QUALITY ASSURANCE AND CONTROL (Section 10 in MI's Fortification Handbook)

This section provides information and guidelines on mill quality assurance (QA) and quality control (QC) related to the fortification of wheat flour and maize meal. It is not meant to be a comprehensive treatise on the subject, which has been well covered in other publications (FAO, 1995) (Nalubola and Nestel 2000; Nestel, Nalubola et al. 2002). Rather, it gives only the key aspects of a QA/QC programs related to cereal fortification, so as to help mills just starting to fortify flour, premix manufacturers and those applying for grants in cereal fortification better understand the nature and requirements of such programs.

There is a tendency in other materials to make this subject highly complex and involved, giving the mistaken impression that considerable cost and time will be needed for a QA/QC program on fortification. The goal here is provide a simplified version of the basic or minimal actions needed for a mill to assure conformance to fortification specifications and regulations. This, fortunately, does not require a large investment in equipment and personnel. The time and effort a mill need spend to effectively control fortification can be quite small.

While flour fortification does not have the same stringent requirements as the manufacture of pharmaceuticals or infant foods, it does require additional QA/QC measures over what the mill is accustomed to with regular, non fortified flour. Millers who fortify flour need to accept and practice these additional QA/QC requirements, recognizing that they will come under closer scrutiny by both their customers and government regulator to make sure that the product is properly fortified.

There are other aspects of fortification QC and testing that are covered in other sections of this manual. Appendix I provides analytical methods that can be used to test for micronutrients in flour. Section 11 on Regulations and Enforcement discusses government testing, monitoring and auditing of mills to see if they are properly fortifying. Special issues related to QA/QC in small mills are discussed in Section 9 while testing related to surveillance of nutrient intakes and effectiveness of fortification programs is covered in Section 14.

Definition of terms and abbreviations

QA/QC has a rich lexicon of special terms and abbreviations. Some of these are defined in the Appendix, but a few deserve special attention.

quality: The composite of material attributes, including performance features and characteristics of a production or service, needed to satisfy a customer's given need.

quality assurance (QA): A planned and systematic pattern of all actions necessary to provide confidence that adequate technical requirements are established, that products and services conform to established technical requirements, and that satisfactory performance is achieved.

quality control (QC): The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it adheres to specifications that meet the needs of its users.

total quality program: A program which is developed, planned, and managed to carry out, cost-effectively, all efforts to effect the quality of material and services from concept through validation, full-scale development, production, deployment, and disposal.

quality audit:. A systematic examination of the acts and decisions with respects to quality in order to independently verify or evaluate the operational requirements of the quality program or the conformance to specifications of a product .

total quality systems audit (TQSA): A comprehensive audit designed to ensure the quality of products provided in U.S. domestic and export food assistance programs.

Indicator nutrient: The use of a single nutrient in flour as an index of all the micronutrients added by a fortification premix. Given that the indicator nutrient is within specification, it follows that all the other micronutrients should be as well providing that the fortification premix is correct.

MSDS: Material Safety Data Sheets.
GMPs: A recognized set of *Good Manufacturing Practices*.
SOP: A S*tandard Operating Procedure* adopted for repetitive use when performing a specific action.
ISO 9000: An internationally recognized set of standards for qualification of global quality assurance and quality control standards. Adherence is accomplished through an application process for ISO 9000 certification in company standards for inspecting production processes, updating records, maintaining equipment, training employees and handling customer relations.
HACCP stands for *Hazard Analysis and Critical Control Point*, a method used to insure food safety by identifying potentially unsafe links in the food processing chain.
CV or COV: *Coefficient of Variation* is a statistical term used to describe the amount of variation within a set of measurements for a particular test. It is calculated by taking the variation of a set of data as a percentage of the mean.
control chart: A visual depiction of analytical results over time.
spot or grab samples: A sample taken at a single point in the process.

In simple terms, QA is the plan on how quality is to be continually achieved and QC is the testing or implementation of that plan.

Objectives of QA/QC

There are a number of reasons for having a QA/QC program on flour fortification:

1. To conform to government regulations or customer specifications.
2. To control costs. That is, not add more premix than needed to meet requirements.
3. To assure product safety.
4. To satisfy customer expectations.
5. To avoid regulatory action or bad publicity for non conformance.

To meet these objectives requires that the correct fortification premix is being uniformly added to the correct flour or meal in adequate amounts to meet the minimum standards and not in excessive amounts as to cause safety, product quality or cost problems. These objectives can be accomplished with standard production and inventory controls along with good manufacturing practices.

QA/QC in different size mills

The type and extent of QA/QC that a mill exerts over fortification will differ greatly depending on the size of the mill. Table 1.1 in the Introduction lists three categories of mills based on size: large (>3 MT/hr), medium (1-3 MT/hr) and small (<1 MT/hr).

Very large mills (> 8 MT/hr) and many large mills (8 to 3 MT/hr) have an active QA/QC program that includes the following:

- A QC manager or employee with QC responsibilities.
- A laboratory or designated space for testing wheat and flour.
- Flour and wheat testing equipment. These tests typically include protein or gluten, moisture or water content, ash content and/or color, amylase enzyme activity (falling number) and dough properties. Some labs also bake bread or prepare other baked products from the flour or meal as a part of their overall quality program.
- A written quality assurance plan that includes sampling procedures and a QC testing schedule.

The larger the mill, the more likely it is to have these capabilities. However, the smaller the mill, the less able it is to afford them or to have a need for them. For large mills the additional QA/QC required by fortification will fit in nicely to their current program, but for medium and small mills, it may necessitate adopting a new and unfamiliar set of procedures. In either case, mill personnel will have to be trained in this component of fortification since it involves unfamiliar procedures and tests.

Fortification premix control

Fortification is always accomplished at the flour mill by adding a *premix* of the vitamins and minerals required under the standards. The first step in quality assurance of flour fortification is to have a well-made premix. The formulation and dilution of the premix is normally not covered by regulations[1] but is agreed to by the mill and premix manufacturer so as to provide the most practical product that will meet the standards for each mill.

All reputable premix manufacturers provide Certificates of Analysis (COAs) for all micronutrients on each lot of premix (a lot or batch being the amount of premix that is blended together at one time). COAs should be kept filed at each mill and be made available to government or customer inspection if asked. The analytical methods used to test for the active ingredients in the premix should be documented. These methods are not those used to test for the levels of micronutrients in fortified flour. Distinct methods are required because of large differences in nutrient concentrations and presence or absence of interfering substances.

The mill should keep good inventory control of the fortification premix. This includes records on:

- Quantities, reception dates and lot numbers of all premix received in the plant.
- Regular (e.g. monthly) inventories of premix on hand.
- Quantity of fortified flour produced during the same period.

Premix suppliers should provide technical information or fact sheets on each type of premix. These sheets should include information on premix composition, application rate, packaging, handling and storage. Many countries will also want an MSDS on the premix. Current information and MSDS sheets from all premix suppliers used should be on file at the mill.

Each container of premix should have a label indicating the type of product (code number or name), lot number, kosher or halal certification if required and application rate.

Premix manufactures should inform the mills what the acceptable shelf life is of their product. Most fortification premixes are good for a couple years when stored in sealed, unopened containers. Premixes containing vitamin A, however, have a shorter shelf life. If there is a question whether a premix is still good after extended storage, a mill should contact the manufacturer providing them the lot number and storage conditions. Mills should not use premix that has sat around in an opened container for months, or exposed to fire or water, nor should they expect the supplier to take back or give credit for such materials. Once a premix has been received in good condition at the mill, the miller is responsible for storing it. A *first in, first out* (FIFO) inventory control should be employed.

All these records should be available to inspectors. One of the simplest ways to see whether a mill has been fortifying flour is to compare the quantities of premix used to the amount of fortified flour produced. This is not a surefire method, since mills can keep false records, but it has proven a very effective means of ensuring compliance when combined with other checks discussed below.

Feeder calibration and maintenance

Most flour mills fortify flour continuously using an ingredient feeder or dosifier to meter the premix into the flour as it flows through the mill. The alternative is to use a batch mixing system where a set quantity of the premix is weighed out and blended into a set quantity of flour. In continuous systems the fortification feeder must be adjusted to add the correct amount of premix based on the *flour flow rate* and the addition rate specified for that premix. Mills have to know, or determine, the flour flow rate, typically in Metric Tons per hour, kg/min or some similar unit, in order to make this calculation.

The premix addition rate should be checked daily by running a "check feed rate" on the feeder, and changed according to any change in the flour flow rate. This is done simply by putting a plate or cup under the discharge spout of the feeder for 60 seconds and weighting the amount of premix collected to an accuracy of 0.1 grams. A standard lab balance or electronic scale can be employed for this purpose.

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[1] An exception to this is South Africa, where regulations cover the premix as well.

Mill QA/QC

The standard procedures utilized by large flour mills to insure that flour is properly fortified includes:

- Use of a quality feeder whose delivery of premix can be tied into the flow rate of the flour and which will stop when the flour flow stops.
- Regular checking of feed rates on feeder. Once per 8 hr. shift is recommended.
- Regular iron spot tests on flour. Once per 8 hr. shift is recommended
- Checking of premix usage against production of flour that should have been fortified. This typically should be done on a monthly basis.
- Optional quantitative testing of iron on a weekly or monthly basis. An outside lab is recommended.
- Optional quantitative testing of all fortification components in a composite sample on a monthly or quarterly basis. This must be done by an outside lab. Some premix companies offer this service to their customers at no charge.

Record keeping

Good record keeping is the key to mill QA/QC in flour fortification. Each mill should have a written plan of what records they want kept, how the data is to be entered, who should collect them and where they are to be kept. Records of all of the above activities should be kept by the mill and made available to government or flour customer inspection or audits when requested.

Analytical testing

Flour samples should be tested to verify that it has been properly fortified. Three types of testing are possible:

- **Qualitative tests** show simply the presence or absence of an added micronutrient. An example is the black light (uv light) test for riboflavin. This type of test is used to see if a flour has been fortified or not.
- **Semiquantitative tests** give a rough indication of the level of an added nutrient. Examples are the iron spot test and a color test recently developed for vitamin A in flour. This type of test tells whether the level added is low, normal or high.
- **Quantitative tests** give an actual value for the level of micronutrient in the sample. Unlike the other two types, which respond to added micronutrients, quantitative tests generally measure total content or both the natural and added levels, but some test methods have been developed to show only the added.

Sampling

The way flour samples are obtained and handled is an important component of the analytical procedure, particularly when submitted for quantitative testing, and should be well documented in the QA plan. The best place to sample is at or directly prior to packout, since this represents the final mill product. Composite samples are preferred to spot samples for quantitative testing, but spot samples are acceptable for the iron spot test.

A good composite samples would have 7 spot samples taken over an 8 hour period. One example is to place 50 gram spot samples into an opaque container holding at least 700 grams of flour. When all 7 samples are collected the composite is mixed by inverting and shaking the container. A single sample is then taken for testing. It is always good practice to retain all or part of the original sample in case something goes wrong with the other sample. A minimum composite sample consists of 3 spot samples taken over a hour period.

Samples of fortified flour or meal must not be exposed to direct sunlight or strong indoor lighting. They should be well labeled with the name of the mill, date, type of flour and whether or not they are fortified (unfortified samples are often collected to establish base line natural levels). If regular samples are to be collected, it is also a good idea to number them consecutively. This sample data should be entered into a mill sample record book. There is no point in taking and testing samples unless they are well documented.

When a mill first starts fortification they may want to have a number of spot samples of fortified protect tested along with some unfortified samples to gain a better picture of their capacity to fortify correctly along with the level and variation they can expect. For example, they may take a spot sample every 6 hours over a 3 day period and have them tested for iron as the indicator nutrient. They may also want to take a couple composite samples and have those tested for all the added micronutrients.

But once fortification has commenced, it is not practical to run that many samples on a regular basis. Medium and small size mills may wish to send in a composite sample a couple times a year, large mills may want to do it every month or quarter, while very large mills may wish to do that more often. Such samples are generally tested for all added micronutrients but testing one or two indicator nutrients may be sufficient.

When an official inspector is collecting samples it is recommenced that the sample taken be separated into three separate samples, one for the test; one for the mill and a third one in case the other two do not agree to their results.

Iron spot test

The iron spot test (AACC method 40-40, Iron- Qualitative Method) (see Appendix I) is universally used by millers to check whether flour has been properly fortified. This is a simple, inexpensive, semiquantitative procedure that should be run on fortified flour on a regular basis, typically every 2 or 4 hours for a large mill. It should be run at least once every eight hour shift at a minimum for flour sampled during production.. It may also be run on flour sampled in the warehouse to verify it has been properly labeled as being fortified.

The test will indicate whether the flour is under-fortified or over-fortified to a sufficient degree for the mill to take corrective action. The iron spot test can also indicate whether the iron being added is reduced, elemental iron, or a salt (ferrous sulfate or ferrous fumarate), in case the mill is adding both types. While this test is only for iron, it can be assumed that if the iron is correct, the other added micronutrients (i.e. folic acid) will be correct as well since they are added as a single premix.

Quantitative testing

While much can be accomplished through simple record keeping, feed rate checks and iron spot tests, there are times when it will be useful if not necessary to do quantitative testing of the micronutrients in flour. There are quantitative analytical procedures available for all the micronutrients that might be added to flour, many of which are provided in Appendix I. These differ greatly in complexity, analytical error (CV), type of equipment and skill needed to run them, as well as cost.

It is strongly recommended that quantitative tests on flour <u>not</u> be run by the flour mill. Rather they should be run by an outside laboratory that is familiar with the methodology and can run the test on a regular basis. The reasons for this are:

- The cost of the equipment and trained personnel needed to run many of these tests are beyond the resources of most milling companies.
- These tests need to be run on a regular basis if accurate results are to be obtained. Mills would only run these on an occasional basis and would never become very skilled in them.
- Mills would not save money by running these tests themselves over sending them out to an outside lab, particularly if they can have it done at no charge by their premix supplier.
- Flour mills in countries that have been fortifying flour for many decades, such as those in the United States and Canada, do not run these tests themselves, which shows that internal testing is not necessary for a successful QA/QC program.
- Quantitative results are normally used to show to a customer or government inspector that a product has been correctly fortified. Results from an outside laboratory have greater credibility than those produced internally.
- Suitable outside testing facilities are generally available for most countries.

There are some rare instances where a mill still believes they need a quantitative testing capacity, despite all the above reasons not to. In that case the spectrophotmetric test (AACC 40-41B) for iron, given in

Appendix I, should be the first method considered. This is a fairly inexpensive method and iron is the most likely nutrient to be used as an *indicator* of adequate fortification in government control. This method requires an ashing oven (generally available in most large mills) and a spectrophotometer or colorimeter. There are a couple of different methods depending on the iron color reagent employed. The wavelength at which the absorption is read depends on the particular reagent employed.

The most difficult part of this procedure is the extraction of the iron from the flour ash. This is prone to contamination and requires boiling acids that must be conducted under a good laboratory exhaust hood. This is a very corrosive process best done with non metal hoods and equipment. Natural iron content in flour can vary greatly and is harder to extract than added iron. Once the extract has been prepared it is a fairly simple procedure to read the iron content with a spectrophotometer.

Since folic acid is now routinely included in flour fortification, some mills may wish to test it along with iron. Others may wish to test for vitamin A when that is being added. Unfortunately, tests for both of these vitamins are quite difficult and are best left to outside labs.

Lab flour standards

It is recommended that the mill make and maintain a set of fortified flour standards for use in both the iron spot tests and any quantitative analyses. A separate set of standards is needed for each type of flour or meal being fortified. These should be kept in a sealed container preferably in a refrigerator and made fresh every couple months or when they are used up.

As an example, if a particular type of flour is being fortified with a premix added at 150 grams/metric ton (0.15 grams/kg), make up the following 4 samples:

1. Unfortified flour A
2. A + 0.10 grams/kg premix
3. A + 0.15 grams/kg premix
4. A + 0.20 grams/kg premix

It is recommended that these standard flours be run along with an unknown sample. Sample #3 with the correct fortification addition should be run every time a quantitative test is performed. The results on this known set of flour samples will help the mill correctly read the iron spot test, particularly during the first couple months that it is performed. In quantitative testing repeated assays on a single known sample will help verify its accuracy of the method, quantify its precision (CV) and help correct for any bias that may exist.

Utilizing nutrient assay data

One of the more misunderstood and contentious aspects of flour fortification is how to interpret and utilize analytical results from quantitative tests, whether performed by the mill or by an outside laboratory. Ideally, one would like to see all the results fall just above the minimum fortification standards or within the minimum-maximum range if there is one. Obviously, that does not always happen.

Some millers even question the value of quantitative testing of micronutrient content considering their high analytical error. They feel that the variability of premix addition in a well run mill is far less than the variability or assay error of the analytical testing. However, government regulatory control and some large flour customers require quantitative testing to confirm compliance to standards. Because there is pressure on the mills to consistently show good results, some mills or laboratories may want to “fudge” or “dry lab” the micronutrient testing. One indication that this might be happening is a CV under 8%, which is not generally achievable. Having a suspect laboratory run blind samples that are known to out of specification is one way to check whether they are reporting actual assay values.

Both mills and government regulators need to understand that there will be considerable variation in the results of quantitative tests of added micronutrients, with CVs of 10 to 20% common, depending on the type of nutrient being tested. A CV higher than 20% indicates a possible problem, either in the production or analytical testing, which needs to be corrected.

Very little can be achieved by having just one or two test results on spot samples. Rather, one needs a collection of results over time, preferably on composite samples, in order to really assess how well a mill is doing in fortifying flour. A mill is doing a good job in fortification if the average of 7 or more tests is above the minimum standard and the CV is less than 16%. It may be that one or two of the results are very low or very high. It the mill can assign a cause to those occurrences, such as a flour choke or the feeder to stop working knowing to have occurred, those results can be excluded from the average.

Following are some possible situations where corrective action is indicated:

Situation	***Possible causes***	***Possible actions***
A single indicator nutrient or all of the nutrients tested are consistently 80% or less of the minimum standard or greater than 140% of minimum.	Wrong feed rate, Wrong premix, High analytical error	Increase feed rate Change premix, Check for bias on test method
One nutrient is consistently high or low but the others are okay.	Wrong premix formulation, High analytical bias on the problem nutrient.	Reformulate premix, Check accuracy of method
High variability (CV) of all nutrients but their relationship to each other stays relatively constant.	This is indicative of high process variability rather than analytical error.	Check for causes of process variability such as erratic flour flow rate, high frequency of chokes and pneumatic separation.
Low thiamin but other nutrients okay.	Thiaminase in maize meal, High pH (>8)	Use different source of maize.
Low riboflavin or folic acid but other nutrients okay.	Exposure to ultraviolet light.	Protect flour and samples from light exposure.
Low iron but other nutrients okay	Low natural iron levels, Magnetic separation, Pneumatic separation	Reformulate premix, Use non magnetic iron, Add premix at different point.
Low vitamin A but other nutrients okay.	Vitamin A separation, Poor quality vitamin A.	Reconfigure point of addition, Use premix with better quality vitamin A.

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