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Test Procedures and Instructions for Hanford Complexant Concentrate Supernatant Cesium Removal using CST

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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 Complexant Concentrate supernatant liquor from tank 241-AN-107, in a bench-scale column. The cesium sorbent to be tested is crystalline silicotitanate. The test plan for which this provides instructions is WHC-SD-RE-TP-023, *Hanford Complexant Concentrate Supernatant Cesium Removal Test Plan.*

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Test Procedures and Instructions for Hanford Complexant Concentrate Supernatant Cesium Removal using CST

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 Complexant Concentrate Supernatant Cesium Removal Test Plan* (Hendrickson *et al.* 1996b) to ensure adequacy of conduct and collection of appropriate samples and data.

This work is funded by the U.S. Department Of Energy Office of Science and Technology Tanks Focus Area under Technology Task Plan (TTP) RL37WT42 *Cesium Flow Studies at Hanford* through Pacific Northwest National Laboratory (PNNL). This task was previously identified under TTP # RL07WT42.

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 five liters of drainable Complexant Concentrate (CC) supernatant liquor from Hanford Tank 241-AN-107 will be acquired through the sampling efforts of the TWRS Characterization Project at Hanford (Hohl 1996) and will be placed in the hot cell with the test apparatus. The CC supernatant liquor will be diluted with a dilute caustic solution to a concentration target of five molar sodium, mixed, settled, decanted, centrifuged or filtered to remove particles, and then placed in the feed tank(s) to be used as the feed for the column flow test. The test will be conducted using the checksheets and data sheets provided in Appendix A.

The test apparatus will contain a primary test column, a backup test column (in case of fouling) and two scavenging (guard) columns in series to reduce any cesium remaining in the effluent. All columns will be packed with crystalline silicotitanate (CST). 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 provided in Figure 1. Physical layout of the test apparatus is provided in Figure 2.

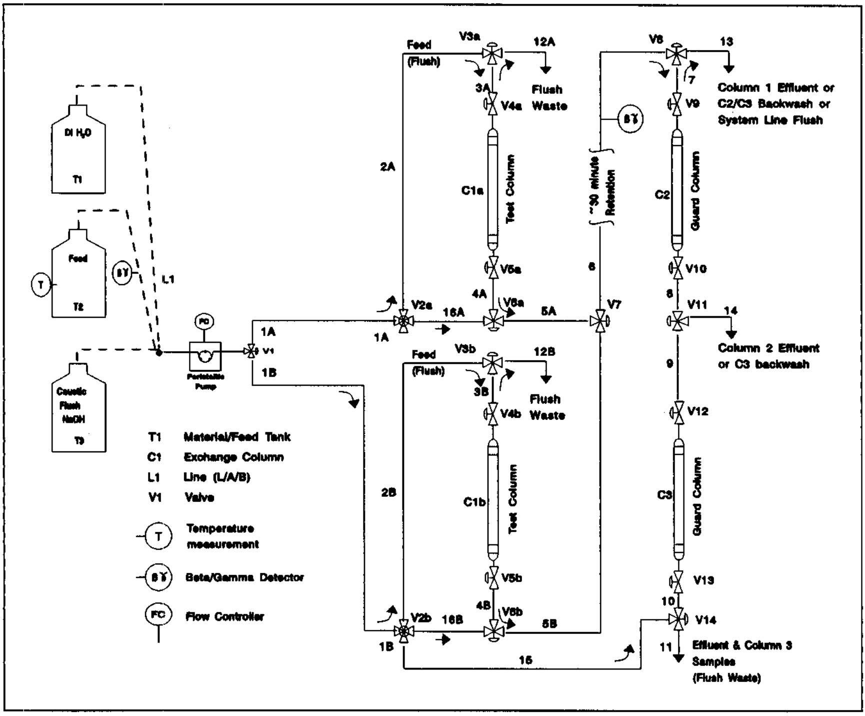

Figure 1: Bench-Scale Cesium Exchange Flows and Instrumentation

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 on-line for cesium breakthrough and will be sampled periodically. Samples will also be taken of the feed and guard column effluent, and digested sorbent as necessary. Sample analysis 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 test column, it will be flushed and drained, removed from the system, digested, sampled, and disposed. Initial plans for the spent CST 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, *et al.* 1996b).

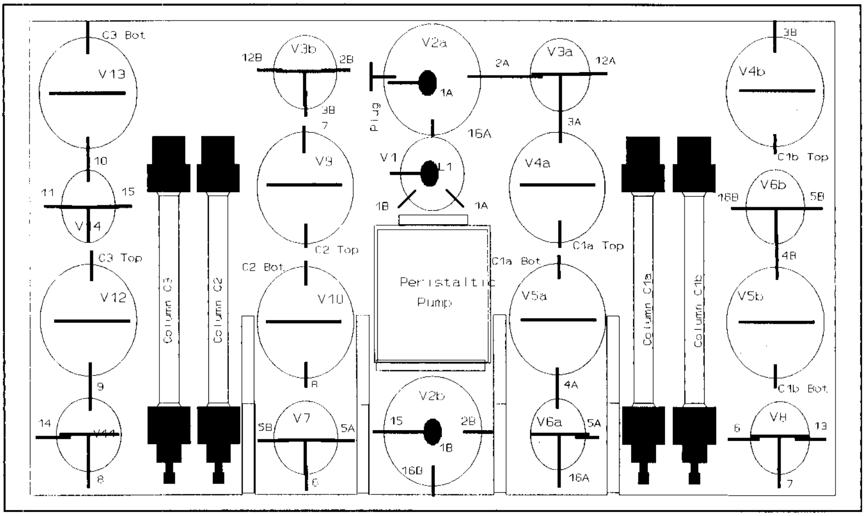

Figure 2: Physical Test Assembly Valve and Column Arrangement

The performance of the sorbents in the removal of cesium from CC waste, and process considerations of such removal, will be evaluated and reported in the test report.

2.1 TEST ENVIRONMENT

The test environment is primarily that of Hanford's 222-S analytical laboratories.

Assembly of the test equipment is largely anticipated to occur in the 306E laboratories (300 Area) followed by 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 (probably 1F) of the 222-S laboratory where the process tests will occur.

Waste material grab sample bottles, will be retrieved from tank 241-AN-107, brought to the 222-S laboratory and transferred to a hot cell for testing.

With the exception of sample analyses at the 222-S laboratory or the Battelle Pacific Northwest National Laboratory 325 laboratory, all tests will be done within a hot cell at 222-S.

2.2 MATERIAL AND EQUIPMENT NEEDS

Equipment needed for the apparatus assembly includes:

2	Exchange Columns, glass, 0.7 cm internal diameter (ID), packing height 7 cm.
2	Exchange Columns, glass, 1 cm internal diameter (ID), packing height 10 cm.
8	column clamps for exchange column retention
1	Peristaltic pump, flow range from 7 to 42 mL/hr
8	gate valves, $\sim$3 mm ID, Teflon[1] core
9	T-valves, $\sim$3 mm ID, Teflon core
2	four way valves, $\sim$3 mm ID, Teflon core
5 m	Tygon[2] tubing, 0.51mm, 0.89mm, 1.59mm, 3.2mm, and 4.8mm ID
1	Waste Feed Tank (T2), polyethylene, 3.8 L
1	Plug, predrilled, for T2, 3.8 cm diameter
4	Waste Feed Bottles, polyethylene, 1L
2	Feed Reagent Tank, polyethylene, 1 L (0.220 M NaOH, DI H_2O)
6	Effluent Receivers, polyethylene, 1 L
1	Flush receiver, glass jar, 250 mL
1	Shears for cutting large feed reagent tanks
52	Glass sample vials with lids, $\sim$20 mL
12	Centrifuge cones, glass, 50 mL
4	sample carriers
1	Continuous γ detector and recorder
1	thermometer
1	Runtime clock
1	Sample clock/stop watch
100 g	CST engineered exchange material (MSDS in Appendix B)
1	Hot/stir plate with stir bars

Reagents required (for Material Safety Data Sheets, see Appendix B), and anticipated volumes include:

Deionized Water	10.0	Liters
Sodium Hydroxide (NaOH), 5.0 M	2.0	Liters
Hydrofluoric Acid (HF), 1 M	0.125	Liter
Hydrochloric Acid (HCl), 2 M	0.25	Liter
Tank Waste, 241-AN-107	5.0	Liters

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

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Table 1: Reagent Densities at Experimental Temperature

Reagent	Molar	Density (g/mL) 27°C
HF	1	0.998
HCl	2	1.031
H_2O	55.51	0.997
NaOH	0.22	1.0060
NaOH	0.4689	1.0166
NaOH	5.0	1.1820

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 scientist 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 in the subtask. In general, the parameters of measurement, when not in chemical assay, are those of interval, volume, mass, and gamma decay. In-cell sample masses should be measured to three significant digits, runtimes should be reported in tenths of minutes, sample intervals (by stop watch) should be recorded in tenths of seconds. The subtasks to be described and executed herein are waste feed preparation exchange column packing, activation, flush, operation, sampling, and waste material handling. Chemical assay quality assurance is described by Meznarich (1995). Quality assurance for the conduct of the unit operations and hot cell activities will be in accordance with the approved process testing quality assurance plan (Meznarich 1996). 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, LabCore.

2.4 CRITERIA/CONSTRAINTS

This is a treatability test whose activities 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 *et al.* 1996b), compliance with these provisions has been met with the 1988 notification.

ALARA principles shall guide all actions in this test.

2.5 WASTE MANAGEMENT AND DISPOSAL

Wastes from the conduct of this test scope will include spent extraction media, extraction columns, sample bottles, feed bottles (tanks), tygon tubing, valves, waste liquor, treatment effluent, 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 poured down the hot cell drain. Excess and spent samples, and undispositioned effluent will be disposed per 222-S laboratory procedures. Glass vessels will be decontaminated and removed from the cell for appropriate waste disposal.

Preliminary dose models (Hendrickson and Duncan 1996) for a similar recent test 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 is not considered safe to remove these materials from the hot cell intact.

3.0 EXPECTED RESULTS

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

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

Guidance from the CST developers indicates that treatment expectation for the CST sorbent is approximately 1500 column volumes of CC waste feed to λ_{50}. Due to the uncertainty of this expectation, on-line 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 three primary subdivisions: system acceptance testing and calibration; CST test run preparation and cesium loading; system flush, necessary decontamination, and waste management. Sampling procedures are a component of each of these subdivisions of the test and are provided as a fourth component of the test.

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

Initial pump calibration and sorbent conditioning will be conducted in non-radiological facilities. Thus portions of test through conditioning (§ 4.2.1) will be conducted 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 306E 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 be followed by conditioning and loading of the exchange material in the columns per manufacturer's specifications. With the exception of § 4.1.5, acceptance testing and calibration 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. 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 CST conditioned in preparation for actual waste flow. The conditioning will be accomplished as outlined in section 4.2.1.

The polyethylene feed bottles, for deionized water (T1), waste feed (T2), and 0.220 M NaOH (caustic flush, T3), 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 0.51 mm. There will be two lines connected from V1 to the test columns, the 1A line to the primary column and the 1B line to the backup column.

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

4.1.1.2 Test Assembly

The test assembly shall be completed using Sheet 2. The function of all valve configurations are detailed in Table 2.

Table 2: Valve Connection/Function for CST Columns

Valve	After	Before	Function
V1	Peristaltic Pump	V2a	Connects pump to Test bed C1a
		V2b	Connects pump to backup Test bed C1b
V2a	V1	V3a	Directs Feed to C1a
		V6a	Directs Reverse Flush to C1a
V3a	V2a	V4a	Directs Feed to C1a
		NA	Forward feed bypass to Line 12A
	V4a	NA	C1a Reverse Flush flow to receiver
V2b	V1	V3b	Directs Feed to C1b
		V6b	Directs Reverse Flush to C1b
		V14	Guard Column Reverse Flow
V3b	V2b	V4b	Directs Feed to C1b
		NA	Forward feed bypass to Line 12B
	V4b	NA	C1b Reverse Flush flow to receiver
V4a	V3a	C1a	Column C1a isolation, forward
	C1a	V3a	Column C1a isolation, reverse
V4b	V3b	C1b	Column C1b isolation, forward
	C1b	V3b	Column C1b isolation, reverse
V5a	C1a	V6a	Column Isolation, Forward
	V6a	C1a	Column Isolation, Reverse

Table 2: Valve Connection/Function for CST Columns

Valve	After	Before	Function
V5b	C1b	V6b	Column Isolation, Forward
	V6b	C1b	Column Isolation, Reverse
V6a	V5a	V7	Directs Flow forward to V7
	V2a	V5a	Directs reverse flush to C1a
V6b	V5b	V7	Directs Flow forward to V7
	V2b	V5b	Directs reverse flush to C1b
V7	V6a	V8	Forward flow from C1a to 30 min
	V6b	V8	Forward flow from C1b to 30 min
V8	V7	V9	Directs feed to C2
	V7	NA	Directs C1a/b effluent or C2/C3
V9	V8	C2	Column C2 isolation, forward
	C2	V8	Column C2 isolation, reverse
V10	C2	V11	Column C2 isolation, forward
	V11	C2	Column C2 isolation, reverse
V11	V10	V12	Forward flow to Column C3
	V10	NA	Directs C2 effluent to receiver
	V12	V10	Directs flush to C2
V12	V11	C3	Column C3 isolation, forward
	C3	V11	Column C3 isolation, reverse
V13	C3	V14	Column C3 isolation, forward
	V14	C3	Column C3 isolation, reverse
V14	V13	NA	Column C3 effluent and samples
	V2b	V13	Directs backwash to C3
	V2b	NA	Directs flush waste to receiver

NA = Not Applicable, flow to receiver vessel

The objective of the assembly is to provide feed solutions from the feed lines through the test and guard columns to sample and effluent points.

4.1.2 Testing in Cold Environment

Using Figure 1 as a reference for flow streams and Figure 2 as reference for physical layout, the following will be carried out to cold test the system in Building 306E. Care will 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. Figures representing all valving configurations are provided in Appendix C.

Config. 1: Full System Forward Feed Valving Configuration
(Forward flow to Line 11)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V8	6 → V8 → 7
V2a/b	1A/B → V2a/b → 2A/B	V9	7 → V9 → C2
V3a/b	2A/B → V3a/b → 3A/B	V10	C2 → V10 → 8
V4a/b	3A/B → V4a/b → C1a/b	V11	8 → V11 → 9
V5a/b	C1a/b → V5a/b → 4A/B	V12	9 → V12 → C3
V6a/b	4A/B → V6a/b → 5A/B	V13	C3 → V13 → 10
V7	5A/B → V7 → 6	V14	10 → V14 → 11

Config. 2: First Guard Column Forward Feed Valving Configuration
(Forward flow to Line 14)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V7	5A/B → V7 → 6
V2a/b	1A/B → V2a/b → 2A/B	V8	6 → V8 → 7
V3a/b	2A/B → V3a/b → 3A/B	V9	7 → V9 → C2
V4a/b	3A/B → V4a/b → C1a/b	V10	C2 → V10 → 8
V5a/b	C1a/b → V5a/b → 4A/B	V11	8 → V11 → 14
V6a/b	4A/B → V6a/b → 5A/B		

Config. 3: Lead Column Forward Feed Valving Configuration
(Forward flow to Line 13)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V5a/b	C1a/b → V5a/b → 4A/B
V2a/b	1A/B → V2a/b → 2A/B	V6a/b	4A/B → V6a/b → 5A/B
V3a/b	2A/B → V3a/b → 3A/B	V7	5A/B → V7 → 6
V4a/b	3A/B → V4a/b → C1a/b	V8	6 → V8 → 13

Config. 4: CST Pump Flow Rate Calibration Valve Configuration
(Foward flush bypass to Line 12A/B)

Valve	Direction
V1	L1 → V1 → 1A/B
V2a/b	1A/B → V2a/b → 2A/B
V3a/b	2A/B → V3a/b → 12A/B

Config. 5: Column C1a/b Reverse Flush Valving Configuration
(Reverse flush to Line 12A/B)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V5A/B	4A/B → V5A/B → C1A/B
V2A/B	1A/B → V2A/B → 16A/B	V4A/B	C1A/B → V4A/B → 3A/B
V6A/B	16A/B → V6A/B → 4A/B	V3A/B	3A/B → V3A/B → 12A/B

4.1.2.1 Assembly Leak Test

The CST will not be loaded in the columns for this portion of the test. All test conduct will be recorded via indicated checksheets and datasheets.

Deionized (DI) water will be pumped through the basic assembly for this phase. The connecting lines are the required ID and length and the valving is connected as indicated in Table 2. Use Sheet 3 and place system into various configurations to check for leaks in full forward flow at middle to high set using C1a, and C1b. Images and valving configurations

Config. 6: Reverse Feed Valving Configuration
(Reverse flush through guard columns)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1B	V11	9 → V11 → 8
V2b	1B → V2b → 15	V10	8 → V10 → C2
V14	15 → V14 → 10	V9	C2 → V9 → 7
V13	10 → V13 → C3	V8	7 → V8 → 13
V12	C3 → V12 → 9		

are provided in Appendix C. The peristaltic pump does not have a digital readout or record. Calibration will be conducted with each flow rate change and recorded on Sheet 3. The flow path for calibration will be Config. 1.

4.1.2.1.1 Column Forward Tests

Use Sheet 5 to record and conduct this procedure.

- Tare receiver bottle (100 mL < receiver < 250 mL)
- Place L1 into feed vessel T1, place line 11 into tared receiver
- Set valve positions to: Config. 1
- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For this test, 6 CV/hr are targeted which is approximately 0.27 mL/min or 16.1 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.
- After the appropriate time period for leak testing in a forward flow mode, shut down the pump. Record masses collected and time interval on Sheet 3.

4.1.2.1.2 Column Reverse Flow Tests

4.1.2.1.2.1 Column C1a/C1b Reverse Flow Leak

Use Sheet 11 to record and conduct this procedure.

- Tare receiver bottle (~100 mL)
- Place line L1 in feed vessel T1, place line 12A into tared receiver
- Set valve positions to: Config. 5
- After ensuring the flow path is correct to the above valve settings, turn on the peristaltic pump. For this test, 6 CV/hr are targeted which is approximately 0.27 mL/min or 16.1 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 collected and time interval on Sheet 11.

4.1.2.1.2.2 Columns C2/C3 Reverse Flush Leak

Use Sheet 12 to record and conduct this procedure.

- Tare receiver bottle (~100 mL)
- Place line L1 into feed vessel T1, place line 13 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 0.27 mL/min or 16.1 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 12.

4.1.3 Reagent Preparation

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

NaOH, aq.	5.0 M	2.0 L
HF, aq.	1.0 M	125 mL
HCl, aq.	2.0 M	250 mL

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

4.1.4 Exchange Material Dissolution

A qualitative evaluation of the dissolution of CST has been carried out in the conduct of test work reported in Hendrickson *et al.* 1996a. It was found that the CST material substrate and binder dissolved satisfactorily with the addition of hydrochloric and hydrofluoric acids. This dissolution will be repeated in this work. Following contact of wastes with the sorbent, the material will be dissolved within the hot cell using the procedure in Sheet 13.

4.1.5 Gamma Probe Calibration

The calibration of the gamma probe will be carried out using 222-S laboratory sources, for a range of 7.4 E9 Bq/L to 7.4 E8 Bq/L (0.2 Ci/L ^{137}Cs to 0.02 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.

An "initial concentration (C_0)" standard will be made by similarly filling the tubing with feed material. Calibration responses from prepared standards will be entered into a Controlled Laboratory Notebook.

4.2 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 1500 column volumes ($\lambda_{50\%} = 1500$ CV). The run is expected to be conducted for up to 2000 column volumes, sampled every 100 column volumes for the first 1000 CV (11 samples) and every 50 CV thereafter (20 samples). Calibrated standards prepared for prior work (Hendrickson *et al.* 1996a) may be applied with appropriate decay correction.

4.2.1 Exchange Column Preparation

Both test columns, C1a and C1b, and the guard columns, C2 and C3, will be charged with CST and conditioned outside of the hot cell. After conditioning and pump calibration, the columns and system will be transferred into the hot cell. To prepare the CST, 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 14 to prepare and measure the bed density of the sorbent.

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

4.2.2 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 0.220 M NaOH. This fluid, used in sorbent conditioning, is also expected to well represent the density and viscosity of waste feed. Following the pump curve acquisition, the flow settings configuration (Config. 4) may be used to ensure adequate flow at the apparatus terminus (line 11).

Use Sheet 3 to record and conduct this procedure

- Tare receiver bottle (~250 mL)
- Place line L1 into T3. Place line 11 into tared receiver
- Set valve positions per Sheet 3

Repeat this procedure, for a total of at least three point calibration, at flow settings of approximately 10%, 40%, and 60% of pump knob range.

4.2.3 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-AN-107 during the 1997 grab sampling operations which are planned for January 1997 (Hohl 1996). The waste feed is of approximately 8.5 M Na^+ and 0.03 M OH^-. The waste feed will be diluted to a target of approximately 5 M Na with 0.47 M NaOH. Analysis of the waste composition for the feed will be carried out by gamma energy analysis, ion chromatography, TIC, TOC, and inductively coupled plasma at 222-S, in addition to inductively coupled plasma mass spectrometry conducted on similar samples acquired for privatization (DOE 1996) for speciation of cesium.

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 (§ 4.2.5.5) cannot proceed unless the material is prepared.

Use Sheet(s) 4 to record and conduct this procedure. To ensure a homogeneous feed stream throughout the run, the 1 L batches of prepared feed liquor will be composited in a 3.8 L vessel. A total of five batches will be prepared in this manner, and the excess over 3.8 L will be stored in 1 L bottles. Two feed samples will be withdrawn for analysis, one at the beginning of the test and the second after addition of the feed from the two 1 L bottles to the 3.8 L vessel.

4.2.4 System Flush

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

Use Sheet 5 to set pump with 0.22 M NaOH and Sheet 6 to record and conduct the system flush for each lead column.

Objective: 6 CV/hr = 16.1 mL/hr

Set Pump:

- Tare receiver bottle (~100 mL capacity)
- Place line L1 into T3 (0.22 M NaOH)
- Place Line 12A/B into tared receiver
- Set valve positions to: Config. 4. 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 11 into tared receiver
- Place Line L1 into T3 (0.220 M NaOH)

- Place System Valving in Config. 1 using the first available lead column. Confirm and record valving alignment.
- Turn on pump and run for 2 hours. Record time on/off, flowrate and feed source.
- Close and confirm closure of all valves.

The 0.220 M NaOH from the conditioning phase (§ 4.2.1) will have been flushed through the column and testing for ^{137}Cs removal may begin.

4.2.5 Cesium Removal Test Run

Based on modeling of recent CST test results (Hendrickson *et al.* 1996a) breakthrough (^{137}Cs $C/C_0 = 0.5$) in this test could occur as early as 800 CV. Ancillary analyses by CST developers and researchers at Sandia National Laboratory indicate that as many as 1,500 column volumes of this feed may be necessary to achieve 50% breakthrough. Therefore, this test run will schedule use of 2000 CV to ensure data past breakthrough will be obtained. The test will proceed until $C/C_0 \geq 0.7$ and will be discontinued by direction of the lead engineer or chemist.

4.2.5.1 Gamma Detector Data Logging

During the test run, the 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 (§ 4.2.5.5) and discontinue with final sample acquisition from the flow stream following the test column. Initial estimate for counting periods is 20 min.

The gamma probe must provide an additional function for this component of the test. If the response from the gamma probe indicates $C/C_0 > 0.20$ prior to 500 CV, the sample regime will accelerate to one sample per 50 CV. Sufficient detector shielding will be provided to ensure that cesium loading on columns does not adversely effect detector background. The gamma probe will be read as detailed in Gamma 1 (pg. 16). Detailed instructions for the Gamma probe operation are contained in Appendix D.

4.2.5.2 Primary Column Sampling

All samples will be recorded with column number, date time/group, sample sequence number (C1E-1, C1E-2, ...C1E-n).

It is anticipated that the CST sorbent will be exposed to 2000 CV before the test is stopped. Samples of 5 mL each will be taken at Line 12A (or 12B). At 2000 CV with a flow rate of 6 CV/h, the test will run for 333 hours. The first sample will be taken at 1 hour, representing the effluent stream front. Additional samples will be taken every 16 hours and 40 minutes (100 CV) through 1000 CV. Subsequent samples will be taken every 8 hours and 20 minutes (50 CV) through the remainder of the test. Expected samples total 31 and are detailed on Table 3. Should the on-line gamma detector indicate that the cesium C/C_0

Reading the Gamma probe:

The instrument computer (in the 19" instrument rack) should be set up with Notepad^{tm} running.

If Notepad^{tm} is not running, start it by clicking its icon in the Program manager, or Alt-TAB to switch to Notepad^{tm}

With the Notepad^{tm} window open, select OPEN from the FILE menu.

Go to the directory C:\practjob

Open the highest numbered file that starts with cs#

It should look like

cs#003.txt

with different numbers.

Read the GROSS value. Compare the Gross value with the values posted on the face of the computer or in the notebook. Note the time the file was created (at the top of the file). Record the result on Sheet 8 (if reading during a sampling) and/or on a separately provided data sheet.

If the number is 20% or greater of the original concentration value, notify the cognizant scientist. Sampling frequency may need to be increased.

If the number is 50% or greater of the original concentration value, notify the cognizant scientist. The experiment is nearly finished.

Gamma 1: Gamma Probe Reading Procedure

exceeds 20%, the sample regime shall immediately (nearest 50 CV increment) switch to the accelerated sampling structure of Table 3. 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 3. Sampling shall be conducted using Sheet 7 for sample acquisition data and Sheet 8 for process parameters of the sample.

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 columns. With the guard columns out of the flow path during sampling, it is expected that flows from the sample point will be slightly higher

Table 3: CST Test Run Primary Column Sampling

Sample	Projected Run Time
C1E-1	1 hr 0 min
C1E-2	17 hr 40 min
C1E-3	34 hr 20 min
C1E-4	51 hr 0 min
C1E-5	67 hr 40 min
C1E-6	84 hr 20 min
C1E-7	101 hr 0 min
C1E-8	117 hr 40 min
C1E-9	134 hr 20 min
C1E-10	151 hr 0 min
C1E-11	167 hr 40 min
C1E-12	176 hr 0 min
C1E-13	184 hr 20 min
C1E-14	192 hr 40 min
C1E-15	201 hr 0 min
C1E-16	209 hr 20 min
C1E-17	217 hr 40 min
C1E-18	226 hr 0 min
C1E-19	234 hr 20 min
C1E-20	242 hr 40 min
C1E-21	251 hr 0 min
C1E-22	259 hr 20 min
C1E-23	267 hr 40 min
C1E-24	276 hr 0 min
C1E-25	284 hr 20 min
C1E-26	292 hr 40 min
C1E-27	301 hr 0 min
C1E-28	309 hr 20 min
C1E-29	317 hr 40 min
C1E-30	326 hr 0 min
C1E-31	334 hr 20 min

than they would be with the added pressure drop across the guard column. Hence, the pump curves of Sheet 3 are required to appropriately monitor flow.

4.2.5.3 Secondary Column Sampling

All samples will be recorded with column number, date time/group and sample sequence number (C2E-1, C2E-2, ...C2E-n).

It is anticipated that the CST sorbent will be exposed to 2000 CV before the test is stopped. Samples of 5 mL each will be taken at Valve 11, Line 14. At 2000 CV with a flow rate of 6 CV/h, the test will run for 333 hours. The first sample will be taken at 17 hours and 10 minutes. Additional samples will be taken every 33 hours and 20 minutes (200 CV) until test completion.

Samples shall be acquired as detailed in Table 4.

Table 4: CST Test Run Secondary Column Sampling

Sample	Projected Run Time
C2E-1	17 hr 10 min
C2E-2	50 hr 30 min
C2E-3	83 hr 50 min
C2E-4	117 hr 10 min
C2E-5	150 hr 30 min
C2E-6	183 hr 50 min
C2E-7	217 hr 10 min
C2E-8	250 hr 30 min
C2E-9	283 hr 50 min
C2E-10	317 hr 10 min

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

4.2.5.4 System Effluent Sampling

The sampling from the second guard column (C3) will be of composite materials. At test initiation place a 1L container under V14/11. This volume should be sufficient for approximately 60 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 C3E-1 to C3E-n, where n is the container number (should not exceed 6).

4.2.5.5 Run Execution

Use Sheet 6 to record and conduct this procedure

Confirm or do:
Objective: 6 CV/hr = 16.1 mL/hr

- Tare receiver bottle (1L)
- Place Line 11 into 1L collection vessel
- Place line L1 into T2 (Waste Feed)
- Confirm valving for Config. 1. Confirm and record valving alignment on Sheet 6.
- Measure and record feed temperature (°C)
- Turn on pump and initiate test runtime clock. Record test initiation on Sheet 6.
- Acquire data and samples as directed above.
- Continue flow until γ probe response is beyond ~70% C/C_0.
- Immediately following last primary column sample, shut down pump, and take the composite effluent samples from the receivers filled below V14/11.
- Close and confirm closure of all valves.

4.2.6 System Flush

After the feed solution has run through the system, the system will be flushed with 0.220 M NaOH for 15 CV at 6 CV/h (2.5 hours). Allow the flush to run to the flush receiver.

Use Sheet 6 to record and conduct this procedure.

- Wash exterior of Line L1 with H_2O, place L1 in T3 (0.220 M NaOH)
- Place 11 into flush waste receiver (1L)
- Establish valving for Config. 1. 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.

4.3 SYSTEM CLEANOUT AND WASTE MANAGEMENT

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).

4.3.1 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)

4.3.2 Sorbent Columns

When the test is completed, use Sheet 13 for instructions and record of removal of spent sorbent from all the columns, followed by dissolution and disposal. No samples will be taken because digested sample analysis results for the last test were not usable. All columns should be dissolved together in one batch.

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

4.3.3 Other Liquors

Effluent materials from test conduct, acquired at line 11, 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.

4.3.4 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.

4.4 SAMPLING AND SAMPLE ANALYSES

Analytical sample selection for analyses of feed wastes, effluents, digest, and eluates are presented in Table 5. Rubidium, potassium, strontium, and cesium isotopic analyses are to be conducted by inductively coupled plasma mass spectroscopy (ICP-MS); 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 analysis (AEA), 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 gamma analysis.

Table 5: Cesium Ion Exchange Summary Sample Analysis Plan

Sample	Method	ICP-MS	ICP-AES	GEA	AEA	IC	TIC	TOC	OH-	Density	On-line γ
	Description										
Core-1	Tank Liquor	X	X	X		X					
Core-2	Tank Liquor	X	X	X		X					
F1	Waste Feed		X	X	X	X	X	X	X		
F2	Waste Feed		X	X	X	X	X	X	X		
FS1	Centrifuge slds		X	X	X						
FS2	Centrifuge slds		X	X	X						
C1E-1	C1a/b Effluent		X	X							X
C1E-2	C1a/b Effluent		X	X							X
C1E-3	C1a/b Effluent		X	X		X	X		X		X
C1E-4	C1a/b Effluent		X	X							X
C1E-5	C1a/b Effluent		X	X							X
C1E-6	C1a/b Effluent		X	X							X
C1E-7	C1a/b Effluent		X	X		X	X		X		X

Table 5: Cesium Ion Exchange Summary Sample Analysis Plan

Sample	Method	ICP-MS	ICP-AES	GEA	AEA	IC	TIC	TOC	OH-	Density	On-line γ
	Description										
C1E-8	C1a/b Effluent		X	X							X
C1E-9	C1a/b Effluent		X	X							X
C1E-10	C1a/b Effluent		X	X							X
C1E-11	C1a/b Effluent		X	X		X	X		X		X
C1E-12	C1a/b Effluent		X	X							X
C1E-13	C1a/b Effluent		X	X							X
C1E-14	C1a/b Effluent		X	X							X
C1E-15	C1a/b Effluent		X	X		X	X		X		X
C1E-16	C1a/b Effluent		X	X							X
C1E-17	C1a/b Effluent		X	X							X
C1E-18	C1a/b Effluent		X	X							X
C1E-19	C1a/b Effluent		X	X		X	X		X		X
C1E-20	C1a/b Effluent		X	X							X
C1E-21	C1a/b Effluent		X	X							X
C1E-22	C1a/b Effluent		X	X							X
C1E-23	C1a/b Effluent		X	X		X	X		X		X
C1E-24	C1a/b Effluent		X	X							X
C1E-25	C1a/b Effluent		X	X							X
C1E-26	C1a/b Effluent		X	X							X
C1E-27	C1a/b Effluent		X	X		X	X		X		X
C1E-28	C1a/b Effluent		X	X							X
C1E-29	C1a/b Effluent		X	X							X
C1E-30	C1a/b Effluent		X	X							X
C1E-31	C1a/b Effluent		X	X		X	X		X		X
C2E-1	C2 Effluent			X							
C2E-2	C2 Effluent			X							
C2E-3	C2 Effluent			X							
C2E-4	C2 Effluent			X							
C2E-5	C2 Effluent			X							
C2E-6	C2 Effluent			X							
C2E-7	C2 Effluent			X							
C2E-8	C2 Effluent			X							
C2E-9	C2 Effluent			X							
C2E-10	C2 Effluent			X							
C3E-1	Comp. C3 Effl.	X	X	X		X	X	X		X	
C3E-2	Comp. C3 Effl.		X	X	X				X		
C3E-3	Comp. C3 Effl.	X	X	X		X	X	X		X	
C3E-4	Comp. C3 Effl.		X	X	X				X		
C3E-5	Comp. C3 Effl.	X	X	X		X	X	X		X	
C3E-6	Comp. C3 Effl.		X	X	X				X		
Total Samples/Method		ICP-MS	ICP-AES	GEA	AEA	IC	TIC	TOC	OH-	Density	On-line γ
184		5	42	52	6	15	13	5	12	3	31

5.0 CHANGE PROCEDURE

5.1 General Changes

As in any experimental test conduct, the possibility of procedural revision exists. Operating personnel may institute any necessary changes with approval of the lead engineer or scientist. Such change will be documented in an engineering change notice at test completion for configuration control where necessary. All changes will be recorded in the laboratory notebook and reported with test results.

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 γ detector will be operated to report effluent activities. Should the activity exceed 20% of breakthrough prior to sampling frequency shift stated in sampling section 4.2.5.5, the operating personnel should contact the lead engineer or scientist 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 recorded in the laboratory notebook and reported with tests results.

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 § 4.2.5.5, deviate from the object by greater than 10%, the pump rate should be reset to the appropriate rate through procedures on Sheet 5. The operating personnel may institute the required change with approval of the lead engineer or chemist. Such change will be recorded in the laboratory notebook.

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 analyses. The quality assurance for the conduct of the unit operations 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

SHEET PAGE

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Sheet 1: Feed System Assembly

See Figure A-1 and Figure A-2: Checksheet#________

Assemble Line L1 as composite of 1.5 m Tygon tubing, 0.51 mm ID, fitted to 10 cm Tygon tubing, 3.175 mm ID. Connect 3.175 mm ID end of L1 to Valve V1. Mount 0.51 mm ID segment of L1 through pump head.

Next, to ensure that the feed line is situated about 1" above the bottom of the feed container, assemble a dip tube for the feed line as follows. Drill a ¼" hole and a ⅛" vent hole in a No. 6 rubber stopper (suitable for the 3.8L feed container). Position a 15½" long x ¼" diameter glass tube through the rubber stopper so that the end of the glass tube is 1" above the bottom of the feed container. For support, attach a piece of tape (as a flag) on the 0.51 mm feed line 15" from the free end and insert the tubing through the glass dip tube.

Assembly confirmed: ____________________________ Date:______________________

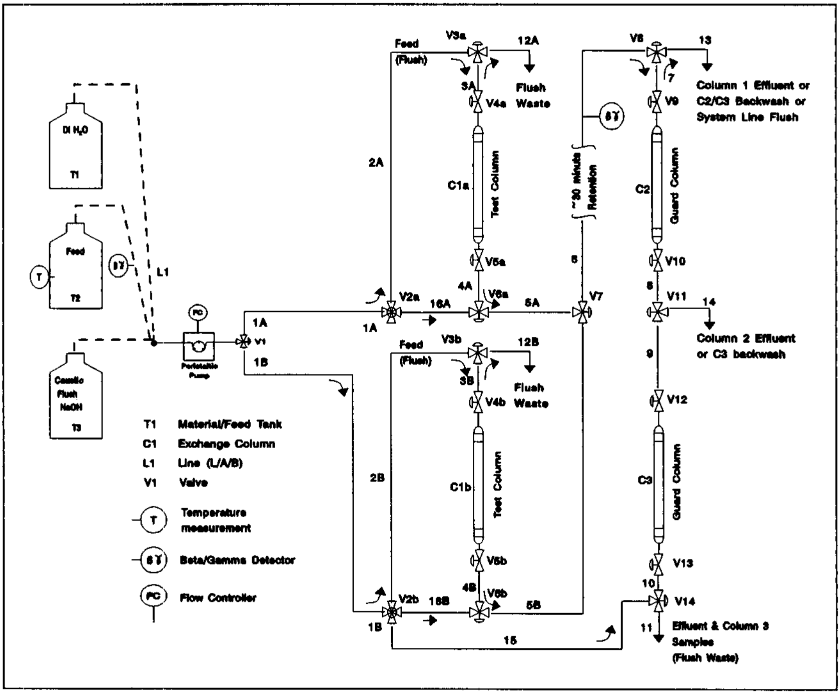

Figure A-1: Bench-Scale Cesium Exchange Flows and Instrumentation

Sheet 1: Feed System Assembly - Continued

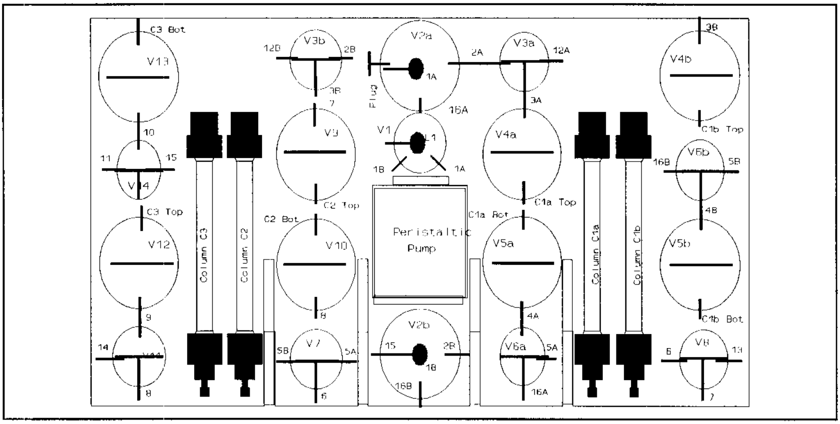

Figure A-2: Physical Test Assembly Valve and Column Arrangement

Sheet 2: Test Assembly Instructions and Checksheet

Checksheet#______________

Mount valves and columns in test assembly (See Figure A-1 for flow intent, Figure A-2 for layout).
Connect line 1A (3.2 mm ID, 20 cm Tygon[3]) to V1 and V2a.
Connect line 1B (3.2 mm ID, 39 cm Tygon) to V1 and V2b.
Connect line 2A (3.2 mm ID, 7 cm Tygon) to V2a and V3a.
Connect line 2B (3.2 mm ID, 44 cm Tygon) to V2b and V3b.
Connect line 3A (3.2 mm ID, 5 cm Tygon) to V3a and V4a.
Connect line 3B (3.2 mm ID, 43 cm Tygon) to V3b and V4b.
Connect line (Composite*) to V4a and C1a (top).
Connect line (Composite*) to V4b and C1b (top).
Connect line (Composite*) to C1a (bottom) and V5a.
Connect line (Composite*) to V5b and C1b (bottom).
Connect line 4A (3.2 mm ID, 5 cm Tygon) to V5a and V6a.
Connect line 4B (3.2 mm ID, 5 cm Tygon) to V5b and V6b.
Connect line 5A (3.2 mm ID, 28 cm Tygon) to V6a and V7.
Connect line 5B (3.2 mm ID, 71 cm Tygon) to V6b and V7.
Connect line 6 (V7 to the following composite: 3.2 mm ID, 2 cm Tygon joined to 4.8 mm ID, 60 cm Tygon joined to 3.2 mm ID, 25 cm Tygon to V8).
Connect line 7 (3.2 mm ID, 71 cm Tygon) to V8 and V9.
Connect line (Composite*) to V9 and C2 (top).
Connect line (Composite*) to V10 and C2 (bottom).
Connect line 8 (3.2 mm ID, 32 cm Tygon) to V10 and V11.
Connect line 9 (3.2 mm ID, 5 cm Tygon) to V11 and V12.
Connect line (Composite*) to V12 and C3 (top).
Connect line (Composite*) to V13 and C3 (bottom).
Connect line 10 (3.2 mm ID, 5 cm Tygon) to V13 and V14.
Connect line 15 (3.2 mm ID, 36 cm Tygon) to V2b and V14.
Connect line 12A (3.2 mm ID, 60 cm Tygon) to V3a.
Connect line 13 (3.2 mm ID, 25 cm Tygon) to V8
Connect line 14 (3.2 mm ID, 25 cm Tygon) to V11.
Connect line 11 (3.2 mm ID, 35 cm Tygon) to V14.
Connect line 12B (3.2 mm ID, 60 cm Tygon) to V3b.
Connect line 16A (3.2 mm ID, 35 cm Tygon) to V2a and V6a.
Connect line 16A (3.2 mm ID, 35 cm Tygon) to V2a and V6a.

Composite* = (3.2mm ID, 2cm Tygon, 3.2mm x 1.6mm polypropylene connector, 1.6mm ID, 2cm Tygon, 1.6mm ID, 79cm Teflon).

Assembly confirmed (init.):______________________ Date:__________________

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

Sheet 3: Pump Calibration for the CST Column Test

Objective: To compare the pump flow rate at different locations in the test apparatus. The flow rate at 12A will be plotted against effluent flow rates at lines 13, 14, and 11. This will be repeated for the flow at 12B and lines 13, 14, and 11 using 0.22 M NaOH.

Begin by adjusting the pump dial to achieve the target flow rate of 0.27 mL/min at line 11. Follow the steps below to measure the flow rate. When this desired flow rate is reached, inscribe a mark on the pump dial and record approximate radial setting. **This procedure will be carried out for two pump settings in the vicinity of the target.** Turn pump knob counterclockwise half distance to zero flow, and repeat flow rate measurement. Then turn the pump knob clockwise until the same distance to the right of the inscribed set point is reached.

Executed for Test Procedure Step: *4.2.2* Checksheet#__________

Executed by (print name): ______________________________

Run Start (Date/time): __________ Completed Date/time:________________

Prior to carrying out the procedure:

A. Place an empty 100 mL beaker on an analytical top loading balance for ease of continual weight measurements. With the aid of a laboratory stand secure discharge lines 12A, 12B, 13, 14, and 11 above this tared receiver.

B. Place line L1 into a feed bottle containing 300 mL of 0.22 M NaOH.

C. Set valve positions per Configuration 1, for C1a; all other valves shall remain closed.

D. Adjust the pump knob setting to an approximate 30° radial position from zero. Once the system has been charged with the caustic solution, begin the test procedure.

Test Procedure:

1. Turn on pump for 20 minutes; use a stop watch.
2. Turn pump off, record time of flow, weigh the receiver.
3. Record all data in the table below.
4. With the same pump setting take flow rate measurements with valves positioned per Config. 2, 3 & 4.
5. Repeat steps B thru 4 for valves positioned for flow through backup column C1b.

Sheet 3: Pump Calibration for the CST Column Test - Continued

Config.1: Full System Forward Feed Valving Configuration (Forward flow to Line 11)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V8	6 → V8 → 7
V2a/b	1A/B → V2a/b → 2A/B	V9	7 → V9 → C2
V3a/b	2A/B → V3a/b → 3A/B	V10	C2 → V10 → 8
V4a/b	3A/B → V4a/b → C1a/b	V11	8 → V11 → 9
V5a/b	C1a/b → V5a/b → 4A/B	V12	9 → V12 → C3
V6a/b	4A/B → V6a/b → 5A/B	V13	C3 → V13 → 10
V7	5A/B → V7 → 6	V14	10 → V14 → 11

Config. 2: First Guard Column Forward Feed Valving Configuration
(Forward Flow to Line 14)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V7	5A/B → V7 → 6
V2a/b	1A/B → V2a/b → 2A/B	V8	6 → V8 → 7
V3a/b	2A/B → V3a/b → 3A/B	V9	7 → V9 → C2
V4a/b	3A/B → V4a/b → C1a/b	V10	C2 → V10 → 8
V5a/b	C1a/b → V5a/b → 4A/B	V11	8 → V11 → 14
V6a/b	4A/B → V6a/b → 5A/B		

Config.3: Lead Column Forward Feed Valving Configuration (Forward Flow to Line 13)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V5a/b	C1a/b → V5a/b → 4A/B
V2a/b	1A/B → V2a/b → 2A/B	V6a/b	4A/B → V6a/b → 5A/B
V3a/b	2A/B → V3a/b → 3A/B	V7	5A/B → V7 → 6
V4a/b	3A/B → V4a/b → C1a/b	V8	6 → V8 → 13

Config. 4: CST Pump Flow Rate Calibration Valve Configuration
(Forward Flush bypass to Line 12A/B

Valve	Direction
V1	L1 → V1 → 1A/B
V2a/b	1A/B → V2a/b → 2A/B
V3a/b	2A/B → V3a/b → 12A/B

Sheet 3: Pump Calibration Flow Measurement Checksheet #

Executed By: Pump Knob Position: ____ °

Column	Discharge point (Line #)	Run 1				Run 2			
		Time	Mass, g		Rate mL/min	Time	Mass, g		Rate mL/min
			Init.	Final			Init.	Final	
C1a	11								
	14								
	13								
	12A								
C1b	11								
	14								
	13								
	12B								

Column	Discharge point (Line #)	Run 3				Avg flow rate ml/min
		Time	Mass, g		Rate mL/min	
			Init.	Final		
C1a	11					
	14					
	13					
	12A					
C1b	11					
	14					
	13					
	12B					

Sheet 4: Waste Feed Preparation

Objective: Prepare 5000 mL batch of waste feed for CST sorbent test.

Executed for Test Procedure Step: *4.2.3* Date/time:________________

Executed by (print name): ____________________________ Checksheet# ____________

1. A composite 3.8 L polyethylene jug will be used in the test. The feed material will be prepared one liter at a time until five liters are ready to begin the test. Excess feed will be stored in 1 L polyethylene bottles and added to the 3.8 L jug when 2 L of feed have been used up. The following masses have been calculated for an estimated as-received waste density of 1.43 g/mL.

2. Decant 4 waste sample bottles into a 2L glass beaker. Record bottle #s in lab notebook. Take an aliquot from this beaker and measure tank waste density,________ g/mL. Add waste from another sample bottle to total 809.7 g (566.25 mL) waste liquor in the 2L glass beaker. Actual mass added = __________ g. Actual mass added (g) / density (g/mL) =__________mL.

3. Add 455.4 g (433.75 mL) 0.4689 M NaOH solution to mixing vessel. Actual mass added____________ g.

4. Mix on magnetic stirring plate until no crystals are visible. Minimize heating during dissolution.
5. Allow to settle, decant clear liquor into feed tank T2, if solids are present then go to either step 6 or 7 before decanting into the 3.8 L vessel.

6. If solids do not settle out, do the following:
   a. Tare centrifuge cones.
   b. Centrifuge the waste material mixture, and decant clear liquid into feed tank T2.
   c. At completion, weigh all centrifuge cones to determine the accumulated solids mass.

7. If solids are present and settle out, then decant the liquid very carefully into the 3.8 L vessel taking care to minimize the amount of liquid left over. Transfer the solids into a 250 mL vial labeled "7AN Solids". Tare wt. of "7AN Solids" = _________ g.

   Repeat steps 2 thru 7 for each 1 L batch. In the lab notebook record observations of liquor clarity, color and any suspended solids.
8. Upon completion of waste feed addition to T2, pipette a 15 mL aliquot into prelabeled sample vial F1. T2 is again sampled, in vial F2, after adding 2 L waste to T2.
9. Take an additional aliquot from T2 and measure, and record density _________g/mL.
10. Dry contents of "7AN Solids". Gross Wt. ______ Net Wt.______
    Place two samples of separated solids in prelabeled sample vials FS1 and FS2.

Sheet 5: Pump Flow Rate Set and Measurement

Executed for Test Procedure Step: 4.2.4 Checksheet#________

Executed by: ____

Date/time: ________________________

Target Flow Rate: ___________ g/hr

Receiver/bottle tare:________________ grams

Feed Material: _____________________ Density_________g/mL

Place Effluent Line (12A/B) into receiver and Feed Line (L1) into Feed Tank.

Valve Placement/Confirmation (See Figure A-1) other valves closed:

Config. 4: CST Pump Flow Rate Calibration Valve Configuration
(Foward flush bypass to Line 12A/B)

Valve	**Direction**
V1	L1 → V1 → 1A/B
V2a/b	1A/B → V2a/b → 2A/B
V3a/b	2A/B → V3a/b → 12A/B

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

Get Regression Curve from Sheet 3 Performance for Configuration 1 relative to Configuration 4.
Full System Flow: ____________________________________mL/hr

Sheet 6: Effluent Collection Record

Executed for Test Procedure Step: ________ Checksheet#__________

Executed by: ____

Date/time: ______________________________

Receiver/bottle tare:____________________ grams

Feed Line (L1) into Feed Tank:___________ Primary Column:__________

Feed Material: __________________________ Density____________g/mL

Effluent Line (11) into _________________________

Valve Placement/Confirmation (See Figure A-1, other valves closed):

Config.1: Full System Forward Feed Valving Configuration (Forward flow to Line 11)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V8	6 → V8 → 7
V2a/b	1A/B → V2a/b → 2A/B	V9	7 → V9 → C2
V3a/b	2A/B → V3a/b → 3A/B	V10	C2 → V10 → 8
V4a/b	3A/B → V4a/b → C1a	V11	8 → V11 → 9
V5a/b	C1a/b → V5a/b → 4A/B	V12	9 → V12 → C3
V6a/b	4A/B → V6a/b → 5A/B	V13	C3 → V13 → 10
V7	5A/B → V7 → 6	V14	10 → V14 → 11

Flow Rate (from Sheet 5, 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 is changed)

Sheet 7: Primary Column Sampling

Executed for Test Procedure Step: 4.2.5.2 Checksheet#________

Executed by: ____

Completed Date/time: ____________________________ Runtime __________

Feed Line (L1) into Feed Tank:__________ Primary Column:_________

Feed Material: _________________________ Sp. gravity (from Sheet 4): _____________

Sample Vial plus stopper Tare weight:_________________g

Place effluent Line (13) into Flush Vial.

Change V8 position: from: 6 → V8 → 7 to: 6 → V8 → 13

Flow Rate (from Sheet 5, Checksheet#____):________ mL/hr

Collect ~2 mL in Flush Vial (approx. 8 min); Start (Runtime) _________
End (Runtime) _________

Place line 13 into sample vial

Collect ~5 mL in sample vial (approx. 19 min) Start (Runtime) _________
End (Runtime) _________

Time of collection:____________min_______sec

Redirect valve V8 back to line 7 as follows:

Change V8 position: from: 6 → V8 → 13 to: 6 → V8 → 7

Weight Sample + stopper:______________g, Net Sample:___________g

Flow rate = Net Sample _________ / sample time________ = ____________g/hr

____________g/hr / density = ___________mL/hr

Observations:

Sheet 8: Primary Column Sample Record Checksheet#________

Executed for Test Procedure Step: *4.2.5.2* Executed by:____________________
Completed Date/time: ______________________________
Run Start (Date/time): ____________________________

Sample	Projected Run Time	Projected Clock Time	Actual Clock Time	On-Line γ	Feed Temp. (C)
C1E-1	1 hr 0 min				
C1E-2	17 hr 40 min				
C1E-3	34 hr 20 min				
C1E-4	51 hr 0 min				
C1E-5	67 hr 40 min				
C1E-6	84 hr 20 min				
C1E-7	101 hr 0 min				
C1E-8	117 hr 40 min				
C1E-9	134 hr 20 min				
C1E-10	151 hr 0 min				
C1E-11	167 hr 40 min				
C1E-12	176 hr 0 min				
C1E-13	184 hr 20 min				
C1E-14	192 hr 40 min				
C1E-15	201 hr 0 min				
C1E-16	209 hr 20 min				
C1E-17	217 hr 40 min				
C1E-18	226 hr 0 min				
C1E-19	234 hr 20 min				
C1E-20	242 hr 40 min				
C1E-21	251 hr 0 min				
C1E-22	259 hr 20 min				
C1E-23	267 hr 40 min				
C1E-24	276 hr 0 min				

Sheet 8: Primary Column Sample Record - continued

Checksheet# _________

Sample	Projected Run Time	Projected Clock Time	Actual Clock Time	On-Line γ	Feed Temp. (C)
C1E-25	284 hr 20 min				
C1E-26	292 hr 40 min				
C1E-27	301 hr 0 min				
C1E-28	309 hr 20 min				
C1E-29	317 hr 40 min				
C1E-30	326 hr 0 min				
C1E-31	334 hr 20 min				

Sheet 9: Secondary (C2) Column Sampling

Executed for Test Procedure Step: _4.2.5.3_ Checksheet#__________

Executed by: ____

Completed Date/time: ____________________________

Feed Line (L1) into Feed Tank:__________

Feed Material: ________________________ Sp. gravity (from Sheet 4): _____________

Sample Vial plus stopper Tare weight:________________g

Effluent Line (14) into _*Flush Vial*_

Change V11 position: from: 8 → V11 → 9 to: 8 → V11 → 14

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

Collect ~2 mL in Flush Vial (approx. 8 min); Start (Runtime) __________
End (Runtime) __________

Place line 14 into sample vial

Collect ~5 mL in sample vial (approx. 19 min); Start (Runtime) __________
End (Runtime) __________

Time of collection:____________min________sec

Redirect valve V11 back to line 9

Change V11 position: from: 8 → V11 → 14 to: 8 → V11 → 9

Weight Sample + stopper:______________g, Net Sample:___________g

Flow rate = Net Sample _________ / sample time________ = ____________g/hr

____________g/hr / density = ___________mL/hr

Observations:

Sheet 10: Secondary (C2) Column Sample Record Checksheet#________

Executed for Test Procedure Step: *4.2.5.3* Executed by:____________________

Completed Date/time: ____________________________

Run Start (Date/time): __________________________

Sample	Projected Run Time	Projected Clock Time	Actual Clock Time	On-Line γ	Feed Temp. (C)
C2E-1	17 hr 10 min				
C2E-2	50 hr 30 min				
C2E-3	83 hr 50 min				
C2E-4	117 hr 10 min				
C2E-5	150 hr 30 min				
C2E-6	183 hr 50 min				
C2E-7	217 hr 10 min				
C2E-8	250 hr 30 min				
C2E-9	283 hr 50 min				
C2E-10	317 hr 10 min				

Sheet 11: Primary Column C1a/b Reverse Feed Valving

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 (12 A/B, Configuration 5) into _______________________

Valve Placement/Confirmation (See Figure A-1) for Configuration 5, other valves closed:

Config. 5: Column C1a/b Reverse Flush Valving Configuration
(Reverse flush to Line 12A/B)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1A/B	V5A/B	4A/B → V5A/B → C1A/B
V2A/B	1A/B → V2A/B → 16A/B	V4A/B	C1A/B → V4A/B → 3A/B
V6A/B	16A/B → V6A/B → 4A/B	V3A/B	3A/B → V3A/B → 12A/B

Pump On: _______________ (time/date); Runtime _________

Observations:

Pump Off:_______________ (time/date); Runtime __________

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

Sheet 12: Guard Columns C2 & C3 Reverse Feed Valving

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 (13, Configuration 6) into _______________________

Valve Placement/Confirmation (See Figure A-1) for Configuration 6, other valves closed:

Config. 6: Reverse Feed Valving Configuration
(Reverse flush through guard columns)

Valve	Direction	Valve	Direction
V1	L1 → V1 → 1B	V11	9 → V11 → 8
V2b	1B → V2b → 15	V10	8 → V10 → C2
V14	15 → V14 → 10	V9	C2 → V9 → 7
V13	10 → V13 → C3	V8	7 → V8 → 13
V12	C3 → V12 → 9		

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 is changed)

Sheet 13: CST Sorbent Dissolution

Objective: To remove and dissolve the CST sorbent at the end of the test. This procedure will facilitate waste disposal.

Sorbent Dissolution Procedure: Checksheet#________

<u>NOTE</u>: The CST sorbent will not be analyzed and will be dissolved in a batch.

1. Confirm the following valve positions:

C1a	V4a	&	V5a	Closed
C1b	V4b	&	V5b	Closed
C2	V9	&	V10	Closed
C3	V12	&	V13	Closed

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

3. Remove column C1a from the retainers.

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

5. Remove the top of the column.

6. Deposit the sorbent into a 2 liter beaker by scraping and rinsing. The beaker will be large enough to hold all the CST sorbent (~ 21 grams) plus the required volume to dissolve the sorbent as indicated in Step 8.

7. Repeat the Steps 3 through 6 for columns C1b, C2, and C3.

8. Digest the sorbent by sequentially adding:
   610 mL DI water
   210 mL of 2<u>M</u> HCL
   105 mL of 1<u>M</u> HF

9. Place on a hot plate with a magnetic stirrer. Heat gently until the CST sorbent is dissolved.

10. Pour cooled solution into waste receiver labeled ____________ pending disposal through hot cell drain.

Sheet 14: CST Sorbent Bed Density Determination

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

Procedure: Checksheet# __________

1. Dry approximately 100 grams of sorbent in a tared evaporating dish at 103°C for 4 hours.

2. Cool in a desiccator and weigh on an analytical balance to find per cent moisture.

   Weight of the sorbent + evap dish: ____________________g

   Weight of the evaporating dish: ____________________g

   Weight of the CST Sorbent: ____________________g

3. Mix approximately 50 grams of the sorbent with 18 Mohm·cm water and slurry the sorbent into each of the glass columns to approximately 50% of the column volume.

4. Condition the sorbent as follows (per instructions from the manufacturer)):

   a. Backflush the column with a high superficial velocity to backwash fines from the bed (usually 200% of the initial height of the bed). When evidence of fines ceases in the effluent, stop the flow of water and allow the bed to settle.

   b. Using 0.22 M NaOH, condition the bed in a downflow manner. Monitor the pH of the bed and when the effluent remains at a pH equal to or slightly greater than the pH of the waste feed (pH ≈ 13.3). Check the pH in the effluent. When the pH value stabilizes, turn off the pump, allow the bed to settle. After settling, mark the top of the bed. Remove the sorbent from the column and determine the volume (mL) that the bed occupied.

   c. Wash the sorbent with 18 Mohm·cm water until the NaOH has been flushed from the system. Analytically transfer the sorbent to a tared evaporating dish, washing all the sorbent from the column. Dry the sorbent overnight at 103° C and determine the dry mass of the sorbent.

Sheet 14: CST Sorbent Bed Density Determination - cont'd. Checksheet#____________

d. Repeat this for five iterations and use the average

Sample	Settled Bed Volume, mL	Wt. of Evap Dish, g	Dry Wt of (Absorbent + Evap Dish) g	Net Sorbent Wt. g	Density (dry g /mL bed)	Avg. Density, g/mL bed
1						
2						
3						
4						
5						

5. Bulk Density Measurements:

Take a graduated cylinder (25 mL) and tare on a top loading balance. Using the other half of the sorbent from step 2, pour into the cylinder and tap the side to settle the sorbent. Record the weight of the cylinder and sorbent on the chart below. Using tongs to handle the cylinder, record the level of the sorbent in mL. Repeat this operation for five iterations and use the average for the density.

Weight of cylinder plus sorbent	Weight of cylinder	Weight of sorbent	mL of sorbent in cylinder

Average Weight of Sorbent =__________________

Average mL of Sorbent =__________________

Density =__________________g/mL

Sheet 15: NaOH Solution Preparation from 5.0 mole/L Stock

Objective: NaOH solutions of different molarity are needed in the hot cell for preparing waste feed and for flushes. This data sheet gives the procedure to prepare 1 kg of each of these NaOH solutions from 5.0 mole/L stock solution. Masses of desired solution other than 1 kg may be linearly scaled using the table below.

NaOH Preparation/Kg	Stock = 5 mole/L		
Prepared Solution (mol/L) (27°C)	Stock Soln (g)	H2O (g)	Total (g)
0.22	51.65	948.35	1000
0.4689	108.67	891.33	1000
5.0	1000.00	0.00	1,000

Procedure: **Checksheet#**________________

1. Quantity of NaOH solution to be prepared ________g; desired strength ________mol/L
2. Quantity of water required ________________________g.
3. For desired strength, from Table above, meter water as:
   Tare wt of Beaker#1 and stirrer:__________________g
   Wt. of Beaker#1, stirrer and water:________________g
   Net wt. of water taken:______________________________g
4. Calculate and meter NaOH, from Table above, as:

$$\frac{(\qquad)\ g\ stock\ 5.0\ \overline{M}\ NaOH}{(\qquad)\ g\ H_2O} \times (\qquad)\ Net\ Wt.H_2O$$
$$= (\qquad)\ g\ stock\ soln.$$

   Tare wt of Beaker#2:______________________g
   Wt. of Beaker#2 and NaOH:_______________g
   Net wt. of NaOH:______________________g
5. Slowly, over a few minutes, pour preweighed amt. of 5.0 mole/L stock NaOH solution into the water in the 1L beaker on a stir plate, allowing time for blending.
   Record final solution temp. ___________°C.
   Final wt. of (beaker+solution) = _____________ g
   Wt. of solution prepared = __________ g.

Appendix B: Material Safety Data Sheets

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List of Material Safety Data Sheets

MSDS Numbers

	MSDS
Sodium Hydroxide, NaOH	045620
Hydrofluoric Acid, HF	039275
Hydrochloric Acid, HCl	036338
UOP IONSIV Ion Exchanger Type IE-911 (Crystalline Silicotitanate)	053329

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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.

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.

TION 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

ICITY 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

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.

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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.

TRANSPORTATION DATA

RTMENT 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

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.

HEALTH EFFECTS AND FIRST AID

INHALATION:
HYDROGEN FLUORIDE:
CORROSIVE/TOXIC. 30 PPM IMMEDIATELY DANGEROUS TO LIFE OR HEALTH.
CUTE 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 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.

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.
ONCRETE: 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.
ODIUM: REACTS WITH EXPLOSIVE VIOLENCE WITH AQUEOUS ACID.
ODIUM 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.

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DECOMPOSITION:
THERMAL DECOMPOSITION MAY RELEASE CORROSIVE HYDROGEN FLUORIDE.

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

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.

CONDITIONS TO AVOID

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

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 ABSORBENT 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.

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.

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

MATERIAL SAFETY DATA SHEET

SPECTRUM CHEMICAL MFG. CORP.
14422 S. San Pedro Street
Gardena, CA 90248-9985
Phone : (213) 516-8000

EMERGENCY CONTACT:
CHEMTREC (800) 424-9300
SPECTRUM TECHNICAL SERVICE (213) 516-8000

MSDS # 039275

PRODUCT #: Z6759 NAME: HYDROCHLORIC ACID REAGENT
MATERIAL SAFETY DATA SHEET, Valid 2/96 - 4/96
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Sigma Chemical Co.	Aldrich Chemical Co., Inc.	Fluka Chemical Corp.
P.O. Box 14508	1001 West St. Paul	980 South Second St.
St. Louis, MO 63178	Milwaukee, WI 53233	Ronkonkoma, NY 11779
Phone: 314-771-5765	Phone: 414-273-3850	Phone: 516-467-0980
		Emergency Phone: 516-467-3535

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.

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.

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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.

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RTECS #: MW4025000
HYDROCHLORIC ACID

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.

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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

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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.

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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

UOP

Material Safety Data Sheet

1. EXPERIMENTAL PRODUCT AND COMPANY IDENTIFICATION

Page 1 of 5
January 1996

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

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

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.

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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.

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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	2315454
Titanium dioxide	2366755
Sodium oxide	2152089
Trade Secret Material	listed
Trade Secret Material	listed
Aluminum oxide	2156916
Copper oxide	2152691
Calcium oxide	2151389
Chromium oxide	2151609
Magnesium oxide	2151719

16. OTHER INFORMATION

For additional information concerning this product contact the following:

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Date: January 1996

Revision: 1

Supersedes: February 1995

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Appendix C: Valving Configuration Figures

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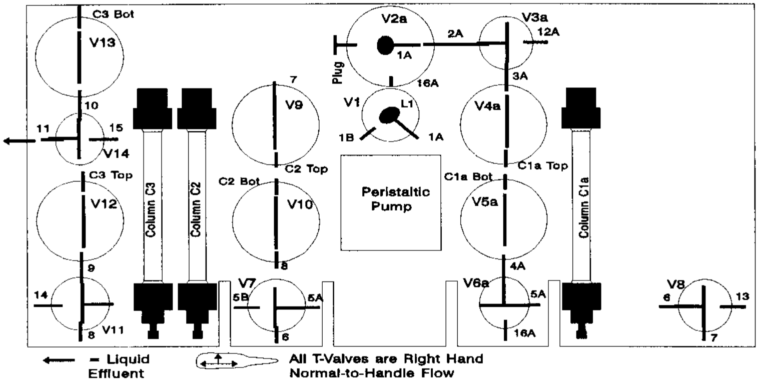

Figure C-1: Config. 1A, Primary Column (C1a) Full System Feed Valving

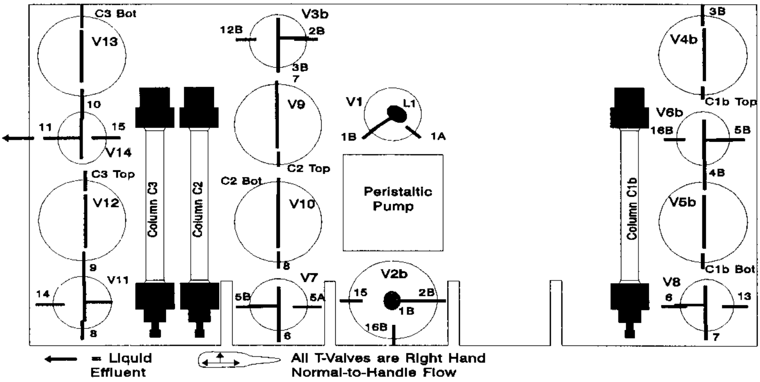

Figure C-2: Config. 1B, Backup Column (C1b) Full System Feed Valving

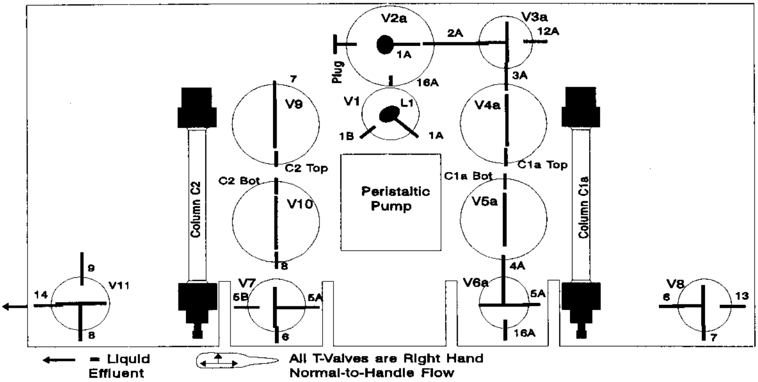

Figure C-3: Config. 2A, Forward Feed through Primary and First Guard Columns

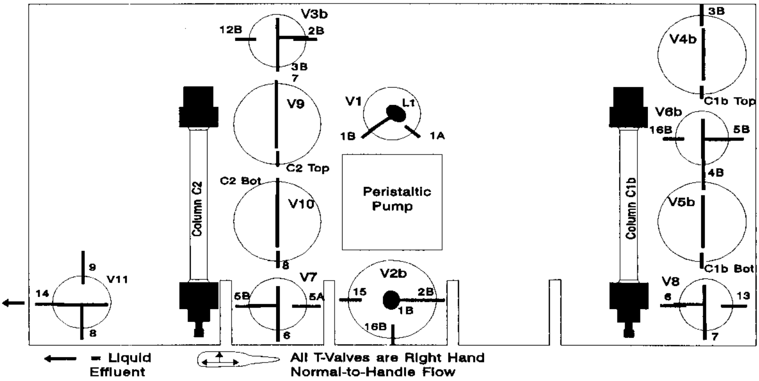

Figure C-4: Config. 2B, Forward Flow Through Backup and First Guard Columns

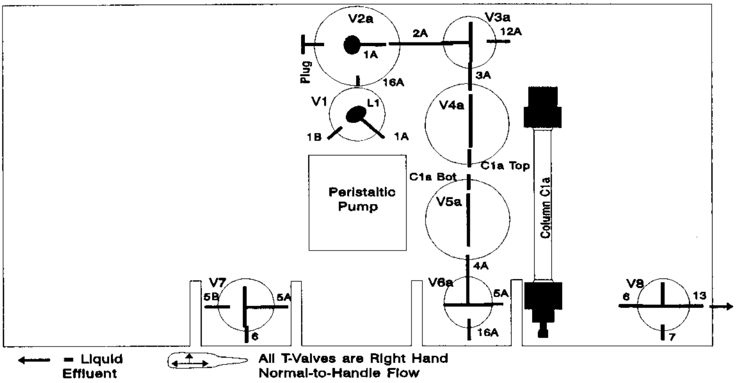

Figure C-5: Config. 3A, Forward Flow Through Primary (C1a) Column to Sample

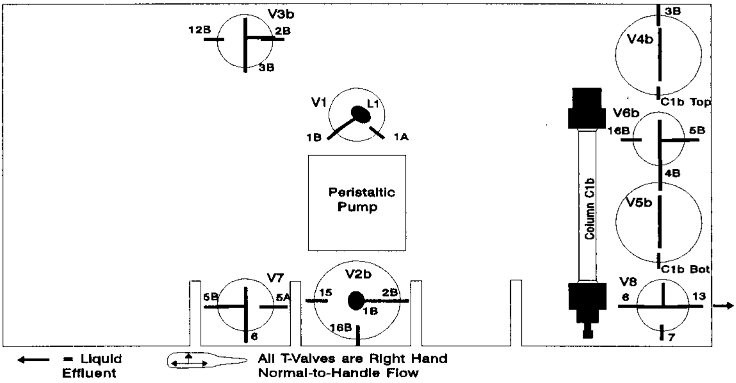

Figure C-6: Config. 3B, Forward Flow Through Backup (C1b) Column to Sample

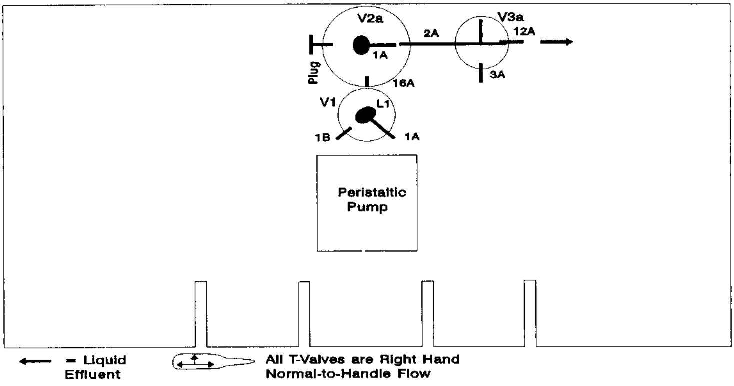

Figure C-7: Config. 4A, Forward Flush Bypass to Line 12A

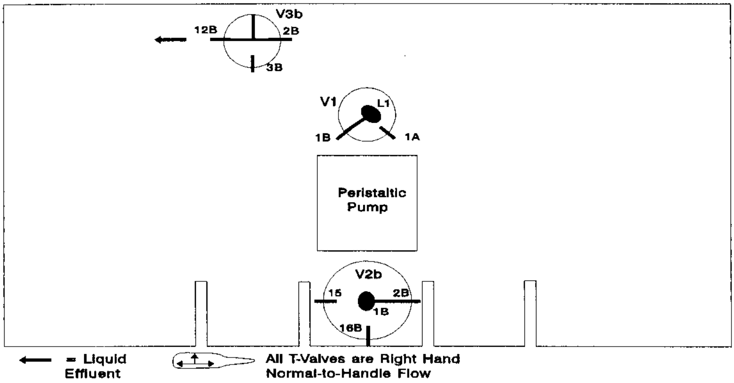

Figure C-8: Config. 4B, Forward Flush Bypass to Line 12B

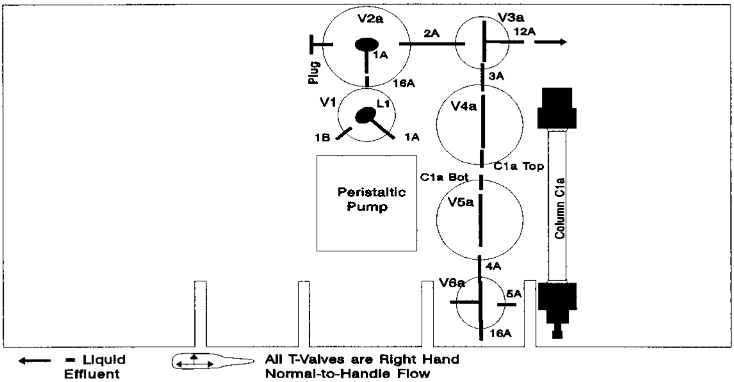

Figure C-9: Config. 5A, Reverse Flush Through Primary (C1a) Column

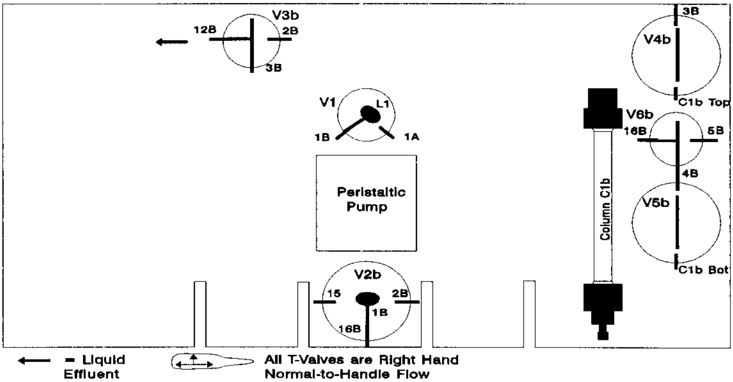

Figure C-10: Config. 5B, Reverse Flush Through Backup (C1b) Column

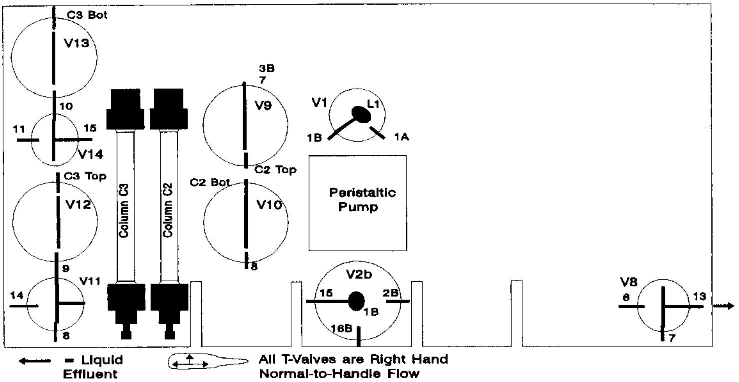

Figure C-11: Config. 6, Reverse Flush Through Guard Columns (C3 and C2)

Appendix D: Gamma Probe Operation

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GENERAL INSTRUCTIONS BETA/GAMMA HOT CELL PROBE
VERSION 1.0

INTRODUCTION

This desk instruction provides checklist-style guidance on producing data with the beta/gamma hot cell probe. This instruction assumes the person has read the GammaVision™ software manual, and understands the basics of Microsoft Windows[1] operating system as well as nuclear spectroscopy.

Setup:

Cable connections:

Ensure that all power cables are connected.

Ensure that cables to the probe are connected.

Powering up:

Ensure that both bias power supplies are switched to OFF and set to 0 volts (V). Reset the beta detector bias supply if necessary.

Turn on the main power switch at the bottom of the relay rack.

Turn on the bottom NIM bin.

Turn on the computer (the bottom NIM bin power MUST be on).

Turn on the top NIM bin.

Turn on the gamma detector bias supply (top NIM bin). Slowly raise the voltage up to 800 V Positive.

Plug the beta amplifier (Top NIM crate, Canberra Fast[2] spectroscopy amp) output to an oscilloscope.

Trademark of Microsoft Corporation, Redmond, Washington.

Trademark of Canberra Industries, Inc., Meridian, Connecticut.

Place a source under the beta detector. This may be needed to provide enough counts to see in the oscilloscope.

Turn on the beta detector bias supply (bottom NIM crate). Very slowly (<20 volts/sec) raise the beta detector bias voltage to 800 V, waiting until the pulse on the oscilloscope returns to normal before proceeding.

When the beta detector bias voltage is at 800 V, reconnect the amplifier output to the T-connector at channel 2 of the single-channel analyzer (SCA).

Turn the pointer on (up). The switch is in a blank NIM module in the top NIM bin.

Starting GammaVision™

At the C: prompt, enter "win" to enter Windows™.

Double-click on the GammaVision™ icon.

Switch to multichannel buffer (MCB) #1 (gamma) if not already there.

Run job "gate1cn.job".

Switch to MCB #5 (beta).

Run Job "gate5off.job"

Spectrometry:

Taking a spectrum in GammaVision™ is as easy as hitting ALT2 (STOP), ALT3 (CLEAR), ALT1 (START), and giving the program all the information it requests. GammaVision™ is set up to ask for collection live time and sample description.

Spectra for most samples will be taken using an automated .JOB file, which only requires the user to input live time and sample description.

Running the counters:

When you are ready to start a count, hit the stop button (or switch) on the timer, then hit the reset (to clear previous counts), and then hit the start button (switch) to start the count.

Daily Energy Calibration and QA Check

Perform startup.

Center the ^{154}Eu quality assurance (QA) source under the probe (at the intersection of the light beams of the pointer).

Set the live time on the MCBs and counters to 2,000 seconds.

Start the counters and the MCBs.

ON MCB #1, Recall qaeusrc.roi.

Compare the peak centroids to the published values for ^{154}Eu.

If the centroids of the peaks are shifted from the published values by more than 10 keV, adjust the amplifier fine gain to match the centroids with published values.

Stop and start the MCBs between adjustments of the fine gain controls.

If the actual values are more than 30 keV apart, consult with the scientist on whether a recalibration is necessary.

Compare the endpoint of the beta spectrum taken to the file C:\betapro\SrtdQA1.chn. If the endpoint is different, adjust the fine gain on the Canberra amplifier.

After completing the calibration and QA check, take a spectrum with both MCBs.

Run .JOB file C:\mcafile\QAcheck.job.

At the end of the count record all counter values.

Compare the current QA check source values to previous QA counts; the values should be within 20%. The values below are from page 10 (repeated on page 37) of laboratory notebook WHC-N-11151 1 (Beck 1994), and the spectra files: Gamma, C\data\gampro\srtdQA.chn; Beta, C:\data\betapro\srtdQA1.chn.

Total Beta (TB):	2,073,772	Betas Above Gammas (BAG):	377
Total Gamma (TG):	1,448,951	Gammas Above Cesium (GAC):	215,372
Cesium-137 (Cs):	72,892		

Eu-154 @248 keV 385,290 counts, @723 keV
85,423 counts @993 keV 10,671 counts @1274 keV 5,667 counts

Running Samples:

If the daily QA check is OK, then samples may be run.

Run the .JOB file gammaV5.job .

Remember which scan you're on.

Scans 1 and 7 are backgrounds, so the sample must be moved away from the probe during those scans.

Label the scans appropriately at 0.5, 5, 9.5, 14, and 18 inches from the bottom of the core segment.

Align the sample with the laser beam crosshairs so as to position the sample for scans 2 through 6.

Return the sample to 11A Hot Cell Operations personnel.

After the end of the run (including backgrounds), start Quattro Pro™.

Open the file appropriate to the geometry of the sample. LogSS5.WQ1 should be used for "log" samples, and TraySS#3.WQ1 for "tray" (flat) samples.

Hit Alt A.

Change the text on the 2nd line of the graph to reflect the identity and nature of the sample.

Print-to-Fit and Print Graph. There is no need to change any settings before printing, as everything is already set up.

Save the Quattro Pro™ file in a directory appropriately named for the sample.

In Windows™ File Manager, copy all the files in the directory

C:\practjob

to a directory that is labeled in such a way as to identify the sample.

Paste all the output into the laboratory notebook.

You're done!...

Normal Shutdown:

Shut the pointer off.

Normally, everything else is left on.

Total Shutdown:

Perform the normal shutdown.

Exit Windows™.

Shut off the computer.

Slowly turn the bias supply voltages down to 0 and lock them there.

Turn off the bias supplies.

Turn off the NIM bins.

Turn off the fans (back of rack).

REFERENCES

Beck, M. A., 1994, Controlled Laboratory Notebook, Hot Cell Beta/Gamma Probe, WHC-N-120 7, Westinghouse Hanford Company, Richland, Washington.

Beck, M. A. et al. WHC-SD-WM-DTR-039 is the source of this instruction set.

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