

# **Environmental Compliance Systems for Gulf of Mexico Synthetic Muds**

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## **ABSTRACT**

The EPA Effluent Limitation Guidelines for the discharge of drill cuttings generated from synthetic-based drilling fluids have been completed and incorporated into the Region 6 General Permit. On the surface of the general permit language, the limitations for meeting stock limitations appear simple. However, imbedded in these limitations are significant requirements for representative sampling and quality control systems. The permit language reflects the need for both the operator and the supplier of the drilling fluid to be prepared to defend their compliance efforts. This responsibility manifests itself in both the company's ISO and quality control systems. This paper will present an example of an established compliance process that includes sample control, product specifications, data validation, process controls and quality management systems. The process is designed to provide a responsible and defensible set of documentation for meeting environmental performance criteria.

In addition to basic quality control measures, service companies also have a deeper knowledge of the test protocols and the artifacts that can develop within those protocols that may cause the base fluids to pass or fail the environmental performance criteria. Moreover, the paper will review laboratory artifacts relating to synthetic fluid quality control systems.

## INTRODUCTION

In the early 1990's synthetic-based drilling fluids were introduced as a new technology (1) with improved environmental performance. Owing to their physical and chemical characteristics, synthetic-based drilling fluids provide excellent drilling performance which reduces the overall volume of waste discharges (2). The capacity of these fluids to inhibit highly reactive shales reduces both wellbore washout and stuck pipe incidents. Further, the superior lubricity of these fluids reduces torque and drag, thereby reducing stuck pipe and allowing for the drilling of difficult directional wells. In addition to performance in the wellbore, inhibition of shale cuttings allow for efficient removal of cuttings from the drilling fluid, thus eliminating the need for high dilution rates and discharge of whole mud required of most water-based muds. Additional advances in surface treatment equipment have allowed a secondary drying step in solids-control equipment, which further reduces discharges from the drilling operations. Further, the comparatively higher rates of penetration of these fluids reduce days on location, thus minimizing both the environmental impact and economics. All of these technical issues have resulted in synthetic-based drilling fluids being recognized as pollution-prevention technology.

The environmental performance criteria employed to identify acceptable and unacceptable synthetic base fluids has been a significant challenge. In the beginning, the focus was segregating synthetic and traditional base fluids by definition and manufacturing processes. As more information became available from microcosm studies and seabed surveys on the environmental performance of synthetic fluids, acceptance criteria shifted towards chemical and biological performance criteria. In the case of the US EPA, the acceptance criteria that were adopted include a chemical test that uses PAH as an indicator of priority pollutants and benthic toxicity and anaerobic biodegradation tests that serve as biological acceptance criteria. Industry work groups devoted substantial efforts in providing the EPA with information that resulted in the best available protocols. Experience gained by regulators, service companies, operators and testing companies indicated that many test procedures used to evaluate acceptability in the North Sea were highly variable because of inconsistent interpretation of protocols and laboratory artifacts. As similar biodegradation and toxicity tests used in the US were considered, the tests were evaluated and modified to reduce variability and minimize testing artifacts. Resulting from these efforts have been three robust testing protocols that serve as a foundation of synthetic-base fluid technology. The implementation of a compliance system built around these protocols insure the integrity of the data used to qualify synthetic fluids and will promote the continued use and discharge of synthetic-based drilling fluids as a pollution-prevention technology.

## PERMIT REQUIREMENTS

### Obvious permit requirements

The Effluent Limitation Guidelines (4) contain three stock limitations for synthetic base fluids that were incorporated into the permit language. The simple language described in the permit is listed below (5).

#### Part I, Section B.2.c;

##### 1) Stock Limitations

*The permittee shall analyze a representative sample of the stock base fluids at the frequencies listed below. The test results shall be reported on the Discharge Monitoring Report.*

*Alternatively, the permittee may provide certification, as documented by the supplier(s), that the stock base fluid being used on the well will meet the limits listed below.*

*Polynuclear Aromatic Hydrocarbons (PAH). The mass ratio in grams of PAH (as phenanthrene) divided by the mass in grams of base fluids shall not exceed 0.00001. Monitoring shall be performed at least once per year on each base fluid blend. See Part I, Section D.10 of this permit.*

*Sediment Toxicity. The ratio of the 10-day LC<sub>50</sub> of C16 - C18 internal olefin or C12- C14 or C8 ester reference fluid divided by the 10-day LC<sub>50</sub> sediment toxicity test with *Leptocheirus plumulosus* of the base fluid shall not exceed 1.0. Monitoring shall be performed at least once per year on each base fluid blend. See Part I, Section D.8 of this permit.*

*Biodegradation Rate. The ratio of the cumulative gas production (mL) of C16 - C18 internal olefin or C12-C14 or C8 ester reference fluid divided by the cumulative gas production (mL) of stock base fluid, both at 275 days, shall not exceed 1.0.*

*Monitoring shall be performed at least once per year on each base fluid blend. See Part I, Section D.9 of this permit.*

Further explanations of the testing requirements are provided in the permit in sections 8, 9 and 10.

#### Part I, Section D;

##### 8. Stock Base Fluid Sediment Toxicity

*The approved test method for permit compliance is identified as: ASTM E1367-99 method: Standard Guide for Conducting Static Sediment Toxicity Tests with Marine and 31 Estuarine Amphipods (Available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA, 19428) with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in Appendix 3 of 40 CFR Part 435, Subpart A and the method found in Appendix A of this permit.*

##### 9. Biodegradation Rate

*The approved test method for permit compliance is identified as: modified ISO 11734:1995 method: "Water quality - Evaluation of the 'ultimate' anaerobic biodegradability of organic compounds in digested sludge - Method by measurement of the biogas production (1995 edition)" (Available from the American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036) supplemented with modifications in Appendix 4 of 40 CFR Part 435, Subpart A and detailed in Appendix B of this permit. Compliance with the biodegradation limit will be determined using the following ratio:*

$$\frac{\% \text{ Theoretical gas production of reference fluid}}{\% \text{ Theoretical gas production of NAF} + 4\%} \leq 1.0$$

Where: NAF = stock base fluid being tested for compliance  
Reference Fluid = C16-C18 internal olefin or C12-C14 or C8 ester reference fluid

#### **10. Polynuclear Aromatic Hydrocarbons**

*The approved test method for permit compliance is identified as: Method 1654A: "PAH Content of Oil by High Performance Liquid Chromatography with a UV Detector," which was published in Methods for the Determination of Diesel, Mineral and Crude Oils in Offshore Oil and Gas Industry Discharges, EPA-821-R-92-008 (incorporated by reference and available from National Technical Information Service at 703/605-6000).*

Finally, in order to minimize variability and artifacts within these tests, the EPA listed very detailed descriptions of the protocols in Appendix A and Appendix B of the Permit for sediment toxicity and biodegradation testing, respectively. Furthermore, contained in these protocols listed in the Permit appendix are detailed QA/QC procedures for each test. The Permit requirements for passing these tests are listed in the Stock Limitations.

## **Imbedded Permit Requirements**

In addition to the obvious stock limitation requirements, the Permit contains standard conditions that apply to all NPDES permits. While these requirements are less obvious, they are not less important. Some of the most critical of these requirements can be found in Part II Section C and D of the permit and relate to Monitoring and Reporting Procedures. Several of these requirements from the Permit are presented below.

### **Part II Section C**

#### **2. Representative Sampling**

*Samples and measurements taken for the purpose of monitoring shall be representative of the monitored activity.*

#### **3. Retention of Records**

*The permittee shall retain records of all monitoring information, including all calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of the sample, measurement, report, or application. This period may be extended by request of the Director at any time.*

*The operator shall maintain records at the platform where the discharges occur or another platform in the Field for a period of three years, whenever practicable and at a specific shorebase site whenever not practicable. For example: in the case of unmanned platforms or platforms where records storage is not practicable, records may be maintained at a central field office platform or a specific shore based site. In either case, the records must be available for review by government inspectors coincident with their inspection. The operator is responsible for maintaining records at exploratory facilities while they are discharging under the operators control and at a specific shore-based site for the remainder of the 3-year retention period.*

#### **4. Record Contents**

*Records of monitoring information shall include:*

- a. *The date, exact place, and time of sampling or measurements;*
- b. *The individual(s) who performed the sampling or measurements;*
- c. *The date(s) and time(s) analyses were performed;*
- d. *The individual(s) who performed the analyses;*
- e. *The analytical techniques or methods used;*
- f. *The results of such analyses; and*
- g. *A copy of the permit and notice of intent to be covered.*

#### **5. Monitoring Procedures**

- a. *Monitoring must be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit or approved by the Regional Administrator.*
- b. *The permittee shall calibrate and perform maintenance procedures on all monitoring and analytical instruments at intervals frequent enough to insure accuracy of measurements and shall maintain appropriate records of such activities.*
- c. *An adequate analytical quality control program, including the analyses of sufficient standards, spikes, and duplicate samples to insure the accuracy of all required analytical results shall be maintained by the permittee or designated commercial laboratory.*

#### **Part II Section D**

#### **5. Additional Monitoring by the Permittee**

*If the permittee monitors any pollutant more frequently than required by this permit, using test procedures approved under 40 CFR Part 136 or as specified in this permit, the results of this monitoring shall be included in the calculation and reporting of the data submitted in the Discharge Monitoring Report (DMR). Such increased monitoring frequency shall also be indicated on the DMR.*

However, the implications of these requirements goes beyond the language in the permit and the guidelines and includes secondary documents such as the *NPDES Compliance Inspection Manual* (6), Third Level References such as the *Handbook for Analytical Quality Control in Water and Wastewater Laboratories* (7); *Handbook for Sampling and Sample Preservation of Water and Wastewater* (8) and Fourth Level References such as *National Environmental Laboratory Accreditation Conference* (9).

With each additional level of references the details of quality assurance and documentation become apparent. While these quality assurance requirements are burdensome, they provide the defensibility of data required by the permit when a critical once-a-year testing requirement is enforced. In many cases, companies may correctly assume that these requirements are being met by their commercial testing lab. However, the necessary procedures and documentation should be developed and preserved before the inspector arrives at the front door. Additionally, laboratory mistakes do not relieve the permittee of the responsibility for the monitoring results or the associated quality assurance procedures. As with many other important issues, operators frequently contract service companies to address these regulatory issues. Consequently, service companies sometimes are contractually obligated to develop quality assurance programs to address both the obvious and imbedded permit requirements. The following section describes one such program to meet the permit requirements for SBM stock limitations.

## **COMPLIANCE PROCESS**

The compliance process has to address several issues imbedded in the permit requirements. The first basic step is the identification and collection of a representative sample. Secondly, the sample has to be tested and the test results validated. Finally, the test results have to be documented to represent the product that is delivered and ultimately discharged.

In the case of stock limitation requirements issues in the permit for barite, it was possible to collect a representative sample of product, quarantine the product until the test results indicated that the stock limits were met, and then provide test results that were tied to a specific lot of barite production.

Due to the 275-day duration of the biodegradation testing procedures, it is not economically realistic to quarantine large volumes of base fluid until testing is complete. Consequently, the stock limitation compliance program for synthetic fluids is more complex than that for barite.

There are many potential platforms for developing a compliance program for those companies with existing ISO 9000 programs which offer an excellent format for maintaining rigorous procedures and documentation. In the case of the example compliance process, local work instructions have been developed in the proper format that allow the systems to run in parallel with other quality control programs in the company. For this summary, the procedures were removed from the ISO work instruction format and summarized. Certain basic elements such as responsibilities, management of change and general elements of the ISO program concerning training, communication, and document control have not been included in this discussion. The purpose of this summary is to provide an example of the sampling, approval and quality control measures that were developed to address the requirements in the NPDES permit.

## **Qualifications of Base Fluids and Base Fluid Blends**

Before a base stock or blend of base stocks can be approved, it must be shown that it will pass base-fluid stock limitation requirements of the permit for the Gulf of Mexico. These requirements include sediment toxicity tests, biodegradation tests and PAH content using the test procedures described in the Permit and Effluent Limitation Guidelines. Identification and approval of blend constituents, blend ratios, and acceptable QA specifications must also be completed.

The toxicity, biodegradation and PAH are to be performed on representative samples of base stock. To accomplish this goal, samples submitted for testing should be accompanied by the chemical specification test results for each sample. In addition, a chain of custody records and specific lot numbers that corresponds with the chemical specification test results should accompany the sample. In those cases where products are blended at a predetermined ratio, the preparation of samples is conducted in a laboratory using calibrated measuring equipment and fully documented procedures.

Each of the protocols that are used for testing includes performance criteria. Some of these criteria, like control survival in the sediment toxicity and the positive, negative, and intermediate control performance in the biodegradation test, are obviously important. While other criteria may not seem as critical, they should be evaluated to determine the acceptability of a test report. An example of these could include the temperature range of the sediment toxicity or

biodegradation test. If the temperature is out of range a judgment would have to be made on using the test/results for compliance purposes. Therefore, an in-depth review of the protocol should be made to determine all of these criteria. Consequently, each test report used to establish compliance with stock limitations should be reviewed against the criteria to confirm the test met the QA/QC parameters for the test protocol. Check lists completed by competent scientists are used to document adherence to specific criteria. It is expected that raw data should be included with the report, along with detailed calculations supporting the conclusions of the report. The detailed manner of the report should be such that another competent scientist could take the same raw data, repeat the calculations, and arrive at the same conclusions. In addition, calibration standards and calibration records also are included in test reports so that the conformation can be reviewed, validated and, if necessary, made available at a later date for confirmation. In some cases this is seen as “over the top” documentation. However, including this information in the report eliminates the possibility of the data being lost, destroyed or otherwise unavailable at a later date.

Once data has been generated and validated on properly documented samples, the next step is analyzing it against the stock limitations. Data generated using approved procedures cannot be excluded from use in analysis of a blend or single-supplier product to be used as a base fluid without a scientific justification. The exclusion of failing results is sometimes called “cherry picking” and prohibited by Part II, Section D of the permit that require reporting of additional monitoring performed by the permittee. However, data can be excluded if it was generated under conditions that do not meet the control parameters of the test or if a defensible scientific approach is used to justify the exclusion of a particular sample result.

Experience with the current system has led to several instances where products that should have passed the stock imitations were excluded because testing artifacts impacted the test results, but were not severe enough to invalidate the test result. In order to address this issue, the industry has requested the EPA modify the permit to allow averaging of results. As with all compliance programs, the stock-limitation program is a living procedure that changes to adapt to permit modifications and changing business conditions.

## **Establishment of QA/QC Chemical Specifications**

Because the 275-day biodegradation test cannot be conducted on specific lots of fluids and the toxicity and PAH tests are not required by the permit to be conducted on each lot of fluid, a surrogate set of quality control parameter must be developed to ensure the sample that passed the stock limitations is representative of the fluids delivered for use and discharged. The basis of this aspect of the certification program is that chemical characteristics of base stocks drive the environmental performance of base stocks. The significance of changes in chemistry can vary from parameter to parameter and from product to product. Therefore, the establishment and defense of chemical specifications for each product is handled on a case-by-case basis. As knowledge of the chemical specifications that significantly impact the environmental performance of the base stock in biological tests increases, new and different chemical specifications may need to be established while others may need to be terminated. The long-term research efforts reflected in published research has shown there are some universal chemical specification elements that should be addressed in any synthetic-base fluid quality assurance program. These chemical specifications should include at minimum the following elements:

**Carbon Chain Distribution** – The distribution of the mass of the carbon chain length, or carbon chain distribution, has been generally established as a driver in toxicity evaluation. Typically, the test method to determine carbon chain distribution is a GC/FID. In some cases surrogate parameters such as flash point can be used to help characterize this parameter.

**Branching** – The degree and nature of branching in base stock molecules has been generally established as driver in biodegradation rates. The test method to determine branching is typically NMR. In some cases surrogate parameters such as pour point can be use to help characterize this parameter.

**Chemical Structure or Speciation** – Chemical structure has been established as a driver of both biodegradation and toxicity performance. The test method to identify the general nature of the chemical structure can vary but may include techniques as IR, NMR or GC/MS.

**Contaminants** – Contaminants cover a wide range of impurities including residual raw materials such as olefins, alcohols, residual catalysts, aromatic or other toxic compounds that may impact performance in the product in the biodegradation, toxicity or PAH test. Test protocols for contaminants could include GC, IR, NMR, AA or other analytical techniques.

Therefore, the establishment of specific chemical parameters includes the following steps:

1. Initial biological testing and identification of manufacturing specifications for supplier products.
2. Establishment of a consistency of manufacturing parameters and chemical specifications in conjunction with performance in environmental tests.
3. Modification of chemical specifications in light of the consistency of manufacturing and performance in environmental tests.

Similarly, the establishment of specific blend ratios includes the following steps: initial biological testing and identification of blend ratio for supplier products and modification of blend ratio in light of the consistency of manufacturing and performance in environmental tests. These chemical specifications should be agreed upon with the suppliers and shared in writing so as not to create any unnecessary surprises.

## **Acceptance of Individual Shipments**

Before a shipment of product is accepted from the supplier, QA specifications should be met. Chemical specifications for each supplier product used in certified base stock should be generated as outline above. Prior to each shipment of a product, the supplier sends a Certificate of Analysis to a qualified QA person within the company. The qualified QA person reviews each certificate of analysis and confirms that it meets the chemical specifications agreed upon by both parties. The QA person then notifies the plant receiving the shipment that the supplier product batch or shipment has been approved.

Any fluid not meeting the chemical specifications should not be accepted for use in a base stock. This fluid could be accepted by concession and tests performed to determine if the base fluid will pass the required compliance tests.

## **Blending and Certifying Base Stock**

Supplier products should be blended at a qualified location using only supplier product shipments that have been approved by QA. Verification of the blend is performed and documented. This verification could include analytical analyses by GC/FID, tank measurements, or calibrated flow meters. The verification method has to be approved by the quality assurance department.

**Verification by GC/FID analytical analyses** – After homogenization, a sample is collected and the batch assigned a unique number. Afterwards, the sample is sent to an approved analytical lab for GC/FID testing following proper chain-of-custody procedures. The GC/FID results are evaluated at the blending plant to determine if the ratio of approved supplier product(s) is within acceptable ratio specifications. If the ratio is within specifications, the batch is added to the base stock supply. If the ratio is not within specifications, additional approved supplier product(s) is added as needed to bring the ratio into specifications.

**Verification by calibrated flow meters** – Documentation of the flow meter readings are recorded with the initials of the person performing the reading/measurements and dates to verify the blend.

**Verification by tank strapping measurements** – Documentation of the levels in the tank are recorded with the initials of the person performing the reading/measurements and dates to verify the blend.

The approval process is completed and documented by attaching the formulation blend sheet to the blend conformation documentation, which may include GC/FID analytical results, tank measurements readings, metering pump readings, or supplier information. The blend facility manager or their designee signs a Certificate of Compliance for any and all shipment(s) of base stock. The certificate of compliance states that only certified supplier products were used in the preparation of the base stock.

## **Mixing and Certifying of Drilling Fluids**

Certifiable drilling fluids should only be mixed with base stocks from an approved blending facility following receipt of the Certificate of Compliance for a particular shipment. Arriving shipments of base stock(s) should be quarantined from the non-aqueous drilling fluid (NAF) liquid mud inventory until a Certificate of Compliance is received from the blending facility. Transfer of the base stock(s) should be handled and stored in dedicated containers and tanks, and transferred using dedicated hoses and pipes.

## **Certification of NAF Drilling Fluids**

The warehouse manager, or his/her designee, can sign the Certificate of Compliance for any and all shipment(s) of NAF liquid mud inventory. The certificate of compliance states that only certified base stock was used in the preparation or reconditioning of the NAF drilling fluid. Certificates of compliance shall be provided to the permittee at the point of loading.

NAF drilling fluids that are not a part of NAF liquid mud inventory could be added to the NAF liquid mud inventory providing:

- that NAF drilling fluid had only base stock(s) additions that meet the base-fluid stock limitations for toxicity, biodegradation and PAH as indicated by NPDES permit, and
- that NAF drilling fluid is documented to be free of formation oil contamination.

## TESTING ARTIFACTS

Because synthetic fluids were introduced as a pollution-prevention technology with improved environmental performance, there is particular interest in identifying, documenting and defending the environmental improvements. In the North Sea, efforts to prove environmental performance ended in failure and the use and discharge of synthetic fluid technology ceased. In the North Sea, one of the primary difficulties not overcome was the resolution of testing artifacts on the acceptance criteria for synthetic base fluids. During the process of evaluating both test procedures and base fluids, it was documented in the literature that simple modifications contained within standardized test procedures resulted in a high degree of variability in test results. (10) Test procedures used to determine aerobic biodegradation rates resulted in significant variability even though the testing labs were complying with all of the quality control procedures documented in the protocol. A number of authors have concluded that the standardized tests needed to be further refined and additionally standardized to minimize testing variability (11,12). Conflicting efforts to qualify less expensive base fluids while defending the environmental acceptability of the existing fluids, continued until synthetic base fluid technology was effectively terminated in the North Sea regulatory arena. In most cases the artifacts of the standardized tests were not resolved as they applied to synthetic base fluids using North Sea protocols.

The US EPA and industry representatives had the benefit of reviewing the lessons learned in the North Sea prior to formal requirements being developed in the country. From these experiences, the industry learned to adapt, modify and further standardize protocols in order to minimize testing variability and protect the ability of the test to discriminate acceptable and unacceptable environmental performance. Consequently, the test procedures detailed in the permit have been further refined and standardized to minimize testing variability. As testing has continued, more information and experience is available to identify and resolve potential testing artifacts. For the companies involved in testing and qualifying base stocks, there is an additional level of responsibility that requires they maintain the overall integrity of the test by continuing to identify and resolve testing artifacts.

An example of this type of ongoing responsibility is the use of additional evaluation controls. In the past two years the use of diesel oil as an observation control has been implemented for the 10-day sediment toxicity test. On-going experience indicates that the reference fluid LC<sub>50</sub> results may be significantly reduced due to animal size or sediment quality. In order to insure the discriminatory power of the test is maintained, each time a test series is conducted, a diesel oil sample is run in parallel as a positive control. Over time, the toxicity ratio between diesel and the reference fluid is monitored to insure the overall test performance is consistent. Similarly, the intermediate control in the 275-day biodegradation test can be monitored to observe the relative performance of the sediment used in the test. While these measures are not full-proof, they offer information that can help maintain the overall integrity of the testing regime. By continuing to protect the overall integrity of the test, responsible industry members eliminate the need for regulators to develop and enforce more burdensome testing requirements.

## CONCLUSIONS

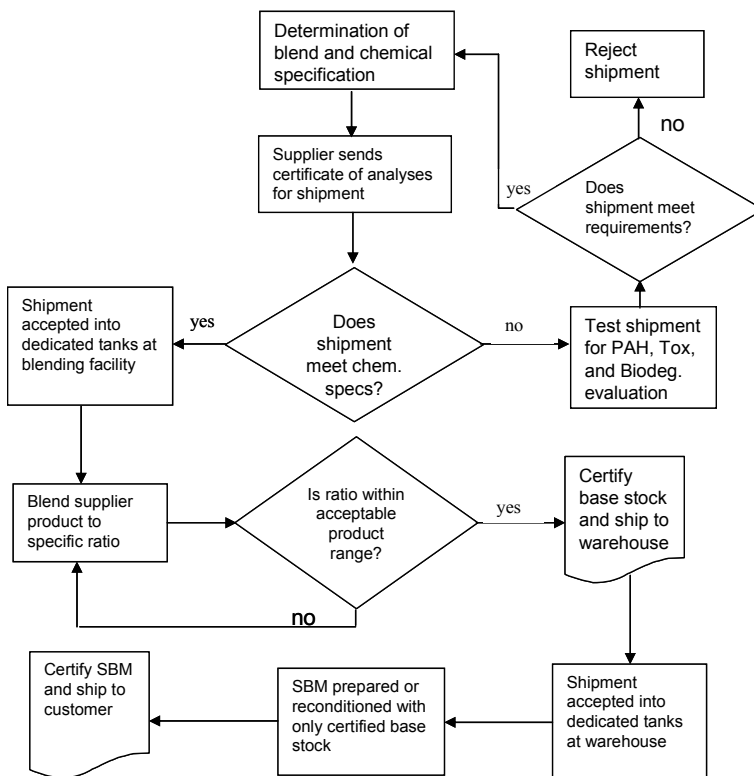
The inclusion of synthetic based mud requirements into the general discharge permit GMG 290000 has added new requirements. Some of these requirements are obvious, while others are imbedded in the general requirements of the permit. In order to address these new requirements, a compliance program has been developed that uses an existing ISO 9000 quality control program as a foundation. New work procedures to collect and analyze samples have been developed as well as new process control procedures. In addition to these measures, a secondary effort has continued to evaluate and resolve testing artifacts as they are identified using additional testing samples and evaluation parameters.

This summary of the certification process for base stock delivered to the wellsite is just one part of a larger program to ensure compliance with the limitations for synthetic base fluids. After three years of active service, the procedures have been incorporated into routine business activities and have served as a defensible basis for certifying synthetic base fluids. Additional sampling and testing procedures conducted at the well site also are subject to both the obvious and imbedded requirements listed in the general permit. These compliance requirements have been addressed with a significant quality assurance program and backed up with training and documentation that provide a high degree of defensibility. While some consider these efforts “over the top” others consider these efforts as reasonable and required.

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**Figure 1.** Base Fluid and Drilling Fluid Certification Flow Chart