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Project Title/Work Order K6JG6/8E140		EDT No. 604204 ECN No.

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**This document was reviewed following the
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7. Abstract This test plan describes the activities associated with the High Gradient Magnetic Filtration (HGMF) of plutonium-bearing solutions (10-L). The 10-L solutions were received from Argonne National Laboratories in 1972, are highly acidic, and are considered unstable. The purpose of the testing is to show that HGMF is an applicable method of removing plutonium precipitates from solution. The plutonium then can be stored safely in a solid form.		
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HGMF of 10-L SOLUTIONS

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1.0 INTRODUCTION

The Engineering and Environmental Demonstration Laboratory and Plutonium Process Support Laboratories personnel will perform a series of tests on plutonium-bearing solutions (10-L). The 10-L solutions were received at Hanford from Argonne National Laboratories in 1972. These solutions are stored in polybottles, and are considered to be reactive and corrosive. The purpose of the tests is to develop technologies to separate plutonium from chemicals that interfere with its long term safe storage. One of the technologies selected for demonstration is known as High Gradient Magnetic Filtration (HGMF)(1).

The HGMF technology was developed in the early 1970's for the wet beneficiation of quality grade kaolin clay. In this application, the magnetic separator removes micron size mineral contaminants. Kaolin clay is a white alumina-silicate mineral. Quality clays are principally employed as filling and coatings agents in paper products. The clays are sold on the basis of their white color. The contaminants removed by the HGMF cleaning are feebly magnetic with susceptibilities typically 10^{-6}cm^{-3} and range in size from 5-10 microns. The contaminants are typically anatase, rutile, mica, and iron pyrite(2).

Extensive laboratory testing and development for installation of an HGMF on the primary loop of Hanford's N-Reactor was conducted in the 1980's. The principal use was to remove activated corrosion products from the primary loop. The HGMF system was not installed due to the shutdown of N-Reactor. However, the testing suggested that the HGMF system would be useful on the removal of transuranic elements from the Neutralized Current Acid Waste (NCAW) tanks in the 200 Areas. A series of tests were performed in 1985 on synthetic NCAW using chromium as the transuranic surrogate. These tests were successful in removing 95.5% of chromium in a single pass. Consequently, an HGMF was moved to the Plutonium Finishing Plant (PFP) for further testing on actual waste. Problems were encountered in getting the unit installed, which resulted in the cancellation of the proposed testing. However, the unit remained at PFP(3).

The HGMF system is rugged, has no moving parts, and is not sensitive to high radiation fields.

2.0 OBJECTIVE

The proposed tests are intended to demonstrate that the HGMF system is an effective method of removing plutonium precipitates from waste solutions.

3.0 PROCESS AND TEST DESCRIPTIONS

3.1 Process Description

Magnetic separation is a process by which magnetically susceptible particles are removed from a fluid by magnetic forces. The HGMF consists of an iron box that encloses the energizing coils, which, in turn, surrounds the filter (Figure 1). The filter is a nonmagnetic vessel that contains a magnetic matrix material. The matrix material can consist of expanded metal mesh, steel wool, steel balls, or steel screens, and has a void volume on the order of 80 to 90 percent. The void volume precludes plugging and facilitates backwashing to remove the solids captured.

The matrix distorts the magnetic field and causes strong magnetic gradients that capture sub-micron and weakly paramagnetic materials on the matrix fibers. Particle capture is a function of the magnetic susceptibility of the material and the applied field. However, a particle in a uniform magnetic field experiences no net forces. Ferromagnetic materials can be separated quantitatively at low field strength. Paramagnetic materials do not magnetically saturate; therefore, increasing the applied field increases the capture efficiency. The HGMF at PFP can produce up to a two Tesla field.

3.2 Test Description

The 10-L solutions contain dissolved plutonium; however, the solutions will be adjusted with sodium hydroxide to a pH range 9 to 12, which precipitates the plutonium. This precipitate is a paramagnetic material and can be removed by the HGMF.

Approximately 3 liters of alkaline 10-L solution, containing less than 9 grams of plutonium will be pumped from the first collection tank (CT1) through the filter at a rate of 1.0 cm/sec, and collected in the second collection tank (CT2). The filtrate solution from CT2 then will be pumped back through the filter at a rate of 1.0 cm/sec, and collected in CT1. A total of four passes through the filter will be performed. Refer to Figure 2 and Table 1.

The filter will then be drained, removed, and dried at 500°C. The drying process will drive off excess water and change the plutonium from the hydroxide form to an oxide form. The filter and its contents then can be stored safely. If in the future, there is a need to remove the plutonium oxide from the filter it can be backwashed with water to recover the material.

If no plutonium is removed from the first test, flocculent, such as magnetite or ferrite, will be added to the solution to help facilitate removal. It has been shown that magnetite increases plutonium removal in a superconducting magnetic filter(4). Ferrite addition has also been shown to be beneficial in the removal of actinides using a magnetic flux density of 3 Tesla (5).

To validate the experimentation a second test will be performed using a new filter.

3.3 Test Equipment

- ▣ Pacific Electric Motor Company, Cross Field High Gradient Magnetic Filter
- ▣ Bell Incorporated, Gauss meter Model 620
- ▣ Pump, MasterFlex[™] L/S Digi-Stalic[™] (MasterFlex and Digi-Stalic are registered trade marks of Cole Parmer instrument Company)
- ▣ 11 KW Chiller, ACME RD-5, or equivalent
- ▣ Filter: Nonmagnetic Vessel, 3/4 inch diameter 304L Stainless Steel Pipe; Matrix, 430 Stainless Steel wool
- ▣ Tube Furnace
- ▣ Mixers, Arrow 1750, or equivalent
- ▣ Combination Stirrer/Hot Plate, Corning PC-351, or equivalent
- ▣ Thermometer
- ▣ Stainless Steel Equipment: Tanks, Tubing, Valves, and Fittings
- ▣ Glassware: Beakers, Volumetric Flasks, and Sample Vials.

3.4 Test Environment

The testing will be conducted in 234-5Z, Room 187. This room is equipped with open face HEPA filtered hood for exhaust ventilation. The Magnetic Separator is located outside of the hood because of its size and weight. All tanks will be within the hood. All tubing/piping exterior to the hood will be double contained.

3.5 Samples and Analysis

At least four 20 milliliter samples will be taken per pass, one sample of the influent and a sample of the effluent every 5 minutes for 15 minutes.

Sample aliquots will be prepared as described in Section 5, and will be analyzed for plutonium using a Packard Instrument Corporation, Tri-Carb 1500 Liquid Scintillation Analyzer, located in Room 183 of 234-5Z. At least 1 blank and 3 duplicate sample aliquots will be analyzed to verify the accuracy of this procedure.

4.0 EXPECTED RESULT

Los Alamos National Laboratories has performed tests on a 3" warm-bore super conducting magnet at 7.5 Tesla and reported 99.8% removal of plutonium activity from their Waste Treatment Plant effluent(4).

The tests will be considered successful if at least 50% of the plutonium is removed from the solution.

5.0 TEST PROCEDURE

Before testing with actual 10-L solution, a water test will be performed using Procedures 5.2, 5.3, and 5.4. This will ensure operator training and procedure refinements prior to actual testing. This test will be conducted using deionized water meeting CAP/NCCLS Type III water standards or better.

5.1 pH Adjustment

The 10-L solution will be adjusted to a pH range of 9 to 12 by the Plutonium Process Support Laboratories personnel.

After pH adjustment a sample will be taken for particle size analysis.

5.2 Pre-Experimental Preparations

- A. Ensure that all valves shown in Figure 3 are closed.
- B. Verify that hood sash is at the correct height for proper airflow velocity.
- C. Transfer the alkalized 10-L solution from the transport containers to collection tank 1 (CT1). Take care to ensure that no spillage of solution occurs. Set aside the transport containers within the hood.

5.3 Filter Operation

- A. Refer to Figure 3 and Table 1.
- B. Turn on mixer located in the feed tank. Allow to mix for minimum of 5 minutes before proceeding to the next step.

NOTE: The feed tank can be tank CT1 or CT2 depending on the valve position refer to table 1. The receiver tank agitator should always be off. For example if tank CT1 is the feed tank, then tank CT2 is the receiving tank. The mixer in CT1 should be on, and the mixer in CT2 should be off.

- C. Position valves as indicated in Table 1. Ensure Valve 5 (V5) and sample ports (SP1 and SP2) are closed.
- D. Turn on pump. Set the pump speed to deliver a velocity of 1.0 cm/sec through the filter.
- E. Check for leaks. If leaks occur shut down pump, mixers, and close all valves. Fix leaks and begin procedure starting with Step A of this section.
- F. Sample Feed Stream as follows:

1. Ensure sample vial is labeled with the sample port number, time interval, and affix a rad label.
 2. Place the appropriate sample vial beneath the sample port. (influent SP1, effluent SP2)
 3. Open Valve and collect enough sample to fill the sample vial. Close valve.
 4. Cap sample vial and set aside within the hood.
- G. If performing the first pass through the filter go to Step K, otherwise continue.

NOTE: The first pass through the filter is to determine if the plutonium precipitates are being mechanically filtered. If mechanical filtration is inherent, then a different matrix material may be needed, such as expanded metal.

- H. Activate chiller and open cooling water valves for the HGMF unit.
- I. Turn on power supply and activate the HGMF unit and stop watch (timer) simultaneously.
- J. Measure the magnetic field intensity using a Gauss Meter and probe following manufacture procedure. Record Data in a bound notebook.
- NOTE:** If no magnetic field intensity is measured, the HGMF system or the Gauss Meter is not operating properly; therefore, the test cannot be performed. Shut down all systems, close valves, store solutions, and then troubleshoot and repair.
- K. Take a sample of effluent (SP2) and record cooling water temperature every 5 minutes for 15 minutes according to Step F.
- L. Continue operation until the inlet collection tank is empty or pump runs dry. Then turn off pump, and mixer. Close all valves.
- M. To begin next pass, repeat procedures starting with step A of Section 5.3, otherwise continue.

5.4 Filter Removal

- A. Refer to Figure 3.
- B. Ensure pump is off.
- C. Place a one-liter bottle under the drain line port, V5, within a containment pan.
- D. Open V1, V2, V3, and V4. This will drain the lines and the filter.

- E. When lines are drained, cap the one-liter drain bottle and place within a hood.
- F. Deactivate the magnet and turn off the power supply.
- G. Deactivate the chiller.
- H. Close all valves.

CAUTION: Make sure the HGMF is off before continuing. Serious injury can result if an attempt is made to remove filter while the magnet is activated.

- I. Disconnect the filter at the quick disconnects 1 and 2 (D1,D2 are above and below the HGMF unit).
- J. Install the quick disconnect plugs.
- K. Remove the filter and place within a hood.
- L. Continue to disconnect all fittings and tubing from the filter. Remove plastic sleeving.
- M. Cap both ends of filter with stainless steel Swagelok® plugs (Swagelok is a trademark owned by Crawford Fitting Company).
- N. Bag the filter out of the zone and transport to room 179. Place filter within the glove box containing the pipe furnace.

5.5 Drying

- A. Remove the filter from the plastic bag and uncap one end.
- B. Place filter within the tube furnace and bake at a temperature of $500 \pm 10^{\circ}\text{C}$ for 2 hours.
- C. Let the filter cool to room temperature inside the oven. After filter has cooled, cap the open end with the stainless steel Swagelok® plug.
- D. The filter vessel is now ready for storage.

5.6 Samples

For scintillation analysis, the plutonium precipitates in the samples must be redissolved. The following procedure will be performed within a hood.

5.6.1 Reagent "B-Acid"

- A. Read Material Safety Data Sheets (MSDS) for 70vol% Nitric Acid and 48vol% Hydrofluoric Acid.

- B. Measure out 10 mL of 70vol% Nitric Acid in a clean dry 10 mL volumetric flask.
- C. Mix 18 μ L of 48vol% Hydrofluoric Acid in the 10 mL of concentrated Nitric Acid.
- D. Cap, mark the flask "B-Acid", and affix a corrosive label. This makes a 0.05 molar Hydrofluoric Acid solution in Concentrated Nitric.

Note: This volume is sufficient for a single sample aliquot dissolution of plutonium precipitate.

5.6.2 Sample Preparation

- A. Label a clean dry glass sample vial (corresponding with markings on the sample). Affix rad label to the lid of the vial.
- B. Weigh the sample vial with a glass covered stirrer bar and cap, record, and tare the scale. Ensure that the balance has been calibrated prior to use.
- C. Shake the sample vigorously for 30 seconds. Quickly decant approximately 5 mL of sample to the clean dry sample vial. Record the weight.
- D. Place the sample aliquot on a combination stirrer heating plate. Stir the sample aliquot.
- E. Add slowly (drop wise) an approximate equivalent amount of B-Acid to the sample aliquot.

CAUTION: The reaction is exothermic. The solution will be corrosive. It is important that B-Acid is added slowly to the solution. If added to quickly the solution could splatter, the vial may break, or the solution may boil over.

- F. If the precipitates are not dissolved or the solution is turbid heat the sample until it begins to boil. Turn off the heat.
- G. If the sample is still turbid add drop wise B-Acid until dissolved. Additional heat may be required.
- H. Remove the vial from the stirrer/hot plate and allow to cool to room temperature. Cap vial, and record the weight.
- I. Add 1 mL of the sample aliquot to the scintillation vial and cocktail, cap and have released from the zone. Transport scintillation vial to room 183 for scintillation analysis. Re-weigh the sample aliquot and record. Repeat this procedure for each sample.

Note: The sample aliquots will be prepared for analysis and analyzed for plutonium content using in-house procedures for the scintillation analyzer.

5.7 Preformed Magnetite

Note: This part of the procedure should only be performed if the analysis from the above procedure shows inadequate removal of plutonium precipitates from the neutralized 10-L solution. Otherwise proceed to the next section.

- A. Review MSDS for Magnetite.
- B. Install a new filter as depicted in Figure 2.
- C. Transfer all alkalized 10-L solution to tank CT1, including the samples, but not the sample aliquots.
- D. Add to tank CT1 14 grams of magnetite.
- E. Mix for 30 minutes.
- F. Repeat the procedures starting with Step A of Section 5.3.

5.8 Insitu Magnetite

Note: This part of the procedure should only be performed if the analysis from the above procedure shows inadequate removal of plutonium precipitates from the neutralized 10-L solution. Otherwise proceed to the next section.

- A. Review MSDSs for Hydrofluoric Acid, Nitric Acid, Magnetite, and Sodium Hydroxide.
- B. Install a new filter as depicted in Figure 2.
- C. Obtain a clean dry PYREX 4 liter beaker, with Stirrer Bar.
- D. Within a hood, transfer 1 liter of alkalized/magnetite 10-L solution to the 4 liter beaker. Place it on a stirrer/hot Plate.
- E. While mixing add slowly 1 liter of B-Acid.

CAUTION: The reaction is exothermic. The solution will be corrosive. It is important that B-Acid is added slowly to the solution. If added to quickly the solution could splatter, the vial may break, or the solution may boil over.

- F. If the precipitates are not dissolved or the solution is turbid, heat the sample until it begins to boil. Turn off the heat.
- G. If the sample is still turbid add, in 5 mL amounts, B-Acid until dissolved. Additional heat may be required.
- H. Remove the beaker from the stirrer/hot plate and allow to cool to room temperature.

- I. Prepare an ice bath.
- J. Place the 4 liter beaker in the bath, and on a stirrer. Cool the solution to approximately 4°C.
- K. Weigh out 640 grams of Sodium Hydroxide or an appropriate amount to neutralize the solution.
- L. Add the Sodium Hydroxide to the 4 liter beaker as rapidly as possible. Do not add so quickly as to form a block of Sodium Hydroxide at the bottom of the beaker.

CAUTION: The reaction is exothermic. It is important that the Sodium Hydroxide is not added so quickly that the solution begins to boil, or splatter. Sodium Hydroxide is corrosive.

NOTE: The pH of the solution must change rapidly in order to form a ferrite complex with plutonium trapped in its lattice. Otherwise ferric hydroxide and ferrous hydroxide will precipitate.

- M. Cool the solution to room temperature.
- N. Obtain a pH meter and probe. Calibrate, and immerse the end of the probe 2 inches into the solution. Adjust the pH of solution between 9 and 12.
- O. Empty tanks CT1 and CT2 by transferring the solution to an appropriate container. Cap and store within the hood.
- P. Transfer the solution in the 4 liter beaker to tank CT1.
- Q. Repeat procedures beginning with Step A of Section 5.3.

5.9 Disposal of Waste

The filtrate liquid and samples will either be discharged to the radioactive drain, absorbed, or calcined using in-house procedures or as directed by the Hazardous Waste Coordinator.

The filters will be stored or disposed as directed by the Hazardous Waste Coordinator.

6.0 DISPOSITION OF EQUIPMENT

Equipment not disposed of will be moved to 1706-KE, 100-K area. Removal of the HGMF unit requires riggers and the use of "load spreaders" to move the unit across a pipe tunnel beneath Corridor 10.

7.0 SAFETY

7.1 Radiological Safety

The 10-L solutions contain relatively low concentrations of plutonium, uranium, and thorium nuclides. The low concentrations and the small volumes will keep dose rates low.

The cooling coil within the HGMF unit also contains a small amount of fixed cobalt-60. Past efforts to flush the cobalt-60 from the coil have failed. There is no reason to believe that the cooling water in these tests will flush the cobalt-60 out; however, the water will be collected and sampled for total activity before being disposed. The small fixed amount of cobalt-60 has no measurable increase in dose rates.

All personnel working to this plan will follow all Radiation Work Procedures (RWPs). RWPs will be written in accordance with HSRCM-1, Hanford Site Radiological Control Manual(6).

7.2 Criticality Safety

All work will be performed within the posted Criticality Prevention Specification (CPS) and postings for Hood 187-3. The small amount of Plutonium and Uranium in the neutralized 10-L solution will preclude any chance of a criticality.

7.3 Electrical Safety

All high voltage connections (480 VAC 3 phase power) to equipment are made via metal conduits through switch boxes that can be locked and tagged when the system is shut down. The lock and tag procedures will be followed for all maintenance on the water chiller and HGMF unit. The HGMF is equipped with short circuit/overload protection.

The mixers and pump will run on 115 volt power via standard receptacles. These pieces of equipment will be unplugged when not in use.

7.4 Chemical Hazards

Material Safety Data Sheets (MSDS) will be reviewed for every chemical in use. All work with chemicals will follow the Chemical Hygiene Plan, WHC-SD-HSP-001(7). The reagents such as Nitric Acid, Hydrofluoric Acid, and Sodium Hydroxide, are corrosive materials. The 10-L solutions contain plutonium. Plutonium and its compounds are extremely toxic.

Proper protective clothing/equipment will be addressed in the RWPs and Job Hazard Analysis Report. The protective clothing shall be a minimum of anti-C's, rubber gloves, safety glasses, and plastic aprons. Personnel will familiarize themselves with location of eye washes and showers prior to testing.

In the event of a spill, the spread of contamination will be contained via double contained tubing, plastic sleeve, and containment pans beneath equipment that might leak. Spill pillows will also be available to absorb the liquid if necessary.

7.5 HGMF Hazards

The parallel plate design of the HGMF contains the magnetic field primarily between the plates. A small magnetic field can occur directly above and below the unit. There are no magnetic hazards in the normal working area around the outside of the HGMF. There is no need to reach in or near the magnetic field during any part of the operation. However, during the water test a survey with the Gauss meter will ensure that magnetic flux density in the working area will be less than the routine occupational exposure limit of 1 Gauss.

The HGMF is cooled by chilled water. In the event of cooling water failure, the HGMF will automatically shut down at 51 °C. The plastic sleeving containment of the filter will remain intact.

8.0 QUALITY ASSURANCE

These tests are assigned an Approval Designator of SQ per MRP 5.43. Work will be performed in accordance with WHC-CM-4-2 Quality Assurance Manual(8). The quality assurance implementation is under the direction of the cognizant engineer. Relevant work will be documented in a bound notebook. Results of testing will be documented via supporting document per WHC-CM-6-1 "Standard Engineering Practices."

One additional test will be conducted for repeatability, and to determine experimental error.

Samples will be analyzed by the Plutonium Process Support Labs using a Packard Instrument Corporation Tri-Carb 1500 Liquid Scintillation Analyzer. A standard that has been calibrated against an National Bureau Standard material will be used to calibrate the analyzer before processing the samples.

The Gauss Meter will be calibrated using an internal standard following the manufacturer's recommended procedures.

9.0 ORGANIZATIONS RESPONSIBILITIES

9.1 Engineering & Environmental Demonstration Laboratory (EEDL)

The EEDL personnel will write test plans, perform tests, document results, and arrange equipment disposition and waste disposal.

9.2 Plutonium Process Support Laboratories (PPSL)

The PPSL personnel will pH adjust the 10-L solutions, analyze samples for plutonium concentrations, provide technical support, and review/approve documents.

9.3 Plutonium Finishing Plant (PFP) Engineering

PFP Operations will provide letter of instruction, test solutions, funding, and review/approve documents.

9.4 PFP Safety Assurance

Industrial, nuclear, and criticality safety will review and approve documents.

9.5 PFP Quality Assurance

Quality Assurance will review and approve documents.

9.6 PFP Maintenance

The PFP Maintenance organization will provide installation and maintenance support.

10.0 SCHEDULE

Refer to the PFP integrated schedule for target dates. The testing should be completed by March 1995.

11.0 REPORTS

Interim reports will be documented via EEDL Weekly Reports. Test results will be issued at completion of testing as a supporting document.

12.0 REFERENCES

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3. Oder, R. R. "High Gradient Magnetic Separation Theory and Applications". IEEE Transactions on Magnetics, Vol. Mag-12, No.5, September 1976.

14.0 ATTACHMENTS

Figure 1 HGMF CROSS SECTION 16
Figure 2 TEST SETUP 17
Table 1 TESTS 18

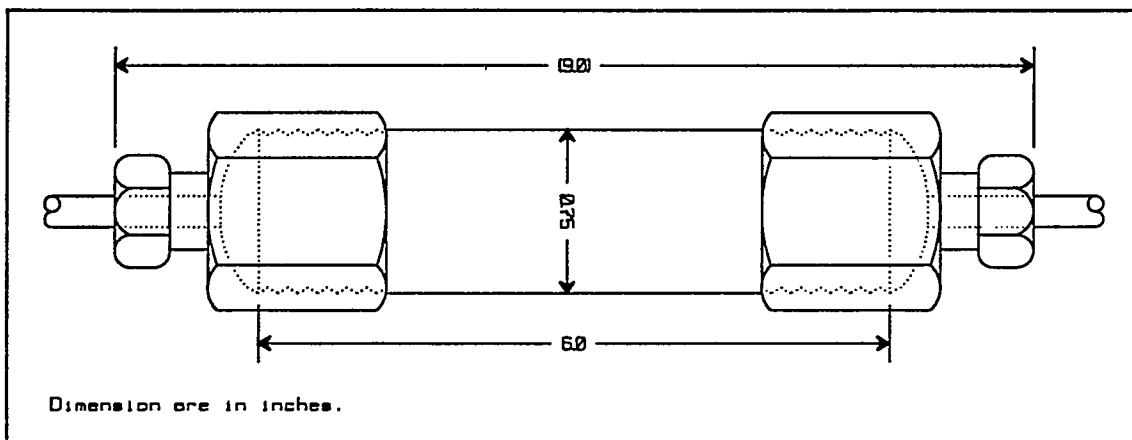


Figure 1 FILTER

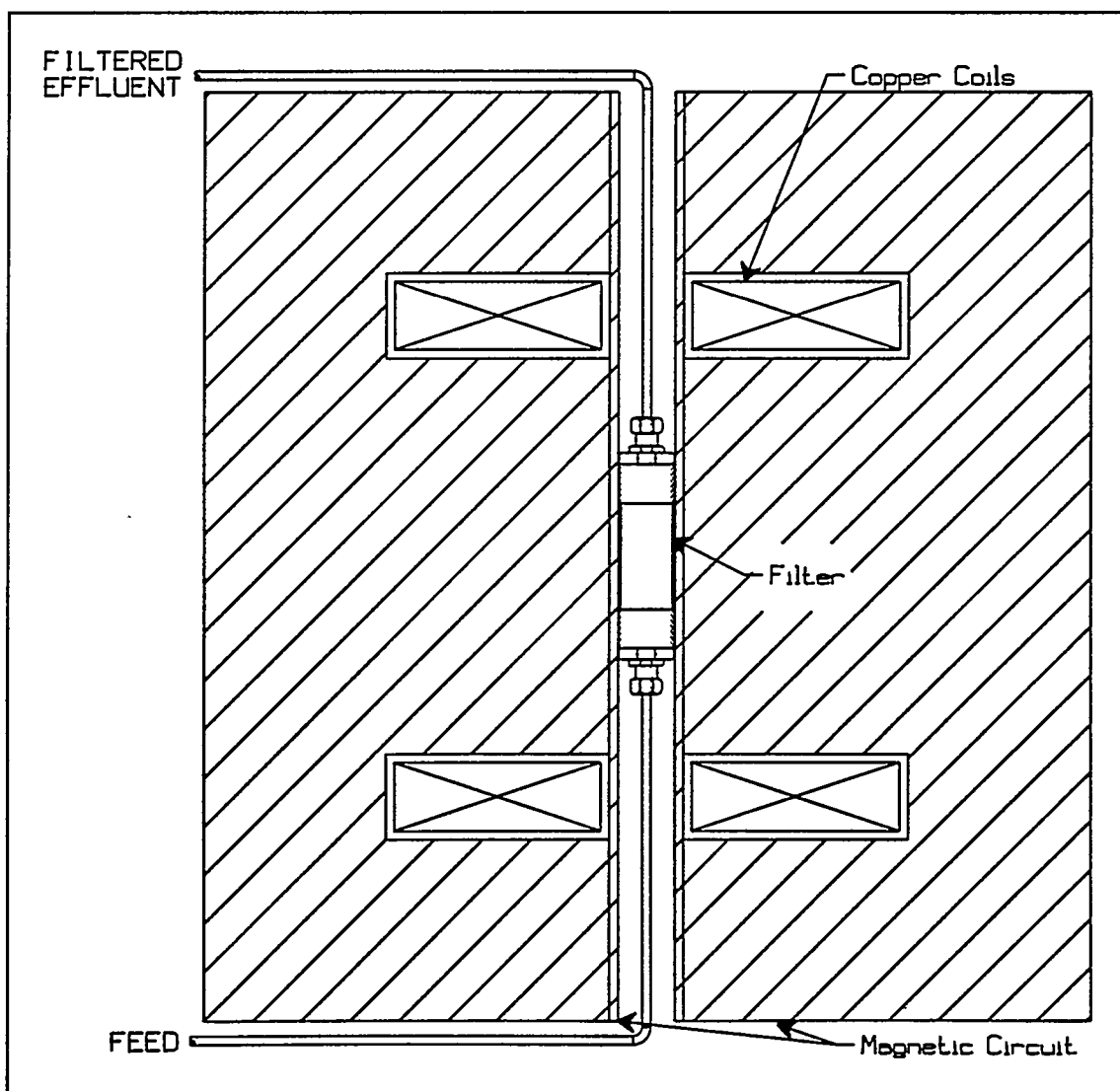


Figure 2 CROSS SECTION OF THE HGMF

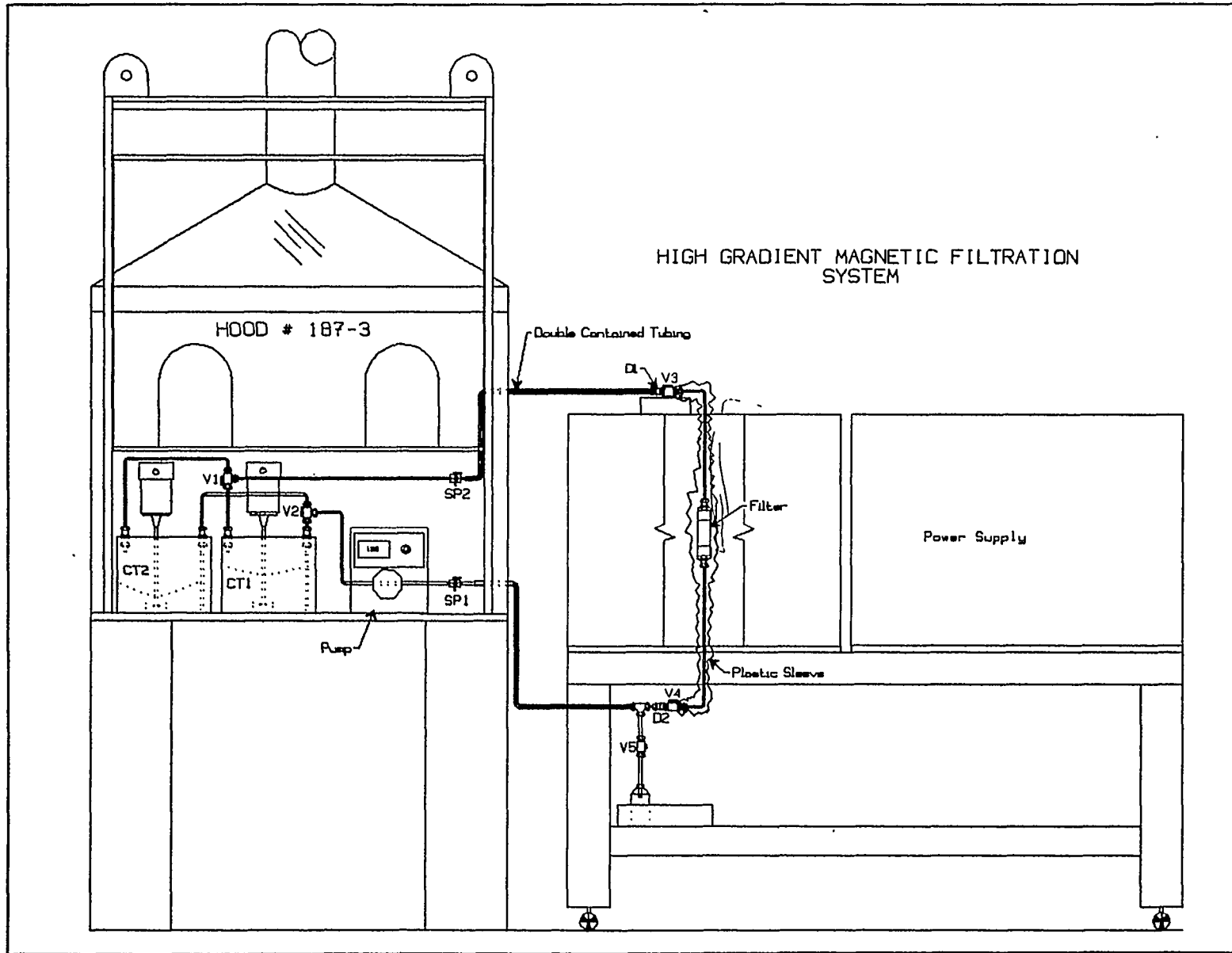


Figure 3 HGMF TEST SETUP

Table 1
 TESTS

TEST 1						
Valve Position	Valve 1	Valve 2	Valve 3	Valve 4	valve 5	Comments
Pass 1	Away from CT1	Towards CT1	Open	Open	Closed	Magnetic field Off. Mechanical filtration pass.
Pass 2	Towards CT1	Away from CT1	Open	Open	Closed	Magnetic field and chiller on. cooling water valves open.
Pass 3	Away from CT1	Towards CT1	Open	Open	Closed	Magnetic field and chiller on. Cooling water valves open.
Pass 4	Towards CT1	Away from CT1	Open	Open	Closed	Magnetic field and chiller on. Cooling water valves open.
TEST 2 repeat of TEST 1						