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DP-MS-85-151, Rev.

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SRL FILE COPY

A paper presented at the
American Ceramic Society
Chicago, ILL.
April 27, 1986

and for publication in the Proceedings

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ABSTRACT

Candidate materials of construction were evaluated for a facility at the Department of Energy's Savannah River Plant to vitrify high-level radioactive waste. Limited operating experience was available under the corrosive conditions of the complex vitrification process. The objective of the testing program was to provide a high degree of assurance that equipment will meet or exceed design lifetimes. To meet this objective in reasonable time and minimum cost, a program was designed consisting of a combination of coupon immersion and electrochemical laboratory tests and pilot-scale tests.

Stainless steels and nickel-based alloys were tested. Alloys that were most resistant to general and local attack contained nickel, molybdenum (>9%), and chromium (where Cr + Mo > 30%). Alloy C-276 was selected as the reference material for process equipment. Stellite 6 was selected for abrasive service in the presence of formic acid. Alloy 690 and Allcorr were selected for specific applications.

INTRODUCTION

Starting in 1989, the Defense Waste Processing Facility (DWPF) at the Department of Energy's Savannah River Plant (SRP) will begin processing high-level radioactive waste accumulated during the past 30 years.[1] This facility will vitrify high-level radioactive waste to make it immobile. The borosilicate glass product will be encapsulated in stainless steel canisters and stored on plant prior to shipment to a federal repository. The overall process is shown in Figure 1.

Radionuclides are generated in nuclear reactors by irradiating an appropriate target material with neutrons from fission of uranium fuels. The irradiated reactor tubes are then dissolved and chemically separated. The acidic wastes are neutralized with caustic and stored in 1.3 million gallon carbon steel tanks.

The radioactive waste from these processes contains radioactive by-products, nonradioactive materials, processing chemicals, and large amounts of soluble salts. The salts contained in the waste, which are critical for corrosion, are nitrates, nitrites, sulfates, chlorides, fluorides, and phosphates. Of the high-level radioactive waste stored at SRP over the past 30 years, 90% is made up of a concentrated salt solution generated from the neutralization of nitric acid-based process solutions by sodium hydroxide. This concentrated nitrate salt solution contains soluble radioactive strontium and cesium which must be removed before the nonradioactive salts can be further processed for safe disposal. Additions of sodium tetraphenylborate and sodium titanate precipitate these radioactive species. The precipitates are filtered from the salt, washed in the tank farm, slurried and sent to the DWPF for further processing. In the DWPF, they are hydrolyzed by formic acid. During hydrolysis, the radionuclides remain in the aqueous phase. Most of the organic

materials (~90%) form a separate phase which is steam stripped, decanted, and distilled for further decontamination. The resulting distillate will be burned in an incinerator as low activity waste, without burdening the glass melter and melter offgas system. The decontaminated salts can be handled as industrial wastes, and will be converted to a cement-based grout and buried in an on-site land fill.

The remaining 10% of the waste is sludge, formed primarily from the precipitation of hydroxides of iron, aluminum, other transition and alkaline earth metals, actinides, and rare earths. A detailed range of compositions of borosilicate glass product is available, based on these high-level radioactive wastes.[2]

SELECTION CRITERIA FOR MATERIALS OF CONSTRUCTION

The DWPF, like the existing SRP separation facilities, is designed for a minimum of hands-on maintenance. Operations in the radioactive process cells are remotely controlled from outside the radioactive area. Therefore, any process equipment to be repaired or replaced must be first removed from the cell and decontaminated. Thus, equipment installation depends primarily on special flanged and gasketed connections rather than welded joints. These connections are especially sensitive to crevice corrosion.

Most major process equipment has been designed for a service life of twenty years, with a five year life for more easily replaced equipment. The glass melter is a special case and is designed for a minimum of two years of continuous service. Melter material performance is discussed in a companion paper.[3]

In the interest of economy, interchangeability of similar DWPF equipment was maximized. Minimizing the number of alloys has improved quality control, reduced fabrication problems, and often expedited delivery schedules.

IDENTIFICATION OF CORROSION CONDITIONS

It was not possible to predict which type of corrosion would be most important, and which chemical species would dominate corrosion. The several wastes to be processed in the facility contain most of the chemical elements of the periodic table, with concentrations and ratios of the elements shifting between waste types. In most of the existing chemical separation processes at SRP, Type 304L stainless steel is used because the solutions are dominated by the oxidizing species nitric acid. Similarly, in the waste storage tanks, the soluble hydroxide, sodium nitrate and sodium nitrite species dominate the environment, and other anions are either insoluble, or not corrosive because of a pH 12. Other anion species present include oxalates, chlorides, fluorides, sulfates, and phosphates. The gelatinous hydroxide sludge also contains ions of transition metals, such as iron, copper, and mercury, which can accelerate electrochemical corrosion by shifting electrical potentials, or by acting as charge carriers inside of pits and crevices. The process requires reacidification with formic acid, which is a reducing acid, to permit the removal of mercury from the sludge, to permit hydrolysis of the tetraphenyl borate precipitate, and to adjust the redox state of the glass. Sludge acidification typically results in pH 3 and the ionization of several thousand parts per million (ppm) of chloride and fluoride. Process temperatures range from ambient to boiling, and many chemical reactions take place between aqueous and organic, as well as oxidizing and reducing species.

To characterize the various potential locations of corrosion, a chemical engineering computer program was used to trace 172 chemical species through over 1100 process streams.[4] This was further expanded by using as inputs to the program the range of anticipated sludge compositions. This permitted the definition of minimum and maximum concentrations for each of the chemical species in each of the process streams. General operating conditions, including pH and temperature, were determined from process development tests using non-radioactive, synthetic sludge feed.[5,6] For reasons of safety and to preclude possible early failure of Type 304L stainless steel pilot equipment, halides and mercury were generally not added to these synthetic sludges.

This multitude of conditions was simplified by grouping process streams with similar conditions together to form the following "Corrosion Control Zones":

- Standard (Industrial [7] or SRP) Practice
- Glass Frit Handling (slurried nonradioactive glass formers)
- Sludge Receipt and Adjustment Tank (SRAT, i.e. formic acid addition to waste sludge)
- Slurry Mix and Evaporation (SME, i.e. glass frit addition to acidified sludge)
- Precipitate Processing (hydrolysis, distillation, and decanting of tetraphenyl borate salts and sodium titanate)
- Melter Feed Tank and Lines
- Melter
- Melter Off-gas Condensate
- Mercury Recovery, Washing, and Decontamination
- Recycle to Waste Tank Farm (caustic treatment, evaporation, and storage)

A simplified flowsheet summarizing the entire processing and solidification operations is given in Figure 1. Because of the complexity of the reactions occurring in the melter, it was not possible to predict the chemical species and concentrations in the melter offgas condensate. To obtain more realistic process information, a pilot scale melter was operated at the Pacific Northwest Laboratory. The melter feed was prepared using pilot scale equipment, with average and maximum expected contents of mercury and halides. Melter air inleakage was controlled to determine the effect of melter redox on offgas species. It was determined that no measureable amount of mercury was dissolved in the glass produced. Analysis of the off-gas condensate indicated that most of the mercury in the sludge feed came through the melter and was trapped in the condensate. It was further determined that the mercury in the offgas system was present in metallic, mercurous, and mercuric oxidation states, with the fraction as mercuric ion varying between one-forth for relatively reducing conditions to three-forths for relatively oxidizing conditions. Analyses of the condensate were used as the basis for the composition and pH of synthetic solutions used for bench top corrosion testing.

Mercury recovery, washing, and decontamination by distillation will be conducted in glass equipment using nitric acid. This is considered standard practice, and was not tested. In the absence of process upsets, the incineration of organic from the precipitate processing is also considered to be standard practice and will not be discussed here.

CHARACTERISTICS OF CORROSION TEST METHODS

A multitude of standardized test methods have evolved for materials selection for the chemical process industries.[8-10] Generally these can be divided into:

- Alloy coupon tests in general or specialized laboratory equipment
- Electrochemical tests with electrical probes
- Pilot scale operations in equipment constructed of candidate materials
- Operating experience with production equipment

The characteristics of test methods used are summarized in Table 1. Because of the uniqueness of the process, limited production experience is available. Materials for processes involving standard industrial solutions, such as support facilities outside the high radiation fields of the DWPF, or process supply and electrical connections inside of the radiation control areas, were selected based upon SRP or industrial standard practice (e.g. Reference 7). These did not require testing.

Laboratory coupon tests are the least expensive and most easily conducted. Typically, results are available about two weeks after the start of testing. However for some types of attack, such as chloride stress corrosion, the times must be on the order of months to permit sufficient time for crack initiation. The tests are easy to conduct as a battery of simultaneous tests, and are commonly used to select from available alloys. They are also versatile enough to be used for the determination of the mode of attack, whether it is general corrosion, pitting, crevice attack, galvanic corrosion, or chloride stress corrosion cracking. They are also applicable to the determination of the critical chemical species and the critical concentration of corrosive species.

They are very effective when applied to complex situations, since a large number of tests can be conducted in a limited amount of space. Through the use of quartz or alumina tubes and crucibles, or specialized equipment, the method can be applied to high temperature corrosion. The credibility of the test depends upon how close the test is able to simulate actual operating conditions. Incorrect temperatures or concentrations of minor chemical species can

lead to erroneous results in this, as in any of the other test methods. Where possible, actual process solutions or commercial chemicals representative of the process should be used, rather than analytical reagents. Bench top tests can be used to duplicate normal process cycles, but it is difficult for these small tests to duplicate the range of conditions experienced in actual process equipment cased by mixed phase flows, temperature gradients, etc.

Electrochemical tests have many of the same constraints as lab coupon tests. Their major advantage is relatively quick results, at costs comparable to those of coupon tests. They are most applicable where the alloy has sufficient resistance to the test solution to avoid high general corrosion rates, or is suspected of being susceptable to localized attack. In relatively simple process solutions, the method is effective in determining the critical corrosive species and critical concentration. For well characterized processes and process solutions, the test is conservative if it shows no evidence of localized corrosion modes. High temperature tests are only available using special probes and custom equipment.

Pilot scale tests are highly dependent upon the resources available. If equipment is being designed for process development, then very representative corrosion tests can be conducted by building the equipment of candidate alloys based upon coupon or electrochemical tests. If existing equipment is being used which is constructed of materials which are known to be attacked by process solutions, then operating personnel are reluctant to use complete simulations of process solutions, and very limited tests are possible. Pilot scale tests which are conducted with incomplete simulations of process chemicals, or outside of actual operating conditions, can be very misleading. Alternatively, pilot scale tests with actual process solutions, or chemically complete synthetic solutions are the closest possible simulation to exposure in actual

process equipment. The ability of pilot scale equipment to realistically simulate actual process gradients, flows, process cycles, and mixed phase effects makes it unique in the ability to determine what actual corrosion conditions will be.

APPLICATION OF TEST METHODS AND DISCUSSION OF TEST RESULTS

Detailed corrosion test methods and results for the SRAT, SME, precipitate processing, melter, and melter offgas system have been discussed.[3,5,6] They will therefore only be discussed as illustrations of the test methods. Compositions of alloys tested are given in Table 2. Initially, Type 304L stainless steel was selected as the reference material of construction for DWPF because of its general corrosion resistance, and operating experience with it in Reactor and Chemical Separations areas. Later, process development testing indicated that halides would not be totally removed during in-tank washing of sludge or precipitate. Coupon testing indicated that Type 304L and 316L stainless steels had acceptable general corrosion rates in formic acid process solutions, but had unacceptable resistance to chloride stress corrosion cracking (SCC).[5] Types 304L and 316L cracked in dilute formic acid with chloride concentrations as low as 100 parts per million (ppm). This was subsequently confirmed when a 304L steam coil installed in a pilot scale high level cell SRAT/SME vessel failed during its first cycle with actual waste.

Since Alloy 20 was known to be significantly more resistant to chloride stress corrosion cracking, readily available and economical, it was selected as the initial reference material for process equipment operating in the melter feed preparation and melter off-gas condensate areas. Alloy C-276, which is still more highly alloyed, was selected as a backup material. Alloy 20 and Alloy C-276 were then tested in simulated SRAT and SME solutions. Results

revealed that Alloy 20 experienced exceptionally severe pitting and crevice corrosion, and is therefore not sufficiently resistant to SRAT and SME process exposure. These same tests indicated that Alloy C-276 was a satisfactory material.[6]

Localized attack was especially severe on Alloy 20, with directional pitting producing holes completely through 1/4 inch samples in 72 hours. This also confirmed the directional pitting on Alloy 20 coupons which had been exposed to off-gas condensate in pilot plant melter tests.[6] The severe corrosion of Alloy 20 has been attributed to an interaction between copper contained in the alloy, and mercury in the solutions.[11]

Coupon corrosion tests of abrasion resistant materials for centrifugal transfer pumps indicated no significant attack of the Abex Co. SPA (TM) alloy in the SRAT/SME environment. However, pitting was observed on this alloy in offgas condensate solutions. Since SPA contains copper, its behavior in SRAT/SME solutions was suspect. A clue to its behavior was found in electrochemical testing in the offgas condensate. In these solutions, the SPA is anodic by about 0.4 volts with respect to Alloy C-276. Because alloys are normally electrically isolated during coupon tests, the initial SPA tests were not representative of actual production equipment, where the pump and tank material are bolted together. When SPA was galvanically coupled to Alloy C-276 in pilot scale SME tests, the SPA cracked after 5 process cycles. The galvanically coupled effect was then confirmed in coupon tests. Haynes Stellite 6 (TM), a cobalt based wear-resistant alloy was tested in galvaniccoupled coupon tests with Alloy C-276 and found to be satisfactory.

Pilot scale abrasion testing indicated minimal wear in a Stellite 6 centrifugal pump, after 2000 hours operation with synthetic melter feed. Because

of lower relative velocities, heavy wall C-276 tubing is sufficiently wear resistant for transfer piping.[12,13]

Candidate material verifications for SRAT and SME were performed in a special 35-gallon vessel constructed of Alloy C-276. This vessel had a flanged top and condenser connection, and could be disassembled for inspection of heating and cooling coils, agitator and baffles, and other interior details, including welds. Corrosion coupon support racks were installed inside the vessel and condenser. This permitted quantitative measurement of the Alloy C-276 corrosion rate, as well as the galvanically coupled tests just discussed. An Alloy C-276 offgas system with a Teledyne Allvac Co. Allcorr (TM) quencher was built to confirm the behavior of these alloys under more realistic conditions. It was dismantled and examined after six weeks operation with the maximum anticipated concentration of halides and mercury. General corrosion rates were acceptably low, with no evidence of localized attack.

Twelve nickel base alloys were exposed to the synthetic off-gas solution in laboratory bench top tests. Of these, only Alloy C-276 and Allcorr were resistant to both general and localized attack. Subsequent coupon testing has indicated both temperature and mercuric chloride thresholds for pitting in C-276. Table 3 indicates the thresholds, and also shows a strong adverse interaction with small amounts of nitric acid. Corrosion coupons exposed during pilot scale runs indicated the off-gas condensate to be extremely corrosive, and confirmed that highly alloyed nickel-based alloys are required.

The only normal requirement for dilute solutions such as melter offgas condensate transferred to the waste tank farm is that the free hydroxide concentration exceed 0.01 molar (pH greater than 12). As is shown in Table 4, this is not adequate for DWPF recycle. X-ray diffraction of the precipitate at pH 12 revealed incomplete conversion of mercurous chloride after 4 hours. It

is believed that this contributed to the attack. Similar x-ray diffraction of offgas indicated that a minimum of 0.3 molar excess hydroxide is necessary to achieve complete conversion in less than 1 hour. Subsequent testing was conducted at 0.5 molar free hydroxide (pH approximately 13). At this level, satisfactory protection of waste tanks and waste evaporators is predicted.

Electrochemical tests were not used extensively because of a tendency for mercury to deposit on cathodes. As a further complication, formic acid reacts with calomel in saturated calomel electrodes, requiring the use of double junction or other reference electrodes.

SUMMARY AND CONCLUSIONS

Extensive bench top laboratory and pilot scale tests have evaluated candidate alloys for construction of the Defense Waste Processing Facility. Type 304L stainless steel is recommended for general service piping and equipment, but is unacceptable for use with solutions derived from waste processing. Since Alloy 20 suffered from localized attack in all the simulated tests, as well as the pilot plant melter tests, it was dropped as the reference material for SRAT, SME, and the melter offgas system. Alloy C-276 was selected as the reference material for these process zones and precipitate processing because of its superior corrosion resistance. Stellite 6 appears satisfactory for abrasive service. Allcorr has been satisfactory in coupon and pilot scale tests of the melter quencher.

Each of the available corrosion test methods has particular advantages and disadvantages. For complex processes with limited operating experience where long operating lives are needed, pilot scale tests are required with as complete a simulation of the chemical environment as possible. The use of coupon

and electrochemical tests permits the expeditious determination of corrosion modes, and critical chemical concentrations at minimum expense.

ACKNOWLEDGMENTS

The authors wish to thank A. R. Greening, R. S. Ondrejcin, and M. K. Carlson for help in designing the experiments. The assistance of S. V. Fox in conducting the bench-top tests is most appreciated, as is that of R. W. Goles and G. J. Sevigny of the Pacific Northwest Laboratory and J. W. Dunaway, S. E. Eissenberg, and B. A. Hacker of the Savannah River Laboratory for pilot scale testing.

The information contained in this article was developed during the course of work under Contract No. DE-AC09-76SR00001 with the U.S. Department of Energy.

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TABLE 1

Typical Characteristics of DWPF Corrosion Test Methods

	Lab Coupon Tests	Electrochemical	Pilot Scale
Time Per Test	2 Weeks	1 Day	0.1-3 Years
Cost Per Sample	$250-350	$500-1,000	$500-100,000
Alloy Selection	Yes	Yes	Limited
Modes of Attack	Screening	Electrochemical Only	All Modes
Identify Critical Chemical Species	Complex Matrix	Simple Matrix	No
Identify Critical Concentrations	Yes	Yes	No
Quantitative Corrosion Rates	Yes	Limited	Yes
High Temperature	Limited	No	Realistic
Credibility	Interpretation and Experience Required	Conservative for Some Modes of Attack	Most Credible
Process Solution	Synthetic or Actual	Synthetic or Actual	Synthetic or Actual
Process Cycles	Limited	No Cycles	Realistic
Special Uses and DWPF Applications	Relative Ranking of Alloy Abrasion Resistance, Melter Quencher	Verification of Abscence of Local Corrosion Modes	Identification and Concentration of Melter Reaction Products, Pump and Pipe Abrasion Tests
DWPF Equipment Saved	Imbedded Piping, Processing Vessels, Offgas Pumps Recycle Waste Tanks	Pumps in SRAT, SME	Pumps in SRAT/SME, Quencher, Melter Parts, Melter Offgas System

TABLE 2

Chemical Composition of Alloys Tested, wt %

Alloy	Ni	Cr	Mo	Cu	Fe	Other
304L	10.0	19.0	-	-	69	
316L	12.0	17.0	2.5	-	66	
Sanicro 28	31.0	27.0	3.5	1.0	38	
800	32.5	21.0	-	0.5	44	
Alloy 20	34.0	20.0	2.5	3.5	40	Cb-1
20Mo-4	37.5	23.5	4.0	1.0	33	Cb-0.25
Hastelloy G-30	43.0	29.5	5.5	2.0	15	Co-1, W-2.5, Cb-0.5
X	48.0	22.0	9.0	-	18.5	Co-1.5
HR13	49.0	25.5	5.7	0.9	16	Ti-1.3
Inconel 617	52.0	22.0	9.0	0.2	1.5	Co-12.5, Al-1.2
ALLCORR	56.5	31.0	10.0	-	-	W-2, Ti-0.25, Al-0.25
C-276	57.5	15.5	16.0	-	5.5	W-3.5, Co-2
Hastelloy C-22	60.0	21.0	13.0	-	3.0	W-3
Inconel 690	62.0	29.0	-	0.3	9.0	
625	64.0	21.5	9.0	-	3.0	Cb-3.5
Hastelloy C-4	65.0	16.0	15.5	-	1.5	Co-1.5
600	76.0	15.5	-	0.5	8.0	W-5, C-1, balance Co
Stellite 6		26.0				

Trademark	Product Of
ALLCORR	Teledyne Allvac
Hastelloy C-4	Cabot Corporation
Hastelloy C-22	Cabot Corporation
Hastelloy G-30	Cabot Corporation
HR13	Sumitamo Metal Industries, Limited
Inconel 617	Inco Alloys International
Inconel 690	Inco Alloys International
Sanicro 28	Sandvik, Incorporated
20Mo-4	Carpenter Technology Company
Stellite 6	Cabot Corporation

TABLE 3

Crevice Attack by Mercury Compounds and Halides on Alloy C-276 in Simulated Offgas Condensate Solutions

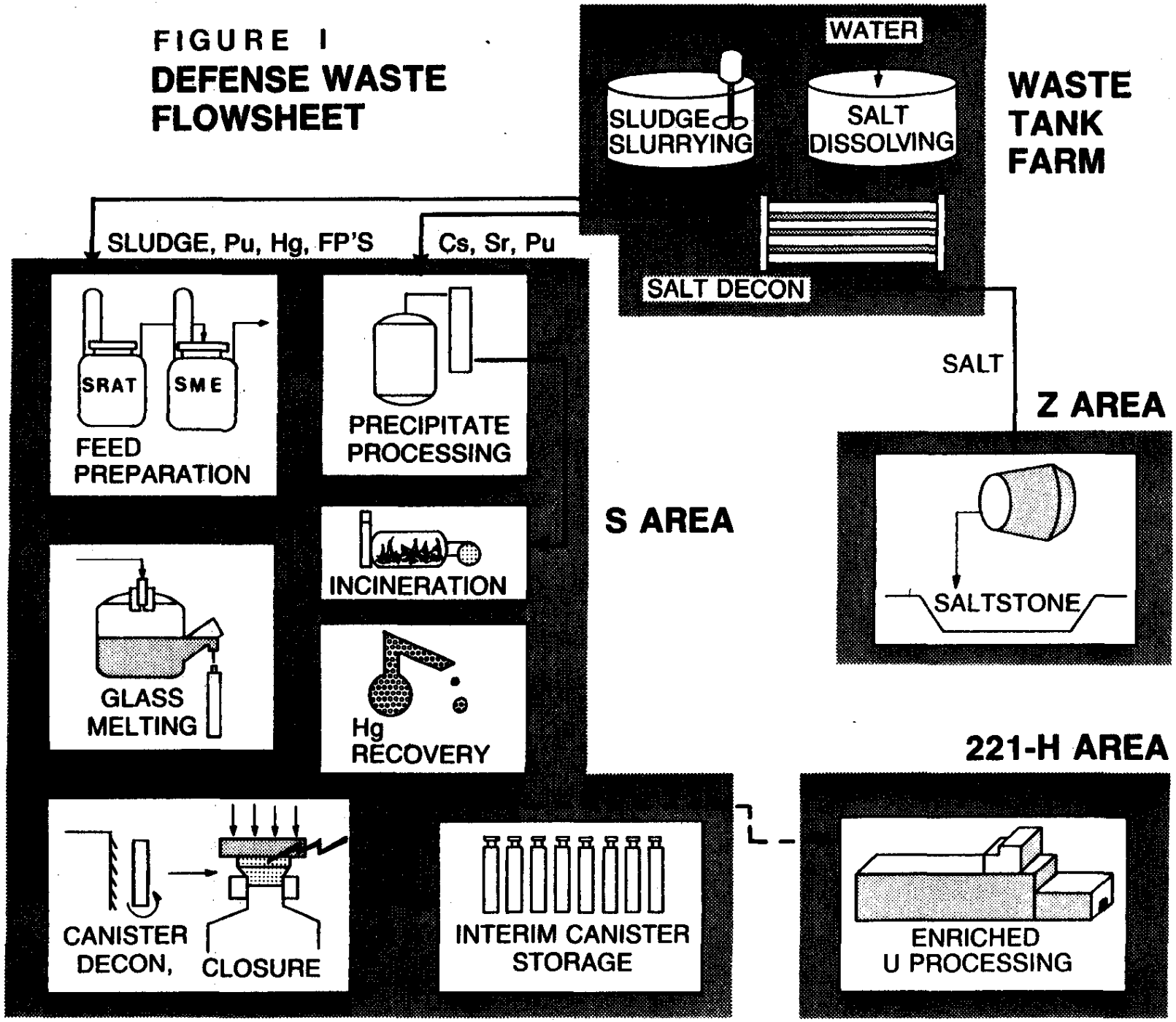
60°C Results	OK	OK	OK	OK	OK	OK	OK	OK	OK
90°C Results	C-1	C-15	OK	C-2	C-15	OK	OK	OK	OK
pH	1.6	1.6	1.6	1.6	1.6	2.2	2.2	2.2	2.2
Chloride, % (as NaCl)	2.01	2.01	2.01	2.01	2.01	2.01	2.01	0.25	0.25
Fluoride, % (as NaF)	0.23	0.23	0.23	0.23	0.23	0.23	0.23	0.03	0.03
Nitrate, % (as NaNO3)	---	0.10	---	---	0.10	---	---	---	0.10
Hg (as $HgCl_2$)	0.45	0.45	---	---	---	---	---	0.10	0.10
Hg (as Hg_2Cl_2)	---	---	0.45	---	---	---	---	---	---
Hg (as 50/50 $Hg_2Cl_2/HgCl_2$)	---	---	---	0.45	0.45	---	---	---	---
Hg (as 90/10 $Hg_2Cl_2/HgCl_2$)	---	---	---	---	---	0.45	---	---	---
Hg (as 99/1 $Hg_2Cl_2/HgCl_2$)	---	---	---	---	---	---	0.45	---	---

C-XX = XX mils crevice corrosion during 14 day test.
OK = Less than 5 mils per year general corrosion, with no visible attack in 14 day test.
pH adjusted with sulfuric acid.

TABLE 4

Corrosion Rates of Caustic Treated Melter Offgas Solution on Waste Farm Tanks (Carbon Steel) and Waste Evaporators (304L)

Alloy	Temp. °C	Free Hydroxide Molar	General Corrosion Mils per year	Localized Corrosion Mils in 14 days
C. Steel	60	0.01	7.3-29	18
C. Steel	90	0.01	2.9-44	Intergranular
C. Steel	90	0.5	0.3-1.4	No Visible Attack
C. Steel	100	0.5	0.8-5	No Visible Attack
304L	60	0.01	0.1	No Visible Attack
304L	90	0.01	0.0-4.4	40
304L	90	0.5	0.1	No Visible Attack
304L	100	0.5	0.1	No Visible Attack
304L	185	0.5	6.6	No Visible Attack

FIGURE I

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