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Mixer performance, cross-contamination testing examined

Registration of feed mills and implementation of "process controls" like mixer performance and cross-contamination testing have been on a national basis in the EU, with feed manufacturers in many countries relying heavily on guidance from industry-sponsored feed research institutes, but testing procedures are still being developed and are not harmonized at this time.

TABLE

Qualitative performance of tracers

Criterion	Fine colored iron	Cobalt	Manganese, low level	Coarse colored iron	Methyl violet	Manganese, high level
Premix uniformity	Inconclusive	Complete	Complete	Complete	Inconclusive	N.A.
Recovery-mash	Excellent	Excellent	Good	Excellent-good	Moderate-high	Excellent
Recovery-pellets	Moderate-poor	Excellent	Good-excellent	Poor (high)	Excellent	Excellent
Mixing uniformity	Complete	Complete	Complete	Complete	Complete	Complete
Carryover mash	Good	Good	(Far) too high	Good	(Far) too high	Good
Carryover pellets	Good	Good	(Far) too high	Good	Too high	Good

By DAVID A. EISENBERG

Reacting to several crises, the European Commission published a January 2000 White Paper on Food Safety that outlined a new concept in farm-to-table legislation and listed 80 legal initiatives, many of which recast regulation to a new "precautionary" hazard analysis and critical control points (HACCP) approach.

The actions are aimed at getting the food and feed industries to anticipate and prevent problems before they occur rather than reacting to them when they occur. Approximately 15 of the proposed or enacted measures have a direct impact on the European Union's feed regulation.

The EU had already been moving in the direction of mandatory HAACP, in particular "process controls," for feed mills and premix plants. Council Directive 395L0069 of Dec. 22, 1995, laid down the conditions and arrangements for approving and registering "establishments and intermediaries operating in the animal feed sector." This included commercial feed manufacturers as well as manufacturers of feed for their own use. This directive states that "the manufacturer must have at his disposal a quality control laboratory having adequate staff and equipment to guarantee and check that the compound feedingstuffs containing admixtures comply with the specifications defined by the manufacturer and that will guarantee and check, in particular, the nature and content and homogeneity of the additives concerned in the compound feedingstuffs, and as low a level as possible of cross-contamination. ... A quality control plan must be drawn up in writing and implemented to include, in particular, checks on the critical control points in the manufacturing process. ... Samples must be taken in sufficient quantity by a procedure pre-established by the manufacturer ... in order to ensure traceability ... ."

Registration of feed mills and implementation of "process controls" including mixer performance and cross-contamination testing has been on a national basis in the EU, with feed manufacturers in many of the countries relying heavily on guidance from industry-sponsored feed research institutes. These would include the IFF and DLG in Germany, Tecaliman in France and TNO in the Netherlands. Certain collaboration has occurred among these research institutes, but testing procedures are still being developed and are not harmonized at this time.

The German, French and Dutch feed research institutes agree mixing at feed mills can be validated by testing for one or more microingredients added to feeds at 100 g per metric ton of feed, 1:10,000 dilution, using the analytical results for such "tracers" to assure all other feed ingredients are completely mixed. Validation of mixing at premix plants in Germany is confirmed by testing for microingredients added to premixes at 10 g/mt, a 1:100,000 dilution.

Europeans agree salt (sodium chloride) is not an adequate tracer because it is added to feeds at 0.5-2.0% of a formula and is therefore not a microingredient. The assumption is that if microingredients are completely mixed, then macroingredients will also be completely mixed but that the converse cannot be expected.

All feed mills, whether they add medications to feeds or not, are subject to EU regulation, and in most if not all countries, mixer and cross-contamination tests are being performed at least annually to satisfy the quality assurance requirements. In Germany, Holland and France, the requirements have been taken seriously, and major efforts have been made to develop meaningful and demanding testing procedures. This has included running extensive studies to validate the consistency between various microingredients used as tracers, including medications, amino acids, vitamins, minerals, methyl violet and colored iron particles and powders.

The IFF and DLG research institutes perform mixer and cross-contamination studies as paid for services often with five or more batches of feed studied and with 300 or more samples analyzed. These organizations use fine powdered methyl violet as a tracer to validate mixing. The methyl violet is added to the feed at 10 g/mt (10 ppm), the feed is mixed, samples are taken, the violet color is eluted with solvent from subsamples of feed and the color of the solutions is read on a spectrophotometer. When feeds are completely mixed, it is possible to achieve a coefficient of variation (CV) of 5% or less from a series of "grab" samples taken from a batch. The methyl violet is not food-grade and is very dusty and is thereby inconvenient to work with. Dye recovery from pelleted feeds also can be incomplete.

The TNO research institute uses cobalt and manganese as tracers to validate mixing as well as cross-contamination of feeds with determination of these elements by atomic absorption. Completely mixed feeds can yield CV from a series of grab samples of 5-7%, and cobalt or manganese at high levels can be used to estimate the level of cross-contamination to following batches of feed, though the level of co-

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bait required to permit such testing is unacceptably high in a finished feed requiring a re-blending step before test feed can be consumed.

The Tecaliman research institute uses elemental iron powder colored with water-soluble FD&C colors or the water-insoluble "lake" forms of such dyes as tracers for mixer performance and cross-contamination testing. These tracers are formulated into the feed with the iron separated magnetically from the feed samples. The dye is eluted and read on a spectrophotometer similarly to the methyl violet test. Completely mixed feeds can yield a CV from a series of grab samples of 5% or less. These tracers are food-grade so the feed can be fed to animals. These tracers also are not dusty, and recovery of the dyes from pelleted feeds may be better than for the methyl violet.

In general, mixing is considered complete if a CV of 10% or less is achieved for the microingredient tested in final feeds and if a CV of 5% or less is achieved for premixes. In interpreting particle count data, the CV should not exceed the CV predicted from the applicable Poisson statistics by more than 5%.

Ideally, complete mixing should yield a CV from analysis of a series of samples equal to the CV found from repeat analysis of one homogenous sample.

Basic issues involved in performing a mixer validation study include:

• Selection of one or more tracers as indicators of mixing of all other ingredients;

• Setting the mixing parameters including formula, batch size, mixing time and speed of mixer;

• Taking samples, ideally from within the mixer but when this is impossible from mixer discharge, the need for grab samples and analysis of grab subsamples taken from each sample to minimize the "level of scrutiny" of the test and thereby strengthen the test results;

• The sample analysis, including cost and speed of obtaining results, and

• Interpretation of the analytical results.

Data from the Netherlands TNO and from the French Tecaliman have shown a generally good correlation of results between the various microingredients used in mixer tests. The cost, ease of use and familiarity with the various tracers and their assays have been the most important criteria in tracer selection.

A TNO Jan. 8 report concluded that "taking into account the constraints of this study, ... performance results show that six of the seven tracers are equally suitable for the measurement of mixing uniformity. Four tracers seem about equally suitable to be used in the assessment of mixing uniformity and carry-over in practice, although none of them are without any technical or practical problems. These (acceptable tracers) are the two colored iron particle tracers — coarse and fine (any color, determined by particle count or color reading), cobalt (at mixing levels greater than 25 ppm) and manganese at a high concentration level ... ."

The Tecaliman research reports primarily compared fine colored iron powder, manganese and methyl violet and have found all these suitable as tracers for mixing uniformity but only certain drug assays and the fine-colored iron powder suitable for cross-contamination studies.

The Table (p. 15) shows the summarized results of TNO evaluation of five tracers used to validate mixing and cross-contamination of feeds.

In addition to the European work, excellent research has been performed in the U.S. as well. A study at Kansas State University on the effect of particle size on mixing and segregation of feeds provides some interesting results.

Cross-contamination testing

IFF and DLG use methyl violet for cross-contamination testing, while TNO uses cobalt, and the French Tecaliman uses colored iron powder. Each of these tracers can yield quantitative estimates of cross-contamination of 0.1% or less.

In 1998, IFF suggested 4% cross-contamination of finished feeds and 1% cross-contamination of premixes as achievable limits. Tecaliman in 2003 has suggested 1% cross-contamination of medicated feeds as being an achievable goal. Within the EU more generally, a 1% level of cross-contamination of genetically modified organisms (GMO) into non-GMO feeds has been considered an achievable upper limit.

In Canada, Quebec enacted mixer performance and cross-contamination testing requirements in 1987 that were more specific than the current EU regulations. They were implemented largely because Japan had condemned pork exports from the province for sulfamethazine residues, and this threatened an important export market for the province.

Quebec's regulations have been in force for more than 15 years and require all medicated feed manufacturers, whether a commercial feed mill or on-farm mixer, to be registered with the provincial government. As part of this registration, commercial feed mills must validate mixing twice each year, and on-farm mixers must validate mixing once each year. The regulations further specify that the tests must be performed by a registered agronomist, nine samples must be taken per batch and analyzed for a substance formulated in the feed at not more than 2% in the feed and a CV of less than 10% must be obtained from the analysis of the samples. Salt (sodium chloride) has been most commonly used as the tracer for such tests.

While Canadian regulations are not as specific as those of Quebec, since December 2001, Canada requires all manufacturers of medicated feeds to be registered with the Canadian Food Inspection Agency (CFIA), and as part of the registration, mixing must be validated at least once each year. The Canadian regulations specify that the American Society of Agricultural Engineers' procedure for validating mixing be used, with salt used as a tracer or other methods verified by CFIA.

In the U.S., the Association of American Feed Control Officials (AAFCO) advocated mandatory mixer performance and cross-contamination testing in 2003. This has not changed as of February, though opposition from the regulated industry may lead to delay or modification of AAFCO's position.

In September 2003, the Food & Drug Administration sponsored a public meeting on the possible development of HAACP regulations applicable to the feed industry. More than 200 people, predominantly from the feed industry and government, attended this meeting.

Many comments from industry cautiously supported the need for HAACP controls but only if they would be accepted and useful in satisfying the demands of our trading partners. Many others from industry advocated voluntary "self-inspection" or third-party certification programs. FDA suggested it would be three to four years before proposed rules could be drafted, commented upon and enacted.

What will it cost the feed industry if FDA requires mixer performance and cross-contamination testing? Costs of the actual testing should not be very great if only one batch of feed must be studied: If 10 samples are taken from the batch, the cost for analysis for a tracer as an indicator of mixing will range from $10 to $100 per sample, or from $100 to $1,000 for the batch depending upon the tracer used. This, of course, does not consider the time involved in running the test or, more importantly, the cost of remedying problems that are discovered.

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