Title:

Developing testing protocols to assure the quality of fertilizer materials for organic agriculture

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Supporters

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2. **Wallace Laboratories,** 365 Coral Circle, El Segundo CA 90245
3. **California Certified Organic Farmers (CCOF)** 2155 Delaware Ave, Suite 150, Santa Cruz, CA 95060

CDFA Funding Request Amount/Other Funding

	Requested:	Other funding:
2010	$ 49,995	$ 10,838
2011	$ 49,995	$ 10,838
2012	$ 49,995	$ 15,689
TOTAL	**$149,985**	**$ 37,365**

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B. Executive Summary

Fertilizer labeled as "suitable for organic production" sold to growers of organic produce was recently found to be adulterated with synthetic fertilizer. Use of synthetic fertilizer is not allowed in organic agriculture. This incident undermined the public trust in the "organic" label of California food products with potential negative consequences for the entire organic agriculture industry. There is an urgent need to bring more transparency and authentication to the array of organic fertilizer products on the market.

The objective of the proposed project is to develop a suite of methods that can be used by test laboratories and regulatory agencies to detect adulteration of organic fertilizers and soil amendments by synthetic fertilizer. As a first step, we will build a comprehensive database of quantifiable properties of naturally occurring substances used in organic fertilizers, potential synthetic adulterants (i.e. synthetic fertilizer), and organic fertilizers and soil amendments. Examples of quantifiable properties are the natural stoichiometric elemental composition or the stable isotope ratios of carbon, nitrogen, and oxygen. The data for this database will come from the scientific literature and through analyses of raw materials, organic fertilizers and soil amendments, and synthetic fertilizers in our laboratory and at the UC Davis Stable Isotope Facility. Expected ranges of values for each of the properties of interest will be deduced from the multitude of data collected. Additional correlation analysis will be used to validate the source and makeup of the primary fertilizer ingredients. Once the datasets have been evaluated and principal trends of properties have been validated, guidelines that outline how an organic fertilizer material is to be tested will be developed. To evaluate the robustness of the testing protocols and to measure the success of the project's final product (i.e. the testing protocol), participating test laboratories will have to distinguish between adulterated and unadulterated materials in "blind" tests by following the protocols. The database will be publicly available and serve as a resource and means to standardize guidelines and protocols for the organic fertilizer industry. Once developed, we hope the outcome of the proposed work will then be used by regulatory agencies to create a framework to effectively deal with adulterated organic fertilizers and soil amendments.

The findings of this research will be disseminated at conferences attended by the fertilizer industry and users of the product (i.e. test protocol) and through various newsletters to make sure that the test labs and regulatory agencies understand the nature and technical limitations of the testing approaches.

Growers, the fertilizer industry, regulatory agencies and test labs, and the public will benefit from the findings and products of this project because the organic fertilizer industry and their products will become more transparent and minimize concerns with authenticity. Manufacturers of organic fertilizers and soil amendments will have guidelines that will ensure their products meet the rigid organic label requirements. Most importantly, trust in organically grown foods will be restored.

C. Justification

There is growing concern about the authenticity and integrity of soil and crop amendments sold for use in organic production in California ("Organic farms unknowingly use synthetic fertilizer", Sacramento Bee, Dec 28, 2008; see also letter from the Executive Director of the Organic Materials Review Institute, Feb. 20, 2009, http://omri.org/OMRI_PR.html). For example, synthetic ammonia, which is not permitted in organic production, may be added to a product claimed to be derived from fish; as expected, the resulting product is very effective as a fertilizer but is much cheaper to produce, ensuring a higher profit for the manufacturer. In addition, the product quality and consistency is enhanced giving the illusion the "organic fertilizer" is of a better grade than other materials or competing products on the market. Since much of the organic amendment certification process is based on trust, such adulterated products are often approved and labeled as suitable for organic agriculture. Depending on the degree of adulteration, basic laboratory tests often cannot indicate a problem. Analysis of nitrogen content, for example, may confirm a product label, but will not indicate if the product has been adulterated. The problem undermined public trust in the "organic" label of produce in California, and this could negatively affect growers of organic foods.

This project will develop guidelines and protocols to test organic fertilizers for their authenticity and will contribute to restoring trust in producers of organic fertilizers and in fairness of the marketplace and confidence of consumers in being offered produce that has been grown according to organic standards. The project is directly related to CDFA's goals of assisting industry efforts to increase public confidence in the food supply and to provide for an equitable marketplace for California agriculture. The proposed organic fertilizer testing procedures that will be developed will benefit the whole organic agriculture industry and the public. Therefore, this project will likely contribute to organic agriculture's future economic success.

The project has the potential to provide methods to test labs and regulatory agencies to detect adulteration of organic fertilizers. In addition, the proposed methods will provide the basis to develop standards to ensure the authenticity of organic amendments. To measure success of the project's product (i.e. the testing protocols), the methods will be will be given to participating test labs to distinguish in "blind" trials between adulterated and non-adulterated organic fertilizers. If successful, the project will eliminate or reduce adulteration of organic fertilizers and amendments. Manufacturers of adulterated organic fertilizers and amendments will be faced with a standard testing process to establish the authenticity of their products. Legitimate producers of fertilizers will benefit by having a defined set of testing protocols to ensure the quality of their products. The project will thus contribute to greater transparency and authenticity of fertilizer products intended for organic agriculture.

Previously, no systematic research has been undertaken to develop comprehensive guidelines on testing the authenticity of organic fertilizers and amendments. However, some of the techniques that will be incorporated into the testing protocols, such as the use of stable isotope analysis, have been used in criminal forensic, ecosystem and physiological studies. Important to this proposal is the biogeochemical literature that addresses the sources, fractionation and pathways of carbon, nitrogen and oxygen isotopes within different trophic levels of food webs and unique organismal metabolic pathways (Schimel 1993; Horwath et al. 2001). A great deal of information that can be used for the present project can be found in the literature although this information is highly fragmented.

This project will contribute to better organize the characterization of materials that can be used in manufacturing and testing of organic fertilizers and amendments, and will supplemented with information from our own analyses. The major new product generated by this project is a method of detecting with high probability adulteration of organic fertilizers and other amendments by synthetic fertilizer and other chemical nutrient sources.

D. Objectives

1. Construct a database of materials used in organic and synthetic fertilizers and their quantifiable properties through thorough search of the literature and additional chemical and physical analyses of such materials.
2. Establish natural ranges for the chosen properties of these materials that can be used to distinguish between pure, or unadulterated, and adulterated materials.
3. Develop a stepwise protocol test that labs and regulatory agencies can follow to identify organic fertilizers that have likely been adulterated by synthetic fertilizers.
4. Carry out blind tests with collaborating test labs to evaluate the robustness of the above protocol.
5. Disseminate the results and products of the project to potential users, such as organic fertilizer test labs and regulatory agencies.

E. Work plans and Methods

Tasks to be completed

Task 1. Conduct a literature review on the materials used in organic and inorganic fertilizer materials.
Task 2. Analyze the collected materials for stable isotope and nutrient content.
Task 3. Build a database.
Task 4. Evaluate and summarize the dataset.
Task 5. Construct usable guidelines to be used by test labs and regulatory agencies.
Task 6. Evaluation: Measuring success.
Task 7. Outreach

Year 1:
In the first year (2010), we will conduct a thorough literature review (task 1), and we will complement the data in the literature with our own analyses of raw materials (task 2). We will further collect fertilizers used in organic agriculture and analyze them. Details are given below.

Task 1. Conduct a literature review on the materials used in organic and inorganic fertilizer materials.

The scientific literature will be comprehensively searched with the goal of compiling quantifiable properties of materials relevant to this study. Materials included will be naturally occurring substances (i.e. collected unaltered), potential synthetic adulterants and some elaborated products. Examples of properties that have been measured are stable isotope ratios of carbon, nitrogen, oxygen and deuterium, the ratio of carbon to nitrogen, concentrations of nitrogen and carbon, ratio of nitrogen to phosphorus, or phosphorus concentration. In addition, quantities of other elements, such as calcium and magnesium, may provide further validation of source of the ingredients in fertilizers. Such data have typically been determined in ecological studies to study food web dynamics and decomposition processes. The information contained in the scientific literature in the aggregate will be most useful both in the initial stage of this project and in the final analysis to evaluate the usefulness of a specific measurement as a potential tool to detect adulteration in organic fertilizer. Examples of this literature compilation are given in Table 1. The literature review will guide and complement our own measurements on materials used as organic fertilizers and will be instrumental in evaluating our results and in constructing guidelines to be used by test labs and regulatory agencies.

Table 1. Examples of measured properties of materials potentially used in organic fertilizers and as adulterants, as reported in the scientific literature. The properties are the isotope ratios of carbon and nitrogen ($d^{13}C$ and $d^{15}N$), carbon content (%C), nitrogen content (%), carbon to nitrogen ratio (C:N), and phosphorus content (%P).

Products	$d^{13}C$	$d^{15}N$	%C	%N	C:N	%P	Reference
Raw anchovy	-18	13	42	11	4.2		Yokoyama et al. 2006
Herring fish meal			11.5			1.7	Luzier et al. 1995
whole fish (pike)						1.8-2.3	Tanner et al. 1999
aqua ammonia		-0.3					Freyer and Aly 1974

Task 2. Analyze the collected materials for stable isotope and nutrient content.

We have begun the process of collecting materials used as organic fertilizers, as well as raw materials used to manufacture commercial products, and synthetic materials that might be used as adulterants. We have so far collected several dozen different organic fertilizers obtained from growers and dealers, but the initial collection only represents a fraction of the materials used in the manufacture of organic fertilizers and amendments. The success of this project will depend on collecting as many products and source materials as possible to accurately develop the proposed methods. . The collection will include all of the major classes of organic fertilizer currently on the market in California, such as fishmeal, liquid fish hydrolyzate, fish emulsion, blood meal, feather meal, guano, and products formulated from more than one raw material. These materials will be analyzed for physical and chemical properties.

The goal of task 2 is to have a comprehensive list of materials used to produce organic fertilizers and amendments. In addition, the collection of conventional fertilizers and their ingredients is also required to assess to potential and degree of their use in organic products. . All of these materials will be associated with a set of properties, such as carbon (C), nitrogen (N) and phosphorus (P) content, C:N and N:P ratios, stable isotope ratios of C, N, and oxygen (O), as well as physical properties, such as solid to liquid ratios, particle size, density etc. This information will be included in the database described under task 3.

Methods for task 2. Physical properties to be measured include density, and solids versus liquid fraction. The stable isotope ratios of carbon, nitrogen and oxygen, i.e. the ratios of $^{13}C/^{12}C$, $^{15}N/^{14}N$, and $^{18}O/^{16}O$, in the fertilizer materials will be determined by a 20-20 ANCA (automated nitrogen and carbon analyzer) coupled to a mass spectrometer (Europa, Cheshire, UK) and by a HEKATech thermal conversion elemental analyzer coupled to a PDZ 20-20 mass spectrometer (Europa, Cheshire, UK) at the UC Davis Stable Isotope Facility. These procedures also yield total C, N, and O content. Additionally, total nitrogen, total phosphorus, and other elements may be determined by sulfuric acid (Lindner 1944) or nitric acid (Ippolito and Barbarick 2000) digestion followed by inductively coupled plasma or atomic adsorption analysis.

Year 2:
In the second year (2011), we will continue with the analysis of materials (task 2, year 1), build a database (task 3), evaluate and summarize the dataset (task 4), and conduct outreach (task 7). Details are given below.

Task 3. Build a database.

The database will be the foundation for the construction of the guidelines and protocols to be used by test labs and regulatory agencies. The database will include both published values of materials' properties from the literature and information on materials obtained through analyses in our lab. The database will be set up to allow continuous updates as materials used in fertilizers emerge. The database will be publically available and serve as a product of this project to serve as a resource and means to standardize guidelines and protocols for the organic fertilizer industry. Additional variables that can used to characterize commercial products or raw materials may be assessed and included in the database.

Methods for task 3. The database will be cross-referenced and annotated with all pertinent information. All data and reference files will be backed up daily on the departmental server. The database will ideally eventually be web based. We will work with the California Certified Organic Farmer (CCOF) (see **Appendix, Letters of Support**) organization to establish a web based database as a future effort derived from the efforts of this project.

Task 4. Evaluate and summarize the dataset.

From the multitude of data, we will deduce expected ranges of values for each of the properties of interest in the diverse range of materials. The establishing of threshold or boundary values is one of the most challenging aspects of this research since in the natural world, multiple factors can influence concentrations of nutrients, and in particular, stable isotope ratios. Therefore, many datasets must be critically evaluated. Once highly probably boundary values for several properties are established, combining information on a range of properties will become a powerful method for detecting ingredients of organic fertilizers, including adulterants.
Based initially on existing literature, an expected or "natural" range of values will be presented for each of the properties of interest. This evaluation will describe how the characteristics of a product might be altered if it has been adulterated, and will suggest threshold values beyond which a product may be suspected of being adulterated. As the database is continually amended, this evaluation will be strengthened by the diversity of samples obtained from different manufacturers. However, since the integrity of products received for analysis is not assured (some products may be adulterated or under suspicion of being adulterated), results obtained in the laboratory will be integrated into this evaluation using the literature database as a guide. Samples that show properties outside the "natural" limits, especially if they are from dubious manufacturers, will still contribute to this evaluation, in this case to confirm suggestions about how a material might change if it has been adulterated.

The following example illustrates how a relatively simple measurement such as carbon to nitrogen ratio (C:N), with a suggested threshold value, may be used to question the integrity of a product. The preliminary data shown in Figure 1 represent fish-based fertilizers from several different suppliers. The N contained in fish tissue is organic, consisting primarily of protein-derived amino acids, which range in C:N from just above 1 to approximately 8. A product derived exclusively from fish should therefore not have a C:N lower than about 1 or 2, even in the extreme case that the protein consisted of mostly N-rich amino acids. Adulteration with ammonia/ammonium would increase the amount of N relative to C, decreasing the C:N ratio; a low C:N could indicate some degree of adulteration. Indeed, all of the products which show a low C:N have been suspected of being adulterated. A more advanced measurement such as nitrogen isotope ratio (expressed as $\delta^{15}N$) can then strengthen this tentative assessment. Synthetic nitrogen has a low (zero to negative) $\delta^{15}N$ value relative to fish-nitrogen. This is because synthetic N is derived from atmospheric N_2 (with a $\delta^{15}N$ of zero), whereas animal tissues have higher $\delta^{15}N$ values reflecting biochemical preference for retaining the heavier isotope as new tissue is synthesized and as the food chain advances to higher trophic levels (Peterson and Fry 1987, Kendall 1998). The addition of synthetic N to a product would therefore tend to lower the $\delta^{15}N$ of an organic fertilizer derived solely from fish. Figure 1 shows that the samples with a low C:N ratio also had a relatively low $\delta^{15}N$ value. As expected, there was a relationship between C:N and $\delta^{15}N$.

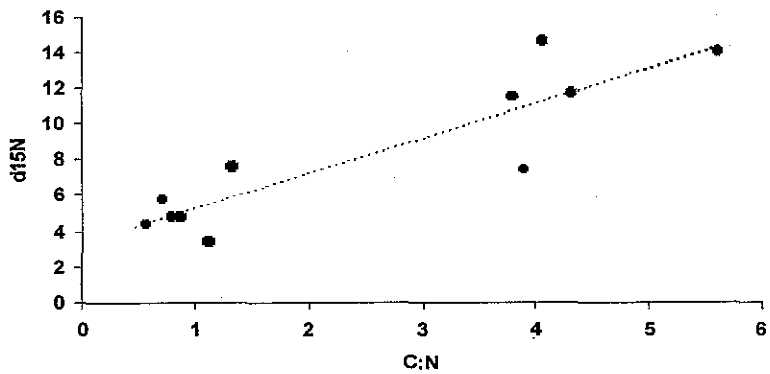

Figure 1. The carbon to nitrogen ratio *vs.* the $\delta^{15}N$ value of 11 fish-based fertilizers analyzed in our lab and at the UC Davis Stable Isotope Facility.

Methods for task 4. Compilation of data, regression analyses and validation of data in the literature will all serve to develop robust "natural" ranges of properties based on many variables and datasets.

Year 3:

In the third year (2012), we will develop the guidelines (task 5), test the guidelines and revise them as necessary (task 6), revise the evaluation of our dataset (task 4, year 2), update the database (task 3, year 2), conduct outreach (task 7).

Task 5. Construct usable guidelines to be used by test labs and regulatory agencies.

Once the datasets have been evaluated and principal trends of properties have been validated, guidelines that outline how a material may be tested for potential adulteration will be developed. These guidelines will include information from Tasks 1, 2, 3 and 4. A simplified version of the guidelines incorporated into a flowchart is shown in Figure 2. The flowchart is based on our preliminary findings, using properties listed in Table 1. According to our present knowledge, the C:N ratio would be an easy-to-measure property giving a strong indication on the authenticity of a tested product. If the C:N ratio was suspect, further tests could be recommended. Measurement of isotope ratios of C, N, and O would be recommended if multiple variables suggested that adulteration of a natural product with synthetic fertilizer might have occurred.

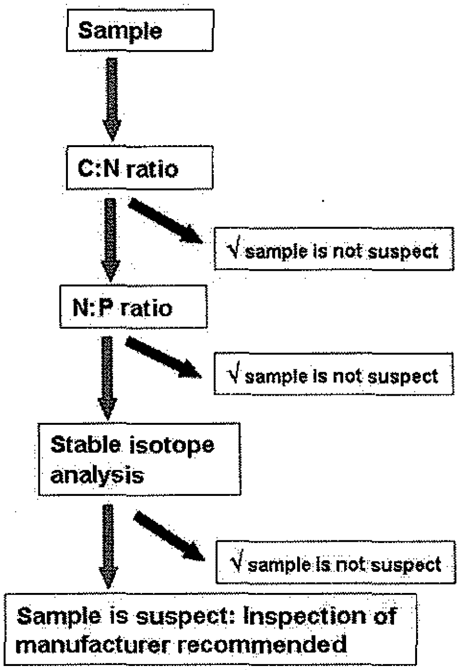

Figure 2. A conceptual flowchart showing an example of an recommended course of action for test labs and regulatory agencies using the guidelines that will be developed. The purpose of these tests is to ascertain the authenticity of organic fertilizers. The example is based on preliminary results and illustrates how a series of tests will lead to a recommended inspection of a manufacturing facility if results of initial relatively simple tests look suspect.

The guidelines for the testing labs and regulatory agencies will include all the information about a material and possible steps that need to be taken to evaluate such a product, but they will be written in a way to be practical for institutions that use the guidelines.

F. Project Management, Evaluation, and Outreach

1. Management

William R. Horwath. Dr. Horwath will be primarily responsible for all analytical procedures outlined in the proposal. Dr. Horwath has published numerous articles using stable isotopes and elemental analysis. Dr. Horwath will also be responsible for data analysis and interpretation, reporting and writing of results for scientific journals.

Stuart Pettygrove. Dr. Pettygrove will be responsible for implementing the extension and outreach activities in the proposal. Dr. Pettygrove will also write the news bulletin and extension article for the project.

The proposed work will be coordinated through regular meetings with Dr. Horwath, Dr. Pettygrove, Dr. Burger, Project Scientist in Horwat's lab and T. Doane, Specialist in Horwath lab. As data is collected and interpreted more meetings will be held to discuss the outreach and education components of the project (See **Task** 7). Dr. Horwath will be primarily responsible for coordinating these meetings and reporting of results.

2. Evaluation (Task 6)

Once the guidelines are ready for use, we will send at least 20 different organic fertilizers, some of them adulterated and some of them unadulterated organic fertilizers, to participating test laboratories (see **letters of support, Appendix**). Following the guidelines, the testing labs should be able to detect, which of the materials were adulterated. This process may be repeated several times as our guidelines improved and refined, and as the scope of materials to be tested expands.

This research has many stakeholders, such as growers, retailers of organic fertilizer, regulatory agencies, as well as the public, because having adequate test procedures will restore trust in the organic label of produce. Therefore, the organic industry as a whole and the consumers will benefit from this project. However, the actual users of the research product, i.e. the guidelines and protocols to test fertilizers used in organic agriculture, are limited in number, and we are confident that we can communicate our findings to them by the means described above.

3. Outreach (Task 7)

The purpose of the outreach program is to educate dealers (retailers) of organic fertilizer, test labs and regulatory agencies to understand the testing approaches.

We will engage in at least three outreach activities in each year. First, we will present our findings at the fall FREP Conference and submit our presentations for publication in the Conference Proceedings. Second, we will participate at the fall Western Plant Health Association seminars. Third, we will write a short summary to be published in the WPHA e-mail newsletter. Fourth, we will present at the California Plant and Soil Conference and will submit an article to the Conference Proceedings. Fifth, we will write an article for the Agriculture and Natural Resources (ANR) Extension Bulletin. Sixth, at the end of the project we will submit at least one article for publication in a scientific journal

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