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AN INVESTIGATION AND COMPARISON OF VARIOUS TECHNIQUES OF DISAGGREGATION AS APPLIED TO SHALES

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William Fay Bradford
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AN INVESTIGATION AND COMPARISON OF VARIOUS TECHNIQUES OF DISAGGREGATION AS APPLIED TO SHALES

by

WILLIAM FAY BRADFORD

Submitted to the College of Science and Arts of Michigan
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Department of Geology

1958

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AN INVESTIGATION AND COMPARISON OF VARIOUS TECHNIQUES OF DISAGGREGATION AS APPLIED TO SHALES

WILLIAM FAY BRADFORD

ABSTRACT

The purpose of this project was to employ a number of disaggregation techniques on shales to determine which techniques produced the best results.

The disaggregation methods employed in this research were both physical and chemical. The physical included ultrasonic vibration, mortar and pestle, and oak board. The chemical included potassium hydroxide and perchloric acid.

Eight rock samples were selected; they included six shales, one sandstone and one siltstone. These samples were treated by the various disaggregation techniques and then pipette analyses were run on each sample. The results of the analyses were compared to determine which methods were most effective.

The results of this research indicate that for the most successful disaggregation of a shale, the ultrasonic vibration

method should be employed. However, the perchloric acid method produced the best results on the coarser-grained siltstone and sandstone.

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AN INVESTIGATION AND COMPARISON
OF VARIOUS TECHNIQUES OF DISAGGREGATION
AS APPLIED TO SHALES

INTRODUCTION

Purpose

The purpose of this research was to employ various disaggregation methods on a number of shales and analyze the results to determine which method of disaggregation produced the best results.

Methods and Scope

The methods used in this research were both physical and chemical. The physical were ultrasonic vibration, mortar and pestle, and oak block. The chemical means utilized potassium hydroxide and perchloric acid.

The scope of the research was to disaggregate the samples, run a pipette analysis, interpret the results, and determine which method gave the best results.

Sample Selection

Eight rocks were selected; they included six shales, one sandstone, and one siltstone (Table I).

TABLE I

ROCK SAMPLES

Sample	Name	Source
A	Marshall sandstone	Grindstone City, Michigan
B	glazed argillaceous shale	Cohoes, New York
C	bituminous shale	Leroy, New York
D	Saginaw shale	Grand Ledge, Michigan
E	petroliferous shale	Fossil, Wyoming
F	siltstone	Mexico
G	carbonaceous shale	Moscow, New York
H	Antrim shale	Charlevoix County, Michigan

PHYSICAL METHODS

Introduction

Because of the lack of information about the ultrasonic vibrator, it was decided to explore the use of this machine extensively to determine if it would be successful in this application. It was not decided to add the mortar and pestle method and the oak block method until after the chemical methods had been started. Because of the lack of sample material after starting the chemical methods, the mortar and pestle method and the oak block method could only be run on one rock type. However, the sample that was used in these two methods was one of the toughest of the samples and it was felt that if disaggregation was successful on this sample, it would also be successful on other shales.

Ultrasonic Vibration

Equipment

The ultrasonic vibration machine had a maximum frequency of 300 kc, with an oscillator plate power output of 300 watts. The power supply was full wave, producing 1500 volts at full power. The vibrations were produced by a disc-shaped quartz crystal transducer about four inches in diameter and about one-half inch thick.

The transducer was located inside a heavy lucite cylinder. The cylinder was filled with oil which transmitted the vibrations produced by the quartz crystal transducer to the samples. Various other transducers are obtainable which would increase the effectiveness of the treatment, however none of them were available. Because of the power loss due to the shape of the transducer, it was decided to use one gram samples in this machine.

Sample Preparation

Because of the lack of knowledge and information about this machine, three samples of each of the eight rocks were subjected to a treatment of five minutes, ten minutes, and fifteen minutes respectively (Table II). The rocks were crushed with a hammer to a maximum diameter of approximately one-sixteenth of an inch and three samples of 1.000 gram each were taken from each of the eight pulverized rocks. The samples were placed in water in a small glass test tube and then treated.

Disaggregation

During the treatment of the samples the machine was run at full power for five minute periods whenever possible. The glass test tube containing the pulverized sample was suspended in the oil during the treatment. At times the machine became so hot the water in the test tube boiled.

TABLE II

ULTRASONIC VIBRATION METHOD

SAMPLE TREATMENT

Sample	Length of Vibration	Power in Watts	Milli-amperes	Container	Remarks
A-1	5	130	171 ma.	Glass	
A-2	10	130	170 ma.	Glass	
A-3	15	130	170 ma.	Glass	Boiled at 15 minutes.
B-1	5	130	170 ma.	Glass	
B-2	10	130	175 ma.	Glass	Used fan.
B-3	15	130	175 ma.	Glass	Used fan.
C-1	5	130	170 ma.	Glass	
C-2	10	130	170 ma.	Glass	Boiled at 5 minutes.
C-3	15	130	170 ma.	Glass	
D-1	5	130	170 ma.	Glass	
D-2	10	130	170 ma.	Glass	Boiled at 4 3/4 min., 8 min., 9 min.
D-3	15	130	167 ma.	Glass	Used fan.
E-1	5	130	170 ma.	Glass	Boiled at 2 minutes and 5 minutes.
E-2	10	130	165 ma.	Glass	Used fan.
E-3	15	130	165 ma.	Glass	Used fan.
F-1	5	130	165 ma.	Glass	Boiled at 1 3/4 minutes and 5 minutes.
F-2	10	130	170 ma.	Glass	Used fan.
F-3	15	130	167 ma.	Glass	Used fan.
G-1	5	130	167 ma.	Glass	Boiled at 1 1/2 min., 2 1/2 min., 5 min.
G-2	10	130	167 ma.	Glass	Used fan.
G-3	15	130	167 ma.	Glass	Used fan.
H-1	5	130	167 ma.	Glass	Boiled at 1 1/2 minutes and 5 minutes.
H-2	10	130	167 ma.	Glass	Used fan.
H-3	15	130	167 ma.	Glass	Used fan.

This necessitated a stop in the treatment to allow the machine to cool. On one occasion a fuse burned out. The addition of an air cooling unit and a strong fan helped prevent overheating so that most of the samples could be treated for the full five minute periods. This process took longer than the prescribed time because the machine had to cool between treatments. After the air cooling unit was added the average timing was to treat the sample for five minutes and then allow the machine to cool for ten minutes.

Pipette Analysis

After all the samples were vibrated, each sample was wet sieved through a 230 mesh sieve to separate the sand from the silt and clay particles. During the sieving many of the samples had aggregates about one-sixteenth of an inch in diameter. In most of the samples these aggregates could be crushed by attrition between the fingers. However, in some samples they could not be separated. The washing was continued until all of the clay and silt particles passed through the sieve. The amount of sample larger than the 230 mesh sieve was dried, weighed, and recorded (Table III).

Due to the small amount of sample used, standard pipette analysis procedures could not be employed. That part of the samples smaller than the 230 mesh sieve was washed into a 600 ml beaker and enough distilled water was added to make the total height 10 cm. The beaker was

TABLE III

ULTRASONIC VIBRATION METHOD

WEIGHT AND PERCENTAGE OF SAMPLE LARGER THAN 0.0625 MILLIMETERS

Sample	Weight of Sample Larger than 0.0625 mm	Percentage of Total Sample
A-1	.449	44.9
A-2	.492	49.2
A-3	.546	54.6
B-1	.733	73.3
B-2	.776	77.6
B-3	.749	74.9
C-1	.231	23.1
C-2	.146	14.6
C-3	.041	4.1
D-1	.008	0.8
D-2	.000	0.0
D-3	.032	3.2
E-1	.046	4.6
E-2	.048	4.8
E-3	.017	1.7
F-1	.697	69.7
F-2	.698	69.8
F-3	.689	68.9
G-1	.468	46.8
G-2	.471	47.1
G-3	.399	39.9
H-1	.201	20.1
H-2	.098	9.8
H-3	.103	10.3

agitated and then allowed to settle for the standard periods of time according to Stokes' Law (Krumbein and Pettijohn, 1938). At the end of the designated periods of time the liquid was decanted off, leaving the sediment which had settled. This sediment was washed into another beaker, dried, weighed, and recorded. Enough distilled water to make a total height of 10 cm was added and the process was repeated for each of the times specified in Stokes' Law (Table IV).

Data Analysis

Table II, which shows the flow sheet for the sample treatment is self-explanatory, except to note that the fluctuation of milliamperes could not be controlled by the author. The milliamperes increased as the temperature of the machine increased.

In Table III, the percentage of sample larger than 230 mesh in diameter was calculated directly from the weight of the material because the original size of each of the samples was 1.000 gram.

Table IV lists the weight of each portion of each sample which settled according to their diameter in millimeters. The depth of settling was 10 centimeters in all cases.

Table V lists the percentage by weight of each portion of each sample according to the diameter in millimeters.

TABLE IV

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

SIZE ANALYSIS

Diameter in Millimeters	Time			Weight in Grams								
	Hr.	Min.	Sec.	A-1	A-2	A-3	B-1	B-2	B-3	C-1	C-2	C-3
-Over .0625				.449	.492	.546	.733	.776	.749	.231	.146	.041
.0625			29	.103	.147	.084	.070	.049	.028	.069	.057	.068
.0442			58	.081	.098	.059	.063	.040	.043	.082	.070	.041
.0312		1	56	.057	.027	.046	.048	.026	.021	.073	.041	.083
.0221		3	52	.066	.038	.037	.041	.027	.028	.063	.060	.097
.0156		7	44	.091	.077	.021	.017	.020	.019	.080	.111	.142
.0110		15		.056	.033	.029	.027	.020	.028	.037	.071	.127
.0078		31		.041	.024	.048	.025	.017	.027	.055	.108	.168
.0055	1	1		.027	.019	.032	.032	.013	.013	.158	.122	.068
.0039	2	3		.039	.020	.064	.010	.011	.025	.056	.070	.101
.00276	4	5		.012	.025	.027	.002	.016	.021	.037	.038	.010
.00195	8	10		.030	.023	.021		.009	.010	.009	.041	.005
.00138	16	21		.014	.010	.029		.002	.005	.015	.007	.019
.00098	32	42		.006	.000	.018						
.00069	65	24				.036						
.00049	130	50										

TABLE IV (Continued)

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

SIZE ANALYSIS

Diameter in Millimeters	Time			Weight in Grams								
	Hr.	Min.	Sec.	D-1	D-2	D-3	E-1	E-2	E-3	F-1	F-2	F-3
-Over				.008	.000	.032	.046	.048	.017	.697	.698	.684
.0625				.089	.083	.078	.071	.107	.079	.050	.050	.045
.0625			29	.054	.062	.081	.069	.088	.081	.042	.036	.029
.0442			58	.060	.078	.089	.077	.089	.088	.017	.029	.016
.0312		1	56	.065	.083	.115	.074	.080	.087	.011	.010	.033
.0221		3	52	.051	.071	.093	.067	.087	.084	.023	.029	.021
.0156		7	44	.147	.103	.070	.082	.108	.104	.037	.020	.029
.0110		15		.158	.120	.061	.100	.096	.086	.015	.051	.040
.0078		31		.131	.159	.046	.088	.047	.049	.019	.014	.012
.0055	1	1		.101	.067	.043	.063	.056	.091	.027	.010	.029
.0039	2	3		.040	.047	.055	.057	.060	.061	.004	.012	.006
.00276	4	5		.020	.014	.056	.040	.037	.048	.011	.001	.018
.00195	8	10		.018	.023	.031	.010	.020	.023	.000	.000	.000
.00138	16	21			.010	.026	.009	.009	.010			.000
.00098	32	42				.011	.008	.002	.015			.000
.00069	65	24				.005	.025	.003	.013			
.00049	130	50										

TABLE IV (Continued)

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

SIZE ANALYSIS

Diameter in Millimeters	Time			Weight in Grams					
	Hr.	Min.	Sec.	G-1	G-2	G-3	H-1	H-2	H-3
-Over .0625				.468	.471	.399	.201	.098	.103
.0625			29	.078	.040	.084	.079	.085	.108
.0442			58	.055	.025	.051	.045	.070	.094
.0312		1	56	.058	.075	.034	.041	.090	.071
.0221		3	52	.060	.085	.061	.081	.086	.065
.0156		7	44	.055	.065	.040	.080	.096	.078
.0110		15		.027	.049	.049	.050	.093	.080
.0078		31		.012	.029	.034	.045	.101	.101
.0055	1	1		.020	.038	.026	.064	.081	.084
.0039	2	3		.011	.015	.026	.088	.064	.056
.00276	4	5		.003	.003	.025	.041	.049	.045
.00195	8	10		.005	.025	.027	.033	.059	.038
.00138	16	21		.005	.010	.007	.021	.016	.028
.00098	32	42		.005	.000	.019	.025	.020	.032
.00069	65	24		.000	.000	.000	.008	.012	.009
.00049	130	50			.000	.012	.007	.009	.012

TABLE V

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

PERCENTAGE BY WEIGHT

Diameter in Millimeters	Time			Percentage								
	Hr.	Min.	Sec.	A-1	A-2	A-3	B-1	B-2	B-3	C-1	C-2	C-3
.0625			29	18.7	28.9	18.5	26.4	21.7	11.1	9.0	6.1	7.1
.0442			58	4.7	19.3	13.0	23.6	17.6	17.1	10.6	8.3	4.3
.0312		1	56	0.4	5.3	10.1	18.0	11.6	8.4	9.5	4.8	8.7
.0221		3	52	12.0	7.5	8.1	15.3	12.0	11.2	8.2	7.0	10.1
.0156		7	44	16.4	15.2	4.6	6.4	8.9	8.0	10.4	13.0	14.8
.0110		15		10.2	6.5	6.4	10.1	8.9	11.2	4.8	8.3	13.2
.0078		31		7.6	4.7	10.6	9.4	7.6	10.8	7.1	12.6	17.5
.0055	1	1		4.9	3.7	7.0	12.0	5.8	5.2	20.5	14.3	7.1
.0039	2	3		7.1	3.9	14.1	3.7	4.9	10.0	7.3	8.2	10.5
.00276	4	5		2.0	4.9	5.9	.7	7.1	8.4	4.8	4.4	1.0
.00195	8	10		5.5	4.5	4.6		4.0	4.0	1.2	4.8	.5
.00138	16	21		2.5	2.0	6.3		.9	2.0	1.9	.8	2.0
.00098	32	42		1.1		4.0						
.00069	65	24				7.9						
.00049	130	50										
				113.1	106.4	121.1	125.6	110.0	107.4	95.3	92.6	96.8

TABLE V (Continued)

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

PERCENTAGE BY WEIGHT

Diameter in Millimeters	Time			Percentage								
	Hr.	Min.	Sec.	D-1	D-2	D-3	E-1	E-2	E-3	F-1	F-2	F-3
.0625			29	9.0	8.3	8.1	7.4	11.3	8.0	16.5	16.5	14.2
.0442			58	5.5	6.2	8.4	7.2	9.3	8.2	13.9	11.9	9.2
.0312		1	56	6.1	7.8	9.2	8.1	9.4	8.9	5.6	9.6	5.7
.0221		3	52	6.6	8.3	11.9	7.8	8.4	8.8	3.6	3.3	10.4
.0156		7	44	5.2	7.1	9.6	7.0	9.1	8.5	7.6	9.6	6.6
.0110		15		14.9	10.3	7.2	8.6	11.4	10.5	12.2	6.6	9.2
.0073		31		16.0	12.0	6.3	10.5	10.1	8.7	5.0	16.9	12.7
.0055	1	1		13.2	15.9	4.8	9.2	5.1	5.0	6.3	4.6	3.8
.0039	2	3		10.2	6.7	4.4	6.6	5.9	9.3	8.9	3.3	9.2
.00276	4	5		4.0	4.7	5.7	6.0	6.3	6.2	1.3	4.0	1.9
.00195	8	10		2.2	1.4	5.8	4.2	3.9	4.9	3.6	.3	5.7
.00138	16	21		1.8	2.3	3.2	1.1	2.1	2.3			
.00098	32	42			1.0	1.1	.9	.9	1.0			
.00069	65	24				.5	.8	.2	1.5			
.00049	130	50					2.6	.3	1.3			
				94.7	92.0	86.2	88.0	93.7	93.1	84.5	86.6	88.6

TABLE V (Continued)

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

PERCENTAGE BY WEIGHT

	Time			Percentage					
Diameter in Millimeters	Hr.	Min.	Sec.	G-1	G-2	G-3	H-1	H-2	H-3
.0625			29	14.7	7.6	14.0	9.9	9.4	12.0
.0442			58	10.4	4.7	8.5	5.6	7.8	10.5
.0312		1	56	10.9	14.2	5.7	5.1	10.0	7.9
.0221		3	52	11.3	16.1	10.2	10.1	9.5	7.3
.0156		7	44	10.4	12.3	6.7	10.0	10.7	8.7
.0110		15		5.1	9.3	8.2	6.3	10.3	8.9
.0078		31		2.3	5.5	5.7	5.6	11.2	11.2
.0055	1	1		3.8	7.2	4.3	8.0	9.0	9.4
.0039	2	3		2.1	2.8	4.3	11.0	7.1	6.2
.00276	4	5		.6	.6	4.2	5.1	5.4	5.0
.00195	8	10		.9	4.7	4.5	4.1	6.5	4.2
.00138	16	21		.9	1.9	1.2	2.6	1.8	3.1
.00098	32	42		.9		3.2	3.1	2.2	3.6
.00069	65	24				0.0	1.0	1.3	1.0
.00049	130	50				2.0	.9	1.0	1.3
				74.3	86.9	82.7	88.4	103.2	100.3

The percentage was calculated by subtracting the weight of the material in the sample larger than 0.0625 mm from the original sample of 1.000 gram size. This value was used to calculate all of the percentages for the certain sample. This results in the value of a certain size representing the percentage of material of this size in the total weight of all the material which is less than 0.0625 mm in diameter. In short, the percentage value represents the percent of material in the silt and clay sizes only.

The cumulative percentage for each sample was obtained by adding each of the percentages for each sample to the percentages above it (Table VI).

Data Interpretation

In examining the portion of the sample larger than 0.0625 mm (Table III), it is evident that the disaggregation method was successful on some of the samples. Sample C, the bituminous shale, shows a definite decrease in the amount of sand particles which varies indirectly with the length of treatment. Sample D, the Saginaw shale, indicates that most, if not all, of the silt and clay particles have been separated from the sand particles. The results obtained by treating sample E, the petroliferous shale, indicates that most, if not all, of the silt and clay particles have been separated from the sand particles. The results of sample H, the Antrim shale, indicates that the majority of the silt

TABLE VI

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

CUMULATIVE PERCENTAGES

Diameter in Millimeters	Time											
	Hr.	Min.	Sec.	A-1	A-2	A-3	B-1	B-2	B-3	C-1	C-2	C-3
.0625			29	18.7	28.9	18.5	26.4	21.7	11.1	9.0	6.1	7.1
.0442			58	33.4	48.2	31.5	50.0	39.3	28.2	19.6	14.4	11.4
.0312		1	56	43.8	53.5	41.6	68.0	50.9	36.6	29.1	19.2	20.1
.0221		3	52	55.8	61.0	49.7	83.3	62.9	47.8	37.3	26.2	30.2
.0156		7	44	72.2	76.2	54.3	89.7	71.8	55.8	47.7	39.2	45.0
.0110		15		82.4	82.7	60.7	99.8	80.7	67.0	52.5	47.5	58.2
.0078		31		90.0	87.4	71.3	109.2	88.3	77.8	59.6	60.1	75.7
.0055	1	1		94.9	91.1	78.3	121.2	94.1	83.0	80.1	74.4	82.8
.0039	2	3		102.0	95.0	92.4	124.9	99.0	93.0	87.4	82.6	93.3
.00276	4	5		104.0	99.9	98.3	125.6	106.1	101.4	92.2	87.0	94.3
.00195	8	10		109.5	104.4	102.9		110.1	105.4	93.4	91.8	94.8
.00138	16	21		112.0	106.4	109.2		111.0	107.4	95.3	92.6	96.8
.00098	32	42		113.1		113.2						
.00069	65	24				121.1						
.00049	130	50										

TABLE VI (Continued)

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

CUMULATIVE PERCENTAGES

Diameter in Millimeters	Time											
	Hr.	Min.	Sec.	D-1	D-2	D-3	E-1	E-2	E-3	F-1	F-2	F-3
.0625			29	9.0	8.3	8.1	7.4	11.3	8.0	16.5	16.5	14.2
.0442			58	14.5	14.5	16.5	14.6	20.6	16.2	30.4	28.4	23.4
.0312		1	56	20.6	22.3	25.7	22.7	30.0	25.1	36.0	38.0	29.1
.0221		3	52	27.2	30.6	37.6	30.5	38.4	33.9	39.6	41.3	39.5
.0156		7	44	32.4	37.7	47.2	37.5	47.5	42.4	47.2	50.9	46.1
.0110		15		47.3	48.0	54.4	46.1	58.9	52.9	59.4	57.5	55.3
.0078		31		63.3	60.0	60.7	56.6	69.0	61.6	64.4	74.4	68.0
.0055	1	1		76.5	75.9	65.5	65.8	74.1	66.6	70.7	79.0	71.8
.0039	2	3		86.7	82.6	69.9	72.4	80.0	75.9	79.6	82.3	81.0
.00276	4	5		90.7	87.3	75.6	78.4	86.3	82.1	80.9	86.3	82.9
.00195	8	10		92.9	88.7	81.4	82.6	90.2	87.0	84.5	86.6	88.6
.00138	16	21		94.7	91.0	84.6	83.7	92.3	89.3			
.00098	32	42			92.0	85.7	84.6	93.2	90.3			
.00069	65	24					85.4	93.4	91.8			
.00049	130	50					88.0	93.7	93.1			

TABLE VI (Continued)

ULTRASONIC VIBRATION METHOD

PIPETTE ANALYSIS OF MATERIAL TREATED

CUMULATIVE PERCENTAGES

Diameter in Millimeters	Time Hr.	Min.	Sec.	G-1	G-2	G-3	H-1	H-2	H-3
.0625			29	14.7	7.6	14.0	9.9	9.4	12.0
.0442			58	25.1	12.3	22.5	15.5	17.2	22.5
.0312		1	56	36.0	26.5	28.2	20.6	27.2	30.4
.0221		3	52	47.3	42.6	38.4	30.7	36.7	37.7
.0156		7	44	57.7	54.9	45.1	40.7	47.4	46.4
.0110		15		62.8	64.2	53.3	47.0	57.7	55.3
.0078		31		65.1	69.7	59.0	52.6	68.9	66.5
.0055		1		68.9	76.9	63.3	60.6	77.9	75.9
.0039		3		71.0	79.7	67.6	71.6	85.0	82.1
.00276		5		71.6	80.3	71.8	76.7	90.4	87.1
.00195		10		72.5	85.0	76.3	80.8	96.9	91.3
.00138		21		73.4	86.9	77.5	83.4	98.7	94.4
.00098		42		74.3		80.7	86.5	100.9	98.0
.00069		24				80.7	87.5	102.2	99.0
.00049		50				82.7	88.4	103.2	100.3

and clay particles have been separated after 10 minutes of treatment. What is indicated by the trend in sample A, the Marshall sandstone, is not known. Sample B, the glazed argillaceous shale, and sample F, the siltstone, do not show any definite trend. Lack of any definite trend indicates that disaggregation has not begun to any large degree. Sample G, the carbonaceous shale, shows that disaggregation may have begun after the treatment exceeded 10 minutes.

The wide variations in total percentage in Tables IV and V were probably due to unavoidable errors in the unorthodox technique employed for the pipette analysis. However, even though the technique employed was unorthodox due to the extremely small size of the sample and the lack of available information on executing pipette analyses on such small samples, the author feels that the data obtained may be at least partially indicative of the true results obtained with this disaggregation method.

Mortar and Pestle Method

Equipment

The equipment used in this method consisted of a large steel mortar and pestle.

Disaggregation

The rock used in this method was sample C, the bituminous shale from Leroy, New York. The rock was pulverized in the mortar and then a sample of 25.000 grams was taken.

Pipette Analysis

The sample was both dry sieved and wet sieved through the 230 sieve to separate the sand particles from the silt and clay particles. The wet sieving was continued until all the silt and clay particles were washed through the sieve. The part of the sample which did not pass through the sieve was dried, weighed, and recorded (Table VII). That portion of the sample which passed through the sieve was washed into a 1000 ml graduated cylinder. To minimize coagulation 0.670 grams of sodium oxalate was added to the solution and enough water was added to make exactly 1000 ml. The procedure determined by Stokes' Law for a pipette analysis was followed.

The suspension was agitated thoroughly, and after the designated time period a 20 cc pipette was inserted to the designated depth and exactly 20 cc of the solution was withdrawn. This was dried in a beaker of predetermined weight. The weight of the material in the beaker was recorded. This weight represents one-fiftieth of the amount of material of a certain diameter present in the suspension. This process was repeated for each of the times designated by Stokes' Law.

TABLE VII

MORTAR AND PESTLE METHOD

PIPETTE ANALYSIS

SAMPLE C

Diameter in Millimeters	Height in Centimeters	Time			Uncorrected Weight in Grams	Corrected Weight in Grams	Corrected Weight times 50	Percentage	Cumulative Percentage
		Hr.	Min.	Sec.					
.0625	20			58	.058	.045	2.250	11.6	11.6
.0442	20		1	56	.059	.046	2.300	11.8	23.4
.0312	10		1	56	.051	.038	1.900	9.8	33.2
.0221	10		3	52	.050	.037	1.850	9.5	42.7
.0156	10		7	44	.046	.033	1.650	8.5	51.2
.0110	10		15		.058	.045	2.250	11.6	62.8
.0078	10		31		.041	.028	1.400	7.2	70.0
.0055	10	1	1		.041	.028	1.400	7.2	77.2
.0039	10	2	3		.033	.020	1.000	5.1	82.3
.00276	10	4	5		.033	.020	1.000	5.1	87.4
.00195	10	8	10		.027	.014	0.700	3.6	91.0
.00138	10	16	21		.023	.010	0.500	2.6	93.6
.00098	5	16	21		.023	.010	0.500	2.6	96.2
.00069	5	32	42		.023	.010	0.500	2.6	98.8
.00049	5	65	25		.019	.006	0.300	1.5	100.3
								100.3	

Weight of Sample larger than .0625 mm	Percentage of Sample larger than .0625 mm
5.539	22.2

Original sample weight was 25.000 grams.

Data Analysis

The data for this method is shown on Table VII. The uncorrected weight in grams was obtained by weighing the beakers of predetermined weight which contained the various sizes of sample. In the pipette analysis 0.670 grams of sodium oxalate was added to the graduated cylinder to minimize coagulation. Because of this addition, the weights had to be corrected. This was done by subtracting 0.013 grams from each of the uncorrected weights. The corrected weights were each multiplied by 50 because the 20 ml sample taken represented one-fiftieth of the total suspended material. The percentages represent the percent of silt and clay only. The cumulative percentages were obtained by adding each of the percentages to those above it in the table.

Data Interpretation

The percentage of the sample larger than 0.0625 mm is still quite large, which indicates that disaggregation by this method has not been very successful.

Oak Board Method

Equipment

The equipment used in this method consisted of a hand jaw crusher and two oak boards. The machine was made of two grooved plates, one solidly fastened and the other attached to an eccentric camshaft. Turning the crank produced a back and forth movement of the plate attached to the camshaft. There was very little up and down movement of the plate. It was felt that the use of this machine in crushing the rock would result in very little attrition. The oak boards were two pieces four inches wide, twelve inches long, and about three-quarters of an inch thick. The two largest surfaces on each board were sanded until they were smooth.

Disaggregation

The rock used in this method was sample C, the bituminous shale from Leroy, New York. The rock was first crushed in the jaw crusher. After crushing with this machine, the largest remaining particles were thin, platy-shaped, and approximately one-quarter of an inch long and one-eighth of an inch wide. The rock was then placed between the large flat surfaces of the oak boards, pressure was applied and as little rotary motion as possible was used to further disaggregate the rock. A 16.000 gram sample was then weighed out.

Pipette Analysis

The procedure followed here was the same as that of the pipette analysis of the mortar and pestle method (Table VIII).

TABLE VIII

OAK BLOCK METHOD

PIPETTE ANALYSIS

SAMPLE C

Diameter in Millimeters	Height in Centimeters	Time			Uncorrected Weight in Grams	Corrected Weight in Grams	Corrected Weight times 50	Percentage	Cumulative Percentage
		Hr.	Min.	Sec.					
.0625	20			58	.048	.035	1.750	13.8	13.8
.0442	20		1	56	.047	.034	1.700	13.4	27.2
.0312	10		1	56	.042	.029	1.450	11.4	38.6
.0221	10		3	52	.042	.029	1.450	11.4	50.0
.0156	10		7	44	.036	.023	1.150	8.6	58.6
.0110	10		15		.035	.022	1.100	8.6	67.2
.0078	10		31		.029	.016	0.800	6.3	73.5
.0055	10	1	1		.027	.014	0.700	5.5	79.0
.0039	10	2	3		.023	.015	0.750	5.9	84.9
.00276	10	4	5		.021	.008	0.400	3.1	88.0
.00195	10	8	10		.021	.008	0.400	3.1	91.1
.00138	10	16	21		.021	.008	0.400	3.1	94.2
.00098	5	16	21		.017	.004	0.200	1.6	95.8
.00069	5	32	42		.018	.005	0.250	2.0	97.8
.00049	5	65	25		.018	.005	0.250	2.0	99.8
								99.8	

Weight of Sample larger than 0.0625 mm
3.267

Percentage of Sample larger than 0.0625 mm
20.4

Original sample weight was 16.000 grams.

Data Analysis

The procedure followed was the same as the procedure in the data analysis of the mortar and pestle method (Page 19).

Data Interpretation

The percentage of sample larger than 0.0625 mm is 20.4 percent of the original sample weight, which indicates that disaggregation by this method has not been very successful.

CHEMICAL METHODS

Introduction

For the chemical methods of disaggregation it was decided to use potassium hydroxide and perchloric acid, two methods which have been utilized before (Ross, 1957 and Kropschot, 1953). It was felt that these previously used methods would provide the necessary basis for a sound comparison of disaggregation techniques.

Potassium Hydroxide Method

Sample Preparation

All samples to be chemically disaggregated were prepared by crushing them down to a maximum diameter of approximately one-sixteenth of an inch with a hammer. The weight of the sample used depended on the amount of rock available (Table IX). The amount of crushed sample available was divided approximately in half so as to insure having enough left to use in the perchloric acid method.

The potassium hydroxide solution was prepared by using a ratio of ten grams of dry potassium hydroxide and ten ml of water to each gram of sample (Ross, 1957). The solution was then placed in a 600 ml beaker and the sample was added

TABLE IX

POTASSIUM HYDROXIDE METHOD

WEIGHT AND PERCENTAGE OF SAMPLE LARGER THAN 0.0625 mm

Sample	Original Sample Weight in grams	Weight of Sample Larger than 0.0625 mm in grams	Percentage Larger than 0.0625 mm
A	30.000	16.960	56.5
B	25.000	7.330	29.3
C	30.000	13.479	45.0
D	25.000	10.996	44.0
E	30.000	1.473	4.9
F	30.000	16.140	53.8
G	20.000	7.065	35.3
H	20.000	13.535	67.8

to the solution. In some cases there was a mild effervescence when the sample was added.

Disaggregation

The disaggregation method consisted of heating the beaker and contents slowly until most of the liquid was driven off, then adding more water and evaporating that. Water was added to each beaker several times. This process took about 24 hours of slow heating. The beakers containing the sample were weighed both before and after the disaggregation to determine if a part of the beaker had been taken into solution. No appreciable difference in weight was noted.

Pipette Analysis

The procedure followed was the same as that used in the pipette analysis of the mortar and pestle method, except that the sample was wet sieved only (Table X).

Data Analysis

The procedure followed was the same as the procedure described in the data analysis of the mortar and pestle method.

TABLE X

POTASSIUM HYDROXIDE METHOD

UNCORRECTED WEIGHT IN GRAMS

Diameter in Millimeters	Height in Centimeters	Time			A	B	C	D	E	F	G	H
		Hr.	Min.	Sec.								
.0625	20			58	.118	.065	.090	.044	.112	.094	.068	.024
.0442	20		1	56	.080	.049	.084	.043	.112	.066	.064	.024
.0312	10		1	56	.039	.039	.058	.042	.092	.053	.061	.023
.0221	10		3	52	.024	.040	.040	.043	.064	.031	.062	.020
.0156	10		7	44	.026	.039	.036	.043	.063	.037	.018	.021
.0110	10		15		.025	.038	.035	.024	.051	.033	.063	.021
.0078	10		31		.022	.040	.030	.021	.049	.023	.011	.020
.0055	10	1	1		.018	.034	.026	.026	.042	.022	.015	.021
.0039	10	2	3		.016	.037	.023	.026	.033	.018		.021
.00276	10	4	5		.017	.035	.019	.026	.030	.016		.024
.00195	10	8	10		.018	.031	.015	.027	.028	.018		.021
.00138	10	16	21			.028	.015	.027	.026			.021
.00098	5	16	21			.024	.020	.027	.023			.021
.00069	5	32	42			.023	.017	.028	.021			.021
.00049	5	65	25			.025	.016	.029	.019			.020

TABLE XI

POTASSIUM HYDROXIDE METHOD

CORRECTED WEIGHT IN GRAMS

Diameter in Millimeters	Height in Centimeters	Time										
		Hr.	Min.	Sec.	A	B	C	D	E	F	G	H
.0625	20			58	.105	.052	.077	.031	.099	.081	.055	.011
.0442	20		1	56	.067	.036	.071	.030	.099	.053	.051	.011
.0312	10		1	56	.026	.026	.045	.029	.079	.040	.048	.010
.0221	10		3	52	.011	.027	.027	.030	.051	.028	.049	.007
.0156	10		7	44	.013	.026	.023	.030	.050	.024	.005	.008
.0110	10		15		.012	.025	.022	.011	.038	.020	.050	.008
.0078	10		31		.009	.027	.017	.008	.036	.010	.000	.007
.0055	10	1	1		.005	.021	.013	.013	.029	.009	.002	.008
.0039	10	2	3		.003	.024	.010	.013	.020	.005		.008
.00276	10	4	5		.004	.022	.006	.013	.017	.003		.011
.00195	10	8	10		.005	.018	.002	.014	.015	.005		.008
.00138	10	16	21			.015	.002	.014	.013			.008
.00098	5	16	21			.011	.007	.014	.010			.008
.00069	5	32	42			.010	.004	.015	.008			.008
.00049	5	65	25			.012	.003	.016	.006			.007

TABLE XII

POTASSIUM HYDROXIDE METHOD

CORRECTED WEIGHT IN GRAMS TIMES 50

Diameter in Millimeters	Height in Centimeters	Time										
		Hr.	Min.	Sec.	A	B	C	D	E	F	G	H
.0625	20			58	5.250	2.600	3.850	1.550	4.950	4.050	2.750	.055
.0442	20		1	56	3.350	1.800	3.550	1.500	4.950	2.650	2.550	.055
.0312	10		1	56	1.300	1.300	2.250	1.450	3.950	2.000	2.400	.050
.0221	10		3	52	0.550	1.350	1.350	1.500	2.550	1.400	2.450	.035
.0156	10		7	44	0.650	1.300	1.150	1.500	2.500	1.200	0.250	.040
.0110	10		15		0.600	1.250	1.100	0.550	1.900	1.000	2.500	.040
.0078	10		31		0.450	1.350	0.850	0.400	1.800	0.500	0.000	.035
.0055	10	1	1		0.250	1.050	0.650	0.650	1.450	0.450	0.100	.040
.0039	10	2	3		0.150	1.200	0.500	0.650	1.000	0.250		.040
.00276	10	4	5		0.200	1.100	0.300	0.650	0.850	0.150		.055
.00195	10	8	10		0.250	0.900	0.100	0.700	0.750	0.250		.040
.00138	10	16	21			0.750	0.100	0.700	0.650			.040
.00098	5	16	21			0.550	0.350	0.700	0.500			.040
.00069	5	32	42			0.500	0.200	0.750	0.400			.040
.00049	5	65	25			0.600	0.150	0.800	0.300			.035

Data Interpretation

Upon examining the percentage larger than 0.0625 mm as shown in Table IX, it is evident that disaggregation was much more successful on sample E, the petroliferous shale, than any of the other samples. The other samples still have a high percentage of sand particles which indicates that disaggregation was not complete when using this method.

The variation in percentages in Tables XIII and XIV is little; the largest error is one percent.

Perchloric Acid Method

Sample Preparation

All of the samples used in this process were prepared by crushing them with a hammer to a maximum diameter of approximately one-sixteenth of an inch. The weight of the sample used varied, depending on the amount of rock left after dividing the sample in half for use in the potassium hydroxide treatment (Table XV).

The solution used in this method consisted of one ml of perchloric acid, one ml of sulfuric acid, and 10 ml of nitric acid to each gram of shale (Kropschot, 1953). This approximate ratio must be maintained to prevent the precipitation of calcium sulphate. This solution was placed in a 600 ml beaker and the sample was added to the solution.

TABLE XIII

POTASSIUM HYDROXIDE METHOD

PERCENTAGE

Diameter in Millimeters	Height in Centimeters	Time Hr.	Time Min.	Time Sec.	A	B	C	D	E	F	G	H
.0625	20			58	40.3	14.7	23.3	11.1	17.4	29.2	21.3	8.5
.0442	20		1	56	25.7	10.4	21.5	10.7	17.4	19.1	19.7	8.5
.0312	10		1	56	10.0	7.4	13.6	10.4	13.9	14.4	18.6	7.7
.0221	10		3	52	4.2	7.6	8.2	10.7	8.9	10.1	19.0	5.4
.0156	10		7	44	5.0	7.4	7.0	10.7	8.8	8.7	1.9	6.2
.0110	10		15		4.6	7.1	6.7	3.9	6.7	7.2	19.3	6.2
.0078	10		31		3.4	7.6	5.1	2.9	6.3	3.6	0.0	5.4
.0055	10	1	1		1.9	5.9	3.9	4.6	5.1	3.2	0.8	6.2
.0039	10	2	3		1.2	6.8	3.0	4.6	3.5	1.8		6.2
.00276	10	4	5		1.5	6.2	1.8	4.6	3.0	1.1		8.5
.00195	10	8	10		1.9	5.1	0.6	5.0	2.6	1.5		6.2
.00138	10	16	21			4.2	0.6	5.0	2.4			6.2
.00098	5	16	21			3.1	2.1	5.0	1.8			6.2
.00069	5	32	42			3.4	1.2	5.4	1.4			6.2
.00049	5	65	25			2.8	0.9	5.7	1.1			5.4
					99.7	99.7	99.5	100.3	100.3	100.2	100.6	99.0

TABLE XIV

POTASSIUM HYDROXIDE METHOD

CUMULATIVE PERCENTAGE

Diameter in Millimeters	Height in Centimeters	Time			A	B	C	D	E	F	G	H
		Hr.	Min.	Sec.								
.0625	20			58	40.3	14.7	23.3	11.1	17.4	29.2	21.3	8.5
.0442	20		1	56	66.0	25.1	44.8	21.8	34.8	48.3	41.0	17.0
.0312	10		1	56	76.0	32.5	58.4	32.2	48.7	62.7	59.6	24.7
.0221	10		3	52	80.2	40.1	66.6	42.9	57.6	72.8	78.6	30.1
.0156	10		7	44	85.2	47.5	73.6	53.6	66.4	81.5	80.5	36.3
.0110	10		15		89.8	54.6	80.3	57.5	73.1	88.7	99.8	42.5
.0078	10		31		93.2	62.2	85.4	60.4	79.4	92.3	99.8	47.9
.0055	10	1	1		95.1	68.1	89.3	65.0	84.5	95.5	100.6	54.1
.0039	10	2	3		96.3	74.9	92.3	69.6	88.0	97.3		60.3
.00276	10	4	5		97.8	81.1	94.1	74.2	91.0	98.4		68.8
.00195	10	8	10		99.7	86.2	94.7	79.2	93.6	100.2		75.0
.00138	10	16	21			90.4	95.3	84.2	96.0			81.2
.00098	5	16	21			93.5	97.4	89.2	97.8			87.4
.00069	5	32	42			96.9	98.6	94.6	99.2			93.6
.00049	5	65	25			99.7	99.5	100.3	100.3			99.0

TABLE XV

PERCHLORIC ACID METHOD

WEIGHT AND PERCENTAGE OF SAMPLE LARGER THAN 0.0625 mm

Sample	Original Sample Weight in grams	Weight of Sample Larger than 0.0625 mm in grams	Percentage Larger than 0.0625 mm
A	30.000	5.965	19.9
B	30.000	2.407	8.0
C	30.000	1.211	4.0
D	24.000	1.362	5.7
E	25.000	5.674	22.7
F	25.000	5.980	23.9
G	20.000	0.046	2.3
H	18.000	1.723	9.6

Disaggregation

The disaggregation method consisted of heating the beaker and contents slowly. The perchloric acid was given off as a white vapor and the nitric acid was given off as nitrogen dioxide. It was essential to use a hood with a strong exhaust system to draw off the fumes. When there was very little liquid remaining, water was added to the beaker and allowed to evaporate. Water was added to each beaker several times. This process took about 72 hours of slow heating to complete.

Pipette Analysis

The procedure followed was the same as the procedure used in the pipette analysis following disaggregation by the mortar and pestle method, except that the sample was wet sieved only (Table XVI).

Data Analysis

The procedure followed was the same as that described in the data analysis of the mortar and pestle method.

Data Interpretation

Upon examining the percentage larger than 0.0625 mm in Table XV, it is evident that disaggregation was not very successful on sample E, the petroliferous shale. Sample A,

TABLE XVI

PERCHLORIC ACID METHOD

UNCORRECTED WEIGHT IN GRAMS

Diameter in Millimeters	Height in Centimeters	Time			A	B	C	D	E	F	G	H
		Hr.	Min.	Sec.								
.0625	20			58	.054	.082	.083	.080	.056	.061	.061	.062
.0442	20		1	56	.052	.073	.145	.077	.065	.059	.064	.063
.0312	10		1	56	.049	.078	.090	.073	.064	.042	.062	.056
.0221	10		3	52	.052	.072	.051	.068	.060	.052	.054	.035
.0156	10		7	44	.053	.058	.071	.056	.057	.053	.059	.043
.0110	10		15		.057	.052	.034	.049	.050	.038	.059	.043
.0078	10		31		.045	.042	.042	.038	.030	.049	.042	.027
.0055	10	1	1		.040	.041	.048	.035	.040	.038	.028	.034
.0039	10	2	3		.040	.040	.045	.030	.035	.030	.022	.032
.00276	10	4	5		.038	.034	.037	.029	.027	.031	.022	.027
.00195	10	8	10		.039	.030	.028	.028	.025	.026	.022	.023
.00138	10	16	21		.040	.044	.028	.022	.018	.025	.025	.020
.00098	5	16	21		.040	.036	.021	.019	.019	.023	.026	.020
.00069	5	32	42		.039	.029	.020	.020	.019	.024	.024	.019
.00049	5	65	25		.038	.026	.027	.023	.017	.025	.024	.017

TABLE XVII

PERCHLORIC ACID METHOD

CORRECTED WEIGHT IN GRAMS

Diameter in Millimeters	Height in Centimeters	Time			A	B	C	D	E	F	G	H
		Hr.	Min.	Sec.								
.0625	20			58	.041	.069	.070	.067	.043	.048	.048	.049
.0442	20		1	56	.039	.020	.132	.064	.052	.046	.051	.050
.0312	10		1	56	.036	.065	.077	.060	.051	.029	.049	.043
.0221	10		3	52	.039	.059	.038	.055	.047	.039	.041	.022
.0156	10		7	44	.040	.045	.058	.043	.044	.040	.046	.030
.0110	10		15		.044	.039	.021	.036	.037	.025	.046	.030
.0078	10		31		.032	.029	.029	.025	.017	.036	.029	.014
.0055	10	1	1		.027	.028	.035	.022	.027	.025	.015	.021
.0039	10	2	3		.027	.037	.032	.017	.022	.017	.009	.019
.00276	10	4	5		.025	.021	.024	.016	.014	.018	.009	.014
.00195	10	8	10		.026	.017	.015	.015	.012	.013	.009	.010
.00138	10	16	21		.027	.031	.015	.009	.005	.012	.012	.007
.00098	5	16	21		.027	.023	.008	.006	.006	.010	.013	.007
.00069	5	32	42		.026	.016	.007	.007	.006	.011	.011	.006
.00049	5	65	25		.025	.013	.014	.010	.004	.012	.011	.004

TABLE XVIII

PERCHLORIC ACID METHOD

CORRECTED WEIGHT TIMES 50

Diameter in Millimeters	Height in Centimeters	Time			A	B	C	D	E	F	G	H
		Hr.	Min.	Sec.								
.0625	20			58	2.050	3.450	3.500	3.350	2.150	2.400	2.400	2.450
.0442	20		1	56	1.950	3.000	6.600	3.200	2.600	2.300	2.550	2.500
.0312	10		1	56	1.800	3.250	3.850	3.000	2.550	1.450	2.450	2.150
.0221	10		3	52	1.950	2.950	1.900	2.750	2.350	1.950	2.050	1.100
.0156	10		7	44	2.000	2.250	2.900	2.150	2.200	2.000	2.300	1.500
.0110	10		15		2.200	1.950	1.050	1.800	1.850	1.250	2.300	1.500
.0078	10		31		1.600	1.450	1.450	1.250	0.850	1.800	2.450	0.700
.0055	10	1	1		1.350	1.400	1.750	1.100	1.350	1.250	0.750	1.050
.0039	10	2	3		1.350	1.850	1.600	0.850	1.100	0.850	0.450	0.950
.00276	10	4	5		1.250	1.050	1.200	0.800	0.700	0.900	0.450	0.700
.00195	10	8	10		1.300	0.850	0.750	0.750	0.600	0.650	0.450	0.500
.00138	10	16	21		1.350	1.550	0.750	0.450	0.250	0.600	0.600	0.350
.00098	5	16	21		1.350	1.150	0.400	0.300	0.300	0.500	0.650	0.350
.00069	5	32	42		1.300	0.800	0.350	0.350	0.300	0.550	0.550	0.300
.00049	5	65	25		1.250	0.650	0.700	0.500	0.200	0.600	0.550	0.200

the Marshall sandstone, and sample F, the siltstone, still have a high percentage of material larger than 0.0625 mm, but since these two rocks are coarser grained, disaggregation may be complete. All of the remainder of the samples have a small percentage of sand particles, which indicates that disaggregation has been successful.

The variation in percentages as shown in Tables XIX and XX is little with the exception of sample G where the error is slightly over five percent.

TABLE XIX

PERCHLORIC ACID METHOD

PERCENTAGE

Diameter in Millimeters	Height in Centimeters	Time			A	B	C	D	E	F	G	H
		Hr.	Min.	Sec.								
.0625	20			58	8.5	12.5	12.1	14.8	11.1	12.6	12.0	15.0
.0442	20		1	56	8.1	10.9	22.8	14.1	13.5	12.1	12.8	15.3
.0312	10		1	56	7.5	11.8	13.3	13.3	13.2	7.8	12.3	13.2
.0221	10		3	52	8.1	10.7	6.6	12.2	12.2	10.3	10.3	6.8
.0156	10		7	44	8.3	8.2	10.0	9.5	11.4	10.6	11.5	9.2
.0110	10		15		9.1	7.1	3.6	8.0	9.6	6.6	11.5	9.2
.0078	10		31		6.6	5.3	5.0	5.5	4.4	9.5	12.3	4.3
.0055	10	1	1		5.6	5.1	6.1	4.9	7.0	6.6	3.8	6.4
.0039	10	2	3		5.6	6.7	5.5	3.8	5.7	4.5	2.3	5.8
.00276	10	4	5		5.2	3.8	4.2	3.5	3.6	5.2	2.3	4.3
.00195	10	8	10		5.4	3.1	2.6	3.3	3.1	3.4	2.3	3.1
.00138	10	16	21		5.6	5.6	2.6	2.0	1.3	3.2	3.0	2.1
.00098	5	16	21		5.6	4.2	1.4	1.3	1.6	2.6	3.3	2.1
.00069	5	32	42		5.4	2.9	1.2	1.5	1.6	2.9	2.8	1.8
.00049	5	65	25		5.2	2.4	2.4	2.2	1.0	3.2	2.8	1.2
					99.8	100.3	99.4	99.9	100.3	101.1	105.3	99.8

TABLE XX

PERCHLORIC ACID METHOD

CUMULATIVE PERCENTAGE

Diameter in Millimeters	Height in Centimeters	Time Hr.	Time Min.	Time Sec.	A	B	C	D	E	F	G	H
.0625	20			58	8.5	12.5	12.1	14.8	11.1	12.6	12.0	15.0
.0442	20		1	56	16.6	23.4	34.9	28.9	24.6	24.7	24.8	30.3
.0312	10		1	56	24.1	35.2	48.2	42.2	37.8	32.5	37.1	43.5
.0221	10		3	52	32.2	45.9	54.8	54.4	50.0	42.8	47.4	50.3
.0156	10		7	44	40.5	54.1	64.8	63.9	61.4	53.4	58.9	59.5
.0110	10		15		49.6	61.2	68.4	71.9	71.0	60.0	70.4	68.7
.0078	10		31		56.2	66.5	73.4	77.4	75.4	69.5	82.7	73.0
.0055	10	1	1		61.8	71.6	79.5	82.3	82.4	76.1	86.5	79.4
.0039	10	2	3		67.4	78.3	85.0	86.1	88.1	80.6	88.8	85.2
.00276	10	4	5		72.6	82.1	89.2	89.6	91.7	85.8	91.1	89.5
.00195	10	8	10		78.0	85.2	91.8	92.9	94.8	89.2	93.4	92.6
.00138	10	16	21		83.6	90.8	94.4	94.9	96.1	92.4	96.4	94.7
.00098	5	16	21		89.2	95.0	95.8	96.2	97.7	95.0	99.7	96.8
.00069	5	32	42		94.6	97.9	97.0	97.7	99.3	97.9	102.5	98.6
.00049	5	65	25		99.8	100.3	99.4	99.9	100.3	101.1	105.3	99.8

CONCLUSIONS

For the purposes of drawing accurate conclusions from the results, we shall consider complete disaggregation to be composed of two parts, primary disaggregation and secondary disaggregation. Primary disaggregation shall be defined as separating the particles larger in diameter than 0.0625 mm (Table XXI). Secondary disaggregation shall be defined as separating the particles smaller in diameter than 0.0625 mm (Refer to the cumulative curves of the samples).

The cumulative curves of sample A rock type show that the perchloric acid method was much more successful in primary disaggregation than any of the other methods. The perchloric acid method was also most effective in secondary disaggregation.

The cumulative curves of sample B show that the perchloric acid method was most successful in primary disaggregation, with the potassium hydroxide method second. The potassium hydroxide method and the perchloric acid method were about equally successful in secondary disaggregation.

The cumulative curves of sample C show that the perchloric acid method and the 15 minute vibration method were equally successful in primary disaggregation. The 10 minute vibration method was most effective in secondary disaggregation with the 15 minute vibration method second.

The perchloric acid method was sixth out of seven in effectiveness.

The cumulative curves of sample D show that all three vibration methods and the perchloric acid method were successful in primary disaggregation. The vibration methods were most successful in secondary disaggregation.

The cumulative curves of sample E show that the 15 minute vibration method was most successful in primary disaggregation. The 5 and 10 minute vibration methods and the potassium hydroxide method were about equally effective, but less so than the 15 minute vibration method. The vibration methods were the most successful in secondary disaggregation with the potassium hydroxide method the least effective.

The cumulative curves of sample F show that the perchloric acid method was much more successful in primary disaggregation than any of the other methods. The three vibration methods and the perchloric acid method were about equally effective in secondary disaggregation.

The cumulative curves of sample G show that the perchloric acid method was much more successful in primary disaggregation than any of the other methods. The three vibration treatments were most successful in secondary disaggregation with the perchloric acid method second.

An examination of the cumulative curves of sample H shows the 10 and 15 minute vibration methods and the perchloric acid method to be about equally successful in primary disaggregation. The cumulative curves show the potassium hydroxide method as the most successful, with the vibration methods second and the perchloric acid method last in secondary disaggregation.

The ultrasonic vibration method was successful in primary disaggregation in four of the shale samples. This method was successful in secondary disaggregation in four of the shales and in the siltstone.

The mortar and pestle method and the oak board method were not successful in either primary or secondary disaggregation.

The potassium hydroxide method was surprisingly unsuccessful. It was successful in primary disaggregation in only one shale sample and in secondary disaggregation in only two shale samples.

The perchloric acid method was the most successful of all the methods in primary disaggregation. It was successful in primary disaggregation in five of the shale samples and in both the sandstone and siltstone samples. This method was successful in secondary disaggregation in two of the shale samples and in both the sandstone and siltstone samples.

On the basis of the above results, the following conclusions can be drawn:

1. If a sandstone or siltstone is to be disaggregated, the perchloric acid method should be used.
2. If a shale is to be fully disaggregated, the ultrasonic vibration method should be used. However, if only primary disaggregation is desired, the perchloric acid method is recommended.

TABLE XXI

PERCENTAGE OF SAMPLE LARGER THAN 0.0625 MILLIMETERS

SAMPLE A
Marshall Sandstone, Michigan

A-1	44.9
A-2	49.2
A-3	54.6
Perchloric acid	19.9
Potassium Hydroxide	56.5

SAMPLE B
Glazed argillaceous shale,
Leroy, New York

B-1	73.3
B-2	77.6
B-3	74.9
Perchloric acid	8.0
Potassium Hydroxide	29.3

SAMPLE C
Bituminous shale,
Leroy, New York

C-1	23.1
C-2	14.6
C-3	4.1
Perchloric acid	4.0
Potassium Hydroxide	45.0
Oak Board	20.4
Mortar and Pestle	22.2

SAMPLE D
Saginaw shale, Michigan

D-1	.8
D-2	.0
D-3	3.2
Perchloric acid	5.7
Potassium Hydroxide	44.0

SAMPLE E
Petroliferous shale, Fossil, Wyo.

E-1	4.6
E-2	4.8
E-3	1.7
Perchloric acid	22.7
Potassium Hydroxide	4.9

SAMPLE F
Siltstone, Mexico

F-1	69.7
F-2	69.8
F-3	68.9
Perchloric acid	23.9
Potassium Hydroxide	53.8

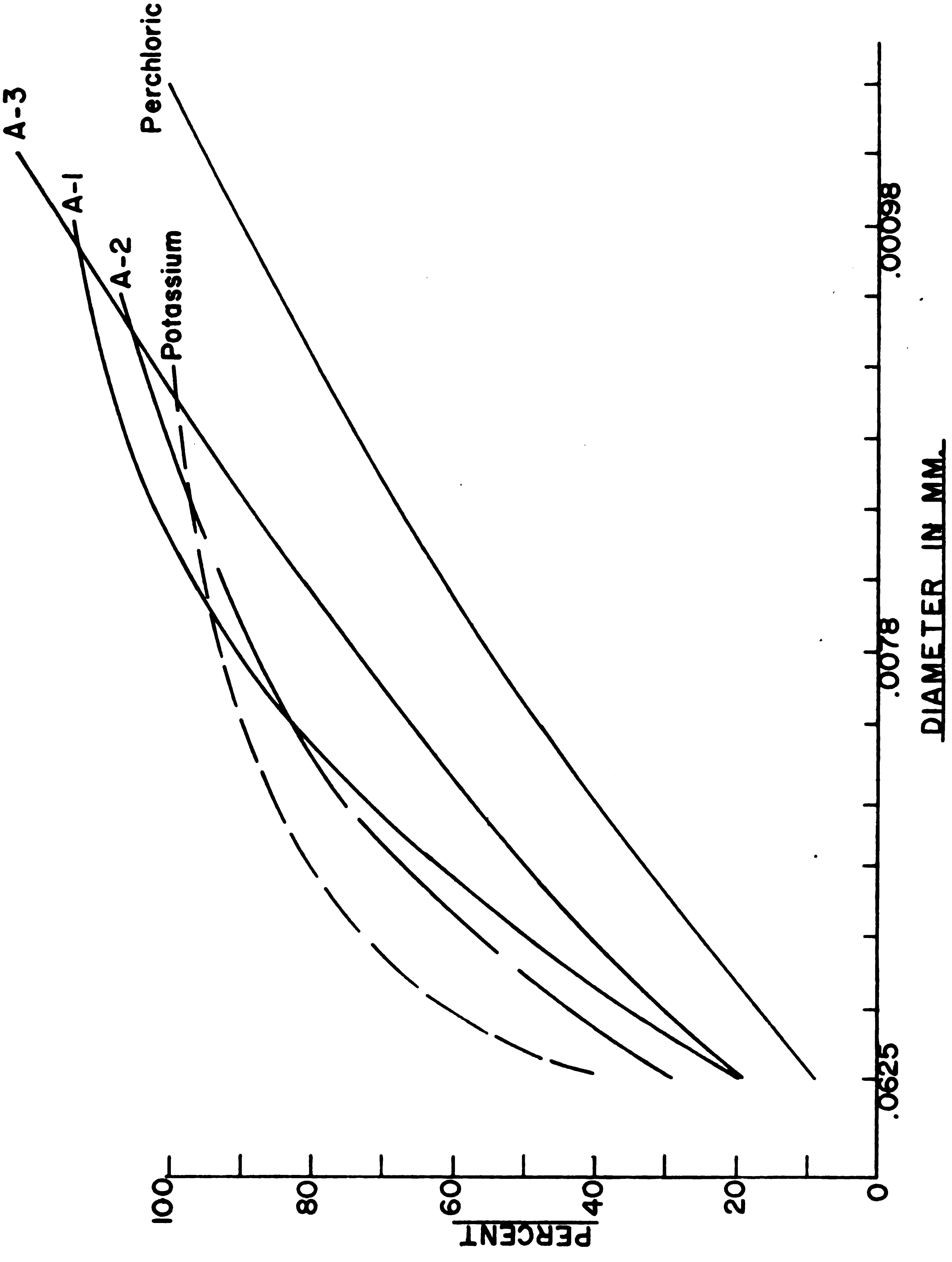
SAMPLE G
Carbonaceous shale,
Moscow, New York

G-1	46.8
G-2	47.1
G-3	39.9
Perchloric acid	2.3
Potassium Hydroxide	35.3

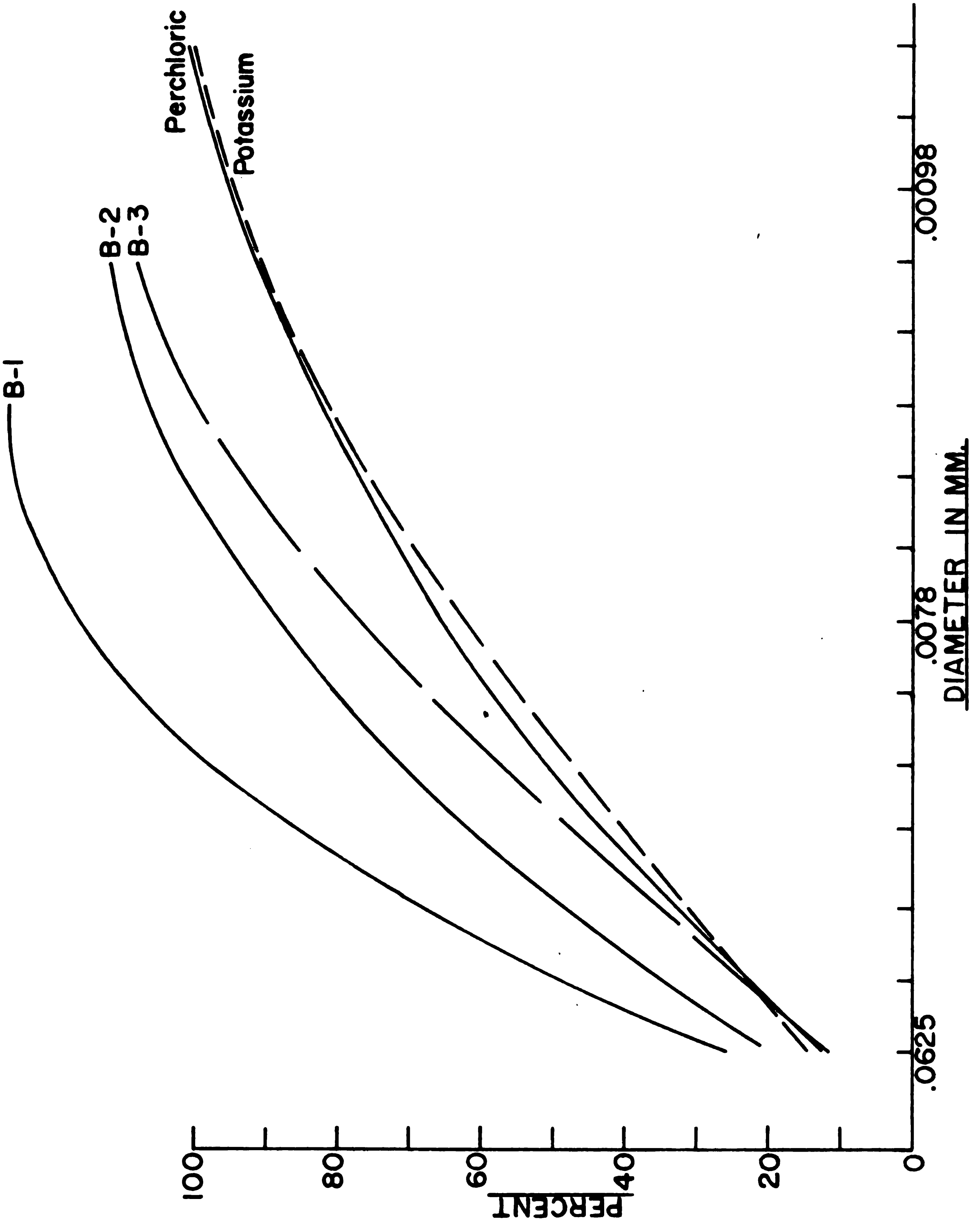
SAMPLE H
Antrim shale, Michigan

H-1	20.1
H-2	9.8
H-3	10.3
Perchloric acid	9.6
Potassium Hydroxide	67.8

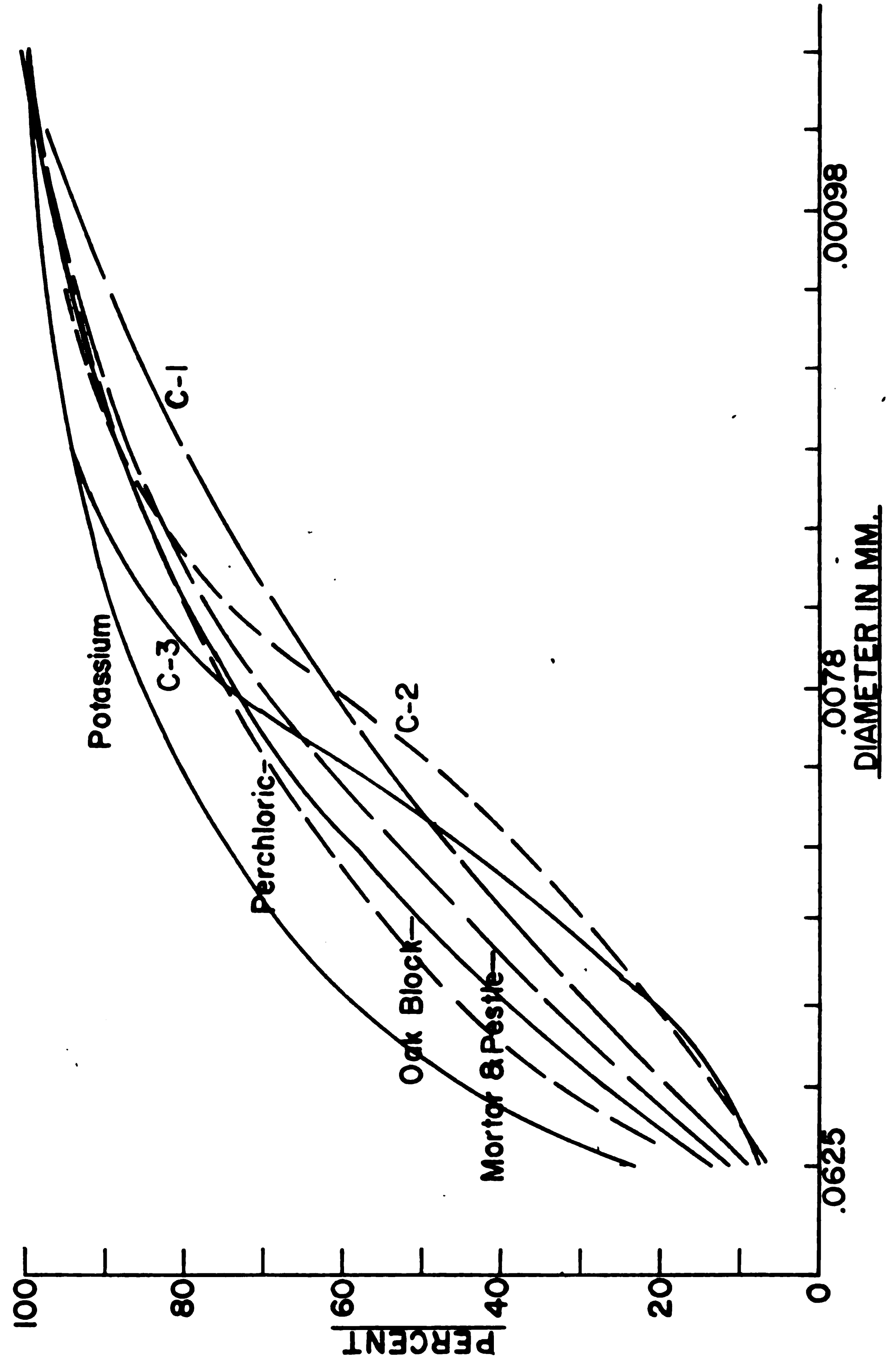
CUMULATIVE CURVES FOR SAMPLE A



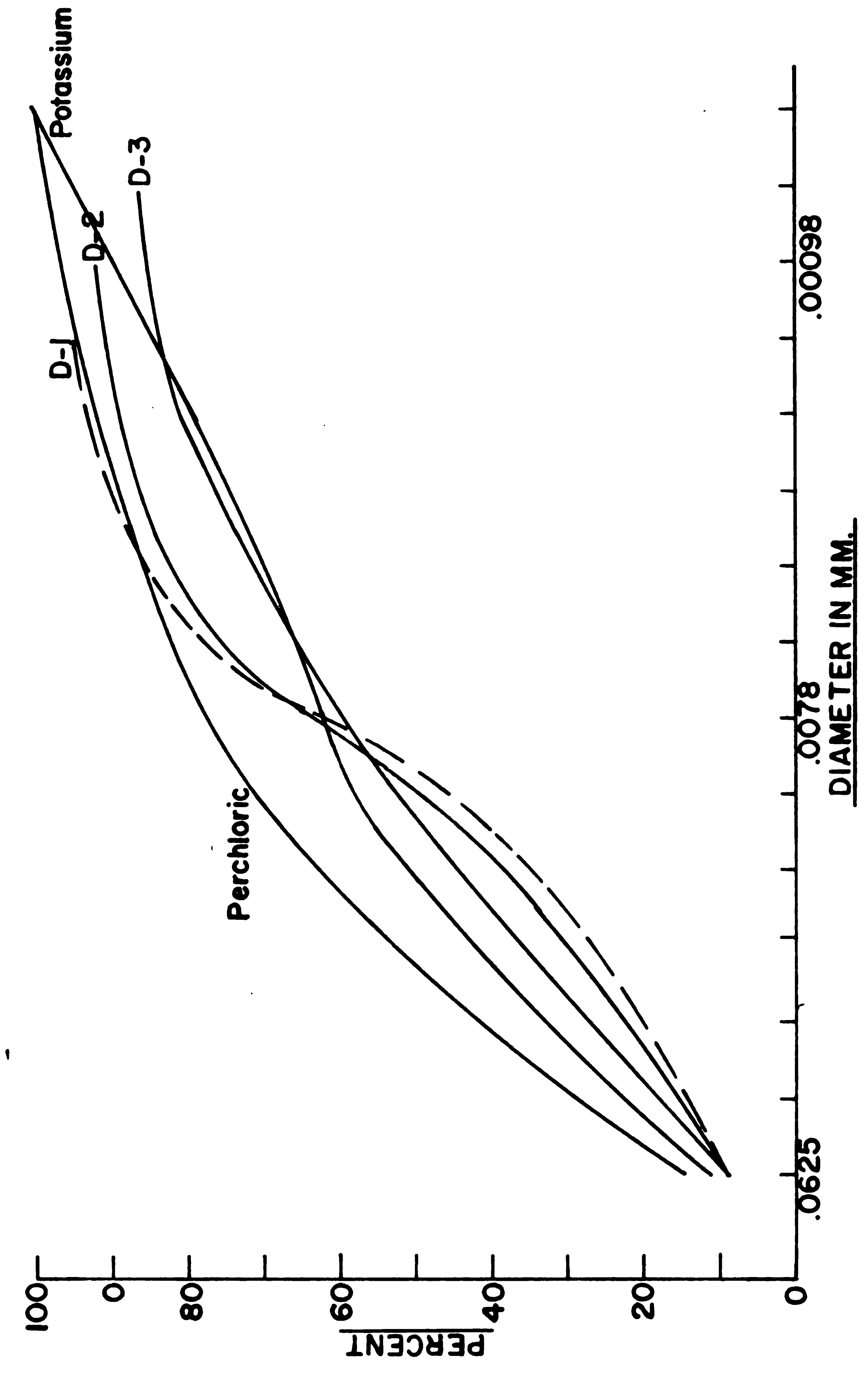
CUMULATIVE CURVES FOR SAMPLE B



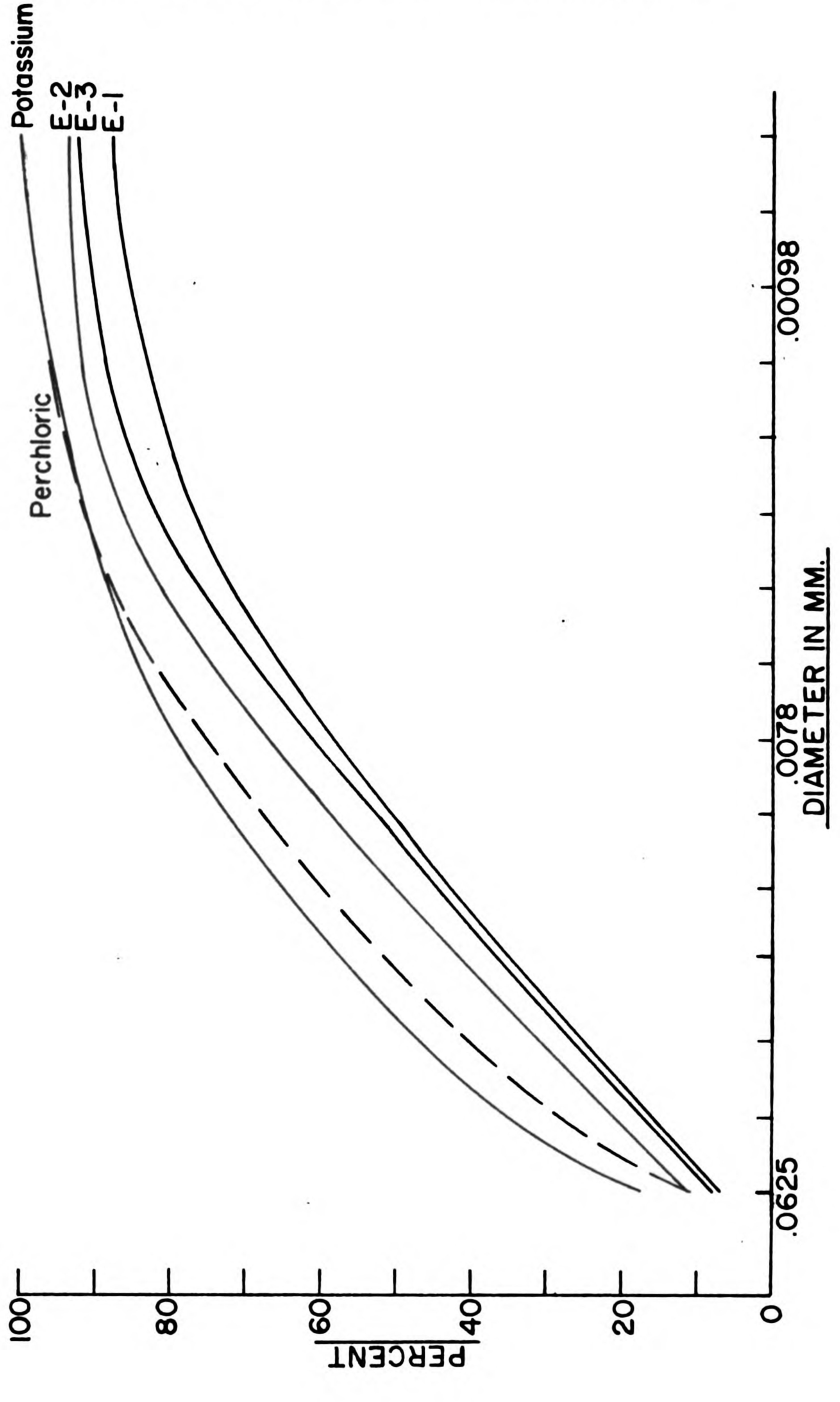
CUMULATIVE CURVES FOR SAMPLE C



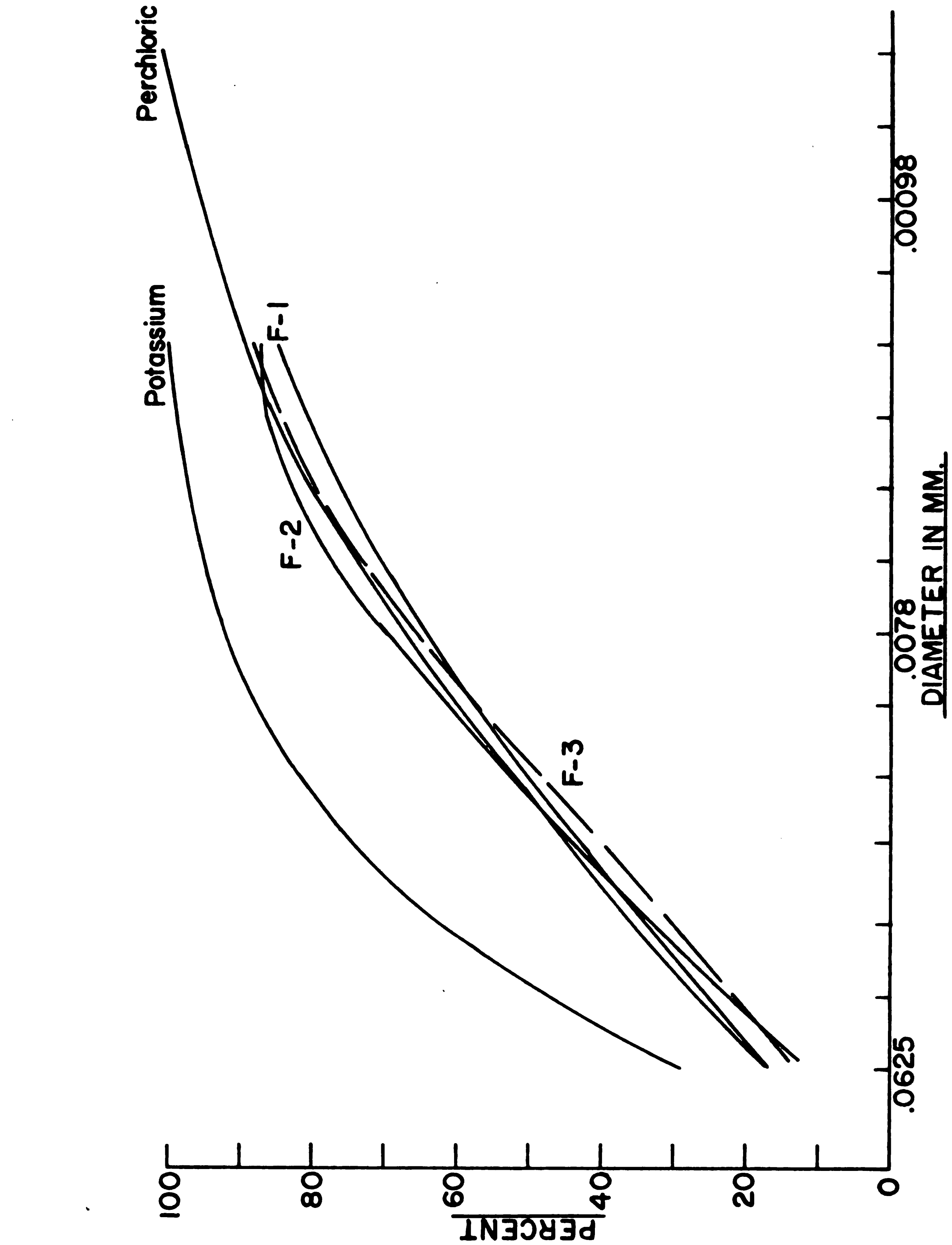
CUMULATIVE CURVES FOR SAMPLE D



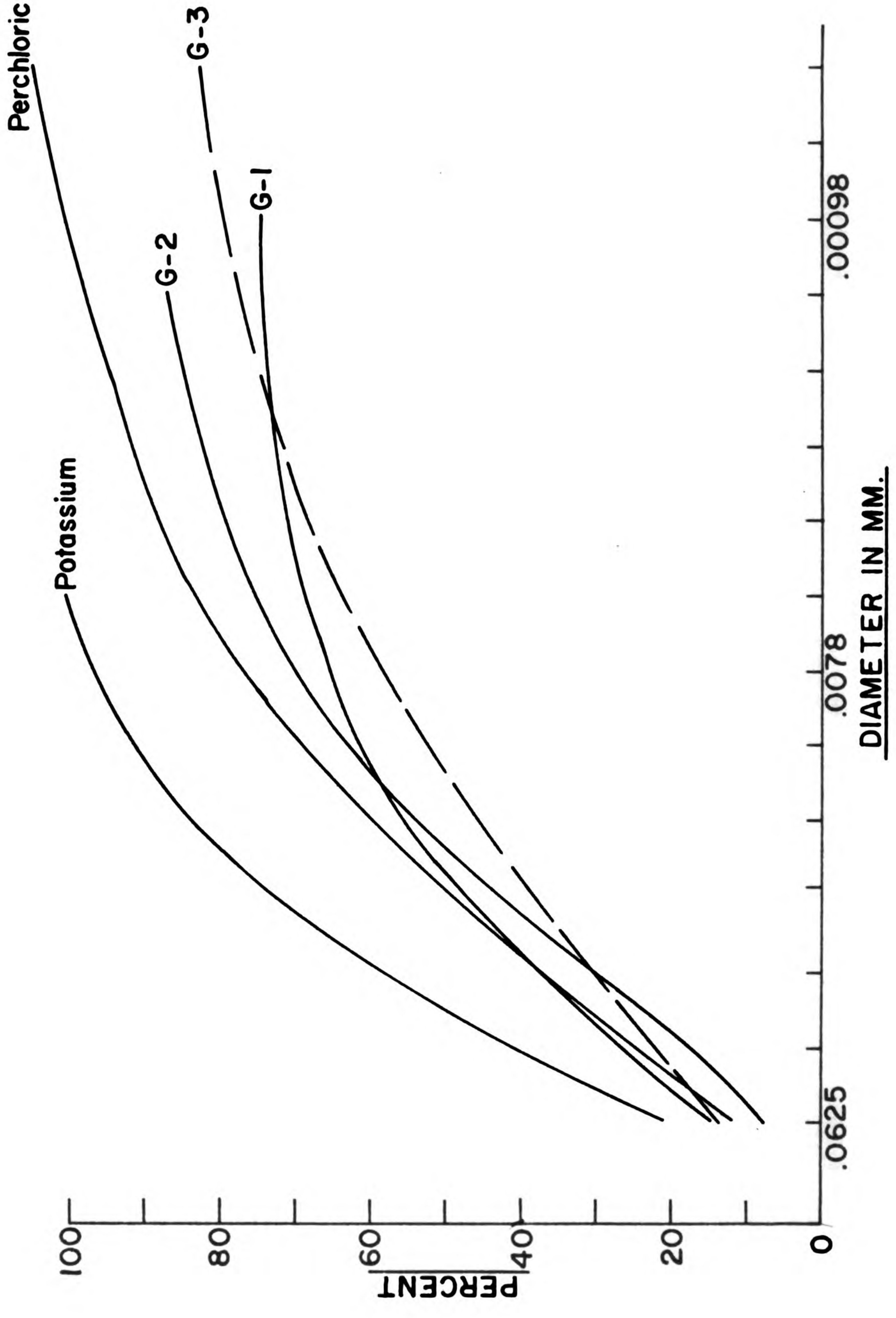
CUMULATIVE CURVES FOR SAMPLE E



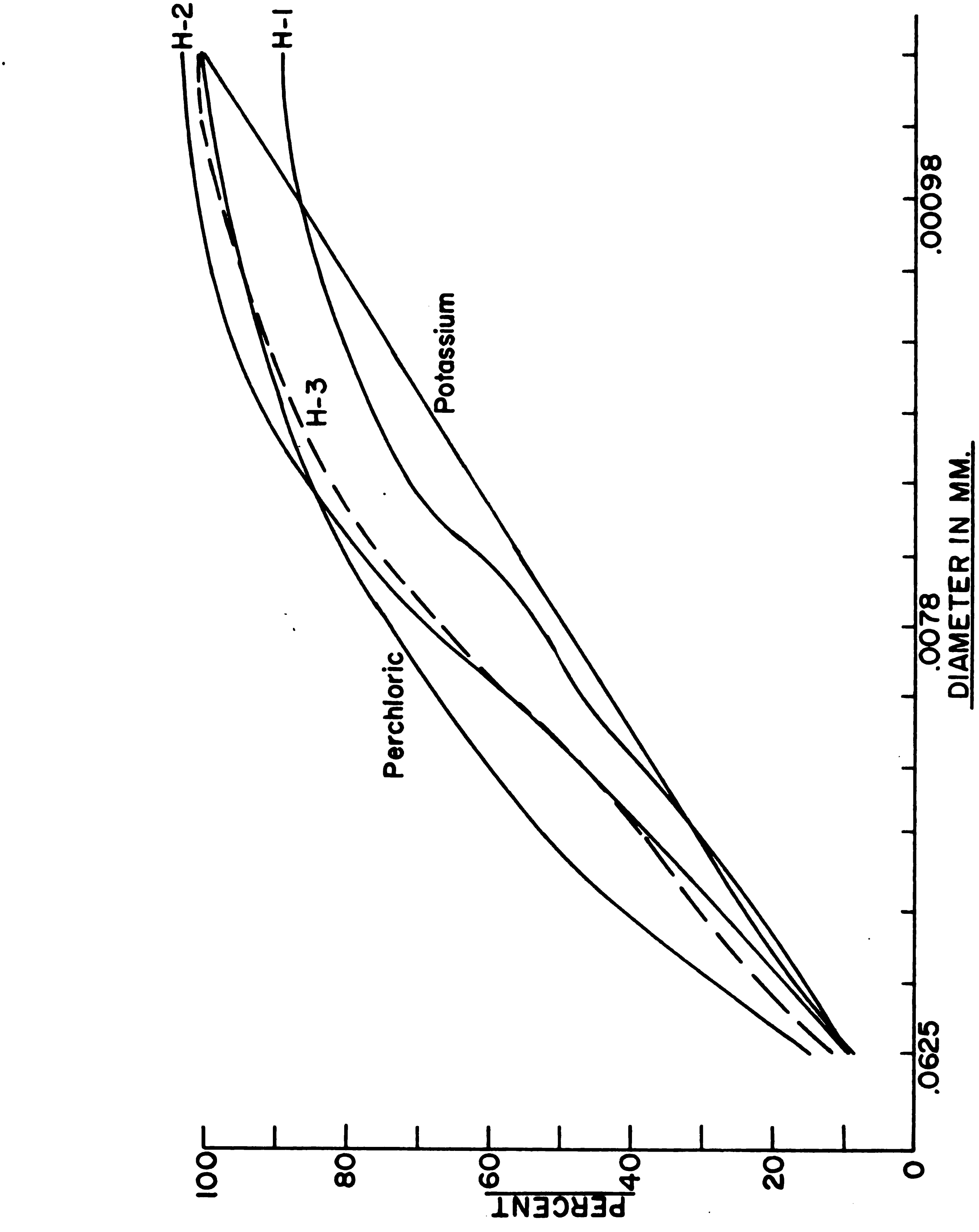
CUMULATIVE CURVES FOR SAMPLE F



CUMULATIVE CURVES FOR SAMPLE G



CUMULATIVE CURVES FOR SAMPLE H



RECOMMENDATIONS

As a result of this research, the author feels there are several further investigations that would be interesting and valuable. These are:

1. The use of the perchloric acid method for primary disaggregation and then the use of ultrasonic vibration for secondary disaggregation may prove to be more successful than either of the methods by themselves.

2. An investigation of more intensive treatment with the ultrasonic vibrator.

3. A study of the application of the ultrasonic vibrator to other fields, such as freeing fossils.

4. Possibly a longer treatment in the oak block and the mortar and pestle method would make these methods successful.

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