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TECHNIQUES FOR EVALUATING AND IMPROVING THE PERFORMANCE OF THE IRON OXIDES USED AS BARITE SUBSTITUTES IN OIL WELL DRILLING FLUIDS

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THE UNIVERSITY OF OKLAHOMA

GRADUATE COLLEGE

TECHNIQUES FOR EVALUATING AND IMPROVING THE PERFORMANCE OF THE IRON OXIDES USED AS BARITE SUBSTITUTES IN OIL WELL DRILLING FLUIDS

A DISSERTATION

SUBMITTED TO THE GRADUATE FACULTY

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degree of

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By

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TECHNIQUES FOR EVALUATING AND IMPROVING THE PERFORMANCE
OF THE IRON OXIDES USED AS BARITE SUBSTITUTES
IN OIL WELL DRILLING FLUIDS

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TECHNIQUES FOR EVALUATING AND IMPROVING THE PERFORMANCE OF THE IRON OXIDES USED AS BARITE SUBSTITUTES IN OIL WELL DRILLING FLUIDS

ABSTRACT

A large variety of industrial minerals and chemicals is used in the formulation of oil well drilling fluids. Each mineral and each chemical used performs a specific function. Among all minerals used in the oil well drilling industry, the nonmetallic mineral barite leads in tonnage and monetary value. Barite is the main weighting agent for drilling fluids because it has the required advantages of high specific gravity together with chemical inertness, and it is non-abrasive.

There is strong evidence that both the U.S. and the world are facing an imminent squeeze in the availability of mud-grade barite. World reserves are estimated at about 200 million tons that will last only about twenty years. There is a growing dissatisfaction in the oil industry with the available barite even when the mineral meets all current American Petroleum Institute specifications. Barite is frequently contaminated with alkaline soluble carbonate and sulfide minerals that are causing serious problems during drilling operations.

In search for a solution in finding a substitute for barite, this work was designed to evaluate and improve the performance of a selected group of iron oxides in drilling fluids. Iron minerals tested and evaluated in this study are the following:

1. Ilmenite, now on the market under the commercial name of "Bargain." It has an average specific gravity of 4.58, approximately 9 percent greater than that of barite.

2. Itibirite, composed of natural hematite ore, with small amounts of quartz under the commercial name of "Densi-mix." This mineral is derived from the processing of the metamorphic hematite bearing itibirite sediments of Brazil. It has a specific gravity of 5.10, more than 20 percent higher than barite.

3. Synthetic hematite, a biproduct resulting from the processing of various iron oxides and sulphides. This mineral has been produced by the Kerr-McGee Chemical Corporation in small amounts for testing purposes. Electron micrographs indicate that synthetic hematite possesses a sub-spherical particle shape. With a specific gravity of 5.16, the material is more than 22 percent denser than barite.

Specially designed experimental techniques have been implemented together with the standard procedures for testing drilling fluids to evaluate the iron oxide weighted mud types under simulated bottom hole conditions. Some of the problems investigated in this study are:

1. The strong abrasivity associated with the various iron oxides due to their hardness and particle shape, at both low and high shear rates.

2. The unfavorable filtration properties at low and high temperatures exhibited by iron minerals weighted drilling fluids.

3. Possible extensive formation damage that may be caused by iron oxide particles and poor mud cakes.

4. The tendency of some iron oxides to precipitate from the drilling fluid and to form a compact plastic cement.

5. Effect of the use of the various weighting agents on the drilling penetration rates.

6. The use of iron oxides may develop interference with the logging operations due to the ferromagnetic and electromagnetic properties associated with some iron minerals.

7. The quick quality deterioration of drilling fluids weighted with barite as a result of grain attrition.

8. The erroneous rheological behavior of some drilling fluids due to drilling fluid-shale formation interaction. This phenomenon was investigated for six various types of mud formulations using different weighting agents. Performance campatibility tests were conducted on the various mud types using shale samples from a troublesome formation in Oklahoma.

The evaluation of the various drilling mud types revealed the advantages of the iron oxide weighted drilling fluids over the barite weighted system. Some of these advantages are as follows:

1. Nondispersed iron oxide drilling fluids exhibited better rheological performance and higher stabilization characteristics at ambient and high temperature.

2. Iron oxide weighted mud system showed superior rheological and gel strength characteristics at high temperature

that qualifies some mud types for drilling deep holes and geothermal formations.

3. Higher penetration rates were estimated for iron oxide weighted mud types based on drilling fluids properties alone.

4. Iron oxide muds are less reactive with shale cuttings than barite mud and therefore are less liable to cause borehole problems.

5. The mineral grain attrition of the iron oxides is slower and less destructive to mud properties.

6. This study shows evidence that there are prospects for salvage of the iron minerals from the drilling fluids after the well completion. The reuse of this material will make the utilization of iron minerals as weighting agents much more economic than barite.

The role of the mineral hardness, particle size and particle shape at the drill pipe and bit nozzles was investigated thoroughly. Special mud formulation incorporating polymer additives are utilized to either eliminate or reduce the mud wear effect and grain attrition of solids. The polymer additives act as coating agents to the abrasive weighting material and encapsulates the shale cuttings to prevent or minimize hydration effects. This process eliminates the need for intensive chemical treatment and continuous mechanical separation to modify the properties of the aged drilling fluids.

TECHNIQUES FOR EVALUATING AND IMPROVING THE PERFORMANCE OF THE IRON OXIDES USED AS BARITE SUBSTITUTES IN OIL WELL DRILLING FLUIDS

CHAPTER I

INTRODUCTION

The United States' drilling fluids total sales estimated at $3 billion in 1981 indicate a significant increase from the 1980 figure of $2.4 billion. The huge growth rate in the drilling fluids business has become as a result of the increase in drilling activities in the United States. Industry sources estimate that more than one-third of the oil and gas demanded in 1990 will have to be met from reserves which have not yet been discovered. Production of oil is projected to range from 8.3 million to 9.3 million barrels daily in 1985 and to 10.1 million in 1990.[23,27,28,30] According to the American Association of Petroleum Geologists, the fulfillment of these production targets will require an estimated 2.6 billion feet of drilling to maintain a discovery rate of 2 billion barrels per year.

The drilling fuilds used in drilling an oil or gas well is an integral element in the drilling program. The primary purpose of the drilling mud was to remove the cuttings produced during drilling. Today much more is expected from newly developed drilling fluids. Modern technology has added more chemically and physically sophisticated materials and drilling fluids have become more complicated mixtures of liquids, solids and chemicals.

The solids component of modern drilling fluids is composed of a variety of industrial minerals. Drilling fluids can be divided into two main groups: weighted and unweighted. The weighted mud is subdivided into several classes on the basis of specific weight. A drilling fluid with a specific weight of 83 lb/cu ft (11.1 ppg) or more may be considered weighted. Weighted drilling fluids require the use of a weighting material such as barite to achieve the desired weight.

Deep wells require the use of weighted drilling fluids to provide sufficient hydrostatic head to prevent the entry of subsurface fluids into the annulus. Various weighting materials may be used to increase the density of the drilling fluid, depending on the type of fluid used and the final density required.

The nonmetallic industrial mineral barite is now the most popular weighting agent. The amount of weighting material used in drilling fluids varies from zero in unweighted fluids to as much as 700 lb/barrel forming nearly 90 percent by weight of the heavy fluids. The rate of consumption of the weighting material depends on the geopressure and depth of drilling, and on increases for exploration wells in new areas where muds have to be overweighted for safety reasons.

Iron oxides are used occasionally as weighting agents. Natural hematite (Fe_2O_3) was widely used in drilling fluids but was eliminated due to claimed undesirable filtration properties. Its present use has declined to infrequent applications.[24] A heavy iron titanate mineral "ilmenite" has been recently claimed to be a superior barite alternative. The mineral is now on the market under the commercial name "Bargain." Its use so far has been very limited to some specific areas in the U.S. A synthetic hematite with rounded grains is now under investigation. Very small amounts have been produced, so far, for testing purposes. If successful, the mineral will be produced on a large scale for use as a partial or total substitute for barite.

1.1 Statement of the Problem

Published work on the use of iron minerals as weighting agents is scarce. Some work has been done outside the United States, in West Germany and Norway. Oil well drilling service companies in the United States are believed to have done extensive work on ilmenite, but little information was released through commercial brochures and published papers.[50]

The need for a barite substitute attracted the attention of the petroleum industry after the recent increased demand for barite which is likely to continue at least for the next 8 years to 1990. The United States reserves of barite, estimated at 25 million tons are not adequate to handle the cumulative demand of 62 million tons throughout the year 2000.[5,6,8,9,12] World reserves of barite estimated at about 200 million tons will last only for twenty years with 80 percent consumed in drilling mud for oil and gas wells.[11]

The rate of barite consumption in the U.S. drilling industry reached more than 2.50 million tons during the year 1980 and an estimated 2.75 to 3 million tons in 1981. As a result of the strong demand a shortage on the supply of the API grade barite has occurred on the Gulf Coast as well as in the North Sea area. This shortage problem could be even worse in the Middle East and the Arabian Gulf areas where no substantial local barite reserves are available.

Although barite is the overwhelming choice as a weighting agent in drilling fluids, there is a growing dissatisfaction with the material. It is usually contaminated with alkaline-soluble carbonate and sulphide minerals that can cause serious problems during drilling. Because of the large amounts of barite used in the drilling fluids to reduce solids content, even small concentrations of impurities in the mineral can cause significant contamination of the drilling fluids.[10]

Field practice shows that the salvage of barite and its separation from the mud solids is very difficult. Therefore, barite particles usually of silt and sand size will be discarded along with other undesired solids. Only with unusual, expensive maintenance of drilling fluids, can up to 90 percent of the usable barite be salvaged. The tendency of the barite mineral grains to deteriorate towards the finer fractions excludes the host of the barite from the coarser and heavier portions of the treated mud which are returned to the active system. This barite loss must be compensated with addition of the fresh mineral to maintain the mud weight.

The wear effect and grain disintegration of barite adds a considerable colloidal fraction to the drilling fluids weighted with it. They also prevent the possible salvage and reuse of the mineral.

1.2 Purpose of the Study

The objectives of this study can be summarized as follows:

1. To design experimental techniques that can be used to evaluate the performance of drilling fluids weighted with high specific gravity iron minerals.

2. To investigate the problems associated with the use of iron oxides. These problems are claimed by some workers in the petroleum industry to be:

a. Erroneous rheological behavior of the drilling fluids as a result of soluble salts and impurities believed to be associated with iron minerals.

b. Effect of high temperature flocculation on viscosity and gel strength for weighted drilling fluids.

c. The abrasive effect caused by the hard iron oxides (between 5.5 up to 6.5 on the Moh's Scale) on the drilling equipment. The abrasive effect may also effect other mud additives leading to changes in the drilling fluid rheological properties, especially at high temperature and high shear rates. For example, with barite-natural hematite mixture, now on the market, a quick deterioration of barite particles may be expected due to the friction between the sharp, abrasive hematite grains (hardness up to 6.5 on the Moh's Scale) with the soft, tabular barite grains (hardness 3.5 on the Moh's Scale). The gradual increase in the barite colloidal fraction will have a significant effect on the hole cleaning capability of the drilling fluids. The increasing solids content of the fluid was found to decelerate the drilling penetration rate.

d. The electric and magnetic properties of the iron minerals could interfere with the electrical and magnetic well logging instrumentation causing inaccuracy in measurements or even a complete hindrance to the measurements. This effect is attributed to the ferroelectric and ferromagnetic characteristics of the iron oxides. In dealing with this problem it is necessary to measure the magnetic permeability of drilling fluids weighted with various iron minerals.

e. There are doubts in the petroleum industry as to whether these iron oxides will remain in true suspension under static conditions and during the gas cutting process.

f. The extensive formation damage that may be caused by the abrasive iron oxides due to the unfavorable filtrate properties and the unstable mud cakes claimed to be possessed by some iron minerals.

3. To improve the performance and rheology of weighted water-base drilling fluid systems. This requires designing of fluids formulation and treatment techniques which can satisfy the following:

a. The abrasion for iron oxides weighted fluids is reduced to the barite abrasion level.

b. Decelerate the deterioration of barite weighted systems. This will allow for a more efficient use of barite in drilling fluids and reduce the amount of barite consumed per hole.

c. Avoid excessive thickening and high gel strengths that may develop with rising temperature or high shear rates.

4. To develop an adequate experimental laboratory technique for determination of the formulation of a compatible drilling fluid capable of safe drilling through a troublesome formation. This technique must be adequate for measuring the reactivity of the drilling fluid and the specified troublesome formation. Certain types of shales, when exposed to fluids, react in various ways to create different types of instabilities. Highly reactive shales hydrate whereas low reactive types may splinter or fracture. A laboratory study of the drilling fluid-shale interaction together with the characteristics of the

shale cuttings should be carried out before progressing with any further drilling through the troublesome formations. The selection of the most compatible drilling fluid should be based on performance tests under simulated drilling conditions.

5. To study the effect of the grain size distribution of the weighting material on the rheological properties and performance of the drilling fluids. Field practice indicates that the study of the particle size distribution is an accurate means of checking the operating characteristics and efficiency of the solids control equipment. Size analysis are also useful in monitoring characteristics of the drilling fluids and whether the same mud system can be used for a prolonged period of time. It would indicate the rate of the build up of the micron and submicron size particles that may cause complete deterioration of the mud properties.

6. To examine the effect of the grain shape of the weighting agents on the mud abrasivity. There are various methods for examining the grain shape. The most accurate and informative is the scanning electron microscope. Two dimensional and three dimensional electron micrographs can be produced at various scales and used as a clear and convenient way to study the grain shape, and its dynamic change with the progressive abrasion. This study may disclose information that can be used to develop a technique to produce rounded, low abrasive mineral grains.

1.3 Scope of the Study and Program Plan

To accomplish the stated purposes of this study a number of experiments had to be performed. Each experiment has its own purpose and technique. Some tests were conducted according to standardized testing procedures as recommended by the American Petroleum Institute. Many of these tests are covered in the API Code RP 13B, 1978. The main task of these tests is to evaluate the drilling fluids by measuring certain properties of the mud and mud filtrate.

Other testing techniques were developed for this particular study. Engineering manuals of Magcobar, 1979, IMCO Services, 1978, and AMOCO Production, 1978, were found very helpful in combining and developing test techniques that simulates the appropriate field conditions.

The experimental study can be divided into the following:

1.3.1 Mud Formulation and Preparation

Batches of water base mud were prepared in a manner that can serve the correlation between the weighting agents under investigation. All muds had the same components with the exception of the weighting agents. Each mud batch was weighted with one of the three iron minerals under investigation with barite as a control. No chemical additives were used, unless necessary to adjust a specific property that might effect the correlation results. Percentages of the mud components were obtained from standard charts and percentages of weighting materials were obtained from charts especially designed for each mineral. The fluid batches were mixed simultaneously in the laboratory using a Magcobar multimixer. The pH values were adjusted between 9-11 to reduce the tendency of fresh water clays to flocculate. Flocculation of clays usually occurs due to salt and calcium contaminations. A relatively high hydroxyl ion content limits the solubility of calcium and other contaminants that may cause the fresh water mud to thicken. Very small amounts of caustic soda were occasionally added to make the pH value adjustments. A core lab pH meter model 125 was used for measuring the pH value.

Rheological properties of the mud were measured by the six speed Fann model 35 viscometer. The gear system in this viscometer allows the operator to use six different speeds (3, 6, 100, 200, 300 and 600 rpm) for shear stress measurement. Because the dial readings can vary markedly with temperature changes, it was observed that readings for various muds were taken at the same temperature in order to obtain accurate correlations. Rheological properties, including plastic viscosity in centipoise, yield point, gel strength in lb/100 ft^2, were measured. Due to variation of the viscosity of non-Newtonian fluids with shear-stress/shear rate ratio, it was most accurate to use the effective viscosity values as estimated at different parts of the circulating system.

1.3.2 High Temperature Rheological Measurements

The surface measurements of viscosity does not reflect the rheological properties of the drilling fluids at well bore temperature and pressure. Testing of the viscous properties under the actual well bore conditions can be performed by the Fann 50B high temperature high pressure viscometer.

The viscous characteristics of mud systems weighted with synthetic hematite, ilmenite, natural hematite and a mixture of ilmenite and natural hematite were measured. A barite weighted mud batch was also tested under the same conditions. The drilling fluids samples were heated from ambient temperature up to 400°F and then cooled back to ambient temperature. A back pressure of 1000 psi is usually applied to prevent boiling of drilling fluids samples. The testing procedure consists of filling a pressure rated sample chamber with the fluid under investigation and immersing a bob sleeve assembly into the sample. The whole sample chamber is then submerged in an oil bath and the following steps are taken:

1. Determining the initial viscous properties at ambient temperature at each shear rate,

2. Recording the shear-stress readings at 300 rpm shear rate continuously as the sample is gradually heated,

3. Determining the viscous properties by taking the shear stress readings at each shear rate,

4. Taking shear stress readings at 300 rpm shear rate while the sample is gradually cooled to ambient temperature,

5. Another set of readings is taken at each shear rate available on the viscometer.

A plot of the apparent viscosity as a function of temperature enables determination of the viscous properties of the drilling fluids under high temperature. The plastic viscosity and yield point of each mud at specified points in the circulating system can be determined. The test also gives the stability limits for the undispersed iron oxides and barite weighted systems.

1.3.3. Measurement of the Electric and Magnetic Properties

In electric well logging resistivity methods, the apparent resistivity R_a is measured by the use of focusing electrode tools. These tools include the laterolog and the spherically focused log. Field measurements are always influenced by the resistivities and geometrical dimensions of all the media around the device including bore hole, drilling fuilds, invaded zones and adjacent beds.

When the laterolog tools and spherically focused logs (shown in Appendix D) are utilized, the mud resistivity value R_m becomes a very important parameter and must be controlled so that the ratio R_t/R_m is sufficiently large to allow for an accurate measurement of the rock's true resistivity R_t. The well logging tools become reliable only when R_m is small; therefore it is necessary to control the mud resistivity to obtain valid results especially when dealing with low resistivity formations.

Another important parameter in well logging operations is the resistivity of the mud filtrate (R_{xo}). A correlation between the mud filtrate resistivity and the true formation resistivity is an oil exploration tool which can indicate hydrocarbon mobility. Where the mud filtrate invasion is quite deep, R_{xo} values can be used to obtain better estimates of the true formation resistivity.

Resistivities of mud and mud filterate also effect the results of the spontaneous potential (S_p), which is the measurement of the electrical potential between a point in the borehole and a grounded electrode at the surface. The S_p cannot be recorded in holes filled with nonconductive muds, because such muds do not provide electrical continuity between the S_p electrode and formation. If the resistivities of the mud filtrate and connate water are about equal, the S_p deflections will be small and the well logging curve will be featureless.

In designing a new drilling fluids formulation, it has to be noted that well logging measurements are always taken with the hole filled with drilling fluids. The fluids have electrical and magnetic properties depending on their components and additives. The influence of these substances on the measurements must be known, and control methods should be available while drilling.

In this study two experiments were performed to investigate the electric and magnetic properties of the iron minerals weighted muds. The first experiment deals with the resistivities of the various iron mineral weighted muds and the mud filtrate invaded rocks. Results were correlated with those obtained for barite weighted muds having the same mud weight and formulation except for the weighting material. All four mud systems (3 different iron mineral weighted systems and a barite weighted system) were filtered through berea sandstone in an evacuated system at the same time. The resistivities were measured precisely using a Fann resistivity meter model 88-A (shown in Appendix D). The resistivity test unit is capable of measuring resistances with an accuracy of 0.001 ohms. Combined with a resistivity cell with a dip cell constant c = 0.01 meter, the mud resistivities were measured to 0.001 ohm, m^2/m. A special core holder (shown in Figure 62) was used to measure the mud filtrate saturated berea sandstone. The standard method for measurement was followed as explained in the Core Laboratories Inc. operating manual (Code 5810-030).

The second experiment was designed to investigate the electromagnetic characteristics of the iron minerals weighted fluids and to compare it with those of the barite weighted systems. The experiment is based on producing a magnetic field in the drilling fluid contained in a circular toroid. The toroid is made of hard plastic tube with a uniform winding of many turns of thin wire carrying an electric current. The magnetic lines of flux are almost entirely confined to the interior of the winding and therefore to the inside medium. The wire terminals were connected to a Q meter (Appendix D) which is capable of applying an electric current and measuring the inductance. The value of the inductance depends on the electromagnetic properties of the medium inside the toroid. Different values for inductance in henrys can be obtained with different material depending on their magnetic permeability values. A plot between the capacity C in picofarad versus $1/f^2$ where f = frequency in kilocycle can be used to determine the magnetic permeability for each individual mud system. These measurements indicate the strength of the magnetic effects that can be expected from each type of drilling fluid as a result of the electric current applied on the mud by the electric and magnetic logging tools. If these effects are strong as the case with the ferromagnetic material, a strong interacting magnetic and/or electric interference with the well logging operations will inevitably occur.

1.3.4. Dynamic Aging by Rolling Technique

In the investigation of performance properties of drilling fluids and the evaluation of the mud additives, it is important to simulate actual drilling conditions as closely as possible. The roller oven manufactured by NL-Baroid provides an excellent method of simulating the heating and agitation the mud experiences during circulation down the hole and back to the surface. A mud contained in a high temperature aging cell can be subjected to drill pipe heat and agitation conditions by adjusting the thermostat of the roller over to the desired temperature and operate the power driven rollers.

1.3.5. Abrasion Tests

Drilling muds weighted with iron minerals are usually more abrasive than those weighted with barites. The abrasivity varies with the hardness, size and shape of the mineral grains. Two different techniques have been adopted in this study for measuring abrasivity. Each method simulates a specific part of the drilling circulating mud system.

a. Abrasion caused by iron oxides weighting agents at drill pipe.

Rolling techniques at temperatures varying from 150-250°F were applied to the abrasive drilling fluids systems. A barite weighted mud was also used as a control. The drilling fluids were placed into a special mud cell provided with a coupon fixed along the axis of the cylindrical cell. The abrasion was measured in weight loss percent. The specially designed mud cell and coupon may be seen in Appendix D.

b. Abrasion under simulated bottom hole conditions.

This part of the drilling system is characterized by high shear and high temperature. The shear rate is believed to vary from 10,000 up to 100,000 rpm. Simulation was achieved by using a high speed Waring blender with a shear rate of approximately 20,000 rpm and an Osterizer blender of approximately 15,000 rpm shear rate. Both types have the advantage of possessing removable electrically insulated blades. Temperature simulation was achieved by preheating the drilling fluid. Heat produced during stirring and planning of stirring periods were adequate in keeping the temperature between 140°-180°F. The abrasion rate was measured in weight loss percent. Weight losses were measured using an electronic analytical balance (mettler H_{35}) with an accuracy of ± 0.0002 grams.

1.3.6. Tests for Mineral Grain Attrition

Weighting agents made of iron oxides are composed of hard minerals which will have less grain to grain attrition than the soft barite particles during use. By combining rolling technique and high shear tests the whole circulating system can be simulated. For this purpose drilling fluids weighted with various types of weighting agents were examined for particle size distribution and particle shape. Size distribution was examined using fine sieve and hydrometer analysis whereas the particle shape was examined by the Scanning Electron microscope. The rheological properties of each mud were determined then the mud was subjected to a high shear aging process for 80 minutes. The aged fluids were then placed in the mud cells and aged for 14 days in the roller oven. After being cooled down to room temperature, the weighted drilling fluids were tested for particle size distribution and grain shape. A correlation between shapes and particle size distributions before and after the combined aging process should give a good indication about changes in particle size and shape as a result of abrasion and grain to grain attrition. Soft weighting material like barite, galena and micaceous hematite produce more fines under borehole conditions than does the weighting material composed of hard iron minerals. The produced fines tend to increase the mud viscosity and cause deterioration of the mud quality. Therefore these fines are usually eliminated from the active mud system.

1.3.7. Coating and Lubrication Techniques

The main purposes for using this technique are:

a. To provide a soft coating for hard iron mineral grains to cut down abrasivity, and
b. To decelerate the progressive comminution of mud additives and weighting agents made of soft material such as barite and galena.

Any lubricant or coating material for use in drilling fluids must be uniformly dispersed or distributed in the water phase. It should be composed of material that would not change the rheological properties of the mud significantly and would not add much to the solids content. The desired material must be stable and effective at high temperature and under high shear rate conditions. A number of substances have been tried in this study. The most effective were found to be; Drispac polymer made by Drilling Specialities Co., H-42-10-4 water soluble polymer and LO-SOL polymer, both manufactured by AMOCO Chemicals Inc. The polymers were added in small amounts, less than 2 percent by volume. The drilling mud had to be reformulated by adding small amounts of thinners like Desco and/or Barafos.

1.3.8 Compatibility Performance Tests

The tests are used to determine the effects of drilling fluids—shale interaction on the mud properties and to ensure the success of the laboratory formulations. The test is developed to suit the purpose of this study and has been conducted in the following manner.

The test was applied on core samples obtained at approximately 11,400 foot depth from drilled hole, Shell 1, Dipple in eastern Oklahoma (see Appendix D for location). A 50 gram of shale cuttings between 4 mesh and 10 mesh screen was used for each test. Drilling fluids formulated for all the individual iron oxides and their mixtures were tested. The cuttings together with the fluids were placed in the laboratory barrels after the initial properties of the muds had been determined. The samples were then aged in the roller oven under dynamic conditions at the desired formation temperature of 200°F for 24 hours. After the samples were removed from the roller oven the cuttings were screened over a 30 mesh screen and weighted to determined the percentage of shale recovered. Comparisons of the Methylene blue testing results of the cuttings were made before and after the tests. Comparatively they indicate the degree of which the cuttings are effected by the various drilling fluids. A measurement of the rheological properties after the test will indicate the changes in mud performance and type of treatment required. The pH value was also meausred and adjusted with minimum changes in mud properties.

A new fresh sample of shale was then mixed with each drilling fluid and the whole test was repeated. The test was carried out for seven days after which the filtration properties together with the final methylene blue capacity (MBT) and pH values were determined. A comparison between the initial and final mud properties gives an excellent indication about the drilling fluids compatibility performance. It also gives information about the necessary mechanical and chemical treatment for each mud and the expected changes during its surface and downhole circulation. The test provides valuable technical data for planning of a drilling fluids program. The test can be adapted to various types of formations under variable drilling conditions.

CHAPTER II

DRILLING FLUIDS TECHNOLOGY

Drilling fluids are defined as materials employed to aid drilling tools in the creation of a bore-hole. The American Petroleum Institute defines a drilling fluid as "A circulating fluid used in rotary drilling to perform any or all of the various functions required in the drilling operation."

2.1. Functions of Drilling Fluids

By definition, the main functions of a successful drilling fluid is to facilitate a speedy, safe and satisfactory completion of the well. To achieve this goal, the drilling fluid must be capable of achieving the following:

1. Cooling and lubrication of the bit, drill pipe and drill coller.

2. Minimize the friction losses and maximize the available hydraulic horse power.

3. Removal of cuttings from the hole.

4. Cleaning of the bottom of the hole.

5. Releasing cuttings at the surface after being suspended and removed from the wellbore.

6. Control formation pressure by preventing the influx of formation fluids in the wellbore without creating excessive differential pressure.

7. Forming an impermeable filtercake on the walls of the borehole which can wall off permeable formations and minimize the formation damage caused by the drilling fluids.

8. Stabilizing the wellbore and preventing caving in of the formations.

9. Aiding in supporting the weight of the drill string and casing.

10. Reducing the cost of the casing.

11. Preventing corrosion fatigue of drillpipe.

12. Allowing interpretation of electric logs by having an appropriate resistivity value and not interfering with the log tools.

13. Suspension of cuttings and weighting materials on stopping the circulation.

14. Performing the above functions without creating any hazard to the crew.

2.2. Factors Influencing the Functions of the Drilling Fluids

2.2.1. Annular and Slip Velocities

One of the most important and primary functions of drilling fluids is the removal of cuttings from the borehole. In order for the cuttings to reach the surface, the slip velocity must be lower than the average annular velocity. The annular velocity is a function of borehole size and condition, pump output, and drillpipe and drillcollar sizes.

$$\text{Annular velocity (ft/min)},\ V_a = \frac{\text{pump output (bbl/min x 100)}}{\text{annular volume (bbl/100 ft)}}$$

The following equations can be used to obtain slip velocities V_s in ft/sec., depending on the type of flow in the annulus (Tschirly 1978).

$$\text{Laminar flow } V_s = \frac{53.5\ (\nu_s - \nu_f) d_s V_a}{6.65\ YP(d_b - d_p) + V_a PV}$$

$$\text{Turbulent flow } V_s = \frac{d_s (\nu_s - \nu_f)}{\nu_f}$$

where ν_s = specific weight of the cutting (lb/gal)

ν_f = specific weight of drilling fluid (lb/gal)

V_a = average annular fluid velocity (ft/sec)

d_s = average cuttings diameter (inches)

YP = yield point (lb/100 sq ft)

d_b = bore diameter or bit size (inches)

d_p = drillpipe OD (inches)

PV = plastic viscosity of drilling fluid (Cp)

According to Moore, 1974, the slip velocity V_s can be determined using drilling fluids parameters from the following equation:

$$V_s = \frac{175\ d_s (\nu_s - \nu_f)^{0.667}}{(\nu_f\ \mu_f)^{0.333}}$$

where d_s = particle diameter (inches)

μ_f = fluid viscosity (Cp)

The Reynolds number R_e which determines the type of flow may be calculated from the following equation.

$$R_e = \frac{15.47\ \nu_f\ \nu_s\ \ d_s}{\mu_f}$$

For Reynolds numbers greater than 2000, the flow is turbulent, the slip velocity can be calculated from the following equation.

$$v_s = 113.4\ [\frac{d_s(\nu_s - \nu_f)}{C_D \nu_f}]^{\frac{1}{2}}$$

where C_D = drag coefficient.

The above equations indicate that to maintain proper hole cleaning, the annular velocity, yield value and gel strengths of the circulating drilling fluids should be maintained at the appropriate values. The effect of the viscosity and gel strength is represented graphically in Figure 1.

2.2.2. Subsurface Pressure Balance

The specific weight of the drilling fluids has a vital role in increasing the lifting capacity. The particle recovery is much higher in the case of heavier drilling fluid at the same circulation rate. This is illustrated in Figure 2. But the greatest importance of the specific weight of the drilling fluids is to establish a balanced drilling condition. Balanced conditions are provided when the hydrostatic head provided by the drilling fluid balance the formation pore

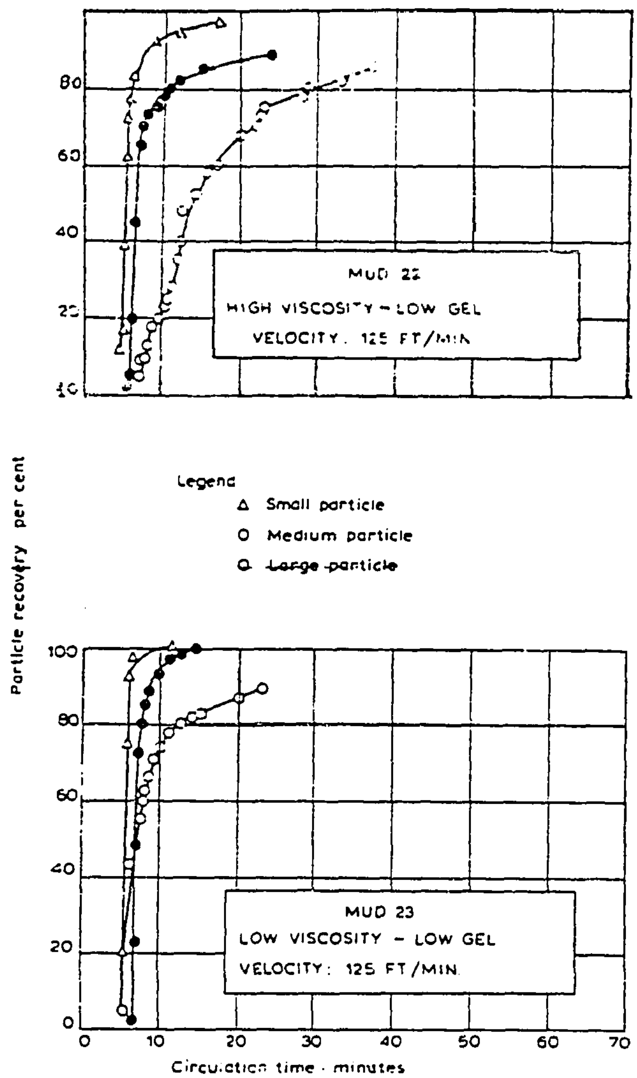


Fig. 1. Effect of viscosity and gel strength on the function of the drilling fluids (Chilingarian and Vorabutr, 1981).

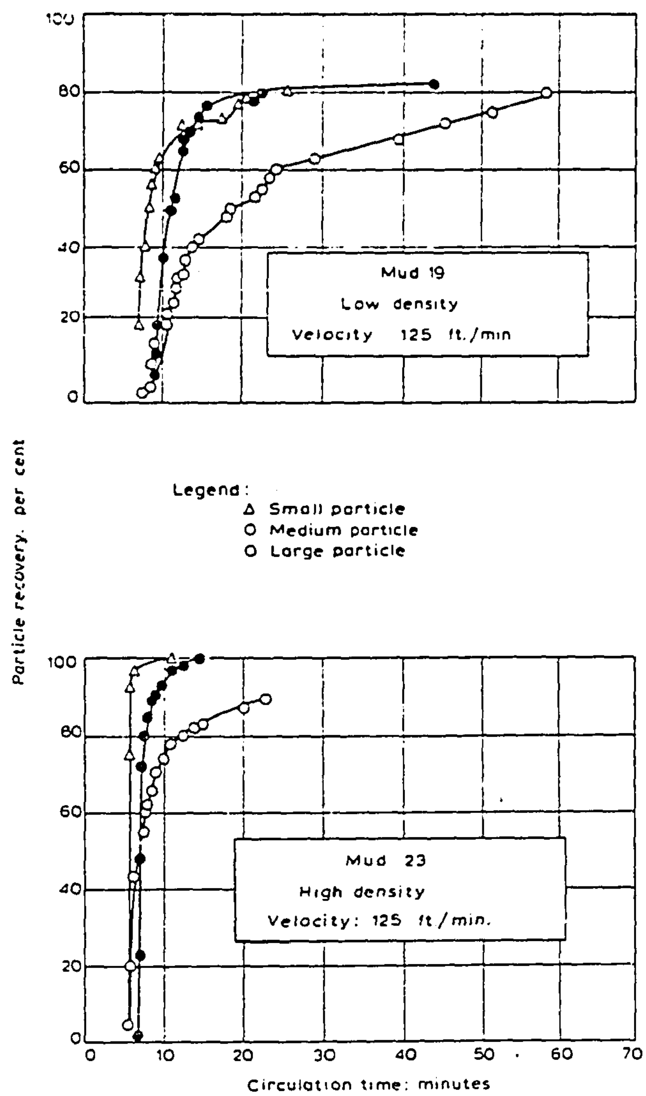


Fig. 2. Effect of the specific weight on the function of the drilling fluids (Chilingarian and Vorabutr, 1981).

pressure and restricts the influx of formation fluids into the wellbore. A control of the subsurface pressure by the drilling fluid can be explained mathematically as follows:

$$P_o = D[(1 - \emptyset)\nu_{rm} + \emptyset\ \nu_{if}]$$

where P_o = overburden pressure (lb/sq. ft)

D_g = depth of the geologic column

$\emptyset$ = porosity (fraction)

ν_{rm} = specific weight of the rock matrix (lb/cu ft)

ν_{if} = specific weight of the interstitial fluids (lb/cu ft)

In selecting the specific weight of the drilling fluids, several factors have to be considered.[18] The most important are: (1) the fracture gradient, (2) pore pressure, (3) kick tolerance, (4) casing-shoe depth, (5) borehole stability, (6) surface pressure control equipment, (7) annular circulating velocity, (8) pressure surges, (9) magnitude and rate of filteration of drilling fluids, (10) gas cutting of drilling fluids, (11) drilling rate, (12) formation porosity and permeability, (13) safety margin, and (14) reservoir data including formation damage and electric log interpretation. The subsurface pressure concept is represented graphically after Fertl and Chilingarian, 1977, in Figure 3. The hydrostatic pressure that a column of drilling fluid exerts at any depth in the hole can be calculated from the formula: hydrostatic pressure (psi) = (0.052) x depth (ft) x drilling fluids weight (lb/gal).

2.2.3. Drilling Rate

Drilling fluid properties are among the important factors effecting rate of penetration properties found to be the most important based on laboratory and field studies are the specific weight, viscosity, filterate loss, solids content and oil content. The drilling fluid must have these properties adjusted in order to allow for optimum penetration rate. The density has to be the lowest possible, minimal solids content, high spurt loss and optimum flow properties.

2.2.4. Hole Stability

In addition to other factors, the chemical composition of the drilling fluids effects the hole stability; therefore the mud should be formulated in order that its chemical composition filtrate causes minimum damage to water sensitive formations.

2.2.5. Formation Evaluation

Damage caused by drilling fluids to the pay zones effects the well testing and the interpretation of the characteristics and potential of the producing zones. Therefore the drilling fluids must be of low solids content and appropriate chemical composition before drilling the productive zones.

2.2.6. Rig Equipment Selection

The rig and the associated equipment should be selected to function property with the drilling fluids program. All necessary solids removal and circulating equipment must be readily available and in good working condition.

2.3. Borehole Problems Associated with Drilling Fluids

The common wellbore problems are due mainly to instability caused by sloughing and heaving shales.[18,24,29] Shales are composed of many different minerals such as quartz, feldspar, dolomite, calcite, siderite, and gypsum. These minerals are forming the inert fraction of the shale. The reactive fraction is formed of kaolinite, illite, chlorite, montmorillonite and mixed layer clays. This fraction contributes to the swelling, heaving and caving. The heaving problem is partly chemical and partly mechanical. Among the important variables effecting both the chemical and mechanical functions influencing shale are the drilling fluids properties. The properties having direct bearing on hole stability are the following:

a. Mud Weight

Insufficient mud weight will cause pressured shale to explode in the well bore especially in geopressure areas or abnormally pressured areas. Geopressures may have any value up to the total weight of the overburden and the density of the mud must be increased accordingly (see Figure 4). Unstable shale formations usually contain large fractions of reactive clay minerals. Therefore their swelling tendency must be controlled by adding special chemicals to the mud system.

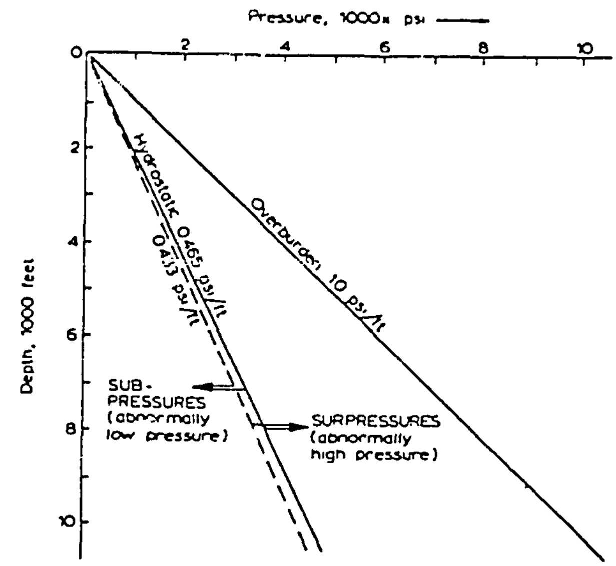


Fig. 3. Graphical presentation of the subsurface pressure concept (After Fertl and Chilingarian, 1977).

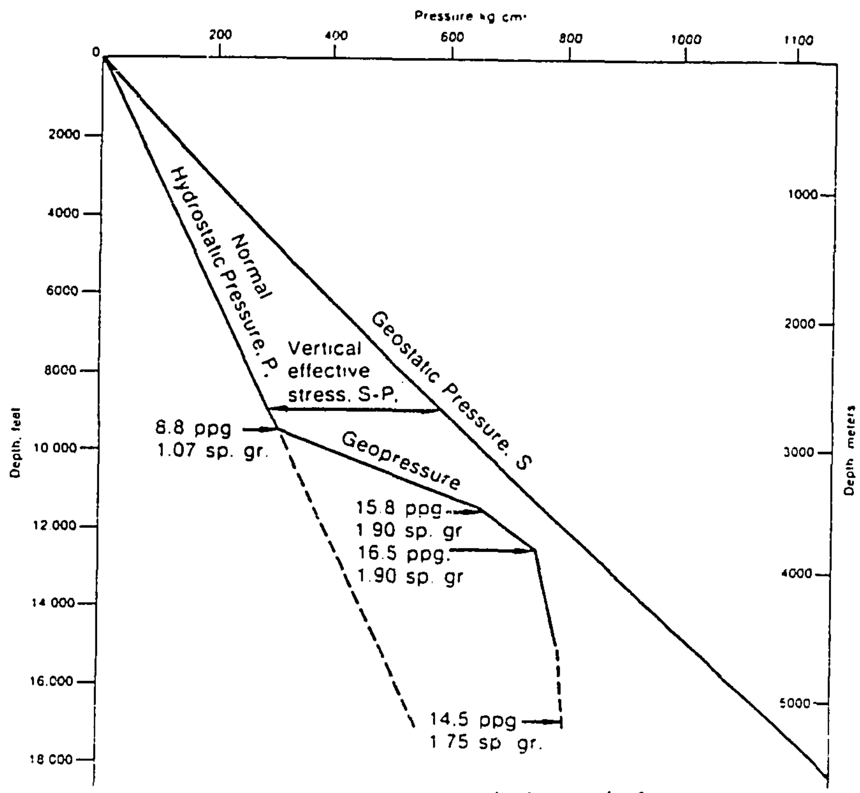


Fig. 4. Increase of mud weight with geopressure (After Grey and Darly, 1981).

b. Mud Viscosity

The mud viscosity must be controlled by chemical and mechanical means. In order to maintain the flow profile in laminar condition, the yield value and circulation rate must be properly adjusted to minimize hole erosion. Turbulent flow usually causes hole enlargement and should be avoided when circulating the drilling fluids.

c. Filtration Properties

The chemistry of the drilling fluid filtrates and adsorption ability of the clays forming the shales are the most significant factors in shale stabilization. Chemical inhibition may be necessary in many cases to prevent the clay hydration and their dispersion in the mud system.

The hole problems can be avoided by the proper selection of drilling fluids and conditioning of these fluids in order to improve their performance and reliability. In this study long duration tests of performance compatibility have been conducted under dynamic bottom hole conditions. These tests investigate the capabilities and limitations of the iron minerals weighted drilling fluids to perform under unstable hole conditions. Drilling fluids-shale interaction tests give the basic techniques for mud selection and treatment desired for the design of a drilling fluids program.

2.4. Classification of Drilling Fluids

2.4.1. Classification According to Base Fluid

Drilling fluids are classified into three main groups according to their base fluid.

1. Water base drilling fluids
2. Oil base drilling fluids
3. Gaseous drilling fluids

Each group has its own physical and chemical properties that enable it to work under specific well conditions. The various groups cover all varieties of well conditions including deep and overpressured wells. All types of drilling fluid must be weighted to provide sufficient hydrostatic head in order to confine formation fluids to their native formations. Different types of basic mud and conditioned muds can be seen in Figure 5.

Types of drilling fluids are a function of composition which are also reflected in the mud properties. Clay content of the drilling fluids imparts many of the important properties like viscosity and influences the thixotropic characteristics of the drilling fluid. Different types of clay minerals have various effects on the fresh water drilling fluids. Montmorrilonite clays disintegrate in water solution into small particles less than 0.1 micrometer in diameter leading to good swelling property which increases viscosity. Koalinite clays do not swell much and are not active because of their low base exchange capacity. The degree of swelling in clay minerals depends on the type and positions of cations. Depending on the type of forces between the clay particles, there are four different states: (1) depersed-defloculated, (2) dispersed-flocculated, (3) aggregated-defloculated and (4) aggregated-flocculated.

The state of the suspension of the clay determines the increase or decrease in surface area and thus controls the plastic viscosity, yield point and gel strength. It also influences the volume of the filtrate loss, thickness and permeability of the filter cake of the mud.[18,24]

2.4.2 Classification of Drilling Fluids on the Basis of Specific Weight

Depending on their specific weights, drilling fluids are divided to weighted and unweighted. Ranges in specific weights of various drilling fluid types are shown in Figure 6.

Unweighted drilling fluids include solids free fluids, (which contain polymers instead of benonite) and water in oil or oil in water emulsions. Weighted drilling fluids are used to drill deep wells to provide sufficient hydrostatic head. They are usually weighted with various weighting material, the most common is the nonmetallic mineral "barite." In this research natural widespread heavy minerals including itibirite (natural hematite and silica), ilmenite and their mixtures will be used as the main components for weighted mud. A synthetic iron oxide will be also used as a partial or full replacement for barite.

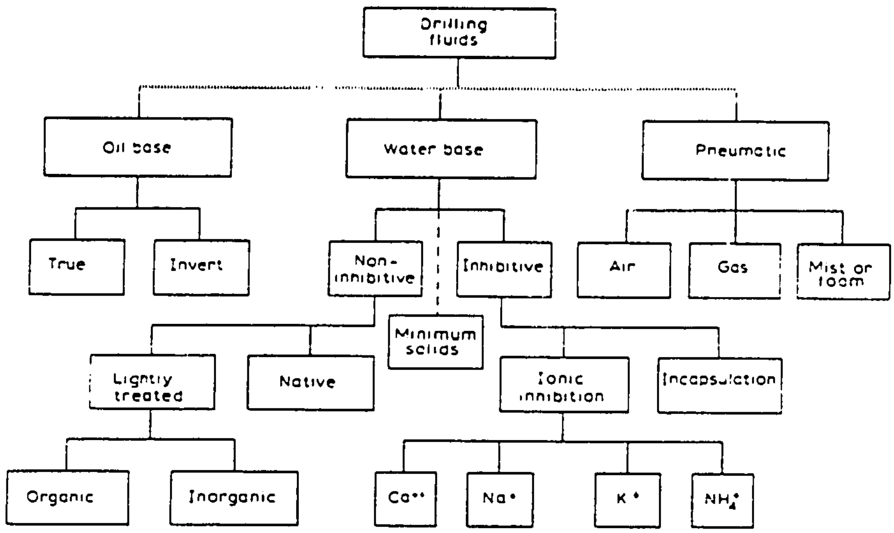


Fig. 5. Classification of drilling fluids according to their base fluid (Courtesy of IMCO Petroleum Service).

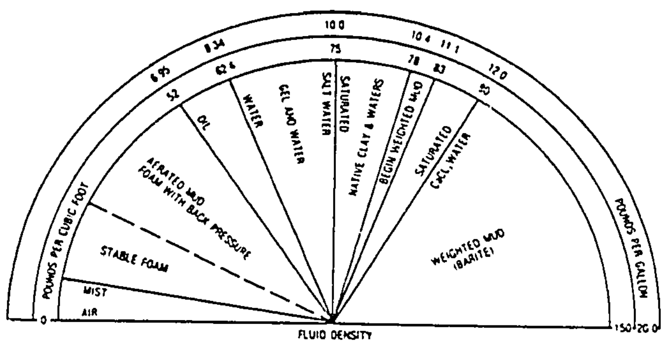


Fig. 6. Classification of drilling fluids on the basis of specific weight (Courtesy of Baroid Pet. Services).

CHAPTER III

RHEOLOGY OF DRILLING FLUIDS

The rheology of drilling fluids primarily deals with the relationship between flow pressure and flow rate and eventually the flow characteristics of the fluid. The flow behavior of the drilling fluid in a borehole can be, like any other fluid, either laminar flow or turbulent flow.[18,25,29] Under laminar flow conditions the pressure-velocity relationship is a function of the viscous properties whereas in turbulent flow it is usually determined by empirical equations governed by the inertial properties of the fluid.

In describing the flow behavior and characteristics of the drilling fluids four models are involved.

3.1. The Newtonian Model

The most important concept of this model is that a Newtonian fluid has a constant viscosity at all shear rates. The flow obeys the mathematical straight line relationship.

$$\tau - \tau_o = - \mu p \frac{dv}{dr}$$

where τ is the shear stress, τ_o = is the stress required to initiate flow.

$\frac{dv}{dr} = \nu$ is the shear rate

and μp is the viscosity.

3.2. The Bingham Plastic Model

3.2.1. Bingham Plastic Fluids

These are fluids with a yield point and plastic viscosity that can be obtained from the intercept and slope of a line drawn through the shear stress measurement at 600 and 300 rpm on the standard viscometer. The model is represented by the equation

$$\tau = - \mu p \frac{dv}{dr} + \tau_o$$

μp is the plastic viscosity

τ_o is the yield stress

This model provides a practical method of defining and measuring fluid flow parameters using the rotational viscometer. The following simple formulas, present by Savins and Roper in 1954, are used for the calculation of plastic viscosity μp and yield point YP.

$PV = \mu p = R_{600} - R_{300}$

$YP = \tau_o = R_{300} - \mu p$

The effective viscosity at a specified shear rate is defined as the viscosity of a Newtonian fluid that exhibits the same shear stress at the same shear rate. It can be calculated at a shear rate ν_1 from the equation

$$\mu_{e1} = \frac{\tau_1 - \tau_o}{\nu_1} + \frac{\tau_o}{\nu_1} = \mu p + \frac{\tau_o}{\nu_1}$$

The major disadvantages of using the Bingham plastic model are:

1. It describes the fluid flow over a range of 511 to 1022 sec^{-1} only, whereas the shear rate in actual drilling situations varies from 1-3 sec^{-1} at the pits, 5-170 sec^{-1} at annulus and from 170-10,000 sec^{-1} at the solid removal equipment and drillpipe.

2. The Bingham plastic model fails to describe the flow at the bit and bit nozzle which ranges from 10,000 to 1,000,000 sec^{-1} shear rate.

3. The model may not accurately describe fluid rheological characteristics in all drilling situations.

4. Drilling fuilds are not ideal Bingham plastics and they deviate from linearity at low shear rates. This becomes obvious when using a multispeed viscometer where curves become linear only at considerably high shear rate. The actual yield point calculated from a multispeed viscometer is well below that indicated by the extrapolation of the 600 and 300 rpm (shear rate) readings. Extrapolation of the mud viscosities at various parts of the circulating system was performed on the appropriate logarithmic forms.

3.2.2. Thixotropic Drilling Fluids

A thixotropic mud will not flow unless the applied stress is greater than the gel strength. A stress magnitude equivalent to the gel strength must be applied to the mud to recirculate it. Thixotropic fluids have the following characteristics:

1. The viscosity of the thixotropic fluid depends on time of shearing and rate of shear. This is different from the viscosity of a Bingham plastic which depends on the shear rate.

2. The structural component changes with time according to the past shear history of the fluid.

3. Bingham plastics may or may not be thixotropic depending on the composition and electrochemical composition.

4. High gel strengths developed by thixotropic muds after prolonged periods of rest create another problem for the drilling engineer.[29] The long term gel strength is a major factor in the pressure required to break circulation after a round trip and in magnitudes of swap and surg pressures.

When measurements of long term gel strengths were conducted in this research as part of the rheological tests, the following measures were observed.

1. The mud was sheared under constant shear rate until equilibrium is reached.

2. The prepared mud batches were mixed at the same time and left overnight before the rheological parameters were measured.

3. Ilmenite weighted mud showed a rheopetic behavior as soon as it was mixed. When left overnight, the viscosity changed significantly and a thixotropic behavior replaced the earlier anomolous rheopexic property.

3.3. Power Law Model (Pseudoplastic Fluids)

The power law accurately describes the annular flow of a drilling fluid. The shear rate range of 5-170 sec^{-1} (3-100 rpm) encountered in the annulus of a wellbore can be described by a straight line relationship on a logarithmic scale.[18,29]

The main characteristics of a pseudoplastic fluid obeying the power law are:

1. It has no yield point.

2. A typical pseudoplastic fluid, a suspension of long chain polymer, tend to allign the suspended particles parrallel to the direction of flow.

The power law model is represented mathematically by the empirical formula

$$\tau = K\left(\frac{dv}{dr}\right)^n$$

K and n are constants which characterize the flow behavior of the fluid. K is called the consistency index ($dynes/cm^2$). n indicates the degree of departure from Newtonian behavior, known as the flow behavior index. When $n < 1$ the fluid is pseudoplastic and the effective viscosity decrease with shear rate. For Newtonian fluids $n = 1$ indicating no change with shear rate. In a dilatant fluid, $n > 1$ showing an increase of viscosity with shear rate.

The effective viscosity of a power law fluid U_e(poises) is obtained from the equation

$$U_e = \frac{\tau}{\nu} = GK(\nu)^{n-1}$$

where $\nu = (\frac{dv}{dr})$ = shear rate (sec^{-1})

τ = shear stress ($dynes/cm^2$)

The flow parameters n and K can be determined using a direct indicating viscometer from the formula

$$n = \frac{\log 0600 - \log 0300}{\log 1022 - \log 511} = 3.32 \log 0600/0300$$

$$K = 0600/(1022)^n \ lb/100 \ ft^2$$

where 0600 and 0300 are the reading of the shear stress at 300 and 600 rpm shear rates.

3.4. The Generalized Power Law Model

The flow behavior of most drilling fluids is intermediate between the Bingham plastic and the power law (pseudoplastic) flow models. Plots of multispeed viscometer data were found to deviate from the typical straight line logarithmic relationship at low shear rates. Low-solid and polymer fluids, heavily treated muds and oil base muds tend to follow the pseudoplastic model. High solid muds, untreated and flocculated muds act more like Bingham plastics.[29]

The generalized power law was developed by Robinowitch and Mooney to cover the flow behavior of these diverse fluids. The flow characteristics were defined by the ratio of the shear stress at the wall of the pipe to the shear rate at the wall of the pipe. The following equation was developed by Metzner and Reed to represent the generalized formula.

$$-\left(\frac{dv}{dr}\right)_w = \frac{3n' + 1}{4n'} \quad \frac{8v}{D}$$

where $\left(-\frac{dv}{dr}\right)_w = \nu_w$ is the shear rate at the wall and the model parameters can be obtained from

$$n' = \frac{d\left(\log \frac{DP}{4L_d}\right)}{d\left(\log \frac{8V}{D}\right)}$$

V = volume of flow

D = borehole diameter

P = pressure

L_d = depth

To find the constant K' the following formula may be used

$$K' = \left(\frac{3n + 1}{4n}\right)^n K$$

where n' is numerically equal to n in the power law. The generalized power law model then becomes

$$\tau_w = \frac{DP}{4L} = K'\left(\frac{8v}{D}\right)^{n'}$$

In terms of K and n the equation may be written in the form

$$\tau_w = K \left(\frac{3n + 1}{4n}\right)^n \left(\frac{8v}{D}\right)^n$$

where $\frac{8v}{D}$ is the shear rate in sec^{-1} and $\frac{D\Delta P}{4L}$ represent the shear stress in lb/ft^2. The effective viscosity of the drilling fluids at the drill pipe can be calculated in terms of flow parameter from the formula.[29,56]

$$U_e = \frac{\tau_w}{\gamma_w} = \frac{K\left(\frac{3n+1}{4n}\right)^n \left(\frac{8v}{D}\right)^n}{\frac{3n+1}{4n} \quad \frac{8v}{D}} = K\left(\frac{3n+1}{4n}\right)^{n-1}\left(\frac{8v}{D}\right)^{n-1}$$

or in the form

$$U_e = K\left(\frac{2n+1}{3n}\right)^{n-1}\left(\frac{12v}{D_2-D_1}\right)^{n-1}$$

The generalized power law model is adopted in all calculations for drill pipe or annulus using rotary viscometer data.

3.5. Effect of Temperature on the Rheology

The effect of temperature on the rheology of the drilling fluids can be summarized as follows:

1. The mobility of the water component increases with elevated temperatures and influences the clay particles dispersion.

2. Active water molecules affect the chemical additives and disturb its stability.

3. High temperature reduces the effectiveness of most mud conditioning agents.[18]

In a weighted mud system, the solids contents are composed of a heavy fraction and a light fraction. The effect of the high specific gravity solids depends on the mud weight. The heavier the mud, the more the dependence of the rheology on the structure and texture of the weighting material. The shape, size and surface changes of the solid particles control the rheological behavior of the fluids together with other factors including the mechanical interaction of solids and liquids and the effective shear rate at elevated temperature. Low shear rate viscosities are greater than the viscosity at higher shear rates and the difference becomes greater as temperature increases.[1,18] This indicates the necessity of measuring the rheological properties of drilling fluids at ranges of temperature reflecting the actual borehole conditions. The extrapolation of mud properties to higher temperatures or shear rates is misleading. Therefore accurate rheological parameters for water base drilling fluids can only be determined by direct measurement at the required temperature.[1,29] High temperature causes severe gelation of mud. This gelation is attributed to the reaction between the hydroxyl anions, calcium cations, and clay in the mud. The chemical reaction forms some type of cement composed of calcium-alumino-silicate.[25]

It is part of this work to evaluate the behavior of the iron oxides weighted drilling fluids at high temperature. To concentrate on the role of the weighting material, any change in ion concentration or shift in relative concentration of various ions was avoided. Amount and kind of clay types that may influence the mud behavior were adjusted and its effects were brought to the same magnitude in all mud systems. The thickening of the tested muds and the changes in gel strength at elevated temperatures are investigated using simply formulated muds. Chemical additives were added whenever necessary at minimum amounts.

3.6. Adjustment of the Flow Properties

Control of the flow properties of the water base mud requires an adjustment of the important rheological properties.

These properties are; viscosity (apparent, plastic, and effective), gel strength and yield point. The plastic viscosity is primarily determined by the size, shape and number of clay particles and their concentration. The plastic viscosity can be reasonably controlled by adjusting the water content and the solids content in the mud. The dilution of drilling fluids decreases the plastic viscosity whereas an increase in the solids content causes an increase in the plastic viscosity. Dilution is very effective for altering the flow properties of mud systems with respect to the Bingham plastic model. The yield point decreases as a result of increasing the liquid content or by addition of dispersants and deflocculants and increases by addition of viscofiers and flocculating agents.

In the power-law model, the addition of any viscosifier like bentonite or xanthan gum will affect the K value (viscosity). Flocculation or beneficiation of bentonite also raises K, whereas deflocculation or dispersion together with dilution will lower K. Mud additives are mainly used to rectify the mud properties. Therefore additives tend to lower the factor n (degree of deviation from the Newtonian fluid) in the power law model.

An important parameter, the gel strength, was not considered in any of the above mentioned models. Many investigators have emphasized the importance of gel strength in mud performance.[18,24, 29] The gel strength should be just high enough to suspend the weighting material and the drill cuttings when circulation is stopped. Higher gel strengths are undesirable because they retard the separation of cuttings and require high pressure to initiate flow. Gel structures build up slowly with time, as particles orient themselves into positions of minimum free energy. Mungan and Jessen suggested that gelation results when layers of water molecules bond clay particles causing isolation of the liquid water in nonconnected chambers. The authors believe that the gel strength arises from bond strength within the clay film.[14] The strength of the drilling fluids gel depends on: (1) flocculation value for the system, (2) the concentration of the clay and of the salt, (3) amount and type of solids, (4) time, (5) temperature, and (6) chemical composition of the mud system. The gel strength is directly measured by a multispeed viscometer at approximately 3 rpm at two intervals of time (after 10 seconds and 10 minutes of agitation). The multispeed Fann model 35 is used in this work to measure the specified gel strength together with long term gel behavior of the weighted mud systems. Measurements were all reported in lb/100 ft^2. The difference between the initial gel strength and that after a 10 minute rest period or more may be used to judge how thick the mud will get during round trips.

3.7. Evaluation of Drilling Fluids Conditions

Desirable properties of a new drilling fluid can be summarized as follows:

1. The rheological properties must be efficient in all parts of the circulating system.

2. The original viscometric characteristics must be retained after exposure to high shear, i.e. bit nozzles.

3. The drilling fluid must be capable of suspending the weighting material in weighted mud systems by having adequate plastic viscosity and gel strength.

4. The drilling fluid should be compatible with the treating chemicals, viscosity builders, fluid loss reducers and other materials.

5. The mud must be stable at elevated temperatures for the operating temperature ranges.

6. The less the sensitivity of the mud to soluble salts, the more efficient it is.

7. The abrasivity to wellbore and surface equipment must be at an acceptable level, i.e., equivalent or below the level of the common mud types.

8. The deterioration of the mud components is slow and does not impose the frequent use of chemicals or the continuous elimination of drilling fluids material from the active system.

The performance of the components of the drilling fluids should be evaluated under conditions simulating as close as possible conditions of use. There are no standardized methods for evaluating drilling fluids components. Evaluation techniques have to be developed according to the type of additive, its function in the drilling fluid and the purpose of using it.

In evaluating weighting agents, it is important to observe changes of the performance of mud due to contamination caused by various types of natural material. Laboratory tests must be directed to the examination of specific properties under controlled conditions. A laboratory test found quite useful in this work in testing weighting material of iron oxide, was a high temperature aging and compatibility test combined with high temperature high pressure filteration test. All conditions applied to the various mud systems were the same except for the weighting agent. Tests were applied to a specific shale formation in Oklahoma (Atoka Shale) and performed in a way that all rheological, physicochemical and filteration property changes with drilling progress, are demonstrated. The tests were able to establish the quality level of each material which can be used for future improvement. The results can be used for selection of mud systems and provide an economic evaluation of the principal functions of the iron oxides weighted drilling fluids system in comparison with the performance of the acceptable barite weighted systems.

3.8. Use of Rheological Parameters Relationships

Based on the Bingham plastic model, the shear stress $\tau = \overline{yp} - \overline{PV}\ (\frac{dv}{dx})$ where $\overline{yp}$ is the yield point, $\overline{PV}$ is the plastic viscosity and $\frac{dv}{dx}$ is the shear rate, it is possible to plot $\overline{PV}$ versus density or $\overline{yp}$ versus density.[18] The graphs may consist of straight-line portions with their points of inflextion reflecting changes in certain changes in the development of the phyisicochemical conditions of the drilling fluid. Riabcheko, et al. (1968) proposed using $\overline{PV}$ versus $\overline{yp}$ curves to evaluate stability of drilling fluids. The major portion of the curve can be represented by the linear equation

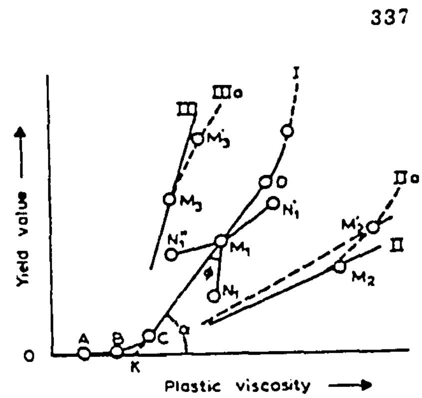
$$\overline{yp} = \tan\alpha\ (\overline{PV} - \overline{PV}_{min})$$

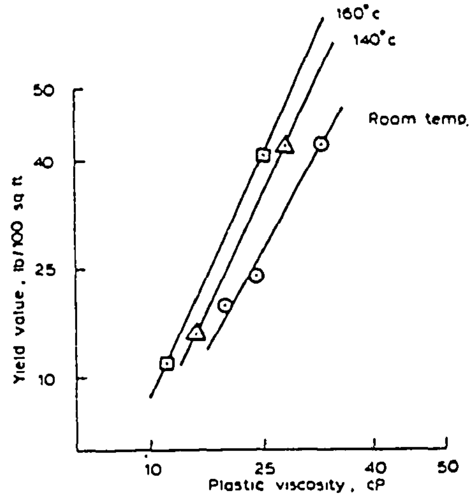
where tan α is the slope of the line, $\overline{PV}_{min}$ represents the plastic viscosity at zero yield value. Typical curves for density-plastic viscosity and $\overline{PV}$ - $\overline{yp}$ are shown in Figure 7 (a and b) plots were found applicable to the water-base weighted mud systems investigated in this research.

The apparent viscosity-specific gravity relationships can be also used in mud systems evaluation. An abrupt change in apparent viscosity at specific mud weights can be detected by breaks in the straight line relationships represented by the formula

$$\tau = - \overline{AV}\ (\frac{dv}{dx})$$

This technique was found to be capable of indicating the stability of the systems of a given clay concentration and its unduly changes with addition of weighting agents. The results obtained together with fluid loss data form useful tools in understanding and predicting the behavior of different mud systems. It can be used as a guideline in the field control of the drilling mud systems and in taking timely actions. The





a. (After Riabchencho, 1968) Curve I untreated fresh water, Curve II, stabilized mud and Curve III coagulated mud.

b. (After Mathur and Chilingar, 1979) Effect of thermal aging on stability

Fig. 7. Relationships between plastic viscosity and yield point.

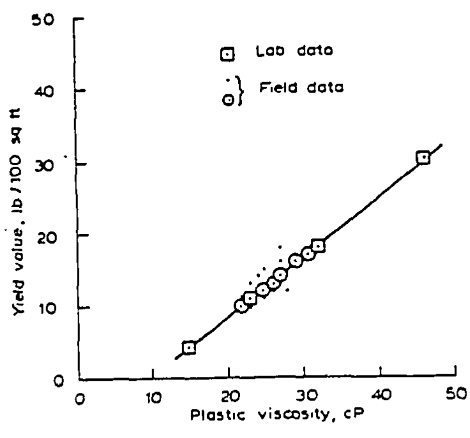


Fig. 8. Plot of field and laboratory results for plastic viscosity—yield point relationship (After Mathur and Chilingar, 1979).

agreement between the laboratory and field data points (shown in Figure 8) encourage the application of this technique to other important mud parameters such as instant and long term gel strengths.

CHAPTER IV

DRILLING FLUIDS WEIGHTING MATERIALS

It is essential during drilling operations that the hydrostatic pressure of the drilling fluid column be slightly higher than the formation pressure in order to prevent blowouts. If the mud is unnecessarily heavy, the following problems can be expected: (1) swabbing and surge pressures during pulling or lowering of drill pipes and casing from or into the borehole, (2) loss of circulation occurring as a result of loss of drilling fluids into fractures induced by excessive mud pressures into preexisting open fractures or openings in the rock. Therefore, it is important to keep a check on the specific weight of the drilling fluids and to add the appropriate weighting material in order to achieve the following results: (a) prevent the inflow of formation fluids, (b) adjust the bottom hole pressure to slightly higher than the formation pressure, and (c) to balance any sudden drop in the mud weight, which may occur as a result of formation fluids inflow.

Material used in drilling fluids in the greatest quantities are those added to increase the drilling mud density. Conditions that must be satisfied in a weighting material for drilling fluids are listed below.

1. Material should have high specific gravity.

2. It must not react with the continuous phase of the drilling fluid.

3. Material should be ground to the optimum particle size distribution.

4. The abrasivity of the material should be very low when ground.

5. The amount of particles larger than 44 microns (+325 mesh screen) must be held to a minimum as high density materials are more difficult to keep in suspension.

6. The hardness should be reasonably low, the harder the material, the more abrasive it becomes.[22] At the same time, if the material is too soft, progressive comminution of particles due to grain-to-grain attrition may occur.[62]

7. Particles having a diameter less than 2 microns are held to a minimum because they increase the effect of the material on the viscosity and gelation of the drilling fluids. Table 1 shows the materials that can be used to increase the mud weight together with some selected properties.

The effect of the specific gravity of the weighting material on the solids concentration can be seen in Figure 9.

TABLE 1

Materials That Can be Used to Increase Density of Drilling Fuilds

Material	Principal Components	Sp. Gravity Kg/m^3	Hardness Moh's Scale
Lead	Pb.	11.31-11.33	1.5
Iron	Fe.	7.6 - 7.8	4.5
Galena	Pbs	7.4 - 7.7	2.5 - 2.7
Ferrosilicon	Feo & Si	6.5 - 6.9	6.5 - 7.0
Hematite	$Fe_2 0_3$	4.9 - 5.3	5.5 - 6.5
Magnetite	$Fe_3 0_4$	5.0 - 5.2	5.5 - 6.5
Iron Oxide	$Fe_2 0_3$	4.7	-
Ilmenite	$Feo\text{-}Ti0_2$	4.5 - 5.1	5.0 - 6.0
Barite	$Ba\ So_4$	4.2 - 4.5	2.5 - 3.5
Siderite	$FeCo_3$	3.7 - 3.9	3.5 - 4.0
Celestite	$SrSo_4$	3.7 - 3.9	3.0 - 3.5
Strontianite	$Sr0_3$	3.6 - 3.7	3.5 - 4.0
Dolomite	$CaCo_3 \cdot MgCo_3$	2.8 - 2.9	3.5 - 4.0
Calcite	$CaCo_3$	2.6 - 2.8	3

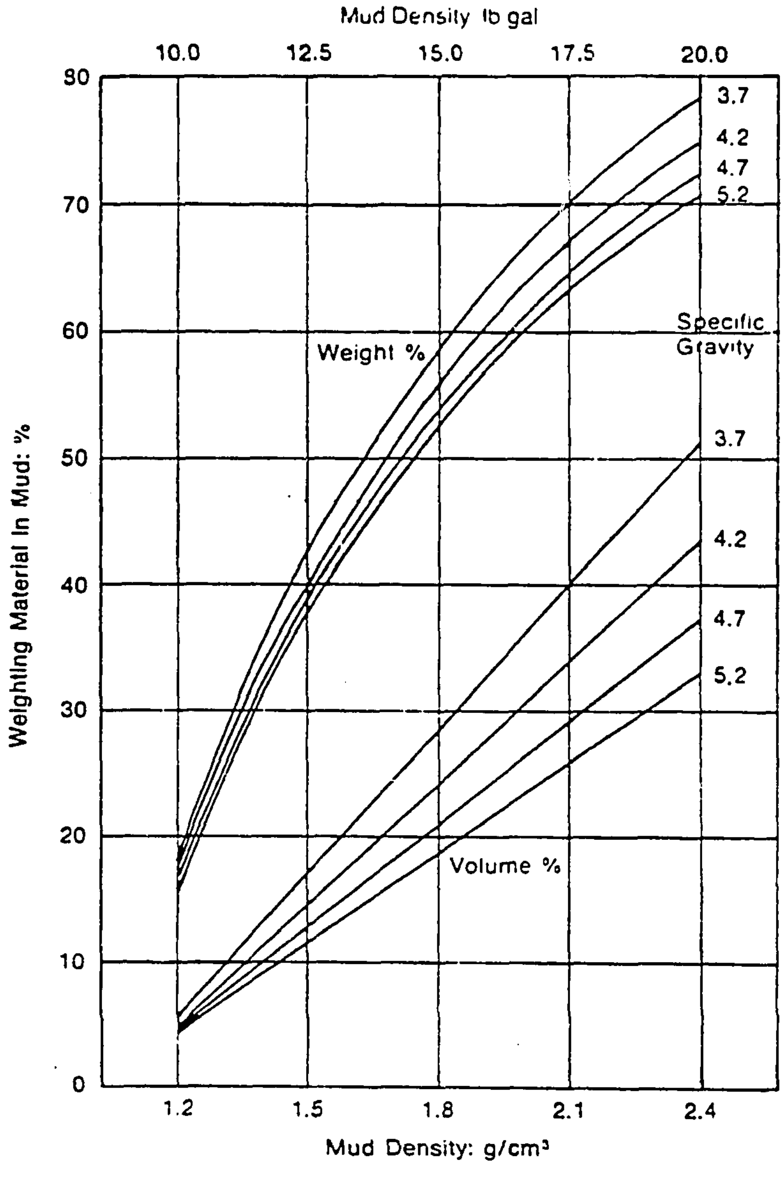


Fig. 9. Effect of specific gravity of weighting material on the solids concentration of weighted mud (After Gray and Darley, 1980, Ref. 29).

4.1. Evaluation of Barite as a Weighting Agent

4.1.1. Barite Properties and Specification

The properties that led to barite becoming the most extensively used material for increasing the density of the drilling fluids are:

1. It has a fairly high specific gravity (s·g = 4.2).

2. Barite has a very low abrasiveness due to its low hardness (3.0-3.5 on Moh's Scale).

3. The material is inert in both oil and water, therefore both water-base and oil-base muds can be weighted with barite.

4. The mineral is economical to mine and to beneficiate.

The American Petroleum Institute specifications for barite are summarized below.

1. The minimum specific gravity is 4.2.

2. The soluble alkaline earth metals (i.e., calcium) should be 250 part per million, maximum.

3. Residue on the U.S. sieve number 325 is 5 percent maximum.

4. Barium sulfate ($BaSo_4$) lies between 83-93 percent.

The above specifications are believed to be too brief to assure a satisfactory performance of barite weighted drilling fluid systems under all possible conditions of use. Therefore further tests must be performed under conditions of actual utilization. These tests are conducted to make certain that the weighting material is low in soluble salts, silica, iron and lead. The following limitations are specified.

1. Least amount of soluble salts having polyvalent cations (i.e., calcium sulphate).

2. Silica is objectionable when in the form of discrete quartz inclusions that make the barite more abrasive.

3. Iron oxides contamination is not objectionable when in the proper particle size and range stated in API specifications for barite. The iron oxide can be an advantage if it increases the specific gravity of the ground product without changing the properties of the drilling fluids.

4. Lead inclusion is beneficial when it occurs as an insoluble form because it increases the specific gravity substantially. But when it replaces barium in the crystal structure it causes undesirable surface activity in the water base drilling fluids.

4.1.2. Barite Demand and Consumption in the United States

The demand for barite rose from approximately 100,000 tons a year in 1942 to approximately 3.0 million tons in 1981 due to acceleration in oil and gas in the United States.[5,6,8,9] The principal use for barite as weighting material in well drilling fluids accounted for 91 percent of the total U.S. consumption. Barite consumption per foot drilled in the United States has shown a general upward trend. The additional mud cost is recovered in a reduction in overall well costs mainly through faster penetration rates and maintenance of optimum hydraulic properties.[36] In the United States a wide scale survey over the past forty years shows that the average depth of a well has increased almost 60 percent, from 3,000 to 5,000 feet, while at the same time the amount of barite consumed per foot of drilling has increased eight fold from 2.8 lb to 23 lb.[32] Crushed barite is also used as an aggregate in the concrete coating in the underwater oil and gas pipelines for its high specific gravity and inertness. With the current, enormous developments of offshore oil fields, the rate of barite consumption for aggregate is expected to increase.

The United States produces about 2 million tons of barite, this amount covers approximately 60 percent of its consumption. Nevada supplies 82.5 percent of the total barite mined in the United States. This production comes from the large bedded reserves of the Battle Mountain area and the Toquima Range in central Nevada.[7,9,11,46] Imports increased steadily from 955,000 tons in 1977 to 1.291 million tons in 1978 then to 1.4 million tons in 1979.

4.1.3. Barite Consumption in Other Parts of the World

Since about 1930, chemical industries and other users of barite consumed less than 20 percent of the barite mined in the Western world. The remaining amount (80 percent of the total production) was consumed in drilling fluids used for oil and gas exploration and development. The United States produces more drilling mud grade barite than all other Western countries.[8,11]

In the North Sea oil drilling operations, barite accounts for 20 to 80 percent of the overall cost of the drilling fluids. The average is taken as 40 percent. Barite consumption in the North Sea is of the order of 180,000 to 220,000 tons and is expected at least to be maintained over the next few years.

After 1959 the barite consumption outside the United States has increased sharply due to growing petroleum exploration activities. The mineral is produced in more than 40 countries. The production is exported or consumed as crude or after processing according to the facilities and industrial activities of each country. The total world barite production was estimated at 6.1 million tons in 1979,[8] and is expected to have maintained approximately the same level until 1981.

4.1.4. Barite Occurrances and Resources

Brobst, 1970[11] estimated the world total barite reserves at 204 million tons. He broadly defined the term "reserves" to include bodies of barite rich rock of sufficient size and grade to be mined now or to warrant serious consideration for mining under slightly more favorable economic conditions. This estimate represents only a 20 year supply assuming that the annual production rates continue to climb in the next 20 years as they have in the past 20 years. Barite reserves of North America are estimated at 32 million tons including 25 million in the United States, 3 million in Canada and 4.0 million in Mexico.[2,11]

Barite occurs mainly as residual bedded, lenticular replacement veins, tabular bodies, mineralized breccia zones and dissiminated deposits. It has been produced as a co-product of fluorspar, base and precious metal and rare earths. It is also a minor constituent of many lead and zinc ores.[51,53] In recent developments, barite was recovered from fluorite mill tailings. The tailings are thickened to about 32 percent solids and the barite floated with sodium cetyl sulphate.[51] Grades as high as 97 percent $BaSo_4$ with recoveries of 80 percent have been achieved.

4.2. Problems Associated with Barite

Future barite supply problems are predicted by authorities, including the U.S. Department of Interior (Mineral Commodity Profile, Feb. 1974).[9] There is also a growing dissatisfaction in the oil industry with the available barite even when the mineral meets all current API specifications. Commercial barite may contain from 10 to 30 percent by weight of a complex mixture of water insoluble mineral impurities. Some of these impurities are soluble in alkaline drilling fluids. Binder et al., 1981[10] found that little trouble may be caused by cations from solublized impurities whereas soluble anions of carbonates and sulphides may cause serious problems. These problems include lost circulation, stuck pipe and even catastrophic loss of the well. Cases of high level carbonate and sulphide contaminations were recorded in wells in southern Louisiana and in Alabama causing excessive gel strengths and repeated drill pipe failures.[10]

There are other problems associated with the widespread use of barite for oil well drilling fluid. A major problem is the transportation of this heavy mineral for long distances significantly increasing the price, especially with the recent increases in freight charge. The mineral is not readily available near centers of petroleum activities. The United States imports barite from South American countries as well as Asian countries such as India and China. The problem is much worse for the North Sea drilling activities where nearby reserves are very limited. The same problem occurs in some Middle Eastern countries such as Egypt, Saudi Arabia and the Arabian Gulf Emirates. These countries have no significant barite product or significant barite deposits to satisfy the needs of growing drilling acitivites.[11,43]

4.3. Proposed Solutions for Problems with Barite

To overcome problems of barite shortage and degrading quality, the following solutions are proposed.

1. The mineral shortage can be dealt with effectively by reclaiming, recycling and reusing barite weighted drilling fluids. This requires an extremely efficient mud treatment system, way above the normal mud maintenance level.

2. Requirements for new barite supplies can be decreased by the efficient use of barite in the drilling mud and by deceleration of the progressive comminution of the soft barite particles. A number of polymers were selected and

tested in this research for use as a lubricant or protective coating for barite. This technique helps to slow down the disintegration of the mineral grains which leads to the deterioration of the mud properties and the immediate elimination of the deteriorated fraction from the active mud system.

3. Blending of a very high specific gravity mineral with low grade barite of specific gravity 4.1 or less to reach specific gravity of 4.2 or more. This solution would probably increase the barite contamination hazard. Therefore in this work, API grade barite was used with other heavy minerals to give a weighting material with less contamination hazards than barite.

4. Using alternative weighting agents among the widespread natural minerals. The selected minerals should satisfy all requirements stated previously for weighting materials in drilling fluids including high specific gravity, chemical inertness and availability near petroleum activity centers.

5. Using barite substitutes among synthetic material or industrial biproducts which satisfy the requirements of weighting agents. A synthetic iron oxide, produced as a biproduct by Kerr-McGee Corporation, has been tested extensively in this work for use as a total or partial barite substitute. Testing techniques and results will be furnished in detail in the following chapters and appendices.

CHAPTER V

USING IRON OXIDES TO INCREASE THE DENSITY OF DRILLING FLUIDS

The most effective solution to the barite problem would be to find an alternative weighting material. This material can be found among the natural or synthetic substances that satisfy all requirements necessary for weighting agents in drilling fluids. This material may be used for full or partial replacement of barite in weighted mud systems.

Although published work on barite alternatives is very scarce, it is evident that some oil service companies are looking into possible barite alternatives. Natural mineral weighting material now on the market are: ilmenite (an iron titanium oxide) and itibirite; a mixture of iron oxide and silica, imported from Brazil. Among synthetics, a new iron oxide has been produced in small amounts for testing purposes by Kerr-McGee Corporation. This mineral was tested extensively in this work together with the itibirite and ilmenite for weighting drilling fluids. Defects in performance were determined using the appropriate testing procedures, and experiments were conducted to find the appropriate remedy for each problem.

5.1 Ilmenite as a Barite Alternative

The single laboratory work published on the use of ilmenite as a weighting material for drilling fluids was conducted by Rogaland Regional University Research Center in Norway (Nederveld and Vieaux, 1980). The Rogaland Laboratory studies cover tests of attrition and physical properties.

1. Study of attrition

In the Rogaland study of attrition, prepared ilmenite and barite muds were rolled in a ball mill up to 24 hours, and tests of rheological characteristics were taken as an indication of particle attrition. It was reported that a significant increase in the viscosity and yield value of barite mud was encountered compared to the ilmenite mud. The results were represented graphically, as shown in Figures 10a and 10b. The mud system, composition and testing temperatures were not specified. It was not mentioned which part of the drilling circulating system this simulation represents or how the measurements were performed.

2. Study of physical properties

Results of the Rogaland Institute study, together with a field test by a team from the ilmenite marketing company, NL Baroid, were reported by Nederveld and Vieaux, 1980. As a result of the study it was concluded that ilmenite ore has three major advantages over barite: (1) it has a higher specific gravity (4.58 compared to 4.3 for barite), (2) it has less attrition of particles during use, (3) it possesses a negligible amount of contaminants. These advantages are

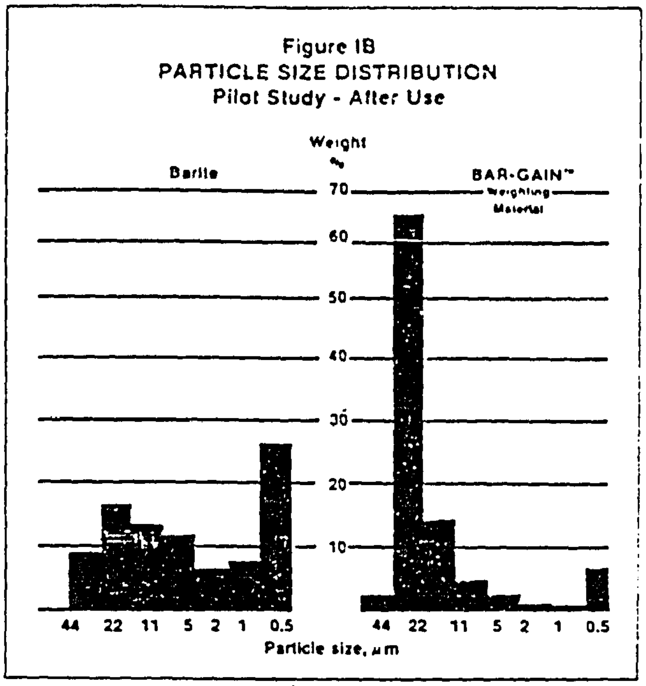


Fig. 10a

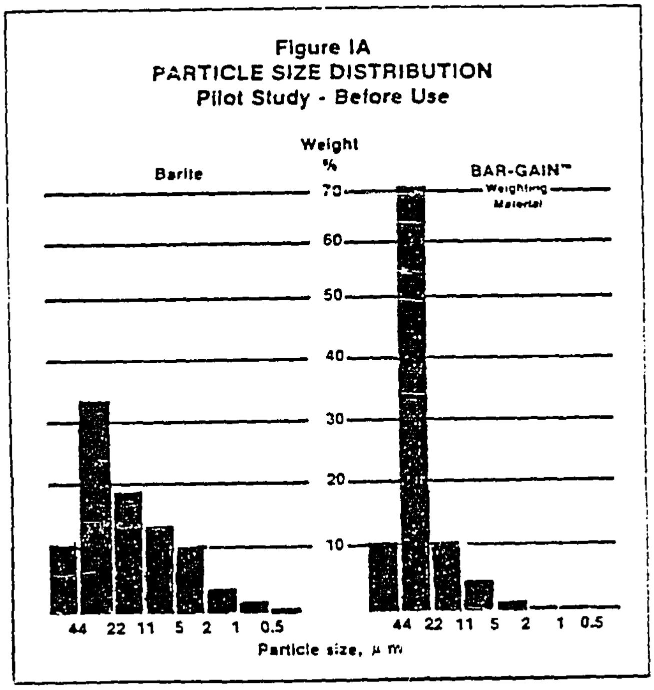
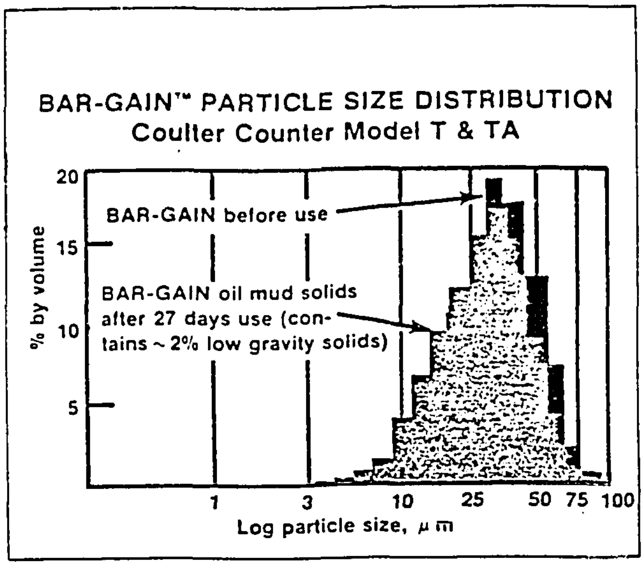


Fig. 10b

Figs. 10a, b. Presentation of results for the Rogaland Institute, Norway, on attrition of barite and ilmenite (after Nederveld and Vieaux, 1980).

believed by the authors to decrease the weight/volume requirements of ilmenite thus saving 2.5 percent by weight and 10.6 percent less by volume of weighting material compared to barite. The authors also concluded that mechanical separation of weighting material and drilled solids becomes easier, and lower solids content can be achieved with ilmenite. The authors proposed that ilmenite being harder than barite (ilmenite 5.0-6.0; barite 3.5 on the Moh's scale) does not erode to a smaller size as readily as barite. This is demonstrated in Figures 10a and 10b, showing both ilmenite and barite before and after use during the Baroid team field experiment. The researchers claim that provisional testing of the ilmenite showed that disadvantages due to abrasiveness were found to be comparable to barite.

The limited field test by the NL Baroid team used data from four wells drilled with barite weighted oil mud, and one well used EZ oil mud weighted with ilmenite ore. Reports showed that ilmenite weighted EZ oil contributed to faster drilling due to lower solids content. The results of this field experiment are represented graphically in Figures 11 and 12. Drilling fluids tested in this field experiment were all oil base systems. No water base, ilmenite weighted system was tested; therefore, the above mentioned conclusions are believed applicable only to the EZ oil base mud and can probably be extended to include other oil base mud systems. The reason for the relative low abrasivity is probably due to the high



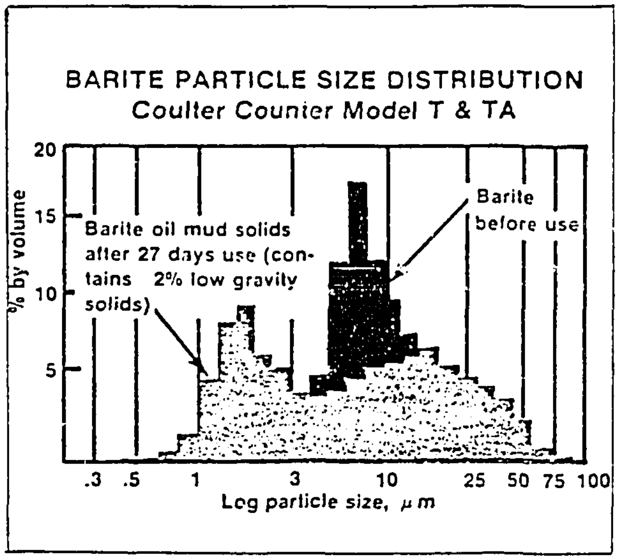


Fig. 11. Particle size distribution for barite and ilmenite before and after field use in oil base mud (Baroid, pilot study, Nederveld and Vieaux, 1980).

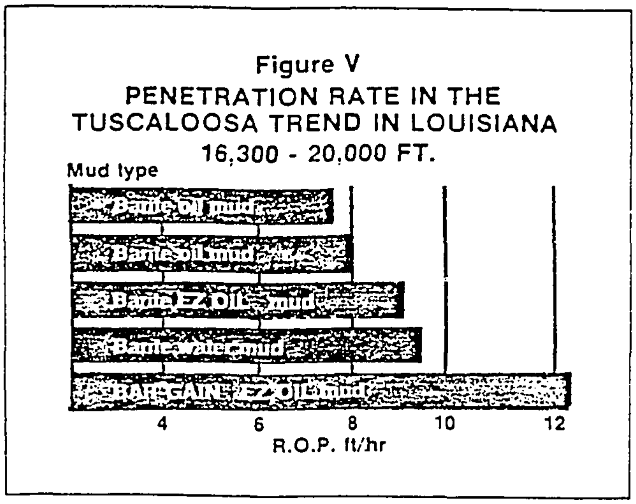


Fig. 12. Comparison between penetration rates for oil muds using barite and ilmenite as weighting agents (Baroid team field study).

lubricity possessed by the EZ oil mud which is reported by NL Baroid to be the highest lubricity of any oil mud systems.

5.2 Specifications of the Drilling Mud Grade Ilmenite

The drilling mud grade ilmenite, now on the market under the commercial name "Bargain" has a specific gravity of 4.58 approximately 9 percent greater than that of barite. To mix an equal density fresh-water gel mud would require 2.5 percent less Barbain by weight. Its abrasion characteristics are claimed by the distributors to be similar to those of barite. Physical and chemical attributes can be summarized in the following:

1. Total soluble salts are less than 500 ppm.
2. Soluble alkaline earths, i.e., calcium are less than 200 ppm.
3. The mineral is slightly acid soluble.
4. It is less than 35 percent iron.
5. It has less than 1 percent moisture.

5.3 Use of Itibirite in Drilling Fluids

Itibirite is composed mainly of micaceous hematite and a small amount of quartz. The mineral has a specific gravity of 5.10, more than 20 percent higher than barite. Itibirite is derived from the processing of the metamorphosed hematite bearing itibirite sediments of Brazil. It has been imported recently (late 1981) and ground to satisfy API specifications for grain size distribution of barite. It is marketed now under the commercial name "Densimix." The only published work on the material is an article in Oil and Gas Digest (December 1981)[16] and several advertisements appearing in scientific magazines. The article claims that drilling fluids weighted with itibirite have less solids content which reduces the need for expensive chemical treatments, a thinner filter cake, lower gel strength, lower plastic viscosity which reduces friction losses down the drill pipe and through the bit nozzles and better cleaning of the bottom of the hole. The article also claims that faster drilling rates can be achieved with itibirite averaging 30 percent or better. A solids reduction was estimated at 37 percent when weighting mud up to 22 ppg. Compared to barite, it is probably more environmentally acceptable because it is free of heavy metals.

Itibirite is composed of a mixture of hematite (Fe_2O_3) and silica (SiO_2), and therefore is expected to have a hardness between 6.0-7.0 on the Moh's scale. The abrasiveness of this material is a disadvantage according to results obtained from wear tests performed on laboratory prepared drilling fluids. The material was subjected to various tests of performance to reveal its actual properties under simulated down hole conditions.

Natural hematite, the main constituent of the itibirite, was used on a large scale as a mud weighting material. Some industrial sources claim that the mineral has undesirable filtration properties. It is believed that it tends to increase the filtrate loss and the filter cake thickness to a considerable degree.[24,25] This claim will be closely examined in this work and the appropriate treatment will be conducted.

5.4 Synthetic Iron Oxides as Weighting Material

5.4.1 Use of Fer-O-Bar for Weighting Drilling Fluids

The idea of using industrial biproducts as weighting agents for drilling fluids was first proposed in West Germany. A specially treated pyrite residue prepared by Sachtleben Chemie of Cologne was composed of 85 percent synthetic iron oxide (Fe_2O_3) and 15 percent silicates of aluminum, zinc and calcium. According to the project manager, W. Bulian, 1973 (Wintershall AG, Barnstorf, NW Germany), the following physical properties were reported for the new material named "Fer-O-Bar."[9,11]

1. The material possess a specific gravity of 4.7.

2. It has a maximum of 0.1 percent water soluble solids and a maximum of 100 ppm water soluble alkaline earth metals.

3. It has a pH of 7.0 and is inert at a pH value greater than 2.0.

4. A proportion ranging from 80 to 85 percent is soluble in hot hydrochloric acid.

5. It has low viscosity and favorable flow properties to drilling muds even at high mud weights.

6. This material produces heavy muds with a lower volume of weighting material solids, at least 16 percent less than in equivalent weighted mud.

7. The material was not expected to cause any electric or magnetic problems.

8. The particle shape of the Fer-O-Bar was claimed to be more rounded than natural hematite.

9. Filtration and rheological properties of muds weighted with this material were reported as similar to muds of the same specific gravity weighted with barite.

No testing details were published to support the claimed advantages of using the product. Fer-O-Bar was found incompatible compared to barite for the following reasons.

1. There were doubts whether the cost would be competitive with barite (based on 1973 low barite prices).

2. The claimed low abrasiveness needed to be verified.

3. There were doubts as to whether the new weighting material would remain in suspension during gas cutting processes, due to lack of testing data.

4. Claims of noninterference with electric and magnetic well logging were not confirmed by data.

5.4.2 K-M - Hematite as a New Weighting Agent

The K-M - hematite is a new synthetic industrial bi-product. It was prepared by the Kerr-McGee Corporation in Oklahoma City by special treatment of some iron oxides and iron sulphides. The material has been produced, so far, in very small amounts at the company's pigment plant. The K-M - hematite has a higher specific gravity than any other iron oxide used in drilling fluids. With a specific gravity of

5.16, the material is more than 22 percent denser than barite. It was tested extensively in this work for compatibility and rheological performance at varying temperatures and shear rates. Results, shown and analyzed in the following chapters, have indicated that the mineral is superior to barite in many aspects. Filteration and abrasive defects revealed by testing have been improved significantly by chemical additives or by mixing with barite depending on desired mud properties.

Scanning electron micrographs indicated that the K-M - hematite possesses a spherical to subspherical grain shape. This phenomenon has reduced the abrasiveness due to the high hardness of the substance (5.5-6.5 on the Moh's scale) below the abrasion level of the similar iron minerals. Although the abrasiveness is still higher than the barite level, it will be shown experimentally that it was possible by adding small proportions of some specified polymers (less than 2 percent by volume) to bring the abrasion level to the barite level under high shear rates (approximately 20,000 rpm). This was achieved without changing the rheological properties and with minimum chemical treatment.

According to Kerr-McGee Corporation sources, the synthetic iron oxide is composed of 60 percent Fe^{+++}, 0.17 percent Fe^{++}. It contains 5.17 percent titanium dioxide (TiO_2), 4.7 manganese dioxide and a minute amount of chlorine, smaller than 0.01 percent. The material contains very minute amounts of heavy metals impurities including Ag, As, Ba, Cd, Cr, Hg, Pb and Se varying from less than 1 ug/l (for Hg and Ag) to a maximum of 46 ug/l (for Pb). This is significantly below the EPA specified permitted maximum for this metals changing from 200 ug/l to 100,000 ug/l according to the Resource Conservation and Recovery Act, 1976. Arc emition data on the synthetic hematite indicated an amount of 1.1 percent by weight of aluminum, 0.1 percent for calcium, 0.3 for magnesium 0.2 percent for silica and minute amounts for Co, Cu, Ni, Pb, Si, Zn and Zr varying from 0.003 percent (Pb) to 0.1 percent (for V, Zn).

CHAPTER VI

PROCEDURES FOR EVALUATING THE IRON MINERALS MUD SYSTEMS

The properties of the water base drilling fluids change markedly with temperature, shear rate and shear history. During their circulation, on the surface and down hole, the drilling fluids are subjected to changeable conditions including laminar and turbulent flow in the drill pipe, intense shearing at the bit and the bit nozzles and variable shear rates at the annulus. When evaluating drilling fluids downhole conditions should be simulated as close as possible to actual borehole conditions and limitations. Standard methods were sometimes inadequate in testing drilling fluids performance under certain conditions, therefore it was necessary to develop testing procedures to cope with the problem-solving requirements and to reflect the downhole behavior as closely as the specially designed equipment allows.

6.1 Mud Formulation and Preparation

Four batches of mud were prepared for testing of rheological, electrical and magnetic properties. All muds possess the same amount of bentonite (3 percent) and contain enough weighting material to increase the density to the required density for all four mud types. The prepared samples differ only in the type of weighting agents. For abrasivity and performance compatibility tests, in addition to the above four batches, muds composed of mixed weighting materials were tested. Various types were mixed until the most compatible mixtures were achieved. The main constituents of the four mud types tested throughout this work are shown in Table 2. Composition of two compatible mud mixtures or muds weighted with mixed weighting material are shown in Table 3.

TABLE 2

DRILLING MUD TYPES

1. Mud A 3% bentonite mud weighted with ilmenite (Bargain).
2. Mud B 3% bentonite mud weighted with K-M hematite.
3. Mud C 3% bentonite mud weighted with itibirite (Densimix).
4. Mud D 3% bentonite mud weighted with API grade barite.

TABLE 3

COMPATIBLE MUD MIXTURES

5. Mud AC 3% bentonite mud weighted with W(0.6 ilmenite + 0.4 itibirite).
6. Mud BD 3% bentonite mud weighted with W(0.5 K-M hematite + 0.5 barite).

W is the total weight of the weighting agent calculated on the basis of the average density of the two components.

All percentages are relative to total final weight of mud. In preparing mud batches for testing, the following procedure was followed.

1. All muds were prepared from dry material, therefore;

   a. Each was given a first mixing using a multimixer,

   b. The mud was aged for 24 hours to allow the colloids time to hydrate,

   c. All muds were subjected to high shear rate for 15 minutes to obtain a constant viscosity.

2. The mud weight was determined using the mud balance.

3. Small adjustments were made to bring the mud weights to the specified value.

4. The mud was remixed for 5 minutes and the pH value was determined.

5. The desirable pH value (between 8 and 11) was obtained with all muds without chemical additives except for barite weighted mud. To adjust the pH value for barite mud, small amounts of concentrated solutions of caustic soda were added.

6. The muds were remixed for 15 minutes before the rheological properties were measured.

To determine the amount of the weighting material required to formulate a specific mud type at a particular mud weight, special charts for each weighting material were constructed.

The weighting material calculations were based on the formula

$$V_w = \frac{V_i(\nu_f - \nu_i)}{(\nu_w - \nu_f)}$$

V_i = volume of mud before weighting; V_w = volume of weighting material added; ν_i = initial specific weight of mud; ν_w = specific weight of weighting material; ν_f = final mud specific weight.

The volume and weight of the weighting material for a specific mud weight can be obtained from the charts shown in Appendix A. A chart for assessing ilmenite addition to mud compared to barite was constructed and shown in Figures 19a and 19b in both metric and field units for laboratory and field applications. Similar charts (Figures 20a and 20b) were designed for the K-M hematite and can be applied to itibirite addition to drilling fluids.

The preparation of the weighted mud usually requires a check for equivalent bentonite content. An increase in the level of bentonite indicates an increasing low gravity solids.[47] In high weight muds there is an upper bentonite toleration level. If this level is exceeded, an excessive thickening of the mud may occur at the bottom of the hole. Therefore, for weighted mud it is important to measure accurately the solids content and to have available means of determining the concentration of the high and low gravity solids contents. The high gravity solids are decided by the amount and concentration of the weighting material. The charts shown in Figures 21 and 22 (Appendix A) are designed for estimating amounts of ilmenite and hematite or itibirite in the mud. These charts were prepared from the mass balance equations and show the advantage of lower solid contents of the iron oxides compared to barite. The charts indicate the high gravity solids content of the mud occurring as a result of adding iron minerals, for any specific mud weight. Composition of various mud types can be seen in Tables 6 to 14 of Appendix B.

6.2 Rheological Measurements

The viscosity of the drilling fluid is the internal resistance of the fluid to flow. Newtonian fluids exhibit linear flow characteristics, whereas all non-Newtonian fluids, including drilling fluids, exhibit non-linear flow characteristics and require more than a single viscosity term to define their viscous behavior. The measurement of viscosity of drilling fluids is either relative measurements, i.e., the apparent viscosity, or absolute measurements which are the quatitative values of the non-Newtonian characteristics, namely the plastic viscosity, the yield value and the gel strength.

To evaluate the rheological properties of the iron oxides weighted drilling fluids, their viscous properties should be compared to the desired viscous properties for drilling fluids, summarized as follows:

1. It should be shear thinning to impart optimum hydraulic horsepower at the bit.

2. It must have sufficient effective viscosity in the annulus for efficient hole cleaning.

3. Its effective viscosity at the surface, should be low enough to allow removal of the cuttings.

4. Its gellation characteristics should be sufficient to suspend cuttings and weighting material under static conditions.

6.2.1 Ambient Temperature Properties

The prepared mud batches were tested for rheological properties at room temperature using the Fann V-G (Model 35) viscometer. The readings were checked occasionally with a Baroid variable speed rheometer. After properly determining the dial readings at 600 RPM and 300 RPM speeds on the viscometer, the plastic viscosity (pv) in centipoises was calculated as follows:

$$pv = \theta_{600} - \theta_{300}$$

θ_{600} is the dial reading in centipoises at 600 RPM and θ_{300} is the dial reading in centipoises at 300 RPM. The yield value (yv) in lbf/100 ft^2 was determined by the equation

$$yv = \theta_{300} - pv$$

The apparent viscosity va in centipoises is the 500 RPM dial reading divided by a factor 2. Values for apparent viscosities were calculated from the multispeed viscometer readings on the basis shown in Table 4. Values for pv, yp and circulating effective velocities are indicated for all types of mud systems in Tables 6 to 14. The effective viscosities are estimated for each mud system to show its performance compatibility at each part of the drilling circulating system. The graphical analysis for determination of the effective viscosities are based on the shear rate ranges in a circulating system shown in Table 5. Constructing the log-log viscosity profile is essential when increasing the density of the mud for the following reasons.

1. It shows the effect of the drilled solids on the rheological properties of the drilling fluid as drilling progresses. The change in colloidal solids content will change the slope of the curve and help in deciding on the mud treatment.

2. The graphs show which part of the circulating system may cause trouble during drilling and which mud parameter is responsible. This helps to decide how much to increase or decrease certain mud or drilling parameters.

3. When combining various weighting material, i.e., ilmenite-itibirite or barite-K-M hematite, these curves can provide valuable guidance to adjust mud parameters when mixing the weighting material to minimize the need for chemical treatment.

4. The effect of mud treatment, used to improve certain mud properties (abrasiveness and filtration properties

TABLE 4

BASIS FOR CALCULATING THE APPARENT VISCOSITY
Example Table

Shear	Rate	Visc. Dial Reading	Multiplier (M)	Apparent Viscosity (CPS)
(RPM)	(sec^{-1})	(R)	(M)	(R x M)
600	1020		0.5	
300	510		1.0	
200	340		1.5	
100	170		3.0	
6	10.2		50.0	
3	5.1		100.0	

TABLE 5

SHEAR RATE RANGES IN A CIRCULATING SYSTEM

Circulating System	Shear-Rate Ranges (sec^{-1})
Bit Nozzles	10,000-100,000
Drill Collars	700-3,000
Drill Pipe	100-500
Solid Removal Equipment	300-6,000
Annulus	10-500

NOTE: To convert shear rates from RPM speeds to reciprocal seconds (sec^{-1}), multiply the RPM with the constant 1.7.

in this work), on the effective viscosities of the mud system were determined for the various types of mud investigated.

Effective viscosity profiles made possible the quantitative correlation between the barite weighted mud and the iron minerals weighted systems at all sections of the drilling circulating system. Furthermore, it helped in analyzing the experimental results.

6.2.2 Measurement of Gel Strength

The gel strength of a drilling fluid is a measure of the minimum shearing stress necessary to initiate flow of the fluid. The gel strength is measured by taking two readings with the viscometer, referred to as initial gel strength reading which is taken immediately after agitation of the mud in the cup, and the ten minutes gel strength taken after the mud has been quiescent for ten minutes. The difference between the two values is considered to be a measure of thixotropy of the mud system. The value of the gel strength of mud depends mainly on composition, degree of flocculation and type and amount of weighting material. Grey and Darley stated that there is no well established means of predicting long-term gel strengths.[29] The authors emphasized the need for a method of predicting long term gel strengths. The following procedures were used in this study to test long-term gel strength.

1. Mud systems A, B, C and D were prepared simultaneously and aged for 48 hours.

2. All muds were stirred using the multimixer for 10 minutes before testing began.

3. Using the Fann viscometer the mud was stirred at high speed for 15 seconds.

4. A rest time was allowed (10 seconds, 10 minutes, 15 minutes up to 90 minutes) and each time the gel strength knob was turned slowly and steadily.

5. The maximum deflection of the dial before the gel broke was recorded in lb/100 sq. ft.

6. From the plot between gel strength (lb/100 sq. ft.) and rest time a classification of the type of gel strength can be made.

The gel strength behavior can be classified as; fragile gels which would be extremely low and nearly identical for any resting period. Good gels are those which build up slowly and steadily. Progressive gels exhibit a low to intermediate 10 second value then build up to extremely high values. Flat gels usually have identical gel strength of medium to high levels.[24,25] Based on this classification, gel strengths for the tested mud system were identified. The interpretation of the graphical presentation shown in Figure 28 indicates which of the mud systems requires a corrective treatment to avoid possible complications. High gel may cause the swabbing of a kick or breaking down of a weak formation. If the gel is too low, it will be unable to keep the cuttings in suspension during connections or trips.

In weighted systems, the 10 second gel strength of at least 2 lbf/100 sq. ft. is required and preferably 3 to 5 lbf/100 ft^2 and 10 minutes gel of 5 to 10 lbf/100 ft^2. Applying this principle to the investigated mud system, the ilmenite mud was found to be of the progressive type. The barite mud is of the flat gel type and so is the itibirite. The K-M hematite can be classified as "good" gel strength, and is the only mud system which does not require any treatment for gel strength.

Values for 10 seconds and 10 minutes gel strength show that a mixture of ilmenite and hematite mud system (type AC) exhibits high gel strength while a mixture of K-M hematite and barite (type BD mud) possesses good gel strength characteristics. Measurements of gel strengths with time may be seen in Table 15.

6.2.3 High Temperature and High Pressure Rheology

To predict the viscous properties of a drilling fluid at well bore temperatures and pressures, the Fann model 50B high temperature viscometer is used. The instrument is shown in Appendix D together with the digital data acquisition unit.

Tests were carried out on the following mud systems

A. 3 percent bentonite mud weighted with ilmenite.

B. 3 percent bentonite mud weighted with synthetic hematite (K-M hematite).

C. 3 percent bentonite mud weighted with itibirite.

D. 3 percent bentonite mud weighted with barite.

E. A mixture of mud systems A and C.

The testing was carried out using the Amoco Research Center high temperature viscometer. It was carried out in the following manner.

1. The instrument pressure rated sample chamber was filled with the specific drilling fluid and the bob sleeve assembly was immersed into the sample.

2. The sample chamber was immersed in an oil bath.

3. Initial viscous properties of the samples were determined at ambient conditions.

4. The viscous properties were determined by recording the shear stress readings at each shear rate at the desired temperature as the sample was heated up.

5. The shear stress-shear rate readings were also recorded while the sample was gradually cooled to ambient temperature.

The purpose of the above measurements is to provide a comprehensive description of the viscosity-temperature relationship for each mud.[24] The charts produced by the instruments are shown in Appendix D. Interpretation and analysis of data are tabulated as shown in Appendix B. The shear stress readings at 100, 200, 300 and 450 rpm were converted into apparent viscosity values in centipoises. Plots of the apparent viscosity as a function of temperature for each mud system at various shear rates are shown in Figures 29, 30, 31 and 32.

6.2.4 High Temperature Gel Strength

The gelation process depends upon temperature as well as time. The effects of temperature on the initial gel were obtained from the high temperature viscometer charts. Some of the tested samples showed large gel strengths. When running the tests, it was somewhat difficult to find the gels because the instrument plotter is not provided with multiple pens. However the gels were marked on the charts as best as possible while the samples were being run. The values for the minimum shear stress in dynes/cm^2 to initiate flow of the mud at elevated temperatures may be seen against shear rates of 3 rpm in Tables 16 to 19. Plots for shear rate values for gels as interpreted from the charts versus temperature for various iron minerals weighted muds and barite muds are shown in Figure 33. The relationships show the critical temperature for maximum gelation values of each mud. The graphs furnish a correlation of gelation characteristics between iron minerals weighted and barite weighted, untreated muds under varying downhole conditions.

6.3 Measurement of Electric and Magnetic Properties

The importance of measuring and controlling the electric and magnetic properties of drilling fluids was discussed in section 1.3.3. Two independent experiments have been performed to measure separately the electric and magnetic characteristics of the iron oxide mud systems and to compare it with the barite mud system. The experimental procedures are explained in the following subsections.

6.3.1 Measurement of Electrical Properties

The experiment was designed to measure the resistivities of the drilling fluids and drilling filtrate invaded rocks. Resistivities of iron oxides weighted mud and mud filterate were correlated with similar resistivities of barite weighted mud. To predict the effect of using iron minerals in drilling fluids on the spontaneous potential logging results, resistivities of the filtrate saturated core plugs were compared with resistivities of brine saturated plugs (as discussed in subsection 1.3.3). The effect of aging on mud resistivity was also determined. The experiment was performed as follows:

1. The pre-weighted and measured clean dry Berea Sandstone core plugs were divided into five groups of four plugs each.

2. The five groups were saturated individually at the same time using a five way saturation equipment.

3. The three iron oxide mud types, barite mud and a 3 percent concentration brine, were applied simultaneously to the core plugs.

4. The specific resistences of the filtrate saturated core plug were determined using the Fann resistivity bridge and a special core holder (shown in Appendix D).

5. Specific resistances of all mud types used were measured using the resistivity bridge and the appropriate mud cell.

6. Resistivities of core plugs and various mud types were calculated and reported.

7. The three types of drilling fluids investigated together with the control muds were aged using the rolling technique for 24 hours at 200°F and the mud resistivities were determined.

8. Mud resistivities were measured again after the various muds were aged for another 24 hours at 300°F.

9. The mean values for the filtrate saturated plugs were calculated together with the mud resistivities before and after aging. The results can be seen in Table 20, Appendix B.

6.3.2 Measurement of Magnetic Characteristics

The most important magnetic properties can be obtained from the measurement of incremental permeability at various polarizing inductions. The induction produced by current in a primary winding is observed in terms of the fluxmeter deflection as the primary current is changed suddenly or reversed. The use of a ring sample eliminates the possibility of errors due to air gaps. To obtain uniformity of magnetizing force throughout the test sample, the ratio of the outside diameter to the inside diameter of the ring should be approximately 1:2.

The properties of magnetic materials are usually described by a magnetization curve plotted between the magnetic induction, B, in the ring sample against the magnetizing force H. In this case the value of B will give a measure of the amount of the magnetization and H represents the magnetizing force required to produce the magnetic induction B. The magnetizing force is usually expressed in oersteds and the magnetic induction in Henrys.

The ease with which a magnetic material can be magnetized is measured by the ratio B/H called the magnetic permeability (μ). The values of the permeability obtained for a given material depend on the methods and conditions of measurement as measured by the Standard Methods of Test for magnetic material (ASTM Designation A34), it is the ratio of the maximum value of induction to the maximum value of the magnetizing force. In dealing with many media, it is often convenient to speak of the relative permeability μ_r as

$$\mu_r = \frac{\mu}{\mu_o}$$

where μ_r = relative permeability (dimentionless ratio)

μ = permeability (henrys meter)

μ_o = permeability of vacuum

In this study μ_o was considered to be the unity (i.e., non-magnetic material), in actual fact air is paramagnetic and has a relative permeability of 1.000,000 4.

Based on this information an experiment was designed to measure the relative permeability of the drilling fluids and the weighting materials in dry conditions. A hard plastic ring-shaped tube, with a uniform windings of 800 turns of insulated wire, was filled with the fluid to be tested. The wire terminals were connected to a Q-meter circuit (see

Appendix D). The voltage was applied through the Q-meter circuit to the toroid inductance in series with a capacitance. By varying the capacitance value C and adjusting the frequency value (f) at a maximum charge density (Q), a maximum voltage was produced across the capacitance, then

$$WL = \frac{1}{WC} \quad \text{for resonance}$$

where $W = 2\pi f$

$$L = \frac{1}{4\pi^2 f^2 C}$$

then $C = 4\pi^2 LC$

The inductance $L = \dfrac{\mu_o N^2 r^2}{2R_s}$ for a toroid

where N = number of turns

R_s = radius of toroid

r = radius of cross section

From the above equations we can drive the relationship

$$\frac{1}{f^2} = \frac{4\pi^2 N^2 r^2}{2R_s} \mu_o C$$

The magnetic permeability for the iron oxides weighted muds and barite mud were obtained from the slopes of the straight line relationship between $\frac{1}{f^2}$ versus C. The magnetic permeability of the weighting material in the powder form was also determined. The permeability calculations together with the tabulation of the readings can be seen in Tables 21 and 22. The graphical presentation of results is shown in Figures 34 and 35 in Appendix B.

6.4 Determination of the Filtration Properties and Fluid Loss

The filtration and fluid loss properties of drilling fluids have a direct impact on the penetration rate, hole problems, formation damage and differential sticking problems. Previous studies by Kreuger and Vogel, 1954, Glenn and Slusser, 1957 and Kreuger, 1973,[40] indicated that the degree of formation damage was observed to be a function of the time of exposure and total volume of filtrate through the rock. Keelan and Koepft, 1977,[37] evaluated the formation due to drilling fluids and concluded that all muds cause damage and that permeability reduction is the result of mud filtrate solids. Return permeability after remedial work was found to decrease as fluid loss increased.

In formulating a compatible drilling fluid, desirable characteristics are:

1. A high spurt loss to maximize the penetration rates and to quickly establish an effective impermeable filter cake.

2. A low ultimate fluid loss to avoid hole instability and formation damage.

The drilling fluids are usually tested for filteration and fluid loss properties using the static procedures. These are considered to be the only practical means of obtaining fluid loss measurements in the field. The measurements are influenced by several factors including temperature, particle type and size, pressure and time.[24,25] The equation governing filteration under static conditions is

$$Q_w^2 = \frac{2kPA^2}{\mu} \times \frac{Q_w}{Q_c} t$$

where A = area of the filter cake in square centimeters

k = permeability in darcies

P = differential pressure in atmospheres

μ = viscosity of the filtrate in centipoises

Q_c = volume of the filter cake in cubic centimeters

Q_w = volume of the filtrate in cubic centimeters

t = time in seconds

q_o = the zero error

Larsen, 1938, found that the filtrate volume that would accumulate in 30 minutes can be predicted from the volume, Q_w observed at time t_1 from the equation

$$Q_{w30} - q_o = (Q_{w1} - q_o) \frac{t_{30}}{t_1}$$

This means that by doubling the filtrate volume measured at 7.5 minutes, the 30 minute standard filtrate volume can be predicted. In many cases, the discrepancy that might occur can be attributed to the spurt loss.[24]

6.4.1 API Low Temperature Test

The testing equipment and testing procedure is specified in API-RP-13. The laboratory model, Fann, 6 cell filter press was used for measuring the filtration properties of iron oxides weighted muds together with barite weighted mud.

The test was also applied to improved and aged muds to investigate changes in filtration properties as a result of mud treatment and aging. The Fann model equipment has a built-in screen mounted on the base of the pressure cell with machined groves to facilitate the run-off of the filtration and minimize the hold-up volume.

The standard dimensions of the filter press used are: filtration area, 7.1 in^2 (45.8 cm^2); minimum height, 2.5 in. (6.4 cm); and standard filter paper. A pressure of 100 psi from a nitrogen cylinder was applied at the top of each cell. The amount of filtrate discharged in 30 minutes and thickness of the filter cake were measured after washing off the excess mud.

To determine the corrected fluid loss and mud spurt, the 2, 5, 10, 15, 20, 25 and 30 minutes fluid loss volumes are plotted against the square root of time as expressed by the equation

$$Q = C \quad t + \text{spurt loss}$$

where Q = filtrate volume

t = time

C = constant

The results for filtration tests for untreated muds, aged muds and polymer treated muds using iron oxides and barite as weighting materials are shown in Tables 23, 24, and 25. The graphic presentation and determination of corrected fluid loss are shown in Figure 36. The effect of mud aging and grain coating are indicated in Figures 37 and 38 and will be explained in the analysis of results (Chapter IIX).

6.4.2 High Temperature, High Pressure Filtration

A special high pressure, high temperature filter press is designed to test muds at elevated temperatures and pressures. The equipment (shown in Appendix D) consists of heating a well with a thermostat, 250-ml filter cell, and pressure unit. The filteration characteristics obtained with this apparatus more truly represents the actual conditions in the wellbore.

The standard procedure in the API-RP 13 was developed in order to allow for testing the compressibility of the filter cake. The filter cakes which are highly compressible will be compacted as the differential pressure is increased, resulting in a reduction of cake permeability. Testing was applied to fresh batches of mud, aged muds and improved (treated muds). The testing procedure was as follows:

1. The heating jacket was preheated to 200°F by adjusting the thermostat.

2. Mud was preheated while stirring up to 120°F.

3. The mud cell was filled to one inch from the top and placed into the heating jacket with both valve stems closed.

4. Both pressure unit and bottom receiver were locked in place, then a pressure of 100 psi was applied through the top valve while the mud was heated.

5. When the required temperature was reached, the bottom valve was opened and the pressure was increased to 600 psi.

6. Filtrate was collected for 2, 5, 10, 15, 20, 25 and 30 minutes.

7. To test the compressibility of the filter cake:

    a. the differential pressure was doubled

    b. filtrate was collected for 5 minutes

    c. a comparison between the collected filtrate for the last 5 minutes to the difference between the cumulative volumes at 25 and 30 minutes interval expresses the efficiency of the mud cake. A sharp increase indicates an incompressible mud cake due to lack of solids control.

The testing cell is so designed that the cross-sectional area of the cell is only half that of the API low temperature cell. In order to maintain consistency, the HT-HP fluid loss volume must be multiplied by a factor of 2. A testing temperature of 200 °F was selected for the HT-HP filtration tests, being the aging temperature of the mud for performance compatibility tests. The compatibility tests use shale cuttings from the Atoka Formation which was found, from the core log records, to have an average borehole temperature of 200 °F at 12,000 feet depth.

Detailed results can be seen in Appendix B, Tables 26, 27, and 28. Fluid loss-time relationships are represented graphically in Figure 39 for untreated mud. The effect of aging with shale cuttings and polymer treatment are expressed graphically in Figures 40 and 41. An overall correlation of fluid losses for untreated, aged and treated drilling fluids is represented in the block diagrams, Figures 42 and 43.

6.5 Procedures for Measuring Abrasivity of Drilling Fluids

Water-based heavy drilling fluids weighted with iron minerals are expected to encounter problems of abrasion which will vary at different parts of the circulating system. The abrasivity of the drilling fluid is due mainly to the hardness of the weighting material, size and shape of the mineral grains. The wear effect at various parts of the circulating system is a function of the drilling fluids composition and the shear rate at the specified part of the system. The shear rate varies significantly from the drill pipe (100 to 500 sec^{-1}) to the bit (10,000 to 100,000 sec^{-1}), and this will eventually change the wear effect caused by abrasive drilling fluids.

Considering the significant contradiction in conditions at various places downhole, laboratory experiments were designed to test the wear effect for the iron weighted mud at temperatures and shear rates as close as possible to those encountered at a specific part of the circulating system. The widely accepted barite level of abrasiveness is considered as the datum level of correlation.

6.5.1 Rolling Technique for Combined Aging and Abrasion Tests

The abrasion rates of the various muds were measured by calculating the weight loss percent of a coupon held rigidly inside a specially designed mud cell. The cells are filled with iron minerals weighted and barite weighted muds and rolled inside a thermally controlled roller oven. This technique provides an excellent method of measuring the abrasivity of the drilling fluids under combined heating and agitation conditions. It simulates conditions that mud experiences in being circulated down the hole and back to the surface pit.[18,29] The roller oven has proved to be a valuable tool in measuring the abrasivity as the mud ages under controlled downhole conditions of heat and agitation. The specially designed stainless steel coupons and mud cells used in the tests are shown together with the roller oven in Appendix D. The testing procedure can be summarized as follows:

1. The reological properties of the three basic iron oxide weighted and barite weighted drilling fluid batches were determined.

2. The four mud cells (400 ml volume each) were filled with a safe volume of mud for the temperature at which the samples will be tested.

3. A coupon, of predetermined weight is fixed in place at each cell.

4. Cells were then placed in the roller oven and heated to the desired temperature while being rolled for 48 hours.

5. Cells were removed and air cooled to ambient

temperature then opened and coupons removed.

6. The mud samples were tested for rheological properties.

7. The coupons were weighted to four decimal figures using an electronic analytical balance.

8. Tests were repeated at gradually increasing temperatures from 140°F to 280°F at 20°F intervals.

The results, listed in Tables 30 to 33, give the weight loss and the weight loss percent of the investigated mud systems with increasing temperature. A correlation between the abrasivity of the iron oxides weighted mud and barite mud during 14 days of rolling and aging can be seen in the bar diagram, Figure 44.

6.5.2 Abrasion Tests Under High Shear Rates

To produce laboratory simulated abrasion effects and particle breakdown, two different types of blenders were used. Both types have the advantages of: (1) producing high shear rates between 10,000-20,000 rpm and (2) possessing removable electrically insulated blades. Temperature simulation was achieved by preheating the mud, adjustment of stirring periods and using water baths when necessary. This procedure was effective in adjusting the temperature throughout the experiment between 140-180°F without causing any evaporation. For each drilling mud, a new blade was used to measure the abrasive activities. The weight loss was measured to four decimal points after each stirring period.

Both the Waring and Osterizer blenders have a shear rate of approximately 20,000 rpm (34,000 sec^{-1}) and can create the type of shear that can be expected at the bit. The weight loss can be calculated from the coupon weight measurements before and after the tests. The weight loss percent are tabulated for each mud type in Tables 34 to 42 (Appendix B). A relationship between weight loss percent and stirring periods was plotted to show the abrasion level of each of the iron oxide weighted mud compared to the barite mud abrasive level.

The tests were found very useful in simulating bottom hole, high shear conditions and producing a convenient, precise means of measuring the abrasivity at selected shear rates. The conditions can be adapted and adjusted to any particular field case. Furthermore, the experiment provides useful information about the role of the mineral grain shape in abrasion and the possible means of cutting the abrasivity down to the widely acceptable barite level either mechanically or by using additives. This will be explained in Chapter VII.

6.6 Tests for Mineral Grain Attrition

The importance of measuring the mud components, grain-to-grain attrition and the effect of the particle progressive comminution on the mud performance has been discussed in Chapter I. The high shear abrasion tests combined with the rolling technique provides a complete simulation of the conditions effecting the drilling fluids during its complete trip.

The experiment was designed to measure the amount of desintegration each of the iron oxides undergoes in comparison to barite. The procedure was performed as follows:

a. Samples were taken from each type of mud and tested for particle size distribution using fine sieve analysis for particles larger than 38 microns. Smaller particles were tested using the hydrometer analysis as explained in the American Standard Specifications for Transportation Materials, 1978. Samples were also obtained for examination under the scanning electron microscope.

b. The drilling fluids after having been subjected to high shear rate for 80 minutes are transferred to the aging mud cells and agitated in the roller oven for 14 days. The temperature was changed regularly from 100°F up to 240°F at a rate of 10°F every 24 hours.

c. The various muds were again sampled and tested for particle size distribution.

d. The samples were collected after the hydrometer analysis screened and separated on number 400 mesh and prepared for examination with the electron microscope.

Results of testing are furnished in Tables 43 to 46 (Appendix B). A graphical presentation of the particle size distribution before and after tests is provided on semilog paper. The graphs show the change in particle size distribution that may be expected with each mud as a result of utilization. The effect of grain coating on the particle attrition of barite is demonstrated in Figure 52.

6.7 Performance Compatibility Tests

The main purpose of the test is to ensure the success of the mud formulation. The test is developed in this research to select the most competent drilling fluid to drill through the Oklahoma Atoka Shale formation. The Atoka Shale was recorded as causing drilling problems with barite mud on several occasions. The effect of the drilling fluids-shale interaction for six various types of mud had been examined. A periodic testing of the rheological properties, pH and solids content was conducted as the drilling mud was provided with new shale cuttings after the old aged cuttings were removed. The mud screening and separation of aged cuttings is a simulation of the desanding procedure which usually takes place on the surface at the end of each round trip. The addition of new cuttings represents the new cuttings produced as the drilling progresses.

The testing temperature was selected on the basis of actual drilling data obtained from borehole logs for drilled hole, Shell 1, Dipple and other drilled holes in eastern Oklahoma. The formation temperature of the Atoka Shale between 11,000 to 12,000 feet depth was found to vary from 180-200°F. Therefore a testing temperature of 200°F was selected for appropriate borehole temperature simulation.

The test procedure as performed in this work can be summarized in the following.

1. Known quantities of the Atoka Shale (50 gms) of given size range taken between number 4 mesh maximum and number 10 minimum mesh size (i.e., between 4.7 and 1.65 mm) were prepared.

2. Drilling fluids type A, B, C, D, AC and BD of known initial properties were placed in the 350cc volume mud cells.

3. 50 gm of the prepared shale, of predetermined initial methylene blue capacity, was added to each cell.

4. The mixture was aged in the roller oven under dynamic conditions at 200°F for 24 hours.

5. The drilling fluids were removed and screened on number 30 mesh screen.

6. The recovered shale was dried out, weighted to 0.1 gm accuracy and tested for methylene blue capacity.

7. The aged mud was tested for rheological properties and gel strength.

8. New fresh shale cuttings (50 gm each) were added to each mud after screening and steps 2, 3, 4, 5, 6 and 7 were repeated for a total duration of 7 days.

A comparison between the rheological characteristics of the various muds after each test and the initial and final properties for each type of mud will proivde useful information about the actual field performance of each mud. The changes in the shale-mud interaction and methylene blue capacity values indicate how a rock from a particular formation reacts with each mud. Based on results of these tests a selection of the most compatible drilling mud to drill some specific formations can be justified.

The graphical presentation for rheological parameters relationships (shown in Figures 53, 54, and 55) were used effectively to analyze the results and to investigate the degree of stability as the mud ages.

CHAPTER VII

TECHNIQUES FOR IMPROVING THE PERFORMANCE OF DRILLING FLUIDS

Testing drilling fluids weighted with iron oxides and barite revealed the nature of the problems associated with each weighting material. In this chapter these problems are dealt with by treating the mud and reformulating it in order to produce an improved mud type that eliminates or reduces the previous defects, thus rendering it feasible to use.

The problems uncovered by the experiments performed in this study are summarized as follows:

1. All iron oxide weighted, water base muds have shown better rheological properties and more compatible performance than barite (as discussed in the analysis of results). However, because of their intrinsic hardness, iron oxides have shown high levels of abrasiveness under simulated bottom hole conditions well above that of barite. This is indicated by the graphical representation of the relative abrasivity of the various muds shown in Figure 45.

2. Ilmenite and itibirite weighted mud exhibited undesirable filtration properties under both low and high temperature conditions. With the exception of the K-M hematite

mud, all drilling fluids made of iron oxides possess compact but incompressible mud cakes under high temperature and high pressure conditions. The filtration characteristics of the iron mineral systems were generally inferior to untreated barite mud. However, all muds investigated, including barite mud, have displayed an inevitable need for filtration control due to the consistent high fluid loss obtained by the high pressure, high temperature filter press. Undesirable filtration properties may cause extensive damage to productive formations unless fluid loss control materials are added.

3. The itibirite has a tendency to precipitate in the high density mud systems (above 15 ppg), and shows low bentonite concentrations (i.e., below 5 percent by weight).

4. The soft characteristics of barite makes it exposed to the attrition of the mineral grains under the effect of the high shear rates produced at various parts of the drilling circulating system. The reduction in particle size results in a significant increase in the apparent viscosity, and in an increase in the solids content which may reduce the penetration rate and disturb the mud performance.

5. Iron minerals weighted drilling fluid systems have shown excellent thinning properties at high shear rates, as indicated by the viscosity profiles (Figures 19 to 24). At low shear rates the effective viscosity (i.e., 3 and 6 rpm) increases to more than 1000 centipoises. This phenomenon requires an effective, long lasting treatment. The viscosity

increase is characterized by an abrupt change in the slope of the effective viscosity profile towards the low shear rate portion. This is attributed to a significant variation of the (n) factor of a pseudoplastic fluid model and makes the prediction of the mud behavior at low shear rates difficult and uncertain.

7.1 Techniques for Treatment of Abrasiveness

Abrasion results in a severe localized attack on the metallic and rubber components of the drilling equipment. Damage usually appears as smooth groves or washout of pipes and valves of surface equipment used for mud treatment or circulation. When combined with corrosion, abrasion can cause a complete destruction of metals. Impingment attack caused by the abrasion-corrosion process occurs after the protective films are eroded and the clean metal surface becomes exposed to the corrosive elements.

The presence of abrasive suspended materials, together with other factors like high circulating velocities, are responsible for the destructive erosive effect on the drilling equipment. Therefore, it is essential to find a means of reducing the abrasion to an acceptable level.

7.1.1 Mechanical and Chemical Techniques

The grain size and grain shape of the suspended particles play an important role in the abrasivity of the drilling mud, especially at relatively low shear rates (i.e., drill pipe and annulus). The effect of grain shape is shown by the scanning electron micrographs in Appendix C. One can see that barite grains possess more sharp edges per grain than any other weighting agent. At high shear rates itibirite, with its highest hardness and sharpest edges, was found to be the most erosive followed by ilmenite. Despite the hardness of the K-M hematite, its relative abrasivity was only 35 percent above the barite level. The reduced hematite abrasivty can be explained by the subspherical grain shape of the material. The distinguished grain shape of the K-M hematite is the result of the previous chemical and physical processing operations. The scanning electron micrographs shown in Figure 58, Appendix C indicate that shearing and aging effects had a milling action on all weighting agents. This effect results in grain rounding and flattening of the abrasivity-stirring time curves. Hydrometer analysis indicated that the amount of fines produced in the attrition process varies from one weighting material to another depending on the physical characteristics of each mineral (see Figures 49 to 52). The outcome of the shear and aging testing techniques can be applied to improve the grain shape of the high abrasive minerals. By milling the highly abrasive itibirite and ilmenite minerals, during the mineral processing and preparation procedure, improved grain shape type minerals can be obtained. The fines produced during the processing being rounded themselves can be used to form the fine fraction of the weighting material blends. Mechanical and chemical processing can reduce the relative abrasivity by 45-50 percent (i.e., to the K-M hematite level, Figure 45). This can be further reduced to the acceptable barite level or below it by the polymer grain coating technique, as will be explained in the following subsection.

7.1.2 Treatment of Abrasivity by Lubrication Technique

As a result of the irregular nature of surfaces, two surfaces brought into contact will touch only in isolated regions. The pressure at these touching points is sufficient to cause deformation leading to multiple contacts, and friction becomes confined to a real area that is much smaller than the apparent area. Rather high local temperatures can develop during rubbing between the interfaces. The consequence is that there is also rather high pressure, as the two surfaces are brought together. The pressure is extremely large at the initial few points of contact and deformation immediately occurs to allow more to develop. The abrasivity depends on the coefficient of friction which is a function of the relative velocity of the two surfaces. At low speeds the effect is usually small and insignificant.

Lubrication can be used to produce a separating medium, called lubrication boundary, between the interacting surfaces. Restricted conditions of lubrication are:

1. The oil film must be thick enough so that the surface regions are independent of each other.

2. The lubricant efficiency will depend on the hydrodynamic properties, especially the viscosity of the oil.

This means that changes in speeds or viscosity can make the film between the interacting surfaces thinner and increase the contact between the two surfaces. The friction between the two surfaces will eventually rise from low values possible for fluid friction to some value that usually is less than that for unlubricated surfaces.

The UK patent application GB2066876,[62] published by the Patent Office, London, 1981, is the only published work on abrasive drilling fluids. The patent applies to weighting agents having a hardness of at least 4.3, preferably 4.5, on the Moh's scale for use in water-base drilling fluids. The patent claims that with the use of a water-dispersible lubricant (i.e., a long chain fatty acid or vegetable oil containing such an acid), with a surfactant no abrasion problems will be encountered on the drill string, bit, etc. The UK patent proposes ilmenite and non-micaceous hematite as suitable weighting agents.

The patent used a drilling mud weighted with specular hematite to test the abrasive characteristics and the effect of adding the proposed anti-wear agent. A Hamilton Beach mixer operating at 15,000 rpm was used to produce laboratory simulated abrasion effects. The mixer blade was examined periodically for weight loss after various stirring periods. The muds were immersed in a cooling bath during the mixing to avoid overheating.

The results were represented by the graph shown in Figure 57. It can be seen from the graph that a reduction in the abrasivity of less than 40 percent was achieved by the UK patent. The testing procedure, as stated in the patent, lacks an efficient simulation of downhole conditions for the following reasons.

1. The temperature of the testing, which is extremely important in boundary lubrication, was not specified. However, it was stated that a cooling bath was used to avoid overheating which indicates that the temperature was kept low.

2. The Hamilton Beach mixer cannot provide the type and rate of shear expected at the bit nozzles due to the wide clearance between the blades and the sides.

The published results indicated that the antiwear agent had undesirable effects on the high temperature, high pressure filtration properties. The filtrate volume increased from 40 ml to 65 ml when the antiwear agent was added.

It is evident from the studies by Hardy[2] that under boundary lubrication conditions like those produced by fatty acids, the reduction in the force fields is the result of adsorbed films. The area of actual contact during the abrasion process is a small fraction of the total area. Therefore, only occasional small patches of film are put under mechanical pressure. As a result, the film molecules tend to escape from the pressured region to adjacent normal regions causing sharp thinning of the lubrication films at the contact area and deterioration of the lubrication effect. Frewing[2] found that on heating, the friction between the two lubricated surfaces rose sharply. The reason is likely to be a significant change in the hydrodynamic properties of the lubricant, mainly viscosity. These factors cause uncertainty and inconsistancy with regard to the efficiency of the UK patent.

7.1.3 Polymer Coating Technique

In contrast to the situation in lubrication where the contact between the surfaces occurs on small patches of the film, adsorption occurs over quite large areas of the actual interfacial contact. Good coating of grains is achieved when the adsorbent spreads on the surface uniformly and effectively.

Polymers are generally polydisperse, and their adsorption is more that of a multicomponent system. It takes several parameters to describe the state of the polymer at an interface. These include the number of points of attachment, the horizontal spread and the thickness. These parameters determine the coating efficiency and the behavior of the suspended particles in the drilling fluids.

A number of polymers were selected for possible use as an additive to abrasive drilling fluids to improve the mud abrasiveness. The most successful polymers among the tested material were found to be the following.

1. Lo-sol polymer

This polymer is produced by Amoco Chemicals. It is normally used to extend the yield of bentonite in fresh water systems. The polymer provides higher viscosity with a given amount of bentonite to reduce the colloidal solids content of the mud. When small amounts of this polymer were added to the abrasive, iron mineral weighted mud, the abrasivity was reduced by 50 to 60 percent of the total relative abrasivity above the barite level. The effectiveness of the lo-sol polymer as an antiwear agent can be seen in Figures 46, 47 and 48. The polymer was also found very effective in decelerating the barite grain attrition as seen from the results of the hydrometer analysis on improved barite mud shown in Figure 51.

2. H-42-10-4 polymer flocculant (Select-Flocculant)

Manufactured and marketed by Amoco Chemicals. It is a fresh water soluble polymer, normally used to flocculate drilled solids to maintain solids free drilling fluids. The polymer flocculates drilled solids without flocculating bentonite. When added to the abrasive mud types, the mud abrasivity was reduced significantly. The effect of the H-42-12 polymer was equivalent to the Lo-Sol as an antiwear agent when used with abrasive muds. Ordinary thinners like Barafos and Desco were found to be effective in reducing the viscosity to approximately the original untreated mud viscosity. Other chemicals and thinners may also be used.

3. Drispac

Drispac is a polyionic cellulose derivative, manufactured and marketed by Drilling Specialities Company. The polymer is despersible in water-base mud. It is nonbiodegradable and does not require a preservative. Drispac is normally used as a dual purpose additive for both fluid-loss control and viscosity extender. The material selectively flocculates the drilled solids and helps its separation. Experimental results on Drispac as an antiwear agent indicate an effective reduction in abrasivity of approximately 20 percent.

Abrasion tests performed on the various abrasive muds have proved the effectiveness of both the Lo-Sol and Amoco selective (H-42-10-4) polymer as antiwear agents. Tests for comparative attrition proved that these polymers can also be used with soft minerals like barite and galena as protective agents for decelerating the attrition of the mineral grains. The nature of the adsorption of the polymers on the crystalline interfaces of the mineral grains was verified by the interpretation of the scanning electron micrographs.

7.1.4 Scanning Electron Microscopy

The University of Oklahoma High Performance Scanning Electron Microscope facility was used to examine samples taken from the investigated mud types. The microscope is of the Etec Autoscan system with a wide magnification range varying from 5X to 240,000X measured from the surface of the sample for more accurate, direct magnification readout. Microphotographs have been obtained for the following purposes:

1. To examine the changes in the shape of the mineral grains due to shearing and aging effects.

2. To evaluate the role of the grain shape in the mud abrasiveness.

3. To study the nature and limitations of the polymer adsorption on the weighting material crystal faces.

The study required that the fluid phase be removed from the specimen before placing it in the instrument. A combination of air drying and oven drying was used to dry the specimens without disturbing the original structure.[30] Before drying, the mud samples were screened on the 38 micron size sieve (number 400 mesh). Both materials greater and smaller than 38 micron were washed with distilled water and dried out under a maximum temperature of 180°F. After drying the specimen, the grain surfaces were exposed for study by applying an adhesive to attach the specimen to a special nonconductive substrate. It was necessary for nonconductive minerals like barite to have a suitable conducting path and, therefore, a thin layer of gold was sufficient to eliminate the problems associated with charging.

The electron micrographs can be seen in Figures 58, 59 and 60 in Appendix C.

7.2 Composition and Properties of Improved Mud Types

Iron minerals weighted drilling fluids have been reformulated as a result of the testing program and electron micrographs interpretation. In the mud reformulation the following limitations were considered.

1. The formulation of mud weighted with abrasive material must incorporate adequate quantities of either lo-sol or select flocculant polymers. Experimental results indicated that an amount of 1.5 percent, by volume, of these polymers will be adequate to reduce the abrasivity of a 14 ppg ilmenite or itibirite mud by approximately 60 percent. The same amount of polymer was capable of reducing the abrasivity of the K-M hematite mud to the barite mud level. The relative abrasivity is expected to go down further at shear rates below 20,000 rpm. In field terms the appropriate volume of the specified polymers, as prepared by Amoco Chemicals, is approximately 0.225 gallons per sack (100 lb) of the abrasive weighting agent in the mud (i.e., 1.85 liter per 100 kg of weighting material).

2. Thickening of the weighted muds as a result of adding the antiwear polymer was treated by adding dispersants to the mud. At the pH of 8 to 9.5 of the untreated mud, polyphosphate additives like Barafos functioned efficiently in reducing the apparent viscosity. Modified tannin (Desco) was occasionally added in small amounts to the mud for its dual effect as a dispersant and for the pH control. Lignosulphonates could also be used for viscosity control.

3. The control of the mud rheological and abrasive properties was not achieved at the expense of the filtration properties, as was the case with the UK patent.

4. The yield point/plastic viscosity ratio was kept

in an acceptable range. High YP/PV ratio present settling fines in pits and increase the solids content in the mud. The composition and rheological properties of the improved mud types F, G, FH and GI can be seen in Tables 12a, 12b, 13 and 14. The effective viscosity profiles are shown in Figures 19 to 27, Appendix B.

The properties of the treated muds as verified by the evaluation testing program are summarized as follows.

1. The improved ilmenite and hematite weighted muds possess abrasive characteristics slightly above the barite level at the bit nozzles. In other parts of the circulating system the abrasivity of iron oxides weighted muds is equivalent to barite.

2. The abrasivity of the treated K-M hematite weighted drilling fluids are either equivalent or below the barite abrasivity at any part of the drilling circulating system.

3. The filtration properties of all treated muds are excellent and sufficient to avoid most well bore problems. The low ultimate fluid loss eliminates any possible formation damage, and the thin compressible mud cake will help in avoiding stuck pipe problems.

4. The polymer additives used can impart maximum viscosity at a minimum solids content. These polymers have provided the mud with better suspension characteristics for weighting agents. The muds were aged under static conditions at ambient temperature for 10 days without suffering from settlement.

5. The treated muds have slightly higher apparent viscosity values at high shear rates, whereas at low shear rates the shear stress was lower. As a result, the general range of shear stresses in various segments of the circulating system has improved. The effective viscosity in the annulus indicates improved hole cleaning and values for the n factor are less liable to significant changes along the log log viscosity profile.

6. The treated mud exhibits lower gel and yield values due to the nature of the dispersed mud systems. The polymer additives in the mud will still provide adequate carrying and suspension capabilities. This advantage eliminates the need for a higher bentonite concentration which may increase the solids content and eventually decrease the rate of penetration.

7. Treatment of barite weighted mud by polymer additives has proved to be a dual purpose process. It reduces the mud abrasiveness, and it decelerates the progressive comminution of the mineral grains. The treatment can be applied to other soft minerals utilized in drilling fluids, as galena (hardness 2.5-2.7 on the Moh's scale).

CHAPTER VIII

INTERPRETATION AND ANALYSIS OF RESULTS

The evaluation of the iron oxides weighting material incorporated various iron minerals. The variation in chemical composition and physical characteristics of these minerals provide a range of variation in their performance as weighting materials for drilling fluids. However, each one of these minerals have exhibited certain characteristics that distinguishes its performance over all other weighting material for some particular job. This study establishes the basis for preparing new mud programs and provides the comprehensive information needed in planning new wells and selection of competent mud. Mud programming can be conducted based on the pertinent mud properties reported in this work.

To be able to properly evaluate and analyze the data on each mud type, a number of important parameters have been selected and type curves generated for each mud type to enhance the interpretation of data. The investigated types of drilling fluids were formulated such that;

1. to avoid and overcome costly drilling problems that may be encountered during drilling as a result of using

incompetent drilling fluids,

2. to achieve the implementation of an effective drilling fluid program which enables an overall optimization of the drilling operation,

3. to minimize the consumption of mud materials and ensure the availability of the material supplies at all times,

4. to proivde the type of mud most suitable for drilling a specific formation without excessive borehole problems.

8.1 Assessment of Iron Weighted Mud Systems

For mud formulation and preparation a number of assessment charts have been designed for each type of weighting agent based on the specific gravity of the mud components and the volumetric weight of each mud. A given volume of any one of the iron minerals (i.e., ilmenite, K-M hematite and itibirite) weights more than the same volume of the API standard barite by an amount varying from 9 percent for ilmenite to more than 20 percent for the itibirite and K-M hematite. It can be ssen from Figure 13 and Figure 14 that to mix an equal density water base gel mud with each of the weighting agents will require less amounts of iron oxides than barite. The difference between the weights required for each type of mud and barite will depend on the specified density of each mineral and the required mud weight. Charts (Figures 17 and 18) indicate that the barite weighted mud always contains a higher percentage of solid material by volume than any of the iron oxide mud systems. Low solids in the mud can improve significantly the rate of penetration. Drilling tests have proven that although all solids have an adverse effect on penetration rates, fine particles less than 1 micron in size are 12 times more detrimental than larger particles.[18,24] Aged barite mud systems contain more fine particles, of the colloidal size, than any of the aged iron minerals weighted system due to the progressive comminution of the barite particles. Iron minerals being relatively hard are less susceptible to grain attrition and less fines are produced as a result of aging and shear. The results of the hydrometer analysis, shown in Figure 49 to 52, indicate that 22 percent barite particles were ground down to a very fine size and become a colloidal suspension, whereas only 6 to 10 percent fines smaller than one micron were obtained by subjecting the iron minerals weighted mud to the same conditions of agitation and shear. As the particle size of the material decreases, the surface area is increased and eventually more water is adsorbed by the grains. The adsorption has the effect of concentrating the solids in the system and results in an increase in plastic viscosity and yield point.

It is necessary in weighted mud systems to remove the fine solids and to add other high quality commercial product to improve the mud rheology. This process has to be carried out with the barite system much more frequently than with any of the iron oxides weighted systems.

8.2 Rheological Testing Results

An interpretation of the log log viscosity profiles for the undispersed mud systems at ambient temperature indicated that a wide variation in rheological behavior has been exhibited by the various tested systems. All muds exhibited excellent shear thinning properties at high shear rates. All mud types A, B, C and D will perform efficiently at the bit. Barite mud imparts high effective viscosities at other parts of the circulating systems including drill pipe, drill collars, solid removal equipment and annulus. The barite mud viscosity profile also shows the steepest gradient that is typical of a high solids content mud type. The viscosity profile for the ilmenite and itibirite (types A and C) muds togethter with the ilmenite-itibirite (type AC) mud have displayed similar rheological characteristics to barite muds. The performance of mud types (A, D and AC) imposes the necessity of chemical treatment or use of dispersants. On the other hand, viscosity profiles for both the K-M hematite mud (type B) and the barite-K-M hematite mud (type BD), show good rheological characteristics for undispersed mud systems. The values obtained for effective viscosities are capable of avoiding drilling problems in all parts of the circulating system.

The distinguished spherical grain shape of the K-M hematite may have an influence on the mud rheology. The spherical shape gives rise to the least surface area possible for any given volume and therefore a minimum water adsorption will occur. This explains the relatively regular change in the apparent viscosity at varying shear rates. A better evaluation of the rheology can be seen when combining the testing results at low and high temperatures. The results of the sophisticated high temperature viscometer is extremely important and decisive as regards to its environmental simulation. The measurements provide a comprehensive description of the viscosity-temperature relationship for the tested mud systems. The data analysis technique was accomplished by converting the shear stress readings into apparent viscosities in cps. The plotting of the apparent viscosity as a function of temperature enables determination of the variation and stability of the viscous properties of the investigated mud types. The apparent viscosity-temperature curves shown in Figures 29 to 32 can be used to treat the mud effectively. The results indicate that the barite weighted mud is more susceptible to viscosity fluctuations at elevated temperature than the iron minerals weighted mud types. Maximum apparent viscosity values were obtained between 250 and 300°F. All mud types A, B and C displayed favorable thermal characteristics and capacity of drilling deep holes and high temperature formations. The barite mud with its high viscosity values and maximum viscosities as low as 200°F, would require extensive chemical treatment at the surface during each circulation.

8.3 Interpretation of Gel Strength Data

The results of the change of gel strength with time for nondispersed drilling muds are plotted in Figure 28. Each one of the tested mud systems is characterized by a unique gel type. The ilmenite weighted mud system exhibits an intermediate 10 second value building up to a high level. This type is called progressive gel. The itibirite mud shows a combination of progressive gel at the first 20 minutes changing to a flat type gel afterwards. The K-M hematite mud system started with a low to medium 10 second gel strength which builds up to medium strength in ten minutes then increases slowly. The gel strength of the barite mud started at an intermediate 10 second value then built up progressively in the next 10 minutes. Identical gel strengths were obtained for 20 minutes and thereafter. The range of gel strengths is indirectly related to the range of the yield value, therefore chemicals which help decreasing the yield value will decrease the gel strength range. Such chemical treatment should render the progressive gel to good gel. The improved mud types F, G, H and I show significant improvement in gel strength. A 10 second/10 minutes gel for ilmenite mud changed from 28/92 to 6/12. For the itibirite mud the 10second/10 minutes gel improved from 28/44 to 9/20 for improved mud including a polymer antiwear agent and a thinner. A significant improvement in gel strength of other mud types was also observed as a result of mud treatment. The composition of the improved mud types can be seen in Tables 12a, 12b, 12c, 12d, 13 and 14, Appendix B.

Temperature-gel strength relationships for iron minerals weighted drilling fluids, shown in Figure 33 indicate good characteristics that favor the use of the mud for deep drilling. Maximum gel values were obtained between 200 and 250°F. The gel strength exhibited by the barite mud was extremely high and hard to control. This gel performance may endanger the drilling operations if the proper treatment is not provided consistently.

8.4 Analysis of Electric and Magnetic Measurements

The resistivities of the mud and the mud filtrate are important parameters in the well logging operations. Results of mud resistivity values for iron mineral mud systems show slightly lower values than the barite mud system. Results indicate that no special treatment will be needed. The mud resistivity of the iron oxides weighted muds of 7.50 to 9 ohm·meter (Table 20) is very close to that obtained from barite mud of 9.58 ohm·meter. No dramatic change in mud resistivities was observed after the mud was aged. Mud resistivity values obtained for fresh and aged mud are low enough to provide good conductive media for well logging measurements. Filtrate saturated core resistivities of 45.7 to 70.65 ohm·meter show similar results to barite mud and deviated strongly from the value obtained for the 30,000 ppm brine saturated cores of 4.53 ohm·meter. Therefore, it can be stated that no well logging problems will be encountered with the use of the iron minerals as weighting agents in drilling fluids. The difference in resistivities between the mud filtrate and connate water is sufficient to eliminate any doubt about interference with the spontaneous potential readings.

It is claimed by several industrial sources that the iron oxides being ferromagnetic material, will interfere with the electric and magnetic well logging tools. Testing results shown in Tables 21 and 22 and plotted in Figures 34 and 35, indicate that the iron oxides weighted drilling fluids possess a relative magnetic permeability of 1.0433 compared to 1.0087 for barite mud. The absolute magnetic permeability is 13.11×10^{-7} (henry/meter) for iron oxide weighted muds whereas barite mud gave a value of 12.68×10^{-7}. Experiments conducted on dry uncompacted powder of the weighting materials gave very similar results to those obtained from measurements on drilling fluids. Based on these results the iron oxides weighting agents can be classified together with barite drilling fluids as paramagnetic or diamagnetic material. In both groups, the material shows only weak magnetic effects whereas materials in the ferromagnetic group show very strong magnetic effects.[38]

8.5 Filtration Properties Testing Results

Detailed testing of the fluid loss and filtration properties revealed the type and nature of problems associated with each type of weighting material. Results indicate that some remedial action has to be taken to prevent the serious borehole problems that may take place as a result of undesired filtration characteristics.

The low and high temperature filtration tests of the nondispersed fresh drilling fluids incorporating iron oxide and barite weighted mud resulted in the following.

1. All iron oxide mud types exhibited higher fluid loss volumes than barite mud. The highest fluid loss volumes were obtained with ilmenite mud indicating the lack of the proper size particles that can give good impermeable filter cakes. The results shown in Tables 23 to 29 and plotted in Figures 36 to 43 of Appendix B, show that the K-M hematite and the itibirite mud types have similar fluid loss characteristics. Filter cakes obtained varied from moderately thick, obtained by the K-M hematite and barite, to thick, which are those obtained by the itibirite and ilmenite. A high pressure and high temperature testing of the filter cake verified that all filter cakes for the tested mud types were incompressible and permeable. To obtain a filter cake with the least permeability, the appropriate particle size distribution varying from sub-micron size to multimicron size is essential in order to plug all the openings between the grains including the minute gaps. However, to control the fluid loss of a drilling fluid strictly by particle size distribution will require an intolerable concentration of solids that will be detrimental to both the penetration rate and flow

properties. Therefore it is necessary to use filtration control additives to adjust the fluid losses of drilling fluids.

2. Aging of the iron oxide weighted drilling fluids after mixing with shale cuttings seem to have improved the filtration properties significantly. It can be seen from Figure 37 and Figure 40 that the fluid loss volumes have been reduced. However the quality of the mud cake apparently has improved without a significant reduction in thickness.

3. The addition of polymers (Lo-Sol and select-flocculant) as antiwear agents helped in providing a tougher and thinner filter cake by coating the iron mineral grains, tying up the bentonite particles and by the plugging action due to their own chain length.[24] Results shown in Tables 25 and 28 show the improvement in mud properties as a result of treating the mud with the antiwear polymers . The effect of these additives on the thickness and efficiency of the mud cakes was extremely beneficial. The improvement in the efficiency of the mud cakes was confirmed by results of the mud cake compressibility test, shown in Table 28. A correlation of mud properties before and after treatment is represented graphically in Figure 42. The results demonstrate the remarkable improvement in the fluid loss volumes at both ambient and elevated temperatures.

8.6 Analysis of the Abrasion Testing Results and Remedial Treatment

The abrasion tests using rolling techniques have indicated that the amount of wear that takes place in the drill pipe is generally low and of minor importance. However, by using a sensitive analytical balance, measurements for the weight loss percent indicated that more wear of the coupons have taken place with the barite mud than with any of the other iron oxide mud types (see Tables 30, 31, and 33). This is quite understandable in case of the K-M hematite, where the spherical shape of the mineral grains provide a reasonable explanation for this contradictory result. The spherical grain shape of the K-M hematite have been confirmed by electron micrographs shown in Figure 59, Appendix C. The explanation for the higher relative abrasivity of the soft barite over the harder ilmenite and itibirite (hardness 6 to 6.5 on the Moh's scale compared to 3.0 for barite), is probably the shape and nature of the mineral particles. Barite crystals seem to have a greater number of edges than ilmenite and hematite. There is abundant evidence that even very smooth-appearing surfaces are irregular on a molecular scale of distances. The surfaces of cyrstalline material may have quite irregular steps of hundreds in hundred or thousands of Angstrom units in depth.[2] This irregular nature of the crystal faces can increase the abrasivity of the material. Barite crystals probably possess this kind of abrasive crystal faces. Results represented in Figure 44 is an evidence that under low shear rates (i.e., drill pipe) the grain shape and the nature of the crystalline faces have more influence on the wear effect than the hardness of the weighting agent.

On the other hand testing of iron oxide mud systems under high shear conditions show high abrasive characteristics for hard minerals like itibirite and ilmenite. Testing results show clearly the role of the grain shape in the high shear abrasion process. A correlation between the abrasivity of the itibirite mud, which is composed mainly of natural hematite and quartz grains, and the spherical grained, K-M hematite give a quantitative evaluation of the role of grain shape in abrasion. Both the K-M hematite and the itibirite possess nearly the same chemical composition and the same hardness. Therefore the difference in their abrasive characteristics can be attributed to the difference in grain shape. This accounted for 60 percent of the relative abrasivity of the itibirite mud system.

In this study it was experimentally verified that the use of Lo-Sol and Select-Flocculant polymers have reduced the abrasivity by an average of 55 percent (see Figures 45 to 48). Details on the rheological properties of the treated mud were briefed in the previous chapter. However, it can be stated that the polymer and dispersant additives to iron oxide weighted drilling fluids system has rendered the system to an efficient nonabrasive mud system that incorporates various mud types to choose from, with regards to dominating field conditions. Furthermore, treatment of barite mud with the polymer additives resulted in a new type mud which has the following characteristics:

1. The improved barite mud has less abrasivity and excellent filteration properties.

2. The new system has a better lifting capacity, hence the polymer additive will also act as a bentonite extender.

3. The polymer acted as a protective coating agent that normally decelerates the comminution of the barite particles and drilling cuttings. As a result less barite particles have to be eliminated from the active mud system and replaced. This makes the utilization of the barite as a weighting material more feasible. The coating agent also helps in decreasing the amount of contaminants in the mud caused by cuttings.

8.7 Interpretation of the Attrition Tests

The results, listed in Tables 43 to 46 show the particle size distribution of the investigated mud types. Graphical representation of these results shown in Figures 49 to 52 indicate the degree of particle attrition suffered by each mud type. It is apparent from the results that there is a significant reduction in the particle size of barite and a considerable increase in the colloidal fraction. This type of fines is responsible for the dramatic increase in viscosity and yield value of the barite mud through utilization. The attrition rate of the iron oxides in the mud has been proved to be much less than barite and the colloidal fines generated during the use of the mud are much lower.

Improved barite drilling fluids containing 1.5 to 2.0 percent Lo-Sol or Select-Flocculant polymers and a thinner displayed a better particle size distribution than untreated mud. The particle size distribution graph for treated mud shown in Figure 52, indicates that there is a tendency of the polymer to adhere to coarse grains more than fine grains, a phenomenon that has been also observed in the scanning electron micrographs. The generated fines are believed to contain a considerable amount of polymer and dispersant fines. These types of fine material can be eliminated from the drilling fluids action system without causing a significant reduction of the mud density.

Treatment with polymer and thinner is also beneficial in case of mixed weighting agent. Drilling fluids made of a mixture of barite and K-M hematite and treated with the anti-wear agent was found to have the following advantages over barite and improved barite mud systems:

1. It is less abrasive than the barite mud.

2. It has better rheological and flow properties as seen from their viscosity profiles.

3. The barite grain attrition in the mud is minimized

due to the effect of grain coating of both barite and K-M hematite grains and the distinguished K-M hematite particle shape.

4. The chemical treatment needed for the improved barite-KM hematite mud is less frequent and more effective than in the case of barite muds.

8.8 Analysis of the Compatibility Performance Results

The comparison of the initial and final mud properties after each mud aging period during the compatibility test indicates the drilling fluids performance that can be expected in the field. The viscous properties and the ionic activity of the mud determined by the Methylene Blue Test, play an important role in controlling the drilled solids content in the circulating system. The ionic activity when properly adjusted and maintained, will retard the hydration of the reactive solids and enable the surface removal techniques to be more effective. The degree to which the cuttings are affected by the various drilling fluids can be determined from the results of the cationic exchange capacity before and after the test and by estimating the percent of shale lost to the mud during the test.

Data collected on various mud types during the compatibility performance tests are summarized in Table 47. A plot for the plastic viscosity versus yield value can be seen in Figures 53, 54 and 55. The PV versus YP relationship, as explained in sections 3.8 and 6.7, shows the stability of each type of mud under simulated conditions of drilling through the Eastern Oklahoma, Atoka shale formations. The broken lines shown on the graphs represent the general trend for the change of the yield value with the plastic viscosity due to thermal aging. The slope of the line decreases with the increase in thermal stability.[18] The effectiveness of the chemical treatment is indicated by its tendency to decrease the slope of the stability line. On this basis the analysis of the results indicate the following.

1. The majority of the iron oxide mud types have shown better thermal stabilization than barite, as indicated by the slopes of the stability lines.

2. The itibirite mud show similar stabilization performance to barite.

3. The decrease in the slope of the barite mud stability lines due to the effect of chemical treatment causes lower increase in yield point per unit increase in plastic viscosity and, therefore high apparent viscosities cannot be controlled easily because thinners become less effective.

Drilling fluids properties are among the important parameters which influence the drilling penetration rate. Although most mud properties are interrelated, it was possible to relate the individual drilling fluid property effects to the penetration rate using curve fits of the results of laboratory tests.[18] The relationship between the penetration rate and plastic viscosity was represented by the formula:

$$(ROP)_2 = (ROP_2 \times 10^{0.003(\overline{PV}_1 - \overline{PV}_2)}$$

where

$(ROP)_1$ = initial rate of penetration (ft/hour)

$(ROP)_2$ = final rate of penetration (ft/hour)

$\overline{PV}_1$ = initial plastic viscosity (centipoises)

$\overline{PV}_2$ = final plastic viscosity (centipoises)

The formula can be written in the form:

$$\frac{(ROP)_2}{(ROP)_1} = 10^{0.003(\overline{PV}_1 - \overline{PV}_2)}$$

This formula was used in this study to determine changes in penetration rates of the various mud systems as a function of the plastic viscosity changes as a result of the thermal aging of the drilling fluids. Penetration rates ratios (R) were calculated from the successive plastic viscosity values shown in Table 47. The above formula was developed to the form:

$$R = \frac{(ROP)_n}{(ROP)_o} = 10^{0.003(\overline{PV}_o - \overline{PV}_n)}$$

$(ROP)_o$ and $\overline{PV}_o$ are the initial rate of penetration and initial plastic viscosity before aging.

$(ROP)_n$ and $\overline{PV}_n$ are the final rate of penetration and final plastic viscosity, where n denotes the aging time varying from 1 to 7 days. In the graphical presentation of results shown in Figures 55a and 55b each mud type is represented by a separate graph and all points on the same graph are only related to the initial point 0. Values for penetration rate ratio on different graphs are unrelated. It can be seen that the best performance was displayed by the K-M hematite, the itibirite and the ilmenite-itibirite muds which also have lower solids than all other mud types. The higher solids content in the barite, ilmenite and barite-K-M hematite muds is due to the relatively lower overall density of the weighting agents and the higher shale percentage lost to these muds during the compatibility tests. The higher shale weight loss percent indicates undesirable reaction between the barite and ilmenite muds with cuttings of the Atoka shale leading to quicker deterioration of the cuttings than with other mud types.

Results of the cationic exchange capacity combined with shale weight loss data indicate that the Atoka shale can be classified as a low methylene blue capacity (MTB). It contains less than 15 ppb of bentonite equivalent clays in 100 lbs of shale and therefore, the Atoka shale are of the low reactive type. The drilling problems associated with this type of shale is due to the greater percentages of inert or low reactivity minerals. The improved mud types (with additives of polymers and dispersants) will be the most compatible muds for drilling through this formation. The coating agent in these drilling fluids will prevent the breakup of the shale and the subsequent dispersion into the mud system.

CHAPTER IX

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

9.1 Conclusions

Evaluation of the iron minerals weighted drilling fluids have been carried out to study the advantages and problems associated with the utilization of the iron minerals in increasing the density of the drilling fluids. The various aspects of the mud properties have been examined and correlated with the performance of the widely accepted barite weighted drilling fluid system under the same conditions. The results of the drilling fluids evaluation testing program were used for the diagnosis of problems associated with the utilization of iron minerals in drilling fluids to initiate remedial solutions for the encountered defects.

Based on the results achieved in this study the following conclusions are made.

1. The investigated iron oxides (ilmenite, synthetic K-M hematite and itibirite) form a prospective group of high density weighting material for drilling fluids. These minerals offer a variety of performances and characteristics that can be used as basis for selection and planning of the most competent

drilling mud programs. This is contrary to the barite mud system which uses only one mineral.

2. The nondispersed iron minerals weighted drilling fluids have better rheological and gel strength characteristics than barite. Most of the iron minerals mud types possess lower effective viscosity than barite in all parts of the drilling circulating system. Superior rheological and flow characteristics were obtained by the K-M hematite weighted mud type. Viscosity profiles show that no mud treatment is needed in any part of the circulating system for the K-M hematite mud.

3. The iron oxides weighted mud types are more reliable for drilling under high temperature conditions than the barite weighted mud system. High temperature and high pressure viscosity results indicate that all iron minerals weighted mud types exhibited the essential temperature stability at various shear rates whereas the barite mud system shows rapid viscosity changes with increasing temperature. High temperature gel strength results indicate that it is safer to drill with iron minerals weighted mud types and that barite weighted drilling fluids can endanger the drilling operation unless an extensive chemical treatment is provided at the surface during the circulation.

4. Based on drilling fluids properties alone, an estimation of the penetration rate show that most iron oxide weighted mud types (K-M hematite and itibirite) exhibited

higher rates of penetration than the barite weighted mud system. These results were obtained from a drilling simulation through the Eastern Oklahoma Atoka Shale using the compatibility performance tests. The results enhanced the importance of the role of the solids content on the rate of penetration.

5. Iron oxide mud types are less reactive with shale cuttings, therefore they develop less solids in the mud and minimize borehole problems.

6. The particle size distribution of the iron oxides can not provide good filtration properties and efficient mud cakes. Therefore all iron mineral weighted mud types, like barite, require filtration control material to be added to the mud. However, the filtration properties of the iron mineral weighted drilling fluids improved significantly when the mud was thermally aged with the Atoka shale cuttings. The filtration and fluid loss characteristics were perfectly controlled for all tested mud types, including those weighted with mixed weighting agents, by the mud reformulation using an antiwear polymer and dispersant additives.

7. The particle comminution of the hard iron mineral grains in the drilling fluids, due to aging and high shear rates, is much slower than barite. The disintegration of the soft barite particles produced a significant amount of undesirable fines smaller than 1 micron during the attrition test. This type of fines has an invert impact on the mud properties and penetration rates.

8. The investigated iron minerals weighted mud types do not possess ferromagnetic properties that may interfere with the electric or magnetic well logging equipment. All tested iron minerals mud types exhibited a paramagnetic or diamagnetic behavior which does not produce strong enough electric or magnetic effects to interfere with logging equipment. However, the relatively high magnetic permeability (compared to barite and other mud components) can be deployed to recover the iron minerals from the drilling fluids. The salvage of the iron minerals can be achieved by applying strong magnetic fields in the form of magnetic separators. This mineral separating technique suggests a convenient method of decreasing the density of any given mud upon requirement without loosing the weighting material.

9. The iron oxides are reported to react and combine with hydrogen sulphide and therefore it may provide a temporary buffer action when a sour gas-containing formation is reached during drilling. The iron weighted mud may also act as a hydrogen sulphide corrosion inhibitor.

10. Based on the results of the mud abrasivity, the investigated drilling fluids can be classified into the following categories.

a. Abrasive drilling fluids: They include the untreated ilmenite and itibirite weighted muds (type A and C). The abrasive mud types can cause excessive wear to the bit and surface equipment unless certain percautions are taken. The ilmenite and itibirite mud types exhibited undesirable filtration characteristics that may cause extensive formation damage unless properly treated. The utilization of these mud types requires the use of the appropriate protective coating and effective protection of the downhole equipment. Intensive maintenance of surface equipment is needed to maintain the wear effect at a convenient level.

b. Moderately abrasive drilling fluids: These include the untreated K-M hematite weighted mud (type B) and the improved ilmenite and itibirite muds (type F and H muds). The improved mud types are those containing a proportion ranging from 1.5 to 2.0 percent by volume polymer (either Lo-Sol or Select-Flocculant) and one or more dispersants to control the plastic viscosity. The proposed modified grain shaped ilmenite and itibirite weighted muds may be also included in this category. A good maintenance system will be needed during the utilization of these mud types to protect the bit and surface equipment.

c. Low abrasive drilling fluids: Mud types falling in this category are the untreated barite mud, the improved K-M hematite mud type G and the improved, modified grain shaped ilmenite and itibirite weighted mud types.

d. Nonabrasive drilling fluids: This category include the improved barite mud (type I) and the improved barite-K-M hematite mud (type GI). Both mud types possess an abrasion level below that of the barite mud.

The utilization of drilling fluids of the b, c or d categories does not require in fact any further percautions than those normally taken for the control of corrosion. The distribution of the various mud types on the different categories indicates that the polymer coating and encapsulation effects together with the proposed mechanical grain modification can render the most abrasive muds to a low abrasive or nonabrasive material. The coating technique also helps to decelerate the grain attrition of the soft barite grains used in the formulation of the nonabrasive drilling fluids.

9.2 Recommendations for Future Research

Barite has been the overwhelming weighting agent for drilling fluids for more than fourty years. There is, therefore, a vast unknown area regarding the utilization of the iron minerals to become the main ingredient of the weighted drilling fluids system. Relative research fields to this area incorporate the chemistry of the drilling fluids, mineral processing and petroleum reservoir engineering.

a. Chemistry of Drilling Fluids

In the field of the chemistry of drilling fluids a study of the reaction between the iron oxide weighted drilling fluids and the acid-forming gases is eminent. Hydrogen sulphide is one of the most serious environmental corrosion accelerators. It is often associated with hydrocarbons of the produced crude oil or gas as well as in formation water.

It is extremely important to investigate the possiblity of using the iron oxide weighted drilling fluids as a distinct indicator of the occurrence of hydrogen sulphide and the capacity of the iron oxide mud as a sour-gas scavenger.

b. Mineral Processing

Two different research projects are proposed in the field of mineral processing. The first research deals with the grain size and grain shape of the mineral grains. Abrasion testing results indicated that under high shear conditions, the grain shape is responsible for at least 50 percent of the relative abrasivity of the ilmenite and itibirite mud types. If the shape of the mineral grain is changed, the surface area is affected and eventually this will affect the amount of adsorbed water. The spherical grain shape provides the least possible mud abrasivity and the best rheological performance. The particle shape and particle size distribution of the weighted drilling fluids control the most vital properties of the mud. Particle shape and size are controlled by the mineral crushing technique which may take place either by compression of the ore between rigid surfaces or by impacting the particle against a massive surface.[54,64] A crushing design based on particle impact against a massive surface may be found effective in controlling the particle size and particle shape. The proposed research should provide a new crushing technique capable of producing the desired size, shape and an optimum particle size distribution which can provide the best possible mud cake.

The weighting material in the drilling fluids accounts for the major part of the drilling fluids cost. If the weighting material can be maintained and recovered after the completion of the well a great saving in the consumption of the weighting agent can be made. The relatively high magnetic permeability possessed by the iron minerals offers the opportunity of recovering the expensive weighting material by using a strong magnetic field. There is also a possibility of using the same technique for reducing the mud weight by the removal of a specific amount of the iron minerals from the mud. Research is needed to verify this potential advantage of the iron minerals and to investigate the feasibility of the project.

C. Field of Reservoir Engineering

The mud solids that form on the well bore wall restrict the filtrate flow into the formation, but allow some fines to move with the filtrate into pore channels. These fines block the flow channels and reduce the productivity. Remedial work is necessary to restore the formation productivity. A research is proposed to investigate the limits of formation damage observed with the various types of the iron oxides weighted drilling fluids. The improved mud types containing the polymer antiwear agent may be also considered. A comparison between the initial and return permeabilities for all mud types including a standard control mud will give an evaluation of the iron oxide weighted system. The testing program will yield a drilling mud type that would cause the minimum formation damage. Tests can be performed either by using regular mud acid for remedial treatment or other types of acids.

NOMENCLATURE

A = area of the filter cake
C = capacity
C_D = drag coefficient
D = borehole diameter
D_g = depth of geological column
d_p = drill pipe outside diameter
d_s = particle diameter
f = frequency
K = consistency index
k = permeability
L_d = depth
L = electric inductance
N = number of turns
n = degree of departrue from Newtonian behavior
p = pressure
p_o = overburden pressure
PV = plastic viscosity, q_o = the zero error
Q = maximum charge density
Q_c = volume of the filter cake
Q_w = volume of the filtrate
R_s = radius of toroid
R = rate of penetration ratio
R_a = apparent resistivity
R_e = Reynolds number
R_m = mud resistivity
Rop = rate of penetration
R_t = true resistivity
R_{xo} = mud filterate resistivity
r = radius of cross section
t = time in seconds
V = volume of flow
V_a = annular velocity
V_i = volume of mud before weighting
V_s = slip velocity
V_w = volume of weighting material added
yp = yield point
yv = yield value
ν = shear rate
ν_{if} = specific weight of the interstitial fluids
ν_{rm} = specific weight of the rock matrix
θ_{600} = viscometer reading at 600 rpm
θ_{300} = viscometer reading at 300 rpm
μ = magnetic permeability
μ_e = effective viscosity, μ_f = fluid viscosity
μ_o = magnetic permeability of vacuum
μ_p = plastic viscosity
μ_r = relative magnetic permeability
t_o = yield stress or shear stress to initiate flow
t_w = shear stress at wall
ϕ = porosity (fraction)

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APPENDIX A

CHARTS FOR

MUD PREPARATION AND ASSESSMENT

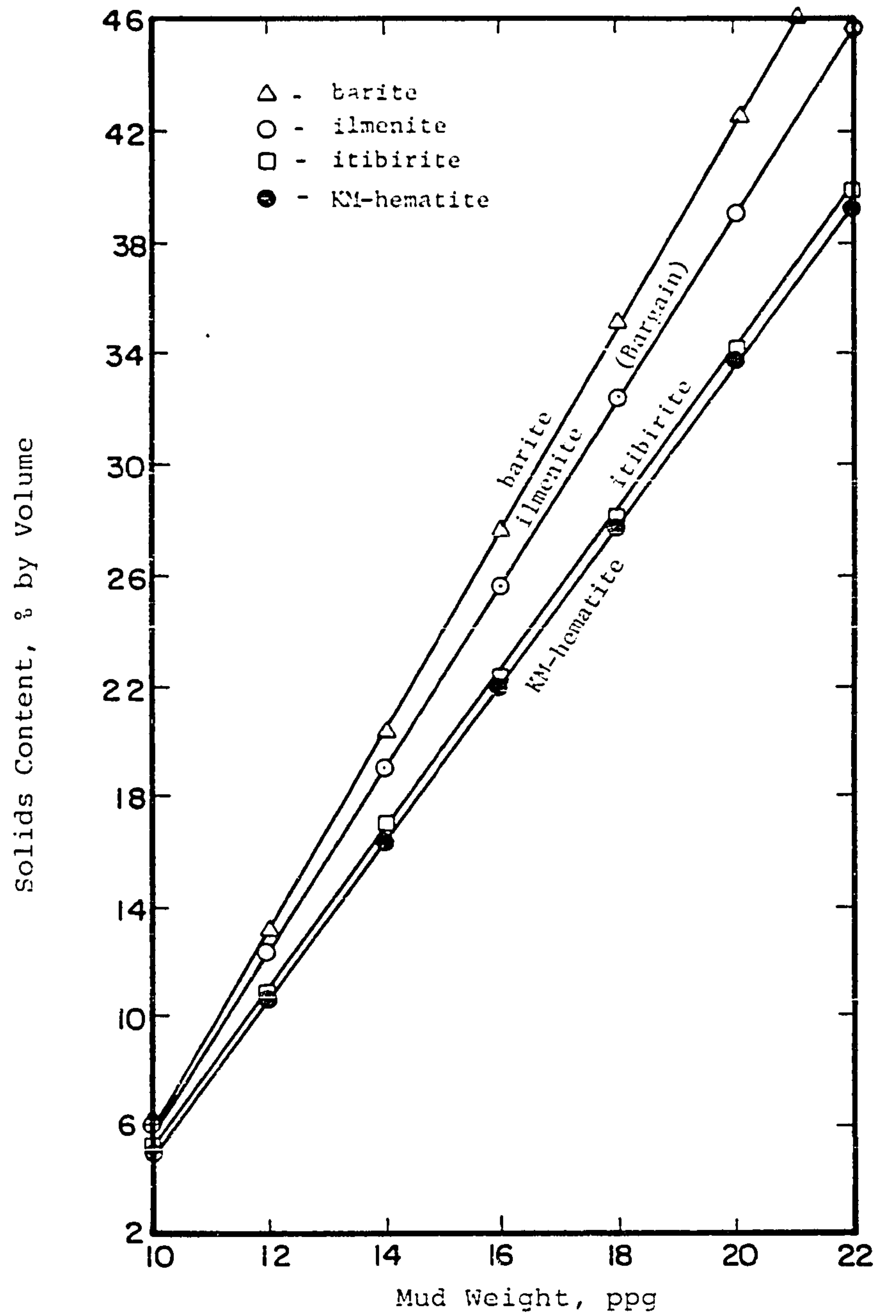


Fig. 13. Mud weight vs. solids content.

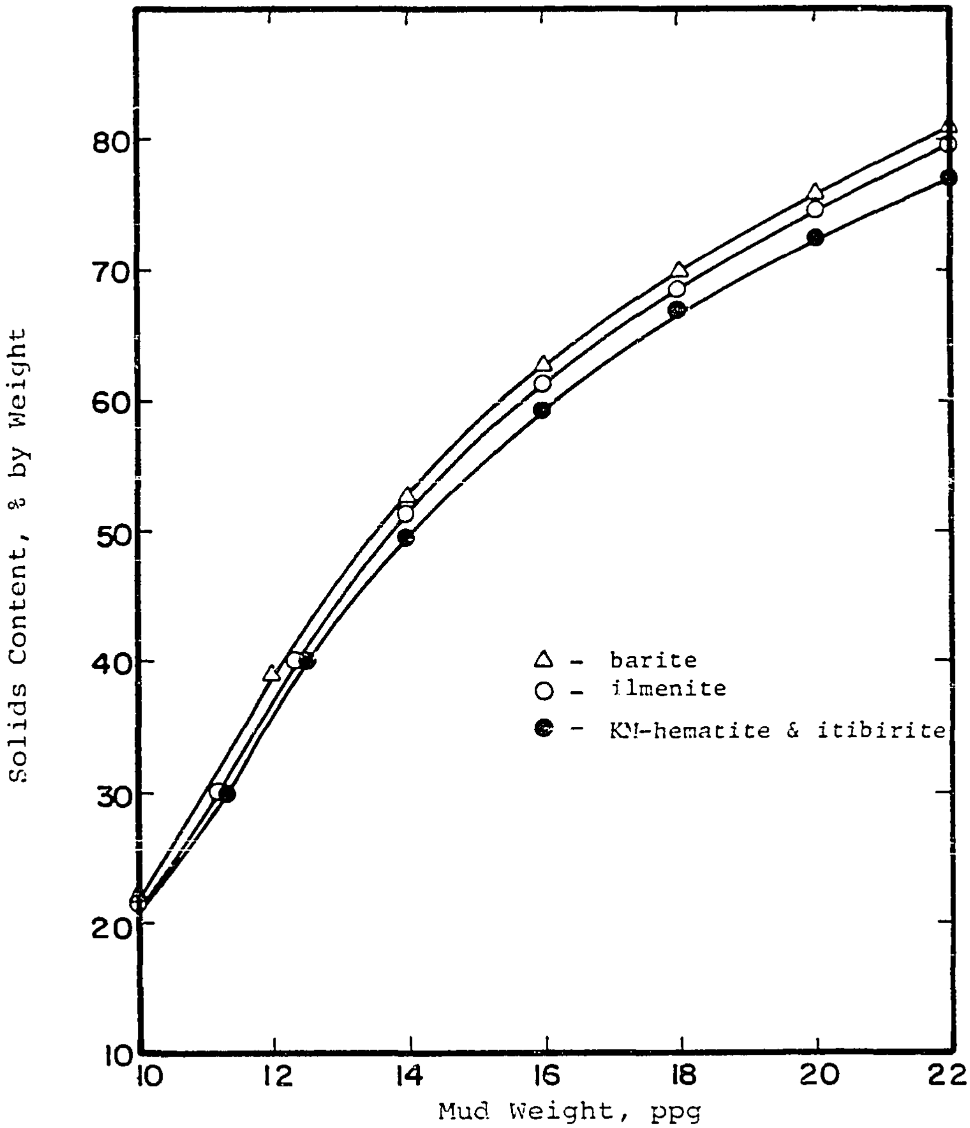


Fig. 14. Mud weight vs. solids content by weight.

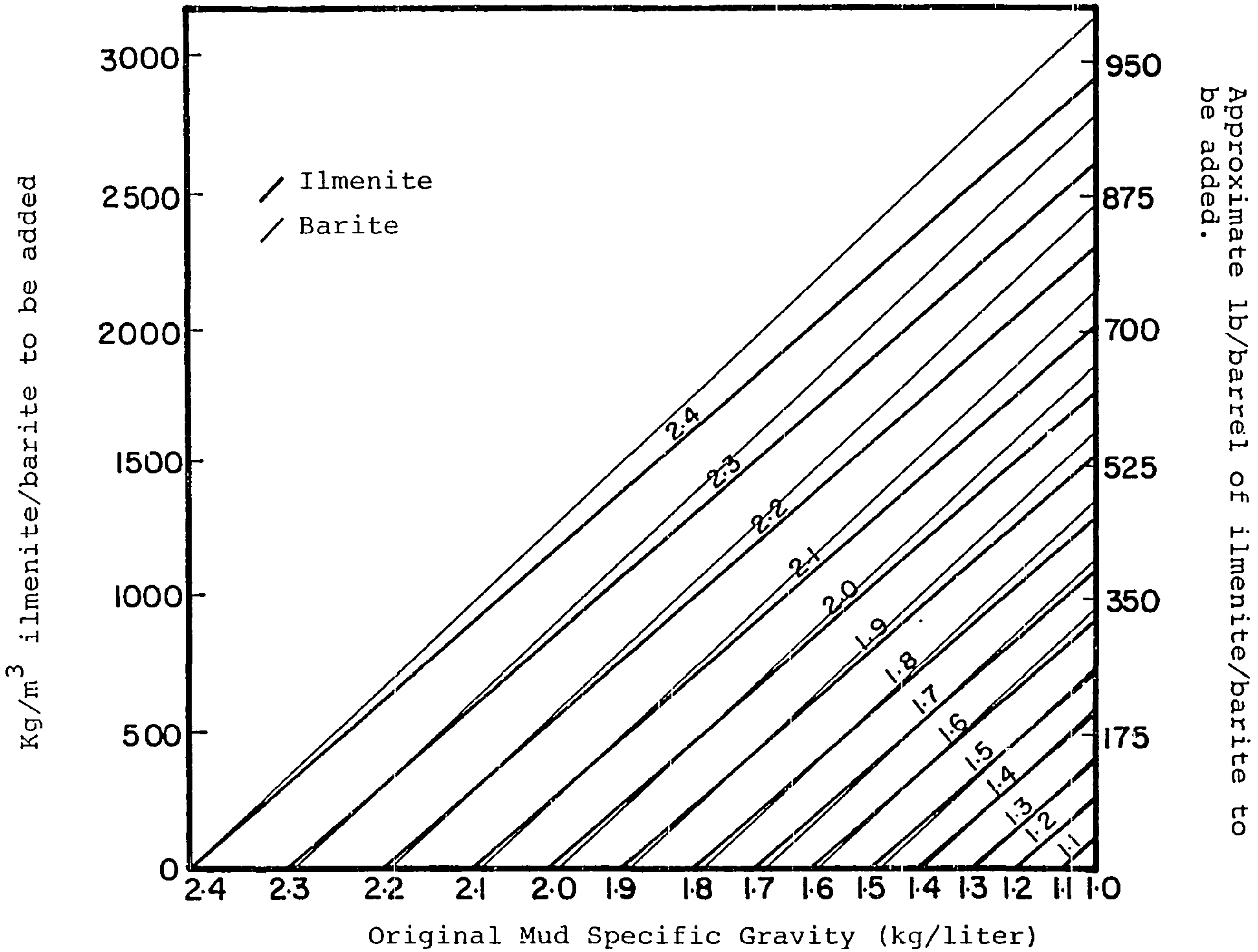


Fig. 15a. A laboratory chart for assessing ilmenite addition to mud compared to barite.

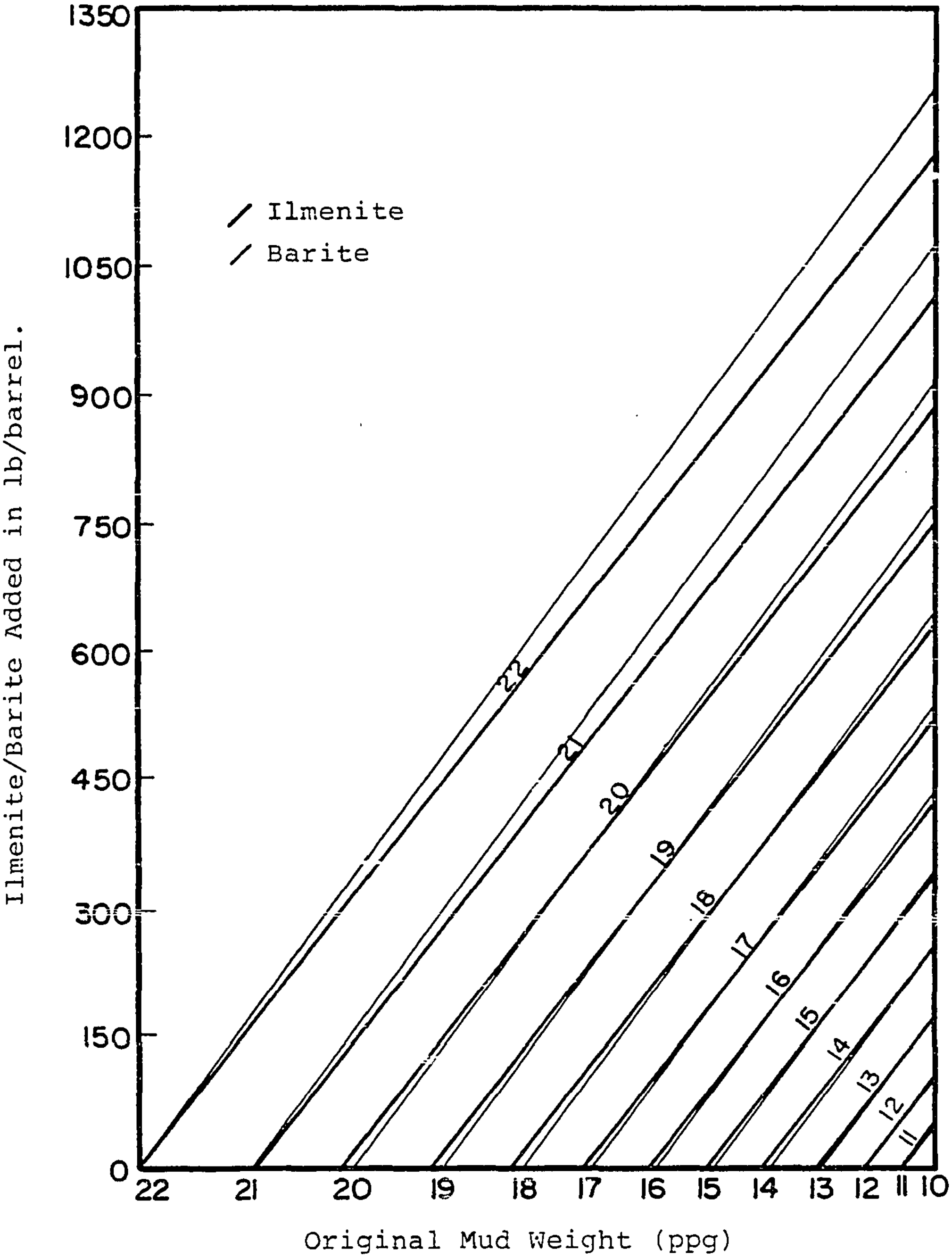


Fig. 15b. Chart for assessing ilmenite addition to mud compared to barite in field units.

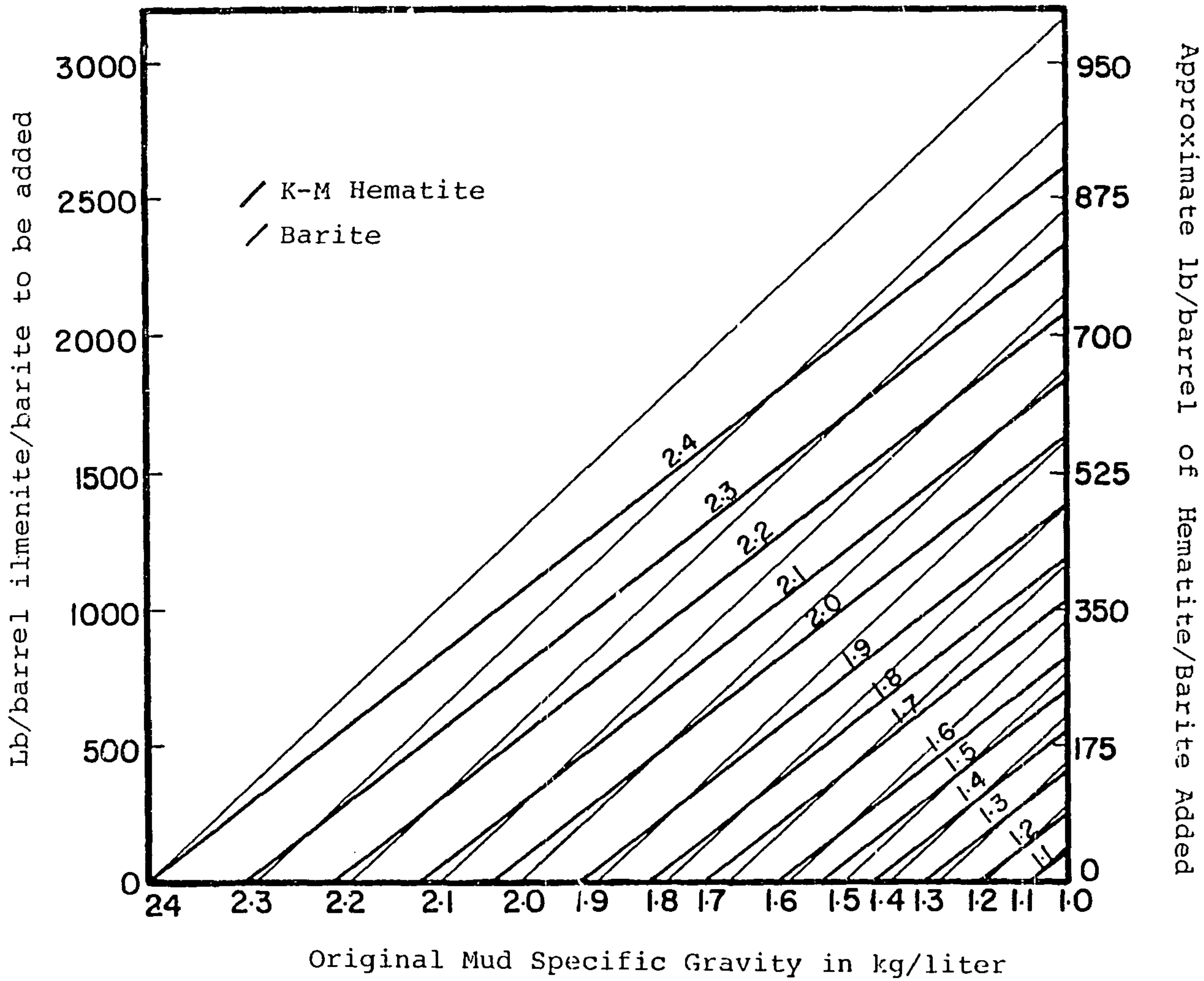


Fig. 16a. Laboratory chart for assessing hematite addition to mud compared to barite.

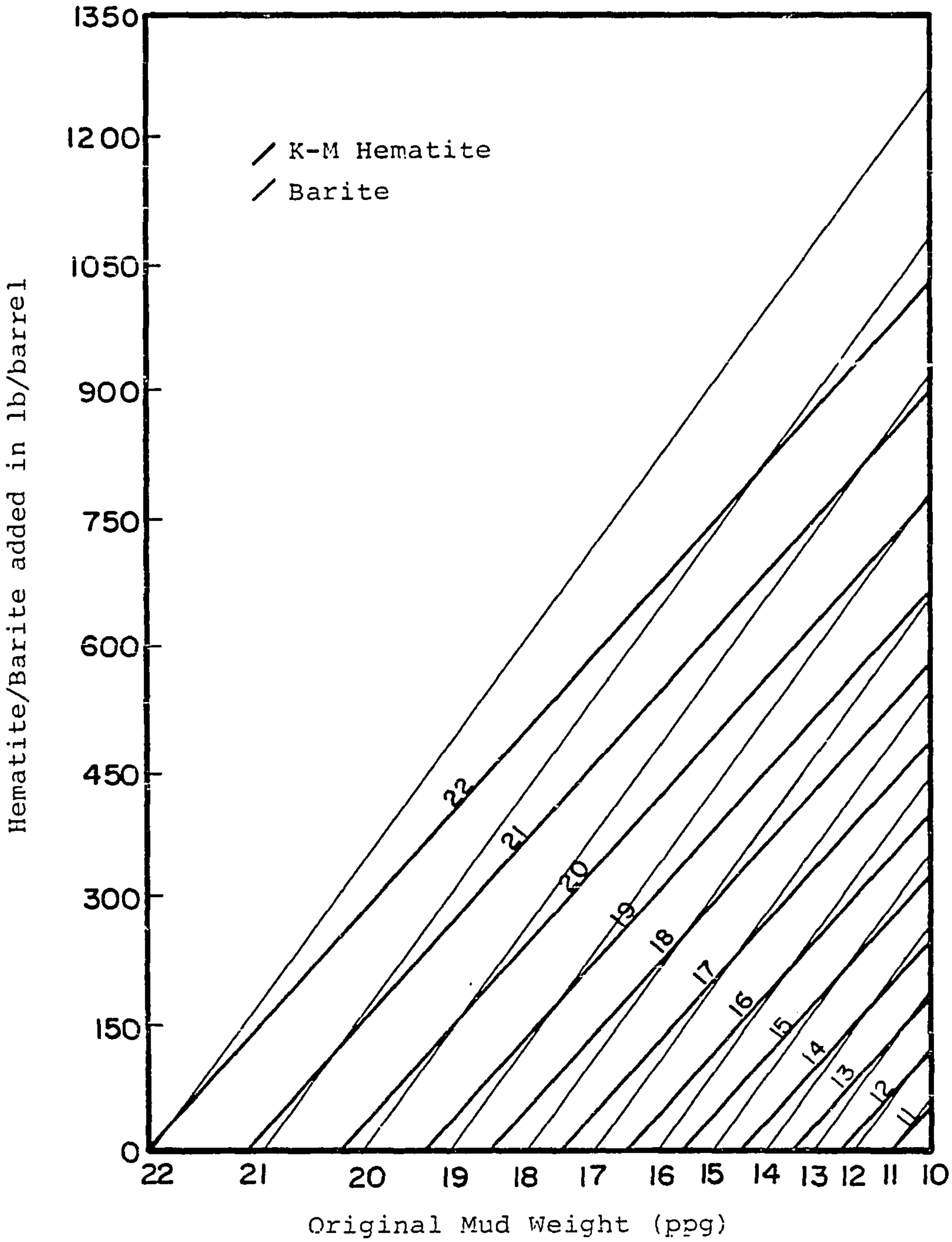


Fig. 16b. Chart for assessing hematite addition to mud compared to barite in field units.

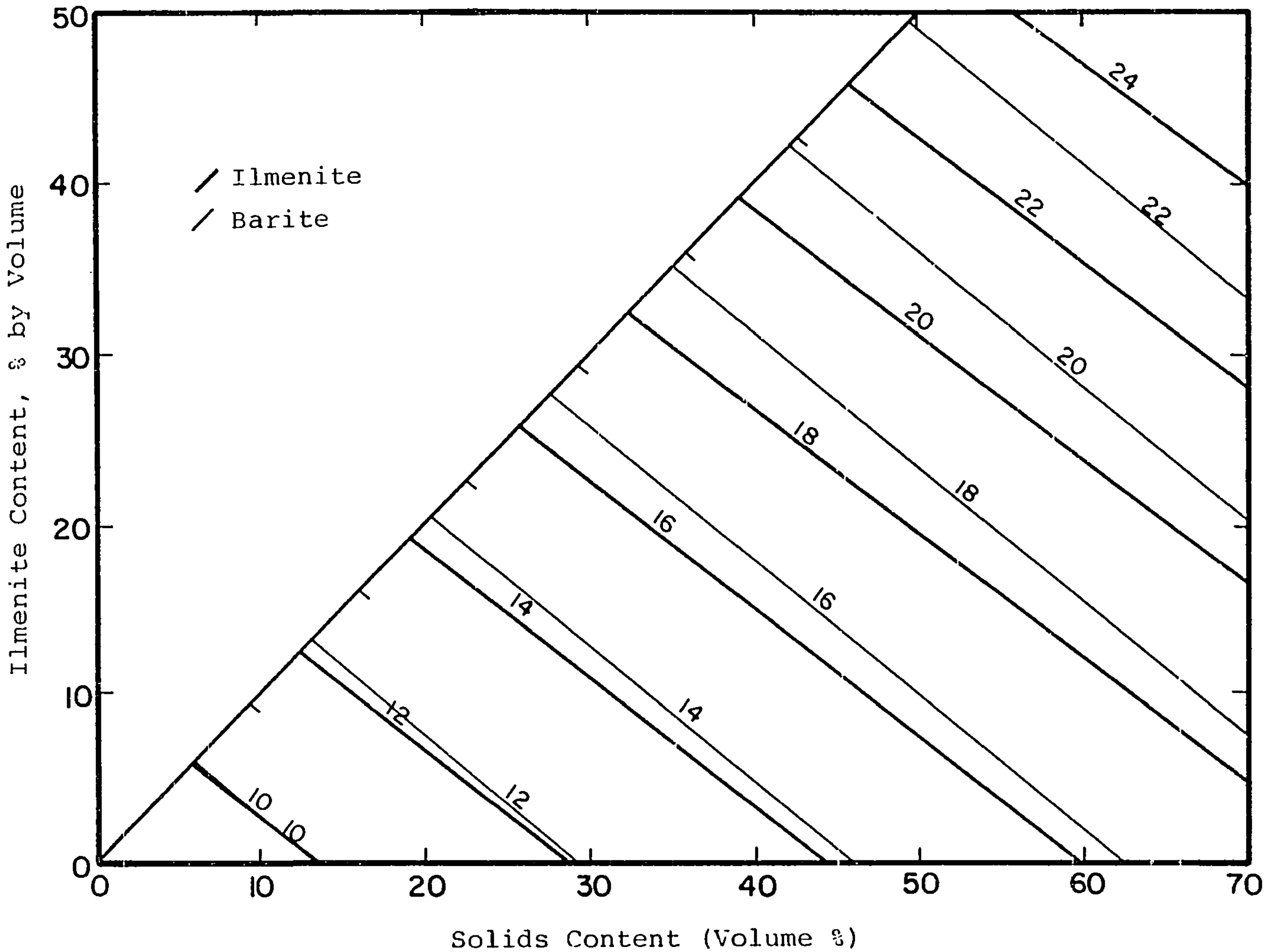


Fig. 17. Ilmenite/barite content vs. solids content.

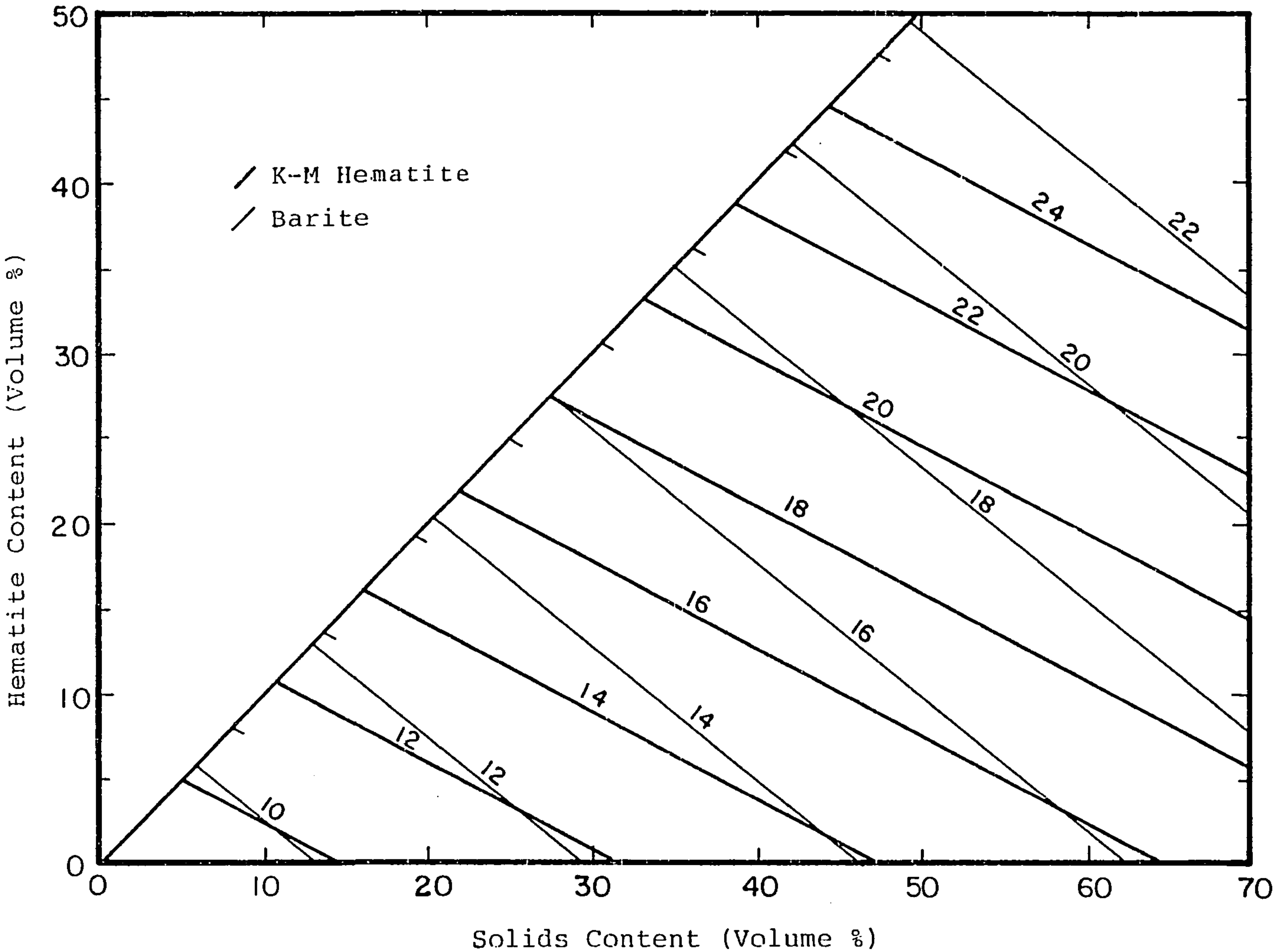


Fig. 18. Hematite/barite content vs. solids content.

APPENDIX B

TABLES AND ILLUSTRATIONS

TABLE 6

TESTING OF RHEOLOGICAL PROPERTIES

Type A Mud

Mud Composition

3% bentonite + ilmenite (Bargain) + distilled water

Mud weight = 14 p

pH value = 9.4

Temperature = 80 °F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	72.0	0.5	36.00
300	48.0	1.0	48.00
200	39.5	1.5	59.25
100	30.0	3	90.00
6	22.5	50	1125.00
3	28.0/92		

Rheological Properties

Plastic Viscosity (PV)	= 24.0	cp
Yield Point (YP)	= 24.0	lb/100 sq. ft.
Initial Gel (10 sec.) (IG)	= 28.0	lb/100 sq. ft.
10 minutes Gel (10 mg)	= 92.0	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

		Minimum	Maximum
Bit Nozzles	=	9	18
Drill Collars	=	27	37
Drill Pipe	=	48	140
Solid Removal Equipment	=	20	60
Annulus	=	45	>1000

NOTE: The mud probably requires adjustment at the annulus.

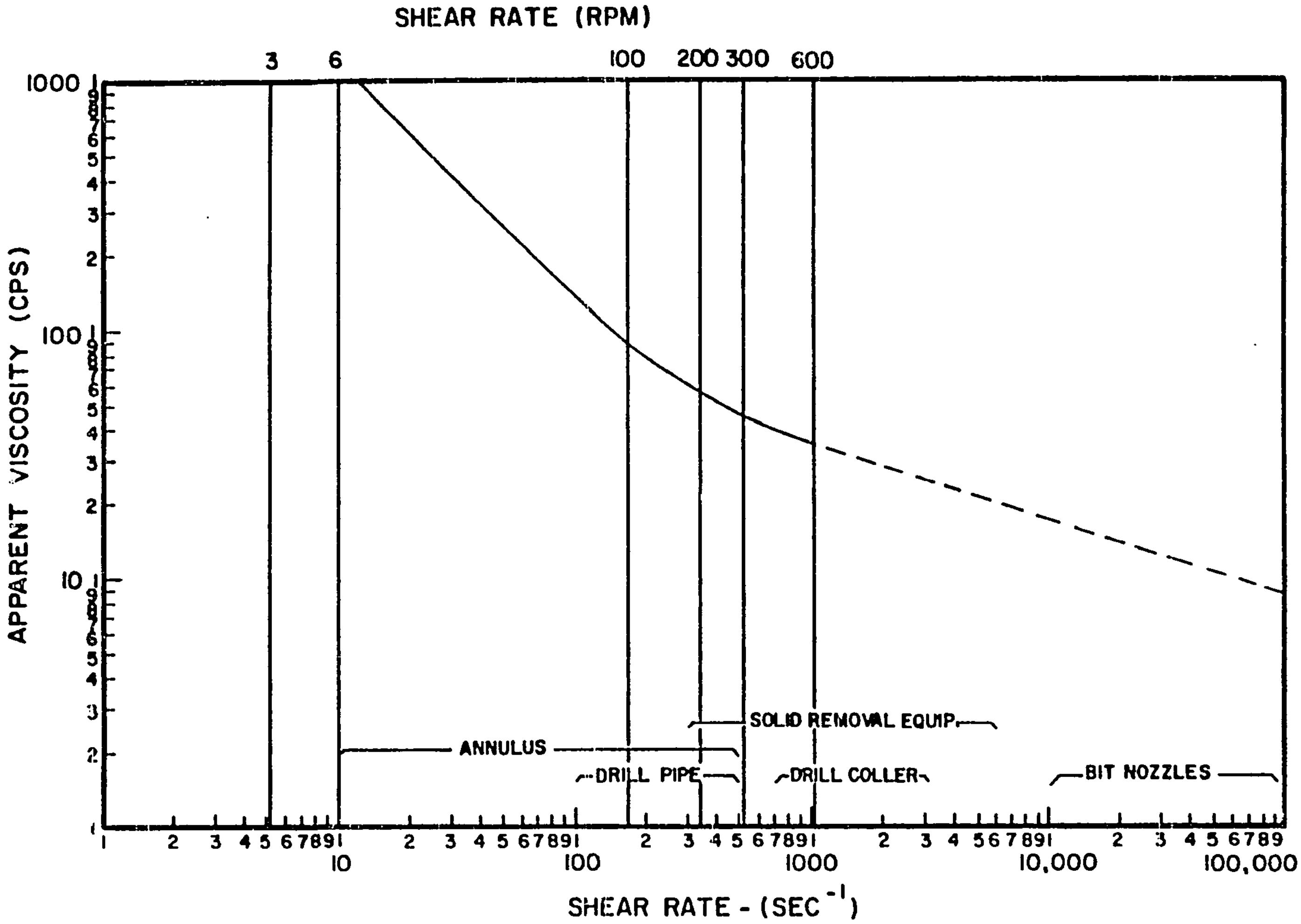


Fig. 19. The log log viscosity profile for type A ilmenite mud.

TABLE 7

TESTING OF RHEOLOGICAL PROPERTIES

Type B Mud

Mud Composition

3% bentonite + K-M hematite + distilled water

Mud weight = 14 ppg

pH value = 9.69

Temperature = 80 °F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	63.5	0.5	31.75
300	37.0	1.0	37.0
200	28.0	1.5	42.0
100	18.0	3.0	54.0
6	5.0	50	250.0
3	4.0	100	400.0

Rheological Properties

Plastic Viscosity (PV)	=	25.5	cp
Yield Point (YP)	=	11.5	lb/100 sq. ft.
Initial Gel (IG)	=	4.0	lb/100 sq. ft.
10 Minutes Gel (10 mg)	=	20.0	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	10	18
Drill Collars	22	32
Drill Pipe	35	70
Solid Removal Equipment	20	42
Annulus	35	230

NOTE: Mud properties are acceptable at all parts of the circulating system. No treatment is necessary.

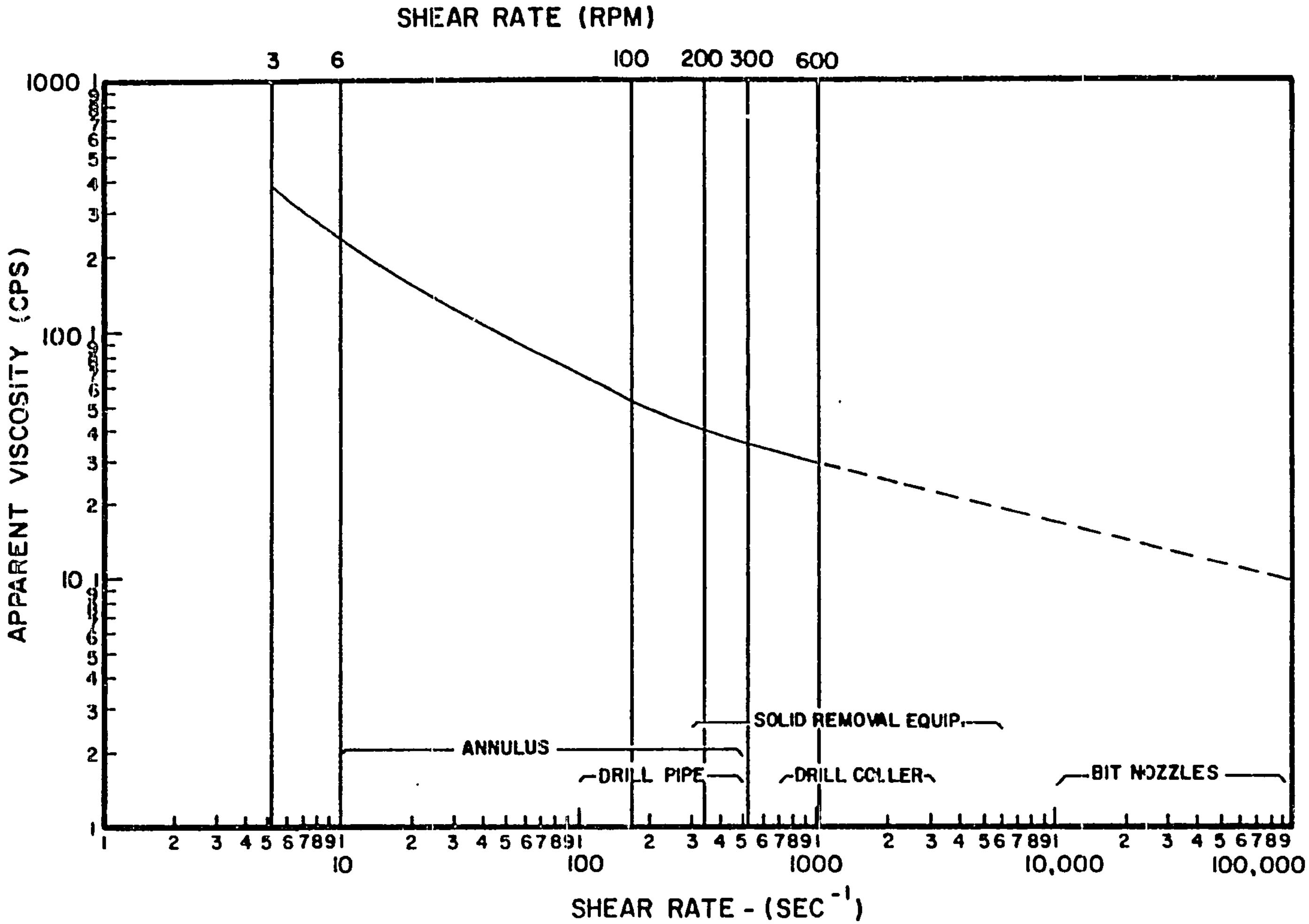


Fig. 20 . The log log viscosity profile for type B mud (K-M hematite).

TABLE 8

TESTING OF RHEOLOGICAL PROPERTIES

Type C. Mud

Mud Composition

3% bentonite + Itibirite (Densimix) + distilled water

Mud Weight = 14 ppg

pH Value = 8.2

Temperature - 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	84.5	0.5	42.25
300	60.0	1.0	60.00
200	51.5	1.5	77.25
100	38.5	3.0	115.50
6	21.0	50	1050
3 (10 sec)	-		

Rheological Properties

Plastic Viscosity (PV) = 24.5 cp
Yield Point (YP) = 35.5 lb/100 sq. ft.
Initial Gel (IG) = 28.0 lb/100 sq. ft.
10 minutes Gel (10 mg) = 44.0 lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	4.5	16
Drill Collars	28	55
Drill Pipe	65	150
Solid Removal Equipment	75	85
Annulus	65	>1000

NOTE: The mud may probably require treatment at the annulus.

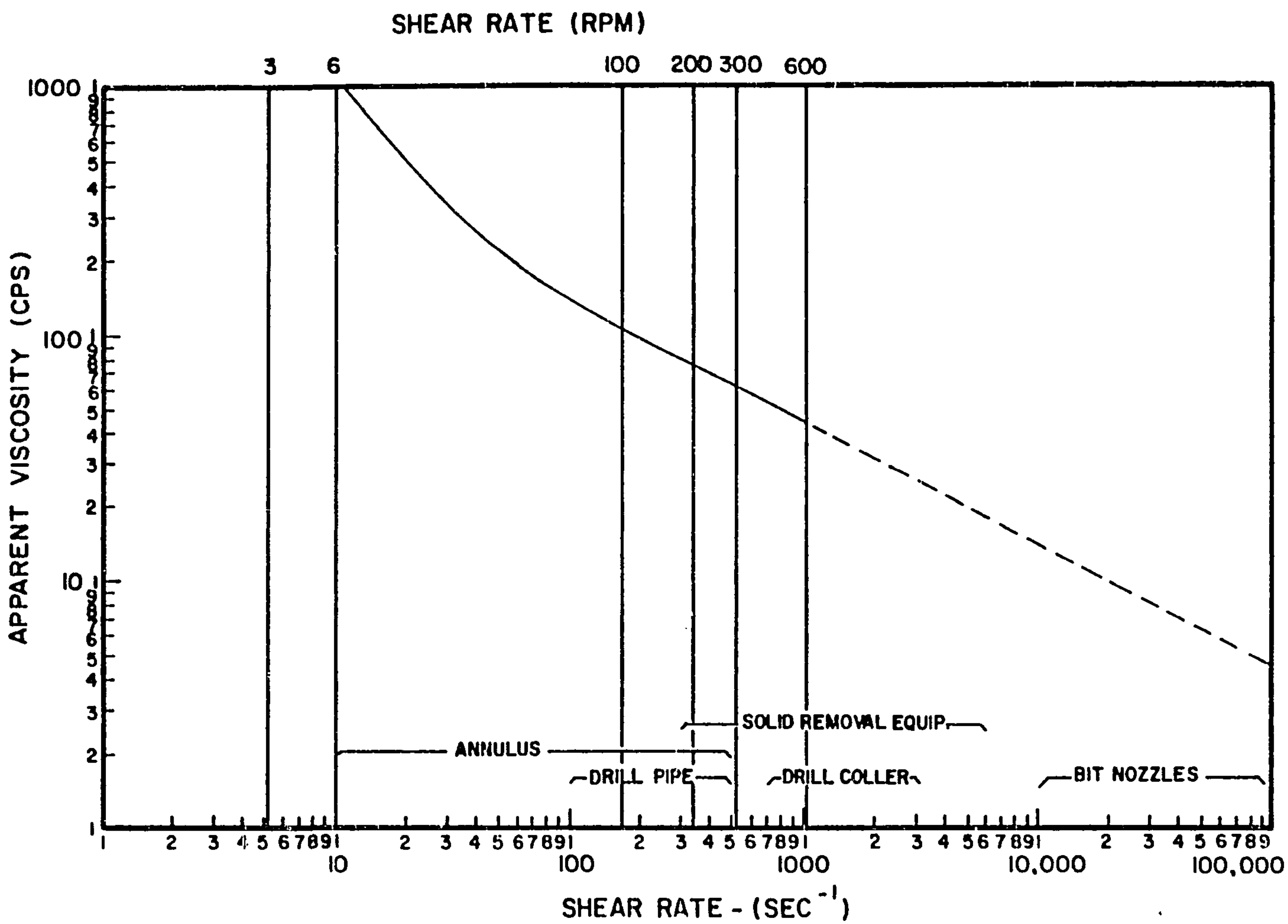


Fig. 21. The log log viscosity profile for type C mud (itibirite mud).

TABLE 9

TESTING OF RHEOLOGICAL PROPERTIES

Type D Mud

Mud Composition

3% bentonite + barite + caustic soda (for pH adjustment)

Mud Weight = 14 ppg

pH Value = 9.0 (adjusted from 7.7)

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	96	0.5	48.0
300	67	1.0	67.0
200	56	1.5	84.0
100	40	3.0	120.0
6	19.5	50	975.0
3 (10 sec)	29		

Rheological Properties

Plastic Viscosity = 29.0 cp

Yield Point = 38.0 lb/100 sq. ft.

Initial Gel = 29.0 lb/100 sq. ft.

10 Minutes Gel = 47.0 lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	5	18
Drill Collars	28	55
Drill Pipe	68	150
Solids Removal Equipment	20	90
Annulus	65	975

NOTE: The mud may require adjustment at the annulus.

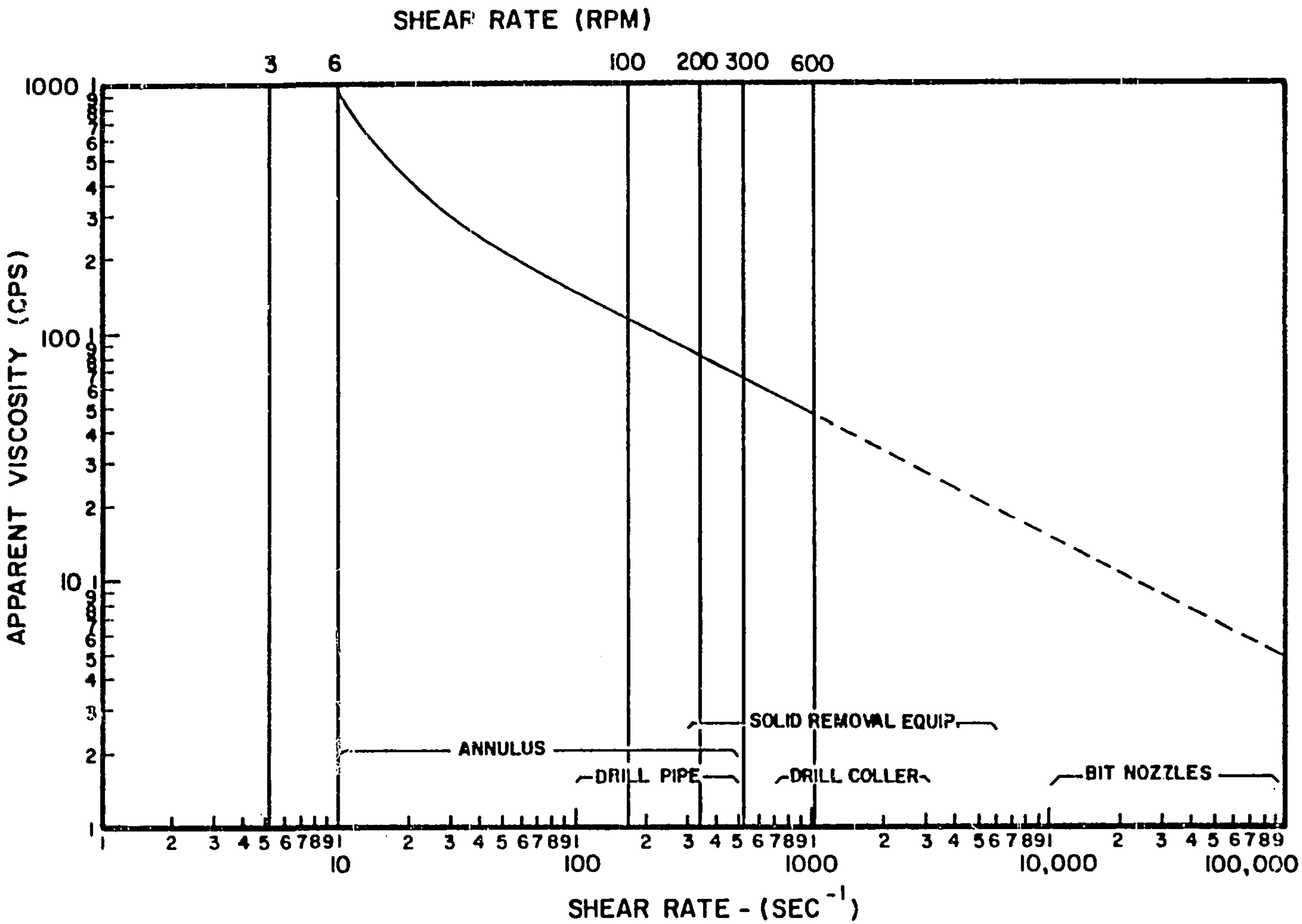


Fig. 22. The log log viscosity profile for type D mud (barite mud).

TABLE 10

TESTING OF RHEOLOGICAL PROPERTIES

Type AC Mud

Mud Composition

60% (by weight) type A mud + 40% (by weight) type C mud
i.e., weighting material is a mixture of ilmenite and itibirite

Mud Weight = 14 ppg

pH Value = 9.07

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	60	0.5	30
300	36	1.0	36
200	28	1.5	42
100	19	3.0	57
6	10	50	500
3 (10 sec)	14		

Rheological Properties

Plastic Viscosity	= 24	cp
Yield Point	= 12	lb/100 sq. ft.
Initial Gel (10 sec)	= 14	lb/100 sq. ft.
10 Minutes Gel	= 31	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	10	19
Drill Collars	22	32
Drill Pipe	35	70
Solids Removal Equipment	22	45
Annulus	36	500

NOTE: No mud treatment will be necessary.

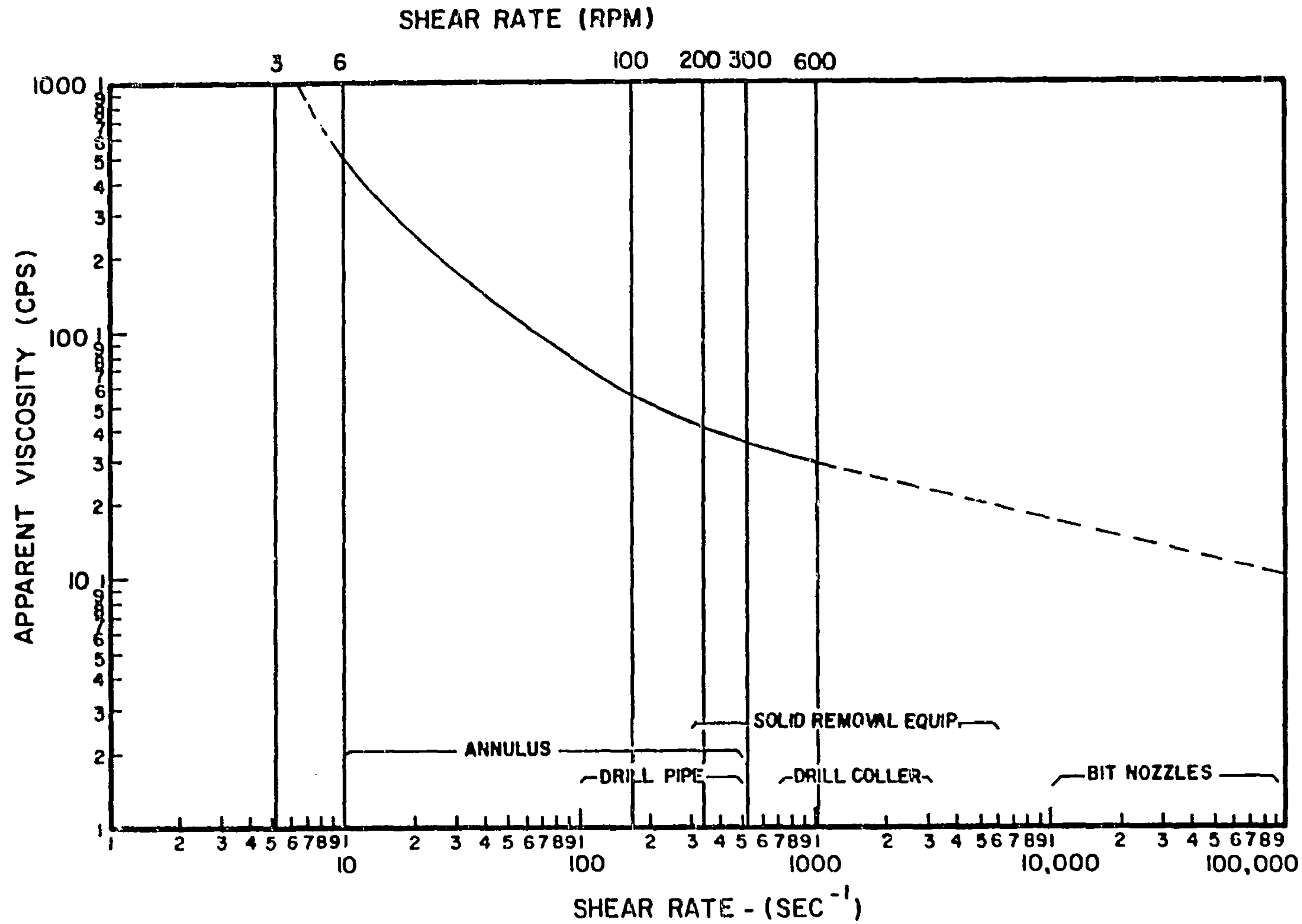


Fig. 23. The log log viscosity profile for type AC mud.

TABLE 11

TESTING OF RHEOLOGICAL PROPERTIES

Type BD Mud

Mud Composition

50% (by weight) type B mud + 50% (by weight) type D mud i.e., weighting material is a mixture of barite and K-M hematite

Mud Weight = 14 ppg

pH Value = 9.00

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	74.0	0.5	37
300	45.0	1.0	45
200	33.5	1.5	50.25
100	21.0	3.0	63.0
6	8	50	400
3 (10 sec)	5.5		

Rheological Properties

Plastic Viscosity	= 29	cp
Yield Point	= 16	lb/100 sq. ft.
Initial Gel	= 5.5	lb/100 sq. ft.
10 Minutes Gel	= 11.0	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	9	38
Drill Collars	27	41
Drill Pipe	46	90
Solids Removal Equipment	22	52
Annulus	43	400

NOTE: No mud treatment will be required.

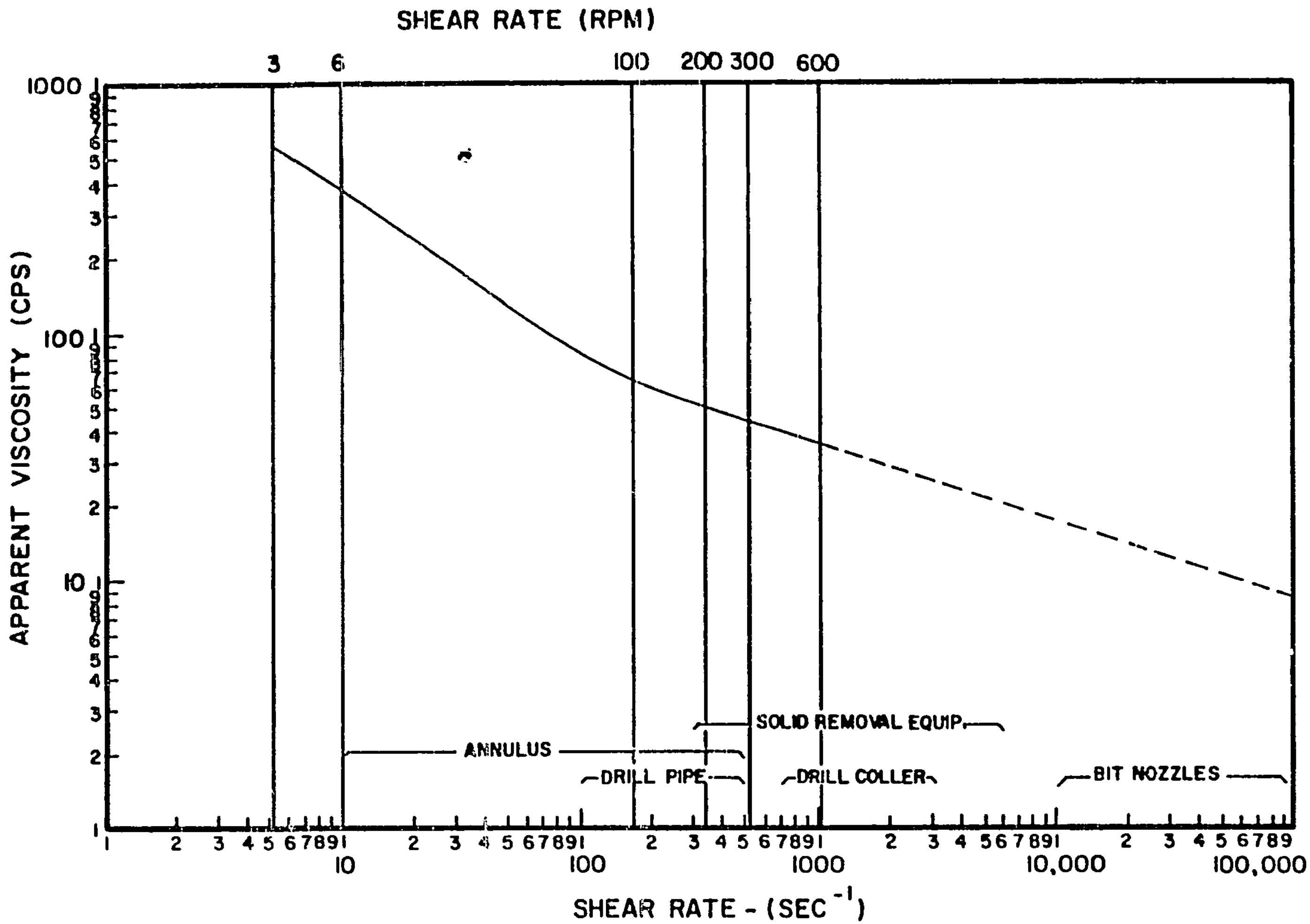


Fig. 24. The log log viscosity profile for type BD mud.

TABLE 12a

TESTING OF RHEOLOGICAL PROPERTIES

*Treated Mud Type F

Mud Composition

400 ml of type A mud + 5 ml of H-42 polymer + 2g Barafos

Mud Weight = 14 ppg

pH Value = 7.0

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	99.0	0.5	49.5
300	56.0	1.0	56.0
200	41.5	1.5	62.25
100	28.0	3.0	84.0
6	9.0	50.0	450
3	6.0	100	600

Rheological Properties

Plastic Viscosity	= 43	cp
Yield Point	= 13	lb/100 sq. ft.
Initial Gel	= 6.0	lb/100 sq. ft.
10 minutes Gel	= 12.0	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	18	30
Drill Collars	38	52
Drill Pipe	58	105
Solid Removal Equipment	35	65
Annulus	58	450

NOTE: No treatment is necessary.

*All treated muds were statically aged for 10 days.

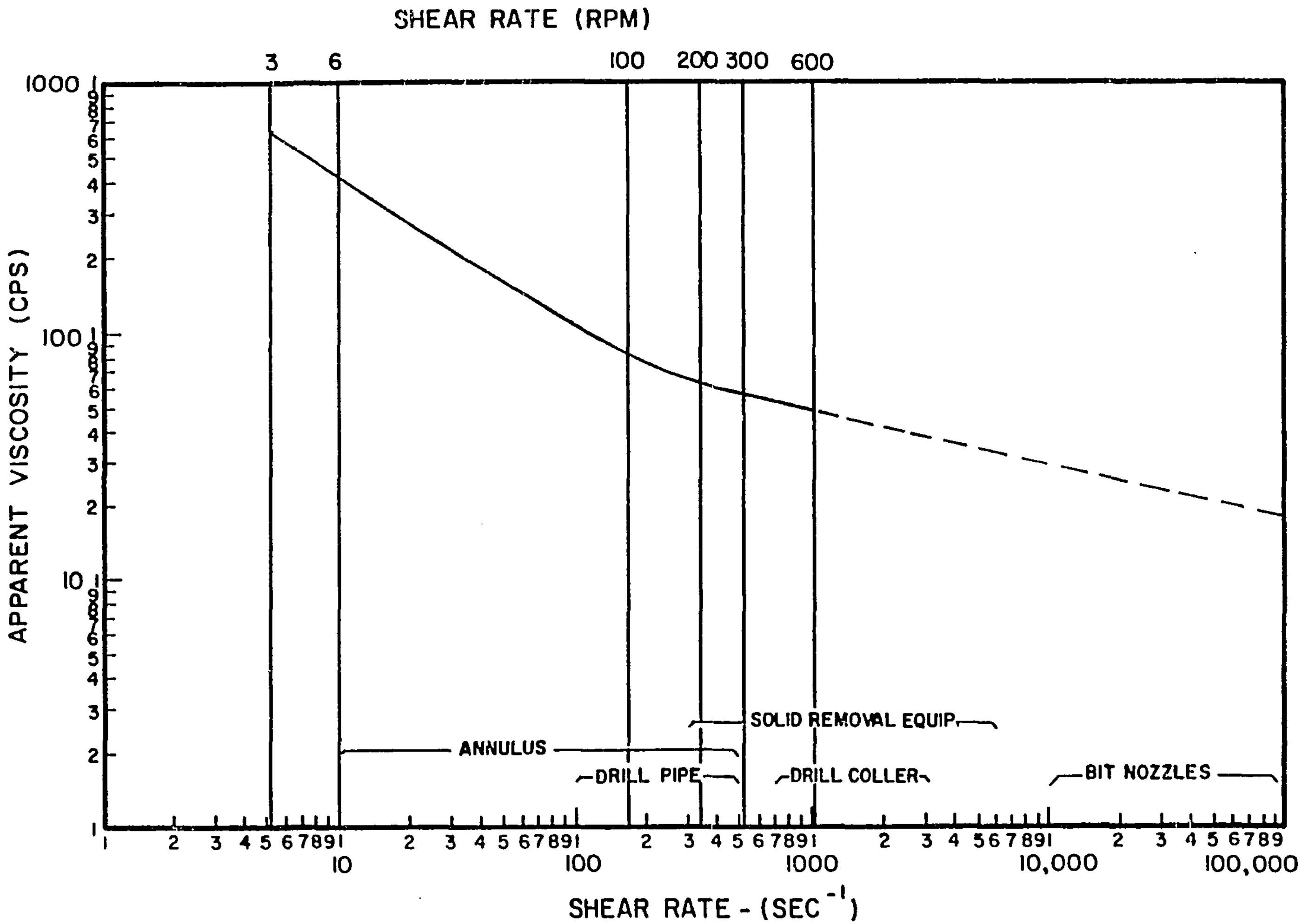


Fig. 25a. The log log viscosity profile for type F treated mud.

TABLE 12b

TESTING OF RHEOLOGICAL PROPERTIES

Treated Mud, Type G*

Mud Composition

400 ml of type B mud + 5 ml of select polymer + 4 g. Barafos

Mud weight = 14 ppg

pH value = 7.5

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Viscosity Multiplier	Apparent Viscosity (cp)
600	116	0.5	58
300	69	1.0	69
200	52	1.5	78
100	33	3.0	99
6	10	50.0	500
3	8	100.0	800

Rheological Properties

Plastic viscosity = 47 cp
Yield point = 22 lb/100 sq. ft.
Initial Gel = 8 lb/100 sq. ft.
10 minutes gel = 10 lb/100 sq. ft.

Effective Viscosity (μ_e) in centipoises

		Min	Max
Bit Nozzles	=	18	32
Drill Collars	=	42	65
Drill Pipe	=	68	120
Solid Removal Equipment	=	38	80
Annulus	=	70	500

*All treated muds were statically aged for 10 days before testing.

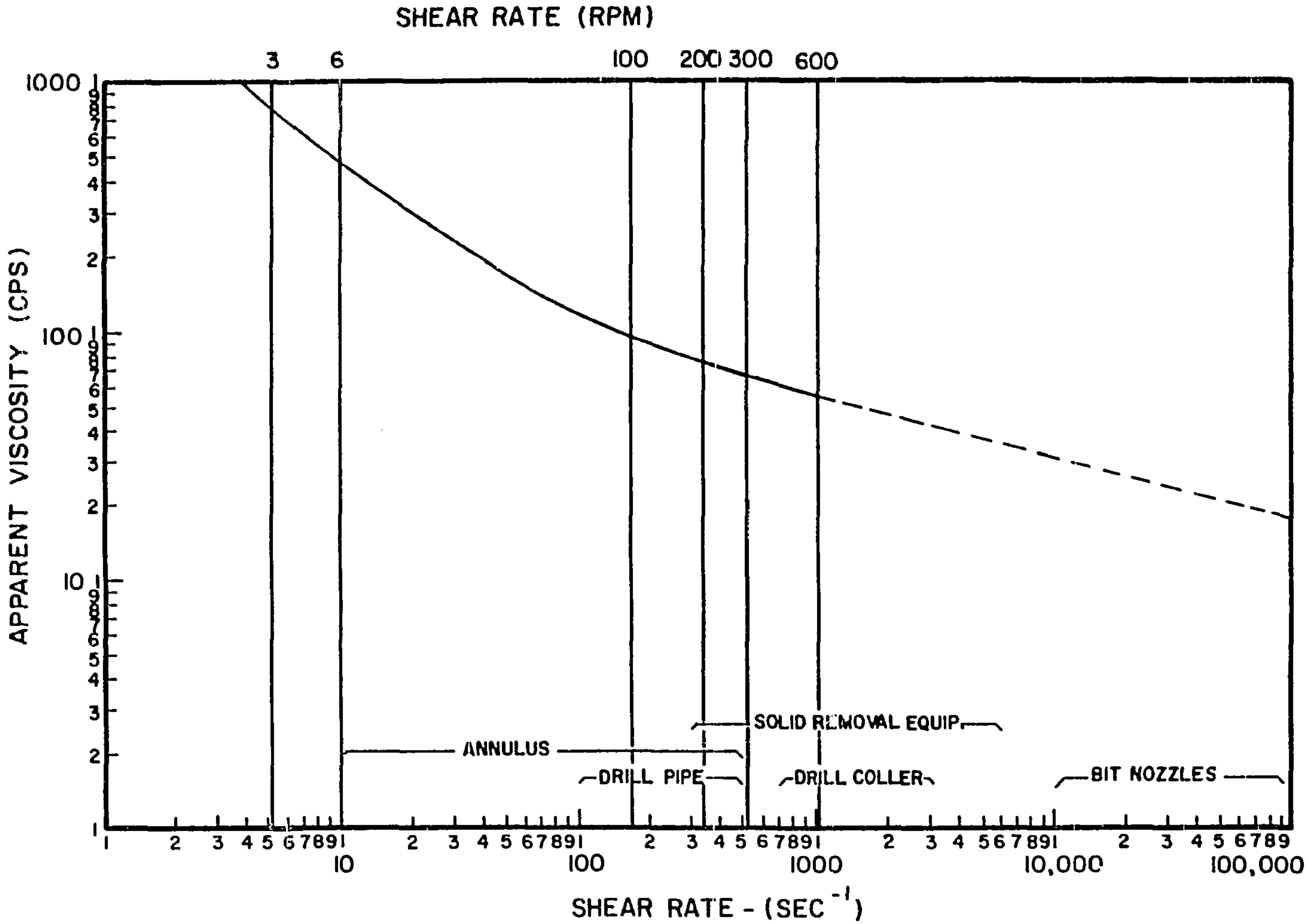


Fig. 25b. The log log viscosity profile for type G treated (K-M hematite) mud.

TABLE 12C

TESTING OF RHEOLOGICAL PROPERTIES

Treated Mud, Type H

Mud Composition

400 ml of type C mud + 5 ml of Lo-Sol polymer + 5 g. Barafos

Mud weight = 14 ppg

pH value = 8.1

Temperature = 80°F

*Apparent Viscosity

RPM	Dial Reading	Viscosity Multiplier	Apparent Viscosity(cp)
600	162	0.5	81.0
300	102	1.0	102.0
200	77	1.5	115.5
100	49	3.0	147.0
6	13	50.0	650.0
3	9	100.0	900.0

Rheological Properties

Plastic viscosity = 60 cp
Yield Point = 42 lb/100 sq. ft.
Initial gel = 9 lb/100 sq. ft.
10 minutes gel = 20 lb/100 sq. ft.

Effective viscosity (μ_e) in centipoises

		Min	Max
Bit Nozzles	=	16	35
Drill Collars	=	55	90
Drill Pipe	=	100	200
Solid Removal Equipment	=	41	130
Annulus	=	100	650

*All treated muds were statically aged for 10 days before testing.

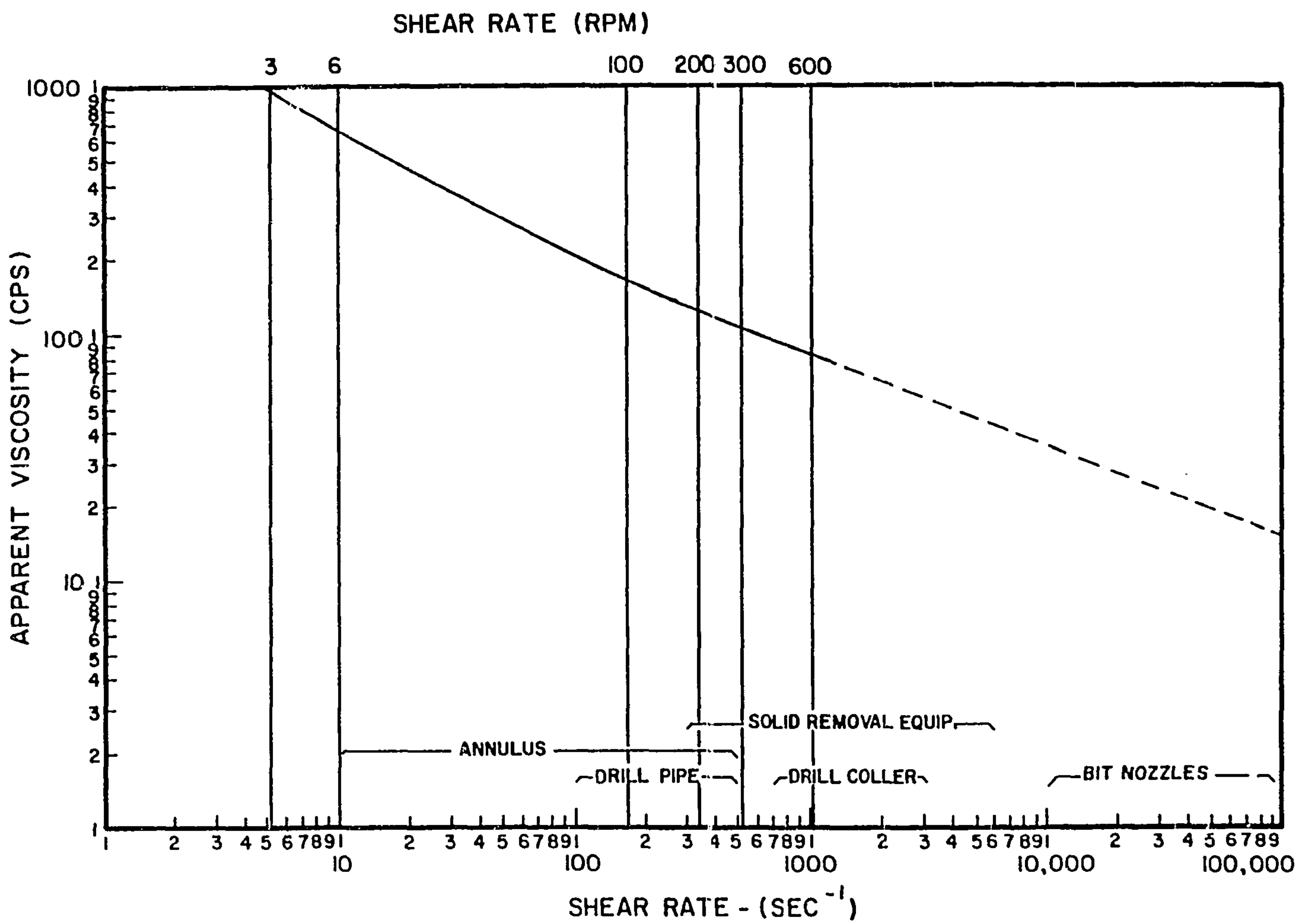


Fig. 25c. The log log viscosity profile for type H (itibirite weighted) mud.

TABLE 12d

TESTING OF RHEOLOGICAL PROPERTIES

Treated Mud, Type I*

Mud Composition

400 ml of type D mud + 5 ml of Lo-Sol polymer + 5 g. Barafos

Mud weight = 14 ppg

pH value = 7.9

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Viscosity Multiplier	Apparent Viscosity (cp)
600	157	0.5	78.5
300	100	1.0	100.0
200	79	1.5	118.5
100	54	3.0	162.0
6	14.5	50.0	725.0
3	9	100.0	900.0

Rheological Properties

Plastic viscosity = 57 cp
Yield point = 43 lb/100 sq. ft.
Initial gel = 9 lb/100 sq. ft.
10 minutes gel = 40 lb/100 sq. ft.

Effective viscosity (μ_e) in centipoises

		Min	Max
Bit Nozzles	=	17	36
Drill Collars	=	50	90
Drill Pipe	=	100	190
Solid Removal Equipment	=	45	110
Annulus	=	95	725

*All treated mud types were statically aged for 10 days

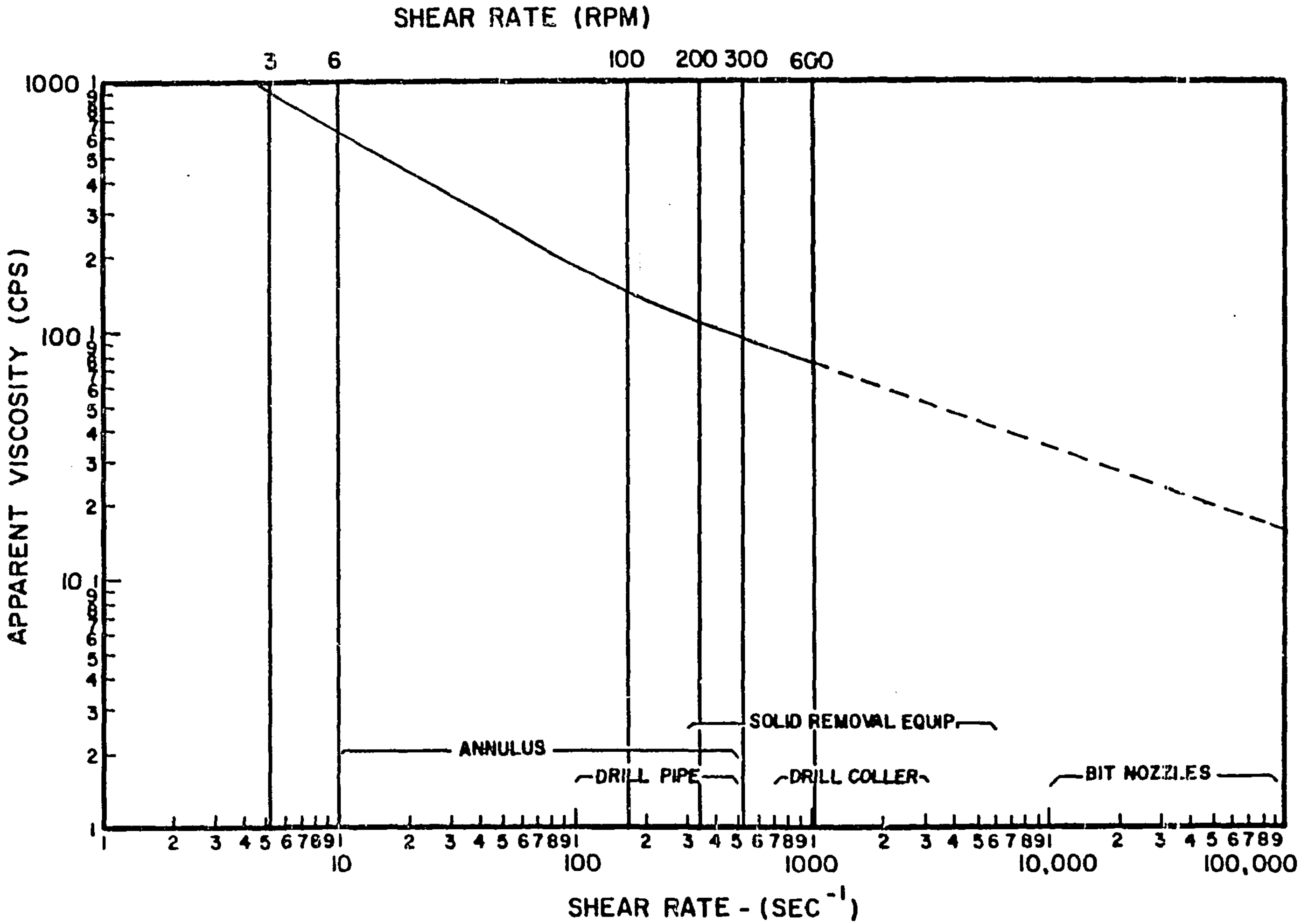


Fig. 25d. The viscosity profile for treated, type I (barite weighted) mud.

TABLE 13

TESTING OF RHEOLOGICAL PROPERTIES

Treated Mud Type FH

400 ml type AC mud + 5 ml of H-42 polymer + 3g Barafos

Mud Weight = 14 ppg

pH Value = 7.0

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	150	0.5	75.0
300	89	1.0	89.0
200	66.5	1.5	99.0
100	41.5	3.0	124.5
6	12.0	50.0	600.0
3	10	100	1000

Rheological Properties

Plastic Viscosity	= 61	cp
Yield Point	= 28	lb/100 sq. ft.
Initial Gel	= 10	lb/100 sq. ft.
10 minutes Gel	= 11.5	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoises

	Minimum	Maximum
Bit Nozzles	25	43
Drill Collars	60	82
Drill Pipe	90	110
Solids Removal Equipment	50	100
Annulus	90	600

NOTE: Treatment may be required at bit nozzles depending on depth and bottom hole temperature.

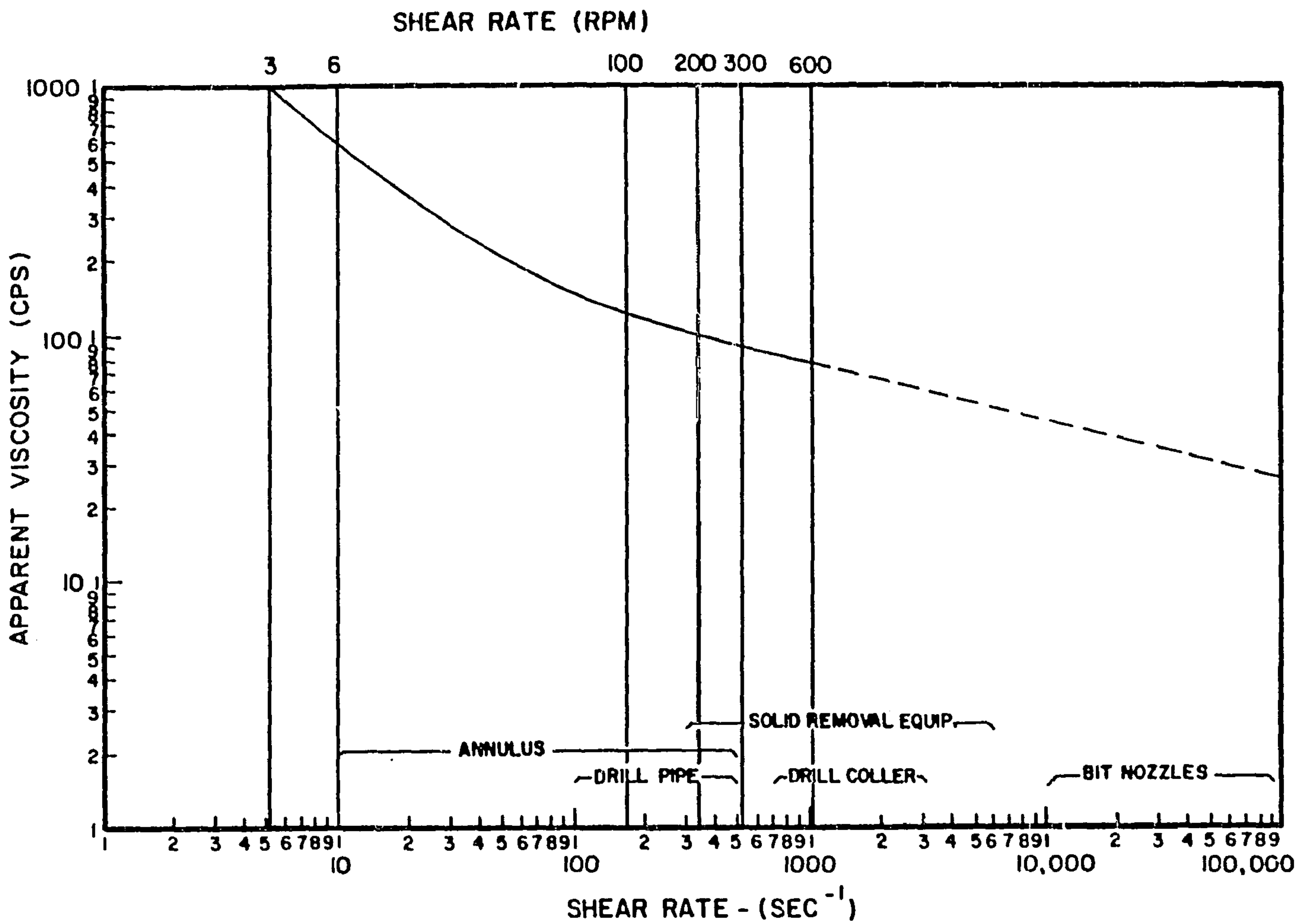


Fig. 26. The log log viscosity profile for type FH treated mud.

TABLE 14

TESTING OF RHEOLOGICAL PROPERTIES

*Treated Mud Type GI

Mud Composition

400 ml of type BD mud + 5 ml of Lo-Sol polymer + 6g Barafos + 1g desco

Mud Weight = 14 ppg

pH Value = 7.35

Temperature = 80°F

Apparent Viscosity

RPM	Dial Reading	Visc. Multiplier	Apparent Viscosity (cp)
600	160	0.5	80
300	102	1.0	102
200	78	1.5	117
100	51	3.0	153
6	14	50	700
3	11	100	

Rheological Properties

Plastic Viscosity	= 58	cp
Yield Point	= 44	lb/100 sq. ft.
Initial Gel	= 11	lb/100 sq. ft.
10 Minutes Gel	= 28	lb/100 sq. ft.

Effective Viscosity (μ_e) in Centipoise

	Minimum	Maximum
Bit Nozzles	17	37
Drill Collars	52	90
Drill Pipe	100	190
Solids Removal Equipment	42	120
Annulus	100	700

*All treated muds were statically aged for 10 days.

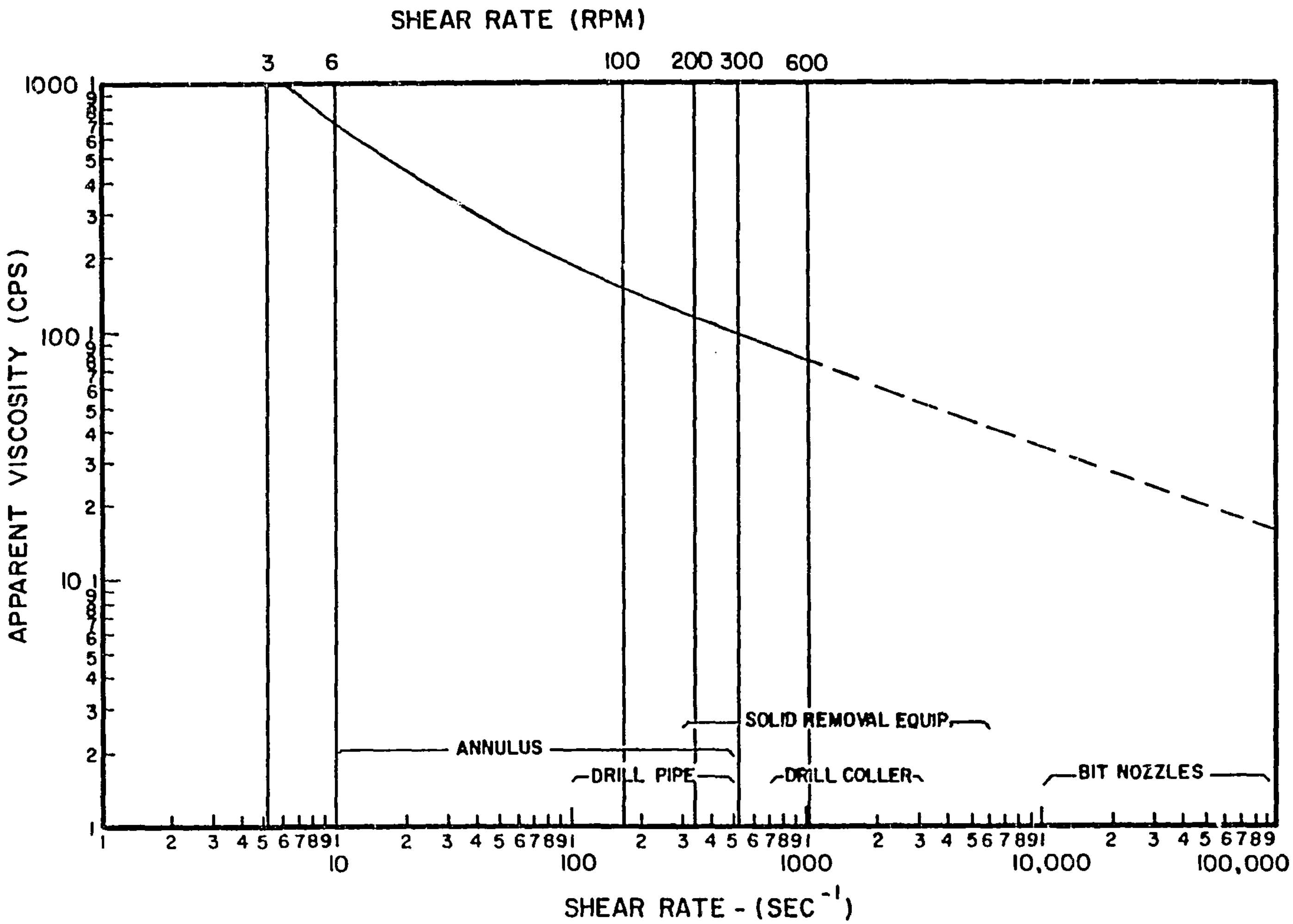


Fig. 27. The log log viscosity profile for type GI treated mud.

TABLE 15

TESTING OF RHEOLOGICAL PROPERTIES

Change of Gel Strength with Time

Composition: 3% bentonite content + weighting agent + distilled water
Mud Weight: 15 ppg
Temperature: 78°F

Weighting Agent	Rest Time in Minutes								
	10 sec.	10 m.	20	30	40	50	60	70	80
Ilmenite	36	58	65	88	82	79	100	106	114
K-M Hematite	5	23	30	35	40	44	48	49	51
Itibirite	22	80	91	97	98	102	103	103	103
Barite	29	47	53	53	53	53	53	53	53

Viscometer Readings at 3 RPM

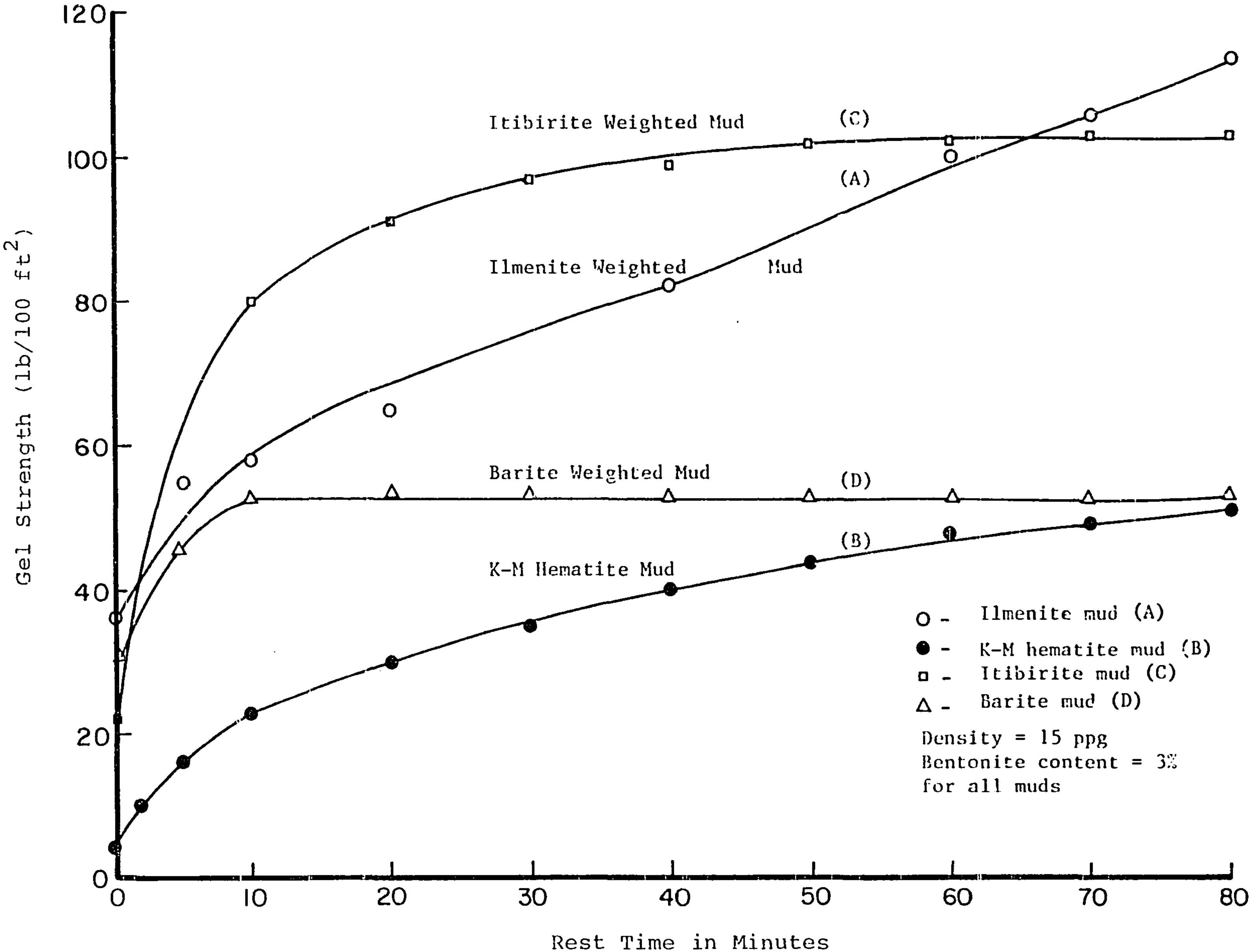


Fig. 28. Change of gel strength with time for untreated iron oxides and barite weighted muds.

TABLE 16

HT AND HP RHEOLOGICAL TESTS

Type A mud (Ilmenite weighted mud)
Mud Weight = 15 ppg
pH Value = 9.0

Rheological Characteristics

Temp. °F	Pressure PSIG	Shear Rate RPM	Shear Rate $Sec.^{-1}$	Shear Stress $Dynes/Cm^2$	Apparent Viscosity Centipoise
150	760	*3	5.1	684.0	1341.2
		100	170.3	572.0	33.6
		200	340.7	570.0	16.7
		300	511.0	566.0	11.1
		450	766.5	564.0	7.4
200		*3	5.1	756.0	1482.0
		100	170.3	740.0	43.5
		200	340.7	736.0	21.6
		300	511.0	732.0	14.3
		438	746.0	730.0	9.8
250		*3	5.1	728.0	1427.0
		100	170.3	720.0	42.3
		200	340.7	710.0	20.8
		300	511.0	700.0	13.7
		450	766.5	680.0	8.9
300		*3	5.1	636.0	1247.0
		100	170.3	600.0	35.2
		200	340.7	590.0	17.3
		300	511.0	580.0	11.3
		450	766.5	558.0	7.28

*Shear rates for determination of gels in the HP and HT viscometer.

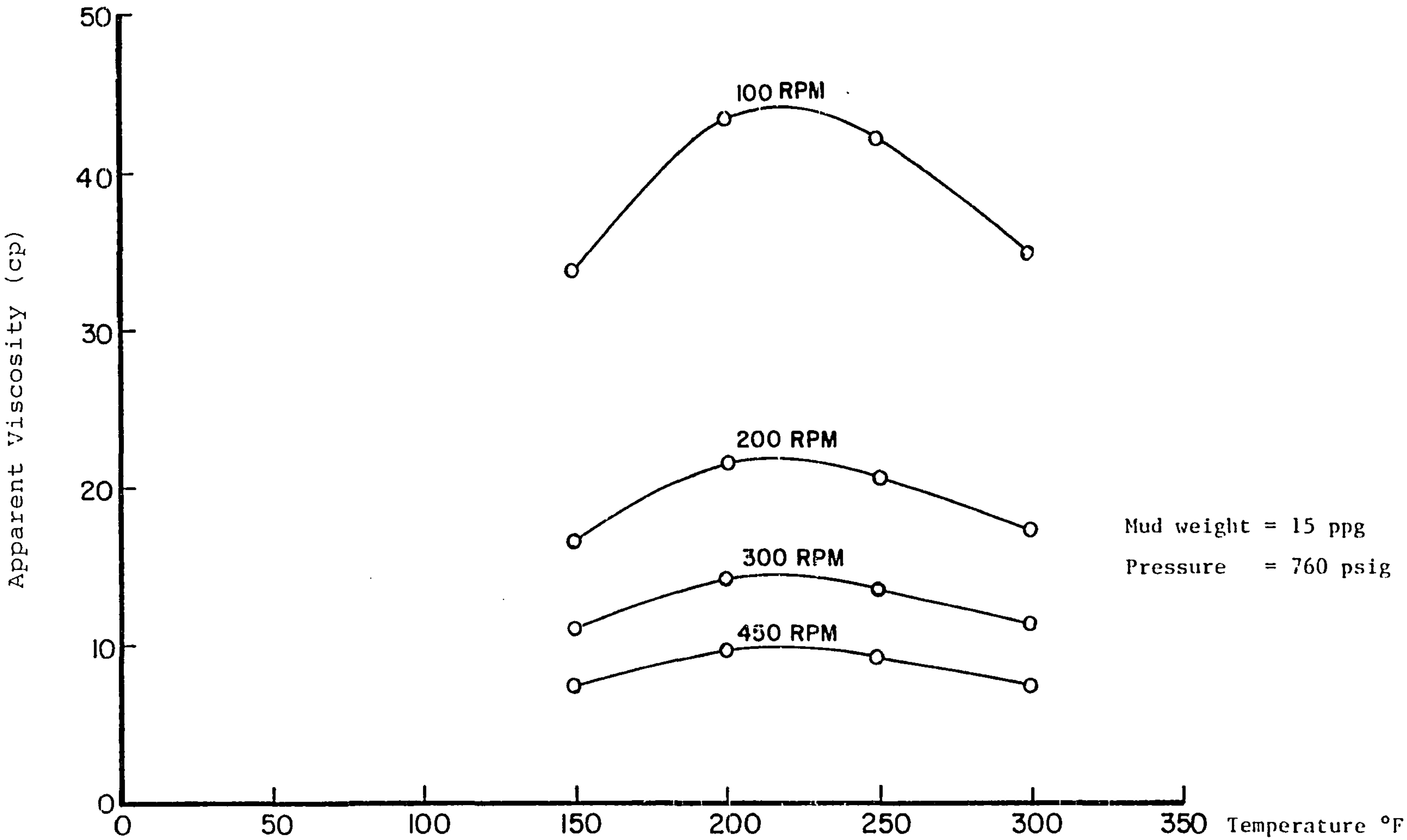


Fig. 29. Effect of temperature on viscosity of ilmenite weighted mud (type A).

TABLE 17

HT AND HP RHEOLOGICAL TESTS

Type B mud (K-M Hematite weighted mud)
Mud Weight = 15 ppg
pH Value = 9.9

Rheological Characteristics

Temp. °F	Pressure PSIG	Shear Rate RPM	Shear Rate $Sec.^{-1}$	Shear Stress $Dynes/Cm^2$	Apparent Viscosity Centipoise
150	750	*3	5.1	360.0	352.9
		100	170.3	400.0	11.9
		200	340.7	600.0	8.8
		300	511.0	632.0	6.2
		450	766.5	780.0	5.1
200	742	*3	5.1	1080.0	1058.0
		100	170.3	1620.0	47.6
		200	340.7	2120.0	31.1
		300	511.0	2360.0	23.1
		455	740.9	2440.0	16.5
250		*3	5.1	7200.0	1411.0
		100	170.3	10600.0	62.2
		200	340.7	10600.0	31.1
		300	511.0	10600.0	20.7
		450	766.5	10600.0	13.8
300		*3	5.1	400.0	784.3
		100	170.3	10680.0	62.7
		200	340.7	10680.0	31.1
		300	511.0	10680.0	20.7
		450	766.5	10680.0	13.8

*Shear rates for determination of gels in the HP and HT viscometer.

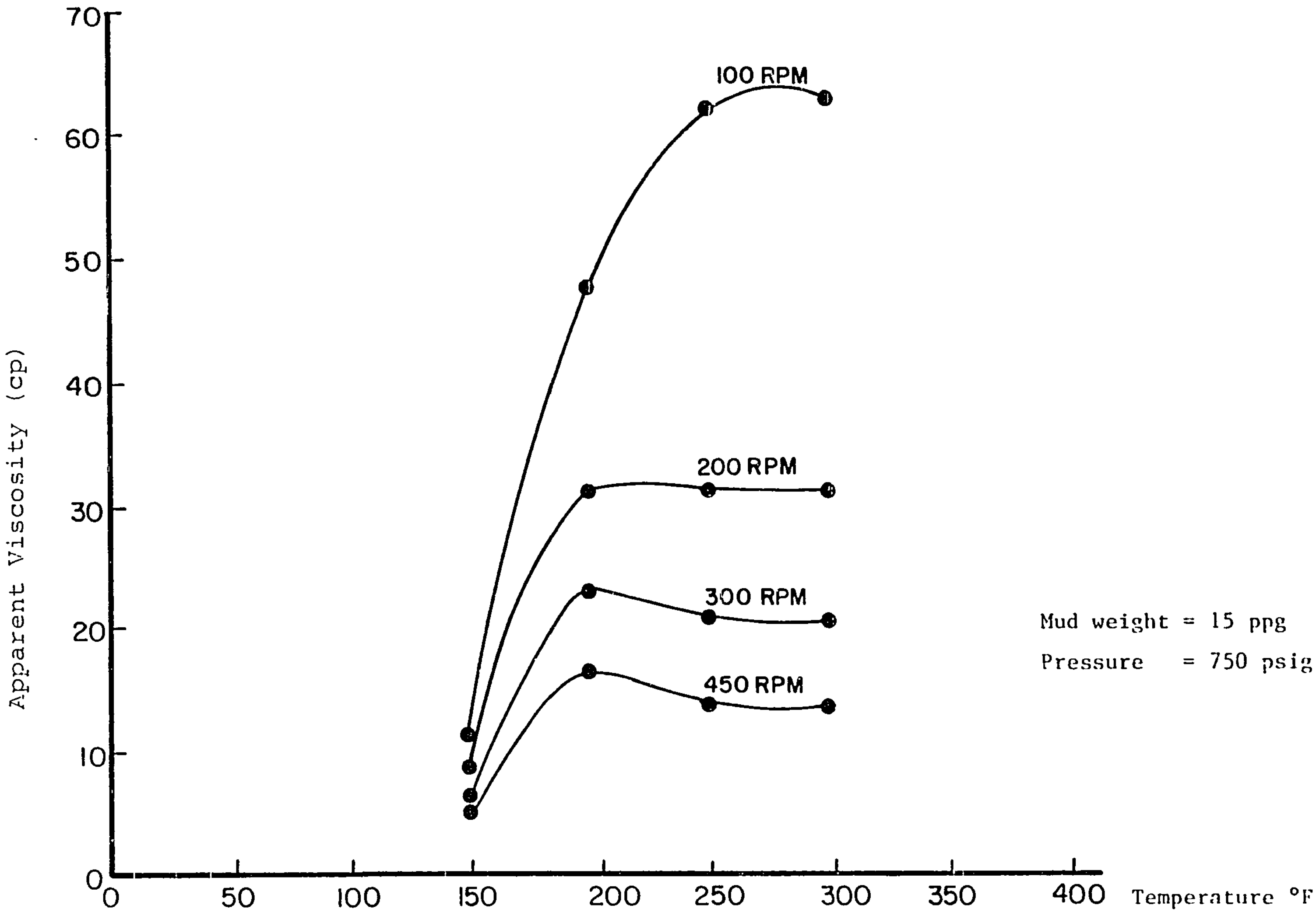


Fig. 30. Effect of temperature on viscosity of K-M hematite weighted mud (type B).

TABLE 18

HT AND HP RHEOLOGICAL TESTS

Type D mud (Barite weighted mud)
Mud Weight = 15 ppg
pH Value = 7.7

Rheological Characteristics

Temp. °F	Pressure PSIG	Shear Rate RPM	$Sec.^{-1}$	Shear Stress $Dynes/Cm^2$	Apparent Viscosity Centipoise
150	790	*3	5.1	1040	2039.2
		100	170.3	900	52.8
		200	340.7	696	28.2
		300	511.0	1036	20.3
		462	786.9	1138	14.5
200	780	*3	5.1	1560	3058.0
		100	170.3	1660	97.5
		200	340.7	1820	53.4
		300	511.0	1812	35.5
		450	766.5	1800	23.5
260	740	*3	5.1	3360	3294.1
		100	170.3	2440	71.6
		200	340.7	2980	43.7
		300	511.0	2580	25.2
		450	766.5	2080	13.6
300	790	*3	5.1	3040	2980.4
		100	170.3	2240	65.8
		200	340.7	2272	33.3
		300	511.0	2140	20.9
		450	766.5	1950	12.7
350	800	*3	5.1	2284	2533.3
		100	170.3	2280	66.9
		200	340.0	2500	36.7
		300	511.0	2480	24.3
		450	766.5	2340	15.3
400	800	*3	5.1	1820	1784.0
		100	170.3	2088	61.3
		200	340.7	2160	31.7
		300	511.0	2088	20.4
		450	766.5	1960	12.8

*Shear rates for determination of gels in the HP and HT viscometer.

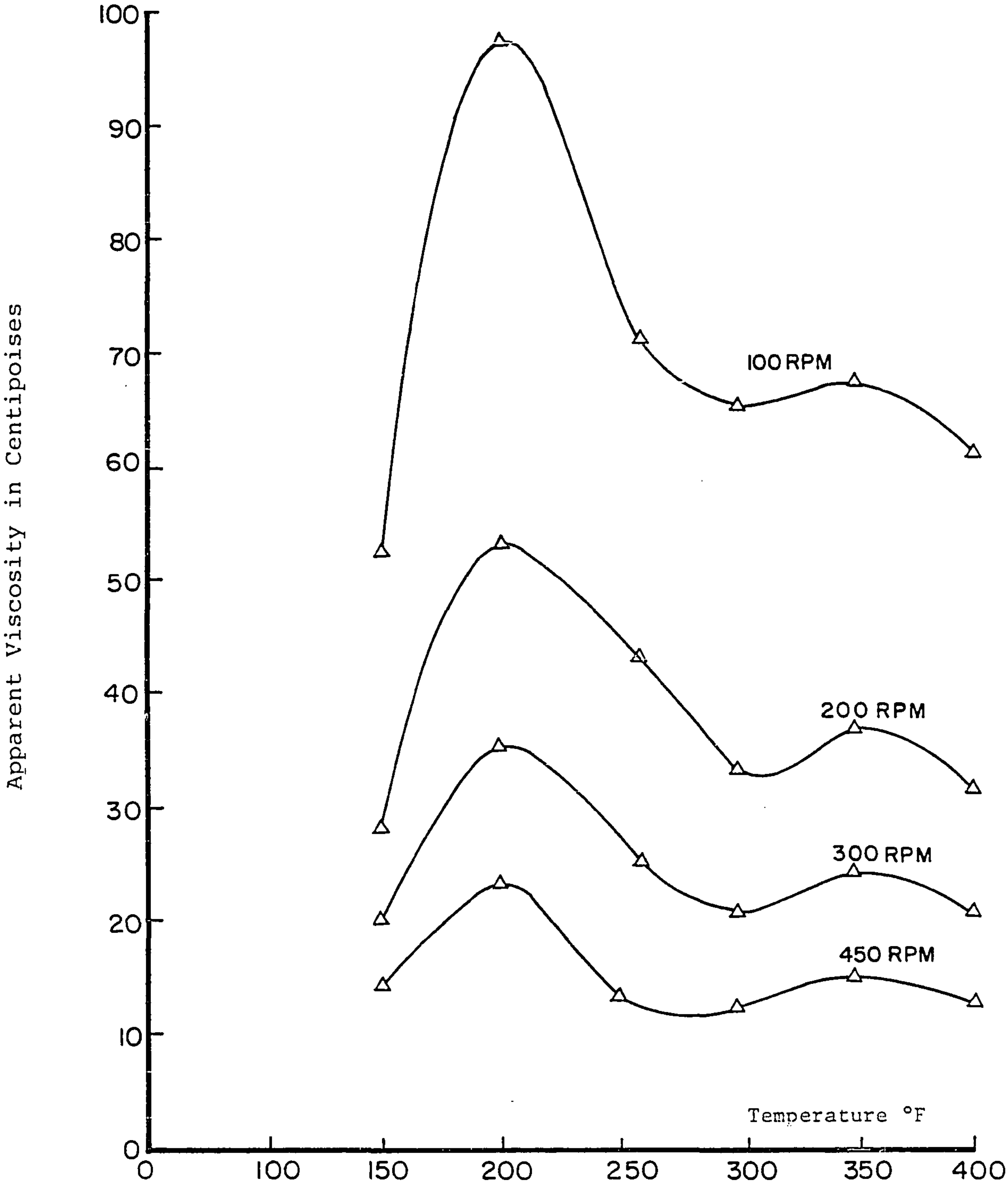


Fig. 31. Effect of temperature on viscosity of barite weighted mud.

TABLE 19

HT AND HP RHEOLOGICAL TESTS

Type AC mud (Ilmenite-Itibirite weighted mud)
Mud Weight = 15 ppg
pH Value = 9.1

Rheological Characteristics

Temp. °F	Pressure PSIG	Shear Rate RPM	Shear Rate $Sec.^{-1}$	Shear Stress $Dynes/Cm^2$	Apparent Viscosity Centipoise
150	760	*3	5.1	320	627.0
		100	170.3	380	22.3
		200	340.7	440	12.9
		300	511.0	510	10.0
		435	740.9	590	8.0
200	760	*3	5.1	816	1600.0
		100	170.3	790	46.4
		200	340.7	760	22.3
		300	511.0	740	14.4
		465	792.0	700	8.8
250	770	*3	5.1	1080	2117.0
		100	170.3	1050	61.7
		200	340.7	1010	29.6
		300	511.0	964	18.9
		480	817.6	920	11.3
300		*3	5.1	830	1627.0
		100	170.3	826	48.5
		200	340.7	824	24.2
		300	511.0	820	16.0
		440	749.4	818	10.9
350	785	*3	5.1	440	880.0
		100	170.3	790	46.4
		200	340.7	784	23.0
		300	511.0	790	15.5
		450	740.9	780	10.5

*Shear rates for determination of gels in the HP and HT viscometer.

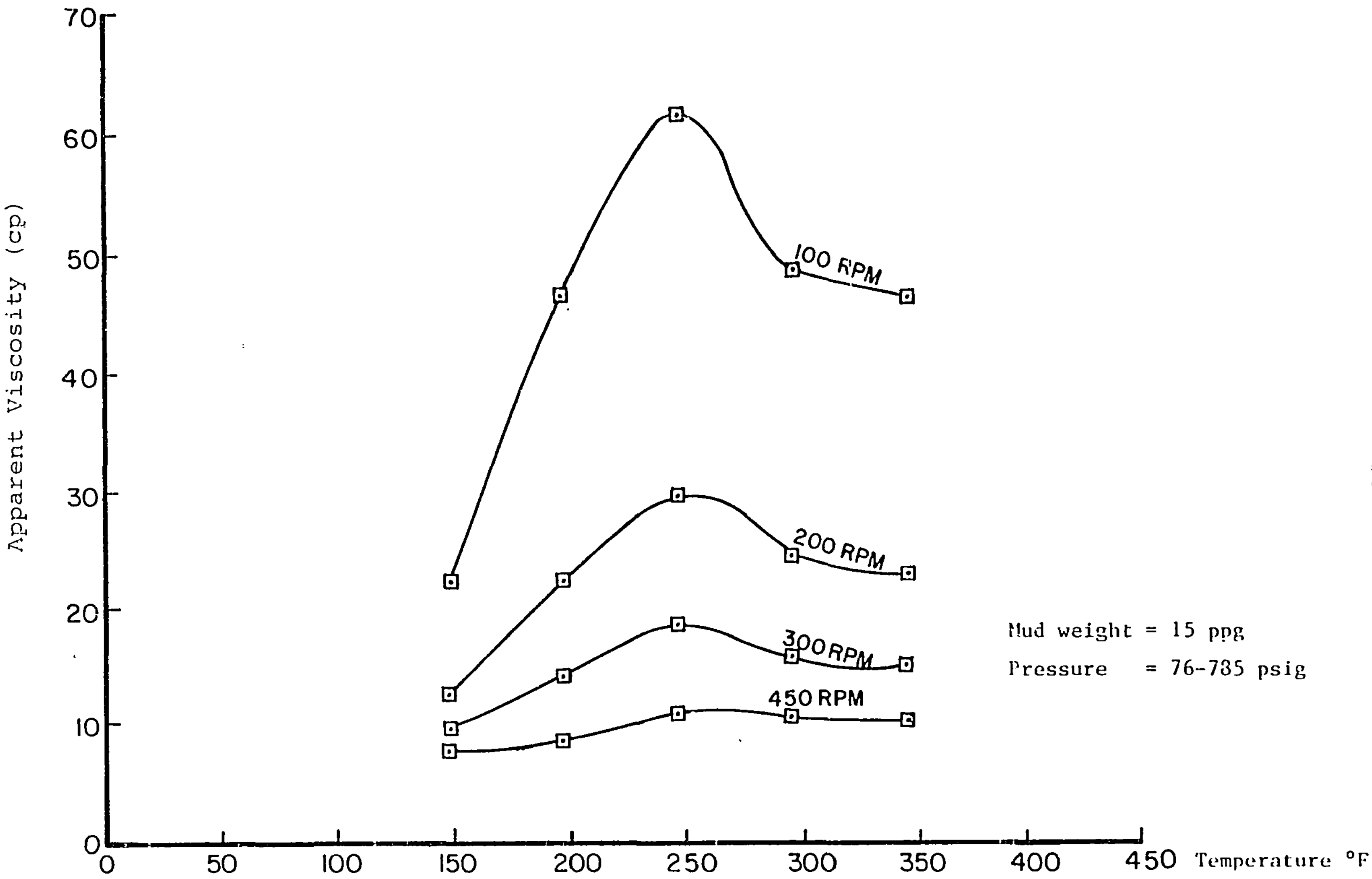


Fig. 32. Effect of temperature on viscosity of ilmenite-itibirite weighted mud (type AC).

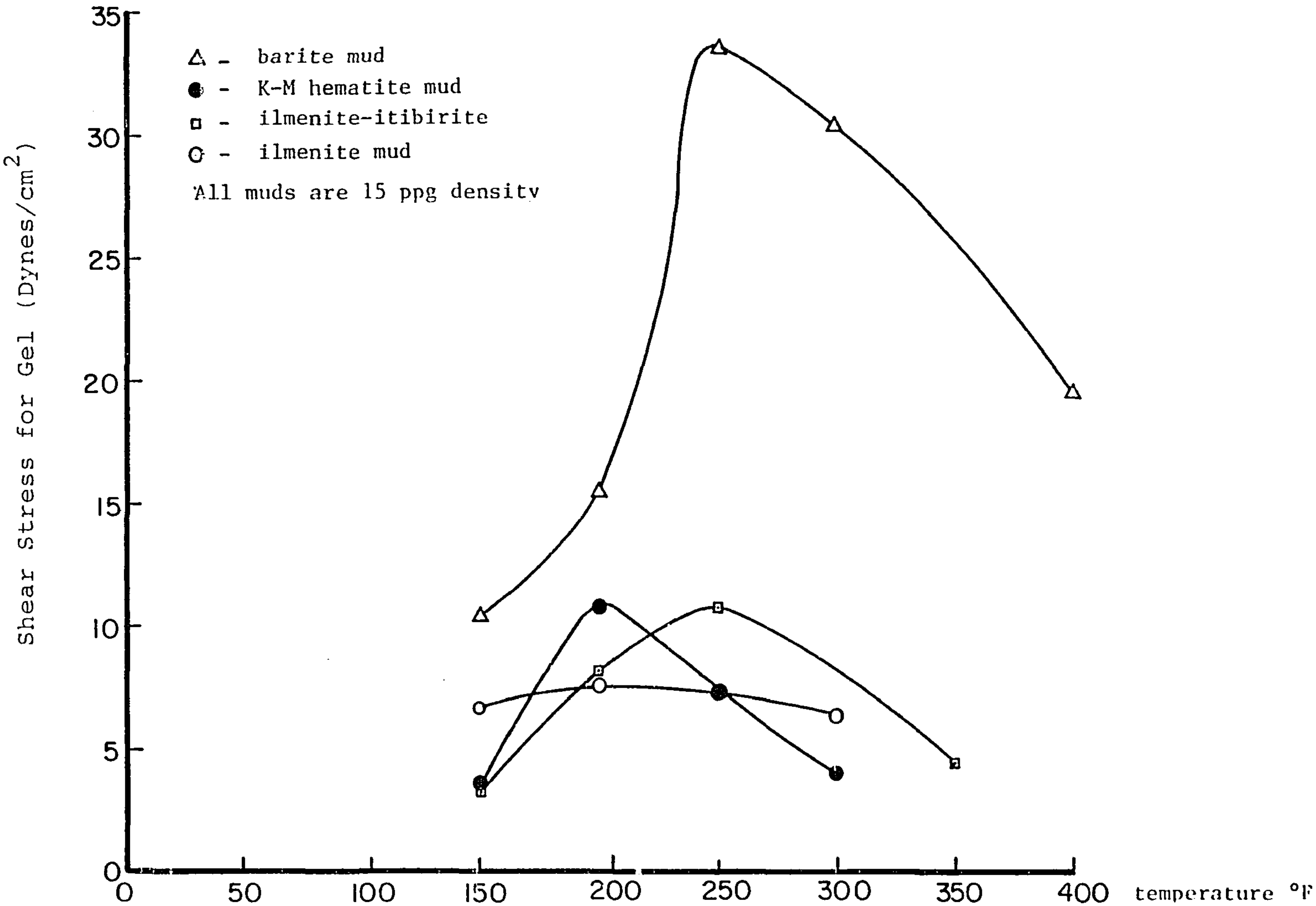


Fig. 33. Temperature - gel strength relationships for untreated mud.

TABLE 20

RESULTS OF RESISTIVITY MEASUREMENTS

Mud Weight: 14 ppg
Porosity of Berea Sandstone: 21.9 percent
Ambient Temperature: 80°F

Mud Type	Weighting Agent	pH	Average Resistivity of Filterate Saturated Cores Ohm Meter	Resistivity of Mud (80°F) Ohm Meter	Resistivity of Ages Mud (200°F) Ohm Meter	Resistivity of Ages Mud at 300°F Ohm Meter
A	Ilmenite	9.08	45.66	7.50	6.87	5.52
B	K-M Hematite	9.53	70.65	8.92	7.54	6.52
C	Itibirite	8.40	49.28	9.00	7.85	6.58
*D	Barite	9.50	68.16	9.58	9.24	8.92
	Brine	-	4.53	-	-	-

*Caustic soda was added to raise pH value from 7.75 to 9.50.

TABLE 21

MAGNETIC PERMEABILITY MEASUREMENTS
A - Drilling Fluids

Medium	(10^{-12} Farad) Capacity (C)	(Kilocycles) frequency (f)	Q (Coulomb) Charge density	$1/f^2$ (KC^{-2}) (x 10^{-6})
Air	50	276	41	13
	100	218	45	21
	150	185	46	29
	200	164	46	37
	250	148	46	46
	300	137	45	53
	350	128	44	61
	400	120	42	69
Ilmenite	880	80.5	18.0	154 x 10^{-6}
Mud	900	79	17.0	160
MW = 15 ppg	940	78.5	18.0	162
	1000	76	18.0	173
	1050	75	18.0	178
	1100	73	18.0	188
	1120	72.5	19.0	190
	1160	71	18.0	198
	1300	67	18.5	223
	1350	66	19.0	230
	1400	65	20.0	237
	1460	64	19.0	244
KM-Hematite	360	123	16	66
Fluid	380	122	18	67
MW = 15 ppg	400	118	16	72
	410	118	15	72
	420	117	16.5	73

TABLE 21 (continued)

Medium	(10^{-12} Farad) Capacity (C)	(Kilocycles) frequency (f)	Q (Coulomb) Charge density	$1/f^2$ (KC^{-2}) (x 10^{-6})
	430	115.5	16.5	75
	440	114	15	77
	450	113	17	78
	460	110.5	17	82
	480	108.2	17	85
N-Hematite	400	118	16	72 x 10^{-6}
Mud	420	115	15.5	76
MW = 15 ppg	440	112	15.5	80
	450	111	15.0	81
	460	110	15.0	83
	480	108	15.0	86
	680	90	15.5	120
	780	86	15.0	135
	830	84	15.0	142
	880	81	14.0	150
	980	77	15.0	168
Barite	1030	76.5	16	171
Mud	1060	75	15	178
	1100	74	15	183
	1140	73	16.5	188
	1180	71	15	198
	1240	70	15	204
	1300	68	14	216
	1400	65.5	15	233
	1440	65	15	237
	1480	64	16	244

TABLE 21 (continued)

Medium	(10^{-12} Farad) Capacity (C)	(Kilocycles) frequency (f)	Q (Coulomb) Charge density	$1/f^2$ (KC^{-2}) ($\times 10^{-6}$)
N. Hematite	400	118	16.0	72
	420	115	15.8	76
	440	112	15.5	80
	450	111	15.0	81
	460	110	15.0	83
	480	108	15.0	86
	680	90	15.5	120
	780	86	15.0	135
	830	84	15.0	142
	880	81	14.0	150
	980	77	15.0	168
Barite	1030	76.5	16.0	171
	1060	75.0	15.0	178
	1100	74.0	15.0	183
	1140	73.0	16.5	188
	1180	71.0	15.0	198
	1220	70.0	15.0	204
	1300	68.0	14.0	216
	1400	65.5	15.0	233
	1440	65.0	15.0	237
	1480	64.0	16.0	244

TABLE 21 (continued)

MAGNETIC PERMEABILITY MEASUREMENTS
B- Weighting Material (uncompacted powder)

Medium	(10^{-12} Farad) Capacity (C)	(Kilocycles) frequency (f)	Q (Coulomb) Charge density	$1/f^2$ (KC^{-2}) (x 10^{-6})
Ilmenite	880	80.5	18.0	154
	900	79.0	17.0	160
	940	78.5	18.0	162
	1000	76.0	18.0	173
	1050	75.0	18.0	178
	1100	73.0	18.0	188
	1120	72.5	19.0	190
	1160	71.0	18.0	198
	1300	67.0	20.0	233
	1350	66.0	19.0	230
	1400	65.0	18.5	237
	1460	64.0	19.0	244
KM-Hematite	100	216	31	21.4
	120	201	37	24.8
	140	190	37	27.7
	160	180	34	30.9
	180	171	36	34.2
	200	163	33	37.6
	220	156	34	41.1
	240	151	35	43.9
	280	141	36	50.3
	320	133	33	56.5
	360	126	36	63.0
	400	120	33	69.4
	440	114	34	76.9
	480	110	34	82.6

TABLE 22

MAGNETIC PERMEABILITY CALCULATION

	Media	(1/slope)	Relative Perm μ_r	Permeability $\mu = 4\pi * 10^{-7}\mu_r$ (henry/meter)
*A	Air	0.165000	1.000,0004	12.47×10^{-7}
	Barite Mud	0.166429	1.0087	12.68×10^{-7}
	Iron Oxide Mud	0.172143	1.0433	13.11×10^{-7}
**B	Air	0.164,000	1.000,0004	12.57×10^{-7}
	Barite (powder)	0.1666,667	1.016260	12.77×10^{-7}
	Iron Oxides (powder)	0.171,333	1.044,713	13.13×10^{-7}

*Part A is obtained from Figure 34.
**Part B is obtained from Figure 35.

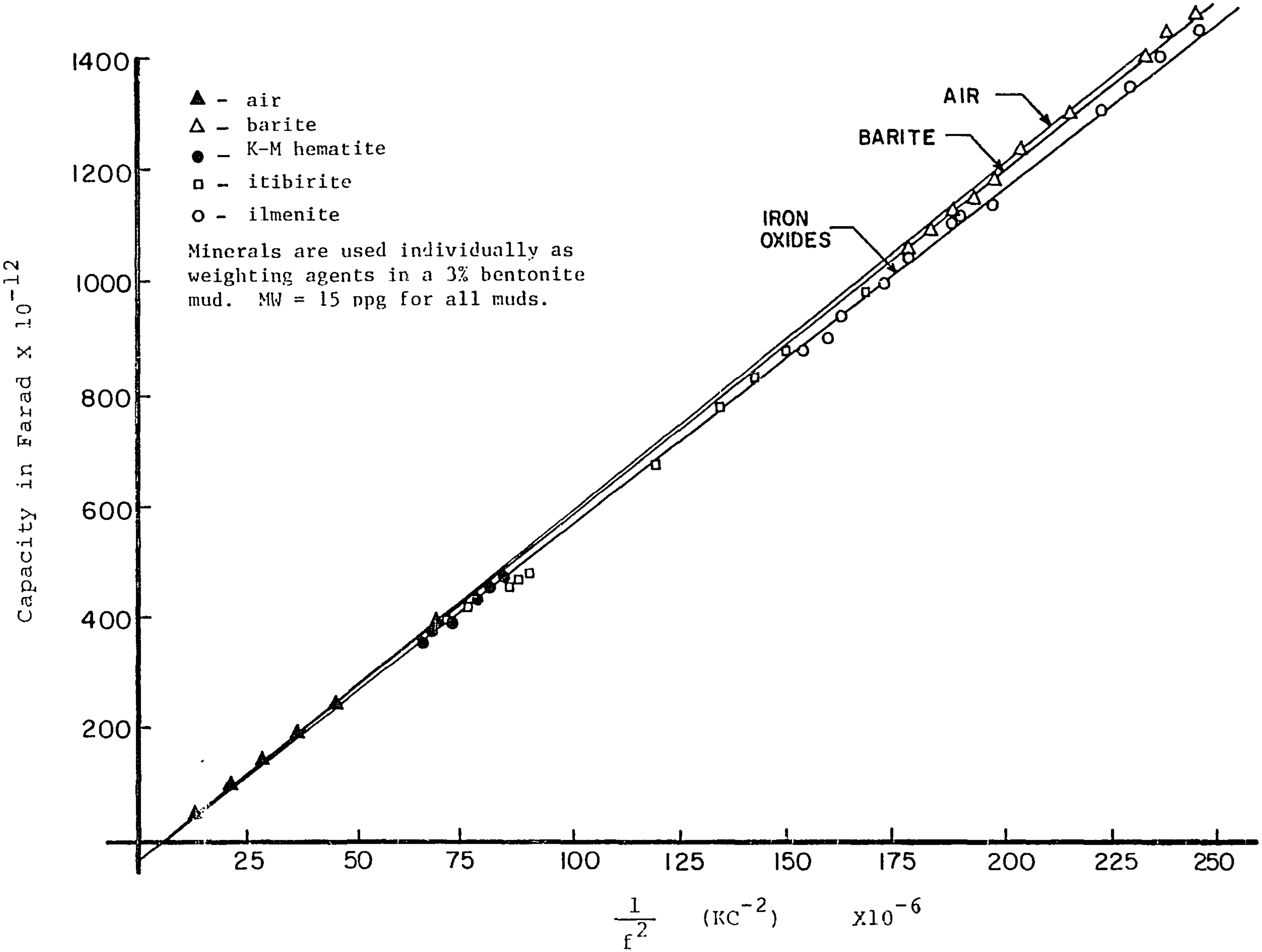


Fig. 34. Magnetic permeability for drilling fluids.

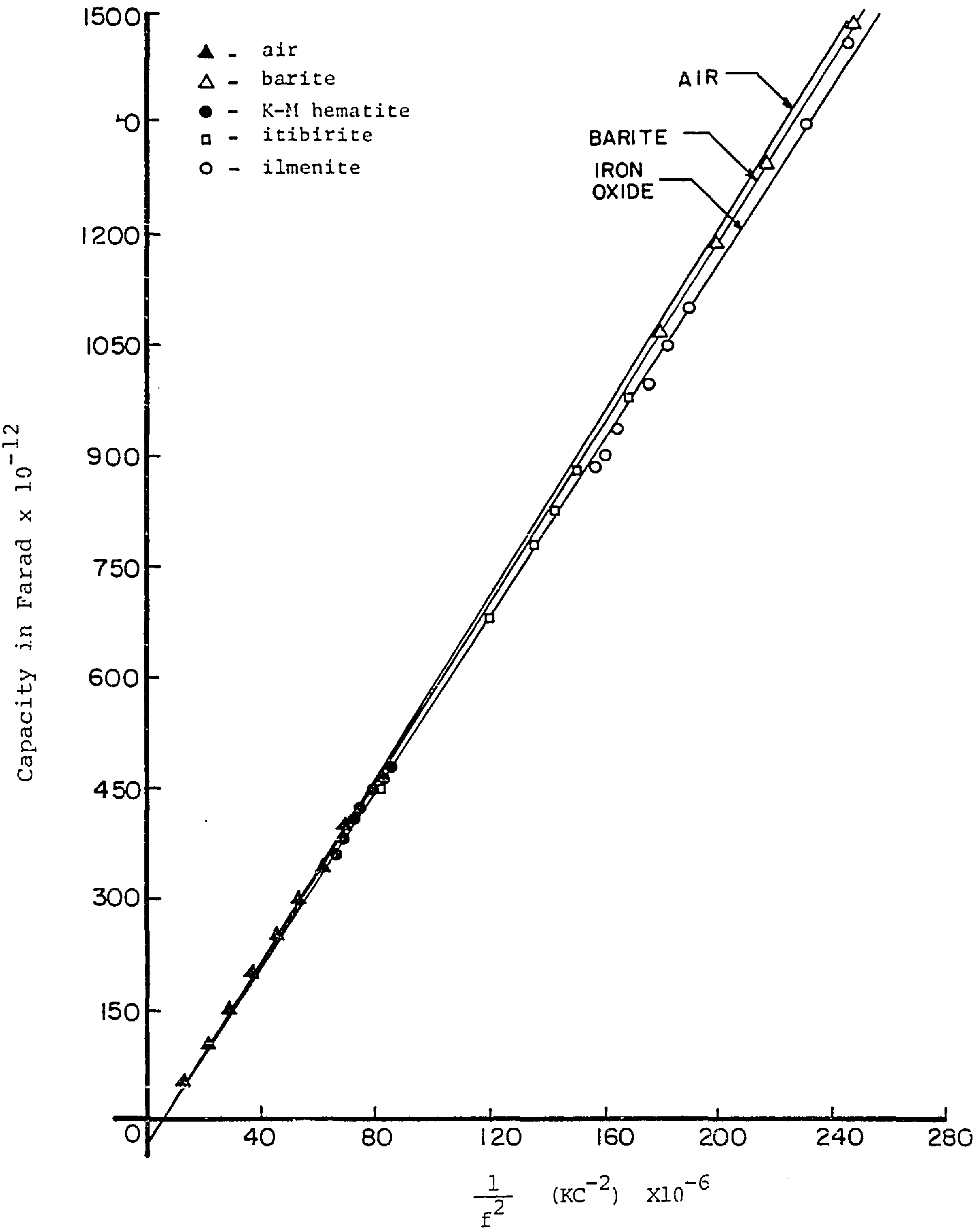


Fig. 35. Magnetic permeability for weighting materials.

TABLE 23

LOW TEMPERATURE FILTRATION TESTS

Mud condition = Untreated mud
Mud types = A, B, C, D, AC, and BD
Mud weight = 14 ppg
Temperature = 80°F
Pressure = 100 psi

Time Minutes	Weighting Material					
	(A) Ilmenite	(B) K-M Hem.	(C) Itibirite	(D) Barite	(AC) Ilm/Itib.	(BD) Barite/KM-Hem.
2	5.6	3.1	3.3	2.4	5.2	3.4
5	10.2	5.0	5.2	3.8	6.8	4.6
10	14.4	7.4	7.4	5.6	9.6	6.9
15	18.0	9.2	9.4	7.1	11.8	7.6
20	20.4	9.8	10.9	8.0	13.3	9.7
25	22.8	12.2	12.2	9.2	15.0	11.0
30	25.0	13.4	13.5	10.2	16.6	12.4
*corrected loss (ml.)	24.6	13.7	13.5	10.5	16.4	12.7
Mud cake thick (cm)	0.62	0.34	0.5	0.26	0.60	0.35
Mud cake condition						
pH	8.51	9.56	8.77	8.56	8.65	9.0

*Obtained from fluid loss versus time graphs.

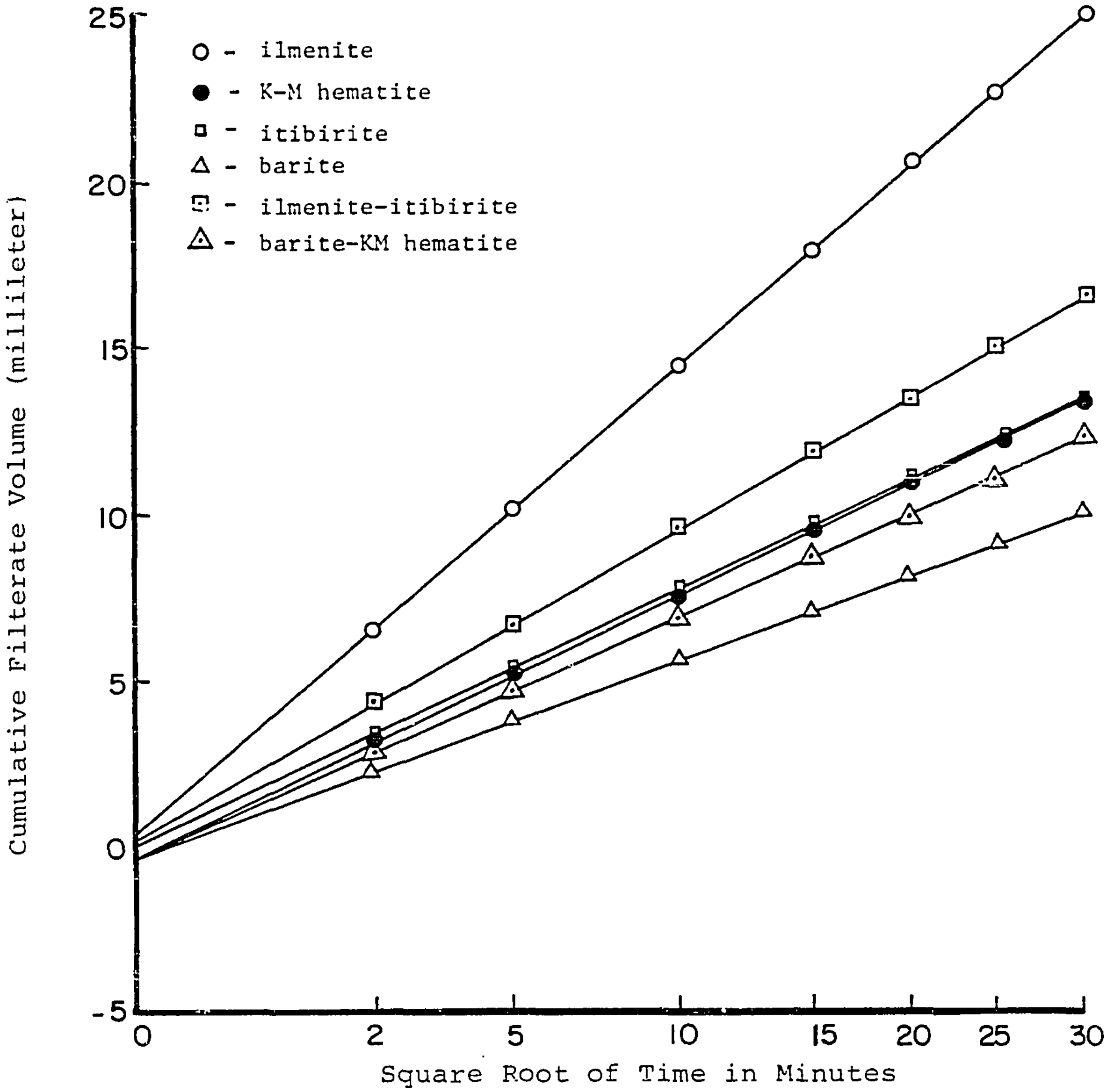


Fig. 36. A low temperature filtrate volume versus time for untreated mud.

TABLE 24

LOW TEMPERATURE FILTRATION TESTS

Mud condition = Mixed with shale and aged for 7 days
Mud types = A, B, C, D, AC and BD
Mud weight = 14 ppg
Temperature = 80°F
Pressure = 100 psi

Time Minutes	Weighting Material					
	(A) Ilmenite	(B) K-M Hem.	(C) Itibirite	(D) Barite	(AC) Ilm/Itib.	(BD) Barite/KM-Hem.
2	3.4	2.7	2.2	1.8	3.8	2.6
5	5.2	4.4	3.3	2.8	5.7	4.2
10	7.3	6.1	4.7	4.2	8.3	5.8
15	9.3	7.6	5.0	5.7	10.1	8.4
20	10.8	8.8	7.0	6.5	11.8	9.7
25	11.9	9.9	7.6	7.4	13.3	11.1
30	13.3	10.8	8.7	8.1	14.6	12.2
corrected loss (ml.)	13.3	10.8	8.7	8.7	14.6	13.2
Mud cake thickness	0.49 cm	0.27 cm	0.30 cm	0.26	0.36	0.30
Mud cake condition	poor	good	good	good	good	good
pH	8.61	9.08	8.83	8.74	8.46	8.90

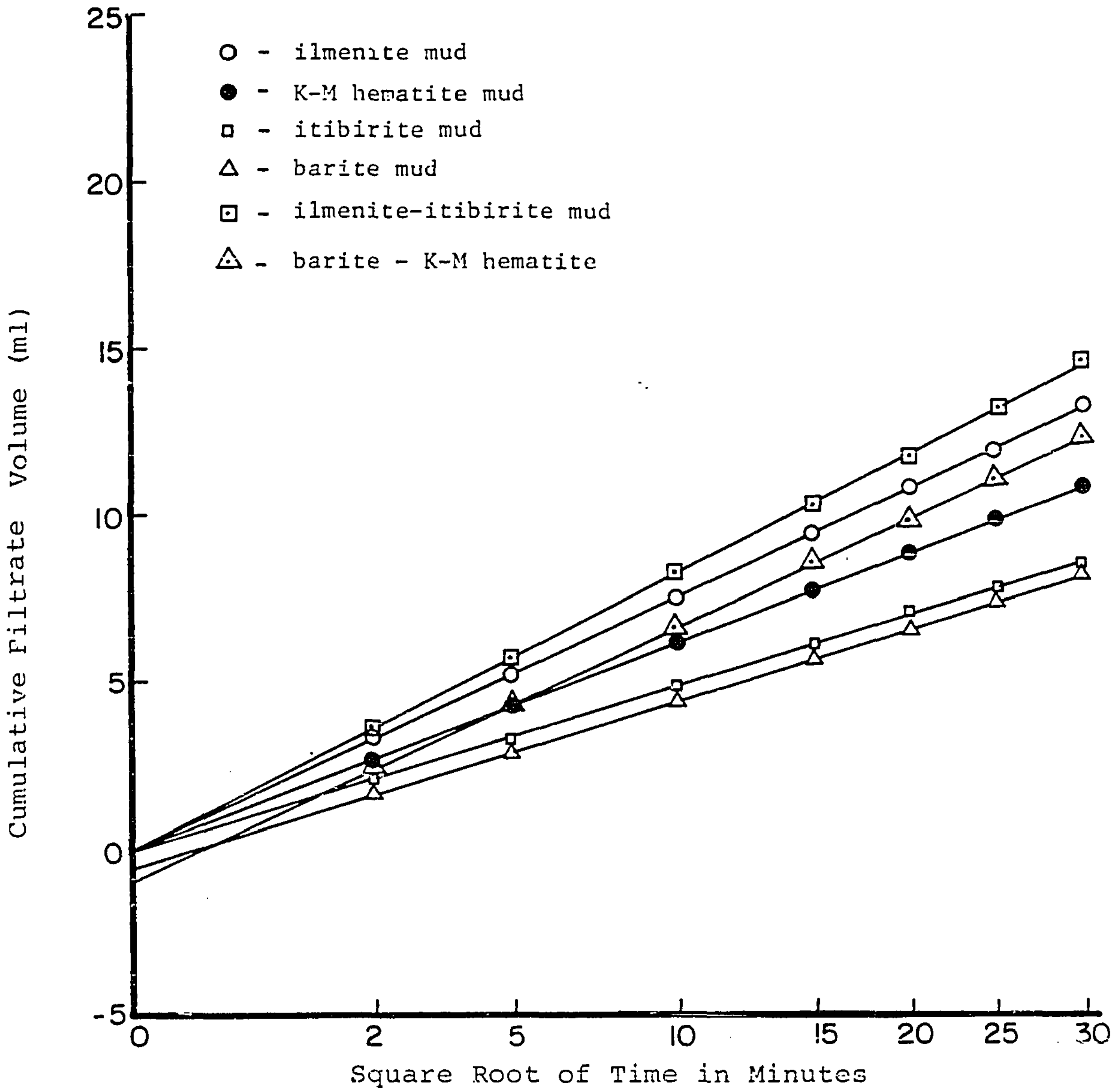


Fig. 37. Effect of mud aging with shale cuttings on low temperature filtration properties.

TABLE 25

LOW TEMPERATURE FILTRATION TESTS

Mud condition = Treated mud, i.e., polymer added + thinner
Mud types = F, G, H, I, FH and GI
Mud weight = 14 ppg
Temperature = 80°F
Pressure = 100 psi

Time Minutes	Weighting Material					
	*(F) Ilmenite	**(G) K-M Hem.	**(H) Itibirite	**(I) Barite	*(FH) Ilm/Itib.	*(GI) Barite/KM-Hem.
2	1.1	1.1	1.1	1.0	1.1	0.3
5	2.2	2.0	2.0	1.9	2.0	1.2
10	3.1	2.6	2.6	2.4	2.8	2.1
15	3.8	3.2	3.2	3.1	3.5	2.4
20	4.6	3.8	3.8	3.6	4.0	3.1
25	5.1	4.2	4.2	4.0	4.5	3.5
30	5.4	4.8	4.8	4.6	4.8	3.8
corrected loss (ml.)	5.4	4.8	4.8	4.7	4.8	4.4
Mud cake thick (cm.)	0.12	0.12	0.12	0.12	0.12	0.12
Mud cake condition	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
pH	6.92	7.73	8.77	8.8	7.24	8.04

*Mud treated with H-42 polymer.
**Mud treated with Lo-Sol polymer.

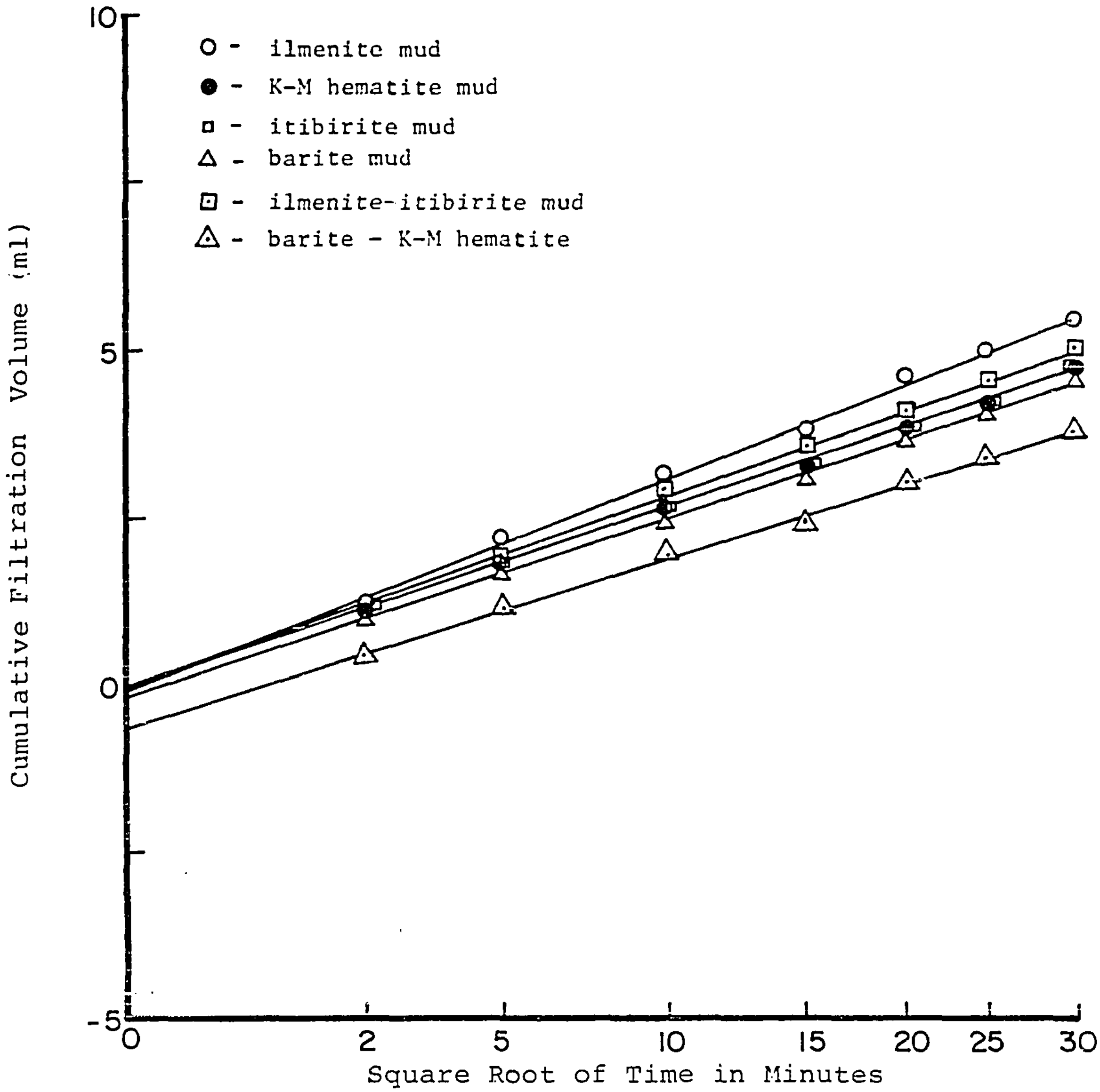


Fig. 38. Effect of grain coating on low temperature filtration tests.

TABLE 26

HIGH PRESSURE AND HIGH TEMPERATURE FILTRATION TESTS

Mud Types: A, B, C, D, AC and BD (untreated mud)
Mud Weight: 14 ppg
Temperature: 200°F
Differential Pressure (P): 500 psi

	Weighting Material					
Time (minutes)	(A) Ilmenite	(B) K-M Hematite	(C) Itibirite	(D) Barite	(AC) Ilm/Itib.	(BD) Bar./K-M Hem.
2	5.5	4.8	7.2	4.7	3.4	5.1
5	10.0	7.7	10.0	7.9	6.2	8.0
10	16.2	11.5	13.6	11.1	10.0	10.6
15	20.8	14.8	16.3	13.8	13.4	13.2
20	25.0	16.8	18.4	15.7	16.2	15.6
25	28.8	19.5	20.4	17.2	18.2	17.0
30	32.2	21.3	21.6	18.8	20.4	18.3
*5(2p)	5.8	3.7	3.2	2.8	4.4	3.8
**corrected filt. (ml.)	71.5	44.1	39.0	37.6	45.8	35.1
Mud cake thickness (cm)	1.55	0.65	1.45	0.75	1.25	0.65
Mud cake condition	incompres- sible	imcompressible	incompressible	incompressible	incompressible	incompressible
pH	8.4	8.48	8.6	8.08	8.3	8.2

*Fluid loss collected at double pressure in 5 minutes.
**Corrected filterate volume = 2(filtrate volume) + correction from the filtrate volume versus time plot.

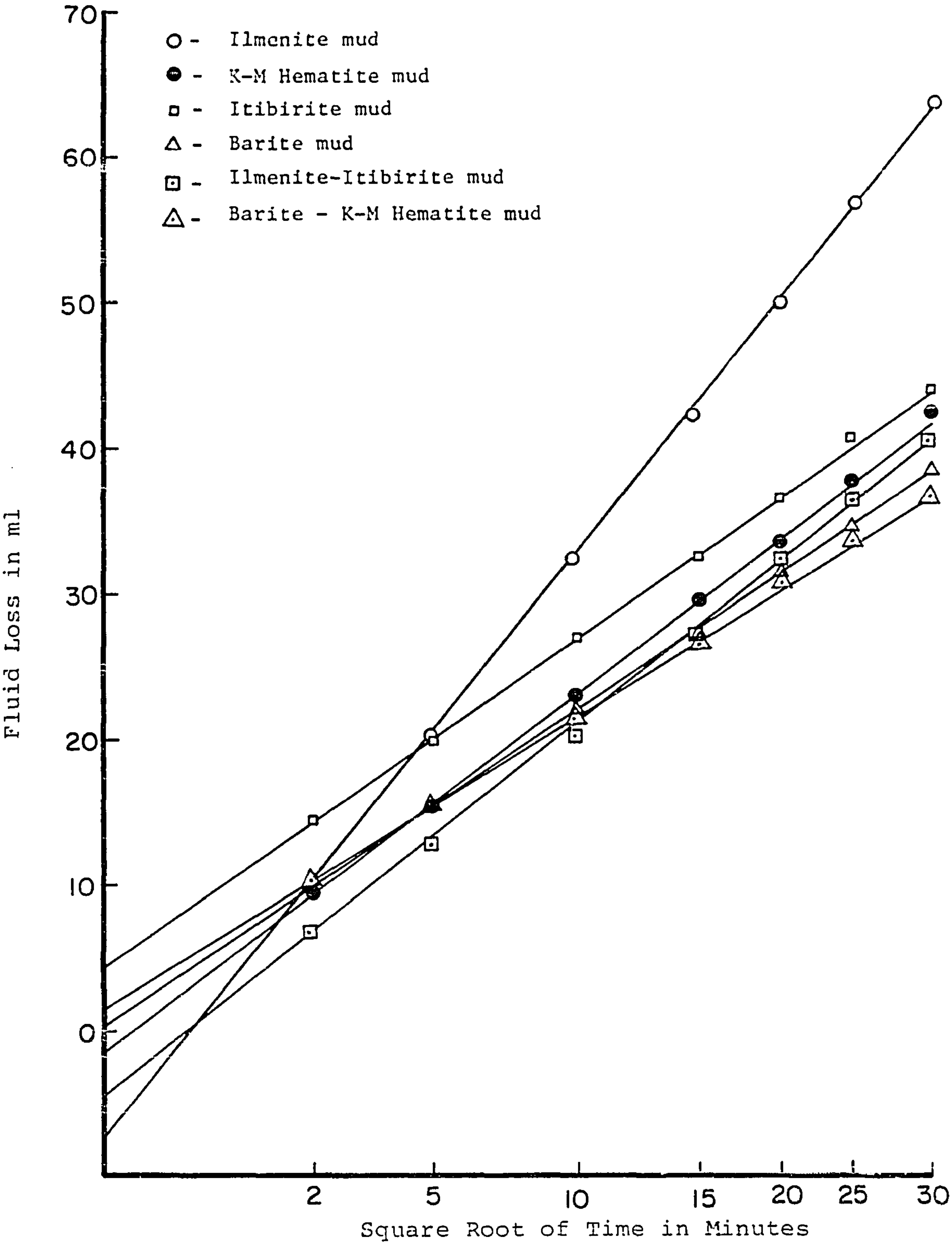


Fig. 39. High temperature and high pressure filtrate volume versus time relationships for untreated mud.

TABLE 27

HIGH TEMPERATURE AND HIGH PRESSURE FILTRATION TESTS

Mud condition = Mixed with shale cutting, aged by rolling for 7 days
Mud types = A, B, C, D, AC, and BD
Mud weight = 14 ppg
Temperature = 200°F
Differential pressure (p) = 500 psi

Time Minutes	Weighting Material					
	(A) Ilmenite	(B) K-M Hem.	(C) Itibirite	(D) Barite	(AC) Ilm/Itib.	(BD) Barite/KM-Hem.
2	4.0	3.0	2.9	2.6	5.6	3.7
5	7.8	5.5	5.0	4.8	9.2	6.6
10	11.0	8.9	7.4	7.2	13.8	9.8
15	14.4	11.7	9.7	8.8	17.8	12.6
20	17.0	14.0	11.0	10.2	21.2	14.7
25	19.4	16.0	12.5	12.0	23.0	16.6
30	21.6	17.6	14.0	13.8	25.4	18.5
*5 (2p)	3.0	2.3	2.6	2.1	4.2	3.0
**corrected filtrate (ml.)	47.2	30.2	30.7	29.5	43.3	40.2
Mud thickness (cc)	1.20	0.48	0.53	0.35	0.85	0.42
cake condition						
pH	8.19	8.70		8.13	7.92	8.33

*Fluid loss collected at double pressure in 5 minutes.
**Corrected filterate volume = 2 (filtrate volume) + correction obtained from the filtrate volume versus time.

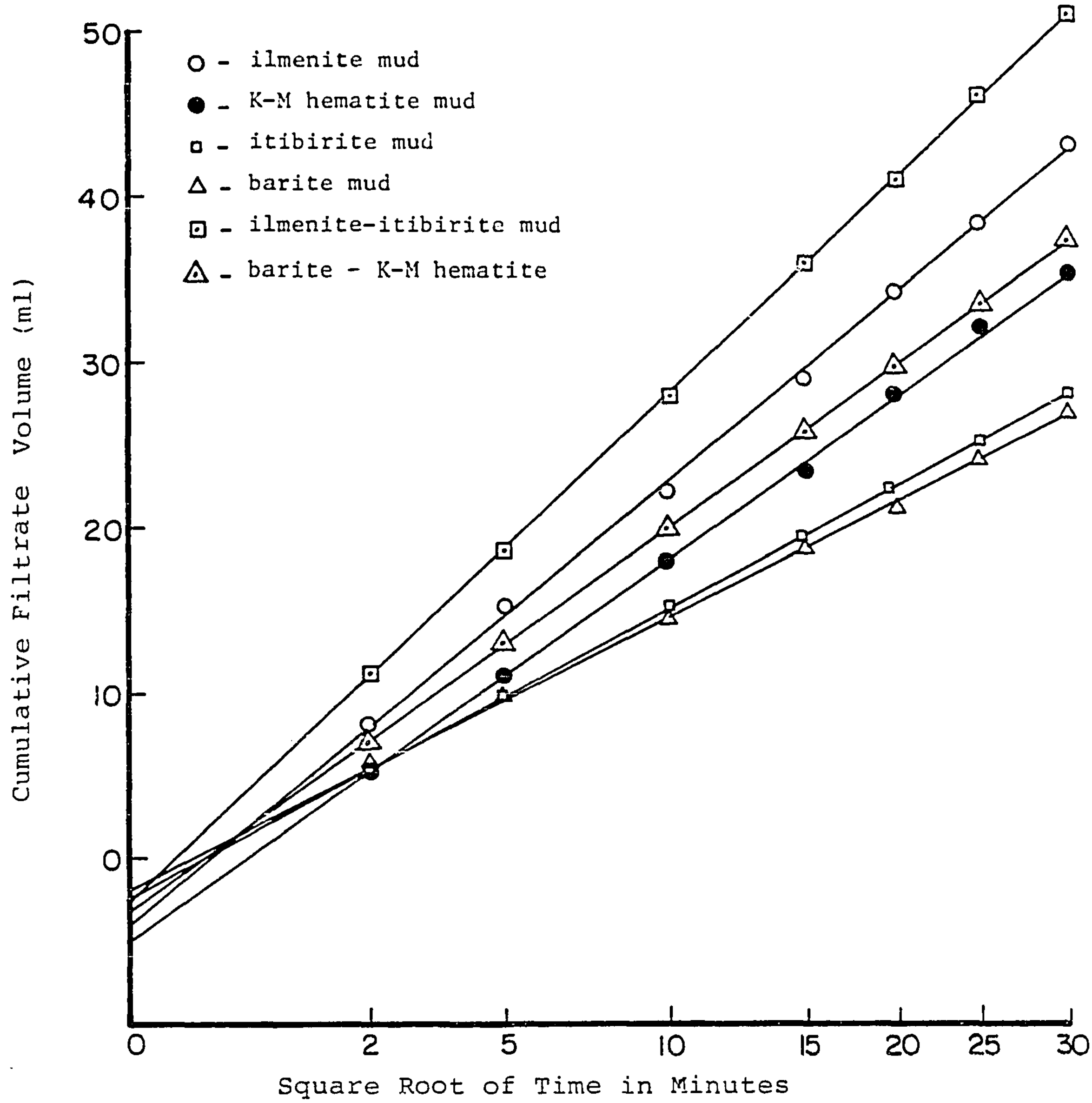


Fig. 40. Effect of mud aging with shale cuttings on the HT and HP filtration properties.

TABLE 28

HIGH TEMPERATURE AND PRESSURE FILTRATION TESTS

Mud condition = Treated mud, i.e., polymer added + thinner
Mud types = F, G, FH, GI (improved mud types)
Mud weight = 14 ppg
Temperature = 200°F
Differential pressure = 500 psi

	Weighting Material					
Time Minutes	(F) Ilmenite	(G) K-M Hem.	(H) Itibirite	(I) Barite	(FH) Ilm/Itib.	(GI) Barite/KM-Hem.
2	1.8	2.2	1.2	1.7	1.4	1.8
5	2.9	3.3	2.0	3.0	2.2	3.1
10	4.4	4.8	3.2	4.2	3.5	4.4
15	5.4	5.9	4.1	5.1	4.4	5.3
20	6.4	6.8	5.0	6.0	5.2	6.2
25	7.1	7.5	5.7	6.6	6.0	6.9
30	7.8	8.2	6.6	7.4	6.8	7.6
5 (2p)	1.2	1.4	1.0	1.0	1.0	1.4
corrected filterate (ml.)	15.1	15.9	15.7	15.8	15.1	15.7
Mud thickness	0.35	0.32	0.30	0.27	0.32	0.30
cake condition	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
pH	6.9	7.13	7.42	6.72	7.20	6.95

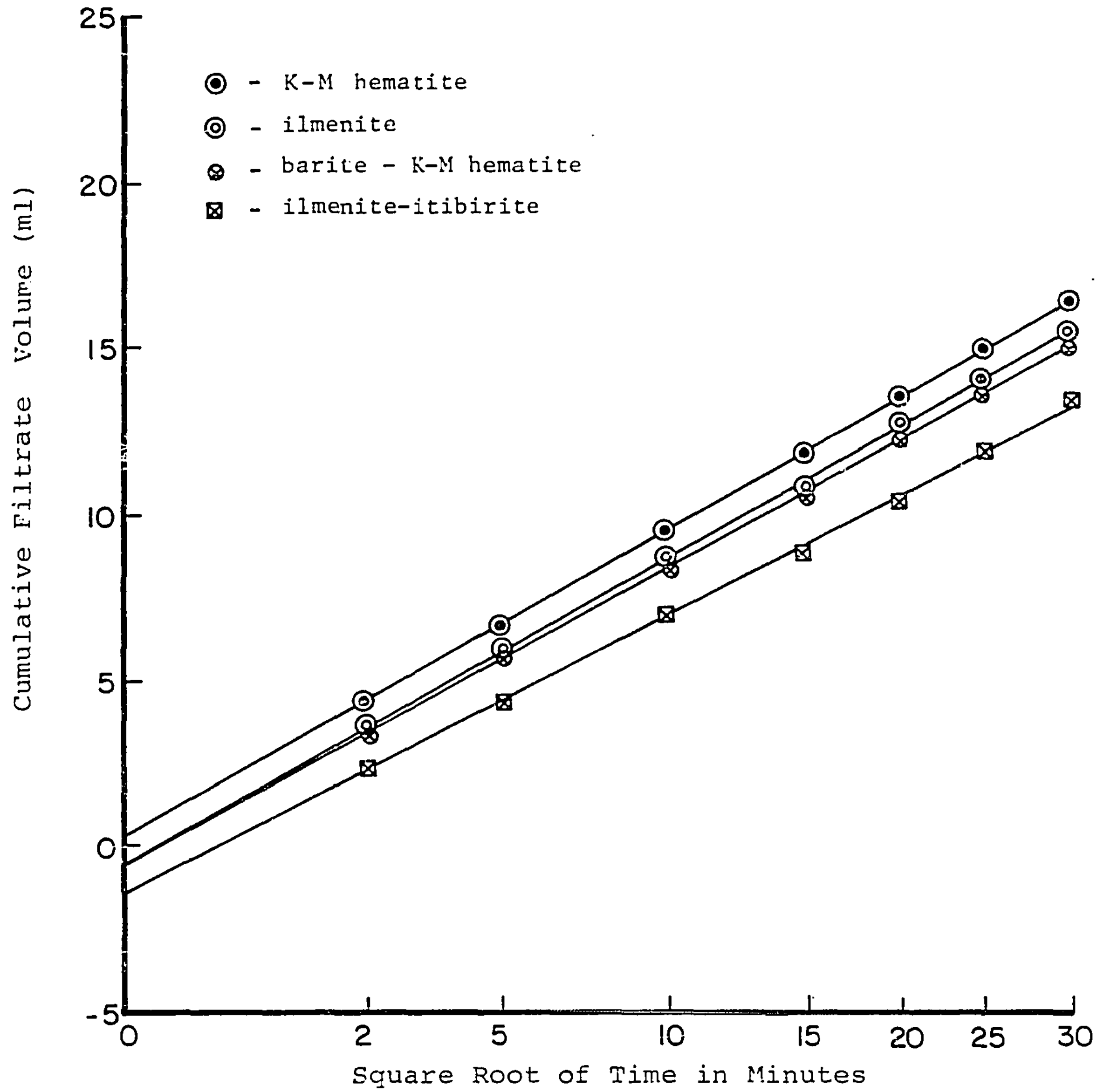


Fig. 41. Effect of grain coating on HT and HP filtration properties of mud.

TABLE 29

CORRELATION BETWEEN THE FILTRATION PROPERTIES

Mud Condition: Fresh, aged and treated muds
Mud Types: All types tested
Mud Weight: 14 ppg
Temperature: 80°F and 200°F
Pressure: 100 psi and 500 psi

Weighting Material	Fluid Loss in Ml.					
	Low Temp. Tests			HP & H.T.*		
	*Untreated	Aged	Treated	Untreated	Aged	Treated
Ilmenite	24.6	13.3	5.4	71.5	47.2	15.1
K-M Hematite	13.7	10.8	4.8	44.1	30.2	15.9
Itibirite	13.5	8.7	4.8	39.0	30.7	15.7
Barite	10.5	8.7	4.7	37.6	29.5	15.8
Ilm./Itibirite	16.4	14.6	4.8	45.8	43.3	15.1
Bar./K-M Hem.	12.7	13.2	4.4	35.1	40.2	15.7

*Values from Tables 39, 40 and 41 are multiplied by the factor 2.

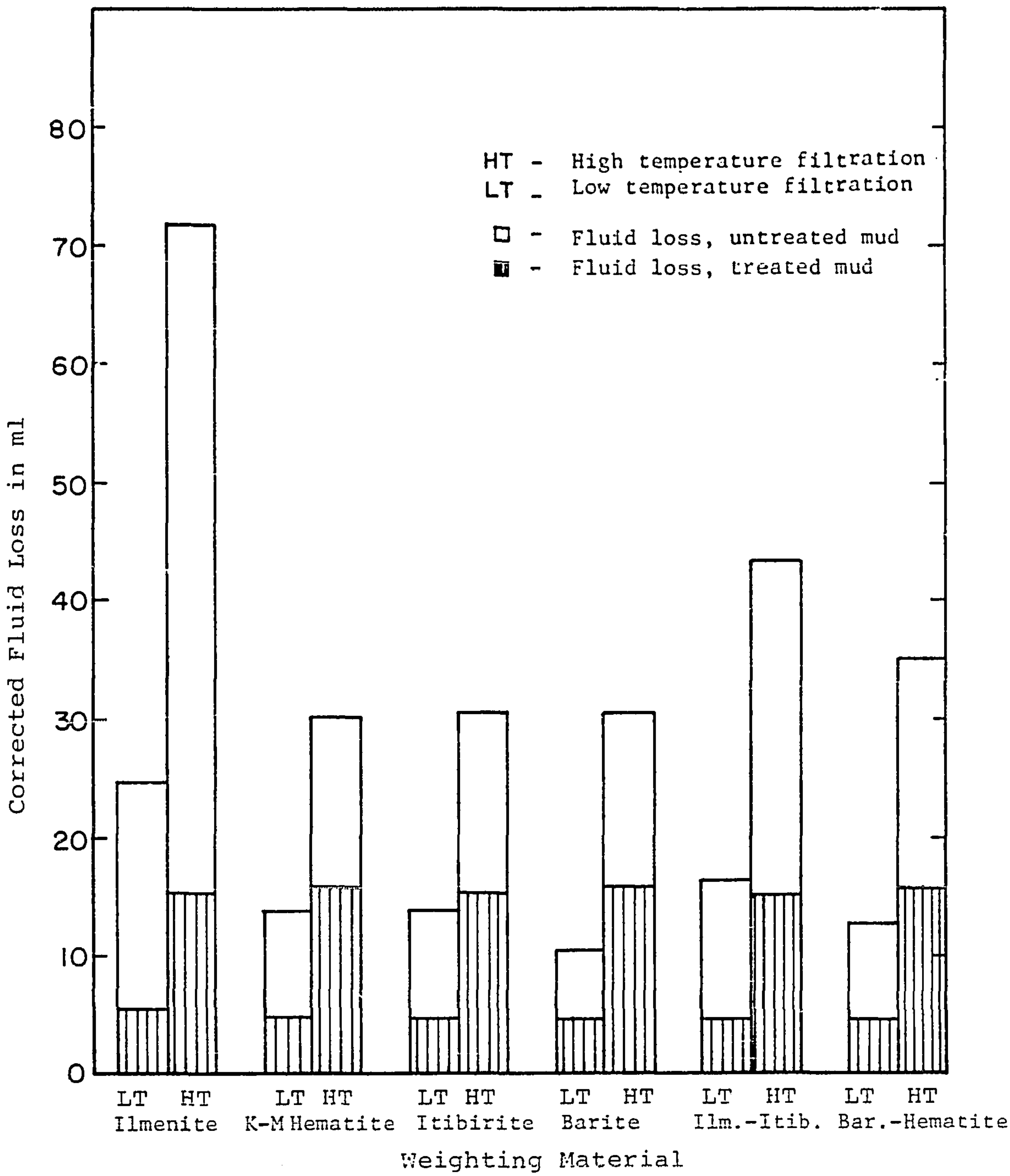


Fig. 42. Correlation between the corrected fluid losses for treated and untreated drilling fluids.

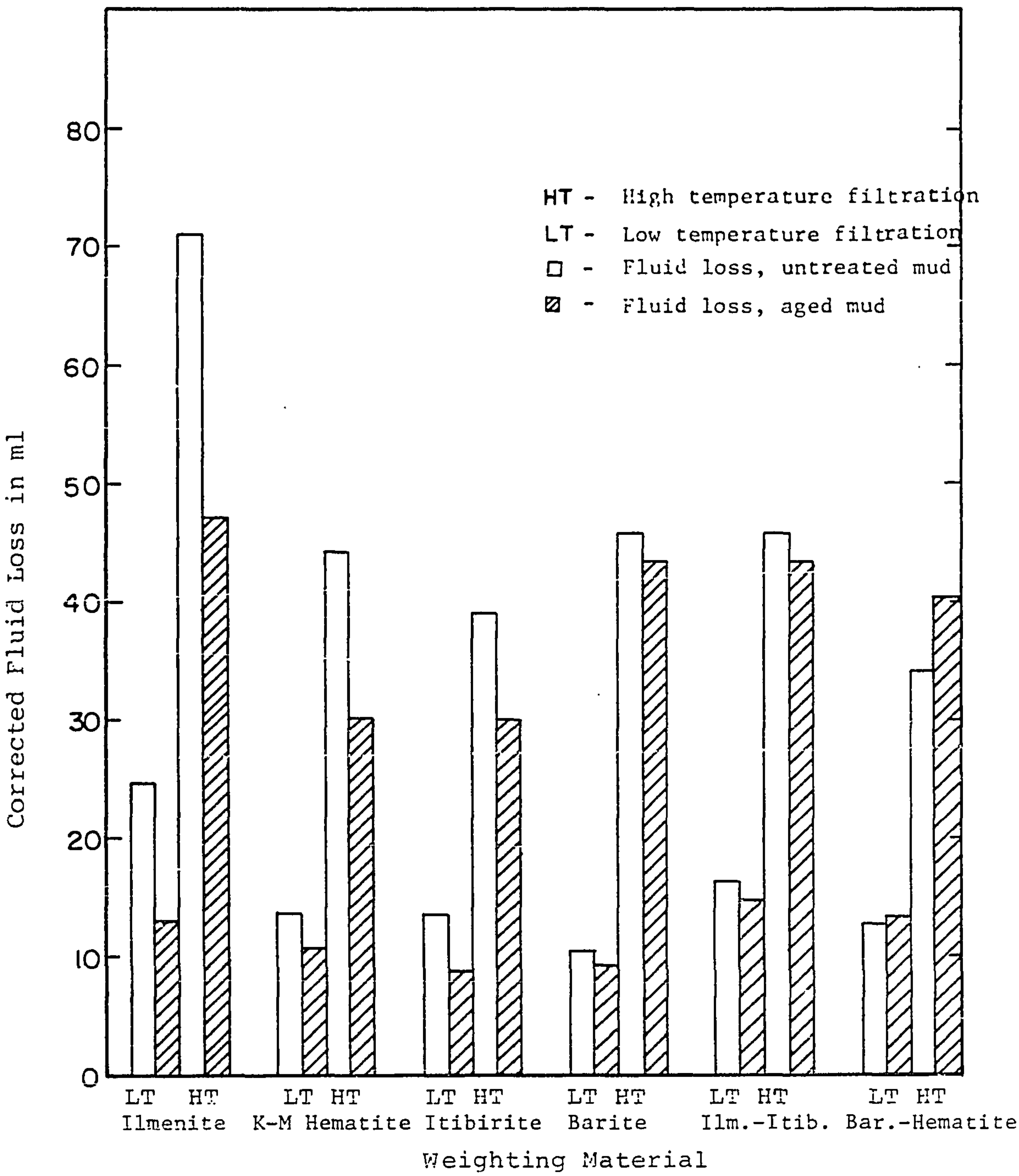


Fig. 43. The effect of aging and compatibility performance tests on the low and high temperature filtration properties.

TABLE 30

COMBINED ABRASION AND AGING TESTS BY ROLLING TECHNIQUE

Mud Type = A
Weighting Agent = Ilmenite
Mud Weight = 15 ppg
pH = 8 to 10

Rolling Time Days	Temp. (°F)	PV (cp)	YP (lb/100 ft^2)	Wt. of Coupon (gms.)	Wt. Loss (gms.)	Wt. Loss (%)	Cumulative Wt. Loss %
0	80	26.0	66.0	65.3550	-	$x10^{-4}$	$x10^{-4}$
2	120	18.5	31.0	65.3560	0.0010	15	15
4	140	21.0	36.0	65.3549	0.0011	17	32
6	160	20.5	38.0	65.3544	0.0005	8	40
8	180	26.0	40.0	65.3536	0.0008	12	52
10	200	21.0	43.0	65.3536	0.0000	0	52
12	220	23.0	49.0	65.3534	0.0002	3	55
14	240	19.5	54.0	65.3529	0.0005	8	63

TABLE 31

COMBINED ABRASION AND AGING TESTS BY ROLLING TECHNIQUE

Mud Type = B
Weighting Agent = K-M Hematite
Mud Weight = 15 ppg
pH = 8 to 10

Rolling Time Days	Temp. (°F)	PV (cp)	YP (lb/100 ft^2)	Wt. of Coupon (gms.)	Wt. Loss (gms.)	Wt. Loss (%)	Cumulative Wt. Loss %
						$x10^{-4}$	
0	80	43.0	27.0	64.9846			
2	120	43.5	21.0	64.9845	0.0001	1.5	1.5
4	140	47.0	23.5	64.9845	0.0000	0	1.5
6	160	54.0	40.0	64.9844	0.0001	1.5	3.0
8	180	50.5	43.0	64.9836	0.0008	12	15.0
10	200	66.5	32.0	64.0829	0.0007	11	26.0
12	220	68.0	36.0	64.9823	0.0006	9	35.0
14	240	69.5	65.0	64.9818	0.0005	8	43.0

TABLE 32

COMBINED ABRASION AND AGING TESTS BY ROLLING TECHNIQUE

Mud Type = C
Weighting Agent = Itibirite
Mud Weight = 15 ppg
pH = 8 to 10

Rolling Time Days	Temp. (°F)	PV (cp)	YP (lb/100 ft^2)	Wt. of Coupon (gms.)	Wt. Loss (gms.)	Wt. Loss (%)	Cumulative Wt. Loss %
0	80	52.0	11.0	65.1498		$x10^{-4}$	
2	120	52.0	26.0	65.1486	0.0012	18	13
4	140	55.5	26.0	65.1482	0.0004	6	24
6	160	58.5	33.0	65.1482	0.0000	0	24
8	180	63.0	46.0	65.1474	0.0008	12	36
10	200	67.0	56.0	65.1474	0.0000	0	36
12	220	94.5	74.0	65.1470	0.0004	6	42
14	240	93.0	79.0	65.1467	0.0003	5	47

TABLE 33

COMBINED ABRASION AND AGING TESTS BY ROLLING TECHNIQUE

Mud Type = D
Weighting Agent = Barite
Mud Weight = 15 ppg
pH = 8 to 10

Rolling Time Days	Temp. (°F)	PV (cp)	YP (lb/100 ft^2)	Wt. of Coupon (gms.)	Wt. Loss (gms.)	Wt. Loss (%)	Cumulative Wt. Loss %
0	80	43.5	23.0	64.8520		$x10^{-4}$	$x10^{-4}$
2	120	49.5	72.0	64.8494	0.0026	40	49
4	140	58.0	54.0	64.8462	0.0032	49	89
6	160	83.5	36.5	64.8401	0.0061	94	183
8	180	91.0	52.0	64.8366	0.0035	53	236
*10	200	62.0	29.0	64.8314	0.0052	80	316
*12	220	62.5	44.0	64.8304	0.0010	15	331
*14	240	65.5	49.0	64.8253	0.0051	79	410

*Mud was treated with caustic sauda and barafos to adjust the pH value and control the increasing plastic viscosity.

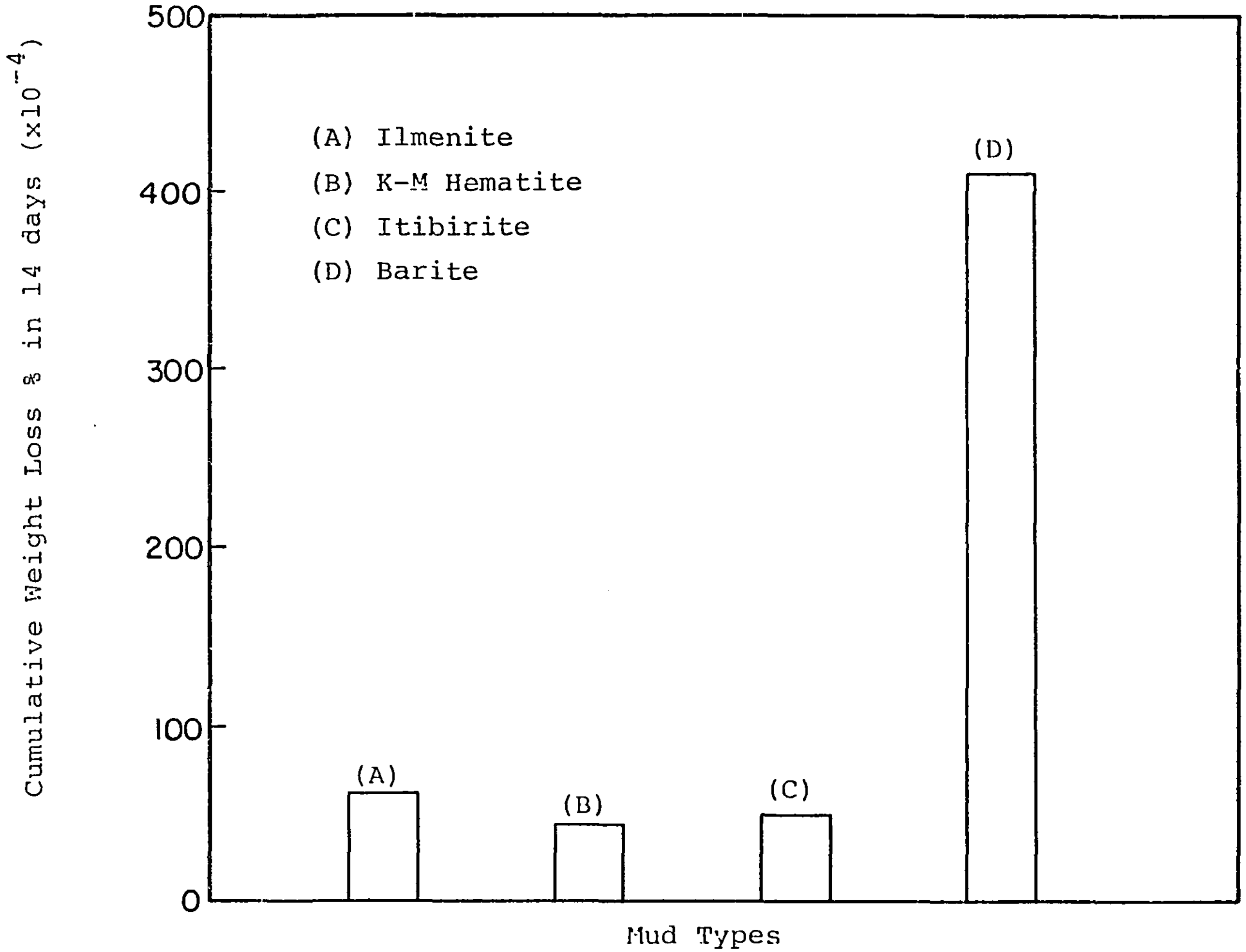


Fig. 44. Bar diagram, correlation between the abrasivity of the various mud types using rolling technique for 14 days.

TABLE 34

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm
Mud type = A (untreated)
Weighting agent = Ilmenite
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	16.0081			Waring Blender
10	15.8084	0.1997	1.247	
20	15.6705	0.3376	2.109	
30	15.5646	0.4435	2.770	
40	15.4957	0.5124	3.201	
50	15.4271	0.5810	3.629	
60	15.3611	0.6470	4.042	
70	15.3035	0.6046	4.402	
80	15.2356	0.7625	4.763	
0	7.7459			Osterizer Blender
10	7.6571	0.0888	1.147	
20	7.5832	0.1627	2.100	
30	7.5366	0.2093	2.702	
40	7.4969	0.2490	3.215	
50	7.4422	0.3037	3.922	
60	7.4036	0.3423	4.420	
70	7.3755	0.3704	4.782	
80	7.3600	0.3859	4.983	

TABLE 35

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm approximately
Mud type = B
Weighting agent = K-M Hematite
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	16.2089	0		
10	16.0163	0.1126	0.695	Waring Blender
20	16.0143	0.1946	1.201	
30	15.9515	0.2574	1.588	
40	15.8945	0.3144	1.940	
50	15.8352	0.3737	2.306	
60	15.7828	0.4261	2.629	
70	15.7371	0.4718	2.911	
80	15.6981	0.5138	3.169	
0	6.7262			Osterizer Blender
10	6.6895	0.0367	0.546	
20	6.6579	0.0683	1.016	
30	6.6220	0.1042	1.550	
40	6.5950	0.1312	1.952	
50	6.5674	0.1588	2.362	
60	6.5515	0.1747	2.598	
70	6.5276	0.1986	2.952	
80	6.5144	0.2118	3.149	

TABLE 36

Shear rate = 20,000 rpm (approximately)
Mud type = untreated C mud
Weighting agent = Itibirite
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	15.9076	0		Waring Blender
10	15.6884	0.2192	1.380	
20	15.5471	0.3605	2.266	
30	15.4453	0.4623	2.906	
40	15.3660	0.5416	3.404	
50	15.3038	0.6038	3.780	
60	15.2324	0.6752	4.244	
70	15.1661	0.7415	4.661	
80	15.1090	0.7986	5.025	
0	7.7450			Osterizer Blender
10	7.6526	0.0933	1.205	
20	7.5715	0.1744	2.252	
30	7.5131	0.2328	3.005	
40	7.4684	0.2775	3.582	
50	7.3653	0.3806	4.913	
60	7.3479	0.3980	5.138	
70	7.3120	0.4339	5.602	
80	7.2748	0.4711	6.082	

TABLE 37

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm
Mud type = D
Weighting agent = Barite
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	16.3857	0	0	Waring Blender
10	16.3428	0.0429	0.262	
20	16.3000	0.0857	0.523	
30	16.2668	0.1189	0.726	
40	16.2362	0.1495	0.912	
50	16.2080	0.1777	0.084	
60	16.1823	0.2034	1.241	
70	16.1569	0.2288	1.396	
80	16.1368	0.2489	1.519	
90	16.1170	0.2687	1.640	
100	16.1057	0.2800	1.709	
0	6.5084			Osterizer Blender
10	6.4853	0.0231	0.402	
20	6.4672	0.0412	0.700	
30	6.4531	0.0553	0.841	
40	6.4403	0.0681	1.042	
50	6.4312	0.0772	1.182	
60	6.4199	0.0885	1.360	
70	6.4168	0.0916	1.393	
80	6.4132	0.0952	1.463	

TABLE 38

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm (approximately)
Mud type - 3% Bentonite
Weighting agent = Base mud
Mud weight = 8.5 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	15.1756	0		
10	15.1726	0.0030	0.020	Waring Blender
20	15.1680	0.0076	0.050	
30	15.1649	0.0107	0.071	
40	15.1627	0.0131	0.086	
50	15.1594	0.0162	0.107	
60	15.1570	0.0186	0.122	
70	15.1539	0.0217	0.143	
80	15.1511	0.0245	0.161	

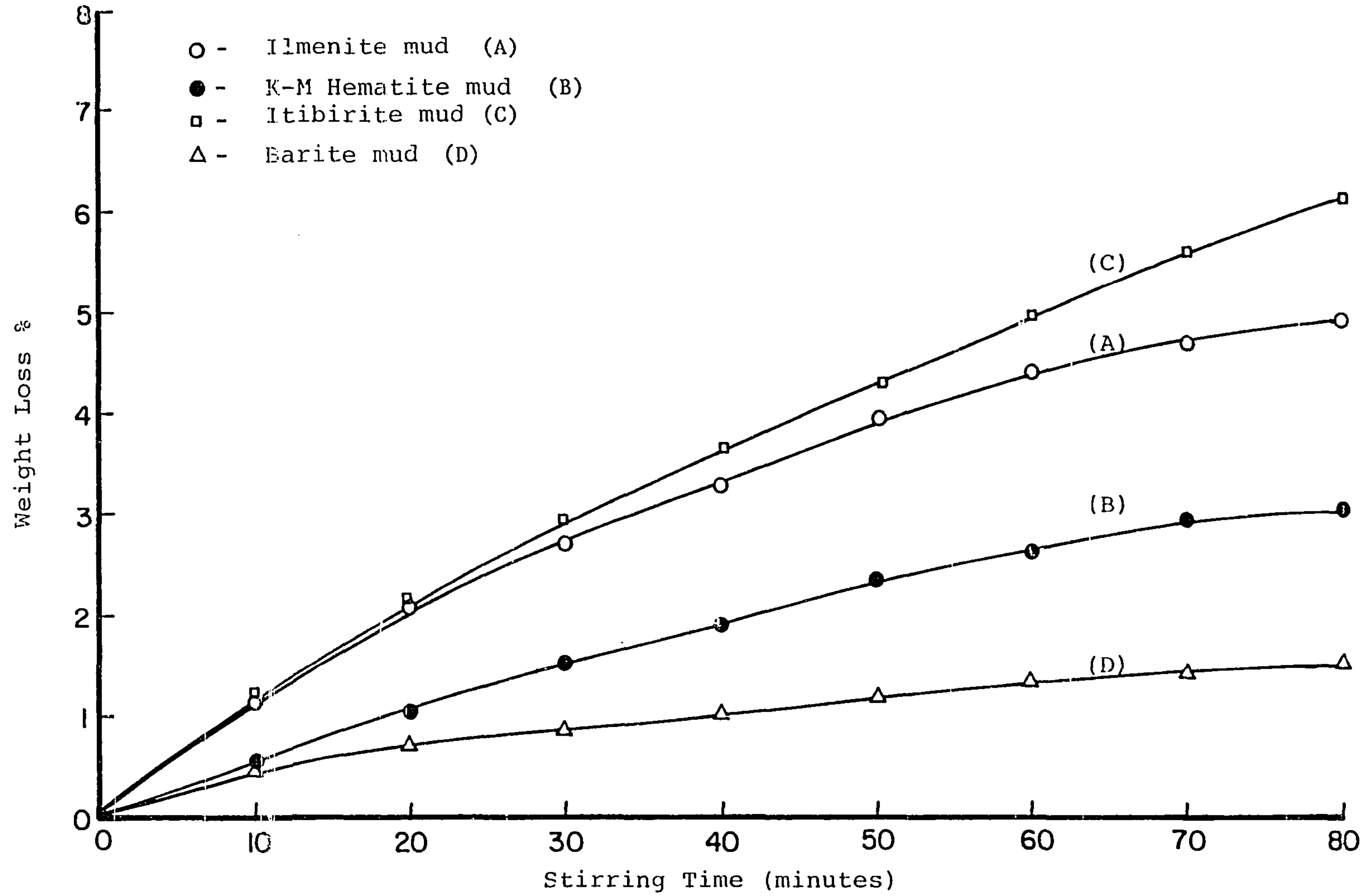


Fig. 45. Correlation between the relative abrasiveness of untreated muds.

TABLE 39

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm (approximately)
Mud type = F (Mud A + polymer + thinners)
Weighting agent = Ilmenite
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	16.0342	0		
10	15.9756	0.0586	0.365	Waring Blender
20	15.9076	0.1266	0.789	
30	15.8464	0.1878	1.171	
40	15.7915	0.2427	1.514	
50	15.7431	0.2911	1.815	
60	15.6990	0.3349	2.089	
70	15.6578	0.3764	2.347	
80	15.6163	0.4179	2.606	

TABLE 40

HIGH SHEAR ABRASION TEST

Shear rate = 20,000
Mud type = G
Weighting agent = 350 ml mud B + 5 ml Amoco H42 polymer + 3g Barafos
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	5.7429			Osterizer Blender
10	5.7264	0.0165	0.288	
20	5.7087	0.0332	0.578	
30	5.6962	0.0467	0.813	
40	5.6761	0.0658	1.145	
50	5.6669	0.0760	1.324	
60	5.6468	0.0951	1.655	
70	5.6380	0.1049	1.826	
80	5.6183	0.1236	2.150	

TABLE 41

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm
Mud type = H
Mud composition = 350 ml of mud C + 5 ml of Lo-Sol polymer + 5 gms Barafos + 2 g. Desco
Weighting agent = Itibirite
Mud Weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	6.0213			Osterizer Blender
10	5.9838	0.0375	0.623	
20	5.9438	0.0775	1.287	
30	5.9114	0.1099	1.825	
40	5.8826	0.1387	2.303	
50	5.8645	0.1568	2.605	
60	5.8382	0.1831	3.040	
70	5.8266	0.1947	3.234	
80	5.8051	0.2162	3.590	

TABLE 42

HIGH SHEAR ABRASION TEST

Shear rate = 20,000 rpm (approximately)
Mud type = I (Mud D + polymer + thinners)
Mud composition = 350 ml Barite mud + 5 ml (H42-10-4) Amoco polymer + 2g Desco + 4g Barafos
Mud weight = 15 ppg

Stirring Time (minutes)	Wt. of Blade (gm)	Wt. loss (gm)	Wt. loss (%)	Remarks
0	16.2605	0	0	Waring Blender
10	16.2330	0.0275	0.169	
20	16.2029	0.0576	0.354	
30	16.1789	0.0816	0.502	
40	16.1568	0.1037	0.638	
50	16.1357	0.1248	0.768	
60	16.1171	0.1434	0.882	
70	16.0979	0.1626	1.000	
80	16.0789	0.1816	1.117	
0	5.8051			Osterizer Blender
10	5.7969	0.0082	0.152	
20	5.7888	0.0163	0.281	
30	5.7818	0.0233	0.402	
40	5.7732	0.0319	0.549	
50	5.7677	0.0374	0.645	
60	5.7560	0.0491	0.846	
70	5.7536	0.0515	0.887	
80	5.7429	0.0622	1.071	

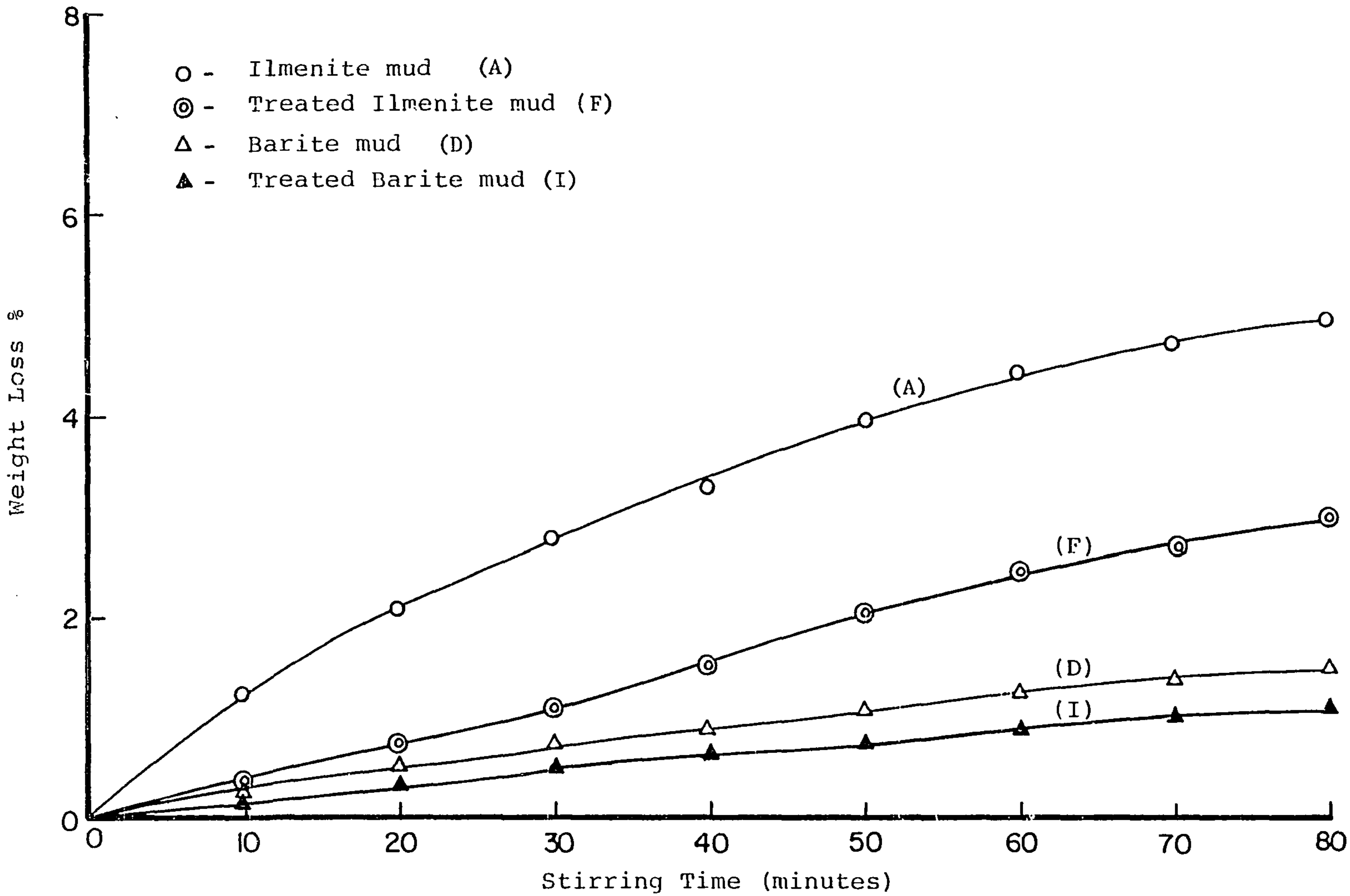


Fig. 46. Effect of mud treatment by polymer additives on the abrasivity of the ilmenite mud (Waring blender).

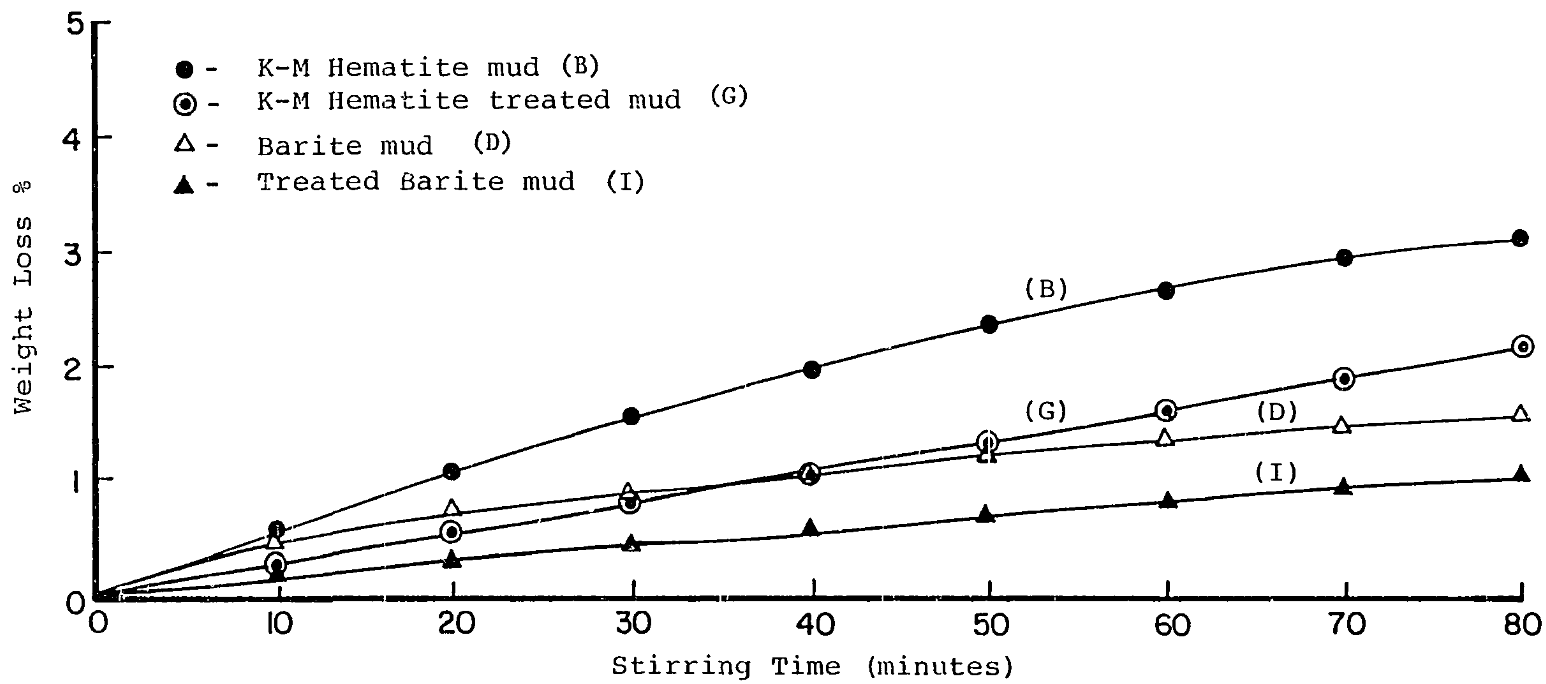


Fig. 47. The effect of mud treatment on the abrasiveness of the K-M hematite and barite weighted muds.

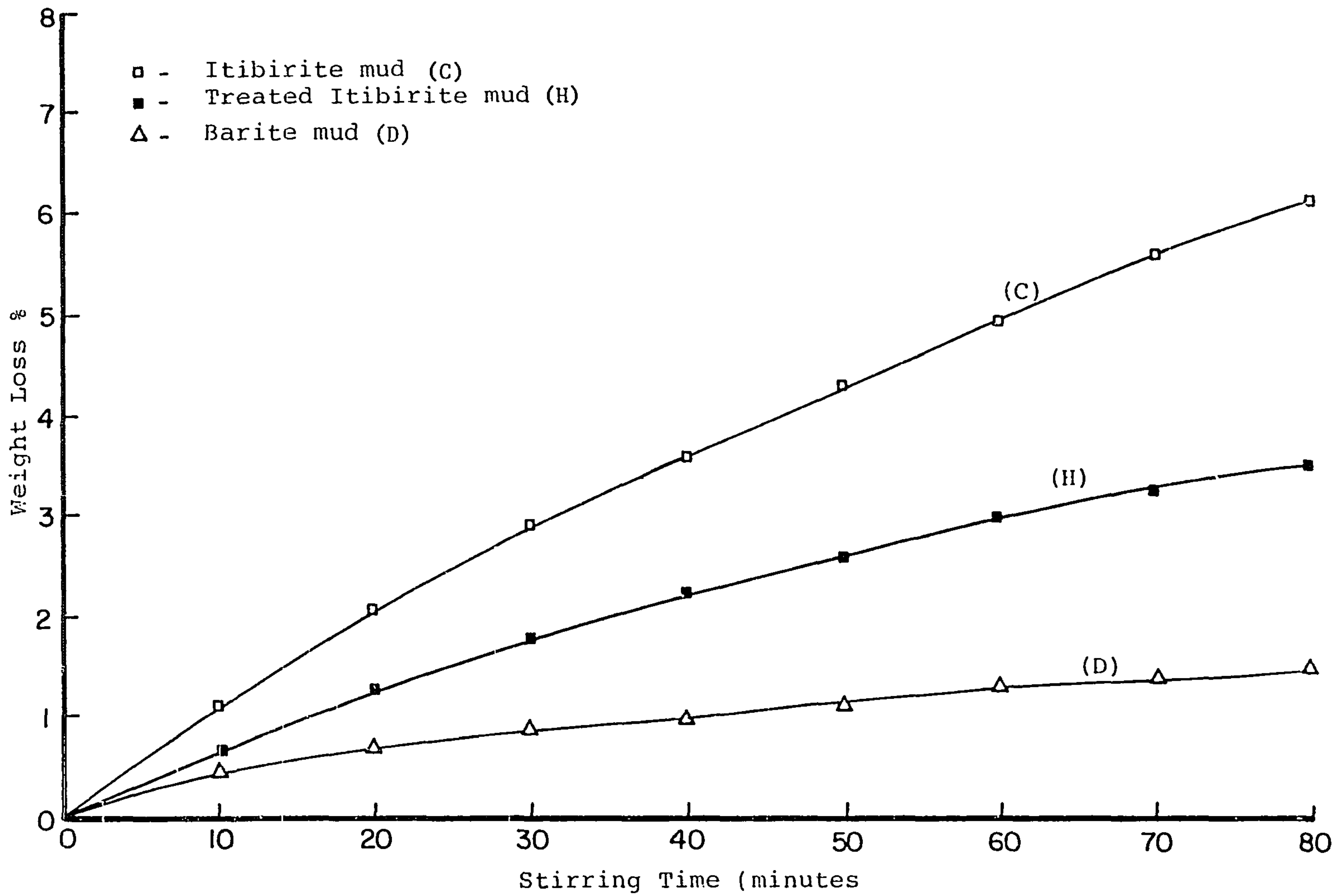


Fig. 48. The effect of mud treatment on the abrasiveness of the itibirite mud.

TABLE 43

PARTICLE ATTRITION TESTS
Sieve and Hydrometer Analysis

Mud type: A
Weighting material: Ilmenite
Temperature: 76-80°F

Time minutes	Fresh Mud		Aged & Sheared Mud	
	particle size µm	% passing	particle size µm	% passing
	74.0	96.0	74.0	96.0
2	25.1	66.8	24.8	67.3
5	16.5	49.6	16.4	52.8
15	10.1	31.2	9.9	40.1
30	7.7	20.4	7.6	28.3
60	5.3	12.6	5.2	21.5
250	2.7	9.3	2.6	17.6
1440	1.1	5.2	1.1	5.9

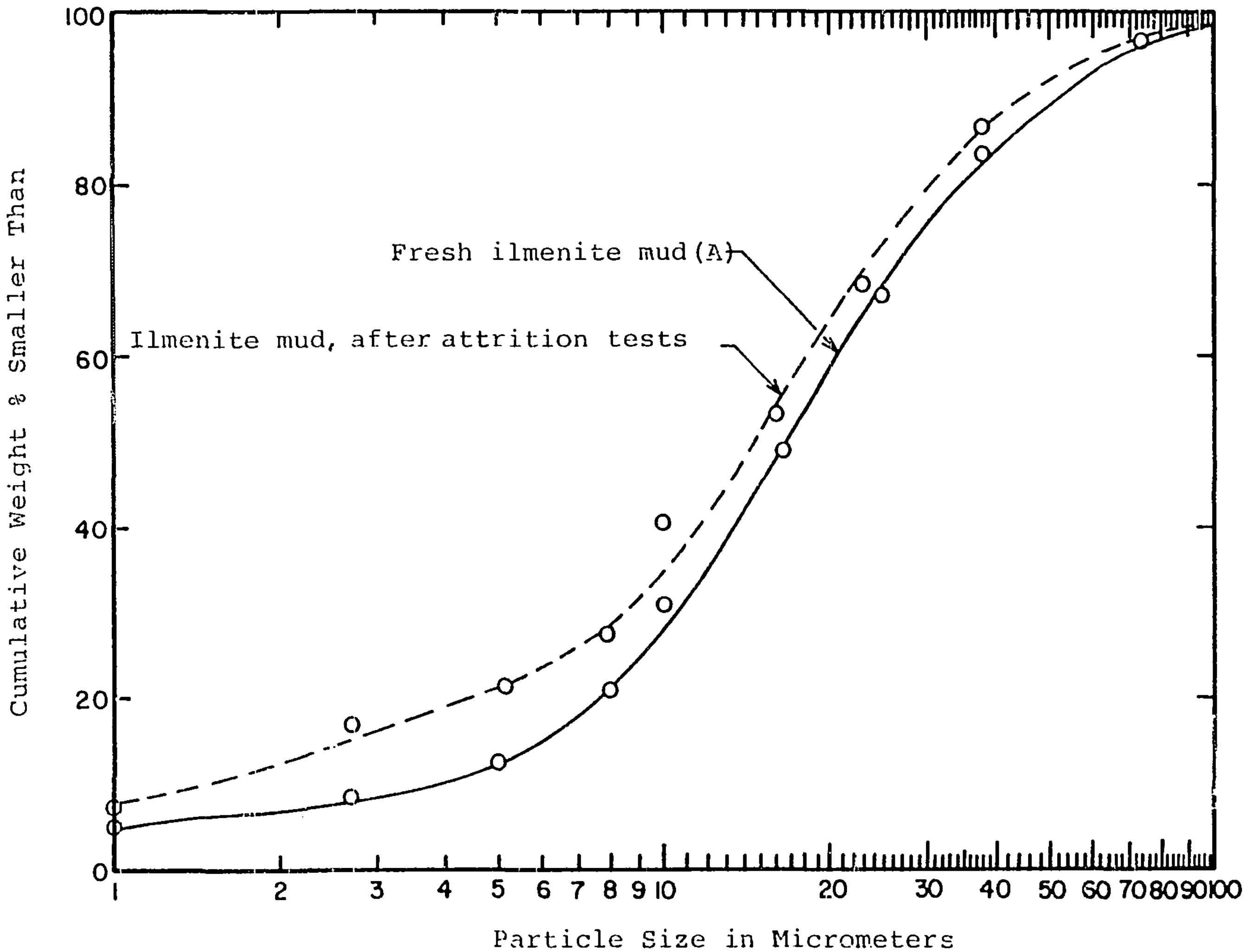


Fig. 49. Particle size distribution of the ilmenite mud (A) before and after the attrition test.

TABLE 44

PARTICLE ATTRITION TESTS
Sieve and Hydrometer Analysis

Mud type: B
Weighting material: K-M Hematite
Temperature: 76-80°F

Time minutes	Fresh Mud		Aged & Sheared Mud	
	particle size µm	% passing	particle size µm	% passing
	74.0	93.8	74.0	98.5
2	22.5	88.8	22.3	89.7
5	14.7	82.0	14.4	84.6
15	9.5	45.9	9.4	47.9
30	7.6	13.4	7.4	25.5
60	5.2	5.3	5.1	14.7
250	2.7	4.9	2.6	10.8
1440	1.1	4.2	1.1	8.2

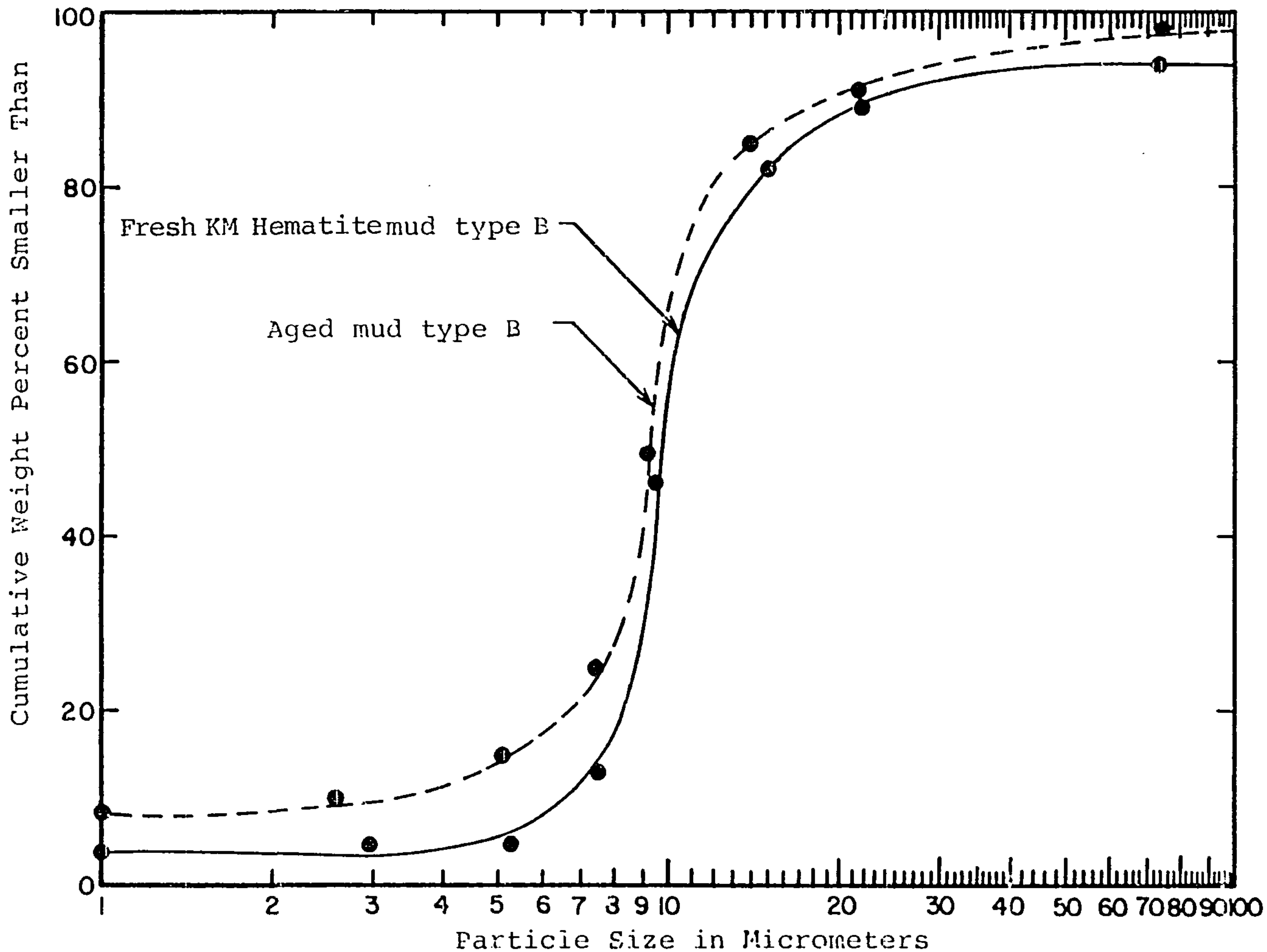


Fig. 50. Effect of shear and aging on the particle attrition of the K-M hematite mud (type B).

TABLE 45

PARTICLE ATTRITION TESTS
Sieve and Hydrometer Analysis

Mud type: C
Weighting material: Ilmenite
Temperature: 79-80°F
Mud Weight: 15 ppg

Time minutes	Fresh Mud		Aged & Sheared Mud	
	particle size μm	% passing	particle size μm	% passing
	74.0	97.7	74.0	98.1
2	25.7	46.5	24.7	73.5
5	16.5	31.3	16.7	47.0
15	10.0	21.5	10.3	25.7
30	7.5	15.2	7.6	21.3
60	5.2	11.6	5.2	17.1
250	2.5	9.2	2.6	15.9
1440	1.1	6.1	1.1	11.0

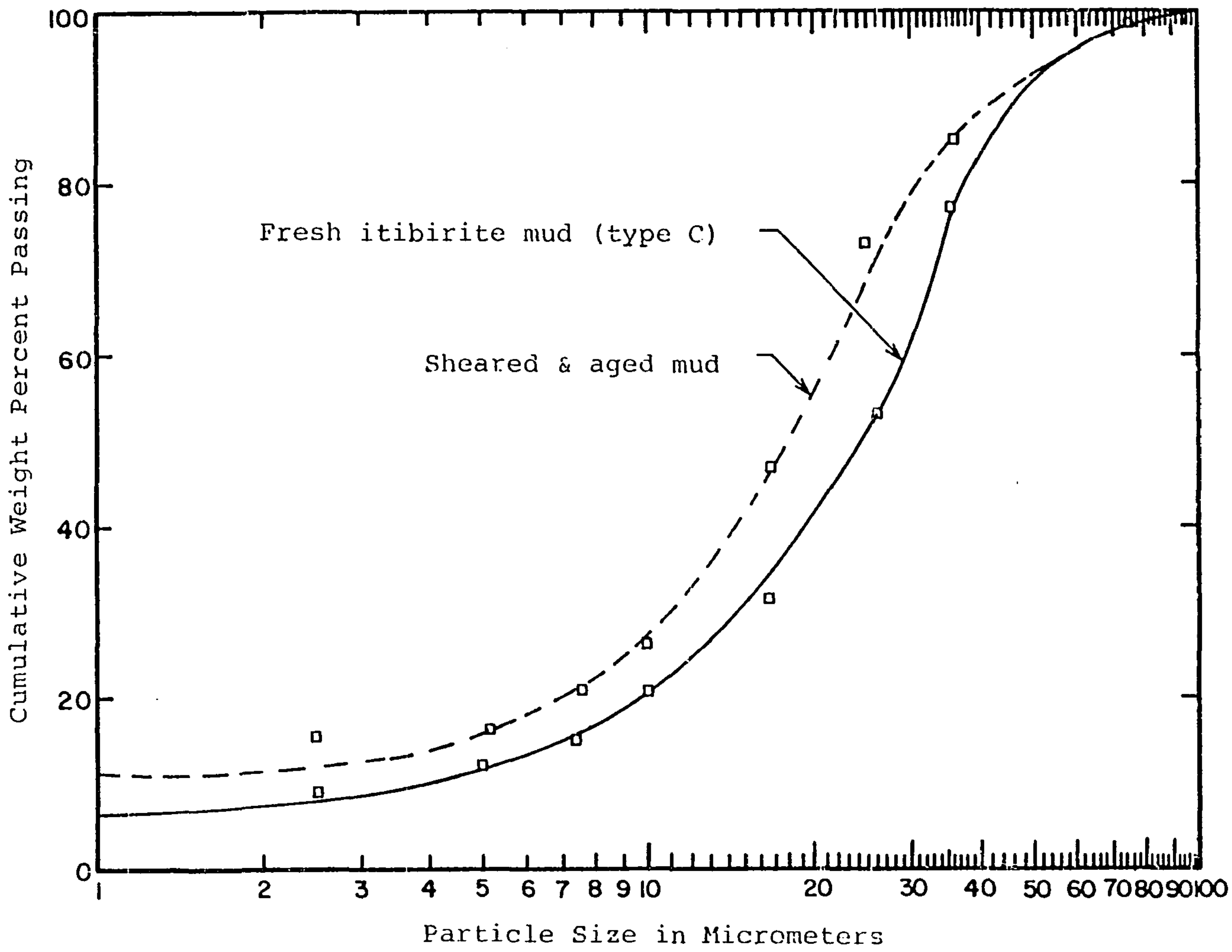


Fig. 51. Particle size distribution of the itibirite mud before and after the attrition tests.

TABLE 46

PARTICLE ATTRITION TESTS
Sieve and Hydrometer Analysis

Mud type: D & I
Weighting material: Barite
Temperature:
Mud weight: 15 ppg

Time minutes	Fresh Mud		Sheared & Aged Mud		Sheared & Aged Treated Mud (polymer added)	
	particle size μm	% passing	particle size μm	% passing	particle size μm	% passing
	74.0	94.0	74.0	98.3	74.0	98.0
	38.0	82.8	38.0	92.2	38.0	88.0
2	26.2	70.6	23.0	77.5	23.6	69.6
5	17.0	60.0	15.5	64.9	15.7	58.3
15	10.5	43.5	9.4	52.3	9.5	48.5
30	7.9	33.8	7.0	47.2	7.1	43.7
60	5.4	27.6	4.9	40.5	4.9	38.0
250	2.8	24.0	2.5	33.7	2.5	30.7
1440	1.1	10.7	1.1	19.4	1.0	21.4

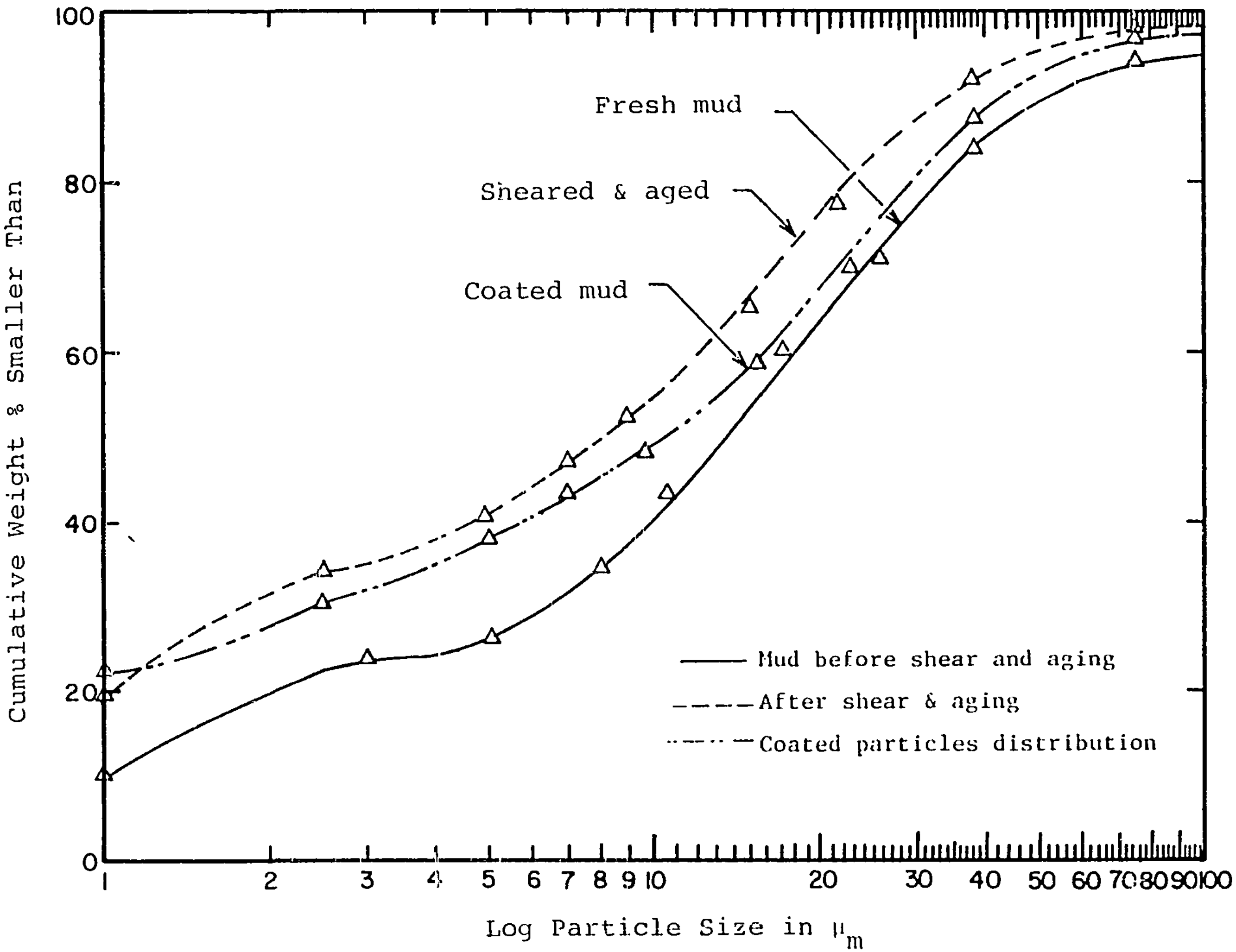


Fig. 52. Effect of shear and aging and mud treatment on the grain attrition of the barite mud.

TABLE 47

COMPATIBILITY PERFORMANCE TESTS

Mud Weight = 14 ppg
Temperature = 200°F

Weighting Material	Aging Time Days n	pH	PV (cps.)	YP ($lb/100ft^2$)	Gel Strength 10 sec	Gel Strength 10 min	Shale Loss %	MBT Shale[b] Cationic Ex Cap milli eq/100g	R[c]
Ilmenite (type A mud)	0	9.4	24	24	28	92	0	3.510	1.00
	1	7.6	19.5	23	25	45	7.1	2.925	1.031
	2	7.8-9.9[d]	33.5	13	9.5	32	1.8	2.925	0.943
	3	8.0	46.0	21.5	7	29	1.0	4.875	0.859
	4	8.1-9.2[d]	57.0	36.0	15	50	1.6	3.900	0.769
	5	8.2	59.0	19.0	4.5	7	2.4	6.825	0.785
	6	8.2	83.0	44	8	15	0.4	5.850	0.665
	7	8.2	83.0	42	9	21	1.6	4.875	0.665
							15.9[a]		
K-M Hematite (type B mud)	0	9.6	28.0	9.5	4	5	0	3.510	1.00
	1	9.2	33.0	11.5	4	4	1.0	2.925	0.966
	2	8.8	49.0	27.5	5	5	1.6	2.925	0.865
	3	8.9	45.0	20.0	4.5	5	3.0	4.875	0.889
	4	8.9	45.0	20.0	4.5	5	2.8	3.900	0.889
	5	8.8	47.0	23.0	4.5	4.5	0	3.900	0.877
	6	8.8	47.0	28.0	4.5	4.5	2.0	5.850	0.877
	7	9.1	47.5	30.0	4.5	5		5.850	0.874
							10.4[a]		

[a] Total shale loss percent

[b] The cationic exchange capacity (milliquivalent/100g.) = methylene blue capacity x 0.78.

[c] $R = \frac{(ROP)n}{(ROP)o} = 10^{0.003(\overline{PV}_o - \overline{PV}_n)}$ (ref. 18)

(ROP)o = initial penetration rate (ROP)n = final penetration rate n = 1,2,3,...7

[d] Chemicals added to adjust pH and/or viscosity

TABLE 47--continued

Mud Weight = 14 ppg
Temperature = 200°F

Weighting Material	Aging Time Days n	pH	PV (cps.)	YP (lb/100ft^2)	Gel Strength 10 sec	Gel Strength 10 min	Shale Loss %	MBT Shale[b] Cationic Ex Cap milli eq/100g	R[c]
Itibirite (type C mud)	0	8.1	65	50	6	8	0	3.510	1.000
	1	7.8-8.2[d]	70	70	9	10	1.0	3.900	0.966
	2	8.1	69	72	8.5	9.5	1.4	4.870	0.973
	3	7.8-9.8[d]	75	80	8	10.5	2.0	3.900	0.933
	4	8.5	88	74	10	11	3.0	3.900	0.853
	5	8.6	90	98	12	13	1.0	4.870	0.841
	6	8.5	83	89	10.5	22	1.8	4.870	0.883
	7	8.2-8.6[d]	71	63	7.5	9	1.8	3.900	0.959
							12.0[a]		
Barite (type D mud)	0	9.2	44	16.0	4	6	0	3.510	1.000
	1	6.6-8.2[d]	58.5	24.5	4.5	7	4.9	3.900	0.905
	2	8.2	60.0	27.5	5	8.5	2.2	5.850	0.895
	3	7.4-9.9[d]	74.0	47	6.5	14.5	0.8	6.830	0.813
	4	8.6	91.0	51	8	18	3.0	3.900	0.723
	5	8.2-8.5[d]	92.0	66	5	13	4.6	4.870	0.718
	6	8.1-8.5[d]	82.0	58	6	12	0.6	6.825	0.769
	7	8.0	87.0	73	8	18	1.6	4.875	0.743
							17.7[a]		

TABLE 47--continued

Mud Weight = 14 ppg
Temperature = 200°F

Weighting Material	Aging Time Days n	pH	PV (cps.)	YP (lb/100ft^2)	Gel Strength 10 sec	Gel Strength 10 min	Shale Loss %	MBT Shale Cationic Ex Cap milli eq/100g [b]	N[c]
Barite-KM Hematite (type BD mud)	0	9.2	29	16	4.5	21	0		
	1	8.6	34	15	4	11	1.8	3.900	0.966
	2	8.1	39	18	5	13	1.6	4.875	0.933
	3	8.6	48	30	4.5	17	2.0	3.900	0.877
	4	8.6	54.5	32	5.5	22	1.4	4.875	0.838
	5	8.7	53	34	7	25	4.0	5.850	0.847
	6	8.7	70	59	11	36	4.0	4.875	0.753
	7	8.5	66	57	11	35	1.8	4.875	0.774
							16.6[a]		
Ilmenite-Itibirite (type AC mud)	0	9.1	24.0	12.0	14	31	0		
	1	8.3	28.5	5.0	4	19	1.0	5.850	0.973
	2	8.0	31.5	8.5	5	20	3.0	4.875	0.953
	3	8.0	33.5	10.5	4	6	0.8	5.850	0.940
	4	8.1	37.5	9.0	4	17	2.0	5.850	0.914
	5	7.6-10.3[d]	43.0	17.0	5	17	1.8	5.850	0.877
	6	8.2	37.0	8.0	4	5.5	2.8	4.875	0.914
	7	8.4	40.5	12.5	6	29	2.8	4.875	0.895
							14.2[a]		

[a] Total shale loss percent

[b] The cationic exchange capacity (milliquivalent/100g.) = methylene blue capacity x 0.78.

[c] $R = \frac{(ROP)n}{(ROP)o} = 10^{0.003(\overline{PV}_o - \overline{PV}_n)}$ (ref. 18)

[d] Chemicals added to adjust pH and/or viscosity

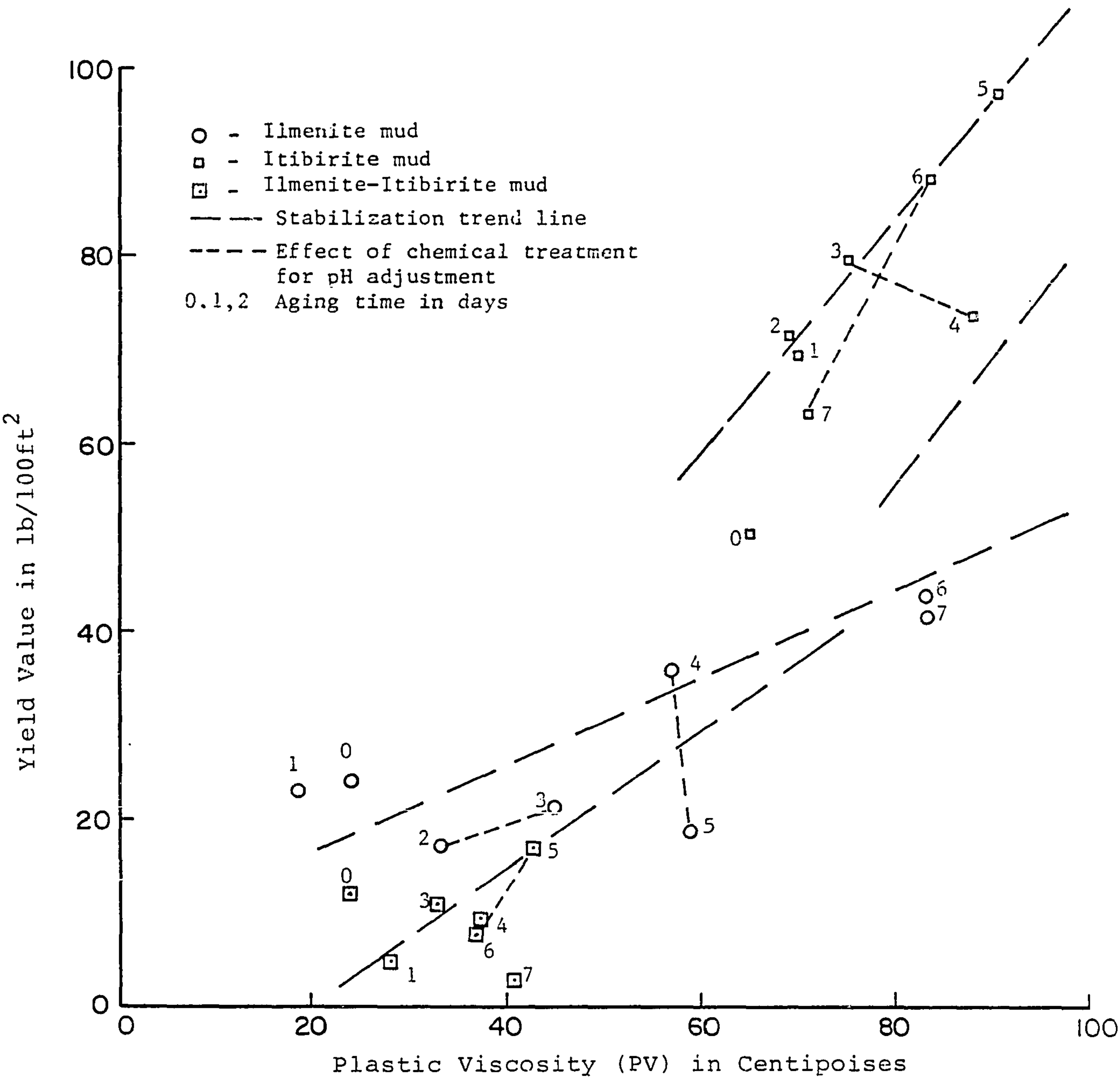


Fig. 53. The effect of thermal aging and compatibility performance tests on the stability of the ilmenite and itibirite muds.

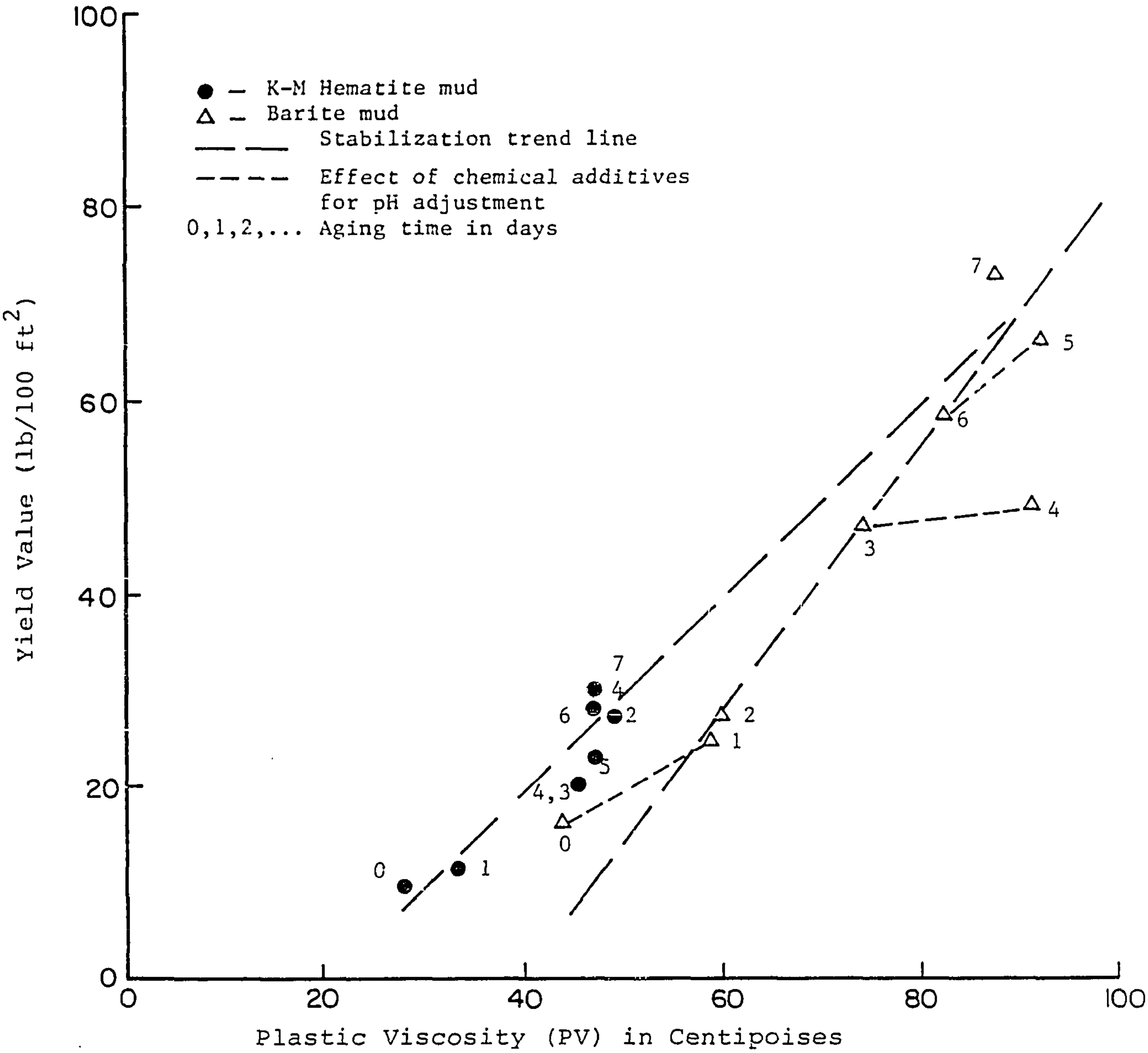


Fig. 54. The effect of thermal aging and compatibility tests on the stability of the K-M hematite and barite muds

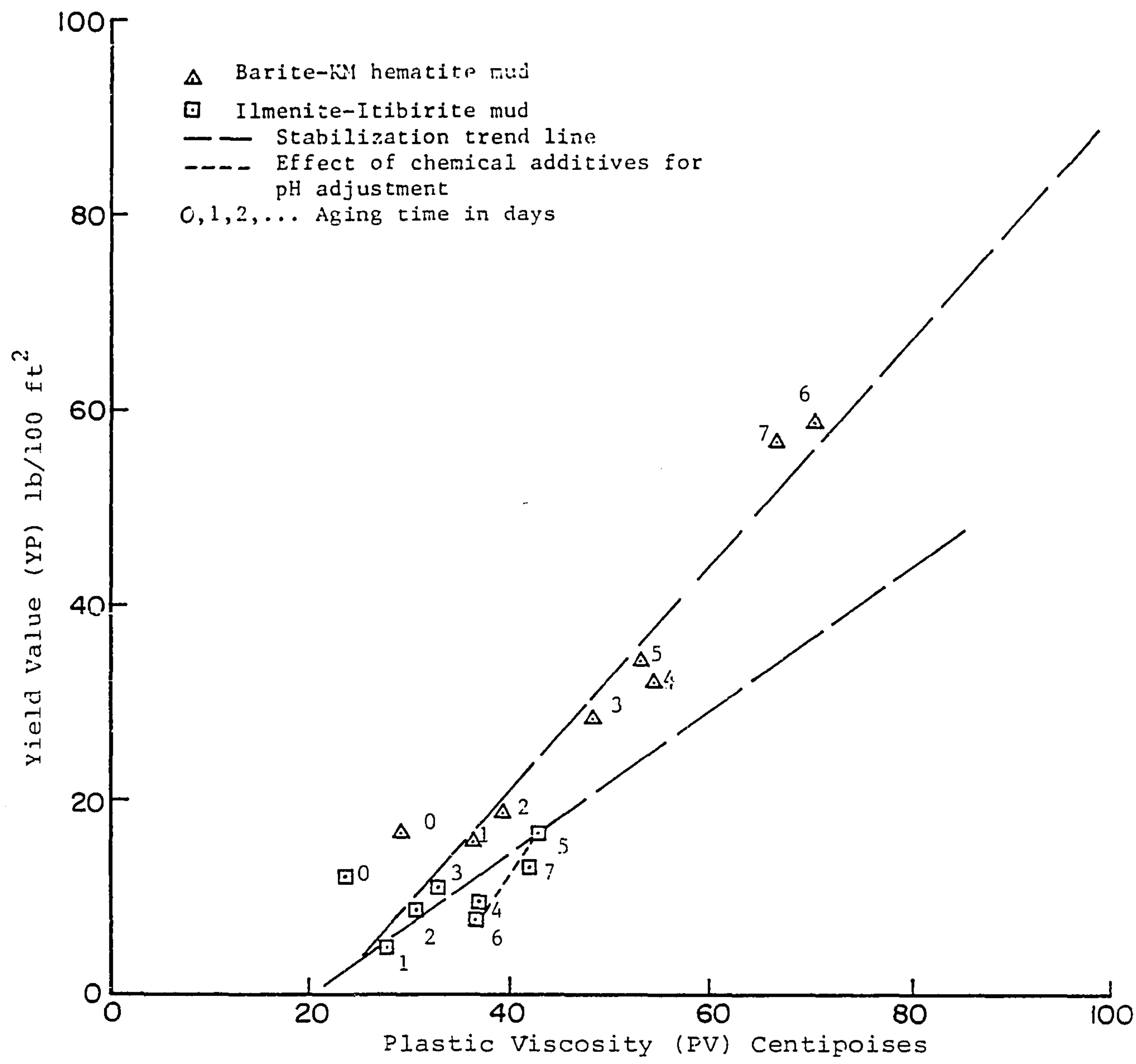


Fig. 55. The effect of thermal aging and compatibility tests on the stability of drilling fluids weighted with mixtures.

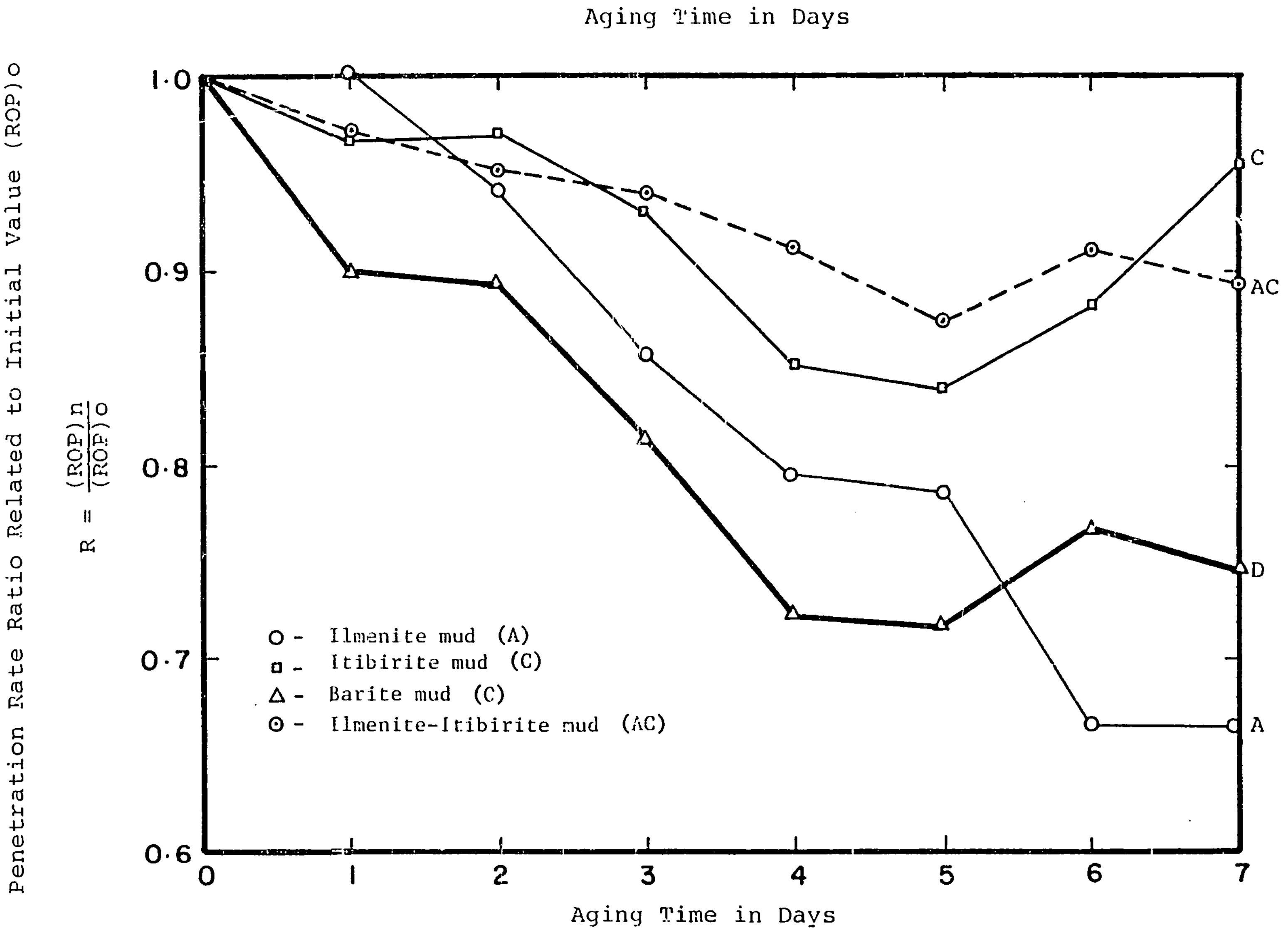


Fig. 56a. Effect of mud aging and chemical treatment on penetration rates.

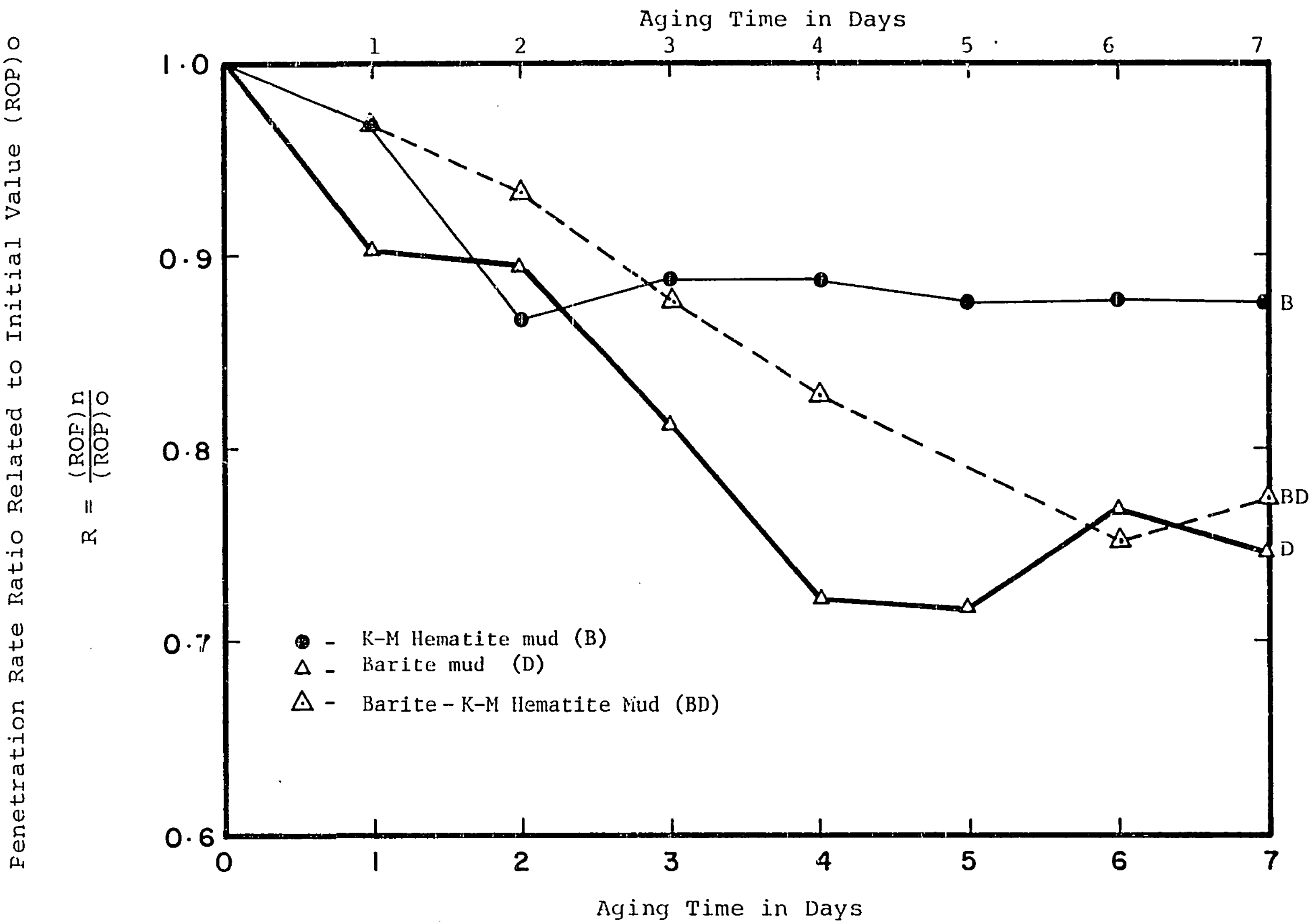


Fig. 56b. Effect of mud aging and chemical treatment on penetration rates.

(A) untreated abrasive mud
(B) mud treated with an antiwear agent

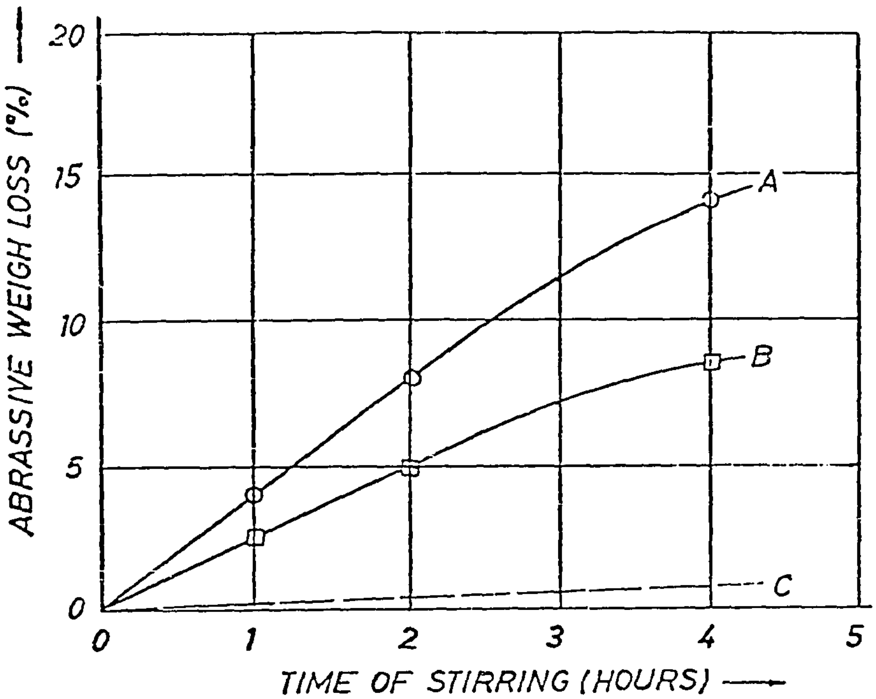


Fig. 57. Time of stirring versus the abrasive weight loss percent for the UK patent application.

(UK patent application GB 2066876A, 1981, published by the Patent Office, London)

APPENDIX C

ELECTRON MICROGRAPHS

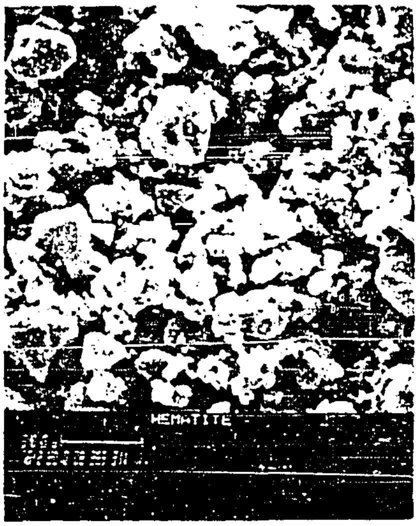
A. Barite crystals before aging

B. Barite showing the effect of attrition due to aging and high shear rate

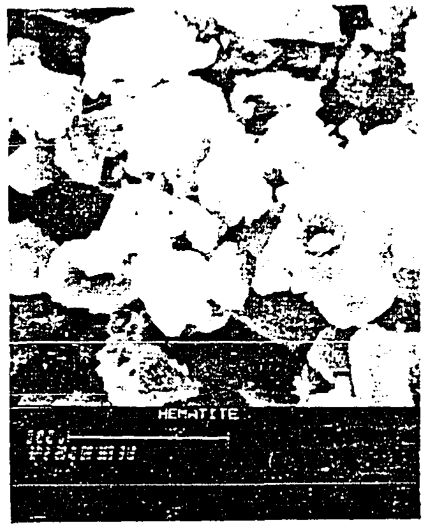
C. Effective coating of barite particle using select flocculant polymer, H42

D. Partial grain coating with Drispac

Fig. 58. Effect of aging and mud treatment on barite particles

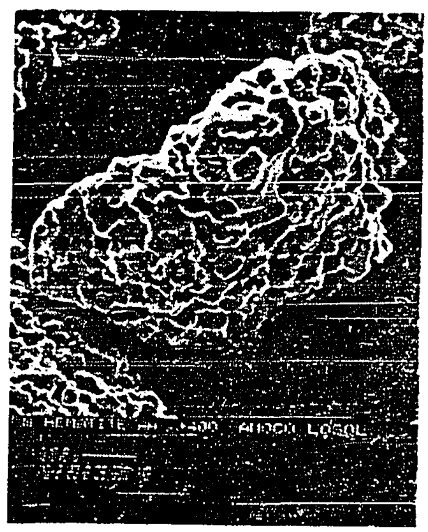


A. The K-M Hematite rounded grain shape



B. Grain surface before treatment

C. Treated K-M Hematite mud with Amoco Lo-sol

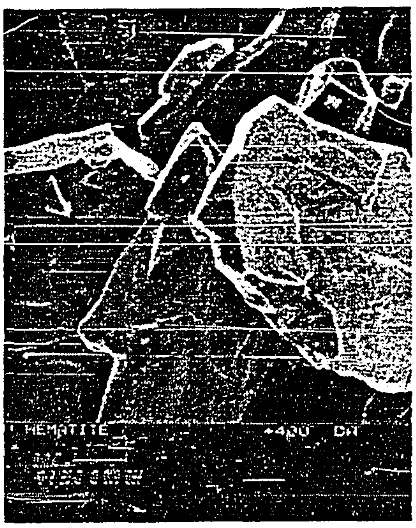


D. Coated K-M Hematite particle

Fig. 59. Grain shape and coated particles of the K-M Hematite

A. Grain shape of ilmenite (Bargain)

C. Coated ilmenite grain (select flocculant polymer, H42)



C. Grain Shape of itibirite

D. Coated itibirite grains (Lo-sol polymer)

Fig. 60. Grain shape and coated ilmenite and itibirite particles

APPENDIX D

SET-UP PHOTOGRAPHS

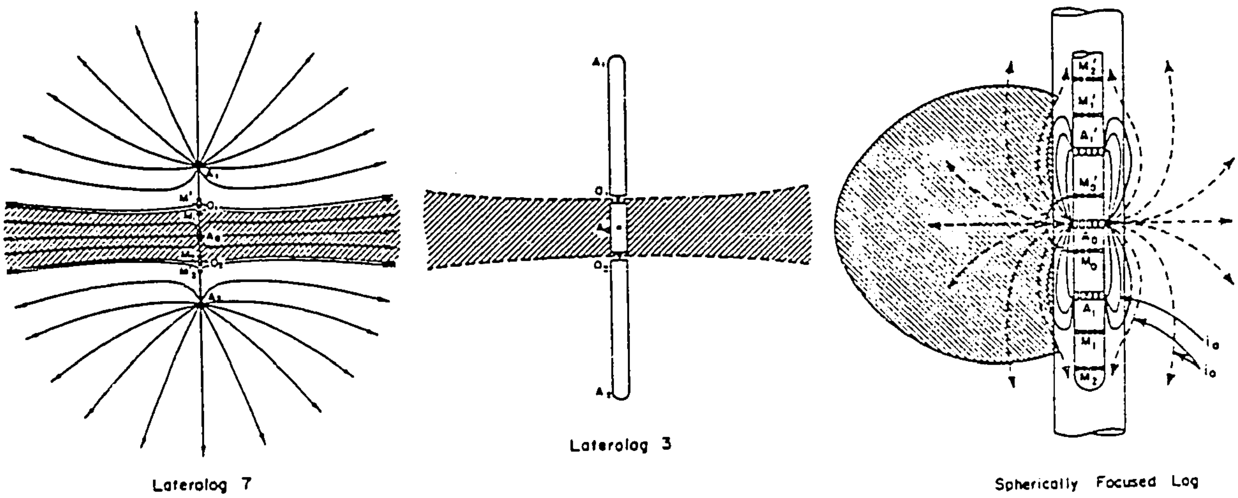


Fig. 61. The laterolog and spherically focused log tools. (Courtesy of Schlumberger, 1975)

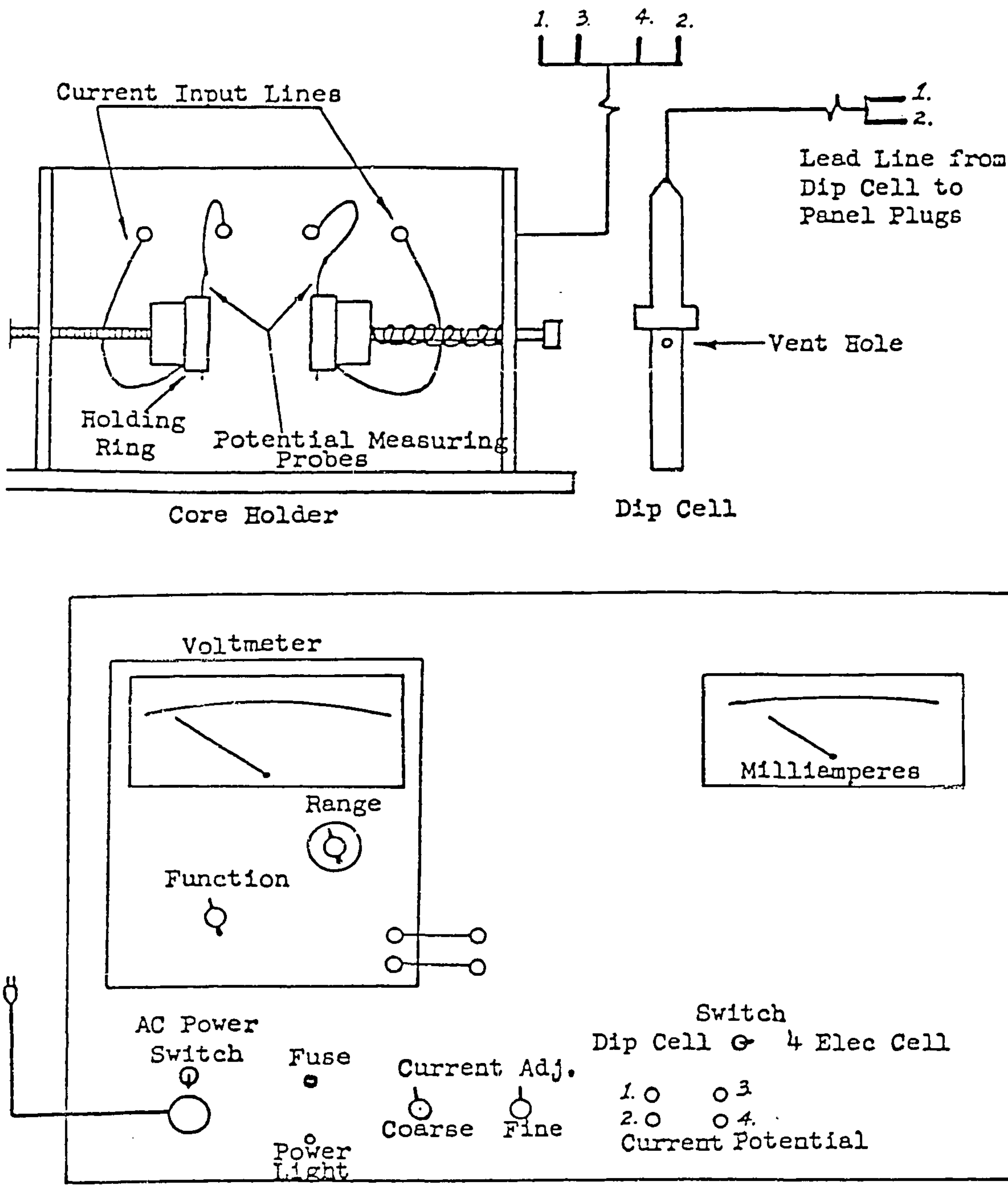
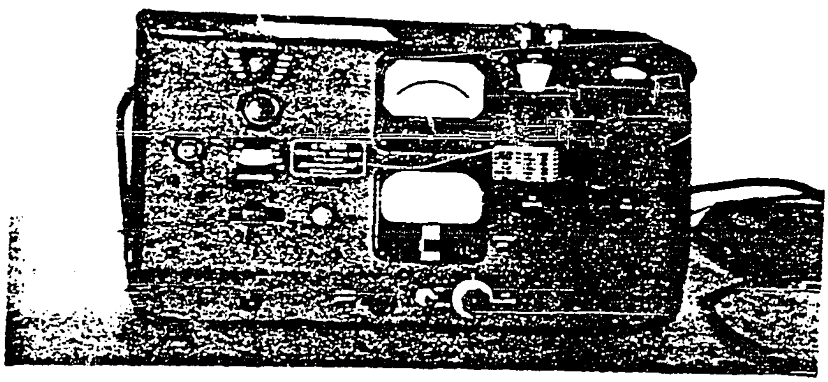


Fig. 62. The Fann Resistivity meter Model 88-A



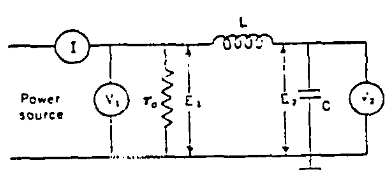


Fig. 63. The Q-meter and the magnetic permeability measurement circuit

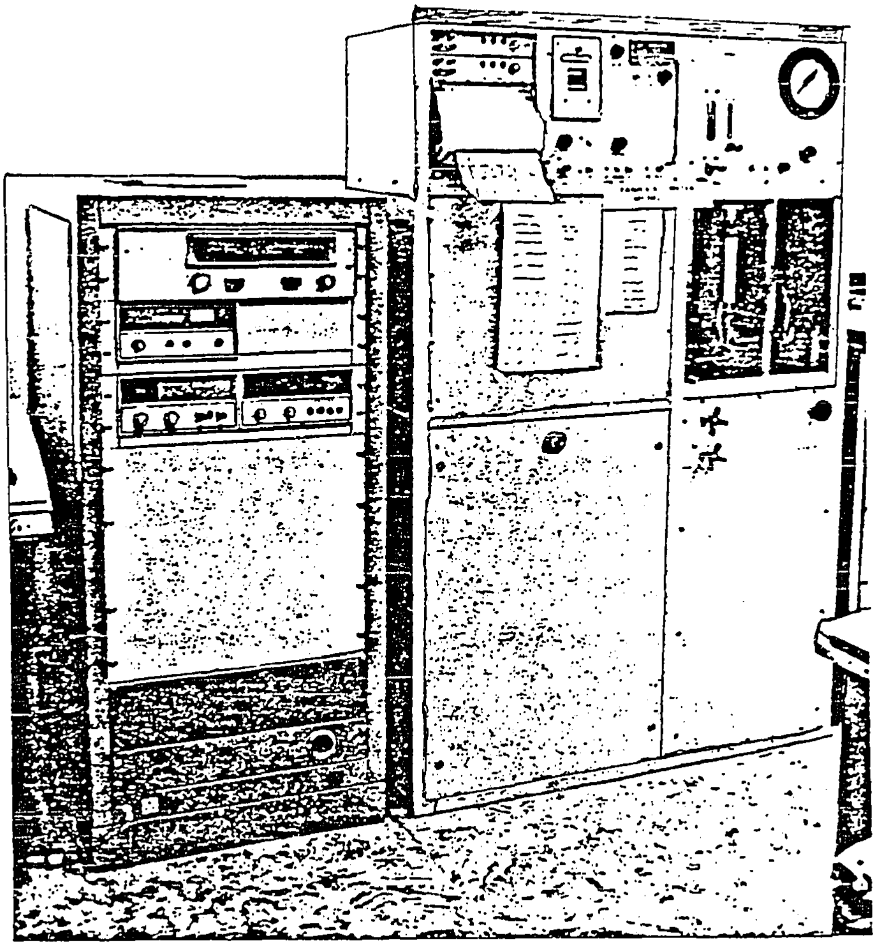


Fig. 64. Fan Model 50B High Temperature Viscometer

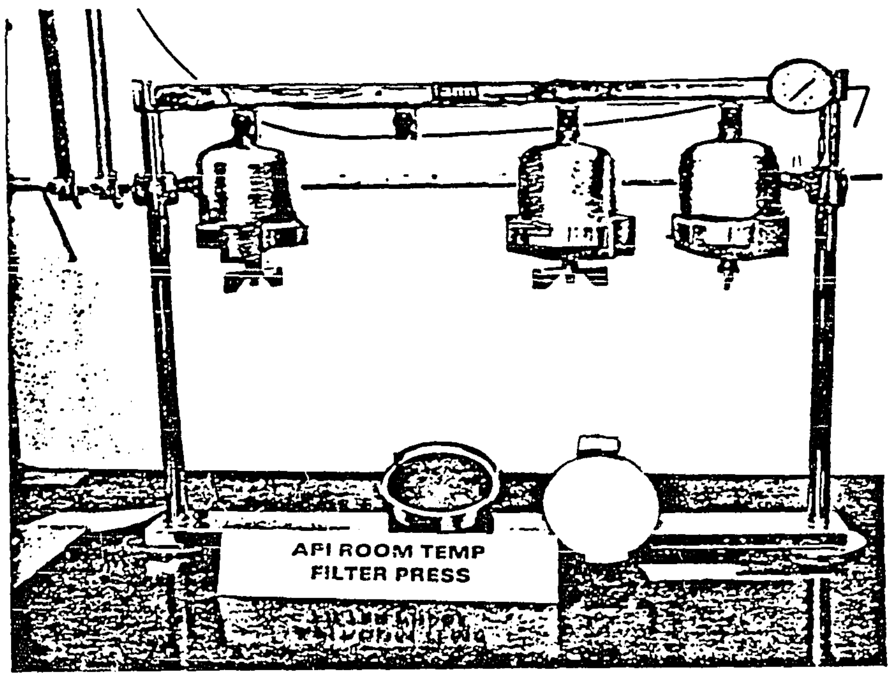


Fig. 65. Low temperature filter press (Lab. Model-Fann)

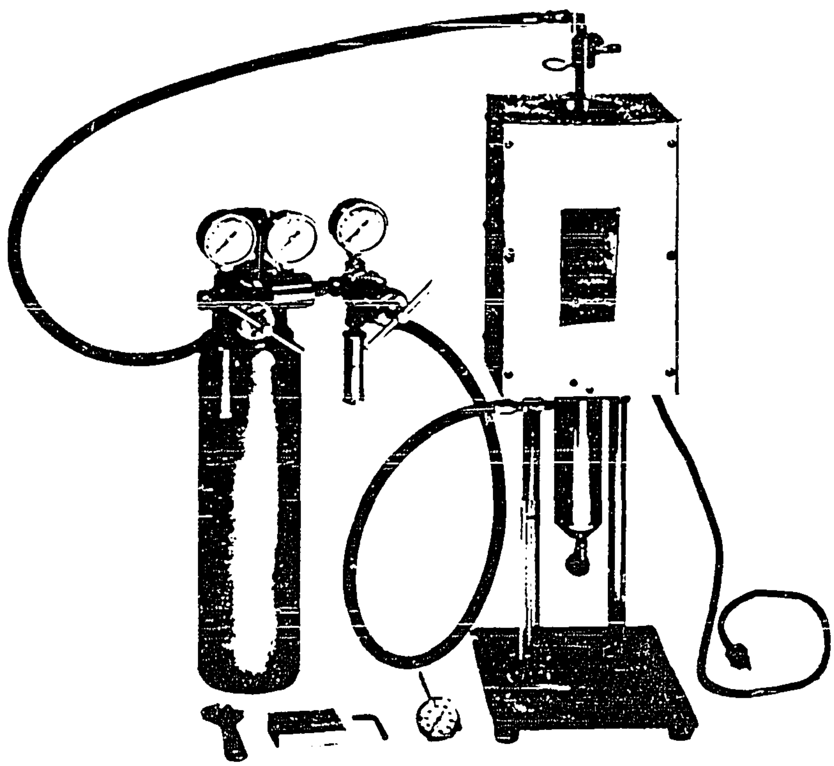


Fig. 66. HT-HP filter press (Baroid)

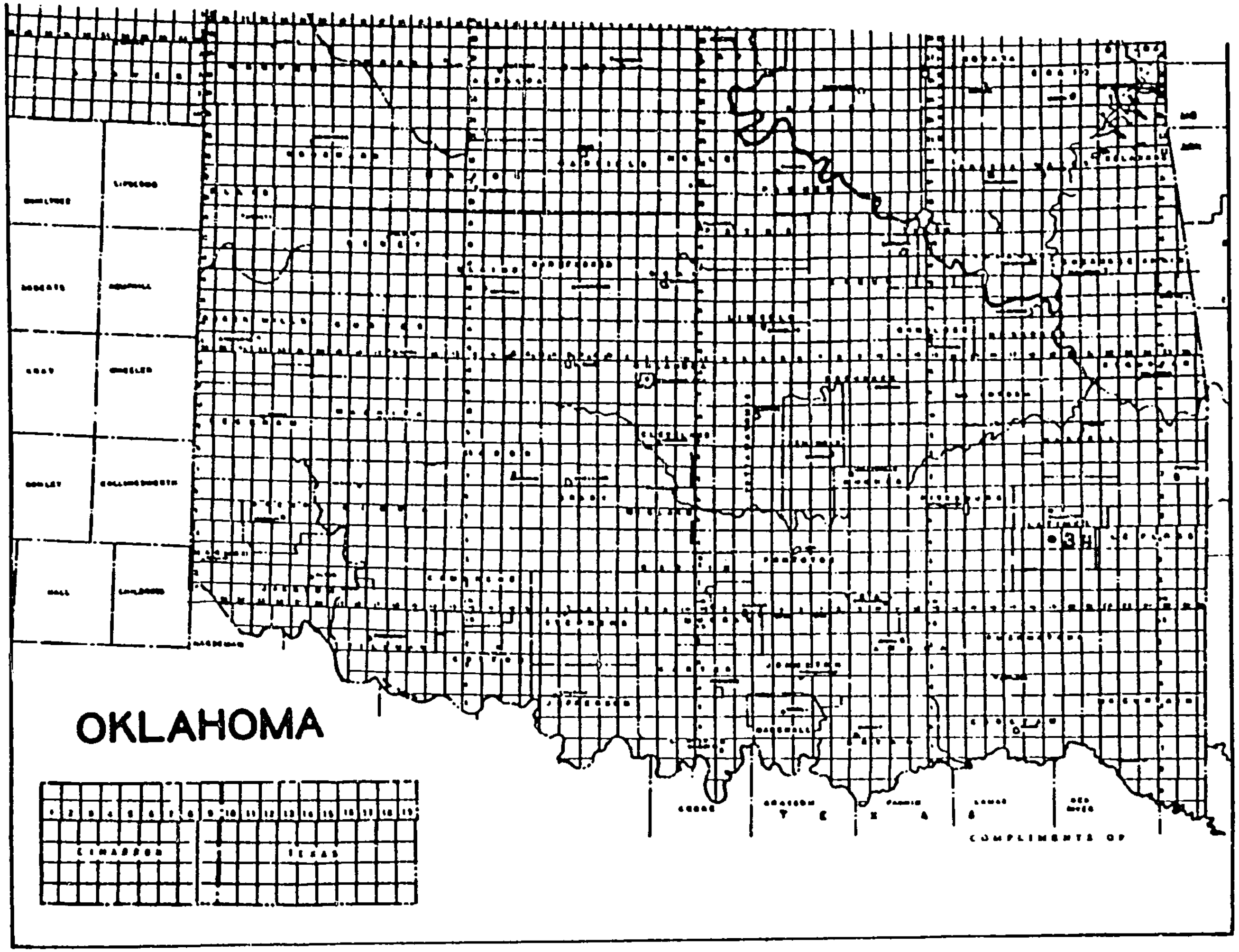


Fig. 67. Location of drilled hole, Shell 1, Dipple, Eastern Oklahoma

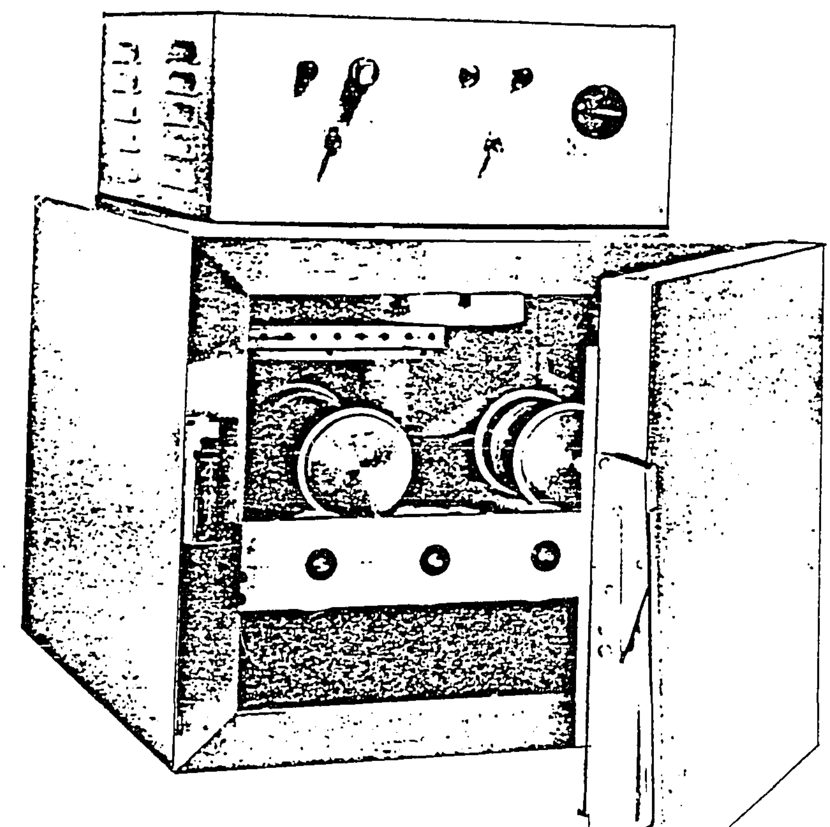


Fig. 68. The roller oven and aging cell.

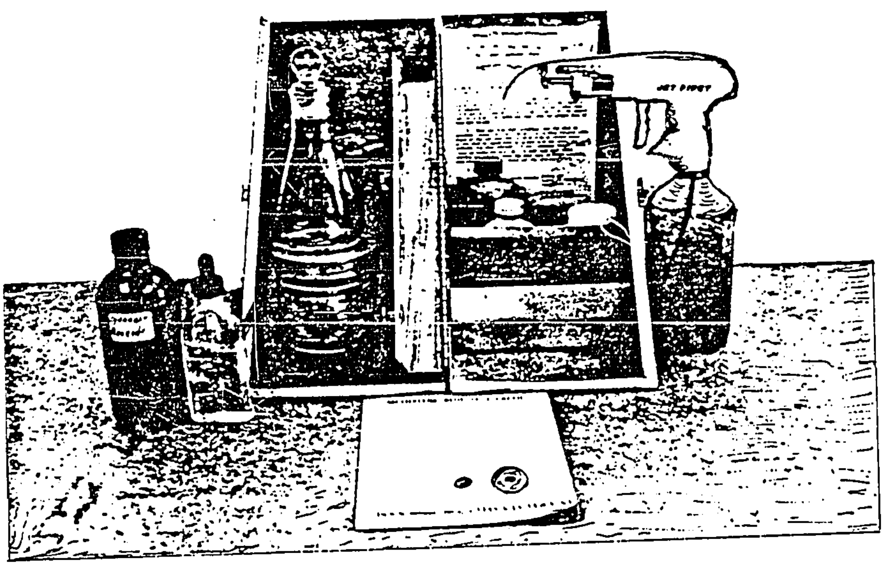


Fig. 69. Methylene blue test kit.

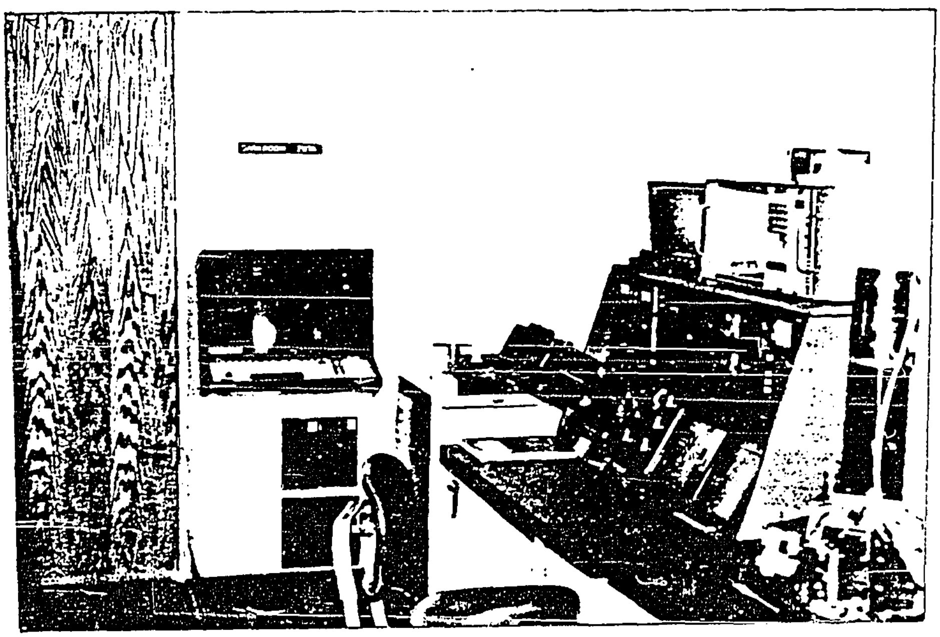


Fig. 70. The Scanning Electron Microscope

APPENDIX E

GRAPHICAL AND LETTER SYMBOLS USED IN APPENDICES

Graphical and Letter Symbols Used in Appendices

Type of Drilling Fluid	Letter Symbol	Graphical Symbol
<u>Untreated Mud</u>		
Ilmenite Weighted Mud	(A)	○
K-M Hematite Weighted Mud (synthetic hematite)	(B)	●
Itibirite Weighted Mud	(C)	□
Barite Weighted Mud	(D)	△
<u>Mixed Mud</u>		
Ilmenite-Itibirite Mixture	(AC)	⊡
Barite-K-M Hematite	(BD)	◬
<u>Improved Mud</u>		
Mud A + polymer + thinner(s)	(F)	◎
Mud B + polymer + thinner(s)	(G)	⦿
Mud C + polymer + thinner(s)	(H)	■
Mud D + polymer + thinner(s)	(I)	▲
<u>Improved Mixed Mud</u>		
Mud AC + polymer + thinner(s)	(FH)	⊠
Mud BD + polymer + thinner(s)	(GI)	⊗

Figure 71.