A STUDY OF METHODS USED IN

MEASURMENT AND ANALYSIS OF SEDIMENT LOADS IN STREAMS



REPORT Z

THEORY AND OPERATION MANUAL FOR THE AUTOPIPET SEMIAUTOMATIC PIPET WITHDRAWAL APPARATUS

A Study of Methods Used in MEASUREMENT AND ANALYSIS OF SEDIMENT LOADS IN STREAMS

A Cooperative Project
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Geological Survey ** Corps of Engineers

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Soil Conservation Service ** Tennessee Valley Authority

Office of Surface Mining ** Forest Service

REPORT Z

THEORY AND OPERATION MANUAL FOR THE AUTOPIPET SEMIAUTOMATIC PIPET WITHDRAWAL APPARATUS

Ву

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THEORY AND OPERATION MANUAL FOR THE AUTOPIPET SEMIAUTOMATIC PIPET WITHDRAWAL APPARATUS

By Joseph P. Beverage

ABSTRACT

The autopipet is a semiautomatic withdrawal apparatus which obtains unattended the six scheduled withdrawals (for 62, 31, 16, 8, 4, and 2 micrometers) of the pipet particle-size procedure. A fixed-elevation, 12-depth siphon sampling scheme is used instead of mechanically lowering the pipet to a predetermined depth for each withdrawal. An optical water-level sensor stops the siphon when the correct volume is obtained. A wasted withdrawal precedes each of the scheduled subsamples to flush the siphon line and to fill the line with a suspension of nearly the same concentration as the subsequent sample.

A brief description of particle settling theory and of the compromises made during the design are given. Operational procedures, circuit descriptions, and a troubleshooting guide are given in appendices.

INTRODUCTION

Information on particle-size distributions of river sediments is essential for many hydrologic studies. The distributions are combined with water discharge for estimating total sediment transport and for predicting deposition patterns in rivers, lakes, and reservoirs. Other uses include studies of soil loss, transport of toxic substances by fine sediments, and the ecological effects of sediment.

Few naturally occurring sediment particles have a smooth spherical shape; most have rough, fractured surfaces and irregular shapes. Regardless, the principal requirements of particle-size distributions are not for the true, physical dimensions of the particles. Rather, a particle-size analysis of river-born sediment is preferred in terms of the fall (settling) diameter and is usually a composite distribution obtained from two methods of analysis. Particles larger than 62 micrometers (μ m) are analyzed by visual-accumulation tube (ICWR Rept. 11, 1957) method. Particles smaller than 62 μ m are mainly analyzed by the pipet method (ICWR Rept. 4, 1941; Guy, 1969).

The conventional pipet procedure is labor intensive. First a prepared sample (Appendix III) is placed in a cylinder. It is mixed and an aliquot is obtained for a concentration determination. Then the operator must manually collect subsamples from the cylinder at predetermined intervals. The collection procedure is to lower the volumetric pipet to the correct depth in the suspension, withdraw an exact volume at the specified time, withdraw the pipet, deliver the subsample to an evaporation dish, and flush and drain the pipet before continuing to successive samples. Several withdrawals are needed to define a complete particlesize distribution. The early withdrawals occur rapidly one after the other, keeping the operator quite busy. Later withdrawals are sufficiently separated to allow the operator to work at something else. Also, the usual practice of performing 6 to 10 or more analyses simultaneously by using staggered withdrawal times further complicates the operator's job. However, the operator must remain aware of the time while doing other things. The apparatus described in this report, the autopipet,

minimizes the labor of obtaining the scheduled pipet subsamples. Once started, the apparatus automatically withdraws the subsamples at the required times.

This report provides a brief, qualitative description of particle settling theory and of the compromises made during the design of the apparatus. The apparatus itself is described in Appendix I along with instructions for installation. Further appendices describe the initial setting up, sample preparation, operation, circuit descriptions, trouble-shooting guide, and a sample withdrawal calculation.

Acknowledgements

J. V. Skinner, U.S. Geological Survey, proposed the fundamental concept of the fixed-elevation (decreasing depth) sampling procedure used in the autopipet design. He designed and built a proof-of-principle model of the sample stand and performed preliminary tests. He also did all mechanical design of the final model autopipet and designed part of the electronics of the sample stand as well. Many helpful suggestions were offered by Dr. L. L. McDowell and associates of the Agricultural Research Service Sedimentation Laboratory at Oxford, Mississippi. Florence Wright, U.S. Geological Survey, typed the manuscript.

THEORY

The autopipet was designed to mechanize most of the subsample withdrawal portion of the pipet procedure (ICWR Rept. 4, 1941, p. 84-90, and Guy, 1969, p. 23-29) for determining particle-size distribution of the silt and clay fraction (< 62 μm) of a soil or sediment sample. The standard pipet procedure begins with a cylinder containing a well-mixed suspension of the fine sediments. Subsamples are withdrawn by volumetric pipet from a fixed depth at time intervals predetermined for the fluid temperature. The pipetted subsamples steadily decrease in concentration in response to the smaller fractions of the finer material. If allowed to settle undisturbed, particles which settle faster than others soon decrease the concentration of the upper layers. At a fixed depth and temperature, the change in concentration with time is related directly to the particle-size distribution.

An equation describing the terminal fall velocity of a particle sufficiently large to overcome Brownian movement was derived by G. G. Stokes in 1851. Stokes balanced the viscous drag on a sphere and the buoyant force of the liquid with the force of gravity. The assumptions and derivation are given in Rept. 4 (ICWR, 1941, p. 29-37). A simplified form of Stokes' equation is (ICWR, 1941, p. 30):

$$\omega = \frac{89.83d^2}{u}$$
 , (1)

where ω is the particle fall velocity in centimeters per second (cm/s), d is the particle diameter in centimeters (cm), and μ is the dynamic (absolute) viscosity of water in dyne-seconds per centimeter (poise). Recasting the equation in more useful form and in more recent units (Petersen, 1980):

$$t = \frac{1.113 \times 10^7 \text{ h}\mu}{\text{d}^2} , \qquad (2)$$

where t is the time (seconds) required for a particle to settle through a distance h (centimeters), μ is the dynamic viscosity in pascal-seconds (Pa·s), and d is in micrometers. The following table gives values of μ

for even temperatures between 20° and 30°C (Celsius) (Bolz and Tuve, 1970, p. 67):

Temperature	Viscosity, μ						
20° C	0.001 002 Pa·s						
22	.000 9548						
24	.000 9111						
26	.000 8705						
28	.000 8327						
30	.000 7975						

Equations 1 and 2 are based on a particle specific gravity of 2.65 for spherical particles falling in water. The assumptions of spherical shape and the specific gravity of quartz are needed in studies where the quartz-equivalent settling diameter (regardless of its shape or specific gravity) is more useful than the physical dimensions of the particle.

It is important that the settling process be understood. Visualize a cylinder filled with a nonturbulent quiescent sediment suspension which at the beginning of the analysis is uniformly mixed. Next, examine a thin, horizontal slice of the suspension at the surface. The concentration of sediment in this slice will decrease as time progresses. Sediment settling from the slice is not replaced by material from above, so the concentration gradually decreases with time. The largest particles leave the slice most rapidly because they have the greatest fall velocity. As the larger particles leave, the median diameter of the remaining particles decreases.

Let us next examine a thin horizontal slice of the suspension at a depth of, say, 10 cm. Particles that fall from this slice are replaced by other particles that enter the volume from above. There will be no change in concentration until the suspension above the slice has been depleted. The concentration within this slice will begin to diminish when the largest particles have fallen through the volume above this slice. This time may be calculated by dividing the 10-cm depth by the fall velocity of the particles in the chosen size range. Once this time has elapsed without disturbance, there will be no large particles available from above for replenishment of the slice's larger-sized particles.

The slice at 10-cm depth will thenceforward behave in a manner similar to the surface slice, except that the deeper the slice the greater the spread between time-of-depletion of disparate particle sizes. For example, at 30°C a 62-um particle will clear a 1-millimeter (mm) thick surface slice in a quarter of a second while a 2-µm particle will take about 3 minutes and 40 seconds. The spread between these sizes is about 3 minutes and 40 seconds. At a depth of 10 cm, $62-\mu m$ particles will have cleared after 23 seconds, while 2-µm particles will take six hours and ten minutes. The spread is now about six hours. Shifting the observation slice downwards has stretched out the difference in time of depletion for different sizes. The converse is also true. In the standard pipet procedure this converse relation is used to reduce waiting time. The initial (62 μ m) withdrawal depth might be a 15-cm depth, intermediate withdrawals at 10 cm, and the final (2 µm) withdrawal at 3 cm (Guy, 1969, p. 24). The withdrawal depth is thus an important variable which determines the time a withdrawal must be made.

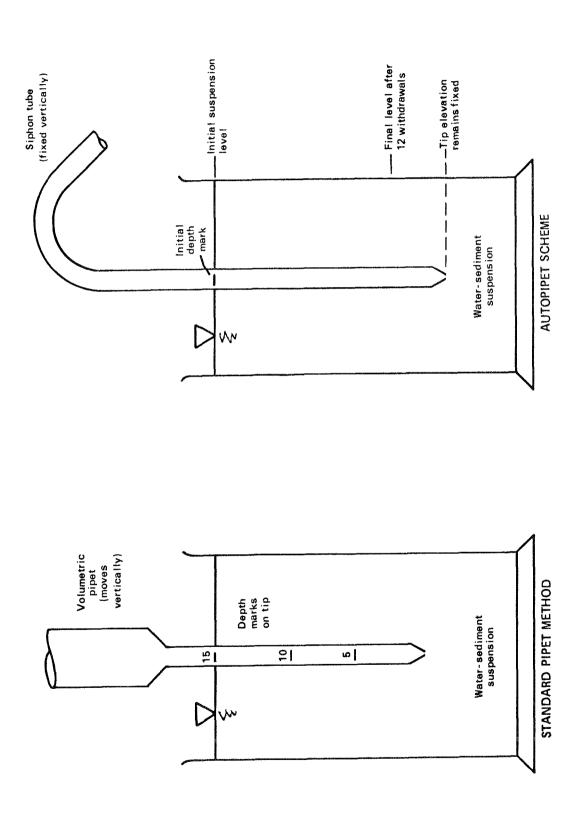
Another important parameter is the water temperature. The viscosity of the water is related to its temperature, decreasing by 20 percent as it warms from 20°C to 30°C. Suspension temperatures thus are normally maintained at a constant value (within 1°C or less of the initial value) to minimize the effect of viscosity.

The pipet analysis is based on the assumption that each particle settles at a rate unaffected by other particles. Thus, another variable is the influence of particles on each other. A large particle will drop rapidly through a suspension, dragging many smaller particles along in its wake. The wake will create turbulent eddies which will serve to stir up other small particles. Also, fluid moving up to replace these falling particles will result in an upward convective current. Particle interactions increase greatly at very high concentrations. Suspension concentrations are normally analyzed in the range of 2 to 5 grams per liter (g/L) (Guy, 1969, p. 23) although some laboratories report acceptable analyses at concentrations of 10 g/L (M. D. Mays, 1981, U.S. Soil Conservation Service, written communication).

DISCUSSION

In the standard pipet procedure, the withdrawal pipet is inserted a fixed depth for each subsample withdrawal (fig. 1). The pipet is withdrawn to dispense the subsample into a container, then a small amount of distilled water is used to flush the pipet. The autopipet does not use the same withdrawal scheme as the standard pipet procedure. In the autopipet procedure, the withdrawal tip is set at a fixed elevation in the cylinder, and is not changed or disturbed during the analysis. subsamples are withdrawn by siphoning. A presample is withdrawn before each subsample to flush the line of material deposited from the suspension remaining in the line from the previous sampling withdrawal and to equilibrate the withdrawal line for the subsample. The standard procedure, then, uses two or three preselected withdrawal depths during a complete analysis; while the autopipet withdraws each of the 12 subsamples from a progressively shallower depth. The standard pipet method can be said to use a two or three-depth withdrawal scheme while the autopipet uses a 12-depth withdrawal scheme.

Each scheme has its advantages and disadvantages. The fixed-elevation, 12-depth withdrawal scheme used with the autopipet minimizes disturbances to the suspension in the cylinder which are a potential hazard of the standard procedure. On the other hand, the withdrawal depth is more difficult to predict and control. Each withdrawal depth is based on average cylinder and test-tube dimensions, and average subsample volumes. The siphoning scheme is much easier to automate than the standard mechanical scheme. However, the standard pipet scheme collects a very accurate sample volume from an accurately set depth. The autopipet's sample volume is quite consistent; but unless the optical level switch is carefully adjusted, the absolute volume of each subsample may not be sufficiently accurate. An incorrect subsample volume means that all following subsamples will be collected at the wrong depth for the calculated particle diameters. The autopipet procedure has twice as many withdrawals (presample flush and sample) as the conventional procedure, and is susceptible



In the standard pipet method, the pipet is lowered to a given depth for withdrawal of the subsample. A 15-cm depth is preferred for initial withdrawals (Guy, 1969, p. 24), 10-cm depth for intermediate withdrawals, and 5-cm depth (even 3 cm) for final withdrawals. The autopipet, on the other hand withdraws each subsample from a shallower depth than the previous one because the tip elevation remains fixed. Figure 1.--Comparison of a standard pipet and autopipet withdrawal schemes.

to cumulative error. Careful control must be maintained, therefore, of all volume-related components.

Measurements of the internal diameter of a hundred test tubes revealed that about half were within the required narrow range and were usable. The tube lengths, however, were variable. The autopipet, therefore, supports the test tubes on a fixed base during the filling operation to eliminate errors caused by variability in test tube length. A go/no-go gage is supplied with the autopipet for testing the inside diameter of replacement tubes. The internal diameter of the suspension cylinder and pipet tubing also must be within narrow limits.

The initial sampling depth is set at 17 cm after the initial-concentration sample is withdrawn. This depth gives sufficient volume for the 12 samples. The 17-cm initial sampling depth ensures that the final sample will be withdrawn from near the surface. Thus, the analysis may be completed within a reasonable time.

The volume of each withdrawal is assumed to be removed from above and below the tip of the siphon tube in equal portions. In other words, each subsample volume is assumed to be withdrawn from a horizontal slice centered at the tip of the siphon tube. The nth subsample will be withdrawn from a depth of

$$H_{n} = h_{0} - (n - \frac{1}{2})\overline{\Delta h} \qquad , \qquad (3)$$

where h_0 (cm) is the initial depth (17.0 cm); n is the withdrawal (subsample) number; and where $\overline{\Delta h}$ (cm) is the average change in water-surface elevation per subsample withdrawal, which is equivalent to the average withdrawal volume divided by the average wetted-surface area of the cylinder.

With all of the volume-related items held to close tolerances, it is possible to write a simplified fall-time equation for 30°C:

$$t_{30} = 8876 \text{ H}_{n}/\text{d}^{2}$$
 , (4)

where H_n is the fall distance (cm) for the <u>n</u>th withdrawal (from Equation 3), and particle diameter d is in μm . This equation computes the time at which the withdrawal is half completed. The average siphoning time is

about ten seconds, so five seconds must be subtracted from the computed withdrawal time to obtain the time for beginning the withdrawal. The use of Equation 4 is shown in more detail in Appendix VII. It is the basis of calculations for autopipet withdrawal times.

SUMMARY

Both the standard pipet procedure and the autopipet utilize the same basic fall velocity principles to determine particle-size distributions. The autopipet incorporates several modifications which simplify the hydraulic and electrical design. The primary modification is the use of a fixed-elevation, 12-depth sampling scheme instead of mechanically lowering the pipet to a predetermined depth. Other concessions are the use of an optical water-level sensor to stop the flow of the siphon when the correct volume is in the test tube, and the use of a presample to flush the siphon line of sediment suspension prior to collecting the "keeper" sample.

Careful attention to laboratory technique should give results similar to those of the standard pipet procedure.

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APPENDIX I - PRECAUTIONS, DESCRIPTION, AND INSTALLATION

PRECAUTIONS

Operator safety should be the primary concern. Certain precautions which may seem obvious should nonetheless be stated explicitly.

- 1. Only grounded power outlets equipped with ground-fault detectors should be used.
- 2. Unplug the power cord:
 - (a) when working on the equipment,
 - (b) when water is spilled on or about the instrument or on the floor.
- 3. Secure loose clothing when working in the vicinity of moving machinery.
- 4. Operate water-bath heater only when it is immersed.
- 5. Do not test live circuits with an ohmmeter, because to do so will destroy the meter. Also, do not test integrated circuits with an ohmmeter, because the battery inside the meter may destroy them.

PHYSICAL DESCRIPTION

The minimum configuration of the autopipet consists of two parts: a control console and a sample stand. A temperature-controlled water bath is optional, but is strongly recommended unless a temperature-controlled room is available. Each console can control from one to four sample stands without modification. Each sample stand can withdraw samples from three pipet cylinders simultaneously.

Control console

The autopipet is controlled from the console depicted in fig. 2. The power switch in the lower left corner connects the 120-volt ac power and the internal batteries when turned on. An ac-power indicating lamp is located above the power switch. The "Heater Current" meter shows the electrical current delivered to the heater in the water bath. The meter in the upper left shows voltage of the 28-volt dc bus when the "Test"

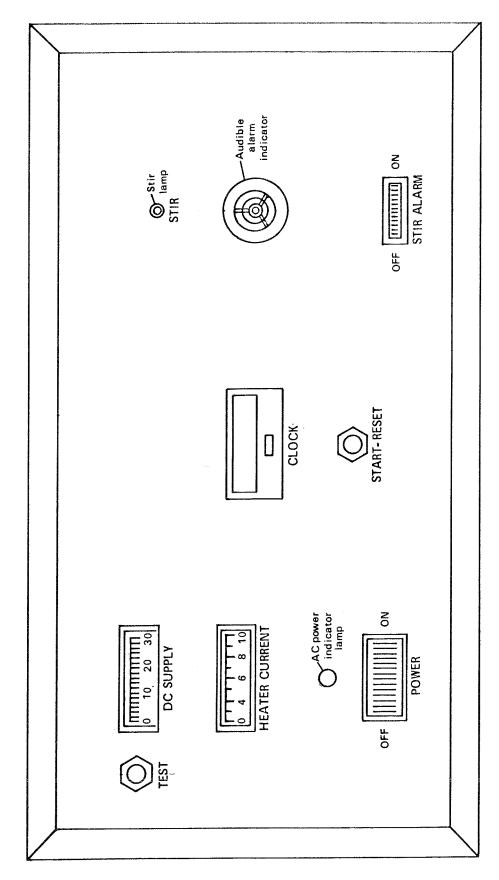


Figure 2.--Drawing of front of control console showing control switches, meters, clock, indicator lamps. and audible alarm indicator.

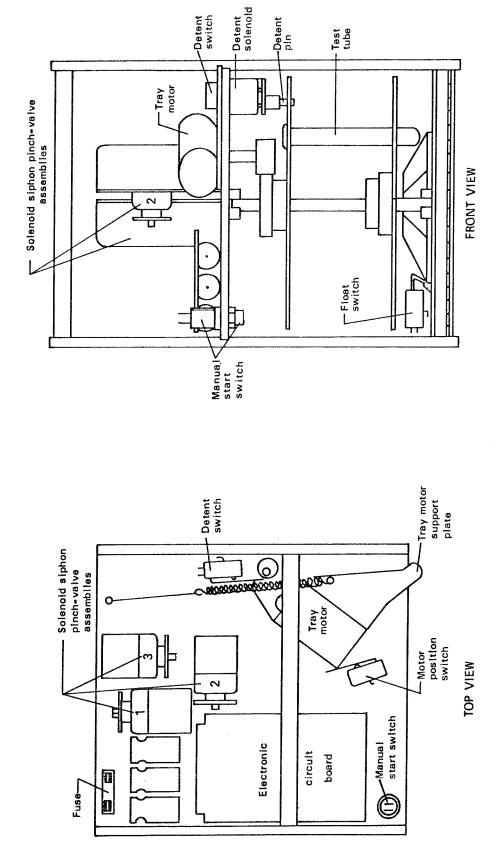
button is pushed. The clock counter in the center indicates the time, in seconds, following the pushing of the "Start-Reset" switch. The "Stir Alarm" switch activates the audible and visual indicators signaling when to mix the suspensions in the siphon cylinders for each sample stand.

The rear of the console has connectors for power, water-bath heater, heater sensor, stirring motor (water bath), and sample stand cables. Two circuit breakers are also at the rear panel, a 10-amp breaker for the heater and 1-amp breaker for the power supply.

Sample stand

The sample stand (fig. 3) is a roughly cubical box containing all of the mechanical actuators necessary to collect subsamples from the siphon cylinders. The sloping front of the box stops just above the internal deck. A slot with a large brass tab is at the top of the vertical front face. The tab is part of the motor support plate: swinging the tab to the left pivots the motor drive hub away from the test-tube tray, and swinging the tab to the right allows the motor to advance the tray. The tray itself is visible when the vertical front face is lowered, drawbridge fashion. The tray is removable when the motor drive hub has been moved away from the tray. With the front panel down, the manual start switch can be seen in the upper left front of the cavity. The float switch and well should be visible in the left rear. Also, the detent solenoid and detent pin can be seen attached to the bottom of the deck on the upper middle right. The cable from the control console attaches to the connector beneath the left rear corner of the deck.

Raising the top hinged panel shows the electronics circuit board, the three siphon-valve assemblies and tray-advance motor. The motor-position switch and motor are hidden beneath the sloping front. The detent switch is visible beside the large spring. The water-level sensors are below the deck beneath the siphon-valve assemblies. Note the location numbers on the assemblies: these determine the order of placement of siphon tubing from the cylinders. The 2-amp fuse is also visible in the left rear corner.



Numbers Figure 3.--Sample stand parts location diagram. The top and front covers have been removed. on solenoids refer to the order of pipet cylinder attachment.

Water bath

Figure 4 shows the water bath including the heating element, stirring motor, and horizontal baffle. Not shown is a vertical baffle separating the heating element and stirring motor from the main body of water. The stirring motor lifts the water from the horizontal baffle up across the heating element, and over the vertical baffle out to the tank.

Dimensions (approx)

	<u>Width</u>		<u>Height</u>		<u>Depth</u>	Width		Height		<u>Depth</u>	
Control console	0.43	x	0.24	х	0.28 m	17	x	9.5	x	11 in	
Sample stand	0.33	x	0.37	x	0.30 m	13	x	14.5	x	12 in	
Water bath	1.8	x	0.66	х	0.41 m	72	x	26	х	16 in	

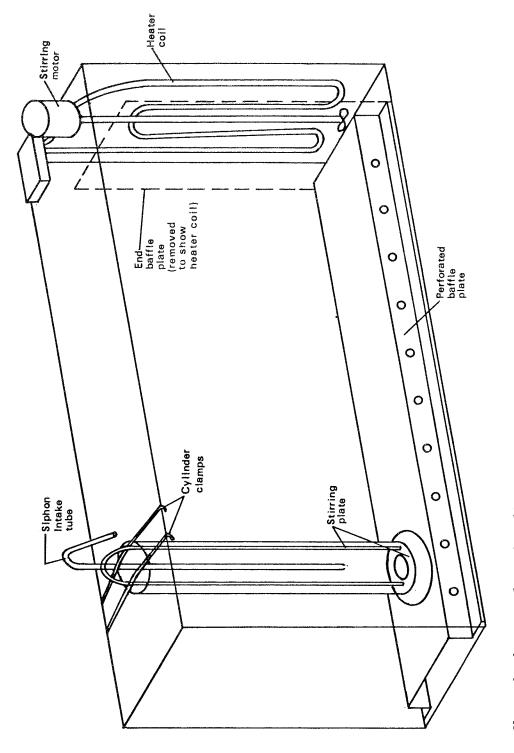
Power requirements

A 15-amp, 120-Vac (60 Hz) grounded three-wire power receptacle will satisfy the power requirements of this instrument. The actual power used will vary depending on the heater demand, the state of charge of the battery, and on whether any of the sample stands is operating. The heater is rated at 1,000 watts. Each sample stand uses about 80 watts, but no more than two ever operate simultaneously.

INSTALLATION

The equipment should be assembled on a stable, sturdy table or bench capable of supporting 300 pounds (140 kg) of equipment. The table top should be at least 30-in wide by 72-in long (0.76 m \times 1.8 m) and should have a comfortable working height. The table should be accessible from three sides.

The equipment may be arranged in any convenient fashion. One possible arrangement is shown in fig. 5. There must be space behind the water bath for the operator(s) to mix the suspensions in the cylinders at the beginning. If the optional water bath is not used, the cylinders must be



The support stand and end baffle plate are not shown. Figure 4.--Water-bath parts-location diagram.

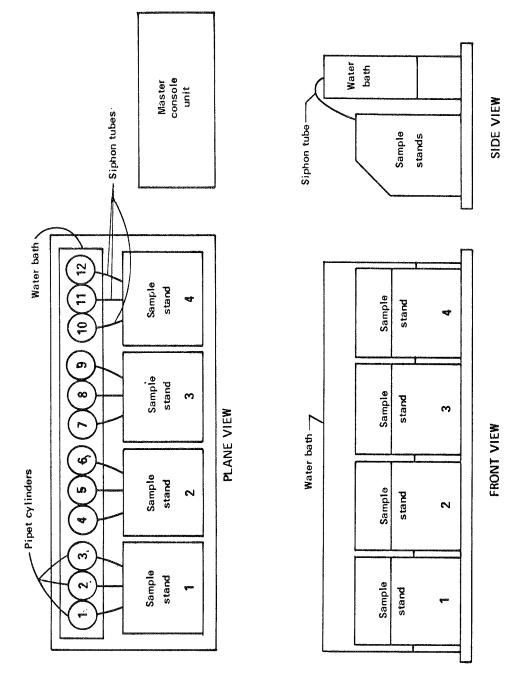


Figure 5.--Possible arrangement of autopipet apparatus.

placed on a 25.4-cm stand to produce the proper siphon rate. Each sample stand is placed to minimize the length of siphon tubing from the cylinders in the water bath to the siphon valves within the sample stand. The sample stands are placed so the test-tube trays may be installed and retrieved. The console control is not shown on the figure, but it should be placed so that all cords and cables reach their proper connectors. The operator should be able to read the clock counter and see the stirring indicator. The operator must also be able to reach the clock/alarm start switch at the beginning of the automatic phase of the analysis.

The sample stands and the legs of the water bath must rest on the same surface to insure the proper siphoning rate. This rate is controlled by the difference in elevation between the water level in the cylinder and the tip of the nozzle below the siphon valve in the sample stand. Because the water level drops during an analysis, the siphon rate will decrease throughout the analysis.

APPENDIX II - INITIAL STARTUP AND TESTS

Fill the water bath with tap water and check for leaks. If any leaks are found, drain the bath and, when dry, seal with clear silicone sealer. Refill the bath to a depth of about eight inches (0.2 m). Place the horizontal baffle (see fig. 4) in the bottom of the bath. The closely spaced holes should be nearest to the heater end. Mount the stirring motor and check its operation. Attach the temperature sensor and heater cables and check their operation. Fill the bath until the heating element is covered. CAUTION: BE SURE SUFFICIENT WATER IS IN THE BATH BEFORE OPERATION. heater is controlled by the power switch on the front panel, the 10-amp circuit breaker on the rear of the console, and by the two potentiometers (coarse and fine) of the temperature controller. The controller is a proportional type. Heater power may be adjusted continuously and smoothly. The coarse adjustment is located on the controller inside the console and the fine adjustment is next to the temperature-sensor connector on the rear panel. The coarse adjustment will be set before shipment. The fine adjustment can be made by measuring the bath with an accurate $(\pm 0.1^{\circ}\text{C})$

thermometer, then turning the potentiometer screw to adjust the power level as desired. The heater-current meter on the front panel of the console will indicate the heater current.

The sample cylinders may be placed in the water bath after they are filled with water. Some water may have to be removed from the tank to prevent displacing bath water into the cylinders. The cylinders are prevented from tipping or floating by attaching the latex tubing clamps, two for each cylinder. The cylinder positions should be numbered to correspond to positions in the sample stands. Normal usage would require numbering from left to right when viewed from the sample-stand side of the bath. After adding the stirrer, pipet, and lid assembly to each cylinder, the siphon tubing may be fitted. The normal numbering within each sample stand is shown on fig. 6, but this is arbitrary. Whatever scheme is chosen should be used consistently through all of the sample stands. The samples from each cylinder collected in the outer row will be delivered to alternate test tubes as shown in the same figure. Note also that the sets of three alternate - the first withdrawal set of three is to flush the pipet and tubing. The second withdrawal set is the "keeper", or subsample. All flush sets may be discarded, but all subsample sets must be retained. It might be wise to paint the keeper sectors of the test-tube trays to help prevent discarding samples.

After the equipment is set in place and cables have been connected, several practice analyses should be performed to train personnel and verify equipment operation. A dry trial run would make good sense for an initial try. Clamping or pinching the siphon tubes is all that is required. Proper operation of the equipment can be verified by following the procedure in Appendix IV and checking times with the clock counter or a stopwatch. When satisfied, a wet run with tap water can be used to test operator dexterity and to check for leaks.

Field samples should be entrusted only to properly trained operators using thoroughly understood equipment.

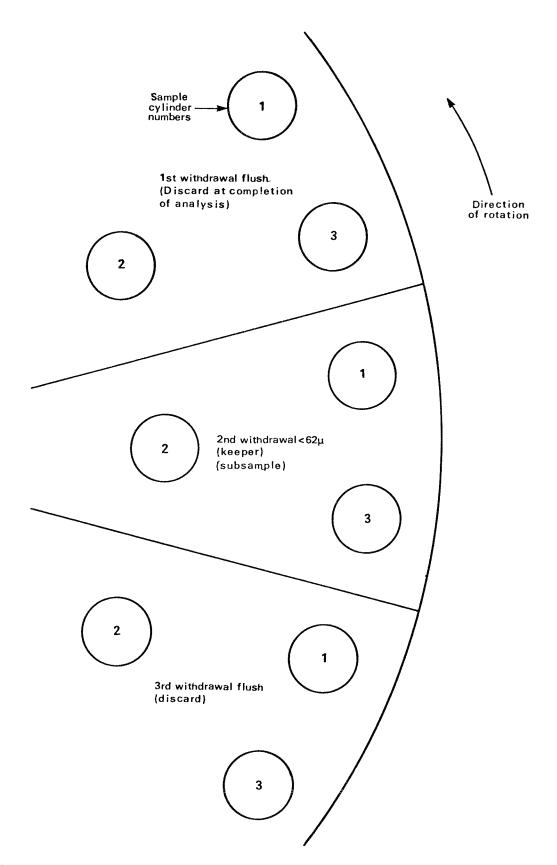


Figure 6.--Arrangement of test tubes in tray, viewed from above.

APPENDIX III - SAMPLE PREPARATION

PRE-OPERATION SAMPLE PREPARATION

A sample received from the field must undergo treatment before it can be analyzed. The preferred condition of a sample ready for analysis is:

- 1) free of organic matter (seeds, twigs, leaves),
- 2) low in dissolved solids,
- 3) free of coarse sediments; that is, all sediments coarser than 62 $\mu\text{m}\,,$
- 4) in the sediment concentration range from 2 to 5 g/L,
- 5) chemically and mechanically dispersed, and
- 6) at a uniform, stable temperature.

Each of these aspects of the preferred condition removes a source of measurement interference or adjusts the sample to optimum subsampling condition. Each aspect will be discussed along with the procedure.

1) Organic matter interferes with settling of inorganic sediments, introduces weighing errors, and could clog small passages and nozzles of the autopipet. Guy (1969, p. 26) gives the following procedure for removal of organic matter after decanting the clear supernatant water from the settled sample from the field:

"Add 5 mL of 6 percent solution of hydrogen peroxide for each gram of (dry) sample which is contained in 40 mL water. Stir thoroughly and cover. Large fragments of organic matter may be skimmed off at this state if it can be assumed that they are free of sediment particles. If oxidation is slow, or after it has slowed, the mixture is heated to 93°C and stirred occasionally. The addition of more of the hydrogen peroxide solution may be necessary to complete the oxidation. After the reaction has completely stopped, wash the sediment two or three times with distilled water."

L. L. McDowell (U.S. Agricultural Research Service, written communication, 1981) has found better results are obtained by adding 10 to 20 mL of 30 percent hydrogen peroxide solution and digesting overnight at room temperature. An additional 10 mL is added the next morning and the sample

is digested on a hot plate with occasional stirring to reduce foaming. McDowell also notes that a slightly acid (pH5) suspension is required for efficient oxidation of the organic matter. The soil or sediment in suspension must be non-calcareous. An alkaline suspension causes rapid, excessive decomposition of the hydrogen peroxide with a rapid generation of oxygen gas.

- 2) Low dissolved solids are desirable to minimize unwanted flocculation or dispersion by these salts. Decanting the clear water prior to the previous step should serve to remove sufficient dissolved solids. Decanting may be accomplished by careful pouring, by using a j-shaped suction tube, or by using a commercially available ceramic filter tube (also called a filter candle) to remove the supernatant water and salts.
- 3) Coarse sediment interferes with normal settling of fine particles, creating turbulent wakes which agitate the mixture and increase the fall velocity of small particles. The particles fall too rapidly to be analyzed accurately in a short pipet column. Also, coarse material deposits in the tubing and passages of the autopipet. Two procedures for removing coarse particles in current practice are the initial-break tube method and the wet-sieve method.

The initial-break tube method separates particles by fall diameter. An apparatus is shown in fig. 7. The tube should be from one to two inches in diameter (2.5 to 5.0 cm) and one to three feet long (30 to 90 cm). The tapered section at the bottom is not too critical; but an angle of 30 degrees or less with the vertical is preferred. The tube is similar to the bottom-withdrawal tube described in Rept. 7 (ICWR, 1943) and Rept. 10 (ICWR, 1953). The distance from the visual-accumulation tube clamp (ICWR, 1957) at the top and the pinch clamp at the bottom should be measured carefully. This distance (centimeters) divided by the fall velocity (centimeters per second) of a 62-µm particle falling in water of the same temperature (0.433 cm/sec at 30°C) gives the fall time for that particle between the clamps. The tube is filled with distilled water.

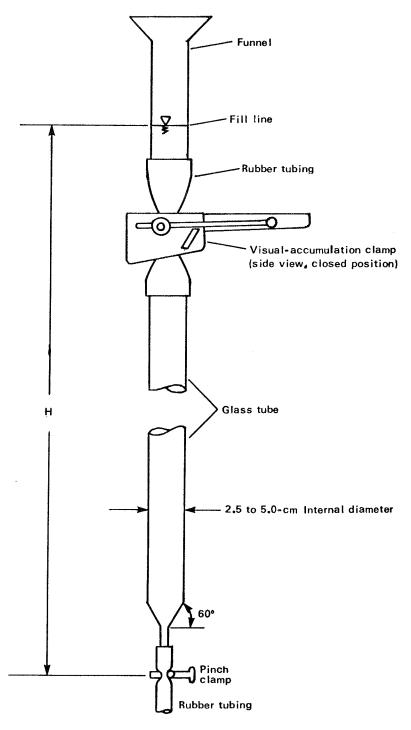


Figure 7.—Possible initial-break tube arrangement. The distance H divided by the fall velocity of a 62-micrometer particle computed from Equation 1 will give the initial-break withdrawal time for the specified temperature. Note that the distance from the fill line above the upper pinch clamp to that clamp should be small relative to the distance between the clamps.

Next the sample is mixed with the water in the tube above the upper clamp. The clamp is opened and a stopwatch is started. The material is allowed to settle for the computed fall time. The upper clamp is closed and the coarse fraction is immediately removed by opening the bottom pinch clamp. The remainder, constituting all of the fine particles for pipet analysis, is drained into a separate beaker by opening both clamps and flushing with distilled water.

The alternate method of initial separation is the wet-sieve method described by Guy (1969, p. 28): using a gentle jet of water to wash the finer particles through a 250-mesh (62 μ m) sieve. "The sieve is tilted, rotated, and tapped gently to facilitate the washing procedure." The material passing the screen is for pipet analysis and the retained coarse material is for separate analysis, usually by visual-accumulation tube.

- 4) The sediment concentration should be adjusted before analysis. Too high a concentration leads to too much interference between particles, while too low a concentration increases weighing errors. The optimum range is from 2 to 5 g/L. If the beaker containing the fine fraction is estimated to contain between 2 and 5 g of sediment, then all that remains is add dispersants, mix, and to dilute to one liter with distilled water. If insufficient material is present (< 2 g) the operator must decide if the increased weighing errors justify the analysis. Use of a smaller cylinder is not possible because the preset sample withdrawal times are computed for drawdown in the standard cylinder. If more than 5 g material is present, a Jones splitter may be used to reduce the mass of material to an acceptable amount.
- 5) Chemical and mechanical dispersion is necessary to break up floccules formed during storage and to minimize additional flocculation during the analysis. Also a standardized preparation method allows analytical results to be compared on the basis of particle-size differences without the influence of variable water-quality characteristics. The National handbook of recommended methods (work group 3 on sediment,

1978, p. 95) gives the following recipe for the dispersant:

"40.0 g of sodium metaphosphate and 8.0 g of sodium bicarbonate in distilled water diluted to 1.0 L. After adding the dispersing agent, (1) transfer the sample to a 250-mL shaker bottle, adding distilled water to bring the volume to 180 mL, and shake overnight in a horizontal reciprocating shaker, or (2) stir with a mechanical analysis stirrer (malt machine) for 2 to 5 minutes."

Ten mL of dispersant should be added per gram of sediment. The dispersant should be added before the 5-min mixing in a mechanical mixer, then the suspension should be rinsed into the pipet cylinder and diluted to one liter.

6) The temperature of the suspension should be allowed to become uniform and stable. Uniformity of temperature is necessary to minimize density-induced convection currents. Temperature stability is necessary to control fluid viscosity and maintain temperature uniformity. Generally, a few hours in the temperature-controlled water bath will suffice to stabilize the water temperature in a cylinder. Pipet analyses may be performed in a constant-temperature room. Prior experience will have to dictate the time necessary for temperature stability in air in such a case.

APPENDIX IV - OPERATIONAL PROCEDURE

START PROCEDURE

Once the suspension has been prepared according to procedures given in Appendix III, "Sample Preparation", the analysis may proceed. The starting procedure will be described for three simultaneous analyses with one sample stand. If fewer analyses are to be performed, make appropriate corrections. If multiple sample stands are to be used, appropriate comments are included.

1) Empty the treated sample into the cylinder and add sufficient distilled water to bring the volume to a liter as indicated by the top line on the cylinder. Place one mixing plunger and one siphon assembly

into each pipet cylinder. The desired pattern for connecting siphon assemblies to the sample stand is indicated in figs. 3 and 5. Check to see that the siphon tube does not interfere with the movement of the plunger. Remove the test tube tray. Place a clean 150-mL beaker beneath the desired nozzle inside the sample stand. Stir the mixture steadily for one minute, then push the solenoid valve open while continuing to stir the suspension. Stir vigorously to prime the siphon. the solenoid is pushed during a lowering of the plunger, siphon action should start. After siphon action starts, continue siphoning until the water surface is at the second line on the cylinder, about 130 mL. subsample is to determine the initial concentration of the mixture. Return the test tube tray to the sample stand. Finally, adjust the siphon tube so that the mark is at water level, 17.0 cm (6.69 in) above the tip. Repeat the above procedure for all other cylinders being analyzed, including those for other sample stands, before proceeding. Turn on the Stir Alarm switch on the lower right of the console front panel (fig. 2) if more than one sample stand will be used.

2) When all is ready, mix the sample with the plunger for one minute. Push the plunger to the bottom, cease stirring, and immediately push the start button on the control console (see fig. 2). CAUTION! This button is pushed only once (even for 12 simultaneous analyses) to initiate the time process. Do <u>not</u> push the start button for each cylinder <u>nor</u> for each sample stand because each push resets the basic time counter circuit. With practice, an operator can mix two cylinders at once. Two persons are required to mix all three cylinders.

One minute after the start button is pushed, the red lamp on the front (upper right) of the console lights. This is the signal to start mixing the cylinder(s) for the next sample stand. The lamp glows for the one-minute mixing period, then as it turns off an audible "beep" is sounded. Stop mixing. Rest until the red lamp lights again (about one minute) while preparing to mix the cylinder(s) in the next sample stand. Repeat this process until the cylinder(s) in the last sample stand have been mixed. The Stir Alarm switch may be turned off after the final mixing. Turning off the switch is not critical, however.

Each sample stand contains all necessary timers and controls for with-drawing a subsample from each of three sample cylinders after receiving a start pulse from the console. That is, once the start pulse is received, the sample stand withdraws a sample from the three cylinders and then advances the test-tube tray. A normal cycle consists of a start pulse from the control console which initiates a preliminary ("flush") cycle followed 23 seconds later by another start pulse which initiates a subsampling ("keeper") cycle.

The autopipet can be left unattended until the subsampling is complete, in about $2\frac{1}{2}$ hours. The machine is set to collect subsamples for 62, 31, 16, 8, 4, and 2 μm sizes without further operator attention. The operator (and assistant) are now free to prepare succeeding samples, weigh prior samples, or do any other necessary activities.

APPENDIX V - CIRCUIT DESCRIPTIONS

Control console

The control console contains all power supplies and controls, and necessary circuitry to initiate withdrawal cycles by each sample stand at the proper time. A block diagram is given in fig. 8, which shows the six fundamental subcircuits:

- 1) power supply,
- 2) time base and clock counter,
- 3) stirring timer with indicators,
- 4) withdrawal-time decoder,
- 5) end-of-analysis (EOA) decoder, and
- 6) water-bath heater control.

Power supply

Three dc voltages (5, 12, and 28 volts) are required by the circuits in the control console. The 28-volt supply is used by the clock counter and the audible indicator. The programable logic array (PLA) and its

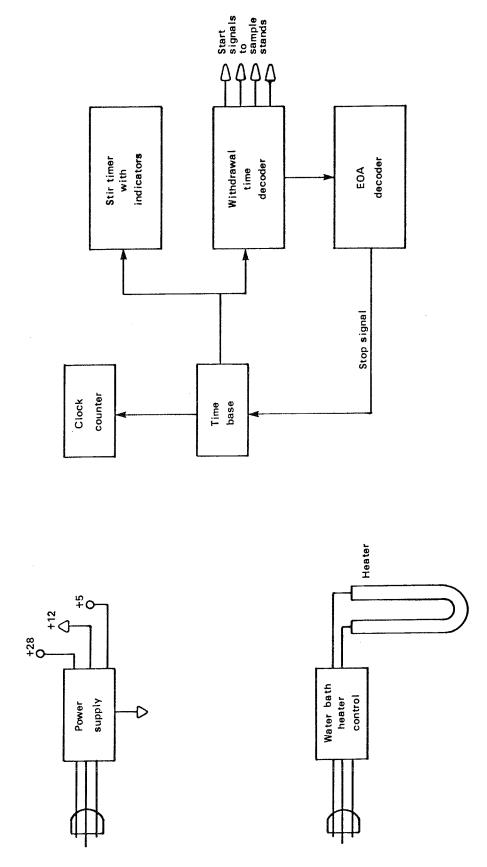


Figure 8.--Block diagram of control console unit. The EOA (end-of-analysis) decoder turns off the time base after all samples have been collected to avoid a repeating of the entire sequence.

buffers require 5 volts and all other logic elements require 12 volts. Both 5 volts and 12 volts are derived from the 28-volt supply. Because a power interruption would disrupt analyses in progress, the 28-volt supply is backed by a 24-volt, 2.5 ampere-hour, sealed lead-acid battery.

The power supply circuits (fig. 9) are conventional in design. The 120-Vac power is passed through the 1-amp circuit breaker to the step-down transformer, T1, which converts it to 35-Vac. Capacitors C110 and C111 provide some filtering before rectifying the ac to dc in the diode bridge CR109-112. Additional filtering is provided by the 3-ohm, 5-watt resistor (R121) and the 1500-microfarad (μ F) capacitor. A 12-volt regulator (IC32), biased by resistors R110 and R120, regulates the output at 28 volts. Diode CR113 protects the regulator from the battery when the 120-Vac power is OFF and the power switch is ON. Resistor R118 limits the charging current to the battery, and diode CR108 conducts battery current when the 28-volt bus falls below about 27 volts and the power switch is ON. Capacitor C104 (0.1 μ F) limits current surges. Resistors R101 and R102 limit current flow to their respective regulators. Capacitors C101, 102, and 103 smooth the regulator outputs.

Another regulated supply, the 24-volt source for the clock counter, consists of R117, C108, CR106, and Q2. If the 120-Vac power fails, battery power is supplied through CR105. In this event, the counter will continue counting but the display will go off. The display will show the correct reading when 120-Vac power is restored.

Except for the water-bath heater control, the remainder of the subcircuit parts are on the wire-wrap board (fig. 10) and are shown schematically in fig. 11.

Time base and clock counter

IC30, the time base oscillator, has a frequency of 4,194,304 Hz. IC29 is a 21-stage binary counter which reduces the oscillator frequency to 2 Hz. Flip-flop IC28A divides the 2-Hz signal to 1 Hz if reset pin 4 is low. The 1-Hz signal is counted by the front-panel clock counter which

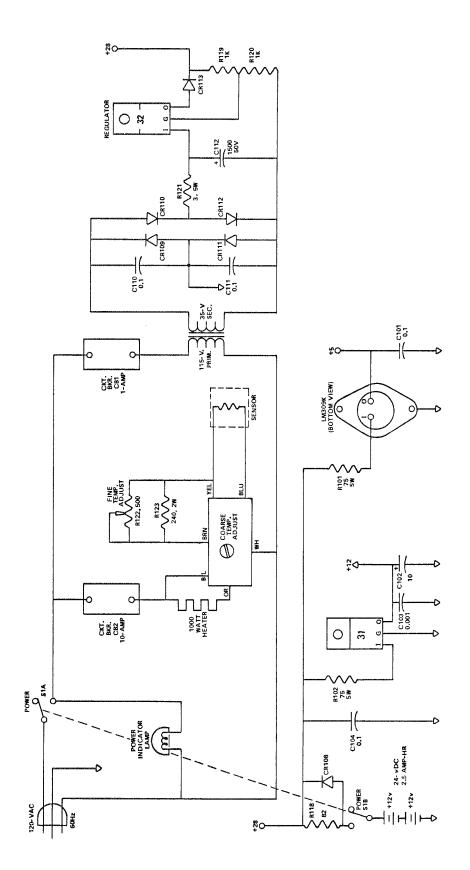
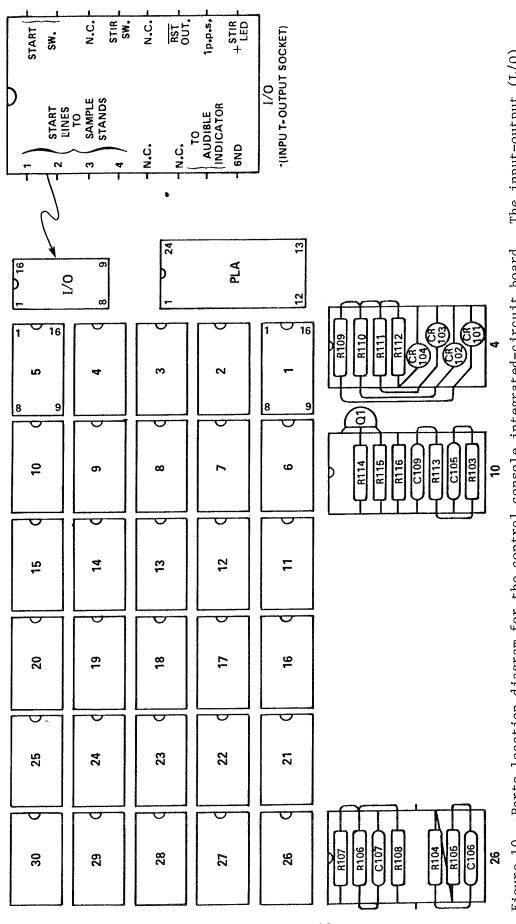
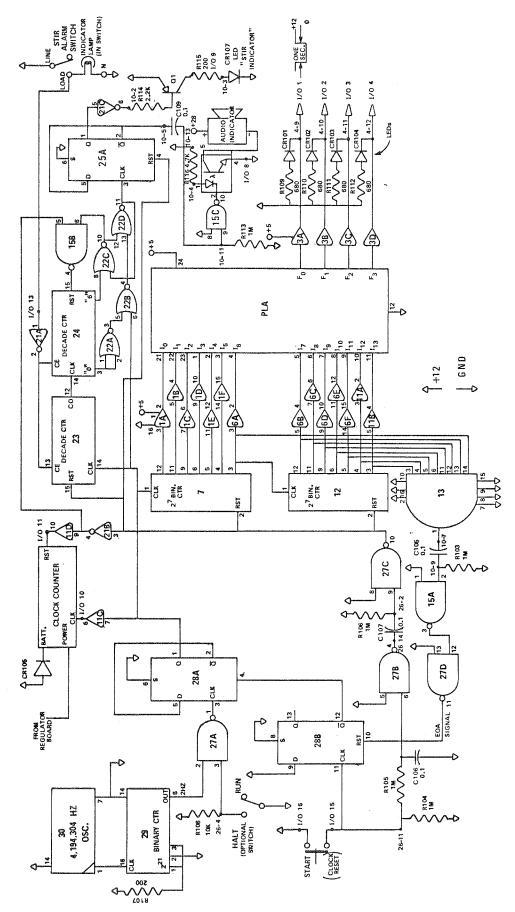


Figure 9.--Control console power supply schematic circuit diagram. All resistance values are given in ohms and all capacitances in microfarads.



Ground and power pins are different for IC 29 and the PLA. Fig. 11 gives pin numbers for The 14-pin ICs are inserted so that their ground pin (7) matches the grounded receptacle 16 pins except for the 24-pin PLA socket. The board contains a mixture of 14 and 16-pin integrated circuits, Figure 10.--Parts location diagram for the control console integrated-circuit board. The input-output (L/O) socket and three component carriers (for positions 4, 10, and 26) are shown enlarged. All sockets have not counting the PLA. pin (8) of the board. these ICs.



All resistance values are given in ohms and all capacitances are given in microfarads. Figure 11. -- Control console schematic circuit diagram.

displays the elapsed time (seconds) since reset. The 1-Hz signal is also used by the stirring timer (IC 23, IC24) and the withdrawal-time decoder (IC7, IC12, and IC13).

Stirring timer with indicators

Decade counters IC23 and IC24 are enabled by the stir alarm switch on the front panel of the control console. After they have been enabled, IC23 divides the 1-Hz time base by 10 and IC24 divides it further by 6. The four gates of IC22 assure an exact divide-by-60, otherwise the first pulse would be at 60 seconds and all other pulses would be 100 seconds apart. The output of IC22D is transmitted to the clockline of IC25A. This signal causes flip-flop IC25A to alternately activate the light-emitting diode (LED) stir indicator through transistor Ql and the audible indicator through gate IC15C. C109 and R113 differentiate the positive-going input to IC15C to produce a short pulse so only a short "beep" is heard.

Withdrawal-time decoder

The 1-Hz signal at the "Q" output of IC28A is accumulated in counters IC7 and IC12, which are connected to form a 14-stage binary counter. All outputs of both counters are connected to the PLA through a 12-volt to 5-volt buffer (IC1, IC6, and IC11). When the PLA senses a match between the binary time count from IC7 and 12 and the programmed code, it drops the appropriate output voltage, which is buffered from 5 volts back to 12 volts by IC3 and passed on to the appropriate sample stand as a start pulse. Whenever an output line is grounded, an LED indicator on the IC board lights. These indicators serve only as diagnostic aids.

End-of-analysis decoder

When all withdrawals have been completed, the decoder halts the 1-Hz time base. Withdrawal completion is sensed by ICl3, an 8-input AND gate. When the voltage at all eight input lines is high, ICl3's output also

goes high. Capacitor C105 and resistor R103 differentiate the rising voltage which gate IC15A converts to a narrow pulse. This pulse, in turn, is inverted by gate IC27D to reset flip-flop IC28B. The output of IC28B resets IC28A which halts the 1-Hz time base. With the time base halted, there is no possibility of recycling through the withdrawal schedule inadvertently.

Water bath heater control

A commercial heater control with a coarse and fine adjustment is used to adjust the temperature of the water bath (fig. 9). The standard PLA has been coded with withdrawal times calculated for 30° Celsius. The bath temperature should be adjusted to within $\frac{1}{2}$ ° of this value.

Sample stand

When the start pulse is received, all three siphon-valve solenoids activate (fig. 3). Each solenoid moves a flat spring to open the siphon tube and to activate the snap-action switch. Moving the flat spring allows the suspension to flow from the cylinder through the siphon tubing, through the plastic nozzle below the deck, and into the test tube centered below the nozzle. When the meniscus passes the beam of the infra-red lamp (IRLED) below the upper arm of the test tube, the beam is diverted away from the sensor, the solenoid relaxes and the flat spring closes the siphon tube. Each siphon is closed individually when its water-level sensor is activated. After all three siphon-valve solenoids have relaxed, the detent solenoid momentarily moves the detent pin out of a notch on the rim of the test-tube tray while the tray advance motor rotates the tray. When the detent pin drops into the next notch, the motor stops and the withdrawal cycle is complete.

The circuit schematic for the sample stand is shown on fig. 12. The circuit may be divided into the following subcircuits for better understanding:

- 1) power supply,
- 2) start circuit,

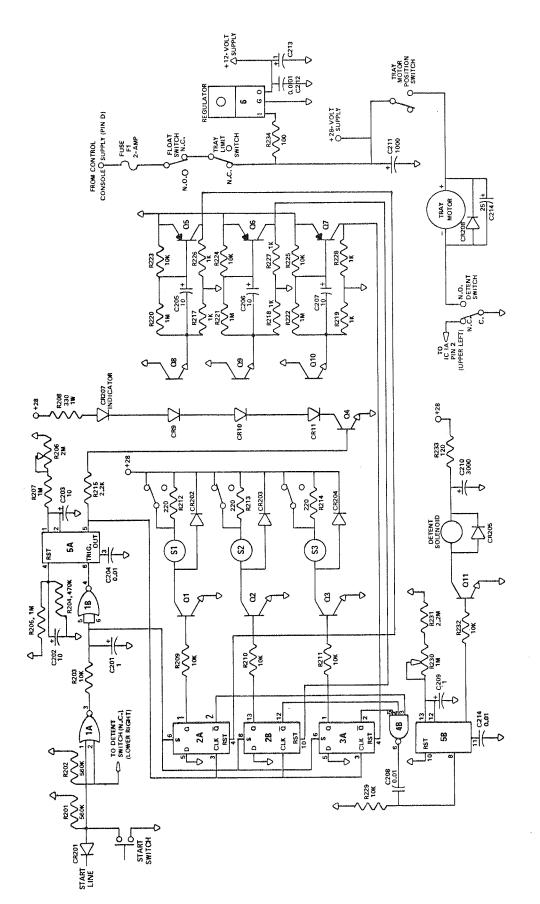


Figure 12. -- Sample stand schematic circuit diagram. All resistances are given in ohms and all capacitances in microfarads.

- 3) LED and siphon solenoid timer,
- 4) individual siphon solenoid control, consisting of
 - a) flip-flop solenoid driver
 - b) water-level sensor
 - c) flip-flop reset,
- 5) detent solenoid timer and driver, and
- 6) test-tube tray motor control.

Parts on the electrical circuit board may be located using fig. 13.

Sample stand power supply

This supply receives its power from the +28-volt console supply. Power may be interrupted by the 2-amp fuse, the float switch, and the tray-table limit switch. The float switch prevents operation if there is a spill or overflow. The limit switch shuts off the machine after the last withdrawal. Capacitor C211 provides continuation of power for short-term power interruptions or dips in the +28-volt supply. R234 dissipates power as the regulator drops the voltage from 24 volts to the 12 volts required by the integrated circuits.

Start circuit

The start pulse is a one-second pulse generated by the PLA in the control console. To initiate a withdrawal cycle, the console drops the clockline voltage to zero volts for a second or two then returns to 12 volts. The one-second pulse width is not critical. Either a start pulse or pushing the start switch (located on front left beneath deck) will cause the output of gate 1A to go high if the ground-enable detent switch is also closed to ground. The pulse from IClA is smoothed by R2O3 and C2O1. The pulse sets flip-flops 2A, 2B, and 3A; and after being inverted by IClB, the pulse triggers timer IC5A. The flip-flops switch transistors Q1, Q2, and Q3. Each transistor activates one of the three siphon-tube solenoids (S1, S2, or S3).

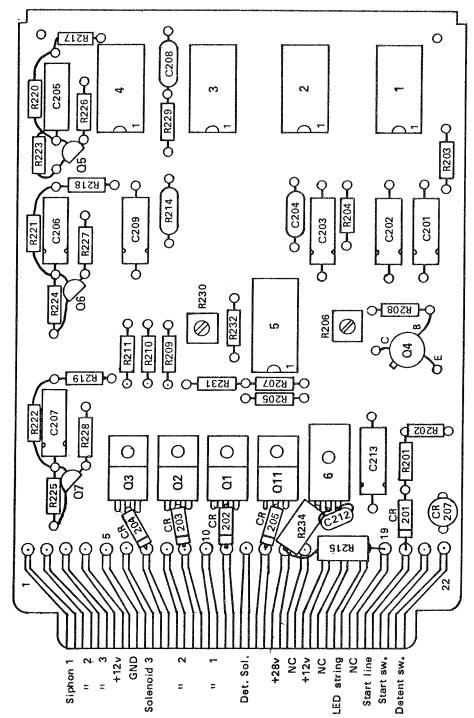


Figure 13. -- Parts location diagram for sample stand electrical circuit board.

LED and siphon solenoid timer

IC5 is a dual timer. The first half, IC5A, when triggered by a sharply dropping voltage from gate 1B, generates a positive pulse with a duration determined by capacitor C203, resistor R207, and potentiometer R206. R206 is used to adjust the pulse duration to 14 seconds. pulse turns on transistor Q4 which feeds power to light-emitting diodes (LEDs) CR9, 10, 11, and CR207. Diodes, termed IRLEDs, CR9, 10, and 11 produce infra-red radiation which is invisible to the eye. CR207 produces visible light as an indicator - if it lights, the IRLEDs are receiving power. The IRLEDs are mounted in brackets below the top deck where the beams will be intercepted by each meniscus of the sediment suspensions as the test tubes are filled. The beams are deflected away from their respective phototransistors (Q8, 9, or 10) by the passing menisci. About 14 seconds after the start pulse, the output of the timer returns to zero volts. Gate IC1C goes high which causes all three flip-flops (IC2A, IC2B, IC3A) to deactivate the three siphon-tube solenoids. This timed deactivation overrides all water-level sensors and prevents excessive spills should one or more sensors fail.

Individual siphon solenoid control

There are three identical siphon solenoid circuits, so only the first will be described. The start pulse from gate 1A sets flip-flop 2A, causing its "Q" output to go high and to turn on transistor Q1 and thus solenoid S1. This solenoid is powered from the 28-volt supply (A+). Initially, the solenoid is powered by the full 28 volts through switch SW2 which is opened by movement of the solenoid armature. After the switch opens, the solenoid is powered through resistor R212. SW2 serves to decrease the power drawn from the A+ supply. As the meniscus rises past the IR beam from CR9, the beam is either deflected away from phototransistor Q8 or is absorbed. When illuminated by the beam of CR9 through the test tube, Q8 will conduct through R217. When the meniscus diverts or absorbs the beam, the voltage across R217 will drop to zero. This voltage

transition is differentiated by C205, and momentarily causes Q5 to conduct current through R226. The voltage which now appears across R226 because of this current is transmitted to the reset (R) input of flip-flop IC2A. Resetting a flip-flop causes its "Q" output to go low (drop to zero volts) and its " \overline{Q} " (read not-Q) output to go high. When the "Q" output goes low, siphon solenoid S1 is deactivated.

Detent solenoid timer and driver

When all three flip-flops have been reset, whether individually through the optical circuit or simultaneously at the end of the timer interval, their \overline{Q} outputs are high. This condition is sensed by the triple-input gate IC4B which goes low, causing a negative pulse to appear at timer 5B. Capacitor C209, resistor R231, and potentiometer R230 are used to adjust the output pulse to between three and five seconds. The output from timer 5B turns on transistor Q8 which activates the detent solenoid on the top deck. Resistor R233 limits the current drain from the 28-volt supply while extra short-term current is provided by capacitor C210.

Test-tube tray motor control

If the tray is in position, the motor position switch will be closed enabling the detent switch to control the tray motor. When the detent solenoid is activated, the detent pin moves to unlock the test-tube tray and to toggle the detent switch. When the detent switch is closed, the tray motor rotates the tray until the detent pin falls into the next index notch. The motor stops the detent solenoid timer (5B) and activates the solenoid long enough for the motor to move the perimeter of the tray so that the detent pin will clear the most recently occupied index notch.

APPENDIX VI - TROUBLESHOOTING GUIDE

Theory

There are two fundamental approaches to troubleshooting electronic equipment: frontwards and backwards. In the frontwards approach, testing is begun at the beginning of the schematic, usually the power supply, and proceeds through each section of the circuit logically until the improperly performing section is located. The same procedure is used within this section to locate the particular part or parts which are not performing as necessary. The backwards approach to troubleshooting begins at the end or final portion of the circuit and proceeds back through the system until the functioning portion of the instrument is located. The most recent nonfunctioning section must therefore be at fault. Again, testing may now proceed to locate the bad part. A trained technician will use the approach appropriate to the problem. For instance, the backwards approach might isolate a problem more quickly if the instrument appears to function properly except at the end of its cycle.

Both approaches to troubleshooting stress a logical approach. Eliminating functioning subcircuits as suspects requires trained, thoughtful observation and deductive logic. The descriptions of operation and circuit functions given in this manual provide the instrument technician or operator a basis for observing out-of-tolerance behavior of the instrument.

One further point should be mentioned. If an instrument is not functioning or is operating improperly, the problem may be caused by parts that have failed altogether, that are working improperly, or that are out of adjustment. Obviously, a failed part should be replaced. However, the technician must decide the fate of an improperly working part: adjustment, repair, or replacement.

General problem isolation

Symptom	Possible problem circuits
"Nothing works"	Power supplies, time base.
No clock counter display	Power supplies, including clock counter's supply.
Clock counter doesn't count	Clock reset (start), time base.
Stir alarm doesn't work	Time base, clock reset, stir-time counter.
Sample stand won't start	Control console: time base, clock reset, withdrawal time decoder, cable including connectors.
	Sample stand: power supply, including fuse, input and siphon solenoid timer, limit switch, float switch.
Siphon fails to shut off	Siphon cut-off, siphon solenoid timer, pinch-valve spring (adjustment).
Tray does not advance	Detent solenoid timer, detent switch, motor-position switch, tray motor.
Clock counter display goes out when more than one sample stand is connected	Check for inadequate power supply capacity.
Test tubes systematically do not fill to proper level	Siphon solenoid timer, check that base of cylinders is 25.4 cm (10 inches) above surface on which sample stand rests.
Tubes fill erratically	See "siphon fails to shut off" above. Check for uneven test tube optical properties—hold clean tube up to a light and rotate, discard if uneven— ness is noted. Check for misalignment or looseness of nozzles—they should discharge against side of test tube away from IR beam. Check for proper IR beam alignment—the very narrow beam should be aimed directly at the phototransistor—note that IRLED and phototransistor leads may be fragile.

Power supplies

There are seven separate power supplies in the autopipet: 120Vac for the water heater, 120Vac for the 28Vdc bus, 28Vdc for charging the 24V batteries, 28Vdc to 24Vdc for the clock counter, 28Vdc to 12Vdc for the IC board, 28Vdc to 5Vdc for the withdrawal-time decoder, and finally 28Vdc to 12Vdc in each sample stand. Each of these will be examined in some detail. Refer to figs. 8 through 13 for parts location and circuit schematics. Further circuit descriptions may be found in Appendix V.

The pilot light indicates that 120Vac is available at least as far as the power switch. The two circuit breakers are powered independently from the switch: the 10-amp circuit breaker supplies power only to the heater circuit and the 1-amp breaker supplies current to all other circuits.

- a. 120Vac for water heater. The operation of the heater circuit may be monitored by observing the heater current meter on the front panel. The heater controller is the proportional type; that is, it will reduce the average current to the heater coil as the temperature of the water bath approaches the sensor's set point. The temperature sensor may be checked with an ohmmeter for continuity after power has been disconnected.
- b. 120Vac for the 28Vdc bus. The 28-volt supply may be checked with a voltmeter. Locate IC32. The voltage between pin 2 and ground should be about 28 volts. If it is 25 volts or less, check pin 1 voltage. If pin 1 voltage is also low, the problem is between the regulator and the circuit breaker. If pin 1 is more than 3 volts higher than the pin 2 voltage, then the problem must be after the regulator.
- c. 28Vdc for charging the batteries. Turn off the 1-amp breaker. Measure the 28-volt bus voltage at the juncture of CR108 and CR113. The bus is now supplied by the batteries and should measure about 24 volts. The batteries are disconnected when the power switch is OFF. Measure the voltage at the batteries or at the battery-side junction of R118 (82 ohm) and CR108 with the power switch ON and with the 1-amp circuit breaker ON. A voltage greater than 24 should be measured depending upon the state of charge of the batteries.

- d. 28Vdc for the clock counter. The voltage at the "power" terminal of the clock counter should measure slightly more than 24 volts. A voltage much less than this would indicate a problem with Q2, CR106, R117, or C108. Turn OFF the 1-amp breaker. Slightly less than 12 volts should be measured at the "battery" input terminal of the counter. The counter will not display when operating from the batteries.
- e. 28Vdc to 12Vdc for integrated circuits (control console). Measure the voltage at pin 2 of IC31. The voltage at this point should be very close to 12 volts. If not, look for a possible short or a failed IC on the 12-volt side of the regulator.
- f. 28Vdc to 5Vdc for withdrawal-time decoder. The voltage at the positive side of capacitor C101 should be nearly equal to 5 volts. The LM309K regulator is mounted on a black, finned heat sink attached to the rear inside of the control console. If the voltage is too low, successively remove ICs 1, 3, 6, 11, and the PLA (the large gold-plated IC at the end of the IC board). Before removing an integrated circuit, turn OFF the power. Be careful not to bend the delicate pins when removing and inserting any ICs. Note the orientation and location of each IC before removing. If more than one IC is faulty, remove all of the above ICs and re-insert sequentially. Note that the PLA and IC3 are the only ICs wholly powered by the 5-volt and 12-volt buses.
- g. 28Vdc to 12Vdc in each sample stand. With the cable connected between the control console and the sample stand, measure the voltage at pin 2 of IC6 in the sample stand. The voltage should be nearly 12. If not, measure the voltage at pin 1. If more than 12 volts is measured, there is a faulty component in the regulator circuit. If no voltage is present at pin 1, check the 2-amp fuse and the float and limit switches.

Electronic subcircuits

Both an oscilloscope and a voltmeter will be needed to trace signal paths. Do not use an ohmmeter unless all ICs have been removed from the board. The battery in an ohmmeter can destroy an IC. Sample stands may be disconnected for sections a through e, because only the control console is tested. In the IC pin location column, the first number refers to the IC number and the second refers to the pin number.

clock counter. It should reset to all zeros and then start counting. If it does, then both this a. Clock reset (start). Turn the power OFF for 10-15 seconds. Turn the power ON and watch the circuit and the time base circuit are functioning normally. If the counter does not operate properly, follow the procedure given below.

Trouble isolator guide

Trouble	Possible cause	Cure or remarks
Counter resets but does not count	Bad connection IC 11C, 28B	Test for signal continuity. Replace faulty IC.
Counter does not reset	No time base Faulty switch IC 11D, 21B, 27B, or 27C	See section b. Replace. Replace.
Circuit tests		
IC-pin number	Signal, voltage, or transition	Remarks
28-12 28-11	High to low Low to high	When start switch is pushed. Do.

IC-pin number	Signal, voltage, or transition Remarks	Remarks
28-12	High to low	When start switch is mushe
14 01	117811 CO 108	אווכיו מכמד ב מאדבכוו דם המסווי
28-11	Low to high	Do.
26-11	Low to high	Do.
27-10	Positive pulse	Do.
21-4	Negative pulse	Do.
15-5	do.	Do.
11-10	Positive pulse (+5 volts)	Do.
7-2	Positive pulse	Do.
12-2	do.	Do.
23-15	do.	Do.

to generate the 1-Hz time base signal. Note that IC29 has power and ground pins (3 and 14, respecbinary counter. The 2-Hz square-wave output is divided once more by one of the flip-flops in IC28 b. Time base. IC 30 is a 4,194,304 Hz (4.19 MHz) oscillator which is divided by IC29, a 21-stage tively) in unusual locations.

Trouble isolator guide

Cure or remarks	Check 28-4, which must be low for 28A to operate. Pin 4 receives a positive pulse at EOA.	Pin 27-3 should be high. Pin 27-2 should have 2-Hz signal.	Replace if pins 1, 2, 3, 8, 14, and 16 do not pass the well-circuit tests.	Replace if pin 14 is high and pin 7 is ground.
Possible cause	IC28A is latched high	IC27A	IC29	1G30
Trouble	No 1-Hz time base	No 2-Hz signal		No 4.194 MHz signal

b. Time base. (Continued)

Circuit tests

IC-pin number	Signal or voltage	Remarks
28-1, 7-1, 11-7, 23-14, 22-6	1-Hz	Time-base output.
28-3, 27-1, 27-2, 29-8	2-Hz	Halt-run gate. Pin 29-8 is the 2-Hz output of the binary counter IC29.
27-3, 26-4	High	When this pin is high the 2-Hz signal is passed on to 28A. If this pin is grounded, the time base will be halted.
30-1, 29-16	4.194 MHz	Fundamental oscillator output.
29-1, 29-3, 30-14	High	Power pins.
29-14, 30-7, 29-2	Low	Grounds.

This circuit includes two decade counters, IC23 and IC24. IC23 divides the STIR ALARM switch on the front panel. The LED turns on after a 1-min delay and stays on for 1-min. is generated at 15-10 which sounds a "beep" from the audible alarm. The circuit is enabled by the condition, sends a reset signal to 15-6, and clocks, or "toggles," flip-flop IC25A. When 25-1 is high, the LED (light-emitting diode) STIR INDICATOR lights. When 25-1 goes low, a brief pulse time base by ten, and IC24 divides the output of IC23 by six. IC22 detects the divide-by-sixty The LED alternates 1-min on and 1-min off until the stir alarm switch is turned OFF. c. Stir-time counter.

Trouble isolator guide

Trouble	Possible cause	Cure or remarks
LED does not light 1-min after stir alarm switch turned ON	No time base	Check pin 23-14 for 1-Hz signal. If signal is not present, go to section b, time base.
	ICs 15B, 21A, 21C, 22, 23, 24, and 25A	Replace if IC fails the well- circuit tests given below.
	Transistor Ql	Replace if 10-3 does not go high when 25-1 is high (21-6 low).
	LED CR107	Replace if LED does not light when 10-3 is high.
No audible alarm when LED shuts off	Audible indicator	Replace if indicator fails to sound when 5-5 goes low. Positive side of indicator should be 28 volts.

c. Stir-time counter. (Continued).

Trouble	Possible cause	Cure or remarks
No audible alarm when LED shuts off	IC5	Pin 5 should go low when pin 2 (same as 15-10) goes low. The low is very short (½-sec or less) and occurs every 2-min.
Circuit tests		
IC-pin number	Signal or voltage	Remarks
23–14	1-Hz	Time base input.
21-1	High	Switched high by stir alarm switch on front panel.
21-2, 23-13, 24-13	Low	Clock-enable input to these decade counters. Counting function disabled if high.
23-15	Low	Short positive pulse resets this counter at EOA.
24-14, 15-4	Low	EOA reset the same as 23-15, and also reset at "60" count.
23-12, 24-14	1/10-Hz	Divided output from IC23.
22-9, 22-11, 25-3	1/60-Hz	Decoded output, alternating 30-sec low and 30-sec high.

c. Stir-time counter. (Continued).

Circuit tests

Remarks	Alternates 1-min high and 1-min low. When high, front panel LED stir indicator is on.	Alternates in opposition to 25-1.	Causes IC5 to turn on the audible indicator (to make a "beep") at the end of each stir indicator "on" period.
Signal or voltage	1/120-Hz	1/120-Hz	Short negative pulse every 120-sec
IC-pin number	25-1, 21-5	25-2, 25-5, 10-5	15–10

times. These times and binary time codes are given in appendix VIII. The four PLA outputs (pins 13, sense the presence of the 14-bit binary time codes corresponding to all of the necessary withdrawal 15, 17, and 19) are normally high (+5 volts), but go low (ground, or zero volts) for the one-second 16,384 seconds (4 hours, 33 minutes, and 4 seconds) each complete counting cycle. ICs 1, 6, and 11 PLA output back to the 12-volt bus used in the sample stands. Four LEDs (CR101, 102, 103, and 104) ICs 7 and 12 constitute a 14-stage binary counter which counts up to period when the binary counter output matches the internal withdrawal-time code. IC3 buffers the array (PLA). The PLA decodes the binary counter output; that is, the PLA has been manufactured are buffers which change the counter's 12-volt high signal to 5 volts for the programable logic serve as indicators of PLA output. Withdrawal-time decoder.

Trouble isolator guide

Trouble	Possible cause	Cure or remarks
No output pulse	IC3	Replace if PLA output pins 13, 15, 17, or 19 go low and the corresponding output pins of IC3 (2, 4, 6, or 8, respectively) do not go low also.
÷	PLA	Replace if output pins do not go low at appropriate inputs. Verify that 5 volts is avail- able at pin 24 and that pin 12 is grounded.
	ICs 1, 6, and 11	Replace if 12-volt high input does not produce a correspond- ing 5-volt high output. The

(low) when the output is low.

output should be at ground

d. Withdrawal-time decoder. (Continued).

Trouble	Possible cause	Cure or remarks
No output pulse	ICs 7 and 12	Replace if counters do not increment when clocked. Be sure that reset lines are low.
Circuit tests		
IC-pin number	Signal or voltage	Remarks
7-1	1-Hz	Time-base input.
12-1	1/64-Hz	IC7 output.
7-2, 12-2	Low	Reset lines. These ICs are reset with a short positive pulse after the start-reset switch is pushed.
7-12 & PLA-21	Low-low High (+12v) - high (+5)	Checking buffer 1A, pins 3 & 2. In like manner, check the other output pins of IC7 and 12 with the corresponding input pins of the PLA.
PLA-13, 15, 17, 19	Normally high (+5v) with 1-sec low at appropriate times.	Appendix VIII lists binary and digital times for decoded output.
3-2	Same as above, except +12v	When 3-2 goes low, LED CRIOL at 4-9 should light. In similar fashion, check the other IC3 buffer outputs and LED indicators.

e. EOA (end-of-analysis) decoder. IC13 senses the "all-high" condition of the eight most-significant bits of the binary time counter, ICs 7 and 12. This count appears 16,320 seconds (4 hours, 32 min) after reset. At this time, a short negative pulse is generated at 15-3. The pulse is inverted to a positive pulse at 27-11 and is used to reset the start-reset flip-flop at 28-10.

Trouble isolator guide

Trouble	Possible cause	Cure or remarks
No EOA pulse (clock counter continues counting beyond 16,320 seconds)	IC13	Replace if output does not go high when all 8 inputs are high (at 16,320 seconds after resetting).
	IC15A or 27D	Replace if faulty. See well-circuit tests for measurements.
Circuit-tests		
IC-pin number	Signal or voltage	Remarks
13-2, 13-16, 13-10	High	Pin 16 is power, pins 2 & 10 help determine the IC's function.
13-7, 13-8, 13-9, 13-15	Low	Pin 8 is power ground, the others help determine the IC's function.
IC3, pins 3-6, 11-14	High	Pin 1 will go high when all 8 input pins are high. Otherwise, these 8 input pins follow the indicated IC7 and 12 output pins.
15-3, 27-12	Negative pulse	When 13-1 goes high.
27-11, 28-10	Positive pulse	When 13-1 goes high IC28 (and the time base signal) is latched off when the pulse reaches

28 - 10.

start circuit: a pushbutton start switch located in the front on the left beneath the top deck, and f. Sample stand start circuit. The 1-sec negative pulse from the control console is inverted to a The positive pulse, one side of the detent switch. The first switch allows the sample stand to be started manually for in turn, is re-inverted by IClB for triggering siphon timer IC5A. There are two switches in the The detent switch disables the input line when the tray motor is operating. 2B, and 3A. positive pulse by IClA for the siphon solenoid flip-flops, ICs 2A, testing purposes.

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Cure or remarks	Check earlier sections. Check start-up with manual start switch.	Locate fault and repair.	Check as indicated in well- circuit tests. Replace if faulty.	Replace if 1-2 is not grounded through normally closed contacts.
Possible cause	Control console	Cable	IC1	Detent switch
Trouble	Will not start			

f. Sample stand start circuit. (Continued).

Circuit tests

Remarks	From control console. A positive pulse should appear at 1-3 if 1-2 is low.	Goes high only when detent solenoid is activated.	Pulse is stretched slightly by R203 and C201.	Positive pulse is inverted by IClB.
Signal or voltage	1-sec negative pulse	Low	Short positive pulse	Short negative pulse
IC-pin number	1-1	1-2	1-5, 1-6, 2-6, 2-8, 3-6	1-4, 5-6

timed period. R206 adjusts the duration. The positive output turns on Q4 which turns on indicator g. Siphon timer. IC5A is an integrated circuit timer controlled by potentiometer R206, resistor R207, and capacitor C203. A negative pulse at 5A-6 causes the output (5A-5) to go high for the LED CR207 (on the IC board) and IRLEDs CR9, 10, and 11 in the brackets beneath the top deck.

Trouble			
	Possible cause	Cure or remarks	
Siphons will not shut off IC5		Replace if faulty. See well-circuit tests.	ee well-
LEDs, and	LEDs, photo-transistors, and flip-flops.	See other sections below.	low.

Circuit tests		
IC-pin number	Signal or voltage	Remarks
5–5	14-sec high	Output to Q4 and IC1C. Pulse duration adjusted by R206.
1-10	14-sec low	Resets flip-flops 2A, 2B, and 3A positive-going edge at end of pulse.
5-4	High	Reset line is buffered against

Q1. The siphon solenoid is switched on from the 28-volt bus. As the solenoid pushes the pinch-valve the solenoid is powered through R212. The circuit is designed to operate in this manner to provide transistor Q1, and solenoid S1. The flip-flops are set by the positive pulse from IC1A. When set, spring from the tubing, the small snap-action switch (SW2) is pushed open. When the contacts open, the "Q" outputs go high and the "Q" outputs go low. In this example, 2-1 goes high which turns on h. Siphon solenoid latch. There are three identical latch circuits. One such circuit is IC2A, high power for the initial thrust then reduce power for the remainder of the withdrawal period.

Trouble isolator guide

Trouble	Possible cause	Cure or remarks
Siphon will not start	Flip-flop IC2 or 3	Replace if faulty. Check with well-circuit tests.
	Transistor: Q1, Q2, or Q3	Replace if collector (wired to low side of solenoid) does not go low when 2-1 ("Q") output goes high.
	Diode short: CR202, CR201, or CR204	Remove power from sample stand. Remove wire-wrap board, and test diode resistance in both directions with ohmmeter. Replace diode if resistances are nearly equal.
	Solenoid open: S1, S2, or S3	With wire-wrap board removed, test solenoid leads for

continuity with ohmmeter.

Replace if open.

h. Siphon solenoid latch. (Continued).

Trouble	Possible cause	Cure or remarks
Siphon will not start	Switch	Push switch and listen for click. Measure voltage at positive side of solenoid (terminal 10 on wire-wrap for Q1-S1) before pushing start switch and afterwards. Voltage should drop from 28 to between 12 and 18 volts.
Siphon will not shut off	Siphon solenoid cut off circuit	See next section (i).
	Flip-flop IC2, 3	<pre>IC fails to reset from reset line or clock line. Check well-circuit tests and replace if faulty.</pre>
	Timer IC5A	See previous section (g).

h. Siphon solenoid latch. (Continued).

Circuit tests

IC-pin number	Signal or voltage	Remarks
2-6 (2-8, 3-6)	Positive pulse	When start signal received, or when start switch is pushed.
2-1 (2-13, 3-1)	High	This output goes high when 2-6 receives its positive pulse. Q1 then turns on solenoid S1.
2-1 (2-13, 3-1)	Low	This output goes low when the reset line 2-4 goes high or when the clock line goes high when timer 5A goes low (at the end of its timed period) and which, in turn, is inverted by 1C.
2-2 (2-12, 3-2)	High	This output goes high when 2-1 goes low, that is, when 2-3 (clock) or 2-4 (reset) goes high.
2-5 (2-9, 3-5)	Low	Data input. Must be low so that flip-flop is reset when clockline goes high.

IRLED CR9 is aimed slightly for stopping the siphon after a fixed sample volume has been withdrawn. Transistors (8 and 05, with Each siphon latch circuit (item h, above) has a complementary circuit to one side of photo-transistor Q8. When the meniscus of the water-sediment mixture passes through pulse to 2-4. This, in turn, causes 2-1 to go low (turning off the solenoid) and 2-2 to go high. the beam of CR9, the beam is deflected away from Q8. The brief turn-off of Q8 causes a negative pulse to be passed to the base of Q5. This transistor now conducts which sends a positive reset their associated resistors and capacitors, perform this function for S1. i. Siphon solenoid cut-off.

Trouble isolator guide

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Trouble	Possible cause	Cure or remarks
Siphon will not shut off	IRLED: CR9, CR10, or CR11	If CR207 (red LED on wire-wrap board) lights, all 3 IRLEDs are OK. If CR207 does not light, replace faulty LEDs. See circuit test for isolating procedure.
	Phototransistor: Q8, Q9, or Q10	Replace if faulty, but check for misalignment first. Transistor should conduct with LED aimed directly at it.
	Transistors: Q5, Q6, or Q7	Transistor should conduct if base goes low. Replace if faulty.

See previous section.

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Flip-flop IC2,

i. Siphon solenoid cut-off. (Continued).

tests	-
Circuit	

Remarks	With sample tray removed, push start switch then move finger between the IRLED and phototransistor pair (in bracket below deck) for each siphon tube. Each siphon solenoid should shut off as the beam is interrupted. The IRLEDs should be aimed 3-5° away from the phototransistors.	Under same conditions as above.	When timer 5A goes high, Q4 switches on and turns on the LED string.	Voltage drop across Q4 when timer 5A switches on and LED string is operating.	Voltage measurement made while operating; measured from anode (positive side) to cathode (negative side).
Signal or voltage	Positive pulse	Negative pulse	Low (3-5 volts)	Very low voltage (less than 0.5 volt)	Approx. 1.2 volts
IC-pin number	2-4, 2-10, or 3-4	Phototransistor emitter	Terminal 18 of wire-wrap board	Transistor Q4 collector	IRLED: CR9, 10, 11

3-A-2) are high. A short negative pulse is sent to 5B-8, the timer's trigger input. Potentiometer R230 adjusts the output pulse to a three to five second duration. When the output goes positive, j. Detent timer. IC4B triggers timer IC5B when all three Q flip-flop outputs (2A-2, 2B-12, and Q11 activates the detent solenoid. Capacitor C210 supplies the initial power surge to minimize momentary dips in the power supply.

Trouble isolator guide

Trouble Detent solenoid will not activate	Possible cause Solenoid Transistor Q11 Timer IC5B	Replace if tests prove that it is faulty. Do.
	Gate IC4B	Do.
Detent solenoid on too long or too short a time	Timer adjustment	Repeat operation after adjusting potentiometer R230. Detent should be on for 3 to 5 second

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j. Detent timer. (Continued).

	Remarks	This pin is high when any of the 3 inputs (2-2, 2-12, and 3-2) are low.	Only when all 3 inputs are high.	Following the high-to-low transition of 4-6.	High output duration adjusted by potentiometer R230.	During output pulse.	Measured after first second.	Measured with detent solenoid relaxed. Voltage should remain high for some time if power cable to control console is removed.
	Signal or voltage	High	Low	Negative pulse	Positive pulse (3-seconds duration)	Low (less than 0.5 volt)	Medium (12-18 volts)	High (28 volts)
Circuit tests	IC-pin number	7-6		5-8	5–9	Transistor (11 collector	Detent solenoid	C210 (3000 µF)

k. Tray motor. The tray motor advances the tray when the detent switch is activated by the detent pin, but only if the motor-support plate is in the proper position. In that position, the motorposition switch connects the positive power lead to the 28-volt bus. The detent switch connects the negative lead to ground when it is activated.

Trouble isolator guide

Motor will not run Motor-support plate position Motor-position switch Detent switch Motor drive not engaged	Replace if motor does not run when proper voltage is applied to positive and negative leads. Support plate should activate motor-position switch. Replace if not activated by motor support plate. Readjust or replace if switch is not activated by detent solenoid.
	try to turn test-tube tray by hand.

(Continued). k. Tray motor.

The common lead should be grounded. The normally-closed lead will grounded <u>only</u> when the detent solenoid is activated. The detent solenoid is activated. switch is manually operated. be grounded except when the motor should run when this The normally-open lead is Remarks Signal or voltage As noted Motor-position switch Detent switch Circuit tests

As noted

APPENDIX VII - AUTOPIPET WITHDRAWAL CALCULATIONS

depth from the surface. Even values of n give the withdrawal times for "keeper" subsamples, and odd values are for flush withdrawals which are discarded. The withdrawal times for the odd n values are average wet-surface area inside the cylinder to find the average change of elevation per withdrawal, the This value, in turn, is used to calculate the mean depth of the siphon intake at the time of withdrawal, $_{
m n}$; and finally, to calculate the time required for a particle to settle through this water temperature and the pertinent physical dimensions of the cylinder, siphon tubing, and the The calculation proceeds by dividing the average volume of each withdrawal by the obtained by subtracting 23 seconds from the next even-n withdrawal time. The sample stands are staggered 120 seconds apart. Calculated withdrawal times and their binary-code equivalents are The calculation of withdrawal times for the autopipet begins with the given parameters: given in Appendix VIII. mixing tool.

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Water temperature,	$T_{W} = 30^{\circ}C$
Cylinder diameter (inside),	$^{\mathrm{D}}$ cyl = 5.813 cm (ave. of 10 samples)
Siphon tubing diameter (outside),	D tube = 0.620 cm
Mixing rod diameter (outside),	$D_{rod} = 0.31 cm$
Average withdrawal volume,	$V_{\rm wd} = 29.1 \mathrm{cm}^3$
Withdrawal number,	$n = 1, 2, \dots 12$
Siphon time	= 9 sec for first withdrawal

sec for last withdrawal

APPENDIX VII - AUTOPIPET WITHDRAWAL CALCULATIONS (Continued)

CALCULATION:

Average wet-surface area inside cylinder,

 $A_{\text{cyl}} = \frac{\pi}{4} (D^2 \text{cyl} - D^2 \text{tube} - 2D^2 \text{rod})$ $= 26.08 \text{ cm}^2$

> Average change in elevation per withdrawal,

 $\Delta h = \frac{V}{v} / A_{cyl} = 1.116 \text{ cm}$

Fall distance for given n,

 $H = h_0 - (n - \frac{1}{2}) \overline{h} \text{ cm}$

where initial depth of intake is

 $h_0 = 17.0 \text{ cm}$

Time (seconds) for a particle of diameter d(µm) to settle through a distance H (cm) at $30^{\circ}C$

 $t_{30} = 8876 \text{ H} / \text{d}^2$

Example: Determine the fall time for the 62-um subsample. This is the second withdrawal (a flush subsample was first), so n = 2.

 $\frac{H}{n} = \frac{h}{0} - (n - \frac{1}{2}) \frac{\Delta h}{\Delta h} = 17.0 - (1.5) \text{ 1.116} = 15.33 \text{ cm}$

 $t_{30} = 8876 \text{ H}/d^2 = 8876 (15.33)/(62)^2 = 35.4 \text{ seconds.}$

Because this is for the midpoint of the withdrawal which takes about ten seconds altogether, the withdrawal begins five seconds earlier, or at 30 seconds.

APPENDIX VIII - CODING FOR PROGRAMABLE LOGIC ARRAY

Herein are given the binary coding for the PLA (programable logic array) for a constant water-bath temperature of $30^{\circ} \pm \frac{1}{2}^{\circ}$ Celsius. Withdrawal times for sample stand 1 were calculated as shown in Appendix VII. A 23-second delay was used between the flush withdrawal and the size-sample withdrawal. The withdrawal times for sample stand 2 were calculated by adding 120 seconds to the times for sample stand 1. Additional 120-sec delays were added in order to calculate withdrawal times for sample stands 3 and 4.

APPENDIX VIII (Continued).

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APPENDIX VIII (Continued).

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