



LOST CIRCULATION GUIDE

Drilling Specialties Company
a division of Chevron Phillips Chemical Company LP

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OVERVIEW OF THE COMPANY

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The Drilling Specialties sales and service engineers possess a wealth of applications expertise to help our customers utilize our products. While the knowledgeable technical service staff is always ready to provide immediate support from our Drilling Fluids, Cement, Stimulation, and Polymer laboratories. Our experienced customer service group ensures quick product availability from our global distribution system. To meet your operation's needs, Drilling Specialties Company products are never far away.

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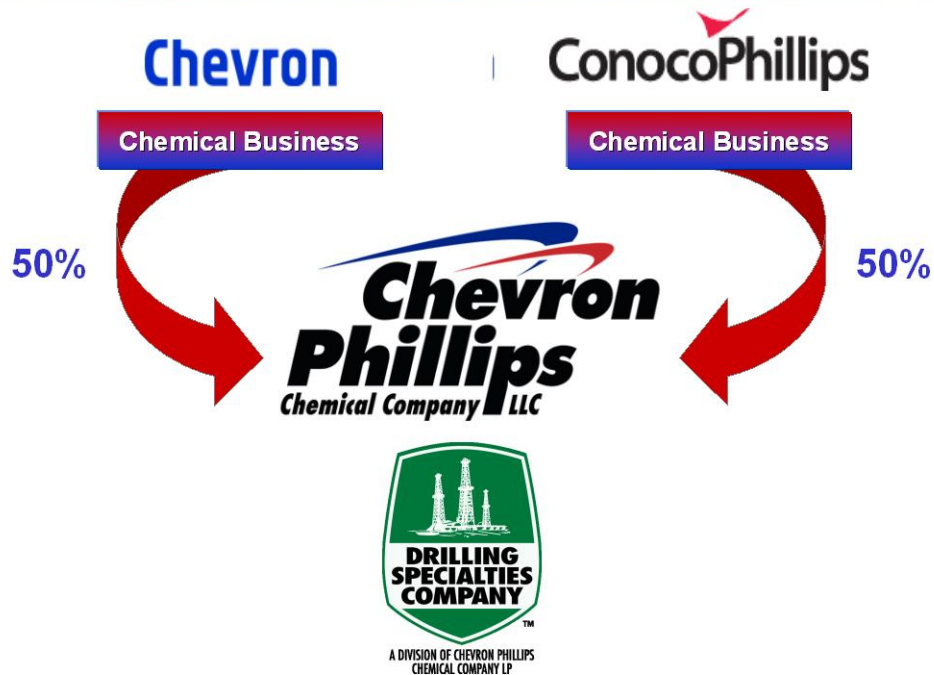
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LOSS OF CIRCULATION

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LOSS OF CIRCULATION

FOREWORD

In a broad survey conducted by Drilling Specialties Company loss of circulation was identified as the most costly problem faced by the oil and gas industry associated with drilling fluids. The purpose of this CD is to inform the reader of the nature of this problem and ways to solve it.

This CD was prepared by a team of individuals that combined have over 180 years experience in the drilling fluids business. The team reports to Bill R. Holvey Western Hemisphere Sales and Marketing Manager for Drilling Specialties Company. The Team wishes to thank management for its support of this project.

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INTRODUCTION

LOSS OF CIRCULATION: Is the loss of whole drilling fluid (mud) at any depth to the formation.

LOSS OF CIRCULATION FORMATION TYPES

1. Coarsely permeable unconsolidated formations such as sand, pea gravel and some coarse gravel beds, shell beds and reef deposits
2. Vugular and cavernous formations such as; reefs, limestone, chalk and dolomite formations.
3. Fissures or fractures, both natural and induced

Arguably, one of the most time consuming and cost inflating events in the drilling operation is that of loss of circulation. It has been estimated to cost the drilling industry over one billion dollars annually in rig time, materials and other financial resources. In a continued effort to inform the reader and contribute some solutions to this problem, Drilling Specialties Company, a division of Chevron Phillips Chemical Company LP, has undertaken the task of producing a CD to address this problem. A particularly unique aspect of this CD is a LCM (loss circulation material) calculator to assist the reader in formulating a suitable remedy. In order to produce a CD that is easy to understand and relatively free of technical abstraction we have divided the content of the CD into three major descriptive sections. These are seepage losses, moderate loss and severe or complete loss of returns. In each of these sections we will describe the nature of the loss and recommend ways to resolve the problem.

SEEPAGE LOSS

For purposes of this discussion we will define a seepage loss of a whole mud that is less than 10 barrels per hour for oil based drilling fluids and 25 bbl/hr for water based drilling fluids. This rate of loss may be caused by a number of factors resulting from the drilling operation or it may only be a perceived loss. That is, fast-drilling rates in a reasonably competent formation can result in the perception of having a seepage situation where none actually exist. Example:

When high drilling rates are attained even in hole sizes as small as 8.5" suspected seepage loss can actually be attributed to other factors. One such factor is normal displacement of drilled solids with fluids. For example, while drilling 50 feet per hour in a 8.5" hole, 3.5 bbls of whole drilling fluid per hour will be required to fill the new hole drilled. Another source of perceived seepage loss is the drilling fluid retained on drilled solids removed from the system. One estimate or rule of thumb of the amount of mud loss attributed to this effect is 1 bbl of mud per bbl of cuttings drilled. So, as in the previous example, where 8.5" hole is drilled at a sustained rate of 50 feet per hour, an additional 3.5 bbl of mud would be lost on the drill cuttings in the period of one hour or 50' of hole. This would total 7 bbl of perceived mud lost per hour or 56 bbls over an 8 hour tour. In a full 24 hours of drilling this loss would be 168 bbl giving the impression of a nonexistent problem. Thus prior to initiating any treatment for suspected seepage loss it is necessary to take these factors into account. Of course, the larger the bore hole, the greater the effect.

To effectively deal with a previously outlined seepage loss, one must understand the possible causes. In addition to the example outlined above there are several common causes of this type of loss of circulation. Factors such as ECD (equivalent circulating density), generally overbalanced drilling fluid to formation pressure, or the nature of the formation being drilled are some of the causative considerations to be taken into account. Perhaps the most important aspect of this drilling condition is to be able to separate actual losses from those merely perceived. Volume totalizers, tattle tails or similar devices can also be used to gauge the magnitude of actual losses.

When the reason for the loss is isolated a course of remedial action can be initiated. Depending on what stage the drilling operation is in and the severity of the seepage loss, the operator may choose to ignore the event entirely. That is, if the operation is making good hole and losses can be tolerated for a short period of time (nearing a casing point) with an inexpensive drilling fluid, a simple economic analysis may indicate ignoring the problem would be the best solution.

Another avenue to consider would be to continue the drilling process and do nothing relying on the accumulated abundance of fine drill solids to heal the hole and stop the mud losses. This approach is recommended only with considerable caution, as a build up of fine-drilled solids will adversely affect the drilling fluids rheological properties. Thus while we remedy one problem we may create a more expensive one.

A second approach to addressing this type of loss would be to stop drilling, shut the mud pumps down, and pull up past the suspected loss zone and allow the hole to heal. Normal rig maintenance (cut and slip drill line etc.) is usually carried out during this type of action to minimize rig down time. The process usually takes about two hours.

A third and possibly the best method to combat this type of loss is to treat the entire system either before drilling the suspected theft zone (preventative) or as the event occurs (remedial). If pre-treatment is chosen as a preferred course of action, it is recommended that the fluid be treated with 4-6 ppb (pounds per barrel) of a fine grade of LCM. This fine LCM may take the form of a fine calcium carbonate (CaCO_3) or an assortment of fine micro-cellulosic fibers or combination of both. Carbon-based materials such as deformable graphite or Gilsonite (uintaite) may also be used. Some of these LCM materials will expand and contract in response to changing temperature and differential pressures and thus may be considered a more permanent solution. These materials can be used alone or in conjunction with other similar size grades of LCM. One precautionary comment about the use of calcium carbonate needs to be made here.

Calcium carbonate will increase the mud weight in use, which may contribute to other problems such as a higher ECD and overbalanced drilling.

Special consideration must also be given to the optimization of solids control equipment. This is necessary to retain the LCM in the fluids system. The following remedial procedures are offered for consideration.

REMEDIAL PROCEDURE: SLUG TECHNIQUE IS AS FOLLOWS

1. Rig up to isolate +/- 75 bbl of returns.
2. Put 50 bbl whole drilling fluid in slugging pit.
3. Add 15 ppb of micro cellulose fiber and 15 ppb of fine calcium carbonate.
4. Mix thoroughly – may need small additions of water, thinner or wetting agent if rheology gets too high.
5. Pump pill to bit, reduce to slow pump rate around to surface.
6. Bypass shale shaker when screens begin to blind, isolate pill for reuse.
7. Gradually increase to full pump rate and monitor system for losses.
8. Repeat if necessary.
9. The reuse of the pill not only saves money but also reduces the build up of fine particles in the mud system that in turn can lead to increased ECD's.

Another treatment procedure that can be used to combat potential seepage losses is to slug the hole periodically with high concentration LCM pills. These pills are usually formulated in the +/- 80 ppb range and can be a blend of several components exhibiting a variety of sized particles. This blend of sizes results in an LCM pill that actually acts as a bridging agent to maximize the pill effectiveness. At the conclusion of the procedure, the pill can simply assimilate into the existing system and allowed to circulate during continued drilling. If seepage losses persist, it may be necessary to drill with LCM in the system,

if so the following is recommended:

1. Add 5 ppb fine calcium carbonate to the entire active system
2. Drill ahead and monitor for losses keeping LCM concentrations steady to accommodate for normal mud losses due to new hole drilled and losses for cuttings.
3. Supplement fine calcium carbonate with micro cellulose fibers.

PREVENTION OF SEEPAGE LOSSES TREAT THE ENTIRE SYSTEM

Quick Reference Guide

<i>Products</i>	<i>Mud Weights</i>				<i>Comments</i>
Mud Weights PPG	7.0 to 12.5	12.5 to 15.0	15.1 to 17.0	17.1+	Add recommended amounts to active system.
<i>Micro-fiber Fine</i>	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	
<i>Micro-fiber Medium</i>	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	
<i>Micro-fiber coarse</i>	0	0	0	0	
<i>Calcium Carbonate sized</i>	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	
Total ppb LCM	4 - 15 ppb	4 - 15 ppb	4 - 15 ppb	4 - 10 ppb	

REMEDIAL TREATMENTS FOR SEEPAGE LOSSES AS A SLUG TECHNIQUE

Quick Reference Guide

<i>Products</i>	<i>Mud Weights</i>				<i>Comments</i>
Mud Weights PPG	7.0 to 12.5	12.5 to 15.0	15.1to17.0	17.1 +	Pump sweeps while drilling depleted sands every connection then periodically as needed to Total Depth.
<i>Micro-fiber Fine</i>	10 - 25 ppb	10 - 25 ppb	10 - 15 ppb	5 - 10 ppb	
<i>Micro-fiber Medium</i>	10 - 25 ppb	10 - 25 ppb	10 - 15 ppb	5 - 10 ppb	
<i>Micro-fiber Coarse</i>	0	0	0	0	
<i>Calcium Carbonates sized</i>	10 - 40 ppb	10 - 30 ppb	15 - 25ppb	10 - 15 ppb	
Total ppb LCM	30 - 80+/- ppb	30 - 75+ ppb	25 - 50 ppb	20 - 30 ppb	

CARBON-BASED MATERIAL USED TO TREAT SEEPAGE LOSS

Carbon-based materials known as deformable graphite are used to prevent loss of circulation in porous and fractured formations and may be used in any drilling fluid. When added to a drilling fluid they become tightly compressed into porous formations and fractures. They will then expand and contract with the formation without being dislodged. As these types of materials are more expensive they may be supplemented with calcium carbonate.

PREVENTION OF SEEPAGE LOSSES TREAT THE ENTIRE SYSTEM

Quick Reference Guide

<i>Products</i>	<i>Mud Weights</i>				<i>Comments</i>
Mud Weights PPG	7.0 to 12.5	12.5 to 15.0	15.1to17.0	17.1+	Add recommended amounts to active system.
<i>Deformable graphite LCM Fine</i>	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	
<i>Calcium Carbonate Fine</i>	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	
<i>Total ppb LCM</i>	10 - 20 ppb	10 - 20 ppb	10-20 ppb	5 - 10 ppb	

MODERATE LOSS

Moderate loss of circulation is usually defined as those losses that are less than total. In oil based drilling fluids more than 10 barrels per hour but less than 30 is commonly accepted as moderate. In water based fluids 25 to 100 bbl/hr is an acceptable range. When circulation is lost while drilling, the cause may be several fold. That is, when the mud pumps are shut down and the hole stands full, the loss is said to be caused by a marginal increase in bottom hole pressure (BHP) due to a hydraulic pressure drop in the annulus. This condition is also a direct result of apparent ECD conditions. Circulation may also be temporarily lost due to pressure surges induced while running casing pipe or because bottom hole pressures are exceeded when breaking circulation after a trip.¹ Losses of this nature are always treated by remedial techniques and procedures. Usually, these remedies are attained by simply adjusting the physical parameters of the drilling fluid in use. A reduction in the solids content of the fluid or yield point (YP) values rather than the application of lost circulation materials is the recommended course of action. The following procedures are also recommended as possible solutions:

1. Evaluate offset well data to determine proper fluid weights and casing placement and carry the lowest mud density consistent with well safety and borehole stability.²
2. Establish formation integrity with pressure test at the most recent casing depth.
3. Use the lowest circulation rate that will clean the hole adequately.³
4. Adjust the rheological properties to give maximum hole cleaning with minimum pressure drop in the annulus.⁴
5. Do not drill with a balled bit, drill collars, stabilizers, or tool joints as this tends to close off the annular clearance. Avoid excessive wall-cake build up by reducing filtration rates.⁵
6. Run pipe slowly, and above all, do not ream down rapidly with the pumps on.⁶
7. Break circulation several times on the way into the hole and rotate the pipe. When on the bottom, break circulation slowly, and raise pipe while doing so.
8. Minimize gel strength of the drilling fluid.⁷ Avoid high temperature gelation.
9. Control rates of penetration to avoid excessive solids loading in the annulus.
10. Finally pull pipe into protective casing or 3 to 4 stands off bottom and allow hole to heal by waiting 4 to 10 hours. Applicable to induced fractures and works about 50 percent of the time.

* Footnotes 1, 2, 3, 4, 5, 6 and 7 are summaries of pages 437 and 438 from The "Composition and Properties of Oil Well Drilling Fluids" 4th addition.

Depending on the magnitude of the loss and the physical characteristics of the formation incurring the loss, a more comprehensive remedial procedure may be

attempted. To maximize the effectiveness of this the drilling fluid in use must be within acceptable conditions. Care should be taken to insure that LCM products to be used do not adversely affect the physical properties of the drilling fluid or tools in the hole.

REMEDIAL PROCEDURES

THE LCM PILL USING STANDARD MATERIALS

1. Spot a 50 bbl pill consisting of 10 ppb of Fine Calcium Carbonate, 10 ppb of Coarse Calcium Carbonate and 10 ppb of fine or medium fiber your choice. *Mica and other flake type LCM's result in a lower frac propagation pressure and are not recommended! Regional differences may mean adaptations run up to 100 ppb LCM materials.* Size LCM to restrictions in Bottom Hole Assembly (BHA) consult tool manufacture or Directional Drilling Service Company if needed.
2. Attempt to keep annulus full with water or existing drilling fluid and monitor fluid level. If drilling fluid level in the annulus stays full – calculate amount of water and cut the mud weight in pits back to the equivalent hydrostatic pressure in hole, if possible.
3. If hole stays full for one hour, stage slowly into open hole and gently break circulation. Attempt to circulate at a slow pump rate.
4. Repeat if necessary 2 to 3 attempts.
5. Avoid the use of coarse LCM's that require by passing of the solids control system as this will result in a build up of fines in the system that will increase viscosities and equivalent circulating densities (ECD's) and in turn lead to more losses.
6. If the above fails then prepare High Solids High Filtration Slurry and perform a squeeze.

MODERATE PARTIAL LOSSES

TREATING THE ENTIRE SYSTEM FOR MODERATE PARTIAL LOSSES

Quick Reference Guide

Products	Mud Weights				Comments
Mud Weights PPG	7.0 to 12.5	12.5 to 15.0	15.1 to 17.0	17.1+	Pump sweeps as needed! Do not over treat as this can lead to a build up of solids and increased ECD's
<i>Micro-Fiber Fine</i>	10 ppb	10 ppb	10 ppb	10 ppb	
<i>Micro-Fiber Medium</i>	10 ppb	10 ppb	10 ppb	10 ppb	
<i>Micro-Fiber Course</i>	10 ppb	10 ppb	10 ppb	10 ppb	
<i>Calcium Carb F.</i>	10 ppb	10 ppb	10 ppb	10 ppb	
<i>Calcium Carb C.</i>	10 ppb	10 ppb	10 ppb	10 ppb	
<i>Total LCM</i>	30 - 50 ppb	30 - 50 ppb	30-50 ppb	30 ppb	

Treating the entire drilling fluid system is another method of resolving a moderate loss of circulation condition. This method is especially effective when dealing with non-weighted systems using a variety of LCM materials and where good solids control equipment is not available or in use. Treating the entire system with 20 – 30 ppb of fine calcium carbonate and 10 – 20 ppb of fine micro cellulose fibers is an option that allows continued use of the shakers with acceptable losses of LCM material.

Weighted drilling fluids, on the other hand, are not usually treated in their entirety due to the requirement of imposed adequate solids control practices to control desired drilling fluids properties. Solids control equipment such as hydro cyclones; flow line cleaners; mud cleaners; and centrifuges cannot be used when treating the total circulating system unless very fine LCM is used. Again caution should be exercised when using products like fine calcium carbonate, as this will cause an increase in mud weight. Similarly, fine flake materials such as mica and some fiber materials **should be avoided** as this may increase the resistance to flow and thus increase surge and swab pressures. Another preventive measure is to squeeze the problem area with deformable graphite.

Note on LCM materials: Lost circulation materials come in many different forms, each may possess a specific advantage such as cost, availability, performance and effect or lack of effect on drilling fluid properties. There is no magical product as performance of an LCM is controlled primarily by its concentration and distribution of its particle size and shape. Particle size is mostly controlled by the sorting or milling process and particle shape by the source of material.⁸

It is commonly thought that the ratio of particle size of the mud to the fracture gap width controls the bridging process. When the ratio of the particle size to fracture gap width is less than 1/6, whole drilling fluid will pass through the formation and bridging will not occur. A ratio of 1/2 or greater will cause bridging and the formation of a filter cake to form immediately. Values in-between these two ratios will permit particle invasion and bridging until the bridges ratio is greater than 1/2 and the filter cake forms.⁹

The decision almost always these days is what concentration of what size material can be pumped through the mud motor / MWD / BHA or bit nozzles and what has the best chance for success for that concentration of particle size distribution.

Footnotes 8 and 9 comes from "Evaluation of Lost Circulation Materials for Water-Based Muds F96-P-72 September 30th, 1996 962780002-Tul, EPTG-AMOCO

WELLBORE STRENGTHENING: USING DEFORMABLE GRAPHITE A SIMPLE APPROACH

A method of treating for potential loss of circulation is to slug the hole with very high (50 - 100 lb/bbl) concentrations but do it by spotting the high concentration slug across the zone of concern during trips. On trips the swab and surge pressure would effectively create fractures for a very short time and then allow them to close. This seems like an obvious best practice. Various oil and gas companies report they have achieved formation strength increases of up to 1000 psi by doing small fracs and allowing them to stay propped open with deformable graphite. This would have the potential to give the desired result with no increase in cost or time. Spotting a pill of deformable graphite ahead prior to running casing and cement is good practice if there is a zone exposed that is unlikely to support the cement. Running casing can give a high surge pressure effectively producing the equivalent of a series of short hesitation squeezes. This gives the formation a very high concentration exposure with the potential to seal any fracs exposed.

WELLBORE STRENGTHENING PROCEDURE

There are two ways to apply a wellbore strengthening treatment to a weak zone.

1. The drilling ahead method.
2. The Pill method or formation integrity test (FIT) method or leak off test (LOT) method.

DRILLING AHEAD METHOD

Treat the entire drilling fluid system with fine deformable graphite LCM, and drill the weak zone with the LCM in the drilling fluid. Since the LCM has a large particle size, the shakers have to be fitted with 20 or 40 mesh screens or coarser to keep the effective material in the system (or bypass the shakers altogether). It is desirable and necessary to keep the full particle size spectrum in the system. As the weak zone is drilled, small short fractures will be created, but filled and plugged with the LCM. The weak zone will be sealed and strengthened. As the mud weight (density) is raised, more small fractures are formed and plugged. This method is the most effective but is best to limit drilling to about 500 feet because of the build up of drill solids in the mud system. See formula below for treating the entire system.

THE PILL METHOD

Before drilling out of the shoe, determine the depth of the expected "weak" section. Conduct a formation integrity test (FIT) or leak off test (LOT). If the shoe test fails, drill this section with treated mud as far as it is necessary to get to a formation that will have the desired rock strength. The depth will have to be predicted based on offsets and other data. All formations below this point should be strong enough to withstand the highest ECD predicted for the rest of the well. In other words, choose a point where a drilling liner would be set.

Change shaker screens to 20 mesh or coarser, or bypass shakers. Before drilling ahead add the graphitic material and CaCO₃ adding CaCO₃ to the mud for shales is not a functional treatment! In sands, with much larger pore throats, CaCO₃ is desirable. Adding the materials through the mud hopper will ensure good mixing and distribution through out the mud system and prevent any plugging of MWD tools or mud motors. The special LCM blend will have to be maintained in the mud at all times. The ECD (equivalent circulating density) at the end of this section will have to be the maximum ECD expected at TD (total depth). This ensures that when TD is reached, the upper section will have already been exposed to this ECD with the strengthening LCM blend in the drilling fluid. The LCM blend may be removed from the drilling fluid, and drilling to TD can continue as per plan, as the weak section has been strengthened.

BORE HOLE STRENGTHENING: SQUEEZE PROCEDURE

Deformable graphite is used as a component of a special fluid to drill formations that would be likely to frac with existing drilling fluid density, and which need to be strengthened. For this application, a very clean fluid is used to keep the solids distribution constant. If the formation of concern is sand, then a blend of calcium carbonate and deformable graphite may work as well as straight deformable graphite. For shale formations straight deformable graphite is clearly superior and the calcium carbonate adds little if anything to hole-strengthening! This special fluid may be mixed and stored until the zone of concern is to be drilled and then used just to drill that zone. These fluids in addition to the deformable graphite also have a very low fluid loss. In this case, it is desirable to run more coarse screens to avoid shaking out the coarse fraction of the material. A particle size distribution, up to the size of the fracture, is optimal.

THE FOLLOWING ARE THE RECOMMENDED STEPS FOR A SQUEEZE PROCEDURE

SPOTTING/FIT METHOD OR PILL METHOD

Drill through the entire weak zone with a low mud weight, treat the zone with the special LCM in a series of FIT squeezes, and then drill ahead raising the mud weight as needed for pore pressure or hole stability. This method is operationally easier, but there is a risk that if the wellbore breaks down during the FIT test, the fracture may extend so far from the wellbore that it cannot be repaired.

Drill to the predetermined top of the stronger rock (bottom of the weak rock). Circulate hole clean at this point. While drilling, build LCM pill volume big enough to cover entire open hole plus 100 feet up into the casing plus 10 bbls. Add graphitic material and CaCO₃ through hopper to ensure good mixing. Use the formula below to treat the entire system, except increase graphitic concentration to 20-25 ppb. As long as it is well mixed, this formulation should go through the MWD, mud motor and other down-hole tools without plugging. Check tool tolerances with manufactures or down hole drilling service companies to reduce

or prevent plugging. This pill should have good suspending properties to prevent dropout of the large CaCO₃ in the drill string.

Spot the pill in the open hole and up into the casing, leaving about 10 bbls in the drill pipe. Perform a series of FIT's to drive "wedges" of the LCM material into the wellbore wall at the weak zones. Pump down the drill pipe at a controlled rate of about ¼ bbl/min. Carefully plot pressure vs. time and carefully record volumes pumped and returned. When the pressure/volume line is definitely bending over, stop pumping. If the well breaks down, stop pumping immediately to limit the growth of the fracture. If the fracture gets too far from the wellbore, the method may fail. Bleed the pressure off over about a minute, if possible. Wait 5 minutes to do the next FIT to allow any leak off to occur in the fracture, if the zone is permeable. Repeat until the desired wellbore pressure is achieved. Some mud should have been lost to the formation (not returned when pressure released). This indicates that mud with the LCM blend was forced into short fractures and leaked off. For a long zone, this might be 2-10 bbls.

This section should now be "strengthened". Circulate the pill out of the hole and either separate it for use as a later LCM pill, or let it mix into the drilling fluid. Put the 200 mesh screens back on the shakers and drill ahead as per program.

THE FORMULATION OF THE SPECIAL LCM IS AS FOLLOWS

For treating the entire mud system:

1. 15 ppb of deformable graphite – 250-600 micron. For a 1500 bbl circulating system, this would require 450 50-lb sacks
2. 10 ppb of CaCO₃ in the 200-600 micron range
3. 10 ppb of CaCO₃ in the 50-150 micron range
4. If unweighted, 10 ppb of CaCO₃ in the 5-10 micron range. If the mud has barite in it, then this CaCO₃ fraction is unnecessary.
5. For a formation with no permeability (shale, marl, and tight siltstone) the mud must be ultra-low fluid loss. This would be < 0.5 ml HTHP (high temperature high pressure) fluid loss at maximum BHT (bottom hole temperature). This can be achieved in OBM with a combination of organophillic lignite, Gilsonite, **Soltex® Additive**, or a polymeric fluid loss material, and emulsifiers. The polymeric material may be a key to getting the low fluid loss. An O/W (oil/water) ratio of no higher than 70/30 is preferred.

For treating with a pill:

Use the same formula above, except increase the deformable graphite concentration to 20-25 ppb.

- 1) For an open hole of 1000 feet with a diameter of 8.75" + 10% hole wash out a pill of 100 bbls is required and will take 40 to 50 sacks of deformable graphite. **Remember you must cover all the open hole plus 100 feet of casing plus 10 bbl. for the drill pipe.** There must be a complete distribution of particle sizes from the largest on down so that a filter cake will form on the large plugging particles at the fracture tip. The deformable graphite has been shown to be important for both its particle size distribution and its characteristic of resiliency. CaCO_3 alone is not as effective, especially in impermeable rock. **When treating impermeable rock use only deformable graphite and treat at 50 to 100 pounds per barrel.** Using the same example above the number of sacks of deformable graphite would then be 100 – 200 sacks.
- 2) Depending on the particle size distribution of available CaCO_3 , adjust the formula above. The idea is to have some CaCO_3 at the same size range as the deformable graphite, and some CaCO_3 in the size range between 50 microns and 200 microns – to bridge the gap between barite and the deformable graphite.
- 3) Note; that for long hole sections, an applied pressure at the surface exerts a higher EMW (equivalent mud weight) pressure at the shoe than at TD (total depth). Calculate the range of EMW that will be applied with various applied surface pressures.

Note: *The method of using deformable graphite to strengthen a wellbore must be applied to a wellbore that has not yet broken down. Wellbore Strengthening is a preventive treatment! Once an induced fracture has been initiated and it extends far from the wellbore, this technique will probably not work! It can not be used as a corrective method for lost circulation once a significant drilling fluid volume has been lost!*

SEVERE LOSSES – TOTAL LOSS OF RETURNS

Total loss of circulation is self-explanatory and is almost always treated with remedial procedures and techniques. In oil based fluids losses over 30 bbl/hr are considered severe enough to warrant action. In water based fluids losses over 100bbl/hr are considered severe. The first step to take is diagnosis of the problem. It is of critical importance to be able to determine why and where the loss is occurring as well as formation type and depth of interest. If the depth of the loss is not known with certainty, a loss circulation survey or log suite should be obtained. The added expense of such a survey and diagnostic costs are easily justified in the overall cost of the well. It enables the best remedial treatment to be applied once and avoids the costly possibility of as trial and error approach. Information can often be gained from circumstantial evidence. For instance, if the loss occurs while drilling ahead in a normally pressured zone with no change in mud weight, the fluid will almost certainly be lost into a pre-existing void, which the bit has just encountered. If the loss occurs when pipe is being run into the hole, one may safely assume that a transient surge pressure has induced a fracture.¹⁰

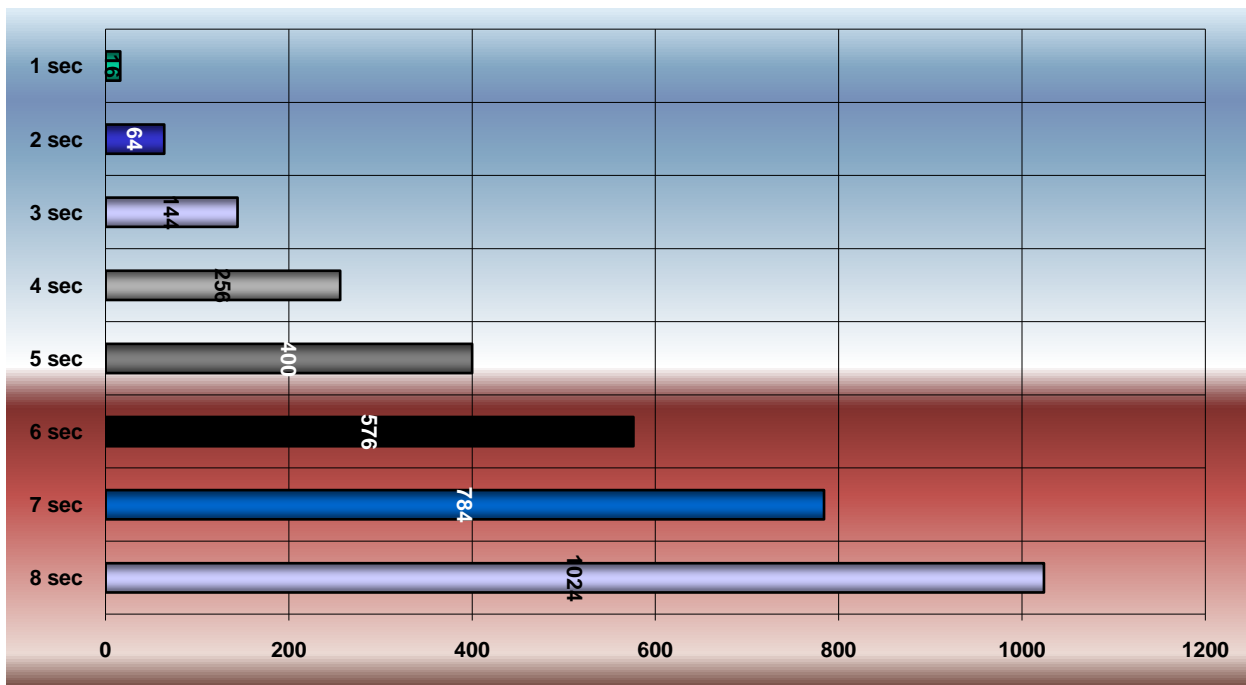
It is also useful to be able to determine the fluid level in the annulus after the pumps are shut down. This is especially true if the local formation pore pressure and fracture gradient is known. Fluid levels may be determined by several methods. Echometer determinations as well as counting pump strokes to fill the annulus are two methods.¹¹



Another technique is illustrated below: FORMULA: $D = \frac{1}{2} at^2$ where D = distance in feet, a = acceleration of 32 ft/sec./sec. And t = time in seconds. Procedure: Form a one-inch diameter ball from a piece of soft clay. Start a stopwatch as the ball is released directly over the borehole. Stop the watch when the ball is heard to hit the fluid in the hole. (If the ball ricochets off the walls on the way down this will delay its fall.) On the graph, follow the proper time-line horizontally to the curve-line intercept. Read downward to the footage scale to read depth to top of fluid in the hole.

DETERMINATION OF FLUID LEVEL IN THE HOLE BY DROPPING A CLAY BALL

APPROXIMATE DISTANCE TO TOP OF FLUID IN HOLE, FEET



** Footnotes 10, 11 are summaries of page 435 Regaining Circulation from Composition and Properties of Oil Well Drilling Fluids 4th addition.

Losses into large caverns occur only at very shallow depths, and are difficult to remedy. Enormous volumes of LCM slurries may be needed to cure such losses, and require non conventional means. Sometimes a cure is not possible and other actions may be necessary. See drilling blind or with aerated fluid page 41.

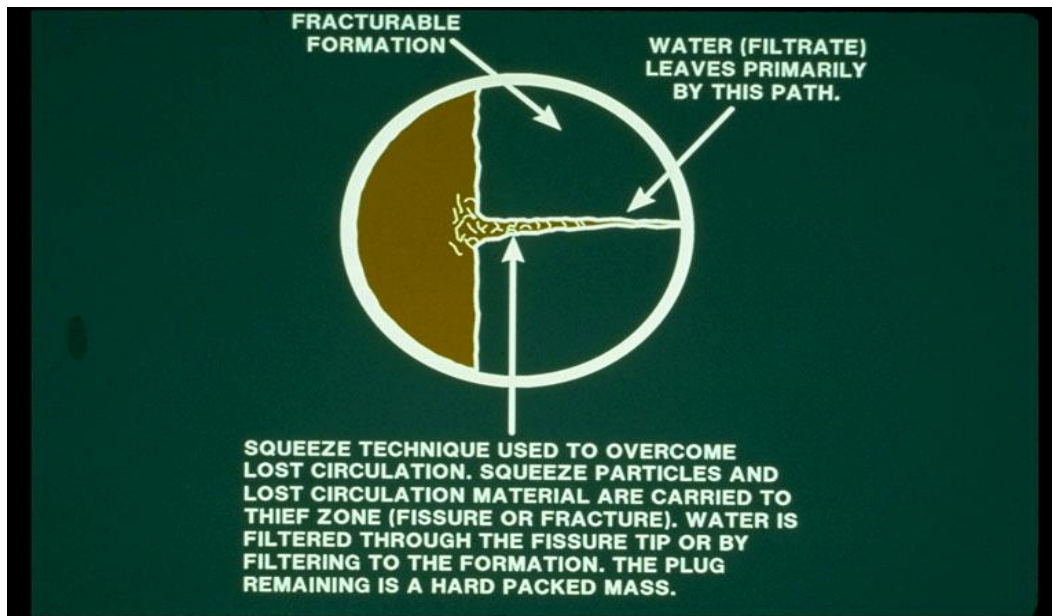
Losses into smaller cavities such as vugular limestone, gravel beds, and induced fractures are best stopped with various types of Reactant Pills, these are loosely classified into three groups depending upon how they achieve their respective plugging action.

REACTANT PILLS

Reactant pills are a treatment to regain control of circulation from either lost circulation or kicks and have been successfully used for many years to seal off zones or pathways to underground flows allowing the flowing zone to be killed. Reactant means that the pill's final properties will be much different after the pill is spotted into the wellbore. The three types of reactant pills are: **Filtration Activated Pills**, **Hydration Pills** and **Chemical Reactive Pills**.

FILTRATION ACTIVATED PILLS OR THE-HIGH-FILTRATE-LOSS SLURRY

The-High-Solid-High-Filtrate-Loss-Slurry and "Hesitation Squeeze" procedure are suitable for sealing fractures or channels in permeable formations. The rapid loss of filtrate deposits a filter cake plug that fills the fracture or small void in the loss zone and not on the surface of the wellbore where it could be dislodged by the drill string. See picture below.



HISTORICAL NOTE

The first high solids high fluid loss squeeze was performed with diatomaceous earth in 1956 by Carl Huber, (Phillips Petroleum Company) and John Crocker (Magcobar) on a Phillips Petroleum Company well north of Pampa, Texas. Drilling Specialties Company then a division of Phillips introduced the first commercial product Diaseal M[®] LCM, in 1964. This one sack product combined several ingredients allowing for a simplified mixing and building of the slurry. Today several companies have altered the formula slightly and present it as something new to the industry. These variations cost several times what a Diaseal M[®] LCM slurry costs to build and pump with little improvement over the original.

Most variations add lime to the product. Both Carl Huber and John Crocker talked of adding extra lime to the slurry if it was deemed necessary so this is not something new. Lime is inexpensive! The idea behind adding additional lime is to give the slurry more compressive strength something the industry solved long ago by adding cement to a Diaseal M[®] LCM slurry. See information on Diaseal M[®] LCM and cement for more detail. There is a big difference in cost between the original and expensive imitations. A recent well in South Texas took 600 sacks of the imitation for a series of squeezes and cost \$39,000.00, if Diaseal M[®] LCM had been used the retail cost would have been approximately \$20,000.00. A typical Diaseal M[®] LCM squeeze is 100 bbls and retails for 6- 7,000.00 dollars.

The price differential is considerable for basically the same material. Just add the price of 6 sacks of lime; a few sacks of fiber and you have the same thing. See Appendix XII for “Diaseal M[®] LCM and generic squeeze material” to compare.

DIASEAL M[®] LCM APPLICATIONS

1. Water based and oil based formulations
2. Open hole remedial squeeze for lost circulation
3. Open hole preventative lost circulation squeeze
4. Cased hole squeeze to seal perforations or casing leaks

Mixing Requirements:

1. Clean, isolated mixing tank or liquid mud plant
2. Cement pump truck needed if low volume pump rate required
3. Oil-wetting agent required to prevent excessive viscosity in weighted oil slurry
4. Pump through open ended pipe if additional LCM is added to slurry

**TABLE I
FOMULA FOR PREPAIRING ONE BARREL DIALSEAL M[®] LCM SLURRY
WITH FRESH OR SEA WATER**

Density lb/gal	Diaseal M[®] LCM lb	Diaseal M[®]LCM sacks	Barite sacks	Water bbl
9.0 ppg	50 ppb	1.25 sacks	0.0 sacks	0.93 bbl
10.0 ppg	50 ppb	1.25 sacks	0.6 sacks	0.89 bbl
11.0 ppg	47 ppb	1.18 sacks	1.2 sacks	0.86 bbl
12.0 ppg	42 ppb	1.05 sacks	1.8 sacks	0.82 bbl
13.0 ppg	39 ppb	0.98 sacks	2.3 sacks	0.79 bbl
14.0 ppg	35 ppb	0.88 sacks	2.9 sacks	0.76 bbl
15.0 ppg	31 ppb	0.78 sacks	3.5 sacks	0.72 bbl
16.0 ppg	28 ppb	0.70 sacks	4.0 sacks	0.69 bbl
17.0 ppg	25 ppb	0.63 sacks	4.6 sacks	0.66 bbl
18.0 ppg	22 ppb	0.55 sacks	5.2 sacks	0.62 bbl
19.0 ppg	17 ppb	0.43 sacks	5.8 sacks	0.59 bbl

Note: If saturated salt water is used, decrease barite by 0.6 sacks per barrel
 If nut plug is used, subtract 0.12 bbl of water from table for each sack used

Note: Should slurry with an inhibited filtrate be needed to prevent formation damage

Sea-water, salt water or other fluids high in concentration of electrolytes may be used in place of fresh water.

Note: If foaming occurs add a defoamer usually one 5 gallon can will treat 100 bbls.

Note: Extra dense Diaseal M[®] LCM slurries may be formulated with Hematite up to 22 ppg see Appendix II for formulations. Also see Conversion of Diaseal M[®] LCM Barite to Hematite Squeezes if barite not available in Appendix I.

TABLE II

FORMULA FOR PREPAIRING ONE BARREL DIASEAL M[®] LCM SLURRY WITH BASE OIL*

Density lb/gal	Diaseal M [®] LCM lb	Diaseal M [®] LCM sacks	Barite sacks	Oil bbl
8.0 ppg	44 lb	1.10 sack	0.38 sacks	0.880 bbl
9.0 ppg	41 lb	1.03 sack	0.88 sacks	0.855 bbl
10.0 ppg	38 lb	0.95 sack	1.38 sacks	0.830 bbl
11.0 ppg	35 lb	0.88 sack	1.88 sacks	0.805 bbl
12.0 ppg	32 lb	0.81 sack	2.38 sacks	0.770 bbl
13.0 ppg	30 lb	0.75 sack	2.90 sacks	0.745 bbl
14.0 ppg	27 lb	0.68 sack	3.43 sacks	0.720 bbl
15.0 ppg	24 lb	0.60 sack	3.97 sacks	0.695 bbl
16.0 ppg	22 lb	0.54 sack	4.52 sacks	0.670 bbl
17.0 ppg	19 lb	0.48 sack	5.08 sacks	0.645 bbl
18.0 ppg	16 lb	0.41 sack	5.65 sacks	0.610 bbl

* Due to variations in materials, pilot tests should be made to determine exact formulations. At approximately 14 ppg the Diaseal M[®] LCM – Barite- Oil Mixture will start to get very thick. At this point start adding the oil-wetting agent, which will have an instant thinning effect on the slurry. Use caution when adding wetting agent adding it in ½ to 1-gallon additions. ***Do not over treat!*** If over treatment does occur add more Diaseal M[®] LCM to the slurry to thicken it up. Observe the slurry, the objective here is to maintain sufficient viscosity for barite suspension and yet have the slurry fluid enough to pump. Remember you are using the base oil to build the slurry not the oil based drilling fluid you are drilling with. Big difference!

MIXING PROCEDURE FOR DIASEAL M[®] LCM SLURRY

1. Mix twice the open-hole volume, or minimum of 100 bbls. (16 m³) slurry for longer open hole intervals. Ideally enough slurry should be available to cover all potential loss zones, as well as to have excess volume available for squeezing operations.
2. No special equipment is needed to pump non-weighted Diaseal M[®] LCM slurries. The unweighted slurry can be mixed in a clean, uncontaminated mud pit and pumped with rig pumps. The operators should consider using a cement company blender and pump truck to place weighted Diaseal M[®] LCM slurries of 12.0 ppg. This gives better control of squeeze pressure and avoids contamination.
3. Begin with approximately 80% of the prescribed volume of fluid, (oil or water Table I or II). Add Diaseal M[®] LCM, barite, and then the remainder of fluid. Follow this with additional LCM as required or desired.
4. If building a weighted slurry, the slurry should be weighted to the same density as the drilling fluid in use. Barite or Hematite may be used.
5. Depending on conditions an additional 5-20 ppg (14-57 kg/m³) LCM in the slurry may be added.
6. Place slurry open-ended if possible. Use combinations of fine-medium LCM, and avoid too much fibrous materials. If placing through the bit, avoid excessive concentrations, and use LCM smaller than nozzle sizes to avoid plugging.

PLACEMENT AND SQUEEZE PROCEDURE FOR DIASEAL M[®] LCM

Note: If an induced vertical fracture of the formation is suspected, the most probable point is just below the casing shoe.

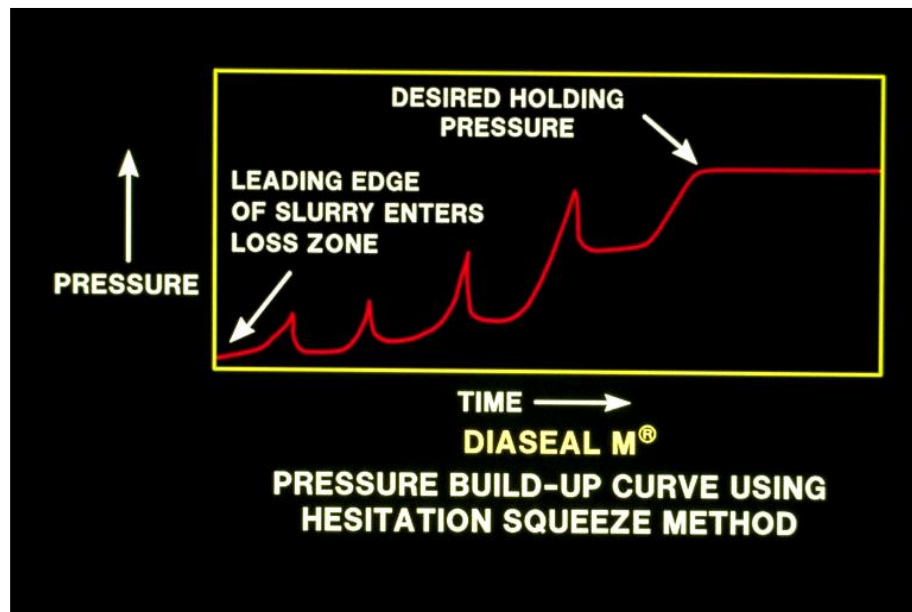
1. Place bottom of drill pipe or bit at a depth that will allow an equivalent of the calculated open-hole volume to remain inside the casing above the casing shoe. It is best to displace the slurry with the drill pipe inside casing as this will insure that the drill string will not get differentially stuck in the open hole. If slurry is to be displaced while in open hole, be sure that the drill pipe is not adjacent to an area with permeability. See Appendix (IV) for more detail
2. Pump slurry at 2-3 bbls/min (bpm) until it reaches the end of the pipe. Before beginning the hesitation squeeze, check the annulus. If no fluid can be seen, use the fill-up line and fill the hole.
3. Close annular bop, and pump slurry at 1 bpm. This will direct the Diaseal M[®] LCM slurry down hole to the point of loss. If unweighted pump the full open hole volume, plus an additional 20-30 bbls. (3-5 m³), leaving remainder of slurry in the drill pipe to squeeze with. If weighted pump the entire slurry and squeeze with drilling fluid as this prevents the weighted slurry from setting up in the pipe.
4. Shut down for 2-3 hours to allow the Diaseal M[®] LCM slurry to dewater (dehydrate). Begin pumping again at ¼ to ½ bpm. When a pressure of 50 psi is obtained, discontinue pumping for 10-15 minutes. Repeat this

procedure until 50 psi can be maintained, and then attempt progressively higher pressures in 25-50 psi increments. With this “hesitation squeeze” method, there will be a pressure bleed off each time the pump is stopped. However, with each successive squeeze, the pressure should stabilize at a higher level. See diagram of hesitation squeeze.

5. A 200-600 psi squeeze is generally considered to be very good, but it may be advisable to squeeze to a higher equivalent mud weight if it is known that a higher fluid density will be needed in this hole interval. When maximum holding pressure is obtained, shut down for 4-6 hours to make sure the slurry is dewatered. Four hours for non-weighted slurry and six hours for weighted slurry.
6. After waiting time is finished bleed the pressure from the annulus slowly, and then circulate out any remaining Diaseal M[®] LCM out of the hole.
7. Run the drill pipe back into the hole slowly, monitoring the weight indicator, and checking for bridges. Wash to bottom, drilling any Diaseal M[®] LCM plug if encountered. The Diaseal M[®] LCM will not set-up like cement, so there is little danger of sidetracking the hole. Any remaining Diaseal M[®] LCM may be retained in the drilling fluid and will act as seepage loss material.

DIAGRAM OF A HESITATION SQUEEZE

If



returns are not fully regained with the first Diaseal M[®] LCM slurry, prepare another of equal or greater size and repeat the procedure. Also see Appendix III for severe loss of return formulas of Diaseal M[®] LCM. These formulas double the amount of Diaseal M[®] LCM in the slurry and therefore double the solids content of the pill to be pumped.

TESTING THE DIASEAL M[®] LCM SLURRY PRIOR TO PUMPING

OIL BASED DRILLING FLUIDS

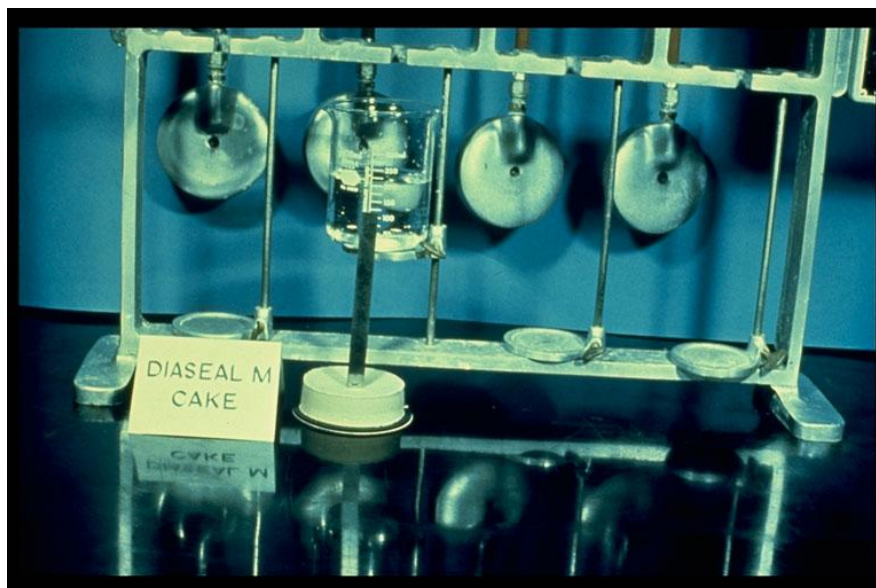
Preheat the HTHP filter press cell to 150° F. Pour slurry into cell, close cell and heat until slurry has time to reach 150° F. Apply 200 psi to cell and observe displacement of oil from slurry. There is no need to attach the lower filtrate collector on the high temp cell. The 150° F oil can be carefully caught in a glass beaker. Safety Precaution: The cell should be pressured with CO₂ for safety. Do not use nitrous oxide to pressure hydrocarbon oil fluids as this might result in an explosion! All the oil should be displaced from the slurry in about 2-3 minutes or less. This will assure one of a good, squeeze slurry that is ready to do its job down-hole. Remove filter cake from filter cell for observation of what can be expected to happen in the loss zone once the slurry is in place.

WATER BASED DRILLING FLUIDS

Testing is simple; pour a sample of slurry to be pumped into a standard API Filtrate cell and pressure up to 100 psi. Filtrate should be completely expelled in 1-3 minutes. Remove filter cake from filter cell for observation of what can be expected to happen in the loss zone once the slurry is in place. See picture of a Diaseal M[®] LCM filter cake.

Note: If the filtrate is slower than 2-3 minutes then try adding 1 ppb of lime to flocculate any clay that may be present especially when using a rig tank for mixing.

PICTURE OF A DIASEAL M[®] LCM FILTER CAKE



In this next variation cement is mixed in with the Diaseal M[®] LCM slurry to give it greater compressive strength and have good application where oil based fluids are in use.

TABLE III

DIASEAL M[®] LCM SQUEEZE MIXED WITH CEMENT

Density lbs/gal	Diaseal M [®] LCM pounds	Cement pounds	Barite pounds	Water gallons	250° F Compressive Strength PSI	150° F Compressive Strength PSI
9.5 ppg	23.40 lb	54.90 lb	0	38.53 gal	215 psi	141 psi
9.5 ppg	26.31 lb	55.91 lb TXI	0	38.05 gal	232 psi	137 psi
10.0 ppg	33.30 lb	78.30 lb	0	37.05 gal	337 psi	221 psi
10.0 ppg	37.50 lb	79.70 lb TXI	0	36.37 gal	265 psi	149 psi
11.0 ppg	53.10 lb	124.80 lb	0	34.10 gal	357 psi	317 psi
11.0 ppg	59.80 lb	127.10 lb TXI	0	33.02 gal	701 psi	338 psi
12.0 ppg	47.00 lb	175.00 lb	14.20 lb	32.16 gal	441 psi	405 psi
12.0 ppg	45.70 lb	155.30 lb TXI	40.40 lb	31.52 gal	397 psi	365 psi
13.0 ppg	20.00 lb	94.00 lb	158.73 lb	32.83 gal	371 psi	341 psi
14.0 ppg	22.00 lb	82.50 lb	222.20 lb	31.38 gal	368 psi	302 psi
15.0 ppg	16.60 lb	83.00 lb	279.50 lb	30.10 gal	334 psi	250 psi
16.0 ppg	16.80 lb	74.00 lb	342.60 lb	28.67 gal	325 psi	265 psi
17.0 ppg	13.70 lb	75.00 lb	398.00 lb	27.26 gal	328 psi	196 psi
18.0 ppg	11.50 lb	54.10 lb	472.89 lb	26.11 gal	362 psi	143 psi

Compressive Strength tests were performed at 250°F for 24 hours. If more compressive strength is required in the squeeze slurry, you should increase the amount of cement required. A service company laboratory should test all slurries before they are pumped. Cement retarders will be required at higher temperatures. At lower temperatures (125-175° F) and depending on the amount

of retarder in the slurry the compressive strength may be lower than reported. Most of these slurries will settle, so if you use a “Hesitation Squeeze” method you will need to use some type of thickening material to reduce this effect. Materials like bentonite should work well. Additional LCM may be added up to 25 ppb with little mixture adjustment. The use of a blender truck or a tank with agitation is recommended and the following procedure followed.

1. Measure water into mixing tan
2. Add Diaseal M[®] LCM while mixing and continue to agitate until pill is pumped
3. If adding a thickener add it now (usually bentonite)
4. Add Barite
5. Add any additional LCM products
6. Add retarder if required

RECOMENDATIONS FOR PUMPING 100 BBL OF DIASEAL M[®] /CEMENT SLURRY

1. Pump the slurry through open-ended drill pipe if possible, a “must do” if adding additional LCM to the slurry.
2. Option a... Spot the drill pipe 50 bbl. above the thief zone. This way when the lead 50 bbl. is displaced into the thief zone the drill pipe will be cleared of the slurry. Once the leading edge of the slurry starts to clear the open-ended drill pipe or drill bit, shut in the annulus to begin bull heading the 50 bbl. of drilling mud ahead of the slurry into the thief zone. If pressure begins to build up before you clear the drill pipe, open up the annulus and let the remaining slurry come up around the drill pipe. Then pull enough drill pipe to get above the remaining slurry. Again shut in the annulus and pressure up. You can pump +/- 3 bpm until the slurry reaches the thief zone then slow down to +/- 1 bmp.
3. Option b... Spot the drill pipe at the casing shoe and clear the drill pipe of the slurry. Pull enough stands of drill pipe to get above the slurry. Shut in the annulus and begin pumping at 3 bpm until the slurry reaches the thief zone then slow down to 1 bpm. If the hole is taking mud while displacing the slurry to the end of the drill string be sure to take that into consideration.
4. Option B would be a better option if the bit is in place.
5. Generally when using Diaseal M[®] LCM you would use the “hesitation squeeze” method, in the case of using cement with Diaseal M[®] LCM it would be best to bull head the slurry into the thief zone without doing the “hesitation squeeze”.
6. Hold pressure on the squeeze for 2 to 4 hours. Bled off the pressure on the annulus slowly.
7. Do not over displace the slurry. It would be better to under displace and leave +/- 5 bbl. in the hole. The slurry should not cause you to sidetrack as the compressive strength is much less that that of cement, however exercise caution when running the drill string back in the hole.
8. These suggestions can be adjusted to fit the actual job.

Because Diaseal M[®] LCM Cement slurries contain cement do not incorporate drilled plug material into the drilling fluid system.

If returns are not fully regained with the first Diaseal M[®] LCM slurry, prepare another of equal or greater size and repeat the procedure. Also see Appendix III for severe loss of return formulas of Diaseal M[®] LCM.

ADDITIONAL DIASEAL M[®] LCM SQUEEZE SUGGESTIONS

1. Pump Diaseal M[®] LCM Slurry open-ended if possible.
2. If slurry must be placed through bit, it is suggested to use a choke nipple, smaller than the bit nozzle size, in the surface line. This will prevent plugging of the bit, and foreign objects can be easily removed at surface to avoid disrupting operations.
3. Always test slurry, to insure high fluid loss, prior to pumping.
4. Don't hurry to build squeeze pressure. Patience and time are necessary to obtain a successful squeeze.
5. Don't mix weighted slurry too far in advance of using it as all weighted fluids are subject to barite settling with time. If weighted slurry has been mixed prematurely, mix additional Diaseal M[®] LCM to prevent long-term settling of barite.
6. Don't contaminate Diaseal M[®] LCM Slurry with low fluid loss mud.
7. Clean and flush all mixing lines to and from the cement mixing equipment to avoid contamination with any left over cement.
8. Additional LCM's maybe added to the Diaseal M[®] LCM Slurry as follows:
Open ended as much as an additional 50 ppb of CaCO₃ or mix of products maybe added if unweighted. Weighted slurries need to be pump able.
Through a bit with jet nozzles none is recommended.

ADVANTAGES

- Diaseal M[®] LCM is compatible with all drilling fluids
- The rig can mix and pump the slurry up to a 12 ppg density
- The reaction is not affected by down-hole conditions
- Additional LCM may be added to the pill

DISAVANTAGES

- Diaseal M[®] LCM must be squeezed in stages (Hesitation Squeeze) to gradually heal the loss zone
- High solids high fluid loss squeezes like Diaseal M[®] LCM are not effective in losses to carbonate formations like limestone and dolomite because this type of formation is not restricted like a fracture.

FILTRATION ACTIVATED PILLS – BARITE PLUGS

Barite intentionally settled from barite-water slurry spotted in a drilling well is called a “barite plug”. It is used mainly as a temporary plug to control gas kicks. For example, suppose you drill into a high-pressure gas zone and returns are partially lost in an upper zone before the mud weight can be raised enough to control the gas. A barite plug in the high pressure zone will shut off the gas and bring the well under control. Remedial action can then be safely taken

Barite settling due to gravitation or settling by dehydration from filtration, or a combination of both forms barite plugs. Of these methods, settling is best because you do not have to rely on filtration for a plug to form. Moreover, these heavy slurries may overcome a problem simply because of their high density. Therefore, the slurry with the highest density and fastest settling rate is best. The lower the viscosity of the slurry, the faster the settling rate and the firmer the barite plug will be. To insure low viscosity, it must perform well at the down hole temperature. The slurry should have a negative yield point (this is merely an indication of barite settling during the measurement of flow properties). A pH of 8.0-10.0 also increases settling rate and either NaOH (sodium hydroxide) or KOH (potassium hydroxide) may be used. **Fresh water must be used because barite does not settle readily in seawater or salt water!**

Enough slurry should be used to give about 450 feet (137.2 m) of fill. Longer barite plugs are not recommended because the drill pipe must be quickly pulled above the slurry before it becomes stuck from settled barite. In addition, it is undesirable to have to pull very far up the hole.

High-density slurries settle more slowly than low-density slurries due to the high solids concentrations and gel strength. Therefore, the lowest appropriate weight should be chosen.

Using SAPP, as a dispersant, is no longer recommended because it is temperature sensitive and a contaminate. Instead use 1 ppg of **Desco[®] Deflocculant**, **CF Desco Deflocculant**, **CF Desco II[®] Deflocculant** or **Drill-Thin[®] Thinner** (See Improved Barite Plug) or 6-8 ppb of Chrome Lignosulfonate (CLS)

Pilot tests of the slurry should be performed prior to running the plug. The barite in the slurry should settle at an ideal rate of ½ the volume in 10 minutes.

Avoid mixing slurry below 16 ppg for barite weight material and 20 ppg for hematite material, as rapid settling could occur and plug the equipment and lines. The maximum recommended density for barite plugs is 22 ppg. Heavier slurries may be produced by combining hematite with barite or by mixing hematite alone.

Barite Plug Mixing: Barite-water slurry is usually mixed with cement equipment pumped through the drill pipe and spotted on bottom. The drill bit jets do not have to be removed.

TABLE IV

MAKE-UP WATER AND AMOUNT OF BARITE NEEDED

Slurry Density (lb_m/gal)	Barite (sacks/bbl)	Gallons water/bbl
14.0 ppg	3.10 sacks/bbl	33.2 gallons
15.0 ppg	3.70 sacks/bbl	31.7 gallons
16.0 ppg	4.20 sacks/bbl	30.1 gallons
17.0 ppg	4.80 sacks/bbl	28.6 gallons
18.0 ppg	5.30 sacks/bbl	26.9 gallons
19.0 ppg	5.94 sacks/bbl	25.5 gallons
20.0 ppg	6.43 sacks/bbl	23.9 gallons
21.0 ppg	7.00 sacks/bbl	22.4 gallons
22.0 ppg	7.50 sacks/bbl	20.6 gallons

PREPERATION

Clean and flush all mixing lines to and from the cement mixing equipment to avoid contamination with any left over cement. Have barite on hand for the job either sack or bulk. Measure the water for plug volume taking into account line volumes and dead space of the mud tank into tank. If possible, mix NaOH or KOH and 6-8 ppb CLS into the mixing water. Mix plug and pump "on the fly". **Use mud not water to chase the barite plug to prevent premature settling!**

CALCULATE THE BARITE PLUG

BARREL OF PLUG:

$$\begin{aligned} &(\text{Length of Plug}) \times (\text{bbl/ft open hole}) = (\text{bbl of Plug}) \\ &(\text{_____ Ft}) \times (\text{_____ bbl/ft}) = (\text{_____ bbl}) \end{aligned}$$

SACKS OF BARITE TO BE USED:

$$\begin{aligned} &(\text{Bbl of plug}) \times (15 \text{ sacks barite/bbl. of vol.}) = (\text{Sacks of barite}) \\ &(\text{_____ Bbl}) \times (15 \text{ sacks barite/bbl. of vol.}) = (\text{_____ Sacks}) \end{aligned}$$

BARREL OF SLURRY: (Table IV)

$$(\text{Sacks of barite}) / (\text{Sacks barite/bbl slurry}) = (\text{bbl of slurry})$$

GALLONS OF WATER: (Table IV)

$$\begin{aligned} &(\text{Bbls of slurry}) \times (\text{Gal of water/bbl of slurry}) = (\text{Gal of Water}) \\ &(\text{_____ bbl}) \times (\text{_____ gal/bbl}) = (\text{_____ gal}) \end{aligned}$$

POUNDS OF DEFLOCCULANT (1 ppb **Desco[®] Deflocculant**, **CF Desco[®] Deflocculant**, **CF Desco II[®] Deflocculant** or **Drill-Thin[®] Thinner** or CLS at 6-8 ppg)

$$\begin{aligned} &(\text{Bbl of slurry}) \times (\text{Lbs of deflocculant}) = (\text{Lbs of Desco[®] Deflocculant or CLS}) \\ &(\text{_____ Bbl}) \times (\text{_____ lb/bbl}) = (\text{_____ lb}) \end{aligned}$$

POUNDS OF KOH OR NaOH: (1-1.5 ppb recommended)

$$\begin{aligned} &(\text{Bbl of slurry}) \times (\text{Lb of KOH or NaOH/bbl of slurry}) = (\text{Lb of KOH or NaOH}) \\ &(\text{_____ Bbl}) \times (\text{_____ lb/bbl.}) = (\text{_____ lb}) \end{aligned}$$

THE IMPROVED BARITE PLUG

Barite plugs weighting 18-22 ppg may be prepared using barite, fresh water and tannin deflocculants. Tannin base thinners (**Desco[®] Deflocculant**, **CF Desco[®] Deflocculant**, **CF Desco II[®] Deflocculant** and **Drill-Thin[®] Thinner**) are highly effective at all temperatures and they are available in all drilling areas. Tannin deflocculants perform well at the natural pH of the barite slurry. Therefore, the use of sodium hydroxide or potassium hydroxide to increase slurry pH and settling rate is unnecessary. This simplifies the recipe and mixing.

MIXING AND PLACING THE SLURRY

Mix the barite in fresh water treated with one ppb (2.85 kg/m³) tannin deflocculant. It is best if pneumatic bulk mixers (cement mixers) are used so the slurry can be mixed and displaced continuously. Displace entire slurry plus 2 barrels (0.3m³), (from the drill pipe (DP) – annulus equalization point) with active drilling fluid. This insures against premature settling, as the slurry will still be moving after the pump is stopped. Barite settling is hindered while the slurry is in motion. Pull the drill pipe above the top of the slurry and begin circulation with mud as soon as possible. Circulate bottom if possible. Since the slurry contains

50% by volume barite, a firm barite plug will be about one-half of the hole fill-up volume of the slurry.

TABLE V

**MATERIALS NEEDED TO MAKE ONE BARREL OF 22 PPF SLURRY
ENGLISH UNITS**

**Water – 0.5 bbl Desco® Deflocculant, CF Desco® Deflocculant CF Desco II®
Deflocculant or Drill-Thin® Thinner – 0.5lb., Barite –750 lb**

Fill-Up data for Various Hole Sizes	12-1/4”	8-3/4”	7-7/8”	6-1/4”
Fill-Up, feet per one barrel of Slurry	6.9 fpb	13.4 fpb	16.6 fpb	26.4 fpb
Barrels of Slurry Needed for 450 ft Hole Fill-Up	66 bbl	33 bbl	27 bbl	17 bbl

TABLE VI

**MATERIALS NEEDED TO MAKE ONE CUBIC METER (m³) OF 2.6 kg/dm³
SLURRY (SI) UNITS**

**Water – 0.5m³ Desco® Deflocculant, CF Desco® Deflocculant, CF Desco II®
Deflocculant or Drill-Thin® Thinner – 1.43 kg Barite 2140 kg**

Fill-Up Data for Various Hole Sizes	31.1cm	22.2cm	20.0 cm	15.9 cm
Fill-Up, m per m³ of Slurry	13.2 m/m³	25.7 m/m³	31.4 m/m³	50.6 m/m³
Cubic Meters (m³) of Slurry Needed for 137.2 m Hole Fill-Up	10.5 m³	5.2 m³	4.3 m³	2.7 m³

HYDRATION PILLS

Description: mixing a high concentration of bentonite into diesel (BDO), mineral (BMO) or other synthetic oil (BSO) forms a Hydration pill. These pills form a very firm but plastic LCM material when mixed with water or water based drilling fluids. These slurries do not develop high compressive strength, but remain plastic. The final strength of BDO is determined by the ratio of BDO to drilling fluid. The starting ratio of the drilling fluid to BDO is usually 8:1 to 4:1 as this concentration produces a softer plug that can be more easily squeezed into the loss zone. As the ratio of drilling fluid to BDO decreases to 3:1 progressively firmer plugs are formed. The 4:1 ratio will be a highly viscous fluid and the 1:3 ratio forms a stiff, plastic grainy solid.

Applications: Hydration pills are intended for situations where conventional lost circulation pills have failed. In addition to lost circulation applications, BDO has been used to shut off down-hole flows, such as from a high-pressure zone to a lower pressure zone. BDO pills are unaffected by down-hole temperature and may be used in fresh water or salt water mud systems.

Mixing Requirements: Two pumping units are used for a BDO squeeze. One pumping unit will pump the BDO down the drill string and the second unit will pump drilling fluid down the annulus so the mixing rate can be changed as the plug is placed. Alternately one pumping unit can be used to pump the pill down the pipe and the rig pump can be used to pump drilling fluid down the annulus. While the BDO pill can be successfully mixed and placed using the rig's mixing and pumping system, better control of the operation and reduced risk of contamination and plugged piping is obtained from two pumping units.

Flush all pumps, mixers, lines and tanks that will be used to prepare the slurry. Avoid contamination of the slurry with drilling fluid or water in the suction lines and pumps as this will cause the slurry to gel and plug lines.

THE BENTONITE-DIESEL OIL (BDO) OR BENTONITE-SYNTHETIC OIL (BSO) SLURRY AND SQUEEZE IN

WATER BASED DRILLING FLUIDS

Formula:

1. 300-400 pounds bentonite
2. 1 bbl of diesel oil, mineral oil or synthetic oil (final volume 1.42 bbl)
3. Lost circulation material optional at 10-12 ppb.
4. Barite as needed for density

Squeeze Procedure:

1. Locate loss zone and run in hole open ended. Ideally, the loss zone should be known so the BDO can be placed efficiently where needed. If hole conditions allow place the pipe just above the loss zone, if not place the end of the pipe inside the casing.
2. Determine the volume of slurry to pump. Typically, a 40 barrel pill is used. It is advisable to mix in 10 bbl increments as to provide easier clean up of the pumping equipment.
3. Pump a 5-10 bbl base oil spacer ahead and behind the slurry.
4. Pump pill to bottom of pipe and follow spacer with drilling fluid.
5. Close the blow out preventers.
6. Pump the slurry out the drill string at 1-2 bbl/minute and pump drilling fluid into annulus at 4-8 bbl/min for a thinner initial slurry pump at 8-16 bbl per minute down the annulus.
7. When one half the slurry from drill string is displaced; reduce pump rates to 1 bbl/min on the drill pipe and 1-3 bbl/min on the annulus.
8. When desired pressure is reached, maintain pressure on casing. Displace the drill string volume with drilling fluid and pull into casing.
9. Maintain pressure on the well for approximately 3 hours.
10. Circulate and condition the drilling fluid in the casing, and wash and ream the open hole slowly to bottom. This avoids pushing the bit into the BDO-slurry and possibly pressuring up the hole causing lost circulation to reoccur.

Note: As the BDO pill is circulated to the surface the shale shaker may blind and should be closely watched to prevent mud losses across the screens. **BDO pills are not incorporated into the drilling fluid and are not saved for future use! Do not attempt to reverse out unused BDO, as the pipe is likely to become plugged!**

THE BENTONITE-CEMENT-DIESEL OIL SQUEEZE (BCDO) IS A REACTIVE PILL FORMULATED WITH CEMENT TO PROVIDE SOME COMPRESIVE STRENGHT

Formula for 50 bbl of slurry is:

1. 77 sacks of (100 lb/sack) cement
2. 77 sacks of (100 lb/sack) bentonite
3. 36 bbl diesel oil or base oil
4. Barite as needed for density

Squeeze Procedure:

Same as above, for greater compressive strength allow hardening for 24 hours.

REVERSE SQUEEZE FOR USE IN OIL BASE DRILLING FLUIDS

Description: mixing a high concentration of organophillic bentonite into water forms a Hydration pill when mixed down hole with oil-based drilling fluids. Organophillic bentonite is used to increase the viscosity of oil based drilling fluids. The organophillic bentonite is added at a very high concentration to the water as it reacts as a relatively inert solid and does not hydrate until it contacts oil. When the reverse slurry is mixed with the oil based drilling fluid in the hole, the organophillic bentonite reacts and becomes an active solid. Because the concentration of organophillic bentonite is very high, a stiff plastic mass results that can effectively seal a loss zone.

Applications: Hydration pills are intended for situations where conventional lost circulation pills have failed. In addition to lost circulation applications, the reverse slurry has been used to shut off down-hole flows, such as from a high-pressure zone to a lower pressure zone. Reverse slurries are unaffected by down-hole temperature and may be used in fresh water or salt water mud systems.

Mixing Requirements: Two pumping units are used for a reverse squeeze. One pumping unit will pump the slurry down the drill string and the second unit will pump drilling fluid down the annulus so the mixing rate can be changed as the plug is placed. Alternately one pumping unit can be used to pump the pill down the pipe and the rig pump can be used to pump drilling fluid down the annulus. While the reverse pill can be successfully mixed and placed using the rig's mixing and pumping system, better control of the operation and reduced risk of contamination and plugged piping is obtained from two pumping units. The initial ratio of the reverse slurry to the drilling fluid is often 8 parts mud to 1 part slurry. This mixture produces a softer plug that can subsequently be squeezed into the loss zone. After pumping $\frac{1}{2}$ the reverse slurry the ratio should change to 3 parts drilling fluid to 1 part slurry to produce a much thicker squeeze. Pump this thicker squeeze at $\frac{1}{2}$ the initial rate. Move pipe periodically to insure it remains free. If the hole stands full pump 1 bbl down the drill pipe and $\frac{1}{2}$ bbl down the annulus to develop pressure. Monitor annular pressure gauge and control pressure to a maximum 1.5 ppg mud weight.

Flush all pumps, mixers, lines and tanks that will be used to prepare the reverse slurry with water... Avoid contamination of the slurry with oil based drilling fluid or oil in the suction lines and pumps as this will cause the slurry to gel and plug lines.

**THE REVERSE FORMULA FOR USE IN OIL BASED DRILLING FLUIDS
FORMULATION FOR 1 BBL**

Material	Density	Density	Density	Density
	10.5 ppg	13.0 ppg	16.0 ppg	18.0 ppg
Water bbl	0.660 bbl	0.628 bbl	0.582 bbl	0.540 bbl
CLS	3.5 ppb	3.5 ppb	3.5 ppb	3.5 ppb
NaOH	1.5 ppb	1.5 ppb	1.5 ppb	1.5 ppb
Organophillic Bentonite	220 ppb	150 ppb	100 ppb	100 ppb
Barite	None	175 ppb	370 ppb	548 ppb

PUMPING PROCEDURE

1. Locate loss zone and run in hole open ended. Ideally, the loss zone should be known so the reverse slurry can be placed efficiently where needed. If hole conditions allow place the pipe just above the loss zone, if not place the end of the pipe inside the casing.
2. Determine the volume of slurry to pump. Typically, a 40 barrel pill is used. It is advisable to mix in 10 bbl increments as to provide easier clean up of the pumping equipment.
3. Pump a 5-10 bbl water spacer ahead and behind the reverse formula slurry
4. Pump pill to bottom of pipe and follow spacer with drilling fluid
5. Close the blow out preventers.
6. Pump the slurry out the drill string at 1-2 bbl/minute and pump drilling fluid into annulus at 4-8 bbl/min. For a initial thinner slurry pump 8-16 bbl drilling fluid down the annulus. This allows the leading edge of the plug to enter the loss zone easier than a thicker one.
7. When one half the slurry from the drill string is displaced reduce pump rates to 1 bbl/min on the drill pipe and 1-3 bbl/min on the annulus. This produces a firmer plug.
8. When desired pressure is reached, maintain pressure on casing. Displace the drill string volume with drilling fluid and pull into casing.
9. Maintain pressure on the well for approximately 3 hours.
10. Circulate and condition the drilling fluid in the casing, and wash and ream the open hole slowly to bottom. This avoids pushing the bit into the BDO-slurry and possibly pressuring up the hole causing lost circulation to reoccur.

Note: As the reverse pill is circulated to the surface the shale shaker may blind and should be closely watched to prevent mud losses across the screens. **BDO pills are not incorporated into the drilling fluid and are not saved for future use! Do not attempt to reverse out unused BDO, as the pipe is likely to become plugged!**

ADVANTAGES/DISADVANTAGES

The advantage of the BDO over BCDO plugs is that the BDO plug remains plastic and does not develop compressive strength. BDO pills have a proven record of solving difficult loss circulation problems and BDO pills are prepared from commonly available materials. Success depends upon achieving the right mixture of drilling fluid and BDO in the hole to seal the loss zone. If too thin the plug will not form, if too thick the plug may not seal the fracture and circulation will be lost again when the plug is drilled. Best results require careful mixing and placement with a cement pumping unit to move both the BDO and the drilling fluid.

The use of diesel or mineral oil may require that all drilling fluid and cuttings be handled in a manner that complies with environmental regulations. Synthetic oil may be acceptable after pilot tests are run.

When a either a BDO or reverse squeeze is circulated out the shale shakers may blind and should be closely watched to prevent drilling fluid losses across the screens. Incorporation of any of the spacers and pill into the drilling fluid system will require treatment to avoid adversely affecting the drilling fluids properties.

If a reverse BDO squeeze is not successful, it may be necessary to displace an oil based fluid with a water based fluid to drill the troubled-hole section or pump cement.

There are many variations of the BDO/BSO squeeze and the largest collections of these can be found in "Lost Circulation" by Joseph U. Messenger Copyright 1981 PennWell Books.

CHEMICAL REACTIVE PILLS TYPES

POLYMER PILLS

When total loss of returns is attributed to large vugular and cavernous formations a polymer plug may be spotted and allowed to hydrate and expand within the formation with time and temperature. Generally consisting of blends of loss circulation materials and a hydrateable cross-linked polymer that is spotted across the zone of interest and then squeezed at 150-200 psi. Polymer plugs are either surface activated as they are mixed and then chemically activated or cross-linked as pumping down hole begins or down hole activated when the pill contacts mud or formation fluid forming a plug. Both surface activated and down hole activated pills react very much like BDO pills when contacting water.

SURFACE ACTIVATED POLYMER PILLS

These pills are usually prepared in water, rather than mud and are especially effective at shallow depths to seal large vugular or cavernous openings. No special equipment is required as they can be mixed and pumped using rig equipment. Surface activated polymer pills may be used in water or oil based muds where a clean mixing tank can be employed. The success rate on carefully prepared and planned jobs is high. Bottom hole temperature must be below 175°F for natural polymers such as guar gum as the polymer will rapidly break down above this temperature.

Advantages

- The surface activated polymer pills (such as Guar Gum) can be mixed and placed with the rig equipment.
- The success rate on carefully prepared and planned jobs is high.
- The reactive type polymer pills are especially suitable to isolate and remote drilling locations using a helicopter rig because the materials require little helicopter time.

Disadvantages

- Bottom hole temperatures must be below 175 degrees F for the natural polymers as the polymer rapidly breaks down above this temperature.
- Generally need to be pumped immediately after mixing!

DOWN HOLE ACTIVATED POLYMER PILLS

These pills are high concentrations of polymer typically 300 to 350 ppb suspended in an inert carrier such as diesel or mineral oil. These polymer plugs form hard very plastic plugs when the pill contacts the mud or formation fluid and some are used to control water flow on producing wells. Mixing a pill in diesel or mineral oil requires two cement-pumping units to mix the pill and move mud on both the drill pipe and annulus or one cement unit to place the pill and the rig

pump to mix the mud with the pill down hole. Down hole activated polymer pills can be selected to work in various brines.

Note: Most Polymer Pills are proprietary and the Service Company involved needs to provide guidance on how to apply them correctly.

Preparation

Pilot test the planned mix before proceeding to mixing the pill. Fill the mixing tank with water. If the pill is to be weighed with barite, pilot testing and very careful adherence to the formulation provided from the supplier is required.

Placement

If hole conditions allow and the loss zone depth is known, place the pipe just above the loss zone, otherwise, place the end of the pipe inside the casing. Also, the hole should be open to TD and not be bridged over to allow the pill to seal all the zone taking fluid.

Pump 3-5 barrels of spacer followed by the pill and another 3-5 barrels of spacer to isolate the polymer pill from the drilling fluid, followed by enough drilling fluid to place the bottom of the pill at the bottom of the pipe.

Close the blow out preventers to allow annular injection down the string.

The planned pumping program should be determined by the site specific objectives and conditions. These factors can alter the following placement procedure.

Commence pumping down the drill string at a maximum rate of 2-3 barrels per minute and down the annulus at a slower rate. The polymer will react very fast with the drilling fluid.

Monitor annular pressure gauge reading and control the pressure to a maximum equivalent of 1.5 ppg mud weight.

After the desired pressure is reached, maintain pressure on the casing. Displace the drill string volume with drilling fluid. Pull into the casing.

Maintain pressure on the well for approximately 3 hours to allow the plug to seal the loss zone. Circulate and condition the drilling fluid at the bottom of the casing for at least two annular mud volumes while carefully monitoring for drilling fluid loss before tripping the pipe.

Circulate and condition the drilling fluid at the casing shoe before entering the open hole. Wash and ream the open hole slowly to avoid pushing the bit into the reacted pill and possibly pressuring up the hole or re-initiating lost circulation.

Advantages

- The down hole activated polymer pills can be formulated to form very hard plastic plugs. Some of these pills (sodium silicate) are also used in water flow control operations on producing wells.

Disadvantages

- Bottom hole temperatures limits of 175 degrees F or below. Many of the natural polymers break down rapidly above this temperature.
- Requires two cement pump trucks to mix and place the pill properly.

CEMENT

The use of Portland cement to plug inside a well bore or cased hole is a remedial cementing procedure sometimes used in drilling a well to regain circulation. Portland Cement is a practical and economical option for combating loss of circulation. The types of cements slurries used for this purpose are neat cement or blends, and light density cements made with either bentonite or diatomaceous earth. Neat Portland cement provides for a higher density non weighted slurry. This slurry develops high compressive strength when it sets. Blends may include Portland cement plus additives such as accelerators, retarders, or light weight additives. Some commonly used light weight additives are bentonite and diatomaceous earth.

Bentonite: Bentonite in cement allows lower density pills (12-15.6 ppg) to be built. Prehydrate part of the bentonite in water as this lowers the density over adding all the bentonite dry. The water used to prehydrate the bentonite is treated with ¼ ppb sodium hydroxide and ¼ ppb of soda ash to remove hardness before the dry cement and the remaining dry bentonite are added. Attapulgate may be use as a substitute for bentonite in fresh or sea water. Preshearing attapulgate in either fresh, sea water or brines will improve the yields and the cement slurry properties.

Aggregates: Adding Gilsonite in concentrations of 25-100 lb/bbl or Frac Sand 10-20 ppb, can be added to decrease the density of the slurry and act as a bridging agent. These materials are mixed with other additives to prevent the sand from settling out or the Gilsonite from floating out and are seldom used alone for lost circulation.

Diacel D[®] Additive (Diatomaceous Earth) cements (10.5-15.6 ppg) may be built by adding various amounts of diatomaceous earth to Neat Portland cement. See Appendix V for formulations.

THE BALANCED PLUG

The term “balancing a plug” refers to spotting a volume of material in the well bore in a way that the heights of the fluid columns on either side of pipe are balanced. This balance is necessary to assure that the plug stays in place once the drill pipe or tubing is removed. If the plug is not balanced the plug could move, either up or down, and become contaminated.

Balancing the plug is easier if the specific gravity of the plug is the same as the fluid in the well bore and annular space. Engineering comes into play when the plug is either heavier or lighter than the fluid in the well bore or the plug is being placed up hole. If the plug is heavier than the fluid in the well bore, such as cement, then a lighter fluid will be needed to balance the fluid columns and if the plug is lighter than the fluid in the well bore, then a weighted pill will be needed to be used to balance the system. When a cement plug is set the plug and the spacer both have a higher density than the drilling mud. The term balance in this case refers to the slurry and spacer column heights both inside and outside of the pipe, i.e. they are equal.)

In most instances a balanced plug is a plug made out of cement. The viscosity difference between the cement plug and the drilling fluid that has been left in the hole is enough to prevent the cement plug from mixing with the drilling fluid. If the drilling fluid has a significantly lower viscosity than the cement plug then a high viscous bentonite pill can be pumped ahead of the plug to hold the plug in place. If a bentonite pill is pumped, then its volume needs to be added to the calculations.

Calculations used in balancing a plug:

1- Determine the volume of plug needed to cover the zone of interest in either bbls per foot or cubic feet per foot.

Volume to cover a specific length of hole= length (height) of plug x hole volume (bbl/ ft or ft³/ft) $V = H \times V_H$

2- Determine the amount of spacer needed in the tubing or drill string to balance the spacer placed in the annulus a head of the plug

Tubing Spacer Volume

$$TSV = V_s \times C_t / A$$

3- Calculate the height of the balanced plug column before pulling the tubing or drill pipe

Height (thickness) of the balanced plug before pulling tubing/drill pipe

$$H = V / (A + C_t)$$

4- Calculate the amount of mud needed to balance the plug column before pulling the tubing or drill pipe

$$V_{MD} = TL \cdot H \cdot C_t - TSV$$

Nomenclature:

TL= tubing or drill pipe length, ft

V_{MD} = Mud displacement volume, ft³ or bbls

V_s = spacer volume, ft³ or bbls

TSV= Tubing Spacer Volume, ft³ or bbls

V_H = open hole volume, ft³ or bbls

V= volume of plug used, ft³ or bbls

H= height of balanced cement column, ft

A= annular volume between tubing or drill string and open hole or casing, ft³/ft or bbl/ft

C_t = capacity of the tubing or drill string, ft³/ft or bbl/ft

Wt= weight of tubing or drill string, lb/ft

Example Problem:

Balance a 10 barrel (bbl) plug in a 8.5" open hole using 4.5" drill pipe (16.6 lbs/f) set so the bottom of the plug is at 8500'. A 5 bbl water spacer will be run ahead of the plug.

Data:

Open Hole Volume = 0.0702 bbls/ft or 0.3941 ft³/ft

Annular volume = 0.0505 bbls/ft or 0.2836 ft³/ft for 4.5 "DP in 8.5" open hole

Drill pipe volume= 0.0142 bbl/ft or 0.07984 ft³/ft

1- Determine the amount of spacer needed in the tubing or drill pipe to balance the 5 bbl spacer run ahead of the plug.

Ratio of tubular volume to annular volume= $(0.0142 \text{ ft}^3/\text{ft}) / (0.0505 \text{ ft}^3/\text{ft}) = 0.281$

The volume of spacer needed to be in the tubing = $5 \times 0.281 = 1.41 \text{ bbls}$

2- Determine the height (thickness) of the plug left in the hole

$$10 \text{ bbls} = 5.61 \text{ ft}^3/\text{bbl} \times 10 = 56.1 \text{ ft}^3$$

$$H = V / (A + C_t)$$

$$= 56.1 \text{ ft}^3 / (0.2836 \text{ ft}^3/\text{ft} + 0.07984 \text{ ft}^3/\text{ft})$$

$$= 154.36 \text{ ft.}$$

3- Determine the depth to the top of the plug

Data:

Bottom of the plug is set at 8500'

Plug Height=154.4'

Depth to the top of the plug = $8500' - 154.4' = 8345.6'$

4- Determine the amount of mud needed to balance the plug

Data:

Mud needed to balance the plug = total depth of the bottom of the plug – (height of the plug + height of the spacer in the drill pipe) x drill pipe volume (bbl/ft)

Data:

Depth= 8500'

Height of the plug = 154.4'

Height of Spacer in the drill pipe = $\text{bbls} / \text{drill pipe capacity (bbl/ft)} = 1.41 \text{ bbl} / .0142 \text{ bbl/ft} = 99.3'$

Mud needed to balance the plug = $(8500' - 154.4' - 99.3') \times 0.0142 \text{ bbl/ft} = 117.1 \text{ bbl}$

DRILLING BLIND OR WITH AERATED FLUID AND SET PIPE

In very severe complete losses drilling through caverns and other large natural horizontal fractures drilling blind (with out returns), drilling with aerated fluid, or stiff foam can be used to cross the loss zone and then set pipe. Drilling with water or brine and sweeping the hole with high viscosity pills to push cuttings back into the loss zone may be required.

See Appendix X; Mist Drilling with Drispac[®] Polymer.

LOST CIRCULATION IN PRODUCTION ZONES

Industry studies indicate that fibrous LCM's can cause formation damage to productive zones. Core tests showed fluid invasion to the extent that fibers passed completely through the cores on return permeability tests. In tight sands, which will be fractured, this may not be of concern; **other wise use of fibrous LCM's in productive zones is not recommended!**

Typical pills are composed of 15-20 bbl of completion fluid brine and 75 lb/bbl of calcium carbonate in equal amounts of fine, medium and coarse. Repeat as needed. For total loss of returns this may increase to 30-40 bbl of completion fluid brine and 200 lb/bbl of calcium carbonate in equal amounts of fine, medium and coarse. Most brine used for this is 2-3% potassium chloride (KCl).

APPENDIX CONTENTS

- Diaseal M[®] LCM weighted with Hematite formulas (I)
- Extra Dense Diaseal M[®] LCM formulations (II)
- Alternate Diaseal M[®] LCM formulations for Severe Applications (III)
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- Diaceal D[®] (Diatomaceous Earth) cements for loss circulation formulations (V)
- Log of a Lost Circulation Zone (VI)
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- Mist Drilling with Drispac[®] Polymer (X)
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APPENDIX I

DIASEAL M[®] LCM WEIGHTED WITH HEMATITE

Density lb/gal	Diaseal M[®] LCM lb	Hematite ppb	Water in Bbl.
9.0 ppg	50 ppb	0 ppb	0.930 bbl
10.0 ppg	50 ppb	60 ppb	0.897 bbl
11.0 ppg	50 ppb	120 ppb	0.875 bbl
12.0 ppg	50 ppb	180 ppb	0.842 bbl
13.0 ppg	47 ppb	230 ppb	0.818 bbl
14.0 ppg	42 ppb	290 ppb	0.795 bbl
15.0 ppg	37 ppb	350 ppb	0.762 bbl
16.0 ppg	34 ppb	400 ppb	0.738 bbl
17.0 ppg	30 ppb	460 ppb	0.716 bbl
18.0 ppg	26 ppb	520 ppb	0.683 bbl
19.0 ppg	21 ppb	580 ppb	0.660 bbl

Corrections to the volume of water were made to compensate for the density differential of barite and hematite.

Corrections in the Diaseal M[®] LCM quantity are estimated based on the difference in the amounts of solids in the heavier systems. Verification in the field should be made to ensure that there is no settling. Adequate mixing is also mandatory.

The revised Diaseal M[®] LCM amounts are estimates only at this time and need to be verified in the laboratory.

APPENDIX II

EXTRA DENSE DIASEAL M[®] LCM FORMULATIONS

FORMULA FOR PREPARING ONE BARREL DIASEAL M[®] LCM SLURRY WITH FRESH WATER

Density lb/gal	Diaséal M [®] LCM lb	Hematite ppb	Water in Bbl.
20 ppg	15.8 ppb	602 ppb	0.645 bbls.
21 ppg	12.4 ppb	665 ppb	0.611 bbls.
22 ppg	10.0 ppb	725 ppb	0.583 bbls.

MIXING ORDER: WATER, DIASEAL M[®] LCM AND HEMATITE

FORMULA FOR PREPARING ONE BARREL DIASEAL M[®] LCM SLURRY WITH DIESEL OIL

Density lb/gal	Diaséal M [®] LCM lb	Hematite ppb	Diesel Oil bbls.
20 ppg	10.0 ppb	672 ppb	0.620 bbls
21 ppg	10.0 ppb	730 ppb	0.590 bbls
22 ppg	10.0 ppb	770 ppb	0.560 bbls

Mixing instructions: Mix diesel and surfactant together for about five minutes and then add hematite, mix for ten minutes, add Diaséal M[®] LCM, mix and add more surfactant and stir for ten minutes. A total of 30 ml. of surfactant was used, 20 ml. initially and 10 ml. after the addition of Diaséal M[®] LCM. The surfactant is required to oil wet the materials other wise the mix is too thick. The surfactant used was an ethoxylated alcohol. It is suggested that individual oil mud wetting agents be pilot tested first to make sure of the correct concentrations needed. Testing was done in the API filtrate cell without using a filter paper only the screen. There is not a dramatic rush as with the water base formulations only a slow steady stream. The 22 ppg slurry is a maybe, we were able to drive the oil out of the mix but it was slower than the 20 ppg and 21 ppg formulations.

Note: also be sure when doing the 22 ppg slurry to keep your finger over the discharge port on the bottom of the API test cell because the slurry will start coming out as soon as you pour the slurry into the API filter press. All test pressures were 100 psi.

APPENDIX III

ALTERNATE DIASEAL M[®] LCM FORMULA FOR SEVERE APPLICATIONS

Subject: Testing 12, 13 and 14 ppb Diaseal M[®] LCM Formulations

Three different weighted slurries (12, 13, and 14 ppg) were tested for rheological properties. The formulation for the slurries was made based on doubling the recommended amount of Diaseal M[®] LCM used in the slurry and adding 1 ppb of CF Desco[®] II Deflocculant.

Test Procedures:

The three different slurries were mixed on a multimixer and then tested for rheological properties.

After the slurries were tested, 1 ppb equivalent of CF Desco[®] II Deflocculant was added and they were stirred for an additional 5 minutes on the multimixer. The slurries were then retested for rheological properties. The results are in table I.

Formulations:

12 ppg: 84 g of Diaseal M[®] LCM, 273.5 ml of tap water, and 150.2 g of barite

13 ppg: 78 g of Diaseal M[®] LCM, 262.2 ml of tap water, and 210.6 g of barite

14 ppg: 70 g of Diaseal M[®] LCM, 251.7 ml of tap water, and 271.9 g of barite

TEST RESULTS

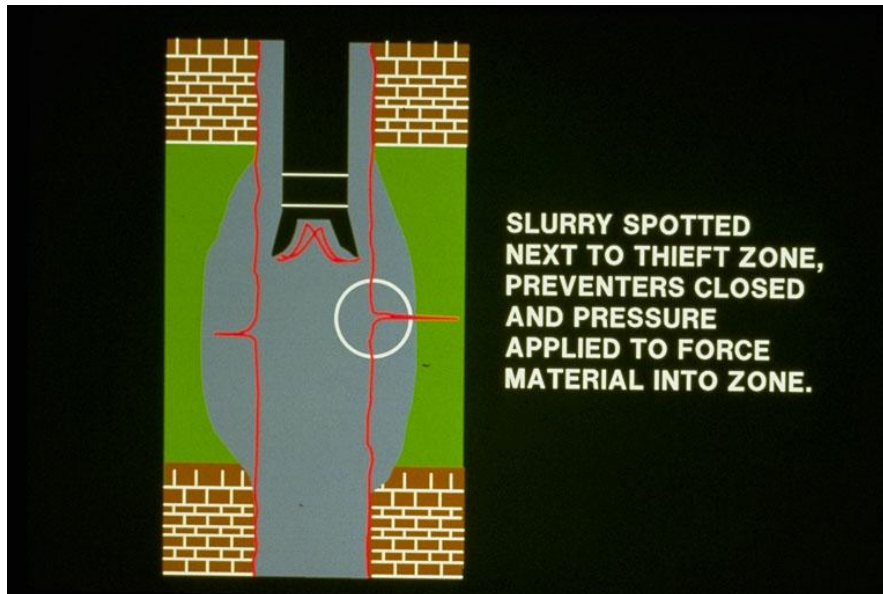
TABLE I

Slurry	600	300	200	100	6	3
12 ppg	160	100	70	50	30	25
13 ppg	165	110	90	63	35	26
14 ppg	180	125	95	60	30	20
	1 ppb of CF Desco II[®] Deflocculant Added					
12 ppg	120	95	70	45	13	10
13 ppg	123	95	75	50	20	15
14 ppg	110	90	68	50	13	13

APPENDIX IV

ALTERNATE DIASEAL M[®] LCM PLACEMENT PROCEDURE

Decide what the maximum pressure will be during this procedure. A 200-600psi holding pressure is generally considered to be very good, but it may be advisable to squeeze to a higher equivalent mud weight if it is known that a higher fluid density will be needed in this hole. Pull off bottom or just above loss zone, be sure that the drill pipe is not adjacent to an area with permeability. See picture below



Pump slurry at 2 bpm until it reaches the end of the pipe. Before beginning the hesitation squeeze, check the annulus. If no fluid can be seen, use the fill-up line and fill the hole. Close annular bop, and pump at 1 ppm. This will direct the Diaseal M LCM slurry down hole to the point of loss. If unweighted pump the full open hole volume, plus an additional 20-30 bbls (3-5 M3) leaving the remainder of slurry in the drill pipe to squeeze with. If weighted pump the entire slurry volume and squeeze with the drilling fluid as this will prevent the weighted slurry from setting up in the pipe.

After spotting the slurry pull, the pipe into the casing and continue the squeeze procedure. Shut down for 2-3 hours to allow the Diaseal M[®] LCM slurry to dewater. Remember that Diaseal[®] M LCM is a high fluid loss, high solids slurry. If there is any permeability a thick filter cake can build up on the face of the well bore and stick the pipe or the drill bit. This will not occur in a thief zone where the slurry is bull-headed into the formation.

After allowing the Diaseal M[®] LCM slurry to dehydrate check to see if the hole stands full. If the hole stands full then close pipe rams and pump through fill up line at a 1/4 to 1/2 bpm rate until 50 psi is obtained; then discontinue pumping for 10-15 minutes. Repeat this procedure with the goal to obtain the

desired pressure. The hesitation squeeze allows the loss zone the best opportunity to become sealed-off. The final squeeze pressure should not exceed the equivalent maximum mud weight hydrostatic pressure for the interval. Once the desired pressure is obtained, shut down for 2-4 hours and wait for any additional fluid left in the slurry to bleed off. Two hours for a non-weighted slurry and 4 hours for weighted slurry.

Slowly bleed the pressure from the annulus. Circulate and condition the mud, before tripping back into the hole. Carefully wash and ream back to bottom while monitoring holes conditions and signs of mud loss. Wash to bottom, drilling any Diaseal[®] M LCM plug if encountered. The Diaseal[®] M LCM plug will not set up like cement, so there is minimal danger of sidetracking the hole as with cement. Any remaining Diaseal[®] M LCM plug material may be retained in the drilling fluid and will act as seepage loss material.

ON LOCATION SOLVING ANOTHER PROBLEM



APPENDIX V

DIACEL D® ADDITIVE CEMENTS FOR LOSS CIRCULATION

TABLE I

40% Diacel D® Additive Cement System Slurry Density and Volume Relationships

Slurry Density Lb/gal	Slurry Density Lb/cu feet	Slurry/sack cement	Water percent	Water gal/sack cement	Sacks cement per cubic foot of slurry	Sacks Diacel D® Per cubic foot of slurry
** 10.8	80.8	4.52	249	28.1	0.22	0.17
10.9	81.5	4.34	237	26.8	0.23	0.17
* 11.0	82.4	4.19	226	25.6	0.24	0.18
* 11.1	83.0	4.03	216	24.5	0.25	0.19
11.2	83.8	3.89	207	23.4	0.26	0.19
11.3	84.5	3.76	199	22.4	0.27	0.20

* Recommended slurry density for most jobs using common Portland cement

** Recommended slurry density for most jobs using Incor SR cement

TABLE II

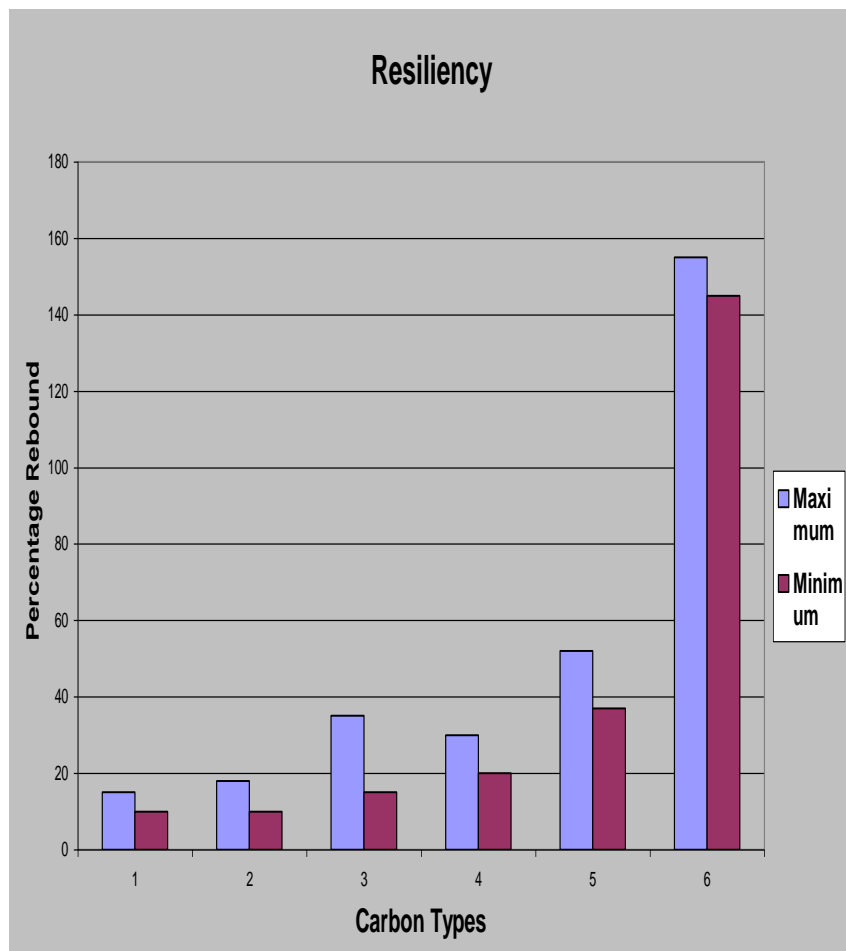
40% Diacel D® Additive Cement System Thickening Time and Compressive Strength Prepared with Class A Common Portland.

Slurry Density		CaCl ₂ %	Compressive strength, psi when cured at atmospheric pressure				API thickening Time		
Lb/gal	Lb/cubic foot		1 Day		3 Days		Hours@ 10,000'		
			80° F	100° F	120° F	140° F	80° F	120° F	230° F
11.1	83.0	0	25	55	110	370	70	610	7.00+
11.1	83.0	4	45	130	300	600	145	870	7.13
11.3	84.5	0	30	85	130	580	80	750	3.22
11.3	84.5	4	70	190	520	600+	200	1220	2.02

APPENDIX VII

REBOUND™ LCM – RESILIENT GRAPHITIC CARBON

Carbon Types:
1-Crystalline Vein
2-Flake
3-Amorphous
4-Petroleum Coke
5-Synthetic
6-Rebound™ LCM



APPEXDIX VIII

THE DYNARED™ FIBER LCM PILL

Material needed per barrel of Pill built:

DynaRed™ Fiber medium or coarse: 50 – 60 lb. per bbl.

Flowzan® Biopolymer: 0.5 gal/bbl or 2 lb/bbl dry

Drispac® Superlo® Polymer: 0.5 – 1.0 ppb (for filtrate control - not a lot but enough to help it carry some particles in the fissures and or vugs).

Mix as follows:

Drispac® Superlo® Polymer or Drispac® Plus Superlo® Polymer
50% of Flowzan® Biopolymer
DynaRed™ Fiber
50% of Flowzan® Biopolymer
Add water if getting un-pump able

Suggested pill size:

25 – 50 bbl.

APPENDIX IX

DYNARED™ FIBER FOR SEEPAGE LOSS

PREVENTION OF SEEPAGE LOSSES TREAT THE ENTIRE SYSTEM

Quick Reference Guide

<i>Products</i>	<i>Mud Weights</i>				<i>Comments</i>
Mud Weights PPG	7.0 to 12.5	12.5 to 15.0	15.1 to 17.0	17.1+	Add recommended amounts to active system.
<i>DynaRed™ fiber Fine</i>	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	
<i>DynaRed™ fiber Medium</i>	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	4 - 6 ppb	
<i>DynaRed™ fiber coarse</i>	0	0	0	0	
Calcium Carbonate	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	5 - 10 ppb	
Total ppb LCM	4 - 15 ppb	4 - 15 ppb	4 - 15 ppb	4 - 10 ppb	

REMEDIAL TREATMENTS FOR SEEPAGE LOSSES AS A SLUG TECHNIQUE

Quick Reference Guide

<i>Products</i>	<i>Mud Weights</i>				<i>Comments</i>
Mud Weights PPG	7.0 to 12.5	12.5 to 15.0	15.1 to 17.0	17.1 +	Pump sweeps while drilling depleted sands every connection then periodically as needed to Total Depth.
<i>DynaRed™ fiber Fine</i>	10 - 25 ppb	10 - 25 ppb	10 – 15 ppb	5 – 10 ppb	
<i>DynaRed™ fiber Medium</i>	10 - 25 ppb	10 - 25 ppb	10 – 15 ppb	5 – 10 ppb	
<i>DynaRed™ fiber Coarse</i>	0	0	0	0	
Calcium Carbonates sized	10 - 40 ppb	10 – 30 ppb	15 – 25 ppb	10 - 15 ppb	
Total ppb LCM	30-80+/- ppb	30 – 75+ ppb	25 - 50 ppb	20 - 30 ppb	

Advantages

- Can be mixed rapidly through the mud hopper and does not require bypassing of solids control equipment. DynaRed™ Fiber Fine has been used with mud cleaners having screens of 180 mesh without loss of product
- Seals off depleted zones without excessive build up of wall cake
- Works in both water based and oil based drilling fluids
- Unlike competitive materials it does not appreciably increase drilling fluid rheology
- Particle size distribution contributes to API filtrate reduction
- Does not readily degrade due to shear
- Resistant to attack by bio-organisms
- Works in drilling fluids of any pH
- Compatible with other common lost circulation materials
- Available in fine, medium and coarse grades

APPENDIX X

MIST DRILLING WITH DRISPAC[®] POLYMER

Mist drilling is an air drilling technique whereby water and a foamer (0.5-2%) are injected into the air stream to remove the produced liquid with minimum back pressure. Most often used to drill in dry formations that produce little formation fluids it can be used as a lost circulation drilling fluid when massive loss circulation occurs. As a loss circulation drilling fluid it offers the lowest possible ECD. An ECD of 0 to 2 ppg is possible with air and mist drilling fluids and this makes it very useful when drilling caverns or vugs where there are no returns. Large capacity high pressure air compressors and a rotating head are required for this type of drilling.

There are three types of Mist:

1. Water and foamer usually a soap solution (mist)
2. Mud and foamer (mud mist)
3. Polymer and foamer (Drispac[®] Polymer mist)

Water and foamer lifts produced water from the well bore but does not protect the dry exposed formation already drilled.

Mud mist is a thin mud instead of water injected along with a foamer to coat and protect the hole from water wetting.

The use of mist containing Drispac[®] Polymer has extended the time a well can stay on mist safely. Drispac[®] Polymer is an effective protective colloid and retards the disintegration and sloughing of shales due to water wetting.

Mist drilling with Drispac[®] Polymer has been used successfully for many years by various operators.

Both mud mist and Drispac[®] Polymer mist are designed to protect sloughing shales. Mud mist is an attempt to plaster off the well bore thus protecting the shales. Mud mist may contain Drispac[®] Polymer but the polymer is adsorbed on the clay and less available to inhibit shale. Drispac[®] Polymer mist on the other hand, has a higher concentration of available polymer and is more successful in controlling shales.

From 0.5 to 2 lb of Drispac[®] Polymer per barrel of water are mixed in the rig mud pit. If the formation water is hard meaning it contains calcium add 1-2 lb of soda ash or bicarb per barrel to treat out the hardness. Then from 0.5 – 2 percent by volume foamer is added to the Drispac[®] Polymer solution in the mist pump tank. The mist pump injects the Drispac[®] Polymer solution and foamer into the air line leading to the standpipe. Drispac[®] Polymer in no way reduces the effectiveness of foamers and tends to give more stable foam. Stiffer foams may be made by increasing the concentration of foamer and polymer to achieve better hole

cleaning capabilities and is especially useful for drilling large holes where air volumes are insufficient to properly clean the hole.

APPENDIX XI THE ACID SOLUBILITY OF DIASEAL M[®] LCM

Diaseal M[®] LCM is a blend of materials that quickly produces a stable, high water, high solids slurry. This slurry is very effective for overcoming lost circulation. It is placed in the well so that the solids from the slurry are deposited within the zone of loss by filtration. This filtration can be caused by squeezing or by normal leak-off caused by the hydrostatic head. The final seal is produced by the mud filter cake behind the deposited Diaseal M[®] LCM slurry solids.

It is unlikely that this type seal will damage a pay zone because of the high permeability of the Diaseal M[®] LCM plug. For example, the standard API water loss test is a relative measure of cake permeability. An unweighted slurry of Diaseal M[®] LCM has an extremely high water loss, in excess of 1000 ml. Thus, the Diaseal M[®] LCM cake or plug will also have a high permeability to air, the usual core test for permeability. This is, in fact true. Carbon dioxide which is used for pressure on filtration cells will blow right through a filter cake of Diaseal M[®] LCM.

However, if desired, Diaseal M[®] LCM cake plug can be removed by acidation. While it is only 18 percent soluble in hydrochloric acid (HCl), it is almost completely soluble in mud acid which is a mixture of HCl and hydrofluoric acid (HF).

SOLUBILITY OF DIASEAL[®] M LCM IN 15% HCL AND 2.1% HF ACIDS

Temperature

Percent Solubility vs. Time at

	1 hour	3 hour	6 hour	24 hour
24°C (75° F)	91%	95%	96%	98%
66 °C (150° F)	98%	98%	98%	99%

From the data, 5.4 gallons of 15% HCl and 2.1% HF acid are needed to dissolve one pound of Diaseal M[®] LCM. This is a large excess of HCl. According to the literature the HF concentration of an HCl-HF mixture can range up to 9% thus, much less total acid would be required. Doubling the HF concentration also roughly doubles the rate of reaction.

Filter cakes of Diaseal M[®] LCM have very high permeability. Therefore, it is unlikely that they would ever reduce production if placed in a pay zone. But if an operator desires to be absolutely sure, he can partially or wholly remove the plug of Diaseal M[®] LCM additive with HCl-HF acid.

APPENDIX XII

ANALYSIS OF A LOOK-A-LIKE SQUEEZE LOST CIRCULATION MATERIAL

From: Sam Ledbetter, Jr. Sr. Technical Service Engineer Drilling Specialties Co.

Subject: Analysis of a Look-a-Like Squeeze Lost Circulation Material

A sample of Lost Circulation Squeeze Material (LCM) from a competitor was sent for evaluation. The material was analyzed using x-ray fluorescence (XRF) and scanning electron microscopy (SEM). The material was also analyzed under an optical microscope and sieved for particle size distribution. For comparison purposes, a sample of Diaseal M[®] LCM was analyzed using the same procedures.

Conclusions:

The competitive product and Diaseal M[®] LCM are very similar materials. They both contain lime, paper, and diatomaceous earth. The difference between the two products appears to be the addition of fibrous material, extra paper, and lime in the competitive product.

Performance of the two materials is very similar at the same loading. The composition of a Diaseal M[®] LCM squeeze can be adjusted to match that of competitive product by the addition of common materials found on most drilling rigs, such as cedar fibers, nutshell, and lime. This would mimic the composition seen in competitive squeeze material.

Using Diaseal M[®] LCM as the nucleus, a very effective lost circulation material can be devised to meet specific requirements as shown in the past. Adjusting the composition of a Diaseal M[®] LCM squeeze slurry is addressed in the literature.

Test Results:

The generic squeeze material is a blend very similar to Drilling Specialties Co. Diaseal M[®] lost circulation material. Both products contain diatomaceous earth, lime and paper. The difference between generic squeeze material and Diaseal M[®] LCM is that the generic squeeze product contains a higher amount of paper and lime along with some type of fibrous material (See Photos of Diaseal M[®] LCM and competitive squeeze material).

The exact type of fiber found in the competitive squeeze product was not determined but looked to be some kind of straw or rice hulls. The increase in lime content of the competitive squeeze samples was determined by measuring the pH of various mixtures of the competitor’s product and water. One of the solutions reached a pH level of greater than 12. The Diaseal M[®] LCM mixture had a pH of 11.57. The additional paper was determined from the sieve analysis done on both samples. See (Graphs 1-3, Percent of Individual Components on Sieves)

The generic-look-a-like squeeze material has broader range of particle sizes than Diaseal M[®] LCM. Diaseal M[®] LCM has higher concentration of components in the 80 mesh or smaller size. See (Table I, Percent by Weight of Material Retained on the Screen)

TABLE I

PERCENT BY WEIGHT OF MATERIAL RETAINED ON THE SCREEN

	10 mesh	20 mesh	40 mesh	60 mesh	80 mesh	<80 mesh
Diaseal M[®] LCM	1.85	2.0	2.60	2.70	10.70	78.35
Look-a-like	6.25	7.95	12.05	7.05	3.95	60.80

X-Ray Refraction Analysis shows that both the generic-look-a-like squeeze product and Diaseal M[®] LCM to be nearly identical in elemental composition and in weight percent of each element measured. The increase in silica and aluminum show a higher concentration of diatomaceous earth in the Diaseal M[®] LCM, while the increase in calcium indicates a higher concentration of lime in the generic look-a-like squeeze sample. See Table II, X-Ray Refraction Analysis

Explanation of the scanning electron microscope images:

The top left image is the Diaseal M[®] LCM or generic material. The pictures next to it are x-ray elemental dot maps (EDM) of the images. To use the EDM, imagine stacking the maps on top of the SEM picture to locate the elements within the image. This will give a sense of what the material are that you are looking at in the SEM image. (See SEM Images 1- 5)

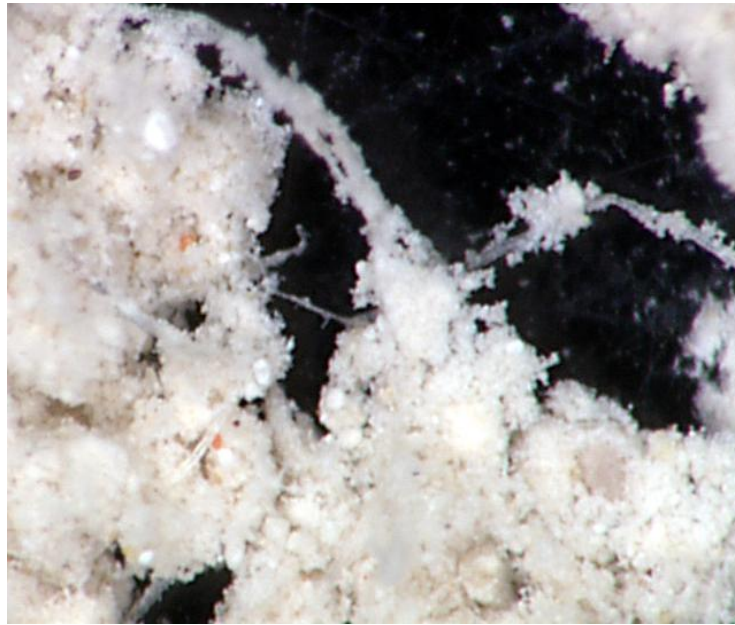


PHOTO OF DIASEAL M[®] LCM



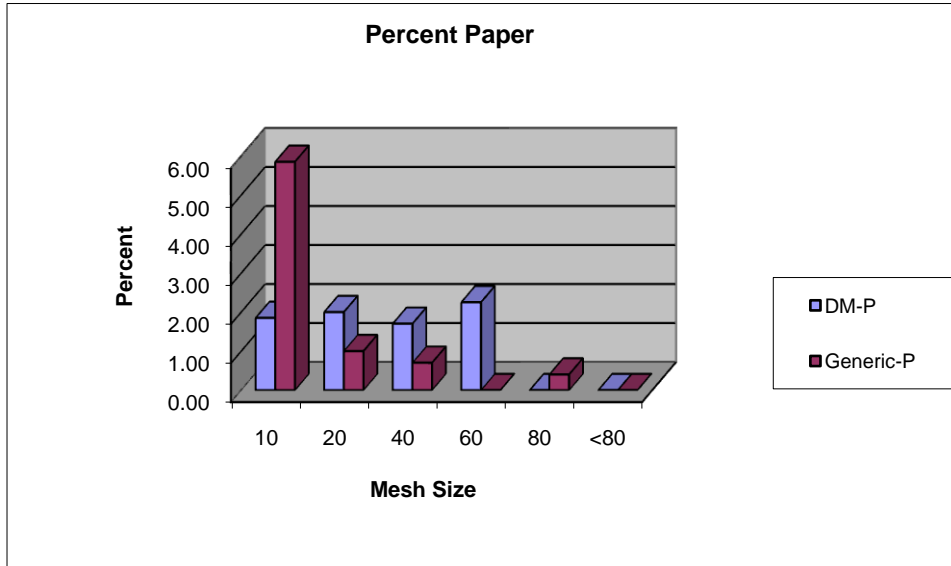
PHOTO OF GENERIC LOOK-A-LIKE PRODUCT

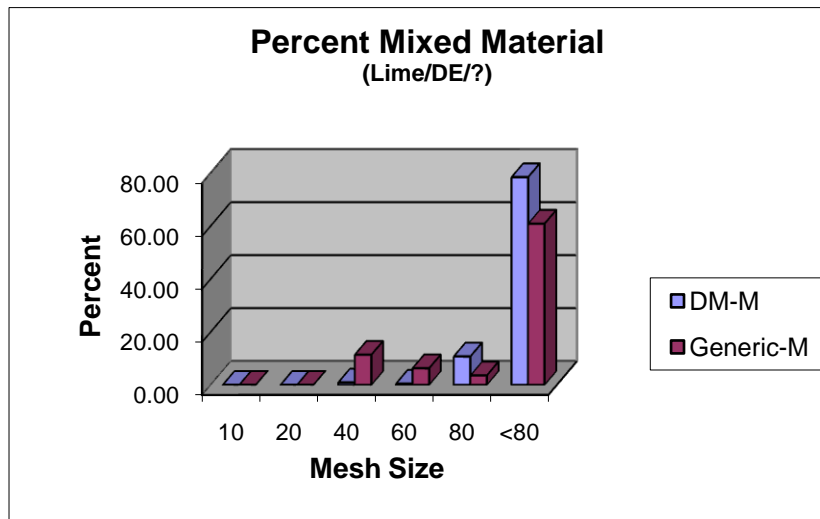
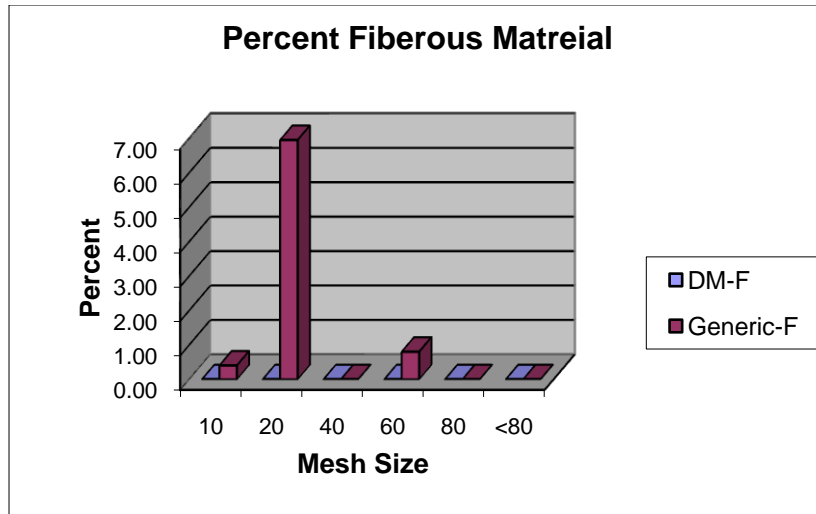
TABLE II

X-RAY REFRACTION ANALYSIS

Diaseal M[®]LCM		Generic look-a-like	
Element	Amount	Element	Amount
Al	3.72Wt %	Al	2.69Wt %
Ca	7.9Wt %	Ca	33.2Wt %
Cl	<Wt %	Cl	<Wt %
Fe	1.6Wt %	Fe	2.01Wt %
K	0.6Wt %	K	0.27Wt %
Na	0.14WT%	Na	<WT%
Mg	0.39Wt %	Mg	0.15Wt %
Mn	<Wt %	Mn	<Wt %
P	0.044Wt %	P	<Wt %
So	0.073Wt %	So	0.063Wt %
Si	15.6Wt %	Si	33.8Wt %
Ti	0.26Wt %	Ti	0.31Wt %
Sr	0.043WT%	Sr	<WT%

**PERCENT OF INDIVIDUAL COMPONTS ON SLEEVES
(Graphs 1-3)**





Test procedures:

Scanning electron microscope (SEM) and optical microscope images were done by Phillips Petroleum Co. analytical branch, as was the X-ray fluorescence spectroscopy. SEM images were done on one sample of Diaseal M[®] LCM and two samples of generic product. The images were done using two different levels of contrast, Grey Scale 1 and 2. Along with the SEM images, elemental dot maps (EDM) were made of the two materials to help identify the materials seen in each image.

The X-ray Fluorescence was done by scanning for fifty pre-determined elements and those elements below the detection limit of the instrument were not listed.

The Sieve analysis was done by taking 20 grams of material and sieving it through a stack of screens that was placed on a Roto-tap. The Roto-tap was run

for 20 minutes. The material that was left on the different screens was cataloged as to type and weight. The screen sizes used were 10, 20, 40, 60, and 80 mesh.

Less than 80-mesh size material was caught in a collection pan at the bottom of the stack of screens. The screens were in a stack in descending screen size, 10 mesh on top with 80 mesh on the bottom. At the start of the sieving process, the material was placed on the 10-mesh screen.

The percentages and total weights do not add up to one hundred percent. Some losses occurred while material was being transferred from the sieves.

IMAGE I
DIASEAL M[®] LCM

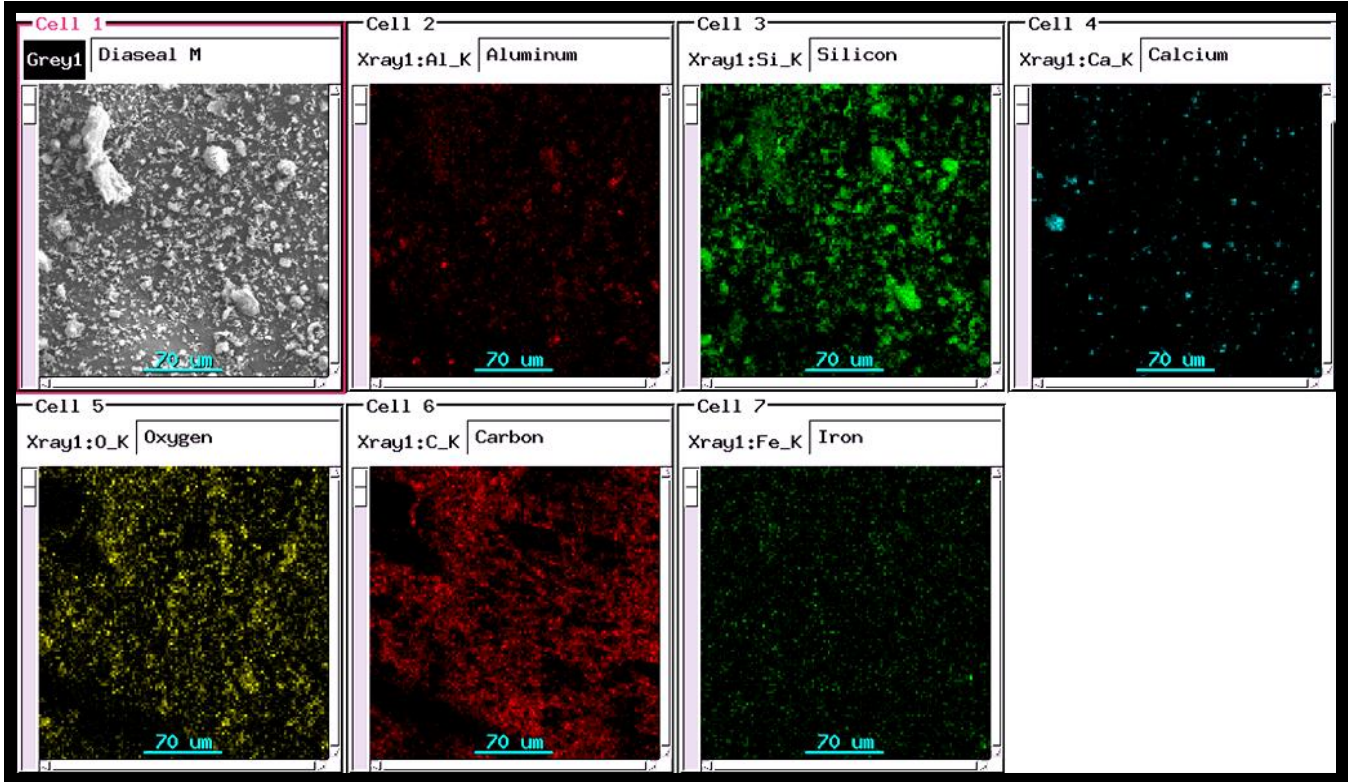


IMAGE II
DIASEAL M[®] LCM

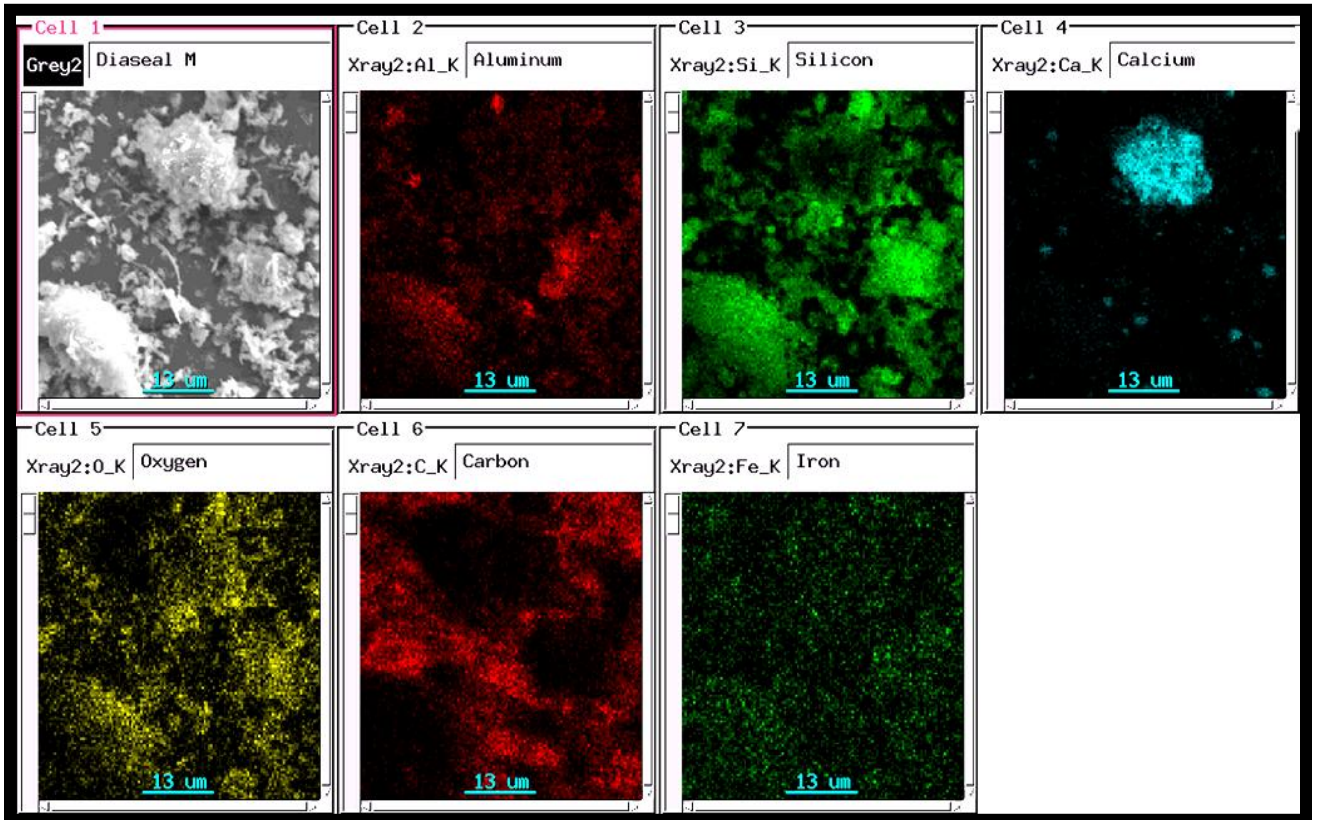


IMAGE III
GENERIC MATERIAL

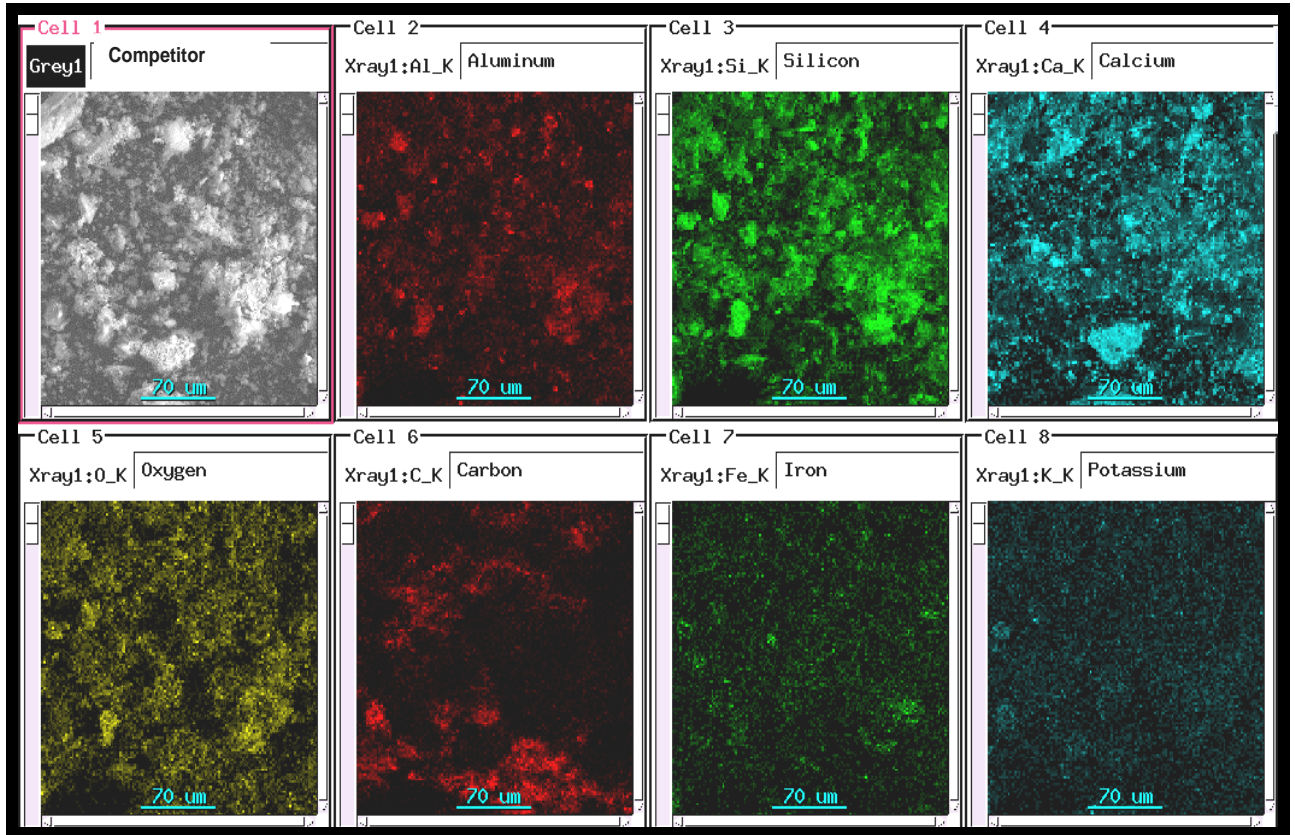
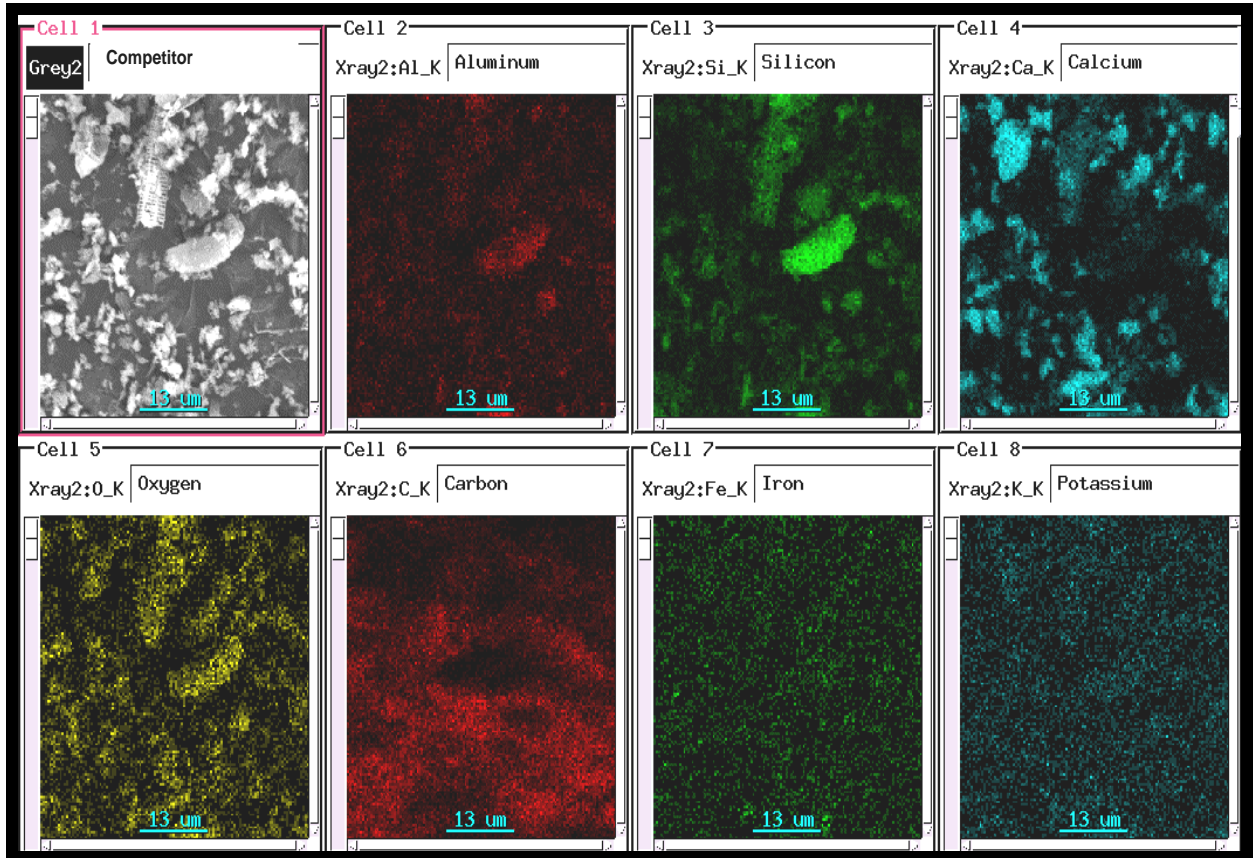


IMAGE IV
GENERIC MATERIAL



APPENDIX XIII

RETURN PERMEABILITY STUDY OF DYNARED™ FIBER

The data showing the effect of DynaRed™ Fiber on return permeability was generated by an independent laboratory. The return permeability test procedure and test results are in this report.

Samples A and B were prepared in the Drilling Specialties Company laboratory. Sample formulations were as follows:

- Sample A: Two 100 ml tap water + 60.0g bentonite clay (mixed 5 minutes) + 0.5 ml of 50% W/V NaOH solution (mixed 15 minutes) + 4.5g Drispac Regular polymer (mixed 20 minutes) + 120g Rev dust to represent drill solids (mixed 2 hours).
- Sample B: Sample A + 5.0 ppb DynaRed™ Fiber – fine grind (mixed 20 minutes)

Procedure:

- Two 1” diameter plugs were drilled from a block of standard Berea sandstone. The plugs were cleaned in a Soxhlet extractor using methanol to remove hydrocarbons, pore water and salt. After drying, ambient permeability to air and the porosity by Boyle’s Law helium expansion was determined for each plug.
- The core plugs were then saturated with a 35,000 ppm NaCl solution and each loaded into Hassler core holders and confined at an overburden pressure of 1,500psi. The permeability to brine was then determined for each plug.
- Each drilling fluid sample was then flowed through a core plug in the opposite direction to the brine flow. A drilling fluid pressure 50 psi greater than the pore pressure was maintained for a period of 4 hours. Brine was then flowed in the original direction and the final brine permeability was determined after 20, 50, and 80 pore volumes of displacement through the core plug.

Results:

The results are presented in the following table and in the attached graphs. Graphical data is presented in two versions:

1. Brine permeability in millidarcies versus pore volumes produced and
2. Normalized permeability versus cumulative pore volumes produced

RETURN PERMEABILITY DATA

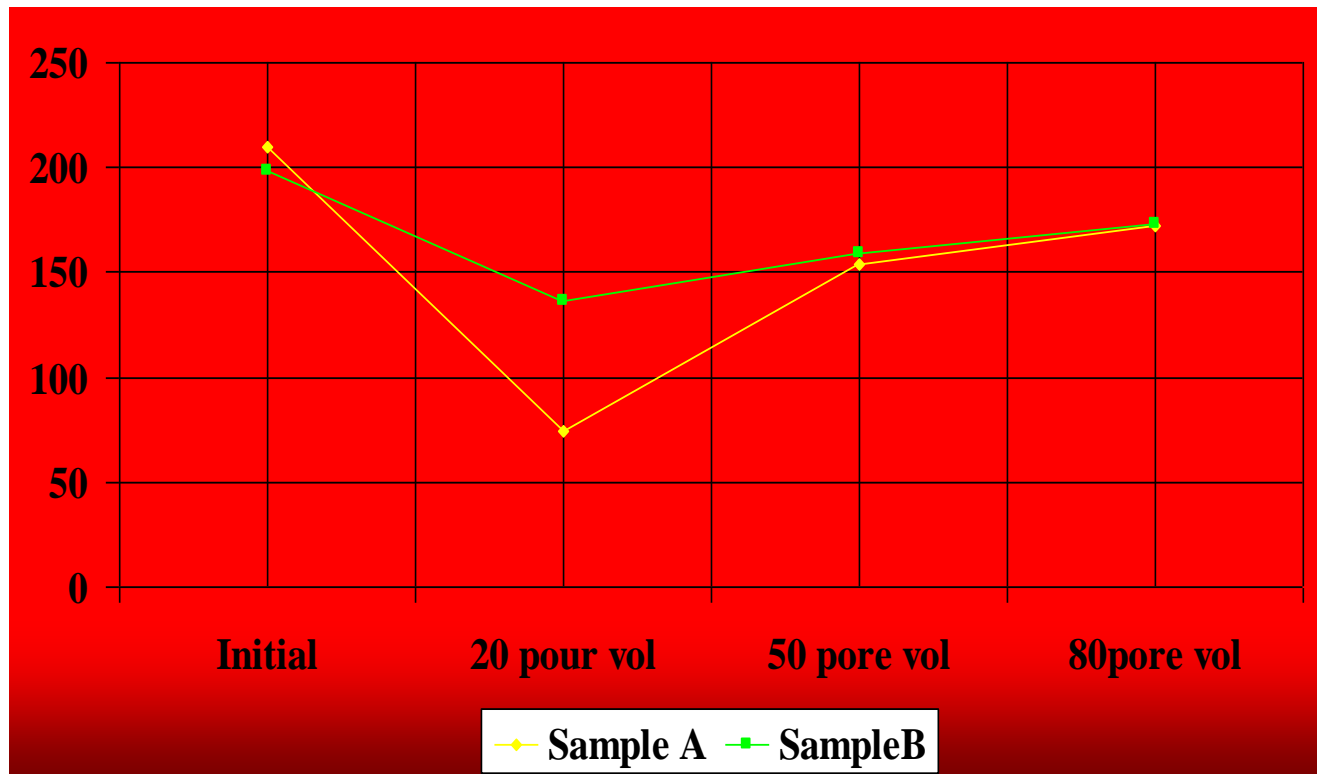
Without DynaRed™ Fiber

With 5 ppb DynaRed™ Fiber

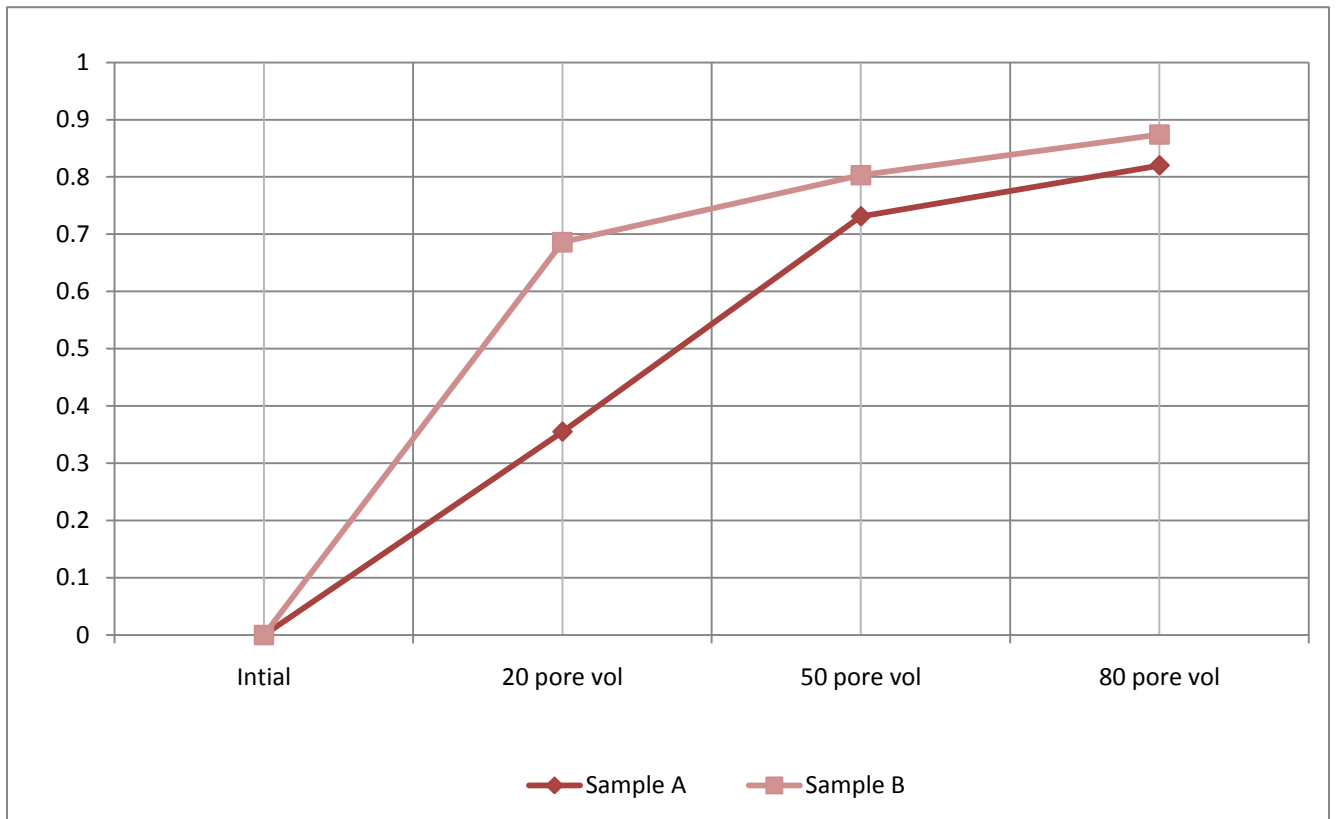
	Sample A	Sample A	Sample B	Sample B
Permeability	Permeability	Normalized Data	Permeability	Normalized Data
Initial to air	450.00		440.0	
Initial to brine	210.0	1.000	198.4	1.000
Brine after mud invasion	74.5	0.355	136.2	0.686
Brine after 50 pore volume	153.5	0.731	159.3	0.803
Brine after 80 pore volume	172.2	0.820	173.5	0.874

Note: The final cleanup return permeability's were similar after 80 pore volumes of displacement. Sample B cleaned up more rapidly.

RETURN PERMEABILITY VS PORE VOLUMES PRODUCED



CLEAN UP RETURN PERMEABILITY NORMALIZED



SOURCES AND RECOMMENDED READING

The “Composition and Properties of Oil Well Drilling Fluids” 4th addition by George R. Gray and H.C.H. Darley, Copyright 1980 by Gulf Publishing Company, Houston, Texas.

“Lost Circulation” by Joseph U. Messenger Copyright 1981 PennWell Books

“A New Approach to Preventing Lost Circulation while Drilling” by Fuh, Giin–Fa., SPE paper 24599

“Drilling Fluids for Wellbore Strengthening” by M.S. Aston, M. W. Alberty, M.R. McLean, H.J. deJong, and K. Armagost, BP Exploration, IADC/SPE paper 87130, published 2004

“Cement & Drilling Fluid Reactive Pills” Technical Memorandum 96-13, Chevron

“Evaluation of Lost Circulation Materials for Water-Based Muds F96-P-72 September 30th, 1996 962780002-Tul, EPTG-AMOCO by Frederic B. Growcock, Tyrone P. Frederick, Angelia R. Reece, and Sabrina H. Sinor

PRODUCT DESCRIPTIONS

Diaseal M[®] LCM

Description:

- A filtration activated pill material
- High fluid loss, high solids, lost circulation squeeze product. Can be enhanced with cement for greater compressive strength

Formations:

- Fractured or highly permeable formations

Mud Types:

- All water and oil based muds

Applications:

- Partial lost returns while drilling
- Total lost returns while drilling
- Squeeze off casing leaks or perforations for well remediation

Rebound[™] LCM Regular and Fine

Description:

- Rebound[™] LCM is a carbon-based resilient material that becomes tightly compressed into the loss zone
- Rebound[™] LCM has the ability to expand and contract without dislodging with changes in differential pressure. Rebound[™] LCM is packaged in fine and coarse grades

Formations:

- Seepage losses in permeable formations

Mud Types:

- All water and oil based muds

Applications:

- Seepage losses while drilling
- Spot as a pill across the loss zone

DynaRed[™] Fiber Fine, Medium and Coarse

Description:

- Cellulose fiber available in fine, medium and coarse grades

Formations:

- Depleted sand and permeable formations.

Mud Types:

- All water and oil based drilling fluids

Applications:

- DynaRed Fiber can be applied to combat seepage or spotted as a lost circulation pill

Poly Block™ LCM

Description:

- Surface activated chemical reactive pill material
- A blend of loss circulation material and a hydrateable cross-linked polymer that swells with time and temperature to seal the thief zone in a lost circulation situation.

Formations:

- Vugular and cavernous formations

Mud Types:

- Water based muds and oil based muds where a clean mixing tank can be employed.

Applications:

- Poly Block™ LCM can be used to combat partial or total lost returns; it acts quickly to seal off the thief zone

CASE HISTORIES

DIASEAL M[®] LOST CIRCULATION MATERIAL CASE HISTORIES

CASE HISTORY # 1 – LOUISIANA, USA

An operator in Perry, Louisiana had drilled out of 7 5/8” casing with an 18.1 ppg mud. After 1,200 feet had been drilled, drilling fluid losses of 71.2 bbl/hour were noted. For the 70 bbl of open hole volume, a 150 bbl Diaseal M[®] LCM pill was squeezed to a 250 psi holding pressure. The procedure was successful and the well was TD'd at 14, 500 feet without further problems.

CASE HISTORY # 2 – ALBERTA, CANADA

A major Canadian operator was able to eliminate a string of intermediate casing in a well near Slave Lake, Alberta, through implementation of a Diaseal M[®] LCM squeeze. Due to total losses in the Wabamum formation, they blind-drilled to intermediate casing point. A 20 m³ (125 bbl) of unweighted slurry with an additional 43 kg/ m³ (15 lb/bbl) Diaseal M[®] LCM was mixed and placed into the wellbore above the uppermost loss zone. After waiting 2 hours while hydrostatic pressure squeezed the slurry, the annulus was filled with drilling fluid. By keeping the annulus filled over the next hour, the hole finally stood full. Closing the annular BOP and pumping into the annulus through the kill line then implemented the “hesitation squeeze” procedure. The desired holding pressure of 1200 kpa (175 psi) was achieved in about 1 hour. The production interval of the well was then cored, logged and cased without further mud losses. This operator has had numerous successes with Diaseal M[®] LCM squeezes in this area.

CASE HISTORY # 3 – MISSISSIPPI, USA

A major operator experienced severe lost circulation in Greene County, Mississippi while drilling at 16,632 feet with an 18.2 ppg oil based drilling fluid. The limestone formation had been broken down during a gas kick where the SIDPP was reported to be 2800 psi. After a number of unsuccessful LCM pills and three unsuccessful cement squeezes, (oil mud losses totaling 2, 621 bbl), a Diaseal M[®] LCM squeeze was attempted. A 100 barrel slurry of Diaseal M[®] LCM and diesel was mixed in a turbine blender and weighted to 18.2 ppg. The slurry was pumped into place with a cement pump truck, and a 10 bbl diesel spacer was pumped ahead and behind the slurry to prevent any cross-contamination. A total of 56 barrels of Diaseal M[®] LCM slurry was squeezed into the formation, utilizing the “hesitation squeeze” technique. A holding pressure equivalent to 19.3 ppg was obtained and circulation was regained. Another Diaseal M[®] LCM squeeze was required at greater depth after the residual cement had been cleaned out. No further loss problems were encountered in this interval, and drilling fluid density was 19.1 ppg at TD.

CASE HISTORY # 4 – LOUISIANA, USA

Severe lost circulation was experienced at 16,550 feet with an 18.0 ppg oil based drilling fluid near Lake Arthur, Louisiana. A 100 barrel 18.0 ppg oil based **Diaseal M[®] LCM** squeeze was implemented and a 375 psi holding pressure was obtained. Drilling resumed, but another loss zone was encountered 50 feet deeper. A second squeeze was applied and it, too, was successful. This procedure had to be applied again for one additional loss zone at greater depth, and TD was reached without further problems.

CASE HISTORY # 5 COLORADO, USA

Location: (Pieance Creek), Colorado
Operator: Independent Oil and Gas Company
Hole-Depth: 11,660'
7" Liner set: 10,990'

Squeeze Formula: 60 bbls H₂O—80 Sx **Diaseal M[®] LCM**—20 Sx CaCO₃—25 Sx Additional LCM and 40 Sx Barite

Squeeze Procedure: RIH to 9,880' (Make sure tool joint is not in middle of BOP)
 Pump squeeze to end of DP + 4 bbls to fill lines (1,380 Strokes @ .070 bbls/Strokes@2 bbls/min) Close the BOP

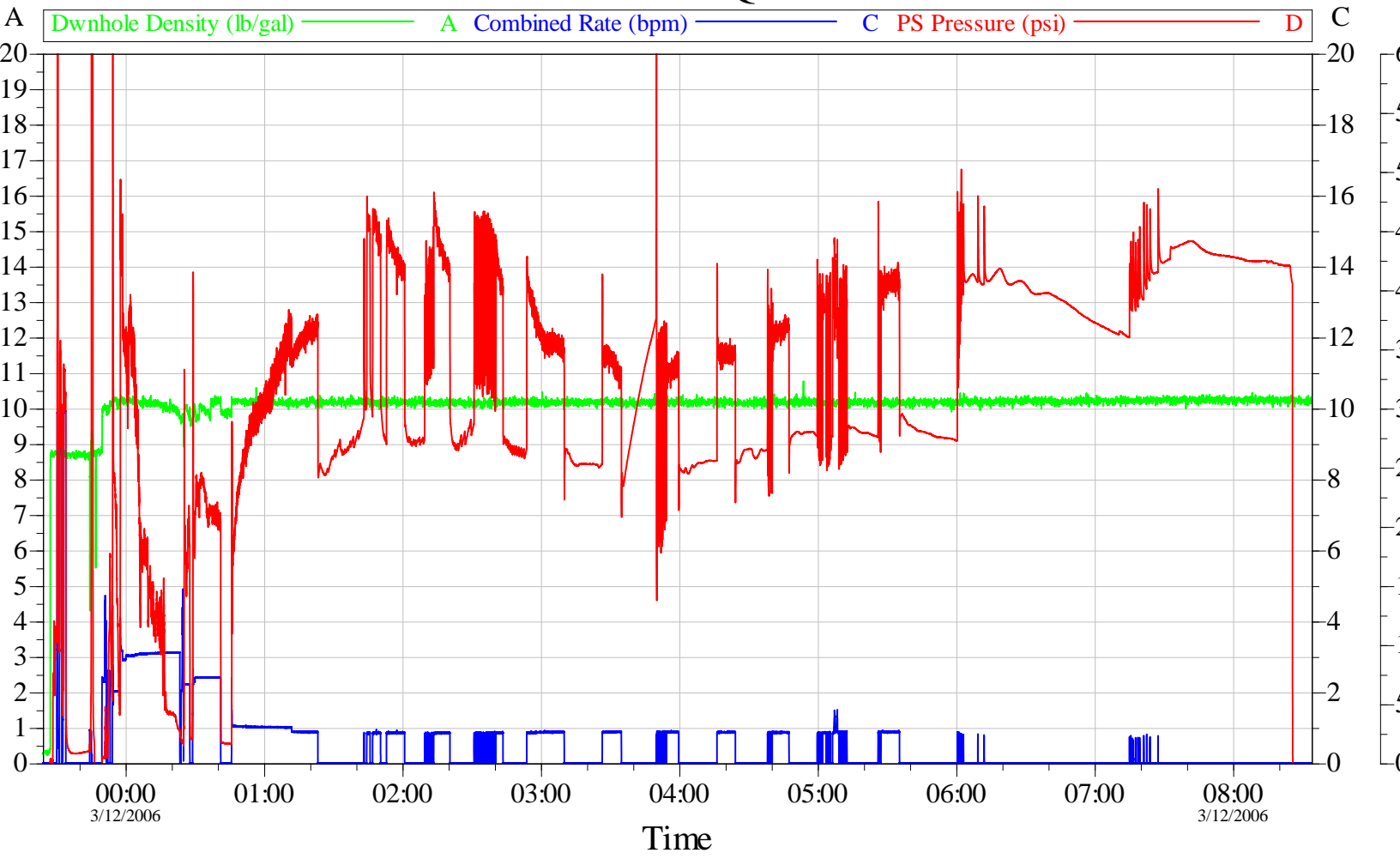
Pump @ 1bbl/Min (Open-hole Vol. +20 bbls) Total 46 bbls (650 Strokes)

Reduce pump rate to ½ bbl /min for 5 bbls or until 50 psi increase is reached.

Time	Diaseal M [®] LCM	Mud Volume	PSI Static	PSI Circulating
23:55	60			240
00:25		30		260
00:40			200	
01:10		50		340
01:23		57	230	
01:45			270	
01:47		58		440
01:48		60		306
01:50				440
01:52		65	270	
02:00				420
02:08			270	
02:20		70		420
02:30			280	
02:45		75		390
02:53			265	
03:10		80		350
03:25				330
03:35		90		330
03:50			372	
04:00		95		345
04:15			255	
04:25		100		345
04:40			265	
04:47		105		370
05:00			276	
05:12		110		360
05:27			276	
05:36		115		407
06:00			272	
06:15		117	414	

**Bumped Static Pressures to 489-467-511-443-435 PSI respectively
 Pressures held for 3+ Hours @ 435 PSI (EMW 15.5+ ppg)**

DIASEAL-M SQUEEZE



DYNARED™ FIBER CASE HISTORIES

CASE HISTORY # 1 – OFFSHORE GULF OF MEXICO

A major international oil and gas company drilled a well in Louisiana waters. The problem was the upper portion of a sand taking fluid and not permitting continued drilling for fear of creating a well control problem. The drilling fluid was very dense (18.3 ppg), and this prevented the addition of large amounts of traditional types of seepage control materials such as Gilsonite, ground fiber and fine mica.

An LCM pill made with combinations of two different competitive ground fibers totaling 10 – 14 ppb produced funnel viscosities of 150+ sec/quart and required extensive diluting and weight up in order to be pump able. This pill was placed on bottom and pumped with the following results. The first attempt to seal the seepage zone resulted in pumping 19 bbl drilling fluid and reaching a high of 257 psi before breaking back to 200 psi. The second attempt resulted in pumping 17 bbl and reached a high of 257 psi before breaking back to 200 psi. The pill was circulated out and not left in the hole.

The pill built with 15 ppb DynaRed™ fiber and 10 ppb of sized calcium carbonate had a resulting funnel viscosity of only 120 sec/quart. The rest of the mud properties were PV = 94, YP = 54 and Gels = 27/44. The pill was diluted back to 100 sec/quart funnel viscosity reweighted to 18.3 ppg and pumped. After placing the pill, 4-5 bbl were pumped, the pressure built to 360 psi and then broke back to 170 psi. After pumping another 4-5 bbl, the pressure built to 406 psi and bled off slowly to 200 psi. This was good enough to come out of the hole, change bits and return to bottom. The mud on bottom after a 20 hour trip was very pump able and the pill was washed out with no problems. The drilling manager for this company credits DynaRed™ fiber with solving his lost circulation problem.

CASE HISTORY # 2 CENTRAL TEXAS GULF COAST USA

An independent turnkey drilling contractor drilled two wells in Jackson County, Texas using DynaRed™ fiber. DynaRed™ fiber was added at 7440 feet to prevent seepage to upper Vicksburg sands that were a known problem. Initial treatment was 1/3 ppb (12 sacks) and additional material was added at a rate of 1 (25 lb sack) every two hours. Mud weight was 15.5 ppg. The mud service engineer reported that DynaRed™ fiber mixed in well and did not cause the drilling fluid to thicken like other fibers did. No problems were encountered and the drilling contractor was pleased enough to reorder DynaRed™ fiber for a second well to be drilled in the same area. No problems were encountered on the second well either.

CASE HISTORY # 3 - CENTRAL TEXAS GULF COAST WASHINGTON CO., USA

An independent operator drilled a chalk well in Washington County, Texas using DynaRed™ fiber. DynaRed™ fiber was added at 4000 feet just 200 feet prior to drilling sands in the upper Wilcox formation. Treatment was 1 sack per hour while drilling and this treatment rate was maintained through 8,200 feet. Sands that normally took large amounts of drilling fluid while drilling were sealed off. The operator felt that DynaRed™ fiber contributed to the success of the drilling operation and continued to use DynaRed™ fiber all the way to casing point.

CASE HISTORY # 4 - UPPER TEXAS GULF COAST - BRAZORIA COUNTY USA

A major oil and gas company drilled a well in the Chocolate Bayou area (Brazoria county, Texas) using a combination of fiber including DynaRed™ fiber. The well was drilled successfully.

CASE HISTORY # 5 - CENTRAL TEXAS GULF COAST - BROOKS COUNTY USA

A turnkey operator drilled a well in Brooks County, Texas. At 8500 feet the water based drilling fluid was displaced with a oil based drilling fluid. This fluid was treated with 2 ppb DynaRed™ fiber and successfully drilled to 11,300 feet.

POLY BLOCK™ LCM CASE HISTORY

CASE HISTORY # 1 OKLAHOMA USA

Date: January 26, 2005

Operator: Major International Oil and Gas Company

Mud Company: Major Independent

County: Stephens, Oklahoma

Problem: Lost Circulation

Mud Type: 16.0 ppg OBM

Product Application: Poly Block™ LCM

A major operator experienced severe lost circulation at 13,650' with 16.0 ppg oil-base mud near Chickasha, Oklahoma. After a number of unsuccessful LCM pills as well as the active mud system having 14 ppb of LCM, circulation was still not obtained. The oil-based mud losses totaled 500 bbls and it was decided to pump a Poly Block™ LCM pill.

A 96 bbl Poly Block™ squeeze pill was mixed and weighted up to 10.7 ppg with barite. The squeeze was pumped into formation. While pumping the Poly Block™ LCM squeeze mud losses were 30 bbls per hour. After the squeeze was set into place full circulation was obtained immediately.