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Liquid Hydrofluoric Acid Sorption Using Solid Media—Part I

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UT-BATTELLE

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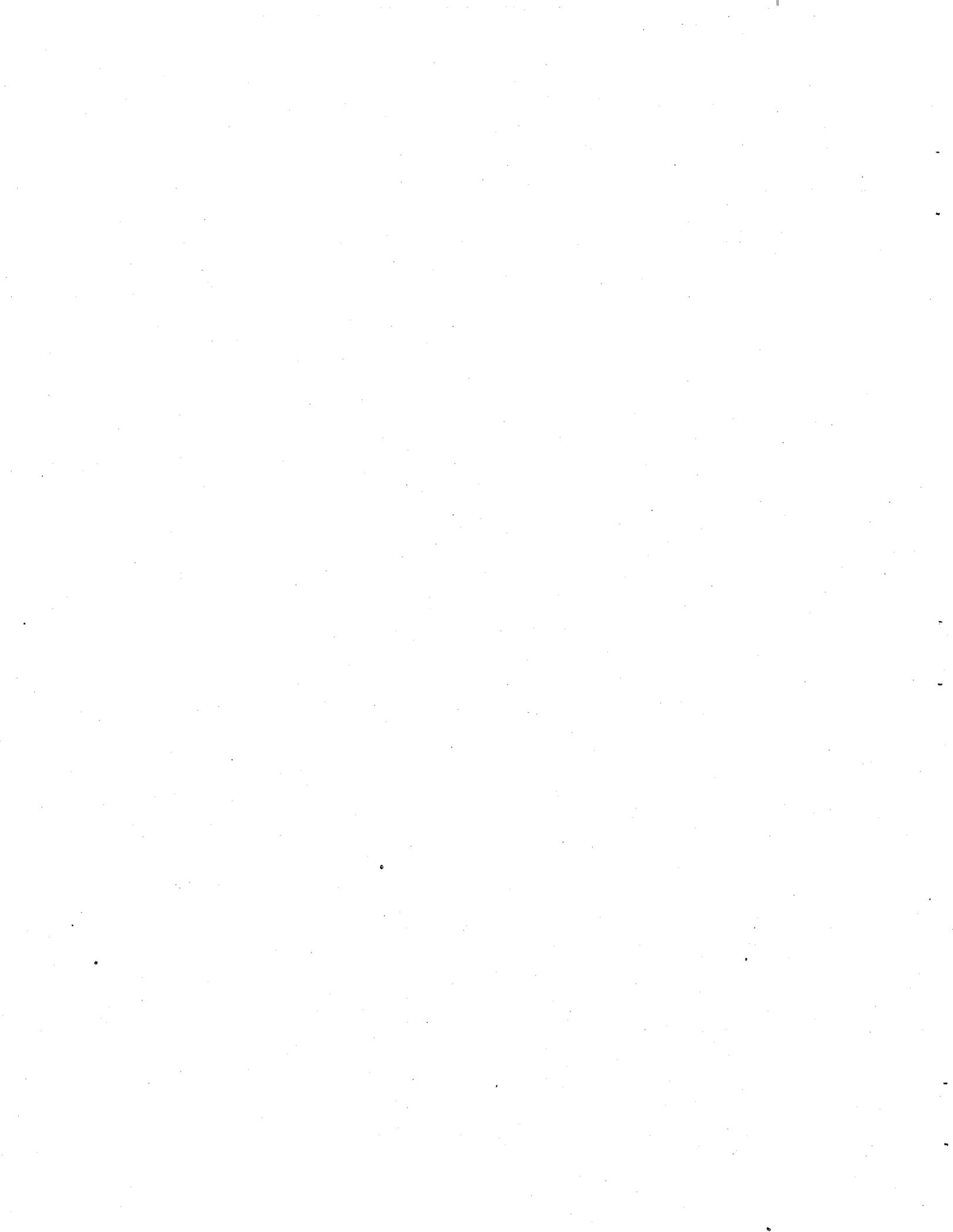
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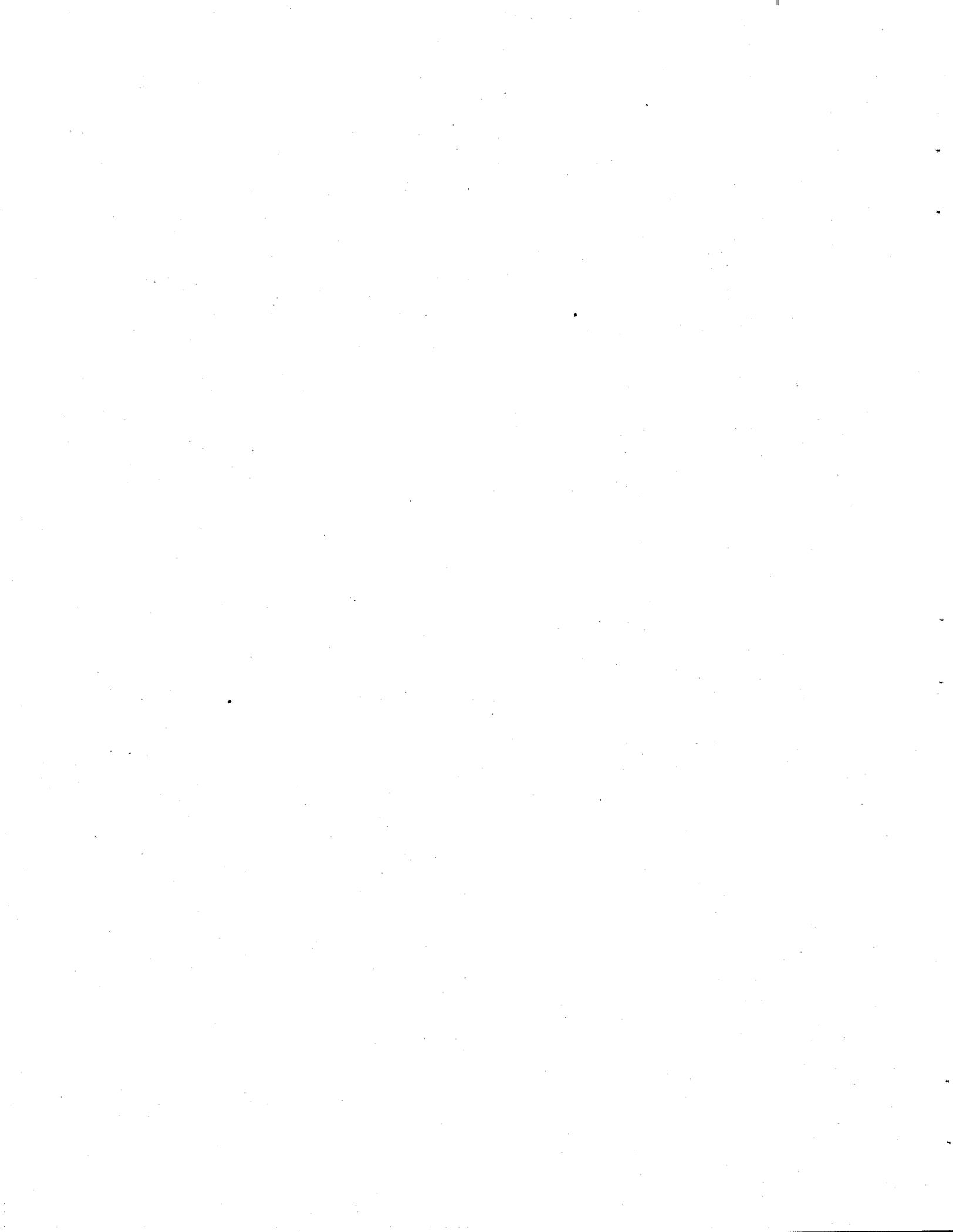
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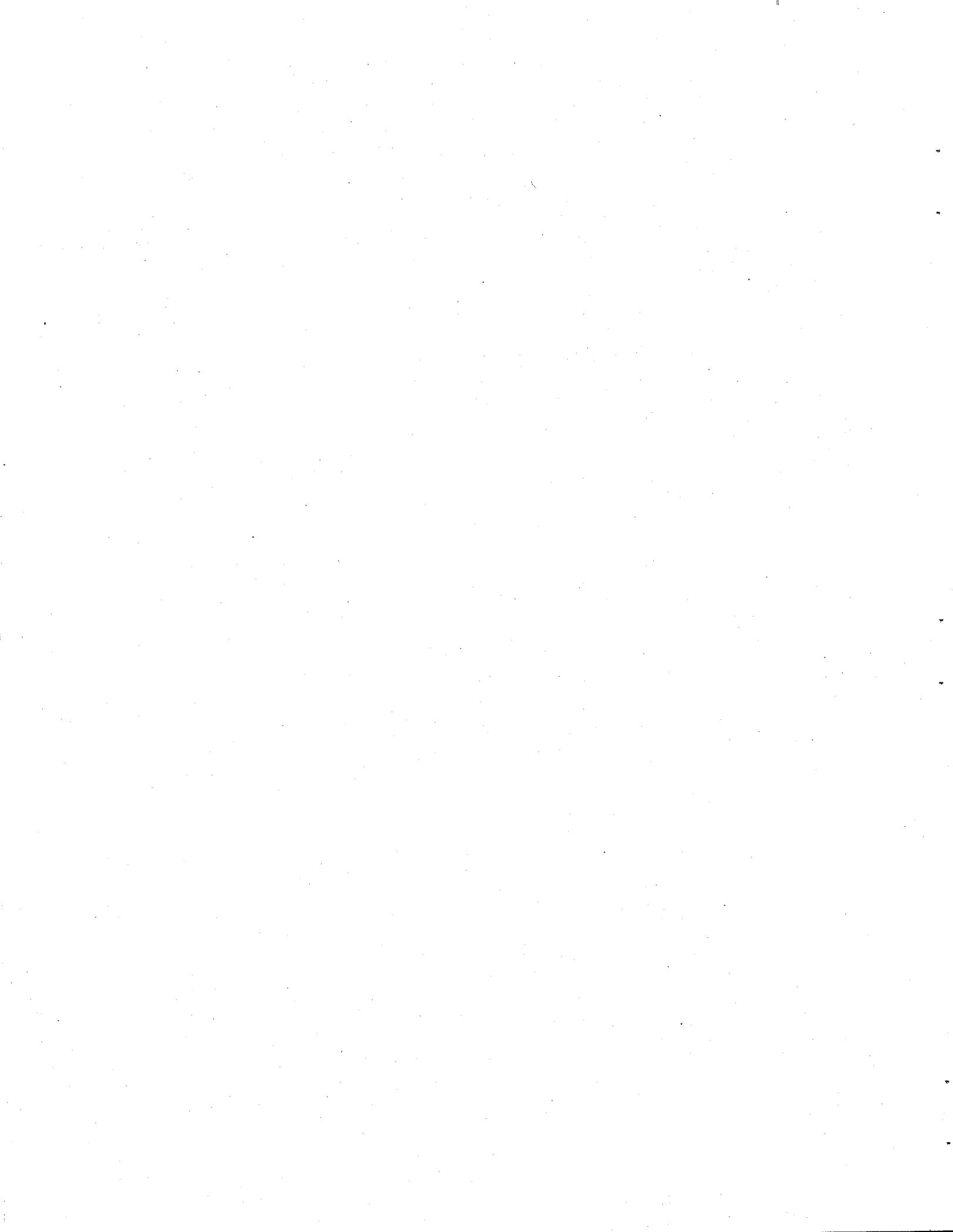
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ACRONYMS

BFS	blast furnace slag
BJC	Bechtel Jacobs Company, LLC
DOE	U.S. Department of Energy
MSRE	Molten Salt Reactor Experiment
ORNL	Oak Ridge National Laboratory
PC	portland cement
PVC	polyvinyl chloride



EXECUTIVE SUMMARY

The conversion of the uranium hexafluoride (UF_6) which is removed from the Molten Salt Reactor Experiment (MSRE), into a stable oxide for long-term storage will produce a significant amount of slightly contaminated, concentrated aqueous hydrofluoric acid (HF). Since the handling of this HF is complicated and dangerous, it was decided to transform it into a stable solid fluoride (e.g., CaF_2 , AlF_3 , and MgF_2). Tests have been performed to identify the best media to use for trapping the HF. These tests are described in this report.

The first series of tests evaluated 37 trapping materials using a 6 wt % solution of HF. The solution was pumped through a 3.8-cm-diam column at a slow rate, and samples were taken in 100-mL batches until it was determined that the media could no longer neutralize the solution. Each bed volume of media was evaluated for its retention of fluoride and for its plugging problems.

Mixtures of calcium hydroxide and blast furnace slag (BFS) with high surface areas (18–30 mesh) performed the best. A mixture of 80 wt % calcium hydroxide and 20 wt % BFS was capable of loading 0.134 g HF per cubic centimeter (cm^3) of media. Other media that performed well were (a) mixtures of calcium hydroxide and portland cement and (b) pure calcium hydroxide.

The second series of tests evaluated media using a 33 wt % HF solution. The best performing media from the first series and some new ones were tested. A 2.54-cm-diam, clear, polyvinyl chloride pipe was used as the column, and solution was introduced to different types or sizes of media using slugs from a pipette or constant flow of ~ 10.7 mL/min from a metering pump. The transparent PVC allowed for observation of acid-media interaction and provided a glimpse into how the media and cartridge were performing in this highly corrosive environment.

Results from the second series of tests showed that many of the best performing media from the first series of tests would not do well under the more concentrated solutions of HF. Plugging and vigorous reactions were common in the second series, and calcium hydroxide-based media was ruled out due to its disintegration at any size (1.25-cm diam to 30 mesh). The best performing media was mid-sized (4–18 mesh) soda and lime (soda lime). This media not only stood up well in the HF solution, but it also had great neutralization capability, effectively neutralizing up to ~ 0.5 g HF/ cm^3 of media. It is expected that a cartridge of this sorbent will be capable of handling approximately seven batches of HF from the uranium conversion.

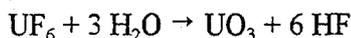
This work was managed by Bechtel Jacobs Company, LLC, (BJC) as prime contractor for the U.S. Department of Energy (DOE) for DOE EM under contract DE-AC05-98OR22700, responsible for MSRE remediation activities. UT-Battelle is the DOE prime contractor for operation of the Oak Ridge

National Laboratory (ORNL) under contract DE-AC05-00OR22725, and tasked by BJC as subcontractor for the MSRE conversion project activities, under a work authorization agreement to provide all the necessary support for operation of these activities. BJC is responsible for expenses associated with the conversion project. The ORNL Chemical Technology Division has responsibility for Building 4501 and for conducting operations and maintenance associated with the conversion project. Conversion project operations in Building 4501 are conducted in accordance to the ORNL Work Smart Standards.

1. INTRODUCTION

This report presents results obtained from the testing of several types of solid media used to remove hydrofluoric acid (HF) from a liquid waste stream. The work was performed at the Oak Ridge National Laboratory (ORNL) in support of the Molten Salt Reactor Experiment (MSRE) remediation. When uranium hexafluoride ($^{233}\text{UF}_6$) is converted to uranium oxide ($^{233}\text{U}_3\text{O}_8$), a large quantity of highly reactive HF is produced. This unwanted HF can be disposed of safely and conveniently as stable fluorides (e.g., CaF_2 and AlF_3). The unique circumstances associated with the MSRE remediation provided many constraints not usually associated with the disposal of HF.

The conversion process can be summarized by the following global reaction:



(UO_3 is not stable at higher temperatures, and it transforms spontaneously in air to U_3O_8 .)

A total of 6 mol of HF are produced for each mole of UF_6 that is converted. The HF is generated in the form of a concentrated aqueous solution and may include traces of uranium and other impurities (e.g. Ni, Cr, Fe, Mo, and W). The concentrated aqueous HF is a dangerous, extremely corrosive, and highly reactive waste stream. Because a remote possibility of criticality exists, all vessels and tubing must maintain a safe geometry by having a diameter of 4 in. or less.

The current plan is to avoid the handling of HF by reacting it with a substance to produce an inert solid. To avoid transporting trace amounts of uranium into the solid reagent, the process includes a distillation step for the HF. As shown in Fig. 1.1, initially the HF aqueous solution is condensed inside a cooled vessel (condenser 1). At the end of the conversion process, the condensed HF is warmed and transferred to an evaporator, in which the HF is distilled, under vacuum, into a pure HF solution. Any dissolved solids will mostly remain in the evaporator, and the purified HF solution will condense inside the second condenser. The evaporator will be periodically treated with F_2 or ClF_3 to fluorinate and recover the accumulated traces of uranium as UF_6 . The clean HF in the second condenser will then be slowly flowed into the HF trap, which is basically a removable plastic cartridge, containing a trapping material. The HF reacts with the media, producing a contained product, which remains in the cartridge, while the water is collected at the bottom of the trap and evaporated. Initially, the pH of the effluent water is alkaline; the pH remains nearly constant for quite some time until the trapping material reaches saturation. The cartridge will be replaced once the pH starts to drop and when there is still some capacity left. An indicator, which changes color when the pH changes, is used to visually determine when the cartridge is exhausted and needs to be replaced.

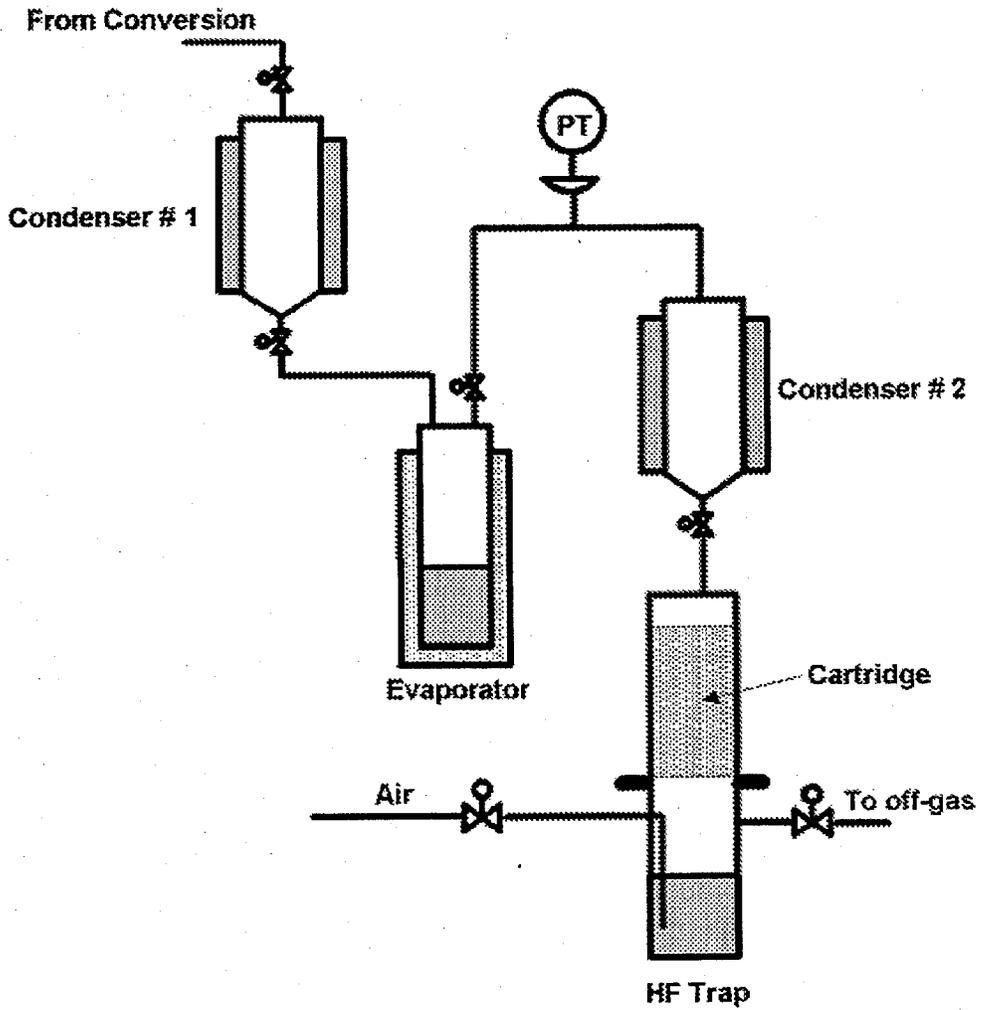


Fig. 1.1. Diagram of the HF handling system.

The HF trapping media tests were conducted in two parts. Part one was a test of several different materials using a solution of 6 wt % HF to (a) observe the reaction of HF with the media and (b) determine if further testing was appropriate. Thirty-seven materials, or variations thereof, were tested using a peristaltic pump to deliver the HF solution through a 3.8-cm-ID polyvinyl chloride (PVC) column. Media were evaluated for fluoride absorption, pH neutralizing capacity, and plugging tendency.

The second phase of the tests involved re-evaluating some of the best performing media (identified in Part one) with a 33 wt % HF solution. Because of the different behavior of the concentrated HF solution, a few other materials not previously tested were tried—totaling 39 types of media, or variations thereof, in this series of tests. The second-phase tests also involved pumping and/or sending slugs of HF solution through a 2.54-cm-ID transparent PVC column to observe the acid-media reaction and to evaluate its use in the prototype. Specific criteria for consideration were (a) the pH neutralization capability and (b) the retention of original physical characteristics (ability to withstand dissolution). This setup was most closely related to the actual conversion process. Several methods to introduce the HF to the column were tested also. These methods included metered flow, diffuser tube, and differently sized aliquots.

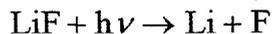
This report provides background information on the MSRE and the conversion process, a description of the experiments, results, and conclusions regarding the recommended media to use for the HF trapping.

2. BACKGROUND

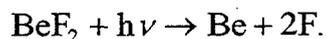
2.1 MSRE

The MSRE was operated at ORNL from 1965 to 1969 to test the concept of a high-temperature, homogeneous, fluid-fueled reactor. The reactor was fueled with a molten salt mixture of LiF-BeF₂-ZrF₄-UF₄ (with a composition of 64.5-30.4-4.9-0.14 mol % for a ²³³U-fueled reactor and 64.1-30.0-5.0-0.81 mol % for a ²³⁵U-fueled reactor), which melts at about 450°C and which served as both the fuel and the primary coolant (Compere et al. 1975). This fluid was circulated by a large impeller pump, which was located between the reactor core and the primary heat exchanger. A secondary coolant of LiF-BeF₂ (66 and 34 mol %, respectively), circulated by a similar impeller pump, transferred heat from the primary heat exchanger to an air-cooled radiator. About 4,350 kg (~2 m³) of fuel salt constituted the fuel charge circulating in the fuel-salt circuit. Originally, the MSRE was fueled with ²³⁵UF₄; however, after successful operation with this isotope, the ²³⁵U was removed by fluorinating the tetrafluoride to volatile UF₆. Afterward, the fuel was reconstituted with ²³³UF₄ (containing 220 ppm ²³²U, an impurity isotope) to demonstrate that the system could function equally well on the product of a ²³²Th thermal breeding cycle. After the successful completion of this campaign, the reactor operation was terminated Dec. 12, 1969, when the fuel salt was drained from the reactor circuit and solidified in two drain tanks at a lower level of the facility. The fuel salt has remained in these tanks for more than 30 years.

During the MSRE operation, no radiolysis of the fuel salt was ever observed. However, radiolysis of the fuel salt was recognized as a problem if the salt were solidified and held below 100°C—with the net effect that F₂ would be liberated from the frozen salt mixture and could then cause corrosion or overpressurization of the drain tank containment system. The relevant radiolysis reactions are (Williams, Del Cul, and Toth, January 1996; Toth and Felker 1990):



and

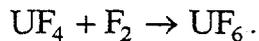


To prevent the accumulation of F₂, the frozen salt (which was normally at about 40°C because of the self-heating by fission product decay) was heated to about 200°C annually. This frequency was selected because of the experimentally observed induction period before release of F₂ from the salt matrix

(Savage et al. January 1964). Hence, it was believed that any generated F_2 would be trapped in the matrix and that heating would lead to the recombination of the F_2 with the reduced metal sites left in the salt. The fluorine pressure in the drain tanks before and after annealing was not monitored; therefore, the effectiveness of this annual procedure was never established.

In the late 1980s, an increase in radioactivity in one of the gas-line protrusions into the North Electrical Services Area, a room adjacent to the drain tank cell, was suspected as coming from UF_6 . Because the annual annealing operation would drive this condensable gas from the drain tanks to cooler surfaces, such as the gas-line protrusion into the North Electrical Services Area, the annual annealing operation was postponed until a better understanding of the fuel salt under long-term storage conditions was obtained.

In early 1994, two 1,000-mL gas samples were withdrawn (from a gas line in the Vent House connected to the drain tanks) and analyzed. Surprisingly, 350 Torr of F_2 , 70 Torr of UF_6 , and smaller amounts of other gases were found in both of the samples (Table 2.1), confirming that the annual annealing operations had not been successful in recombining the fluorine with the fuel salt and, more importantly, that the temperature gradient created during the annealing operation had definitely—as was later shown (Williams, Del Cul, and Toth, January 1996)—contributed to the formation and displacement of UF_6 from the fuel salt. The UF_6 was formed by the following reaction (Williams 1999):



Upon further investigation, it was found that the gas-line from the drain tank also ran to large charcoal beds (U-tubes of 6-in. diam and 24-ft length), which could not be isolated because a shutoff valve had failed in the open position. Gamma scans and thermal analyses indicated that about 2.6 kg of the uranium from the drain tanks had been deposited at the charcoal-bed inlet. Because F_2 was also present with the UF_6 , it was believed that the charcoal bed containing both carbon-fluorine reaction products (C_xF) and uranium presented both chemical and radiological hazards. The C_xF was an explosive compound, which could result in major dispersion of the ^{233}U contained in the charcoal bed. On Nov. 20, 1995, the shutoff valve was closed to prevent the further movement of uranium and fluorine onto the charcoal bed. Steps were taken to minimize (and ultimately eliminate) the possibility of explosive decomposition of the C_xF in the charcoal beds.

Table 2.1. Gas analysis of two samples taken from the off-gas MSRE system^a

Species	Gas composition (Torr)	
	First sample	Second sample
UF ₆	70	68
HF	1,200 ppm	1,000 ppm
MoF ₆	10	10
CF ₄	5	5
F ₂	<i>b</i>	350
He, Ar, N ₂ , O ₂ ^c	305	305

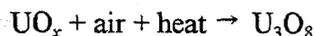
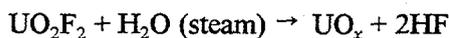
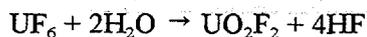
^a Adopted from ORNL/TM-2000/92, *Prototype Tests for the Recovery and Conversion of UF₆ Chemisorbed in NaF Traps for the Molten Salt Reactor Remediation Project*, April 2000 (Del Cul et al.).

^b Not determined analytically but assumed to be the same as second sample.

^c Quantity determined by difference from total sample pressure. Qualitative identification by mass spectroscopy.

The remediation of the MSRE site includes the removal of the total uranium inventory of about 37 kg. Of that inventory, about 23 kg have been removed from the piping system and sorbed on NaF pellets (as a 2NaF•UF₆ complex). The containers for the NaF pellets are referred to as NaF traps, and 25 such traps have been produced. The traps are being stored in Bldg. 3019 at ORNL. The planned recovery of ~11 kg of uranium from the fuel salt will generate another 15 to 19 NaF traps. The remaining 2 to 3 kg of uranium are laden into activated charcoal beds, which are also scheduled to be removed from the reactor site. Since all these materials, NaF traps and the uranium-laden charcoal, are not suitable for long-term storage, they will be converted to a chemical form (U₃O₈), which is suitable for long-term storage.

The conversion project has been challenged with the conversion of pressurized traps containing the highly radioactive UF₆ into a stable oxide (U₃O₈) for long-term storage. The amount of UF₆ absorbed by each cylinder was carefully documented and is in the range of 1.1–1.3 kg (~4 mol) of UF₆ per trap. The first step of the process will convert the UF₆ to UO₂F₂ and HF, after an addition of water (H₂O). The product is then heated, and more water is added (in the form of steam), converting it to UO_x. The by-product of this reaction is an additional 2 mol of HF. The final step will involve converting the UO_x to U₃O₈ by heating in air. The entire conversion is demonstrated in the following set of reactions.



The first two reactions evolve 6 mol (120 g) of HF gas or ~24 mol (480 g) per batch based on the 4 mol of UF_6 loaded on each NaF trap. This will be diluted by ~1,500 mL of water, which is added to the system, producing an average concentration of 33 wt % HF. The HF, which is produced by this process, will leave the system as a gas and steam mixture, which will be condensed in a condenser. The entire conversion process is summarized by Fig. 2.1.

2.2 HF TREATMENT TECHNOLOGY

HF is a hazardous and highly reactive compound, which has many unique characteristics (classified as a “weak” acid in most chemistry books because of its lack of complete dissociation when diluted), and it is considered one of the most dangerous of the acids. It is highly reactive and will readily attack a wide range of materials and compounds including (1) glass, concrete, and silica-based compounds; (2) most metals; (3) alkalis; (4) elastomers; and (5) many organic materials such as leather. Some of the reactions are very violent and can be highly exothermic—potentially forming explosive hydrogen gas (Farrar and Barber 1979).

In addition to its corrosivity, HF has extreme health consequences for ingestion and contact. Once on the skin, the HF disassociates itself from other diluting molecules and is absorbed deep into the tissue, where it searches for calcium, magnesium, sodium, and potassium. The fluorides it then forms can lead to cell death, bone demineralization, organ failure, and arrhythmias. HF is extremely toxic and can lead to death from exposure of only 2% of the body or 50 ppm in air (“Improving HF First Aid”, C&EN September 1999).

Disposal of aqueous HF in industrial settings is performed in several different ways. One of the most prevalent methods of disposal is to use a wet-scrubber system. This process is sometimes accomplished by neutralizing the HF with potassium hydroxide before sending it to a concurrent scrubbing chamber with a lime (calcium hydroxide) slurry. The lime slurry serves to floc and precipitate the calcium fluoride where it can then be filtered (Farrar and Barber 1979). Similar methods involve scrubbing with water and initiating precipitation by adding lime, or transporting the HF directly to a lime-slurry system, known as a gas-liquid dispersion scrubber (Kohl and Riesenfeld 1960, Story 1975).

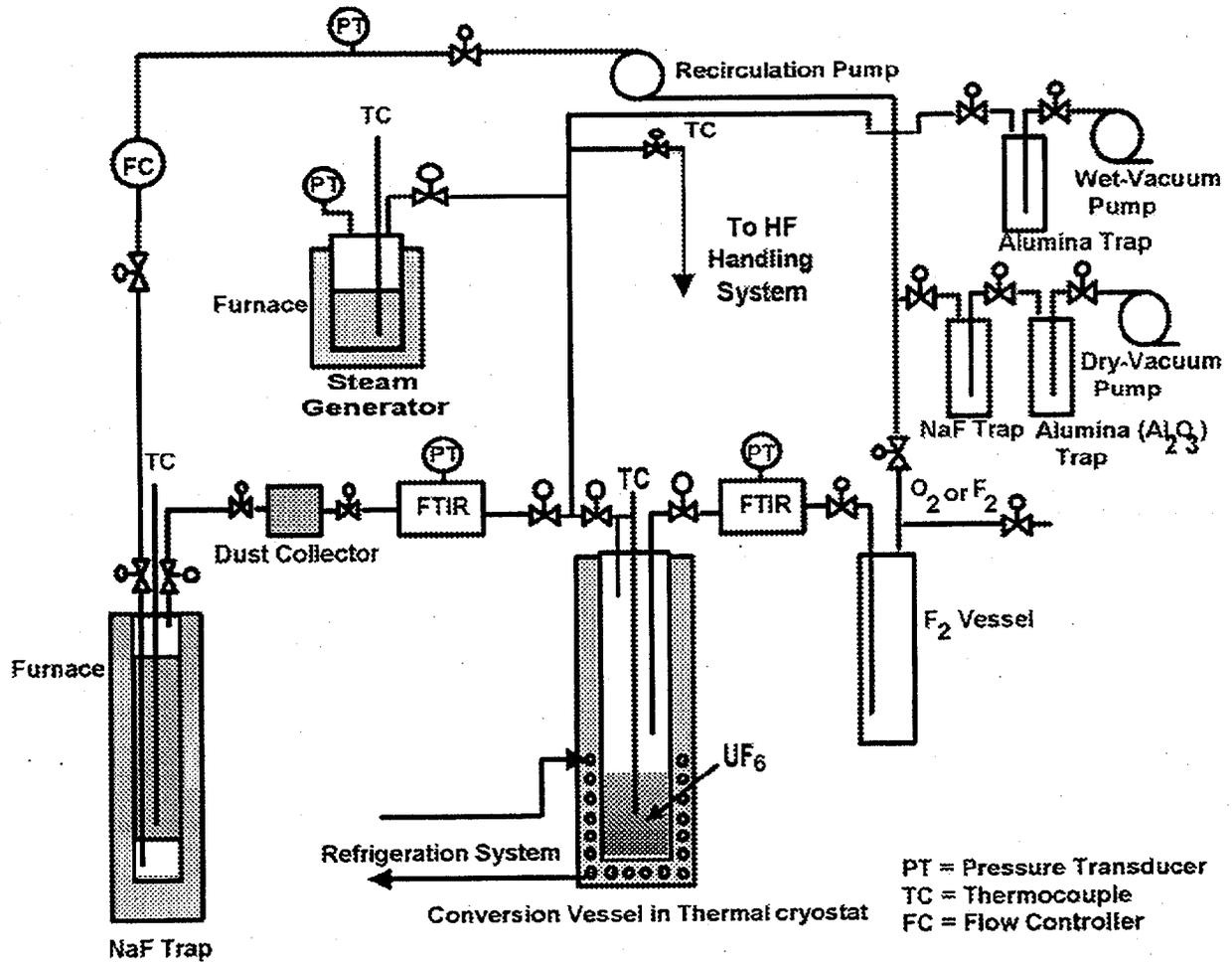


Fig. 2.1. Diagram of uranium recovery and conversion process for NaF traps.

Although each of these methods is currently feasible and used in different parts of industry, each has limitations. These methods are used primarily where large amounts of low concentration (<10 wt %) fluorine and HF are being disposed. Since these systems are designed to handle large quantities of chemicals, more space and equipment are required.

Other studies have documented the treatment of HF in gas streams. Most of these involve static beds of limestone (CaCO_3) as the compound of choice (Liimatainen and Mecham, February 1955, Gilbert, et al. March 1953). Although both of these studies proved good removal efficiency (~90 % or better) they, again, were designed to handle large flows of dilute (<10 wt %) HF in gas streams and therefore were not conducted under conditions representative of the current study. Advantages and disadvantages of disposal of small amounts of fluorine and HF by soda lime traps or reaction with carbon have also been discussed (Detamore, April 16, 1983, Farrar and Barber 1979). One study of interest involved treating the off-gas from a uranium dioxide conversion reactor, which contained ~50 vol % HF (Brown and Fowler, January 1985). This study looked at potential solid compounds as possible alternatives to wet scrubbing. The media of choice was slaked calcium oxide (calcium hydroxide) pieces, which were used in copper baskets. Results from this study indicated a tendency for the reactor to plug with calcium fluoride above a length to diameter ratio of eight. However, some excellent loading efficiencies of 0.13 to 0.26 g HF/cm³ $\text{Ca}(\text{OH})_2$ [0.3 to 0.6 g HF/g $\text{Ca}(\text{OH})_2$] were demonstrated. It was decided that the HF in the current study should be treated in a column configuration because of the remote possibility of uranium carryover in the HF, causing a criticality hazard.

3. EXPERIMENTAL

Approximately 953 mol of HF will be produced as a by-product of the conversion of 37 kg of fissile UF_6 to U_3O_8 . The concentrated HF has proven to be a hazardous and highly reactive compound, and it is desirable that the HF be neutralized as soon as possible after formation.

The testing was performed to determine which material would best sorb HF and leave as few free fluoride ions as possible that might reform as another corrosive substance. Two concentrations of HF were used in testing the media. A 6 wt % solution was initially used to help identify potential sorbents, and a 33 wt % solution was used in the second phase of the testing. The primary objective of the testing was to identify a solid sorbent that would work in a column to handle as much HF as possible, thereby minimizing the number of cartridges used.

3.1 EVALUATION OF MEDIA USING 6 wt % HF

The testing of the 6 wt % HF is covered in Sect. 3.1.1, with the subsequent section (3.1.2) devoted to the results.

3.1.1 Setup

Part one of the media testing of the media involved initially identifying the best available materials to neutralize a 6 wt % solution of HF. Thirty-seven different types or variations of media were evaluated on their respective (1) ability to produce an insoluble reaction product with fluoride, (2) ability to neutralize the HF, and (3) durability or ability to allow liquid to pass—yet not plug.

These tests were performed only for selection of the materials that have the best affinity for fluoride. Before the first set of column tests, numerous materials were tested in beakers in a batch-test mode to select the best potential performers. One set was tested at ambient temperatures while the second set was tested at elevated temperatures (80°C). Very little difference was seen in the reactivity of HF at the two temperatures.

The materials were tested in the following manner. An aliquot of 50 mL of a 6 wt % HF solution was transferred into a container of deionized water. The material to be tested was added slowly, and the pH was monitored (with a pH electrode or pH paper strips) after each addition. The addition of material was stopped when a pH value around neutral was obtained or when no pH change was observed despite a large amount added. The amount of initial fluoride in a diluted aliquot for the batch tests was about 30,000 mg/L.

For powdered chemicals, the theoretical specific gravity was used in the calculation, and a porosity of 50% was assumed. For other materials, a measurement of the apparent density was performed by weighing a known volume of material in a graduated cylinder. The material was not compacted in the cylinder when it was introduced, reproducing the porosity which would result while filling the trap.

The pH and fluoride concentrations were measured at the completion of the test. The pH was measured either with a pH electrode or with pH paper strips. The fluoride concentration was measured by using an ion-specific electrode. The calibration was performed with 1-, 10-, 100-, and 1,000- mg/L standards. Samples having very high concentrations of fluoride were analyzed with the same calibration curve without dilution; therefore, these results are only estimated.

Some of the media were further tested using 3.8-cm ID PVC column connected to a ring stand and filled with a specific media. A peristaltic pump then pumped 6 wt % HF solution at a slow rate through the column, where the effluent was collected in 100-mL samples. The samples were monitored for fluoride content and pH until breakthrough occurred.

3.1.2 Results

Results from the tests varied widely, but two patterns were observed. The first observable pattern was that media with a high surface area (small mesh) tended to perform best. The second pattern showed that the best overall performing media contained $\text{Ca}(\text{OH})_2$. BFS mixed with $\text{Ca}(\text{OH})_2$ [80% $\text{Ca}(\text{OH})_2$ and 20% BFS] performed best with a loading capacity of 0.164 g HF/cm³ of media. The other media which performed well were those comprised of mixtures of $\text{Ca}(\text{OH})_2$ and portland cement (PC), and $\text{Ca}(\text{OH})_2$ by itself. The pure $\text{Ca}(\text{OH})_2$ and the smallest mesh materials (30 mesh) of the variations proved too exothermic for this column and even melted through the side of the PVC column when CaO , in the form of burned lime, was used. Many of the other materials evaluated gave a nominal or poor performance. These results are listed in Appendix B.

For some materials, tests were performed at 20 and 85°C; however, the difference observed between the two temperatures was not significant enough such as to justify the excess time necessary for the hot tests. From the powdered vs granular batch tests, the results obtained at 20°C are presented in Appendix A, while Table 3.1 presents the best performing media.

Two sorbents outperformed the others in their neutralizing and fluoride-binding capabilities, yet they did not react too exothermically such as to cause concern. For the two best performing materials—100% $\text{Ca}(\text{OH})_2$, fraction size 18–30 mesh, and the mixture of 80% $\text{Ca}(\text{OH})_2$ plus 20% BFS, fraction size 18–30 mesh—another set of experiments were performed to evaluate the concentration of fluoride

remaining in the solution (under the same conditions as those described earlier) when various amounts of material were added.

Table 3.1. Best performing media using 6 wt % HF

Media	Weight (g)		Loading g HF/cm ³ media
	Media	HF	
80% Ca(OH) ₂ + 20% BFS (18–30 mesh)	164.5	38.85	0.164
75% Ca(OH) ₂ + 25% BFS (7–30 mesh)	220	38.85	0.123
85% Ca(OH) ₂ + 15% PC (10–20 mesh)	216.2	38.85	0.119
50% Ca(OH) ₂ + 50% BFS (18–30 mesh)	216.3	27.75	0.102
Ca(OH) ₂ (18–30 mesh)	150.3	27.75	0.092
25% Ca(OH) ₂ + 75% BFS (20–30 mesh)	275	27.75	0.088
17% Ca(OH) ₂ + 83% BFS (7–30 mesh)	240	22.2	0.096
75% Ca(OH) ₂ + 25% BFS (7–18 mesh)	202	22.2	0.077
Ca(OH) ₂ (7–18 mesh)	142.5	22.2	0.076

3.2 EVALUATION OF MEDIA USING 33 wt % HF

The following sections are devoted to the setup and results obtained from the testing of 33 wt % HF. This concentration of HF is what is expected from the uranium conversion process.

3.2.1 Setup

Part two involved the testing of media using a more concentrated solution of HF and at a slightly larger scale. A new column was fabricated from clear 2.54-cm-ID PVC. The column measured 38.1 cm in length and had a coupling and cleanout plug at the bottom. The plug had three 0.95-cm (3/8-in.) holes drilled into it for flow and was covered on the inside by a 10.5-squares-per-centimeter polypropylene screen to retain the media. The top of the column was open to allow for pressure equalization and the introduction of HF, but the top also contained a polypropylene screen to distribute the flow and diminish channeling through the media. The column was held in place by a ring stand over a 250-mL beaker, which was placed on a stir plate with a magnetic stir bar and held an initial volume of 50 mL of deionized water. The water and effluent were monitored by an Orion® pH and temperature probe throughout the tests. Another temperature probe was connected to the outside of the column ~2 cm

above the bottom of the media. The effluent was tested intermittently for fluoride using an ion-specific electrode, and flow into the column was stopped if the effluent fluoride level reached 2,000 ppm. Most tests were conducted with ~21.6 cm (109.4 cm³) of media. The HF was made by diluting a 50 wt % solution with deionized water to make a 33 wt % solution. The HF was most commonly introduced into the column using a peristaltic pump at a rate of 3.7–10.7 mL/min, but it was also tested with some media by pipette using 5–20-mL aliquots of HF.

Effluent was caught in the beaker below the cartridge and monitored for breakthrough in pH. When the pH in the beaker dropped below 5.0, the addition of HF to the column was stopped. The effluent was saved and evaporated on a hot plate at ~75°C until all liquid was gone. The dried precipitate from the beaker was then weighed and sampled.

3.2.2 Results

Results from this series of tests proved to be somewhat surprising. Many of the most promising media from the first set of tests performed poorly using the concentrated HF. Results from the tests are found in Appendix C.

All sizes (30 mesh–1.5 cm diam) of the 80% Ca(OH)₂ 20% BFS mix had good neutralizing capability, but (after a vigorous reaction) it was found to partially disintegrate in the concentrated HF. This disintegration led to a slurry, which severely restricted flow and quickly plugged the column. A mixture of 80% Ca(OH)₂ and 20% PC performed somewhat better in neutralizing the HF with a loading efficiency of 0.32 g HF/cm³ media, but the mixture also proved to be highly susceptible to disintegration and plugging, producing 7.2 g of precipitate per 109 cm³ of media.

Sodium hydroxide (NaOH) pellets were tested alone and then mixed with CaCO₃. The former proved too reactive (melting the column in one instance), forming sodium fluoride, which is soluble. Most of the media in the column was converted to NaF and ended up in the collection beaker. Consequently, NaOH was mixed with limestone to slow the reaction and dislodge the sodium ion with a calcium ion to produce an insoluble product of calcium fluoride (CaF₂). This mixture proved partially successful when small amounts of NaOH were used, although still more precipitate was left than occurred with most of the other media.

Three potential compounds from the first test were retested to see if they functioned better with concentrated HF. All three compounds [calcium carbonate (marble pieces), alumina spheres, and silica gel] performed just as poorly at the higher concentration of HF. The poor results stemmed from very little neutralizing capability.

A smaller size of activated alumina (8–14 mesh) was tested, but this alumina reacted much like the calcium hydroxide-based media, producing a slurry that plugged the column. When a sample of activated alumina was combined with soda and lime (40:60 vol %, respectively), the media became ineffective, and the HF broke through almost immediately. In two separate tests, the activated alumina was combined with limestone to see if the limestone might be used as a moderator for the reaction. Again, the activated alumina was too reactive and plugged the column.

Because of evidence that some types of limestone work well as a gas-phase HF neutralizer in tower or column reactors, two samples of limestone were obtained and evaluated. The initial limestone sample was a manufactured sand (<3/8-in. fine aggregate) from the Rogers Group in Oak Ridge, Tennessee. The sand performed well in neutralizing capability at ~ 0.29 g HF/cm³ media, and was the best media for the amount of precipitate evolved (presumably because of its density). A second test using this material was performed at a slower flow rate (3.7 mL/min) to see if the neutralizing capability was flow dependent. The slower rate appeared to have very little, if any, effect on the outcome; in fact, the media performed slightly worse (0.14 g HF/cm³ media). A second sample of limestone was obtained from Genlime, Inc., in Genoa, Ohio. This sample was a dolomitic limestone with about the same characteristics as the first sample. It, however, performed poorly and was unable to neutralize the HF at even the slowest rates.

The overall media of choice from the tests was a mixture of soda and lime (soda lime). It did not disintegrate and proved to be a good neutralization agent. Many sizes were tested, and the best formula proved to be a mix of sizes with 4–8 mesh on top of 7–18 mesh. With a loading efficiency of ~ 0.5 g HF/cm³ soda lime, it holds promise to be used for about seven batches of HF per cartridge. Results from the most promising forms of media are found in Table 3.2.

3.3 DELIVERY MECHANISMS

Because of the highly reactive nature of the trapping media and its tendency to slurry and plug in concentrated solutions of HF, several systems were attempted. One delivery system involved introducing the HF through a diffuser tube down the center of the media. In theory, this process would increase the surface of the reacting front so that the column would be less susceptible to plugging. The results were much the same, however. Because such a vigorous reaction took place at each of the diffuser holes, slurry was pushed into the diffuser tube, causing the tube to clog.

Another method of HF introduction was by aliquot transfer. Aliquots of 5–50 mL were transferred (via pipette) into the top of the column and allowed to react and slowly drain before another aliquot was transferred. This method did not yield any better results, although an interesting phenomenon was repeatedly observed. If the introductory aliquots were small, they would sometimes result in a higher capacity and less dissolution and plugging of the media when larger aliquots were added later. It also indicated that some passivation of the media might be taking place.

Table 3.2. Best performing media using 33 wt % HF^a

Media	Packing g/cm ³	Loading (g HF/cm ³ media)	Expected ^b batch/ column	Precipitation (g)	Expected ^b precipitation/ column (g)	HF flow (mL/min.)
Ca(OH) ₂ /BFS (7–18 mesh)	0.40	0.200	2.78	1.21	73.83	10.7
Ca(OH) ₂ /PC (7–18 mesh)	0.48	0.323	4.49	7.2	439.31	10.7
Limestone (<3/8 in. diam)	1.61	0.290	4.04	2.7	164.74	10.7
Limestone (<3/8 in. diam)	1.63	0.234	3.25	1	61.01	3.7
Limestone/2.5% NaOH	1.71	0.358	4.98	5.5	335.58	3.2
Limestone/5.1% NaOH	1.72	0.489	6.79	6.9	421.00	2.8
NaOH pellets	0.43	0.427	6.03	Column melted		8.3 ^c
NaOH pellets	0.43	0.420 ^d	>16.53	60.1	10,557.04	10.7
Soda lime (4–18 mesh)	0.74	0.516	7.17	4.6	280.67	10.7
Soda lime (4–18 mesh)	0.74	0.537	7.47	4.7	286.77	10.7
Soda lime (4–18 mesh)	0.73	0.457	6.35	N/A	N/A	15 ^e

^aBased on 10.7-mL/min flow of 33 wt % HF solution into 1.0-in. column containing 109.4 cm³ media.

^b“Expected” results are based on a column 4.026 in. diam × 32 in. long with capacity of ~6,675 mL. Assumes 480 g HF/batch.

^cIndicates flow in slugs (i.e., by pipette) vs metered flow.

^dIndicates that column still had some neutralizing ability but HF stock solution ran out.

4. CONCLUSION

HF is a reactive and potentially dangerous by-product of the conversion of UF_6 to U_3O_8 . Because of its extreme (and sometimes violent) corrosivity and health effects, it is desired that the concentrated HF be neutralized soon after its formation. The purpose of this study was to identify the solid sorbent that would most effectively neutralize the HF. Because of criticality, space, and radiation constraints, it was desired to use the sorbent in a disposable cartridge, which could be easily replaced.

After two thorough tests of many different types and combinations of media, including a study of three types of delivery mechanisms, the conclusion is that there seems to be no all-inclusive media. Different concentrations of HF are handled better by different media because of the characteristics of dilute vs concentrated HF.

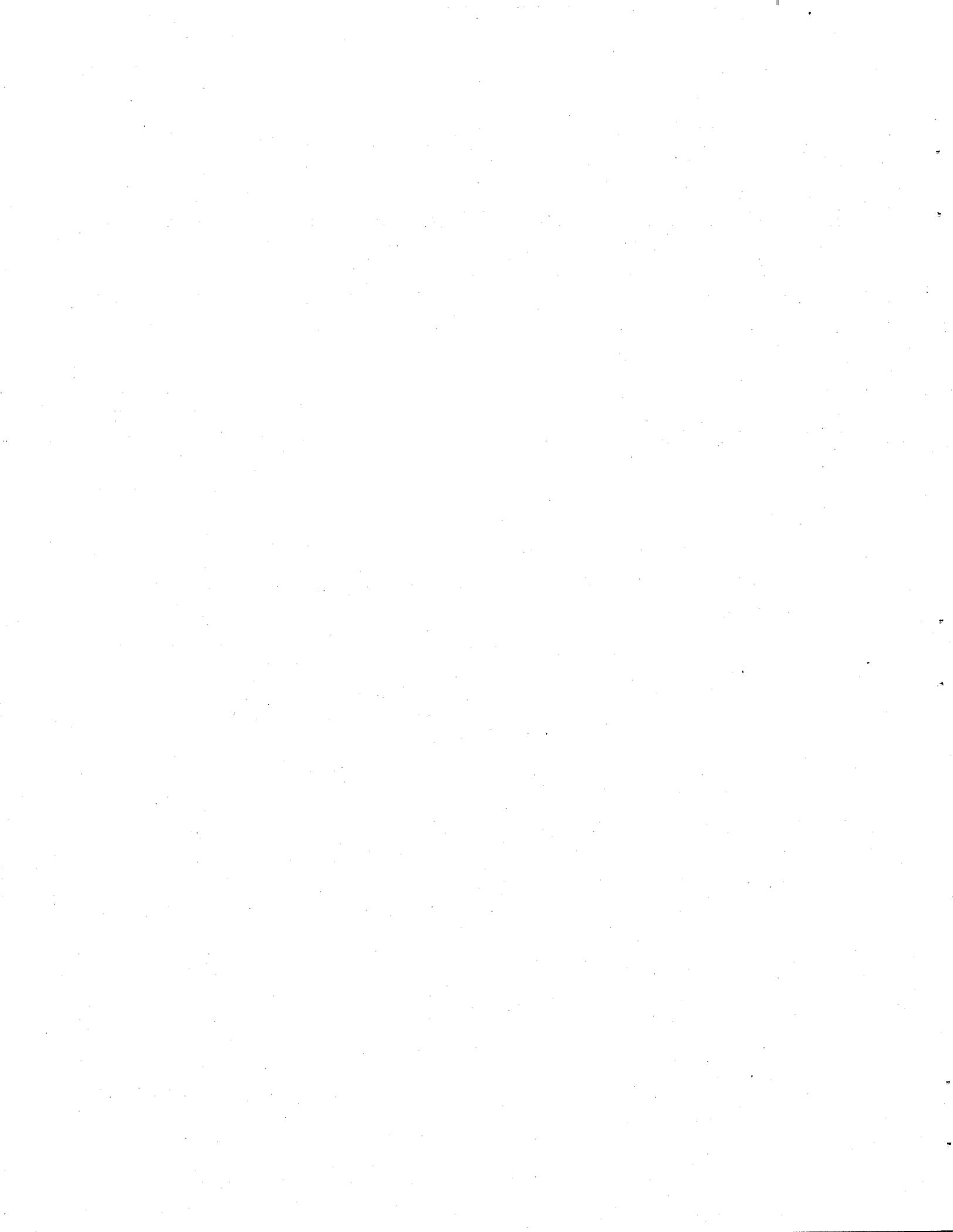
The first half of the study involved observing interactions of 6 wt % HF with different sorbents in batch tests. Once a promising sorbent was identified, it was then tested in a column with a metered flow of HF. The observed pattern from part one of the media evaluation indicated that the best media for the process would be a high-surface-area (small mesh) and calcium-hydroxide-based material. The media which outperformed all others at sorbing the 6 wt % HF solution was a mixture of 80% $Ca(OH)_2$ and 20% BFS. All sizes of this material performed well, but the 18–30 mesh material proved to be the best at sorbing the HF.

Part two of the media testing involved flowing concentrated (33 wt %) HF through a column filled with different sorbents to test for reactivity and effluent. The higher concentrated solution revealed a much different pattern. The higher concentration of HF dramatically changed the results of much of the media testing. The observable pattern from this set of tests indicated that a lower surface area and harder material would be needed to stand up to the high corrosivity of a 30% HF solution. The media that performed best was soda lime at sizes of 4–18 mesh. Soda and lime together neutralized up to 0.5 g HF/cm³ sorbent when using the buffering capacity of the total effluent. Crushed limestone also proved noteworthy with a high durability, a low dissolution rate, and good neutralization capabilities.

With respect to delivery mechanisms, metered flows tended to provide the best and most consistent results. Slugs of HF solution passed through pipettes gave variable results. It appears that the size of the first aliquots of solution may play a critical role in how the media performs overall, but further testing will be needed for verification. Introduction of HF by a diffuser tube worked slightly better than adding aliquots to the top of the media in some instances; however, even for these tests, the column plugged.

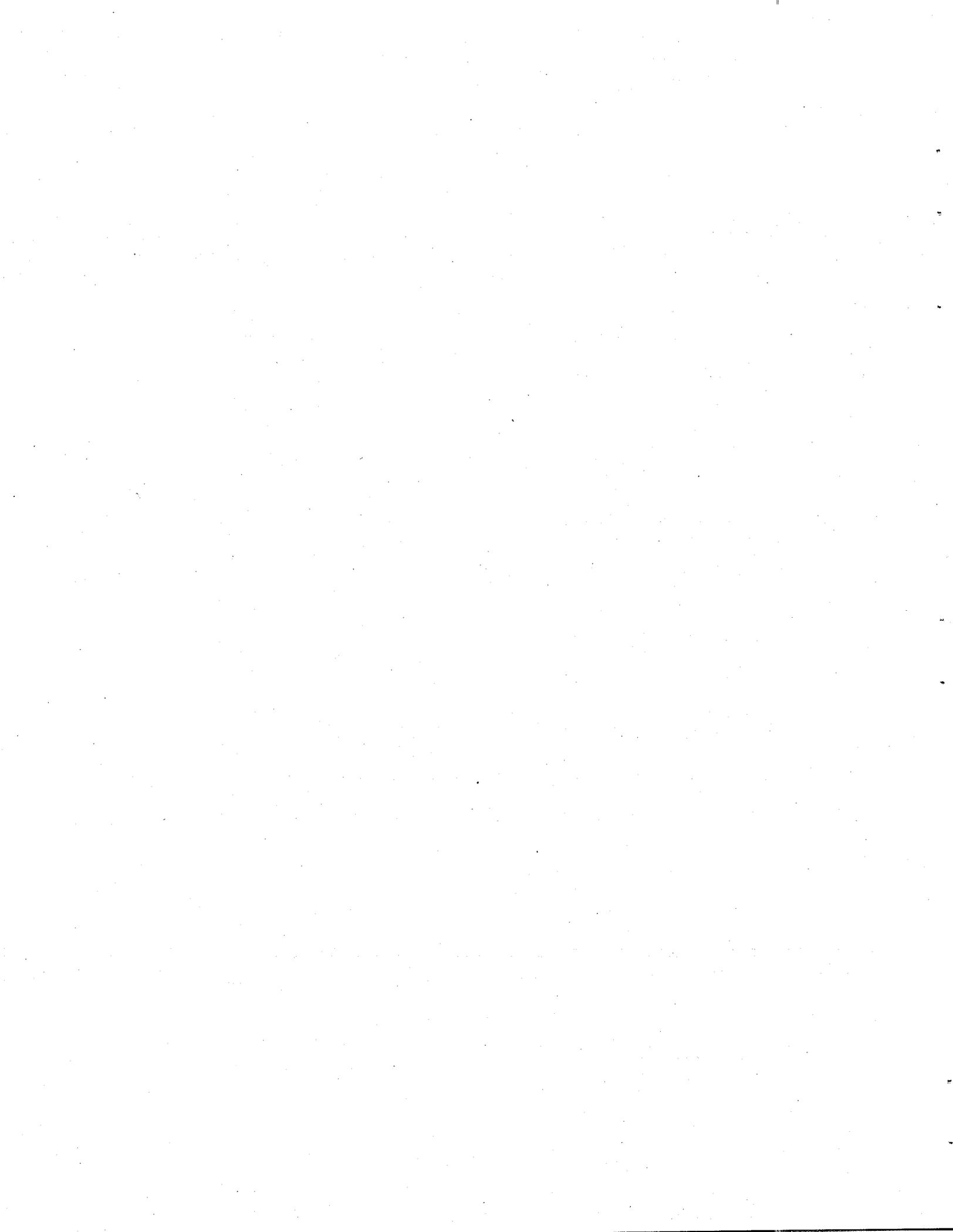
Based on the results of this study, it is recommended that soda lime be used as the trapping media for the conversion system. Because 18–30 mesh material is not a standard size, a slightly larger size of 6–12

mesh, in conjunction with 4-8 mesh, has been proven to be nearly as effective and is the recommended sorbent. The method of introduction of HF into the cartridge should be a metered flow of ~20 mL/min. Using a soda lime-filled cartridge ~81 cm in length and 10.2 cm diam, it is expected that ~3,800 g of HF can be effectively neutralized before the pH in the effluent collection drops below 5. This amount of HF equates to about 7-8 UF₆ traps being processed in the conversion system.



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APPENDIX A: Summary of Batch Tests

Table A.1. Summary of the batch tests at 20°C with materials in a powdered form

Material tested	Density (g/cm ³)	Wt sorbent (g)	Vol sorbent (cm ³)	Residual [F] (mg/L)	mg F ⁻ / cm ³ of sorbent	Final pH
Al ₂ O ₃ (80–200mesh)	2.2	86.32	39.236	2,057	712.2	4.8
Al(OH) ₃ (powder)	2.42	99.36	41.058	1,606	691.6	2.8
CaCO ₃ (powder)	2.8	21.97	7.846	466	3,764.2	7
Ca(OH) ₂ (powder)	2.24	6.07	2.710	234	10,983.8	10.5
CaSiO ₃ (powder)	2.9	11.82	4.076	51	7,347.6	8.5
4MgCO ₃ Mg(OH) ₂ 5H ₂ O (powder)	2.16	11.06	5.120	435	5,774.4	7
Mg(OH) ₂ (powder)	2.36	12.62	5.347	688	5,482.0	8.6
Magnesium aluminate (powder)	3.6	64.59	17.942	2,387	1,539.0	2.4
Sodium aluminate (powder)	2.6	6.1	2.346	2,241	11,832.5	10.1
Fluorisil (powder)	NA ^a	15.29	NA	8,483	NA	3.2
BFS (powder)	2.74	23.7	8.650	213	3,443.6	8.4
Class C fly ash (powder)	2.4	24.9	10.375	332	2,859.6	7.4
Class F fly ash (powder)	2.37	46.5	19.620	4,812	1,233.3	1.8
Diatomaceous earth (powder)	NA	24.57	NA	35,000	NA	1
Pumice stone (powder)	NA	53.59	NA	28,000	NA	1
Celite 503 (powder)	NA	34.4	NA	35,000	NA	1
Kaolin (powder)	NA	30.3	NA	1,836	NA	1
<u>Anhydrous PC type II (powder)</u>	3.2	44.08	13.775	493	2,142.1	11

^aNA = not applicable.

APPENDIX A: Summary Batch Tests

Table A.2. Summary of the batch tests at 20°C with materials in a granular form

Material tested	apparent d (g/cm ³)	Wt sorbent (g)	Vol sorbent (cm ³)	Residual [F] (mg/L)	mg F/ cm ³ of sorbent	Final pH
Sodium borate (granular)	1.27	39.1	30.684	8,450	702.3	6.9
Marble chips (big pieces)		14.9	11.693	14,343	1,339.0	2.5
Dolomitic limestone (spheres)	1.39	42.8	30.836	1,049	938.9	3.7
Molecular sieve 5A (8–12 mesh)	0.73	30.08	41.205	1,914	681.6	4.0
Molecular sieve 3A (8–12 mesh)	0.73	35.7	48.770	1,301	588.4	6
Molecular sieve 4A (8–12 mesh)	0.73	36.2	49.589	3,085	542.8	4.7
Soda lime (8–12 mesh)	0.63	34.92	55.795	22	537.3	13
Molecular sieve 13X (30–60 mesh)	NA	31.55	NA	945	NA	5
17% Ca(OH) ₂ – 83% BFS (7–20 mesh)	0.85	31.38	36.918	89	810.2	10
25% Ca(OH) ₂ – 75% BFS (7–18 mesh)	0.84	77.77	92.583	1,957	302.9	11
50% Ca(OH) ₂ – 50% BFS (7–18 mesh)	0.65	50	76.455	909	380.5	11
75% Ca(OH) ₂ – 25% BFS (7–18 mesh)	0.57	30.9	54.290	1,037	533.5	12
100% Ca(OH) ₂ (7–18 mesh)	0.40	20.6	50.880	108	587.5	13
Burned lime CaO (7–18 mesh)	0.87	13.27	15.307	380	1,935.1	14
Sodium aluminate (7–16 mesh)	0.83	15	18.016	1,482	1,582.9	13
Dolomitic limestone (7–16 mesh)	1.41	65	45.990	474	642.0	4–5
85% Ca(OH) ₂ – 15% PC type II (7–20)	0.54	25	46.183	663	635.2	12
Hydrated PC type II (7–20mesh)	1.04	56.32	54.133	6,100	441.5	3–4
Limestone (7–20mesh)	1.49	87.93	59.170	3,943	440.4	2
Marble chips (7–20mesh)	1.49	98	65.772	29,680	4.9	1
Titanium sponge (fine)	1.53	7.11	4.638	43,600	NA	5
Titanium metal (coarse)	1.53	7.4	4.837	49,300	NA	5
Magnesium (40 mesh)	NA	19.97	NA	46,300	NA	1
50% Ca(OH) ₂ – 50% BFS (7–10 mesh)	0.65	30	46.487	337	632.1	9–10
50% Ca(OH) ₂ – 50% BFS (10–18 mesh)	0.64	30	46.928	429	630.1	12
50% Ca(OH) ₂ – 50% BFS (18–30mesh)	0.60	30	49.669	518	593.6	12
50% Ca(OH) ₂ – 50% BFS (<30mesh)	0.60	20	33.333	65	898.0	12
25% Ca(OH) ₂ – 75% BFS (7–10mesh)	0.77	88.2	113.925	308	260.6	11–12
25% Ca(OH) ₂ – 75% BFS (10–16mesh)	0.75	67.41	89.403	130	334.1	12
25% Ca(OH) ₂ – 75% BFS (16–20mesh)	0.72	52.05	72.430	123	412.5	11–12
25% Ca(OH) ₂ – 75% BFS (20–30mesh)	0.71	41.18	58.321	127	512.2	11–12
50% Ca(OH) ₂ – 50% BFS (<30mesh)	0.77	20.88	27.060	65	1,106.2	10–11

APPENDIX B: Media Summary for 6 wt % HF

Table B.1. Media summary for 6 wt % HF

Media	Conditions	Result	HF sorbed (g/cm ³)
Sodium aluminate powder	Slow metered	Bad (plugged)	NA
Sodium aluminate–limestone (67:33%)	Slow metered	Bad (plugged)	NA
Calcium silicate–dolomite (37:63%)	Slow metered	Bad (plugged)	NA
Molecular sieve 3A–dolomite–calcium silicate (33:43:23%)	Slow metered	Bad (plugged)	NA
Sand (30–100 mesh)–sodium aluminate (76:24%)	Slow metered	Bad (little reaction)	NA
Silica gel–sodium aluminate (67:33%)	Slow metered	Bad (little reaction)	NA
Dolomite–Ca(OH) ₂ (81:19%)	Slow metered	Bad (plugged)	NA
Silica gel–sodium aluminate (45:55%)	Slow metered	Bad (little reaction)	NA
Sand (20–30 mesh)–sodium aluminate (66:34%)	Slow metered	Bad (plugged)	NA
Sand (20–30 mesh)–Ca(OH) ₂ (76:24%)	Slow metered	Bad (plugged)	NA
Dolomite	Slow metered	Bad (plugged)	NA
Molecular sieve 3A (8–12 mesh)	Slow metered	Bad (little reaction)	NA
Sand (20–30 mesh)–sodium aluminate (73:27%)	Slow metered	Bad (little reaction)	NA
Sand (20–30 mesh)–Ca(OH) ₂ (86:14%)	Slow metered	Bad (plugged)	NA
Sand (20–30 mesh)–calcium silicate (72:28%)	Slow metered	Bad (slow flow)	NA
17% Ca(OH) ₂ – 83% BFS (7–16 mesh)	Slow metered	Good	0.072
Sodium aluminate (7–16 mesh)	Slow metered	Bad (plugged)	NA
Molecular sieve 13X	Slow metered	Bad (little reaction)	NA
17% Ca(OH) ₂ – 83% BFS (16–30 mesh)	Slow metered	OK (plugged)	0.076
17% Ca(OH) ₂ – 83% BFS	Two size 7–30 mesh Slow metered	Good	0.096
17% Ca(OH) ₂ – 83% BFS	Two size 7–30 mesh Slow metered	OK (plugged)	0.071
17% Ca(OH) ₂ – 83% BFS (7–16 mesh)	Slow metered	OK (too slow)	0.048
17% Ca(OH) ₂ – 83% BFS (7–16 mesh)	Slow metered	OK (too slow)	0.073
25% Ca(OH) ₂ – 75% BFS (7–20 mesh)	Slow metered	OK (too slow)	0.035
75% Ca(OH) ₂ – 25% BFS (7–18 mesh)	Slow metered	OK	0.077
Limestone (7–16 mesh)	Slow metered	OK (too slow)	0.038

Table B.1. Media summary for 6 wt % HF

Media	Conditions	Result	HF sorbed (g/cm ³)
100% Ca(OH) ₂ (7-18 mesh)	Slow metered	OK (too slow)	0.076
100% CaO (7-18 mesh)	Slow metered	Bad (plugged)	NA
Hydrated PCII (7-20 mesh)	Slow metered	OK (too slow)	0.039
75% Ca(OH) ₂ - 25% BFS	Two size 7-30 mesh Slow metered	Good	0.123
85% Ca(OH) ₂ - 15% PCII (10-20 mesh)	Slow metered	Good	0.119
25% Ca(OH) ₂ - 75% BFS (20-30 mesh)	Slow metered	OK (too slow)	0.088
50% Ca(OH) ₂ - 50% BFS (18-30 mesh)	Slow metered	Good	0.102
Ca(OH) ₂ (16-30 mesh)	Slow metered	Good	0.092
66.6% Ca(OH) ₂ - 33.3% CaSiO ₃ (16-30 mesh)	Slow metered	Bad (plugged)	NA
Silica gel (8-20 mesh)	Slow metered	Bad (little reaction)	NA
80% Ca(OH) ₂ - 20% BFS (18-30 mesh)	Slow metered	Good	0.164

APPENDIX C: Media Summary for 33 wt % HF

Table C.1. Media summary for 33 wt % HF

Media	Conditions	Result	Expected Batch/Col.	Expected Precip/Col.
Al ₂ O ₃ (8-14 mesh)	9.2 mL/min metered	Bad (plugged)	NA	NA
Al ₂ O ₃ (alumina spheres)	10.7 mL/min metered	Bad (too fast)	NA	NA
Ca(CO) ₃ (marble chips)	10.7 mL/min metered	Bad (too fast)	NA	NA
Ca(OH) ₂ - BFS 1-2 cm diam	25 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 18-30 mesh	10 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 2 cm - 30 mesh	3 size, strata, 25 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 2 cm - 30 mesh	3 size, wet first, 25 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 7-18 mesh	10-25 mL pipette	Good	4.49	439.3
Ca(OH) ₂ - BFS 7-18 mesh	25-50 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 7-18 mesh	10 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 7-18 mesh	Cooled HF, 10 mL pipette	OK	2.96	NA
Ca(OH) ₂ - BFS 7-18 mesh	Diffuser tube, 10 mL pipette	OK	1.69	NA
Ca(OH) ₂ - BFS 7-18 mesh	Diffuser tube, 10 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 7-18 mesh	Diffuser tube, 10 mL pipette	Bad (plugged)	NA	NA
Ca(OH) ₂ - BFS 7-18 mesh	10.7 mL/min metered	OK (plugged)	2.78	NA
Ca(OH) ₂ - MgO powder	4.2 mL/min	Bad (destructured)	NA	NA
Ca(OH) ₂ - PC II (7-18 mesh)	10.7 mL/min metered	OK (plugged)	4.48	439.3
Dolomitic limestone pellets	10.7 mL/min metered	Bad (plugged)	NA	NA
Limestone - GenLime	2.3 mL/min metered	Bad (little reaction)	NA	NA
Limestone (< 3/8 in.) - Rogers	10.7 mL/min metered	Good	4.04	164.7
Limestone (< 3/8 in.) - Rogers	3.7 mL/min metered	Good	3.25	61
Limestone-Al ₂ O ₃ (50:50%)	9.9 mL/min metered	Bad (plugged)	NA	NA
Limestone-Al ₂ O ₃ (80:20%)	9.9 mL/min metered	Bad (plugged)	NA	NA
Limestone-NaOH (84:14%)	3 strata, 10.7 mL/min	Bad (plugged)	NA	NA
Limestone-NaOH (85:15%)	Mixed, 10.7 mL/min	Bad (plugged)	NA	NA
Limestone-NaOH (97.5:2.5%)	3.2 mL/min metered	Good	4.98	335.6
Limestone-NaOH (95:5%)	2.8 mL/min metered	Good	6.79	421
NaOH pellets	5-20 mL pipette	OK (destructured)	86.92	NA

Table C.1. Media summary for 33 wt % HF

Media	Conditions	Result	Expected Batch/Col.	Expected Precip/Col.
NaOH pellets	10.7 mL/min metered	Good	> 16.53	10,557
Silica gel (18 mesh)	5 mL pipette	Bad (too fast)	NA	NA
Soda lime (18-30 mesh)	10-25 mL pipette	OK (plugged)	3.38	NA
Soda lime (4 mesh)	10-25 mL pipette	Good	3.59	NA
Soda lime (4-18 mesh)	10.7 mL/min pipette	Good	7.47	286.8
Soda lime (4-18 mesh)	10.7 mL/min pipette	Good	7.17	280.7
Soda lime (4-8 mesh)	10-25 mL pipette	Good	2.33	NA
Soda lime (6-12 mesh, dried)	9.9 mL/min metered	Good	8.08	530.8
Soda lime (7-30 mesh)	2 size, 10-25 mL pipette	Good	4.65	NA
Soda lime-Al ₂ O ₃ (60:40%)	9.2 mL/min metered	Bad (little reaction)	NA	NA
Sodium Aluminate powder	10.7 mL/min metered	Bad (plugged)	NA	NA

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