University of Idaho

Biodiesel Production Quality Control Plan

for the

Biodiesel Lab

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Executive members of the University of Idaho Biodiesel Education Team approved the Quality Control Plan herein.

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Attachments:
Flow Charts
Biodiesel Certificate of Analysis Form

I. EXECUTIVE SUMMARY

The University of Idaho Biodiesel Laboratory (the lab) is an education and research facility located at the James Martin Lab (JML) building on the Moscow campus. Bulk production of biodiesel produced from this lab powers campus vehicles with a batch-type reactor.

This document was developed as a directive to those making biodiesel in this lab to assure that the biodiesel produced at this facility meets nationally recognized standards and addresses processes for corrective action and the prevention of nonconformity. The biggest challenge for this lab to produce a quality fuel is for the variety of people producing the biodiesel to use a consistent, standardized process. Consequently, the bulk of this plan is a guide for procedures to produce a high-quality product while maintaining safety and protection for personnel. It is the policy of this lab that biodiesel production is done safely and the final product (biodiesel) will always meet or exceed the quality standard set by ASTM D6751.

In conjunction with standardized process quality, assurance procedures have been implemented in the industry and this lab. Quality assurance items that are instituted in this lab are:

QC Management & Audits

1. Quality control management consists of the supervisor and the manager.
2. The supervisor will be the ultimate authority on QC and biodiesel processing and will conduct an audit of the lab at least once a year by June 1 that requires signed approval.
3. The manager is the Quality Control Representative (QCR) and plays a primary role in biodiesel production and is responsible for production success and provides clear, orderly, complete, and easy assessable documentation of the following:
    a) Laboratory purchases,
    b) Laboratory equipment calibration,
    c) Feedstock log,
    d) Production reports,
    e) Test charts, and
    f) A Biodiesel Certificate of Analysis

Procurement & Equipment Calibration

4. All raw materials, consumables, spare parts and safety items will be purchased from reliable and trusted sources.
5. Laboratory equipment, used to make biodiesel or certify quality, will be calibrated as frequently as the frequency of calibration schedule provided in this document.

Feedstock

6. Different feedstocks can be used for bulk biodiesel production but the supervisor, along with any additional procedures the supervisor deems necessary, must be employed for feedstocks that are not commonly used.
7. Documentation of the feedstock used for bulk production will be made easily accessible and retained for no less than two years.
8. The feedstock used for biodiesel will be filtered with a 100-micron filter.
9. The oil will be dried until there is no more than 0.05% (500 ppm) of water.
10. If FFA levels exceed 3%, the QMR will need to receive instructions from the supervisor on how to proceed.
11. A regiment of measuring the sulfur content of the feedstock will be instituted if the biodiesel does not meet the sulfur limit. This regiment will continue until the issue is fully resolved.

Production

12. Prior to starting a bulk production batch, pumps, hoses, containers, and anything else that comes in contact with the ingredients must be flushed or cleaned.
13. The alcohol used is methanol of industrial grade with a purity of at least 99.9%. If there is any concern for its quality, methanol will be tested for water content using Karl-Fisher Coulometer and have no more than 1,000 ppm of water.
14. All bulk production of biodiesel must follow all of the steps and procedures described herein.
    a) The reactor lid is closed and temperature of the oil in the reactor is below 65°C. The methanol condenser vent tube is installed and turned on.
    b) 80% of the methanol used in the calculation is pumped into the reactor below the surface of the oil.
    c) Using the value derived from the calculation, 80% of the catalyst solution is added to the reactor.
    d) The reactor is maintained at 55-60^{o}C and the reaction mixture is mixed vigorously for at least one (1) hour for the first reaction.
    e) After the first reaction, the agitation is turned off to allow the phase separation of biodiesel and glycerol for at least two (2) hours before the glycerol is removed.
    f) The remaining 20% of the alcohol and of the catalyst solution are then added using the same method described above.
    g) Water washing is done by turning off the heat to the reactor and pumping water at one-quarter (¼) of the amount of fuel through the perforated tube above the surface of the biodiesel under gentle mixing.
    h) This process of raining water under gentle agitation, separation and draining are repeated until the soap content is below 5 ppm. Once the soap is below 5 ppm, the mixer is then turned off to allow separation of grey water and the grey water is removed.

i) The biodiesel is dried until the water level is below 500 ppm.
j) An antioxidant is added at a dose of 200 ppm and the oxidative stability is measured. If this amount of additive does not result in an oxidative stability reading of 3 hours or more, an additional 200 ppm will be added and the fuel retested.
k) A series of tests are made on the finished product to certify it meets the ASTM biodiesel standards. If the fuel fails any test, attempts will be made to rectify the situation.
l) Once the fuel has been shown to meet the standards, a biocide additive is added to the fuel at a dosage prescribed by the maker.
m) The finished biodiesel is allowed to cool down to a minimum of 30°C, then pumped into clean and dry storage containers. Full storage containers are sealed and labeled.

Testing & Sampling

15. The finished biodiesel will be tested in accordance with the most current biodiesel standard (ASTM D 6751). A copy of the most current ASTM Biodiesel Standard will be maintained in the testing lab.
16. Testing of the biodiesel will be performed on each production lot. In no event will biodiesel be used for vehicular use unless it meets the ASTM biodiesel standards.
17. Each batch will be sampled by obtaining representative samples from the reactor for testing and retained samples (retains) in accordance with the ASTM D-4057 Standard Guide for the Sampling of Petroleum Products, which will be maintained in the testing lab.
18. Retains shall be kept in a well-ventilated, cool, dark location to avoid spoilage, and labeled as a "Retain" with the lot number. Samples will be retained for 60 days, or the estimated life of the product.
19. A series of critical tests as identified by BQ-9000 will be made on every production lot. This is a subset of ASTM D6751:
20. The laboratory will maintain production records in a laboratory notebook documenting the analysis of QC samples and retained for a minimum of two years
21. All lots approved as biodiesel for vehicular use will be covered by a certificate.
22. QC test data will be plotted on a control chart.
23. Procedures for corrections or additions to a production test report must be approved and initialed by the supervisor.

Storage & Delivery

24. Biodiesel storage containers should be clean and dry before being filled with biodiesel.
25. A completed batch of fuel will be filtered to 5-microns.
26. Once the storage containers are filled, they will be sealed, and resealed after each use until empty.

27. All fuel going into storage will be additized with an antioxidant at a rate of 200 ppm and kept in closed containers at indoor ambient temperatures.
28. When biodiesel is being stored longer than six (6) months, it will be tested for oxidative stability every two (2) months thereafter.
29. Biodiesel that cannot pass the stability test will not be used for vehicular fuel.
30. The fuel that is dispensed from the green truck slip tank will pass through an additional 5-micron fuel filter.
31. A specific fuel delivery plan will be developed for each user.
32. Partnering with campus made fuel users means the biodiesel program will provide adequate fuel storage tank housekeeping and monitoring expertise and guidance.
33. Biodiesel will receive special handling during each winter.
34. The cloud point of the biodiesel, diesel, and blended mixture will be provided to the campus made biodiesel users by December 1 of each year.
35. Drivers, equipment operators and technicians will be educated on the use of biodiesel.

II. INTRODUCTION

The University of Idaho Biodiesel Laboratory (the lab) is an education and research facility located at the James Martin Lab (JML) building on the Moscow campus. The lab has about 2,640 square feet space with a large overhead door, and is equipped with a pilot-scale biodiesel production facility, including 2 oilseed crushers, a 400 and a 740-gallon volume capacity stainless steel biodiesel batch reactor, a flash evaporator for biodiesel polishing and methanol recovery. Pumps and mixers have TEFC motors or are pneumatic.

The facility includes two fuel testing and analytical laboratories that provide testing and analysis on oils and fuels. Capabilities include the instruments and equipment needed for testing a majority of the standards included in ASTM D6751.

As a long-time biodiesel research facility, about 30 different feedstocks have been used to produce biodiesel. Currently, the main biodiesel production feedstock is from used frying oil from one or more of the campus dining facilities. The biodiesel produced from this oil powers campus vehicles including three demonstration vehicles the department owns for its Biodiesel Education Program.

The lab generally produces biodiesel using a conventional batch system with the 400-gallon reactor. The facility is properly maintained with procedures in place for (1) the procurement and handling of raw materials and supplies, (2) the testing and possible pretreatment of the feedstock, (3) the execution of the production processes, and (4) the final testing and storage of the product before it goes out the door. The lab is strictly compliant with safety rules and regulations at all times.

This document was developed as a directive to those making biodiesel in this lab to assure that the biodiesel produced at this facility meets nationally recognized standards and addresses processes for corrective action and the prevention of nonconformity. The quality of biodiesel produced in this facility conforms to the requirements of the BQ-9000 program for biodiesel producers and in some cases exceeds BQ-9000 standards. BQ-9000 is an important quality assurance to customers that enhance the industry, but since the lab does not produce biodiesel for commercial purpose and as an education and research facility, following exact BQ-9000 such as periodical external audits would be irrelevant and hence this lab does not follow the exact protocol of BQ-9000.

This plan is a guide to be followed at the lab to ensure that sound procedures are implemented and that a high-quality biodiesel is produced while maintaining safety and protection for personnel. It is the policy of this lab that biodiesel production is done safely and the final product (biodiesel) will always meet or exceed the quality standard set by the most recent version of ASTM D6751.

III. QUALITY CONTROL MANAGEMENT & AUDITS

The management of the lab consists primarily the supervisor and the manager. The supervisor has direct authority over the manager, provides guidance to the manager regarding priorities and goals for the lab, and must approve significant process changes. The supervisor is responsible for assigning and monitoring lab work, and verifying work affecting quality. The supervisor will direct actions on any nonconformities relating to product, process and quality system. The supervisor meets other Biological Engineering faculty and the manager to discuss any significant process changes before implementation.

The supervisor will be the ultimate authority on QC and biodiesel processing in the lab and will conduct an audit of the biodiesel lab at least once a year by June 1. The audit requires verification and satisfactory approval of the process of making and delivering biodiesel used for campus vehicles and the appropriate documenting of the production. Documentation will be recorded in a specifically designated binder or file (electronic or otherwise) in the laboratory, made easily accessible, and retained for a minimum of two years for the purposes listed below.

1. Laboratory purchases,
2. Laboratory equipment calibration,
3. Feedstock log,
4. Production reports,
5. Test charts, and a
6. Biodiesel Certificate of Analysis

The manager plays a primary role in biodiesel production and is responsible for production success. The manager is designated as the quality management representative (QMR), reporting to the supervisor, and must get approval from the supervisor regarding major changes in the lab. The manager's job is to produce or direct production of biodiesel with strict adherence to this document, within the specified period, and under the established budget, while achieving the objectives. The duties of the manager include the following:

1. Obtain all the necessary materials for biodiesel production,
2. Produce or direct production of biodiesel with strict adherence to this document,
3. Dispose of by-products in a matter acceptable to the supervisor,
4. Deliver the fuel as specified in the customer biodiesel delivery plan,
5. Initiate action to prevent the occurrence of any nonconformities relating to product, process, quality system, and fuel delivery,
6. Identify and record any problems relating to the product, process and quality systems,
7. Initiate, recommend or provide solutions,
8. Verify the implementation of solutions,
9. Control further processing or delivery of nonconforming product until the deficiency or unsatisfactory condition has been corrected and
10. Provide required documentation in an orderly fashion.

IV. PROCUREMENT & EQUIPMENT CALIBRATION

All raw materials, consumables, spare parts and safety items will be purchased from reliable and trusted sources. Purchasing orders and correspondence related to biodiesel will be documented as described in section II above and be backed up by university financial services.
Laboratory equipment, used to make biodiesel or to certify quality, are listed below along with the frequency of calibration. Calibrations may include a comparison of results with a certified laboratory. Specific calibration instructions for each instrument can be found in equipment manuals or in the ASTM standard methods for each test associated with a given instrument. Equipment manuals will be kept in an easily accessible convenient location.

Instrument	***Calibration Frequency***
Weighing scale	1 year
Mettler Balance	1 year
Karl Fisher	6 months
Rancimat	1 year
Flash Point	1 year
Kinematic Viscometer	1 year
Spectrophotometer (FTIR)	1 year
Gas Chromatograph (GC)	3 months
TS 100 Sulfur Detector	6 months
Cloud/Pour point Analyzer	1 year
Vacuum Distillation System	1 year

Documentation will include the instrument calibrated (including a unique instrument identification traceable to a serial number), method or procedure used for calibration, the date of the last calibration, the person performing the calibration, the values obtained and the nature and traceability (if applicable) of the calibration standards.

Quantitative calibration standards will be prepared from constituents of known purity. Primary calibration standards or certified reference material (CRM) will be used. Where appropriate, values for reference materials should be traceable to national or international standard reference materials.

V. FEEDSTOCK & FEEDSTOCK PREPARATION

Most feedstock for biodiesel production is used frying oil from fryers at the on-campus dining facilities. Other feedstocks may be used but must follow the procedures outlined in this document. Feedstock for production will be documented in a specifically designated binder or file, made easily accessible, and retained for no less than two years. The feedstock records will include the date the feedstock was obtained, amount, and any noteworthy comments.

The feedstock/oil used for making biodiesel will first be filtered through a 100-micron filter to ensure no particles, grime, or dirt enter the processing scheme. The amount of oil needed for a production batch for this facility is described in the Production chapter of this document. The container(s) of oil is placed on the digital scale along with the pump, and tared. The pump is then turned on, and the calculated amount of oil is pumped from the oil containers into the reactor.

Excess water in the oil will result in serious processing complications that may include a complete failure of the oil to react, incomplete reactions, soap, and emulsions. It is important that water be kept out of the production process. While the process can tolerate up to 1-percent water, it should be reduced as much as possible. Higher water content will increase soap production and measurably affect the completeness of the transesterification reaction.

The reactor uses electrical band heaters for temperature control, and a hydraulic mixer and a recirculation pump for agitation. To remove water, the oil is brought up to 55-70^{o}C and agitated with the reactor lid open. The oil is dried using heat, agitation, and ventilation until there is no more than 0.05% (500 ppm) of water, which is tested using a Karl Fisher Coulometer. This usually takes about eight (8) hours. The oil temperature is then reduced to 55-60^{o}C before it is ready for transesterification.

The first test to do before making biodiesel is to measure the amount of free fatty acids (FFA) in the oil feedstock. The amount of FFA is determined by ASTM D664.The percent of FFA will be used to adjust the alkali catalyst application rate to ensure an adequate amount of catalyst is added for a complete reaction. FFA reacts with the alkali catalyst to form soap instead of biodiesel. FFA binds catalyst and slows the transesterification reaction and produces less biodiesel. Produced soap adds complication to the separation process. The oil collected from dining facilities generally contains less than 1% of FFA. In case, if FFA levels exceed 3%, the QMR will report to the supervisor and follow instructions from the supervisor before proceeding.

Measuring feedstock quality is generally limited to water content and acid value. However, some feedstocks have sulfur levels above the ASTM standard. Lowering sulfur levels before the transesterification process may be the most desirable option towards meeting the sulfur limits. If it is anticipated that the biodiesel produced in this facility will exceed sulfur limits, a regiment of measuring the sulfur content of the feedstock will be instituted. This regiment will continue until the issue is fully resolved.

VI. PRODUCTION

At this point the reactor has been filled with feedstock, water has been removed, and the FFA content is known and is below 3% by weight. The typical inputs of the transesterification process for the lab are used frying oil, methanol and sodium methoxide (CH_3ONa). The transesterification process will typically be conducted in the 400-gallon reactor. Safety operation guidelines specify that only two-thirds (⅔) of the working volume be used for this type of chemical reaction.

The four steps for the production of biodiesel in the lab are: 1) determination of the quality and quantity of methanol and catalyst needed, 2) transesterification, 3) polishing after the reaction, and 4) storage of the finished product. Prior to starting a production batch, pumps, hoses, containers, and anything else that comes in contact with the ingredients must be flushed or cleaned to avoid adding contaminants to the process.

I. Methanol and Catalyst Amounts

The alcohol used is transesterification reaction is industrial grade methanol with a purity of at least 99.9%. The purity will be assured by buying the methanol from trusted vendor, checking the packing label, storing it in a prescribed manner and using it within the dates specified. Methanol will be tested for water content using Karl-Fisher Coulometer if there is any concern of its quality. Methanol used in reaction, including methanol recovered from previous batches, shall have no more than 1,000 ppm of water. The stoichiometric molar ratio of methanol to oil in transesterification is 3:1. Since transesterification is a reversible reaction, excess alcohol is needed to push the reaction toward completion. In this production, methanol is added at a 6:1 molar ratio to the oil.

In addition to using excess methanol, a two-step (80/20) reaction strategy is used to ensure a complete reaction. Eighty percent of the methanol/catalyst is reacted, the glycerol is drained, and the remaining 20% of the alcohol/catalyst is added and reacted.
The amount of methanol needed is determined by the following equation:

$$\boldsymbol{Methanol\ Weight} = \mathbf{6} \times \frac{\boldsymbol{Oil\ Weight}}{\boldsymbol{Molar\ Weight\ of\ Oil}} \times \boldsymbol{Molar\ Weight\ of\ Methanol}$$

The molar weight of methanol is 32.04 g/mol. The molar weight of used frying oil varies, but can be determined by a fatty acid profile. The fatty acid profile for the used oil from Dining Services found the molar weight to be about 876 g/mol. Since most oil has a molecular weight close to this value and 100% excess methanol is used for the reaction, accuracy of the actual molar weight of the oil is not that critical. Therefore, unless there is an extreme change in feedstock type, the molecular weight of 876 can be used for calculating methanol weight.

The batch size for bulk production at this facility is generally determined by the amount of methanol. This is provided in a 55-gallon barrel. To convert methanol volume to weight, use methanol density as 6.6 lb/gal, unless a more accurate value is available. An example of the equation to determine the amount of oil that will be reacted follows.

OIL AMOUNT EXAMPLE

Calculation of barrel weight of anhydrous methanol:

Barrel Volume x Methanol Density = Weight of Methanol in Barrel
55 gallons x 6.6 lb/gal = 363 lb/barrel

Calculation of oil weight to consume 1 barrel of methanol:

(**Methanol Weight x Molar Weight of Oil) / (6 x Molar Weight of Methanol) = Oil Weight**
(363 lb x 876) / (6 x 32.04) = 1,655 lb

Calculation of oil volume to consume 1 barrel of methanol:

Oil Weight / Oil Density = Volume of Oil
The density of oil is 7.6 lb/gal, therefore:
1,655 lb / 7.6 lb/gal = 218 gallons of oil

A form showing this calculation will be included in the documentation for each batch.

The catalyst typically used in this laboratory for bulk production (vehicular biodiesel use) is sodium methoxide (CH_3ONa), which is typically in the form of sodium methoxide-methanol solution and is also toxic. If the catalyst to be used is not sodium methoxide (CH_3ONa), the calculations need to be revised accordingly.

The amount of catalyst solution used depends on the amount of oil and the FFA content of the oil. For sodium methoxide (CH_3ONa), the standard application rate is 0.5% of the oil weight, plus the quantity for neutralizing the FFA that is present in the oil as determined in the Feedstock and Preparation chapter. The standard recipe for CH_3ONa is shown below.

$$\frac{(\%\boldsymbol{FFA} \times \mathbf{0.19}) + \mathbf{0.5}}{\mathbf{100}} \times \boldsymbol{the\ weight\ of\ oil} = \boldsymbol{amount\ of\ sodium\ methoxide}$$

An example of the equation follows. A form showing this calculation will be included in the documentation for each batch.

CATALYST AMOUNT EXAMPLE

Using the quantity of the oil from the example above, (1,655 lb) and assuming the oil contains 1% of FFA (Note that the actual percentage of FFA, as determined in the previous chapter, must be used),

(((1 x 0.19) + 0.5) / 100) x 1,655 lb = 11.4 lb

This figure represents the weight of pure sodium methoxide. Since it comes as a concentrate in methanol, the figure must be divided by the concentration (25% or 30%). For a 25% concentration:

11.4/0.25 = 45.6 lb

Since, the density of 25% sodium methoxide (CH3ONa) is estimated as 8.5 lb/gal, this amount is approximately equivalent to:

45.6 lb / 8.5 lb/gal = 5.2 gallon of catalyst-methanol solution.

2. Transesterification

Once the reactor has been filled with the amount of pre-prepared oil necessary to react with a given amount of methanol, and a known quantity of FFAs, and calculations to determine the amount of feedstock/methanol and catalyst have been made, the next step is to get ready for the transesterification process. All bulk production of biodiesel must follow the procedures described herein.

The reactor lid is closed and temperature of the oil in the reactor is set to 55-60^{o}C for the reaction. The next step is to carefully add methanol, which is extremely toxic and highly flammable. Before methanol is added to the reactor, the oil temperature should be below 65^{o}C. If the oil temperature is being lowered, the mixture is agitated for another 20 minutes after it reaches 65^{o}C for temperature stabilization. The methanol condenser vent tube is installed and turned on. The condenser is chilled using a chiller, which pumps ethanol at -20^{o}C through a copper tubing attached to the vent tube. This forces the methanol vapor to condense and return to the reactor.

While the oil temperature is being stabilized, the barrel of anhydrous methanol is placed on the digital scale along with the pneumatic pump specifically intended for this purpose, and tared. The exhaust fan above the reactor is turned on, to remove potential methanol vapor escaping from the reactor vent. The pneumatic pump is then turned on and 80% of the methanol used in the calculation is pumped from the barrel into the reactor by a perforated tube just under the lid (see water washing photo).

The methanol barrel is then removed from the scale and is replaced by a container of the 25% catalyst solution, and tared with the pneumatic pump attached. Using the value derived from the calculation, 80% of the catalyst solution is added with the pneumatic pump in the same method used as with the methanol.

The reactor is maintained at 55-60°C, and the reaction mixture is mixed vigorously for at least one (1) hour for the first reaction. After the first reaction, the agitation is turned off to allow the phase separation of biodiesel and glycerol overnight or at least two (2) hours. The temperature control system is maintained on during the setting period. After the settling, the glycerol is removed from the bottom of the reactor and pumped into a crude glycerol barrel using an electric pump attached to the reactor.

After the glycerol is removed from the reactor, the remaining 20% of the alcohol and of the catalyst solution are then added using the same method described above. The second reaction lasts for one (1) hour at 55-60°C under agitation and recirculation. After the reaction, glycerol is again allowed to settle for at least two (2) hours and is removed from the reactor using the same process as described above.

3. Polishing after the Reaction

The biodiesel is prepared for water washing by turning off the heat to the reactor and pumping water at one-quarter (¼) of the amount of fuel through the perforated tube above the surface of the biodiesel under gentle mixing. This process of raining water under gentle agitation, separation and draining are repeated until the soap content is below 5 ppm. The mixer is then turned off to allow separation of grey water from the biodiesel. The grey water is removed with an electric pump attached to the reactor, using the same method as removing the crude glycerol from the reactor, and disposed of appropriately.

Water Washing

The biodiesel is dried by heating it to 110°C with gentle agitation until the water level is below 500 ppm. Once moisture level is below 500 ppm, an antioxidant is added at a dose of 200 ppm and the oxidative stability is measured, in accordance to AOCS method Cc 17-79. If this amount of additive does not result in an oxidative stability reading of 3 hours or more, an additional 200 ppm will be added and the fuel retested.

A series of tests are made on the finished product to certify it meets the ASTM biodiesel standards as specified in the following chapter. If the fuel fails any test, attempts will be made to rectify the situation. No fuel will be used for vehicular use unless it meets or exceeds the ASTM biodiesel standards. Once the fuel has been shown to meet the standards, a biocide is added to the fuel at a dosage prescribed by the manufacturer.

4. Storage of the Finished Product

The finished biodiesel is allowed to cool down to 30°C or lower, and then pumped into clean and dry storage containers. The storage containers typically used are 55-gallon steel barrels and when full are sealed to keep the fuel protected from oxidation. Each barrel will be clearly labeled in accordance with the Storage & Fuel Delivery chapter of this document.

VII. TESTING & SAMPLING

The finished biodiesel will be tested in accordance with the most current biodiesel standard (ASTM D 6751) to assure that it complies with the specifications. A copy of the most current ASTM Biodiesel Standard will be maintained in the testing lab.

Testing of the biodiesel will be performed on each production lot. A production lot is a homogeneous production volume of finished biodiesel. At the UI facility, a lot is the equivalent of one reactor batch of fuel and will be represented by a batch number and (end) date of production. If the fuel fails any test, the biodiesel team will be consulted to determine a reprocessing strategy. Biodiesel will not be used for vehicular use unless it meets or exceeds the ASTM biodiesel standards.

Each batch will be sampled by obtaining representative samples from the reactor for testing and retained samples (retains) in the event that additional testing is needed. Representative samples will include product taken at the upper, middle and lower regions of the reactor. Samples will be taken in accordance with the ASTM D-4057 Standard Guide for the Sampling of Petroleum Products, which will be maintained in the testing lab. Retains shall be kept in a well-ventilated, cool, dark location to avoid spoilage, and labeled as a "Retain" with the lot number. Samples will be retained for 60 days, or the estimated life of the product, whichever is longer. Each batch will be tested as described herein.

A series of critical tests will be made on every production lot. This is a subset of ASTM D6751 which includes the following tests that can be made on site:

- Acid Number
- Water and Sediment
- Free Glycerin
- Monoglycerides
- Total Glycerin
- Cloud Point
- Flash Point
- Sulfur
- Oxidation Stability
- Cold Soak Filterability

The additional tests included in ASTM D6751 will be determined at the discretion of the UI biodiesel team. These are those of which properties are not anticipated to change from batch to batch.

- Phosphorous
- Sulfated Ash
- Calcium & Magnesium
- Sodium/Potassium
- Kinematic Viscosity
- Cetane

The laboratory will maintain production records in a laboratory notebook documenting the analysis of QC samples and retained for a minimum of two years. Production laboratory notebook will be legible, identifiable and accessible. Items included in laboratory notebook will be:

1) Date of performance of test,
2) Lot number on each page of the report,
3) Identification of the test sample location (upper, middle, or lower),
4) Test calculations and results,
5) Any deviations, additions to or exclusions from the specified test requirements, and any other information relevant to a specific test,
6) Any other information which might be useful.

All lots approved as biodiesel for vehicular use will be covered by a "Biodiesel Certificate of Analysis" that accurately and clearly presents the test results, all other relevant information, and a signature of person(s) accepting responsibility for the certification and the date of issue. An example of the certificate is provided in the attachments. The certificates will be placed in a binder or file specifically designated for biodiesel certificates, made easily accessible, and retained for no less than five years.

QC test data will be plotted on a control chart and evaluated to determine if the results obtained are within the method specifications and laboratory-established control limits. The charts used should be appropriate for the testing conditions and statistical objectives. Corrective action should be taken and documented for any analyses that are out of equipment parameters.

Procedures for corrections or additions to a production test report must be approved and initialed by the supervisor.

VIII. STORAGE & FUEL DELIVERY

In the lab, the primary concerns associated with biodiesel storage and delivery are solvency, oxidative stability and low-temperature properties. Typically, the biodiesel storage containers in the lab are 55-gallon barrels. Storage containers will be clean and dry before being filled with biodiesel. Due to biodiesel's solvent effect, it will scrub off any deposits in the tank and carry them straight through to the pump and into the vehicle fuel tank. A completed batch of fuel will be filtered to 5-microns as it is transferred from the reactor into storage containers and again at the point of delivery. Once the storage containers are filled, they will be sealed and resealed after each use until empty, to protect from potential oxidation. Each storage container will be clearly labeled with the lot number and date of production.

Chemical changes in biodiesel during storage usually consist of oxidation due to contact with the air. Often these changes are accompanied by a darkening of the biodiesel color from yellow to brown and the development of a "paint" smell. All fuel going into storage will be additized with an antioxidant and a biocide and kept in closed containers at indoor ambient temperatures. When biodiesel is being stored longer than six (6) months, it will be tested for oxidative stability every two (2) months thereafter. Biodiesel that cannot pass the stability test will not be used for vehicular fuel.

Delivery of the biodiesel will be from the aluminum 100-gallon slip tank located in the 1999 Dodge green pick-up truck. The fuel that is dispensed from the green truck slip tank will pass through an additional 5-micron fuel filter.

Biodiesel is provided directly into vehicles as B100 or splash blended into vehicles or other storage tanks as B10 or B20. Since the situation is different for each campus made biodiesel user, and to accommodate changes, a specific fuel delivery plan will be developed for each user. Fuel delivery plans will have a signature showing approval of the QMR and the motor pool manager, as well as the date of the signatures.

Partnering with campus made fuel users means the biodiesel program will provide adequate fuel storage tank housekeeping and monitoring expertise and guidance. This usually means minimizing water in contact with the fuel. Water bottoms must be removed from tanks, and standing tanks will be sampled and tested for microbial contamination.

Biodiesel will receive special handling during each winter. The green truck will be kept indoors in a heated environment during the months when temperatures may drop below the cloud point of the fuel. When blending between November 1 through March 1, the biodiesel will be a minimum of 10 to 15°F above its respective cloud point to avoid temperature shock. The cloud point of the biodiesel, diesel, and blended mixture will be provided to the campus made biodiesel users by December 1 of each year.

Moscow Cold Month Averages & Records - °F

Month	Average Low	Record Low
January	25°	-30° (1937)
February	27°	-26° (1996)
March	31°	-5° (1955)
November	30°	-14° (1955)
December	24°	-42° (1968)

Generally, new campus biodiesel users will begin with B10 and after four (4) fill-ups increase to a B20. Once the vehicles have been run on B20 for a considerable time, higher blends of biodiesel will be considered.

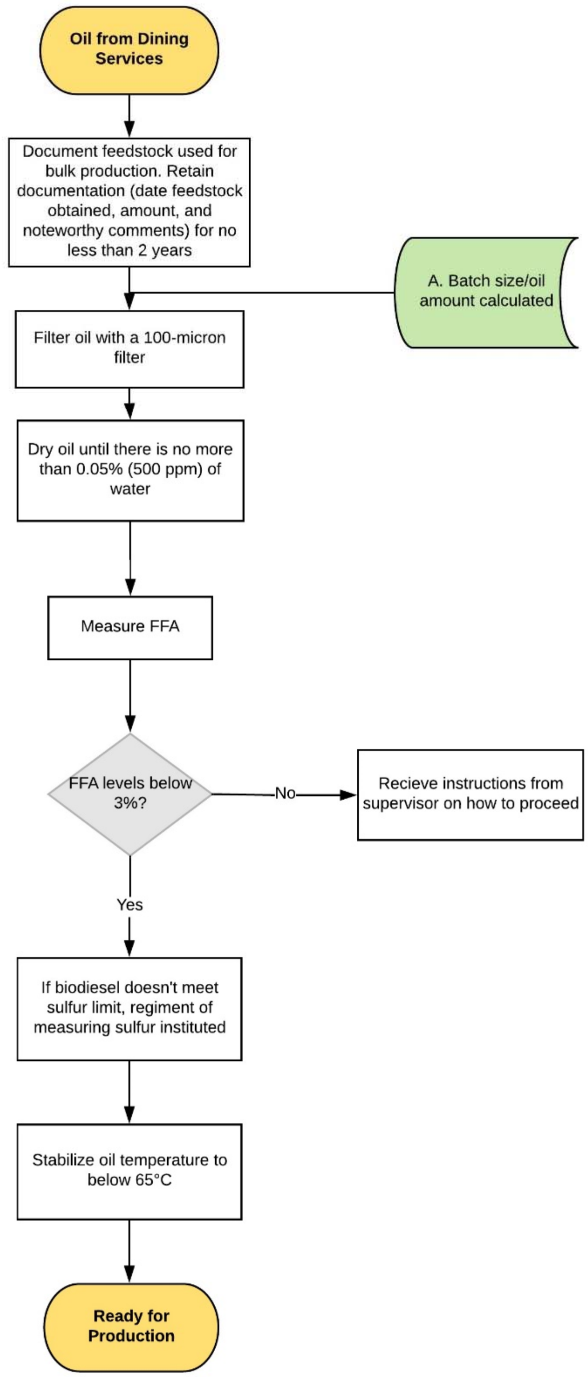
All vehicles and equipment that use biodiesel should carry a spare fuel filter. The cleansing property of biodiesel means filters will become clogged more quickly (during the initial stages of transition) if the equipment's fuel tank and system contain sludge and sediment from years of diesel buildup.

Drivers, equipment operators and technicians will be educated on the use of biodiesel and informed that if they notice any degradation in vehicle power, rough engine idling, or similar problems, they should bring the vehicle into the shop immediately to replace the fuel filters. That will solve the problem most of the time.

ATTACHMENTS

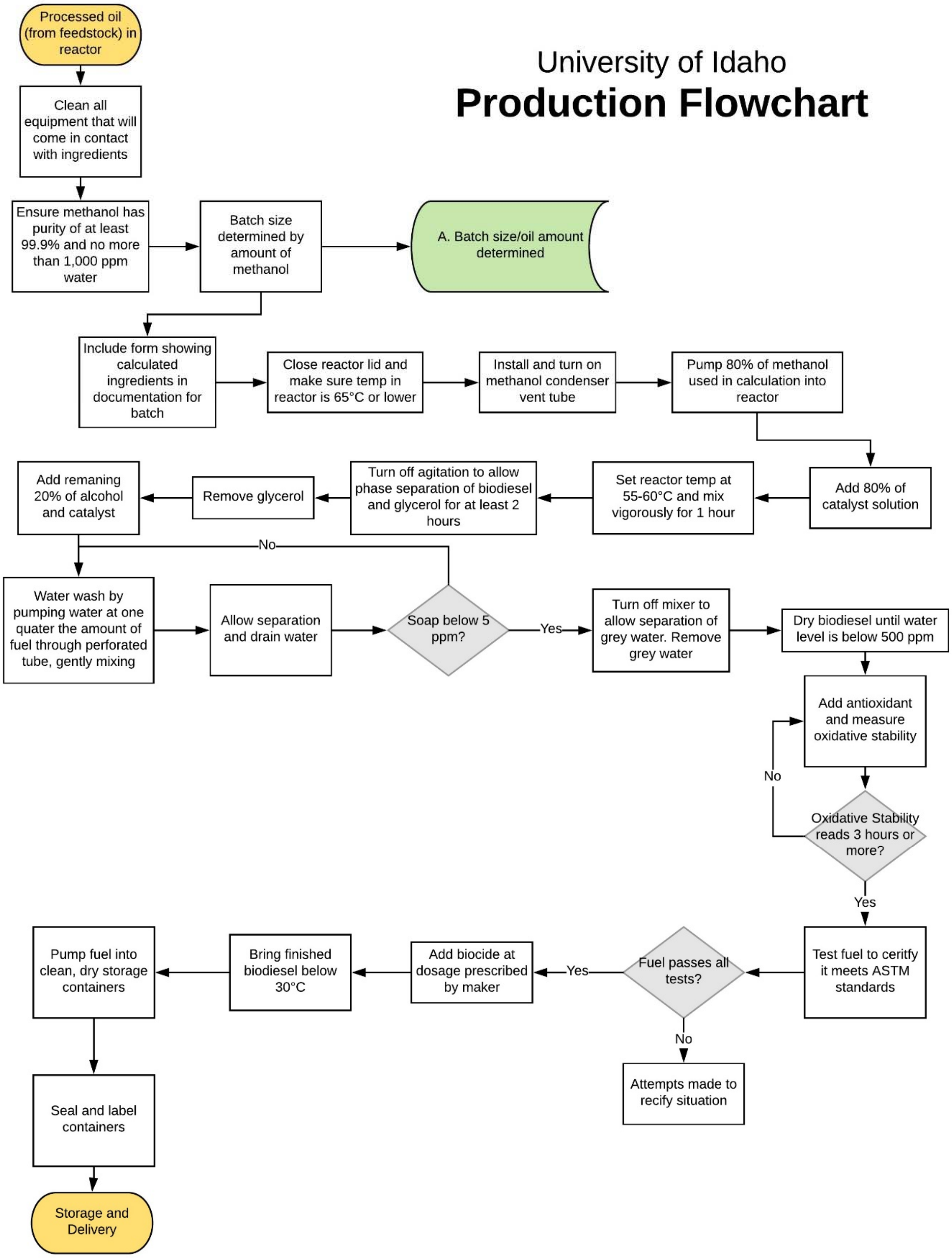
1. Feedstock Flowchart
2. Production flowchart
3. Testing flowchart
4. Storage flowchart
5. Delivery flowchart
6. Biodiesel Certificate of Analysis Form

University of Idaho
Feedstock Flowchart

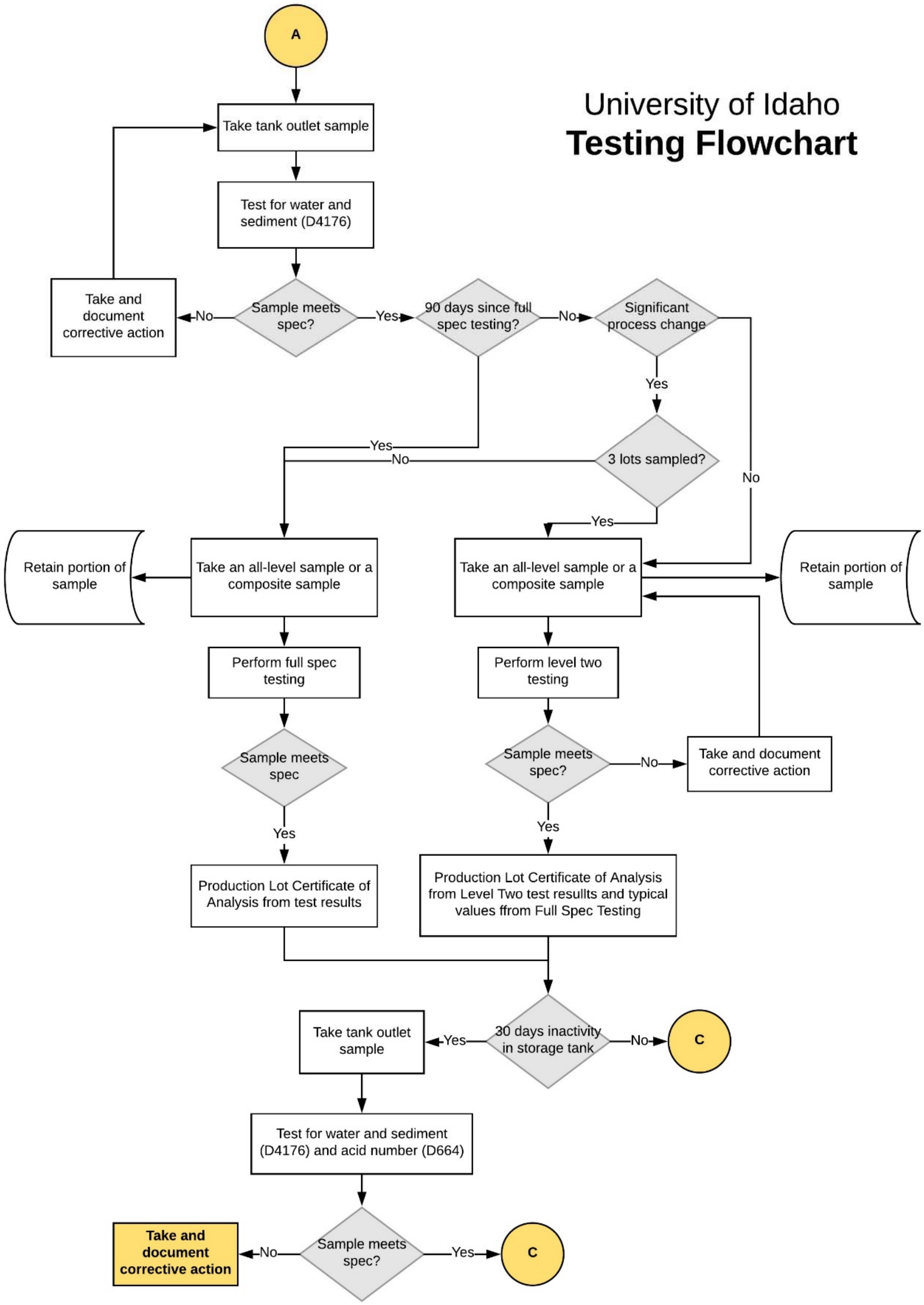


University of Idaho

Production Flowchart

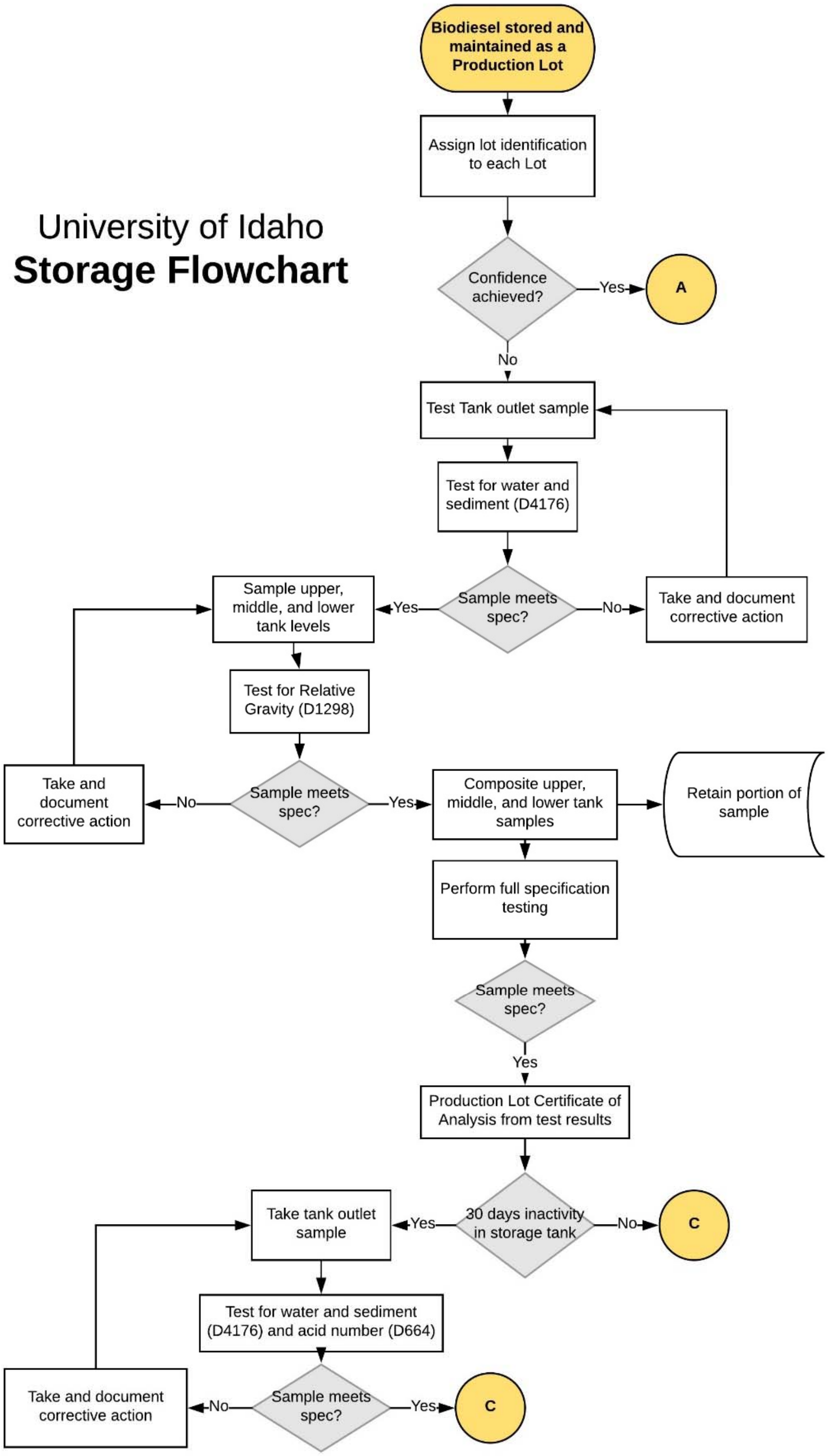


University of Idaho
Testing Flowchart

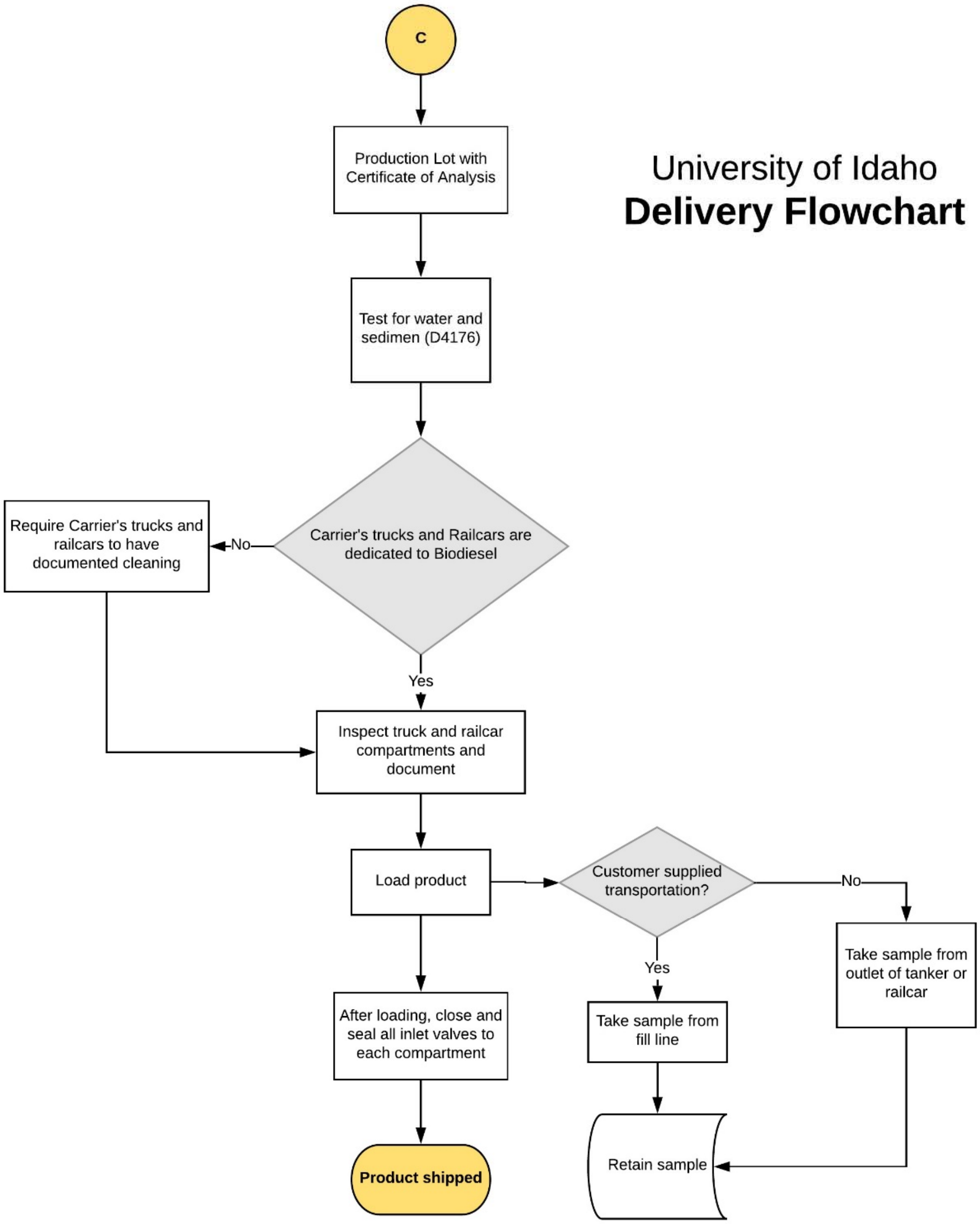


University of Idaho

Storage Flowchart



University of Idaho
Delivery Flowchart



BIODIESEL CERTIFICATE OF ANALYSIS

Lot Number: ______________________

Feedstock: ______________________

PROPERTY	MEASURED VALUE	UofI TARGET	ASTM LIMITS	UNITS	TEST METHOD
Acid Number		0.3	0.50 Max.	mg KOH/g	D 664
Water & Sediment		0.03	0.050 . Max.	% Volume	D 2709
Free Glycerin		0.02	0.020 Max.	% mass	D 6584
Monoglycerides		0.4	0.4 Max	% mass	D6584
Total Glycerin		0.18	0.24 Max.	% mass	D 6584
Cloud Point			Report	°C (°F)	D 2500
Pour Point			Report during winter months		
Flash Point		100°C	130 Min	°C (°F)	D 93
Oxidation Stability		6 hr.	3 hr.	hours	EN 15751
Cold Soak Filtration		200	360 Max.	seconds	D 7501
Sulfur – S 15/500 Grade			15/500 Max.	ppm (mg/kg)	D 5453

______________________ Antioxidant Brand

______________________ PPM Antioxidant Additive

______________________ Biocide Brand

______________________ PPM Biocide Additive

______________________ Cold Flow Additive Brand

______________________ PPM Cold Flow Additive

NOTES: ______________________

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Prepared by: ______________________

Name Date