineer or chemist and institute a sampling frequency shift as directed. That shift must be defined in terms of run time (i.e. flow volume). Such change will be incorporated in an engineering change notice at test completion for appropriate configuration control.

### 5.3 Pump Flow Rate Reset

Due to the potential of pump flow rate drift through tubing crimping, exchange material degradation, or other causes, the test apparatus flow may depart from the test objective rate. Should the observed rate under 4.2.6.4 and 4.3.6.4, deviate from the object by greater than 10%, the pump rate should be reset to the appropriate rate through procedures on Sheets 20 and 21 for R-F and CST respectively. The operating personnel may institute the required change with approval of the lead engineer or chemist. Such change will be incorporated in an engineering change notice at test completion for appropriate configuration control.

## 6.0 QUALITY ASSURANCE

Quality assurance requirements are guided by 10 CFR 830.120 *Quality Assurance Requirements* (DOE 1994a) and by the *Implementation Guide for Use with 10 CFR 830.120* (DOE 1994b). The implementation of 10 CFR 830.120 is through the *Quality Assurance Manual* (WHC 1996) and facility specific quality assurance plans.

Existent quality assurance requirements encompassed by the Quality Assurance Plan (Meznarich 1995) of the laboratory facilities will be met in the conduct of this work and its chemical analysis. The quality assurance for the conduct of the unit operation and hot cell activities will be in accordance with the approved process testing quality assurance plan (Meznarich 1996).

## 7.0 REFERENCES

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# Appendix A: Checksheets and Data Sheets

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## LIST OF CHECKSHEETS AND DATASHEETS

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Sheet 1: Feed System Assembly

See Figure A-3 and Figure A-4:
Assemble Line L1 as composite of 1.5 m Tygon tubing, 0.89 mm ID, fitted to 10 cm Tygon tubing, 3.175 mm ID
Connect 3.175 mm ID end of L1 to Valve V1
Mount 0.89 mm ID segment of L1 through pump head

Assembly confirmed: Date:

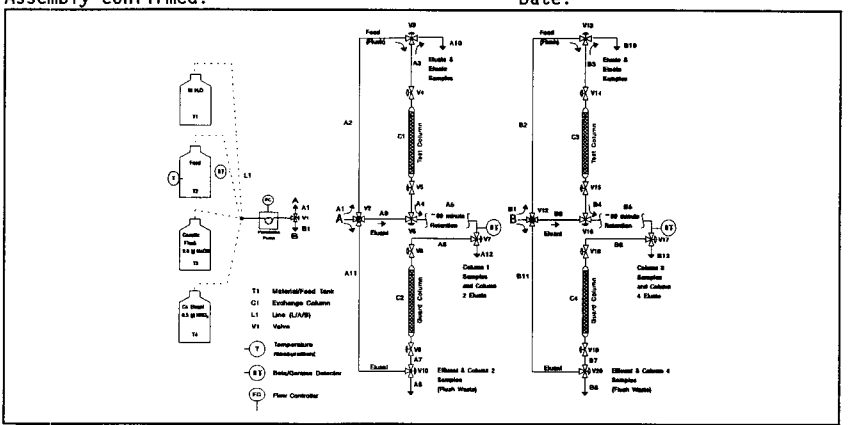

**Figure A-3:** Bench-Scale Cesium Exchange Flows and Instrumentation

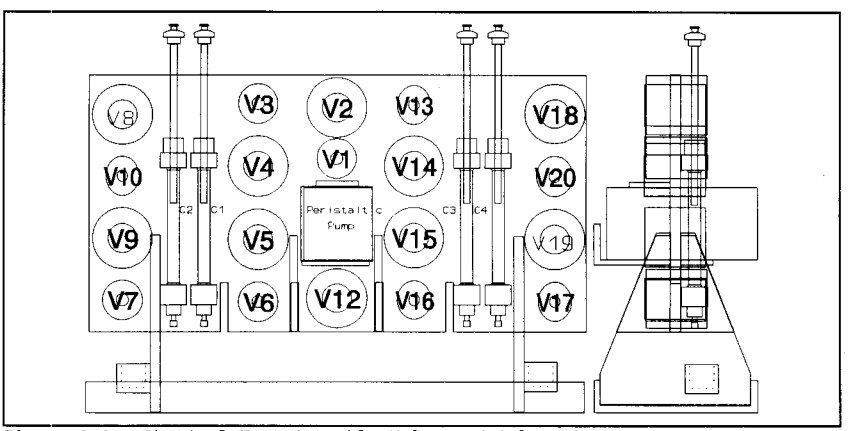


**Figure A-4:** Physical Test Assembly Valve and Column Arrangement

Sheet 2: R-F Test Assembly Instructions and Checksheet

Mount valves and columns in test assembly (See Figure A-3 for flow intent, Figure A-4 for layout).
Connect line A1 (3.2 mm ID, 11 cm Tygon[5]) to V1 and V2.
Connect line A2 (3.2 mm ID, 7 cm Tygon) to V2 and V3.
Connect line A3 (3.2 mm ID, 11 cm Tygon) to V3 and V4.
Connect line (Composite, 3.2 mm ID, 10 cm Tygon to 0.5 mm ID, 32 cm Teflon) to V4 and C1 (top).
Connect line (Composite, 0.5 mm ID, 19 cm Teflon to 3.2 cm, 4 cm Tygon) to C1 (bottom) and V5.
Connect line A4 (3.2 mm ID, 25 cm Tygon) to V5 and V6.
Connect line A5 (V6 to composite: 3.2 mm ID, 3 cm Tygon joined to 5mm ID, 191 cm Tygon joined to 3.2 mm ID, 32 cm Tygon to V7)
Connect line A6 (3.2 mm ID, 27 cm Tygon) to V7 and V8.
Connect line (Composie, 0.5 mm ID, 45 cm Teflon to 3.2 mm ID, 3 cm Tygon) to V8 and C2 (top).
Connect line (Composite, 0.5 mm ID, 29 cm Teflon to 3.2 mm ID, 3 cm Tygon) to C2 (bottom) and V9.
Connect line A7 (3.2 mm ID, 8 cm Tygon) to V9 and V10.
Connect line A8 (3.2 mm ID, 48 cm Tygon) to V10.
Connect line A9 (3.2 mm ID, 41 cm Tygon) to V2 and V6.
Connect line A10 (3.2 mm ID, 58 cm Tygon) to V3.
Connect line A11 (3.2 mm ID, 37 cm Tygon) to V2 and V10.
Connect line A12 (3.2 mm ID, 21 cm Tygon) to V7. init. _____

Assembly confirmed: _____ Date: _____

[5] Tygon is a trademark of Norton Performance Plastics, Akron, OH.

Sheet 3: CST Test Assembly Instructions and Checksheet

Mount valves and columns in test assembly (See Figure A-3 for flow intent, Figure A-4 for layout).
Connect line B1 (3.2 mm ID, 32 cm Tygon) to V1 and V12.
Connect line B2 (3.2 mm ID, 45 cm Tygon) to V12 and V13.
Connect line B3 (3.2 mm ID, 12 cm Tygon) to V13 and V14.
Connect line (Composite, 3.2 mm ID, 5 cm Tygon to 0.5 mm ID, 57 cm Teflon) to V14 and C3 (top).
Connect line (Composite, 0.5 mm ID, 25 cm Teflon to 3.2 mm ID, 4 cm Tygon) to C3 (bottom) and V15.
Connect line B4 (3.2 mm ID, 21 cm Tygon) to V15 and V16.
Connect line B5 (V16 to composite: 3.2 mm ID, 4 cm Tygon joined to 4.8 mm ID, 290 cm Tygon to 3.2 mm ID, 4 cm Tygon to V17)
Connect line B6 (3.2 mm ID, 27 cm Tygon) to V17 and V18.
Connect line (Composite, 3.2 mm ID, 2 cm Tygon to 0.5 mm ID, 54 cm Teflon) to V18 and C4 (top).
Connect line (Composite, 0.5 mm ID, 59 cm Teflon to 3.2 mm ID, 3 cm Tygon) to C4 (bottom) and V19.
Connect line B7 (3.2 mm ID, 9 cm Tygon) to V19 and V20.
Connect line B8 (3.2 mm ID, 48 cm Tygon) to V20.
Connect line B9 (3.2 mm ID, 16 cm Tygon) to V12 and V16.
Connect line B10 (3.2 mm ID, 58 cm Tygon) to V13.
Connect line B11 (3.2 mm ID, 51 cm Tygon) to V12 and V20.
Connect line B12 (3.2 mm ID, 21 cm Tygon) to V17.

init_____

Assembly confirmed: _____ Date: _____

Sheet 4: Config. 1: R-F Pump Flow Rate Valving and Calibration

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Target Flow Rate: _____ g/hr

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (A10) into receiver

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A2 |
| V3 | Open | A2 → V3 → A10 |

| Time (min) | Mass (g) | | | Flow (g/hr) | Time (min) | Mass (g) | | | Flow (g/hr) |
|---|---|---|---|---|---|---|---|---|---|
| | Tare | Gross | Net | | | Tare | Gross | Net | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |

Flow Rate = Average of last three test periods without pump setting change

Flow Rate = _____ g/hr = _____ mL/hr

Sheet 5: Config. 2: R-F Subassembly Forward Feed Valving Configuration

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (A8) into _____

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A2 |
| V3 | Open | A2 → V3 → A3 |
| V4 | Open | A3 → V4 → C1 |
| V5 | Open | C1 → V5 → A4 |
| V6 | Open | A4 → V6 → A5 |
| V7 | Open | A5 → V7 → A6 |
| V8 | Open | A6 → V8 → C2 |
| V9 | Open | C2 → V9 → A7 |
| V10 | Open | A7 → V10 → A8 |

Flow Rate (from Sheet 4, Checksheet#____):_____ mL/hr

Pump On: _____ (time/date)
Observations:

Pump Off:_____ (time/date)

Mass Collected (gross receiver): _____ g (net collected):_____ 9
(Use additional sheets for mass collection if collection vessel changed)

Sheet 6: Config. 3: R-F Subassembly Column 1 Reverse Feed Valving

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (A10) into _____

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A9 |
| V6 | Open | A9 → V6 → A4 |
| V5 | Open | A4 → V5 → C1 |
| V4 | Open | C1 → V4 → A3 |
| V3 | Open | A3 → V3 → A10 |

Flow Rate (from Sheet 4, Checksheet#____):_____ mL/hr

Pump On: _____ (time/date)

Observations:

Pump Off:_____ (time/date)

Mass Collected (gross receiver): _____ g (net collected):_____ g
(Use additional sheets for mass collection if collection vessel changed)

Sheet 7: Config. 4: R-F Subassembly Column 2 Reverse Feed Valving

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____Checksheet#___

Executed by: _____

Date/time: _____

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (A12) into _____

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → A1 |
| V2 | Open | A1 → V2 → A11 |
| V10 | Open | A11 → V10 → A7 |
| V9 | Open | A7 → V9 → C2 |
| V8 | Open | C2 → V8 → A6 |
| V7 | Open | A6 → V7 → A12 |

Flow Rate (from Sheet 4, Checksheet#____):_____ mL/hr

Pump On: _____ (time/date)

Observations:

Pump Off:_____ (time/date)

Mass Collected (gross receiver): _____ g (net collected):_____ 9
(Use additional sheets for mass collection if collection vessel changed)

## Sheet 8: R-F Waste Feed Preparation

### Checksheet and Data Sheet

Objective: Prepare ~700 mL of waste feed for R-F resin test.

Executed for Test Procedure Step: __4.2.4_____ Checksheet#___

Executed by: _____

Date/time: _____

Waste: Core / Riser / Segment #'s:_____

Take an aliquot of the waste for density analysis in a prelabeled vial W1D.

Density Analysis of Waste:_____g/mL

1. By mass, add 350 mL equivalent of waste liquor from waste sample bottles (550 g at SPG = 1.56) of waste liquor into 1L mixing vessel. Actual mass added / density _____g/mL) = _____mL.

2. Add DI water to mixing vessel in aliquots to equal waste mass divided by density . Actual volume added_____.

3. Mix on magnetic stirring plate until no crystals are visible.

4. Allow to settle, decant clear liquor into feed tank T2.

5. Tare centrifuge cones.

6. Centrifuge the waste material mixture, and decant into feed tank T2.

   Observations of liquor clarity, color and of any suspended solids:

7. Upon completion of waste material addition to feed tank T2, pipette a 5 mL aliqout into prelabeled sample vial F1.

8. Take an additional aliquot from T2 and submit it for density analysis, into a prelabeled sample vial F1D.

9. Place any centrifuge solids in prelabeled sample vial FS1.

10. Weigh all centrifuge cones to determine the accumulated mass.

11. Place ~1 gram of the centrifuge solids in a prelabeled sample vial FS1.

Sheet 9: R-F Test Run Primary Column Sampling

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Completed Date/time: _____

Feed Line (L1) into Feed Tank:_____

Feed Material: _____

Sample Vial plus stopper Tare weight:_____g

Flush Vial Tare _____g

Effluent Line (A12) into Flush Vial

Valve Placement/Confirmation: (See Figure A-3) Configuration 2

Change V7 position:
from: A5 → V7 → A6
to: A5 → V7 → A12

Flow Rate (from Sheet 4, Checksheet#____):_____ g/hr

Collect ~2 mL in Flush Vial

Place line A12 into sample vial

Collect ~5 mL in sample vial;

Time of collection:_____min_____sec

Redirect valve V7 back to line A6

Change V7 position:
from: A5 → V7 → A12
to: A5 → V7 → A6

Weight flush:_____g Net flush:_____g

Weight Sample + stopper:_____g, Net Sample:_____g

Flow rate = Net Sample _____ / sample time_____= _____g/hr

_____g/hr / density = _____mL/hr

Observations:

Sheet 10: R-F Test Run Primary Column Sample Record

Sample Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Completed Date/time: _____

Run Start (Date/time):

| Sample | Projected Run Time | Projected Clock Time | Actual Clock Time | On-Line β-γ | Feed Temp. (°C) |
|---|---|---|---|---|---|
| C1E-1 | 1 hr 32 min | | | | |
| C1E-2 | 2 hr 47 min | | | | |
| C1E-3 | 4 hr 2 min | | | | |
| C1E-4 | 5 hr 17 min | | | | |
| C1E-5 | 6 hr 32 min | | | | |
| C1E-6 | 7 hr 47 min | | | | |
| C1E-7 | 9 hr 2 min | | | | |
| C1E-8 | 10 hr 17 min | | | | |
| C1E-9 | 11 hr 32 min | | | | |
| C1E-10 | 12 hr 47 min | | | | |
| C1E-11 | 14 hr 2 min | | | | |
| C1E-12 | 15 hr 17 min | | | | |
| C1E-13 | 16 hr 32 min | | | | |
| C1E-14 | 17 hr 47 min | | | | |
| C1E-15 | 19 hr 2 min | | | | |
| C1E-16 | 20 hr 17 min | | | | |
| C1E-17 | 21 hr 32 min | | | | |

Sheet 11: Config. 5: CST Pump Flow Rate Valving and Calibration

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Target Flow Rate: _____ g/hr

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (A10) into receiver

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V11 | Open | L1 → V11 → B1 |
| V12 | Open | B1 → V12 → B2 |
| V13 | Open | B12 → V13 → B10 |

| Time (min) | Mass (g) | | | Flow (g/hr) | Time (min) | Mass (g) | | | Flow (g/hr) |
|---|---|---|---|---|---|---|---|---|---|
| | Tare | Gross | Net | | | Tare | Gross | Net | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |

Flow Rate = Average of last three test periods without pump setting change

Flow Rate = _____ g/hr / density = _____ mL/hr

Sheet 12: Config. 6: CST Subassembly Forward Feed Valving Configuration

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (B8) into _____

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B2 |
| V13 | Open | B2 → V13 → B3 |
| V14 | Open | B3 → V14 → C3 |
| V15 | Open | C3 → V15 → B4 |
| V16 | Open | B4 → V16 → B5 |
| V17 | Open | B5 → V17 → B6 |
| V18 | Open | B6 → V18 → C4 |
| V19 | Open | C4 → V19 → B7 |
| V20 | Open | B7 → V20 → B8 |

Flow Rate (from Sheet 11 Checksheet#____):_____ mL/hr

Pump On: _____ (time/date)
Observations:

Pump Off:_____ (time/date)

Mass Collected (gross receiver): _____ g (net collected):_____ 9
(Use additional sheets for mass collection if collection vessel changed)

Sheet 13: Config. 7: CST Subassembly Column 3 Reverse Feed Valving

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____g/mL

Effluent Line (B10) into _____

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B9 |
| V16 | Open | B9 → V16 → B4 |
| V15 | Open | B4 → V15 → C3 |
| V14 | Open | C3 → V14 → B3 |
| V13 | Open | B3 → V13 → B10 |

Flow Rate (from Sheet 11 Checksheet#_____):_____ mL/hr

Pump On: _____ (time/date)

Observations:

Pump Off:_____ (time/date)

Mass Collected (gross receiver): _____ g (net collected):_____ 9
(Use additional sheets for mass collection if collection vessel changed)

Sheet 14: Config. 8: CST Subassembly Column 4 Reverse Feed Valving

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Date/time: _____

Receiver/bottle tare:_____ grams

Feed Line (L1) into Feed Tank:_____

Feed Material: _____ Density_____ g/mL

Effluent Line (B12) into _____

Valve Placement/Confirmation (See Figure A-3) other valves closed:

| Valve | Position | Direction |
|---|---|---|
| V1 | Open | L1 → V1 → B1 |
| V12 | Open | B1 → V12 → B11 |
| V20 | Open | B11 → V20 → B7 |
| V19 | Open | B7 → V19 → C4 |
| V18 | Open | C4 → V18 → B6 |
| V17 | Open | B6 → V17 → B12 |

Flow Rate (from Sheet 11 Checksheet#____):_____ mL/hr

Pump On: _____ (time/date)

Observations:

Pump Off:_____ (time/date)

Mass Collected (gross receiver): _____ g (net collected):_____ 9
(Use additional sheets for mass collection if collection vessel changed)

## Sheet 15: CST Waste Feed Preparation

### Checksheet and Data Sheet

Objective: Prepare 1000 mL batch of waste feed for CST resin test.

Executed for Test Procedure Step: __4.2.4_____ Checksheet#___

Executed by: _____

Date/time: _____

1. By mass, add 500 mL equivalent of waste liquor from waste sample bottles (780 g at SPG = 1.56) into 2L mixing vessel.
   Actual mass added / density _____g/mL) = _____mL.

2. Add DI water to mixing vessel in aliquots to equal waste mass divided by density. Actual volume added_____.

3. Mix on magnetic stirring plate until no crystals are visible.

4. Allow to settle, decant clear liquor into feed tank T2.

5. Tare centrifuge cones.

6. Centrifuge the waste material mixture, and decant into feed tank T2.

   Observations of liquor clarity, color and of any suspended solids:

7. Upon completion of waste material addition to T2, pipette a 5 mL aliqout into prelabeled sample vial F___. (seq. number by batch starting at 2)

8. Take an additional aliquot from T2 and submit it for density analysis.

9. Place any centrifuge solids in prelabeled sample vial FS___. (seq. number by batch starting at 2)

10. Weigh all centrifuge cones to determine the accumulated mass.

11. Place ~1 gram of the centrifuge solids in a prelabeled sample vial FS___ (seq. number by batch starting at 2)

Sheet 16: CST Test Run Primary Column Sampling

Configuration Checksheet and Datasheet

Executed for Test Procedure Step: _____ Checksheet#___

Executed by: _____

Completed Date/time: _____

Feed Line (L1) into Feed Tank:_____

Feed Material: _____

Sample Vial plus stopper Tare weight:_____g

Flush Vial Tare: _____g

Effluent Line (B12) into _Flush Vial_____

Valve Placement/Confirmation: (See Figure A-3) Configuration 5

Change V17 position:
from: B5 → V17 → B6
to: B5 → V17 → B12

Flow Rate (from Sheet 11, Checksheet#____):_____ g/hr

Collect ~2 mL in Flush Vial

Place line B12 into sample vial

Collect ~5 mL in sample vial;

Time of colleciton:_____min_____sec

Redirect valve V17 back to line B6

Change V17 position:
from: B5 → V17 → B12
to: B5 → V17 → B6

Weight flush:_____g Net flush:_____g

Weight Sample + stopper:_____g, Net sample:_____g

Flow rate = Net sample _____ / sample time _____ = _____g/hr

Observations:

## Sheet 17: CST Test Run Primary Column Sample Record

### Sample Datasheet

Executed for Test Procedure Step: _____ Checksheet#___
Executed by: _____
Completed Date/time: _____
Run Start (Date/time):

| Sample | Projected Run Time | Projected Clock Time | Actual Clock Time | On-Line β-γ | Feed Temp. (°C) |
|---|---|---|---|---|---|
| C3E-1 | 1 hr 30 min | | | | |
| C3E-2 | 68 hr 10 min | | | | |
| C3E-3 | 72 hr 20 min | | | | |
| C3E-4 | 76 hr 30 min | | | | |
| C3E-5 | 80 hr 40 min | | | | |
| C3E-6 | 84 hr 50 min | | | | |
| C3E-7 | 89 hr 0 min | | | | |
| C3E-8 | 93 hr 10 min | | | | |
| C3E-9 | 97 hr 20 min | | | | |
| C3E-10 | 101 hr 30 min | | | | |
| C3E-11 | 105 hr 40 min | | | | |
| C3E-12 | 109 hr 50 min | | | | |
| C3E-13 | 114 hr 0 min | | | | |
| C3E-14 | 118 hr 10 min | | | | |
| C3E-15 | 122 hr 20 min | | | | |
| C3E-16 | 126 hr 30 min | | | | |
| C3E-17 | 130 hr 40 min | | | | |
| C3E-18 | 134 hr 50 min | | | | |
| C3E-19 | 139 hr 0 min | | | | |
| C3E-20 | 143 hr 10 min | | | | |
| C3E-21 | 147 hr 20 min | | | | |
| C3E-22 | 151 hr 30 min | | | | |
| C3E-23 | 155 hr 40 min | | | | |
| C3E-24 | 159 hr 50 min | | | | |
| C3E-25 | 164 hr 0 min | | | | |
| C3E-26 | 168 hr 10 min | | | | |
| C3E-27 | 172 hr 20 min | | | | |

Sheet 18: R-F Resin Dissolution Test

Sample Datasheet

Executed for Test Procedure Step: _____ Checksheet#____

Executed by: _____

Completed Date/time: _____

Run Start (Date/time): _____

Procedure:

1. In an evaporating dish, weigh out 1 g of R-F resin.
2. Add 2 mL of 3M $HNO_3$ acid.
3. Allow ~ 1 hour for digestion, do not heat above 60°C.
4. Analytically transfer to 100 mL volumetric flask and dilute with DI water to the mark.
5. Subsample ~ 5 mL and retain. Label the subsample as SRF1.

Observations:

Sheet 19: CST Resin Dissolution Test

Sample Datasheet

Executed for Test Procedure Step: _____ Checksheet#____

Executed by: _____

Completed Date/time: _____

Run Start (Date/time): _____

Procedure:

1. In an evaporating dish, weigh out 0.1 g of CST resin.
2. Add 3 mL (+/- 0.5 mL) of DI water.
3, Add ~ 1 mL of 2M HCL.
4. Add 0.5 mL of 1M HF.
5. Heat gently to dissolve.
6. Analytically transfer to a 100 mL volumetric flask and dilute with DI water to the mark.
7. Subsample ~ 5 mL and retain. Label the subsample as SCST1.

Observations:

## Sheet 20: Pump Calibration for the R-F Resin Column Test

Sample Datasheet

Objective: To calibrate the pump flow rate against effluent flow rates at V3 (line A10), V7 (line A12), and V10 (line A8), using 2.5 M NaOH. **This procedure will be carried out for pump flow settings of approximately 15%, 40%, 50%, 60% and 75% of the pump knob range.**

Executed for Test Procedure Step: _____ Checksheet#____

Executed by: _____

Run Start (Date/time): _____Completed Date/time:_____

Prior to carrying out the procedure:

1. Tare the receiver bottle (~250 mL). Note: The receiver bottle will be used and weighed at each sample point.
2. Place line L1 into a feed bottle containing 250 mL of 2.5 M NaOH.
3. Set valve positions to : Configuration 2 (Sheet 5).
4. Place the pump knob setting to the desired flow configuration and allow effluent to flow into a effluent collection bottle located after column C2. Once the system has been charged with the caustic solution, begin the test procedure.

Test Procedure:

1. Close Valve V10.
2. Open Valve V3 to Line A10.
3. Check that the pump knob is set to the desired rate.
4. Turn on pump for 20 minutes, record time on.
5. Turn pump off, record time off, weigh the receiver.
6. Reset Valve V3 to Line A3.
7. Place Line A12 into the tared receiver.
8. Open Valve V7 to Line A12.
9. Turn on pump for 20 minutes, record time on.
10. Turn off pump, record time off, weigh receiver.
11. Reset Valve V7 to Line A6.
12. Place A8 into tared receiver.
13. Open Valve V10 to line A8.
14. Turn on pump for 20 minutes, record time on.
15. Turn off pump, close all valves, record time off, weigh receiver.

Sheet 20 Continued. Checksheet #_____

| A10 Sample | | A12 Sample | | A8 Sample | |
|---|---|---|---|---|---|
| Sample Time | Sample Mass (g) | Sample Time | Sample Mass (g) | Sample Time | Sample Mass (g) |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Flow= Net Sample Mass/Sample Time

| A10 Sample | A12 Sample | A8 Sample |
|---|---|---|
| Flow rate | Flow rate | Flow rate |
| | | |
| | | |
| | | |
| | | |
| | | |

Sheet 21: Pump Calibration for the CST Resin Column Test

Sample Datasheet

Objective: To calibrate the pump flow rate against effluent flow rates at V13 (line B10), V17 (line B12), and V20 (line B8), using 2.5 M NaOH. **This procedure will be carried out for pump flow settings of approximately 15%, 40%, 50%, 60% and 75% of the pump knob range.**

Executed for Test Procedure Step: _____ Checksheet#____

Executed by: _____

Run Start (Date/time): _____Completed Date/time:_____

Prior to carrying out the procedure:

1. Tare the receiver bottle (~250 mL). Note: The receiver bottle will be used and weighed at each sample point.

2. Place line L1 into a feed bottle containing 300 mL of 2.5 M NaOH.

3. Set valve positions to : Configuration 6 (Sheet 12).

4. Place the pump knob setting to the desired flow configuration and allow effluent to flow into a effluent collection bottle located after column C4. Once the system has been charged with the caustic solution, begin the test procedure.

Test Procedure:

1. Close Valve V20.
2. Open Valve V13 to Line B10.
3. Check that the pump knob is set to the desired rate.
4. Turn on pump for 20 minutes, record time on.
5. Turn pump off, record time off, weigh the receiver.
6. Reset Valve V13 to Line B3.
7. Place Line B12 into the tared receiver.
8. Open Valve V17 to Line B12.
9. Turn on pump for 20 minutes, record time on.
10. Turn off pump, record time off, weigh receiver.
11. Reset Valve V17 to Line B6.
12. Place B8 into tared receiver.
13. Open Valve V20 to line B8.
14. Turn on pump for 20 minutes, record time on.
15. Turn off pump, close all valves, record time off, weigh receiver.

Sheet 21 Continued. Checksheet #_____

| B10 Sample | | B12 Sample | | B8 Sample | |
|---|---|---|---|---|---|
| Sample Time | Sample Mass (g) | Sample Time | Sample Mass (g) | Sample Time | Sample Mass (g) |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Flow= Net Sample Mass/Sample Time

| B10 Sample | B12 Sample | B8 Sample |
|---|---|---|
| Flow rate | Flow rate | Flow rate |
| | | |
| | | |
| | | |
| | | |
| | | |

## Sheet 22: R-F Resin Dissolution

Objective: To remove and dissolve the R-F resin at the end of the test. The dissolved resin from Column C1 will be sampled and submitted for analysis by AEA, ICP and GEA.

Checksheet #_____

Resin Dissolution:

Prior to carrying out the procedure:

1. Confirm the following valve positions:

   | | | | | |
   |---|---|---|---|---|
   | C1 | V4 | & | V5 | Off |
   | C2 | V8 | & | V9 | Off |

2. Crimp the tubing to inhibit leaks, two crimps approximately 1" apart on each side of the column.

Procedure:

1. Remove column C1 from the retainers.
2. Cut the line between the crimps above and below the column.
3. Remove the top of the column.
4. Deposit the resin into a receiver by scraping and rinsing.
5. From the determination of the amount of acid needed (Sheet 18), digest proportionatly, sampling as SRF2, SRF3, and SRF4 using prelabeled sample vials.
6. Digest the resin in Column C2 in the same manner, no samples are to be taken. Slurp the remaining resin.

## Sheet 23: CST Resin Dissolution

Objective: To remove and dissolve the CST resin at the end of the test. The dissolved resin from Column C3 will be sampled and submitted for analysis by AEA, ICP and GEA.

Checksheet #_____

Resin Dissolution:

Prior to carrying out the procedure:

1. Confirm the following valve positions:

   C3 V14 & V15 Off
   C4 V18 & V19 Off

2. Crimp the tubing to inhibit leaks, two crimps approximately 1" apart on each side of the column.

Procedure:

1. Remove column C3 from the retainers.

2. Cut the line between the crimps above and below the column.

3. Remove the top of the column.

4. Deposit the resin into a receiver by scraping and rinsing.

5. From the determination of the amount of acid needed (Sheet 19), digest proportionatly, sampling as SCST2, SCST3, and SCST4 using prelabeled sample vials.

6. Digest the resin in Column C4 in the same manner, no samples are to be taken. Slurp the remaining resin.

## Sheet 24: R-F Resin Bed Density Determination

Objective: To determine the volume to mass ratio of the conditioned R-F resin.

Checksheet #_____

Procedure:

1. Dry approximately 5 grams of resin in a tared evaporating dish at 103°C for 4 hours.

2. Cool in a dessicator and weigh on an analytical balance.

   Weight of the resin + evap dish:_____g

   Weight of the evaporting dish: _____g

   Weight of the R-F Resin: _____g

3. Wash the resin to remove fines in a 250 mL beaker with a magnetic stirrer, allow the resin to settle, carefully decant the water without disturbing the resin. Repeat until fines are removed.

4. After the fines are removed, condition the resin as per specifications in the TPI 4.2.2.

5. After the resin is conditioned, analytically decant into a volumetric cylinder. Allow the resin to settle and record the volume.

6. Analytically decant the resin into a tared evaporating dish and dry at 103°C for 4 hours.

   Weight of the resin + evap dish:_____g

   Weight of the evaporting dish: _____g

   Weight of the R-F Resin: _____g

   Volume occupied by the resin: _____mL

   Mass/Volume = _____g/mL

## Sheet 25: CST Resin Bed Density Determination

Objective: To determine the volume to mass ratio of the conditioned CST resin.

Checksheet #_____

Procedure:

1. Dry approximately 5 grams of resin in a tared evaporating dish at 103°C for 4 hours.

2. Cool in a dessicator and weigh on an analytical balance.

   Weight of the resin + evap dish:_____g

   Weight of the evaporting dish: _____g

   Weight of the CST Resin: _____g

3. Wash the resin to remove fines in a 250 mL beaker with a magnetic stirrer, allow the resin to settle, carefully decant the water without disturbing the resin. Repeat until fines are removed.

4. After the fines are removed, condition the resin as per specifications as per specifications provided by the manufacturer.

5. After the resin is conditioned, analytically decant into a volumetric cylinder. Allow the resin to settle and record the volume.

6. Analytically decant the resin into a tared evaporating dish and dry at 103°C for 4 hours.

   Weight of the resin + evap dish:_____g

   Weight of the evaporting dish: _____g

   Weight of the CST Resin: _____g

   Volume occupied by the resin: _____mL

   Mass/Volume = _____g/mL

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Appendix B: Material Safety Data Sheets

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## List of Material Safety Data Sheets



## MSDS Numbers

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Product #: S0899 Name: SODIUM HYDROXIDE PELLETS ACS REAGENT
Material Safety Data Sheet Valid 2/95- 4/95
Printed: 02/22/1995 15:33:32

MSDS # 045620

Sigma Chemical Co.
P.O. Box 14508
St. Louis, MO 63178
Phone: 314-771-5765

Aldrich Chemical Co., Inc.
1001 West St. Paul
Milwaukee, WI 53233
Phone: 414-273-3850

Fluka Chemical Corp.
980 South Second St.
Ronkonkoma, NY 11779
Phone: 516-467-0980
Emergency Phone: 516-467-3535

SECTION 1. - - - - - - - - - - CHEMICAL IDENTIFICATION- - - - - - - - - -
PRODUCT #: S0899
NAME: SODIUM HYDROXIDE PELLETS ACS REAGENT
SECTION 2. - - - - - - COMPOSITION/INFORMATION ON INGREDIENTS - - - - - -
CAS #:1310-73-2
MF: HNAO
SYNONYMS
CAUSTIC SODA * HYDROXYDE DE SODIUM (FRENCH) * LEWIS-RED DEVIL LYE * LYE * NATRIUMHYDROXID (GERMAN) * NATRIUMHYDROXYDE (DUTCH) * SODA LYE * SODIO(IDROSSIDO DI) (ITALIAN) * SODIUM HYDRATE * SODIUM HYDROXIDE (ACGIH,OSHA) * SODIUM HYDROXIDE, SOLID (UN1823) (DOT) * SODIUM HYDROXIDE, SOLUTION (UN1824) (DOT) * SODIUM(HYDROXYDE DE) (FRENCH) * UN1823 (DOT) * UN1824 (DOT) * WHITE CAUSTIC *
SECTION 3. - - - - - - - - - - HAZARDS IDENTIFICATION - - - - - - - - - -
LABEL PRECAUTIONARY STATEMENTS
CORROSIVE
CAUSES BURNS.
HARMFUL BY INHALATION, IN CONTACT WITH SKIN AND IF SWALLOWED.
IN CASE OF CONTACT WITH EYES, RINSE IMMEDIATELY WITH PLENTY OF WATER AND SEEK MEDICAL ADVICE.
TAKE OFF IMMEDIATELY ALL CONTAMINATED CLOTHING.
WEAR SUITABLE PROTECTIVE CLOTHING, GLOVES AND EYE/FACE PROTECTION.
SECTION 4. - - - - - - - - - - FIRST-AID MEASURES- - - - - - - - - - -
IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES OR SKIN WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES.
ASSURE ADEQUATE FLUSHING OF THE EYES BY SEPARATING THE EYELIDS WITH FINGERS.
IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.
IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS.
CALL A PHYSICIAN.
WASH CONTAMINATED CLOTHING BEFORE REUSE.
DISCARD CONTAMINATED SHOES.
SECTION 5. - - - - - - - - - - FIRE FIGHTING MEASURES - - - - - - - - - -
EXTINGUISHING MEDIA
USE EXTINGUISHING MEDIA APPROPRIATE TO SURROUNDING FIRE CONDITIONS.
DO NOT USE WATER.
SPECIAL FIREFIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.

Product #: S0899 Name: SODIUM HYDROXIDE PELLETS ACS REAGENT
Material Safety Data Sheet Valid 2/95- 4/95
Printed: 02/22/1995 15:33:38

MSDS #045620

UNUSUAL FIRE AND EXPLOSIONS HAZARDS
EMITS TOXIC FUMES UNDER FIRE CONDITIONS.
SECTION 6. - - - - - - - - - ACCIDENTAL RELEASE MEASURES- - - - - - - - -
EVACUATE AREA.
WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
SWEEP UP, PLACE IN A BAG AND HOLD FOR WASTE DISPOSAL.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.
SECTION 7. - - - - - - - - - - HANDLING AND STORAGE- - - - - - - - - - -
REFER TO SECTION 8.
ADDITIONAL INFORMATION
CONTACT WITH ALUMINUM, TIN AND ZINC LIBERATES HYDROGEN GAS. CONTACT WITH NITROMETHANE AND OTHER SIMILAR NITRO COMPOUNDS CAUSES FORMATION OF SHOCK-SENSITIVE SALTS.
SECTION 8. - - - - - - - EXPOSURE CONTROLS/PERSONAL PROTECTION- - - - - -
WEAR APPROPRIATE NIOSH/MSHA-APPROVED RESPIRATOR, CHEMICAL-RESISTANT GLOVES, SAFETY GOGGLES, OTHER PROTECTIVE CLOTHING.
SAFETY SHOWER AND EYE BATH.
USE ONLY IN A CHEMICAL FUME HOOD.
FACESHIELD (8-INCH MINIMUM).
DO NOT BREATHE DUST.
DO NOT GET IN EYES, ON SKIN, ON CLOTHING.
AVOID PROLONGED OR REPEATED EXPOSURE.
READILY ABSORBED THROUGH SKIN.
WASH THOROUGHLY AFTER HANDLING.
TOXIC.
CORROSIVE.
KEEP TIGHTLY CLOSED.
EXTREMELY HYGROSCOPIC
AIR SENSITIVE
STORE IN A COOL DRY PLACE.
SECTION 9. - - - - - - - - PHYSICAL AND CHEMICAL PROPERTIES - - - - - - - -
APPEARANCE AND ODOR
WHITE PELLETS
MELTING POINT: 318 C
VAPOR PRESSURE: <18MM 20 C 3MM 37 C
VAPOR DENSITY: >1
SPECIFIC GRAVITY: 2.130
SECTION 10. - - - - - - - - - -STABILITY AND REACTIVITY - - - - - - - - - -
INCOMPATIBILITIES
STRONG OXIDIZING AGENTS
STRONG ACIDS
ORGANIC MATERIALS
CHLORINATED SOLVENTS
ABSORBS CO2 FROM AIR.
PROTECT FROM MOISTURE.
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
NATURE OF DECOMPOSITION PRODUCTS NOT KNOWN.
SECTION 11. - - - - - - - - - - TOXICOLOGICAL INFORMATION - - - - - - - - -

Product #: S0899 Name: SODIUM HYDROXIDE PELLETS ACS REAGENT
Material Safety Data Sheet Valid 2/95- 4/95
Printed: 02/22/1995 15:33:43

MSDS # 045620

ACUTE EFFECTS
HARMFUL IF SWALLOWED, INHALED, OR ABSORBED THROUGH SKIN.
MATERIAL IS EXTREMELY DESTRUCTIVE TO TISSUE OF THE MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT, EYES AND SKIN.
CAUSES SEVERE BURNS.
INHALATION MAY BE FATAL AS A RESULT OF SPASM, INFLAMMATION AND EDEMA OF THE LARYNX AND BRONCHI, CHEMICAL PNEUMONITIS AND PULMONARY EDEMA.
SYMPTOMS OF EXPOSURE MAY INCLUDE BURNING SENSATION, COUGHING, WHEEZING, LARYNGITIS, SHORTNESS OF BREATH, HEADACHE, NAUSEA AND VOMITING.
TO THE BEST OF OUR KNOWLEDGE, THE CHEMICAL, PHYSICAL, AND TOXICOLOGICAL PROPERTIES HAVE NOT BEEN THOROUGHLY INVESTIGATED.
RTECS NO: WB4900000
SODIUM HYDROXIDE
IRRITATION DATA

| | |
|---|---|
| EYE-MKY 1%/24H SEV | TXAPA9 6,701,64 |
| SKN-RBT 500 MG/24H SEV | 28ZPAK -,7,72 |
| EYE-RBT 400 UG MLD | OYYAA2 26,627,83 |
| EYE-RBT 1% SEV | AJOPAA 29,1363,46 |
| EYE-RBT 50 UG/24H SEV | 28ZPAK -,7,72 |
| EYE-RBT 1 MG/24H SEV | TXAPA9 6,701,64 |
| EYE-RBT 1 MG/30S RINSE SEV | TXCYAC 23,281,82 |

TOXICITY DATA

| | |
|---|---|
| IPR-MUS LD50:40 MG/KG | COREAF 257,791,63 |

ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATION.
SECTION 12. - - - - - - - - - - ECOLOGICAL INFORMATION - - - - - - - - - -
DATA NOT YET AVAILABLE.
SECTION 13. - - - - - - - - - - DISPOSAL CONSIDERATIONS - - - - - - - - - -
FOR SMALL QUANTITIES: CAUTIOUSLY ADD TO A LARGE STIRRED EXCESS OF WATER. ADJUST THE PH TO NEUTRAL, SEPARATE ANY INSOLUBLE SOLIDS OR LIQUIDS AND PACKAGE THEM FOR HAZARDOUS-WASTE DISPOSAL. FLUSH THE AQUEOUS SOLUTION DOWN THE DRAIN WITH PLENTY OF WATER. THE HYDROLYSIS AND NEUTRALIZATION REACTIONS MAY GENERATE HEAT AND FUMES WHICH CAN BE CONTROLLED BY THE RATE OF ADDITION.
OBSERVE ALL FEDERAL, STATE AND LOCAL ENVIRONMENTAL REGULATIONS.
SECTION 14. - - - - - - - - - - TRANSPORT INFORMATION - - - - - - - - - -
CONTACT SIGMA CHEMICAL COMPANY FOR TRANSPORTATION INFORMATION.
SECTION 15. - - - - - - - - - - REGULATORY INFORMATION - - - - - - - - - -
REVIEWS, STANDARDS, AND REGULATIONS

| | |
|---|---|
| ACGIH TLV-CL 2 MG/M3 | 85INA8 6,1416,91 |

EPA FIFRA 1988 PESTICIDE SUBJECT TO REGISTRATION OR RE-REGISTRATION
FEREAC 54,7740,89
MSHA STANDARD:AIR-CL 2 MG/M3
DTLVS* 3,233,71
OSHA PEL:8H TWA 2 MG/M3
FEREAC 54,2923,89
OSHA PEL FINAL:CL 2 MG/M3

Product #: S0899 Name: SODIUM HYDROXIDE PELLETS ACS REAGENT
Material Safety Data Sheet Valid 2/95- 4/95
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MSDS # 045620

FEREAC 54,2923,89
OEL-AUSTRALIA:TWA 2 MG/M3 JAN93
OEL-BELGIUM:STEL 2 MG/M3 JAN93
OEL-DENMARK:TWA 2 MG/M3 JAN93
OEL-FINLAND:TWA 2 MG/M3 JAN93
OEL-FRANCE:TWA 2 MG/M3 JAN93
OEL-GERMANY:TWA 2 MG/M3 JAN93
OEL-JAPAN:STEL 2 MG/M3 JAN93
OEL-THE NETHERLANDS:TWA 2 MG/M3 JAN93
OEL-THE PHILIPPINES:TWA 2 MG/M3 JAN93
OEL-SWEDEN:TWA 2 MG/M3 JAN93
OEL-SWITZERLAND:TWA 2 MG/M3;STEL 4 MG/M3 JAN93
OEL-THAILAND:TWA 2 MG/M3 JAN93
OEL-TURKEY:TWA 2 MG/M3 JAN93
OEL-UNITED KINGDOM:TWA 2 MG/M3;STEL 2 MG/M3 JAN93
OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA CHECK ACGIH TLV
OEL IN NEW ZEALAND, SINGAPORE, VIETNAM CHECK ACGIH TLV
NIOSH REL TO SODIUM HYDROXIDE-AIR:CL 2 MG/M3/15M
NIOSH* DHHS #92-100,92
NOHS 1974: HZD 69070; NIS 359; TNF 112525; NOS 192; TNE 1122583
NOES 1983: HZD X3782; NIS 164; TNF 20410; NOS 117; TNE 332750; TFE 123903
NOES 1983: HZD 69070; NIS 394; TNF 107562; NOS 231; TNE 2233346; TFE 740163
EPA GENETOX PROGRAM 1988, NEGATIVE: CELL TRANSFORM.-SA7/SHE
EPA TSCA CHEMICAL INVENTORY, JUNE 1993
EPA TSCA SECTION 8(E) STATUS REPORT 8EHQ-0485-0552
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, JULY 1994
NIOSH ANALYTICAL METHODS: SEE ALKALINE DUSTS, 7401

SECTION 16. - - - - - - - - - - - OTHER INFORMATION- - - - - - - - - - - -

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. SIGMA, ALDRICH, FLUKA SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITIONS OF SALE.

MATERIAL SAFETY DATA SHEET

Ashland

ASHLAND CHEMICAL, INC.
Subsidiary Of Ashland Oil, Inc
P.O. BOX 2219
COLUMBUS, OHIO 43216
(614) 889-3333

24-HOUR Emergency Telephone
1(800) 274-5263 or
1(800) ASHLAND

# NITRIC ACID REAGENT ACS



000026

THIS MSDS COMPLIES WITH 29 CFR 1910.1200 (THE HAZARD COMMUNICATION STANDARD).

Product Name: NITRIC ACID REAGENT ACS
CAS NUMBER: 7697-37-2

05 92 022 4074722-226

Data Sheet No: 0000898-006
Prepared: 12/27/88
Supersedes: 04/15/88

HANFORD ENVIRON. HEALTH FNDTN.
ATTN: DANIEL L. SNIDER
905 GOETHALS DR.
MSIN: B2-75
RICHLAND, WA 99352

PRODUCT:
INVOICE: REQST
INVOICE DATE: 02/24/92
TO:

MSDS #043200

## SECTION I PRODUCT IDENTIFICATION

General or Generic ID: INORGANIC ACID

DOT Hazard Classification: CORROSIVE (173.240) AND OXIDIZER (173.151)

## SECTION II COMPONENTS

IF PRESENT, IARC, NTP AND OSHA CARCINOGENS AND CHEMICALS SUBJECT TO THE REPORTING REQUIREMENTS OF SARA TITLE III SECTION 313 ARE IDENTIFIED IN THIS SECTION.
SEE DEFINITION PAGE FOR CLARIFICATION

| INGREDIENT | % (by WT) | PEL | TLV | Note |
|---|---|---|---|---|
| NITRIC ACID<br>CAS #: 7697-37-2 | 70 | 2 PPM | 2 PPM | ( 1) |

Notes:

( 1) OSHA/ACGIH SHORT TERM EXPOSURE LIMIT (STEL) FOR NITRIC ACID IS 4.0 PPM. NIOSH RECOMMENDS A LIMIT OF 2.0 PPM, 15-HOUR TWA.

THIS CHEMICAL IS SUBJECT TO THE REPORTING REQUIREMENTS OF SECTION 313 OF SARA TITLE III.

## SECTION III PHYSICAL DATA

| | | |
|---|---|---|
| Boiling Point | for PRODUCT | 250.00 Deg F<br>( 121.11 Deg C)<br>@ 760.00 mm Hg |
| Vapor Pressure | for PRODUCT | 5.50 mm Hg<br>@ 68.00 Deg F<br>( 20.00 Deg C) |
| Specific Vapor Density | AIR = 1 | 1.3 |
| Specific Gravity | | 1.413<br>@ 68.00 Deg F<br>( 20.00 Deg C) |
| Percent Volatiles | | 100.00% |
| Evaporation Rate | | SLOWER THAN ETHER |

## SECTION IV FIRE AND EXPLOSION INFORMATION

FLASH POINT NOT APPLICABLE

EXPLOSIVE LIMIT NOT APPLICABLE

EXTINGUISHING MEDIA: WATER FOG

HAZARDOUS DECOMPOSITION PRODUCTS: MAY FORM TOXIC MATERIALS:, NITROGEN COMPOUNDS, ACID VAPORS

FIREFIGHTING PROCEDURES: WEAR SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE OPERATED IN THE POSITIVE PRESSURE DEMAND MODE AND FULL BODY PROTECTION WHEN FIGHTING FIRES.

WATER MAY BE USED TO KEEP FIRE-EXPOSED CONTAINERS COOL UNTIL FIRE IS OUT.

SPECIAL FIRE & EXPLOSION HAZARDS: ACID REACTS WITH MOST METALS TO RELEASE HYDROGEN GAS WHICH CAN FORM EXPLOSIVE MIXTURES WITH AIR.

THIS MATERIAL IS A POWERFUL OXIDIZER. CONTACT WITH COMBUSTIBLE MATERIALS CAN RESULT IN FIRE OR EXPLOSION.

NFPA CODES: HEALTH- 3 FLAMMABILITY- 0 REACTIVITY- 0

## SECTION V HEALTH HAZARD DATA

PERMISSIBLE EXPOSURE LEVEL 2 PPM

THRESHOLD LIMIT VALUE 2 PPM

EFFECTS OF ACUTE OVEREXPOSURE:

EYES - CAUSES SEVERE DAMAGE AND EVEN BLINDNESS VERY RAPIDLY.
SKIN - CAUSES BURNS, POSSIBLE DEEP ULCERATION.
BREATHING - OF FUMES CAN CAUSE DAMAGE TO NASAL AND RESPIRATORY PASSAGES.
SWALLOWING - RESULTS IN SEVERE DAMAGE TO MUCOUS MEMBRANES AND DEEP TISSUES.



CONTINUED ON PAGE: 2

05/29/96 09:01:10 1-589-376-9771-> 589 376 9964 Page 003

| MATERIAL SAFETY DATA SHEET | Ashland | ASHLAND CHEMICAL, INC. Subsidiary Of Ashland Oil Inc P.O. BOX 2219 COLUMBUS, OHIO 43216 (614) 889-3333 | 24-HOUR Emergency Telephone 1(800) 274-5263 or 1(800) ASHLAND |
|---|---|---|---|

000028 NITRIC ACID REAGENT ACS MSDS #043200 Page: 2

SECTION V HEALTH HAZARD DATA (Continued)

FIRST AID:

IF ON SKIN: IMMEDIATELY FLUSH EXPOSED AREA WITH WATER FOR AT LEAST 15 MINUTES. GET MEDICAL ATTENTION. REMOVE CONTAMINATED CLOTHING. LAUNDER CONTAMINATED CLOTHING BEFORE RE-USE.

REMOVE CONTAMINATED SHOES PROMPTLY. DISCARD SHOES SATURATED WITH THIS PRODUCT.

IF IN EYES: IMMEDIATELY FLUSH WITH LARGE AMOUNTS OF WATER FOR AT LEAST 15 MINUTES, LIFTING UPPER AND LOWER LIDS OCCASIONALLY. GET IMMEDIATE MEDICAL ATTENTION.

IF PHYSICIAN IS NOT IMMEDIATELY AVAILABLE, CONTINUE FLUSHING WITH WATER.

DO NOT USE CHEMICAL ANTIDOTE.

IF SWALLOWED: DO NOT INDUCE VOMITING. VOMITING WILL CAUSE FURTHER DAMAGE TO THE THROAT. DILUTE BY GIVING WATER. GIVE MILK OF MAGNESIA. KEEP WARM. QUIET. GET MEDICAL ATTENTION IMMEDIATELY.

IF BREATHED: IF AFFECTED, REMOVE INDIVIDUAL TO FRESH AIR. IF BREATHING IS DIFFICULT, ADMINISTER OXYGEN. IF BREATHING HAS STOPPED GIVE ARTIFICIAL RESPIRATION. KEEP PERSON WARM, QUIET AND GET MEDICAL ATTENTION.

PRIMARY ROUTE(S) OF ENTRY:

INHALATION, SKIN CONTACT

SECTION VI REACTIVITY DATA

HAZARDOUS POLYMERIZATION: CANNOT OCCUR

STABILITY: STABLE

INCOMPATIBILITY: AVOID CONTACT WITH:, STRONG ALKALIES, ORGANIC MATERIALS, REDUCING AGENTS

SECTION VII SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED:

SMALL SPILL: COVER THE CONTAMINATED SURFACE WITH SODIUM BICARBONATE OR A SODA ASH/FLAKED LIME MIXTURE (50-50). MIX AND ADD WATER IF NECESSARY TO FORM A SLURRY. SCOOP UP SLURRY AND WASH SITE WITH SODA ASH SOLUTION. PROPER MIXING PROCEDURES ARE WARRANTED. TRAINED PERSONNEL SHOULD CONDUCT THIS PROCEDURE. UNTRAINED PERSONNEL SHOULD BE REMOVED FROM THE SPILL AREA.

LARGE SPILL: PERSONS NOT WEARING PROTECTIVE EQUIPMENT SHOULD BE EXCLUDED FROM AREA OF SPILL UNTIL CLEAN-UP IS COMPLETED. STOP SPILL AT SOURCE. DIKE TO PREVENT SPREADING. PUMP TO SALVAGE TANK.

WASTE DISPOSAL METHOD:

SMALL SPILL: DISPOSE OF IN ACCORDANCE WITH ALL LOCAL, STATE AND FEDERAL REGULATIONS.

DISPOSE OF IN ACCORDANCE WITH ALL LOCAL, STATE AND FEDERAL REGULATIONS.

SECTION VIII PROTECTIVE EQUIPMENT TO BE USED

RESPIRATORY PROTECTION: IF WORKPLACE EXPOSURE LIMIT(S) OF PRODUCT OR ANY COMPONENT IS EXCEEDED (SEE SECTION II), A NIOSH/MSHA APPROVED AIR SUPPLIED RESPIRATOR IS ADVISED IN ABSENCE OF PROPER ENVIRONMENTAL CONTROL. OSHA REGULATIONS ALSO PERMIT OTHER NIOSH/MSHA RESPIRATORS (NEGATIVE PRESSURE TYPE) UNDER SPECIFIED CONDITIONS (SEE YOUR SAFETY EQUIPMENT SUPPLIER). ENGINEERING OR ADMINISTRATIVE CONTROLS SHOULD BE IMPLEMENTED TO REDUCE EXPOSURE.

VENTILATION: PROVIDE SUFFICIENT MECHANICAL (GENERAL AND/OR LOCAL EXHAUST) VENTILATION TO MAINTAIN EXPOSURE BELOW TLV(S).

PROTECTIVE GLOVES: WEAR RESISTANT GLOVES SUCH AS:, NEOPRENE, POLYVINYL CHLORIDE

EYE PROTECTION: CHEMICAL SPLASH GOGGLES AND FACE SHIELD (8" MIN.) IN COMPLIANCE WITH OSHA REGULATIONS ARE ADVISED; HOWEVER, OSHA REGULATIONS ALSO PERMIT OTHER TYPE SAFETY GLASSES. (CONSULT YOUR SAFETY EQUIPMENT SUPPLIER)

OTHER PROTECTIVE EQUIPMENT: TO PREVENT SKIN CONTACT, WEAR IMPERVIOUS CLOTHING AND BOOTS.

SECTION IX SPECIAL PRECAUTIONS OR OTHER COMMENTS

ADDITION TO WATER RELEASES HEAT WHICH CAN RESULT IN VIOLENT BOILING AND SPATTERING. ALWAYS ADD SLOWLY AND IN SMALL AMOUNTS. NEVER USE HOT WATER.

CONTAINERS OF THIS MATERIAL MAY BE HAZARDOUS WHEN EMPTIED.SINCE EMPTIED CONTAINERS RETAIN PRODUCT RESIDUES (VAPOR, LIQUID, AND/OR SOLID), ALL HAZARD PRECAUTIONS GIVEN IN THE DATA SHEET MUST BE OBSERVED.

THE INFORMATION ACCUMULATED HEREIN IS BELIEVED TO BE ACCURATE BUT IS NOT WARRANTED TO BE WHETHER ORIGINATING WITH THE COMPANY OR NOT. RECIPIENTS ARE ADVISED TO CONFIRM IN ADVANCE OF NEED THAT THE INFORMATION IS CURRENT, APPLICABLE, AND SUITABLE TO THEIR CIRCUMSTANCES.

 LAST PAGE--SEE ATTACHMENT PAGE ENCLOSED--LAST PAGE

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SUBSTANCE IDENTIFICATION

CAS-NUMBER 7664-39-3

SUBSTANCE: HYDROFLUORIC ACID REAGENT ACS

MSDS # 039275

TRADE NAMES/SYNONYMS:
FLUORIC ACID; HYDROFLUORIC ACID SOLUTION; UN 1790; RCRA U134; HF; OHS11172

CHEMICAL FAMILY:
INORGANIC ACID

MOLECULAR FORMULA: H-F

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=0 REACTIVITY=1 PERSISTENCE=0
NFPA RATINGS (SCALE 0-4): HEALTH=4 FIRE=0 REACTIVITY=1

---

COMPONENTS AND CONTAMINANTS

COMPONENT: HYDROGEN FLUORIDE PERCENT: 49.0

COMPONENT: WATER PERCENT: 51.0

EXPOSURE LIMITS:
HYDROGEN FLUORIDE, AS F:
3 PPM OSHA TWA; 6 PPM OSHA STEL
3 PPM (2.5 MG/M3) ACGIH CEILING
3 PPM (2.5 MG/M3) NIOSH RECOMMENDED 10 HOUR TWA;
6 PPM (5 MG/M3) NIOSH RECOMMENDED 15 MINUTE CEILING

100 POUNDS SARA SECTION 302 THRESHOLD PLANNING QUANTITY
100 POUNDS SARA SECTION 304 REPORTABLE QUANTITY
100 POUNDS CERCLA SECTION 103 REPORTABLE QUANTITY
SUBJECT TO SARA SECTION 313 ANNUAL TOXIC CHEMICAL RELEASE REPORTING

---

PHYSICAL DATA

DESCRIPTION: COLORLESS TO SLIGHTLY YELLOW LIQUID WITH A PUNGENT ODOR.

BOILING POINT: 225 F (107 C) MELTING POINT: -35 F (-37 C) (APPROX.)

SPECIFIC GRAVITY: 1.15-1.18 VAPOR PRESSURE: 25 MMHG @ 20C PH: <2

SOLUBILITY IN WATER: SOLUBLE

---

FIRE AND EXPLOSION DATA

FIRE AND EXPLOSION HAZARD:
NEGLIGIBLE FIRE HAZARD WHEN EXPOSED TO HEAT OR FLAME.

**MSDS #1039275**

FIREFIGHTING MEDIA:
DRY CHEMICAL, CARBON DIOXIDE, HALON, WATER SPRAY OR ALCOHOL FOAM
(1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4).

FOR LARGER FIRES, USE WATER SPRAY, FOG OR STANDARD FOAM
(1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4).

FIREFIGHTING:
MOVE CONTAINERS FROM FIRE AREA IF POSSIBLE. COOL CONTAINERS EXPOSED TO FLAMES WITH WATER FROM SIDE UNTIL WELL AFTER FIRE IS OUT. STAY AWAY FROM STORAGE TANK ENDS (1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4, GUIDE PAGE 59).

DO NOT USE WATER ON MATERIAL. EXTINGUISH USING AGENTS SUITABLE FOR TYPE OF FIRE. USE FLOODING AMOUNTS OF WATER AS FOG. COOL CONTAINERS WITH FLOODING AMOUNTS OF WATER, APPLY FROM AS FAR A DISTANCE AS POSSIBLE. AVOID BREATHING CORROSIVE VAPORS, KEEP UPWIND.

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TRANSPORTATION DATA

DEPARTMENT OF TRANSPORTATION HAZARD CLASSIFICATION 49CFR172.101:
CORROSIVE MATERIAL

DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS 49CFR172.101 AND SUBPART E:
CORROSIVE

DEPARTMENT OF TRANSPORTATION PACKAGING REQUIREMENTS: 49CFR173.264
EXCEPTIONS: 49CFR173.244

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TOXICITY

HYDROGEN FLUORIDE:
50 MG EYE-HUMAN SEVERE IRRITATION; 100 MG/M3/1 MINUTE INHALATION-MAN TCLO; 50 PPM/30 MINUTES INHALATION-HUMAN LCLO; 1276 PPM/1 HOUR INHALATION-RAT LC50; 342 PPM/1 HOUR INHALATION-MOUSE LC50; 1774 PPM/1 HOUR INHALATION-MONKEY LC50; 260 MG/M3/7 HOURS INHALATION-RABBIT LCLO; 4327 PPM/15 MINUTES INHALATION-GUINEA PIG LC50; 500 MG/KG SKIN-MOUSE LDLO; 25 MG/KG INTRAPERITONEAL-RAT LDLO; MUTAGENIC DATA (RTECS); REPRODUCTIVE EFFECTS DATA (RTECS).
CARCINOGEN STATUS: NONE.
HYDROGEN FLUORIDE IS TOXIC AND A SEVERE EYE, SKIN AND MUCOUS MEMBRANE IRRITANT. CHRONIC EXPOSURE MAY PRODUCE FLUOROSIS OF THE SKELETAL SYSTEM.

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HEALTH EFFECTS AND FIRST AID

INHALATION:
HYDROGEN FLUORIDE:
CORROSIVE/TOXIC. 30 PPM IMMEDIATELY DANGEROUS TO LIFE OR HEALTH.
ACUTE EXPOSURE- TWO HUMAN SUBJECTS EXPOSED TO 120 PPM EXPERIENCED MARKED RESPIRATORY IRRITATION. THIS WAS THE HIGHEST CONCENTRATION THAT COULD BE TOLERATED FOR MORE THAN ONE MINUTE. 30 PPM CAUSED MILD NASAL IRRITATION

AND COULD BE TOLERATED FOR SEVERAL MINUTES. HIGHER CONCENTRATIONS MAY CAUSE TRANSIENT CHOKING, COUGHING, CHILLS, CHEST PAIN AND CONSTRICTION, AND DYSPNEA. AN ASYMPTOMATIC PERIOD OF 12-48 HOURS MAY BE FOLLOWED BY FEVER, COUGH, DYSPNEA, CYANOSIS, RALES, AND PULMONARY EDEMA OR BRONCHIAL PNEUMONIA. IN HUMANS, KIDNEY DAMAGE HAS ONLY BEEN REPORTED IN SEVERE, ACUTE OVEREXPOSURES. IN FOUR SEPARATE EVENTS, 9 WORKERS WERE SPLASHED WITH HYDROFLUORIC ACID; 6 DIED. DEATH OCCURRED 2-10 HOURS AFTER EXPOSURE AND WAS CAUSED BY PULMONARY EDEMA, HEMORRHAGIC PULMONARY EDEMA AND ULCERATIVE TRACHEOBRONCHITIS, OR CARDIAC ARREST. IN ONE INSTANCE, THE BREATHING ZONE CONCENTRATION WAS ESTIMATED TO BE ABOVE 10,000 PPM.

CHRONIC EXPOSURE- 5 HUMAN SUBJECTS EXPOSED 6 HOURS/DAY, 5 DAYS/WEEK, FOR 10-50 DAYS AT AVERAGE CONCENTRATIONS OF UP TO 4.7 PPM EXPERIENCED SLIGHT NASAL IRRITATION. REPEATED EXPOSURE TO LOW CONCENTRATIONS MAY CAUSE NASAL CONGESTION, NOSEBLEEDS, SINUS PROBLEMS, AND BRONCHITIS. ABSORPTION OF EXCESSIVE AMOUNTS OF FLUORINE MAY RESULT IN FLUOROSIS, A SYNDROME CHARACTERIZED BY OSTEOSCLEROTIC BONE CHANGES. CASES OF VARYING DEGREES OF OSTEOSCLEROSIS HAVE BEEN REPORTED IN WORKERS EXPOSED TO HYDROGEN FLUORIDE FOR A NUMBER OF YEARS, USUALLY 3 OR MORE. THE FIRST EVIDENCE OF CHANGE IS MOST APPARENT IN THE PELVIS AND LUMBAR SPINE AND MAY BE ACCOMPANIED BY MILD TO MODERATE BACK PAIN AND STIFFNESS. PRESUMABLY, OTHER SYMPTOMS OF FLUOROSIS, WEIGHT LOSS, GENERAL ILL HEALTH, ANEMIA, BRITTLENESS OF THE BONES, AND DISCOLORATION OF DEVELOPING TEETH, ARE ALSO POSSIBLE. ANIMAL STUDIES INDICATE THAT REPEATED EXPOSURE MAY CAUSE PULMONARY, HEPATIC AND RENAL TISSUE DAMAGE. EXPOSURE OF PREGNANT RATS FOR 22 DAYS RESULTED IN IN EFFECTS ON FERTILITY AND ON THE FETUS.

FIRST AID- REMOVE FROM EXPOSURE AREA TO FRESH AIR IMMEDIATELY. IF BREATHING HAS STOPPED, GIVE ARTIFICIAL RESPIRATION. MAINTAIN AIRWAY AND BLOOD PRESSURE AND ADMINISTER OXYGEN IF AVAILABLE. KEEP AFFECTED PERSON WARM AND AT REST. TREAT SYMPTOMATICALLY AND SUPPORTIVELY. ADMINISTRATION OF OXYGEN SHOULD BE PERFORMED BY QUALIFIED PERSONNEL. GET MEDICAL ATTENTION IMMEDIATELY.

SKIN CONTACT:
HYDROGEN FLUORIDE:
CORROSIVE.

ACUTE EXPOSURE- HYDROGEN FLUORIDE BURNS ARE CHARACTERIZED BY A BLANCHED APPEARANCE OF THE SKIN WITH PERSISTENT EXCRUCIATING PAIN, EDEMA AND NECROSIS. WITH CONCENTRATIONS LESS THAN 20%, PAIN AND ERYTHEMA MAY OCCUR AFTER A LATENT PERIOD OF 24 HOURS. WITH 20-50% SOLUTIONS, BURNS MAY BE APPARENT WITHIN 1-8 HOURS. WITH CONCENTRATIONS GREATER THAN 50%, IMMEDIATE PAIN AND RAPIDLY APPARENT TISSUE DAMAGE OCCUR ON CONTACT. SMALL AMOUNTS OF HYDROGEN FLUORIDE WHICH ARE NOT WASHED OFF MAY CAUSE DELAYED DEVELOPMENT OF NON-HEALING ULCERS. FINGERNAILS AND NAIL BEDS MAY BE COMPLETELY DESTROYED. PENETRATION OF THE FLUORIDE ION TO DEEP TISSUES MAY RESULT IN SLOW HEALING NECROSIS OF SOFT TISSUES AND DECALCIFICATION OF BONE. VAPORS AT A CONCENTRATION OF OF 120 PPM CAUSED SMARTING OF EXPOSED SKIN IN PEOPLE IN ONE MINUTE. SYSTEMIC FLUORIDE POISONING THROUGH SKIN ABSORPTION MAY OCCUR. STUPOR, UNRESPONSIVENESS TO STIMULI OTHER THAN PAIN, SEVERE NAUSEA, VOMITING, AND REDUCED PULSE RATE WERE REPORTED IN ONE CASE.

CHRONIC EXPOSURE- REPEATED OR PROLONGED EXPOSURE MAY CAUSE IRRITATION OR BURNS. SLIGHT IRRITATION OCCURRED IN PEOPLE EXPOSED TO VAPOR CONCENTRATIONS AVERAGING 2.6 AND 4.7 PPM FOR PERIODS OF UP TO 50 DAYS. SLIGHT DESQUAMATION OF THE SUPERFICIAL EPITHELIUM OF THE FACE WAS OBSERVED IN ONE SUBJECT AFTER TEN DAYS OF EXPOSURE TO HYDROGEN FLUORIDE AT 3.4 PPM.

FIRST AID- REMOVE CONTAMINATED CLOTHING AND SHOES IMMEDIATELY. WASH AFFECTED AREA WITH SOAP OR MILD DETERGENT AND LARGE AMOUNTS OF WATER UNTIL NO EVIDENCE OF CHEMICAL REMAINS (AT LEAST 15-20 MINUTES). IN CASE OF CHEMICAL BURNS, COVER AREA WITH STERILE, DRY DRESSING. BANDAGE SECURELY, BUT NOT TOO TIGHTLY. GET MEDICAL ATTENTION IMMEDIATELY.

EYE CONTACT:
HYDROGEN FLUORIDE:
CORROSIVE.
ACUTE EXPOSURE- EXPOSURE TO HYDROGEN FLUORIDE AVERAGING 4.7 PPM FOR 6 HOURS, OR 30 PPM FOR SEVERAL MINUTES CAUSED MILD IRRITATION, WHILE 120 PPM RESULTED IN MARKED CONJUNCTIVAL IRRITATION WITHIN 1 MINUTE IN HUMANS. DIRECT CONTACT WITH THE LIQUID OR SOLUTIONS MAY CAUSE CORNEAL BURNS. IF NOT PROMPTLY REMOVED, PERMANENT VISUAL DEFECTS OR BLINDNESS MAY RESULT. ONE WORKER EXPOSED TO A FINE SPRAY OF CONCENTRATED HYDROGEN FLUORIDE, HAD LOSS OF EPITHELIUM FROM THE CORNEA AND CONJUNCTIVA, AND MARKED EDEMA OF THE EYELIDS, CONJUNCTIVA, AND CORNEAS; PROMPT TREATMENT WAS ADMINISTERED. NORMAL VISION WAS REGAINED WITHIN 19 DAYS.
CHRONIC EXPOSURE- HUMANS EXPERIMENTALLY EXPOSED TO CONCENTRATIONS WHICH AVERAGED 2.6-4.7 PPM FOR UP TO 50 DAYS DEVELOPED MILD EYE IRRITATION. IN ANIMALS, REPEATED OR PROLONGED EXPOSURE TO LOW VAPOR CONCENTRATIONS CAUSED SLIGHT LACRIMATION.

FIRST AID- WASH EYES IMMEDIATELY WITH LARGE AMOUNTS OF WATER, OCCASIONALLY LIFTING UPPER AND LOWER LIDS, UNTIL NO EVIDENCE OF CHEMICAL REMAINS (AT LEAST 15-20 MINUTES). CONTINUE IRRIGATING WITH NORMAL SALINE UNTIL THE PH HAS RETURNED TO NORMAL (30-60 MINUTES). COVER WITH STERILE BANDAGES. GET MEDICAL ATTENTION IMMEDIATELY.

INGESTION:
HYDROGEN FLUORIDE:
CORROSIVE.
ACUTE EXPOSURE- INGESTION MAY CAUSE BURNS OF THE MOUTH, ESOPHAGUS, STOMACH AND SMALL INTESTINE WITH GASTRITIS, GASTRIC HEMORRHAGES, VOMITING, NAUSEA, ABDOMINAL PAIN, AND DIARRHEA. LARGE DOSES MAY CAUSE EXTENSIVE NECROSIS WITH PERFORATION OF THE STOMACH, SHOCK AND DEATH. SYSTEMIC POISONING MAY CAUSE HYPOGLYCEMIA, HYPERKALEMIA, HYPOMAGNESEMIA, AND SEVERE HYPOCALCEMIA RESULTING IN TETANY, ESPECIALLY OF THE EXTREMITIES, AND PARESTHESIAS. HYPOTENSION, CIRCULATORY SHOCK AND CARDIAC ARRHYTHMIAS INCLUDING SINUS TACHYCARDIA OR VENTRICULAR FIBRILLATION, SOMETIMES PRECEDED BY TACHYCARDIA, MAY OCCUR. CENTRAL NERVOUS SYSTEM SYMPTOMS MAY INCLUDE HEADACHE, EXCESSIVE SALIVATION, NYSTAGMUS AND DILATED PUPILS, LETHARGY, STUPOR, COMA, AND RARELY, TRANSIENT CONVULSIONS. DEATH IS USUALLY DUE TO RESPIRATORY PARALYSIS OR CARDIAC FAILURE. IN NON-FATAL CASES, JAUNDICE AND KIDNEY DAMAGE WITH ALBUMINURIA, HEMATURIA, OLIGURIA OR ANURIA MAY OCCUR, BUT ARE GENERALLY REVERSIBLE. ASPIRATION MAY LEAD TO CHEMICAL PNEUMONITIS.
CHRONIC EXPOSURE- CHRONIC INGESTION OF SMALL AMOUNTS MAY CAUSE FLUOROSIS WITH OSTEOSCLEROTIC THICKENING WITH CALCIFICATION IN LIGAMENTOUS ATTACHMENTS OF SKELETON, WEIGHT LOSS, BRITTLENESS OF BONES, REDUCED BONE MARROW SPACE WITH ANEMIA, WEAKNESS, GENERAL ILL HEALTH, STIFFNESS OF JOINTS, AND DISCOLORATION OF DEVELOPING TEETH. RARELY, CENTRAL NERVOUS SYSTEM INVOLVEMENT OCCURS.

FIRST AID- DO NOT USE GASTRIC LAVAGE OR EMESIS. DILUTE THE ACID IMMEDIATELY BY DRINKING LARGE QUANTITIES OF WATER OR MILK. IF VOMITING PERSISTS, ADMINISTER FLUIDS REPEATEDLY. INGESTED ACID MUST BE DILUTED APPROXIMATELY

100 FOLD TO RENDER IT HARMLESS TO TISSUES. MAINTAIN AIRWAY AND TREAT SHOCK (DREISBACH, HANDBOOK OF POISONING, 12TH ED.). GET MEDICAL ATTENTION IMMEDIATELY. IF VOMITING OCCURS, KEEP HEAD BELOW HIPS TO HELP PREVENT ASPIRATION.

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ANTIDOTE:
NO SPECIFIC ANTIDOTE. TREAT SYMPTOMATICALLY AND SUPPORTIVELY.

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REACTIVITY

REACTIVITY:
REACTS EXOTHERMICALLY WITH WATER OR STEAM WITH THE RELEASE OF TOXIC AND CORROSIVE FUMES.

INCOMPATIBILITIES:
HYDROGEN FLUORIDE:
- ACETIC ANHYDRIDE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- 2-AMINOETHANOL: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- AMMONIUM HYDROXIDE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- ARSENIC TRIOXIDE: INCANDESCENT REACTION.
- BISMUTHIC ACID: VIOLENT REACTION EVOLVING OZONISED OXYGEN.
- CALCIUM OXIDE: VERY VIOLENT REACTION WITH INCANDESCENCE.
- CHLOROSULFURIC ACID: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- COATINGS: ATTACKED.
- CONCRETE: ATTACKED.
- CYANOGEN FLUORIDE: EXPLOSIVE POLYMERIZATION REACTION.
- DIPHOSPHOROUS PENTOXIDE: VIOLENT REACTION.
- ETHYLENEDIAMINE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- ETHYLENEIMINE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- FLUORINE: ENERGETIC REACTION WITH IGNITION.
- GLASS: ATTACKED.
- LEATHER: ATTACKED.
- METALS: MAY GENERATE FLAMMABLE HYDROGEN GAS UPON CONTACT.
- MERCURY OXIDE: EXOTHERMIC REACTION UNLESS ADEQUATE COOLING KEEPS REACTION TEMPERATURE BELOW 0 C.
- METHANESULFONIC ACID: ELECTROLYSIS OF A MIXTURE PRODUCED OXYGEN DIFLUORIDE WHICH EXPLODED.
- NITRIC ACID: IGNITION.
- NITRIC ACID + GLYCEROL: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- NITRIC ACID + LACTIC ACID: UNSTABLE MIXTURE.
- NITRIC ACID, PROPYLENE GLYCOL, AND SILVER NITRATE: UNSTABLE MIXTURE.
- OLEUM: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- ORGANIC MATERIALS: ATTACKED.
- N-PHENYLAZOPIPERIDINE: VIOLENT REACTION.
- PHOSPHORUS(V) OXIDE: VIGOROUS REACTION BELOW 20 C.
- PLASTICS: ATTACKED.
- POTASSIUM PERMANGANATE: VIOLENT, EXOTHERMIC REACTION WITH CONCENTRATED ACID.
- POTASSIUM TETRAFLUORISILICATE: VIOLENT EVOLUTION OF SILICON TETRAFLUORIDE.
- PROPIOLACTONE (BETA): TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- PROPLYLENE OXIDE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
- RUBBER (NATURAL): ATTACKED.
- SILICA CONTAINING MATERIALS: CORROSIVE.
- SODIUM: REACTS WITH EXPLOSIVE VIOLENCE WITH AQUEOUS ACID.
- SODIUM HYDROXIDE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.

SULFURIC ACID: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.
TETRAFLUOROSILICIC ACID: VIOLENT REACTION.
VINYL ACETATE: TEMPERATURE AND PRESSURE INCREASE IN A CLOSED CONTAINER.

DECOMPOSITION:
THERMAL DECOMPOSITION MAY RELEASE CORROSIVE HYDROGEN FLUORIDE.

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POLYMERIZATION:
HAZARDOUS POLYMERIZATION HAS NOT BEEN REPORTED TO OCCUR UNDER NORMAL TEMPERATURES AND PRESSURES.

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## STORAGE AND DISPOSAL

OBSERVE ALL FEDERAL, STATE AND LOCAL REGULATIONS WHEN STORING OR DISPOSING OF THIS SUBSTANCE. FOR ASSISTANCE, CONTACT THE DISTRICT DIRECTOR OF THE ENVIRONMENTAL PROTECTION AGENCY.

**STORAGE**

PROTECT AGAINST PHYSICAL DAMAGE. STORE IN WELL-VENTILATED AREA, SEPARATED FROM OTHER STORAGE (NFPA 49, HAZARDOUS CHEMICALS DATA, 1975).

STORE AWAY FROM INCOMPATIBLE SUBSTANCES.

**DISPOSAL**

DISPOSAL MUST BE IN ACCORDANCE WITH STANDARDS APPLICABLE TO GENERATORS OF HAZARDOUS WASTE, 40 CFR 262. EPA HAZARDOUS WASTE NUMBER D002.

100 POUND CERCLA SECTION 103 REPORTABLE QUANTITY.

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## CONDITIONS TO AVOID

MAY BURN BUT DOES NOT IGNITE READILY. MAY IGNITE COMBUSTIBLES (WOOD, PAPER, OIL, ETC.).

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## SPILL AND LEAK PROCEDURES

SOIL SPILL:
DIG HOLDING AREA SUCH AS LAGOON, POND OR PIT FOR CONTAINMENT.

DIKE FLOW OF SPILLED MATERIAL USING SOIL OR SANDBAGS OR FOAMED BARRIERS SUCH AS POLYURETHANE OR CONCRETE.

USE CEMENT POWDER OR FLY ASH TO ABSORB LIQUID MASS.

NEUTRALIZE SPILL WITH SLAKED LIME, SODIUM BICARBONATE OR CRUSHED LIMESTONE.

AIR SPILL:
APPLY WATER SPRAY TO KNOCK DOWN AND REDUCE VAPORS. KNOCK-DOWN WATER IS CORROSIVE AND TOXIC AND SHOULD BE DIKED FOR CONTAINMENT.

WATER SPILL:
NEUTRALIZE WITH AGRICULTURAL LIME, SLAKED LIME, CRUSHED LIMESTONE, OR SODIUM BICARBONATE.

NEUTRALIZE WITH CAUSTIC SODA.

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ADD SUITABLE AGENT TO NEUTRALIZE SPILLED MATERIAL TO PH-7.

USE MECHANICAL DREDGES OR LIFTS TO EXTRACT IMMOBILIZED MASSES OF POLLUTION AND PRECIPITATES.

OCCUPATIONAL SPILL:
DO NOT TOUCH SPILLED MATERIAL. STOP LEAK IF YOU CAN DO IT WITHOUT RISK. USE WATER SPRAY TO REDUCE VAPORS. FOR SMALL SPILLS, TAKE UP WITH SAND OR OTHER ABSORDENT MATERIAL AND PLACE INTO CONTAINERS FOR LATER DISPOSAL. FOR LARGER SPILLS, DIKE SPILL FOR LATER DISPOSAL. KEEP UNNECESSARY PEOPLE AWAY. ISOLATE HAZARD AREA AND DENY ENTRY.

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PROTECTIVE EQUIPMENT

VENTILATION:
PROCESS ENCLOSURE RECOMMENDED TO MEET PUBLISHED EXPOSURE LIMITS.

RESPIRATOR:
THE FOLLOWING RESPIRATORS AND MAXIMUM USE CONCENTRATIONS ARE RECOMMENDATIONS BY THE U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES, NIOSH POCKET GUIDE TO CHEMICAL HAZARDS; NIOSH CRITERIA DOCUMENTS OR BY THE U.S. DEPARTMENT OF LABOR, 29 CFR 1910 SUBPART Z.
THE SPECIFIC RESPIRATOR SELECTED MUST BE BASED ON CONTAMINATION LEVELS FOUND IN THE WORK PLACE, MUST NOT EXCEED THE WORKING LIMITS OF THE RESPIRATOR AND BE JOINTLY APPROVED BY THE NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH AND THE MINE SAFETY AND HEALTH ADMINISTRATION (NIOSH-MSHA).

HYDROGEN FLUORIDE:

30 PPM- ANY SUPPLIED-AIR RESPIRATOR
ANY SELF-CONTAINED BREATHING APPARATUS.
ANY POWERED AIR-PURIFYING RESPIRATOR WITH CARTRIDGE(S) PROVIDING PROTECTION AGAINST HYDROGEN FLUORIDE.
ANY CHEMICAL CARTRIDGE RESPIRATOR WITH CARTRIDGE(S) PROVIDING PROTECTION AGAINST HYDROGEN FLUORIDE.
ANY AIR-PURIFYING FULL FACEPIECE RESPIRATOR (GAS MASK) WITH A CHIN-STYLE OR FRONT- OR BACK-MOUNTED CANISTER PROVIDING PROTECTION AGAINST HYDROGEN FLUORIDE.

ESCAPE- ANY AIR-PURIFYING FULL FACEPIECE RESPIRATOR (GAS MASK) WITH A CHIN -STYLE OR FRONT- OR BACK-MOUNTED CANISTER PROVIDING PROTECTION AGAINST HYDROGEN FLUORIDE.
ANY APPROPRIATE ESCAPE-TYPE SELF-CONTAINED BREATHING APPARATUS.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

SELF-CONTAINED BREATHING APPARATUS WITH FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

SUPPLIED-AIR RESPIRATOR WITH FULL FACEPIECE AND OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE IN COMBINATION WITH AN AUXILIARY

SELF-CONTAINED BREATHING APPARATUS OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

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CLOTHING:
EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE (IMPERVIOUS) CLOTHING AND EQUIPMENT TO PREVENT ANY POSSIBILITY OF SKIN CONTACT WITH THIS SUBSTANCE.

GLOVES:
EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE GLOVES TO PREVENT CONTACT WITH THIS SUBSTANCE.

EYE PROTECTION:
EMPLOYEE MUST WEAR SPLASH-PROOF OR DUST-RESISTANT SAFETY GOGGLES AND A FACESHIELD TO PREVENT CONTACT WITH THIS SUBSTANCE.

EMERGENCY WASH FACILITIES:
WHERE THERE IS ANY POSSIBILITY THAT AN EMPLOYEE'S EYES AND/OR SKIN MAY BE EXPOSED TO THIS SUBSTANCE, THE EMPLOYER SHOULD PROVIDE AN EYE WASH FOUNTAIN AND QUICK DRENCH SHOWER WITHIN THE IMMEDIATE WORK AREA FOR EMERGENCY USE.

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AUTHORIZED BY- SPECTRUM CHEMICAL MFG. CORP.
CREATION DATE: 03/08/89 REVISION DATE: 11/02/89

---------- MATERIAL SAFETY DATA SHEET ----------

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MSDS # 039275

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PRODUCT #: Z6759 NAME: HYDROCHLORIC ACID REAGENT
MATERIAL SAFETY DATA SHEET, Valid 2/96 - 4/96
Printed Thursday, February 01, 1996 10:50AM

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MSDS # 036338

SECTION 1. - - - - - - - - - CHEMICAL IDENTIFICATION- - - - - - - - - -
CATALOG #: Z6759
NAME: HYDROCHLORIC ACID REAGENT
SECTION 2. - - - - - - COMPOSITION/INFORMATION ON INGREDIENTS - - - - - -
CAS #: 7647-01-0
EC NO: 231-595-7
SYNONYMS
ACIDE CHLORHYDRIQUE (FRENCH) * ACIDO CLORIDRICO (ITALIAN) * ANHYDROUS HYDROCHLORIC ACID * CHLOORWATERSTOF (DUTCH) * CHLOROHYDRIC ACID * CHLOROWODOR (POLISH) * CHLORWASSERSTOFF (GERMAN) * HYDROCHLORIC ACID, SOLUTION (UN1789) (DOT) * HYDROCHLORIDE * HYDROGEN CHLORIDE (ACGIH: OSHA) * HYDROGEN CHLORIDE, ANHYDROUS (UN1050) (DOT) * HYDROGEN CHLORIDE, REFRIGERATED LIQUID (UN2186) (DOT) * MURIATIC ACID * SPIRITS OF SALT * UN1050 (DOT) * UN1789 (DOT) * UN2186 (DOT) *
SECTION 3. - - - - - - - - - - HAZARDS IDENTIFICATION - - - - - - - - -
LABEL PRECAUTIONARY STATEMENTS
CORROSIVE
CAUSES BURNS.
REACTS VIOLENTLY WITH WATER.
MAY DEVELOP PRESSURE.
POISON
IN CASE OF CONTACT WITH EYES, RINSE IMMEDIATELY WITH PLENTY OF WATER AND SEEK MEDICAL ADVICE.
TAKE OFF IMMEDIATELY ALL CONTAMINATED CLOTHING.
WEAR SUITABLE PROTECTIVE CLOTHING, GLOVES AND EYE/FACE PROTECTION.
DO NOT BREATHE VAPOR.
SECTION 4. - - - - - - - - - - FIRST-AID MEASURES- - - - - - - - - - -
IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES OR SKIN WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES.
ASSURE ADEQUATE FLUSHING OF THE EYES BY SEPARATING THE EYELIDS WITH FINGERS.
IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.
IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS.
CALL A PHYSICIAN IMMEDIATELY.
WASH CONTAMINATED CLOTHING BEFORE REUSE.
DISCARD CONTAMINATED SHOES.
SECTION 5. - - - - - - - - - FIRE FIGHTING MEASURES - - - - - - - - - -
EXTINGUISHING MEDIA
NONCOMBUSTIBLE.
USE EXTINGUISHING MEDIA APPROPRIATE TO SURROUNDING FIRE CONDITIONS.
SPECIAL FIREFIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.

PRODUCT #: Z6759 NAME: HYDROCHLORIC ACID REAGENT
MATERIAL SAFETY DATA SHEET, Valid 2/96 - 4/96
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USE WATER SPRAY TO COOL FIRE-EXPOSED CONTAINERS.
UNUSUAL FIRE AND EXPLOSIONS HAZARDS
EMITS TOXIC FUMES UNDER FIRE CONDITIONS.

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SECTION 6. - - - - - - - - ACCIDENTAL RELEASE MEASURES- - - - - - - - -
EVACUATE AREA.
WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
COVER WITH DRY-LIME, SAND, OR SODA ASH. PLACE IN COVERED CONTAINERS USING NON-SPARKING TOOLS AND TRANSPORT OUTDOORS.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.

SECTION 7. - - - - - - - - - - HANDLING AND STORAGE- - - - - - - - - - -
REFER TO SECTION 8.

SECTION 8. - - - - - - EXPOSURE CONTROLS/PERSONAL PROTECTION- - - - - -
CHEMICAL SAFETY GOGGLES.
SAFETY SHOWER AND EYE BATH.
FACESHIELD (8-INCH MINIMUM).
NIOSH/MSHA-APPROVED RESPIRATOR IN NONVENTILATED AREAS AND/OR FOR EXPOSURE ABOVE THE ACGIH TLV.
MECHANICAL EXHAUST REQUIRED.
RUBBER GLOVES.
AVOID BREATHING VAPOR.
DO NOT GET IN EYES, ON SKIN, ON CLOTHING.
AVOID PROLONGED OR REPEATED EXPOSURE.
WASH THOROUGHLY AFTER HANDLING.
CORROSIVE.
POISON
KEEP TIGHTLY CLOSED.
MAY DEVELOP PRESSURE.
REACTS VIOLENTLY WITH WATER.
STORE IN A COOL DRY PLACE.

SECTION 9. - - - - - - - PHYSICAL AND CHEMICAL PROPERTIES - - - - - - - -
APPEARANCE AND ODOR
COLORLESS LIQUID
PHYSICAL PROPERTIES

| | |
|---|---|
| FLASHPOINT | NONE |
| VAPOR PRESSURE: | 3.23PSI 21.1 C 7.93PSI 37.7 C |
| SPECIFIC GRAVITY: | 1.200 |
| VAPOR DENSITY: | 1.3 |

SECTION 10. - - - - - - - - -STABILITY AND REACTIVITY - - - - - - - - -
INCOMPATIBILITIES
BASES
AMINES
ALKALI METALS
COPPER, COPPER ALLOYS
ALUMINUM
CORRODES STEEL
DO NOT ALLOW WATER TO ENTER CONTAINER BECAUSE OF VIOLENT REACTION.
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
TOXIC FUMES OF:
HYDROGEN CHLORIDE GAS

SECTION 11. - - - - - - - - - TOXICOLOGICAL INFORMATION - - - - - - - -
ACUTE EFFECTS
MAY BE FATAL IF INHALED, SWALLOWED, OR ABSORBED THROUGH SKIN.
CAUSES BURNS.

PRODUCT #: Z6759 NAME: HYDROCHLORIC ACID REAGENT
MATERIAL SAFETY DATA SHEET, Valid 2/96 - 4/96
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MATERIAL IS EXTREMELY DESTRUCTIVE TO TISSUE OF THE MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT, EYES AND SKIN.
INHALATION MAY BE FATAL AS A RESULT OF SPASM, INFLAMMATION AND EDEMA OF THE LARYNX AND BRONCHI, CHEMICAL PNEUMONITIS AND PULMONARY EDEMA. SYMPTOMS OF EXPOSURE MAY INCLUDE BURNING SENSATION, COUGHING, WHEEZING, LARYNGITIS, SHORTNESS OF BREATH, HEADACHE, NAUSEA AND VOMITING.

RTECS #: MW4025000
HYDROCHLORIC ACID

MSDS # 036338

IRRITATION DATA
EYE-RBT 5 MG/30S RINSE MLD TXCYAC 23,281,82

TOXICITY DATA

| | |
|---|---|
| IHL-HMN LCLO:1300 PPM/30M | 29ZWAE -,207,68 |
| IHL-HMN LCLO:3000 PPM/5M | TABIA2 3,231,33 |
| UNR-MAN LDLO:81 MG/KG | 85DCAI 2,73,70 |
| IHL-RAT LC50:3124 PPM/1H | AMRL** TR-74-78,74 |
| IHL-MUS LC50:1108 PPM/1H | JCTODH 3,61,76 |
| IPR-MUS LD50:1449 MG/KG | COREAF 256,1043,63 |
| ORL-RBT LD50:900 MG/KG | BIZEA2 134,437,23 |

TARGET ORGAN DATA
SENSE ORGANS AND SPECIAL SENSES (OTHER EYE EFFECTS)
LUNGS, THORAX OR RESPIRATION (RESPIRATORY STIMULATION)
SKIN AND APPENDAGES (AFTER SYSTEMIC EXPOSURE: DERMATITIS, OTHER)
ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATION.

SECTION 12. - - - - - - - - - - ECOLOGICAL INFORMATION - - - - - - - - - -
DATA NOT YET AVAILABLE.

SECTION 13. - - - - - - - - - - DISPOSAL CONSIDERATIONS - - - - - - - - - -
FOR SMALL QUANTITIES: CAUTIOUSLY ADD TO A LARGE STIRRED EXCESS OF WATER. ADJUST THE PH TO NEUTRAL, SEPARATE ANY INSOLUBLE SOLIDS OR LIQUIDS AND PACKAGE THEM FOR HAZARDOUS-WASTE DISPOSAL. FLUSH THE AQUEOUS SOLUTION DOWN THE DRAIN WITH PLENTY OF WATER. THE HYDROLYSIS AND NEUTRALIZATION REACTIONS MAY GENERATE HEAT AND FUMES WHICH CAN BE CONTROLLED BY THE RATE OF ADDITION.
OBSERVE ALL FEDERAL, STATE AND LOCAL ENVIRONMENTAL REGULATIONS.

SECTION 14. - - - - - - - - - - TRANSPORT INFORMATION - - - - - - - - - -
CONTACT SIGMA CHEMICAL COMPANY FOR TRANSPORTATION INFORMATION.

SECTION 15. - - - - - - - - - - REGULATORY INFORMATION - - - - - - - - - -
EUROPEAN INFORMATION
EC INDEX NO: 017-002-00-2
CORROSIVE
R 34
CAUSES BURNS.
R 14
REACTS VIOLENTLY WITH WATER.
S 26
IN CASE OF CONTACT WITH EYES, RINSE IMMEDIATELY WITH PLENTY OF WATER AND SEEK MEDICAL ADVICE.
S 27
TAKE OFF IMMEDIATELY ALL CONTAMINATED CLOTHING.
S 36/37/39
WEAR SUITABLE PROTECTIVE CLOTHING, GLOVES AND EYE/FACE PROTECTION.

PRODUCT #: Z6759 NAME: HYDROCHLORIC ACID REAGENT
MATERIAL SAFETY DATA SHEET, Valid 2/96 - 4/96
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S 23
DO NOT BREATHE VAPOR.
REVIEWS, STANDARDS, AND REGULATIONS
OEL=MAK
ACGIH TLV-CL 5 PPM 85INA8 6,773,91
IARC CANCER REVIEW:HUMAN INADEQUATE EVIDENCE IMEMDT 54,189,92
IARC CANCER REVIEW:ANIMAL INADEQUATE EVIDENCE IMEMDT 54,189,92
IARC CANCER REVIEW:GROUP 3 IMEMDT 54,189,92
EPA FIFRA 1988 PESTICIDE SUBJECT TO REGISTRATION OR RE-REGISTRATION
FEREAC 54,7740,89
MSHA STANDARD:AIR-CL 5 PPM (7 MG/M3)
DTLVS* 3,129,71
OSHA PEL (GEN INDU):CL 5 PPM (7 MG/M3)
CFRGBR 29,1910.1000,94
OSHA PEL (CONSTRUC):CL 5 PPM (7 MG/M3)
CFRGBR 29,1926.55,94
OSHA PEL (SHIPYARD):CL 5 PPM (7 MG/M3)
CFRGBR 29,1915.1000,93
OSHA PEL (FED CONT):CL 5 PPM (7 MG/M3)
CFRGBR 41,50-204.50,94
OEL-AUSTRALIA:TWA 5 PPM (7 MG/M3) JAN93
OEL-AUSTRIA:TWA 5 PPM (7 MG/M3) JAN93
OEL-BELGIUM:STEL 5 PPM (7.7 MG/M3) JAN93
OEL-DENMARK:STEL 5 PPM (7 MG/M3) JAN93
OEL-FINLAND:STEL 5 PPM (7 MG/M3);SKIN JAN93
OEL-FRANCE:STEL 5 PPM (7.5 MG/M3) JAN93
OEL-GERMANY:TWA 5 PPM (7 MG/M3) JAN93
OEL-HUNGARY:STEL 5 MG/M3 JAN93
OEL-JAPAN:STEL 5 PPM (7.5 MG/M3) JAN93
OEL-THE NETHERLANDS:TWA 5 PPM (7 MG/M3) JAN93
OEL-THE PHILIPPINES:TWA 5 PPM (7 MG/M3) JAN93
OEL-POLAND:TWA 5 MG/M3 JAN93
OEL-RUSSIA:STEL 5 PPM (5 MG/M3) JAN93
OEL-SWEDEN:STEL 5 PPM (8 MG/M3) JAN93
OEL-SWITZERLAND:TWA 5 PPM (7.5 MG/M3);STEL 10 PPM (15 MG/M3) JAN93
OEL-THAILAND:TWA 5 PPM (7 MG/M3) JAN93
OEL-TURKEY:TWA 5 PPM (7 MG/M3) JAN93
OEL-UNITED KINGDOM:TWA 5 PPM (7 MG/M3);STEL 5 PPM (7 MG/M3) JAN93
OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA CHECK ACGIH TLV
OEL IN NEW ZEALAND, SINGAPORE, VIETNAM CHECK ACGIH TLV
NIOSH REL TO HYDROGEN CHLORIDE-AIR:CL 5 PPM
NIOSH* DHHS #92-100,92
NOHS 1974: HZD 38580; NIS 360; TNF 87434; NOS 156; TNE 824985
NOES 1983: HZD 38580; NIS 321; TNF 60309; NOS 183; TNE 1238572; TFE 388130
EPA GENETOX PROGRAM 1988, NEGATIVE: CELL TRANSFORM.-SA7/SHE
EPA TSCA SECTION 8(B) CHEMICAL INVENTORY
EPA TSCA SECTION 8(D) UNPUBLISHED HEALTH/SAFETY STUDIES
EPA TSCA SECTION 8(E) RISK NOTIFICATION, 8EHQ-0892-9246
ON EPA IRIS DATABASE
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, OCTOBER 1995
NIOSH ANALYTICAL METHOD, 1994: ACIDS, INORGANIC, 7903
U.S. INFORMATION
THIS PRODUCT IS SUBJECT TO SARA SECTION 313 REPORTING REQUIREMENTS.

MSDS # 036338

PRODUCT #: Z6759 NAME: HYDROCHLORIC ACID REAGENT
MATERIAL SAFETY DATA SHEET, Valid 2/96 - 4/96
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SECTION 16. - - - - - - - - - - OTHER INFORMATION- - - - - - - - - - -
THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. SIGMA, ALDRICH, FLUKA SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITIONS OF SALE.


MSDS # 036338

# Material Safety Data Sheet

May be used to comply with OSHA's Hazard Communication Standard, 29CFR 1910. 1200. Standard must be consulted for specific requirements.

Cesium Ion Exchange Resin
QUICK IDENTIFIER
Common Name: (used on label and list)

RF Resin
(BSC-187)

SECTION 1 -

Manufacturer's Name: BOULDER SCIENTIFIC COMPANY

Address: P.O. BOX 548 598 THIRD STREET

City, State, and ZIP: MEAD, CO 80542

Emergency Telephone No.: 303-442-1199

Other Information Calls: 303-535-4494

Signature of Person Responsible for Preparation (Optional)

Date Prepared: Revised 6/14/93

SECTION 2 - HAZARDOUS INGREDIENTS/IDENTITY

| Hazardous Component(s) (chemical & common name(s)) | OSHA PEL | ACGIH TLV | Other Exposure Limits | % (optional) | CAS NO. |
|---|---|---|---|---|---|
| NONHAZARDOUS -- TOTALLY CROSSLINKED RESORCINOL FORMALDEHYDE RESIN | | | | | |

MSDS # 023312

SECTION 3 - PHYSICAL & CHEMICAL CHARACTERISTICS

Boiling Point: DOES NOT BOIL

Specific Gravity (H2O = 1): 0.7

Vapor Pressure (mm Hg): NONE

Vapor Density (Air = 1): NONE

Solubility in Water: NONE

Reactivity in Water: NONE

Appearance and Odor: RED AMORPHOUS SOLID, NO ODOR

Melting Point: NONE

SECTION 4 - FIRE & EXPLOSION DATA

Flash Point: NONE C.

Method Used:

Flammable Limits in Air % by Volume: LEL Lower: NONE UEL Upper: NONE

Auto-Ignition Temperature: NOT AVAILABLE

Extinguisher Media: DRY CHEMICAL, CARBON DIOXIDE, WATER

Special Fire Fighting Procedures: DUSTY CONDITIONS MAY CREATE DUST EXPLOSION HAZARD

Unusual Fire and Explosion Hazards: REQUIRES SUSTAINED FLAME TO MAINTAIN IGNITION

NONFLAMMABLE

COMBUSTIBLE

## SECTION 5 - PHYSICAL HAZARDS (REACTIVITY DATA)

Stability: Unstable ☐ Stable ☒ Conditions to Avoid

Incompatability (Materials to Avoid): STRONG OXIDIZING ACID

MSDS # 023312

Hazardous Decomposition Products: CARBON MONOXIDE, CARBON DIOXIDE

Hazardous Polymerization: May Occur ☐ Will Not Occur ☒ Conditions to Avoid

## SECTION 6 - HEALTH HAZARDS

1. Acute 2. Chronic

HARMFUL IF INHALED OR SWALLOWED

Signs and Symptoms of Exposure: MAY BE HARMFUL TO MUCOUS MEMBRANES AND RESPIRATORY TRACT.

PROLONGED EFFECT NOT KNOWN.

Medical Conditions Generally Aggravated by Exposure:

Chemical Listed as Carcinogen or Potential Carcinogen: National Toxicology Program Yes ☐ No ☒; I.A.R.C. Monographs Yes ☐ No ☒; OSHA Yes ☐ No ☒

Emergency and First Aid Procedures: WASH WELL WITH SOAP AND WATER AFTER CONTACT.

ROUTES OF ENTRY

1. Inhalation: DUST HAZARDOUS
2. Eyes: NONE
3. Skin: NONE
4. Ingestion: DUST HAZARDOUS

## SECTION 7 - SPECIAL PRECAUTIONS AND SPILL/LEAK PROCEDURES

Precautions to be Taken in Handling and Storage: WEAR RESPIRATOR WHILE HANDLING, GRINDING, ETC.

Other Precautions: WASH AFTER HANDLING. DO NOT BREATHE DUST.

Steps to be Taken in Case Material is Released or Spilled: SWEEP UP AND PUT IN ENCLOSED CONTAINER. WEAR GLOVES AND DUST MASK.

Waste Disposal Methods (Consult federal, state, and local regulations): BURN IN CHEMICAL INCINERATOR WITH AFTER BURNER AND SCRUBBER.

## SECTION 8 - SPECIAL PROTECTION INFORMATION/CONTROL MEASURES

Respiratory Protection (Specify Type): SIMPLE DUST MASK

Ventilation: Local Exhaust; Mechanical (General); Special; Other XXXX

Protective Gloves: RUBBER CHEMICAL RESISTANT

Eye Protection: SAFETY GLASSES

Other Protective Clothing or Equipment: NONE

Work/Hygienic Practices: AVOID UNNECESSARY CONTACT. WASH AFTER HANDLING.

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. BOULDER SCIENTIFIC COMPANY SHALL NOT BE HELD LIABLE FOR ANY DAMAGE OR INJURY RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT.

UOP

# Material Safety Data Sheet

## 1. EXPERIMENTAL PRODUCT AND COMPANY IDENTIFICATION



PRODUCT: UOP™ IONSIV™ Ion Exchanger Type IE-911

UOP
25 E. Algonquin Road
Des Plaines, IL 60017-5017
Telephone: 847-391-3189
FAX: 847-391-2953
Telex: 211442

Emergency Assistance
24 Hour Emergency Telephone Numbers:

| | | |
|---|---|---|
| USA: | UOP | 847/391-2123 |
| | Chemtrec | 800/424-9300 |
| Canada: | Canutec | 613/996-6666 |
| Outside USA: | Chemtrec | 202/483-7616 |

## 2. COMPOSITION

MSDS # 053329

| MATERIAL | CAS No. | ~WT% | 1995-96 ACGIH TLV-TWA (1995 OSHA PEL-TWA) |
|---|---|---|---|
| Silicon dioxide | 7631-86-9 | 15 - 45 | 10 mg/m³ as Si total dust (15 mg/m³ as Si total dust) (5 mg/m³ as Si respirable dust) |
| Titanium dioxide | 13463-67-7 | 20 - 40 | 10 mg/m³ (15 mg/m³ as respirable dust) |
| Sodium oxide | 1313-59-3 | 5 - 20 | None established |
| Trade Secret material | Trade Secret | 15 - 25 | None established |
| Trade Secret metal oxide | Trade Secret | 0 - 25 | 5 mg/m³, STEL 10 mg/m³ (5 mg/m³, STEL 10 mg/m³) |
| Aluminum oxide (non-fibrous) | 1344-28-1 | 0 - 10 | 10 mg/m³ total dust 5 mg/m³ respirable dust (15 mg/m³ total dust) (5 mg/m³ respirable dust) |
| Copper oxide | 1317-38-0 | 0 - 2 | None established |
| Calcium oxide | 1305-78-8 | 0 - 2 | 2.0 mg/m³ (5.0 mg/m³) |
| Chromium oxide | 1308-38-9 | 0 - 2 | None established |
| Magnesium oxide | 1309-48-4 | 0 - 2 | 10 mg/m³ (15 mg/m³) |

ACGIH - American Conference of Governmental Industrial Hygienists
OSHA - Occupational Safety and Health Administration
TLV - Threshold Limit Value
TWA - Time Weighted Average
PEL - Permissible Exposure Limit

## 3. HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW:

This white crystalline powder may cause skin and eye irritation. Inhalation of dust may cause respiratory irritation. Product is considered to have a low oral toxicity.

POTENTIAL HEALTH EFFECTS:

Primary Routes of Exposure: Contact with skin and eyes. Exposure may also occur via inhalation or ingestion if product dust is generated during handling. Product ingestion is unlikely but may occur if proper safety/hygiene procedures are not followed.

Skin Contact: The solid or dust may cause irritation with repeated or prolonged exposure.

Eye Contact: Solid or dust may cause irritation or reddening due to mechanical action. Mild to moderate irritation of eye membrane may also occur possibly resulting in swelling.

Inhalation: May cause irritation of the nose and throat, accompanied by cough and chest discomfort. Prolonged inhalation may cause lung damage.

Ingestion: This product is considered to have a low order of toxicity.

Carcinogenicity Classification:

IARC: Titanium dioxide and Silicon dioxide - Not classifiable as human carcinogens (Group 3). None of the other components are classified.
NTP: None of the product components are classified.
OSHA: None of the product components are classified.

MSDS # 053329

## 4. FIRST AID MEASURES

Skin: Wash affected area with soap and water. If irritation occurs, obtain medical attention.

Eyes: Flush with water for at least 15 minutes. If irritation persists, obtain medical attention.

Inhalation: Remove affected person to fresh air. If breathing is difficult, oxygen may be needed; obtain medical attention.

Ingestion: Do not induce vomiting. Victim should drink large quantities of milk, gelatin solution or water. Obtain medical attention.

## 5. FIRE FIGHTING MEASURES

Flash Point: Not applicable
Method: Not applicable
Extinguishing Media: Material does not burn. Use media appropriate for surrounding fire (carbon dioxide, dry chemical or foam).
Fire and Explosion Hazard: Used material may contain products of a hazardous nature. The user of this product must identify the hazards of the retained material and inform the fire fighters of these hazards.

## 6. ACCIDENTAL RELEASE MEASURES

Isolate the affected area; restrict entry to the affected area to personnel wearing proper personal protective equipment. Special attention should be given to respiratory and eye protection, because recovery of material can be expected to generate dust. Vacuum or shovel up spilled material, placing it into appropriate recovery drums or containers.

## 7. HANDLING AND STORAGE

Store in tightly closed, properly labeled containers. Do not take internally. Avoid repeated or prolonged contact with skin. Avoid contact with eyes and inhalation of dust.

## 8. EXPOSURE CONTROLS AND PERSONAL PROTECTION

Respiratory: Where natural ventilation is inadequate, use mechanical ventilation, other engineering controls or a toxic dust respirator (in USA NIOSH/MSHA-approved) to prevent inhalation of dust.

Skin: Chemical-resistant gloves and work uniform as necessary to prevent repeated or prolonged skin contact.

Eye: Safety glasses or goggles as necessary to prevent eye contact.

MSDS # 053329

## 9. PHYSICAL AND CHEMICAL PROPERTIES

These data do not represent technical or sales specifications.

| | | | |
|---|---|---|---|
| Boiling Point: | Not applicable | Solubility in Water: | Insoluble |
| Bulk Density: | Not available | % Volatile: | Not applicable |
| Vapor Pressure: | Not applicable | Appearance: | White Crystalline Powder |
| Vapor Density: | Not applicable | Odor: | Odorless |
| Pour Point: | Not applicable | pH in 10% aqueous slurry: | approximately 11 |
| Freezing Point: | Not applicable | Physical State: | Solid |
| Specific Gravity | Not applicable | | |

## 10. STABILITY AND REACTIVITY

| | |
|---|---|
| Stability: | Stable. |
| Conditions to Avoid: | None known. |
| Hazardous Decomposition Products: | None. |
| Hazardous Polymerization: | Will not occur. |
| Incompatible Materials: | Avoid contact with acid and easily oxidized materials. |

## 11. TOXICOLOGICAL INFORMATION

No data available for this product, the following data is for a similar product:

| | |
|---|---|
| Oral LD50 | >5 g/kg (rat) |
| Eye Irritation | The product is no more than moderately irritating to the eyes of rabbits. No corneal damage was seen. Iritis was seen in 2/6 animals after 1 hour, but none was seen after 48 hours. All 6 rabbits showed irritation of the conjunctiva after 1 hour, but after 72 hours only 1 still showed the irritation. |
| Skin Irritation | The product is no more than slightly irritating to the skin of rabbits; only barely perceptible erythema was noted at the 1-hr score time in 2/6 rabbits, score of 1 for 2/6 rabbits (maximum possible score is 8). |
| Skin Sensitization | The product was found not to produce skin sensitization in guinea pigs. |
| Cytotoxicity | The product is classified in the nondetectable category according the IERL cytotoxicity scheme (Sandhu, 1979). The product was considered to be noncytotoxic to rabbit alveolar macrophage cells at concentrations less than or equal to 1000 µg/ml. |

## 12. ECOLOGIC INFORMATION

No data currently available.

## 13. DISPOSAL CONSIDERATIONS

Dispose of product in accordance with all applicable government regulations. The unused product and its components are not listed by generic name or trademark name in the U.S. EPA's Resource Conservation and Recovery Act (RCRA) Hazardous Waste Management Regulations and do not possess any of the four identifying characteristics of hazardous waste.

## 14. TRANSPORTATION INFORMATION

MSDS # 053329

| | |
|---|---|
| DOT Hazard Classification: | Not Regulated |
| ID Number: | Not Applicable |
| IMO Hazard Classification: | Not Regulated |
| ID Number: | Not Applicable |

## 15. REGULATORY INFORMATION

U.S. TOXIC SUBSTANCES CONTROL ACT (TSCA): All the ingredients of this product are registered in accordance with TSCA.

U.S. SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (SARA) TITLE III, SECTION 313: The following component(s) in this product is subject to the reporting requirements of section 313 of the Emergency Planning and Community Right-To-Know Act, 40 CFR 372: —none—

EUROPEAN INVENTORY OF EXISTING COMMERCIAL CHEMICAL SUBSTANCES (EINECS): The components of this preparation are included on the EINECS.

| | |
|---|---|
| Silicon dioxide | 231545 4 |
| Titanium dioxide | 236675 5 |
| Sodium oxide | 215208 9 |
| Trade Secret Material | listed |
| Trade Secret Material | listed |
| Aluminum oxide | 215691 6 |
| Copper oxide | 215269 1 |
| Calcium oxide | 215138 9 |
| Chromium oxide | 215160 9 |
| Magnesium oxide | 215171 9 |

## 16. OTHER INFORMATION

For additional information concerning this product contact the following:

MSDS # 053329

For health, safety & environmental information, please contact:

Product Stewardship Manager
Health, Safety and Environmental Department
UOP
25 E. Algonquin Rd.
Des Plaines, IL 60017-5017
Telephone: (847) 391-3189
Fax: (847) 391-2953

For technical or product purchasing information, please contact:

Account Manager
UOP
307 Fellowship Road
Suite 207
Mt Laurel, New Jersey 08054
Telephone: (609) 727-9400
Fax: (609) 727-9545

### PRODUCT EMERGENCIES

If you have a product-related emergency, resulting in an incident such as a spill or release of product, human exposure, etc., and need assistance from UOP, please call us at the following number:

**24 Hour EMERGENCY Telephone Number: (847) 391-2123**

The data and recommendations presented in this data sheet concerning the use of our product and the materials contained therein are believed to be accurate and are based on information which is considered reliable as of the date hereof. However, the customer should determine the suitability of such materials for his purpose before adopting them on a commercial scale. Since the use of our products by others is beyond our control, no guarantee, express or implied, is made and no responsibility assumed for the use of this material or the results to be obtained therefrom. Information on this form is furnished for the purpose of compliance with Government Health and Safety Regulations and shall not be used for any other purposes. Moreover, the recommendations contained in this data sheet are not to be construed as a license to operate under, or a recommendation to infringe, any existing patents, nor should they be confused with state, municipal or insurance requirements, or with national safety codes.

UOP

Date: January 1998

Revision: 1

Supersedes: February 1995

# DISTRIBUTION SHEET

| To | From | Page 1 of 1 |
|---|---|---|
| Distribution | ETTP/PE | Date 05/31/96 |
| Project Title/Work Order | | EDT No. 611414 |
| Test Procedures and Instructions for Hanford Tank Waste Supernatant Cesium Removal | | ECN No. |

| Name | MSIN | Text With All Attach. | Text Only | Attach./ Appendix Only | EDT/ECN Only |
|---|---|---|---|---|---|
| Akita, R. | T6-20 | X | | | |
| Appel, J. N. | G3-21 | X | | | |
| Beck, M.A. | T6-09 | X | | | |
| Benar, C. J. | R2-12 | X | | | |
| Berger, J.D. | H6-34 | X | | | |
| Brown, G.N. | P7-25 | X | | | |
| Brown, L. C. | L5-62 | X | | | |
| Duncan, J. B. (4) | L5-55 | X | | | |
| Eberlein, S. J. | R2-12 | X | | | |
| Edmondson, D. W. | T6-09 | X | | | |
| Hall, M. J. | T6-12 | X | | | |
| Hendrickson, D. W. (8) | L5-31 | X | | | |
| Herting, D. L. | T6-09 | X | | | |
| Hunter, J.A. | L5-31 | X | | | |
| Hyatt, J. E. | S3-31 | X | | | |
| Kurath, D.E. | P7-20 | X | | | |
| Jewett, J.R. | T6-09 | X | | | |
| Jaten, K.H. | T6-04 | X | | | |
| Klem, M.J. | H5-27 | X | | | |
| Lamson, S. B. | T6-28 | X | | | |
| McDonald, G. T. | L5-31 | X | | | |
| Narquis, C. T. | T6-16 | X | | | |
| Smith, J. R. | T6-09 | X | | | |
| Stewart, T. | K9-91 | X | | | |
| TCRC (2) | R2-12 | X | | | |
| Voogd, J. A. | H5-03 | X | | | |
| Central Files (orig + 2) | A3-88 | X | | | |
| ~~DPC~~ | ~~A3-94~~ | X | | | |