SELECTION OF PROCESS PARAMETERS
FOR SODIUM REMOVAL VIA THE
WATER VAPOR NITROGEN PROCESS

by

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July 1976

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PRELIMINARY DOCUMENT

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I. INTRODUCTION

A. PURPOSE

The purpose of this document is to state the background on the technical decisions that went into the selection of the water-vapor-nitrogen (WVN) processing parameters as shown in standard RDT SDT F5-9.⁽¹⁾ Information is included which will guide the facility designers and operators in process applications.

This short summary paper presents the rationale for selected standard parameters. This paper shows where variations in the standard process may be made within the limits of experimental work. The WVN process has not been entirely optimized; there are areas where improvements can be made if additional development is undertaken. For those interested in details, several specific references on experiments are included on each topic.

B. SUMMARY

For the vapor phase of the WVN process the 160 to 190°F temperature limit is shown to be well within the 145 to 208°F minimum/maximum range. Decreasing process time by expansion of the temperature range is not expected to aid processing. The most productive area for improvement would be an increase in the water vapor concentration above the present 5% level. However, this would require confirmatory testing before approved use.

The rinsing process was shown to be mainly controlled by component crevice geometry. Improvements in rinse time may be made by increasing the water temperature, but the concern over the caustic stress corrosion cracking will tend to limit the available increase. Although directed jets or sprays of rinse flows was not recommended, methods were suggested for conserving rinse water.

Drying (as well as heating and cooling) of components was again shown to be constrained mostly by individual geometry and not processing parameters. A gas only, vacuum only, or a combination of the two modes were shown to be generally accepted methods. The hot gas only mode was recommended for its simplicity.

II. VAPOR PROCESSING

A. HISTORIC EVOLUTION

Theory

In reviewing the conditions necessary for a water vapor reaction with sodium, it is instructive to examine the temperature-concentration diagram shown in Figure 1.

To maintain a gaseous solution of a liquid, its partial pressure in the gas phase must be less than its vapor pressure at a given temperature. Therefore, a homogeneous phase will result only with the concentration and temperature above the line AB; below AB a two phase "rain" or "fog" situation will exist. The region ABC will then be the atomized water system which will be discussed separately.

If, at a given temperature, the partial pressure of water in the gas phase is less than the vapor pressure of water over a caustic solution, the transfer of water will be from the liquid to the gas phase which is just the opposite of what is desired and is illustrated by line DE. In order to provide water to react the sodium, the temperature and composition must then be to the right of DE.

Two other restraints are also indicated. The first restraint is a minimum temperature. If the reaction is carried out below ambient, then a cooling or refrigeration system is needed. Further, the maximum water concentration is rather low and, since reaction rate generally increases with both temperature and concentration, low process temperatures seemed unwarranted. Therefore, ambient temperature was selected as the lower processing limit (line FA on Figure 1). The second constraint was the upper temperature limit. It appeared prudent to stay below the melting point of sodium (line GH on Figure 1) to prevent changes in the metal configuration during processing. This also allowed very high water concentrations to be reached. Therefore, the values of the temperature and concentration parameters should be maintained within the area GHAFDG in Figure 1 for the gas phase of WVN.

FIGURE 1. WVN Phase Diagram

Water Mist

During 1973 a number of experiments were carried out in plastic bags and glass columns using the atomized water process.⁽²⁾ The initial tests had resulted in high temperature spikes and generally unstable reactions while the latter tests were better controlled to prevent direct impingement of the atomized water on the test pieces. However, unstable reactions still occurred because of condensing water droplets falling onto large sodium surfaces. The results are summarized in Table 1. This led to the conclusion that wall heaters should be used to prevent condensation.

In identical comparison runs between the atomized water and WVN processes, the WVN showed better control and faster processing. Therefore, the atomized water process was abandoned in favor of WVN.

Window

The previously mentioned area in Figure 1 was explored by a number of test runs in the Sodium Removal Development Apparatus (SRDA) using mostly 4-in. diameter, nondrainable pools of sodium. The following criterion for the

TABLE 1. Comparison of the Atomized Water and the Water Vapor Process

Run Number	FEED				RECIRCULATION			RESULTS			
	H ₂ (g) (ℓ/hr)	H ₂ O () (ℓ/hr)	H ₂ O/N ₂ Mole (Percent)	Temp (°F)	Total Flow ℓ/hr	H ₂ O ℓ/hr	H ₂ O/N ₂ Mole (Percent)	Run Time (hr)	Sodium-Wet Surface hr to Remove	3-in. long 13-mils gap (Estimated Percent Na Removed)	5g Na Estimated Reacted
ATOMIZED WATER PROCESS											
1	660	0.492 (liq)	48	RT	2220	0.033	6	48	2	60% in 48 hr 0.02 in./hr	60 mils depth in 48 hr
2	660	0.732 (liq)	58	RT	8880	0.087	16	25	2	22% in 25 hr	Spike >500°F Reaction incomplete
3	660	0.732 (liq)	58	RT	8880	0.087	16	56	3	82% in 56 hr 0.02 in./hr	Spike >500°F Reaction incomplete
WATER-VAPOR-NITROGEN PROCESS											
1 concurrent with Run 2 above	1080	54 (gas)	5(a)	150- 170	0	0	0	25	3	75% in 25 hr 0.05 in./hr	Reaction complete
2 concurrent with Run 3 above	1080	54 (gas)	5(a)	150- 170	0	0	0	56	4	100% in 56 hr; 70.03 in./hr	Reaction complete

a. Mole % = vol %

success of a run was selected: If the reaction was completed without a temperature excursion (or spike) over 250°F, the run was successful. The 250°F limit was selected because that was expected to be below the minimum temperature needed to initiate caustic stress corrosion cracking in stainless steel.

A few dozen preliminary runs showed that the area ABCDE of Figure 2 always gave uncontrolled and unsuccessful runs. The spikes were often above 500°F for tens of minutes or longer. However, within the area AEF of Figure 2, the runs were usually successful although there were occasional temperature excursions over 250°F but only for a few minutes. Some runs were conducted at temperatures as low as 100°F and were successful, but the reaction rate was found to be very slow. Some of these typical data are shown as Figure 3 and Tables 2 and 3.⁽³⁾

At this point several conclusions were drawn. Among them, that a successful operating envelope of parameters had been found and that changes in the temperature or concentration during the reaction almost always upset the reacting mass and led to spikes.

HEDL 7606-116.1

FIGURE 2. SRDA Bulk Sodium Water Vapor Reaction - Steady State and Specified Operating Conditions

HEDL 7606-116.2

FIGURE 3. SRDA Bulk Sodium Water Vapor Reaction - Steady State and Specified Operating Conditions

TABLE 2. SRDA Run Results Within Stable Envelope of Figure 1

Run Number	OPERATING CONDITIONS			RESULTS		POOL CONFIGURATION	
	Conclusion H ₂ O (g) % Percent	TEMPERATURE		TIME		Sodium Depth, in.	Sodium Area, in. ²
		Initial N ₂ Inlet °F	Maximum Sodium, °F	To Complete Reaction, hr	Above 250°F, hr		
3	7.5	170	<250	20	0	0.5	14
5	12	150	<250	19	0	0.5	14
8	6.0	170	<250	48	0	1	14
13	15	150	<250	57	0	0.5	14
17	7.5	150	<250	60	0	0.5	14
29	5.0	120	<250	41	0	0.5	14
32	5.0	160	<250	75	0	0.5	14
35	15	140	<250	17	0	0.5	14
36	5.0	140	<250	63	0	0.5	14
37	10	130	<250	68	0	0.5	14
39	15	160	<250	26	0	0.5	14
41	10	140	285	32	0.16	0.5	14
54	10	150	<250	107	0	2	14
55	10	150	<250	60	0	1	14
56	10	150	<250	82	0	2	14
59	10	150	<250	82	0	2	14

TABLE 3. SRDA Run Results Within Specified Operating Limits
(7.5-15% H₂O, 140-160°F) Inlet

Run Number	OPERATING CONDITIONS			RESULTS		POOL CONFIGURATION	
	Conclusion H ₂ O (g) % Percent	TEMPERATURE		TIME		Sodium Depth, in.	Sodium Area, in. ²
		Initial N ₂ Inlet °F	Maximum Sodium, °F	To Complete Reaction, hr	Above 250°F, hr		
5	12	150	<250	19	0	0.5	14
13	15	150	<250	57	0	1	14
35	15	140	<250	17	0	0.5	14
39	15	160	<250	26	0	0.5	14
41	10	140	285	32	0.16	0.5	14
54	10	150	<250	107	0	2	14
55	10	150	<250	60	0	1	14
56	10	150	<250	82	0	2	14
59	10	150	<250	82	0	2	14

More runs were made and the "stable" area was cropped to HIJH⁽⁴⁾ (Figure 2). The limits or "window" for the process was then formally set as area KLMNK of Figure 2 which was $150 \pm 10^\circ$ and $7.5 \pm 2.5\%$ water. The reaction rate was also approximated at 0.02 in./hr under these conditions,⁽³⁾ see Figure 4.

It was now recognized that some of the unsuccessful runs had probably been caused by the nonwetting condition of the pools which was an experimental anomaly. That is, the sodium had been cast into a stainless steel container, but not heated high enough to wet the steel. During the reaction, the caustic solution would creep between the steel and the sodium. When the disc of sodium floated, the reaction surface was greatly increased, the system then went unstable and a spike resulted. To prevent this, all pots were sodium wetted at 1050°F for a minimum of 48 hr after they had been filled with sodium.

This then was the origin of the "window" concept and was essentially a first examination of the problem. The size of the undrained pools for these tests was 1, 2, and 4-in. in diameter by 1/4 to 1-in. deep. A continuing test campaign was started using larger diameter pots for "scale up" purposes.

FIGURE 4. Reaction Rate of Sodium - Water Vapor Within Specified Operating Limits of 7.5-15% H₂O and 140-160°F Inlet

It was soon discovered that larger pools spiked more often than smaller ones even though they were processed within the window. A few runs at other temperatures and concentrations showed that the propensity to spike was higher outside the window area. A great deal of effort was spent analyzing the thermal characteristics of these pools and comparing them to hardware of various sizes.⁽⁵⁻⁷⁾ It was eventually found that pools were not appropriate to test since few existed in real hardware and the thermal analogue of large pools never exists in real reactor components (see Figure 5). That is, when more sodium surface is available there is also more steel cross section to carry the heat away. Nevertheless, it was considered appropriate to solve the "pool problem".

Programmed Feed

During mid-1974 a test program was initiated to determine the exact chemistry of the system. This was done with small beakers of sodium in 4-in. diameter glass pipes. The apparatus eventually allowed the separation and rate characterization of the two principal reactions, "condensation" and "consumption" as shown below:

FIGURE 5. The Maximum Expected Sodium Temperatures for Various Pot Diameters and Wall Thicknesses

These reactions allow observation and quantification of many interesting and useful phenomena that had previously been either suspected or unknown. Perhaps the most important factor was the high precision and very small concentration at which the hydrogen product gas could be measured. Where previously only overall average reaction rates were determined, the sodium consumption reaction could now be monitored on a continuing basis with minute-to-minute variations in the reaction clearly detectable.

It was determined that during the initial hour or so of pool reaction the rate was very rapid and then decreased. It usually continued stably for some time (many tens of hours), usually until the sodium was consumed to the point that the available reaction area started to significantly decrease. Since the window concept calls for a constant feed rate it was clear that an inventory of unreacted water could accumulate if condensation was greater than consumption. This situation could eventually lead to a spike.

The concept of programmed feed then evolved as a method of decreasing the water feed in anticipation of the expected changes in the reaction rate.⁽⁸⁾ This would give a more nearly constant water inventory resulting in a more stable reaction system and fewer spikes. This is illustrated in Figure 6 which illustrates that the water inventory will be fairly constant until the end of the reaction. At this point it is of no particular concern that a theoretical spike may occur since the remaining sodium is so small compared to the solution that a temperature rise would be very modest even with the instantaneous and complete consumption of the sodium inventory. This was observed to be the case experimentally.

The results of SRDA test using the programmed feed on 8- and 12-in. diameter pots were encouraging and were significantly better than had been obtained with constant feed conditions on similar size pools even though there were still some spikes. It was determined that different programs might be required for different configurations and that practice on these various configurations would be required to successfully apply these on a large scale. It was also determined that there could be difficulties in getting an exact characterization on real components since the degree of sodium draining could vary in ways unknown to the cleaning operators. Thus, the programmed feed formed a useful, but short duration, stepping stone in the total WVN development.

FIGURE 6. Hypothetical Programmed Feed Reaction History

Feedback Control

During late 1974 the work in the glass columns was very productive. As had been suspected from theoretical considerations, the reaction rate was characterized as a power function of the concentration and the formula was experimentally determined to be:⁽⁹⁾

$$\text{rate } \left(\frac{\text{in.}}{\text{hr}} \right) = 0.0018 (\% \text{ H}_2\text{O})^{1.2} = \frac{\text{in.}^3}{\text{hr-in.}^2}$$

It was also found that the temperature dependency was very slight. This is because the driving force is the difference in partial pressure of water between the gas phase and the caustic phase. As long as the processing condition is above the line AB in Figure 1, the partial pressure in the gas is independent of temperature and the partial pressure of the liquid (line DE) is seen to be only weakly dependent on temperature. However, the dependency is negative, that is raising the temperature decreases the reaction rate.

A very important phenomenon had also been observed. Spikes during a run were always preceded by oscillation in the hydrogen output of the reacting pool.⁽⁹⁾ This was confirmed on all glass pipe runs by reexamination of the records. Figure 7 illustrates this for a typical run. It also seemed to be true on all SRDA runs. Here, the record was not complete because some of the early instrumentation was not capable of detecting this phenomena. Confirmatory runs made in the glass pipe showed that when the hydrogen output started to oscillate that if the process temperature was raised the usually inevitable spike was prevented. Development runs in SRDA showed that the technique was fully applicable on a larger scale.^(10,11) In fact, since the initial development was conducted the method, when properly applied in a timely manner, has never failed to stabilize an undrained sodium pool.⁽¹²⁾ This has been continually demonstrated in SRDA and AMCF.

The criterion for success was also tightened at this point. Not only did the temperature have to stay below 250°F but there could be no significant uncontrolled oscillations in the temperature or hydrogen record. This new criteria would have changed the box score on many previously "successful" runs to "unsuccessful."

FIGURE 7. Typical Hydrogen and Temperature History of Pot Run

These runs showed that at least a 5 to 7°F increase was necessary to effect a change in the reacting system and that 10°F seemed to always effectively accomplish the change. Several changes up through the temperature range from 160 to 190°F were all effective.

This completes the summary of pertinent points in the development of the WVN Feedback Control concept and leads to illumination of the present control criteria as given in RDT F5-9.⁽¹⁾ This standard calls for the component to be brought to a constant temperature of 160°F. Steam is then admitted to bring the gas concentration up to 5% where it is held. If instabilities are indicated by oscillations in the hydrogen record the process temperature is increased by 10° to dampen and eliminate the oscillation. The higher temperature is maintained throughout the remainder of the process unless further increases are required because of more instabilities. When the reaction is complete the steam concentration is raised for a time as a double check. This completes the gas phase of the process.

B. PRESENT PARAMETERS

The values of various parameters in RDT Standard F5-9, Table 1, herein reproduced as Table 4, are justified in the following subsections.

Equipment

A recirculating gas system was selected to conserve reactants and to give clear information about the progress of the reaction. An engineering judgment was made that the vessel volume should be changed at least every 5 min to maintain the well-stirred criterion.

Instrument requirements (as far as possible) were selected to utilize off-the-shelf equipment normally available to the chemical process industry.

Procedure

Experience has shown that minute attention to the details of pre-processing, especially as regards draining, frost, possible plugs, reaction time, etc. will greatly facilitate the overall cleaning operation.

TABLE 4. WVN Process Variables and Control Criteria

Unit Operation/ Media	Preprocess	Start Up	Steady State	Reaction Termination	Rinse Wash	Drying	Allowable Variation
<u>System Feed</u>							
Temp °F	70 to 400	160	160 to 190	m/nc	170 to 190	200 to 220	±3
Temp °C	21 to 204	71	71 to 88	m/nc	77 to 88	93 to 104	±2
N ₂ (vol.%)	100	99	nm	nm	nm	100	±5
H ₂ O (vol.%)	0.005	1	5 to 10	15	100 (liq)	-76 dp	--
H ₂ (vol.%)	nm	nm	nm	nm	nm	--	--
O ₂ (vol.%)	nm	nm	nm	nm	nm	--	--
<u>System Discharge</u>							
Temp °F	m/nc	m/nc	m/nc	≤190	170 to 190	230 to 250	±3
Temp °C	m/nc	m/nc	m/nc	≤88	m/nc	110 to 121	±2
N ₂ (vol.%)	100	nm	nm	nm	nm	100	--
H ₂ O (vol.%)	≤-76 dp	nm	nm	nm	100 (liq)	<-55 dp	--
H ₂ (vol.%)	<4	<4	<4	<0.005	m/nc	nm	--
O ₂ (vol.%)	<2	<2	<2	<2	nm	nm	--

nm = not monitored

m/nc = monitored, not controlled

dp = dew point, °F

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III

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Inerting is required to decrease the oxygen level below 4% as this concentration will not support hydrogen combustion under any circumstances. Because safety considerations are very conservative, a 2% level is shown in the Standard.⁽¹⁾ As a matter of practicality, the actual concentration will be much lower. The highly sensitive hydrogen meter, upon which much depends, must read zero before steaming is started. And, since the thermal conductivity types will also detect other gases (although not to the very low concentration as hydrogen) these gases must be at low levels in order to zero the meters. Therefore, total gas impurities in the nitrogen will probably be less than 50 ppm before steaming is initiated.

The upper limit of 4% hydrogen is based upon a misunderstanding. A 4 to 5% hydrogen mixture in air will be flammable (but not explosive), but hydrogen in any concentration in nitrogen is nonflammable. In fact, a mixture of 4% hydrogen in nitrogen could be safely vented in air without any hazard whatsoever. A mixture of at least 7% hydrogen in nitrogen is necessary when mixed with air to form a flammable (but not detonatable at the 7% level) mixture somewhere in the vent area. The only way a hazardous situation can exist in the vessel is for large amounts of air to be left in (which implies a simultaneous failure of not one but many instruments and procedures) while at least 8% steam is injected (again contrary to procedures and instrumentation) to finally reach a gas mixture which may be flammable but almost certainly will not be detonatable. Once inerting is accomplished, up to 7% hydrogen in the nitrogen can be safely vented to the atmosphere without any flammability hazard.

To determine what initial concentration of steam should be used one has to assume that the component may have large surfaces and/or frost areas present. This is generally true and implies that a low concentration (approximately 1%) of water should be used to be conservative. After the large areas have reacted (or the hydrogen meter indicates that they were not present) the feed may be quickly raised to 5% for the majority of the processing. When the reaction has been completed (hydrogen less than 5 ppm at normal nitrogen feed rates, which is nearly the lower detection limit) it is well to raise the steam concentration to a high

value (that is, greater than 30%) and wait several hours to be sure that the hydrogen concentration is still zero. This will prevent the flooding of small amounts of sodium that might still be reacting.

This high steam concentration may be obtained in several ways. One way is to maximize the steam flow and/or minimize the nitrogen flow. Another way is to partially flood the vessel and sparge the gas through the water to raise its moisture content. Any method that is acceptable from an equipment viewpoint is satisfactory to accomplish the purpose.

The initial reaction surface temperature should be at least 146°F because of the following reaction:

which takes place at that temperature. This reaction will lead to large instabilities in the consumption reaction because of the "loss" or "gain" of water at the consuming surface. This is probably the major cause of spikes where processing was under 150°F and has been vividly demonstrated in the laboratory to be destabilizing.

The usual inaccuracies in instrumentation coupled with the unknowns about the temperature at all parts of different equipment configurations lead to the judgment to use 160°F as the specified starting temperature. A temperature as low as 150°F could be used if the equipment and procedures insured that all surfaces were truly at least 150°F.

The reasoning for the 190°F maximum temperature is similar. The sodium surface temperature during reaction may be 10 to 15°F hotter than ambient, but the sodium must not be allowed to melt. If it does, then the available reaction area can drastically change and this has repeatedly been demonstrated in the laboratory as an unstabilizing factor.

The 10°F increase in operating temperature has proven very effective in dampening reaction rate oscillation. Temperature increases significantly

below this value would probably not be effective in accomplishing the stabilizing action. Higher increases would unnecessarily use up the available temperature range for restabilizing the reaction at a future time. Since it is the reacting surface temperature that is important and the gas has quick access to those surfaces, stability is obtained very quickly. It is of no importance that the bulk interior temperatures of a large component may not make the 10°F increase for several hours.

C. EXTENDED RANGE OF VARIABLES

In reviewing the present criteria, changes in operating temperatures do not appear productive. The absolute limits are the previously mentioned 146 to 208°F. Enlarging the 160 to 190°F condition to come closer to the absolute limit would not appear to effect any practical saving in equipment, design, operation, or procedures.

The limits on the oxygen and hydrogen could safely be enlarged without requiring better instrument accuracy or tighter procedures. In practice, the initial impurity level will be very close to zero regardless of what is specified in the Standard.⁽¹⁾ By the same token the chance of a real component operating at a hydrogen concentration above 1% is small. Raising the hydrogen specification may reduce the unnecessary writing of "incident reports" for situations where no hazard really existed.

Changes in water vapor concentration could significantly reduce the vapor phase processing time. Since the reaction rate is dependent on the 1.2 power of concentration, raising the water from 5 up to 10% would increase the rate by a factor of 2.7, or reduce the vapor reaction time by 63%. But, this may only amount to a 20 to 30% decrease in total processing time from loading, through inerting, through vapor, through rinse, through drying to unloading.

Early developments showed much greater instability at the higher water concentrations (above 15%). The feedback concept was tested only with 5% water. Concentrations up to 10% may be feasible but would definitely require development work to confirm stability.

It is of interest to note that European systems run at much higher water concentrations (in either the steam or mist mode) and do have indications of instability. However, they have chosen to disregard this and apparently do not consider it a problem. If there were no undrained sodium pools, then low water concentration would be unnecessary and a suggested operating level would be near 100% water vapor. Although designers generally seek to avoid pools, there is some risk of small ones occurring from time to time which may manifest themselves in "bangs," "pops," etc., high temperatures, or other indications from a rapid uncontrolled reaction. Conservatism indicates remaining with the lower concentration, slower processing.

Starting at 190°F has often been suggested to avoid instability and simplify the process. This high temperature start would make very little real saving in capital equipment or procedures and would only slow the reaction rate, but not prevent possible instabilities. However, it would prevent the option of taking effective action to regain a stable situation.

In summary, the vapor phase of the WVN process is confined by a number of practical constraints that prevent major changes within the moist gas concept. The area most lucrative to improvement would be a modest increase in the normal water vapor concentrations.

III. RINSING

A. CREVICE STUDIES

Crevicees were considered to be the most difficult configuration to clean and could be most effectively done in the rinse phase. Most of the testing was done in 1972-73. The primary motivation at that time was the IHX and IVHM crevice problems (which were typical of many other components) and what design changes might be made to improve the situation. One study⁽¹³⁾ showed that crevices could be effectively and repeatedly cleaned if the reactants had access to the sodium.

A series of tests⁽¹⁴⁾ on crevices from 1 to 29 mils wide revealed a number of interesting points (see Figures 8, 8A and Tables 5 and 6):

- Increased water vapor concentration gave faster penetration into a given crevice.
- Hot (185°) water as opposed to ambient water gave penetration rates 10 to 100 times faster.
- Removal depth increased with the log of time.
- Removal from crevices smaller than 1 mil tended to be erratic.
- Orientation of crevices was also important--the open end down attitude was the most difficult to clean.

The equation developed for gas phase crevice penetration was

$$k = 0.50 + 1.527 \ln t$$

k = inches removed

t = hours

which is for the condition of 100% steam at 215 to 250°F with 26 to 56 mil gaps.

B. RINSE STUDIES

In early 1975 a number of low priority tests were conducted to determine the results of bulk rinsing. These tests were set up to measure only the

FIGURE 8. Time Required to Remove 100% of Sodium from 13-Mil Gap Crevices

FIGURE 8A. Removal of Sodium From 1 mil Radial Crevice with Hot (180 to 200°F) Liquid Water

TABLE 5. Effective Sodium Removal from Crevices with 4-hr Exposure

Condition of Specimen	Specimen Length, in.	Diametral (a) Gap, Mils	H ₂ O Vapor (b) Vol/%	Sodium (c) Removed, Percent
Both Ends Open	3	2	30	73-100
	3	8	10	100
	3	8	15	100
	3	8	20	97.5
	3	8	30	100
	3	8	75	100
	3	14	10	96
	3	14	20	100
	3	14	30	98
	3	14	40	100
	3	14	50	100
	3	14	75	100
	3	14	100	100
	3	26	20	70
	3	26	30	75
	3	26	50	99.5
	3	26	75	100
	3	26	100	100
	3	58	30	90
	6	26	30	73
	6	26	50	88
	6	26	75	91
	6	26	100	98.5
	6	58	50	55
	6	58	75	69
	6	58	100	100
One End Open	3	26	20	53
	3	26	30	58
	3	26	50	79
	3	26	75	100
	3	26	100	100
	3	58	30	55
	3	58	50	84
	6	26	30	36
	6	26	50	40
	6	26	75	63
	6	26	100	67
	6	58	75	49

a. Radial gap equals average gap half that listed.

b. Measured by Flowmeter.

c. Calculated Values.

TABLE 6. Effective Sodium Removal from 13-mils Crevices^(a)
for Various Times

<u>Specimen Length, in.</u>	<u>Moisture, Percent</u>	<u>Time, hr</u>	<u>Sodium Removed, Percent</u>
6	30	1	70
6	30	2	67
6	30	4	73
6	30	18	100
6	50	1	65
6	50	2	75
6	50	4	88
6	50	8	100
6	50	18	100
6	100	2	88
6	100	4	98.5
6	100	6	100

a. 26 mils diametral equals average gap width of 13 mils

effects of large surface areas and pools so there would be no confusion with logarithmic effect of crevices. Results were compared with the small amount of SRDA operating data.

The test showed surface rinsing was effective in a very short time (one residence time or less) and that agitation had little effect. The only case where turbulence was of value was where direct impingement was obtained on the nondrainable area to "splash out" the remaining caustic solution. Minimum volume rinses were also confirmed to make the most efficient use of rinse water. It was concluded that the most effective rinsing campaign might be as follows: Use one or two quick rinses to remove the majority of caustic and bring conductivity meters on scale. Then use one long rinse to allow for diffusion into and out of crevices and small pockets or pools. One additional rinse could be made for a quick "polish" to insure surface cleanliness.

With the experience of this and the various crevice test, it would be useful to examine the performance of some real components. In the case of

the IVHM, the rinses were changed every few hours but the rinse conductivity did not seem to improve very rapidly. This is probably due to the myriad of large and small crevices and pockets the caustic has to diffuse from. In retrospect, leaving the rinses in some arbitrarily longer time (24 to 48 hr) may not have extended the actual cleaning time much but would have used less water. Since this device is fairly open access of rinse to all areas was presumed to be good.

The IHX tube bundle model was another item cleaned in the large AMCF vessel. After one long rinse there was caustic in the interior which had not been present in the previous test where it had been rinsed for the same length of time (but many more cycles). It was reasoned that because of the shrouding there was no mechanical interchange of fluid so the rinsing was not as effective. On a successive try the rinse was raised and lowered above and below the model every 24 hr. This was equal to a "new" rinse every day with a mechanical drain and refill of all voids. For the large vessel little extra water over the initial fill volume was used. The result was that all caustic was removed from the model by this technique.

C. PARAMETERS

From the foregoing it can be determined that high temperatures are beneficial to rinsing. The reaction rate of water with sodium is increased, the solubility of sodium products in water is increased, the diffusivity of sodium compounds in water increases, and the viscosity of water decreases. All of these speed up sodium removal operation.

The water concentration should also be as high as possible. The early crevice test showed that the present 160°F, 5% water during vapor phase processing would be rather ineffective (100% steam at over 215°F was required to give reasonable rates). There were some tests in liquid water that showed a temperature of 185° as being effective.

At this point a judgment was made to attempt crevice cleaning during the rinse phase only rather than extending the vapor phase to high temperatures and concentration. Water temperatures over 212° would require a pressurized vessel

which introduces a number of mechanical difficulties. There is also a problem of nearing the caustic stress corrosion cracking condition for some areas of the component. If a gas sparge is to be maintained at temperatures over 200°F large quantities of condensate begin to be present in the vent piping of the cleaning system. Therefore, the 180 to 200°F range was picked as a temperature high enough to be effective but low enough to simplify the requirements for process hardware.

The initial rinse temperature had originally been ambient. It was reasoned that this would decrease the sodium water reaction rate somewhat and, therefore, be a safer way of flooding the component. However, it would also freeze high concentration caustic in crevices and puddles which take more time to redissolve even when hotter rinses are finally used. Since the experience with better gas phase control showed that small amounts of remaining sodium could be detected before flooding, the initial rinse temperature was changed from ambient to 180°.

A high rinse water resistivity endpoint was chosen as the most realistic for various component configurations. Significant amounts of caustic had not been found when the circulating rinse water maintains a Grade B conductivity. The number (a few or many) or duration (a few hours or a few days) of rinses was not felt to be amenable to facility design but should be the decision of the operator and would vary from component to component. It is then necessary to provide some rinse flexibility in the facility design.

The major rinse variable, subject to design manipulation, is temperature. Low ambient temperatures would seem to save little in the way of equipment and would greatly extend processing time. Higher temperatures would speed up rinsing but would also require pressurizing of the system and components.

Agitation is a possibility for speeding the rinsing in critical areas. But it would be difficult to design a general device to impinge the rinse at the right place or places on a variety of components, many of which are very effectively shrouded. A simpler method would be "twin tanks" where the rinse could be pumped back and forth to fill and drain the component volumes without using any new water.

IV. DRYING

A. THEORETICAL CONSIDERATIONS

There are three main methods of drying in the WVN process: vacuum, hot gas only, or a combination of the two. The fastest drying method would be preferred to minimize processing time, but there is no clear cut advantage to any method for the general case.

Evaporation of water is difficult because of its high heat of vaporization (970 Btu/hr at 212°F and 1036 at 100°F). It is also complex because it involves the two mechanisms of heat and mass transfer simultaneously. The use of a vacuum will greatly increase the mass transfer rate of water vapor from a puddle, but the movement of water soon stops unless the needed amount of heat is added to the pool by some means. And, the only method of heat transfer under a vacuum is by radiation which gives very low heat fluxes at the temperatures we might expect in normal operation (100 to 200°F).

On the other hand, the use of dry gas does provide a steady means of heat input to the component even though it does inhibit the mass transfer rate. While heat transfer by natural convection is poor, it is several times better than would be expected by radiation under the same temperature differences.

The choice of method then depends more upon the peculiarities of each component being processed. A component with a large mass of metal (relative to the water), that is in good thermal contact with the water, may dry quicker under a vacuum. An example would be the IVHM where a small amount of water may be present in both counter bore holes and in crevices of massive parts. Here, there is more than enough thermal energy stored in the metal to quickly evaporate all the water.

On the other hand a component with a small mass in relatively poor thermal contact with water would dry much quicker by hot gas. An example of this would be the baffle sections of many components or the IHX where the tube bundle is shrouded. Tubes in inner areas would be "blind" to radiant heat transfer under a vacuum, but would absorb heat much faster from a circulating gas.

Other components may be intermediate in drying performance with either operation. Or they maybe extremely good in one particular mode depending on their configuration of surfaces that may act as good heat gathering fins in radiant or convective transfer.

With this in mind, it is now worthwhile to look at the various equipment experiences.

B. EQUIPMENT EXPERIENCE

During sodium removal testing in SRDA of the pump bearing pocket one test was made of a vacuum-versus-gas drying cycle.⁽²⁾ The two modes were: 1) three 15-min cycles of 180°F nitrogen interrupted by two 15-min vacuum cycles for a total of 75-min, and 2) 90-min of 180°F nitrogen. For this particular configuration of crevices (the predominant feature of this model) the first cycle was more effective. This is not surprising since most combination cycles do show better performance over either pure drying or pure vacuum.

Experience in AMCF was more limited. There have been no direct tests of vacuum-versus-gas only versus vacuum/gas combinations. During cleaning of the IVHM a hot gas only cycle was used and found to be effective--no water was found in any part of the machine. The moisture content of the exit gas had remained at 300 to 350 ppm for a number of hours before the component was removed. A pool of water in some connecting piping was later found which must have been the source of the low-level moisture. This demonstrates that pools of water can actually be detected in large systems.

A DHX was recently cleaned with ethanol. Some preliminary drying was attempted with essentially ambient temperature gas. The gas was initially hot, but because of the heat transfer characteristics of the device, it quickly cooled to ambient. A shift was made to vacuum and drying was successfully terminated after a low pressure had been reached. A sample of the back-filled (but not recirculated) gas showed less than 1 ppm ethanol, but no visually inspection was possible. Presumably this demonstrated that vacuum could be effective on large objects although in this case it should be understood that the object was very effectively warmed by the ambient air.

A short study was conducted in SRDA on the three drying modes: vacuum only, gas only and vacuum/gas combination. The "component" was a 12-in. diameter pot filled with water and sitting on a grate in the vessel (see Table 7 for the resulting data). In this case the vacuum mode was more effective but the component was hardly typical. A simple pot 1/8-in. thick had a mass much less than the water contained. Since there was little metal mass, the experiment would tend to emphasize the biggest difference between the two drying modes. It should also be noted that the grating upon which the pot sat acted as a much more effective heat gathering fin in the vacuum mode than in the natural gas convection mode.

TABLE 7. Results of SRDA Drying Mode Tests

<u>Mode</u>	<u>Pot or Wet Bulb Temperature, °F</u>	<u>Evaporation Rate, lb/water/hr-ft² of Water Area</u>
Hot gas	150	0.40
Vacuum	80	0.69
Mixed	80/150	0.50

C. PARAMETERS

From a practical viewpoint there are several considerations: The first viewpoint is that in real components the difference in evaporation rates between the several modes would tend to be less than shown by any specific test or example. Second, the pockets of water in components will usually be less than 1-in. deep thereby giving fairly short drying times in either mode. Third, is that the equipment for the hot gas mode is already in being as part of the WVN process. The fourth, is that vacuum drying does require additional equipment, maintenance, and procedures adding to the installed and operating cost of the facility. Pursuant to this it should be noted that in large real systems it will take on the order of at least 1 hr to change valve line ups, shutdown/startup equipment, etc. This effort is required to change from hot gas to a vacuum mode and the same time required to change back again.

For the present RRD standard the hot gas only mode was selected as the simplest under the variety of conditions that would probably be encountered in the normal cleaning facility. A vacuum mode could certainly be used in addition or in combination with the hot gas drying. In view of the marginal decrease in total processing time that a vacuum drying system might give versus the extra cost it would seem reasonable to not include vacuum drying as a part of the WVN process.

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