

Humic Acid Labeling Aid

If your product label has a guarantee for humic acid, please answer the following questions:

1. Is your product liquid or dry?

- a) If liquid, must guarantee at least 0.06%* of humic acid.
- b) If dry, must guarantee at least 0.6%* of humic acid.

2. What is the type of your product?

- a) If your product is a **Packaged Soil Amendment**, add humic acid and its source to the ingredients list. For example: humic acid (from leonardite).
- b) If your product is an **Auxiliary Soil and Plant Substance**, add humic acid guarantee under a “NONPLANT FOOD INGREDIENT” heading, a percentage value to the left, and the source of humic acid. For example:

NONPLANT FOOD INGREDIENT:
X humic acid (from leonardite)

- c) If your product **guarantees nutrients**, add humic acid guarantee under an “ALSO CONTAINS NONPLANT FOOD INGREDIENT” heading, a percentage value to the left, and the source of humic acid. For example:

ALSO CONTAINS NONPLANT FOOD INGREDIENT:
X% humic acid from leonardite

* The humic acid guarantee should be based on the CDFA method for determination of humic acid (see below);

** If your product contains a significant amount of potash, you must add a potash guarantee on the label. See also “Notice to Industry” for more information

Humic Acid Method

1. Scope:

The procedure is to be used for humic acid analysis of solid samples containing a minimum of 0.5% humic acid and for liquid samples containing a minimum of 0.05% humic acid.

2. Principle:

The humic acids are dissolved by treatment with 0.5N sodium hydroxide and then precipitated with hydrochloride acid.

3. Safety:

Care must be taken when working with acids and bases. Follow the proper safety procedures outlined in the Injury and Illness Prevention Program Manual.

Refer to the Instruction Manual for the proper and safe use of the centrifuge.

4. Interferences:

None

5. Apparatus and Equipment:

- Centrifuge (9.5 inch radius)
- Mechanical shaker
- Drying oven (100-110°C)
- Centrifuge bottle with screw caps (100 mL)
- General laboratory glassware

6. Reagents and Supplies:

- 0.5N Sodium Hydroxide (NaOH)
- 1% Sodium Hydroxide (NaOH)
- Hydrochloric acid, Concentrated (HCl)

7. Standards Preparation:

- Obtain reference material from the International Humic Substances Society.
- Use the reference material as received. Keep covered.

8. Sample Preservation and Storage:

- Store solid fertilizer samples in glass jars.
- Store liquid fertilizer samples in plastic bottles.

9. Test Sample Preparation:

- No additional test sample preparation is needed.
- Tumble the glass jar to mix the solid fertilizer sample prior to weighing.
- Shake the plastic bottle to mix the liquid fertilizer sample prior to weighing.

10. Instrument Calibration:

None

11. Analysis:

- 1) Weigh an amount of finely ground solid or well-mixed liquid fertilizer sample into a centrifuge bottle to give approximately 500 mg of dry humic acid precipitate. Record the weight.
- 2) Add 50 ml 0.5N NaOH. Cap and seal tightly.
- 3) Shake on mechanical shaker for 1.5 hours for solid samples or 30 minutes for liquid samples.
- 4) After shaking, rinse the cap with 1% NaOH.
- 5) Centrifuge for 20 minutes at -2000 rpm.
- 6) Decant the supernatant into a second, previously weighed centrifuge bottle.
- 7) Add 5 – 10 ml 1% NaOH to the first bottle, shake vigorously, and centrifuge.
- 8) Combine this second supernatant with the first by decanting into the second centrifuge bottle. Discard the first bottle with the precipitate.
- 9) To the combined extracts in the second bottle, add enough concentrated HCl (approximately 10 ml) to lower the pH to s 1.
- 10) Centrifuge the sample for 20 minutes at - 2000 rpm.
- 11) Carefully decant and discard the liquid.
- 12) Add 25 ml distilled H₂O previously adjusted to pH s 1 (with HCl) to the bottle, cap and shake vigorously to free all precipitate from the bottom, and centrifuge.
- 13) Carefully decant and discard the liquid.
- 14) Repeat Steps 11.12 - 11.13 one final time.
- 15) Dry the bottle with humic acid overnight at 100-110°C
- 16) Cool in a desiccator (2–3 hours) and weigh.

12. QA/QC:

Run a reference material obtained from the International Humic Substances Society or a well-characterized humic acid sample as a check sample. For the International Humic Substances Society Reference Material, an acceptable recovery is 90%. For a well characterized humic acid sample, an acceptable recovery is $\pm 10\%$ of the average value (based on a minimum of 10 results).

The minimum reporting limit for solid samples is 0.5%, and for liquid samples is 0.05%.

13. Calculations:

% Humic acid= "Weight of Dried Precipitate"/ "Sample weight" X 100

14. Discussion and References:

- 1) John Husler, University of New Mexico, Department of Geology, Albuquerque, New Mexico
- 2) A.L. Page, Methods of Soil Analysis Part 2, American Society of Agronomy, Inc., Madison, Wisconsin, 1982
- 3) R.S. Swift, Methods of Soil Analysis Part 3, American Society of Agronomy, Inc., Madison, Wisconsin, 1996

Humic Acid Method
Revision 2
Revision Date:03/11/2009
Original Date: 09/13/1983

State of California
DEPARTMENT OF FOOD AND AGRICULTURE
1220 N STREET
SACRAMENTO CA 95814-5621
916-445-0444

GRAY DAVIS, Governor

**MANUFACTURERS AND DISTRIBUTORS OF HUMIC ACID PRODUCTS
REGARDING ACCEPTABLE PRODUCT CLAIMS FOR HUMIC ACIDS**

The Department of Food and Agriculture has received numerous inquiries from humic acid manufacturers and distributors regarding label statements and product claims. We would like to take this opportunity to clarify the acceptable labeling statements for humic acid products.

At this time, the Department only allows one product claim for humic acid: "Humic Acid may aid in the uptake of micronutrients". The statement "NOT A PLANTFOOD INGREDIENT" in all capital letters is also required on the product label. The Department has not received any efficacy data that substantiates additional or expanded product claims for humic acids.

We are aware that several companies have made expanded product claims for humic acids in brochures and advertisements. It is a violation of the Fertilizing Materials Law and Regulations to make any product claim that is not approved on the registered label. Companies that make expanded claims will be subject to enforcement action.

If your company is considering an efficacy study for the purpose of expanding your product label claims for humic acid products, we ask that you submit an experimental protocol to the Department prior to conducting the study. While this is not mandatory, it allows our staff to review the experimental design and resolve any concerns before you undertake the study.

If you have any questions regarding fertilizing material product registration, please call and ask to speak to a fertilizing material registration specialist as the above number.

Sincerely,

Steven D. Wong, Branch Chief
Agricultural Commodities and Regulatory Services

Original Date of this notice: January 3, 1988

Updated: February 13, 2003

March 15, 2009

State of California
Arnold Schwarzenegger, Governor
California Department of Food and Agriculture
A.G. Kawamura, Secretary

NOTICE TO FERTILIZER MANUFACTURERS WITH PRODUCTS CONTAINING HUMIC ACID

The California Department of Food and Agriculture (CDFA) Fertilizing Materials Inspection Program is responsible for sampling fertilizing materials in the State of California. Over the past 18 months, the program discovered that over half of the humic acid products were deficient from the manufacturer's guaranteed analysis and in violation of the California Fertilizing Materials Law and Regulations code 14682(b). CDFA allows a 10% variance for humic acids; therefore, solid and liquid humic acid products must meet 90% of the label guarantee. The program has recognized that some companies maybe using testing methods that are not consistent with the CDFA method for humic acid analysis.

In an attempt to standardize the humic acid testing method, CDFA is recommending the following:

Institute an in-house or third party laboratory test that is consistent with the method used by CDFA for any product(s) that guarantee humic acid. A copy of the method used by CDFA is included with this letter. CDFA uses and recommends standards available from the International Humic Substances Society to determine the accuracy of the method.

The program acknowledges that products may require re-labeling, new registration or reformulation to accurately reflect the humic acid guarantee. As a result, CDFA has instituted a six-month grace period on all humic acid products. **All products with a humic acid guarantee must be in compliance by September 1, 2009.** Violations for humic acid after September 1, 2009, may be subject to immediate quarantine of product inventory or cancellation of the fertilizer license.

Please contact Dr. Amadou Ba, Research Manager at the number below if you have any questions.

Sincerely,
Asif Maan, Ph.D.
Branch Chief
Fertilizing Materials Inspection Program
Feed, Fertilizer, Livestock Drugs and Egg Regulatory Services

Inspection Services,
1220 N Street, Sacramento, California, 95814
Telephone: 916.445.0444; Fax: 916.445.2171; www.cdfa.ca.gov