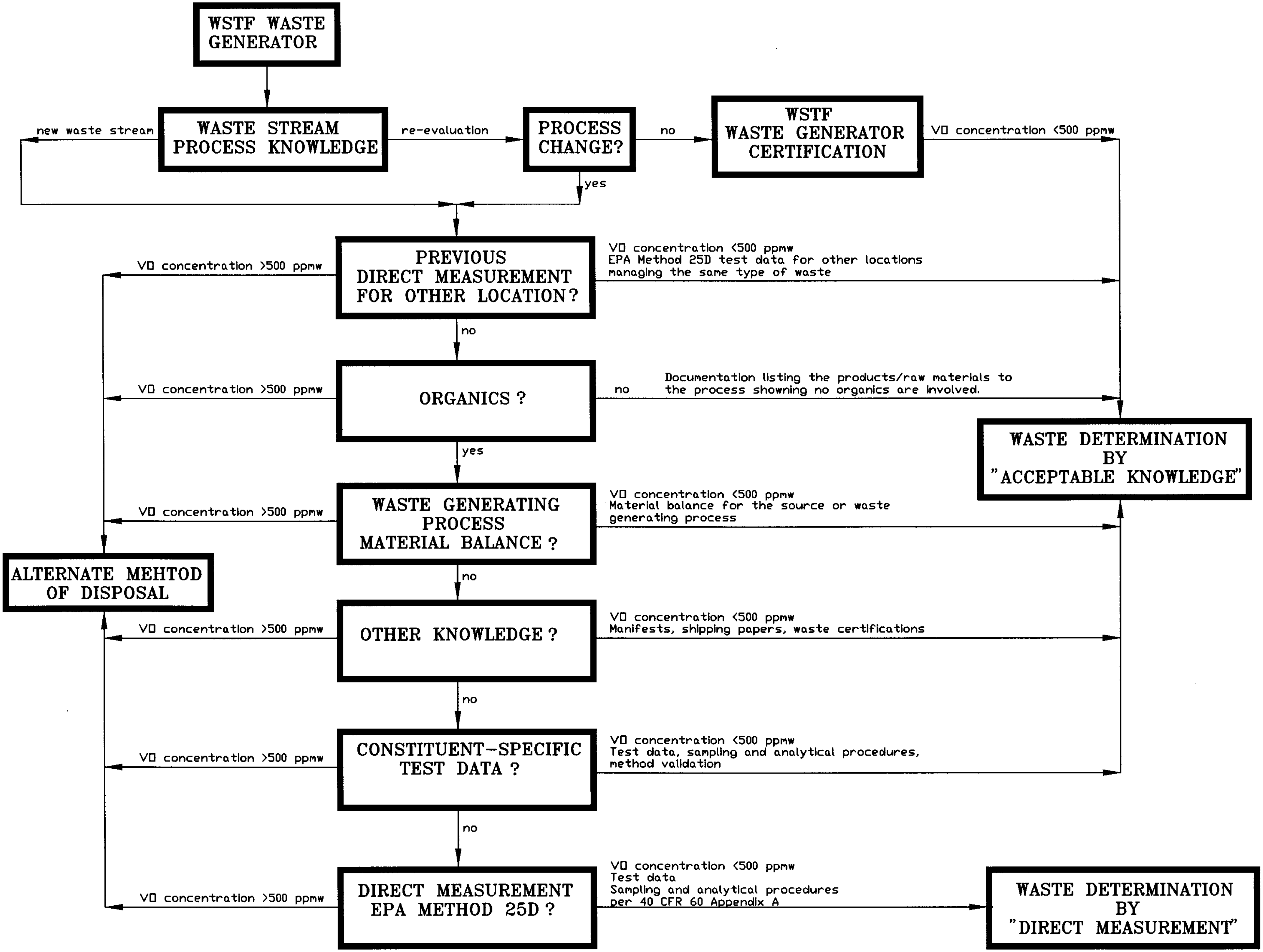
Attachment 1

Waste Determination Flow Chart

WASTE DETERMINATION FLOW CHART



Attachment 2

EPA Method 25D

Sampling Procedure

and

Sampling Apparatus

NASA JSC WSTF

TEST PREPARATION SHEET

(1) TPS NUMBER - MOD. 2-HWM 950343 R	(2) PAGE 1 of 8	
(3) A - CONFIGURATION CHANGE		☐
(3) B - NO CONFIGURATION CHANGE		☒

(4) SYSTEM/TEST STAND
200 Area Hazardous Waste Evaporation Tank System

(5) QA COORDINATION
9 VO
Apr 21 '95

(6) MIPS					
✓	NONE		STEPS		ALL

(7) TITLE
EPA Method 25D Sampling the 200 Area Hazardous Waste Drain Line Waste Streams

(8) DRAWING(S), DOCUMENT(S), OCP(S), PART NO(S) AND DCN(S)
40 CFR 265 Subpart CC, 40 CFR 60 appendix A (EPA METHOD 25D), US EPA Document <u>SW-846, Test Methods for Evaluating Solid Waste, Physical and Chemical Methods</u>

(9) FILE NAME

(10) TASK ORDER NUMBER
038-JAA-00

(11) NEED DATE
4/21/95

(12) SPECIAL SAFETY REQUIREMENTS ☒ YES ☐ NO

(13) PREPARED BY EXT.
Tanaka

(14) DATE CLOSED BY QA
CONTRACTOR Tanaka 1/21/98
QA

(15) CONSTRAINTS
None

(16) SUMMARY/STATUS/INSTRUCTIONS:

SCOPE:

The following repetitive TPS authorizes sampling and analyses per EPA Method 25D of waste streams that are discharged to the 200 Area Hazardous Waste Drain Lines.

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
Tanaka 4/20/95	4/21/95
4/20/95	

WSTF-392 (1/91)

NASA JSC WSTF

TEST PREPARATION SHEET

(17) TPS NUMBER - MOD. 2HWM-950343 R		(18) PAGE 2 of 8
(19) QA COORDINATION	(20) MIPS ✓ NONE	STEPS ALL

(21) ITEM NUMBER	(22) DESCRIPTION	(23) PERFORMED BY	(24) INSPECTED BY

EQUIPMENT:

All sampling techniques and equipment used to sample the 200 Area Hazardous Waste Drain Line waste streams shall conform to the instructions provided in the US EPA Document <u>SW-846, Test Methods for Evaluating Solid Waste, Physical and Chemical Methods</u> and the requirements of EPA Method 25D in 40 CFR 60 appendix A.

EPA Method 25D sampling apparatus that includes:
- 0.25" ID teflon sampling tube; and
- 0.25" ID 304 SS cooling coil with a thermocouple at the coil outlet.

Prepared EPA Method 25D sample vials. Store vials on ice until one (1) hour before the sampling event.

Ice chest with ice.

MANPOWER REQUIREMENTS:

2 - Waste Samplers certified per WSTF Operations Certification Plan, Appendix VI, Category 2.5 - <u>Waste Sampler</u>. (8 to 16 hours/each)

NOTE:

ENVIRONMENTAL DEPARTMENT PERSONNEL PERFORMING THE SAMPLING PROCEDURES OF THIS TPS WILL CHARGE TASK ORDER 038-JAA00.

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
4/20/95	4/20/95
4/20/95	

NASA JSC WSTF

TEST PREPARATION SHEET

(17) TPS NUMBER - MOD.: 2HWM 950343 R

(18) PAGE 3 of 8

(19) QA COORDINATION: QA 6 21

(20) MIPS: [✓ NONE] STEPS ALL

(21) ITEM NUMBER	(22) DESCRIPTION	(23) PERFORMED BY	(24) INSPECTED BY

ENVIRONMENTAL DEPARTMENT WASTE SAMPLERS

SAFETY NOTES:

THE FOLLOWING MINIMUM SAFETY EQUIPMENT IS REQUIRED DURING SAMPLE COLLECTION.

A. TYVEX OR SPLASH SUITS.
B. RUBBER GLOVES.
C. FACE SHIELD OR GOGGLES.
D. INTERSCAN WHEN HYDRAZINE WASTES ARE SAMPLED.
E. BREATHING AIR IS REQUIRED ANY TIME HYDRAZINE LEVELS AS INDICATED BY INTERSCAN EQUALS OR EXCEEDS 0.1 ppm OR AS DIRECTED BY THE CONTRACTOR ENVIRONMENTAL DEPARTMENT.
F. "TWO MAN OPERATION" IS REQUIRED.
G. ORGANIC RESPIRATOR AS DIRECTED BY THE CONTRACTOR ENVIRONMENTAL DEPARTMENT.

1. Contact the responsible section supervisor or designated alternate. Section supervisor or designated alternated will escort Environmental Department samplers during the sampling operation. For each sampling event the section supervisor shall provide the following minimum information:

   A. The waste quantity represented by the samples; and
   B. Details of the operating conditions for the source or process generating the waste represented by the samples.

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
4/20/95	4/21/95
4/20/95	

WSTF-392 (7/91)

NASA JSC WSTF

TEST PREPARATION SHEET

(17) TPS NUMBER - MOD. 2HWM-950343 R		(18) PAGE 4 of 8
(19) QA COORDINATION QA 6	(20) MIPS ✓ NONE	STEPS ☐ ALL ☐

(21) ITEM NUMBER	(22) DESCRIPTION	(23) PERFORMED BY	(24) INSPECTED BY
2.	One (1) hour before the sampling event, remove selected sample vials from ice and allow the vials to reach room temperature.		
3.	Set up work station and sampling apparatus at the "point of waste origination" as directed by Environmental Department personnel. The sample shall be taken at a point which is most representative of the unexposed waste.		
4.	Purge the sampling apparatus with a minimum of four (4) apparatus volumes of waste. Collect the waste in a container and dispose as directed by Environmental Department personnel. Check the temperature as indicated by the thermocouple thermometer. During the sampling event the temperature should be kept below 10°C. NOTE: DURING THE SAMPLING EVENT ENSURE THE FOLLOWING: A. THE SAMPLE TUBE IS KEPT BELOW THE SURFACE OF THE POLYETHYLENE GYLCOL (PEG). B. THE TEMPERATURE AT THE COOLING COIL OUTLET IS LESS THAN 10°C. C. PEG IS NOT LOST OR REMOVED FROM THE SAMPLE VIAL. D. HEAD SPACE IS MINIMIZED. E. SAMPLE VIALS ARE CAPPED IMMEDIATELY AFTER SAMPLING AND STORED ON ICE. F. DO NOT ADD ANY EXTRA LABEL, SEALS OR TAPE TO THE SAMPLE VIALS.		

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
4/20/95	4/21/95
4/20/95	

NASA JSC WSTF

TEST PREPARATION SHEET

(17) TPS NUMBER - MOD. 2HWM-95034B R		(18) PAGE 5 of 8
(19) QA COORDINATION QA 6 APR 21 '95	(20) MIPS ✓ NONE ☐ STEPS ☐ ALL	

(21) ITEM NUMBER	(22) DESCRIPTION	(23) PERFORMED BY	(24) INSPECTED BY
5.	After purging, stop flow and direct the sample tube to a prepared sample vial. Keep the sample tube below the surface of the PEG during sampling to minimize contact with the atmosphere. Sample at a flowrate such that the temperature as indicated by the thermocouple thermometer is less than 10°C. Add just enough sample media to completely fill the sample vial. Try to minimize headspace; however, do not remove or lose any PEG from the sample vial. Cap the vial within five (5) seconds of filling the vial and store on ice. Do not add any extra labels, seals or tape to the sample vials. Open field blanks as directed by Environmental Department personnel.		
6.	Repeat step 5 as required. Take eight (8) sample vials of sample media, four (4) for the samples and four (4) for the duplicate samples (under normal circumstances duplicate samples will not be analyzed).		
7.	Annotate the evaporation tank logbook with a brief description of the sampling operation which includes the type of sampling, WIWPS #, point of waste origination, TPS #, date, and personnel present during the sampling event. ENSURE THE FOLLOWING ARE NOTED IN THE LOGBOOK: A. Sample parameter; B. Sample preservation; C. Sample I.D. # (commercial lab # and corresponding WSTF #); D. Sample location (WIWPS # and point of waste origination);		

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
4/20/95	4/21/95
4/20/95	

NASA JSC WSTF **TEST PREPARATION SHEET**	(17) TPS NUMBER - MOD. 2HWM-950343 R	(18) PAGE 6 of 8
	(19) QA COORDINATION QA 6 APR 21 '95	(20) MIPS ✓ NONE STEPS ALL

(21) ITEM NUMBER	(22) DESCRIPTION	(23) PERFORMED BY	(24) INSPECTED BY
	E. Lab performing analyses; and F. Type of sample (sample or duplicate)		
8.	Repeat steps 1 through 7 for other waste streams or locations as directed by the Environmental Department.		
	NOTE: FOR ALL SAMPLES, FOLLOW "CHAIN-OF-CUSTODY" PROCEDURES. ENSURE THE "CHAIN OF CUSTODY" IDENTIFIES THE COMMERCIAL LAB ID NUMBER, CORRESPONDING WSTF ID NUMBER AND LOCATION.		
9.	Deliver the samples and the corresponding "Chain of Custody" documentation to the warehouse for shipment.		

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
4/20/95	4/21/95
4/20/95	

WSTF-392 (7/91)

NASA JSC WSTF

TEST PREPARATION SHEET

(17) TPS NUMBER - MOD. 2HWM-950343 R		(18) PAGE 7 of 8
(19) QA COORDINATION 6	(20) MIPS ✓ NONE	STEPS ALL

(21) ITEM NUMBER	(22) DESCRIPTION	(23) PERFORMED BY	(24) INSPECTED BY

EPA METHOD 25D SIGNOFF SHEET

	WIWPS#'s/ DESCRIPTION	E. D. AUTHORIZATION	
	(20-01-01) Rinse Water (1-A → 4B)	Tanaka	
	Clean Room Cleaning Tanks		
Oakite 33	(20-01-11) Oakite ~~[illegible]~~ 33 Acid (5A — 8B)	Tanaka	
	Clean Room Cleaning Tanks		
oakite 126	(20-01-10) ~~[illegible]~~ (9A - 12B) Oakite 126	Tanaka	
	Clean Room Cleaning Tanks		
	(20-01-18) Simple Green	Tanaka	
	Clean Room/Lab Con (13A — 16B)		
	(20-01-38) Citric Acid	Tanaka	
	Clean Room/Lab Con (17A — 20B)		
	(20-01-13) Oakite Rustripper	Tanaka	
	Clean Room/Lab Con (21A — 24B)		
	Photo Fixer (25A — 28B)	Tanaka	
	(20-03-15) (20-03-31)		
	Water Soluble Oil	Tanaka	
	(29A — 32B		
	ChemLab Aqueous Waste 20-04-01	Tanaka	
	20-04-01		
	MetLab Photo Flow	Tanaka	
	(37A — 40B)		
	Silkscreen Rinse Water	Tanaka	
	(41A — 44B)		
	1% Simple Green	Tanaka	9/12/95
	Oakite 33	Tanaka	9/12/95
	ChemLab Fuel Scrubber	Tanaka	9/12/95
	10% UDMH	Tanaka	9/12/95
	10% MMH	Tanaka	9/12/95
	10% HZ	Tanaka	9/12/95
	Clean Room Oakite 31	Tanaka	7/16/96
	500 Area F1F Treated Fuel	Tanaka	7/16/96
20-01-45	Clean Room Brulin 815 GD	Tanaka	8/28/96

Close this TPS Effective January 21, 1998

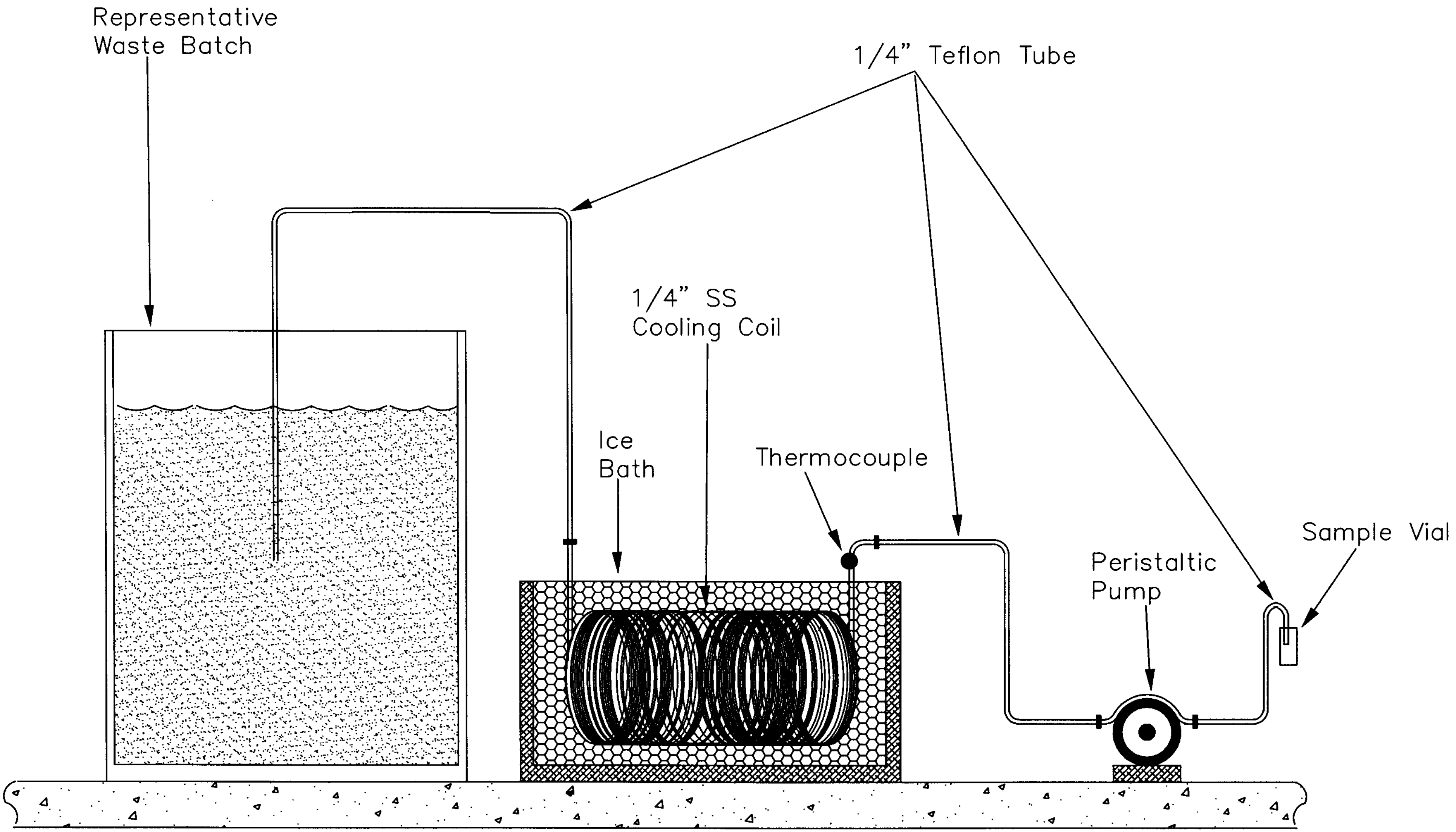
Tanaka 1/21/98
Cont- Sig

NASA Sig

CONTRACTOR AUTHORIZED SIGNATURES	NASA AUTHORIZED SIGNATURES
Tanaka 4/20/95	4/21/95
4/20/95	

WSTF-392 (7/91)

EPA Method 25D Sampling Apparatus



Attachment 3

NMED Correspondence

State of New Mexico
ENVIRONMENT DEPARTMENT
Hazardous & Radioactive Materials Bureau
2044 Galisteo
P.O. Box 26110
Santa Fe, New Mexico 87502
(505) 827-1557
Fax (505) 827-1544

1912

GARY E. JOHNSON
GOVERNOR

MARK E. WEIDLER
SECRETARY

EDGAR T. THORNTON, III
DEPUTY SECRETARY

VIA TELEFAX

April 23, 1996

Mr. D. Tanaka
Allied Signal Team
White Sands Test Facility
FAX No. (505) 527-8925

Dear Mr. Tanaka:

RE: Waste Determination Procedures pursuant to 40 CFR §265.1084

This telefax transmittal is in response to your request for NMED's interpretation of §265.1084(a)(2)(ii)(A). NMED interprets this section to mean that once an initial waste determination of the average VO concentration for a batch is made by laboratory analysis, an update of the information by use of knowledge of process is acceptable assuming the process generating the waste does not change. Therefore, if an initial waste determination was performed by using the approved EPA test method, a simple statement on record that the process has not changed and that therefore knowledge of process is used to make the determination will normally be acceptable in subsequent years. Please be reminded that the generator or manager of the waste is responsible for being correct when using knowledge of process.

Please be aware that the New Mexico Hazardous Waste Management Regulations (20 NMAC 4.1) will not adopt the Subpart CC regulations until at least October 1, 1996. Therefore, these regulations will not be enforceable by NMED until they are adopted into 20 NMAC 4.1 If you have any further questions or need additional clarification, please contact me at (505) 827-1558.

Sincerely,

Coby Muckelroy

Coby Muckelroy
RCRA Inspection/Enforcement Program Manager
Hazardous and Radioactive Materials Bureau

Appendix 6-E

NASA/WSTF ETU
Fuel Waste Water Management Plan

200 AREA EVAPORATION TANK UNIT (ETU)

FUEL WASTE WATER

MANAGEMENT PLAN

1.0 Introduction

Fuel contaminated waste water (P068, U098 and U133 listed hazardous wastes) is restricted from discharge to the ETU. NASA has developed this 200 Area ETU Fuel Waste Water Management Plan to segregate and define the P068, U098 and U133 listed hazardous waste waters from non-hazardous waste water which can be discharged to the ETU.

2.0 Fuel Decontamination and Disassembly Waste Water

WSTF decontamination processes involve the management of P068, U098 and U133 listed hazardous wastes. Per the “Mixture Rule”, 40 CFR 261.3 (a)(2)(iv) and (b)(2), a solid waste mixed with a listed hazardous waste (hazardous wastes listed in 40 CFR 261 subpart D) is a hazardous waste. Per the “Mixture Rule”, these waste codes could potentially be carried through other subsequent processes that are discharged to the ETU. To demonstrate compliance, this Plan incorporates the following approach to segregate and define the P068, U098 and U133 listed hazardous wastes associated with WSTF’s propellant decontamination and disassembly process.

2.1 Empty Container Rule

NASA manages the fuel decontamination and disassembly processes according to the “Empty Container Rule”, 40 CFR 261.7(a)(1) and 40 CFR 261.7(b)(3)
40 CFR 261.7(a)(1):

“Any hazardous waste remaining in either (i) an empty container or (ii) an inner liner removed from an empty container, as defined in paragraph (b) of this section, is not subject to regulation under parts 261 through 265, 267, 268, 270 or 124 of this chapter or to the notification requirements of section 3010 of RCRA.”

40 CFR 261.7(b)(3):

> *“A container or an inner liner removed from a container that has held an acute hazardous waste listed in §§261.31, 261.32, or 261.33(e) is empty if:*
>
> > *(i) The container or inner liner has been triple rinsed using a solvent capable of removing the commercial chemical product or manufacturing chemical intermediate;*
> > *(ii) The container or inner liner has been cleaned by another method that has been shown in the scientific literature, or by tests conducted by the generator, to achieve equivalent removal; or*
> > *(iii) In the case of a container, the inner liner that prevented contact of the commercial chemical product or manufacturing chemical intermediate with the container has been removed.”*

40 CFR 260.10 defines a container as follows:

> *"Container means any portable device, in which a material is stored, transported, treated, disposed of, or* ***otherwise handled*** *(emphasis added)".*

NASA interprets the definition of a container per 40 CFR 260.10 to include containers and container-like equipment involved in product propellant handling. This includes, but not limited to, sample (Hoke) bottles, laboratory equipment, valves, piping, fittings, and other aerospace components.

2.2 WSTF Decontamination and Disassembly Process Description

A flowchart describing the decontamination and disassembly process is presented in Attachment 1. A general description of the steps involved in the process follows:

1. WSTF containers and container-like equipment involved in product fuel propellant handling (including items such as sample (Hoke) bottles, laboratory equipment, valves, piping, fittings, and other aerospace components) are removed from service.

2. Within 24 hours, the containers and container-like equipment are triple rinsed or pressure rinsed at locations including but not limited to the 300, 400, and 800 Area fuel decon stations, the Fuel Treatment Unit (FTU) or 200 Area fuel hoods. Through this process these items are rendered RCRA empty per 40 CFR 261.7(b)(3)(i) and (b)(3)(ii).

3. Under the "Empty Container Rule" the rinsate from triple or pressure rinsing (rinsate from the first three rinses) is listed hazardous waste (P068, U098 and U133 waste codes). After decontamination (triple or pressure rinsing is complete) any waste residues and the container and container-like equipment are not regulated under RCRA.

4. The non-regulated containers and container-like equipment/components are transported to the 200 Area Components Services Section for disassembly, rinsing, and aerospace cleaning. Under the "Empty Container Rule", the rinsate from the fourth rinse and liners/softgoods materials removed during disassembly are non-hazardous. Although non-hazardous, WSTF may manage these waste streams as hazardous waste. The waste may be declared to be hazardous (P068, U098, U133 waste coded) to support off-site disposal.

5. All subsequent cleaning processes or other operations are no longer associated with, defined as, or declared P068, U098 and U133 listed hazardous wastes.

2.3 Equivalency of the Method

Under 40 CFR 261.7 (b)(3), a container is a RCRA "Empty Container" if:

- the container has been triple rinsed, or
- the container has been cleaned by another method that has been shown by

scientific literature, or by tests conducted by the generator, to achieve equivalent removal.

Triple rinsing and pressure rinsing are approved by Federal agencies in the U.S. and Canada for the rinsing or decontamination of pesticide containers prior to disposal. NASA has performed tests to demonstrate that the WSTF fuel decontamination process achieves removal equivalent to triple rinsing. Published data (Iowa State University, PAT 1442, revised June 2000) indicates that the 3rd rinse contains less than 2 ppm of the active ingredient (one ounce the 3rd rinse from a pesticide container contains 0.00005 grams). The non-regulated 4th rinse can be assumed to contain at least an order of magnitude less than the 3rd rinse.

3.0 Non-Hazardous Contact Contaminated Waste Water

Non-hazardous contact contaminated waste water (waste water that is not a hazardous waste as defined by 40 CFR 261.3) includes:

- Accumulated precipitation and precipitation run-off from the ETU and FTU, and
- Non-hazardous waste water, generated from test or test support activities, that has been determined not to be a listed waste per 40 CFR Part 261 Subpart D.

These items are discussed below in further depth.

3.1 Accumulated Precipitation and Precipitation Run-Off

Demonstration of compliance for accumulated precipitation and precipitation run-off from the ETU and FTU will be performed by “acceptable knowledge”. Pertinent regulatory citations with respect to the management of these waste streams follow.
40 CFR 261.3(c)(2)(i) states:

> *“Except as otherwise provided in paragraph (c)(2)(ii), (g) or (h) of this section, any solid waste generated from the treatment, storage, or disposal of a hazardous waste, including any sludge, spill residue, ash emission control dust, or leachate* ***(but not including precipitation run-off)(emphasis added)*** *is a hazardous waste. (However, materials that are reclaimed from solid wastes and that are used beneficially are not solid wastes and hence are not hazardous wastes under this provision unless the reclaimed material is burned for energy recovery or used in a manner constituting disposal.)”*

40 CFR 264.193(c)(4) states:

> *“Sloped or otherwise designed or operated to drain and remove liquids resulting from leaks, spills, or precipitation. Spilled or leaked waste and accumulated precipitation must be removed from the secondary containment system within 24 hours, or in as timely a manner as is possible to prevent harm to human health and the environment, if the owner or operator can demonstrate to the Regional*

> *Administrator that removal of the released waste or accumulated precipitation cannot be accomplished within 24 hours.*

[Note: If the collected material is a hazardous waste under Part 261 of this chapter, it is subject to management as a hazardous waste in accordance with all applicable requirements of parts 262 through 265 of this chapter. If the collected material is discharged through a point source to waters of the United States, it is subject to the requirements of sections 301, 304, and 402 of the Clean Water Act, as amended. If discharged to a Publicly Owned Treatment Works (POTW), it is sub]ect to the requirements of section 307 of the Clean Water Act, as amended. If the collected material is released to the environment, it may be subject to the reporting requirements of 40 CFR part 302.]"

Per 40 CFR 261.3(c)(2)(i) and the note per 40 CFR 264.193(c)(4), accumulated precipitation and precipitation run-off is not presumed to be hazardous waste. Accumulated precipitation and precipitation run-off from the ETU and FTU would be hazardous waste only when it is mixed with a listed hazardous waste.

The ETU and FTU are managed in accordance with 40 CFR 264 Subpart J and inspected daily in accordance with 40 CFR 264.193(f)(1) through (f)(4) and 264.195(b). All spills or leaks are cleaned up within 24 hours in accordance with 40 CFR 264.193(c)(4). From "Acceptable Knowledge", it is known that the accumulated precipitation and precipitation run-off from the ETU and FTU are not mixed with listed hazardous wastes (P068, U098, and U133). Therefore, accumulated precipitation and precipitation run-off from the ETU and FTU will be discharged directly to the ETU.

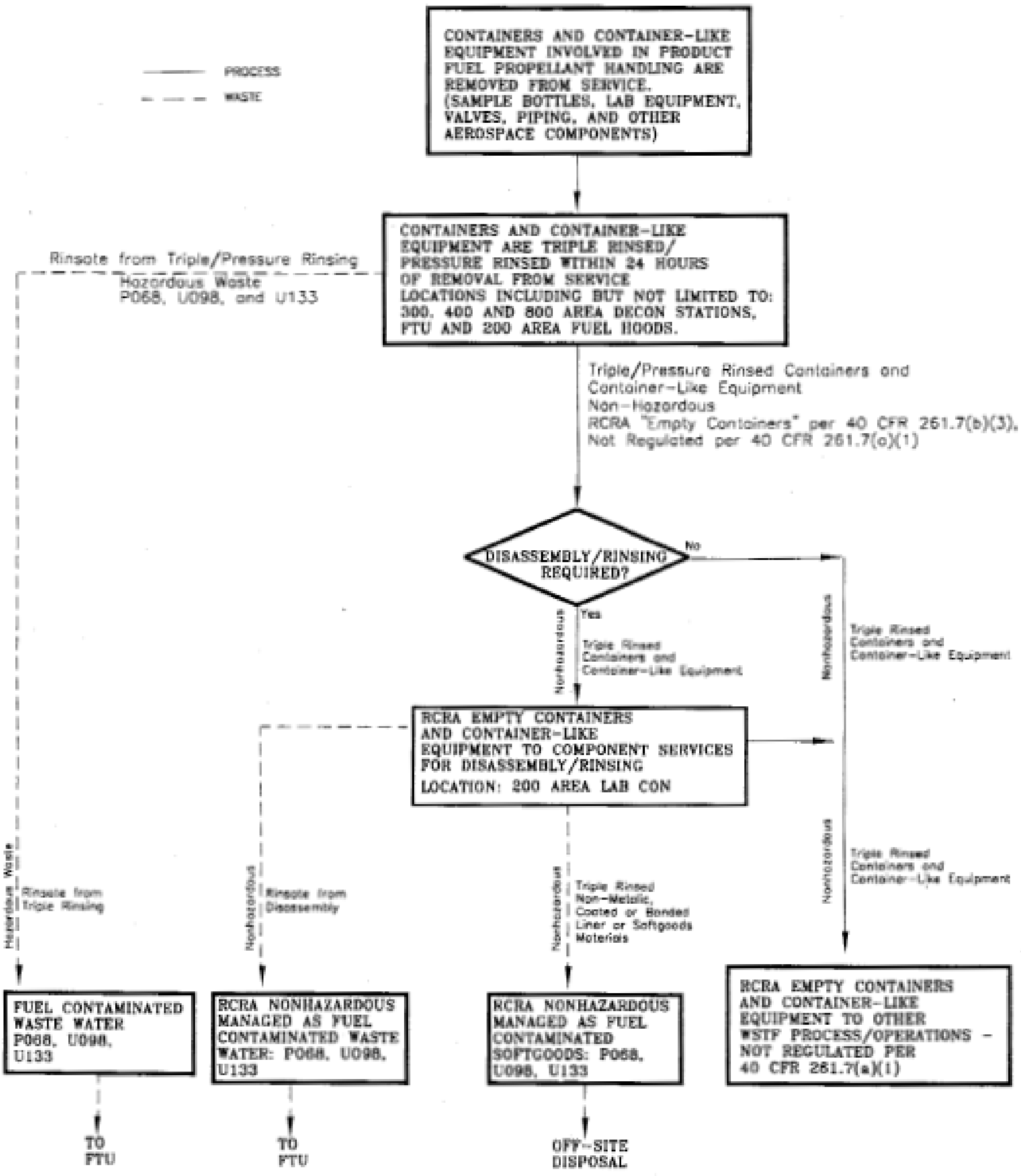
3.2 Non-Hazardous Contact Contaminated Waste Water

Non-hazardous waste water generated from test or test support activities that has the potential to contain trace levels of hydrazine constituents, and are not a listed waste per 40 CFR 261 Subpart D will be discharged to the ETU

ATTACHMENT 1

WSTF FUEL DECONTAMINATION AND DISASSEMBLY PROCESS FLOW SCHEMATIC

WSTF FUEL DECONTAMINATION AND DISASSEMBLY PROCESS FLOW SCHEMATIC



Appendix 6-F

NASA/WSTF ETU
Corrosive Waste Water Management Plan

1.0 Introduction

The ETU is operated as an exempt unit in accordance with 40 CFR 264.1082(c)(1) (RCRA Subpart CC regulations) and under the provisions of 40 CFR 268.50 (e) (LDR regulations). All waste discharged to the ETU is limited to waste with an average volatile organic (VO) concentration of less than 500 ppmw as determined at the point of waste origination and waste that meets LDR treatment standards. The waste discharged to the ETU is limited to investigative derived waste (IDW), oxidizer contaminated waste water and corrosive wastes that meets LDR treatment standards. RCRA metals (waste codes D004, D005, D006, D007, D008, D009, D010, and D011), and inorganic UHC's above LDR treatment standards are restricted from discharge to the ETU. To assure compliance the LDR regulations, all corrosive waste discharged to the ETU will be below LDR treatment standards or treated below LDR treatment standards (in exempt units regulated under 40 CFR 262.34). Under 40 CFR 268.7(a)(5), a WAP is required for treatment in exempt units to meet LDR treatment standards. NASA has developed the Corrosive Waste Management Plan to provide details with respect to the management of corrosive waste water discharged to the ETU and a WAP for the treatment of corrosive waste with potential inorganic UHC's. The NASA WSTF Corrosive Waste Management Plan follows.

2.0 ETU Waste

The corrosive waste and non-hazardous waste water discharged to the ETU includes the following waste categories:

- Acidic Cleaning Solution
- Ammonia Waste Water
- Caustic Cleaning Solution
- Citric Acid Cleaning Solution
- Pickling/Etching Solution
- Nitric Hydrofluoric Acid
- Clean Room Rinse Water (non-hazardous)
- Contact Contaminated Waste Water (non-hazardous)
- Detergent-Type Waste Water (non-hazardous)
- Met Lab Waste Water
- Chem Lab Waste Water

A general description of the waste categories discharged to the ETU is presented in Table 6.2 of the WAP. Typical waste generation scenarios are presented in Appendix 6-A of the WAP.

3.0 Corrosive Waste - ETU LDR Compliance

All corrosive waste discharged to the ETU will be below LDR treatment standards or treated below LDR treatment standards (in containers regulated under 40 CFR 262.34). The regulations allow waste generators to use testing, knowledge of the waste or both to demonstrate compliance with LDR treatment standards. Details of what constitutes "acceptable knowledge" are provided in section 6.3.2 of the WAP.

Whenever possible, "acceptable knowledge" will be used to document compliance with LDR treatment standards. A flow chart of the overall approach to demonstrate compliance with LDR treatment standards and the management of corrosive waste is presented in Attachment 1. The steps in the process follow.

1) Whenever possible "acceptable knowledge" will be used to demonstrate compliance. Product information/MSDS's and the waste generating process are reviewed to identify organic UHC's. Organic UHC's are defined as organic constituents identified per 40 CFR 268.48 that have a reasonable potential to be present. Corrosive waste with potential to contain organic UHC's above LDR treatment standards will shipped off-site for disposal.

2) Product information/MSDS's and the waste generating process are reviewed to identify potential inorganic UHC's. Inorganic UHC's are defined as inorganic constituents identified per 40 CFR 268.48 that have a reasonable potential to be present. If inorganic UHC's are not involved in the waste generating process, the corrosive waste will be discharged to the ETU.

3) In cases where "acceptable knowledge" can not be used to document compliance with LDR treatment standards, sampling and analysis will be performed with respect to the potential inorganic UHC's present in the corrosive waste. If inorganic UHC's (as demonstrated by sampling and analysis) are below LDR treatment standards, the waste will be discharged to the ETU.

4) Sampling and analysis by conventional methods may be inconclusive or is not feasible. Under some circumstances, sampling and analysis by conventional methods to document compliance with the LDR treatment standards may not conclusively demonstrate compliance. Many of the commercial products used in standard operations contain a high concentration of detergents and surfactants. Due to matrix interference, conventional analytical methods such as EPA Method 1311/6010 may have laboratory reporting limits that are higher than the LDR treatment standard as specified per 40 CFR 268.48. As a result, the analytical data may not conclusively demonstrate that the inorganic UHC's are below LDR treatment standards. Under these conditions, the compliance will be documented by one of the following:

    - The potential inorganic UHC's will be assumed or declared to be present at levels above LDR treatment standards and the waste will be treated to below LDR treatment standards as described in Section 5.0.

    - Compliance documentation will be based on constituent-specific sampling and analysis (acceptable knowledge) will be performed by methods other than those referenced in SW-846, or

  - Worst case test data (acceptable knowledge) showing that under worst case conditions the potential inorganic UHC's will be below LDR treatments standards.

5) At WSTF, waste water from laboratory and laboratory-scale processes is generated in milliliter to liter quantities from a continuous series of steps in a laboratory procedure. Under strict interpretation, each slightly different step in a laboratory process has the potential to be interpreted as a unique waste generating process and a unique point of generation. Although the aggregated waste is non-hazardous, some of the individual steps in the laboratory process may have the potential to be corrosive and contain inorganic UHC's. The segregation, sampling and analysis of each slightly different step in a laboratory process that generates waste water may not be feasible. Under these conditions, the potential inorganic UHC's will be assumed or declared to be present at levels above LDR treatment standards. The waste will either be shipped off-site for disposal or treated as described in Section 5.0.

4.0 Corrosive Waste Water Treatment Process- Regulatory Approach

Under 40 CFR 261.3 (c)(2)(i) and (d)(1), characteristic hazardous wastes can be treated so that the waste no longer exhibits the characteristics of a hazardous waste. For characteristic hazardous waste to become non-hazardous, the generator must remove the characteristics of a hazardous waste and comply with the applicable LDR treatment standards per 40 CFR 268.48. In some cases, compliance with LDR treatment standards is difficult using conventional sampling and analytical methods. An optional approach is to assume or declare that the corrosive waste contains inorganic UHC's above LDR treatment standards and treat the waste to below LDR treatment standards. The regulatory approach follows.

4.1 Corrosive Treatment

Per 40 CFR 268.40(a), a prohibited waste identified in the table "Treatment Standards for Hazardous Wastes" may be land disposed only if it meets the requirements found in the table. For corrosive waste, the treatment standard is the technology standard "DEACT". Per 40 CFR 268.42, Table 1, "DEACT" is defined as,

"Deactivation to remove the hazardous characteristics of a waste due to its ignitability, corrosivity, and/or reactivity."

Corrosive waste can be treated by "NEUTR". Per 40 CFR 268.42, Table 1, "NEUTR" is defined as

"Neutralization with the following reagents ***(or waste reagents) (emphasis added)*** *or combinations of reagents: (1) Acids; (2) bases; or* ***(3) water (including wastewaters)***

(emphasis added) *resulting in a pH greater than 2 but less than 12.5 as measured in the aqueous residuals."*

A pertinent citation with respect to the EPA's interpretation (Federal Register, June 1, 1990, Vol. 55, No. 106, 22549) is as follows:

"As a result, EPA is promulgating a general treatment standard for wastes in the D002 Acid and Alkaline Subcategories that allow the use of any appropriate treatment technology, namely "Deactivation (DEACT to Remove the Characteristic of Corrosivity". ***This means that the facility may use any treatment (including neutralization achieved through mixing with other wastewaters) that results in a pH above 2 but less than 12.5 and thereby removes the characteristic of corrosivity (emphasis added)****."*

A description of the treatment process is provided in Section 5.0. Deactivation or neutralization of corrosive waste will be performed using waste reagents (waste acidic or caustics solutions) or combination of waste reagents and water (including wastewaters). Neutralization will be achieved when the resulting solutions reaches a of pH greater than 2 but less than 12.5 as measured in the aqueous residuals. At this point the corrosive waste will be rendered non-hazardous.

4.2 The LDR Dilution Prohibition

The "LDR Dilution Prohibition" per 40 CFR 268.3(a) prohibits dilution as a substitute for adequate treatment. 40 CFR 268.3(a) states as follows:

"Except as provided in paragraph (b) of this section, no generator, transporter, handler, or owner or operator of a treatment, storage, or disposal facility shall in any way dilute a restricted waste or the residual from treatment of a restricted waste ***as a substitute for adequate treatment (emphasis added)*** *to achieve compliance with subpart D of this part, to circumvent the effective date of a prohibition in subpart C of this part, to otherwise avoid a prohibition in subpart C of this part, or to circumvent a land disposal prohibition imposed by RCRA section 3004."*

The treatment process will aggregate waste to allow the use of waste reagents and wastewaters for deactivation of corrosive wastes as discussed in Section 4.1, and for centralized treatment of potential inorganic UHC's.

The EPA's interpretation with respect to the "LDR Dilution Prohibition" as it applies to centralized treatment is provided by the June 1, 1990, Federal Register, Vol. 55, No. 106, 22664- 22666. Pertinent citations are as follows:

"In interpretive preamble discussions, the Agency explained that these rules are not intended to discourage legitimate centralized treatment, and ***that aggregation of wastes preceding legitimate centralized treatment is not considered to be impermissible dilution (emphasis added)****."*

"*The existing rules on dilution and EPA's interpretive statements regarding those rules indicate that* ***the dilution prohibition has a two-fold objective: (1) To ensure that prohibited wastes are actually treated: and (2) to ensure that prohibited wastes are treated by methods that are appropriate for that type of waste. The EPA has acknowledged that prohibited wastes which are aggregated are not diluted impermissibly if they are treated legitimately in a centralized treatment system, irrespective of the dilution inherent in such a system. Thus, if "dilution" is a legitimate type of treatment, or a necessary pretreatment step in a legitimated treatment system, such dilution is permissible (emphasis added).*** *"*

"The Agency is able to provide limited additional guidance today on the issue of when treatment methods involving dilution are permissible. The issue frequently arises when prohibited wastes are aggregated for the purposes of treatment. First, ***if the wastes are all legitimately amenable to the same type of treatment, and this is a method of treatment is utilized for the aggregated wastes, the aggregation step is not impermissible dilution (emphasis added)****."*

The inorganic UHC's are amenable to the same type of treatment technology. Legitimate treatment technologies for inorganic UHC's can include technologies such as metal recovery and stabilization. Per 40 CFR 268.42 Table 1, metal recovery or "RMETL" is defined as:

"Recovery of metals or inorganics utilizing one or more of the following direct physical/removal technologies: (1) ***Ion exchange (emphasis added)****; (2)resin or solid (i.e., zeolites) adsorption; (3)reverse osmosis; (4) chelation/solventextraction; (5) freeze crystalization; (6)ultrafiltration and/or (7) simple precipitation(i.e., crystalization) Note: This does not preclude the use of other physical phase separation or concentration techniques such as decantation, filtration (including ultrafiltration), and centrifugation, when used in conjunction with the above listed recovery technologies."*

The treatment process will aggregate waste for centralized treatment of potential inorganic UHC's by ion exchange. Based on the EPA's intent and interpretation of the "LDR Dilution Prohibition", the waste aggregation associated with the treatment process is not in violation of 40 CFR 268.3(a).

4.3 "90-Day Generator Rule"

The treatment process will treat corrosive waste in containers under the provisions of the "90 Day Generator Rule" which allows on-site treatment in tanks or containers with out a permit. 40 CFR 262.34 (a) states as follows:

"Except as provided in paragraphs (d), (e), and (f) of this section, a generator may ***accumulate (emphasis added)*** *hazardous waste on-site for 90 days or less without a permit or without having interim status, provided that:*

(1) The waste is placed:

> *(i) In containers and the generator complies with the applicable requirements of subparts I, AA, BB, and CC of 40 CFR part 265; and/or*
>
> *(ii) In tanks and the generator complies with the applicable requirements of subparts J, AA, BB, and CC of 40 CFR part 265 except §§265.197(c) and 265.200;*

The EPA's interpretation of the above regulation is provided by the March 24, 1986 Federal Register, Vol. 51., No. 56, 10168. A pertinent citation follows:

"Of course, no permitting would be required if a generator chooses to treat their hazardous waste in the generator's accumulation tanks or containers in conformance with the requirements of 40 CFR 262.34 and Subparts J or I of Part 265. ***Nothing in 40 CFR 262.34 precludes a generator from treating waste when it is in an accumulation tank or container covered by that provision. Under the existing Subtitle C system, EPA has established standards for tanks and containers which apply to both the storage and treatment of hazardous waste (emphasis added)****."*

The treatment process will treat waste in containers in accordance with the requirements of 40 CFR 262.34 and 40 CFR 265 Subpart I. Per 40 CFR 260.10, a container is defined as follows,

"Container means any portable device in which a material is stored, transported, treated, disposed of, or otherwise handled".

5.0 Corrosive Waste Water Treatment Process WAP

WSTF treats corrosive waste in containers regulated under 40 CFR 262.34 to meet LDR treatment standards. Under 40 CFR 268.7(a)(5), a WAP is required for treatment in units regulated under 40 CFR 262.34 to meet LDR treatment standards. This section provides the WAP for the WSTF Corrosive Waste Water Treatment Process and serves to provide facility procedures to assure compliance with LDR requirements.

5.1 Description of Processes and Activities

Corrosive waste water is generated from the 200 Area Clean Room, Chemistry Laboratory, and Metallurgy Laboratory. The description of the associated facility processes and activities that generate corrosive waste or corrosive waste water is provided in Section 6.1.2 of the WSTF WAP.

5.2 Identification of Hazardous Wastes Managed At WSTF

The identification of hazardous wastes managed at WSTF is provided in Section 6.2.1 of the WSTF WAP. A general description of the waste categories managed at the ETU is provided in Table 6.2. The potential waste categories associated with the corrosive waste water treatment process and the approximate quantities are presented in Table 1.

Table 1 - Corrosive Waste Water Treatment Potential Waste Categories

Waste Generator	**Waste Category**	**Approximate Quantities (gallons)**	
		weekly	**annual**
200 Area Clean Room	Acidic Cleaning Solution	58	3,000
	Caustic Cleaning Solution	87	4,500
	Citric Acid Cleaning Solution	19	1,000
	Pickling/Etching Solution	1	50
	Nitric Hydrofluoric Acid	2	110
	Detergent-Type Waste Water (non-hazardous)	29	1,500
	Clean Room Rinse Water (non-hazardous)	962	50,000
	Total:	1158	60,160
Metallurgy Laboratory	Met Lab Waste Water	19	1,000
Chemistry Laboratory	Chem Lab Waste Water	192	10,000

5.3 Description of Hazardous Waste Management Units

WSTF will utilize acceptable knowledge and perform sampling and analysis as required to characterize the waste and identify potential UHC's at the point of waste generation. Prior to discharge to the ETU, all corrosive waste with potential UHC's will be treated by deactivation or neutralization so that the waste no longer exhibits the characteristics of a corrosivity and is in compliance with LDR treatment standards. Deactivation or neutralization of corrosive waste will be performed using primarily waste reagents (waste acidic or caustics solutions) or combination of waste reagents and water (including wastewaters). Neutralization will be achieved when the resulting solutions reaches a of pH greater than 2 but less than 12.5 as measured in the aqueous residuals. At this point the corrosive waste will be rendered non-hazardous. The waste will be aggregated to allow the use of waste reagents and waste waters for the deactivation of corrosive waste and for centralized treatment of potential UHC's. A general description follows.

Rinse water and corrosive waste from the Building 200, Room 111 cleaning tank assembly and Lab Con Highbay will be aggregated for centralized treatment. The lift station that serves the Building 200, Room 111 cleaning tank assembly will be plumbed to a container managed in accordance with 40 CFR 262.34. Waste from the Lab Con Highbay area will be pumped directly to the same container that serves the lift station.

The approximate waste quantities are presented in Table 1. About 1,200 gallons of waste are generated from the Clean Room per week consisting of about 80% Clean Room Rinse Water (tap water and ultra pure rinse water) and the remainder consisting of primarily acidic, caustic or detergent waste waters which are either slightly corrosive or non-hazardous.

The waste will be aggregated and neutralized in a 3,000 gallon container (polyethylene, stainless steel, or carbon steel with a chemical resistant coating or lining). When the container has reached 75% capacity (about a 2 week accumulation period), the container will be transported to a designated area for treatment of potential inorganic UHC's.

A conceptual design of the Clean Room treatment process is presented in Attachment 2. The system consists of four basic processes. A general description follows

1. pH adjustment - After accumulation, the resulting pH will be within the range 3 to 8 or adjusted to within the pH range of 3 to 8 (or as specified by the ion exchange resin manufacturer). Caustic (waste reagents or sodium hydroxide) or acidic (waste reagents or hydrochloric acid) will be pumped into the container to adjust the pH as required to support the ion exchange process.

2. Filtration for solid particulate removal.

3. Carbon filtration for removal of organics and other compounds that could attack or foul the exchange resins, and

4. Ion exchange for removal of target metals – The process consists of passing an influent stream through a columnar fixed –bed type unit filled with the exchange resin specially designed to target only the ions required to be removed from the waste stream. Cation exchange resins are resins that attract positive ions such as nickle, copper, lead, cadimuim, and trivalent chrome. These types of resins prefer metals over ions such as H^+ or Na^+ ions that initially present at the resin ion exchange sites. During the exchange process, the metal ions replace the H^+ or Na^+ ions at the exchange sites and the H^+ or Na^+ ions are released to effluent stream. Anion exchange resins utilized the same basic process, but remove anions such as sulfate, chloride, hexavalent chrome, etc. and release ions such as OH^-.

The system design assures a constant, controllable flow rate (within the range of 2 to 10 gallons per minute) that allows optimum contact with the exchange resin. Typically the ion exchange process consists of two stages (identical fixed bed units) in series. The design capacity of the system is based on the ion exchange capacity of a single stage. The ion exchange or contaminant removal process occurs in the first stage. The second stage is installed as an added safeguard to prevent the breakthrough of contaminants in the final effluent.

The effluent from the first stage will be sampled to determine when breakthrough occurs. Breakthrough occurs when the concentration of the target ion in the effluent from the first stage reaches a predetermined limit (such as LDR treatment standards). At this time the exchange media or columns in the first stage are removed for off-site disposal, the exchange media or columns in the second stage are shifted to the first stage position and virgin media or columns are installed in the second stage position.

For every batch of treated waste, the pH will be measured prior to treatment (after accumulation), after pH adjustment (if required) and prior to discharge to the ETU. Inorganic UHC's will be sampled and analyzed prior to discharge to the ETU. During the treatment process, the treated effluent will be transferred into a second container. The treated effluent will be held until receipt of analytical data demonstrates that the potential inorganic UHC's have been treated to below LDR treatment standards. At that time the waste will either retreated (if required) or discharged to the ETU. The entire process from the start of accumulation to discharge to the ETU will be completed in about 30 days, well within the 90 day regulatory time frame.

For every batch of treated waste, the pH will be measured prior to treatment (after accumulation), after pH adjustment (if required) and prior to discharge to the ETU. Inorganic UHC's will be sampled and analyzed prior to discharge to the ETU.

The treatment of waste water from the Chemistry and Metallurgy Laboratories follows a similar approach and as previously described, will utilize exchange media specially designed to remove the potential inorganic UHC's. The waste water will be accumulated in container(s) managed in accordance with 40 CFR 262.34. The containers are sized to allow the entire process from the start of accumulation to discharge to the ETU will be completed in about 30 days, well within the 90 day regulatory time frame. Waste water from the Chemistry Laboratory will be accumulated in a 500 gallon container (polyethylene, stainless steel, or carbon steel with a chemical resistant coating or lining) and waste from the Metallurgy Laboratory will be collected in a 50 gallon container (polyethylene, stainless steel, or carbon steel with a chemical resistant coating or lining). The approximate waste quantities are presented in Table 1. The treatment systems will utilize scaled-down versions of the system used to treat waste from the Clean Room (treatment rate within the range of one-half to one gallon per minute). A conceptual design of the treatment systems are presented in Attachments 3 and 4.

5.4 Criteria and Rationale for Parameter Selection and Special Parameter Selection Requirements

The criteria and rationale for parameter selection, and special parameter selection requirements are addressed per Section 6.4.1 of the site WAP.

5.5 Sampling Strategies and Equipment, Sample Preservation and Storage and Sampling QA/QC Procedures

Details with respect to sampling strategies, equipment, sample preservation, storage and sampling QA/QC are addressed per sections 6.4 and 6.5 of the site WAP.

5.6 Re-Evaluation Frequencies

Annual re-evaluation of the treatment process and associated waste streams will be performed per Section 6.3.1.4 of the site WAP.

5.7 Special Procedural Requirements

WSTF does not receive waste from off-site generators, therefore, no special procedures for the receipt of off-site waste is required**.** The general requirements for ignitable, reactive, or incompatible wastes per 40 CFR 264.17(b) and 40 CFR 270.14 (b)(9) are addressed in Section 11.1 of the site WAP..

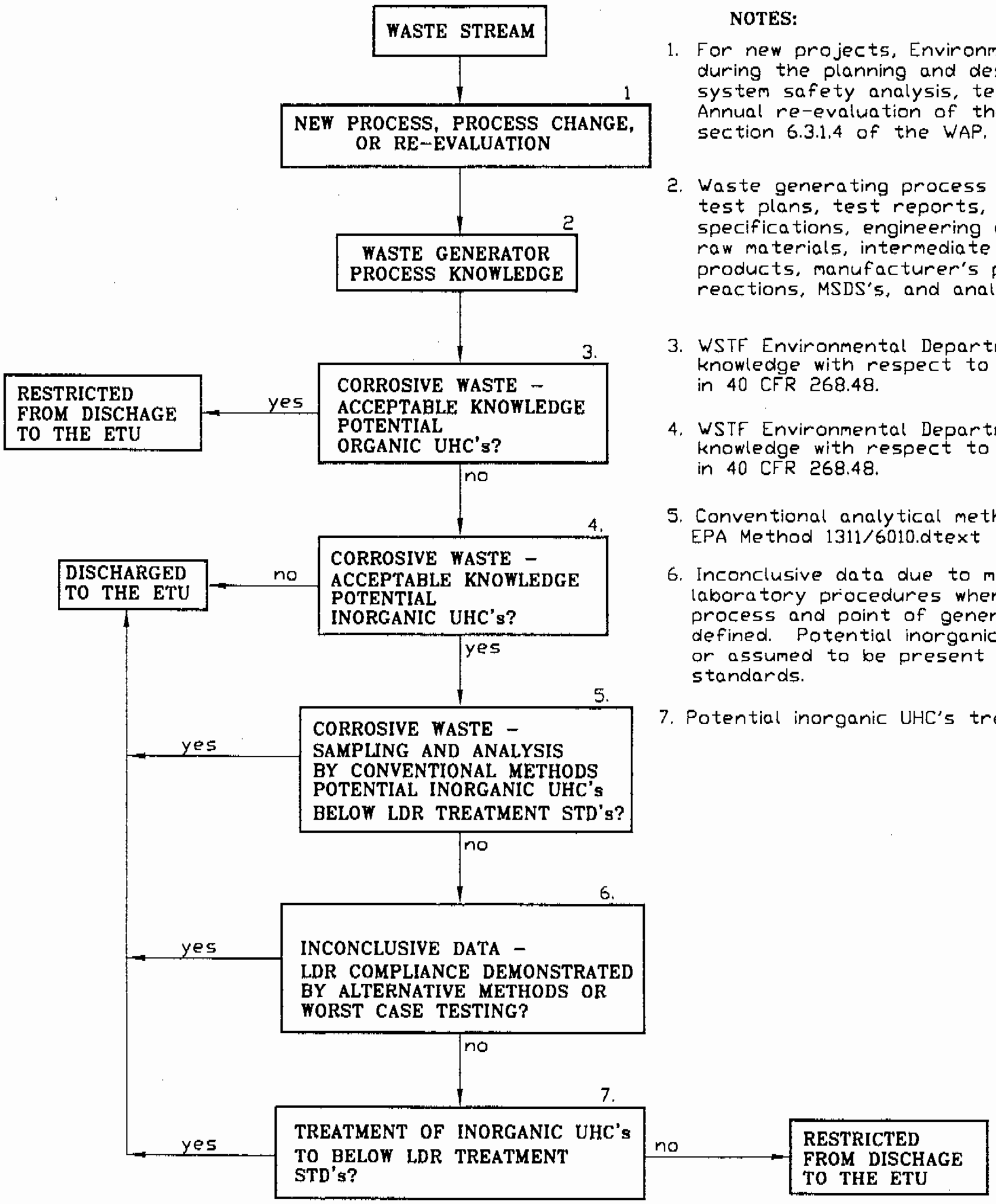
5.8 Compliance with LDR Requirements

WSTF treats corrosive waste in containers regulated under 40 CFR 262.34 to meet applicable LDR treatment standards. This WAP serves to document the procedures for complying with the LDR regulations. Section 3.0 addresses the regulatory methods of treatment, the “LDR Dilution Prohibition” and provisions for treatment with out a permit under the “90 Day Generator Rule”. Section 5.0 defines the treatment process for the treatment for corrosivity and for inorganic UHC’s. Prior to discharge to the ETU all waste will be non-corrosive and all potential inorganic UHC’s will documented by sampling and analysis to be below LDR treatment standards.

Appendix 1

ETU Corrosive Waste
LDR Compliance Flow Chart

ETU CORROSIVE WASTE LDR COMPLIANCE FLOW CHART



NOTES:

1. For new projects, Environmental Department support during the planning and design phase (design reviews, system safety analysis, test readiness reviews, etc.) Annual re-evaluation of the waste generating process per section 6.3.1.4 of the WAP.

2. Waste generating process information such as test plans, test reports, test procedures, test specifications, engineering calculations, material balances raw materials, intermediate materials, by-products, final products, manufacturer's product information, process reactions, MSDS's, and analytical data.

3. WSTF Environmental Department review of process knowledge with respect to organic constituents listed in 40 CFR 268.48.

4. WSTF Environmental Department review of process knowledge with respect to inorganic constituents listed in 40 CFR 268.48.

5. Conventional analytical methods such as EPA Method 1311/6010.dtext

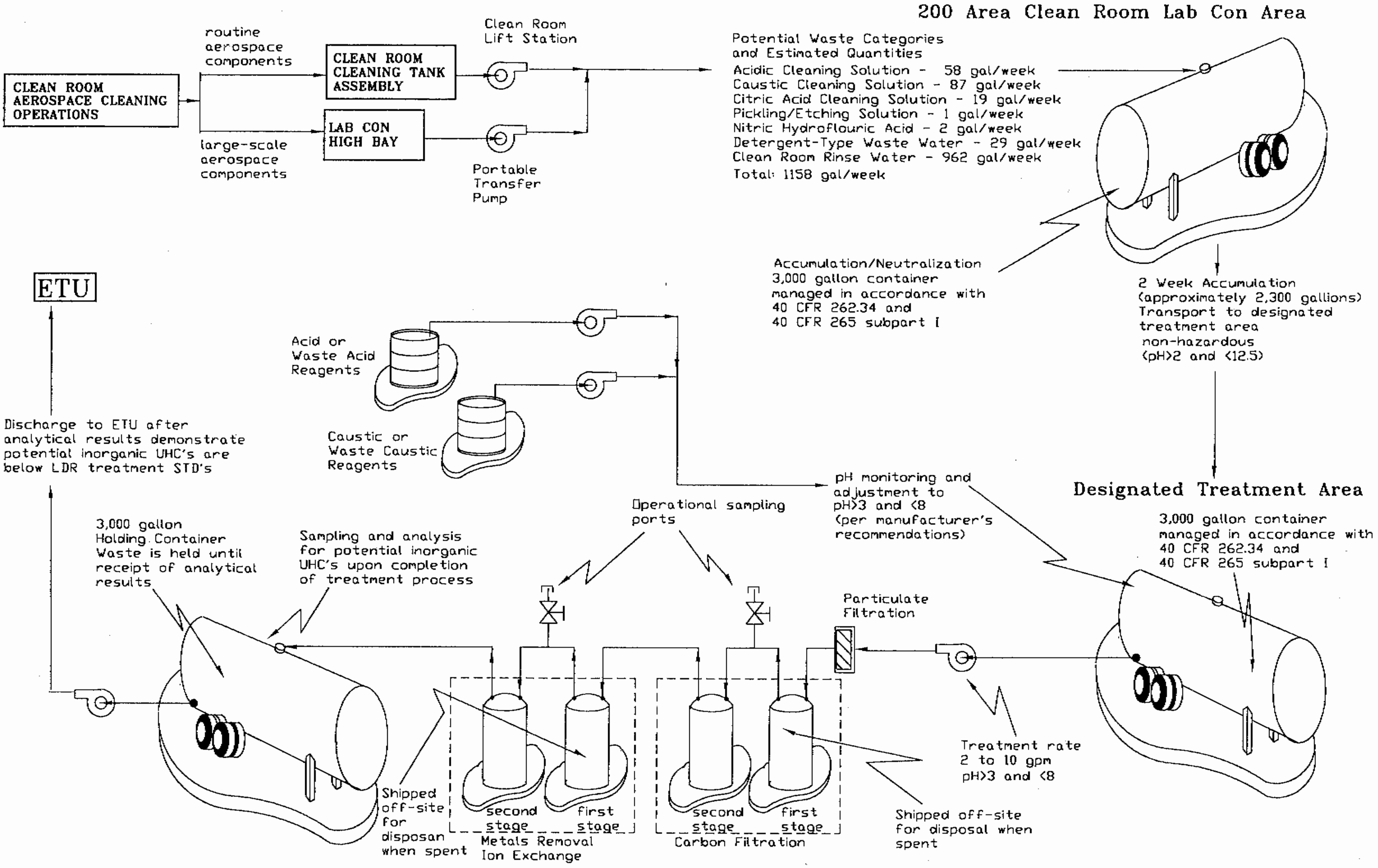
6. Inconclusive data due to matrix interference or laboratory procedures where the waste generating process and point of generation are not clearly defined. Potential inorganic UHC's are declared or assumed to be present above LDR treatment standards.

7. Potential inorganic UHC's treated by ion exchange

Appendix 2

200 Area Clean Room
Conceptual Design of Treatment Process

200 AREA CLEAN ROOM – CONCEPTUAL DESIGN OF TREATMENT PROCESS

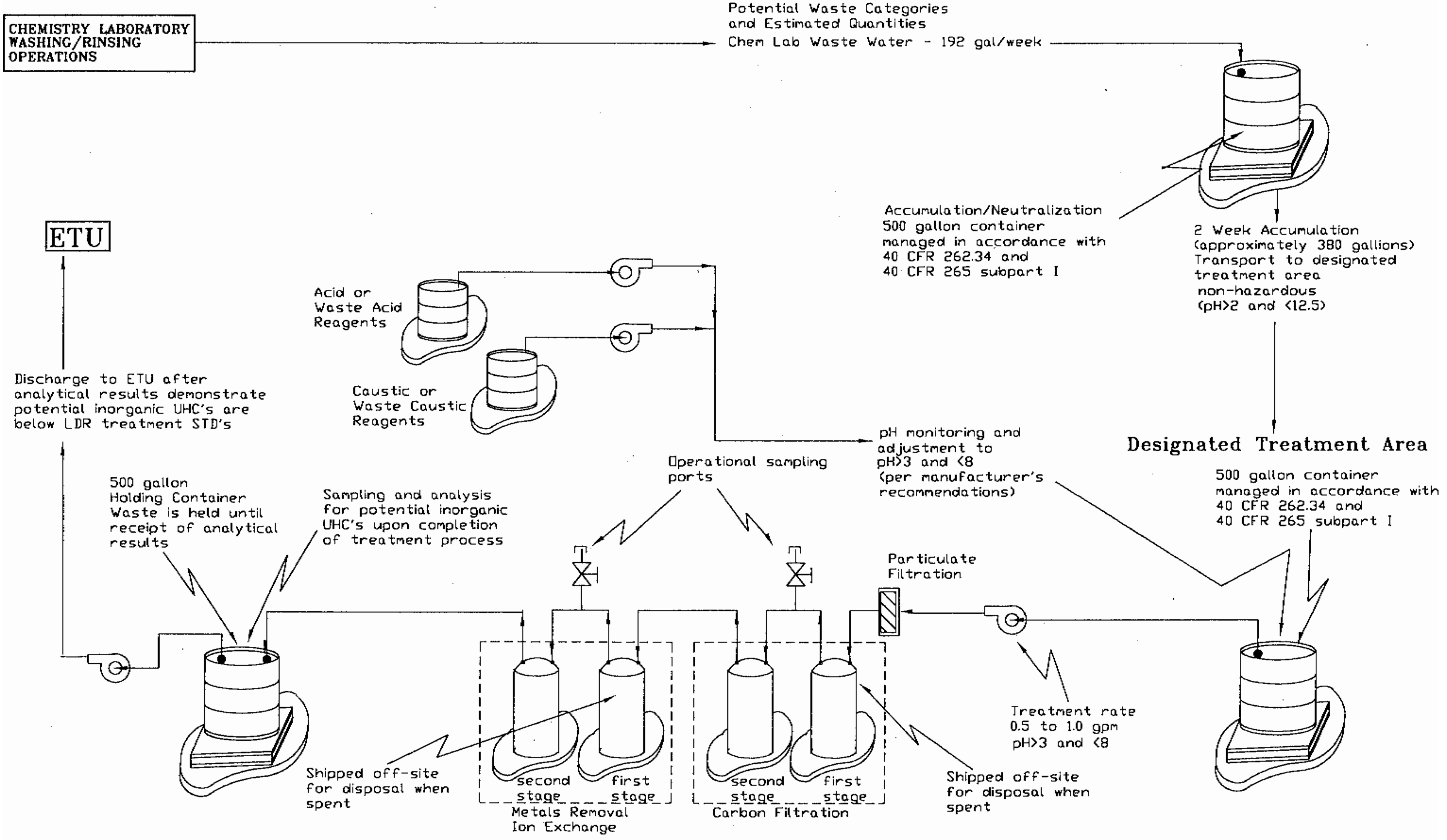


Appendix 3

200 Area Chemistry Laboratory Conceptual Design of Treatment Process

200 AREA CHEMISTRY LABORATORY – CONCEPTUAL DESIGN OF TREATMENT PROCESS

200 Area Chemistry Laboratory Area

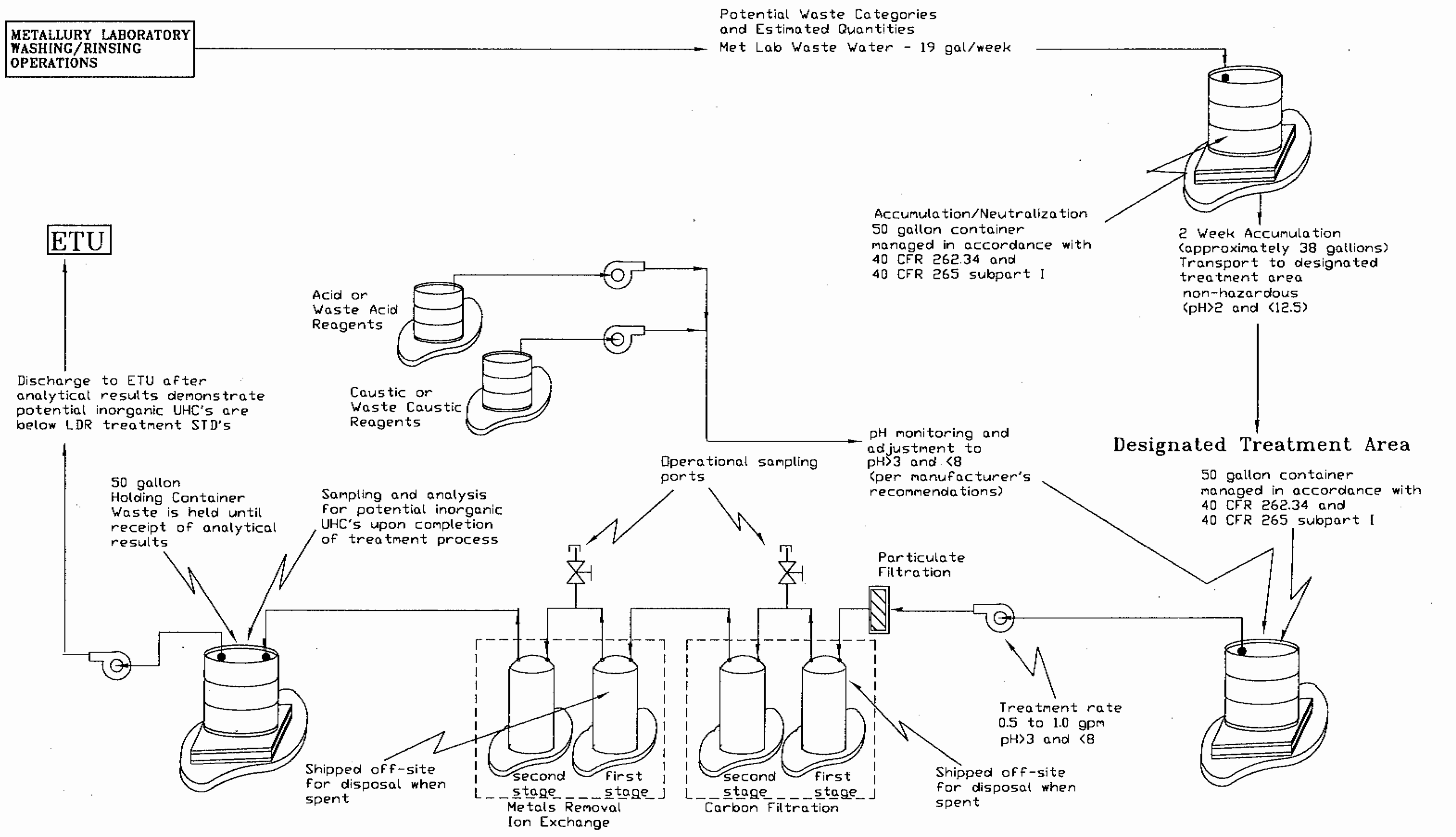


Appendix 4

200 Area Metallurgy Laboratory Conceptual Design of Treatment Process

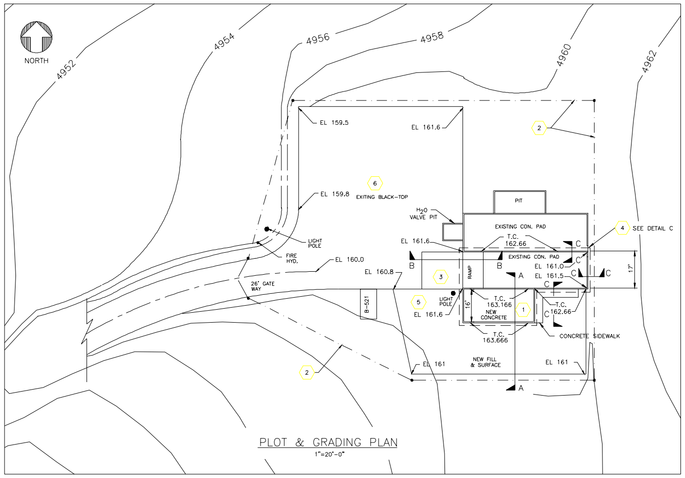
200 AREA METALLURGY LABORATORY – CONCEPTUAL DESIGN OF TREATMENT PROCESS

200 Area Metallurgy Laboratory Area



Appendix 6-G

FTU Diagrams



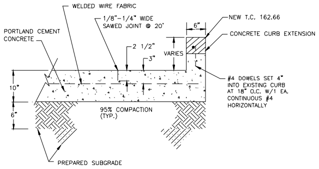
PLOT & GRADING PLAN

1"=20'-0"

LEGEND

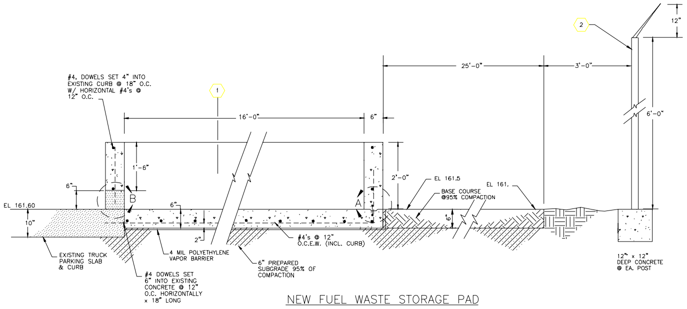
- - - - SECONDARY CONTAINMENT BOUNDARY

═══ CONCRETE CURB (SECONDARY CONTAINMENT) (T.C.=TOP OF CURB)



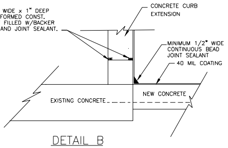
EXISTING TRUCK PARKING LANE
TYP. CONCRETE SECTION
SECTION C–C

NTS



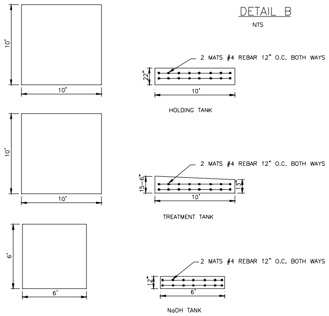
NEW FUEL WASTE STORAGE PAD
TYP. CONCRETE SECTION
SECTION A–A

NTS



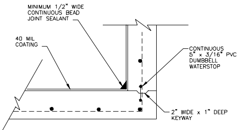
DETAIL B

NTS



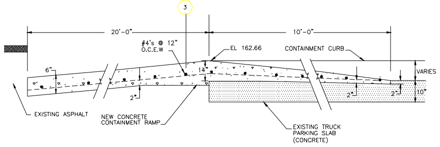
CONCRETE TANK FOUNDATIONS

N.T.S.



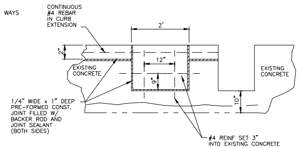
DETAIL A

NTS



NEW RAMP
TYP. CONCRETE SECTION
SECTION B–B

NTS



DETAIL C

NTS

NOTES:

⟨1⟩ NEW FUEL WASTE STORAGE PAD

- PREPARED SUBGRADE TO BE COMPACTED TO 95% OF MODIFIED PROCTOR.
- REINFORCING TO BE ASTM D1557 GRADE 60 UNLESS OTHERWISE NOTED
- CONCRETE TO BE 3000PSI DESIGNED PER ASTM C94, W/ 5-1/2 SACK/CY PORTLAND TYPE I-II CEMENT @ W.C. RATIO = .5, SLUMP = 4", 1" C.A. WATER REDUCING AND AIR ENTRAINING ADMIXTURES MAY BE USED RESULTING IN A DECREASE OF .91 GAL H2O/SACK OF CEMENT.
- ALL CONSTRUCTION JOINTS AND SURFACE OF CONCRETE TO BE COATED WITH 40MIL THICKNESS OF TECHNI-PLUS EN25 NOVOLAC COATING, OR EQUAL AND TECHNI-PLUS FLEX 3 JOINT SEALANT, OR EQUAL.
- 1'-8" HIGH CONTAINMENT BERM PROVIDES A SECONDARY CONTAINMENT VOLUME OF 6,400 GALLONS. THE CALCULATED VOLUME OF THE WASTE STORAGE TANK, NaOH TANK, AND ASSOCIATED PIPING IS 4,600 GALLONS.

⟨2⟩ NEW ISOLATION FENCE WITH WARNING SIGNS & THREE STRAND BARBED WIRE OUTRIGGER.

- 6-FOOT CHAIN-LINK FENCE TO BE INSTALLED AROUND FACILITY AND JOINED TO EXISTING CHAIN-LINK GATES ACROSS ROAD.
- FENCING MATERIAL TO HAVE HOT-DIPPED GALVANIZED COATING CONFORMING TO FS RR-F-191, INSTALLED PER MANUFACTURERS INSTRUCTIONS.

⟨3⟩ NEW TRUCK PARKING LANE CONTAINMENT RAMP.

- EXISTING ASPHALT TO BE REMOVED UNDER WEST HALF OF RAMP.
- REINFORCING TO BE ASTM D1557 GRADE 60.
- CONCRETE TO BE 3000PSI PER ⟨1⟩ ABOVE.
- THE NEW CONTAINMENT RAMP AND RAISED CURB PROVIDE A SECONDARY CONTAINMENT VOLUME OF 8,600 GALLONS. THE MAXIMUM CONTENTS OF THE TREATMENT TANK AND ASSOCIATED PIPING IS 4,200 GALLONS.
- EAST SIDE OF EXISTING TRUCK PARKING LANE TO BE COATED & JOINTS SEALED WITH SAME EPOXY COATING PER NOTE ⟨1⟩

⟨4⟩ GAP IN EXISTING CURB CLOSED PER DETAIL C

⟨5⟩ VEHICLE ACCESS AREA AROUND FUEL WASTE STORAGE PAD.

⟨6⟩ VEHICLE TURN-AROUND

SYMBOL	LETTER	ZONE	DESCRIPTION	DATE	APPROVAL
	A		INC REDLINES AS PER DCN A-1 R.E.M. 6-16-93 REVISED PER DCN A-2 C. CURTIS 7-7-93	1/27/93	A. SILVA D. OGDEN C.WOLF R. MONJARAS
	B		INC REDLINES AS PER DCN B-1 GABE. 12-13-93 REVISED PER DCN B-2 C. CURTIS 03/04/94	3-4-94 3-4-94 3-10-94 3-10-94	D. DAY G. HACKLER C. WOLF A. SILVA
	C		REVISED PER DCN C-1 C. CURTIS 05-25-94	5-25-94 5-31-94 6-1-94 6-1-94	D. DAY L. GANONG C. WOLF A. SILVA
	D		REVISED PER DCN D-1 G.GAMBOA 06-24-94	7-21-94 7-26-94 8-1-94 8-4-94	D. DAY L. GANONG C. WOLF A. SILVA
	E		REVISED PER DCN E-1 THRU E-3 R.CARPENTER 06-20-96	6-28-96 12-9-96 12-18-96 12-20-96	D. DAY L. GANONG D. AMIDEI A. SILVA
	F		REVISED PER DCN F-1 AND F-2 G. HOLCOMB 08-26-98	9-04-98 9-11-98 9-14-98 9-16-98	C. CURTIS L. GANONG D. AMIDEI A. SILVA
	G		REVISED PER DCN G-1 G. HOLCOMB 10-13-99	10-18-99 5-01-00 5-02-00 5-02-00	D. DAY L. GANONG D. AMIDEI A. SILVA
	H		REVISED PER DCN H-1 K. BERGLUND 05/16/01		

REQ'D PER ASS'Y	ITEM NO.	PART NO.	DESCRIPTION	MATERIAL	SPECIFICATION	SHT. NO.

NEXT ASS'Y / RELATED DWG'S

DIMENSIONAL TOLERANCE UNLESS NOTED OTHERWISE
.0± ANGLES ±
.00± FRACTIONS ±
.000±
DIMENSIONS ARE IN INCHES.

SURFACE FINISH IN MICRO INCHES RMS UNLESS OTHERWISE NOTED. √

UNLESS NOTED OTHERWISE REMOVE ALL SHARP EDGES AND BURRS.

DRAWN BY	G.P. MONTALVO	2-17-93
ENGINEER	D. OGDEN	2-17-93
DESIGNED BY	D. OGDEN	2-17-93
CHECKED BY	A. SILVA	3-8-93
APPROVAL	G. HACKLER	3-8-93
APPROVAL	C. WOLF	3-8-93
AUTHORIZED	J. DAWDY	3-9-93
RELEASE	R. MONJARAS	3-10-93

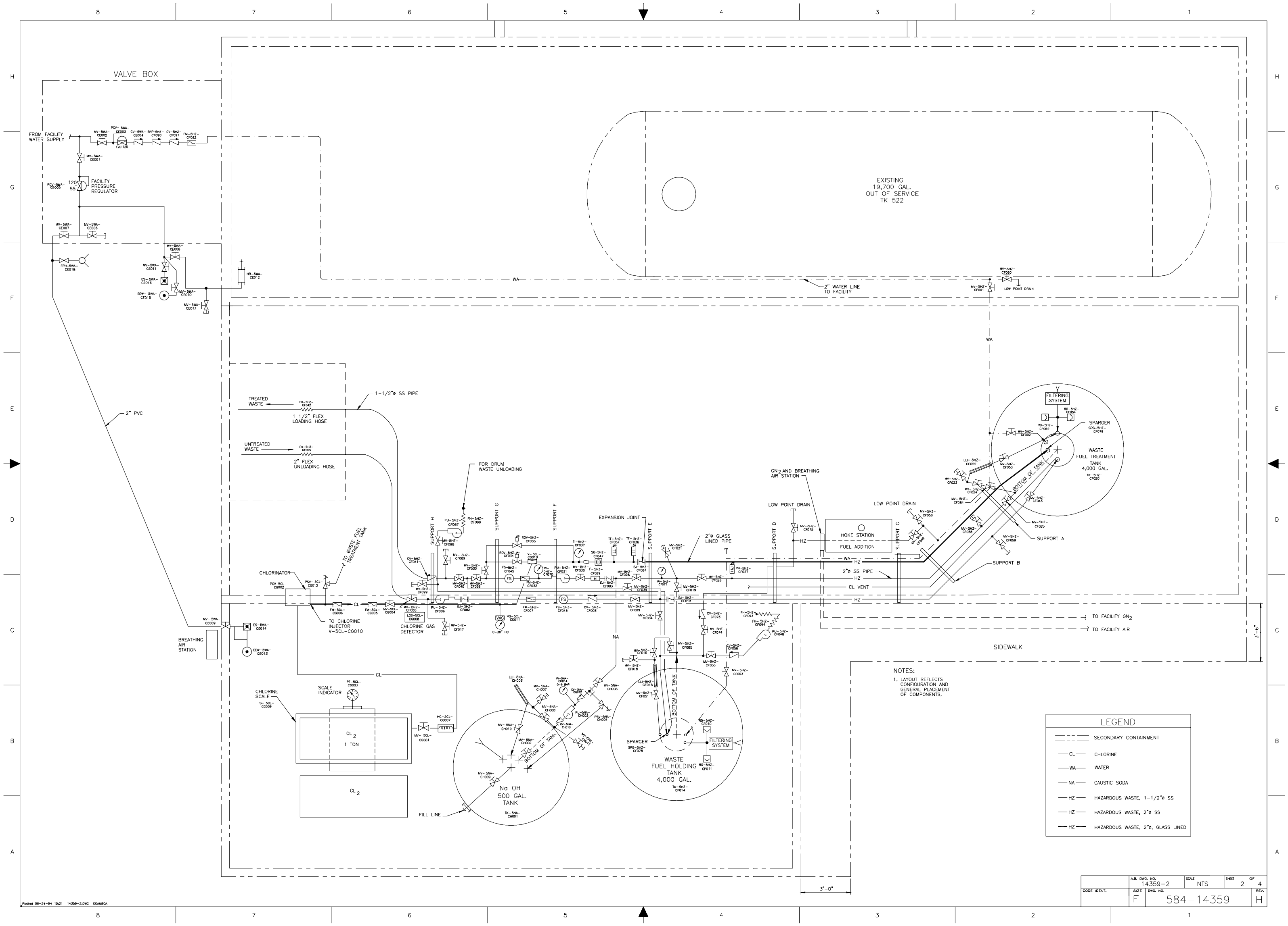
National Aeronautics and
Space Administration
NASA

Lyndon B. Johnson Space Center
White Sands Test Facility

500 AREA
WASTE FUEL TREATMENT FACILITY
PLUMBING PLAN

CODE IDENT.	SIZE	A.B. DWG. NO. / DWG. NO.	SCALE	SHEET	REV.
	F	584-14359		1 OF 4	H

FTU Process Piping Diagram



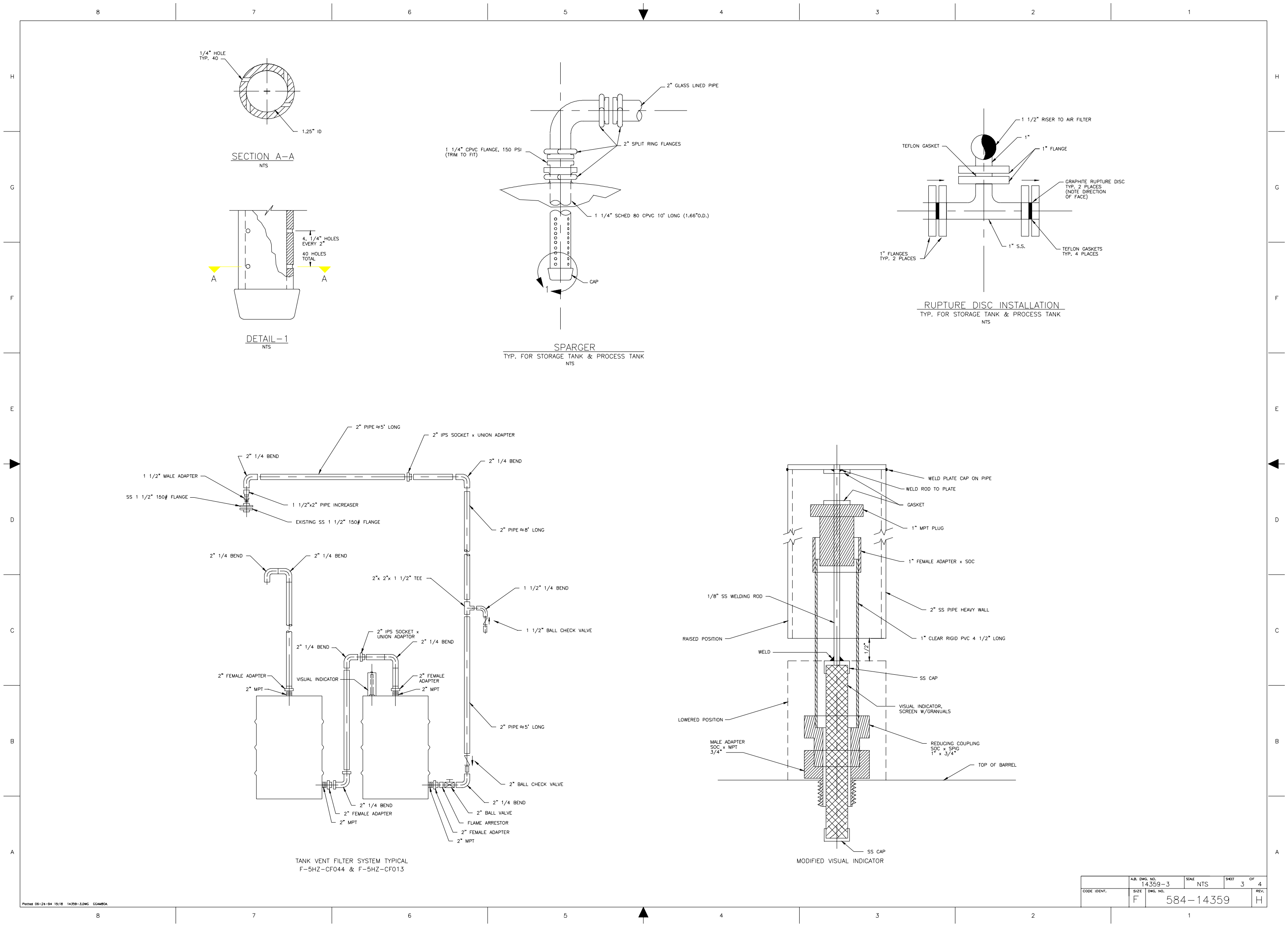
NOTES:

1. LAYOUT REFLECTS CONFIGURATION AND GENERAL PLACEMENT OF COMPONENTS.

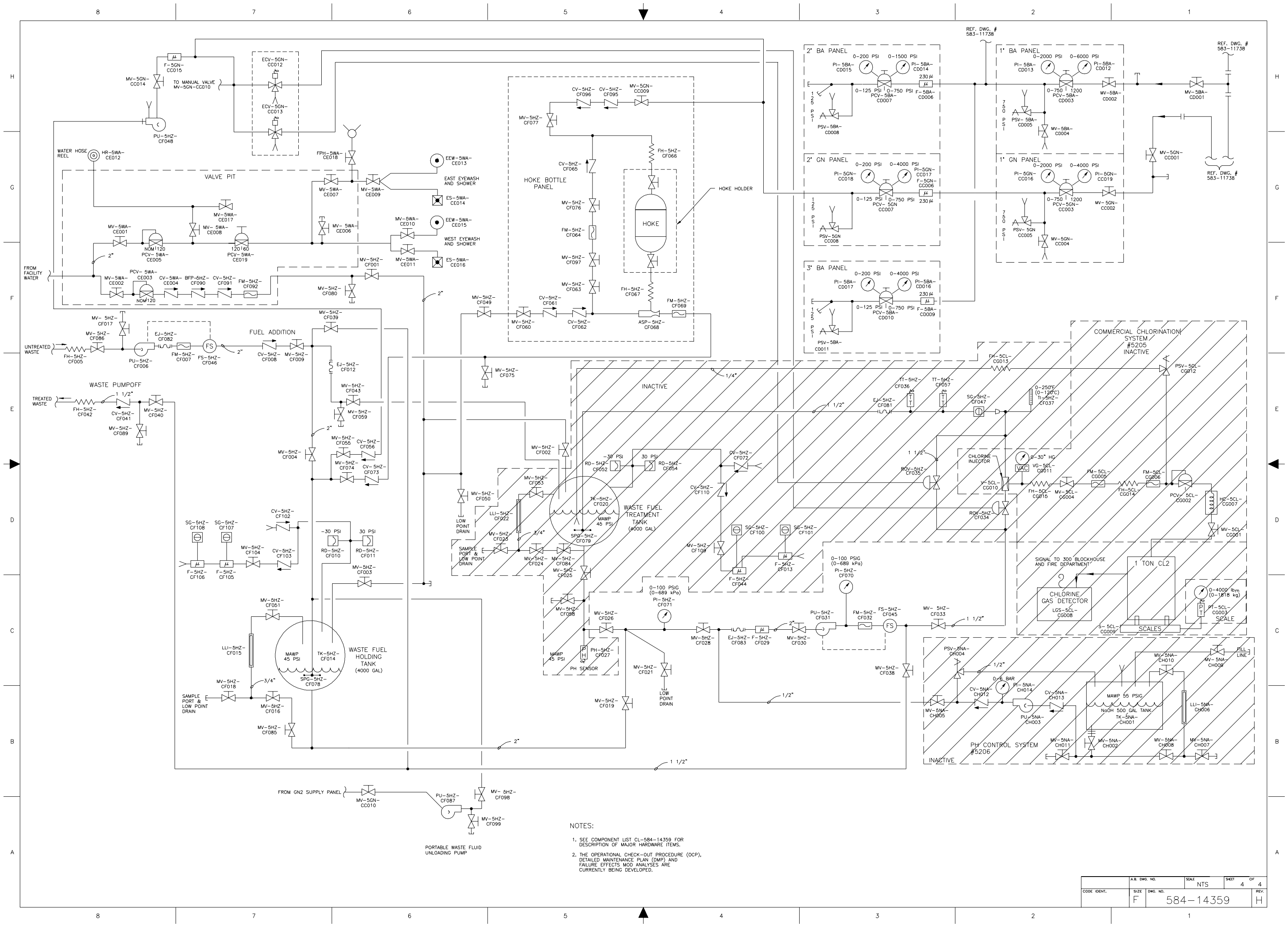
LEGEND	
═ ═ ═	SECONDARY CONTAINMENT
—CL—	CHLORINE
—WA—	WATER
—NA—	CAUSTIC SODA
—HZ—	HAZARDOUS WASTE, 1-1/2"ø SS
—HZ—	HAZARDOUS WASTE, 2"ø SS
━HZ━	HAZARDOUS WASTE, 2"ø, GLASS LINED

	A.B. DWG. NO. 14359-2		SCALE NTS	SHEET 2 OF 4
CODE IDENT.	SIZE F	DWG. NO. 584-14359		REV. H

FTU Supplemental Construction Diagram



FTU Pressure Vessel/System Certification Diagram

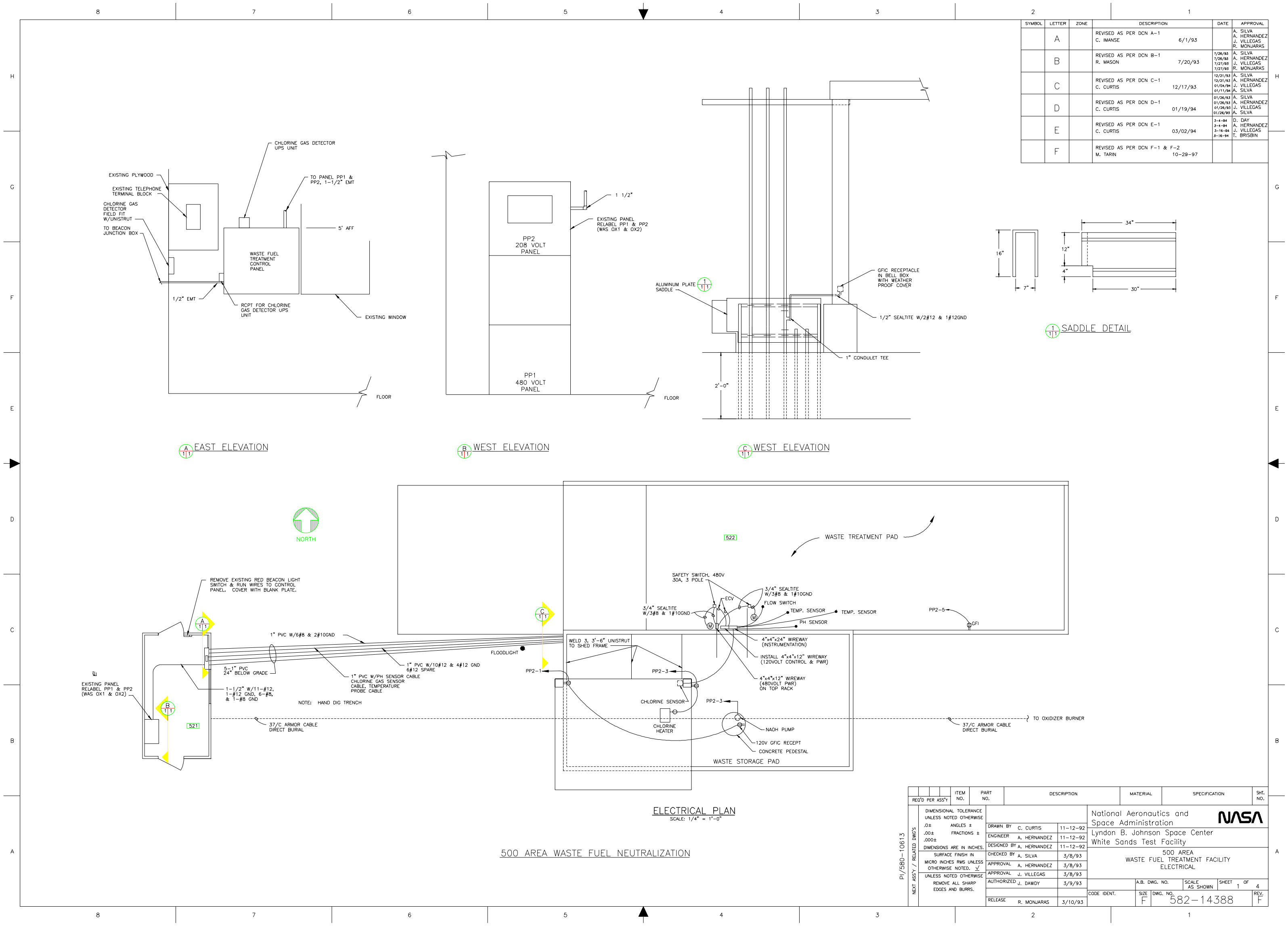


NOTES:

1. SEE COMPONENT LIST CL-584-14359 FOR DESCRIPTION OF MAJOR HARDWARE ITEMS.
2. THE OPERATIONAL CHECK-OUT PROCEDURE (OCP), DETAILED MAINTENANCE PLAN (DMP) AND FAILURE EFFECTS MOD ANALYSES ARE CURRENTLY BEING DEVELOPED.

SCALE	NTS	SHEET 4	OF 4
SIZE F	DWG. NO. 584-14359	REV. H	

FTU Electrical Diagram #1



500 AREA WASTE FUEL NEUTRALIZATION

SYMBOL	LETTER	ZONE	DESCRIPTION	DATE	APPROVAL
	A		REVISED AS PER DCN A-1 C. IMANSE 6/1/93		A. SILVA A. HERNANDEZ J. VILLEGAS R. MONJARAS
	B		REVISED AS PER DCN B-1 R. MASON 7/20/93	7/26/93 7/26/93 7/27/93 7/27/93	A. SILVA A. HERNANDEZ J. VILLEGAS R. MONJARAS
	C		REVISED AS PER DCN C-1 C. CURTIS 12/17/93	12/21/93 12/21/93 01/04/94 01/11/94	A. SILVA A. HERNANDEZ J. VILLEGAS A. SILVA
	D		REVISED AS PER DCN D-1 C. CURTIS 01/19/94	01/26/93 01/26/93 01/26/93 01/26/93	A. SILVA A. HERNANDEZ J. VILLEGAS A. SILVA
	E		REVISED AS PER DCN E-1 C. CURTIS 03/02/94	3-4-94 3-4-94 3-16-94 3-16-94	D. DAY A. HERNANDEZ J. VILLEGAS T. BRISBIN
	F		REVISED AS PER DCN F-1 & F-2 M. TARIN 10-29-97		

REQ'D PER ASS'Y	ITEM NO.	PART NO.	DESCRIPTION	MATERIAL	SPECIFICATION	SHT. NO.

DIMENSIONAL TOLERANCE UNLESS NOTED OTHERWISE
.0± ANGLES ±
.00± FRACTIONS ±
.000±
DIMENSIONS ARE IN INCHES.
SURFACE FINISH IN MICRO INCHES RMS UNLESS OTHERWISE NOTED. √
UNLESS NOTED OTHERWISE REMOVE ALL SHARP EDGES AND BURRS.

DRAWN BY	C. CURTIS	11-12-92
ENGINEER	A. HERNANDEZ	11-12-92
DESIGNED BY	A. HERNANDEZ	11-12-92
CHECKED BY	A. SILVA	3/8/93
APPROVAL	A. HERNANDEZ	3/8/93
APPROVAL	J. VILLEGAS	3/8/93
AUTHORIZED	J. DAWDY	3/9/93
RELEASE	R. MONJARAS	3/10/93

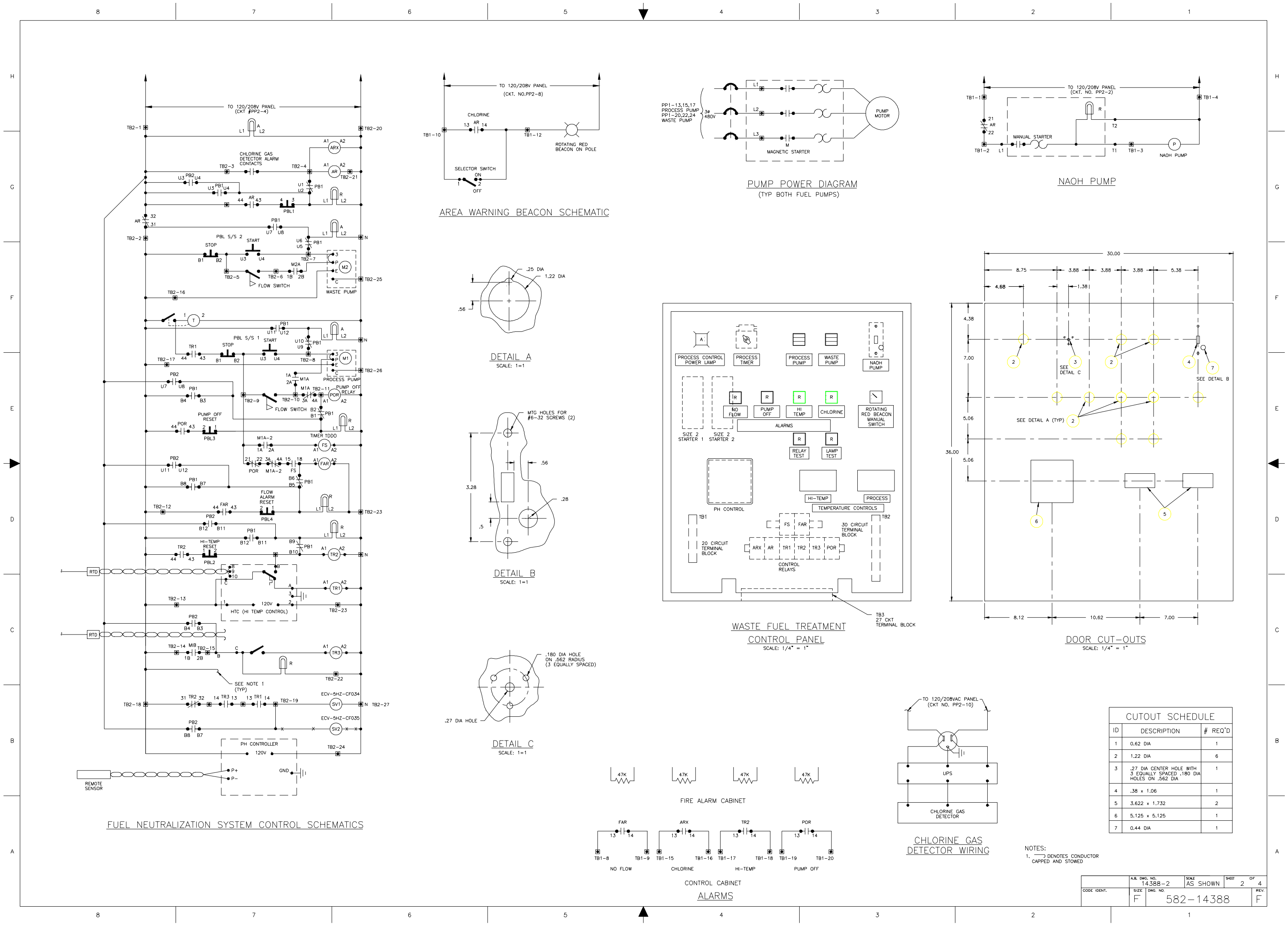
National Aeronautics and
Space Administration
NASA
Lyndon B. Johnson Space Center
White Sands Test Facility

500 AREA
WASTE FUEL TREATMENT FACILITY
ELECTRICAL

	A.B. DWG. NO.	SCALE	SHEET	OF
		AS SHOWN	1	4

CODE IDENT.	SIZE	DWG. NO.	REV.
	F	582-14388	F

FTU Electrical Diagram #2



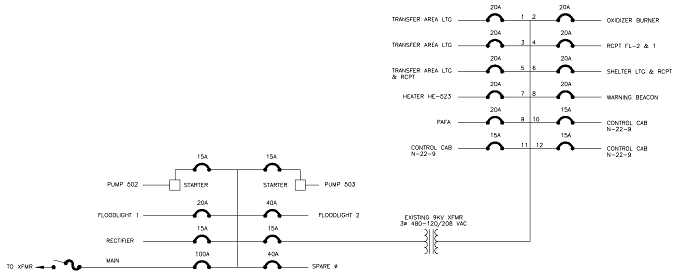
CUTOUT SCHEDULE

ID	DESCRIPTION	# REQ'D
1	0.62 DIA	1
2	1.22 DIA	6
3	.27 DIA CENTER HOLE WITH 3 EQUALLY SPACED .180 DIA HOLES ON .562 DIA	1
4	.38 x 1.06	1
5	3.622 x 1.732	2
6	5.125 x 5.125	1
7	0.44 DIA	1

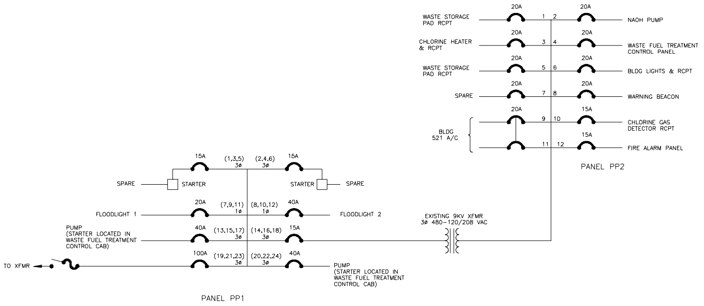
A.B. DWG. NO. 14388-2	SCALE AS SHOWN	SHEET 2 OF 4
CODE IDENT.	SIZE F	DWG. NO. 582-14388

REV. F

FTU Electrical Diagram #3



EXISTING POWER DISTRIBUTION

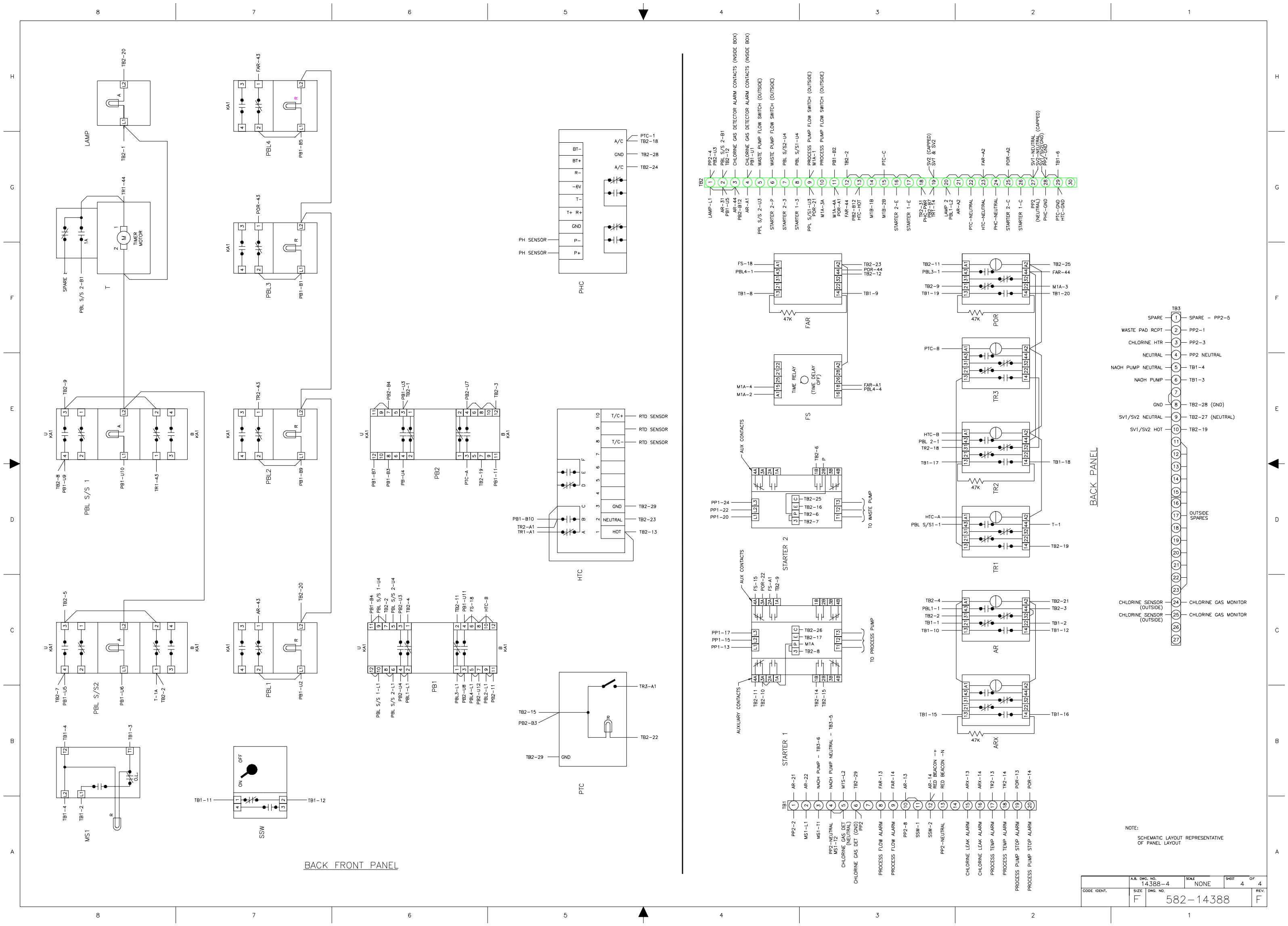


NEW POWER DISTRIBUTION

	A.B. DWG. NO.		SCALE	SHEET	OF	
	14388-3		NONE	3	4	
CODE IDENT.	SIZE	DWG. NO.				REV.
	F	582-14388				F

Plotted 07-28-93 13:10

FTU Electrical Diagram #4



Appendix 6-H

FTU Photos

Front entrance to the WSTF waste fuel treatment unit (FTU). This view incorporates the required fencing and signage, bldg. 521, light tower and tanks. (June 2002)

FTU work area. Active storage tank is to the right behind the process piping. Inactive caustic tank is to the right of the active storage tank. Inactive treatment tank is at the end of the tanker slot/work area. Large flex hoses are the tanker on-load and off-load hoses. Small flex hoses are the container on-load and off-load hoses. Stile crosses secondary containment wall. Secondary containment is sealed with epoxy/novalac coating (gold color on concrete). (June 2002)

Active storage tank showing sight glass, vent, and addition piping. Pipe rack showing filter, pressure gauge, pipe labels, and low-point drain. (June 2002)

Inactive treatment tank showing red valve lockouts w/yellow tags, tank pedestal, sight glass, and circulation piping. Site process water is copper line coming from overhead left. (June 2002)

FTU process piping rack showing active storage tank in rear. Also showing is the breathing air and GN2 panels with the HOKE station panel with S.S. sink in the background. Also shown are the secondary containment gold and white coatings. (June 2002)

Process piping with active storage tank and inactive caustic tank in background. Pumps on pedestals are Cranes (green) and Aro (behind galvanized sheet metal box). Crane pump emergency shut-off boxes are gray boxes on the pipe supports. (June 2002)

Active storage tank in background. Breathing air and gaseous nitrogen panels on left side. Test Stand 302 in far background. Water hose reel is in foreground with the copper site-water line. (June 2002)

HOKE panel with S.S. sink containment. Shows HOKE holder, flow meters, aspirator, and water bypass components. Also shows coal tar urethane joint in flooring. (June 2002)

Appendix 6-I

WSTF Job Instruction
WJI-CHEMLAB-0268

White Sands Test Facility Job Instruction

Issued: 03/17/08
Expires: N/A

WJI-CHEMLAB-0268.D

DETERMINATION OF PROPELLANT HYDRAZINES IN AQUEOUS SOLUTION USING HEWLETT PACKARD HPLC 1100 SERIES WITH AMPEROMETRIC DETECTION

Work-Authorizing Document: Laboratory Work Order

Prepared By:	Original Signed By Tom P. Martinez Chemist NASA Test and Evaluation Contract (NTEC)
Reviewed By:	Original Signed By Ben Greene Laboratory Safety and Quality Assurance NASA Test and Evaluation Contract (NTEC)
Approved By:	Original Signed By John Caruso III, PhD, Branch Manager Chemistry and Materials Section NASA Test and Evaluation Contract (NTEC)
	Original Signed By Miguel J. Maes NASA Laboratories Office

Rescissions: None.

Record of Revision

Issue	Date Revised	Pages Affected	Description of Changes
WJI-CHEMLAB-0268.B	09/15/99	All	Corrections and Updated Procedures
WJI-CHEMLAB-0268.C	07/20/06	All	Fire Brick Preparation Updated, and Minor Corrections Throughout
WJI-CHEMLAB-0268.D	03/17/08	All	Utilize new HPLC column, change flowrate from 1.0 ml/min to 1.5 ml/min, add MSM document, add holding times for samples and firebrick, and update applicable waste dispositions

1. PURPOSE

The purpose of this WJI is to establish a routine procedure for the operation of the Hewlett Packard 1100 Series equipped with a Hewlett Packard 1049A Programmable Electrochemical Detector for the determination of hydrazine, monomethylhydrazine, and 1, 1-dimethylhydrazine in aqueous solutions.

2. REFERENCES

2.1 RD WSTF-0004, *WSTF Laboratories Department Chemical Hygiene Plan*

2.2 Management System Manual (MSM), 4.2.1.6, *Environmental*

2.3 WJI-LSAFETY-0229, *Hazardous Chemical Spill Procedures*

2.4 User's Manual, *Hewlett Packard 1049A*

2.5 Installation and Maintenance Manual, *Autosampler*

2.6 User's Manual, *Hewlett Packard 1100 Series*

2.7 User's Manual, *Understanding Your ChemStation*

2.8 Johnson, D. L. and Baker, D. L. *Determination of Hydrazine, MMH, and UDMH in Aqueous Solution Using Cation Exchange HPLC with Amperometric Detection; Application to Airborne Propellant.* CPIA-PUB-588, August 1992.

2.9 Luskus, L. J. and Kilian, H. J. *Field Sampling and Analysis of Hydrazines and UDMH Vapors in Air: The Firebrick Method*. USAF School of Medicine. Report SAM-TR-82-29, October 1982.

3. ABBREVIATIONS AND ACRONYMS

CIC	Chemist in Charge
ICS	Instrument Check Standard
HPLC	High Performance Liquid Chromatography
LWO	Laboratory Work Order
MMH	Monomethylhydrazine
MSDS	Material Safety Data Sheet
S/N	Serial Number
OSHA	Occupational Safety and Health Administration
PPE	Personal protective equipment
RPD	Relative Percent Difference
UDMH	1,1-dimethylhydrazine
WJI	White Sands Test Facility Job Instruction

4. ATTACHMENTS

4.1 Attachment A, Validation Data and Calibration Data

5. SAFETY REQUIREMENTS

5.1 OSHA Requirements

To meet the OSHA requirements of the Office of the Federal Register National Archives and Records Administration Title 29, Part 1910, Section 1450 “Occupational Exposure to Hazardous Chemicals in Laboratories,” the *WSTF Laboratories Chemical Hygiene Plan* and all relevant MSDS’s must be read.

5.2 Personal Protective Equipment (PPE)

The following personal protective equipment must be worn when handling dilute standards, samples, inorganic salts, and alcohols to prevent exposure:

- Safety goggles
- Lab coat
- Nitrile gloves
- Non-porous leather footwear without eyeholes that could admit liquid

The following PPE must be worn when handling concentrated liquid acids or neat hydrazines and concentrated samples:

- Chemical splash goggles and face shield meeting the requirements of ANSI Z87.1
- Lab coat
- Rubber apron
- MAPA Stanflex V-20 (yellow Pylox) gloves or butyl rubber gloves
- Non-porous leather footwear without eyeholes that could admit liquid.

Hydrazines used in this method are known or suspected carcinogens. Chemicals that are encountered in this procedure include:

- Hydrazine
- Monomethylhydrazine
- 1,1-dimethylhydrazine
- Phosphoric acid
- Potassium phosphate, monobasic
- Potassium chloride
- Isopropanol
- Methanol
- Hydrochloric acid
- Sulfuric acid

.

6. GENERAL

6.1 Methodology

This method is used for the quantitative determination of propellant hydrazines at trace concentrations. Analytes are separated by cation exchange chromatography in a potassium phosphate buffer at pH 2.3 and detected amperometrically on a platinum working electrode. Retention times vary as the column ages. Hydrazine is the first analyte to elute, then MMH, followed by UDMH. This method measures free hydrazines.

The maximum holding times (shelf lives) of samples from the data of sampling to the date of analysis are as follows:

Liquid samples, acidified (pH <2)	14 days
Solid samples (soil, concrete, etc.)	14 days
Firebrick samples	14 days
Solution eluted from firebrick samples	14 days

Liquid and solid samples should always be analyzed as soon as possible after receipt. Firebrick samples should be eluted and the eluant analyzed as soon as possible after receipt.

The samples and standards analyzed in this procedure are generally preserved by acidification to pH<2 with hydrochloric acid. When un-acidified samples are encountered, they must be kept in a hood in sealed vials and analyzed as soon as possible. Un-acidified samples may be acidified in the laboratory as required.

Analytical standards have a nominal expiration date of one year, at which time they should be discarded and re-prepared.

6.2 Personnel Qualifications

Personnel performing the operation must be qualified as a Liquid Chromatograph Analyst according to the *WSTF Certification Plan* and demonstrate the ability to meet the performance criteria summarized in Table 1.

6.3 Quality Control

Data quality from this analysis is monitored by the analysis of check standards, blanks, spikes, duplicates, and control samples. Table 1 specifies the minimum required QA/QC samples. The analysis of additional QA/QC samples is up to the discretion of the CIC.

Table 1. QA/QC Requirements for Fuels by HPLC

Control Type	Minimum	Control Limits	Comments

	Frequency		
Instrument Check Std.	Beginning and end of analysis	± 15% of Known Value	Verify Calibration
Reagent Blanks	Beginning and end of analysis	< Reporting Limit	Establish the System is Clean
Spiked Samples	Chemist's discretion	75% - 125%	Verify Accuracy
Duplicate Samples	Chemist's discretion	RPD ≤ 20%	Verify Precision

6.3.1 CALIBRATE BEFORE USE

The HP HPLC 1100 Series is a "calibrate before use" instrument. Quantitative analysis is achieved by the comparison of the instrument's response to a sample versus a calibration curve obtained by using propellant grade hydrazine fuels conforming to their current military specifications. The identity of the fuels and the traceability to the military specification analysis of the fuels must be recorded in the Analytical Laboratory Liquid Chromatography Standards Logbook. The identity of the instrument check standard(s) (and the calibration standards, if applicable) and the corresponding samples analyzed must be recorded in the HP 1100 HPLC Calibrate Before Use Logbook with enough information provided to achieve traceability of samples to standards. At a minimum the logbook will include the following:

- Date of Analysis
- Analysts Initials
- Date last calibrated.
- ICS concentration and preparation date.
- Samples Analyzed (or reference to samples). At a minimum annotate the sequence file name.

Calibration reports/curves must be maintained in the Calibration Logbook. This logbook will include, at a minimum, the instrument's ID number, a reference to previous Calibration Logbooks, and a complete record of all calibration activities performed to date, including identification and source of standards. The calibration standard preparation dates will also be included.

All instrument maintenance must be recorded in the instrument maintenance logbook.

6.3.2 INSTRUMENT CHECK STANDARD

One ICS must be run at the beginning of an analytical batch. The CIC will determine the appropriate ICS concentration to be analyzed. It must contain all the fuels that are analyzed in the batch. The result must be within ± 15 percent of the nominal value. If not within accepted limits, re-analyze the check standard. If the results are still outside accepted limits, notify the CIC for resolution of the problem. The instrument may require recalibration.

The check standard must be run at the end of the batch to verify system calibration throughout the analyses. If the check standard fails, all results for samples analyzed after the last passing check standard are considered invalid and these samples must be reanalyzed or appropriately qualified.

6.3.3 BLANK

After calibration has been verified, a reagent blank must be analyzed before any samples to ensure the system is free of contamination. The reagent blank must be run at the end of the batch to verify system cleanliness throughout the analyses. Eluant should be run as the reagent blank. If an analyte is found at a level greater than or equal to the reporting limit all results for samples analyzed after the last passing blank are considered invalid and these samples must be reanalyzed or appropriately qualified.

6.3.4 MATRIX SPIKE

A matrix spike is an aliquot of sample spiked with a known concentration of a target analyte. The spike concentration should be at least 10 times the WSTF reporting limit for the analyte of interest or greater than the sample concentration, whichever is higher. Consult the CIC when preparing a matrix spike. Prepare the matrix spike again if the recovery is not within ± 25 percent of the known spike concentration. If it fails again, consult the CIC.

6.3.5 DUPLICATES

A duplicate is the analysis of the same sample twice. The calculation of duplicate precision is described in Step 8.7.3. If the RPD is not ≤ 20 % consult the CIC.

6.4 Data Acquisition

ChemStation is a Hewlett Packard product for data acquisition and reduction as well as instrument control. The HP ChemStation user interface is designed into Views that group software functionality according to typical analytical tasks. Three standard views are present in all software configurations:

- Method and Run Control view, for controlling and acquiring data from the instrument.
- Data Analysis view, for reviewing and reevaluating data that has been acquired.
- Report Layout view, for designing specific report layouts.

A method is comprised of three components:

- Method information, descriptive information about the method.
- Instrument control, defines the parameters that control the instrument or its components. Parameters such as mobile phase composition, flow rate, injection volume, the detector settings, etc.
- Data analysis defines the parameters that control the data processing: signal details, integration events, peak identification, peak quantification, calibration, and the report.

The graphical user interface of the HP ChemStation system comprises toolbars and a status bar in the Method and Run Control View of the HP ChemStation. The status bar comprises a system status field and information on the currently loaded method and sequence. If they were modified after loading they are marked with a red triangle. A yellow EMF symbol reminds the user that usage limits set for consumables have been exceeded. Consult the ChemStation manual for more detailed information on EMF.

Method and Run Control View illustrates a graphical representation of your HPLC system. An icon represents each component. The diagram allows quick access to dialog boxes for setting parameters for each system component. Using the following color-coding, the current status is displayed:

- yellow- not ready
- green- ready
- blue- run
- red- error

7. SYSTEM VALIDATION

The HPLC 1100 Series system was validated in accordance with good analytical practices. System validation documentation is found in Attachment A. Also refer to Reference 2.7.

8. PROCEDURE

8.1 Reagents and Apparatus

All chemicals should conform to analytical reagent grade specifications or otherwise be of suitable purity (free of interfering substances). The water used to prepare the reagents and buffer should be purified such as that from the Milli-Q system. For best system performance and results, filter particulates from all solutions including samples.

8.1.1 ELUANT SOLUTION

The eluant is a potassium phosphate buffer with a pH of 2.3. The buffer is prepared by dissolving 0.35 g of KCl, 9.00 g of KH_2PO_4, and 4.0 mL of conc. H_3PO_4 diluted up to 1.00 L of deionized water. The buffer is filtered under vacuum through a 0.45 µm nylon filter. Transfer to the eluant reservoir. The reservoir should be labeled with the contents (KCl, KH_2PO_4, and H_3PO_4, hazard (toxic, corrosive), your initials and date prepared. Use of the WSTF 394 label is encouraged. Excess eluant should be similarly labeled and then refrigerated.

8.1.2 STOCK FUEL STANDARDS

When preparing stock standards from neat propellant, use the proper handling procedures associated with this task. Prepare 500 mL of 1% v/v H_3PO_4 by bringing 5 mL of conc. H_3PO_4 up to 500 mL with deionized water. Clean three 100 mL volumetric flasks thoroughly with deionized water. Add 1% v/v H_3PO_4 almost to the line on the flasks. Stopper the flasks. Solutions should not be stored in the volumetric flasks. Transfer the solutions to clean plastic bottles and label each bottle with the fuel name, concentration, hazard (toxic, carcinogen, corrosive), your initials and date prepared. Use of the WSTF 394 label is encouraged. In the Analytical Laboratory Liquid Chromatography Standards Logbook record the grams of each stoppered flask to 4 decimal places. Add 0.10 g of the appropriate fuel to the appropriate flask.

Fuel	Density
Hydrazine	1.0036 g/mL
MMH	0.874 g/mL
UDMH	0.782 g/mL

Stopper the flasks and record the grams to 4 decimal places. Use the difference of the gross weight and the initial weight to determine the mg of fuel added. Bring each flask to volume with 1% v/v H_3PO_4 and calculate the concentration in mg/L taking into account the actual purity of the neat propellant. Record the Hoke S/N of each propellant used with the associated LWO number of the military specification analysis performed on that propellant, date of preparation, and preparer’s initials in the Analytical Laboratory Liquid Chromatography Standards Logbook.

Transfer each standard to a plastic bottle or other suitable storage container (i.e. I-Chem). Label each with the calculated analyte concentration, matrix, Hoke S/N, LWO number of the military specification analysis, date of preparation, and preparer’s initials.

8.1.3 CALIBRATION STANDARDS

Use the formula $C_1V_1 = C_2V_2$ to calculate the volume of stock fuel standard needed to result in a 100 mg/L fuel standard mix in a 100 mL volumetric flask. Prepare the dilution with eluant.

From the 100 mg/L fuel standard mix, perform the appropriate dilutions, with eluant, required to generate the necessary calibration curve. Store the calibration standards in suitable storage containers labeled with concentration of standard, matrix, preparation date, preparer’s initials, and reference the stock standards prepared from. Record the preparation of each standard in the Analytical Laboratory Liquid Chromatography Standards Logbook.

8.1.4 INSTRUMENT CHECK STANDARD

The ICS and calibration standards must be prepared from independently prepared stock fuel standards. The preparation of these standards and the traceability to each Hoke and military specification analyses, will also be recorded in the Analytical Laboratory Liquid Chromatography Standards Logbook.

8.1.5 APPARATUS

In addition to general laboratory glassware, required apparatus includes 5 mL plastic syringes, 13 mm filters with 0.2 or 0.45 micron porosity, HPLC vials and caps, pipettes, and plastic transfer pipettes.

8.2 Instrument Setup

The liquid chromatograph, column, and conditions are given below. Virtually all of the operating parameters are retained in the memory and controlled through HP ChemStation.

8.2.1 HPLC

Hewlett Packard Model 1100 Series high performance liquid chromatograph equipped with a Model 1049A Electrochemical Detector. Injections are made using an autosampler.

8.2.2 COLUMN

The column is a strong cation exchange column (i.e. Phenomenex Luna 10 SCX, 100A[250 x 4.60mm], p/n 00G-4401-E0) See Section 9.3 for column maintenance.

8.2.3 DEGASSER

Eluant flows from the reservoir through channel A of the vacuum degasser. The degasser is generally left on and is operating properly when both lights on the front panel (Power and Monitor) are illuminated.

8.2.4 PUMP

The pump is set to deliver eluant at a flow rate of 1.500 mL/min during analyses. The backpressure from the system is typically about 60 bar at this flow rate. If the pressure exceeds 80 bar, indicating a blockage, investigate. The high pressure limit parameter is set to 400 bar; the pump will go into ERROR mode if this pressure is reached.

8.2.4.1 Pump Parameters (ChemStation)

Accessed by using the mouse to click on the pump/injection portion of the diagram

FLOWRATE	1.500 mL/min	
STOPTIME	10.00min (may be increased if needed)	
SOLVENT	A: 100% Phosphate Buffer (isocratic)	
PRESSURE LIMITS	Max 400 bar	Min 0 bar
INJECTION WITH NEEDLE WASH		
INJECTION VOLUME	Variable Method dependent	
WASH VIAL	71	
AUXILLIARY	draw speed 200 μL eject speed 200 μL draw position 0.0 min compressibility $100x10^{-6}$ max flow rate 100.00 mL/min	

8.2.5 AUTOSAMPLER

The design of the autosampler provides for very little carryover. Contamination of the outside of the autosampler sampling needle or needle seat, are the sources of cross contamination or carryover. The HP 1100 Series has a rinse cycle programmed in the method.

These precautions do not protect against needle seat contamination. Analytes from highly concentrated samples are carried on the needle tip to the needle seat during injections. A rinse is not practical during this part of the injection. The best safeguard against needle seat contamination is to simply dilute samples that are suspected to be highly concentrated. If needle seat contamination occurs, consult the maintenance CD-ROM.

8.2.2.1 HP 1100 Series Autosampler

The parameters are set up in the **HP 1100 SERIES PUMP, (Section 8.2.4.1)**.

8.2.3 DETECTOR

The analytes are detected by oxidation at a platinum working electrode relative to a Ag/AgCl reference electrode. The cell body is made of graphitized Teflon and functions as the counter electrode.

The detector is programmed to condition the platinum working electrode by pulsing the voltage between it and the auxiliary electrode for 84 seconds prior to each run. The detector begins pretreatment when it receives a START REQUEST from the autosampler. The detector sends a NOT READY signal to the autosampler during the pretreatment period. This period is 4.5 minutes to allow the baseline to stabilize in the direct current mode before injection.

The HP 1100 series system is equipped with the 1049A electrochemical detector controlled through ChemStation. Use the mouse to click on the detector to edit the parameters.

8.2.3.1 HP 1049A Electrochemical Detector Parameters

STOPTIME	as pump 10.00 min (may be increased if needed)
ECD POTENTIAL	LC-ECD
MODE	Pretreat
MORE	Potential
	P1 = 1.100V
	P2 = -0.200V
	P = 1.000V

	Time
	T1 = 20ms
	T2 = 100ms
	T3 = 20ms
	Cycles = 600
	Pretreat = Prepare
POTENTIAL	+ 1.000V
LIMITS	- 0.400V to + 1.400V
INCREMENT	OFF
REPETITIONS	not used
DRIFTTRIGGER	OFF
PREPARE TIME	4.5 min
STOPTIME	as pump 10.00 min (may be increased if needed)
POSTTIME	OFF
PEAKWIDTH	0.10min (Response time 2.00 sec)
ZERO CONTROL	STOP
INSTRUMENT FULL SCALE	500(μA)
POLARITY	oxidation
THERMOSTAT	not used
SIGNAL DETAILS	LC-ECD
AVAILABLE SIGNALS	ECD1 A, Pretreat, Pot = 1.000 start 0.000 end 0.000 delay 0.000

8.3 Analysis of Samples

8.3.4 SAMPLE PREPARATION

8.3.4.1 Firebrick Sample Tube Preparation

Firebrick sampling tubes can either be purchased or prepared in the laboratory. The following steps outline the preparation of a firebrick tube within the laboratory.

8.3.4.1.1 Glass Tubing

Sampling tubes are prepared in 7 mm OD glass tubing. Rinse the tubing with 5% (v/v) HNO_3 followed by deionized water. Dry with GN_2. Cut into 3.5 in segments. Fire-polish each end and create a restriction near one end by heating. Purge each tube with GN_2 to remove any condensate.

8.3.4.1.2 Inert Support Material

To prepare 100 g of solid sorbent, place 100 g of 45/60 Gas Chrom R (Alltech Associates) in 450 mL of ACS reagent grade HCl contained in a 1000-mL beaker and heat to near boiling. Maintain the temperature for 2 h with occasional swirling. Decant the HCl and add a second 450 mL portion of HCl. Heat the mixture again for 2 h with occasional swirling. Decant the HCl and add 450 mL of purified water. Heat this mixture to near boiling and decant the water. Repeat the hot water wash twice more then add 450 mL more purified water and filter the mixture using a Buchner funnel. Wash the retained Gas Chrom R thoroughly with purified water then transfer the damp cake on the filter to a 70°C nitrogen-purged oven to dry for 30-40 minutes.

8.3.4.1.3 Active Media

NOTE

ADDITION OF CONCENTRATED SULFURIC ACID TO WATER WILL GENERATE HEAT. MAKE THE ADDITION SLOWLY WITH SWIRLING AND COOL EXTERNALLY WITH ICE OR WATER AS REQUIRED.

To prepare a 6M H_2SO_4 add approximately 50 mL deionized water to a 100 mL volumetric flask, then slowly with swirling add 33 mL concentrated H_2SO_4 (18M). Bring to volume with deionized water.

To prepare a 0.86M H_2SO_4/methanol solution, add approximately 400 mL of methanol to a 500 mL volumetric flask, then add 72 mL of 6M H_2SO_4. Bring to volume with methanol.

Place 40 to 50g of dried firebrick into a tared 1000 mL beaker. Determine the weight in grams of firebrick in the beaker. Multiply the weight by 2.75. The result is the volume of 0.86M H_2SO_4 methanol solution to add to the firebrick. Pour the resulting slurry into several large petri dishes and dry in a 60°C N_2 purge oven for 30 minutes. The resulting sulfuric acid content will be approximately 60 mg per 300 mg acidified firebrick or 19% (w/w). Store the material in a closed labeled glass container. The container should be labled Firebrick (acidified with Sulfuric Acid), hazard (toxic, corrosive), your intials and date prepared. Use of the WSTF 394 label is encouraged.

8.3.4.1.4 Tube Loading

Place a small wad of Pyrex or glass wool in the tube from the end that is not restricted and gently press it down to the restriction. Using a funnel, pour 300 mg ± 15 mg of sulfuric acid coated firebrick into the tube. Gently press a second wad of Pyrex wool onto the top of the firebrick. Keep packing of the sorbent to a minimum. Fit clean, unused plastic caps to the tube ends and store the tubes in a closed glass container away from amines and hydrazines.

NOTE

USED CAPS WILL CONTAMINATE THE FIREBRICK!!!!

8.3.4.2 Firebrick Elution

Transfer the firebrick material and glass wool plugs from the firebrick tube to a test tube that will hold at least 12 mL. Add 10 mL of eluant solution using a digital or volumetric pipette. Cap and **agitate** (vortex, shake, invert or gently sonicate) the tube periodically or continually for 10 minutes. Use of a laboratory rocker is encouraged. Draw approximately 2 mL of the firebrick eluant into a 5 cc syringe. Equip the syringe with a 13 mm 0.45 micron porosity filter. Expel the eluant through the filter into an HPLC vial. Cap the vial and label with the sample ID and total eluant volume.

8.3.4.3 Solid Sample Elution

Determine if the requestor requires hydrazine concentration by dry weight or wet weight. Dry the sample if required. Accurately weigh a portion of the sample (typically 1-2 g) into a clean Erlenmeyer flask or test tube. Record the sample weight on the work authorizing document. Add 10 mL of eluant solution using a digital or volumetric pipette. Cover and agitate the container for 10 minutes then sonicate for 10 minutes. Draw approximately 2 mL of the eluant into a 5 cc syringe. Equip the syringe with a 13 mm 0.45 micron porosity filter. Expel the eluant through the filter into an HPLC vial. Cap the vial and label with the sample ID and total eluant volume.

8.3.4.4 Liquid Samples

If the customer requires a total µg determination of hydrazines measure the total volume of the sample and record on the work authorizing document. Filter the sample through a 13mm 0.45 micron porosity filter via a 5 cc plastic syringe into an HPLC vial. Cap the vial and label with the sample ID.

8.3.4.5 Cleanup

Triple rinse the filters and syringes with water. Dispose the rinsed filters and syringes as fuel contaminated soft goods in accordance with WIWPS 20-04-18 (Fuel Contaminated Soft Goods). Dispose the rinse water in accordance with WIWPS 20-04-102 (Aqueous Fuel Contaminated Waste).

8.3.1 INSTRUMENT STARTUP

8.3.1.1 Open the HP ChemStation folder. Open LC-ECD online. A diagram of the system appears on the screen. You are in the **Method and Run Control View**. It will be **red**; indicating the system is **not ready**.

8.3.1.2 Turn on the pump by clicking the mouse on the button next to the pump diagram. The pressure should increase to about 60 bar. If not, contact the CIC. Reservoir A, containing the phosphate buffer pH 2.3, should be selected indicated by the arrow. When the pump is on, the diagram will turn **green** indicating the system is in **ready** mode. When the instrument is not in use, the system should be rinsed with deionized water before it is brought down. This also will minimize corrosion and system blockage from salt build up.

8.3.1.3 Turn on the detector by clicking the **ON** button. The software will alert the operator to ensure that there is adequate flow of eluant.

8.3.2 AUTOSAMPLER

8.3.2.1 Fill a vial with eluant, cap it, and place it in position 71 of the tray. This is the wash vial. Label the vial “Blank”.

8.3.2.2 Place another blank and a fuels standard in the tray, usually in positions 1 and 2 respectively. Load the vials to be analyzed. The sequence file must match the vial order.

8.3.3 SEQUENCE FILE

Create or edit a sequence file by clicking on Sequence then New Sequence or Load Sequence, respectively. Then click on Sequence and Sequence Table. Create or edit the sequence table. Enter the method, the number of injections per vial, and sample type or identification, and the dilution factor if necessary. When the sequence is complete, click OK, then under **Sequence** go to the **Save Sequence As** command and save the sequence as the run date (e.g. 971107).

To save data files click on the directory path, under the illustration of the autosampler tray, then sequence parameters. Change the subdirectory to the run date (e.g. 971107). Click on **OK.** The files will automatically be saved as the vial number, sequence line, and injection number under the subdirectory which is saved as the run date. For example, the first injection of the first sample written in the first line of the sequence run on 11/07/97 will be 001-0101.d (sequence line, vial number, injection number) in the subdirectory 971107. The data file is located in **hpchem\2\data\971107\001-0101.d**. The software will warn you when there is a danger of overwriting files that already exist.

To program the sequence file for automatic pump and detector shutdown see Step 8.4.1

Click on **START**. If at anytime you wish to terminate the analysis, click on **STOP**. Real time view of the analysis in progress can be seen by maximizing the **Online Plot** window.

When analyzing a single vial, select the single vial icon at the top left of the Method and Run Control diagram. Then click on the vial titled MAIN SEAT. In the Sample Info screen enter the file name, and the run date under subdirectory. Identify the vial position. Click on OK, then START. To run a partial sequence, select the partial sequence option under sequence. Click the boxes in the Sel column of the vials to be analyzed, then click on Run Sequence.

8.3.4 REINTEGRATION OF CHROMATOGRAMS

To reintegrate a chromatogram and save the new file, select Data Analysis. Select file, load signal and then select the file to be reintegrated. Click OK. Select the appropriate integration task icons and reintegrate. To save reintegration select view, window functions, save window then click OK twice.

8.4 Automatic Instrument Shut Down

8.4.1 PUMP AND DETECTOR

In the **SEQUENCE PARAMETERS**, under **SHUTDOWN**, check **Post-sequence cmd/Macro** and then select **standby**. Standby will turn off the lamp and the pump immediately after the last run in the sequence table.

8.4.2 DETECTOR

Automatic shutdown of the cell can be obtained by pressing <CELL ON/OFF> on the detector front panel. Press <⮟> to display AUTO CELL = CELL OFF. Press <⮟> again to display AFTER = 0; off. Determine how many runs remain in the sequence and multiply by 15 minutes (don’t forget the blanks and standards). Use the numeric keypad to enter this number of minutes, then press <ENTER>. The cell will shut off after the specified time. Turning the cell on, resets this condition.

8.5 Manual Shutdown

8.5.1 DETECTOR

Press <CELL ON/OFF> <➢> <⮟> <ENTER> on the detector to turn the cell off **OR preferably** click on the **OFF** button on the diagram on the computer screen to turn the cell off and the pump off simultaneously. **NEVER** leave the cell on while the pump is off.

8.5.2 PUMP

Turn off the pump by clicking on the button next to the pump and hitting **OK** to turn off the pump.

8.6 Cleanup and Disposal

8.6.1 Empty the instrument waste eluant in accordance with WIWPS 20-04-102 (Aqueous Fuel Contaminated Waste).

8.6.2 Dispose of spent samples, firebrick, blanks, standards, vials, and caps in accordance with the current waste profiles described below.

For safety and quality reasons, aqueous hydrazine fuel samples in concentrations approaching 1% or and higher must be removed to an approved fuel hood in Building 200 Room 143 (Fuel Laboratory), disposed down a fuel drain, and logged appropriately as 20-04-102 (Aqueous Fuel Contaminated Waste) or 20-04-03 (Waste Fuel) depending on its origin. Never leave samples approaching these concentration ranges in a non-fuel laboratory in a manner they could be confused for dilute samples.

Dilute samples (<1%) and rinses from fuel containers (sample containers, syringes, syringe filters, transfer pipettes, etc) must be disposed in the Building 203 Room 124 (Analytical Lab) hood cup sink in accordance with WIWPS 20-04-102 (Aqueous Fuel Contaminated Waste).

Firebrick material and glass tubes containing firebrick must be disposed in accordance with WIWPS 20-04-31 (Waste Firebrick Material). Firebrick materials or glass tubes containing firebrick must be collected in a plastic beaker then bagged in plastic, checked with a hydrazine fuel vapor monitor and if free of fuel vapors disposed in the Fuel Contaminated Soft Goods Container.

Contaminated wipes, tissues, plastic ware, vial caps, firebrick tube caps and other solids meeting the definition of soft goods (or with the approval of the WSTF Environmental Department) must be disposed of as Fuel Contaminated Soft Goods (20-04-18) after verifying free of hydrazine fuel vapors using a hydrazine fuel vapor monitor.

Autosampler vials used during the analysis of samples that are made up in phosphate buffer or do not contain free (not-acidified) hydrazine fuels may be crushed using the vial crusher located in Building 203 Room 136. The vials containing liquid must be thoroughly crushed and the fines triple rinsed with water. The liquid from these vials and the rinsates are disposed in accordance with WIWPS 20-04-102 (Aqueous Fuel Contaminated Waste). The rinsed solids are bagged in plastic, verified free of hydrazine fuel vapors, then sealed and disposed in the fuel contaminated soft goods container in accordance with WIWPS 20-04-63 (Hydrazine Autosampler Refuse).

Hydrazine fuel-contaminated metal and glass (non-soft goods) are decontaminated by triple rinsing with water. The decontaminated metal and glass are considered non-hazardous waste and may then be disposed of as ordinary trash in the outdoor dumpster. Syringes must be rendered unusable prior to disposal such as by clipping off the needle. Teflon- or other polymer-tipped or polymer plungers (or that portion thereof) must be disposed as fuel contaminated soft goods in accordance with WIWPS 20-04-18. Rinse water must be disposed in accordance with WIWPS 20-04-102 (Aqueous Fuel Contaminated Waste).

Used or old chromatographic columns must be triple rinsed (normal eluant rinses suffice) then wrapped or bagged and disposed as Fuel Contaminated Soft Goods (20-04-18) because of the porous chromatographic support material inside.

8.6.3 Leave the area clean for the next person. Pick up stray debris from the floor, empty the trashcans, and place any dirty glassware on the cart for cleaning. If any glassware has been broken, clean it up immediately and place it in a broken glass receptacle.

8.7 Data Reduction and Reports

8.7.1 RESPONSE FACTORS

Linear calibration has been established for the three propellant hydrazines from 1.0 ng to 1000 ng injected (Reference 2.7). Calibration is based on peak areas. Response factors for each component are calculated as amount/response. Calibration of each analyte is done in the **Data Analysis View** under the “**scales**” or calibration version of the window.

8.7.2 Matrix spike recoveries are calculated by:

$$((C_{ms} - C_s) / C_{sp}) * 100 \qquad \textbf{(1)}$$

Where:
C_{ms} is the concentration of matrix spike
C_s is the concentration of sample
C_{sp} is the concentration of spike

Matrix spike recoveries will be between 75 and 125 percent; if not, they will be re-analyzed or another matrix spike will be prepared and analyzed.

8.7.3 Duplicate precision is defined as relative percent difference and is calculated as follows. The RPD must be ≤ 20%; if not, they must be re-analyzed.

$$RPD = \frac{/ D_1 - D_2 /}{Mean} * 100 \qquad \textbf{(2)}$$

Where:
D_1 = Duplicate 1
D_2 = Duplicate 2

8.8 Data Archival

8.8.1 The data obtained by this WJI are recorded on the report attached to the LWO and are archived with the data package from Chemistry Metallurgy Section.

8.8.2 ChemStation data files are archived frequently to appropriate storage media.

9. MAINTENANCE

NOTE

ALL INSTRUMENT MAINTENANCE MUST BE LOGGED IN THE INSTRUMENT MAINTENANCE LOGBOOK.

9.1 Detector Working Electrode

Fouling of the electrode is evidenced by broad, tailing peaks and/or increased baseline noise. A spare electrode is kept by the responsible chemist for replacement purposes. Locate the spare electrode and polish it as described on page 7-5 of the *1049A Operator's Handbook*. Turn the cell OFF and the pump OFF then replace the electrode as described on page 7-4 of the Operator's Handbook.

9.2 Autosampler Needle Seat Cleaning

Highly concentrated samples can leave residues of analytes on the needle seat of the autosampler. This will result in carryover that can linger for many injections. In extreme cases, replace the needle seat using the procedure on page 4-10 of the Autosampler Installation and Maintenance manual. Minor contamination can be rinsed from the needle seat with deionized water. Follow the procedure on page 4-10 to put the autosampler in the CHANGE NEEDLE mode, and remove the inner cabinet as described. Place a dry Kimwipe in the trough between the needle seat and the leak sensor. Rinse the needle seat using a 5-mL syringe filled with deionized water. Repeat the rinse several times using a fresh Kimwipe each time. Replace the inner cabinet and exit the CHANGE NEEDLE mode as described in the manual.

Fuel-contaminated Kimwipes are disposed as fuel-contaminated soft goods in accordance with WIWPS 20-04-18 (Fuel Contaminated Soft Goods after verifying free of hydrazine fuel vapors after checking with a hydrazine fuel vapor monitor as described above. Fuel-contaminated needle seats requiring disposal must be triple rinsed with water or other aqueous solution, the water disposed in accordance with WIWPS 20-04-102 (Aqueous Fuel Contaminated Waste), and the rinsed seat disposed as ordinary trash.

9.3 Column Replacement

As the column ages, the column capacity decreases and retention times also decrease. A new column should be installed when peaks are no longer resolved.

Since new columns are shipped with methanol in them, they must be purged with deionized water before buffer is used. Flush the lines with deionized water (Reservoir C). Turn the pump off and disconnect the old column. Connect the new column. Turn the pump on and pump deionized water through the column for 30 minutes. Switch from the deionized water (Reservoir C) to the eluant reservoir (Reservoir A) and continue to pump through the column for at least 2 hours. Turn the detector cell on and run a standard multiple times.

Used or old columns must be triple rinsed (normal eluant rinses while the column is still installed suffice) then wrapped or bagged and disposed as Fuel Contaminated Soft Goods (20-04-18) because of the porous chromatographic support material inside.

9.4 Other

Procedures for non-routine maintenance are described in the instrument manuals and in the maintenance CD-ROM.

Permanent Deviation Index

Dev. No.	Dev. Sheet	Affected Page(s)	Affected Step(s)	Description	Entered By

Temporary Deviation Index

Dev. No.	Dev. Sheet	Affected Page(s)	Affected Step(s)	Description	Entered By

Attachment A Issued: 07/20/06

Attachment A:
Validation Data and Calibration Data

Shown in the table below, standards independent of the calibration standards containing hydrazine, MMH, and UDMH were analyzed and the data reduced for precision and accuracy statistics.

TABLE I

Fuel Standard	Precision (%RSD)	Accuracy (%)
Hydrazine (1 mg/L)	1.2	99.0
Hydrazine (10 mg/L)	0.40	109
MMH (1 mg/L)	1.9	101
MMH (10 mg/L)	1.6	109
UDMH (10 mg/L)	0.82	99.8

Accuracy is also shown through the analysis of Blind Controls (CAH093 and 1CAH097B). Reference LWO 685164.

Calibration curves generated from duplicate injections of four standards (0.1 mg/L, 1.0 mg/L, 10 mg/L and 100 mg/L) establish linearity from 0.1 mg/L to 100 mg/L with correlation coefficients greater than 0.999 with an injection volume of 10 μL. This data satisfies the requirement outlined in TPS 2-LAB-972609.

Appendix 6-J

WSTF Chain of Custody Form

WSTF CHAIN OF CUSTODY RECORD

Date ____________________ Page ____ of ____

Laboratory / PO #

Analysis Requirements

Return Address for Analytical Reports:

NASA / WSTF / Honeywell
P.O. Box 20

Las Cruces, NM 88004

Attn: ☐ Lori Minnick ☐ Other

____

(505) 524-5119 (505) 524-_________

<u>Special Instructions</u>

Please return coolers and contents of coolers as soon as possible.

Return Address:

NASA / WSTF / Honeywell
Building 120 / Route 70

Las Cruces, NM 88012

Attn: Lori Minnick

Sample No.	Well ID.	# of Containers	Sample Type (Aqueous)	Halogenated Volatile Organics Ice/No Headspace (40 ml Vial) ( )	Volatile Organics Ice/No Headspace (40 ml Vial) ( )	Nitrosamines Ice (1 L Glass Bottle) ( )	RCRA Metals (As, Ba, Cd, Cr, Pb, Hg, Se and Ag) Ice/HNO_3 (500 ml Poly)					Comments

Relinquished By:	Date/Time:	Accepted By:	Date/Time:

WSTF – 381B (02/00)

CHEMISTRY/METALLURGY
LABORATORY WORK ORDER

REQUESTED BY	RECEIVED BY	DATE OF REQUEST	NEED DATE
ORGANIZATION	PHONE	TASK ORDER	PROMISED DATE
SUPERVISOR	DELIVER RESULTS TO/PHONE/MAIL CODE		LW0 NO.

SAMPLE ID

SAMPLE RELINQUISHED BY	DATE/TIME	ACCEPTED BY	DATE/TIME

SERVICES REQUESTED

APPLICABLE DOCUMENTS/SPECS/WJI/TPS

☐ RESULTS BELOW ☐ REFER TO ATTACHED REPORT

ANALYST	REVIEWER	SUPERVISOR

WSTF Form 122 (Rev. 9-95)