### **National Organic Program Petition**

### **CBI-Deleted Copy**

Submitted by SHAC Environmental Products Inc. PO Box 73 Medicine Hat, Alberta, Canada T1A 7E5 Phone: 403.528.4446 Toll-free: 888.533.4446 Website: <u>www.shac.ca</u> Email: <u>office@shac.ca</u>

Submitted on June 22, 2011

**Confidential Business Information (CBI) Statement** 

The sections designated as CBI-deleted within this petition are limited solely to the product manufacturing process, which is considered a 'trade secret'. This process, in its entirety, is considered confidential in nature, and has been marked CBI-deleted in this copy. The enclosed appendices do not contain confidential business information.

### Item A - Section for petitioned inclusion on the National List:

Synthetic substances allowed for use in organic crop production, 205.601

### Item B - Petition Data:

- The Substance's Chemical and Common Names: For the purposes of this petition, the substance is defined as Humic Acid Derivatives – Hydrogen Peroxide extracted. The common name of the product, as manufactured, is liquified oxidized lignite. Oxidized lignite is also known as leonardite.
- 2. The Manufacturer's Contact Information:

SHAC Environmental Products Inc. Mailing Address: PO Box 73 Medicine Hat, AB Canada T1A7E5 Physical Address: 35, 2269 2<sup>nd</sup> Ave Dunmore, AB Canada T1B0K3 P: 403.528.4446 / 888.533.4446 F: 403.529.9334 E: office@shac.ca

3. The Intended Uses of the Substance:

All intended uses: Soil Amendment, Livestock Management Tools and Production Aids, Livestock Feed Ingredient.

Organically certified intended uses: Soil Amendment.

4. List of Intended Uses:

The petitioned material is currently being used in a variety of markets (non-organic) as outlined below. Please note that the product names identified below reflect identical product formulations, marketed under different trade names for specific intended uses.

- a. **Soil Amendment:** Not being marketed or sold at this time. Currently seeking registration as a soil amendment with various state departments of agriculture under the trade name SHAC *Revitagro* as a humic acid based amendment for turf, soil and foliar applications.
- b. Livestock Management Tools and Production Aids: currently marketed in Canada, US and Mexico as a water treatment product and as a manure management aid under the trade names SHAC *Ponder* and SHAC *Manure Digester* respectively. SHAC *Ponder* is intended for the reduction of odors, turbidity and organic solids in water bodies such as reservoirs, ponds and farm dugouts and is NSF certified Std.61 (see Appendix 1). SHAC *Manure Digester* is intended for the reduction of odors, ammonia gases, and organic solids in livestock manure handling and storage systems.
- c. Livestock Feed Ingredient: Not currently marketed in US (no FDA/AAFCO approval), but is sold under the trade name SHAC *Feed Additive for Odour Control in Swine* in Canada (Canadian Food Inspection Agency Registration No. 480549 and 480573)

# The primary intention of this petition is to achieve 'synthetic - allowed' status for the petitioned material as a soil amendment, in order to be eligible for organic certification for the amendment product, Revitagro.

Labels for each of the above mentioned products, which specify rate and methods of application, are enclosed in Appendix 2. Please note that the SHAC Revitagro label lists only CDFA-method

analyzed humic acid. However, it is common for manufacturers of humic acid products to analyze using a variation of the Mehlich method, which quantifies all humic acids (derivatives).

5. Source of Substances and Manufacturing Process:

All SHAC products are manufactured identically; and are packaged and labeled differently for marketing purposes only. The final product contains only oxidized lignite (leonardite) and water. The following manufacturing process contains proprietary information regarding the production of SHAC products.

Source of Ingredients and Suppliers:

- Product Name/Description: Black Earth Mini Granule (CAS# 129521-66-0) (also known as: humalite, oxidized lignite, leonardite, oxidized sub-bituminous coal) Supplier: Black Earth Humic LP
- 2. Product Name/Description: Cypress Spring Water (non-chlorinated) Supplier: Shortgrass Ranch #704869 Alberta Ltd.
- 3. Product Name/Description: 35% Hydrogen Peroxide Food Grade (CAS# 7722-84-1 ) Supplier: Canada Colors and Chemicals Ltd.

The oxidized lignite supplier, Black Earth Humic LP, periodically analyzes the material at the supplier's source for consistency and quality control purposes using internal quality criteria. The company has access to the oxidized lignite from several mines which all draw from the same coal field spanning the Alberta and Saskatchewan border in Canada. The oxidized lignite is ground at the mining site to produce uniform homogenous granules. It is then packaged for shipment and delivered to the SHAC manufacturing facility.

The spring water supplier (#704869 Alberta Ltd.) provides water from a natural spring in the Cypress Hills of southeast Alberta, Canada. It is considered high quality drinking water and is marketed for human consumption. The water is transported in a stainless steel tank truck and delivered to the SHAC manufacturing facility.

The food grade hydrogen peroxide is supplied by Canada Colors and Chemicals Ltd. 50% hydrogen peroxide is manufactured by FMC of Canada Ltd. and then transported in bulk trucks to the Canada Colors and Chemicals Ltd. plant located in Leduc, Alberta. The hydrogen peroxide (50%) is then diluted to 35% hydrogen peroxide product at the plant in Leduc. The 35% hydrogen peroxide is then stored in drums and delivered to the SHAC manufacturing facility.

It should be noted that the hydrogen peroxide is a strong oxidizer and decomposes completely during manufacturing, resulting in the formation of oxygen and water. The final product has been tested by Alpha Laboratory Services Ltd. There were no detectable traces of hydrogen peroxide present in the final product, and therefore it is not considered an ingredient in the final product. Hydrogen peroxide analytical results are enclosed in this petition package in Appendix 3.

### Manufacturing Process:

**CBI** Deleted Copy

CBI Deleted Copy

- 6. Previous Reviews: See Appendix 4 for copies of OMRI (Organic Materials Review Institute) review letters.
- 7. Current Registrations: No EPA or FDA registrations exist for the products. While in the process of registering the substance with various state departments of agriculture, only the Oregon Department of Agriculture registration has been completed. Ponder is NSF certified to standard 61 for use in potable water intended for human consumption; and SHAC Feed Additive is registered with the Canadian Food Inspection Agency (CFIA) for use in complete feed rations.
- CAS Numbers: The CAS numbers for the individual ingredients are listed in Section 5 (Sources of Substances and Manufacturing Process). A substance/product-specific CAS number does not exist.
- 9. The Substance's Physical Properties and Chemical Mode of Action

The petitioned material is composed of liquefied oxidized lignite; is dark brown in colour and contains both dissolved and suspended oxidized lignite particles. See Appendix 5 for copies of laboratory analysis reports for various chemical and physical parameters including: Metals analysis (ICP), PAH analysis, Humic Acids analysis, CHNOS ash analysis (analyzed dry weight then converted), Particle size analysis, and physical product data.

a. Chemical interaction with other substances:

Humic acid derivatives (and oxidized lignite in general) act by various mechanisms including the following: ion-exchange, chelation, sorption, and the formation of complexes; and as a result may interact with a multitude of elements and compounds in a variety of ways. As the specific molecular structure of humic acid derivatives is complex and variable in nature, it is difficult to predict all possible interactions. However, it is generally recognized that various inorganic minerals and certain organic compounds may become complexed by humic acid derivatives. In addition, heavy metals may become complexed (and therefore less bio-available) within an environment containing humic acid derivatives.

Chemical Mode of Action between the Oxidized Lignite, water and Hydrogen Peroxide to create substance defined as *Humic Acid Derivative – Hydrogen Peroxide extracted*: During formulation of the petitioned substance, the hydrogen peroxide aggressively degrades into water and oxygen in the presence of the naturally occurring oxidized lignite and physically agitates the large lignite particles during the reaction, resulting in smaller-sized lignite particles.

Hydrogen peroxide decomposition (degradation) may be catalyzed by many substances, including most of the transition metals and their compounds. Hydrogen peroxide used in the SHAC manufacturing process encounters various trace metals as it contacts the oxidized lignite and decomposes exothermically into water and oxygen gas spontaneously.

$$2 \text{ H}_2\text{O}_2 \rightarrow 2 \text{ H}_2\text{O} + \text{O}_2$$

In the final product, 14 transition metals (such as Manganese, Copper, and Zinc – see metals analysis in Appendix 5 for complete list) were detected in varying quantities as summarized in the enclosed total metals analysis. These metals act as catalysts rather than reactants. While a chemical reaction converts reactants to products (reactants are consumed and products are produced), a catalyst is neither consumed nor produced as a result of the chemical reaction. Therefore, these metals are not included in the reaction formula shown above.

The rate of decomposition is also dependent on the temperature and concentration of the peroxide, as well as the pH and the presence of impurities and stabilizers. The warm temperature of the water, concentration of the peroxide, and the presence of catalytic metals in the oxidized lignite are all factors which contribute to the aggressive rate of hydrogen peroxide decomposition during the manufacturing process.

To summarize, the hydrogen peroxide decomposes rapidly (into water and oxygen) resulting in oxidation and aggressive agitation of the product, thereby reducing the particle size of the oxidized lignite granules, as well as dissolving the humic acid derivative fractions that are soluble under acidic conditions. The final product has no detectable traces of hydrogen peroxide, but rather contains only oxidized lignite (in solution and suspension), and water.

- b. Toxicity: Non-toxic under practiced use conditions. Environmental Persistence: Oxidized lignite is generally considered resistant to further short-term degradation.
- c. Environmental Impacts from Use/Manufacture: Minimal.
- d. Effects on Human Health: Unknown unstudied. However, as mentioned previously, the Ponder product is NSF certified to Standard 61 for use in potable water intended for human consumption.
- e. Effects on soil organisms, crops or livestock: Humic acids may have a bio-stimulatory effect on micro-organisms; act as amendments in soil/crop conditions; and may reduce ammonia volatilization in livestock applications, thereby improving overall health/environmental conditions within barn settings. It should be noted however, that the SHAC Feed Additive product is not recommended for use when medicating, as the humic acids may bind/complex certain medications/compounds, thereby reducing effectiveness of the medication.
- 10. Safety Information: Please refer to Appendix 6 for product Material Safety Data Sheets. A substance report from the National Institute of Environmental Health Studies does not exist for this product.
- 11. Research Reviews / Information:

Please refer to Appendix 7 for the NOSB review of humic acid derivatives dated 1996 as this substance is most closely related to the petitioned substance defined as Humic Acid Derivatives – Hydrogen Peroxide extracted. There are no NOP/NOSB reviews on the petitioned substance specifically, to our knowledge.

Please refer to Appendix 8 for additional research information pertaining to the use of oxidized lignite (leonardite) in crop production.

- 12. Petition Justification Statements:
  - Why the synthetic substance in necessary: Most oxidized lignite/humic acid products in liquid form are formulated by dissolving the humic acid derivatives. This is generally achieved by increasing the pH (usually between 10-12) through the addition of sodium hydroxide or potassium hydroxide to produce humates (the salts of humic acids). The difference with the petitioned formulation is that the product contains very small particle size (<200 mesh) *suspension* of humic acids, in their acid form, rather than dissolved humic acids in humate form; as well the soluble derivative fractions in *solution*.

In many applications of such products intended for agricultural use, consumer perception of high pH products is not ideal. The pH of the petitioned material falls within the range of 2.7-3.2 (close to pH of vinegar) so will not present any concern regarding alkalinity to concerned end users.

The same can be said for consumer perception of the addition of salts. Some landowners may not wish to add an amendment product that has been augmented with alkaline materials or 'salts'. There is also concern with the application of humic acid on a land application basis when applied with acid fertilizers, which in some cases may cause the pH of the humate product to decrease and the humic acids to come out of solution. If this occurs, blockages may become a problem. The petitioned material may be applied in conjunction with either alkaline or acidic fertilizers, without concern regarding further precipitation of particles. It should be noted that using SHAC products in conjunction with alkali fertilizers may result in dissolving the small suspended humic acid particles.

- Alternatives: To our knowledge, there is no equivalently formulated product on the market, as our process is proprietary. All other liquefied oxidized lignite/humic acid based products that we have encountered on the market have been humic acid alkali-extraction products.
- **Benefits:** A non-synthetic substance equivalent is not available in liquid form, but rather in powder or granular form. The only liquid forms of oxidized lignite/humic acid known to us are synthetic, and formulated by alkali extraction. In liquid form, the product provides the properties of oxidized lignite/leonardite (as source of humic acids), in a medium that is simple to apply as outlined in the *Intended Uses* section of this petition. For example, a liquid soil amendment product is simple to apply using existing irrigation and fertilizer spraying equipment. Another example: the addition of a liquid product to a farm dugout or a manure pit enables the product to circulate without the need for complicated application equipment; whereas non-synthetic powdered oxidized lignite tends to float on the surface and agglomerate, and granular oxidized lignite simply sinks directly to the bottom.

The differences between the SHAC process and alkali-extraction process for liquifying humic acids, is detailed as follows: SHAC's liquified oxidized lignite is unique in that the product is liquefied using hydrogen peroxide as an oxidizer and catalyst in the aggressive agitation and cleaving of lignite particles into smaller particle sizes. The pH of the source material remains acidic rather than becoming alkaline. The SHAC process does not introduce any additional alkaline metals or 'salts' into the material. And lastly, the hydrogen peroxide used during manufacture is consumed completely, thereby decomposing/degrading into water and oxygen, with no residual remaining.

# Appendix 1

 $\left( \begin{array}{c} \end{array} \right)$ 

(....)

6

NSF Certificate for SHAC Ponder

.

NSF International

Ę.

RECOGNIZES

SHAC ENVIRONMENTAL PRODUCTS INC. Facility: DUNMORE, ALBERTA, CANADA

AS COMPLYING WITH NSF/ANSI 61 AND ALL APPLICABLE REQUIREMENTS. PRODUCTS APPEARING IN THE NSF OFFICIAL LISTING ARE AUTHORIZED TO BEAR THE NSF MARK.







Certification Program Accredited by the Standards Council of Canada

This certificate is the property of NSF International and must be returned upon request. For the most current and complete information, please access NSF's website (www.nsf.org).

X David Purkiss, General Manager Water Distribution Systems

January 12, 2009 Certificate# C0023333 - 01

Appendix 2

(

¥----

SHAC Product Labels

#### EASY TO USE

#### • INVERT & SHAKE WELL BEFORE EACH USE

• DO NOT ALLOW PRODUCT TO FREEZE

#### • IF SOLIDS REMAIN RINSE CONTAINER WITH WATER

### DO NOT APPLY OR MIX WITH CHEMICALS

SHAC REVITAGRO is a humic acid based amendment that has been developed for turf, soil and foliar applications.

### DIRECTIONS FOR USE:

**REVITAGRO** contains oxidized lignite (source of humic acids) and is to be used only as recommended as an amendment for turf, soil and foliar applications. Always invert and shake container thoroughly before dispensing. It may be necessary to dilute product with water prior to application in order to achieve even coverage.

Apply 1-2 Litres (1-2 qt.) of *REVITAGRO* per 2000 m<sup>2</sup> (½ acre). Dilute with water and apply as desired to achieve the required coverage. A suggested dilution rate of 1 L (1 qt.) per 25 to 55 gals may be used as a guideline. A higher dilution rate (1L per 50-55 gals) should be used for foliar applications.

**REVITAGRO** may be applied using existing irrigation system or tank sprayer to achieve even coverage. If using a hose-end spray applicator, fill canister with required amount of partially diluted **REVITAGRO**, and spray consistently to achieve even coverage. If applying with sprayers containing high mesh screens (>100 mesh), remove screens prior to product use. Do not apply or mix **REVITAGRO** with chemical products such as pesticides or herbicides.

This jug will typically cover between 2 ½ - 5 acres. For small applications (yards, residential gardens), *REVITAGRO* is also available in 1 litre (1 qt.) bottles (check availability in your region).

**REVITAGRO** is not a fertilizer or plant food. For best results, applications should be made prior to regular fertilizer applications or once every 8 weeks during the growing season. For applications made by turf-care professionals (or other specialized applications), the product distributor may be contacted for site-specific application instructions.

### Guaranteed Analysis

### CONTAINS NON-PLANT FOOD INGREDIENTS:

Active Ingredients: 1.3 % Humic Acid (derived from Leonardite) Inert Ingredients: 98.7%

**REVITAGRO** is a liquid-suspension product and contains only water and oxidized lignite from one of the highest quality sources in North America; and has not been chemically altered by alkali extraction.

pH: 2.5-3.4 Density: 1.1 Kg per litre at 20°C / 10.7 lbs per gal at 68°F

Information regarding the contents or levels of metals in this product is available on the Internet at: http://www.aapfco.org/metals.htm

Neither the manufacturer nor distributor express or imply any warranty, or shall be liable for any damage caused by this product due to misuse, mishandling, or any application not specified on the label.

This product is manufactured and distributed by:



35, 2269 2 AVE DUNMORE ALBERTA, CANADA T1B 0K3 PO BOX 73 MEDICINE HAT ALBERTA, CANADA T1A 7E5 (mailing address) Toll Free: 1-888-533-4446 www.shac.ca



### SHAC Revitagro™ Humic Acid Amendment

# SHAC Revitagro is a humic acid based amendment that has been developed for turf, soil and foliar applications.

Easy to mix and apply - Contains no chemicals - Source of Humic Acids

### Directions for use

SHAC Revitagro contains oxidized lignite (source of humic acids) and is to be used only as recommended as an amendment for turf, soil and foliar applications. Always invert and shake container thoroughly before dispensing. It may be necessary to dilute product with water prior to application in order to achieve even coverage.

**Lawn Applications:** Apply 500 ml (½ qt.) per 2500 ft<sup>2</sup>. Dilute with water and apply as desired to achieve the required coverage. Apply prior to regular fertilizer applications in the spring, summer and fall.

**Greenhouse Applications:** Apply 175 ml (6 oz.) of SHAC Revitagro to the existing irrigation system (diluted in a minimum of 6 gals of water) per 4000 ft<sup>2</sup>. Apply every 4 weeks during growing season.

**Plants/Shrubs/Gardens and Foliar Applications:** dilute 1 tsp (5 ml) of Revitagro in 1 L (qt.) of water and apply as a watering solution or spray on plants and surrounding soil for foliar applications. Apply every 4 weeks during growing season.

SHAC Revitagro is not a fertilizer or plant food. Do not apply or mix Revitagro with chemical products such as pesticides or herbicides. SHAC Revitagro may be applied using a portable tank sprayer to achieve even coverage. If using a hose-end spray applicator, fill canister with required amount of partially diluted Revitagro, and spray consistently to achieve even coverage. If applying with sprayers containing high mesh screens (>100 mesh), remove screens prior to product use.

Do not apply or mix with chemicals. Do not allow product to freeze.

### Guaranteed Analysis by weight (as is): CONTAINS NON-PLANT FOOD INGREDIENTS

Humic Acids (all derivatives): 6.4% Derived from oxidized lignite source in Alberta, Canada Humic Acid (single derivative via CDFA method): 1.3%

Other Determinable Non-Plant Food Ingredients (water): 93.56%

Revitagro is a liquid-suspension product and contains only water and oxidized lignite from one of the highest quality sources in North America; and has not been alkali extracted.

pH: 2.5-3.4 Density: 1.1 Kg per litre at 20°C / 10.7 lbs per gal at 68°F

Information regarding the contents or levels of metals in this product is available on the Internet at: http://www.aapfco.org/metals.htm

Neither the manufacturer nor distributor express or imply any warranty, or shall be liable for any damage caused by this product due to misuse, mishandling, or any application not specified on the label.

### 1 Litre / 1 qt. LIQUEFIED OXIDIZED LIGNITE

This product is manufactured and distributed by: SHAC Environmental Products Inc. 35, 2269 2<sup>nd</sup> Ave Dunmore, Alberta, Canada T1B 0K3 PO Box 73 Medicine Hat, Alberta, Canada T1A 7E5 (mailing address) Toll Free: 1-888-533-4446 www.shac.ca

### SHAC Revitagro™ Humic Acid Amendment

# SHAC Revitagro is a humic acid based amendment that has been developed for turf, soil and foliar applications.

Easy to mix and apply - Contains no chemicals - Source of Humic Acids

#### **Directions for use**

SHAC Revitagro contains oxidized lignite (source of humic acids) and is to be used only as recommended as an amendment for turf, soil and foliar applications. Always invert and shake container thoroughly before dispensing. It may be necessary to dilute product with water prior to application in order to achieve even coverage.

Apply 1-2 Litres (1-2 qt.) of Revitagro per 2000 m<sup>2</sup> ( $\frac{1}{2}$  acre). Dilute with water and apply as desired to achieve the required coverage. A suggested dilution rate of 1 L (1 qt.) per 25 to 55 gals may be used as a guideline. A higher dilution rate (1L per 50-55 gals) should be used for foliar applications.

Revitagro may be applied using existing irrigation system or tank sprayer to achieve even coverage. If using a hose-end spray applicator, fill canister with required amount of partially diluted Revitagro, and spray consistently to achieve even coverage. If applying with sprayers containing high mesh screens (>100 mesh), remove screens prior to product use. Do not apply or mix Revitagro with chemical products such as pesticides or herbicides.

This jug will typically cover between 2 ½ - 5 acres. For small applications (yards, residential gardens), Revitagro is also available in 1 litre (1 qt.) bottles (check availability in your region).

SHAC Revitagro is not a fertilizer or plant food. For best results, applications should be made prior to regular fertilizer applications or once every 8 weeks during the growing season. For applications made by turf-care professionals (or other specialized applications), the product distributor may be contacted for site-specific application instructions.

### Do not apply or mix with chemicals. Do not allow product to freeze.

### Guaranteed Analysis by weight (as is): CONTAINS NON-PLANT FOOD INGREDIENTS

Humic Acids (all derivatives): 6.4% Derived from oxidized lignite source in Alberta, Canada Humic Acid (single derivative via CDFA method): 1.3%

Other Determinable Non-Plant Food Ingredients (water): 93.6%

Revitagro is a liquid-suspension product and contains only water and oxidized lignite from one of the highest quality sources in North America; and has not been chemically altered by alkali extraction.

pH: 2.5-3.4 Density: 1.1 Kg per litre at 20°C / 10.7 lbs per gal at 68°F

Information regarding the contents or levels of metals in this product is available on the Internet at: http://www.aapf<u>co.org/metals.htm</u>

Neither the manufacturer nor distributor express or imply any warranty, or shall be liable for any damage caused by this product due to misuse, mishandling, or any application not specified on the label.

### 10 Litre / 2.64 US Gal LIQUEFIED OXIDIZED LIGNITE

This product is manufactured and distributed by: SHAC Environmental Products Inc. 35, 2269 2<sup>nd</sup> Ave Dunmore, Alberta, Canada T1B 0K3 PO Box 73 Medicine Hat, Alberta, Canada T1A 7E5 (mailing address) Toll Free: 1-888-533-4446 www.shac.ca



## SAFE & EASY TO USE

#### • INVERT & SHAKE WELL • IF SOLIDS REMAIN RINSE CONTAINER WITH WATER • POUR IN ONE LOCATION OF THE BODY OF WATER • DO NOT ALLOW PRODUCT TO FREEZE • THIS PRODUCT IS NOT AN ALGAECIDE · DO NOT USE CHEMICALS WITH THIS PRODUCT

### APPLICATION RATES - For Large Ponds

| 1 <sup>st</sup> Year                    |  |   | 2 <sup>nd</sup> Year & Bey   | /ond   |
|---|--|---|--|--|
| Type of<br>Reservoir                    | First Time<br>Treatment<br>Rate<br>PONDER (L)<br>per<br>500,000<br>Imp.gal | Maintenance<br>Treatment<br>Rate<br>PONDER (L)<br>per<br>500,000<br>Imp.gal | Spring<br>Treatment<br>Rate<br>PONDER (L)<br>per<br>500,000<br>Imp.gal | Maintenance<br>Treatment<br>Rate<br>PONDER (L.)<br>per<br>500,000<br>Imp.gat |
| Run Off<br>Collection                   | 10 L   | 2 to 10 L   | 2 to 10 L  | 2 to 10 L  |
| Golf Course/<br>Stormwater<br>Reservoir | 20 L   | 10 L  | 10 L   | 10 L   |
| Irrigation<br>Filled                    | 10 L   | 2 to 10 L<br>every refill   | 2 to 10 L  | 2 to 10 L<br>every refill  |

### NTENANCE RATES - For Large Ponds

|   | A REAL PROPERTY AND ADDRESS OF THE OWNER |
|---|--|
| Site Conditions:  | Additional amount<br>of PONDER to apply<br>per site condition  |
| River / Creek filled  | 1 L  |
| Filled from standing water (e.g. slough)                    | 2 L  |
| Copper Sulfate eg. Blue Stone used historically             | 1 L  |
| Bermiess or ineffective berms                               | 1 L  |
| Direct cattle access  | 2 L  |
| intended for human consumption                              | 2 L  |
| Intended for animal consumption                             | 1L   |
| Stock fed fish  | 1 L  |
| Surrounded by trees and vegetation                          | 1 L  |
| Age of dugout or since last<br>dredged is more than 5 years | 1L   |

APPLY MAINTENANCE RATES EVERY 8 TO 10 WEEKS OF OPEN WATER

Maintenance rate is dependant on individual site conditions.



P.O. BOX 73 MEDICINE HAT ALBERTA, CANADA T1A 7E5 Toll Free / Sans frais : 1-888-533-4446 www.shac.ca

au moment de l'application du produit.



### • RETOURNER ET BIEN AGITER

SECURITAIRE ET FAGILE À UTILISER

· S'IL RESTE DES MATIÈRES SOLIDES, RINCER LE CONTENANT À L'EAU • VERSER À UN SEUL ENDROIT DU PLAN D'EAU · CE PRODUIT CRAINT LE GEL · CE PRODUIT N'EST PAS UN ALGICIDE

• NE PAS UTILISER DE PRODUITS CHIMIQUES DE CONCERT AVEC CE PRODUIT

### TAUX D'APPLICATION - Grands bassins

| 1ª année  |  |   | 2º année et au  | -delà   |
|---|--|---|---|---|
| Type de<br>bassin                               | Taux<br>d'application<br>initiale<br>PONDER (L) /<br>500 000<br>gallons<br>impériaux | Taux<br>d'application -<br>Maintenance<br>PONDER (L) /<br>500 000<br>gallons<br>impériaux | Taux<br>d'application -<br>Printemps<br>PONDER (L) /<br>500 000<br>gallons<br>impériaux | Taux<br>d'application -<br>Maintenance<br>PONDER (L) /<br>500 000<br>gallons<br>impériaux |
| Collecte des eaux<br>de ruissellement           | 10 L   | 2à10L   | 2 à 10 L  | 2 à 10 L  |
| Terrain de golf /<br>Bassin d'eaux<br>pluviales | 20 L   | 10 L  | 10 L  | 10 L  |
| Alimenté par<br>irrigation                      | 10 L   | 2 à 10 L<br>à chaque<br>remplissage   | 2 à 10 L  | 2 à 10 L<br>à chaque<br>remplissage   |

# TAUX D'APPLICATION - MAINTENANCE - Grands bassins

| Situation :   | Quantité additionnelle<br>de PONDER à appliquer<br>selon la situation |
|---|---|
| Alìmenté par une rivière ou un ruisseau                     | 1 L   |
| Eaux dormantes (p. ex. ; marécage)                          | 2 L   |
| Sulfate de cuivre utilisé par le passé                      | 1 L   |
| Sans berme ou avec bermes inefficaces                       | 1L  |
| Accès direct du bétail                                      | 2 L   |
| Destiné à la consommation humaine                           | 2 L   |
| Destiné à la consommation animale                           | 1L  |
| Bassin d'élevage de poisson                                 | 1L  |
| Entouré d'arbres et de végétation                           | 1 L   |
| Mare-réservoir ou travaux de dragage<br>de plus de cinq ans | 1L  |

APPLIQUER LES TAUX DE MAINTENANCE TOUTES LES 8 À 10 SEMAINES EN PÉRIODES D'EAUX LIBRES

Le taux est fonction des conditions qui prévalent



#### **• INVERT & SHAKE WELL BEFORE EACH USE** • DO NOT ALLOW PRODUCT TO FREEZE • IF SOLIDS REMAIN RINSE CONTAINER WITH WATER • DO NOT USE CHEMICALS WITH THIS PRODUCT

SAFE & EASY TO USE

# APPLICATION RATES: Hog Manure Pits

| Weaners a | nd Finishers: |          | Sows:     |                |          |
|-----------|---------------|----------|-----------|----------------|----------|
| Number of | Initial       | Monthly  | Number of | Initial        | Monthly  |
| Hogs      | Treatment (L) | Rate (L) | Hogs      | Treatment (L.) | Rete (L) |
| 50        | 1             | 0.5      | 50        | 2              | 1        |
| 100       | 2             | 1        | 100       | 4              | 2        |
| 200       | 4             | 2        | 200       | 8              | 4        |
| 1000      | 20            | 10       | 1000      | 36             | 18       |

### OTHER APPLICATIONS

|                             | Ra  |   |   |
|-----------------------------|---|---|---|
| Application                 | initial   | Maintenance   | How to Apply*   |
| Lagoon*                     | 40 L (10 US gai) MD per<br>375,000 L (100,000 US<br>gal) manure                 | 20 L (5 US gai) MD per<br>375,000 L (100,000 US<br>gei) manure every 6-8<br>weeks | Initially apply in 2 or 3 locations<br>directly in the lagoon.<br>Maintenance dose cen be applied<br>through barn flush<br>or drainage systems. |
| Barn<br>Cleaning<br>Systems | 1 L (1/4 US gal) MD in<br>20 L (5 US gal) water                                 | 1 L (1/4 US gal) MD in<br>20 L(5 US gal) water                                    | Pour into scraper pit<br>or along gutters.  |
| Manure:<br>before<br>piling | 1 L (1/4 US gal) MD in<br>20 L (5 US gal) water<br>per 75 m <sup>a</sup> manure | 1 L(1/4 US gal) MD in<br>20 L (5 US gal) water<br>per 75 m <sup>a</sup> manure    | Spray on<br>manure.   |
| Manure:<br>After<br>piling  | 1 L MD (1/4 US gal) in<br>20 L (5 US gal) water<br>per 75 m <sup>3</sup> manure | 1 L(1/4 US gal) MD in<br>10 L(2.5 US gal) water<br>per 75 m <sup>a</sup> manure   | Spray on pile and/or inject<br>into pile with spray wand<br>or probe.   |

\* NOTE: Inclusion rates can be modified based on individual site conditions. Some applications may require the addition of water in order to optimize solids reduction. Lagoon maintenance conditions are not necessary if barns are treated on a per head basis.

MANURE DIGESTER may be applied to manure storage systems/piles for other livestock as well as composting systems. Please visit www.shac.ca or contact us at 1-888-533-4446 for correct application rates and procedures.

### SÉCURITAIRE ET FACILE À UTILISER

### INVERSER ET AGITER VIGOUREUSEMENT AVANT CHAQUE USAGE PROTÉGER DU GEL • S'IL RESTE DES SOLIDES, RINCER LE CONTENANT À L'EAU

• NE PAS UTILISER DE PRODUITS CHIMIQUES DE CONCERT AVEC CE PRODUIT

## TAUX D'APPLICATION : Fosses à lisier de porc

| Porcs sevrés et de finition : |                                   | Trules :                             |                           |                                   |                                      |
|-------------------------------|-----------------------------------|--------------------------------------|---------------------------|-----------------------------------|--------------------------------------|
| Nombre                        | Traitement initial<br>(en litres) | Traitement<br>mensuel<br>(en litres) | Nombre                    | Traitement initial<br>(en litres) | Traitement<br>mensuel<br>(en litres) |
| 50<br>100<br>200<br>1 000     | 1<br>2<br>4<br>20                 | 0,5<br>1<br>2<br>10                  | 50<br>100<br>200<br>1 000 | 2<br>4<br>8<br>36                 | 1<br>2<br>4<br>18                    |

## AUTRES USAGES

|                                      | Tau   | лх*  | Ň  |
|--------------------------------------|---|--|--|
| Usage                                | Traitement initial  | Traitement de<br>maintenance   | Instructions*  |
| Lagune*                              | 40 litres (10 gal US)<br>par 375 000 litres<br>(100 000 gal US)<br>de fumier    | 20 litres (5 gal US)<br>par 375 000 litres<br>(100 000 gal US)<br>de fumier toutes les<br>6 à 8 semaines | Initialement, appliquer à 2 ou 3<br>endroits directement dans le lagune.<br>La dose de maintenance peut être<br>appliquée dans le système de<br>rinçage ou de vidange. |
| Systèmes<br>de nettoyage<br>d'étable | 1 litre (1/4 gat US)<br>par 20 litres<br>(5 gat US) d'eau                       | 1 litre (1/4 gai US)<br>par 20 litres<br>{5 gal US) d'eau  | Verser dans le fosse à<br>racloir ou le long des dalots.   |
| Fumier :<br>avant<br>l'entassement   | 1 litre (1/4 gal US) par 20<br>§itres (5 gal US) d'eau<br>pour 75 m³ de furnier | 1 litre (1/4 gal US) par 20<br>litres (5 gal US) d'eau<br>pour 75 m³ de fumier                           | Vaporiser sur le furnier.  |
| Fumier :<br>après<br>l'entassement   | 1 litre (1/4 gal US) par<br>20 litres (5 gal US) d'eau<br>pour 75 m³ de furnier | 1 litre (1/4 gal US) par<br>20 litres (5 gal US) d'eau<br>pour 75 m <sup>3</sup> de fumier               | Vaporiser sur le tas ou injecter<br>le produit dans celui-ci à l'aide<br>du tube de vaporisation.  |

\* NOTE : Les doses d'inclusion peuvent être modifiées en fonction de l'état de chaque site. Dans certains cas, l'ajout d'eau peut être nécessaire pour optimiser la réduction des solides. Dans les étables où les animaux sont traités individuellement, les instructions relatives au traitement de maintenance ne s'appliquent pas.





P.O. BOX 73 MEDICINE HAT ALBERTA, CANADA T1A 7E5 Toll Free / Sans frais : 1-888-533-4446 www.shac.ca

environmental products inc.



# SHAKE WELL BEFORE USE APPLICATION RATES

SHAC Liquid Feed Additive is a solution produced from oxidized bituminous coal. It has been developed as a feed additive for the purpose of reducing manure odours and harmful ammonia gas in swine facilities.

### **DIRECTIONS FOR USE**

• MIX THOROUGHLY TO ENSURE ALL SEDIMENT IN CONTAINER IS IN SUSPENSION.

• USE IMMEDIATELY OR RE-AGITATE BEFORE USING.

Results may vary depending on the type and amount of feed used; the health condition of the hog; and the farm management practices. The following application rates will produce excellent results in most cases.

### FOR GROWER / FINISHER HOGS:

Apply 250ml of SHAC Liquid Feed Additive to 1 tonne of complete feed ration. Blend thoroughly into each batch.

### FOR SOWS / BOARS / WEANERS:

Apply 500ml of SHAC Liquid Feed Additive to 1 tonne of complete feed ration. Blend thoroughly into each batch.

For best results, apply SHAC *Manure Digester* to pit or lagoon prior to starting the feeding program to reduce any solid build-up. See your local dealer for details.

Ensure that all hogs sharing common manure storage (pit or lagoon) are consuming feed treated with SHAC Liquid Feed Additive. Manure from untreated hogs will reduce the effect of the product.

Typically, odour/ammonia control can be expected within 2-6 weeks of use.

INGREDIENTS: Oxidized Bituminous Coal solution

DO NOT ALLOW PRODUCT TO FREEZE

10 Litres Density = 1.1kg per litre at 20°C The viscosity of this product will vary inversely with temperature. CFIA Reg. No. #480573



Registered by: P.O. Box 73, MEDICINE HAT ALBERTA, CANADA T1A 7E5 Toll Free: 1-888-533-4446 www.shac.ca



Appendix 3

(

t.

Analytical Results confirming the absence of Hydrogen Peroxide in SHAC Products



# ALPHA LABORATORY SERVICES LTD.

# Analytical and Consulting Services

17225 - 109 Avenue Edmonton, Alberta T5S 1H7 Phone: (780) 489-9100 Fax: (780) 489-9700

To: SHAC Environmental Products Inc. PO Box 73 Medicine Hat AB T1A 7E5 **TECHNICAL REPORT** 

File: Date: Client PO: Attention: 22909 November 2, 2004

Ashley Gavey

Project: Product Samples

|                   | Sample ID:    | SHAC<br>New  | SHAC<br>4 mo. |           |          |
|-------------------|---------------|--------------|---------------|-----------|----------|
|                   | Date Sampled: | Not Supplied | Not Supplied  | Date      | Analyst  |
| Parameter         | Unit          |              |               | Analyzed  | Initials |
| Hydrogen Peroxide | wt %          | <1           | <1            | Nov. 1/04 | B.L.     |



# ALPHA LABORATORY SERVICES LTD.

# Analytical and Consulting Services

17225 - 109 Avenue Edmonton, Alberta T5S 1H7 Phone: (780) 489-9100 Fax: (780) 489-9700

# **TECHNICAL REPORT**

To: SHAC Environmental Products Inc.

File: 22909

Project: Product Samples

Analysis Verified by:

Kenlicht

Lisa Reinbolt Supervisor

Report Authorized by:

Bob Lickacz, B.S., P.Biol President

Note: All samples will be disposed of 30 days after analysis. Please advise the laboratory if you require additional sample storage time.

Appendix 4

# **OMRI** Review Letters



PD Soc 11566, Eugene, Bregen 87440-3758 USA S41, 343 7600 - Soc 541, 343, 8971 9(fo@emii.org)

March 8, 2011

Ashley Gavey SHAC environmental products inc. PO Box 73 Medicine Hat, AB T1A 7E5 CANADA

### Dear Ashley Gavey:

The OMRI Review Panel has reviewed SHAC environmental products inc.'s products, SHAC® Revitagro (she-2363), and SHAC® Ponder (she-2361), and has recommended that they be *Prohibited* for use in organic production. This decision indicates that SHAC® Revitagro and SHAC® Ponder do not comply with the *OMRI Policy and Standards Manual*, which is based on the requirements of the USDA National Organic Program (NOP) Rule (7 CFR Part 205).

Specifically, the Review Panel determined that SHAC® Revitagro is prohibited because the reaction taking place between the hydrogen peroxide and Black Earth Mini Granule ingredients clearly results in a chemical breaking of bonds. The Panel reviewed the manufacturing process, and disagreed with the ascertain that the reaction strictly produces a mechanical change. Since this reaction is not one specifically allowed in the NOP rule, the Panel has determined that it is prohibited for use in organic crop production.

SHAC environmental products inc. can petition the NOP to have the prohibited substance considered for use in organic production. For information on the petition procedure, see the NOP website. You may also choose to reformulate your product to remove any prohibited substances, and submit a new product application and fee to OMRI for the review of a reformulated product at any time.

Please be advised that the OMRI Listed Seal and wording can not be used for this product. Any unauthorized use of the OMRI Listed Seal and name may result in legal action against the company that violates the OMRI Seal Use Policy.

Prohibited listings are circulated to subscribing certifiers and provided to other OMRI subscribers when requested. Please be aware that organic certification agents retain the right to make final certification decisions concerning use of products in organic production. These certifiers may choose not to recognize OMRI's recommendation. OMRI is not responsible for any losses that may occur as a result of the OMRI Prohibited Status of SHAC® Revitagro or SHAC® Ponder.

This letter serves as OMRI's Final Response Letter to SHAC environmental products inc. regarding the status of SHAC® Revitagro and SHAC® Ponder. If SHAC environmental products inc. wishes to rebut or appeal this decision, please refer to the "Decision Rebuttals, Appeals and Mediation" section of the OMRI Policy and Standards Manual. Please be advised that a notice of your decision to reformulate does not constitute a proper rebuttal. Thank you for your participation in the OMRI Review Program. Please contact me with any questions.

Sincerely,

Andria Schulze Product Review Coordinator (541)343-7600 x112 andrias@omri.org

| R    | ĒĈ              | en      | VEC               | )      |
|------|-----------------|---------|-------------------|--------|
|      |                 | 18      | 2011              |        |
| in m | w ize init init | ين ير م | . 305 423 425 ==- | na. 97 |

09ProhibitedNotice1F

she-2363



P.O. Box 11558, Eugene, Oregon 97440-3758 USA 541.343.7600 • fax 541.343.8971 info@omri.org

March 11, 2011

Ashley Gavey SHAC environmental products inc. PO Box 73 Medicine Hat, AB T1A 7E5 CANADA

### Dear Ashley Gavey:

The OMRI Review Panel has reviewed SHAC environmental products inc.'s product, SHAC® Manure Digester (she-2362), and has recommended that it be *Prohibited* for use in organic production. This decision indicates that SHAC® Manure Digester does not comply with the *OMRI Policy and Standards Manual*, which is based on the requirements of the USDA National Organic Program (NOP) Rule (7 CFR Part 205).

Specifically, the Review Panel determined that SHAC® Manure Digester is prohibited because the reaction taking place between the hydrogen peroxide and Black Earth Mini Granule ingredients clearly results in a chemical breaking of bonds. The Panel reviewed the manufacturing process, and disagreed with the ascertain that the reaction strictly produces a mechanical change. Since this reaction is not one specifically allowed in the NOP rule, the Panel has determined that it is prohibited for use in organic crop production.

SHAC environmental products inc. can petition the NOP to have the prohibited substance considered for use in organic production. For information on the petition procedure, see the NOP website. You may also choose to reformulate your product to remove any prohibited substances, and submit a new product application and fee to OMRI for the review of a reformulated product at any time.

Please be advised that the OMRI Listed Seal and wording can not be used for this product. Any unauthorized use of the OMRI Listed Seal and name may result in legal action against the company that violates the OMRI Seal Use Policy.

Prohibited listings are circulated to subscribing certifiers and provided to other OMRI subscribers when requested. Please be aware that organic certification agents retain the right to make final certification decisions concerning use of products in organic production. These certifiers may choose not to recognize OMRI's recommendation. OMRI is not responsible for any losses that may occur as a result of the OMRI Prohibited Status of SHAC® Manure Digester.

This letter serves as OMRI's Final Response Letter to SHAC environmental products inc. regarding the status of SHAC® Manure Digester. If SHAC environmental products inc. wishes to rebut or appeal this decision, please refer to the "Decision Rebuttals, Appeals and Mediation" section of the *OMRI Policy and Standards Manual.* Please be advised that a notice of your decision to reformulate does not constitute a proper rebuttal.

Thank you for your participation in the OMRI Review Program. Please contact me with any guestions.

Sincerely,

Andria Schulze Product Review Coordinator (541)343-7600 x112 andrias@omri.org she-2362



09ProhibitedNotice1F

# Appendix 5

# Chemical and Physical Properties – Laboratory Analysis

Chemical Properties: Metals, PAH, Humic Acids, CHNOS ash Analyses Physical Properties: Particle Size Analysis and Physical Product Data Sheet

Exova 7217 Roper Road NW Edmonton, Alberta T6B 3J4, Canada T: +1 (780) 438-5522 F: +1 (780) 438-0396 E: Edmonton ⊛exova.com W: www.exova.com

eport Transmission Cover Page



| Bill To:    | SHAC Environmental       | Project:   |                       | Lot ID:         | 797593       |
|-------------|--------------------------|------------|-----------------------|-----------------|--------------|
| Report To:  | SHAC Environmental       | ID:        | SHAC Routine Analysis | Control Number: | A176052      |
|             | P. O. Box 73             | Name:      |                       | Date Received:  | Apr 8, 2011  |
|             | Medicine Hat, AB, Canada | Location:  |                       | Date Reported:  | Apr 15, 2011 |
|             | T1A 7E5                  | LSD:       |                       | Report Number:  | 1421892      |
| Attn:       | Ashley Gavey             | P.O.:      |                       |                 |              |
| Sampled By: | A. Gavey                 | Acct code: |                       |                 |              |
| Company:    | SHAC                     |            |                       |                 |              |

| Contact & Affiliation | Address                                      | Delivery Commitments                                  |   |
|-----------------------|--|---|---|
| Ashlev Gavev          | , P. O. Box 73                               | On [Report Approval] send                             |   |
| SHAC Environmental    | Medicine Hat, Alberta T1A 7E5                | (COC, Test Report) by Email - Merge Reports           |   |
|                       | Phone: (403) 528-4446                        | On [Lot Approval and Final Test Report Approval] send |   |
|                       | Fax: (403) 529-9334<br>Email: ashley@shac.ca | (COC, Test Report, Invoice) by Post                   | м |

### Notes To Clients:

Particle Size by Microtrax analysis was performed by a subcontract laboratory. See attached 1 page report.

The information contained on this and all other pages transmitted, is intended for the addressee only and is considered confidential. If the reader is not the intended recipient, you are hereby notified that any use, dissemination, distribution or copy of this transmission is strictly prohibited. If you receive this transmission by error, or if this transmission is not satisfactory, please notify us by telephone.

| Exova<br>7217 Roper Road NW<br>Edmonton, Alberta<br>T6B SJ4, Canada | T: +1 (780) 438-5522<br>F: +1 (780) 438-0396<br>E: Edmonton & exova.com<br>W: www.exova.com |                          |                       |  | Exova                                  | in a substantia a substantia<br>bate a substantia a substantia a substantia<br>bate a substantia a substantia a substantia a substantia a substantia<br>bate a substantia a substantia a substantia a substantia a substantia<br>bate a substantia a substantia a substantia a substantia a substantia a substantia<br>bate a substantia a substantia a substantia a substantia a substantia a substantia<br>bate a substantia a substantia a substantia a substantia a substantia a substantia<br>substantia a substantia a substantia<br>substantia a substantia a substantia<br>substantia a substantia a substantia<br>substantia a substantia a substantia<br>substantia a substantia a su |
|---|---|--------------------------|-----------------------|--|--|--|
| ample Cust  | ody   |                          |                       |  |  |  |
| Bill To:<br>Report To:  | SHAC Environmental<br>SHAC Environmental  | Project:<br>ID:<br>Nomo: | SHAC Routine Analysis | Lot ID:<br>Control Number:                         | <b>797593</b><br>A176052               |  |
|   | P. O, Box 73<br>Medicine Hat, AB, Canada<br>T1A 7E5   | Location:<br>LSD:        |                       | Date Received:<br>Date Reported:<br>Report Number: | Apr 8, 2011<br>Apr 15, 2011<br>1421892 |  |
| Attn:   | Ashley Gavey  | P.O.:                    |                       |  |  |  |
| Sampled By:<br>Company:   | A. Gavey<br>SHAC  | Acct code:               |                       |  |  |  |

# Sample Disposal Date: May 15, 2011

All samples will be stored until this date unless other instructions are received. Please indicate other requirements below and return this form to the address or fax number on the top of this page.

|   | Extend Sample Storage Until   | (MM/DD/YY)  |
|---|---|---|
| · | The following charges apply to extended sample storage<br>Storage for an additional 30 days<br>Storage for an additional 60 days<br>Storage for an additional 90 days | :<br>\$ 2.50 per sample<br>\$ 5.00 per sample<br>\$ 7.50 per sample |
|   |   |   |
|   | Return Sample, collect, to the address below via.   |   |
|   | Greyhound   |   |
|   | DHL   |   |
|   | Purolator   |   |
|   | Other (specify)   |   |
|   |   | Name  |
|   |   | Company   |
|   |   | Address   |
|   |   | Phone   |
|   |   | Fax   |
|   |   | Signature   |
|   |   |   |
|   |   |   |

ę. •

Exova 7217 Roper Road NW Edmonton, Alberta T6B 334, Canada T: +1 (760) 438-5522 F: +1 (760) 438-0396 E: Edmontos@exova.com W; www.exova.com



### halytical Report

Bill To: SHAC Environmental Report To: SHAC Environmental P. O. Box 73 Medicine Hat, AB, Canada T1A 7E5 Attn: Ashley Gavey Sampled By: A. Gavey Company: SHAC Project: ID: Name: Location: LSD: P.O.: Acct code:

SHAC Routine Analysis

Lot ID: **797593** Control Number: A176052 Date Received: Apr 8, 2011

Date Received:Apr 8, 2011Date Reported:Apr 15, 2011Report Number:1421892

|                    |                 | Reference Number<br>Sample Date<br>Sample Time<br>Sample Location<br>Sample Description<br>Matrix | 797593-1<br>Apr 05, 2011<br>NA<br>SHAC041146<br>Water |         |         |                   |
|--------------------|-----------------|---|---|---------|---------|-------------------|
| Analyte            |                 | Units   | Results   | Results | Results | Nominal Detection |
| Metals Total       | <u></u>         |   |   |         |         |                   |
| Calcium            | Total           | mg/L  | 725   |         |         | 0.2               |
| lron               | Total           | mg/L  | 152   |         |         | 0.05              |
| Magnesium          | Total           | mg/L  | 105   |         |         | 0.1               |
| Manganese          | Total           | mg/L  | 14.6  |         |         | 0.005             |
| Potassium          | Total           | mg/L  | 27  |         |         | 0.4               |
| Silicon            | Total           | mg/L  | 93.0  |         |         | 0.05              |
| Sodium             | Total           | mg/L  | 264   |         |         | 0.4               |
| Sulfur             | Total           | mg/L  | 314   |         |         | 0.3               |
| Mercurv            | Total           | mg/L  | 0.0006  |         |         | 0.0001            |
| Aluminum           | Total           | mg/L  | 408   |         |         | 0,005             |
| Antimony           | Total           | mg/L  | 0.02  |         |         | 0.0002            |
| Arsenic            | Total           | mg/L  | 0.16  |         |         | 0.0002            |
| Barium             | Total           | mg/L  | 7.0   |         |         | 0.001             |
| Beryllium          | Total           | mg/L  | 0.05  |         |         | 0.0001            |
| Bismuth            | Total           | mg/L  | 0.24  |         |         | 0.0005            |
| Boron              | Total           | mg/L  | 15.6  |         |         | 0.002             |
| Cadmium            | Total           | mg/L  | 0.006   |         |         | 0.00001           |
| Chromium           | Total           | mg/L  | 0.26  |         |         | 0.0005            |
| Cobalt             | Total           | mg/L  | 0.09  |         |         | 0.0001            |
| Copper             | Total           | mg/L  | 0.5   |         |         | 0.001             |
| Lead               | Total           | mg/L  | 0.42  |         |         | 0.0001            |
| Lithium            | Total           | mg/L  | 0.3   |         |         | 0.001             |
| Molybdenum         | Total           | mg/L  | <0.1  |         |         | 0.001             |
| Nickel             | Total           | mg/L  | 0.29  |         |         | 0.0005            |
| Selenium           | Total           | mg/L  | 0.10  |         |         | 0.0002            |
| Silver             | Total           | mg/L  | 0.0161  |         |         | 0.00001           |
| Strontium          | Total           | mg/L  | 9.4   |         |         | 0.001             |
| Thallium           | Total           | mg/L  | <0.005  |         |         | 0.00005           |
| Tin                | Total           | mg/L  | 0.6   |         |         | 0.001             |
| Titanium           | Total           | mg/L  | 19.9  |         |         | 0.0005            |
| Uranium            | Total           | mg/L  | 0.12  |         |         | 0.0005            |
| Vanadium           | Total           | mg/L  | 0.67  |         |         | 0.0001            |
| Zinc               | Total           | mg/L  | 0.9   |         |         | 0.001             |
| Zirconium          | Total           | mg/L  | 3.2   |         |         | 0.001             |
| Physical and Aggre | nate Properties | 5   |   |         |         |                   |
| Polide             | Total           | mg/L  | 20500   |         |         | 5                 |

Terms and Conditions: www.exova.ca/terms&conditions

| Exova<br>7217 Roper Road NW<br>Edmonton, Alberta<br>T5B 3J4, Canada | T: +1 (780) 438-5522<br>F: +1 (780) 438-0396<br>E: Edmonton@exova.com<br>W: www.exoya.com                       |  |                     |  | Page 2 of 3<br><b>EXOVQ</b>  | Restances of the second |
|---|---|--|---------------------|--|--|--|
| nalytical Re  | port  |  |                     |  |  |  |
| Bill To:<br>Report To:<br>Attn:                                     | SHAC Environmental<br>SHAC Environmental<br>P. O. Box 73<br>Medicine Hat, AB, Canada<br>T1A 7E5<br>Ashley Gayey | Project:<br>ID: SH/<br>Name:<br>Location:<br>LSD:<br>P.O.: | AC Routine Analysis | Lot ID:<br>Control Number:<br>Date Received:<br>Date Reported:<br>Report Number: | <b>797593</b><br>A176052<br>Apr 8, 2011<br>Apr 15, 2011<br>1421892 |  |
| Sampled By:   | A. Gavey  | Acct code:   |                     |  |  |  |
| Company:  | SHAC  |  |                     |  |  |  |
| - <u>many - Pangasana ay</u> Salah                                  |   | Reference Number   | 797593-1            |  |  |  |
|   |   | Sample Date  | Apr 05, 2011        |  |  |  |
|   |   | Sample Time  | NA                  |  |  |  |
|   |   | Sample Location  |                     |  |  |  |
|   |   | Sample Description   | SHAC041146          |  |  |  |
|   |   | Matrix   | Water               |  |  |  |

Analyte Units Results
Subcontracted Analysis

Subcontractor Report Id Umicore

1

Anthony Weuman

Nominal Detection Limit

Results

Results

Approved by: Anthony Neumann, MSc Laboratory Operations Manager Exova 7217 Roper Road NW Edmonton, Alberta T6B 3J4, Canada

ethodology and Notes

T: +1 (780) 438-5522 F: +1 (780) 438-0396 E: Edmonlon@exova.com W: www.exova.com



#### Lot ID: 797593 Project: Bill To: SHAC Environmental SHAC Routine Analysis Report To: SHAC Environmental ID: Control Number: A176052 Name: Date Received: Apr 8, 2011 P. O. Box 73 Medicine Hat, AB, Canada Location: Date Reported: Apr 15, 2011 LSD: T1A 7E5 Report Number: 1421892 P.O.: Attn: Ashley Gavey Sampled By: A. Gavey Acct code: Company: SHAC

### **Method of Analysis**

| Method Name                        | Reference | Method  | Date Analysis<br>Started | Location       |
|------------------------------------|-----------|---|--------------------------|----------------|
| Mercury (Total) in water           | US EPA    | * Determination of Hg in Sediment by<br>Cold Vapor Atomic Absorption Spec,<br>245.5           | 11-Apr-11                | Exova Edmonton |
| Metals ICP-MS (Total) in water     | US EPA    | <ul> <li>Determination of Trace Elements in<br/>Waters and Wastes by ICP-MS, 200.8</li> </ul> | 08-Apr-11                | Exova Edmonton |
| Metals Trace (Total) in water      | АРНА      | <ul> <li>Inductively Coupled Plasma (ICP)<br/>Method, 3120 B</li> </ul>                       | 08-Apr-11                | Exova Edmonton |
| Solids (Total, Fixed and Volatile) | APHA      | * Total Solids Dried at 103-105'C, 2540<br>B  | 13-Apr-11                | Exova Edmonton |
| Sublet to Umicore                  | Ext. Lab  | See attached test report,   | 12-Apr-11                | Umicore        |
|                                    |           | * Reference Method Modified   |                          |                |

### eferences

l---

| APHA   | Standard Methods for the Examination of Water and Wastewater |
|--------|--|
| US EPA | US Environmental Protection Agency Test Methods              |

### Comments:

Particle Size by Microtrax analysis was performed by a subcontract laboratory. See attached 1 page report.

Please direct any inquiries regarding this report to our Client Services group. Results relate only to samples as submitted.

The test report shall not be reproduced except in full, without the written approval of the laboratory.

## **Particle Size Distribution**



| Sample Name: 797593-1 SHAC041146 |                 |                      | Analysed:          | April 15, 2011 10:28:05 |
|----------------------------------|-----------------|----------------------|--------------------|-------------------------|
| Run No. 95680                    | Measured by:    | Amy                  |                    | AM                      |
| Particle Name: Fraunhofer        | Accessory Name: | Hydro 2000S (A)      | Obscuration:       | 26.26 %                 |
| Particle RI: 0.000 Absorption: 0 | Analysis model: | General purpose      | Dispersant RI:     | 1.330                   |
| Dispersant Name: Water           | Size range:     | 0.020 to 2000.000 um | Weighted Residual: | 0.269 %                 |
| D(0.10) : 0.94 µm D(0.50) : 2.99 | μm D(0.90       | 0) : 14.36 µm D(0.9  | 5) : 26.23 μm D(1. | 00) : 111.20 µm         |

### Operator notes:



| S | Size (um) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | [ | Size (µm) | Volume In % |   | Size (µm) | Volume In % |
|---|-----------|-------------|-----------|-------------|-----------|-------------|---|-----------|-------------|---|-----------|-------------|
| F | 0.020     | 0.00        | 0.224     | 0.00        | 2.518     | 4.57        |   | 28.251    | 0.67        |   | 316.979   | 0.00        |
| 1 | 0.022     | 0.00        | 0.252     | 0.00        | 2.825     | 4.51        |   | 31.698    | 0.62        |   | 355.656   | 0.00        |
|   | 0.025     | 0.00        | 0.283     | 0.00        | 3.170     | 4.42        |   | 35.566    | 0.57        |   | 399.052   | 0.00        |
|   | 0.028     | 0.00        | 0.317     | 0.00        | 3.557     | 4.97        |   | 39,905    | 0.53        |   | 447,744   | 0.00        |
|   | 0.032     | 0.00        | 0.356     | 0,00        | 3,991     | 4.07        | 1 | 44.774    | 0.48        |   | 502.377   | 0.00        |
|   | 0.036     | 0.00        | 0.399     | 0.12        | 4.477     | 3.82        |   | 50.238    | 0.43        |   | 563.677   | 0.00        |
|   | 0.040     | 0.00        | 0.448     | 0.341       | 5.024     | 3.54        |   | 56.368    | 0.37        |   | 632,456   | 0.00        |
|   | 0.045     | 0.00        | 0.502     | 0.38        | 5.637     | 3 23        |   | 63.246    | 0.30        |   | 709.627   | 0,00        |
|   | 0.050     | 0.00        | 0.564     | 1 18        | 6.325     | 2 90        |   | 70.963    | 0.23        |   | 796.214   | 0.00        |
|   | 0.056     | 0.00        | 0.632     | 1.10        | 7.096     | 2.58        |   | 79.621    | 0.17        |   | 893.367   | 0.00        |
|   | 0.063     | 0,00        | 0.710     | 1.89        | 7,962     | 2.26        |   | 89.337    | 0.10        |   | 1002.374  | 0.00        |
|   | 0.071     | 0,00        | 0.796     | 2 27        | 8,934     | 1.97        |   | 100.237   | 0.07        |   | 1124.683  | 0.00        |
|   | 0.080     | 0.00        | 0,893     | 2.65        | 10,024    | 1.72        | ' | 112,468   | 0.00        |   | 1261.915  | 0.00        |
|   | 0.089     | 0,00        | 1.002     | 3.02        | 11.247    | 1.51        |   | 126.191   | 0.00        |   | 1415.892  | 0.00        |
|   | 0.100     | 0.00        | 1.125     | 3 37        | 12.619    | 1.33        |   | 141.589   | 0,00        |   | 1588.656  | 0.00        |
|   | 0.112     | 0.00        | 1.262     | 3.69        | 14.159    | 1 19        |   | 158.866   | 0.00        |   | 1782.502  | 0.00        |
|   | 0.126     | 0,00        | 1.416     | 3.05        | 15.887    | 1 07        |   | 178.250   | 0.00        | l | 2000.000  |             |
|   | 0.142     | 0.00        | 1.589     | 4 20        | 17.825    | 0.96        | 1 | 200.000   | 0,00        |   |           |             |
|   | 0.159     | 0.00        | 1.783     | 4 38        | 20,000    | 0.87        |   | 224.404   | 0.00        |   | 1         |             |
| 1 | 0,178     | 0.00        | 2.000     | 4.50        | 22.440    | 0.79        |   | 251.785   | 0.00        |   |           |             |
|   | 0.200     | 0.00        | 2.244     | 4.50        | 25.179    | 0.73        |   | 282,508   | 0.00        |   |           |             |
|   | 0.224     | 0.00        | 2.518     | 4.57        | 28,251    |             |   | 316.979   |             |   | 1         |             |

Malvern Instruments Ltd.

Maivern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : 34123-34

## **Report Transmission Cover Page**

| Bill To:<br>Report To:<br>Attn:<br>Sampled By:<br>Company: | SHAC Environme<br>SHAC Environme<br>P. O. Box 73<br>Medicine Hat, AB<br>T1A 7E5<br>Ashley Gavey<br>A. Gavey<br>Shac | ntal<br>ntal<br>, Canada  | Project:<br>ID:<br>Name:<br>Location:<br>LSD:<br>P.O.:<br>Acct code:                   | Shac Feed Additive Product  | Lot ID:<br>Approval Status:<br>Invoice Frequency:<br>COD Status:<br>Control Number:<br>Date Received:<br>Date Reported:<br>Report Number:             |  |   |
|--|---|---|--|---|---|--|---|
| Contact & Affilia<br>Ashley Gavey<br>SHAC Enviro           | tion<br>onmental  | Address<br>, P. O. f<br>Medicin<br>Phone:<br>Fax: (40<br>Email: a | 3<br>3ox 73<br>1e Hat, Alberta T1A<br>(403) 528-4446<br>03) 529-9334<br>ashley@shac.ca | Delivery Com<br>On [Report Ap<br>7E5 (COC, Test<br>On [Report Ap<br>(COC, Test<br>On [Lot Appro<br>(COC, Test | nitments<br>proval] send<br>Report) by Email - Merg<br>proval] send<br>Report) by Email - Merg<br>val and Final Test Repo<br>Report, Invoice, Test Re | e Reports<br>e Reports<br>ort Approval] send<br>eport) by Post | Ň |
| Carrie Larson<br>SHAC Enviro                               | onmental  | , Box 7;<br>Medicir<br>Phone:<br>Fax: (4)<br>Email: (             | 3<br>ne Hat, Alberta T1A<br>(800) 533-4446<br>03) 529-9334<br>carrie@shac.ca           | On [Lot Verific<br>7E5 (COA) by Er<br>On [Report Aj<br>(COC, Test<br>On [Report Aj<br>(COC, Test              | cation] send<br>nail - Multiple Reports<br>oproval] send<br>Report) by Email - Multi<br>oproval] send<br>Report) by Email - Multi                     | ple Reports  |   |

#### Notes To Clients:

Ashley was contacted and informed of the deviation due to her container that was sent in, she would like to go ahead with the analysis.

Dibenzo(a,I)pyrene analysis was performed by a subcontract laboratory. See attached 3 page report.

 Report was issued to include addition of Dibenzo(a,I)pyrene analysis on samples 1-3 as originally requested. Report 1228046 is an addendum to report 1222540.

 Sample sample #1, #2 and #3 was received in a plastic container which does not meet the sample requirements for PAH1 as specified by the reference method.

The information contained on this and all other pages transmitted, is intended for the addressee only and is considered confidential. If the reader is not the intended recipient, you are hereby notified that any use, dissemination, distribution or copy of this transmission is strictly prohibited. If you receive this transmission by error, or if this transmission is not satisfactory, please notify us by telephone.

6

## Sample Custody

| Bill To:<br>Report To:<br>Attn:<br>Sampled By:<br>Company: | SHAC Environmental<br>SHAC Environmental<br>P. O. Box 73<br>Medicine Hat, AB, Canada<br>T1A 7E5<br>Ashley Gavey<br>A. Gavey<br>Shac | Project:<br>ID:<br>Name:<br>Location:<br>LSD:<br>P.O.:<br>Acct code: | Shac Feed Additive Product | Lot ID:<br>Control Number:<br>Date Received:<br>Date Reported:<br>Report Number: | <b>684191</b><br>A 011986<br>May 26, 2009<br>Jun 16, 2009<br>1228046 |  |
|--|---|--|----------------------------|--|--|--|
|--|---|--|----------------------------|--|--|--|

## Sample Disposal Date: June 28, 2009

All samples will be stored until this date unless other instructions are received. Please indicate other requirements below and return this form to the address or fax number on the bottom of this page.

| Extend Sample Storage Until   | (MN  | 1/DD/YY) |
|---|--|----------|
| The following charges apply to extended sample storage<br>Storage for 1 to 5 samples per month<br>Storage for 6 to 20 samples per month<br>Storage for 21 to 50 samples per month<br>Storage for 51 to 200 samples per month<br>Storage for more than 200 samples per month | :<br>\$ 10.00<br>\$ 15.00<br>\$ 30.00<br>\$ 60.00<br>\$ 110.00 |          |
| Return Sample, collect, to the address below via:   |  |          |
| <br>Greyhound<br>Loomis<br>Purolator<br>Other (specify)   | Name<br>Company  |          |
|   | Phone<br>Fax   |          |
|   | Signature  |          |

6

ſ

## **Analytical Report**

.

| Bill To:<br>Report To:<br>Attn:<br>Sampled By:<br>Company:  | SHAC Environmental<br>SHAC Environmental<br>P. O. Box 73<br>Medicine Hat, AB, Canada<br>T1A 7E5<br>Ashley Gavey<br>A. Gavey<br>Shac | Project:<br>ID:<br>Name: Sha<br>Location:<br>LSD:<br>P.O.:<br>Acct code:                | ac Feed Additive Produc     | Lot IC<br>Control Numbe<br>t Date Received<br>Date Reported<br>Report Numbe | e: <b>684191</b><br>r: A 011986<br>d: May 26, 2009<br>d: Jun 16, 2009<br>r: 1228046 |                            |
|---|---|---|-----------------------------|---|---|----------------------------|
| <u>- 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 199</u> | annan ann an Anna ann an An                     | Reference Number<br>Sample Date<br>Sample Time<br>Sample Location<br>Sample Description | 684191-1<br>SHAC Lot 051309 | 684191-2<br>SHAC Lot 030308 \$  | 684191-3<br>HAC Lot 022609  |                            |
|   |   | Matrix  | Liquids                     | Liquids   | Liquids   |                            |
| Analyte   |   | Units   | Results                     | Results   | Results   | Nominal Detection<br>Limit |
| Metals Total  |   |   |                             |   |   |                            |
| Calcium   | Total   | mg/L  | 509                         | 476   | 422   | 0.2                        |
| Iron  | Total   | mg/L  | 116                         | 130   | 115   | 0.05                       |
| Magnesium   | Total   | mg/L  | 81                          | 76  | 89  | 0.1                        |
| Manganese   | Total   | mg/L  | 9.53                        | 8.96  | 8.02  | 0.005                      |
| Potassium   | Total   | mg/L  | 20                          | . 10  | 10  | 0.4                        |
| Silicon   | Total   | mg/L  | 35.4                        | 31.7  | 34.2  | 0.05                       |
| Sodium  | Tot <b>al</b>   | mg/L  | 263                         | 275   | 253   | 0.4                        |
| Sulfur  | Total   | mg/L  | 170                         | 190   | 160   | 0.3                        |
| Aluminum  | Total   | mg/L  | 246                         | 227   | 223   | 0.005                      |
| Antimony  | Total   | mg/L  | 0.007                       | 0.008   | 0.008   | 0.0002                     |
| Arsenic   | Total   | mg/L  | 0.068                       | 0.12  | 0.11  | 0.0002                     |
| Barium  | Total   | mg/L  | 1.8                         | 1.4   | 1.4   | 0.001                      |
| Beryilium   | Total   | mg/L  | 0.028                       | 0.029   | 0.02  | 0.0001                     |
| Bismuth   | Total   | mg/L  | <0.01                       | <0.01   | <0.01   | 0.0005                     |
| Boron   | Total   | mg/L  | 7.59                        | 8.16  | 7.76  | 0.002                      |
| Cadmium   | Total   | mg/L  | 0.002                       | 0.002   | 0.001   | 0.00001                    |
| Chromium  | Total   | mg/L  | 0.473                       | 0.576   | 0.581   | 0.0005                     |
| Cobalt  | Total   | mg/L  | 0.097                       | 0.12  | 0.089   | 0.0001                     |
| Copper  | Total   | mg/L  | 0.1                         | 0.1   | 0.1   | 0.001                      |
| Lead  | Total   | mg/L  | 0.095                       | 0.085   | 0,071   | 0,0001                     |
| Lithium   | Total   | mg/L  | 0.2                         | 0.2   | 0.2   | 0.001                      |
| Molybdenum  | Total   | mg/L  | 0.03                        | 0.03  | <0.02   | 0.001                      |
| Nickel  | Total   | mg/L  | 0.24                        | 0.323   | 0.264   | 0.0005                     |
| Selenium  | Total   | mg/L  | 0.087                       | 0.080   | 0.085   | 0.0002                     |
| Silver  | Total   | mg/L  | 0.0106                      | 0.0107  | 0.00965   | 0.00001                    |
| Strontium   | Total   | mg/L  | 6.57                        | 5.85  | 5.28  | 0.001                      |
| Thallium  | Total   | mg/L  | 0.0025                      | 0.0027  | 0.002   | 0.00005                    |
| Tin   | Total   | mg/L  | 0.64                        | 0.65  | 0.59  | 0.001                      |
| Titanium  | Total   | mg/L  | 10.2                        | 10.1  | 8.31  | 0.0005                     |
| Uranium   | Total   | mg/L  | 0.02                        | 0.02  | 0.02  | 0.0005                     |
| Vanadium  | Total   | mg/L  | 0.638                       | 0.611   | 0.717   | 0.0001                     |
| Zinc  | Total   | mg/L  | 0.75                        | 2.4   | 0.74  | 0.001                      |
| Zirconium   | Total   | mg/L  | 1.9                         | 2.0   | 1.8   | 0.001                      |
| Polycyclic Aro  | matic Hydrocarbons - Water  |   |                             |   |   |                            |
| Naphthalene   |   | ug/L  | <0.1                        | <0.1  | <0.1  | 0.1                        |
| Quinoline   |   | ug/L  | <3.4                        | <3.4  | <3.4  | 3.4                        |
| Acenaphthyler   | าย  | ug/L  | <0.1                        | <0.1  | <0.1  | 0,1                        |

 Bodycote Testing Group
 www.bodycote.com
 www.bodycotetesting.com

 7217 Roper Road NW - Edmonton - AB - T6B 3J4 - Canada - Tel; +1 (780) 438-5522 - Fax: +1 (780) 438-0396
 Terms and Conditions:
 www.bodycotetesting.com/terms&conditions

Cie

6

( )

## **Analytical Report**

| Bill To:<br>Report To: | SHAC Environmental<br>SHAC Environmental<br>P. O. Box 73<br>Medicine Hat, AB, Canada<br>T1A 7E5 | Project:<br>ID:<br>Name:<br>Location:<br>LSD: | Shac Feed Additive Product | Lot ID:<br>Control Number:<br>Date Received:<br>Date Reported:<br>Report Number: | <b>684191</b><br>A 011986<br>May 26, 2009<br>Jun 16, 2009<br>1228046 |
|------------------------|---|---|----------------------------|--|--|
| Attn:                  | Ashley Gavey  | P.O.:   |                            |  |  |
| Sampled By:            | A. Gavey  | Acct code:                                    |                            |  |  |
| Company:               | Shac  |   |                            |  |  |

|                         | R                                  | eference Number<br>Sample Date<br>Sample Time<br>Sample Location | 684191-1        | 684191-2        | 684191-3        |                   |
|-------------------------|------------------------------------|--|-----------------|-----------------|-----------------|-------------------|
|                         | Sa                                 | mple Description   | SHAC Lot 051309 | SHAC Lot 030308 | SHAC Lot 022609 |                   |
|                         |                                    | Matrix   | Liquids         | Liquids         | Liquids         |                   |
| Analyte                 |                                    | Units  | Results         | Results         | Results         | Nominal Detection |
| Polycyclic Aromatic Hyd | rocarbons - Water - Conti          | nued   |                 |                 |                 |                   |
| Acenaphthene            |                                    | ug/L   | <0.1            | <0.1            | <0.1            | 0.1               |
| Fluorene                |                                    | ug/L   | <0.1            | <0.1            | 0.1             | 0.1               |
| Phenanthrene            |                                    | ug/L   | 0.1             | 0.2             | 0.2             | 0.1               |
| Anthracene              |                                    | ug/L   | <0.005          | <0.005          | <0.005          | 0.005             |
| Acridine                |                                    | ug/L   | <0.1            | <0.1            | <0,1            | 0.1               |
| Fluoranthene            |                                    | ug/L   | 0.04            | 0.08            | 0.08            | 0.01              |
| Pyrene                  |                                    | ug/L   | 0.07            | 0.14            | 0.13            | 0.01              |
| Benzo(a)anthracene      |                                    | ug/L   | <0.01           | 0.01            | 0,01            | 0.01              |
| Chrysene                |                                    | ug/L   | <0.1            | <0.1            | <0.1            | 0.1               |
| Benzo(b+j)fluoranthene  |                                    | ug/L   | <0.1            | <0.1            | <0.1            | 0.1               |
| Benzo(k)fluoranthene    |                                    | ug/L   | <0.1            | <0.1            | <0.1            | 0.1               |
| Benzo(a)pyrene          |                                    | ug/L   | <0.008          | <0.008          | <0.008          | 0,008             |
| Indeno(1,2,3-c,d)pyrene |                                    | ug/L   | <0.05           | <0.05           | <0.05           | 0.05              |
| Dibenzo(a,h)anthracene  |                                    | ug/L   | <0.05           | <0.05           | <0.05           | 0.05              |
| Benzo(g,h,i)perylene    |                                    | ug/L   | <0.05           | <0.05           | < 0.05          | 0.05              |
| CB(a)P                  | Carcinogenic Potency<br>Equivalent | ug/L   | <0.01           | <0.01           | <0.01           | .01               |
| PAH - Water - Surrogate | Recovery                           |  |                 |                 |                 |                   |
| Nitrobenzene-d5         | PAH - Surrogate                    | %  | 100             | 110             | 130             | 23-130            |
| 2-Fluorobiphenyl        | PAH - Surrogate                    | %  | 30              | 30              | 30              | 30-130            |
| p-Terphenyl-d14         | PAH - Surrogate                    | %  | 20              | 40              | 20              | 18-137            |

Approved by:

Paron Crichto

Darren Crichton, BSc, PChem Operations Chemist
### **Methodology and Notes**

| Bill To   | : SHAC Environmental     | Project:   |                            | Lot ID:         | 684191       |
|-----------|--------------------------|------------|----------------------------|-----------------|--------------|
| Report To | : SHAC Environmental     | ID:        |                            | Control Number: | A 011986     |
|           | P. O. Box 73             | Name:      | Shac Feed Additive Product | Date Received:  | May 26, 2009 |
|           | Medicine Hat, AB, Canada | Location:  |                            | Date Reported:  | Jun 16, 2009 |
|           | T1A 7E5                  | LSD:       |                            | Report Number:  | 1228046      |
| Attr      | n: Ashley Gavey          | P.O.:      |                            |                 |              |
| Sampled B | r: A. Gavey              | Acct code: |                            |                 |              |
| Compan    | /: Shac                  |            |                            |                 |              |

### **Method of Analysis**

| Method Name                    | Reference | Method   | Date Analysis<br>Started | Location     |
|--------------------------------|-----------|--|--------------------------|--------------|
| Metals ICP-MS (Total) in water | US EPA    | * Determination of Trace Elements in<br>Waters and Wastes by ICP-MS, 200.8                               | 27-May-09                | BTG Edmonton |
| Metals Trace (Total) in water  | APHA      | <ul> <li>Inductively Coupled Plasma (ICP)<br/>Method, 3120 B</li> </ul>                                  | 27-May-09                | BTG Edmonton |
| PAH - Water                    | US EPA    | <ul> <li>Semivolatile Organic Compounds by<br/>Gas Chromatography/Mass<br/>Spectrometry, 8270</li> </ul> | 28-May-09                | BTG Calgary  |
|                                |           | * Bodycote method(s) based on reference method   |                          |              |

### References

| APHA   | Standard Methods for the Examination of Water and Wastewater |
|--------|--|
| US EPA | US Environmental Protection Agency Test Methods              |

### Comments:

Ashley was contacted and informed of the deviation due to her container that was sent in, she would like to go ahead with the analysis.

- Dibenzo(a,i)pyrene analysis was performed by a subcontract laboratory. See attached 3 page report.
- Report was issued to include addition of Dibenzo(a,I)pyrene analysis on samples 1-3 as originally requested. Report 1228046 is an addendum to report 1222540.
- Sample sample #1, #2 and #3 was received in a plastic container which does not meet the sample requirements for PAH1 as specified by the reference method.

Please direct any inquiries regarding this report to our Client Services group.

Results relate only to samples as submitted.

The test report shall not be reproduced except in full, without the written approval of the laboratory.



| Certificate of Analysis |  |  |  |
|-------------------------|--|--|--|
| 09-299415               |  |  |  |
|                         |  |  |  |
| 2009-06-08              |  |  |  |
| 2009-06-10              |  |  |  |
| 1                       |  |  |  |
| lysis                   |  |  |  |
| Analysis                |  |  |  |
|                         |  |  |  |

### Client

### **BODYCOTE (CALGARY)**

#9, 2712-37 AVENUE N.E. CALGARY, Alberta, Canada T1Y5L3

| ار<br>ا | P.O. Number | Your project ID. | Project Manager   |
|---------|-------------|------------------|-------------------|
| ſ       | 106305      | 684191           | Mme Ginger Pecson |

### Comments

This version replaces and cancels all earlier version.

NA : Information Not Available

AVIS DE CONFIDENTIALITÉ : Ce document est à l'usage exclusif du requérant ci-dessus et est confidentiel. Si vous n'êtes pas le destinataire, soyez avisé que tout usage, reproduction, ou distribution de ce document est strictement interdit. Si vous avez reçu ce document par erreur, veuillez nous en informer immédiatement. / CONFIDENTIALITY NOTICE : This document is intended for the addressee only and is considered confidential. If you are not the addressee, you are hereby notified that any use, reproduction or distribution of this document is strictly prohibited. If you have received this document by error, please notify us immediately.

> Certificate of Analysis No. 298555 - Revision 1 - Page 1 of 2 Terms and conditions: <u>http://www.bodycotetesting.com/terms&conditions</u>

> > ·····

This certificate must not be reproduced, except in its entirety, without written consent from the laboratory. The official version of this certificate is protected and cannot be modified. The above-mentioned samples will be retained for a period of 30 days following the issue of this certificate with the exception of microbiology samples or as instructed by the client. Results pertain only to the samples submitted for analysis.



#### 09-299415 **Request Number:**

#### Client: **BODYCOTE (CALGARY)**

| P.0                               | . Number                            |                       | Your Project ID.   |                                    |                                       | Project Manager          |       |  |
|-----------------------------------|-------------------------------------|-----------------------|--|------------------------------------|---------------------------------------|--------------------------|-------|--|
| 1                                 | 106305                              |                       | 684191   |                                    |                                       | Mme Ginger Pecson        |       |  |
| L                                 |                                     |                       |  | Sample(s)                          |                                       |                          |       |  |
|                                   |                                     |                       | Lab. No.   | 1337840                            | 1337841                               | 1337842                  |       |  |
|                                   |                                     |                       | Your<br>Reference  | #1 Shac Lot<br>051309              | #2 Shac Lot<br>030308                 | #3 Shac Lot<br>022609    |       |  |
|                                   |                                     |                       | Matrix   | Liquid                             | Liquíd                                | Liquid                   |       |  |
|                                   |                                     |                       | Sampled by   | CLIENT                             | CLIENT                                | CLIENT                   |       |  |
|                                   |                                     |                       | Site sampled   | NA                                 | NA                                    | NA                       |       |  |
|                                   |                                     |                       | Date sampled   | NA                                 | NA                                    | NA                       |       |  |
|                                   |                                     |                       | Date received  | 2009-06-08                         | 2009-06-08                            | 2009-06-08               |       |  |
| Paramete<br>Method<br>Reference - | er(s)                               |                       |  |                                    |                                       |                          |       |  |
| Polynucle                         | ear aromatic hydrocarbo             | ns (PAH's)            | Preparation  | 2009-06-08                         | 2009-06-08                            | 2009-06-08               |       |  |
| QC058-97 / dic                    | hloromethana extraction, GC-MS anat | ysis                  | Analysis   | 2009-06-09                         | 2009-06-09                            | 2009-06-09               |       |  |
| EPA3510, 8270                     | 07 MA400 HAP 1.1                    |                       | Sequential No.   | 286824                             | 286824                                | 286824                   |       |  |
| Dibenzo (a,                       | i) pyrene                           |                       | μg/L   | <1.0                               | <1.0                                  | <1.0                     |       |  |
| Acenaphter                        | ne-d10                              |                       | %  | 49%                                | 59%                                   | 46%                      |       |  |
| Fluoranther                       | ne-d10                              |                       | %  | 45%                                | 49%                                   | 44%                      |       |  |
| Chrysene-c                        | i12                                 |                       | %  | 36%                                | 33%                                   | 34%                      |       |  |
| Comments                          |                                     |                       |  |                                    | · · · · · · · · · · · · · · · · · · · |                          |       |  |
| 1337840                           | #1 Shac Lot 051309                  | Detectio<br>recupera  | n limit was i⊓crea<br>tion percentage c  | sed because of the<br>f surrogate. | complexity of the m                   | atrix and because of the | e low |  |
| 1337841                           | #2 Shac Lot 030308                  | Detection<br>recupera | Detection limit was increased because of the complexity of the matrix and because of the low recuperation percentage of surrogate. |                                    |                                       |                          |       |  |
| 1337842                           | #3 Shac Lot 022609                  | Detection             | n limit was increa   | sed because of the                 | complexity of the m                   | atrix and because of the | e low |  |

Note: Results pertain only to the samples submitted for analysis.

CHIMIS ٢ ain Ferrar Alain Perron 2003-052 uése' Alain-Perron, chemist Starmer 15.

Certificate of Analysis No. 298555 - Revision 1 - Page 2 of 2 Terms and conditions: http://www.bodycotetesting.com/terms&conditions

This certificate must not be reproduced, except in its entirety, without written consent from the laboratory. The official version of this certificate is protected and cannot be modified. The above-mentioned samples will be retained for a period of 30 days following the issue of this certificate with the exception of microbiology samples or as instructed by the client. Results pertain only to the samples submitted for analysis.



www.bodycotetesting.com

# Certificate of Analysis

### Request Number: 09-299415

# Client: BODYCOTE (CALGARY)P.O. NumberYour Project ID.Project Manager106305684191Mme Ginger Pecson

| Qu  | ality Con | trol Result | s (CQ) |        |                |  |
|---|-----------|-------------|--------|--------|----------------|--|
| Certified Control                         |           |             |        |        |                |  |
| Parameters<br>(Sequential ID No.)         | Units     | RDL         | Blank  | Result | Expected Range |  |
| Polynuclear aromatic hydrocarbons (PAH's) |           |             |        |        |                |  |
| Sequential ID No.: 286824                 |           |             |        |        |                |  |
| Dibenzo (a,l) pyrene                      | μg/L      | < 0.04      | <0.04  | 1.2    | 1.2 - 2.8      |  |

Comments



#### **RDL** : Reported Detection Limit

Appendix 1 of Certificate no.298555 - Page 1 of 1

#### 

This certificate must not be reproduced, accept in its entirely, without written consent from the laboratory. The official version of this certificate is protected and cannot be modified. The above-mentioned samplas will be retained for a pariod of 30 days following the issue of this certificate with the exception of microbiology samples or as instructed by the client. Results pertain only to the samples submitted for enalysis. A & L WESTERN AGRICUL' JAAL LABORATORIES

(i

1311 WOODLAND AVE #1 • MODESTO, CALIFORNIA 95351 • (209) 529-4080 • FAX (209) 529-4736

CLIENT: 9999-D

REPORT NUMBER: 11-024-032

SUBMITTED BY: A GAVEY

SHAC011131 **CUSTOMER:** 

> SHAC ENVIRONMENTAL PRODUCTS INC PO BOX 73/MEDICINE HAT ALBERTA, CAN T1A7E5, SEND TO:

FERTILIZER AND LIME ANALYSIS REPORT

DATE OF REPORT: 01/28/11

-PAGE:

|   |            | <br>         | <br>            |
|---|------------|--------------|-----------------|
|   |            | <br>         |                 |
| HUMIC<br>ACID*<br>%                                     | 1.30       | <br>         | <br>            |
| HUMIC<br>ACID%  | 6.44       | <br>         |                 |
| %<br>SOLIDS   | 4.53       | <br><u> </u> |                 |
| Calcium<br>Carbonate<br>Equiv. %<br>CaCO <sub>3</sub>   |            | <br>         |                 |
| Magnesium<br>% Mg                                       |            |              |                 |
| Calcium<br>% Ca   |            |              |                 |
| Sulfate<br>Sulfur<br>%                                  |            |              | <br>            |
| Non-Ortho<br>Phosphate<br>% of Total<br>P2O5            |            |              | <br>            |
| Available<br>Phosphate<br>P <sub>2</sub> O <sub>5</sub> |            |              | <br>            |
| Zino<br>% Zn  |            | <br>         |                 |
| Sulfur<br>% S   |            | <br>         |                 |
| Potash<br>% K <sub>2</sub> O                            |            | <br>         | <br><del></del> |
| Total<br>Phosphate<br>% P <sub>2</sub> O <sub>5</sub>   |            | <br>         | <br>            |
| Nitrogen<br>% N   |            | <u></u>      | <br>            |
| Lab<br>Number   | 26008      |              |                 |
| Sample<br>Identification                                | SHAC011131 |              |                 |

REMARKS: \* Humic acid analyzed by CDFA method.

A & L WESTERN LABORATORIES, INC. Our reports and letters are for the exclusive and confidential use of our clients, and may not be reproduced in whole or in part, nor may any reference be made to the work, the result or the company in any advertising, news release, or other public announcements without obtaining our prior written authorization. Copyright 1977



This report applies only to the sample(s) tested. Samples are

Robert Butterfield



#### Attention: ASHLEY GAVEY

SHAC ENVIRONMENTAL PRODUCTS INC. PO BOX 73 MEDICINE HAT, AB CANADA T1A 7E5

### Report Date: 2008/01/08

 Job/Sample
 Analysis Type
 Well Name/Sample ID

 A760768/
 I25318
 Certificate of Analysis
 SHAC ENVIROMENTAL PRODUCTS

Sample Point N/A

Encryption Key Michelle Parker Michelle Parker 08 Jan 2008 16:19:09 -07:00

Please direct all questions regarding this Certificate of Analysis to your Project Manager.

MICHELLE PARKER, Project Manager, Petroleum Customer Service Email: michelle.parker@maxxamanalytics.com Phone# (780) 468-3500

Maxxam has procedures in place to guard against improper use of the electronic signature and have the required "signatories", as per section 5.10.2 of ISO/IEC 17025:2005(E), signing the reports. SCC and CAEAL have approved this reporting process and electronic report format.

 Report Distribution
 SHAC ENVIRONMENTAL PRODUCTS INC.
 PO BOX 73
 MEDICINE HAT, CANADA

 KEVIN DALLISON, CET
 Manager, Compositional Analysis Laboratory
 kevin.dallison@maxxamanalytics.com

 BEATA KARPINSKA, MSc
 Manager, Industrial Water Laboratory
 beata.karpinska@maxxamanalytics.com

 BRANKO BANJAC, B.Sc.
 manager, Petroleum Properties Laboratory
 branko.banjac@maxxamanalytics.com

Date of Issue

2008/01/08



C

### **CERTIFICATE OF ANALYSIS**

|                                     |               |   |               |                     |                     | Až            | 60768:125318     |              |
|-------------------------------------|---------------|---|---------------|---------------------|---------------------|---------------|------------------|--------------|
| :                                   | MaxxiD        | Client ID                               | u             | Meter Number        |                     | La            | boratory Number  |              |
| SHAC ENVIRONMENTAL PRODUCTS         | INC.          |   |               |                     |                     |               |                  |              |
| Operator Name                       |               |   |               | LSD<br>N/A          |                     | SHAC          |                  |              |
| SHAC ENVIROMENTAL PRODUCTS          |               |   |               | Initials of Sampler |                     | Sampling Con  | npany            |              |
| Weli Nama                           |               |   | N/A           | •                   |                     | PLASTIC       | BOTTLE           |              |
| Field or Ama                        | Pool or Zona  |   | Sample Point  |                     |                     | Containet Ide | niity            | Percent Full |
|                                     |               |   |               |                     |                     |               |                  |              |
| Test Recovery Inte                  | rval 1 Intern | rai 2 ——— Interval 3 ——                 | Elø           | vations (m)         | Sample Gathering Po | lof -         | Solution         | Gas          |
| From:                               |               |   |               |                     | Well Fluid Status   |               | Well Status Mode |              |
| Test Type No. Multiple Recovery 10: |               |   | КВ            | GRD                 |                     |               |                  |              |
| - Production Rates                  | G             | auge Pressurøs kPa 🗕 —                  | Tempe         | 230                 | Well Status Type    |               | Well Type        |              |
|                                     | Source        | As Beceived                             | Source        | As Received         | Gas or Condensale F | miect         | Licence No.      |              |
| Water move Oir mare Gas Tobolitare  |               | // //////////////////////////////////// |               |                     | Gas of Containation |               |                  |              |
|                                     |               | 2007/12/06                              | 2008/01/08    | 2008/01/0           | 3                   | EF , MP2      |                  |              |
| Date Sampled Start Date Sampled End |               | Date Received                           | Date Reported | Date Reissuen       |                     | Attalyst      | MDI              |              |
| PARAMETER DESCRIPTION               |               | Result                                  | unit          |                     |                     |               |                  |              |
| Elemental Analysis                  | <u> </u>      |   |               |                     |                     |               |                  |              |
| Carbon (C)                          |               | 52.56                                   | mass%         |                     |                     |               | 0.01             |              |
| Hydrogen                            |               | 2.2                                     | mass%         |                     |                     |               | 0.01             |              |
| Nítrogen                            |               | 2.28                                    | mass%         |                     |                     |               | 0.01             |              |
| Oxygen                              |               | 21.22                                   | 11123376      |                     |                     |               |                  |              |
| Physical Properties                 |               |   |               |                     |                     |               |                  |              |
| Ash Content                         |               | 0.21                                    | mass%         |                     |                     |               | 0.001            |              |
| Properties                          |               |   |               |                     |                     |               | 4                |              |
| Total Suspended Solids              |               | 21100                                   | mg/L          |                     |                     |               | l                |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |
|                                     |               |   |               |                     |                     |               |                  |              |

\*\* Information not supplied by client -- data derived from LSD information

Results relate only to items tested

Bemarks: Sulphur = 0.93%

Смахдат

### A760768:125318

| te et state state state state state state state st |        | Analyzed     |
|--|--------|--------------|
| Parameter  | Unit   | On dry basis |
|  |        |              |
| Carbon C   | Mass%  | 52.56        |
| Hydrogen H   | Mass%  | 2.20         |
| Nitrogen N   | Mass%  | 2.28         |
| Oxyden O   | Mazs%  | 21.22        |
| Sulphur S  | Mass%  | 0.93         |
| Total  | Mas5%  | 79.19        |
| Ach Contost  | Marris | 0.21         |

|  | Calculated   |              | Analyzed                               |
|--|--|--------------|--|
| Normalized to<br>Egnite content in<br>sample (w1%) | Normalized to fighte<br>content in sample<br>and element mass<br>fractions (wt%) | On wet basis | On wet basis (first<br>report A740497) |
| 2.02   | 1.603  | 1.603        | 1,15                                   |
| 0.08   | 0.067  | 10.839       | 11.48                                  |
| 0.09   | 0.070  | 0.070        | 0.09                                   |
| 0.82   | 0.647  | 86.825       | 85,48                                  |
| 0.03   | 0,028  | 0.028        | not analyzed                           |
| 3.04   | 2.42   | 99,365       |  |
| t stand og som sere som så                         | <i></i>  | add          |  |

| Ash Content | Mass% | 0.21  |            |
|-------------|-------|-------|------------|
| TSS         | Mass% | 3.05  |            |
| Water H2O   | Mass% | 96,95 | calculated |

| _     | 0.63   | _includes ash |
|-------|--------|---------------|
| Total | 99,993 | from lign     |

ncludes ash content and other elements from lignite contamination

|            | Atomic<br>weight | Transferred to<br>wt% | Normalized to<br>water content in<br>sample (wt%) |
|------------|------------------|-----------------------|---|
| Hydrogen H | 2                | 11.11                 | 10.77   |
| Oxygen O   | 16               | 88.89                 | 86.18   |
| Water H2O  | 18               | 100.00                | 96.95   |



(

### PHYSICAL PRODUCT DATA

- Density of Ponder Product = 1.1 kg/L or 1.1 g/ml
- Prior to bottling, the product is screened through 200 mesh screen (refer to particle size analysis).

The following data was reported by Zalco Laboratories Inc. in Bakersfield, CA:

- Ponder has no flash point, is not corrosive, and does not release reactive gases. The product should be stirred well prior to application to re-suspend any settled matter 1997.
- Product contains approximately 5% solids to 95% liquids by volume (there is a small degree of variation in the ratio between product batches).

Appendix 6

.

(

()

2

SHAC Product Material Safety Data Sheets (MSDS)

SECTION 1. PRODUCT IDENTIFICATION

CHEMICAL FAMILY: organic carbon liquid MANUFACTURED BY: SHAC Environmental Products Inc. PO Box 73, Medicine Hat, AB, T1A 7E5

EMERGENCY TELEPHONE NUMBER: 1-888-533-4446 DESCRIPTION: blackish / brown liquid TRADE NAME: **SHAC Revitagro** PRODUCT USE: 1. soil amendment. 2. improve fertilizer retention NOT A CONTROLLED PRODUCT UNDER WHMIS

### SECTION 2. HAZARDOUS INGREDIENTS

INGREDIENTS: Liquefied lignite coal (water, oxidized lignite coal) Lignite Coal CAS # 129521-66-0 LC<sub>50</sub> (Species & route): not applicable TVL (AGGIH): None currently established - used mainly outdoors

### SECTION 3. PHYSICAL DATA

PHYSICAL STATE: liquid BOILING POINT: not available APPEARANCE: blackish/brown ODOR: clean coal SPECIFIC GRAVITY: not applicable VAPOR DENSITY: (air = 1) not available DENSITY: 1.1 g/ml COEFFICIENT OF WATER/OIL DISTRIBUTION: n/a ODOR THRESHOLD: not applicable VAPOR PRESSURE: not applicable EVAPORATION RATE: not applicable

SECTION 4. FIRE AND EXPLOSION HAZARD FLAMMABLE: No FLASH POINT: (Test method); not applicable AUTOIGNITION TEMPERATURE: not applicable FLAMMABLE LIMITS IN AIR, % BY VOLUME: n/a EXTINGUISHING MEDIA: not applicable HAZARDOUS COMBUSTION PRODUCTS: not known LEL: not applicable UEL: not applicable

#### SECTION 5. REACTIVITY DATA STABILITY: stable INCOMPATIBLE PRODUCTS: none known HAZARDOUS POLYMERIZATION: will not occur CONDITIONS OF CHEMICAL UNSTABILITY: not applicable.

CONDITIONS OF REACTIVITY: none

### <u>SECTION 6. HEALTH HAZARDS</u> LD<sub>50</sub> (Mixture): not applicable

 $LC_{50}$  (Mixture): not applicable

EFFECTS OF SINGLE (ACUTE) OVEREXPOSURE: SWALLOWING: No evidence of adverse effects from available information. SKIN ABSORPTION: No evidence of adverse effects from available information. INHALATION: No evidence if adverse effects from available information. SKIN CONTACT: Staining may occur - wash with soap and water. No harmful effects expected. EYE CONTACT: No harmful effects expected from vapour. If splashed in eyes, rinse with water to remove particles. EFFECTS OF REPEATED (CHRONIC) OVEREXPOSURE: No evidence of adverse effects from available information.

effects from available information. SIGNIFICANT LABORATORY DATA WITH POSSIBLE RELEVANCE TO HUMAN HEALTH HAZARD EVALUATION: None currently known.

### SECTION 7. PREVENTATIVE MEASURES PERSONAL PROTECTIVE EQUIPMENT TO BE USED:

RESPIRATORY PROTECTION: Not required PROTECTIVE GLOVES: Wear loose-fitting gloves. EYE PROTECTION: It is recommended that

goggles be worn when spraying.

### SECTION 8. FIRST AID

INHALATION: Not applicable.. INGESTION: Dilute the stomach contents by giving water or milk. EYES: Flush with water to remove particles, which may cause irritation. SKIN: Remove clothing and wash with soap and water.

### SECTION 9. PREPARATION DATA

PREPARED BY: SHAC Environmental Products Inc. January 4, 2011 PHONE NUMBER: 1-403-528-4446 FAX NUMBER: 1-403-529-9334 WEB SITE: www.shac.ca



SECTION 1. PRODUCT IDENTIFICATION CHEMICAL FAMILY: organic carbon liquid MANUFACTURED BY: SHAC Environmental Products Inc. PO Box 73, Medicine Hat, AB, T1A 7E5

EMERGENCY TELEPHONE NUMBER: 1-888-533-4446 DESCRIPTION: blackish / brown liquid TRADE NAME: **SHAC PONDER** PRODUCT USE: 1. reduces odors. 2. promotes biodigestion of organic sludge and reduces suspended solids in water NOT A CONTROLLED PRODUCT UNDER WHMIS

**SECTION 2.** HAZARDOUS INGREDIENTS INGREDIENTS: Liquefied lignite coal (water, oxidized lignite coal) Lignite Coal CAS # 129521-66-0 LC<sub>50</sub> (Species & route): not applicable TVL (AGGIH): None currently established - used mainly outdoors

SECTION 3. PHYSICAL DATA PHYSICAL STATE: liquid BOILING POINT: not available APPEARANCE: blackish/brown ODOR: clean coal SPECIFIC GRAVITY: not applicable VAPOR DENSITY: (air = 1) not available DENSITY: 1.1 g/ml COEFFICIENT OF WATER/OIL DISTRIBUTION: n/a ODOR THRESHOLD: not applicable VAPOR PRESSURE: not applicable EVAPORATION RATE: not applicable

SECTION 4. FIRE AND EXPLOSION HAZARD FLAMMABLE: No FLASH POINT: (Test method); not applicable AUTOIGNITION TEMPERATURE: not applicable FLAMMABLE LIMITS IN AIR, % BY VOLUME: n/a EXTINGUISHING MEDIA: not applicable HAZARDOUS COMBUSTION PRODUCTS: not known LEL: not applicable UEL: not applicable

SECTION 5. REACTIVITY DATA STABILITY: stable INCOMPATIBLE PRODUCTS: none known HAZARDOUS POLYMERIZATION: will not occur CONDITIONS OF CHEMICAL UNSTABILITY: not applicable. CONDITIONS OF REACTIVITY: none

SECTION 6. HEALTH HAZARDS LD<sub>50</sub> (Mixture): not applicable

LC<sub>50</sub> (Mixture): not applicable

EFFECTS OF SINGLE (ACUTE) OVEREXPOSURE: SWALLOWING: No evidence of adverse effects from available information. SKIN ABSORPTION: No evidence of adverse effects from available information. INHALATION: No evidence if adverse effects from available information. SKIN CONTACT: Staining may occur - wash with soap and water. No harmful effects expected. EYE CONTACT: No harmful effects expected from vapour. If splashed in eyes, rinse with water to remove particles. **EFFECTS OF REPEATED (CHRONIC)** OVEREXPOSURE: No evidence of adverse effects from available information. SIGNIFICANT LABORATORY DATA WITH POSSIBLE RELEVANCE TO HUMAN HEALTH HAZARD EVALUATION: None currently known.

SECTION 7. PREVENTATIVE MEASURES PERSONAL PROTECTIVE EQUIPMENT TO BE USED: RESPIRATORY PROTECTION: Not required PROTECTIVE GLOVES: Wear loose-fitting gloves. EYE PROTECTION: It is recommended that goggles be worn when spraying.

#### SECTION 8. FIRST AID

INHALATION: Not applicable.. INGESTION: Dilute the stomach contents by giving water or milk. EYES: Flush with water to remove particles, which may cause irritation. SKIN: Remove clothing and wash with soap and water.

#### SECTION 9. PREPARATION DATA

PREPARED BY: SHAC Environmental Products Inc. January 4, 2011 PHONE NUMBER: 1-403-528-4446 FAX NUMBER: 1-403-529-9334 WEB SITE: <u>www.shac.ca</u>



SECTION 1. PRODUCT IDENTIFICATION CHEMICAL FAMILY: organic carbon liquid MANUFACTURED BY: SHAC Environmental Products Inc. PO Box 73, Medicine Hat, AB, T1A 7E5

EMERGENCY TELEPHONE NUMBER: 1-888-533-4446

DESCRIPTION: blackish / brown liquid TRADE NAME: SHAC Manure Digester PRODUCT USE: 1. reduces odors. 2. promotes biodigestion and waste solids breakdown NOT A CONTROLLED PRODUCT UNDER WHMIS

#### SECTION 2. HAZARDOUS INGREDIENTS

INGREDIENTS: Liquefied lignite coal (water, oxidized lignite coal) Lignite Coal CAS # 129521-66-0 LC<sub>50</sub> (Species & route): not applicable TVL (AGGIH): None currently established - used mainly outdoors

SECTION 3. PHYSICAL DATA PHYSICAL STATE: liquid BOILING POINT: not available APPEARANCE: blackish/brown ODOR: clean coal SPECIFIC GRAVITY: not applicable VAPOR DENSITY: (air = 1) not available DENSITY: 1.1 g/ml COEFFICIENT OF WATER/OIL DISTRIBUTION: n/a ODOR THRESHOLD: not applicable VAPOR PRESSURE: not applicable EVAPORATION RATE: not applicable

SECTION 4. FIRE AND EXPLOSION HAZARD

FLASH POINT: (Test method); not applicable AUTOIGNITION TEMPERATURE: not applicable FLAMMABLE LIMITS IN AIR, % BY VOLUME: n/a EXTINGUISHING MEDIA: not applicable HAZARDOUS COMBUSTION PRODUCTS: not known LEL: not applicable

UEL: not applicable

SECTION 5, REACTIVITY DATA STABILITY: stable INCOMPATIBLE PRODUCTS: none known HAZARDOUS POLYMERIZATION: will not occur CONDITIONS OF CHEMICAL UNSTABILITY: not applicable.

CONDITIONS OF REACTIVITY: none

#### SECTION 6. HEALTH HAZARDS

LD<sub>50</sub> (Mixture): not applicable LC<sub>50</sub> (Mixture): not applicable EFFECTS OF SINGLE (ACUTE) OVEREXPOSURE: SWALLOWING: No evidence of adverse effects from available information. SKIN ABSORPTION: No evidence of adverse effects from available information. INHALATION: No evidence of adverse effects from available information. SKIN CONTACT: Staining may occur - wash with soap and water. No harmful effects expected. EYE CONTACT: No harmful effects expected from vapour. If splashed in eyes, rinse with water to remove particles. EFFECTS OF REPEATED (CHRONIC) OVEREXPOSURE: No evidence of adverse effects from available information. SIGNIFICANT LABORATORY DATA WITH POSSIBLE RELEVANCE TO HUMAN HEALTH

HAZARD EVALUATION: None currently known.

#### SECTION 7. PREVENTATIVE MEASURES PERSONAL PROTECTIVE EQUIPMENT

TO BE USED: RESPIRATORY PROTECTION: Not required. PROTECTIVE GLOVES: Wear loose-fitting gloves. EYE PROTECTION: It is recommended that

goggles be worn when spraying.

### SECTION 8. FIRST AID

INHALATION: Not applicable. INGESTION: Dilute the stomach contents giving water or milk. EYES: Flush with water to remove particles, which may cause irritation SKIN: Remove clothing and wash with soap and water.

#### SECTION 9. PREPARATION DATA

PREPARED BY: SHAC Environmental Products Inc. January 4, 2011 PHONE NUMBER: 1-403-528-4446 FAX NUMBER: 1-403-529-9334 WEB SITE: www.shac.ca



SECTION 1. PRODUCT IDENTIFICATION CHEMICAL FAMILY: organic carbon liquid MANUFACTURED BY: SHAC Environmental Products Inc. PO Box 73, Medicine Hat, AB, T1A 7E5

EMERGENCY TELEPHONE NUMBER: 1-888-533-4446

DESCRIPTION: blackish / brown liquid TRADE NAME: SHAC Liquid Feed Additive for Odour Control in Swine

PRODUCT USE: 1. reduces odors. 2. reduces ammonia gas produced in confined hog operations

NOT A CONTROLLED PRODUCT UNDER WHMIS

SECTION 2. HAZARDOUS INGREDIENTS INGREDIENTS: water, oxidized bituminous coal (CAS # 129521-66-0) LC<sub>50</sub> (Species & route): not applicable TVL (AGGIH): None currently established - used mainly outdoors

SECTION 3. PHYSICAL DATA PHYSICAL STATE: liquid BOILING POINT: not available APPEARANCE: blackish/brown ODOR: clean coal SPECIFIC GRAVITY: not applicable VAPOR DENSITY: (air = 1) not available DENSITY: 1.1 g/ml COEFFICIENT OF WATER/OIL DISTRIBUTION: n/a ODOR THRESHOLD: not applicable VAPOR PRESSURE: not applicable EVAPORATION RATE: not applicable

SECTION 4. FIRE AND EXPLOSION HAZARD

FLAMMABLE: No FLASH POINT: (Test method); not applicable AUTOIGNITION TEMPERATURE: not applicable FLAMMABLE LIMITS IN AIR, % BY VOLUME: n/a EXTINGUISHING MEDIA: not applicable HAZARDOUS COMBUSTION PRODUCTS: not known LEL: not applicable UEL: not applicable

SECTION 5. REACTIVITY DATA

STABILITY: stable INCOMPATIBLE PRODUCTS: none known HAZARDOUS POLYMERIZATION: will not occur CONDITIONS OF CHEMICAL UNSTABILITY: not applicable.

CONDITIONS OF REACTIVITY: none

SECTION 6. HEALTH HAZARDS

LD<sub>50</sub> (Mixture): not applicable LC<sub>50</sub> (Mixture): not applicable EFFECTS OF SINGLE (ACUTE) OVEREXPOSURE: SWALLOWING: No evidence of adverse effects from available information. SKIN ABSORPTION: No evidence of adverse effects from available information. INHALATION: No evidence if adverse effects from available information. SKIN CONTACT: Staining may occur - wash with soap and water. No harmful effects expected. EYE CONTACT: No harmful effects expected from vapour. If splashed in eyes, rinse with water to remove particles. EFFECTS OF REPEATED (CHRONIC) OVEREXPOSURE: No evidence of adverse effects from available information. SIGNIFICANT LABORATORY DATA WITH

POSSIBLE RELEVANCE TO HUMAN HEALTH HAZARD EVALUATION: None currently known.

### SECTION 7. PREVENTATIVE MEASURES

PERSONAL PROTECTIVE EQUIPMENT TO BE USED: RESPIRATORY PROTECTION: Not required PROTECTIVE GLOVES: Wear loose-fitting gloves. EYE PROTECTION: It is recommended that goggles be worn when spraying.

#### SECTION 8. FIRST AID

INHALATION: Not applicable.. INGESTION: Dilute the stomach contents by giving water or milk. EYES: Flush with water to remove particles, which may cause irritation. SKIN: Remove clothing and wash with soap and water.

### SECTION 9, PREPARATION DATA

PREPARED BY: SHAC Environmental Products Inc. January 4, 2011 PHONE NUMBER: 1-403-528-4446 FAX NUMBER: 1-403-529-9334 WEB SITE: <u>www.shac.ca</u>



# Appendix 7

 $\langle \widehat{\phantom{a}} \rangle$ 

NOSB Review of Humic Acid Derivatives (1996)

And

Technical Evaluation Report – Humic Acids – Crops (2006)

# **NOSB NATIONAL LIST FILE CHECKLIST**

# **CROPS**

MATERIAL NAME: #7 Humic acid derivatives



**NOSB** Database Form



References

**MSDS (or equivalent)** 

TAP Reviews from: William Zimmer, James A. Johnson, Paul Sachs

Crops -September 1996

\_\_\_\_\_

# NOSB/NATIONAL LIST COMMENT FORM CROPS

# Material Name: #7 Humic acid derivatives

Please use this page to write down comments, questions, and your anticipated vote(s).

**COMMENTS/QUESTIONS:** 

,

(

1. In my opinion, this material is: \_\_\_\_\_ Synthetic \_\_\_\_\_ Non-synthetic.

2. This material should be placed on the proposed National List as: \_\_\_\_\_Prohibited Natural \_\_\_\_\_Allowed Synthetic.

# TAP REVIEWER COMMENT FORM for USDA/NOSB

(

.....

Use this page or an equivalent to write down comments and summarize your evaluation regarding the data presented in the file of this potential National List material. Complete both sides of page. Attach additional sheets if you wish.

| T           | his file  | is due bac  | k to us by:  | Aug.  | 5,1996   |      |
|-------------|---|---|--|---|--|------|
| L<br>N<br>R | lame of<br>leviewer   | Material:<br>Name:  | Humic<br>BART HAL  | Acid  | Derivatives  |      |
| ا:<br>a     | s this su<br>oppropriat<br>If syn<br>form<br>Deriva<br>Strong | ubstance S<br>e) <u>GEN</u><br>athetic, how is<br>is blank)<br>twes, esp<br>alkali. | ynthetic or no<br>ERALLY NON<br>: the material made<br>ecially Itquic<br>Natural TRON        | n-synthei<br>SYNTHE<br>? (please an<br>? produce<br>A Aux       | tic? Explain (if<br><u>TIC</u><br>swer here if our database<br>its have been fluxed<br>only permitted. | lup  |
| T           | rhis mat<br><br>or, _<br>HAD                                  | Synthetic A<br>Non-synthetic A  | ld be added to<br>llowed<br>netic (This material<br>in alkali offun                          | the Na<br>Pr<br>does not be<br>than Ma                          | tional List as:<br>ohibited Natural<br>long on National List)<br>fural frome                           |      |
|             | Are ther<br>placed o<br>fermuth<br>and<br>Please co           | e any use<br>in this man<br>al only w<br>they, toul<br>mment on th                  | restrictions or<br>terial on the M<br>hen liquids of<br>y if all office<br>re accuracy of th | limitational<br>National<br>re diss<br>er ingr<br>e information | ons that should be<br>List?<br>olved with Maturel<br>edients comply.<br>tion in the file:              | Tro  |
|             | Any add<br>S<br>Oure (  | sitional con<br>er affact<br>lefinitely   | nments? (attac<br>ment. Als<br>bizarre Sory  | hments<br>o, soul<br>n, coula                                   | Welcomed)<br>H,A.D. products<br>innig all sorts of stu<br>visil?                                       | fl - |
|             | Do you h<br>Signatu   | ave a comm<br>re <u>Barto</u>   | m. Hell_   | Dat   | e <u>96.08.06</u>  |      |

Please address the 7 criteria in the Organic Foods Production Act: (comment in those areas you feel are applicable)

 the potential of such substances for detrimental chemical interactions with other materials used in organic farming systems;

NONE

(

(2) the toxicity and mode of action of the substance and of its breakdown products or any contaminants, and their persistence and areas of concentration in the environment;



(3) the probability of environmental contamination during manufacture, use, misuse or disposal of such substance;



(4) the effect of the substance on human health;

Unknown.

(5) the effects of the substance on biological and chemical interactions in the agroecosystem, including the physiological effects of the substance on soil organisms (including the salt index and solubility of the soil), crops and livestock;

Too complex and unstudied to defermine at this point.

(6) the alternatives to using the substance in terms of practices or other available materials; and

See a Hachment

(7) its compatibility with a system of sustainable agriculture. Annal Mat always effective

### HUMATES and HUMIC ACID DERIVATIVES

### Prepared by Bart Hall, October, 1995

Humates and humic acid derivatives are a diverse family of products, generally obtained (directly or indirectly) from various forms of oxidized coal.

Coal deposits are of three types. Anthracite coal is very dense and hard with quite low sulphur content. Bituminous coal is a softer coal, usually with rather high sulphur levels. Lignite coal is a very soft, coarse coal with highly variable sulphur content and often marginal fuel value. Softer coals, particularly lignite, are (as a result of their more open texture) subject to oxidation, especially if found in a near-surface deposit.

While oxidation decreases the fuel value of lignite coals, it increases the percent of alkalineextractable humic matter. In the western part of the Dakotas and the eastern part of Montana there are millions of tons of oxidized, near-surface lignite, surrounded by millions of hectares of alkaline soils capable of releasing assorted humic substances from these lignites.

Oxidized-coal-derived (OCD) humus and humic substances are essentially the same as humus extracts from soil (1), but there has been a reluctance to accept OCD humus as a worthwhile soil additive. In part, this stems from the belief (unjustified, in my opinion) that only humus derived from recently-decayed organic matter is beneficial.

Production and recycling of organic matter in the soil cannot be replaced by OCD humus — the sugars, gums, hemi-celluloses and similar materials from fresh organic matter play a vital role in both soil microbiology and structure, but they are not humus. Sod-based rotations, green manures and cover crops, preservation of crop residues and additions of manure or compost remain fundamental elements of any healthy soil management system, but especially *organic* management systems. However, only a small portion of the organic matter added to the soil will ever be converted to humus (most will return to the atmosphere as a result of microbial activity and cultivation), and only then on a time scale greatly exceeding current management frameworks.

The reluctance to accept OCD humus as a worthwhile soil amendment may also arise not only from the plethora of definitions for "humus," but also from the chemical complexity of humus, making it impossible to demonstrate that OCD humus and soil humus are identical. As a result, most of the research with OCD humus has involved indirect field trials and similar "bio-assay" methods. Such studies, by nature, involve many more variables and unknowns than do simple head-to-head chemical comparisons, and are much more subject to uncertainties and variations in their results. Additionally, there is an extensive body of anecdotal and experiential information surrounding OCD humus, arising from studies of varying sophistication and independence by farmers and vendors of the product.

HUMATES and HUMIC ACID DERIVATIVES (B. HALL)

page 1

HUMATES.DOC

Even determining the most fundamental characteristics of OCD humus can be challenging, especially in the case of farmer or vendor trials, because the degree of oxidation is something of a continuum and the exact origin and/or composition of the tested material has not necessarily been recorded. Many forms of oxidized coal are available, and are generally classified by stages of oxidation eventually ending in the complete humification of the starting material (1). In the case of lignite coal, the apparent end-product of natural oxidation is a soft, loose-textured, almost earthy OCD humus known as **leonardite** (2). Leonardite usually occurs at lignite outcrops, or at the top of very shallow beds of lignite, grading into the parent lignite seam.

Partially-oxidized lignite is called slack lignite and contains far less OCD humus than leonardite, but nevertheless more than lignite. The following table (I) summarizes approximate chemical properties of potential sources of OCD humus:

|                                 | LIGNITE | SLACK LIG. LEC | DNARDITE |
|---------------------------------|---------|----------------|----------|
| Oxygen in source material       | 20%     | 25%            | 30%      |
| Extracted humic acids           | 5%      | 30%            | 85%      |
| Oxygen in extracted humic acids | 25%     | 30%            | 30%      |

### Summaries of some leonardite studies

For Kennebec potatoes treated with 200 lb./ac. 10-20-10 fertilizer, plots treated with pulverized slack lignite (1 ton/ac.) showed a dry-matter-corrected yield increase of 9% over the control. Plots treated with leonardite (1 ton/ac.) showed a 28% dry-matter-corrected yield increase compared to the control (1).

Soybeans are a difficult crop to grow in the northern Great Plains, due to alkaline soils and short growing seasons, so leonardite was studied in that region to determine if it had significant enough impact on crop yield to make the difference between crop failure and success. The limited study suggests that it does (2). Unfertilized soybeans, both rhizobia-inoculated and uninoculated, were treated with 1 ton/ac. or leonardite *in the row*. Inoculated, untreated soybeans yielded 65% more than the uninoculated, untreated control. Both uninoculated and inoculated leonardite-treated soybeans yielded roughly three times the control, while the treated, inoculated soybeans managed to double the yield of their untreated (but inoculated) counterparts.

HUMATES.DOC

Other leonardite research in Illinois (3) on corn and soybeans shows no benefit from the material. In general there have been far more positive results on Western soils, typically high pH soils with low available iron, low organic matter and low extractable humic acids.

With most crops, quality was improved, and yield increases noted for some of the crops normally responsive to additions of organic matter. Humate increased root growth and root formation, deepened the color of the leaves, flowers, and fruits, and at high rates increased branching and reduced terminal extension (4).

Humic acids retain nutrient ions against leaching, yet hold them in a way that they are nevertheless readily available to plants. This results from humic acids' high cation exchange capacity (5).

Humic acids mobilize the phosphate ion. In the presence of humates, plants use phosphate fertilizers much more fully than otherwise. This is probably because the humic molecule and phosphate anions compete almost equally for the anion exchange sites on clays, and for the multivalent cations, such as aluminum (6).

Humic acids appear to chelate certain metallic cations, and may be important for plants growing in alkaline soil by enabling increased uptake of micronutrients (7). Humate fertilizers added to a sandy loam soil had no beneficial effect on plant growth; rather, they decreased soil permeability (5). Humate has also been shown to improve the chipping quality of white potatoes so greatly that chippers pay a premium for such potatoes (8).

Eastern studies (9) suggest that corn yields were best at 5 ppm humic acid and that the addition of humates to a hydroponic solution stimulated both root and shoot development, resulting in an increase of 87%. These studies also show that as soil humic acid levels increased, so did phosphorus in the plant, indicating that humates probably play an important role in plant phosphate utilization. These data also seem to show that if soil humate levels are already high, further additions may not benefit the crop.

Humic acids may protect plants against the harmful effects of aluminum, by preventing phosphorus deficiency in the presence of high aluminum and by suppressing toxie effects of aluminum (10). It has also been suggested that soil aluminum and iron may inactivate humates. This may be one important reason why humate products often generate disappointing results when used on acid soils in humid areas. Such acid soils often contain large amounts of soluble aluminum and iron, and will be problematic (with or without humates) unless limed to optimal levels. Western soils, in contrast, often have high levels of free lime, resulting in very low iron and aluminum levels as calcium tends to precipitate those ions, along with manganese; in such situations, humates seem inherently more effective.

HUMATES and HUMIC ACID DERIVATIVES (B. HALL)

page 3

HUMATES.DOC

In sum, then, OCD humus has shown very promising results as a natural soil amendment in areas of alkaline, low-organic-matter soils. Such soils are common across a wide range of agricultural production zones in central and western North America. Leonardite and similar products appear to be entirely consistent with organic production practices, given that they are natural products with proven benefit in certain situations.

### Citations:

G

- Freeman, Philip C. 1970. Technology and Use of Lignite. Proceedings: Bureau of Mines - Univ. North Dakota Symposium, Grand Forks, North Dakota. BOM Circular 8471.
- Fowkes, W.W. and C.M. Frost. 1960. Leonardite: A lignite byproduct. Bureau of Mines Rept. of Inv. 5611. 12 p.
- 3) Anonymous. 1976. NCR-103 Committee report.
- 4) Martin, J.A, et al. 1962. Influence of humic and fulvic acids on the growth, yield, and quality of certain horticultural crops. Clemson Univ. Dept. Hort. Research Series No. 30, May.
- 5) Acock, B. 1963. Effects of humic acids on the growth of tomato plants and the physical properties of sand and sandy soils. M.Sc. Thesis. Clemson Univ., South Carolina.
- Bosse, A. 1957. Information supplied to Doggett-Pfeil Co., Springfield, New Jersey Cited in: Stearman, R.J., et al. 1989. Characterization of Humic acid from no-tilled and tilled soils using Carbon-13 nuclear magnetic resonance. Jour. Soil Sci. Soc. Am. Vol. 53, p. 744-49.
- 7) Heintze, S.C., and P.J.G. Mann. 1949. Jour. Agric. Sci. Vol 39, p. 80-95.
- 8) Moore, M.D. 1964. Unpublished data, Clemson Univ. Dept. Hort. Cited in: -Stearman, R.J., et al. 1989. Characterization of Humic acid from no-tilled and tilled soils using Carbon-13 nuclear magnetic resonance. Jour. Soil Sci. Soc. Am. Vol. 53, p. 744-49.
- 9) Lee, Y.S. and R.J. Bartlett. 1976. Stimulation of plant growth by humic substances. Jour. Soil Sci. Soc. Am. Vol. 40, p. 876-79.
- 10) Tan, K.H. 1985. The effects of humic acid on aluminum toxicity in com plants. Agronomy Abstracts.

HUMATES and HUMIC ACID DERIVATIVES (B. HALL)

page 4

# TAP REVIEWER COMMENT FORM for USDA/NOSB

Use this page or an equivalent to write down comments and summarize your evaluation regarding the data presented in the file of this potential National List material. Complete both sides of page. Attach additional sheets if you wish.

|  | No   |  |
|--|--|--|
| Humic Aria   | Derivariurs  |  |
| illiam A. Zimme  | - D.V. MRECEIVED JUL   | - 3 D 1995   |
| etic or non-synthe<br>effic<br>naterial made? (please an<br>by mining natural a<br>ay be extracted using<br>humic acid, futbic a | tic? Explain (if<br>swerhere if our database<br>epocsits of humates or<br>alkaliaes to produce wa  | humic<br>ter   |
| e added to the Na  | tional List as:  |  |
| ed Pr<br>This material does not be   | ohibited Natural<br>long on National List)   |  |
|  | etic or non-synthes<br>etic or non-synthes<br>etic or non-synthes<br>effc<br>outerial made? (please an<br>by mining inchard a<br>ay be extracted using<br>humic acid, futbric a<br>e added to the Na<br>ed Pr<br>This material does not be | etic or non-synthetic? Explain (if<br><u>Hidh A. Zimmer D.V. MRECEIVED JUR</u><br>etic or non-synthetic? Explain (if<br><u>Hic</u><br>uaterial made? (please answer here if our database<br>by mining inclosed deposits of humates or<br>ay be extracted using alkalines to produce use<br>humic acid, fullic acid,<br>e added to the National List as:<br><u>ed</u> Prohibited Natural<br>This material does not belong on National List) |

Please comment on the accuracy of the information in the file: minimal information;

### Any additional comments? (attachments welcomed)

Composition - humic acid, fulvic acid, natural salts of these acids such as calcium humates Siroperties - Dark brown, black grownlar or pewder solid mined from national humate deposits. OR How Made Dark brown, black light consisting of acids extracted by treatment with weak alkalles. OR Golden, clear liquid of water extracted humates.

Uses - source of humic and, fullise acid, Carbon, numerous trace minerals bound as colloids or mark electrostatic bonds. Nutrient for plants, animals. Do you have a commercial interest in this material? Yes; No

illen M. finner 1) M. Date 7-3-96 Signature

Please address the 7 criteria in the Organic Foods Production Act: (comment in those areas you feel are applicable)

(1) the potential of such substances for detrimental chemical interactions with other materials used in organic farming systems;

minimal - naturally occurring, ubiquitus compounds

(2) the toxicity and mode of action of the substance and of its breakdown products or any contaminants, and their persistence and areas of concentration in the

environment; minimal under practical lese conditions

(3) the probability of environmental contamination during manufacture, use, misuse or disposal of such substance;

none-

 $( \square$ 

( ·

- (4) the effect of the substance on human health; fositive offects when used as a nutrient source.
- (S) the effects of the substance on biological and chemical interactions in the agroecosystem, including the physiological effects of the substance on soil organisms (including the salt index and solubility of the soil), crops and livestock;
- (6) the alternatives to using the substance in terms of practices or other available materials; and unknown

(7) its compatibility with a system of sustainable agriculture.

compatible

# TAP REVIEWER COMMENT FORM for USDA/NOSB

Use this page or an equivalent to write down comments and summarize your evaluation regarding the data presented in the file of this potential National List material. Complete both sides of page. Attach additional sheets if you wish.

This file is due back to us by:

August 5, 1996

| Name of Material: | Humic Acid Derivatives | RECEIVED AUG 0 5 1996 |
|-------------------|------------------------|-----------------------|
| Reviewer Name:    | Paul Sachs             |                       |

### Is this substance synthetic or non-synthetic? Explain (if appropriate) Synthetic; it comes from natural materials but is extracted via chemical reactions.

If synthetic, how is the material made? (please answer here if our database form is blank) Humic acid derivatives are usually extracted from lignite or leonardite (soft coals) by reactions with potassium hydroxide or ammonia (use of ammonia is very rare). The extract usually contains potassium from the reaction.

# This material should be added to the National list as:

Prohibited Natural, or Synthetic Allowed 11

Non-synthetic (This material does not belong on National List

### Are there any use restrictions or limitations that should be placed on this material on the National List?

Per Label instructions.

 $\square$ 

# Please comment on the accuracy of the information in the file:

None provided

### Any additional comments? (attachments welcomed)

Humic soid derivatives seem to be very helpful in poor soil that are deficient in organic matter or humus. Research indicates, however, that very little benefit is provided in soils that are already rich in organic matter and humic substances.

| To vou have a commercial interest in this material Yes | No 🛛     |        |  |
|--|----------|--------|--|
| Signature Jave D. Jack                                 | <br>Date | 8/5/96 |  |

#### P. 3

### Please address the 7 criteria in the Organic Foods Production Act: (comment in those areas you feel are applicable)

### 1. the potential of such substances for detrimental chemical interactions with other materials used in organic farming systems;

None that I know of.

# 2. the toxicity and mode of action of the substance and of its breakdown products of any contaminants, and their persistence and areas of concentration in the environment;

These products are not known for any type of toxicity to plants, soil organisms, or higher animals. Breakdown by soil organisms is near complete.

# 3. the prohability of environmental contamination during manufacture, use, misuse or disposal of such substance:

There is very little waste produced in the manufacturing process. The main by-product is inorganic material similar in analysis to the inorganic component of soil.

### 4. the effect of the substance on human health;

None that I know of.

# 5. the effects of the substance on biological and chemical interactions in the agroecosystem, including the physiological effects of the substance on soil organisms (including the salt index and solubility of the soil), crops and livestock;

Beneficial reactions have been recorded in poor soils. Neutral reaction are noted in good soils. I have never seen negative research data.

### 6. the alternatives to using the substance in terms of practices or other available materials; and

Soil building practices.

#### 7. its compatibility with a system of sustainable agriculture.

Ycs.

### TAP REVIEWER COMMENT FORM for USDA/NOSB

Use this page or an equivalent to write down comments and summarize your evaluation regarding the data presented in the file of this potential National List material. Complete both sides of page. Attach additional sheets if you wish.

| Name of Material: | Humic | Aeld | Derivatives      |      |
|-------------------|-------|------|------------------|------|
| Reviewer Name:    |       |      | RECEIVED JUL 3 0 | 1998 |
|                   |       |      |                  |      |

This material should be added to the National List as:

\_\_\_\_\_ Synthetic Allowed \_\_\_\_\_ Prohibited Natural

or, \_\_\_\_\_ Non-synthetic (This material does not belong on National List)

Are there any use restrictions or limitations that should be placed on this material on the National List?

| Please | comment | on the         | accuracy o | f the        | informatio | n in the | file: | ١ | 1 |   |
|--------|---------|----------------|------------|--------------|------------|----------|-------|---|---|---|
|        | 1.2.19  | а <u>– к</u> . |            | <del>د</del> | in drafter |          |       | 1 | 1 | ٠ |
|        | in the  | ·              |            | 1.0          | i (ey.e.)  |          |       |   |   |   |

### Any additional comments? (attachments welcomed)

Confirmation of transition metals such as Co, Zn, Fe Win & others by monit is an interpreter a force of a long parties of policies. The first an of human elements (contraction of Soil Colution may continue to the megiover available elements (contraction of Soil Colution may continue to the megiover available of the policies (ed.) The take of monitor metals we have the policies of the metal Do you have a commercial interest in this material? Yes; No 173-15

Thuson Date 7/29/96 ames ( Signature

Please address the 7 criteria in the Organic Foods Production Act: (comment in those areas you feel are applicable)

 the potential of such substances for detrimental chemical interactions with other materials used in organic farming systems;

124

(

( )

- (2) the toxicity and mode of action of the substance and of its breakdown products or any contaminants, and their persistence and areas of concentration in the environment;
- (3) the probability of environmental contamination during manufacture, use, misuse or disposal of such substance;
- (4) the effect of the substance on human health;

(S) the effects of the substance on biological and chemical interactions in the agroecosystem, including the physiological effects of the substance on soil organisms (including the salt index and solubility of the soil), crops and livestock;

Lines and Bring Schutzer Coldina and

(6) the alternatives to using the substance in terms of practices or other available materials; and

(7) its compatibility with a system of sustainable agriculture.

Very compative

# **NOSB Materials Database**

Chemistry

# **Identification**

### Common Name Humic Acid Derivatives

Other Names

Code #: CAS

N. L. Category UN

unknown

# Chemical Name

Code #: Other

MSDS ⊖yes ⊛no

1

-----

Family

Composition Properties How Made

HOW Made

Type of Use Crops

**Use/Action** 

Specific Use(s) Action

Combinations

Status

. . . . .

• • •

. . ..

OFPA

N. L. Restriction EPA, FDA, etc

Directions

Safety Guidelines

Historical status InternationI status

------

# **Humic Acids**

Crops

|  | Identification of Petitioned Substance  |   |  |  |  |  |  |
|--|---|---|--|--|--|--|--|
| Chamical Names   | 14  | Trade Names.  |  |  |  |  |  |
| NA   | 15  | NA  |  |  |  |  |  |
|  |   |   |  |  |  |  |  |
| Other Name:  |   | CAS Numbers:  |  |  |  |  |  |
| Humic acid   |   | 1415-93-6   |  |  |  |  |  |
| Humic acid, sodium salt  |   | 68131-04-4  |  |  |  |  |  |
| Humic acid, potassium salt   |   | 68514-28-3  |  |  |  |  |  |
| Humates  |   | (EPA, 2004)   |  |  |  |  |  |
|  |   | Other Codes:  |  |  |  |  |  |
|  |   | NA  |  |  |  |  |  |
|  |   |   |  |  |  |  |  |
| Characterizatio  | on of Pe  | etitioned Substance   |  |  |  |  |  |
|  |   |   |  |  |  |  |  |
| Composition of the Substance:  |   |   |  |  |  |  |  |
| Humis substances are a group of complay areas  | nic cor   | nounde consisting of humic scide fulvic scide   |  |  |  |  |  |
| natural salts of these acids (a.g. calcium humat   | ee) and   | sponge-like substances called humin (Weber  |  |  |  |  |  |
| undated) Humic substances (which includes h  | umic a  | cids) naturally constitutes a large fraction of the   |  |  |  |  |  |
| organic matter in soil and is formed through the   | ie proce  | ess known as "humification." Humification is th   |  |  |  |  |  |
| natural conversion of organic matter into humi   | c substa  | ances by microorganisms in the soil (Mayhew,  |  |  |  |  |  |
| 2004). This process begins with microorganism  | is separ  | ating out sugars, starches, proteins, cellulose, an   |  |  |  |  |  |
| other carbon compounds from the organic mat  | ter. The  | e microorganisms use these components in their  |  |  |  |  |  |
| own metabolic processes. Subsequently, the mi  | icroorg   | anisms transform the majority of the organically  |  |  |  |  |  |
| bound nutrients into a mineral form that are us  | ed by p   | plants and other organisms. The portions of the   |  |  |  |  |  |
| organic matter that are not digested by the micr   | roorgai   | bound nutrients into a mineral form that are used by plants and other organisms. The portions of the organic matter that are not digested by the microorganisms accumulate as humic substances (Sachs).   |  |  |  |  |  |
| organic matter that are not digested by the inicroorganisms accumulate as numic substances (Sachs, undated). Humification does not occur in one step, but involves an intermediate substance called  |   |   |  |  |  |  |  |
| undated). Humification does not occur in one s   | step, bu  | nisms accumulate as humic substances (Sachs,<br>it involves an intermediate substance called  |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic  | step, bu<br>substar   | nisms accumulate as humic substances (Sachs,<br>it involves an intermediate substance called<br>inces and partially decomposed organic matter. A  |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic<br>the humification process proceeds, various cher   | step, bu<br>substar<br>micals (   | nisms accumulate as humic substances (Sachs,<br>it involves an intermediate substance called<br>nces and partially decomposed organic matter. A<br>dominate at different times until conversion to  |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic s<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).   | step, bu<br>substar<br>micals (   | nisms accumulate as humic substances (Sachs,<br>It involves an intermediate substance called<br>Inces and partially decomposed organic matter. A<br>dominate at different times until conversion to   |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic s<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is c   | step, bu<br>substar<br>micals o   | nisms accumulate as humic substances (Sachs,<br>it involves an intermediate substance called<br>ices and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of   |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic s<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is c<br>available free oxygen, the amount of moisture t  | step, bu<br>substar<br>micals<br>depend   | nisms accumulate as humic substances (Sachs,<br>it involves an intermediate substance called<br>nces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The  |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic s<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is c<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aer  | step, bu<br>substar<br>micals<br>depend<br>present<br>obic or   | nisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the   |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humics<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is c<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aer<br>decomposition process. Aerobic microorganism   | step, bu<br>substar<br>micals of<br>lepend<br>present<br>obic or<br>ns deco   | nisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do   |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humic s<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is of<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aero<br>decomposition process. Aerobic microorganism<br>anaerobic organisms. However, greater amour   | step, bu<br>substar<br>micals<br>depend<br>present<br>obic or<br>ns deco<br>nts of h  | nisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by   |  |  |  |  |  |
| undated). Humification does not occur in one a<br>compost, which consists of a mixture of humic a<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is of<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aer<br>decomposition process. Aerobic microorganism<br>anaerobic organisms. However, greater amour<br>anaerobic organisms because in these condition  | step, bu<br>substar<br>micals of<br>present<br>obic or<br>ns deco<br>nts of his<br>accus  | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic  |  |  |  |  |  |
| <ul> <li>undated). Humification does not occur in one s<br/>compost, which consists of a mixture of humics<br/>the humification process proceeds, various cher<br/>humic substances is complete (Mayhew, 2004).</li> <li>The decomposition of organic matter in soil is c<br/>available free oxygen, the amount of moisture p<br/>amount of free oxygen determines whether aer<br/>decomposition process. Aerobic microorganism<br/>anaerobic organisms. However, greater amour<br/>anaerobic organisms because in these condition<br/>substances. Although microorganisms need more</li> </ul>  | step, bu<br>substar<br>micals of<br>present<br>obic or<br>ns deco<br>nts of his<br>accur<br>oisture   | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can   |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humics<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is d<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aer<br>decomposition process. Aerobic microorganism<br>anaerobic organisms. However, greater amour<br>anaerobic organisms because in these condition<br>substances. Although microorganisms need mod<br>decrease the rate of decomposition. Increasing  | step, bu<br>substar<br>micals of<br>present<br>obic or<br>ns deco<br>nts of hi<br>as accur<br>oisture<br>soil ter   | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and  |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humics<br>the humification process proceeds, various cher<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is of<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aer<br>decomposition process. Aerobic microorganism<br>anaerobic organisms. However, greater amount<br>anaerobic organisms because in these condition<br>substances. Although microorganisms need mod<br>decrease the rate of decomposition. Increasing<br>decreased humic substance content because dec   | step, bu<br>substar<br>micals of<br>present<br>obic or<br>ns deco<br>nts of hi<br>ns accur<br>oisture<br>soil ter<br>composition  | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulat   |  |  |  |  |  |
| <ul> <li>undated). Humification does not occur in one s<br/>compost, which consists of a mixture of humics<br/>the humification process proceeds, various cher<br/>humic substances is complete (Mayhew, 2004).</li> <li>The decomposition of organic matter in soil is of<br/>available free oxygen, the amount of moisture p<br/>amount of free oxygen determines whether aere<br/>decomposition process. Aerobic microorganism<br/>anaerobic organisms. However, greater amount<br/>anaerobic organisms because in these condition<br/>substances. Although microorganisms need mod<br/>decrease the rate of decomposition. Increasing<br/>decreased humic substance content because dec<br/>(Sachs, undated).</li> </ul>   | step, bu<br>substar<br>micals of<br>present<br>obic or<br>ns deco<br>nts of hi<br>as accur<br>oisture<br>soil ter<br>composition  | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulat   |  |  |  |  |  |
| undated). Humification does not occur in one s<br>compost, which consists of a mixture of humics<br>the humification process proceeds, various cher-<br>humic substances is complete (Mayhew, 2004).<br>The decomposition of organic matter in soil is c<br>available free oxygen, the amount of moisture p<br>amount of free oxygen determines whether aer-<br>decomposition process. Aerobic microorganism<br>anaerobic organisms. However, greater amour<br>anaerobic organisms because in these condition<br>substances. Although microorganisms need mo<br>decrease the rate of decomposition. Increasing<br>decreased humic substance content because dec<br>(Sachs, undated).   | step, by<br>substar<br>micals of<br>present<br>obic or<br>ns decc<br>nts of his<br>accur<br>oisture<br>soil ter<br>compos   | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulat   |  |  |  |  |  |
| <ul> <li>undated). Humification does not occur in one s<br/>compost, which consists of a mixture of humics<br/>the humification process proceeds, various cher<br/>humic substances is complete (Mayhew, 2004).</li> <li>The decomposition of organic matter in soil is d<br/>available free oxygen, the amount of moisture p<br/>amount of free oxygen determines whether aer<br/>decomposition process. Aerobic microorganism<br/>anaerobic organisms. However, greater amour<br/>anaerobic organisms because in these condition<br/>substances. Although microorganisms need modecrease the rate of decomposition. Increasing<br/>decreased humic substance content because dec<br/>(Sachs, undated).</li> <li>In addition to humic substances originating fro</li> </ul>   | step, bu<br>substar<br>micals of<br>depend<br>present<br>obic or<br>ns deco<br>nts of h<br>is accur<br>oisture<br>soil ter<br>composition<br>m soil,  | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulat   |  |  |  |  |  |
| <ul> <li>undated). Humification does not occur in one s<br/>compost, which consists of a mixture of humics<br/>the humification process proceeds, various chen<br/>humic substances is complete (Mayhew, 2004).</li> <li>The decomposition of organic matter in soil is d<br/>available free oxygen, the amount of moisture p<br/>amount of free oxygen determines whether aer<br/>decomposition process. Aerobic microorganism<br/>anaerobic organisms. However, greater amour<br/>anaerobic organisms because in these condition<br/>substances. Although microorganisms need modecrease the rate of decomposition. Increasing<br/>decreased humic substance content because dec<br/>(Sachs, undated).</li> <li>In addition to humic substances originating fro<br/>derived from coal. Leonardite is a highly oxidi</li> </ul>   | step, bu<br>substar<br>micals of<br>present<br>obic or<br>ns deco<br>nts of hi<br>ns accur<br>oisture<br>soil ter<br>compos<br>m soil,<br>ized low  | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>hoces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulat  |  |  |  |  |  |
| <ul> <li>undated). Humification does not occur in one s compost, which consists of a mixture of humic s the humification process proceeds, various chern humic substances is complete (Mayhew, 2004).</li> <li>The decomposition of organic matter in soil is c available free oxygen, the amount of moisture p amount of free oxygen determines whether aero decomposition process. Aerobic microorganism anaerobic organisms. However, greater amour anaerobic organisms because in these condition substances. Although microorganisms need modecrease the rate of decomposition. Increasing decreased humic substance content because dec (Sachs, undated).</li> <li>In addition to humic substances originating fro derived from coal. Leonardite is a highly oxidi serves as a key mined source for the production.</li> </ul>   | step, bu<br>substar<br>micals of<br>depend<br>present<br>obic or<br>ns deco<br>nts of his<br>accur<br>oisture<br>soil ter<br>composi-<br>om soil,<br>ized low<br>n of hus   | hisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulat<br>commercially available humic substances are<br>w-rank coal originating from plant matter that<br>mic acids. The humification process that yields   |  |  |  |  |  |
| <ul> <li>undated). Humification does not occur in one s compost, which consists of a mixture of humic s the humification process proceeds, various chern humic substances is complete (Mayhew, 2004).</li> <li>The decomposition of organic matter in soil is c available free oxygen, the amount of moisture p amount of free oxygen determines whether aero decomposition process. Aerobic microorganism anaerobic organisms. However, greater amour anaerobic organisms because in these condition substances. Although microorganisms need modecrease the rate of decomposition. Increasing decreased humic substance content because dec (Sachs, undated).</li> <li>In addition to humic substances originating fro derived from coal. Leonardite is a highly oxidi serves as a key mined source for the production leonardite may take 70 million years. In comparison process.</li> </ul> | step, bu<br>substar<br>micals of<br>lepend<br>present<br>obic or<br>ns decc<br>the obic or<br>ns decc<br>the obic or<br>ns decc<br>the obic or<br>soil ter<br>composi-<br>om soil,<br>ized low<br>n of hus<br>arison, | tisms accumulate as humic substances (Sachs,<br>at involves an intermediate substance called<br>aces and partially decomposed organic matter. A<br>dominate at different times until conversion to<br>ent on several factors, including the amount of<br>in the soil, and the temperature of the soil. The<br>anaerobic microorganisms will conduct the<br>ompose organic material at a faster rate than do<br>umic substances are found in soils produced by<br>mulation is favored over destruction of humic<br>to function, too much or too little water can<br>nperature leads to greater microbial activity and<br>sition is occurring at a faster rate than accumulati<br>commercially available humic substances are<br>v-rank coal originating from plant matter that<br>mic acids. The humification process that yields<br>peat can be formed in about a few thousand year |  |  |  |  |  |

.....

( )

Technical Evaluation Report

53 much higher humic/fulvic acid content than other sources (such as black peat at 10 to 40 percent, 54 sapropel peat at 10 to 20 percent, manure at 5 to 15 percent, and compost at 2 to 5 percent). Natural soil 55 typically has 1 to 5 percent humic and fulvic acids. Additionally, in contrast to compost, leonardite-56 sourced humic acids do not compete with plants for nutrients, because leonardite-sourced humic acids 57 are completely decomposed (Humintech, undated).

58

70 71

72

1 (

Humic acids are not a substance with a single molecular formula. Figure 1 shows one example of a humic 59 acids molecule. Humic acids may be described as "complex colloidal supermixtures" (Mayhew, 2004) 60 that are characterized by their functional groups (Heise and Brendler., undated). The composition and 61 structure of humic acids can vary from one soil to another (Sachs, undated). Generaly, humic acids are 62 considered to be aromatic in structure with amino acids, amino sugars, peptides, and aliphatic 63 compounds linking the aromatic groups. The hypothetical structure of humic acid, as shown by the 64 example in Figure 1, is believed to consist of free and bound phenolic hydroxyl groups, quinines, oxygen 65 and nitrogen bridges, and carboxy groups (Weber undated). Adding to their complexity, the structure of 66 humic acids is continuously influenced by their surroundings. For example, changes in pH can result in 67 broken hydrogen bonds. (Heise and Brendler, undated). 68 69





73 Source: Weber, Jerzy. Undated. Definition of soil organic matter. Available online at:

<u>http://www.humintech.com/001/articles/article\_definition\_of\_soil\_organic\_matter.html</u>. Last Accessed:
 January 10, 2006.

76

The surface of humic substance particles consists of compounds that contain hydrogen ions capable of being replaced by cations (positively charged ions) like calcium, magnesium, potassium or sodium. In soil with high hydrogen ion activity (low pH), humic substances are saturated with hydrogen ions forming humic acid. Humic acid, in turn, reacts with mineral particles in the soil causing these particles to release basic cations (e.g., calcium, potassium, and magnesium). Humic acid then replaces its hydrogen ions with the basic cations and is converted into a salt called humate (Sachs, undated).

# 8485 Properties of the Substance:

86

ſ

As previously stated, humic substances consist of humic acid, fulvic acid, and a sponge-like 87 substance called humin. Humic substances are divided into three fractions on the basis of their 88 solubility characteristics. These fractions are soluble over a wide pH range. The first fraction, 89 humic acid, is not soluble in water under acidic conditions (pH < 2), but is soluble at higher pH90 values. Humic acid is the major extractable fraction of humic substances and ranges from dark 91 brown to black in color. It can be extracted from soil by various reagents. The second fraction, 92 fulvic acid, is soluble in water under all pH conditions. Fulvic acid, which stays in solution after 93 the extraction of humic acid, ranges from light yellow to yellow-brown in color. After the first two 94 fractions have been extracted, the third fraction, called humin, remains. This fraction, which is 95 black in color, is not soluble in water at any pH, nor in any alkali solution (Weber, undated). 96

Technical Evaluation Report

(\*\*\*)

Crops

| 07  |  |
|-----|--|
| 97  | Company the humin fraction of humic substances is the dominant organic material in soils and   |
| 98  | Generally, the human fraction of human substances is the dominant of game indication (Kohl and   |
| 99  | seatments. However, inde information exists regarding the properties of this nation (rectil and<br>n: 100(). Variations between humis and fulvis acids include differences in molecular weight.  |
| 100 | Kice, 1990). Variations between numer and rulere actus include differences in morecular melecular  |
| 101 | number of functional groups, and extent of polymenzation. Think and OP and OP groater carbon   |
| 102 | weight, fewer functional groups that are actor in hardre (e.g. COOT and OT), greater carbon  |
| 103 | content, and lower oxygen content than fulvic acid. Finally, the majority of oxygen in humic acid  |
| 104 | is located as a structural constituent of its nucleus, while the oxygen in fulfile acid is follow  |
| 105 | predominantly in the functional groups (Weber, undated).   |
| 106 |  |
| 107 | Specific Uses of the Substance:  |
| 108 | The second all all and the second all all all are apprendix on the National  |
| 109 | Humic acids (naturally occurring deposits, water and alkali extracts only) are currently on the radional   |
| 110 | List for use as a plant or soil amendment. Specifically, furnic actus are used by organic growers as a   |
| 111 | component of traditional fertilizers.  |
| 112 | that 1 the stand leaves a foliar spray   |
| 113 | Although numic acids are most commonly used as a son amenument, mey are also used us a roman spray   |
| 114 | (Jackson, undated). In general, plants are capable of absorbing small anounts of nutrients this way:   |
| 115 | foliar sprays on leaves. However, it is not possible to supply significant another or nutrents and may,  |
| 110 | root uptake is more encient (Whiley, 1990). Application of numer actuals a tonal spray a senerou to  |
| 11/ | promote the photosynthesis of leaves, increase yield and quality of phints, promote root develop methy   |
| 110 | and improve numeric uptake unough the root system (runtareets me) and access   |
| 119 | Commercially available humic substances do not provide additional nutrients to plants, but rather affect   |
| 120 | coil fortility by making micronutrients (e.g. iron) more readily available to plants. By chelating (or   |
| 121 | binding) putrients (especially iron), humic substances cause insoluble and unstable, and therefore   |
| 122 | unavailable compounds in the soil to remain available for plant uptake (Obreza et al, 1989).   |
| 123 |  |
| 124 | The use of humic acids for agricultural purpose continues to grow, and as result, the number of products   |
| 125 | and vendors continues to grow. However, there is currently a lack of standardized analysis for   |
| 127 | substances marketed as humic substances, resulting in the marketing of some products that produce  |
| 128 | minimal to no results. However, the benefits of using humic acid substances in agriculture have been   |
| 129 | researched and documented by scientists (Mayhew, 2004).  |
| 130 |  |
| 131 | Approved Legal Uses of the Substance:  |
| 132 |  |
| 133 | As stated above, humic acids (naturally occurring deposits, water and alkali extracts only) are currently  |
| 134 | on the National List for use as a plant or soil amendment.   |
| 135 |  |
| 136 | The US Environmental Protection Agency (EPA) granted an exemption from the requirement for a   |
| 137 | tolerance for residues of humic acids, sodium salts, used as an inert ingredient (adjuvant, UV protectant)   |
| 138 | in pesticide formulations applied to growing crops and raw agricultural commodities after harvest (ErA,  |
| 139 | 2000).   |
| 140 | a sease to TRA to the table of tabl |
| 141 | In 2003, the EPA proposed to amend the existing tolerance exemption for numic actu, southin sait to  |
| 142 | include numic acid, potassium sait and numic acid. The EFA stated that such number that Enderal Food   |
| 143 | used as mert ingredients in pesticide formulations applied to growing crops under the Federal Food,  |
| 144 | Drug, and Cosmence Act (FFDCA), as amended by me rood Quanty From the need to establish a  |
| 145 | 2003). In 2004, the existing toterance exemption was affectude eminiating the fact to establish a  |
| 140 | maximum permissible level for residues of numic acid, numic acid, southin said, and numic acid,  |
| 14/ | potassium san (ETA, 2004).   |
| 140 |  |

 $\lambda = j l$ 

Page 3 of 10

|                 | Technical Evaluation Report   | Humic Acids   | Crops                  |
|-----------------|---|---|------------------------|
| 149             | Action of the Substance:  |   |                        |
| 150             | )   |   |                        |
| 151             | <ol> <li>According to Mayhew (2004), hur</li> </ol>   | nic substances have demonstrated the ability to:        |                        |
| 152             | 2   |   |                        |
| 153             | <ul> <li>Chelate (bind) soil nutries</li> </ul>   | nts;  |                        |
| 154             | <ul> <li>Improve nutrient uptake;</li> </ul>  |   |                        |
| 155             | <ul> <li>Reduce the need for nitro</li> </ul>   | gen fertilizer;   |                        |
| 156             | <ul> <li>6 • Remove toxins from soils</li> </ul>  | ;   |                        |
| 157             | <ul> <li>Stimulate soil biological a</li> </ul>   | ctivity;  |                        |
| 158             | <ul> <li>Solubilize minerals;</li> </ul>  |   |                        |
| 159             | <ul> <li>Improve soil structure; at</li> </ul>  | ıd  |                        |
| 160             | <ul> <li>Improve water holding ca</li> </ul>  | ipacity.  |                        |
| 16.             | 1   |   |                        |
| 162             | 2 In general, commercially-availabl   | e humic substances do not promote plant growth by       | providing              |
| 163             | 3 substantial amounts of nutrients to substantis amounts of nutrients to substantial amounts of nut | o plants. Instead, humic substances affect soil fertili | ty by making           |
| 164             | 4 nutrients (e.g., iron) more readily   | available to plants. In order for plants to take up nu  | trients, the           |
| 165             | 5 nutrients need to be in solution, or  | r dissolved in water. However, nutrients predomina      | intly exist in soil as |
| 166             | 6 the insoluble forms of soil minera  | ls and organic matter. Humic substances, which are      | negatively charged,    |
| 16'             | 7 make nutrients more available by  | attracting the positively charged nutrients and hold    | ing them in reserve.   |
| 168             | 8 These readily available nutrients   | are subsequently released into solution to replace nu   | trients taken up by    |
| 169             | 9 the plant roots (Cogger, 2000; Ob   | reza et al., 1989; Senn and Kingman, undated).          |                        |
| 170             | 0   |   |                        |
| 17              | <ol> <li>Humic acids also promote plant §</li> </ol>  | rowth by enabling root penetration in soils with hig    | h clay content.        |
| 172             | 2 These types of soil can become ex   | tremely dense and compact due to salts located on the   | heir surface. The      |
| 173             | 3 salts cause the negatively-charge   | l clay particles to become neutral and move together    | . This compaction      |
| 174             | 4 can create resistance to plant root   | ing. The addition of humic acids results in the remo    | val of the salts,      |
| 17:             | 5 which causes the clay soil to loos  | en up for greater root penetration (Bio Ag, 1999). See  | d germination and      |
| ( j <b>17</b> ) | 6 top growth also are stimulated (C   | breza et al 1989). In the presence of humic acid, both  | n a larger             |
| 17              | 7 percentage of seeds germinate an  | d germination occurs at a faster rate. This increased   | rate and occurrence    |
| 173             | 8 of germination is related to the g  | eater efficiency of binding of nutrients and water that | it takes place in the  |
| 179             | 9 presence of humic substances (Bi  | 5 Ag, 1999).  |                        |
| 18              |   | 1 dia (ha maniana manahallarua far araatar)             | unter population in    |
| 18              | Additionally, the same action des   | crited in the previous paragraph allows for greater of  | sontial in arid areas  |
| 18              | 2 clay soils. Humic acid also acts in   | a decreasing water evaporation from sons. This is es    | t wara proviouely      |
| 18              | 3 with sandy soils that retain little   | to no water. With water present, the compounds that     | l compound's           |
| 184             | 4 bound by numic acid are partial   | restored Subsequently the negatively charged over       | ren atom of the        |
| 18.             | 5 positive attractive force is party.   | the positively charged compound while the bydroge       | on and of the water    |
| 18              | 6 water molecule loosely bolids to  | This loads to another negatively charged oxygen fro     | om a water molecule    |
| 18              | / molecule becomes more positive.   | bydrogon ion of the original water molecules. This      | continues until the    |
| 10              | 8 binding to the positively charged   | The inverse stabilized (Bio $\Delta \alpha$ 1999)       | continues inter are    |
| 10              | o attractive forces of the water mor  | ectiles are statistized (Dio rig, 1999)                 |                        |
| 19              | V   |   |                        |
| 19              | 1   | Status  |                        |
| 19              | 2   |   |                        |
| 19              | 3 <u>International</u>  |   |                        |
| 19              | 4   |   |                        |
| 19              | 5 Canada - Canadian General Stan  | dards Board -   |                        |
| 19              | 6 <u>http://www.pwgsc.gc.ca/cgsb/</u>   | <u>032_310/32.310epat.pdf</u>                           |                        |
| 19              | 7 No information was identified a   | t the listed site.                                      |                        |
| 19              | 8   |   |                        |
| 19              | 9 The Certified Organic Associatio  | ns of British Columbia (undated) allows:                |                        |
| 20              | 0   |   |                        |
| 20              | <ul> <li>Humates (if not fortified</li> </ul>   | with synthetic nutrients); and                          |                        |
| · ·             |   |   |                        |

1.1

|     |                                 | Technical Evaluation Report   | Humic Acids  | Crops   |
|-----|---------------------------------|---|--|---|
|     | 202<br>203                      | <ul> <li>Humic acid derivatives (no fortify the product – no otherward)</li> </ul>  | n-synthetic or using potassium hydroxide as an extra<br>er sources are allowed)  | actant but not to                                       |
| ÷   | 204<br>205<br>206               | They prohibit humic acids extracte potassium hydroxide.   | d by ammonium or soium hydroxide or synthetic bas  | ses other than  |
|     | 207<br>208<br>209               | CODEX Alimentarius Commissio  | n –<br>/ <u>Y2772e/Y2772e.pdf</u>  |   |
|     | 210<br>211                      | No information was identified at the  | ie listed site.  |   |
|     | 212<br>213<br>214<br>215        | European Economic Community (<br>http://europa.eu.int/eur-lex/en/e<br>No information was identified at th   | EEC) Council Regulation 2092/91 -<br><u>consleg/pdf/1991/en_1991R2092_do_001.pdf</u><br>ne listed site.  |   |
|     | 213<br>216<br>217<br>218<br>219 | Humic acids are not allowed in the<br>from certain fossilized organic mat<br>more common in recognized third<br>2002).                              | EU. However, some certifiers/authorities may cons<br>er to be 'peat' products, which are covered under An<br>countries like Hungary than in the EU (Organic Trad   | ider the extracts<br>nex IIA. This is<br>le Association |
|     | 220<br>221<br>222<br>223        | Japan Agricultural Standard for O<br>http://www.ams.usda.gov/nop/<br>No information was identified at t   | <b>Frganic Production</b> —<br><u>NOP/TradeIssues/JAS.html</u><br>ne listed site.  |   |
|     | 224<br>225<br>226<br>227        | California Certified Organic Grov<br>http://www.ccof.org/pdf/Global   | vers International (CCOF) -<br>MarketAccessDraftForReview.pdf  |   |
| 1 ) | 228<br>229<br>230               | According to the 2005 CCOF's Dra<br>acids are prohibited and/or restric<br>International Federation of Organi                                       | ft Manual III: Global Market Access Program, alkali-<br>ted on crops for the USDA/Export, European Union<br>c Agriculture Movements (IFOAM) export programs  | extracted humic<br>Export, and                          |
|     | 231<br>232<br>233<br>234        | Washington State Department of<br>http://agr.wa.gov/FoodAnimal/   | Agriculture: European Organic Verification Program   | m (EOVP) -  |
|     | 235<br>236<br>237               | According to this program, alkali-<br>exported to Japan.  | extracted humic acid may not be used on raw or proc  | essed organic food                                      |
|     | 238                             | Evaluation Questions for S  | Substances to be used in Organic Crop or Livestock   | Production  |
|     | 239<br>240<br>241               | <i>Evaluation Question #1:</i> Is the pe<br>process? (From 7 U.S.C. § 6502 (21  | titioned substance formulated or manufactured by a<br>))   | a chemical  |
|     | 242<br>243<br>244<br>245<br>246 | Extraction of humic substances fro<br>other coals (Karr, 2001), is commo<br>(Original TAP Database Form, 199<br>paragraphs:                         | m terrestrial sources, such as soil, peat, compost, oxionly conducted using potassium hydroxide, water, or 5). Various extraction processes are described in the   | dized lignites, and<br>(rarely) ammonia<br>following    |
|     | 247<br>248                      | Alkali Extraction of Humic Subs   | ances:   |   |
|     | 249<br>250<br>251<br>252<br>253 | The process begins with the separ<br>and clay. The terrestrial source is<br>other positively-charged ions and<br>alkaline reagents. Next, a stronge | ation of organic matter from the inorganic matrix of s<br>leached with hydrochloric acid (HCL) to remove calc<br>to increase the efficiency of extraction of organic mat<br>r sodium hydroxide solution is used to create a liquic | and, silt,<br>ium and<br>iter with<br>I solution        |
| 1   | 254<br>255<br>256               | (Weber, undated). The extracted l<br>alkaline, in the range of 8 to 12 pH<br>acid portion (Mayhew, 2004).   | iquid solution is incompatible with acids because it is<br>., and can be treated with an acid to precipitate out th  | s very<br>1e humic                                      |

|       | 257   |   |
|-------|-------|---|
| <br>1 | 258   | Alkali extraction can also be conducted using potassium hydroxide, which is a typical alkali used by        |
|       | 259   | manufacturers to extract humic acid from leonardite. The extracted liquid solution is also incompatible     |
|       | 260   | with acids because it is very alkaline, in the range of 8 to 12 pH, and can be treated with an acid to      |
|       | 261   | precipitate out the humic acid portion (Mayhew, 2004).  |
|       | 262   |   |
|       | 263   | Processes Inconsistent with the Current Listing   |
|       | 264   |   |
|       | 265   | Continued experimentation has led to development of a recently patented process that solubilizes the        |
|       | 266   | "humin" fraction of leonardite and mixes it with liquid phosphate fertilizer. This solution is used as an   |
|       | 267   | early-season soil treatment or as a foliar spray, and enhances the efficiency of the phosphate in the       |
|       | 268   | fertilizer. (Kline and Wilson, 1994)  |
|       | 269   |   |
|       | 270   | Lignite and other coals serve as a natural source that can now be synthetically oxidized to produce         |
|       | 271   | biochemically active humic substances. The coal is converted to humic substances through either "dry or     |
|       | 272   | wet oxidative depolymerization" or "nitric acid oxidation and ammonation."                                  |
|       | 272   | net ontaal to acpely meneration and a second  |
|       | 274   | A recent inpovation in extraction of humic acid uses microbial digestion of lignite to form a humic         |
|       | 275   | substance with the trade name Actosol (Kline and Wilson, 1994).   |
|       | 275   |   |
|       | 210   | Evaluation Question #2: Is the petitioned substance formulated or manufactured by a process that            |
|       | 278   | chemically changes the substance extracted from naturally occurring plant, animal, or mineral sources?      |
|       | 270   | (From 7 II S.C. § 6502 (21).)   |
|       | 280   |   |
|       | 281   | Humates and humic acids are extremely complex and varied in chemical structure. Some chemical               |
|       | 201   | reactions do occur during the processes by which humic materials are obtained from terrestrial sources,     |
|       | 202   | such as soil peat compost oxidized lignites, and other coals (Karr, 2001). However, it may not be           |
|       | 287   | possible to characterize the reactions and the extent to which there is a chemical change beyond the        |
| 1 1   | 204   | simple effects of shifting pH on hydrogen ion availability for displacement.                                |
|       | 205   | simple circles of similing prior try diogen for a value sincy for engineering                               |
|       | 280   | Evaluation Question #3: Is the netitioned substance created by naturally occurring biological               |
|       | 287   | processes? (From 7 U.S.C. 8 6502 (21).)   |
|       | 289   |   |
|       | 202   | In nature, organic matter is converted into humic substances by microorganisms. This natural                |
|       | 291   | humification process, however, does not occur as a result of the specific process (i.e., application of     |
|       | 292   | NaOH) used for commercial manufacturing.  |
|       | 293   | Nuclif) used for commercial manufacturing.  |
|       | 292   | Evaluation Question #4: Is there environmental contamination during the petitioned substance's              |
|       | 295   | manufacture, use, misuse, or disposal? (From 7 U.S.C. § 6518 (m) (3).)                                      |
|       | 296   |   |
|       | 297   | Humic acids are a natural substance that can also be manufactured from natural sources (e.g., coals).       |
|       | 298   | There is no information available from EPA to suggest that environmental contamination results from the     |
|       | 299   | manufacture, use, misuse, or disposal. Improper disposal of acids or bases used in the extraction process   |
|       | 300   | could be a source of environmental contamination, and the mining of lignite/leonardite or other source      |
|       | 301   | materials has environmental impacts.  |
|       | 302   |   |
|       | 302   | Evaluation Question #5: Is the petitioned substance harmful to the environment? (From 7 U.S.C. §            |
|       | 304   | 6517 (c) (1) (A) (i) and 7 U.S.C. § 6517 (c) (2) (A) (i).)  |
|       | 305   |   |
|       | 306   | No. Humic acids are a primary component of soil. Humic acids are nearly completely broken down by           |
|       | 307   | soil organisms, and are not know to produce toxicity to plants, soil organisms, or higher animals (Original |
|       | 308   | TAP Database Form, 1995). According to Humintech (undated), humic acids will not harm soil or               |
|       | 300   | contaminate groundwater or soil. As discussed above (see "Action of the Substance"), humic acids have a     |
|       | 310   | number of beneficial properties in soil.  |
|       | 311   | number of Statement Propagato inter-  |
|       | J I I |   |
Technical Evaluation Report

|     | 312 | Evaluation Question #6: Is there potential for the petitioned substance to cause detrimental chemical  |
|-----|-----|--|
| C   | 313 | interaction with other substances used in organic crop or livestock production? (From 7 U.S.C. § 6518  |
|     | 314 | (m) (1).)  |
|     | 315 |  |
|     | 316 | Based on the intended use of the substance, no information was uncovered to suggest that use of humic  |
|     | 317 | substances could cause detrimental chemical interaction with other substances used in organic crop   |
|     | 318 | production. Humic acids are naturally occurring and are a primary component of soil.   |
|     | 319 |  |
|     | 320 | Evaluation Question #7: Are there adverse biological or chemical interactions in the   |
|     | 321 | agrorecosystem by using the petitioned substance? (From 7 U.S.C. § 6518 (m) (5).)  |
|     | 321 | agro-cosystem by asing the permonent substances (  |
|     | 322 | Based on the intended use of humic acids, no evidence of adverse biological or chemical interaction in the   |
|     | 223 | based on the interfaced use of number actus, no evidence of adverse biological of chemical interfaced  |
|     | 324 | agro-ecosystem was identified.   |
|     | 325 | E - Insting Ownsting #9. Are there detrimonted abusical offects on soil arganisms groups or  |
|     | 326 | Evaluation Question #6: Are there detrimental physiological effects on soli organisms, crops, or   |
|     | 327 | Investock by using the permoned substance: (From 7 0.5.C. 9 6516 (m) (5).)   |
|     | 328 | out the first of the little of the little of the state of the Contesting ITAP Detabases file (Out at the State of the 1995)                              |
|     | 329 | Soil organisms are affected positively by the addition of numic acids (Original TAP Database file, 1995).  |
|     | 330 | Crops generally benefit from, or do not react to, addition of numic acids.   |
|     | 331 |  |
|     | 332 | Evaluation Question #9: Is there a toxic or other adverse action of the petitioned substance or its  |
|     | 333 | breakdown products? (From 7 U.S.C. § 6518 (m) (2).)  |
|     | 334 |  |
|     | 335 | Soil humic acids in nature undergo biochlorination, facilitated by ubiquitous soil enzyme  |
|     | 336 | chloroperoxidase. The reaction yields chlorinated humic acid, which in turn is converted to  |
|     | 337 | chlorophenols, chloraceitc acids, and chloroform. The chlorophenols have been shown in nature to   |
|     | 338 | dimerize to form dioxins. However, this process is natural, and since humic acid is a primary component  |
|     | 339 | of soil the intended use would not significantly affect the environment through this process. (Euro Chlor,   |
| ( ) | 340 | undated)   |
|     | 341 | '  |
|     | 342 | Evaluation Question #10: Is there undesirable persistence or concentration of the petitioned substance   |
|     | 343 | or its breakdown products in the environment? (From 7 U.S.C. § 6518 (m) (2).)  |
|     | 344 | •  |
|     | 345 | Humic acids are nearly completely broken down by soil organisms, and are not known to produce  |
|     | 346 | toxicity to plants, soil organisms, or higher animals (Original TAP Database Form, 1995).  |
|     | 347 |  |
|     | 348 | Evaluation Ouestion #11: Is there any harmful effect on human health by using the petitioned   |
|     | 349 | substance? (From 7 U.S.C. § 6517 (c) (1) (A) (i), 7 U.S.C. § 6517 (c) (2) (A) (i)) and 7 U.S.C. § 6518 (m) (4).)   |
|     | 350 |  |
|     | 351 | Based on the intended use, no adverse effects on human health from use of humic acids were identified.   |
|     | 352 |  |
|     | 353 | Evaluation Question #12: Is there a wholly natural product which could be substituted for the  |
|     | 354 | netitioned substance? (From 7 II S C & 6517 (c) (1) (A) (ii).)   |
|     | 255 |  |
|     | 256 | Monurse and ward waste compost also contain humic acids and can be applied to soil as alternatives to  |
|     | 250 | humic acid as a fortilizer. However, no alternatives are listed for use of humic acids as a soil amendment.  |
|     | 337 | number actual as a fertilizer. Thowever, no alternatives are instea for use of number actus as a son unchannel.<br>(M = 1) of a memory line for $2002$ ) |
|     | 328 | (Maryland Cooperative Extension, 2003).  |
|     | 329 | E Insting Ourseling #12. Are there also duallowed substances that could be substituted for the   |
|     | 360 | Evaluation Question #13: Are there other already allowed substances that could be substituted for the $\frac{1}{1000}$                                   |
|     | 361 | petitionea substance: (From / 0.5.C. 9 0510 (m) (0).)  |
|     | 362 | Other It and the first second second second by Martine all Linking local second  |
|     | 363 | Other allowed plant or soil amendments on the National List include:   |
|     | 364 |  |
|     | 365 | Aquatic plant extracts (other than hydrolyzed) - Extraction process is limited to the use of   |
|     | 366 | potassium hydroxide or sodium hydroxide; solvent amount used is limited to that amount   |
|     | 367 | necessary for extraction;  |

|   |            | Technical Evaluation Report  | Humic Acids   | Crops                       |  |  |
|---|------------|--|---|-----------------------------|--|--|
|   | 260        | - Elemental culture  |   |                             |  |  |
|   | 308        | • Elemental Sulfur,  | no example duct cumprogramt floctation agont                |                             |  |  |
|   | 309        | <ul> <li>Lignin suiforate - chelating agent, dust suppressant, notation agent,</li> <li>Magnesium suifate, allowed with a documented soil deficiency:</li> </ul> |   |                             |  |  |
|   | 370        | Magnesium suifate - allowed with a documented soil deficiency;   |   |                             |  |  |
|   | 3/1        | • Micronutrients - not to be used as a defoliant, nerbicide, or desiccant. Those made from intrates of   |   |                             |  |  |
|   | 3/2        | Chlorides are not allowed  | Son denciency must be documented by testing,                |                             |  |  |
|   | 3/3        | <ul> <li>Soluble boron products;</li> <li>Linuid fish mendutata</li> </ul>   | n he nU adjusted with sulfurie sitrie or phosphoric         | acid. The amount of         |  |  |
|   | 3/4        | <ul> <li>Liquid fish products - call</li> <li>asid used shall not evere</li> </ul>   | d the minimum needed to lower the pH to 3.5; and            | acid. The miloun of         |  |  |
|   | 313        | With which B1 C and F (I   | In the minimum needed to lower the pri to 5.5, and          |                             |  |  |
|   | 3/0<br>277 |  | 5DA 2005).  |                             |  |  |
|   | 279        | Evaluation Question #14. Are th  | are alternative practices that would make the use           | of the petitioned           |  |  |
|   | 370        | substance unnecessary? (From '   | UISC. § 6518 (m) (6).)                                      | or the Permonent            |  |  |
|   | 380        | substance uniccessury. (rion )   |   |                             |  |  |
|   | 381        | Potential alterative practices incl  | ude composting or use of an alternative soil organic        | r soil amendment (see       |  |  |
|   | 382        | Evaluation Ouestions #12 and #1  | 3). Other soil building practices that could be cons        | idered as alternatives      |  |  |
|   | 383        | to the use of added humic acids (  | and soil amendments) include tilling, rotating crop         | s, and planting cover       |  |  |
|   | 384        | crops (Giannangelo Farms South   | west, 2005).  |                             |  |  |
|   | 385        | 1 . 0  |   |                             |  |  |
|   | 386        | References   |   |                             |  |  |
|   | 387        |  |   |                             |  |  |
|   | 388        | Bio Ag Technologies Internation  | al. 1999. Humid acid structure and properties. Av           | ailable Online at:          |  |  |
|   | 389        | http://www.phelpstek.com/gra   | <pre>phics/bioag/humic_acid.pdf. Last Accessed: Janua</pre> | ary 11, 2006.               |  |  |
|   | 390        |  |   | 01111111                    |  |  |
|   | 391        | California Certified Organic Gro   | wers International (CCOF). 2005. Draft Manual III           | : Global Market             |  |  |
|   | 392        | Access Program. Available Onli   | ne at:  | J. J                        |  |  |
|   | 393        | http://www.ccot.org/pdf/Glob   | alMarketAccessDraftForKeview.pdf. Last Accessed             | 1: January 11, 2006.        |  |  |
|   | 394        |  | British Columbia Undeted Crop production ma                 | toriale list Available      |  |  |
| 1 | 395        | Certified Organic Associations of  | brass (standards/b2y7sas13.5 htm. Last Accessed)            | January 11 2006             |  |  |
|   | 390<br>207 | Omme at: <u>www.certinedorgatic</u>  | De.ca/standards/ D2V/Secis.S.ntin. Last Accessed.           | Junuary 11, 2000.           |  |  |
|   | 308        | Corret C 2000 Soil managem   | ent for small farms Farming West of the Cascades set        | ries of the Washington      |  |  |
|   | 300        | State University Food and Farm Co  | nnections Team EB1895: 1-24.                                |                             |  |  |
|   | 400        | Divid Childenbirg I box wha I white Co   |   |                             |  |  |
|   | 401        | Euro Chlor, Undated, Naturally   | -occurring organochlormes. Available Online at:             |                             |  |  |
|   | 402        | http://www.eurochlor.org/upl   | pad/documents/document56.pdf. Last Accessed: J              | anuary 11, 2006.            |  |  |
|   | 403        |  |   | -                           |  |  |
|   | 404        | Giannangelo Farms Southwest.   | 2005. What is sustainable organic gardening? Soil h         | ouilding. Available         |  |  |
|   | 405        | Online at: <u>http://www.avant-ga</u>  | rdening.com/ogardening.htm. Last Accessed: Jan              | uary 19, 2006.              |  |  |
|   | 406        |  |   |                             |  |  |
|   | 407        | Heise, K-H and V. Brendler. Un   | lated. Humic acids as object of environmental rese          | earch. Available            |  |  |
|   | 408        | Online at:   |   |                             |  |  |
|   | 409        | http://www.humintech.com/00  | <u>1/articles/article_humic_acids_as_object_of_envir</u>    | <u>onmental_research.ht</u> |  |  |
|   | 410        | <u>ml</u> . Last Accessed: January 11, 2   | J06.  |                             |  |  |
|   | 411        |  |   | 1 / /                       |  |  |
|   | 412        | HumaTech, Inc. Undated. Hum  | ics explained. Available Unline at: <u>http://humated</u>   | <u>cn.com/ spage.ntm</u> .  |  |  |
|   | 413        | Last Accessed: January 17, 2006.   |   |                             |  |  |
|   | 414        | TToon State In TTo Jorn J. A set of the  | resinformation Available Online at                          |                             |  |  |
|   | 415        | Hummtech. Undated. Agricult  | are mornation. Available Online at:                         | record: January 11          |  |  |
|   | 410        | nttp://www.nuinintecn.com/ut   | 17 agriculture/ huormation/ general.hum. Last Acc           | coocu, january 11,          |  |  |
|   | 41/<br>/10 | 2006.  |   |                             |  |  |
|   | 418<br>/10 | Jackson W Undeted The Une  | us Cycle Available Online at:                               |                             |  |  |
|   | 419<br>700 | bttp://www.humintoch.com/00  | 11 /articles/article the humus cycle html. Last Ac          | cessed: January 11.         |  |  |
| : | 420<br>421 | 2006   | A manager and an and an and cyclestant. Fuel ne             |                             |  |  |
|   | 42.2       | 2000   |   |                             |  |  |
|   |            |  |   |                             |  |  |

Humic Acids Crops Technical Evaluation Report Karr, M. 2001. Oxidized lignites and extracts from oxidized lignites in agriculture. Available Online at: 423 http://humates.com/Humates%20in%20Agriculture%20-%20Karr.pdf. Last Accessed: January 19, 2006. 424 425 Klime, S.W and C.E.Wilson. 1994. Proposal for experimentation with Arkansas lignite to identify organic 426 soil supplements suitable to regional agricultural needs. Available Online at: 427 http://www.humintech.com/001/articles/article\_arkansas\_tech\_university.html. Last Accessed: 428 429 January 11, 2006. 430 Kohl, S. and J.A. Rice. 1996. The binding of organic contantinants to humin. Available Online at: 431 http://www.engg.ksu.edu/HSRC/96Proceed/kohl.pdf. Last Accesses: January 17, 2006. 432 433 434 Maryland Cooperative Extension. 2003. Soil amendments and fertilizers. Home and Garden Mimeo #42. 435 Mayhew, L. 2004. Humic substances in biological agricultural systems. Acres 34(1&2). 436 437 Obreza, T.A., R.G. Webb, and R.H. Biggs. 1989. Humate materials: their effects and use as soil 438 439 amendments. At www.livearth.com/articles/art4.htm. 440 Organic Trade Association. 2002. Compariative Analysis of the United States NationalOrganic Program 441 (7 CFR 205) and the European Union Organic Legislation (EEC2092/91 & Amendments). Available 442 Online at: http://www.ota.com/pics/documents/NOPEUunifiedreport.pdf. Last Accessed: January 11, 443 444 2006. 445 446 Original TAP Database. 1995. from NOSP Materials Database. 447 Sachs, P. Undated. Humus: still a mystery? Available Online at: 448 http://www.humintech.com/001/articles/article\_humus\_still\_a\_mystery.html. Last Accessed: January 449 11,2006. 450 451 452 Senn, T.L., A.R. Kingman, and W.C. Godley. Undated. A review of humus and humic acids. Clemson 453 University Horticulture Department Research Series No. 165. Available Online at: 454 www.livearth.com/articles/art2.htm. Last Accessed: January 11, 2006. 455 U.S. Department of Agriculture's National Organic Program (NOP). 2005. National List: Regulatory 456 Text. Subpart G - Admistrative. The National List of Allowed and Prohibited Substances. § 205.600 457 Evaluation criteria for allowed and prohibited substances, methods, and ingredients. Available Online at: 458 http://www.ams.usda.gov/nop/NOP/standards/ListReg.html. Last Accessed: January 11, 2006. 459 460 U.S. Environmental Protection Agency. 2000. Humic Acid, Sodium Salt, Exemption Tolerance. Federal 461 Register 65(138): 44469-44472. Available Online at: http://www.epa.gov/fedrgstr/EPA-462 463 PEST/2000/July/Day-18/p18097.htm. Last Accessed: January 11, 2006. 464 465 U.S. Environmental Protection Agency. 2003. Humates; Exemption from the Requirement of Tolerance. Federal Register 68(114): 35349-35354. Available Online at: http://www.epa.gov/fedrgstr/EPA-466 PEST/2003/June/Day-13/p14881.htm. Last Accessed: January 11, 2006. 467 468 U.S. Environmental Protection Agency. 2004. Humates; Exemption from the Requirement of Tolerance. 469 Federal Register 69(115): 33576-33578. Available Online at: http://www.epa.gov/fedrgstr/EPA-470 PEST/2004/June/Day-16/p12913.htm. Last Accessed: January 17, 2006. 471 472 Weber, J. Undated. Definition of soil organic matter. Available Online at: 473 http://www.humintech.com/001/articles/article\_definition\_of\_soil\_organic\_matter.html. Last Accessed: 474 475 January 11, 2006. 476

|     | Technical Evaluation Report  | Humic Acids  | Crops |  |  |
|-----|--|--|-------|--|--|
| 477 | Witney, G. 1996. Soil versus foliar fertilizer application. Available Online at: |  |       |  |  |
| 478 | http://hort.tfrec.wsu.edu/Orchard  | <u>l/soilvfoli.html</u> . Last Accesses: January 17, 2006. |       |  |  |

478

( )

. [\_\_\_\_\_

## Appendix 8

Additional Research Information:

Oxidized Lignites and Extracts from Oxidized Lignites in Agriculture (Karr)

## Oxidized Lignites and Extracts from Oxidized Lignites in Agriculture

By Michael Karr, Ph.D. ARCPACS Cert. Prof. Soil. Sci.

May, 2001

### **Table of Contents**

| PART I. Effects Of Oxidized Lignites And Derivatives From<br>Humic Substances On Plants And Soils |
|---|
| Introduction3   |
| Effects Of Oxidized Lignites and Derivatives from Humic   |
| Substances on Plant Growth3   |
| Effects Of Oxidized Lignites And Derivatives From Humic   |
| Substances On Nutrient Availability and Uptake6   |
| Plant Uptake of Humic Substances9   |
| Biochemical Effects of Humic Substances on Plants10   |
| Drought Tolerance and Water Use Efficiency11  |
| Humic Substances and Soil Microbial Activity12  |
| Effects of Humic Substances on Soil Physical Properties   |
| The Humin Fraction13  |
| Additional Research Needs13   |
| Conclusions14   |
| PART II. COMMERCIAL USE OF OXIDIZED LIGNITES AND EXTRACTS OF OXIDIZED<br>LIGNITES                 |
| Types and Uses of Oxidized Lignite Products14   |
| Application Rates of Oxidized Lignites and Extracts   |
| Conditions where Benefits from Oxidized Lignite Products are reduced16                            |
| The Fulvic Acid Fraction and Claims about Fulvic Acid Content17                                   |
| Chemical and Heat Treatment of Oxidized Lignites18  |
| REFERENCES19  |

# PART I. EFFECTS OF OXIDIZED LIGNITES AND DERIVATIVES FROM HUMIC SUBSTANCES ON PLANTS AND SOILS.

#### Introduction

Most of the published research on the effects of humic substances on plants has been done in nutrient and sand cultures. Field research or soil pot studies on the response of agricultural crops to applications of oxidized lignite is less abundant. More work has been done using humic acid derivatives from oxidized lignite or peat than by using raw lignites. Because of this, the literature reviewed on the effect of these substances on plants and soils will come from all of these areas.

It is true that humic and fulvic acid fractions extracted from different terrestrial sources (soil, peat, compost, oxidized lignites, other coals, manure, etc.) and vegetation types do show differences in molecular size, chemical structure and functional groups. However, when highly humified extracts are purified and examined, the differences are fewer. For example, Amalfitano (1995) looked for major differences or similarities between the chemical structure of humic acids derived from the light fraction litter of soils with widely varying vegetation types, and found that the spectra from highly humified extracts were similar.

Reports by commercial enterprises on the beneficial effects oxidized lignite are often a series of side-by-side comparisons without statistical analyses, or are performed at a single location or over a single year, and thus have a narrow inference space. In addition, commercial enterprises have a vested interest in demonstrating positive outcomes from their experiments. Because of this, corporate research literature on the effects of oxidized lignites and derivatives on plants and soils will not be reviewed in this paper.

A note on terminology; often the term used in the literature for oxidized lignites is leonardite. Leonardite refers to a particular geologic deposit of oxidized lignite in North Dakota, but has often been misapplied to lignitic deposits found elsewhere. Humate is a common term meaning a source of humic and fulvic acids. The term humic acid, which is the alkali soluble but acid insoluble portion of a source of humic substances, often is applied to alkali extracts of these materials. These alkali extracts include the acid soluble portion (fulvic acid) component. Generally it is to be assumed that the term "humic acid" includes the fulvic acid component unless the author of the research literature has specified otherwise.

#### Effects Of Oxidized Lignites and Derivatives from Humic Substances on Plant Growth

#### Germination:

In the application of humic acid extracts to plants, Smidova (1962) found increased water absorption, respiration and germination rate in wheat, and Ishwaran and Chonker (1971) in soybeans (Glycine max L.). Dixit and Kishore (1967) found an increased germination rate in barley (Hordeum Vulgare L.), corn (Zea mays L.), and wheat (Triticum aesthivum L.). However Piccolo et al. (1993) observed no increase in the germination percentage or rate for either lettuce or tomato seeds treated in Petri dishes with unfractionated humic acids derived from an oxidized lignite. No evidence that humic substances increase the viability of seeds has been reported

#### Root growth - solution and sand culture:

Increases in root mass, length or number of initials were reported on the several crops grown in sand or nutrient solutions to which were added humic or fulvic acids, or extracts from oxidized lignites. Here are some examples:

beans - (Phaseolus vulgoris L.) Schnitzer and Piapst.1967.

corn - (Zea mays) Ivanova ,1965; Alexandrova ,1977;

cucumber - (Cucumis sativus L.) plants by Rauthan and Schnitzer,1981;

grapes - (Reynolds et al. 1995)

millet - (Pennin'tum sp. L.) Alexandrova, 1977;

pepper - (Capsicum annuum L.) Sanchez-Conde and Ortega, 1968);

sugar beet - (Beta vulgaris L.) Sanchez-Conde et al., 1972;

tomato - (Lycopersicon esculentem L·) Sladky ,1959a; Lineham ,1976; Adani et al.,1998;

#### **Root Growth - Soils**

Lee and Bartlett (1976) investigated the response of corn to 8 mg L-1 Na-humate additions to a low organic matter soil, and found increased root proliferation.

Kelting et al., 1998a tested three types of humate (oxidized lignite) derived products on root growth and sapflow of balled and burlapped red maple (Acer rubrum L.) trees. Treatments included oxidized lignite as 1) an extract applied as a soil drench; 2) a liquid formulation to which various purported root growth -promoting additives had been added, also applied as a soil drench; 3) as a dry granular formulation, applied as a topdress. They found that no treated trees had more root length than non-treated controls, but all humate derived treatments increased sap flow.

Kelting et al. (1998b) also tested soil treatments of compost, peat and oxidized lignite on posttransplant growth of red maple (Acer rubrum L.) and Washington hawthorn (Crataegus phaenopyrum Hara) trees. Granular oxidized lignites increased total root length in Washington hawthorn but not in red maple.

#### Foliar applications of humic substances on root growth:

Sladky (1959b) applied humic materials as a foliar spray on begonia (*Begonia semperflorens* L.) plants grown in nutrient solutions and found increased root growth. Similar observations were obtained by Sladky (1965) with sugar beets grown in distilled water.

#### Shoot growth:

As is the case for root growth studies, most of the early publications on shoot growth enhancement are limited to young plants grown in pots or in nutrient solutions.

Piccolo et al. (1993) Treated lettuce and tomato seeds in Petri dishes with unfractionated humic acids (UHA) derived from an oxidized lignite at strengths ranging from 40 to 5000 mg/L. The fresh weight of total seedlings and per seedling increased in treatments with UHA and with increasing concentrations for both lettuce and tomato plants without showing signs of growth inhibition up to 5000 mg/L. The authors attributed this to cell elongation and more efficient water uptake.

Adani et al. (1998) Studied the effects of humic acids extracted from peat (CP-A) and from leonardite (CP-B) on the growth and mineral nutrition of tomato plants (Lycopersicon esculentum L.) in hydroponics culture were tested at concentrations of 20 and 50 mg/L. Both the humic acids tested stimulated plant growth. The peat derived humic acids stimulated only root growth, while the leonardite derived humic acids showed a positive effect on both shoots and roots, especially at 50 mg/L.

Lee and Bartlett (1976) studied stimulation of corn seedling growth in low organic matter soil with 8 mg/Kg Na-humate and found increases in seedling growth of 30 to 50%.

Tan and Tantiwiramanond (1983) applied humic and fulvic acids to sand cultures of soybeans (Glycine max L.), peanuts (Arachis hypogea L.) and clover (Trifolium sp.). Shoot, root, and nodule dry weights increased in response to treatments up to 400 to 800 mg/kg soil.

Reynolds et al. (1995) planted greenhouse-grown 'Chardonnay' vines (Vitis vinifera L.) in a sand medium to which was added one of five levels of granular Gro-Mate (GM), a commercial humate. Shoot length responded to increasing level of granular humates. Fresh and dry weights of leaves, shoots, and roots, as well as leaf count and area, exhibited increasing linear or quadratic trends in response to increased level of granular GM.

Reynolds et al. (1995) found that very high granular applications of oxidized lignite may result in leaf necrosis and retarded growth on grapes in sand culture.

Kelting et al. 1998b, tested several types of organic materials on post-transplant growth of red maple (Acer rubrum L) and Washington hawthorn (Crataegus phaenopyrum Hara) trees. Soil treatments applied at planting included additions of compost, peat and oxidized lignite. They found that all soil treatments did increase top dry mass for Washington hawthorn, with the oxidized lignite treated trees showing the greatest increase. No treatments significantly increased dry mass for red maple.

#### Foliar applications of humic substances and shoot growth:

Sladky and Tichy (1959) sprayed tomato plants with a solution of 300 mg/ L humic acid, and found that both fresh and dry weight of shoots was increased. They reported that higher application rates inhibited growth and deformed leaves.

Sugar beets (*Beta vulgaris* L.) also responded with increased shoot growth when foliar sprayed with humic acids A. (Sladky, 1965).

(Sladky, 1959b) sprayed begonia plants with either humic or fulvic acids and found increased shoot growth. The investigator also indicated that fulvic acid was slightly more effective than humic acid.

In a review of published reports, Chen & Aviad (1990) found that fulvic and humic acids may stimulate shoot growth of various plants when applied either as foliar spray at concentrations of 50 to 300 mg/L, or when applied in nutrient solutions at concentrations of 25 to 300 mg/ L. This stimulatory effect often extended to roots, regardless of the mode of application.

#### Crop yields in soil pots and field trials

Martin and Senn (1967) found that the use of humic acid derivatives (HAD) added to tomatoes grown in 3-gallon pots increased yields, especially during the latter stages of growth. Applications of HAD resulted in a greater number of fruits of comparable size to the check for the first 5 harvests. Quality and grade of fruit was superior to controls, with HAD treatments resulting in more than 200 percent increase in yield of number 1 tomatoes.

Brownell et al. (1987) conducted field trials on tomatoes, cotton and grapes after application of two commercially available extracts from leonardite (oxidized lignite). One extract was used as an early season soil treatment, while the other was used as a foliar spray. Results from both treatments on tomatoes produced average yield increases of 10% over controls; on cotton the average yield increase was 11%. Unreplicated, large field trials on various cultivars of grapes produced yield increases ranging from 3 to 70% over untreated controls.

Wang et al. (1995) added humic acids to an alkaline soil with P fertilizer and examined wheat yields in field trials. Humic acid treated plots increased both P uptake and yields by 25%.

Crowford et al. 1968 conducted a three-year test to determine if humic acids could effectively influence sprout production and yield of sweet potatoes. Treatments included either soaking the seed potatoes in a 10% humic acid derivatives (HAD) solution or by incorporating HAD into the soil beds at the rate of 2 grams/lb. soil. The results were averaged over three years. Soil treatment increased sprout production from 69 to 231, and potatoes showed a 10 - 20% yield increase over controls. For the 10% HAD potato seed treatment, three year averaged yields increased 30 - 40% over controls. All humic treatments resulted in a significantly higher percentage of number one grade potatoes.

Duval (1998) Applied varying rates of leonardite up to 400 lbs./acre on turnip (*Brassica rapa* L) and mustard greens (*Brassica hirta* L.) with 3 plantings over a one-year period, and found no differences in the plant growth parameters studied. However, they did report that excessive rain over a 6-week period (13.5 inches, and 6.5 inches in one day on a fine sandy loam soil) eroded the soil and caused a nitrogen deficiency in the crop. In addition, they could not find detectable quantities of humic acid in the soils after the experiment was concluded. They also reported an infestation of yellow margined black turnip beetles (*Microtheca ochrolma* Stal), which destroyed the stand of plants 4 weeks after the 2<sup>nd</sup> planting, and then was replanted after applying an additional 120 lbs. ammonium sulfate per acre.

#### Effects Of Oxidized Lignites And Derivatives From Humic Substances On Nutrient Availability and Uptake

#### Uptake of macroelements in solution or sand culture:

Humic substances have been demonstrated to increase the uptake of plant nutrients. Many studies report increased growth, together with increasing uptake of plant nutrients. Studies that isolate the growth hormone-like response from growth resulting from increased uptake of limiting plant nutrients will be presented in the section on the biochemical effects of humic substances.

Bean (Phaseolus vulgaris) and rough fescue (Festuca scabrella Torr.). Dormaar (1975) added humic acids at 1 to 50 mg L-1 to plants grown in nutrient solutions. Nitrogen uptake increased at 20 to 50 mg L-I, but uptake of P, K, Na, Ca, and Mg was not significantly affected.

#### Corn (Zea mays L.)

Lobartini et al. (1998) investigated the effect of humic (HA) and fulvic acid (FA) on the dissolution of aluminum phosphate (AIPO4) and iron phosphate (FePO4), and assessed their availability to plants. The results indicated that the amount of P released by HA or FA increased with time, with free orthophosphates present with small amounts of P-humic acid complexes. Humic acid was more effective than fulvic acid in dissolving the metal phosphates. The plant-availability of phosphate dissolution products was confirmed by growing corn plants in hydroponic solutions with AIPO4 or FePO4 as the source of P, and HA or FA at pH 5.0. Corn plants exhibited better P uptake and growth performance when HA or FA is present.

#### Cucumber (*Cucumis sativus*)

Rauthan and Schnitzer (1981) added up to 2000 mg L-I soil-derived fulvic acid to nutrient solutions. The uptake of N, P, K, Ca, and Mg increased to the shoots. Maximum uptake and maximum growth occurred at concentrations of 100 to 300 mg/ L FA.

#### Grapes

Reynolds et al. 1995, planted greenhouse-grown 'Chardonnay' vines (Vitis vinifera L.) in a sand medium to which was added one of five levels of granular commercial humate. They found that the granular humate increased petiole Fe and lamina P, K, and Fe.

#### Ryegrass (Lolium perenne L.)

Gaur (1964) found enhanced uptake of N, P, and K; and reduced uptake of Ca in ryegrass grown in pots in a soil with added humic acid extracted from compost.

#### Pepper

Sanchez-Conde and Ortega (1968) found increased uptake of N, P, and Mg, and reduced uptake of K, Ca, and Na on pepper plants irrigated with solutions containing 8 -100mg/ L humic acid.

#### Tomatoes (Lycopersicon esculentum L.)

Adani et al. (1998) studied the effects of humic acids extracted from peat (CP-A) and from leonardite (CP-B) on the growth and mineral nutrition of tomato in hydroponics culture. Both extract treatments showed increases in the uptake of N, P, and Fe.

#### Wheat (Triticum aestivium).

Vaughan et al. (1978) studied radioactively labeled <sup>32</sup>P uptake on excised roots and cell cultures of winter wheat. Concentrations of 5 to 50 mg/L humic acid enhanced <sup>32</sup>P uptake, but 500 mg/L reduced uptake.

More research has been done recently regarding the stimulation of nitrate uptake by humic substances:

Piccolo (1992) obtained humic extracts with distinct physical-chemical characteristics, by using various soil extractants and from different sources, in order to study their effect on nitrate uptake by barley seedlings. Results showed that the most effective humic fraction on plant nitrate

uptake had the highest acidic functionality and the smallest molecular size, whereas the aliphatic and aromatic content of extracts did not appear to play a role.

The uptake of major anionic macronutrients like nitrate is substrate inducible and requires energy. Santi et. al., 1995 found that the activity and amount of plasma membrane -ATPase was increased in maize roots induced for nitrate uptake.

Pinton and Cesco (1999) studied the effect of the water extractable humic substances fraction (WEHS) on nitrate uptake of maize roots. They found significant increases in both nitrate uptake and plasma membrane H<sup>+</sup>ATPase activity. Results supported the idea that the plasma membrane proton pump might be one of the primary targets of the action of humic substances on plant nutrient acquisition.

Nardi et. al. (2000) tested a low molecular weight humic fraction (LMW-HA) for its biological activity in maize seedlings. Results showed that LMW-HA increased nitrate uptake. The authors hypothesized that LMW-HA stimulated nitrate uptake by decreasing the pH at the surface of roots, thus facilitating the H+/NO3- symport. The nitrogen regulatory properties of LMW-HA appeared to depend on the combination of low molecular size, gibberellin-like activity and to the content of phenolic and carboxyl carbon.

#### Nutrient availability in soils:

Humic substances may influence the rate of release of nutrients from soil minerals. Tan (1978) has demonstrated that both humic and fulvic acids can enhance the release of fixed K from illite or montmorillonite.

Wang et al. (1995) studied the effect of humic acids on transformation of phosphorus fertilizer in an alkaline soil. Soil P was fractionated following 4 and 15 days incubation after humic acids were applied with phosphorus fertilizer to the soil. The availability of phosphate in the soil and in plants was determined at heading stage and at maturity in a pot experiment, and wheat yield was examined in a field trial. Addition of humic acids to soil with P fertilizer significantly increased the amount of water-soluble phosphate, strongly retarded the formation of occluded phosphate, and increased P uptake by 25%.

#### Nutrient uptake from soils:

Jelenic et al. (1966) added <sup>32</sup>P-labeled superphosphate plus Na-humic acids derivatives (HAD) extracted from lignite to two soils at rates of 2 to 12 mg HAD/kg soil. They found increased uptake of both soil-P and superphosphate-P by corn, with a maximum uptake observed at 3 to 8 mg HAD/kg of soil.

Wang et al. (1995) added humic acids to an alkaline soil with P fertilizer with wheat grown in field trials, and observed increased P uptake and yield; both by 25%.

Xudan, (1986) in pot studies and field trials with wheat, found that spraying the leaves with fulvic acid resulted in greater uptake of <sup>32</sup>P by the roots.

#### Uptake of microelements:

Improved availability of micronutrients by solubilization from their inorganic forms in soils or in nutrient solutions plays an important role in the promotion of plant growth in soils by humic substances.

Studies by Varadachari et al. (1997) on the complexation of humic substances with oxides of iron and aluminum indicated two major modes of HA bonding - cation bridges forming oxide-M-HA links and direct bonding of HA to coordination centers at the oxide surface.

Dekock (1955) found that lignite-derived humic substances increased the solubility of Fe in solution, and increased Fe uptake and translocation from roots to shoots. This effect was observed even at high phosphate concentrations.

Lee and Bartlett (1976) found that 5 mg L-I Na-humate in a nutrient solution increased the Fe concentration in roots and shoots of corn.

Rauthan and Schnitzer, 1981 found that fulvic acid increased the uptake of Fe, Zn, Cu, and Mn by cucumber (Cucumis sativus) plants grown in Hoagland's solution.

Aso and Sakai (1963) found that the chlorosis exhibited by barley (Hordeum vulgare L.) and rice (Oryza sativa L.) was alleviated by additions of Fe (III)-humic substance complexes, while unferrated humic substances alone were ineffective.

Linehan and Shepherd (1979) observed that the addition of fulvic acid at concentrations up to 25 mg/L to nutrient solutions increased Fe uptake to shoots of wheat seedlings (Triticum aestivium).

Bar-Tal et al. (1988) demonstrated that solutions with fulvic acid added would maintain a zinc level of  $10^{-3.5}$  mM in the presence of Ca-montmorillonite at pH 7.5, whereas solution Zn levels decreased to  $10^{-5.5}$  mM without fulvic acid.

#### Plant Uptake of Humic Substances

If humic substances have direct effects on plant growth, then they must be absorbed and translocated by plants. Studies commonly use 14C-labeled humic substances to trace their uptake into and movement through plants.

Fuhr and Sauerbeck (1967b) reported that much of the absorbed radioactivity from 14C-humic acid was incorporated into the epidermis of sunflower (Helianthus annus L.), radish (Raphanus safivus L.) and carrot (Daunts carora L.) roots. In addition, the radioactivity that was observed in the stele originated from low molecular weight components of the humic materials.

Vaughan and Linehan (1976) found that 14C-labeled humic acid was taken up by wheat roots, and a small percentage (5%) was transported to the shoots.

Fuhr & Sauerbeck, (1967a) showed that fulvic acid is transported to the shoot to a greater extent than is humic acid.

Vaughan and McDonald (1976) also suggested that only low molecular weight fractions of the humic acids are biologically active. They examined the uptake of <sup>14</sup>C-humic acid by intracellular components of beet roots. The greatest amount of radioactivity was associated with cell walls and smaller levels with mitochondria and ribosomes.

Additional clarification of this issue resulted from an investigation on excised pea (Pisum safivum L.) roots (Vaughan and Ord 1981). Results showed greater uptake of the low molecular weight substances. They found that both low molecular weight humic acid and fulvic acid fractions are taken up both actively and passively, but humic substances with molecular weights above 50,000 daltons are absorbed up only passively.

#### **Biochemical Effects of Humic Substances on Plants**

#### Molecular size and activity:

Piccolo (1992) obtained humic extracts with distinct physical-chemical characteristics, by using various soil extractants and from different sources, in order to study their effect on growth regulation in watercress and lettuce. Results showed that the most effective humic fraction on hormone-like activity had the highest acidic functionality and the smallest molecular size, whereas the aliphatic and aromatic content of extracts did not appear to play a role. Fulvic acids had a smaller molecular size, and tended to have higher acidic functionality than humic acids.

#### Membrane permeability:

Many investigators have proposed that these humic substances affect membrane permeability, increasing permeability to some ions and decreasing it to others. This could be due to the surface activity of humic substances on cell membranes.

Pinton and Cesco, (1999) studied the effect of the water extractable humic substances fraction (WEHS) on plasma membrane H<sup>+</sup>ATPase activity of maize roots, and found significant increases in plasma membrane H<sup>+</sup>ATPase activity. Results supported the idea that the plasma membrane proton pump might be one of the primary targets of the action of humic substances on plant nutrient acquisition

#### Respiration rate:

Sladky (1959a) grew tomato plants in nutrient solutions containing either humic acid (50 mg/L), or fulvic acid (50 mg/L)(Sladky 1959a). Oxygen consumption increased by 23% in HA treated plants, and by 34% in FA treated plants, compared to control plants.

Foliar applications of solutions of humic materials may also increase respiration rates. When leaves of begonia were sprayed with humic acid solution, a large increase in oxygen uptake was observed (Sladky, 1959b; Sladky & Tichy, 1959).

#### Chlorophyll density:

Sladky, (1959a) showed an increase in chlorophyll contents (HA =+63%, FA = +69%) resulting from applications of humic substances in nutrient solutions to tomatoes. Humic acid treatment increased chlorophyll density by 63%, and fulvic acid increased chlorophyll by 69%.

Xudan (1986) also found that spraying wheat with fulvic acid in pot experiments and field trials resulted in a higher level of chlorophyll in the leaves

Hormonal effects:

Mato et al. (1971, 1972a, 1972b) have shown that humic acid and fulvic acid fractions of humic substances inhibit indoleacetic acid (IAA)-oxidase. Although unfractionated humic acid was more effective than humic or fulvic acid fractions at suppressing the destruction of the IAA plant hormone, the smallest molecular fraction (mol. Wt. < 700) showed the greatest inhibition of IAA-oxidase.

Nardi et al. (2000) tested a low molecular weight humic fraction (LMW-HA) for its biological activity in maize seedlings. Results showed that LMW-HA strongly inhibited K+ stimulated ATPase of maize microsomes and H+ extrusion in a manner similar to gibberellic acid (GA). Studies of changes in messenger RNA after the humic treatment was performed and an analysis of synthesized polypeptides demonstrated a positive post-transcriptional effect of HA on protein synthesis. The gibberellin-like activity of LMW-HA appeared to depend on the combination of low molecular size, and to the content of phenolic and carboxyl carbon.

Effects on enzyme activity:

Humic substances have been shown to inhibit the activity of the several other enzymes. They include:

carboxypeptidase A, (Ladd & Butler, 1971) choline esterase (DeAlmeida et al., 1980) chymotrypsin A, (Ladd & Butler, 1971) invertase (Malcolm & Vaughan, 1979b), peroxidase (Vaughan & Malcolm, 1979). phosphatase (Malcolm & Vaughan, 1979a,c), pronase, (Ladd & Butler, 1971) trypsin (Ladd & Butler, 1971)

The following enzymes were stimulated by the presence of humic substances:

H<sup>+</sup> stimulated ATPase (Pinton and Cesco,1999) K<sup>+</sup> stimulated ATPase (Nardi et al., 2000) ficin (Ladd and Butler, 1971) papin (Ladd and Butler, 1971)

#### **Drought Tolerance and Water Use Efficiency**

It has been widely claimed by commercial vendors of oxidized lignites that these materials increase drought tolerance or decrease water consumption. In a landmark study, Xudan (1986) investigated the effects of foliar application of fulvic acid on water use and yield of wheat in pot and field experiments. When subjected to a 9-day drying cycle, the stomatal conductance of control plants fell from 0.85 cm S<sup>-1</sup> to nearly zero at the end of the cycle. Plants sprayed with fulvic acid at the start of the drying cycle maintained stomatal conductance of 0.30 cm S<sup>-1</sup> for the entire interval. Fulvic acid applied to well-watered plants in pots also rapidly reduced stomatal conductance from 0.80 to a constant 0.25 cm S<sup>-1</sup>.

When wheat plants were subjected to drought stress at head development stage, grain yield by control plants was depressed by 30% compared to the irrigated control. However, fulvic acid treated plants suffered only a 3% yield loss compared to the irrigated control.

Xudan (1986) also conducted field trials on wheat in north China. Fulvic acid was sprayed on plants just before head development, and allowed to grow to maturity over time when hot, dry

winds are prevalent. He found that grain yield was increased by 7 to 18% over the untreated controls.

Piccolo et al. (1993) Treated lettuce and tomato seeds in Petri dishes with unfractionated humic acids derived from an oxidized lignite at strengths ranging from 40 to 5000 mg/L. They attributed the increase in fresh weight of the seedlings to cell elongation and more efficient water uptake.

It is clear that more research is needed to more firmly establish the effects of humic substances on water stress and water use efficiency.

#### Humic Substances and Soil Microbial Activity

Bkardwaj and Gaur (1972) found that humic acid as sodium humate and fulvic acid had a marked growth stimulating effect on Rhizobium trifolu. The maximum effect was at 500 mg/L. Humus extract dialyzed for fulvic acid exerted appreciable growth stimulating influence (over 200 percent greater growth rate than check) while undialysed sodium humate was less effective (52 percent over check). The growth promoting effect of farmyard manure containing an equivalent amount of humic acid was less than half as effective as that of sodium humate.

Vallini et al. (1997) investigated the effect of humic acids on activity and growth of Nitrosomonas europaea and Nitrobacter agilis in vitro under axenic conditions. Humates from compoststabilized vegetable waste or leonardite were added to the chemolithotrophic-culturing medium. They found that both types of humic acids increased either NH4+ or NO2-oxidation and cell growth of nitrifying bacteria. By combining these results with data from a comparative growth evaluation of N. agilis, evidence was obtained that nitrifiers cannot use humic acids as an alternative carbon and energy source. They attributed the stimulating effect of humic acids on these bacteria to an increase in microbial membrane permeability favoring a better utilization of nutrients.

#### Effects of Humic Substances on Soil Physical Properties

#### Soil structure:

Piccolo and Mbagwu (1989) found a significant increase in water-stable aggregates in a sandy loam and a strong clay soil after treatment with humic substances derived from coal. If so, increased water infiltration and percolation, reduced runoff and resistance to erosion, and increased aeration are other beneficial effects that are indirectly supported by humic substances.

Piccolo et al. 1997 added humic substances from oxidized coal to two soils with severe structural problems and assessed their effect in reducing runoff erosion with simulated rainfall. They observed a reduction of soil loss of 36% on one soil treated with 100 kg/ha; and the same approximate magnitude of reduction on the other soil treated with 200 kg/ha. They found that the improvement in the water retention capacity more than aggregate stability accounted for the reduced runoff erosion. This delayed the onset of runoff and favored water entry through the stable interaggregate pore spaces within the soil beds. Percent moisture retained at field capacity increased from 26.3% to 29.3% at the 0.05g/kg rate for the Principina silt loam (Orthic Xerofluvent), and from 26.9% to 33.0% at the same rate for the Bovolone loam (Udic Ustochrept).

#### Available water:

Humic acids are heterogeneous substances, which include in the same macromolecule, hydrophilic acidic functional groups (made up of the carboxylic and phenolic groups) and the hydrophobic groups (made up of the aliphatic and aromatic carbon groups) (Stevenson,1994). The humic acid hydrophilic groups (carboxyl and phenols) attract hydration water thus increasing the water retention capacity in soils.

#### **Oxidized Lignite and Odor Control of Manure**

Georgacakis, D. (1996) . found that ground lignite (humate), due to its excellent odor- and moisture-absorbing capacities, allowed for the successful incorporation of the wet and malodorous swine manure into the compost process. More work on the use of oxidized lignite for odor control and in the composting process is needed.

#### **The Humin Fraction**

Researchers have commonly overlooked the role of humins in soils. Humin is the alkali (and acid) insoluble portion of humic substances. The "humin fraction" includes humin, plus mineral impurities and other insoluble compounds. Humin benefits soils by holding water and by sorbing cations, polar and nonpolar compounds.

Kohl et al. (1998) studied the binding of 3 polycyclic aromatic hydrocarbons (PAHs) and 2 polychlorinated biphenyl (PCBs) contaminants to the humin fraction of organic matter from 3 different soil types. In all soils and contaminants, the humin fraction contained more than 50% of the bound residue and typically between 70 and 80%. Unfortunately, chemically extracted liquids from oxidized lignites leave the humin fraction behind.

Studies have shown that organic P compounds of from several sources, including manure, become bound to high molecular weight organic colloids (humin). The organic P associated with humic substances may exist, in part, as complexes with simple phosphate esters (e.g. inositol phosphates) Brannon and Sommers (1985a) have reported

#### **Additional Research Needs**

Additional research is needed to determine the effects of oxidized lignite and derived products in the following areas:

More field research conducted on soils with varying amounts of organic matter

Field research on broadcast applications of oxidized lignite, or banding of oxidized lignite with fertilizer, affects nutrient availability and uptake. This type of research needs to be conducted on soils that vary in pH, presence of free lime, available P and metal micronutrients.

Field research on nitrogen use efficiency using oxidized lignite.

Water use efficiency and abiotic stress tolerance.

Effects on microbial respiration and mineralization of organic matter.

Development of a reliable inexpensive fulvic acid test

Effects on ruminants, ruminant microbial diversity and numbers, efficiency of conversion of cellulose, disease incidence and severity. Although research in this area has been done by corporations, the results are almost always proprietary, and thus not available to the public. Research from public institutions is practically non-existent.

#### CONCLUSIONS

Solution and sand culture studies have demonstrated that soluble derivatives of humic substances will increase length and fresh and/or dry weights of shoots and roots, number of lateral roots, root initiation, seedling growth after germination, nutrient availability and nutrient uptake. These substances also affect a wide range of enzymatic processes.

Field trials and soil pot studies have also demonstrated these effects using oxidized lignite or derivatives of humic substances. The difference is that less of this type of research has been performed.

Additions of oxidized lignite to soils with low humic content may help to increase aggregate stability and available water capacity.

Recent research data has increased our understanding of the role of humic substances play in nitrate uptake by plants.

A limited amount of research exists on specific effects of oxidized lignites or derivatives of humic substances on plant drought tolerance, water use efficiency, and enhancement of soil microbial activity.

#### PART II. COMMERCIAL USE OF OXIDIZED LIGNITES AND EXTRACTS OF OXIDIZED LIGNITES

#### **Uses of Oxidized Lignite Products**

Vendors of oxidized lignite products commonly advise the following uses. Of course, the amount of research that supports each recommendation varies widely.

Soil treatment - broadcast for broad-spectrum benefits to soils and plants

Soil treatment - banded with micronutrient and phosphate fertilizers to increase availability

Foliar treatment for growth enhancement and stress tolerance

Applied to organic materials to increase the rate of the composting products.

Applied to manure for odor control

Extracts added to liquid fertilizers to help keep phosphates and micronutrient metals soluble.

#### Types of Oxidized Lignite Products Available

Raw, ground oxidized lignites. Cheapest cost of production per kilogram of humic substances

<u>Liquid extracts of oxidized lignites</u>. Generally base-treated, with a final concentration of 6 - 12% humic plus fulvic acids. More expensive to produce than oxidized lignites, due to the extraction process, low analysis, high transportation and storage costs per kilogram of humic substances.

<u>Dry water-soluble base extracts of oxidized lignites</u>. Most expensive to produce per kilogram humic substances. It is the dried down residue of liquid extracts of oxidized lignites. Drying costs are very high.

<u>Base treated raw oxidized lignites</u>. Addition of a base, generally KOH, sprayed on the oxidized lignite and dried. About the same cost to produce as liquid extracts per kilogram of humic substances

<u>Raw oxidized lignite suspensions</u>. Recent patent-pending process of micronizing and suspending oxidized lignite in water, without chemical alteration. Up to 37% oxidized lignite yielding 24% humic plus fulvic acids. Includes the humin component. Cost to produce per kilogram humic substances greater than raw oxidized lignite.

<u>Fulvic acids</u> - extracted from highly oxidized lignites and peats by various methods. Actually the fulvic acid fraction is what is marketed. Very expensive per kilogram of humic acid fraction extracted.

#### Application Rates of Oxidized Lignites and Extracts

Based on the ranges in the concentrations of humic substances used by researchers in studies reviewed by Chen and Aviad (1990), they calculated the following rates for field applications. These rates are based on a midpoint average from which benefits were reported.

Assumptions for soil treatment are: 1) the plow layer weight was 2500 Mg/ha, 2) water content at field capacity is 30% by weight (quite high) and 3) the increase required in humic substances to be most effective is 100 mg/L. (range is 25 to 300 mg/L).

Soil Application: = 75 kg. humic substances per hectare (66 lbs./ac.). (range from about 20 to 225 kg/ha).

Assumptions for foliar treatment is a spray volume of 2000 liters per hectare and a midpoint concentration of 250 mg/kg soluble HA + FA. (range 50 to 300 mg/L).

Foliar Spray = 500 grams of HA + FA per 2000 liters. (range from about 100 to 600 g/2000 L).

#### General comments on application rates:

For soil applications at the rate of 75 kg/ha as suggested by Chen and Aviad (1990), using an oxidized lignite with 70% humic + fulvic acid content, the amount required would be about 110 kg/ha (97 lbs./ac.). The range would be from about 30 to 350 kg/ha. Vendors generally recommend from 40 to 750 kg/ha. Three assumptions here are that the entire humic and fulvic acid fraction will dissolve and remain in the soil solution without reacting with the soil mineral phase, and without leaching in the interval between applications. This rarely, if ever, is the case. Because of this, agronomic benefits probably decline below application rates of 100 kg/ha (88 lbs./ac.).

For foliar applications at the rate of 500g HA and FA per hectare, as suggested by Chen and Aviad (1990), using a 6% HA + FA extract of oxidized lignite, the amount required would be about 8.5 liters per hectare (about 1 gallon per acre). For 12% HA + FA liquid extracts, the rate would be half that. Vendors have suggested rates ranging from 1/2 gallon to 3 gallons (4 to 26 liters per hectare).

#### Usage on soils with high humus levels:

Soils have widely varying ranges of soluble humic substances in the organic fraction. In fertile soils with high total humus levels, soluble organic matter may reach levels up to 400 mg/L (Chen & Schnitzer, 1978), while in soils of arid regions it may not exceed 20 mg/L (Chen & Katan, 1980). It would seem that the beneficial effects due to application of humic substances become diminished as native humus content increases. Because of this, the author does not recommend using broadcast applications of oxidized lignites on soils with more than 5% total humus by weight. On these soils, the banding of oxidized lignites or foliar applications of oxidized lignite extracts should be tested for efficacy by the grower on small areas of his crop, before general use is adopted. More research on the effects of varying soil humus levels on the performance of oxidized lignites and extracts is clearly needed.

On calcareous soils of moderately high humus levels, where solubility of P or metal micronutrients is limiting plant growth, banding acidic oxidized lignite (pH 3.4 - 4.0) with fertilizer sources of these nutrients may result in increased availability to plants. Generally, 5 - 15 lbs. oxidized lignite per acre is applied in a band with fertilizer. Liquid humate extracts can also be banded with liquid fertilizers at the rate of 1 - 3 gallons per acre. Again, this approach should be tested in small areas by the grower, and again, more research data on the benefits of this strategy is needed.

#### Excessively low recommendations by vendors.

Occasionally vendors of humate products recommend rates so low that little practical benefit is realized. This happens more often with the more expensive oxidized lignite extracts. Vendors do this for two reasons:

- 1. To support their claim that their product works better than their competitors and
- 2. To enjoy huge markups on their product, but still keep the product affordably priced at the usage rates recommended.

The damage done is twofold - the customer does not get the purported benefits he/she purchased and, researchers report little benefits from using humates at the rates recommended by the producer. For example:

In a study of humate-based biostimulants on Turkish hazelnut seedlings, Kelting (1997) found no significant differences in root or top growth compared to untreated controls. All treatments were applied at the manufacturers' recommended rates. In the dry-water soluble oxidized lignite treatment only 2.5 mg was applied to each 3.8 liter pot; a rate recommended by the producer. This amount would provide a HA concentration of just over 1ppm at 50% moisture by volume - if no leaching occurred over the course of the experiment. This is in comparison to the 25 - 300 ppm HA range where most investigators found significant growth effects Chen and Aviad (1990).

One vendor of a dry-water soluble product recommends a rate of 2 oz per acre, and sells the product to retailers at \$16,000 per ton!

#### Conditions in which benefits from oxidized lignite products are reduced

High organic matter soils - especially over 5% humus.

Optimum fertility and growing conditions.

Long term compost or manure additions

Compost additions over 5 tons/acre in a given year

Severe limiting factors. Examples include severe deficiencies of N, extremely high or low pH, excessive wetness, excessive cold, severe compaction, heavy foliar disease pressure, etc.

Growth enhancement is decreased if oxidized lignite products are applied after other biostimulants like kelp extracts, yucca extracts, growth hormones, etc.

#### The Fulvic Acid Fraction and Claims about Fulvic Acid Content

Oxidized lignites vary not only in the total amount of humic substances, they also vary in the relative proportion split between humic acids and fulvic acids. New Mexico oxidized lignites tend to be higher in the fulvic acid fraction than North Dakota lignites, for example. Reports on specific fulvic acid contents result from the confusion between the "fulvic acid fraction" and the "fulvic acid content". This relates to the fact that there is not currently a reliable and inexpensive fulvic acid test. The two chief ways in which oxidized lignite products are analyzed for humic acid are:

1. <u>Colorimetric</u> tests of a 0.5N NaOH extract of a humate source. The numbers are usually reported as "humic acid content", when it is more precisely the spectrally active humic acid plus fulvic acid content. Included but not reported are other water or base-soluble constituents that may show some absorbance at the 450nm analytical wavelength. These other soluble constituents make up a very small proportion of the total absorbance of the extract, and may vary among oxidized lignite products. Still, it is a good test for routine quantitative analysis of humic substances, and includes both humic and fulvic acids. It is also relatively inexpensive and easy for labs to do

2. A <u>gravimetric</u> test of humic acids precipitated from the 0.5N NaOH extract of a humic acid source, using 6N HCl to bring the pH of the extract at or below 2.0. The humic acid fraction then precipitates out, then washed, dried and weighed. What is being measured is chiefly the humic acids content. This value is always lower than the colorimetric test for humic plus fulvic acids, because what is not included or measured are the fulvic acids and other components still soluble at this acidic pH.

Since there is not currently a reliable fulvic acid test, companies report the "fulvic acid content" as being the difference between the colorimetric test and the gravimetric tests, attributing that difference entirely to fulvic acids. It is more accurate to represent that number as the "fulvic acid fraction", which contains fulvic acid plus other soluble organic components that usually are present. At this time most producers and customers do not know the difference between "fulvic acid" and "fulvic acid fraction".

Although we have recognized procedures, endorsed by the International Humic Substances Society, for extracting and purifying fulvic acid from soils, peats, oxidized lignites and aquatic sources, they are very expensive, time consuming and, most importantly, <u>give variable yields</u>.

Until a good fulvic acid test is developed, there are several ways for states to resolve this matter. Listed below are three options often considered by states:

- 1. Use both the colorimetric test for humic plus fulvic acids, and the gravimetric test for humic acids, and allow producers to report the difference as a "fulvic acid fraction".
- 2. Use only the colorimetric test for humic plus fulvic acids; and do not allow producers to report a "fulvic acid fraction"
- 3. Use only the gravimetric test for humic acids, do not include the fulvic acid fraction, and do not allow producers to report a "fulvic acid fraction"

The third option, which ignores the fulvic acid fraction entirely, as California currently does by only allowing the base extract - precipitation test for humic acids, is problematic for the following reasons:

1. Fulvic acids are the most biologically active fraction of humic substances, and have the highest amount of reactive functional groups.

2. Materials with a low ratio of "fulvic acid fraction to humic acid fraction" will enjoy a higher reported numerical humic acid content compared to materials with a higher ratio of "fulvic acid fraction to humic acid fraction". For example:

One oxidized lignite material with a relatively high proportion of low molecular weight humic substances, and may test at 70% HA + FA by the colorimetric test, and at 45% HA with the precipitation test, leaving a 25% fulvic acid fraction. If limited to just reporting humic acids by precipitation, they can only report 45% humic acids in their material.

Another oxidized lignite material with a relatively low proportion of low molecular weight humic substances, may test the same as the first material in the colorimetric test (70% HA + FA), but their precipitation test may give a result of 60% HA, leaving only a10% fulvic acid fraction. In this case they can report 60% humic acids in their material.

In other words, a producer of oxidized lignite with a relatively high proportion of biologically active small molecules is penalized relative to the producer of a material with a lower proportion of biologically active humic substances!

3. At present "fulvic acid" is a magic buzzword in agriculture, and is often promoted as having greatly enhanced effects on soils, microbes and plants; compared to other humic substances. Because of this, vendors can afford to go through the relatively expensive process of extracting the humic acid fraction for sale to the public at large markups.

The public needs to be educated about the proven benefits of fulvic acid and fulvic acid fraction products. Ignoring the existence of fulvic acid by restricting the reporting of humic substances to humic acid only, via the base extract-precipitation test, is a disservice to the public.

#### **Chemical and Heat Treatment of Oxidized Lignites**

Leonardite has been treated in a variety of ways to increase yield of humic and fulvic acids, their extracts, or the relative abundance of functional groups. Chemical treatment with oxidants, organic acids, and prolonged aeration of poorly oxidized coals, has been used with varying degrees of success. All add to the cost of production.

Heat treatment during base extraction for liquids has also been used frequently. Cegarra et al. (1994) used solutions of potassium hydroxide (0.1M and 0.25M) to extract humic substances from peats at temperatures ranging up to 80 degrees C. Although yields increased with temperature, the HA released from the extraction exhibited less oxidized molecules, a lower content of functional groups and larger molecular sizes than extracts performed at room temperature.

#### Oxidized Lignites and Extracts from Oxidized Lignites as Energy Sources

Humates have been touted as an energy source for microbes by vendors. Aside from small concentrations of readily oxidizable carbohydrates and organic acids that may be present via illuviation from overburden materials, oxidized lignites are, by definition, extremely resistant to further oxidation. The mean residence time for highly humified substances in temperate soils ranges from 250 to 1000 years or longer (Stevenson, 1994). Oxidized lignites and extracts from oxidized lignites should not be promoted as energy sources for soil microbes.

#### REFERENCES

- Adani, F., Genevini, P., Zaccheo, P., Zocchi, G. 1998. The effect of commercial humic acid on tomato plant growth and mineral nutrition. J. plant nutr. 1998. v. 21 (3) p. 561-575.
- Alexandrova, I.V. 1977. Soil organic matter and the nitrogen nutrition of plants. Sov. Soil Sci. (Engl. Transl.) 9:293-301.
- Amalfitano, C. Quezada, R.A., Wilson, M.A. and J.V. Hanna.1995. Chemical composition of humic acids: a comparison with precursor 'light fraction' litter from different vegetations using spectroscopic techniques. Soil science. June 1995. v. 159 (6) Pages: p. 391-401
- Aso, S., and I. Sakai. 1963. Studies on the physiological effects of humic acid. 1 Uptake of humic acid by crop plants and its physiological effects. Soil Sci. Plant Nutr. (Tokyo) 9:85-91.
- Bar-Tal, A., B. Bar-Yosef, and Y. Chen. 1988. Effects of fulvic acid and pH on zinc sorption on montmorillonite. Soil Sci. 146:367-373.
- Bkardwaj, K.K.R. and A.C. Gaur. 1972. Growth Promoting Effect of Humic Substances on Rhizobium trifolu. Ind. J. of Microbiol. (12), 19-21
- Brannon, C.A., and L.E. Sommers. 1985a. Preparation and characterization of model humic polymers containing organic phosphorus. Soil Biol.Biochem 17:213-219.
- Brannon, C.A., and L.E. Sommers. 1985b. Stability and mineralization of organic phosphorus incorporated into model humic polymers. Soil Biol.Biochem 17:221-227..
- Brownell, J.R., O. Nordstrom, I. Marihart, and G. Jorgensen. 1987. Crop responses from two new Leonardite extracts. Sci.Total Environ. 62:492-499.
- Cegarra, J., Garcia, D., Navarro, A. and M.P. Bernal. 1994. Effects of heat on the alkali extraction of humic substances from peat. Communications in soil science and plant analysis. v. 25 (15/16) p. 2685-2695.
- Chen, Y. and T. Aviad 1990. Effects of Humic Substances on Plant Growth. In P. MacCarthy et al. Eds. Humic Substances in Soil and Crop Sciences: Selected Readings. Amer. Soc. of Agron., Madison WI. p. 161-186.
- Chen, Y., and M. Schnitzer. 1978. The surface tension of aqueous solutions of soil humic substances. Soil Sci. 125:7-15.
- Conte, P. and A. Piccolo. 1999. High pressure size exclusion chromatography (HPSEC) of humic substances: molecular sizes, analytical parameters, and column performance. Chemosphere v. 38 (3) Pages: p. 517-528.
- Crowford, J.H., Senn, T.L. and G. E. Stembridge. 1968. The Influence of Humic Acid Fractions on Sprout Production and Yield of the Carogold Sweet Potato. S. Carolina Ag. Exp. Sta. Tech. Bull. 1028.

- De Almeida, R.M., F. Pospisil, K. Vackova, and M. Kutacek. 1980. Effect of humic acids on the inhibition of pea choline esterase and choline a-cyltransferase with malathion. Biol. Plant. 22:167-175.
- Dekock, P.C. 1955. The influence of humic acids on plant growth. Science (Washington, DC) 121:473-474.
- Dixit, V.K., and N. Kishore. 1967. Effect of humic acid and fulvic acid fraction of soil organic matter on seed germination. Indian J. Sci. Ind. Sec. A 1:202-206.
- Dormaar, J.F. 1975. Effects of humic substances from chernozemic Ah horizons on nutrient uptake by *Phaseolus vulgaris* and *Festuca scabrella*. Can. J. Soil Sci. 55:111-118.
- Duval, J.R. 1998. Evaluating leonardite as a crop growth enhancer for turnip and mustard greens. HortTechnology. Oct/Dec 1998. v. 8 (4) Pages: p. 564-567. Texas A & M.
- Fuhr, F., and D. Sauerbeck. 1967a. The uptake of colloidal organic substances by plant roots as shown by experiments with "C-labelled humus compounds. p. 73-82. *In* Report FAO/IAEA Meeting, Vienna, Pergamon Press, Oxford.
- Fuhr, F., and D. Sauerbeck. 1967b. The uptake of straw decomposition products by plant roots. p. 317-327. In Report FAO/IAEA Meeting, Vienna, Pergamon Press, Oxford.
- Gaur, A.C. 1964. influence of humic acid on growth and mineral nutrition in plants. Bull. Assoc. Fr. Itude Sol. 35:207-219.
- Georgacakis, D., Tsavdaris, A., Bakouli, J., and S. Symeonidis. 1996. Composting solid swine manure and lignite mixtures with selected plant residues. Bioresource technology: May/June 1996. v. 56 (2/3). p. 195-200.
- He, X.T., Stevenson, F.J., Mulvaney, R.L. and K.R. Kelly. 1988. Incorporation of newly immobilized 15N into stable organic forms in soil. Soil Biol. Biochem. 20:75-81.
- Iswaran, V., and P.K. Chonkar. 1971. Action of sodium humate and dry matter accumulation of soybean in saline alkali sail. *In* B. Novak ct al. (ed.) Humus et Planta. V:613-615. Prague.
- Ivanova, L.V. 1965. Influence of humic substances on growth of excised maize roots. Dokl. Abad. Nauk. BSSR 9:255-257.
- Jelenic, D.B., M. Hajdukovic, and Z. Aleksic. 1966. The influence of humic substances on phosphate utilization from labeled superphosphate. p. 85-88. *In* The use of isotopes in soil organic matter studies. FAO/IAEA Tech. Meet., Pergaman Press, Oxford.
- Kelting, M. 1997. Humate-based biostimulants do not consistently increase growth of containergrown Turkish hazelnut. Journal of environmental horticulture.v. 15 (4) p. 197-199.
- Kelting, M., Harris, J.R., and J. Fanelli. 1998a Humate-based biostimulants affect early posttransplant root growth and sapflow of balled and burlapped red maple. HortScience . v. 33 (2) Pages: p. 342-344.
- Kelting, M., Harris, J.R., Fanelli, J., and B. Appleton.1998b. Biostimulants and soil amendments affect two-year posttransplant growth of red maple and Washington hawthorn. HortScience. v. 33 (5) Pages: p. 819-822.
- Kohl, S.D. and J.A. Rice. 1998. The binding of contaminants to humin: a mass balance. Chemosphere v. 36 (2) p. 251-261.
- Ladd, J.M., and J.H.A. Butler. 1971. Inhibition and stimulation of proteolytic enzyme activities by sail humic acids. Austr. J. Sail Res. 7:253-261.

- Lee, Y.S., and R.J. Bartlett. 1976. Stimulation of plant growth by humic substances. Soil Sci. Sec. Am. J. 40:876-879.
- Lobartini, J.C., Tan, K.H., and C. Pape. 1998. Dissolution of aluminum and iron phosphate by humic acids. . v. 29 (5/6) Commun. soil sci. plant anal. p. 535-544.
- Linehan, D.J. 1976. Some effects of a fulvic acid component of soil organic matter on the growth of cultivated excised tomato roots. Soil Biol. Biochem. 8:511-517.
- Linehan, D.J., and H. Shepherd. 1979. A comparative study of the effects of natural and synthetic ligands on ion uptake by plants. Plant Soil 52:281-289.
- Malcolm, R.E., and D. Vaughan. 1979a. Comparative effects of sail organic matter fractions on phosphatase activities in wheat roots. Plant Sail 51:117-126.
- Malcolm, R.E., and D. Vaughan. 1979b. Effects of humic acid fractions on invertase activities in plant tissues. Soil Biol. Biochem. 11:65-72.
- Malcalm, R.E., and D. Vaughan. 1979c. Humic substances and phosphatase activities in plant tissues. Sail Biol. Biochem. 11:253-259.
- Martin, J.A. and T.L. Senn. 1967 The Influence of Various Rates of Nitrogen and Humic Acid Derivatives on the Growth and Yield of Greenhouse Tomatoes. S. Carolina Ag. Exp. Sta. Research. Series No. 95.
- Mato, M.C., R. Fabregas, and J. Mendez. 1971. inhibitory effect of soil humic acids on indoleacetic acid oxidase. Soil Biol. Biochem. 3:285-288.
- Mato, M.C., M.G. Olmedo, and I. Mendez. 1972a. Inhibition of indoleacetic acid oxidase by soil humic acids fractionated in Sephadex. Soil Biol. Biochem. 4:469-473.
- Mato, M.C., L.M. Oonralez-Alonso, and J.Mendez. 1972b. Inhibition of enzymatic indoleacetic acid oxidation by fulvic acids. Soil Biol. Biochem. 4:475-478.
- Nardi, S., Pizzeghello, D., Gessa, C., Ferrarese, L. Trainotti, L., and G. Casadoro. 2000 A low molecular weight humic fraction on nitrate uptake and protein synthesis in maize seedlings. Soil Biol. & Biochem. 32(2000) 415- 419.
- Piccolo, A., Celano, G. and G. Pietramellara 1993. Effects of fractions of coal-derived humic substances on seed germination and growth of seedlings (Lactuca sativa and Lycopersicon esculentum). Biology and fertility of soils. v. 16 (1) Pages: p. 11-15.
- Piccolo, A. and J.S.C. Mbagwu. 1989. Effects of humic substances and surfactants on the stability of soil aggregates. Soil science. Jan 1989. v. 147 (1) p. 47-54.
- Piccolo, A., Nardi, S., Concheri, G. 1992. Structural characteristics of humic substances as related to nitrate uptake and growth regulation in plant systems. Soil biol. and biochem. v. 24 (4) p. 373-380.
- Piccolo, A., Pietramellara, G. and J.S.C. Mbagwu. 1997. Reduction in soil loss from erosionsusceptible soils amended with humic substances from oxidized coal. Soil technology. v. 10 (3) Pages: p. 235-245.
- Pinton, R., Cesco, S., Iacolettig, G., Astolfi, S. and Z. Varanini. 1999. Modulation of NO3uptake by water-extractable humic substances: involvement of root plasma membrane H+ATPase. Plant and soil. v. 215 (2) p. 155-161.
- Rauthan, B.S., and M. Schnitzer. 1981. Effects of soil fulvic acid on the growth and nutrient content of cucumber (*Cucumus sativus*) plants. Plant Soil. 63:491-495.

- Reynolds, A.G., Wardle, D.A., Drought, B., and R. Cantwell 1995. Gro-Mate soil amendment improves growth of greenhouse-grown 'Chardonnay' grapevines. HortScience. v. 30 (3)Pages: p. 539-542.
- Sanchez-Conde, M.P., and C.B. Ortega. 1968. Effect of humic acid on the development and the mineral nutrition of the pepper plant. p. 745-755. *In* Control de la Fertilizacion de las plantas cultivadas, 2" Cologuia Evr. Medit. Cent. Edafal. Biol. Aplic. Cuarto, Sevella, Spain.
- Sanchez-Conde, M.P., C.B. Ortega, and M.I. Perz Brull. 1972. Effect of humic acid on sugar beet in hydroponic culture. Arales de edafologia y'i\grobialogia 31:319-331.
- Santi, S., Locci, G., Pinton, R., Cesco, S. and Z Varanini. 1995. Plasma membrane H<sup>+</sup> -ATPase in maize roots induced for NO<sub>3</sub><sup>-</sup> uptake. Plant Physiol. 109, 1277-1283
- Schnitzer, M., and P.A. Poapst. 1967. Effects of a soil humic compound on roof initiation. Nature (London) 213:598-599.
- Sladky, Z. 1959a. The effect of extracted humus substances on growth of tomato plants. Biol. Plant. 1:142-150.
- Sladky, Z. 1959b. The application of extracted humus substances to overground parts of plants. Biol. Plant. 1:199-204.
- Sladky, Z. 1965. Anatomic and physiological alternations in sugar beet receiving foliar applications of humic substances. Biol. Plant. 7:251-260.
- Sladky, Z. and V. Tichy. 1959. Applications of humus substances to overground organs of plants. Biol. Plant. 1:9-15.
- Smidova, M. 1962. Effect of sodium humate on swelling and germination of plant roots. Biol. Plant. 4: 112-118.
- Stevenson., F.J. 1994. Humus Chemistry: Genesis, Composition, Reactions, 2<sup>nd</sup> Ed. John Wiley & Sons. p.13, 236-256.
- Tan, K.H. 1978. Effects of humic and fulvic acids on release of fixed potassium. Geoderma 21:67-74.
- Tan, K.H., and D. Tantiwiramanond. 1983. Effect of humic acids an nodulation and dry matter production of soybean, peanut and clover. Soil Sci. Soc. Am. J. 47:1121-1124.
- Valdrighi, M.M. 1995. Effects of humic acids extracted from mined lignite or composted vegetable residues on plant growth and soil microbial populations. Compost science & utilization. Winter 1995. v. 3 (1) Pages: p. 30-38.
- Vallini, G. Pera, A. Agnolucci, M., and M.M. Valdrighi . 1997. Humic acids stimulate growth and activity of in vitro tested axenic cultures of soil autotrophic nitrifying bacteria. Biol. and fert, of soils. 1997. v. 24 (3) p. 243-248.
- Varadachari, C., Chattopadhyay, T. and K. Ghosh. 1997 Complexation of humic substances with oxides of iron and aluminum. Soil science. v. 162 (1) Pages: p. 28-34.
- Vaughan, D, and D.1. Linehan. 1976. The growth of wheat plants in humic acid solutions under axenic conditions. Plant Soil 44:445-449.
- Vaughan, D., and R.E. Malcolm. 1979. Effect of soil organic matter on peroxidase activity of wheat roots. Soil Biol. Biochem. 11:57-63.
- Vaughan, D., and I.R. McDonald. 1971. Effects of humic acid on protein synthesis and ion uptake in beet discs. J. Exp. Bot. 22:400-410.

- Vaughan, D., and I.R. McDonald. 1976. Some effects of humic acid on the cation uptake by parenchyma tissue. Soil Biol. Biochem. 8:415-421.
- Vaughan, D., and B.G. Ord. 1981. Uptake and incorporation of 14C--labeled soil organic matter by roots of *Pisum sativum* L. J. Exp. Bet. 32:679-687.
- Wang, X.J., Wang, Z.Q., and S.G.Li. 1995. The effect of humic acids on the availability of phosphorus fertilizers in alkaline soils. Soil Use Manage. v. 11 (2) p. 99-102.
- Xudan, X. 1986. The effect of foliar application of fulvic acid on water use, nutrient uptake and wheat yield. Aust. J. Agric. Res. 37:343-350.