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HEME AND HEPA FILTER ELEMENT DISSOLUTION PROCESS (U)

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HEME AND HEPA FILTER ELEMENT DISSOLUTION PROCESS (U)

SUMMARY

High Efficiency Mist Eliminators (HEME) and High Efficiency Particulate Airfilters (HEPA) are to be used in the Defense Waste Processing Facility (DWPF) to remove volatile and semi-volatile effluents from the off-gases generated during the vitrification process. When removed, these filters are likely to contain radioactive contaminants, organics, and hazardous materials, which make their disposal by normal methods impractical. Hence, an alternative disposal method is needed.

The alternative disposal method evaluated in this study is dissolution of the filters with caustic and acid solutions. Dissolution converts the waste into an aqueous stream, which can be transferred to the Tank Farm and disposed of by normal means. This process was shown to be effective on a small scale in earlier studies, but the results were not well documented and the studies were not performed on fouled filters.

The previously studied dissolution process, along with some potential improvements, was tested on clean and fouled filter samples. Although the process was effective in breaking down small samples (one inch cubes), its effectiveness diminished for larger sized samples. For larger samples, the process tended to break down the filter elements, but the pieces were not small enough for easy transfer. An experiment imitating the conditions of the Decon Waste Treatment Tank (DWTT), while also not completely successful, produced a particle free solution, with the 24% remaining filter encased in the filter basket.

Though complete dissolution was not achieved for every test condition, filter dissolution utilizing 48 hours of 5% NaOH treatment, 96 hours of 5% HNO₃ treatment, and 4 hours of 5% NaOH treatment at near boiling temperatures seems to be a viable dissolution process if there is sufficient agitation. A larger scale test using the DWTT processing conditions is recommended.

INTRODUCTION

The DWPF vitrification process will generate off-gases, which contain significant quantities of radioactive Cs-137, Sr-90, and TRU contaminants.¹ HEME and HEPA filters are used in the off-gas system to remove these contaminants. The HEME is responsible for removing approximately 98% of the Sr-90 and 83% of the TRU contaminants, while

the HEPA removes virtually all of the Cs-137 and approximately 17% of the TRU contaminants.²

These filters will probably need changing on an annual basis, and since the filters contain high levels of contamination, they cannot be disposed of by normal methods. In order to dispose of the filters and minimize solid waste generation, DWPF decided that it would be best to try and break down the filters and send them to the Tank Farm as an aqueous stream.

To achieve this end, a dissolution process using 48 hours of 5% NaOH treatment, 96 hours of 5% HNO₃ treatment, and a final 4 hours of 5% NaOH treatment at near boiling temperatures was developed by J.L. Bransford.³ His process was derived from preliminary work done by B.A. Daugherty, who used 5% NaOH to dissolve the filters. Daugherty's process was not completely successful because of the soluble and insoluble reaction products that remained in solution.⁴ Bransford's experiments proved sufficient on a small scale, but information on the post-digestion solutions and products was not documented.

Analysis of the dissolution products was performed by C. M. Jantzen. She concluded that the complete caustic-acid-caustic dissolution process was necessary because insoluble zeolite reaction products remained after the first caustic treatment, which could only be dissolved by using the acid treatment. She further concluded that only dissolved amorphous silica remained after the acid dissolution step and the final caustic step was only necessary to adjust the pH for recycle to the Tank Farm.⁵

While Bransford's dissolution process was sufficient in breaking down the filters, it was only performed on clean filter elements, and thus the effects of foulants on the filter dissolution process were not known. Recent experience with fouling of the HEME at TNX indicated that organic materials from the hydrolysis of tetraphenylborate could coat the filter elements, possibly making dissolution more difficult, and resulting in difficult to control reactions.⁶ The ability to transfer the aqueous waste stream, from a chemical component and physical characteristic standpoint, also had to be proven. Therefore, demonstrations with filter elements which had been exposed to processing conditions were needed.

The scope of this study was to duplicate the previous experiments and determine if the process would effectively dissolve a fouled filter. In addition, the chemical composition and important physical properties of the products of the dissolution were to be documented. Analyses of the components of the dissolution solutions aided in determining how well the filters were dissolved.

In order to meet all the requirements of the task assignment, numerous experiments were performed in this study. A clean HEPA, fouled HEPA, and fouled HEME were obtained for the work. The HEPAs are supplied by Flanders Filters Inc. and are representative of those that will be used

in the DWPF. The size of the filter fibers range from 0.33 to 10 microns. Their analyzed composition, along with the manufacturer supplied composition, is shown in Table 1. The fouled HEPA was removed from the Integrated DWPF Melter System (IDMS) where it was used during several campaigns.

Table 1 - HEPA Filter Composition

Oxides	Flanders Comp. (wt%)	Clean Filter (wt%)	Fouled Filter (wt%)
Al ₂ O ₃	5.0 - 7.0	5.282	4.439
B ₂ O ₃	9.0 - 11.0	8.745	8.039
BaO	2.5 - 5.5	3.412	3.712
CaO	2.0 - 4.0	1.100	0.809
Cr ₂ O ₃		<0.006	0.802
Fe ₂ O ₃	0.0 - 0.3	<0.003	2.422
K ₂ O	1.5 - 3.0	2.344	3.446
Li ₂ O		0.038	0.225
MgO	0.0 - 2.0	0.776	0.310
Na ₂ O	10.0 - 12.0	9.938	10.789
NiO ₂		<0.006	0.472
SiO ₂	57.0 - 63.0	57.438	46.089
ZnO	0.0 - 4.5	2.882	2.800
Other Species			
H ₂ O		4.697	5.308
Hg		0.000	0.061
Cl		0.151	0.507
F		0.656	0.678
F ₂	0.0 - 0.8	1.311	1.356
NO ₃		0.519	6.678
SO ₄		0.614	1.058
Totals		100.002	100.000

The fouled HEME was supplied by Monsanto Environmental Chemistry and is also representative of the DWPF filters. It is composed of loose 7 micron to 20 micron fibers surrounded by a woven mat of 16 micron to 80 micron fibers. The composition was not supplied by Monsanto for proprietary reasons, but its analyzed composition is shown in Table 2. The HEME used in these experiments and the HEMEs to be used in the DWPF contain less CaO, but more Na₂O and K₂O than those studied in Bransford's experiments. It was removed from the IDMS after being located in the Formic Acid Vapor Condenser (FAVC) system. A clean HEME was not obtained for these studies due to the long procurement time and high cost, but it is likely that the fouled HEME will be more difficult to digest.

Table 2 - HEME Filter Composition

Oxides	Clean Filter (wt%)	Fouled Filter (wt%)
Al ₂ O ₃	3.972	4.358
B ₂ O ₃	4.859	5.424
BaO	0.208	0.295
CaO	2.096	2.033
K ₂ O	0.920	0.685
Li ₂ O	0.037	0.019
MgO	2.995	3.075
Na ₂ O	18.510	15.392
SiO ₂	63.771	63.502
ZnO	0.013	0.007
Other Species		
H ₂ O	2.120	2.950
Hg	0.000	0.056
Cl	0.255	0.267
F	0.048	0.031
NO ₃	<0.300	2.578
SO ₄	0.195	0.182
Totals	99.999	99.999

EXPERIMENTAL

All experiments were performed using procedure GTOP-3-055, Revision 0 or 1.^{7,8} Deviations from the procedure were noted on the data sheets, which can be found in WSRC-NB-92-39. Solutions were sampled for analyses and physical property determination after each processing step. These analyses are described in WSRC-RD-92-01.⁹ The viscosity and water loss of the final solutions were recorded, as well as composition of the final solid components.

For ease in recognizing experiments and samples, a sample identification system was developed. The sample number was of the form FDT-XX-Y-Z, where the FDT represents Filter Dissolution Task, the XX represents the type of filter (CP-Clean HEPA, FP-Fouled HEPA, and FM-Fouled HEME), Y represents the experiment trial number (from each filter), and Z represents the processing step (this applies only to samples, not experiments, the S stands for the remaining solid residue). All dissolution experiments and parameters are identified in Table 3 using this nomenclature.

Table 3 - Sample Numbers

Caustic-Acid-Caustic Experiments

Number	Size	Mass	Initial Vol.	Additional Information
FDT-CP-3	1" Cube	0.464 g	111 ml	SA/V = 0.35 cm ⁻¹
FDT-FP-3	1" Cube	0.765 g	111 ml	SA/V = 0.35 cm ⁻¹
FDT-FM-3	1" Cube	2.817 g	111 ml	1st Fouled Section, SA/V 0.35 cm ⁻¹
FDT-FM-4	1" Cube	3.477 g	111 ml	2nd Fouled Section, SA/V 0.35 cm ⁻¹
FDT-FM-5	1" Cube	3.447 g	111 ml	3rd Fouled Section, SA/V 0.35 cm ⁻¹
FDT-CP-7	1" Cube	0.810 g	430 ml	SA/V = 0.09 cm ⁻¹
FDT-FP-5	1" Cube	0.734 g	430 ml	SA/V = 0.09 cm ⁻¹ , Coupon Used
FDT-FM-7	1" Cube	3.384 g	430 ml	SA/V = 0.09 cm ⁻¹ , Coupon Used
FDT-FP-6	3" Cube	19.048 g	4000 ml	SA/V = 0.09 cm ⁻¹
FDT-FM-8	3" Cube	37.601 g	4000 ml	SA/V = 0.09 cm ⁻¹
FDT-FP-7	6" Cube	27.775 g	3981 ml	SA/V = 0.35 cm ⁻¹
FDT-FM-9	6" Cube	318.78 g	3981 ml	SA/V = 0.35 cm ⁻¹

C-A-C with Decanting Experiments

Number	Size	Mass	Initial Vol.	Additional Information
FDT-CP-8	1" Cube	0.764 g	430 ml	SA/V was maintained between steps
FDT-FP-9	1" Cube	0.881 g	430 ml	SA/V was maintained between steps
FDT-FM-11	1" Cube	2.976 g	430 ml	SA/V was maintained between steps

C-A-C with Air Sparging Experiments

Number	Size	Mass	Initial Vol.	Additional Information
FDT-CP-5	1" Cube	1.301 g	430 ml	Air Sparge Rate > 8.55 e ⁻² cfm
FDT-FP-8	1" Cube	1.442 g	430 ml	Air Sparge Rate > 8.55 e ⁻² cfm
FDT-FM-10	1" Cube	3.386 g	430 ml	Air Sparge Rate > 8.55 e ⁻² cfm

Acid-Caustic Experiments

Number	Size	Mass	Initial Vol.	Additional Information
FDT-CP-6	1" Cube	1.226 g	430 ml	96 hrs acid & 4 hrs caustic
FDT-FP-10	1" Cube	1.067 g	430 ml	72 hrs acid & 72 hrs caustic
FDT-FM-12	1" Cube	2.214 g	430 ml	72 hrs acid & 72 hrs caustic
FDT-FP-11	1" Cube	1.104 g	430 ml	96 hrs acid & 48 hrs caustic
FDT-FM-13	1" Cube	2.182 g	430 ml	96 hrs acid & 48 hrs caustic

Large Scale Experimentation

Number	Size	Mass	Initial Vol.	Additional Information
FDT-FM-14	6" Cube	446.7 g	43.5 liters	Performed in MFT, Air Sparge Rate = 0.064 cfm

DWTT Mock-up Experiment

Number	Size	Mass	Initial Vol.	Additional Information
FDT-FM-15	6" Ring	72.259 g	4 liters	6" OD, 1.5" thick, 3" H, Air Sparge Rate = 0.065 cfm

Caustic-Acid-Caustic Dissolutions

Bransford's dissolution processing conditions of 48 hours of 5% NaOH treatment, 96 hours of 5% HNO₃ treatment, and 4 hours of 5% NaOH treatment were used as the basis for all experiments. This process was tested on one inch, three inch, and six inch cubes of filter element. While the one inch cube tests were performed with all types of filters, the three and six inch tests were only performed with the fouled filters, being that they represented the worst case scenarios. Different sized samples were tested so the scale-up potential could be determined.

Various surface area to volume (SA/V) ratios were tested with Bransford's process so that the smallest amount of dissolution solution needed could be determined to ensure waste minimization. The main two ratios used in the experiments were 0.35 and 0.09 cm⁻¹. The 0.35 cm⁻¹ SA/V ratio was based on past leaching experiments and the 0.09 cm⁻¹ SA/V ratio was based on Bransford's experiments.

Both stainless steel and teflon vessels were used in the experiments. The purpose was to allow for comparison of the effects of the caustic, acid, and organics on the vessel walls, as well as to help define the species that will dissipate into the solution. Coupons made of 304 L stainless steel were also used to help determine the extent of corrosion on the metal, so that any precautions necessary for the DWPF could be determined. As mentioned earlier, the DWPF will be using a stainless steel tank, the DWTT, to perform their dissolutions.

Caustic-Acid-Caustic Dissolutions with Decanting

Decanting was used with Bransford's dissolution process to determine any benefits. The rationale behind this approach was to rid the processing vessel of the saturated solution, so further dissolution would be possible. In these tests, the remaining dissolution solutions were decanted off after each dissolution step.

The decanting experiments were performed on one inch cubes of all filter types, using the 0.09 cm⁻¹ SA/V ratio. This ratio was maintained every time solution was added.

Caustic-Acid-Caustic Dissolutions with Air Sparging

The benefits of air sparging with Bransford's dissolution process were examined. The DWTT does not have the ability to agitate when filters are being dissolved; however, it does have a permanently installed air sparge ring, which can serve as an agitator.

In the lab scale experiments, teflon hosing connected to an air supply line and a flowmeter was used to simulate air sparging. The air sparging experiments were performed on one inch cubes of all filter

types, using the $0.09 \text{ cm}^{-1} \text{ SA/V}$ ratio. The teflon vessels were used, and the sparge line was inserted through a septum in the lids.

Acid-Caustic Dissolutions

Experiments were performed using the acid and final caustic step of Bransford's dissolution process. The benefits to be gained were time and waste minimization. The main purpose of the acid step in Bransford's process, according to Jantzen, was to break down the zeolite products that remained after the initial caustic step; therefore if the acid were strong enough to perform the dissolution alone, then time and waste minimization could be achieved.

These experiments were initially performed on an one inch cube of the clean HEPA for a total of 100 hours, which is equal to the time of the last two steps in Bransford's process. The experiments were later performed on one inch cubes of the fouled HEPA and HEME for 144 hours, which is equal to the duration of Bransford's process minus the 4 hours required for conversion to a basic solution. The last 4 hours would no longer be necessary since the solution would be basic already.

Effluent Penetration Depth

A vertical section of the fouled HEME was removed and divided into three sections to determine what effect, if any, the degree of effluent penetration had on the dissolution process. Each section was dissolved using the $0.35 \text{ cm}^{-1} \text{ SA/V}$ ratio in the stainless steel Parr bombs. These experiments were performed at the same time as the caustic-acid-caustic dissolution experiments.

Large Scale Dissolution

In order to ensure that the process would suffice on a larger scale, a dissolution experiment with a six inch cube of the fouled HEME was performed in the 774-A Melter Feed Tank (MFT). The MFT provided adequate space and volume capacity to better simulate the processing conditions of the DWTT.

The SA/V ratio for this experiment was 0.032 cm^{-1} , which is still 10 times less solution than the ratio to be seen in the DWTT. An air sparge tube was also used in this experiment with a flow rate of 0.064 cfm, which is proportional to the flow rate to vessel volume of the DWTT. An immersion heater was used as the heat source for the experiments.

DWTT Mock-up Dissolution

To more accurately reflect the DWTT dissolution conditions, an experiment was performed with the fouled HEME encased in a stainless steel "basket" and utilizing an air sparge ring. When the filters are dissolved in the DWTT, they will still be in their filter housings and

will be placed in a basket that is lowered into the DWTT. Hence, the effects of having the filter concentrated in one place, where the solution would be not accessible to all sides, had to be studied.

The filter sample for this experiment was not a cube, but rather a 1 1/2" thick, 3" high, and 6" outer diameter ring of filter, reflecting the actual design of the HEME. A 1/4 scale ring air sparger was used to represent the DWTT's sparger. This sparge ring was made of teflon tubing with holes drilled in the tubing, and this in turn was connected to an air supply line. The sparge ring rested on the bottom of the vessel, similar in design to the DWTT. The volume of solution was limited by the vessel volume, the SA/V ratio was 0.205 cm^{-1} .

Triton

The effects of the chemical surfactant Triton on cleaning and dissolving the fouled HEME filter were studied. This chemical, when used with KOH, had proven satisfactory in destroying organics in some previous TNX experiments.

Experiments were performed on small samples of the HEME filter using 3000 ppm Triton with 5% NaOH and 5% KOH in beakers on a hot plate. The solution and remaining filter for each sample were chemically analyzed. In addition, the final weight loss for each sample was calculated to see how well the solutions dissolved the filters.

RESULTS AND DISCUSSION

The effectiveness of the dissolutions was determined from how well the fibers were dissolved. Additional insight was provided from the analyses of the dissolution solutions and the dissolution products that remained. Analyses of the remaining filter products were only performed, when a sizeable quantity of filter remained.

When Bransford's products were analyzed, Jantzen found that the residue remaining after the first caustic step was a Na-zeolite of the Linde B₁ type, with a composition of $0.95\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 3.35\text{SiO}_2 \cdot 4.79\text{H}_2\text{O}$. Zeolites of this structure tend to incorporate Ca, K, and Mg; thus, by monitoring their presence in the dissolution solution, the completeness of the acid dissolution could be monitored. Jantzen noted that this zeolite was completely dissolved by the end of the acid step, and all that remained was an amorphous silicate based gel. After the final four hours of caustic treatment, the main crystalline materials detected were NaNO_3 and NaNO_2 , indicating thorough breakdown of the filter material.⁵

The appearance of Bransford's observed products, as well as the filter material components, were monitored in these studies for confirmation of filter dissolution.

Caustic-Acid-Caustic Dissolutions

Bransford's reference process proved effective on dissolving one inch cubes of the clean HEPA (FDT-CP-7), fouled HEPA (FDT-FP-5), and the fouled HEME (FDT-FM-7) using a SA/V ratio of 0.09 cm^{-1} . The first caustic treatment caused the elements to break down into much smaller pieces, which were dissolved even further by the conclusion of the acid treatment. Only a few very small filter particles remained settled at the bottom of the vessels by the end of the tests.

Bransford's process, however, did not dissolve as completely when a 0.35 cm^{-1} SA/V ratio was used. The HEPA (FDT-CP-3 and FDT-FP-3) filter cubes dissolved more than the HEME (FDT-FM-3) did, but pieces of the filters remained undissolved in all experiments at this ratio.

Though the smaller volume of solution failed to entirely dissolve the filter elements, this does not present an obstacle to the DWPF, since the DWTT will require a much smaller SA/V ratio. Approximately 6,000 gallons of solution will be required to operate the DWTT when dissolving the filters, due to the coil coverage requirements. This equates to a SA/V ratio of roughly 0.0032 cm^{-1} .

The caustic-acid-caustic dissolution process was further tested on three inch cube samples of the fouled HEPA (FDT-FP-6) and HEME (FDT-FM-8) filter using the 0.09 cm^{-1} SA/V ratio. These tests did not prove as successful as the one inch tests. Large pieces of the filter remained intact and were found floating on the solution surface, while small pieces had settled to the bottom of the vessel by the end of the experiment. Stirring between the steps did seem to help in the fouled HEME dissolution, but it was not significant enough to completely break down the filter.

To finish the dissolution scale-up experiments, the process was attempted on six inch cubes of the fouled HEPA (FDT-FP-7) and HEME (FDT-FM-9). A SA/V ratio of 0.35 cm^{-1} was utilized, due to vessel volume limitations. Both attempts resulted in large pieces of the filter remaining intact, with the large pieces once again floating on the surface. The solutions for each test were very saturated. The one important difference to note between the two filter dissolutions was the appearance of hard agglomerated matter during the HEME acid dissolution step. This substance conformed to the vessel walls and was difficult to break apart. It can probably be attributed to the small volume of the dissolution vessel and the small amount of dissolution solution available to effectively break down the filter. Stirring between the steps helped to break the large pieces into smaller pieces, but it did not completely break down the elements.

The chemical analyses results of the caustic-acid-caustic dissolution solutions are given in Attachment 1. The chemical analyses results for FDT-CP-7-1 indicate that the filter elements had started to dissolve during the initial caustic dissolution, as evidenced by the appearance

of Zn, Al, B, and Si in the solution. Results for FDT-CP-7-2 show that more of these elements, along with Ca, Mg, K, and Ba, were dissolved into solution during the acid treatment. This supports Jantzen's theory that the Ca and Mg that were incorporated in the zeolites are released in the acid step. The final elemental concentration results, FDT-CP-7-3, suggest that the primary solution components remaining are Na and NO_3 , which once again supports Jantzen's theory.

All of the elemental analyses for FDT-FP-5 and FDT-FM-7 were not available because of problems that occurred when the samples were analyzed. The results available do reflect the presence of the major filter components in the dissolution solutions, as seen with the clean HEPA filter cube. An observation from the fouled filter experiments not seen in the clean filter experiment was the behavior of mercury during dissolution. Most of the mercury dissolved from the filter during the acid dissolution step, as attested by the increase in mercury concentration in solution. By the completion of the tests, a large portion of the released mercury had evaporated off, which was verified by the decrease in mercury concentration of the final solution. This is important considering the toxicity of mercury. Overall, the fouled filter experiments had greater final concentrations of the filter components than the ones for the clean HEPA under the same conditions, but the physical evidence from the tests indicate that the filter cubes were broken down enough to be transferrable.

The XRD results indicate mostly the presence of sodium compounds, and not zeolites as seen in past experiments. The SEM results, however, indicate that the components of the zeolites were in solution, as well as large quantities of the filter material. The SEM results also support most of the findings of the XRD and chemical composition analyses.

The 0.35 cm^{-1} SA/V ratio dissolution results were similar to the 0.09 cm^{-1} SA/V ratio results, in that the largest amounts of filter components were released during the acid dissolution step. However, in general, the concentrations of the filter components in solution were greater during the 0.35 cm^{-1} SA/V ratio tests. Since the concentrations were higher, it would seem that the 0.35 cm^{-1} SA/V ratio test was more effective, but this was not the case. The most probable explanation is that the 0.35 cm^{-1} SA/V ratio solutions were too saturated to dissolve the filter any further.

The XRD results at this ratio did not indicate the presence of sodium silicate as seen at the 0.09 cm^{-1} SA/V ratio, which indicates that the dissolution was not as effective. On the other hand, the SEM results revealed that some of these sodium compounds were present, along with components of the filter, but in great of quantities as the 0.09 cm^{-1} SA/V ratio.

For the three inch cube tests, some of the data for FDT-FP-6 was not available, once again, due to problems during analyses. In general, the results indicate the same dissolution trend as mentioned above; however,

large quantities of the filter components remained at the end of dissolution, as can be seen from the high B, Si, K, and Al concentrations. Analyses of the remaining solid products revealed that most of the major filter components had not been dissolved.

When larger quantities of the filter were dissolved, more complex products appeared in the XRD and SEM results. The same basic compounds were identified as in the one inch cube tests, but compounds with more than two species began to appear. Less of the filter components were released freely into solution and CeF_3 started to form.

Review of the analytical results for the six inch cube tests lead to the same conclusions as those of the three inch tests. The largest dissolutions occurred during the second processing steps, and considerable amounts of the filters remained undissolved at completion of the experiments, as verified by the solid residue analyses. The major difference in the XRD and SEM results was that the six inch HEPA sample tended to form barium compounds as opposed to the more complex compounds seen for the six inch HEME tests and both three inch filter tests. The SEM results supported the XRD findings.

The corrosion potential of Bransford's dissolution process on stainless steel was investigated. The Materials Technology Section (MTS) examined the stainless steel parr bombs used in the first experiments and the 304 L coupons suspended in the teflon vessels and did not discover any signs of corrosion. From the literature search performed by MTS, they concluded that 304L was not susceptible to stress corrosion cracking in 5 wt% NaOH or 5 wt% HNO_3 at boiling temperatures.

Caustic-Acid-Caustic Dissolutions with Decanting

Decanting proved to be an effective method for dissolving the filter elements. The tests proved as successful as did caustic-acid-caustic dissolution alone. Very little, if any, of the filters remained intact by the end of dissolution. However, when using decanting, more solution is required for the overall process. Since a main goal of all waste management activities is to minimize the waste produced, this consideration must be taken into account when deciding on the best method for dissolution.

The analytical results for the decanting experiments are contained in Attachment 2. When the results from the clean filter decanting experiment (FDT-CP-8) are compared to the normal dissolution clean filter results (FDT-CP-7), the results from the first processing step are similar, as are the results from the second processing step with a few exceptions. Decanting dissolved less Zn, Al, B, Si, and K than the normal processing did during the second step. On account of this, the decanting final solution contained greater concentrations of the filter components. The final Na and NO_3 levels were much lower for the regular processing also.

Due to the fact that not all of the results are available for the fouled filter normal dissolutions, direct comparison to the decanting experiments is difficult. However, from the data that is available, the results indicate that the decanting experiments dissolved most of the filter components by the end of the dissolutions, whereas the normal processing final solutions still contained large concentrations of the filter components.

The combination of the decanting results from the clean and fouled filter experiments are not consistent when compared to the normal processing results. While results from the clean filter decanting experiments seemed worse than the normal processing results, the results from the fouled filter decanting experiments seemed much better. A possible explanation of this behavior might be that the processing solutions for the fouled filter experiments become saturated with the organics and other foulants and are unable to dissolve the filters further. By changing the solutions, as is done in decanting, more of the solution is free to break down the filter elements.

The XRD results were consistent for all of the decanting experiments. Sodium carbonate and silicate were identified in the caustic solutions, while sodium nitrate was identified in the acid solutions. The SEM results support the presence of these components as well as the filter materials identified in the chemical analyses.

Caustic-Acid-Caustic Dissolutions with Air Sparging

Experiments performed with air sparging proved very effective in breaking down the one inch cube samples. None of the filter elements remained intact at the end of the process and very little appeared in solution. However, one draw-back to this method was that the volume had to be constantly monitored, due to the evaporation from the dissolution vessels that occurred.

While these tests were successful, the flow rate to vessel volume ratio of the teflon vessels (0.327 cfm/gal) was much greater than the flow rate to vessel volume ratio of the DWTT (0.0022 cfm/gal). The larger ratio present in the teflon vessels would make dissolution much simpler, so this factor must be considered when determining the effectiveness of air sparging.

The analytical results for the air sparging experiments are contained in Attachment 3. Chemical analyses results for the first dissolution solutions are not available because most of the liquid had evaporated out of the vessels. For the clean HEPA with air sparging (FDT-CP-5), the acid dissolution step resulted in smaller amounts of filter components in solution than with the normal dissolution processing, with the exception of B, Na, and Si. Whereas the opposite effect held true for the final solution of the air sparging experiments, much more of the filter components remained in solution than with the normal process. These results do not support the actual physical evidence from the

experiment, since hardly any of the filter remained by the end of the experiment using air sparging.

Once again, the results for the fouled filter experiments (FDT-FP-8 and FDT-FM-10) seemed to be opposite of the clean filter results. When comparing the final solutions of the fouled filter air sparging experiments to the fouled filter normal processing experiments, the normal processing final solutions contained larger concentrations of the filter components in solution, with the exception of Na, B, K, and Si. The solid residue that did remain still contained some quantities of the filter components, but it was not a significant amount.

Sodium carbonate was easily identified in all of the XRD results for the air sparging experiments, perhaps due to the air that was able to get into the vessel. Sodium silicate was also identified, which indicates dissolution of the filter. The SEM results supported both the XRD and chemical analyses results.

Acid-Caustic Dissolutions

The initial acid-caustic dissolution was performed on a clean HEPA. While dissolution did not appear to occur until the caustic was added and pieces of the filter still remained at the end of the test, analytical results indicated that the filter had begun to dissolve during the acid test, and the releases were equivalent to those experienced in the caustic-acid-caustic dissolution process. This is consistent with Jantzen's results.⁵

Since the initial test duration was only 100 hours and the filter element had shown some signs of dissolution after the 4 hours of caustic treatment, it was decided to try the process for 144 hours on fouled filters. One set of tests consisted of 96 hours of acid and then 48 hours of caustic treatment, while the other set consisted of 72 hours of acid and then 72 hours of caustic treatment. For both time durations, the results were the same. The fouled HEPA elements had dissolved slightly and then settled to the bottom, while the undissolved pieces remained floating on the surface. The fouled HEME loose fibers had completely dissolved, but the woven fibers remained undissolved. Once again, evidence of break-down appeared in the analytical results.

The analytical results for the acid-caustic dissolutions are given in Attachment 4. For the clean HEPA dissolution (FDT-CP-6), the data in Attachment 4 indicates that a significant amount of the filter had dissolved in the acid step. These concentration numbers were roughly equivalent to the sum of the element concentrations for the first two steps of the normal clean HEPA filter dissolution (FDT-CP-7). However, the difference in dissolution effectiveness is apparent when the final solution concentrations are compared. The filter component concentrations (Zn, Al, Ba, B, K, and Si) for FDT-CP-6 were much higher than those of FDT-CP-7. In addition, the Na and NO₃ levels were higher in the final solutions. The effectiveness of dissolving the Na portion

of the filter could not be determined based on a strict comparison since NaOH is used in normal processing and this biased the Na concentration.

For the fouled filter experiments with acid-caustic dissolution, the elemental concentration results do not display much of a difference between the different times of acid-caustic dissolution. The physical results also support this, since no difference was seen between the amount of filter that was dissolved for the 72-72 and the 96-48 hour tests.

When compared to the normal dissolutions, the two filters portrayed different behaviors. The fouled HEPA experiments (FDT-FP-10 and -11) retained larger concentrations of the filter components in the final solution, yet the fouled HEME experiments (FDT-FM-12 and -13) dissolved more of the filter components than the normal processing method. The Cl, Na, and NO₃ levels were much higher for the acid-caustic dissolutions for both filter types than in the normal process.

XRD results for all acid-caustic dissolutions revealed the presence of sodium nitrate in the first solutions. In fact, only the clean HEPA (FDT-CP-6) and the 72-72 hour fouled HEPA (FDT-FP-10) initial solutions indicated the existence of any other compounds. The remaining solutions and final products exhibited the same components. The SEM results supported the XRD and chemical analyses results.

The original intent of this process was for waste and time minimization. While it was shown that waste would be minimized, due to omitting the extra dissolution step, time would not be because of the extra time needed to completely dissolve the filters. Even after the extended time period tests, the results were not good enough to justify recommendation of this process.

Effluent Penetration Depth

The degree of penetration of effluents seemed to have no effect on the dissolution process. None of the sections tested dissolved any more than the others. All physical characteristics of the final solutions were approximately the same. The only difference present during processing was the colors of the solutions, perhaps indicating the existence of different organics.

The analytical results are presented in Attachment 5 for all 3 sections of the HEME filter. In general, the outermost section of the filter (FDT-FM-3) showed the smallest concentrations of elements and the innermost section (FDT-FM-5) showed the highest concentrations of elements in the solutions. The variation amongst the concentrations was not significant, but it was enough to be noticeable. We are not sure as to which end of the filter the vertical section of filter element was removed from, since we did not know which way the filter was placed in the FAVC system. From the results, it would appear that what we are calling the inner section (FDT-FM-5) was located closest to the off-gas in-take. In either case, the amount of dissolution on the filter

elements was approximately the same, so it does not appear that the effluent penetration had an effect on dissolution.

From comparison of the XRD results, the same conclusion can be drawn. The compounds identified were practically the same with the exception of the sodium nitrate identified in FDT-FM-3, as opposed to the potassium calcium aluminum silicate hydrate identified in FDT-FM-4 and -5. The SEM results tended to confirm the findings of the XRD and chemical analyses. The major difference between the results was the appearance of sulfides in FDT-FM-4 and -5.

Large Scale Dissolutions

The six inch cube experiments performed in the MFT using an air sparge tube proved very unsuccessful. Only the pieces of filter located near the air sparge tube dissolved. The remaining filter was as intact as when it was first put in the solution and appeared floating on the solution surface. These results led to the necessity of an actual DWT Mock-up dissolution.

Analytical results for the large scale dissolution experiment (FDT-FM-14) are presented in Attachment 6. The elemental concentrations were not indicative of an effective dissolution. The concentrations for the second processing step should have been much greater, since this is the step where most of the filter components are dissolved into solution. In addition, the final elemental concentrations should have been much lower, since the filter and reaction products should be dissolved by this point. Theoretically, this experiment should have been more successful than the fouled HEME six inch cube experiment without air sparging (FDT-FM-9) because a larger tank and solution volume were used, along with air sparging; however, this was not the case.

The XRD results are consistent with other experiments; however, the SEM results point out that the filter materials did not appear until later in the process. They also indicate that a large portion of the filter material remained in the final product.

DWT Mock-up Dissolutions

The tests performed with the DWT simulated sparger were much more successful than the six inch cube study performed with the air sparge line. Approximately 76% of the filter dissolved and the remainder stayed in the "basket". A peculiar observation to note from this experiment was that the filter seemed to dissolve most during the last 4 hours of treatment; however, since the mass loss was not measured after each processing step, this notion cannot be substantiated. Due to the fact that the undissolved filter was still in the "basket", the resulting solution was essentially free of particles. Even though this experiment was not a total success, important insight into dissolution conditions was obtained.

Results from the analyses of the dissolution solutions for the mock-up experiment are presented in Attachment 7. The results indicate that the filter had begun to dissolve during the first step of dissolution, as substantiated by the amount of Al, B, Si, and K that had dissolved into the solution. The second step further dissolved the filter and released the Mg and Ca that had been incorporated in the first step, as attested to from the concentrations determined. However, the analyses of the final solution indicated that a substantial part of the filter components still remained in solution, and the analyses of the solid residue also indicated that a substantial part of the filter components had not been dissolved.

XRD results for this test revealed the expected compounds (sodium silicate, carbonate, and nitrate), as well as sodium hydroxide and barium nitrate. The fact that sodium hydroxide was able to be identified indicates that a large portion of the first dissolution solution remained unreacted; thus, maybe the reason why the last caustic step dissolved the filter further. SEM results for this test are not available.

Triton Tests

The small scale tests that were performed with Triton proved non-beneficial. While the HEME filter sample did dissolve more in the Triton laced 5% NaOH (Sample #2) than the pure 5% NaOH (Sample #1), chemical analyses results did not show a large difference in the amount of the elements released into solution or the elements remaining in the filter. The attempts with Triton laced 5% KOH (Sample #3) proved even less effective. Not only did less of the filter dissolve, but the releases were even smaller. The concentrations of the elements in solution (in ppm) and the composition of the remaining filter (in wt%) are presented in Table 5.

Table 5 - Triton Test Results

Element	Sample #1		Sample #2		Sample #3	
	Solution	Filter	Solution	Filter	Solution	Filter
Al	217.900	0.518	217.320	0.736	175.790	0.592
B	251.780	0.054	262.930	0.043	201.450	0.060
Ba	3.415	0.044	5.667	0.135	2.736	0.014
Ca	0.581	1.739	0.610	2.548	1.749	2.312
Cr	<0.364	0.007	0.430	0.007	<0.364	<0.004
Fe	0.713	0.063	0.641	0.125	<0.182	0.264
Hg	3.019	N/A	2.519	N/A	N/A	N/A
K	99.598	0.131	102.696	0.129	N/A	N/A
Mg	0.174	1.057	0.033	1.642	0.028	1.410
Na	51295	N/A	50119	N/A	1261.5	3.840
Na (AA)	61648	10.805	52071	16.523	N/A	N/A
Si	3943.7	10.827	4179.9	8.879	3090.2	20.438
Zn	6.345	0.148	20.739	0.149	7.027	0.135
Weight Loss		29.52%		32.71%		22.90%

The benefits of using Triton were not realized in these experiments, therefore it is not recommended that this chemical be used in helping to clean the filters. In addition, some very violent reactions were experienced when the temperatures of the Triton laced solutions rose above 80°C.

CONCLUSIONS

The experiments performed in this study, though not completely successful, have provided insight as to a dissolution process that may be effective on dissolving the DWPF's HEME and HEPA filters. The dissolution process developed by Bransford of 48 hours of 5% NaOH treatment, 96 hours of 5% NO₃ treatment, and 4 hours of 5% NaOH treatment exhibited limited success. While the process worked well on dissolving one inch cubes of filter, it was not as successful on larger sized samples. However, with the appropriate processing conditions, namely air sparging, the dissolution process proved more effective.

Even though the current tests were not as successful as the experiments performed by Bransford, the analytical results reflected some similarities. Zeolite was not detected in the XRD results as with Bransford's experiments, but their presence was indicated in the SEM results. The dissolution solutions, however, contained the elements necessary to substantiate Bransford's and Jantzen's ideas on what is occurring during the dissolution process. In the successful experiments, dissolution of the major components of the filter began in the first step, the second step further dissolved these components along with releasing the Ca and Mg that was believed to be incorporated in the first step, and the final step dissolved anything else that was remaining, so that the only thing remaining in solution was trace quantities of the filter components.

Based on the findings obtained thus far, it is our suggestion that a larger scale experiment be performed using the DWTT operating parameters and conditions. The results of this experiment should ensure that the process is effective in breaking down the fouled filters, before recommending the alternative disposal method to the DWPF.

Organic analyses of the filters and the dissolution solutions and products was not available at this time. Once the samples can be analyzed, the results will be issued in a separate document. The downstream effects of the final dissolution solutions were also not determined, since the transfer criteria for the DWPF to the Tank Farm have not been adequately established. Upon establishment of these criteria, the effects will be determined and a separate report will be issued.

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ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Na (AA)	Hg (AA)	K (AA)	Cl	F	NO2	SO3	NO3
DT-CP-7-1	0.359	0.025	36.302	49.420	16.003	<0.182	<0.455	68.067	<0.364	487.0698	26808	N/A	N/A	N/A	3	13	<2	1.64	3.64
DT-CP-7-2	20.522	9.066	52.915	37.482	58.880	<0.182	<0.455	42.008	<0.364	333.4656	24017	40300.5	0.013	44.178	1.2	11	<20	21.5	79735
DT-CP-7-3	0.736	<0.004	4.364	<0.910	<0.036	<0.182	<0.455	<0.364	<0.364	<0.546	27954	15085.75	<0.010	5.988	<1	<1	<25	<1	209
DT-FP-5-1	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	75300.5	<0.014	104.624	N/A	N/A	<25	15.4	519
DT-FP-5-2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	63300.4	0.058	93.911	N/A	16	N/A	N/A	N/A
DT-FP-5-3	1.391	0.028	70.213	36.187	17.680	2.855	<0.455	68.847	5.437	499.3901	>119056	128100.8	0.024	87.301	N/A	2	<25	<12.5	96928
DT-FM-7-1	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	67800.4	0.472	24.834	N/A	N/A	N/A	N/A	N/A
DT-FM-7-2	1.403	0.022	10.796	13.259	2.089	<0.182	<0.455	88.114	<0.364	1784.832	>86585	84600.5	0.965	34.744	N/A	2	<100	<50	157790
DT-FM-7-3	3.067	7.811	11.705	12.405	2.945	1.108	<0.455	65.643	<0.364	1411.482	>137591	105600.7	0.257	31.760	N/A	N/A	<100	<50	129516
DT-CP-3-1	1.254	0.370	30.762	79.018	13.406	<2.000	<5.000	113.822	<4.000	939.8682	>26548	2535.76	<0.01	6.770	375	28	Si	28.3	25.6
DT-CP-3-2	41.487	18.361	139.890	96.449	119.356	12.349	<1.471	94.628	3.064	525.0355	>23075	7537.00	<0.01	20.119	105	21	Si	75003	75003
DT-CP-3-3	0.161	<0.011	70.221	68.624	34.016	<0.526	<1.316	93.628	<1.053	745.8098	>87970	32855.9	<0.01	20.890	103	19	Si	<2.63	65153
DT-FP-3-1	0.337	<0.023	28.289	59.374	10.504	<1.067	<4.167	155.188	<3.333	1167.565	>24430	3008.85	<0.01	15.486	342	34	Si	43.7	283
DT-FP-3-2	85.741	23.650	168.930	123.864	164.813	108.425	9.388	151.027	27.283	474.4822	>24205	7721.73	0.330	42.532	116	28	Si	51.9	75647
DT-FP-3-3	0.055	0.010	41.472	44.285	9.048	1.658	<0.455	58.512	2.908	237.4926	>47777	48179.5	0.014	667.534	24	25	Si	<2.17	81588
DT-FM-3-1	0.775	<0.025	10.616	54.242	1.436	1.699	<3.125	254.207	<2.500	3368.98	>22075	3917.18	0.131	15.181	41	4	Si	34.1	334
DT-FM-3-2	300.578	341.323	28.856	393.115	47.081	12.739	<1.471	243.443	4.073	295.0451	>23110	7736.75	2.588	33.789	22	2	Si	43.5	80665
DT-FM-3-3	1.492	0.049	10.273	194.437	3.018	<0.500	<1.250	244.823	<1.000	4037.53	>99468	38134.6	0.457	38.216	28	2	Si	<2.50	78450
DT-FP-6-1	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	40983.9	<0.0105	159.077	36	24	<20	65.2	491
DT-FP-6-2	30.757	10.308	116.770	110.410	103.414	81.102	14.069	115.721	17.515	68.785	>27394	17355.1	<0.0105	118.462	31	22	<100	<50	79670
DT-FP-6-3	0.043	<0.004	90.957	90.482	18.616	3.648	<0.500	113.646	2.508	892.041	>71801	47059.0	<0.0105	123.877	17	25	<100	62	108577
DT-FP-6-S	1.047	0.308	1.959	1.068	2.965	2.129	0.564	1.064	0.341	8.088	>294.25	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
DT-FM-8-1	2.440	0.073	7.796	74.982	0.244	<0.182	0.958	106.062	<0.364	1590.064	27518	36545.8	0.655	55.189	66	1	<100	<12.5	304
DT-FM-8-2	104.120	108.917	18.115	101.688	13.503	5.556	<0.455	73.574	<0.364	1208.115	24714	21390.1	5.886	55.318	70	0.83	<25	19	91151
DT-FM-8-3	0.451	<0.004	2.741	23.427	<0.036	<0.182	<0.455	77.594	<0.364	1155.82	38649	68919.7	0.181	59.861	104	0.96	<25	<12.5	105599
DT-FM-8-S	0.922	0.828	0.150	0.234	0.039	0.201	0.009	<0.004	<0.004	2.193	12.939	66750.3	0.126	35.917	49	1	<12.6	<1	108.000
DT-FP-7-1	0.765	0.117	236.210	138.013	3.610	<0.182	<0.455	923.288	<0.364	5731.635	26433	N/A	0.014	N/A	190	87	<25	<105	572
DT-FP-7-2	166.310	48.991	665.000	503.861	987.471	102.050	7.391	774.898	31.040	104.7757	25469	28202.0	3.461	725.661	198	42	<25	<12.5	120184
DT-FP-7-3	140.338	39.072	468.960	363.971	851.513	28.614	4.041	784.547	17.047	136.3254	26581	37547.0	4.104	709.653	136	72	<25	<12.5	114893
DT-FP-7-S	0.156	0.059	0.899	0.528	1.019	0.300	0.028	<0.004	0.072	4.615	7.091	494.8	0.049	793.338	472	83	<8654	<865	94403
DT-FM-9-1	0.420	0.305	4.887	4.982	<0.036	<0.182	<0.455	963.869	<0.364	11819.01	33782	38900.9	3.709	422.796	91	4	77	108	1861
DT-FM-9-2	274.812	276.735	2.532	<0.910	37.129	<0.182	<0.455	601.393	<0.364	0.559	31376	50235.0	19.468	398.578	97	2	<25	104	120711
DT-FM-9-3	3.819	7.527	6.735	60.184	2.162	<0.182	<0.455	851.372	<0.364	18446.34	101760	95786.0	1.329	371.334	90	3	57.3	35.8	97284
DT-FM-9-S	4.503	5.045	0.000	5.685	0.656	0.393	0.111	3.577	<0.041	11.862	>224.51	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

XRD RESULTS

SAMPLE #	Compounds Identified
FDT-CP-7-1	Sodium Carbonate, Sodium Silicate
FDT-CP-7-2	Sodium Nitrate
FDT-CP-7-3	Sodium Carbonate, Sodium Silicate
FDT-FP-5-1	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-5-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-5-3	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-5-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-7-1	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-7-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-7-3	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-7-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-CP-3-1	Sodium Carbonate Hydrate
FDT-CP-3-2	Sodium Nitrate
FDT-CP-3-3	Sodium Nitrate, Sodium Carbonate Hydrate
FDT-FP-3-1	Sodium Nitrate
FDT-FP-3-2	Sodium Nitrate, Sodium Carbonate Hydrate
FDT-FP-3-3	Sodium Nitrate
FDT-FM-3-1	Sodium Nitrate
FDT-FM-3-2	Sodium Nitrate
FDT-FM-3-3	Sodium Nitrate, Sodium Carbonate Hydrate, Sodium Hydrogen Carbonate Hydrate
FDT-FP-6-1	Sodium Silicate, Sodium Carbonate
FDT-FP-6-2	Sodium Nitrate
FDT-FP-6-3	Sodium Carbonate, Sodium Nitrate
FDT-FP-6-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate, Potassium Sodium Calcium Aluminum Silicate Hydrate
FDT-FM-9-1	Sodium Carbonate, Sodium Silicate, Sodium Aluminum Carbonate Silicate
FDT-FM-9-2	Sodium Nitrate
FDT-FM-9-3	Sodium Silicate, Sodium Nitrate
FDT-FM-9-S	Sodium Nitrate
FDT-FP-7-1	Barium Hydroxide Hydrate, Sodium Silicate, Sodium Carbonate
FDT-FP-7-2	Sodium Nitrate, Barium Nitrate
FDT-FP-7-3	Sodium Nitrate, Barium Nitrate
FDT-FP-7-S	Sodium Nitrate, Sodium Carbonate
FDT-FM-9-1	Sodium Carbonate, Sodium Silicate
FDT-FM-9-2	Sodium Nitrate
FDT-FM-9-3	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-FM-9-S	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-FM-9-S	

SEM RESULTS

Compounds Identified
Filter material (Na, Si, K, Ca, Fe, Al), Na zeolite (Al, Si, Na), Sodium carbonate, Sodium silicate
Sodium compound (possibly nitrate), Filter material (Al, Si, Ca, Ba, Zn), Sodium carbonate
Sodium silicate, Sodium carbonate, Filter material (Na, Al, Si, Ca, Ce, Ni, Zn)
Sodium silicate, Sodium carbonate, Na zeolite (Na, Al, Si), trace K
Sodium carbonate, Filter material (K, Al, Si, Ni, Ba), trace (Na, K)Cl
Sodium compound (could be the nitrate), Filter material (Al, Si, K, Zn, Fe)
Sodium compound (could be the nitrate), Filter material (Ca, Si, Al, Fe, Ni, K, Mn, Ba), (Na, K)Cl
Na zeolite (Na, Si, Al), Sodium silicate, trace of filter material (Cr, Fe)
Sodium silicate, Sodium carbonate, Na zeolite (Na, Al, Si), Filter material (K, Na, Al, Si, Fe)
Sodium compound (possibly nitrate), Sodium carbonate, Filter material (Fe, Al, Si, Ca, Cr, Ni)
Sodium compound (possibly nitrate), Filter material (Al, Fe, Mn, Ca, Si, Ba, K), CeF ₃
Na zeolite (Na, Al, Si), Filter material (Ca, Mg, Zn), BaS, NaCl
Sodium carbonate, Sodium silicate, Filter material (Al, Si, Ba)
Sodium carbonate, Filter material (Al, Si, Ca, Ba, Mg, K, Zn, Ni)
Filter material (Cr, Al, Si, Ba, Ca, Fe, Zn), Sodium carbonate, Sodium sulfate
Sodium sulfate, Sodium carbonate, Filter material (K, Fe, Al, Si, Cr), Barium sulfide
Sodium compound (could be the nitrate), trace Si
Filter material (Na, Si, Fe, Mg, Ca), (Na, K)Cl, Sodium compound (possibly nitrate), Na zeolite (Na, Al, Si)
Sodium compound (perhaps silicate), Filter material (Al, Si, K, Ca, Fe, Na)
Sodium compound (possibly carbonate), Na zeolite (Na, Si, Al), trace filter material (K, Fe)
Sodium Silicate, Filter material (K, Al, Si, Ba, Fe), Sodium compound (possibly carbonate), CeF ₃
Filter matl (Fe, Zn, K, Al, Si, Ba, Cr, Ca), BaS, Na compound (possibly silicate, alumina, or a combo), (Na,)
Not available
Sodium aluminum carbonate silicate, Filter material (Ca, Si, Al), Sodium silicate, CeF ₃
Filter material (Ca, Si, Ba, Mg, S, K, Fe), Sodium carbonate, CeF ₃
Sodium silicate, Filter material (Ca, Al, Si, Na, Ce, Fe), Sodium carbonate
Filter material (Ca, Na, Si, Mn, Ni, Cr, Mg), Sodium silicate, Na Zeolite (Ca, Mg, Al, Si, Na), CeF ₃
Sodium silicate, Filter material (K, Zn, Si, Na, Al, Ca, Fe, Ce), Na zeolite (Na, Al, Si)
Filter material (Na, Si, Al, Ca, Fe, Ba, Zn, K), Sodium carbonate, Sodium silicate, CeF ₃
Filter matl (Na, Al, Si, Cl, Ca, Ba, K, Zn, Fe), Ba compound (possibly NO ₃), CeF ₃ , Na compound (possible NO ₃)
Sodium silicate, Filter material (Al, K, Si, Na, Fe, Ca, Ba, Mg, Mg), Sodium carbonate, (Na, K)Cl, CeF ₃
Sodium carbonate, Sodium silicate, Filter material (Al, Si, K, Na, Ce, Ca)
Sodium compound (possibly nitrate), Filter material (Al, Si, Ca, Mg, K, Ba, S), CeF ₃
Sodium aluminum silicate, Filter material (Ca, Mn, Fe, Cr, Ni, Na, Si, Al)
Na compound (possibly NO ₃), Filter matl (Al, Si, Ca, Na, Mg, Cr, Fe, Ni), BaS, (Na, K)Cl,
Sodium aluminum silicate

ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Na (AA)	Hg (AA)	K (AA)	Cl	F	NO2	SO3	NO3
FDT-CP-8-1	0.438	<0.004	30.063	36.952	16.591	<0.182	<0.455	58.601	<0.364	457.267	28774	23608	0.014	41.682	2.8	13	<5	<2.5	<7.5
FDT-CP-8-2	17.050	7.719	15.612	1.892	10.813	0.505	<0.455	2.340	<0.364	49.272	2477	9313	<0.010	3.154	<1	1	<20	14.7	29082
FDT-CP-8-3	0.503	<0.004	30.011	17.307	13.354	<0.182	<0.455	42.128	<0.364	371.968	60811	89525	0.010	43.430	2.4	11	<25	<12.5	70055
FDT-FP-9-1	0.305	<0.004	30.144	34.535	9.867	<0.182	<0.455	58.728	<0.364	414.128	28564	34004	0.011	77.624	8.3	14	<25	28.8	147
FDT-FP-9-2	14.686	5.073	16.944	4.159	42.150	66.530	4.132	1.314	9.739	40.510	1753	2599	0.132	5.151	<1	1	<25	13.5	33286
FDT-FP-9-3	0.470	<0.004	4.816	<0.910	<0.036	<0.182	<0.455	<0.364	<0.364	<0.546	27722	38553	<0.01	2.904	2.9	<1	<25	<2.5	171
FDT-FM-11-1	0.469	<0.004	5.225	85.161	0.277	<0.182	<0.455	106.467	<0.364	1441.89	29002	34004	0.118	47.174	3.2	<1	<25	<2.5	148
FDT-FM-11-2	92.429	107.367	9.823	39.144	12.279	5.767	<0.455	5.872	<0.364	158.252	2248	4115	0.015	6.762	<1	<1	<25	<12.5	33924
FDT-FM-11-3	0.871	<0.004	4.038	<0.910	<0.036	<0.182	<0.455	<0.364	<0.364	<0.546	26414	38202	0.013	1.815	<1	<1	<50	<2.5	379

XRD RESULTS

SAMPLE #	Compounds Identified
FDT-CP-8-1	Sodium Carbonate, Sodium Silicate
FDT-CP-8-2	Sodium Nitrate
FDT-CP-8-3	Sodium Carbonate, Sodium Silicate, Sodium Nitrate
FDT-FP-9-1	Sodium Carbonate, Sodium Silicate
FDT-FP-9-2	Sodium Nitrate
FDT-FP-9-3	Sodium Carbonate, Sodium Silicate
FDT-FM-11-1	Sodium Carbonate, Sodium Silicate
FDT-FM-11-2	Sodium Nitrate
FDT-FM-11-3	Sodium Carbonate, Sodium Silicate

SEM RESULTS

Compounds Identified
Sodium carbonate, Filter material (Na,Al,Si,K,Mg,Ca,S,Zn,Ni), Sodium silicate
Sodium compound (possibly nitrate), Filter mat'l (Na,Al,Si,Ca,Zn,K,Ba,Mn,Fe), BaS, Sodium carbonate
Sodium silicate, Sodium aluminum silicate, Filter material (Na,Al,Si,K,Ca,Fe)
Na compound (possibly nitrate), Sodium silicate, Filter mat'l (Na,Al,Si,K,Ce), Na zeolite (Na,Al,Si)
Sodium compound (possibly nitrate), Filter mat'l (Na,Al,Si,K,Ca,Ba,Cr,Mn,Fe,Ni,Zn), Sodium silicate
Sodium silicate, Filter material (Na,Al,Si,S,Cl)
Sodium silicate, Sodium compound (possibly Na2O or NaOH)
Na zeolite (Na,Al,Si), Filter mat'l (Na,Mg,Al,Si,Ca,Fe,Zn,Ce,Cr), Na compound (possibly nitrate), BaS
Sodium silicate, Filter material (Na,Al,Si,Cr,Fe)

ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Hg (AA)	K (AA)	Cl	F	#02	SO3	NO3
FDT-CP-5-2	1.584	<0.004	1.182	<0.910	28.480	<0.182	<0.455	249.537	<0.364	29.427	93044.91	99100.34	<0.01	N/A	N/A	SI	63.3	252683
FDT-CP-5-3	1978.7	<0.004	48.013	2.638	17.248	<0.182	<0.455	83.412	<0.364	551.997	63190.38	250.448	<0.01	1.168	16	<100	<1	66061
FDT-FP-8-2	51.735	18.167	211.110	136.368	300.542	153.009	15.502	173.902	31.951	<0.548	85983.68	57240.23	0.1285	N/A	29	SI	75.8	388946
FDT-FP-8-3	0.134	<0.004	70.627	29.286	16.504	<0.182	<0.455	89.324	<0.364	684.288	92474.07	56326.03	<0.01	130.380	18	<50	<1	174448
FDT-FP-8-S	2.490	0.620	2.027	1.057	4.113	7.839	0.643	0.044	1.265	5.539	N/A	N/A	N/A	N/A	20	<50	46.3	188630
FDT-FM-10-2	0.355	0.039	<0.055	<0.910	<0.036	<0.182	<0.455	125.624	<0.364	69.373	88094.28	N/A	<0.01	N/A	1	<50	<25	189038
FDT-FM-10-3	0.305	<0.004	7.584	<0.910	<0.036	<0.182	<0.455	131.950	<0.364	1665.13	130347.5	126810.7	N/A	72.809	7	<50	<25	144974
FDT-FM-10-S	7.548	9.929	1.331	11.926	0.769	2.012	0.265	<0.041	<0.041	34.186	N/A	N/A	N/A	3.96	4	<50	<25	153000

XRD RESULTS

Compound Identified

FDT-CP-5-1	Sodium Carbonate Hydrate
FDT-CP-5-2	Sodium Nitrate
FDT-CP-5-3	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-CP-5-S	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-FP-8-1	Sodium Carbonate Hydrate, Sodium Carbonate
FDT-FP-8-2	Sodium Nitrate
FDT-FP-8-3	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-FP-8-S	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-FM-10-1	Sodium Carbonate Hydrate, Sodium Carbonate
FDT-FM-10-2	Sodium Nitrate, Sodium Carbonate
FDT-FM-10-3	Sodium Nitrate, Sodium Carbonate, Sodium Silicate
FDT-FM-10-S	Sodium Nitrate, Sodium Carbonate, Sodium Silicate

SEM RESULTS

Compounds Identified

Sodium compound (possibly Na2O or NaOH), Filter material (Na,Al,Si,Ca,Zn,K,Ba), Sodium silicate
Sodium compound (possibly nitrate), Filter material (Ce,Fe,Na,Mg,Si,Fe,Cu,S,Ba,Ca,Ni)
Sodium silicate, Filter material (Na,Al,Si,Ca,F,Mg,Fe,Cr,Ce), Sodium carbonate
Sodium silicate, Filter material (Na,Al,Si,K,Zn,Ba), (K,Na)Cl
Sodium carbonate, Filter material (Na,Al,Si,K,Ca,Zn,Ba,Fe,Cl,S)
Sodium compound (possibly nitrate), Filter material (Na,Al,Si,K,Ca,Fe,Zn,Ce,Cr,Ni), BaS
Sodium silicate, Filter material (Na,Al,Si,Ca), CeF3, Zeolite (Na,Mg,Al,Si,Ca)
Sodium silicate, Filter material (Na,Al,Si,K,Mg,Zn,Ba,Cr,Ca,Ce)
Sodium silicate, Filter material (Na,Al,Si,K,Ca), Sodium carbonate, Na zeolite (Na,Al,Al)
Sodium compound (possibly nitrate), Filter material (Na,Al,Si,Ca,Ce), Na crystal, Sodium silicate, Ca
Sodium compound (possibly nitrate), Filter material (Na,Al,Si,Ca,Ce,Fe,S,Cl,K)
Sodium silicate, Filter material (Na,Al,Si,Ni,Cu,Cr,Fe,Zn), Sodium carbonate

ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Hg (AA)	K (AA)	Cl	F	MO2	SO3	MO3
FDT-CP-6-1	28.124	11.991	78.526	35.334	86.444	<0.182	<0.455	72.280	<0.364	148.1626	269.038	N/A	64.090	9	13	SI	31.9	44802
FDT-CP-6-2	0.157	<0.004	60.346	66.715	18.926	<0.182	<0.455	90.989	<0.364	1122.304	46209.9	66686.98	N/A	10	21	SI	9.88	35932
FDT-FP-10-1	13.024	3.873	52.890	39.280	79.052	66.466	4.890	58.056	13.897	148.4115	141.5844	N/A	N/A	23.4	9	<100	36.8	32668
FDT-FP-10-2	0.846	0.356	70.627	72.313	32.355	10.419	0.930	77.915	5.946	582.6458	54560.44	N/A	<0.01	16.2	13	<50	<25	31126
FDT-FM-12-1	34.822	34.244	16.667	40.768	6.541	8.607	1.155	37.556	0.652	157.696	271.1892	N/A	2.7677	N/A	<1	<50	<25	33540
FDT-FM-12-2	0.801	0.881	10.281	113.901	4.206	0.943	<0.455	68.287	<0.364	1128.195	53829.31	N/A	0.0485	N/A	<1	<50	<25	30797
FDT-FP-11-1	22.594	5.525	80.136	60.632	102.036	50.342	15.110	73.619	13.548	137.769	239.5518	N/A	6.8283	N/A	11	<50	34.9	38866
FDT-FP-11-2	0.902	0.388	75.562	75.984	51.063	9.832	0.907	78.420	6.269	605.8291	67905.94	N/A	0.199	N/A	16	<50	<25	37952
FDT-FM-13-1	27.186	25.385	14.049	31.242	4.894	9.238	0.761	29.285	0.520	145.3158	221.1201	N/A	6.333	N/A	<1	<50	<25	35938
FDT-FM-13-2	0.765	1.110	6.865	55.225	1.984	0.915	<0.455	51.519	<0.364	1175.552	61468.81	N/A	0.1465	N/A	<1	<50	<25	33907
FDT-FM-13-S	18.442	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0	194.8594	183.2653	0.3998	N/A	N/A	N/A	N/A	N/A

XRD RESULTS

SAMPLE #	Compound Identified
FDT-CP-6-1	Barium Nitrate, Sodium Nitrate
FDT-CP-6-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-CP-6-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-10-1	Zinc Hydroxide, Sodium Nitrate
FDT-FP-10-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-10-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-12-1	Sodium Nitrate
FDT-FM-12-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-12-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-11-1	Sodium Nitrate
FDT-FP-11-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FP-11-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-13-1	Sodium Nitrate
FDT-FM-13-2	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-13-S	Sodium Silicate, Sodium Carbonate, Sodium Nitrate

SEM RESULTS

Compound Identified

Sodium carbonate, Filter material (Na,Al,Si,K,Ca,Ba,Zn,Mg,Ce), Na zeolite (Na,Al,Si), BaS
Sodium Silicate, Filter material (Na,Al,Si,Ce,Cr,Mn,Fe,Ni,Ba)
Sodium Silicate, Filter material (Na,Al,Si), Na2O
Zinc compound (possibly hydroxide), Filter material (Zn,Al,Si,S,K,Ca,Ba,Cr,Fe,Zn), BaS
Sodium silicate, Filter material (Na,Al,Si,K,Zn,Ba,Ni,Fe)
Sodium silicate, Filter material (Na,Al,Si,Ni,Ca,Ce,Ba), Sodium aluminum silicate
Filter material (Na,Mg,Al,Si,S,K,Ca,Cr,Mn,Fe,Ni), possibly some zeolites (Na,Mg,Ca,Al,Si,Fe,K)
Sodium compound (possibly nitrate), Sodium silicate
Na zeolite (Na,Al,Si), Filter material (Na,Al,Si,Ca,K,Cl)
Sodium silicate, Na zeolite (Na,Al,Si), Filter material (Na,Al,Si,S,K,Ca,Ba,Fe,Cr,Zn,Ni,Mn), Sodium compound (possibly nitrate)
Sodium compound (possibly nitrate), Filter material (Na,Si,K,Zn,Cu), (Na,K)Cl,
Sodium compound (possibly nitrate), Filter material (Na,Si,K,Ca,Ba,Al,Mn,Fe,Ni), (Na,K)Cl,
Sodium compound (possibly nitrate), Filter material (Na,Mg,Al,Si,Mn,Fe,Ni,K,Ca,Cu)
Filter material (Na,Mg,Al,Si,S,K,Ca,Fe,Ce,Ci,Cr,Mn,Zn), BaS, Al2O3
Sodium silicate, Sodium compound (possibly nitrate)
Sodium silicate, Sodium carbonate, Filter material (Na,Mg,Al,Si,Mn,Fe,Ni,K,Ca,Cu)

ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Hg (AA)	K (AA)	Cl	F	MO2	SO3	MO3
FDT-FM-3-1	0.775	<0.025	10.816	54.242	1.436	1.899	<3.125	254.207	<2.500	3368.06	>22075	3917.18	0.131	15.909	41	4	SI	304
FDT-FM-3-2	300.578	341.323	28.956	393.115	47.081	12.739	<1.471	243.443	4.073	296.0451	>23110	7739.75	2.588	35.465	22	2	SI	80665
FDT-FM-3-3	1.492	0.049	10.273	194.437	3.018	<0.500	<1.250	244.823	<1.000	4037.53	>89469	38134.6	0.467	40.101	28	?	SI	78450
FDT-FM-4-1	3.199	<0.040	11.161	41.003	0.974	<2.000	<5.000	338.001	<4.000	4411.187	>25805	2150.98	0.054	13.317	26	5	SI	851
FDT-FM-4-2	374.513	425.949	26.443	485.801	57.728	14.967	<1.316	310.721	4.150	278.4051	>25973	10086.3	2.141	50.558	13	2	SI	84205
FDT-FM-4-3	1.780	0.181	21.481	141.712	2.050	<0.500	<1.250	289.988	<1.000	5625.549	>104825	32421.5	0.221	47.037	15	2	SI	50478
FDT-FM-5-1	1.722	<0.040	9.803	34.898	1.624	<2.000	<5.000	350.564	<4.000	4539.904	>24945	2708.07	0.085	13.838	50	5	SI	424
FDT-FM-5-2	369.376	415.574	26.971	473.401	55.813	12.579	1.734	320.668	3.687	316.2931	>24435	9125.68	3.106	46.97	16	2	SI	75808
FDT-FM-5-3	1.954	0.050	25.518	212.412	1.972	<0.526	<1.316	310.997	1.304	4919.091	>100441	38589.7	0.383	47.778	23	2	SI	90595

XRD RESULTS

SAMPLE #	Compound Identified
FDT-FM-3-1	Sodium Nitrate
FDT-FM-3-2	Sodium Nitrate
FDT-FM-3-3	Sodium Nitrate, Sodium Carbonate Hydrate, Sodium Hydrogen Carbonate Hydrate
FDT-FM-4-1	Potassium Calcium Aluminum Silicate Hydrate
FDT-FM-4-2	Sodium Nitrate
FDT-FM-4-3	Sodium Nitrate, Sodium Hydrogen Carbonate Hydrate, Sodium Carbonate Hydrate
FDT-FM-5-1	Potassium Calcium Aluminum Silicate Hydrate
FDT-FM-5-2	Sodium Nitrate
FDT-FM-5-3	Sodium Nitrate, Sodium Hydrogen Carbonate Hydrate, Sodium Carbonate Hydrate

SEM RESULTS

Compound Identified

Filter material (Na, Si, Fe, Mg, Ca), (Na, K)Cl, Sodium compound (possibly nitrate), Na zeolite (Na, Al, Si)
Sodium compound (possibly silicate), Filter material (Al, Si, K, Ca, Fe, Na)
Sodium compound (possibly carbonate), Na zeolite (Na, Si, Al), trace filter material (K, Fe)
Sodium carbonate, Filter material (Na, Si, Al, Mg, Ca, S, Fe, Ni, Zn, Co, Cr, Hg), Na zeolite (Na, Al, Si)
Zeolite (Na, Al, Si, Mg, Ca), Filter mat'l (Na, Al, Si, Ca, K, Ni, Fe, Cr, S), BaS, Na compound (possibly nitrate)
Sodium carbonate, Filter material (Na, Al, Si, Ca, Mg)
Zeolite (Na, Mg, Al, Si, Ca), Filter material (Al, Na, Si, K, Ca, S, Mg, Hg, Fe, Cl, Cr, Ce)
Sodium carbonate, Filter material (Na, Al, Si, K, Ca, Mg, Co, Fe), BaS, CeS
Sodium carbonate, Filter mat'l (Na, Fe, Al, Si, Cl, Ca, Cr, Fe, Zn, Ni, Mn), BaS, Na compound (possibly nitrat

ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Na (AA)	Hg (AA)	K (AA)	Cl	F	NO2	SO3	NO3
FDT-FM-14-1	1.275	0.082	0.869	182.187	<0.026	<0.102	<0.455	185.132	<0.364	2621.133	>46897	43156.9	1.580	94.259	N/A	N/A	<50	34.3	505
FDT-FM-14-2	10.004	4.481	0.570	<0.910	0.674	<0.182	<0.455	142.107	1.114	142.8686	>41772	37413.6	1.774	84.175	N/A	N/A	<50	36.3	100029
FDT-FM-14-3	1.003	0.112	6.247	254.663	<0.026	<0.182	<0.455	207.056	1.569	3473.203	>78019	71636.55	3.580	103.027	N/A	N/A	<50	36.2	107495
FDT-FM-14-S	4.070	5.195	0.000	3.046	0.044	<0.020	<0.051	0.346	<0.040	60.68176	110.320	N/A	0.151	N/A	N/A	N/A	<1956	<978	55849

XRD RESULTS

SAMPLE #	Compound Identified
FDT-FM-14-1	Sodium Carbonate, Sodium Silicate
FDT-FM-14-2	Sodium Nitrate
FDT-FM-14-3	Sodium Carbonate, Sodium Silicate, Sodium Nitrate
FDT-FM-14-S	Sodium Nitrate, Sodium Carbonate Hydrate

SEM RESULTS

Compound Identified

Sodium silicate, Filler material (Na, Si, Al, K, Cl)
 Na carbonate, (Na, K)Cl, Filler mat'l (Na, Al, Si, Zr, Ca, Fe, Cr, Ni), BaS, Na compound (possibly nitrate)
 Sodium compound (possibly nitrate), Sodium silicate, Filler mat'l (Na, Al, Si, Ca, K, S, Ba, Fe, Mn, Ni, Zn, Cl)
 Na compound (possibly nitrate), Filler mat'l (Na, Al, Si, Ca, Mn, Fe, Ni, Cu, Mg, K, Cr, Hg), SiO2 crystals

ELEMENTAL RESULTS (ppm)

SAMPLE #	Ca	Mg	Zn	Al	Ba	Fe	Ni	B	Cr	Si	Na	Na (AA)	Hg (AA)	K (AA)	Cl	F	MO2	303	NO3
FDT-FM-15-1	0.144	0.137	2.921	134.679	4.885	0.216	0.104	152.716	0.065	2651.136	30655	37067.8	0.505	82.762	N/A	N/A	<100	<16	802
FDT-FM-15-2	98.787	121.075	3.396	128.708	15.573	9.114	1.841	86.955	0.902	809.8202	24084	24137.2	0.576	56.048	N/A	N/A	<100	<2.5	64554
FDT-FM-15-3	41.772	55.788	2.557	109.514	9.447	5.762	1.004	87.316	0.647	1942.528	86825	86817.3	0.386	57.404	N/A	N/A	SI	<10	66894
FDT-FM-15-S	5.250	7.200	0.041	2.726	0.158	<0.20	0.172	0.282	<0.041	48.760	334.7	31680.07	0.121	2.817	N/A	N/A	<2724	<987	215180

XRD RESULTS

SAMPLE #	Compound Identified
FDT-FM-15-1	Sodium Hydroxide, Barium Nitrate, Sodium Silicate, Sodium Carbonate
FDT-FM-15-2	Sodium Nitrate
FDT-FM-15-3	Sodium Silicate, Sodium Carbonate, Sodium Nitrate
FDT-FM-15-S	Sodium Carbonate, Sodium Nitrate

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6 / 10 / 93