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# EXPERIMENTS ON COLD-TRAP REGENERATION BY NaH DECOMPOSITION

by

MASTER

C. C. McPheeters, S. B. Skladzien,
and D. J. Raue

BASE TECHNOLOGY

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# EXPERIMENTS ON COLD-TRAP REGENERATION BY NaH DECOMPOSITION

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Chemical Engineering Division

June 1980

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# EXPERIMENTS ON COLD-TRAP REGENERATION BY NaH DECOMPOSITION

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C. C. McPheeters, S. B. Skladzien,
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## ABSTRACT

Cold-trap regeneration may be very important in future LMFBRs because of the expected high hydrogen source from the steam generators. This hydrogen precipitates as NaH in the cold trap and may fill the trap within one year of operation. Several methods of cold-trap regeneration were considered, but the simplest and least expensive appears to be decomposition of NaH under vacuum at elevated temperatures.

Experiments were done to assess the feasibility of this method for cold-trap regeneration. Small-scale simulated cold traps (SCT) were loaded with NaH and NaH plus $Na_2O$, and were heated both under vacuum and under a sweep gas at 100 kPa. The evolved hydrogen was converted to water by a CuO bed and collected in a weighing tube.

The experimental results indicate that significant hydrogen evolution rates occur $\sim$100 K lower in temperature under vacuum (600 K) than under 100 kPa pressure (700 K). The results also indicate that the hydrogen evolution rate is reduced (at 600 K) more than one order of magnitude (to $\sim$0.15 $\mu$g/s) by the addition of $\sim$50% $Na_2O$.

## I. INTRODUCTION

Operation of liquid sodium systems such as the LMFBR heat-transfer systems requires continuous removal of impurities such as oxygen and hydrogen from the sodium. Cold trapping is the most widely used method for this purification. Cold traps are usually operated until they become filled with impurity deposits to the extent that a flow blockage occurs. At that point, the cold trap is removed from the system and replaced with a new one. The choked cold trap is usually stored indefinitely or cleaned with steam or alcohol for refurbishing and reuse. The practice of replacing choked cold traps may be acceptable in cases where the useful lifetime of the trap is five to ten years; however, if the expected time to plugging were on the order of one to two years, the replacement operation would be too expensive and time-consuming.

Measurements of diffusion of hydrogen from water-side corrosion of steam-generator tubes through the tube wall to the sodium side[1] indicate that the hydrogen source rate may be sufficient to fill the cold trap with NaH within one year of full-power operation. This short cold-trap lifetime is expected only in the intermediate heat-transport system (IHTS), while the expected lifetime of the primary heat-transport system (PHTS) cold traps is longer because the impurity sources are much smaller.

Since the primary impurity of concern in the IHTS is hydrogen, thermal decomposition of NaH should be well suited for regeneration of the IHTS cold traps. This method consists of draining some of the sodium from the cold trap to provide a cover-gas space, heating the trap to 670 K (400°C) under vacuum, collecting sodium vapors in a high-efficiency vapor trap, converting the evolved hydrogen to water, and collecting the water for disposal as tritiated water. Of course, impurities other than NaH, such as $Na_2O$, will remain in the cold trap. After many regeneration cycles, these stable impurities may accumulate sufficiently to make cold-trap replacement necessary; however, extending the cold-trap lifetime from one year to, perhaps, five to ten years justifies use of the NaH decomposition method.

Several methods of cold-trap regeneration other than thermal decomposition of NaH have been attempted with varying degrees of success. These methods include replacement of the mesh section, steam or alcohol cleaning, dissolution of impurities with hot sodium, and reaction of $Na_2O$ and NaH to form NaOH to be drained from the trap. These methods are discussed in the following paragraphs to place them in perspective; however, they are not discussed in detail because the primary objective of the paper is to present the results of recent studies of the thermal decomposition of NaH.

The method of replacing the mesh-packed section has been used as a means of renewing cold traps.[2,3] This method requires isolating the cold trap from the main system, draining the sodium, opening the trap, removing the loaded mesh section, cleaning impurity deposits from internal surfaces, replacing the mesh section with a clean unit, and resealing the trap. The method is simple and direct; however, it suffers from the following disadvantages: the need for a special configuration to allow opening the trap and removing the mesh section, the need for special sodium-handling equipment, the problem of handling and disposal of tritium and other radionuclides, and possible problems in removing the mesh sections due to impurity deposits in close-tolerance regions.

The method of cleaning choked cold traps with steam or alcohol has been used with success.[4,5] The method requires removal of the trap from the sodium system, draining the sodium from the trap, injecting the steam or alcohol into the trap to dissolve the impurity deposit, drying the trap, and replacing it on the system. Although this method has been used successfully, it suffers from the requirement of cutting the system piping to remove the trap. A method for performing the regeneration *in situ* is much more desirable.

Removal of impurities from a choked cold trap by dissolution in hot sodium (∿670 K) is attractive because of its simplicity and the ability to process the trap *in-situ*. The process involves pumping hot sodium through the trap and cooling the sodium to precipitate the impurities in a less expensive vessel that may be processed at a more leisurely pace. Although this method has obvious advantages, the waste-management problems associated with handling the radioactive sodium waste are more difficult than those involved in handling gaseous tritium or tritiated water.

The Na-NaOH vertical section of the phase diagram of the $Na-Na_2O-NaOH-NaH$ system[6] shows a region of two immiscible liquids, Na(ℓ) and NaOH(ℓ), above 685 K (412°C). The concept of heating a cold trap above 750 K to allow $Na_2O$ and NaH to react and form liquid NaOH was explored by McKee.[7] In this method, the liquid NaOH could be drained directly from the trap for disposal. While the method is very attractive for its simplicity and direct approach, the corrosion problems associated with the molten NaOH at 750 K must be examined more closely before it can be used on a reactor system.

Compared with the other methods discussed above, the NaH decomposition method has the advantages of simplicity, true *in-situ* operation, rapid regeneration, and an easily disposable waste product (either tritiated water or a tritiated metal hydride). Although similar potential corrosion problems may exist with this method as observed in the molten NaOH method, the operating temperatures are significantly lower, thus the corrosion rates should be much lower.

## II. PRINCIPLES OF HYDROGEN EVOLUTION FROM NaH IN Na

The NaH decomposition method of cold-trap regeneration may be operated either under vacuum or with an inert sweep gas. In either of these cases, the sodium must be, at least partially, drained from the trap to provide a gas space through which to flow the inert gas or to allow a vacuum space above the sodium. This gas space may be created by pressurizing one side of the trap (either inlet or outlet) to force sodium out the opposite side and into the main sodium system. In the sweep-gas case, the gas is pumped through the trap to sweep out the hydrogen as the temperature is raised to ∿700 K (430°C). In the vacuum case, the gas space is evacuated while the temperature is raised to ∿670 K (400°C).

Hydrogen may be evolved from the trap in either case by two mechanisms: 1) diffusion of hydrogen from the NaH through the liquid sodium to the sodium/gas interface either in a quiet sodium pool or aided by thermal convection, and 2) formation of hydrogen bubbles on the NaH surface that disengage and rise to the sodium/gas interface. The diffusion mechanism is obviously much slower in a quiet pool than when aided by thermal convection. In either case, the diffusion mechanism should be slower than the bubble formation mechanism. The diffusion mechanism would be expected to predominate at lower temperatures (<600 K) where the hydrogen pressure over NaH is insufficient to nucleate bubbles.

The rate of hydrogen transfer from the NaH deposit to the liquid surface by diffusion may be expressed by

$$\phi = DA \frac{dC}{dh} \tag{1}$$

where $\phi$ = the hydrogen flux, μg/s

$D$ = the hydrogen diffusivity in sodium, $m^2/s$

$A$ = the cross-sectional area perpendicular to the diffusion path, $m^2$, and

$dC/dh$ = the hydrogen concentration gradient along the diffusion path, $\mu g/m^3 \cdot m$.

The hydrogen concentration in the sodium is assumed to be at saturation at the SCT temperature adjacent to the NaH deposit and essentially zero at the liquid/gas or liquid/vacuum interface. The diffusivity of hydrogen in sodium is not known; however, it may be estimated as $\sim 10^{-7} m^2/s$ at 670 K as a maximum. The solubility of hydrogen in sodium was measured by Vissers *et al.*,[8] and is expressed by

$$\log C(g/m^3) = 5.998 - \frac{2880}{T(K)} \qquad (2)$$

Using Eqs. 1 and 2, and the estimate of hydrogen diffusivity in sodium, the maximum hydrogen flux that could be achieved at 670 K without thermal convection is $\sim 0.14$ μg/s (for the geometry used in the tests described below). This hydrogen flux would be increased by thermal convection; however, these experiments were conducted in isothermal vessels so that significant thermal convection would not be expected. Hydrogen fluxes much greater than 0.14 μg/s could be achieved by nucleation and growth of hydrogen bubbles on the NaH deposit. The pressure required to nucleate a bubble in a homogeneous liquid is given by the Laplace equation:

$$\Delta P_c = \frac{2\sigma}{r_c}$$

where $\Delta P_c$ = the pressure differential across the liquid-gas interface of a bubble of critical size,* kPa,

$\sigma$ = the liquid surface tension, N/m,

and $r_c$ = the radius of a bubble of critical size, mm.

Bubbles that nucleate on a solid-liquid interface require a lower pressure differential which may be expressed by

$$\Delta P_c = \sigma\left(\frac{1}{r_c} + \frac{1}{r_c'}\right) \qquad (4)$$

where $r_c$ and $r_c'$ are the principal radii of curvature of the bubble surface. In cases where the solid is poorly wetted by the liquid, the bubble-solid contact angle is large and the bubble is significantly distorted from a spherical shape. This distortion results in a significant difference in the radii of curvature. Conversely, when the solid is very well wetted by the liquid, the contact angle approaches zero, and the bubble shape approximates a sphere. Contact angles have not been measured for hydrogen bubbles in sodium in contact with sodium hydride; however, sodium is expected to wet sodium hydride very well, so the bubble shape should be approximately spherical. Since the greatest pressure is required to sustain a spherical bubble, assumption of a spherical shape is the most conservative case.

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* A bubble smaller than the critical size is thermodynamically unstable relative to the gas in solution, while a bubble larger than the critical size is stable in the same solution.

The hydrogen pressure required to form a bubble of critical size is equal to the sum of the sodium hydraulic pressure and the pressure differential across the bubble wall. The sodium hydraulic pressure arises from the gas pressure over the sodium, $P_g$, plus the head due to the sodium depth, $P_h$. The hydrogen pressure in the bubble is then expressed by

$$P_H = P_g + P_h + \Delta P_c \tag{5}$$

For sodium at 670 K, Eq. 3 may be written

$$P_H(\text{kPa}) = P_g(\text{kPa}) + 0.00825h + 0.334/r_c \tag{6}$$

where h is the sodium depth, mm, and $r_c$ is the critical bubble radius, mm.

The equilibrium hydrogen pressure over NaH has been measured by several investigators, and the most reliable seem to be Banus *et al.*,[9] who determined the relationship

$$\log P(\text{kPa}) = 9.61 - \frac{5070}{T(\text{K})} \tag{7}$$

The temperature required to produce hydrogen bubbles in sodium due to decomposition of NaH may be calculated as a function of critical radius by combining Eqs. 6 and 7 to obtain

$$T(\text{K}) = 5070/[9.61 - \log(P_g + 0.00825h + 0.334/r_c)] \tag{8}$$

Figure 1 is a plot of the bubble nucleation temperature *vs.* the critical radius for two cases: 1) an inert gas pressure of 100 kPa over the sodium and 2) vacuum over the sodium. Equation 8 and Fig. 1 show two important features, *i.e.*, the sodium depth is relatively unimportant up to approximately 1 m, and if the critical bubble radius is less than ∿2 μm, the bubble nucleation temperature should be approximately the same under vacuum as it is at 100 kPa pressure. For critical bubble radii greater than 2 μm, bubble nucleation should occur much more readily under vacuum than under a 100-kPa cover gas. The critical radius of hydrogen bubbles in sodium has not, to our knowledge, been measured.

Once the bubble has reached the critical size, its rate of growth is limited only by the rate at which hydrogen becomes available by decomposition of the NaH. The kinetics of NaH decomposition was measured by Naud[10] in the temperature range of 550 to 585 K. Naud determined that the NaH decomposition reaction is first-order and follows the relationship

$$-\frac{dC}{dt} = kC \tag{9}$$

where C is the amount of NaH present in grams, t is the time in seconds, and k is the rate constant in reciprocal seconds. If Naud's data may be extrapolated to 670 K, the rate constant is 0.0019 $s^{-1}$, and (assuming 5 g NaH for these experiments) the limiting initial rate of hydrogen evolution

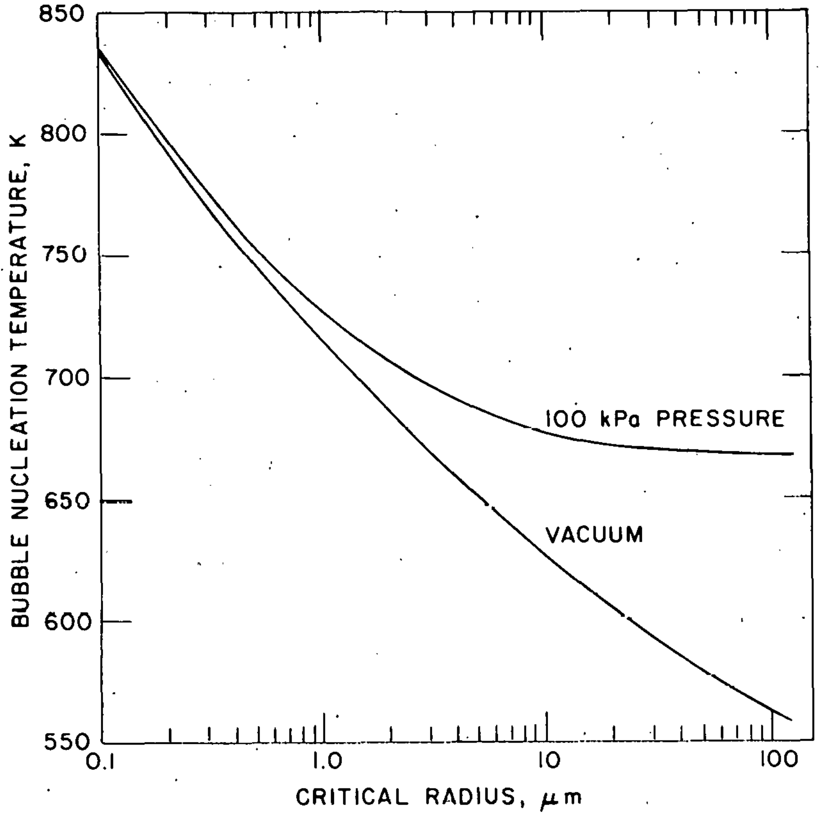

Fig. 1. Temperature Required to Nucleate Bubbles of Various Sizes under Vacuum and Atmospheric Pressure.

that could be expected in these experiments in 400 μg/s. This rate could only be achieved under ideal conditions with no rate-inhibiting factors such as competing reactions, solution/dissolution steps, or diffusion barriers.

## III. EXPERIMENTAL APPARATUS

The apparatus used to perform the experiments is shown schematically in Fig. 2. In the sweep-gas mode of operation, the inert gas flows through a flowmeter into the simulated cold trap (SCT), which is a sealed stainless steel vessel 40 mm in dia and 125 mm high. NaH was placed in the bottom of the SCT and covered with stainless steel wire mesh typical of that used as cold-trap packing. Sodium was poured into the SCT to fill it to the top of the wire mesh. The initial sodium depth was ∿50 mm in all of the tests.

The inert gas flows from the SCT, carrying a burden of sodium vapor and hydrogen, into the reflux condenser. The condenser is 40 mm in dia and 575 mm high. The upper 400 mm is filled with wire mesh packing and the lower 175 mm is intended for collecting the condensed sodium. The vapor-laden gas enters at ∿725 K at the approximate midpoint of the condenser. A temperature gradient is maintained so that the top and bottom ends are ∿400 K. As the gas flows upward, it is cooled, the sodium condenses and flows down

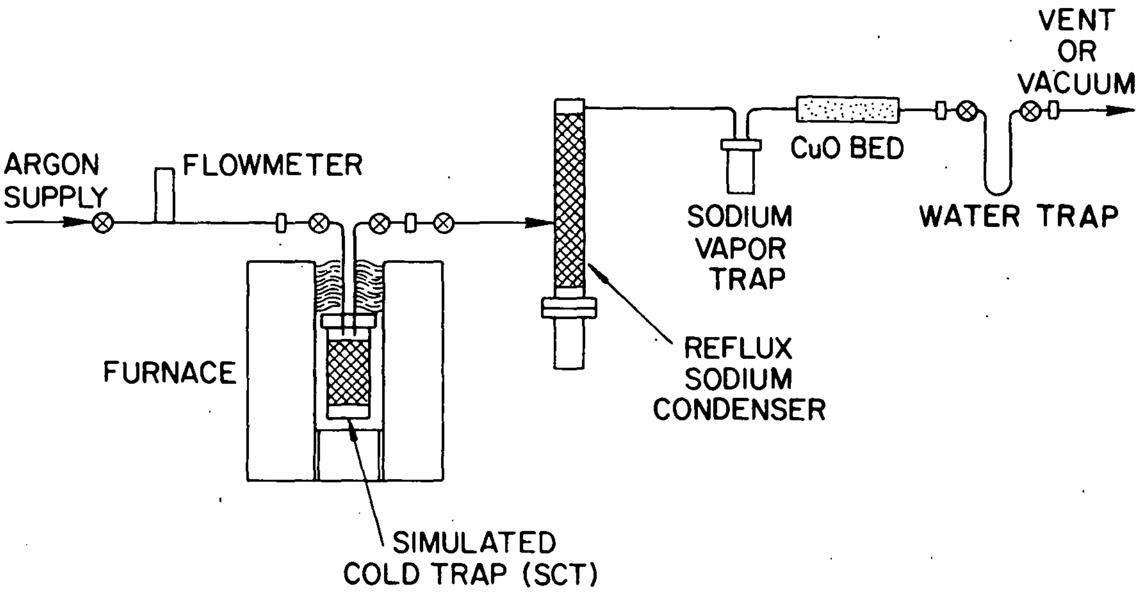

Fig. 2. Test Apparatus for Removing Hydrogen from Simulated Cold Traps (SCT)

through the mesh packing. The downward-flowing sodium aids the condensation of the incoming vapor. No doubt, some of the condensed sodium reevaporates in the hot zone; however, it eventually reaches the cool lower end where it is retained for the duration of the test.

Gas leaving the condenser is cooled to room temperature and enters a final vapor trap for removal of residual sodium aerosol. This vapor trap provides a quiet volume for aerosol settling and the gas exits through a porous stainless steel filter of ∿10-μm pore size.

The gas flows from the vapor trap to a CuO bed that is maintained at 775 K to convert the hydrogen to water. The water vapor is collected in a trap filled with Molecular Sieve 5A adsorbent, and the quantity of water is determined by the weight gain of the trap. Although only one trap is shown in Fig. 2, two traps were actually used in parallel so that one trap could be weighed while the other continued to collect water.

When the system is operated under vacuum, the valve at the inlet to the SCT is closed and all other features of the system remain the same. The molecular sieve trap was found to retain adsorbed water under vacuum even at room temperature and with very low water loadings. Therefore, the traps were operated at room temperature during these tests. Hydrogen recoveries ranging from ∿70% in one experiment to >97% were observed.

Corrosion tabs were placed in the SCT for three of the tests. They were cut from cold-rolled type 304 stainless steel sheet and were 12 mm by 25 mm by 0.2 mm thick. The flat surfaces were left as received and the edges were filed smooth. The tabs were ultrasonically cleaned in acetone, rinsed in alcohol, and dried before being placed in the SCT in the NaH on the bottom.

The parameters of the experiments performed in this series are summarized in Table 1.

Table 1. Summary of Cold-Trap Unloading Experiment Parameters

| Test No. | SCT Temp, K | Test Time, ks | Compounds in SCT; (Weight, g) | Ar Flow or Vac., $mm^3/s$ |
|---|---|---|---|---|
| 1 | 718 | 18.0 | NaH(6.16) | 14 |
| 2a | 693 | 10.8 | NaH(5.56) | 14 |
| 2b | 710 | 18.0 | | 14 |
| 3a | 523 | 79.2 | NaH(3.81) | Vac. |
| 3b | 598 | 202 | | Vac. |
| 4a | 543 | 26.3 | $Na_2O$(3.14) | Vac. |
| 4b | 568 | 93.6 | NaH(2.08) | Vac. |
| 4c | 588 | 59.8 | | Vac. |
| 4d | 603 | 86.0 | | Vac. |

The procedure for performing these tests was to load (in an inert-gas glove box) the NaH into the SCT along with the corrosion tab, insert the wire mesh, and pour liquid sodium over the mesh to a height sufficient to cover the mesh. The SCT was then sealed with a Varian bakeable vacuum flange using a nickel gasket. The SCT inlet and outlet valves were closed, and the SCT was transported to the test laboratory and connected to the test apparatus. The valves were then opened and argon flow was initiated while the components were at room temperature. The CuO bed, the sodium condenser, and finally, the SCT were heated to operating temperature to initiate the experiment. The SCT operating temperatures listed in Table 1 were maintained for the listed test times, and, at regular intervals during the test, the water-collection tubes were weighed to monitor the hydrogen evolution.

Neutron radiographs of the SCT were taken before and after the tests to qualitatively determine the effect of the unloading procedure. Since thermal neutrons are preferentially absorbed by hydrogen, any hydrogen-bearing compounds (such as NaH) are easily detected in an all-stainless-steel system as light, relatively unexposed areas on the film. Figures 3 and 4 are contact prints of neutron radiographs of the SCT used in test number 2. In the contact prints, the NaH appears as a dark area in the bottom of the SCT. Figure 3 is the pretest radiograph and Fig. 4 is the post-test radiograph. Note that the NaH deposit obscures the corrosion tab in Fig. 3, and that in Fig. 4, the NaH appears to be completely eliminated. (The weight gain of the water traps also confirms complete decomposition of the NaH in test number 2.) These radiographs also indicate that the sodium appears

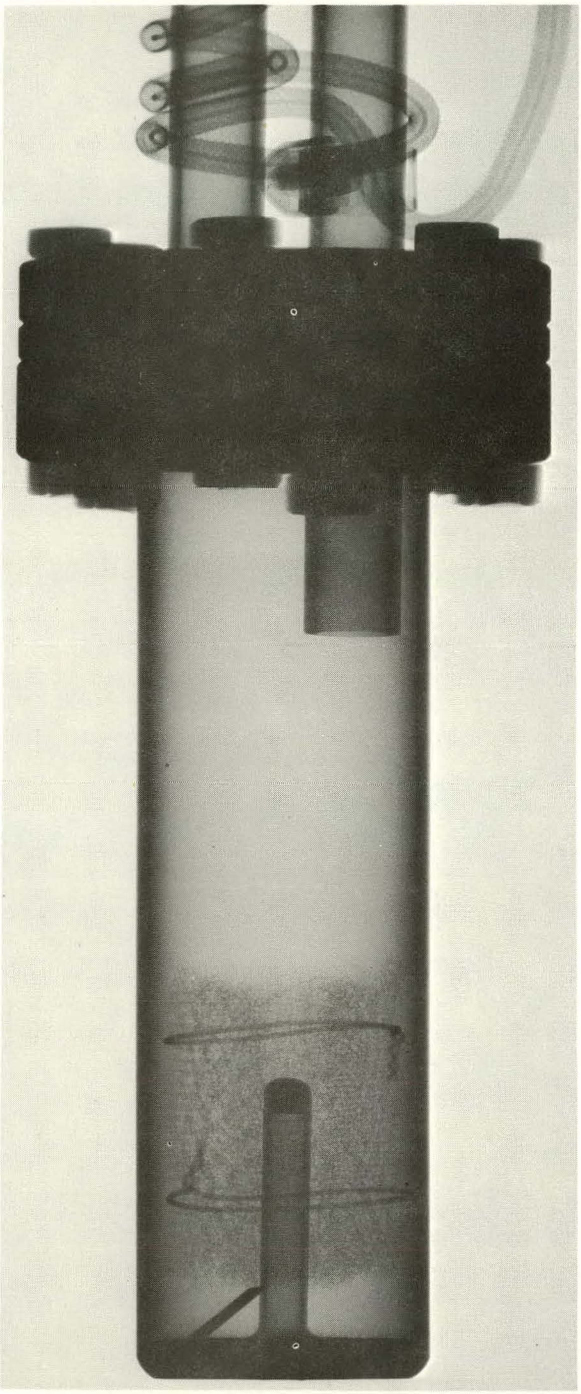

Fig. 3. Contact Print of Pretest Neutron Radiograph of SCT Number 2

to be completely removed from the SCT by the test procedure. The sodium appears as a lightly shaded region in Fig. 3 up to a height just below the inlet and outlet tubes. This region is not shaded in Fig. 4, indicating that the sodium has been removed. Post-test examination confirmed this sodium removal.

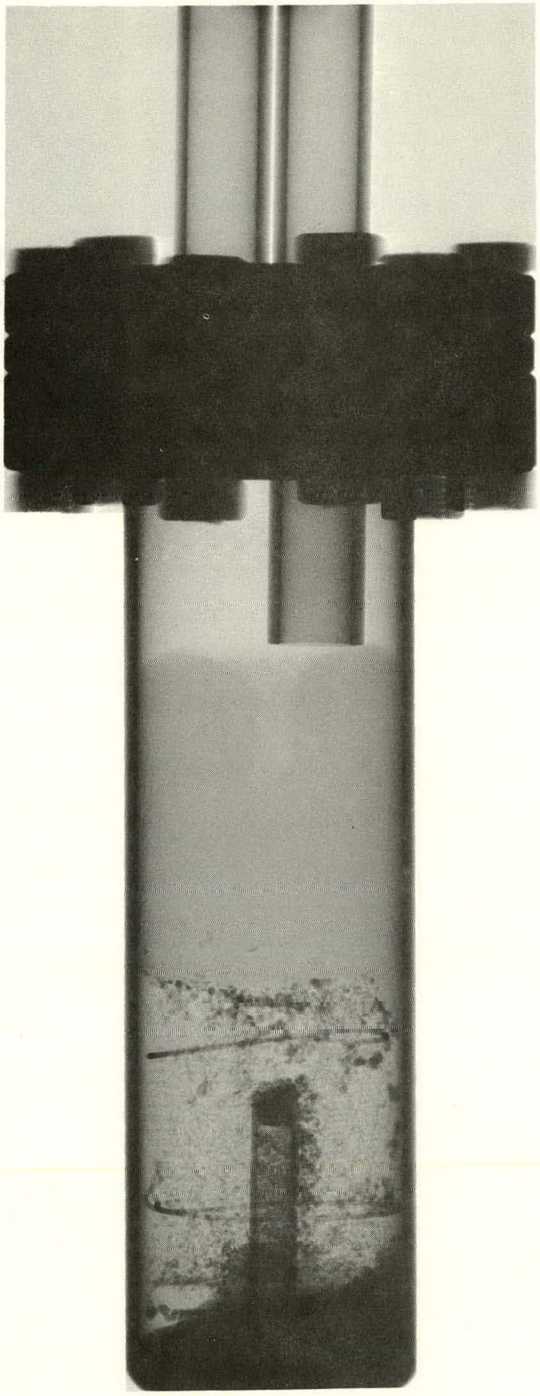

Fig. 4. Contact Print of Post-Test Neutron Radiograph of SCT Number 2

Accumulation of water in the traps occurred at different rates during each test, depending upon the test conditions. Figure 5 shows the results of two tests using the same SCT loading. The first test involved heating the SCT at 690 K which resulted in a slow release of hydrogen, whereas the second test was run at 710 K at which temperature the hydrogen was released rapidly. The results of these tests and the others are discussed in the following sections.

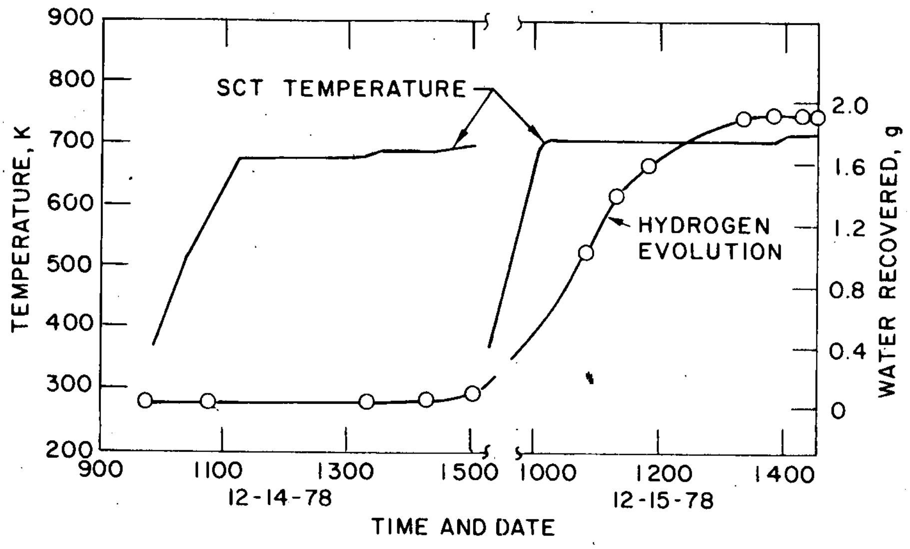

Fig. 5. Hydrogen Evolution Observed during Tests 2a and 2b with SCT Number 2

## IV. RESULTS AND DISCUSSION

The hydrogen evolution rates and the percent recovery of hydrogen from the SCT are summarized in Table 2 for each of the tests of this series. Tests number 2, 3, and 4 were done in several steps using the same impurity loading in a single SCT. The results of tests 2a and 2b are shown in Fig. 5 above, and tests 3a, 3b, 4a, 4b, 4c, and 4d were done in a similar manner. The temperature was raised in each test in the sequence and the hydrogen evolution rate was measured at each temperature.

Table 2. Summary of Cold-Trap Unloading Test Results

| Test No. | Test Temp, K | Test Pressure, kPa | Hydrogen Loaded (as NaH), mg H | Hydrogen Recovery, % | Hydrogen Evolution Rate, μg/s |
|---|---|---|---|---|---|
| 1 | 718 | 100 | 257 | 95.4 | 56.9 |
| 2a | 693 | 100 | 232 | | 0.833 |
| 2b | 710 | 100 | 232 | 90.3 | 17.8 |
| 3a | 523 | 0 | 159 | | 0.142 |
| 3b | 598 | 0 | 159 | 72.1 | 2.47 |
| 4a | 543 | 0 | 87 | | 0.042 |
| 4b | 568 | 0 | 87 | | 0.044 |
| 4c | 588 | 0 | 87 | | 0.089 |
| 4d | 603 | 0 | 87 | 97.3 | 0.142 |

The results presented in Table 2 may be divided into three groups: tests 1, 2a, and 2b which were run at atmospheric pressure, tests 3a and 3b in which NaH was decomposed under vacuum, and tests 4a through 4d in which NaH in the presence of $Na_2O$ was decomposed under vacuum. The hydrogen evolution rates for these three groups are plotted *vs.* temperature in Fig. 6. Although straight lines are drawn through these data points, the amount of information available is insufficient to determine the nature of the relationships. However, several observations can be made from these results. A significantly higher temperature is needed to evolve hydrogen at atmospheric pressure than under vacuum. This observation is consistent with the theory that the primary mechanism for rapid hydrogen evolution is by bubble nucleation, growth, and release. Hydrogen evolution at 3 μg/s is arbitrarily considered "rapid" in these tests because it is ∿20 times the rate calculated for simple diffusion. The temperature difference between hydrogen evolution at 3 μg/s under atmospheric pressure and the same rate under vacuum was found to be 100 K. This temperature difference corresponds (in Fig. 1) to a critical

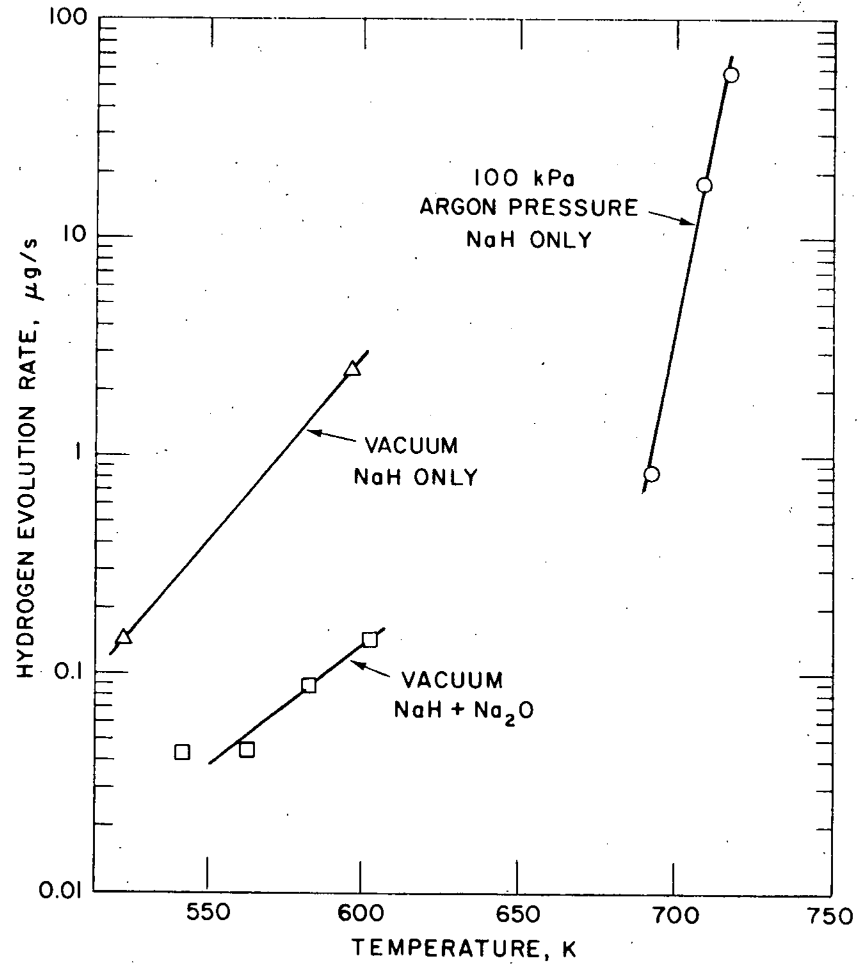

Fig. 6. Hydrogen Evolution Rates Observed in All Tests

bubble radius of between 50 and 100 μm. Although the bubble nucleation temperatures shown in Fig. 1 (670 and 570 K for a 75-μm critical radius and atmospheric pressure and vacuum, respectively) do not agree exactly with the observed temperatures shown in Fig. 6 (700 and 600 K for a hydrogen evolution rate of 3 μg/s), the agreement is better than would be expected for this simple analysis.

Another important observation is that the presence of $Na_2O$ appears to inhibit the hydrogen evolution rate by as much as one order of magnitude. This reduction of evolution rate is probably due to interaction of the hydrogen and oxygen species either in solution, or at interfaces between phases. The data obtained in this study are insufficient to determine the mechanisms of this interaction, and more work is needed in this area. This interaction could have a significant impact on the time required to regenerate cold traps that have large quantities of $Na_2O$. However, IHTS cold traps should contain relatively little $Na_2O$, so the regeneration rate should be acceptable in the IHTS case.

Little significance should be given to the relatively low hydrogen recovery achieved from SCT number 3. The emphasis of the test was on determination of the maximum rate of hydrogen evolution rather than on total hydrogen recovery. The test was terminated before all the hydrogen had been evolved.

Corrosion tabs were present in the bottom of SCT's 2, 3, and 4. Post-test examination of these tabs showed very little visible effect of exposure to the test environment. The post-test appearance was very slightly duller than the initial appearance; however, weight losses were not detectable beyond the normal handling, cleaning, and weighing errors.

## V. CONCLUSIONS AND FUTURE DIRECTIONS

Although this work was preliminary in nature, several significant conclusions may be drawn from the results:

1) Thermal decomposition of NaH is an effective method for removal of NaH from sodium in a configuration similar to a cold trap.

2) Effective decomposition of NaH can be achieved either at one atmosphere pressure using a sweep gas or under vacuum.

3) The hydrogen evolution rate becomes significant (>3 μg/s in these tests) at temperatures ∿100 K lower when the vacuum method, rather than the sweep-gas method, is used.

4) The presence of $Na_2O$ significantly reduces the rate of hydrogen evolution from NaH.

5) The NaH decomposition method should be suitable for regenerating IHTS cold traps because the deposit should be primarily NaH with little $Na_2O$ present.

The NaH decomposition method for cold-trap regeneration--particularly under vacuum--appears attractive for regeneration of IHTS cold traps. Additional studies are being conducted to more completely establish the relationships between temperature, pressure, and hydrogen flux; to establish the effect of $Na_2O$ on the hydrogen flux; to determine the impact of tritium on the equipment design and operating requirements; and to test the NaH decomposition method on a circulating sodium system.

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