

Sampling Techniques

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1. Introduction

It is important to be able to detect and quantify the mycotoxin concentration in food and feedstuffs destined for human and animal consumption. In research, regulatory, and quality assurance activities, correct decisions concerning the fate of commercial lots can only be made if mycotoxin test procedures are accurate and precise. However, it is difficult to estimate accurately and precisely the mycotoxin concentration in a large bulk lot because of the large variability associated with the mycotoxin test procedure (1–8). A mycotoxin test procedure is a complicated process and generally consists of 3 steps: (a) a sample is taken from the lot, (b) the sample is ground in a mill to reduce particle size, and a subsample is removed from the comminuted sample for extraction, and (c) the mycotoxin is extracted from the comminuted subsample and quantified. There have been several reviews published describing accepted procedures for sampling, sample preparation, and analysis for agricultural commodities (9–15). Even when using accepted procedures, there are errors (the term error will be used to denote variability) associated with each of the above steps of the mycotoxin test procedure. Because of these errors, the true mycotoxin concentration in the lot cannot be determined with 100 percent certainty by measuring the mycotoxin concentration in the sample taken from the lot.

In this chapter we will discuss the different sources of variability that are associated with testing agricultural commodities for mycotoxins. Specifically, we will concentrate on the testing of agricultural commodities for aflatoxin since most published literature is concerned with this mycotoxin. We will show how to reduce the variability of mycotoxin test results and how to design testing programs to determine the mycotoxin level of a contaminated lot as accurately and precisely as resources will permit.

Table 1
Distribution of Aflatoxin Test Results for Ten 5.4 kg Samples
from Each of Six Lots of Shelled Peanuts^{a,b}

Lot Number	Sample Test Result (ppb)										Mean (ppb)	SD ^c (ppb)	CV ^d (%)
1	0	0	0	0	2	4	8	14	28	43	10	15	150
2	0	0	0	0	3	13	19	41	43	69	19	24	126
3	0	6	6	8	10	50	60	62	66	130	40	42	105
4	5	12	56	66	70	92	98	132	141	164	84	53	63
5	18	50	53	72	82	108	112	127	182	191	100	56	56
6	29	37	41	71	95	117	168	174	183	197	111	66	59

^aFrom Whitaker et al. (1972).

^bAflatoxin test results are order by aflatoxin ppb.

^cSD = Standard Deviation.

^dCV = Coefficient of Variation = (SD/mean × 100).

2. Variability of Mycotoxin Test Procedures

Assuming accepted test procedures are used to estimate the mycotoxin concentration of a bulk lot, random variation still exists among replicate mycotoxin tests on the same bulk lot. For example, 10 replicated aflatoxin test results from each of 6 contaminated shelled peanut lots are shown in **Table 1 (16)**. Each test was made by (a) comminuting a 5.45 kg sample of peanut kernels in a subsampling mill developed by Dickens and Satterwhite (17,18), (b) extracting aflatoxins from a 280 g subsample with the AOAC Method II (BF method), and (c) quantifying the aflatoxins densitometrically using thin layer chromatography (TLC). The 10 aflatoxin test results from each lot are ranked from low to high to demonstrate several important characteristics about replicated aflatoxin test results taken from the same contaminated lot.

First, the wide range among replicated test results from the same lot reflects the large variability associated with estimating the true mycotoxin content of a bulk lot. In **Table 1**, the variability is described by both the standard deviation and the coefficients of variation (CV). The maximum test result can be four to five times the lot concentration (the average of the 10 test results is the best estimate of the lot concentration). Secondly, the amount of variation among the 10 test results appears to be a function of the lot concentration. As the lot concentration increases, the standard deviation among test results increases, but the standard deviation relative to the lot mean, as measured by the CV, decreases. Thirdly, the distribution of the 10 test results for each lot in **Table 1** are not always symmetrical about the lot concentration. The distributions are positively skewed, meaning that more than half of the sample test

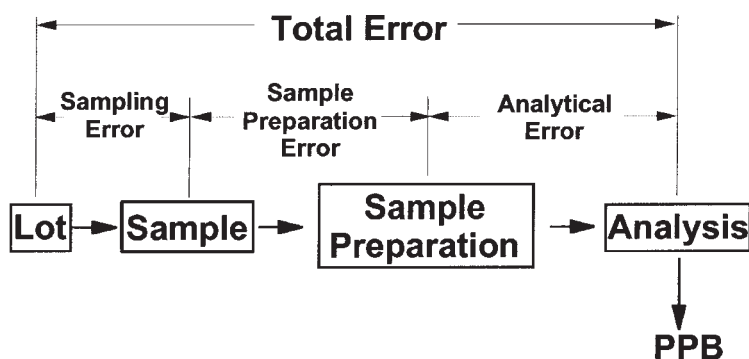


Fig. 1. Types of error associated with mycotoxin testing.

results are below the lot concentration. However, the distribution of sample test results becomes more symmetrical as the lot concentration increases. This skewness can be observed by counting the number of test results above and below the lot concentration in **Table 1**. If a single sample is tested from a contaminated lot, there is more than a 50% chance that the sample test result will be lower than the true lot concentration. The skewness is greater for small sample sizes and the distribution becomes more symmetrical as sample size increases (19).

The variability shown in **Table 1** is the sum of the variability associated with each step of the mycotoxin test procedure. As shown in **Fig. 1**, the total variability (using variance as the statistical measure of variability) associated with a mycotoxin test procedure is equal to the sum of the sampling, sample preparation, and analytical variances associated with each step of the mycotoxin test procedure.

$$VT = VS + VSS + VA \quad (1)$$

Examples of the magnitude of the variability associated with each step of a mycotoxin test procedure (**Eq. 1**) are given in the sections below.

3. Sampling Variability

There are two important aspects that can affect sampling variability. First is the sample selection procedure, and second is the distribution among contaminated particles within a lot. Generally, using proper sampling equipment and procedures can minimize any effect of sample selection, but only increasing sample size can reduce the effects of the distribution among contaminated particles within a lot on sampling variability. These two aspects affecting sampling variability are discussed below.

3.1. Sample Selection Methods

Procedures used to take a sample from a bulk lot are extremely important. Every individual item in the lot should have an equal chance of being chosen (called random sampling). Biases will be introduced by the sample selection methods if equipment and procedures used to select the sample prohibit or reduce the chances of any item in the lot from being chosen (20). If the lot has been blended thoroughly from the various material handling operations, then the contaminated particles are probably distributed uniformly throughout the lot. In this situation, it is probably not too important from what location in the lot the sample is drawn. However, if the product is contaminated because of moisture leaks or for other reasons, then the mycotoxin contaminated particles may be located in isolated pockets in the lot (21). If the sample is drawn from a single location, the contaminated particles may be missed or too many contaminated particles may be collected. Because contaminated particles may not be distributed uniformly throughout the lot, the sample should be an accumulation of small portions taken from many different locations throughout the lot (22,23). The accumulation of many small incremental portions is called a bulk sample. If the bulk sample is larger than desired, the bulk sample should be blended and subdivided until the desired sample size is achieved. The smallest size sampling unit used before the sample preparation step to estimate the lot mycotoxin concentration is often called the test sample. It is generally more difficult to obtain a representative (lack of bias) sample from a lot at rest (static lot) than from a moving stream of the product (dynamic lot).

3.1.1. Static Lots

Examples of static lots are commodities contained in storage bins, railcars, or many small containers such as sacks. When drawing a sample from a bulk container, a probing pattern should be developed so that product can be collected from different locations in the lot. An example of several probing patterns used by the Agricultural Marketing Service to collect samples from bulk peanut lots is shown in **Fig. 2**. The sampling probe should be long enough to reach the bottom of the container when possible. As a general rule, several hundred grams of sample should be drawn per 1000 kg of commodity.

When sampling a static lot in separate containers such as sacks, the sample should be taken from many containers dispersed throughout the lot. When storing sacks in a storage facility, access lanes should be left in order to gain access to interior sacks. The recommended number of sacks sampled can vary from one-fourth of the sacks in small lots to the square root of the number of sacks for large lots (24).

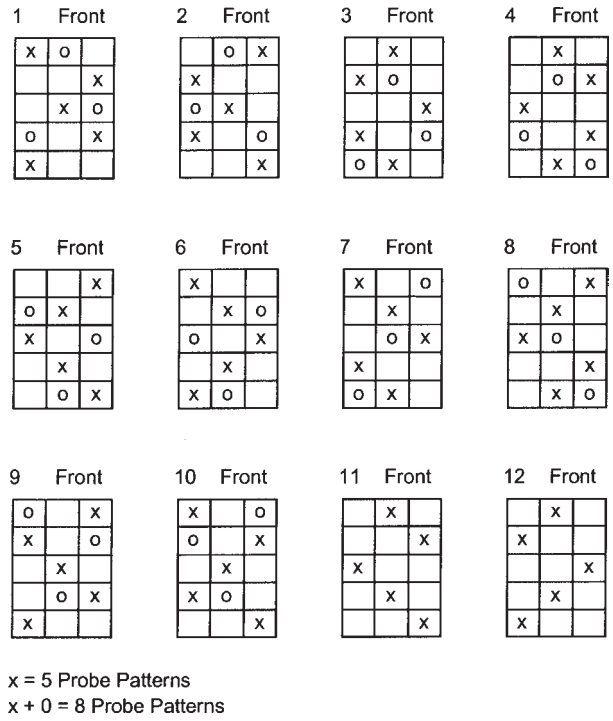


Fig. 2. Example of several five- and eight-probe patterns used by the USDA to sample farmers stock peanuts for grade and support price.

If the lot is in a container where access is limited, the sample should be drawn when the product is either being removed from or being placed into the container. If the accumulated bulk sample is larger than required, the bulk sample should be thoroughly blended and reduced to the required test sample size with a suitable device such as a riffle divider.

3.1.2. Dynamic Lots

True random sampling can be more nearly achieved when selecting a bulk sample from a moving stream as the product is transferred (i.e., conveyor belt) from one location to another. When sampling from a moving stream, small increments of product should be taken along the entire length of the moving stream; composite all the increments of product to obtain a bulk sample; if the bulk sample is larger than required, then blend and subdivide the bulk sample to obtain the desired size test sample.

Automatic sampling equipment such as cross-cut samplers (**Fig. 3**) are commercially available with timers that automatically pass a diverter cup through

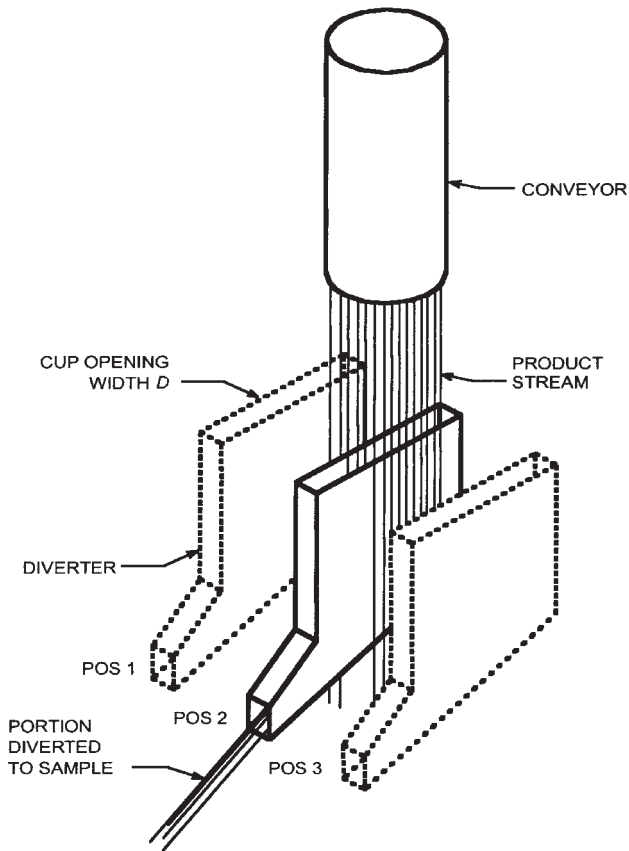


Fig. 3. Automatic cross-cut sampler.

the moving stream at predetermined and uniform intervals. When automatic equipment is not available, a person can be assigned to manually pass a cup through the stream at periodic intervals to collect the bulk sample. Whether using automatic or manual methods, small increments of product should be collected and composited at frequent and uniform intervals throughout the entire time product flows past the sampling point.

Cross-cut samplers should be installed in the following manner: (1) the plane of the opening of the sampling cup should be perpendicular to the direction of flow; (2) the sampling cup should pass through the entire cross sectional area of the stream; and (3) the opening of the sampling cup should be wide enough to accept all items of interest in the lot. As a general rule, the width of the sampling cup opening should be two to three times the largest dimensions of the items in the lot.

The size of the bulk sample, S in kg, taken from a lot by a cross-cut sampler is:

$$S = (D) (L) / (T) (V) \quad (2)$$

where D is the width of the sampling cup opening in cm, L is the lot size in kg, T is interval or time between cup movement through the stream in seconds, and V is cup velocity in cm/s.

Eq. 2 can also be used to compute other terms of interest such as the time between cuts, T . For example, the required time, T , between cuts of the sampling cup to obtain a 10 kg sample from a 30,000 kg lot where the sampling cup width is 5.08 cm (2 inches), and the cup velocity through the stream 30 cm/s. Solving for T in **Eq. 2**, $T = (5.08 \text{ cm} \times 30,000 \text{ kg}) / (10 \text{ kg} \times 30 \text{ cm/s}) = 508 \text{ s}$.

If the lot is moving at 1000 kg/min, the entire lot will pass through the sampler in 30 min and only three or four cuts will be made by the cup through the lot. This may be considered too infrequent, in that too much product passes through the sampler between the time the cup cuts through the stream. The interaction among the variables in **Eq. 2** need to be fully understood in terms of the amount of sample accumulated and the frequency of taking the product.

3.2. Contamination Distribution

Studies by researchers on a wide variety of agricultural products (peanuts, cottonseed, shelled corn, and pistachio nuts) indicate that, especially for small sample sizes, the sampling step is usually the largest source of variability associated with the mycotoxin test procedure (1–8). Accepted sample selection equipment and procedures were used to minimize any effects due to sample selection methods. Sampling error is large because of the extreme distribution among contaminated particles within a lot. Aflatoxin studies on peanuts suggest about 0.1% of the kernels in the lot are contaminated and the concentration on a single kernel may be extremely high. Cucullu et al. (25,26) reported aflatoxin concentrations in excess of 1,000,000 ng/g (parts per billion, ppb) for individual peanut kernels and 5,000,000 ng/g for cottonseed. Shotwell et al. (27) reported finding over 400,000 ng/g of aflatoxin in a corn kernel.

Because of this extreme range in aflatoxin concentrations among a few contaminated kernels in a lot, variation among replicated sample test results tends to be large. As an example, the sampling variance associated with testing shelled corn, VSs, was estimated empirically (3) and is shown in **Eq. 3**.

$$VS_s = 3.95M/WS_s \quad (3)$$

where M is the aflatoxin concentration in the lot in nanograms of total aflatoxin per g of corn (ng/g) or parts per billion (ppb), WS_s is the mass of shelled corn in the sample in kg (kernel count per gram was 3.0). From **Eq. 3** one can see that the sampling variance is a function of the lot concentration M and

sample size WSSs. The sampling variance associated with taking a 0.91 kg (2 lb) sample from a lot of shelled corn at 20 ppb is 86.9. The coefficient of variation is 47%.

Researchers have developed equations to describe the sampling variance for several commodities and several mycotoxins (1–8). The equations are specific for the type of mycotoxin and the type of product studied.

4. Sample Preparation Variability

Once the test sample has been taken from the lot, the sample must be prepared for aflatoxin extraction. Since it is not practical to extract the mycotoxin from a large test sample, the mycotoxin is usually extracted from a much smaller portion of product (subsample) taken from the test sample. If the commodity is a granular product such as shelled corn, it is essential that the entire test sample be comminuted in a suitable mill before a subsample is removed from the test sample (9). Removing a subsample of whole seed from the test sample before the comminution process would eliminate the benefits associated with the larger size sample of granular product. After the sample has been comminuted, a subsample is removed for mycotoxin extraction. It is assumed that the distribution of contaminated particles in the comminuted sample is similar to the distribution among contaminated kernels found in the lot. As a result, there is also variability among replicated subsamples taken from the same test sample. However, the sample preparation variance is not as large as the sampling variance due to the large number of comminuted particles in the subsample. An example of sample preparation variance for aflatoxin and shelled corn, VSSs, is shown below in Eq. 4 (3).

$$\text{VSSs} = 0.0125M/\text{WSSs} \quad (4)$$

where M is the aflatoxin concentration in the comminuted test sample in ppb, and WSSs is the mass in kg of comminuted shelled corn in the subsample. The variance in Eq. 4 also reflects a particle size that will pass through a number 20 screen. From Eq. 4, it can be seen that the sample preparation variance is also a function of the aflatoxin concentration in the sample and the subsample size. The sample preparation variance associated with a 0.05 kg subsample taken from a sample at 20 ppb is 5.0 and the CV is 11%.

Researchers have developed equations to describe the sample preparation variance for several commodities, type mills, and mycotoxins (1–8,28). The equations are specific for the type mycotoxin, type mill, and the type product used in the study. The type mill effects the particle size distribution. If the average particle size decreases (number of particles per unit mass increases), then the subsampling variance for a given size subsample decreases.

5. Analytical Variability

Once the subsample is removed from the comminuted test sample, the mycotoxin is extracted. Analytical methods usually involve several steps such as solvent extraction, centrifugation, drying, dilution, and quantification (10). As a result, there can be considerable variation among replicated analyses on the same subsample extract. The analytical variance, V_{Abf} , associated with AOAC method II extraction and clean-up procedures along with TLC and densitometric quantification techniques to measure aflatoxin in peanuts (BF method) is given by Eq. 5 (1).

$$V_{Abf} = 0.064M^{1.93}/N_{bf} \quad (5)$$

where M is the aflatoxin concentration (ppb) in the subsample, and N_{bf} is the number of aliquots quantified by TLC methods. For example, at 20 ng/g, the variance and CV associated with the BF method is 20.9 and 22.8%, respectively. Studies on the BF method (30) indicate that the thin layer chromatography quantification step is the major source of variability in the analytical process associated with analyzing peanuts for aflatoxin.

If extraction and cleanup contribute only a small portion of the total analytical variance, then the immunoassay and high performance liquid chromatography (HPLC) type analytical methods should have lower variances than methods that use TLC quantification techniques. Hagler and Whitaker (31), and Dorner and Cole (32) independently measured the analytical variance associated with HPLC type methods to measure aflatoxin in peanuts. Even though Hagler and Dorner used slightly different extraction and cleanup procedures (31–33), both obtained almost identical results. The relationship between variance and aflatoxin concentration of Hagler's study for HPLC are given below.

$$V_{Ah} = 0.0048M^{1.75}/N_h \quad (6)$$

where M is the aflatoxin concentration in the subsample and N_h is the number of aliquots quantified by the HPLC procedure. At 20 ng/g, the variance and CV associated with the HPLC method is 0.9 and 4.8%, respectively. A CV of 4.8% associated with HPLC is much lower than the 22.8% associated with the BF method using TLC quantification techniques.

Immunoassay techniques are a more recent analytical development to measure mycotoxins in agricultural commodities such as peanuts, corn, and cottonseed. Food and feed industries, researchers, and regulatory agencies have studied the variability associated with immunoassay-type analytical methods (34,35; Whitaker, unpublished data, 1991). The variability one might expect using an immunoassay-type analytical method to quantify aflatoxin in peanut products is given below.

$$VA_i = 0.013M^{1.57}/N_i \quad (7)$$

where M is the aflatoxin concentration in the subsample and N_i is the number of aliquots quantified by the immunoassay procedure. **Eq. 7** reflects the pooling of variance data from corn, cottonseed, and peanuts. From **Eq. 7**, the variance associated with quantifying the aflatoxin in a subsample at 20 ppb using an immunoassay method is 1.9, and the CV is 7%. The variability associated with immunoassay type methods appears to be less than TLC methods and more than HPLC methods.

All of the analytical variance information described above reflects results from single laboratories and do not reflect among laboratory variances. As a result, some laboratories may have higher or lower variances than those reported in **Eq. 5**, **6**, and **7**. Among laboratory variance is about double the within laboratory variance (**36**).

6. Reducing Variability of Test Procedure

The only way to achieve a more precise estimate of the true lot concentration is to reduce the total variability or the variability associated with each step of the mycotoxin test procedure. The sampling variability can be reduced by increasing the size of the sample. The sample preparation variability can be reduced either by increasing the size of the subsample and/or by increasing the degree of comminution (increasing the number of particles per unit mass in the subsample). The analytical variance can be reduced by either increasing the number of aliquots quantified by the analytical method and/or using more precise quantification methods (using HPLC instead of TLC). If the variability associated with one or more of these steps can be reduced, then the total variability associated with a mycotoxin test result can be reduced.

For example, the expected total variance associated with testing a shelled corn lot at 20 ppb when using a 0.91 kg sample, taking a 50 g subsample from a comminuted sample, and using an immunoassay analytical method for quantification can be estimated by summing the variances (equation 1) calculated using **Eqs. 3**, **4**, and **7**.

$$VT = 86.9 + 5 + 1.9 = 93.8 \quad (8)$$

The variance, standard deviation, and CV associated with the total aflatoxin test procedure described above is 93.8, 9.7, and 48.4%, respectively. The sampling, subsampling, and analytical variances account for 92.6, 5.3, and 2.1% of the total testing variance, respectively. The major variance component is sampling which accounts for 92.6% of the total testing variation. It appears that the best use of resources to reduce the total variance of the test procedure would be to increase sample size. Increasing the sample size by a factor of five from 0.91

Table 2
Range of Aflatoxin Estimates for 95% Confidence Limits
When Testing a Contaminated Lot of Shelled Corn
with 20 ppb Using Different Sample Sizes.

Sample Size (kg)	Standard Deviation ^a (ppb)	Low ^b (ppb)	High ^c (ppb)
1	9.2	2.0	38.0
2	6.8	6.7	33.3
4	5.2	9.8	30.2
8	4.1	12.0	28.0
16	3.4	13.3	26.7
32	3.1	13.9	26.1

^aStandard deviation reflects sample size shown in table plus a 50 g subsample that will pass a #20 screen and immunoassay analytical method. Sample preparation plus analytical standard deviation = 2.6 and is constant for all sample sizes.

^bLow = 20 - 1.96(standard deviation)

^cHigh = 20 + 1.96(standard deviation)

to 4.54 kg will cut the sampling variance in **Eq. 8** by a factor of five to 17.4. The total variance with the 4.45 kg sample now becomes (**Eq. 9**):

$$VT = 17.4 + 5.0 + 1.9 = 24.3 \tag{9}$$

The variance, standard deviation, and CV associated with the total testing procedure has been reduced to 24.3, 4.9, and 24.6%, respectively.

The range of mycotoxin test results associated with any size sample and subsample, and number of analyses can be estimated from the standard deviation SD (square root of the total variance). Approximately ninety-five percent of all test results will fall between a low of $(M - 1.96*SD)$ and a high of $(M + 1.96*SD)$. The two expressions are only valid for a normal distribution where test results are symmetrical about the mean. The distribution among aflatoxin test results is usually skewed, but will approach a symmetrical distribution as the sample size becomes large. The effect of increasing sample size on the range of test results when testing a contaminated lot of shelled corn that has 20 ppb aflatoxin is shown in **Table 2**. We can see that the range doesn't decrease at a constant rate as sample size increases. For example, doubling sample size has a greater effect on decreasing the range at small sample sizes than at large sample sizes. This characteristic suggests that increasing sample size beyond a certain point may not be the best use of resources and that increasing subsample

size or number of analyses may be a better use of resources in reducing the range of test results once sample size has become significantly large.

As indicated above, there are methods other than increasing sample size to reduce the total variance associated with testing a commodity for a mycotoxin. Different costs are associated with each step of the mycotoxin test procedure, and careful study is required to determine the test procedure that will provide the lowest variance for a given cost. The optimum balance in sample size, degree of comminution, subsample size, number and type of analysis will vary with the costs involved with each step of the testing procedure. In general, the costs of properly designed mycotoxin test procedures will increase as the total variance is reduced.

7. Conclusions

Because of the variability associated with a mycotoxin test procedure, it is difficult to determine with 100% certainty the true concentration of a bulk lot. Even when the sample is correctly selected, there will be variability associated with the mycotoxin test procedure. The variance associated with a mycotoxin test procedure is the sum of sampling, sample preparation, and analytical variances. For small sample sizes, sampling is usually the largest source of variability. The variability associated with a mycotoxin test procedure can be reduced by increasing sample size, the degree of sample comminution, subsample size, and the number of aliquots quantified.

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Mycotoxin Protocols

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2001, XII, 244 p., Hardcover

ISBN: 978-0-89603-623-9

A product of Humana Press