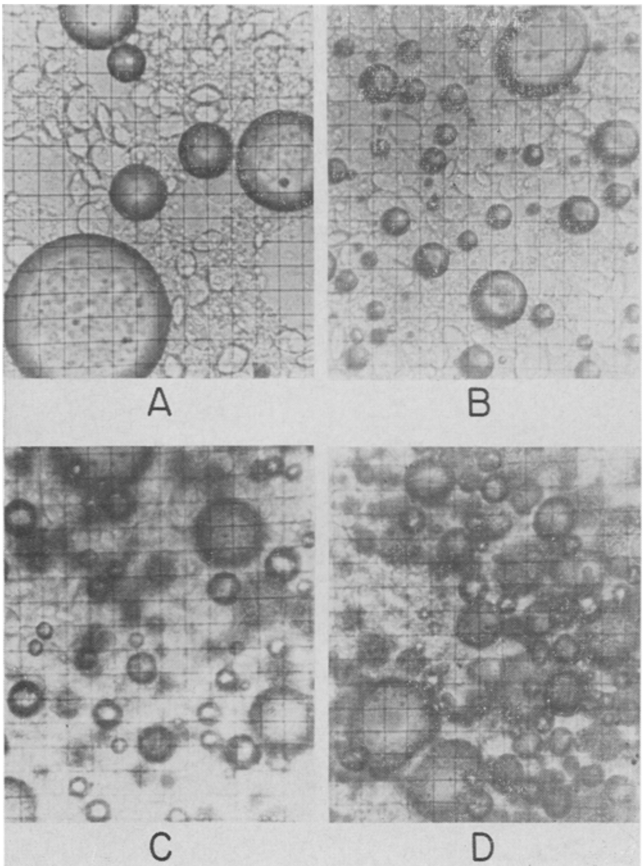

FIG. 5. Air bubbles in cake batters. Grid represents 9 microns between lines.

shown by Figure 5. In general, the more numerous the air bubbles in the batter, the higher will be the final volume of the cake, and the smaller the bubbles, the finer will be the texture of the cake (2). The batters shown in pictures 5A and 5B produced cakes of substandard volumes. Batter 5C produced a cake of excellent volume and texture. Batter 5D, on the other hand, was far too frothy and yielded a cake which rose too rapidly during baking and subsequently "fell" upon cooling.

Familiarization with general microscopy and examination of the accompanying illustrations will suggest many variations of the above applications. For example, with a little practice, quantitative approximations of the relative proportions of various constituents can be estimated with a reasonable degree of accuracy. From the average crystal size and the total volume of sample, the ratio of solid to liquid can be judged (3). By simple calculation of the volumes of the air bubbles in a given volume of cake batter, the percentage of incorporated air can be determined.

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Automation–Instrumental Analysis

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TODAY with the ever increasing demand on industry to provide more product, better product of more uniform quality—the role of instrumentation, control, and now automation takes on a new significance. Automation has various meanings for various people. This paper will be concerned with automation as the development of new types of sensing elements that have emerged from theory into practical laboratory instruments and then finally into the plant for instantaneous control.

Lewis Malter

The modern system of instrumentation has produced improvements in plant process control in many industries. The need is apparent for a general review of some of the present laboratory instruments and methods to determine if further savings of money, time, and manpower can be accomplished in other industries. Instrumentation has outstripped the conventional measurements of temperature, pressure, flow, etc., and is being extended to measure other important process variables used in the petroleum, chemical, and food industries: electrochemical, color and turbidity, and chemical composition.

It has become necessary for the plant instrument engineers to transfer the useful analytical instruments and methods from the laboratory and make them work under the more difficult plant conditions. One of the early analytical tools to be moved from the laboratory to the process plant was that for the measurement of pH. During the early development of pH instrumentation very delicate electrometers were used, and for industrial applications these were out of the question. Attempts were then made to use galvanometers with large thin-walled electrodes, but the extreme fragility of the electrodes and the unstable galvanometers again would not stand up in the plant. Later with the development of stable low current amplifiers the standard glass electrode was made practical for industrial application.

Applications of pH control can be found in practically every industrial process where aqueous solutions are involved. pH will affect the quality of the product as well as the speed and yield of reactions. Emulsification, electrolysis, neutralization, biochemical processing, bleaching and dyeing, coagulation and precipitation, and hydrolysis all require some degree of pH control. The most obvious applications, of course, are water-conditioning and waste and sewage treatment. The latter problem is becoming more important as state waste requirements become more demanding.

TODAY, more than ever, we are becoming color-conscious in our every-day living. Products are often classified and priced according to either arbitrary or fixed color standards. Oils, resins, pigments, and dyes have to be selected with the utmost care to maintain uniform quality and color. The quality and price of edible oils often can be determined by color. Instruments have been applied as a means of standardizing color tolerances in the laboratory and in the plant.

Because of the great strides after the development of photoelectric cells, designs of direct reading and now automatically operated spectrophotometers have been developed, and product laboratory methods of color standardization have taken hold. These instruments are now the fundamental basis of colorimetric analysis, standardization, and specifications. A routine set of specifications usually controlled by the laboratory can be placed on the plant stream to provide continuous data for quality control.

The flow colorimeter offers a means of indicating or controlling process streams where intermittent data do not supply adequate information for watching the process where sudden changes in concentration or contamination can be damaging. In the case of chlorine dioxide where the material is dangerous and difficult to handle, the rugged stream colorimeter is used effectively. The flow colorimeter consists basically of the same elements which make up a laboratory colorimeter, and the principles are identical. A light source in the colorimeter passes through the flowing sample where some of the radiant energy is absorbed and the remaining energy is detected by a phototube. The phototube then sends its signal to an amplifier, then to a meter, and next possibly to a recorder-controller. This indication gives a relative reading of concentration to some reference fluid which has previously been set for full-scale deflection. Unlike the laboratory where extreme care is taken to make some standardization, checks are made frequently, and equipment is kept clean, the flow colorimeter has to live with the abuse of the plant process. Maintenance and frequent cleaning should be made on such installations. Variables such as temperature, pressure concentration, flow rate, and flow transients can usually be compensated or engineered out of the process.

For example, such an instrument can be used to determine chlorophyll in vegetable oils. Color filters can increase the sensitivity by selecting the most sensitive portion of the spectrum. Perhaps where a filtrate or precipitate has to be carefully maintained for maximum yields, a flow colorimeter measuring the turbidity of the filtrate will continue to recirculate the filtrate until a cake is built up on the filter screen. When the colorimeter senses a preset turbidity concentration, it will open the stream to the process and will now act as a "monitor" for possible breaks in the filter screen. Applications can also be made in other than liquid streams. Successful measurements have been made on gas streams of chlorine and chlorine dioxide.

It was not too long ago that infrared analysis changed from a scientific curiosity to a practical process control variable. It took World War II to provide the stimulus for further investigation of infrared spectrophotometry for the control of butadiene in synthetic rubber production and the control of aviation gasoline.

In the early instruments the accumulation of data was slow and laborious. Sensitive galvanometers were relied upon to indicate exceedingly small signals. Because of the extreme problem of temperature variations and vibration the instruments were usually mounted in the basements on huge piers, and night operation was necessary for minimum vibration. With the advent of low thermal mass thermocouples and bolometers, chopped beam systems came into existence and faster scanning techniques became feasible. In order to build infrared instruments commercially, large quantities of infrared transparent material for prisms, windows, and lenses had to be produced. Synthetically grown salt crystals soon filled this need.

Meanwhile manufacturers of electronic tubes developed subminiature tubes of low noise characteristics and filled the gap necessary in the attendant circuity. Now with transistors becoming available, the electronic portions can be cut down in the sheer mass of the instrument and performance improved.

An infrared spectrophotometer is used to investigate almost any organic molecule regardless of the size, composition, or weight. Very simply, the principle of infrared analysis involves the absorption of radiation by virtue of molecular rotation and vibration. Any small molecular group attached to an organic molecule will exhibit a particular vibration of the remainder of the molecule. Research on the structure of molecules can quickly give a quantitative analysis. Infrared can also be used as a quantitative analytical tool based on the assumption that the absorption will follow the Lambert-Beer Law.

The extreme usefulness of specific qualitative and quantitative analyses by infrared spectroscopy persuade the instrument engineer to place a unit on the process stream. Because even the most rugged laboratory instruments will not stand up under plant conditions, a simplified version using a non-dispersive technique is being used successfully. This principle actually has been used as far back as 1926 for a self-filter type of instrument. But in the last 10 or 12 years the instrument has increased in usefulness many-fold to the point where about a half-dozen gas analyzers are being manufactured commercially. By far the majority of the infrared stream analyzers have been used for gas streams.

HOWEVER just as important is the need for good control of liquid streams. Dow Chemical in 1951 developed a dispersive instrument that could be fitted into a 11 x 13 x 7 explosion-proof housing. They have been quite successful in applying this unit to many applications in the plant. Although there are seemingly difficult problems in sampling, various systems have been devised to supply a representative sample without undue lags. This same instrument can be used for gas samples as well.

The mass spectrometer is another classic example of the transformation of an instrument for laboratory use into one of the most useful process controls. In practice the spectrometer ionizes particles and fires them across a magnetic field where they are bent into a circular path. The heavier masses make a wider arc than the lighter ones and hence go to different targets where they are measured. This arc diffusion can be compared to the dispersion of light through a prism.

In practice there are several different methods of dispersing the various molecules—180 degree magnetic, 60 degree segment, radio-frequency and ion-resonance.

Again the need for more exacting means of measurement during World War II in development of atomic research prompted the phenomenal growth of mass spectrometry. As recently as 1940 there were less than a dozen instruments throughout the country. Now there are hundreds.

The analytical mass spectrometer has been used extensively for both liquid and gas samples, usually analyzing mass from 2 to 150, although it can be operated on masses somewhere over 400. This type of instrumentation is extremely useful in the petroleum field, where compounds have properties that are so nearly similar that they are quite difficult to identify by ordinary chemical means. The outstanding features of this means of measurement is the speed by which samples can be measured without previous fractionation or concentration.

Because of the high resolution and detectability of the instrument it is used for measuring trace materials in process streams as well as the key component or components in the process stream.

In order to reach the ultimate goal of practical automation for the chemical plant, more and better analytical measurements will have to be made continuously on the plant streams. The preceding paper tells of some of the useful laboratory instruments and how they are being successfully used in plant operation today.

Statistics Applied to Research and Control in the Oil and Fat Industry

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FOR MANY YEARS the subject of statistics was considered to be associated chiefly with the collection of large masses of data and the presentation of such data in tables, charts, or graphs. Today that conception is extremely outmoded, and those more or less routine operations are now only an incidental part of the function of statistics in research and industry.

H. P. Andrews

Prof. George W. Snedecor, of Iowa State College, considers statistics to be an integral part of the scientific method. "Some people," he says, "seem to think that it is only an aid to science, a luxury to be indulged in by only the more leisurely scientists. At the other extremes there are those who attribute to statistics a kind of magic to elicit reliable information from shoddy and inadequate experimental data."

As a vital and integral part of the scientific method, statistics provides special tools and techniques for the research workers who pursue scientific investigations. It is concerned with two main features of the scientific method: the performance of experiments and the drawing of objective conclusions from the experimental observations. Accordingly the science of statistics may be thought of as being divided into the two corresponding areas: the design of experiments and the analysis of data leading to statistical inference.

In practically every field of inquiry the attainment of new knowledge and the development of specialized technology has been chiefly by the process of inductive reasoning: the process of arguing from observational facts to the theories which might explain them. In this process it is necessary to draw inferences from the results of testing or experimentation, to argue from consequences to causes, from the particular to the general; in statistical terms, to argue from a sample to the population from which it was drawn.

This process is logically hazardous, and the conclusions must always be considered to have a degree of uncertainty. All of us—laymen, specialists, administrators, or research workers are constantly sampling, estimating, and drawing conclusions, often without even being conscious of it and frequently with little or no actual knowledge of the hazards involved.

In the last few years public opinion surveys have come into great popularity, and although they are admittedly fraught with considerable possible error, they have been invaluable in many economic and business decisions. Often in research a dozen experimental animals may suffice to disclose useful information concerning a population of millions; a carload of oil may be accepted or rejected upon evidence gained from testing a sample of only a few pounds; the physician makes inferences about a patient's blood from the examination of only a single drop. Yet anyone who has ever taken samples, run analyses, or conducted experiments knows that the results from any one sample, analysis, or experiment may be far from the true values for the aggregate of material or population.

The conclusions drawn by these procedures are not altogether infallible and are attended always with a degree of uncertainty. The process of statistical inference provides a quantitative evaluation of this uncertainty. It utilizes the theories of mathematical probability and enables the scientist to assess the reliability of his conclusions in terms of probability statements.

ONE of the simplest yet most fundamental problems to which the statistical approach may effectively be applied concerns the answer to such questions as: how accurate is my observation, what is the precision