



Micro-Tracers Inc.

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

Quality Assurance With Microtracer F-Ni

Principle:

Microtracer F-Ni (iron/nickel particles) is an easily identifiable "harmless marker" used to assure the quality of mixed formula feeds, especially feeds containing high levels of moisture (i.e. in excess of 15%).

When assayed quantitatively, Microtracer F-Ni can be used to document efficacy of mixing as well as adequacy of cleanout of mixers and other feed manufacturing equipment.

Microtracer F-Ni may be isolated from high moisture feeds by drying such feeds, then grinding them to a fine mash, and finally separating the tracer using a Microtracer Rotary Detector magnetic separator.

This tracer is use primarily for testing mixing of high moisture feeds or in gastrointestinal throughput studies.

Specifications:

Microtracer F-Ni consists of uniformly sized iron/nickel particles, a minimum of 99% passing 35-mesh and a maximum of 3% passing 200-mesh screens (USA Standard Sieves).

Microtracer F-Ni will withstand pelleting and prolonged exposure to high moisture.

Microtracer F-Ni has a specified count of 50,000 particles per gram. In practice, the tracer count will fall in the range 40,000 to 65,000 particles per gram.

Tracer recovery should be 100% from samples taken directly from a mixer to which the tracer was added and 75% for both mash and pelleted feeds made from the batch. These recoveries assume use of a Rotary Detector™ to retrieve the magnetic particles from the feed.

Tracer recovery with the Mason Jar procedure will be qualitative only.

Applications and Amount to Use:

1. Routine Identification of Premix in Finished Feeds

Premixes should be formulated to yield approximately 5 grams of tracer per metric ton of finished feed. If a premix is added to the feed at 500 g per ton, then 10 grams of tracer should be formulated per kilogram of premix.

This will yield a theoretical count of 16 tracer particles per 65 grams of feed, an amount that can be conveniently analyzed utilizing a Mason Jar with magnetic lid. If tracer recovery for a pelleted

feed is 65%, then on an average test one would find 10 tracer particles. If a feed is completely mixed and one expects to find ten tracer particles, the likelihood of finding none based on the Poisson statistics would be less than 1 in 100 tests.

For greater confidence and to measure "carryover" of premixes coded with Microtracer F-Ni, one should use a Rotary Detector to test for microtracers. This permits complete tracer recovery and analysis of larger feed samples (i.e. 500 grams). The chances of obtaining a "false negative" (coded premix present at formulated level but no tracer found) will be nil. The likelihood of finding at least one tracer particle if 10% "carryover" of the premix to a non-target feed occurs will be better than 95%.

2. Mixer Efficiency

To determine completeness of mix, formulate Microtracer F-Ni at 25 grams of tracer per metric ton of feed. One must use a Rotary Detector to obtain quantitative information. Usually, one will analyze 75 gram feed samples obtaining tracer counts of about 100 particles. A series of such counts from a "perfectly" mixed feed will yield a coefficient of variation (CV) of about 10%. If 10 samples are taken from a batch and one finds a 20% coefficient of variation, this will evidence a "statistically significant" deviation from complete mixing. Please refer to *Literature Item P - The Use of Microtracers to Determine Completeness of Mix*.

3. Product Identification

Microtracer F-Ni may be formulated at 5 grams per metric ton to code a feed as proprietary. This is useful in protecting patent or distribution rights, in servicing improper product liability claims or requests for services and in controlling use of proprietary feed (i.e. misuse of feed by contract growers).

Detection Procedure - Rotary Detector Technique: *(from mash or pelleted feeds)*

Retrieval

Materials:

- a) Coffee mill, or equivalent, for grinding pelleted samples to a fine consistency

Quality Assurance With Microtracer F-Ni

- b) Rotary Detector™ magnetic separator, for isolating the tracer from premixes or ground feed samples.
- c) Scale

Procedure:

Weigh 500 g to 1.0 kg coarse or pelleted samples and grind in the coffee mill. Pour the finely ground feed through the Rotary Detector™ twice to isolate very nearly 100% of the retrievable iron from the feed. Brush this magnetically retrieved material into a weigh scoop and "demagnetize" it using a bulk tape eraser. The tracer is now prepared to be developed (alternatively, you may just directly brush the tracer particles onto a large filter paper).

Development

Materials:

- c) E & K Scientific 601 Grade filter paper, 18.5 cm circles (or larger).
- d) A slightly larger square of glass to support the filter paper.
- e) The Acid Reagent
[Dissolve 25 g of tartaric acid in water to make 100 mL, and then mix with 50 mL of hydrochloric acid].
- f) The Color Reagent
[Mix a 1% alcoholic solution of dimethylglyoxime with an equal volume of ammonium hydroxide].
- g) A hot plate (a household griddle will do) in a laboratory hood.
- h) A wash tub filled with water to rinse filter papers in a laboratory hood.

Procedure:

Caution! Wear gloves and work under a hood!

1. Place a filter paper on a double layer paper towel. Sprinkle the demagnetized iron particles retrieved from a feed sample onto the filter paper, thoroughly spreading them with the help of a fan brush.
2. With an eye dropper, pour 6-8 mL of Acid Reagent onto the glass plate and carefully place the filter paper with the tracer particles over the glass plate. Allow 1-2 minutes for the paper to absorb the Acid Reagent and make sure the paper is thoroughly wet (avoid puddles

- of excess Acid Reagent).
3. After 2-3 minutes, transfer the filter paper from the glass plate back to the paper towel so that excess moisture is absorbed.
 4. Allow the particles to sit on the filter paper for at least 30 minutes.
 5. Gently lift the glass plate and spray the Color Reagent onto the particles on the filter paper (make sure you do this under a laboratory hood). Red spots will develop immediately. With an eye dropper, pour 10-12ml of the Color Reagent by portions 3-4 ml and check the reading of pH with an indicator strips. The addition of the Color reagent should be continued until pH reaches 8.0. There will be relatively large spots which will eventually diffuse. The excess of the Color Reagent will aid in the diffusion of the large red spots.
 6. Place the filter paper on the hot plate (preheated to about 100°C or less) and dry, being careful not to burn the paper. The corresponding ferrous compound is fugitive under the specified conditions. Iron spots will either disappear or possibly turn brown. The remaining red spots indicate the presence of nickel.
 7. When the paper is dry, mark it for identification. When time is available, count the colored spots by making hash marks next to the colored spots.
 8. Employ Poisson statistics and chi-squared calculations to interpret the results of the test. Please refer to Microtracer *Literature Item P - The Use of Microtracers to Determine Completeness of Mix..*

Total elapsed time: 40 minutes

Detection Procedure - Mason Jar Technique

Note: There is no real advantage in using the Mason Jar Technique for this particular tracer since the developing procedure is quite involved, but it provides a means to retrieve the tracer particles from the feed if no Rotary Detector is available.

Materials:

- a) A scale suitable for weighing 65 grams of feed (if this is unavailable, feed may be measured volumetrically in the Mason Jar itself; 1/2 Jar roughly equals 65 grams).
- b) E & K Scientific 601 Grade filter paper, 7.0 cm circles.
- c) For pelleted feeds, a coffee mill or grinder.
- d) A Mason Jar with a magnetic lid (supplied by Microtracers, Inc.).

Quality Assurance With Microtracer F-Ni

Procedure:

- a) Prepare pelleted feeds for analysis by grinding them to mash.
- b) Transfer 65 grams of feed to Mason Jar.
- c) Insert one sheet of filter paper into special magnetic lid and screw lid onto Mason Jar.
- d) Shake the jar for one minute, exposing all feed to the magnetic lid.
- e) Remove the lid, placing it upside down with filter paper fully exposed. Carefully lift the filter paper and transfer the tracer particles to a large filter paper. Continue as indicated under Developing Procedure - Rotary Detector Technique, part 1.

Comments:

The usefulness of this method is obviously limited by the "noise level" of nickel in ferromagnetic particles present in feeds from other sources. Of 15 feed samples examined during another investigation, particulate iron ranged from 10 ppm to 267 ppm, averaging 55 ppm. Such "noise" results primarily from wear of hammers in hammer mills, and these generally do not contain nickel.

Last modified: 1-02-13 NB