

The Generation of Test Atmospheres
for Occupational Hygiene Laboratory Evaluation
of Organic Vapour Monitoring Devices

**Report prepared for the Occupational Health
and Radiation Control Branch
South Australian Health Commission**

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- S.A.H.C. Industrial Hygiene Laboratory**

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**PART 1: REVIEW OF METHODS FOR THE GENERATION
OF TEST ATMOSPHERES IN THE LABORATORY**

Methods for the generation of test atmospheres are generally divided into two categories: static methods and dynamic methods.

Methods in the former category involve the introduction of a known weight or volume of contaminant into a container of fixed dimensions. Non-rigid containers holding metered volumes of gases are also used.

Dynamic methods, also referred to as flow-dilution methods, involve continuous introduction of contaminant, at a controlled rate, into a stream of diluent gas, usually air.

A. STATIC METHODS

Static systems are usually employed when comparatively small volumes are required. Applications include instrument calibration and the production of gas phase standards for gas chromatography, mass spectrometry and infrared spectroscopy. Static devices may be used at any desirable pressure but laboratory applications are generally limited to systems at atmospheric pressure or slightly below.

Advantages of static systems include simplicity, low cost and ease of operation. Disadvantages include adsorption and reaction on container walls as well as permeation through walls in the case of plastic bags.

A convenient static system is one which involves the introduction of a predetermined amount of contaminant into a rigid vessel, e.g. glass container, of known volume as in Figure 1. A disadvantage of this technique is that the concentration decreases during the process of withdrawing a

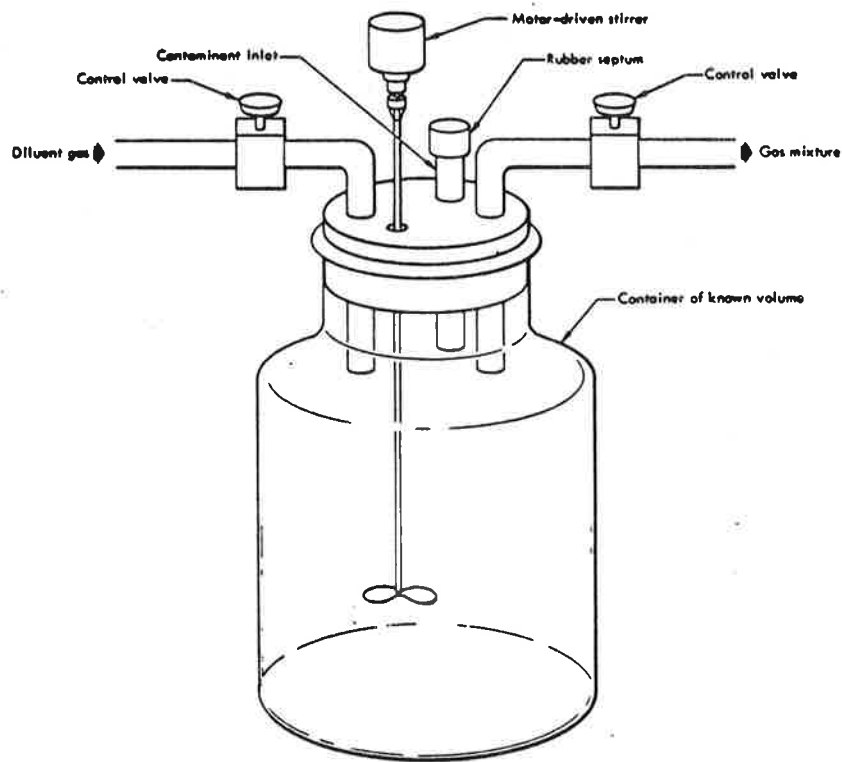
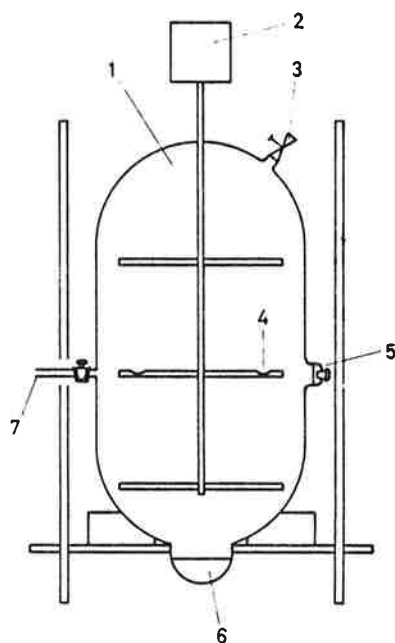


Figure 1 : Sketch of a rigid static system for producing gas and vapour mixtures



- 1 Container
- 2 Stirrer
- 3 Ventilation
- 4 Evaporator dish
- 5 Feed opening for admixture
- 6 Cleaning opening
- 7 Sampling nozzle (the detector tube is connected at this point)

Figure 2 : 400-litre container (borosilicate glass) for static test gas settings

sample. Assuming the worst possible case of complete turbulent mixing in the bottle, the change in concentration is given by

$$C/C_0 = \exp(-W/V)$$

where C = final concentration in bottle

C₀ = initial concentration

W = volume withdrawn, litres

V = original volume of mixture, litres.

A withdrawal of up to 5% of the atmosphere is acceptable (5% error). Figure 2 [1] illustrates a 400 litre container used for evaluating Drager detector tubes. Figure 3 shows a setup which avoids sample dilution. Alternatively, relatively large volumes of test gas may be withdrawn from rigid chambers in series [2].

A recirculating pump and in-line detector are occasionally incorporated into a static system as in Figure 4 [3].

Non-rigid chambers, e.g. plastic bags, allow the entire sample to be withdrawn without any troublesome volume dilution with replacement air. Gas mixtures in plastic bags can be carried into the field and used for instrument calibration.

Caution should be applied when using plastic bags for certain materials at low concentration. Conditioning of bags is essential to minimize losses [4].

Static methods are inferior to dynamic methods for the assessment of the performance of passive monitors or charcoal tubes. The system in Figure 4 relies on infrared monitoring of concentration for the entire exposure period.

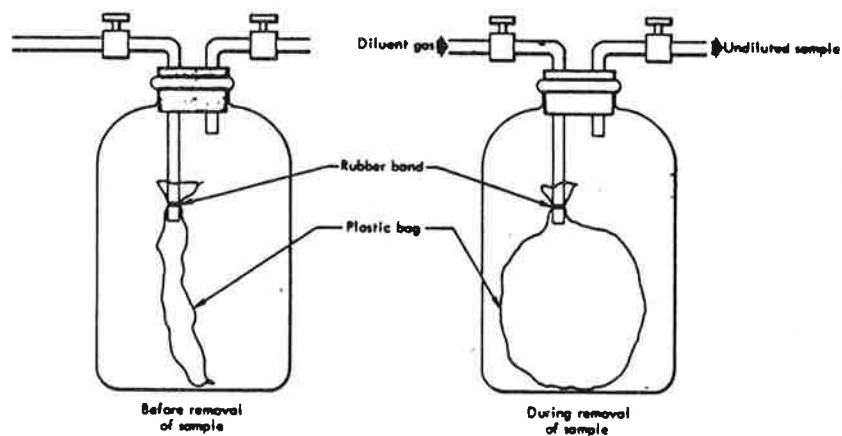


Figure 3 : Setup for removing a test gas from a rigid static system without causing sample dilution

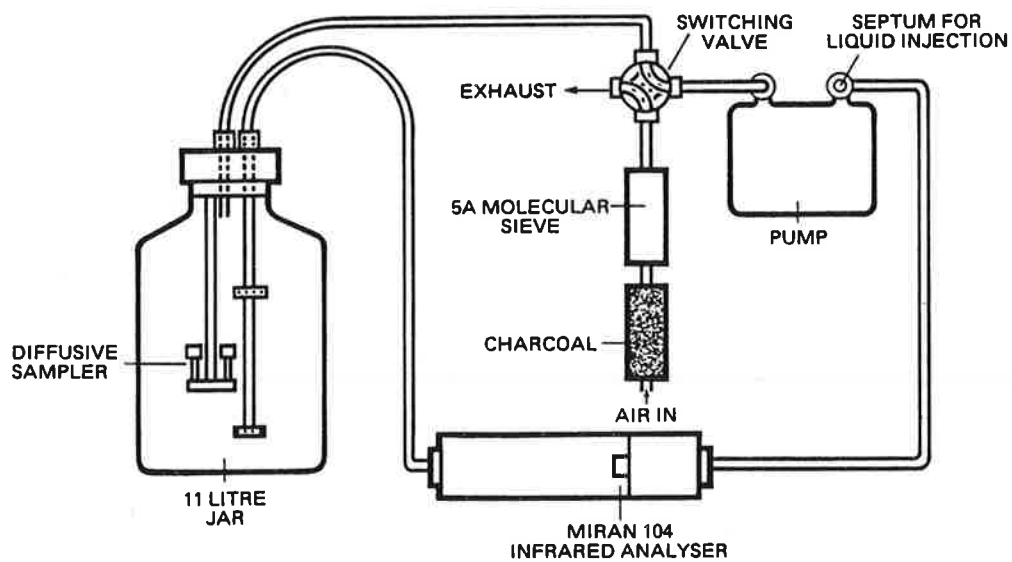


Figure 4 : Recirculating system for generation of standard atmosphere

B. DYNAMIC METHODS

Dynamic systems have many advantages over static systems, particularly in relation to the production of reactive gas mixtures. If the mixture is prone to decomposition or chemical reaction, the undesirable reaction products can be swept away and continuously replaced by the relatively pure and unreacted test gas mixture. Large volumes can be produced for extended time intervals with equipment which may be reasonably compact.

Wall adsorption, a problem in most static systems, usually becomes negligible since an equilibrium is established after operating for a long enough time period. In a properly designed system concentrations may be changed very rapidly and explosive hazards associated with dynamic systems are typically less than those of static systems. Because of these advantages, dynamic systems are popular for accurate work with most substances. Applications include toxicological, inhalation and odour investigations as well as kinetic studies and analytical standard preparation. On the other hand, dynamic methods are usually more elaborate and expensive than static methods. Various types of dynamic systems which have been used for the generation of test atmospheres are described in Nelson's book [2] and also in references 1 and 5. These will now be briefly reviewed.

Gas stream mixing

Since a flow dilution system is generally comprised of a metered test substance source, a metered diluent gas source and a mixer, flow monitoring devices are almost always incorporated into the system. Rotameters are commonly used but orifice meters and critical orifices are also frequently employed.

The most widely used and successful method of mixing two or more gases is to dilute the gases with one another after measuring their flow rates. Figure 5 shows the components of a test mixture being initially metered through previously calibrated rotameters. They are subsequently combined into a single test mixture after passing through a mixing chamber. Multiple dilution allows the generation of very low contaminant concentrations without the use of extremely low flow rotameters. Figure 6 depicts a system for making double dilutions. Note that pressure buildup in the first stage must be determined and corrected for.

One disadvantage of multiple dilution systems is that a very high proportion of contaminant gas or vapour is wasted after each dilution stage.

The gas stream mixing (dilution) method is very convenient for contaminants which can be purchased in pure (compressed) gas form or in the form of a certified gas mixture with nitrogen, e.g. ethylene oxide.

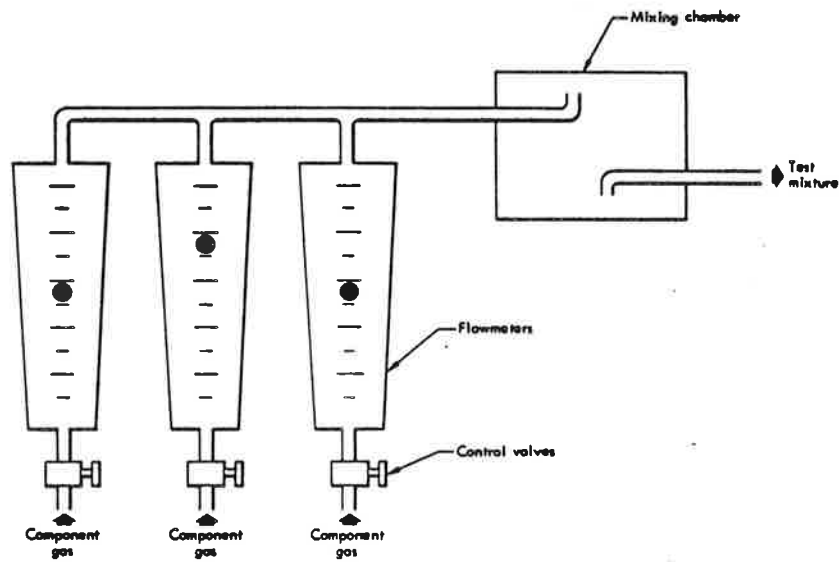


Figure 5 : Sketch of a system for mixing three-component gas streams

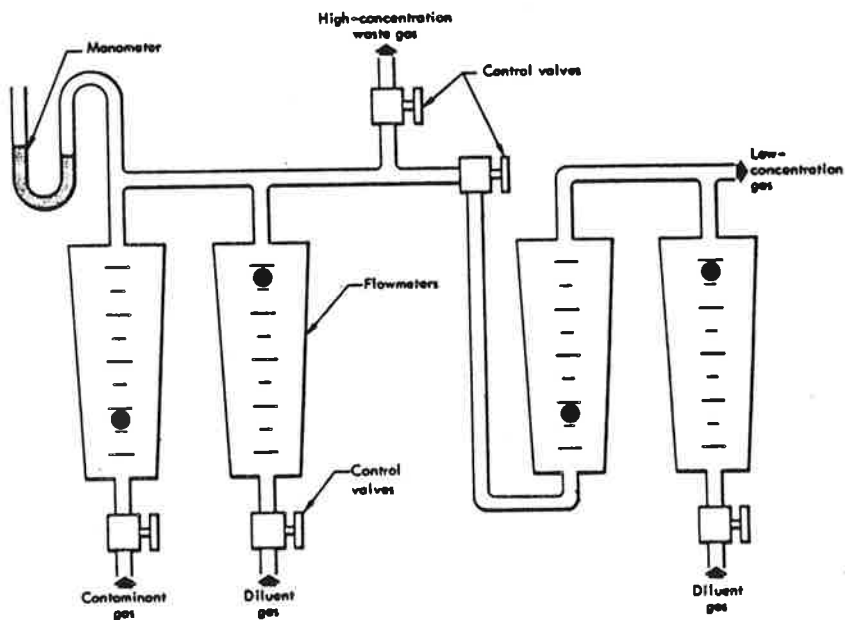


Figure 6 : Sketch of a system for making double dilutions

Injection

Contaminant gases and liquids can be added to moving gas streams by means of a wide variety of mechanical dosers, injectors and pumps as well as gravity and electrolytic feeding units [2].

The most common device is a motor driven syringe, although HPLC pumps are now often used for liquids [6].

In the case of liquid injection, smooth evaporation and mixing is required for reliable vapour concentrations. In order to increase the rate of evaporation for liquids of low volatility, three main methods are employed, viz, (1) increase the surface area between diluent gas and liquid, e.g. by using a glass wool plug [7] or atomiser; (2) heat the liquid, or preferably diluent gas, as in Figure 7 and (3) increasing the amount of gas turbulence around the emerging liquid.

Motor driven syringe systems, as in Figure 8, are extremely versatile and have the advantage of little or no waste. Addition of liquid vapour or gas does not need to be smooth and continuous but may be pulsed [1] provided that downstream mixing is efficient and uniform.

Diffusion and Permeation

Systems based on diffusion tubes or permeation tubes are convenient for producing low concentrations of certain liquid vapours in a moving gas stream.

Figure 9 illustrates a system based on a diffusion tube kept at constant temperature. Flow rates past the diffusion

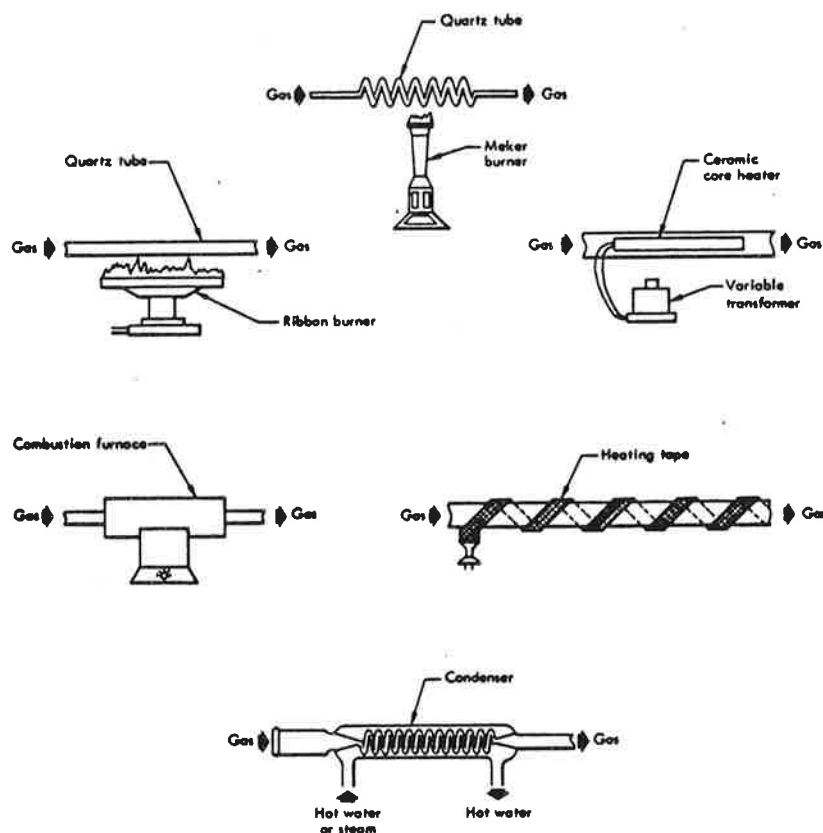


Figure 7 : Sketches of several methods for heating gases so that they can later vapourize liquids

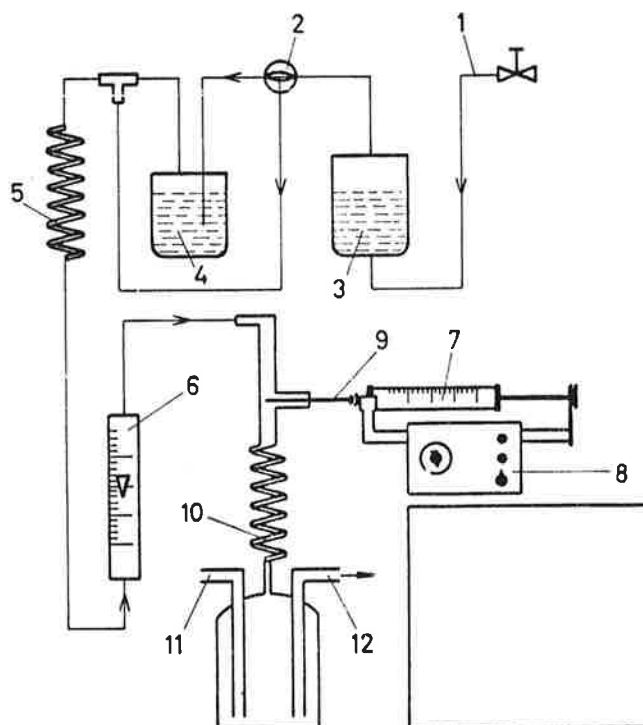


Figure 8

Continuous injection of liquids with vapourization

- 1 Inlet of basic gas
- 2 Change-over cock
- 3 Filter pot (for gas purification)
- 4 Humidifier
- 5 Temperature compensation
- 6 Flow meter (for basic gas)
- 7 Glass piston sampler (filled with the liquid admixture)
- 8 Transport device with gear motor
- 9 Capillary tube
- 10 Evaporation section
- 11 Sampling nozzle (the detector tube is connected at this point)
- 12 to the exhaust

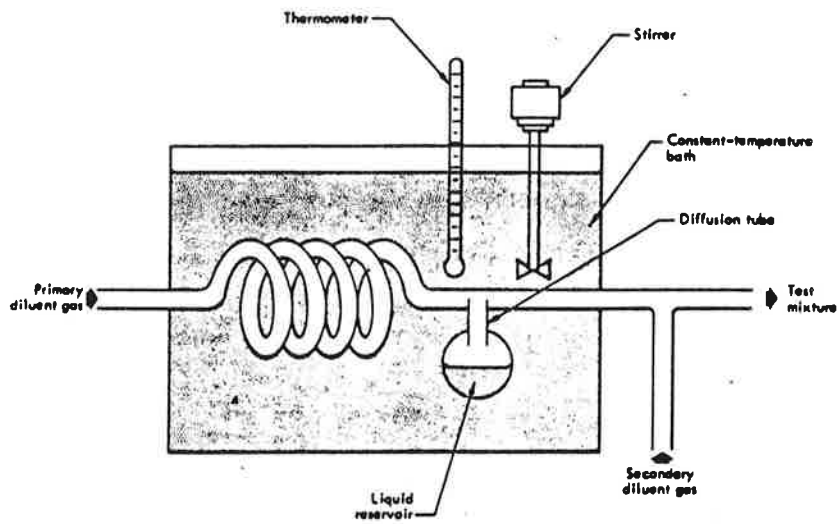


Figure 9 : Sketch of a dynamic diffusion system

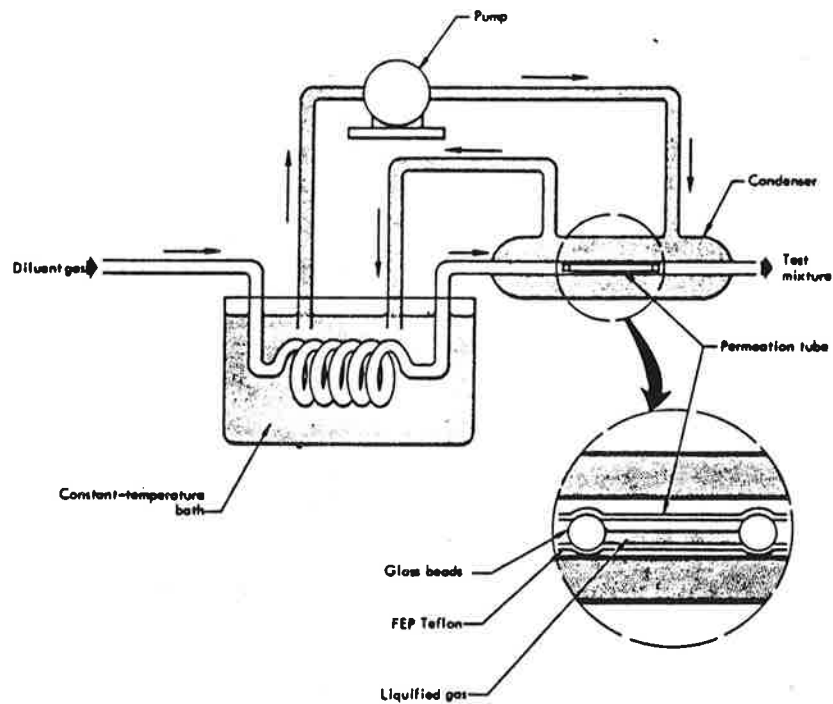


Figure 10 : Sketch of a method for dynamically producing gas mixtures with a permeation tube

tube should not usually exceed one litre per minute since diffusion depends on concentration gradients as well as tube geometry and temperature. Recent literature applications are given in references 8 - 10 and a good theoretical description with examples is to be found in Nelson's book [2].

Permeation tubes can be used to produce standard gas mixtures in the parts per million range as in Figure 10. Permeation rates are generally calibrated gravimetrically or volumetrically at the experimental temperature desired. Methods relying on permeation tubes can be very accurate but there are relatively few materials which permeate fast enough to be useful at even a low flow rate.

Evaporation

This technique, one of the most generally useful, involves passing diluent gas in close proximity to the liquid or volatile solid to be vaporized. The gas stream can be saturated and further diluted but partial saturation is perhaps more desirable for volatile liquids. Classic types of evaporation units are shown in Figure 11. The extent of vaporization is temperature dependent so that temperature must be controlled. In the case of partial saturation, the effluent gas mixture must be independently analysed, e.g. by infrared spectroscopy or gas chromatographic techniques [11]. Weight loss from the evaporation vessel, where appropriate, can also be used to compute concentrations. If the diluent gas has reached

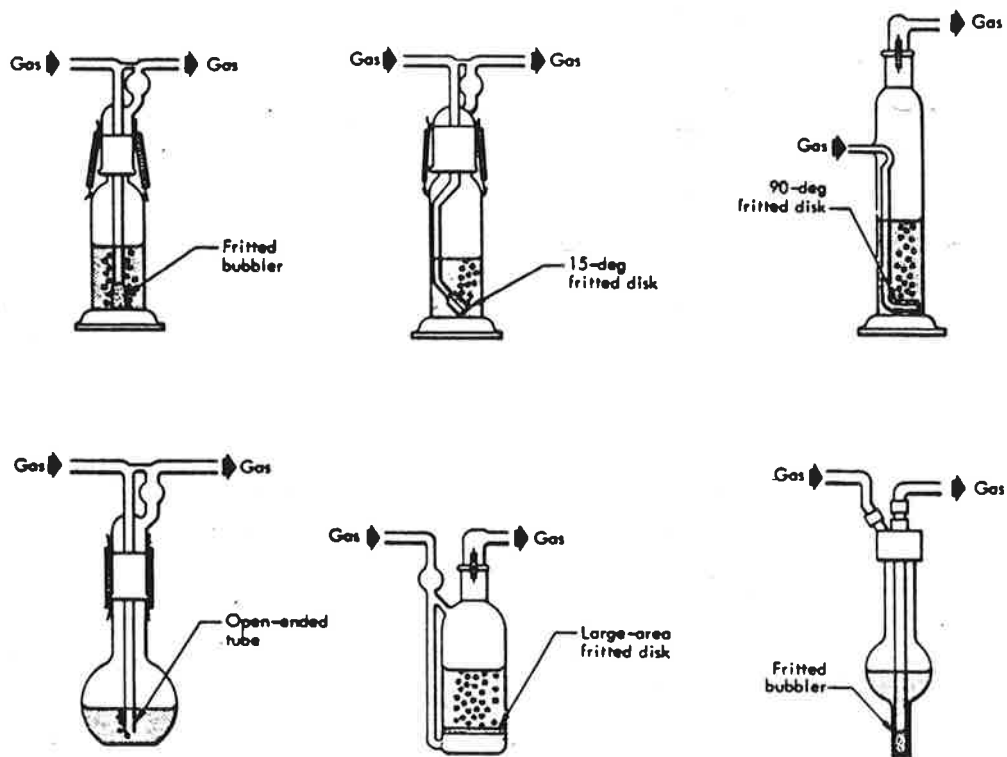
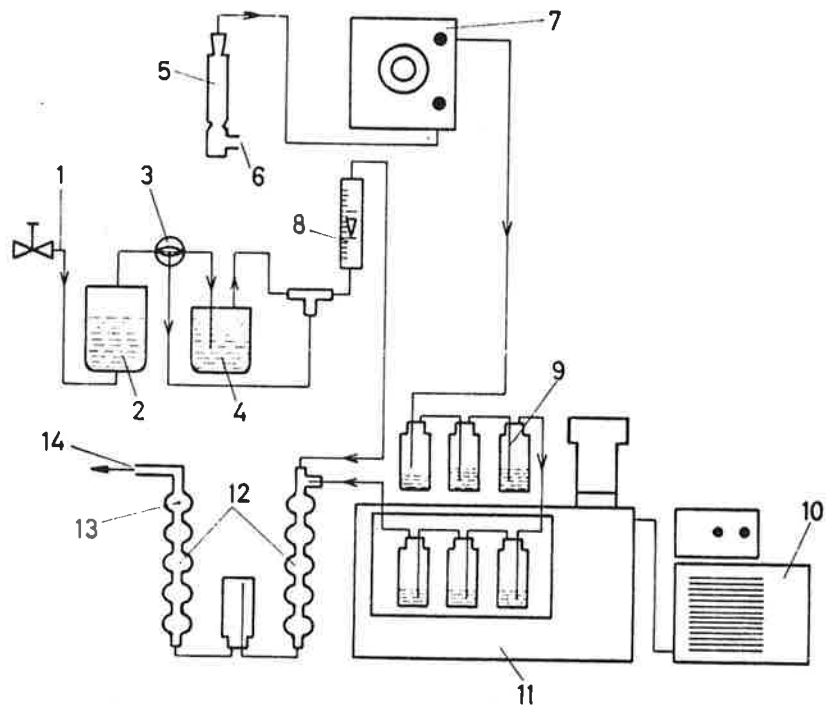


Figure 11 : Sketches of six typical gas-dispersion bottles

Figure 12

**Periodic injection of vapours
by means of valve-
controlled diaphragm pumps**

- 1 Inlet of basic gas
- 2 Filter pot (for gas purification)
- 3 Change-over cock
- 4 Humidifier
- 5 Drying tower
- 6 Inlet of delivered gas (carrier gas)
- 7 Diaphragm pump (to measure the delivered gas)
- 8 Flow meter (for basic gas)
- 9 Presaturation of the delivered gas at about 23 °C by vaporous admixture
- 10 Temperature control
- 11 Thermostat (20 °C) with saturation section for admixture
- 12 Mixing section for basic gas and admixture
- 13 Sampling nozzle (the detector tube is connected at this point)
- 14 to the exhaust



theoretical saturation at a known temperature (usually less than ambient temperature) concentrations may be calculated directly from vapour pressure data.

An example of a total saturation system is shown in Figure 12.

Potts and Steiner [12] have described an apparatus for the generation of vapours from liquids of low volatility. The method is basically countercurrent volatilization employing a multi-plate high efficiency distillation column. It is most useful if there is a danger of thermal decomposition of the test liquid.

SRI International have made use of a vapour saturation generator (as well as a diffusion tube generator) for the preparation of U.S. NIOSH Proficiency Analytical Testing Program samples [13].

Electrolysis and Chemical Reaction

Electrolytic methods are sometimes useful for the production of certain gases such as arsine and stibine. Figure 13 depicts a U-shaped electrolytic reaction vessel. Under ideal conditions, seldom achieved, the rate of gas production is directly proportional to the current supplied. Potential advantages include the ability to quickly produce very low gas concentrations. However, the method has limited applicability and subtle problems may arise if the electrolytic system has not been investigated properly.

Laboratory chemical reactions may be used to produce unstable, extraordinarily reactive, commercially unavailable

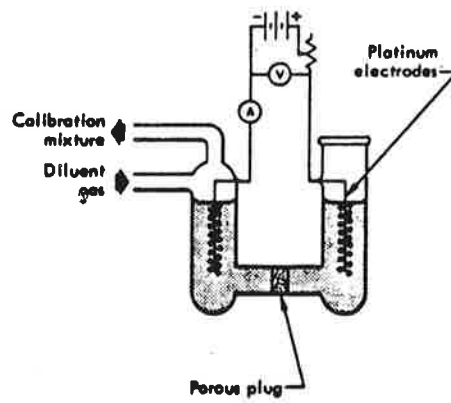


Figure 13 : Sketch of a U-shaped electrolytic reaction vessel in which the unwanted gas generated by the right-hand electrode is vented to the atmosphere

or prohibitively expensive test materials. Very few reaction systems, however, have been practiced [14].

In situ contaminant formation probably represents the last resort in test atmosphere generation because of often poorly investigated kinetic and thermodynamic factors.

PART 2: DYNAMIC TEST ATMOSPHERE GENERATOR

SOUTH AUSTRALIAN HEALTH COMMISSION

INDUSTRIAL HYGIENE LABORATORY

A. PRINCIPLES OF OPERATION

The apparatus utilizes syringe injection and gas stream mixing methods in a dynamic system.

It is based upon a U.K. Health and Safety Executive (HSE) design presented as Appendix 2.3. More specific details are available from HSE specifying GCIS 3/1978.

Whilst the HSE apparatus was designed for mobile laboratories, the South Australian Health Commission (SAHC) system is not strictly portable: It consists of three separate mobile units as in Figure 14. The central unit is normally located in a fume cupboard. In the SAHC Industrial Hygiene Laboratory one fume cupboard has had its bench top cut out, allowing easy ingress and egress of the central unit, approximately one metre wide. Toxic emissions are contained in the fume cupboard. The heater/humidifier unit deals only with diluent gas and the detector unit has an exhaust vented back into the fume cupboard.

Construction materials for the system are borosilicate glass, teflon, Kel-F, viton and 316 stainless steel. Other materials such as nylon, brass and silicone rubber are used where only diluent gas is present.

Humidification of the diluent gas stream is accomplished by using a split stream saturator (see Section C.3) and monitored by a capacitance sensor at the exhaust.

Heating of the diluent gas stream is accomplished by passage through a cylindrical electric furnace followed by tubing wrapped with electrical heating tape. A temperature controller prevents overheating of the teflon tubing.

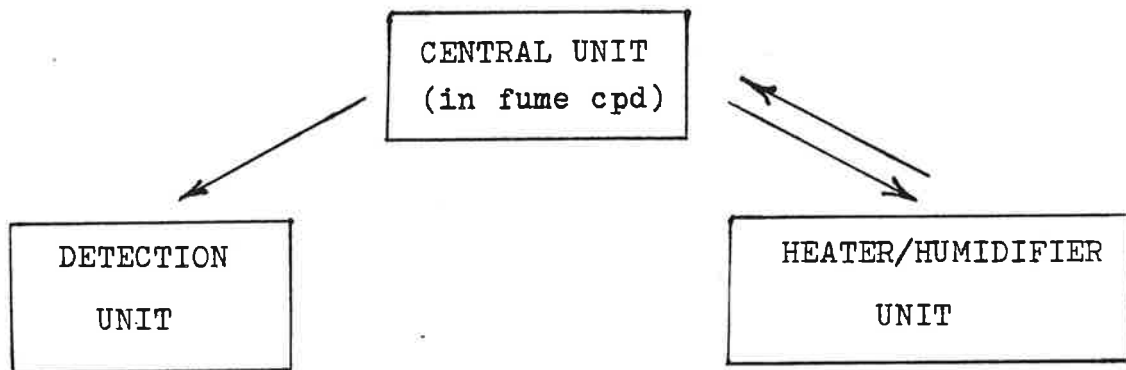


Figure 14 : System Units. S.A.H.C. Industrial Hygiene Laboratory Test Atmosphere Generator

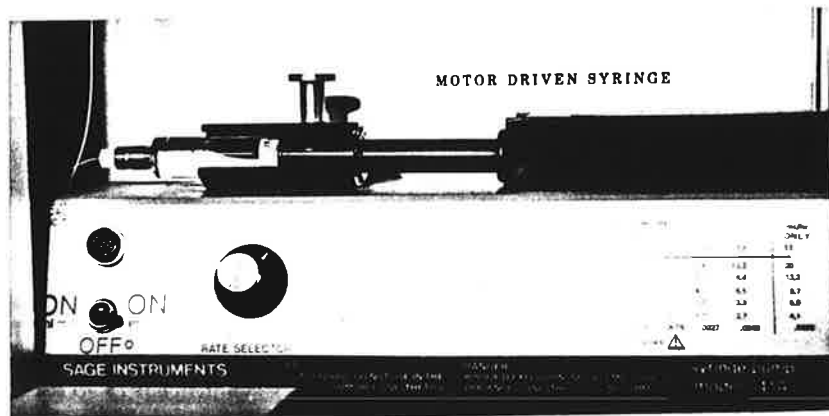


Figure 15 : Motor driven syringe injector

single dilution and double dilution of the test material is performed using rotameters. The atmosphere inside the exposure chamber is stirred by a pneumatic stirrer. A small portion of this atmosphere is typically diverted through a Miran infrared analyser. Voltage output, proportional to concentration, is then continuously displayed digitally, plotted and integrated.

B. COMPONENT DESCRIPTIONS

Motor driven syringe injector

A Sage Model 341A syringe pump is used for liquid and gas injection. A technical description is provided in Appendix 2.1.

Twenty one speeds may be selected which gives enormous flexibility in terms of injection rate. Only Hamilton gas tight teflon luer lock syringes have been used. Ground glass syringes leak noticeably.

Figure 15 shows this component in operation (with a 10ml syringe connected to 0.33mm i.d. teflon tubing).

A minor problem with teflon gas tight syringes is that the teflon piston skirt tends to stick to the glass barrel in an irregular way. This does not cause difficulties if a deviation of +/- 5% over a period of several hours is acceptable. Very slow syringe speeds may cause increased sticking and possibly other problems. The use of smaller

syringes will overcome most of the problems. Before a syringe is used for liquid injection, the interior should be wetted or "lubricated" with the liquid that is to be injected in order to reduce sticking.

Compressed air source

Air supplied from an *Ingersoll-Rand* compressor is first passed through a coarse particulate filter/water trap prior to fine filtration through a two stage *Domnick Hunter* filter. The elements remove particulates (down to 0.01 microns) and organic vapours. The compressor cuts in at 650 kPa and cuts out at 850 kPa. Pressure is then stabilized and reduced in three steps using regulators shown in Figure 16, Figure 17 (rear of central unit) and Figure 18. The relative humidity at room temperature (21 C) is between 5 and 10%.

Rotameters

Four rotameters are used in the system. Each have removable borosilicate glass tapered tubes with 316 stainless steel "plumb-bob" floats and integral needle valves. Stainless steel connection blocks and viton O-rings are used for those rotameters (2 and 4) measuring flows of diluted or pure test material. Calibration details are given in Section E and the physical arrangements are described in Section C.

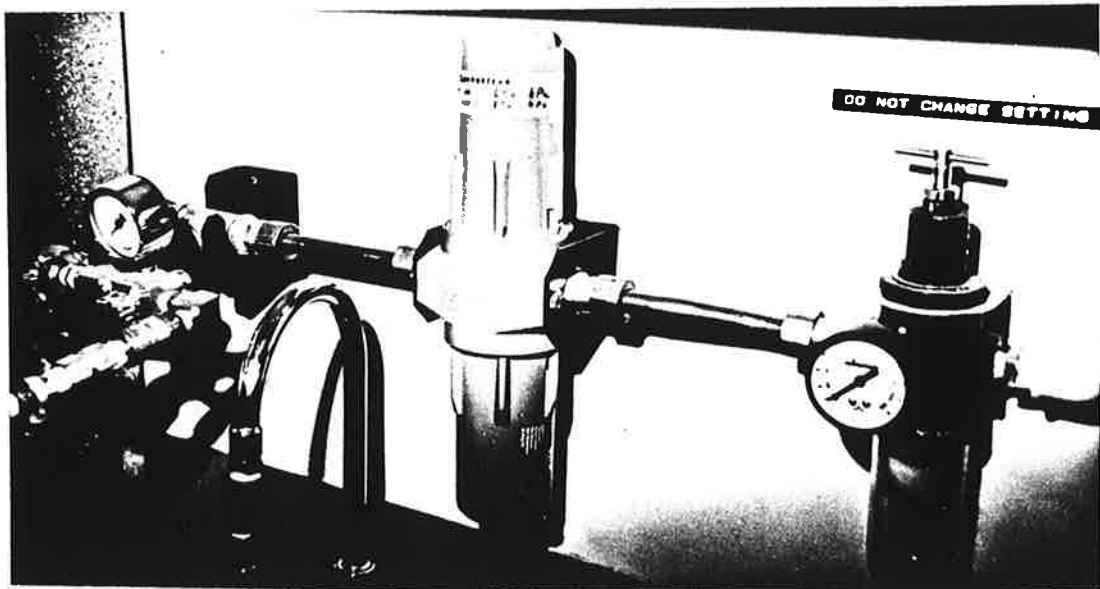


Figure 16: Purification of compressed air

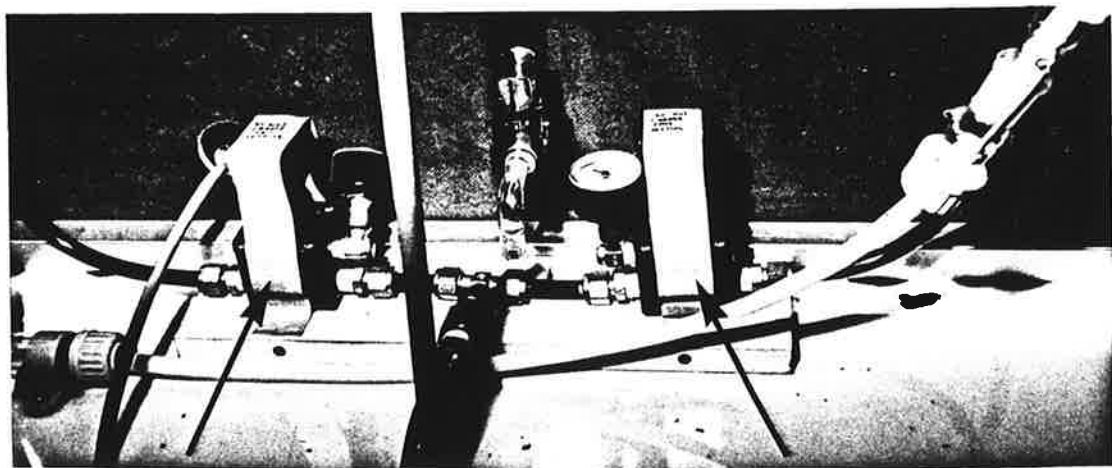


Figure 17 : High accuracy pressure regulators at rear of central unit

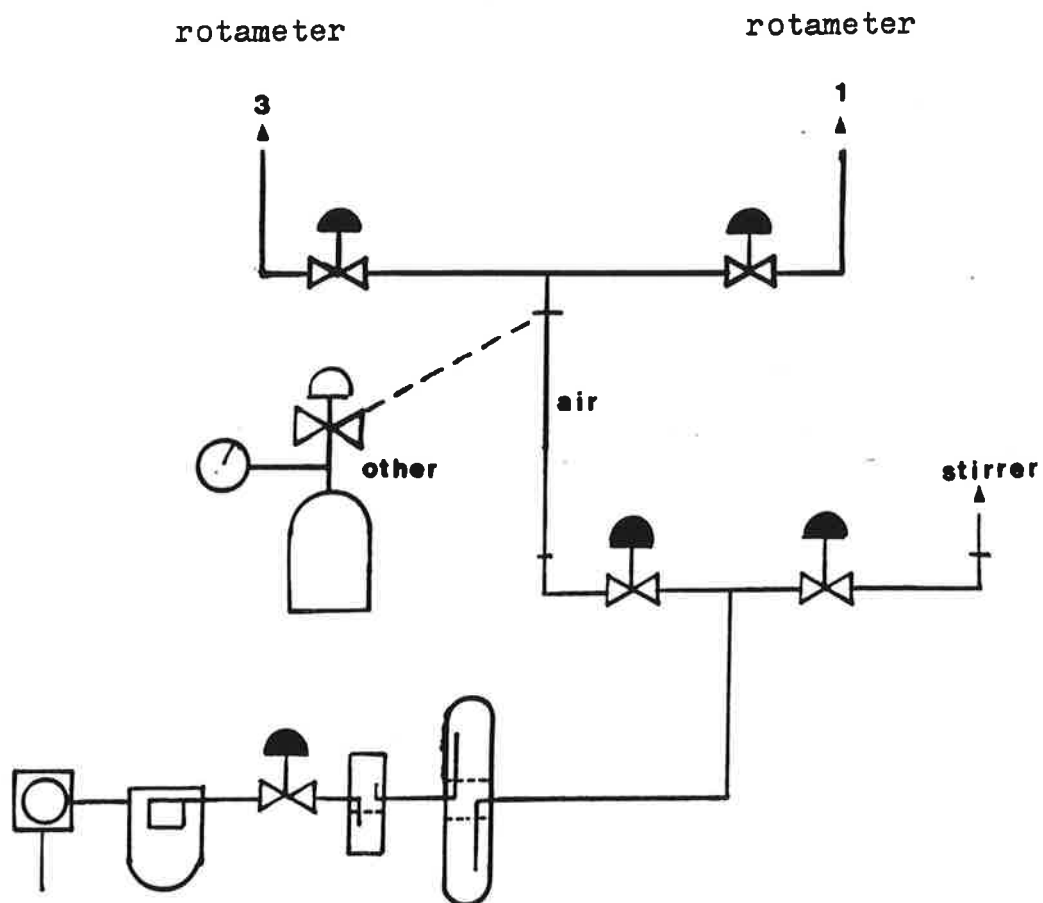


Figure 18 :

DILUENT GAS SUPPLY



= pressure regulator

= compressor

Mixing chambers

The primary and secondary mixing chambers are the same as those described in Appendix 2.3. Ball and socket joints are used to avoid strain and facilitate interchangeability of parts. Silicone grease is used to seal all ground glass connections.

Exposure chamber

The details of this component, taken directly from the HSE GCIS 3/1978, are given in Figures 19 and 20. Extensive use is made of screwthread joints (SQ13 and SQ18), which are suitable for connecting teflon or glass tubes (e.g. detector tubes, charcoal tubes etc.).

Figure 21 is a schematic diagram of the exposure chamber in normal operation. As might be expected, such complex glass apparatus is expensive to construct and repair. Less fragile arrangements are probably more practical. The chamber is supported on a wooden base and secured with large rubber rings.

Pneumatic stirrer

The stirrer is spark resistant and gives a continuously variable rotor speed which is indicated by a tachometer. The

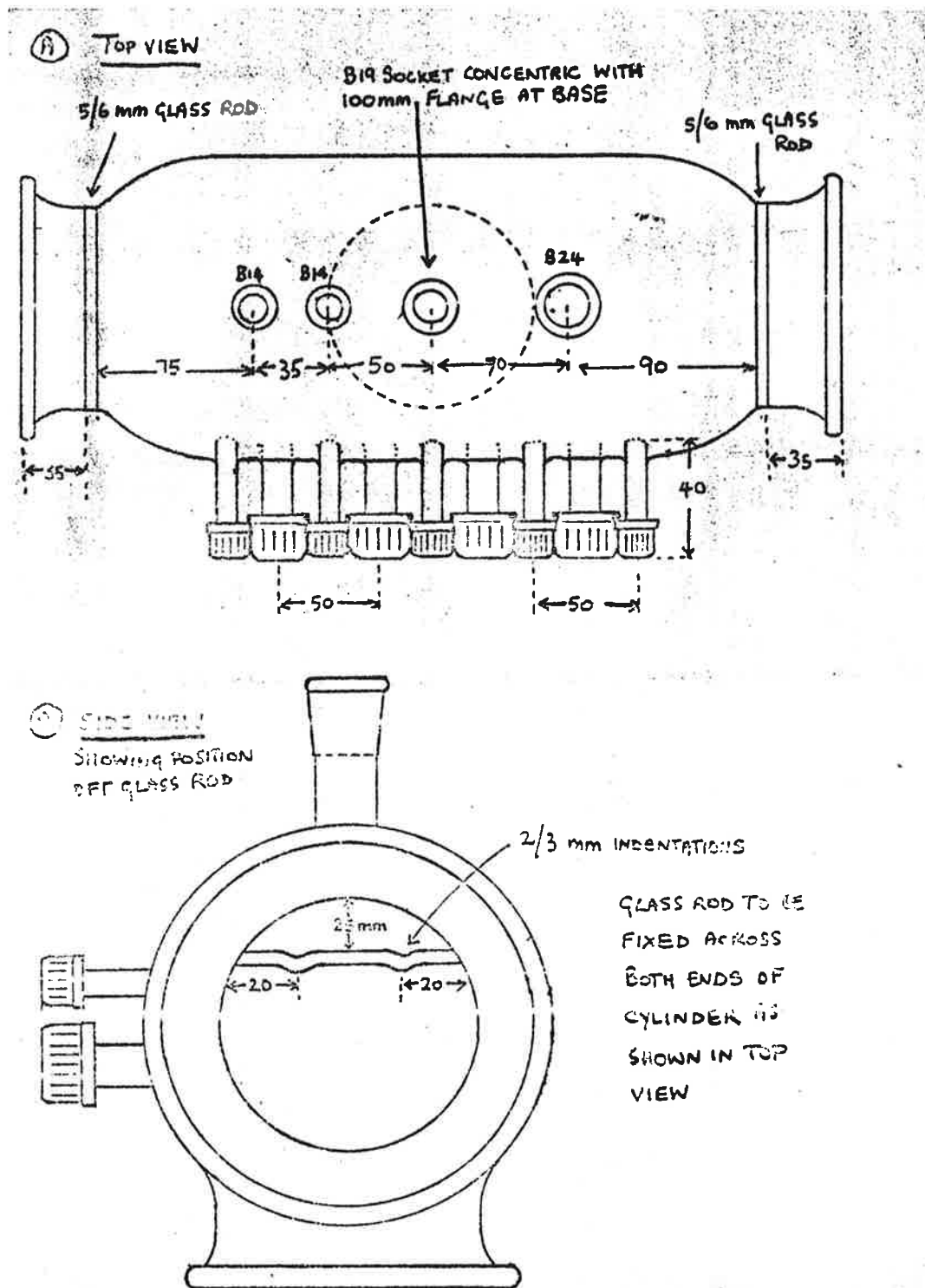
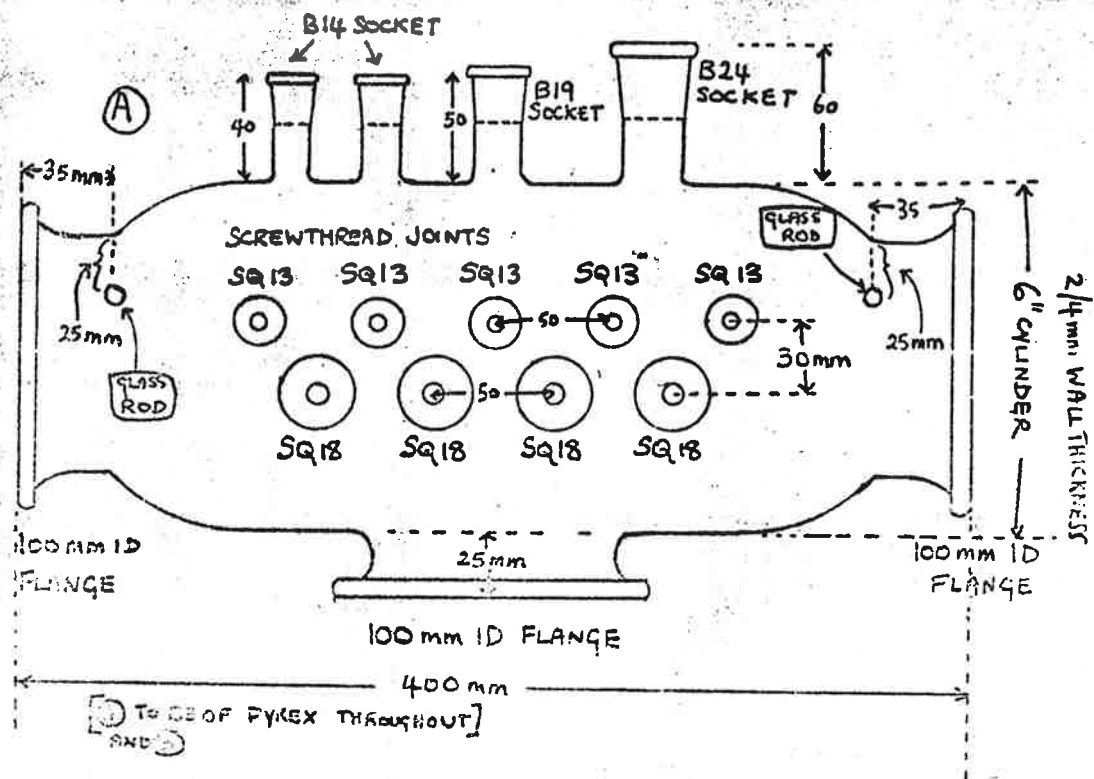
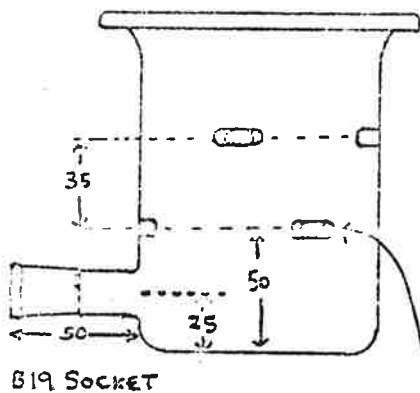


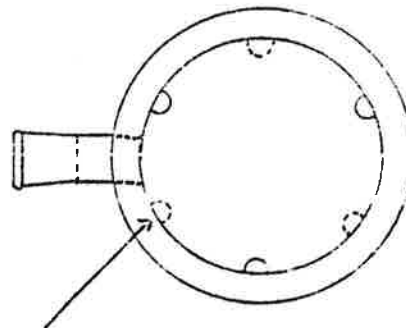
Figure 19 : Glass exposure chamber (HSE)



(B) FV-1L FLASK WITH 100 mm FLANGE



(C) TOP VIEW FV-1L FLASK



5/8 mm DIAMETER FLAT TOPPED LEDGES WELDED TO INSIDE WALL IN 2 LAYERS OF 3. 120 DEGREES APART BUT WITH THE 2 LAYERS STAGGERED BY 60 DEGREES WITH RESPECT TO EACH OTHER.

Figure 20 : Glass exposure chamber (HSE)

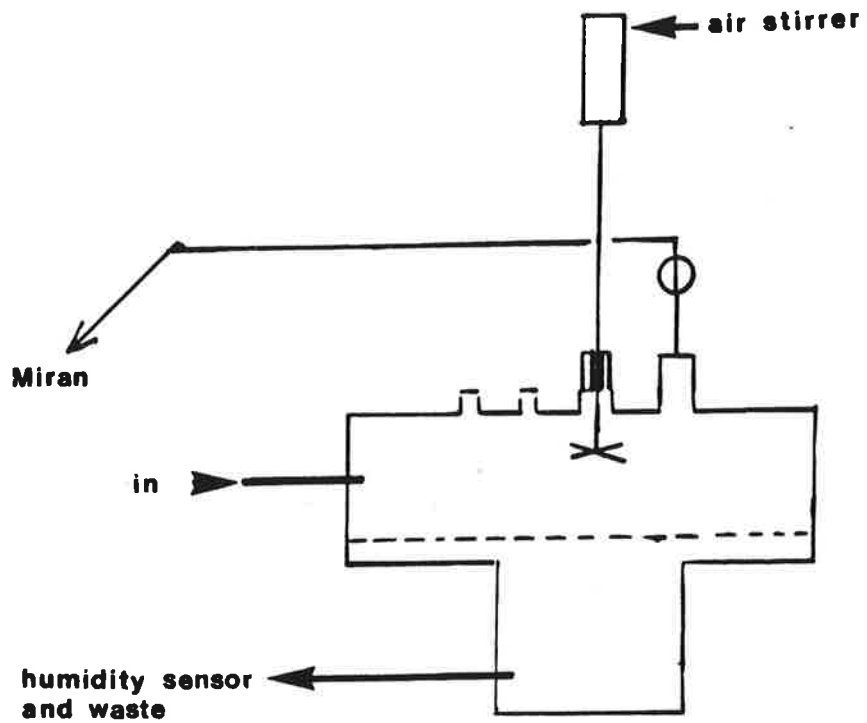


Figure 21 : **EXPOSURE CHAMBER**

stainless steel drive shaft is centred by a teflon gland (19/26 cone) as in Figure 22. The alignment of the shaft with respect to the glass socket is important since it is possible to break the glass exposure chamber by lateral movement. The pneumatic stirrer is supported by a retort rod fixed to the wooden frame which supports the chamber. This means that the glass chamber and stirrer cannot move independently, which might otherwise create strain.

Heater

a) Furnace

The resistance furnace (Figure 23) is regulated by a simmerstat control (Figure 24, right), i.e. it shuts off the power supply intermittently rather than controlling voltage. A 10 amp Variac is required to perform the latter.

Inside the cylindrical furnace cavity is a quartz tube with interior indentations in order to transfer heat more efficiently to the diluent gas flowing through it. The outlet side of the furnace is joined to teflon tubing via a tube fitting incorporating a viton O-ring. The connection is made only finger tight. This viton O-ring is rated to approximately 230 C but the system is capable of heating beyond that point.

Therefore, it is important to impose an upper limit on the furnace temperature. This is done via two methods:

- Simmerstat, which should be set at no more than 2.5
- Thermocouple in line

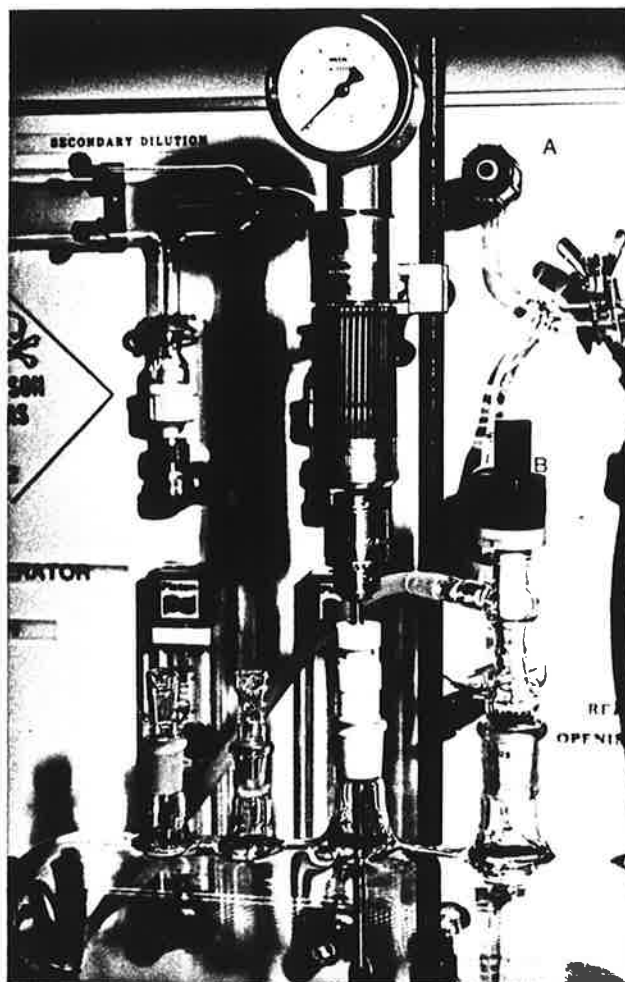


Figure 22 : Pneumatic stirrer with tachometer

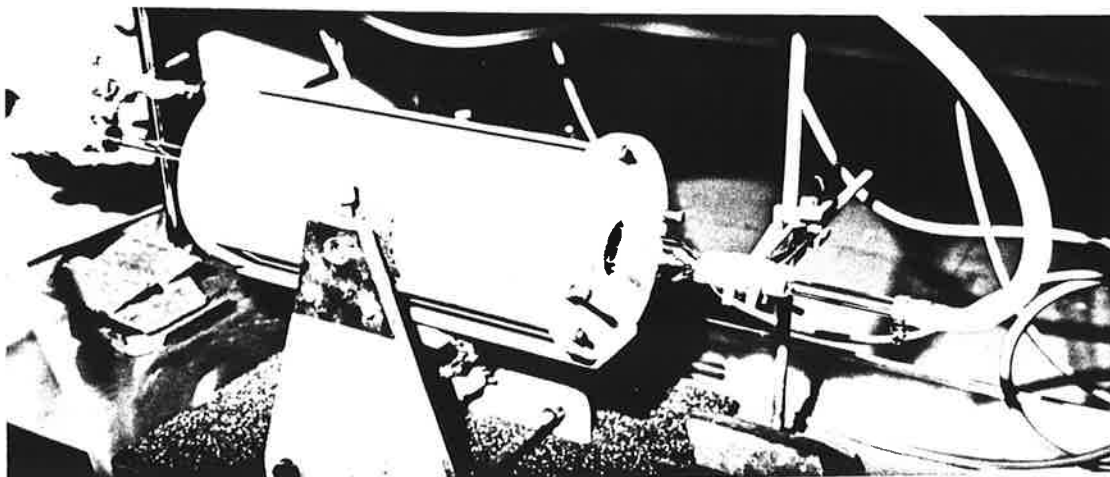


Figure 23 : Cylindrical furnace with quartz tube
in cavity



Figure 24 : Simmerstat controls for furnace (right) and heating tape (left)

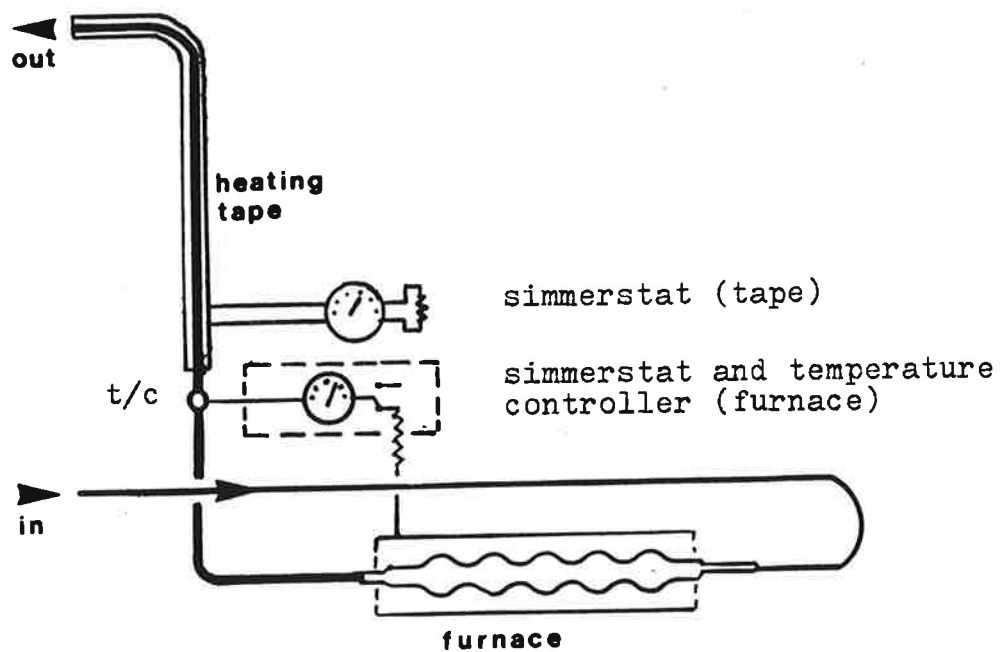


Figure 25 :

HEATER

The thermocouple acts as a sensor for the Shimaden Indicating Temperature Controller (Figure 25).

If the thermocouple temperature exceeds the preset value (100 C) the power to the furnace is cut off. Note that this is not a quick response - air cooling of the furnace is slow and, therefore, it is important not to set the simmerstat beyond 2.5.

b) Heating tape booster

This is required in order to maintain an injected gas stream temperature of >50 C since the thermal losses in the system are substantial, despite the use of kaowool rope and foam insulation around the teflon tubing. The heating tape temperature is controlled by a simmerstat (Figure 24, left) which should be set at no more than 1. Settings higher than 1 cause decomposition (with smell) of the foam insulation and high temperatures cause breakdown of the silicone rubber matrix of the heating tape.

Humidifier

Humidification of the diluent gas is achieved by using a split stream saturator which is depicted schematically in Figure 26. Taps D and E control humidity which is continuously sensed at the rear of the central unit using the KM-8004 humidity/temperature probe as in Figure 27. Note that relative humidity is displayed digitally on the front of the heater/humidifier unit. (A coiled cable

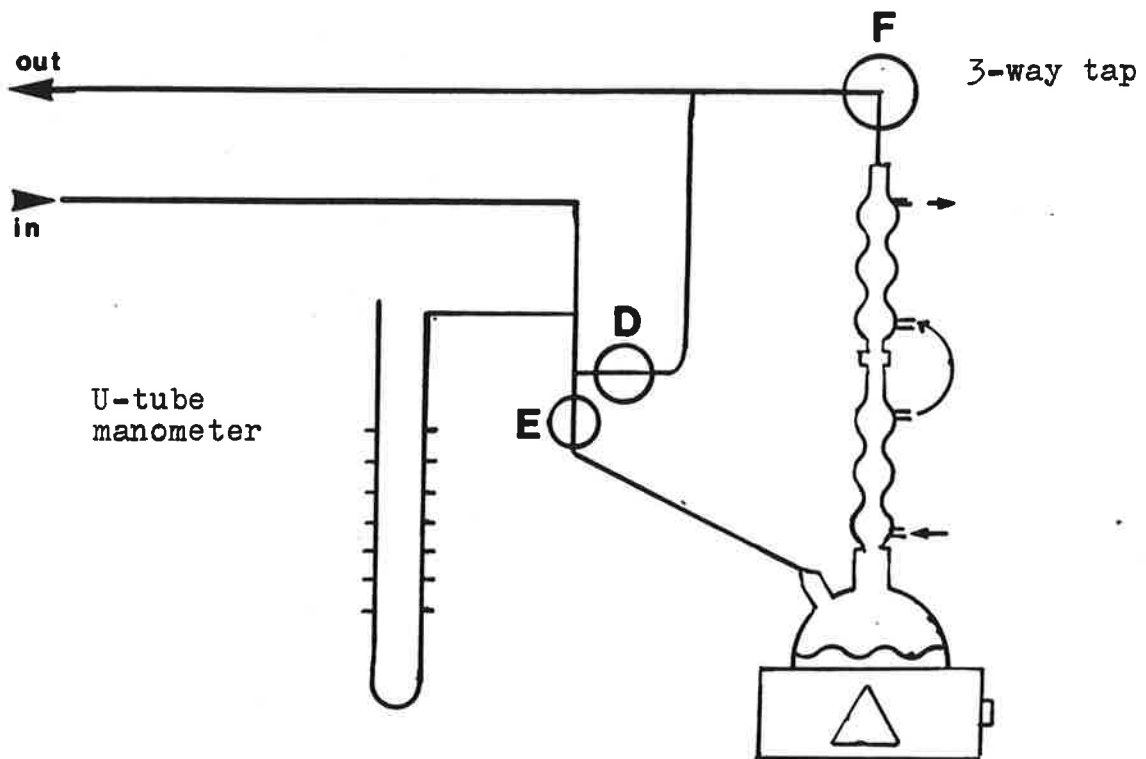


Figure 26 :

HUMIDIFIER

Split stream saturator

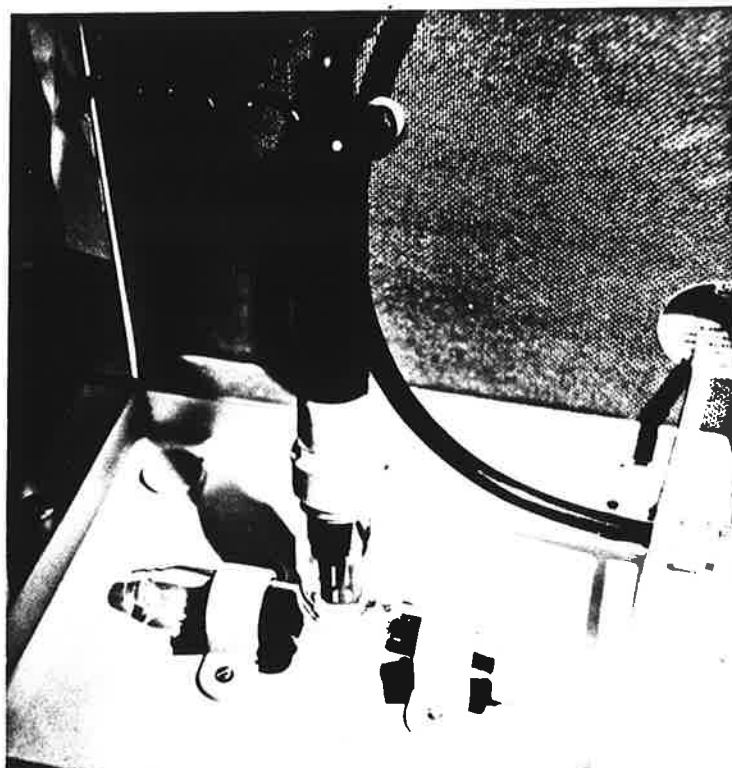


Figure 27 : Relative humidity sensor at rear of central unit

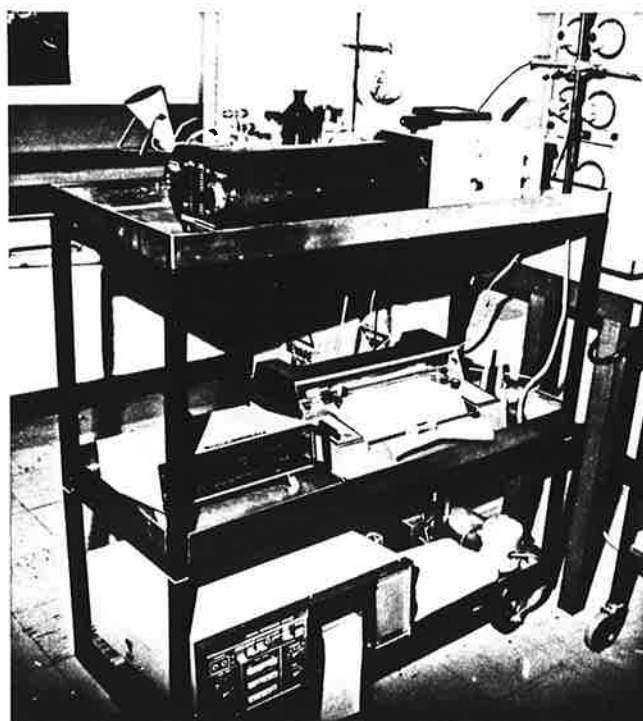


Figure 28 : Detection unit

provides the connection between the sensor and the display meter)

A heating mantle, with temperature control typically set at 3, provides heat for a flask containing distilled water. The temperature of cooling water for the condensers is relatively stable and slightly below ambient air temperature so that when tap D is closed completely, the outlet for the system provides nearly saturated diluent gas.

Backpressure is measured using a U-tube mercury manometer but the pressure corrections for rotameters have been small (typically 3% under worst conditions).

Detection unit

This comprises a Miran 1A infrared analyser, chart recorder, digital voltage display (0-1V) and integrator. As may be seen from Figure 28, the unit is mobile. Exhaust from the Miran is directed back into the fume cupboard.

C. OPERATING MODES

C.1. Single Dilution

The most common mode of operation is single dilution of test material and is depicted schematically in Figure 29. The gas mixture from the primary mixing chamber is required to pass through tap A. Tap B is closed. Note that the test material may be introduced either by syringe injection or by the use of rotameter 4. Figure 30 shows the (primary mixing chamber) inserts that are employed for syringe injection (right) and mixing using rotameter 4 (left). Figures 31 (rear of central unit) and 32 (front of central unit) show how the connection is made between rotameter 4 and the insert when rotameter 4 is used to introduce toxic gas. Figures 33 and 34 show that another teflon tube is used to connect the toxic gas source (gas cylinder) to rotameter 4.

When the gas mixture is hot (see Section C.4), there may be minor heating of the glass exposure chamber. To remove this heat, teflon tubing, connected to the condenser system (Figure 35, rear of heater/humidifier unit) may be interposed between the outlet of the primary mixing chamber and tap A as in Figure 36 (small arrows). The tube, marked "single dilution" is connected to the exposure chamber, whilst the tube marked "double dilution" is disconnected.

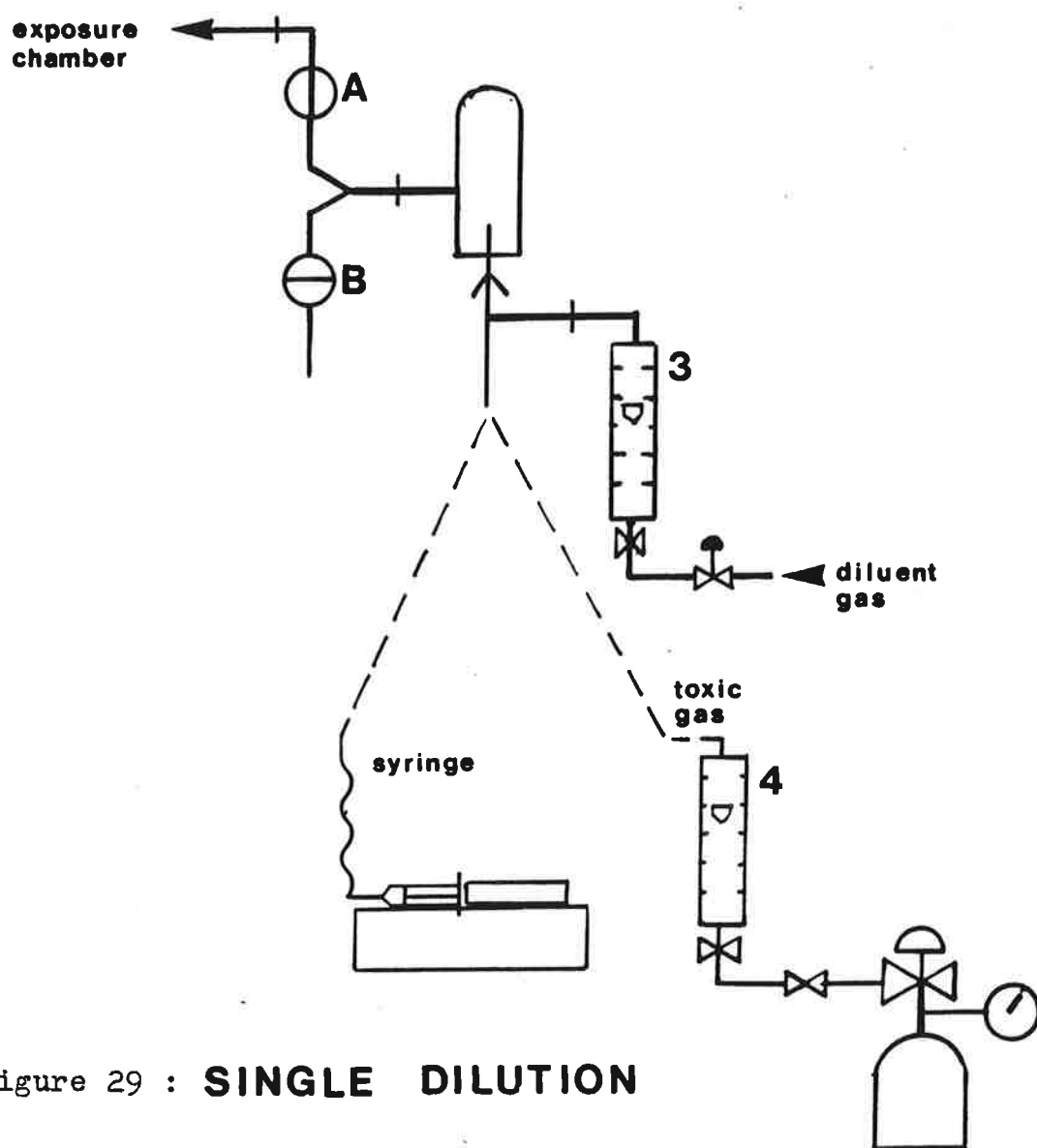


Figure 29 : **SINGLE DILUTION**

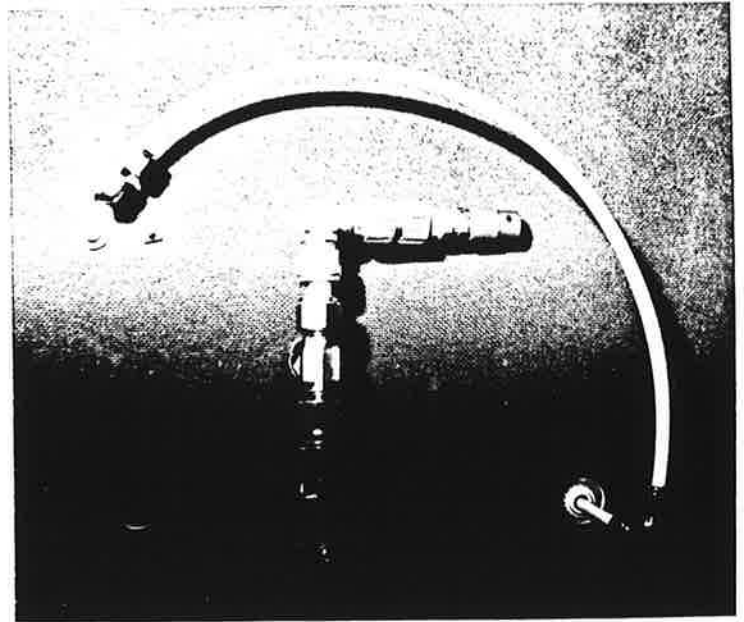


Figure 31

← Figure 30 : Primary mixing chamber inserts

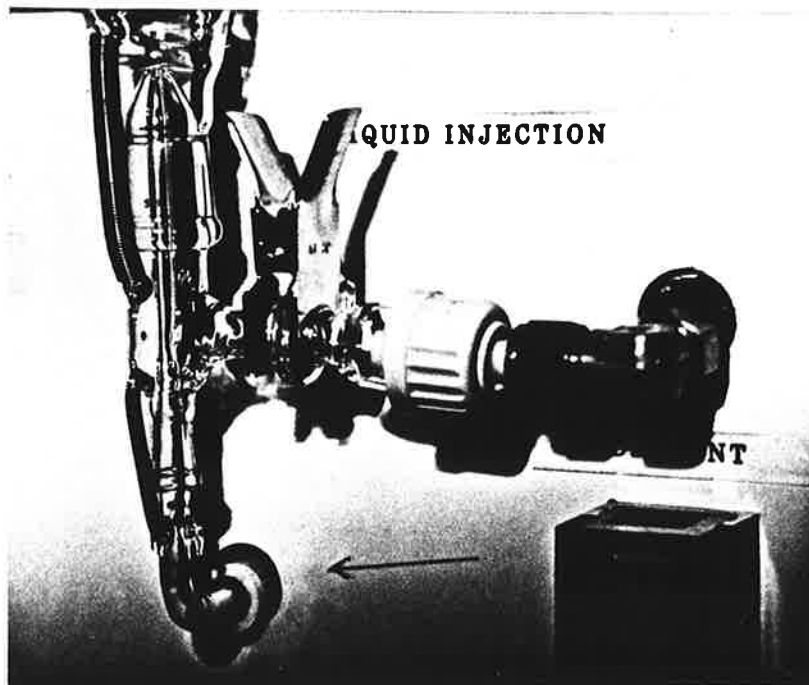


Figure 32 : Connections at front of central unit when toxic gas is introduced using rotameter 4



Figure 33 :
Toxic gas cylinder

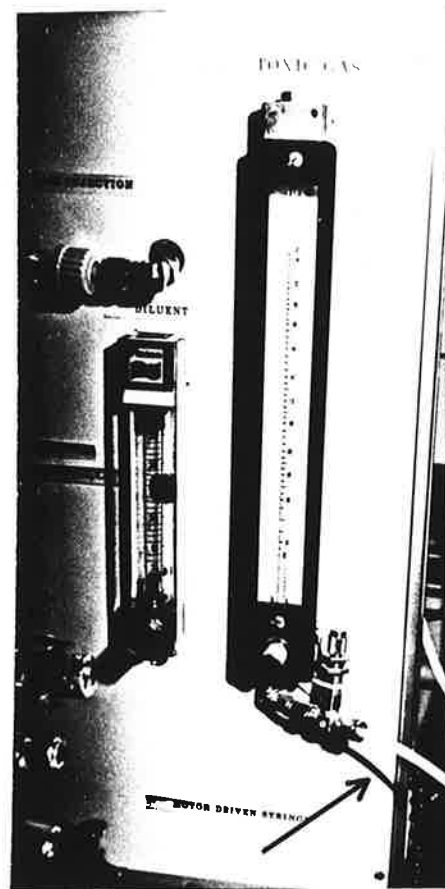


Figure 34 :
Front connection
to rotameter 4



Figure 35 :
Dual condensers for
cooling of gas stream

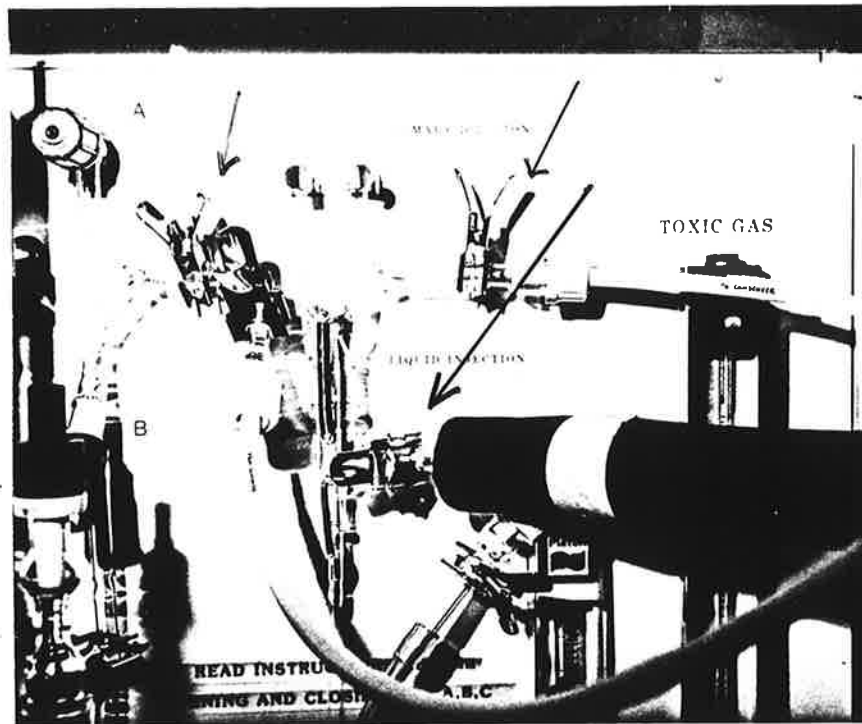


Figure 36 : Connections for heater and condenser

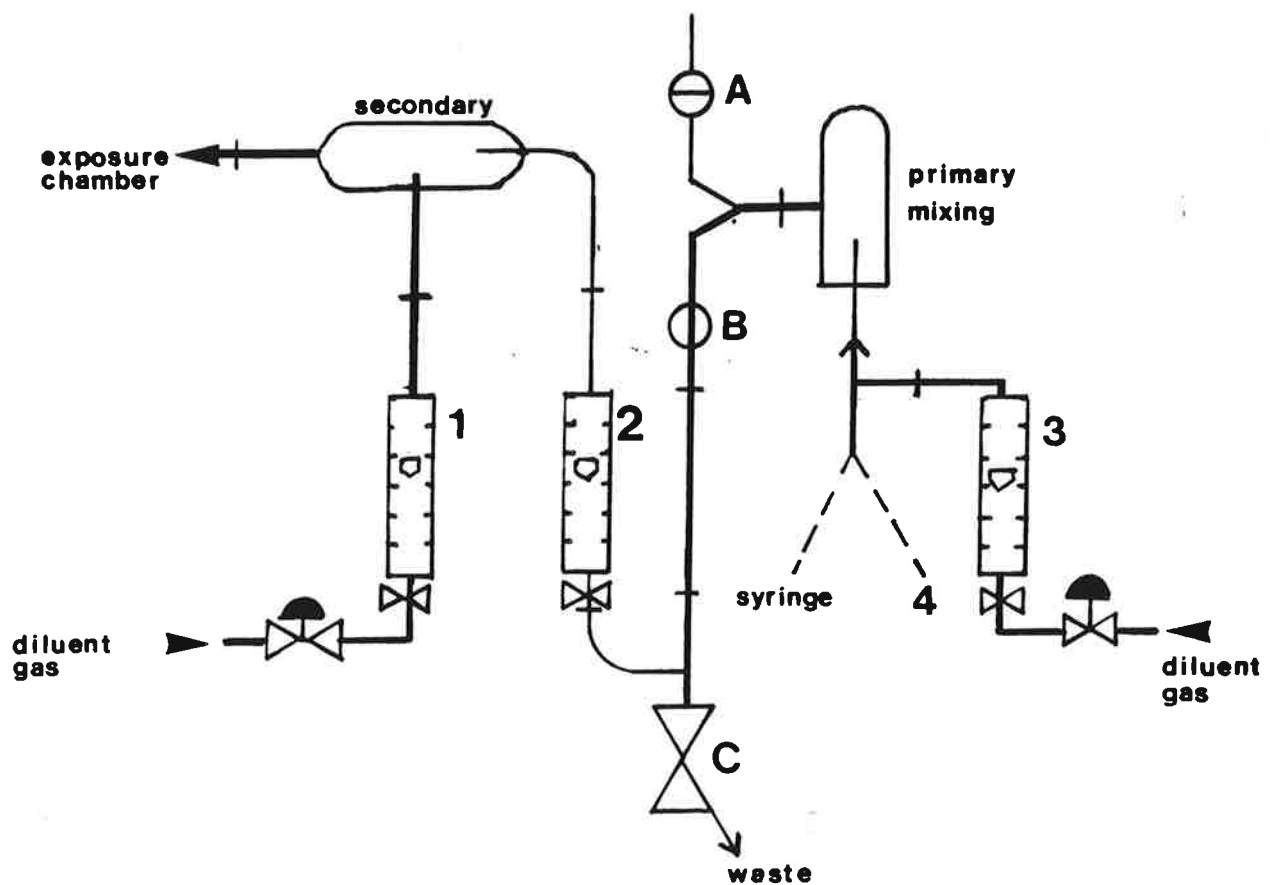


Figure 37 : **DOUBLE DILUTION**

Summary:

Prior to passing gas through system check that:

- (1) tap A is open and tap B is closed
- (2) if heater is on, that tubing leading to and from condenser is connected
- (3) tubing marked "single dilution" is connected
- (4) tubing marked "double dilution" is disconnected

C.2. Double Dilution

This is shown schematically in Figure 37.

The gas mixture is required to pass through tap B. Tap C is open (Figure 38) and tap A is closed.

When the gas mixture is hot, teflon tubing, connected to the condenser, needs to be interposed between the outlet of the primary mixing chamber and tap B as in Figure 36 (small arrows).

The teflon tubing marked "double dilution" is connected to the exposure chamber, whilst the tube marked "single dilution" is disconnected. The valve for rotameter 2 is opened fully.

At this stage, the valve for rotameter 3 is opened to allow diluent gas into the primary mixing chamber. Set this flow to approximately 15 litres/min. Slowly begin to close tap C, at the same time observing the float in rotameter 2. When the float (rotameter 2) rises to about one litre/min, partially close the valve (rotameter 2) until the value is

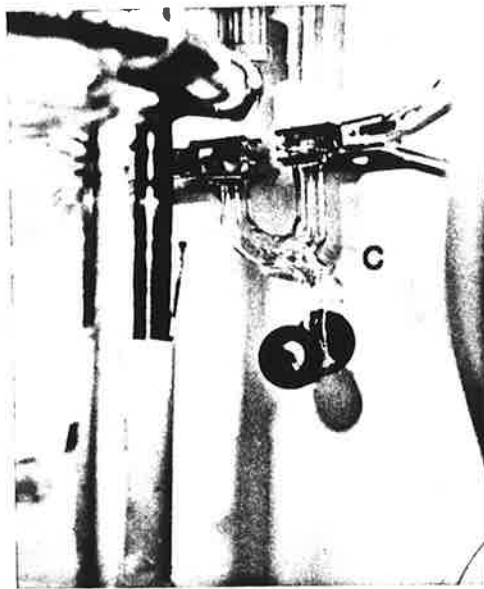


Figure 38 : Tap C

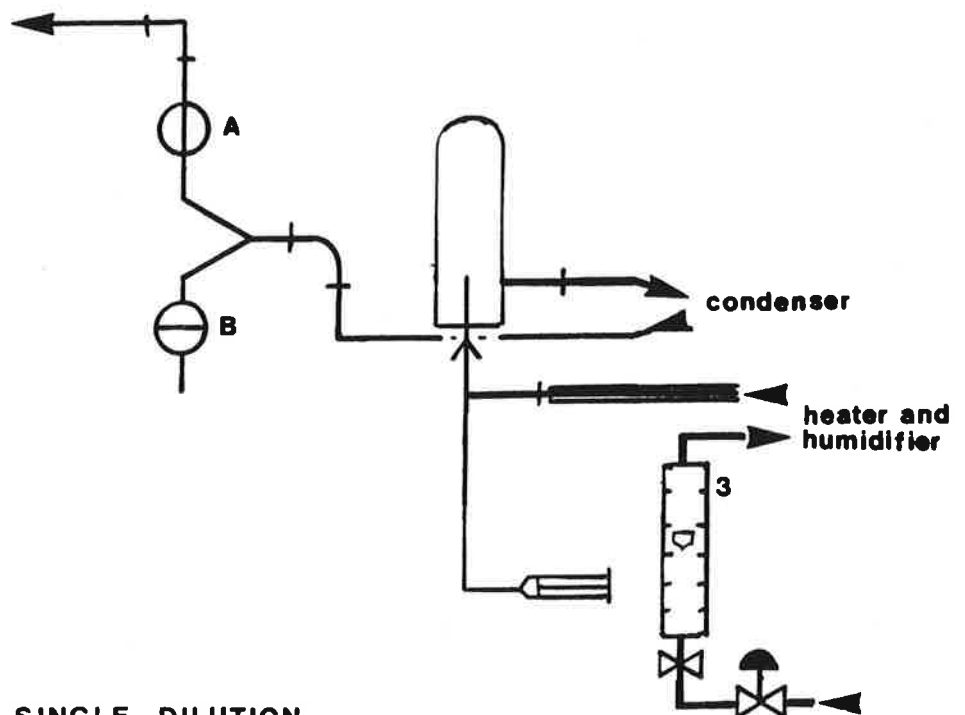


Figure 39 : **SINGLE DILUTION**
HEATING AND HUMIDIFICATION

approximately 700 ml/min. Open rotameter 1 and adjust flows of rotameters 2 and 3 by using their valves. Do not attempt to make adjustments using tap C, since complete closure of tap C will cause high internal pressures.

Prior to passing gas through system check that:

- (1) tap B is open, tap C is open and tap A is closed
- (2) if heater is on, that tubing leading to and from condenser is connected
- (3) tubing marked "single dilution" is disconnected
- (4) tubing marked "double dilution" is connected

C.3. Diluent Gas Humidification

This is represented in Figures 39 and 40. Humidification of diluent gas occurs by passage over hot water in a flask. Use of the humidifier requires approximately 30 minutes lead time in order to warm up water in the flask.

To start up humidifier (refer to Figure 26):

- (1) close tap E
- (2) open tap D
- (3) open tap F to room atmosphere
- (4) make appropriate connections (ball and socket joints) to outlet of diluent gas rotameter (diluent gas passes into humidifier) and inlet of mixing chamber (gas from

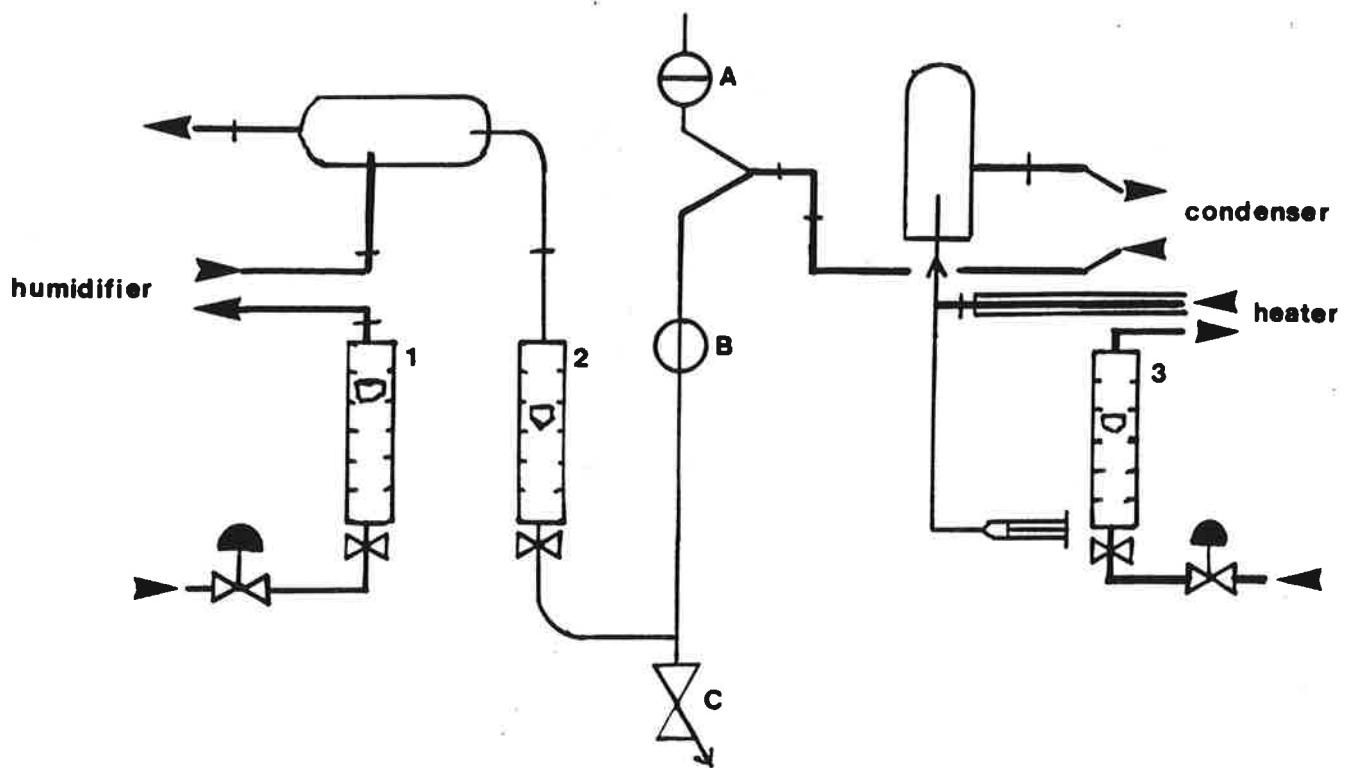


Figure 40 : **DOUBLE DILUTION**
HEATING AND HUMIDIFICATION

humidifier)

e.g. for single dilution, as in Figures 39 and 41

e.g. for double dilution, as in Figures 40 and 42

(5) switch on heating mantle (Figure 43) and set control to 3

(6) switch on water for cooling condensers (Figure 43)

(7) switch on humidity meter (Figure 44)

(8) after about 30 minutes, open tap E (figure 26)

(9) turn tap F so that it is closed to room atmosphere and open in the opposite direction, as in Figure 26

A nearly saturated diluent gas stream is obtained by closing tap D, with tap E open. Intermediate humidity can be obtained by partial closure of tap D, with tap E open (Figure 45). The driest conditions are obtained by having tap D open, tap E closed and tap F open to the room atmosphere.

Note that if both taps D and E are closed, gas pressure will be released through the U-tube manometer (Figure 26) possibly causing some liquid mercury to squirt out.

Water losses from the flask (by evaporation) are usually small. Replenishment of water to the flask can be accomplished by carefully pouring distilled water through the sidearm until the flask is about two thirds full. All of the glass joints should be "greased" with a small amount of silicone grease periodically to avoid minor leakages and sticking of glass.

The relative humidity value is displayed continuously by a KM 8004 humidity meter on the heater/humidifier board as in Figure 44. Output can be directed to a chart recorder. The relative humidity reading should be taken after several



Figure 41 :
Connection for single
dilution, humidification

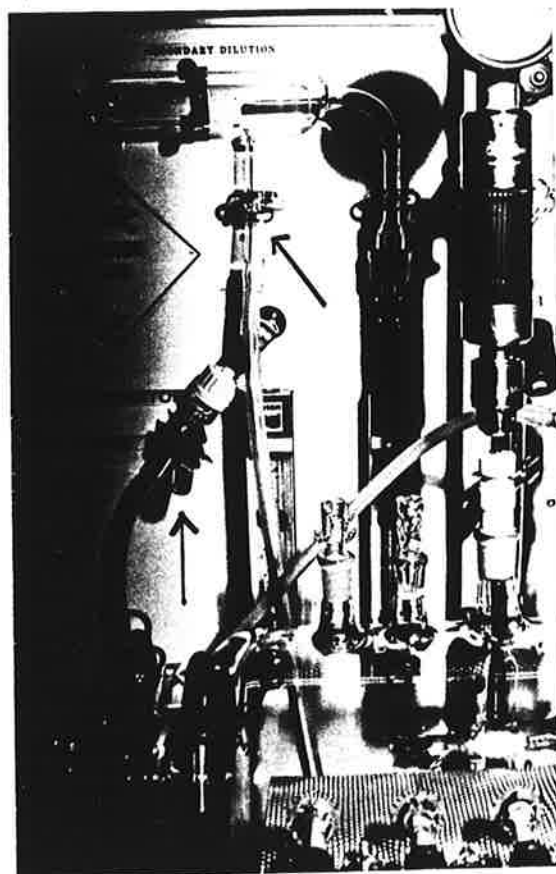


Figure 42 :
Connection for double
dilution, humidification

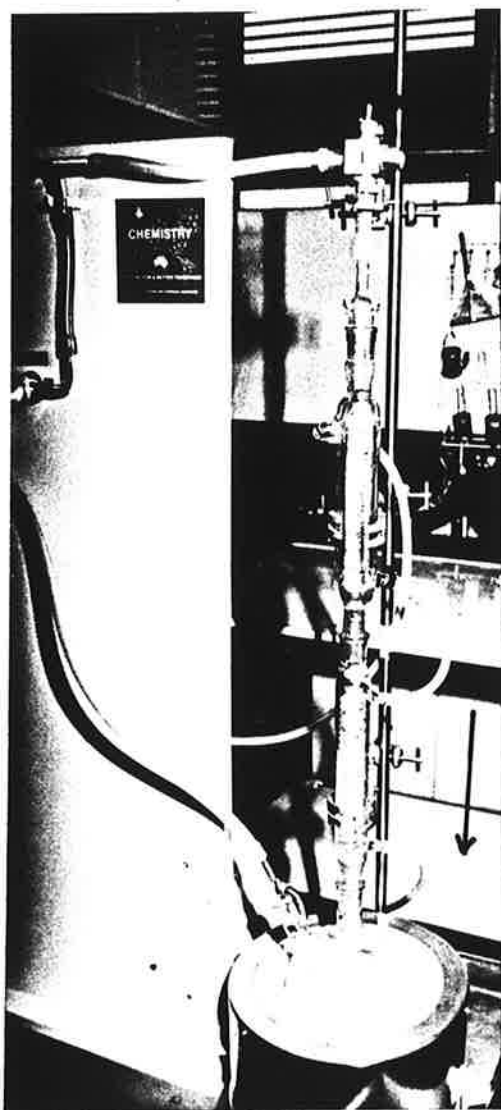


Figure 43 : Flask and condensers
for humidification



Figure 44 :
Humidity meter (top)
Temperature limiter (bottom)



Figure 45 :
Taps D and E
(Humidifier)

minutes and before any toxic gas or liquid vapour is introduced into the chamber, since some polar vapours will influence the capacitative sensor. Humidity values are usually quite stable over an 8 hour period. The sensor may need to be recalibrated approximately every six months and this can be done using reference humidity "bottles" (to adjust the "zero" and "span" potentiometers) according to the Kane-May 8004 Instruction Manual.

Prior to the removal of the central unit out of the fume cupboard, the coiled cable for the KM 8004 meter should be disconnected.

C.4. Diluent Gas Heating

Procedure (refer to Figures 39 and 40)

- (1) connect tubing marked "inlet to furnace" to rotameter output as in Figure 41 or to humidifier exit if humidification is required prior to heating.
- (2) connect large black insulated hose to primary mixing chamber as in Figure 36 (large arrow)
- (3) connect tubing marked "to condenser" to outlet of primary mixing chamber as in Figure 36 (small arrow, right)
- (4) connect tubing marked "from condenser" as in Figure 36 (small arrow, left). Note the use of the right angled glass connection.
- (5) turn on water for condenser
- (6) open or close taps A and B as necessary for single or

double dilution (Figures 39 and 40).

- (7) if prior humidification of gas stream is necessary, set up humidifier as indicated in Section C.3.
- (8) switch on simmerstats labelled "tape" and "furnace" (Figure 24), using the switches on the powerboard and the circular knobs. The furnace simmerstat should be set at no more than 2.5. The heating tape simmerstat should be set at no more than 1 (corresponding roughly to 100 C exit gas temperature). Higher values will cause decomposition of the foam insulation.
- (9) the temperature limiter for the furnace should be set at 100 C (see Figure 44, under humidity meter).

Note that the quartz tube is fragile and could be broken if the furnace is jolted in a vertical direction, up or down.

Finally, Figure 46 shows the arrangement (central unit) for syringe injection, double dilution with heating and humidification of diluent gas.

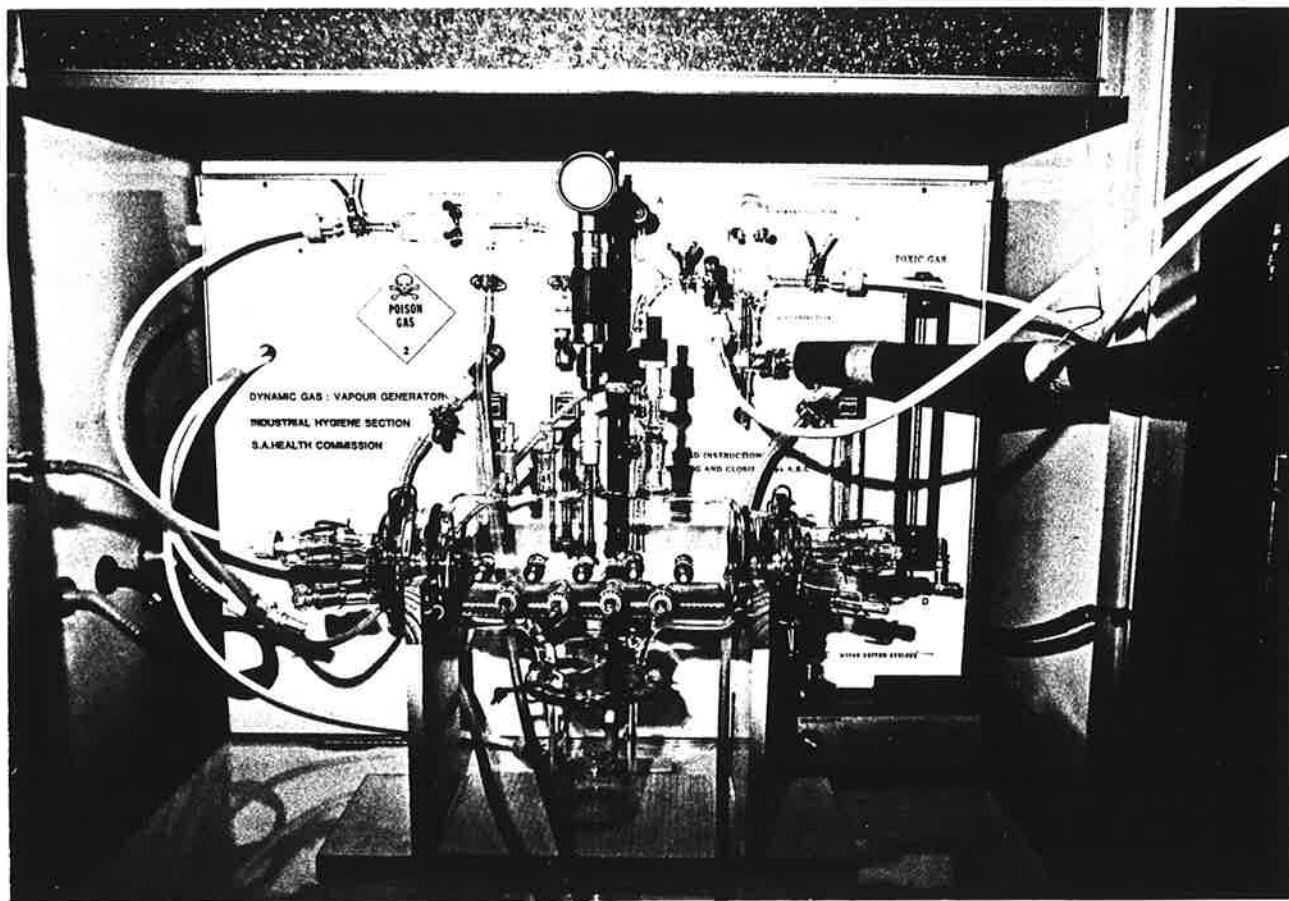


Figure 46 : The arrangement (central unit) for syringe injection, double dilution with heating and humidification of diluent gas

D. EXAMPLE SETTINGS

The rotameters used for this system are most accurate in their mid-range so that it is desirable to maintain flows in the following ranges:

Rotameter 1 (2-25 l/min) 10-20 l/min
Rotameter 2 (100-1200 ml/min) 400-1000 ml/min
Rotameter 3 (2-25 l/min) 10-20 l/min
Rotameter 4 (20-250 ml/min) 80-200 ml/min

The final gas output should be at least 10 litres/minute, which corresponds to changing the atmosphere in the exposure chamber approximately once per minute. This output is usually sufficient to render insignificant losses of test material due to passive monitors and charcoal tubes in the exposure chamber.

The most useful syringe pump settings, for exposure periods of several hours or more, are 3-10 (ml/hr) which represent a 20-fold range in injection rate (see Appendix 2.1).

Even with a limited choice of syringe sizes, say 1-10 ml, a 200-fold range is comfortably achieved. In fact, the syringe size/syringe pump setting combination provides excellent flexibility in the system.

Typically, operating boundary limits might be:

Syringe pump setting 3-10 (ml/hr)
Syringe size 1,5,10 ml
Rotameters 1,3 10-20 l/min
Rotameter 2 400-1000 ml/min
Rotameter 4 80-200 ml/min

NOTES:

- (1) Rotameter 4 is not used if the syringe pump is used and vice versa
- (2) If heating of the diluent gas is necessary for improved liquid evaporation, then this is done after gas has passed through rotameter 3. Hence, rotameter corrections for temperature are irrelevant.
- (3) If humidification of diluent gas is required, then this is also done after the (dry) diluent gas has passed through the rotameters (1 or 3).

However, in this case, water vapour contributes to the total volume flow and must be accounted for:

Fully saturated air (100% r.h.) at 21 C and 760 mm Hg has 24,500 ppm of water vapour. Therefore, the maximum increase in flow relative to dry air at this temperature is 2.45%. At 50% r.h. (21 C, 760 mm Hg), the increase is 1.22% and so the final concentration of test material is reduced by approximately 1%.

EXAMPLE 1 : Syringe injection of a pure volatile liquid

- (a) 200 ppm toluene in air (5-10% r.h. at room temp (21 C) and pressure (760 mm Hg))

Procedure (one of several that could be used)

Choose a suitable injection rate considering the volatility of toluene e.g. 1 ml/hr. This conveniently corresponds to a syringe pump setting of 7 (ml/hr) with a 10 ml syringe (see Table 2, Section E).

Required air flow rate (litres/min) at 21 C is:

$$\approx \frac{\text{dens(g/ml)} \times \text{mol vol. (l)} \times \text{inj. rate(ml/hr)} \times 1000000}{\text{mol wt.} \times \text{ppm} \times 60}$$

$$\frac{0.865 \times 24.13 \times 1.009 \times 1000000}{92.14 \times 200 \times 60} = 19.0$$

This is within the range for single dilution. The required flow of 19.0 l/min is obtained if rotameter 3 is set to 18.4 l/min (see Figure 50, calibration of rotameter 3)

Note :

- (1) Always check to see that the liquid is evaporating smoothly, without spattering on the walls of the primary mixing chamber
- (2) If there is a significant backpressure (p mm Hg) then
true flow = indicated flow $\times \sqrt{(760+p)/760}$

(b) 5 ppm toluene in air (50% r.h. at room temp (21 C) and pressure (760 mm Hg))

Required air flow (l/min) for 1 ml/hr injection rate (10 ml syringe) is

$$\approx \frac{0.865 \times 24.13 \times 1.009 \times 1000000}{92.14 \times 5 \times 60} = 762 \text{ l/min}$$

Therefore, double dilution is necessary.

From example (a) 200 ppm was achieved using a 10 ml syringe with pump speed 7 (ml/hr) and rotameter 3 set to 18.4 l/min. A further 40-fold dilution is required so that the ratio of flows (rotameter 1:rotameter 2) = 39:1. Since rotameter 1 flows should be within the range 10-20 l/min and rotameter 2 flows should be within the range 0.4-1 l/min, a suitable

flow ratio is 19.5:0.5 (l/min).

However, at 50% r.h. final concentration would be diluted by approximately 1% because of water vapour. Hence, flow ratio should be 38.6:1

i.e. rotameter 1: 19.3 l/min

rotameter 2: 0.5 l/min

Final settings (see Figures 48-50)

Rotameter 1: 18.6 (actual 19.3) l/min

Rotameter 2: 460 (actual 500) ml/min

Rotameter 3: 18.4 (actual 19.0) l/min

Syringe pump speed 7 (ml/hr) with 10 ml syringe.

Note: If the backpressure for rotameter 1 or rotameter 3 is greater than 1" Hg then an appropriate pressure correction needs to be made.

EXAMPLE 2 : Toxic gas dilution

2 ppm ethylene oxide in air (5-10% r.h. at room temp (21 C) and pressure (760 mm Hg)) from a gas cylinder containing 15000 ppm ethylene oxide in nitrogen

Dilution factor is 7500, hence double dilution is required

A suitable combination of rotameter settings would be:

Rotameter 1: 19.2 (actual 20.0) l/min (see Figure 48)

Rotameter 2: 460 (actual 500) ml/min (see Figure 49)

Rotameter 3: 14.2 (actual 14.6) l/min (see Figure 50)

Rotameter 4: 82 (actual 80) ml/min (see Figure 51)

E. CALIBRATION OF SYRINGE PUMP AND ROTAMETERS

The syringe pump settings and individual syringes are calibrated as follows:

The gas tight syringe is filled with distilled water and placed in the syringe pump holder. A pump flow rate is selected and water is collected in pre-weighed (small) beaker for a sufficient period (5-30 minutes). Ambient temperature should be stable and measured during the period. The approximate evaporation rate of water (mg/min) in the beaker should be established beforehand. The weight of water collected (corrected for evaporation to 0.1 mg) is then used (along with the density of water at the ambient temperature) to determine true flow rate (ml/hr) for each syringe pump setting.

Sample calibrations are given in Tables 1 (5ml syringe) and 2 (10 ml syringe) and these correspond well with the flow rate chart printed on the syringe pump.

Each rotameter is calibrated using either a soap bubblemeter (Figure 47) or, in the case of flows greater than 5 litres/minute, with a pre-calibrated dry gas meter (Parkinson Cowan).

In this procedure, small backpressures (up to 1" Hg) are set up (corresponding to those which are encountered in the system for syringe injection).

Rotameter corrections for pressure, temperature and gas density are described in references 2 and 15. Sample calibrations are given in Figures 48-51.

Each measurement needs to be done at least in duplicate.

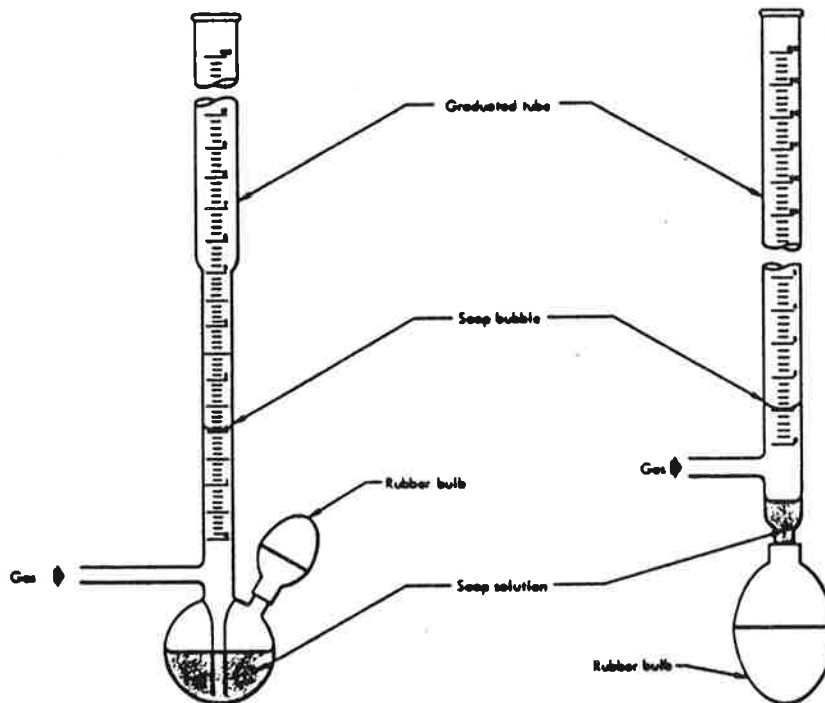


Figure 47 : Two kinds of soap bubblemeters for measuring low gas flows

TABLE 1: Calibration of 5 ml Hamilton syringe (Temp = 21 C)

<u>Setting (ml/hr)</u>	<u>ml/hr</u>
4	0.165
5	0.242
6	0.360
7	0.496
8	0.745
9	1.235

TABLE 2: Calibration of 10 ml Hamilton syringe (Temp = 21 C)

<u>Setting (ml/hr)</u>	<u>ml/hr</u>
2	0.137
3	0.190
5	0.489
7	1.009
8	1.506
9	2.49
10	3.940

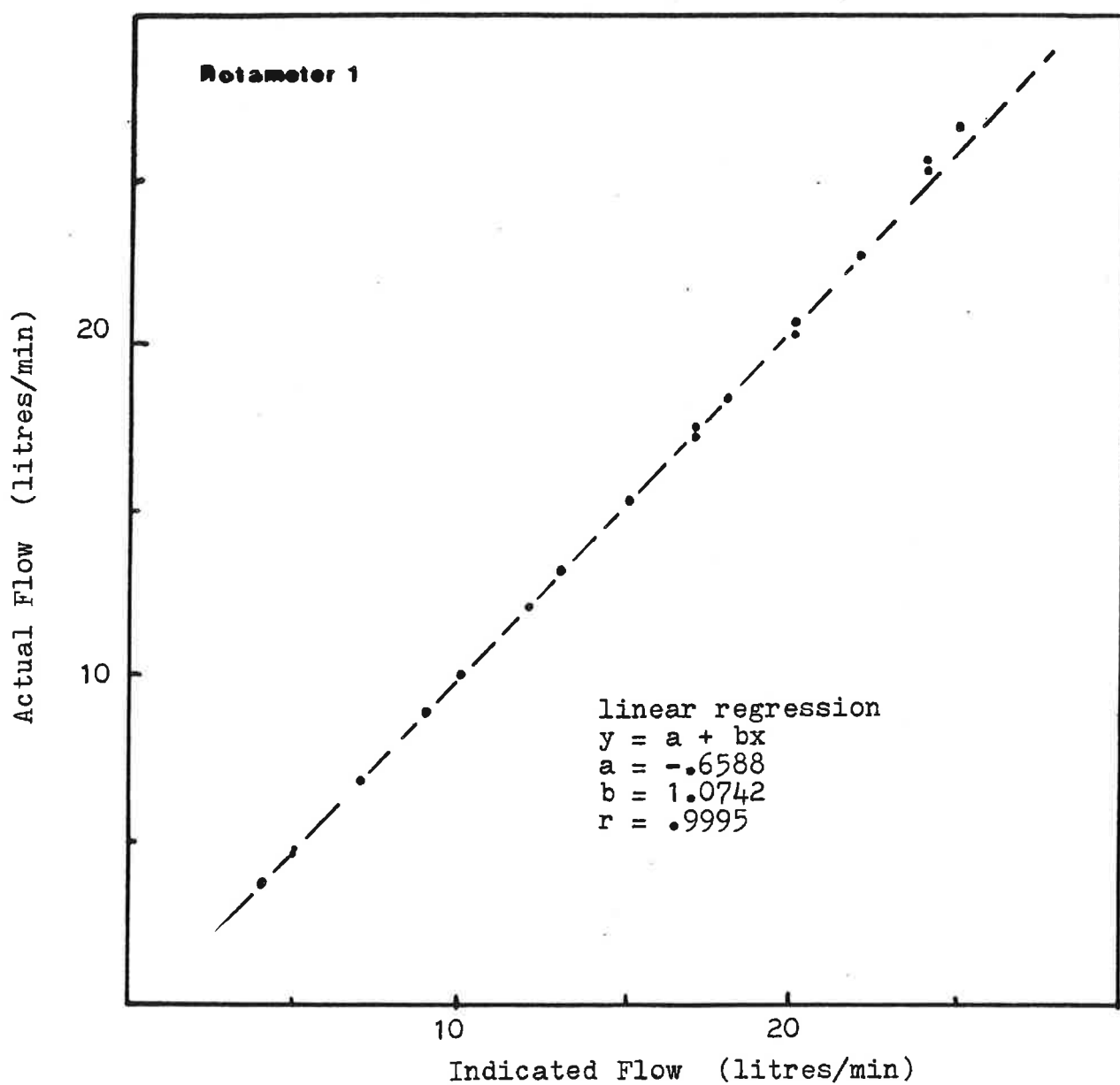


Figure 48 : Rotameter 1 calibration
Operating conditions: air (5-10% r.h. @ 21 C)
 $\frac{1}{2}$ " Hg backpressure, dry gas meter/bubblemeter

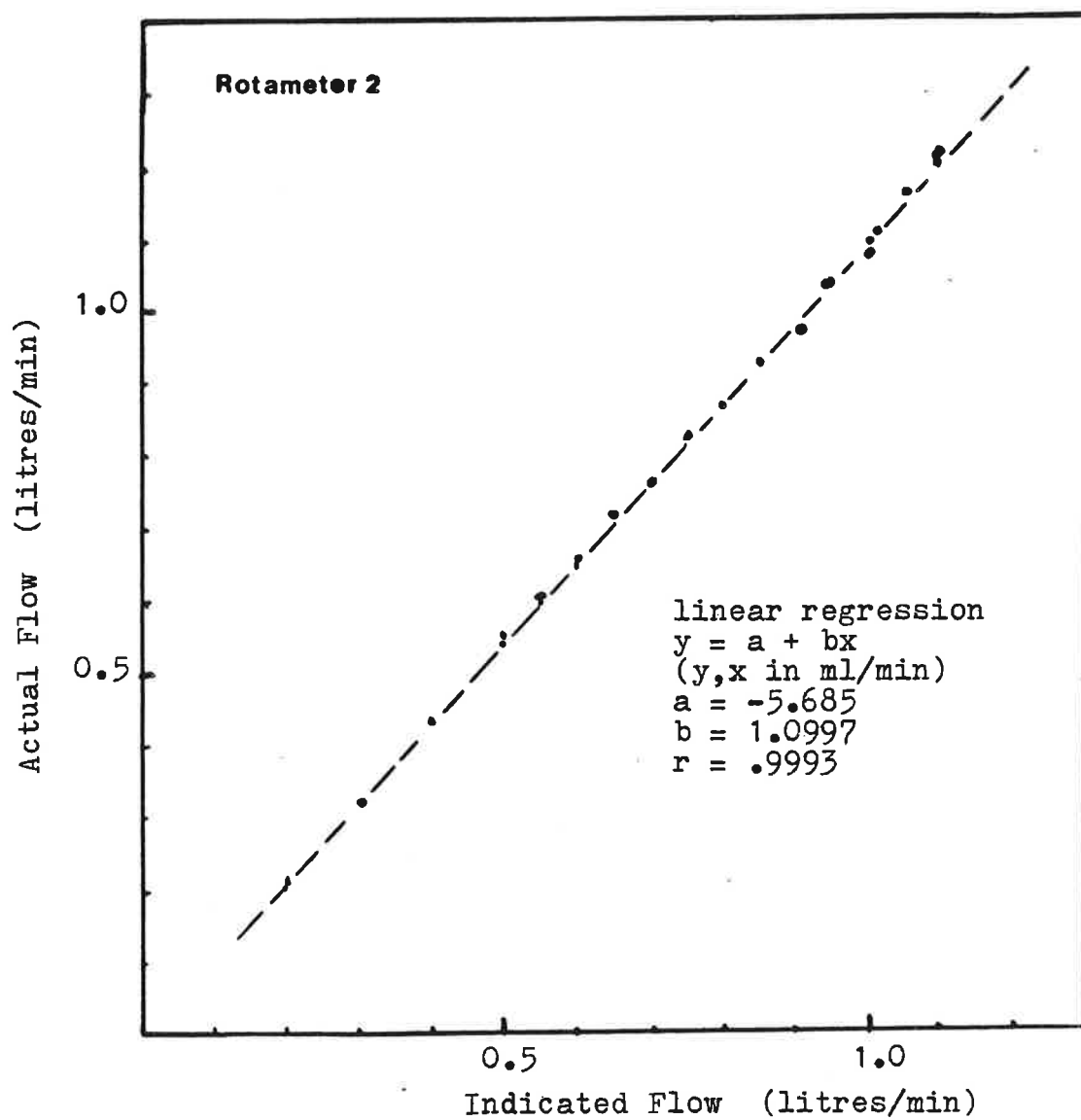


Figure 49 : Rotameter 2 calibration
Operating conditions: air (5-10% r.h. @ 21 C)
 $\frac{1}{2}$ " backpressure, soap film bubblemeter

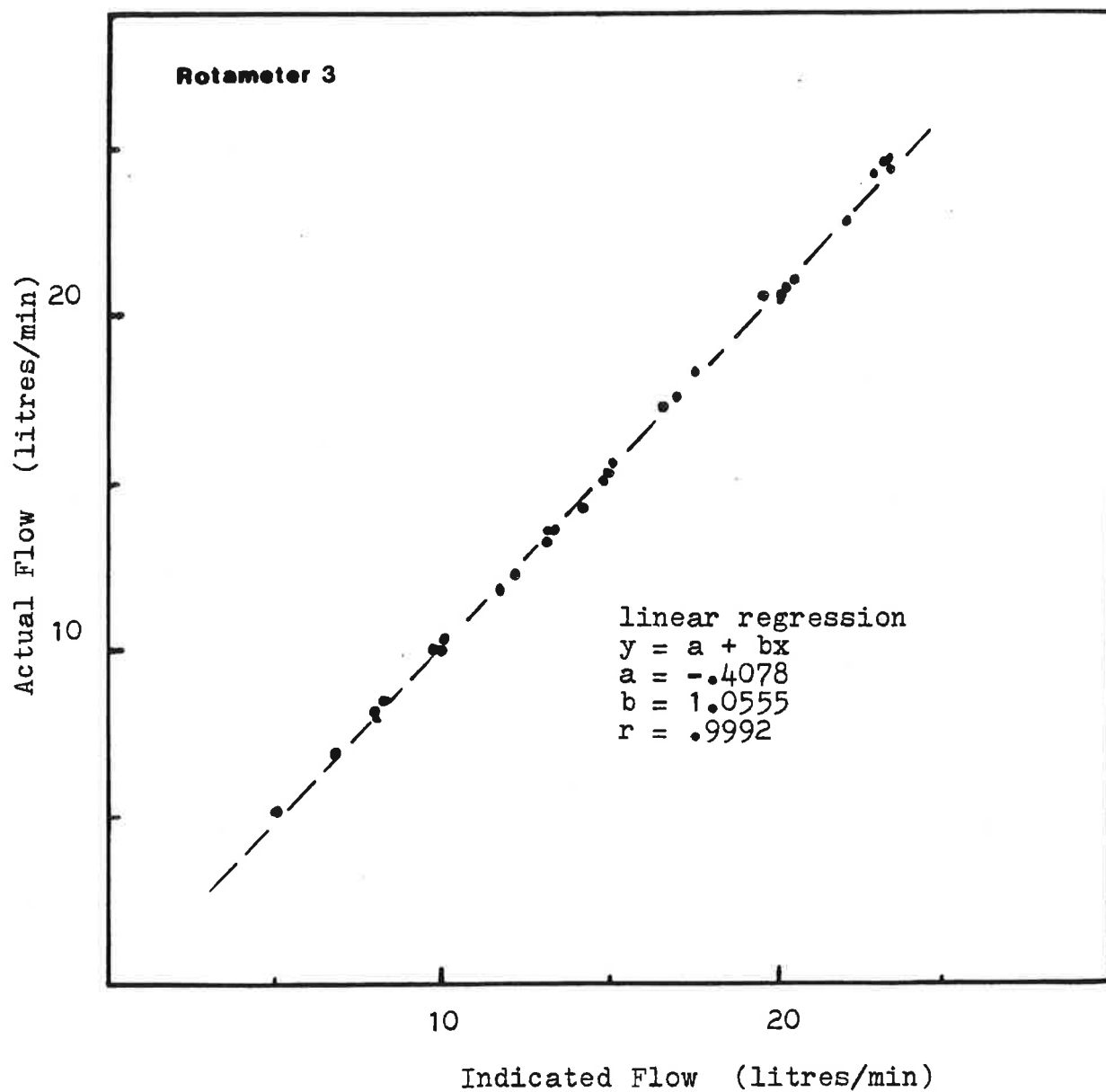


Figure 50 : Rotameter 3 calibration
Operating conditions: air (5-10% r.h. @ 21 C)
 $\frac{1}{2}$ " backpressure, dry gas meter/bubblermeter

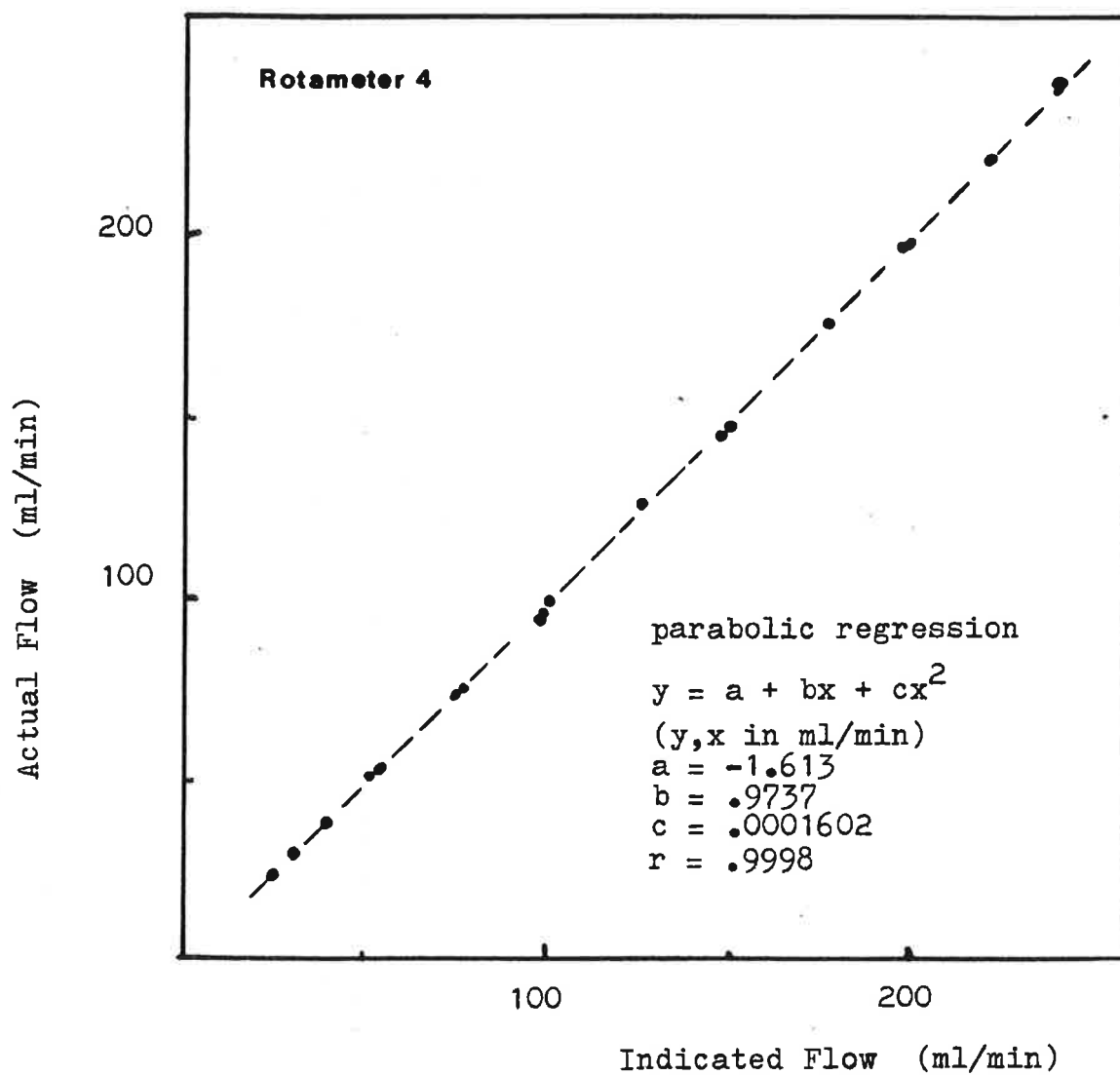


Figure 51 : Rotameter 4 calibration
Operating conditions: air (5-10% r.h.
@ 21 C) soap film bubblemeter

F. PARTS LIST (major items only)

a) Central Unit and Compressed Air Supply

Glassware

- * Exposure chamber (Figures 19,20)
- * Mixing chambers (Appendix 2.3).
- * Rotaflo taps HP-2/10, HP-6/10 (Taps A,B,C, Figure 36)
- * Various Quickfit screwthread joints, ball and socket joints, clamps, springs, clips, cones, sockets.

Tubing

- * Teflon 10 mm o.d., 8 mm i.d.
- * Teflon 1/4" o.d., 1/8" i.d. (for toxic gas)
- * Teflon .031" o.d., .013" i.d. (for syringe, with CTFE luer connector)
- * Nylon 3/8" o.d. (diluent gas)

Metal fittings

- * Various 3/8", 1/4", 10 mm s/s and brass (diluent gas only) tube fittings, 1/4" BSP pipe fittings
- * Nupro B-4CPA2-3 relief valve (diluent gas) 3-50 psi nominal cracking pressure range.

Stirrer assembly

- * PLR 31 T stirrer with tachometer
chrome plated chuck, s/s fly-vane stirrer blade 1352
- * Quickfit PTFE gland (19/26 cone) ST 3/2 F

Rotameters

- * Platon GTV rotameters
2-25 l/min s/s float (rotameters 1 and 3)
100-1200 ml/min s/s float, body, valve, viton O-ring (rotameter 2)
- * Fisher series 1100 flowmeter
20-250 ml/min, s/s float, body, valve, viton O-ring (rotameter 4, toxic gas)
- * Nupro SS-4MG-TFE metering valve (for rotameter 4)

Syringe pump

- * Sage Instruments model 341A pump (Figure 15)
- * Hamilton 1001, 1005, 1010 TTL gas tight syringes

Pressure regulators

- * Norgren 11-818-100 regulators with 0-60 psi gauge (Figure 17)
- * 2-stage regulators for compressed air (Figure 16) or other diluent gas
- * 2-stage regulator for compressed toxic gas mixture

Compressed air purification

- * Water trap, prefilter for compressed air (Figure 16)
- * Domnick Hunter Oil-X combination unit AC-0007G
microfibre and activated carbon filters (Figure 16)

b) Heater/Humidifier Unit

Glassware

- * Quartz tube (for furnace, Figure 23) with 1/2" o.d. outlet
- * Rotaflo plain bore teflon stopcocks (taps D and E)
- * Quickfit double surface Davies condensers C5/12
- * Quickfit r.b. flask 1 litre, 2 neck 24/29, 19/26 sockets
- * Various Quickfit screwthread joints, ball and socket joints, clamps, springs, cones and sockets

Tubing

- * Silicone rubber 3/8" i.d.
- * Teflon 10 mm o.d. 8 mm i.d.

Metal fittings

- * Whitey B-44XF6 3-way ball valve (tap F)
- * Cajon SS-8-UT-1-8 1/2" connector (quartz tube fitting with viton O-ring)
- * Various 1/2", 10 mm, 6 mm, 3/8" s/s and brass tube and pipe fittings
- * Cooling water flow control valve

Heating equipment

- * Electrothermal 1 litre size heating mantle with control
- * Cylindrical electric furnace as in Figure 23
- * SR 1.5 silicone rubber electrical heating tape, 225 W

Temperature control

- * Shimaden model 01 indicating temperature controller 0-200C with thermocouple, s/s sheath (Appendix 2.2)
- * Simmerstats as in Figure 24

Humidity measurement

- * Kane-May KM 8004 humidity/temperature meter with probe as in Figures 27 and 44.

Pressure measurement

- * U-tube manometer

Sage Instruments

DIVISION, ORION RESEARCH INCORPORATED

model 341A syringe pump instruction manual

description

Sage Model 341A is a U.L./CSA medically approved, single-channel pump which answers most requirements for continuous infusion of fluids. It accepts all types of syringes, in any size from microliter to 50 cc. Rates are precalibrated and highly reproducible and are offered in two ranges - ml/min and ml/hr. Twenty-one speeds may be chosen; eleven in the ml/hr mode and ten in the ml/min mode. This provides rates from 0.00011 ml/hr (with a 10 μ l syringe) to 13.3 ml/min (with a 50 cc syringe).

The pump (**figure 1**) is housed in an aluminum case with a scratch-and chemical-resistant paint. It is powered by a motor which drives a set of gears on top of the pump housing. The gears engage mating racks inside the plastic drive carriage and move the syringe piston smoothly down its barrel (**figure 2**). The motor operates in a series of pulses*- approximately 50 pulses per second at the fastest rate, and 47 seconds per pulse at the slowest. Each pulse moves the carriage 6.9×10^{-4} cm (2.7×10^{-4} inch). At the end of an infusion run, the motor stalls itself, preventing syringe damage and in no way harming the motor.

controls and chart

mode switch

A three-position toggle switch that turns the motor OFF or selects one of two speeds: "ml/min" and "ml/hr".

CAUTION: Changing the switch from "ml/hr" to "ml/min" increases the speed by a factor of 60.

When the rate selector switch is in position eleven, the mode switch must be in the ml/hr position. A power light indicates when the switch is set to either of the ON positions.

rate selector switch

Eleven positions ranging from one (1) to eleven (11).

CAUTION: Do not attempt to operate pump with rate selector between numbered positions. Do not switch selector settings while operating. Over- or under-infusion could occur.

flow rate chart

Nominal flow rates (in ml/min or ml/hr, depending on the mode switch setting) are listed for the eleven settings of the rate selector switch and six syringe sizes.

Flow rates are given on the flow rate chart for five different B-D Plastipak® syringe sizes. Microliter syringes manufactured by Hamilton in 1, 10, 50, 100, and 250 μ l sizes may also be used. Flow rates for microliter syringes only are proportional to syringe volume. For example, a 50 μ l syringe is used with the mode switch set to ml/hr and the rate selector switch set at 1. The flow rate chart shows that these settings would result in a flow rate of .00011 ml/hr (0.11 μ l/hr) with a 10 μ l syringe. The flow rate with a 50 μ l syringe is then:

$$\text{rate} = 0.11 \text{ l/hr} \times 50/10 = 0.55 \mu \text{ l/hr}$$

operation

1. Connect power cord to a suitable power source. See **grounding** below.
2. Refer to the flow rate chart to select a suitable syringe size, flow rate, and mode (ml/min or ml/hr) for your application. If the flow must be known accurately or if the B-D Plastipak[®] syringes are not used, measure the flow rate by timing the flow and measuring the volume delivered.
3. Remove drive carriage by lifting it vertically from the gears.
4. Lift the knob of the spring-loaded syringe holder high enough to accommodate the syringe barrel (**figure 3**).
5. Using the other hand, place the filled syringe in the holder with the syringe barrel flange flush against the syringe holder (**figure 1**), and lower the knob to hold the syringe in place.
6. Replace drive carriage on the gears, making sure the carriage is parallel to the edge of the pump.
7. Advance drive carriage by turning rate selector switch to position 10, and run the pump in the ml/min mode. Turn it off just before the carriage reaches the piston flange. The pump is now ready for immediate operation.
8. Turn the rate selector switch to the required setting.

CAUTION: DO NOT ATTEMPT TO OPERATE PUMP WITH RATE SELECTOR SWITCH BETWEEN NUMBERED POSITIONS. DO NOT SWITCH SELECTOR SETTINGS WHILE INFUSING. THESE ACTIONS COULD CAUSE OVER- OR UNDER-INFUSION.

9. Turn the mode switch to the required position, ml/min or ml/hr.

CAUTION: CARE MUST BE TAKEN WHEN SELECTING THE APPROPRIATE FLOW RATE MODE. PLACING THE SWITCH IN THE WRONG POSITION COULD OVER OR UNDER INFUSE THE PATIENT WHICH MAY RESULT IN SERIOUS INJURY.

Position eleven can be used only when the mode switch is in the ml/hr position. Moving the switch to either position will turn on the pump motor, and pumping will start immediately.

At the end of an infusion run, the pump motor will automatically stall to prevent damage to the syringe and the motor. Reset by returning to step 3.

grounding

Grounding reliability can only be achieved when the equipment is connected to an equivalent receptacle marked Hospital Grade.

NOTE: B-D Plastipak[®] syringes are recommended for use with the 341A pump, although glass or other syringes can be used. To assure operation against maximum back pressure use each B-D Plastipak[®] syringe only once. All flow rates are based on B-D Plastipak[®] syringes. Flow rates may be nominal if other types of syringes are used.

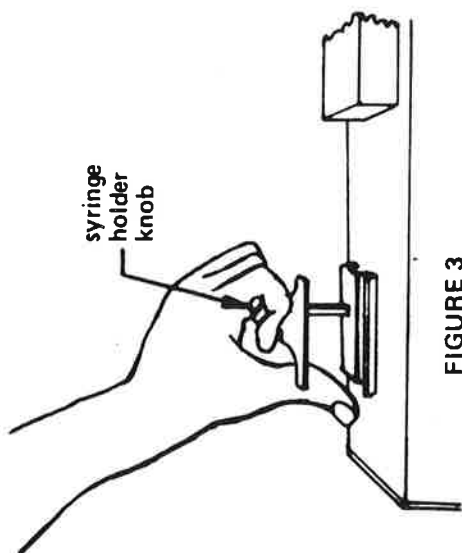


FIGURE 3

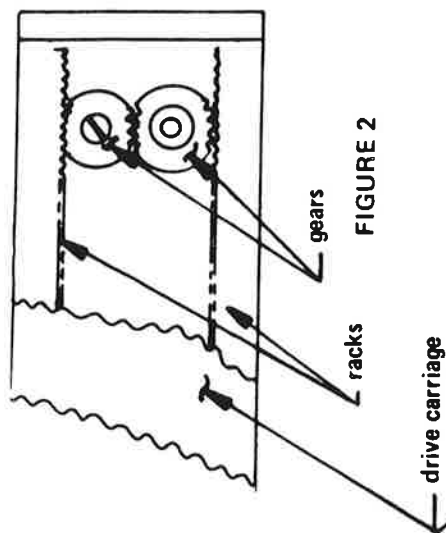
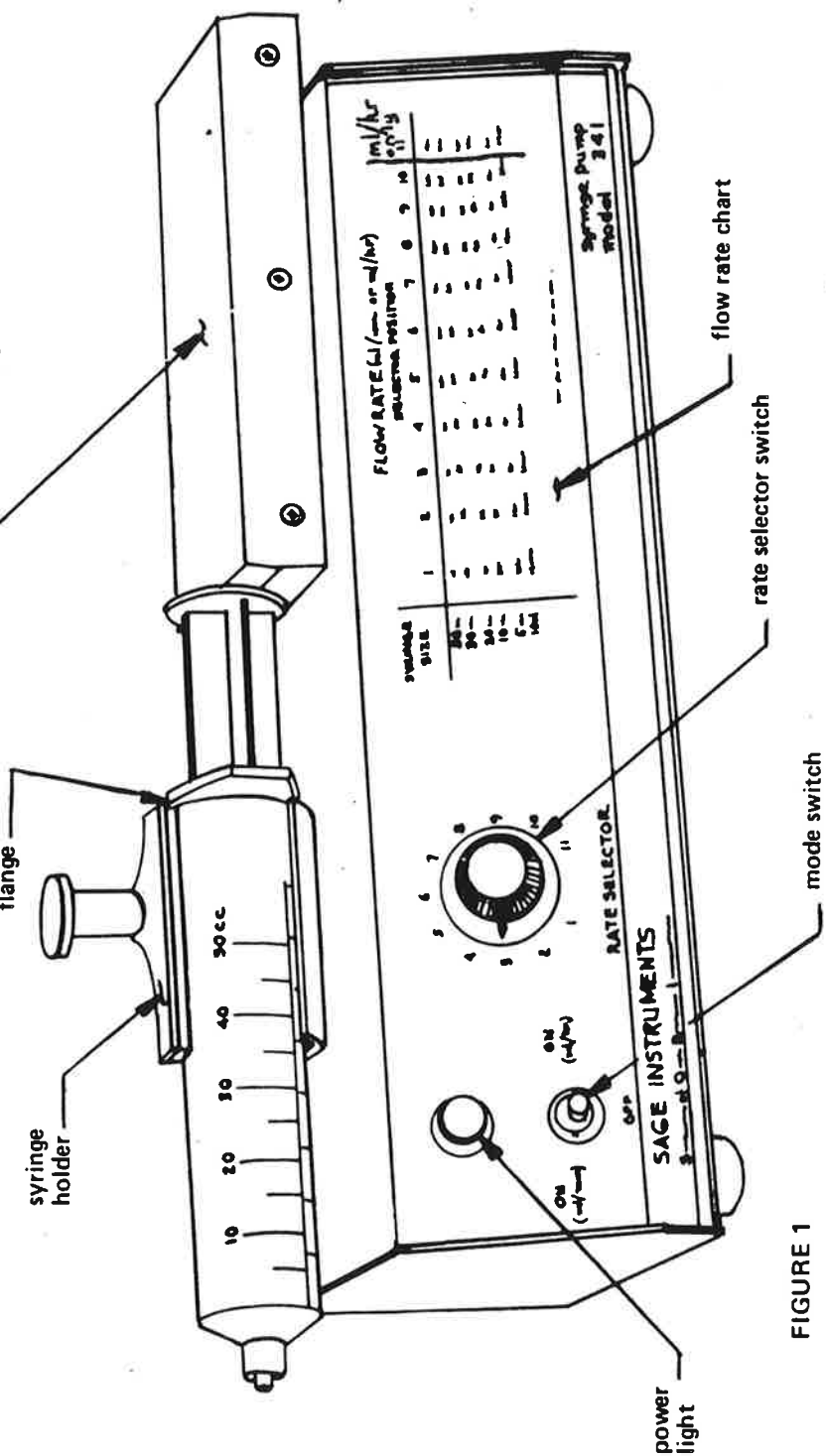


FIGURE 2



specifications

power requirements	115/230 VAC, 50/60 Hz	
accuracy	± 10% (based on B-D Plastipak® syringes)	
reproducibility	± 2% of setting	
maximum back pressure	syringe size	back pressure (mm Hg)
	50 ml	250
	30 ml	400
	20 ml	500
	10 ml	900
	5 ml	1300
case	aluminum with chemical-resistant paint	
weight	1.8 kg	
size	31 cm x 13 cm x 11.5 cm	

CSA File No. CR41766-1

U.L. File No. R57114

specifications subject to change without notice

IMPORTANT NOTICE: In order for ORION's warranty of the pump to be effective, you must complete and return the enclosed warranty card. ORION WILL NOT HONOR THE WARRANTY UNLESS THIS CARD IS RETURNED.

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Form IM341A/1890D
Printed in U.S.A.

ORION RESEARCH INCORPORATED 840 Memorial Drive, Cambridge, MA 02139 (617) 864-5400/telex 921466

OPERATING INSTRUCTIONS

Appendix 2.2

2-Position
(Control Mode 02)

01 Series — Electronic Indicating Controller
02 Series — Electronic Non-indicating Controller

Shimaden 01/02 series controller are the highest faculty controller which have been manufactured under Shimaden's long-experienced and the newest technology combined with high reliable parts.

Before your operation, please read this instruction sheet so as to attain the best performance.

Please contact the dealer in case when you have question and trouble.

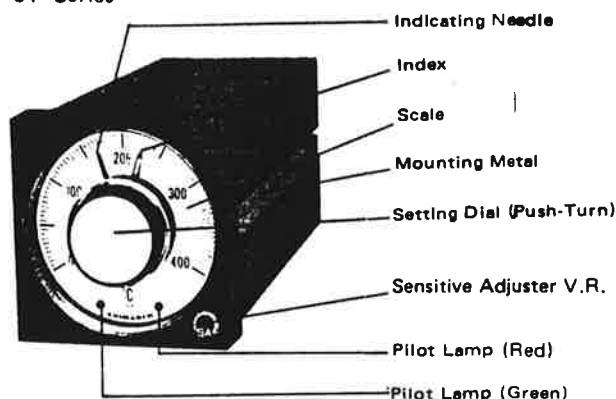
SPECIFICATION

Measuring Mode DC potentiometric for thermo-couple input models
DC bridge for resistance temperature detector input models
Indication Moving coil drive
Indication Accuracy $\pm 1.5\%$ of scale range
Setting Push-and-turn dial setting
Setting Accuracy $\pm 1.0\%$ of scale range
Burnout Circuit Provided for thermocouple input models only

Operating Ambient Temperature -10°C to $+50^{\circ}\text{C}$
External Resistance Allowable Range for Thermocouple 100Ω maximum
Lead Resistance Allowable Range for Resistance Thermometer 2Ω maximum
Supply Voltage AC 110/120V, 220/240V, $\pm 10\%$, 50/60Hz
Control Relay Capacity AC 220V — 10A (resistive load)

DESIGNATIONS

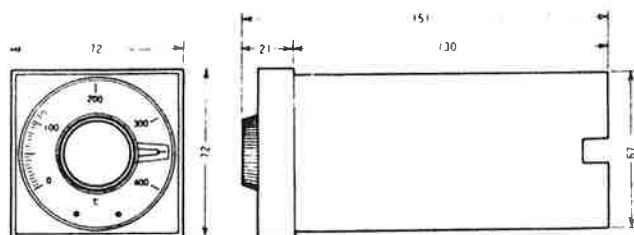
01 Series



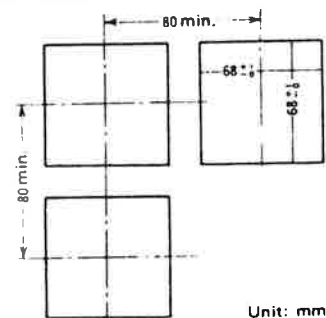
02 Series



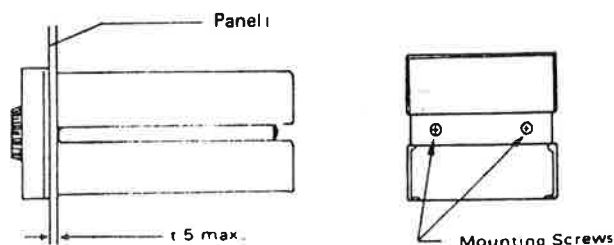
EXTERNAL DIMENSIONS



PANEL CUTOUT



INSTALLATION



Drill mounting hole (68 x 68 mm) in the panel is illustrated above.

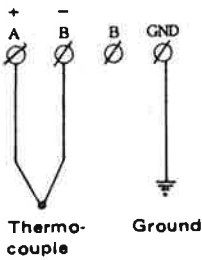
Remove the metal fitting attached to the instrument, fit the instrument into the mounting hole, put the metal fitting back to the instrument and fix the instrument in the panel by means of two mounting screws.

AMBIENT CONDITION

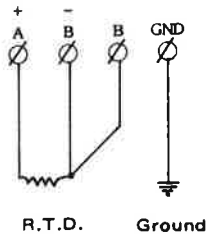
Controller is to be placed where its temperature is within 0°C — 40°C and its humidity is under 80% RH.
Avoid Corrosive gas and inflammable gas.

INPUT CIRCUIT WIRING

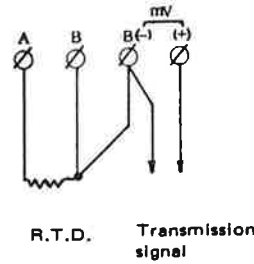
THERMOCOUPLE TYPE



R.T.D. TYPE



R.T.D. TYPE w/TRANSMISSION SIGNAL (TRANSMISSION SIGNAL ONLY FOR R.T.D.)



DC 0 - 10mV/Full Scale

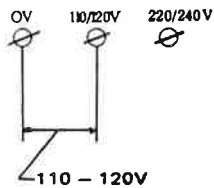
Output Characteristics: Non-Linear

The input circuit is of weak current and the wiring to it shall not be made up into a bundle with heavy current circuit, especially with 200V line. Use shielded wires to the maximum extent possible in wiring in the panel.

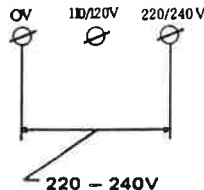
- * Use compensation wires up to the instrument terminal in case of thermocouple. Exercise care not to confuse polarity.
- * External resistance shall be kept minimum, i.e., 100Ω maximum in thermocouple input and 2Ω maximum per single wire in 3-wire resistance bulb input.
- * Connections on the way shall be soldered or firmed up by use of terminal board. Loose connections may become a cause of errors or malfunctioning of instrument.

POWER SUPPLY WIRING

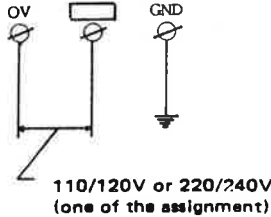
110 - 120V



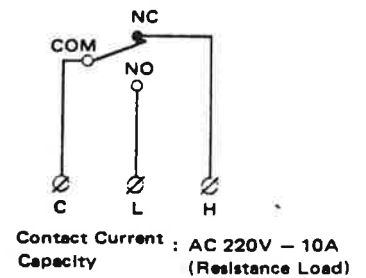
220 - 240V



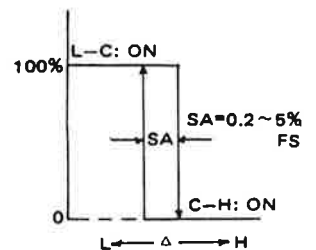
W/TRANSMISSION SIGNAL



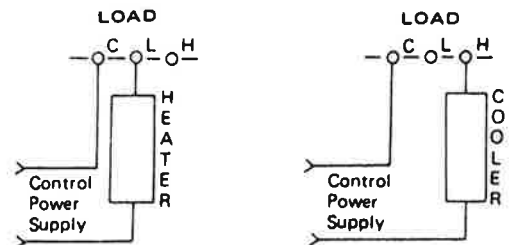
CONTROL TERMINAL



CONTROL PATTERN



WIRING DIAGRAM



Power supply to the instrument can be AC 110-120V, 50/60Hz or 220-240V, 50/60Hz.

- AC 110-120V: Connect OV terminal and 110/120V terminal
- AC 220-240V: Connect OV terminal and 220/240V terminal

OPERATION

1. Push and turn the setting dial to the scale required.
2. Make power source then instrument operates.
When measured value is lower than set value, green lamp lights and relay for C-L contacts.
When measured value is higher than set value, red lamp lights and relay for C-H contacts.
3. Adjust control sensitivity adjuster (S.A.) V.R. to the accuracy required. (S.A.) V.R. is indicated in percentage of full scale. Small percentages means narrow sensitivity band.

MDHS 3

Methods for the
Determination of
Hazardous Substances

March 1981

Generation of test atmospheres of organic vapours by the syringe injection technique

Portable apparatus for laboratory and field use

Health and Safety Executive: Occupational Medicine and Hygiene Laboratories

INTRODUCTION

1 Organic vapours are, in general, toxic and may also be flammable. Handle pure chemicals and concentrated solutions in a ventilated fume cupboard. The apparatus or vapour exit should be placed in a ventilated fume cupboard or suitably vented to the outside atmosphere. Consult suitable source reference for toxicity and first-aid advice.

SCOPE

Range

2 This method is suitable for the generation of atmospheres of gases or low- to medium-boiling liquids from the volume percent level down to 0.1 ppm in one to three dilution steps. The atmosphere may be generated at a rate between 1 and 100 litres/min. The apparatus is suitable for liquids with boiling points of up to about 140°C, but the range may be extended by solvent dilution of the injected liquid.

Accuracy

3 Assuming there are no leaks and the (dynamic) system has reached equilibrium, there are three main source of error:

Atomisation

4 The liquid may not be evaporating in a smooth and predictable manner, particularly if it is relatively high-boiling.

Syringe

5 The motor-driven injector is expected to have precision of 1%, measured as a relative standard deviation and, if calibrated, should have a bias of less than 1%.

Rotameters

6 The rotameters have a specified precision of 2%, measured as a relative standard deviation. A bias may occur if temperature and pressure corrections are not made (para 35).

Stability

7 There will be some short-term variation in the concentration of the delivered atmosphere, owing to slight

irregularities in the syringe drive, atomisation and pressure control. This variation occurs over time periods of the order of a few seconds and should not exceed $\pm 5\%$ of the mean delivered concentration. Some smoothing of the atmosphere may be achieved by incorporating a large (about 10 litres) glass vessel at the output stage of the design.

Construction

8 The construction is of glass with ball and socket joints to avoid strain and facilitate interchangeability of parts. The apparatus is mounted on a board of height 560 mm and width 660 mm, which is sufficiently compact to be placed in a small fume cupboard. The apparatus can be made fully portable, requiring only a source of compressed air and, for toxic vapours, fume extraction.

PRINCIPLE

9 Organic gases or liquids are loaded into a gas-tight syringe connected by PTFE tubing to an atomisation chamber.

10 The organic material is injected into the atomisation chamber by means of a motor-drive on the syringe.

11 A regulated supply of air (or nitrogen, or calibrated gas mixture) is simultaneously applied to the atomisation chamber.

12 If desired, the atmosphere generated in the atomisation chamber may be diluted with a second or third regulated supply of air.

13 The concentration of atmosphere generated is calculated from the rate of injection of the gas or vapour and the rate of supply of the air or diluent gas.

REAGENTS

Compressed air

14 A compressor or compressed air cylinder may be used. The apparatus includes a purification unit.

Bulk gas or liquids

15 These should be analytical grade of known purity.

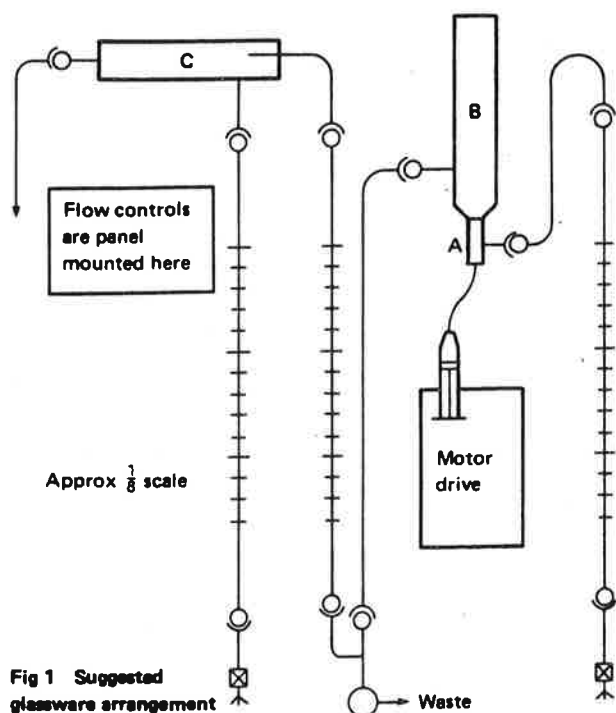


Fig 1 Suggested glassware arrangement

APPARATUS

16 The apparatus is illustrated in Fig 1. The main items required are:

Glassware (Figs 2 and 3)

17 Borosilicate glass is used for the mixing chambers and connecting tubing. Joints are all ball and socket. The connecting tubing should be at least 9 mm ID to minimise resistance to air flows.

Injector tip enlarged x2

Epoxy resin
PTFE
Weeble

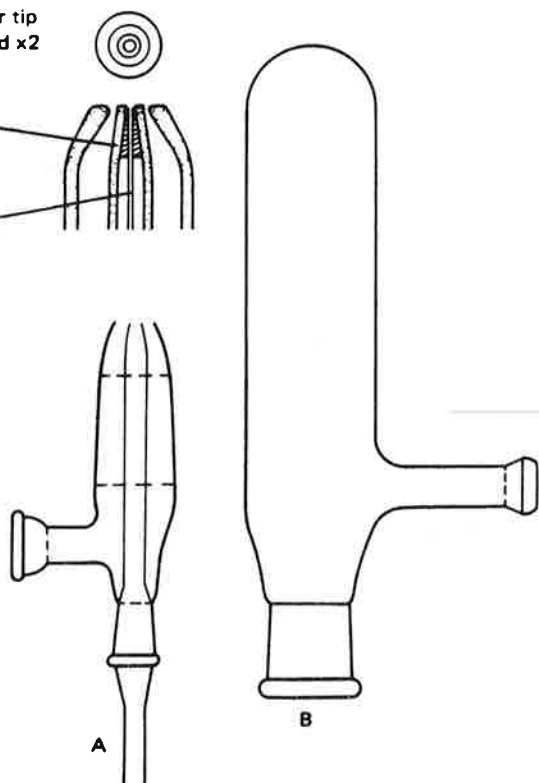


Fig 2

Flow meters

18 A range of rotameters from 30–350 ml/min to 5–50 litres/min.

Motor-driven syringe unit

19 Suitable for injecting liquids in the range 5–30 μl /min from a 1-ml glass syringe.

Regulators

20 Pressure regulators, needle valves, on–off valves, 3-way valves, and an air filtration and purification unit.

PROCEDURE

21 The apparatus is set up as in Fig 1 (for a two-stage dilution system).

22 Calculate the gas flows required for generating the specified atmosphere, selecting syringe size, syringe drive speed, and rotameters for gas flows to atomiser and to second or third dilution stage as appropriate.

23 A 1-ml gas-tight precision glass syringe is generally used for liquids; a 10-ml gas-tight precision glass syringe is generally used for pure gas.

24 The syringe drive speed should be a minimum of 0.3 mm/min to avoid irregularities in the test atmosphere due to thermal expansion or contraction of the syringe reservoir.

25 Individual dilutions should not exceed a ratio of 1:100 v/v. It may be necessary to include a further dilution stage to achieve generated concentrations in the sub ppm range.

26 Switch on and adjust syringe drive, pressure and flow regulators.

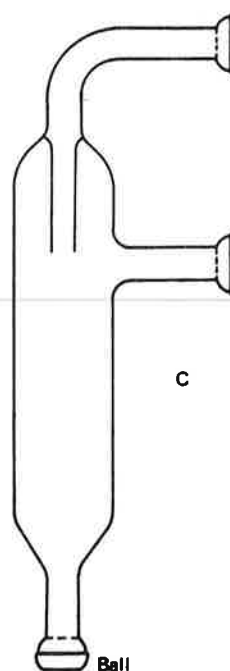


Fig 3

27 Checks should be made to ensure even evaporation of liquid (if used) in the atomiser.

28 Allow system to stabilise; dynamic equilibrium should be reached within 10 min.

CALIBRATION

Syringe drive

29 Fill the 1-ml glass syringe with distilled water. Operate the motor-drive syringe at the drive setting to be used and collect the dispensed water into a previously weighed 10-ml flask. Run the motor for 60 minutes. Reweigh the flask and calculate the volume of water collected.

$$q_l = \frac{v}{t}$$

where q_l = injection rate ($\mu\text{l}/\text{min}$)
 v = volume of water collected (μl)
 t = time (min)

Rotameters

30 The true flow rate of a gas passing through a rotameter calibrated at T_1 K and P_1 mm Hg for air may be obtained by multiplying the flow indicated on the rotameter by the factor $f = \left[\frac{P_2 \times T_1}{\rho_g \times P_1 \times T_2} \right]^{1/2}$

where T_2 = operating temperatures (K)
 ρ_g = density of gas relative to air
 P_2 = operating pressure (mm Hg)

Atmosphere

31 The concentration of vapour delivered by the apparatus may be checked by independent analysis, e.g. infra-red gas analyser, gas chromatography (after collection and desorption from adsorbent of known desorption efficiency), wet chemical methods.

CALCULATIONS

To calculate diluent gas flows for standard atmospheres of liquid vapours, the following expression may be used:

$$q_g = \frac{\rho_l \times q_l \times 1000}{C \times f}$$

where q_g = indicated gas flow (litres/min)
 ρ_l = liquid density (g/ml)
 q_l = liquid injection rate ($\mu\text{l}/\text{min}$)
 C = required vapour concentration (mg/m^3) at operating temperature and pressure
 f = rotameter correction factor (paras 30 and 35).

33 To calculate diluent flows for gases, the following expression may be used:

$$q_2 = \frac{q_1}{C'}$$

where q_2 = diluent gas flow (litres/min)
 q_1 = primary gas flow of pure vapour ($\mu\text{l}/\text{min}$)
 C' = required vapour concentration (vol. ppm)

34 Concentrations in vol. ppm and mg/m^3 are related by:

$$C' = C \times \frac{24.45}{\text{MW}} \times \frac{760}{P_2} \times \frac{T_2}{298}$$

where C' = vapour concentration (ppm)
 C = vapour concentration (mg/m^3)
 24.45 = molar volume (litres) at 25°C and 760 mm Hg
 MW = molecular weight of substance used.

35 For temperatures and pressures within 10°C or 30 mm Hg of calibration conditions, rotameter corrections are less than 2% and may be ignored. Care should be taken, particularly in the design of the atomiser, to avoid significant back pressure which might introduce further rotameter pressure corrections. For flows greater than 15 litres/min a larger orifice than that shown in Fig 2 (A) may be required.

ADVICE

Advice on this method and the equipment used may be obtained from the Health and Safety Executive, Occupational Medicine and Hygiene Laboratory, 403-405 Edgware Road, London NW2 6LN (telephone 01-450 8911).

A number of methods are available for generating standard atmospheres, including others described in this series.¹ The use of other methods not included in this series is acceptable provided they have the accuracy and reliability appropriate to the application.

The Health and Safety Executive wishes, wherever possible, to improve the methods described in this series. Any comments that might lead to improvements would therefore be welcome and should be sent to the above address.

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Further copies of this publication, price 50p, can be obtained from the Directorate of Information and Advisory Services, (IAS 5), Baynards House, 1 Chepstow Place, London W2 4TF, tel. 01-229 3456.



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