AIChE

EQUIPMENT TESTING PROCEDURE

PACKED COLUMNS

A GUIDE TO PERFORMANCE EVALUATION

Second Edition

MERICAN INSTITUTE OF CHEMICAL ENGINEERS
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AIChE Equipment Testing Procedure

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CONTENTS

100.0 PURPOSE AND SCOPE

101.0 Purpose

This testing procedure offers methods of conducting and interpreting performance tests on packed distillation columns. Such tests may be made to accumulate reliable data in one or more of the following areas of interest: packing efficiency, capacity limitations, energy consumption and pressure drop considerations. These data are useful for troubleshooting, for debottlenecking, for new designs, for correlations, and for determination of the operating range of a column as well as the optimum operating conditions. Such data are often most useful in verifying or calibrating computer simulations which are then used for optimizing, debottlenecking and design studies. These data may also be required for an "acceptance test."

102.0 Scope

Rather than compulsory directions, a collection of techniques is presented to guide the user. Emphasis is placed on principles rather than on specific steps.

This procedure covers continuously operated packed distillation columns and batch columns operating either at total reflux or with distillate returned to the stillpot. The packings include all types of randomly dumped, structured and grid type packings with appropriate supports and distributors. Testing of auxiliary equipment is not included; however, capacity may be restricted by auxiliaries. Other established procedures are available for checking the capacities of the auxiliary equipment.

103.0 Liability

The AIChE and members of the various committees involved make no representation, warranties or guarantees, expressed or implied, as to the application or fitness of the testing procedures suggested herein for any specific purpose or use. Company affiliations are shown for information only and do not imply procedure approval by the companies listed. The user ultimately must make the judgement as to the testing procedures to utilize for a specific application.

200.0 DEFINITION AND DESCRIPTION OF TERMS

201.0 Flow Quantities (Refer to Figure 802.1)

201.1 *Feed* is the material to be separated, including multiple feed streams.

201.2 *Bottoms* is the high-boiling product leaving the bottom of the column (or the reboiler).

201.3 *Distillate* is the product distilled overhead. It may leave the distillation system as a vapor, liquid, or combination of both.

201.4 *Side-stream product* is a product withdrawn from an intermediate section of the column.

201.5 *Overhead vapor* designates the vapor from the top of the column and includes material to be condensed for reflux. It is the combined distillate and overhead reflux.

201.6 *Reflux* is used to designate the quantity of liquid returned to the column.

201.6.1 *Overhead (external) reflux* is the quantity of liquid returned to the top of the column. Overhead reflux may be subcooled.

201.6.2 *Internal reflux* is the quantity of liquid leaving the top theoretical stage.

201.7 *Throughput* refers to the combined liquid and vapor traffic passing through a cross section of the column.

201.7.1 *Internal liquid* is the calculated quantity of liquid flowing from point to point in the column.

201.7.2 *Internal vapor* is the calculated quantity of vapor passing from point to point in the column.

201.7.3 *Entrainment* is the liquid carried upward from one point to another by the vapor stream.

202.0 Key Components

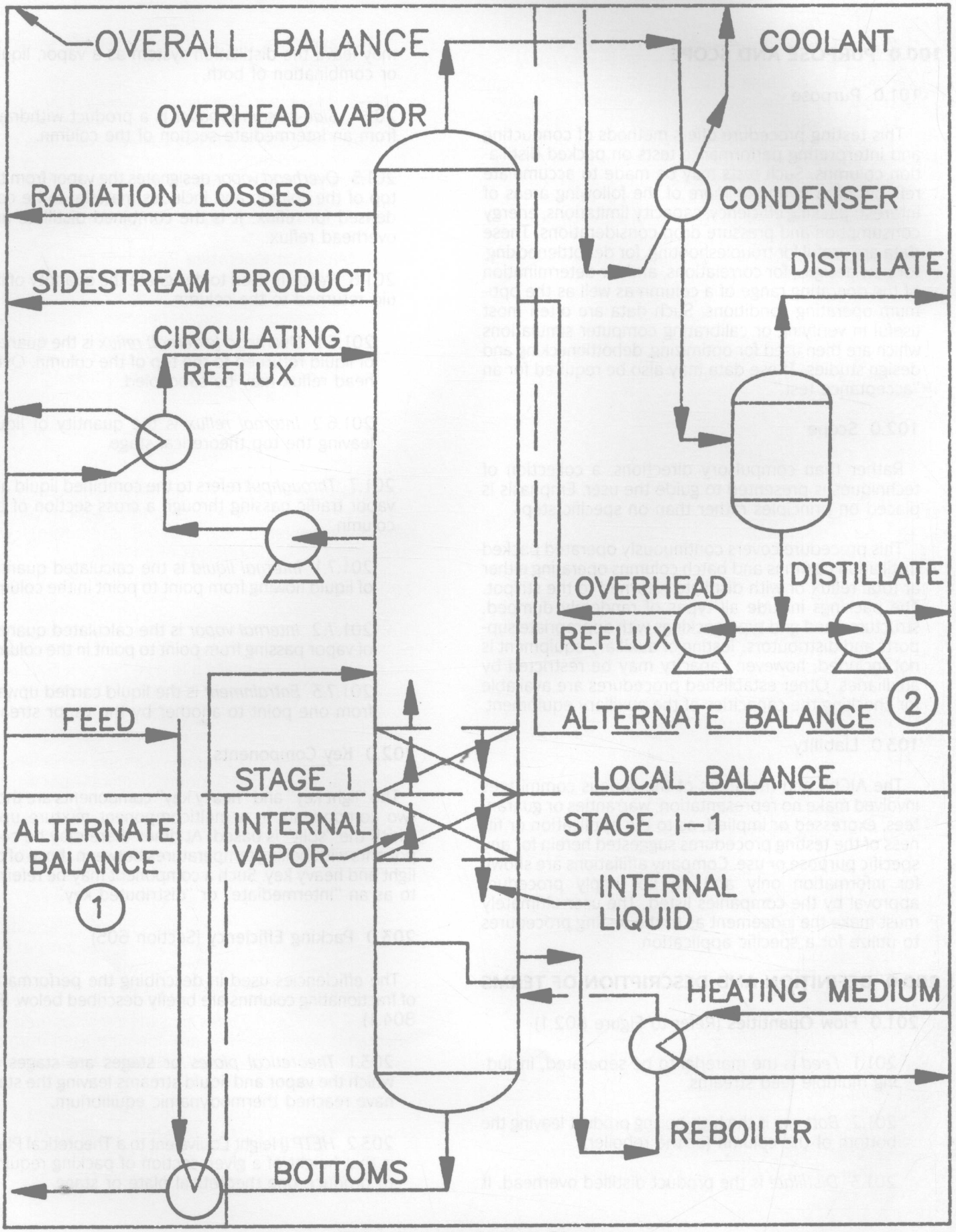
The "light key" and "heavy key" components are those two components in a multicomponent mixture upon which the "split" is based. At times there may be components with boiling temperatures between those of the light and heavy key. Such a component may be referred to as an "intermediate" or "distributed key."

203.0 Packing Efficiency (Section 605)

The efficiencies used in describing the performance of fractionating columns are briefly described below. (Ref 804.1)

203.1 *Theoretical plates* or stages are stages on which the vapor and liquid streams leaving the stage have reached thermodynamic equilibrium.

203.2 *HETP* (Height Equivalent to a Theoretical Plate) is that height of a given section of packing required to produce one theoretical plate or stage.



802.1 Enthalpy Balance Diagram

203.3 *Overall column efficiency* refers to the performance of the column as a whole. The *HETP* for the column as a whole is the ratio of the overall height of packing in the column to the number of theoretical stages in the column. The number of theoretical stages in the column is the total number of theoretical stages required for the separation less the sum of theoretical stages equivalent to the reboiler and to the partial condenser. This efficiency is useful in comparing one test with another or in comparing the test with design. The overall efficiency of sections of a column may also be of interest. For example, the overall efficiency below the feed point may be different from the overall efficiency above the feed.

203.4 *HTU* (Height of a Transfer Unit) is the height of packing required to accomplish a separation of standard difficulty. It is theoretically related to mass transfer coefficients and, for heat transfer, to the heat transfer coefficient. (See Section 605.2.)

204.0 Operating Lines

These are the material-balance lines on a McCabe-Thiele type of diagram for a binary system (Ref 804.2). The use of operating lines has been extended to multicomponent systems (Ref 804.3).

205.0 Pinch

This term describes a local condition within the column under which there is no appreciable change in composition of the liquid or vapor components from point to point when such lack of change is due to lack of mass transfer driving force (not column malfunctioning, flooding, high entrainment, dry packing). For a binary system a pinch is graphically depicted when an operating line is approaching or intersects the equilibrium curve on a McCabe-Thiele diagram.

206.0 Maximum Throughput

206.1 *Maximum hydraulic throughput* is defined as the highest loading at which a column can operate without flooding. Since the loading is affected by both liquid and vapor rates, there are many combinations of those rates which define a maximum hydraulic throughput curve. This maximum may in practice be limited by the packed bed or one of the internals.

206.1.1 *Flooding* describes the condition of the column when the throughput capacity is exceeded. At loadings slightly exceeding the flooding point, liquid accumulates uncontrollably and continued operation becomes impossible. The flooding point depends on both vapor and liquid velocities, system properties and packing geometry. (Ref 804.105) The flooding point can be recognized by the existence of one or more of the situations described in Section 502.

206.2 *Maximum Operational Capacity* is the highest loading at which stable operation giving an acceptable over-all column efficiency is achieved. This point may occur well below the column's maximum hydraulic capacity. Since loading involves both liquid and vapor, a maximum operational capacity curve can be defined on a plot of liquid vs. vapor rates.

207.0 Minimum Operating Rate

Is the smallest loading at which acceptable performance is obtained. The separation may become unacceptable because of loss of packing efficiency due to incomplete wetting of the packing, liquid or vapor maldistribution, or because of column instability. (The operating range of a liquid distributor is very often much less than that of the packing).

208.0 Operating Section

An operating section of a column is defined as a portion of a column to which no feed is added, product removed nor external heat is added or removed. It may consist of one or more beds of packing.

209.0 Hardware

A packed column consists of one or more sections of packing, each section having internals consisting of a liquid distributor and usually a bed limiter or a hold down plate at the top and support plate at the bottom (See Figure 802.7.) Hardware is described more fully in Section 604.

209.1 *Packing* is randomly dumped or structured material used inside columns to effect vapor and liquid contact. (Sect. 604.2, Figure 802.7, 802.8).

209.2 *Support plates* are devices installed at the bottom of a packed section which support the packing while allowing vapor and liquid to flow freely into and out of the bed.

209.3 *Bed Limiters* are devices fixed in place above a packed section to retain packing elements at high vapor flow rates. They are used with metal or plastic packings and may be part of the liquid distributor.

209.4 *Hold Down Plates* are similar devices used with ceramic packings. They are not supported from the column wall but rest directly on the packing, preventing packing fluidization.

209.5 *Liquid Distributors* are devices installed at the top of a packed section by which liquid entering the section is distributed uniformly over the top of the packing.

209.6 *Liquid Redistributors* are devices used to improve column performance by collecting and redistributing liquid downflowing from a packed section above.

209.7 *Vapor Distributors* and *Flashing Feed Distributors* have the added feature of distributing incoming vapor over the underside of a packed bed.

300.0 TEST PLANNING

301.0 Preliminary Preparation

The cost of performing plant tests goes far beyond the time and material expended during the actual test run. Careful planning and preparation are essential to maximize the economic and technical benefits of a test. (See Ref 804.4, 804.106 for an in-depth discussion of preliminary test preparation).

301.1 Test Objectives

Specific objectives should be stated for the plant test. If the test is proposed for plant troubleshooting, a logic diagram similar to Figure 802.2 is useful for defining the nature of the problem and in formulating test objectives (Ref 804.5). This phase of test planning should include techniques for determining whether or not the objectives are being met while the test is in progress.

301.2 Organizational Resources

The test objectives help determine which organizational resources should be involved in the test. Plant tests frequently involve people from many disciplines such as plant maintenance, analytical services, research, design organization representatives, and equipment supplier representatives.

301.3 Schedule

It is best to schedule the test for a time where the plant is expected to run at the desired rates under as stable conditions as possible. In setting the schedule, consideration should be given to availability of plant feedstocks and raw materials, conditions of upstream and downstream units, the time it will take to complete the required preparations, personnel availability, availability of analytical services and the anticipated ambient conditions. It should be realized that the test may be conducted under extremely unfavorable conditions, and alternative plans should be provided. The length of the test will depend on the test objectives, the number of conditions tested, and the time required to reach steady state for each test condition. (See Sect 503.0.)

301.4 Column Control and Instrumentation

The column control scheme should be well understood by the test participants so that the column's response to deliberate or consequential disturbances can be anticipated. Equipment diagrams showing the locations for temperature and pressure measurement and sample points should be available. Accuracy provided by column instrumentation is an important factor for a successful test. Critical instruments should be recalibrated prior to starting the test and controllers retuned for test stability if necessary. If direct flow metering is not possible on critical streams, plans should be devised for calculating the flows based on known data (where flow meters are not installed, external bolt-on meters may possibly be used). Preparations must be made for piping, instrument and equipment changes where necessary to obtain the operating conditions, samples, and data required.

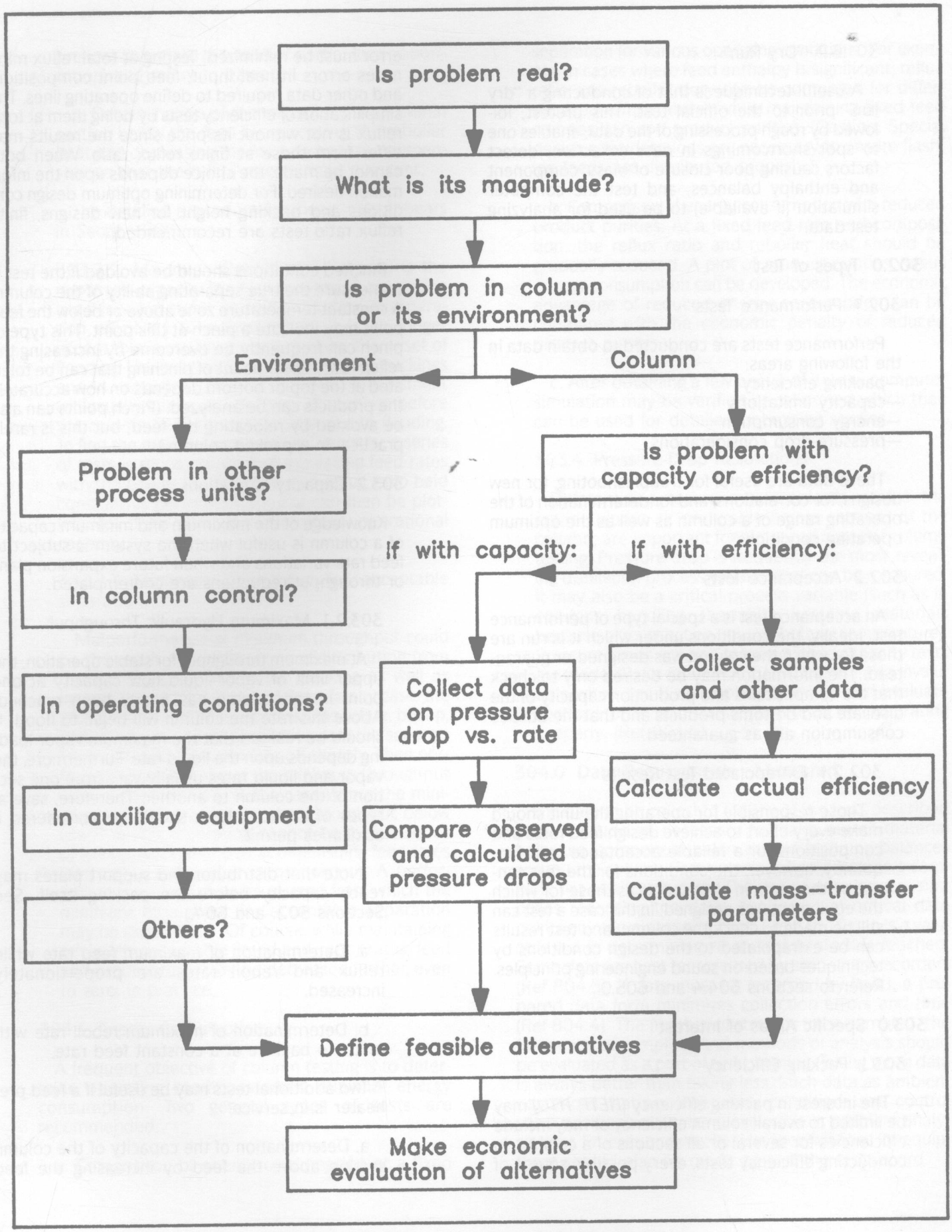
301.5 Peripheral Equipment

Potential limitations of peripheral equipment such as reboilers, condensers, pumps and valves should be considered. A good supply of feed of the desired compositions, temperature and pressure must be available at a correct and steady rate. Effects of the test on downstream equipment or other processing units must be considered. This is especially important when testing distillation columns that are thermally integrated with other operations.

301.6 Pretest Calculations

301.6.1 Process Simulation

Simulation of anticipated test conditions is helpful when computer models of the process are available. Flow diagrams showing enthalpy and material balances are instructive for test participants. Such simulations may identify key debottlenecking parameters such a column pressure, reflux ratio, etc. and help to define the ranges of the variables to be tested. Frequently, results from simulating a sequence of step changes in operating conditions can be shown graphically. These graphs are useful for comparing actual and anticipated test results while the test is in progress. Another useful pretest calculation is the expected pressure drop for zones of packing and for the entire column. The importance of these calculations is discussed in Sections 303.4, 502.1 and 604.0. The pretest calculations will require the accumulation of physical and thermodynamic property data as summarized in Section 305.



802.2 Logic Diagram for Column Troubleshooting

301.6.2 "Dry Run"

A useful technique is that of conducting a "dry run" prior to the official test. This pretest, followed by rough processing of the data, enables one to spot shortcomings in data collection; detect factors causing poor closure of mass, component and enthalpy balances; and test the computer simulation (if available) to be used for analyzing test data.

302.0 Types of Test

302.1 Performance Tests

Performance tests are conducted to obtain data in the following areas:
—packing efficiency
—capacity limitations
—energy consumption
—pressure-drop considerations.

These data are useful for troubleshooting, for new designs, for correlations, and for determination of the operating range of a column as well as the optimum operating conditions.

302.2 Acceptance Tests

An acceptance test is a special type of performance test. Ideally, the conditions under which it is run are those for which the column was designed or guaranteed. The information may be desired only to check that the compositions and production capacity of the distillate and bottoms products and that the utilities consumption are as guaranteed.

302.2.1 Extrapolated Test Results

Those responsible for operating the unit should make every effort to achieve design feed rates and compositions for a reliable acceptance test. Frequently, however, the conditions for the test cannot be the same in all respects as those for which the equipment was designed. In this case a test can still be made to check the column, and test results can be extrapolated to the design conditions by techniques based on sound engineering principles. Refer to sections 604.4 and 605.0.

303.0 Specific Areas of Interest

303.1 Packing Efficiency

The interest in packing efficiency *(HETP, HTU)* may be limited to overall column efficiency or may include efficiencies for several or all sections of a column. In conducting efficiency tests, every possible source of error must be minimized. Testing at total reflux minimizes errors in heat input, feed point composition, and other data required to define operating lines. The simplification of efficiency tests by doing them at total reflux is not without its price since the results may differ from those at finite reflux ratio. When both cannot be made, the choice depends upon the information desired. For determining optimum design conditions and packing height for new designs, finite reflux ratio tests are recommended.

Pinched conditions should be avoided if the test is to measure the true separating ability of the column. A constant temperature zone above or below the feed point may indicate a pinch at this point. This type of pinch can frequently be overcome by increasing the reflux ratio. The amount of pinching that can be tolerated at the top or bottom depends on how accurately the products can be analyzed. (Pinch points can also be avoided by relocating the feed, but this is rarely practical in a packed column.)

303.2 Capacity Limitations

Knowledge of the maximum and minimum capacity of a column is useful when the system is subject to feed rate variations and when future expansion plans or throughput reductions are contemplated.

303.2.1 Maximum Hydraulic Throughput

At maximum throughput for stable operation, the upper limit of vapor-liquid flow capacity at one point in the column has nearly been reached. Above this rate the column will begin to flood. It should be realized that the maximum vapor loading depends upon the liquid rate. Furthermore, the vapor and liquid rates usually vary from one section of the column to another. Therefore, several types of capacity tests should be considered if auxiliaries permit:

(Note that distributor and support plates may reach capacity before the packing itself. See Sections 502. and 604.)

a. Determination of maximum feed rate while reflux and reboil rates are proportionately increased.

b. Determination of maximum reboil rate with reflux to balance at a constant feed rate.

Two additional tests may be useful if a feed preheater is in service:

a. Determination of the capacity of the column section above the feed by increasing the feed

preheat (even into the zone of partial feed vaporization) and reflux to balance.

b. Determination of the capacity of the column section below the feed by increasing the reboiler heat and decreasing the feed preheat to compensate so that the reflux rate remains constant.

The detailed procedure for these tests appears in Section 500.

303.2.2 *Maximum Operational Capacity* is the maximum capacity at which acceptable separation is achieved in stable operation. A drop in separation efficiency to a level where it is no longer acceptable is usually associated with the onset of flooding. However, with low pressure drop packings and in vacuum distillation excessive entrainment will usually cause an efficiency drop much before the column becomes inoperable due to flooding. To find the maximum operational capacity, a series of tests is recommended at increasing feed rates with the boilup to feed ratio and reflux ratio held constant. A performance curve can then be plotted to determine the maximum operational capacity.

303.2.3 Minimum throughput for acceptable separation

Malperformance at minimum throughput could be the result of liquid/vapor maldistribution from the distributor or within the packing as well as incomplete wetting of the packing. In any case, the procedure would be to decrease the reflux, boilup, and feed by the same proportion until the separating action of the column falls off sharply. Then perform the test just above the minimum throughput value so determined. In the case where the minimum throughput is to be that throughput below which design separation cannot be met, a series of tests is recommended at diminishing feed rates (and reflux and boilup in proportion). A performance curve can then be plotted from which the minimum throughput for acceptable separation may be determined. Of course, while maintaining minimally acceptable liquid and vapor rates, feed and product rates may be decreased further, even to zero, in practice.

303.3 Energy Consumption

A frequent objective of column testing is to determine operating conditions which minimize energy consumption. Two general types of tests are recommended:

a. Comparison of energy consumption at a fixed separation for various operating conditions. For example, in cases where feed enthalpy is significant, reflux ratio and reboiler duty can be compared for different feed temperatures while maintaining fixed feed composition and product purities. (Caution: Special design considerations are necessary to ensure flashing feeds are distributed properly.)

b. Comparison of energy consumption with reduced product purities. At a fixed feed rate and composition, the reflux ratio and reboiler heat should be gradually reduced. A plot of product purities versus energy consumption can be developed. The economic advantage of reduced energy consumption can be compared with the economic penalty of reduced product purity.

c. After obtaining a few points of data, a computer simulation may be verified or calibrated which then can be used for detailed optimizing studies.

303.4 Pressure-Drop Restrictions

Differential pressure measurements across the entire column, or perferably across sections of the column, are important for almost all types of column testing. Pressure drop is frequently the most revealing distillation process variable that can be measured. It may also be a critical process variable (such as in columns handling thermally unstable materials because of its effect on column bottom temperature). Pressure drops are also required when capacity tests are made. (Refer to Sect 502.1). These are also invaluable when one is attempting to locate and evaluate sections of a column suspected of not operating properly. (Ref 804.5)

304.0 Data Requirements—Measured

The data to be collected for any of the tests described above include all necessary to make overall material balances, component balances and enthalpy balances around the column. Test planning should include a complete list of all such data. The use of data loggers simplifies recording the data. A printout of critical data points at intervals ranging from 1 min to 10 min is a valuable aid in determining when steady state is reached. Some data are best represented by strip-chart recorders (Ref 804.5). If data must be recorded by hand, a prepared data form minimizes collection errors and time (Ref 804.4). The number of streams to be sampled, the frequency of sampling, and methods of analysis should be evaluated as a part of test planning. Taking more data is always better than taking less. Such data as ambient conditions, battery limit utility conditions and control valve positions may prove invaluable in problem analysis later. Process and mechanical specifications for equipment should also be made part of the test record.

304.1 Conditions of External Streams

Conditions of external streams must be known to allow computation of overall and component material and heat balances.

304.1.1 Overall and Component Material Balances

The flow rate, temperature, pressure, and composition of the feed stream, distillate product stream, bottoms product and side-stream products must be obtained. In the case of two phase feeds, the temperature and pressure conditions at the last point where the feed is known to be single phase, should be obtained. The flow rate, temperature and composition are required for the reflux liquid stream returning to the top of the column, for any circulating reflux in and out of the column, and for any special streams such as the solvent in extractive distillation or the entrainer in azeotropic distillation. The pressure and temperatures of the overhead vapor should also be obtained. (Where one such variable is theoretically redundant, it may be used as a check.)

304.1.2 Overall Enthalpy Balance

To confirm the overall enthalpy balance it is necessary to determine the heat quantities added to or removed from the system by measuring the flow rate, temperatures and pressure (where vapor exists) of the heating or cooling fluids from exchangers such as reboilers, condensers, feed preheater, etc., passing through the enthalpy envelope. (See Fig 802.1.) These heat quantities, together with the enthalpies calculated for the feed and products, will permit checking the overall enthalpy balance. Radiation and convection losses are usually negligible except in special cases. (Vacuum columns are an example since the flow rates are small compared to the size of the equipment.)

304.2 Internal Temperatures

Temperature measurements at several points within the column are extremely useful in aiding the calculations of internal flows and in analyzing the performance of the column.

304.2.1 Heat Balances

If heat balances are to be hand calculated, the temperatures at points where the maximum or minimum flows occur are required. (Refer to section 603.2). This permits making a heat balance with the envelope intersecting the column at these points so that the internal flows may be calculated.

304.2.2 Internal Profiles

As discussed in Section 603.2, the calculation of internal flow rate and temperature profiles is much easier (and usually more accurate) when a distillation computer simulation program is available. It is still beneficial to have temperature measurements at several points within the column to be compared with the calculated profiles. However, measured temperatures where maximum or minimum flows occur would not be necessary.

In tests where efficiency is to be determined and where a critical analysis of the separation performance is desired, a carefully measured temperature profile of the whole column is advantageous. Ideally, the temperature points should be strategically placed to cover the zones of maximum temperature change. Practically, the temperature points should be evenly distributed about every five theoretical stages. In multicomponent columns where there are moderately large amounts of non-key components in the feed, the composition of the non-keys changes rapidly at the feed point and near the bottom and top of the column; consequently these regions may have large temperature changes.

304.3 Internal Samples

Samples of vapor and liquid from within the column are frequently difficult to obtain and should be attempted only if absolutely necessary to achieve the test objectives. Obtaining a representative sample of the vapor from a large column is unlikely. It is strongly recommended that one rely on liquid samples, obtaining the average vapor composition leaving a stage by calculation. Samples taken above and below the feed point may be desirable for facilitating interpretation of test results.

304.3.1 Internal Samples for Efficiency Checks

For accurate efficiency determination where practicable, internal samples (preferably liquid) should be withdrawn from a number of points in the column sufficient to establish a complete concentration gradient along the section in question. (See sections 303.1 and 405.0.)

304.3.2 Internal Samples for Overall Performance

In tests for maximum and minimum throughputs and overall column efficiency, internal samples are not required since internal compositions can be

estimated accurately enough for estimation of fluid densities and enthalpies. This point should be checked, however, by a preliminary heat balance.

304.4 Pressure Profiles

Pressure-drop measurements are always desirable, especially in packed columns. (See Sect 303.4.) Accurate differential pressure meters or manometers are required. (See Sect 404.)

305.0 Data Requirements—Physical Properties

305.1 Test Mixtures

The test mixture must be suited to the column being tested. In particular, the relative volatility and composition must be such that extremes of purity are not reached at either end of the column. Such extremes can exaggerate the effect of errors in composition due to the pinches that result. Also, where one component is in low proportion, it can be largely held up in the column, reflux drum or sump, leading to difficulty in reaching steady state. Most convenient, of course, would be the use of a mixture of which the column was intended, but because of the reasons cited above, this may not be practical. Descriptions of test mixtures suited to various heights of columns and operating pressure are available (Ref 804.95, 804.96).

305.2 Essential Data

Essential physical property data must be available for calculating and interpreting the performance data. Essential data of vapor and liquid streams over the range of column conditions include densities, molecular weights, latent heats and heat capacities (or enthalpy correlations). Heats of solution should be included when this property has an appreciable effect on stream enthalpies. Vapor-liquid equilibrium data are required for graphical stage counting or computerized column simulation. Accurate VLE data or correlations are essential for meaningful determination of theoretical stages. (Physical property data are available in a number of data bases sponsored by AIChE and major technical societies, worldwide. A number of texts also are devoted to the estimation of these properties.)

305.3 Auxiliary Data

Other physical properties useful in developing and testing correlations and for analyzing the performance data include viscosity, diffusivity, and surface tension.

306.0 Safety

Any equipment testing must conform to plant, industry, local, state, and federal safety regulations. It is recommended that all testing be conducted under the supervision of local personnel fully experienced in plant and equipment operating practices.

307.0 Environmental

The test procedure must conform to the latest requirements of applicable environmental standards which include plant, industry, local, state and federal regulations. Environmental conditions that apply to the equipment in normal operation should also apply during testing.

308.0 Test Procedure Documentation

The final phase of test planning should be a written document containing all pertinent test information. A complete document would include a summary of test objectives, manpower requirements, essential physical property data, pretest calculations, data collection procedures, sampling schedules, test sequence and any special safety and environmental considerations. Reference should be made to the relevant standard sampling procedures, equipment data, and safety and environmental regulations. A summary report for management could include only the test objectives, manpower requirements, safety and environmental considerations. Finally, a session should be held with the unit operators to fully explain the test and their duties and responsibilities during its operation (Ref 804.4).

400.0 METHODS OF MEASUREMENT AND SAMPLING

401.0 System Controls and Operating Stability

Reliable measurements and samples can only be obtained from a column operating smoothly within its design operating range. For example, pressure swings in a vacuum column will result in vapor rate fluctuations, temperature changes, and partial dumping of the liquid from the packing. Consequently, before rigorous testing begins, any serious deficiencies which interfere with the steady operation of the column should be diagnosed and corrected.

A few of the problems frequently encountered are listed as follows.

1. Changing feed compositions
2. Improperly tuned control loops
3. Changing steam or cooling water supply pressure
4. Oversized or sticking control valves
5. Surging reboiler
6. Liquid level controller failure

402.0 Measurement of Temperatures

Any reliable thermometer may be used, but thermocouples or resistance thermoelements placed in thermowells are preferred in commercial installations due to their ruggedness and availability. Before use, all temperature-measuring devices should be checked and calibrated in the temperature ranges to be used.

402.1 Accuracy

The temperatures should be measured with an accuracy such that the resulting heat balances meet the established error requirement (See Sect 603.1) For example, in measuring the heat removal from a condenser by water temperatures, measurements within a fraction of a degree are sometimes required. A relatively accurate temperature difference can be obtained by connecting two thermocouples in series opposing each other.

402.2 Errors

Errors in temperature measurement can still occur in spite of the fact that the sensing elements are accurate.

To eliminate errors due to conduction and radiation, adhere to recommended practices. (Ref 804.6). Thermowells must have an adequate length of insertion into the process fluid being measured. Both the thermowell connection as well as the external surface of the column in the vicinity of the thermowell must be well insulated. Good thermal contact between the sensor and the thermowell is important.

402.2.2 Errors due to stratification of a stream may be reduced by installing the thermowell in a location of high fluid turbulence such as downstream from mixing sections or bends. Part of the piping system may serve as a mixing section if heat losses from the piping are small.

Temperatures inside the column are sometimes measured by inserting the thermowell into the packing itself. In this case it is advisable, particularly in larger diameter columns, to install approximately three thermowells at each sensing level. These are equally spaced around the perimeter of the column, but each is inserted to a different depth into the packing. The average reading of these sensors is used as the measured temperature at the subject sensing level, while the differences between the average and individual readings can be used as a qualitative measure of liquid and vapor distribution across the column.

Liquid temperatures in the voids between packed beds are obtained directly under the packing support grid, in the liquid collector rings or trays, in the downcomers from the liquid collector trays, or in the liquid re-distributors to the bed below the void.

Temperatures of liquids in distributors receiving feed will most probably not be at equilibrium.

Accurate average vapor temperatures within columns are difficult to obtain, and should only be sought when the need is a serious. The accurate measurement of average vapor temperatures in voids between and below packed beds calls for probes which permit vapor to flow past them, but which are effectively shielded from liquid impingement plus other sources of conducted or radiated heat to or from the sensing point. Multiple probes at each vertical location, or a single probe with externally variable lengths of insertion into the column are recommended.

402.2.3 All thermowells should be inspected before a test is made in installations where deposits may occur that would result in erroneous measurements.

402.2.4 In thermosyphon and some other types of reboilers where the outlet line to the column contains two fluid phases, quite different temperature readings will result depending upon the location of the thermocouple in the outlet line, with respect to height. This is due to the continual flashing of the material as the hydrostatic head becomes less in the rising fluid. It is recommended that the thermocouple be placed as near to the column as possible in order to get a meaningful fractionator temperature. Of course a thermocouple directly at the reboiler outlet will be useful for observing reboiler performance.

402.2.5 Special attention is required to determine the correct enthalpies of feeds to columns. If a liquid feed coming from a preceding unit flashing at column pressure, the feed temperature must be measured at a location in the feed line or preceding unit where single phase liquid flow exists. If the feed is converted from a liquid to two phases in a feed preheater, the temperature of the liquid entering the preheater plus the heat input to the preheater per unit of feed are required to determine the feed enthalpy.

403.0 Measurement of Flow Rates

The rate of flow may be measured by means of instruments such as orifices, venturi meters, rotameters, displacement meters, vortex meters, mass flow meters, or by direct volume and weight measurement. Whenever possible, the instruments should be calibrated in place with the fluid to be measured, and at the temperature at which it is to be used. Meters must be accurate enough to the material and heat balancee requirements.(see Sect 602.0, 603.1)

403.1 Orifice Meters

Orifice meters have been extensively investigated, and their performance can be accurately predicted for a wide variety of conditions. Details concerning the construction, installation, and calibration of orifices and nozzles can be found in Reference 804.7. The procedure outlined in Reference 804.1 may also be followed.

The reading of orifice or venturi-type meters is affected by the density of the flowing material. If the density differs from that for which the meter was calibrated, the reading, if in volumetric units, must be corrected as follows:

$$\text{Volumetric flow rate} = (\text{Volumetric reading}) \times (\text{Design Density})^{1/2} / (\text{Actual Density})^{1/2}$$

If the meter is also designed to read volumetric flow corrected to a standard temperature and pressure, a further correction is needed if the density differs from the design density of the meter. The combined corrections give:

$$\text{Volumetric Flow Rate (at standard conditions)} = \text{Volumetric Reading} \times \frac{(\text{Actual Density})^{1/2}}{(\text{Design Density})^{1/2}} \times \frac{(\text{Design Density at standard cond.})}{(\text{Actual Density at standard cond.})}$$

For viscous materials an additional correction for viscosity may be required (Ref 804.7).

403.1.2 The orifice plate should be checked to verify that it is the proper size and that it is in good condition. Failure to check the orifice plate has ruined results of many test runs. Whenever possible, it is recommended that all flow meters be recalibrated for the purposes of the test run to read directly in weight units per unit time. Direct reading instruments facilitate immediate heat balances during testing, thereby helping identify errors in flow rates.

403.2 Rotameters

When rotameters cannot be calibrated in place, generalized charts from manufacturers are usually available. The theory developed by Head (Ref 804.8) may be used to convert the calibration from one fluid to that for another as well as for making corrections for variation in temperature and pressure. (see Ref 804.36, p. 5-16, for procedure)

403.3 Direct Volume or Weight Measurement

This should be made whenever practical as a check on other flow measurements. The fluid in tanks or gas holders is weighed or measured volumetrically at specificed intervals of time, and a plot of several measurements versus time is desirable. In situations where there are no steam meters or when it is desirable to check their accuracy, steam condensate can be collected into a large container. When accurately timed, this technique gives an accurate measure of steam flow rate. To minimize condensate flashing and for safety considerations, a condensate cooler should be used.

404.0 Measurement of Column Pressure Drop

An invariant top pressure is necessary for efficient packing performance, and for the proper operation of feedback controls based upon internal column temperatures. Consequently, the system selected to monitor the internal column pressure must have adequate sensitivity to record small percentage perturbations in the pressure and have adequate response so that significant variations are recorded rather than being damped out in the sensing system.

Differential pressure drops across packed sections are used to monitor and in some cases control the vapor rate through the beds, and to monitor the condition of the beds. Pressure taps should be provided so that the performance of each bed can be checked as needed.

404.1 Instruments

Pressure drops across packed beds are usually quite small, generally ranging from 0.1 to 1 inch of water per foot of packed height. Therefore, care is required to obtain readings which are accurate and reproducible to evaluate packing performance. Differential instruments must be used; pressure differences measured between separate gauges are not acceptable. If a pressure guage must be used, the same gauge must be moved and used for all readings.

Where safe to use, either vertical or inclined manometers, are the most convenient instruments for temporary measurement of pressure drop. They should be equipped on both sides with seal pots. They should also be inert gas purged on both ends to prevent condensate from collecting in them if the column vapors condense at ambient temperatures. Differential pressure cells are recommended for high pressure columns and for permanent installations. They should be calibrated before using in the test.

404.2 Pressure Taps

Column pressure taps must be located where there is no danger of submergence below a liquid level in the column. Gas or vapor filled lines leading to the pressure differential sensing device are preferred where applicable. Instruments which use liquid-filled capillaries to transmit pressures from diaphragms to the differential pressure sensor are subject to pressure measurement errors. The errors are especially acute in systems with elevation differences or systems operating under vacuum.

Where practical to do so, the differential pressure sensing device should be installed high enough so that gas or vapor filled lines leading from the column to the sensing device drain freely back into the column. (1/2″ diameter lines are usually required for this to prevent liquid hold-up due to capillary action.). Low spots in these lines which can collect condensate should be avoided. Line diameters should be adequate so that pressure drops due to gas purging or minor leaks are negligible, and so that the lines are free draining. Provision must be made to drain or blow condensate from the sensing system during startup and normal operations without interfering with the performance of the column. When there is a possibility of vapors condensing and freezing in the sensing lines, the lines must be heat traced or jacketed, or continuously purged with an inert gas or vapor. Pressure tap lines should also be purged or filled with a suitable liquid or gas if the material in the column is corrosive or toxic.

404.2.1 Gas Purge

Refer to Figure 802.3 for typical installation. This technique is the preferred method for keeping lines free from column vapors. The purge flow rate must be steady and low to avoid line pressure drop. A rotameter or bubbler is used to indicate the flow rate, which is adjusted by the needle valves. A pressure controller is installed to hold the purge-gas pressure constant at a value higher than the column pressure. Before operation, a pressure test of the system is advisable at column pressure with the purge gas. As an extra precaution, seal pots (Sect 404.3) will allow condensed vapors from the column to drain back to the column and will dampen pressure fluctuations. In high-pressure columns the reading will include the static head of high-density gas in the pressure taps as well as the static head of high-density vapor in the column. Compensate for the static head in the pressure taps by taking the zero reading at the operating pressure. Note the provision for blowing out the pressure sensing lines during startup and operations without disturbing the performance of the column. Gas purge systems are not recommended for systems in which build-up of non-condensables in the condenser cannot be tolerated.

404.2.2 Liquid Purge

The use of liquid may sometimes be more convenient than a purge gas, but the principles for gas purge would also apply for liquid purge. In addition, the installation should be made to permit the complete elimination of air bubbles above the liquid in the manometer to the seal pots on the tower. The zero reading is also made with purge liquid flowing at the desired rates with the side vents on the seal pots open. See Figure 802.4 for a typical installation.

404.2.3 Static Liquid Seal

A static liquid seal is used on columns where the process fluids must be kept out of the instrument lines but where continuous purge is impractical. The sealing liquid in this case should be immiscible with and heavier than the column liquid or condensate. Refer to Figure 802.5 for a typical installation. The installation should be made to permit complete elimination of air bubbles in instrument and lines to the seal pots, which are filled to the level of the column connections. The zero reading is made with the vent valves open.

404.2.4 Vapor-filled Lines

If the column vapors are not condensable at ambient temperature or if the lines can be steam or electrically heated to prevent condensation, no sealing fluid is necessary. The seal pots as shown on Figure 802.3 for the gas purge are still recommended, however, to trap column liquid which may surge into the line.