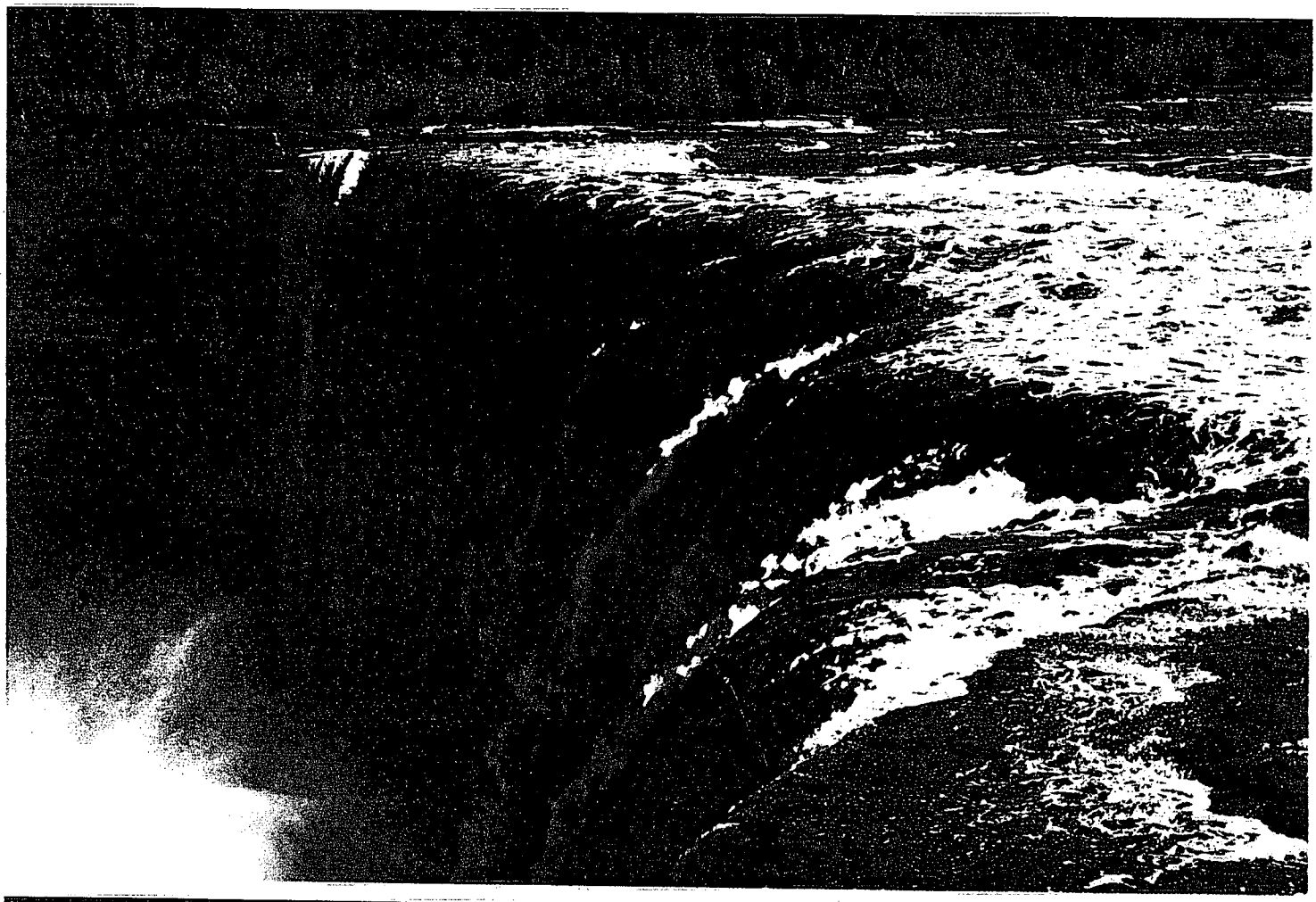
Evaluation of Lyophilization Method of Disaggregating Sand, Silt and Clay

E.F. Bolan and M.A. Cashman



TECHNICAL BULLETIN NO. 106

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INLAND WATERS DIRECTORATE
WATER RESOURCES BRANCH
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ABSTRACT

The Sediment Survey program includes the collection of "bed material" samples that are analyzed for particle size distribution. The present method of oven-drying requires vigorous grinding with a mortar and pestle to disaggregate the particles. The adaptation of the freeze dryer reduces this grinding time considerably.

This report evaluates the freeze dryer or lyophilization method of disaggregating sands, silts and clays for particle size analysis and compares it with the oven-dried method presently used in the Sediment Laboratories of the Water Survey of Canada.

RÉSUMÉ

Le programme d'études des sédiments consiste à prélever des échantillons de "matériaux de fond" pour en faire l'analyse granulométrique. La méthode actuelle de séchage au four exige que le matériau soit d'abord vigoureusement broyé dans un mortier afin de le désagréger. La nouvelle méthode de séchage à très basse température réduit considérablement le temps passé à le broyer.

Dans le présent rapport, on évalue la méthode de séchage à très basse température ou lyophilisation, employée pour désagréger les sables, les limons et les argiles destinés à l'analyse granulométrique, par rapport à la méthode de séchage au four actuellement utilisée dans les laboratoires d'études des sediments de la Division des relevés hydrologiques du Canada.

INTRODUCTION

In the present conventional oven-drying method used in the Sediment Laboratories, the classification of soil separates (sand, silt, clay) is based on sieve diameters of the soil particles. If more than 10% of a grain size sample passes the 63-μm sieve, a hydrometer analysis must be done (see "Bed Material Hydrometer Analysis"). The bed material samples are oven-dried (usually overnight) from aqueous suspensions and ground to a workable powder in preparation for analysis. Samples with high clay fractions form hard flakes and require vigorous grinding. Preparation time varies from 15 to 30 min per sample.

A theoretical treatment of lyophilization and the application to large-scale drying of clays has been given by Strickland-Constable and Bruce (1954). With this method a sample is quick-frozen and the water removed by sublimation under vacuum to a cold condenser surface. The heat transfer equivalent to the heat of sublimation of water is sufficient to keep the sample frozen until it is dry.

The lyophilization equipment manufactured by Labconco Corporation (1978) was used not only because of its simplicity, capacity and rugged nature but mainly because of its availability (Fig. 1). The 12-port drying chamber, fabricated in stainless steel, has eight ½-in. valves and four ¾-in. valves with a clear acrylic top plate ¾-in. thick. The cabinet has an electrical outlet and adequate space for enclosing the Sargent-Welch high-vacuum pump, which has a free-air displacement rate of 100 litres per minute. The high-capacity 1/3 HP freon refrigeration system efficiently cools the condenser to -50°C (-60°F). The stainless steel condensing coils are within the drying chamber. Three litres of ice may be condensed before defrosting and cleaning are necessary. The effluent

drain line at the base of the drying chamber is easily accessible from the front of the cabinet. The condenser temperature is continuously indicated on a thermocouple gauge on the front panel. The electronic vacuum gauge, which operates on the thermocouple principle, gives a continuous indication of the relative vacuum conditions. The switches for the condenser refrigeration and the vacuum pump are illuminated.

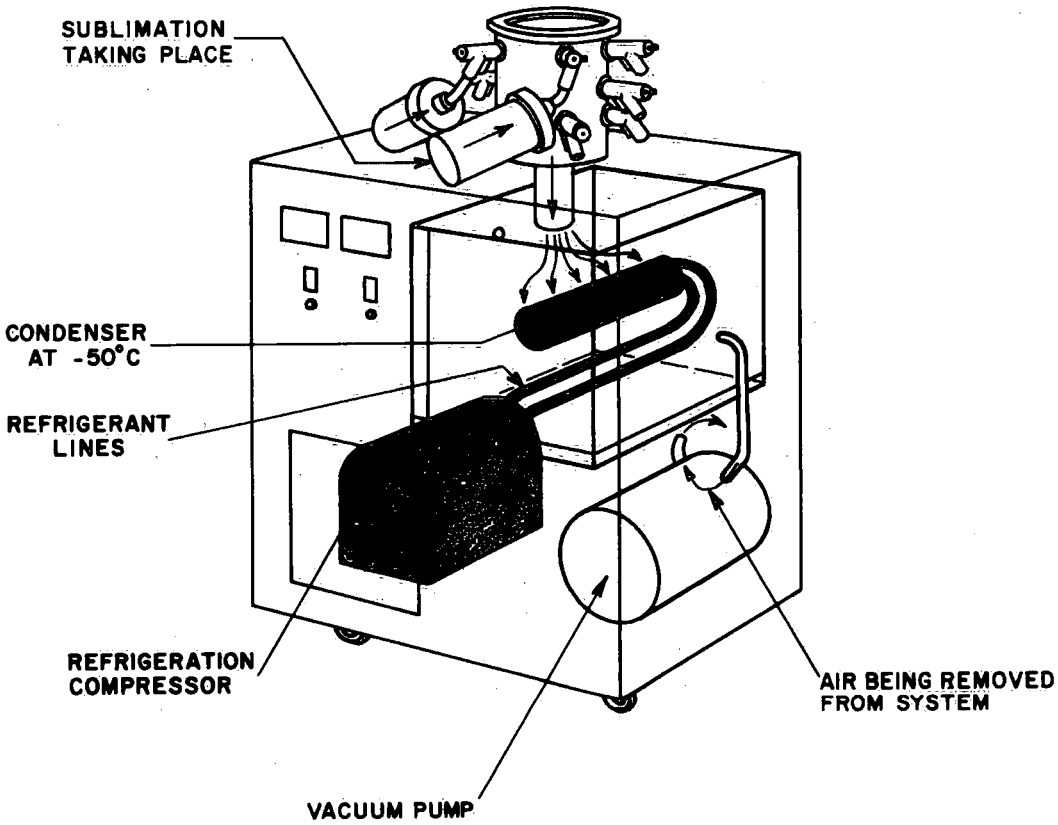

FIGURE I - LABCONCO FREEZE DRYER-3

The basic techniques used in freeze-drying are quite simple. The aqueous solution or suspension of bed material to be dried is transferred to a 600-mL freeze-dry flask. After connecting the flask through the drying chamber to the high-vacuum pump, the system is evacuated and left until the sample is dry. Although the time of drying varies from 2 to 24 hr, the usual practice is to allow the apparatus to run overnight.

The sample is dry when all of the frost has disappeared from the outer surface of the sample container and no cold spots can be detected by handling the container.

For samples with high silt and clay fractions, a volume of about 150 mL is the maximum which can be used in each flask. A larger volume prevents the development of a uniform frozen shell. The shell is important because, if the layer of dry clay is thick towards the end of the drying period, the tortuous path through the clay causes an increase in vapour pressure and a decrease in the rate of drying. The heat of transfer is decreased by the insulating property of the thicker layer of dry clay and the ice melts before drying is complete. For coarser materials and samples with low moisture contents, larger volumes can be used.

TESTS

Test (a)

Three dry bed material samples were chosen with high sand fractions. Each sample was poured through a Jones sample splitter. The splitter consists of a series of inclined chutes leading alternately to two pans placed on opposite sides of the apparatus (Fig. 2). The sample was poured into the hopper using a rectangular pan, the width of which was equal to the width of the set of chutes. The sample was split to the desired volume and random particle size distribution by re-splitting the contents of the right and left receiving pans alternately. Thus, two representative samples of the original were obtained.

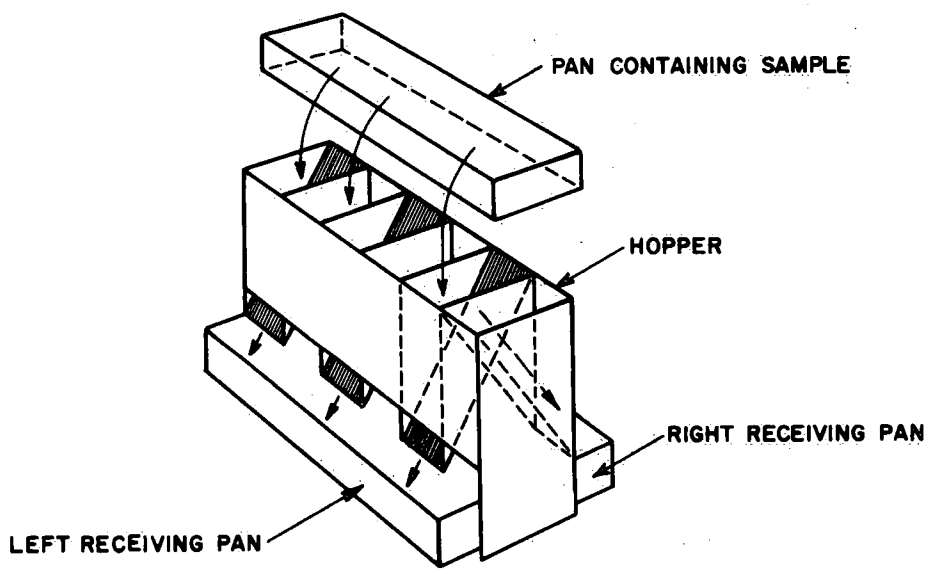

FIGURE 2 - PRINCIPLE OF A CHAMBER SAMPLE SPLITTER

A volume of 50 mL of distilled water was added to each portion. One portion of each of the three samples was then freeze-dried, while the other was treated by the conventional oven-drying method. The resulting dried portions were analyzed using the standard sieve series:

8.000 mm, 4.000 mm, 2.000 mm, 1.000 mm, 0.500 mm, 0.250 mm, 0.177 mm, 0.125 mm and 0.0625 mm.

Test (b)

Three bed material samples with high clay-silt fractions were chosen. As in Test (a), the samples were split and distilled water was added to each portion. One portion of each sample was freeze-dried and the other oven-dried. The dry samples were analyzed by the hydrometer analysis, since the criteria for such analyses were met (i.e., more than 10% of the sample passed the 0.0625 mm sieve).

Test (c)

For this test, two samples of 50 g were prepared with predetermined grain sizes as follows:

Approximate Percentage Finer Than Indicated Size	Grain Size (mm)
100.0	0.500
90.0	0.250
80.0	0.177
60.0	0.125
30.0	0.0625

A volume of 20.0 mL of distilled water was added to each sample. One sample was freeze-dried and the other oven-dried. The dried samples were analyzed by the hydrometer method.

OBSERVATIONS AND RESULTS

Test (a)

For the test samples with high sand fractions, the method of drying - freeze-drying or oven-drying - had little effect on the results. The freeze-dried samples required only a slight grinding with the mortar and pestle (Fig. 3) in preparation for analysis. The differences in the "percentage finer than indicated size" values (Table 1) and the graphical comparisons (Figs. 4 to 6) indicate the slight variation in the results. This variation could be attributed to the method of splitting the samples (variance in sample uniformity) and human error in the plotting and drawing of the particle size curves.

Figure 3. Mortar, pestle and freeze-dried sample.

Table 1. Results of Test (a).

METHOD OF SAMPLE PREPARATION	SAMPLE NUMBER	TOTAL SAMPLE DRY WEIGHT (g)	PERCENTAGE FINER THAN INDICATED SIZE										% SILT 0.062–0.004 mm.	% CLAY <0.004 mm.
			U.S. STANDARD SIEVE NUMBER											
			5/8	5/16	5	10	18	35	60	80	120	230		
			OPENING IN MILLIMETRES											
			16.000	8.000	4.000	2.000	1.000	0.500	0.250	0.177	0.125	0.062		
FREEZE-DRIED	1 A	201.7	100.0	99.6	94.3	84.2	68.6	40.5	9.9	2.7	0.5	0.1	0.1	0.0
OVEN-DRIED	1 B	207.8	100.0	97.6	94.1	85.7	71.4	42.8	10.8	2.6	0.4	0.1	0.1	0.0
DIFFERENCE IN PERCENT FINER VALUES	1 A-1B		0.0	2.0	0.2	-1.5	-2.8	-2.3	-0.9	0.1	0.1	0.0	0.0	0.0
FREEZE-DRIED	2 A	227.2	100.0	98.8	95.7	90.1	79.0	41.0	7.1	2.2	0.4	0.1	0.1	0.0
OVEN-DRIED	2 B	221.7	100.0	97.7	94.8	89.4	78.6	39.7	6.6	2.0	0.4	0.1	0.1	0.0
DIFFERENCE IN PERCENT FINER VALUES	2 A-2B		0.0	1.1	0.9	0.7	0.4	1.3	0.5	0.2	0.0	0.0	0.0	0.0
FREEZE-DRIED	3 A	228.2		100.0	99.8	99.3	97.7	70.3	31.2	15.0	6.0	1.4	1.4	0.0
OVEN-DRIED	3 B	248.7		100.0	99.5	98.9	97.2	66.1	31.0	15.6	6.2	1.4	1.4	0.0
DIFFERENCE IN PERCENT FINER VALUES	3 A-3 B			0.0	0.3	0.4	0.5	4.2	0.2	-0.6	-0.2	0.0	0.0	0.0

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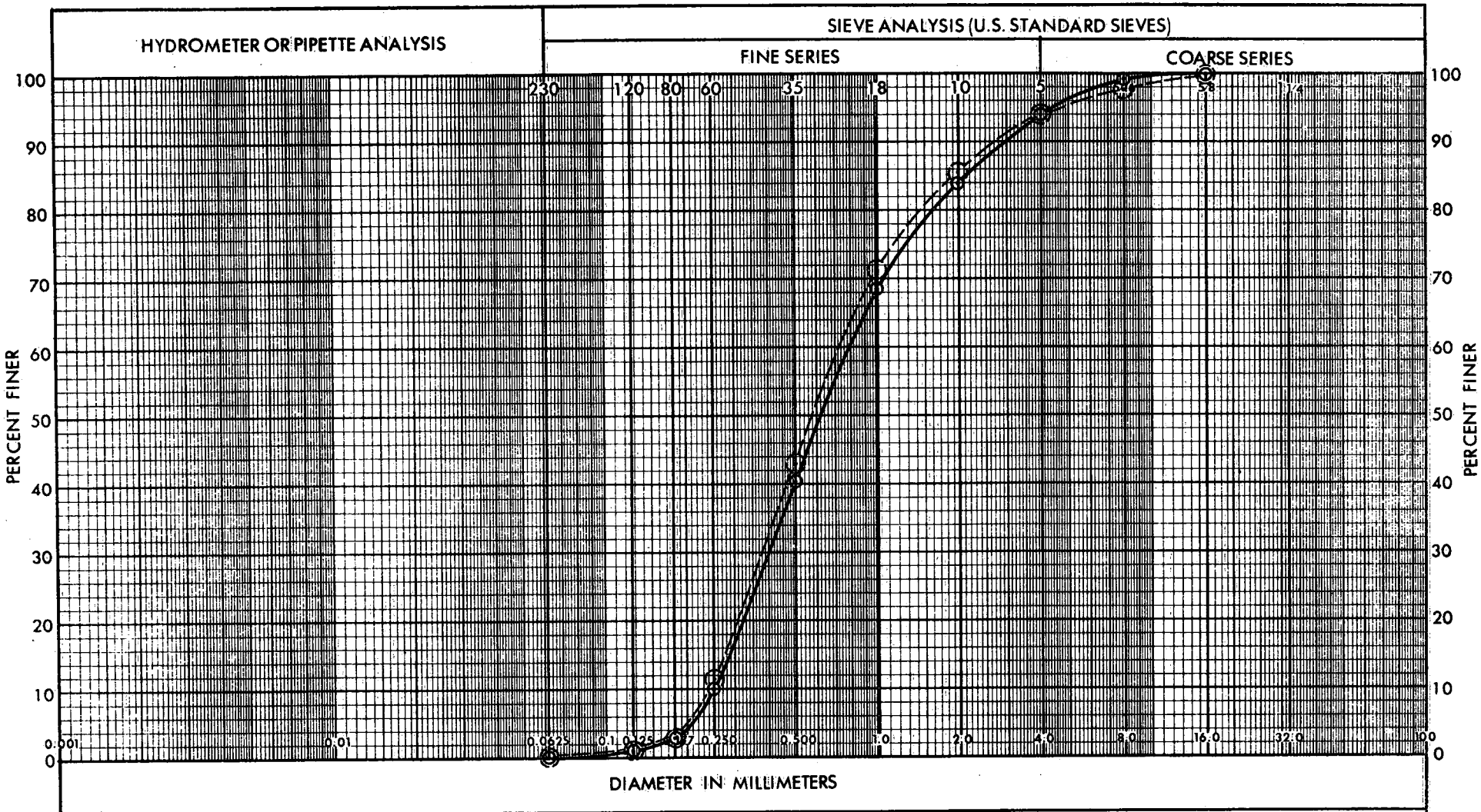

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE - DRIED 1A —⊙—

OVEN - DRIED 1B —⊙—

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BED LOAD AND BED MATERIAL

FIGURE 4

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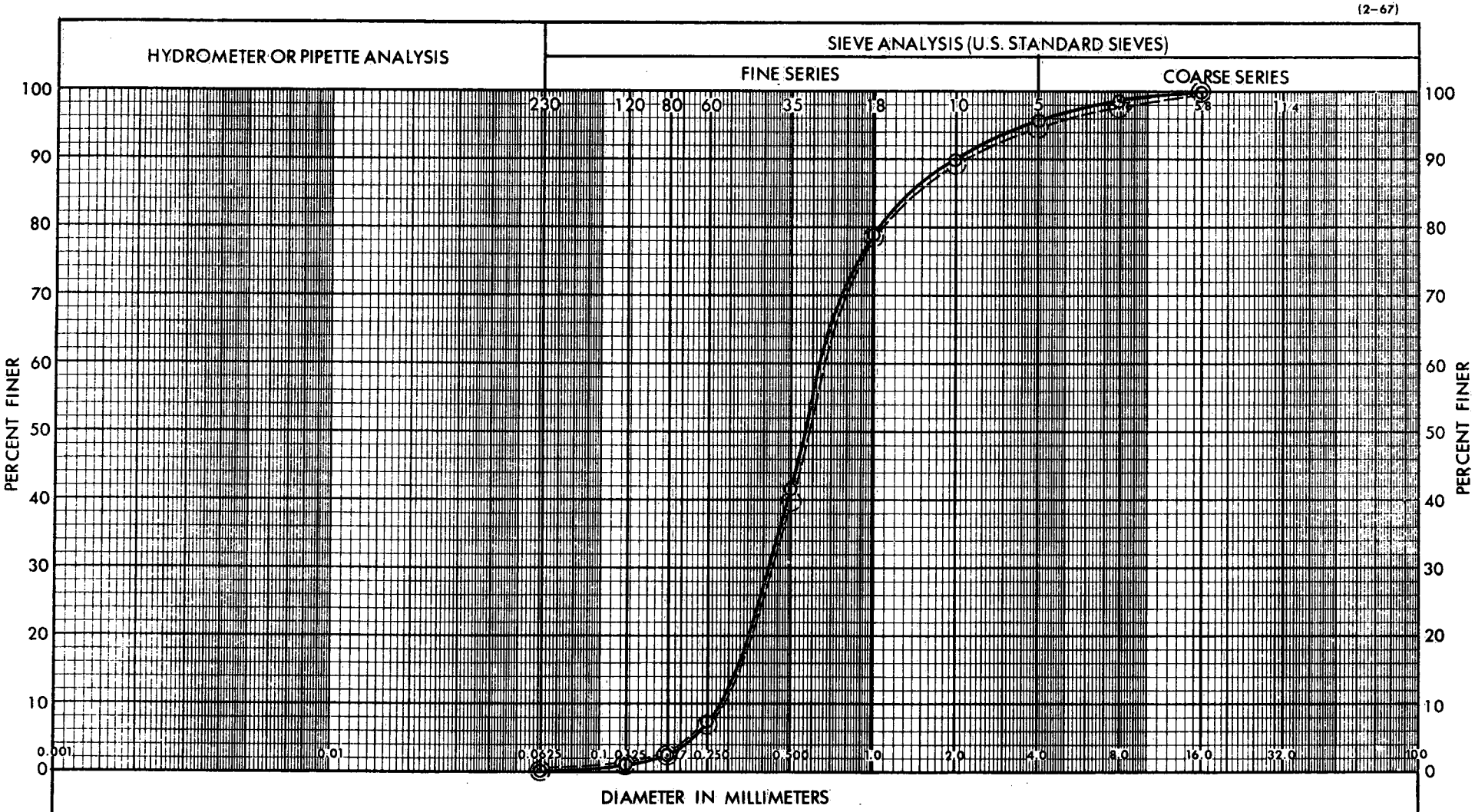

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE – DRIED 2A —⊙—

OVEN – DRIED 2B – –⊙– –

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BED LOAD AND BED MATERIAL

FIGURE 5

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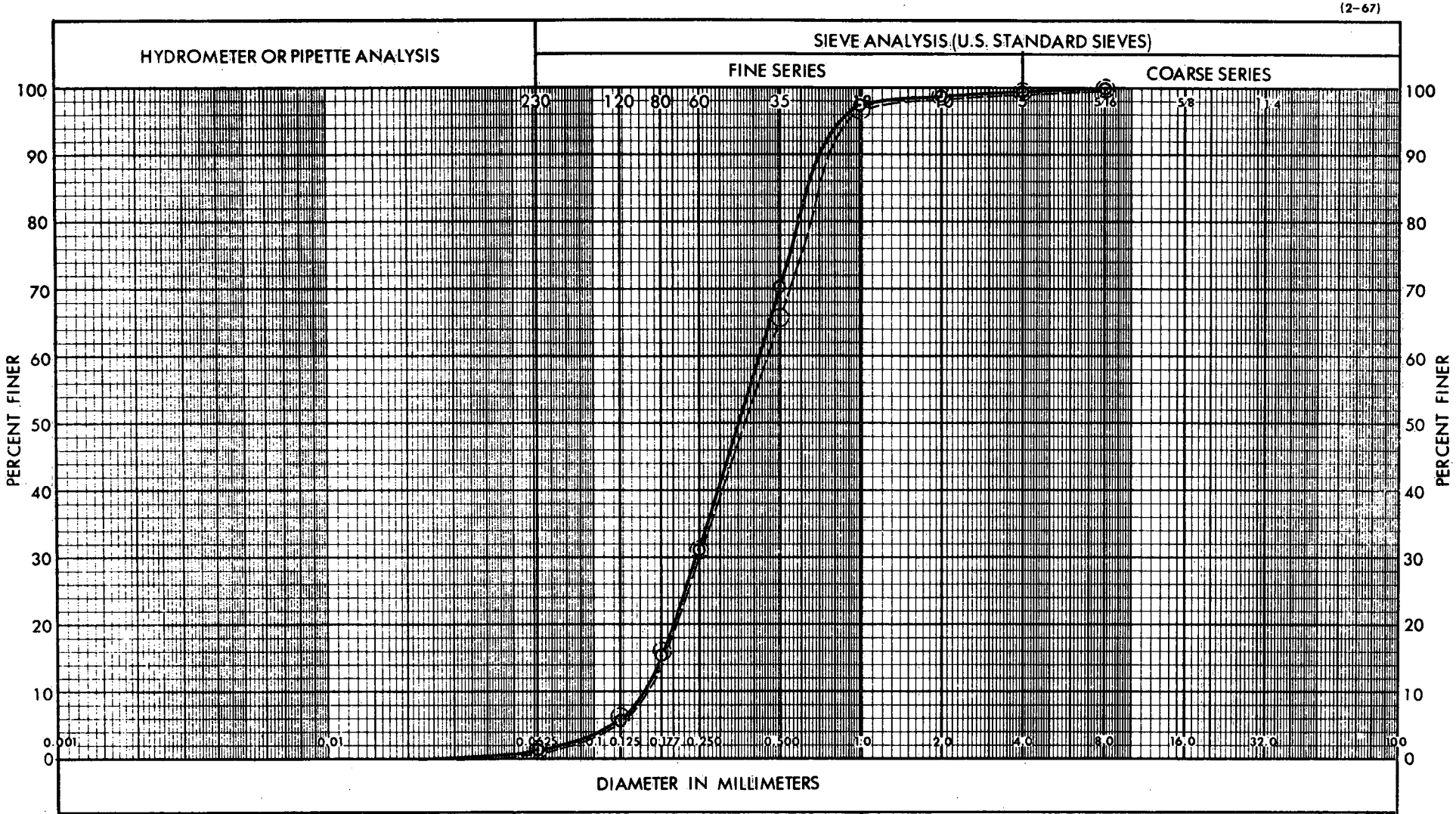

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE - DRIED 3 A

OVEN - DRIED 3 B

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BED LOAD AND BED MATERIAL

FIGURE 6

Test (b)

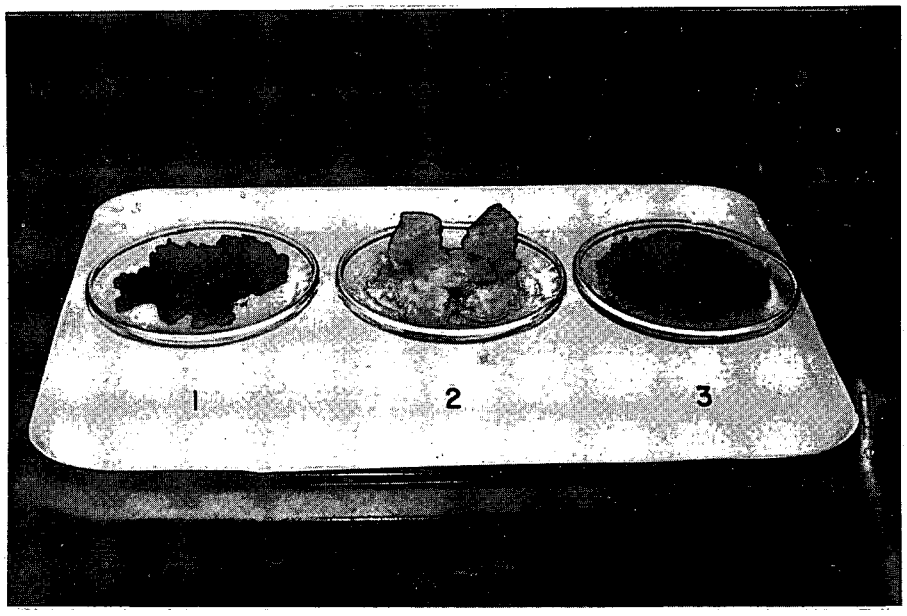
Because of the extreme temperature ranges, glass beakers containing the samples with high silt and clay fractions proved to be unsatisfactory when placed in the freeze flask for freeze-drying. However, 300-mL Nalgene bottles with their tops removed held up very well during freeze-drying. Each container was covered with a Kimwipe tissue which was held in place by a rubber band. The tissue prevented the slurry of bed material from splashing from the plastic bottle before it was frozen.

The oven-dried samples were very compact and required a vigorous grinding - 15 to 30 min per sample. On the other hand, the freeze-dried samples had a velvet-like appearance and required only a slight crushing - 5 min. (Fig. 7).

The freeze-dried samples tended to have a higher "percentage finer" value below 0.125 mm in diameter than their oven-dried counterparts on examination of the differences in the "percentage finer" values (Table 2) and the particle size curves (Figs. 8 to 10). This could indicate that the freeze-dried samples with high silt and clay fractions disaggregate more readily when re-wetted. This is substantiated by comparing the percentage silt values and the percentage clay values for the test samples. In each case, the percentage silt value (0.062 - 0.004 mm) is lower and the percentage clay value (less than 0.004 mm) is higher for the freeze-dried sample than for its oven-dried counterpart.

Table 2. Results of Test (b).

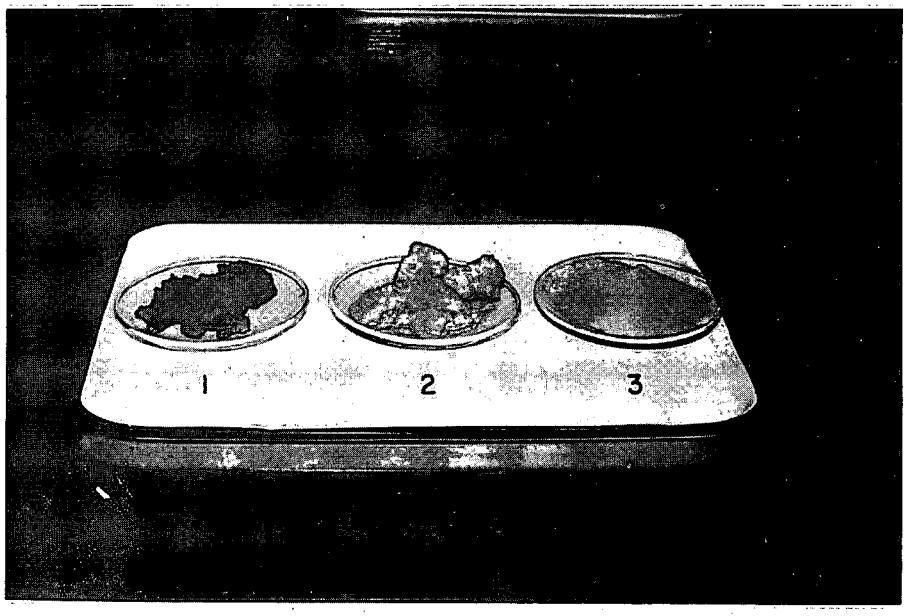
METHOD OF SAMPLE PREPARATION	SAMPLE NUMBER	TOTAL SAMPLE DRY WEIGHT (g)	PERCENTAGE FINER THAN INDICATED SIZE										% SILT 0.062-0.004 mm.	% CLAY <0.004 mm.
			U.S. STANDARD SIEVE NUMBER											
			5/8	5/16	5	10	18	35	60	80	120	230		
			OPENING IN MILLIMETRES											
			16.000	8.000	4.000	2.000	1.000	0.500	0.250	0.177	0.125	0.062		
FREEZE-DRIED	4 A	136.2				100.0	99.0	96.8	91.3	87.3	82.4	70.2	53.0	17.2
OVEN-DRIED	4 B	148.2				100.0	99.0	98.1	95.2	88.5	82.3	69.0	55.7	13.3
DIFFERENCE IN PERCENT FINER VALUES	4 A-4 B					0.0	0.0	-1.3	-3.9	-1.2	0.1	1.2	-2.7	3.9
FREEZE-DRIED	5 A	172.0				100.0	99.6	91.1	66.1	58.8	51.8	39.5	29.7	9.8
OVEN-DRIED	5 B	174.6				100.0	99.6	96.9	75.6	65.5	55.8	40.0	31.7	8.3
DIFFERENCE IN PERCENT FINER VALUES	5 A-5 B					0.0	0.0	-5.8	-9.5	-6.7	-4.0	-0.5	-2.0	1.5
FREEZE-DRIED	6A	124.1					100.0	98.9	94.3	90.8	86.5	75.5	60.8	14.7
OVEN-DRIED	6 B	121.3					100.0	99.0	96.1	93.0	88.5	75.6	65.4	10.2
DIFFERENCE IN PERCENT FINER VALUES	6 A-6 B						0.0	-0.1	-1.8	-2.2	-2.0	-0.1	-4.6	4.5



1 . WET SAMPLE

2 . OVEN-DRIED

3 . FREEZE-DRIED, BEFORE GRINDING



1 . WET SAMPLE

2 . OVEN-DRIED

3 . FREEZE-DRIED, AFTER GRINDING

FIGURE 7- FREEZE-DRIED SAMPLE
BEFORE AND AFTER GRINDING

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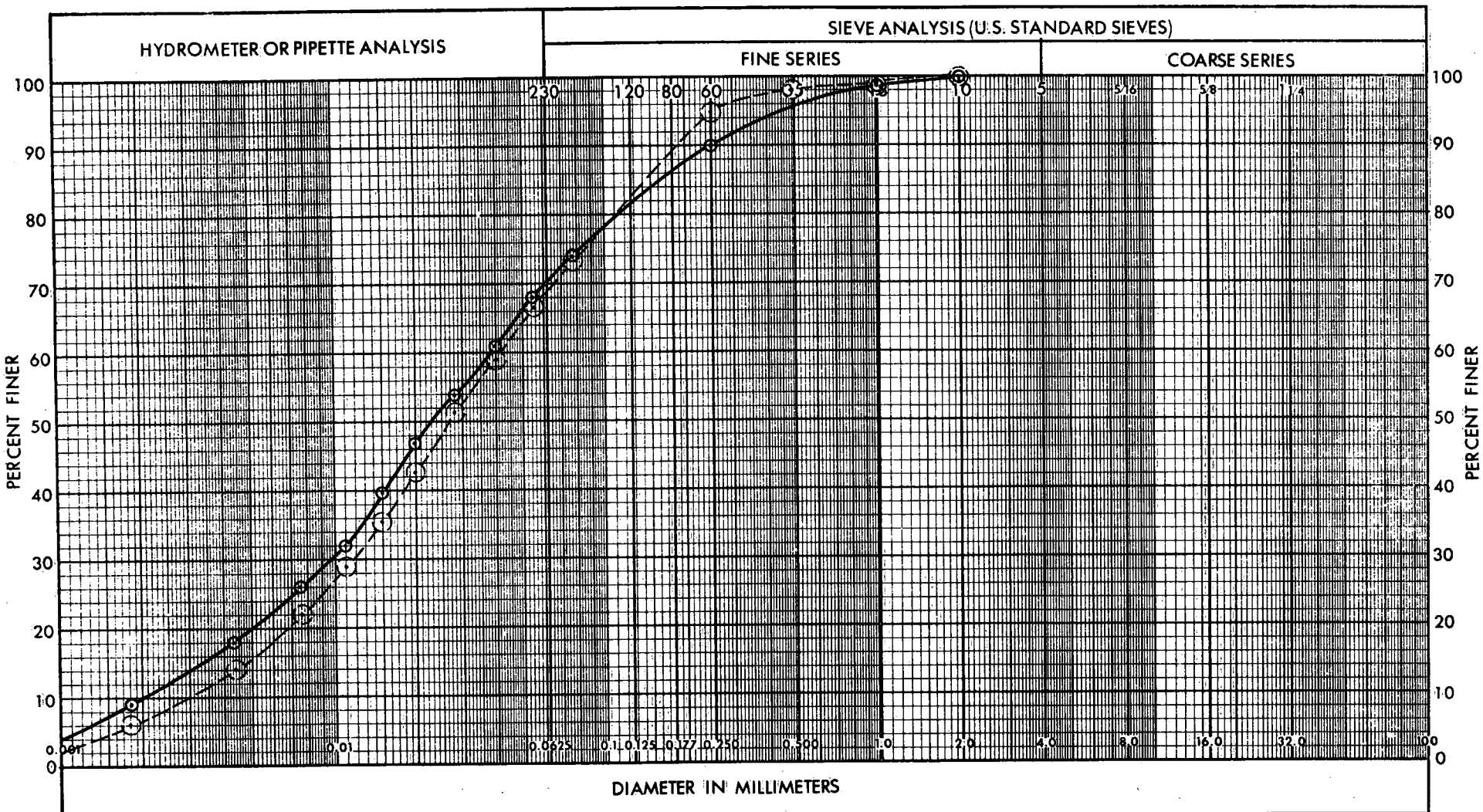

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE - DRIED 4 A —⊙—

OVEN - DRIED 4 B —⊙—

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BED LOAD AND BED MATERIAL

FIGURE 8

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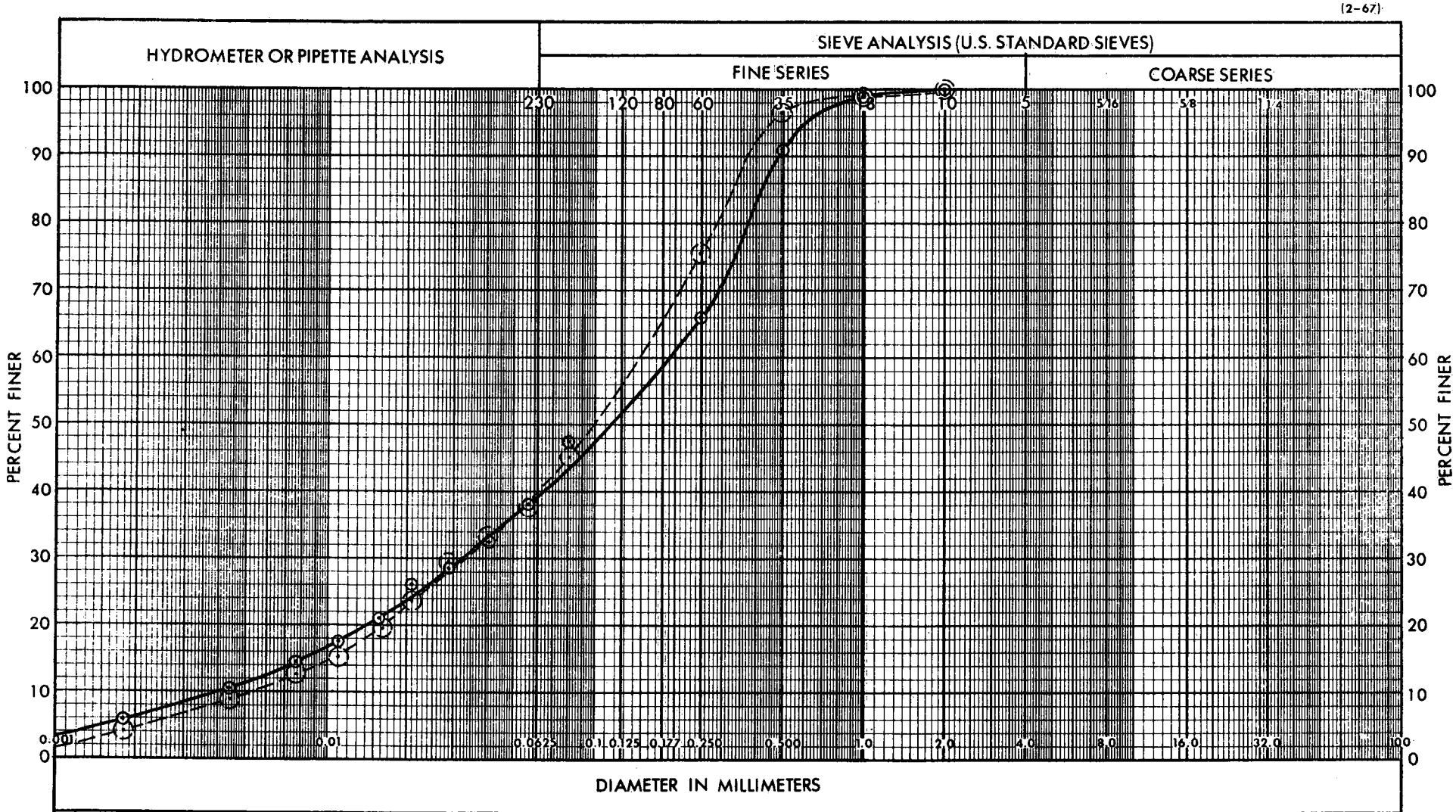

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE - DRIED 5 A —⊙—

OVEN - DRIED 5 B —⊙—

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BED LOAD AND BED MATERIAL

FIGURE 9

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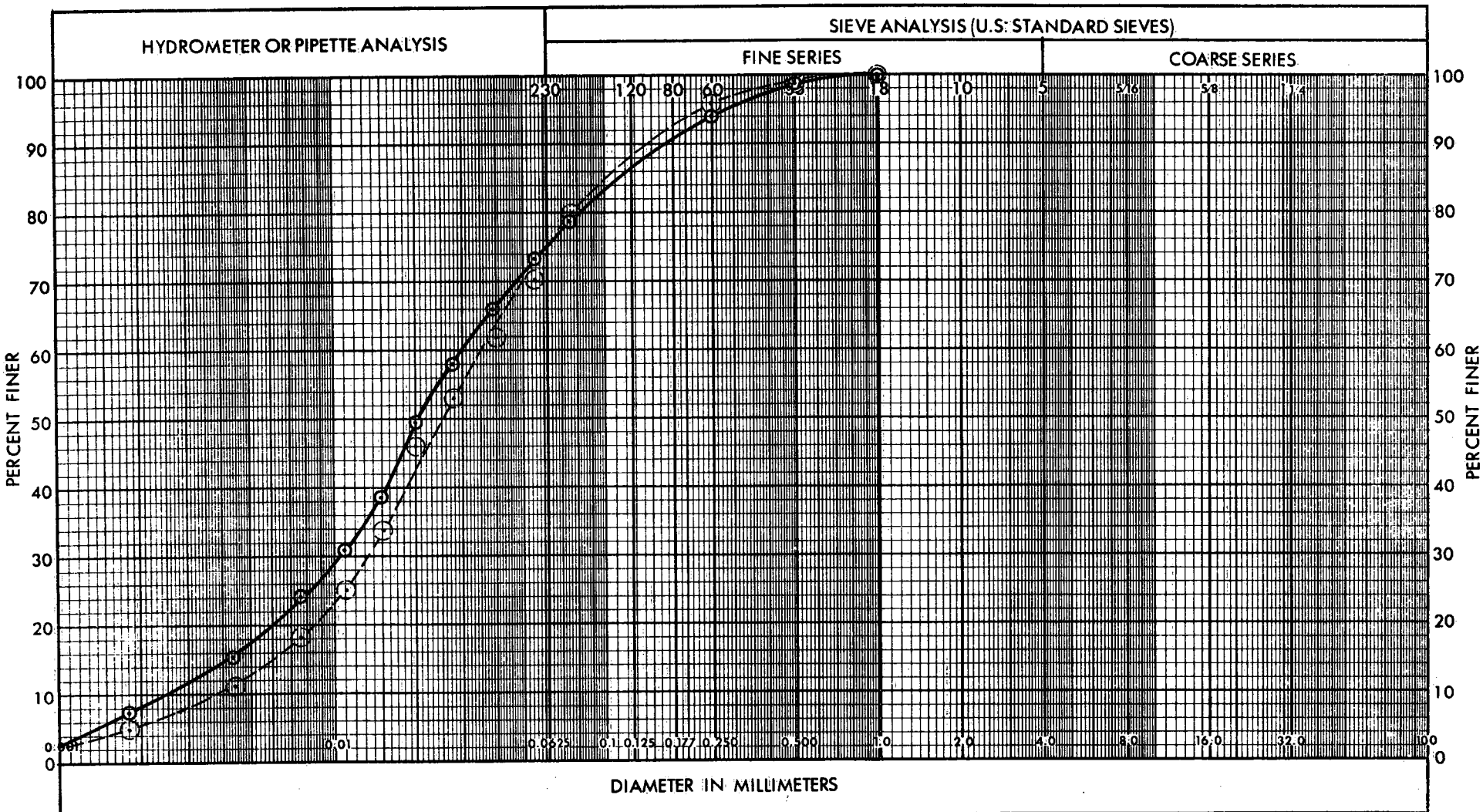

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE - DRIED 6 A —⊙—

OVEN - DRIED 6 B --⊙--

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BED LOAD AND BED MATERIAL

FIGURE 10

Test (c)

Since there was only a slight difference in the "percentage finer" values (Table 3), the particle size curves (Fig. 11) were very similar for the freeze-dried and oven-dried samples. However, the freeze-dried sample had a larger value for the percentage of clay (particle size less than 0.004 mm in diameter). Again, this could indicate that the freeze-dried sample disaggregated more completely when re-wetted.

Table 3. Results of Test (c).

METHOD OF SAMPLE PREPARATION	SAMPLE NUMBER	TOTAL SAMPLE DRY WEIGHT (g)	PERCENTAGE FINER THAN INDICATED SIZE										% SILT 0.062–0.004 mm.	% CLAY <0.004 mm.
			U.S. STANDARD SIEVE NUMBER											
			5/8	5/16	5	10	18	35	60	80	120	230		
			OPENING IN MILLIMETRES											
			16.000	8.000	4.000	2.000	1.000	0.500	0.250	0.177	0.125	0.062		
FREEZE-DRIED	7 A	49.5						100.0	84.0	76.5	69.5	55.8	34.6	21.2
OVEN-DRIED	7 B	49.3						100.0	84.5	77.8	70.5	56.5	35.7	20.8
DIFFERENCE IN PERCENT FINER VALUES	7 A-7 B							0.0	-0.5	-1.3	-1.0	-0.7	-1.1	0.4

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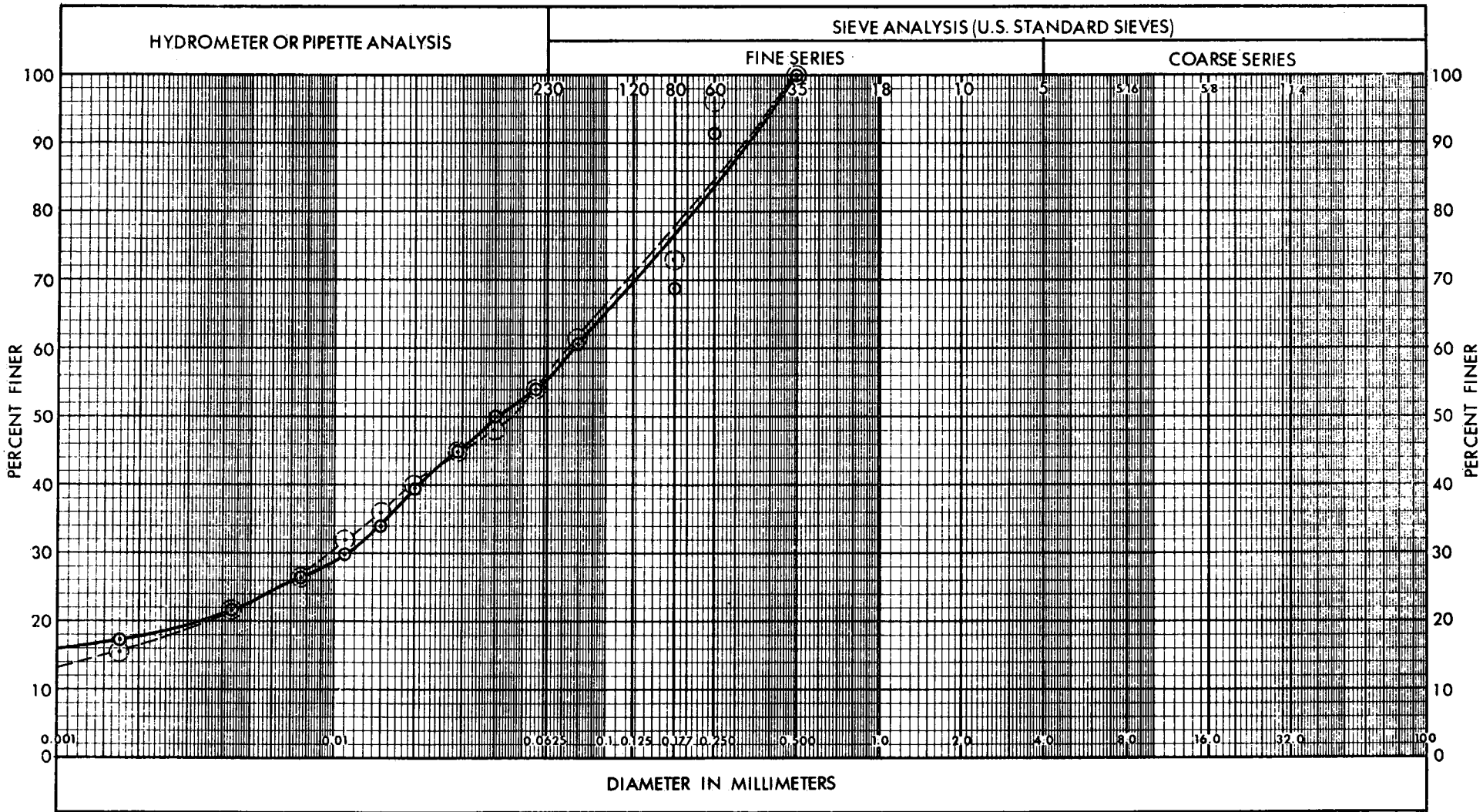

Date _____ Time of Sampling _____

Gauge Height _____ Discharge _____ cfs

Sample No. _____ Gr/lt _____

Vertical _____ Depth _____

Analysis _____

FREEZE-DRIED 7 A

OVEN-DRIED 7 B

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BED LOAD AND BED MATERIAL

FIGURE 11

CONCLUSIONS

In general, the freeze-dried samples, especially the samples with a high percentage of fine sediment particles, required only a slight crushing rather than the vigorous grinding required to reduce the oven-dried samples to a workable, homogeneous powder. From the preceding results, it was concluded that the difference between the two methods of drying was negligible. Nevertheless, the advantages of freeze-drying fine sediments are the following:

1. The size fractions are in a powdered form, and are ready for analysis or storage. No further processing of the sample is necessary.
2. There is less chance of crushing larger sediment particles because the samples do not require vigorous grinding.
3. The preparation time for each sample is reduced, resulting in a saving of man hours.

RECOMMENDATIONS

The Sediment Survey Section, Ottawa, recommends that the Regional Offices consider purchasing a Freeze Dryer unit for our Sediment Laboratories. The use of a Freeze Dryer not only reduces the handling of bed material samples but also reduces the grinding time required for each sample by approximately 15 to 20 min.

It has been proven by our test samples (Figs. 4-6, 8-11) that the difference in the "percentage finer" particle size distribution between the oven-dried and freeze-dried samples is negligible.

The Freeze Dryer is available in various sizes, but the one we would recommend is the Freeze Dryer-3, (12-port chamber) available from Labconco Coporation, Kansas City, Missouri, through Fisher Scientific Company Limited.

The following is a list of the associated equipment required and the price as of April 1, 1978.

	ITEMS	CATALOG NO.	PRICE
1	Freeze Dryer-3 (12-port chamber)	75200	$1395.00
1	Vacuum gauge	75800	505.00
1	Vacuum pump		572.00
6	Fast-freeze flasks, 600 mL	75408	279.00
6	Fast-freeze flasks, 1200 mL	75410	300.00
12	Fast-freeze 45° adaptors (glass)	75458	35.40
		TOTAL	$3086.40

FREEZE DRYER OPERATION AND USE

Steps in the Freeze-Drying Process

The freeze-drying process follows two steps:

(a) primary drying, in which the ice evaporates, or sublimes, without melting, and

(b) secondary drying, in which some of the moisture that is still held by the material is allowed to sublimate, leaving a residue which is critical to the stability of the sample.

The basic requirements for freeze-drying a sample are:

1. Pre-frozen samples, if possible, in suitable containers.
2. A chamber or manifold into which the containers may be placed.
3. A vacuum system to facilitate vapour flow.
4. A heat source for evolution of vapour.
5. A vapour trap to prevent large quantities of vapour from contaminating the vacuum pump.

Accessories which measure vacuum and temperature are also important to the overall success of the freeze-drying operation. There are a vast number of combinations of these requirements which will result in efficient freeze-drying, such as the use of a mechanical refrigeration system; the use of a manifold or drying chamber; the use of freeze-drying flasks or other containers.

Freeze-drying is carried out under vacuum to allow the water vapour which sublimes from the ice to flow easily from the pores and away from the remaining material.

Heat energy is needed to change ice into vapour. The sample will reach an equilibrium temperature which is a balance between the heating

effect of the surroundings and the cooling effect produced by the sublimation of water vapour from the sample.

Because vacuum is also a good insulator, the material placed in containers inside a vacuum chamber usually dries slowly, therefore the system is run overnight.

Figure 12 shows what actually happens when freeze-drying takes place. Water vapour sublimes from the surface of the material and is drawn into the vacuum trap.

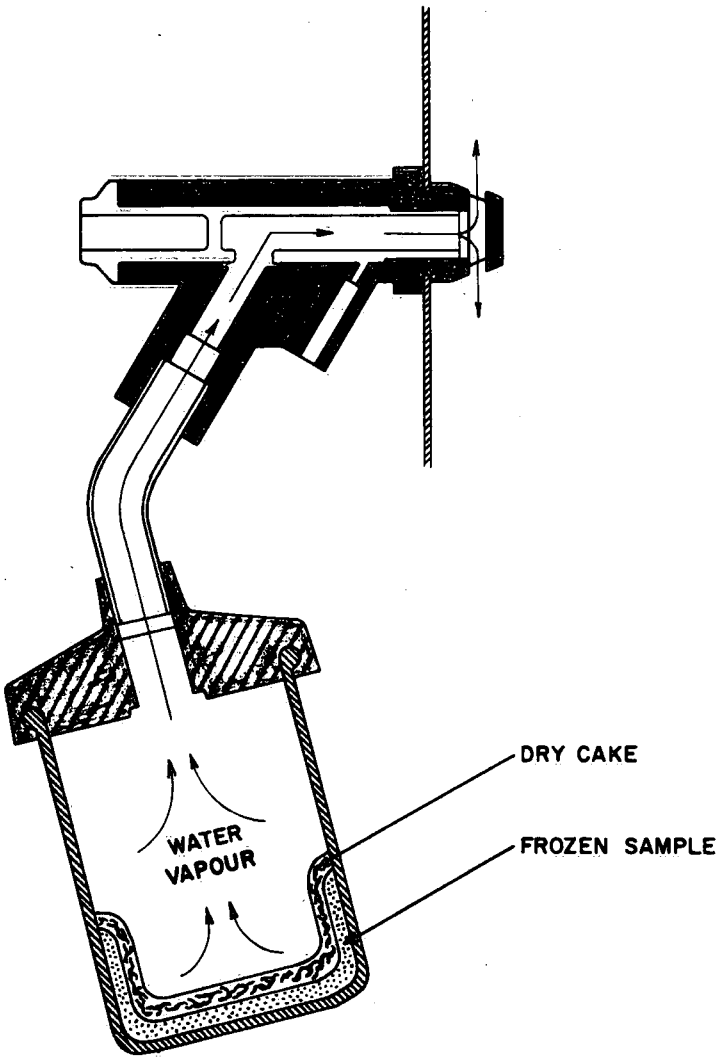


FIGURE 12 - FREEZE-DRYING FLASK AND VALVE

Steps to Operate Freeze Dryer

1. Clean the rubber gasket on the clear acrylic drying chamber closure to remove dirt and other contaminants that could be a source of vacuum leaks. Vacuum grease is not usually required to obtain a vacuum tight seal. If grease is used, apply only a thin film of a silicone high-vacuum grease.
2. Remove any moisture within the drying chamber by wiping with a soft cloth or paper towel. Close the drying chamber with the acrylic cover.
3. Close all manifold valves on the drying chamber (except the $\frac{3}{4}$-in. valve connected to the vacuum outlet). The valves are closed when the word "VENT" is up.
4. The $\frac{3}{4}$-in. vacuum outlet valve must be open ("VAC" position up).
5. Check to see if the plug is secured in the drain line for the water vapour condenser.
6. Turn on the condenser refrigeration and allow condenser temperature to reach approximately -50°C or lower.
7. Check vacuum pump oil condition (clear) and quantity (level marking) before starting the pump. Change the oil if it is dirty or cloudy.
8. Turn on the vacuum pump and allow the system pressure to reach 25 μm Hg (.025 Torr) or lower.
9. If there are no vacuum leaks and the vacuum pump is operating properly, the Freeze Dryer-3 should obtain no-load condenser temperature of -45°C or lower and a system pressure of 10 μm (.010 Torr) or lower. The Freeze Dryer-3 is now ready for freeze-drying.

   NOTE: 1 millitorr = 1 μm Hg = 1×10^{-3} torr

   1 torr = 1 mm Hg

Freeze-Drying

1. After connecting the freeze flask containing the sample to a vacuum valve, turn the plastic valve knob to the "VAC" up position to open the valve.
2. Allow the system pressure to return to a vacuum of 100 μm (.1 Torr) or lower before adding another sample. Any combination of valves and sample sizes may be used at one time provided that the system vacuum remains below 100 μm and the condenser temperature remains below -40°C.
3. Allow the samples to freeze-dry. Ambient room temperature plus water vapour in the room condensing on the outer surface of the sample container will usually supply sufficient heat for efficient sublimation.
4. End Point: When all of the frost has disappeared from the outer surface of the sample container and no cold spots can be detected by handling the container, the sample is nearly dry. It is wise to continue drying for several more hours to be certain of low final moisture content (less than 1% residual moisture content).
5. To remove a flask after drying is complete, turn the plastic knob to closed position ("VENT" in up position).

NOTE: Keep the vacuum gauge on the freeze dryer cabinet, as the vibrations keep the gauge indicator moving freely in its bearings.

Defrosting

1. Turn off the machine in the opposite sequence:
   (a) turn off vacuum pump,
   (b) release system vacuum by opening one or more vacuum valves,
   (c) turn off the condenser refrigeration.

2. Open front access door, place the drain hose for the water vapour condenser in a suitable receptacle and remove the drain plug.
3. The ice on the stainless steel condensing coils may simply be permitted to melt and drain.
4. For rapid defrost, simply pour warm water over the coils. Ice will fall off the coils and may be removed by hand. DO NOT ATTEMPT TO CHIP ICE FROM THE COILS. THIS CAN SERIOUSLY DAMAGE THE REFRIGERATION SYSTEM.

BED MATERIAL HYDROMETER ANALYSIS

The purpose of the hydrometer analysis is to determine the individual grain sizes of the fine-grained particles of a bed material sample. This analysis is performed on material passing the 1.0 mm sieve (U.S. sieve series No. 18). The hydrometer analysis must be done if more than 10% of a grain size sample passes the 63 μm sieve (U.S. sieve series No. 230).

Procedure for Hydrometer Analysis

1. Weigh out the required amount of sample:
   - for sandy and/or silty material, 60 g
   - for clays, 40 g
2. Place the weighed sample in a 400-mL beaker; add the dispersing agent (calgon, 1 g). Mix thoroughly with about 100-200 mL of distilled water and let soak overnight. This allows any adhering particles to separate.
3. Pour approximately 500 mL of distilled water and the sample into the mechanical mixing cup. Mix for 1 min.
4. Transfer the mixed sample into a 1000-mL graduated cylinder. Add distilled water until the liquid level reaches the 1000-mL graduation.
5. Place the cylinder in the constant temperature bath if one is available and let stand until the temperatures are equalized. A constant temperature bath will eliminate the need for periodic temperature readings during the analysis.
6. Using a perforated brass stirring rod, mix the suspension for 1 min. Use long even strokes, trying not to bring the rod out of the suspension; if the rod breaks the surface, it creates a sudsy foam which inhibits the reading of the hydrometer.

7. Immediately after stirring, start the timer and slowly immerse the hydrometer to a depth slightly beyond its floating point and then allow it to float freely. Take readings at intervals of 30 sec, 1 min, 2 min, 4 min, 8 min, 15 min, 30 min, 60 min, 3 hr and 20 hr. The readings may be discontinued when either the 10% finer or the 2-μm reading has been reached.

NOTE:

(a) Inserting and removing the hydrometer must be done carefully; allow about 10 sec for each operation.

(b) Initially do not remove the hydrometer from the sample until after the 2-min reading. Dry the hydrometer after each removal.

(c) The hydrometer reading is taken at the upper rim of the meniscus on the hydrometer stem. If the meniscus is not fully developed around the stem, it could be caused by greasy fingerprints, etc. The hydrometer should be washed occasionally with soap and water.

8. Calculate W% by making use of the nomographic chart and the observed data in the formula

$$W\% = \frac{100}{W_s} \cdot \frac{S_s}{S_s - 1} \cdot (R_H + m_T - C_d)$$

The quantity $\frac{100}{W_s} \times \frac{S_s}{S_s - 1}$ is a constant for any given test.

W% is the percentage by weight containing all grains smaller than the grain size D.

W_s is the weight of the sample used in the analysis.

C_m is the meniscus correction, which is a constant for each hydrometer and is dependent on the diameter of the hydrometer stem.

C_d is the correction for the increase in density of the liquid phase of the suspension due to the addition of a dispersing agent. This quantity is determined by observing the difference in the hydrometer readings obtained by inserting the hydrometer first in distilled water and then in a solution of this water containing the amount of dispersing agent which is used in a given test.

m_T is the correction due to the temperature. This value may be obtained from the temperature correction chart (Fig. 13).

R_H is the hydrometer reading taken at the upper rim of the meniscus. The reading may be 1.0225 but should be recorded as $R_H = 22.5$.

D is the grain size determined by means of the nomographic solution of Stokes' Law.

S_s is the specific gravity of soil.

γ_s is the specific gravity of solid.

Use of the Nomographic Chart

Each hydrometer requires a separate nomographic chart (Fig. 14) because the "R_H" scale depends on the dimensions of the instrument.

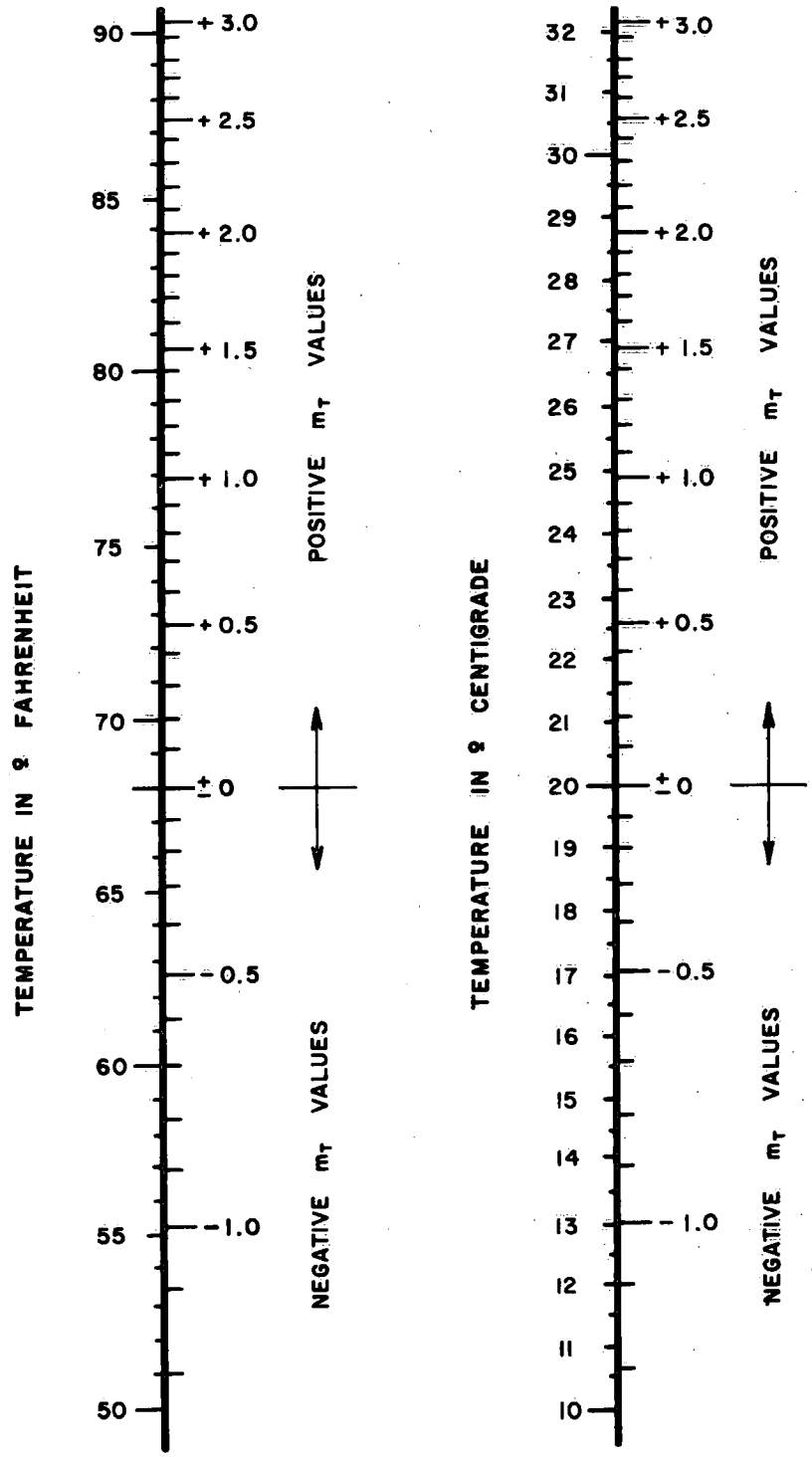
The intersection of a straight line across the "R_H" and "t" scales yields the velocity on the "v" scale. Hold this point and rotate the straightedge until it intersects the "B" reading corresponding to the γ_s and the temperature. In this position the intersection with the "D" scale represents the grain size.

NOTE : W% = FRACTION CONTAINING ALL GRAINS SMALLER THAN CORRESPONDING GRAIN SIZE IN PERCENT OF TOTAL SAMPLE (W_s)

S_s = SPECIFIC GRAVITY OF SOIL.
R_H = $(r_H - 1)\,10^3$
r_H = DENSITY READING ON HYDROMETER
C_d = DENSITY CORRECTION
m_T = TEMPERATURE CORRECTION

TO BE COMPUTED FROM EQUATION :

$$W\% = \frac{S_s}{S_s - 1}\,\frac{100}{W_s}\,(R_H + m_T - C_d)$$



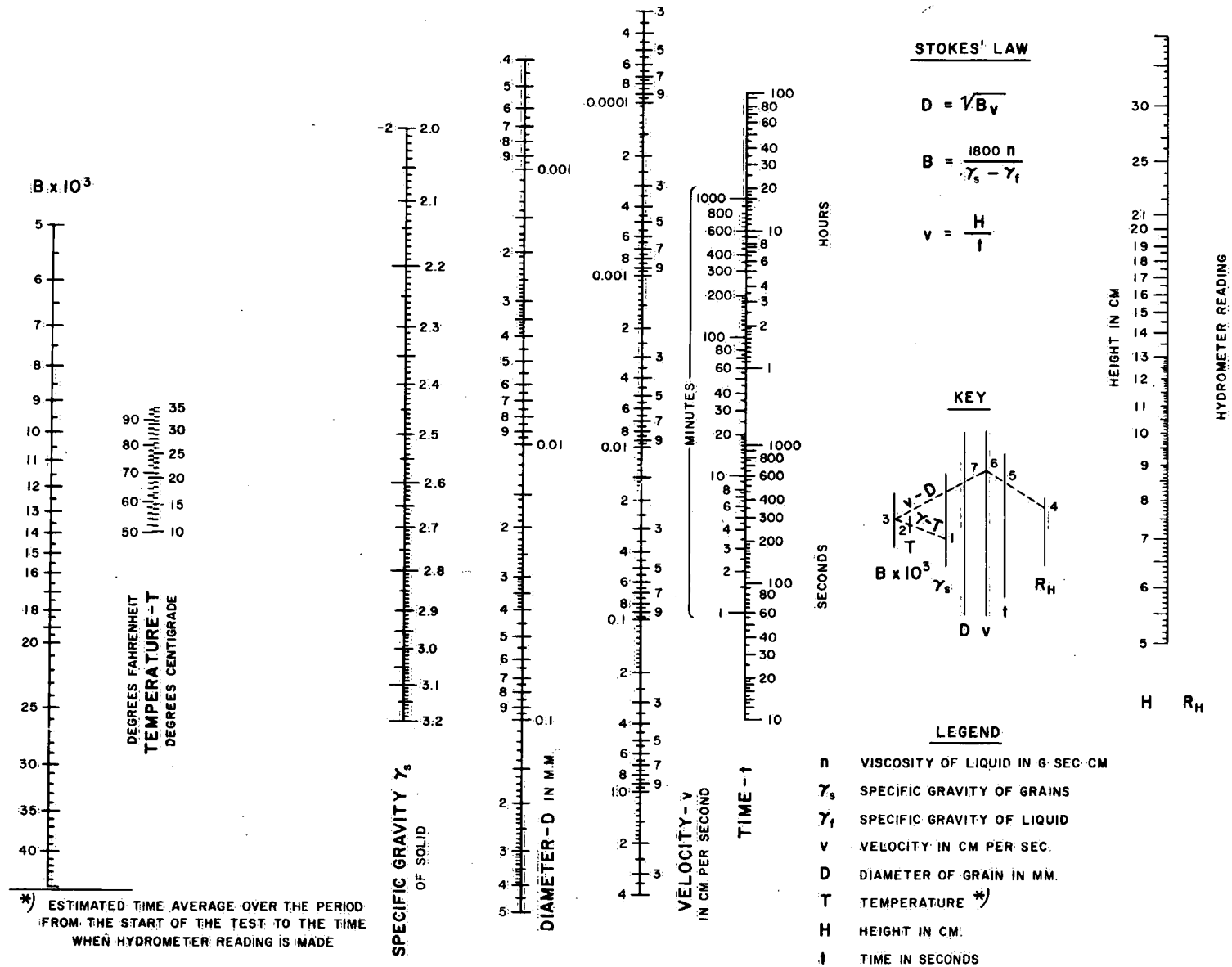
T = 68°F = 20°C

TEMPERATURE CORRECTION CHART

FOR HYDROMETERS CALIBRATED IN DENSITY AT TEMPERATURE 'T'

FIGURE 13

NOMOGRAPHIC CHART FOR SOLUTION OF STOKES' LAW



*) ESTIMATED TIME AVERAGE OVER THE PERIOD FROM THE START OF THE TEST TO THE TIME WHEN HYDROMETER READING IS MADE

FIGURE 14

WET SIEVE ANALYSIS AFTER HYDROMETER ANALYSIS

Using the portion of the bed material sample in the hydrometer cylinder and/or a larger representative portion of the original sample, determine the grain size distribution for particles larger than 63 μm. To accomplish this, wet sieve the sample through a No. 230 sieve and allow the material to dry. The dried sample is then put on the sieve shaker for 10 min to sieve through the fine sieves (Nos. 18, 35, 60, 80, 120 and 230). The weight of the material retained on each sieve is recorded and its percentage of the total is computed (Fig. 15). A particle size distribution curve (Fig. 16) is plotted using the "percentage finer than" values from the wet sieve analysis and the W% values from the hydrometer analysis.

DEPARTMENT OF THE ENVIRONMENT
INLAND WATERS BRANCH–WATER SURVEY OF CANADA

R 270
(9-67)

Stream EXAMPLE
Type of Sampler _____ Vertical 350
Sample Number T-4097 Sample taken by _____ Date APRIL 10, 1977

HYDROMETER ANALYSIS

DATE	TEMP. °C	TIME	ELAPSED TIME	R'_H	$R_H = R'_H + C_m$	D mm	$R_H + m_T - C_d$	W%	% BASIS ORIGINAL SAMPLE	REMARKS
	20.5	1:00	0.0							Mt
	"	1:00.5	0.5	18.2	18.7	0.077	18.5	74.3		0.1
	"	1:01	1	16.7	17.2	0.055	17.0	68.3		"
	"	1:02	2	14.9	15.4	0.040	15.2	61.0		"
	"	1:04	4	13.2	13.7	0.028	13.5	54.2		"
	"	1:08	8	11.4	11.9	0.020	11.7	47.0		"
	"	1:15	15	9.6	10.1	0.015	9.9	39.7		"
	20.6	1:30	30	7.7	8.2	0.011	8.0	32.1		"
	20.3	2:00	60	6.2	6.7	.0075	6.5	26.1		"
	20.7	4:00	3 hr.	4.2	4.7	.0043	4.5	18.1		"
	21.8	9:00	20 hr.	1.6	2.1	.0018	2.2	8.8		0.4

Analyzed by _____ Computed by _____ Checked by _____

HYDROMETER NUMBER I

$W\% = \frac{100}{W_S} \cdot \frac{S_S}{S_S - 1} \cdot (R_H + m_T - C_d) =$ 4.015 $(R_H + m_T - C_d)$

MENISCUS CORRECTION = C_m = 0.5 SPECIFIC GRAVITY OF SOLIDS = G = 2.65

DISPERSING AGENT USED CALGON AMOUNT 1.0 grams

C_d = 0.3 = CORRECTION FOR INCREASE IN DENSITY OF LIQUID DUE TO ADDITION OF DISPERSING AGENT

m_T = _____

DESCRIPTION OF SAMPLE _____

METHOD OF PREPARATION _____

REMARKS: _____

WEIGHT SAMPLE AND DISH (DRY) gr. _____
WEIGHT DISH gr. _____
WEIGHT SAMPLE gr. 40.0

SIEVE ANALYSIS AFTER HYDROMETER ANALYSIS

TOTAL WT. 136.2

SIEVE NO.	SIZE OF OPENING MM	WEIGHT RETAINED gr.	TOTAL WT. FINER THAN gr.	PERCENT FINER THAN	% FINER THAN BASIS ORIG. SAMPLE
5	4.000				
10	2.000		10.1	100.0	
18	1.000	0.1	10.0	99.0	
35	0.500	0.1	9.9	98.0	
60	0.250	0.8	9.1	90.1	
80	0.177	1.5	7.6	75.2	
120	0.125	2.2	5.4	53.5	
PASS 120	0.063	4.2	1.2	11.9	
	PAN	1.2			

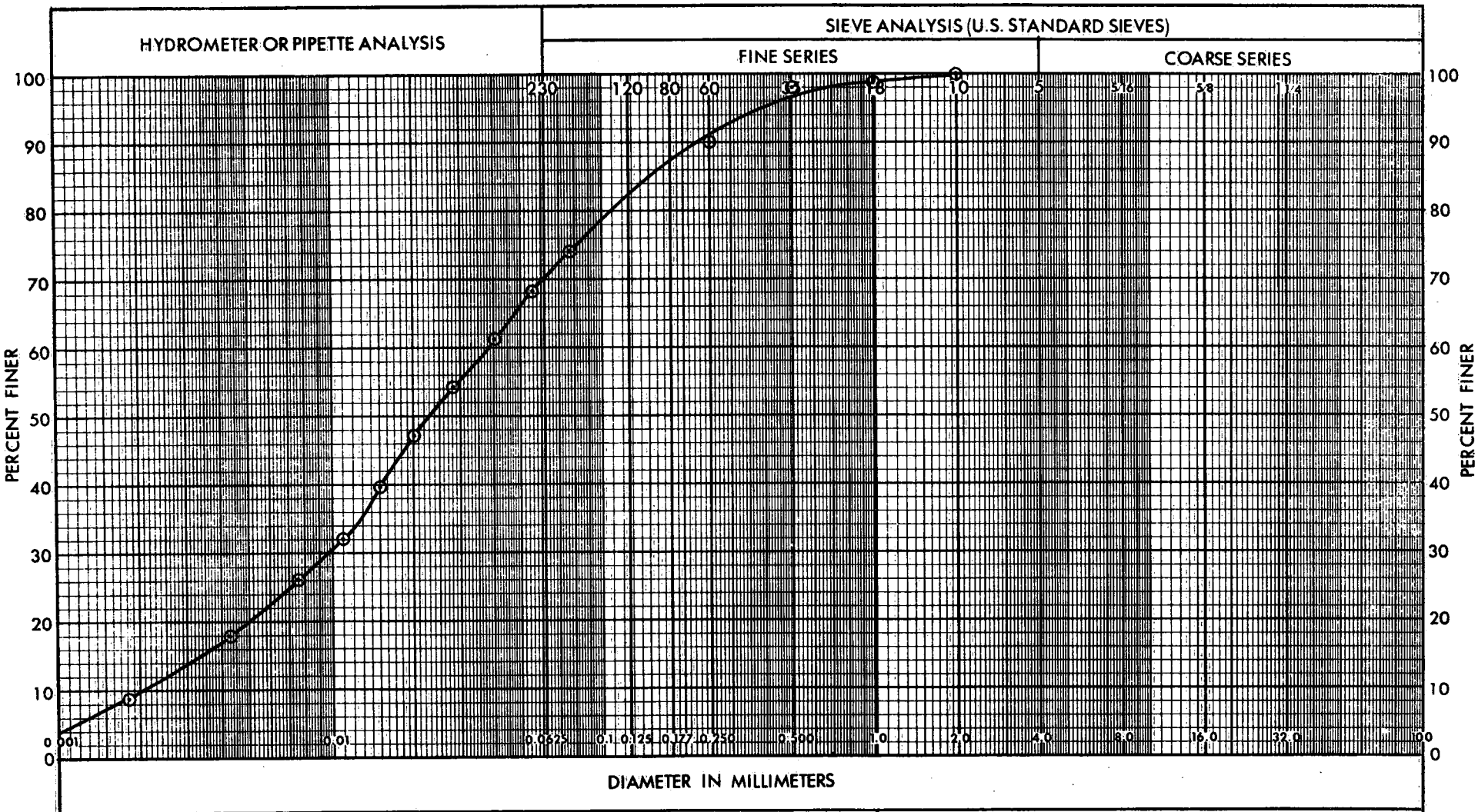
WET SIEVE WT. 10.1

DRY WEIGHT OF SAMPLE BEFORE WASHING 40.0 gr.
DRY WEIGHT OF SAMPLE AFTER WASHING _____ gr.
DRY WEIGHT OF SAMPLE PASSING NO. 230 _____ gr.

FIGURE 15

G9–10172

R.–286
(2-67)



Date APRIL 10/1977 Time of Sampling 1325

Gauge Height _____ Discharge _____ cfs

Sample No. T-4097 Gr/lt _____

Vertical 350 Depth _____

Analysis HYDROMETER (SWH)

DEPARTMENT OF ENERGY, MINES AND RESOURCES
INLAND WATERS BRANCH

BED LOAD AND BED MATERIAL

EXAMPLE

FIGURE 16

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