Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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221H-LIB-F-21-003

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November 15, 1985

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EVALUATION OF TEST AUTHORIZATION #2-1102
IODINE REMOVAL USING SILVER MORDENITE

SUMMARY

The original Test Authorization (TA)[1] is evaluated. A new silver mordenite (Ag°Z) cartridge design has been developed and presented to Separations personnel. They expressed interest in the design, which should provide higher iodine removal efficiency, longer bed life, and lower personnel exposure than the present Berl saddle cartridge. A sampling program to obtain iodine DF data for the existing reactor packing has been started. The indirectly measured ^{129}I DF values obtained thus far are 2 to 4. Separations Technology reported an average annual overall process ^{129}I DF of 4.7.

The future direction for this TA, mutually agreed to by SRP and SRL personnel, is to:

- Document current program status. (Hsu, via this memorandum)
- Continue the sampling program to refine the method and gather baseline DF data. (Hsu, HP)
- Finalize new cartridge design and obtain cost and timing estimates for its procurement. (Hsu, Separations)

- Complete cost, benefit analysis for silver mordenite. (Hsu, Sep Tech)

- Conduct literature search on available technology to divert more iodine to the dissolver off-gas and thus to the iodine reactor. (Hsu)

A decision on whether to conduct a plant test will be made after the last three items are completed.

INTRODUCTION

The dissolution of uranium fuel slugs releases iodine-129 and iodine-131. Historically, SRP has always used a $AgNO_3$-coated Berl saddle packed bed to remove iodine from the dissolver off-gas (DOG). The average bed life of the reactor (vessel 7.1R) in F-Area is about ten months; the shortest is one month.[2] The average bed life at H-Area is in excess of five years.[3] The less than expected reactor life at F-Area appears due to plugging by melted and washed away $AgNO_3$,[3] solids from deterioration of the Berl saddles, and solids from entrained caustic solution.[2]

TA 2-1102 was routed for approval to test silver mordenite as an alternative adsorbent in iodine removal. Silver mordenite offers the benefits of more efficient removal of iodine;[3,4,5] therefore, it would result in lower offsite releases, lower waste volume, and reduced radiation exposure to personnel. Moreover, successful testing of mordenite provides an alternate iodine adsorbent, as future supply of $AgNO_3$-coated Berl saddles is uncertain.[1] Though the current emission levels are well within the DOE concentration guide,[6] future regulatory changes may require more efficient iodine removal.[7,8]

In August 1985, the chemical stability of silver mordenite was questioned. It was found that Ag°Z disintegrated in a boiling 2 M NaOH solution.[9] When drops of simulated dissolver solution were added to mordenites at 200°C, the liquid bubbled and evaporated, and mordenite extrudates fused together in the beaker.[10]

As a result, the present evaluation was initiated to study the engineering aspects of the original TA and to propose a new cartridge design if necessary. This report describes the results of the evaluation, proposes a new Ag°Z packed bed design, and documents the consensus of SRL and SRP personnel on this program.

PRESENT IODINE SILVER REACTOR

Silver Nitrate-Coated Berl Saddles

SRP has used silver nitrate-coated ceramic Berl saddles to remove iodine from the DOG stream since plant start-up. The $AgNO_3$-coated Berl saddles have performed smoothly in H-Area with average reactor bed life greater than five years.[3]

The silver reactor in F-Area, however, has not always operated as smoothly.[2]

Generically, it is reported[11] that, although the silver reactor is capable of DF's up to 10^3, it has historically operated well below that. Having recently reviewed the Hanford Purex silver reactor performance, Evoniuk (1981)[12] concludes that an average DF of 100 is probably achievable. One possible reason for the poorer-than-expected performance is the reactor's narrow effective temperature range. If the temperature is too low, entrained acid/caustic and water wash off the silver nitrate; if too high, the silver nitrate melts and drains away. It's also possible the captured iodine species is not stable at process conditions. In addition, a fully acceptable waste treatment process has not been developed for the $AgNO_3$- coated Berl saddles. Simple equipment and low operating costs, excluding the cost of silver, are the saddles' advantages.

Description of the Reactor

The F-Area silver reactor consists of a reactor shell containing a removable cartridge or "basket," Figure 1. The cartridge contains about 25 cubic feet of 1/2-inch $AgNO_3$-coated Berl saddles.[13] The cylindrical cartridge with slight taper at the very bottom is two feet in outside diameter and is packed to a depth of about eight feet. The cartridge has an open top fitted with a cross-shaped beam structure with a lifting bail formed of 1-inch stainless steel rod. There is also a screen cover with a 1 3/4-inch wide slot at the center to slide over the lifting bail as it covers the cartridge. The bottom of the cartridge is a grating made of thin bars on 3/8-inch centers.[13]

The reactor sits in a steam jacket[14] through which the DOG enters at the top through two 4-inch pipes coiled helically downward around the reactor. Near the bottom of the steam vessel, the two DOG-carrying pipes turn upward to feed the iodine reactor from the bottom.[15,16] The 150 psig steam in the jacket provides a temperature of about 185°C. As the DOG

goes through the reactor, the iodine is adsorbed by and reacted with $AgNO_3$. The DOG stripped of iodines then leaves the reactor at the top. Reactor 7.1R was installed in 1964.[2] Since that time, 24 cartridges have been used with an average life of 9.8 months; the shortest life was one month, and the longest 35 months.

Problems with the Reactor

DOG from both the caustic decladding and the acidic dissolution cycles goes through the same iodine silver reactor.[16]

Plugging of the cartridge is conceivably due to the two following causes:[3] (1) Introduction of caustic dissolver solution into the iodine reactor where, as the water in the solution is evaporated off, solids are left behind, slowly plugging the bed. This can be from normal entrainment or more likely abnormal flooding. (2) Chemical degradation of the Berl saddles and the washing away and melting off of the $AgNO_3$ produce materials that settle and eventually plug at the bottom of the cartridge or pipes. The design of the cartridge/reactor system[14] does not allow entrapped solution to drain. The moisture slowly evaporates and solids stay behind. Saddle degradation is probably due to alternate exposure to base and acid vapor and solution during decladding and dissolution cycles.[2] It is also possible to have localized temperatures exceeding the melting point of $AgNO_3$ due to exothermic reaction between NH_3 and $AgNO_3$.[17]

MORDENITE

Mordenite is a commercially available molecular sieve material with a silica/alumina ratio of 10/1[18] which gives the material good acid and thermal stability. Zeolon 900® extrudates are stable to reflux (103°C) in 6 N HCl and in environments with pH as high as 12.[18] Typically, these molecular sieves have internal surface area in the range of 800 m^2/g.[19]

Silver mordenite lowers the waste volume produced because its silver concentration is much higher than that of $AgNO_3$-coated Berl saddles; for a given amount of iodine removed, a much smaller quantity of silver mordenite is required. Fully exchanged silver mordenite contains 19 weight percent versus 3 weight percent silver on Berl saddles, Table 1. In addition, more of the silver in mordenite is available to react with iodine,[3] because of its higher internal surface area.

Thus, 2.6 ft^3 of Ag°Z will collect as much iodine as the present 25 ft^3 of $AgNO_3$-coated Berl saddles.[3,20] A standard 30-gallon drum should be adequate to contain the smaller volume of 2.6 ft^3 of Ag°Z.

Since Ag°Z is more efficient in removing iodine, the offsite releases would be lower.[3] Radiation exposure to personnel occurs during the handling of the waste container for burial. Since it takes less time to load, seal, and remove a 30-gallon drum than a container 10 times as large, the total radiation exposure should be less with the smaller waste volume.

Very promising results have been obtained using Ag°Z in a small scale system for the adsorption of simulated LWR (light water reactor) fuel reprocessing DOG.[4] The DF's thus obtained are 10^3 to 10^4 for gases containing up to 6% H_2O, 2% NO_2, and 2% NO. A bed loading about 190 mg I_2/g Ag°Z was achieved.

The Separations Technology Laboratory conducted tests to determine the chemical resistance of silver mordenite.[9] Silver mordenite disintegrated when separately soaked in 2 M and 19 M caustic at 85-100°C. Tests were also run in SRL to simulate entrainment of dissolver solution into a cartridge operating at 175-200°C.[10] Liquid bubbled and evaporated from the hot silver mordenite extrudates, the material turned dark gray, and the extrudates fused together.[10]

ENGINEERING EVALUATION OF TA 2-1102

The proposed test details are described in the original TA[1] as follows:

> Silver mordenite will be tested in F-Canyon using an existing reactor cartridge. The bottom 24 inches of the cartridge will be packed with stainless steel wool to act as a demister. About 150 pounds of silver mordenite will be placed on a No. 7 or 8 mesh screen on top of the steel wool. The initial pressure drop across the reactor is expected to be 5 to 10 inches of water column which is higher than with Berl saddles. The higher pressure drop is due to the smaller particle size (1/8 inch extrudate) of the mordenite. However, the higher initial pressure drop should cause no problems during dissolver operation. The reactor operating temperature will continue to be 170 to 190°C. The existing stack iodine monitor will be used to determine if iodine releases change during the test.

Several comments can be made about the original proposed packing design:

1. The 24-inch thick layer of steel wool will collect any entrained liquid. Because of fine voids, the thick steel wool layer will also promote plugging and increased pressure drop.

2. With 10 inches of mordenite and 2 feet of steel wool, the total height is less than 3 feet from the bottom of the reactor, Figure 1. With the thermowell hanging 1'8" from the top of the cartridge, there is a gap of 3'8" between the thermowell and the top of the mordenite bed. The reactor bed temperature, an important operating variable, cannot be measured directly.

3. The volume above the Ag°Z bed which could be used to raise the bed height and reduce the probability of liquid reaching the mordenite was not utilized.

4. Also, the practical aspects of installing 2 feet of steel wool and 150 lbs of mordenite evenly at the bottom of the 9-foot cartridge with cross beams and a thermowell in the way was not considered.

NEW SILVER MORDENITE BED DESIGN

Due to the deficiencies identified in the original TA, a modified design of the silver mordenite cartridge was developed.

Conceptual Design

To structurally convert the old cartridge design to the new configuration, the cross beams anchoring the lifting bail will be lowered, while the lifting bail will be extended upward by the same 14 1/2 inches, Figure 2. A 23-inch high mordenite cartridge with a slightly smaller diameter will sit on the lowered crossed beams inside the reactor cartridge. The top lip of the mordenite cartridge is welded to a 3-foot O. D. flange. The reactor cover, the mordenite-cartridge flange, and the large cartridge flange will all be bolted together with gasket materials between the sealing surfaces. In the center of the small cartridge, there is a 17-inch slot-shaped well that will slide over on the lifting bail. Several layers of Koch or similar mist eliminator mesh[21] supported by a rigid

frame will be at the bottom of this cartridge. At the center of the cross beams in the large cartridge, an oval stainless steel plate with a vertical sleeve will prevent DOG from bypassing the mordenite bed, Figures 2 and 3. The small cartridge can be lifted out separately for maintenance or disposal by the pivoting bail welded to its top cross beam.

The bottom 7 feet of the large reactor cartridge will be filled with 1-inch 304 stainless steel Pall rings.[22] The deep bed of Pall rings (Figure 2) will demist, collect solids, and reduce gas velocity by 20% due to increased cross sectional area as compared to existing Berl saddles. Since the randomly dumped Pall ring bed has a 94% void volume,[22] there is very little pressure loss.[23] This large surge volume below the mordenite bed greatly reduces the probability of caustic solution reaching the bed.

If the mordenites are fused and cannot be poured out, the small cartridge can be lifted out and its contents loosened with caustic. It may be reasonable to consider the small cartridge as disposable to reduce personnel exposure associated with repacking.

The total initial pressure drop across the entire length of the reactor cartridge is calculated to be less than 5 inches of water.[23,24]

Benefits

The new packed bed system will greatly reduce the likelihood of caustic dissolver solution reaching the silver mordenites. Several potential benefits of Ag°Z can therefore be realized:

- Iodine removal efficiency will be improved reducing offsite dose.
- Pluggage caused by melted silver nitrate can be eliminated.
- Longer bed life.
- Lower personnel exposure.
- Waste volume will be reduced.
- Easier maintenance (change-out).
- Lower operating cost.

Safety

No safety problems associated with the use of Ag°Z have been identified.[1] Personnel radiation exposure may be reduced due to the easier access of the much smaller mordenite cartridge and the smaller volume of packing handled. Personnel exposure may be reduced even further if the small cartridge is disposed as a whole.

Risks of Plant Test

The chance of the new silver mordenite bed failing prematurely should be less than with the present Berl saddles. Below the mordenite bed, there is 7 feet of Pall rings with a 94% void volume versus 66% for the Berl saddle bed. The Berl saddle bed has no surge volume below it in the cartridge. The 20.7 ft^3 surge volume of the pall ring zone is larger than the 16.6 ft^3 void volume of the entire current saddle bed. Caustic solution will wash away the $AgNO_3$ coating and attack the ceramic saddles when it gets into the cartridge. However, when that same flooding volume of caustic solution that would cover the entire saddle bed gets into the new mordenite cartridge, it reaches a height of only 5 feet 7 inches. This flood level is still 17 inches below the mordenite bed.

Even if it does fail, a spare cartridge is available for prompt packing with Berl saddles and insertion into the reactor. Also, the new cartridge can be easily modified for use as a spare Berl saddle cartridge.

In the worst possible scenario, the mordenites are completely fused in the small cartridge. In such event, the small cartridge can be separately removed and cleaned and its mesh bottom replaced, or the whole small cartridge may be replaced to reduce personnel exposure. If the pall-ring zone is seeing a large pressure increase, the whole cartridge can either be soaked and cleaned out or the plugged pall rings can be replaced with new ones. However, if the Ag°Z test is to be discontinued, the pall rings can simply be poured out, the small cartridge re-inserted without its bottom, and the main cartridge loaded with the saddles. The operation then returns to the traditional configuration.

F-Area Versus H-Area as Test Site

H-Area is not as suited as F-Area for the Ag°Z plant test for the following reasons:

In H-Area:

- I_2 emissions are much lower (2% into DOG versus 50% at F-Area[25]).
- Iodine reactor life is much longer (> 5 years).
- The required sampling capacity is not available.
- Vacuum capability is limited.
- Presence of Hg may complicate evaluation of the Ag°Z effectiveness for iodine removal.

Therefore, F-Area is recommended as the site for a plant test.

MONITORING PROGRAM

A sampling program for F-Area iodine reactor performance was initiated this September. It gathers baseline iodine reactor DF data and prepares for the monitoring needs of a plant test. Routine weekly charcoal and molecular sieve stack samples are taken and measured for ^{129}I and ^{131}I by HP and SRL (ETD). In addition, the sand filter discharge sample was reactivated. A F8 sample taken between the NO_x adsorber and the stack was also added for direct measurement of the dissolver iodine releases, Figure 4.

Indirectly, the difference between the releases at the main stack and the sand filter discharge gives the iodine release from the reactor. The iodine input to the reactor is assumed to be one-half of the calculated iodine content of the slugs charged to the dissolver.[8,25,26] On this basis, for the period of September 3-24, the ^{129}I DF is 3, and the ^{131}I DF is 15, Table 2. This compares with an average annual overall process DF of 4.7 for ^{129}I reported by Sep Tech, Table 3. Additional monitoring data will be reported as they become available.

FURTHER REDUCTION OF IODINE RELEASES

When fuel is dissolved in the F-Area, no more than 50% of the radioactive iodine is evolved in the DOG stream.[8,25] The remainder is spread through the system with some released to the stack via the process vessel vent system.

Total iodine discharge could be reduced by diverting more iodine to the DOG, provided Ag°Z gives substantially improved iodine removal capability. Such techniques have been reported by Henrich, et al.,[27,28] and Johnson and Stone.[29] Henrich has demonstrated greater than 99% removal of radioactive iodine from simulated fuel solution by sparging with NO_2 gas towards the end and after fuel dissolution plus addition of KIO_3 towards the end of the desorption process to maintain the iodine concentration at levels high enough for efficient stripping of radioactive iodines by sparging.[28] Johnson and Stone were able to strip more than 90% of the ^{129}I from the dissolver solutions with air sparging only.[29]

Removal of radioactive iodine species at high concentrations from dissolver off-gas stream with fairly low volumetric flowrates in existing facilities is certainly more efficient and economical than removal of the same species from subsequent gas streams with much higher volumetric flowrates, using new equipment.

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concentration is 10^{-2} to 10^{-6} g/M^3. Corresponding residence time for F-Area configuration is 0.40 to 1.1 second. For MK31A fuel, the concentration of iodine feed gas is about 10^{-3} g/M^3.

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TABLE 1.

Comparison of Three Bed Configurations

		Ag°Z (TA 2-1102)	
	Existing	Original	Modified
Demister Used	None	Steel Wool	1-in 304 Pall Rings
Height from Grating, ft	0	2	7
Void Volume, %	-	Unknown	94
Pressure Drop, H_2O/ft	-	Unknown	0.06
Adsorbent and Size	1/2" $AgNO_3$-Coated Ceramic Berl Saddle	1/8" Ag°Z Extrudate	
Silver Content, wt %	3	19	19
Iodine Loading, lb/lb adsorbent	0.007	0.10	0.10
Bed Depth, in	98.5	10	10
Bed Level above Grating, ft	0	2	7
Total Pressure Drop, in H_2O	< 5	5-10*	< 5**

*Reference 3 (DPST-82-348).
**Calculated using References 23 and 24.

TABLE 2.

Recent 1985 Iodine Data

F-Area

PERIOD	DF*		% EMISSION FROM DOG		# OF CHARGES DISSOLVED	WEIGHTED AVERAGE COOLING TIME, DAYS
	^{131}I	^{129}I	^{131}I	^{129}I		
SEPTEMBER 3 - 10	38	4	55	73	2	262
SEPTEMBER 10 - 17	75	2	52	97	2	222
SEPTEMBER 17 - 24	3	3	85	77	1	229
SEPTEMBER 3 - 24	15	3.0	77	86	5	239

*DF = Input/Output; Input is assumed to be 1/2 of the calculated content of the iodine isotope, per DPST-66-582, p. 2 and DPST-74-533.

TABLE 3.

Annual Overall Process ^{129}I DF Values*

F-Area

Year	% 240-Pu	MTU to 1st Cycle	F- & H-Area I-129 Released mCi	F-Area I-129 Released mCi	F-Area I-129 Charged mCi	I-129 DF
1985	3%	1012				
1984	3%	1298	35.4	25	208.81	8.35
1983	3%	985	41.0	29	158.46	5.46
1982	3%	1127	59.0	44	181.30	4.12
1981	6%	1550	166.0	111	675.24	6.08
1980	6%	1300	160.0	112	566.33	5.06
1979	6%	840	130.0	91	365.94	4.02
1978	6%	915	130.0	91	398.61	4.38
1977	6%	667	140.0	98	290.57	2.97
1976	6%	1284	150.0	105	559.36	5.33
1975	6%	505	140.0	98	220.00	2.24
1974	6%	1353	170.0	119	589.42	4.95
1973	6%	1234	212.0	148.4	537.58	3.62
				Average I-129 DF Across Process		4.7

Annual I-131 DF Across Process was approximately 5.0 in 1983.

*Presented by R. L. Eubanks, Separations Technology, on November 5, 1985.

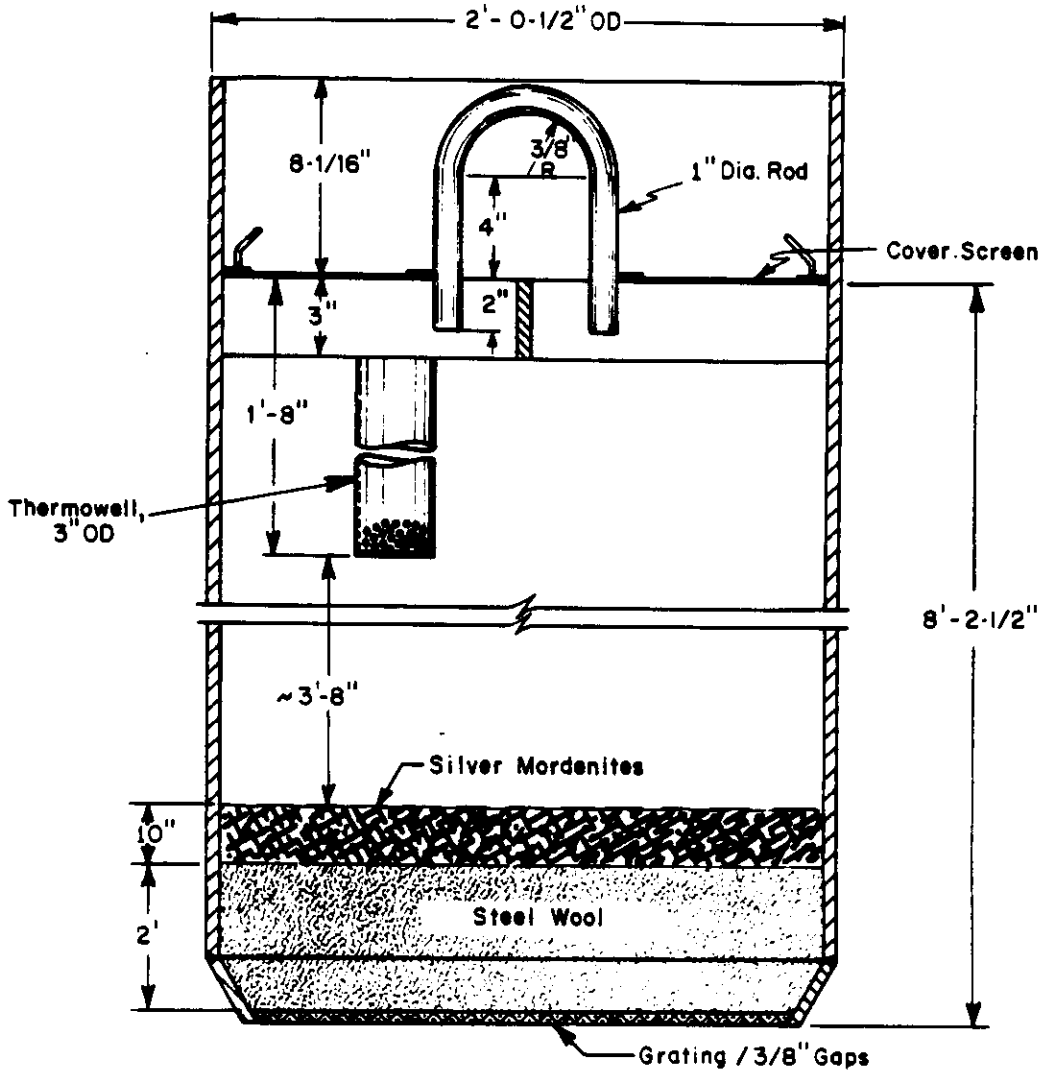

FIGURE 1. Iodine Cartridge for Existing and Original TA 2-1102 Configurations

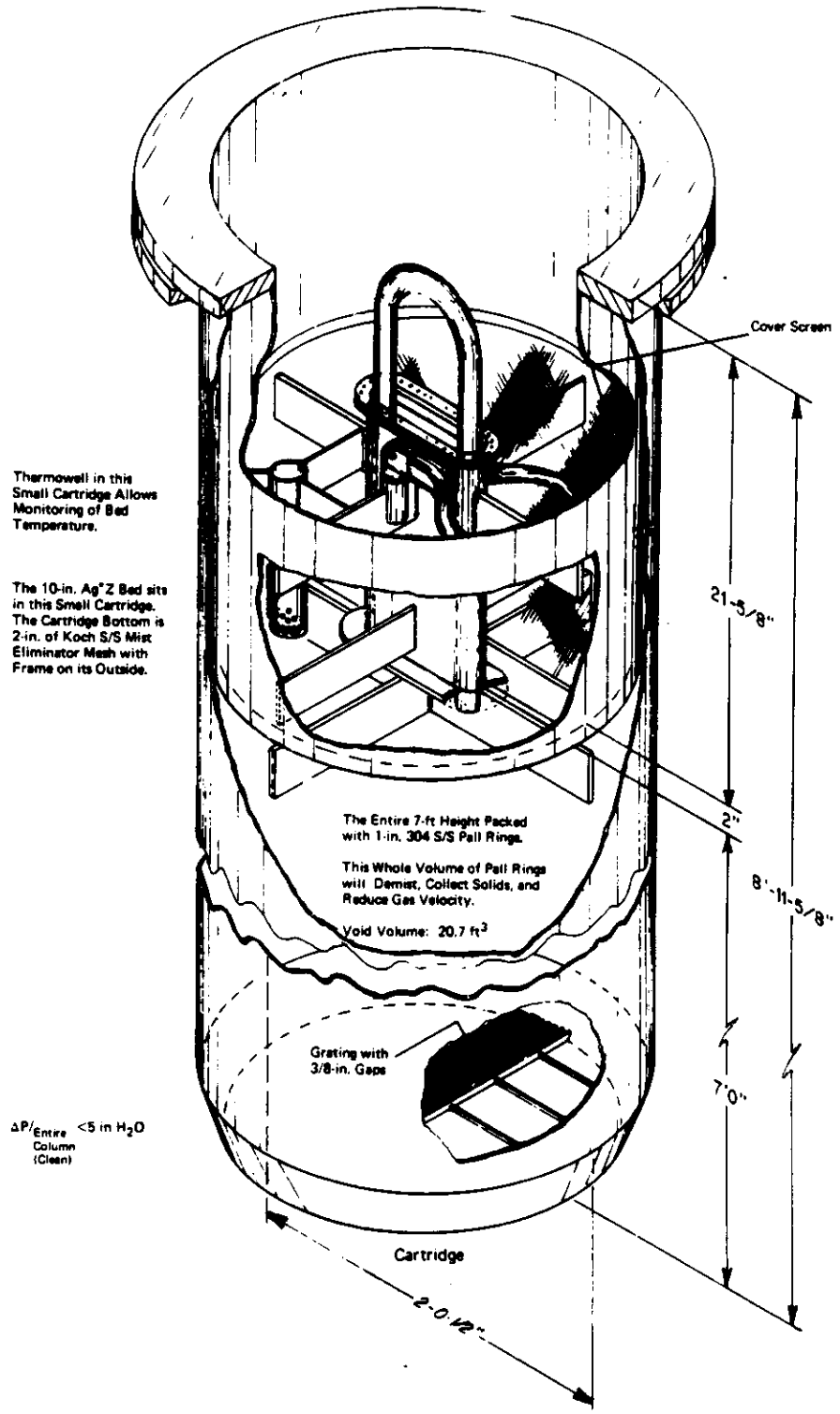

FIGURE 2. A New Design for Silver Mordenite (Ag°Z) Bed

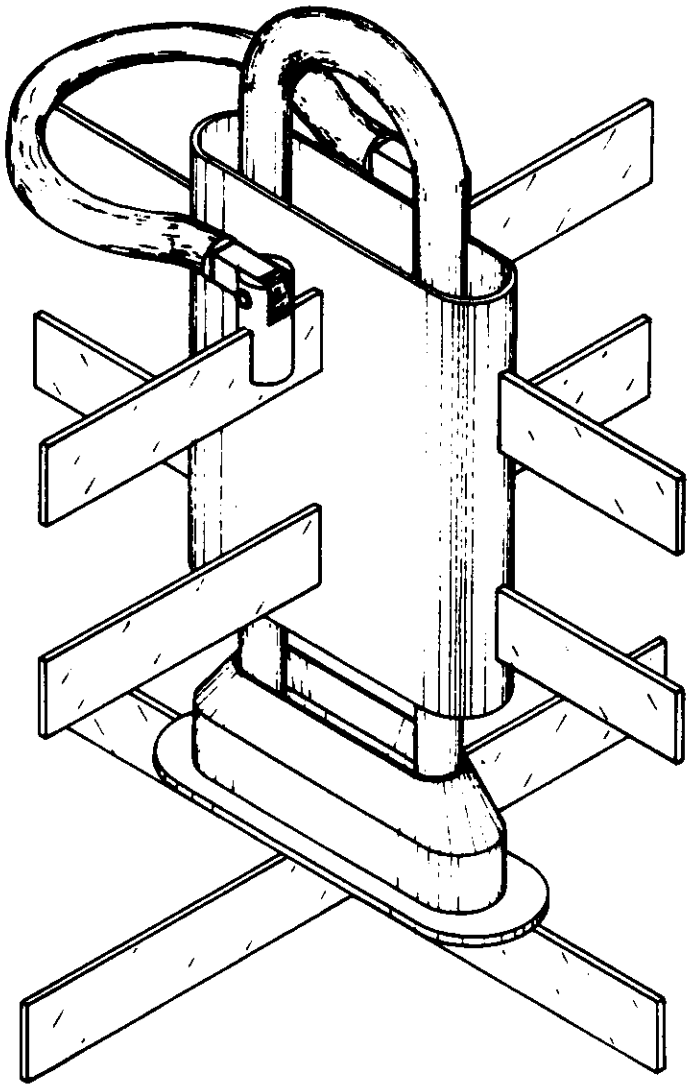

FIGURE 3. Sealing Mechanism for Mordenite Cartridge

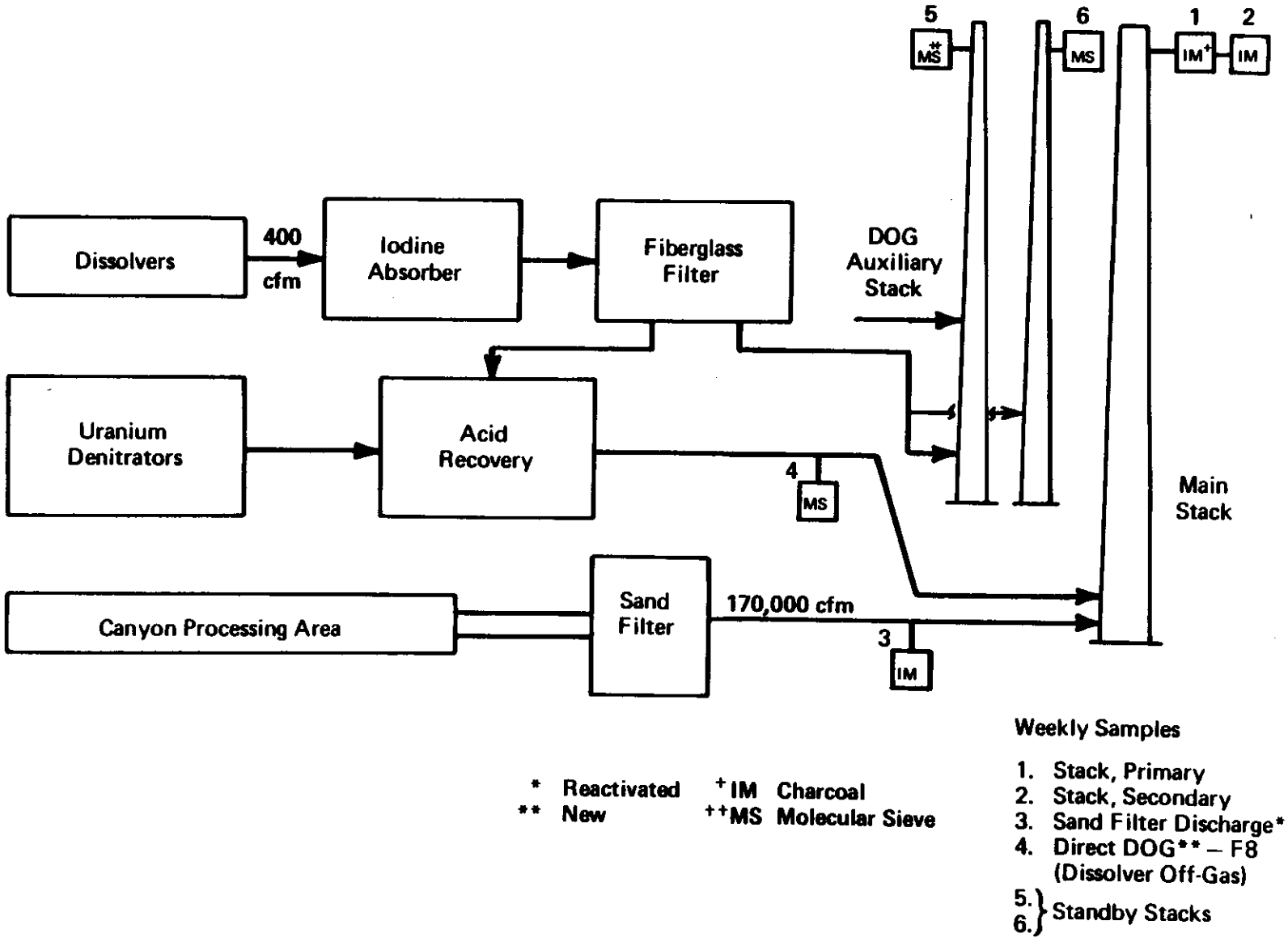

FIGURE 4. Iodine Sampling in F-Area