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Title: Report on the Testing of Raschig Rings

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Submitted to: This work was performed at the request of V. Majidi of CST-9 and this report documents the results

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REPORT ON THE TESTING OF RASCHIG RINGS

Los Alamos National Laboratory
MST-6, Metallurgy

Loren Jacobson, David Sandoval and Debbie Rodella

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I. Introduction:

Raschig Rings are hollow glass cylinders made from Pyrex glass. They are used in containers for the storage of liquid radioactive waste, and perform two useful functions. The boron content of the glass helps to absorb neutrons from the waste. The volume of the glass rings helps to reduce the local density of the liquid waste and avoid potential criticality problems. Mechanical tests have been performed on such rings in earlier years, involving some nine different styles of testing. (Reference 1) The present work was performed in order to compare the strength of fresh, unexposed rings with rings that had been used in various waste storage containers. In all, ten batches of rings were tested, one batch of unexposed rings and nine batches of exposed rings. The following sections describe the test procedures and the results that were obtained. An appendix contains a series of photographs of the different test methods, and sample failure appearances.

II. Test Procedures:

Raschig Rings are right circular cylinders of Pyrex glass of approximate dimensions 1.5" (38.1 mm) outside diameter, 0.25" (6.35 mm) wall thickness and 1.75" (44.45 mm) high. The load carrying ability of these rings has been evaluated before by several different techniques. (Reference 1) Two of the previously used testing methods (Test Method 7 and Test Method 5) were used in this study, and the test method nomenclature was the same as that used in the earlier work. Test Method 7 involves stacking two rings on their sides within a steel fixture that is 1.65" wide, so that the centers of the cylinders are not vertically aligned. This arrangement is then loaded in compression with a steel block until failure occurs. The test setup is shown schematically in Figure 1. Usually only one of the cylinders failed, but in one case, both cylinders failed simultaneously. Test Method 5, Figure 2, required a fixture that held one raschig ring with its central axis tilted at a 45 degree angle. Then a second cylinder was placed on top of the first, with its central axis vertical, and roughly aligned with the topmost point of the bottom cylinder. The arrangement was then loaded in compression until one of the cylinders failed, usually the top cylinder. The loading was performed with an MTS hydraulic test system using a stroke rate of about 0.008" per minute. The load, stroke and time were recorded in a data file that was timed to write a data point every 0.5 second. A running maximum load indicator was used to record the actual maximum failure load of the ring assembly.

Ten different batches of rings were tested One batch was unexposed, i.e. had not been exposed to the radioactive waste environment. The other nine batches had been exposed. The conditions of exposure were not given. The batches of exposed specimens are designated by the numbers that were given with each batch.

Table I gives all of the test results. The columns labeled Test Method 7 and Test Method 5 record the failure load in pounds, and then in kilograms using the conversion 1 lb. = 0.454 kg. Three tests were conducted for each condition. The right two columns are results of seven additional tests of seven sets of samples using the Test Method 7. A total of ten results for unexposed specimens are compared with ten results from one of the batches that appeared to be strongly affected by exposure, batch 4743-1. In addition to the average and standard deviation for these sets, a Weibull analysis was performed on each data set, although the sample size was too small in number for truly valid Weibull parameters.

III. Discussion:

Three fracture tests of a brittle material are not sufficient for drawing statistically significant conclusions. A preliminary examination of the results showed that some samples in the exposed population fell either above or below the range of unexposed test results. For Test Method 7, the entire10 specimen unexposed population was used. Failure loads varied from 425 kg. to 749 kg. For Test Method 5, only the range of three values was used for the unexposed population, 406 to 525 kg. Table II summarizes the results of this preliminary examination. This Table shows that the only batch of exposed specimens that was consistently low was batch 4743-1, which had two Test Method 7 test results and two Test Method 5 test results that fell below the range of unexposed specimens for these test types. It was this factor that led to the selection of batch 4743-1 for additional Test Method 7 tests, to generate a total of 10 data points.

The Weibull Distribution Function has been used for many years to determine the nature of a statistical distribution of fracture strengths of brittle materials. This distribution function has two independent parameters, allowing it to be fit to skewed or truncated distributions. For brittle fracture strength data, the failure stress values are typically used. However, for this data the failure load data was used, because the irregularities among the test specimens and the nature of loading of two samples at a time, precluded the determination of actual failure stress values. The distribution function is given by the equation below:

$$P_f = n/(N+1) = 1 - \exp - [\frac{(L_n - L_0)}{L_u}]^m$$

In this equation P_f is the probability of failure, given by the order number, n, of the load value in a list of failure loads ordered from lowest value to highest value. N is the total number of specimens. P_f is then related to the failure load of the n-th specimen, L_n, by the relationship on the right side of the equation. In this relationship, L_0 is the "zero strength", one of the independent parameters that must be determined for the load distribution. L_u is a normalizing factor and m is the other independent parameter which is a measure of the spread of the data. The double logarithm is taken of this expression, and the Log Log of P_f is plotted versus the Log of the quantity (L_n - L_0). L_0 is adjusted until the best straight line is obtained, giving the value of L_0, and then m is the slope of this line.

Figures 3. and 4. show the Weibull plots for the Unexposed and the Exposed specimens. It was found that an L_0 value of 0 gave the best straight line for the Unexposed specimens, and an L_0 value of 100 kg. gave the best straight line for the Exposed specimens. Since the magnitude of L_0 is the load value below which the probability of failure is equal to 0, it could be inferred that the exposed specimens are "stronger" than the unexposed specimens, at the low end of the specimen population. However, it would be more correct to infer nothing at all from this value; it is only a parameter that is adjusted to give a better fit to the measured values. The m values for each of these groupings of specimens were 5.6 and 2.44, respectively. The narrowness of the distribution, as indicated by the m value, is consistent with the more conventional statistical quantity of standard deviation, which is 98.73 for the unexposed specimens and 135.05 for the exposed group, batch 4743-1. The average strengths are 622.4 for the unexposed samples and 442.7 for the exposed samples. It is believed that the average values represent a significant difference between the populations, since the unexposed average minus the unexposed standard deviation, 523.7, is still greater than the exposed average, and the exposed average plus the exposed standard deviation, 577.7, is below the unexposed average.

Comparison with the previous study results for Test Method 7 (Reference 1), shows that the average for unexposed specimens in this study, 622 kg., is considerably higher than previously reported, 462 kg. The prior result for standard deviation was higher as well, 155 kg, compared to 99 kg in the present study. The load values given in the previous study were obtained from only two or three tests, and original data was not presented. The average value of the failure load for the three unexposed rings tested in Test Method 5 was 501 kg. for this study. This value compares favorably with the average value of all Test Method 5 results of the previous study, 504 kg. This may reflect the greater severity of the loading arrangement used in Test Method 5, but such comparison may not be justified, based on the small number of specimens tested.

The ten unexposed specimen failure load values makes up an interesting population, because it represents the loads to failure of only ten specimens. Some of the specimens were tested more than one time, because during each test, only one of the two specimens failed, and the other specimen could be used in one or more additional tests. Also, two of the specimens were tested alone in order to obtain a failure load. This was done in order to economize on specimen usage, but would probably bear repeating if the statistics of only the stacked ring failure were of interest. Table III lists the results of the ten tests, also indicating which specimens were tested together, and which specimen failed when two rings were tested. Note that specimens 5 and 10 were tested alone, this being the third time of loading for specimen 5 and the second loading for specimen 10. Specimen 10 had experienced a previous higher load, whereas specimen 5 had not. The single failure loads fall within the overall range for the ten tests. Specimen 1 was also loaded three times, failing the third time at a load level that was less than the one it had previously experienced, but at a higher load than it experienced in its first test. While obvious damage to the surviving specimens was not apparent after one load application, it might be inferred from these results that some damage to the ring could occur during prior loading, which does not lead to catastrophic failure.

A final category of observations on the failure of exposed versus unexposed specimens must be remarked upon. All of the unexposed specimens failed by shattering into numerous pieces, too numerous to count. However, the exposed specimens were quite variable in the number of pieces following failure. Table II indicates in parentheses the number of pieces into which the failed ring was divided, Small, Medium or Large. Quantitatively, Small corresponds to less than 10 pieces, Medium corresponds to 10-20 pieces and Large is greater than 20 pieces. Failure into a small number of pieces suggests that the exposure conditions caused some damage to the rings and could have led to a small number of internal cracks. Note that there is no consistent correlation between the observation of a small number of pieces and lower failure load.

It is important to note that these results refer to two approximate conditions of Raschig ring loading that might be experienced in service. The test results do not, nor were they intended to, reflect actual material properties that could be associated with the borosilicate glass. The failure loads appear to be high enough to far exceed the load imposed on any ring due to the weight of other rings that would be loaded into a waste container, for both the unexposed and exposed rings. Thus, there would appear to be no damage due to exposure that could lead to undesirable failure of Raschig rings during service. Of course, this situation may not apply to dynamic loading conditions that might be associated with container transport operations. Dynamic loading could lead to lower measured failure loads, but this consideration was not part of this study, and would require further work.

IV. Conclusions:

Based on only three tests per condition, it is very difficult to draw any clear conclusions about the effect of exposure on the load bearing capability of Raschig rings.

Results of this study generally agree with the results of the previous study for Test Method 5, but give higher values of load bearing capability for Test Method 7.

Expanding the test population to 10 specimens for the unexposed rings, and to 10 specimens for one of the batches of exposed rings allowed treatment by the Weibull distribution. The results clearly indicate that these two populations are different. The fracture load values for the exposed batch that was selected for greater numbers of tests (4743-1), were lower and more variable compared to the values for the unexposed rings.

More quantitative conclusions cannot be drawn without additional testing to enlarge the data sets to at least 10 samples for all conditions. For the best statistically meaningful results, a batch size of about 30 is recommended.

V. References:

Reference 1. Rothe, R. E., "The Mechanical Strength of Borosilicate Glass Raschig Rings", Unidentified Rocky Flats Report

TABLE I RESULTS

Raschig Ring Failure Load Test Results

Unexposed		Test Method 7 lb.	kg.	Test Method 5 lb.	kg.	unexposed Type 7, kg.		exposed Type 7, kg.
1		1054.3	478.6522	895	406.33	425		247
2		935.4	424.6716	1261	572.494	479		321
3		1295.6	588.2024	1156.2	524.9148	588		341
						609		331
						615		430
						670		453
						671		465
						688		513
						730		620
						749		706
						622.4	Average	442.7
						98.73	Std. Dev.	135.05
Exposed								(4743-1)
0476-1		900.4	408.7816	1014	460.356			
	2	482.4	219.0096	1333.8	605.5452			
	3	1450.6	658.5724	1026.1	465.8494			
4694-1		1154.4	524.0976	967.4	439.1996			
	2	1996.7	906.5018	851.7	386.6718			
	3	1652.5	750.235	1018.2	462.2628			
4665-1		1114.6	506.0284	1164.3	528.5922			
	2	1546.2	701.9748	1081.8	491.1372			
	3	1827	829.458	1106.8	502.4872			
4658-1		807.2	366.4688	962.3	436.8842			
	2	485.1	220.2354	1135.9	515.6986			
	3	709.4	322.0676	937.5	425.625			
4700-1		1627.7	738.9758	1047.2	475.4288			
	2	994	451.276	716.3	325.2002			
	3	1999.8	907.9092	931.8	423.0372			
4773-1		643.4	292.1036	1196.9	543.3926			
	2	1832.5	831.955	1078.3	489.5482			
	3	1227	557.058	1094.7	496.9938			
4668-1		1164	528.456	973.5	441.969			
	2	1494.9	678.6846	781.6	354.8464			
	3	1918.8	871.1352	832	377.728			
4674-1		929.4	421.9476	907.6	412.0504			
	2	1065.6	483.7824	1490.1	676.5054			
	3	557.3	253.0142	936.4	425.1256			
4743-1		949	430.846	675.6	306.7224			
	2	544	246.976	886.6	402.5164			
	3	728.2	330.6028	998.6	453.3644			

TABLE II Results Summary

Batch Number, Exposed	Type 7 Test Results Compared to Unexposed Range	Number of Failed Pieces	Type 5 Test Results Compared to Unexposed Range	Number of Failed Pieces
0476-1	2 low	(Small)	1 high	(Small)
4694-1	1 high	(Small)	1 low	(Large)
4665-1	1 high	(Sm-Med)	3 in range	(Med-Lg)
4658-1	3 low	(Large)	3 in range	(Large)
4700-1	1 high	(Sm-Med)	1 low	(Medium)
4773-1	1 low,1 high	(Sm-Med)	3 in range	(Large)
4668.1	1 high	(Sm-Lg)	2 low	(Small)
4674-1	2 low	(Large)	1 high	(Large)
4743-1	2 low	(Sm-Lg)	2 low	(Sm-Lg)

TABLE III Unexposed Specimens, Identified

Samples Tested	Load, lb.	Load, kg	failed
1-2	1054.3	478.6522	2
3-4	935.4	424.6716	4
5-6	1295.6	588.2024	6
1-3	1477.3	670.6942	3
1-5	1340.6	608.6324	1
5 (single ring)	1474.9	669.6046	5
7-8	1649.9	749.0546	7
8-9	1515.2	687.9008	8
9-10	1608.2	730.1228	9
10 (single ring)	1354.5	614.943	10

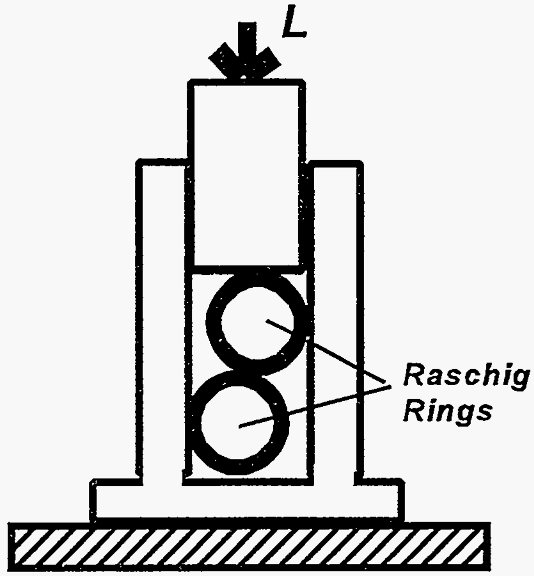


Figure 1. Raschig Ring Test Method 7.
Load is applied until failure occurs in one of the rings.

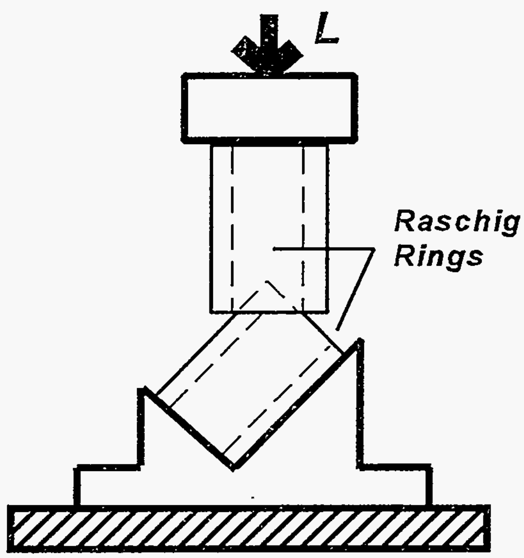


Figure 2. Raschig Ring Test Method 5.
Load is applied until failure occurs in one of the rings.

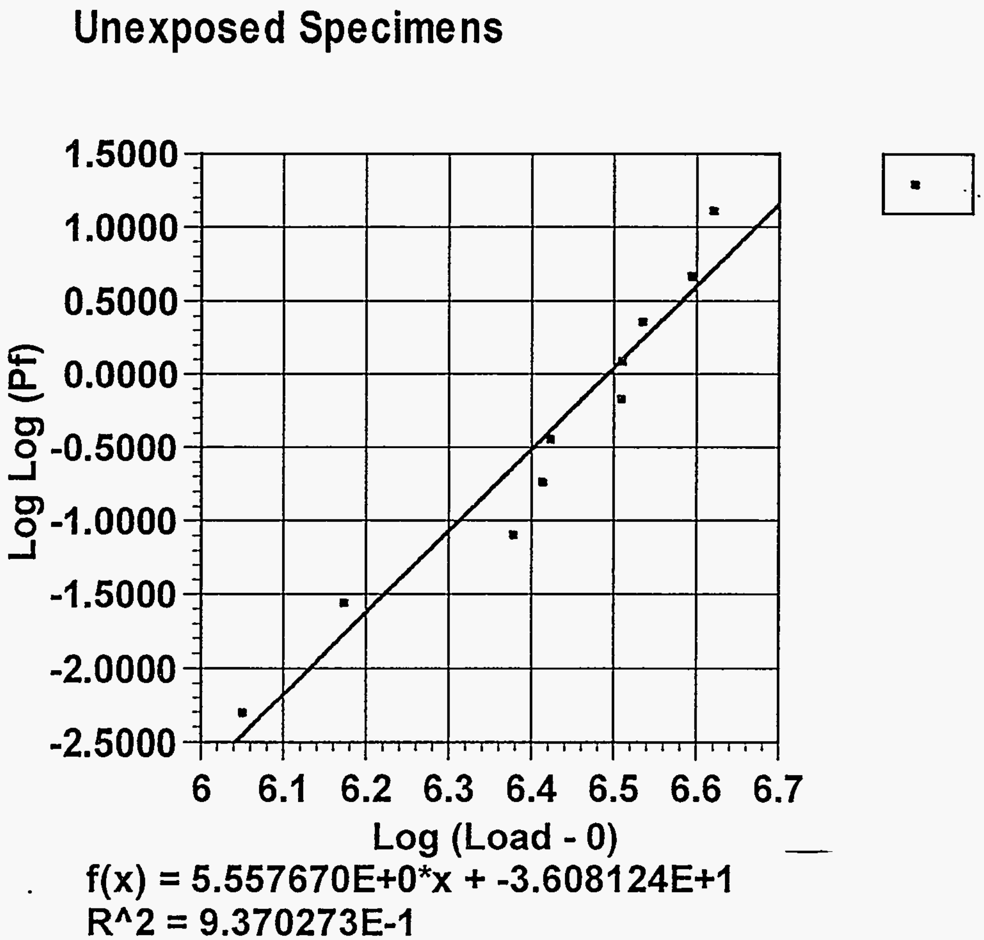


Figure 3. Weibull Plot for Unexposed Specimens, m = 5.6, Lo = 0

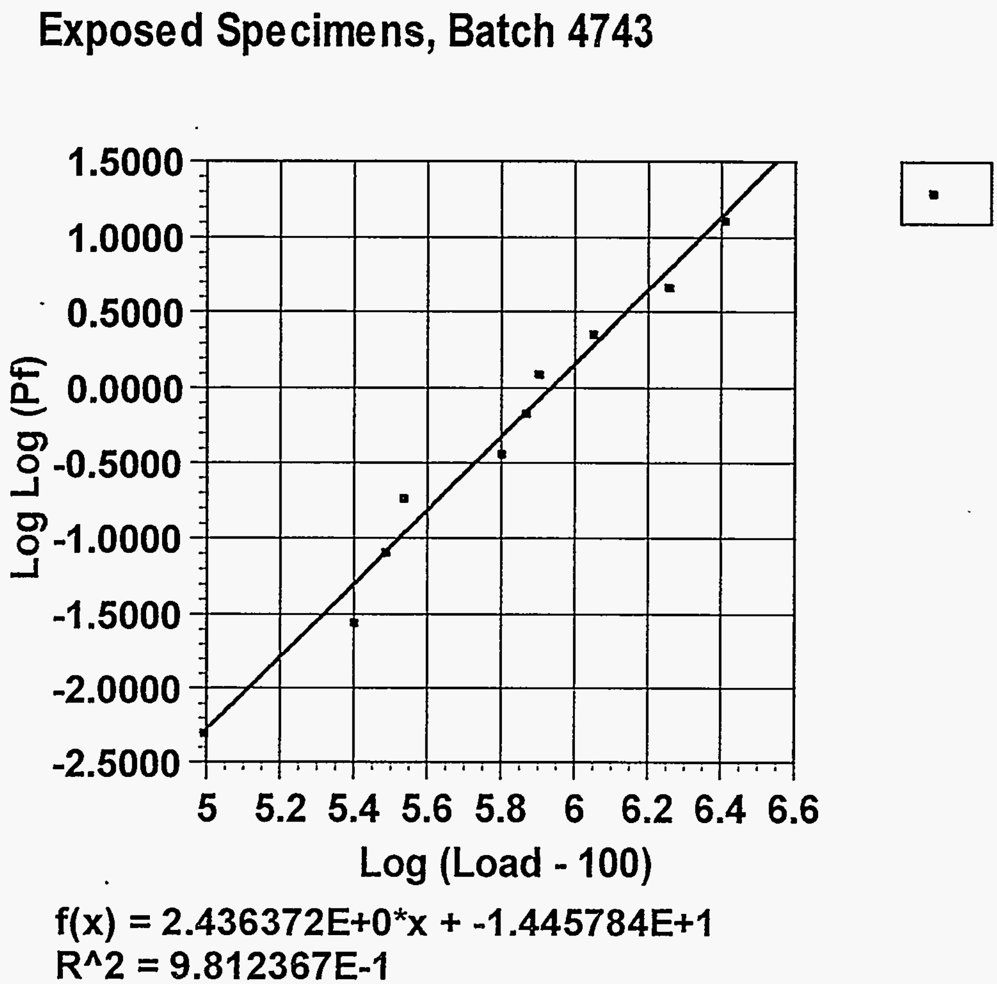
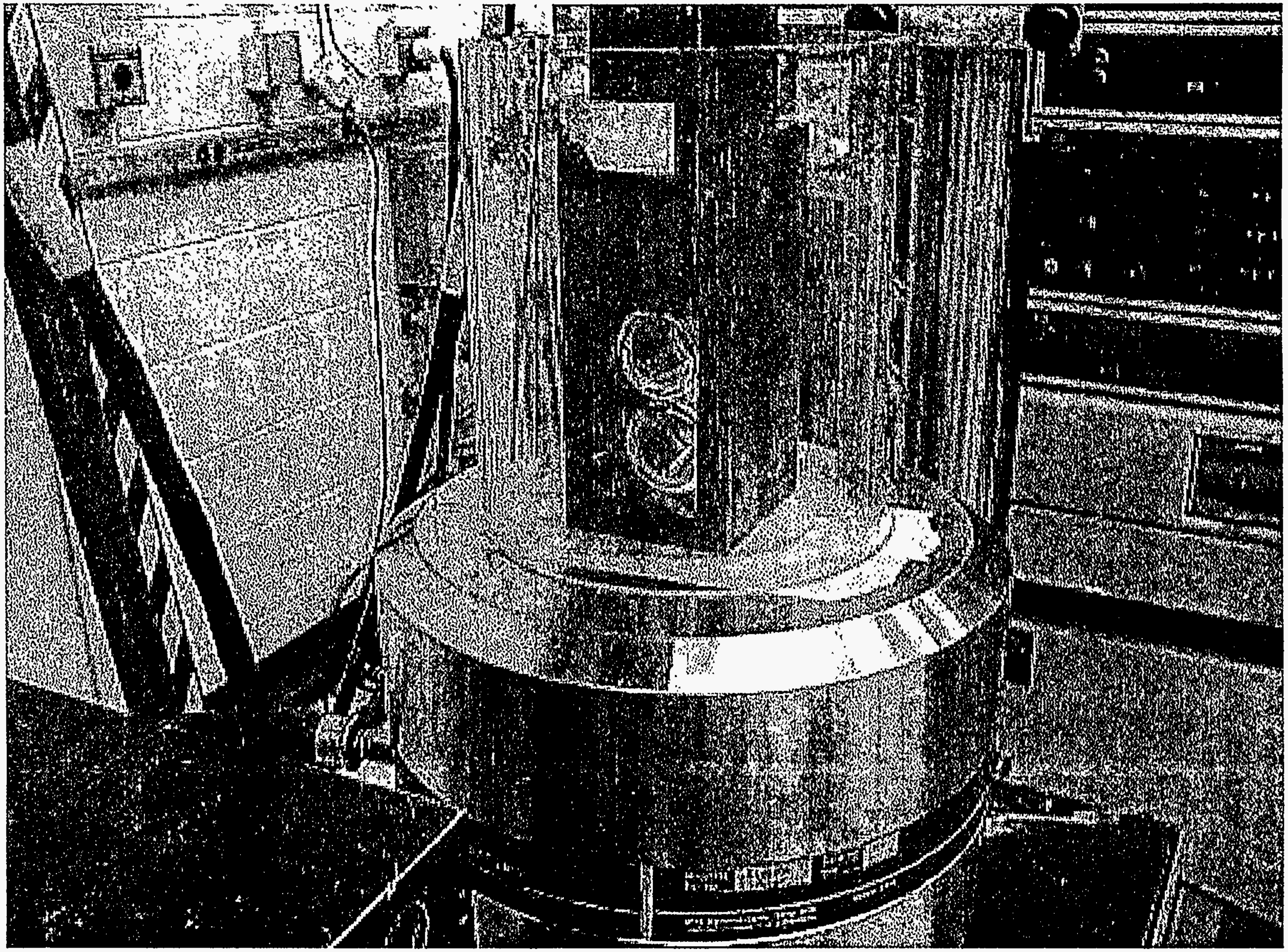


Figure 4. Weibull Plot for Exposed Specimens, m = 2.44, Lo = 100 kg.

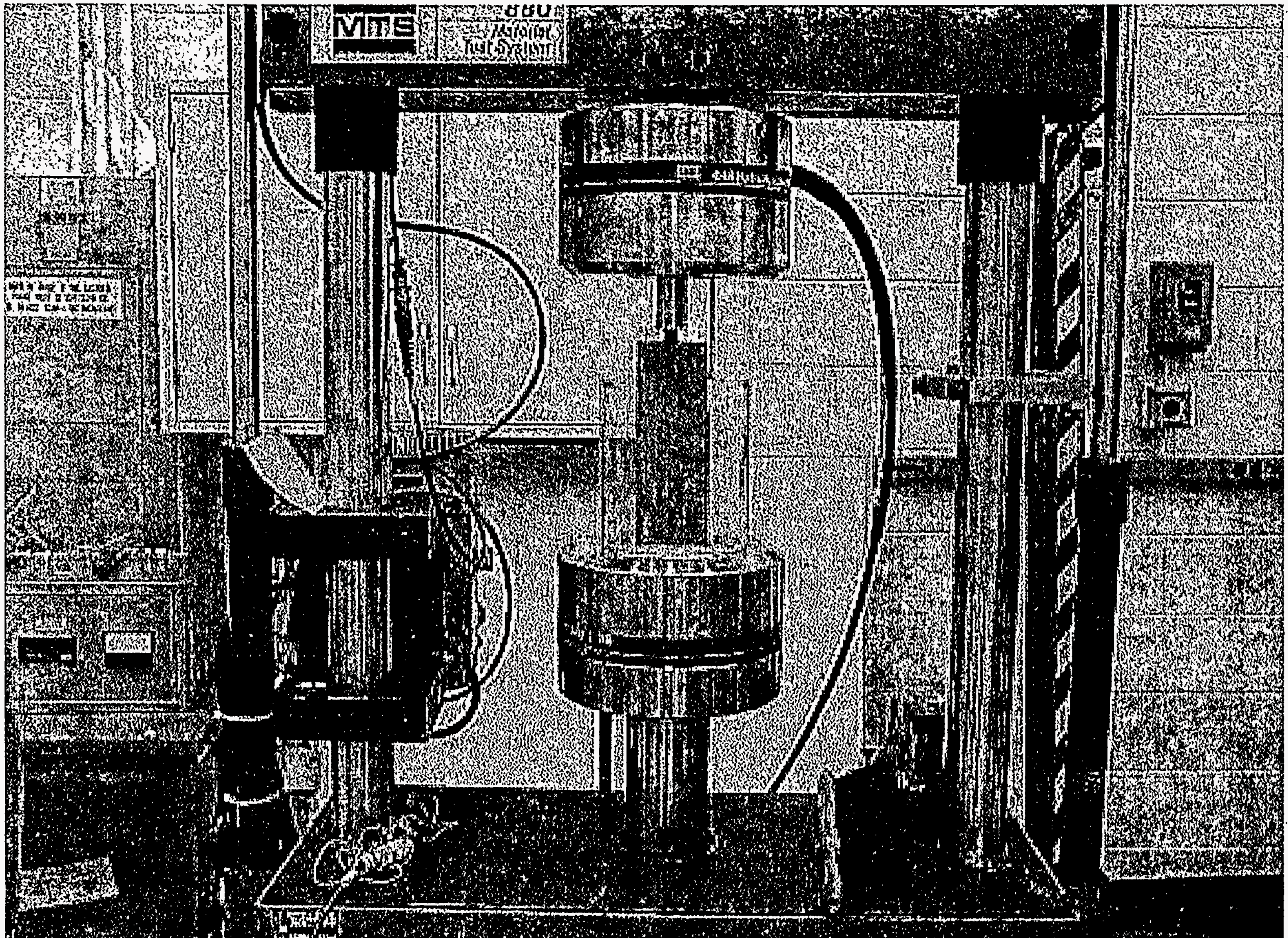
APPENDIX I.

Photos of Various Test Methods as Requested

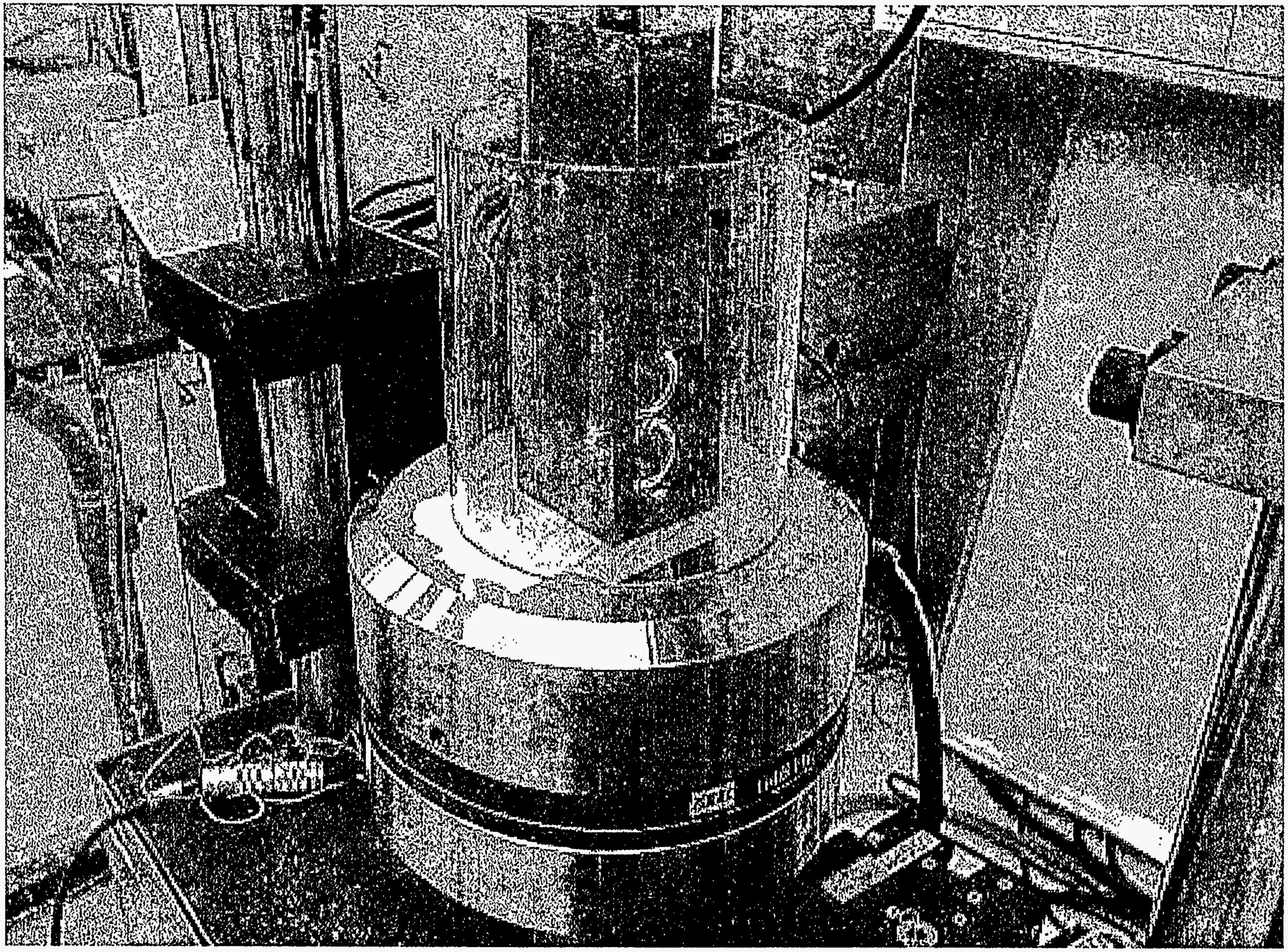
Test 1 (Samples 1and 2: G0476 D-16 Drum#4)



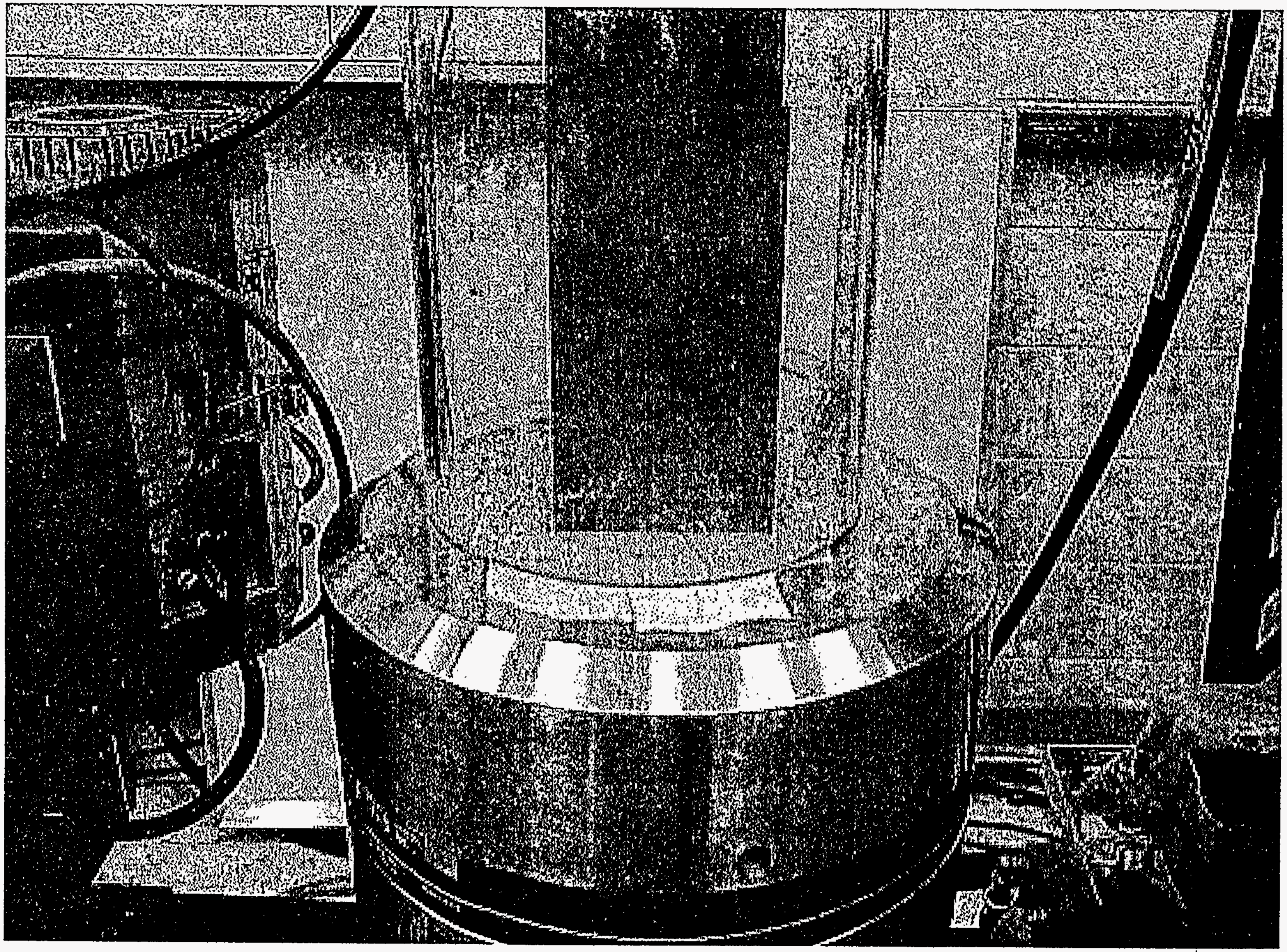
Test #1 (Front View)



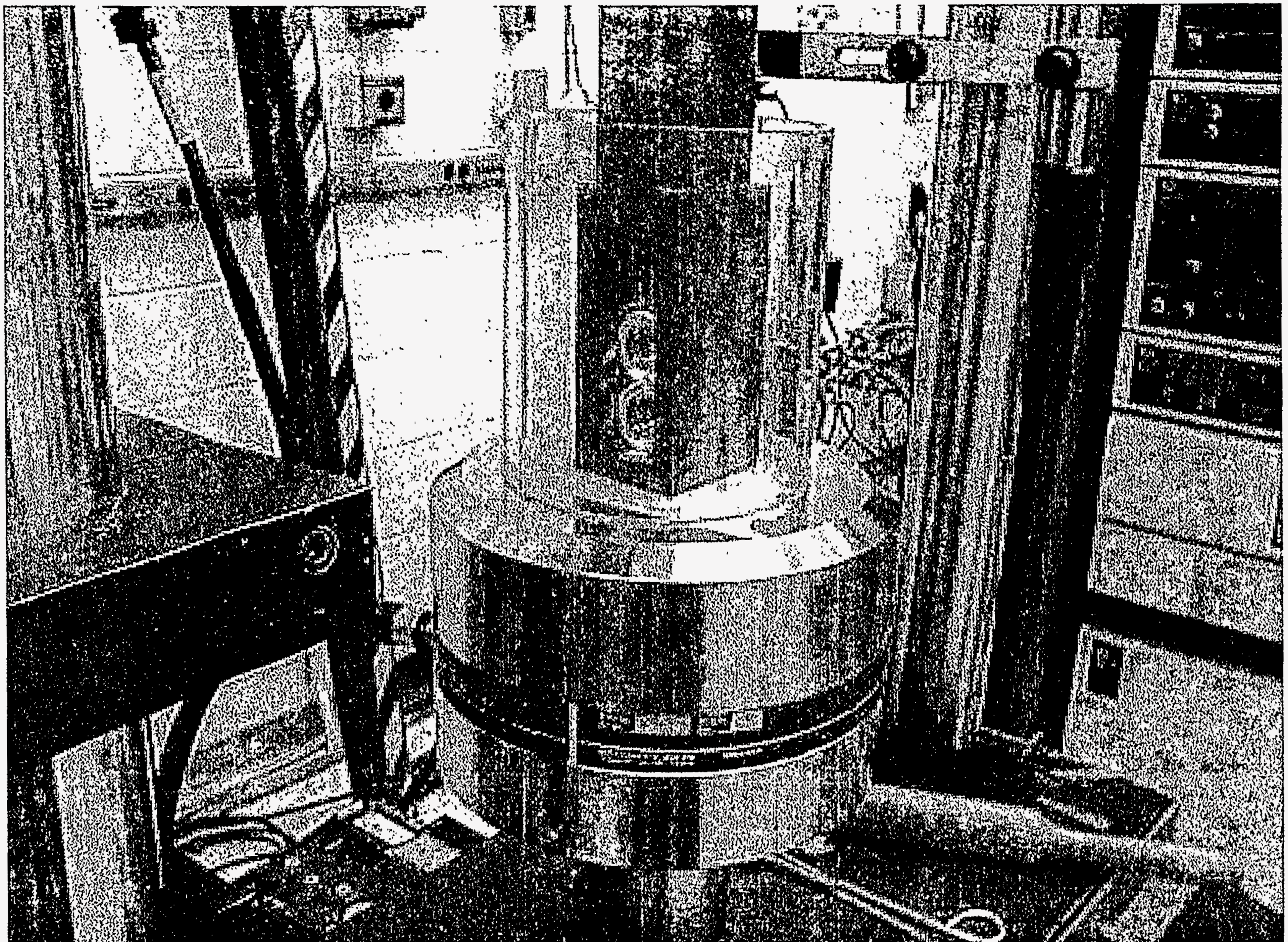
Test #1 (Side View)



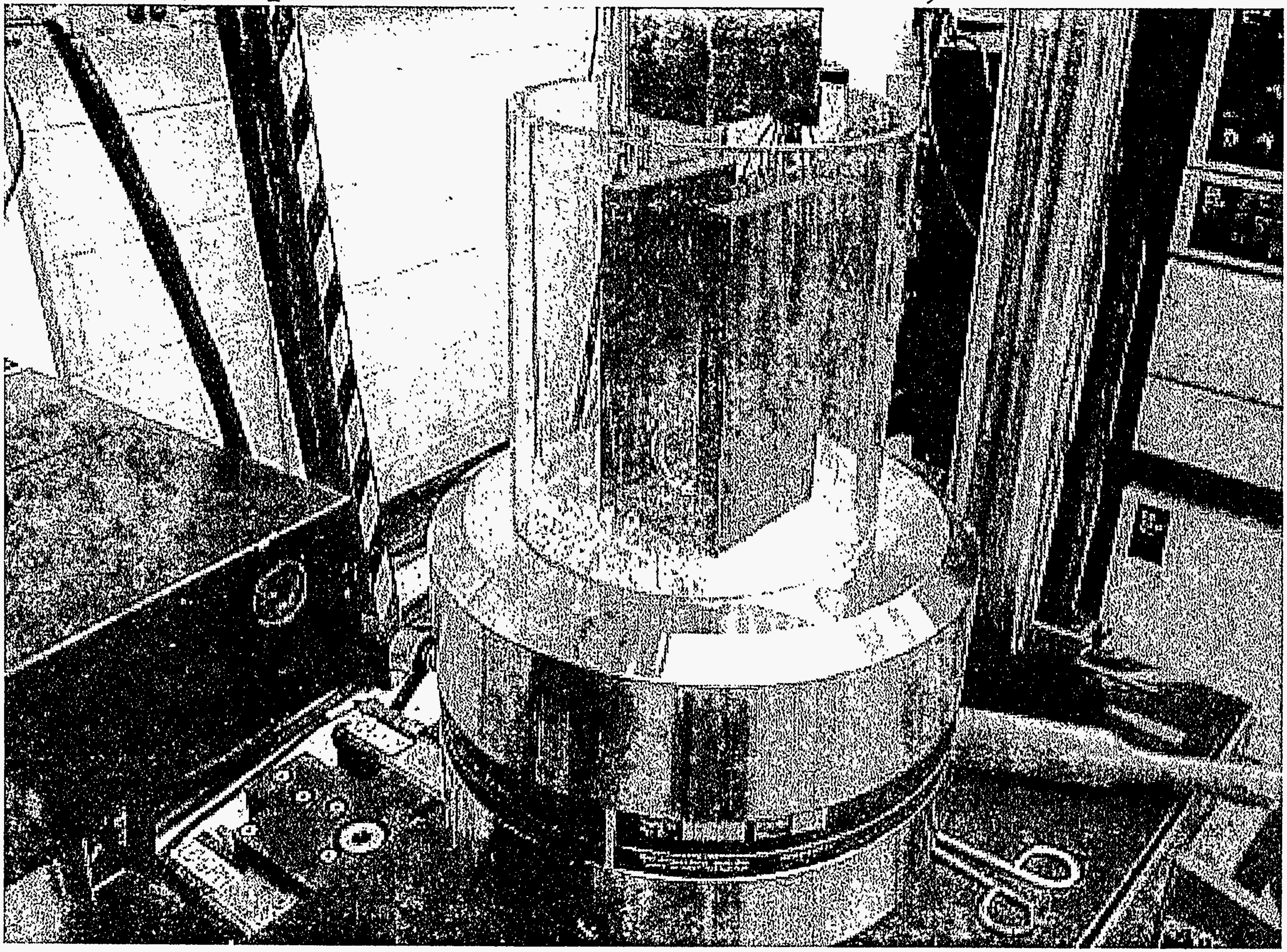
Test #1 (Samples #1 and 2:G0476 D-16 Drum#4)



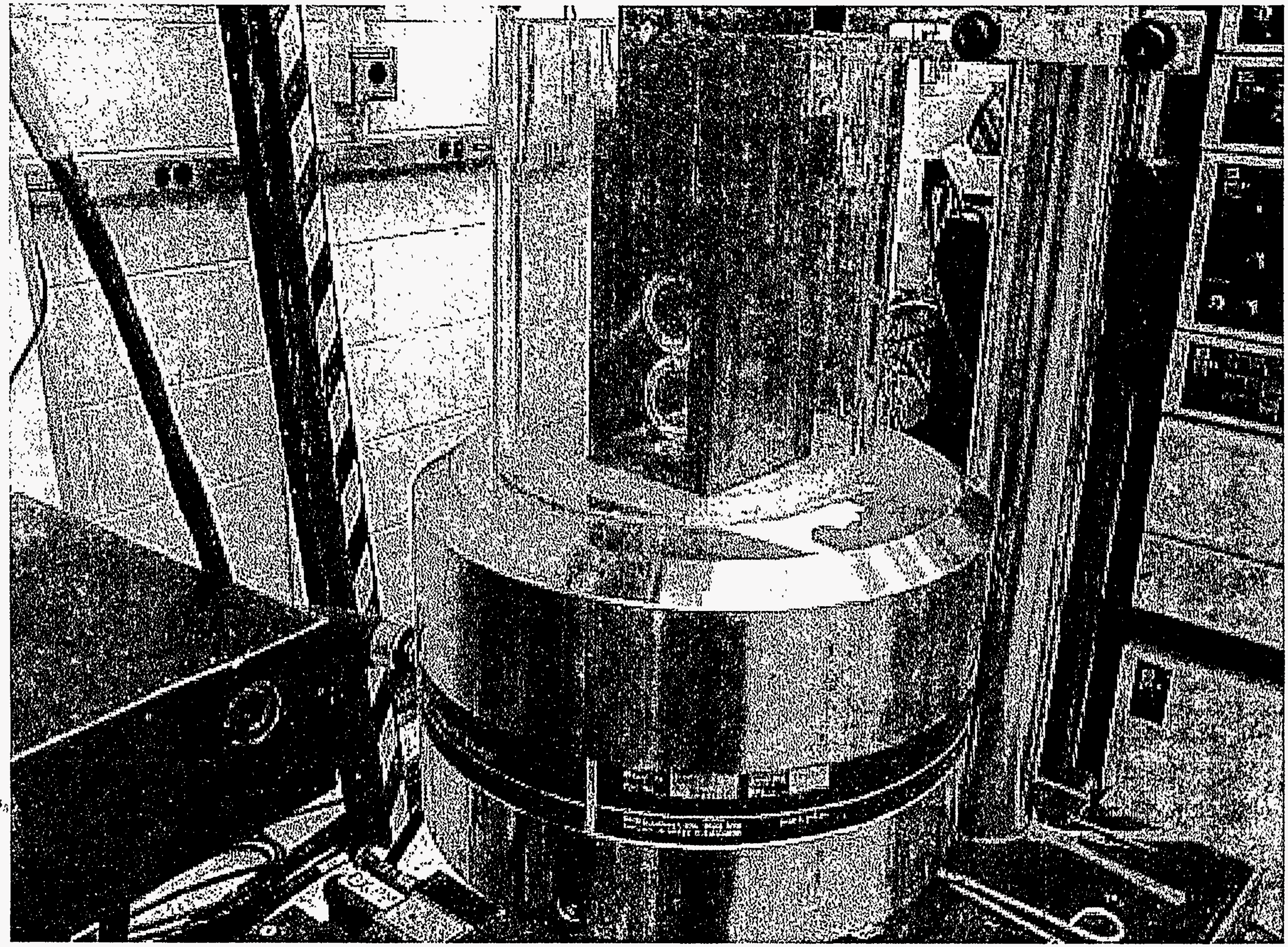
Test #4 (Samples #7 and 8: D046 D-48 Drum#4)



Test #4 (Samples #7 and 8: D046 D-48 Drum#4)



Test #7 (Samples #13 and 14:Drum #4 D04665 D-65)



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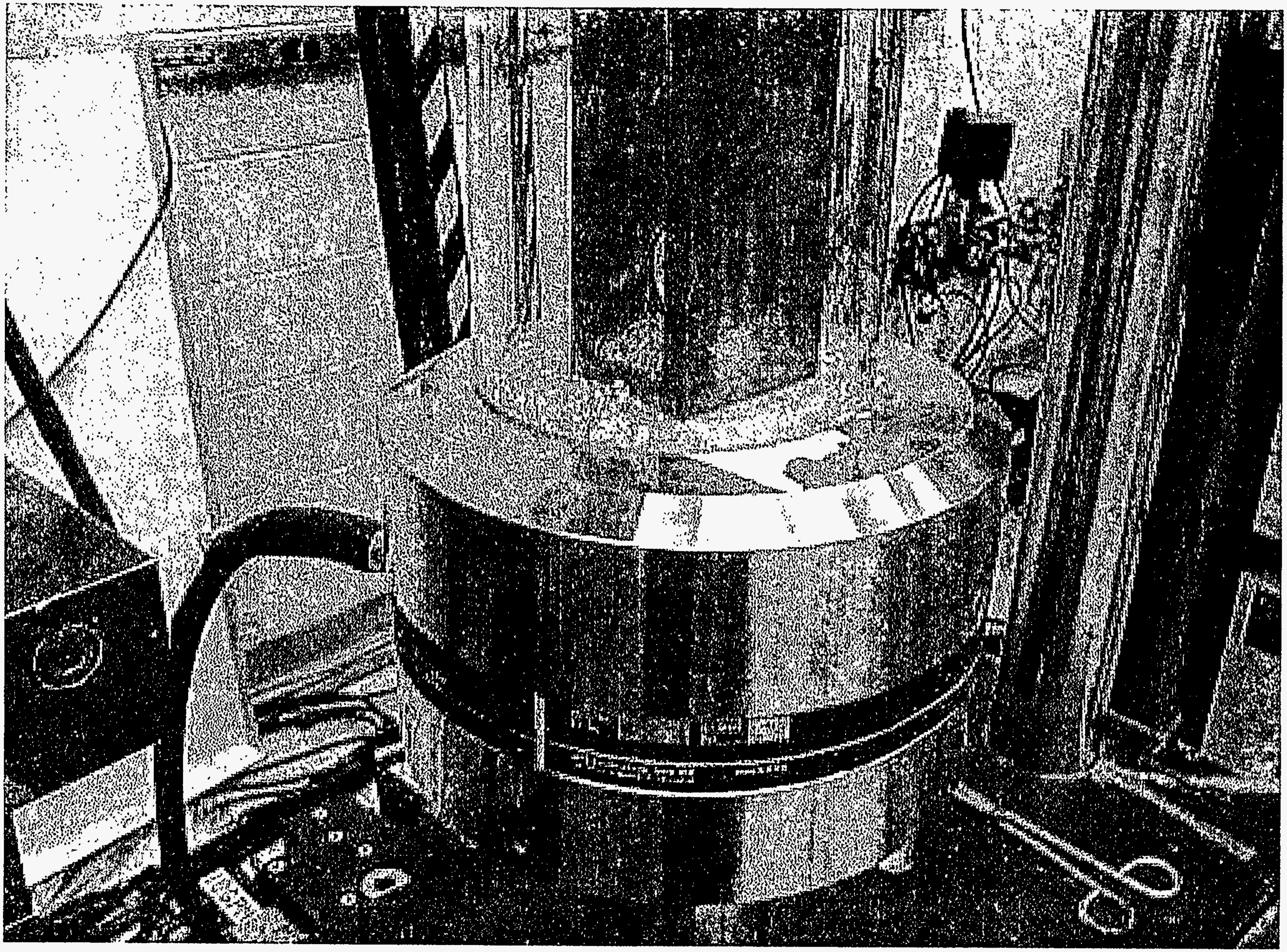
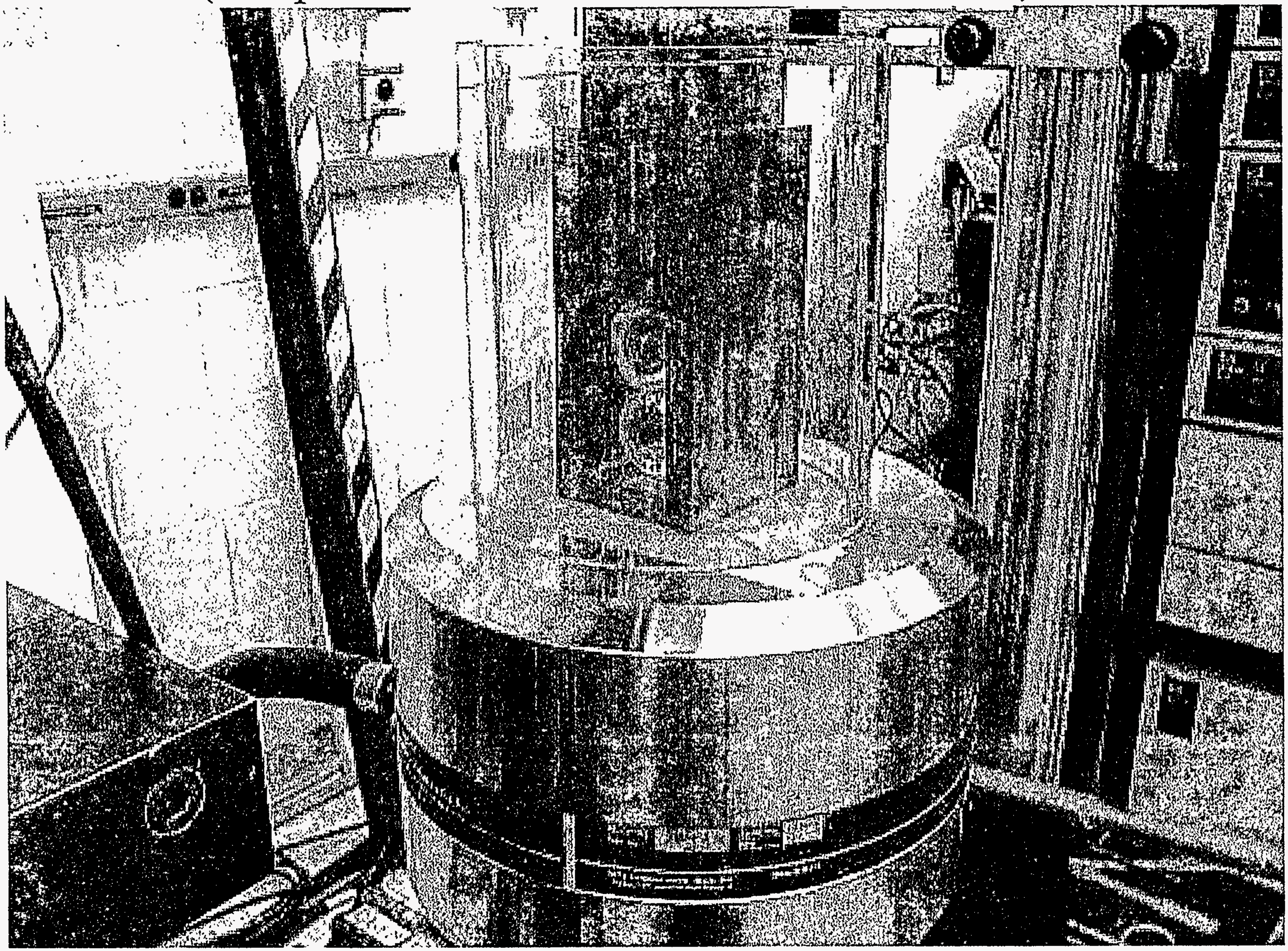
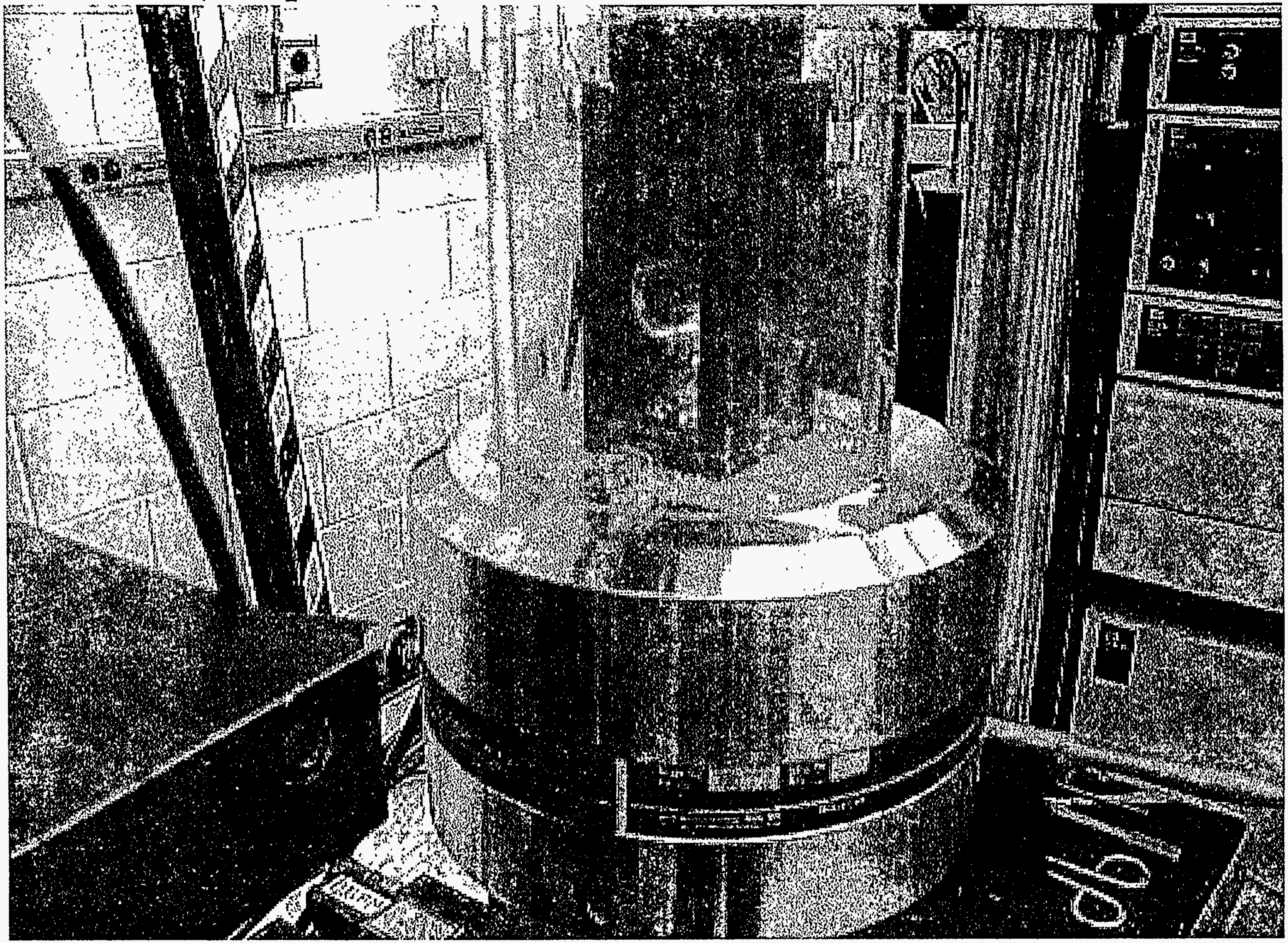


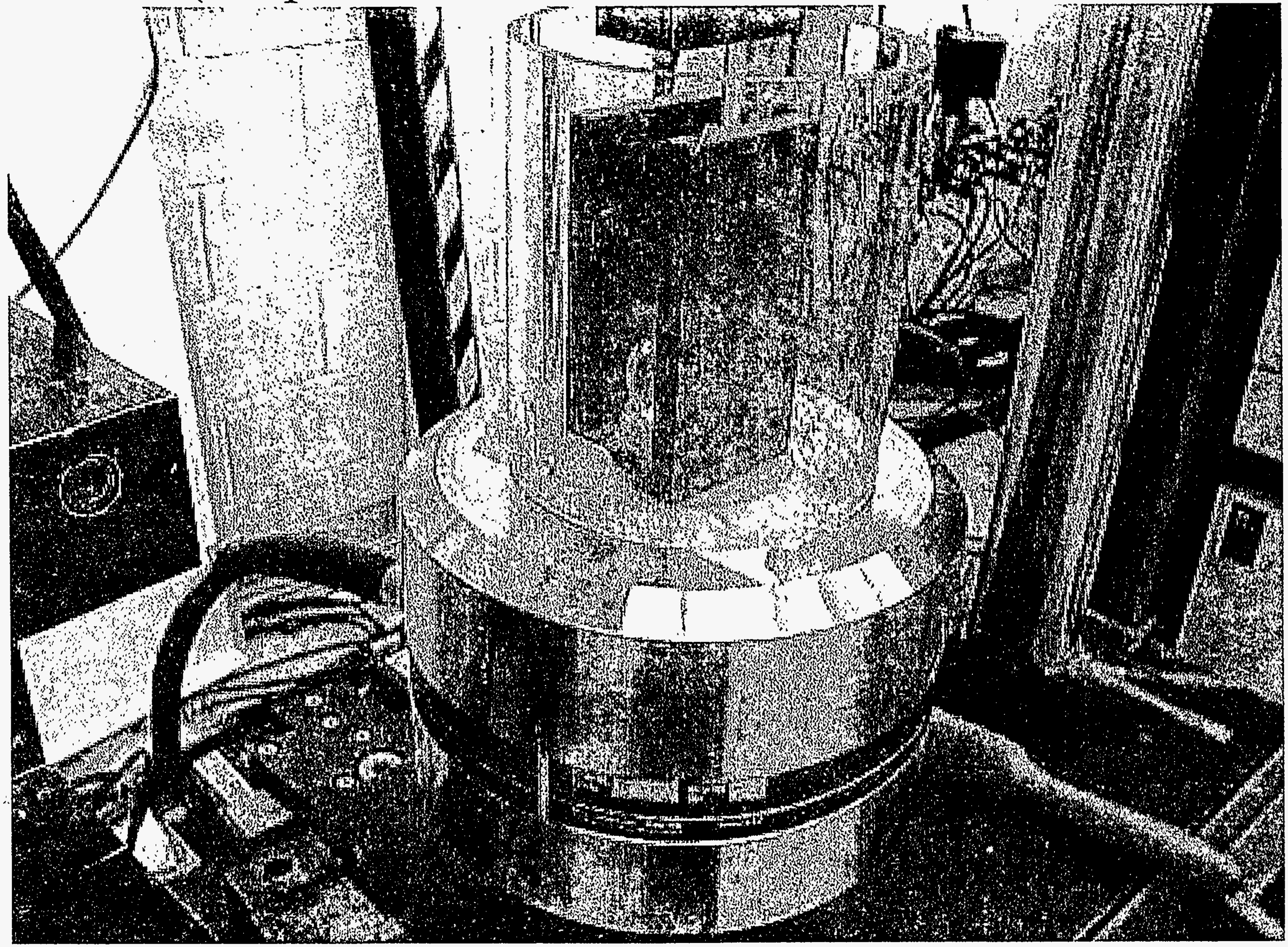
Test # 10 (Samples #19 and 20:Drum #4 D99 G04658)



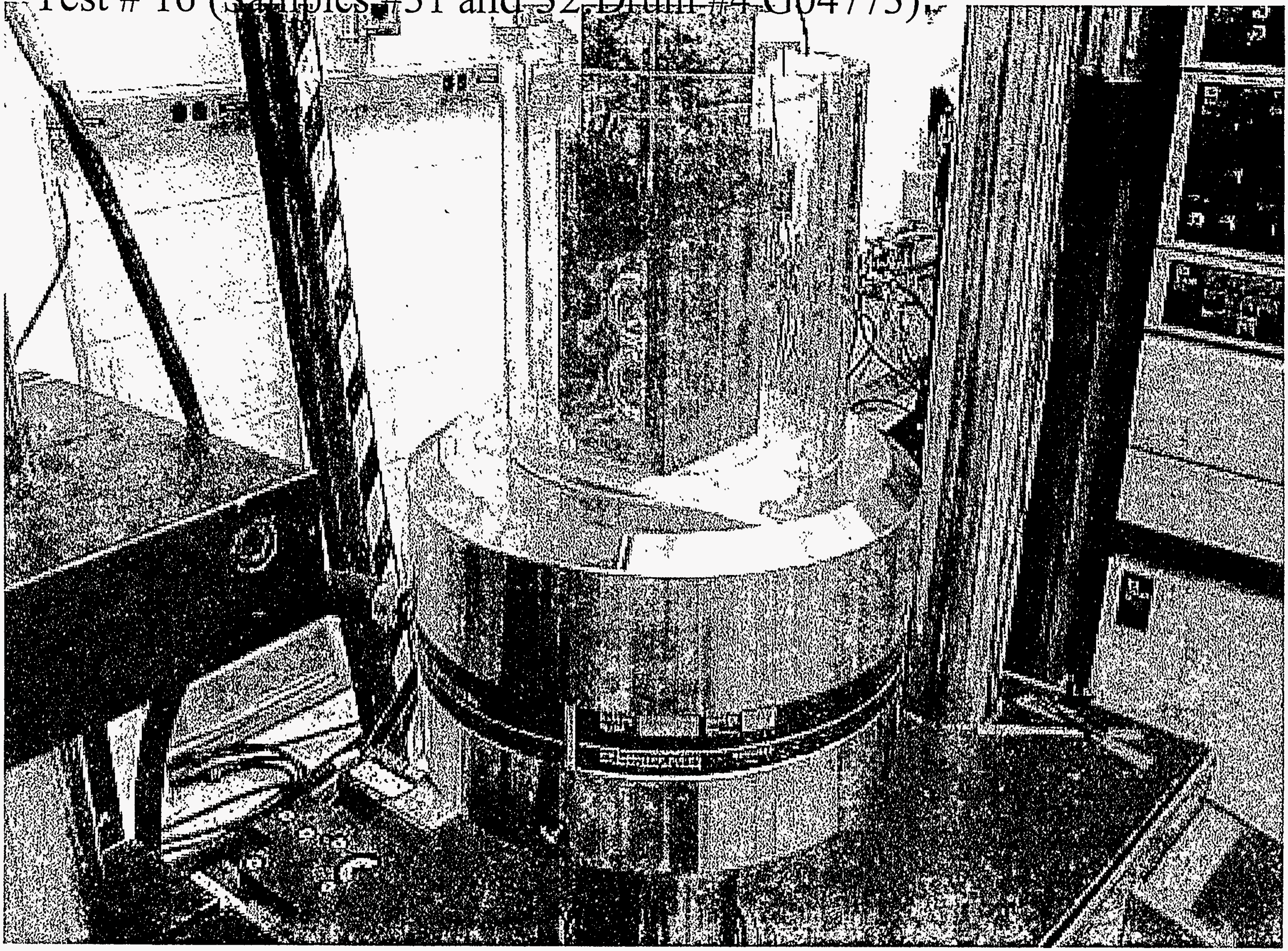
Test #10 (Samples #19 and 20:Drum #4 D99 G04658)



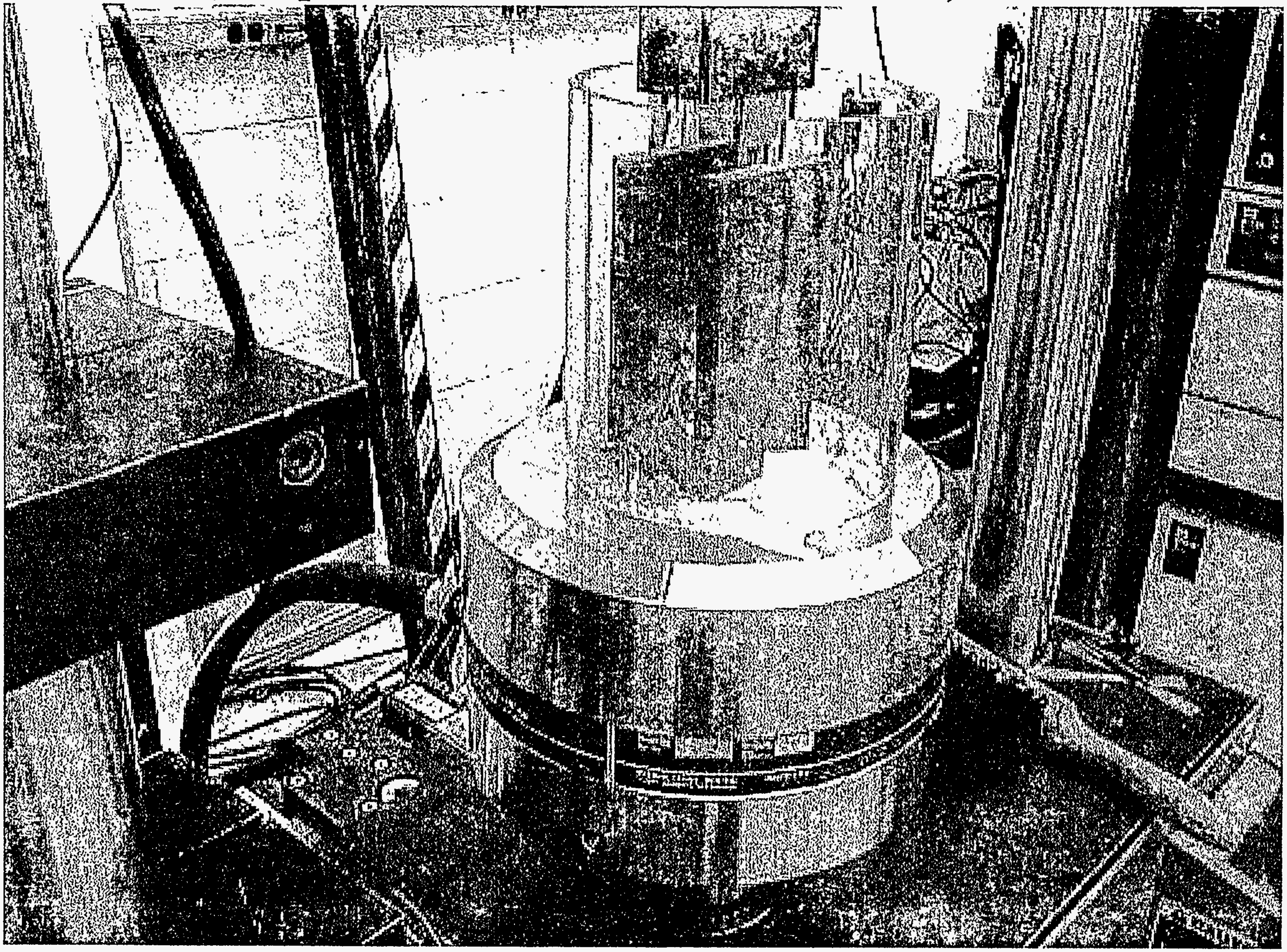
Test # 13 (Samples #25 and 26:Drum#4 D58 G0470)



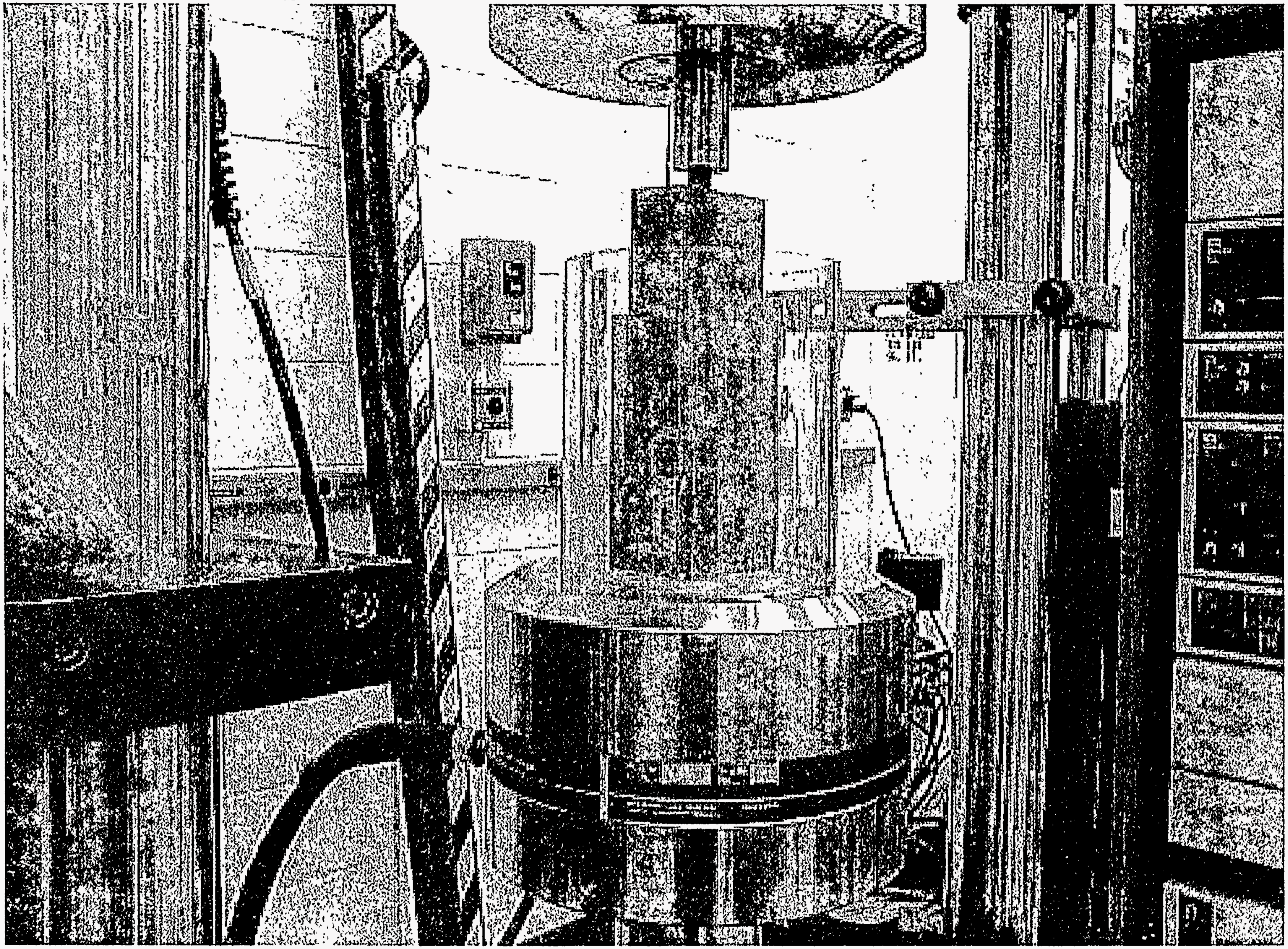
Test # 16 (Samples #31 and 32:Drum #4 G04773).



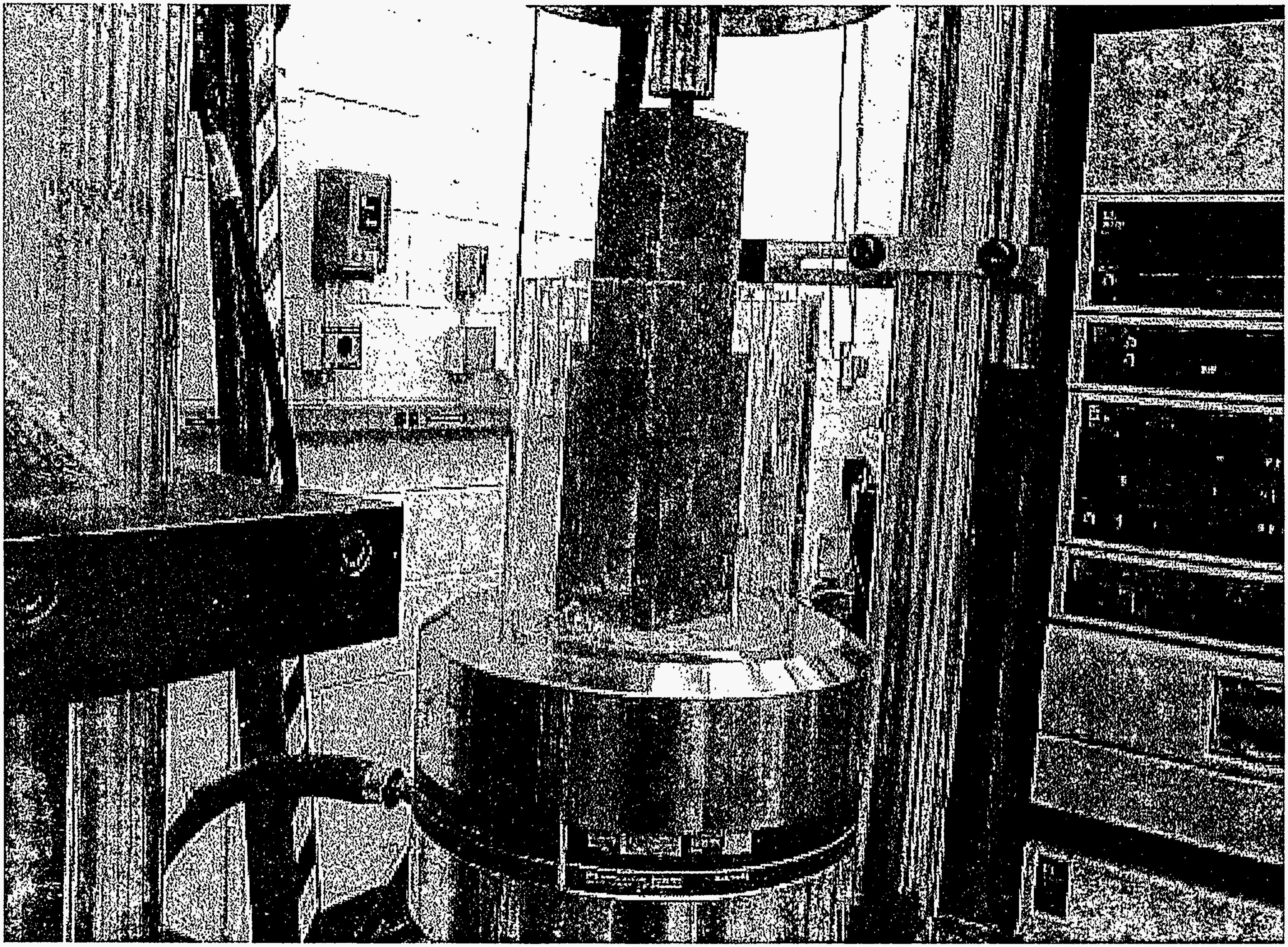
Test # 16 (Samples # 31 and 32:Drum #4 G04773)



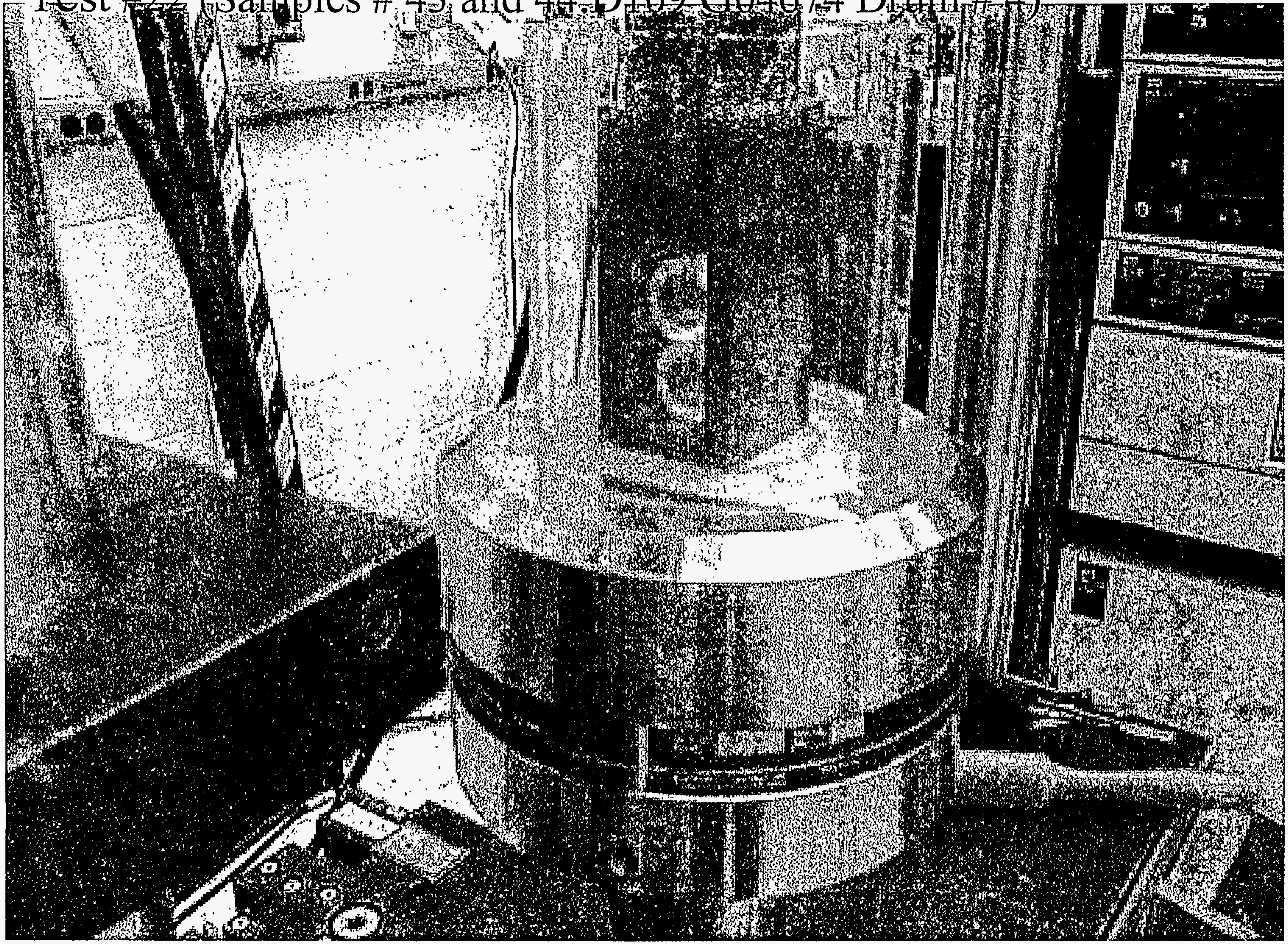
Test # 19 (Samples #37 and 38 :D85 G04688 Drum # 4 Chipped)



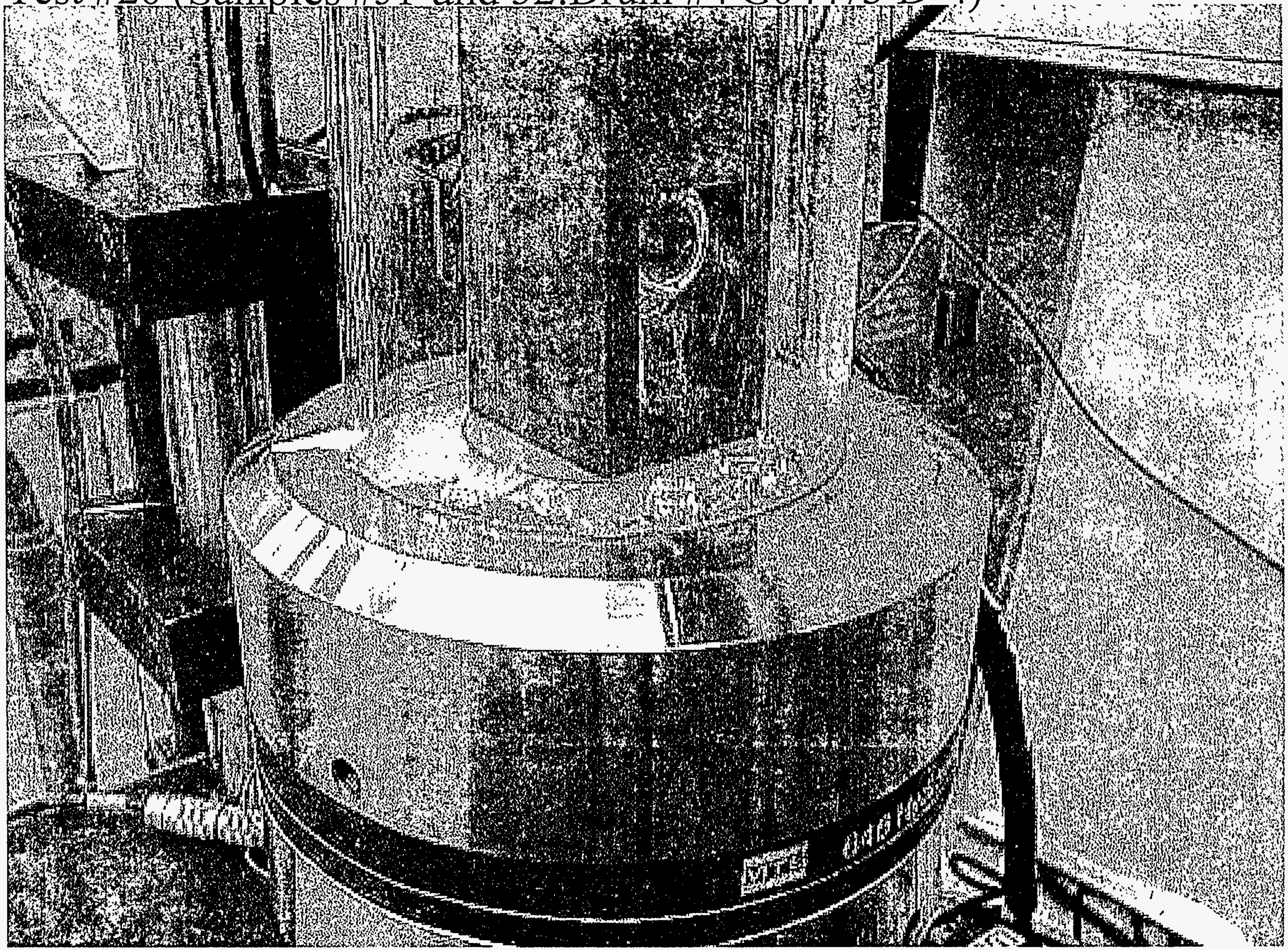
Test # 19 (Samples # 37 and 38:D85 G04688 Drum #4 Chipped)



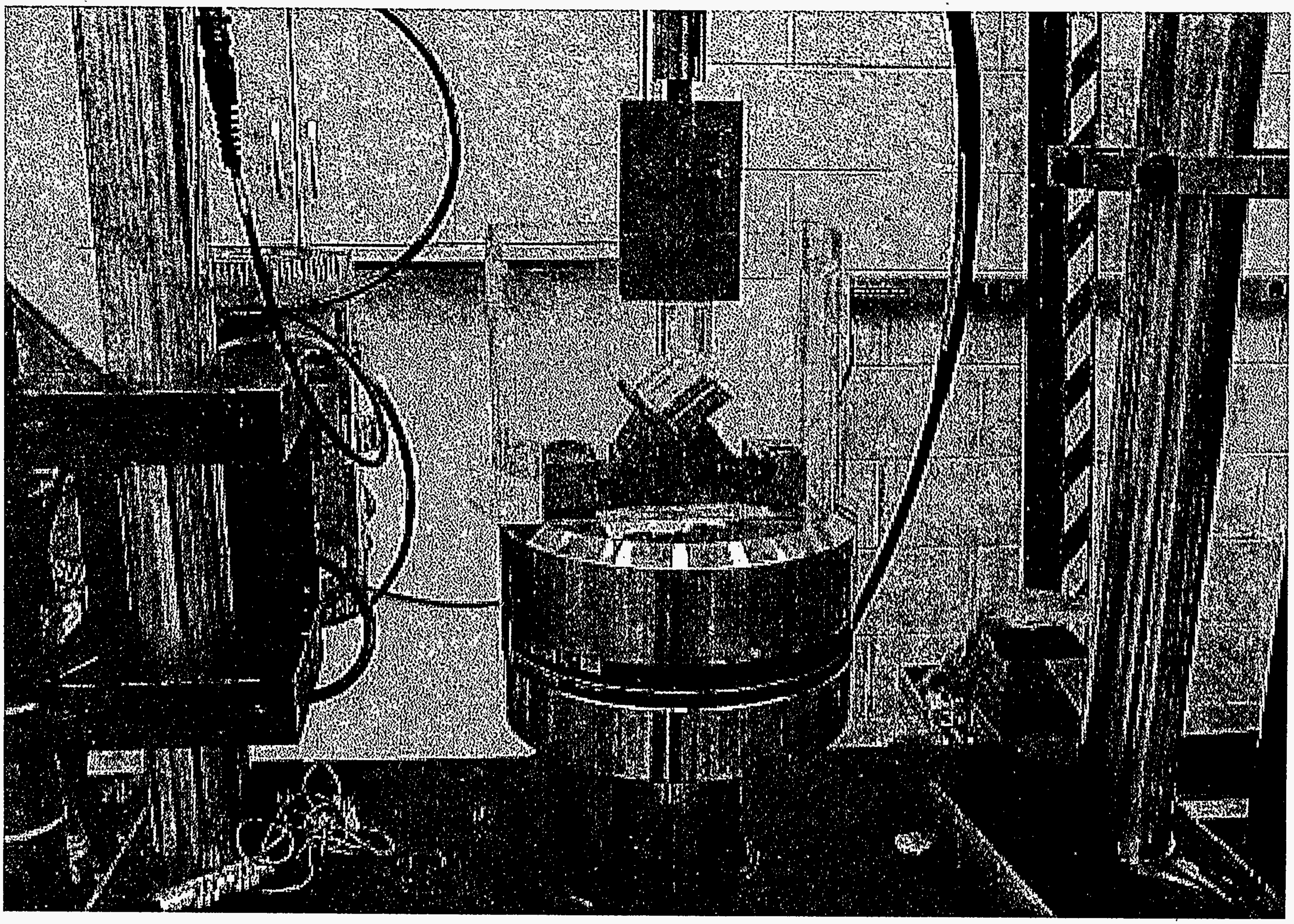
Test #22 (Samples # 43 and 44:D109 G04674 Drum # 4)



Test #26 (Samples #51 and 52:Drum #4 G04473 D-4)



Style #5 Test #1: Samples #1 and 2(G0476 D-16 Drum#4)



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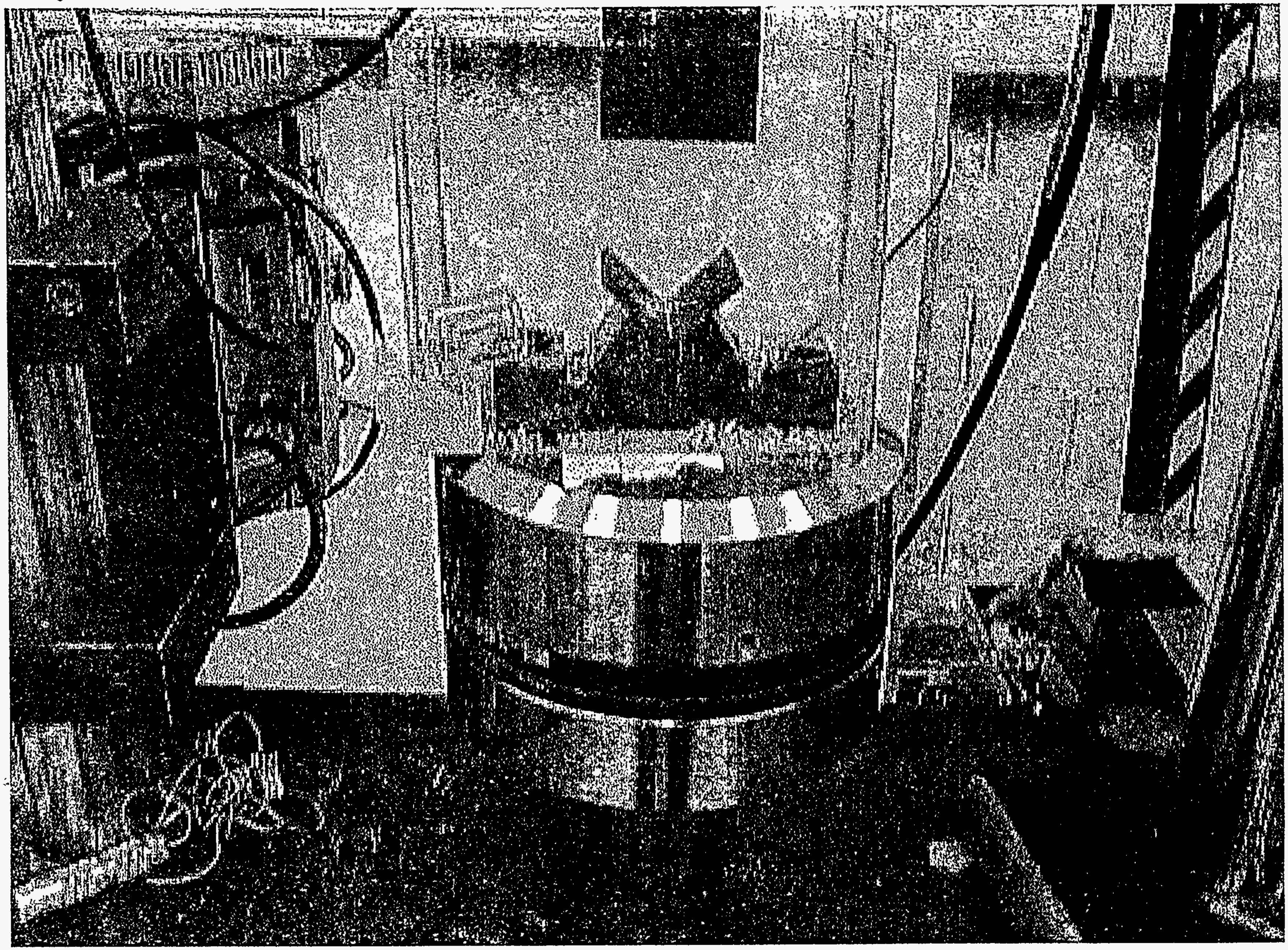
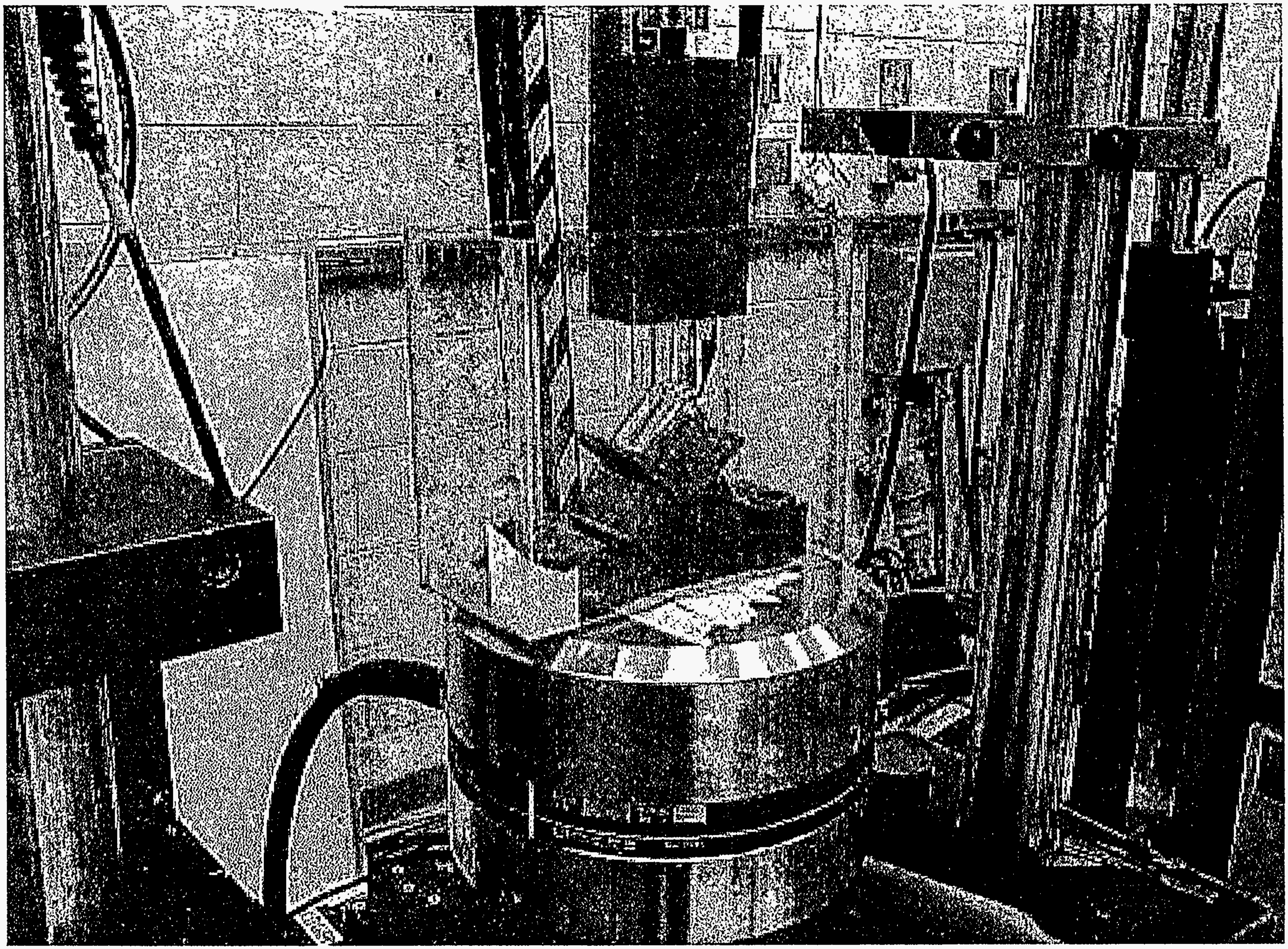
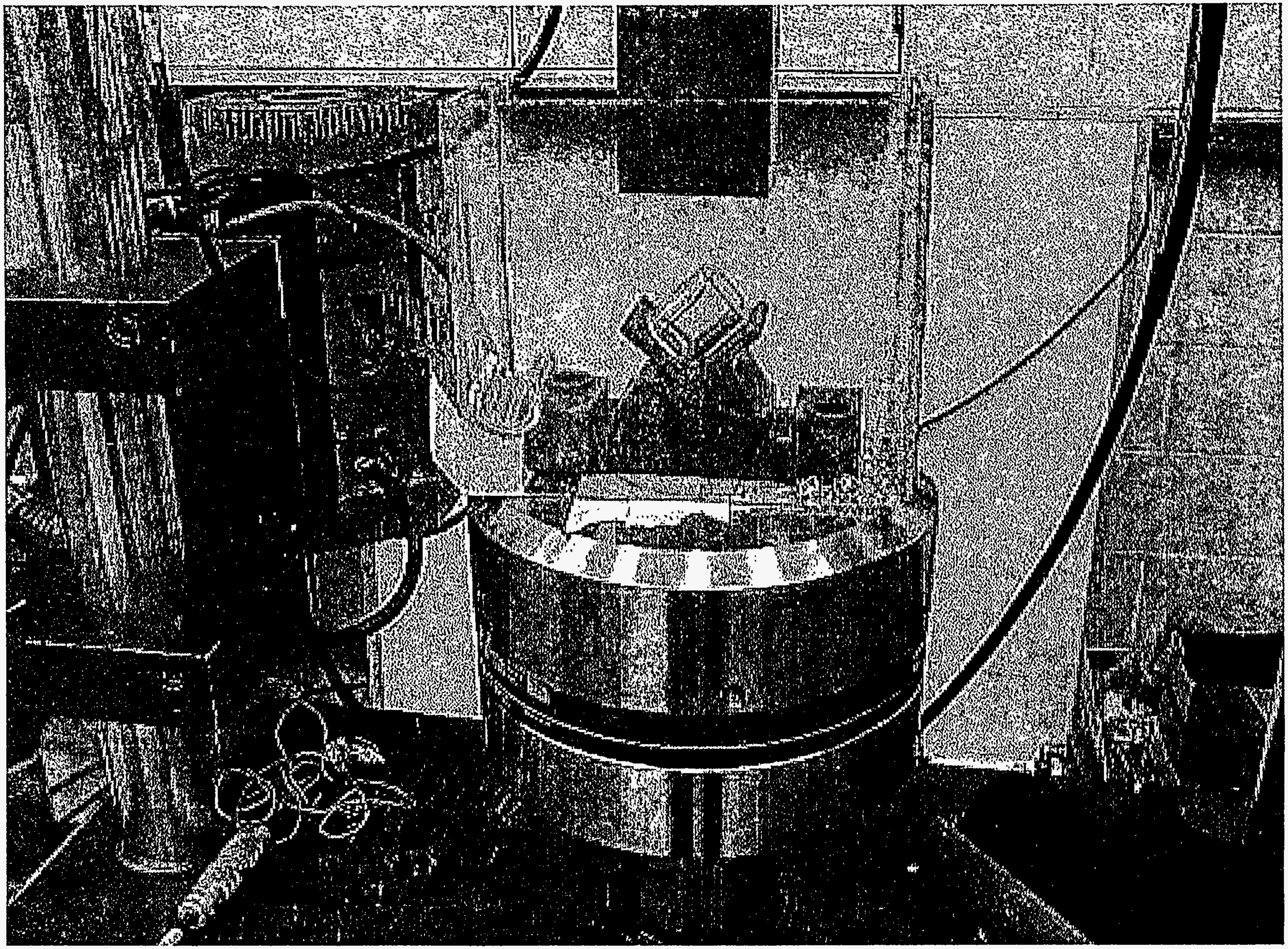
Style #5 Test #1 4/13/98



Style #5 Test #6 Samples # 11 & 12:(04694 D-48 Drum #4)



Style #5 Test #6



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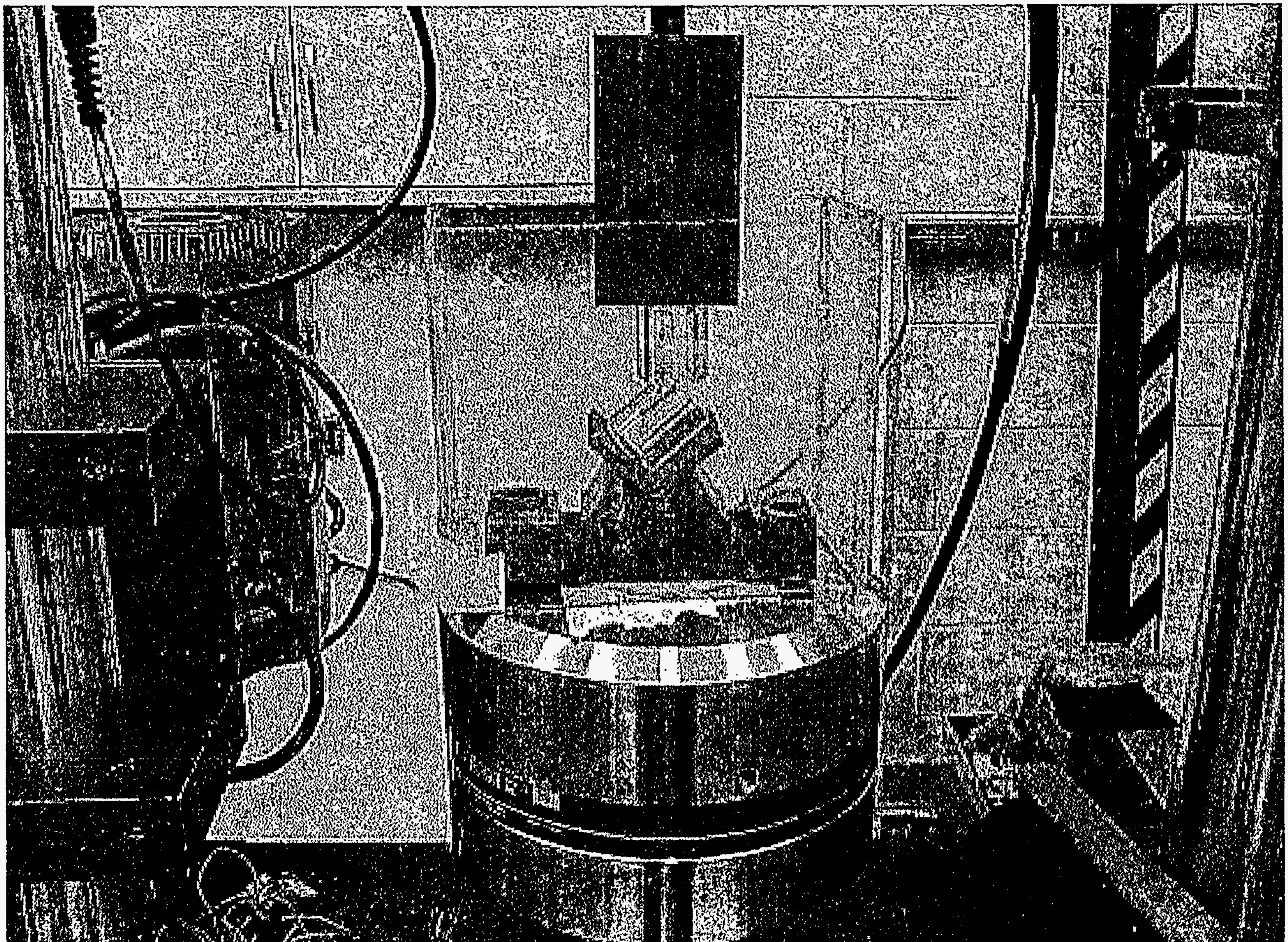
Styl #5 Test #7:Samples # 13 & 14(D04665 D-65 DRUM #4)



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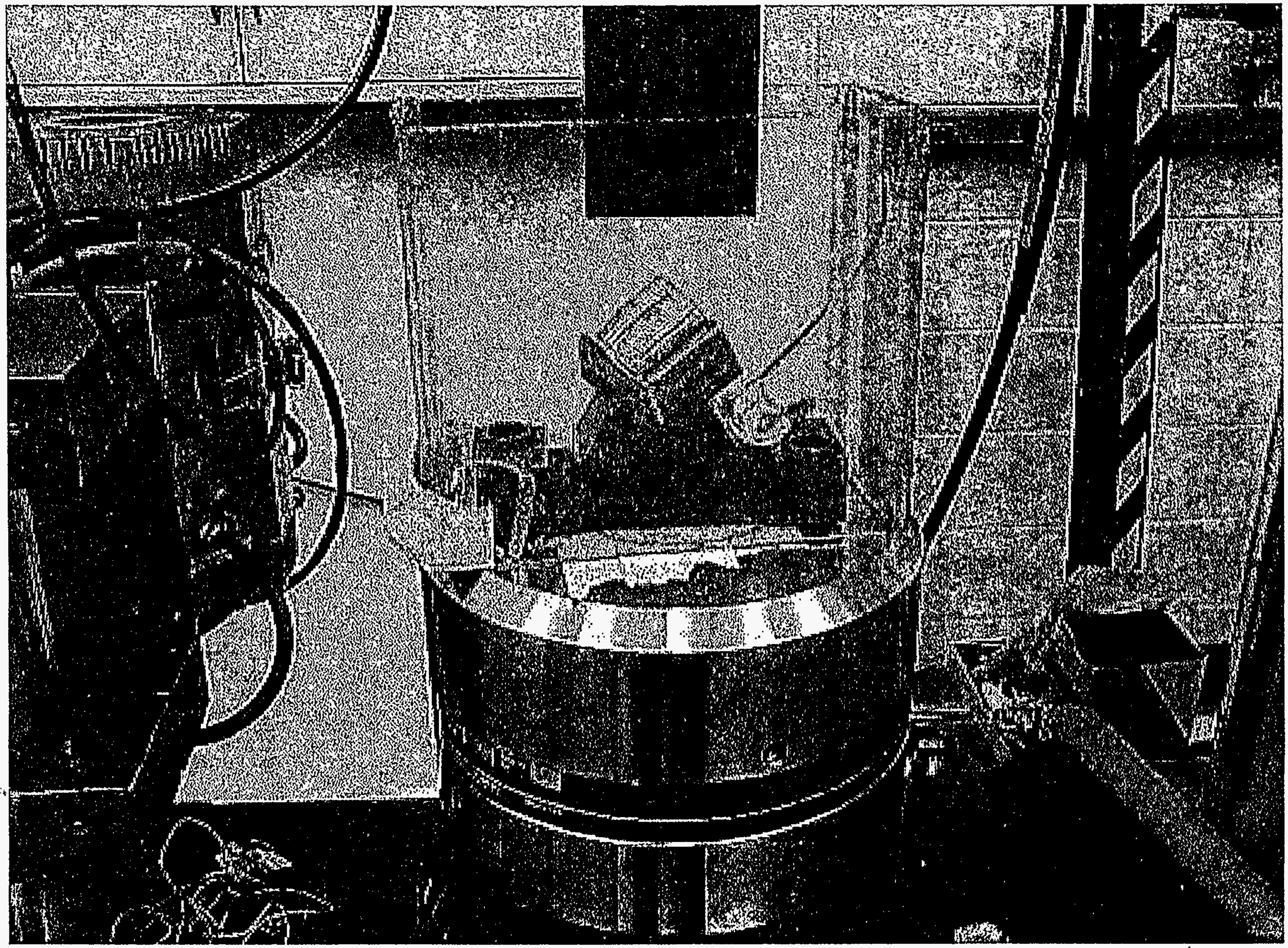
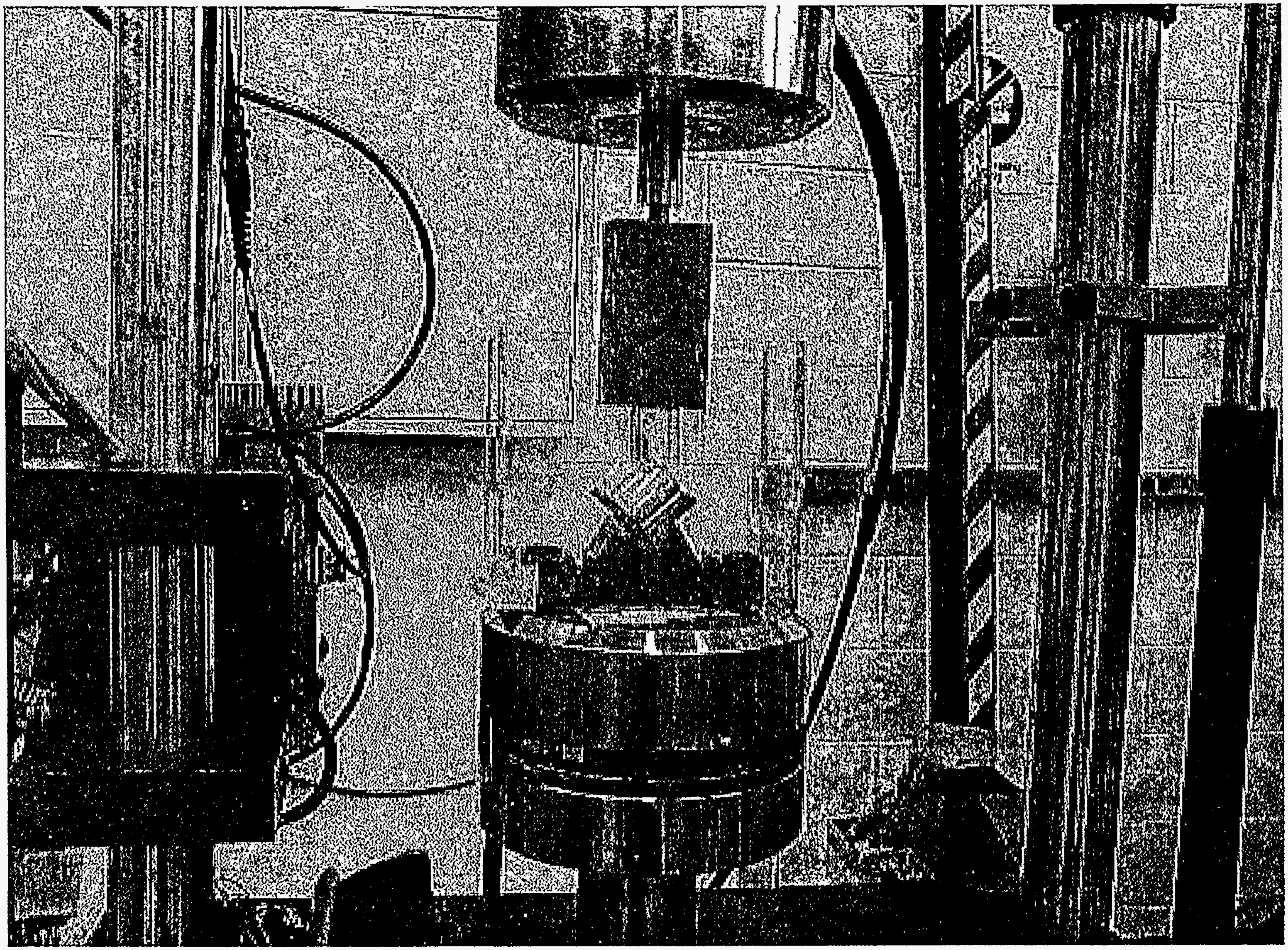
Style 5 Test #7



Style #5 Test# 10 Samples # 19 & 20(G04658 D99 Drum #4)



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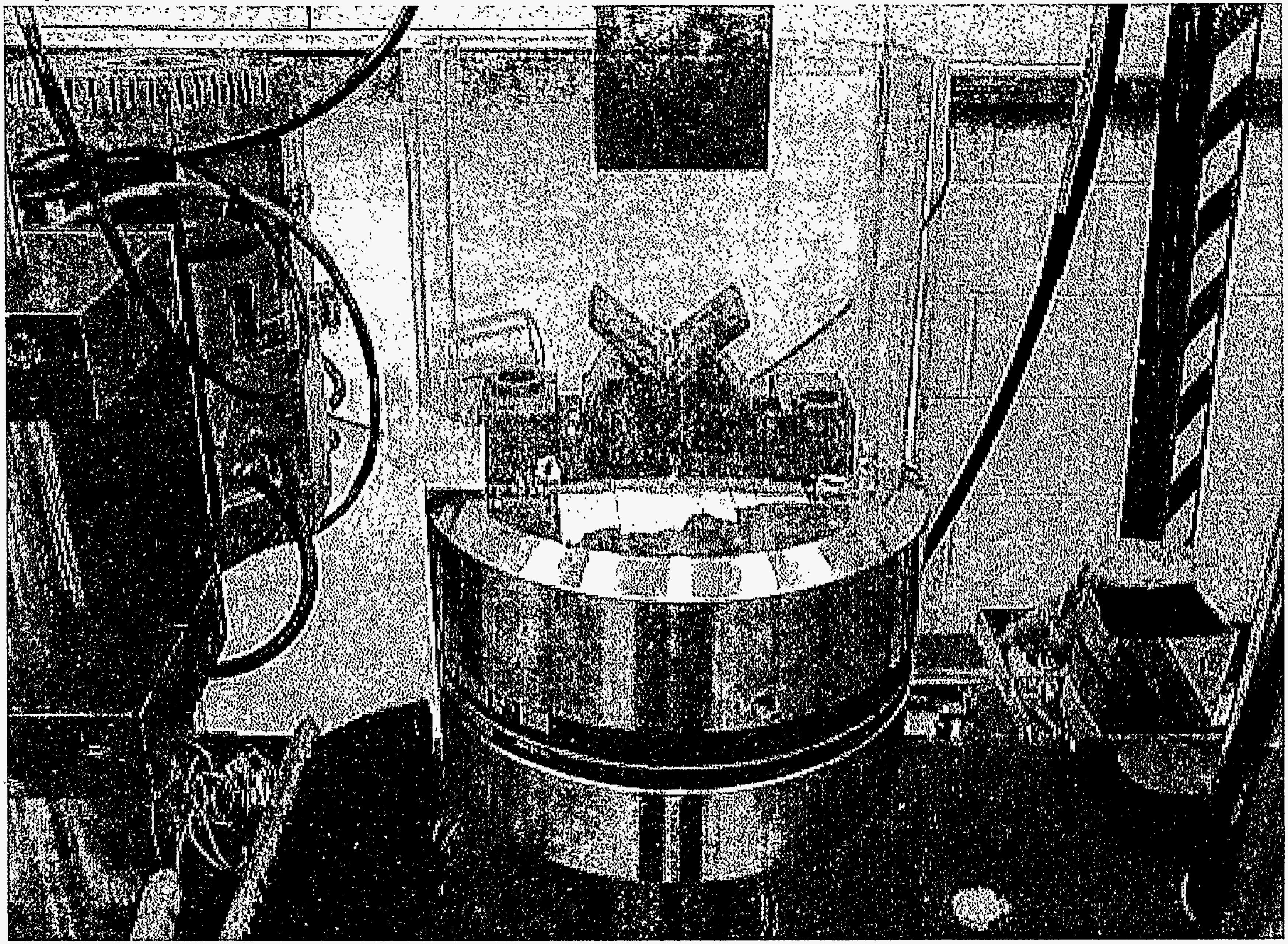
Style #5 Test #10



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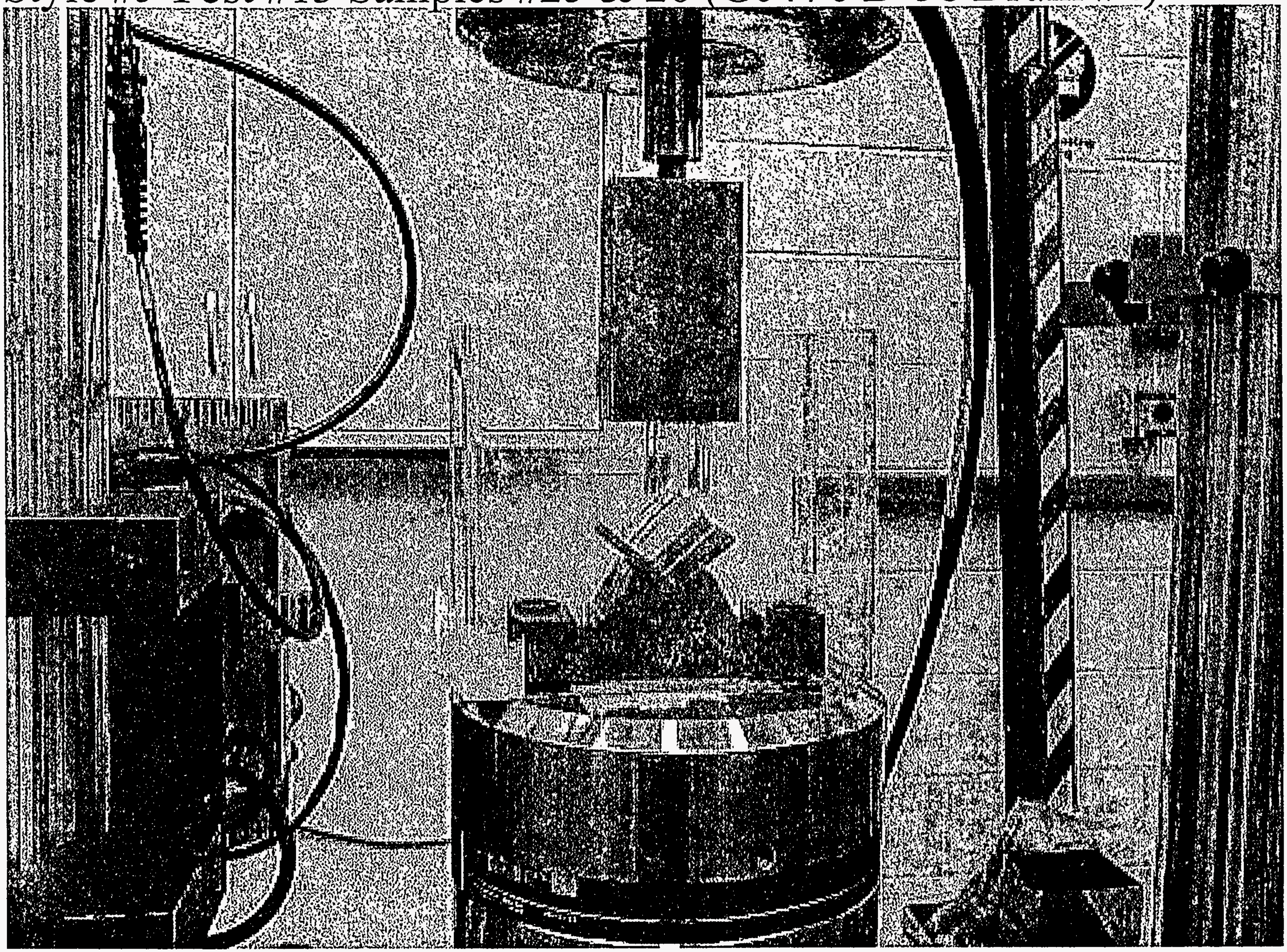

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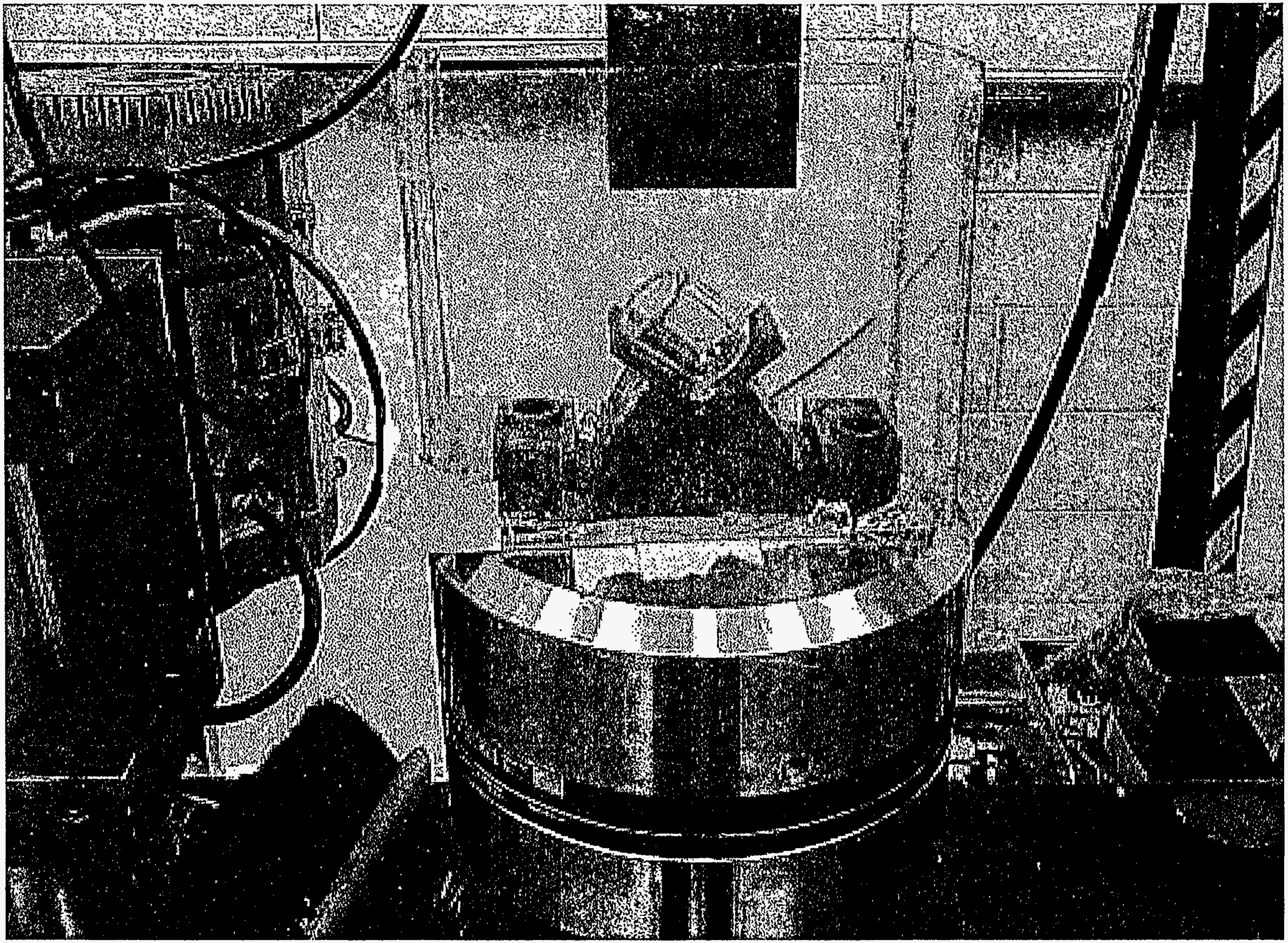

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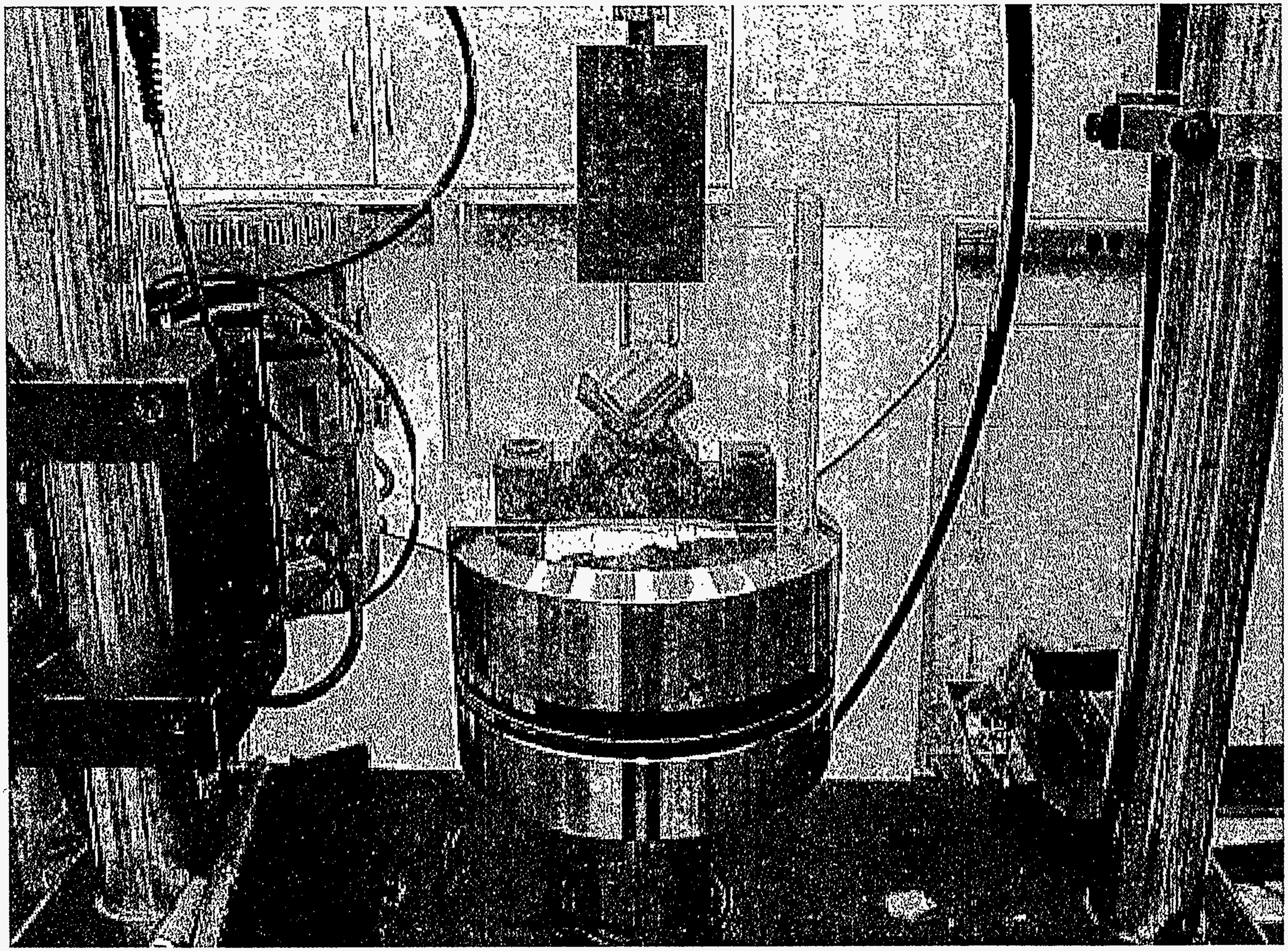
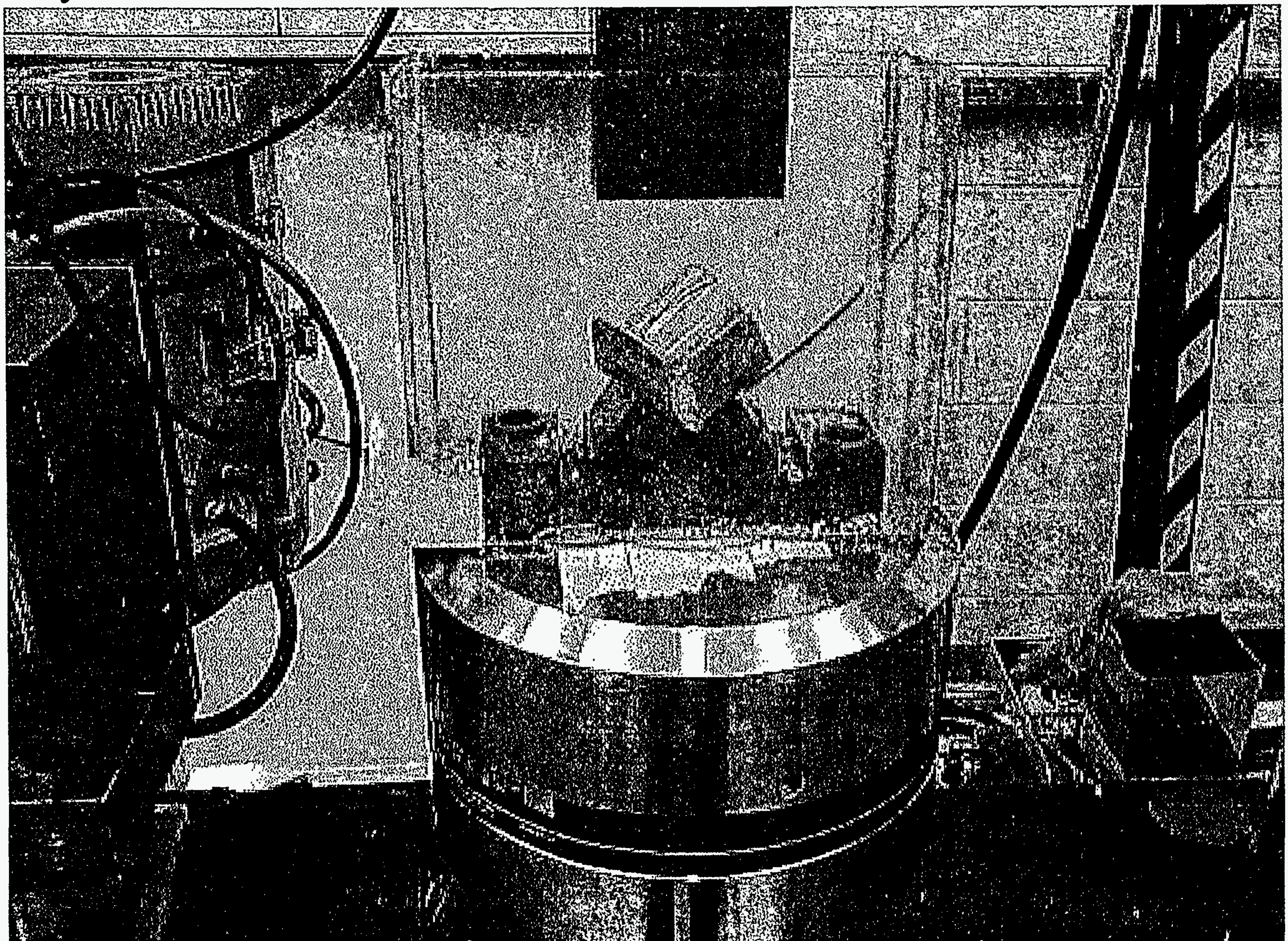
Style # 5 Test #16 Samples #31 and 32(G04773 Drum #4)



Style #5 Test 16



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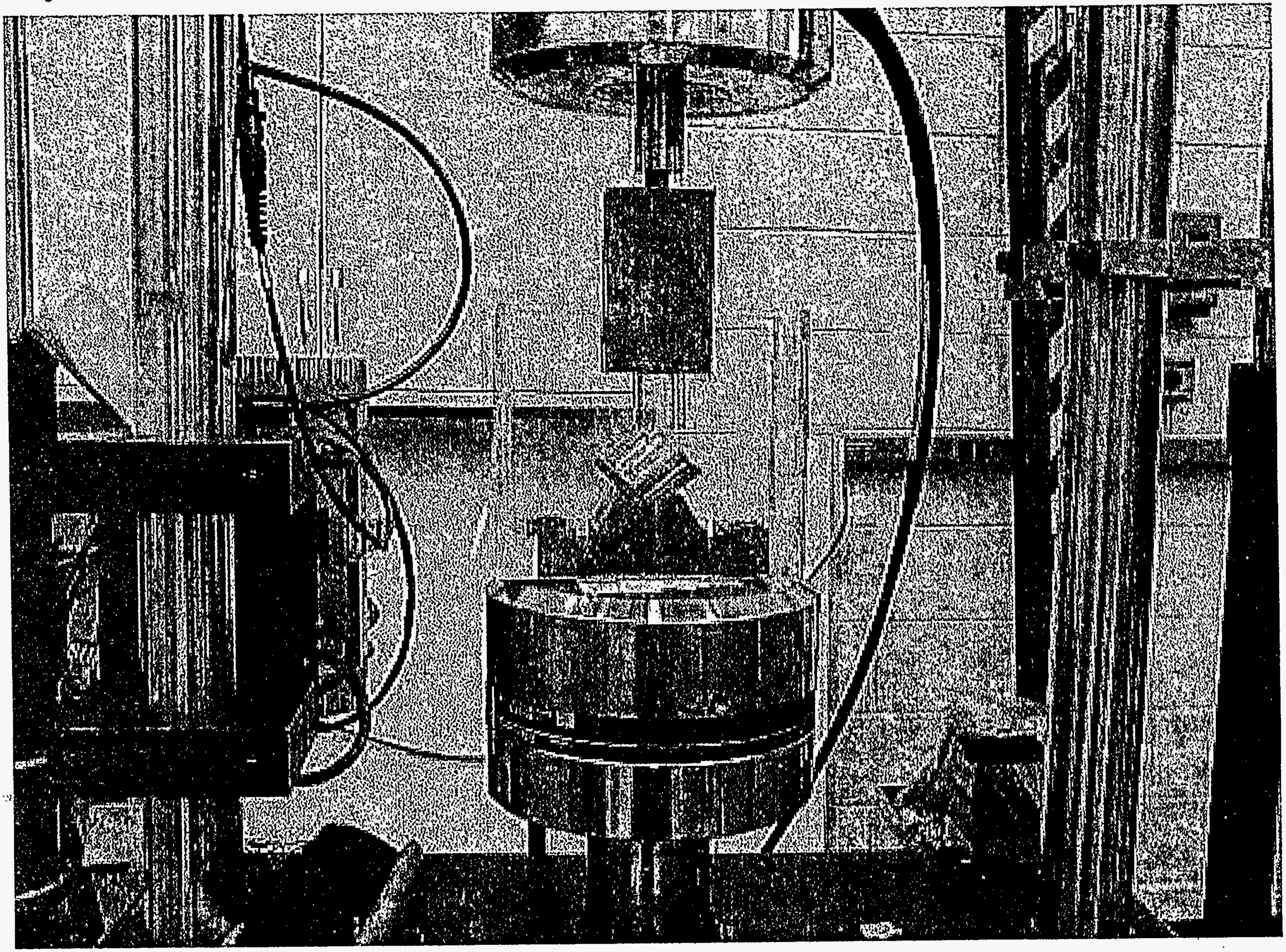
Style #5 Test 19 Samples #37 & 38 (G04688 D85 Drum #4 Chipped)



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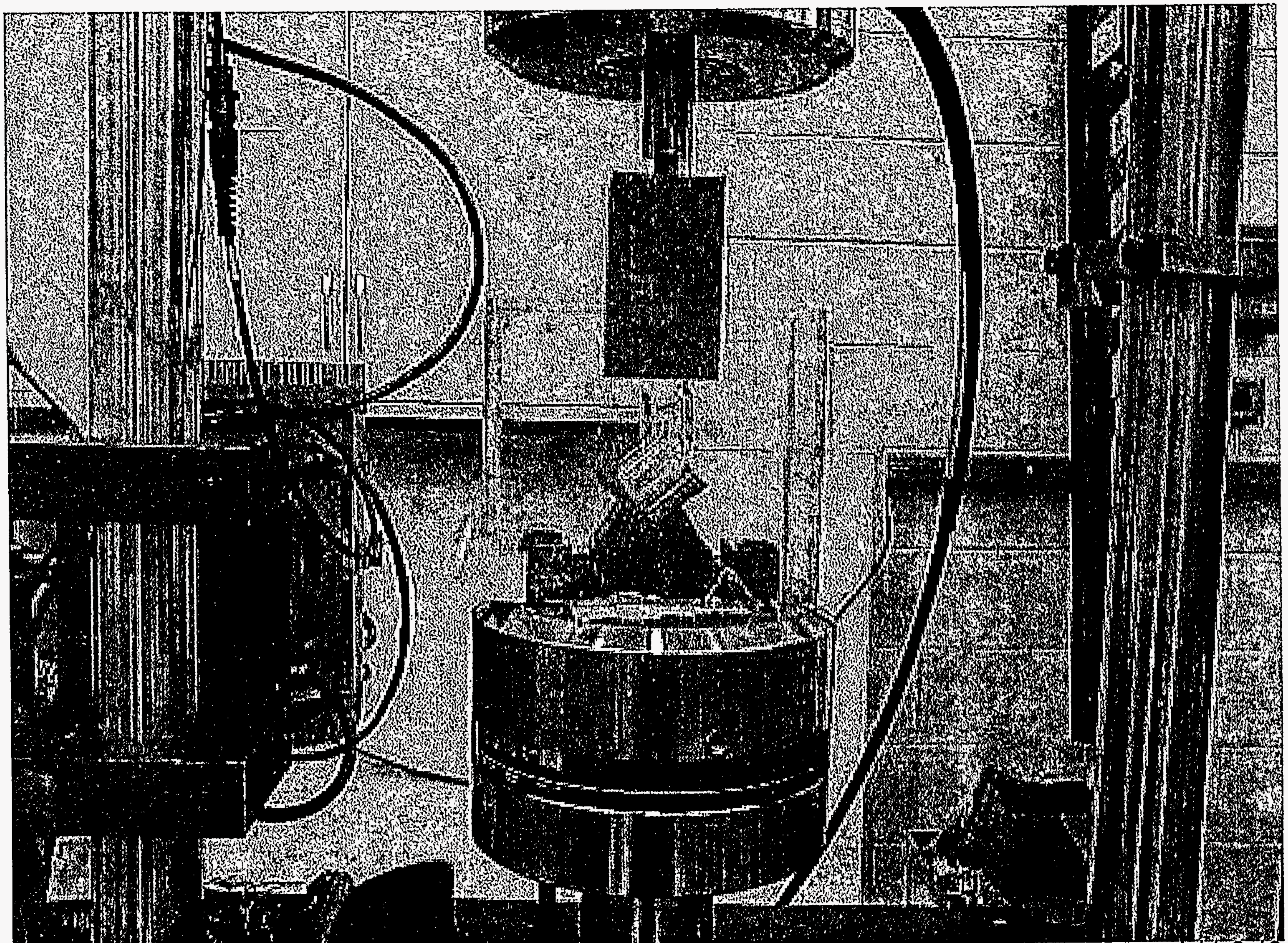
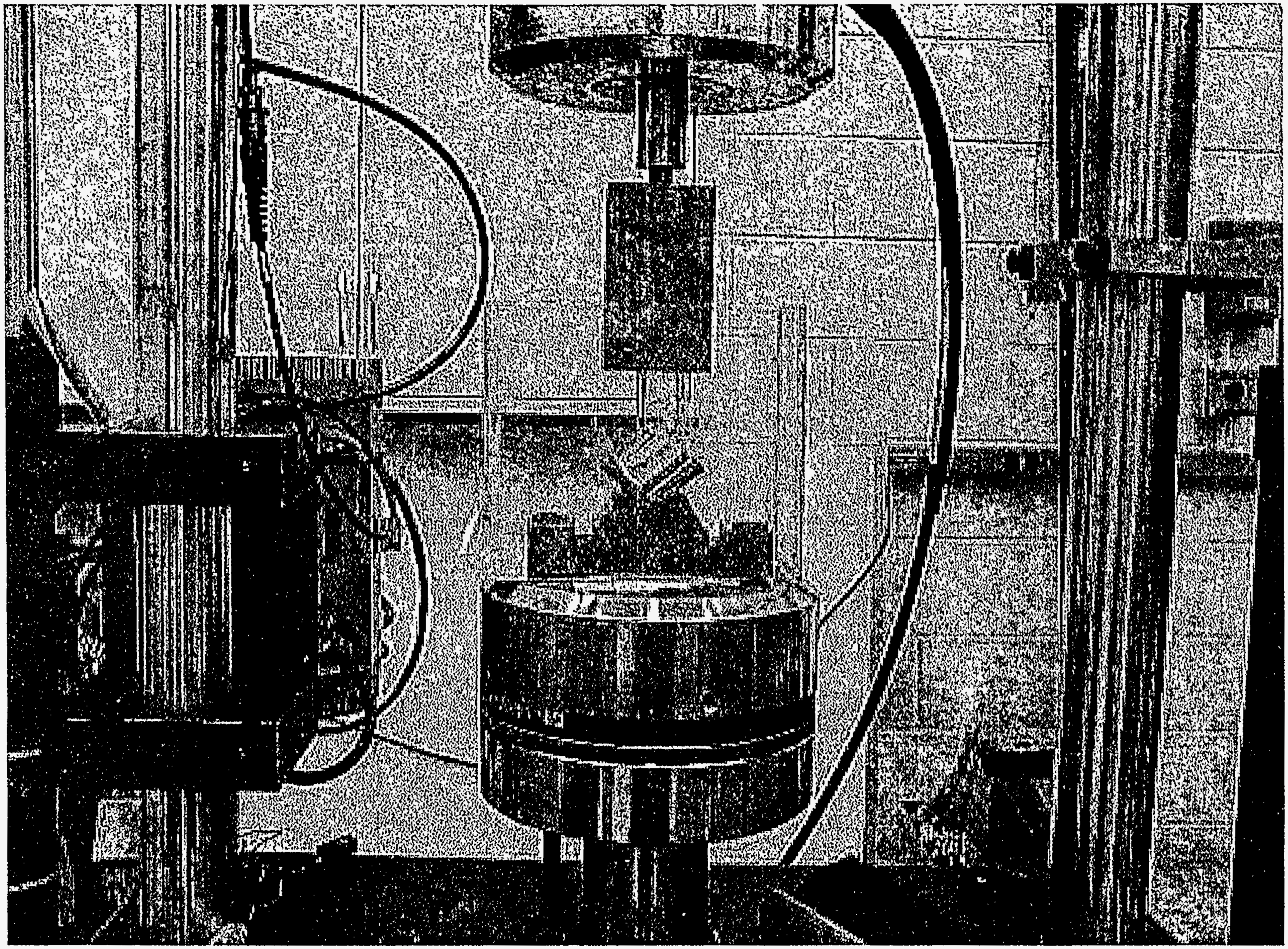
Style #5 Test 19



Style #5 Test #22 Samples # 43 & 44(G04674 D109 Drum#4)



Note:No Picture Broken.

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