

# Aging of Water Based Drilling Fluids

A discussion of the techniques and equipment commonly used to mix and age water based drilling fluid formulations, including some basic guidelines and safety considerations.

## Description

Drilling fluid aging is the process in which a drilling fluid sample, previously subjected to a period of shear, is allowed to more fully develop its rheological and filtration properties. The time period needed to more fully develop properties varies from as little as several hours (usually 16 hours) to as much as several days. The aging can be done at either ambient or elevated temperatures.

# PREPARATION, HANDLING AND TESTING

#### Water-Based Drilling Fluids and Components

Most drilling fluid formulations contain a base liquid and additives which must be dissolved or mechanically dispersed into the liquid to form a homogenous fluid. The resulting fluid may contain one or more of the following: water-dispersible (soluble) polymers or resins, clays or other insoluble but dispersible fine solids, and soluble salts. The fluids are mixed or sheared for times appropriate to achieve a homogenous mixture and are then set aside to "age." Aging is done under conditions which vary from static to dynamic and from ambient to highly elevated temperatures.

#### Mixing/Blending/Shearing Devices

Drilling fluid formulations are commonly mixed with various shearing devices which may be either fixed speed or variable speed. The motors may turn mixing shafts with rounded "propellers," sharp blades, wave-form shapes, or others. Single shaft or multiple shaft devices are used. Some examples of the more widely used mixer types are: Hamilton Beach<sup>®</sup> Model 936, Dispersator<sup>®</sup> high shear mixer, Waring Blendor<sup>®</sup>, Multi-Mixer<sup>®</sup> Model 9B with 9B29X impeller, Silverson<sup>®</sup> Model 14LR mixer, and Oliani<sup>®</sup> mixer. Nozzle shear devices are also used to prepare some formulations.

Shearing devices vary widely in the amount of shear they impart. Longer shearing times may be required for low shear devices to achieve complete dissolution/hydration of fluid components; while high shear devices may produce nearly completely yielded drilling fluid blends in a few minutes. Aging of drilling fluid samples tends to minimize differences in properties which can result from shearing treatment.

#### pH Levels of Drilling Fluids or Base Fluids

The pH of a drilling fluid formulation containing bentonite clays usually never falls below a value of 8.5 unless acidic material are added to these based fluids. Except for some drilling fluid systems viscosified with certain water-soluble polymers, the pH of these formulations is usually raised above pH 8.5 with alkalinity control agents, such as sodium or potassium hydroxide (caustic soda or caustic potash), or calcium hydroxide (lime).

**CAUTION:** Wear eye protection whenever drilling fluids are formulated, handled or tested.

Fluid alkalinity is lowered by the reaction of hydroxide groups with aluminosilicates (clays), gradually at ambient temperature and rapidly at elevated temperature. Some drilling fluid additives require that alkalinities be maintained within certain narrow, but elevated, ranges in order to function at optimum levels. Therefore, pH levels are often raised after aging if there has been a substantial pH drop.

# Drilling Fluid Sample Storage, Disposal

Drilling fluid formulations are usually kept until used up or until properties move outside acceptable ranges. Some laboratories do not routinely keep unused portion of test muds at ambient temperatures longer than some arbitrary time, such as one week or one month. Prolonged storage is best done in refrigerated units with capability of storage up to one year at 40°F (4°C).

Drilling fluid samples which contain certain organic materials or polymer products subject to fermentation (i.e., starches, biopolymers, etc.) should either have a preservative added or should be discarded after a suitable time which precedes the expected onset of biological degradation. Unused samples should be discarded in an environmentally appropriate manner based upon known ingredients.



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Fann Instrument Company offers a complete line of equipment, materials and supplies for use in aging drilling fluids in accordance with *API Recommended Practice 13I*.

# AMBIENT TEMPERATURE PREPARATION AND AGING

## Description

Drilling fluid samples may be mixed at ambient temperatures in a variety of open containers made from metal, plastic or glass. Little or no incompatibility exists between normal water-based drilling fluid and these materials under ambient temperature conditions.

# Equipment

a. Containers: Crack-resistant glass, such as home canning jars, supplied by Mason<sup>®</sup>, Kerr<sup>®</sup>, or BALL<sup>®</sup>, or equivalent jars or bottles, and Heat-resistant containers, such as Pyrex<sup>®</sup> or equivalent materials. Metal containers (aging cells) may be used at ambient temperatures. Commonly used metal include various grades (303, 304 or 316) of stainless steel. More inert metal formulations may be used but these are expensive for ambient temperature use.

**Note:** Metal containers (aging cells) made of aluminum bronze, are not recommended for any drilling fluid formulations. Reactions between bronze aging cells and numerous drilling fluid systems or additives have been observed.

- b. Coverings/lids for glass vessels are commonly plastic or metal, often lined with plastic, rubber, enamel, or other relatively "inert" material. Plastic containers and lids may be made from polyethylene, polypropylene, or other suitably inert, mechanically strong, and durable materials.
- c. Mixers, as described above.
- d. Balance with precision of  $\pm 0.01g$ .

## Procedure

After the initial shearing/blending stages of drilling fluid base, or after full sample preparation, most additional aging at ambient temperatures is done statically.

Prepared samples are left overnight (16 hours) or for days, if necessary, to reach stable or desirable properties.

Rolling or tumbling (rotating) the prepared samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.

Drilling fluids are routinely aged at ambient temperatures in metal, plastic or glass containers capped to prevent loss of moisture. The fluid properties may continue to change until the components are fully hydrated.

The properties may continue to change as some components react further in the aqueous suspension to degrade due to secondary reactions between various components, or due to bacteria attached on susceptible materials. Biocides may be added to prolong shelf life of these susceptible formulations.

Drilling fluid formulations which are to be kept for extended periods are usually stored under refrigeration at 40°F (4°C). Drilling fluids stored at ambient temperatures are often discarded after several days and are seldom kept for longer than a month. Formulations are monitored to ensure that the properties remain within acceptable ranges.

# MODERATE TEMPERATURE AGING (UP TO 150°F (66°C))

#### Description

Drilling fluid samples to be aged at moderately elevated temperatures are mixed at ambient temperatures as described above. Additional materials may be added to slurries which have already been aged at ambient or elevated temperatures.

# Equipment

- a. Containers: Most of the acceptable glass, plastic or metal containers used for ambient aging have been found to be acceptable for static or dynamic aging up to and including 150°F (66°C).
- b. Covers: Acceptable covers are ones which seal the container.
- c. Oven: Regulated to 150°F (66°C), or other suitable temperature.
- d. Mixer: As described above.

# Procedure

Aging at moderately elevated temperature is usually done to hasten the equilibrium hydration level of clays and/or polymers in the fluid system, or to expose the fluid to thermal conditions similar to field conditions.

For exposure to moderate temperature, drilling fluid samples are placed in one of a wide variety of commercially available or custom-built ovens. These ovens range from being tabletop, portable models small enough to hold only a few one-pint samples, to large floorstanding units having very large capacities. These ovens also vary from static units to those equipped with rollers or pulley systems to roll the containers or to tumble (rotate) strapped-in cells.



Preliminary studies indicate that the methods of heating and cooling the drilling fluid samples (i.e., preheating the oven versus no preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequate air circulation), and exact heating exposure time will affect the data values measured. To optimize data repeatability between test runs, use the same heating and cooling methods and heat exposure time for each sample in a series of comparative tests.

One artifact of aging at temperatures through 150°F (66°C) is that some glass and plastic vessels containing drilling fluids, especially those with elevated salinity (e.g., to sea water salinity of approximately 19,000 mg/L chlorides) develop modes pressures in the air overlying the liquid drilling fluid. After 15-30 minutes of heating, these vessels are removed from the oven, the lids carefully loosened to allow excess pressure to escape ("burping"), the lids restored tightly and the containers replaced in the oven to continue heating to the desired temperature. Sometimes a second check for pressure buildup is advisable.

## Maintaining Fluid Properties at Moderate Temperatures

As previously mentioned, alkalinity levels drop with time when exposed to clay (or other drilling fluid additives which exhibit weakly acidic behavior). The reaction between clays and hydroxyl ions is accelerated with increasing temperature. Therefore, alkalinities need to be more closely monitored and more frequently adjusted, if desired, in drilling fluids which are exposed to elevated temperature.

Purging and blanketing samples with nitrogen gas appears to impart strong benefits in reducing oxidative degradation of polymer-treated samples. The use of nitrogen leads to better reproducibility in testing polymer drilling fluids.

## Storage and Testing Practices for Samples Aged at Moderate Temperatures

Even though samples may have been aged at moderately elevated temperatures for time periods ranging from hours to days, prolonged storage of such samples, if desired, is usually done at ambient temperatures or under refrigeration, depending upon the length of time for anticipated storage.

# ELEVATED TEMPERATURE AGING (OVER 160°F (70°C))

## Description

Drilling fluid samples to be aged at substantially elevated temperatures are also blended at ambient temperatures as described previously. Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperature may be added to slurries which have already been aged at ambient or elevated temperatures.

#### Equipment

Once the 150-160°F (66-71°C) temperature threshold is crossed, only aging cells constructed from suitable metals are used to expose drilling fluids to elevated temperatures.

Note: Aluminum bronze cells are not recommended for testing drilling fluids at any temperature.

Since aging temperatures are often selected to be near the estimated or anticipated bottom-hole temperatures, aging cells chosen must be designed and constructed in such a way as to meet, or exceed, the pressure and temperature requirements of the aging tests.

Beyond the issue of being able to meet the temperature and pressure requirements of specific aging conditions, the choice of the proper metal is complex. It depends both upon the ultimate temperature which the sample and cell will experience as well as the salinity & ionic activity of the fluid sample.

The largest percentage of aging between 150 and 350°F (66 and 177°C) is done in cells constructed from various grades of stainless steel (303, 304 or 316). Sometimes high carbon-content steel cells are used if there is a strong desire to simulate field conditions wherein "mild steel" pipe is in use, and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e., they have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

For prolonged exposure to elevated salinity at high temperatures, e.g., 20,000 mg/L chlorides at 350°F (177°C), cells constructed from premium metals may be desirable. Examples of these premium metals are; Hastelloy<sup>®</sup> C-276 alloy, Inconel<sup>®</sup> alloy 600 and Incoloy<sup>®</sup> alloy 825. Cells made from these premium metals with the same wall thickness as standard stainless steel cells, however, suffer from lower strength ratings. Therefore, cells made from premium metals do not have the same pressure ratings as stainless steel and must be de-rated to lower pressure limits.

A key consideration in the use of metal aging cells at elevated temperatures is that care must be taken to ensure that the cells not be overfilled. When enclosed liquids expand with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as liquid hydraculically loads against the cell cap. Leaving gaps of 1.5 to 2 in. (3.8 to 5.1cm) between the top of the liquid and the cell cap, or filling the cell to only 85 - 90% of its volume, appear to be adequate for safe operation during most aging conditions between 150-400°F (66-190°C).

## Safety Considerations

Metal aging cells should only be used to age drilling fluids containing the usual types or classes of mud additives. Do not use materials whose high temperature compatibility with drilling fluid chemical and minerals are in doubt. Test such compatibilities in equipment, such as autoclaves, specifically designed for extreme pressure service. For protection, perform these compatibilities in specially designed enclosures.

The only known instances of catastrophic failures (explosions) of stainless steel aging cells resulted from either the cells being used inappropriately as chemical reaction autoclaves or being over-filled.

The types of failures of metal aging cells reported either from inadequate inspection and maintenance or from prolonged use for drilling fluids aging, were simply leaks which developed in the cells. The drilling fluids leaked into the aging ovens but caused no danger to laboratory personnel.

Rupture disks may be installed in the cell caps if there is concern about cell failure. Since the use of rupture disks essentially de-rates the pressure rating of the metal aging cell, conduct tests at temperatures that keep the pressure lower.

#### Metal Aging Cell Maintenance

Cell bodies and caps should have serial numbers recorded along with the cell contents whenever they are used. Separate sets of metal aging cells should be maintained for testing fresh water fluids, high chloride fluids, and oil-based fluids. The cells exposed to fresh water should have the longest useful life.

Stainless steel aging cells tend to experience chloride-stress corrosion cracking or pitting in environments where there has been prolonged exposure at high temperatures, e.g., over 220°F (105°C) to fluid with substantially elevated chloride levels (over several thousand mg/L). Such concerns are legitimate, but there are a number of mitigating circumstances which exist in drilling fluids aging technology. These tend to lessen the severity of the effects experienced.

One drilling fluid aging practice that mitigates accelerated corrosion is that metal aging cells are washed and visually inspected after each use to see if they have been affected by the exposure. The onset of pitting and cracking corrosion is often detected at such a time. Affected stainless steel cells are then usually sand blasted to remove corrosion spots. These cells can be returned to useful service.

Even when no obvious corrosion in a metal aging cell is detected, historical experience has shown that periodic sand blasting of the cells removes developing problem sites, keeps the surface passive, and significantly increases the useful life of the cell.

#### Other mitigating circumstances include:

- a. The cells are used intermittently and cleaned, and are not used in continuous process exposures.
- b. The cells used in drilling fluids testing are made from metal bar stock and are not welded. The absence of the stress point which would be present in welded or cast cell bodies mitigates against the likelihood or severity of corrosion which might be experienced under certain aggressive conditions of exposure. *Fann aging cells are manufactured from solid metal bar stock*.
- c. Drilling fluid formulations have high pH levels and typically low oxygen contents.

The recommended equipment and procedures for aging waterbased drilling fluids may be found in *API Recommended Practice 13B-1* and *API Recommended Practice 13I*.

# Procedure

If the correct match of cell metallurgy and drilling fluid is established for a given temperature regime, then the samples may be statically or dynamically aged in a suitable oven, as described earlier.

Dynamic aging should be performed in rolling or rotating ovens capable of achieving and safely maintaining temperatures of 150-400°F (66-190°C). A 16-hour minimum aging time is recommended for such exposures. Depending on the temperature stability of the product(s) being evaluated, the time and temperature may need to be adjusted.

For static aging, any qualified oven or the same ovens may be used but with their rollers/rotating mechanisms turned off. Static aging at elevated temperatures simulates the conditions when a drilling fluid is left quiescent down-hole during regular rig operations.

For high temperature wells over 300°F (135°C), the usual 16-hour aging interval is a reasonable simulation of the time a drilling fluid is left in the hole during a bit trip. For longer operations, such as extended electric well logging runs, 48 to 72-hour aging periods is appropriate. In choosing a test temperature, it should be noted that fluid left in the hole may take many hours to even approach the actual bottom-hole temperature since well-bores are cooled by the circulating fluid. Therefore, a test temperature below the bottom-hole temperature may be the most realistic simulation of down-hole conditions for bit trip periods.

**Note:** A meaningful API test to use in evaluating long term gelation of heat-aged drilling fluids is the shear strength test found in *API Recommended Practice 13B-1*, Appendix entitled "Shear Strength Measurement Using Shearometer Tube."

## CAUTIONS ABOUT INERTNESS AND CHEMICAL COMPATIBILITY

#### Chemical Compatibility of Materials within Metal Aging Cells

As mentioned earlier, do not subject materials of unknown or suspicious reactivity to high temperature aging in cells containing drilling fluids.

Do not include materials which are known to produce, or suspected of initiating or taking part in highly exothermic (explosive) reactions in heat aged drilling fluid formulations.

Exclude materials that are known or suspected strong redox (oxidation-reduction) reagents from heataged drilling fluid situations.

#### Inertness of Metal Aging Cells to Chemicals

The greatest degree of inertness to potentially corrosive conditions is provided by the nickel-based alloys, such as the Hastelloy<sup>®</sup> C-276 alloy, Inconel<sup>®</sup> alloy 600 and Incoloy<sup>®</sup> alloy 825 formulations.

Iron may be leached from the various stainless steels by high chloride fluids at high temperatures. This iron removal causes pits and cracks after stainless steel cells are exposed to severe temperature/chloride conditions.

#### Lining Materials in Metal Aging Cells

Some investigators have found that test results from high chloride-content drilling fluids containing watersoluble polymers may not have been reproducible when stainless steel vessels are used. Iron appears to be leached from the cell bodies causing these anomalies. If the more economical stainless steel cells are used under aggressive fluid aging conditions, lining materials may be inserted in the aging cells. Materials which have seen limited to widespread acceptance as liners include: Teflon<sup>®</sup>, glass, and ceramic materials.

It is generally agreed that Teflon liners may be safely used to at least 400°F (190°C) and offer the highest degree of inertness to a wide variety of drilling fluid formulations.

Glass liners may be attacked at the high pH levels used in many drilling fluid formulations. These are also susceptible to attack by certain highly saline conditions at elevated temperatures.

## Metal Plating to Enhance Contamination Resistance of Aging Cells

Plating of stainless steel cells with inert or noble metals, such as gold, have been suggested to avoid the much greater expense of fabricating new cells from premium metal alloys. This otherwise attractive consideration has one serious drawback: if the plated cell is scratched, the exposed steel will experience accelerated severe localized corrosion rates above what unplated steel would experience. Therefore, scratching of plated cells leads to an earlier likelihood of cell leaks and failures.

Since drilling fluid samples aged at elevated temperatures often become very viscous or even hard, it is often necessary to scrape solidified samples from the cells. Despite great care being taken, there is a risk that scratches can occur when removing solidified drilling fluids. Therefore, gold plated cells are not recommended for aging drilling fluids at elevated temperatures.

## Contrast between Drilling Fluid Material Performance in Inert and Real Work Environments

Be cautious about interpreting results from laboratory aging studies using cells which contain inert metallurgy or inert material liners. Drilling fluids in field applications are exposed to the steel in drill pipe, usually made from mild carbon steel.

While the amount of steel surface exposure to drilling fluids is much less than that experienced in steel aging cells, it is still more than would be experienced within inert aging cells.

Therefore, field results can be at variance with the results obtained from aging tests conducted under more ideal inert conditions in the laboratory environment.

# SAFETY AND DESIGN ADVICE FOR THE AGING OF DRILLING FLUID SAMPLES

## Metallurgical Consultants

For best advice on acquiring the proper metals for aging cells required for severe service (elevated chlorides at high temperatures), contact a reputable consulting metallurgist or firm which specializes in metallurgical consulting.

#### Pressure Vessel Consultants

Fann Instrument Company employs qualified Engineers with experience in design and manufacture of pressure vessels for many applications. We can provide assistance with product application, pressure vessel design, drilling fluid aging, or advice on custom products to meet your specifications and situation.