**ORIGINAL SUBMISSION**

#1641

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March 28, 2016

GRN 000646

Paulette Gaynor, Ph.D.
Deputy Division Director
Office of Food Additive Safety (HFS-200)
Center for Food Safety and Applied Nutrition
Food and Drug Administration
5100 Paint Branch Parkway
College Park, MD 20740-3835

RE: Pecan Shell Fiber GRAS Notification

Dear Dr. Gaynor:

In accordance with proposed 21 CFR § 170.36 (a notice of a claim for exemption based on a GRAS determination) published in the Federal Register (62 FR 18937-18964), I am submitting as the agent of the notifier, the Southeastern Reduction Company, a Division of South Georgia Pecan Company, Inc., 309 South Lee Street, Valdosta, GA 31601, a GRAS notification for the use of pecan shell fiber as a source of fiber and antioxidants in the diet at a maximum consumption level of 10,233.6 mg/day. A GRAS expert panel dossier, setting forth the basis for the GRAS determination, as well as *curriculum vitae* of the members of the GRAS panel, are enclosed.

Best regards,

(b) (6)

**George A. Burdock, Ph.D., DABT, FACN**
*Diplomate*, American Board of Toxicology
*Fellow*, American College of Nutrition

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FOOD ADDITIVE SAFETY

## 1. GRAS Exemption Claim

### A. Claim of Exemption from the Requirement for Premarket Approval Pursuant to Proposed 21 CFR 170.36(c)(1)

Southeastern Reduction Company, a Division of South Georgia Pecan Company, Inc., on the advice of qualified experts, has determined pecan shell fiber to be generally recognized as safe (GRAS) as a food ingredient and therefore, exempt from the requirement of premarket approval, under the conditions of its intended use as described below. The basis for this finding is described in the following sections.

Signed,

(b) (6)

Date 28 March 2016

**George A. Burdock, Ph.D.**
*Diplomate*, American Board of Toxicology
*Fellow*, American College of Nutrition
859 Outer Road
Orlando, FL 32814

**(i) Name and Address of the Notifier**

John Nizio
Southeastern Reduction Company
Division of South Georgia Pecan Company, Inc.
309 South Lee Street
Valdosta, GA 31601

**Agent of the Notifier:**

George A. Burdock, Ph.D.
*Diplomate*, American Board of Toxicology
*Fellow*, American College of Nutrition
Burdock Group
859 Outer Road
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Telephone: 407-802-1400
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**(ii) Common Name of the Notified Substance**

The common name pecan shell fiber, for the purposes of this GRAS Notification has been defined as:

Pecan Shell Fiber

**(iii) Conditions of Use**

Pecan shell fiber may be used as an ingredient in the food groups shown in Table 1 in order to provide a source of fiber and antioxidants for foods. Such use could potentially increase dietary (aggregate) exposure of pecan shell fiber to 10,233.6 mg/day. The ingredient is intended to be used solely for purposes other than coloring, and any color imparted is unimportant insofar as the appearance, value, marketability, or consumer acceptability is concerned.

**(iv) Basis of GRAS Determination**

Pursuant to 21 CFR § 170.3, the use of pecan shell fiber as an ingredient in food categories shown in Table 1 at an intended maximum 90$^{th}$ percentile consumption of 10,233.6 mg *per* day, has been determined GRAS by scientific procedures for its intended conditions of use. The safety of pecan shell fiber for this use is supported by publicly available information including, but not limited to 13-week dietary toxicity and genotoxicity studies on pecan shell fiber (Dolan *et al.*, 2016), plus additional genotoxicity studies with an aqueous extract of the fiber. This determination is based on the views of experts who are qualified by scientific training and experience to evaluate the safety of substances used as ingredients in food.

**Table 1. Food groups selected for pecan shell fiber supplementation***

| Food Category | Intended use level (%) |
|---|---|
| Baked goods and baking mixes | 2-5 |
| Breakfast cereals | 5 |
| Confections and frostings | 5 |
| Gelatins, puddings and fillings | 5 |
| Grain products and pastas | 2 |
| Meal replacement | 10 |
| Snack food | 2 |
| Soft candy | 10 |
| Sweet sauces | 5 |

*The food categories correspond to those listed in 21 CFR § 170.3(n), with the exception of meal replacement. Certain categories may contain foods for which a standard of identity exists, which prohibit the addition of ingredients to the food not identified as mandated or optional ingredients under the regulation. Therefore, addition of pecan shell fiber to a food for which a standard of identity exists would demand that the food product be named other than that as indicated under the standard of identity or a waiver of that standard be obtained.

### (v) Availability of Information

The data and information that serve as a basis for this GRAS determination are available for FDA review and copying during conventional office hours at:

Burdock Group
859 Outer Road
Orlando, FL 32814
Telephone: 407-802-1400
Facsimile: 407-802-1405
Email: gburdock@burdockgroup.com

Alternatively, data and information that serve as a basis for this GRAS determination may be sent to FDA upon request.

## 2. Detailed Information about the Identity of the Notified Substance

### A. Identity

Pecan shell fiber (also called pecan shell flour or dried, ground pecan shells) is a fine, reddish brown powder, mainly comprised of insoluble fiber. Pecan shell fiber is included in Everything Added to Food in the United States (EAFUS) (FDA, 2015) and has been determined generally recognized as safe (GRAS) by the Flavor and Extract Manufacturers Association (FEMA, No. 4385) for use as a flavoring substance in meat products at a maximum level of 3000 ppm (Waddell *et al.*, 2007). The American Association of Feed Control Officials (AAFCO) includes Ground Pecan Shells (definition 60.110) in the 2010 and later issues of the Official Publication (OP) of feed ingredients (AAFCO, 2015).

The general descriptive characteristics of pecan shell fiber are presented in Table 2. Packaging for pecan shell fiber will be labeled "May Contain Pecans".

**Table 2. General description of pecan shell fiber**

| Characteristic | Value |
|---|---|
| Source | *Carya illinoensis* |
| Appearance | Fine, reddish brown powder |
| Odor | No characteristic odor |
| Total dietary fiber | 80-85% |
| Insoluble fiber | 78-83% |
| Stability | Up to one year when stored at ambient temperature in unopened containers |
| Labeling | "May contain pecans" included on packaging |

**Common or Usual Name:**

The common name is pecan shell fiber. Pecan shell fiber is also called pecan shell flour or dried, ground pecan shells.

**B. Composition**

The specifications of pecan shell fiber are summarized in Table 3. The primary constituents of the ingredient are cellulose and lignin (53-71% of the ingredient), collectively measured as acid detergent fiber (ADF). Hemicellulose is also present, and is included with cellulose and lignin in the neutral detergent fiber (NDF) measurement (68-78% of the ingredient). Pecan shell fiber also contains moisture (5-10%), polyphenols (3-9%) and proanthocyanidins (8-18%), as well as small amounts of protein (< 3%), fat (< 4.5%) and ash (< 3%). There is low potential for fungal, bacterial, aflatoxin or heavy metal contamination.

**C. Method of Manufacture of Pecan Shell Fiber**

Pecan shell fiber is produced entirely from ground shells of pecan nuts according to current Good Manufacturing Practice (cGMP), with no additional substances added during processing. Pecans in the shell (before cracking; also known as "inshell" pecans) are washed in an ambient temperature water bath to remove any organic material. Pecans in the wash water float on the surface and are tumbled over (forced underwater by paddles) to clean the entire shell. The inshell pecans are then sanitized by submersion in hot water.

After the shells are cracked, the pecan meat is separated from the shells using a system of blowers, vacuum aspirators, water baths, high temperature driers and electronic shell sorters by a continuous process. The shells are transferred pneumatically to the shell grinding facility where they are inspected, passed through magnets and screenings to remove unwanted material and ground into pecan shell fiber. Pecan shell fiber is subjected to air separation and several passes over a magnet during the grinding process. Fine particles are pulled upward and captured, leaving heavier, larger particles behind. Temperature inside the grinding mills is controlled to yield a final moisture content within specification. The final pecan shell fiber product is packaged into 50 lb. multiple layer paper (kraft) bags with polypropylene liners, or polyethylene lined "super sacks" or drums (variety of sizes).

**Table 3. Specifications of pecan shell fiber**

| Analysis | Method | Specification | Batch Analysis Results (*N*=5) Range | Average |
|---|---|---|---|---|
| **Physical/Chemical** | | | | |
| Appearance | Visual | Reddish brown powder | Meets specification | Meets specification |
| US 100 mesh screen (thru) (%) | SRC Wet Screen | ≥ 98 | 100* | 100 |
| US 200 mesh screen (thru) (%) | SRC Wet Screen | ≥ 90 | 95-97 | 96.6 |
| US 325 mesh screen (thru) (%) | SRC Wet Screen | ≥ 75 | 88-91 | 90.0 |
| Moisture content (wt %) | Forced Draft AOAC 930.15 | 5-10 | 6.19-6.61 | 6.42 |
| Fiber, Acid Detergent (%) (Cellulose + Lignin) | Ankom ADF 05/03 or equivalent | 53-75 | 64.6-67.4 | 66.4 |
| Fiber, Neutral Detergent (%) (Cellulose + Lignin + Hemicellulose) | Ankom NDF | 65-78 | 70.9-74.4 | 72.4 |
| Protein (g/100 g) | AOAC 990.03 or 992.15 | < 3 | 1.94-2.74 | 2.26 |
| Fat (g/100 g) | Acid Hydrolysis | < 4.5 | 1.64-3.8 | 3.17 |
| Ash (g/100 g) | AOAC 942.05 | < 3 | 1.90- 2.18 | 1.98 |
| Calories (*per* 100 g) | Atwater calculation | 350-400 | 374-386 | 381.6 |
| Polyphenols (% dry basis)** | Singleton *et al.* (1999) | 3-9 | 3.81 – 8.22 | 5.4 |
| Proanthocyanidins (% dry basis) | USP 29/NF24 | 8-18 | 8.14 – 17.1 | 11.52 |
| **Heavy metals** | | | | |
| Arsenic (ppm) | Modified AOAC 986.15 | < 0.5 | < 0.02* | < 0.02 |
| Cadmium (ppm) | Modified AOAC 986.15 or EPA 3050/6020 USP730 | < 0.5 | 0.021 – 0.025 | 0.023 |
| Lead (ppm) | Modified AOAC 986.15 | < 0.5 | < 0.02 – 0.07 | 0.03 |
| Mercury (ppm) | Modified AOAC 986.15 or EPA 3050/6020 USP730 | < 0.5 | < 0.01* | < 0.01 |
| **Microbiological** | | | | |
| Total Aerobic (cfu/g) | AOAC 966.23 | < 10,000 | <10 – 2750 | 1152 |
| Coliforms (cfu/g) | AOAC 991.14 | < 100 | < 10* | < 10 |
| *Escherichia coli* (cfu/g) | AOAC 991.14 | < 10 | < 10* | < 10 |
| *Salmonella* spp. (*per* 25 g) | AOAC-Rl-100201 | Negative | Negative | Negative |
| Yeast and mold (cfu/g) | FDA-BAM, 7th Ed. | < 100 | 10-80 | 46 |
| Sum of Aflatoxins B1, B2, G1, G2 (ppb) | Modified AOAC 991.31 | < 5 | < 5* | < 5 |

*N* = 9 for polyphenols and proanthocyanidins. * all samples, ** Gallic aid equivalents.
Ppb = parts *per* billion; ppm = parts *per* million

### 3. Self-Limiting Levels of Use

The quantity of pecan shell fiber used in food would be self-limiting due to potential unpalatability.

### 4. Basis of GRAS Determination

The determination that pecan shell fiber is GRAS is on the basis of scientific procedures, as outlined in the attached Dossier in Support of the Generally Recognized as Safe (GRAS) Status of Pecan Shell Fiber as a Food Ingredient, dated January 29, 2016. On the basis of the data and information described in the attached dossier and other publicly available information, there is consensus among experts qualified by scientific training and experience to evaluate the safety of substances added to food, that there is reasonable certainty that pecan shell fiber is GRAS under the intended conditions of use.

### 5. References


AAFCO (2015) 60.110 Ground Pecan Shells. Official names and definitions of feed ingredients. *In 2015 Official Publication. Association of American Feed Control Officials Incorporated.* p. 415.

Dolan, L., Matulka, R., Worn, J. and Nizio, J. (2016) Safety studies conducted on Pecan Shell Fiber, a food ingredient produced from ground pecan shells. *Toxicology Reports* 3:1-11.

FDA (2015) Everything Added to Food in the United States (EAFUS), Doc No. 3779 <<http://www.accessdata.fda.gov/scripts/fcn/fcnDetailNavigation.cfm?rpt=eafuslisting&id=3167>>, (site visited November 16, 2015).

Waddell, W. J., Cohen, S. M., Feron, V. J., Goodman, J. I., Marnett, L. J., Portoghese, P. S., Rietjens, I. M., Smith, R. L., Adams, T. B., Gavin, L. C., McZGowen, M. M. and Williams, M. C. (2007) GRAS Flavoring Substances 23. The 23rd publication by the FEMA Expert Panel presents safety and usage data on 174 new generally recognized as safe flavoring ingredients. *Food Technology* 22-49.

Pages 000009-000068 of duplicate material has been removed under Freedom of Information Exemption E6

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# DOSSIER IN SUPPORT OF THE GENERALLY RECOGNIZED AS SAFE (GRAS) STATUS OF PECAN SHELL FIBER AS A FOOD INGREDIENT

**January 29, 2016**

**FINAL**

**Panel Members**

**Marilyn Aardema, Ph.D.**

**Julie Miller Jones, Ph.D.**

**Joanne Slavin, Ph.D.**

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**DOSSIER IN SUPPORT OF THE GENERALLY RECOGNIZED AS SAFE (GRAS) STATUS OF PECAN SHELL FIBER AS A FOOD INGREDIENT**

**TABLE OF CONTENTS**

# DOSSIER IN SUPPORT OF THE GENERALLY RECOGNIZED AS SAFE (GRAS) STATUS OF PECAN SHELL FIBER AS A FOOD INGREDIENT

## 1. EXECUTIVE SUMMARY

The undersigned, an independent panel of recognized experts (hereinafter referred to as the Expert Panel)[1], qualified by their scientific training and relevant national and international experience to evaluate the safety of food ingredients, was requested by Southeastern Reduction Company, Division of South Georgia Pecan Company, Inc. (hereafter referred to as SRC) to determine the Generally Recognized As Safe (GRAS) status of pecan shell fiber, based on scientific procedures. Pecan shell fiber is to be added to foods identified herein, such that the 90$^{th}$ percentile consumption from all categories, including already approved foods, may be up to 10,233.6 mg/day (170.6 mg/kg bw/day). SRC assures Burdock Group that all relevant, unpublished information in its possession related to the safety of pecan shell fiber has been supplied to Burdock Group and has been summarized in this dossier. A comprehensive search of the scientific literature was conducted through December, 2015 for safety and toxicity information on pecan shell fiber and related substances and has been summarized in this document. Information from the literature search, along with supporting documentation, was made available to the Expert Panel. In addition, the Expert Panel independently evaluated materials deemed appropriate and necessary. Following an independent, critical evaluation, the Expert Panel conferred and unanimously agreed that pecan shell fiber is safe when consumed up to 10,233.6 mg/day.

## 2. INTRODUCTION

The typical American consumes approximately 15 g fiber/day, well under the recommended 14 g/1000 calories, or 25 g/day for women and 38 g/day for men (USDA, 2010). Health benefits associated with high fiber intakes include lower risk for developing coronary heart disease, stroke, hypertension, diabetes, obesity, gastrointestinal diseases and certain forms of cancer, including colon cancer (IOM, 2005; Anderson *et al.*, 2009; Slavin, 2013; Baena and Salinas, 2015; Kunzmann *et al.*, 2015). In an attempt to increase the fiber content of the diet, developers of food ingredients have created new fiber ingredients, some of which are produced by genetically modified organisms. However, due to increased demand by consumers for foods containing "natural" ingredients, there is an increased need for new fiber ingredients from "natural" sources.

Pecan shell fiber (also called pecan shell flour or dried, finely ground pecan shells) is produced from shells of pecan nuts, excluding the husks and nut kernels. Pecan shell fiber is predominantly composed of insoluble fiber (78-83%) and contains small amounts of fat (< 4%) and protein (< 3%). It also contains approximately 5% polyphenols and 12% proanthocyanidins, molecules recognized for antioxidant activity. Therefore, pecan shell fiber has potential as both a fiber ingredient and antioxidant for food. This dossier is a summary of the scientific evidence that supports the general recognition that pecan shell fiber is safe for human consumption as a food ingredient.

[1] Modeled after that described in Section 201(s) of the Federal Food, Drug, and Cosmetic Act, as amended. See also attachments (*curriculum vitae*) documenting the expertise of the Panel members.

## 2.1. Description

Pecan shell fiber is produced in its entirety from the ground shells of pecan nuts. The pecan tree is the common name for a species of hickory (*Carya illinoensis*) belonging to the walnut family. The pecan is native to the United States (US) and was harvested and stored by Native Americans prior to European colonization (Ty Ty Nursery, 2013). Pecan trees thrive in the warm, long growing season of the southern United States, in bottomlands near rivers or lakes. The states of Georgia, Alabama, Louisiana, Texas, Oklahoma, New Mexico and Arizona produce more than 93% of the commercial pecan crop in the US. Natural stands of pecan trees also are found in Kansas, Missouri, Iowa, Illinois, Indiana, Mississippi, Tennessee, Ohio and Kentucky (Reid and Hunt, 2000; Virginia Tech, 2001). Pecan orchards can be planted with grafted trees or seedling trees that are started from nuts, depending on orchard grower preference. Grafted trees reach maturity - by the seventh year (seedling trees require another 2-3 years), and most cultivars remain productive for decades when properly managed. Some pecan tree cultivars will produce a full crop of pecan nuts (pecans) every year, while other varieties are considered alternate-year bearing orchards (Huntrods *et al.*, 2013). In Georgia, the peak harvesting time for pecans is October – December (Georgia Pecan Commission, 2015).

Pecans rank among the most preferred of all nuts and are an economically important crop in the United States (De La Rosa *et al.*, 2011). Pecans tie with walnuts as the second most frequently consumed tree nut in the United States after almonds (De La Rosa *et al.*, 2011). Pecans can be harvested from trees that grow wild or varieties specially bred for superior traits of nut size, ease of shelling, production habit and resistance to insects and diseases (originally referred to as "papershell pecans") (Nesbitt *et al.*, 2013). Shells from both native and papershell pecans are used to produce pecan shell fiber. [2] Papershell pecan varieties include Stuart, Desirable, Wichitaw and Kiowa (Hoyt, 2015).

The pecan fruit's green, fleshy outer husk or shuck splits into four valves upon ripening and exposes one large, single-seeded "nut" (Figure 1). The husk is composed of leathery tissue derived (at least in part) from fused sepals. Each endocarp (nut) contains one seed with two cotyledons. The nut is composed of the seed (also called kernel or "meat"), outer shell, and packing material between the meat and shell (Figure 2). Pecan shells are formidable, but not impervious barriers to the kernel. The hard woody shell is 0.5-1.5 mm thick, but contains small pores between cells,which allow for gas and water exchange (Dimalla and Van Staden, 1978). Substances found in pecan shells include cellulose, hemicellulose, lignin, phenolic compounds, flavonoids and proanthocyanidins (Preston and Sayer, 1992; Villarreal-Lozoya *et al.*, 2007; De La Rosa *et al.*, 2011); the values for phenolic compounds, flavonoids and proanthocyanidins are higher in the shell than in the kernel (Villarreal-Lozoya *et al.*, 2007; De La Rosa *et al.*, 2011).

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[2] Correspondence from Southeastern Reduction Company, November, 1. 2012.

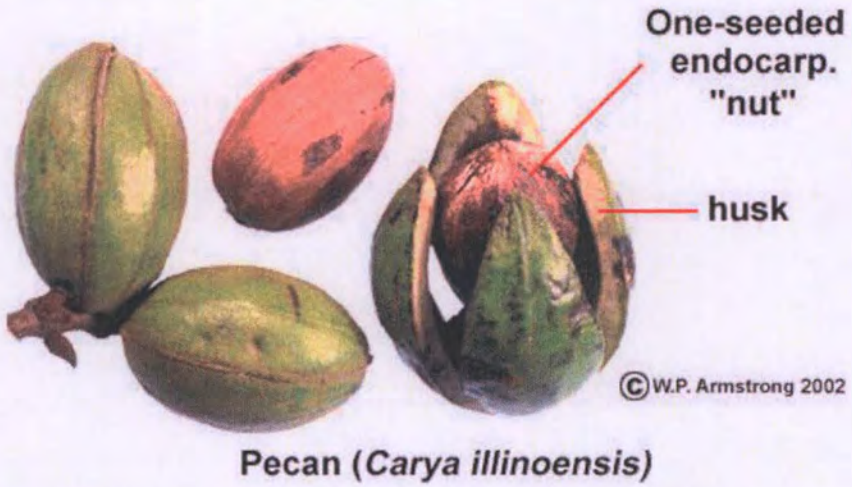


**Figure 1. Appearance of the pecan fruit at harvest (Armstrong, 2009)**

**Figure 2. Appearance of a fully developed unshelled pecan nut (Giles variety) (Reid, 2011)**

## 2.2.History of use

Historically, pecan shells have been viewed as a waste product of the pecan industry. In 2003, the New Mexico and Texas pecan industries produced about 59 million pounds of shells (Stafne *et al.*, 2009). However, some uses of pecan shell fiber or ground or intact pecan shells have been reported.

Pecan shell fiber is used as a flavoring substance for meat (Burdock, 2010) and an inert ingredient for granulated pesticides (EPA, 2015). Pecan shells have been added to barbeque briquette grills or used in smokers and other cooking devices as an alternative to charcoal (Antal Jr. *et al.*, 2000; Revermann, 2016). They also have been used to make liquid smokes (Van Loo *et al.*, 2012). Pecan shells have been used as a roughage source in cattle feed as far back as 1973, at up to 20% in the ration (Cullison *et al.*, 1973).

Several non-food uses of pecan shell fiber or ground or crushed pecan shells have been reported. Pecan shell fiber has been used as filler in plywood phenol-formaldehyde resin adhesives and flakeboard adhesives (Sellers, 1989). It is also mixed with resin to provide a base material for handcrafted/sculptured figurines (Artmolds, 2015). Ground pecan shells are used as a soil conditioner, mulch, or organic carrier for granular pesticide formulations (Goss, *et al.*,1996; Stafne *et al.*, 2009). Crushed pecans shells are used as a source of activated carbon, a lost circulation aid in the oil well drilling industry, and an abrasive for cleaning chemical and refinery plant equipment, boiler tubes, oil field equipment oil field drilling rigs, storage tanks, compressors, turbine compressor components, polishing soft metals, wood plastics, fiberglass and stones. Crushed pecan shells are also effective filter media for removing metals from water and are frequently used in waste treatment systems. In addition, ground pecan shells are suitable for polishing and tumbling items such as brass (ammunition) shell casings and jewelry. In electronic and electrical applications, the material is used to deflash and deburr moldings and castings (Reade Advanced Materials, 2015). Pecan shells also have been used to make a natural dye that gives a warm brown color to textiles (Lazo, 2015).

## 2.3.Regulatory Status

Pecan shell fiber is included in Everything Added to Food in the United States (EAFUS) (FDA, 2015) and has been determined generally recognized as safe (GRAS) by the Flavor and Extract Manufacturers Association (FEMA, No. 4385) for use as a flavoring substance in meat products at a maximum level of 3000 ppm (Waddell *et al.*, 2007). The United States Environmental Protection Agency (US EPA) authorizes use of pecan shell fiber as an inert ingredient in granulated pesticides (EPA, 2015).

The American Association of Feed Control Officials (AAFCO)[3] includes Ground Pecan Shells (definition 60.110) in the 2010 and later official publication of feed ingredients (AAFCO, 2015). In 2008, a letter from the director of the FDA Center for Veterinary Medicine to a pet food company stated that "safety, utility and manufacturing data in support of the use of ground pecan shell as a source of dietary fiber in animal feed have been satisfactorily addressed in previous submissions" and stated that they would not anticipate that they would initiate action against the

[3] The Food and Drug Administration (FDA) currently recognizes feed ingredients that have definitions in the Official Publication of AAFCO.

ingredient or feed product containing ground pecan shell (FDA, 2008). To our knowledge, no adverse events have been reported about use of ground pecan shell in animal feed.

### 2.4.Proposed use or uses

The proposed uses of the ingredient are as a source of fiber and antioxidants for certain foods, at amounts ranging from 2-10%. The ingredient is intended to be used solely for purposes other than coloring, and any color imparted is unimportant insofar as the appearance, value, marketability, or consumer acceptability is concerned.

## 3. MANUFACTURING PROCESS, DESCRIPTION AND SPECIFICATIONS

### 3.1.Manufacturing process

Pecan shell fiber is a co-product of the manufacturing process to produce pecan meat for human consumption. Pecan shell fiber is produced entirely from ground shells of pecan nuts according to current Good Manufacturing Practice (cGMP), with no additional substances added during processing. Pecans in the shell (before cracking; also known as "inshell" pecans) are washed in an ambient temperature water bath (estimated [redacted] water/pecan shell ratio) for [redacted] minutes to remove any organic material. Pecans in the wash water float on the surface and are tumbled over (forced underwater by paddles) to clean the entire shell. The inshell pecans are then submerged in hot water for sanitation. This sanitation process has been validated as effective when conducted at [redacted] for at least [redacted]. Typically this process runs at [redacted] for [redacted] minutes, well above the validated process time and temperature required for adequate sanitation.

After the shells are cracked, the pecan meat is separated from the shells using a system of blowers, vacuum aspirators, water baths, driers and electronic shell sorters by a continuous process. The time in each process is standard. The drying temperature for shells is kept around [redacted] (air temperature) for approximately [redacted]. The shells are transferred pneumatically to the shell grinding facility where they are inspected, passed through magnets and screenings to remove unwanted material (including the reddish-brown inner membranes or "packing material")[4], and ground into pecan shell fiber. Pecan shell fiber is subjected to air separation and several passes over a magnet during the grinding process. Fine particles are pulled upward and captured, leaving heavier, larger particles behind. Temperature inside the grinding mills is controlled to yield a final moisture content within specification. The final pecan shell fiber product is packaged into 50 lb. multiple layer paper (kraft) bags with polypropylene liners, or polyethylene lined "super sacks" or drums (variety of sizes). Packaging for pecan shell fiber will be labeled "May Contain Pecans".

### 3.2. Description and Specifications

The physical and chemical properties and specifications for pecan shell fiber are provided in Table 1 and Table 2 respectively. Pecan shell fiber is a fine, reddish brown powder with no odor. Pecan shell fiber is mainly comprised of insoluble fiber and as such, is poorly soluble in water. The ingredient is stable for up to one year when stored at room temperature in its original, unopened packaging.

[4] Personal communication from Southeastern Reduction Company, November 1, 2012.

**Table 1. Physical and chemical properties of pecan shell fiber**

| Characteristic | Value |
|---|---|
| Source | *Carya illinoensis* |
| Appearance | Fine, reddish brown powder |
| Odor | No characteristic odor |
| Total dietary fiber | 80-85% |
| Insoluble fiber | 78-83% |
| Stability | Up to one year when stored at ambient temperature in unopened containers |

The specifications of pecan shell fiber are shown in Table 2. Analyses from five nonconsecutive lots confirm that pecan shell fiber can consistently meet specifications. The primary constituents of the ingredient are cellulose and lignin (53-71% of the ingredient), collectively measured as acid detergent fiber (ADF). Hemicellulose is also present, and is included with cellulose and lignin in the neutral detergent fiber (NDF) measurement (68-78% of the ingredient). Pecan shell fiber also contains polyphenols (3-9%) and proanthocyanidins (8-18%), as well as small amounts of protein (<3%), fat (<4.5%) and ash (<3%). The moisture content is 5-10%. The microbial and heavy metals specifications and analyses for pecan shell fiber show a low potential for fungal, bacterial or heavy metal contamination. The fact that microbial counts in the ingredient are relatively low is consistent with extracts of pecan shells exhibiting antimicrobial properties (Van Loo *et al.*, 2012; Babu D. *et al.*, 2013). The total amount of aflatoxins B1, B2, G1, and G2 in the ingredient is < 5 ppb[5], the limit of detection of the assay, under the FDA action level of 20 ppb aflatoxin (FDA, 2000). Levels of commonly measured pesticides and herbicides and the antifungal metconazole in two representative lots of pecan shell fiber are beneath limits of detection, with the exception of permethrin and piperonyl butoxide, which were present at 0.03 ppm and 0.02 ppm (respectively) in the two lots (APPENDIX I).

### 3.3.Stability

Pecan shell fiber is stable for twelve months at ambient temperature and humidity (25°C/60%) and accelerated temperature and humidity (40°C/75%), when stored in sealed containers, for all parameters measured except polyphenols (PPh) and proanthocyanidins (PAC), which decrease in concentration following the six month measurements for both ambient and accelerated conditions (Table 3 and Table 4).

[Remainder of this page is blank]

[5] ppb = parts *per* billion, or µg/kg.

**Table 2. Specifications of pecan shell fiber**

| Analysis | Method | Specification | Batch Analysis Results (N=5) Range | Average |
|---|---|---|---|---|
| **Physical/Chemical** | | | | |
| Appearance | Visual | Reddish brown powder | Meets specification | Meets specification |
| US 100 mesh screen (thru) (%) | SRC Wet Screen | ≥ 98 | 100* | 100 |
| US 200 mesh screen (thru) (%) | SRC Wet Screen | ≥ 90 | 95-97 | 96.6 |
| US 325 mesh screen (thru) (%) | SRC Wet Screen | ≥ 75 | 88-91 | 90.0 |
| Moisture content (wt %) | Forced Draft AOAC 930.15 | 5-10 | 6.19-6.61 | 6.42 |
| Fiber, Acid Detergent (%) (Cellulose + Lignin) | Ankom ADF 05/03 or equivalent | 53-75 | 64.6-67.4 | 66.4 |
| Fiber, Neutral Detergent (%) (Cellulose + Lignin + Hemicellulose) | Ankom NDF | 65-78 | 70.9-74.4 | 72.4 |
| Protein (g/100 g) | AOAC 990.03 or 992.15 | < 3 | 1.94-2.74 | 2.26 |
| Fat (g/100 g) | Acid Hydrolysis | < 4.5 | 1.64-3.8 | 3.17 |
| Ash (g/100 g) | AOAC 942.05 | < 3 | 1.90- 2.18 | 1.98 |
| Calories (*per* 100 g) | Atwater calculation | 350-400 | 374-386 | 381.6 |
| Polyphenols (% dry basis)** | Singleton *et al.* (1999) | 3-9 | 3.81 – 8.22 | 5.4 |
| Proanthocyanidins (% dry basis) | USP 29/NF24 | 8-18 | 8.14 – 17.1 | 11.52 |
| **Heavy metals** | | | | |
| Arsenic (ppm) | Modified AOAC 986.15 | < 0.5 | < 0.02* | < 0.02 |
| Cadmium (ppm) | Modified AOAC 986.15 or EPA 3050/6020 USP730 | < 0.5 | 0.021 – 0.025 | 0.023 |
| Lead (ppm) | Modified AOAC 986.15 | < 0.5 | < 0.02 – 0.07 | 0.03 |
| Mercury (ppm) | Modified AOAC 986.15 or EPA 3050/6020 USP730 | < 0.5 | < 0.01* | < 0.01 |
| **Microbiological** | | | | |
| Total Aerobic (cfu/g) | AOAC 966.23 | < 10,000 | <10 - 2750 | 1152 |
| Coliforms (cfu/g) | AOAC 991.14 | < 100 | <10* | < 10 |
| *Escherichia coli* (cfu/g) | AOAC 991.14 | < 10 | <10* | < 10 |
| *Salmonella* spp. (*per* 25 g) | AOAC-Rl-100201 | Negative | Negative | Negative |
| Yeast and mold (cfu/g) | FDA-BAM, 7th Ed. | < 100 | 10-80 | 46 |
| Sum of Aflatoxins B1, B2, G1, G2 (ppb) | Modified AOAC 991.31 | < 5 | < 5* | <5 |

*N* = 9 for polyphenols and proanthocyanidins. * all samples, ** Gallic aid equivalents. The protocol for particle sizing (Wet Screen Method) is shown in APPENDIX 2.
ppb = parts per billion; ppm = parts per million

**Table 3. Stability of pecan shell fiber at room temperature and humidity**

| Lot Number | Time point (mos) | Appearance | Odor | Moisture (%) | ADF (%) | NDF (%) | PPh (%) | PAC (%) | TPC (CFU/g) | Coliforms/ E coli CFU/g) | Salm | Yeast (CFU/g) | Mold (CFU/g) | AFL (ppb) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| [redacted] | Baseline | RB | None | 5.72 | 63.6 | 71.8 | 4.65 | 10.50 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 5.85 | 68 | 71.7 | 4.13 | 10.20 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 6 | RB | None | 6.03 | 68.7 | 72.8 | 4.49 | 10.90 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 10 | RB | None | 6.04 | 61.4 | 70.6 | 3.15 | 5.69 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 6.73 | 67.4 | 70.7 | 3.03 | 5.42 | 50 | <10* | Neg | <10 | 10 | <5 |
| [redacted] | Baseline | RB | None | 6.00 | 56.6 | 72.3 | 4.24 | 10.70 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 3 | RB | None | 6.20 | 69.2 | 72.1 | 3.70 | 10.60 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 6.29 | 67.9 | 69.2 | 3.87 | 11.00 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 5.83 | 63.9 | 71 | 2.54 | 4.03 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 6.91 | 68.1 | 70.7 | 2.63 | 4.61 | 20 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 5.41 | 53.2 | 75.2 | 3.81 | 8.73 | 30 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 5.79 | 68.2 | 75 | 3.52 | 8.65 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 5.60 | 67.2 | 73.7 | 3.87 | 9.61 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 5.17 | 61.4 | 74.1 | 2.88 | 5.25 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 6.87 | 68.5 | 71.9 | 2.47 | 4.76 | <10 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 5.62 | 69 | 74.2 | 4.10 | 8.97 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 5.89 | 68 | 73.1 | 4.03 | 9.29 | 10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 5.86 | 68.8 | 71.8 | 4.37 | 10.40 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 6.57 | 62.7 | 72.5 | 2.16 | 3.36 | 10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 6.85 | 69.5 | 72.8 | 2.61 | 4.61 | <10 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 5.83 | 69.5 | 74.6 | 3.89 | 8.14 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 5.93 | 66.3 | 72.4 | 3.61 | 9.04 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 5.92 | 66.4 | 72.6 | 3.98 | 10.40 | 10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 5.70 | 62.3 | 75.5 | 2.58 | 4.62 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 6.88 | 66.1 | 73.1 | 2.35 | 4.68 | <10 | <10* | Neg | <10 | <10 | <5 |

* < 10 for both analyses; Values for PPh and PAC are reported on a dry matter basis. ADF = acid detergent fiber; AFL = Aflatoxins B1+B2+G1+G2; CFU = colony forming units; mos = months; NDF = neutral detergent fiber; Neg = negative; PAC = proanthocyanidins; ppb = parts *per* billion; PPh = Polyphenols; RB = reddish brown; Salm = *Salmonella per* 25 g; TPC = total plate count. Methods of analysis: Moisture: Forced Draft AOAC 930.15, ADF: Ankom ADF 05/03, NDF: Ankom NDF, PPh: Singleton *et al.* (1999); PAC: USP 29/NF24, Coliforms/*E. coli*: AOAC 991.14, *Salmonella:* AOAC-RI-100201, Yeast/Mold: FDA-BAM, 7th Ed., AFL: Modified AOAC 991.31.

**Table 4. Stability of pecan shell fiber at accelerated temperature and humidity**

| Lot Number | Time point (mos) | Appearance | Odor | Moisture (%) | ADF (%) | NDF (%) | PPh (%) | PAC (%) | TPC (CFU/g) | Coliforms/ *E coli* (CFU/g) | Salm | Yeast (CFU/g) | Mold (CFU/g) | AFL (ppb) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| [redacted] | Baseline | RB | None | 5.72 | 63.6 | 71.8 | 4.65 | 10.50 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 6.65 | 62.2 | 71.4 | 3.81 | 9.78 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 7.53 | 65.6 | 69.3 | 4.00 | 10.30 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 8.38 | 59.8 | 69.6 | 2.06 | 3.00 | 10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 8.61 | 65.3 | 69 | 2.47 | 4.04 | <10 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 6.00 | 56.6 | 72.3 | 4.24 | 10.70 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 3 | RB | None | 7.13 | 55.3 | 71.5 | 3.66 | 8.79 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 6.78 | 68.1 | 71.4 | 3.65 | 10.10 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 7.48 | 63 | 69.7 | 2.83 | 5.27 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 6.95 | 70.6 | 71.1 | 2.11 | 3.18 | <10 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 5.41 | 53.2 | 75.2 | 3.81 | 8.73 | 30 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 6.35 | 67.2 | 74.5 | 3.25 | 8.09 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 7.30 | 63.2 | 72.9 | 3.47 | 8.59 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 10 | RB | None | 8.60 | 69.1 | 68.8 | 2.32 | 3.61 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 9.20 | 60.8 | 75.2 | 1.91 | 2.93 | 10 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 5.62 | 69 | 74.2 | 4.10 | 8.97 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 7.01 | 63 | 72.8 | 3.48 | 8.40 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 6 | RB | None | 7.09 | 67.5 | 71.3 | 3.58 | 9.34 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 6.90 | 62.1 | 72.2 | 2.79 | 4.71 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 9.86 | 62.9 | 70.5 | 2.04 | 2.81 | <10 | <10* | Neg | <10 | <10 | <5 |
| [redacted] | Baseline | RB | None | 5.83 | 69.5 | 74.6 | 3.89 | 8.14 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 3 | RB | None | 6.86 | 63.6 | 72.8 | 3.50 | 8.74 | <10 | <10* | Neg | <10 | 10 | <5 |
| | 6 | RB | None | 7.40 | 67.5 | 70.9 | 3.54 | 8.45 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 10 | RB | None | 7.79 | 59.7 | 73.2 | 2.30 | 3.65 | <10 | <10* | Neg | <10 | <10 | <5 |
| | 12 | RB | None | 9.03 | 64.5 | 71 | 2.26 | 3.04 | <10 | <10* | Neg | <10 | <10 | <5 |

* < 10 for both analyses; Values for PPh and PAC are reported on a dry matter basis. ADF = acid detergent fiber; AFL = Aflatoxins B1+ B2,+G1,+G2; CFU = colony forming units; mos = months; NDF = neutral detergent fiber; Neg = negative; PAC = proanthocyanidins; ppb = parts *per* billion; PPh = Polyphenols; RB = reddish brown; Salm = *Salmonella* per 25 g; TPC = total plate count. Methods of analysis: Moisture: Forced Draft AOAC 930.15, ADF: Ankom ADF 05/03, NDF: Ankom NDF, ,PPh: : Singleton *et al.* (1999); PAC: USP 29/NF24, Coliforms/*E. coli*: AOAC 991.14, *Salmonella*: AOAC-RI-100201, Yeast/Mold: FDA-BAM, 7th Ed., AFL: Modified AOAC 991.31.

## 4. ESTIMATED DAILY INTAKE

The intake profile (amount and frequency) by individuals in USDA's What We Eat in America (WWEIA) Continuing Survey of Food Intakes by Individuals 2011-2012 (CDC, 2015) was used to calculate the estimated daily intake (EDI) of pecan shell fiber for individuals consuming the food groups selected for the addition of pecan shell fiber *per* this GRAS evaluation. The food groups as defined by 21 CFR §170.3(n) are provided in Table 5. The levels of use of pecan shell fiber in foods are self-limiting as undesirable taste characteristics develop when recommended levels are exceeded.

**Table 5. Food groups selected for pecan shell fiber supplementation***

| Food Category | Intended use level (%) |
|---|---|
| Baked goods and baking mixes | 2-5 |
| Breakfast cereals | 5 |
| Confections and frostings | 5 |
| Gelatins, puddings and fillings | 5 |
| Grain products and pastas | 2 |
| Meal replacement | 10 |
| Snack food | 2 |
| Soft candy | 10 |
| Sweet sauces | 5 |

*The food categories correspond to those listed in 21 CFR 170.3(n), with the exception of meal replacement. Certain categories may contain foods for which a standard of identity exists, which prohibit the addition of ingredients to the food not identified as mandated or optional ingredients under the regulation. Therefore, addition of pecan shell fiber to a food for which a standard of identity exists would demand that the food product be named other than that as indicated under the standard of identity or a waiver of that standard be obtained.

The means and 90$^{th}$ percentile EDIs were calculated for: (1) current pecan shell fiber intake from natural sources (current); (2) pecan shell fiber intake following addition of pecan shell fiber to the selected food groups (added) and; (3) total estimated EDI from natural sources combined with levels from addition to the foods (total).

Pecan shell fiber is currently permitted for use as a flavor ingredient in meat products (Burdock, 2010). Possible daily intake from this use is 156.8 mg/day (Burdock, 2010). Using this value as the mean for current consumption, the 90$^{th}$ percentile current EDI of pecan shell fiber is estimated by assuming two times greater consumption than the reported mean current EDI. Thus, the estimated 90$^{th}$ percentile current EDI is 313.6 mg pecan shell fiber *per* day (5.2 mg/kg bw/day).

If pecan shell fiber is added to the selected foods at the levels specified in Table 5, the added mean and 90$^{th}$ percentile pecan shell fiber consumption is 5,020.6 mg/day (83.7 mg/kg bw/day) and 9,920.0 mg/day (165.3 mg/kg bw/day), respectively.

Combining the current and added intake levels gives the total pecan shell fiber consumption level. The estimated total mean and 90$^{th}$ percentile consumption of pecan shell fiber, if pecan shell fiber were added to the selected foods at the levels specified in Table 5, is 5,177.4 mg/day (86.3 mg/kg bw/day based on a 60 kg bw[6]) and 10,233.6 mg/day (170.6 mg/kg bw/day), respectively

[6] bw = body weight

(Table 6). This is a conservative estimate of intake, as it is unlikely that people will consume all of the pecan shell fiber-containing products at the 90th percentile levels of intake.

**Table 6. Pecan shell fiber: current intake, predicted intake following supplementation of selected foods at the indicated levels (Figure 4) and total intake (predicted + current) for individuals consuming selected supplemented foods**

| | *Per* User (mg/day) | |
|---|---|---|
| **Pecan shell fiber intake from:** | **Mean** | **90th Percentile** |
| Current consumption from food | 156.8 | 313.6 |
| Possible maximum consumption with pecan shell fiber as an added ingredient to food | 5,020.6 | 9,920.0 |
| Total from conventional food (current + added) | 5,177.4 | 10,233.6 |

As mentioned in Section 3.1, 0.02 ppm permethrin and 0.02 ppm piperonyl butoxide were detected in pecan shell fiber. Based on an estimated 90th percentile consumption of 170.6 mg/kg bw/day of pecan shell powder, approximately 3 ng/kg of permethrin and piperonyl butoxide could be consumed by 90th percentile consumers of pecan shell fiber. The intakes of these pesticides from pecan shell fiber are considerably lower than the acceptable daily intake (ADI) of 0.05 mg/kg bw permethrin and 0.2 mg/kg bw piperonyl butoxide (FAO/WHO, 1999; 2013), and as such will not constitute a hazard.

## 5. ABSORPTION, DISTRIBUTION, METABOLISM, AND ELIMINATION (ADME)

No information on the ADME of pecan shell fiber was identified in the literature search. Pecan shell fiber primarily consists of cellulose, hemicellulose and lignin. The cellulose, hemicellulose and lignin components are resistant to digestion by enzymes present in the human stomach and will pass through the stomach along with indigestible fiber from other sources. Studies in humans have shown that hemicellulose and cellulose (but not lignin) may be broken down in the human intestine (Holloway *et al.*, 1978; Slavin *et al.*, 1981; Cummings, 1984a; Slavin, 2013). It has been hypothesized that this hydrolysis is accomplished *via* fermentation by cellulolytic bacteria (Cummings, 1984b; Wedekind *et al.*, 1988). Products of microbial fermentation of fiber include carbon dioxide, methane, hydrogen and short-chain fatty acids (primarily acetate, propionate and butyrate) (IOM, 2005), the latter of which can be used as an energy source. Because the process of fermentation is anaerobic, less energy is recovered from fiber than carbohydrate. It is generally thought that the amount of energy recovered from fiber is 1.5 to 2.5 kcal/g (IOM, 2005). The small amounts of protein and fat in the ingredient will also be digested along with protein and fat from other foods and will provide energy. Proanthocyanidins are known to be poorly absorbed in the gut due to their polymeric nature and high molecular weight (Choy and Waterhouse, 2014).

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## 6. SAFETY EVALUATION

### 6.1. Acute Studies

The maximum tolerated oral (gavage) dose of pecan shell fiber in male and female NMRI mice is 10 g/kg bw, administered in three split doses of 333 mg/kg bw suspended homogeneously in cottonseed oil, with two hours between each dose (Dolan *et al.*, 2016). No deaths occurred in the study, which was terminated 68 hours after the last dose. A mild reduction of spontaneous activity and/or half eyelid closure were observed in the male group 30 minutes and one hour after the last application of test material, which abated by two hours.

### 6.2. Subchronic Studies

Results of a study conducted in accordance with OECD Guideline No. 408[7] indicate that the 90-day dietary no observable adverse effect level (NOAEL) for pecan shell fiber in Sprague-Dawley rats is 150,000 ppm[8] (9,947.5 mg/kg bw/day in males and 11,082.8 mg/kg bw/day in females), the highest dose administered (Dolan *et al.*, 2016). All animals exposed to pecan shell fiber survived to scheduled termination. Results of functional observational, forelimb/hindlimb grip strength, hindlimb foot splay and motor activity assessments of test animals were comparable to controls and there were no adverse clinical signs attributed to administration of the test material. Results of ophthalmologic examinations of animals were normal, except for one female that had an abnormal eye exam at the beginning and end of the study. Mean body weights (Figure 3 and Figure 4) and body weight gain (data not shown) of animals receiving pecan shell fiber were similar to controls, with the exception of a statistically significant increase in body weight gain of females receiving 100,000 ppm test material (the mid-dose group) from Days 56-63 ($p < 0.05$).

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[7]OECD Guidelines for the Testing of Chemicals, Section 4. Test No. 408. Repeated Dose 90-Day Oral Toxicity Study in Rodents. Available at http://www.oecd-ilibrary.org/environment/test-no-408-repeated-dose-90-day-oral-toxicity-study-in-rodents_9789264070707-en; site last visited December 21, 2015.

[8] The highest dose was equivalent to 15% of the diet.

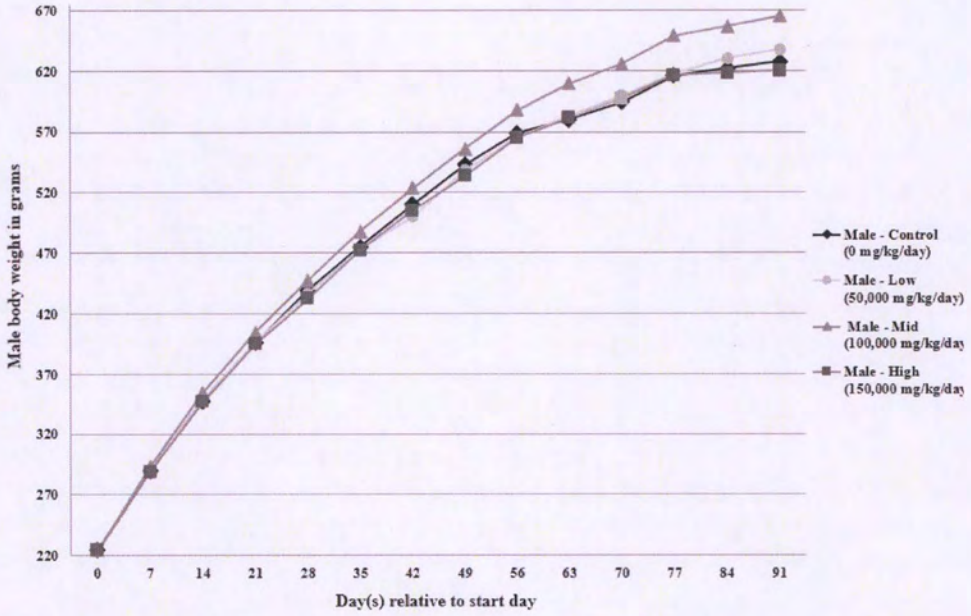


**Figure 3. Mean body weights of male rats during the study. Standard deviations, which tended to be large for treated animals, are omitted for figure clarity.**

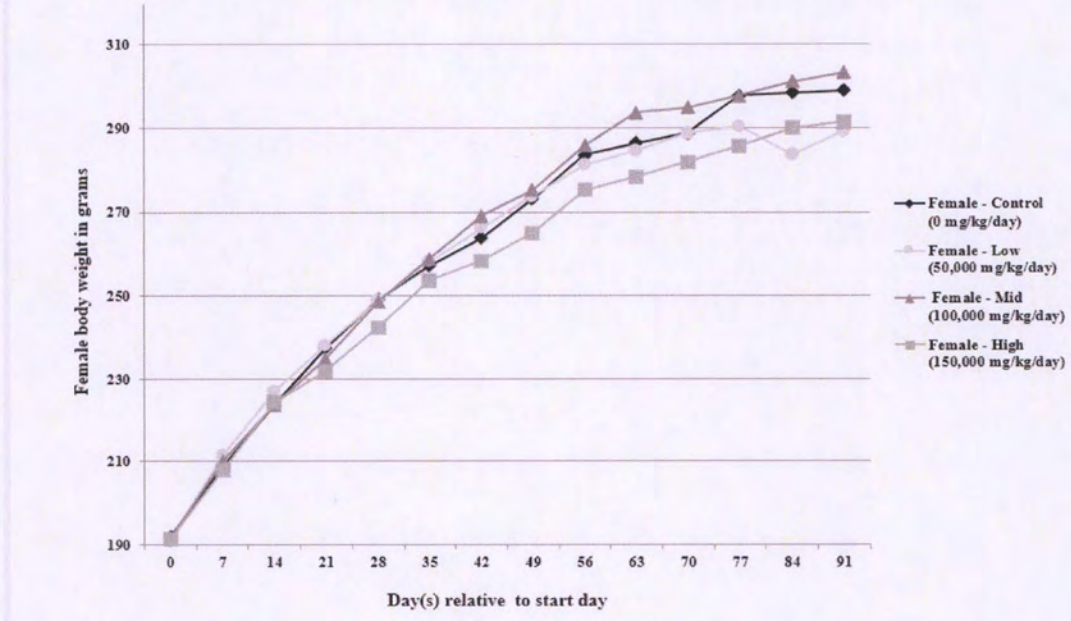


**Figure 4. Mean body weights of female rats during the study. Standard deviations, which tended to be large for treated animals, are omitted for figure clarity.**

Statistically significant increases ($p < 0.01$-$0.05$) in food consumption were reported on Days 63-70 and 77-91 in 100,000 ppm males and on Days 0-7, 63-84, and overall (Days 0-91) in 150,000 ppm males (Table 7). Feed efficiency decreased in males receiving 150,000 ppm on Days 0-7, 21-28, and overall (Days 0-91) ($p < 0.01$), but was not affected at lower concentrations. Overall (Days 0-91) and mean daily food consumption for female rats exposed to the test substance were comparable with control values throughout the study, and there was no effect of pecan shell fiber on food efficiency of females.

**Table 7. Mean food consumption of male and female rats during the study**

| Day(s) Relative to Start Date | Control | 50 000 mg/kg/day | 100 000 mg/kg/day | 150 000 mg/kg/day |
|---|---|---|---|---|
| **Number of Males** | **9** | **10** | **10** | **10** |
| 0→7 | 24.86±2.43 | 26.26±2.85 | 27.59±2.62 | 28.21±2.47 * |
| 7→14 | 27.19±3.11 | 28.60±4.15 | 30.20±2.69 | 30.89±3.49 |
| 14→21 | 28.11±2.86 | 29.80±4.51 | 30.66±3.32 | 32.10±3.24 |
| 21→28 | 28.76±2.95 | 29.84±4.67 | 31.20±3.53 | 32.30±2.95 |
| 28→35 | 29.17±2.52 | 29.94±4.82 | 31.99±3.56 | 32.94±3.31 |
| 35→42 | 30.16±2.76 | 30.91±5.05 | 32.86±4.22 | 34.17±4.32 |
| 42→49 | 30.34±2.76 | 30.14±6.88 | 33.13±3.74 | 34.10±3.67 |
| 49→56 | 30.77±2.58 | 30.74±5.29 | 33.53±3.59 | 34.87±3.84 |
| 56→63 | 29.97±1.49 | 29.21±5.57 | 33.46±3.63 | 33.09±3.56 |
| 63→70 | 28.24±1.73 | 29.36±4.98 | 32.61±3.51 * | 33.27±3.28 * |
| 70→77 | 29.48±2.13 | 28.33±3.66 | 32.30±4.03 | 33.57±3.26 * |
| 77→84 | 27.21±1.71 | 29.20±4.52 | 31.54±3.24 * | 31.61±3.34 * |
| 84→91 | 24.63±1.91 | 27.21±4.76 | 30.61±5.94 ** | 27.17±2.78 |
| 0→91 | 28.04±1.46 | 29.20±4.31 | 31.67±3.24 | 32.18±3.03 * |
| **Number of Females** | **10** | **10** | **10** | **10** |
| 0→7 | 17.23±1.77 | 17.94±2.03 | 18.70±1.95 | 18.50±2.52 |
| 7→14 | 18.66±3.08 | 17.83±2.93 | 19.81±4.14 | 19.79±3.09 |
| 14→21 | 19.83±3.79 | 18.24±2.91 | 20.84±4.87 | 20.06±3.63 |
| 21→28 | 19.71±3.71 | 18.34±3.61 | 20.53±3.68 | 19.44±3.25 |
| 28→35 | 18.60±3.63 | 18.44±3.71 | 19.96±4.11 | 19.66±2.68 |
| 35→42 | 19.04±3.29 | 18.74±3.14 | 19.01±3.63 | 19.07±2.14 |
| 42→49 | 17.91±3.09 | 17.17±2.71 | 20.31±4.61 | 18.84±3.50 |
| 49→56 | 17.86±2.64 | 17.43±2.19 | 20.29±3.58 | 19.06±3.13 |
| 56→63 | 18.84±3.72 | 18.14±2.28 | 21.50±4.45 | 19.33±2.34 |
| 63→70 | 20.59±7.47 | 17.93±3.50 | 19.80±3.68 | 18.33±2.89 |
| 70→77 | 18.43±3.90 | 16.49±2.18 | 18.46±3.68 | 18.37±4.59 |
| 77→84 | 17.49±3.30 | 15.96±6.19 | 20.21±5.06 | 18.66±3.62 |
| 84→91 | 14.99±1.97 | 15.50±2.87 | 17.51±2.87 | 16.94±3.59 |
| 0→91 | 18.40±2.60 | 17.55±2.47 | 19.76±3.30 | 18.93±2.71 |

Data are presented as mean ± standard deviation (SD). *Significantly different from control by Dunnett's test (2 Sided), $p < 0.05$; ** Significantly different from control by Dunn's test (2 Sided), $p < 0.01$.

Dietary exposure to pecan shell fiber did not induce any toxicologically significant changes in hematology or coagulation parameters (Table 8). While values for mean corpuscular hemoglobin (MCH), mean corpuscular hemoglobin concentration (MCHC) and mean corpuscular volume (MCV) were lower than control and for red blood cell distribution width (RDW) were higher than control in high dose males ($p<0.05$), they were within laboratory historical ranges for Sprague-Dawley rats (15.3-20.7 pg MCH, 30.1-35.3 g/dL MCHC, 47.5-68.6 fL MCV and 11.4-31.2% RDW). An increase in the absolute number of basophils was observed in females administered the high dose; however, the value was within the historical control range (0-0.7 103/µL), and in light of the absence of changes in other white blood cells, this change was considered to be of no toxicological significance. Other changes that occurred in low or medium dose animals (decreased platelet counts in mid-dose males, increased MCH and MCV in mid-dose females and decreased RDW in low dose females ($p<0.05$)) are toxicologically insignificant because they did not occur in dose dependent manner and are within laboratory historical ranges for control animals.

There was no effect of pecan shell fiber on urinalysis. Minor differences in some clinical chemistry parameters (Table 9) occurred (decreased bilirubin (BILI) in high dose males, increased cholesterol (CHOL) in high dose females and decreased triglycerides (TG) in mid dose females, $p<0.05$). The changes in BILI and CHOL are likely incidental, because the values are within historical values (0.10-0.27 mg/dL BILI, 34-145 mg/dL CHOL) and the changes are small in magnitude and not accompanied by any other corresponding clinical or histopathological change. Although the TG values for all groups of exposed females were reduced by 22-29%, the only group that differed significantly from control was the TG value for low dose females. An increase in TG, rather than a decrease, is considered an adverse effect.

There were no statistically significant changes in absolute or relative organ weights of females or absolute organ weights of males (Table 10). Decreases in spleen and liver relative to body weight (Table 10) or spleen to brain weight (data not shown) were observed in high dose males. Values were within historical control values (0.87-4.12 and 0.21-1.12 for spleen to body and brain weight and 14.62-35.04 for liver to body weight) and corresponding absolute organ weight changes or histopathological correlates did not occur; thus the statistical changes are interpreted to be of no toxicological relevance.

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**Table 8. Hematology and coagulation data for rats administered pecan shell fiber for 91 days**

| Parameter | Control | 50 000 mg/kg/day | 100 000 mg/kg/day | 150 000 mg/kg/day |
|---|---|---|---|---|
| **Number of Males** | **9** | **10** | **10** | **10** |
| RBC ($10^6$/µL) | 8.43±0.40 | 8.53±0.31 | 8.70±0.43 | 8.73±0.40 |
| HGB (g/dL) | 15.4±0.6 | 15.1±0.3 | 15.4±0.5 | 15.0±0.2 |
| HCT (%) | 45.8±2.0 | 45.5±1.1 | 46.0±1.8 | 45.4±1.0 |
| MCH (pg) | 18.2 ±0.5 | 17.7±0.6 | 17.7±0.4 | 17.2±0.7** |
| MCHC (g/dL) | 33.6±0.4 | 33.2±0.5 | 33.4±0.3 | 33.1±0.4* |
| MCV (fL) | 54.3±1.2 | 53.3±1.3 | 52.9±1.1 | 52.1±1.6* |
| RET ($10^3$/ µL) | 182.4±26.6 | 203.1±52.0 | 190.2±35.3 | 173.3±26.4 |
| PLT ($10^3$/ µL) | 1128±147 | 1011±138 | 963±139* | 1088±125 |
| RDW (%) | 12.7±0.4 | 13.2±0.9 | 13.5±0.4 | 14.3±0.8* |
| WBC ($10^3$/ µL) | 13.47±3.65 | 12.54±2.29 | 13.71±3.53 | 11.69±2.08 |
| LYM ($10^3$/ µL) | 9.74±2.76 | 9.68±2.05 | 10.37±3.20 | 8.82±1.94 |
| MON ($10^3$/ µL) | 0.38±0.15 | 0.34±0.10 | 0.36±0.09 | 0.37±0.10 |
| NEU ($10^3$/ µL) | 2.98±2.47 | 2.15±1.15 | 2.50±0.57 | 2.18±0.98 |
| EOS ($10^3$/ µL) | 0.23±0.03 | 0.23±0.05 | 0.29±0.09 | 0.20±0.06 |
| BAS ($10^3$/ µL) | 0.07±0.03 | 0.06±0.03 | 0.09±0.05 | 0.05±0.03 |
| LUC ($10^3$/ µL) | 0.08±0.03 | 0.07±0.03 | 0.08±0.04 | 0.06±0.03 |
| PT (sec) | 10.6±0.3 | 10.6±0.3 | 10.8±0.3 | 10.9±0.3 |
| APTT (sec) | 16.8±1.7 | 16.8±1.6 | 18.9±2.8 | 18.2±1.4 |
| **Number of Females** | **10** | **10** | **10** | **10** |
| RBC ($10^6$/ µL) | 8.35±0.23 | 8.22±0.41 | 8.18±0.33 | 8.19±0.23 |
| HGB (g/dL) | 15.1±0.6 | 15.1±0.8 | 15.2±0.5 | 15.2±0.5 |
| HCT (%) | 45.1±1.7 | 44.3±2.3 | 45.6±1.8 | 45.2±1.5 |
| MCH (pg) | 18.1±0.3 | 18.4±0.4 | 18.6±0.5* | 18.5±0.6 |
| MCHC (g/dL) | 33.5±0.4 | 34.1±0.6 | 33.4±0.4 | 33.6±0.2 |
| MCV (fL) | 54.1±1.1 | 53.9±1.5 | 55.8±1.5* | 55.1±1.8 |
| RET ($10^3$/ µL) | 147.9±29.5 | 122.1±29.4 | 155.5±26.9 | 173.8±38.2 |
| PLT ($10^3$/ µL) | 979±173 | 957±160 | 1108±96 | 979±143 |
| RDW (%) | 12.0±0.1 | 11.7±0.4** | 11.8±0.3 | 12.0±0.3 |
| WBC ($10^3$/ µL) | 6.89±2.36 | 9.34±3.41 | 7.74±2.25 | 8.72±2.39 |
| LYM ($10^3$/ µL) | 5.48±1.84 | 7.07±2.52 | 5.95±1.70 | 7.18±2.24 |
| MON ($10^3$/ µL) | 0.18±0.09 | 0.28±0.18 | 0.21±0.09 | 0.19±0.06 |
| NEU ($10^3$/ µL) | 1.04±0.49 | 1.77±1.07 | 1.35±0.58 | 1.13±0.49 |
| EOS ($10^3$/ µL) | 0.13±0.05 | 0.14±0.04 | 0.16±0.04 | 0.14±0.04 |
| BAS ($10^3$/ µL) | 0.02±0.01 | 0.03±0.02 | 0.03±0.01 | 0.03±0.02* |
| LUC ($10^3$/ µL) | 0.04±0.02 | 0.05±0.02 | 0.05±0.02 | 0.05±0.03 |
| PT (sec) | 10.2±0.2 | 10.2±0.2 | 10.2±0.2 | 10.2±0.2 |
| APTT (sec) | 17.3±1.6 | 16.5±1.7 | 16.8±1.3 | 16.4±1.6 |

Data are presented as mean ± standard deviation (SD). * Significantly different from control by Dunnett's test (2 sided), $p < 0.05$; ** Significantly different from control by Dunnett's test (non-parametric, 2 sided), $p < 0.05$. APTT = activated partial thromboplastin time; BAS = basophils; dL = deciliter; EOS = eosinophils; fL = femtoliter; g = grams; HCT = hematocrit; HGB = hemoglobin; L = liter; LUC = large unstained cells; LYM = lymphocytes; MCH = mean corpuscular hemoglobin; MCHC = mean corpuscular hemoglobin concentration; MCV = mean corpuscular volume; $mm^3$ = cubic millimeter; MON = monocytes; NEU = neutrophils; pg = picograms; PLT = platelets; PT = prothrombin time; RBC = erythrocytes; RDW = red blood cell distribution width; RET= reticulocytes; sec = seconds; WBC = white blood cells (leukocytes).

**Table 9. Clinical chemistry data for rats administered pecan shell fiber for 91 days**

| Parameter | Control | 50 000 mg/kg/day | 100 000 mg/kg/day | 150 000 mg/kg/day |
|---|---|---|---|---|
| **Number of Males** | **9** | **10** | **10** | **10** |
| AST (U/L)* | 82±10 | 85±12 | 89±20 | 80±5 |
| ALT (U/L) | 27±13 | 25±9 | 25±6 | 25±4 |
| SDH (U/L)* | 7.7±3.8 | 8.5±1.5 | 6.3±2.3 | 8.7±2.1 |
| ALKP (U/L) | 90±19 | 83±15 | 86±22 | 81±13 |
| BILI (mg/dL) | 0.16±0.03 | 0.17±0.02 | 0.15±0.03 | 0.13±0.02** |
| BUN (mg/dL) | 11±2 | 13±2 | 12±1 | 10±2 |
| CREA (mg/dL) | 0.32±0.04 | 0.30±0.03 | 0.31±0.03 | 0.30±0.04 |
| CHOL (mg/dL) | 75±13 | 94±38 | 97±24 | 81±20 |
| TRIG (mg/dL) | 82±28 | 82±32 | 96±45 | 79±28 |
| GLUC (mg/dL) | 123±12 | 129±19 | 136±16 | 127±15 |
| TP (g/dL) | 6.4±0.3 | 6.5±0.4 | 6.4±0.3 | 6.3±0.1 |
| ALB (g/dL) | 3.2±0.1 | 3.2±0.1 | 3.1±0.1 | 3.1±0.1 |
| GLOB (g/dL) | 3.2±0.3 | 3.3±0.3 | 3.3±0.2 | 3.2±0.2 |
| CALC (mg/dL) | 10.1±0.3 | 10.1±0.5 | 10.3±0.5 | 10.0±0.2 |
| IPHS (mg/dL)* | 6.6±0.4 | 6.4±0.7 | 6.4±0.5 | 6.2±0.5 |
| Na (mmol/L) | 146.4±5.8 | 145.0±6.7 | 148.1±14.0 | 142.6±5.1 |
| K (mmol/L) | 5.09±0.32 | 4.99±0.51 | 5.23±0.42 | 5.00±0.21 |
| CL (mmol/L) | 106.2±4.0 | 105.2±4.0 | 107.5±9.1 | 103.6±3.5 |
| **Number of Females** | **10** | **10** | **10** | **10** |
| AST (U/L) | 63±13 | 90±50 | 86±38 | 72±23 |
| ALT (U/L) | 16±3 | 34±38 | 28±20 | 24±12 |
| SDH (U/L) | 6.8±2.3 | 10.1±4.2 | 10.6±9.2 | 8.6±3.9 |
| ALKP (U/L) | 46±9 | 63±31 | 50±22 | 52±15 |
| BILI (mg/dL) | 0.18±0.03 | 0.19±0.03 | 0.18±0.02 | 0.17±0.03 |
| BUN (mg/dL) | 13±3 | 13±2 | 14±3 | 12±2 |
| CREA (mg/dL) | 0.34±0.04 | 0.39±0.08 | 0.39±0.05 | 0.35±0.03 |
| CHOL (mg/dL) | 82±12 | 86±17 | 77±14 | 99±15** |
| TRIG (mg/dL) | 62±19 | 44±7** | 47±15 | 48±12 |
| GLUC (mg/dL) | 122±6 | 126±19 | 123±18 | 125±14 |
| TP (g/dL) | 7.3±0.5 | 7.0±0.6 | 7.3±0.5 | 7.3±0.5 |
| ALB (g/dL) | 4.0±0.4 | 3.7±0.5 | 4.0±0.3 | 3.9±0.3 |
| GLOB (g/dL) | 3.3±0.2 | 3.4±0.3 | 3.3±0.2 | 3.4±0.3 |
| CALC (mg/dL) | 10.5±0.5 | 10.3±0.8 | 10.5±0.4 | 10.6±0.3 |
| IPHS (mg/dL)* | 5.0±0.8 | 4.6±0.7 | 4.8±0.6 | 4.9±0.6 |
| Na (mmol/L) | 143.4±6.8 | 146.3±10.4 | 147.8±6.1 | 148.2±7.1 |
| K (mmol/L) | 4.22±0.26 | 4.16±0.72 | 4.28±0.37 | 4.34±0.33 |
| CL (mmol/L) | 104.7±5.4 | 106.9±9.1 | 108.3±4.4 | 108.0±4.7 |

Data are presented as mean ± standard deviation (SD). * Number of males: (Control: 7; 50 000 ppm: 7; 100 000 ppm: 9); ** = Significantly different from control by Dunnett's test (2 sided), $p < 0.05$; ALB = albumin; ALKP = alkaline phosphatase; ALT = alanine aminotransferase; AST = aspartate aminotransferase; BILI = total bilirubin; BUN = urea nitrogen; CALC = calcium; CHOL = cholesterol; CL = chloride; CREA = creatinine; dL = deciliter; g = grams; GLOB = globulin; GLUC = glucose; IPHS = inorganic phosphorus ; U = international units; K = potassium; L = liter; mg = milligrams; mmol = millimoles; Na= sodium; SDH = sorbitol dehydrogenase; TP = total protein; TRIG = triglycerides

**Table 10. Absolute organ weights (g) and relative organ to body weights (%) of rats administered pecan shell fiber for 91 days.**

| | Control | 50 000 mg/kg/day | 100 000 mg/kg/day | 150 000 mg/kg/day |
|---|---|---|---|---|
| **Number of Males** | **9** | **10** | **10** | **10** |
| Body weight (g) | 603.7±39.7 | 614.4±106.1 | 637.5±71.3 | 595.6±60.1 |
| Adrenals (g) | 0.0628±0.0076 | 0.0638±0.0125 | 0.0706±0.0105 | 0.0632±0.0100 |
| Adrenals/TBW (Ratio) | 0.1046±0.0159 | 0.1043±0.0151 | 0.1113±0.0154 | 0.1070±0.0189 |
| Brain (g) | 2.341±0.101 | 2.285±0.106 | 2.289±0.065 | 2.350±0.083 |
| Brain/TBW (Ratio) | 3.890±0.263 | 3.798±0.536 | 3.629±0.397 | 3.981±0.418 |
| Epididymides (g) | 1.499±0.255 | 1.504±0.168 | 1.586±0.156 | 1.556±0.085 |
| Epididymides/TBW (Ratio) | 2.4871±0.4261 | 2.5035±0.4895 | 2.5079±0.3011 | 2.6380±0.3177 |
| Heart (g) | 1.752±0.164 | 1.778±0.227 | 1.785±0.192 | 1.772±0.209 |
| Heart/TBW (Ratio) | 2.914±0.345 | 2.923±0.292 | 2.805±0.185 | 2.976±0.183 |
| Kidneys (g) | 3.748±0.379 | 3.729±0.407 | 3.912±0.527 | 3.773±0.597 |
| Kidneys/TBW (Ratio) | 6.215±0.573 | 6.140±0.599 | 6.149±0.571 | 6.330±0.701 |
| Liver (g) | 15.201±1.222 | 15.441±3.592 | 15.804±2.088 | 13.613±1.939 |
| Liver/TBW (Ratio) | 25.239±2.200 | 25.019±2.514 | 24.765±1.584 | 22.795±1.405* |
| Spleen (g) | 1.040±0.102 | 0.983±0.222 | 1.033±0.241 | 0.858±0.120 |
| Spleen/TBW (Ratio) | 1.727±0.177 | 1.595±0.188 | 1.616±0.294 | 1.441±0.152** |
| Testes (g) | 3.592±0.419 | 3.652±0.368 | 3.806±0.470 | 3.622±0.259 |
| Testes/TBW (Ratio) | 5.960±0.680 | 6.053±0.896 | 5.998±0.685 | 6.120±0.569 |
| Thymus (g) | 0.3463±0.0914 | 0.4124±0.0602 | 0.4020±0.1200 | 0.3478±0.0813 |
| Thymus/TBW (Ratio) | 0.5735±0.1458 | 0.6835±0.1329 | 0.6328±0.1883 | 0.5815±0.1000 |
| **Number of Females** | **10** | **10** | **10** | **10** |
| Body weight (g) | 285.8±28.4 | 276.0±23.2 | 286.8±41.4 | 275.6±34.8 |
| Adrenals (g) | 0.0657±0.0121 | 0.0609±0.0104 | 0.0702±0.0104 | 0.0623±0.0170 |
| Adrenals /TBW (Ratio) | 0.2285±0.0248 | 0.2227±0.0436 | 0.2477±0.0413 | 0.2245±0.0514 |
| Brain (g) | 2.087±0.084 | 2.077±0.106 | 2.082±0.102 | 2.046±0.085 |
| Brain /TBW (Ratio) | 7.376±0.882 | 7.568±0.680 | 7.371±0.889 | 7.511±0.799 |
| Heart (g) | 1.034±0.100 | 0.996±0.132 | 0.990±0.103 | 0.974±0.104 |
| Heart /TBW (Ratio) | 3.623±0.158 | 3.612±0.393 | 3.485±0.395 | 3.558±0.366 |
| Kidneys (g) | 1.938±0.172 | 1.877±0.127 | 1.921±0.171 | 1.906±0.299 |
| Kidneys /TBW (Ratio) | 6.800±0.427 | 6.844±0.725 | 6.803±1.077 | 6.900±0.436 |
| Liver (g) | 7.500±1.106 | 7.169±0.694 | 7.409±0.861 | 7.345±0.995 |
| Liver /TBW (Ratio) | 26.194±2.139 | 26.128±3.277 | 26.046±2.912 | 26.644±1.308 |
| Spleen (g) | 0.569±0.075 | 0.638±0.138 | 0.560±0.076 | 0.576±0.119 |
| Spleen /TBW (Ratio) | 1.999±0.254 | 2.321±0.527 | 1.972±0.310 | 2.090±0.344 |
| Ovaries (g) | 0.0728±0.0127 | 0.0733±0.0126 | 0.0746±0.0158 | 0.0641±0.0164 |
| Ovaries /TBW (Ratio) | 0.2549±0.0364 | 0.2665±0.0446 | 0.2647±0.0661 | 0.2318±0.0471 |
| Thymus (g) | 0.2947±0.0691 | 0.2463±0.0457 | 0.2765±0.0653 | 0.3144±0.0705 |
| Thymus /TBW (Ratio) | 1.0307±0.2226 | 0.8913±0.1435 | 0.9674±0.2006 | 1.1499±0.2559 |
| Uterus and Oviduct (g) | 0.885±0.198 | 0.726±0.179 | 0.935±0.308 | 0.744±0.250 |
| Uterus and Oviduct /TBW (Ratio) | 3.119±0.722 | 2.650±0.718 | 3.281±1.133 | 2.732±1.000 |

Data are presented as mean ± SD. Relative organ weights (Ratios) presented in the table are times 1000.
*Significantly different from control by Dunn's test (2 sided), $p < 0.05$. **Significantly different from control by Dunn Test after rank transformation (2 sided), $p < 0.01$. TBW = terminal body weight

There were no macroscopic or microscopic findings related to administration of the test material. Fluid-filled uteri that were noted in three to five females/group (including controls) corresponded to cyclical dilation of the uterine lumen that was attributable to variation in the estrous cycle in individual animals. The uterus of a high dose female presented with a 3 x 4 mm irregular clear, tan cyst encompassing the right horn and adjacent cervix. Microscopically, this finding correlated to uterine cyst and was considered incidental and unrelated to test substance exposure. A unilateral renal carcinoma in was found in one mid dose female, which was poorly demarcated, composed of polyhedral cells, intensely basophilic, and exhibited a high nucleus-to-cytoplasm ratio that focally formed tubular-like structures. The mitotic index was approximately 5 to 7 mitoses *per* high power field. Considering the lack of dose-response relationship, the solitary incidence and the absence of pre-neoplastic or neoplastic lesions in any other animal from this study, the renal tubular carcinoma observed in this animal was considered incidental and unrelated to the test substance exposure. Renal tubular tumors are sometimes observed as an incidental finding in rats (Hall *et al.*, 2007; Lanzoni *et al.*, 2007). One mid dose male exhibited a 2 x 3 mm, irregular wall area, associated with a white, 2 x 5 mm, luminal concretion in the urinary bladder. This finding did not have a microscopic correlate and was considered incidental. All recorded microscopic findings occurred sporadically or at a similar incidence in control- and test substance-treated groups and were generally of the type commonly seen in rats of this strain and age; therefore they were considered incidental and unrelated to treatment.

### 6.3.Genotoxicity

Pecan shell fiber was nonmutagenic in a bacterial reverse mutation (OECD Guideline 471) assay using a plate incorporation (experiment 1) and a preincubation (experiment 2) method (Dolan *et al.*, 2016). Strains used in the study included *Salmonella typhimurium* TA98, TA100, TA1535 and TA1537 and *Escherichia coli* WP2*uvr*A. Each assay was conducted in the presence and absence of metabolic activation with S9 mix prepared from the S9 microsomal fraction of rat liver. The test substance was suspended homogeneously in distilled water and added to plates to achieve concentrations of 5000, 2500, 1000, 316, 100 and 31.6 µg/plate.[9] The positive controls in the absence of S9 mix were 4-nitro-o-phenylene-diamine (4-NOPD) for *S. typhimurium* TA98 and TA1537, sodium azide ($NaN_3$) for *S. typhimurium* TA100 and TA1535 and methylmethanesulfonate (MMS) for *E. coli* WP2*uvr*A. The positive control for all bacterial strains in the presence of S9 mix was 2-aminoanthracene (2-AA) and the negative control for all strains in the presence or absence of S9 mix was distilled water.

The test substance did not induce any significant or dose-dependent increases in the numbers of revertant colonies in any strain tested in the absence or presence of S9 mix (Table 11). Precipitation of the test item was observed in all tester strains used in experiment 1 at concentrations of 1000 µg/plate and higher (with and without metabolic activation) as well in all tester strains used in experiment 2 at a concentration of 2500 µg/plate (with and without metabolic activation), as evidenced by a decrease in the mutation frequency. In experiment 1, toxic effects of the test substance were observed in *S. typhimurium* TA1537 at concentrations of 2500 µg/plate and higher (without metabolic activation). In experiment 2, the top concentration was selected as

[9] Five thousand micrograms/plate is the maximum required concentration in the bacterial mutation test for non-toxic test articles Due to the strong precipitation at 5000 µg/plate in experiment 1, 2500 µg/plate was chosen as the highest concentration for experiment 2.

2500 µg/plate, the lowest precipitating concentration. Toxic effects of the test item (decrease in mutation frequency and a reduction in the background lawn) were noted in *S. typhimurium* TA100 at a concentration of 2500 µg/plate (without metabolic activation) and in *S. typhimurium* TA1537 at concentrations of 1000 µg/plate and higher (without metabolic activation). The assay was valid, as the positive controls showed a distinct increase in revertants meeting the criteria for a positive response.

A water extract of dried, finely ground pecan shells (31.8% w/w[10]) tested negative for mutagenicity in *S. typhimurium* TA98, TA100, TA102, TA1535 and TA1537 at up to 5000 µg/plate in the absence or presence of S9 (Porto *et al.*, 2013). There was no mention of cytotoxicity or precipitation in the assay. The results of this study suggest that the water soluble components of pecan shell fiber are not mutagenic.

**Table 11. Reverse mutation assay of pecan shell fiber in *Salmonella typhimurium* and *Escherichia coli*: mean number of revertants / plate**

| Concentration (µg) | TA98 | | TA100 | | TA1535 | | TA1537 | | WP2*uvr*A | |
|---|---|---|---|---|---|---|---|---|---|---|
| | -S9 | +S9 | -S9 | +S9 | -S9 | +S9 | -S9 | +S9 | -S9 | +S9 |
| **Experiment 1** | | | | | | | | | | |
| 0[a] | 21 | 27 | 107 | 97 | 9 | 6 | 13 | 7 | 38 | 43 |
| 10.0 | 24 | 31 | 102 | 118 | 12 | 9 | 13 | 15 | 40 | 32 |
| 31.6 | 18 | 32 | 93 | 108 | 7 | 8 | 8 | 13 | 30 | 36 |
| 100 | 26 | 33 | 97 | 100 | 8 | 6 | 11 | 11 | 42 | 34 |
| 316 | 28 | 32 | 106 | 113 | 7 | 5 | 8 | 9 | 32 | 37 |
| 1000 | 26* | 38* | 102* | 118 | 16* | 8* | 9* | 8* | 40* | 38* |
| 2500 | 26* | 42* | 88* | 94 | 12* | 10* | 7* | 10* | 39* | 42* |
| 5000 | 40* | 26* | 105* | 102 | 8* | 4* | 5* | 8* | 34* | 48* |
| Positive Control | 346[b] | 1665[c] | 412[d] | 1437[c] | 539[d] | 84[c] | 120[b] | 306[c] | 289[e] | 127[c] |
| **Experiment 2** | | | | | | | | | | |
| 0[a] | 18 | 31 | 87 | 95 | 16 | 16 | 11 | 11 | 46 | 41 |
| 3.16 | 16 | 32 | 93 | 93 | 19 | 12 | 10 | 12 | 39 | 49 |
| 10.0 | 16 | 29 | 91 | 90 | 18 | 11 | 9 | 10 | 42 | 47 |
| 31.6 | 23 | 33 | 92 | 101 | 13 | 16 | 8 | 10 | 39 | 50 |
| 100 | 25 | 31 | 100 | 109 | 16 | 10 | 13 | 8 | 39 | 46 |
| 316 | 30 | 35 | 111 | 100 | 19 | 15 | 7 | 13 | 50 | 53 |
| 1000 | 20 | 31 | 103 | 109 | 23 | 15 | 9† | 14 | 46 | 55 |
| 2500 | 23* | 24* | 67*,† | 104* | 15* | 15* | 1*,† | 10* | 40* | 44* |
| Positive Control | 329[b] | 1007[c] | 597[d] | 1124[c] | 586[d] | 57[c] | 95[b] | 126[c] | 572[e] | 164[c] |

Pecan shell fiber was tested using the standardized plate incorporation assay (Experiment 1) and the pre-incubation method (Experiment 2). * Precipitate observed; † toxicity observed

[a] Distilled water
[b] 4-Nitro-*o*-phenylene diamine
[c] 2-Aminoanthracene
[d] Sodium azide
[e] Methylmethanesulfonate

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[10] weight/weight

An OECD Guideline 474[11] study was performed to investigate the potential of pecan shell fiber to induce micronuclei in polychromatic erythrocytes (PCE) in peripheral blood of male and female adult NMRI mice (minimum seven weeks of age) (Dolan *et al.*, 2016). In the micronucleus test, groups of mice ($n$ = five/sex) were dosed with 3333 mg/kg bw test material suspended homogeneously in cottonseed oil, three times daily (with two hours between each dose) for a cumulative dose of 10 g/kg bw/day (the maximum tolerated dose determined by a pre-experiment). The volume of each application was 10 ml/kg bw. The vehicle control (cottonseed oil) was administered by gavage three times *per* day to vehicle control mice (five mice/sex), in an identical manner as the test group animals. The positive control material (cyclophosphamide) was administered once intraperitoneally to a separate group of five mice/sex, at a dose of 40 mg/kg bw. Samples of blood were taken from the tail vein 44 hours (all groups) and 68 hours after the last dose (vehicle and test material groups only). Blood cells were immediately fixed in ultracold methanol. Before analysis (at least 24 hours after fixation), fixed blood cells were washed in Hank's balanced salt solution, centrifuged at 600 x g for five minutes (4°C) and the supernatant discarded. Blood cell populations were discriminated using specific antibodies against CD71 (expressed only at the surface of immature erythrocytes) and CD61 (expressed at the surface of platelets). DNA content of micronuclei was determined by the use of a DNA specific stain (propidium iodide, PI). Anti-CD71 and anti-CD61 antibodies were then labeled with fluorescein isothiocyanate (FITC) and phycoerythrin (PE), respectively, and samples were analyzed using flow cytometry. For all dose groups (including positive and negative controls), 10,000 PCE *per* animal were scored for the incidence of micronucleated immature erythrocytes. To detect a possible cytotoxic effect of the test item, the ratio between immature and mature erythrocytes was determined. The result is expressed as relative PCE (proportion of polychromatic (immature) erythrocytes among total erythrocytes). The test substance was judged positive if any test group exhibited a statistically significant ($p<0.05$) increase in the frequency of micronucleated immature erythrocytes compared with the concurrent negative control (using the nonparametric Mann-Whitney Test) and the results are outside the distribution of the historical negative control data (*e.g.*, Poisson-based 95% control limits).

The mean relative PCE values for the 44 hour negative controls were 1.82% (male mice) and 0.80% (female mice). The corresponding values for treated mice were 0.75% (males) and 1.35% (females). The relative PCE value for the treated male group was decreased relative to the concurrent negative control but this decrease was not statistically significant. However, the value was below the historical control limits. The relative PCE value for the treated female group was statistically significantly increased compared to the concurrent negative control.For 68 hours, the mean relative PCE values were 2.10% (control males) and 1.46% (control females), 1.86% (treated males) and 2.23% (treated females). The value in treated males was decreased and the value in treated females was increased compared to the concurrent negative control, but the changes were not statistically significant. The fact that relative PCE values were slightly affected by in animals receiving the test material results suggests that erythrocytes were exposed to the test material.

[11] OECD Guidelines for the Testing of Chemicals, Section 4. Test No. 474. Mammalian Erythrocyte Micronucleus Test. Available at http://www.oecd.org/env/test-no-474-mammalian-erythrocyte-micronucleus-test-9789264224292-en.htm; site last visited December 21, 2015.

Under the conditions of the test, pecan shell fiber did not cause a biologically relevant increase in the frequency of MN-PCE. Mean frequencies of micronucleated PCE (MN-PCE) for treated mice at 44 hours were 0.22% (males) and 0.28% (females) (Figure 5). For males, the mean MN-PCE values were within the range of the concurrent negative control as well as within the historical control limits for negative controls. The mean value observed in the female group was statistically significantly increased compared to the concurrent negative control, but within the distribution of the historical negative control data (e.g., Poisson-based 95% control limits).. Therefore, the significant increase in MN-PCE in treated females at 44 hours with respect to the concurrent control did not meet all criteria for a positive response. For the 68-hour measurement, the mean values of both males and females (0.25% and 0.2%, respectively) were within the range of the concurrent negative control and the distribution of the historical negative control data (e.g., Poisson-based 95% control limits historical controls (Figure 6). The assay was considered valid as the MN-PCE frequencies for the negative control rats were within the expected historical distribution and an adequate positive control response was obtained (3.51% MN-PCE in males and 1.90% MN-PCE in females).

A water extract of dried, ground pecan shells (31.8% w/w) tested negative for clastogenicity/aneugenicity in a bone marrow micronucleus study and DNA damage in a comet assay performed on healthy or diabetic rats provided 100 mg/kg bw/day of the extract by oral gavage for 28 days (Porto *et al.*, 2013; 2015) In male Wistar rats administered 20% ethanol in drinking water for 12 weeks, inclusion of a 1:40 (2.5%) aqueous extract of dried, crushed pecan shells (AEPS) in drinking water prevented ethanol from causing an increase in micronucleated red blood cells (Müller *et al.*, 2011). Results of these studies suggest that the water soluble components of pecan shell fiber are not clastogenic/aneugenic or induce DNA damage, and exert a protective effect on ethanol-induced erythrocyte genotoxicity.

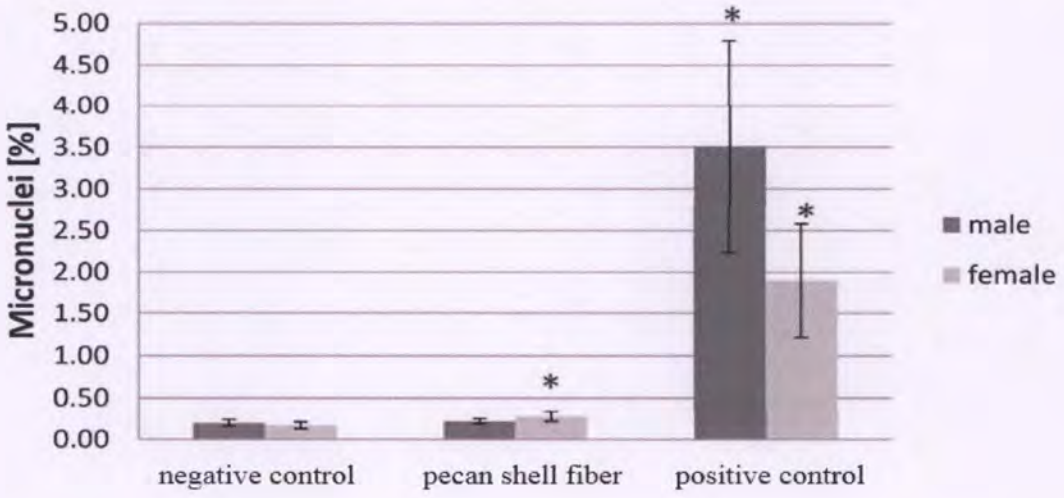


**Figure 5. Incidences of Micronucleated PCE [%] after 44 hours (mean ± standard deviation) (Dolan *et al.*, 2016)**

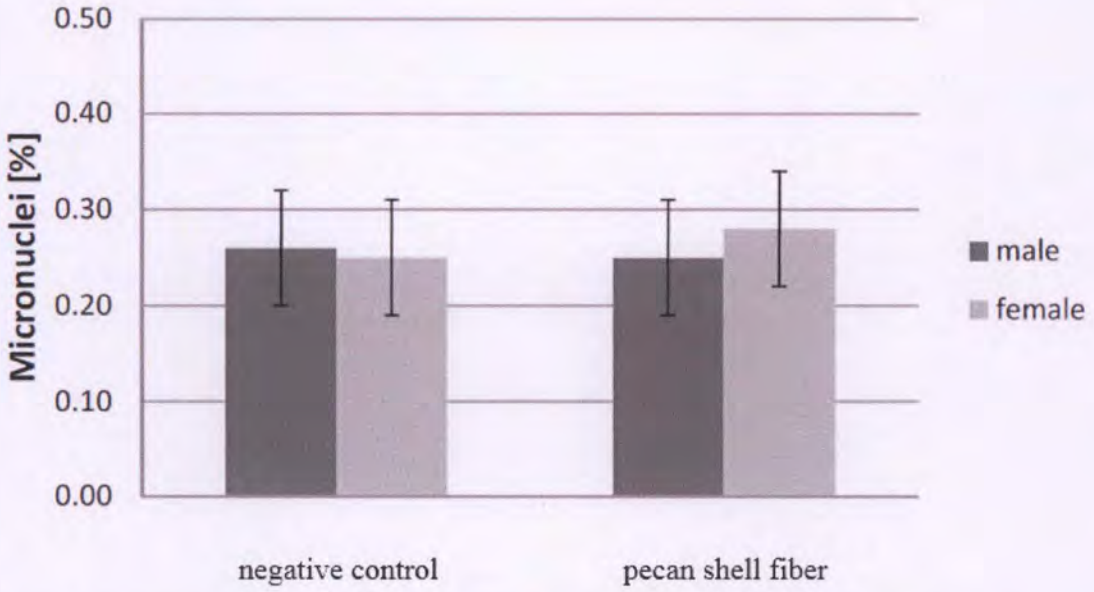


**Figure 6. Incidences of Micronucleated PCE [%] after 68 hours (mean ± standard deviation) (Dolan *et al.*, 2016)**

## 7. EVALUATION

Pecan shell fiber is produced entirely from ground shells of pecan nuts, with no additional substances added during processing. The primary constituents of the ingredient are cellulose and lignin (53-71% of the ingredient), measured collectively as acid detergent fiber (ADF). The ingredient also contains significant quantities of hemicellulose and proanthocyanidins, and smaller amounts of polyphenols, protein, fat and ash. Pecan shell fiber is stable for twelve months at ambient temperature and humidity and accelerated temperature and humidity when stored in sealed containers, for all parameters measured except polyphenols and proanthocyanidins, which are stable to six months. Packaging for the ingredient will be labeled "May Contain Pecans".

Pecan shell fiber is intended to be added to selected foods at 2-10%, as a source of dietary fiber (mainly insoluble). Addition of pecan shell fiber to a product may also provide antioxidant effects. The levels of use of pecan shell fiber in foods are self-limiting as undesirable taste characteristics develop when recommended use levels are exceeded. Ground pecan shells have a history of use in animal feed use as a source of fiber, and the ingredient is included in the AAFCO OP. Pecan shell fiber is included in Everything Added to Food in the United States (EAFUS) and has been determined GRAS by FEMA for use as a flavoring substance in meat products at a maximum level of 3000 ppm. The estimated total mean and 90$^{th}$ percentile consumption of pecan shell fiber from all sources, if pecan shell fiber were added to the selected foods at the levels specified in this document, is 5,177.4 mg/day (86.3 mg/kg bw/day based on a 60 kg bw) and 10,233.6 mg/day (170.6 mg/kg bw/day), respectively. This is a conservative estimate of intake, as it is unlikely that people will consume all of the pecan shell fiber-containing products at the 90th percentile levels of intake.

Experiments that were conducted to determine the safety of pecan shell fiber include a bacterial reverse mutation assay, an *in vivo* mouse micronucleus study and a 13-week dietary toxicity study in rats. The NOAEL in the 13-week dietary study in rats is 150,000 ppm (9,947.5 mg/kg bw/day in males and 11,082.8 mg/kg bw/day in females), the highest dose administered. Pecan shell fiber was not mutagenic in a bacterial reverse mutation assay using plate incorporation and preincubation methods, with and without metabolic activation. Pecan shell fiber tested negative for clastogenicity/aneugenicity in a mouse micronucleus study conducted at the maximum tolerated oral gavage dose of 10 g/kg bw, administered in three split doses of 333 mg/kg bw. Excepting the renal tubular carcinoma observed in one mid dose female, which was ruled incidental, no pre-neoplastic or neoplastic lesions were found in any tissues of rats provided up to 150,000 ppm pecan shell fiber in the diet for 13 weeks. Studies performed with an aqueous extract of dried, ground pecan shells show that the water soluble components of pecan shell fiber are not mutagenic or clastogenic.

In conclusion, the overall database supports the safety of pecan shell fiber when used in food at a level providing a 90$^{th}$ percentile intake of 10,233.6 mg/day (170.6 mg/kg bw/day). This is a conservative estimate of intake, as it is unlikely that people will consume all of the pecan shell fiber containing products at the 90$^{th}$ percentile level.

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## 8. CERTIFICATION

The undersigned authors of this document—a dossier in support of GRAS status for the use of pecan shell fiber —hereby certify that, to the best of their knowledge and belief, this document is a complete and balanced representation of all available information, favorable as well as unfavorable, known by the authors to be relevant to evaluation of the substance described herein.

(b) (6)

February 4 2016

**Laurie C. Dolan, Ph.D.**
Senior Toxicologist, Burdock Group

**Date**

(b) (6)

4 February 2016

**George A. Burdock, Ph.D.**
*Diplomate*, American Board of Toxicology,
*Fellow*, American College of Nutrition
President, Burdock Group

**Date**

## 9. CONCLUSION

Following a critical evaluation of the information available, the Expert Panel has determined that, based on common knowledge throughout the scientific community knowledgeable about the safety of substances directly or indirectly added to food, there is reasonable certainty that pecan shell fiber, produced in accordance with current Good Manufacturing Practice (cGMP), is safe under the intended conditions of use, and is therefore Generally Recognized As Safe (GRAS), by scientific procedures, when used as a source of fiber and antioxidants for certain foods, so that total daily consumption of pecan shell fiber from all sources may be up to 10,233.6 mg/day (170.6 mg/kg bw/day). In particular, the Expert Panel has evaluated the proposed use of pecan shell fiber at specified levels in the foods listed in Table 5 of this document and has concluded that such use results in an estimated intake of pecan shell fiber that is GRAS.

It is our opinion that other experts qualified by scientific training and experience to evaluate the safety of food and food ingredients would concur with these conclusions.

## 10. SIGNATURES

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2-12-16

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Date

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Date

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Date

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## APPENDIX 1

See attachment

eurofins | Nutrition Analysis Center

Eurofins Scientific Inc.
Nutrition Analysis Center
2200 Rittenhouse Street, Suite 150
Des Moines, IA 50321
Tel. +1 515 265 1461
Fax. +1 515 266 5453

**Eurofins Sample Code:** 464-2014-08015080
**Sample Description:** Pecan Shell Fiber
**Client Sample Code:** [redacted]
**PO Number:** 48135
**Client Code:** [redacted]

**Entry Date:** 08/01/2014
**Reporting Date:** 08/14/2014

South Georgia Pecan - SRC
attn: John Nizio
309 South Lee Street
PO Box 5366
Valdosta, GA 31601

South Georgia Pecan - SRC
Attn: John Nizio
309 South Lee Street
PO Box 5366
Valdosta, GA 31601

# CERTIFICATE OF ANALYSIS

AR-14-QD-109161-01

| Test | Result | |
|---|---|---|
| **QD002 - Fiber, Acid Detergent** | | **Completed: 08/07/2014** |
| Ankom ADF 05/03 | | |
| * Fiber, Acid Detergent | 67.4 % | |
| **QD161 - Fiber, Neutral Detergent** | | **Completed: 08/07/2014** |
| Ankom NDF | | |
| * Fiber, Neutral Detergent | 75.9 % | |
| **QD101 - Hemicellulose** | | **Completed: 08/07/2014** |
| Ankom NDF/Ankom ADF | | |
| Hemicellulose | 8.5 % | |
| **QD112 - Lignin** | | **Completed: 08/07/2014** |
| Ankom Lignin | | |
| Lignin | 28.4 % | |
| **QD039 - Cellulose** | | **Completed: 08/07/2014** |
| Ankom ADF/Ankom Lignin | | |
| Cellulose | 39.0 % | |
| **QA133 - Arsenic (ICP-MS)** | | **Completed: 08/14/2014** |
| AOAC 986.15 mod. | | |
| Arsenic (As) | <0.02 mg/kg | |
| **QA417 - Lead (ICP-MS)** | | **Completed: 08/14/2014** |
| AOAC 986.15 mod. | | |
| Lead (Pb) | <0.02 mg/kg | |
| **QD00T - Visual Appearance- Other** | | **Completed: 08/04/2014** |
| Internal Method | | |
| Appearance | Required | |
| *Sample is a homegenous light reddish-brown fine, fibrous powder. No off odors.* | | |
| **UMZY9 - Salmonella spp. - AOAC 2009.03** | | **Completed: 08/02/2014** |
| AOAC 2009.03 | | |
| * Salmonella spp. | Negative /25 g | |
| **QAA69 - Metconazole** | | **Completed: 08/14/2014** |
| No Reference | | |
| Metconazole | <0.01 mg/kg | |
| **QA01P - Pesticides Quechers GC-MSMS** | | **Completed: 08/14/2014** |
| AOAC 2007.01 | | |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Acephate | <0.01 mg/kg |
| Acetochlor | <0.01 mg/kg |
| Aclonifen | <0.01 mg/kg |
| Acrinathrin | <0.01 mg/kg |
| Aldrin | <0.01 mg/kg |
| Allethrin | <0.05 mg/kg |
| Ametryn | <0.01 mg/kg |
| Atrazine | <0.01 mg/kg |
| Azaconazole | <0.01 mg/kg |
| Azinphos-ethyl (Ethyl Guthion) | <0.01 mg/kg |
| Azinphos-methyl | <0.01 mg/kg |
| Benalaxyl | <0.01 mg/kg |
| Benfluralin | <0.01 mg/kg |
| Bifenox | <0.01 mg/kg |
| Bifenthrin | <0.01 mg/kg |
| Bromacil | <0.01 mg/kg |
| Bromocyclen | <0.01 mg/kg |
| Bromophos | <0.01 mg/kg |
| Bromophos-ethyl | <0.01 mg/kg |
| Bromopropylate | <0.01 mg/kg |
| Butafenacil | <0.01 mg/kg |
| Butylate | <0.01 mg/kg |
| Captafol | <0.02 mg/kg |
| Captan | <0.02 mg/kg |
| Carbetamide | <0.01 mg/kg |
| Carbophenothion | <0.01 mg/kg |
| Carbophenothion-methyl | <0.01 mg/kg |
| Chlordane, cis- | <0.01 mg/kg |
| Chlordane, oxy- | <0.01 mg/kg |
| Chlordane, trans- | <0.01 mg/kg |
| Chlordene, beta | <0.01 mg/kg |
| Chlordene, gamma- | <0.01 mg/kg |
| Chlordimeform | <0.01 mg/kg |
| Chlorethoxyfos | <0.01 mg/kg |
| Chlorfenapyr | <0.01 mg/kg |
| Chlorfenson | <0.01 mg/kg |
| Chlorobenzilate | <0.01 mg/kg |
| Chloroneb | <0.01 mg/kg |
| Chloropropylate | <0.01 mg/kg |
| Chlorothalonil | <0.01 mg/kg |
| Chlorpropham (CIPC) | <0.01 mg/kg |
| Chlorpyrifos | <0.01 mg/kg |
| Chlorpyrifos-methyl | <0.01 mg/kg |
| Chlorthal-dimethyl | <0.01 mg/kg |
| Chlorthiofos | <0.01 mg/kg |
| Chlorthion | <0.01 mg/kg |
| Chlozolinate | <0.01 mg/kg |
| Cloquintocet-mexyl | <0.01 mg/kg |
| Coumaphos | <0.01 mg/kg |
| Crimidine | <0.01 mg/kg |
| Crotoxyphos | <0.01 mg/kg |
| Cyanazine | <0.05 mg/kg |
| Cyanofenphos | <0.01 mg/kg |

**Eurofins Sample Code:** (b) (6)
**Client Sample Code:** [redacted]

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Cyanophos | <0.05 mg/kg |
| Cyfluthrin | <0.01 mg/kg |
| Cyhalothrin lambda- | <0.01 mg/kg |
| Cypermethrin | <0.01 mg/kg |
| Dacthal (DCPA) | <0.01 mg/kg |
| DDD, o,p'- | <0.01 mg/kg |
| DDD, p,p'- | <0.01 mg/kg |
| DDE, o,p'- | <0.01 mg/kg |
| DDE, p,p'- | <0.01 mg/kg |
| DDT, o,p'- | <0.01 mg/kg |
| DDT, p,p'- | <0.01 mg/kg |
| DEF (Butifos) | <0.01 mg/kg |
| Deltamethrin | <0.01 mg/kg |
| Demeton-O | <0.01 mg/kg |
| Demeton-S | <0.01 mg/kg |
| Demeton-S-methyl | <0.01 mg/kg |
| Dialifos | <0.01 mg/kg |
| Diallate | <0.01 mg/kg |
| Diazinon (O Analog) | <0.01 mg/kg |
| Dichlobenil | <0.01 mg/kg |
| Dichlofenthion | <0.01 mg/kg |
| Dichlofluanid | <0.01 mg/kg |
| Dichlone | <0.05 mg/kg |
| Dichlorvos | <0.01 mg/kg |
| Diclobutrazol | <0.01 mg/kg |
| Dicloran | <0.01 mg/kg |
| Dicofol, p,p- | <0.01 mg/kg |
| Dicrotophos | <0.01 mg/kg |
| Dieldrin | <0.01 mg/kg |
| Diflufenican | <0.01 mg/kg |
| Dimethachlor | <0.01 mg/kg |
| Dimethenamid | <0.01 mg/kg |
| Dimoxystrobin | <0.01 mg/kg |
| Dioxathion | <0.01 mg/kg |
| Diphenamid | <0.01 mg/kg |
| Diphenyl | <0.01 mg/kg |
| Diphenylamine | <0.01 mg/kg |
| Dipropetryn | <0.01 mg/kg |
| Disulfoton | <0.01 mg/kg |
| Disulfoton-PS-sulfone | <0.01 mg/kg |
| Endosulfan I (alpha-endosulfan) | <0.01 mg/kg |
| Endosulfan II (beta-Endosulfan) | <0.01 mg/kg |
| Endosulfan sulphate | <0.01 mg/kg |
| Endrin | <0.01 mg/kg |
| Endrin ketone | <0.01 mg/kg |
| Endrin-aldehyde | <0.01 mg/kg |
| EPN | <0.01 mg/kg |
| EPTC | <0.01 mg/kg |
| Esfenvalerate | <0.01 mg/kg |
| Ethalfluralin | <0.01 mg/kg |
| Ethion | <0.01 mg/kg |
| Ethoprophos | <0.01 mg/kg |
| Ethoxyquin | <0.01 mg/kg |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Etoxazole | <0.01 mg/kg |
| Etridiazole | <0.01 mg/kg |
| Etrimfos | <0.01 mg/kg |
| Famophos | <0.01 mg/kg |
| Famoxadone | <0.01 mg/kg |
| Fenamidone | <0.01 mg/kg |
| Fenchlorphos | <0.01 mg/kg |
| Fenfluthrin | <0.01 mg/kg |
| Fenitrothion | <0.01 mg/kg |
| Fenoxaprop-p-ethyl | <0.01 mg/kg |
| Fenpropathrin | <0.01 mg/kg |
| Fenson | <0.01 mg/kg |
| Fensulfothion | <0.01 mg/kg |
| Fenthion | <0.01 mg/kg |
| Fenvalerate | <0.01 mg/kg |
| Fluazifop-P-butyl | <0.01 mg/kg |
| Fluchloralin | <0.01 mg/kg |
| Flucythrinate | <0.01 mg/kg |
| Flumioxazin | <0.01 mg/kg |
| Fluotrimazole | <0.01 mg/kg |
| Fluquinconazole | <0.01 mg/kg |
| Flutriafol | <0.01 mg/kg |
| Fluvalinate | <0.01 mg/kg |
| Folpet | <0.01 mg/kg |
| Fonofos | <0.01 mg/kg |
| Gamma-cyhalothrin | <0.01 mg/kg |
| gamma-HCH (Lindane) | <0.01 mg/kg |
| Halfenprox | <0.01 mg/kg |
| HCH, alpha- | <0.01 mg/kg |
| HCH, beta- | <0.01 mg/kg |
| HCH, delta- | <0.01 mg/kg |
| Heptachlor | <0.01 mg/kg |
| Heptachlor Epoxide (cis, trans) | <0.01 mg/kg |
| Hexachlorobenzene (HCB) | <0.01 mg/kg |
| Hexazinone | <0.01 mg/kg |
| Iprobenfos | <0.01 mg/kg |
| Iprodione | <0.01 mg/kg |
| Isazophos | <0.01 mg/kg |
| Isocarbamid | <0.01 mg/kg |
| Isocarbofos | <0.01 mg/kg |
| Isodrin | <0.01 mg/kg |
| Isofenphos-methyl | <0.01 mg/kg |
| Isopropalin | <0.01 mg/kg |
| Isoprothiolane | <0.01 mg/kg |
| Isoxadifen-ethyl | <0.01 mg/kg |
| Lenacil | <0.01 mg/kg |
| Leptophos | <0.01 mg/kg |
| Malathion | <0.01 mg/kg |
| Mefenpyr-diethyl | <0.01 mg/kg |
| Mepronil | <0.01 mg/kg |
| Metazachlor | <0.01 mg/kg |
| Methacriphos | <0.01 mg/kg |
| Methamidophos | <0.01 mg/kg |

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Methoprothryn | <0.01 mg/kg |
| Methoxychlor, o,o' | <0.01 mg/kg |
| Methoxychlor, p,p' | <0.01 mg/kg |
| Mevinphos | <0.01 mg/kg |
| Mexacarbate | <0.01 mg/kg |
| MGK-264 | <0.01 mg/kg |
| Mirex | <0.01 mg/kg |
| Monocrotophos | <0.01 mg/kg |
| Naproanilide | <0.01 mg/kg |
| Napropamide | <0.01 mg/kg |
| Nitralin | <0.01 mg/kg |
| Nitrapyrin | <0.01 mg/kg |
| Nitrofen | <0.01 mg/kg |
| Nitrothal-isopropyl | <0.01 mg/kg |
| Nonachlor, cis- | <0.01 mg/kg |
| Nonachlor, trans- | <0.01 mg/kg |
| Norea | <0.01 mg/kg |
| Norflurazon | <0.01 mg/kg |
| Ofurace | <0.01 mg/kg |
| Omethoate | <0.01 mg/kg |
| o-Phenylphenol | <0.01 mg/kg |
| Oxyfluorfen | <0.01 mg/kg |
| Paclobutrazol | <0.01 mg/kg |
| Parathion | <0.01 mg/kg |
| Parathion oxygen analog | <0.01 mg/kg |
| Parathion-methyl | <0.01 mg/kg |
| Parathion-methyl oxygen analog | <0.01 mg/kg |
| PCB 101 | <0.01 mg/kg |
| PCB 138 | <0.01 mg/kg |
| PCB 153 | <0.01 mg/kg |
| PCB 180 | <0.01 mg/kg |
| PCB 28 | <0.01 mg/kg |
| PCB 52 | <0.01 mg/kg |
| Pebulate | <0.01 mg/kg |
| Pentachloranisole | <0.01 mg/kg |
| Pentachloroaniline | <0.01 mg/kg |
| Pentachlorobenzene | <0.01 mg/kg |
| Pentachlorobenzonitrile | <0.01 mg/kg |
| Pentachlorothioanisole | <0.01 mg/kg |
| Penthiopyrad | <0.01 mg/kg |
| Permethrin | 0.02 mg/kg |
| Perthane | <0.01 mg/kg |
| Phenothrin | <0.01 mg/kg |
| Phenthoate | <0.01 mg/kg |
| Phorate | <0.01 mg/kg |
| Phorate-O-analogue | <0.01 mg/kg |
| Phorate-sulfone | <0.01 mg/kg |
| Phosmet | <0.01 mg/kg |
| Phosphamidon | <0.01 mg/kg |
| Picolinafen | <0.01 mg/kg |
| Picoxystrobin | <0.01 mg/kg |
| Procymidone | <0.01 mg/kg |
| Profenofos | <0.01 mg/kg |

**Eurofins Sample Code:** (b) (6)
**Client Sample Code:** [redacted]

| Test | Result | |
|---|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | | **Completed: 08/14/2014** |
| AOAC 2007.01 | | |
| Profluralin | <0.01 mg/kg | |
| Prometryn | <0.01 mg/kg | |
| Propachlor | <0.01 mg/kg | |
| Propamocarb | <0.01 mg/kg | |
| Propazine | <0.01 mg/kg | |
| Propetamphos | <0.01 mg/kg | |
| Propyzamide | <0.01 mg/kg | |
| Prothiofos | <0.01 mg/kg | |
| Pyrazophos | <0.01 mg/kg | |
| Pyridalyl | <0.01 mg/kg | |
| Pyrifenox | <0.01 mg/kg | |
| Quinalphos | <0.01 mg/kg | |
| Quinomethionate (Morestan) | <0.01 mg/kg | |
| Quintozene | <0.01 mg/kg | |
| Quizalofop-P-ethyl | <0.01 mg/kg | |
| S 421 (Octachlordipropylether) | <0.01 mg/kg | |
| Secbumeton | <0.01 mg/kg | |
| Silaneophan | <0.01 mg/kg | |
| Silthiofam | <0.01 mg/kg | |
| Simazine | <0.01 mg/kg | |
| Tebupirimfos | <0.01 mg/kg | |
| Tecnazene | <0.01 mg/kg | |
| Tefluthrin | <0.01 mg/kg | |
| Terbacil | <0.01 mg/kg | |
| Terbuthylazine | <0.01 mg/kg | |
| Tetrachloroaniline, 2,3,4,6- | <0.01 mg/kg | |
| Tetradifon | <0.01 mg/kg | |
| Tetramethrin | <0.01 mg/kg | |
| Tetrasul | <0.01 mg/kg | |
| Thiometon | <0.01 mg/kg | |
| Thionazin | <0.01 mg/kg | |
| THPI (Tetrahydrophthalimide) | <0.01 mg/kg | |
| Tolclofos-methyl | <0.01 mg/kg | |
| Tolylfluanid | <0.01 mg/kg | |
| Toxaphene (camphechlor) | <0.05 mg/kg | |
| Tralomethrin | <0.01 mg/kg | |
| Transfluthrin | <0.01 mg/kg | |
| Triazophos | <0.01 mg/kg | |
| Trichlorfon | <0.01 mg/kg | |
| Trichloronat | <0.01 mg/kg | |
| Trifluralin | <0.01 mg/kg | |
| Triticonazole | <0.01 mg/kg | |
| Vinclozolin | <0.01 mg/kg | |
| **QA636 - Rimsulfuron** | | **Completed: 08/14/2014** |
| No Reference | | |
| Rimsulfuron | <0.01 mg/kg | |
| **QA730 - Penoxsulam** | | **Completed: 08/14/2014** |
| No Reference | | |
| Penoxsulam | <0.01 mg/kg | |
| **QA0AC - Flubendiamide** | | **Completed: 08/14/2014** |
| No Reference | | |
| Flubendiamide | <0.01 mg/kg | |

**Eurofins Sample Code:** (b) (6)
**Client Sample Code:** [redacted]

| Test | Result | |
|---|---|---|
| **QAA54 - Spinetoram** | | **Completed: 08/14/2014** |
| No Reference | | |
| Spinetoram | <0.010 mg/kg | |
| **QA714 - Emamectin (LC-MSMS)** | | **Completed: 08/14/2014** |
| No Reference | | |
| Emamectin | <0.01 mg/kg | |
| **QA679 - Pyraflufen-ethyl** | | **Completed: 08/14/2014** |
| No Reference | | |
| Pyraflufen-ethyl | <0.01 mg/kg | |
| **QA021 - Aflatoxin Profile (LC-MSMS)** | | **Completed: 08/14/2014** |
| AOAC 999.07 Modified | | |
| Sum of Aflatoxins B1,B2,G1,G2 | < 4.0 µg/kg | |
| Aflatoxin B1 | < 1.0 µg/kg | |
| Aflatoxin B2 | < 1.0 µg/kg | |
| Aflatoxin G1 | < 1.0 µg/kg | |
| Aflatoxin G2 | < 1.0 µg/kg | |
| **QA01R - Pesticides Quechers-LC-MS/MS** | | **Completed: 08/14/2014** |
| AOAC 2007.01 | | |
| Alachlor | <0.01 mg/kg | |
| Carbendazim/Benomyl (sum) | <0.01 mg/kg | |
| Climbazole | <0.01 mg/kg | |
| Clodinafop-propargyl | <0.01 mg/kg | |
| Demeton-S-methyl-sulfone | <0.01 mg/kg | |
| Demeton-S-sulfone | <0.01 mg/kg | |
| Desmetryn | <0.01 mg/kg | |
| Dimethametryn | <0.01 mg/kg | |
| Dimethylvinphos | <0.01 mg/kg | |
| Etobenzanid | <0.01 mg/kg | |
| Isofenphos | <0.01 mg/kg | |
| Mecarbam | <0.01 mg/kg | |
| Naled | <0.01 mg/kg | |
| Naphthalene Acetamide | <0.01 mg/kg | |
| Phenkapton | <0.01 mg/kg | |
| Promecarb | <0.01 mg/kg | |
| Prometon | <0.01 mg/kg | |
| Propanil | <0.01 mg/kg | |
| Propaquizafop | <0.01 mg/kg | |
| Propargite | <0.02 mg/kg | |
| Propham | <0.05 mg/kg | |
| Propiconazole | <0.01 mg/kg | |
| Propoxur | <0.01 mg/kg | |
| Proquinazid | <0.01 mg/kg | |
| Prosulfocarb | <0.01 mg/kg | |
| Pymetrozine | <0.02 mg/kg | |
| Pyraclostrobin | <0.01 mg/kg | |
| Pyridaben | <0.01 mg/kg | |
| Pyridaphenthion | <0.01 mg/kg | |
| Pyridate | <0.01 mg/kg | |
| Pyrimethanil | <0.01 mg/kg | |
| Pyriproxyfen | <0.01 mg/kg | |
| Quinoxyfen | <0.01 mg/kg | |
| Rotenone | <0.01 mg/kg | |
| Sethoxydim | <0.01 mg/kg | |
| Simetryn | <0.01 mg/kg | |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Spinosad | <0.01 mg/kg |
| Spirodiclofen | <0.01 mg/kg |
| Spiromesifen | <0.01 mg/kg |
| Spiroxamine | <0.01 mg/kg |
| Sulfotep | <0.01 mg/kg |
| Sulprofos | <0.01 mg/kg |
| Tebuconazole | <0.01 mg/kg |
| Tebufenozide | <0.01 mg/kg |
| Tebufenpyrad | <0.01 mg/kg |
| Tebuthiuron | <0.01 mg/kg |
| Teflubenzuron | <0.01 mg/kg |
| Tepraloxydim | <0.01 mg/kg |
| Terbufos | <0.01 mg/kg |
| Terbumeton | <0.01 mg/kg |
| Terbutryn | <0.01 mg/kg |
| Tetrachlorvinphos | <0.01 mg/kg |
| Tetraconazole | <0.01 mg/kg |
| Thiabendazole | <0.01 mg/kg |
| Thiacloprid | <0.01 mg/kg |
| Thiamethoxam | <0.01 mg/kg |
| Thiobencarb | <0.01 mg/kg |
| Thiodicarb | <0.01 mg/kg |
| Thiofanox-sulfoxide | <0.01 mg/kg |
| Thiophanate-methyl | <0.01 mg/kg |
| Tralkoxydim | <0.01 mg/kg |
| Triadimefon | <0.01 mg/kg |
| Triadimenol | <0.01 mg/kg |
| Triallate | <0.01 mg/kg |
| Triazamate | <0.01 mg/kg |
| Tricyclazole | <0.01 mg/kg |
| Tridemorph | <0.01 mg/kg |
| Trifloxystrobin | <0.01 mg/kg |
| Triflumizole | <0.01 mg/kg |
| Triflumuron | <0.01 mg/kg |
| Triforine | <0.01 mg/kg |
| Trimethacarb 2.3.5- | <0.01 mg/kg |
| Trimethycarb, 3,4,5- | <0.01 mg/kg |
| Uniconazole-P | <0.01 mg/kg |
| 1-Naphthol | <0.01 mg/kg |
| 2,4-D | <0.01 mg/kg |
| 3-Hydroxycarbofuran | <0.01 mg/kg |
| 3-ketocarbofuran | <0.01 mg/kg |
| Abamectin | <0.01 mg/kg |
| Acetamiprid | <0.01 mg/kg |
| Acibenzolar-s-methyl | <0.01 mg/kg |
| Aldicarb | <0.01 mg/kg |
| Aldicarb-sulfone | <0.01 mg/kg |
| Aldicarb-sulfoxide | <0.01 mg/kg |
| Aminocarb | <0.01 mg/kg |
| Amitraz | <0.01 mg/kg |
| Azadirachtin | <0.01 mg/kg |
| Azinphos-methyl oxon | <0.01 mg/kg |
| Azoxystrobin | <0.01 mg/kg |

**Eurofins Sample Code:** (b) (6)
**Client Sample Code:** [redacted]

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Bendiocarb | <0.01 mg/kg |
| Bensulide | <0.01 mg/kg |
| Bifenazate | <0.01 mg/kg |
| Bitertanol | <0.01 mg/kg |
| Boscalid | <0.01 mg/kg |
| Bromuconazole | <0.01 mg/kg |
| Bupirimate | <0.01 mg/kg |
| Buprofezin | <0.01 mg/kg |
| Butocarboxim-sulfoxide | <0.05 mg/kg |
| Carbaryl | <0.01 mg/kg |
| Carbendazim | <0.01 mg/kg |
| Carbofuran | <0.01 mg/kg |
| Carbofuran (Phenol) | <0.01 mg/kg |
| Carbosulfan | <0.01 mg/kg |
| Carboxin | <0.01 mg/kg |
| Carfentrazone-ethyl | <0.01 mg/kg |
| Chlorfenvinphos | <0.01 mg/kg |
| Chloridazone | <0.01 mg/kg |
| Chloroxuron | <0.01 mg/kg |
| Clethodim | <0.01 mg/kg |
| Clofentezine | <0.01 mg/kg |
| Clomazone | <0.01 mg/kg |
| Clothianidin | <0.01 mg/kg |
| Cyazofamid | <0.01 mg/kg |
| Cycloate | <0.01 mg/kg |
| Cycloxydim | <0.01 mg/kg |
| Cymoxanil | <0.01 mg/kg |
| Cyproconazole | <0.01 mg/kg |
| Cyprodinil | <0.01 mg/kg |
| Cyromazine | <0.05 mg/kg |
| Desmedipham | <0.01 mg/kg |
| Pirimicarb, desmethyl-formamido- | <0.01 mg/kg |
| Pirimicarb, desmethyl- | <0.01 mg/kg |
| Diafenthiuron | <0.01 mg/kg |
| Diazinon | <0.01 mg/kg |
| Diethofencarb | <0.01 mg/kg |
| Difenoconazole | <0.01 mg/kg |
| Diflubenzuron | <0.01 mg/kg |
| Dimethoate | <0.01 mg/kg |
| Dimethomorph | <0.01 mg/kg |
| Diniconazole | <0.01 mg/kg |
| Dioxacarb | <0.01 mg/kg |
| Diuron | <0.01 mg/kg |
| Dodine | <0.01 mg/kg |
| Edifenphos | <0.01 mg/kg |
| Epoxiconazole | <0.01 mg/kg |
| Etaconazole | <0.01 mg/kg |
| Ethiofencarb | <0.01 mg/kg |
| Ethiofencarb-sulfone | <0.01 mg/kg |
| Ethiofencarb-sulfoxide | <0.01 mg/kg |
| Ethofumesate | <0.01 mg/kg |
| Etofenprox | <0.01 mg/kg |
| Fenamiphos | <0.01 mg/kg |

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 08/14/2014** |
| AOAC 2007.01 | |
| Fenarimol | <0.02 mg/kg |
| Fenazaquin | <0.01 mg/kg |
| Fenbuconazole | <0.01 mg/kg |
| Fenhexamid | <0.01 mg/kg |
| Fenobucarb | <0.01 mg/kg |
| Fenoxycarb | <0.01 mg/kg |
| Fenpropimorph | <0.01 mg/kg |
| Fenpyroximate | <0.01 mg/kg |
| Fipronil | <0.01 mg/kg |
| Flonicamid | <0.01 mg/kg |
| Fludioxonil | <0.01 mg/kg |
| Flufenacet | <0.01 mg/kg |
| Flufenoxuron | <0.01 mg/kg |
| Fluopicolide | <0.01 mg/kg |
| Flusilazole | <0.01 mg/kg |
| Fluthiacet-methyl | <0.01 mg/kg |
| Forchlorfenuron | <0.01 mg/kg |
| Fosthiazate | <0.01 mg/kg |
| Furalaxyl | <0.01 mg/kg |
| Furathiocarb | <0.01 mg/kg |
| Heptenophos | <0.01 mg/kg |
| Hexaconazole | <0.01 mg/kg |
| Hexaflumuron | <0.01 mg/kg |
| Hexythiazox | <0.01 mg/kg |
| Imazalil | <0.01 mg/kg |
| Imidacloprid | <0.01 mg/kg |
| Indoxacarb | <0.01 mg/kg |
| Iprovalicarb | <0.01 mg/kg |
| Isoprocarb | <0.01 mg/kg |
| Isoproturon | <0.01 mg/kg |
| Isoxaben | <0.01 mg/kg |
| Isoxaflutole | <0.01 mg/kg |
| Kresoxim-methyl | <0.01 mg/kg |
| Linuron | <0.01 mg/kg |
| Lufenuron | <0.01 mg/kg |
| Malaoxon | <0.01 mg/kg |
| Mepanipyrim | <0.01 mg/kg |
| Metalaxyl | <0.01 mg/kg |
| Metamitron | <0.05 mg/kg |
| Methabenztiazuron | <0.01 mg/kg |
| Methidathion | <0.01 mg/kg |
| Methiocarb | <0.01 mg/kg |
| Methiocarb-sulfone | <0.01 mg/kg |
| Methiocarb sulfoxide | <0.01 mg/kg |
| Methomyl | <0.01 mg/kg |
| Methoxyfenozide | <0.01 mg/kg |
| Metolachlor | <0.01 mg/kg |
| Metolcarb | <0.01 mg/kg |
| Metoxuron | <0.01 mg/kg |
| Metribuzin | <0.01 mg/kg |
| Molinate | <0.01 mg/kg |
| Monolinuron | <0.01 mg/kg |
| Myclobutanil | <0.01 mg/kg |

Eurofins Sample Cod (b) (6)
Client Sample Code:

| Test | Result | |
|---|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | | **Completed: 08/14/2014** |
| AOAC 2007.01 | | |
| Neburon | <0.01 mg/kg | |
| Nitenpyram | <0.05 mg/kg | |
| Nuarimol | <0.01 mg/kg | |
| Oxadiazon | <0.01 mg/kg | |
| Oxadixyl | <0.01 mg/kg | |
| Oxamyl | <0.01 mg/kg | |
| Oxydemeton-methyl | <0.01 mg/kg | |
| Penconazole | <0.01 mg/kg | |
| Pencycuron | <0.01 mg/kg | |
| Pendimethalin | <0.01 mg/kg | |
| Phenmedipham | <0.01 mg/kg | |
| Phorate-sulfoxide | <0.01 mg/kg | |
| Phosalone | <0.01 mg/kg | |
| Piperonyl butoxide (PBO) | 0.02 mg/kg | |
| Pirimicarb | <0.01 mg/kg | |
| Pirimiphos-methyl | <0.01 mg/kg | |
| Pirimiphos-ethyl | <0.01 mg/kg | |
| Prochloraz | <0.01 mg/kg | |
| Profoxydim | <0.01 mg/kg | |
| **QAA80 - Avermectin B1a** | | **Completed: 08/14/2014** |
| No Reference | | |
| Avermectin B1a | <0.010 mg/kg | |
| **QA0AA - Chlorantraniliprole** | | **Completed: 08/14/2014** |
| No Reference | | |
| Chlorantraniliprole | <0.01 mg/kg | |
| **QA16C - Fluopyram (Foods, GC-MSMS)** | | **Completed: 08/14/2014** |
| No Reference | | |
| Fluopyram | <0.01 mg/kg | |
| **QA0AD - Fluridone** | | **Completed: 08/14/2014** |
| No Reference | | |
| Fluridone | <0.01 mg/kg | |
| **QA14Q - Oryzalin (LC-MSMS)** | | **Completed: 08/14/2014** |
| No Reference | | |
| Oryzalin | <0.01 mg/kg | |
| **QA09S - Saflufenacil (LC-MSMS)** | | **Completed: 08/14/2014** |
| No Reference | | |
| Saflufenacil | <0.01 mg/kg | |
| **QA0AB - Spirotetramat** | | **Completed: 08/14/2014** |
| No Reference | | |
| Spirotetramat | <0.01 mg/kg | |

**The test result is covered by our current A2LA accreditation.*

**Eurofins Sample Code:** (b) (6)
**Client Sample Code:** [redacted]

Respectfully Submitted,
Eurofins Scientific Inc.

(b) (6)

David Gross
Support Services Manager

Biological Testing
Cert:3329:01

Chemical Testing
Cert:2927:01

Results shown in this report relate solely to the item submitted for analysis.
All results are reported on an "As Received" basis unless otherwise stated.
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Eurofins Scientific, Inc. Measurement of Uncertainty can be obtained upon request.

eurofins | Nutrition Analysis Center

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**Eurofins Sample Code:** (b) (6)
**Sample Description:** Pecan Shell Fiber
**Client Sample Code:** [redacted]
**PO Number:** 50492
**Client Code:** QD0005157

**Entry Date:** 05/26/2015
**Reporting Date:** 06/26/2015

South Georgia Pecan - SRC
attn: John Nizio
309 South Lee Street
PO Box 5366
Valdosta, GA 31601

South Georgia Pecan - SRC
Attn: John Nizio
309 South Lee Street
PO Box 5366
Valdosta, GA 31601

# CERTIFICATE OF ANALYSIS

AR-15-QD-069755-04

This analytical report supersedes AR-15-QD-069755-03.

| Test | Result | |
|---|---|---|
| **QD309 - Moisture-Forced Draft (Food Products)** | | **Completed: 05/29/2015** |
| AOAC 930.15 | | |
| * Moisture, Forced Draft | 6.45 % | |
| **QD252 - Protein - Combustion** | | **Completed: 05/29/2015** |
| AOAC 990.03; AOAC 992.15 | | |
| * Protein | 2.53 % | |
| **QD250 - Ash** | | **Completed: 05/29/2015** |
| AOAC 942.05 | | |
| * Ash | 2.29 % | |
| **QD226 - Calories, Calculated** | | **Completed: 06/03/2015** |
| CFR - Atwater calculation | | |
| * Calories Calculated | 386 kcal/100 g | |
| **QD038 - Carbohydrates, Calculated** | | **Completed: 06/03/2015** |
| CFR 21-calc. | | |
| * Carbohydrates, Calculated | 84.53 % | |
| **QD002 - Fiber, Acid Detergent** | | **Completed: 06/02/2015** |
| Ankom ADF 05/03 | | |
| * Fiber, Acid Detergent | 69.2 % | |
| **QD161 - Fiber, Neutral Detergent** | | **Completed: 06/02/2015** |
| Ankom NDF | | |
| * Fiber, Neutral Detergent | 74.1 % | |
| **QD101 - Hemicellulose** | | **Completed: 06/05/2015** |
| Ankom NDF/Ankom ADF | | |
| Hemicellulose | 4.9 % | |
| **QD112 - Lignin** | | **Completed: 06/05/2015** |
| Ankom Lignin | | |
| Lignin | 19.5 % | |
| **QD039 - Cellulose** | | **Completed: 06/05/2015** |
| Ankom ADF/Ankom Lignin | | |
| Cellulose | 49.7 % | |
| **QD059 - Fat by Acid Hydrolysis** | | **Completed: 06/22/2015** |
| AOAC 954.02 | | |
| * Crude Fat By Acid Hydrolysis | 4.20 % | |

| Test | Result | |
|---|---|---|
| **QD059 - Fat by Acid Hydrolysis (Cont.)** | | **Completed: 06/22/2015** |
| AOAC 954.02 | | |
| * Crude Fat by Acid Hydrolysis Retest 1 | 4.17 % | |
| * Crude Fat by Acid Hydrolysis Retest 2 | 4.15 % | |
| **QA133 - Arsenic (ICP-MS)** | | **Completed: 06/05/2015** |
| AOAC 986.15 mod. | | |
| Arsenic (As) | <0.02 mg/kg | |
| **QA417 - Lead (ICP-MS)** | | **Completed: 06/05/2015** |
| AOAC 986.15 mod. | | |
| Lead (Pb) | 0.06 mg/kg | |
| **QD610 - Mercury (ICP-MS)** | | **Completed: 06/05/2015** |
| AOAC 986.15 mod. | | |
| Mercury (Hg) | <0.010 mg/kg | |
| **QD07J - Wet Screen Particle Size - 100, 200, 325 (South Georgia Pecan Method)** | | **Completed: 06/26/2015** |
| Proprietary Method | | |
| Mesh Screen 100 | 100.0 % | |
| Mesh Screen 100 Retest 1 | 100.0 % | |
| Mesh Screen 100 Retest 2 | 100.0 % | |
| Mesh Screen 100 Retest 3 | 100.0 % | |
| Mesh Screen 200 | 95.0 % | |
| Mesh Screen 200 Retest 1 | 96.0 % | |
| Mesh Screen 200 Retest 2 | 96.0 % | |
| Mesh Screen 200 Retest 3 | 96.0 % | |
| Mesh Screen 325 | 96.0 % | |
| Mesh Screen 325 Retest 1 | 97.0 % | |
| Mesh Screen 325 Retest 2 | 96.0 % | |
| Mesh Screen 325 Retest 3 | 94.0 % | |
| **QD00T - Visual Appearance- Other** | | **Completed: 06/08/2015** |
| Internal Method | | |
| Appearance | Required | |
| *Sample is a homogenous light reddish-brown fine, fibrous powder. Some small clumping. No off odors* | | |
| **QAA69 - Metconazole** | | **Completed: 06/05/2015** |
| No Reference | | |
| Metconazole | <0.01 mg/kg | |
| **QA01P - Pesticides Quechers GC-MSMS** | | **Completed: 06/10/2015** |
| AOAC 2007.01 | | |
| Acephate | Not Detected mg/kg | |
| Acetochlor | Not Detected mg/kg | |
| Aclonifen | Not Detected mg/kg | |
| Acrinathrin | Not Detected mg/kg | |
| Aldrin | Not Detected mg/kg | |
| Allethrin | Not Detected mg/kg | |
| Ametryn | Not Detected mg/kg | |
| Atrazine | Not Detected mg/kg | |
| Azaconazole | Not Detected mg/kg | |
| Azinphos-ethyl (Ethyl Guthion) | Not Detected mg/kg | |
| Azinphos-methyl | Not Detected mg/kg | |
| Benalaxyl | Not Detected mg/kg | |
| Benfluralin | Not Detected mg/kg | |
| Bifenox | Not Detected mg/kg | |
| Bifenthrin | Not Detected mg/kg | |
| Bromacil | Not Detected mg/kg | |
| Bromocyclen | Not Detected mg/kg | |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Bromophos | Not Detected mg/kg |
| Bromophos-ethyl | Not Detected mg/kg |
| Bromopropylate | Not Detected mg/kg |
| Butafenacil | Not Detected mg/kg |
| Butylate | Not Detected mg/kg |
| Captafol | Not Detected mg/kg |
| Captan | Not Detected mg/kg |
| Carbetamide | Not Detected mg/kg |
| Carbophenothion | Not Detected mg/kg |
| Carbophenothion-methyl | Not Detected mg/kg |
| Chlordane, cis- | Not Detected mg/kg |
| Chlordane, oxy- | Not Detected mg/kg |
| Chlordane, trans- | Not Detected mg/kg |
| Chlordene, beta | Not Detected mg/kg |
| Chlordene, gamma- | Not Detected mg/kg |
| Chlordimeform | Not Detected mg/kg |
| Chlorethoxyfos | Not Detected mg/kg |
| Chlorfenapyr | Not Detected mg/kg |
| Chlorfenson | Not Detected mg/kg |
| Chlorobenzilate | Not Detected mg/kg |
| Chloroneb | Not Detected mg/kg |
| Chloropropylate | Not Detected mg/kg |
| Chlorothalonil | Not Detected mg/kg |
| Chlorpropham (CIPC) | Not Detected mg/kg |
| Chlorpyrifos | Not Detected mg/kg |
| Chlorpyrifos-methyl | Not Detected mg/kg |
| Chlorthal-dimethyl | Not Detected mg/kg |
| Chlorthiofos | Not Detected mg/kg |
| Chlorthion | Not Detected mg/kg |
| Chlozolinate | Not Detected mg/kg |
| Cloquintocet-mexyl | Not Detected mg/kg |
| Coumaphos | Not Detected mg/kg |
| Crimidine | Not Detected mg/kg |
| Crotoxyphos | Not Detected mg/kg |
| Cyanazine | Not Detected mg/kg |
| Cyanofenphos | Not Detected mg/kg |
| Cyanophos | Not Detected mg/kg |
| Cyfluthrin | Not Detected mg/kg |
| Cyhalothrin lambda- | Not Detected mg/kg |
| Cypermethrin | Not Detected mg/kg |
| Dacthal (DCPA) | Not Detected mg/kg |
| DDD, o,p'- | Not Detected mg/kg |
| DDD, p,p'- | Not Detected mg/kg |
| DDE, o,p'- | Not Detected mg/kg |
| DDE, p,p'- | Not Detected mg/kg |
| DDT, o,p'- | Not Detected mg/kg |
| DDT, p,p'- | Not Detected mg/kg |
| DEF (Butifos) | Not Detected mg/kg |
| Deltamethrin | Not Detected mg/kg |
| Demeton-O | Not Detected mg/kg |
| Demeton-S | Not Detected mg/kg |
| Demeton-S-methyl | Not Detected mg/kg |
| Dialifos | Not Detected mg/kg |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Diallate | Not Detected mg/kg |
| Diazinon (O Analog) | Not Detected mg/kg |
| Dichlobenil | Not Detected mg/kg |
| Dichlofenthion | Not Detected mg/kg |
| Dichlofluanid | Not Detected mg/kg |
| Dichlone | Not Detected mg/kg |
| Dichlorvos | Not Detected mg/kg |
| Diclobutrazol | Not Detected mg/kg |
| Dicloran | Not Detected mg/kg |
| Dicofol, p,p- | Not Detected mg/kg |
| Dicrotophos | Not Detected mg/kg |
| Dieldrin | Not Detected mg/kg |
| Diflufenican | Not Detected mg/kg |
| Dimethachlor | Not Detected mg/kg |
| Dimethenamid | Not Detected mg/kg |
| Dimoxystrobin | Not Detected mg/kg |
| Dioxathion | Not Detected mg/kg |
| Diphenamid | Not Detected mg/kg |
| Diphenyl | Not Detected mg/kg |
| Diphenylamine | Not Detected mg/kg |
| Dipropetryn | Not Detected mg/kg |
| Disulfoton | Not Detected mg/kg |
| Disulfoton-PS-sulfone | Not Detected mg/kg |
| Endosulfan I (alpha-endosulfan) | Not Detected mg/kg |
| Endosulfan II (beta-Endosulfan) | Not Detected mg/kg |
| Endosulfan sulphate | Not Detected mg/kg |
| Endrin | Not Detected mg/kg |
| Endrin ketone | Not Detected mg/kg |
| Endrin-aldehyde | Not Detected mg/kg |
| EPN | Not Detected mg/kg |
| EPTC | Not Detected mg/kg |
| Esfenvalerate | Not Detected mg/kg |
| Ethalfluralin | Not Detected mg/kg |
| Ethion | Not Detected mg/kg |
| Ethoprophos | Not Detected mg/kg |
| Ethoxyquin | Not Detected mg/kg |
| Etoxazole | Not Detected mg/kg |
| Etridiazole | Not Detected mg/kg |
| Etrimfos | Not Detected mg/kg |
| Famophos | Not Detected mg/kg |
| Famoxadone | Not Detected mg/kg |
| Fenamidone | Not Detected mg/kg |
| Fenchlorphos | Not Detected mg/kg |
| Fenfluthrin | Not Detected mg/kg |
| Fenitrothion | Not Detected mg/kg |
| Fenoxaprop-p-ethyl | Not Detected mg/kg |
| Fenpropathrin | Not Detected mg/kg |
| Fenson | Not Detected mg/kg |
| Fensulfothion | Not Detected mg/kg |
| Fenthion | Not Detected mg/kg |
| Fenvalerate | Not Detected mg/kg |
| Fluazifop-P-butyl | Not Detected mg/kg |
| Fluchloralin | Not Detected mg/kg |

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Flucythrinate | Not Detected mg/kg |
| Flumioxazin | Not Detected mg/kg |
| Fluotrimazole | Not Detected mg/kg |
| Fluquinconazole | Not Detected mg/kg |
| Flutriafol | Not Detected mg/kg |
| Fluvalinate | Not Detected mg/kg |
| Folpet | Not Detected mg/kg |
| Fonofos | Not Detected mg/kg |
| Gamma-cyhalothrin | Not Detected mg/kg |
| gamma-HCH (Lindane) | Not Detected mg/kg |
| Halfenprox | Not Detected mg/kg |
| HCH, alpha- | Not Detected mg/kg |
| HCH, beta- | Not Detected mg/kg |
| HCH, delta- | Not Detected mg/kg |
| Heptachlor | Not Detected mg/kg |
| Heptachlor Epoxide (cis, trans) | Not Detected mg/kg |
| Hexachlorobenzene (HCB) | Not Detected mg/kg |
| Hexazinone | Not Detected mg/kg |
| Iprobenfos | Not Detected mg/kg |
| Iprodione | Not Detected mg/kg |
| Isazophos | Not Detected mg/kg |
| Isocarbamid | Not Detected mg/kg |
| Isocarbofos | Not Detected mg/kg |
| Isodrin | Not Detected mg/kg |
| Isofenphos-methyl | Not Detected mg/kg |
| Isopropalin | Not Detected mg/kg |
| Isoprothiolane | Not Detected mg/kg |
| Isoxadifen-ethyl | Not Detected mg/kg |
| Lenacil | Not Detected mg/kg |
| Leptophos | Not Detected mg/kg |
| Malathion | Not Detected mg/kg |
| Mefenpyr-diethyl | Not Detected mg/kg |
| Mepronil | Not Detected mg/kg |
| Metazachlor | Not Detected mg/kg |
| Methacriphos | Not Detected mg/kg |
| Methamidophos | Not Detected mg/kg |
| Methoprothryn | Not Detected mg/kg |
| Methoxychlor, o,o' | Not Detected mg/kg |
| Methoxychlor, p,p' | Not Detected mg/kg |
| Mevinphos | Not Detected mg/kg |
| Mexacarbate | Not Detected mg/kg |
| MGK-264 | Not Detected mg/kg |
| Mirex | Not Detected mg/kg |
| Monocrotophos | Not Detected mg/kg |
| Naproanilide | Not Detected mg/kg |
| Napropamide | Not Detected mg/kg |
| Nitralin | Not Detected mg/kg |
| Nitrapyrin | Not Detected mg/kg |
| Nitrofen | Not Detected mg/kg |
| Nitrothal-isopropyl | Not Detected mg/kg |
| Nonachlor, cis- | Not Detected mg/kg |
| Nonachlor, trans- | Not Detected mg/kg |
| Norea | Not Detected mg/kg |

| Test | Result |
|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Norflurazon | Not Detected mg/kg |
| Ofurace | Not Detected mg/kg |
| Omethoate | Not Detected mg/kg |
| o-Phenylphenol | Not Detected mg/kg |
| Oxyfluorfen | Not Detected mg/kg |
| Paclobutrazol | Not Detected mg/kg |
| Parathion | Not Detected mg/kg |
| Parathion oxygen analog | Not Detected mg/kg |
| Parathion-methyl | Not Detected mg/kg |
| Parathion-methyl oxygen analog | Not Detected mg/kg |
| PCB 101 | Not Detected mg/kg |
| PCB 138 | Not Detected mg/kg |
| PCB 153 | Not Detected mg/kg |
| PCB 180 | Not Detected mg/kg |
| PCB 28 | Not Detected mg/kg |
| PCB 52 | Not Detected mg/kg |
| Pebulate | Not Detected mg/kg |
| Pentachloranisole | Not Detected mg/kg |
| Pentachloroaniline | Not Detected mg/kg |
| Pentachlorobenzene | Not Detected mg/kg |
| Pentachlorobenzonitrile | Not Detected mg/kg |
| Pentachlorothioanisole | Not Detected mg/kg |
| Penthiopyrad | Not Detected mg/kg |
| Permethrin | 0.03 mg/kg |
| Perthane | Not Detected mg/kg |
| Phenothrin | Not Detected mg/kg |
| Phenthoate | Not Detected mg/kg |
| Phorate | Not Detected mg/kg |
| Phorate-O-analogue | Not Detected mg/kg |
| Phorate-sulfone | Not Detected mg/kg |
| Phosmet | Not Detected mg/kg |
| Phosphamidon | Not Detected mg/kg |
| Picolinafen | Not Detected mg/kg |
| Picoxystrobin | Not Detected mg/kg |
| Procymidone | Not Detected mg/kg |
| Profenofos | Not Detected mg/kg |
| Profluralin | Not Detected mg/kg |
| Prometryn | Not Detected mg/kg |
| Propachlor | Not Detected mg/kg |
| Propamocarb | Not Detected mg/kg |
| Propazine | Not Detected mg/kg |
| Propetamphos | Not Detected mg/kg |
| Propyzamide | Not Detected mg/kg |
| Prothiofos | Not Detected mg/kg |
| Pyrazophos | Not Detected mg/kg |
| Pyridalyl | Not Detected mg/kg |
| Pyrifenox | Not Detected mg/kg |
| Quinalphos | Not Detected mg/kg |
| Quinomethionate (Morestan) | Not Detected mg/kg |
| Quintozene | Not Detected mg/kg |
| Quizalofop-P-ethyl | Not Detected mg/kg |
| S 421 (Octachlordipropylether) | Not Detected mg/kg |
| Secbumeton | Not Detected mg/kg |

| Test | Result | |
|---|---|---|
| **QA01P - Pesticides Quechers GC-MSMS (Cont.)** | | **Completed: 06/10/2015** |
| AOAC 2007.01 | | |
| Silaneophan | Not Detected mg/kg | |
| Silthiofam | Not Detected mg/kg | |
| Simazine | Not Detected mg/kg | |
| Tebupirimfos | Not Detected mg/kg | |
| Tecnazene | Not Detected mg/kg | |
| Tefluthrin | Not Detected mg/kg | |
| Terbacil | Not Detected mg/kg | |
| Terbuthylazine | Not Detected mg/kg | |
| Tetrachloroaniline, 2,3,4,6- | Not Detected mg/kg | |
| Tetradifon | Not Detected mg/kg | |
| Tetramethrin | Not Detected mg/kg | |
| Tetrasul | Not Detected mg/kg | |
| Thiometon | Not Detected mg/kg | |
| Thionazin | Not Detected mg/kg | |
| THPI (Tetrahydrophthalimide) | Not Detected mg/kg | |
| Tolclofos-methyl | Not Detected mg/kg | |
| Tolylfluanid | Not Detected mg/kg | |
| Toxaphene (camphechlor) | Not Detected mg/kg | |
| Tralomethrin | Not Detected mg/kg | |
| Transfluthrin | Not Detected mg/kg | |
| Triazophos | Not Detected mg/kg | |
| Trichlorfon | Not Detected mg/kg | |
| Trichloronat | Not Detected mg/kg | |
| Trifluralin | Not Detected mg/kg | |
| Triticonazole | Not Detected mg/kg | |
| Vinclozolin | Not Detected mg/kg | |
| Other screened pesticides | Not Detected | |
| **QA636 - Rimsulfuron** | | **Completed: 06/05/2015** |
| No Reference | | |
| Rimsulfuron | <0.01 mg/kg | |
| **QA730 - Penoxsulam** | | **Completed: 06/05/2015** |
| No Reference | | |
| Penoxsulam | <0.01 mg/kg | |
| **QA0AC - Flubendiamide** | | **Completed: 06/05/2015** |
| No Reference | | |
| Flubendiamide | <0.01 mg/kg | |
| **QAA54 - Spinetoram** | | **Completed: 06/05/2015** |
| No Reference | | |
| Spinetoram | <0.010 mg/kg | |
| **QA714 - Emamectin (LC-MSMS)** | | **Completed: 06/05/2015** |
| No Reference | | |
| Emamectin | <0.01 mg/kg | |
| **QA679 - Pyraflufen-ethyl** | | **Completed: 06/05/2015** |
| No Reference | | |
| Pyraflufen-ethyl | <0.01 mg/kg | |
| **QA205 - Cadmium (ICP-MS)** | | **Completed: 06/05/2015** |
| AOAC 986.15 mod. | | |
| Cadmium (Cd) | 0.023 mg/kg | |
| **QA01R - Pesticides Quechers-LC-MS/MS** | | **Completed: 06/10/2015** |
| AOAC 2007.01 | | |
| Alachlor | Not Detected mg/kg | |

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Other screened pesticides | Not Detected |
| Carbendazim/Benomyl (sum) | Not Detected mg/kg |
| Climbazole | Not Detected mg/kg |
| Clodinafop-propargyl | Not Detected mg/kg |
| Demeton-S-methyl-sulfone | Not Detected mg/kg |
| Demeton-S-sulfone | Not Detected mg/kg |
| Desmetryn | Not Detected mg/kg |
| Dimethametryn | Not Detected mg/kg |
| Dimethylvinphos | Not Detected mg/kg |
| Etobenzanid | Not Detected mg/kg |
| Isofenphos | Not Detected mg/kg |
| Mecarbam | Not Detected mg/kg |
| Naled | Not Detected mg/kg |
| Naphthalene Acetamide | Not Detected mg/kg |
| Phenkapton | Not Detected mg/kg |
| Promecarb | Not Detected mg/kg |
| Prometon | Not Detected mg/kg |
| Propanil | Not Detected mg/kg |
| Propaquizafop | Not Detected mg/kg |
| Propargite | Not Detected mg/kg |
| Propham | Not Detected mg/kg |
| Propiconazole | Not Detected mg/kg |
| Propoxur | Not Detected mg/kg |
| Proquinazid | Not Detected mg/kg |
| Prosulfocarb | Not Detected mg/kg |
| Pymetrozine | Not Detected mg/kg |
| Pyraclostrobin | Not Detected mg/kg |
| Pyridaben | Not Detected mg/kg |
| Pyridaphenthion | Not Detected mg/kg |
| Pyridate | Not Detected mg/kg |
| Pyrimethanil | Not Detected mg/kg |
| Pyriproxyfen | Not Detected mg/kg |
| Quinoxyfen | Not Detected mg/kg |
| Rotenone | Not Detected mg/kg |
| Sethoxydim | Not Detected mg/kg |
| Simetryn | Not Detected mg/kg |
| Spinosad | Not Detected mg/kg |
| Spirodiclofen | Not Detected mg/kg |
| Spiromesifen | Not Detected mg/kg |
| Spiroxamine | Not Detected mg/kg |
| Sulfotep | Not Detected mg/kg |
| Sulprofos | Not Detected mg/kg |
| Tebuconazole | Not Detected mg/kg |
| Tebufenozide | Not Detected mg/kg |
| Tebufenpyrad | Not Detected mg/kg |
| Tebuthiuron | Not Detected mg/kg |
| Teflubenzuron | Not Detected mg/kg |
| Tepraloxydim | Not Detected mg/kg |
| Terbufos | Not Detected mg/kg |
| Terbumeton | Not Detected mg/kg |
| Terbutryn | Not Detected mg/kg |
| Tetrachlorvinphos | Not Detected mg/kg |
| Tetraconazole | Not Detected mg/kg |

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Thiabendazole | Not Detected mg/kg |
| Thiacloprid | Not Detected mg/kg |
| Thiamethoxam | Not Detected mg/kg |
| Thiobencarb | Not Detected mg/kg |
| Thiodicarb | Not Detected mg/kg |
| Thiofanox-sulfoxide | Not Detected mg/kg |
| Thiophanate-methyl | Not Detected mg/kg |
| Tralkoxydim | Not Detected mg/kg |
| Triadimefon | Not Detected mg/kg |
| Triadimenol | Not Detected mg/kg |
| Triallate | Not Detected mg/kg |
| Triazamate | Not Detected mg/kg |
| Tricyclazole | Not Detected mg/kg |
| Tridemorph | Not Detected mg/kg |
| Trifloxystrobin | Not Detected mg/kg |
| Triflumizole | Not Detected mg/kg |
| Triflumuron | Not Detected mg/kg |
| Triforine | Not Detected mg/kg |
| Trimethacarb 2.3.5- | Not Detected mg/kg |
| Trimethycarb, 3,4,5- | Not Detected mg/kg |
| Uniconazole-P | Not Detected mg/kg |
| 1-Naphthol | Not Detected mg/kg |
| 2,4-D | Not Detected mg/kg |
| 3-Hydroxycarbofuran | Not Detected mg/kg |
| 3-ketocarbofuran | Not Detected mg/kg |
| Abamectin | Not Detected mg/kg |
| Acetamiprid | Not Detected mg/kg |
| Acibenzolar-s-methyl | Not Detected mg/kg |
| Aldicarb | Not Detected mg/kg |
| Aldicarb-sulfone | Not Detected mg/kg |
| Aldicarb-sulfoxide | Not Detected mg/kg |
| Aminocarb | Not Detected mg/kg |
| Amitraz | Not Detected mg/kg |
| Azadirachtin | Not Detected mg/kg |
| Azinphos-methyl oxon | Not Detected mg/kg |
| Azoxystrobin | Not Detected mg/kg |
| Bendiocarb | Not Detected mg/kg |
| Bensulide | Not Detected mg/kg |
| Bifenazate | Not Detected mg/kg |
| Bitertanol | Not Detected mg/kg |
| Boscalid | Not Detected mg/kg |
| Bromuconazole | Not Detected mg/kg |
| Bupirimate | Not Detected mg/kg |
| Buprofezin | Not Detected mg/kg |
| Butocarboxim-sulfoxide | Not Detected mg/kg |
| Carbaryl | Not Detected mg/kg |
| Carbendazim | Not Detected mg/kg |
| Carbofuran | Not Detected mg/kg |
| Carbofuran (Phenol) | Not Detected mg/kg |
| Carbosulfan | Not Detected mg/kg |
| Carboxin | Not Detected mg/kg |
| Carfentrazone-ethyl | Not Detected mg/kg |
| Chlorfenvinphos | Not Detected mg/kg |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Chloridazone | Not Detected mg/kg |
| Chloroxuron | Not Detected mg/kg |
| Clethodim | Not Detected mg/kg |
| Clofentezine | Not Detected mg/kg |
| Clomazone | Not Detected mg/kg |
| Clothianidin | Not Detected mg/kg |
| Cyazofamid | Not Detected mg/kg |
| Cycloate | Not Detected mg/kg |
| Cycloxydim | Not Detected mg/kg |
| Cymoxanil | Not Detected mg/kg |
| Cyproconazole | Not Detected mg/kg |
| Cyprodinil | Not Detected mg/kg |
| Cyromazine | Not Detected mg/kg |
| Desmedipham | Not Detected mg/kg |
| Pirimicarb, desmethyl-formamido- | Not Detected mg/kg |
| Pirimicarb, desmethyl- | Not Detected mg/kg |
| Diafenthiuron | Not Detected mg/kg |
| Diazinon | Not Detected mg/kg |
| Diethofencarb | Not Detected mg/kg |
| Difenoconazole | Not Detected mg/kg |
| Diflubenzuron | Not Detected mg/kg |
| Dimethoate | Not Detected mg/kg |
| Dimethomorph | Not Detected mg/kg |
| Diniconazole | Not Detected mg/kg |
| Dioxacarb | Not Detected mg/kg |
| Diuron | Not Detected mg/kg |
| Dodine | Not Detected mg/kg |
| Edifenphos | Not Detected mg/kg |
| Epoxiconazole | Not Detected mg/kg |
| Etaconazole | Not Detected mg/kg |
| Ethiofencarb | Not Detected mg/kg |
| Ethiofencarb-sulfone | Not Detected mg/kg |
| Ethiofencarb-sulfoxide | Not Detected mg/kg |
| Ethofumesate | Not Detected mg/kg |
| Etofenprox | Not Detected mg/kg |
| Fenamiphos | Not Detected mg/kg |
| Fenarimol | Not Detected mg/kg |
| Fenazaquin | Not Detected mg/kg |
| Fenbuconazole | Not Detected mg/kg |
| Fenhexamid | Not Detected mg/kg |
| Fenobucarb | Not Detected mg/kg |
| Fenoxycarb | Not Detected mg/kg |
| Fenpropimorph | Not Detected mg/kg |
| Fenpyroximate | Not Detected mg/kg |
| Fipronil | Not Detected mg/kg |
| Flonicamid | Not Detected mg/kg |
| Fludioxonil | Not Detected mg/kg |
| Flufenacet | Not Detected mg/kg |
| Flufenoxuron | Not Detected mg/kg |
| Fluopicolide | Not Detected mg/kg |
| Flusilazole | Not Detected mg/kg |
| Fluthiacet-methyl | Not Detected mg/kg |
| Forchlorfenuron | Not Detected mg/kg |

Eurofins Sample Code: (b) (6)
Client Sample Code: [redacted]

| Test | Result |
|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | **Completed: 06/10/2015** |
| AOAC 2007.01 | |
| Fosthiazate | Not Detected mg/kg |
| Furalaxyl | Not Detected mg/kg |
| Furathiocarb | Not Detected mg/kg |
| Heptenophos | Not Detected mg/kg |
| Hexaconazole | Not Detected mg/kg |
| Hexaflumuron | Not Detected mg/kg |
| Hexythiazox | Not Detected mg/kg |
| Imazalil | Not Detected mg/kg |
| Imidacloprid | Not Detected mg/kg |
| Indoxacarb | Not Detected mg/kg |
| Iprovalicarb | Not Detected mg/kg |
| Isoprocarb | Not Detected mg/kg |
| Isoproturon | Not Detected mg/kg |
| Isoxaben | Not Detected mg/kg |
| Isoxaflutole | Not Detected mg/kg |
| Kresoxim-methyl | Not Detected mg/kg |
| Linuron | Not Detected mg/kg |
| Lufenuron | Not Detected mg/kg |
| Malaoxon | Not Detected mg/kg |
| Mepanipyrim | Not Detected mg/kg |
| Metalaxyl | Not Detected mg/kg |
| Metamitron | Not Detected mg/kg |
| Methabenztiazuron | Not Detected mg/kg |
| Methidathion | Not Detected mg/kg |
| Methiocarb | Not Detected mg/kg |
| Methiocarb-sulfone | Not Detected mg/kg |
| Methiocarb sulfoxide | Not Detected mg/kg |
| Methomyl | Not Detected mg/kg |
| Methoxyfenozide | Not Detected mg/kg |
| Metolachlor | Not Detected mg/kg |
| Metolcarb | Not Detected mg/kg |
| Metoxuron | Not Detected mg/kg |
| Metribuzin | Not Detected mg/kg |
| Molinate | Not Detected mg/kg |
| Monolinuron | Not Detected mg/kg |
| Myclobutanil | Not Detected mg/kg |
| Neburon | Not Detected mg/kg |
| Nitenpyram | Not Detected mg/kg |
| Nuarimol | Not Detected mg/kg |
| Oxadiazon | Not Detected mg/kg |
| Oxadixyl | Not Detected mg/kg |
| Oxamyl | Not Detected mg/kg |
| Oxydemeton-methyl | Not Detected mg/kg |
| Penconazole | Not Detected mg/kg |
| Pencycuron | Not Detected mg/kg |
| Pendimethalin | Not Detected mg/kg |
| Phenmedipham | Not Detected mg/kg |
| Phorate-sulfoxide | Not Detected mg/kg |
| Phosalone | Not Detected mg/kg |
| Piperonyl butoxide (PBO) | 0.02 mg/kg |
| Pirimicarb | Not Detected mg/kg |
| Pirimiphos-methyl | Not Detected mg/kg |
| Pirimiphos-ethyl | Not Detected mg/kg |

**Eurofins Sample Code:** (b) (6)
**Client Sample Code:** [redacted]

| Test | Result | |
|---|---|---|
| **QA01R - Pesticides Quechers-LC-MS/MS (Cont.)** | | **Completed: 06/10/2015** |
| AOAC 2007.01 | | |
| Prochloraz | Not Detected mg/kg | |
| Profoxydim | Not Detected mg/kg | |
| **QAA80 - Avermectin B1a** | | **Completed: 06/05/2015** |
| No Reference | | |
| Avermectin B1a | <0.010 mg/kg | |
| **QA0AA - Chlorantraniliprole** | | **Completed: 06/05/2015** |
| No Reference | | |
| Chlorantraniliprole | <0.01 mg/kg | |
| **QA16C - Fluopyram (Foods, GC-MSMS)** | | **Completed: 06/05/2015** |
| No Reference | | |
| Fluopyram | <0.01 mg/kg | |
| **QA0AD - Fluridone** | | **Completed: 06/05/2015** |
| No Reference | | |
| Fluridone | <0.01 mg/kg | |
| **QA14Q - Oryzalin (LC-MSMS)** | | **Completed: 06/05/2015** |
| No Reference | | |
| Oryzalin | <0.01 mg/kg | |
| **QA09S - Saflufenacil (LC-MSMS)** | | **Completed: 06/05/2015** |
| No Reference | | |
| Saflufenacil | <0.01 mg/kg | |
| **QA0AB - Spirotetramat** | | **Completed: 06/05/2015** |
| No Reference | | |
| Spirotetramat | <0.01 mg/kg | |

**The test result is covered by our current A2LA accreditation.*

Respectfully Submitted,
Eurofins Scientific Inc.

(b) (6)

David Gross
Support Services Manager

Biological Testing
Cert:3329:01

Chemical Testing
Cert:2927:01

Results shown in this report relate solely to the item submitted for analysis.
All results are reported on an "As Received" basis unless otherwise stated.
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Eurofins Scientific, Inc. Measurement of Uncertainty can be obtained upon request.

## APPENDIX 2

See attachment

SOUTHEASTERN REDUCTION COMPANY
DIVISION OF SOUTH GEORGIA PECAN COMPANY
POST OFFICE BOX 5366, VALDOSTA, GEORGIA 31603-5366

PHONE (229) 259-0111
FAX (229) 249-0370

Quality Nut Shell Products

## Test on Ground Pecan Shell Fiber:

Wet Screen Particle size analysis (method)

1. Wet screen particle size analysis method-1 (conservative):

(1). Pre-wet all screens to be used in the test to eliminate static electricity and any surface tension that may hold water on the screens. As the test is being done, it is expected that water is flowing through the screens and not building up and filling the screen. If water build up occurs, slow the water volume and gently rub the screen with your hand to open the area for water and material to flow through the screens. Rinse you hand over the screen as you remove it so that no solids are removed. It is recommended that no more than 4 screens at a time be used when the bottom screen is a 325 mesh.

(2). Pre mix the sample with water in an Erlenmeyer flask with rubber stopper by shaking and mixing. Pour and wash out the flask of all solids on the top screen.

(3). Use a small fairly hard spray of water on the top sieve for 3 minutes (when using 325 mesh this must be done in intervals to allow sufficient drainage time). Remove the top screen and follow same procedure for next screen until all are finished.

(4). Put each screen into oven at 220 °F for 45 minutes.

(5). Using 1/2" wide bristle brush, brush the dry material out of the sieves and weight immediately.

(6). Record particle size distribution as follows:

$$\frac{10 - \text{dry wt on screen}}{10g} \times 100\% = \%\text{thru screen.}$$

Notes: For 200 mesh Lignoflex we use a US 100 stacked on a 200 mesh screen.
For 325 mesh Lignoflex we use a US 100 stacked on a 200 stacked on a 325 mesh screen.

Pages 000129-000178 of Curriculum Vitae removed in accordance with the Privacy Act of 1974.

SUBMISSION END