

Haynesville Performance Review: Unique Clay-Free Water-Based Polymer Drilling Fluid System for Application-Specific Unconventional Shale Production Intervals

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Abstract

Operators in the Haynesville Shale have long sought an eco-appropriate, water-based drilling fluid solution to the rigorous demands of its long lateral production intervals. High bottom-hole temperatures, troublesome formation contaminants, high pore pressure and wellbore lubricity requirements combine to provide a stressful challenge to conventional water-based formulations. Diesel-based mud has become the default drilling fluid choice.

But utilitarian oil-based mud (OBM) has its own shortcomings, including ancillary transportation and disposal costs and liabilities, environmental concerns, and gas solubility issues that can complicate well control procedures.

A clay-free, water-based polymer system was introduced in late 2009 as an alternative to OBM for this use. Based upon several components new to drilling fluid technology, the system has seen multiple Haynesville applications by numerous operators. A unique polymeric viscosifier/suspension agent, a blend of environmentally sound lubricants and a versatile low-end rheology modifier form the heart of the system.

This field case study examines drilling operational results and overall drilling fluid performance achieved using Haynesville application-specific fluid formulation, drawing from ample data collection and tracking programs developed to evaluate system performance and to provide guidance for continuous improvement and optimization. Penetration rates, drilling lubricity, wellbore management, contaminant tolerance, thermal stability and logistics management are discussed.

Characterization of Haynesville and Bossier shales, and initial developmental performance goals are reviewed. Resultant fluid components and formulation are described. Best Practices and continuous improvement strategies and results are also discussed.

Results achieved with the new water-based technology are evaluated versus offset OBM results.

Introduction

Numerous Haynesville Shale operational factors have introduced a sense of urgency to find an effective and economical alternative to OBM traditionally employed for long lateral sections in that drilling environment. But developing a water-based solution would require evolutionary advances in component stability and performance capabilities.

Among factors driving the search are regional liquid

mud infrastructure inadequacies, growing environmental stewardship focus and targeted cost/efficiency improvement related to OBM-related peripheral activities.

As the world's operational cradle of unconventional shale hydrocarbon development, the Haynesville is a fundamental proving ground for drilling fluid technology developed specifically for this application. But the Haynesville also presents operators with a hostile combination of elevated wellbore thermal conditions, troublesome contaminants and high temperature/high pressure (HTHP) lubricity requirements that have historically limited the effectiveness of conventional water-based drilling fluids (WBM).

Clay-based WBMs have generally been considered deficient in such applications based upon unacceptable conditioning costs and general operational effectiveness. Prior polymer-based WBM formulations have likewise exhibited thermal- and lubricity-related limitations.

A newly developed clay-free, polymer-based WBM was introduced for Haynesville Shale production interval applications in late 2009. An HTHP performance enhancer providing high drilling penetration rates and OBM-like lubricity forms the platform for system capabilities. A high performance water-based system was formulated by combining this performance enhancer with a hardy new viscosifying polymer whose outstanding thermal stability and contaminant resistance matched Haynesville-specific fluid operational requirements.

A review of system laboratory developmental process and direct comparison of Haynesville field results achieved with the new WBM versus those of traditional OBM suggests its validity for this application.

Establishing System Performance Criteria

A thorough characterization of the two primary shales encountered in Haynesville production intervals provided a valuable basis upon which to conceptualize performance requirements for the new fluid. These essential shale characteristics, along with Haynesville operational factors—elevated wellbore temperature, formation contaminants and lubricity requirement—provided a basis for establishing system performance developmental goals.

Haynesville/Bossier Shale Chemistry and Mineralogy

The Haynesville and Bossier shale beds are located along the Texas-Louisiana state boundary. Both are encountered while drilling Haynesville production intervals. The shallower

Bossier Shale is generally drilled vertically after exiting intermediate casing, and the target Haynesville is drilled horizontally to maximize exposure to the production zone.

Twenty-four samples of drilled cuttings from the Haynesville Shale formation were analyzed. Samples were collected from various counties and parishes of the region including Red River, DeSoto, Panola, and Shelby. Three cuttings samples were taken from the Bossier Shale formation. All samples were tested using the methodology cited previously^[2] and results were compiled over a two year period.

Analysis performed included x-ray diffraction, x-ray fluorescence, linear swell meter, shale dispersion, Mercury Injection Capillary Pressure (MICP) porosity/permeability, shale water activity, CEC analysis and soluble salts.

All x-ray diffraction (XRD) data showed the samples to be predominantly clay. Haynesville samples fell in the range of 53 - 31% clay, while Bossier samples ranged from 36 - 27% clay, % of weight of the sample. Other minor phases indicative to these shales were quartz, calcite and mica. Further analysis identified the primary clay present as illite. Haynesville shale sample illite content was in the range of 78 - 57% and Bossier shales had illite concentrations of 58 - 53%, weight of the total clay. All samples exhibited extremely low levels of smectite (7 - 0% weight). High aluminum and silicon concentrations as seen in the x-ray fluorescence (XRF) data confirmed these findings.

Both shales were characterized as virtually non-swelling and featured low reactivity, with Cation Exchange Capacity (CEC) values of 13.9 - 3.6 meq/100g. Most samples of both formations exchanged calcium (89% of all samples tested). All other samples had equivalent amounts of sodium and calcium exchanged when tested. The Haynesville Shale formation could best be differentiated from the Bossier by its concentration of soluble salts. The Haynesville formation had minimal water soluble salts within the sample in the range of 3.6 - 17.8 meq/100g. The Bossier samples showed elevated concentrations in the range of 17.2 - 29.5 meq/100g. The Haynesville formation could be characterized by the fact that its main soluble salts were sodium and bicarbonate. The Bossier shales exhibited equal amounts of sodium and calcium as well as chloride as its main water soluble ions.

Summarizing other shale analysis results:

- Both shales exhibited relatively low porosity and permeability in MICP analysis;
- Shale dispersion analysis showed extremely low reactivity with various drilling fluids tested; and
- In linear swell meter testing, most shale swelling occurred in initial 60 minutes of exposure, and very little additional swelling occurred after that in the 22-hour test period.

Typical Haynesville and Bossier shale analyses are depicted in Figures II and III.

DSC Testing

A sample was tested via Downhole Simulation Cell (DSC)^[1,3,4]. Before testing commenced on the Haynesville

shale formation core taken from 12,606 feet TVD. CEC was determined to be 19.5 meq/100g. Its main exchangeable base was calcium. This sample showed low to moderate amounts of soluble salts (10.8 meq/100g). Interestingly, this sample showed almost equivalent amounts of chloride and bicarbonate concentrations. The XRD showed this sample to be mainly clay, 45% total weight of the sample, and as expected the clay was mainly illite, 86% of total clay. After exposure to the specialized drilling fluid the CEC slightly decreased to 11.5 meq/ 100g. Calcium remained the primary base that exchanged. Soluble sodium increased and soluble calcium decreased. Soluble anions remained fairly constant. The mineralogy as indicated by XRD & XRF did not show any significant changes in the bulk sample. However, the clay species appeared to show a transition to a more mixed-layered configuration. The DSC test is summarized in Figures IV & V.

The DSC test was conducted on a preserved Haynesville core under conditions specified in Figure I. The shale showed no signs of swelling or compaction during the test. The total transfer of fluid during the 120 hours after drilling the borehole through the sample was 7.0 ml. The fluid transfer is believed to have been through a sand bedding plane in the sample, as the core had the same water activity (0.520) and moisture content (<1%) before and after the test.

Haynesville Operational Criteria

The Haynesville Shale's drilling environment has made diesel oil-based mud the common choice for drilling its horizontal production intervals. Its drilling conditions have long challenged conventional water-based drilling fluid formulations based upon either clay-based or polymer viscosifiers.

Haynesville production interval wellbore temperatures are generally above 300° F. At this temperature the standard viscosifying polymer of choice, xanthan gum, deteriorates rapidly, and replacement/treatment costs become economically unrealistic.

The use of commercial bentonite and conventional clay-based formulations pose other challenges. The combined effects of high temperature and significant CO₂ influxes while drilling the shale cause severe rheological problems in clay-based formulations.

CO₂ must be removed from bentonite mud using a calcium source, generally lime. At elevated Haynesville wellbore temperatures, maintaining a large excess of lime is not a viable option, as elevated temperatures may cause cementation of the fluid. At lower lime concentrations, fluid viscosity can become unstable as CO₂ influx levels vary. The resultant viscosity "hump" from either the calcium or the CO₂, introduces several mud-related issues, including hole cleaning problems, fluid loss control issues and much higher coefficients of friction. These factors often create wellbore management problems which are sometimes incorrectly attributed to shale instability/inhibition.

Achieving adequate lubricity for this horizontal application is also an issue in conventional bentonite muds, further exacerbated by higher viscosities resulting from lime

additions, wellbore CO₂ intrusions and high mud weights (>15.0 ppg) required in Haynesville wells. Addition of lubricants to a clay-based mud with a high mud weight is generally not very effective, typically contributing only modest reductions in the coefficient of friction (COF) as compared to the base mud with no lubricant present. Any additions of commercial clays will actually increase the coefficient of friction in muds with MBT above 5-10 lb/bbl. Drilled solids will have a similar effect above 6%. As a result, achieving adequate lubricity in a water-based fluid for this application requires that the fluid have an MBT of less than 10—even with up to 6-8% drilled solids.

Fluid System Concept and Description

Based upon shale characterization findings and fluid performance criteria, a water-based polymer system was conceptualized for this application of high temperature and contaminants. The system and its components should:

- exhibit thermal stability exceeding 400° F;
- provide contaminant resistance to CO₂, drilled solids, H₂S, cement and others;
- provide HTHP lubricity comparable to invert emulsion (oil-based) fluids;
- provide wellbore stabilization;
- be reusable after re-conditioning;
- adequately support barite in high density applications;
- offer eco-appropriate formulation for widened fluid and cuttings disposal options and HSE advantages; and
- compete with oil-based fluid overall cost and drilling/operational performance, with emphasis upon penetration rates while drilling and sliding and support of casing running operations

Four new primary components identified and developed for this concept form the basis for the proprietary system.

- *Product A* advanced polymeric viscosifier and suspension agent exhibits conspicuous thermal stability, laboratory proven to 425°F, and resistance to common field contaminants including CO₂ and H₂S. *Product A* is shear thinning throughout the temperature range from 75-400° F. The product's viscosifying properties remain robust as the temperature increases (Figures VI - X), and it exhibits modest temperature degradation tendencies. It is unaffected by high solids (being stable up to at least 18.0 ppg with 12% Haynesville shale drill solids) (Figure X) and CO₂.
- *Product B* HTHP performance enhancer provides OBM-like coefficient of friction values (.04-.09) to enhance penetration rates and to enable efficient tripping of the drillstring and casing or liner running operations. The economical lubricant is effective in low concentrations, and like *Product A*, is laboratory proven to 425°F.

- *Product C* rheology modifier adjusts low-end rheology to optimize drilling hydraulics and hole cleaning capabilities in extremely low concentrations.
- *Product D* fluid conditioner, a general purpose fluid conditioner.

Several characteristics and capabilities enhance system applicability to unconventional shale use. Primary among these are the lubricating capabilities of the polymeric viscosifier and the HTHP performance enhancer to promote efficient weight transmission to the bit in long horizontal sections. The clay-free formulation and low-end rheology modifier also enhance drilling efficiency. The system's recyclability enhances its cost effectiveness. A relatively simple system with few components, wellsite housekeeping is simplified by virtue of fewer products required at the rig.

Formulation Testing and Validation

Rheology

Establishing a rheology profile for the fluid under stressful conditions of temperature and contaminants was central to optimizing fluid formulation. Hundreds of formulation iterations were examined in the laboratory for this purpose, resulting in a broad yet concise understanding of fluid capabilities and characteristics.

Results shown in Table VI – X illustrate the fluid's shear thinning characteristic, while its viscosity is not significantly affected by elevated temperature. The primary effect of elevated temperature is upon plastic viscosity (PV), which drops significantly with temperature. This (along with its lubricity) is likely one fluid system mechanism that promotes drilling penetration rates exhibited by the fluid system.

Comparing the data in Tables VI and VII, it can be seen that laboratory-prepared muds have very similar properties to the actual muds used in the field.

Tables VIII – X illustrate the stability of fluid samples with elevated drilled solids content and high static aging temperatures, prior to testing the rheology of the fluid. Tables VIII – X also show that even after 18 or more hours of static aging at high temperatures, fluid properties have not changed significantly from measurements taken prior to the static aging.

Initial Field Results

In cooperation with a major independent operator, a Haynesville shale well whose anticipated wellbore parameters were considered typical of this unconventional play was chosen for the system's initial field test. The specialized fluid would be used to drill the horizontal production interval, replacing the OBM historically used. Ultimately, the system was employed to drill production intervals of four consecutive DeSoto and Caddo Parish wells in order to gauge its field performance and results.

The specialized fluid system for the initial well was built on location, thereby eliminating most transportation costs and liabilities associated with OBM.

In this initial field trial, the intermediate casing shoe and cement were drilled using the low solids, non-dispersed fluid used to drill the prior interval. Pits were then thoroughly cleaned and a 15.5 lb/gal specialized polymer-based fluid was displaced into the wellbore. (In successive wells, the polymer fluid was displaced immediately behind the cement plug and employed to drill the shoe, with no problems related to cement contamination.)

After exiting the 7 5/8" casing shoe at about 10,500 feet, drilling began with a 6.5" PDC bit (sliding and rotating as needed) to arrive at the kickoff point at about 11,300 feet. Some mechanical fluid aeration related to hopper discharge proved manageable through mechanical means and use of defoamers. Mechanical solutions included the use of a flooded hopper, baffle plates, and by mixing regular system treatments in the slugging pit rather than directly to the active system. A standard alcohol-based defoamer proved more effective than a glycol-based alternative for this purpose. (*Note: Subsequent field experience showed that a silicone-based defoamer provided superior results.*)

Drilling through the curve section was without incident. High viscosity sweeps were pumped regularly to aid hole cleaning. Drilling continued with an average rate of penetration (ROP) of 19ft/hr until landing the curve at about 12,200 feet. After a trip for a mud motor, drilling resumed with an average ROP of 38ft/hr. After drilling 600 feet another trip was made for tool failure. As drilling resumed, fluid conditioner concentration was increased by .25 ppb, based upon Houston laboratory recommendations to improve cuttings integrity and low gravity solids removal. A weighted sweep brought no visible increase in cuttings at the shakers.

ROP increased to the 90-120 ft/hr range, culminated in 1,003 feet being drilled in a 24-hour period. This was a daily footage record by this active Haynesville operator as compared to any previous Haynesville well, nearly all of which had employed OBM. After a short cleanup trip, drilling resumed, averaging 30 ft/hr while sliding and 50 to 80 ft/hr while rotating. In the next 24-hour period, 1,110 feet were drilled, setting a second consecutive footage record by this operator.

Another trip for a broken shaft on the mud motor served as a cleanup trip. At this point the low gravity solids (LGS) concentration of the fluid had increased to above 6% adversely affecting fluid lubricity, as measured by coefficient of friction testing. To improve lubricity by reducing LGS concentration, 250 barrels of active fluid was pumped into a frac tank and replenished with newly-built fluid.

As drilling continued and approached total depth, the proprietary drilling performance enhancer/lubricant concentration was increased to 4% to aid the casing run. The ROP for the last 1000 feet of the lateral averaged 30-45 ft/hr. Upon reaching total depth at about 17,000 feet, cleanup began consisting of wiper trips, backreaming and circulating during each. Any tight spots were backreamed until clean.

Total interval length was about 5,400 feet.

Casing (5.0" OD/4.04" ID) was run into the well to beyond 11,500 feet without incident, then washed and reamed

to within 100 feet of total depth (about 17,000 feet MD). Casing is also typically rotated in Haynesville applications when OBM is employed.

Overall, the system performed as expected, with penetration rates and wellbore stability matching those of prior OBM applications.

Subsequent Wells in the Series

System performance continually improved and total fluid cost was reduced on subsequent wells in the four-well evaluation as lessons learned were applied and as the fluid was recycled from well to well.

The system was displaced immediately following cement and used to drill the shoe on the second well with no adverse affects, and on every well thereafter. This practice is aided by pre-treating the suction pit with 1 ppb sodium bicarbonate prior to displacement.

Experimentation established low vis sweeps followed by high vis sweeps as the most effective method to optimize hole cleaning.

Penetration rates mirrored those achieved on the initial well, as highlighted by a 900-foot day on the last day of a 5100-foot lateral on well #2, a 1,262-foot day in the lateral section of well #3 and a 1,062-foot day on well #4.

Casing was successfully run and cemented in place at total depth without incident on all three subsequent wells.

High Temperature Applications

The system was later introduced in the higher temperature environment of Red River Parish, in wells featuring bottom hole temperatures exceeding 350°F. In two initial applications there, the system was further stressed by hostile conditions that included elevated chloride levels for the fluid exceeding 5000 ppm.

Superlatives in those applications included drilling one lateral interval of 6,100 feet in six days, and a record casing run for the operator in a Haynesville well. Both production intervals exceeded 5,000 feet in length.

Haynesville Results Overview

This application-specific formulation has been successfully employed to repeatedly and successfully drill and case dozens of Haynesville wells. It has consistently provided outstanding ROP and reductions in torque and drag equal to or exceeding those achieved with OBM. Reduced interval days and casing days achieved with the new system prove repeatability and validate initial results.

Reductions in circulating temperature as compared to direct OBM offsets have been observed with the water-based fluid in use. This temperature reduction results from improved thermal conductance of water as compared to diesel, which results in more rapid cooling of the fluid while on the surface. Typical circulating temperature reductions as observed range from 10° up to over 20° F. These lower circulating temperatures should also diminish rate of MWD failure resulting from elevated OBM circulating temperatures.

Overall cost of the recyclable system also compared

favorably to OBM and its inherent ancillary costs.

Statistical Evaluation: Haynesville Performance Results

A 24-well evaluation^[5] compared operational results achieved with the WBM versus 11 OBM applications, using data provided by area operators. Haynesville wells used for the analysis encompassed three Louisiana parishes—DeSoto, Red River and Sabine—where use of the new system was most common in its first year. The similarity of horizontal intervals on Haynesville wells in terms of interval length, relevant geology and mud system deployment allowed for a straightforward comparison of cost and operational data in comparing WBM and OBM results.

Though all wells used in the evaluation were virtually identical in terms of total depth, kick-off point, production interval length and lateral length, those factors were normalized for analysis. The average total depth of all wells used in the analysis was 16,330 ft measured depth; the lateral production interval length averaged 5,350 ft. These averages were weighted according to the number of wells drilled by each operator in the analysis.

OBM presents key ancillary cost issues to Haynesville operators. Operator-provided spreadsheet costs regarding OBM logistics and disposal averaged about US\$170,000 per well in the eleven OBM offset wells studied, compared to \$14,700 per well for the 24 wells employing the new water-based fluid—a savings of over \$155,300 per well on fluids handling.

The actual fluid cost of the WBM also compared favorably to OBM. In terms of cost/ft, the WBM was more cost-effective at \$25.41/ft versus \$27.66 for the OBM. Calculated OBM cost included diesel used on location for system dilution as drilling progressed. The WBM's favorable cost was aided by its ability to be re-cycled from well to well after standard reconditioning, including adjustment of low-gravity solids. Recycling the fluid from well to well resulted in progressively lower mud cost over repeated use in a series of wells.

In the evaluation group comparison, wells drilled with the WBM averaged 7.5 fewer days to drill the interval as compared to the OBM group. From an operator-provided average of \$60,000 total spread cost/day, those days saved resulted in cost savings for operators of about \$450,000 per well.

Average interval drilling footage for wells employing the water-based system was about 332 ft/day, compared to about 242 ft/d for the OBM offsets. This daily footage improvement resulted from consistently improved penetration rates and elimination of non-productive time associated with preparing the rig and location for use of oil-based fluid.

Time spent reaming and running casing was virtually the same regardless of the system in use. In both OBM and WBM wells, these totals were consistent with generally accepted field averages. The method by which total well cost and drilling days were calculated also inherently includes total

non-productive time on all wells analyzed, and is therefore included in comparative results.

All told, savings realized from the elimination of OBM ancillary cost, lower overall drilling fluid cost, days saved per well, improved penetration rates and daily footage totaled about \$700,000 per well.

Mixing Equipment Requirements

The efficiency of the fluid mixing process and initial quality of freshly-mixed fluid depend largely on the equipment employed and its arrangement. Recommended equipment consists of: (1) a mixing tank of 200-bbl or greater capacity, with a self-contained high-volume diesel pump and mixing hopper; (2) a bulk barite tank to be used only for weighting up volume in the mixing tank; and (3) sufficient storage (frac tanks) to accommodate the required operating volume as well as any excess volume generated during drilling.

While the pump dedicated to the mixing tank can transfer volume to the frac tanks, a second pump is recommended for transferring fluid from the frac tanks to the active system. All mixing-related equipment should be arranged to allow access by barite and vacuum trucks to their respective tanks, and fork-lift access to the hopper. Once the initial fluid is mixed, it is recommended that all tanks and pumps remain on location for the duration of drilling operations for subsequent mixing of dilution volume. Location size and design should consider the footprint and configuration of this equipment when possible, or if other factors dictate that volume be mixed off-site and transported to the rig.

Rig Preparation

Pits, lines, and equipment should be cleaned during the casing run, and the procedure should be completed as thoroughly as it would be for the transition to OBM. If the polymer fluid is to be used to displace cement and bump the plug, sufficient volume should be transferred to a frac tank positioned near, and connected to the cement unit. The remainder of the polymer fluid can be transferred from storage to the active pits when clean-up is finished.

Shakers should initially be dressed with API 100-mesh screens for the first two or three circulations until the fluid system is homogenized and sheared, then replaced with API 140-mesh, which have proven optimum in most cases.

Conclusion

This application-specific drilling fluid formulation has achieved solid field performance results that confirm laboratory-based projections of its lubricity, thermal stability and contaminant resistance under demanding Haynesville drilling conditions. Its successful application marks the first ever water-based fluid to repeatedly be used in these long lateral production intervals.

The proprietary drilling performance enhancer provides

outstanding ROP and excellent wellbore lubricity that met or exceeded OBM results in support of drilling and casing operations. The viscosifying polymer/coating agent, newly introduced for drilling fluid function, exhibits excellent thermal stability and contaminant resistance in field use.

When fostered by prudent drilling practices, the system offers expanding promise as a cost-effective, eco-appropriate alternative to OBM for unconventional hydrocarbon field development in the Haynesville and in other similar applications.

Nomenclature

<i>meq</i>	=milligram equivalent
<i>bbl</i>	= barrel
<i>HSE</i>	= Health, Safety and Environmental
<i>ppb</i>	=pounds per barrel
<i>ppm</i>	=parts per million
<i>ppg</i>	=pounds per gallon
<i>MWD</i>	=Measurement While Drilling
<i>ECD</i>	= Equivalent Circulating Density
<i>RPM</i>	=revolutions per minute

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Figure I

Downhole Simulation Test Conditions

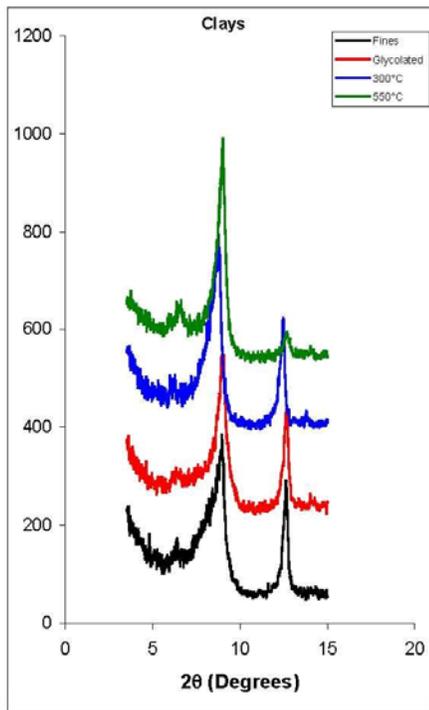
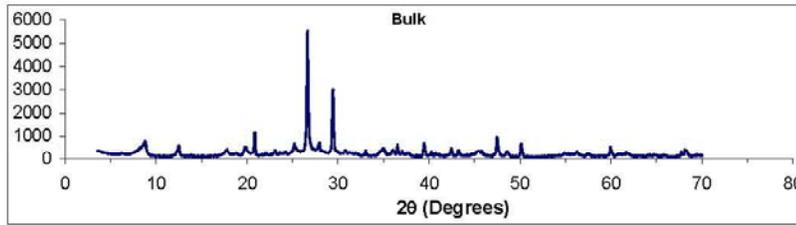
Axial stress:	2500 psi
Horizontal:	2200 psi
Wellbore:	1500 psi
Pore:	1500 psi
Temperature Shale:	235° F
Temperature Fluid:	235° F
Circulating rate while drilling:	1.0 gpm
Circulating rate after drilling:	1.0 gpm
Sand pack (Pore) fluid:	LVT-200

Figure II

X-Ray Diffraction Interpretation and Data

Project: Haynesville Shale

Sample: 7/14/2010



Bulk Composition -	wt%
Total Clay	52
Muscovite-Mica	16
Quartz (SiO ₂)	16
Calcite (CaCO ₃)	14
Barite (BaSO ₄)	2

Clay Composition -	wt%
Illite	68
Kaolinite	17
Mixed-layer	9
Chlorite	4
Smectite	2
Illite/smectite	35 / 65

CEC -	meq/100 g
	11.0

Exchangeable Bases -	meq/100 g
Calcium	10.7
Sodium	1.7
Potassium	1.3
Magnesium	0.3

X-Ray Fluorescence Data -	Mass%
Oxide	
SiO ₂	58.0
Al ₂ O ₃	24.0
Fe ₂ O ₃	4.3
CaO	3.2
K ₂ O	3.0
MgO	2.4
SO ₃	1.8
Na ₂ O	1.4
TiO ₂	0.5
BaO	0.3
P ₂ O ₅	0.2
PbO	0.2
ZrO ₂	0.1
SrO	0.1
ZnO	0.1
MoO ₃	0.1

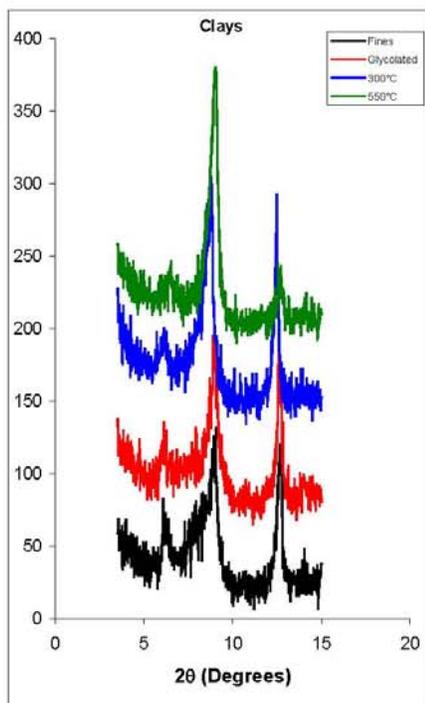
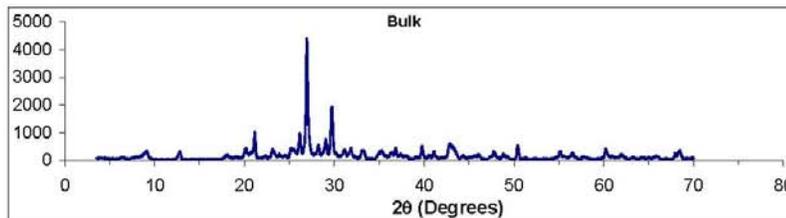
Soluble Cations and Anions -	meq/100 g		
Sodium	6.0	Bicarbonat	4.7
Calcium	0.9	Carbonate	2.4
Potassium	0.0	Sulfate	0.4
Magnesium	0.0	Chloride	0.1

Figure III

X-Ray Diffraction Interpretation and Data

Project: **Bossier Shale**

Sample: **6/16/2009**



Bulk Composition -	wt%
Total Clay	45
Muscovite-Mica	33
Calcite (CaCO ₃)	10
Barite (BaSO ₄)	6
Quartz (SiO ₂)	5

Clay Composition -	wt%
Illite	58
Kaolinite	20
Mixed-layer	14
Smectite	5
Chlorite	4
Illite/smectite	28 / 72

CEC -	meq/100 g
	13.3

Exchangeable Bases -	meq/100 g
Calcium	10.8
Potassium	0.8
Sodium	0.4
Magnesium	0.4

X-Ray Fluorescence Data -	Mass%
Oxide	
SiO ₂	52.8
Al ₂ O ₃	19.8
BaO	7.0
Fe ₂ O ₃	4.1
CaO	4.0
SO ₃	3.4
K ₂ O	2.5
Na ₂ O	2.4
MgO	2.3
TiO ₂	0.4
Cl	0.2
P ₂ O ₅	0.2
PbO	0.2
SrO	0.2
ZnO	0.1
ZrO ₂	0.1

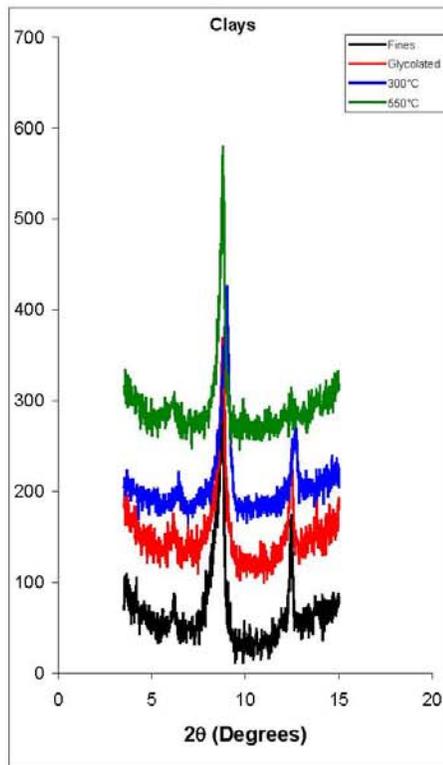
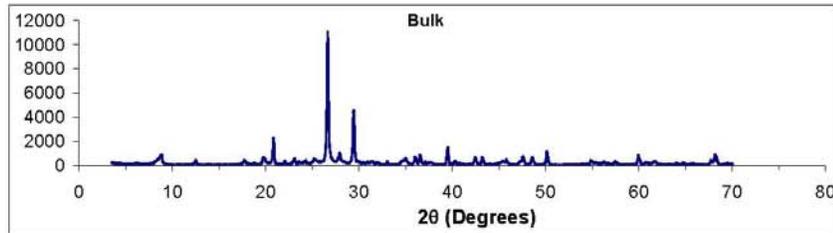
Soluble Cations and Anions - meq/100 g			
Sodium	5.5	Chloride	7.5
Calcium	5.1	Bicarbonat	1.4
Potassium	1.0	Sulfate	1.1
Magnesium	0.3	Carbonate	0.0

Figure IV

X-Ray Diffraction Interpretation and Data

Project: Initial DSC Test

Sample: Haynesville Core



Bulk Composition -		wt%
Total Clay		45
Quartz (SiO ₂)		23
Muscovite-Mica		17
Calcite (CaCO ₃)		15

Clay Composition -		wt%
Illite		86
Kaolinite		21
Smectite		6
Chlorite		3
Illite/smectite		na

CEC -	meq/100 g
	19.5

Exchangeable Bases -		meq/100 g
Calcium		9.3
Sodium		0.9
Potassium		0.7
Magnesium		0.4

X-Ray Fluorescence Data -		Mass%
Oxide		
SiO ₂		64.7
Al ₂ O ₃		19.0
CaO		4.2
K ₂ O		3.0
Fe ₂ O ₃		2.7
MgO		2.0
SO ₃		1.5
Na ₂ O		1.4
TiO ₂		0.3
BaO		0.3
P ₂ O ₅		0.3
PbO		0.2
ZrO ₂		0.1
SrO		0.1

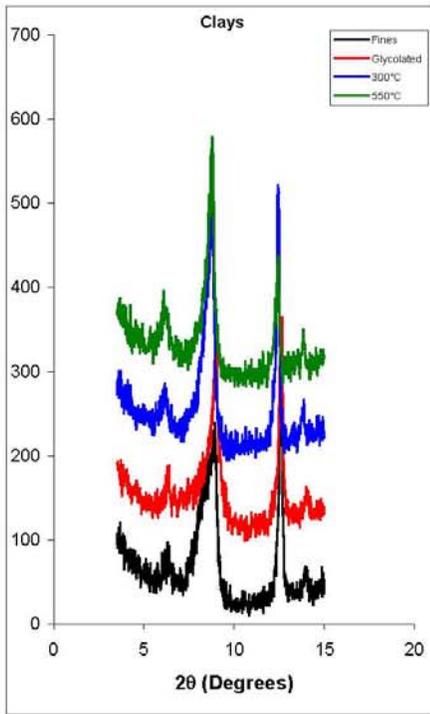
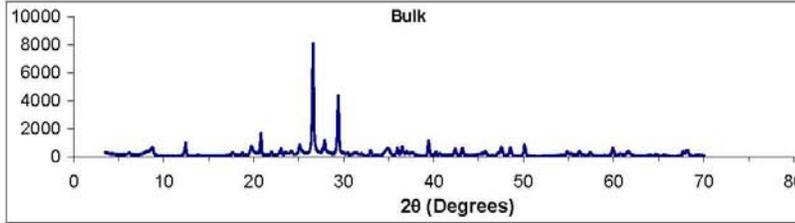
Soluble Cations and Anions - meq/100 g			
Sodium	3.6	Chloride	2.5
Calcium	2.2	Bicarbonat	2.1
Potassium	0.2	Sulfate	0.1
Magnesium	0.1	Carbonate	0.0

Figure V

X-Ray Diffraction Interpretation and Data

Project: After DSC Test

Sample: Haynesville Shale



Bulk Composition -	wt%
Total Clay	48
Muscovite-Mica	19
Quartz (SiO2)	17
Calcite (CaCO3)	14
Barite (BaSO4)	1

Clay Composition -	wt%
Illite	61
Kaolinite	21
Mixed-layer	13
Chlorite	4
Smectite	1
Illite/smectite	26 / 74

CEC -	meq/100 g
	11.5

Exchangeable Bases -	meq/100 g
Calcium	9.8
Sodium	0.9
Potassium	0.9
Magnesium	0.5

X-Ray Fluorescence Data -	Mass%
SiO2	60.6
Al2O3	20.8
CaO	4.6
Fe2O3	3.7
K2O	2.9
MgO	2.3
SO3	2.1
Na2O	1.7
TiO2	0.4
P2O5	0.3
BaO	0.2
PbO	0.2
Cl	0.1
MoO3	0.1
SrO	0.1
ZnO	0.1
ZrO2	0.1

Soluble Cations and Anions -	meq/100 g		
Sodium	6.8	Bicarbonat	2.7
Calcium	0.8	Chloride	1.8
Potassium	0.8	Sulfate	0.7
Magnesium	0.5	Carbonate	0.2

Figure VI

Temperature °F	160	175	186	225	258	284	310
Pressure psig	0	850	1,700	4,240	6,360	9,330	13,570
Plastic Viscosity	40	36	32	25	22	20	20
Yield Point	18	16	17	14	11	11	10
10 sec gel	6	5	5	5	5	5	5
10 min gel	6	5	6	5	6	6	6
6 rpm	8	7	7	6	6	6	6
3 rpm	6	5	5	5	5	5	5

16.3 ppg Fluid with 3% LGS

Figure VII

Temperature °F	160	175	186	225	258	284	310
Pressure psig	0	850	1,700	4,240	6,360	9,330	13,570
Plastic Viscosity	44	37	35	27	25	22	22
Yield Point	14	15	14	17	14	15	14
10 sec gel	10	9	9	9	9	10	10
10 min gel	11	10	9	10	10	11	12
6 rpm	11	10	10	9	10	10	10
3 rpm	10	10	9	9	9	9	9

16.2 ppg Field Mud with 8.5% LGS

Temperature °F	120	200	275	350	425	425	350	275	200	120
Pressure psig	2.5K	5K	7.5K	10K	14K	14K	10K	7.5K	5K	2.5K
Plastic Viscosity	50	32	26	29	41	38	34	23	28	46
Yield Point	37	29	21	27	80	23	22	17	26	30

Figure VIII

17.5 ppg with 6% LGS

Temperature °F	120	200	275	350	400	400	350	275	200	120
Pressure psig	2.5K	5K	7.5K	10K	14K	14K	10K	7.5K	5K	2.5K
Plastic Viscosity	50	31	24	31	47	47	52	67	59	66
Yield Point	35	27	23	29	65	119	87	49	38	43

Figure IX17.5 ppg with 9% LGS
18.5 hours static at
400°F

Temperature °F	120	200	275	350	400	400	350	275	200	120
Pressure psig	2.5K	5K	7.5K	10K	14K	14K	10K	7.5K	5K	2.5K
Plastic Viscosity	67	40	30	31	26	31	38	40	43	45
Yield Point	27	12	11	21	46	39	36	25	26	26

Figure X17.5 ppg with 12% LGS
21 hours static at
400°F

Figure XI

