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ROCKY FLATS PLANT SOIL TREATMENT BENCH-SCALE TREATABILITY STUDIES

FINAL REPORT TO EG&G ROCKY FLATS, INC.
ROCKY FLATS PLANT
GOLDEN, CO 80402-0464

EG&G ROCKY FLATS SUBCONTRACT
ASC 353007J03

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CONTENTS

FIGURES

TABLES

ACRONYMS

Americium	Am
Centimeters	cm
Comprehensive Environmental Response, Compensation, and Liability Act	CERCLA
Counts per minute	cpm
Decontamination Factor	DF
Department of Energy	DOE
EG&G Rocky Flats, Inc.	EG&G
Environmental Protection Agency	EPA
Field Instrument for Detection of Low-Energy Radiation	FIDLER
General Atomics	GA
Individual Hazardous Substance Sites	IHSSs
Interagency Agreement	IAG
Methyl-isobutyl Carbinol	MIBC
Nuclear Remediation Technologies	NRT
Plutonium	Pu
Quality Assurance/Quality Control	QA/QC
Resource Conservation and Recovery Act	RCRA
Rocky Flats Plant	RFP
Superfund National Priority List	NPL

1. INTRODUCTION

This report describes the preliminary treatability studies performed by General Atomics (GA) personnel on a sample of contaminated surficial soil from the Rocky Flats plant. The studies focused on the removal of plutonium (Pu) and americium (Am) contamination from the soil using techniques previously employed by GA on the treatment of contaminated soils from other sites. GA funded the treatability studies and EG&G Rocky Flats funded the preparation of this report.

1.1. SITE DESCRIPTION

1.1.1. Site Name and Location

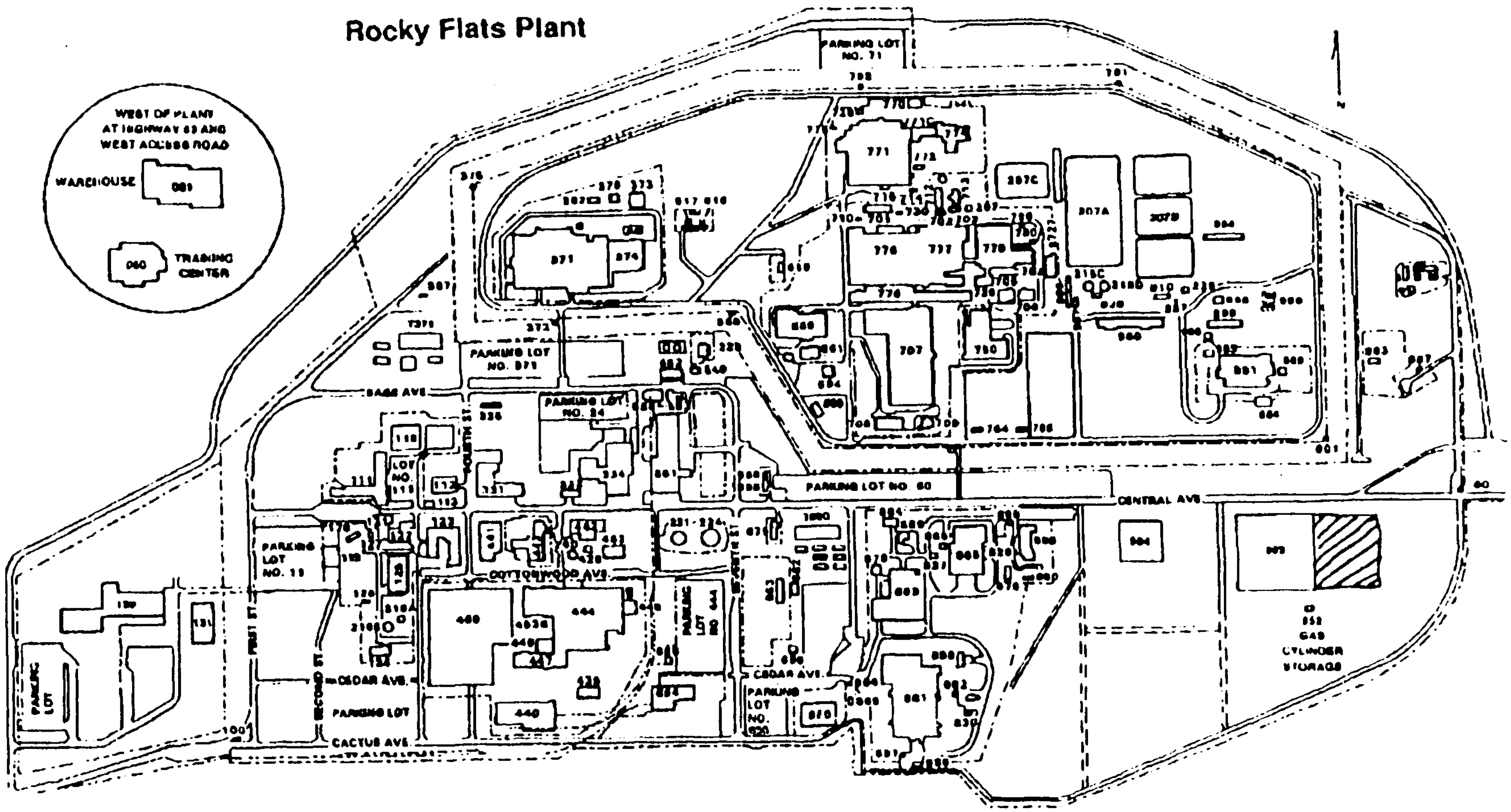
The Rocky Flats plant site covers approximately 6,550 acres in Jefferson County, Colorado, Sections 1 through 4 and 9 through 15 of R70W T25. The facility is centered at 105 degrees 11'30" west longitude, 39 degrees 53' 30" north latitude. This location is 16 miles northwest of Denver and 9 to 12 miles from the communities of Boulder, Broomfield, Golden, and Arvada. It is approximately bounded on the north by State Highway 128, on the west by State Highway 93, on the south by State Highway 72, and on the east by Jefferson County Highway 17 (Indiana Street). Major plant structures, including all production buildings, are located within a 384-acre security fenced area. The plant is divided into several areas consisting of different operational complexes. The major production facilities and associated complexes are in the 300, 400, 600, 700, 800 and 900 areas (Fig. 1-1). The soil sample location is indicated on Fig. 1-1.

1.1.2. Remediation History

The Rocky Flats plant is a key facility in the federal government's nationwide nuclear weapons research, development, and production complex. It supports the nuclear weapons program and other work related to national defense with unique processing capabilities for fabricating weapons components from Pu, uranium, beryllium and stainless steel. The plant also plays a key role in the decommissioning and maintenance of nuclear weapons and would be instrumental in the implementation of any future arms reduction agreement.

Construction began in 1951, and initial operations occurred the following year. The plant was operated at that time by Dow Chemical U.S.A., a unit of the Dow Chemical Company, for the U.S. Atomic Energy Commission. When the Energy Reorganization Act of 1974 dissolved the U.S. Atomic Energy Commission, federal government responsibility for the plant was assigned to the Energy Research and Development Administration.

Rocky Flats Plant



▨ SAMPLING AREA FOR PLUTONIUM CONTAMINATED SOILS

Fig. 1-1. Rocky Flats plant plot plan

On July 1, 1975, Rockwell International assumed operation of the plant for the Energy Research and Development Administration. Two years later, the Energy Research and Development Administration was changed to the U.S. Department of Energy, the federal agency currently responsible for the plant. EG&G Rocky Flats, Inc. (EG&G) took over the operating contract from Rockwell International on January 1, 1990.

The plant is involved in manufacturing the "pit assembly" Pu component of nuclear weapons, reprocessing scrap and Pu from dismantled weapons, laboratory research on properties of nuclear materials, and fabrication of other metals such as steel and beryllium. Wastes produced include hazardous wastes, low-level and transuranic radioactive wastes, and mixed wastes. Historically, these wastes have been either disposed onsite, stored in containers onsite, or disposed off site. The Rocky Flats plant was proposed for inclusion on the Superfund National Priority List (NPL) in 1984, and included on the NPL in October 4, 1989, Federal Register. Cleanup has been conducted under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The Environmental Protection Agency, DOE, and the Colorado Department of Health are involved in assessment and cleanup at the plant. A draft Interagency Agreement between the three was released for public comment in December 1989 and was produced to clarify the roles and responsibilities of each agency.

Various areas at the Rocky Flats Plant (RFP) are being closed and/or remediated in accordance with the provisions of the 1991 Interagency Agreement (IAG) signed between the U.S. Department of Energy (DOE), The U.S. Environmental Protection Agency (EPA), and the State of Colorado (IAG 1991) to ensure protection of human health and the environment. The IAG integrates the closure and corrective action provisions of the Resource Conservation and Recovery Act (RCRA) with the remediation requirements contained in the CERCLA. The Various areas to be closed or remediated, called Individual Hazardous Substance Sites (IHSSs), are divided into 16 Operable Units.

1.2. WASTE STREAM DESCRIPTION

The waste stream of concern in this study is contaminated surficial soil from an area east of the 903 Pad (IHSS 155). The following is a brief discussion of the history of IHSS 155, obtained from the "Phase II RI/FS Work Plan Rocky Flats Plant 903 Pad, Mound, and East Trenches Areas Operable Unit No. 2," December 1989:

> *During drum removal and cleanup activities associated with 903 Drum Storage Site (903 Pad), winds redistributed Pu to the south and east. The most contaminated area was immediately adjacent to the pad to the south and southeast. A survey at the time of the drum removal project and subsequent annual soil sampling from 1969 to 1972 show a maximum Pu concentration of 2,258 pCi/g in the top five centimeters (cm) of soil at the 903 Lip Site.*

Soil cleanup efforts were undertaken in 1976, 1978, and 1984 to remove Pu-contaminated soils from the 903 Lip Site. The 1976 soil removal consisted of hand-excavating contaminated soils until the contamination levels were below the lower detection limit of the Field Instrument for Detection of Low-Energy Radiation (FIDLER). The lower detection limit of the FIDLER is 250 counts per minute (cpm). The excavated area was covered with clean topsoil and reseeded with native grasses.

During the 1978 soil removal project, all soil that exceeded 2,000 cpm, as determined by the FIDLER, was removed. The excavated areas were resurveyed with the FIDLER, and soil removal continued until background readings (approximately 250 cpm) were obtained. Topsoil was added to the excavated areas, and the site was reseeded with native grasses.

A third soil removal effort was performed during 1984. An area along the eastern edge of the 903 Lip Site was excavated and backfilled with clean topsoil.

1.3. TREATMENT TECHNOLOGY DESCRIPTION

Soil treatment technologies used in the study to reduce of level Pu and Am contamination in the surficial soil were:

a. flotation

b. attrition scrubbing

c. acid leaching

These technologies are adaptations of techniques commonly used in commercial ore refining.

All studies were performed in the GA radiochemistry laboratory using bench-scale equipment.

2. CONCLUSIONS AND RECOMMENDATIONS

2.1 CONCLUSIONS

2.1.1 Sample Characterization

Dry screening of the 27.5 Kg (as-received) soil sample yielded the following weight fractions:

Fraction	Weight Fraction
+3/8 in.	0.274
–3/8 in.	0.723
dried vegetation	0.003

Clay (–400 mesh material) is the major fraction in the –3/8-in. split (about 45 weight percent). Americium-241 (Am-241) and Pu concentrations in the clay fraction are approximately twice those in the remainder of the –3/8-in. fraction. Thus, about three-fourths of the contamination present is in the clay (see Fig. 4-1).

2.1.2. Flotation/Attrition Scrub Tests

Two treatability studies were made on intermediate sized fractions using a combination of flotation followed by attrition scrubbing in 6 molar sodium hydroxide. Neither test yielded significant Pu removal. The following table summarizes the results.

			Product Concentration	
Test	Feed Size	Pu DF*	Pu (pCi/g)	Am (pCi/g)
1	–8/+400 mesh	1.22	15.4	—
2	–4/+400 mesh	2.03	9.88	2.10

* DF (decontamination factor) is defined as the concentration of the contaminant in the feed divided by its concentration in the product.

Analytical results and weight measurements are shown in Figs. 4-2 and 4-6.

2.1.3. Leaching Tests

Six separate leaching (stir washing) studies were performed. Four studies used a –400 mesh (clay) feed and two studies used a –4/+400 mesh fraction. In all tests, sufficient liquid was added to yield a slurry containing 25 to 30 percent soil. The slurry was maintained at 70°C. All slurries were agitated sufficiently to suspend the solids. Two tests used a single contact (one stage) and four tests used two successive contacts (two stage). Test highlights are tabulated on the next page.

Test	Soil Fraction	No. of Stage	Reagents	Leach Time	Decontamination Factor		Product Concentration	
					Pu	Am-241	Pu (pCi/g)	Am-241 (pCi/g)
1	–400 mesh	1	HNO_3 + HF	6 hrs	1.6	2.9	26.1	2.13
2	–400 mesh	1	48% HBr	6 hrs	2.6	3.8	15.8	1.62
3	–400 mesh	2	48% HBr/ 48% HBr + HF	3 hrs/ 3 hrs	4.0	6.2	10.4	1.06
4	–400 mesh	2	6.8% HBr/ 24% HBr	3 hrs/ 3 hrs	2.6	7.9	15.1	0.847
5	–4/+400 mesh	2	HNO_3 + HF/ HNO_3 + HF	3 hrs/ 4 hrs	14.1	—	1.65	—
6	–4/+400 mesh	2	48% HBr/ 48% HBr	4 hrs/ 4 hrs	6.3	—	3.81	—

Analytical results are listed in Appendix II.

With the clay fraction, hydrobromic acid yields better soil decontamination than a mixture of nitric and hydrofluoric acid, strong hydrobromic acid is better than dilute acid and a two-stage contact is better than a single stage. Strong hydrobromic acid dissolves a significant fraction of the clay.

With the –4/+400 mesh fraction, a nitric acid/hydrofluoric acid mix is superior to hydrobromic acid but dissolves twice as much of the soil. Leaching is more effective with the –4/+400 mesh fraction than with clay. Leaching of the larger-sized fraction yields much better soil decontamination than does flotation/attrition scrubbing.

2.2. RECOMMENDATIONS

Flotation/attrition scrubbing resulted in low decontamination factors (less than 2) and should no longer be considered for Rocky Flats plant soil decontamination. Flotation, however, should be considered as one of the possible methods for isolating the clay fraction (which contains the highest contamination levels) from the bulk of the soil on a production scale. Specific testing should be undertaken to explore isolation of the clay fraction by flotation and to determine suitable frother/conditioner combinations and concentrations.

Additional bench-scale leaching tests should be done, with emphasis on countercurrent flow of the leaching acids and soil, to optimize operating conditions and reagent concentrations with due consideration to liquid waste disposal, acid recycling and costs. Promising conditions developed by these additional bench-scale tests should then be tested in engineering-scale equipment at the Rocky Flats plant site.

3. TREATABILITY STUDY APPROACH

3.1. TEST OBJECTIVES AND RATIONALE

The test objective was to conduct bench scale treatability studies on possible methods of reducing Pu and Am levels in the contaminated Rocky Flats surficial plant soil. With soil contamination, the contaminants are typically on the surface of the soil particles. Consequently, the contaminant concentration is expected to be higher in the small-sized (fines) particle fractions. Therefore, treatment methods should focus on separating the fine particles from the larger (coarse) particles followed by leaching of fines. An alternate treatment is to abrade the contaminants from the particle surfaces followed by separating the fines generated by abrasion from the bulk of the soil.

3.2. EXPERIMENTAL DESIGN AND PROCEDURES

The treatability studies were designed to first separate the coarse particle fractions which have low contaminant concentrations from the smaller-sized particle fractions. Dry screening was used for this separation. Fine particles clinging to the large-sized particles were washed off with water, dried and combined with the smaller-sized fraction for further treatment. No further treatment of the coarse fraction was done. The very fine clay fraction, which has the highest contaminant concentration, was separated from the smaller-sized particle fraction by wet sieving and treated separately. Leaching was used to remove contaminants from the clay fraction. Two alternate approaches were used for the intermediate-sized fraction — leaching and a combination of flotation and attrition scrubbing. Flotation removes fines and attrition scrubbing abrades surface contaminants from the particulates. Leaching was done by agitation of a mixture of the soil fraction and leaching agent (stir washing).

Am-241 concentrations in the various fractions were determined by gamma analysis. Following gamma analysis, the samples were used in the treatability tests. Pu analysis requires sample dissolution. The sample could no longer be used for further treatability studies.

3.3. EQUIPMENT AND MATERIALS

Much of the equipment used consists of normal chemistry laboratory hardware. This includes screens, sieves, mixers, thermometers, heating plates, balances, filters, etc. Specialized equipment used was a bench-scale WEMCO attrition scrubber with a float cell attachment, a Canberra S-100 gamma ray spectrometer and a Packard Instruments liquid scintillation counter. Stir wash contacts (leaching) used four-liter beakers with agitation provided by a laboratory mixer. Standard analytical chemistry laboratory grade chemicals were used for stir washing and attrition scrubbing.

Two types of reagents are used in the flotation treatment. The first, frothers, helped form a stable froth of air bubbles upon which the desired fractions float to the surface of the agitated and aerated slurry. The frother consists of three parts methyl-isobutyl carbinol (methylamyl alcohol) to one part F65 (Aerofroth® 65 frother) (MIBC/F65). MIBC is made by EM Science, Gibbstown, New Jersey. F65 is a polypropylene glycol manufactured by American Cyanamid Co., Wayne, New Jersey.

The second flotation reagent is a collector (promoter). This reagent type provides the sediments with a water repellent, air-avid coating that attracts air bubbles. The conditioners used are emulsified high-density mineral oil and Armac® T.

The mineral oil was supplied by Eastman Kodak Co., Rochester, New York. It consists of 20 parts water, 2 parts mineral oil and 0.3 parts emulsifying agent. Armac® T is a tallow amine acetate manufactured by AKZO Chemicals, Inc., Chicago, Illinois. It was added as a 1 wt % aqueous solution.

3.4. DATA MANAGEMENT

The laboratory technician performing the treatability studies recorded the steps taken and measurements in GA Laboratory Notebook No. 10802. Measurements and analyses were reviewed by the laboratory manager for consistency and remeasurement and/or reanalysis made when deemed to be necessary. Consistent with the feasibility nature of the studies, only single analyses were performed on the various samples.

3.5. DEVIATIONS FROM THE WORK PLAN

The original work plan is contained in a fax from GA to EG&G Rocky Flats[1]. Deviations include

1. Flotation was added as a treatability method.
2. Untreated mineral appearance was not described.
3. The bulk density was not determined.
4. Wet sieve sizes were altered.

This work plan is included as an appendix to this report.

[1] Fax from Brenda Anderson to Mike Harris, "Work Plan for Initial Bench-Scale Treatability Study — Rocky Flats Plant — O.U.2 Soil Contaminated with Pu and Am.," June 10, 1992.

4. RESULTS AND DISCUSSION

These experimental tests were limited to bench scale treatability studies for the removal of Am and Pu contamination from a sample of Rocky Flats plant surficial soil. The studies did not address the treatment and disposal of secondary wastes arising from the various treatments.

4.1. DATA ANALYSIS AND INTERPRETATION

The Rocky Flats plant soil sample sent to GA (lot no. SS01107ST) was divided into several portions and fractions as shown in Fig. 4-1. Two series of tests were performed on selected portions. Figure 4-1 and subsequent figures contain the measured weights as well as Am-241 and Pu analyses. Appendix II contains a tabulation of the analyses.

4.1.1. Analysis of Waste Stream Characteristics

Based on prior experience involving treatment of contaminated soil, as well as previous characterization work at the RFP site[2], it was found that the larger sized soil components contain only minor quantities of contaminants. The contaminants are on the surface of the soil particles. Per unit of weight, the smaller the particle, the greater the surfaces area and attendant contamination. Larger-sized soil particles are also more difficult to analyze for Am-241 and Pu as well as to process through the bench-scale equipment.

The entire as-received soil sample, weighing about 27.5 kg, was screened using a 3/8-in. screen. Dried vegetation (grass) was manually removed from the sample. No analysis or treatment of the dried vegetation was undertaken.

The –3/8-in. fraction was split into three equal samples — a test sample, a backup sample and an archive sample. The test sample was then split into five subsamples and three of these were used in the subsequent treatability studies. Fine soil particles adhering to the +3/8-in. soil fragments were removed by washing, dried (100°C overnight) and combined with the dried subsample used in the first test series. On a dried-weight basis, the –3/8-in. fines washed from the +3/8-in. fraction was about 9% of pre-washed weight. Significant moisture was present in the as-received sample with smaller particle size fractions retaining more moisture than the larger particle size fractions.

2 Private communication from Scott Hollowell, EG&G Rocky Flats, 6/9/94.

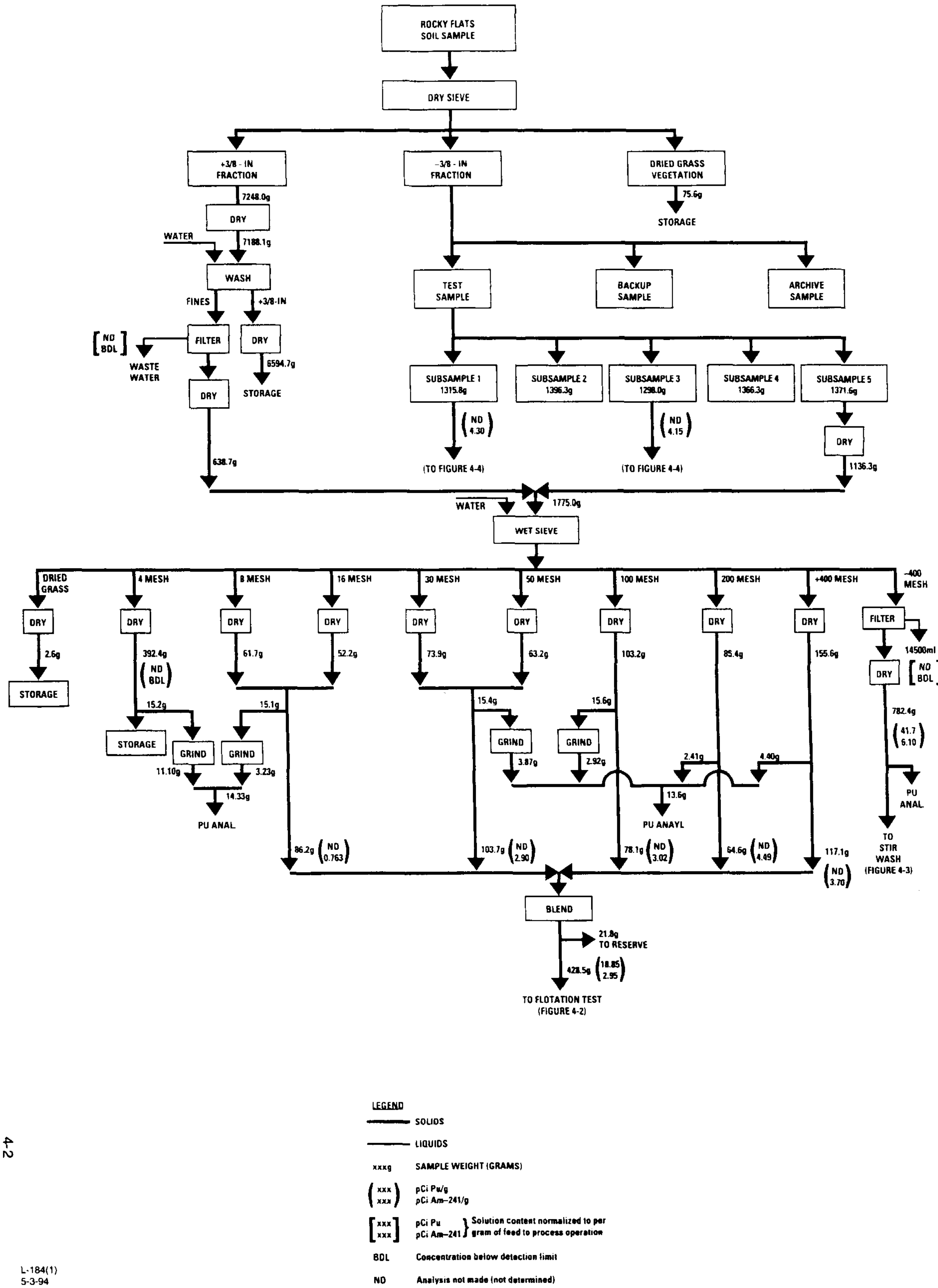


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Fig. 4-1. Soil sample screening and test sample preparation

Weight losses on drying at 100°C overnight were as follows:

entire as-received sample	12.6 %
+3/8-in. fraction (prior to washing)	0.83%
-3/8-in. fraction	17.16%

Estimated weight distribution (dried basis) of the as-received soil sample is

+3/8-in. fraction (after washing)	27.4 wt %
–3/8-in. fraction	72.3 wt %
dried vegetation	0.3 wt %

After drying and weighing, the fines washed from the +3/8-in. fraction and subsample 5 of the –3/8-in. test sample, were combined (weight = 1775 g) and wet-sieved into several fractions. The weight percent breakdown is listed in Table 4-1.

Table 4-1
Soil Sample Weight Distribution by Particle Size

Fraction	Wt %	Fraction	Wt %
dried vegetation	0.15	+50 mesh	3.57
+4 mesh	22.14	+100 mesh	5.82
+8 mesh	3.48	+200 mesh	4.82
+16 mesh	2.94	+400 mesh	8.78
+30 mesh	4.17	–400 mesh	44.14

Since all the fines washed from the +3/8-in. fraction were combined with this subsample, the size distribution is not necessarily representative of the entire –3/8-in. fraction. Because of the low levels of Am-241 present, Am-241 determination by gamma spectrum requires at least 100 cubic centimeters of soil. This required combining the +8 and +16 mesh fractions as well as the +30 and +50 mesh fractions. The Am-241 concentration in the +4 mesh fraction was below its detection limit. Consequently, this fraction was excluded from further testing. Proportionate samples were taken from the various +400 mesh fractions and combined for Pu analyses. This analysis was used for the Pu content in the feeds to the treatability tests on the –8/+400 mesh blends. Similarly, the Pu analysis of a sample from the –400 mesh fraction provided the basis for the Pu content of the feeds to the treatability tests on the –400 mesh fraction.

As noted in Table 4-2, about 75% of the Pu and Am-241 contamination is in the –400 mesh fraction.

Initial Flotation and Attrition Scrub Tests

In the first test series, a two-step process of flotation and attrition scrubbing was used for treatment of the –8/+400 mesh blend from the test sample preparation (see Fig. 4-1). Flotation should remove the fines which have higher-than-average contaminant concentration.

Table 4-2 Pu and Am-241 Distribution in Sample				
Fraction	Weight (dry) Grams	Weight (dry) %	% of Am-241	% of Pu
–8/+400 mesh	595.2	43.2	26.9	25.6
–400 mesh	782.4	56.8	73.1	74.4

Attrition scrubbing should abrade contaminants off the particle surfaces which are subsequently isolated by sieving and filtration. Attrition scrubbing uses a thick slurry (about 65% pulp density). Fig. 4-2 shows the process steps, material flow and contaminant analyses. Am-241 concentrations in the effluent streams were too low for measurement. Consequently, Am-241 decontamination factors could not be determined. Table 4-3 summarizes the test results.

Table 4-3 Initial Flotation/Attrition Scrub Test Results						
Process Step	Reagents	Weight Ratio (Dry Basis) (+400 Mesh Product/Feed)	Pu DF $\left(\frac{\text{Feed Conc.}}{\text{Product Conc.}}\right)$	Material Balances $\left[\frac{\text{Products}}{\text{Feed}}\right]$ Ratio: Weight	Material Balances Ratio: Pu	Product Concentration (pCiPu/g)
Flotation	mineral oil conditioner	0.80	1.0	0.99	1.0	19.0
Attrition Scrub	6.0M NaOH	0.92	1.23	0.96	–	15.4
Overall		0.75	1.22	0.96	–	–

Flotation was of no value in reducing the Pu concentration in the –8/+400 mesh fraction. Attrition scrubbing resulted in only a minor reduction of the Pu concentration.

Initial Stir Wash Tests

In stir washing, a mixture of soil and a leaching agent is agitated at an elevated temperature to dissolve the contaminants. Subsequent filtration separates the dissolved material from the soil. In the bench-scale tests, sufficient reagent was used to yield a pulp density of 25 to 30 percent. Agitation times were 6 hours at 70°C. The initial stir wash tests used portions of the –400 mesh fractions (see Fig. 4-1). One test used HNO_3/HF solution and the other an aqueous solution containing 48 weight percent HBr. Figure 4-3 illustrates the stir wash conditions, quantities and analyses. Table 4-4 summarizes the test results.

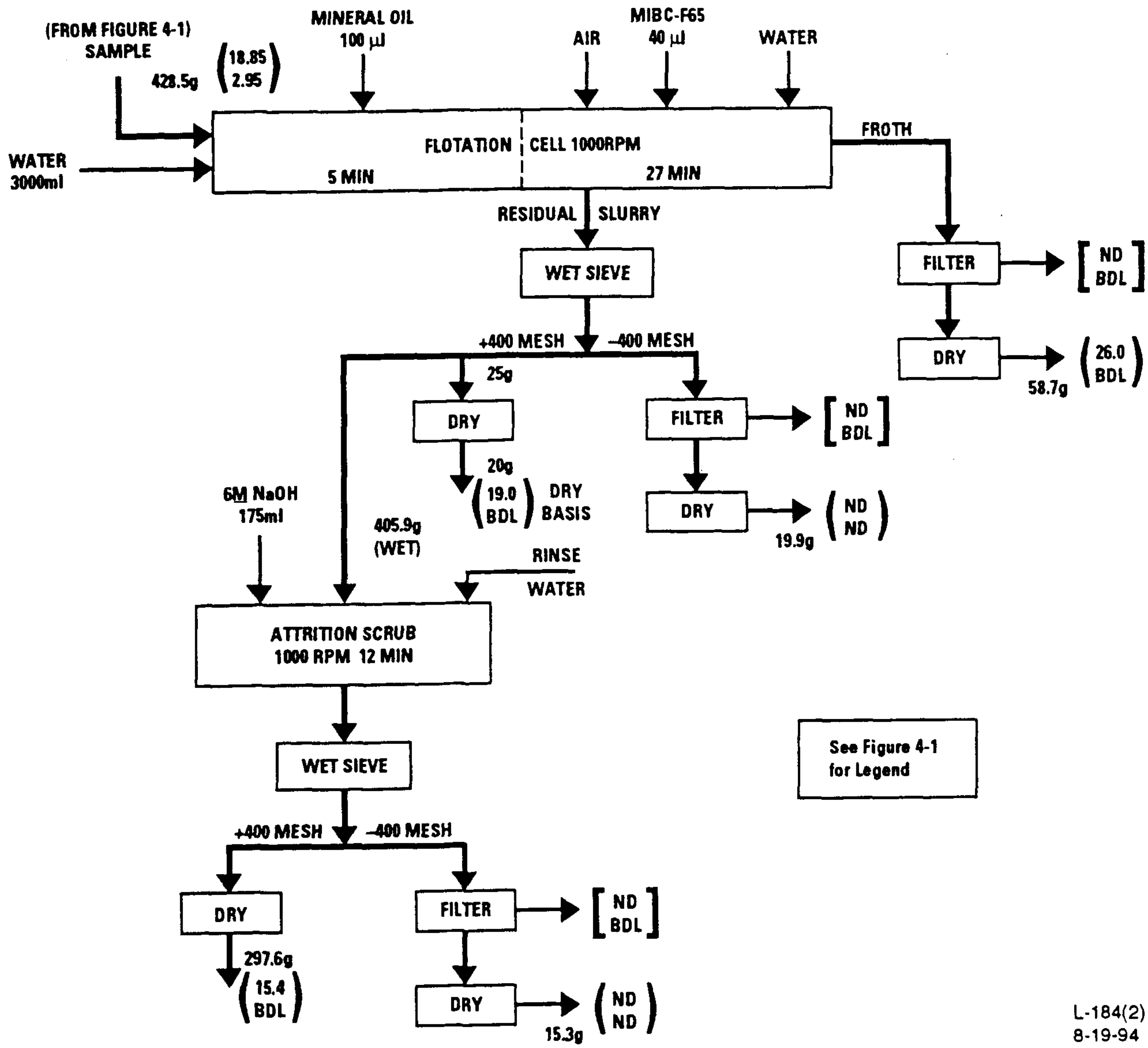

Fig. 4-2. Initial flotation and attrition scrub tests

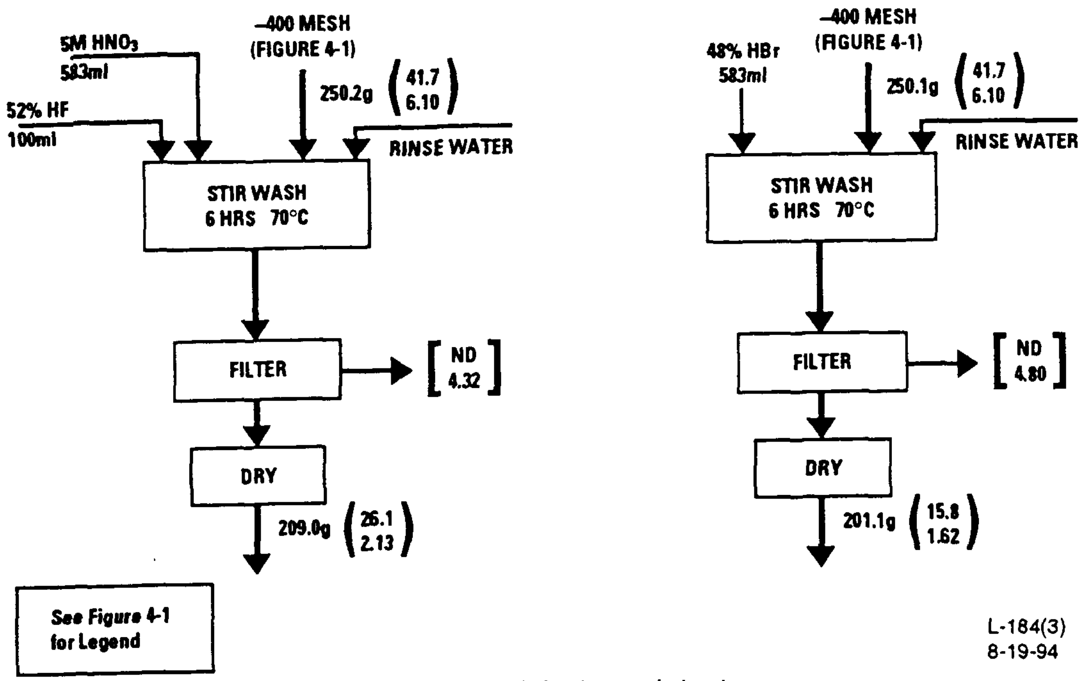

Fig. 4-3. Initial stir wash tests

Table 4-4 Initial Stir Wash Test Results					
		DF		Product Concentration (pCi/g)	
Stir Wash Test	Wt % Soil Dissolved	Pu	Am-241	Pu	Am-241
HNO_3/HF	16.5	1.6	2.9	26.1	2.13
48% HBr	19.6	2.6	3.8	15.8	1.62
Note: Both tests used –400 mesh soil fraction, 6 hour agitation at 70°C.					

The initial stir wash tests yielded modest decontamination factors with HBr better than HNO_3/HF and Am-241 better than Pu. Both reagents, however, dissolved a significant fraction of the feed (16 to 20 percent).

Preparation of Feed Samples for Second Test Series

Subsamples 1 and 3 (–3/8-in.) from the initial soil sample screening (Fig. 4-1) were wet sieved to isolate the +4 mesh and –400 mesh fractions. Both +4 mesh fractions and the –400 mesh fraction from subsample 1 were set aside and not used in the second test series. Oven drying of the –400 mesh fraction yielded a hard monolith (a brick) which required vigorous mechanical effort to break down. This obviously altered the nature of the fraction. Consequently, the –400 mesh fraction from subsample 2 to be used for additional treatability tests was air dried only. This consisted of placing the wet material from filtration in a pan and allowing to dry at room temperature for several days. The air-dried soil still retained significant moisture (approximately 50%). The –4/+400 mesh fractions from both subsamples were blended, split, analyzed for Am-241, re-blended and split into feed samples for the second test series. Figure 4-4 shows the material flow paths, quantities and analyses. The feed samples produced were not analyzed for Pu. Pu concentrations in the feed were calculated based on Pu analyses and quantity measurements of the product and effluent streams.

Stir Wash Treatment of –4/+400 Mesh Fraction

These two stir wash treatability tests used the same reagents as the initial tests (HNO_3/HF and 48% HBr) but differed in two important aspects. Each of these second series tests used the –4/+400 mesh fraction rather than the –400 mesh fraction and each test was a two-stage test. Figure 4-5 shows the flow paths, materials, quantities, analyses and stir wash conditions used for these tests. Table 4-5 summarizes the test results. Am-241 material balances and decontamination factors were not determined because the concentrations in the effluent and product streams were too low to analyze.

Good decontamination factors were obtained by both acids. Overall, the HNO_3/HF treatment resulted in a better DF than the 48% HBr (by a factor of 2) but dissolved twice as much of the soil.

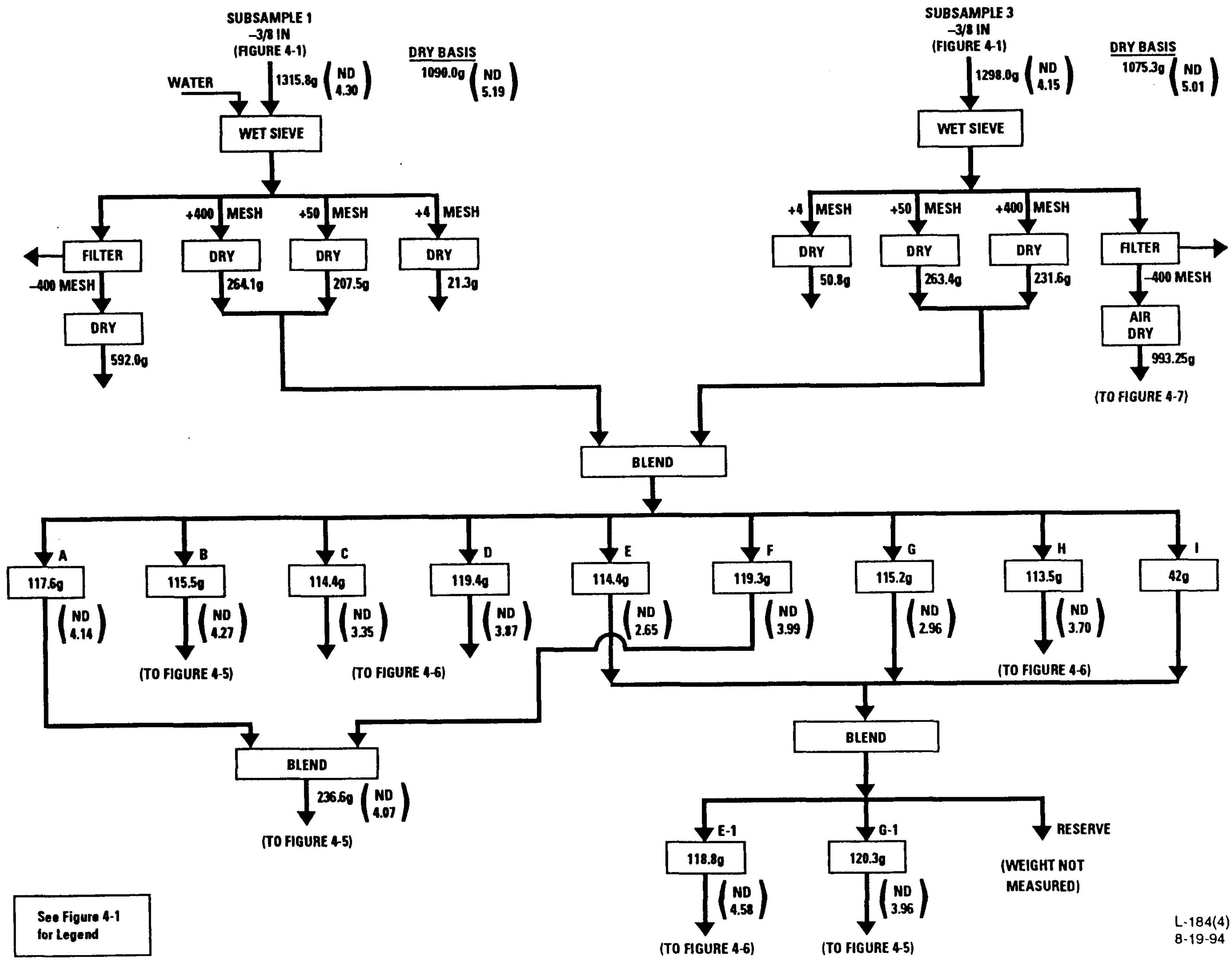

Fig. 4-4. Test feed specimen preparation — second test series

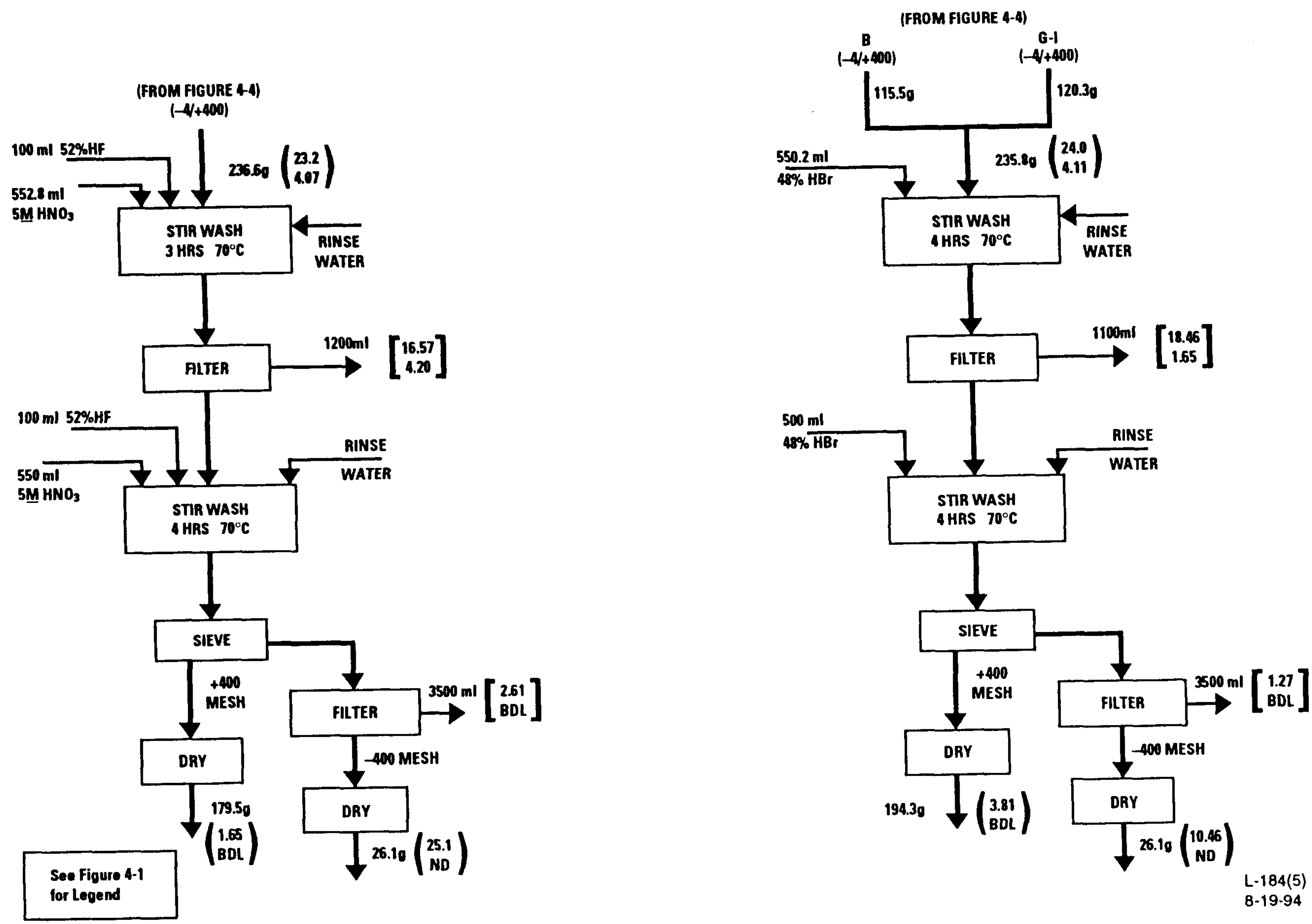

Fig. 4-5. Second test series — stir wash treatment of –4/+400 mesh fraction

Table 4-5 Stir Wash Treatment of –4/+400 Mesh Fraction							
		% Pu in Acid			Overall Pu DF	Production Concentration (pCi/g)	
Stir Wash Test	Wt % Soil Dissolved	1st Stage	2nd Stage	% Pu in Filter Fines	(Feed Conc. / Product Conc.)	Pu	Am-241
HNO_3/HF	13.1	71.4	11.3	11.9	14.1	1.65	—
48% HBr	6.5	76.8	5.3	4.8	6.3	3.81	—

Note: Both tests used a 2-stage stir wash at 70°C. The first and second stage stir wash times were 3 hours and 4 hours for HNO_3/HF and 4 hours each for the 48% HBr.

Second Flotation/Attrition Scrub Test

Four of the –4/+400 mesh feed splits from the subsample 1 and 3 blend (see Fig. 4-4) were used as feed to the second flotation/attrition scrub treatability test. This test was similar to the initial flotation/attrition scrub test (Fig. 4-2) except Armac® T was also added as a conditioner. Figure 4-6 illustrates the material flows and quantities, operating conditions, as well as the Am-241 and Pu analyses. Test results are summarized in Table 4-6.

Table 4-6 Second Flotation/Attrition Scrub Test Results							
		Weight Ratio (Dry Basis)	DF (Feed Conc. / Product Conc.)		Material Balances	Product Concentration (pCi/g)	
Process Step	Reagents	(+400 Mesh Product/Feed)	Pu	Am-241	(Feed Weight / Product Weight)	Pu	Am-241
Flotation	mineral oil conditioner MIBC-F65 frother Armac® T	0.82	1.34	—	0.97	14.91	—
Attrition Scrub	6.0M NaOH	0.92	1.51	—	1.00		
Overall		0.75	2.03	1.85	0.97	9.88	2.10

Although the Pu decontamination factors were better than those attained in the initial flotation/attrition scrub test (overall 2.01 versus 1.22), the Pu DF is still low. Pu decontamination factors achieved by stir washing of the same soil fractions were at least a factor of three higher. Flotation, however, may provide a production-scale method of separating the soil fines (–400 mesh) from the bulk of the soil.

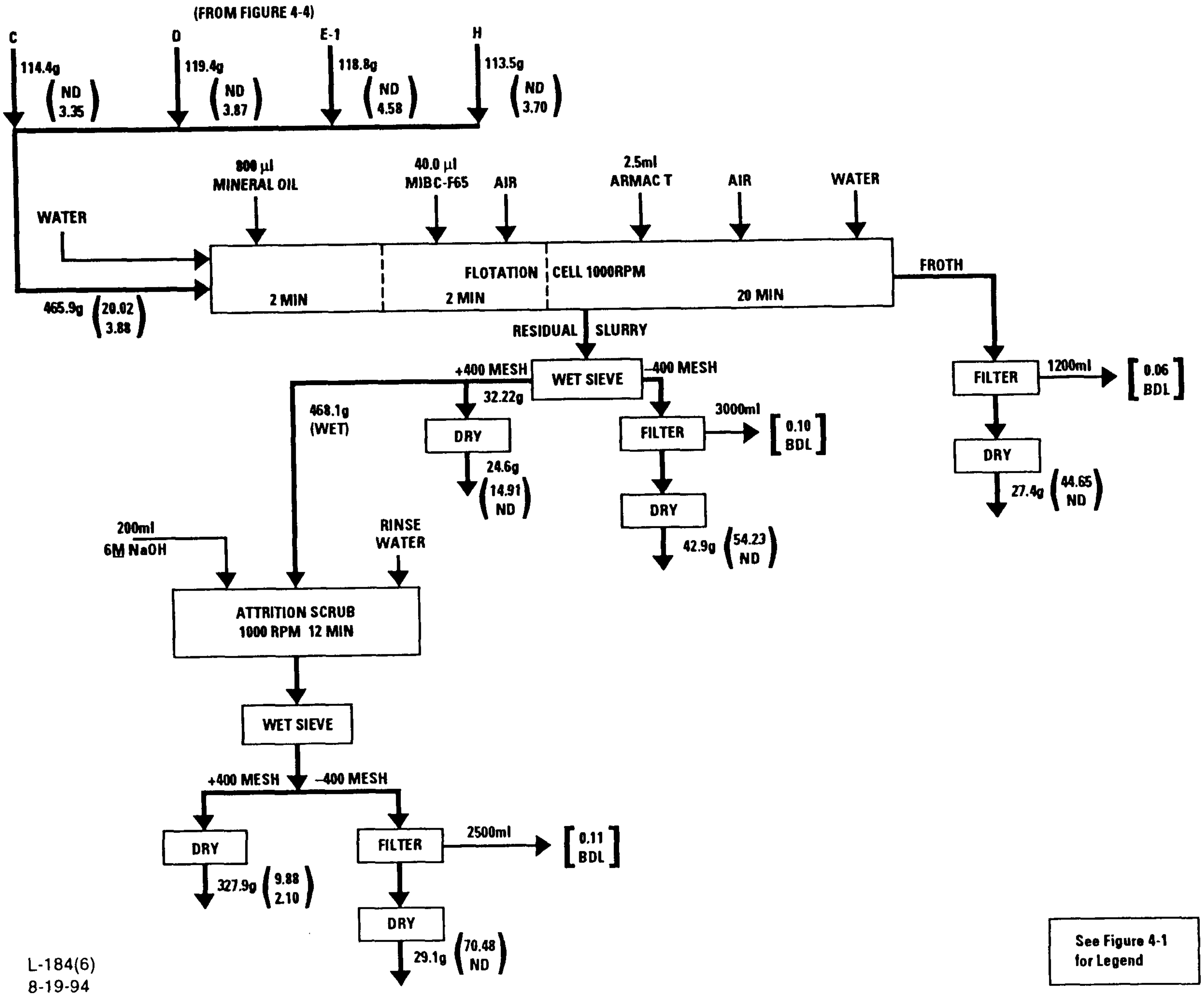

Fig. 4-6. Second flotation/attrition scrub test

Second Stir Wash Tests with –400 Mesh Fraction

The final treatability tests performed were stir washes of sample splits from the –400 mesh fraction sieving of subsample 3 (see Fig. 4-4). The air-dried –400 mesh fraction was split into seven portions. Six portions were weighed and analyzed for Am-241 then portions pairs were combined to make up the feed used for three stir wash tests. Portion seven was weighed, oven dried and reweighed to provide a factor for converting the stir test feeds to a dry weight basis. One attempted stir wash test used a mixture of 24% HBr and a small quantity of concentrated HF. Some of the material overflowed the stir wash beaker causing this test to be aborted.

Both of the completed stir wash tests used two stages with each stage lasting 3 hours at 70°C. Each stage in each test used different leaching agents. Figure 4-7 shows the material quantities, flows, reagents and the Am-241 and Pu analyses for these tests. Table 4-7 contains a summary of the test results. The stir washes are designated by the identification letters assigned to the splits from the –400 mesh fraction.

Table 4-7
Second Stir Wash Tests of with –400 Mesh Fraction

Stir Wash Test	Reagents	Wt % Soil Dissolved	% Pu Removed 1st Stage	% Pu Removed 2nd Stage	Overall DF (Feed Conc. / Product Conc.) Pu	Overall DF (Feed Conc. / Product Conc.) Am-241	Product Concentration (pCi/g) Pu	Product Concentration (pCi/g) Am-241
AF	1st: 600 ml 48% HBr	24.7	67.2	6.1	4.0	6.2	10.4	1.06
	2nd: 600 ml 24% HBr + 2.5 ml 52% HF							
CE	1st: 100 ml 48% HBr + 600 ml water	7.5	38.9	24.2	2.6	7.9	15.1	0.847
	2nd: 600 ml 24% HBr							

Note: Both tests used a three-hour agitation per stage and leaching temperatures of 70°C.

A comparison of the two test results indicates that Pu decontamination factor is related to the HBr concentration. More concentrated HBr yields a better Pu DF. A lower HBr concentration seems to improve Am DF but the low Am-241 concentrations increases the uncertainty of their values. The more concentrated HBr, however, dissolves a larger fraction of the soil. A comparison with the initial stir wash tests (Fig. 4-2) indicates a two stage stir wash improves decontamination. This suggests it would be advantageous to design a large-scale facility for countercurrent flow of the soil and leaching solutions.

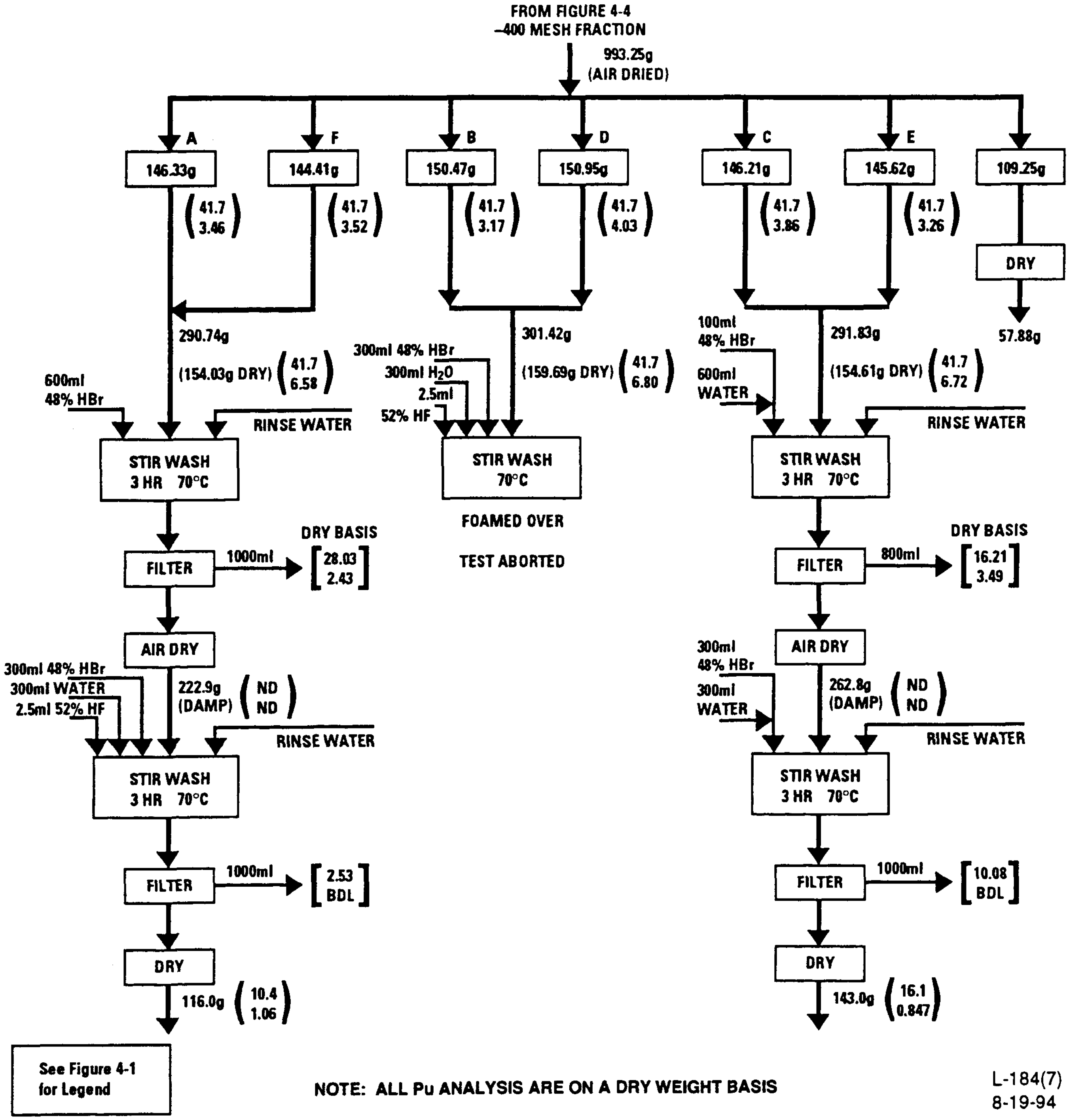

Fig. 4-7. Second stir wash tests with –400 mesh fraction

A comparison of the –400 mesh and the –4/+400 mesh stir wash results indicates the following:

a. Better decontamination is obtainable with the larger-sized fractions.

b. HNO_3/HF is better for +400 mesh material, but HBr is better for –400 mesh material.

c. A small fraction of the +400 mesh material dissolves.

4.1.2. Comparison of Test Objectives

The objective of the treatability study was to perform characterization and bench-scale treatment studies on a sample of contaminated Rocky Flats plant soil. Treatment methods to be studied were attrition scrubbing and leaching[3] (See Appendix I). The studies performed included flotation as well as attrition scrubbing and leaching. The test objectives were met and the number of treatment methods tested exceeded the work plan.

4.2. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

These treatability tests were intended only as initial feasibility studies. The level of QA/QC used was commensurate with a feasibility study. Specific QA/QC measures used were as follows:

a. A QC representative monitored the soil sample screening, sample splitting and initial sample weighing.

b. All laboratory work was reviewed by the laboratory manager. Results that appeared questionable to the manager required the work to be repeated.

c. As results became available, material balances were made to determine data consistency. Inconsistent data required repeating suspect measurements.

d. All work done was recorded in a laboratory notebook (GA Laboratory Notebook No. 10802).

4.3. COSTS/SCHEDULE FOR PERFORMING THE TREATABILITY STUDY

All costs of performing the treatability studies at GA were borne by GA. Only the cost of preparing this final report was paid by a contract with EG&G Rocky Flats.

No formal schedule was established for performing the study. Initiation of the testing was controlled by the collection, packaging and shipping of the soil sample. Over 6 months elapsed between work plan approval and sample receipt. The test work was carried out over a four-month period when laboratory personnel became available to perform the tests. Funding for report preparation was approved eleven months after completion of the experimental studies.

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3 Fax from Brenda Anderson to Mike Harris, "Work Plan for Initial Bench-Scale Treatability Study - Rocky — Flats Plant- O.U.2 Soil Contaminated with PU and Am.," June 10, 1992.

APPENDIX I

WORK PLAN FOR INITIAL BENCH-SCALE TREATABILITY STUDY
ROCKY FLATS PLANT
O.U.2 SOIL CONTAMINATED WITH Pu AND Am

JUNE 10, 1992

1. SUMMARY

1.1 INTRODUCTION

This work plan outlines the initial, bench-scale, soil cleaning, treatability study for soils contaminated with plutonium and americium from Rocky Flats Plant Operable Unit 2. NRT's objective is to demonstrate the capability of our soil cleaning process to meet the anticipated 0.9 pCi/gm Rocky Flats cleanup standard. The results of the study will identify the separation and cleaning efficiency of major process steps and provide data on the likelihood of success and applicability of NRT's technology to Rocky Flats soils.

1.2 PROJECT APPROACH

For Phase 1A work, the parameters addressed by the treatment studies are restricted to those of the soils from O.U.2. to be provided by EG&G Rocky Flats. Most of the radionuclide contamination is expected to be associated with the finest (clay) fraction of the soil. A smaller amount would be expected to be associated with the course fraction (sand and larger). Ultimately, treatment process parameters would be designed to have sufficient flexibility and throughput to accommodate the range of soil/contaminant characteristics likely to be encountered at various impacted sites across Rocky Flats. The approach for the full scale implementation of a soil cleaning volume reduction process would be divided into three phases. This work plan describes only the testing to be performed during Phase 1A.

- Phase 1: Treatability Study
  - 1A - Initial Bench-Scale Soil/Treatability Study
  - 1B - Engineering Treatability Tests/Conceptual Design
- Phase 2: Site Integrated Pilot Demonstration
- Phase 3: Site Remediation

1.3 PHASE 1A WORK SCOPE

The Phase 1A objective is to characterize the soils and contaminant distributions and to obtain sufficient bench-scale treatability test data to evaluate the effectiveness of various components of the soil cleaning process on Rocky Flats soils from O.U.2.

In order for NRT to carry out the Phase 1A work, EG&G - Rocky Flats will obtain and furnish a sample of approximately 20 to 30 kg of contaminated soil from O.U.2. EG&G will acquire the soil and composite the samples to be vertically and laterally representative of the area, with total activity level of approximately 100 pCi/gm (50 to 200 pCi/gm).

NRT will perform a series of analyses and tests on the composite soil sample to establish the physical and chemical characteristics and the behavior of the soil when subjected to bench-scale physical and chemical separation processes.

Phase 1A includes the following tasks:

- Qualitative Examination of contaminated soil
- Sieve Analysis
- Baseline activity of contaminated soil by sieve size
- Attrition Scrub Tests
- Leach Tests

The information developed during this phase will confirm the conceptual viability of the soil cleaning approach, and provide a preliminary estimate of overall volume reduction.

2. DESCRIPTION OF BENCH-SCALE TESTS

2.1 QUALITATIVE EXAMINATION

The soil will be examined to qualitatively determine handling properties, natural organic content, particle size distribution, fraction oversize, and untreated mineral appearance. This information will influence the following test types, conditions and sequence.

2.2 SAMPLE BLENDING AND SPLITTING

The soil received from Rocky Flats Plant will be thoroughly blended and split into 3 approximately equal samples. The first sample will be used for the analytical and test work, the second sample will be retained as a archive sample or for further tests and the third sample will be retained for EG&G Rocky Flats Plant.

2.3 AS RECEIVED MOISTURE AND BULK DENSITY

The as received moisture will be measured by ASTM D 2216 at 60°C. Rough bulk density will be determined by appropriate methods.

2.4 SIEVE ANALYSIS

Wet sieve 500 grams of sample to determine contaminated soil size distribution (3/8", 4, 10, 24, 35, 60, 100, 150, 200, 325, 400 mesh). All results will be reported as dry weight percent. The results from this test will influence the selection of test conditions and size splits.

2.5 BASELINE ACTIVITY

Wet sieve sufficient sample for attrition scrub and leach tests, approximately 5000 grams, into appropriate mesh fractions. Typical splits are: +4, -4/+200, -200/+325 and -325. Measure Pu and Am activity of composite, the sieve samples and the water used for screening.

Pu will be determined using representative samples and a destructive chemical analysis: acid digestion, Pu extraction followed by alpha spectroscopy (GA procedure ACD:RC-016). Am will be quantified using gamma spectroscopy. Once the baseline activity and the Pu to Am ratio have been determined, the additional activity measurements after each attrition or leach test will track Am only, assuming the Am and Pu were scrubbed or leached to approximately the same degree. Once the tests are complete, Pu measurements will be used to verify this assumption.

2.6 ATTRITION SCRUB TESTS

Attrition scrub tests will be performed on selected size splits. The typical attrition scrub sample size is 700 to 1000 grams. It is anticipated that attrition scrub reagents will be selected from the following: deionized water, dilute surfactant solution, caustic solution.

After each attrition scrub, the slurry is vacuum filtered through a fine filter and the Pu and Am activities of both filtrate and solid residue are measured. The dry weight of the solid is determined.

2.7 LEACH TESTS

Leach tests using acidic and/or basic lixivants will be performed on the selected, fine, size split. Multiple leaches may be performed on the same sample to determine the appropriate leach residence time and minimum activity level achievable.

After each leach, the slurry is filtered and the Pu and Am activities of both filtrate and solid residue are measured. The dry weight of the solid is determined.

3. TEST REPORT

The results of the bench-scale tests will be reported in a memo with a summary of the test conditions. The report will include:

- a summary of the results of the characterization tests described in sections 2.1 through 2.5 of the work plan
- an activity analysis of the residue after the final scrub or leach
- a calculation of the mass of decontaminated material as a percentage of starting mass

Since the process used to decontaminate the soil is proprietary, only a very limited description of the test conditions and reagents will be included.

4. TEST RESIDUES

All solid test residues, the remaining sample material and laboratory solid waste generated during the testing will be returned to EG&G Rocky Flats. These solid materials may include some or all of the following: contaminated soil, materials used to package the soil, plastic containers and beakers, gloves, disposable lab coats, waste paper, respirator cartridges, other laboratory trash. The volume of waste to be returned to EG&G will not exceed one 55 gallon drum. The waste will be radioactive, but will not be hazardous according to RCRA standards.

5. QUALITY CONTROL

The bench-scale, soil cleaning, treatability study for O.U.2 soils will be conducted in accordance with the GA Quality Assurance Program. All test activities will be conducted according to standard laboratory practices and procedures. Since this is not a comprehensive treatability study, duplicate samples will not be routinely analyzed.

6. HEALTH AND SAFETY

Testing will be conducted under the safety regulations of the GA Health and Safety Plan.

APPENDIX II

Am-241 and Pu Analyses					
		Am-241			
Sample	Figure	Sample Wt (g)[1]	pCi/g	pCi/Sample	Pu
-3/8 subsample 1	4-1 / 4-4	474.1	4.30		ND[2]
-3/8 subsample 3	4-1 / 4-4	488.0	4.15		ND
+3/8 wash filtrate	4-1			BDL[3]	ND
+4 mesh fraction	4-1	137.7		BDL	ND
+8, +16 mesh fractions	4-1	98.9	0.763		ND
+30, +50 mesh fractions	4-1	89.7	2.90		ND
+100 mesh fraction	4-1	87.6	3.02		ND
+200 mesh fraction	4-1	85.4	4.49		ND
+400 mesh fraction	4-1	90.8	3.70		ND
-400 mesh fraction	4-1 / 4-3	110.5	6.10		41.7
-400 mesh filtrate	4-1			BDL	ND
Sample blend	4-1 / 4-2		[2.95][4]		18.85
Froth filtrate	4-2			BDL	ND
Froth solids	4-2	58.7	BDL		26.0
Slurry filtrate	4-2			BDL	ND
-400 mesh solids	4-2		ND		ND
+400 mesh solids	4-2	20.0	BDL		19.0
Attrition scrub filtrate	4-2			BDL	ND
-400 mesh soilds	4-2	15.3	ND		ND
+400 mesh product	4-2		BDL		15.4
HNO_3/HF filtrate	4-3		(4.32)[5]	1080	ND
HNO_3/HF product	4-3	93.1	[2.13]		26.1
HBr filtrate	4-3		(4.80)	1200	ND
HBr product	4-3	83.8	[3.77]		15.8
Split A	4-4	117.6	4.14		ND
Split B	4-4	115.5	4.27		ND
Split C	4-4 / 4-6	114.4	3.35		ND
Split D	4-4 / 4-6	119.4	3.87		ND
Split E	4-4	114.4	2.65		ND
Split F	4-4	119.3	3.99		ND
Split G	4-4	115.2	2.96		ND
Split H	4-4 / 4-6	113.5	3.70		ND
HF blend	4-4		[4.07]		ND
Blend split E-1	4-4 / 4-6	118.8	4.58		ND
Blend split G-1	4-4	120.3	3.96		ND
HNO_3/HF feed	4-5		[4.07]		[23.2]
HNO_3/HF stage 1 filtrate	4-5		(4.20)	993	(16.57)

Am-241 and Pu Analyses					
		Am-241			
Sample	Figure	Sample Wt (g)(1)	pCi/g	pCi/Sample	Pu
HNO_3/HF stage 2 filtrate	4-5			BDL	(2.61)
HNO_3/HF -400 mesh solids	4-5		ND		25.1
HNO_3/HF product	4-5	162.7	BDL		1.65
HBr feed	4-5		[4.11]		[24.0]
HBr stage 1 filtrate	4-5		(1.65)	389	(18.46)
HBr stage 2 filtrate	4-5			BDL	(1.27)
HBr -400 mesh solids	4-5		ND		10.46
HBr product	4-5	139.8	BDL		3.81
Flotation feed	4-6		[3.88]		[20.02]
Froth filtrate	4-6			BDL	(0.06)
Froth solids (-400 mesh)	4-6		ND		44.65
Residue filtrate	4-6			BDL	(0.10)
Flotation residue (-400 mesh)	4-6		ND		54.23
Flotation residue (+400 mesh)	4-6		ND		14.91
Attrition scrub filtrate	4-6			BDL	(0.11)
Attrition scrub -400 mesh solids	4-6		ND		70.48
Attrition scrub +400 mesh solids	4-6	129.2	2.10		9.88
Split A	4-7	146.3	3.46		[22.1](6)
Split B	4-7	150.47	3.17		[22.1](6)
Split C	4-7	146.21	3.86		[22.1](6)
Split D	4-7	150.95	4.03		[22.1](6)
Split E	4-7	145.62	3.26		[22.1](6)
Split F	4-7	144.41	3.52		[22.1](6)
48% HBR stir wash feed	4-7		[6.58]		[41.7]
48% HBr stage 1 filtrate	4-7		(2.43)	375	(28.03)
48% HBr stage 2 filtrate	4-7			BDL	(2.53)
48% HBr product	4-7	57.0	1.06		10.4
HBr/HF stir wash feed	4-7		[6.80]		[41.7]
Dilute HBr stir wash feed	4-7		[6.72]		[41.7]
Dilute HBr stage 1 filtrate	4-7		(3.49)	540	(16.21)
Dilute HBr stage 2 filtrate	4-7			BDL	(10.08)
Dilute HBr product	4-7	80.9	0.847		16.1

(1) Weight of sample analyzed
(2) Not determined
(3) Below detection limit
(4) Calculated value [xxx]
(5) Solution content normalized to per gram of feed to process operation (xxx)
(6) Plutonium analyses on a dry weight basis were 41.7 pCi/g