GENERAL ELECTRIC

ENERGY SYSTEMS AND
TECHNOLOGY DIVISION

XL-898-90717/P2761

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ADVANCED REACTOR SYSTEMS DEPARTMENT

September 25, 1979

MASTER

DOE/SF/70030--T21

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Nuclear and Magnetic Fusion Division
Department of Energy
San Francisco Operations Office
1333 Broadway
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Records

File 17T03-76 **SF 70030**
8-Z

Dear Mr. Harbison:

SUBJECT: Contract DE-AM03-76SF00893, Task 3C, WPT SG029

Milestone G-6.2, Chemical Cleaning Specification - Few Tube Test Model

This letter transmits subject cleaning specification for the Few Tube Test Model and completes Milestone G-6.2 of SG029.

Mr. J. L. Simpson, San Jose extension 52215, may be contacted for any questions pertaining to this specification.

Very truly yours,

W. V. Leeburn, Manager
Steam Generator Projects

WVL:VH:mcj

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Mr. S. G. Harbison

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September 25, 1979

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CHEMICAL CLEANING SPECIFICATION - FEW TUBE TEST MODEL

L.V. Hampton
J.L. Simpson

September 1979

General Electric Company
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CHEMICAL CLEANING SPECIFICATION - FEW TUBE TEST MODEL

1.0 Objective

The objective of this specification is to provide the Few Tube Test Model (FTTM) operator with the necessary data for a successful waterside chemical cleaning event. It is assumed that only the evaporator section of the system will be cleaned. The cleaning is to proceed until both the fouling product layer and the base metal scale are removed from the 2-1/4Cr-1Mo steel steam generator tubes.

This specification describes the reagents and conditions for post-chemical cleaning passivation of the evaporator tubes.

Ancillary equipment specification, e.g., pumps, heaters, piping, etc., is outside the scope of this document. When actual cleaning is required, these components will be supplied by the contractor.

2.0 Chemical Cleaning Solvent

Based on tests conducted to date of prototypically fouled 2-1/4Cr-1Mo steel steam generator tubes, the solvent ammoniated ethylenediamine tetraacetic acid (EDTA) + citric acid is the prime choice for FTTM application.¹ The solvent offers the best combination of ease of handling, scale removal ability, and low base metal corrosion rate of all candidate solutions investigated. The solvent composition is shown in Table I.

2.1 Solvent Component Characteristics

Of the solvent components, citric acid, hydrazine and ammonium hydroxide are readily soluble in water. The corrosion inhibitor Rodine 33 is not readily soluble and because of its relatively high specific gravity (1.22 @ 16°C), seeks the low point in a water solution. This viscous fluid will dissolve in room temperature water upon vigorous mechanical mixing. (The inhibitor prevents, as completely as possible, the solvent action of the acid cleaning solution on bare metal without significant reduction of the oxide dissolution properties of the solvent.)

The solid EDTA will not dissolve directly into neutral room temperature water. Conversion of EDTA to the ammonium salt form by the addition of ammonium hydroxide is required to make up this solution.

All solutions should be made up in secondary containers and chemically analyzed before insertion into the steam generator. This will avoid any

precipitation due to improper mixing.

2.2 Toxological Data

EDTA and citric acid are not particularly hazardous, however, dust from these compounds can irritate the eyes and throat, hydrazine and ammonium hydroxide are to be handled with extreme caution avoiding the eyes, nose, skin and throat, as these liquids give off very pungent vapors. Use of all the above chemicals is to be restricted to well ventilated areas. Thirty-five percent hydrazine in water should be used as a starting solution.

Toxological data for Rodine 33 are provided by the manufacturer in Appendix I.

All personnel should wear safety glasses, gloves, and protective masks during the mixing operation.

3.0 Flow Conditions

3.1 Flow Circuit

It is assumed that the loop is filled with layup quality water. The flow circuit will consist of a loop containing the evaporator, steam drum, and downcomer line. The solvent should not contact the feedwater line, feedwater pump, feedwater heaters, or the recirculation pump. Check valves should not contact solvent where possible.

Temporary pipework for solvent injection and pumping will be installed at this time. The connection of this piping to the cleaning loop should be made near the evaporator inlet of the flow circuit.

3.2 Loop Checkout

Following completion of the pipework, the boiler water should be circulated and tested to confirm that the chloride concentration is below 1 ppm. The temperature of the water in the loop should be raised by an auxiliary heat source and the vents on the boiler closed as they become free of air.

3.3 Circulation of the Ammoniated EDTA + Citric Acid

The loop pressure should be increased to 50 lb/in². The loop inlet temperature should be increased to 96°C (200°F). (It is assumed that all attempts at minimizing system heat losses have been made at this time.)

The slurry containing the EDTA, citric acid, hydrazine, and Rodine 33, pH adjusted with ammonium hydroxide, will be injected when the loop

temperature stabilizes (chloride content of the slurry will be checked before injection; final solution to be below 1 ppm chloride).

During cleaning, the loop will be adjusted to 0.4 GPM; overflow provisions will be made in advance. The system will be vented regularly during the cleaning operation.

4.0 Control Methods

4.1 Chemical Tests

Chemical analysis is required of the cleaning solution of samples taken from the downcomer line. These chemical tests will be carried out and recorded in a log book of the loop pH, iron, copper and nickel content, total acidity and free acidity. Acid increases will be countered by ammonium hydroxide injections and decreases will be adjusted by additional cleaning solvent injection.

Sampling intervals and immediate analyses will be 1/2 hour after initial injection of cleaning solution, 1-1/2 hours, 2-1/2 hours, and every two hours thereafter. It is imperative that the analytical chemist be in readiness prior to initial injection of the cleaning solution.

When the nickel and copper contents become stable, the fouling product layer on the evaporator tube walls will have been dissolved.

When the iron content becomes stable, cleaning will have been completed. Cleaning can be expected to take up to 12 hours.

4.2 Nondestructive Tests

It is recommended that air-gaging be performed on a tube ID in a region extending from the upper nucleate boiling zone into the film boiling region both before and after the cleaning event.

5.0 Passivation

5.1 Draining

The solvent will be drained by displacement with nitrogen.

5.2 Flushing

After draining the unit, immediate flushing will be accomplished by circulating lay-up water from the feedwater line into the drum, through the downcomer into the evaporator and out the drain line at the evaporator outlet. Flushing will be performed at 8-10 GPM, once-through.

Flushing is to continue until the effluent water is clear and has a

conductivity of 25µmho/cm maximum. Analyze final flush water for EDTA and citric salts. Concentration must be below 10 ppm before complete flushing has been achieved.

5.3 Passivation

Following the flushing operation, the tube surfaces will be passivated by circulating three each solutions containing 500 ppm hydrazine and 40 ppm ammonia. After passivation, the unit will be filled with lay-up quality water.

6.0 References

1. L.V. Hampton, *Fouling and Chemical Cleaning Studies - CRBRP Evaporators-Part II*, GEFR-00449, May 1979.

TABLE I

Solvent Composition* (Weight Percent)

10% EDTA
2% Citric Acid
1% Hydrazine
pH 7 (NH₄OH)
(0.2ml/1 Rodine 33)

*Solvent = water

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

Form Approved
OMB No. 44-R1387

MATERIAL SAFETY DATA SHEET

CONFIDENTIAL
Proprietary Information
Amchem Products, Inc

Required under USDL Safety and Health Regulations for Ship Repairing,
Shipbuilding, and Shipbreaking (29 CFR 1915, 1916, 1917)

SECTION I

MANUFACTURER'S NAME Amchem Products Co., Inc.		EMERGENCY TELEPHONE NO. 1-215-628-1321
ADDRESS (Number, Street, City, State, and ZIP Code) Brookside Ave., Ambler, Pa. 19002		
CHEMICAL NAME AND SYNONYMS	TRADE NAME AND SYNONYMS Rodine 33	
CHEMICAL FAMILY	FORMULA	

SECTION II - HAZARDOUS INGREDIENTS

PAINTS, PRESERVATIVES, & SOLVENTS	%	TLV (Units)	ALLOYS AND METALLIC COATINGS	%	TLV (Units)
PIGMENTS			BASE METAL		
CATALYST			ALLOYS		
VEHICLE			METALLIC COATINGS		
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX		
ADDITIVES			OTHERS		
OTHERS					
HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES				%	TLV (Units)
nitrogen heterocyclic thiourea				26-28	
formalin				17-19	
emulsifiers				26-28	
sulfuric acid				26-28	

SECTION III - PHYSICAL DATA

BOILING POINT (°F.)		SPECIFIC GRAVITY (H ₂ O=1)	1.22 @ 60°F.
VAPOR PRESSURE (mm Hg.)		PERCENT VOLATILE BY VOLUME (%)	
VAPOR DENSITY (AIR=1)		EVAPORATION RATE (_____ = 1)	
SOLUBILITY IN WATER	soluble in aqueous	acid solutions.	
APPEARANCE AND ODOR	brown viscous liquid		

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used)	none	FLAMMABLE LIMITS	Lel	Uel
EXTINGUISHING MEDIA	N. A.			
SPECIAL FIRE FIGHTING PROCEDURES	N. A.			
UNUSUAL FIRE AND EXPLOSION HAZARDS	N. A.			

SECTION V - HEALTH HAZARD DATA	
THRESHOLD LIMIT VALUE	MAE (8 hour weighted average) 3 ppm
EFFECTS OF OVEREXPOSURE	May cause severe burns to skin and mucous membranes
EMERGENCY AND FIRST AID PROCEDURES	
Skin contact - wash thoroughly with soap and water immediately.	
Eye contact - Flush with copious amounts of water. Call a doctor	
Ingestion - Drink milk of magnesia, aluminum hydroxide gel or limewater. Call a doctor	

SECTION VI - REACTIVITY DATA			
STABILITY	UNSTABLE		CONDITIONS TO AVOID
	STABLE	X	
INCOMPATIBILITY (Materials to avoid)			
Strongly alkaline compounds			
HAZARDOUS DECOMPOSITION PRODUCTS			
oxides of sulfur or nitrogen			
HAZARDOUS POLYMERIZATION	MAY OCCUR		CONDITIONS TO AVOID
	WILL NOT OCCUR	X	

SECTION VII - SPILL OR LEAK PROCEDURES	
STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED	
Transfer contents to a clean glass, poly lined or plastic container. Discard broken container and flush away residual matter with water.	
WASTE DISPOSAL METHOD	
Neutralize with lime to pH 7-8	

SECTION VIII - SPECIAL PROTECTION INFORMATION		
RESPIRATORY PROTECTION (Specify type)		
VENTILATION	LOCAL EXHAUST X	SPECIAL
	MECHANICAL (General)	OTHER
PROTECTIVE GLOVES	Rubber	EYE PROTECTION Goggles & face shield
OTHER PROTECTIVE EQUIPMENT Aprons		

SECTION IX - SPECIAL PRECAUTIONS	
PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING	
Store in a cool, dry area. Does not freeze at -20°F.	
OTHER PRECAUTIONS	

TOXICOLOGICAL AND SAFE HANDLING INFORMATION
(For Use by Medical and Safety Personnel)

1. PRODUCT: Rodine® 33
2. MANUFACTURER: Amchem Products, Inc., Ambler, PA 19002
3. STORAGE REQUIREMENTS: Store in a cool, dry area. Do not store Rodine 33 at temperatures below -20°F.
4. PROTECTIVE EQUIPMENT: The use of rubber gloves, aprons, face shields, goggles, etc., is recommended when handling Rodine 33.
5. CARE OF BREAKAGE OR LEAKAGE: Transfer the contents to a clean glass, plastic, or polyethylene lined container. Discard the broken container and flush away any residual materials with plenty of water.
6. FIRST AID:
 - a. Skin Contact: Wash thoroughly with soap and water immediately after contact. Prolonged contact with Rodine 33, especially in hot, humid weather, can cause dermatitis or chemical burns.
 - b. Eye Contact: Flush with copious amounts of water immediately after contact for at least 15 minutes. Call a doctor.
 - c. Ingestion: Drink milk of magnesia, aluminum hydroxide gel, or lime-water followed by several glasses of water. Call a doctor.
7. PROPERTIES:
 - a. State: Rodine 33 is a liquid; its specific gravity at 60°F is 1.22.
 - b. Description: Rodine 33 is an aqueous acid solution containing a nitrogen heterocyclic thiourea, formalin, mineral acids, and emulsifiers.
 - c. Flammability: Rodine 33 is a nonflammable chemical.

Rodine® is a registered Trademark of Amchem Products, Inc., for acid inhibiting chemicals.

DATE: April 1976 (rev.)