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Microtracers (tm) F*) and their Uses in assuring the Quality of Mixed Formula Feeds

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For what purposes are Microtracers F used to assure the quality of mixed formula feed? They are used for at least the following purposes: to test for completeness of mix, to test for adequacy of batch to batch cleanout of feed manufacturing equipment, to code th e p resence (absence) o f c ritical microingredients in f eeds and to identify feed additives and feeds containing such additives as proprietary.

1. **Testing for completeness of** mixing Microtracers have proven useful for this purpose. They offer several advantages over other techniques that may be used to evaluate mixing quality. To understand their use for this purpose, it is best to examine how one tests a mixer.

1.1 **Four steps to test a mixer**

To test a mixer, one will perform at least four tasks:

a) formulation of one or more ,tracers",
b) sampling the mix,
c) analyzing the samples,
d) interpreting results.

1.2 Formulation of the tracer

The ideal tracer would be added at a ,,micro" level, similar to the addition level of critical microingredients (i.e. vitamins, minerals or medications). While it is reasonable to make the inference that if microingredients are completely mixed then macroingredients will also be mixed, it is not necessarily adequate to assume the converse. Since Microtracers F are formulated at not more than 50 grams of tracer (per colour) per tonne of feed, they reasonably meet the criteria of being a,,micro" additive.

Since critical microingredients are generally added to feeds as a component of a premix added at about 1/2 kilo per metric tonne, it is reasonable to formulate the tracer as an ingredient in such a premix. Microtracers are generally premixed with ground corn, salt or with vitamin, mineral or medicated premixes prior to their use in testing mixing.

The location of tracer addition to a mixer can be critical. While most feed manufacturers would first replicate their current production procedures, they often will also test alternate premix addition locations that may yield better mixing.

For certain mixers (i.e. paddle type), it may be best to formulate the premix (with tracer) at opposite ends. The following data (table 1) illustrates typical results when samples are also taken from both ends of the mixer:

Table 1. Typical results when samples are also taken from both ends of the mixer

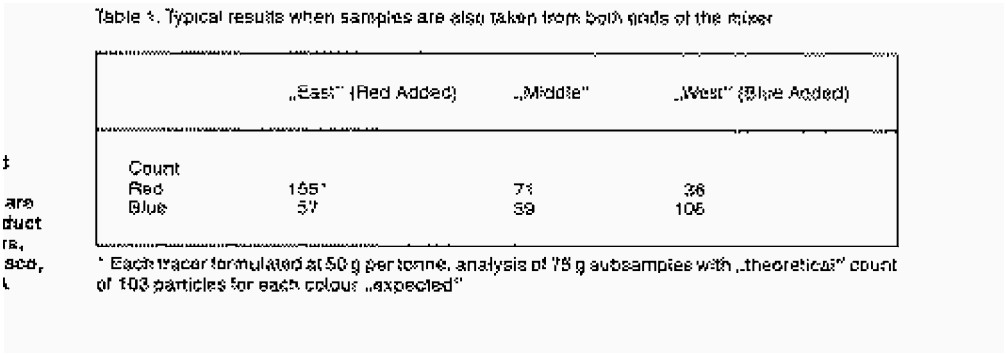
Table 1. Typical results when samples are also taken from both ends of the mixer

	„East" (Red Added)	„Middle"	„West" (Blue Added)
Count			
Red	155*	73	36
Blue	57	59	105

* Each tracer formulated at 50 g per tonne, analysis of 75 g subsamples with „theoretical" count of 103 particles for each colour „expected"

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This data would occur by chance from a ,,perfectly" mixed feed in fewer than five in 10,000 tests (for either colour). The mix is certainly incomplete, yes if the tracer were added either at the centre of the mixer or ideally one-half at each end, the tracer results would be dramatically improved.

1.3 Sampling the batch

To evaluate mixer performance, samples should be taken either from within the mixer or as near the mixer as possible in the manufacturing system. Samples must be ,grab" and not composited, as compositing would be de facto mixing. Samples should be of adequate size to permit repeat analysis of samples yielding deviant or unexpected results. Normally 200 g samples are adequate, with 50 to 75 g subsamples analyzed.

If samples are taken at loadout or at an intermediate location in the manufacturing system, they may include feed from nontarget batches and the feed may have separated during conveyance.

1.4 Analysis of the samples

Microtracers F are isolated from feed samples magnetically using a ,Rotary Detector" magnetic separator or similar device. They are ,demagnetized" so they are not clumped together and then they are sprinkled on a large filter paper wetted with a solvent that will dissolve the dyes from the tracer, usually 70 % alcohol. When the dyes from the tracers begin to make spots on the paper, the paper is dried and when dry the paper is identified and the spots are counted. In practice, it is feasible to analyze approximately 12 samples per hour counting 100 spots for each of two coloured tracers. This is far simpler than analyzing for vitamins, minerals or medications and each tracer analysis may yield two or more sets of data, one set for each coloured tracer. Further, if necessary the testing can be performed ,on-the-spot" at the feedmill. This can be especially useful when diagnosing mixing problems as immediate feedback of information can suggest the parameters for further tests that can be performed immediately.

1.5 Interpretation of results

Microtracer counts are interpreted using Poisson particle statistics. If a series of counts are as would be expected from a Poisson Distribution, the mix is judged complete. If the counts would occur by chance from a ,perfect" mix in fewer than five of 100 tests, the mix is judged ,,probably incomplete". If the counts would occur from a,,perfect" mix in fewer than one of 100 tests, the mix is judged incomplete".
The following data is typical of a **"com*plete mix":***

Red - 87, 86, 77, 73, 97, 93, 87, 85, 83, 80,104

n = 11 (number of samples), mean (average) = 86.5
Found chi-squared value = 9.1; probability of obtaining this value from a ,,perfectly" mixed feed = 0.437
Found coefficient of variation = 10.3 expected coefficient of variation from a ,,perfect" mix = 10.9 %

The following data is typical of an **"in*complete" mix:***

Red - 49, 136, 90, 82, 98, 144, 97, 92, 111,64,90

n = 11 (number of samples), mean (average) = 95.7
Found chi-squared value = 79.7; probability of obtaining this value from a ,,perfectly" mixed feed = less than 0.0005
Found coefficient of variation = 28.9 %, expected coefficient of variation from a ,,perfect" mix = 10.2 %

It should be noted this data was obtained from an actual feedmill test. The first set of data was from a newly installed ribbon mixer mixing a 10,000 lb. (5 ton) batch of broiler feed three minutes. The second set of data was from an adjacent ribbon mixer installed four years earlier and tested under identical conditions. In both cases ,grab" samples were taken from a screw conveyer leaving a surge bin. Precautions were taken to assure as well as possible that batches of feed were not comingled in the surge bin or conveyer.

Since microtracer analyses are relatively inexpensive to perform and since the data is usually available quickly, some feed manufacturers have found it useful to analyze 200 or more samples from a series of batches to refine their mixing parameters as sharply as possible.

These parameters include not just mixing time but also batch size, location of microingredient addition, rpm (revolutions per minute) of the mixer, and feed formula (high roughage feeds may take longer to mix).

Common sense must prevail in planning a mixer test as conditions are significantly different between mills. Often when a feed manufacturer is testing mixing, he can also easily obtain information on the performance of his microingredient addition system (is the same amount added to each batch) as well as for batch to batch ,,carryover" of the tracer as evidence of adequacy of mixer and feed manufacturing system cleanout procedures.

2. **Testing for batch to batch carryover" of tracer as evidence of adequacy of feed manufacturing cleanout procedures:**

Microtracers have been used successfully for this purpose by many feed manufacturers, especially when diethylstilbesterol (DES) was still widely legal and when sulfamethazine was widely used. Generally, the feed manufacturer would add a relatively high level of tracer (i.e. 50 g/t) to a batch of feed formulated with the medication. The

manufacturer would then make the batch of feed (or production run) and take samples both from medicated feed formulated with the tracer as well as from the following ,flushes" and production batches of non-medicated feed. In many cases it has been impossible to correlate microtracer ,carryover" with the carryover of specific feed medications. This is because for many medications, assay procedures are inadequate at trace ,carryover" levels in feeds. Qualitatively, however, microtracer results have almost always been confirmed by assay for the coded medication. The following results from a test at a premix plant may be illustrative (table 2):

In this test, 22 lbs. (10 kg) of Microtracer F-Red was formulated into a 2,500 lb. batch of premix formulated with 500 lbs. of American Cyanimid ASP-250. Four ,,grab" samples were taken from the discharge of the initial batch and from five subsequent ,flushes" and four following production batches of premix. ,,Grab" subsamples were taken from all samples for tracer analysis with results determined ,on-the-spot". The remainder of all samples was sent to American Cyanimid Company for chemical assay. These results were reported approximately two weeks after sample submission. Examples of the actual microtracer results used to prepare table 2 are as follows:

Batch 1-1.66 g of premix analyzed with tracer count = 259

Flush 1-80 g of flush analyzed with tracer count = 363

Table 2. Premix plant ,carryover" test-analytes - Microtracer F-Red and powdered sulfamethazine

Table 2. Premix plant ,,carryover'' test-analytes – Microtracer F-Red and powdered sulfamethazine

Batch #	Total Particles	Particles Per Pound	% of* total	Chemical Assay	% of* total
1	177,060,000	70,824	96.7	8,800 ppm	94.2
Flush 1	618,300	2,061	2.8	331 ppm	3.6
Flush 2	69,600	232	0.3	82 ppm	0.9
Flush 3	15,000	50	0.07	65 ppm	0.7
Flush 4	54,000	54	0.07	32 ppm	0.3
Flush 5	11,000	11	0.02	10.6 ppm	0.1
Followup 1	10,000	5	0.01	1.7 ppm	0.02
Followup 2	18,000	9	0.01	19.4 ppm	0.2
Followup 3	4,000	2	0.003	2.4 ppm	0.02
Followup 4	4,000	2	0.003	2.5 ppm	0.02

* This assumes each production batch and flush were the same size.
** The initial batch was not assayed for sulfamethazine so the ,,Chemical assay'' result for the initial batch is taken to be its specification

This assumes each production batch and flush were the same size. The initial batch was not assayed for sulfamethazine so the ,Chemical assay" result for the initial batch is taken to be its specification

Flush 5-80 g of flush analyzed with tracercount = 20

Followup Batch 1-800 g analyzed with tracer count = 9

Followup Batch 4-800 g analyzed with tracer count = 4

The detection limit for the tracer was lowered in this test by analyzing progressively larger samples of premix. This test did, however, illustrate that at trace levels of contamination, tracer results could underestimate contamination of the medication. In this case, this was probably because the

powdered sulfamethazine was highly electrostatic whereas the tracer was not. If the test were repeated with a granulated sulfa product, the tracer and sulfamethazine results would almost certainly correlate more closely when evaluated by linear regression. A curvillinear regression yielded a correlation Coefficient of 0.98.

Just as many feed manufacturers have found it useful to take 200 or more samples to test for microtracers as evidence of mixing adequacy, they have also found it useful to take large numbers of samples to evaluate the quality of their cleanout procedures. How many and how large must ‚flushes" be? Microtracer results are used to answer such questions as. What sequencing procedures are adequate to reduce the likelihood of significant contamination to de minimus? Manufactures will often use the tracer as a screening tool to isolate "problem" samples where tracer results may be confirmed by subsequent assay of the active medication. The cost of microtracers for periodic testing of mixing and cleanout procedures is trivial as even if a high level of tracer is formulated the number of tonnes containing the tracer is very limited. While microtracers have proven useful for periodic testing, their greatest use has been to identify coded critical microingredients (i.e. vitamins, minerals, medications) in premixes and feeds on a continuous basis.

3. The use of microtracers to code the presence of critical microingredients in feeds for routine quality assurance purposes

For more than ten years, microtracers mineral premixes not only to code the presence of such premixes in feeds but also to code the feeds themselves (i.e. by species, date of feed manufacture, premix supplier or by specific formula starter, grower, finisher). It has only been during the past few years, however, that major pharmaceutical companies have begun to include tracers in their medicated premixes for the benefit of feed manufacturers who can then test *every truckload* of sensitive feed for the coded feed additive.

The first use was by Agri-Bio Corporation of Gainesville, Georgia who responded to the request of a major poultry integrator that a specific microtracer be included in Biocoxsalinomycin premix. The poultry integrator's feed mill was one of the largest and most modern in the United States. The mill produced predominantly broiler and turkey feeds. Unfortunately, the mill had made several formulation or delivery mistakes where feed containing Biocox was delivered and fed to adult turkeys in error. A large number of turkeys were killed, causing an economic loss as well as concern for the welfare of the birds. The feed manufacturer wanted to initiate a program whereby their truck drivers would test *every truckload* of turkey feed before it was shipped to be as certain as possible no further errors would occur. If only one error occurred before per year, if 95 % of such errors could be eliminated th e d rug c ould b e u sed w ith a dequate s afety. T he p rogram s ucceeded w ith more than 2,000 truckloads of turkey feed being tested each year and with only one loss occurr- **The use of** ing during a period of more than five **microtracers** years. In th is o ne in stance, th e tru ck d river failed to ru n th e te st (or keep a "retain sample" of the feed). Further, the dispatcher failed to notice the failure to test. When the loss occurred, feed was taken from the effected birds and the tracer was found at the normal level for a Biocox formulated feed. The level of Biocox was confirmed two or three weeks later by chemical analysis for salinomycin.

After microtracers had proven themselves of value as a routine testing mechanism at one poultry integrator, Agri-Bio offered to include tracers for have been formulated in vitamin and other mills formulating both broiler and
turkey feeds. Five or six major feed manufacturers adopted programs calling for inclusion of a tracer and for continuous testing of their turkey feeds for the presence of the tracer.

Soon there after, MSD Agvet (Division of Merck & Cc) became interested in utilizing a specific , exclusive" microtracer in their Nicarbazin (tm) premix to serve the dual purposes of identifying their premix and feeds containing it as proprietary while also providing feed manufacturers a simple ,quick test" for the medication in feeds. Nicarbazin while being an excellent coccidiostat for **broiler chicken** can destroy shell egg quality when fed to breeders or laying hens. The cost of one mistake with the medication can be 250,000 US$ or more. In the USA, many feed manufacturers refused to consider the medication because of the losses that could occur if it was misformulated or delivered.

MSD Agvet initiated their use of a "Special Blue" tracer in Nicarbazin in 1987 and were the first pharmaceutical company to actively publicize and promote their use of microtracers to permit routine testing to assure their medication is formulated correctly.

Table 3. Microtracers results - analysis of samples from 30 batches of feed - 7 formulated with Biocox-salinomycin and 23 supposed to contain none of the feed additive

Batch #	Biocox Formulated	Tracer Count*
1	yes	34
2	no	2
3	no	1
4	no	1
5	yes	37
6	no	none
7	no	2
8	no	none
9	no	none
10	yes	39
11	no	1
12	no	none
13	yes	27
14	yes	30
15	no	1
16	no	none
17	no	none
18	no	none
19	no	none
20	yes	32
21	no	none
22	no	none
23	no	none
24	no	none
25	no	none
26	no	1
27	no	none
28	yes	26
29	no	2
30	no	none

* Microtracer F-Natural Yellow formulated at 5 g/2,000-lbs. of feed, analysis of samples ranging in weight from 212 to 302 g. 231 tracer particles found from seven feeds formulated with Biocox (a total of 1,708 g of feed) and 11 tracer particles found from 23 feeds not formulated with Biocox (a total of 5,335 g of feed)

Soon thereafter, Hoechst-Roussel AgriVet Co., New Jersey, USA, initiated use of a ,Special Orange" tracer in their coccidiostat premix Stenorol (tm) halofuginone hydrobromide. While this coccidiostat is not toxic if it reaches nontarget birds or animals, USA regulatory authorities set very low tolerances for the medication in poultry tissue. This problem was made more complicated by the facts that while the assay of poultry tissue was extremely sensitive the assay of the feed was not. Feed manufacturers faced a potentially irreconcilable problem of having their birds at risk to condemnation for residues while being unable to test their feeds

promptly and with accuracy for the medication. The Hoechst Program has permitted feed manufacturers to test *every truckload* of broiler or turkey finisher feed *before it is shipped or as it is delivered to a farm* to be as certain as possible it does not contain the medication. Many feed manufacturers have also found the tracer test valuable in testing feeds for the presence of the coccidiostat when they fear an outbreak of coccidiosis may be beginning.

More recently, Elanco Division of Ely Lilly & Co, Pfizer and others have initiated routine Microtracer use in their feed medications or have offered tracers as options for feed manufacturers who request the tracer as in their premixes. In general, when microtracers are used routinely, they are formulated at between 2 and 5 g per tonne of feed at a cost of about 10 cents (USA). The relatively low tracer addition is compensated for by analyzing a relatively large sample. Pfizer Canada recommends testing 500 g feed samples to be as certain as possible the tracer test will provide a correct qualitative test for Coxistat-salinomycin in feeds.

How reliable have tracer tests been for a coded feed additive? The data from a feedmill may be illustrative (table 3).

This data. indicated 1.37% of the total tracer formulated in Biocox premix was eventually found in feeds not supposed to contain the medication. It also illustrates a potential problem. What does one do when he finds trace levels of a tracer in a non-target feed? In this case , the feed manufacturer established acceptance/rejection criteria including taking extra samples when trace contamination occurred, posting a ,control positive" specimen tracer test paper, and requiring testing of at least one known positive feed each day to assure the method was working.

What is the future for routine tracer testing for coded feed additives? In the USA, one major poultry integrator now includes microtracers in virtually every coccidiostat it uses and not only tests every sensitive truckload of feed at the feedmill but goes further and tests every truckload of breeder feed as it is delivered to each farm. No mistakes have occurred since this program was initiated about three years ago.

3.1 Other tracer applications

It has already been mentioned that microtracers can be used to code feed additives and feeds containing the additive as being proprietary. While many such tracers are widely known (as with MSD Agvet's use in Nicarbazin), several are secret and likely will only be known by the feed additive manufacturer. Microtracer (tm) RF-Se-4% is a unique tracer application. This product consists of about 92% food grade reduced elemental iron, 8.76% sodium selenite and traces and sodium carbonate. The product is used as a source of sodium selenite for feed premixes. When a premix manufacturer wants to analyze a premix batch for its selenium content, he takes a sample, isolates magneticaly retrievable material on a magnet and weighs the iron on an analytical balance. For many premix formulations, the tracer analysis will be more accurate and reliable than chemical assays.

3.2 The future

Micro Tracers, Inc. has received patents for ,Micro-ingredient Containing Tracers" consisting of a formulation of about 30 % extremely fine iron, 60% inert carrier, 1 % food dye and 9 % medication or other feed additive. Such a formulation will permit the retrieval of the coded feed additive in relatively pure form from a feed. The dye can be eluted from the particle onto a wetted filter paper yielding an ,,on-the-spot" quick test for the additive. Further, the tracer may then be analyzed for the additive permitting chemical or microbiological assays that would otherwise be impossible.

It is unrealistic to expect no feed manufacturing errors will occur. It is possible using existing tracer methodology to reduce the incidence and severe- of such errors. The ,cost" of testing while real is generally far less than the cost of the errors that are prevented.

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Summary

While Microtracers (tm) have been marketed actively in the United States and elsewhere for some years, they have been promoted actively in continental Europe only recently. Further, it has only been during the past few years that major pharmaceutical companies have included microtracers in medicated premixes for the benefit of their customers, manufacturers of medicated feeds.

This paper describes briefly the uses of Microtracers F in assuring the quality of mixed formula feeds. These uses include: testing for completeness of mixing, testing for adequacy of batch to batch cleanout procedures, coding the presence (absence) of critical microingredients in feeds and identifying premixes and feeds containing such premixes as proprietary. To the extent consumers and government regulatory officials are demanding increased control over the use of medications in feeds and to the extent microtracers offer the possibility of such control, they are more relevant today than in the past. Their use in medicated premixes is especially important in permitting feed manufacturers to perform extensive ,on-the spot" testing that conventional analytical procedures do not permit. Such testing may have both economic and animal welfare implications when a medication may be toxic to non-target animals or avian species (i.e. nicarbazin breeder feeds, salinomycin in turkey feeds, monensin sodium in horse feeds) as well as food safety implications to the extent routine testing for microtracers may reduce the incidence of drug residues in animal and poultry meat.

Mikromarkierungsstoffe ,(tm) F" und ihre Anwendung zur Qualit5tssicherung in Futtermischungen

Bei den Mikromarkierungsstoffen (microtracers) ,(tm) F" handelt es sich um feingefarbte Eisenteilchen einheitlicher GroBe Sie dienen als Indlikator zur Kon- der GleichmaBigkeit von Mischun(Anwendungsmenge nicht mehr als 50 g/t Mischfutter) und der gleichmaBi- Verteilung von in Vormischungen zu- Mikrokomponenten (Vitamine, Mineralstoffe, Medikamente), wobei sie hier in einem bestimmten Anteil in der Vormischung gleichmaBig verteilt enthalsind. Zur Uberprufung der Mischung werden die Teilchen des -Microtracers F" magnetisch aus der zu untersuchen- Probe abgetrennt, entmagnetisiert und auf einem groBen Filterpapier solan- mit einem Losungsmittel bespruht big sich die Farbflecke von den einzelnen Teilchen auf dem Filter abzeichnen. Die se werden nach dem Trocknen des Filters ausgez~ihlt. Die Ergebnisse k6nnen statistisch ausgewertet werden.

Mikromarkierungsstoffe werden schon seit langerem in den USA benutzt, in Europa dagegen erst seit kurzem. Der Beibeschreibt ihre vielseitigen Anwen- Sie konnen auBer in den erwahnten Fallen auch zur Kon- beim Reinigen von Mischern (Kon- der folgenden Mischungen mit unerwunschten Bestandteilen) und zum Markieren von kritischen Mikrokomeingesetzt werden, wobei sich je nach Rezeptur die An- oder Abwesen- dieser Komponenten kennzeichnen laBt Das ist besonders wichtig bei mediVormischungen mit Inhaltsstof- die bei nicht fur sie bestimmten Tier- gefahrliche Nebenwirkungen verursachen konnen Da des Ergebnis der Markierungsstoff-Kontrolle im Ge- zu der bei solchen Stoffen in der Regel zeitaufwendigen Analytrik schon nach verhditnismcHig kurzer Zeit vorliegt, kann sie vor Ort benutzt werden, um Fehlmischungen schnell zu erkennen und gr6l3ere Schdden zu vermeiden.

Las microsubstancias de **marcaje "(tm)F" y su empleo para el aseguramiento de la calidad de las mezclas de piensos**

Con referencia a las microsubstancias de marcaje (microtracers) "(tm)F", se trata de finisimas part(culas coloreadas de hierro de tamafio uniforme. Sirven de indicador para controlar la regularidad de las mezclas (cantidad empleada no superior a 50 gr. por Tm. de pienso mixto) y la distribuci6n regular de los microcomponentes (vitaminas, substancias

minerales, medicamentos) a~adidos a las premezclas; a cuyo efecto estAn contenidas, uniformemente distribuidas, en la premezcla en una porci6n determinada. Para comprobar la mezcla, las particulas del "microtracers V se separan magn6ticamente de [a muestra a ensayar, se desmagnetizan y se recfan con un disolvente sobre un gran papel de filtro hasta que aparecen sobre el filtro manchas de color de las particulas individuales. Despu6s de secar el filtro, se cuenta el nOmero de 6stas. Los resultados se pueden valorar estadisticamente.