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Process Control

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PROCESS CONTROL has increased in importance in recent years and will doubtless increase even further. Proper process control is becoming more important because of a number of developments, including a) the trend from batch to continuous manufacturing operations, b) the increasing rates of production output, and c) the increase in complexity of numerous processes.



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Many benefits can be obtained from the wise selection, adoption, and administration of a good process control system. These benefits include cost control, efficient and relatively trouble-free operations in the factory or pilot plant, and control of the quality of finished product. All of these factors are interrelated, all are of major importance.

Cost Control

Successful operation of a process or manufacturing procedure requires rigid cost control. This success is often dependent upon reliable analytical techniques or instrumental analyses applied to samples which are carefully selected at strategic steps in the manufacturing operation. Some of the cost factors requiring control are as follows.

Raw Materials. The process should be developed to use commercially available raw materials of a suitable grade, concentration, and purity which will ensure the most efficient operation of the process at minimum cost. Each delivery of raw materials subsequently received must be sampled by a method which will ensure that the sample is as uniform and as representative of the entire delivery as possible. This sample must be subjected to sufficient laboratory tests to make certain that it is the specified material, of the

desired concentration and purity, and of the proper quality to perform satisfactorily. For example, a triglyceride to be used in preparing an edible product should be tested for moisture and impurities, free fatty acid content, color and bleachability, iodine value, titer, and its effect on the stability and flavor of the finished product.

By-Products and Losses. Analytical and instrumental control methods can materially affect cost control by minimizing losses and the formation of by-products. For example, in the process for the hydrolysis of triglycerides to fatty acids (4) and the subsequent distillation of the fatty acids, the yield of distilled fatty acids and the amount of still-bottoms is adversely affected by the presence of a high percentage of unsaponifiable material in the feed stock and by the presence of any materials which will react with the crude fatty acids during the distillation operation.

Concentration and Purity of Finished Product. Cost control requires proper control of the finished product concentration and purity. Sufficient samples must be analyzed during the processing operation to control the finished product within specified limits.

Efficient and Trouble-Free Operations

Good process control methods and procedures can do much to increase the efficiency of a plant process and contribute toward the goal of trouble-free operations. All known techniques should be employed to help the manufacturing operation make it right the first time. Numerous examples can be cited. For example, in the hydrogenation of fatty materials, the selection and grading of the catalyst can have a major effect on the reaction time during hydrogenation, and the quality of the finished product. The pre-testing and analysis of the catalyst before approval for plant use is vitally important. Likewise the selection and purification of the fatty materials to be hydrogenated has a direct effect not only on the properties of the hydrogenated material but also on the effective life of the catalyst. Extremely careful and precise analysis of the fatty raw materials is an important factor in this plant operation.

Control of the Quality of Finished Product

Most of the analytical techniques previously described in this short course are necessary to control the quality of the finished product. Papers presented in previous short courses also described these controls. The analysis of soaps was described by Stillman (1) in the 1952 Series. The analysis of Syndets was described by Nevison (2). A. E. King, in his paper (3) on Performance Desiderata, describes the desirable qualities of soaps.

Aids in Obtaining Process Control

Process control is obtained not by one but by many means. Each of the following is an important factor in this control: the chemical control laboratory; in-plant testing and instrumentation in plant operations; quality control inspections; and process investigation work and process audits.

The Chemical Control Laboratory. The chemical control laboratory is a basic tool for process control. An efficient laboratory should have the following: adequate facilities and personnel; methods standardized, accurate, and rapid; sampling representative of entire lot; specifications and control limits defined accurately; and results reported promptly.

The chemical laboratory is the company's means of controlling the quality of raw materials purchased, the quality of the products during the making process, and the quality of finished products. Analyses of raw materials serve not only as a check on the supplier but are used by the manufacturing department for grading and storage operations and to determine the amount to use in formulation. On some materials, analyses are necessary before unloading is permitted. On others unloading may proceed, but the analyses are necessary before calculation of final payment is made. The general purpose of the Chemical Control Laboratory then is to serve as a quality and process control tool. This is done by analyzing representative samples accurately and supplying the information to the proper persons in a form and at the proper time to be of greatest value to them.

The speed required for completion and reporting of analyses is dependent upon the nature of the process and the extent of storage facilities available in the manufacturing operation. For example, if a large inventory of raw material is maintained and adequate provisions are available for segregating new deliveries until laboratory analyses are fully completed, laboratory results need not be rushed. However if small inventories are maintained and the incoming delivery is to be blended in storage with present supplies, rush analyses are necessary before unloading this delivery.

The same principle applies to process control and finished product samples. If batch operation is employed in which each batch can be completed, sampled, and analyzed prior to packing, laboratory results are not as urgent as for continuous flow operations. Continuous flow operations require adequate sampling at strategic points in the process and prompt and accurate analyses together with prompt reporting of these results to the manufacturing department.

Laboratory facilities should be adequate to supply information promptly to the supervisor of the process which will enable him to make wise decisions on the adjustment of process variables. Many of the ana-

lytical techniques previously mentioned in this short course, as well as numerous other methods, will be required in a laboratory serving many operations. In a single operation of a relatively uncomplicated nature, only meager facilities may be required. However the basic requirements are the same. The laboratory methods employed should generally be the ones universally accepted in the industry, both by the suppliers of the raw materials and by the purchasers of the finished goods. For example, wherever applicable the approved methods of the American Oil Chemists' Society should be used, or the methods of the American Society for Testing Materials, or the Association of Official Agricultural Chemists.

Another basic principle which should be observed is that each testing operation should be of basic value. Each test should be scrutinized and thoroughly studied to determine its value.

Adequate, properly trained personnel and adequate facilities and equipment should be available for conducting these tests, with provisions for checking the accuracy and precision of the methods and equipment. Provisions should be made for increased frequency of testing in case of processing difficulty in the plant. Good process control may require resampling at numerous points in the manufacturing operation to determine accurately the prevailing quality condition.

There has been a trend in recent years to determine and describe laboratory results more precisely. For example, the general terms of acidic, neutral, and alkaline are now expressed in specific terms of pH to the second decimal place. Likewise noteworthy improvements have been made and are being further developed to describe color measurements more accurately. Other trends of this nature should be encouraged.

Control laboratory results should not only be reported numerically but, to be of maximum value in controlling the process, should be reported as indicating whether or not they are within specifications and how near they come to falling outside the specification limits. These results should be assembled, organized, and reported in a manner which will clearly indicate to production personnel any trends which might lead to off-specification materials. Likewise they should give immediate warning to manufacturing personnel of any substandard results.

In-Plant Testing and Instrumentation in Plant Operations. Prompt testing results are required in numerous processing operations. Continuous flow operations in general require this type of control. In-plant testing is frequently adopted to meet this requirement. These tests are performed either by laboratory or by specially trained operating personnel.

Instrumentation is not only of major importance when applied to laboratory samples but also is becoming of increasing importance in the manufacturing departments in controlling the processes. Instrumentation can be adopted to do the following and many other functions: sampling, temperature controlling, pH controlling, proportioning and flow control, dilution control, gravity control, moisture control, weight control, and outage control. These and other controllers are a major factor in process control.

Process Inspection and Quality Control. Increasing emphasis is being placed on the importance of quality control inspection during processing and finishing operations by skilled, impartial observers who generally

are not members of the manufacturing departments. The basic purpose of these quality control programs is to aid the production department to manufacture a more uniform product within the desired limits of variability. Quality control today exists in many forms ranging from crude to highly complicated. However they should basically be regarded as tools to help improve process and product control. The complexity of the control program should depend on the complexity of the process being operated. The program should be designed basically to ensure producing a product which will have buyer acceptance, packed in suitable containers, having the desired functional properties and shelf life. The quality control inspection data should be correlated with laboratory results to be of maximum value in obtaining this quality assurance.

Process Investigation and Audits. Another tool some companies use in controlling quality is the use of process investigators or "trouble-shooting" personnel who are available to work on production problems which have no obvious or immediate solution. These technically trained men study the process, go into the plant to observe the operations, and enlist all available help in overcoming the difficulty or in improving the manufacturing procedure. The solution of existing problems as well as modernization plans for the future can result from these investigations.

Application of Process Control Techniques

The practical application of the general principles just discussed can be illustrated by describing a processing operation. The hydrolysis of triglycerides with water (4) under controlled conditions of temperature and pressure is a practical example.

The raw material fats and oils purchased for use in this process are sampled and analyzed by standard

procedures described in the Official Methods of the American Oil Chemists' Society. Determination of impurities, moisture, color, bleachability, iodine value, titer, and other laboratory tests are performed.

The oils are pumped together with water to the plant hydrolysis unit through proportioning devices. Instrumental control devices maintain the desired ratio of raw materials and also the specified temperature and pressure conditions in the hydrolysis unit.

The products emerging from the reaction zone are cooled, allowed to settle, and are then separated into storage tanks for the resulting crude fatty acids and dilute glycerine. Laboratory analyses of both materials are obtained to determine the yield of free fatty acids and glycerine produced and to determine the amount and chemical composition of the impurities present.

The crude fatty acids then are distilled totally or fractionally if they are to be used in making an edible product.

The process for making the edible product is controlled instrumentally at the specification temperature and pressure. Control samples are withdrawn at specified intervals and tested in the plant for free fatty acid content to determine the progress of the reaction. The finished product is analyzed in the laboratory for color, odor, iodine value, melting point, stability, percentage of purity, and flavor evaluation. When approved by the control laboratory, the finished product is filled into containers which also have been approved by the laboratory. Quality control personnel check the filling operation, using standardization inspection techniques.

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Establishment of Specifications

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SPECIFICATIONS may be defined as the formulated, definite, and complete written statements which describe the properties or performance of a product. They may also describe what the buyer requires of the seller. Through the medium of specifications the producer and consumer are enabled to speak a common language.

For a specification to be truly effective it should give in clear and concise terms detailed information concerning the type, composition, grade, and quality of the product to be manufactured or delivered. Specifications should be so worded as to preclude any misunderstanding on the part of those who must use them as to what is actually wanted.

A good specification should also contain definite statements relative to the necessary methods for in-

specting or testing the product delivered in order to determine whether or not it complies with the requirements set forth therein. It is important at the outset that a distinction be made between the establishment of specifications and their enforcement. This discussion will be limited, as the title implies, to the procedures commonly used in establishing a specification.

In setting up specifications, the essential factor is to define the elements of quality desired. These may or may not represent the ideals. In many cases, particularly in the purchase of raw materials or supplies, specifications already exist which can be adopted as, for example, the U.S.P., A.S.T.M., or the quality standards for vegetable oils defined in the N.C.P.A. or N.S.P.A. Trading Rules. Where these do not exist, one approach is to obtain the manufacturer's specifications, not the so-called typical analyses which may be well within limits on an average, but the averages of the plus and minus ranges on the important components. Obviously, if at all possible, a grade of raw material available commercially should be used to avoid the extra costs of a special tailor-made product.

The question of uniformity *versus* purity sometimes enters into consideration. A classic example would be the actual monoglyceride and diglyceride content of a so-called commercial monoglyceride. It is



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