

The BASIS of IMPROVED *Electronics*

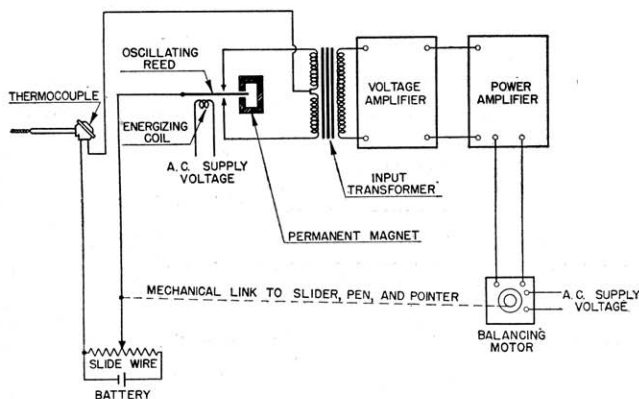
PROCESS instrumentation and chemical engineering have come a long way in the past decade. Consider for a moment how difficult it would be to conduct pilot plant developments or operate the great continuous processes of the chemical industry without precise, reliable measuring instruments which automatically and continuously indicate and record vital data. The industry's debt to instrumentation is high. Conversely, instrumentation is equally indebted to the industry because instruments are designed and developed to meet process needs. Instrumentation and processing are so interrelated and so interdependent that it would seem logical that instrumentation should be regarded by chemical engineers as a unit operation along with distillation, evaporation, crushing, and others.

Instrumentation has outgrown its swaddling clothes and no longer can be summarized in terms of mercury thermometers, pressure gages, flow meters, and the like. The utilization of electronic principles in industrial measurements has launched instrumentation into a new era.

ELECTRONIC POTENTIOMETERS

The increasing application of the self-balancing recording potentiometer to chemical processing has been brought about largely by the advent of basic improvements in the detecting and rebalancing mechanisms employed. One improvement is a direct result of using electrical means, other than the conventional galvanometer, for detecting changes in the measured variable. Further improvement has resulted by replacing the cyclic rebalancing mechanism with an electronically controlled motor which continuously rebalances the indicating or recording system to correspond to changes in the measured variable.

The detecting and rebalancing means used in one type of electronic potentiometer are illustrated schematically in Figure 1. Briefly, the direct-current millivoltage created by the thermocouple is changed to an alternating-current voltage of proportional magnitude



Courtesy of the Brown Instrument Company

FIGURE 1.—SCHEMATIC DIAGRAM OF ELECTRONIC POTENTIOMETER

DOUGLAS M. CONSIDINE
DONALD P. ECKMAN

*Brown Instrument Company,
Philadelphia, Pennsylvania*

in a converter. The converter is essentially a flat metal reed oscillating between two contacts connected to the opposite ends of the primary winding on an input transformer. The unbalanced direct-current voltage is impressed across the converter and the center tap of the primary winding on the input transformer. As the reed moves from one contact to the other, any unbalanced direct-current voltage will cause direct current to flow first in one direction through one-half the primary winding, then in the opposite direction through the other half. This action generates an alternating flux in the input transformer core, which in turn induces an alternating voltage on the transformer secondary.

The action of the converter is related to the alternating-current supply voltage by the energizing coil which is excited by the alternating-current supply voltage through a step-down transformer. The reed is polarized by a permanent magnet and, therefore, is actuated by the energizing coil to oscillate in synchronism with the a.-c. supply voltage. The reed closes one contact to the transformer for one-half the supply voltage cycle and the other contact for the other half, making one complete oscillation for each cycle. The direct current flowing in each half of the transformer primary winding, therefore, will create an alternating voltage in the transformer secondary of the same frequency as the supply voltage. This alternating voltage is amplified in voltage and power to where it will actuate a two-phase balancing motor.

If there is an unbalance between the slide wire voltage and the thermocouple voltage, the balancing motor functions to rebalance the two voltages by moving the slider on the slide wire. The direction in which the motor turns is determined by a definite phase relationship between the a.-c. supply voltage and the amplified a.-c. thermocouple voltage. This relationship depends, of course, upon whether the measured variable is increasing or decreasing in magnitude.

This principle in potentiometric measurement has brought about higher speed of operation, greater sensitivity and accuracy of measurement, and better reproducibility. Consequently, research laboratory investigations, pilot plant operations, and processing in the chemical industry can be performed under closer supervision and control. In addition to temperature, such process variables as pH, high vacuum, spectrographic quantities, rotative speeds, and mechanical strain are particularly adaptable to measurement with this type of continuously balancing potentiometer.

Higher indicating and recording speeds, down to approximately four seconds across scale for many of these

instruments, have brought about the first really adequate means for measuring multiple temperatures. Instruments indicating with speed and precision as many as 48 separate temperatures permit scanning of a chemical unit operation as has never been done before. To plot data on a unit of operation where it is often necessary to know the temperatures at many intermediate points is but a matter of a few minutes. New processes are being aided in their development through the practicability of obtaining operating data quickly and efficiently.

Greater sensitivity of measurement has been accomplished through improvements in the rebalancing mechanism. In most cases, positive detection of changes as small as three-hundredths of one per cent of full scale is realized. On a pyrometer calibrated from 0 to 300°F., for example, this sensitivity will allow consistent measurement of changes in temperature as low as nine-hundredths of one degree.

The advantages of higher speeds of operation and greater sensitivity of measurement are multiplied in automatic control. It has been shown that in automatic control any increase in the speed of measurement will result in a considerable increase in the speed at which the measured variable will be returned to the desired value. This fact is true since the period of cycling of a process under control is about four times the total lag of the system. If this lag is reduced by employing a faster means of measurement, then the process will stabilize much more quickly.

The advantage of low measuring lag is easily noted in the automatic control of processes where the total lag is small, such as in the control of blower air temperatures. In some cases it has been found that if the measuring lag is reduced to a minimum by employing the newer electronic types of potentiometers, proportional or throttling control may be replaced by the simple type of on-off or two-position control.

On those processes which have a relatively large total lag or large heat capacity, such as heat exchangers, an instrument with a high sensitivity of measurement is of great advantage to automatic control. For example, in controlling temperatures associated with heat exchangers, changes in load and other unpredictable variations in processing cause the controlled temperature to deviate slowly from the desired value. The newer types of potentiometer controllers, which enable speedier response to small changes, apply a corrective action much sooner and reduce the deviation of temperature from the desired value.

The use of electronic and electro-mechanical components has virtually eliminated the multiplicity of moving mechanical parts common to previous potentiometers. Longer life is attained because of reduced wear on fewer moving parts. Reproducibility is also improved as a result of reduction in wear. The sturdiness and reliability of the electronic potentiometer has been proved in severe service. The absence of a delicate galvanometer movement and its associated mechanism make an instrument which, in many re-

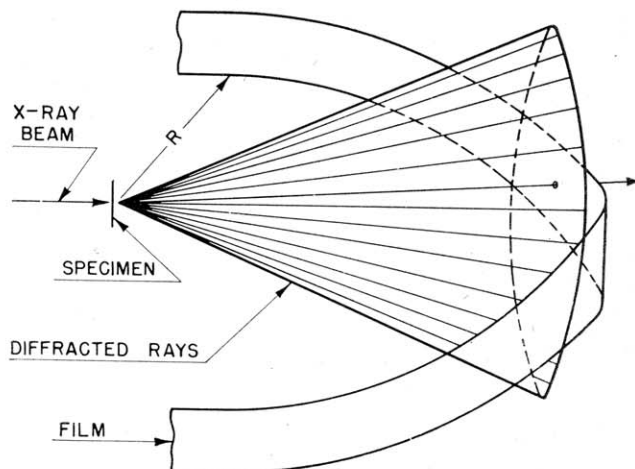


FIGURE 2.—FILM TYPE X-RAY SPECTROMETER

spects, is more powerful and rugged than the simplest pressure type thermometer.

The cases which follow point to a few of many applications which either are rendered possible through use of the newer types of potentiometers, or which are increased considerably in their over-all usefulness.

GEIGER-COUNTER X-RAY SPECTROMETER

The use of X-ray diffraction by the chemical process industries as an analytical tool to determine the ultimate structure of materials has been employed for several years, for example, (1) in the rubber industry—to control the addition of dyes, fillers, and accelerators to the rubber batch—and (2) in the battery manufacturing industry—to select manganese dioxide ores required to impart specific battery characteristics and performance.

Until recently the time and technique required to perform and evaluate an analysis have limited the usefulness of this tool. The development of an X-ray spectrometer which greatly reduces the time required for analysis and which practically eliminates the need for special techniques in performing analyses is principally the result of two achievements in electronics—namely, (1) the Geiger-Müller tube, and (2) the continuously balanced high speed recording potentiometer.

When a finely collimated, essentially monochromatic beam of X-radiation bathes a properly prepared sample

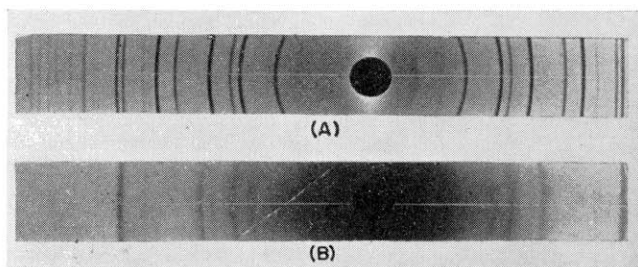


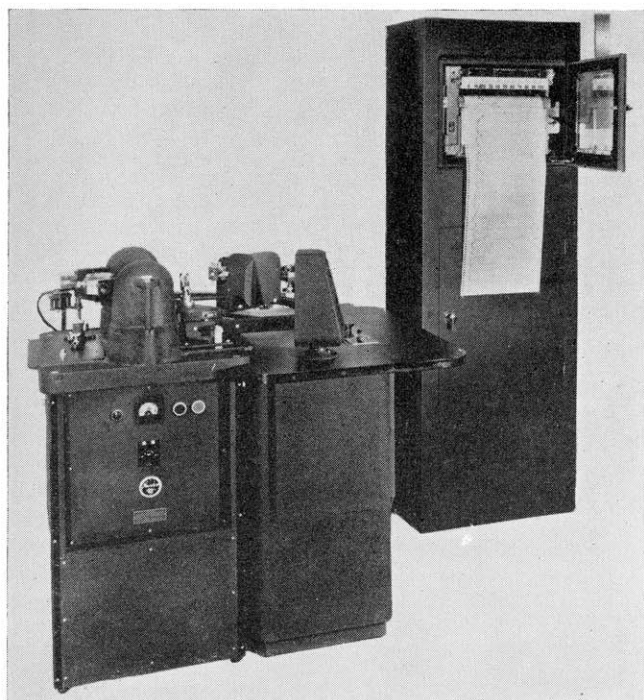
FIGURE 3.—DIFFRACTION FILM PATTERNS OF CATALYST SAMPLES
(a) Poor sample—sharp lines indicate grain growth and large particle size. (b) Good sample—film indicates small particles with large surface area.



Courtesy of North American Philips Company, Inc.

FIGURE 4.—GEIGER-MÜLLER TUBE

of material under test, a series of secondary reflected beams emerges. As illustrated in Figure 2, the secondary beams emerge from the sample in the form of a diffraction cone. By intercepting these beams with a strip of properly sensitized photographic film, a series of diffraction lines in the form of arcs of concentric circles is obtained (illustrated in Figure 3). The spacings of the lines are indicative of the materials that are present, and the densities of lines are indicative of the relative amounts of each material present in the sample. These lines occur wherever there is reinforcement of reflections from the surface and underlying atomic planes in the material. The phenomenon ap-



Courtesy of the North American Philips Company, Inc.

FIGURE 5.—RECORDING X-RAY DIFFRACTION SPECTROMETER

plies to all crystalline and to many amorphous substances. No two different materials have been found to exhibit the same diffraction pattern and hence identification by this means is positive.

With the photographic method considerable time is required (1) to set up the unit and prepare the film before exposure, (2) to expose the film (10 minutes minimum, 10 hours maximum), and (3) to develop the film after exposure. Once the film is developed, there remains a time-consuming manual task of measuring the line spacings and evaluating the line densities. Careful measurements and computations must be made to develop and to apply film shrinkage or extension factors since the film dimensions are affected during development. Likewise the density of the diffraction lines on the film versus the actual intensities of the reflected beams causing the lines is not a linear function, especially at the end zones. Integrity of results can be expected only for the middle ranges except through the adoption of rather involved time-consuming end-range techniques. It became obvious that improvements could be made on the photographic method.

The photographic film has been replaced by the Geiger-Müller tube, illustrated in Figure 4. This tube actually counts the quanta of X-ray energy reflected from the sample. The tube is prepared with close to 100 per cent efficiency for the characteristic X-ray wave length used in the spectrometer. In other words, one count is produced for almost every X-ray quantum entering the tube. It is interesting to compare this sensitivity with that of film which requires from 10^4 to 10^5 quanta to produce a diffraction line of minimum detectable darkness.

The Geiger-Müller tube is essentially a sealed tube containing an ionizable gas. Energy impinging on the tube causes partial ionization of the gas. The formation of a single ion pair anywhere within the active volume of the counter tube releases a flow of current sufficiently large to operate a relay directly. This current is amplified electronically over a range of 0 to 50 millivolts, which is fed to the high speed recording potentiometer illustrated in Figure 5. The recorder scale is evenly divided and is graduated from 0 to 100.

In the photographic method the strip of film is stationary. Where the counter tube is used, however, the tube is rotated about the sample so that it will scan the field of diffraction lines—that is, so that it will be progressively exposed to the various diffracted beams reflected from the sample.

Illustrated in Figure 6, the counter tube is rotated through 90 degrees of arc by means of a motor-driven scanning arm. This speed of rotation is precisely coordinated with the chart speed of the potentiometer to create an exact space relationship of the various intensity peaks and dips from the sample. Ninety minutes are required to obtain a complete diffraction pattern with the high-speed recorder. Where an automatic recorder is not used, it is necessary to position manually the scanning arm each 0.05 degree, requiring a total of 1800 positions.



FIGURE 6.—PARTIAL VIEW OF X-RAY SPECTROMETER

The type of pattern obtained with the electronic recorder is illustrated in Figure 7. This curve shows the proportions of cuprous and cupric oxides in a marine antifouling paint. The ratio of cuprous and cupric oxides must be held within close limits for optimum results, a difficult problem in the past. By use of the X-ray spectrometer, differentiation of the two products and determination of their relative proportions are easily effected.

The continuous high-speed operation of the recorder renders it possible to detect almost instantaneously the changes in the secondary radiation from the sample as the counter tube rotates. The fast pen speed of four and one-half seconds across scale and the high chart speed of 120 inches per hour make it possible to spread out the record, contributing to convenience and accuracy in analyzing the diffraction pattern.

THE RECORDING POLAROGRAPH

The Polarograph is an analytical instrument, both qualitative and quantitative, for determining the chemical constituents of either aqueous or nonaqueous solutions. Although extremely small quantities of material can be detected, it is not limited to micro analysis. Both in micro- and macro- analytical work, it replaces tedious and slow wet chemical methods for determining metallic and organic constituents.

The principle of the dropping mercury electrode, the basis of the Polarograph, dates back to 1873 when Lippman—using a capillary containing a stationary thread of mercury—first measured the surface tension of mercury successfully. Several years later Kucera—in making further studies of the surface tension of mercury—employed a dropping mercury electrode quite similar to the one now used in polarographic analysis. Kucera's method produced anomalous results which he could not explain nor eliminate from his experiments. In 1918 Heyrovsky—in attempting to find an explanation of Kucera's results—continued the investigation of electrocapillarity. A galvanometer was employed to measure the currents flowing through the electrode system, and it was discovered that the

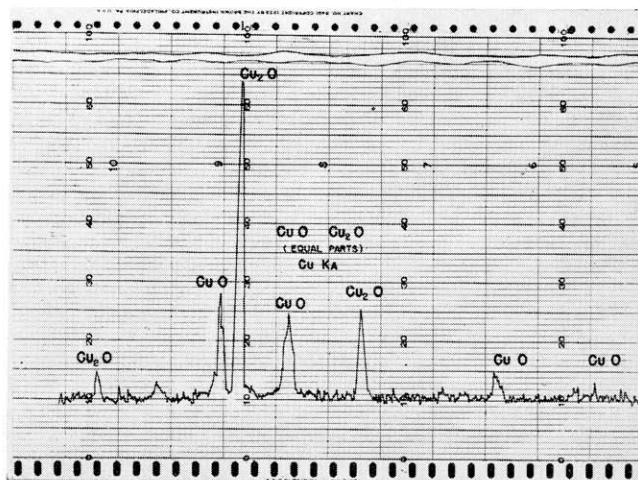


FIGURE 7.—TYPICAL X-RAY DIFFRACTION PATTERN OBTAINED WITH HIGH-SPEED RECORDING POTENTIOMETER

current-voltage curves of solutions which were electrolyzed with a dropping mercury electrode possessed several remarkable qualities, including: (1) the curves had a characteristic step formation, (2) the curves were a function of the nature of the solution and were reproducible, (3) the voltages at which the steps of the curves occurred were characteristics of the components in the reducible substance, permitting qualitative analysis, and (4) the height of the steps was a function of the concentration of the components of the substance in solution, thereby making quantitative analysis possible. The method of analysis was named polarography; the instrument developed by Heyrovsky was called the Polarograph; and the current-voltage curve was termed Polarogram.

The dropping mercury electrode, illustrated schematically in Figure 8, comprises a very fine bore capillary tube connected by a neoprene tube to a mercury reservoir and placed in the solution so that very fine drops of mercury are formed beneath the surface. The rate of mercury flow may be adjusted by varying the height of the mercury reservoir and normally

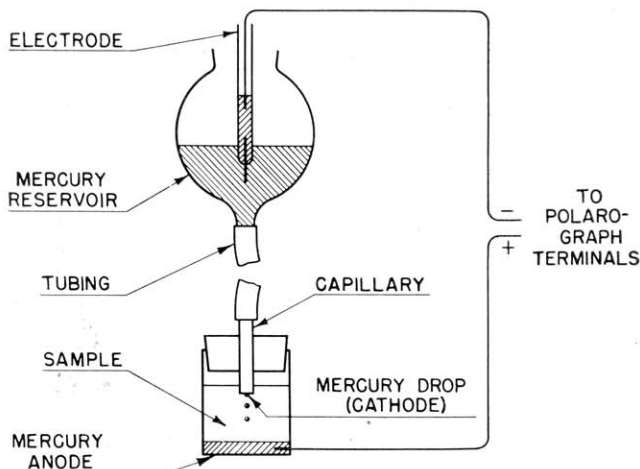


FIGURE 8.—DROPPING MERCURY ELECTRODE

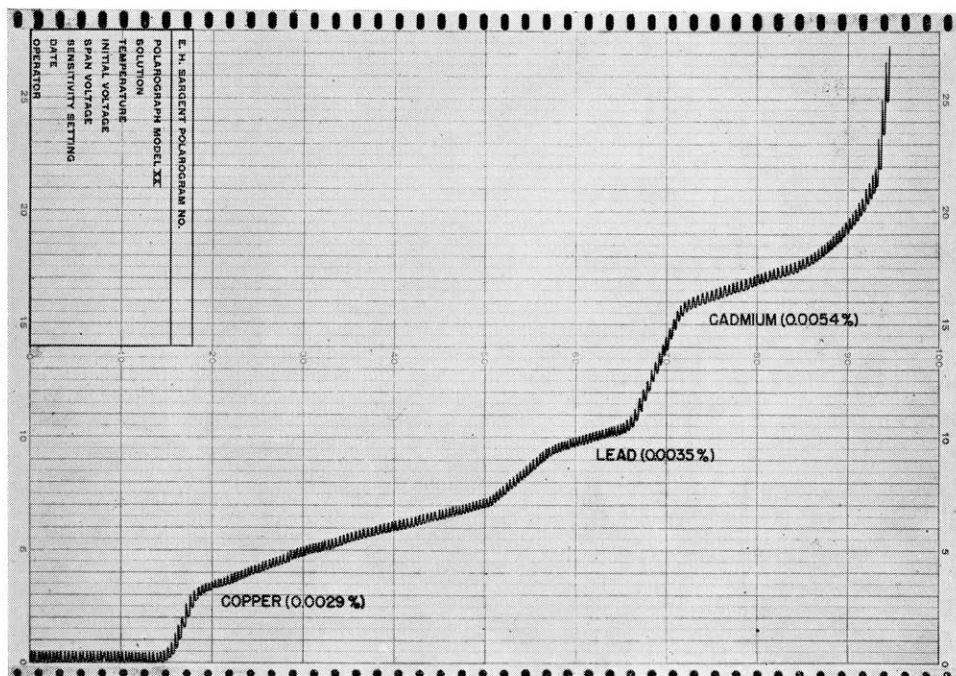


FIGURE 9.—POLAROGRAPH OF COPPER, LEAD, AND CADMIUM IN A HIGH-PURITY, ZINC-BASE, DIE-CAST ALLOY

amounts to a drop every one to three seconds. The mercury reservoir is connected to one terminal of the Polarograph; the other cell terminal is a pool of mercury in the bottom of the solution vessel.

In operation a few drops of mercury added to the sample act as one electrode of the cell. The dropping mercury electrode is next inserted. An inert gas, usually nitrogen, is then bubbled through the solution to remove atmospheric oxygen which is reducible and gives a polarographic wave not usually of interest to the analyst. As the voltage impressed on the cell is slowly raised, the current remains nearly constant until a voltage is reached at which a component of the solution is reducible. The current then rises sharply to a new level at which it remains constant until the voltage is reached at which a second component is reducible. Another sharp rise in current to a new constant value then occurs. These sharp rises in current are called polarographic steps or waves.

Since the polarographic steps for the various components in the sample occur at different voltages, several substances may be determined in one solution on a single polarogram, as illustrated in Figure 9. Note the small percentages of copper, lead, and cadmium determined in a high-purity, zinc-base, die-cast alloy.

During his early experiments Heyrovsky found that manual plotting of polarograms was laborious and subject to experimental error. An automatic instrument was needed with the following characteristics: (1) continuous operation to show all details of the curves, (2) fast operation since current-voltage changes occur rapidly, and (3) high sensitivity since the currents involved are often fractions of a microampere.

A photographic recorder was developed which partially answered the needs. Although a great improvement, photographic recording, as in the case of X-ray diffraction previously described, had many limitations, including: (1) time and labor required for photographic development of polarograms were considerable; (2) current-voltage curves were not visible while the analysis was being made. Visible chart recording permits prompt readjustment of operation conditions at any stage in the process of the polarograms; (3) precision was limited by the size of the record produced. With a strip chart record 12 inches wide, step heights of polarograms can be made very large, permitting greater accuracy in analyzing results.

The ruled chart also simplifies the linear measurements required. By employing a continuously balanced high-speed recording potentiometer the above limitations



Courtesy of E. H. Sargent and Company

FIGURE 10.—HIGH-SPEED VISIBLE RECORDING POLAROGRAPH

have been overcome. The new unit is illustrated in Figure 10.

The output from the polarographic cell is fed to the recorder so that the displacement of the pen from zero is determined by the amount of current flowing through the cell. The chart is driven by a constant speed synchronous motor. The polarographic bridge which supplies voltage to the cell is also driven by a synchronous motor so that the chart displacement is a linear function of the voltage applied to the cell—that is, the time ordinate of the chart becomes, in essence, the voltage ordinate. The record obtained, therefore, is current versus voltage. Since the recorder pen is continuously visible, the operator is in position to determine when a step is completed and to make immediate readjustments if the curve is too small or too large.

Examples of practical applications of the Polarograph to process control include (1) the determination of carryover of aluminum chloride catalyst in petroleum processing, and (2) the indication of residual oxides of nitrogen in high explosives.

ULTRAVIOLET AND INFRARED SPECTROPHOTOMETERS

Spectrophotometry is based upon the fact that various chemical substances, in either the gas, liquid, or solid form, absorb light at certain wave lengths. When light of a single color or wave length is passed through a layer of any substance, the light may be transmitted completely through the substance, or it may be partially or completely absorbed, depending upon the chemical identity of the substance and the amount of the substance in the path of the light.

If a plot is made of wave length versus percentage of light absorbed under various conditions an irregular curve of hills and valleys will result. The hills correspond to wave lengths or colors for which the substance is relatively opaque; the valleys correspond to wave lengths for which the substance is relatively transparent. In some cases the hills show sharp absorption peaks; in other cases the absorption maxima are broad.

The absorption curve for any substance is unique. No two chemical substances absorb light in exactly the same fashion. The absorption spectrum of a substance serves as a fingerprint to identify the substance when in pure form. When the substance is mixed with another, usually it will be found that at least some of the absorption peaks of the substance will not coincide with the absorption peaks of other substances.

Substances which appear colored to the eye obviously absorb light in the visible region of the spectrum. Bromine vapors and copper sulfate solutions are common examples. Many chemical substances, particularly organic compounds, are colorless—that is, they are transparent for all wave lengths in the visible regions. Such substances will nevertheless absorb radiation in invisible spectral regions, depending upon the types of chemical bonds in the molecules. Thus molecules which include the benzene ring in their structure, such as benzene, toluene, and xylene, show distinctive absorption patterns in the ultraviolet region. Con-

jugated diolefins, of which butadiene is the most familiar example, also absorb in the ultraviolet region. Ordinary saturated hydrocarbons, such as propane and butane, and simple unsaturated compounds, such as butylenes, do not absorb extensively in the ultraviolet. For the analysis of such compounds the infrared region of the spectrum is utilized.

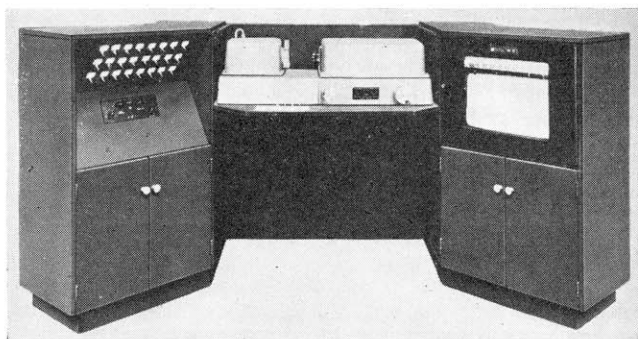
Ultraviolet Spectrophotometer: As illustrated in Figure 11, this instrument comprises (1) a source of ultraviolet radiation, usually a hydrogen lamp, (2) a monochromator to isolate radiation of any desired wave lengths, (3) an absorption cell through which a stream of the fluid being measured is continuously passed, (4) a phototube and electronic amplifier for measuring the amount of radiation transmitted through the sample, and (5) an electronic potentiometer which records the percentage of light transmitted by the sample. Chart readings of less than 100 per cent will be produced whenever the sample stream absorbs some of the radiation. By employing suitable calibration data, the per cent transmission can be correlated with the concentration of the measured substance in the fluid stream.

Infrared Spectrophotometer: In the infrared spectrophotometer, illustrated in Figure 12, a globar is used as the source of infrared radiation. Radiation from the globar is focused through a sample cell. Radiation transmitted by the sample cell is dispersed by means of a prism, usually made of rocksalt. By rotating a Littrow mirror, upon which radiation from the dis-



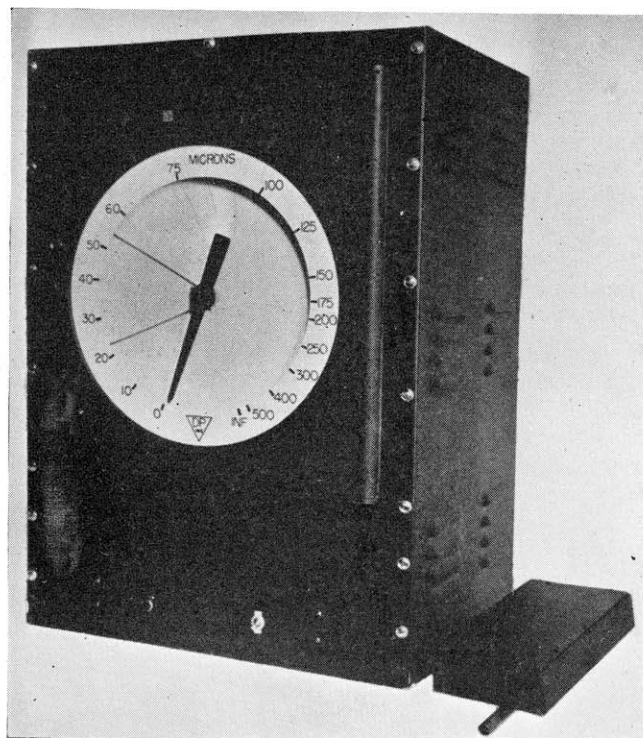
Courtesy of the National Technical Laboratories

FIGURE 11.—RECORDING ULTRAVIOLET SPECTROPHOTOMETER



Courtesy of the Perkin-Elmer Corporation

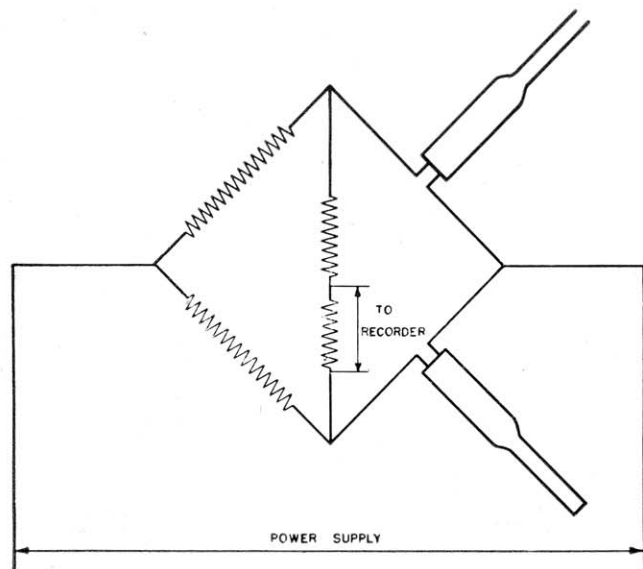
FIGURE 12.—HIGH-SPEED RECORDING INFRARED SPECTROMETER



Courtesy of Distillation Products, Inc.

FIGURE 13.—RECORDING PIRANI HIGH-VACUUM GAUGE

persing prism strikes, it is possible to select the radiation of desired wave length which will be focused upon the measuring thermocouple. The thermocouple is a high vacuum compensated type with a bismuth-bismuth tin hot junction. The e. m. f. generated by the thermocouple is on the order of one microvolt. The amount of energy for a given wave length picked up by the thermocouple is a function of the amount of radiation of that wave length absorbed by the sample.



Courtesy of Distillation Products, Inc.

FIGURE 14.—PIRANI CIRCUIT

By carefully rotating the micrometer wave-length control, which rotates the Littrow mirror, the complete infrared spectrum of the sample—from about 2.5 to 15 microns—is obtained. For automatic scanning of the spectrum where a high speed recording potentiometer is employed, the shaft of the wave-length micrometer is coupled to an electric motor drive. The thermocouple e. m. f. is amplified and recorded on a high-speed strip chart electronic potentiometer.

Advantages of continuously and automatically indicating and recording ultraviolet and infrared spectra include (1) a substantial reduction in the time and skill required to perform analyses is effected, (2) a permanent record is obtained which may be consulted for reference at any time, and (3) unknown absorption in samples often can be identified at a later date in the light of subsequent work, providing a permanent record is at hand.

MEASURING HIGH VACUUMS

Until recently an absolute pressure of 40 to 50 millimeters of mercury was considered a high vacuum. With exception of the manufacture of vacuum tubes, high vacuum techniques were generally confined to the laboratory. Today absolute pressures in the low millimeter and micron ranges are becoming commonplace and are making practical many processes formerly impossible to carry out and are improving the performance of many products.

Some processes in which high vacuums are now being applied include: (1) evaporation of metals and metallic salts, as in the manufacture of magnesium, surface plating of optical lenses, and metallic plating of cellophane; (2) drying blood plasma, penicillin, and other biologicals by sublimation; (3) distillation of vitamin oils, essential oils, and aromatics; (4) extraction of foods and drugs and the recovery of volatile solvents; (5) impregnation of coils, condensers, cables, armature windings, textiles, and wood; (6) manufacture of sealed unit refrigeration systems. The efficiency of the unit with removal of the last traces of water vapor and non-condensable gases is greatly increased; and (7) low temperature drying of foods and chemicals.

Continuous measurement of vacuums is indispensable to these processes because of the following reasons: (1) certain processes become inoperative if the pressure becomes too high. Continuous pressure measurement will indicate when the vacuum is more or less than that desired; (2) in certain processes there is a critical point at which undesirable effects will occur, if the pressures become too low. For example, arcing occurs in electronic drying if the pressures become too low; (3) leaks in vacuum equipment will be recorded by the pressure gage and an alarm will be set to give warning before damage occurs, such as melting in freeze-drying processes.

High-vacuum measurement roughly falls into three broad ranges—namely, 0 to 5000 microns, 0 to 500 microns, and 0 to 50 microns. Two types of high vacuum recorders are discussed in this paper—namely, (1) the Pirani Gauge, and (2) the McLeod Gauge.

Recording Pirani Gauge: The Pirani Gauge, illustrated in Figure 13, is essentially a coil of resistance wire mounted in an envelope which is connected to the system whose pressure is to be measured. The gage is connected into a Wheatstone bridge as one of the four arms. A second gage is usually sealed off at a pressure considerably lower than one micron of mercury, is connected to the bridge as a second arm, and is so arranged that changes in its resistance will balance changes in the gage tube resistance due to temperature fluctuations but not due to pressure changes. The remaining two arms of the bridge are equal fixed resistances. The measuring circuit is illustrated in Figure 14.

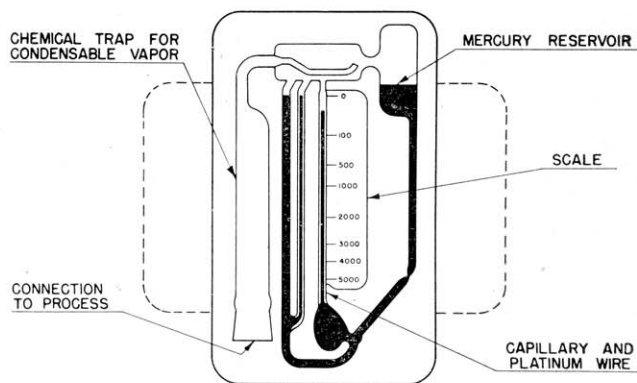
The heat loss from the filament of the gage tube, in pressure range from atmospheric pressure in one millimeter of mercury, is very largely due to convection and conduction. Below one micron of mercury the major heat loss is due to radiation and conduction. Between the pressures of one millimeter and one micron of mercury there is a considerable change in the heat loss from the filament due to the sharp change in the convection loss in this region, which causes its temperature to rise and this, in turn, causes its resistance to change and unbalances the bridge. This unbalance results in a current flow through the indicating arm of the bridge. A resistance placed in this arm will have an IR drop developed across it during unbalance which may be used to actuate a recording potentiometer. The potentiometer may be calibrated directly in terms of the pressure in microns.

The balance point of the gage, shown in Figure 14, is taken as the pressure of the sealed-off comparison tube, which means that the sensitivity is greatest at the low pressure end of the scale and least at the upper end of the scale. In the range of one micron a change in pressure as little as one-quarter micron may be observed, while at 500 microns a change of 10 to 25 microns may be the minimum change observable with accuracy. This change in sensitivity is due in part to the nonlinearity of the scale. The accuracy of calibration is of the order of ± 1 to 2 microns at the low pressure end, ± 10 to 15 microns at the high pressure end of the scale.

The pen of the potentiometer travels across scale in about 4.5 seconds so that even wide variations in pressure are almost instantly recorded. Contacts in the potentiometer recorder may be used for actuating alarm signals or even control circuits. Special sequence programs to fit particular applications may also be worked out with the potentiometer.

Recording McLeod Gauge: The McLeod Gauge after 70 years is still the ultimate standard for vacuum measurement and is used in the calibration of other vacuum gages. Noncondensable gases, such as hydrogen, cause no error in readings as with other types of vacuum gages. Condensable vapors, such as water or alcohol, can cause errors unless they are removed by a chemical trap or other means before they reach the measuring chamber of the gage.

The Stokes McLeod Gauge (Flosdorf-Hall) Type,



Courtesy of the F. J. Stokes Machine Company

FIGURE 15.—MCLEOD TYPE VACUUM GAUGE

illustrated in Figure 15, is now available for both measuring and recording high vacuums. Measurement is automatically made and recorded at regular intervals so that substantially a continuous record is obtained.

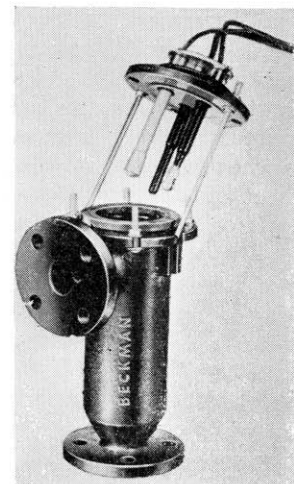
In Figure 15 the gage is illustrated in its measuring position. The gage is turned periodically, as shown dotted, each cycle to permit the measuring chamber to be exposed again to the pressure of the process. The height to which mercury rises in the capillary of the measuring chamber is determined, of course, by the pressure in that chamber. This height is electrically indicated.

To reduce the sampling-measuring cycle of the gage, advantage of a high-speed electronic recording potentiometer may be taken. The steplike action of a galvanometer-type potentiometer limits somewhat the speed with which the complete cycle can be made, as sufficient time must be allowed for the galvanometer to come to balance on the measuring end of the cycle.

Ultra-High Vacuums: For pressures lower than one micron of mercury, the ionization gage, with a range of 1 micron to 0.001 micron, and the Philip's Gauge, with a range of 20 microns to 0.01 micron, also can be adapted for use with recording potentiometers.

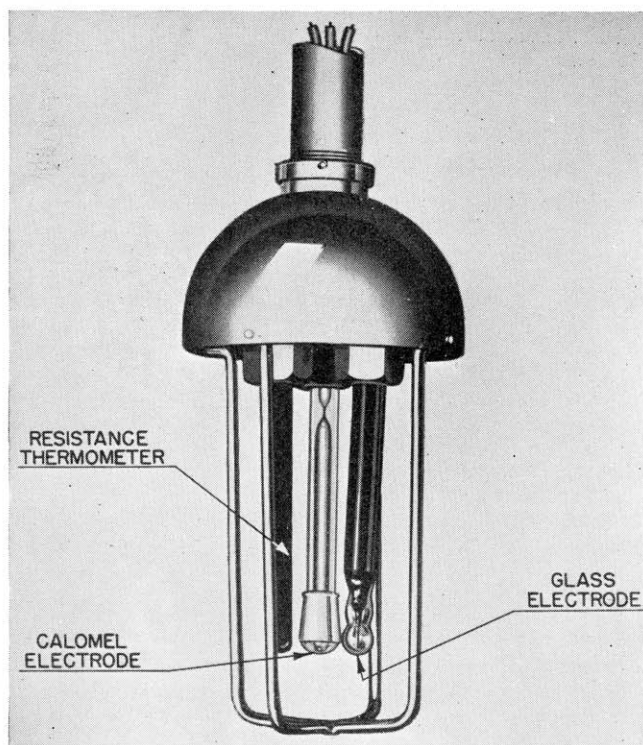
REFINEMENTS IN pH MEASUREMENT

Hydrogen ion concentration has been measured successfully in the laboratory for many years. The adoption of early laboratory measuring means to continuous indication and automatic recording and controlling of pH in industrial processes was not so successful. The industrial limitations of the hydrogen electrode, which requires a platinum catalytic surface



Courtesy of National Technical Laboratories

FIGURE 16.—FLOW TYPE pH ELECTRODE ASSEMBLY



Courtesy of the National Technical Laboratories

FIGURE 17.—IMMERSION TYPE pH ELECTRODE ASSEMBLY

and a pure supply of hydrogen gas, included (1) susceptibility of the catalyst to poisoning and (2) requirements for a continuous supply of pure hydrogen gas. Quinhydrone and antimony measuring electrodes offered some improvement for certain industrial applications, but these were (1) generally subject to errors in oxidizing and reducing solutions and (2) susceptible to poisoning by certain ions and salts.

Development of the glass electrode several years ago was the first real step toward industrial pH measuring and controlling applications. These first electrodes were not the complete answer, however. Considerable research into various types of glasses and into the measuring circuits was necessary before many of the shortcomings of the early glass electrodes could be overcome. The shortcomings included (1) limitation of the electrode to relatively low temperatures, (2) effect of alkaline salts, especially sodium ions, above a pH of 9, (3) fragile construction which made careful

handling mandatory, and (4) effects of changing humidity and stray currents upon the measuring system.

As a result of research during the past five years, the situation has been changed to the effect that (1) continuous pH measurements can be made in boiling aqueous solutions, (2) accurate measurement in highly alkaline solutions containing sodium ion concentrations of two mols per liter and up to a pH of 12 is obtainable, (3) delicate handling of the electrodes is no longer required, and (4) by improving the electronic measuring circuit and shielding the electrode leads, the effects of stray currents have been practically eliminated. It is not claimed, of course, that the ultimate in pH measurement has been reached, but recent progress has made industrial pH measurement and control possible where heretofore practical considerations seriously limited the applications.

Sturdy construction of electrode assemblies has also been responsible for a wider industrial usage of pH measurement. The flow assembly, illustrated in Figure 16, can be installed directly in process pipelines. Numerous materials of construction, including porcelain-covered iron, rubber, stainless steel, Monel metal, and Pyrex glass, can be used to fabricate the chamber. The immersion assembly, illustrated in Figure 17, is designed for permanent installation in tanks, vats, and other process vessels and is capable of operating under pressures of 30 pounds per square inch and over. In this assembly, note the three major parts—namely, the glass electrode, the calomel reference electrode, and the resistance thermometer. The resistance thermometer automatically and continuously compensates for the effects of temperature upon the resistance of the glass in the electrode.

Numerous special electrodes have been developed to satisfy peculiar process conditions. For example, an electrode assembly has been designed which does not expose any metal parts to the process solution. The production of penicillin requires a special electrode which permits the convenient measurement of pH of two liquid layers, a water layer, and a chloroform layer. pH measurement is important since it greatly affects the mutual solubilities of the liquids involved.

While the saturated calomel cell is generally used as the reference electrode, special mercury-less electrodes are available for processes, such as photographic film manufacture, where mercury cannot be tolerated.