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A Dynamic Gas-Mixing System

By C. R. Carpenter, J. E. Chilton, and G. H. Schnakenberg, Jr.



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ði	UNIT OF MEASURE A	ABBREVIATIONS USED	IN THIS REPORT
A	ampere	ml	milliliter
° C	degree Celsius	ml/min	milliliter per minute
cm	centimeter	msec	millisecond
ft	foot	μΑ	microampere
hr	hour	μF	microfarad
Hz	hertz	μm	micrometer
in	inch	μs	microsecond
in H	g inch of mercury	μμΕ	micromicrofarad
k k	kilohm	Ω	ohm
К	kelvin	pct	percent
kHz	kilohertz	ppm	part per million
1/mi	n liter per minute	psi	pound per square inch
М	megohm	sec	second
mb	millibar	V	volt
MHz	megahertz	vol-pct	volume-percent
min	minute	edecation consector	-1, Time base generation, and entry 1-1. Control and getting logic se

A DYNAMIC GAS-MIXING SYSTEM

By C. R. Carpenter, 1 J. E. Chilton, 2 and G. H. Schnakenberg, Jr. 3

ABSTRACT

A dynamic gas-mixing system assembled by the Bureau of Mines for the generation of precise gas mixtures from sources of concentrated or pure gases and diluent gases is described. A set of electronic mass flow controllers with maximum flows ranging from 10 to 5,000 ml/min, a socalled Pure Air Generator, and gases in cylinders are used to generate differing gas concentrations. The repeatability of the delivery rate of a flow controller, measured on different days, has a precision of 0.3 pct of the setting. This dynamic gas-mixing system reduces the cylinder inventory and the demurrage charges for special gas mixtures. Gas mixtures that cannot be shipped commercially, such as flammable mixtures of methane in air, can be conveniently prepared by this system. The maximum dilution ratio is 2.5×10^4 . Thus, gas mixtures can be made over a wide range of concentrations, from the percent region (by dilution of a pure single-component gas) to fractional parts per million (by dilution of a premixed standard, e.g., a 1,000-ppm mixture). This system is especially useful in determining the response of gas detection devices over the entire range of their measurement. Because the controllers are voltage controlled they lend themselves easily to automated control using computer-based systems.

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INTRODUCTION

There are many applications for gas detector devices for personal safety and property protection in the mining industry. The devices are used to detect the presence and concentration of (1) toxic gases (e.g., hydrogen sulfide), (2) gases that are precursors of fire (e.g., carbon monoxide), (3) gases that may accumulate to form explosive mixtures (e.g., methane), and (4) life-threatening atmospheres (e.g., deficiency of oxygen). These gas detectors must be sensitive over a wide range of concentrations to selected species of gas. The performance of each type of detector should be evaluated prior to its general use to insure its adequacy and suitability.

The evaluation of gas instrumentation designed to detect low concentrations (parts per million) of CO, NO, NO₂, SO₂, and $\rm H_2S$ and high concentrations (volume-percent) of $\rm O_2$, $\rm CH_4$, and $\rm CO_2$ for use in mining environments requires gas mixtures of high accuracy for both instrument calibration and instrument performance evaluation. The performance evaluation of gas instrumentation will use well-characterized and accurately determined gas calibration mixtures for tests

including those for accuracy-over-range, drift, and precision.

Calibration gases may be purchased in cylinders from commercial sources but not without several disadvantages: high initial cost; uncertainty in accuracy of analysis; slow, unpredictable delivery; a finite cylinder capacity; and extensive demurrage charges incurred in the storage of a large number of reference gas mixtures in cylinders. Certain mixtures of gases may not be available because of their flammability. One solution to this problem of obtaining different concentrations of a gas is to dilute a highconcentration gas accurately to a lower A dynamic gas dilution concentration. system involves the mixing of two gas streams to produce a predetermined specific concentration of a gas in a flowing gas sample.

This report describes the design and fabrication of a dynamic gas dilution system that satisfies the demands of gas instrument evaluation test methods that require a flowing source of several accurately known gas mixtures.

BACKGROUND

The Bureau of Mines instrumentation group at the Pittsburgh Research Center has used various methods in its attempts to design and build a universal dynamic gas-mixing system. One system employed precision needle valves to control the flows of the test gas and the diluent gas. The flow rates were monitored with high-quality Labcrest⁴,⁵ rotameters.

Although this method was exclusively used for several years, it had several deficiencies. The main deficiency was its imprecision or lack of resetability;

each and every flow setting required verification using a soap bubble meter to obtain desired mixing accuracies (±1 pct or less error). Another deficiency was its lack of long-term stability. One source of this instability was the variation in the regulation of pressure from the cylinder gases. At high dilution ratios, a small change in input gas pressure of the minor component gas caused a large change in the diluted gas concentration.

In an effort to improve our testing facilities, we experimented with restrictors. We found the usable range of flow rates of each resistor to be limited owing to their nonlinearity. Additionally, the instability problem was not significantly improved even though

⁴Use of trade names is for identification only and does not imply endorsement by the Bureau of Mines.

⁵Suppliers' addresses are listed in appendix A.

additional pressure regulators were placed in the input lines.

Our next major effort was centered on the use of electronic mass flow controllers (Tylan model 261). Preliminary tests on the devices indicated that they would be very suitable in our application. We had several reasons for selecting electronic mass flow controllers (MFC):

- 1. As flow controllers, the mass flow rate would be independent of the pressure upstream of the controller.
- 2. The mass flow could be set by applying a voltage between 0.1 and 5 V, and voltages of sufficient stability could be easily produced. Furthermore, the flow rate would be approximately proportional to the control voltage over the range of the controller.
- 3. The controller provided an output signal of 0.1 to 5 V, also approximately proportional to the mass flow of the gas. This signal could be accurately read by a

digital panel meter, and it is also equal to the flow rate control signal when control of the flow was attained by the controller.

- 4. The manufacturer claimed a precision (resetability) of 0.2 pct of full scale, a precision which we thought necessary for our gas detection instrument investigations and evaluations.
- 5. Once a calibration curve was established for a particular controller for one gas, a simple application of the gas laws and well-known physical properties of gases could be applied to this curve to produce a calibration curve for other gases or for other ambient pressures and temperatures.
- 6. Since flows are voltage controlled, these controllers could be easily used in an automated testing facility should this be needed. The following sections describe first the design concepts and rationale and then the full system design and performance of our dynamic gas dilution system using mass flow controllers.

SYSTEM DESIGN

BASIC CONCEPTS

The purpose of this gas-mixing system is to dilute a high concentration of test gas species to a more appropriate lower concentration for use in gas instrument testing, thus conserving gas and permitting a variety of concentrations from one gas cylinder. The inputs to the system are the one or more test gas species to be diluted (source gas) and the diluting gas (diluent). The output is the diluted gas mixture.

Because the output concentration is determined by flow rates, they become the critical elements in the system design. Figure 1 depicts the basic elements of the gas dilution system involving two components (source and diluent). The flow controllers are adjusted by the operator to produce the desired flow ratio and, therefore, the concentration of the blended gases. After mixing, the gas

is conducted by suitable connectors to the system output. Figure 2 emphasizes the flow control and mixing elements of the system.

The flow controllers control the flow rates of the gas in response to a command (set point) voltage. In our systems, this voltage, between 0.1 and 5 V, is obtained from a front panel control that is set by the operator (fig. 3). A voltage proportional to the flow rate is developed by the flow controller. This voltage, which, as previously mentioned, is equal to the command voltage when the flow is stabilized, is displayed by a digital panel meter (DPM). As a refinement, since each controller has a DPM dedicated to it and since each covers different flow ranges, we have adjusted the calibration of each meter to provide a display that is approximately numerically equal to the flow in milliliters per minute. Since it is necessary for

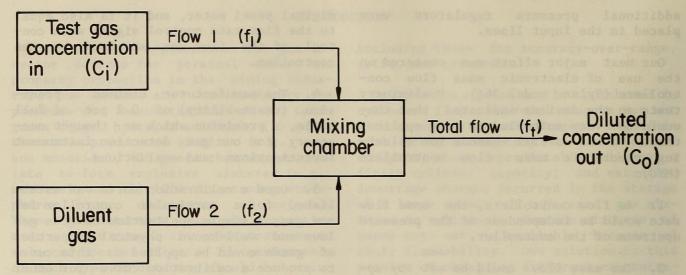


FIGURE 1. - Basic elements of the gas dilution system: source of a minor component at concentration C_i ; flowing at rate f_1 ; a diluent gas, e.g., air flowing at rate f_2 ; and a mixing element. The final concentration of the mix is $C_i \times f_1/f_1$ flowing at a rate of $f_1 = f_1 + f_2$.

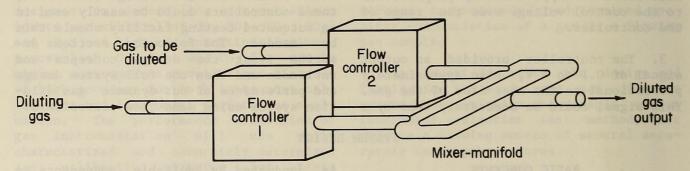


FIGURE 2. - Flow control and mixing elements of the gas dilution system.

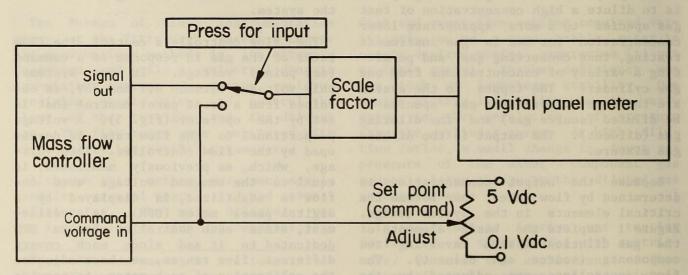
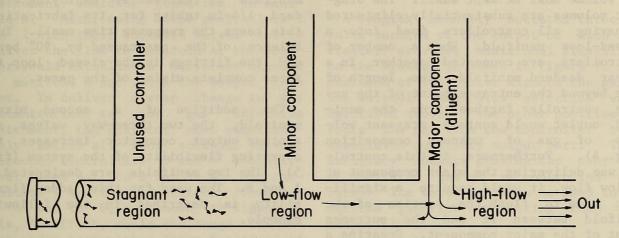


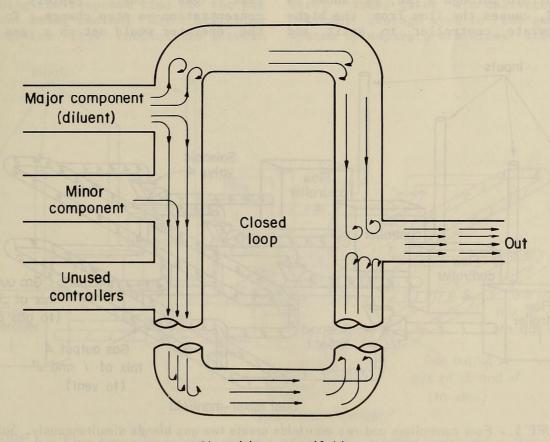
FIGURE 3. - Electrical elements of the flow system. Each mass flow controller has associated with it a power supply (not shown) and a command signal to be set to the desired value by the user. A digital panel meter is appropriately scaled to read flow rate directly in milliliter per minute and is used to display either the command signal to the controller or (normally) the flow rate signal output from the controller.

the operator to see the flow setting command voltage to set the particular controller, this voltage can be displayed on the same digital display, using a momentary contact pushbutton switch.

The output flows of the controllers should be mixed thoroughly and be available with a minimum response (lag) time. To accomplish thorough mixing, the



Linear manifold



Closed-loop manifold

FIGURE 4. - Linear and loop mixing manifold patterns.

manifold into which the controllers deliver the gases should have no stagnant areas that could contain unmixed gases that would slowly diffuse into the mixture. To minimize response time, the total volume must be kept small. The stagnant volumes are substantially eliminated by having all controllers feed into a closed-loop manifold. When a number of controllers are connected together in a linear deadend manifold, the length of tube beyond the entrance point of the accontroller farthest from the manioutlet would contain stagnant volumes of gas of unknown composition Furthermore, if this control-(fig. 4). ler was delivering the minor component at a low flow, it would require a significant time to flush the section of the manifold between it and the entrance point of the major component. Creating a loop manifold by connecting the ends of a linear manifold to each other and exiting the manifold through a tee, as shown in figure 2, causes the flow from the highest flowrate controller to split and

sweep the entire manifold at relatively high flows, picking up and mixing with the minor component in the process.

An internal volume of about 2 ml in the manifold is realized by employing standard 1/4-in tubing for its fabrication; this keeps the response time small. Turbulence of the gas caused by 90° bends and the fittings in the closed loop insures complete mixing of the gases.

The addition of a second mixer-manifold, the two three-way valves, and another output connector increases the operating flexibility of the system (fig. 5). The two manifolds are designated as A and B. The need for this added flexibility is described in the following example.

Suppose the operator wanted to challenge a gas detection instrument with a test gas that rapidly changes concentration—a step change. To do this the operator would set up a gas mixture

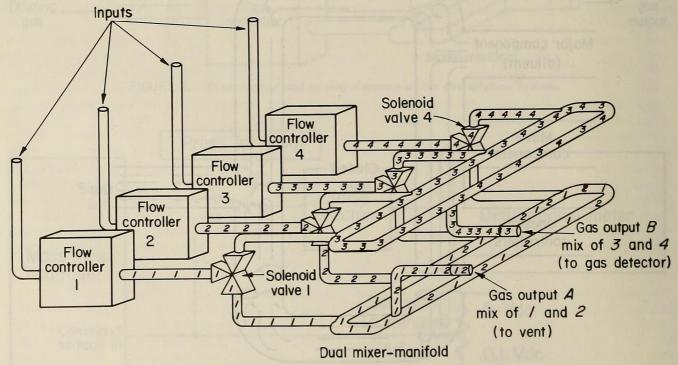


FIGURE 5. - Four controllers and two manifolds create two gas blends simultaneously. Solenoid valves for controllers 1 and 2 direct the gases into manifold A, with controller 1 having the larger of the two flows. Similarly controllers 3 and 4 feed manifold B, with controller 3 having the greater flow. Compare output blends with those in figure 6.

in manifold B using the flow controllers 3 and 4 in figure 5. The solenoid valves, 3 and 4, associated with these controllers would be set to deliver the flows to manifold B. The gas detection instrument would be connected to manifold B. Next, using flow controllers 1 and 2 with their solenoid valves set to deliver the flow to manifold A, the operator would establish a different mixture in manifold A and route the output to a vent. To deliver a step change in gas concentration to the gas detection instrument, the operator must simultaneously switch all four solenoid valves, whereupon controllers 3 and 4 would deliver flows not to B but to A, and controllers 1 and 2 would deliver flows not to A but to B, as shown in figure 6. This, in effect, would challenge the device under test with a "step function" of This "step function," the test gas. among other things, would be useful in determining the response time of the gas instrument being tested. Rapid switching of all of the flow controllers between manifolds A and B is assured by using three-way electrical solenoid valves at the output of each flow controller. Simultaneous switching of all valves is accomplished using a single switch.

A Swaglok manually operated three-way valve located at the output of each controller provides additional flexibility to the system, as shown in figure 7. One position of this valve directs the gas to the solenoid manifolds selection valve described above. The other position directs the gas from the controller to an associated auxiliary output port. These auxiliary outputs provide the user with an undiluted gas at a controlled flow rate. The valve may also be used to cut off the gas flow by setting them to a midposition.

The source gases and the nitrogen diluent gas are usually obtained from cylinders. These gases are delivered from a cylinder storage area to the gas-mixing system by separate stainless steel tubes

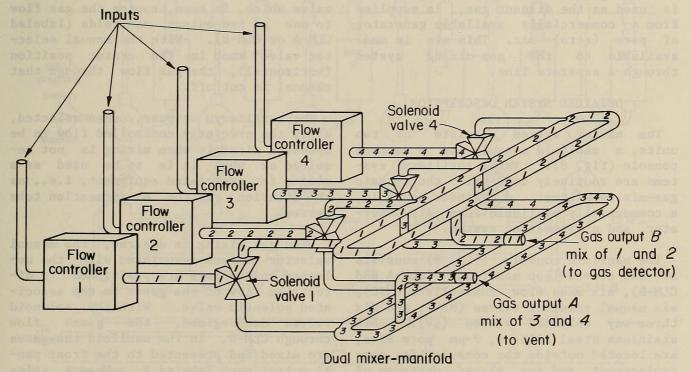


FIGURE 6. - Flow controller solenoid valves simultaneously actuated (energized or deenergized depending on original state) to direct flow to alternate manifold. Thus, at manifold A the gas mix rapidly changed from a blend of 1 and 2 to a blend of 3 and 4; similarly the output from B changed from a blend of 3 and 4 to a blend of 1 and 2. Compare with figure 5.

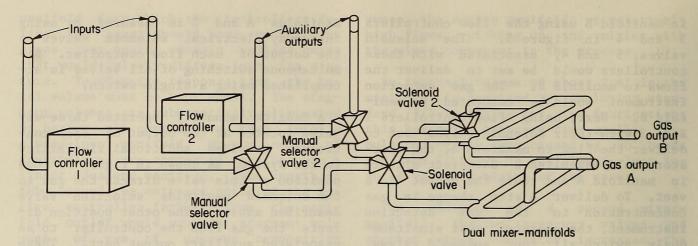


FIGURE 7. - System with manually operated three-way valves.

from each cylinder. The pressure is regulated to the operating input pressure range (10 to 40 psi differential) of the controllers. Some gases, such as NO2, H2S, and SO2, are conveniently obtained from a permeation tube system. gases are normally diluted in a fume hood with the diluent gas controlled by the gas-mixing system; this is an ideal use of the auxiliary outputs. Air, when it is used as the diluent gas, is supplied from a commercially available generator of pure (zero) air. This air is made available to the gas-mixing system through a separate line.

DETAILED SYSTEM DESCRIPTION

The mixing system consists of two units, a manifold console and a control console (fig. 8). Three auxiliary systems are routinely used with the dynamic gas-mixing system: a cylinder manifold, a commercially available Pure Air Generator, and a permeation system.

The manifold console (fig. 9) consists of two closed-loop manifolds (CLM-A and CLM-B), six mass flow controllers (MFC), six manual selector valves (MSV), and six three-way solenoid valves (SV). Six stainless steel filters, 7-µm pore size, are located outside the console for easy replacement and are placed upstream of the MFC to prevent aerosols from entering the system.

The gases to be mixed are connected to the system by compression seal bulkhead fittings on the top of the console, with one corresponding to each controller. Each gas flows through the filter and then directly to a mass flow controller. Immediately downstream of each controller is a manual selector valve that is used to route the gas to either an auxiliary output port or an associated solenoid valve which, in turn, routes the gas flow to one of two output manifolds (labeled CLM-A or CLM-B). With the manual selector valve knob in the center position (horizontal), the gas flow through that channel is cut off.

The auxiliary outputs, when selected, allow the precisely controlled flow to be accessed directly when mixing is not required or when it is to be used as a source for associated equipment, i.e., as the dilution gas for a permeation tube system.

When gas mixing is desired, the manual selector valves associated with the selected controllers are positioned to direct the flow of the gases to the associated solenoid valve. With the solenoid valves deenergized, the gases through CLM-B. In the manifold the gases are mixed and presented to the front panel output port labeled B. When a solenoid valve is energized, the controlled flow from its associated controller

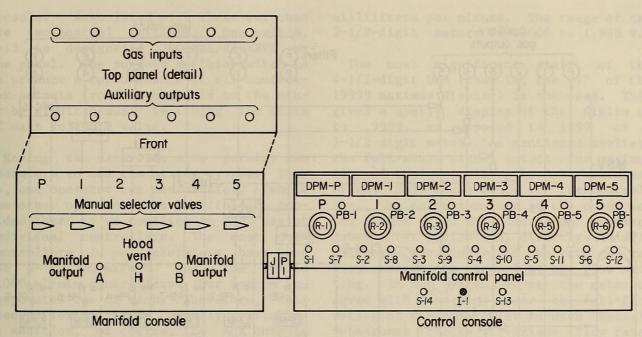


FIGURE 8. - Location of controls and connections for the manifold console and the control console.

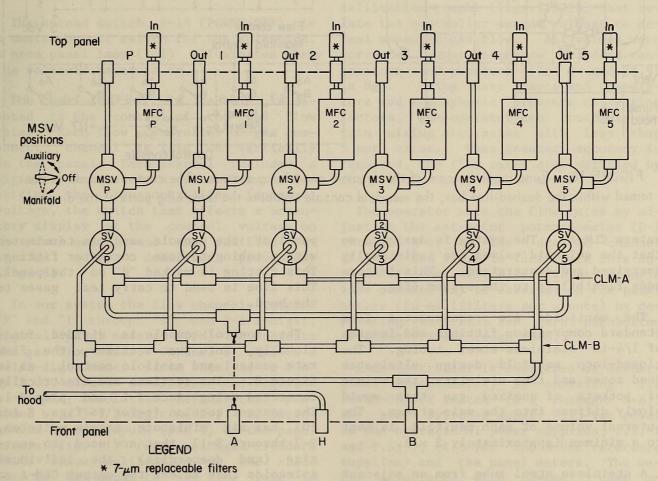


FIGURE 9. - Parts identification and functional layout of the manifold console.

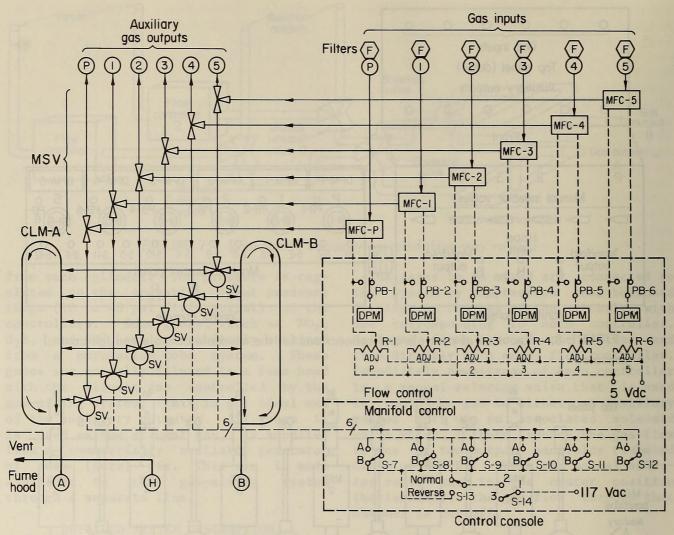


FIGURE 10. - Functional diagram of the dynamic gas-mixing system. The control console is contained within the dashed-line box; the manifold console contains the remaining parts of the diagram.

enters CLM-A. The system is designed so that the solenoid valves are individually energized and deenergized. This feature adds flexibility to the system (fig. 10).

The manifolds are constructed from standard compression fittings and lengths of 1/4-in stainless steel tubing. The closed-loop manifold design eliminates dead zones and thus minimizes the volume of pockets of unmixed gas that would slowly diffuse into the main stream. The internal volume of each manifold is kept to a minimum (approximately 2 ml).

A stainless steel tube from an adjacent fume hood is brought out to the front

panel of the console and is terminated with a tubing-to-hose connector fitting. This fitting is marked "H" on the panel. This line is used to carry test gases to the hood.

The control console is divided, functionally, into two sections, the flow rate control and manifold control, as in figure 8. The sections are electrically connected using jack J-l and plug P-l. The control section (refer to figs. 8 and 10) has six miniature toggle switches, S-7 through S-12, that are used to energize (and deenergize) the individual solenoids that select the output CLM-A or CLM-B for each controller in the manifold

console. Associated with these switches are two control switches. One switch, S-13, is designated "NORMAL/RESERVE" on the panel. The purpose of this switch is to produce a rapid change of all controller outputs from one manifold to the other by simultaneously switching the state of each solenoid valve.

Having two manifolds also permits two operators to use the mixing system. That is, one operator can use manifold A while the other operator uses manifold B, provided there is no conflict in the use of individual controllers. The most used controllers in our laboratory have been the 4- to 200-m1/min the 40- to and 2,000-m1/min controllers. Our system incorporates two of each. Therefore, this needed flexibility is usually available. In addition, of course, the AUX outputs can be used simultaneously and blended external to the system.

The second switch, S-14 (POWER/OFF), is a master on-off switch for the solenoids. A neon panel lamp (I-1) illuminates when the solenoid power is on.

The remainder of this console is devoted to the control of the gas flow rates. Each flow controller or gas component "channel" is laid out vertically on the panel. Each channel includes a digital panel meter, a potentiometer for setting the flow-controlling (command) voltage, the switch that effects a momentary display of the control voltage on the panel meter, and the manifold selection switch described previously (fig. 8).

In our system the flow channels labeled "P" and "1" through "4" use 3-1/2-digit, digital panel meters (DPM) to indicate the gas flow rates. Channel 5 employs a 4-1/2-digit meter since this is a 100-to 5,000-m1/min controller and requires a display resolution beyond that provided by a 3-1/2-digit meter. Potentiometers at the inputs (pin 1) of the panel meters (fig. 11) form voltage dividers which allow calibration of the readouts to be equal to the flow rate in

milliliters per minute. The range of the 3-1/2-digit meters is 0.000 to 1.999 V.

The most significant digit of the 4-1/2-digit DPM (that is, the "1" of the 19999 maximum display) is not used. This gives a usable display of the digits up 9999, as opposed to 1999 3-1/2-digit meter. As mentioned earlier, the full-scale signal output for maximum flow of the controllers is 5 V regardless of the full-scale range of the controller. The potentiometers at the panel meter inputs allow adjustment for an exact panel meter indication, directly in milliliters per minute, of the rate of gas flow through the channel at one flow setting. In our system, we set the meter to agree with the flow near the full-flow rating of the controller in each channel. Subsequent checks at various flow rates within the range of each controller were performed and used to develop a set of calibration graphs (figs. 13-18) that relate the controller output voltage to actual measured gas flow. All values were corrected to our reference standard temperature and pressure (25° C and 28.92 in Hg). Using these graphs and temperature and atmospheric pressure correction factors, the operator can routinely obtain mixing accuracies with less than 5 pct error. When greater accuracy is required, the flow rates are verified by the use of a soap-bubble meter.

The operator sets the flow rates by adjusting the set-point potentiometer (R-1 through R-6) in the desired channel (figs. 8 and 11). To facilitate this adjustment, the controller input signals (set points) may be observed on the panel meters (in milliliters per minute) by depressing the PRESS FOR INPUT pushbutton switches (PB-1 through PB-6).

Each controller is energized or deener-gized independently by operating its POWER-ON/OFF switch (S-1 through S-6). The power switches supply 115-Vac power to the operational power supplies, P.S.-2 and P.S.-1, (±15-Vdc and 5-Vdc reference supplies) and the panel meters. The meter associated with channel 5 uses an

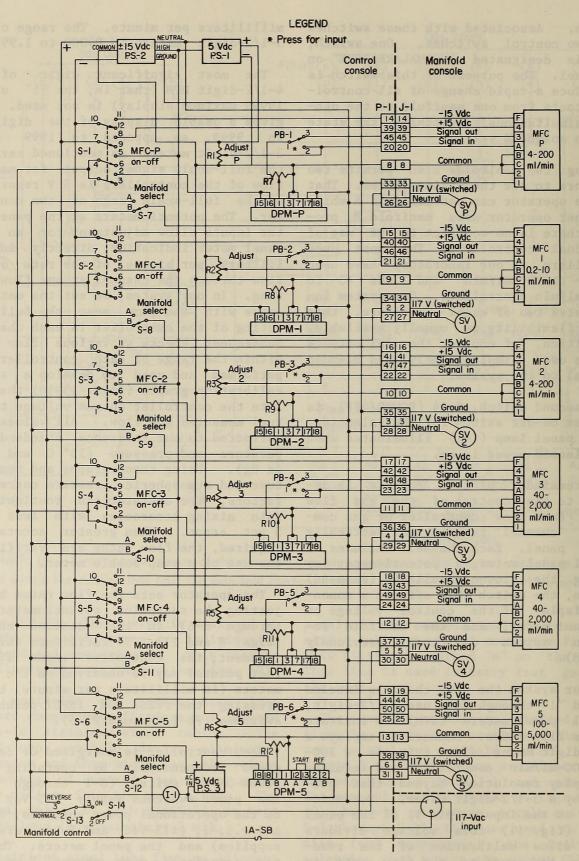


FIGURE 11. - Electrical schematic of the gas-mixing system.

external 5-Vdc (P.S.-3) supply; the other meters are powered directly from the switched 115-Vac line. The output from the 5-V reference supply is permanently connected to the clockwise end of all six set-point (command) potentiometers. The ±15 Vdc is connected to the individual controllers through separate poles of the power switches. Thus only the controllers in use are powered, saving wear and tear on any unused controllers.

MASS FLOW CONTROLLER DESCRIPTION

The electronic mass flow controllers used in this system accurately and reliably measure and control the mass flow of gases. The principles of heat transfer along a capillary tube are used to develop a linear output signal of 0.1 to 5 V over a selected flow range. The controllers incorporate a valve and appropriate electronics to automatically regulate flow rates in response to an external command signal (0.1 to 5 V).

The valve (fig. 12), made of 316 stainless steel, is a unique thermal expansion design that eliminates friction and moving seals. It is a small thin-walled tube with a ball welded to the end. The seat is a cone. Inside the tube is a heater wire that causes the tube to expand relative to the outer shell, moving the ball relative to its seat and thereby varying the flow.

The sensor section consists of a small, stainless steel capillary with external

resistance temperature sensors and heaters. This is shown in figure 12. A more complete explanation can be found in the Tylan manual.

FLOW RATE CALIBRATION

Calibration of the system was performed using zero air produced by a Pure Air Generator, a soap-bubble flowmeter set, an electronic timer, an electronic digital thermometer, and an aneroid barometer.

The Pure Air Generator, according to the manufacturer (AADCO), produces air with less than 0.005 ppm hydrocarbons, carbon monoxide, carbon dioxide, methane, ozone, sulfur, hydrogen sulfide, and oxides of nitrogen but with 22.5 pct 02 in nitrogen. A methane reactor accessory, mounted within the cabinet, is a low-temperature catalytic oxidizer which removes all combustible hydrocarbons, including methane. The air compressor for this unit is capable of supplying up to 10 1/min of zero-air and is mounted in a housing to reduce noise to an acceptable level.

The Teledyne soap-bubble flowmeter set contains a set of three glass tubes of a calibrated volume and traceable to the National Bureau of Standards (NBS). Each tube has a different volume (10, 100, and 1,000 ml) and fits in a glass base which contains a soap solution such as those available for detecting gas leaks. (We use SNOOP.) The gas is directed into the

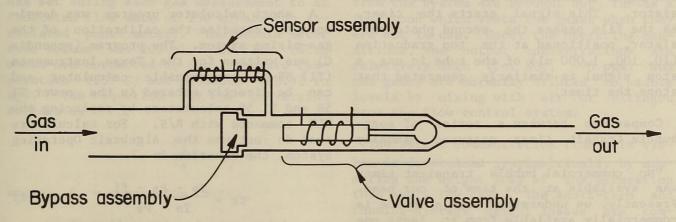


FIGURE 12. - Functional representation of the electronic mass flow controller sensor and valve.

glass base and passes out through the graduated tube. A squeeze bulb, fitted on the base, is used to raise the level of the solution in the base to block the entrance to the graduated tube. When the bulb is released, the liquid level drops; a film of the solution remains across the flow passage and moves up the graduated tube as it is pushed by the flowing gas. The flow rate is determined by measuring the transit time for the film to pass through a given volume. That is,

flow rate =
$$\frac{\text{volume (m1)}}{\text{transit time (min)}}$$
.

Corrections for temperature, atmospheric pressure, and gas composition if other than air are then applied to obtain the mass flow rate (i.e., a volume flow rate referenced to a given pressure and temperature). Because our laboratory is slightly greater than 1,000 ft above sea level, and operating equipment keeps it warm, we standardize at 28.92 in Hg (979.3 mbar) and 25° C.

To improve precision and eliminate the random operator errors in measuring bubble transit times, we developed a digital timer (appendix B) for which the start and stop signals are obtained from two phototransistors which detect the passage of the moving film (bubble) that is illuminated from above.6 The first phototransistor is positioned at the bottom or zero graduation on the tube. As the edge of the film passes, it acts as a "light pipe" that momentarily increases the light transmission to the phototransistor. This signal starts the timer. As the film passes the second phototransistor, positioned at the top graduation (10, 100, 1,000 ml) of the tube in use, a stop signal is similarly generated that stops the timer.

Comparison between a series of soapbubble transit times measured manually and those simultaneously measured by the phototransistor-timer verified the accuracy of the phototransistor separation and the increased precision obtainable with the timer.

For a particular measurement a tube is selected to provide a sufficient transit time to insure adequate resolution at the flow rate being measured.

The timer, designed and constructed specifically for this job, incorporates other features that allow its use as a general purpose laboratory timer. A block diagram and schematics for the timer are included in this paper as appendix B. For use with the soap-bubble flowmeters, the 0- to 10-min range is normally selected. This gives a resolution of +0.0001 min for times up to 9.9999 min.

For gas temperature measurements, we use an electronic digital thermometer from Stow Laboratories. The platinum resistance sensor is attached to the graduated tube of the flowmeter to measure, indirectly, the ambient temperature, and hence, the temperature of the gas. The digital display on this unit provides a resolution of +0.1° C.

Barometric pressure is measured on either an aneroid barometer or an electronic meter; the aneroid barometer is an analog device, and resolution of 0.01 in Hg is obtainable. The Serta Systems electronic meter resolves to 0.1 mbar.

A short calculator program was developed to expedite the calibration of the gas-mixing system. The program (appendix C) was written for the Texas Instruments (TI) SR-52 programmable calculator and can be directly entered in the newer TI 58 and TI 59 calculators by replacing the HLT commands with R/S. For calculators that do not use the Algebraic Operating System, the equation is

$$fc = \frac{Ts \cdot Pa \cdot fi}{Ta \cdot Ps}, \quad (1)$$

⁶No commercial bubble transient timer was available at the time of our need. Presently, we understand such a device is commercially available from at least one source (Mast).

where fc = corrected flow rate, ml/min,

Ts = standardized temperature (25° C + 273.15), K,

Pa = actual barometric pressure, Hg or mbar,

Ta = actual temperature (° C + 273.15), K,

Ps = standardized barometric pressure (28.92 in Hg or 979.3 mbar),

and fi = indicated flow rate, ml/min.

The permeation of the gas through the soap bubble did not cause any significant error in our flow measurements with transit times of less than 2 min.

The flow controllers, as received, were calibrated for air by the manufacturer. One must use a correction factor to use the controllers with gases that have different molecular structure. In their Operation and Service Manual, Tylan Corp. provided a table of correction factors for various species of gas. We conducted experiments in the laboratory to determine the corrections required for methane, carbon monoxide, and carbon dioxide. The values obtained were in very close agreement with the values published in the Tylan table.

Typical examples of this correlation are shown in table 1. Flow controller 2 was set during each gas measurement to an indicated flow rate of 150 ml/min (on associated panel meter). The bubble meter tube volume was 100 ml. The flow rates shown are corrected to our standard temperature (25° C) and pressure (28.92 in Hg). The equation for the conversion factors is

$$C_f = \frac{f_g}{f_a}$$

where $C_f = conversion factor,$

fa = flow rate of air, ml/min,

and $f_g = flow rate of other gas,$ ml/min.

TABLE 1. - Flow conversion factors

10000	Manufacturer's	Experimental	Measured
Gas	conversion	conversion	flow
000	factor	factor	rate,
10 3	the design gos	almo tersion a	ml/min
Air	1.00	1.00	147.1
CH ₄	.72	.72	105.9
co	1.00	.99	146.1
CO ₂	.74	.74	108.9

Most of the test gases used in our testing programs are obtained from cylinders that are located remotely from the dynamic gas-mixing system. We have found it undesirable to store some gases in cylinders (NO_2 SO_2 , H_2S , etc.) because of reaction with the cylinder walls (unstable concentration) or toxicity of the gas. For generating mixtures of these gases, we use a permeation tube system.

Our permeation system uses a Forma Scientific water bath in which the temperature is held constant at 20°±0.1° C by an integral refrigeration and heating system. Two Pyrex glass permeation tube holders (U-tubes) are immersed in the water bath. This allows us to generate two species of test gas simultaneously. We use 10-cm-long permeation tubes, which gives an active tube life of more than 12 weeks at the operating temperature. Dry nitrogen is used as the carrier gas for these tubes at a flow rate of 100 ml/min for each tube holder. The two outputs from the system are brought out inside a fume hood which vents the gases when they are not in use. Under the operating conditions, the output concentrations are relatively high (for NO_2 , ~ 75 ppm) and the gases are normally diluted to usable levels by mixing with air or nitrogen from the flow control system.

The output concentration from this system is determined gravimetrically by measuring the weight loss of the permeation tube every few days. The short-term and running-average weight losses are computed, recorded, and graphed.

SYSTEM OPERATION AND PERFORMANCE

DILUTION RATIOS

The concentrations of test gas required by the instrument evaluation laboratory range from high percentage to low parts per million levels, a range of 1,000,000 to 1. As a compromise the design goal of a dilution ratio of 1:1,000 was chosen. This will produce a test gas concentration of 0.1 pct (1,000 ppm) from a source of pure gas or, if a cylinder of accuknown premixed gas (e.g., a 1,000-ppm concentration) is used as the source, low-parts-per-million gas concentration levels can be achieved (i.e., 1,000 ppm/1,000 = 1 ppm). Of course, intermediate concentrations can be obtained by adjusting the dilution ratio; therefore, any test gas concentration between 1 ppm and 100 pct is available for our gas detection device evaluation efforts. This satisfies our flexibility requirements and reduces the cylinder gas inventory to a maximum of two cylinders for each species of gas required for testing. In our experience gas detection devices require flows of up to approximately 1 1/min. To test several devices from a common source, flows up to 5 1/min might be required. Therefore, for 1:1,000 dilution ratio, we selected four flow rate ranges for our system controllers to cover this dilution range at the desired output flow rates: 0.2 to 10, 4 to 200, 40 to 2,000, and 100 to 5,000 m1/min. These ranges allow the total output flow rates to be adjusted, as required for testing, between 200 ml/min and 5 1/min at the maximum dilution ratio. The maximum dilution ratio is thus 0.2 to 5,000, or 1 to 25,000.

FLOW SETTING REPRODUCIBILITY

The reproducibility of setting the gas flow of the Tylan mass flow controller was calculated by setting a fixed flow value on a controller and periodically measuring the resulting gas flow on different days over a period of several weeks. Since the measurements were made on different days and thus at different ambient temperatures and barometric

pressures, the daily average flow readings were corrected from measured temperature and pressure to our chosen reference temperature and pressure of 25° C and 28.92 in Hg (979.3 mbar). This value of the pressure corresponds to normal atmospheric pressure 29.92 in Hg or (1,013.2 mbar) corrected for a 1,000-ft altitude above mean sea level (laboratory elevation). A soap-bubble meter was used to measure the flows. The mean of 10 values of bubble transit time was taken for each flow determination. The grand average flow for a setting of 604 on the digital panel meter corresponded to 661.19 ml/min when corrected to the reference temperature and pressure. The standard deviation of the flows was 1.95 ml/min. The reproducibility of the flow measurement and setting (the coefficient of variation) is 0.3 pct of reading; the measured flow values are summarized in table 2.

TABLE 2. - Reproducibility of a flow setting, ml/min

Measured	Standard	Flow corrected to
flow	deviation	reference temper-
		ature and pressure
661.08	2.29	660.65
660.99	1.64	659.12
658.72	1.30	660.57
657.07	.32	660.22
671.42	1.86	664.77
667.33	2.82	661.82

FLOW CONTROLLER LINEARITY

We performed our own check of the linearity of each mass flow controller. The control voltage of each mass flow controller was set at selected values, and the corresponding gas flows were measured using 10-, 100-, or 1,000-ml soap-bubble tubes as appropriate, as described earlier. The flow values and the calibrated digital panel meter readings are summarized in figures 13 through 18. The flows were corrected to a flow at our reference temperature of 25° C and pressure of 28.92 in Hg (979.3 mbar). A least squares analysis of the data was

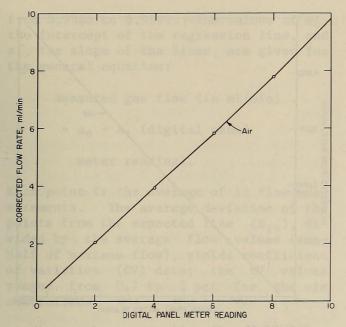


FIGURE 13. - The calibration curve for controller 1 is the measured controller flow rate corrected to 25°C, 28.89 in Hg versus scaled digital panel meter reading of controller output voltage (command signal).

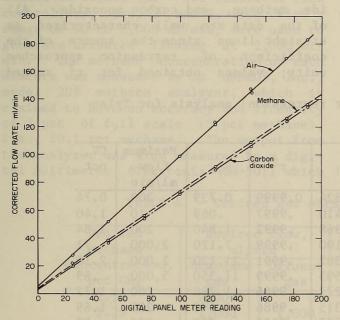


FIGURE 15. - The calibration curve for controller 2 is the measured controller flow rate corrected to 25°C, 28.89 in Hg versus scaled digital panel meter reading of controller output voltage (command signal).

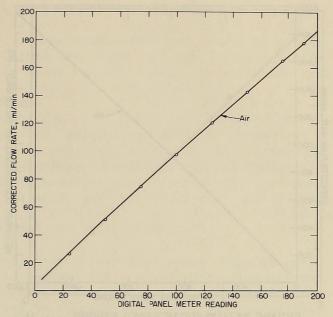


FIGURE 14. - The calibration curve for controller P is the measured controller flow rate corrected to 25° C, 28.89 in Hg versus scaled digital panel meter reading of controller output voltage (command signal).

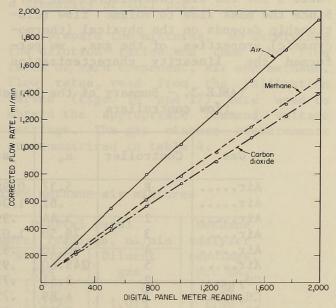


FIGURE 16. - The calibration curve for controller 3 is the measured controller flow rate corrected to 25°C, 28.89 in Hg versus scaled digital panel meter reading of controller output voltage (command signal).

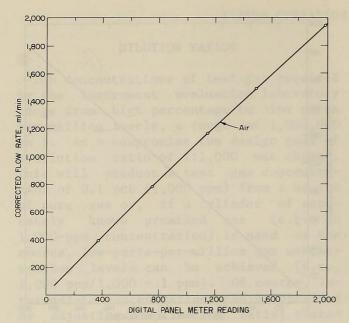
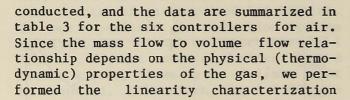


FIGURE 17. - The calibration curve for controller 4 is the measured controller flow rate corrected to 25°C, 28.89 in Hg versus scaled digital panel meter reading of controller output voltage (command signal).



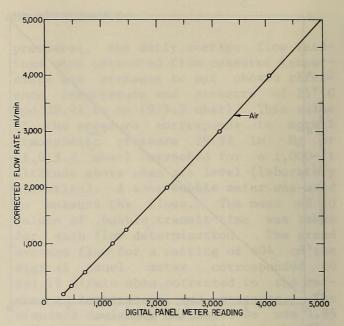


FIGURE 18. - The calibration curve for controller 5 is the measured controller flow rate corrected to 25°C, 28.89 in Hg versus scaled digital panel meter reading of controller output voltage (command signal).

of controller 2 for pure carbon dioxide, methane, and carbon monoxide. All of the data are well characterized as straight lines since the square of the coefficient of regression approaches unity; values obtained for r² ranged

TABLE 3. - Summary of the linear regression 1 analysis for Tylan flow controllers

1000	5,100 Ea. 5,				4 - 1	Maximum	CV,
Gas	Controller	ao	a ₁	r ²	Syx	flow,	pct
						ml/min	
Air	P	5.37	0.9824	0.9999	0.739	200	0.74
Air	1	.82	1.0410	.9997	.069	10	1.40
Air	2	5.44	.9968	.9992	1.840	200	1.84
Air	3	78.50	1.0100	.9999	7.120	2,000	.71
Air	4	40.10	.9382	.9991	21.120	2,000	2.10
Air	5	243.60	.9505	.9999	17.350	5,000	.69
CO2	2	3.51	.7277	.9986	1.770	200	1.77
CH4	2	4.89	.7315	.9986	1.690	200	1.69
CO	2	5.83	.9973	.9997	.890	200	.89

¹Regression equation: measured flow = $a_0 + a_1$ (panel meter reading) and a_0 = intercept,

 $a_1 = slope,$

r² = coefficient of regression,

 S_{yx} = standard deviation of y on x,

CV = coefficient of variation = $\frac{S_{yx}}{\bar{x}}$ · 100 pct.

from 0.9986 to 0.9999. The values of a_0 , the intercept of the regression line, and a_1 , the slope of the lines, are given for the general equation:

measured gas flow (in ml/min) = $a_0 + a_1$ (digital panel meter reading).

Each point is the average of 10 flow measurements. The average deviation of the points from the expected line (S_{yx}) , divided by the average flow values (one-half of maximum flow), yields coefficient of variation (CV) data; the CV values ranged from 0.7 to 2 pct for the six controllers.

BINARY MIXTURE PREPARATION

We used the dynamic gas-mixing system to make different concentrations of methane gas in air from a standard tank of 2.58 pct CH4, and the results are presented in figure 19. Two identical flow controllers, Nos. 3 and 4, each with a range of 40 to 2,000 ml/min, were used to generate mixtures flowing at 2,000 ml/ The methane concentration of each min. mixture was measured by an ANDROS, Inc., model 209 methane analyzer, which is claimed to have an accuracy and linearity of 2 pct of full scale (5 pct methane), i.e., ±0.1 pct methane. The output from the analyzer was also measured by a digital multimeter (Tektronix DM-501) which

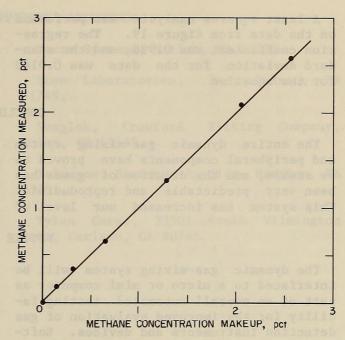


FIGURE 19. - Measured methane concentration versus desired values. Flow controller settings were selected using figures 16 and 17 and the desired flow rates.

has an accuracy of ± 0.1 pct ± 2 counts and a linearity of ± 0.1 pct.

To prepare the mixtures, we determined the dilution ratios and thus the flow rates of each component, air and methane. Flow rates, read from the calibration curves (figs. 16 and 17), were used to obtain the appropriate command voltage settings. The gas mixture measurements are summarized in table 4.

TABLE 4. - Desired and obtained methane-air mixtures

Desired	Desired	flow,	Digital	Methane	
methane	m1/m	in	meter readin	ngs, ml/min	analyzer
concentration,	Standard	Diluent	Standard	Diluent	readings,
pct CH ₄	gas	gas	gas	gas	pct CH ₄
2.58	2,000	0	1,830	0	2.58
1.29	1,000	1,000	1,035	1,015	1.30
.645	500	1,500	515	1,570	.64
.322	250	1,750	265	1,850	.34
.161	125	1,875	144	1,998	.17
2.064	1,600	400	1,695	385	2.09

A least squares analysis was performed on the data from figure 19. The regression coefficient was 0.998, and the standard deviation for the data was 0.0143 for the equation (measured methane concentration, pct)

= 1.01 (makeup methane concentration, pct).

CONCLUSION

The entire dynamic gas-mixing system and peripheral components have proved to be stable, and the dilution of gases has been very predictable and reproducible. This system has increased our level of

confidence in the preparation and use of test gases. It is in continual use and contributes significantly to reducing the costs of operating the instrument evaluation laboratory.

FUTURE PLANS

The dynamic gas-mixing system will be interfaced to a micro or mini computer as part of an overall automated testing facility for the improved evaluation of gas detection instruments and devices. Software programs will be used to specify

temperature, humidity, gas flow (gas concentration), and time for conducting tests of gas monitors for the determination of linearity, accuracy, response time, precision, and stability.

APPENDIX A.--SUPPLIERS' ADDRESSES

AADCO, Inc., 2257 Lewis Ave., Rock-ville, MD 20851.

ANDROS Analyzers, Inc., 2332 Fourth St., Berkeley, CA 94710.

Forma Scientific, Inc., Marietta, OH 45750.

Labcrest, Fischer Porter Co., Warminster, PA 18974.

Litronix, Inc., 19000 Homestead Rd., Vallco Park, Cupertino, CA 95014.

Mast Development Co., 2212 East 12th St., Davenport, IA 52803.

Serta Systems, Natick, MS 01760.

Stow Laboratories, Inc. Hudson, MS 01749.

Swaglok, Crawford Fitting Company, Solon, OH 44139.

Teledyne, Hastings-Raydist, Hampton, VA 23661

Tylan Corp., 23301 South Wilmington Ave., Carlson, CA 90745.

APPENDIX B .-- DIGITAL TIMER OPERATION AND CONSTRUCTION

The digital timer is used to manually or automatically time soap-bubble flowmeter transit times. It is constructed from readily available parts, mainly integrated circuits and standard lightemitting diode (LED) displays. To simplify flow rate calculations, the display is formatted in decimal fractions of minutes. The resolution is 100 µsec and 1 msec in the 10-min and 100-min ranges, The 10-min range provides respectively. a maximum indication of 9.9999 min and is usually used for the bubble meter transit time measurements. Hour and second time bases are also available, with corresponding resolutions.

TIME BASE GENERATION AND SELECTION

All time bases are derived from a 2-MHz crystal oscillator (fig. B-1). The 2-MHz signal is divided by 2,120, and 7,200 to produce frequencies of 1 MHz (F_1), 16.66...kHz (F_2), and 277.7...Hz (F_3), respectively.

The desired time base is selected by SW-3 (fig. B-2). With SW-3 in the position shown, the control input to select gate 2 (pin 4) will float high, and the output (pin 6) will toggle at the F_2 rate. The two inputs to both invert gates are pulled high, which forces a low at the outputs and inhibits select gates 1 and 3.

Placing SW-3 in the "Sec" position inhibits select gate 2 (through the coupling diode) and places a low (ground) on the inputs (pins 2 and 3) of the upper invert gate in figure B-1. The high output from this inverter enables select gate 1, and allows frequency F_1 to appear at the output (pin 3). A similar action occurs when SW-3 is in the "Hr" position. In this case, select gates 1 and 2 are inhibited; select gate 3 is enabled and passes F_3 to its output.

The selected time base $(F_1, F_2, \text{ or } F_3)$ is buffered and passed on to the input of a decade counter and to the range switch, SW-4B. The output from the counter (F_n/F_1)

10) is connected to the opposite side of the range switch. The output from the time base generation and selection section is F_n or $F_n/10$, depending on the position of the range switch. Switch SW-4A sets the decimal point to display one (range = 10) or two (range = 100) integers in the five-digit display.

The selected time base is routed through one of two run gates in the control and gating logic section (fig. B-3) to the counters and displays section (fig. B-4). In this section the time base is divided by 100 and then fed sequentially to five display counters. The BCD outputs from the counters are decoded by the 7447 drivers (fig. B-4) into seven drive lines to the LED readouts (RO).

TIME BASE CONTROL

The starting and stopping of the selected time base frequencies to the counters is controlled in two ways: One uses a pair of phototransistors, one to start, the other to stop the counter; The other uses a single switch to start, stop, and reset the timer. The circuit for the time base control is shown in figure B-3. The double-pole switch, SW-2A and SW-2B, is used to select either of these two ways: In the X position shown, the phototransistors control the time base to the counters and SW-1 is used to reset the counters in preparation for another measurement; in the Y position, the switch SW-1 performs the start, stop, and reset operations in succession, thus controlling the timer as a manually operated Each of these time-basestopwatch. controlling methods is described in detail below.

Switch SW-1, connected to J-3 (fig. B-3), is used to reset the timer when switch SW-2 is set to the X position. When momentary contact switch SW-1 is closed, a low is coupled through the diode to pin 3 of the one-shot multivibrator OS-2. This triggers the one-shot multivibrator and produces two 500-µsec

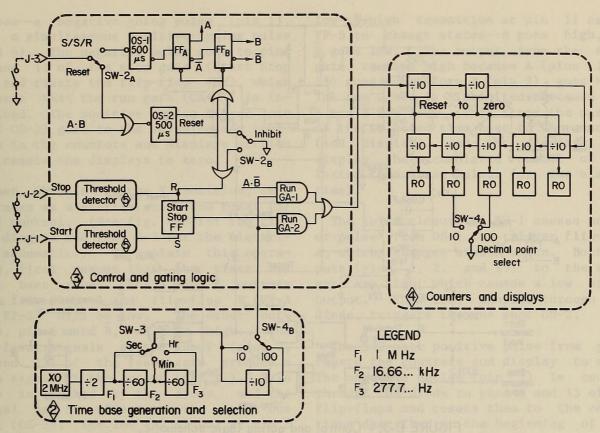


FIGURE B-1. - Functional block diagram of the digital timer.

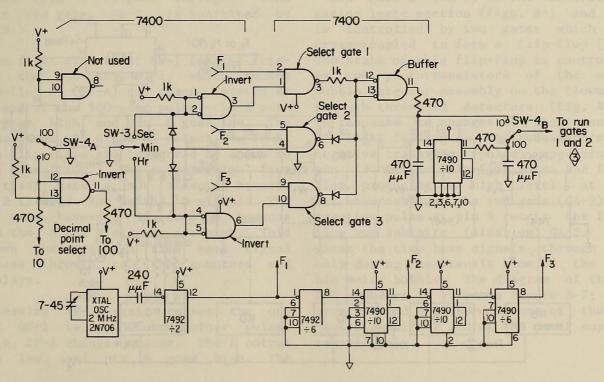


FIGURE B-2. - Time base generation and selection schematic.

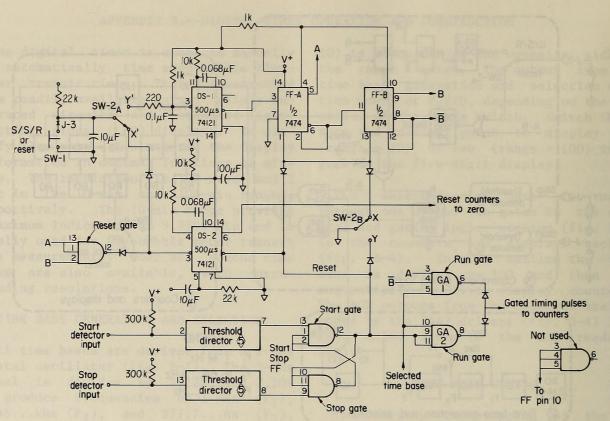


FIGURE B-3. - Control and gating logic schematic.

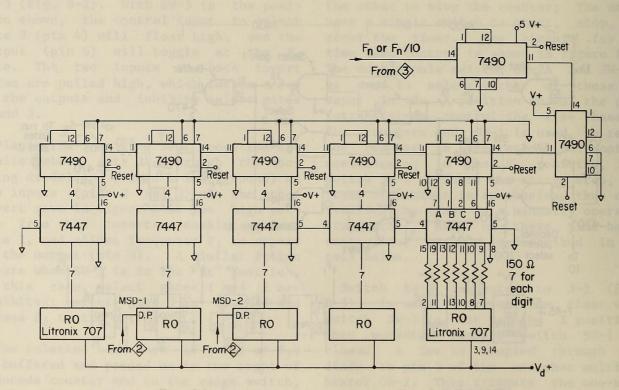


FIGURE B-4. - Counters and display schematic.

pulses—a negative going pulse (pin 1), and a simultaneous positive going pulse (pin 6). The negative pulse goes to pins 10 and 11 of the stop gate (Start Stop FF) and resets the flip—flop (FF), which insures that the run gate (GA-2) is in—hibited. The positive pulse (from pin 6 of OS-2) goes to the seven decade counters in the counters and displays section and resets the displays to zero.

When SW-2 is in the Y position, SW-1 operates as a start, stop, and reset (S/ S/R) control. (See fig. B-7 for the timing diagram and fig. B-3 for the electrical schematic.) To explain this operation, first assume that the timer has just been reset. The A and B outputs from flip-flop A and flip-flop B (FF-A and FF-B) will be low. The other outputs, pin 6 and \overline{B} , will be high. low-level signals at the input (pins 1, 2, and 13) of the reset gate produce a high signal at the output (pin 12). This high is blocked by the diode, and the signal has no effect on the reset one shot (OS-2). The low (A) signal at pin 3 of the run gate (GA-1) inhibits this gate and does not allow the selected time base signal (pin 5) to pass through. other run gate, GA-2, is inhibited by SW-2B.)

The first closure of SW-1 (start) triggers the one shot OS-1, which causes flip-flop A (FF-A) to change states at the end of the 500-µsec pulse--the A output goes high and pin 6 goes low. The signal from pin 6 has no effect on FF-B. (The flip-flops are triggered by a low to high transition.) The output (high) from the reset gate is not affected because pin 2 is held low by the B signal. The run gate, however, is enabled because both the A and B signals are high. This allows the selected time base signal to pass through to the counters and displays.

Pressing SW-1 again causes the one shot OS-1 to produce another pulse. Again, FF-A changes states. The A output goes low, and pin 6 goes high. The

low-to-high transition at pin 11 causes FF-B to change states—B goes high, and \bar{B} goes low. The output from the reset gate remains high because A (pins 1 and 13) goes low before B (pin 2) goes high. The run gate is inhibited because both A and \bar{B} are low. This stops the passage of the selected time base to the counters (and display), and the displays will display the accumulated number of selected time base pulses (or the elapsed time).

The third closure of SW-1 causes another pulse from OS-1 to trigger flip-flop A, which changes states again. Both inputs (pins 1, 2, and 13) to the reset gate are high, which causes a low at the output. This low, coupled through the diode, triggers the one shot OS-2.

The $500\text{-}\mu\text{sec}$ positive pulse from pin 6 resets the counters and display to zero. The negative pulse (pin 1) is coupled through the diode to pins 1 and 13 of the flip-flops and resets them to the conditions described at the beginning of this discussion.

Run gate 2 (GA-2) in the control and gating logic section (figs. B-1 and B-3) is controlled by two gates which are cross-coupled to form a flip-flop (FF). The state of this flip-flop is controlled by two phototransistors of the soapbubble detector assembly on the flowmeter through threshold detectors (fig. B-5), in response to changes in the intensity of light falling on the transistors. A negative transition (pulse) appearing at pin 13 of the flip-flop sets the flipflop producing a high level at the output--enabling the run gate (GA-2). A negative pulse at pin 9 resets the flipflop and inhibits (disables) GA-2. This gates the time base signals through GA-2 only during the transit time of the bubble meter bubble. The diagram of the S/ S/R sequence is given in figure B-7; figures B-5 and B-6 are diagrams of the detector amplifier and of the power supply, respectively.

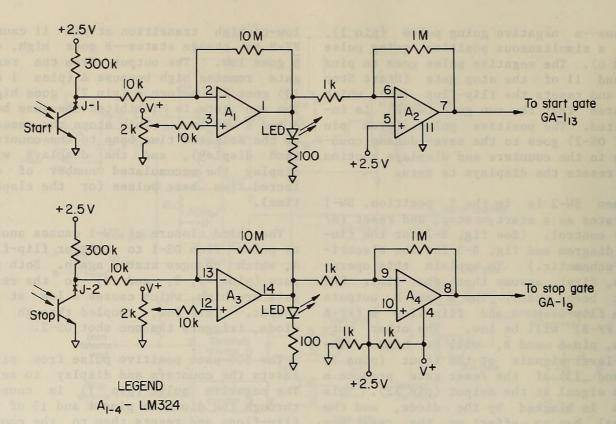


FIGURE B-5. - Soap bubble transit detection circuitry.

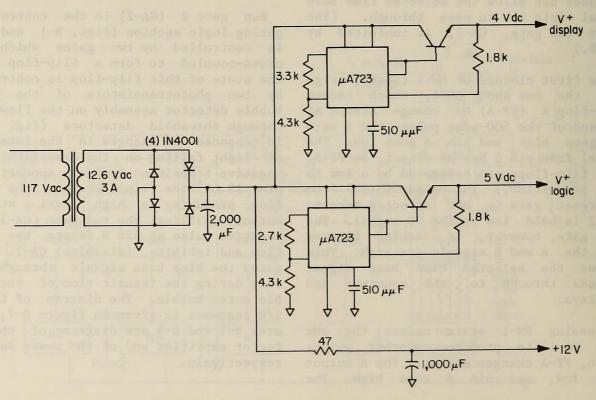


FIGURE B-6. - Timer power supply schematic.

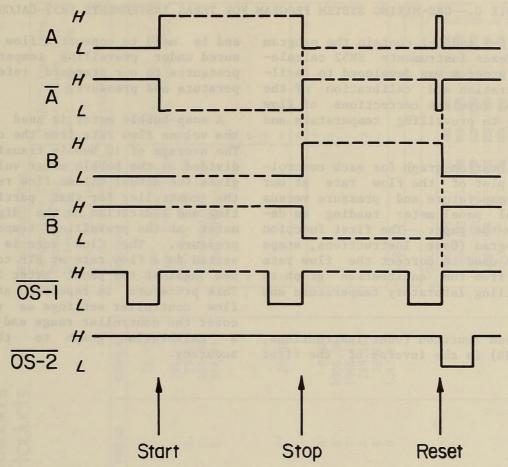


FIGURE B-7. - Timing diagram of logic states of control line of the timer.

APPENDIX C.--GAS-MIXING SYSTEM PROGRAM FOR TEXAS INSTRUMENTS SR52 CALCULATOR

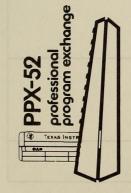
Figures C-1 and C-2 contain the program for the Texas Instruments SR52 calculator. The program was developed to facilitate operation and calibration of the system and involves corrections of flow rate owing to prevailing temperature and pressures.

The calibration graph for each controller is a plot of the flow rate at our standard temperature and pressure versus the digital panel meter reading as described in the paper. The first function of the program (User Instructions, steps IA-3A) is used to correct the flow rate obtained from the calibration graph to the prevailing laboratory temperature and pressure.

The second function (User Instructions, steps 1B-3B) is the inverse of the first

and is used to convert a flow rate measured under prevailing temperatures and pressures to our standard reference temperature and pressure.

A soap-bubble meter is used to measure the volume flow rate from the controller. The average of 10 bubble transit times is divided by the bubble meter volume. This gives the actual volume flow rate through the controller for that particular setting and indication on the digital panel meter at the prevailing temperature and The flow rate is then conpressure. verted to a flow rate at STP to be plotted against the panel meter indication. This procedure is repeated at as many flow controller settings as required to cover the controller range and to produce calibration graph to the desired accuracy.



FEXAS INSTRUMENTS Calculator Products Division

Program Title GAS MIXING SYSTEM

V. F INIT.		*Transit Time (min.) *Tube Volume (ml) cCalc. Meas. Flow	o. FInitialize		10 Tamp (9V)	Std.	13	₹ ¥	. Q	17	18	19	66	5		8	6
T-TIME VOLUME CALC. TEMP PRESS FLOW	USER DEFINED KEYS	A Temperature (°C) B Pressure ("Hg) c Desired Flow Rate	D Calc. STP Flow E Calc. Flow Setting	REGISTERS	00 Tama (ok)		₀ Desired Flow Rate	04Standardized Flow	90	07	90	60	86	0	2 30 00 08 22	378 33 03 08	4 34 10 70 00
				DISPLAY	28.92	205 15	29.43	950.00	751.17		8000	28.92		0.94	1000.00	299.65	28.45

ECBA

22 29.43 950

A. Enter Operating Temperature B. Enter Operating Pressure C. Enter Desired Flow Rate D. Calculate Controller Setting (Use this value as actual flow rate to determine DPM setting

Find "Indicated" Flow Rate

ū

DBACBA

0.938 1000 -26.5 28.45

Enter Bubble Meter Tube Size

Calculate Rate of Flow Enter Present Temperature

Standardize Flow to S.T.P. A. Enter Average of 10 Bubble Transit Times

on calibration graph.) Load Program Card

Initialize

38 38

Enter Present Pressure Calculate Flow Rate at S.T.P.

(For use on calibration

graph)

PRESS

ENTER

PROCEDURE

STEP

Load Program Card

Initialize

1A 2A 3A

User Instructions

4		[] [set	[] read] [] list	[] pr	ded []	181 [] [BI		_
		1 [] [2′] 3	E 4'	2 2	,9	05 7"	8 8	·6 [
3		P/R [_] dsz	T () T	CMs st fly	EXC [] IT US	PROD [] If err	x pos	rtn [] d 2r0	
2		HLT	RUN	log	Z	1/2	IND ON	O WS	D/R
						+			☐ -/+
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FIGHRE C.1.	-
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PPX-52 Professional Program Exchange

For TI use only

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	- 1	*LBL C STO 03 HLT	*LBL *A' STO 06 HLT	*LBL *C' RCL 05 ÷ RCL 06 RCL 06 STO 03	HLT	*LBL *B' STO 05 HLT	*LBL A + 273 .15	STO 01 HLT	20	POCE
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FIGURE C-2. - Program listing for gas dilution mixing program.

