

AN AUTOMATIC PROPORTIONAL
SAMPLING INSTRUMENT

By

ROYCE MARVIN WHITE

Bachelor of Science

Louisiana State University

Baton Rouge, Louisiana

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SAMPLING INSTRUMENT

Thesis Approved:


Thesis Adviser




Dean of the Graduate School

PREFACE

This paper is an attempt to solve a long-standing problem in chemical process work. The problem is that of obtaining representative samples for analytical purpose, and the thesis describes the design and application of an instrument that will automatically perform that duty.

Research, design, and testing has been conducted over the past two and one-half years. The instrument has been subjected to actual field tests at a natural gasoline plant since July, 1954. Testing has been continuous since that date.

I wish to thank my advisor, Dr. Robert Maddox, for his suggestions and criticisms of the thesis material. I wish also to thank Phillips Petroleum Company for their permission to use the project as a basis for this paper. Credit is due Miss Jeane Williams for her efforts in the preparation of the final manuscript.

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INTRODUCTION

The need for representative samples in analytical work is often stressed. This is especially so in the petroleum industry where many analyses are performed, in a routine manner, to forecast daily and monthly plant operation.

Oftentimes, a continuous or intermittent sample is extracted from a flow stream over an extended period so that such sample may take the place of, and perhaps give better results than, a number of individual sample analyses. Such a sample is called a composite. Generally, the extraction of the composite sample is made without regard to instantaneous flow rate of the line being sampled. Such a procedure gives results with accuracies that vary as the volume rate in the process stream varies.

In those instances where the composite volume must reflect the flow stream history with extreme accuracy then a sampler of the proportionating pump type is used. Such an installation costs approximately \$2,500. Even at this price, the flexibility and general merit of the installation is sometimes questionable.

Automatic samplers, that give no attention to sample-flow proportionality, are not uncommon. These make use of an electric or spring-wound interval timer and a suitable valve arrangement.

This paper proposes an inexpensive instrument that will automatically take a flow-proportional sample.

The proposed unit has been designed to function unattended for a period of as long as one month. The cost of material and labor for such

an installation is estimated to be \$600 plus or minus \$100 depending upon existing facilities.

In some applications the instrument may be considered a manpower saving device; however, the designer's intentions were to make available a means to obtain a sample that would be more truly representative of flow stream history than those given by normal procedures now in use at a typical petroleum sampling gathering station.

The proportional aspect of the instrument described here is afforded by the linear variation with time that is experienced by the fall of a mercury column in an orifice meter when the orifice differential to the meter is reduced to zero. During the course of mercury fall an electric circuit holds a sampling valve open. The sampling valve allows a liquid to discharge from a container and the sample fills the void in that container. Such is normally called liquid displacement sampling. The meter mercury is a component of the circuit that maintains energy on the sampling valve. When the mercury ceases to fall, due to the assumption of the "no differential" position in the meter manometer, the electric circuit to the sampling valve is broken since the meter mercury no longer completes the circuit. Since the original height of the mercury was related to flow rate in the header, the duration of sample extraction and consequently the amount of sample is likewise a proportional function of header flow rate.

A plot of sample volume drawn per unit of main line flow rate is shown on Figure 8. The linear relationship between flow and sample extraction is seen to be essentially perfect. Sample extraction is generally within 0.5 cubic centimeter of the designated extraction for absolute proportionality. Thus, when a 50 c.c. sample is indicated, the deviation will be 1 per cent.

In using this proposed method, the mathematical true average composition would be approached as the frequency of sampling increased. As discussed later, a balance among equipment wear, sample accuracy, and container size is sought.

OPERATION OF THE INSTRUMENT

This automatic weighted average sampling device is designed to extract a sample from a line in an amount that is directly proportional to the "root" reading (see Appendix) of the flow meter on the line. The term, root reading, means the square root of the differential pressure and as such is directly proportional to flow. This terminology is very convenient as will be seen later. At a reading of zero roots, no sample is drawn; at ten roots maximum sample volume is drawn; at nine roots, nine-tenths of maximum sample is drawn, etc. If the sampling is frequent enough, then an analysis of this monthly composite sample will indicate the amount of liquid gasoline that can be extracted from an inlet rich gas stream and should check closely with production figures. Moreover, should such an instrument be installed on each of two (or more) gas streams going to a common absorption system, the monthly analysis of each inlet sample would furnish the basis upon which the one gasoline production stream can be credited to the individual inlet gas streams.

The mechanics of extraction of the sample is by means of conventional liquid displacement, and the subject instrument is employed to regulate this liquid draw-off. Liquid displacement type sampling is the extraction of a sample by means of removing a liquid from a container, usually at the bottom, and allowing the sample to fill the void. Either gravity or pressure difference may be employed to cause the sample to enter the sampling container. The pressure method is used almost exclusively. Though the displacement fluid is usually called "water," pure water is rarely employed.

The procedure described in these pages outlines methods of obtaining a sample of gas from a gas stream at about 60 psig. A gas regulator reduces this to 50 psig constant pressure. Gas at very low pressure will ordinarily have to be sampled with oversized containers and in larger incremental amounts in order to have sufficient standard condition volume for analysis.

Although the utility of the instrument is explained here in terms of natural gasoline plant operations, it may find application in any plant where gases or liquids are handled. The instrument was born of necessity in a special gas sampling situation. Accordingly, the description is centered around gas operations. Liquid sampling, as will be seen later, requires less special consideration and will be discussed briefly. This is not to diminish the importance of liquid sampling for, considering the number and relative financial value of liquid process streams, liquid sampling would be of more general interest.

The proportionality between flow rate and sample extraction is made possible through the use of an electrode (1) (see Figure 1) immersed in the mercury of the low pressure side of the orifice meter. At two-hour intervals an automatic electrical timer (2) opens a solenoid valve (3) that equalizes the pressure in the two legs of the orifice meter. The mercury in the high leg begins to fall to an equilibrium position of zero height with reference to the other mercury leg. If the electrode has been adjusted in length to relay to an external electrical circuit the precise time that the mercury column has reached its rest position then the time of mercury fall, which is a function of original mercury height, could be relayed to a second solenoid valve (4) that was extracting a gas sample. The duration of sample extraction, conducted at constant sample flow rate,

is thus a direct function of gas flow in the gas header and the amount of sample extraction is consequently directly proportional to gas flow. Mathematical proof of this proportionality is shown in the appendix.

In practice, the electric output from the timer (2) is fed to the pressure equalizing valve (3) and the water discharge (sample extraction) valve (4) simultaneously. This timer output is presently set for 75 seconds duration per each two-hour period. During this entire time the pressure equalizing valve is open, and the water discharge valve is open for that portion of the 75 seconds that will be required according to the original height of the mercury column. The rate of fall of mercury is calibrated, by means of the meter dampening-drain valve (5), so that a mercury height equivalent to the total range of the meter (100 inches of water differential, or ten roots) will fall to equilibrium in exactly 60 seconds. Should a 50-inch differential pressure meter be used, the full scale reading would be 7.07 roots on a 0-10 square root chart. Full scale mercury fall could be calibrated to the arbitrary 60 seconds or any other convenient time. Proportionality of sample extraction to process flow rate would be the same. With reference to the 100-inch meters, each root that is registering on the meter at the beginning of a sampling cycle will result in a sample extraction of six seconds duration. Thus, the maximum open time of the water discharge valve would always be less than the timer output period. When 75 seconds have elapsed, the valve closes and the mercury columns assume the position that is indicative of gas header flow rate. This time, 75 seconds, must be chosen such that it is slightly longer than the full scale mercury fall time and yet no longer than is necessary for the extraction of the sample. The 15 seconds extra time is as small as can be safely adjusted on the present experimental equipment.

The recording pen of the flow meter will fall to zero as the mercury differential falls and will remain there until orifice differential is restored to the meter at the end of the 75-second period. An actual 24-hour chart showing this feature is seen on Page 65.

With a sampling frequency of two hours and a composite period of one month, approximately 372 water-equivalent gas samples will have to be stored in the sample container. It is field practice to use a standard 9-1/2 gallon stainless steel oxygen bottle for this service. A further general stipulation is that the monthly composite gas volume be divided into three portions (for check analyses) with the pressure of the original sample necessarily being the driving force to fill the two extra check sample containers; also the last container filled must be at atmospheric pressure or above. The three desired volumes are usually not equal but are most often two-9 1/2 gallon containers plus one 5 gallon glass bottle, a total of 24 gallons gas volume, at a minimum pressure of atmospheric. Thus, an operating pressure held on the monthly gathering container must be used that will allow 9-1/2 gallons of gas volume to be expanded to 24 gallons volume and yet have the expanded volume no lower than atmospheric pressure.

The high pressure on the sampling bomb is the driving force to displace water from the sampling container to atmospheric drain in the process of extracting the periodic gas samples. This pressure, approximately 50 psig, would cause water flow rates, during sampling periods, far in excess of sample volumes that are compatible with the above unless the displacement liquid flow line were restricted. The installation of an orifice or other restriction in the water discharge line would require a passage in the order of pin hole size, at these pressure drop conditions, 50 psi to

0 psi, and such a passage would undoubtedly become plugged. If it is considered that the previously mentioned volumes and pressure are unalterable, the upstream pressure of the water must be reduced between the sample storage space (6) and atmospheric discharge (7). The use of a liquid pressure reducing valve would be highly impractical, if not impossible. The desired pressure reduction can be affected by adding an auxiliary container (8) that receives water at high pressure through a 3-way solenoid valve (4) and discharges through another port of the same valve at a pressure that is due only to the elevation (9) of the auxiliary container. Discharge from the auxiliary container takes place during a sampling cycle and the auxiliary container is automatically refilled when the cycle is over. The sample is extracted when the displacement liquid is pressured into the auxiliary container. This container should be of dimensions such that the fall in head, while sampling, is negligible compared to the total head. A special valve (13) has been installed that functions as a vacuum breaker and as a liquid full closure device. This item is actually a diaphragm that allows the top of the auxiliary tank to float. When sampling pressure is placed on the tank, the diaphragm is extended to its upper position and held there by a metal retaining wall. When the auxiliary tank is discharging its contents, the diaphragm hugs the surface of the liquid and discharge is due to hydrostatic head only. A vent valve is installed in the center of the diaphragm so that gas may be expelled after the initial liquid fill and at such other times as may be necessary. With the addition of a nominally sized orifice (10) between this container and atmospheric discharge, the water flow rates can be brought in line with those required and yet keep within a moderately sized monthly water inventory.

The orifice (10) serves the dual purpose of restricting the flow and allowing dependable calculation of flow rates. An orifice diameter of 0.042 inches and a water discharge head of about 14 inches has been found to give flow rates that will yield satisfactory monthly volumes. This water discharge rate is calculated from the formula

$$q = 19.64 \times D^2 \times K \times \sqrt{h} \text{ (Eq. 1)}$$

wherein q = flow in GPM

D = diameter of orifice, inches

h = head at orifice, in feet of fluid flowing

K = orifice discharge coefficient

The value of "K" is the only unknown in the above equation, for a given set of conditions, and is readily determined by solving the equation.

"K" is a function of the type of orifice and its individual characteristics. Since "K" for the orifice in the present instrument has been determined (see Appendix) to be 0.685, a flow rate for the 0.042 inch diameter orifice and 14-inch head is calculated to be 97.2 cc/min. A complete set of calculations is shown in the appendix.

It is to be understood that the 97.2 cc/min. is the displacement liquid flow rate, and its magnitude does not change unless a substitution is made in Equation 1. At ten roots meter reading and at sampling intervals of two hours this is a liquid consumption rate of 9.52 gal./mo. At five roots constant meter readings this figure becomes $9.52 \times 5/10 = 4.76$ gal./mo. As shown, monthly water consumption would depend upon meter reading.

There would be a low average monthly meter reading that is insufficient to extract the required sample volume. This value is calculated to be 5.25 roots. This spread of 4.75 roots is considered wide in

comparison to normal flow fluctuations at an installation of the type that the instrument would serve and the range limits can be moved by merely changing the elevation of the auxiliary container, or the size of either the metering or the sampling orifice. If the instrument is adjusted in anticipation of ten roots monthly average meter reading and the average should actually be three roots, there would be insufficient sample for analysis. Conversely, if a predicted three roots adjustment should meet with an actual monthly rate of ten roots, the entire liquid contents would be displaced when the month was three-tenths gone. Figure 11 shows a low liquid level shut-off float to prevent loss of gas sample should this occur. The range of meter readings should be kept in mind in calculating monthly water requirements. The sample container must be filled with liquid at the beginning of each month. Calculations to indicate the proper elevation (9) of the discharge point, once the extremes of operating flow rates are known, can be accomplished with the use of Equation 1.

Any root reading over ten roots will extract a proportional sample within the overrange limits of the meter.

To reduce the possibility of plugging, the use of a sampling orifice (10) diameter of less than .042 inches is inadvisable. The use of a circular, sharp edge orifice was found to be the most practical method of restricting the flow, especially in view of the flow rates and pressures anticipated. This is true because the circular orifice offers a greater area for the passage of fine extraneous particles. Trial runs have proven that various needle valves, bourdon restrictors, etc., due to the geometric configuration of their flow area, would, at settings commensurate with required flow, become plugged with extremely fine particles.

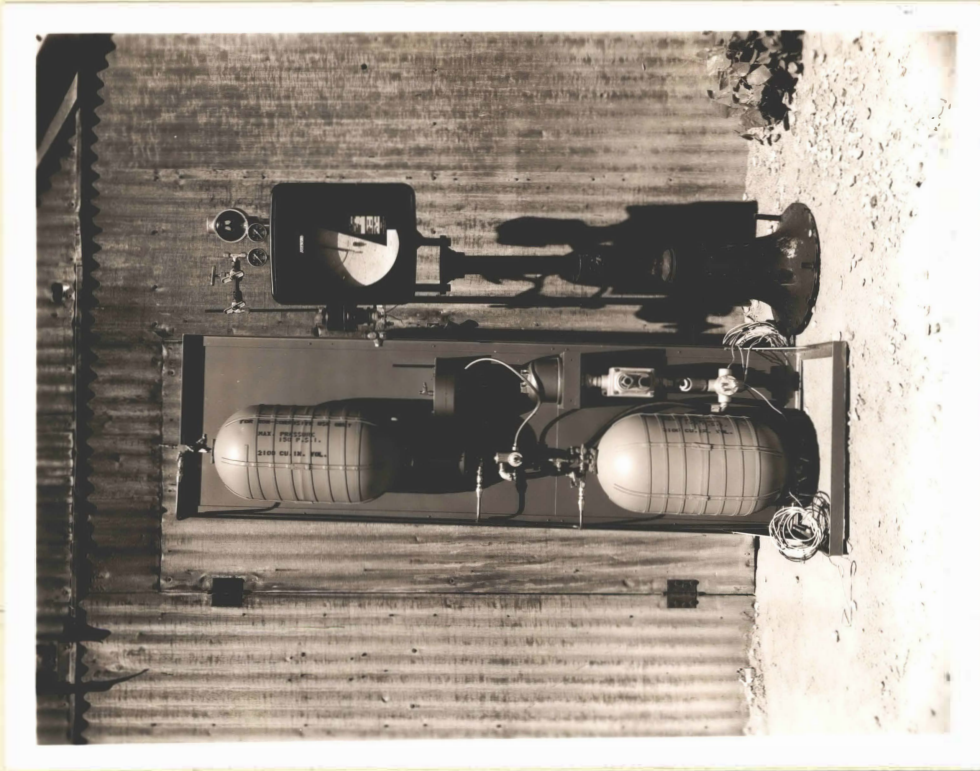
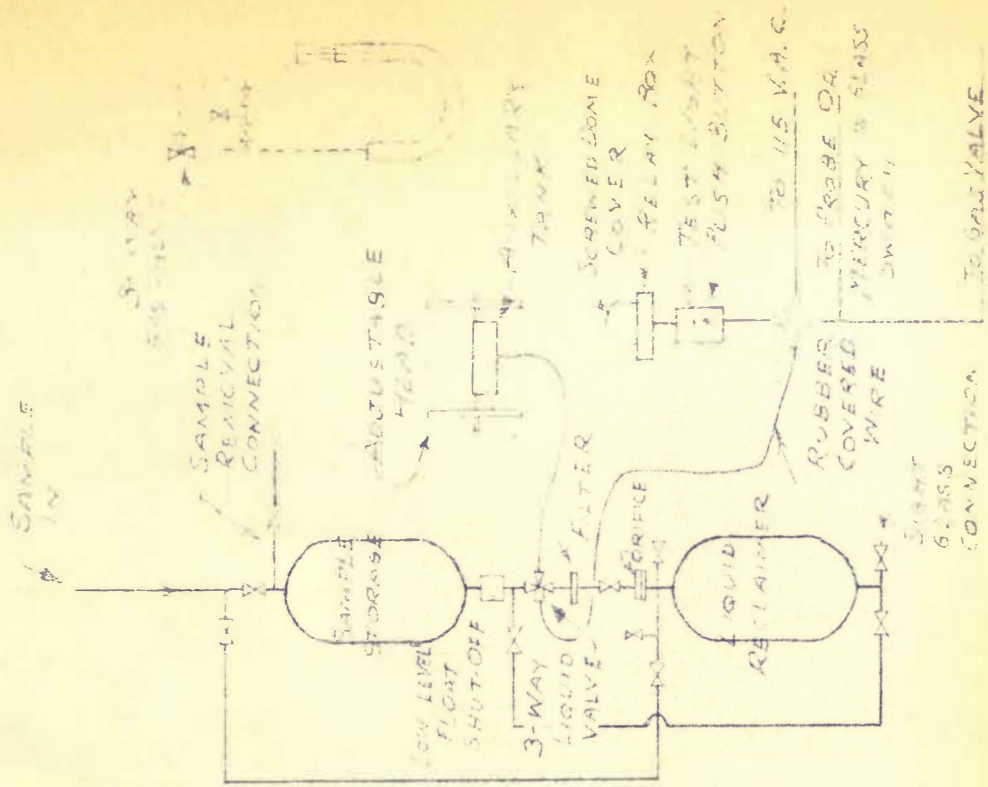


FIG. 2
 PHOTOGRAPH AND SCHEMATIC LAYOUT
 AUTOMATIC PROPORTIONAL SAMPLER

			DRAWN		AFE. NO.	DWG. NO.
			CHECKED		SCALE	SHEET NO.
REVISION	BY	DATE	APPROVED			

THE ELECTRIC CIRCUIT

The period of electrical contact in the electrode circuit, as previously explained, is a rigorously true function of metered gas flow, but certain practical reasons necessitate that a side circuit be introduced. To obtain the desired proportionality between gas flow and sample extraction, the electrode would have to be adjusted to within an infinitely small distance from the rest position of the mercury surface. Here, various mechanical malfunctions could open or close the intervening gap indiscriminately whereupon the expected loss of electrical contact at rest position might not occur. Further, and of more real importance, the temperature coefficient of expansion of mercury, along with the other metals involved, is such that yearly ambient temperatures could vary the rest position surface of the mercury within a range equivalent to about one root. The incremental height of the mercury column is not directly proportional to gas flow rates but is proportional to the square of the flow rate. This is shown in Appendix. The fall of the mercury column from ten roots to nine roots value is nearly two inches where the fall from one root to zero is about $1/12$ inch even though the times of fall from ten to nine and from one to zero roots are equal. The $1/12$ inch figure is arrived at in the following manner. One root is 1 inch of water differential; 1 inch of water is approximately $1/12$ inch of mercury. Flow meters are calibrated to read inches of water differential but the differential indicating fluid is actually mercury except in cases of meters measuring extremely low differential. The proportionality

feature is gained from the fact that the higher mercury columns fall faster and cover the progressively smaller distances in a linear manner with respect to time. This explains why a small discrepancy between the end of the electrode and the mercury equilibrium point could destroy the intent and purpose of the time measurement, and consequently, the proportional-to-flow sampling.

Fortunately, the electrode can be withdrawn to a finite distance above the equilibrium surface provided that a time delay function is installed in the electrode circuit, such time delay being precisely equal to the known time equivalent of the withdrawal distance, 6 seconds for one root, 9 seconds for 1-1/2 roots, etc. These values are on the premise that full scale mercury fall occurs in 60 seconds. In practice, the internal circuit would be broken when the mercury fell below the electrode; this loss of energy would activate the time delay circuit and when the delay had elapsed this delay circuit would then de-energize the water discharge solenoid valve. The time delay circuit (11 of Figure 1) maintains 115 volts (through a relay contact) to the water discharge valve. The actual time of delay is variable to suit operating preference and to serve as what may be termed a zero adjustment. In practice, the disruption of current in the wire (12 of Figure 1), due to mercury falling underneath the probe, causes the time delay circuit to be energized, through a relay, and at the expiration of the pre-set time delay period a relay breaks the water discharge valve electric circuit and the 3-way valve ports reverse. The gas sample is extracted from the header when liquid from the collecting (sampling) tank is pressured into the partially void auxiliary tank.

A detailed account of an electric cycle is described as follows: Refer to Figure 3; when the timer motor indicates expiration of the two-hour period, the timer switch applies 115 volts to the other components of the circuit. Note that through binding post number 6 the gas valve and the liquid valve are thus connected to source voltage. The gas valve has a direct and continuous return to ground through binding post number 5, and the liquid valve has a path to ground through binding post number 7, through the delay switch, through the rectifier, then to the ground wire. The rectifier is in the circuit because the liquid discharge valve, in 1/8 inch pipe connection size, has better mechanical characteristics in the D.C. model than does the A.C. model. This has application to the particular brand used rather than to solenoid valves in general. The delay switch is closed because the delay motor, at this part of the cycle, has not begun to rotate since the simple relay is holding the relay switch open. The delay motor is an electric clock mechanism. At the instant the mercury falls below the probe, the simple relay windings lose energy and the delay motor has its circuit completed. When the cam finger on the delay motor shaft has revolved its pre-set angular distance, it mechanically opens the delay switch and the liquid valve ceases to discharge. When the active period of the timer motor has elapsed, then the gas valve reverts to its normal position that places differential pressure on the orifice meter.

Calibration of the flow meter is typical of any flow meter. Adjusting the electrode tip to a certain distance from the mercury surface is done by observing the meter reading at the time the internal electric circuit is broken as evidenced by the test light. This light will signify the time that mercury falls underneath the electrode since the mercury

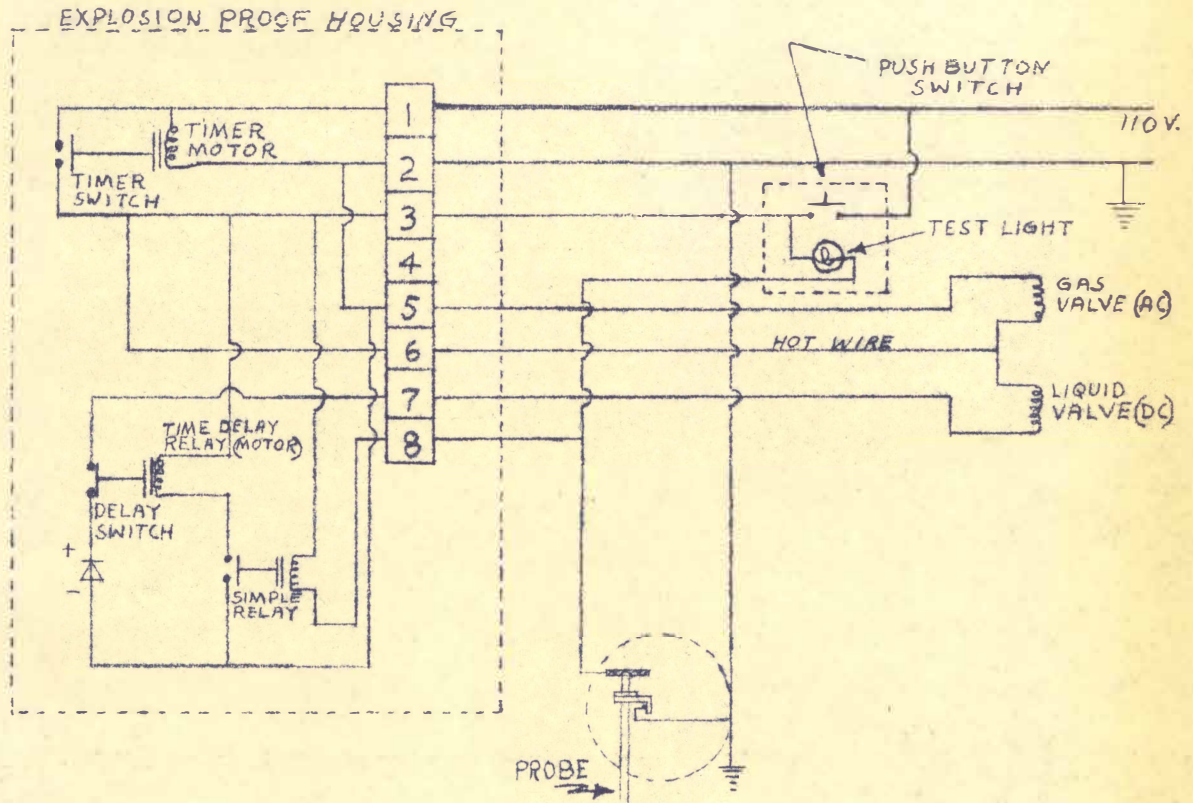


Fig. 3
ELECTRIC DIAGRAM
AUTOMATIC PROPORTIONAL SAMPLER

DRAWN	RMW	AFE. NO.	DWG. NO.
CHECKED		SCALE	SHEET NO.
APPROVED			

REVISION

BY DATE

serves as ground return path for the light. Thus, to make an electrode length setting for a nine second delay, the electrode length is adjusted to approximately the correct height and evidence of the proper setting is seen when the test light goes out, at falling mercury conditions, at the instant the meter pen reads 1-1/2 roots or 2.25 inches of water if the chart is of the inches differential type. The compatibility of nine seconds with 1-1/2 roots has been previously discussed. It must be understood that some means, real or simulated, must be provided to actuate the mercury column through its full scale deflection for calibration purposes. Zero to 3.6 pounds per square inch of positive pressure on the upstream tap of the meter manometer will accomplish this.

Another way of changing the relative electrode height is to add or remove mercury from the meter and re-zero the meter calibration. This would be limited to a correction of plus or minus one root.

Since the various relay mechanisms must be enclosed in an acceptable explosion-proof box for petroleum plant locations, the test light is an item of necessity. Without the test light there would be no way to sense the beginning of the time delay period with the metal cover of the relay box tightly closed. Thus, a diagnosis of that part of the circuitry that may be causing unproportional liquid discharge rates would be impossible without the test light if explosion-proof conditions are to be maintained.

Included as an integral part of the test light assembly is a manual push button switch. The housing for this is likewise explosion-proof. An examination of Figure 3 will show that the switch will allow an observer to begin a sampling cycle, for test or observation, without waiting for the end of a two-hour period. Holding this switch closed will cause the instrument to take a proportional sample at any time, and

momentarily depressing the switch will show the observer that the instrument is working.

The existence of the electrode-to-mercury contact, in the light of a possible safety hazard, has been the subject of considerable study. A volume of transformer oil will fill the area above the mercury surface of the meter. Consequently, when the contact is broken, the small arc will form at the mercury-oil interface. The guaranteed presence of the oil over the mercury surface is maintained with the installation of the seal pots (as shown on Figure 1) and the two mercury check valves that are normally incorporated into the meter body. Efforts have been made to design for all procedural errors or operational mishaps that would place a sudden unbalance of differential pressure on the meter manometer fluid (mercury) and possibly expelling the fluid from the manometer body, thereby tending to cause an arc at the mercury-gas interface. It is felt that the described arrangement will be adequate. Raw natural gas or any normal petroleum plant fluid is not an explosive mixture, due to the absence of oxygen. The application of the transformer oil renders the electrode arc non-hazardous according to the National Electric Code, Article 500, Paragraph 5016-b-1. See appendix for a statement of this code. Inasmuch as the current in the internal circuit is only that which is necessary to operate the relay and the test light, any spark formed would be extremely minute.

CHOICE OF DISPLACEMENT LIQUID MEDIUM

Probably 95 per cent of petroleum gas samples collected are extracted by means of connecting a container to the gas line in question, and after thoroughly purging the container with gas, the sample is introduced.

In some instances it is desired to collect the sample by allowing a container full of liquid to discharge its contents and allowing the gas sample to expand into the evacuated space. It has been long recognized that the solubility of natural gas in pure water is such that erroneous gas-sample compositions may be reported on samples collected with this medium. Consequently, a brine solution is usually employed. The concentration, for general oil field use, is most often reported as "a handful of salt in a jug of water."

The chief drawbacks of using brine solutions are itemized as (1) probable corrosiveness to sampling containers, (2) lack of sufficient freezing point depression at concentrations that might otherwise be suitable, and (3) danger of salting out of the solute with the contingent stoppage of small orifices should maximum solubility be exceeded.

In the initial trial run of the instrument the sampler was filled with water to expedite a mechanical check of the apparatus. It had previously been decided to use a 50 per cent glycerine solution as the permanent displacement medium. Connections were made for a field test which was to sample the inlet gas to a 50 pound natural gasoline absorber. After about two days of automatically sampling on a two-hourly schedule,

it was noticed that the instrument was discharging more liquid than calculations indicated. As time progressed, the discrepancy between calculated and observed sample volumes was getting progressively worse.

Extracted sample volume was measured by collecting the displaced liquid in a 100 cubic centimeter graduated cylinder.

The vent valve in the auxiliary tank was opened and a quantity of "gas" was discharged to atmosphere before the valve discharged liquid, which indicated that the small tank was then full of liquid. The next day, after the probable nature (hydrocarbon, water vapor, or air) of the gas had been argued to some length by the observers, the phenomena was again evident. A water-full gas burette was connected to the vent on top of the auxiliary tank and the entrapped gas was collected in the burette. Upon removing the burette to a non-hazardous location, the gas was swept out, with water, to the atmosphere and a lighted match was applied. The characteristic yellow flame indicated the presence of natural gasoline heavy ends.

The mechanism of the above occurrence is explained as:

1. Solution of the gas in the main sampling tank at 50 psi pressure.
2. Normal transport of this liquid to the auxiliary tank.
3. The gas boiling out of solution in the auxiliary tank each time that tank was reduced to atmospheric pressure (during a sampling cycle).
4. Collection of this gas in the top of the tank.
5. At each sampling period a higher-than-normal liquid displacement would result since the tank would begin discharging at sampling pressure rather than atmospheric

pressure since the tank was not full of liquid as it should have been.

An excess pressure would remain on the tank during its discharge cycle until enough liquid had been expelled to allow the entrapped gas to expand to atmospheric pressure.

Filling the system with the glycerin solution corrected the situation. Gas in the auxiliary tank has not been noticed after weeks of trial runs using the 50 per cent glycerin.

It is improbable that a perfectly suited liquid will be found to meet all the needs of the displacement medium in an instrument of this type. Glycerin stands up well to the two main qualifications; namely, chemical inertness and low freezing point. Other desirable characteristics would be low price and general availability. The viscosity index of glycerin would be a matter of concern, especially if sampling were to be conducted in an area that was subject to wide extremes in temperature. This is true because the volume of sample extraction will vary directly as the flow of displacement medium varies. Viscosity index becomes less significant as the solution strength is reduced, but conversely, weaker solutions would tend to allow hydrocarbon absorption by the displacement medium. This would cause unrepresentative samples to be stored. This is not to say that there is no suitable glycerin solution strength, but that optimum strength will depend upon (1) freezing point depression desired and (2) the composition of the hydrocarbon mixture in question; that is, a predominance of the heavier ends would require a stronger solution to resist a significant absorption by the displacement medium. Any detectable interchange that occurs between the gas sample and the

displacement medium would result in a gas sample analysis that is erroneous.

The viscosity of glycerin solutions at 25 degrees Centigrade is shown on Figure 4. The effect of temperatures on viscosity and the consequent rate of flow change of the displacement medium is the important consideration. A flow chart of the pipe line type is found on Page 379 of Perry's Chemical Engineer's Handbook, Third Edition. This shows that the effect of viscosity on the flow mechanism is only to the 0.16 power of viscosity, in centipoises with density having a similar, but inverse, effect on flow rate. This formula is ordinarily used for large flow rates and nothing should preclude its use on small flow rates. However, in small flow rates such factors as surface tension become important, especially in small bore piping and orifices.

It is thought that such reasons as this were responsible for lack of correlation between calculated flow rate change and observed flow rate change. The observed rates were obtained from an improvised efflux viscosimeter that was fashioned in the laboratory. This was done to observe the percentage-wise flow rate changes as a 50 per cent glycerin solution passed through a sharp edge orifice with the solution temperature made to vary from 120° F to 35° F. Other physical constants in this test were similar to those that would be experienced in the sampling instrument. A plot of the data is shown on Figure 5. The most important conclusion is the fact that the flow rate, with all factors constant except temperature, varied only about 4 per cent from summer to winter ambient temperatures.

Evidence of the lack of natural gas absorption in a 50 per cent glycerin solution is shown on Table 1. Two identical samples were held for one month in field containers with the volumes in the containers

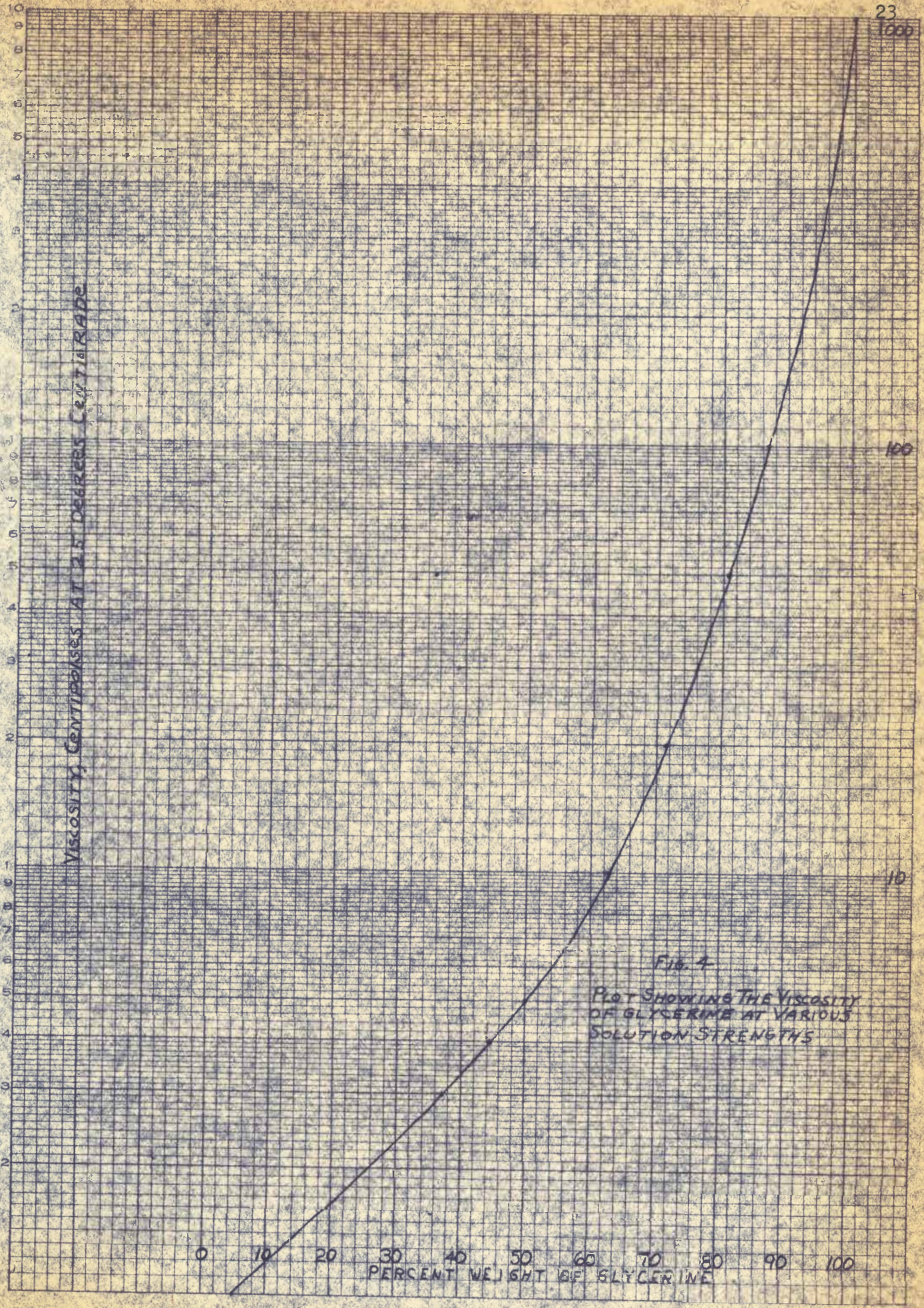


FIG. 4
 PLOT SHOWING THE VISCOSITY
 OF GLYCERINE AT VARIOUS
 SOLUTION STRENGTHS

23
1000

100

10

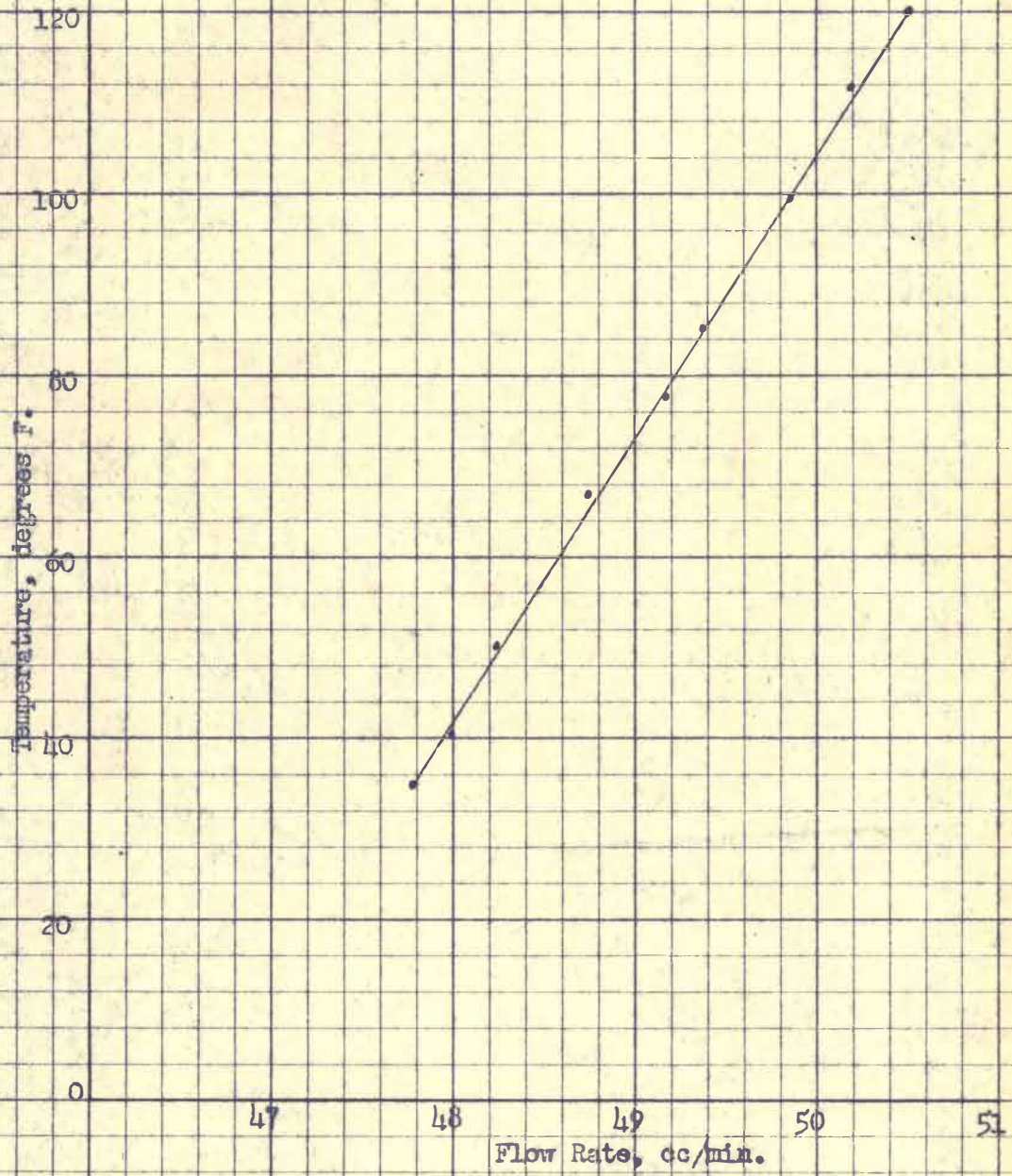


Figure 5

PLOT SHOWING THE EFFECT OF TEMPERATURE ON FLOW RATE OF A 50% GLYCERINE WATER SOLUTION.

Composition, Mol Per Cent

Component	Original Sample	Sample Stored at 75 to 80° F.	Sample Stored at Summer Outdoor Temp. 80 to 110° F.
H ₂ S / CO ₂	1.8	1.0	1.0
N ₂ / O ₂	3.5	4.1	3.9
C ₁	72.8	73.3	73.5
C ₂	11.0	10.6	10.9
C ₃	6.0	6.2	5.9
IC ₄	0.9	0.8	0.8
NC ₄	1.8	1.9	1.8
I-C ₅	0.6	0.6	0.7
N-C ₅	0.5	0.5	0.3
C ₆	0.7	0.5	0.6
C ₇	0.4	0.5	0.5

TABLE I

SOLUBILITY OF NATURAL GAS IN GLYCERIN-WATER SOLUTION

The gas used in this study had a calculated dew point of about 75° F. This may explain the difference in the heptane plus content of the original and test samples. The samples were transferred above 100° F., whereas the original sample was analyzed at about 75° F. There may have been some condensation at the lower temperature.

being roughly half liquid solution and half gas. At the end of the month, the samples were analyzed in a Podbielniak apparatus and compared to an analysis of the gas (by the same method) that had been previously made on a sample of the same gas prior to contact with the glycerin solution. The data show no loss of any particular component. Minor inconsistencies in the analysis are regarded as outside the limit of accuracy of the analytical apparatus. Since any settlement analysis, that would furnish the basis for the exchange of money between a buyer and seller, would be made with the same type apparatus it is practical to conclude that the glycerin solution will absorb no detectable amount of natural gas at the test conditions. The sample investigated was so rich in heavy components that the temperature of the tests almost fell below the dew point of the mixture. Thus, every opportunity was afforded the solution to absorb the gas.

Glycerin, of a quality suitable for this work, costs about \$3.50 per gallon. Although volume requirements may vary, it seems that a 9-1/2 gallon main container is well suited in size. Using a 50 per cent solution means the consumption of \$17 glycerin for an initial fill. With the use of the liquid reclaimer, shown in Figure 6, additional glycerin should be required only on rare occasions.

The possibility of a "dual" medium has been given some consideration. Here, a low specific gravity liquid, with good chemical characteristics, would be in contact with the gas sample and act as a buffer for a layer of higher specific gravity liquid, comprising the major portion of the displacement bulk. The basis for selection of the lower fluid would be constant flow rate with changing temperature.

INTERPRETATION OF PROPORTIONAL SAMPLING

Since proportional sampling is an integration of composition with volume and time, the attempt to correlate extended period composition analyses with actual production data will be fruitless if the sampling technique ignores this fact. The composite sample is a total of a series of instantaneous composition values. As such, it pre-supposes that flow volume in the header was held constant over the composite sampling period. Should this not be the case, then the composite sample is limited in worth since it tells only of the composition of a fixed rate of sample product withdrawal and is not a composite of the mass content through the system over the composite period.

To visualize the difference in the data gathered from the two methods, assume a case of conventional composite sampling with values exaggerated for simplicity.

Assume: A predicted flow of 100 per cent volume in a stream that is composed of 5 per cent of valuable component "A" and 95 per cent inert component "B"; a composite period of 30 days; that at the end of the first 15 days the flow changes from 100 per cent predicted to 105 per cent caused entirely by a volume increase in "A" which is now at the rate of 10 per cent. The stream is 100 per cent richer in valuable components (A) during the last 15 days of the month, but this fact would not be entirely represented in ordinary composite sampling procedures. Here, no extra sample amount would be taken to allow for the fact that though the composition over the last half month is twice as rich it is also

5 per cent more in volume. The material extracted for subsequent analysis would have the following respective "weight" in that analysis: 5 per cent of component A x 15 days x 100 per cent flow factor plus 10 per cent of component A x 15 days x 100 per cent flow factor, all to be divided by 2 for the monthly average. The truly representative sample would have 105 per cent as the flow factor for the latter 15-day period. The reported monetary worth of the gain in component A is reduced for the last 15-day period in the same percentage as the sample flow volume is in error, thus $100/105$ rather than the actual $105/105$.

The above figures are strictly arbitrary and were chosen only to show the method of examining the two cases. It is immediately seen that a flow proportional sampler may or may not be used advantageously. Factors that would indicate the installation of one (assuming that composition does vary) are:

1. Variation of flow, in amount and frequency, from some flow rate that may be considered a normal flow rate.
 2. Variation of the individual constituents of the product with particular emphasis on those constituents that have a special value.
 3. The probability of composition variation occurring simultaneously with flow variation. Should composition change at constant flow rate, then proportional sampling is not justified under normal circumstances.
 4. The degree of accuracy by which the composite must reflect the true mass-composition history of the flow stream.
 5. The probability that the analysis may later be questioned.
- It can be seen that should the initial and terminal end of

a pipe line be sampled by the two different methods and if the two analyses are not in agreement then the sample collected by means of the proposed sampling instrument, with other factors equal, could hardly be disputed since it is by theory, design and application an improved method. This fact is valid for all cases but becomes more significant if Factors 1, 2, and 3 above are in evidence.

APPLICATIONS

The instrument would find application in extracting a weighted average sample from a process stream with the subsequent analysis of the composite as being a representation of the true history of the stream, on a mass-composition basis, over the composite period.

There are six main categories of use that the instrument may fall into.

Case I. To sample each of two or more streams of varying composition and volume when the streams are physically combined at a downstream junction. Such a sampling technique would be the only method to allocate, for accounting and other purposes, the pro-rated origin of the input to the combined stream.

Case II. To sample a liquid stream of varying composition and volume that is fed to a large tank for storage and/or mixing purposes. The advantages of this case are two-fold. First, such a procedure would allow accounting records to reflect the actual contents of the storage tank for inventory purposes. Second, having this information at hand, blending specialists could use it in making calculations that would allow blends of more uniform character and with less chance for errors that would result in blending re-runs.

Case III. To sample the initial point of a pipeline delivery. The advantages of having accurate volume-composition records here would be in improved accounting and engineering efficiencies, and perhaps the elimination of some civil court suits.

Case IV. To use the device in plant material balance checks and plant trouble-shooting checks. Should a natural gasoline absorber, for instance, be suspected of sub-par operation then the inlet and outlet streams could be monitored with the instrument. Variation in process flow rates, with special reference to composition changes, would not be an indeterminate factor in the test since sampling would allow for this. Should a certain set of operating conditions, either natural or artificially imposed, prevail while an abnormally poor absorber operation is in evidence, then if the process conditions are changed to alleviate the poor situation it would not be known whether the observed results were due to the manipulated change or due to a lowered (or higher) flow condition. Sampling in this prescribed manner would not tell the source of the trouble, but it would put the flow volume in its proper perspective; it would yield a composite sample that was sensitive to flow irregularities.

Case V. The instrument should serve very well, with some modifications, as a flow-proportional injector. One possible application of this would be the injection of an odorant to a natural gas delivery line.

Case VI. The automatic feature of the instrument will guarantee to management that periodic samples of a definite volume rather than slugs were added to the composite volume. Reference is made to those instances where the worker forgets or is too busy to manually extract a sample. Too often the less experienced plant personnel labor under the misapprehension that a number of samples extracted at one time will save time and will be of no detectable consequence. Field laboratory analytical personnel and process engineers with their preponderance of accumulated data and with an intimate knowledge of locational and seasonal variations can, as often as not, spot the samples collected in this manner. Then

the only alternative is to use the analysis as it is reported or adjust the analysis to some more plausible figure. No statement is made here as to the general prevalence of this condition but it can be seen that the foregoing represents a possible case for an automatic procedure to remove entirely the human element.

LIQUID SAMPLING

If the specific gravity of a liquid is less than that of the displacement liquid in the sampler (a 50% glycerine-water solution has a specific gravity of 1.13), then sample extraction of that liquid can be conducted under the same procedures as described for gas sampling. Less attention can be given to pressure regulation since the pressure would have no effect on the amount of sampled liquid available to the laboratory for analysis. This is true due to the comparative incompressibility of liquids. Also, a much smaller volumetric amount of composite sample would be required for analysis purposes.

Should it be desired to convert a gas sampling installation to liquid service, the same main sample container can be used and the individual liquid sample amounts can be reduced by any one of the following methods, listed below in order of preference:

1. Decrease the elevation of the auxiliary tank with respect to the sampling orifice.
2. Increase the size of the header orifice plate, if possible, to make less meter differential. Here, since the root reading will be less, then sample per unit root will be less. Proportionality will be unaffected.
3. Decrease the dampening of the meter which will allow faster approach to displacement liquid valve shut-off. The time delay setting must be adjusted also.
4. Reduce the size of the sampling orifice; keep stoppage in mind.

A pressure reducing valve would serve no practical purpose except in cases where excessive main line pressures must be isolated from the sampler to prevent damage to the equipment. A check valve should be installed to prevent flow from the sampler to the process line. Fluctuating header pressures would have no effect on the quantity or quality of the sample. If a high pressure liquid line is sampled and the sampling pressure is reduced to protect the equipment, it must be remembered to hold an excess of the vapor pressure of the sample to prevent flashing. A great many hydrocarbon liquids are so volatile that if their pressure is reduced, as pressure drop across a valve, the liquid will vaporize.

VARIATION IN HEADER PRESSURE AS RELATED TO
DESIRED SAMPLE GATHERING PRESSURE

In general, a check valve or regulator should be installed between the sample source and the sampling tank to prevent the stored composite samples from flowing from the sampling tank back to the header in the event the header pressure fell below the pressure in the sampling tank. A block valve should also be installed to be used when the header is completely shut down.

It should be understood that for gas samples the required sampling tank pressure is only that which will insure sufficient standard condition sample volume for laboratory analysis.

Whether the sample should be admitted to the storage at a constant pressure or at the pressure of the header is determined by the character of the pressure control on the process. If the header experiences pressure variations but a pressure control valve smooths this to a constant value at the process, then to sample at the upstream header pressure would be technically incorrect. This is true because more standard condition volume, at unit flow meter reading, would be collected at times of high feeder pressure. The movement of material to the process would be at its normal level. The simple solution to this would be to have the sampling point at the process.

If the process is conducted at the same fluctuating pressure as the feeder, it would be desirable to store the samples at the various pressures so that the individual sample mass would be equivalent to the main stream flow mass. According to the above paragraph, this cannot be done.

The inability to integrate the individual sample pressure to the system pressure is not serious since the pressure fluctuations would generally be of a minor magnitude and the error would be only by the fractional amount that the absolute pressure varied from the setting of the sample pressure regulator. For instance, in a nominal 50 psig stream the pressure regulator may be set for 48 psig. A pound or two of pressure variation above or below this would result in no detectable error of the sample's proportionate volume.

Since liquid volume is not appreciably affected by pressure, the foregoing considerations do not apply to the sampling of liquids.

AN IMPROVED METHOD FOR TRIGGERING THE ELECTRIC CIRCUIT

Although an electrode immersed in the meter mercury is the ultimate in giving a precise indication of the near zero height, this simple mechanism cannot be used in some instances.

Consider the sampling of a liquid hydrocarbon of a specific gravity heavier than the di-electric oil. The sampled liquid would displace the oil from around the electrode and the safety precaution of the oil-quenched spark would be lost. The sampling of dry gases would not be affected by this feature, and the sampling of wet gas could be conducted, provided a satisfactory liquid trap is installed in the meter differential line.

However, since a number of probable applications of the instrument would be handling gases of high liquid hydrocarbon content plus free water with a comparatively high salt content, it would be desirable to eliminate the previously described electrode with its oil bath. A trace of salt water will change the transformer oil from a di-electric to a conductor.

The electrode can be eliminated if (1) the external electric circuit can be actuated at the same time as previously described and (2) the spark, that is formed when electrical contact is broken, occurs in a safe atmosphere. These requirements can be met with a mercury-in-glass switch.

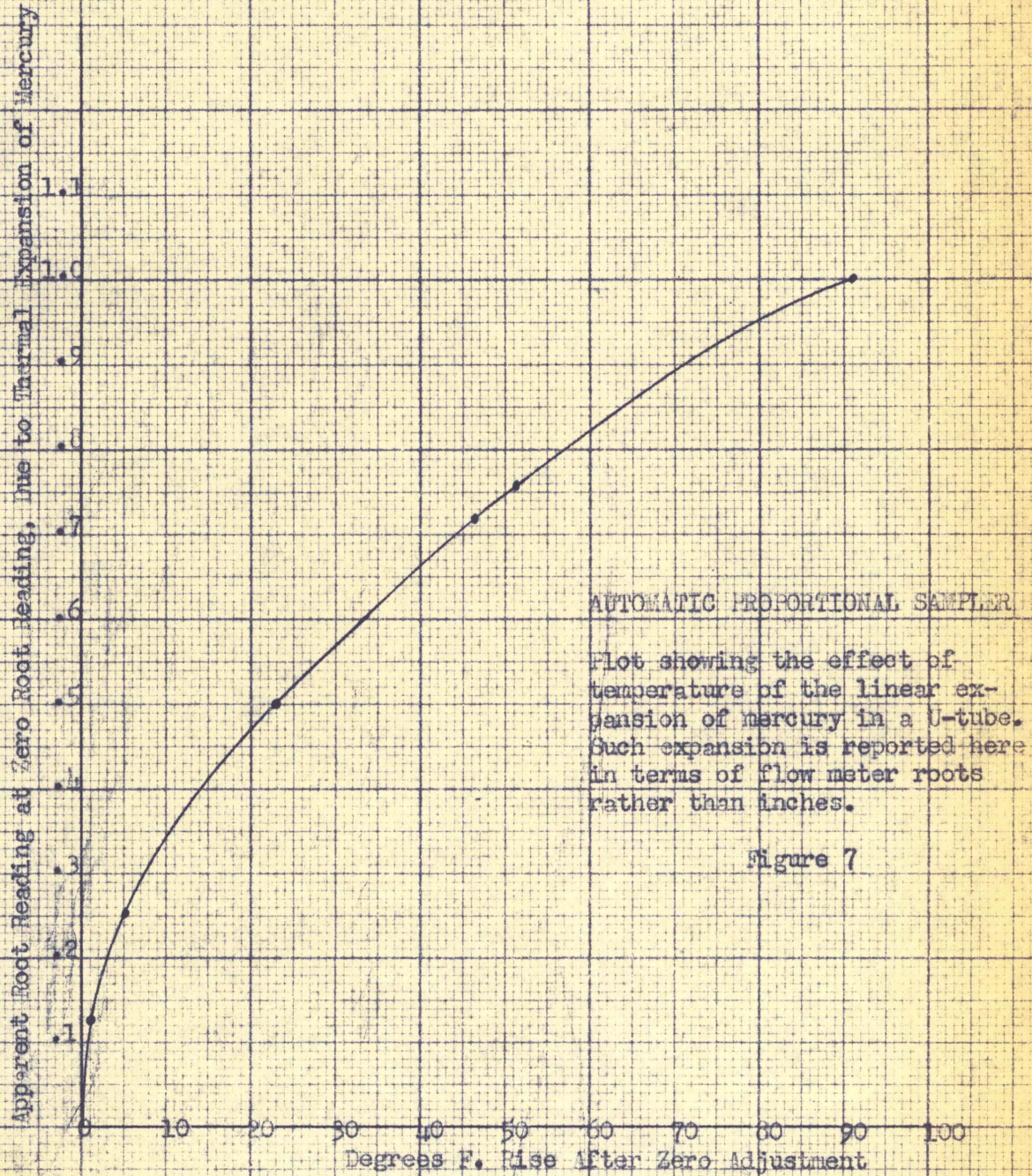
Nothing that has been previously described will have to be altered except that when the meter mercury reaches its near zero position the meter pen (or its linkage) should be made to tilt the switch to break contact, or, preferably, move a small permanent magnet that will separate

the tiny electrodes inside the glass switch. Such is a standard industrial application of these switches.

For the case where the proportionality of the sample must be held to very close tolerances, use of the electrode would be definitely more accurate due to no loss of sensitivity because of mechanical play, backlash, etc. However, this method would ordinarily give a degree of proportionality that would greatly exceed the limits of accuracy of other measured variables affecting the final results. The mercury-in-glass switch would be desired from an operational viewpoint; that is, adjustment, parts replacement, etc.

The manner of mounting and actuating this switch can be so varied that it will not be discussed here. It will suffice to say that the electrical circuitry would be as described previously except that the point of electrical detection would be moved from within the meter body to a point outside.

Using this switch will magnify the need of the delay switching apparatus. This is true because the zero position of the mercury will vary with temperature and also the mechanics of actuating the switch will require power at a time that the power source, which is the height of the mercury column, is essentially zero. It should be understood that the fraction of an inch variation of the meter mercury that is of so much concern here is of no consequence in the operation or the accuracy of an orifice meter. An inspection of the sample calculations and Figure 7 will show that the measured variable, the height of the mercury, is practically zero before the reported function, the volumetric flow in the process stream, has reached a significantly low value. Therefore, if the sampling cut-off point should be fixed at some mercury height that is near



zero, a sizeable error could result since the instrument would not be zero as far as flow measurement is concerned. The sample proportionality, over the entire meter range, would be in error by the amount of the volumetric reading at the time of sampling cut-off point. Thus, in a 100-inch water column meter 1 inch of meter differential can hardly be read on the chart and, in the meter body, is represented by approximately $1/12$ inch of mercury. However, this 1 inch of reading is one root, since the square root of "one" is "one", and as such is 10 per cent of the meter range, which was ten roots. If a sampling system measuring ten roots of flow should shut off at 1 inch of water differential, without the benefit of the time delay circuit, then 90 units of sample would be extracted rather than 100 units.

COST AND ECONOMIC CONSIDERATION

The cost of this instrument, installed, will be approximately the same as a plant temperature or pressure controller. The worth of the instrument can be examined from two separate viewpoints. First, as a device to improve the efficiency of using laboratory analytical data to make process plans, forecasts, or settlements. Second, as a labor saving device.

Regarding the first, if a certain sample can be proven to more closely approximate the true process conditions, then that sample, or its method of collection, is a definite improvement. Whether or not the installation of such an instrument, at a certain location, would be financially justified would depend largely on the value placed on the assurance that is given by the instrument. Sometimes proposed process ventures are held in abeyance due to mistrust of available analytical data. The discriminative application of this instrument would permit a new sort of dependability on the reported composition values of plant streams. Thus, there would be less risk in planning process work of any sort where stream composition were a critical feature. Oftentimes the success or failure of a proposed manufacturing project depends on a small percentage-wise composition change. Due to large volumes handled, the small percent becomes large in dollars. The singular purpose of this instrument is to report process compositions that are as truly representative as modern means will allow.

The instrument was not conceived to conserve labor, but to extract a

sample of a quality not ordinarily obtained by human manipulation. It has been estimated that \$144 per month of operator's time would be given to manually extracting a sample each two hours assuming twelve minutes' absence from productive labor per sample. However, the instrument would find rapid payout in those instances where field personnel frequently travel a considerable distance to take a sample of a stream that is under constant special surveillance.

The unit has been designed to function unattended for a period of one month. The monthly pick-up and re-charge of the sampling containers would be no more trouble or expense than that incurred in manual sampling. Although there are some novel features in the instrument's mechanism, there is nothing to indicate that maintenance would be greater than in an ordinary pneumatic process controller.

There have been numerous considerations as to whether the sampler meter and the recording differential meter should be one and the same piece of equipment. Original intentions were to use a U-shaped pipe as the mercury manometer and have no meter, per se, in the sampling apparatus. In that event, the incremental height of mercury in the field fabricated manometer would be as precise as in a factory built orifice meter. After such a device had been calibrated and proven at a field installation, an orifice meter, with reference to the sampler installation, would be surplus equipment. When it developed that the critical proportional detection as given by the mercury probe was not warranted in the general sampling case, thoughts were directed towards the use of a standard meter body with the mercury-in-glass shut-off switch. Since any process stream that would warrant the application of this sampling system

would, in all probability, have an existing orifice meter, then the expense of a meter for the sampling installation may be disregarded.

The reader is reminded of the periodic fall to zero that the meter pen would experience at each sampling period. Although flow measurement would not be actually affected, since the meter pen was falsely reading zero for a short time, industrial flow measurement experts are usually not in accord with any operation that adversely affects a continuous flow meter chart record. It may be speculated that for inter-departmental work this feature would be of no particular consequence but would probably be questioned in cases of inter-company measurement and sampling. In that event, one meter would serve the sampler and another meter, entirely isolated from the sampling installation but connected to a second set of orifice taps in the meter flange, would serve as the referee flow meter.

Ultimate use will depend on field procedure and preference.

The complete setting would be of a portability that would lend itself to ready disconnection from a stream and connection to another stream that might be deserving of two or three months' special composition study. Thus, in a year one instrument could serve a number of different points.

A list of material and cost summary is shown on Table II and Table III. It is estimated that an installation can be made for \$75 less than the figures shown if time were allocated to the redesign of certain items of special fabrication. At this writing only one production type model is in service. The usual cost reduction due to mass assembly would result in a further lowering of price.

Probe and mounting	\$ 30
1/4 inch 3-way solenoid (gas) valve	53
1/8 inch 3-way (liquid) valve	19
Pilot light and test switch assembly	30
Dome cover for relay box	11
Contents of relay box:	
Simple relay	2
Time delay	15
Selenium rectifier	2
Condenser	1
Micro switch	1
Timer motor	5
Teflon packing	6
Stainless steel float	4
Stainless steel float cage assembly	28
GUAC-47 junction box	4
Auxiliary and sampling tank	33
Displacement liquid tank	12
Tubing, fittings and valves	42
Frame and supports	25
Regulator	11
Filter	3
Electric wire and conduit	15
Main power switch	25
Seal pots	20
	<u>\$114</u>
Shop labor: (at \$4 per hour)	
Auxiliary tank, 8 hours	32
Frame and supports, 3 hours	12
Float cage fabrication, 2 hours	8
Probe unit fabrication, 4 hours	16
Assembly, 10 hours	40
Installation, 10 hours	40
	<u>\$148</u>
Parts:	\$114 (less meter)
Labor:	148
	<u>\$262</u>

TABLE II

AUTOMATIC PROPORTIONAL SAMPLER
LIST OF MATERIAL

Case I

Parts and labor	\$562
Standard orifice meter	200
	<u>\$762</u>

Case II

Parts and labor	\$493
Standard orifice meter with \$3 switch replacing probe arrangement	203
	<u>\$696</u>

Case III

Parts and labor	\$493
Standard meter body with switch, less meter case, clock and chart	110
	<u>\$603</u>

Case IV

Parts and labor	\$493
Mercoid switch installed in existing flow meter	3
	<u>\$496</u>

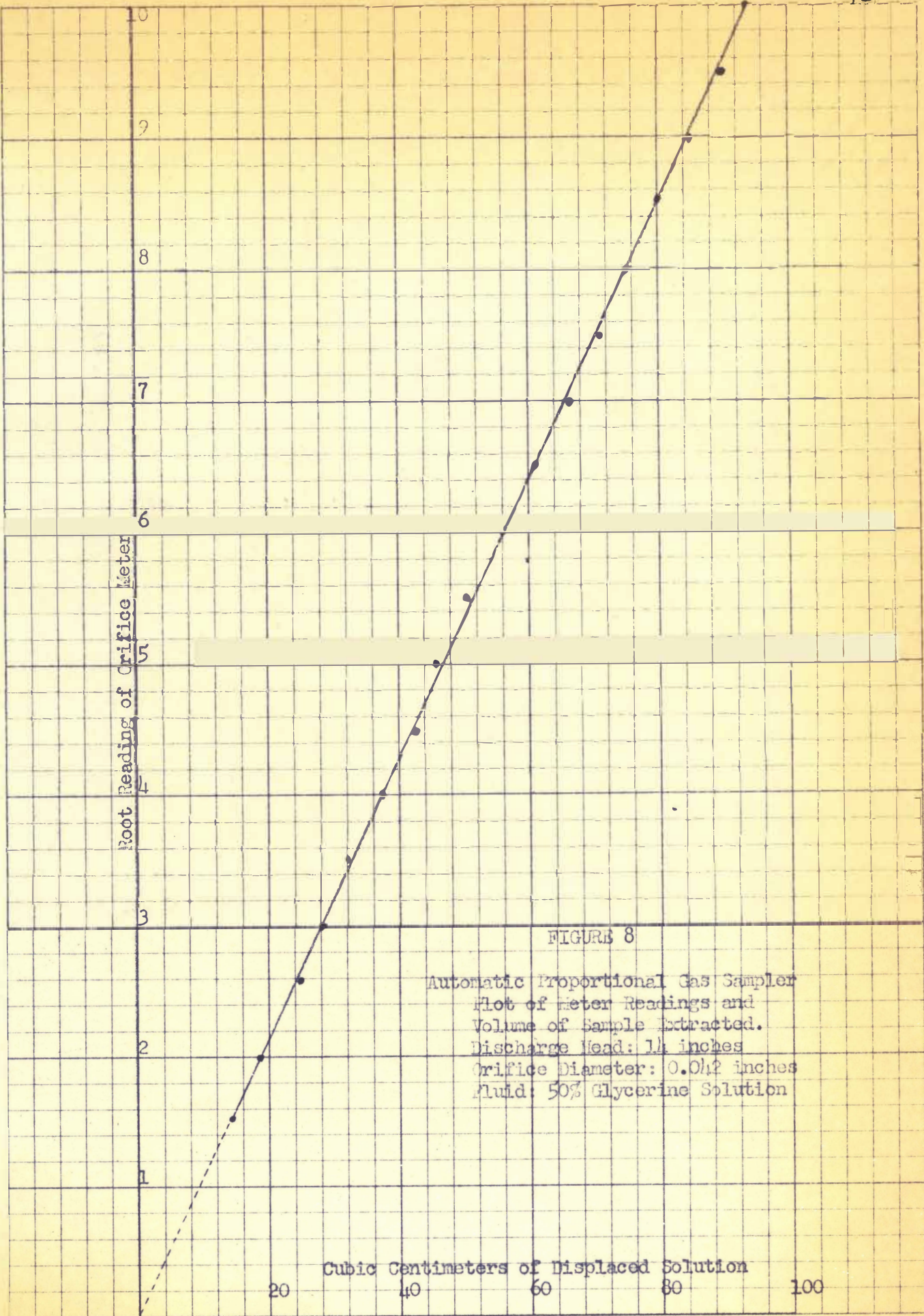
TABLE III

COST SUMMARY

DISCUSSION OF RESULTS

An inspection of Figure 8 shows the proportionality between meter reading and sample extraction. The extrapolation at the lower end of the plot is due to the delay time added to the sampling period and is to be regarded as a blind spot only when attempts to take a proportional sample are made when the meter reading is within this range (approximately 0 to 1-1/2 roots). The comparatively insignificant size of a sample in this range plus the unlikelyhood of operating here (it represents near shut-down flow conditions) would indicate that the blind spot constitutes no threat to the intent and purpose of the instrument. This contention would be supported by the fact that the sample or samples that would fail to be extracted due to this feature are probably of the same general composition as those that had been extracted. Furthermore, this dead zone can be made considerably shorter by moving the shut-off point to a lower root reading and making the equivalent change in the electrical delay circuit; however, as pointed out previously, to shorten the gap may subject the instrument to indeterminate operation. Admittedly, the margin of safety in this respect is very generous as the calibration presently stands, and the user may change the spacing to suit his needs.

Should the time delay function be omitted, but with the same pre-shutoff conditions existing, then a plot showing sample volume at various meter readings would show a straight line with its upper point the same as that shown on Figure 8 but the lower point would intercept the Y axis at a meter reading indicative of the actual shut-off point (here, about 1.5 roots). Proportionality between the X and Y axis would be lost. It can



be immediately recognized that for the amount of sample to be directly proportional to the main stream flow rate then a plot of this function must (1) be a straight line and (2) the straight line must go through the origin of the rectangular coordinates.

FIELD PERFORMANCE. The instrument has been under study in field operations for twenty months. This was for the purpose of mechanical checks and observation to avoid a premature integration of the instrument with process forecasts, etc. During this period, the instrument has sampled at the following points in a natural gasoline plant:

50 pound absorber feed stream	30 days continuous
600 pound absorber feed stream	30 days continuous
50 pound absorber feed stream	30 days continuous
Flash tank vapors	15 days continuous

Connections were made to other process points for periods of varying length.

No efforts have been made to observe any special analytical results due to the new method of sampling. The reason for this is present lack of comparative data that would disclose the fact of improved sampling at the points that had been selected for the tests.

Name: Royce Marvin White

Date of Degree: May, 1956

Institution: Oklahoma A. and M. College Location: Stillwater, Oklahoma

Title of Study: AN AUTOMATIC PROPORTIONAL SAMPLING INSTRUMENT

Pages in Study: 68

Candidate for Degree of Master of Science

Major Field: Chemical Engineering

Scope and Method of Study: The recognized failure of arbitrary volume samples to reflect the true flow stream history of a petroleum-type process line prompted this study. Flow-proportional sampling, when such is mandatory, is commonly done with a proportionating pump type of equipment but this is usually expensive, unwieldy and oftentimes not as accurate as the equipment would suggest.

A project was devised that was directed towards the design, applicability, fabrication and testing of an instrument that would gather and store flow proportional samples. Theoretical soundness was considered to be only the first requisite of the instrument; other factors were to be cost, ruggedness and freedom from human attendance or maintenance for comparatively long periods.

Findings and Conclusions: An automatic sampler was designed that made use of the fact that the height of mercury in an orifice meter is related to rate of flow. The mercury, being an electrical conductor, was made to close a switching mechanism at the beginning of a sampling cycle and end the sampling cycle when a volume of sample had been extracted that was directly proportional to main line rate of flow. The column of mercury in the meter body fell from its indicating height to zero height, due to a separate switching circuit, and the time of mercury fall was actually the function that determined the volume of sample extracted.

The instrument has performed on a test basis for almost two years. Its nominal cost, dependability, and apparent general merit exceeded the designers expectations.

ADVISER'S APPROVAL

Robert N. Maddox

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APPENDIX

APPENDIX A

DEFINITION OF TERMS

Bomb. Any small container that is used to collect or store petroleum products, usually for the purpose of analysis of the contents. These containers are often elliptical in shape and vary from 3-inch by 10-inch size to 12-inch by 36-inch.

Composite. A sample that is composed of many small samples. The large sample is considered to be an average, in composition, of all the small samples.

Grab sample. The opposite of composite. Usually obtained hurriedly and without much regard to factors that would contribute to the sample's being truly representative of its parent volume.

Head. Pressure expressed in terms of the elevation of the fluid whose height is causing the pressure.

Header. A large pipe whose contents are discharged to a number of small pipes.

Heavy components. Constituents of a petroleum product that have a comparatively high carbon content. In this paper, constituents of natural gasoline that have from five to eight carbon atoms per molecule; pentane through octane in the paraffin series.

Heavy ends. See heavy components.

"K" The orifice discharge coefficient. It is an efficiency factor that allows for the fact that a measurement of the loss of kinetic energy across an orifice plate will not give a practical measurement of the flow rate through the orifice plate. The formula Kinetic Energy = $V^2/2g$ theoretically will give the velocity of a fluid through a known orifice area, and a measurement of the loss in kinetic energy should be representative of the flow through that orifice. But the effective orifice area is smaller than the actual orifice area due to an orifice flow phenomenon. Thus, volumetric measurement, from a consideration of Volume = Area x Velocity, must be adjusted to allow for this phenomenon. The name vena contracta is given to this imaginary orifice and it occurs a short distance downstream from the real orifice. A correction, K, allows for this feature and also allows for departure from theoretical considerations due to loss in energy due to turbulence, etc. K may vary from 0.6 with a round, sharp edge orifice to 0.98 with a Venturi section.

Orifice taps. The holes provided in the meter flange for piping to the orifice meter.

Oxygen bottle. So named for its use as an oxygen storage container in high altitude aircraft during World War II. Actually, a thin walled metal tank, usually ellipsoidal in shape.

Proportionating pump. A device whose function is to move a small quantity of fluid in a certain ratio, usually 1:1, that is according to the flow rate of the pumped stream or another stream.

Root reading. The square root of the inches of water differential in an orifice meter. According to the formula $Q = c\sqrt{h}$ this would be a figure that is directly related to Q which is flow. See sample calculations.

Settlement. The agreement, particularly of methods of calculations, analysis, etc., upon which two parties will mutually arrive at decisions regarding credit for goods received. In this paper, settlement information is intended to be critical and exact as compared to information that is gathered for rough consideration only.

Settlement Analysis. That laboratory report which will furnish the basis for an important transfer of goods. It should be noted that settlements are often made between agencies within one company.

Slugs. Volumes of material that are added to a larger quantity in an indiscriminate and haphazard manner.

APPENDIX B

THE ORIFICE EQUATION

This equation will not be derived here but some of the factors that constitute it will be considered. Kinetic energy is expressed as $V^2/2g$ where

V = velocity in feet per second

g = gravitational acceleration

Thus, $KE \times 2g = V^2$, or $Velocity = \sqrt{Energy \times 2g}$. The work term, Pv , pressure times specific volume, may be replaced with h if h is considered to be head pressure loss, expressed in feet of flowing fluid. Thus $h_2 - h_1$ may be a measure of the work required to force fluid from Section 2 to Section 1 of a flow line. $Velocity = Volume/Area = C \sqrt{2gh}$, or at constant area, $Flow = C' \sqrt{2gh} = C'' \sqrt{h}$. This would be true for liquids where pressure had no appreciable effect on specific volume.

As mentioned previously, the velocity through the orifice bore is not the maximum velocity of the measured fluid. An adjustment must be made that will be compatible with the type of orifice used. Consequently, $V = .6 \sqrt{2gh}$ is commonly used for a round sharp edge orifice. Likewise, $Q/A = .6 \sqrt{2gh}$ is the flow formula in its simplest form. The form $Q = A \times .6 \times C''' \sqrt{2gh}$ is most often used, where C''' represents a great number of corrections in the order of 1.01 or .99 each.

APPENDIX C

SAMPLE CALCULATIONS

1. Calculation of K, the orifice constant, in the experimental apparatus was done by the method shown below. It should be realized that this method actually yields an apparent K since the discharge coefficient of the orifice, a strainer and some length of tubing are lumped as one figure. This was true because pressure drop (e.g., head loss) was taken as 14" H₂O upstream and one atmosphere downstream ($P_2 - P_1 = 14"$ H₂O) rather than measuring the pressure drop across the orifice proper. Since actual flow, rather than a study of K, was the object then this procedure is thereby permissible.

The formula: $GPM = 19.64 \times D^2 \times K \times \sqrt{h}$

where: D = diameter of orifice in inches

h = head at orifice in feet of fluid flowing

The data: from actual observation

$$GPM = 0.02565$$

$$D = 0.042 \text{ inches}$$

$$h = 14/12 \text{ feet}$$

$$K = \frac{0.02565}{19.64 \times (.042)^2 \times \sqrt{14/12}} = 0.685$$

2. To say that the experimental flow rate was calculated would be something of a misnomer since flow rate was observed in order to provide a basis for calculating K. However, knowing K, we can now calculate a flow rate for any set of experimental conditions. Thus,

$$GPM = 19.64 \times 0.001762 \times 0.685 \times \sqrt{14/12} = 0.02565$$

$$\text{cc/min.} = 0.02565 \text{ GPM} \times 3785 \text{ cc/gal.} = 97.2 \text{ cc/min.}$$

3. The monthly water consumption, at ten roots meter reading each sampling period, is calculated to be 9.52 gallons per month:

$$97.2 \text{ cc/min.} \times 12 \text{ samples/day} \times 31 \text{ days/month} \times 1/3785 \text{ cc/gal.} = 9.52 \text{ gal./mo.}$$

4. The minimum meter reading that will extract the required sample volume is calculated to be 5.25 roots:

Premise: 24 gallons of gas sample per month at 13.2 psia.

$$24 \text{ gal.} \times 3785 \text{ cc/gal.} = 90,900 \text{ cc/mo. at atmospheric pressure.}$$

$$90,900 \text{ cc at 13.2 psia} = X \text{ cc at 50 psig.}$$

$$X = \frac{13.2}{50 + 13.2} \times 90,900$$

$$X = 18,980 \text{ cc/mo. required at 50 psig.}$$

$$12 \text{ samples/day} \times 31 \text{ days/mo.} = 372 \text{ samples/mo.}$$

$$\frac{18,980 \text{ cc/mo.}}{372 \text{ samples/mo.}} = 51 \text{ cc/sample}$$

$$\frac{51 \text{ cc/sample}}{97.2 \text{ cc/min.}} = 0.525 \text{ minutes/sample}$$

By calibration, 10 roots = 1 minute. Then 0.525 min/sample = 5.25 roots/sample.

It should be remembered that this calculation was based on a sample gathering pressure of 50 psig. A higher pressure would cause the minimum meter reading (5.25 roots) to be lower.

An atmosphere of 13.2 psia is used in these calculations since it is actual local conditions for the experimental work.

5. The preparation of Figure 7, showing the effect of temperature on the sampler's zero calibration, was done in the following manner.

The temperature coefficient of expansion of mercury is 10.9×10^{-5}

inches per inch per degree F. A tabulation of meter roots, inches of water and inches of mercury is listed below for convenience.

Roots	Inches of Water	Inches of Mercury
10	100	7.96
9	81	6.45
8	64	5.10
7	49	3.90
6	36	2.87
5	25	1.98
4	16	1.27
3	9	.716
2	4	.319
1-1/2	2-1/4	.179
1	1	.0796
3/4	9/16	.0448
1/2	1/4	.0198
1/4	1/16	.0049

For example, the temperature necessary to expand the mercury surface from 0 to 1/2 root is calculated:

$$1/2 \text{ root} = 1/4 \text{ inch of water} = 0.0198 \text{ inch of mercury}$$

$$10.9 \times 10^{-5} \times 16 \text{ inches} \times t \text{ } ^\circ\text{F.} = 0.0198 \text{ inch of mercury}$$

$$t = 11.4 \text{ } ^\circ\text{F.}$$

Since only half of this expansion would take place in the U-tube leg that is under consideration, 11.4°F. x 2 would be the temperature necessary to increase the mercury level from zero to 0.0198 inches in each leg. Accordingly, a 22.8°F. temperature increase from reference temperature would raise the mercury level from 0 to 0.5 roots. Figure 7 shows that one such calculation was made at this point.

APPENDIX D

MATHEMATICAL PROOF

The following consideration will prove that gravitational flow of fluid through a small orifice will respond to the general theme of this paper; namely, an equation of the form

$$Q = K \sqrt{h} = K^1 t$$

where t = time of surface fall

K, K^1 = constants

Q = an amount of flow whose rate is being indicated by the height of fluid in this equation.

h = head, in feet of flowing fluid, of the fluid whose surface will fall from h to 0.

Specifically, the following will prove that the time of fluid surface fall is directly related to \sqrt{h} , and since \sqrt{h} has been shown to be directly related to Q then it will be concluded that the time of mercury fall, from h to 0, in a meter manometer is a precise measurement of Q .

Consider a cylinder of convenient dimensions that will roughly approximate those in the subject apparatus; that is, one foot high, one inch in diameter and with a 0.1 inch diameter orifice in the bottom.

The velocity of gravitational flow through such an orifice is given by $V = 0.6 \sqrt{2gh}$ feet per second.

In time dt the fluid that flows through the orifice generates a cylinder of height $V dt$ feet; a radius of $1/20$ inches and a volume of $\pi r^2 V dt$ cubic ft. Since V equals $0.6 \sqrt{2gh}$ then the volume may be stated $\pi r^2 \times 0.6 \sqrt{2gh}$ cubic feet at depth h and time t .

Let $-dh$ be the corresponding surface level at time t . This loss in volume is $-dh\pi r^2$ cubic feet. Volume may be equated to loss in volume, thus

$$.6\pi r^2 \sqrt{2gh} dt = -\pi dh r^2$$

$$.6 \times 0.0000173 \times 8.04 \sqrt{h} dt = -dh \times 0.00173$$

(note that one radius is 1/2 inch and one is 0.1/2 inch)

$$dt = \frac{-dh \times 0.00173}{4.824 \times 0.0000173 \times \sqrt{h}} = -20.7 \frac{dh}{\sqrt{h}}$$

$$dt = -20.7 h^{-\frac{1}{2}} dh$$

$$t = -41.4 \sqrt{h} + C$$

At $t = 0$ and $h = 1$, $C = 41.4$

At $h = 0$ (which is at the completion of one foot of fall) then $t = 41.4$ seconds.

GLYCERINE GRADES AND SPECIFICATIONS

U.S.P. glycerine (also referred to as C.P.-U.S.P.) is a high grade, water white product with taste and odor characteristics desirable for pharmaceutical or food uses. Its glycerol content is 95 per cent (specific gravity 1.249 at 25° C./25° C.).

C.P.-98 per cent or super C.P. glycerine is generally the same quality as U.S.P. but is offered at a higher concentration, typically 98.0 per cent glycerol (specific gravity 1.2602 at 15.5° C./15.5° C.).

High gravity glycerine is a common designation for a commercial grade of glycerine, close to white in color, and otherwise conforming to Federal Specification O-G-491 Grade B. This product also conforms to ASTM (tentative) specification 424c "High Gravity Glycerine for Alkyd Resins." It contains over 98.7 per cent glycerol. (Minimum specific gravity 1.2620 at 15.5° C./15.5° C.) or 1.2587 at 25/25° C.

Synthetic glycerine, a product derived from propylene, conforms to Federal Specifications for high gravity glycerine. It has been accepted for use in foods, but is not offered as U.S.P. Its glycerol concentration is 99 per cent.

Dynamite glycerine normally designates a pale yellow grade of glycerine meeting the particular specifications of explosives manufacturers such as DuPont, Hercules, Federal Specification O-G-491 (Grade B) and others. (Minimum specific gravity 1.2620 at 15.5° C./15.5° C. or 98.7 per cent glycerol).

Paragraph 5016-b-1 of the National Electric Code

5016-b. Class 1, Division 2. Switches, circuit-breakers, motor controllers and fuses in Class 1, Division 2 locations, shall conform to the following:

5016-b-1. Type Required. Circuit-breakers, motor controllers and switches intended to interrupt current in the normal performance of the functions for which they are installed shall be provided with enclosures approved for Class 1 locations, unless general purpose enclosures are provided and (1) the interruption of current occurs within a chamber hermetically sealed against the entrance of gases and vapors, or (2) the current interrupting contacts are oil-immersed and the device is approved for locations of this class and division.

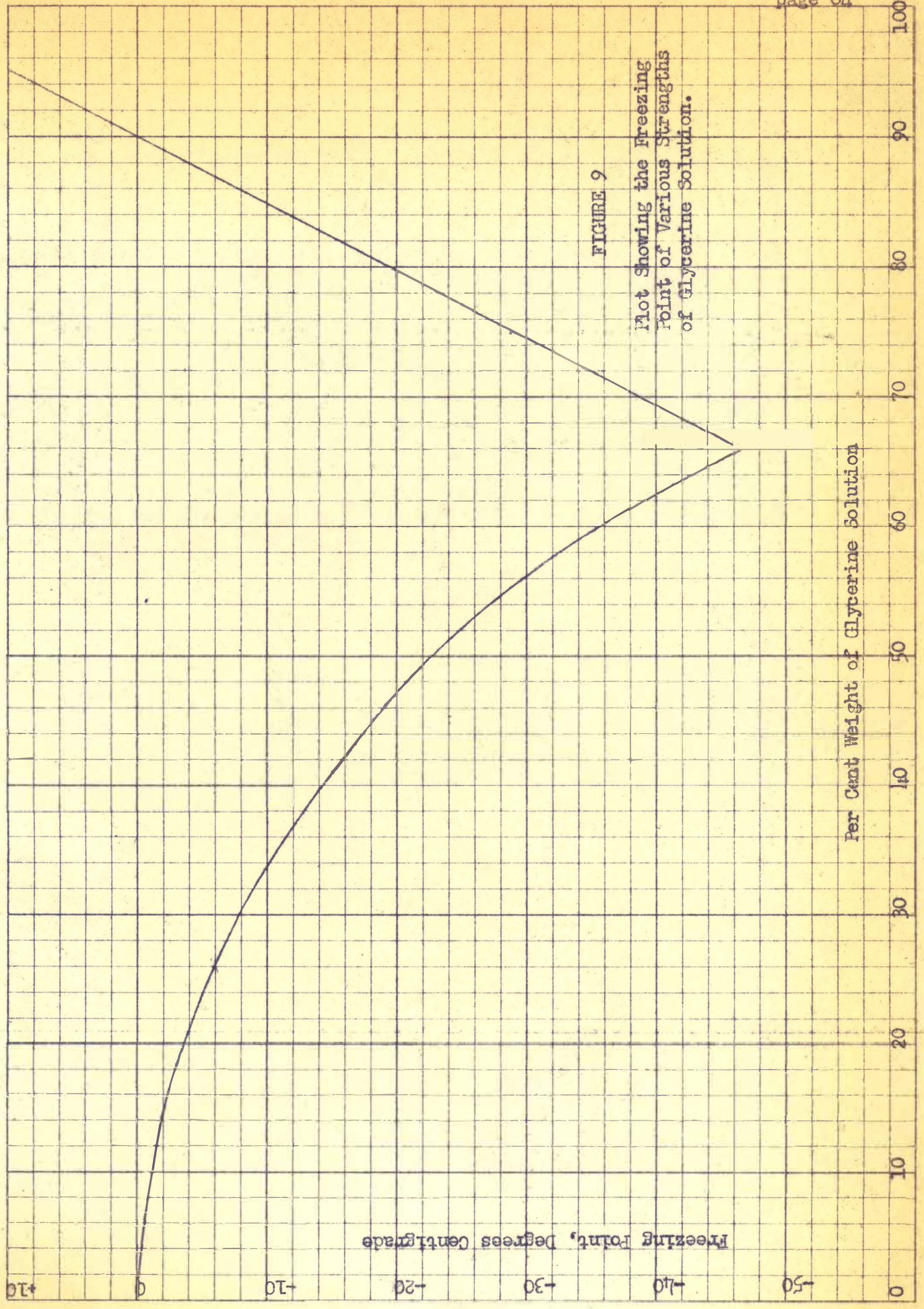


FIGURE 9
Plot Showing the Freezing
Point of Various Strengths
of Glycerine Solution.

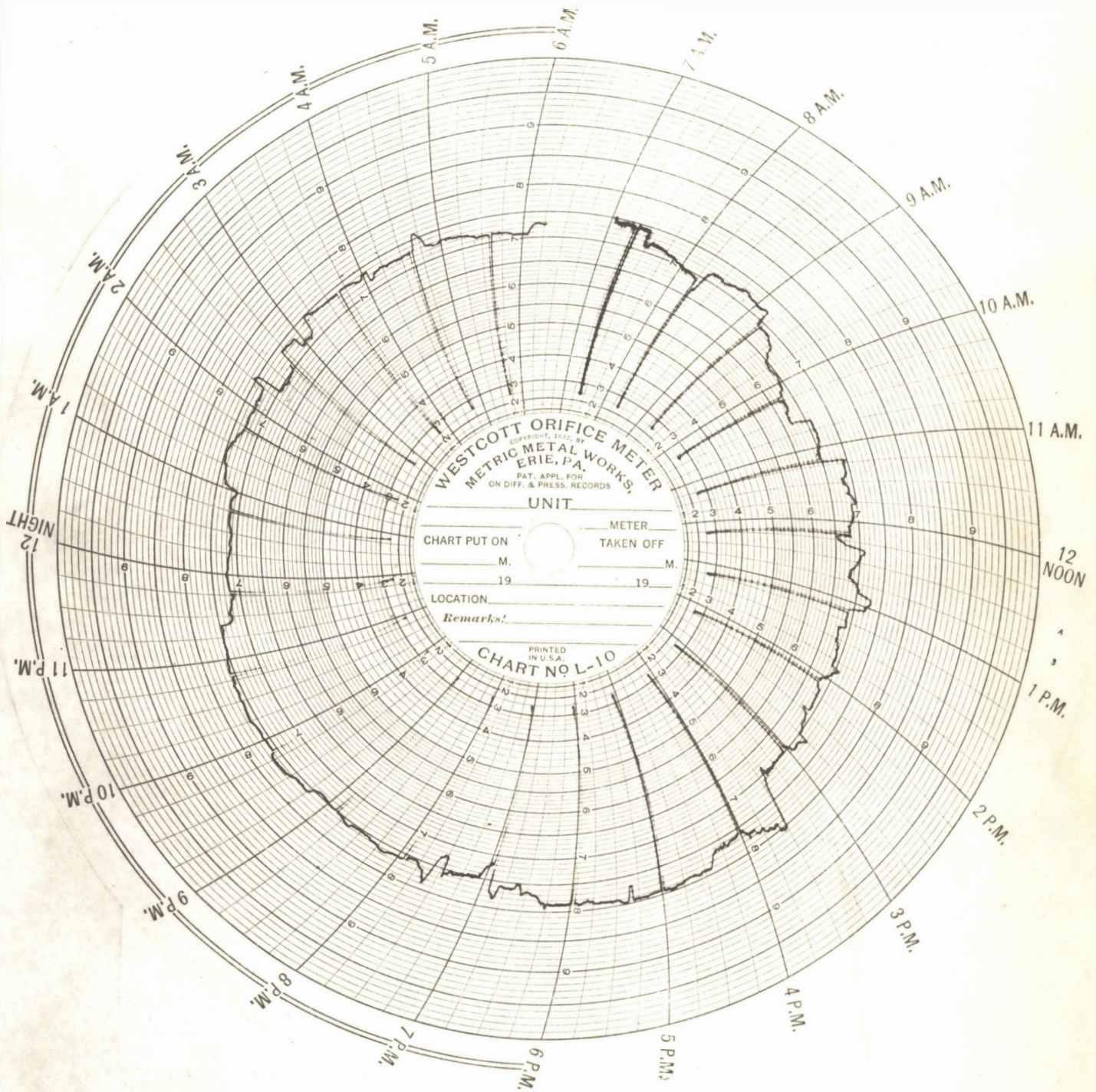


FIG. 10

METER CHART FROM SAMPLING INSTRUMENT

1/2" x 1 1/2" x 1/8" LS

LEGEND

Part No.	Description
1	Haydon Mfg. Co. Timer Motor 1/120 R.P.M. "L" rotation 115 V. 60 cycle Series 1600A with cam
2	Microswitch, Type W, BZ-RW13, 15 amp S.P.D.T.
3	K. W. Cramer Co. Time Delay Relay, Cat #TP2-1-20S 15 A, 110 V. switch.
4	Potter Brumfield, Relay, #KR11A, 115 V. 60 cycle D.P.D.T.
5	Federal, Selenium Rectifier, No. 1028A, 250 M.A.
6	Mallory, Condenser, Dual 10-10MFD, 450 V. Type FP-231
7	Automatic Switch Co., 3-Way Explosion-proof Solenoid Valve, Cat. #830225R for Gasoline Service, Max. Press. 150 psig, 3/16 Dia. Ports, 1/4 N.P.T. Connections, 115 V. 60 cycle Form "H".
8	Skinner Electric Valve Co. 3-Way Explosion-proof Solenoid Valve, Cat #X5-3100.
9	Crouse-Hinds Dome Cover, GUB714
10	" " Junction Box, GUAC-47
11	" " Pushbutton & Pilot Light #EFDC3473-J1
12	Cinch Jones Terminal Strip #8-140
13	W. H. Nicholson & Co., Spherical S.S. Float, 3" Dia. x 18 Ga. 1/8" N.P.T. Style 1 Connection. Not for liquid sampling.
14	Linear, 1820-5 "O" ring.
15	2 Tanks, 2100 Cu. In. Capacity.
16	Block Valve, 1/4" Metric
17	Check Valve, 1/4" Parker

FIG. 11

CONSTRUCTIONAL FEATURES

AUTOMATIC PROPORTIONAL SAMPLER

VITA

Royce Marvin White
candidate for the degree of
Master of Science

Thesis: AN AUTOMATIC PROPORTIONAL SAMPLING INSTRUMENT

Major: Chemical Engineering

Biographical and Other Items:

Born: August 5, 1920, at Helena, Arkansas

Undergraduate Study: Mississippi State College, 1946-47;
Louisiana State University, 1947-51; University of Tulsa
(night classes), 1952

Graduate Study: Oklahoma Agricultural and Mechanical College
(night and Saturday classes), 1953-55

Experiences: Steam power plant fireman-operator, 1940-42; U.S.
Army Signal Corps, 1942-45, Commissioned Officer, overseas
duty with Headquarters of Southwest Pacific Area; Municipal
water plant operator-chemist, City of Columbus, Mississippi,
1947; Employment by Phillips Petroleum Company as Engineer,
in various capacities, February, 1951, to date.

Member of American Institute of Chemical Engineers, Instrument Society
of America.

Date of Final Examination: May, 1956

THESIS TITLE: AN AUTOMATIC PROPORTIONAL SAMPLING INSTRUMENT

AUTHOR: Royce Marvin White

THESIS ADVISER: Dr. Robert N. Maddox

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TYPIST: Jeane Williams