SOURCES OF VARIABILITY IN LABORATORY MILLING YIELDS OF LONG-GRAIN RICE-DRYING EXPERIMENTS[1]

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ABSTRACT

Experiments were conducted to determine sources and magnitudes of components of variance, σ_i^2, affecting laboratory head yields of long-grain rice-drying experiments. Since interest centers on the *difference* between head yields of entering green rice and final-product dried rice, any variability due to days of milling and operator can be eliminated from experimental comparisons by the simple technique of saving early samples from a given drying run and evaluating all samples from such a run on the same day and by the same operator. The variance of head-yield changes is decreased faster by increased replication from more intensive sampling of the process stream than from any other form of increased replication.

Mill-scale drying investigations with short- and medium-grain rices have successfully charted changes in milling yields of rough rice attributable to various steps in the drying procedure (1,2,3). These results have been achieved by laboratory milling of samples taken at strategic points in the drying process, despite the fact that such samples cannot be evaluated without further drying, the very factor under investigation. This has been made possible by use of the laboratory sample dryer (2) developed at the Western Regional Research Laboratory for drying rice samples without other and extraneous effects. Owing to unforeseen difficulties, drying investigations with long-grain rice by the same procedures were disappointing in that yields of head rice from similar samples and duplicate determinations were often widely different.

To properly appraise the true milling characteristics of a lot or sample of rice, whether it be fresh from the field, or partially or fully dried, one must determine the variability of the test itself as well as the influence of incidental factors such as operator judgment, day on which the rice is milled, nonuniform conditions in the sample dryer, and imperfect methods of sampling. Studies were therefore undertaken to determine these quantities. In the procedure used, the rice was dried in the Western Regional Research Laboratory dryer and milled accord-

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ing to the constant milling time method outlined in the Rice Inspection Manual of the Agricultural Marketing Service (4). Milling tests were performed by the Rice Inspection Service, Grain Division, Agricultural Marketing Service, Beaumont, Texas. Because head yields of rice are affected by their moisture content (5), rice samples were dried to a constant moisture content of 12% prior to milling.

The work was planned to be accomplished in two experiments. In the first, designed to evaluate questions concerning the milling determination procedure, samples were prepared by the following schedule. A quantity of rice was withdrawn from each of two lots of rough rice thought to differ in head yield. Each withdrawal was thoroughly mixed and divided into 18 samples, each large enough for duplicate milling determinations. Nine samples from each lot were labeled and hermetically sealed; the other nine samples were split into pairs of subsamples large enough for single determinations, labeled, and hermetically sealed. On a given day one double sample and one *pair* of single samples (originally a single double sample) were taken from each lot, assigned code numbers to prevent identification, and given to one of three operators for evaluation. The operator's instructions were to make duplicate determinations on the samples which were large enough, but otherwise, only single determinations. Thus an operator made four milling determinations on each lot on a single day, two of these being the evaluation of samples he knew to be duplicates and the other two being the evaluation of duplicate samples he did not know to be duplicates. To sample the maximum number of days, no two operators were allowed to evaluate samples on the same day. The data obtained were used to answer the following three questions.

1. What is the order of magnitude of the basic variations in head yield existing between duplicate determinations made on the same sample of rice by a single analyst under some single set of conditions?

2. Do analysts obtain consistent milling results from day to day? If not, do results of a single analyst show wider variation when obtained on different days than when obtained on the same day?

3. Are there consistent differences among the milling yields obtained by different analysts when presented with samples from the same rices?

The second experiment of the study, designed to answer questions concerning sources of head yield differences existing among experimental samples as actually presented for evaluation, was somewhat more complex. For this experiment two similar lots of rice were subjected to the same plant drying schedule. Each lot was sampled by removing two large samples (5 gal.) from the process stream at each of three points, incoming green rice, rice after first drying pass, and rice after last drying pass. This made 12 large samples altogether — three sets of duplicate samples from each of the two drying lots. The two samples taken at the same point of the same lot were independent of each other in the sense that each consisted of 20 independently taken 1-qt. samples which were composited to a single 5-gal. sample.

The 5-gal. samples were allowed to temper or equilibrate overnight, after which they were divided into two equal portions. One portion was dried starting immediately; the other was held under refrigeration for 48 hr. to be dried on a different day.

Each of the two portions of a 5-gal. sample was subjected to the procedure described below and outlined in Fig. 1. The rice of a portion was divided between two trays which were dried simultaneously. After drying, each tray was divided into two samples which were labeled and hermetically sealed. Two of the samples (one from each tray) were milled together, i.e., on the same day; the other two were milled on another day different from the first. Duplicate 5-gal. samples were dried on the same schedule. All determinations in the second experiment were performed by a single operator, and all determinations in both experiments were performed on a single machine with a single huller which was reserved for these experiments only. The data obtained were used to answer the following four questions.

1. Does splitting and drying a subsample on different trays of the dryer create larger differences in head yield than would exist between determinations made on rice dried in a single tray?

2. Does the laboratory dryer give consistent results from day to day? Specifically, do subsamples from the same large bulk sample show more variation if dried on different days than if dried on the same day?

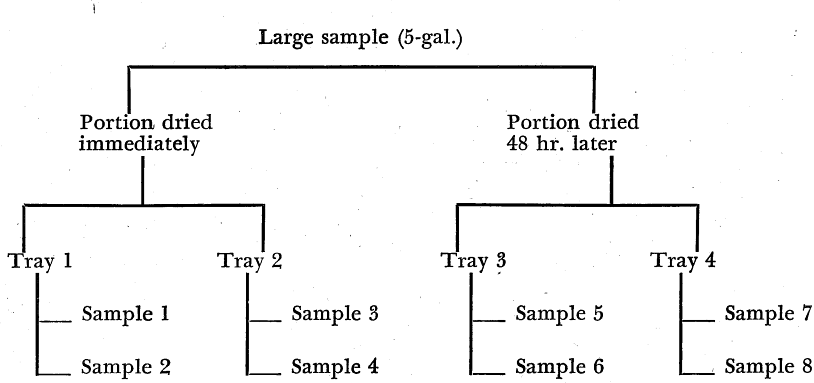

Fig. 1. Procedure for obtaining the eight milling samples of rough rice from a single 5-gal. sample.

3. Is it possible to homogenize and divide a sample so that the resulting subsamples are identical (within the limitations imposed by the variability in the remainder of the experimental and measuring process)?

4. Do large or sizable differences in yield of head rice exist between duplicate 5-gal. samples taken from a single drying lot by means of twenty 1-qt. increments from the process stream?

It was obvious before the experimentation was begun that differences among operators could be eliminated from treatment comparisons by the simple expedient of having each operator evaluate all treatments to the same extent. It was also plain that day-to-day variability of any single operator could be obviated simply by having him make equal numbers of milling determinations on each treatment each day. However, it was of some importance to the co-operators to evaluate these components under their operating conditions.

The experimental results were subjected to analysis of variance procedures to isolate, and estimate magnitudes of, the components of variance affecting head yield (6,7,8).

Results and Discussion

The analyses of variance for the two experiments are given in Tables I and II. Estimates of the variability between duplicate determinations are summarized in Table III, and estimates of other components or sources of uncontrolled variability are summarized in Table IV. The components and their estimates are discussed in the following sections.

Basic-Component Differences between Duplicate Determinations. The magnitude of the basic variability, that between duplicate determinations made on the same day on the same small sample, is estimated in the first experiment (Table I) for two possibly differing situations: 1) the operator knows that the samples are duplicates, or

TABLE I

Analysis of Variance of Experiment to Evaluate Milling Procedure

Sources of Variation	Degrees of Freedom	Mean Square	Sources of Variation	Degrees of Freedom	Mean Square
Total	70	....	M × L	1	0.007
Lots (L)	1	1.150	M × O	2	0.428
Operators (O)	2	1.640	M × D in O	6	0.300
Days of milling (D) in O	6	3.244	M × L × O	2	0.113
			M × L × D in O	6	0.385
L × O	2	0.275	Detn. of unknown duplicates	18	0.398
L × D in O	6	0.073			
Methods of sampling (M)	1	0.211	Detn. of known duplicates	17	0.669

TABLE II

ANALYSIS OF VARIANCE OF EXPERIMENT TO EVALUATE EXPERIMENTAL PROCEDURES

Sources of Variation	Degrees of Freedom	Mean Square	Sources of Variation	Degrees of Freedom	Mean Square
Total	95	......	Error (b), H × L + H × R × L	3	0.837
Lots (L)	1	205.335	Samples from process stream (S)	6	2.715
Stage of drying (R)	2	103.996	S × H	6	1.120
Error (a), L × R	2	1.820	Trays (T) in S × H	24	0.461
Age of sample, or effect of holding (H)	1	2.470	Days of milling (D)	24	0.666
H × R	2	2.193	D × T	24	0.285

TABLE III

ERROR IN DUPLICATE DETERMINATIONS OF HEAD YIELD OF LONG-GRAIN RICE

Estimate	Degrees of Freedom	Mean Square or Variance	Standard Deviation
Second experiment (tray × day interaction)	24	0.285	0.534
First experiment (unknown duplicates)	18	0.398	0.631
First experiment (known duplicates)	17	0.669	0.818
Pooled estimate	59	0.430	0.656

TABLE IV

COMPONENTS OF VARIANCE IN DETERMINING HEAD YIELD OF LONG-GRAIN RICE

Source of Component	Magnitude	
	Variance, $\hat{\sigma}^2_i$	Standard Deviation
Duplicate determinations	0.400[a]	0.632
Days of milling	0.178	0.422
Operators	0.0	0.0
Drying days	0.076	0.276
Trays dried together	0.030	0.173
Splitting samples	0.0	0.0
Samples from process stream	0.199	0.446

[a] See text discussion on splitting samples for explanation of difference from 0.430 in Table III.

2) the operator does not know that the samples are duplicates. The variability between duplicate determinations is also estimated in the second experiment (Table II), but in this case by cross-classifying the four samples from a single portion by drying tray and day of milling, determining the variability due to these two sources, attributing the residual to duplicate determinations, and pooling these results over the 24 portions. Despite the fact that the duplicate samples split from a single tray in the second experiment are milled on different days, this residual or interaction is still a measure of the basic determination error because of the balancing-out of day (and tray) effects from the interaction sums of squares.

The three estimates of determination error (variance of duplicate determinations) are given in Table III. Though the estimates may seem widely different to those not accustomed to estimating variability, no significance should be attached to the differences. Testing for homogeneity by Bartlett's procedure (8) discloses that more than 10% of variance estimates with these degrees of freedom would be as discrepant as these estimates due merely to chance, even though all estimates came from a single population. Thus it may be assumed that the three estimates are estimates of the same population variance. They are, therefore, pooled to obtain their weighted average. This pooled estimate of the variance of duplicate determinations is given in Table III, as 0.430 with 59 degrees of freedom. For the benefit of those who might think that the variability between known duplicates would be less than that between duplicates not known to be duplicates, it should be pointed out that the observed results are the opposite of this. For those who might, on first thought, have expected any estimate of determination variability derived from the second experiment (in which duplicate members are evaluated on different days) to be larger than either estimate from the first experiment (in which duplicates were evaluated on the same day), it should be pointed out that, again, the observed results dispute the supposition.

Component Due to Splitting Samples. The variability due to splitting a single small sample of rice and drying in separate trays, but at the same time, was examined for all of the 24 "portions" in the second experiment by isolating and pooling the sums of squares due to trays in the cross-classification by trays and milling days of the four milling samples of a portion described earlier. If a component of variability identifiable with trays should exist, it might be due to either of two things: 1) inability to divide the original sample equally (with respect to head yield) or 2) real changes induced by the individual tray experiences after splitting. Some additional evidence is obtainable from the first experiment on the variability due merely to the splitting of samples without much opportunity for different experiences to develop after splitting. On each of 9 days the two lots were each examined by the two "methods" of sample splitting — samples split beforehand without the operator's knowledge and then hermetically sealed, and samples split at the time of milling. In the analysis (Table I) the identity of these two "methods" was maintained. As already discussed, the variability of duplicate determinations was probably not affected. Further, no general difference between the two "methods" was demonstrated (Table I), and no interactions of any other variables with the "methods" were indicated (the chi-square test for homogeneity indicated that estimates as variable as "Methods" and "Method interactions" would be expected in fully one-half of all such estimates even if from a single population). In consequence, the samples representing the two "methods" (of splitting) should more nearly be regarded as just two random samples (about the size of those for drying trays) obtained by splitting still larger samples. As a consequence of this, the several estimates of mean squares for "methods" and "method" interactions of the first experiment should be pooled into a single estimate of the mean square of tray-sized samples obtained by splitting larger samples when opportunity is lacking for developing individuality by vagaries of experience after splitting. This pooled mean square turns out to be 0.301 with 18 degrees of freedom. This is less than the determination error for either of the estimates of that experiment and also less than the estimate 0.430 obtained when all other estimates of determination error were pooled. This would indicate almost no chance of a real and sizable component due to inability to divide a sample of this size (roughly two milling samples) into subsamples of equal head yield.

Additional Estimate of Determination Variability. Accepting the viewpoint that the component due to splitting samples down to tray size is either nonexistent or negligible, the mean square 0.301 with 18 degrees of freedom obtained from pooling sums of squares for "Methods" and method interactions with "Methods" in the first experiment should be taken as representing merely another estimate of determination variance. Pooling this with those estimates previously pooled in Table III yields a new pooled estimate of variance of determinations = 0.400 with 77 degrees of freedom. This is the estimate of basic variability that will be used throughout the rest of the paper.

Variability among Trays Dried Together. Having decided that there is no added variability due to inability to divide samples, one concludes that any component in the second experiment (Table II) attributable to trays would have to be attributed to changes induced by individual tray experiences (exposure to the drying process) rather than the splitting procedure proper. The mean square for trays pooled over the 24 portions was 0.461 (Table II). Compared to the pooled mean square for determinations, this does not indicate a significant component of variance due to drying trays:

$$F = \frac{0.461}{0.400} = 1.15.$$

However, since such a component could very easily exist, let it be admitted, and the magnitude of the variance component due to drying trays is then estimated from these same mean squares to be

$$(0.461 - 0.400)/2 = 0.030.$$

Day-to-Day Variability. There is evidence in both experiments that an analyst's results with the same lot of rice vary more from day to day than can be accounted for by the already known sources of variation; see "Days of milling" in Tables I and II. This variation would seem to be attributable directly to days (or some as yet unknown characteristic of days), since it is found to consistently affect all lots of rice evaluated on a given day, but not to inflate any of the terms measuring interactions with days. It is assumed, therefore, that there are factors associated with days which cause an analyst's results to vary, and that the factors seem to affect all lots and samples in the same manner and to the same extent. Any additional real component due to sample splitting would have shown up in increased magnitude of the interactions of milling days with lots and samples. If sizable, it could have increased these interactions to the point that the foregoing conclusion of nonexistence could not have been drawn. This is still further evidence that there is no component of variance due to splitting or subsampling. In the first experiment the added component due to days is estimated to be

$$(\text{Mean square for days} - \text{Det'n error})/8 = (3.244 - 0.400)/8 = 0.356,$$

and in the second experiment is estimated to be

$$(\text{Mean square for days} - \text{Det'n error})/2 = (0.666 - 0.400)/2 = 0.133.$$

The difference in divisors is occasioned by the fact that, while each daily mean of the second experiment is the mean of only two determinations from a single tray, each daily mean of the first experiment is the mean of eight determinations (two determinations by each of two methods on a sample from each of two lots). Since there is no good test of whether these two estimates of the component due to days differ, and since the estimates do not violently disagree, they are averaged and weighted by the degrees of freedom of the mean square in which they occurred (since the mean square subtracted is the same in both instances), to give a pooled estimate. Thus the pooled estimate of the variance component measuring the inability of an operator to repeat himself from day to day on the same rice is

$$[6(0.356) + 24(0.133)]/30 = 0.178.$$

Variability among Samples from Process Stream. The component due to variability between duplicate 5 gal. samples from the process stream was estimated from "Samples" mean square and "Samples $\times$ Age (S $\times$ H)" mean square in Table II as

$$(2.715 - 1.120)/8 = 0.199.$$

Effects of Holding Rice before Drying. It is uncertain whether there is a consistent and significant difference due to holding the rice under refrigeration until a later date ("Age of sample (H)," Table II). It is also uncertain whether the holding effect, if any, is more pronounced at some stages of drying than others ("H × R," Table II). But it is not very important to know, because, when the procedure is finally standardized, the length of the holding period should be constant for all samples, so that the consistent effect, whatever it may be, will not affect treatment comparisons. Further, the interaction of such an effect with stage will become, for any particular stage, a constant and permanent part of all values obtained at that stage, thus becoming a part of the true value for that stage *when evaluated under the specified conditions.*

Random Components Due to Day of Drying. Splitting and drying samples on different days contributed additional variability of a random nature due to interaction of the samples with age, i.e., with a 2-day holding period under refrigeration. This variability is probably due to day of drying and is thought to be of a random nature rather than due to any consistent type of effect caused by the rice being always older on the second date than on the first. The estimate of this random component due to drying days is

$$\{1.120 - [0.400 + 2(0.178) + 2(0.030)]\}/4 = 0.076,$$

where 1.120 is the mean square for "S × H" in Table II; and 0.400, 0.178, and 0.030 are the numerical estimates of the components due to determination, day of milling, and drying tray.

Additional Evidence of No Variability Due to Splitting Samples. Any inconsistent or differential effect from one mill run to another attributable to holding the rice, such as would become apparent if the rice samples could not be split into homogeneous subsamples when divided for the purpose of drying one subsample and refrigerating the other, probably does not exist. This effect is estimated from the mean squares for "Error (b)," i.e., pooled "(H × L)" and "(H × R × L)," and for "Samples × Age, (S × H)," or more exactly (H × S in L), to be negative, since

$$0.837 - 1.120 = -0.283.$$

The negative value probably means that such a component does not exist, and constitutes additional evidence that samples can be subdivided and split without additional variability being created.

Variability among Operators. The differences among operators were no larger than those explainable by the basic variability of duplicate determinations, plus the day-to-day variability of operators; i.e., the mean square for "Operators," Table I, is smaller than either the mean square for "Days of milling" in Table I or 0.400 + (8)(0.178). Thus, it is concluded that there is no component due to operators; i.e., in the population of operators represented by these particular individuals, operators are able to duplicate each other's work.

Considerations General to Experimental Design and Sampling. The results summarized in Table IV and discussed in the preceding paragraphs would indicate that future work may ignore the error introduced by splitting or subdividing samples if future samples are as thoroughly and carefully mixed and divided as these. They also indicate that the variability due to using different operators may be ignored, assuming operators will be as carefully chosen and trained for accuracy as in these studies. Since, however, it is difficult to ascertain with certainty whether operator results are identical but fairly easy to schedule work so that each operator does evaluate equal numbers of samples from all treatments, it is suggested this latter procedure be followed in future evaluations of experimental work as a precautionary measure against the possibility of operators who do perform differently.

From Table IV it would seem that the components or sources of variance creating the most variability or uncertainty in experimental results with plant drying of rice are those due to variability of the determination procedure itself (variance of duplicate determination = 0.400), to variability among supposedly duplicate samples from the process stream (= 0.199), and to the variable results obtained from day to day by the same operator with the same rice (= 0.178).

One might immediately suggest, wholly on an intuitive basis, that a possible means of reducing the variability of the estimate of head yield at some stage in the drying of a lot of rice would be to take a more thorough sample, i.e., composite more individual dips from the process stream, thus cutting down on the component due to differences among duplicate samples. This is in agreement with theory, but unfortunately the experiment obtained no evidence on the variability of samples taken by any other scheme. It is reasonable, however, to expect that the component of variability representing differences among duplicate 5-gal. samples is composed of two parts – that due to the individual dips of rice composited into the sample, and that due to the handling or dividing procedure. *But,* it was indicated at several points in the analysis of the experiments that variability due to handling probably does not exist. This leaves only the variability of the small quantities taken from the stream. It seems fair, because of the lack of variability in handling and subdividing, to assume that the value obtained for this composite is really an average of the 20 individual quarts of rice. This assumption implies that the component 0.199 due to samples is really a component due to means of 20 dips:

$$\hat{\sigma}^2_{\bar{x}} = \frac{\hat{\sigma}^2}{20} = 0.199,$$

so that the variance of dips is estimated to be

$$\hat{\sigma}^2 = (20)(0.199) = 3.98.$$

The advantage of this manipulation is that, assuming the dips are normally and independently distributed, decreases in the component due to sampling the process stream depend in the following manner upon the number of dips taken:

$$\hat{\sigma}^2_{\bar{x}} = \frac{\hat{\sigma}^2}{\text{No. of dips}} = \frac{3.98}{\text{No. of dips}}$$

Consideration Explicit to Drying Experiments. At this point it should be brought out that, in drying experiments, the experimenter is probably not interested in the direct comparison of head yields from rice dried under two different drying schemes because of possible differences in head yield existing between the lots even before treatment. Rather, he would prefer to compare the *change* in head yield from green to dry rice of the first drying procedure with the *change* in head yield of the second drying procedure. Thus the only averages which are to be *directly* compared with each other are those taken *at different stages from the same drying lot of rice.* It seems quite possible that early samples from a particular drying lot could be held until all are ready for milling, then all could be milled on the same day. In this way the *change* in head yield could be estimated *completely free* of the components of variability due to operators and to days, because all determinations are made by the same operator on the same day. This procedure could be followed for all drying regimes, with the result that all treatment comparisons could be made on the basis of their *changes* from entering green rice to completely dried rice, completely free of any uncertainty due to differences among operators and day-to-day variability of a given operator.

In view of the foregoing discussion, the following scheme is suggested for evaluating a rice-drying procedure. 1) Take duplicate samples from the process stream with as many dips from the stream as practicable. 2) Split each sample and dry on as many different days, and in as many trays per day, as seem practicable. (This can be one tray on one day only.) 3) Hold early samples from a particular drying scheme until all are ready, when they should be presented to a single operator for equal numbers of analyses per day.

Use of the Variance Component Estimates to Evaluate Different Sampling Schemes. If the foregoing suggestions are followed, the mean difference in head yield between incoming green rice and final dried product will not be affected by day of milling or operators, and the variance of such a difference may be estimated as

$$\hat{\sigma}^2_{\bar{x}} = \frac{0.400}{\text{ktds}} + \frac{0.030}{\text{tds}} + \frac{0.076}{\text{ds}} + \frac{(3.98/\text{n})}{\text{s}}$$

where s is the number of large samples taken from the process stream;
n is the number of dips composited from the process stream in obtaining a large sample;
d is the number of different days on which a particular sample is dried;
t is the number of trays of a particular rice sample dried at any one time;
k is the number of replicate (duplicate, triplicate, etc.) determinations made on any particular sample.

Suppose a sampling of four determinations obtained by the scheme of two samples consisting of 25 dips from the process stream with two trays per sample dried on one day only and single determinations made on the resulting dry samples, then the variance of the mean would be estimated to be

$$\hat{\sigma}^2_{\bar{x}} = \frac{0.400}{(1)(2)(1)(2)} + \frac{0.030}{(2)(1)(2)} + \frac{0.076}{(1)(2)} + \frac{(3.98/25)}{2} = 0.226.$$

The variance of the *change* in head yield from green to dried rice of the same lot would then be twice the foregoing result or $\hat{\sigma}^2_{\text{change}} = 2(0.226) = 0.452$. From this one would estimate the 95% confidence limits on the difference between the changes in two drying procedures (or the least significant difference between changes) to turn out to be about

$$\text{LSD} = t_{0.05}\sqrt{2\hat{\sigma}^2_{\text{change}}} = 2\sqrt{2(0.452)} = 1.9\% \text{ in head yield,}$$

where $t_{0.05}$ is the value of student's t at the 0.05 probability level.

If one should double the number of determinations to eight by making duplicate instead of single determinations, or by drying four trays instead of two or by drying on two days instead of one or by taking four samples from the process stream instead of two, then the estimated variances of the means would be reduced to 0.176, 0.172, 0.153, and 0.113 respectively, leading to least significant differences between changes of 1.7, 1.7, 1.6, and 1.3 respectively. Thus it becomes apparent that replication created early in the procedure is more efficient than that created later, the most efficient replication being that obtained by taking more samples from the process stream. This replication reduces the amount of all components found in the mean. The least efficient replication is that resulting from duplicate or triplicate determinations made on a given dry sample. This replication reduces only one component, the determination error. Use of at least duplicate samples from the process stream enables a continuing check on the full variability of the sampling and evaluating procedure.

Conclusions

Because the real concern in drying experiments relates to the *change* in head yield from green to dried rice, the component due to milling days, as well as the possibility of a component due to operators, can be obviated by having all the samples of a particular drying run evaluated by the same operator on the same day. Increased numbers of evaluations are most effective if they result from taking more samples from the process stream, and least effective if they result from performing duplicate determinations on the samples presented.

It is suggested that the process stream be sampled as intensively as possible, and if multiple dryings on the sample dryer are to be made, that these not be made on the same day, that one or some be held under refrigeration until the next day. It is strongly suggested that no effort be expended on duplicate determinations; that if duplicate determinations are feasible, one should consider instead the possibility of splitting into different trays before drying; or better yet, splitting into portions for drying on different days; or best of all, doubling the number of samples from the process stream.

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