successful in certain instances in differentiating between meals of high and low nutritive value but has been less successful in differentiating between meals of intermediate nutritive value or in comparing meals produced by one process with those produced by another process. Similarly for soybean meal, solubility measurements have been successful in limited application.

The fact that the increased resistance to peptization of cottonseed proteins in each of the solvents occurs concomitantly with the impairment of the nutritive value of cottonseed meal suggests that chemical modifications of the proteins are induced because of the application of heat. The solubility studies may well serve as a clue to the type of chemical reactions that take place during the heating of cottonseed meals and might serve as a guide to developing improved processes even though, eventually, solubility may be superseded as means for correlating nutritive value with chemical properties.

The conclusion that the protein solubility data obtained with the three solvents are of equal value in grading of cottonseed meal for broilers is implicit in the results from the statistical computations. The absolute values of the coefficients of correlation are specific to these studies and might be slightly different for studies of other cottonseed meals.

Summary

The correlations between the growth response of chicks to the nine cottonseed meals fed as a protein supplement and the solvent powers of 0.02N NaOH, 6N HCl, and 0.5N NaCl for cottonseed meal proteins are almost identical. The correlations between the solvent power of 6N HCl, 0.02N NaOH, and 0.5N NaCl and the gossypol contents of the meals are not as good as the correlations between the solvent powers of these solutions and the growth response of chicks.

[Received August 26, 1957]

Certain Uses of the Analysis of Variance with Standard Product Specifications

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EVERYONE will agree that standard product specifications are a modern-day necessity. They ensure constant product quality and a consequent healthy brand growth. The need is even more obvious when the production facilities used are numerous and widely separated and utilize different sources of raw material in different equipment. In some cases the same customer will obtain production from two or even three factories in successive purchases. The usual objective of industry is to minimize the variations in product sufficiently that the changes in source go unnoticed by the consumer. These conditions make it difficult to provide those responsible for the manufacturing process with standard specifications that are capable of being met uniformly. The use of the statistical technique of the Analysis of Variance has provided a helpful answer to this problem, and some discussion of its use is warranted.

Standard specifications may relate to finished product characteristics by which the consumer will be directly influenced in his evaluation of the product, such as color, odor, shape, package outage. However these have little relationship to the real performance of the product. They may apply to characteristics that can be measured objectively as dimension or sudsing efficiency, or subjectively as flavor or odor. Once market research has determined that the characteristic is important to the consumer and once the acceptable level of the characteristic is defined, then production must conform to this level to meet acceptance. The inherent variability of the process must not dictate the quality of the product; instead the necessary level of quality dictates the permissible variation in the process conditions.

If the process imparts greater variability in finished-product quality than the standard specification permits, then the reason for this variability must be isolated and controlled. While it may be that the process itself is not under good enough control, the variability may be caused by raw material nonuniformity or the lack of effective training of operators. It is often found that the variability is more apparent than real, because of sampling difficulties or lack of precision in the analytical techniques used to define the quality level of the production.

In the determination of the relative importance of the numerous possible causes of excessive variability of finished-product quality in order to start the right corrective measures, the Analysis of Variance technique has been used successfully. Obviously if there is no difficulty in meeting the standard specification uniformly, no problem exists and the following is unnecessary.

Where excessive variability does appear to occur however, it is helpful to make a preliminary separation of the total variability into the fraction contributed by the analytical techniques, by the sampling techniques, and by the remainder which will all be considered as process variability. When the relative magnitude of the three classes has been determined, the area is apparent in which to work first to improve quality or its uniformity, and the search for the specific cause or causes of the lack of control can go forward.

There are two specific aspects of the above discussion which will be illustrated by a practical example: *Problem 1.* the determination of the relative magnitudes of the components of variation in a process (this will assist in making decisions as to where in the process suitable adjustments should be made in order to decrease the variability of the finished product.); *Problem 2.* the determination of an optimum

TABLE I

Unit	1		2		3		4		5		6	
Sample	1	2	1	2	1	2	1	2	1	2	1	2
Analyses												
1	2.55	2.68	2.80	2.45	2.60	2.96	2.80	2.26	2.83	2.83	2.87	3.27
2	2.71	2.58	2.80	2.83	2.83	3.03	2.64	2.64	2.93	2.89	2.56	3.02
Unit	7		8		9		10		11		12	
Sample	1	2	1	2	1	2	1	2	1	2	1	2
Analyses												
1	2.55	2.74	2.51	2.44	2.87	2.81	2.84	3.04	2.56	2.45	2.72	3.02
2	2.60	2.65	2.39	2.57	2.71	2.73	2.99	2.71	2.35	2.52	2.48	2.64

$\bar{X} = 2.71\%$.

sampling plan, which will yield a finished-product quality estimate of desired precision at a minimum cost.

Problem 1. The Determination and Use of the Components of Variance. The Factory Service Group in conjunction with the Products Research Group have determined a manufacturing specification for Total Organic Builder (T.O.B.) in one of our synthetic detergents. This specification reads "Target 2.7%, not permitted below 2.5%."

The factory determines the T.O.B. content of its production by means of a chemical analysis of samples from the production lines. From the results of such analyses, decisions are made concerning the adherence of production to standard specifications and hence the shippability of the product. The precision of these analytical results becomes an important aspect of such decisions. For example, if the T.O.B. were reported as 2.7% with a standard deviation of $\pm$ 1.0%, one would question the adherence of the process to specifications. However, if the standard deviation has been only $\pm$ 0.02%, there would be no question of possible specification deviation.

How can we measure the variation in a single T.O.B. estimate, and, further, can we attribute the major part of the variation in the estimate to any one aspect of the estimation process?

The first requirement is to determine the steps in the T.O.B. estimation procedure. There are three specific subdivisions in the sampling procedure: a) the process naturally falls into "t" distinct process units; b) from any process unit "k" samples are chosen; and c) on each of these "tk" samples, "n" chemical analyses are performed.

Thus there are three possible sources of variation in a T.O.B. estimate: the variation caused by differences from process unit to process unit; the variation present within each unit which represents nonhomogeneous mixing; and the variation resulting from differences between duplicate analytical results on a uniform sample.

The relative magnitudes of these sources of variation can be determined through the use of the "Analysis of Variance" and an experimental design called "A Nested Design." For example, the following sampling scheme was decided upon:

a) The day's production was divided into 24 units of 1-hr. duration. This division was effected because a natural change occurred about every hour in the process. It was decided to choose 12 of these 24 units at random ($(t = 12)$.
b) Two samples were selected at random from each of these units ($k = 2$).
c) Duplicate analyses were done on each sample ($n = 2$).

The T.O.B. analyses obtained by this sampling scheme are shown in Table I.

The calculation of the Analysis of Variance for the Nested Design can be found in any statistical text (1). Detailed calculations will not be shown here. The Analysis of Variance for this example is shown in Table II.

The total variance of a single T.O.B. analysis is estimated as:

$$V(Y) = V(a) + V(s) + V(p) \qquad (1)$$

where $V(Y)$ = total variation of a single observation
$V(a)$ = the variance due to analytical method
$V(s)$ = the variance due to sampling
$V(p)$ = the variance due to the process unit
$V(Y) = 0.0216 + 0.0074 + 0.0158 = 0.0448$

The standard deviation of a single estimate $= \sqrt{0.0448} = 0.2117$.

This indicates that a large amount of variability is present. For example, suppose the T.O.B. of a day's production was estimated by analyzing a single random sample and the T.O.B. result was 2.6%. The 95% confidence limits on the true T.O.B. measure would be:

$$2.6 \pm (1.96)(0.2117) \text{ or } (2.2 \longleftrightarrow 3.0)$$

This is a rather untenable position in which to place a manufacturer.

What is needed is a more precise estimate of the T.O.B. or a more lax specification. In general, it is not possible to relax specification limits since these are determined through consumer requirements. A more precise estimate is necessary. This can be obtained in two ways: a) by decreasing the total variance, σ^2; and b) by increasing the sample size and taking an average of all results.

Decreasing the Variance. Through the use of the

TABLE II

Source of variation	Degrees of freedom	Sum of squares	Mean square	Expected mean square
Between units	11	1.0937	0.0994	$\sigma_a^2+2\sigma_s^2+4\sigma_t^2$
Between samples within units	12	0.4355	0.0363	$\sigma_a^2+2\sigma_s^2$
Between analyses within units within samples	24	0.5181	0.0216	σ_a^2
Total	47	2.0473		

The calculation of the components of variance is done by equating the observed mean squares to their respective expected mean squares and solving simultaneously:[1]

$$S_a^2 = 0.0216$$

$$S_s^2 = \frac{0.0363 - 0.0216}{2} = \frac{0.0147}{2} = 0.0074$$

$$S_t^2 = \frac{0.0994 - 0.0363}{4} = \frac{0.0631}{4} = 0.0158$$

[1] The S_i^2 are used to denote the fact that these are estimates of variance components and not population parameters.

information gleaned from the formula for Total Variance the relative magnitudes of the components of variance provide clues concerning the process variability. In the example shown, the total variance of a single observation was 0.0448. This variance was composed of three parts: due to analytical method= 0.0216, due to sample=0.0074, and due to process= 0.0158.

Thus, if we desire to decrease the variance, the most likely places are, first, the analytical method, and secondly, the unit to unit variation in the process itself. For example, modifying the analytical technique might result in a more uniform duplication of T.O.B. results on the single sample of finished product.

Similarly the exercise of closer control from unit to unit would reduce V(p). The problems of reducing variation are usually operational in nature and require insight and careful experimental scrutiny.

Thus the Nested Design and Analysis of Variance are useful in that they sort out the total variation into component parts and provide clues as to the major source of variation. In the example above, the design additionally indicated that the material within each time increment was relatively homogeneous.

However, since more than one observation was taken in this investigation, a more precise estimate of the T.O.B. of the finished product is available.

In the example just shown, the mean T.O.B. result was 2.71% (Table I). The 95% confidence limits on this mean are obtained as follows:

Given: the variance of a single observation, formula (1)

Then the variance of the mean is:

$$V(\overline{Y}) = V(a)/tkn + V(s)/tk + V(p)/k \qquad (2)$$

$$V(\overline{Y}) = 0.0216/(12)(2)(2) + 0.0074/(12)(2) + 0.0158/12 = 0.002075$$

Thus the standard error of the mean is 0.0456. The 95% confidence limits for the true T.O.B. are (2.62 $\longleftrightarrow$ 2.80). There is little doubt that the T.O.B. of our product is well within specifications.

Problem 2. The Determination of an Optimum Sampling Plan. If one considers the cost of the sampling scheme, a less precise estimate of mean T.O.B. measurement might be adequate.

The optimum sampling scheme, which will minimize the cost of sampling and simultaneously yield a T.O.B. estimate of specified precision, is obtained through the following procedure:

a) Write down the sampling cost function. In the illustrated case above

$$C = tC_p + tkC_s + tknC_a \qquad (3)$$

where t = number of process units
C_p = cost of sampling a unit of the process
k = number of samples per unit
C_s = cost of taking a sample within a unit
n = number of analyses per sample per unit
C_a = cost of a single analysis

b) Minimize this cost, subject to the restriction that the variance of the mean be specified as no greater than some constant, D [$V(\overline{Y}) \leqslant D$]. The resulting function, using a Lagrangian multiplier, λ, is:

$$F = tC_p + tkC_s + tknC_a + \lambda\,[V(\overline{Y}) - D]$$

where $V(\overline{Y})$ is given by formula (2). D = specified variance of the mean.

By solving the simultaneous equations obtained through partial differentiation, the following formulas for n, k, and t are found:

$$n = \sqrt{\frac{V(a)}{C_a} \cdot \frac{C_s}{V(s)}} \qquad (4)$$

$$k = \sqrt{\frac{V(s)}{C_s} \cdot \frac{C_p}{V(p)}} \qquad (5)$$

$$t = \sqrt{\frac{V(p)}{C_p}} \cdot \sqrt{\frac{C_pV(p) + C_sV(s) + C_aV(a)}{V(\overline{Y})}} \qquad (9)$$

It should be noted that the above solutions probably will give answers in fractions of whole units, a situation which leads to possible uneasiness in practice. The following routine is suggested:

1. First, solve for n ($n \geqslant 1$). If n is a fraction, e.g., 1.42, consider Cameron's inequality (2)

$$\frac{V(a)}{C_a} \cdot \frac{C_s}{V(s)} \geqslant l(l+1)$$

where l is the next lowest integer below the calculated value of n.
If the inequality holds, choose $n = l+1$
If the inequality does *not* hold, choose $n = l$.

2. Solve for k, and apply a similar inequality to determine an integer evaluation.
3. Solve for t, using the following formula:

$$t = [V(p) + V(s)/k + V(a)/nk]/V(\overline{Y}) \qquad (7)$$

The substitution of integer solutions for n and k necessitate the adoption of this new formula (7) for guaranteeing the specified $V(\overline{Y})$.

The previous formula for t (6), while completely valid, will not exactly fit the specifications if the integer approximations are used. It should also be noted that the solution for k depends on the solution for n; if one wishes to be more exact, a correction similar to the last step above should be made at each calculation step. However those steps shown above will lead to workable solutions in practical cases.

In the sampling scheme previously considered the following specifications are desired:

1. $V(\overline{Y}) = 0.02$
2. C_a = \$2.00 cost of doing a single analysis
3. C_s = \$0.20 cost per sample per unit
4. C_p = \$0.50 cost of sampling a unit

Using the estimates of the variance components obtained in Table II, the number of analyses, n, is:

$$n = \sqrt{\frac{0.0216}{2.000} \times \frac{0.20}{0.0074}} = 0.54$$

Since one must do one analysis to get any quality estimate, n = 1.

The number of samples per unit is:

$$k = \sqrt{\frac{0.0074}{0.20} \times \frac{0.50}{0.0158}} = 1.08$$

Applying Cameron's inequality

$$\frac{0.0074}{0.20} \times \frac{0.50}{0.0158} \stackrel{?}{\geqslant} l(l+1)$$

$$1.17 \stackrel{?}{\geqslant} 2.$$

Since inequality does not hold: k = 1.

The number of process units to be sampled is:

$$t = |0.0158 + 0.0074/(1) + 0.0216/(1)(1)|/0.02$$

$$t = \frac{0.0448}{.02} = 2.24.$$

The choice of 2 or 3 is a decision that must be made by the sampler since $t = 2$ will yield a mean variance slightly larger than 0.02 and a $t = 3$, a mean variance slightly smaller than 0.02.

Conclusion

The following sampling scheme was decided upon. Three process units were to be selected at random each day. From each unit one random sample would be analyzed for T.O.B. The average of these three T.O.B. results would determine whether the process was adhering to specifications. This sampling scheme cost $8.10 per day; it guarantees that the variance of the mean $\leqslant 0.02$.

Note

1. Other refinements of sampling scheme determinations have been proposed. R. A. Fisher proposed the criterion: minimum cost (effort) per unit of information (3). Summaries and critical analyses of the various methods for the selection of optimum sampling plans can be found in an unpublished M.S. thesis by F. E. Free (4).
2. The experimental design suggested by the Fat Analysis Committee of the American Oil Chemists' Society for determining inter- and intralaboratory variation is this same "nested design." If one translates the elements of the design as follows: a) process units become laboratories; b) samples become laboratory technicians; and c) analyses remain the same, then the estimation of the variance components are obtained in a similar fashion shown below for the general case of "p" laboratories, "q" technicians in each lab, "n" analyses done on each test material.

Analysis of Variance

Source of variation	d.f.	s.s.[a]	m.s.[a]	E(m.s.)
Between laboratories	p-1			$\sigma_A^2+n\sigma_T^2+nq\sigma_L^2$
Between technicians within laboratories	p(q-1)			$\sigma_A^2+n\sigma_T^2$
Between analyses within technicians within laboratories	pq(n-1)			σ_A^2

[a] Omitted for convenience.

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[Received October 2, 1957]

ABSTRACTS . . . R. A. REINERS, Editor

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• Oils and Fats

Protection of fats and oils from oxidation. Z. K. Lebedeva and A. G. Sergeev. *Masloboĭno-Zhirovaya Prom.* 23(9), 17–20 (1957). Factors responsible for the oxidative processes occurring during storage, clarification, filtration, and collection of refined oil, as well as the protective methods involved are discussed. (*C. A.* 52, 3365)

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