

ENERGY LABORATORY

MASSACHUSETTS INSTITUTE
OF TECHNOLOGY

URANIUM FROM SEAWATER RESEARCH:
FINAL PROGRESS REPORT, FY 1982

by

J. Borzekowski, M. J. Driscoll, F. R. Best

Energy Laboratory Report No. MIT-EL-82-037

Nuclear Engineering Department

Report No. MITNE-254

September 1982



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Funded by:

U.S. Department of Energy
Grand Junction Office
Bendix Field Engineering Corporation

Administered by:

MIT Energy Laboratory

Energy Laboratory Report No. MIT-EL-82-037
Nuclear Engineering Department Report No. MITNE-254
Sub-Contract No. 80-499E

September 1982

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ABSTRACT

During the FY '82 campaign 14 new ion exchange resin formulations, prepared by the Rohm & Haas Company, were tested by MIT at the Woods Hole Oceanographic Institution. The best of these chelating resins was again of the acrylic amidoxime type; it picked up approximately 100 ppm uranium in seven days' exposure to seawater, which represents a factor of better than two improvement over the seven-day results for the best FY '81 candidate (which saturated at roughly 100 ppm U after 30 days' exposure). Saturation was not reached and, within experimental accuracy, uranium accumulated at a constant rate over the seven-day period; it is speculated that a useful capacity of over 300 ppm U would be achieved.

All resins of the styrenic amidoxime type were found to be an order of magnitude lower in their effective capacity for uranium in seawater than the best of the acrylic forms. Particle size effects, which were found to be less than expected from theoretical computations of both fluid and solid side mass transfer resistance, can not account for this difference.

Scanning electron microscope examination by R & H scientists of ion exchange resin beads from beds subjected to seawater flow for 30 days in MIT's WHOI columns showed that the internal pores of the macro-reticular-type resins become filled with debris (of undetermined nature and effect) during exposure.

ACKNOWLEDGEMENTS

This work was performed as part of the "Uranium-from-Seawater Project" carried out by the MIT Energy Laboratory/Nuclear Engineering Department under contract to the US Department of Energy, Grand Junction Office, Bendix Field Engineering Corporation. The leadership provided by Mr. Paul Devergie (DOE, Grand Junction) and Mr. Robert Osterstock (Bendix) is greatly appreciated.

The principal authors gratefully acknowledge the many contributions of a number of MIT undergraduate students: Robert Meya, Robert Rozier, Christina Bellinger and Kaikobad Irani. The assistance of the MIT Undergraduate Research Opportunities Program, which provided both student support and waiver of overhead authorization, is greatly appreciated, as is the student support provided by the MIT Seagrant Office.

Thanks are due the staff of the MIT Nuclear Reactor Laboratory - in particular: William Fecych (Reactor Utilization), John Bernard and Kwan Kwok (Reactor Operations), and Edward Karaian (Reactor Radiation Protection Office). Our thanks also to Bill Ting and Barry Wessels (MITR Staff), as well as Nancy Marcus, Diana Franks, John Waterbury and Frederica Valois of the Woods Hole Oceanographic Institution for their assistance and generous loan of laboratory facilities and equipment.

Finally, and most noteworthy, are the efforts of the research scientists at the Rohm and Haas Company, especially Drs. Bernice Chong and Peter Osei-Gyimah.

Typing was capably handled by Ms. Delphine Radcliffe.

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CHAPTER 1

INTRODUCTION

1.1 Foreword

Work has been carried out at MIT for several years now on technology for the extraction of uranium from seawater [1-7]. During FYs 1981 and 1982 a DOE-supported project has focused on the evaluation of advanced ion exchange resins for this application. Results for FY 1981 have been reported in Ref. 7; the accomplishments of the FY 1982 campaign are summarized in this report.

In preparing this summary we have relied heavily on the documented record set down in our previously published material, Reference 7 in particular. In the current write-up, attention is restricted to methods and results which are new and/or different, and not previously reported. In addition, this is a record of the effort on advanced sorber assessment under the subject DOE contract. A parallel effort on recovery system design has been carried out outside of the DOE contract, and its progress is being separately documented (e.g. Ref. 8).

1.2 Background

Since the late 1970's there has been increased activity worldwide to assess the practicality of "mining the sea" for uranium. The motivation is clear: the world's oceans contain over 4,000 million tons of U_3O_8 - enough to fuel thousands of light water reactors for thousands of years. The difficulty is also apparent when one considers that the concentration of uranium in seawater is only 3.3 parts per billion. Reference 2 summarizes the proceedings of a topical meeting on this subject held at MIT in December 1980, in which the status of the international effort is recorded.

It is clear that the prospects for ultimate commercial exploitation of the sea's uranium resource depend on the development of a highly efficient and selective sorber. Prior to 1980 the material of almost universal choice was hydrous titanium oxide - and it remains a prime candidate for this service, being the subject of a number of ongoing research efforts in several countries today. Recently, however, ion exchange resins of the chelating type have been suggested for serious consideration. In all of this sorber research the goal is to develop a material which can be loaded to an effective capacity of several thousand ppm in seawater - that is, roughly to the same uranium content as terrestrial ores of current commercial interest.

In the summer of 1980 the U.S. Department of Energy, through its Grand Junction office, Bendix Field Engineering Corporation, awarded a research contract to the MIT Energy Laboratory/Nuclear Engineering Department to evaluate the capabilities of advanced ion exchange resins for uranium-from-seawater applications. These resins were formulated by research scientists at the Rohm and Haas Company and forwarded to MIT for field tests in rigs installed at the Woods Hole Oceanographic Institution. The first round of tests, completed during FY 1981, and reported in Ref. 7, identified an acrylic amidoxime resin which loaded to over 100 ppm uranium in a 30-day exposure. This was sufficiently encouraging to warrant a proposal, subsequently approved by the DOE, for a second round of tests in the summer of 1982. It is these experiments which are the subject of the present report.

The purpose of the FY 1982 campaign, then, was to test roughly a dozen different variations on the best 1981 resin. Thus, most of the resins evaluated had an amidoxime functionality and an acrylic backbone,

but a styrenic backbone was also re-examined, since it was the runner-up in the 1981 tests. Important variations tested were key parameters such as the degree of cross-linking, which is well known to affect resin performance. Appendix A spells out the details of the chemistry involved. Table 1.1 summarizes the variations in question.

1.3 Organization of this Report

The key facilities used to carry out the subject research - the ion exchange columns installed at the Woods Hole Oceanographic Institution, and the delayed neutron counting system used in conjunction with the MIT Research Reactor - are described in Chapter 2, together with a summary of the uranium assay data measured on candidate ion exchange resins supplied by Rohm and Haas under subcontract to MIT. (The chemical nature of the IX resins tested is documented in Appendix A, as provided by Rohm and Haas.)

In Chapter 3 the key results are summarized and interpreted to provide the basis for drawing conclusions and making recommendations.

Table 1-1

New IX Resins Tested During FY 1982 Campaign

R&H Code Number	% DVB	Functionality	Capacity (meq/g)
POG-501	6	Acrylic Amidoxime	3.83
POG-503 ^a	8	Acrylic Amidoxime	4.49
POG-505 ^a	6	Acrylic Amidoxime	4.54
EB-1958C	3	Styrenic Amidoxime	2.67
EB-1961A	6	Styrenic Amidoxime	4.44
EB-1966B ^b	6	Acrylic Amidoxime	3.50
EB-1966D ^b	6	Acrylic Amidoxime	3.42
EB-1966E	8	Acrylic Amidoxime	3.05
EB-1968A	4	Acrylic Amidoxime	5.04
EB-1970A ^c	3	Acrylic Amidoxime	3.90
EB-1970B ^c	3	Acrylic Amidoxime	3.33
EB-1968C	3	Styrenic Amidoxime	2.93
EB-1972B	3.8	Styrenic Amidoxime	3.15
EB-1976C ^d	3	Acrylic Amidoxime	4.72

^aPOG-503 and POG-505 copolymers were prepared using different suspending agents.

^bTo give beads of different porosity, the copolymer of EB-1966D was phase extended with toluene while that of EB-1966B was prepared without any phase extender.

^cIn an attempt to increase the amidoxime content of the resin, EB-1970A was functionalized using a two molar excess of hydroxylamine hydrochloride. However, a large difference in capacity was not observed between EB-1970A and EB-1970B which was functionalized using an equimolar ratio of reagents.

^d+70 mesh size* (210 microns); other acrylic amidoximes with 3% DVB are -20 +40 mesh (420 - 840 microns).

*US series sieves.

1.4 References

1. F. R. Best and M. J. Driscoll, Prospects for Uranium Recovery from Seawater, MIT-EL-80-001 (January 1980).
2. F. R. Best and M. J. Driscoll, Proceedings of a Topical Meeting on the Recovery of Uranium from Seawater, MIT-EL-80-031 (includes two MIT papers) (December 1980).
3. F. R. Best and M. J. Driscoll, "The Prospects for Uranium Recovery from Seawater", Trans. Am. Nucl. Soc. 34 (June 1980).
4. F. R. Best and M. J. Driscoll, "Uranium from Seawater: An Update on Worldwide Progress", Trans. Am. Nucl. Soc. 38 (June 1981).
5. F. R. Best, M. J. Driscoll, and C. Nitta, "Recovery of Uranium from Seawater" in AIChE Conference 74th Annual Meeting, New Orleans, Louisiana (November 8-12, 1981).
6. M. J. Driscoll and F. R. Best, "Systems Studies on the Extraction of Uranium from Seawater", MIT-EL-81-038 (November 1981).
7. C. K. Nitta, F. R. Best and M. J. Driscoll, "Delayed Neutron Assay to Test Sorbers for Uranium-from Seawater Applications", MIT-EL-82-008 (February 1982); also submitted as SM Thesis, MIT Nucl. Eng. Dept. by principal author.
8. P. B. Romanik, "Process Design Using ASPEN of a Uranium From Seawater Recovery System", SB Thesis, MIT Chemical Engineering Department, May 1982.

CHAPTER 2

EXPERIMENTAL APPARATUS, PROCEDURES AND RESULTS

2.1 Introduction

This chapter reviews the highlights of the procedures used in the column loading and delayed neutron assay experiments, emphasizing those areas in which modifications were made to the techniques employed last year [1]. The procedural review is followed by a discussion of resin loading results, including a comparison of new tests (performed in 1982 on the 1981 resins) with the original 1981 results, together with the principal data on the performance of the 1982 batch of resins. The results of several auxiliary experiments are also included.

2.2 WHOI Columns

2.2.1 Design Objectives

Figures 2.1 and 2.2, from Ref. [1], show the key features of the test rigs operated by MIT at the Woods Hole Oceanographic Institution. Their sole function is to expose a thin bed of sorber particles to a stream of seawater of measured volume. The design philosophy adopted here was to use a high flow loading (averaging $\sim 140 \text{ gpm/ft}^2$) to reduce fluid-side resistance to mass transfer by the maximum practicable amount, and to insure that all particles in the bed are exposed to essentially undepleted seawater. Table 2.1 shows that a high performance sorber should be capable of accumulating on the order of 1000 ppm uranium in a week-long test run.

2.2.2 Preparation for Loading

In order to preserve their adsorption properties, several of the 1981 sorbers had to be kept wet prior to loading. The variation in

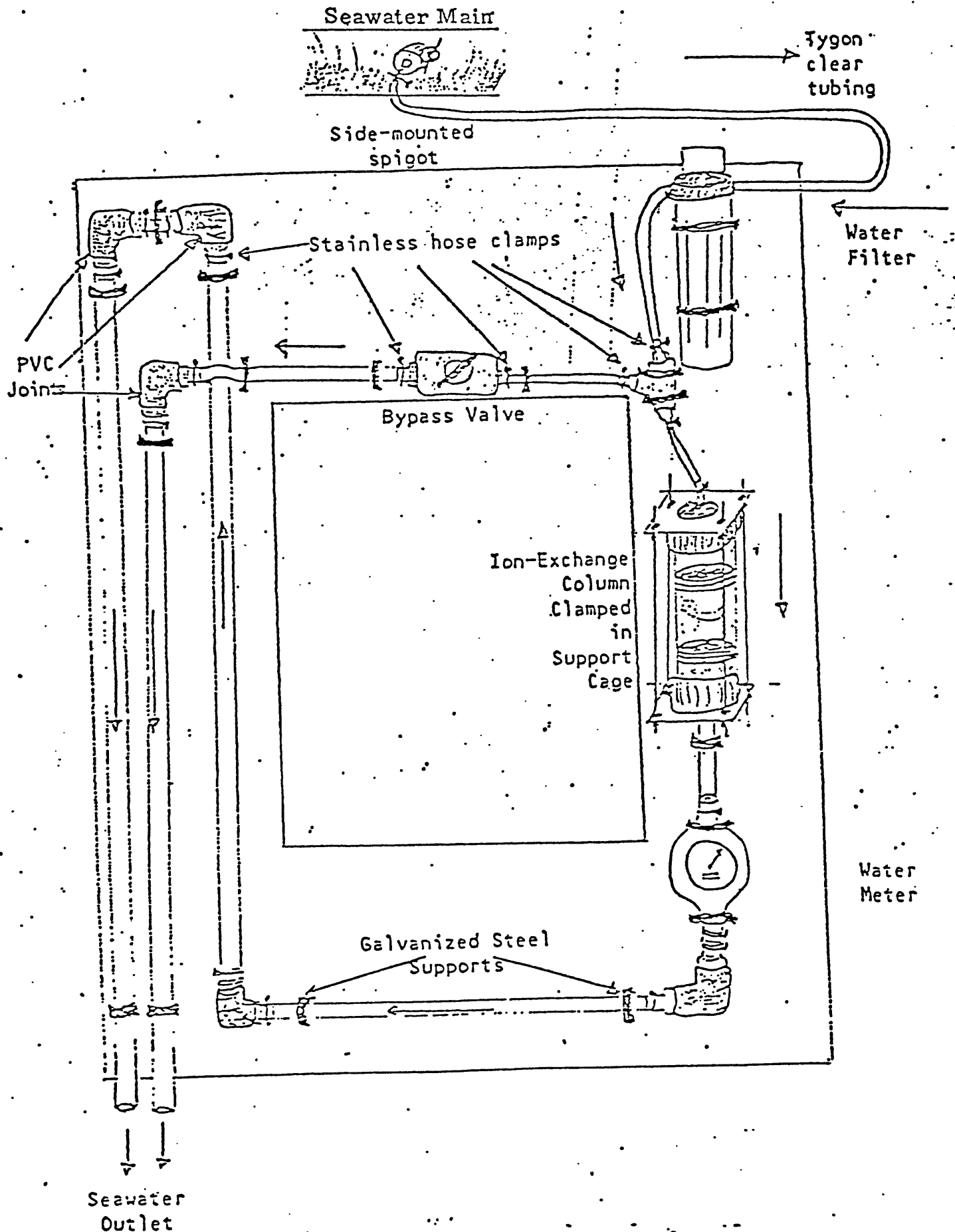


Fig. 2.1 Schematic of an Ion-Exchange System Installed at the Woods Hole Oceanographic Institution.

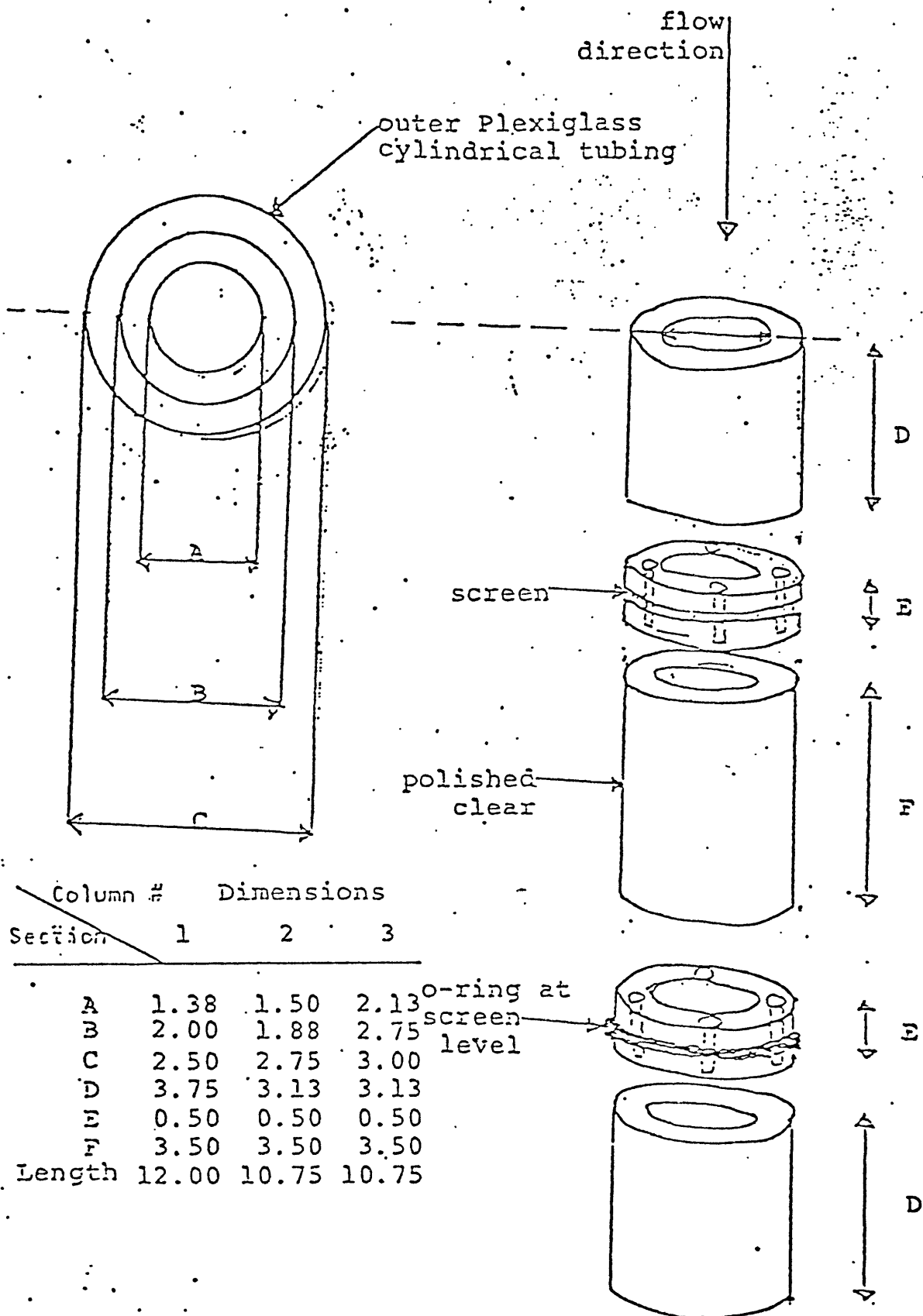


Fig. 2.2 Schematic of Plexiglas Ion-Exchange Column Section (all dimensions in inches).

Table 2.1

Approximate Uranium Pickup Rates:
Sorber Particle Loading in One Week

<u>Basis</u>	<u>Pickup Rate (ppm/week)</u>
100% removal of U transported to MIT's WHOI rigs at 140 gpm/ft ²	~ 700,000
Fluid side resistance only at 140 gpm/ft ² (see (a) below)	~ 10,000
Fluid side resistance only at 1 gpm/ft ² (see (b) below)	~ 2,000
Hydrous titanium oxide at 140 gpm/ft ² (see (c) below)	~ 100
Hydrous titanium oxide at 1 gpm/ft ² (see (c) below)	~ 40

- (a) A flow loading of 140 gpm/ft² would be practically usable only in an expanded (i. e. wool-type) sorber bed.
- (b) A flow loading of 1 gpm/ft² is representative of fluidized bed conditions.
- (c) As per experimental data, MIT and elsewhere.

hydration weight due to a variety of factors prevents the accurate weighing of these samples. This effect is not very important because, in fact, the pre-loaded sample weight provides only information regarding the overall sample recovery efficiency. While this parameter is of general interest (e.g. in identifying major sources of sample loss), it does not have to be determined to high precision. This is in contrast to the final recovered weight, which is determined to 6 decimal places using a Mettler microbalance.

The aforementioned sample loss was due primarily to passage of the smaller sorber particles through the retention screens - both under normal flow conditions and during backwash. The column retention screens used in this series of runs were made of stainless steel wire having a 0.008 inch (~200 microns) diameter and an opening (hole size) of ~300 microns. The use of too fine a mesh results in a greater pressure drop, increased clogging, and hence reduced flow. Our solution was to prescreen the sorbers to remove fines using a 60 x 60 mesh with a wire diameter of 0.0075 inch and an opening of 0.0092 inch (hole size of ~230 microns). This minimized subsequent sorber losses, and helped to achieve and maintain satisfactory flow characteristics.

The system used in 1981 to pre-filter particulates from the seawater employed a Facet Model P2 filter housing in conjunction with two Facet Model CF 10MCE 'wound' type filter cartridges. There is some play, and resulting leakage, between the two 10-inch long cartridges, which are stacked vertically in the housing. While so-called multiple length cartridges are available, they would have had to be specially made, necessitating a protracted delay. As an interim measure, plastic

couplers (Model SP-1000) were used to link Model CF97MCE cartridges (which are 1/4 inch shorter than the CF10MCE cartridges). This arrangement provided a more reliable cartridge alignment and reduced the accumulation of larger particulates on the upper screens of the sorber columns.

Another site of system clogging was at the feedwater spigots (all mounted horizontally on the main line as opposed to vertically downwards) which, when plugged, could completely cut off flow to the columns. These were cleared with pipe cleaners and thoroughly flushed at every filter change (on the third and seventh days of each loading).

2.2.3 Column Dismantling and Loading

Dismantling the Plexiglas columns, for both loading and removing samples, was facilitated by the use of wooden plungers cut to fit the columns' inside diameters, and the application of a small amount of vacuum grease to the lower screens' o-ring. The grease, which is water-insoluble, provided sufficient lubrication to ease moving the lower retention screen within the column, while the plunger helped deliver a more uniform and steady force.

Time was saved by initially loading a column with sufficient sorber material for several samples. In this way, 1, 3, and 7 day loadings of a given resin type could be effected concurrently, thereby minimizing the total time required for each test series. For 'partial' sample removal (i. e., 1 or 3 day loadings), the lower screen was pushed to within half an inch of the column top, after removing the top collar, upper screen and central collar; while for 'complete' sample removal (i. e. after 7 or 30 days) the lower screen was removed through the bottom of the column.

After each partial sample removal, the upper screen was cleaned

by applying a fine jet of seawater to its bottom surface in order to flush particulates from the top surface and rinse trapped resin beads from the lower surface. During complete sample removals this procedure was applied to both retention screens, in addition to flushing all the column components. The lower screen was then reloaded with a new sample and the entire column reassembled.

2.2.4 Preparation for Irradiation

Recovered samples were brought back to M.I.T., where they were cleaned of fine particulates by immersion in water, agitating the sample to free most low density contaminants, and draining off the natant liquid and suspension. After several iterations the sample was allowed to dry in a large cardboard cabinet - where its hydration weight could equilibrate with the atmosphere in the laboratory.

The combination of oven drying and desiccator storage formerly used was abandoned to avoid errors in the final sample weighing caused by absorption of ambient moisture during the measurement. While the new air drying technique is susceptible to an inherent inaccuracy in sample weight (i.e. variation in the residual moisture content), this effect is not appreciable. All results in the present report were determined in this manner, including the FY 81 reruns; thus all are on a consistent basis. Air-dried samples typically weigh ~ 8% more than oven-dried samples.

2.3 Delayed Neutron Counting System (DNC)

2.3.1 Introduction/Background

A detector assembly obtained from the Lawrence Livermore National Laboratory, through the Grand Junction office of D.O.E., comprises the heart of the DNC system. It is comprised of 20 Reuter-Stokes He³ proportional counters, and was outfitted with a variable high

voltage power supply and a counter/scaler unit, mounted on a portable stand to allow transport between storage, diagnostic and irradiation sites.

The required thermal neutron flux was provided by the 1PH1 irradiation facility of the MITR-II research reactor - a 5 MW thermal, H₂O-moderated unit. The 1PH1 facility provides a pneumatic tube system (Fig. 2.3) which allows the remote delivery of samples to a graphite reflector region whose flux is nominally 8×10^{12} neutrons/cm²-sec.

2.3.2 System Maintenance

Prior to the start of this year's experiments, the DNC unit was recalibrated using a Cr²⁵² source ($\sim 5 \mu\text{Ci}$), and a new operating voltage of 1360 V was chosen to provide maximum counting stability with respect to small voltage variations. (According to a performance specialist at Reuter-Stokes, although the He³ tubes would not sustain any damage at voltages less than 4000 V, regular usage above 1700 V would rapidly degrade the characteristics.) A variety of tests performed to determine an optimum discriminator setting - to filter out gamma interference - indicated that the -0.3 V setting (on the IC pin 11) used in 1981 was still appropriate.

In order to extend tube life it was determined that the apparatus should be kept off during storage between runs. Several stability tests indicated that a minimum warm-up time of 4 hours was sufficient before an irradiation, but that an overnight warm-up was preferable.

2.3.3 Irradiation Preparations and Procedures

The M.I.T. reactor schedule typically involves a Monday startup, so that by midweek power levels, shim settings, and regulating rod

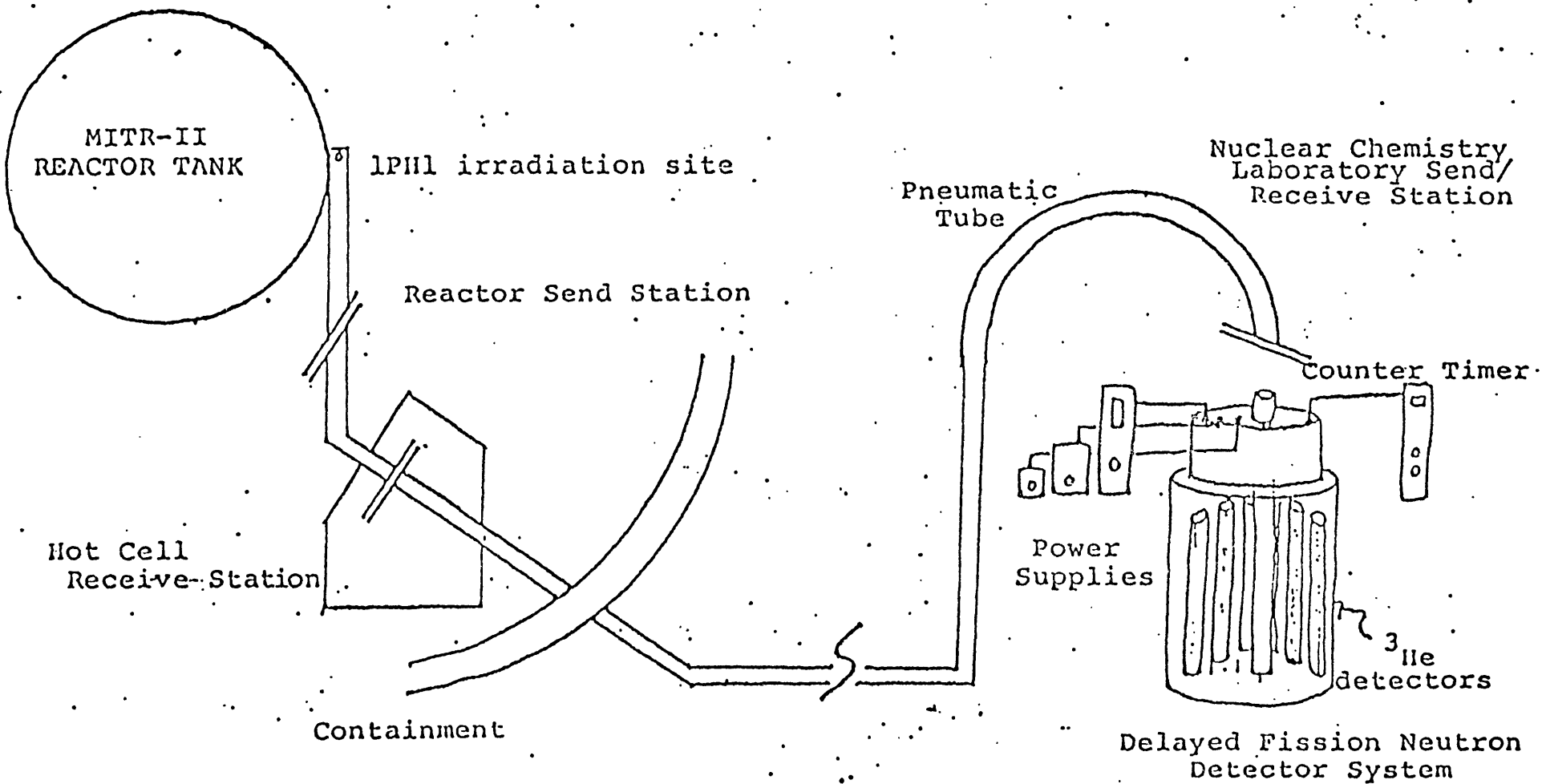


Fig. 2.3 Schematic of Irradiation and Counting Facilities.

positions are relatively stable. Although the variation in flux at the 1PH1 port due to these sources of fluctuation is supposedly minor, irradiations were always scheduled for late-week afternoons, and care was taken to avoid the re-shimming of control rods during a run.

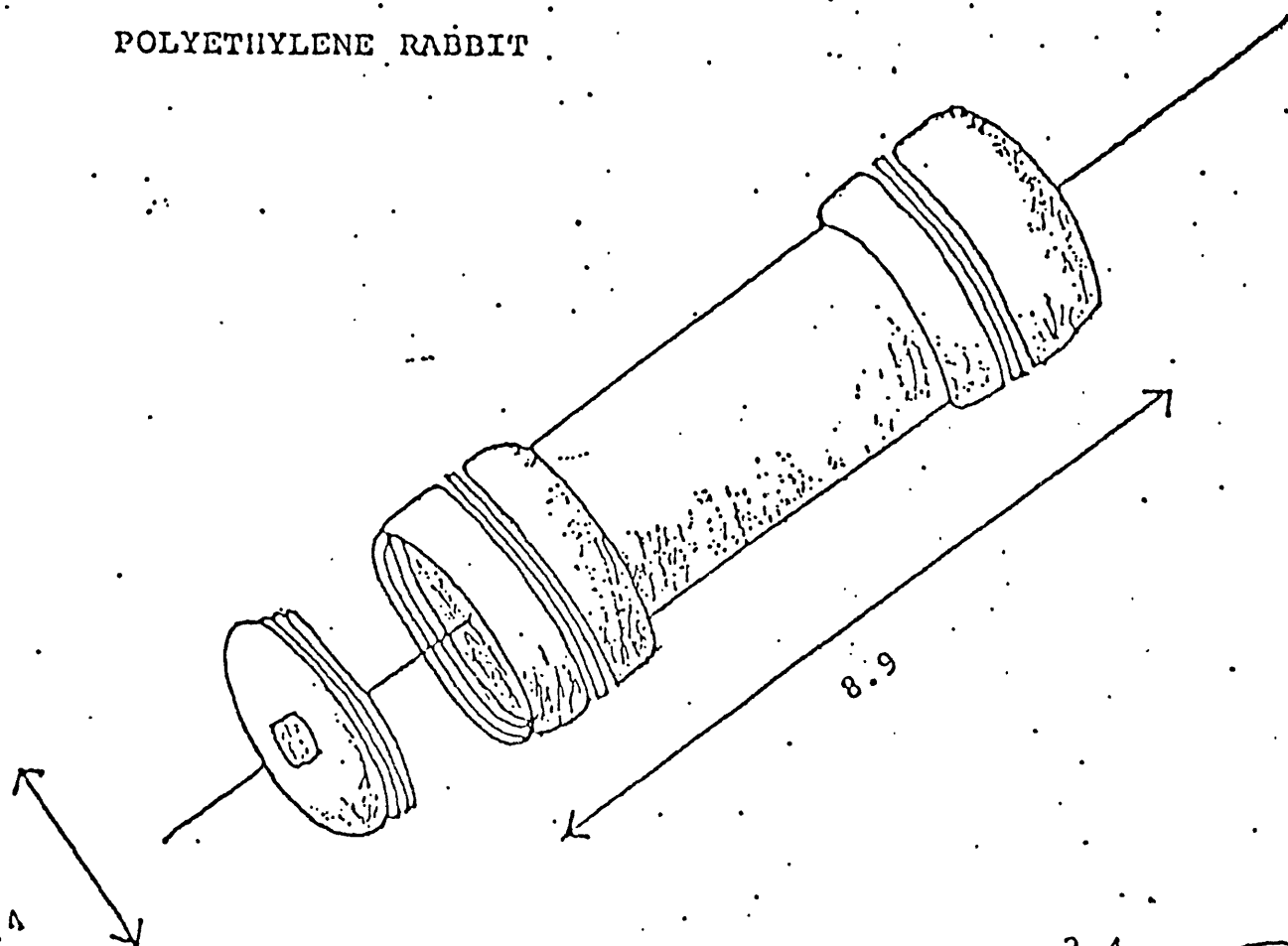
Prior to actually beginning an irradiation the DNC was checked for stability, using the Cf^{252} source, and the laboratory background was determined by counting the empty DNC chamber, 'air' blanks (consisting of a poly-bagged polyethylene vial in a rabbit) or empty rabbits (Fig. 2.4). This simply provides a check of base background levels due to other sources and contaminants present in the laboratory. It is extremely important to note that the DNC is highly sensitive to an increased neutron background, so that any extraneous sources should be eliminated. This is particularly true of the Cf^{252} calibration source, which should be removed from the room prior to making any measurements. Similarly, strong gamma sources should be kept away from the detector array.

Any given irradiation consisted, in order, of a one-minute exposure, a one-minute decay period, and a one-minute counting period using the DNC unit. During a run the DNC detector assembly should be placed as far away as possible from the hood used for storing irradiated rabbits, as these can induce a significant increase in background radiation.

To monitor this room background effect, the empty DNC chamber was counted periodically during the run; variations in reactor induced background were monitored by rerunning an "air blank" (or empty, but irradiated, rabbit) approximately every ten samples.

Geometrical considerations also figure prominently in DNC counting measurements, so that care was taken to ensure a consistent

POLYETHYLENE RABBIT



POLYETHYLENE VIALS

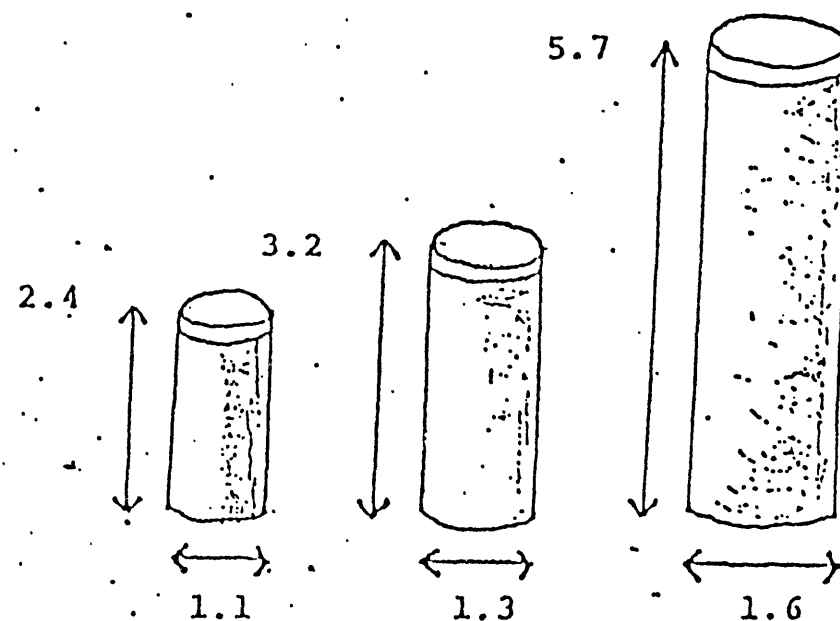


Fig. 2.4 Rabbit and Vial Dimensions (cm).

handling of all samples. Each vial (first heat-sealed, then poly-bagged) was placed upright in the rabbit, and secured with a styrofoam plug. Because the end of the rabbit which enters the pneumatic tube first sees the highest flux (variations of $\sim 40\%$ along a rabbit's length have been recorded), every rabbit was loaded into the tube upside down.* Before counting, however, the samples were placed right side up in the DNC chamber.

In order to correlate results between test dates, an NBS standard of known uranium content was irradiated at the beginning and end of each run. This standard then provided a correction factor (relative), accounting for any variations in flux and/or counting efficiency. All data were subsequently normalized to the NBS value attained during the reference run made on the 8th of July, 1982. In order to compare last year's data on a consistent basis, they too were normalized in this manner, as well as being corrected for the depleted uranium content of the NBS standard (it had previously been assumed to contain natural uranium). For the record, the NBS standard now in use by the project is an SRM-941 calibrated glass standard with a uranium concentration of 461.5 ± 1.1 ppm by weight and a U-235 isotopic abundance of 0.2376 percent [2].

Gold foils were used to provide a supplementary flux monitor. Foil activity was measured using a digital dose calibrator to indicate the decay rate of the Au-198 activity. The initial activity and hence the incident flux could then be calculated based on the known decay time. While some problems were encountered with reproducibility, in general the results indicated a 1PH1 flux of $\sim 7.2 \times 10^{12}$ n/cm²-sec, which is in close agreement with similar measurements made by the reactor

* The intake is vertically oriented, hence the vial-end of the rabbit enters first.

operations staff. Although further refinement in technique is required, this approach may prove quite useful for future experiments.

2.4 Results

Tables 2.2 and 2.3 summarize the most significant data measured during the FY '82 campaign; in particular, Table 2.2 presents the screening test results for the new ion exchange resins prepared during the summer of 1982 by the Rohm & Haas Company. Table 2.3 reports "other" measurements: most were either repeat DNC assay runs on FY '81 samples, or complete test-rig loading/DNC assay repeat runs on resins from the FY '81 inventory. In addition, two natural biosorbers were tested for Prof. Tsezos of McMaster University; he has previously reported on the capabilities of these materials [3,4], and we will leave further data analysis on these substances to him. Likewise, no further discussion of the repeat runs in Table 2.3 will be registered here: the results support our previous conclusions as to the superiority of the SGM-251 formulation, and its effective capacity of ~ 100 ppm uranium. Table 2.3 does contain one interesting new piece of information, however - uranium content measurements on "sludge", the particulate matter which collects on the pre-filters upstream of the sorber beds under test. The 2 ppm or so of uranium is not unexpected, and is not significant with respect to the interpretation of our experiments: even if "sludge" was present in WHOI seawater at a concentration of 100 ppm (1000 times open ocean values), less than 6% of the seawater's uranium content would be tied up in this material; and if as much as 10% of our resin sample weights were really "sludge", only 0.2 ppm uranium would be falsely attributed to the sorber's account.

The results in Tables 2.2 and 2.3 are in general the average of two

Table 2.2

Uranium Uptake Data for New Sorbers Tested During FY 1982

Sorber	3-Day Loading		7-Day Loading	
	Uptake (ppm)	Exposure (gallons)	Uptake (ppm)	Exposure (gallons)
1. POG-503	51.81	8221	94.26	16,143
2. POG-505	43.62	8609	81.80	18,101
3. EB-1970A	48.75	6823	70.80	14,045
4. EB-1976C	43.69	5030	NA	11,255
5. EB-1970B	31.34	6119	NA	13,262
6. POG-501	28.19	8114	56.43	17,176
7. EB-1968A	21.21	6799	33.93	14,940
8. EB-1966B	13.94	8851	26.03	17,298
9. EB-1966E	10.45	3413	20.01	9,755
10. EB-1966D	3.09	7051	6.94	16,158
11. EB-1958C	3.75	6594	11.80	12,380
12. EB-1961A	5.39	9188	7.66	21,699
13. EB-1968C	5.53	7327	5.64	16,387
14. EB-1972B	0.*	7027	NA	14,808

*No uranium uptake within statistical uncertainty.

Table 2.3

Other Sorbers Tested During FY 1982

Sorber	Exposure Time	Uranium Uptake, ppm		
		I	II	III
1. SGM-251 acrylic amidoxime	7 days 30 days 30 days (fines) 30 days (larger beads)	57.4 129.5	85.3 123.0 70.6	26.5 80.2 73.6 41.6
2. SGM-245 styrenic amidoxime	7 days 30 days	0.87 12.0	16.3	3.3
3. XE-318 styrenic iminodiacetate	7 days 30 days	0.3 9.3		3.0
4. Hydrous Titanium Oxide	7 days 30 days	97.5 156.1	142.9	52.8
5. Sludge (material collected on filter cartridges)	7 days			2.0 → 3.5
6. Biosorbers				
(a) R. Arrhizus	1 day 3 days 7 days			5.74 0.24 1.93
(b) P. Chrysogenum	1 day 3 days 7 days			0.* 0.* 0.*

Key: I = results from FY 1981 (for comparison)
 II = loaded during FY 1981, re-assayed for U in FY 1982
 III = complete re-run in FY 1982: loading plus assay

*Within statistical fluctuations, there was no net uptake of uranium for the biosorber, for which unloaded samples registered a U content of ~4.5 ppm.

independent determinations each (two separate resin samples, separately subjected to DNC assay). All results are corrected using an appropriate "blank" (unloaded sorber, similarly encapsulated in a vial and rabbit). The data handling procedure was essentially the same as employed on the FY '81 data, as reported in Ref. 1.

While the results were more self-consistent and reproducible than in FY '82, reflecting our greater experience with all of the phases of resin loading and delayed-neutron assay, several anomalous runs (out of over 200) were again experienced, principally in the form of "blanks" which yielded improbably high, false uranium contents. These were easily spotted and rejected because of the large mismatch vs their supposedly identical twin run. This experience is similar to incidents in FY '81, and the cause has not yet been determined.

Note that the results in Tables 2.2 and 2.3 have been reported to more digits than warranted in terms of strict statistical significance. Although once uranium content exceeds around 10 ppm the counting statistic uncertainty is insignificant; other errors dominate - principally that in sample-to-sample exposure. Weighing errors per se are negligible, but several percent uncertainty is introduced because of inevitable variations in moisture and "sludge" content in the resin samples. Finally, there is an unknown, but assuredly larger variation in the environment at WHOI (i. e. all those factors which characterize the exposure). Our best judgement is that a one-sigma uncertainty of roughly $\pm 10\%$ should be assigned the typical results reported here.

2.5 References

1. C. K. Nitta, F. R. Best and M. J. Driscoll, "Delayed Neutron Assay to Test Sorbers for Uranium-from-Seawater Applications", MIT-EL-82-008, February 1982.
2. NBS Special Publication 260-49, Standard Reference Materials: Calibrated Glass Standards for Fission Track Use, June 1974.
3. M. Tsezos and B. Volesky, "Biosorption of Uranium and Thorium", Biotechnology and Bioengineering, Vol. XXIII, p. 583, John Wiley & Sons, Inc., 1981.
4. M. Tsezos and B. Volesky, "The Mechanism of Uranium Biosorption by *Rhizopus arrhizus*", Biotechnology and Bioengineering, Vol. XXIV, p. 385, John Wiley & Sons, Inc., 1982.

CHAPTER 3

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

3.1 Synopsis of FY '82 Data

Table 3.1 summarizes the most significant new data measured during the FY '82 campaign - the screening test results for the new ion exchange resins prepared during the summer of 1982 by the Rohm & Haas Company.

Figures 3.1 and 3.2 plot the results for the POG series of resins, which were best as a group. Smooth curves have been drawn through the origin (since all resins were measured to be uranium-free as received from R & H), and the 3 and 7 day data points. Note, however, that within experimental uncertainty (on the order of $\pm 10\%$), linear pickup over the entire range is a justifiable assertion - particularly when gallons exposure is used instead of time, as in Fig. 3.2.

Comparison of these figures with similar plots in Ref. 3, which extend through 30 days (and $\sim 50,000$ gallons) suggest that we may just be reaching the knee of the loading curve at the 7-day point. In any event, it is clear that the post-7-day pickup can not proceed faster than linearly; hence the projected 30-day loading must be ≤ 400 ppm, well below our goal of 1000^+ ppm in this time frame.

Table 3.1

Summary of FY 1982 Resin Performance Results

R&H Code Number	Functionality	PPM Loading*	
		3 days	7 days
POG-501	Acrylic Amidoxime	28.19	56.43
POG-503	Acrylic Amidoxime	51.81	94.26
POG-505	Acrylic Amidoxime	43.62	81.80
EB-1958C	Styrenic Amidoxime	3.75	11.80
EB-1961A	Styrenic Amidoxime	5.39	7.66
EB-1966B	Acrylic Amidoxime	3.94	26.03
EB-1966D	Acrylic Amidoxime	3.09	6.94
EB-1966E	Acrylic Amidoxime	10.45	20.01
EB-1968A	Acrylic Amidoxime	21.21	33.93
EB-1970A	Acrylic Amidoxime	48.75	70.80
EB-1970B	Acrylic Amidoxime	31.34	NA
EB-1968C	Styrenic Amidoxime	5.53	5.64
EB-1972B	Styrenic Amidoxime	0**	NA
EB-1976C	Acrylic Amidoxime	43.69	NA

*In test rigs at the Woods Hole Oceanographic Institution at flow loading of $\sim 140 \text{ gpm/ft}^2$.

**No uranium uptake within statistical uncertainty.

NA = not available

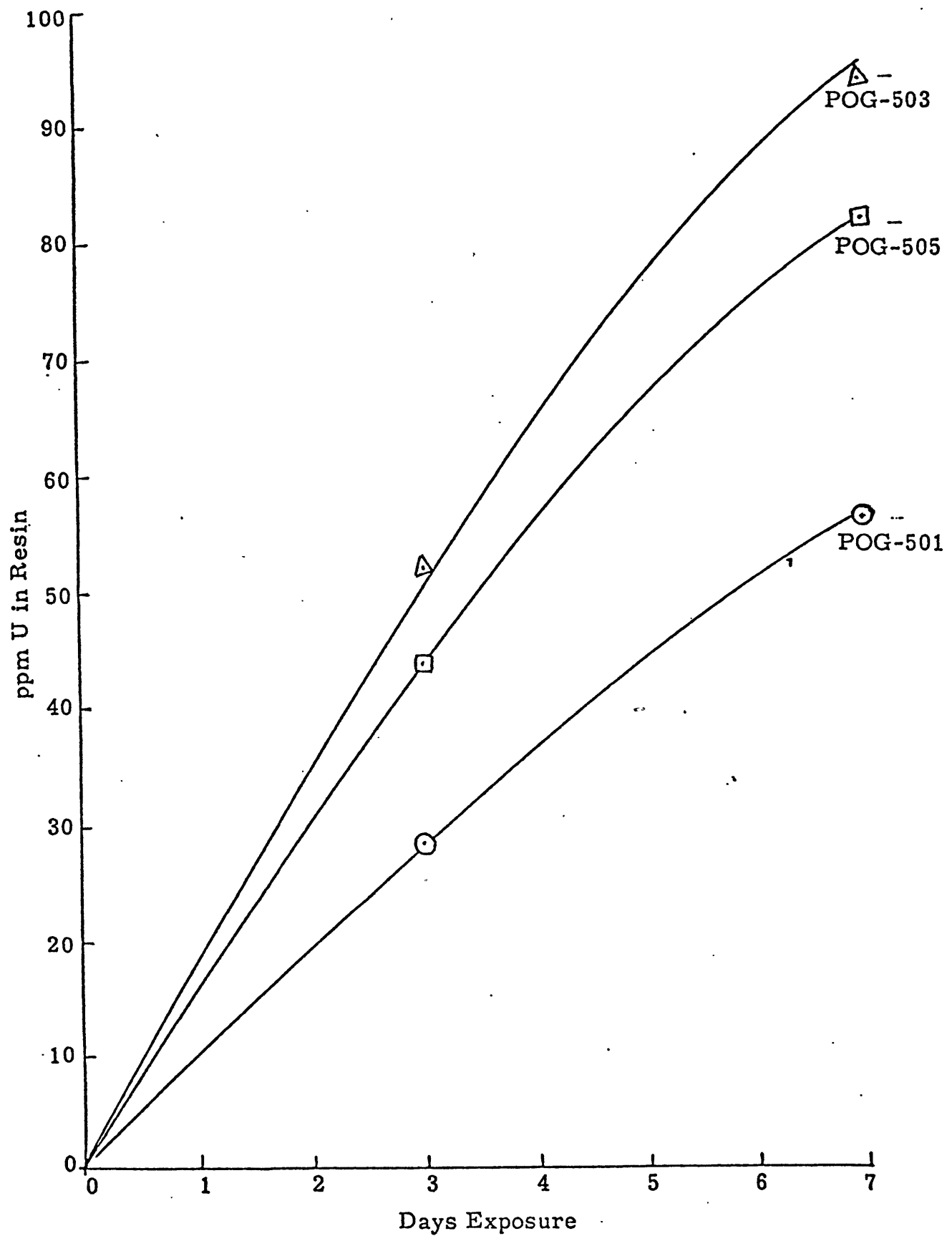


Fig. 3.1 Time dependent uranium pickup for best FY '82 IX resins.

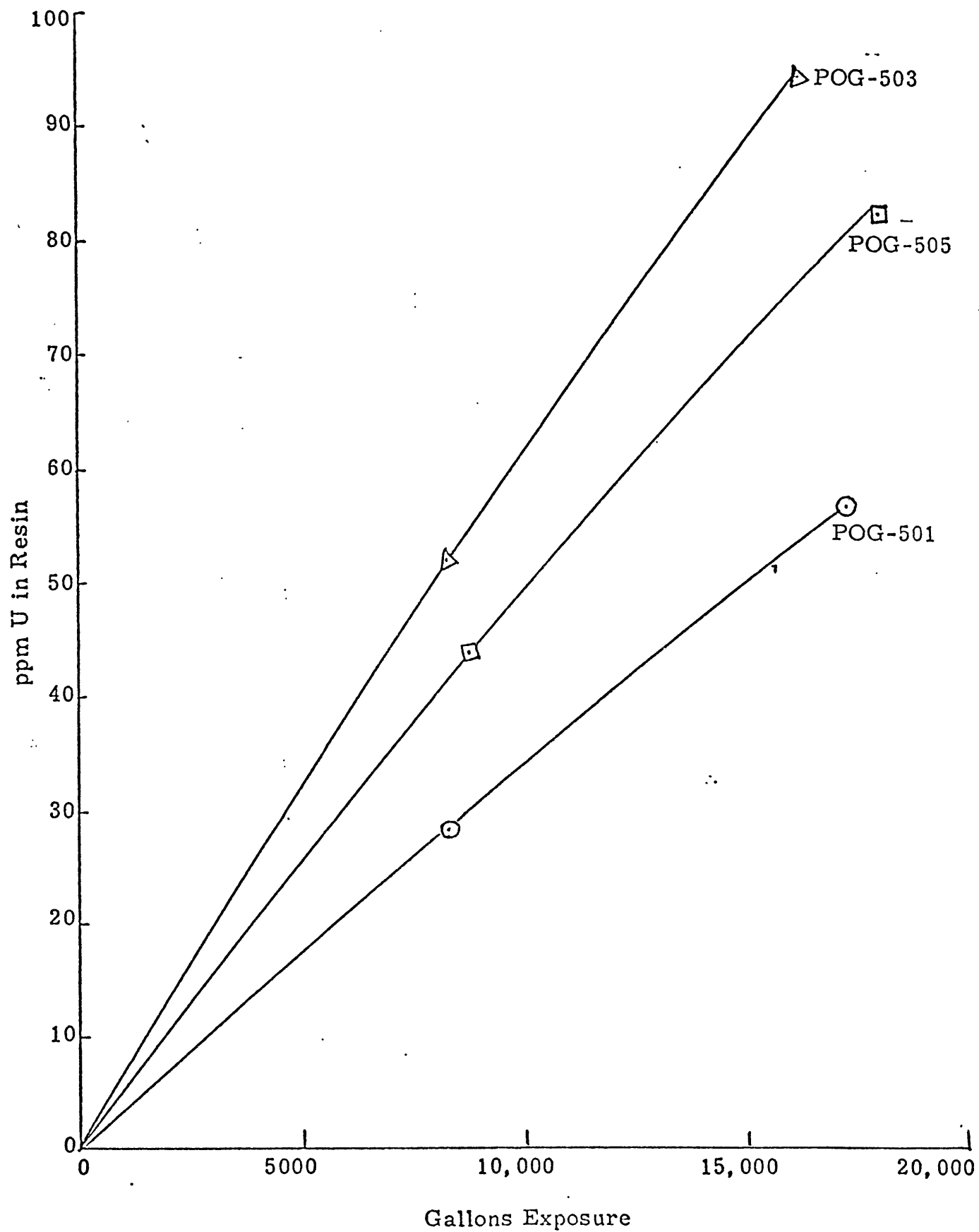


Fig. 3.2 Uranium pickup as a function of seawater throughput.

3.2 Interpretation and Discussion

Sorber performance in the subject screening tests is limited by several factors: transport of uranium to the bed and through the liquid film surrounding the individual particles; then by diffusion into the particle itself; and finally by its inherent ability to selectively sorb uranium in the presence of a (relatively) high concentration of other species. As noted in Chapter 2, the WHOI test rigs were designed to minimize the fluid-side limitations, hence we focus on the solid-side constraints here.

Weisz [1] has characterized the solid-side diffusion in a particularly useful way; he finds for the time for a sorber to reach half-saturation:

$$T_h = u^2 \frac{d^2 b}{4D} \frac{C}{px} \quad (3.1)$$

where u is a constant ranging, in the case of a sphere, between 0.175 and 0.136 in the limits of weak and strong sorption, respectively. Weisz shows that these limits include behavior described by the Nernst, Freundlich and Langmuir isotherms. The other parameters are:

- b = tortuosity factor, usually $\leq \sqrt{3}$
- p = porosity fraction in sorber particle (~ 0.5)
- D = diffusivity of uranyl tricarboxylate ions in seawater
($\sim 2 \times 10^{-6} \text{ cm}^2/\text{sec}$)
- d = particle diameter (~ 300 microns)
- C = concentration at equilibrium in sorber (say 300 ppm)
- x = concentration in solution (3.3 ppb)

For the above parameter values, and strong sorption, $T_h \approx 1.5$ weeks, which suggests that the uranium uptake is potentially restrained by intra-particle diffusion in a short screening test. In one week approximately $\frac{1}{3}$ of saturation would be attained according to this analysis; the loading of ~ 100 ppm is, in fact, what we see in practice.

The very strong (squared) dependence on particle diameter in Eq. (3.1) is noteworthy. The resins tested have a spectrum of particle sizes. To test for this effect we screened SGM-251 into fine and coarse particle fractions, and independently measured their uranium content (see Table 2.3). The fine and coarse fractions differed in particle diameter by (roughly) a factor of four, which would suggest a factor of ~ 16 difference in pickup rate, but the experimental data shows a factor of less than two difference. This is hard to explain, because the other limiting case (fluid-side resistance only) would yield a diameter-to-the-first-power dependence, and hence a predicted factor of four difference in pickup: again less than observed.

The strong dependence on particle porosity in Eq. (3.1) is to be noted. Since solid phase resistance is characteristically much higher than in the liquid phase, it is important that the sorber be configured in a porous structure. This is the principle behind "macroreticular" ion exchange resins (the type used in the present work), in which the resin particles are made up of smaller particles having representative mean particle (and pore) diameters of $\sim 10^{-2}$ micron (which may be compared to the diameter of the uranyl tricarbonate complex anion of $\sim 10^{-3}$ micron). It is the lack of such porosity which is usually cited as a major reason for the slow uptake of uranium by hydrous titanium oxide in gel-particulate form.

Thus a limited uptake in the present work can be attributed either to blockage of the resin's pores, or to its limited inherent affinity for uranium. Since tests were carried out under comparable conditions (and, in fact, three at a time in parallel rigs), the relative ranking among candidate sorbers is not affected by these considerations.

Pore blocking could occur in a number of ways: ingress of fine organic or inorganic debris which passes through the prefilter, precipitation of insoluble salts (e.g. carbonates - as is known to occur in HTO, for example), or by growth of biological organisms which find the pores hospitable, or even feed upon the resin. South African experience with ion exchange recovery of uranium suggests that solution turbidity alone will not harm resin performance, and blinding by silicates (not expected to be a problem in seawater) is more of a problem [2].

There is some evidence that resin pores are blocked in the present case. Rohm & Haas researchers have examined resin particles from MIT/WHOI runs using an electron microscope, and have noted the presence of interstitial matter of an unidentified nature. Whether the presence of this material significantly degraded resin performance can not be established at present. If one proceeds on the hypothesis that in-situ biological growth is responsible, then it may be effective to treat the resin with a biocide prior to use (and after each regeneration).

3.3 Conclusions and Commentary

It was a major objective of the FY '82 program to demonstrate an effective resin capacity of $\sim 1000^+$ ppm. From the results summarized in Table 3.1 it is evident that this goal has not yet been reached. However, uranium pickup rates roughly 2-3 times those achieved in FY '81 were attained, and the best of the FY '82 resins accumulated a

uranium inventory in 7 days equal to that which the best FY '81 resin' logged in 30 days.

The results also show the striking superiority of the acrylic backbone over the styrene formulations: none of the latter reached uranium levels of any practical interest. It appears clear that this line of development should be abandoned. Also (particularly since it appears that German and Japanese researchers are experimenting with still other backbones) this entire area may be a worthwhile focus for future U.S. efforts.

In interpreting these results it is important to note the following points:

- (a) To some extent the 7-day tests are more an indication of the kinetic behavior than the ultimate capacity of the better-performing resins, since they are not driven to saturation during this interval. A screening test of this duration is quite adequate, however, to demonstrate the limitations of the mediocre candidates in an unambiguous manner.
- (b) All things considered, while the pickup rate by the better-performing resins is probably limited by solid-side rather than fluid-side resistance, we did not see the strong (diameter squared) dependence that this should entail in the small number of experiments in which we examined pickup differences among screened resin batches. Comparison of the results for EB-1976C/1970A/1970B also suggests a smaller-than-expected particle size effect. This anomalous behavior could be quite important, since our systems studies, which are based upon theoretical mass transfer

calculations, have predicted a strong size dependence, which has driven our particle size optima into the 100 micron diameter range.

- (c) While uranium loadings of 2000-2400 ppm have been reported by German and Japanese workers for their ion exchange resin formulations, this has apparently been achieved using 50-200 day exposures (publicly available published data is very sketchy on such details). If our resins sustained their 7-day rates over this long an exposure, they would reach an equivalent uranium content. Test periods this long are prohibitive during the materials development phase (and, we might add, rates this slow are marginal for systems of ultimate commercial interest). Thus a different experimental approach is indicated - e.g. start with a uranium-loaded resin and find the equilibrium capacity via elution by seawater.
- (d) While it is true that the styrene-form resins generally had larger particle sizes than those with an acrylic backbone, it is not believed that this could explain their inferior performance in view of the other evidence we have cited on the modest effect of particle size within the family of acrylic resins. It should also be noted that there are particle-size effects associated with resin production and functionalization which may obscure the effects associated with resin loading which we have been discussing.

3.4 Recommendations for Future Work

While a continuing effort in search of even better sorbers is clearly in order, considerable progress can still be made by pursuing several lines of action based upon use of the best resin identified in the FY '82 campaign:

- (a) Since saturation is not indicated from the 3 and 7 day loading experiments (see Fig. 3.1), a 30-day exposure should be carried out, and, if need be, even longer runs made, until saturation is achieved.

A companion experiment is also suggested, in which the resin is loaded to ~50,000 ppm in the laboratory, and then subjected to a standard 7-day run in a test rig to determine the asymptotic capacity by approaching it from the opposite direction.
- (b) The loaded resin should be assayed for vanadium and molybdenum in addition to uranium, since there are indications that these valuable co-products might also be collected by chelating resins of the type we have employed. If removed in proportion to their concentration in seawater, their value could exceed that of uranium based on current market prices!
- (c) The feasibility of elution and re-use of the resin through several cycles should be demonstrated; the retention of ion exchange properties and resistance to biological attack after 6 months and one year of exposure to the marine environment should also be investigated. If degradation is evident, pre-treatment with a biocide should be tested.
- (d) Loading tests should be carried out at lower flow rates. In view of the slower-than-anticipated pickup exhibited by the resin

particles it may be possible to reduce the flow rate without further detriment. If so this would have an important effect on system design choices.

- (e) The particle size effect needs further investigation, down into the 100 micron range used in prior MIT systems studies. The FY '82 resins typically had mean particle diameters of ~ 300 microns, and uranium pickup rates ~ 15 ppm/day. Even if the theoretical diameter to the 1.0 or 2.0 power relations hold, this does not support our previously computed ~ 1500 ppm/day loading rate at 100 microns - especially since most literature concurs that packed particle beds have higher mass transfer coefficients than fluidized beds or expanded beds (e.g. the "steel-wool" configuration we have previously recommended) of the same particles. Lower pickup rates will significantly alter the design tradeoffs in the optimization process.

Finally, since we have already carried out our screening tests under conditions more severe than those anticipated in projected full-scale plant operations, it may now be possible to accelerate the test program and realize significant economies by returning to the laboratory for many of these third-round tests.

3.5 References

1. P. B. Weisz, "Sorption-Diffusion in Heterogeneous Systems, Parts 1-3", Trans. Faraday Society, Vol. 63, pp. 1801-1823 (1967).
2. A. K. Haines, "The Development of Continuous Fluidized-Bed Ion-Exchange in South Africa and Its Use in the Recovery of Uranium", J. S. Afr. Inst. Min. Metall., Vol. 78, p. 303 (1978).
3. C. K. Nitta, F. R. Best and M. J. Driscoll, "Delayed Neutron Assay to Test Sorbers for Uranium-from-Seawater Applications", MIT-EL-82-008, February 1982.

APPENDIX A

ROHM & HAAS CONTRIBUTION

The appendix which follows is a reproduction of the report submitted by Rohm & Haas Company researchers on their FY '82 subcontract efforts (with minor typographic corrections as per telephone conversation M. J. Driscoll/P. Osei-Gyimah on 9/28/82).

Important points covered are:

- The chemical structure (functionalization and polymer backbone) of the resins prepared for this year's screening tests.
- The physical and chemical characterization of the subject resins (e.g. particle size, cross-linking, porosity, surface area, pore diameter, capacity).
- The results of special tests, such as electron microscope scans of resin beads exposed to seawater service in the MIT/WHOI test rigs.

To aid in the interpretation of resin particle size data, the following equivalence should be noted:

<u>U.S. Series Mesh No.</u>	<u>Opening, . microns</u> (i. e. ~ particle dia.)
18	1000 (\approx 1 mm)
20	840
40	420
70	210
140	105

Also, 1 micron = 10,000 angstroms.

*Uranium Recovery from Seawater
with Chelating Resins*

REPORT TO MIT ON WORK DONE UNDER

Purchase Order No. GC-A-290995

*Peter Osei-Gyimah
ROHM AND HAAS COMPANY
Spring House, PA*

I. Introduction

The availability of a powerful and cheap sequestering agent for radionuclides will find wide application in areas of the nuclear industry such as the extraction of uranium from seawater, the extraction of radioactive elements from contaminated nuclear reactor cooling fluids and the removal of radioactive elements from mining and ore processing activities.

Among the many materials that have been investigated to adsorb uranium from seawater, hydrous titanium oxide (HTO) appears to be the best at the present time. However, the slow rate of adsorption and mechanical weakness suffered by HTO have prompted the experimentation with composite adsorbents with HTO to alleviate these drawbacks and have spurred the search for chelating resins as a completely different type of adsorbent to replace HTO.

Earlier work by Rohm and Haas done under a previous contract from the Department of Nuclear Engineering at Massachusetts Institute of Technology (MIT) concentrated on the synthesis and screening of a series of chelating resins some of which bore amidoxime functional groups as adsorbents for uranium ions from seawater. The results of this work identified the amidoxime functional group as a potentially powerful chelating group for uranium. In the present study it was decided to synthesize additional amidoxime functionalized acrylic and styrenic-based resins in order to identify the most promising chelating resin. The resins will be screened and further tested by collaborators at MIT at their Woods Hole Oceanographic Institute test facility.

2. Experimental

A. Acrylic Amidoximes:

Acrylic amidoxime, SGM-251 was found previously to have a capacity about comparable to HTO for uranium. It exhibited the highest uranium capacity among the group of macroporous resins synthesized. The resin was derived from an acrylonitrile/15% divinylbenzene copolymer by treatment with hydroxylamine. In the present study, analogues of SGM-251 were synthesized to attempt to improve upon the adsorption rate and capacity of the resins. Thus, resins were synthesized that possessed lower levels of crosslinker than SGM-251. Four acrylonitrile (AN)/divinylbenzene (DVB) copolymers containing 8, 6, 4, and 3 percent levels of DVB were synthesized and converted to the amidoxime functionality by treatment with hydroxylamine. To influence the porosity and pore-size distribution, two AN/6% DVB copolymers were prepared using different phase-extenders, toluene and methylisobutylcarbinol, Table 1. For another of these copolymers the acrylonitrile monomer served as phase-extender. In the case of the AN/3% DVB copolymer, two mesh sizes of -20+40 and -70 were converted to the amidoxime functional group. All the other resins were of uniform -40+70 mesh size. Table 1 provides a list of the acrylic amidoxime resins including their porosity and capacity data. The resins were additionally characterized by elemental analysis and IR data.

Table 1

Summary of Acrylic Amidoxime Resins

<u>Code^a</u>	<u>% DVB</u>	<u>Resin Capacity (meq/g dry)</u>	<u>Surface Area (m²/g)</u>	<u>Porosity (cc/g)</u>	<u>Ave Pore Diameter (Å⁰)</u>
POG-503	8 ^b	4.49	28.45	0.341	516
EB-1966E	8	3.05	21.59	0.322	597
POG-501	6 ^b	3.83	8.22	0.198	964
POG-505	6	4.54	46.83	0.529	452
EB-1966B	6 } ^c	3.50	15.73	0.088	225
EB-1966D	6	3.42	11.81	0.253	856
EB-1968A	4	5.04	13.60	0.138	405
EB-1970A	3 } ^d	3.90	—	—	—
EB-1970B	3	3.33	4.57	0.364	3188
EB-1976C	3 ^e	4.72	27.58	0.599	869

^a Satisfactory elemental analyses were obtained for these resins.

^b MIBC was used as phase-extender in copolymer synthesis.

^c Copolymers of EB-1966D and POG-505 were phase-extended with toluene and MIBC respectively. No phase-extender was used for EB-1966B.

^d -20+40 mesh size; to increase amidoxime content, EB-1970A was functionalized using two-molar excess of hydroxylamine over copolymer. Equimolar ratio of reagents was used for EB-1970B.

^e +70 mesh size.

B. Styrenic Amidoxime:

A group of amidoxime functionalized resins on styrene/DVB backbones which are analogs of a previously reported resin, SGM-209, were prepared and are summarized in Table 2. A styrene/3% DVB gel copolymer and three macroreticular copolymers with different levels of crosslinker were converted to their corresponding amidoxime functionality according to Scheme I. Chloromethylated styrene/DVB intermediate was aminated with iminodiacetonitrile followed by treatment with hydroxylamine in methanol solution to give the desired benzyloiminodiacetoamidoxime resins. The resins were characterized by IR, elemental analysis and capacity determination.

Scheme 1

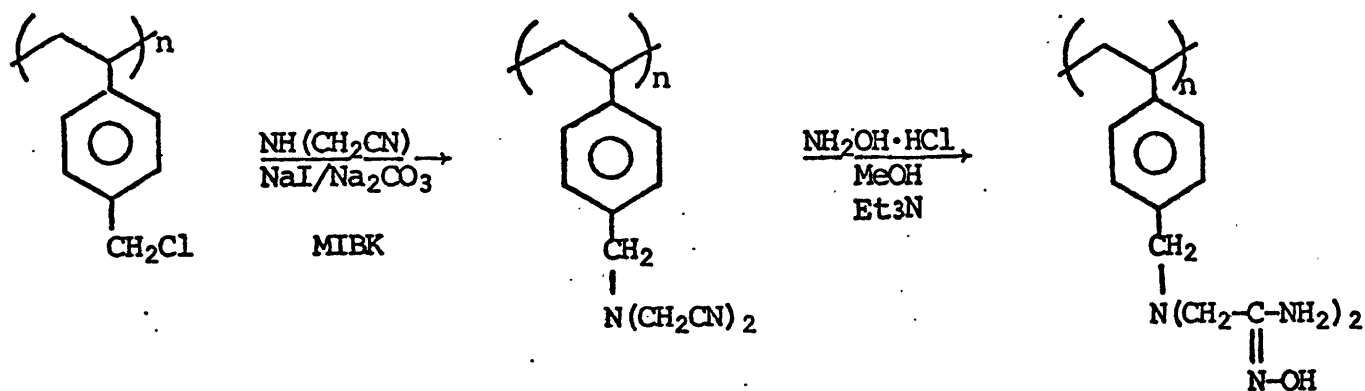


Table 2

Summary of Styrenic Amidoxime Resins

<u>Code^a</u>	<u>% DVB</u>	<u>Resin Capacity (meq/g dry)</u>	<u>Surface Area (m²/g)</u>	<u>Porosity (cc/g)</u>	<u>Ave Pore Diameter (Å^o)</u>
EB-1958C	3	2.67	82.59	0.365	177
EB-1961A	6	4.44	58.24	0.821	564
EB-1968C	3+4% trimethylolpropane trimethacrylate	2.93	26.49	0.688	1038
EB-1972B	3.8	3.15	28.16	0.075	107

^a Satisfactory elemental analyses were obtained for these resins.

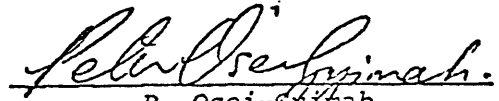
3. Determination of Extent of Surface Fouling of Seawater Treated
SGM-251 (POG-699) by Scanning Electron Microscopy (SEM):

It has been suspected that the surfaces of resins undergo fouling by extraneous materials during the period of exposure to seawater to allow the resins to adsorb uranium. After a 30-day exposure to seawater,

acrylic amidoxime SGM-251 (relabelled POG-699), supplied by MIT, was submitted for SEM to determine the extent of surface fouling. The SEM's were taken by Rohm and Haas Analytical Research which issued a report on the micrographs. The attached analyst report and micrographs confirm the suspicion of debris build-up on the resin surface during the seawater loading process.

4. Conclusion:

We have focused on the amidoxime functionality as a powerful uranium chelating group and have prepared a number of acrylic and styrenic-based resins bearing this functionality. In order to identify the most promising resin and obtain the highest uranium capacity, we varied the physical characteristics of the resins in terms of particle size, DVB level, porosity and pore-size distribution, and degree of functionalization. It is hoped the uranium capacity data we accumulate with these various resins will augment our knowledge and enable the eventual design and synthesis of a commercial uranium chelating resin.


P. Osei-Gyimah

PO/eae
Attachment

ANALYTICAL RESEARCH

SECTION 57 TECHNICAL REPORT NO. 82-353

cc: K. Fletcher
D. B. Fordyce
W. D. Sheasley
*Microscopy
*File

SUBJECT : Surface Fouling of Seawater
Treated Resin POG-699

August 5, 1982

WRITTEN BY : L. M. McGreevy

REQUESTED BY : P. Osei* ✓

PROJECT NO. : 3900.12

OBJECTIVE : Determine extent of surface fouling of resin POG-699.

SUMMARY : POG-699 resins have been treated with seawater to chelate uranium ions. It was suspected that the surfaces of the beads were fouled by extraneous matter during the treatment.

SEM of bead interiors revealed what appears to be a fine macroreticular structure with EDX indicating only low levels of Mg, S and Ca.

SEM of bead surfaces again revealed an apparently reticular structure but with varying amounts of foreign material on the surface. This material ranges from a thin amorphous looking film covering most of the surface to large amounts of crystalline looking material holding together and filling in gaps between adjoining beads. Close examination of this material reveals the presence of the silicon, skeletal remains of diatoms, minute unicellular algae that live in the sea (see D in micrograph). In addition to Mg, S and Ca, EDX indicates the presence of considerable amounts of Al, Si and Fe with lower levels of K and V.

Because of the presence of diatoms in the fouling material, it is quite likely that fouling took place during the seawater treatment.

*Micrographs attached.

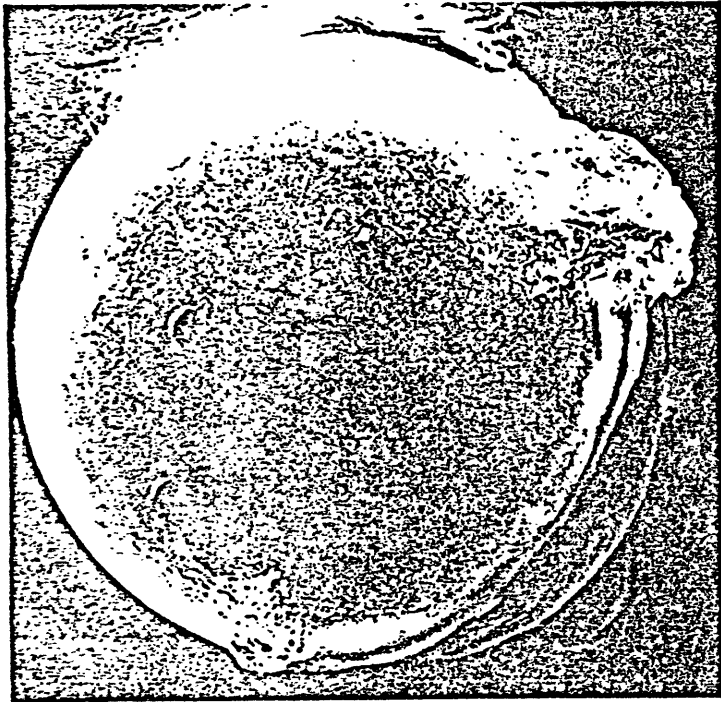
KEY WORDS: seawater, fouling, IFR, diatom,
SEM, EDX

Work Done by: J. Cronin

C4027-0882

SEM AND EDX OF FOULED RESIN POG-699

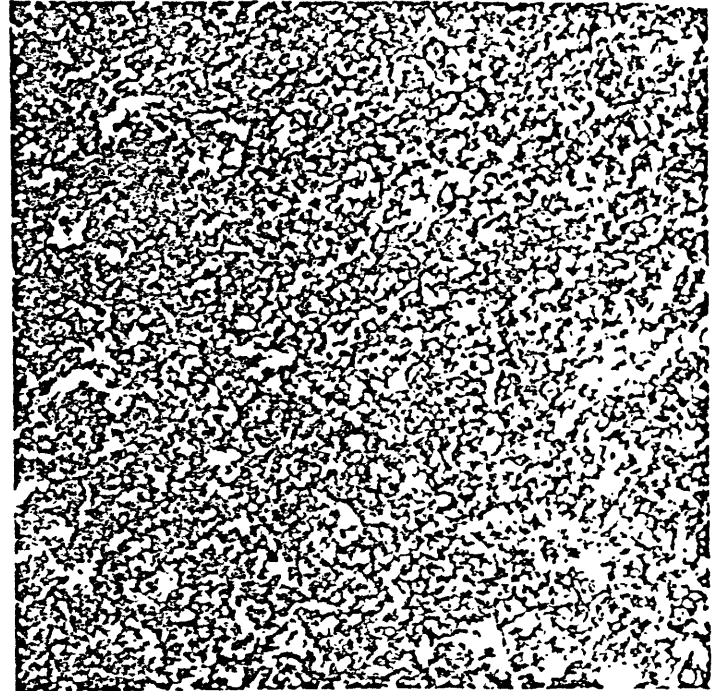
SURFACE



X100

100 μ

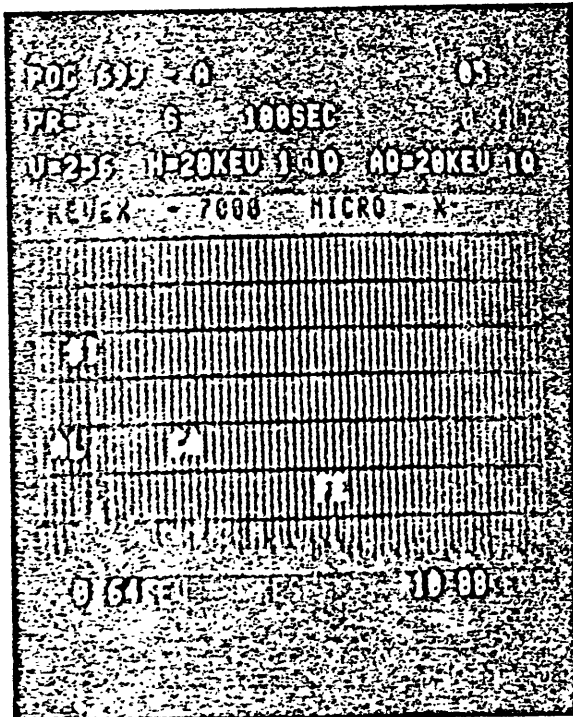
INTERIOR



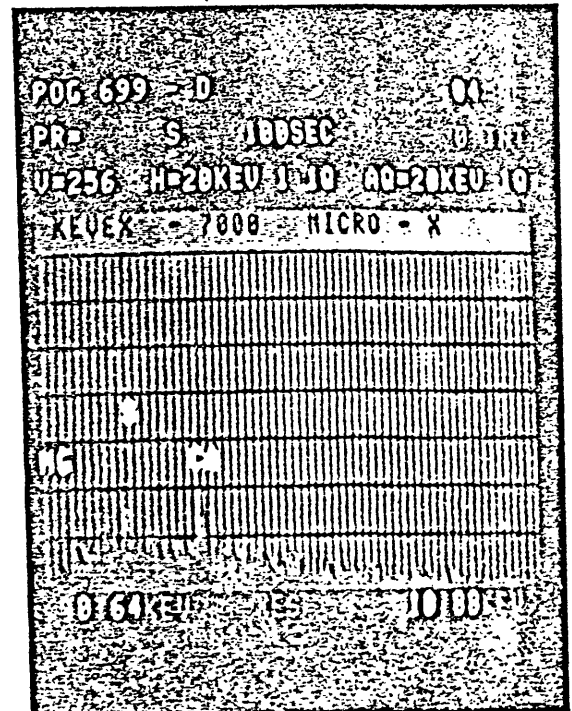
X5,000

2 μ

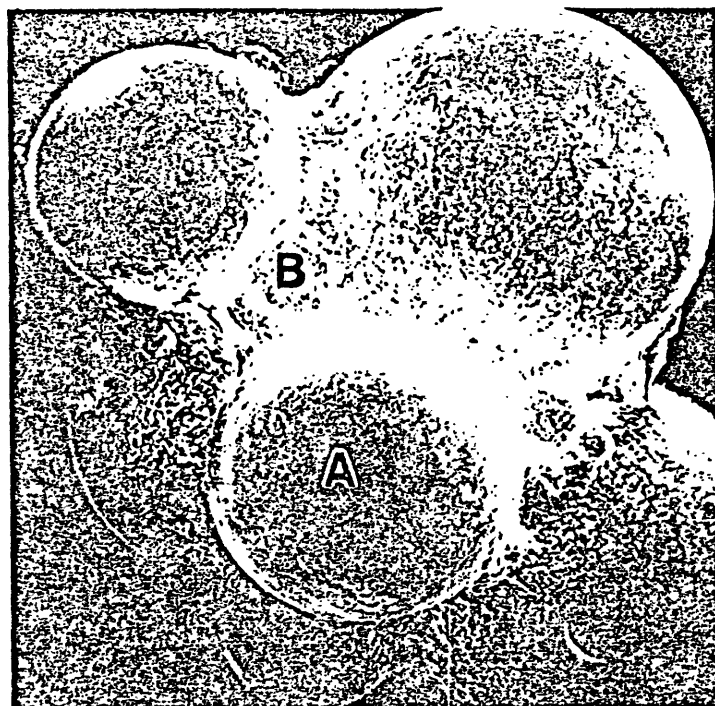
SURFACE



INTERIOR



SEM: FOULED RESIN POG-699



SURFACE

X100

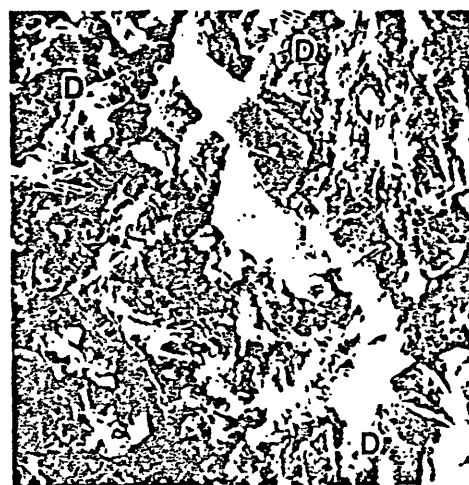
100.μ



A

1μ

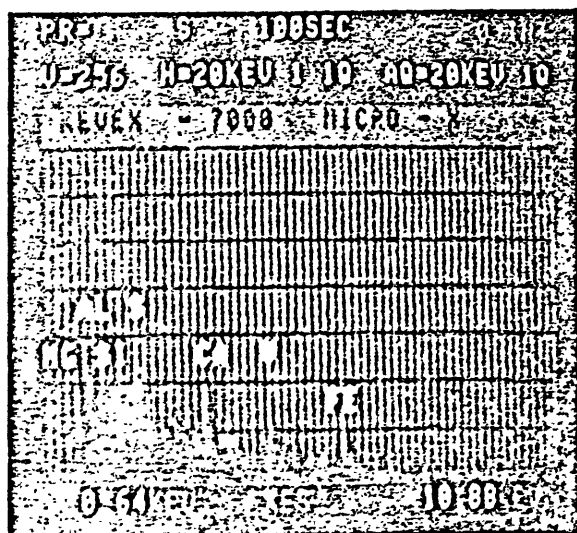
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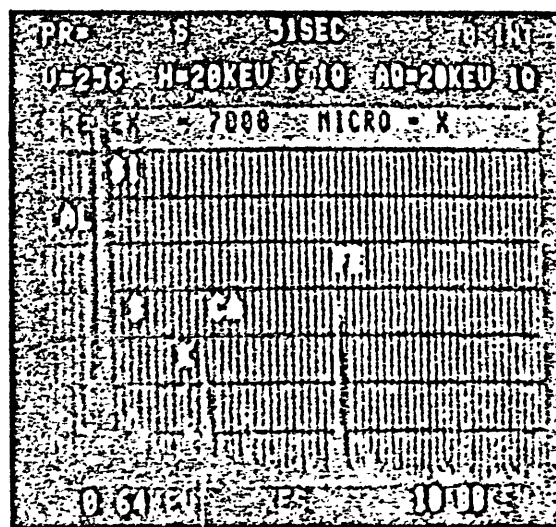
B

10μ

X1,000



A



B