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SEPTEMBER 30, 1971

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EXAMINATION
OF
LMEC'S T-1D TEST FILTERS
FOR
PARTICULATE IMPURITIES

HANFORD ENGINEERING DEVELOPMENT LABORATORY

UNITED STATES ATOMIC ENERGY COMMISSION

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**EXAMINATION OF LMEC'S T-1D TEST FILTERS
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W. O. Greenhalgh

September 30, 1971

**HANFORD ENGINEERING DEVELOPMENT LABORATORY
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EXAMINATION OF LMEC'S T-1D FILTERS FOR PARTICULATE IMPURITIES

W. O. Greenhalgh

INTRODUCTION

Methods for the analysis of sodium impurities are undergoing tests in preparation for the operation of the Fast Flux Test Facility (FFTF). Several analytical methods have been standardized and the procedures detailed in the latest Interim Standards Methods Manual.⁽¹⁾ The basic concept of the approach to particulate identification has been outlined in ANL/ST4.⁽²⁾ Details of the standard particulate analyses have not been defined and experience with the outlined general procedure has been limited. An examination of T-1D filters was undertaken to evaluate the particulate identification capability in place at Hanford Engineering Development Laboratory (HEDL), to determine weaknesses in the procedures, to provide information about the sodium system in which the filters were operated, and to provide experience in the handling and requalification of heavy sodium hardware. All analytical operations performed were consistent with the available Argonne National Laboratory (ANL) guidelines.

The characterization of the particulates within a sodium reactor system is important in several aspects: (1) A particulate analysis can provide information about corrosion processes in the system. (2) Particulate analyses should detect any impurity that is present in excess of its sodium solubility. (3) Particulate analyses can provide information about material transport phenomena. (4) Particulate analyses can provide information about fuel element failure and help define the extent of wash out.

SUMMARY

A T-1D filter from the Large Components Test Loop (LCTL) has been disassembled, the particulate matter contained on the filter examined and the vessel requalified for sodium service. A reasonable (one month maximum) turn around time for the filters has been demonstrated. The applicability of HEDL particulate examination procedures to a large filter has been demonstrated and areas for improvement in handling technique have been defined.

The particulates identified were primarily stainless steel fragments of regular geometry the source of which is postulated to be corrosion or mass transport phenomena within the system. Non-stainless steel particulates were found to contain unexpectedly high concentrations of calcium and silicon. The amount of particulate matter on the filter was found to be quite small, probably due to the inadvertent back flush of the filter with sodium during removal from the system.

HARDWARE DESCRIPTION AND HANDLING PROCEDURES

The T-1D test filters were located in the LCTL operated by the Liquid Metal Engineering Center (LMEC). The filters were positioned at the head end of the FFTF T-1B erosion test modules. The filters had been in operation continuously for six months at a service temperature of 750 °F. An earlier set of similar filters was examined at LMEC and the results of the examination have been reported.⁽³⁾

The vessel containing the filters is 33.5 inches long and 12.75 inches in diameter. Figure 1 shows the size of the vessel by comparison with the 2-foot wide handling cart. It contains nine filter elements mounted in 1-1/2 inch plate. Each element is 18 inches long and is 3 inches in diameter. The filters are sintered stainless steel and they have an average pore size of 20 microns. The weight of the as-received vessel was about 175 lbs. Approximately 50 lbs of this weight was internal hardware and sodium. In preparation for opening the vessel, the locations to be cut were outlined and machined to a depth of 9/32 of an inch. The last 3/32 of an inch was cut inside a high purity inert atmosphere glove box by hand using an ordinary hacksaw. The cuts were made on each side of the filter mounting plate (see Figures 2 and 3) and each filter element was cut off. The elements as they were removed in the glove box were placed in large plastic bags to keep them clean. Figure 4 shows the vessel just after it was cut open. It can be seen that the residual sodium covered three of the filters. These were removed with the sodium intact so that any reactive particulate material present would be protected by the sodium until the sampling could be carried out. It should be pointed out that sodium was layered in the bottom of the vessel as well as layered up the side around the three filter elements as illustrated in Figure 2. We could not hypothesize a mechanism which would account for this unusual configuration of the sodium.

FIGURE 1. T-ID FILTER VESSEL

FIGURE 2. DIAGRAM OF THE T-ID FILTER VESSEL

FIGURE 3. T-ID FILTER VESSEL MACHINE CUTS

FIGURE 4. T-ID FILTER VESSEL OPENING

Making a cut through stainless steel hardware containing sodium with an ordinary hacksaw was found to be difficult at best. The sodium is sticky and tends to gum up the sides and teeth of the saw blade. Although the manual technique employed was successful, a better technique using a power-driven cutter needs to be developed. Use of a Saw Zall fitted with coarse blades has been considered but has not been tested on sodium-exposed hardware. Figure 5 shows the relationship of the size of the vessel to that of the front half section of the inert glove box. Obviously space problems will have to be taken into consideration during the development of techniques to use power equipment. Future glove boxes set up for this type of analysis should have a larger air lock chamber preferably 36 inches in length and possibly a larger diameter. The present air lock is only 20 inches long and has a 15 inch inside diameter.

Frequently it is desirable to disassemble, sample and requalify a system filter during a maintenance shutdown of limited duration. The organization examining the filter should therefore be able to provide a turn around time of less than a month. In this regard, agreement was made with LMEC whereby HEDL would receive the filter, open it, remove the filters and sodium, clean it, refit the vessel, reweld it, recertify it for sodium service, and ship it back to LMEC within about a month. HEDL's commitments to LMEC were met except for a delay of approximately 10 days due to unexpected problems in the requalification of one of the original LMEC components. The vessel arrived Friday, February 26, 1971, and was loaded into the sodium glove box the first part of the following week. The vessel was removed from the glove box and cleaned by March 15, and it was scheduled to be refitted and shipped by March 22. J. Yount was the engineer in charge of handling and refitting the vessel. The operation demonstrated that a large filter vessel could be removed from service, sampled, and returned to service in the relatively short time of about a month. The turn around time can undoubtedly be improved and possibly cut in half by perfecting the developed techniques.

SAMPLING AND ANALYSES

Particulate samples taken prior to this time have generally been collected by a small in-line sintered stainless steel filter designed for 1/2-inch tubing and measuring less than an inch in diameter. For nondestructive analyses such as electron microscopy the filters were mounted directly. The T-1D filters, however, are enormous in comparison so a new technique of

FIGURE 5. THE T-ID FILTER VESSEL INSIDE IN THE INERT-GAS GLOVE BOX

sampling was required. This sampling technique employed a 1-1/4 inch steel hole saw. Figure 6 shows the results of the core sampling that was used. A 1-1/4 inch round hole saw blade was attached to a 1/4 inch bit and a 3/8 inch portable drill was used to core out the sample. The rounded core sample was flattened by a light tap with a hammer using the smooth edge of a cold steel chisel. The chisel left a small thin groove, but did not otherwise damage or contaminate the sample. Saw chips were surprisingly well-contained in the sodium immediately adjacent to the cut and fewer precautions than anticipated were required to prevent contamination of the sample. Saw chips were quite characteristic in appearance and could readily be distinguished from particulate matter originally present in the sample.

Sodium metal samples were taken from the bulk material by scraping off all the exposed sodium surface and gouging out a chunk with a clean all steel tool (spatula, flat chisel, etc.). Samples taken prior to the removal of the filter vessel from the glove box could not be kept free of moisture due to large surface area of the vessel. The vessel was longer than the airlock and it had been passed directly from the air into the box atmosphere without being evacuated. Once the vessel was removed, sodium samples could be taken with minimum moisture contamination.

Individual particles and compounds that were located or collected were identified by electron microscopy, X-ray fluorescence and X-ray diffraction. The electron microscopic technique was used to locate individual particles and photograph their geometric shape. The X-ray fluorescent attachment was then used to identify the elements present in the individual particle except for elements lighter than aluminum. The X-ray diffraction analysis was used to identify the compounds present both in material that could be collected from the filter surface and in the residue from the distillation of the sodium sample.

The filter core samples for the electron microscope instrument were mounted directly on an aluminum block sample stage inside the glove box. They were loaded into the sealed transfer cell shown in Figure 7. Samples were transferred out of the box and into the electron microscope with no noticeable contamination. For X-ray diffraction work, the surface of the filter was carefully scraped inside the inert box to collect the material.

FIGURE 6. CORE SAMPLED T-ID FILTER ELEMENTS

FIGURE 7. ELECTRON MICROSCOPE TRANSFER CELL

This was placed into thin wall capillaries that had been sealed on one end. Upon loading, a small quantity of grease was put over the capillary as a temporary seal until the capillary could be removed from the box and flame sealed. Several filter surfaces were also examined by visual microscopy but the technique did not prove too valuable because of the very small size of the crystals present.

The other analyses made were carried out on the sodium left in the vessel, or a distillation residue of that sodium. Atomic absorption and emission spectrographic analyses were carried out on distillation residues. Total carbon and carbon species determinations were made on the sodium metal. And using ordinary microscopic techniques, a statistical analysis of the size distribution of particles in a 3-gram sample was made.

RESULTS

Filter Surface Analyses

Electron microscope and X-ray probe analyses showed the particulates present on the filter surface to be primarily stainless steel in the form of regular shaped platelets. (See Figure 8-A.) These platelets do not resemble either the filter surface or saw shavings and have apparently resulted from corrosion or mass transport processes within the system.

The only other elements detected in the particulate impurities in significant concentrations were calcium and silicon. Figures 8-B and 8-C show some of the particulates containing these elements. The source of these particulates is not known, but the calcium might have come from the sodium feed stock. The silicon was probably leached out of the other steel loop components.

X-ray diffraction analyses identified Na_2O , $\text{NaOH}\cdot\text{H}_2\text{O}$ (probably resulting from atmospheric contamination of the sample), Na_2SiO_3 , CaSiO_3 . There was some indication of iron carbides and other compounds in the calcium-silicate group. Visual microscopy showed a thin scattered layer of oxide and hydroxide compounds, but sample resolution was poor so efforts were not vigorously pursued with the microscope.

Sodium Analyses

The emission spectrographic analysis is shown in Table 1. Aluminum, calcium, iron, magnesium, lead, silicon, and tin are the major impurities.

A. STAINLESS STEEL PLATELETS

B. CALCIUM SILICATE PARTICULATE

C. SILICATE PARTICULATE 1000X

FIGURE 8. PARTICULATES

TABLE I
SPECTROCHEMICAL ANALYSIS REPORT

<u>Element</u>		<u>Element</u>		<u>Element</u>		<u>Element</u>	
Ag	T	Hg	--	Re		Dy	
Al	M	In	--	Rh	--	Er	
As	--	Ir	--	Ru	--	Eu	
Au	--	K		Sb	--	Gd	
B	T	La	--	Sc		Ho	
Ba		Li		Si	M	Lu	
Be	--	Mg	T-M	Sn	M	Nd	
Bi	--	Mn	T	Sr	--	Pr	
Ca	T-M	Mo	<T	Ta	?	Sm	
Cd	--	Na	M-S	Th	--	Tb	
Ce		Nb	--	Ti	<T	Tm	
Co	--	Ni	T	Tl	--	Yb	
Cr	T	Os		U	--		
Cs		P	--	V	--		
Cu	T	Pb	T-M	W	--		
Fe	M	Pd	--	Y	--		
Ga	--	Pt	--	Zn	--		
Ge		Pu		Zr	--		
Hf	--	Rb					

KEY FOR QUALITATIVE ANALYSES

<u>Symbol</u>	<u>Meaning</u>	<u>App'x. Conc.</u>
SS	Major detectable constituent	
S	Strong	Greater than 1%
M	Moderate	1% to 0.01%
T	Trace	Less than 0.01%
--	Not detected	
*	Interference	
?	Detection uncertain, interference	

These results are only semiquantitative in nature. Atomic absorption data showed the impurity concentration to be approximately 10 ppm for iron, 0.3 ppm for magnesium, and 1 ppm for calcium with an acid insoluble residue of 15 ppm containing chromium, nickel, and iron. The atomic absorption sample was obtained from the bulk sodium near the bottom of the vessel. A sample from the more typical sodium showed 4 ppm iron, 0.2 ppm magnesium; and lead, tin, and calcium were below the detection limit. There was a small portion of the distillation residue that wouldn't dissolve in aqua regia. A carbon analysis of the sodium by total combustion⁽⁴⁾ indicated 5 ppm carbon was present. A carbon specie analysis indicated 2 to 3 ppm total carbon with about half of the total being carbonate.

A statistical analysis of the particulates was successful with aqueous insoluble solids. The size distribution of the particulates in the sodium sample analyzed are given below and the results are extrapolated to give an estimated distribution per pound of sodium.

<u>Particle Size</u> <u>(μ)</u>	<u>Total Count/3 gm</u>	<u>Count/lb of Sodium</u> <u>(estimate)</u>
5-15	980	140,000
15-25	170	24,000
25-50	90	13,000
50-100	18	2,600
> 100	8	1,100

Comparison of particulate size distribution data with similar data from other sodium sources would be of interest, but such data is not available at the present time.

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