NASA Contractor Report 165663

MINERALOGICAL, OPTICAL, GEOCHEMICAL AND PARTICLE SIZE PROPERTIES OF FOUR SEDIMENT SAMPLES FOR OPTICAL PHYSICS RESEARCH

Karen L. Bice and Stephen C. Clement

COLLEGE OF WILLIAM AND MARY
Department of Geology
Williamsburg, Virginia

NASA Contract NAS1-16042
February 1981

National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665

SUMMARY

X-ray diffraction and spectroscopy were used to investigate the mineralogical and chemical properties of the Calvert, Ball Old Mine, Ball Martin, and Jordan sediments. The particle size distribution and index of refraction of each sample were determined also. The samples are composed primarily of quartz, kaolinite, and illite. The clay minerals are most abundant in the finer particle size fractions. The chemical properties of the four samples are similar. The Calvert sample is most notably different in that it contains a relatively high amount of iron. The dominant particle size fraction in each sample is silt, with lesser amounts of clay and sand. The indices of refraction of the sediments are the same with the exception of the Calvert sample which has a slightly higher value.

INTRODUCTION

A primary goal of marine and inland water optical physics research is quantitative characterization of the spectral properties of natural and pollutant particulate materials. If unique spectral properties are determined, remote-sensing techniques can be developed to research and/or monitor sources, transport, and sedimentation of aquatic suspended particulates.

In this report, four sediment samples were analyzed in the laboratory to determine mineralogy, particle size distribution, index of refraction, and selected chemical properties. The samples studied were mined sediments available in large quantities for repeated laboratory optical studies.

MINERALOGY

Mineralogical analyses were done using an X-ray diffractometer and the clay mineral identification procedures outlined by Carroll (ref. 1) and Griffin (ref. 2). Table 1 summarizes the position of the two major peaks for each of the minerals present in the samples. To determine basic mineralogy, one

powder mount of each sediment was prepared using the smear-on-glass slide technique outlined by Gibbs (ref. 3). The method yields a fairly well-oriented mount necessary for the examination of the $d_{(001)}$ spacing in clay minerals. These smear samples, composed of the entire range of particle sizes present in the samples, were examined on the diffractometer using a goniometer scanning speed of 2 degrees 2θ per minute over the range from 2 degrees 2θ to 64 degrees 2θ.

In order to determine differences in mineralogy among different particle size fractions, three size ranges were separated out of each sample. This was done by sedimentation using the settling velocities calculated with Stoke's Law and given in table 2. Fifty milliliters of approximately 0.1 M sodium oxalate solution were added to each liter of sediment/water suspension to prevent flocculation. Powder mounts were prepared from the following three size fractions: (1) 6 ϕ and finer, (2) 8 ϕ and finer, and (3) 10 ϕ and finer. The slides were then examined on the diffractometer using a scanning speed of 4 degrees 2θ per minute over the range from 8 degrees 2θ to 64 degrees 2θ.

The similarity in mineralogical composition of the four sediments is shown by the composite of diffractograms labeled figure 1. The samples are composed primarily of kaolinite, illite, and quartz, differing only in the relative proportions of these three minerals. The difference in mineralogy among the particle size fractions is also quantitative. With a decrease in particle size there is a decrease in quartz content and an increase in kaolinite content, based on relative peak heights. This is shown in figures 2-5.

SEMIQUANTITATIVE MINERALOGY

Methods for the semiquantitative analysis of sediments are discussed by

Brindley (ref. 5), Carroll (ref. 1), Griffin (reg. 2), and Ruhe and Olsen (ref. 6). Most techniques involve the addition of standard clay minerals to sediment samples and the measurement of diffraction peak areas or heights. However, when using any such method it must be kept in mind that clay standards and naturally occurring clay sediments vary widely in degree of crystallinity and composition. Such differences greatly affect the accuracy of results obtained. For the purposes of this study, a comparison of relative ratio percentages of the peak intensities of the three major minerals is sufficient. If more detailed results than those given here are required, the use of the method outlined by Ruhe and Olsen (ref. 6) is recommended.

The peak intensity above background radiation was calculated by three 10-second diffraction radiation counts at the center of the peak and on either side. The data shown in table 3 represent the relative ratio percentages of the kaolinite $d_{(001)}$, illite $d_{(001)}$, and quartz $d_{(101)}$ peak intensities.

The data in table 3 show that with a decrease in particle size there is a decrease in the relative intensity of the quartz peak. The Ball Old Mine and Ball Martin sediments show a sharper decrease in quartz content than the Calvert and Jordan samples. In each of the four samples, there is an increase in kaolinite content with decreasing particle size. The Ball Old Mine and Ball Martin sediments show a decrease in illite peak intensity while the Calvert and Jordan samples have an increasing illite content.

CHEMICAL PROPERTIES

Analysis was done using the X-ray spectrometer to investigate the relative abundances of elements in the four sediment samples. The relative ratio percentages of the spectroscopic peak intensities for Al, Si, K, Ca, Ti, and Fe were determined for each of the four particle size fractions and

are given in table 4. The intensity of each peak was determined by the number of radiation counts under the K-alpha peak for each element, with the exception of calcium. Because the calcium K-alpha peak coincides with the potassium K-beta peak, the number of counts under the K-beta peak for calcium was used to determine the intensity of that element. The data given here are meant for comparative use only and are not quantitative.

The data show that with a decrease in particle size, there is an increase in the relative intensities of the Ca and Al peaks. The Si and Ti peaks decrease in intensity while the K and Fe peaks fluctuate. The data suggest an overall higher iron content for the Calvert sample. Iron oxide coatings on the clay mineral particles are visible under high magnification.

PARTICLE SIZE DISTRIBUTION

Particle size analyses of the four sediment samples were done by personnel at the Virginia Institute of Marine Science. A Coulter Counter was used to determine the particle size distribution represented by figures 6-9. The limiting particle diameters for sand, silt, and clay are given in table 5 and are based on the Wentworth Scale of particle sizes. Table 6 characterizes the four sediments according to percentages of sand, silt, and clay.

The data show that the samples are composed primarily of silt and clay-sized particles with a minor amount of sand. The Ball Martin and Jordan samples contain slightly coarser particles than the Ball Old Mine and Calvert sediments.

INDEX OF REFRACTION

The indices of refraction of the kaolinite in the four sediments were determined by comparison with index of refraction oils. The results are

shown in table 7. The n_y and n_x values were obtained by direct comparison with standardized index of refraction oils. The n_z value was then obtained by subtracting the birefringence value of 0.007 from the n_y value. The same indices were found for the Ball Old Mine, Ball Martin, and Jordan samples. However, the Calvert sample yielded a slightly higher value.

CONCLUDING REMARKS

The Ball Old Mine, Ball Martin, Calvert, and Jordan sediments are composed primarily of kaolinite, illite, and quartz. The finer particle size fractions contain larger amounts of kaolinite and lesser amounts of quartz. The chemical properties of the four sediments are also similar. The Calvert sample contains a higher amount of iron than the other three sediments. The dominant particle size of the four sediments is silt with a moderate amount of clay and minor sand-sized particles. The Jordan and Ball Martin sediments are slightly coarser than the Calvert and Ball Old Mine samples. The indices of refraction of the Ball Old Mine, Ball Martin, and Jordan samples are the same while the Calvert sediment had a slightly higher index value.

REFERENCES

1. Carroll, D.; Clay Minerals: A Guide to Their X-Ray Identification. Geol. Soc. Am. Special Paper 126, 1970.

2. Griffin, G. M.: Interpretation of X-Ray Diffraction Data. In Procedures in Sedimentary Petrology, R. E. Carver, Ed., 1971, pp. 541-569.

3. Gibbs, R. J.: X-Ray Diffraction Mounts. In Procedures in Sedimentary Petrology, R. E. Carver, ed., 1971, pp. 531-539.

4. Krumbein, W. C., and Pettijohn, F. J.: Manual of Sedimentary Petrology. Appleton-Century-Crofts, Inc., 1938.

5. Brindley, G. W.: Quantitative Analysis of Clay Mixtures. In The X-Ray Identification and Crystal Structures of Clay Minerals, G. Brown, ed., 1961, pp. 489-516.

6. Ruhe, R. V., and Olsen, C. G.: Estimate of Clay-Mineral Content: Additions of Proportions of Soil Clay to Constant Standard. Clays and Clay Minerals, vol. 27, 1979, pp. 322-326.

Table 1.- Criteria for identification of minerals present in the samples

	Kaolinite		Illite		Chlorite		Quartz	
Peak Position °2θ Cu Kα radiation	12.4°	24.9°	8.8°	17.3°	6.2°	12.3°	20.8°	26.6°
d- spacing	7.13Å	3.56Å	10.0Å	5.0Å	14.2Å	7.1Å	4.25Å	3.34Å
Order of reflection (hkl)	(001)	(002)	(001)	(002)	(001)	(002)	(100)	(101)
Relative intensity of peak, JCPDS File	100	66	80	80	80	100	20	100

Table 2.- Particle diameters and settling times used in sedimentation procedures

Particle diameter		Time required to settle 10 cm.*		
microns	ϕ units	Hr.	Min.	Sec.
62	+4	0	0	29
31	+5	0	1	56
16	+6	0	7	44
8	+7	0	31	-
4	+8	2	3	-
2	+9	8	10	-
1	+10	32	42	-
0.5	+11	130	50	-

*Settling times are based on Stoke's Law, assuming an average specific gravity of 2.65 for the sediment and and average temperature of 20°C.

(Adapted from Krumbein and Pettijohn (ref. 4), p. 166)

Table 3.- Relative ratio percentages of mineral peak intensities

Sample	Relative ratio percentage		
	Kao	Ill	Qtz
Ball Old Mine			
A)	77.55	2.96	19.48
B)	91.51	2.20	6.27
C)	96.07	1.36	2.55
Ball Martin			
A)	74.85	5.88	19.26
B)	89.05	3.72	7.21
C)	92.26	2.54	5.20
Calvert			
A)	39.23	11.63	49.13
B)	57.72	11.73	31.53
C)	63.85	14.05	22.08
Jordan			
A)	32.61	10.57	56.80
B)	50.93	13.46	35.60
C)	62.33	13.53	24.13

A)- sample containing the particle size fraction 6 ϕ and finer, B)- particle size fraction 8 ϕ and finer, C)- particle size fraction 10 ϕ and finer.

Table 4.- Relative ratio percentages of element peak intensities

	Al	Si	K	Ca	Ti	Fe
Ball Old Mine						
A)	19.07	58.14	3.90	0.66	8.74	9.46
B)	20.56	55.92	4.14	1.09	7.92	10.34
C)	23.85	52.77	4.28	1.33	6.16	11.57
D)	26.45	51.78	3.83	1.97	5.47	10.48
Ball Martin						
A)	16.89	61.89	4.90	0.70	8.21	7.38
B)	20.43	55.78	5.82	0.99	6.76	10.18
C)	25.59	50.19	5.23	1.47	6.51	10.98
D)	25.40	51.80	4.96	1.72	5.64	10.46
Calvert						
A)	11.97	45.55	5.60	0.53	4.93	31.40
B)	13.30	43.65	6.57	0.66	4.22	31.57
C)	16.51	39.73	7.19	0.82	3.25	32.48
D)	17.15	38.23	7.16	1.02	2.75	33.65
Jordan						
A)	13.17	57.09	6.86	0.52	5.47	16.87
B)	13.61	54.94	7.77	0.68	4.63	18.34
C)	18.45	44.99	9.03	0.78	3.66	23.06
D)	19.33	44.96	9.42	1.24	2.44	22.57

A)- sample containing the entire range of particle sizes, B)- particle size fraction 6ϕ and finer, C)- particle size fraction 8ϕ and finer, D)- particle size fraction 10ϕ and finer.

Table 5.- Limiting particle sizes for sand, silt, and clay.

Particle size fraction	Limiting diameter (mm)
Sand	< 0.0625
Very coarse silt	0.0322 - 0.0625
Coarse silt	0.0156 - 0.0322
Medium silt	0.0078 - 0.0156
Fine silt	0.0039 - 0.0078
Very fine silt	0.0019 - 0.0039
Clay	< 0.0019

Table 6.- Characterization of the four sediments according to percentage of sand, silt, and clay

	Ball Old Mine	Ball Martin	Calvert	Jordan
Very fine sand	0.4	1.4	0.4	4.1
Very coarse silt	1.3	4.4	1.7	8.1
Coarse silt	7.5	14.1	6.1	16.1
Medium silt	11.7	18.4	11.9	12.4
Fine silt	21.4	20.0	23.5	17.0
Very fine silt	23.9	18.2	26.6	19.2
Clay	33.8	23.5	29.8	23.1

Table 7.- Indices of refraction of the four sediment samples

Sample	Index of refraction	
	n_x and n_y	n_z
Ball Old Mine	1.561	1.554
Ball Martin	1.561	1.554
Calvert	1.562	1.555
Jordan	1.561	1.554

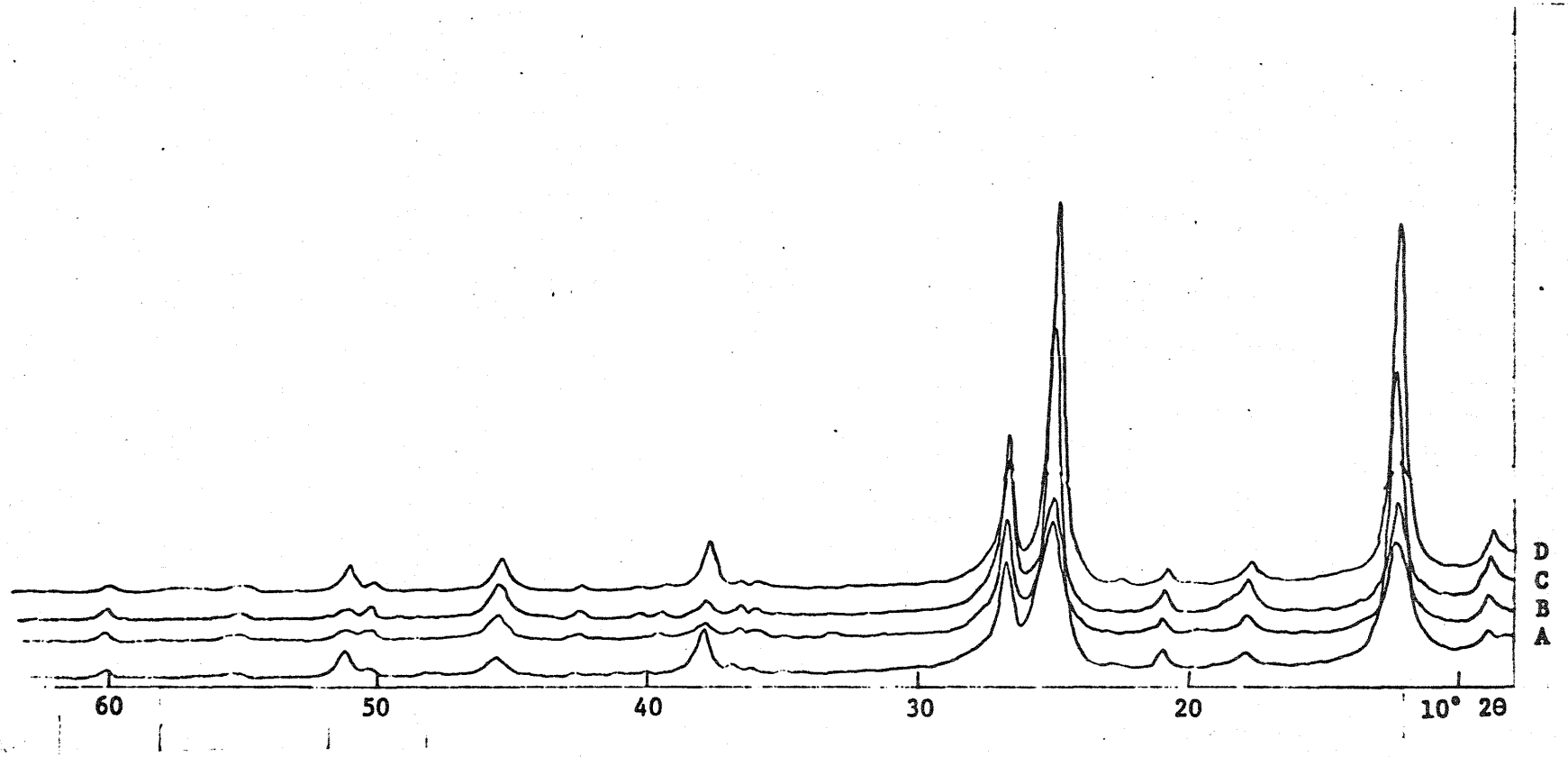

Figure 1 - Composite of diffractograms for Ball Old Mine (A), Calvert (B), Jordan (C) and Ball Martin (D).

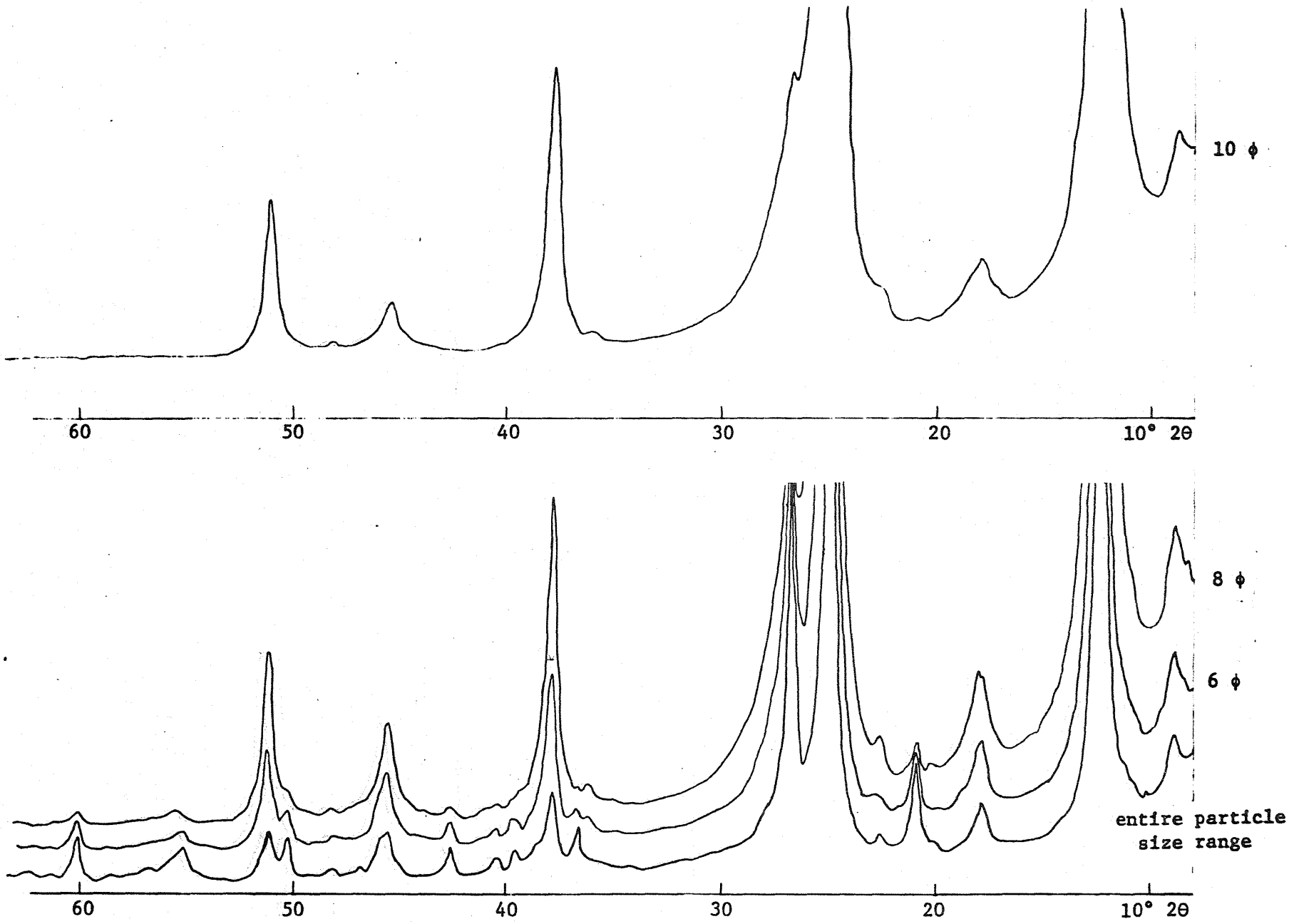

Figure 2 - Diffractograms for Ball Old Mine sediment.

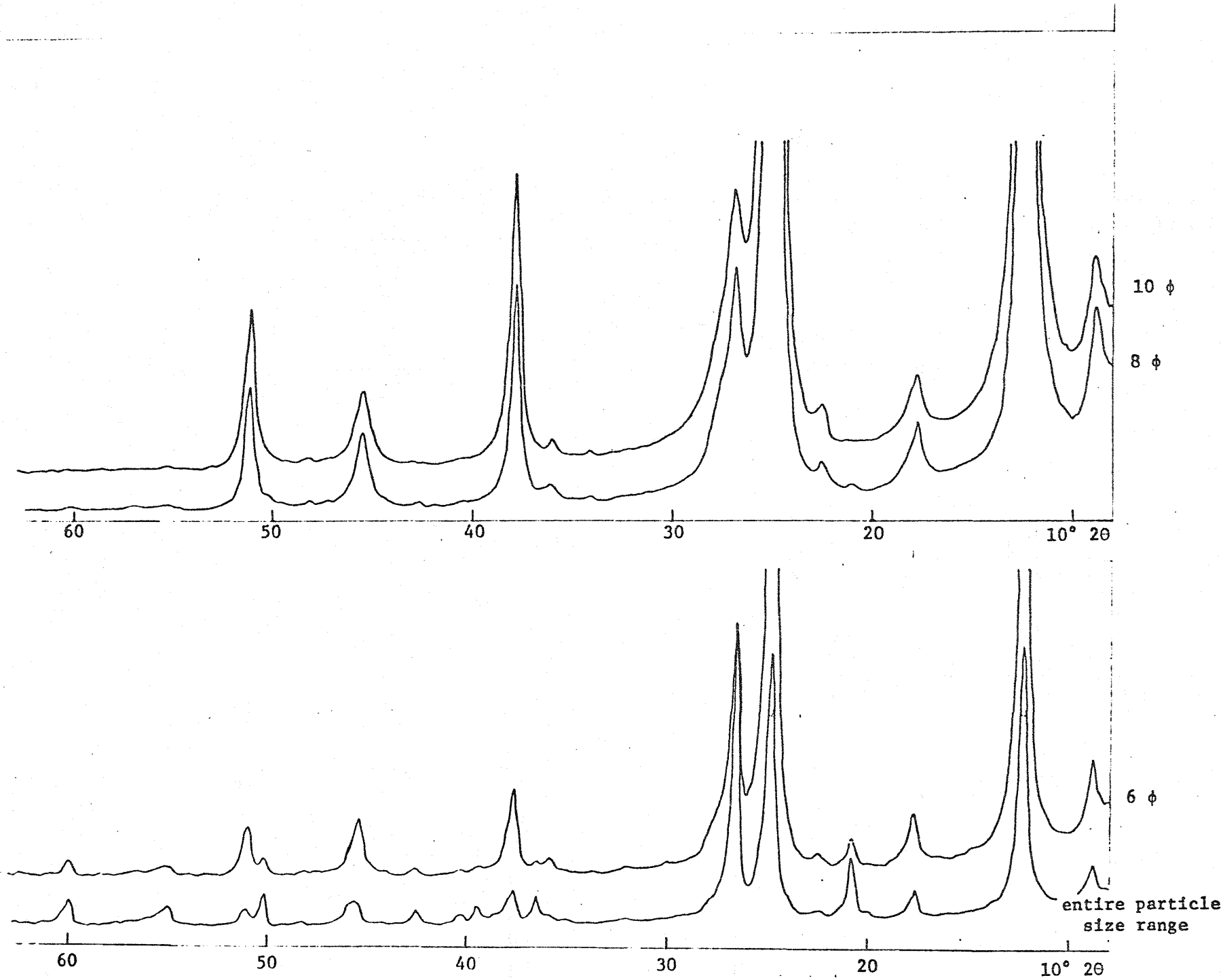

Figure 3 - Diffractograms for Ball Martin sediment.

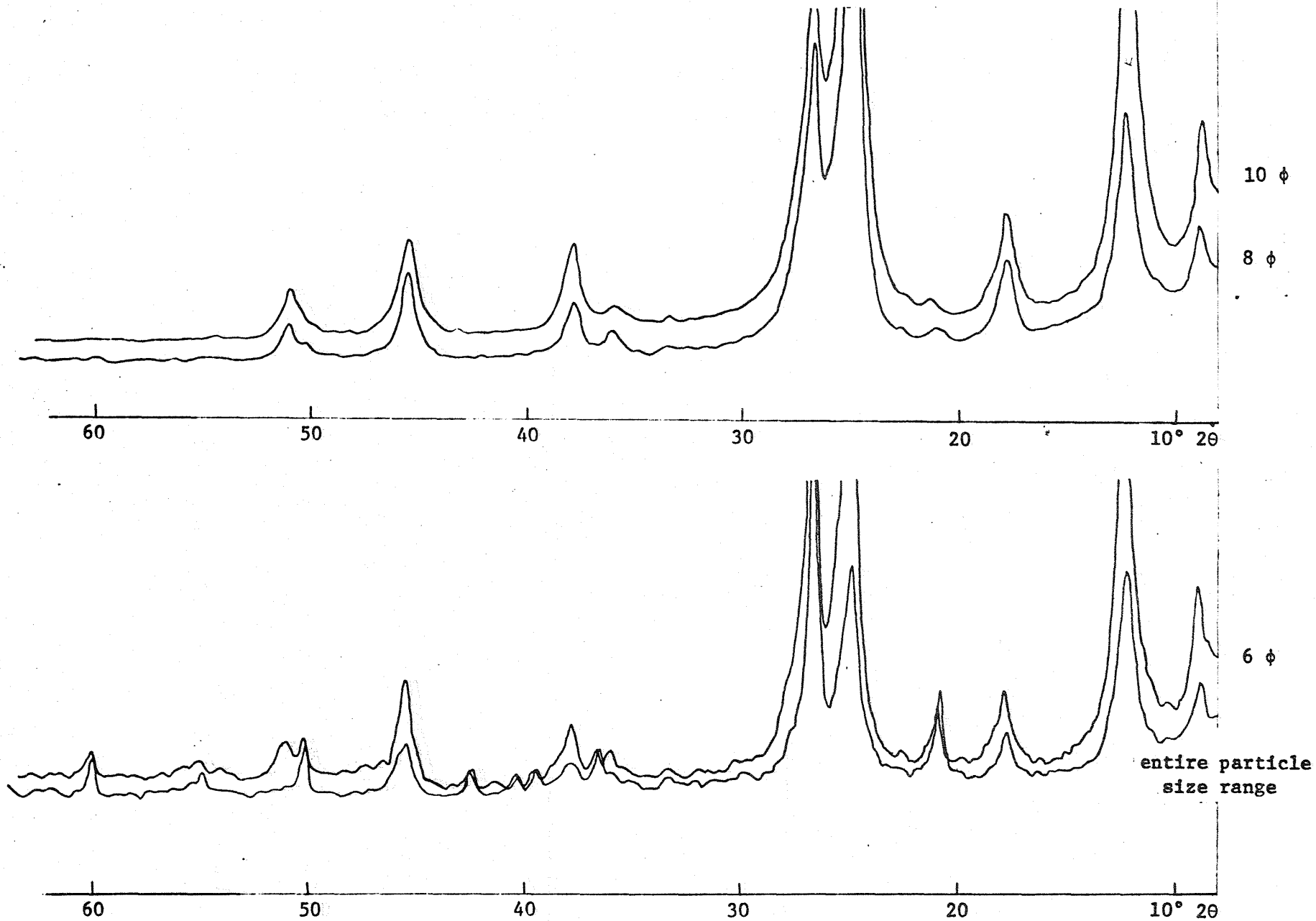

Figure 4 - Diffractograms for Calvert sediment.

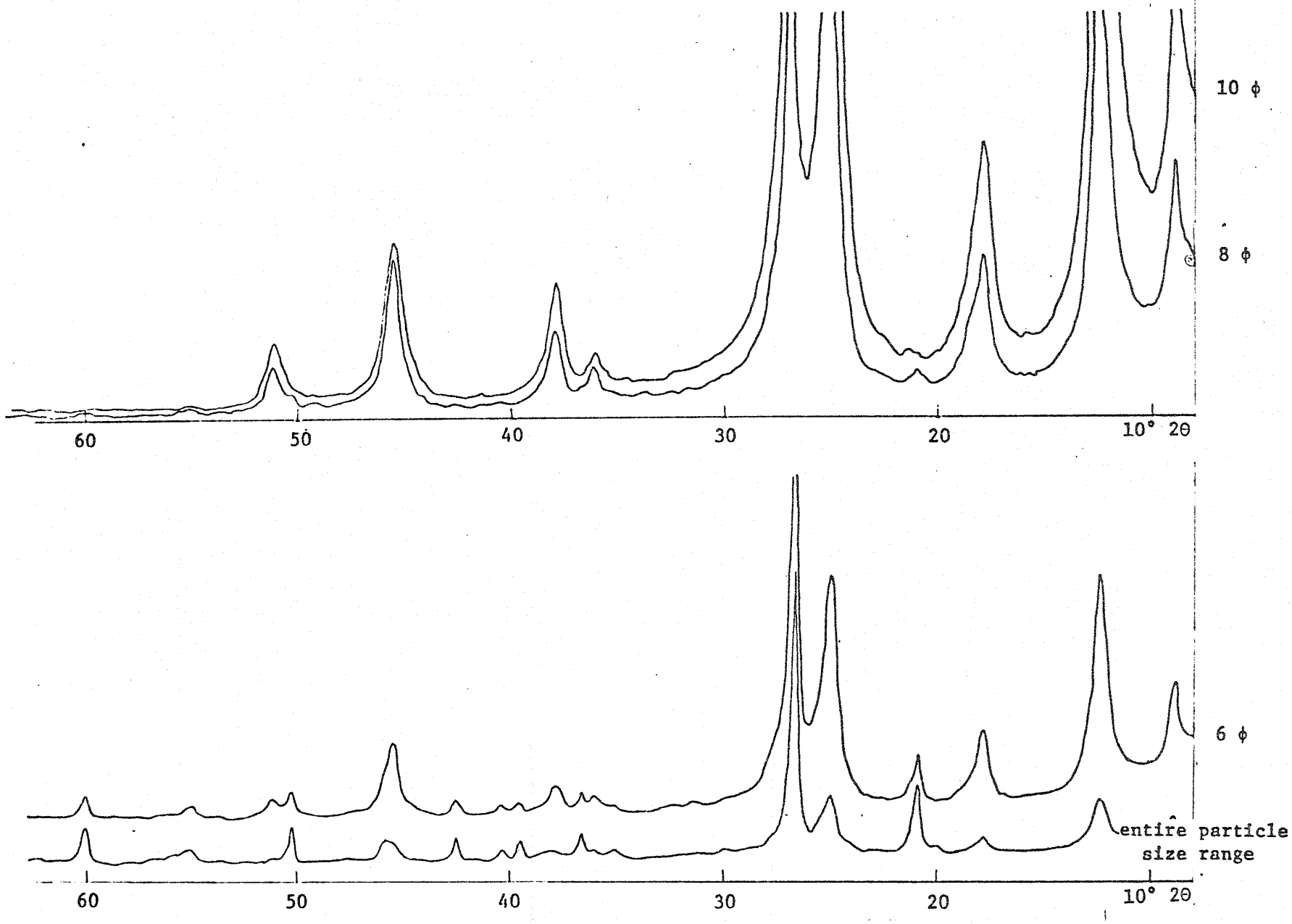

Figure 5 - Diffractograms for Jordan sediment.

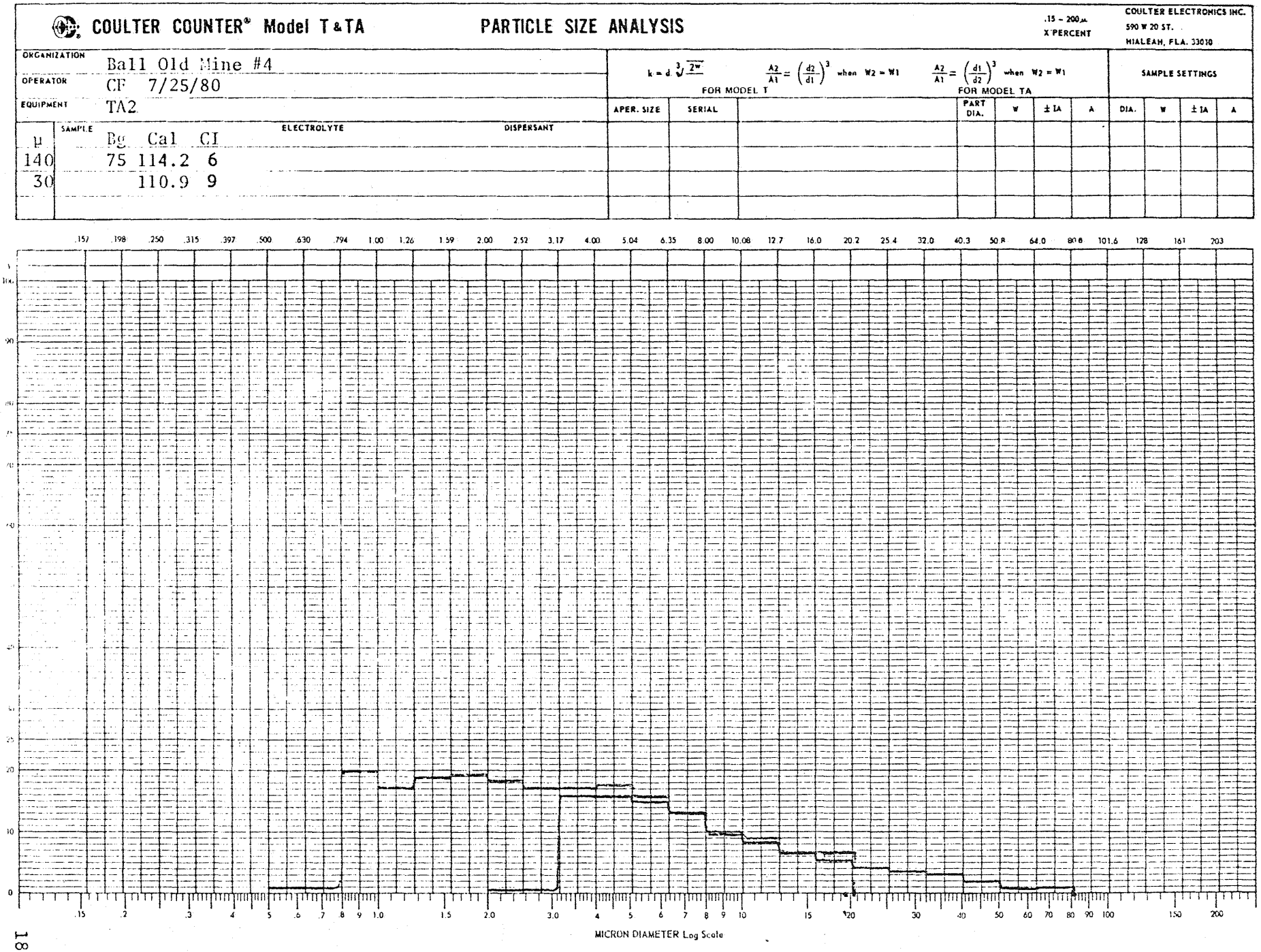

Figure 6 - Particle size analysis for Ball Old Mine sediment.

COULTER COUNTER® Model T&TA

PARTICLE SIZE ANALYSIS

.15 – 200 μ
X'PERCENT

COULTER ELECTRONICS INC.
590 W 20 ST.
HIALEAH, FLA. 33010

ORGANIZATION: Ball Martin #5
OPERATOR: CF 7/28/80
EQUIPMENT: TA2

$k = d\sqrt[3]{2w}$ FOR MODEL T

$\frac{A2}{A1} = \left(\frac{d2}{d1}\right)^3$ when W2 = W1 FOR MODEL TA

$\frac{A2}{A1} = \left(\frac{d1}{d2}\right)^3$ when W2 = W1

μl	SAMPLE	Bg	Cal	CI	ELECTROLYTE	DISPERSANT
140		27	1142	7		
30		18	1109	8		

APER. SIZE	SERIAL		PART DIA.	W	± IA	A

SAMPLE SETTINGS

DIA.	W	± IA	A

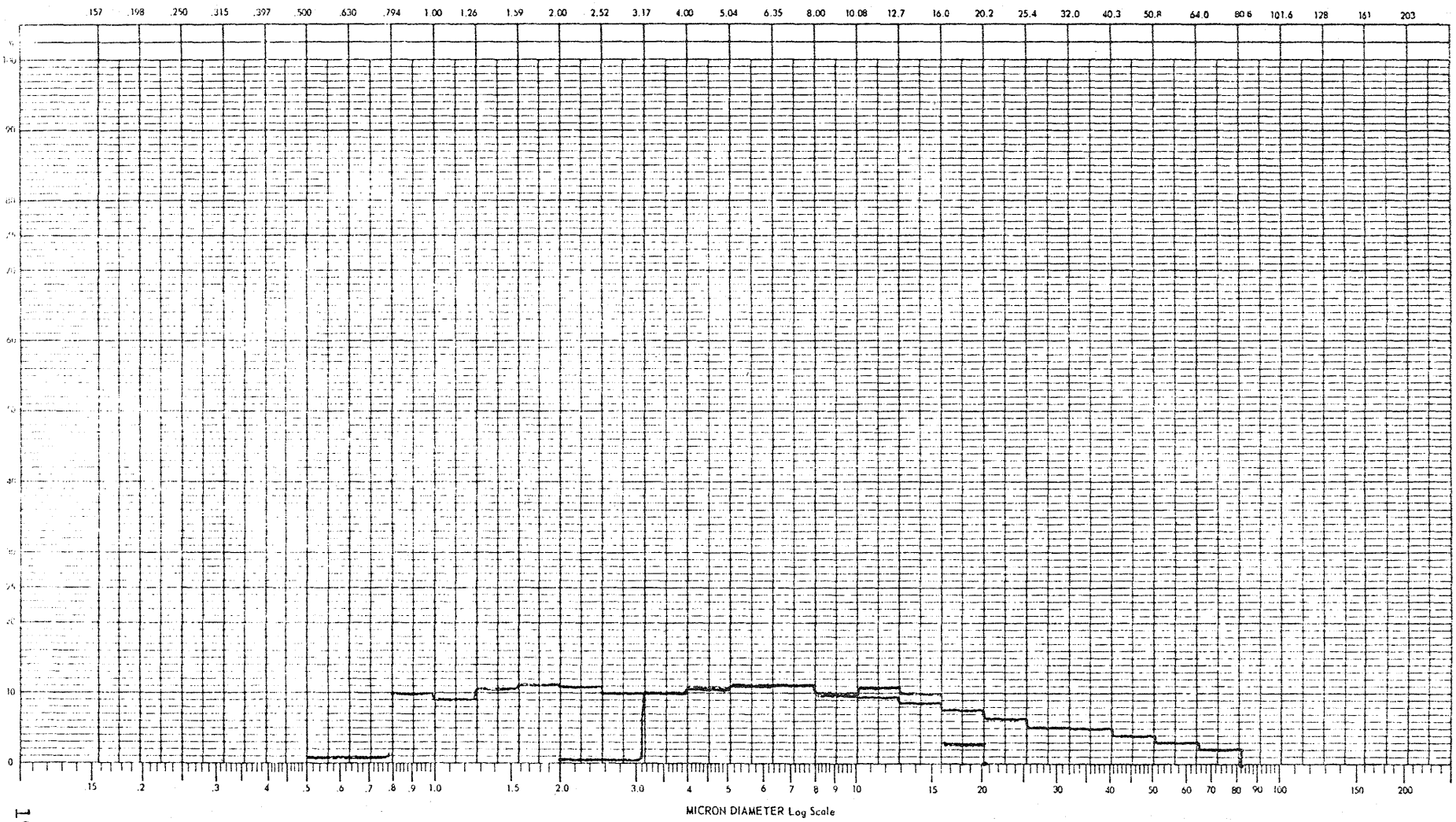

Figure 7 - Particle size analysis for Ball Martin sediment.

COULTER COUNTER® Model T&TA — PARTICLE SIZE ANALYSIS — .15 – 200 μ X PERCENT

COULTER ELECTRONICS INC.
590 W 20 ST.
HIALEAH, FLA. 33010

ORGANIZATION: Calvert
OPERATOR: CF 7/28/80
EQUIPMENT: TA2

$k = d\sqrt[3]{2w}$ FOR MODEL T

$\frac{A2}{A1} = \left(\frac{d2}{d1}\right)^3$ when W2 = W1 $\frac{A2}{A1} = \left(\frac{d1}{d2}\right)^3$ when W2 = W1 FOR MODEL TA

SAMPLE μ	Bg	Cal	CI	ELECTROLYTE	DISPERSANT
140	19	114.2	7		
30	13	110.9	8		

APER. SIZE	SERIAL		PART DIA.	W	± IA	A

SAMPLE SETTINGS DIA.	W	± IA	A

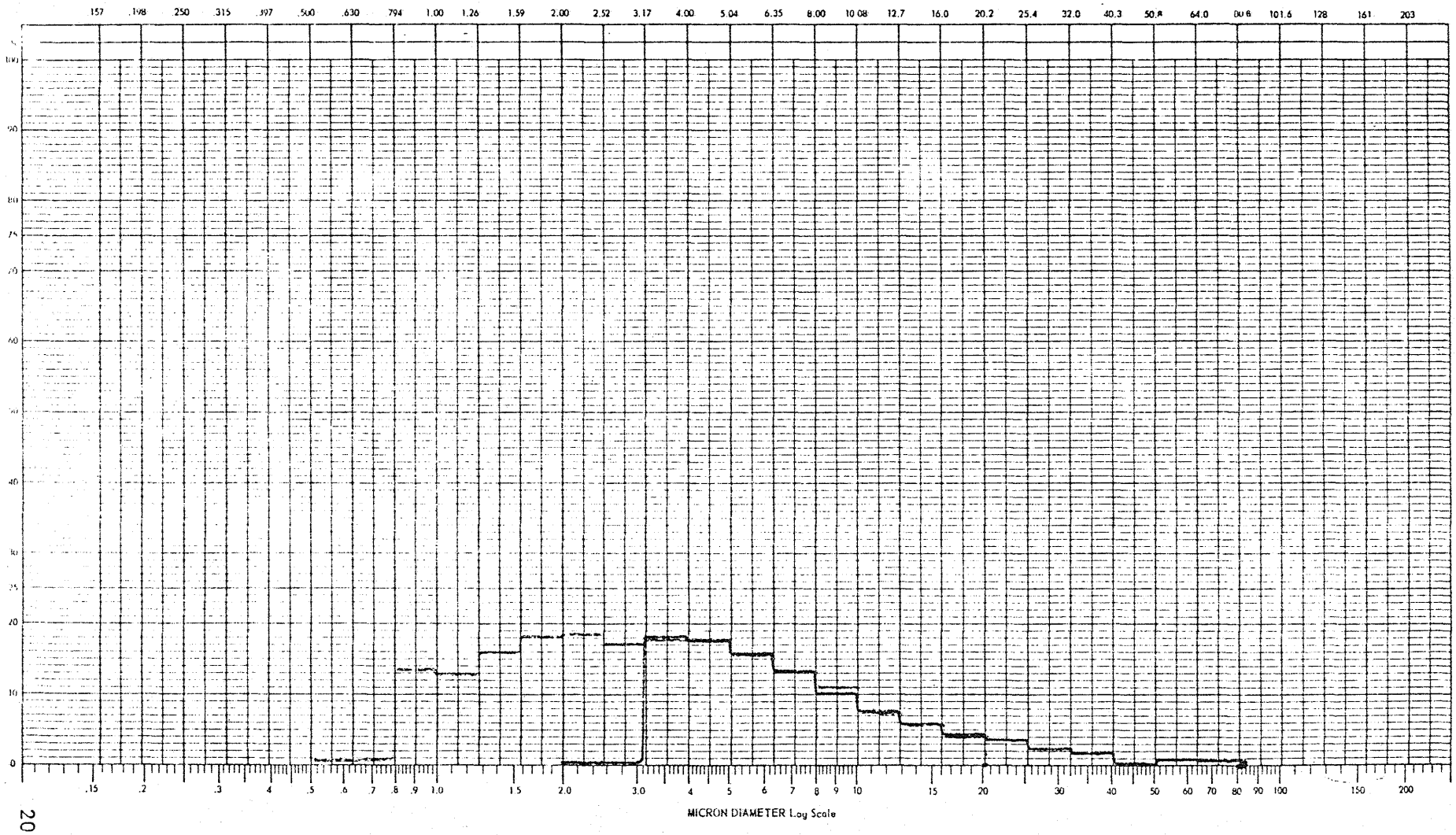

Figure 8 – Particle size analysis for Calvert sediment.

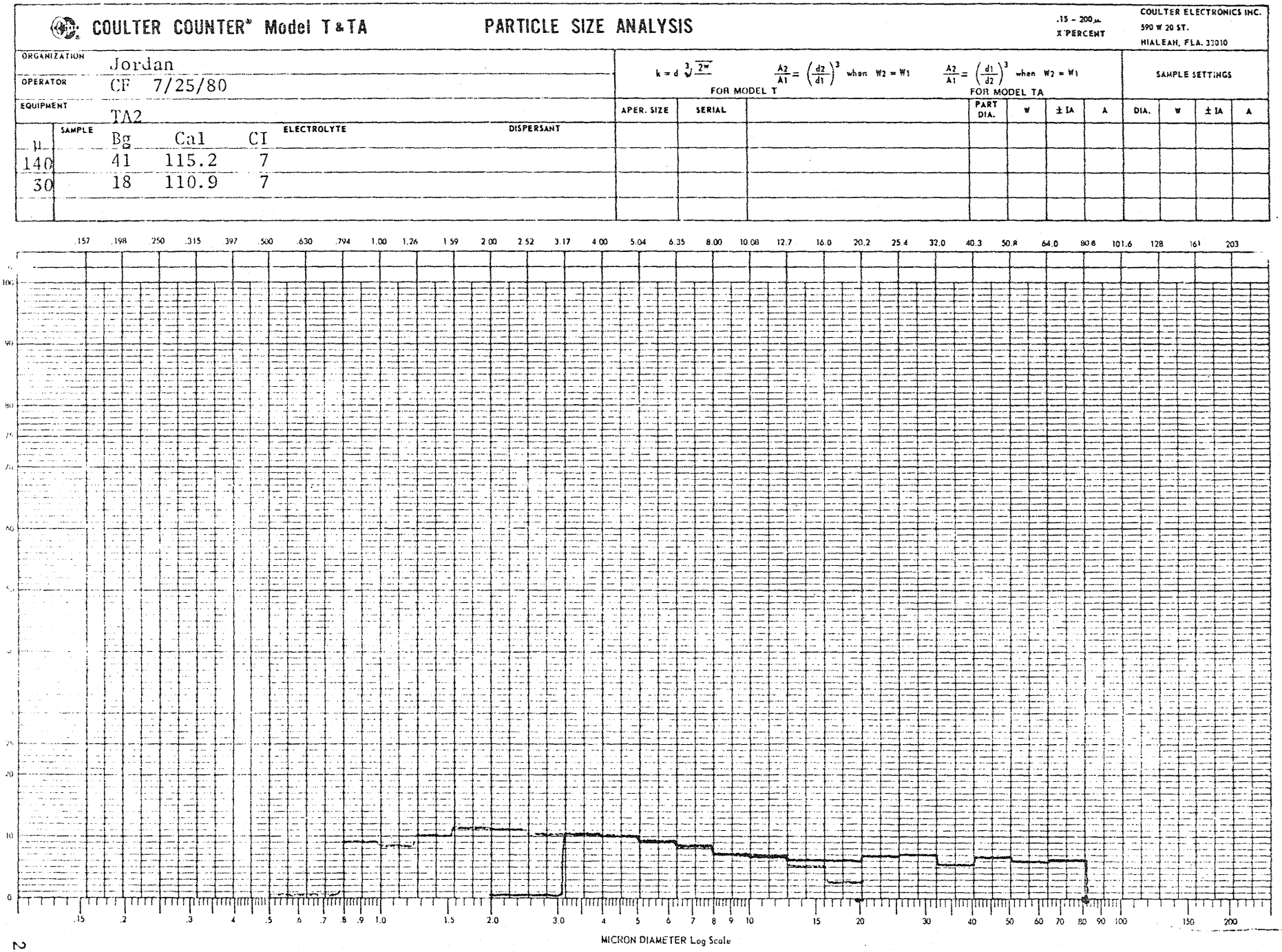

Figure 9 - Particle size analysis for Jordan sediment.

1 Report No	2 Government Accession No	3 Recipient's Catalog No
NASA CR-165663		

4 Title and Subtitle	5 Report Date
Mineralogical, Optical, Geochemical, and Particle Size Properties of Four Sediment Samples for Optical Physics Research	February 1981
	6 Performing Organization Code
7 Author(s)	8 Performing Organization Report No
Karen Bice and Stephen C. Clement	
	10 Work Unit No
9 Performing Organization Name and Address	11 Contract or Grant No
College of William and Mary Department of Geology Williamsburg, Virginia	NAS1-16042
	13 Type of Report and Period Covered
12 Sponsoring Agency Name and Address	Contractor Report
National Aeronautics and Space Administration Washington, DC 20546	14 Sponsoring Agency Code

15 Supplementary Notes

Langley Technical Monitor: Dr. Charles H. Whitlock

16 Abstract

X-ray diffraction and spectroscopy were used to investigate the mineralogical and chemical properties of the Calvert, Ball Old Mine, Ball Martin, and Jordan sediments. The particle size distribution and index of refraction of each sample were determined also. The samples are composed primarily of quartz, kaolinite, and illite. The clay minerals are most abundant in the finer particle size fractions. The chemical properties of the four samples are similar. The Calvert sample is most notably different in that it contains a relatively high amount of iron. The dominant particle size fraction in each sample is silt, with lesser amounts of clay and sand. The indices of refraction of the sediments are the same with the exception of the Calvert sample which has a slightly higher value.

17 Key Words (Suggested by Author(s))	18 Distribution Statement
Remote Sensing Water Quality Optical Coefficients	Unclassified - Unlimited Subject Category 45

19 Security Classif (of this report)	20 Security Classif (of this page)	21 No of Pages	22 Price*
Unclassified	Unclassified	21	A02

For sale by the National Technical Information Service, Springfield Virginia 22161

End of Document