

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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much more important that the percentage of monoglyceride and the ratio of mono- to diglyceride be controlled within reasonably close limits than it is that the maximum percentage of mono be the highest possible. A specific formulation based on a regularly obtainable commercial blend could fail miserably if a pure monoglyceride were used and *vice versa*.

Specifications may originate from one of two sources: a) they may be set up by the manufacturer himself for the purpose of producing a product which will meet the usually accepted standards required by the trade; and b) they may be established by a buyer who requires a product which deviates in some manner or degree from the products usually offered for sale on the general market.

Subspecifications may also be established by the manufacturer to control or govern the production of a product from the purchase of the raw material to the completion of the manufacturing operation.

As a case in point let us consider the manufacture of a shortening from crude vegetable oils. Let us assume further that the product is to be a blend of cottonseed and soybean oils, the production of which is to be dictated by economics and performance characteristics. The properties of a high quality shortening are well known and need not be repeated in detail. Crapple (1) has ably described these factors, and anyone interested may refer to his original article.

There are however a series of steps which must be followed in general in setting up specifications for the manufacture of a shortening. These include specifications governing the purchase of the raw materials and those designed to control the various manufacturing steps. An attempt to outline these is given below.

Purchase of the Crude Oils. Most manufacturers of shortening are equipped to refine crude oils as they are obtained from the oil mills. Soybean oil purchases are almost invariably made under the trading rules of the National Soybean Processors' Association. One specific rule defines the grade and quality of the crude oil and spells out in detail the permissible deviations under which oil may be delivered on a standard purchase contract. It further outlines the analytical procedures which are to be used and the premiums and discounts which are applicable for quality standards above or below an accepted median.

The following definition of standard quality is an excerpt from the rules of the National Soybean Processors' Association (2).

The standard of quality shall be the designated type of pure soybean oil of fair average quality based on the season's production which must conform to standard specifications of the Association, which are herewith made a part of the Trading Rules and which are subject to modification from time to time as conditions may warrant, upon recommendation of the Technical Committee.

A. Types of Crude Soybean Oil. Edible crude soybean oil shall be of any of the following designated types produced from domestic-grown, undamaged, mature yellow soybeans:

1. Expeller-pressed
2. Expeller-pressed degummed¹
3. Hydraulic-pressed
4. Hydraulic-pressed degummed¹

5. Solvent-extracted (state solvent used)
6. Solvent-extracted degummed (state solvent used)
7. Mixtures of any of the above types, in which case seller shall specify that analysis shall be made corresponding to one of the specific types. (state solvent used)

Crude cottonseed oil is purchased under a somewhat similar set of trading rules established for the purpose by the National Cottonseed Processors' Association. These rules also are reviewed annually, and the necessary changes are made to adapt them to changing conditions.

The following quality standards are taken from the Rules of the National Cottonseed Processors' Association (3).

Definition of Quality. Crude cottonseed oil means the oil as produced from cottonseed only, by either the hydraulic, expeller, or screw press, prepress solvent, or solvent extraction process.

The Cottonseed Trading Rules take cognizance of several quality grades of crude cottonseed oil. These are Prime Crude, Basis Prime Crude, Off-Crude, Reddish Off-Crude, and Low Grade Crude. Separate rules provide the price adjustments for the various quality variations.

An important point to observe in this connection is that these specifications are the result of a trial and error procedure over a long period of years and were not necessarily developed by the use of scientific or mathematical techniques.

The N.C.P.A. rules were first adopted in 1897 and have been steadily amended ever since. The N.S.P.A. rules were adopted in 1933 and likewise have been amended regularly over the years. Both sets of rules or specifications are the result of many years of study and evaluation to develop rules which impartially protect the interest of buyer and seller alike. They are so carefully worded that the possibility of misunderstanding is virtually eliminated. Both buyer and seller know exactly what is required insofar as quality of product is concerned.

Thus we see that there are in existence well defined standards which have stood the test of time and which may be used to purchase the necessary raw materials.

Refining. After the oil has been purchased under these rules, the next step in the production of a shortening is the refining of the oil. While crude cottonseed or soy oils may be refined by one of several methods, the most popular and commonly used are those employing alkaline refining agents, either caustic soda, soda ash, or a combination of both.

Regardless of the method of refining used, the refined oil must meet certain quality standards if it is to function properly in the subsequent processing steps. A specification which has been found satisfactory is the following:

FFA .05%
Soap 10 P.P.M.
M&V .05%

A well refined oil also is clear and brilliant in appearance and entirely free from visible moisture and settlings. The impurities commonly present in crude oils must be completely removed and, in addition, the oil must be low in free fatty acid content

¹Tentative Definition. Degummed soybean oil shall be the product resulting from removal of phosphatides from crude soybean oil and shall contain not more than 0.03% phosphorus determined by A.O.C.S. Tentative Method Ca 12-53.

and moisture and volatile matter. The soap which is formed as an adjunct to the refining process must also be substantially removed. Failure to observe these operating precautions may result in the production of an oil which might bleach poorly, possess uncertain color stability, poison the catalyst or at least interfere with the desired selectivity of hydrogenation, and finally deodorize with greater difficulty than may be desirable.

However there may be times when it is desirable or necessary to purchase refined oil on the outside market. When this is done, it is again necessary to rely upon the established standards of N.S.P.A. and N.C.P.A. For purposes of comparison the following trade standards are quoted:

“TENTATIVE REFINED SOYBEAN OIL
SPECIFICATIONS (2)

Refined Soybean Oil shall meet the following specifications:

1. Clear and brilliant in appearance at 70-85°F.
2. Free from settlings
3. Shall contain not more than 0.10% moisture and volatile matter, using A.O.C.S. Official Method Ca 2d-25
4. Free Fatty Acids shall not be in excess of 0.10%
5. Color when bleached, according to A.O.C.S. Method Ce 8b-52 Procedure (a), shall not be darker than 3.5 Red and shall not have a predominantly green color.”

Likewise Refined Cottonseed Oil is usually sold under the Prime Summer Yellow Cottonseed Oil specifications of the National Cottonseed Products Association (3). These rules are as follows:

“Rule 156. *Prime Summer Yellow Cottonseed Oil.* Prime summer yellow cottonseed oil must be free from visible foreign material, clear at temperatures sufficient to melt the stearine, sweet in flavor and odor, of a color no higher than A.O.C.S. 7.6, and shall contain not more than .25% free fatty acid nor in excess of .10% moisture and volatile matter.”

As will be noted, these specifications are less stringent than those previously quoted, and oils purchased under them may require additional processing to bring them to the standard of quality desired for first-class commercial shortenings. After the oils have been suitably refined, they may or may not require bleaching, depending on the quality of the oil and the end-product specifications to be met.

High quality vegetable shortenings usually have a color in the range of 1.0 to 1.5 R on the Lovibond scale. Whether or not the oils require bleaching prior to hydrogenation in order to attain the final color desired depends entirely upon the initial color of the oil, its bleachability characteristics, and its freedom from impurities.

The best index of quality is usually the bleachability of the oil. Generally a good grade of bleachable oil, which is free from impurities and low in soap content, will not require preliminary bleaching as many of the naturally occurring pigments will be decolorized in the hydrogenation process. Usually, however, if impurities are present and it becomes necessary to bleach the product, it is generally found necessary to bleach to a color below 2.0 R Lovibond.

Hydrogenation. In the hydrogenation process a number of reactions occur simultaneously. Allen and Kiess (4), Bailey (5), and others have shown that considerable isomerization occurs during the hydrogenation reaction. Therefore the conditions of hy-

drogenation must be controlled to bring about the desired result, that is, operating conditions must be selected which favor the formation of the maximum quantities of desirable end-products and minimize the undesirable.

The characteristic most directly affected is the iodine number, which decreases in direct proportion to the amount of hydrogen absorbed.

There are many factors which affect the progress of a hydrogenation reaction. The wide variation in converter design alone would preclude the possibility of stating a generalized set of hydrogenation conditions which would fit every installation. Other variables besides the oil which can influence the course of hydrogenations, are purity of hydrogen, type and concentration of catalyst, temperature, pressure, and age of catalyst.

The hydrogenation end-points are usually controlled by iodine value determinations although some authorities prefer the refractive index for reasons of convenience.

However the iodine number alone is seldom a sufficiently accurate index upon which to predicate hydrogenation end-points. The iodine value measures the average degree of unsaturation of the fatty acid chain and nothing more. It takes no account of the selectivity or non-selectivity of the reactions, the degree of isomerization, the relative distribution of saturated and unsaturated fatty acids, etc. To supplement the information afforded by the iodine number, it is usually necessary to determine the melting point and the congealing point (set point) or its equivalent. Dilatometry is not rapid enough for control purposes.

The melting point used may be determined by the traditional capillary tube method or by the Wiley method.

The “set” or congealing point may be determined by procedures which have not been too well standardized. Other methods have been suggested and used as alternatives to shorten the time required or to eliminate as far as possible the “human” factor.

Admittedly if such factors as the purity of the oil, composition of the blend, activity and selectivity of the catalyst, purity of the hydrogen gas, etc., could be rigidly controlled, the iodine value alone would provide sufficient information, assuming of course that the other operating factors are also controlled.

However rarely is it possible to control all factors and therefore, in establishing end-point specifications for hydrogenation, it is necessary to determine two and possibly three independent variables, *i.e.*, the iodine value, congeal point, and melting point.

The reproducibility of each determination should first be determined. Thus if the iodine number is found by statistical methods to be reproducible within $\pm .25$ unit at the range required for this product, consideration must next be given to the processing variation which may be allowed. Obviously, it would be futile to require a plant operator to control a hydrogenation to $\pm .25$ unit since the accuracy of the method itself varies that much and no leeway is permitted for variables in the operation itself. The end-product specifications should be scrutinized at this point to determine how much variability may be acceptable in the final product before it becomes unsuitable for the purpose for which it was produced.

The degree to which a product has been hydrogenated is reflected in the texture and the stability of the product. If the reaction has been carried too far, the texture is likely to be hard and brittle; if not far enough, it may be too soft and, in addition, the residual unsaturated acid content may be high enough so that the stability may be adversely affected.

The stability may be measured by well known accelerated methods. The texture measurement presents more of a problem. There is a difference of opinion in regard to how best to measure the texture of a finished product. Some authorities prefer a micro penetration method, others the macro penetration, while still others depend upon inspection of the product and actual performance tests at the temperature ranges within which a product may be expected to perform. One of these methods must be selected and conclusions based on the results obtained.

The statistical approach provides the most satisfactory tool in determining which variables are significant and which are not, as well as how much variation may be permitted. Thus it has been found that in the case we have selected I. V. may vary ± 1.0 units, congealing point $\pm .75^\circ\text{C}$. and melting point $\pm 1.0^\circ\text{C}$. without significantly affecting performance characteristics. Therefore as much latitude as practicable should be allowed for operating variables.

Other characteristics of importance are color and flavor. Color has already been discussed under bleaching, and if the oil has been properly processed and was of sufficient good quality to begin with, little difficulty is experienced with this factor. If the color of a high quality oil deteriorates subsequent to hydrogenation, the difficulty may almost invariably be traced to accidental mishandling, such as overheating in storage or to deodorizer malfunction. This latter is generally accompanied by other quality degradation manifestations.

Flavor is much more difficult to define objectively. It would be beyond the scope of this discussion to attempt to describe flavor panel techniques. They have been described elsewhere, and anyone interested may consult the original literature (6, 7). While analytical means have been suggested for evaluating flavor, up to the present time the taste panel is the only reliable method which has been developed. Arbitrary values for flavor scoring are of course meaningless in themselves unless the panel members have demonstrated their discrimination, reliability, and ability to reproduce their own results. Generally a capable panel will grade a satisfactory shortening 7 or better. When a shortening is graded 8 by a critical panel, it generally may be considered to be in the high quality category.

Other properties which are of importance are the baking characteristics. These must be sufficiently good to satisfy the commercial baker or the product would not be deemed acceptable.

Following are given typical analyses of several commercial brands of high grade hydrogenated vegetable shortenings.

A study of the table indicates a rather wide variation in analytical characteristics. Thus the iodine value varies quite widely, reflecting different methods of hydrogenation. The same considerations prevail in regard to the congeal and Wiley melting points. However it will be noted that from a performance standpoint the products are, with two notable exceptions, almost all uniformly good. The texture and plastic ranges, as measured by penetrations at 70° and 98°F ., are remarkably close together. Samples 1, 5, and 8, which are appreciably firmer in texture as evidenced by the penetration values, were produced by southern companies and undoubtedly were purposely so manufactured to compensate for the higher temperatures prevailing in that part of the country.

The creaming volumes and pound cake volumes with the exception of samples 1 and 9, indicate that the products should give satisfactory performance in the bake shop. It will be noted in the case of samples 1 and 9 that while the creaming volume is satisfactory, the pound cake volume is significantly below the average. It would behoove the manufacturers of these products to review operating practices and adopt the necessary remedial measures to bring the products back in line.

Unfortunately, the properties of a shortening cannot be described fully in terms of melting point, congealing point, iodine number, or any other group of physical constants. For these terms to have meaning, the composition and method of manufacture of the shortening must be known. For this reason it is more realistic to base primary specifications on texture, workability and plastic range, as measured by performance tests, penetrations, etc., rather than on the physical constants.

It is apparent from these data that different manufacturers use varying methods to arrive at essentially the same end-result. It is quite possible that methods of control other than those outlined here may have been used. In any event however each manufacturer must of necessity have correlated his operating conditions with the actual performance of his product, making the necessary adjustments until it possessed characteristics necessary to meet trade requirements.

Another type of specification which should be discussed at this time are those written by users to control product manufactured for a specific purpose.

TABLE I
Analyses of Typical Commercial Hydrogenated Vegetable Shortenings

Product	FFA%	Lov. color		I.V.	Flavor grade	Congeal point $^\circ\text{C}$.	Wiley M.P. $^\circ\text{C}$.	Pene. 0.1 mm.		Stab. AOM hrs.	Volume creaming cc./100	Lb. cake cc./100 gm.
		Y	R					70°	98°			
1	.04	15	1.5	69.6	8.0	35.5	47.3	145	199 ^a	100	177	246 ^b
2	.03	9	1.2	70.0	7.0	34.9	49.5	158	259	100	180	262
3	.03	8	0.9	72.8	7.5	36.0	50.0	169	230	100	180	287
4	.03	10	1.2	72.4	8.0	34.4	46.1	155	263	174	190	272
5	.03	5	0.6	75.1	8.0	31.7	44.7	168	286	110	190	278
6	.15	6	0.5	74.0	8.0	36.7	48.3	137	215 ^a	190	190	274
7	.02	6	0.8	73.3	8.0	34.4	46.4	151	263	100	185	260
8	.04	15	1.2	72.8	7.5	33.5	45.5	161	250	100	192	291
9	.05	9	0.9	72.5	8.0	36.1	47.7	135	218 ^a	78	180	273
10	.03	8	1.2	72.8	7.5	32.4	45.8	169	287	90	180	254 ^b

^a Product of southern manufacturers.

^b Low Baking Volumes.

Good examples of this are Federal Standard Stock Catalog Specifications EES-321 and JJJ-O-00361 and amendments thereto.

These specifications were promulgated by the Federal Specifications Board and cover Shortening and Salad Oils, respectively. Both are well written in that they define precisely the various types of products encompassed, the raw materials which may go into the formulations, the analytical requirements and the procedures to be used and, finally, the performance characteristics to be expected. The packaging methods and materials are also defined.

Another example of a government specification which is considerably more restrictive is Mil S 10170 A developed and used by the Chicago Quartermaster Depot. This specification fulfills the requirements of a good specification but is open to the objection of being restrictive in the sense that only peanut and cottonseed oils may be used. While there unquestionably were compelling reasons why this restriction was adopted, the fact remains that no cognizance is taken of the most excellent quality characteristics of soybean oil and processed meat fats. These latter products have found a well-established place in civilian uses, some of which are almost as exacting in regard to stability requirements as those of the military establishment.

The practical consequence of this of course is that fats which represent a preponderance of the available supply are barred from these outlets, and only products manufactured from relatively scarce and high priced oils may be used.

Other examples of well written specifications are those of several prominent manufacturers of bakery and home type pre-mixes. Below are given a typical specification for a hydrogenated vegetable shortening put out by a leading company in that field:

TABLE II

Moisture.....	0.10% max.
Stability.....	50 hrs. min.
FFA (as oleic).....	0.075% max.
Melting Points	
Wiley.....	118 ± 3°F.
Capillary.....	124 ± 3°F.
Congel Point.....	106 ± 3°F.

The spread between Wiley and Capillary melting points shall be 6 ± 2°F. Product shall be free from lumps and large crystals and must be delivered at 74°F. ± 6°F.

Secondary Specifications

Methoxyl Content.....	0.005% max.
Insoluble Impurities.....	0.005% max.
Iron.....	5 p.p.m. max.
Color.....	20 Y 2.0 R
Odor.....	Practically none
Flavor.....	Bland

Product must be plasticized and equivalent to previous samples submitted and approved with respect to performance.

The specification further defines the packaging and marking requirements. Also the analytical procedures to be used are carefully outlined.

The specifications for a hydrogenated vegetable oil from another prominent firm in the same field are given below:

TABLE III

FFA	0.15% max.
Capillary M.P.....	110-114°F.
Iodine Number.....	66-72
A.O.M.....	100 hrs. min.
Penetration @ 72°F.....	105-130

Product should not contain more than 30% soybean oil.

Attention is called to the variations between the two products.

Other examples could be cited in which specifications for hydrogenated vegetable shortening vary from either of those given above.

While in many cases the unit quantities purchased by major pre-mix manufacturers are considerable and justify production of "tailor-made" products according to certain specifications, the fact remains that the problems of the shortening manufacturer are enormously complicated by the varying specifications proposed by different users. It would seem that some means of standardization of similar products in the pre-mix industry should be feasible. A similar situation exists in the baking industry where many large bakers have differing specifications for products designed to do essentially the same job.

This practice is bound to be costly in the long run to both producer and consumer, and it would be to the mutual benefit of both parties if some degree of standardization could be made effective. Perhaps the shortening industry itself should take the initiative by proposing a set of specifications for uniform products. The advice and counsel of the various interested industries should of course be sought in an undertaking of this kind. The successful culmination of such a program would result in manifold advantages to both buyer and seller, making possible the manufacture of fewer and better products at lower cost.

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