Micro-Tracers Inc.
1370 Van Dyke Avenue, San Francisco, California 94124 USA
Tel: (415)822-1100 Fax: (415)822-6615 Website: www.microtracers.com

Microtracer F
Testing For Completeness of Mix

The Problem:

The worldwide formula feed industry manufactures more than 300 million tons annually. Manufacturers waste labor, energy and capitol when they mix feeds longer than necessary to achieve a complete blend. Excess mixing may also cause degradation of vitamins and medications.

If feed is not completely mixed, portions of the feed will contain either too much or too little of the formulated ingredients. This excess variability causes economic losses to users of the feed and may increase the incidence of illegal drug residues.

Periodic routine mixer testing is both economically and ethically justified.

Comparative Methods:

Feed manufacturers often test mixing equipment by analyzing their feed for one or more nutrients (or medications) normally present in the feed or by adding a "tracer" specifically for the test.

When a nutrient is tested, the manufacturer uses this nutrient as a “tracer" for purposes of evaluating mixing Quality.

Feed manufacturers often test for the following:

1. Macronutrients (i.e. protein, moisture, fat)
2. Salt (i.e. chloride)
3. Elements (calcium, manganese, zinc etc).
4. Vitamins or medications.
5. Microtracers (tm)

For all of these except drug and Microtracer™ assays, results may be confused by background "noise" where the nutrient is contributed to the feed from more than one source. If many feed ingredients contain protein (or salt) at significant levels, then the feed could appear mixed even if no mixing occurred.

Results may further be confused by imprecise analytical methodology (i.e. for drug assays) If an analytical method yields results no better than +/-30% CV, this can hardly be used to determine if a feed is adequately mixed.

Microtracers™ offer an excellent technique to validate mixing because:

1. Microtracer analyses have little analytical error.
2. Background "noise" does not interfere with results.
3. Cost per analysis is very low and several different tracers can be tested in the same test batch. This allows evaluation of several mixing times or microingredient addition locations in one test.
4. Testing can be performed "on the spot" allowing immediate evaluation of results and further testing the same day.

Testing a Mixer:

There are four problems one must satisfy in any mixer test:

1. Addition of the tracer (where, when, how much, any required premixing, use of multiple tracers etc).
2. Sampling the mix (where, when, how much, how many samples)
3. Analysis of the samples (method of analysis, how much, when are repeat analyses justified or required)
4. Interpretation of results.

These problems are common to any mixer test, whether one employs Microtracers or some other procedure. The remainder of this paper will discuss these problems specifically as they apply to the use of Microtracers.

Addition of Microtracers:

Each mixer test presents a unique set of circumstances and "common sense" must prevail. A few general statements may, however, be appropriate.

1. Microtracers F (colored iron particles) are usually added at 50 grams of tracer per ton of mix. (i.e. 100 grams of a Red tracer may be added to a two ton batch)
2. This tracer should be premixed in one pound of diluent (i.e. ground corn, salt etc) before adding the tracer to the mix.
3. The tracer can be added to the mix at the same time and location as a "hand added" vitamin or medication. Alternately, a tracer can be incorporated in a vitamin premix and added to feed via a computerized micro-ingredient addition system.
4. A second tracer can be added to the test batch one minute after the first tracer or at a second location. This will yield a second series of information from the same test.

Sampling the Feed:

1. Ideally, one takes "grab" samples from the mixer either at spaced intervals during the mix or on completion of the mix.
2. Samples should weigh at least 1/2-lb. and <u>must be</u> "grab" and not composites, for composite sampling tells nothing about mixing quality.
3. If one cannot take samples from the mixer, then take them as near the mixer in the production system as possible. Often, the most feasible location is from a screw conveyer leading from the surge bin.

4. If one samples from a mixer, one should take at least three samples, one from the middle and one from each end. If one samples from the screw conveyer after the surge bin, one should take at least five and preferably ten samples from spaced portions of the mix discharge.
5. One may also want to sample from the following batch of feed to determine batch to batch tracer "carryover".

<u>Microtracer Analyses:</u>

Please refer to Microtracer literature items "A-1" (Quality Assurance with Microtracers F), "A-2" (Microtracer "Rotary Detector") and "A-3" (Microtracers F Quantitative Procedure)

Microtracers F (colored uniformly sized iron particles) are removed from sub-samples (usually 75 grams) of each sample taken from the batch utilizing a "Rotary Detector" magnetic separator. These particles are transferred to a weigh scoop, demagnetized using a bulk tape eraser and then sprinkled on a large (i.e. 15 to 18.5 cm Whatman #1) filter paper moistened with a 60% ethanol solution.

When spots begin to develop, one transfers the paper to a pre-heated hot plate or oven and dries it.

When the paper is dry, one marks it for identification and then counts all the particles of one color noting the total and then counts all the particles of a second color noting the total.

<u>Interpreting Microtracer Results:</u>

One interprets Microtracer™ mixer testing results utilizing Poisson Statistics and related chi-squared calculations and tables.

If a mix is "complete" or "perfect", Microtracer counts will exhibit variability characteristic of a Poisson Statistical Distribution. If Microtracer counts are more variable than one would expect from a Poisson Distribution, one concludes the mix is not complete.

Please contact Micro-Tracers, Inc. for further information on the theory of the Poisson Distribution and the applicability of it and chi-squared calculations to evaluating Microtracer counts.

<u>Use of Chi-Squared Calculations:</u>

Chi-squared calculations are derived from the Poisson Distribution and are used to evaluate Microtracer counts as evidence of mixing.

One determines Microtracer counts (xl, x2, x3...) from a number of feed samples (n). One then calculates the average count (the mean) X.

One then determines the difference between each count (xl) and the mean (X), squares each difference and adds each squared difference to obtain the sum of the squared differences.

One then divides the sum of the squared differences by the mean to obtain the chi-squared value.

One then refers to a table of chi-squared probabilities (Table A)

One locates the number of independent samples (top horizontal column), subtracting 2 to reflect 2 degrees of freedom. One then locates the found chi-squared value (left side vertical column).

The intersection of the horizontal and vertical columns yields a probability (anywhere from .999 to ** -less than .0005). This is the probability the chi-squared value found in the test would be exceeded by chance from a "perfect" Poisson mix.

If the data from a test would occur by chance from a "perfect' mix more than 5 times in *100* tests (probability over 0.05*),* one assumes the data is typical of a "perfect" mix.

If the data from a test would occur by chance from a "perfect" mix between 1 and 5 times in 100 tests (probability between 0.05 and 0.01), one assumes the data is exhibiting a variability "probably significantly deviant" from a "perfect" mix.

If the data from the test would occur by chance from a "perfect" mix fewer than 1 time in 100 tests (probability less than 0.01), one assumes the data is exhibiting a variability that is "statistically significantly deviant" from a "perfect" mix and the feed is not completely mixed.

Please refer to Table B for a sample chi-squared calculation as well as for illustrative data from several actual mixer tests.

Comparing "Found" with "Theoretical" Coefficients of Variation:

A key attribute of the Poisson Distribution is that if a mix is "perfect", the standard deviation of a series of counts should (on the average) equal the square root of the mean count. If the mean (average) count from a mixer test is 100, the standard deviation from a series of counts should (on the average) be 10 and the coefficient of variation (CV) of the data should be *10%* (the coefficient of variation being the standard deviation divided by the mean).

If one completes a Microtracer™ mixer test, one can determine the "found" coefficient of variation and compare this with the "theoretical" value expected from a "perfect" mix. If the found value is greater than the theoretical, this will give some measure of the economic loss incurred due to incomplete mixing. For example, if the found coefficient of variation (CV) is 20% when it should theoretically be *10%,* one might argue 10% of the value of micro-ingredients is being lost due to incomplete mixing.

Micro-Tracers, Inc. has generated an excel program for calculating chi-squared values, standard deviations, found and actual coefficients of variation, and for reporting data with interpretation. Please contact Micro-Tracers, Inc. or visit www.microtracers.com for this Program.

Table A: Probability that X^2, Derived From “d.f.” Independent Counts will be Exceeded Solely Through Errors of Random Sampling[1]

Probability Integral of x^2

Number of independent elements, (n-2)

X^2	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*	*13*	*14*	*15*	*16*	*17*	*18*	*19*	*20*
1	.317	.607	.801	.910	.963	.986	.995	.998	.999	.999	.999	.999	.999	.999						
2	.157	.368	.572	.736	.849	.920	.960	.981	.991	.996	.998	.999	.999	.999	.999	.999	.999	.999		
3	.083	.223	.392	.558	.700	.809	.885	.934	.964	.981	.991	.996	.998	.999	.999	.999	.999	.999	.999	.999
4	.046	.135	.261	.406	.549	.677	.780	.857	.911	.947	.970	.983	.991	.995	.998	.999	.999	.999	.999	.999
5	.025	.082	.172	.287	.416	.544	.660	.758	.834	.891	.931	.958	.975	.986	.992	.996	.998	.999	.999	.999
6	.014	.050	.112	.199	.306	.423	.540	.647	.740	.815	.873	.916	.946	.966	.980	.988	.993	.996	.998	.999
7	.008	.030	.072	.136	.221	.321	.429	.537	.637	.725	.799	.858	.902	.935	.958	.973	.984	.990	.994	.997
8	.005	.018	.046	.092	.156	.238	.333	.433	.534	.629	.713	.785	.844	.889	.924	.949	.967	.979	.987	.992
9	.003	.011	.029	.061	.109	.174	.253	.342	.437	.532	.622	.703	.773	.831	.878	.913	.940	.960	.973	.983
10	.002	.007	.019	.040	.075	.125	.189	.265	.350	.440	.530	.616	.694	.762	.820	.867	.904	.932	.953	.968
11	.001	.004	.012	.027	.051	.088	.139	.202	.276	.358	.443	.529	.611	.686	.753	.809	.857	.894	.924	.946
12	.001	.002	.007	.017	.035	.062	.101	.151	.213	.285	.363	.446	.528	.606	.679	.744	.800	.847	.886	.916
13	**	.002	.005	.011	.023	.043	.072	.112	.163	.224	.293	.369	.448	.527	.602	.673	.736	.792	.839	.877
14	**	.001	.003	.007	.016	.030	.051	.082	.122	.173	.233	.301	.374	.450	.526	.599	.667	.729	.784	.830
15	**	.001	.002	.005	.010	.020	.036	.059	.091	.132	.182	.241	.307	.378	.451	.525	.595	.662	.723	.776

X^2	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*	*13*	*14*	*15*	*16*	*17*	*18*	*19*	*20*
16	*	*	.00	.00	.00	.01	.02	.04	.06	.10	.14	.19	.24	.31	.38	.45	.52	.59	.65	.71
	*	*	1	3	7	4	5	2	7	0	1	1	9	3	2	3	4	3	7	7
17	*	*	.00	.00	.00	.00	.01	.03	.04	.07	.10	.15	.19	.25	.31	.38	.45	.52	.59	.65
	*	*	1	2	4	9	7	0	9	4	8	0	9	6	9	6	4	3	0	3
18	*	*	**	.00	.00	.00	.01	.02	.03	.05	.08	.11	.15	.20	.26	.32	.38	.45	.52	.58
	*	*		1	3	6	2	1	5	5	2	6	8	7	3	4	9	6	2	7
19	*	*	**	.00	.00	.00	.00	.01	.02	.04	.06	.08	.12	.16	.21	.26	.32	.39	.45	.52
	*	*		1	2	4	8	5	5	0	1	9	3	5	4	9	9	2	7	2
20	*	*	**	**	.00	.00	.00	.01	.01	.02	.04	.06	.09	.13	.17	.22	.27	.33	.39	.45
	*	*			1	3	6	0	8	9	5	7	5	0	2	0	4	3	5	8
21	*	*	**	**	.00	.00	.00	.00	.01	.02	.03	.05	.07	.10	.13	.17	.22	.27	.33	.39
	*	*			1	2	4	7	3	1	3	0	3	2	7	9	6	9	7	7
22	*	*	**	**	.00	.00	.00	.00	.00	.01	.02	.03	.05	.07	.10	.14	.18	.23	.28	.34
	*	*			1	1	3	5	9	5	4	8	5	9	8	3	5	2	4	1
23	*	*	**	**	**	.00	.00	.00	.00	.01	.01	.02	.04	.06	.08	.11	.14	.19	.23	.28
	*	*				1	2	3	6	1	8	8	2	0	4	4	9	1	7	9
24	*	*	**	**	**	.00	.00	.00	.00	.00	.01	.02	.03	.04	.06	.09	.11	.15	.19	.24
	*	*				1	1	2	4	8	3	0	1	6	5	0	9	5	6	2
25	*	*	**	**	**	**	.00	.00	.00	.00	.00	.01	.02	.03	.05	.07	.09	.12	.16	.20
	*	*					1	2	3	5	9	5	3	5	0	0	5	5	1	1
26	*	*	**	**	**	**	.00	.00	.00	.00	.00	.01	.01	.02	.03	.05	.07	.10	.13	.16

	*	*					1	1	2	4	6	1	7	6	8	4	4	0	0	6
27	**	**	**	**	**	**	**	.00	.00	.00	.00	.00	.01	.01	.02	.04	.05	.07	.10	.13
								1	1	3	5	8	2	9	9	1	8	9	5	5
28	*	*	**	**	**	**	**	**	.00	.00	.00	.00	.00	.01	.02	.03	.04	.06	.08	.10
	*	*							1	2	3	6	9	4	2	2	5	2	3	9
29	*	*	**	**	**	**	**	**	.00	.00	.00	.00	.00	.01	.01	.02	.03	.04	.06	.08
	*	*							1	1	2	4	7	0	6	4	5	8	6	8
30	*	*	**	**	**	**	**	**	**	.00	.00	.00	.00	.00	.01	.01	.02	.03	.05	.07
	*	*								1	2	3	5	8	2	8	6	7	2	0

i A.E. Treloar, *Elements of Statistical Reasoning, 1939,* p. 246-247,
Courtesy John **Wiley & Sons, Inc. * Greater than** *.9995.*
* * Less than .0005.

Table B: Illustrative Theoretical Chi-Squared Calculations:

Example 1: Complete Mix

Sample#	Found Count	Mean Count	Difference	Squared Difference
1	85	100	15	225
2	105	100	5	25
3	95	100	5	25
4	115	100	15	225
5	100	100	0	0
	Mean (Average)	100	Sum =	500

Sum of Squared Difference divided by Mean =
Found Chi-squared 500 divided by 100 = 5

Probability a "perfect" mix would yield a chi-squared value in excess of 5 (from Table A, n = 5 -2 = 3) = 0.172 or 17.2%.

Conclusion : This test yielded data typical of a "perfect" mix.

Example 2: Incomplete Mix

Sample#	Found Count	Mean Count	Difference	Squared Difference
1	85	100	15	225
2	65	100	5	1,225
3	115	100	15	225

4	135	100	35	1,225
5	100	100	0	0
	Mean (Average)	100	Sum =	2,900

Sum of Squared Differences divided by Mean=

Found Chi-Squared 2,900 divided by 100 = 29

Probability a "perfect" mix would yield a chi-squared value in excess of 29 (from Table A, n = 5 - 2 = 3) = ** (less than .0005)

Conclusion: This test yielded data typical of an incomplete mix.

<u>Table C – Illustrative Chi-Squared Calculations from Actual Feedmill Tests</u>

<u>Example 1: Incomplete Mix</u>

Sample#	Found Count (Red)	Mean Count	Difference	Squared Difference
North	50	95	45	2,025
Center	96	95	1	1
South	139	95	44	1,936
	Mean (Average)	95	Sum =	3,962

Sum of Squared Differences divided by Mean = 40.8

Probability a "perfect" mix would yield a chi-squared value in excess of 20 (from Table A, n =3-2 =1) = ** (less than 0.0005)

Conclusion: This mix is not complete. Further, the Red tracer was added at the South End of the mixer and movement of this tracer to the opposite end of the mixer is incomplete.

<u>Example 2: Incomplete Mix</u>

Sample#	Found Count (Blue)	Mean Count	Difference	Squared Difference
North	201	133	68	4,624
Center	132	133	1	1
South	65	133	68	4,624
	Mean (Average)	100	Sum =	9,229

Sum of Squared Differences divided by Mean = 92.29

Probability a "perfect" mix would yield a chi-squared value in excess of 35 (from Table A, n=.3 -2 =1) = ** (less than 0.0005)

Conclusion: This mix is not complete. Further, the Blue tracer was added at the Center of the mixer and failed to distribute completely to the ends of the mixer.

<u>Example 3: Complete Mix at "Smith Foods" (1989)</u>

"1 November 1989

Smith Foods
#1- Main Street
Webfoot, Oregon 97979

TO: Mr. William Smith

RE: Microtracer (tm) Mixer Test- Your letter dated 13 October 1989; Ref; DFK/89/026; 40 Feed Samples (one lost in analysis); samples received 20 October 1989.

Blue Tracer Counts									
117	121	139	130	116	105	117	122	113	131
121	112	113	126	120	148	111	128	134	130
133	138	120	134	125	128	135	139	140	126
133	137	101	128	120	139	123	153	154	
Red Tracer Counts									
130	120	120	106	131	117	143	114	118	134
125	131	121	134	140	140	114	111	149	131
122	147	115	132	105	121	109	118	116	132
129	116	107	136	130	130	124	129	122	

	Red	Blue
Number of Data Points	39	39
Degrees of Freedom	37	37
Mean =	127.18	124.85
Standard Deviation = +/-	12.09	11.16
Coef. of Variation (CV), % +/-	9.51	8.94
CV (Poisson), % +/-	8.87	8.95
Chi-Squared =	43.70	37.91
Probability, %	20.82	42.76

Conclusion: Results for both Microtracers are typical of a complete "perfect" mix. Data was for samples weighing from 54 to 89 grams with data adjusted to a constant weight of 75 grams.

Tracer recovery was aproximately 124% for the Blue and 120% for the Red tracer assuming each was formulated at 50 gms per ton.

Micro-Tracers, Inc.

David Eisenberg, President"

Example 4: Incomplete Mix at "Jones Equipment Company" (1990)

"10 May 1990
Jones Equipment Company
1313- Maple Street
Jones, Iowa 50505

TO: Mr. John Jones

RE: Microtracer (tm) Mixer Test- Eight (8) Samples marked 5-A thru 5-H; received San Francisco 7 May 1990; refer your letter dated 7 May 1990; 5-Minute mix.

Red Tracer Counts							
200	279	182	103	268	340	186	118
Blue Tracer Counts							
20	13	148	290	36	68	263	343

	Red	Blue
Number of Data Points	8	8
Degrees of Freedom	6	6
Mean =	209.50	147.63
Standard Deviation = +/-	81.41	133.61
Coef. of Variation (CV), % +/-	38.86	90.51
CV (Poisson), % +/-	6.91	8.23
Chi-Squared =	221.46	846.51
Probability, %	0.00	0.00

Conclusion: Results for both tracers indicate the mix is not complete.

Micro-Tracers, Inc.

David A. Eisenberg, President"

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