

**HANDBOOK OF LABORATORY
UNIT OPERATIONS FOR
CHEMISTS AND CHEMICAL
ENGINEERS**

JAN PINKAVA,

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PREFACE

The first edition of this book, in Czech, was published some ten years ago by the Czechoslovak Academy of Sciences. It was immediately translated into Russian and German, and all editions were sold out in a short time. The majority of readers welcomed the book, finding it a valuable and stimulating aid in designing and modelling equipment and systems for continuous processes in chemical engineering laboratories.

Although the framework of the present book is that of the original Czech edition, the whole work has been extensively revised and expanded, and this English edition now contains considerably more material than the original book. Perhaps an idea of the extent of this revision and expansion can best be obtained from the greatly increased number of references; the Czech edition contained less than five hundred (up to 1957), whereas nearly two thousand are given in the present book. I think that from this it is also very obvious how rapid and intense has been the expansion in this branch of chemistry. Where the book will be of particular value to the chemical engineer is in helping him to reduce considerably the cost of construction and testing of experimental and small-scale installations, emphasising as it does the use of glass as a constructional material. In small models of this type glass can be used satisfactorily at pressures of up to ten atmospheres and, in comparison with apparatus fabricated in metal, its modification is far simpler and more rapid, while heating, cooling, measurement and direct observation of the processes is, of course, greatly simplified.

In the interests of clarity it has been necessary to omit or abridge parts of the original text, while the chapters have also been rearranged in a more logical way. In selecting material from new papers I concentrated on that which was of novel or of particular interest, referring only to others in passing, although giving brief details in some cases. Papers published after completion of the manuscript (up to the first half of 1969) are listed in an extensive new bibliography.

My aim has been to produce a book which is easy to read and which informs the reader of the most recent developments; how successful I have been in this is for him to decide. The selection and evaluation are, of course, subjective, and could have been made in another way.

I consider it a pleasant duty to express my thanks to Professor Bažant, Director of the Institute of Chemical Process Fundamentals, for his permanent

interest and some valuable suggestions for the compilation of the manuscript, to Mr. Rudinger for his especially careful translation and correction of the English text, to all the other people who were helpful in some way and, last but certainly not least, to my wife Mileva and to my son Nick for their readiness in undertaking the exhausting work of typing the manuscript, the list of references and the index.

The Author

Zürich, September, 1969

INTRODUCTION

WHY BUILD PLANTS FOR CONTINUOUS OPERATION ?

Some manufacturing processes in the chemical industry are typically continuous while others are typically batch. Between these two extremes there is a wide range of processes that can be carried out in either way. Economical and technical factors influence the decision for or against the continuous system of manufacture⁹¹³.

Yields and product quality are far higher as a rule in continuous processes²⁴. Plant is better utilized and productivity is invariably higher. In many instances the plant can be installed in the open, hence the costs of buildings are lower. The plants are smaller and therefore less expensive.

On the other hand, continuous plants are generally designed for a particular product, and major modifications are necessary if a different process is to be used. They are more sensitive to seasonally fluctuating loads and to varying qualities of raw materials⁷. Breakdowns affect the manufacture far more seriously than in batch processes, and the start-up and shut-down of a continuous process plant is much more difficult. Process engineering, planning and charting is invariably more expensive in terms of both time and money, and as a rule measuring and control instruments cost far more⁸⁹².

As to the technical aspect; the nature of the reaction is of paramount importance. The residence time of the raw materials in reactors and other equipment is far shorter in continuous processes. If very short reaction times are required, say a few seconds, continuous processing is essential^{1049,1087,1236}. Continuous processes are also preferable in the manufacture of unstable or explosive substances. Moreover, they permit the use of noble structural materials and often glass. Continuous processes are always more easily automated.

The demands on the skills of both the process engineer and the workshop manager are invariably higher in continuous manufacture. The proportioning of solid and semisolid materials is often a source of serious difficulties, and so are corrosion or clogging. Long reaction times, lasting several hours for instance, can also be a problem in continuous processes.

Adopting a continuous process, after due deliberation, is not an easy decision. It is influenced by the technical and economic information available, by experience, and often by the engineer's fifth sense. In well chosen and appropriate cases, however, the change to continuous operation yields great improvements.

REASONS FOR THE CONSTRUCTION OF MODELS

The experimental operation of laboratory models is intended to provide data for planning pilot-plants. The course of chemical reactions in a continuous process frequently differs from that in a batch process. The same is true for the product quality and the kind and amount of by-products. Moreover, some reactions are not possible at all in a batch process. No heat balance can be computed for an indistinct reaction; but comparatively accurate measurements can be taken on a model⁶⁹⁴.

In the preparation of very pure substances (e.g. monomers for polymerization, drugs, etc.) calculations cannot be relied upon fully. In such cases even those operations that are normally designed theoretically, such as rectification, are modelled in the laboratory. Clogging and corrosion of equipment, or the effect of materials of construction upon a reaction, can be estimated only approximately; a model however can provide reliable data. In some cases an intermediate, needed for further processing, cannot be made available; it is then often prepared in an experimental model, although its production does not require any measurements. Models are also used to try out proposed manufacturing processes and to study their optimum conditions. Measuring and control systems too are based on data obtained in model experiments.

All this information could naturally be derived from pilot-plants. However, experimental investigations using model plant are incomparably quicker and cheaper, although often tedious.

The basic concept of a laboratory model must take full account of the continuity and the potential automation of the process. Wherever possible, working with solids is avoided since proportioning and conveying them is more difficult than handling liquids. In drafting the detailed plan all calculable data are computed, the procedure being similar to the course followed in designing a pilot-plant project. For this purpose all quantities that are measurable without a model (e.g. physico-chemical data) have to be ascertained, since model measurements would be more difficult.

Small models are generally preferable to large ones: they consume less material and work is quicker. In small-size models glass can be used throughout for pressures up to 10 atm gauge. The construction of metal apparatus, and more particularly modifying it, takes far longer and holds up work. It is good practice

to put into the glass devices samples of the metals which are to be used as constructional materials in the pilot-plant or full-size equipment. They are thus tested for corrosion and for any effects which they may have on the course of the process. The individual steps of the model process may be allowed to differ from the final procedure, provided these deviations expedite the work and do not affect the functioning of the model and the accuracy of the measurements. For instance, electrical heating can be used although this would be uneconomic in the industrial process under investigation; similarly a heater and a cooler can be substituted for a heat-exchanger, etc. Such substitutions are permissible since power economy in the model is not important.

If the manufacturing process to be measured consists of a succession of several partial stages, it is advisable to measure each stage separately. Before the measurements that are to reveal the optimum conditions, the apparatus must work perfectly, the more so if several manufacturing stages are to be measured, one immediately following the other.

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1 MEASUREMENT

The measurement of various physical and physico-chemical quantities is essential since the measured values enable the process to be examined and controlled. The accuracy required depends on the nature of the operation and other factors. Unnecessary accuracy raises the cost of equipment and complicates work, while insufficient accuracy minimizes the usefulness of the results. The optimum accuracy is usually a matter of judgement.

In most cases measurements should be recorded. This is generally done by converting the measured quantities into electrical analogues (voltage, current) or into related air-pressures, i.e. quantities that can be recorded by commercial instruments. Recording instruments are difficult to improvise and makeshift ones often give poor results.

1.1 FLOW MEASUREMENT

1.1.1 FLOW OF GASES

The gas flows to be metered in the laboratory are usually far smaller than in industrial practice and frequently the requisite equipment must be specially made. Therefore simplicity is essential and the choice of measuring methods is more limited than in the factory.

Flowmeters with capillaries or porous glass plates are used most frequently for metering instantaneous flow rates. Venturi and Pitot tubes are seldom applied for metering flow through large diameter tubes. Commercially available Rotameters are used to advantage.

When large volumes of gas are involved, factory-made dry or wet gas-meters are used to measure the total volume discharged. They are not suitable for very small volumes and in such cases some of the integrating flowmeters described below are used.

METERING THE INSTANTANEOUS FLOW RATE OF GASES

In technical practice gas flow is measured by a variety of methods^{327,712}. In the laboratory there is less choice because the flow rates are usually small, and the apparatus must often be made specially, so its design has to be simple. The usual way of metering the instantaneous flow rate of gases is to measure the pressure drop across some form of restriction. The relation between the drop and the flow rate can be either linear or quadratic. The linear dependence, given by a capillary tube, a porous sintered-glass plate or a plug of glass wool, facilitates calibration and is suitable for even the smallest flow rates. The capillary tube has the additional advantage that its resistance is easily modified by changing its length. For the same flow rate it has a larger diameter than an orifice plate and is therefore less sensitive to clogging. Flow in the capillary should be laminar which means that the Reynolds number must be below the critical value.

The Reynolds number is a dimensionless quantity defined by the relation

$$Re = vd\rho/\eta$$

where v is the average velocity of the flowing gas or liquid [cm s^{-1}], d the diameter of the capillary [cm], ρ the specific gravity of the fluid [g cm^{-3}], and η absolute viscosity [poise]. A Reynolds number less than 2,300 is characteristic of laminar flow, a higher value indicates turbulent flow.

Vortices are always formed at the inlet of a capillary. In order to obtain a truly linear relation between the flow rate and the pressure drop the capillary must be long in relation to its diameter. In practice its length is chosen to be at least 100 times its diameter. Provided the gas is absolutely clean, even the smallest flows can be measured with a capillary flowmeter.

For higher flow rates capillary meters are inconvenient because the capillary would need to be very long. If linear relation between the pressure drop and the flow rate is considered essential, either a sintered-glass plate or glass wool is used to meter higher flow rates. The pore size of a sintered-glass plate cannot be modified except during its manufacture (cf. p. 14). The resistance of glass wool may be increased by compressing it.

Orifice plates are not suitable for low flow rates because they are easily blocked. For metering higher flows, however, they are very convenient. Making them, and in particular producing the correct diameter, is not easy. Usually the plate has to be exchanged to alter the resistance. A great advantage of orifice plates is their comparatively low sensitivity to the viscosity (and hence to the temperature) of the gas. In such meters the pressure difference is a function of the square of the flow rate; sometimes this renders process control based on the meter reading somewhat difficult. If the flow fluctuates, the reading does not represent the mean but (unlike capillaries) a lower value.

The characteristics of some resistors used in practice are between those of the capillary and the orifice plate. Such, for instance, is a capillary tube whose resistance is increased by a constriction, and not by increasing its length. It is difficult to calibrate such a flowmeter, and if it has to be discarded (because it is clogged up or broken) it is virtually impossible to replace it with an identical tube. The resistance of a capillary should therefore be adjusted solely by altering its length or by exchanging it for one with a different diameter.

Venturi or Pitot tubes can be used to meter high flow rates. Such methods, however, are hardly ever used on a laboratory scale. In pilot-plants, flow rates are frequently metered with Rotameters. Commercial types are preferred although laboratory production of Rotameters is not difficult (cf. p. 29). The relation between the flow rate and the displacement of the float depends on the shape of the float. It can be similar to the capillary characteristic (for a cylindrical float) or to that of an orifice plate (float with sharp trailing edge), in which case it becomes virtually independent of the viscosity of the fluid.

If the gas is contaminated with dust or mist, the flow cannot be measured accurately and errors, initially small, tend to grow. This is true for capillaries and orifice plates as well as for other resistors, e.g. glass wool or sintered-glass plates. In all these instances the gas must be filtered and freed from entrained solids and liquids. The removal of dust is comparatively simple whereas droplets tend to clog filters fairly rapidly. Various separators are used before filtering, depending on the nature of the dispersed liquid, such as tubes filled with Raschig rings, sawdust, active carbon or other materials. Acid and alkaline vapours, including for instance sulphur trioxide mist, are removed by passing the gas through a layer of ion exchange resin containing 50 to 70 weight per cent water.

Sintered-glass plates are very useful gas filters. The pore size is selected according to the permissible pressure drop, but as complete a purification of the medium as possible should always be attempted. It is worth noting that commercial gases marketed in pressure cylinders often contain considerable quantities of solid particles. The sources of this contamination are probably corrosion products from the cylinder walls and impurities from regulators and valves. Compressed air always contains some oil, water, and rust particles from the piping. A moisture separator such as a sawdust filter is inserted into the air supply line, and if necessary the air is dried with calcium chloride or sulphuric acid before entering the filter.

Hydrogen chloride prepared from hydrochloric acid contains a very persistent mist of sulphuric acid. The purification of hydrogen chloride in large quantities is difficult, and it is preferable to use two flowmeters connected in parallel, which can be switched off and cleaned in turn. A satisfactory method of cleaning smaller quantities consists in passing the gas through large jars filled with active carbon.

Other gases are cleaned by adsorption, scrubbing, electrostatic precipitation or filtration, depending on the nature of their contamination. Complete purification is frequently difficult as well as expensive and is not always economic if carried out

on an industrial scale. In small-size models, however, it is invariably worth while because it facilitates smooth operation and adds to the reliability of the results.

Small quantities of a gas which must not come into contact with water may be introduced into the flowmeter from a dry gas container, a thick-walled bottle containing the gas in a rubber bladder. Compressed air introduced into the bottle displaces the gas from the bladder¹²⁸². An ordinary polyethylene bottle may also be used for the purpose⁶⁹⁹.

Flowmeters can be easily calibrated if the dependence of the gas flow upon the pressure drop conforms to the theoretical relation for either the capillary or the orifice plate.

When the relation is somewhere between these two extremes, the calibration of meters requires a series of measurements (cf. p. 5).

A flowmeter with linear resistance can be calibrated using either the gas to be metered, or a different gas and subsequently converting the results using the relation

$$\frac{Q_1}{Q_2} = \frac{\eta_2 \Delta p_1}{\eta_1 \Delta p_2} \quad (1)$$

where Q_1 is the calculated flow rate of the metered gas, Q_2 the flow rate of the gas used for calibration, η_1 and η_2 are the respective viscosities, and Δp_1 and Δp_2 the pressure drops. When the author of Ref. 505 converted the readings obtained with a glass-wool flowmeter on one gas to the flow of a different gas, the proportionality constant (which should be 1) worked out to 0.94–1.01. Acceptable results are attained only at low velocities and with laminar flow, when the flow is related linearly to the pressure drop. Re should be selected to be as low as possible. In air flow through a capillary, turbulent flow was observed with Re as low as just above 1,000¹²⁸⁰.

If the calibration curve plotted for one gas is to be used for a different one, the data can be converted. The procedure recommended by Ergun is feasible provided the physical constants of the second gas are known. The computed results are valid for capillaries as well as for orifice plates or resistors ranging between the two extremes, and for both laminar and turbulent flow³⁷⁴.

The pressure loss for isothermal flow through a capillary or orifice plate consists of the sum of the viscous loss (or frictional loss, i.e. the pressure drop due to the viscosity of the gas) and the kinetic energy losses

$$\Delta p = a\eta Q_m + b\rho_m Q_m^2 \quad (2)$$

where Δp is the loss of head [dyn cm⁻²], η the viscosity of the gas [dyn s cm⁻²], ρ the specific gravity [g cm⁻³], Q the gas flow [cm³s⁻¹], and a and b are constants. Subscript m corresponds to temperature T and to the mean value between pressures

p_1 at the gas inlet and p_2 at the outlet. Since $\varrho_0 Q_0 = \varrho_m Q_m$, where subscript 0 indicates normal conditions, Eq. (1) can be rewritten to read

$$\Delta p / Q_m = a\eta + b\varrho_0 Q_0 \quad (3)$$

where

$$Q_m = Q_0 \left(\frac{T_m}{T_0} \right) \left(\frac{2p_0}{p_1 + p_2} \right). \quad (4)$$

Equations (2) and (3) are solved for Q_0 neglecting the negative root

$$Q_0 = \frac{1}{4} \left(\frac{a\eta}{b\varrho_0} \right) + \frac{\Delta p}{b\varrho_0} \left(\frac{\Delta p + 2p_2}{2p_0} \right) \left(\frac{T_0}{T_m} \right) - \frac{1}{2} \frac{a\eta}{b\varrho_0}. \quad (5)$$

The constants a and b of the flowmeter can be calculated from the measured dependence of the flow rate on the pressure drop according to Eq. (2). If $\Delta p / Q_m$ is plotted against Q_0 it gives a straight line whose y-intercept and gradient equal $a\eta$ and $b\varrho_0$ respectively. If the constants are known, the flowmeter can be used on a different gas of known density and viscosity without recalibration. The flow is calculated according to Eq. (4). The author presents the computation of the constants from the values measured in the flow of nitrogen and carbon dioxide. Errors were corrected by the method of least squares. The actual flow rates agreed with the computed values within limits of approximately $\pm 0.3\%$.

Although this method permits the conversion of flow rates, its practical value is affected by the rather laborious calculations. A quicker and more convenient conversion method is suggested by the authors of Ref. 233 who write the variables in a dimensionless form. It holds for laminar and turbulent flows as well as for the transition region, for capillary and orifice plate resistances, and for both gases and liquids; hence it may be used to convert flow rates from gas to gas, from liquid to liquid, and from liquid to gas or vice versa.

Denote the variables occurring in the problem by the following symbols:

- Δp — pressure drop [$\text{g cm}^{-1} \text{s}^{-2}$]
- Q — flow rate (by volume) [$\text{cm}^3 \text{s}^{-1}$]
- ϱ — specific gravity of the flowing gas or liquid [g cm^{-3}]
- η — viscosity of the flowing gas or liquid [$\text{g cm}^{-1} \text{s}^{-1}$]
- d — diameter of nozzle or orifice plate [cm]
- D — length of capillary tube or diameter of tubing with orifice plate [cm]

The variables can be written as three dimensionless groups

$$\pi_1 = \left(\frac{\Delta p \varrho d^2}{\eta^2} \right); \quad \pi_2 = \left(\frac{Q \varrho}{\eta d} \right); \quad \pi_3 = \left(\frac{d}{D} \right) \quad (6)$$