MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL

AECU-3780

UNION CARBIDE NUCLEAR COMPANY
DIVISION OF UNION CARBIDE CORPORATION

MEMORANDUM

EPS-Y-363

KT-335

MASTER

April 9, 1958

TO: G. B. Marrow

FROM: G. R. Seiler, P. R. Ammann, and A. b. Newey

SUBJECT: Resin Attrition

DISTRIBUTION:

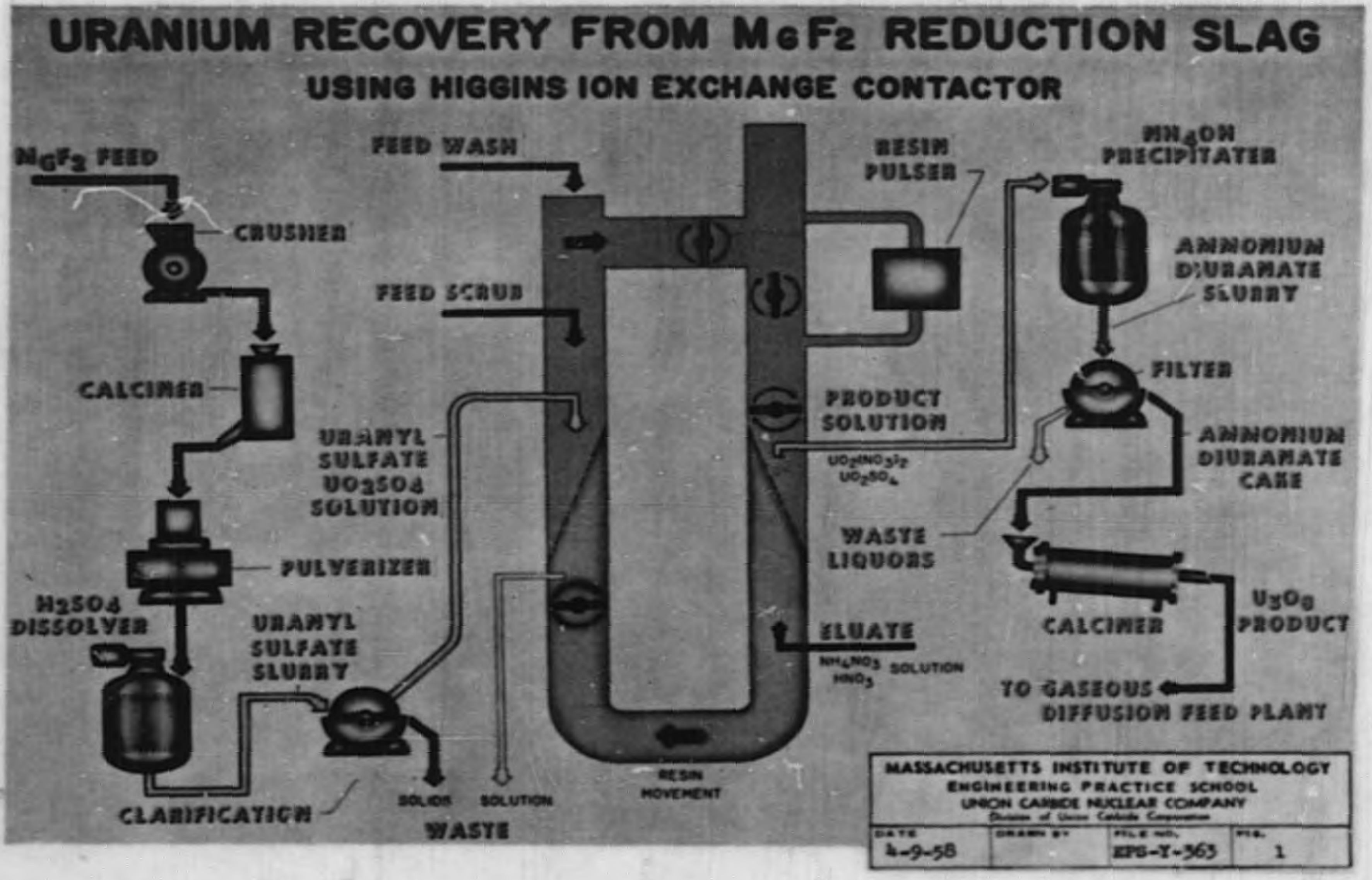
M. J. Fortenbery
J. M. Googin
I. R. Higgins
F. O. Lewis
G. B. Marrow
W. C. Moore
R. C. Olson
R. C. Reid
T. F. Wagner
R. D. Williams
ORGDP Records
ORGDP Records (RC)
Y-12 Plant Records (4)
M. I. T. Practice School (10)

INTRODUCTION

Uranium Recovery Process

At present, uranium metal is produced by the bomb reduction of uranium tetrafluoride with magnesium. The slag from this reduction contains appreciable quantities of uranium, either in the metallic or the tetrafluoride form, in addition to the magnesium fluoride. A process for recovering low enrichment uranium from this slag has recently gone into operation in the Y-12 plant. Briefly, the process (Figure 1) involves the following steps: 1) size reduction of the slag; 2) conversion of any metals in the slag to their oxides by calcining; 3) further size reduction; 4) digestion of the slag in sulfuric acid; 5) clarification to remove the bulk of the magnesium fluoride; 6) uranium

NOTE: This document contains information of a preliminary nature and was prepared primarily for internal use. It is subject to revision and/or correction, and does not represent a final report.


URANIUM RECOVERY FROM MgF2 REDUCTION SLAG
USING HIGGINS ION EXCHANGE CONTACTOR
MgF2 FEED
CRUSHER
CALCINER
PULVERIZER
H2SO4 DISSOLVER
CLARIFICATION
URANYL SULFATE SLURRY
URANYL SULFATE UO2SO4 SOLUTION
FEED WASH
FEED SCRUB
SOLIDS
SOLUTION
WASTE
RESIN MOVEMENT
RESIN PULSER
PRODUCT SOLUTION
UO2(NO3)2
UO2SO4
WASTE LIQUORS
ELUATE
NH4NO3
HNO3
SOLUTION
NH4OH PRECIPITATER
AMMONIUM DIURANATE SLURRY
FILTER
AMMONIUM DIURANATE CAKE
CALCINER
U3O8 PRODUCT
TO GASEOUS DIFFUSION FEED PLANT
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
DATE
4-9-58
DRAWN BY
FILE NO.
EPS-Y-363
FIG.
1

KY-335

adsorption on and elution from an ion exchange resin; 7) conversion from the uranyl salt to ammonium diuranate; and 8) conversion to uranium oxides.

The Higgins Semi-continuous Contactor

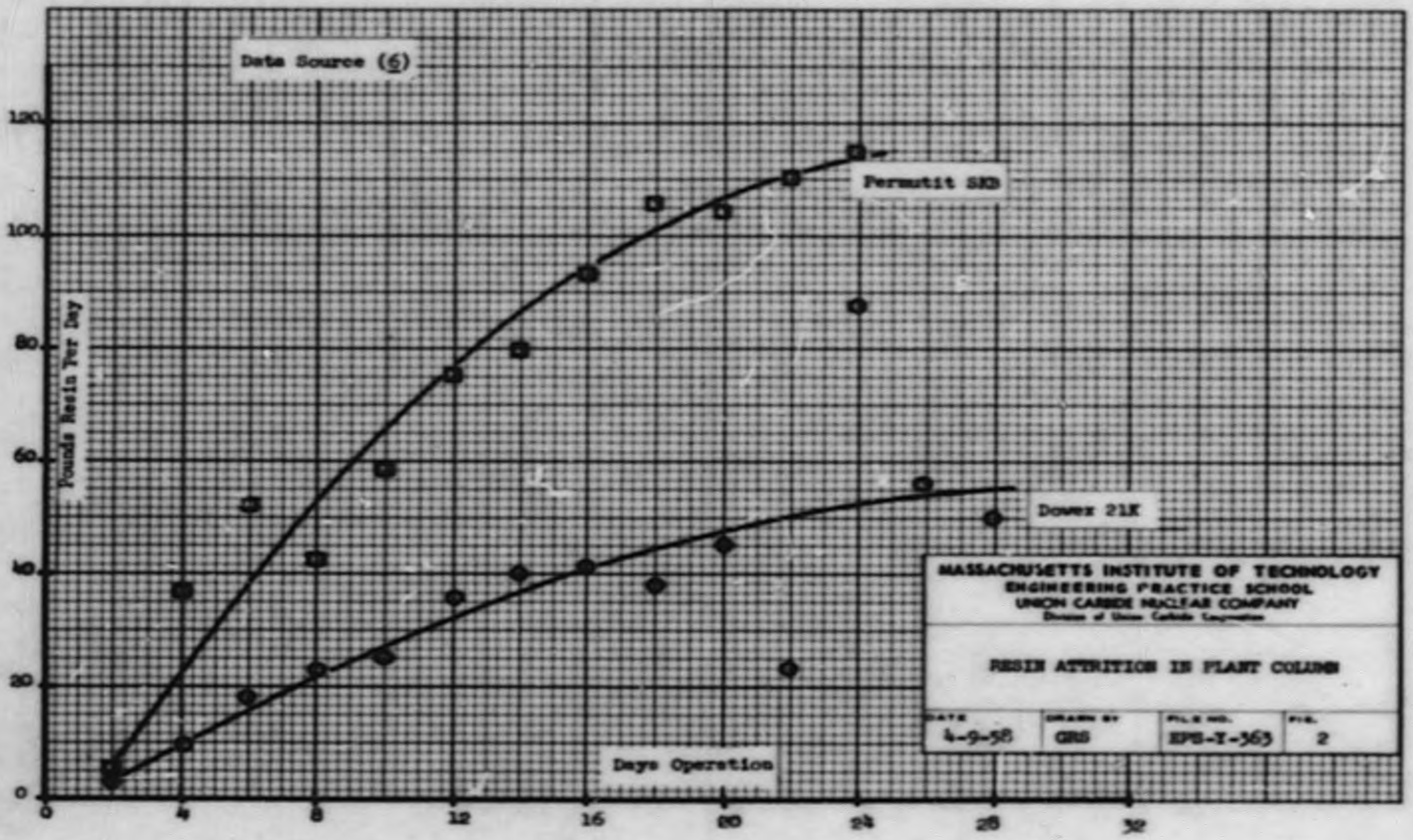
The most unusual phase of this operation is the first application of the Higgins semi-continuous ion exchange contactor to an industrial scale operation. The column operates in cycles with countercurrent movement of the feed liquid down through the resin and the resin up through the adsorption column taking place alternately once in each cycle. Similarly, the eluate passes up through the resin and the resin passes down through the stripping column. In the feed part of a cycle the feed liquid (actually a slurry containing less than two per cent of very fine solids) is first forced down through the column operated as a fixed bed. Simultaneously, a feed scrub (dilute $(NH_4)_2SO_4$) followed by a water wash flows through loaded resin before it is discharged to the stripping column. The scrub and wash sections are at the top of the adsorption column and above the feed point. After operating in this manner for an appropriate length of time, the feed and water wash valves are shut and a valve at the top of the standpipe is opened. A pulse pump operates recycling feed wash liquid, forcing fresh resin into the adsorption column, and causing a general movement of the resin. The resin in the wash section is moved by freshly loaded resin into the similarly operated stripping column. To complete the cycle the resin from the stripping column is returned to the loading column. During the pulse resin is elutriated into the top of the stripping column, the fines removed, and the large particles returned to the pulser (see Figure 1). The height of the column currently used is fifty feet and the diameter is one foot.

Criteria for Resin

The exchange mechanism from the feed liquid to the resin is one of adsorption and is controlled by the diffusion of the ions into the resin beads (1). Thus smaller resin particle sizes enhance mass transfer and, for a given weight of resin in the column, result in higher column capacity. However, attendant problems with small particles are the increased pressure drop through the column and possible blockage of column flow by the resin particles. Furthermore, the bead size must be different from the solid particle size in the feed slurry to effect countercurrent flow. The optimum resin particle size has been found to be - 16 + 20 mesh. Thus one of the problems associated with column operation is the attrition of the resin particles (see Figure 2). The resin originally used (Permutit SKB) in the column was replaced on January 20, 1958, by Dowex 21K because of excessive particle size reduction. The present attrition (about 0.5% per day on the basis of elutriated fines), however, is still higher than that desired (6).

Previous Work on Resin Attrition

The reasons for this attrition are not well known, but several possible causes have been advanced: 1) friction between wall and/or resin during bed movement; 2) crushing by the butterfly valves during operation; 3) effects due to the internal stresses set up by the alternate swelling and shrinking of the beads during the repeated chemical cycles; and 4) crushing of the beads due


Data Source (6)
Pounds Resin Per Day
0
20
40
60
80
100
120
0
4
8
12
16
20
24
28
32
Days Operation
Permutit SK
Dowex 21K
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
RESIN ATTRITION IN PLANT COLUMN
DATE
4-9-58
DRAWN BY
GRS
FILE NO.
EPS-X-363
FIG.
2

to the weight of the resin bed. Synergistic effects may also be present and affect the amount of attrition.

Kunin and Meyers (4) mention briefly three tests to determine the attrition characteristics of resin particles; 1) rapid hydraulic cycling of the resin (which they feel is an unreliable test); 2) grinding of the particles in a ball mill; and 3) chemical tests to determine particle stability during the cycling. No positive results have been noted.

Some preliminary work has been done on the proposed mechanisms of attrition by the SD Chemical Development group at the Y-12 plant. Essentially all resin attrition noted is due to particle breakage rather than wearing away at the surface (1). The effect of valve operation was tested by opening and closing a butterfly valve in a short, vertical pipe filled with resin. While the valve was open, the resin bed was fluidized by air. The attrition rates found were not significant compared to that of the actual plant operation (6). Analysis of plant data from both experimental and theoretical aspects indicate that the "simple hydraulics" of the column operation do not significantly contribute to the resin attrition (5). However, the weight of the resin bed upon the particles in the lower sections of the column exceeded the bead strength of the Permutit resin (1). A test was made in which the resin was circulated in a pilot Higgins' column to study the friction effects. No significant attrition was noticed (5).

Very little conclusive work has been reported on resin attrition, even though the economy of ion exchange processes is such that the proper balance rests solely on a prior estimation of the loss of resin that may be caused by either mechanical or chemical attrition (4). Resin costs for the uranium recovery operation are currently over $2,000 per month, or over ten per cent of the total raw materials cost for the operation (6).

Character of This Investigation

This memorandum is the result of a request to study the causes of resin attrition and their relative magnitude both from an overall point of view and with specific reference to the Higgins' column previously described and Dowex 21K resin.

Equipment was designed, assembled, and tested to investigate the effects of 1) valve action, 2) wall to resin and resin to resin friction, 3) repeated chemical cycling, and 4) column height. Certain mechanical properties of the resin beads were investigated, and some existing plant data were analyzed.

A discussion of the probable mechanisms of attrition and their contribution to the overall amount of attrition is presented.

PROCEDURE

Chemical Cycling

An apparatus (Figure 3) was set up to investigate (a) chemical attrition and (b) variation of bead strength, of Dowex 21K resin, resulting from chemical cycling through the uranyl sulfate and nitrate forms. A resin sample was contained in a flexible unichrome tube (1.94 in. ID, 22.5 in. long) to isolate it from mechanical disturbances; the tube was fitted with a screen and distributor plate at the bottom. Chemical conversion was accomplished by dropwise addition of either uranyl sulfate column feed or 0.1N HNO_3 - 0.9N NH_4NO_3 solutions. Fines generated from chemical attrition were washed overhead (elutriated) with water flowing through the rotameter to the bottom of the bed, collected on a polyethylene screen filter, dried and weighed. Small samples of both forms of the resin were taken from the bed periodically during the series of cycles, and bead strength under shear and compression forces was measured.

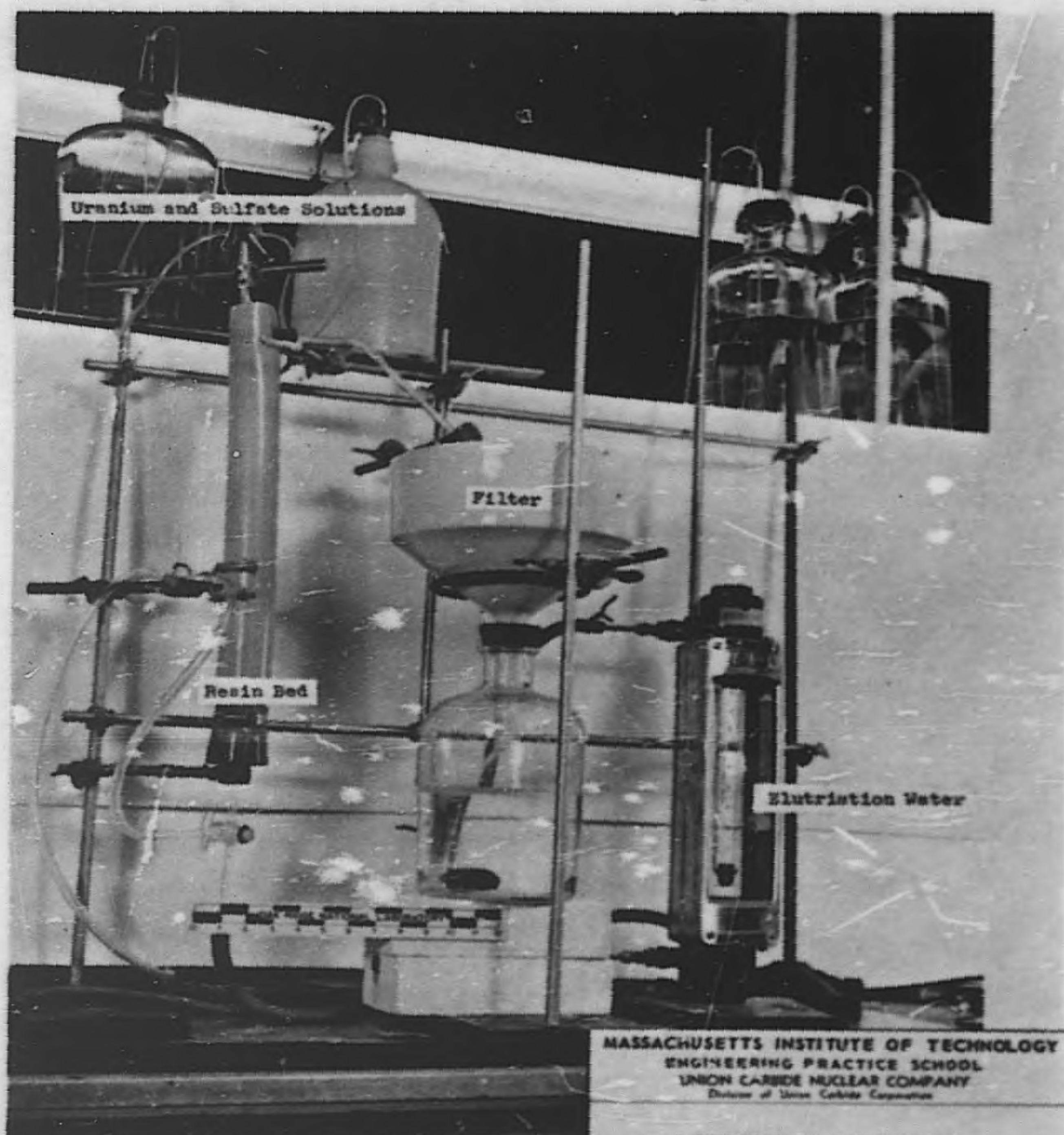
With the shear test apparatus (Figure 4) a couple is applied to a bead by the free moving top plate, to which force is in turn applied by loading the pan with Meriam No. 3 fluid from a burette. With the compression test apparatus (Figure 5) the bead is compressed between the right-hand pan of the balance and the lucite rod supported just above it, by weighting the other pan with water measured from a burette. Both tests were conducted to determine only the applied force at complete bead fracture.

Initially, 100 cc of new Dowex 21K resin (chloride form) was placed in the unichrome tube, elutriated to remove initial fines and treated for a seven-hour period with the nitrate solution to convert it to the nitrate form. The resin was then uranium-loaded for four hours and the fines produced in this first cycle removed by a one-half hour elutriation at a water rate of 0.355 gpm. To begin the second and all subsequent cycles, the nitrate elution step (reduced to two hours) was repeated, the following uranium load period remaining at four hours. Ten complete cycles were run, and resin samples for the bead strength tests were taken during and fines elutriated after the first, second, and all even numbered cycles through ten. The dry weight of the original 100 cc of resin, taken to be the same as that of a second sample of equal volume, was 29.9 gm.

The amounts of fines collected during the series of cycles were very small, and, as a final check on the chemical attrition, individual whole beads and broken pieces of the ten cycle uranium form resin were visually counted. For comparison purposes a similar count was made of new Dowex resin from which the "initial fines" had been elutriated in the test apparatus.

Transparent Valve Study

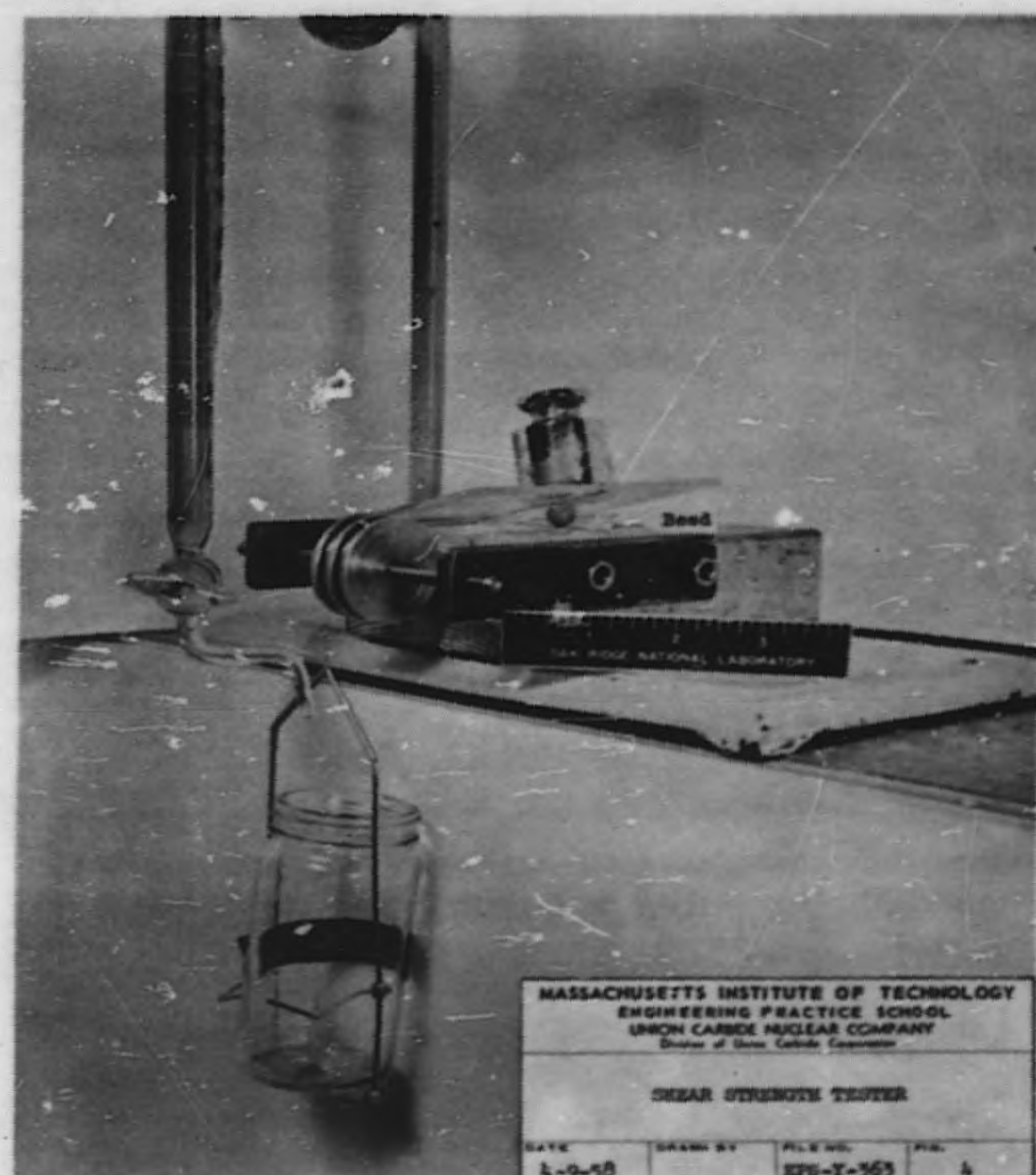
A lucite butterfly valve (Figure 6) was designed and built in order to study the flow patterns of the resin as the valve opened and closed. The valve was placed inside a four-inch lucite tube which was eighteen inches



MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

CHEMICAL TEST APPARATUS

DATE	DRAWN BY	FILE NO.	FIG.
4-9-58		EPS-Y-363	3


Bead
OAK RIDGE NATIONAL LABORATORY
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
SHEAR STRENGTH TESTER
DATE 4-9-58
DRAWN BY
FILE NO. EPS-X-363
FIG. 4

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

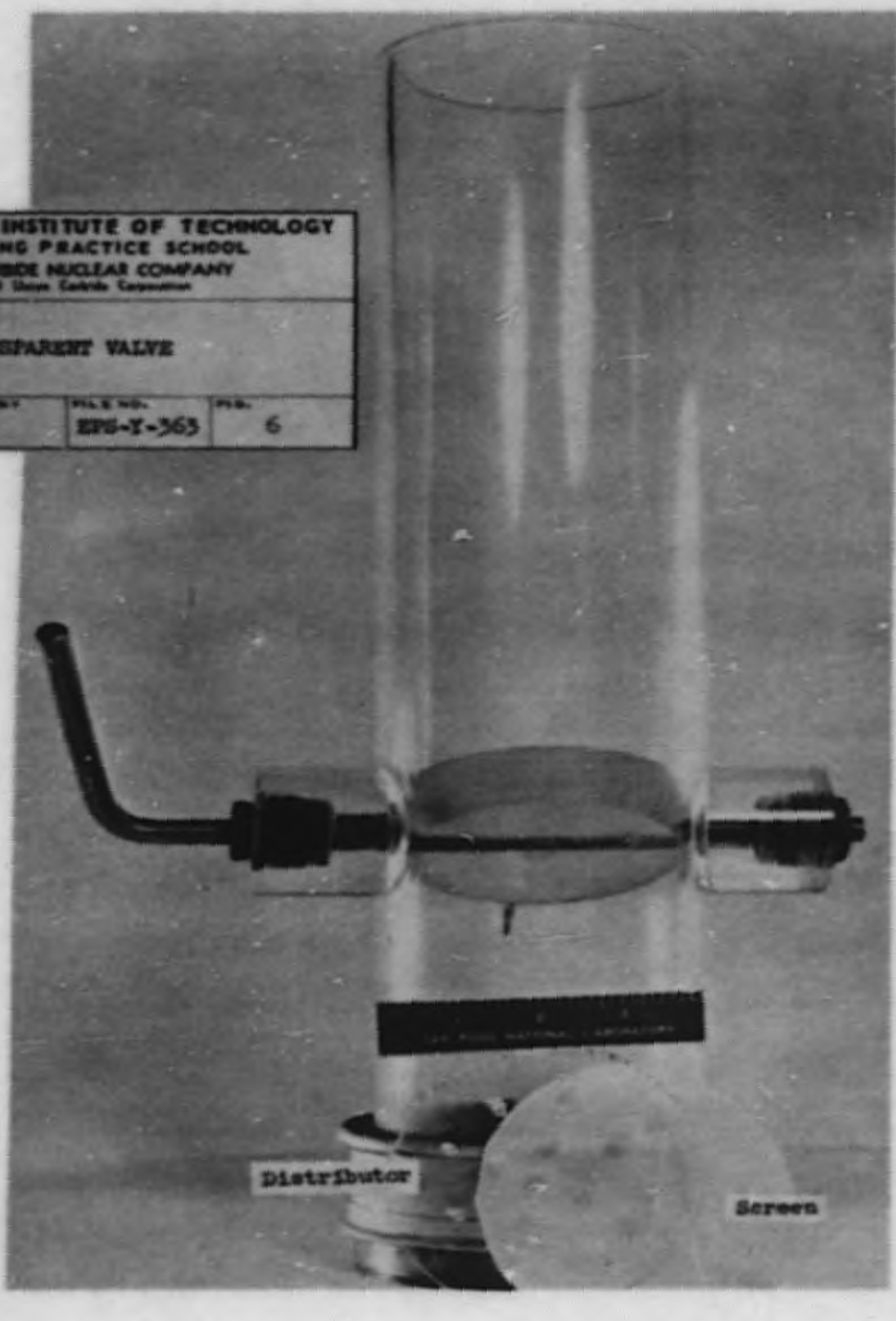
COMPRESSION STRENGTH TESTER

DATE	DRAWN BY	FILE NO.	FIG.
4-9-58		EPS-Y-305	5

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

TRANSPARENT VALVE

DATE	DRAWN BY	FILE NO.	FIG.
4-9-58		EPS-X-363	6



long. A sixty mesh screen was placed on the bottom of the tube to contain the resin beads. A distributor and screen were made to fit the top of the resin column.

Visual observations were made of the flow characteristics of the resin, the effect of packing (loading), and the attrition at the valve to wall contact points. The butterfly valve was operated with water running downward through the resin bed and with a mechanical load of zero, 4.5 kg., 10.65 kg., and 24.2 kg. The mechanical loads were lead weights which were placed on top of the distributor. Also, the apparatus was run with water flowing upward through the bed and no loading on the top.

Column Pulse and Valve Testing

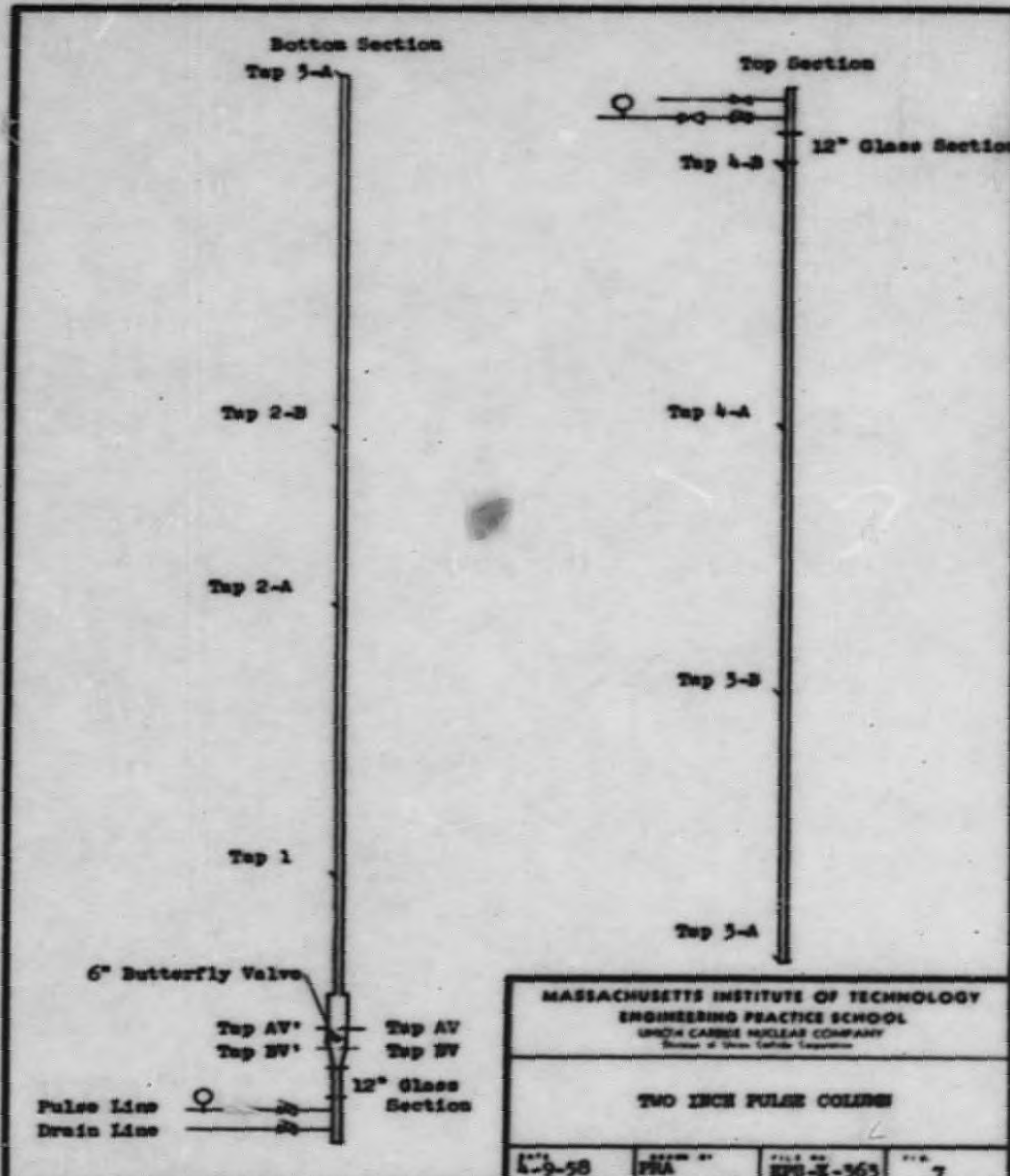
A fifty-foot column of 2-inch steel pipe (Figure 7) was erected in order to study and isolate the mechanical causes of resin attrition under simulated production conditions. Details of the 6-inch butterfly valve section are shown in Figure 8.

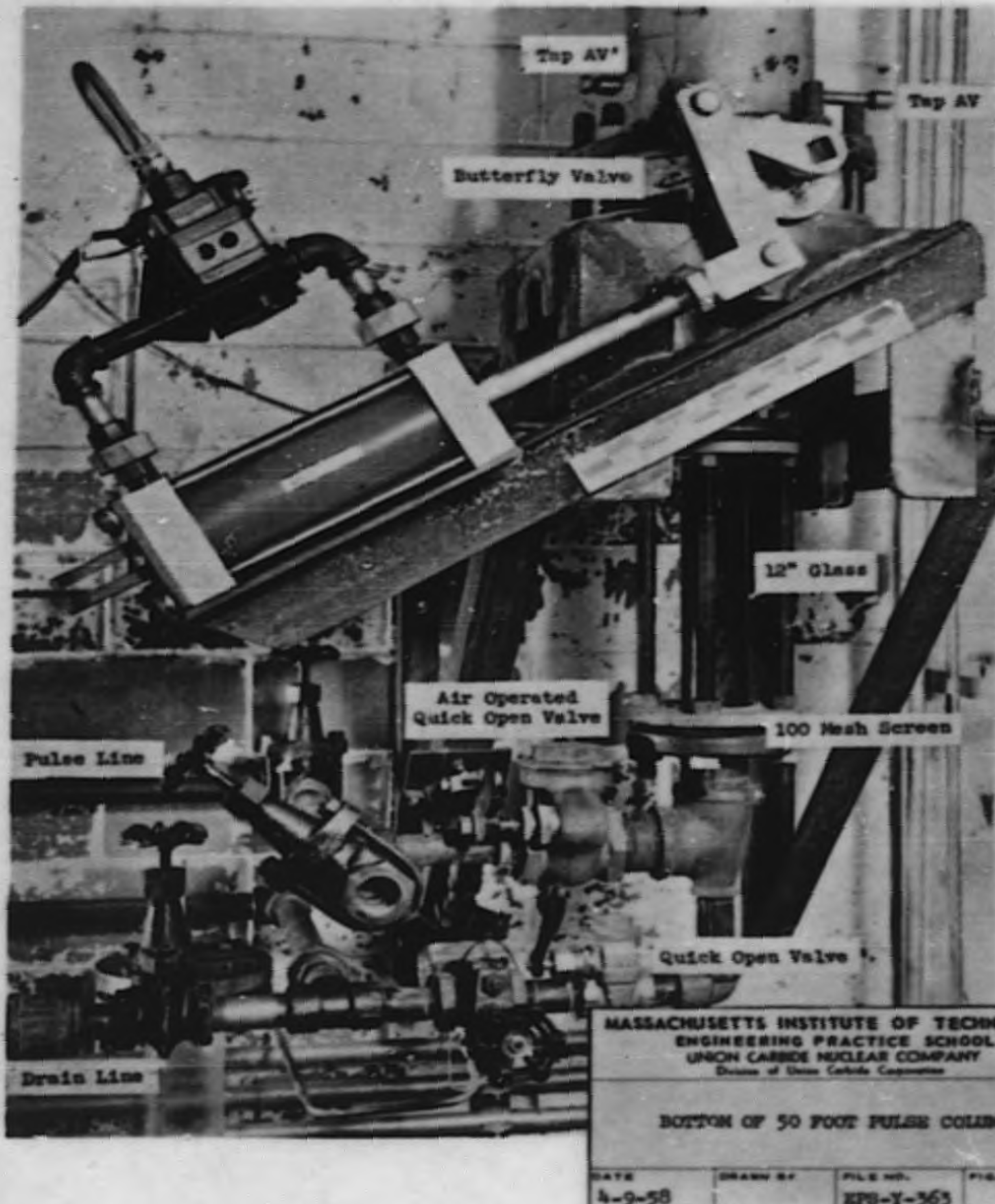
The column was filled with new Dowex 21K resin (chloride form), and samples were taken at taps BV, 2-B, and 3-A (Figure 7) before starting operation. With the butterfly valve open, the column was pulsed 15 seconds and then drained 15 seconds for 214 cycles. During the operation the top water line was on and the top vent cracked to maintain 40 psig pressure. The pressure in the pulse line (bottom) was 65 psig. The resin bed averaged an eight-inch rise on each pulse. Then samples were taken at the same points.

The drain line was then closed and the column operated on a 15 second on-45 second off pulse cycle for 4 1/2 hours. However, sometime during this phase of the operation the pulse valve jammed, so that the actual number of cycles run with this procedure is unknown. Samples were again taken.

Following these tests, attrition by the butterfly valve was tested by applying the following cycle: pulse 5 seconds, drain 11 seconds, butterfly valve closed 5 seconds, and equilibrate 9 seconds. Prior to this test, resin was added to the column and samples were taken at BV, AV, 2-B, and 3-A. Water was continually fed to the top of the column and the vent cracked to maintain 40 psig. The pressure in the pulse line was 75 psig. The resin bed rise averaged 10 inches per pulse. This procedure was followed for 225 minutes (450 cycles), and samples were taken at the taps mentioned above. The same procedure was repeated for another 230 minutes (460 cycles), and samples were again taken at taps BV, BV', AV, AV', 2-B, and 3-A. All samples were then tested for resin breakage.

The samples were wet screened to determine the per cent fines and the fines size distribution. Standard screening methods were used. A measured aliquot of the sample, normally 100 ml, was screened through 20, 30, 50, and 325 mesh screens with water running over the samples at all times. The volumes of resin remaining on each screen were then measured and reported as a per cent of the total volume tested.


Bottom Section
Tap 3-A
Tap 2-B
Tap 2-A
Tap 1
6" Butterfly Valve
Tap AV'
Tap BV'
Tap AV
Tap BV
12" Glass Section
Pulse Line
Drain Line
Top Section
12" Glass Section
Tap 4-B
Tap 4-A
Tap 3-B
Tap 3-A
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
TWO INCH PULSE COLUMN
4-9-58
PHA
EPS-X-363
7



MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

BOTTOM OF 50 FOOT PULSE COLUMN

DATE	DRAWN BY	FILE NO.	FIG.
4-9-58		EPS-Y-363	6

The following roll test was made on each sample to determine the per cent of non-spherical (broken) particles. The samples were dried and placed on a glass plate inclined at approximately three degrees to the horizontal. The particles which rolled down the plane were spherical particles which had not undergone attrition. The fraction of the resin particles which are spherical is reported as that per cent (of the total final volume) which rolled down the plane.

DISCUSSION

Chemical Tests

A. Production of Fines

A distinction should be made between the bases on which attrition figures are reported. In general, they are calculated from either (1) the amount of fines (measured by wet volume, wet weight or dry weight) elutriated from a resin bed, or (2) the relative numbers of broken and whole beads as counted in a given sample. For a given sample, the volume and weight per cent fines would be expected to be roughly equivalent to each other but lower than the true per cent of broken beads because, as is discussed below, elutriation does not remove all of the fines present. The current plant attrition rate of 0.5% per day is calculated from the wet weight of fines removed in the column elutriator.

Negligible amounts of fines were elutriated during the chemical cycling experiment (Table I) compared to the 0.2% per cycle of wet volume of elutriated fines obtained in previous work on the Permutit resin using a similar fixed bed arrangement (2), and the Dowex 21K is therefore definitely superior to the Permutit in this respect. The elutriation water velocity in the present experiment (approximately 0.38 ft./sec.) was somewhat higher than that used in the latter study but only half that presently employed in the plant column, 0.77 ft./sec. (7). This velocity could not be achieved in the 22 in. tube because it fluidized the resin bed to the extent that whole beads were passed over the top.

TABLE I

Elutriated Fines

Cycles Completed	Weight of Fines (grams)
1	0.0088
2	0.0028
3 and 4	0.0026
5 and 6	0.0340*
7 and 8	0.0020
9 and 10	0.0012

Dry weight of 100 ml resin sample (chloride form): 29.9 gms.
Initial Fines (removed prior to start of the chemical cycles): 0.49 gms.

*Prior to this elutriation the water line had been disconnected and some air was in the system. Many of the particles were carried over by air bubbles, which may account for the discrepancy between this and the other figures. Air was not observed in any of the other elutriations.

A comparison to the plant attrition rate is further complicated by the fact that the elutriation process does not afford a clear cut separation between particle mesh size fractions and fails to remove a considerable portion of the fines in the plant column, undoubtedly because they are trapped and retained by the surrounding resin mass even though it is fluidized. The bead counts (Table II) showed that this was also true of the experimental fixed bed; and until information on the relative efficiencies of the plant versus the bench elutriations is available, a direct conclusion from the results of this phase of the chemical tests (i.e., that negligible amounts of elutriatable fines are produced from chemical cycling) cannot be made.

The resin bead counts were made by taking several independent samples of the ten cycle uranium loaded resin and new Dowex 21K resin, placing them on a dark surface, and visually counting individual particles to determine the number of whole particles and the number of parts of fractured beads (chips) in each sample. The per cent fractured was calculated assuming that each chip was one-third of a whole bead.

TABLE II

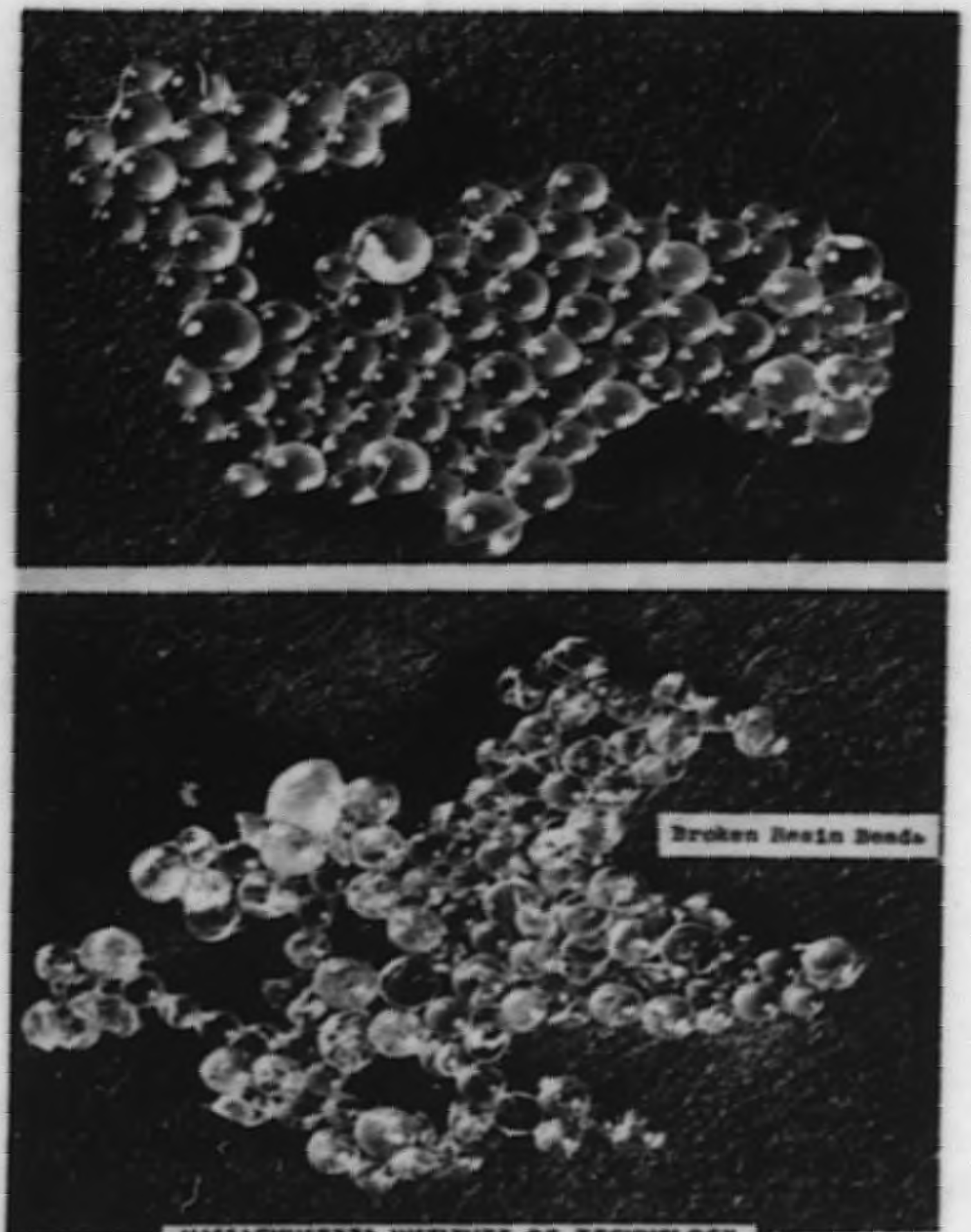
Individual Bead Counts

	Whole Beads		Chips
(a) Ten Cycle Resin:			
	336		75
	293		52
	750		107
	716		81
	960		196
Total:	3055		511
Per Cent Fractured:		5.6	
(b) New Dowex 21K Resin:	**Whole Beads**		**Chips**
	406		39
	542		43
Total:	948		82
Per Cent Fractured:		2.8	

From the counts, the rate of attrition in terms of beads fractured averaged about 0.28% per cycle over the ten cycles completed. There were many more beads which were cracked and deformed after cycling than in the new resin samples. These were taken as whole beads and are not included in the attrition figure given above. Figure 9 shows photographs of new and used Permutit resin. The effects of attrition--cracking, scoring, and deformation--are clearly visible and, although more pronounced, are qualitatively similar to the ten cycle Dowex appearance.

For the reasons discussed above, the fraction of counted fines which would be elutriated under plant column conditions is unknown, and again no direct comparisons are possible. Significant bead fracture appears to result from chemical cycling. Because all the fractured particles are not removed in the plant elutriation, the plant attrition rate on a beads fractured basis is undoubtedly considerably higher than the quoted 0.5% per day and chemical effects are not the major contributor to total attrition, assuming that long-term chemical break-up is not more serious.

The long-term effects of chemical attrition should be further studied over a large number of cycles. A longer tube for the resin bed would be desirable, so that higher elutriation rates may be achieved and a valid comparison with plant data perhaps be made. If whole beads are brought over during elutriation, they should be screened and the +20 mesh fraction returned to the resin bed as is done in the plant. Samples should be taken periodically and individual bead counts made to determine the rate of breakage. Investigation should be made as to the use of the flying spot scanner available at ORNL to facilitate the counting operation.



MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

PERMUTIT RESIN: NEW (TOP)
AND CYCLED (BOTTOM)

DATE	DRAWN BY	FILE NO.	FIG.
4-9-58		EPS-X-365	9

B. Bead Strength

The following qualitative conclusions are drawn from the results of the shear and compression tests (Tables III and IV). (1) Bead strength distribution, particularly of the uranium loaded resin, shifts downward with continued chemical cycling. The data from both the shear and compression tests of the uranium form clearly indicate this downward trend. (2) The nitrate form of the resin is stronger than the uranium form and appears to weaken less from chemical cyling. Thus from Table III (b) in the 300-400 gm. range the per cent which fractured increased from 9.5% to 27.3% over ten cycles, but at the same time the per cent fractures at a compression force greater than 700 gms. increased from 21.8% to 36.4%. Obviously the size of the sample tested was too small, but there is no trend apparent which parallels that of the uranium form resin. (3) The fracture strength of individual beads varies over a wide range, for example, at least 555 gms. compression load.

TABLE III

Bead Strength Distribution Compression Test

(a) Uranium Form:

	Percentage of Beads in Range				
Cycle Number:	1	4	6	8	10
Fracture Strength Range (gms.):					
250-300		7.7	12.5		9.1
300-400	15.4		12.5	15.4	27.3
400-500		23.1	6.3	23.1	27.3
500-600	30.8	7.7	31.2	23.1	
600-700	15.4	23.1		15.4	9.1
+700	38.4	38.4	37.5	23.0	27.2
Total Beads Tested:	13	13	16	13	11

(b) Nitrate Form:

	Percentage of Beads in Range			
Cycle Number:	1	6	8	10
Fracture Strength Range (gms.):				
250-300	9.5	6.2	5.8	6.0
300-400	9.5	6.2		27.3
400-500	21.8	25.0	11.8	9.1
500-600	18.7	6.2	11.8	12.1
600-700	18.7	18.9	17.6	9.1
+700	21.8	37.5	53.0	36.4
Total Beads Tested:	32	16	17	32

TABLE IV

Average and Range of Bead Strengths from Shear Tests

Cycle Number	Form	Range (gms.)	Average (gms.)	Total Beads Tested	"No Shears"*
New	Chloride	110-191	160	10	0
1	Uranium	149-249	156	10	0
1(repeat)	"	150-201	174	7	2
4	"	111-209**	152	10	0
4(repeat)	"	94-178	134	5	0
6	"	108-137	123	10	5
8	"	103-149	122	7	2
1	Nitrate	105-278	168	11	1
1(repeat)	"	93-173	125	14	8
4	"	125-200	158	10	0
6	"	91-208**	159	10	0
10	"	141-175	158	12	9

*"No Shears" refers to beads which pulled through the tester without fracturing. The average is based only on the actual fractures obtained.

**These figures may be high by as much as 20 gms. due to a change in the friction calibration of the tester.

Shear tests were also run on a sample of Permutit resin (sulfate form) which had been run through ten uranium-nitrate-sulfate cycles and on a sample of K-1 (plant adsorption column) resin (nitrate form).

	Range	Average	Number Tested
Permutit:	14-27	19.4	10
K-1 Resin:	82-287	162	10

Some discrepancies are evident in both sets of figures. A primary reason for this is that an insufficient number of beads were tested. Because the range of strengths is large, tests should be run on perhaps a hundred or more beads from any given resin sample to obtain consistent information. In a small sample, one or two particles fracturing in statistically "improbable" ranges may badly distort an average value. Also, in the case of the compression tests, some beads did not fracture in the 250-700 gm. range and their strengths are reported only as being greater than 700 gms. For these reasons, the averages may not be particularly significant, and analysis of moderate sized samples on the basis of distribution of strengths, as of the compression tests, which were run on somewhat larger bead samples than the shear tests, is the more promising approach.

The "no shear" beads in the shear test were in many cases simply those small enough to slip through the cross wires without being deformed sufficiently to fracture. The majority of no shears were of the nitrate form beads, which are in general smaller than the uranium form, and in addition they may not suffer an equivalent drop in strength, as noted above.

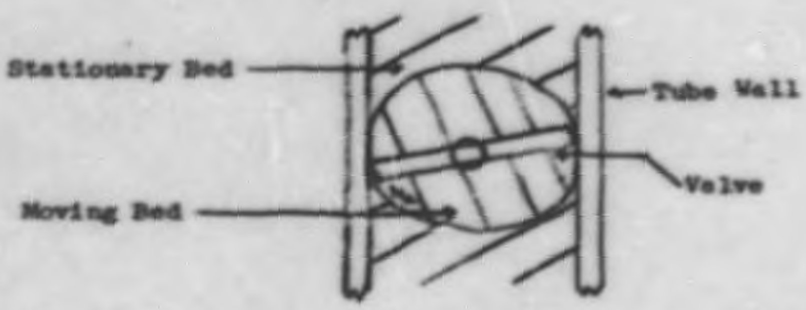
The resin beads are much less resistant to impact than to slowly applied forces. In the production operation the beads are subjected to impact forces when the valves are opened and closed and when the bed is pulsed, and a test to investigate the effects of impact forces upon the resin particles should be devised. Strength tests should be done on partially converted (chemically) beads, which may be weaker than either the complete uranium or complete nitrate form. For example, on the uranium load cycle the outer portion of a bead expands and pulls away from the core as it is converted to the uranium form, the core remaining in the nitrate form until uranyl sulfate ions begin to diffuse into it, and severe opposing internal stresses may be set up.

Effect of Uranium Loading

Data (Figure 10) taken from the production column, supplied by G. B. Marrow (6), on attrition versus the amount of uranium loaded onto the resin indicates that the attrition rate varies almost directly with the amount of uranium loaded.

Transparent Valve Study

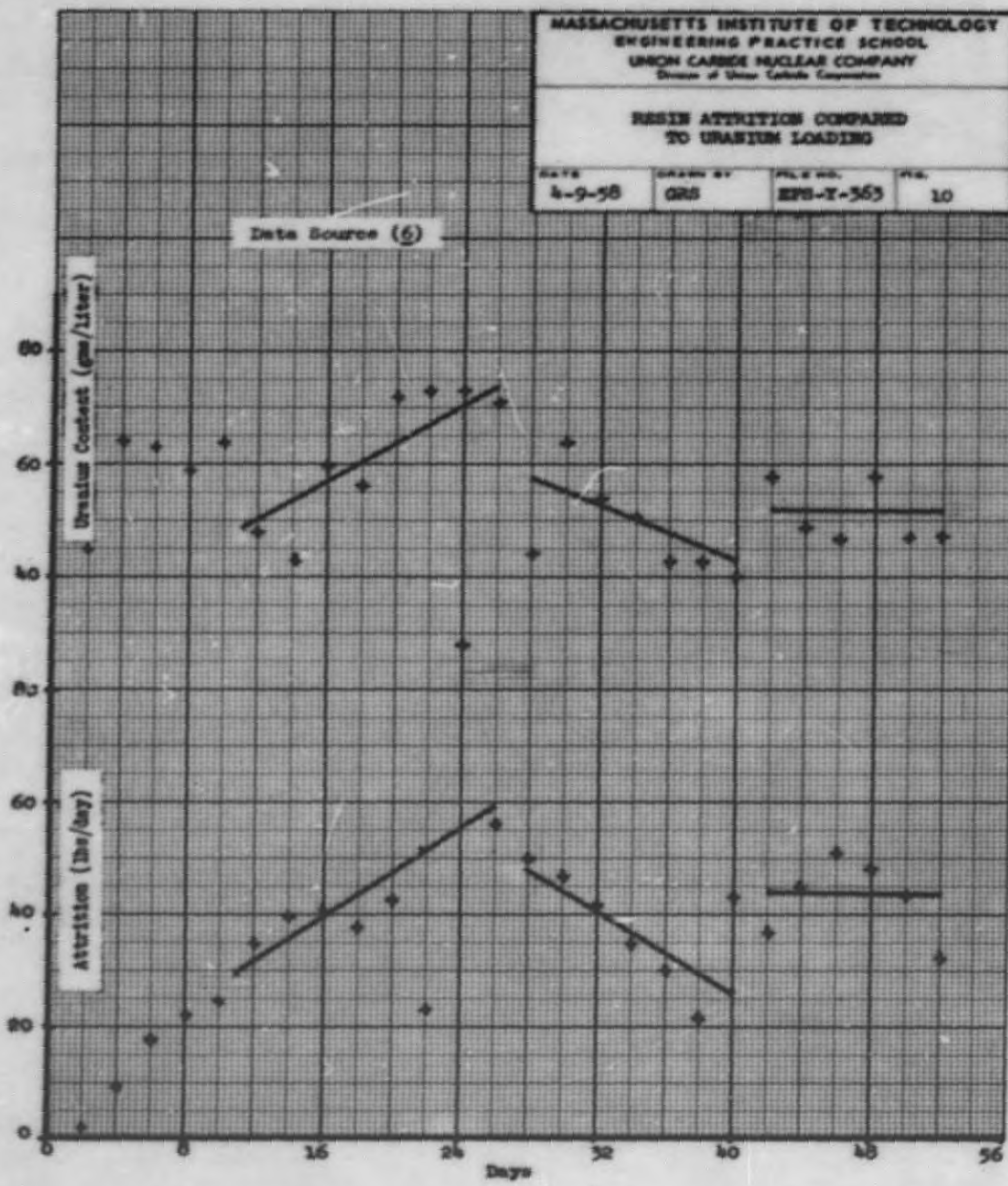
The resin which moves with the valve is shown in the following sketch:



Cross Section

There was essentially no difference in the pattern shown for the various conditions studied. The "moving section" was somewhat compressed as mechanical load was added.

As the lucite butterfly valve was opened and closed, a band of light-colored resin particles gathered at the valve-to-wall contact point and another gathered at the boundary between the moving and the stationary resin. This phenomenon


MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
RESIN ATTRITION COMPARED TO URANIUM LOADING
4-9-58
GRS
EPS-Y-363
10
Data Source (6)
Uranium Content (gm/liter)
Attrition (lbs/day)
Days

is clearly shown in Figure 11. The broken particles are lighter in color than those which are whole (due to changes in light reflecting and refracting properties of the shapes). Also, the attrition occurring when the valve closed could be both seen and heard. This clearly indicates that the butterfly valve causes attrition.

There appears to be a direct relationship between the amount of load applied to the column and the force necessary to operate the valve.

When the resin bed was somewhat fluidized (water flowing upward through the bed) there was some tendency for the flow to sweep the valve-to-wall contact area partially free of resin when the valve was closed slowly. However, the patterns described above were still quite pronounced.

Fifty-Foot Column Study

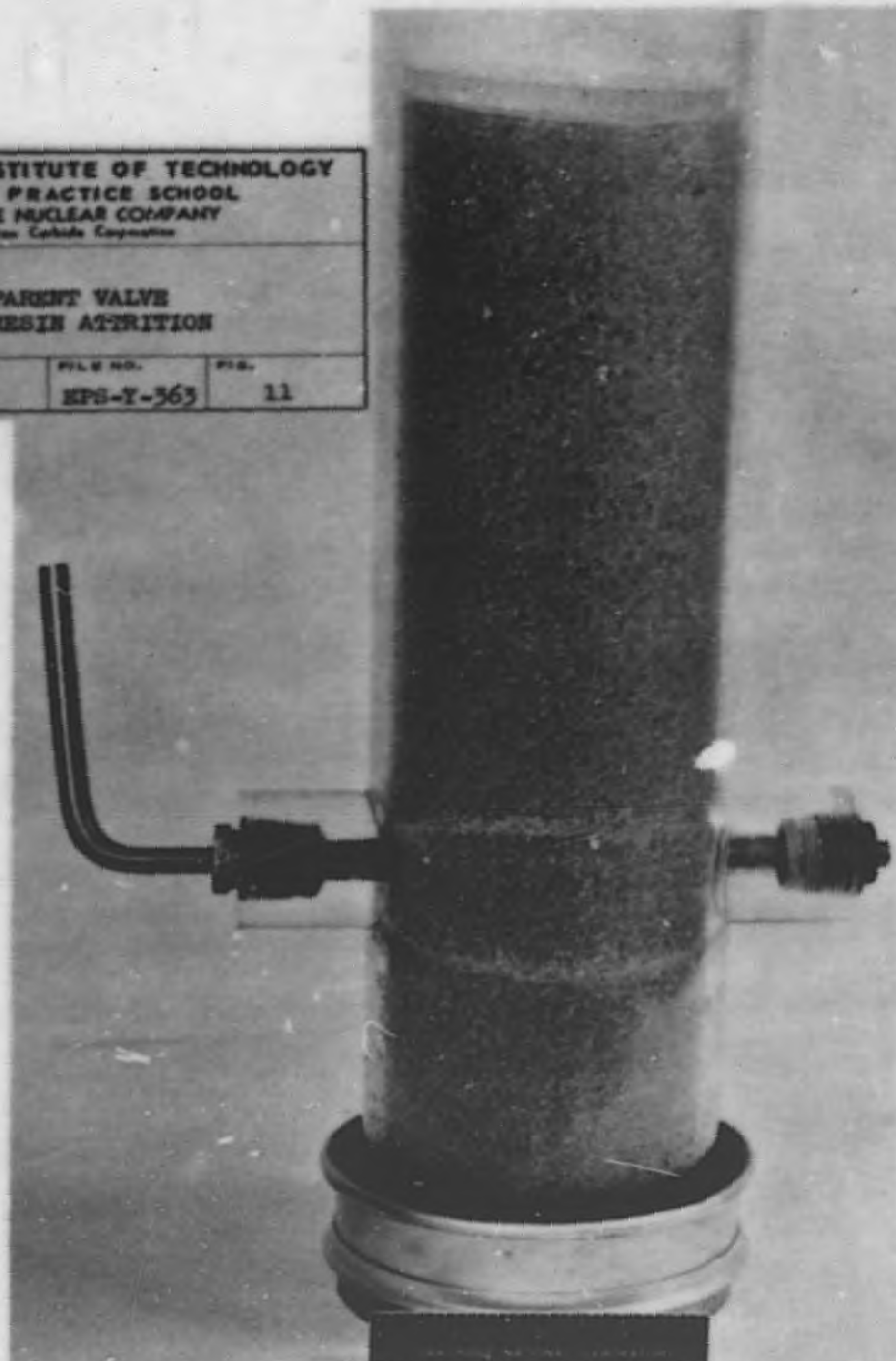
Analysis of the data obtained from the operation of the fifty-foot column (Appendix A, Table V) gives only qualitative results, but does show that attrition is caused both by the friction of the resin against the walls and against itself (during pulsing) and by the operation of the butterfly valve. The breakage caused by the pulsing alone appears to be relatively small compared to that of the other mechanisms of attrition. On the strength of the data mentioned above, as well as the chemical and transparent valve studies, the action of the butterfly valve seems to be the major overall contributor to the resin attrition. The data from the pulsing operation between sampling times 1 and 2 have been discounted as equipment difficulties led to doubt as to what actually took place in the column.

The column, as set up, should be operated over much longer cycle periods for both the pulsing alone and the valve operation. If enough data are gathered, resin attrition may be treated quantitatively and a direct comparison with plant data may be possible. Also, investigation should be made to determine if attrition is decreased by opening and closing the valves during the pulse. The sweeping effects of the water flowing through a narrow annulus may decrease crushing effects of the valve. An alternate test might include injection of water through the column wall and around the periphery of the valve blade when closed.

The production column was originally designed to use plug valves, but butterfly valves were substituted because they were cheaper, easier to procure, and believed to cause no more attrition (7). Also, plug valves have the disadvantage of being greased, and grease in the column could cause operational difficulties. However, a plug valve should be substituted for the butterfly valve in the fifty-foot column to determine the differences, if any, in the attrition caused by the valve action.

The principal criteria for valve selection for use in the column is that the restriction of flow must be kept to a minimum. Only gate-type or plug-type valves fulfill this criteria. Large gate valves are not recommended as they are quite expensive and would probably cause a great deal more resin breakage than the plug or butterfly types.

Another suggestion is that a determination of the force applied to a bead when it is compressed between the edge of the valve blade and the rubber



MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

TRANSPARENT VALVE
SHOWING RESIN ATTRITION

DATE	DRAWN BY	FILE NO.	FIG.
4-9-58		EPS-Y-363	11

gasket (valve closed) should be made. This should shed some light upon the crushing mechanism in the valve (shearing or compression) and upon the amount of attrition caused by the valve action.

Roll and Screen Tests

Neither the roll test nor the screen test in itself is capable of measuring attrition. The former test was devised by Gregory (2) to separate the round particles from the chipped or broken ones. However, during the test, deformed beads and small chips of resin also skid down the smooth glass surface. Hemispheres either rolled on their sharp edge or slid down the plate on their round surface. Some particles were lost to the spherical portion by sliding over the edges of the plate. Thus, considering that only all spherical beads rolled, this test should give a low value for the fraction that were not spherical. Errors in the broken fraction may be as large as -20%.

To improve the meaning and accuracy of the roll test several modifications may be made. First, a surface of less polished material, such as hard rubber, should be used to partially prevent the hemispheres and chips from sliding down the inclined plane. Second, sides of 1/4 inch or higher should be attached to the edges. Third, a one-inch strip of 30-mesh screen across the inclined plane with a bin below it should be placed near the bottom of the plane. This would serve to separate the small fragments, which might slide down the surface, from the large beads.

As the new Dowex resin is essentially all +20 mesh spherical beads, attrition may be measured by the number of fragments produced. Wet screening is one method of measuring the fracture that has occurred by determining the particle size distribution. The major error in this method is that the hemispherical particles would not pass through the 20-mesh screen. A significant fraction of the total attrition results in bead hemispheres (the beads are split in half). Thus, the screen test necessarily results in low values for resin attrition, but does give a distribution of particle sizes.

The resin fractions were measured on a volume basis and the sum of the final volumes used in calculating the per cent resin on each screen. A more accurate measurement may be determined by drying the resin samples from each screen and then calculating the particle size distribution on a weight basis.

CONCLUSIONS

1. Resin attrition is a result of: 1) crushing by the butterfly valve during opening and closing; 2) expansion and contraction of the beads during the chemical cycling; and 3) movement of the resin up and down the column.

2. The butterfly valve is the largest contributor to resin attrition, and friction between the wall and/or resin during bed movement is the smallest.

3. The uranyl sulfate form weakens from cycle to cycle; the nitrate form may retain its structural strength.

4. Bed movement during operation of the butterfly valve is limited to an ellipsoid of resin above and below the valve blade.

5. Resin attrition is increased by an increase in the uranyl sulfate loading.

RECOMMENDATIONS

1. Attrition should be studied on a long-term basis in the fifty-foot column in order to more accurately determine the effects of: 1) valve crushing, 2) friction on resin, 3) weight of resin producing non-uniform force distribution on particles, and 4) forces on particles in the moving bed.

2. The chemical cycling tests should be repeated for a larger number of cycles. A longer unichrome tube would allow higher water velocities during elutriation so that a comparison could be made with plant data. Samples taken should be counted for resin attrition.

3. A study of attrition by butterfly valve which opens and closes during the pulsing should be made. As an alternative, injection of water around the periphery of the closed valve to reduce crushing could be studied.

4. A study of resin movement and attrition in a plug valve could be used to compare its operation with a butterfly valve.

5. The force exerted on a resin bead wedged between the periphery of the closed valve blade and the rubber gasket should be measured.

6. The use of a flying spot scanner for attrition measurement should be investigated.

7. An impact test should be devised to determine the fracture point of beads, which, in column operation, are subjected to various impact forces.

8. Strength tests should be performed on resin beads which have been partially converted to determine if the strength characteristics are affected by the formation of internal stresses in the particles.

9. The data on the strength tests should be expanded so that the data may be treated statistically.

10. The apparatus used in the rolling test should be modified to give more reliable attrition data.

11. Techniques in the screen test procedure could be improved to give more meaningful data.

G. R. Seiler
G. R. Seiler

P. R. Ammann
P. R. Ammann

A. B. Newey
A. B. Newey

APPENDIX

A. Summary of Attrition Data for 50 Foot Column

TABLE V

Sample	% +20	%-20+30	%-30+50	%-50+325	%Rolled	%Not Rolled
3A-O*	92.9	5.1	2.0	--	94.7	5.3
2B-O	93.7	4.1	2.2	--	91.8	8.2
BV-O	96.9	2.1	1.0	--	93.0	7.0
3A-1	92.8	4.0	3.0	0.2	94.7	5.3
2B-1	93.6	3.9	2.4	0.1	90.9	9.1
BV-1	93.7	3.6	2.6	0.2	94.2	5.8
3A-2	94.4	4.0	1.1	0.5	90.7	9.3
2B-2	96.3	2.2	1.0	0.5	92.3	7.7
BV-2	94.5	3.4	2.0	0.1	88.9	11.1
3A-3	80.1	16.4	3.5	--	55.6	44.4
2B-3	95.8	3.1	1.1	--	92.0	8.0
AV-3	97.0	1.7	1.2	0.1	94.9	5.1
BV-3	93.4	4.4	1.1	0.1	94.6	5.4
3A-4	66.2	13.9	19.2	0.7	56.7	43.3
2B-4	95.1	3.7	1.0	0.2	91.8	8.2
AV-4	94.8	2.8	2.1	0.3	91.8	8.2
BV-4	94.5	2.9	2.0	0.6	88.2	11.8
3A-5	81.0	9.9	8.7	0.4	80.5	19.5
2B-5	92.5	5.4	1.6	0.5	92.0	8.0
AV-5	94.3	2.8	2.6	0.3	91.4	8.6
AV'-5	95.0	2.5	2.0	0.5	92.0	8.0
BV-5	91.8	3.7	3.4	1.1	94.9	5.1
BV'-5	91.1	4.1	3.1	1.7	90.1	9.9

*Legend: The first two characters of the sample numbers denote the sample tap (see Figure 7), while the last number denotes the time of sampling as follows:

0 : Before pulsing run No. 1
1 : Between pulsing runs Nos. 1 and 2
2 : After pulsing run No. 2
3 : Before pulsing and valve run No. 1
4 : Between pulsing and valve runs Nos. 1 and 2
5 : After pulsing and valve run No. 2.

B. Location of Original Data

Data were gathered between March 27, 1958, and April 8, 1958, and are recorded on pages 52 through 112 in Unclassified Data Book No. 31, on file at the M.I.T. Engineering Practice School, ORGDP, Union Carbide Nuclear Company, Oak Ridge, Tennessee.

C. Literature Citations

1) Googin, J. M., Personal Communication, Y-12, Union Carbide Nuclear Company, Oak Ridge, Tennessee, March 27, 1958.

2) Gregory, J. F., Personal Communication, Y-12, Union Carbide Nuclear Company, Oak Ridge, Tennessee, April 3, 1958.

3) Higgins, I. R., Personal Communication, ORNL, Union Carbide Nuclear Company, Oak Ridge, Tennessee, March 28, 1958.

4) Kunin, R., and Myers, R. J., "Ion Exchange Resins", 145-147, John Wiley and Sons, Inc., New York, 1950.

5) Levey, R. P., Personal Communication, Y-12, Union Carbide Nuclear Company, Oak Ridge, Tennessee, March 28, 1958.

6) Marrow, G. B., Personal Communication, Y-12, Union Carbide Nuclear Company, Oak Ridge, Tennessee, March 27, 1958.

7) Setter, N. J., Personal Communication, Y-12, Union Carbide Nuclear Company, Oak Ridge, Tennessee, April 7, 1958.

END