TEXTURAL ANALYSIS OF BENTHIC SEDIMENTS

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by

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Textural analysis involved the handling of approximately 3,700 benthic samples by the Sedimentation Laboratory of the Bureau of Economic Geology. In addition to determination of gravel-sand-mud ratios, particle-size distribution within the sand fraction (-1.0ϕ to 4ϕ) was determined using a Rapid Sediment Analyzer and included sand-sized shell material. Most of the shell material was broken and apparently transported. Size distribution within the mud fraction (4.0ϕ to 10.62ϕ) was determined with a Coulter TA II electronic suspended particle counter. Because the gravel fraction (larger than -1.0ϕ) consisted largely of unbroken shell material (much of which was probably not transported), no size distribution within this fraction was determined.

Most aspects of the basic sample preparation, and particle-size analysis used by this laboratory, have been treated by Krumbein and Pettijohn (1938), Ingram (1971), and Folk (1974), among others. This paper outlines a procedure for sample preparation and determination of particle-size distribution, with special attention given to problems associated with the handling of marine benthic sediments containing an abundance of soluble salts, organic matter, and shell material.

The objectives of this procedure are as follows:

1) the removal of soluble salts which affect clay flocculation;

2) removal of disseminated organic matter which bonds otherwise discrete mud particles and generates misleading mud analysis data;

3) preservation of shell material in its original form so that sand analysis reflects original sediment texture more accurately.

SAMPLE PREPARATION

Carefully transfer into labeled jar entire sample unless it is obviously homogenous; e.g., shoreface sands containing no significant shell or mud. Homogenous samples may be

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dried and disaggregated in order to obtain a smaller representative split. Let sample dry slowly at a temperature below 35ºC.

OXIDATION

When the sample is fairly dewatered, add a small amount (10-20 ml) of hydrogen peroxide (30 percent) and 3 to 4 times more distilled water. Pick clumps of sample apart carefully to avoid damaging included shell material. Samples with no significant shell may be mixed as vigorously as desired. Monitor mix closely for at least one-half hour in case a violent reaction threatens to overflow sample. A clean catch-pan may be placed beneath an over-reacting sample to catch any overflow, allowing its return to the jar later. Vigorous stirring of the rising foam with a glass rod generally prevents overflow. Do not stir the boiling mix in the bottom of the jar if overflow threatens because this increases the reaction rate.

Typically, darker samples (higher mud and organic content) produce a short-lived, violent reaction, while sandier materials react less violently, but will often exhibit a delayed reaction resulting in an overflow. Ideally, strive to produce a mixture of sufficiently low viscosity so that stirring does not alter shell material. However, too wet a mixture often results in a delayed overflow. Let mixture sit overnight at room temperature.

The sample is then re-worked as needed with more hydrogen peroxide and water to reduce any remaining lumps. After each subsequent oxidation let sample sit overnight for maximum oxidation of organic contents. Once lumpiness has been satisfactorily reduced and the most recent oxidation is complete, evaporate any liquid standing on top of the sample by setting it beneath a heat lamp.

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DESALTATION

To remove soluble salts, add distilled water to an 800-ml level in sample jar. Return any material from overflow pan and jar rim to jar. Place watertight lid on jar and shake contents vigorously for 15-30 seconds. Use rubber policeman to free material from glass on inside of jar. Agitate again and let sit for several hours. Agitate once more. Let sit until the water column has cleared, usually 24 hours for sandiest samples to 72 hours for muddiest samples. Siphon off the clear supernatant liquid carefully so that sample remains undisturbed. This desaltation procedure can be repeated for most samples for further removal of salt; however, too successful a removal results in a semipermanently dispersed mud fraction. This is not desirable at this point. Two desaltations are optimum for most samples.

SAMPLE DRYING

After the final desaltation is completed, add approximately 10 ml hydrogen peroxide (30 percent) to a labeled pre-weighed plastic cup. Wash every grain from the jar into the cup. If the cup is filled to the rim, place it in a clean overflow pan. Then place sample under a heat lamp to evaporate excess liquid. As the sample surface is exposed be sure that the temperature around the sample is no warmer than $35^{\circ}C$ to prevent irreversible clumping of clay micelles.

As the topmost material begins to shrink and crack, or curl up at the edges, add a few drops of hydrogen peroxide to further digest organic material. As sample continues to dry add more peroxide to compact lumps that may be present. By the time a sample is completely dry, a scoriaceous texture should be evident. This foam-like texture also aids in subsequent dispersion.

SAMPLE WEIGHT

Once dry, weigh cup with sample, subtract weight of cup, and record the difference as the sample weight.

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DISPERSION

Carefully return weighed sample to its labeled jar and add enough 0.5-percent sodium hexametaphosphate to fill jar to 400-500 ml. Replace lid and shake vigorously for 15-30 seconds. Let sit 24 hours. If a clear column of liquid is standing above sample after 24 hours, siphon it off and add fresh dispersant to the 400-500 ml level. Shake vigorously and let sit. When sample is satisfactorily dispersed it is ready for separation of the gravel, sand, and mud fractions.

WET SIEVING

Assemble a sieving apparatus consisting of a no. 10 mesh wet sieve placed atop a no. 230 mesh wet sieve. Place sieves in the top of a plastic funnel large enough to allow the bottom sieve to sit several inches below the funnel rim. The lab uses eight-inch sieves and a ten-inch-diameter funnel. This assembly is placed in a tripod ring stand of sufficient height to hold the funnel spout above the surface of the working area, e.g., a sink. Place a clean 1,000-ml beaker beneath the funnel spout.

Shake sample vigorously for a few seconds and carefully pour contents into top sieve, allowing flow down through lower sieve and into the beaker. Wash every grain from the jar into the sieve. Clean jar and lid completely with water and set aside.

Carefully work sample with 0.5-percent sodium hexametaphosphate in a wash bottle and use a soft brush to reduce mud lumps and assist in passing the mud fraction through the no. 230 screen and into the beaker. Small lumps can be reduced by gentle finger pressure against the screen. Care must be taken when fragile shell material is present. Finally, wash off the bottom of the no. 230 screen into the funnel. Wash down any material remaining in funnel and on its spout into beaker. Transfer all mud into clean labeled jar and fill to approximately 800 ml with fresh sodium hexametaphosphate. Replace lid and store for further analysis.

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Restack no. 10 sieve and no. 230 sieve and use a small stream of tap water to wash any sand remaining in the coarser sieve into the finer sieve. Work sand still in the no. 230 sieve gently to remove any remaining mud.

GRAVEL/SAND/MUD PROPORTIONS

Transfer gravel and sand to appropriately labeled cups and dry. Pour gravel fraction into a dry no. 10 screen to remove any sand-sized material retained by water tension on the screen. Add this additional sand to the sand fraction obtained by wet sieving. (Borderline sand/gravel shell material is especially prone to retention upon the wet sieve.) Weigh sand and gravel fractions. Record these weights. Mud weight is calculated as follows:

MUD WEIGHT = SAMPLE WEIGHT - (SAND WEIGHT + GRAVEL WEIGHT)

The fractional weights are as follows:

GRAVEL % = GRAVEL WEIGHT/SAMPLE WEIGHT X 100

SAND %   = SAND WEIGHT/SAMPLE WEIGHT X 100

MUD %   = MUD WEIGHT/SAMPLE WEIGHT X 100

Conscientious attention to techniques is important since the sand and mud fractions are analyzed separately on devices that are sensitive to contamination by particles of inappropriate sizes. Mud contamination of the sand fraction increases the viscosity of the water column in the RSA settling tube, thus affecting particle descent rate. Also, a one-gram representative split of the sand fraction is used for settling tube analysis. If a significant proportion is silt, the actual weight of true sand-sized material is reduced, effective viscosity near the water surface is increased, and resolution of the graphical output of the RSA recorder is reduced.

Additionally, any sand introduced, through inattentive technique, into the mud fraction results in an apparently coarser distribution as measured by the Coulter counter.

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The delicate aperture central to the Coulter principle of operation can become hopelessly obstructed with even a few sand grains in the prepared mud fraction.

Rapid Sediment Analyzer Operation

A. Check settling tube alignment

    1. The two weighing pan support wires should be equally spaced from the sides of the tube. If necessary, turn the transducer with its special wrench to center the wires.

    2. Sight down the tube from the top. Wires should be straight and the weighing pan should be centered in the tube. If necessary, turn the support bolts in the steel base of the tube support using the transducer wrench.

    3. Put the sample holder in place. It should be spaced equally between the two pan support wires. If necessary, move the sample holder support arm more to the center of the tube.

B. Sample preparation (described in detail elsewhere)

    1. Wet sieve the sample with a 200-mesh sieve.

    2. Dry the sample and remove large particles, if desired.

    3. Split the sample down to 0.75g to 1.5g.

   4. Weigh the sample split to 0.01g.

C. Sample run

    1. Flip the motor toggle switch "up" to raise the sample holder support arm.

    2. Pull on one of the weighing pan support wires to tilt the weighing pan and dump accumulated sediment from its top. Such dumping is necessary only after each four runs.

    3. Turn on the amplifier.

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4. Set the amplifier sensitivity to match the sample split weight.

5. Turn the amplifier zero adjust for a meter reading slightly above zero.

6. Turn on the chart recorder "source" switch.

7. Set chart drive speed to 360 mm/min.

8. Mark on the chart: sample number, date, sample weight, and water temperature.

9. Dip the sample holder into a standard Calgon or Photoflo solution.

10. Invert the sample holder and sprinkle the sample split evenly over the fabric surface.

11. Place the sample holder on the sample holder support arm and align the wire with the black mark.

12. Flip the chart recorder "chart" switch on and lower the pen.

13. Flip motor switch to "down."

14. As the sound of the motor stops, mark the chart with a starting mark and begin timing.

15. When 30 seconds have elapsed, turn the chart speed to 120 mm/min.

16. When three minutes have elapsed from the start of timing, raise the pen and turn off the "chart" switch.

17. The run is complete. Repeat the appropriate procedures in section C for further runs.

D. Interpretation (See fig. 1.)

1. Extend a line down the length of the chart which is equivalent to the initial weight.

2. Extend a line down the length of the chart which is equivalent to the final weight.

3. Draw a line through the starting time mark and perpendicular to the length of the chart.

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FIG 1
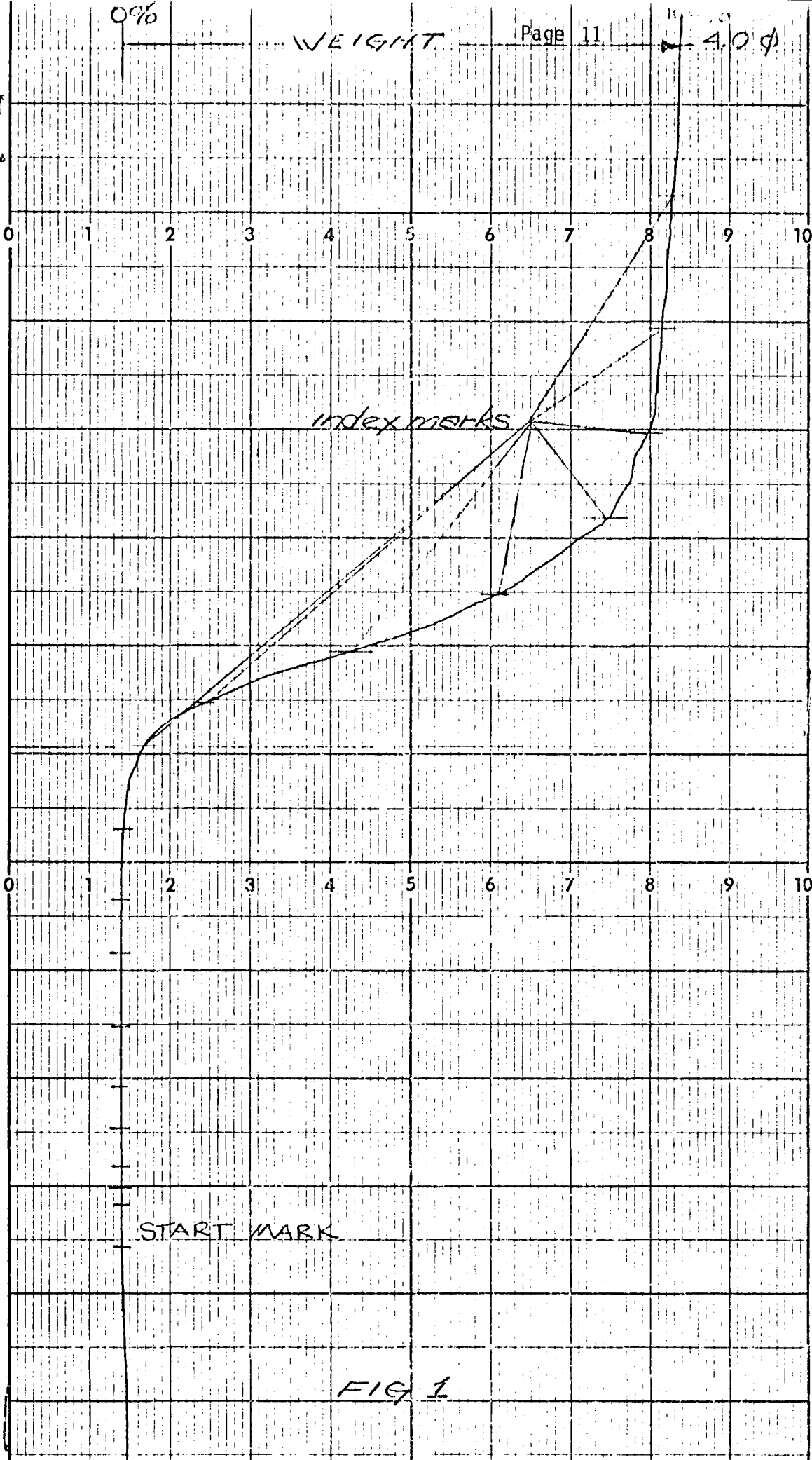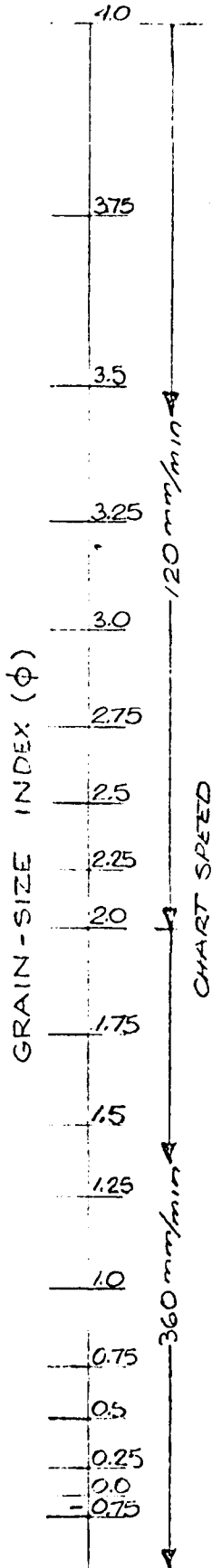
4. Place the calibrated overlay on the chart with the overlay start line on the chart's starting time line.

5. Transfer the φ marks from the overlay onto the chart curve.

6. Using a Gerber variable scale, measure the weight at each φ mark as a percentage of the difference between the initial and final weights on the chart.

ANALYSIS OF MUD FRACTION

Particle-size distribution within the 62.5μ to 0.63μ range is determined with a Coulter TA II electronic fine particle counter. A brief summary of the operational principles of the Coulter counter follows.

An electrical current of constant amperage is propagated through an electrolytic solution between two electrodes. One electrode is positioned in a beaker of clean electrolyte containing a small representative amount of sample. A stirrer device maintains even particle dispersion as electrolyte and sample are drawn by vacuum through a small aperture located in a submerged glass tube within which is located the second electrode. Thus the electrical pathway extends through the aperture. An electrically resistant particle drawn into the aperture creates a resistance proportional to its volume. A proportional voltage pulse results and is assigned to one of 16 separate channels depending on its magnitude.

Four aspects of the analysis are displayed: 1) total particle count; 2) sampling time; 3) a frequency histogram and corresponding numerical values; 4) a cumulative histogram and corresponding numerical values.

TWO-TUBE ANALYSIS

Coulter mud analysis involves the use of two separate aperture tubes and two separate analyses. A 200μ aperture is used to count particles in the 62.5μ to 4μ range

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while a 30μ aperture is used for the 10.08μ to 0.63μ range. The overlap region is used to reconcile the different scales inherent in the separate analyses.

A "scalping" sieve of 20μ pore size is used to remove larger particles between 200μ tube and 30μ tube analysis thus preventing plugging of the aperture by particles larger than its diameter.

ELECTROLYTE FILTERING

The electrolyte used for Coulter analysis must be quite free of particles in its sampling range and, of course, particles large enough to plug an aperture. In addition, some particles of electrolytic solute smaller than the sampling range of a tube (especially the 30μ tube) can act as a radio antenna producing background counts above tolerable limits. Therefore, they must be eliminated.

A dual filtering system has been constructed utilizing a recirculation pump, several feet of plastic hose, a reservoir in-line, and two in-line filters--0.45μ and 0.2μ. A 4-percent sodium hexametaphosphate solution is warmed beneath a heat lamp, poured into the reservoir and recirculated continuously.

This filtered electrolyte is used for cleaning glassware and the micromesh sieve prior to scalping between 200μ and 30μ tube analysis in addition to being used as an electrolyte.

200μ ANALYSIS

The mud fractions of processed samples are dispersed in 0.5-percent sodium hexametaphosphate. A sample is then transferred to a 1,000-ml beaker and placed on a magnetic stirrer set to create a vortex within the sample without exposing the stirring bar. After one minute of agitation, the material is subsampled from three levels within the beaker--10 cm below the surface, 10 cm above the bottom, and at the middle station--and injected into a sampling beaker containing clean 4-percent sodium

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hexametaphosphate. This procedure is repeated with appropriately sized microliter pipets until the particle concentration is 8,000-10,000 counts per 2 ml of electrolyte on the 200μ stand. The sample is then counted until the displayed histogram stabilizes and the trend is obvious. Several more runs are made to confirm a given distribution. A final run is made for 2-3 minutes and the graphic data is recorded by the X-Y plotter and the numerical data is recorded on a work sheet by the operator (fig. 2).

30μ ANALYSIS

A background electrolyte count is made on the 30μ aperture stand. When the background is sufficiently quiet the electrolyte is expended. All parts of the apparatus that are to be in contact with the sample are now clean. The beaker is removed from the 200μ stand and the contents poured through a clean 20μ sieve to remove all particles too large for analysis with the 30μ aperture. Additional clean electrolyte is poured through the sieve to further wash particles into the beaker as well as dilute the sample to approximately 250 ml.

The diluted sample is placed onto the 30μ sample stand. Sample concentration is checked for a count less than 74,000 in 13.5 seconds. If the count is too high, further dilution with electrolyte is required. Once concentration is satisfactory several runs are made to determine characteristic distribution. A final run is made for 2-3 minutes and the data recorded as on the 200μ analysis.

The net result is a pair of overlapping histograms--one covering the range of 3.17μ to 62.5μ and another covering 0.63μ to 12.7μ (fig. 3).

The numerical data is forwarded to a computer which is programmed to combine the two sets of data and produce a single curve. This "mud curve" is then combined with sand analysis data and MUD/SAND/GRAVEL ratio data to produce a single grain-size distribution curve ranging 0.63μ to 62.5μ.
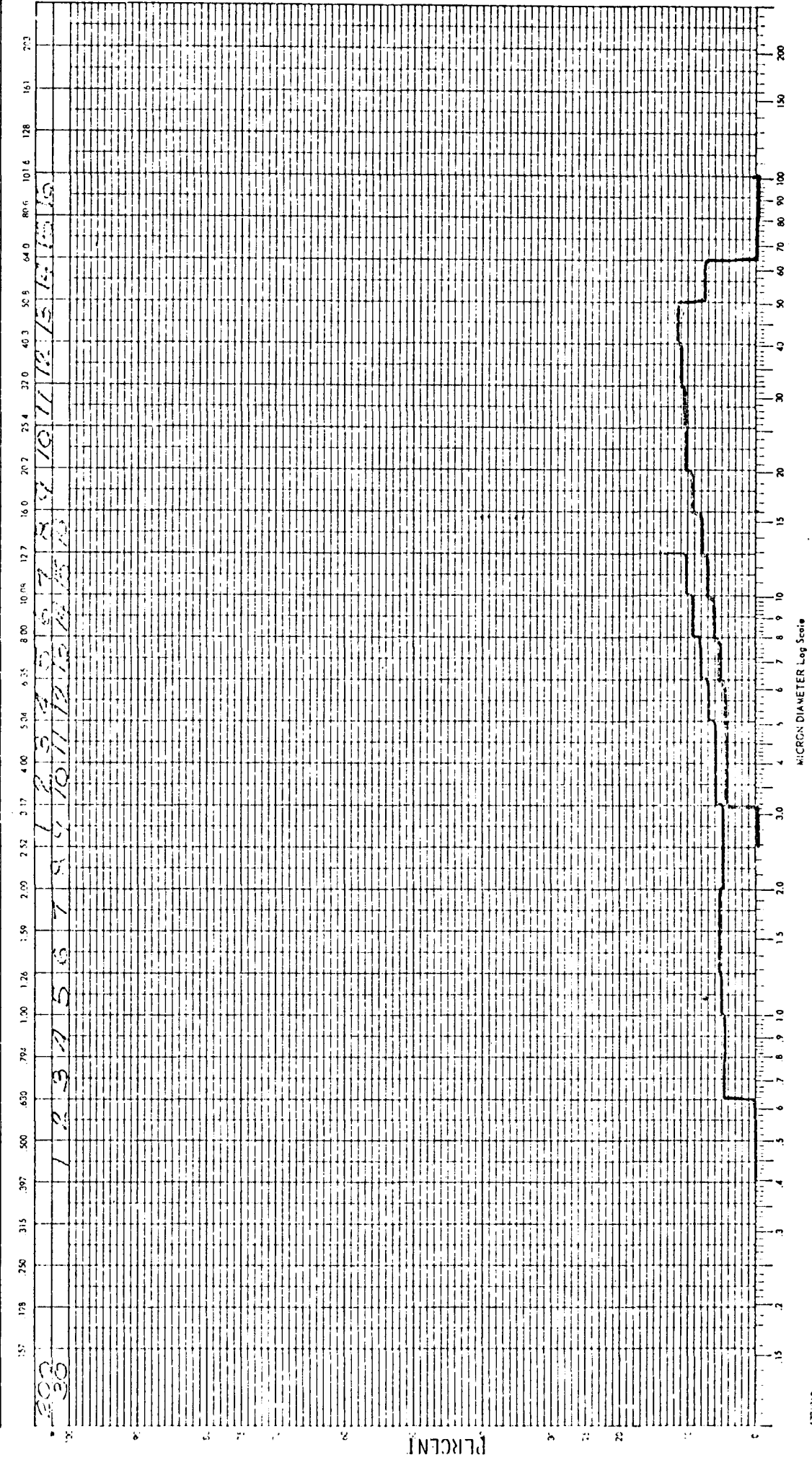
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FIGURE 2

COULTER COUNTER® Model T & T$_A$ Worksheet

SAMPLE 3161	Med. Brownish Gray								

ELECTROLYTE 4% Na-Hexametaphosphate		DISPERSANT 0.5% Na-Hexametaphosphate							

EQUIPMENT TA II	SERIAL	Aper. Dia.	Ser. No.	CALIBRATION DATA	Part. Dia.	W	± I A	A
ORGANIZATION BEG, Austin, Texas		200		Latex	19.75	10		
OPERATOR DAUZAT	DATE 7-2-69	30		Latex	2.02	8		

200μ200, 188 / 210.1 sec

30μ250, 921 / 187.5 sec

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

± I A				
CALIB. A				
APERTURE DIA.				

$$\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3 2^{(W_2 - W_1)} \text{ For Model T}$$

$$\frac{A_2}{A_1} = \left(\frac{d_1}{d_2}\right)^3 2^{(W_2 - W_1)} \text{ For Model T}_A$$

SAMPLE DATA

Geometric Mean μ³	Volume μ³	Diameter μ	200¹	30²	ΔV%¹	Δ V%²	FACTOR	REV 30	ΣV%	NORM	DIF
.00575	.004091	.198									
.0115	.008181	.250									
.0231	.01636	.315									
.0462	.03272	.397	1		0		0				
.0925	.06545	.500	2		0		0				
.1851	.1309	.630	3		4.5		3.1		123.6	100.0	2.5
.3702	.2618	.794	4		4.3		3.0		120.5	97.5	2.4
.7405	.5236	1.00	5		4.8		3.3		117.5	95.1	2.7
1.481	1.047	1.26	6		5.2		3.6		114.2	92.4	2.9
2.962	2.094	1.59	7		5.3		3.7		110.6	89.5	3.0
5.924	4.189	2.00	8		4.6		3.2		106.9	86.5	2.6
11.85	8.378	2.52	1	9	0	4.5	3.1		103.7	84.0	2.5
23.70	16.76	3.17	2	10	4.7	5.6	.839	3.9	100.6	81.4	3.2
47.39	33.51	4.00	3	11	4.5	5.6	.804	3.9	96.7	78.2	3.2
94.78	67.02	5.04	4	12	4.6	6.7	.687	4.7	92.8	75.1	3.8
189.6	134.0	6.35	5	13	5.5	7.7	.714	5.4	88.1	71.3	4.4
379.1	268.1	8.00	6	14	6.2	9.0	.689	6.3	82.7	66.9	5.1
758.3	536.2	10.08	7	15	7.3	9.9	.737	6.9	76.4	61.8	5.6
1516.	1072.	12.7	8	16	8.1	0	–	0	69.5	56.2	6.6
3033.	2145.	16.0	9		9.5				61.4	49.7	7.7
6066.	4289.	20.2	10		10.5		.697		51.9	42.0	8.5
12.13 x 10³	8579.	25.4	11		10.6				41.4	33.5	8.6
24.27 x 10³	17.16 x 10³	32.0	12		11.1				30.8	24.9	9.0
48.54 x 10³	34.31 x 10³	40.3	13		11.8				19.7	15.9	9.5
97.18 x 10³	68.63 x 10³	50.8	14		7.9				7.9	6.4	6.4
194.4 x 10³	137.3 x 10³	64.0	15		0						
388.7 x 10³	274.5 x 10³	80.6	16		0				*NORMALIZED*		
777.4 x 10³	549.0 x 10³	101.6							*CUMULATIVE*		
1.555 x 10⁶	1.098 x 10⁶	128.									
3.109 x 10⁶	2.196 x 10⁶	161.							*NORMALIZED*		
6.219 x 10⁶	4.392 x 10⁶	203.							*HISTOGRAM*		
12.44 x 10⁶	8.784 x 10⁶	256.									
24.88 x 10⁶	17.57 x 10⁶	322.									
49.75 x 10⁶	35.14 x 10⁶	406.									
99.50 x 10⁶	70.27 x 10⁶	512.									
199.0 x 10⁶	140.6 x 10⁶	645.									
398.0 x 10⁶	281.1 x 10⁶	812.									
796.0 x 10⁶	562.2 x 10⁶	1024.									

4221008

FIGURE 3--GRAPHIC DATA

COULTER COUNTER® Model T & TA   PARTICLE SIZE ANALYSIS

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

ORGANIZATION: *Bureau of Economic Geology*
OPERATOR: *Dalziel*
EQUIPMENT: TA II
SAMPLE: 3161
ELECTROLYTE: 4% Calgon
DISPERSANT:

MICRON DIAMETER Log Scale

PERCENT

APPENDIX A

REFERENCES

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