Accuracy of Subsampling Mill for Granular Materials[1]

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ABSTRACT

A subsampling mill was constructed with 19 subsample spouts similar to the 1 or 2 subsample spouts on the conventional subsampling mills. Samples of peanuts, each of which contained 1 kernel made radioactive by neutron activation, were comminuted in the mill. For treatment M the discharge from each spout was kept separate. For treatment R, the discharge from all 19 spouts was blended together in a twin-shelled blender and subdivided with a riffle divider into 16 subsamples which averaged the same weight as the M subsamples. Radioactivity counts/gram of each subsample were measured. Eleven samples were comminuted for each treatment which produced a total of 209 M samples and 176 R samples. An analysis of variance on the pooled data from each treatment showed a mean square error of 1318.3 for the M treatment and 1278.6 for the R treatment. The null hypothesis that the mean square errors for the two treatments are equal was not rejected by the F test (P = 0.421).

INTRODUCTION

A subsampling mill designed to simultaneously comminute and subsample large samples of granular material has been specified for use by the peanut industry to prepare peanut samples for aflatoxin analyses (1,2). The mill has been suggested for preparation of samples of other products for mycotoxin analyses (3).

The subsampling mill is shown in Figure 1. During operation, the cylindrical screen is fastened to a circular platform beneath the blades. As the sample is poured into the chamber thus formed, it is comminuted by the rotating blades. The particles swirl within the chamber until they pass through the screen. A subsample enters the spout formed by two vanes that radiate from the screen surface to the outer shell of the mill. The remainder falls through the opening between the screen and the outer shell. Ratio of subsample to sample weight is determined by the percentage of the total screen area that is between the vanes that form the subsample spout. Comminuted particle size is determined by the openings in the screen. Openings of ca. 3.2 mm are required to prevent the screen from clogging when peanuts are comminuted.

All of the aflatoxin in a sample of peanuts may be confined to only one contaminated kernel (4,5). An accurate subsample would contain the same aflatoxin concentration as the sample. Variance of subsample concentrations about the sample concentration is a measure of subsampling error (4). Due to the particulate nature of comminuted material produced by the subsampling mill, there is an "inherent" variance in aflatoxin concentration among subsamples composed of particles taken at random (5). Variance in excess of the "inherent" variance may be attributed to a bias in the way subsamples are taken by the mill.

The purpose of this study was to compare the variance among subsamples of peanut material comminuted and subsampled with the subsampling mill to the "inherent" variance among subsamples taken with a riffle-type divider from a thoroughly blended sample of peanut material that was comminuted with the subsampling mill.

PROCEDURE

Comminution and Subsampling

A 20-spout subsampling mill constructed for the study is shown in Figure 2. The spacing between the vanes that formed each sample spout was 1/20 the circumference of the screen. Due to the thickness of the vanes, the 20th spout was narrower than the other spouts. The spouts were consecutively numbered 1-20 in a clockwise direction, as viewed from the top, with the narrow spout numbered 20. As a sample passed through the mill, the entire sample was comminuted and subdivided into 20 subsamples that were caught in plastic bags fastened to the discharge of the spouts. Except for the narrow spout, the ratio of subsample to sample size, the screen size, and all other characteristics of the 20-spout mill were the same as for the single- or double-spout mills now used for peanuts.

Aflatoxin analyses are imprecise (4); so peanut kernels made radioactive by neutron activation were used in the study. Stoloff et al. also used radioactive peanut kernels to study subsampling error (5). The peanut kernels were neutron-activated to ca. 6 μc/kernel by the North Carolina State University Nuclear Service Laboratory. This was

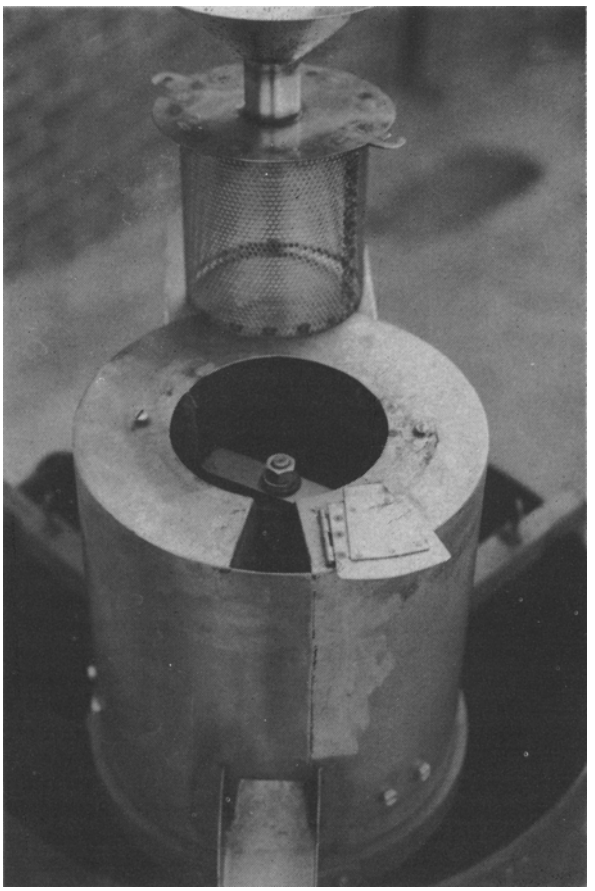

FIG. 1. Single-spout subsampling mill with screen removed.

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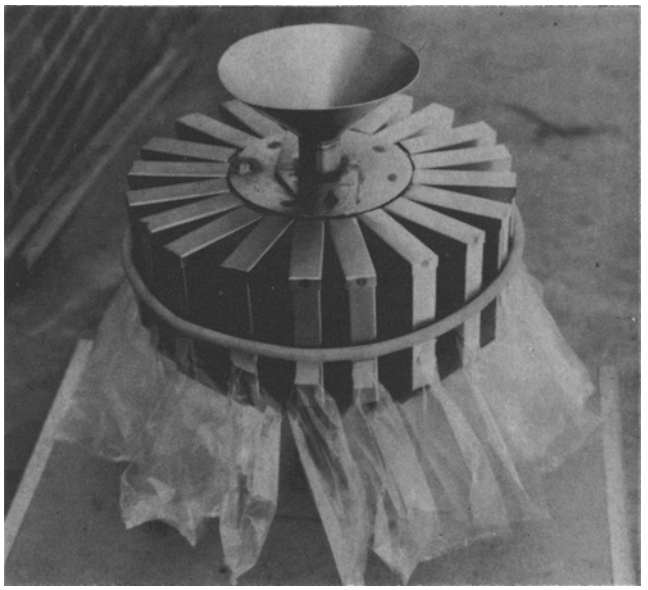

FIG. 2. Twenty-spout subsampling mill used in the experiment.

accomplished by a 10 minute irradiation at a flux of 1.5 x 10^{13} n/cm^2-sec in the vertical exposure port of a 1 megawatt Pulstar reactor. These procedures were designed to produce enough radioactivity of sufficiently long half life that even when the peantus were blended with nonradioactive peanuts to dilution factors of 1200 or more, the experiment could be conducted easily and accurately. The activities measured were gamma rays from the Na-24 and K-42 isotopes. For each subsampling run, a 600 g sample of nonradioactive peanut kernels, a single radioactive kernel, and another 600 g sample of nonradioactive peanut kernels were fed into the mill in rapid sequence so that feeding was continuous. The first 600 g sample preloaded the mill and the second 600 g sample purged the mill of radioactive particles.

Subsamples from one type of subsampling run were numbered M1 through M19 to indicate they were taken with the mill and to indicate the spout that collected the subsample. (Discharge from spout 20 was discarded for all runs). Another type of subsampling run was then made from which all 19 subsamples were blended in a twin-shell blender for 15 min. Because 16 approximately equal subdivisions were more easily obtained with a riffle divider than are 19, a portion of the blended material equal to 16 times the average weight of subsamples M1 through M19 of the immediately preceding run was divided from the blended material with a riffle divider that had 6.4 mm-wide slots (Fig. 3). This portion was then divided into 16 subsamples, numbered R1 through R16, with the riffle divider. Thus, the average weight of the M subsamples and R subsamples were the same for each pair of runs. Each subsample was weighed and sealed in a glass sample jar that was then sealed in a small plastic bag. When each pair of runs was completed, radioactivity of the samples was measured. The 11 pairs of subsampling runs produced a total of 209 M subsamples (11 runs x 19 subsamples per run) and 176 R subsamples (11 runs x 16 subsamples per run).

Radioactivity Measurements

Radioactivity was measured with a large volume Ortec 24% Ge(Li) detector coupled to a computerized ND6603 data acquisition system. The sample jar sealed within the plastic bag was placed in the Cu-Cd lead-lined counting chamber and 20 second counts were made for each subsample at 30 second intervals. Thus, 9 min elapsed from the start of counting subsample M1 until the start of counting M19, and 7.5 min elapsed from start of counting R1 until the start of counting R16. Data were derived from the counts in the gamma spectrum from Na-24 (1.37 MeV gamma) and K-42 (1.52 MeV gamma). Errors due to sample geometry were minimized by counting each subsample in the same detector location and about the same subsample volume.

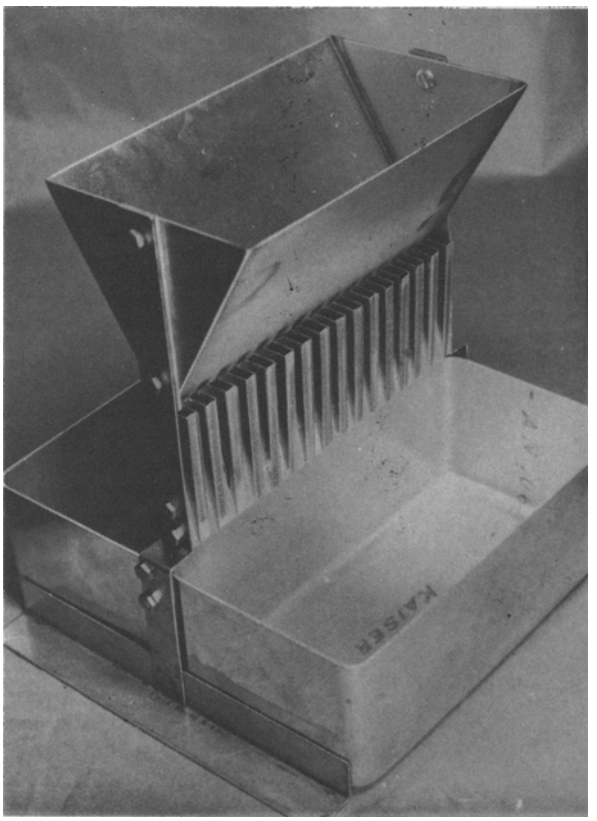

FIG. 3. Riffle-type divider used in the experiment.

The total variance T among the 19 radioactivity counts/gram (CPG) for a set of subsamples M1 through M19 or a set of subsamples R1 through R16 may be expressed by the following equation:

$$T = S + X,$$

where S is the variance due to the difference in radioactivity/gram among the subsamples in the set at the time counting started on the set (subsampling error), and X is the variance due to error in measurement of this radioactivity. Some causes of the variance in radioactivity measurement are (i) variance in CPG due to normal counting error of the instrument, (ii) variance in CPG due to decay of radioactivity between the time counting started and was completed on the set, and (iii) variance in CPG due to the effects of radioactive particle distribution within the bottle of material (configuration). Subsamle weight and density were assumed to be constant within each set of subsamples. Use of approximately equal sample volumes, the 6 μc/kernel specific activity and isotopes with long half-lives greatly reduced these sources of error.

The value of X was estimated by computing the variance among 19 counts made on the same subsample. The 20 second counts were started at 30 second intervals, and 9 minutes elapsed from the start of count 1 until the start of count 19. The subsample was thoroughly blended before each count by shaking the bottle for 15 seconds. Six estimates of X were obtained by applying this procedure to 6 different subsamples.

TABLE I

Results of Analysis of Variance on Pooled Data from 6 Counting Tests to Estimate Errors in Measurement of Radioactivity in the Subsamples (X)

Source	19 Counts/test	16 Counts/test
Mean square errors (X)	63.5	65.9
Degrees of freedom	108(18 x 6)	90(15 x 6)
Standard deviation	8.0	8.1
Mean count/gram	230.5	232.0
Coefficient of variation	3.4%	3.5%

TABLE II

Results of Analysis of Variance on Pooled Data and Related Statistical Data for 11 Subsampling Runs[a]

Source	Subsampling mill	Riffle divider
Mean square errors (T)	1318.3	1278.6
Degrees of freedom	198(18 x 11)	165(15 x 11)
Standard deviation	36.3	35.8
Mean count/gram	237.4	241.7
Coefficient of variation	15.3%	14.8%

[a]Null Hypothesis: Mill T = Riffle T. F Test: F = 1318.3:1278.6 = 1.07. P = 0.421.

RESULTS AND DISCUSSION

Results of an analysis of variance on the pooled data from the 6 tests to determine the variance due to error in measurement of radioactivity X are shown in Table I. An analysis of variance was made on the first 16 counts in each of the 6 tests to determine X for the riffle divider. All 19 counts in each test were used to determine X for the subsampling mill. For the subsampling mill and the riffle divider, X equaled 63.5 and 65.9, respectively, with respective CVs of 3.4% and 3.5%.

The results of the analysis of variance on the pooled data from the 11 runs each on the subsampling mill and the riffle divider are presented in Table II. The mean square errors T for the subsampling mill and the riffle divider are 1318.3 and 1278.6, respectively. The values of X given in Table I are small in comparison with T; so S is approximately equal to T. The null hypothesis that values of T for the two treatments are equal was not rejected by the F test (P = 0.421).

Failure of the experiment to show a statistically significant difference between the T values, and therefore the S values, for the two subsampling methods indicates that subsampling with the subsampling mill is as accurate as subsampling a thoroughly blended sample of the same material with a riffle divider. Although this paper deals with subsampling for radioactivity, the same conclusion would hold for aflatoxin or other compounds. However, subsampling error probably would be different depending upon the distribution of the compound in the sample. Also, if the physical properties of the contaminated kernels or particles were such that they were more finely comminuted than the radioactive kernels in this study, the subsampling error would be reduced.

Increasing the size of the subsample would also decrease subsampling error. According to statistical theory, doubling the subsample size would halve the subsampling error variance. Subsample size may be increased by using two spouts, by using wider spouts or by using a larger sample. The cost for larger quantities of extraction solvents and/or other problems of analysis would have to be considered in relation to the use of larger subsamples. Use of a screen with smaller perforations would reduce particle size and subsampling error. However, slow operation, screen clogging and overloading of the mill would result if screen perforations were too small.

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