

## GUIDELINES FOR ESTABLISHING ETHANOL PRODUCTION FACILITY QUALITY ASSURANCE AND QUALITY CONTROL PROGRAMS



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## Introduction

The Renewable Fuels Association (RFA) is the national trade association for the U.S. fuel ethanol industry. Membership is comprised of numerous ethanol producers, both large and small, as well as suppliers to the industry and other interested parties. Founded in 1981, the RFA's primary objective is to promote public policy initiatives that increase the market for fuel grade ethanol produced from a variety of feed stocks including grains, agricultural wastes, and various biomass feedstock sources.

As the ethanol industry has grown, so has the Renewable Fuels Association's areas of responsibility to its membership. Today, the RFA not only focuses on legislative/regulatory and public policy type issues but also maintains several committees and task groups to address industry needs. These committees include a technical committee to address various technical issues and to assist with technical industry publications (such as this one); plant and employee safety, environmental compliance, and co-product committees monitoring efforts in each of these respective areas. These committees are comprised of representatives of our member companies, staff, and when necessary technical consultants and other interested stakeholders. The RFA provides support for educational outreach programs through its research and education arm, the Renewable Fuels Foundation.

For many years, RFA has promoted the use of denatured fuel ethanol in all its various applications. Denatured fuel ethanol is blended in nearly all the nation's gasoline. This includes not only E10 (90% gasoline/10% ethanol), reformulated gasoline (RFG) and oxygenated fuels, but developing markets such as E15 (85% gasoline, 15% ethanol), and flex fuels such as E85 (15% gasoline/85% ethanol). This document focuses on product quality and integrity which is of the utmost importance to the Renewable Fuels Association and its members. This updated version of the RFA's Quality Assurance and Quality Control guideline has been expanded to address not only denatured fuel ethanol but also other products commonly produced at an ethanol production facility. To promote the use of quality systems, the Renewable Fuels Association has put together this information offering a discussion of specifications, sampling and sample retention, general laboratory practices and techniques and recommendations on quality assurance and quality control practices. The purpose of this document is to serve as a condensed technical reference for ethanol producers, ethanol blenders, and other interested parties who need such information.

For an expanded discussion of the importance of specifications and properties of denatured fuel ethanol, technical/laboratory personnel may wish to review RFA Publication "**Fuel Ethanol-Industry Guidelines, Specifications and Procedures.**" This document and other RFA publications are available on the RFA website at [www.EthanolRFA.org](http://www.EthanolRFA.org).

## **Guidelines for Establishing Ethanol Plant Quality Assurance and Quality Control Programs**

Product quality and integrity is important to the Renewable Fuels Association. To achieve a quality manufactured product accompanied by robust and accurate laboratory data from an ethanol plant, the Renewable Fuels Association has put together this information, which includes a discussion of methods for analyses, sampling, general laboratory procedures and techniques and instruction on quality assurance and quality control practices. Consistently providing high-quality products to the customer should be the primary goal of every Quality Assurance/Quality Control program. This document is intended to be an educational tool and information resource to outline minimum requirements for producing valid lab results and assuring denatured fuel ethanol, distillers dried grains, carbon dioxide and corn distiller's oil product quality meets the customer's expectations.

It's important to remember that a Quality Assurance program can extend past the "gate" at the production facility. There may be a need to include transportation, storage and handling requirements and procedures in the Quality Assurance/ Quality Control programs. This document should be used as a best practices reference to assist in providing the highest quality products from an ethanol manufacturing facility. The information herein was developed to complement internal company specifications, general industry specifications and customer expectations.



**TABLE OF CONTENTS**

Basic Principles for Quality Assurance	4
Basic Principles for Quality Control	4 - 5
Certification of Analysis	5 - 6
Testing Frequency	6 - 7
Sampling, Sample Retention, Sample Shipment	7 - 8
Record Keeping	8
Batch Description	8
Shipment Information, Logistics Plan	9
Regulatory Inspections, Customer Audits	10
Product Quality Discussion:	
Denatured Fuel Ethanol Specifications	11
Individual State Specifications	11 - 12
Fuel Ethanol as the Primary Fuel Component	12
Example Certification of Analysis – Denatured Fuel Ethanol	13
Animal Food Co-Product Specifications	14
Association of American Feed Control Officials (AAFCO)	14
Labeling	15 - 16
Example Feed Tag – Corn Distillers Dried Grains with Solubles	17
Example Feed Tag– Corn Distillers Oil	17
Recommended Standard Test Methods for DDGS	17
Food Safety Modernization Act	18
Carbon Dioxide Specification	19
Annex:	
International Product Quality Considerations	20
Statistical Approach to Testing Frequency	21
Statistical Confidence Interval Analysis	22
Process Susceptibility Analysis	23
Additional References	23

## **BASIC PRINCIPLES OF QUALITY ASSURANCE**

Quality Assurance (QA) is a set of operating principles that, when strictly followed during plant processing and operations, ensure a product will be produced meeting the expectations of the customer. The program should provide assurance of compliance to any applicable quality expectations and regulatory requirements. The Quality Assurance program has the primary function of setting operating expectations, to ensure the process operates within defined conditions and limits. One essential function of the QA program is the nomination of a Quality Manager, whose sole function is to manage and improve the process. The Quality Manager is responsible for development of:

- ✓ Facility quality manual which defines roles and responsibilities,
- ✓ Document control program,
- ✓ Raw material specifications,
- ✓ Finished product specifications,
- ✓ Standard operating procedures that include corrective action expectations, and
- ✓ Management commitment to quality.

*Quality Assurance: A program for the systematic monitoring and evaluation of the various aspects of a project, service, or facility to ensure the standards of quality are being met.*  
-Merriam Webster

An established routine Quality Assurance program applied in each facility provides the basis for a controlled process. An effective quality assurance program can provide financial benefits from running a process within the operational guidelines. An effective Quality Assurance program can also provide a level of confidence in regulatory requirements and a customer inquiry into the company's manufacturing capabilities.

## **BASIC PRINCIPLES OF QUALITY CONTROL**

Quality Control (QC) is an analysis methodology used to assure a process is in control. Although all analysts practice quality control somewhat instinctively, under actual working conditions a sufficiently detailed QC program may be neglected. Quality Control Programs ensure that the process is running as designed and that all analytical methods provide results that are within acceptable accuracy and tolerance limits. Through various process checks such as sampling methods, production representative samples confirm that laboratory analyses are reliable, and verify that the reported results are suitable to support decisions. Each manufacturing location should have a written Quality Assurance Plan while each laboratory should have a written Quality Control Plan. These plans should include details of program management, standard operating procedures, process and product specifications and ultimately planned details for process assessment. The Quality

*Quality Control: An aggregate of activities (as design analysis and inspection for defects) designed to ensure adequate quality especially in manufactured products.*  
-Merriam Webster

Control plan should describe sampling and analytical procedures and frequencies and procedures for addressing out of specification (OOS) instances. The Quality Control plan should detail the process for generating Certificates of Analysis for finished products. The laboratory methods used should be described in detail, such that an experienced analyst unfamiliar with the method can obtain acceptable results. The lab must be staffed appropriately with trained, educated personnel with access to the necessary reference materials.

Both industry accepted standard specifications and customer specifications are updated on a regular basis and it must be verified that the most current version is the appropriate version that is guaranteed to the customer or required by a regulating agency. See the Annex for a useful reference list of general industry accepted standard specifications.



Initially, the QC program should continually monitor the reliability (accuracy and precision) of the results being reported. It should answer the question, "How good (accurate and precise) are the results obtained?" This function is the determination of quality. The second function is the control of quality to meet the program requirements for reliability. Each analytical method has a rigid protocol. Similarly, QC associated with a test must include definite required steps for monitoring the test and insuring results are

correct. The steps in QC vary with the type of analysis. For example, in titration, standardization of the titrant on a frequent basis is an element of QC. In any instrumental method, calibrating and checking of instrument response are also QC functions. All the testing variables that affect the final results should be considered, evaluated and controlled. Each laboratory should have a written program for instrument operation and calibration activities. Calibration checks and samples should be documented. Each laboratory should provide a program for data verification and validation. This can be accomplished by running known samples or through participation in inter-laboratory cross check programs.

### **CERTIFICATE OF ANALYSIS**

A means of communicating product quality to the ultimate customer is by providing a Certificate of Analysis for the lot of material the customer is purchasing. Certificates of Analysis (COA) can be considered a legal document ensuring compliance to the agreed upon quality standard. Another valuable means of communicating product quality is a Certificate of Conformance (COC.) The two documents are very similar with subtle differences.

A Certificate of Analysis certifies the actual product analysis results from a specific container, batch or shipment of product; a Certificate of Conformance certifies the product will conform to specified product quality but does not include actual analytical data from a specific container, batch or shipment of product.

The content of a Certificate of Analysis can vary from product to product and customer to customer. There may be regulatory requirements that must appear on a COA, such as statement of "food grade" or



- ✓ **Company name, address, manufacturing location,**
- ✓ **Lot or batch identification,**
- ✓ **Product name, and possibly product code or number,**
- ✓ **Date of analysis,**
- ✓ **Test methods and results as performed, and**
- ✓ **Signature of the person responsible for ensuring plant quality or person performing the analysis.**

It may be helpful to provide contact information should any questions arise from the customer. In many instances, testing frequency should be indicated next to the test parameter and if any typical properties are listed it must be indicated as such. Clear instructions on how to correctly complete a Certificate of Analysis will provide for the utmost accurate and truthful communication to the customer. COAs may be duplicated and issued on a time-based approach (daily, shift wise, etc.) or process conditional approach (batch, shipment, etc.).

It is good practice to keep a copy of all Certificates of Analysis and Certificates of Conformance generated; Certificates of Analysis are official product quality records. Follow company policy for record retention specifics. See example on page 13.

To ensure fuel compliance to the specification, testing frequency should be based on a risk assessment of the process operations and variability. Processes that are tightly controlled through Standard Operation Procedures, have thorough raw material and process aid purchase specifications, and have a robust quality monitoring program to assist in the decision-making process are at lower risk and would allow a reduced testing frequency. There may be parameters listed in the specification which are more “dynamic” than other requirements and would therefore need to be on an increased monitoring and testing frequency. There may be parameters not inherent to the facility operations that may be tested less frequently due to laboratory instrumentation limitations or economic reasons. Each requirement of the specification is expected to comply for every shipment and ultimate proof of compliance is the

responsibility of the manufacturing location. Reduced testing frequencies must be justified and documented in the quality assurance manual to ensure the product complies during the everyday variations of the process.

Anytime a manufacturing location undergoes process equipment modifications, process changes or upsets, including a change in raw material or processing aids quality or supplier, it is recommended that the specified properties be tested more frequently to ensure compliance. A significant change should trigger a new risk assessment to establish appropriate testing frequency. This will establish confidence that the changes made to the process have not adversely affected compliance with property limits. There must be evidence that the process and the product being produced by the process meet the specifications.

A recommended statistical approach to determining reduced testing frequency is listed in the Annex.

### **SAMPLING, SAMPLE RETENTION, SAMPLE SHIPMENT**

Employees should be properly trained in the various sampling techniques, specific to each type of product being sampled, and whether the sample is for analysis or retention, for internal or external use, or being sent to a customer or regulatory agency for evaluation. The chemical or physical characteristics of the sample must be evaluated to ensure the safe handling and choice of sample container is appropriate to the product. Many times, there are regulatory requirements pertinent to the product sample such as safety precautions and hazardous materials training. The U.S. Department of Transportation requires specific packaging for hazardous materials; see §49 Code of Federal Regulations.

Another aspect of sampling is the type of sample desired, whether that is a running production sample, finished product sample or a sample representative of specific or multiple batches. The procedure for sampling products can be an in-house procedure found in the quality manual or a standard procedure from an independent standards organization, such as an ASTM International. Common examples of sampling procedures are:

- ✓ *ASTM D4057 Standard Practice for Manual Sampling of Petroleum and Petroleum Products* can be utilized for sampling denatured Fuel ethanol. <https://www.astm.org/Standards/D4057.htm>  
Additional sampling information is also covered in *ASTM D4806 Standard Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel* <https://www.astm.org/Standards/D4806.htm>
- ✓ United States Department of Agriculture Grain Inspection, Packers and Stockyards Administration Federal Grain Inspection Service, Grain Inspection Handbook - Book I Sampling; [https://gipsa.usda.gov/fgis/handbook/BK1/BookI\\_2015-09-18.pdf](https://gipsa.usda.gov/fgis/handbook/BK1/BookI_2015-09-18.pdf)

The documented sample management plan should identify collection, frequency and documentation procedures. The plan should also provide for retaining individual samples. Retention time should be based on consumption statistics for the product. For example, if a shipment of denatured fuel ethanol will not reach the customer for 60 days, a 90-day sample retention timeframe should be ample time to



ensure a representative sample is available in the event of any questions arising from the original batch. Proper labeling is important for each sample for future identification. Sample information should include: date the sample was obtained, product identification, sampling location, lot/ batch identification (if necessary), and sampler's initials.

Denatured Fuel Ethanol samples, typically less than one-gallon quantities, needing to be shipped or transported from the production facility are regulated under the Hazardous Material Regulations in 49CFR172 for ground transport. Anyone involved in the packaging and shipment of hazardous materials must be trained and certified through an approved training program.

## **RECORD KEEPING**

Each producer should establish and maintain a written procedure that describes the company policy on record keeping. A general list of each type of record to be kept, location of storage, retention times for each specific record type and the individuals/positions responsible for maintaining the records should be included in the record keeping policy. Many times, the record keeping policy can be communicated on the document or fillable form to be kept. Records should be maintained in a neat and orderly fashion to allow easy access. Electronic storage of records may be an efficient means of storage. The record file names should be documented for future access. The computer system storing the records must be able to collect, index and file data as it's entered and the data integrity protected via user login and passwords.

A "record" may include production and/or laboratory data, raw data, Certificates of Analysis, raw material receipts, etc. Training and training completion documents may also be considered records subject to a retention policy.

Regulatory agencies may have a record keeping requirement specific to their area of jurisdiction; further agencies with a record keeping requirement may have differing retention time for storage for records. Refer to the each regulating agency for specific storage requirements for not only what types of records must be kept but also for the specific length of time.

## **BATCH DESCRIPTION**

Each producer should establish and maintain a batch/lot or similar system of production identification. The batch/lot should be segregated, then sampled and tested with the analytical procedures outlined in Quality Control program. The results of the approved batch/lot should be documented through a Laboratory Information Management program or a COA. This documentation provides for traceability of the product quality from the process and should be maintained according to company document retention standards.

A robust batch/lot identification system allows for product traceability that is accurate.

## **SHIPMENT INFORMATION, LOGISTICS PLAN**

Product quality and integrity must be maintained from the production facility through to the destination, ultimately the customer. Commonly, product quality may be the responsibility of the manufacturing company until reaching the customer, which could be a storage location, another manufacturing facility or a consumer at retail. Whether the product is being transported via truck, rail, barge or pipeline, a logistics plan must be formulated and have requirements pertinent to maintaining product quality and integrity. Many quality assurance plans include details on the varying modes of transportation and maintaining the desired product quality of the customer.



There may be regulatory requirements that impact the logistics plan, for example:

- For Hazardous materials (e.g. denatured fuel ethanol) shipping vessels should be compliant with the regulatory requirements of the Department of Transportation Hazardous Materials Shipping Requirements. These requirements are found in 49 CFR Chapter 1. Another aspect for consideration is to ensure the transportation mode/type selected is capable of being sealed and the prior contents of the vessel will not contaminate, degrade or otherwise make the product unsuitable for the ultimate application. When shipping denatured fuel ethanol products by truck or rail, the U.S. Department of Transportation requires shippers to be registered according to the details in 49 CFR 107-109. Be sure to incorporate company securement requirements for sealing vessels. Furthermore, a Safety Data Sheet should be available for all hazardous materials along with the Bill of Lading, COA, or other shipping documents.
- For products carrying a sanitary designation, such as “food grade” or “feed grade”, ensure the products are stored and transported under conditions that will protect the product from physical and chemical contamination. Food and feed product storage practices should minimize the potential for contamination and provide for appropriate inventory rotation, “first in, first out” (FIFO) inventory practices, etc. All transport vessels should be inspected at the load-out area prior to loading to insure the vessel is free of contamination.



To assure quality product at delivery it may be necessary to maintain a dedicated transportation fleet. If the transportation fleet is not dedicated, third party contract carriers should be apprised of the company expectations for appropriate prior backhauls, and proper cleanout procedures. The same communication is needed for non-dedicated transportation vessels. RFA has published information specific to denatured fuel ethanol on backhaul considerations, cleaning instructions and transportation equipment in general in the publication **“Fuel Ethanol Industry Guidelines, Specifications, and Procedures.”** This publication is available on the RFA website, [www.EthanolRFA.org](http://www.EthanolRFA.org).

## **REGULATORY INSPECTIONS, CUSTOMER AUDITS**



Regulatory inspections and customer audits provide an opportunity to showcase the business, production facility and employees. Planning and having a procedure for handling inspections leads to a successful net result. Always follow three basic rules when hosting either regulatory officials or customers at your facility, production site or office to ensure the best possible outcome from the discussion, inspection and facility tour.

1. Be prepared.
  - a. When setting up compliance systems, consider what compliance documentation an inspector or customer may seek and whether you can provide it.
  - b. Properly organize pertinent records.
  - c. Separate records not subject to inspection authority, such as privileged communications, from other compliance documents.
  - d. Understand the scope of the agency's inspection authority, what to expect in an inspection, how the company expects personnel to conduct themselves, how to react if the inspector finds a problem, who to call and when to call them.
  - e. In advance of an announced inspection, take the opportunity to conduct a pre-audit or inspection and pursue corrective action, if appropriate.
2. Follow the internal procedures that you have set up.
3. Remain calm and be respectful.

Inspectors must furnish credentials upon arrival at the facility; be sure to verify their identity prior to proceeding with any discussion or tour of the facility. Ahead of the inspection, ask for any audit documents or questionnaires that may be helpful prior to the inspector or customer's arrival at the facility. Take contemporaneous notes during the audit to memorialize the details of the discussions and any concerns that were expressed. If corrective action is warranted, pursue it promptly.

## Product Quality Discussions

### DENATURED FUEL ETHANOL SPECIFICATIONS

In the United States, the most common standards for denatured fuel ethanol used in motor fuel are the consensus standards developed by ASTM International. These standards are:



- Denatured Fuel Ethanol: **ASTM D4806** *Standard Specifications for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel*
- Denaturant and Hydrocarbon Blendstocks: **ASTM D8011** *Standard Specification for Natural Gasoline as a Blendstock in Ethanol Fuel Blends or as a Denaturant for Fuel Ethanol*
- Ethanol Flex Fuel: **ASTM D5798** *Standard Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines*
- Mid-level Ethanol Fuel Blends: **ASTM D7794** *Standard Practice for Blending Mid-Level Ethanol Fuel Blends for Flexible-Fuel Vehicles with Automotive Spark-Ignition Engines*

These specifications are available from ASTM International electronically or hard copy at [www.astm.org](http://www.astm.org).

*ASTM D4806 Standard Specification for Denatured Fuel Ethanol for Blending with Gasolines for use as an Automotive Spark-Ignition Engine Fuel* describes the necessary quality for fuel ethanol as a blend component for various unleaded gasolines. This standard is used primarily as the basis for fuel ethanol transactions between the buyer and the seller as well as Federal and State regulatory requirements. There may be agreements made beyond the content of the ASTM D4806 standard and those details are established through contractual language or purchase orders. As the ASTM specification has very detailed information regarding the importance and effect on total fuel quality, testing frequencies are not defined. It is the recommendation of the RFA that each individual production facility evaluate their own process and ability to meet the quality criteria. There are conscience decisions to be made on product quality, such as the nature of the raw material and processing aid quality expectations, any inherent effect each process step has on the finished fuel quality, and any regulatory requirements such as the Alcohol and Tobacco Tax and Trade Bureau (TTB). Plants that have established a fuel profile that indicates continual compliance with a specific property may elect to reduce testing frequency.

### INDIVIDUAL STATE SPECIFICATIONS

The D4806 specification is considered a complete fuel component specification, however there may be other regulatory requirements, such as individual state specifications that may need to be adhered to. It is also important to note that individual state fuel regulations may not adopt the most current version available of the ASTM standard and should be referenced individually.

For example, the State of California Air Resources Board (CARB) has passed legislation governing the specifications of denatured fuel ethanol when blended into California Reformulated Gasoline, California Code of Regulations, Title 13 Motor Vehicles, Division 3 Air Resources Board, Chapter 5 Standards for Motor Vehicle Fuels, Article 1 Standards for Gasoline, Sub article 2 Standards for Gasoline, *Section 2262.9 Requirements Regarding Denatured Ethanol Intended for Use as a Blending Component* in California Gasoline for the most current information.

<b>California Denatured Ethanol Standards (In addition to the Performance Requirements of ASTM D 4806)</b>		
<b>Property</b>	<b>Specification Limit</b>	<b>Test Method</b>
Sulfur, ppm max.	10	ASTM D5453
Benzene, vol. %, max.	0.06	ASTM D7576
Olefins, vol. %, max.	0.05	ASTM D7347
Aromatics, vol. %, max.	1.7	ASTM D7576

Additionally, the State of California places limits on the denaturants used to denature ethanol that is blended into their gasoline. There requirements are set forth in the following table.

<b>California Denaturant Standards</b>		
<b>Property</b>	<b>Specification Limit</b>	<b>Test Method</b>
Benzene, vol. %, max.	1.1	ASTM D 5580
Olefins, vol. %, max.	10	ASTM D 6550
Aromatics, vol. %, max.	35	ASTM D 5580

## **FUEL ETHANOL AS THE PRIMARY FUEL COMPONENT**

*ASTM D5798 Standard Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines* describes the necessary quality for fuel ethanol as the predominant fuel component. This standard is also used primarily as the basis for fuel ethanol transactions between the buyer and the seller. There may be agreements made beyond the content of the D5798 standard and those details established through contractual language or purchase orders. The following table describes the testing parameters with limits as well as suggested test methods. As the ASTM specification has very detailed information regarding the importance and effect on total fuel quality, testing frequencies are not defined. It is the recommendation of the RFA that each individual production facility evaluate their own

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process and ability to meet the quality criteria. There are conscious decisions to be made on product quality, such as the nature of the raw material and processing aid quality expectations, any inherent effect each process step has on the finished fuel quality, and any regulatory requirements such as Alcohol and Tobacco Tax and Trade Bureau (TTB).

Each batch/lot/shipment of DFE should be represented by a Certificate of Analysis. An example is provided below.

<b>CERTIFICATE OF ANALYSIS</b>				
<b>Denatured Fuel Ethanol</b>				
Manufacturing Location				
Address				
City, State, Zip				
<b>Product Loading Date:</b> <u>SHIPMENT DATE HERE</u>		<b>Batch / Lot Number:</b> <u>INSERT NUMBER HERE</u>		
<u>Quality Parameter</u>	<u>Result</u>	<u>Limit</u>	<u>Method</u>	<u>Note</u>
Ethanol, % by volume, minimum	96.62	92.1	ASTM D5501	
Methanol, % by volume, maximum	0.00	0.5	ASTM D5501	
Water, % by mass, maximum	0.50	1.26	ASTM E1064	
Acidity ( <i>as acetic acid</i> ), % by mass, maximum	0.0023	0.0070	ASTM D7795	
pHe	7.40	6.5 - 9.0	ASTM D6423	
Denaturant Content, % by volume	2.2	1.96 - 3.0	metered	③
Inorganic Chloride, mg/kg, maximum	< 2	6.7	ASTM D7319	
Existent Sulfate, mg/kg, maximum	0.63	4	ASTM D7319	
Copper, mg/kg, maximum	<0.02	0.1	ASTM D1688	①
Sulfur, mg/kg, maximum	2.0	10.	ASTM D5453	②
Solvent washed gum, mg/100mL, maximum	<0.2	5	ASTM D381	①
Benzene, % by volume, maximum	0.03	0.06	ASTM D7576	②
Aromatics, % by volume, maximum	0.06	1.7	ASTM D7576	②
Olefins, % by volume, maximum	<0.05	0.5	ASTM D7347	②
Appearance	Clear & Bright			
<p>① Performed monthly by an independent third-party laboratory (last updated: <u>insert date</u>)</p> <p>② Vendor supplies Certificate of Analysis and results are calculated based on denaturant content.</p> <p>③ Certified Ethanol Denaturant suitable for use in the manufacture of denatured fuel ethanol meeting EPA Tier 3 standards and CARB specifications for fuel ethanol denaturants.</p>				
<p><b>Signature or Name:</b> _____ <b>Title:</b> Lab Manager or Analyst</p>				



## **ANIMAL FOOD CO-PRODUCT SPECIFICATIONS**

An important co-product from the grain ethanol production process is the simultaneous production of highly nutritious animal foods. The grain ethanol industry provides an enormous contribution to the global animal food supply. In fact, one-third of every bushel of grain that enters the ethanol process is enhanced and returned to the animal food market, most often in the form of distiller's grains, corn gluten feed and corn gluten meal. These nutrient-dense co-products are fed to beef cattle, dairy cows, swine, poultry, and fish in nations around the world.

Two processes are primarily used to make ethanol in the United States: **dry milling** and **wet milling**. Both the wet and dry mill processes utilize only the starch portion of the grain for ethanol production. The remaining protein, fat, fiber and other nutritional components remain available for use as animal food. Examples of several different types of animal feed co-product produced at ethanol production facilities:

Typical Animal Food Co-Products from the U.S. Grain Ethanol Industry	
<b><u>Dry Mill Process</u></b>	<b><u>Wet Mill Process</u></b>
Distillers Dried Grains with Solubles (DDGS)	Corn Gluten Feed (CGF)
Distillers Dried Grains (DDG)	Corn Gluten Meal (CGM)
Wet Distillers Grains with Solubles (WDGS)	Heavy Steep Water
Condensed Distillers Solubles (CDS)	Corn Germ Meal
High-Protein DDGS	Crude Corn Oil
Germ Meal	
Distiller's Oil	

These co-products are differentiated by the level of processing and amount of water removed from the valuable coarse grain stillage remaining after ethanol distillation. The ultimate application or market for the varying animal food co-products is determined by their nutritional value.

It is recommended for production facilities understand the co-products quality specifications and parameters, and conduct a quality assurance program for sampling and analysis of the nutrient profile and mycotoxins.

## **THE ASSOCIATION OF AMERICAN FEED CONTROL OFFICIALS**     <https://www.aafco.org/>

The Association of American Feed Control Officials (AAFCO) is a voluntary membership association of local, state and federal agencies charged by law to regulate the sale and distribution of animal foods. Although AAFCO has no regulatory authority, the Association provides a forum for the membership and industry representation to achieve three main goals:

- ✓ Safeguarding the health of animals and humans
- ✓ Ensure consumer protection
- ✓ Providing a level playing field of orderly commerce for the animal feed industry.

These goals are achieved by developing and implementing uniform and equitable laws, regulations, standards, definitions and enforcement policies for regulating the manufacture, labeling, distribution and sale of animal feeds.

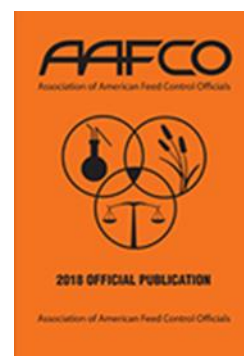
## **LABELING**

A Certificate of Analysis is rarely issued for an animal food, instead they are labeled. States have varying laws, rules, and regulations, especially as to guaranteed nutrients listed.

One major emphasis of all animal food laws and regulatory programs centers on a properly manufactured product being distributed to the ultimate consumer with proper instructions detailing nutritional claims, ingredients being used, cautions, warnings and directions for use. The vehicle to accomplish the task is the label. If an animal food is properly formulated for swine yet fed to cattle, disastrous results may be expected. The label serves as a communication system between the manufacturer and the purchaser of the animal food. Effective, meaningful labeling is essential to inform the purchaser of the purpose of the animal food, its composition and how to use it. Typically, our co-products are not a sole ration they are considered animal food ingredients.

The principle components of a label for animal food ingredients includes the **Product Name**. Each animal food has its own unique name to identify it. Dry Mill and Wet Mill co-products are defined in the AAFCO manual. AAFCO first developed feed ingredient definitions for “Distillers Products” in 1960s.

- 27.4   \*   Distillers Dried Solubles (1964)
- 27.5   \*   Distillers Dried Grains (1964)
- 27.6   \*   Distillers Dried Grains w/Solubles (1964)
- 27.7   \*   Condensed Distillers Solubles (1970)
- 27.8 Distillers Wet Grains (1982)
- 33.10        Distillers Oil, Feed Grade (2015)



\* The predominating grain becomes the first word of the definition. For example, Corn Distillers Dried Grains with Solubles.

A **Guaranteed Analysis** is required by law to advise the user of the composition of the feed. Guarantees must be determinable by recognized laboratory methods. Most are labeled with the minimum level of crude protein and crude fat and the maximum level of crude fiber.

**Typical Label Guarantees for DDGS:**

- Minimum percentage of Crude Protein
- Minimum percentage of Crude Fat
- Maximum percentage of Crude Fiber
- Maximum percentage of Ash Content



**Typical Label Guarantees for Distillers Oil:**

- Minimum percentage of Total Fatty Acids
- Maximum percentage of Insolubles
- Maximum percentage of Moisture
- Maximum percentage of Free Fatty Acids
- Maximum percentage of Unsaponifiables



It should be noted that Corn Distillers Oil is the only current co-product that has nutrient guarantee imposed by the AAFCO definition. Other animal food co-products nutrient guarantees are determined by the manufacturer.

Also on included on the label is the **Directions for Use** to inform the purchaser of how the manufacturer intends for the feed to be used.

The label also contains **Manufacturer Identification** which identifies the name and principle mailing address of the manufacturer or the person responsible for distributing the commercial feed.

## Feed Tag Examples:

**CORN DISTILLERS OIL,  
FEED GRADE**

**GUARANTEED ANALYSIS**  
*as is basis*

Moisture	%, Max	1.0
Free Fatty Acids	%, Max	15.0
Total Fatty Acids	%, Min	85.0
Unsaponifiables	%, Max	2.5
Insolubles	%, Max	1.0

**Manufactured By:**

**XYZ Feed, Inc.**

**1234 Main Street**  
**Anytown, Kansas 56789**  
**(555) 555-5555**

**CORN DISTILLERS DRIED  
GRAINS WITH SOLUBLES**

**GUARANTEED ANALYSIS**  
*as is basis*

Crude Protein	%, Min	27.0
Crude Fat	%, Min	6.0
Crude Fiber	%, Max	15.0
Ash Content	%, Max	5.0

**Manufactured By:**

**XYZ Feed, Inc.**

**1234 Main Street**  
**Anytown, Kansas 56789**  
**(555) 555-5555**

This product is intended as an animal food  
ingredient for further manufacturing and not  
fed as a sole ration

**RECOMMENDED STANDARD TEST METHODS FOR DDGS**

To address this important ethanol industry issue, an ad hoc committee performed An Evaluation of Analytical Methods for Analysis of Dried Distillers Grains with Solubles and made recommendations for standardized testing procedures for distiller's grains co-products. Since 2007 the industry recommends using the following National Forage Testing Association and AOAC International® methods as the most accurate methods for determining various nutrient compounds typically evaluated in the DDGS market.

**DDGS Analytical Test Method Recommendations**

Property	Test Method	Description
Moisture	NFTA 2.2.2.5	(105 °C for 3 hours)
Crude Fat	AOAC® Official Method 945.16	(petroleum ether extract)
Crude Protein	AOAC® Official Method 990.03	(combustion method)
Crude Protein	AOAC® Official Method 2001.11	(copper catalyst)
Crude Fiber	AOAC® Official Method 978.10	(F.G. Crucible method)

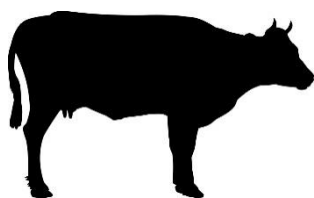
Crude Protein methods are statistically similar and either is acceptable for use on DDGS

## **FOOD SAFETY MODERNIZATION ACT**

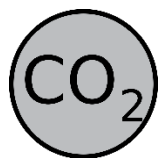
While industry is ultimately responsible for the quality of the products they manufacture, the U.S. Food and Drug Administration (FDA) is responsible for safeguarding the food supply including animal food.

The FDA Food Safety Modernization Act (FSMA) was signed into law on January 4, 2011 to better protect human and animal health by helping to ensure the safety and security of the food supply. The final rule for facilities producing animal food was published in September 2015 requiring animal food facilities to comply with Current Good Manufacturing Practices (CGMP) requirements and to have a written Food Safety Plan in place that includes a Hazard Analysis and Preventive Controls for foreseeable hazards. Once a facility has identified a preventive control for a hazard, the facility must ensure the effectiveness of the control with oversight and management of those controls. More information can be found at the FDA website: <https://www.fda.gov/Food/GuidanceRegulation/FSMA/ucm366510.htm>

The objectives of an animal food regulatory program are many. First and foremost is to protect the food supply. Products must be manufactured properly, free of adulterants or contaminants, and labeled properly so that they may be used effectively and safely.



## **CARBON DIOXIDE SPECIFICATIONS**



Another valuable co-product from ethanol fermentation is carbon dioxide. Carbon dioxide has a wide variety of end-use applications ranging from beverage carbonation, a pressurizing agent for enhanced oil recovery, and as a solid for dry ice applications. With such a wide range of uses by the marketplace, the specifications for carbon dioxide vary greatly as well. Specifications, handling and certificate of analysis expectations are published by specialty industries such as the International Society of Beverage Technologists (ISBT) and independent standards organizations such as the U.S. Pharmacopeia Convention's Food Chemical Codex (FCC). The FCC is a compendium of internationally recognized standards for the purity and identify of food ingredients; the FCC includes a monograph describing purity requirements for carbon dioxide for food applications.

Each batch / lot /shipment should have an associated Certificate of Analysis. An example of the parameters associated with beverage carbon dioxide quality and test methods utilized is shown below.

### **Carbon Dioxide**

<b><u>Quality Parameter</u></b>	<b><u>Limit</u></b>	<b><u>Method</u></b>
Purity, v/v%	> 99.9%	ISBT 2.0
Water, v/v%	< 20 ppm	ISBT 3.0
Oxygen, v/v%	< 30 ppm	ISBT 4.0
Carbon Monoxide, v/v%	< 10 ppm	ISBT 5.0
Ammonia, v/v%	< 2.5 ppm	ISBT 6.0
Nitrogen Monoxide (NO), v/v%	< 2.5 ppm	ISBT 7.0
Nitrogen Dioxide (NO <sub>2</sub> ), v/v%	< 2.5 ppm	ISBT 7.1
Non-volatile Residue, w/w%	< 10 ppm	ISBT 8.0
Total Hydrocarbons (THC), v/v%	< 50 ppm	ISBT 10.0, 10.1
Odor, Taste	No foreign odor or Taste	ISBT 16.0
Appearance in Water	No color or turbidity	ISBT 16.0

This product meets the Quality Guidelines for Beverage Grade CO<sub>2</sub> as listed in the International Society of Beverage Technologist Bulk Carbon Dioxide Quality Guidelines and Analytical Methods Reference, 2010





## **Annex**

### **INTERNATIONAL PRODUCT QUALITY CONSIDERATIONS**

Manufacturing products intended for shipment outside of the United States can be a critical element of the commercial portfolio of an ethanol production facility. Understanding the requirements, from both a legal and regulatory perspective, is key to a successful internationally based business. At the manufacturing level, plant personnel must ensure production processes, testing and release procedures meet the international customer's needs. Product quality and certification must be maintained throughout the logistics chain and until the product reaches the ultimate consumer. It is imperative that quality standards agreed to between the supplier and buyer are communicated to all parties involved in the production, transport and ultimate delivery to the final buyer. There may be additional negotiated terms in the commercial agreements for transfer of product. Examples of negotiated terms may be treatment with additives, supplier certifications, etc.

ASTM International fuel product specifications used primarily in the United States are growing in popularity among other countries. No matter the origin of the specification used in the agreement, adherence to the requirements, including specific laboratory procedures, must be completed. In some cases, laboratory methods may be proven equivalent to other procedures as published. Documentation of the analytical method equivalency should be kept on file.

In the case of exporting a food or feed product, there may be a requirement to provide a phytosanitary certificate. Phytosanitary certificates are official documents which certify that the plants or plant products have been officially inspected, are free from quarantined pests, are practically free from other injurious pests and conform to the current phytosanitary regulations of the importing country. Each importing country determines its own phytosanitary regulations.

## **STATISTICAL APPROACH TO TESTING FREQUENCY**

The testing frequency required to ensure and prove fuel ethanol compliance to the respective analyte specifications in D4806 should be based on a *statistical confidence interval analysis* coupled with a *process susceptibility analysis* for each analyte.

## **STATISTICAL CONFIDENCE INTERVAL ANALYSIS**

The statistical confidence interval (CI) analysis must contain a minimum of 20 samples per analyte spanning a period of no less than two months of operation. The mean, standard deviation, and mean +/- (depending on whether the specification is a minimum or a maximum) one, two, three, four, five, and six standard deviations for each analyte should be calculated and entered Table 1 below:

**Table 1**

Analyte	
Units	
Mean	
Standard Deviation	
(+/-) 1 Std Dev (84.1% CI)	
(+/-) 2 Std Dev (97.6% CI)	
(+/-) 3 Std Dev (99.8% CI)	
(+/-) 4 Std Dev	
(+/-) 5 Std Dev	
(+/-) 6 Std Dev	
D4806 Spec	
Min/Max	

The point at which the mean  $\pm$  a multiple of the standard deviation exceeds or goes below the specification (depending on whether the specification is a minimum or a maximum) should be highlighted. An example including two different analytes is provided in Table 2 below:

**Table 2**

Analyte	Solvent-washed gum	Ethanol Purity
Units	mg/100 mL	volume %
Mean	1.3	95.8
Std Deviation	0.30	1.1
( $\pm$ ) 1 Std Dev (84.1% CI)	1.6	94.7
( $\pm$ ) 2 Std Dev (97.6% CI)	1.9	93.6
( $\pm$ ) 3 Std Dev (99.8% CI)	2.2	92.5
( $\pm$ ) 4 Std Dev	2.5	91.4
( $\pm$ ) 5 Std Dev	2.8	90.3
( $\pm$ ) 6 Std Dev	3.1	89.2
D4806 Spec	5.0	92.1
Min/Max	Max	Min

If a statistical confidence interval of the mean  $\pm$  (depending on whether the specification is a minimum or a maximum) six standard deviations (six sigma) is not achieved, then it should be considered required to measure that analyte on an individual batch basis to ensure and prove compliance. In the example provided in Table 2 above, the ethanol purity mean minus four standard deviations is below the ASTM minimum specification of 92.1; therefore, it would be required to test each batch of ethanol for purity before releasing product. The other analyte in the example, solvent washed gum, has a value of 3.1 when taking the mean plus six standard deviations which is still below the ASTM maximum specification of 5.0. Therefore, in this case, six sigma statistical compliance has been achieved and only periodic third-party oversight testing is required to ensure and prove compliance.

## **PROCESS SUSCEPTIBILITY ANALYSIS**

The process susceptibility analysis involves a detailed evaluation of the process by an experienced scientist or engineer to determine if there is a high susceptibility to some upset event in the process to cause an analyte concentration to supersede historical statistical trends. In other words, it takes the before described statistical confidence interval analysis and treats it as the initial criteria that must be met to rely on oversight testing only to ensure and prove compliance and then asks if any of those parameters that were proven to be six sigma or better are prone to process upsets that would render the statistical analysis inadequate and therefore unable to predict out-of-spec product. One such example is sulfate; since many ethanol processes are susceptible to sulfate excursions that cannot be predicted by historical trends due to the use of oxygenated sulfur based chemicals in the process, it is thereby necessary in the clear majority of cases to analyze for sulfate on an individual batch basis to ensure and prove compliance to the 4 mg/Kg D4806 maximum specification even if the facility has achieved the six-sigma requirement in the statistical confidence interval analysis.

Any process upset should trigger a thorough investigation into the possible quality impact to the finished products. During the process upset and investigation stage, more frequent analysis is recommended.

## **ADDITIONAL REFERENCES**

There are resources available to quality assurance and quality control professionals to assist with decision making that directly affects the release of final product from a production facility. Organizations like ASTM International publish standards to assist with the disposition of product. Examples of resources are:

- *ASTM D3244 Standard Practice for Utilization of Test Data to Determine Conformance with Specifications* <https://www.astm.org/Standards/D3244.htm>
- *ASTM D6299 Standard Practice for Applying Statistical Quality Assurance and Control Charting to Techniques to Evaluate Analytical Measurement System Performance* <https://www.astm.org/Standards/D6299.htm>
- *ASTM D6792 Standard Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories* <https://www.astm.org/Standards/D6792.htm>

For more information contact the Renewable Fuels Association

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Website: [www.EthanolRFA.org](http://www.EthanolRFA.org)