Sampling and Analytical Variability Associated with the Determination of Total Aflatoxins and Ochratoxin A in Powdered Ginger Sold As a Dietary Supplement in Capsules

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The U.S. Food and Drug Administration is studying the need to monitor dietary supplements for mycotoxins such as total aflatoxins and ochratoxin A. An effective mycotoxin-monitoring program requires knowledge of the sampling and analytical variability associated with the determination of total aflatoxins (AF) and ochratoxin A (OTA) in dietary supplements. Three lots of ginger sold as a powder in capsule form and packaged in individual bottles were analyzed for both AF and OTA. The total variability associated with measuring AF and OTA in powdered ginger was partitioned into bottle-to-bottle, within bottle, and analytical variances. The variances were estimated using a nested design. For AF and OTA, the within-bottle variance associated with the 5 g laboratory sample size was the largest component of variability accounting for about 43% and 85% of the total variance, respectively; the analytical variance accounted for about 34% and 9% of the total variability, respectively; and the bottle-to-bottle variance accounted for about 23% and 7% of the total variance, respectively. When the total variance is converted into the coefficient of variation (CV or standard deviation relative to the mean concentration), the CV is lower for AF (16.9%) than OTA (24.7%).

KEYWORDS: Sampling; analytical uncertainty; aflatoxins; ochratoxin A; ginger capsules; dietary supplements

INTRODUCTION

Ginger roots are widely used for digestive problems and dietary supplements. In China, ginger roots are medicinal plants used to treat the common cold. Currently, it is estimated that half of all health care delivered in China is based on traditional herbal medicines. In the developed countries, ginger is used as a food condiment or as a nutritional supplement. They are marketed in many forms such as fresh or dried products; liquid or solid extracts; and tablets, capsules, powders, and tea bags. Ginger is not approved for the treatment or cure of any disease and is consumed as food or as dietary supplements. The major contaminants have been microbial, pesticides, heavy metal, and

mycotoxins. Mycotoxins, specifically the aflatoxins (AF), a group of chemical structurally related compounds consisting of aflatoxins B_1, B_2, G_1, and G_2 (**Figure 1**), and ochratoxin A (OTA) (**Figure 2**) are among the major mycotoxins found in agricultural commodities (*1*). AF and OTA have shown adverse effects to human and animal health. The few occurrences and incidences studied thus far have indicated that the levels of contamination of these toxins in botanicals are probably minimal. However, under adverse weather conditions and poor storage practices, high levels of AF and OTA can occur. Several surveys of AF and OTA in botanicals have been published (*2−7*). In order to accurately estimate the true level of these mycotoxins in dietary supplements, the variability associated with the mycotoxin testing procedure is needed. A test procedure is usually composed of sampling, sample preparation, and analytical steps.

Powdered ginger is often marketed in capsules, and the capsules are packaged in bottles. A lot at the retail level is considered to be some number of bottles that are collectively

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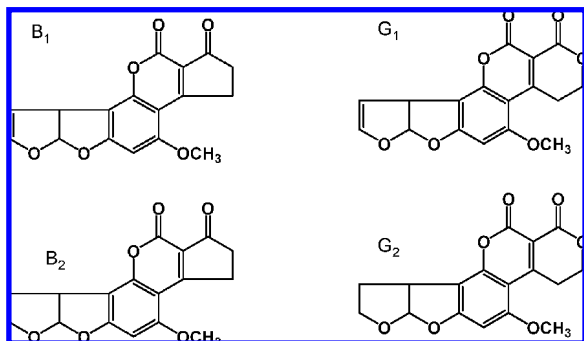[⊥] National Institutes of Health.

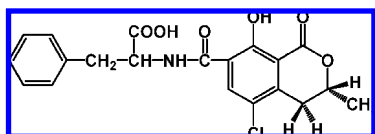**Figure 1.** Chemical structures of aflatoxins B₁, B₂, G₁, and G₂.

Figure 2. Chemical structure of ochratoxin A.

identifiable by some product code established by the processor. If the powdered ginger in the processing plant (source) is well mixed prior to packaging in capsules and bottles, the laboratory sample (smallest sample from which the mycotoxin is extracted) can be selected from any bottle or number of bottles taken from the lot. However, if the powdered ginger is not well mixed and the contaminated particles are not homogeneously distributed throughout the lot before packaging, the contents from a number of bottles should be pooled into an aggregate sample. The aggregate sample can be thoroughly mixed before the laboratory sample is removed and analyzed for specific mycotoxins.

There is variability among replicated laboratory sample concentrations taken from the same aggregate sample even if the laboratory samples are assumed to be representative samples. The total variability associated with the mycotoxin test procedure, as measured by the variance, is the sum of the sampling and analytical variance components (8, 9). Since powdered ginger from capsules is finely comminuted before packaging in capsules, there is no sample preparation step (additional grinding and subsampling) in our study, and we focused only on sampling and analytical variability. Specifying the number of bottles to select and pool the contents into an aggregate sample and the size of the laboratory sample taken from the aggregate sample depend on knowledge of the variability associated with the sampling and analytical steps of the mycotoxin test procedure.

This study was designed to (a) determine if the dietary supplement ginger sold as a powder in capsule form and packaged in individual bottles is contaminated with AF and OTA and (b) determine both the within-bottle variability among 5 g laboratory sample results taken from individual bottles of powdered ginger and the bottle-to-bottle variability among AF and OTA sample test results. The bottle-to-bottle variance can be used to make recommendations for the number of incremental samples (bottles) that need to be selected from a lot and pooled to form an aggregate sample to overcome any lack of homogeneity among contaminated particles in the lot. The within-bottle variance can be used to recommend the size of the laboratory sample (or number of laboratory samples) to be selected from an aggregate sample to get an accurate estimate of the true mycotoxin concentration in the lot.

MATERIALS AND METHODS

Ginger Capsules. On three separate occasions, separated by six month intervals, 20 bottles of ginger (*Zingiber officinale*), each

containing 60 capsules (625 mg of ginger per capsule) was purchased from Penn Herb (Philadelphia, PA). On each occasion, 20 bottles were purchased at the same time. The three groups of 20 bottles were assumed to have come from three separate lots since there were no lot identification codes on the bottles. Each bottle contained 60 capsules or a total of 37.5 g of powdered ginger. For a given group of 20 bottles, all 60 capsules in each bottle were broken open, and the powdered ginger was combined for a total of 37.5 g of powdered ginger per bottle. The 37.5 g of ginger from each bottle was tumbled for 4 h to thoroughly mix the ginger.

Chemical Analysis of AF and OTA. A published method was used to simultaneously extract and purify the AF and OTA in the powered ginger (*10*). The isolated AF and OTA were then separated and quantified with two separate reversed phase liquid chromatographic (RPLC) systems as described below. The 5 g laboratory sample of powdered ginger was placed in a 50 mL centrifuge tube, and 1 g NaCl was added. After adding 25 mL of a mixture of methanol and 0.5% sodium bicarbonate (7 + 3 v/v), the laboratory sample was vigorously shaken with a mechanical shaker for 10 min and was centrifuged for 5 min. A portion of the supernatant, 7 mL, was diluted with 28 mL of 100 mM phosphate buffered saline (PBS, pH 7.4) containing 1% Tween 20 (Sigma-Aldrich, St. Louis, MO). The diluted extract was filtered through a glass microfiber filter paper. The 25 mL filtrate was then passed through a multitoxin (AF and OTA) immunoaffinity column, AflaochraTest column, (G1017, Vicam, Watertown, MA). The column was washed first with 5 mL of 10 mM PBS, pH 7.4, and finally with 5 mL of water. The toxins were eluted with 2 × 1 mL methanol. The eluate was collected into a 3 mL volumetric flask and was diluted with water to volume.

AF was separated and determined by reversed phase liquid chromatography (RPLC) using a Waters 4.6 × 150 mm column (catalog No. AQ12S031546WT, YMC ODS-AQ S-3) and a postcolumn photochemical derivatization cell (AURA Industries, New York, NY), and fluorescence detection with the fluorescence detector (2475 fluorescence detector, Waters, Milford, MA) set at excitation wavelength 362 nm and emission wavelength 440 nm. The mobile phase was methanol/acetonitrile/water (25 + 15 + 60 v/v), and the flow rate was 0.8 mL/min.

OTA was separated and determined by RPLC using a Beckman 4.6 × 250 mm, 5 μm, C-18 column (catalog No.235335, Utrasphere) and fluorescence detection with the detector set at excitation wavelength 333 nm and emission wavelength 360 nm. The mobile phase was acetonitrile/water/acetic acid (47 + 53 + 1 v/v), and the flow rate was 1 mL/min.

Recovery Study. Average recoveries (n = 4 per level) of AF added at 2, 4, 8, and 16 ng/g and OTA added at 1, 2, 4, and 8 ng/g were 75−80% and 86−95%, respectively (*10*).

Experimental Design. The nested design used to measure sampling and analytical variances is represented in **Figure 3**. Initially, two 5 g laboratory samples were removed from each of the 20 bottles in lot 1. It was later decided to remove four 5 g samples from each of the 20 bottles for lots 2 and 3. The 5 g laboratory samples were identified by lot (1, 2, or 3), bottle (1 to 20), and laboratory sample number (1 to 4). For all lots, one AF and one OTA measurement was made per 5 g sample, except for lot 3 where two AF and two OTA measurements were made for laboratory sample 1 to estimate analytical variability.

Measurement of Variability. From the nested design (**Figure 1**), the total variance (s^2_t) among all sample test results per lot is the sum of the bottle-to-bottle variance (s^2_{btb}), within-bottle variance (s^2_{wib}), and analytical variance (s^2_a).

$$s^2_t = s^2_{btb} + s^2_{wib} + s^2_a \qquad (1)$$

For lots 1 and 2, the experimental design did not allow for a direct measurement of the analytical variance. Instead the bottle-to-bottle (s^2_{btb}) and the combined within-bottle and analytical variance (s^2_{wiba}) was measured.

$$s^2_t = s^2_{btb} + s^2_{wiba} \qquad (2)$$

where

Sampling of Mycotoxins in Ginger Capsules

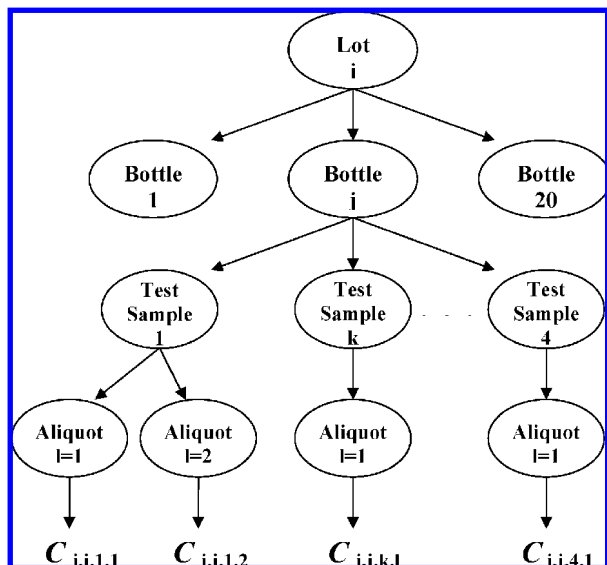*J. Agric. Food Chem.,* Vol. 57, No. 2, 2009 **323**

Figure 3. Nested design used to partition the total variance associated with measuring AF and OTA concentration (C) in powdered ginger for lot 3. Lot 1 had a similar design except that the number of laboratory samples (*k*) was equal to 2, and the number of aliquots quantified (*l*) was 1 for all laboratory samples. Lot 2 was similar in design except that the number of aliquots quantified was 1 for all laboratory samples.

$$s^2_{wiba} = s^2_{wib} + s^2_a \qquad (3)$$

For lot 3, the experimental design allowed the combined within-bottle plus analytical variances to be separated into within-bottle variance and analytical variance.

The variance components in eqs 1 and 2 were determined using Proc Nested in the Statistical Analysis System (SAS). For lots 1 and 2, estimates of s^2_t, s^2_{btb}, and s^2_{wiba} were made. For lot 3, estimates of s^2_t, s^2_{btb}, s^2_{wib}, and s^2_a were made.

RESULTS AND DISCUSSION

Variances Associated with Measurements of AF. The total variance associated with measuring AF in a 5 g laboratory sample by RPLC methods was partitioned into bottle-to-bottle and combined within-bottle plus analytical variances for lots 1, 2, and 3 (**Table 1**). In addition, the analytical variance associated with the AF analytical method is shown in **Table 1** for lot 3. Since the AF concentrations among the three lots were similar, the variances for the three lots were averaged, and the results are shown in **Table 1**. One sample test result from lot 1 and five sample test results from lot 3 were considered outliers and were not used in the statistical analysis. AF values that fell outside the range defined by the lot mean ± 3 times the standard deviations were identified as outliers.

The total, bottle-to-bottle, and combined within-bottle plus analytical variances averaged across the three lots was 1.544, 0.354, and 1.190, respectively. If the analytical variance from lot 3 is subtracted from the average combined within-bottle plus analytical variance (eq 2), the within-bottle variance is 0.662 (assume that the analytical variance for lots 1 and 2 is the same as that of lot 3). The total variance (eq 1) is equal to the sum of the bottle-to-bottle, within-bottle, and analytical variances or 1.544 (0.354 + 0.662 + 0.528). The bottle-to-bottle, within-bottle, and analytical variances account for 22.9, 42.9, and 34.2% of the total variance, respectively. The above variances are specific to using RPLC methods to measure AF in powdered ginger with 5 g laboratory samples taken from a lot at 7.34 ng/g total AF.

The analytical variance (0.528) associated with measuring AF in one aliquot by RPLC can be used to predict the analytical variance for any number of aliquots, na, quantified for AF. The analytical variance for any number of aliquots is

$$s^2_a = (1/na)\ 0.528 \qquad (4)$$

Increasing the number of aliquots, na, quantified for AF and averaging the results can reduce the analytical variance by the amount predicted from eq 4.

The within-bottle variance (0.662) associated with measuring AF in a 5 g laboratory sample taken from an aggregate sample can be used to predict the within-bottle variance for any given laboratory sample size, tns, in grams.

$$s^2_{wib} = (5/tns)\ 0.662 \qquad (5)$$

The within-bottle variance can be reduced by increasing the laboratory sample size, tns, by an amount predicted by eq 5.

The bottle-to-bottle variance (s^2_{btb}) of 0.354 reflects additional variability over and above the within-bottle variance due to the heterogeneity of the AF contaminated particles from bottle to bottle in the lot. The bottle-to-bottle variance (s^2_{btb}) provides an indication of the heterogeneity of the AF contamination from bottle to bottle in the lot and can be used to decide on how many bottles should be taken from the lot and combined to form an aggregate sample. The bottle-to-bottle variance (s^2_{btb}) for any given aggregate sample size bns in number of bottles can be estimated from 0.354 for a single bottle of ginger.

$$s^2_{btb} = (1/bns)\ 0.354 \qquad (6)$$

The total variance associated with pooling ginger powder taken from capsules from bns bottles to form an aggregate sample, taking a laboratory sample of tns grams from the aggregate sample, and quantifying the AF by RPLC in any number of aliquots, na, can be determined by summing eqs 4, 5, and 6 (as shown in eq 1).

$$s^2_t = (1/bns)\ 0.354 + (5/tns)\ 0.662 + (1/na)\ 0.528 \qquad (7)$$

In order to reduce the total variance, one or more of the variance terms in eq 7 must be reduced. Since there is a different cost associated with reducing each variance component, one must decide the most cost-effective method to achieve a given level of variance for s^2_t. However, within-bottle variance is the largest source of variability (accounts for 42.9% of the total variability), and increasing laboratory sample size should be the first consideration.

Variances Associated with Measurements of OTA. Since OTA was measured in the same 5 g laboratory sample as AF, the same statistical analysis was applied to OTA sample test results as described above for AF. The total variance associated with measuring OTA in a 5 g laboratory sample was partitioned into bottle-to-bottle and combined within-bottle plus analytical variances for lots 1, 2, and 3 (**Table 2**). In addition, the analytical variance associated with the OTA analytical method is shown in **Table 2** for lot 3. Since the OTA concentrations among the three lots were similar, the variances for the three lots were averaged, and the results are shown in **Table 2**. Two sample test results from lot 2 and five sample test results from lot 3 were considered outliers and were not used in the statistical analysis. OTA values that fell outside the range defined by the lot mean ± 3 times the standard deviations were identified as outliers.

Table 1. Total, Bottle-to-Bottle, Within-Bottle, and Analytical Variances Associated When Sampling Ginger for Aflatoxins[a]

				variance components (proc mixed)			
lot	number of samples tested	average total aflatoxin concentration (ng/g)	median total aflatoxin concentration (ng/g)	total	bottle to bottle	within bottle and analytical	analytical
1	39	6.54	6.36	0.617	0.052	0.565	
2	80	8.26	7.96	2.966	1.010	1.956	
3	75	7.22	7.25	1.048	0.000	1.048	0.528
all lots averaged	194	7.34	7.19	1.544	0.354	1.190	0.528

[a] Note: One sample test result from lot 1 and five sample test results from lot 3 were considered outliers and were not used in the statistical analysis. AF values that fell outside the range defined by the lot mean $\pm$ 3 times the standard deviations were identified as outliers.

Table 2. Total, Bottle-to-Bottle, Within-Bottle, and Analytical Variances Associated When Sampling Ginger for Ochratoxin A[a]

				variance components (proc mixed)			
lot	number of samples tested	average total OTA concentration (ng/g)	median total OTA concentration (ng/g)	total	bottle to bottle	within bottle and analytical	analytical
1	40	1.96	1.86	0.161	0.000	0.161	
2	78	2.19	2.15	0.166	0.003	0.163	
3	75	1.65	1.45	0.355	0.041	0.314	0.020
all lots averaged	193	1.93	1.82	0.228	0.015	0.213	0.020

[a] Note: Two sample test results from lot 2 and five sample test results from lot 3 were considered outliers and were not used in the statistical analysis. OTA values that fell outside the range defined by the lot mean $\pm$ 3 times the standard deviations were identified as outliers.

The total, bottle-to-bottle and combined within-bottle plus analytical variances averaged across all three lots was 0.228, 0.015, and 0.213, respectively. If the analytical variance from lot 3 (0.020) is subtracted from the average combined within-bottle plus analytical variance (0.213), the within-bottle variance is 0.193 (assume that the analytical variance for lots 1 and 2 are the same as that for lot 3). The total variance (eq 1) is equal to the sum of bottle-to-bottle, within-bottle, and analytical variances or 0.228 (0.015 + 0.193 + 0.020). The bottle-to-bottle, within-bottle, and analytical variances account for 6.6, 84.6, and 8.8% of the total variance, respectively. The above variances are specific to using RPLC methods to measure OTA in powdered ginger with 5 g laboratory samples taken from a lot at 1.93 ng/g OTA.

The analytical variance (0.020) associated with measuring OTA in one aliquot can be used to predict the analytical variance for any number of aliquots, na, quantified for OTA. The analytical variance for any number of aliquots is

$$s^2_a = (1/na)\ 0.020 \qquad (8)$$

The analytical variance can be reduced by increasing the number of aliquots, na, quantified for OTA and averaging the results by the amount predicted from eq 8.

The within-bottle variance (0.193) associated with measuring OTA in a 5 g laboratory sample taken from an aggregate sample can be used to predict the within-bottle variance for any given laboratory sample size, tns, in grams.

$$s^2_{wib} = (5/tns)\ 0.193 \qquad (9)$$

The within-bottle variance can be reduced by increasing the laboratory sample size, tns, by the amount predicted from eq 9.

The bottle-to-bottle variance (s^2_{btb}) of 0.015 reflects additional variability over and above the within-bottle variance due to the heterogeneity of the OTA contamination from bottle to bottle in the lot. The bottle-to-bottle variance (s^2_{btb}) provides an indication of the heterogeneity of the OTA contamination from bottle to bottle in the lot and can be used to decide on how many bottles should be taken from the lot and combined to form

an aggregate sample. The bottle-to-bottle variance (s^2_{btb}) for any given aggregate sample size bns in number of bottles can be estimated from 0.015 for a single bottle of ginger.

$$s^2_{btb} = (1/bns)\ 0.015 \qquad (10)$$

The total variance associated with pooling ginger powder from bns bottles to form an aggregate sample, taking a laboratory sample of tns grams from the aggregate sample, and quantifying the OTA in any number of aliquots by the RPLC method can be determined from eqs 7, 8, and 9 (as shown in eq 1).

$$s^2_t = (1/bns)\ 0.015 + (5/tns)\ 0.193 + (1/na)\ 0.020 \qquad (11)$$

In order to reduce the total variance associated with the OTA test procedure, one or more of the variance terms in eq 11 must be reduced. Since there is a different cost associated with reducing each variance component, one must decide the most cost-effective method to achieve a given level of variance for s^2_t. However, within-bottle variance is the largest source of variability (accounts for 84.6% of the total variability), and increasing laboratory sample size should be the first consideration.

AF and OTA were found in three commercial lots of ginger sold in capsule form. The AF levels in all three lots were similar in magnitude and averaged 7.34 ng/g total AF, which was below the FDA action limit of 20 ng/g. The average OTA level of 1.93 ng/g was lower than the AF levels.

The total variance associated with measuring AF in powdered ginger is greater than measuring OTA when using the same laboratory sample size and analytical method. Since the variance associated with a mycotoxin test procedure increases with concentration (*11, 12*), it is not clear if the differences in variance are due to concentration differences or differences in the method of contamination by the two fungi that produce AF and OTA. However, when the total variance is converted into the coefficient of variation (*CV* or standard deviation relative to the mean concentration), *CV* is lower for AF (16.9%) than OTA (24.7%).

The total variability associated with measuring AF and OTA in powdered ginger was partitioned into bottle-to-bottle, within-bottle, and analytical variances. For both AF and OTA, the within-bottle variance associated with the 5 g laboratory sample size was the largest component of variability accounting for about 42.9 and 84.6% of the total variance, respectively; the analytical variance accounted for about 34.2 and 8.8% of the total variability, respectively; and the bottle-to-bottle variance accounted for about 22.9 and 6.6% of the total variance, respectively.

When measuring OTA, emphasis on error (variability) reduction should focus on using larger laboratory sample sizes, tns, since the within-bottle variance associated with a 5 g laboratory sample accounted for 84.6% of the total variance. The bottle-to-bottle variance (reflecting nonuniform distribution of contaminated ginger particles through out the lot) was only 6.6% of the total variance and can be minimized by pooling the contents of a relatively few bottles.

When measuring AF, error reduction should focus on all three variance components. The bottle-to-bottle, within-bottle, and analytical variances account for 22.9, 42.9, and 34.2% of the total variability, respectively. However, more emphasis should be given to the combined within-bottle and analytical variance that collectively accounts for 77.1% of the total variability.

Estimates of the bottle-to-bottle and combined within-bottle plus analytical variances differed among the three lots tested. One would expect the manufacturer of the powdered ginger to blend all lots in a similar manner. These variance differences from lot to lot are probably due to experimental error since it is difficult to get precise estimates of variance. To be conservative, it may be better to use the larger variance estimates associated with lot 2 for AF and lot 3 for OTA to make recommendations for the number of bottles for the aggregate sample, the number of 5 g laboratory samples, and the number of aliquots to use in their respective test procedures. From **Tables 1** and **2**, the variance models for AF (s^2_{tAF}) and OTA (s^2_{tO}) are shown in eqs 12 and 13, respectively.

$$s^2_{tAF} = (1/bns)\ 1.010 + (5/tns)\ 1.428 + (1/na)\ 0.528 \tag{12}$$

$$s^2_{tO} = (1/bns)\ 0.041 + (5/tns)\ 0.294 + (1/na)\ 0.020 \tag{13}$$

If both AF and OTA were measured in the same laboratory sample, variance reduction methods related to measuring AF (eq 11) would be more than adequate for OTA measurements.

ABBREVIATIONS USED

AF, Aflatoxins (sum of aflatoxins B_1, B_2, G_1, and G_2); OTA, ochratoxin A; RPLC, reversed phase high performance liquid chromatography.

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