

Foth & Van Dyke

REPORT

**Slurry Cutoff Wall System
Preconstruction Report**

Scope ID: 91F6

*Flambeau Mining Company
Ladysmith, Wisconsin*

July 1992

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July 14, 1992

Mr. Gordon Reinke, Coordinator
Mine Reclamation Unit
Bureau of Solid and Hazardous Waste Management
101 South Webster Street, GEF II
Madison, WI 53707

RECEIVED
JUL 14 1992
BUREAU OF SOLID &
HAZARDOUS WASTE MANAGEMENT

Dear Mr. Reinke:

RE: Flambeau Project - Preconstruction Report, Slurry Cutoff Wall System


On behalf of the Flambeau Mining Company (Flambeau), we are submitting 12 copies of the Preconstruction Report pursuant to Part 2 - Item 23 of the Mine Permit approval for the Flambeau Project in Rusk County, Wisconsin. This report is a supplement to the Preconstruction Report submitted to you on June 18, 1991.

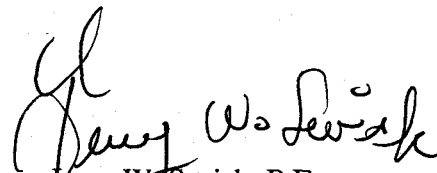
As we have recently discussed, the slurry wall construction described in the preconstruction, slurry wall system report is tentatively scheduled to be constructed in early September. Therefore, we are requesting that the Department review this document as expeditiously as possible and provide its approval such that construction activities can proceed on schedule.

If you have any comments or questions regarding this submittal, please contact us at 414-497-2500.

Sincerely,

Foth & Van Dyke


James B. Hutchison, P.E.
Project Engineer


Jerry W. Sevick, P.E.
Group Vice President

JBH:JWS:cac

Enclosure

Distribution

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**Slurry Cutoff Wall System
Preconstruction Report**

Scope ID 91F6

Prepared for
Flambeau Mining Company

Submitted by
Foth & Van Dyke and Associates Inc.

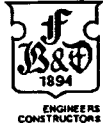
July 1992

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F B & D
TECHNOLOGIES, INC.

Slurry Wall System Submittal
For The
Flambeau Project

Prepared For:

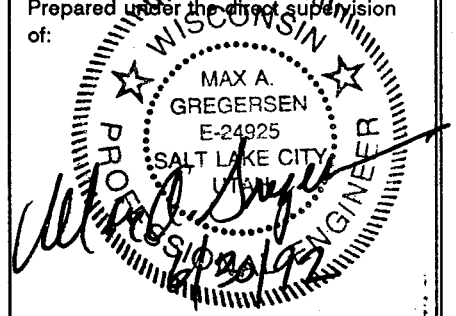
Flambeau Mining Company

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July 1992

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Slurry Cutoff Wall System Preconstruction Report for the Flambeau Project

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Drawing 120-C-027 Slurry Cutoff Wall, Site Grading Plan

1 Introduction

1.1 Purpose and Need

During the development of the Mining Permit Application for Flambeau Mining Company's Flambeau Project, discussions were held between representatives of the Wisconsin Department of Natural Resources (WDNR) and Flambeau Mining Company (Flambeau) regarding the project's slurry cutoff wall system design. As a result of those discussions, it was agreed that the information regarding the slurry cutoff wall system that was to be ultimately included in the permit application would be based on a preliminary design and that more detailed information regarding the final system design would be submitted to the WDNR in a preconstruction report to be prepared by Flambeau. This agreement was formalized by an approval condition (No. 23) included in Part 2 of the Mine Permit as set forth in the document titled "Decision, Findings of Fact, Conclusions of Law, and Permits (Docket No. IH-89-14)" dated January 14, 1991 as issued by the Wisconsin Division of Hearings and Appeals. This condition requires Flambeau to submit a preconstruction report to the WDNR regarding the slurry cutoff wall system a minimum of 60 days prior to the commencement of slurry cutoff wall construction. This report fulfills the requirements of the Mining Permit, Part 2, Condition No. 23.

The report consists of a description of the slurry cutoff wall system, identification of the contractor selected to construct the system; product identification, a description of slurry mixing equipment, and a description of bentonite sources and gradation. Also included in the report narrative is a quality assurance and monitoring plan. Included in appendices to this report are contractor qualifications; construction specifications; bentonite product data; specifications for drilling fluid materials and geotechnical calculations.

1.2 Project Description

Flambeau Mining Company (Flambeau), a wholly owned subsidiary of Kennecott Corporation, proposes to mine by the open pit method; a small copper ore deposit near Ladysmith, Wisconsin. The ore deposit contains economically valuable quantities of copper and gold. At its maximum size, the open pit will be approximately 32 acres in size and would be approximately 550 feet wide, 2,600 feet long, and 225 feet deep. Ore produced at the mine will be crushed and shipped via rail to an existing out-of-state processing plant.

Major project facilities, other than the open pit, include: stockpiles for ore, waste rock, overburden, and topsoil; an ore crusher; a haul road; a wastewater treatment plant; run-off control basins; a flood control dike; a slurry cutoff wall system; a rail spur; and various support buildings. The site will cover about 181 acres.

At the end of mine production, the project area will be reclaimed and returned to its approximate original contours and vegetation. All waste rock, overburden, and soils would be returned to the open pit in their approximate original sequence. The entire site will be replanted to grasslands and trees. A 7.5 acre wetland will be created over the western end of the reclaimed pit. The flood control dike and slurry wall system will remain as part of the reclamation plan.

1.2.1 Project Location

The project is located about 1.6 miles south of the City of Ladysmith, Wisconsin adjacent to State Highway 27 (STH 27). Ladysmith, the county seat of Rusk County, is located in rural northwestern Wisconsin approximately 130 miles east and north of Minneapolis-St. Paul; 240 miles north and west of the Wisconsin state capitol at Madison; and approximately 400 miles north and west of Chicago. It is near the junction of major north-south (STH 27) and east-west (US Highway 8) highways and a major Wisconsin Central, Ltd. railroad junction, as shown on Figure 1, Project Location Map.

The site is situated east of the Flambeau River, west of State Highway 27, north of the south line of Section 9, and south of Blackberry Lane. Also included within the site is a 24-to-36-foot wide corridor east of State Highway 27 on which the railroad spur line is to be constructed.

1.2.2 General Description of the Slurry Cutoff Wall System

During geological investigations conducted to support Flambeau's December 1989 Flambeau Project Mining Permit Application, a permeable zone of sand and gravel was located between the Flambeau River and the northwest corner of the proposed pit. To minimize the contribution of groundwater inflow into the pit from this zone, a slurry cutoff wall was included in the project design.

As originally envisioned through a preliminary design, the slurry cutoff wall was to be approximately 550 feet long. (Note that the Mining Permit Application narrative stated the length was to be approximately 400 feet. Figures 4-7 and 4-8, in the application, which are correct, show the length to be approximately 550 feet). The wall was to be constructed from the ground surface to the top of the Precambrian bedrock. The alignment of the slurry cutoff wall was to be along the western edge of the proposed pit. The wall alignment was to take it along and through the flood control dyke planned to be built across Stream B.

Drawing No. 120-C-027 contains plan view and section information depicting the final design for the project's slurry cutoff wall system. The system consists of two components. The first component is a slurry soil-bentonite wall. The second component is a slurry concrete diaphragm wall.

The concrete diaphragm wall will serve to structurally stabilize the overburden on the west end of the pit where the slurry soil-bentonite system component comes closest to the pit wall. The need to provide additional structural support in this area was developed during final system design when it was determined that an additional factor of safety against sideslope failure in the overburden between the pit and slurry soil-bentonite wall was desired. Structural stability calculations prepared by Call & Nicholas, Inc. (Appendix A) show that the system as designed has a factor of safety of 1.5, which meets the typical minimum desired factor of safety. Call & Nicholas, Inc. did the structural stability calculations for the design of the pit proper and are therefore intimately familiar with the project.

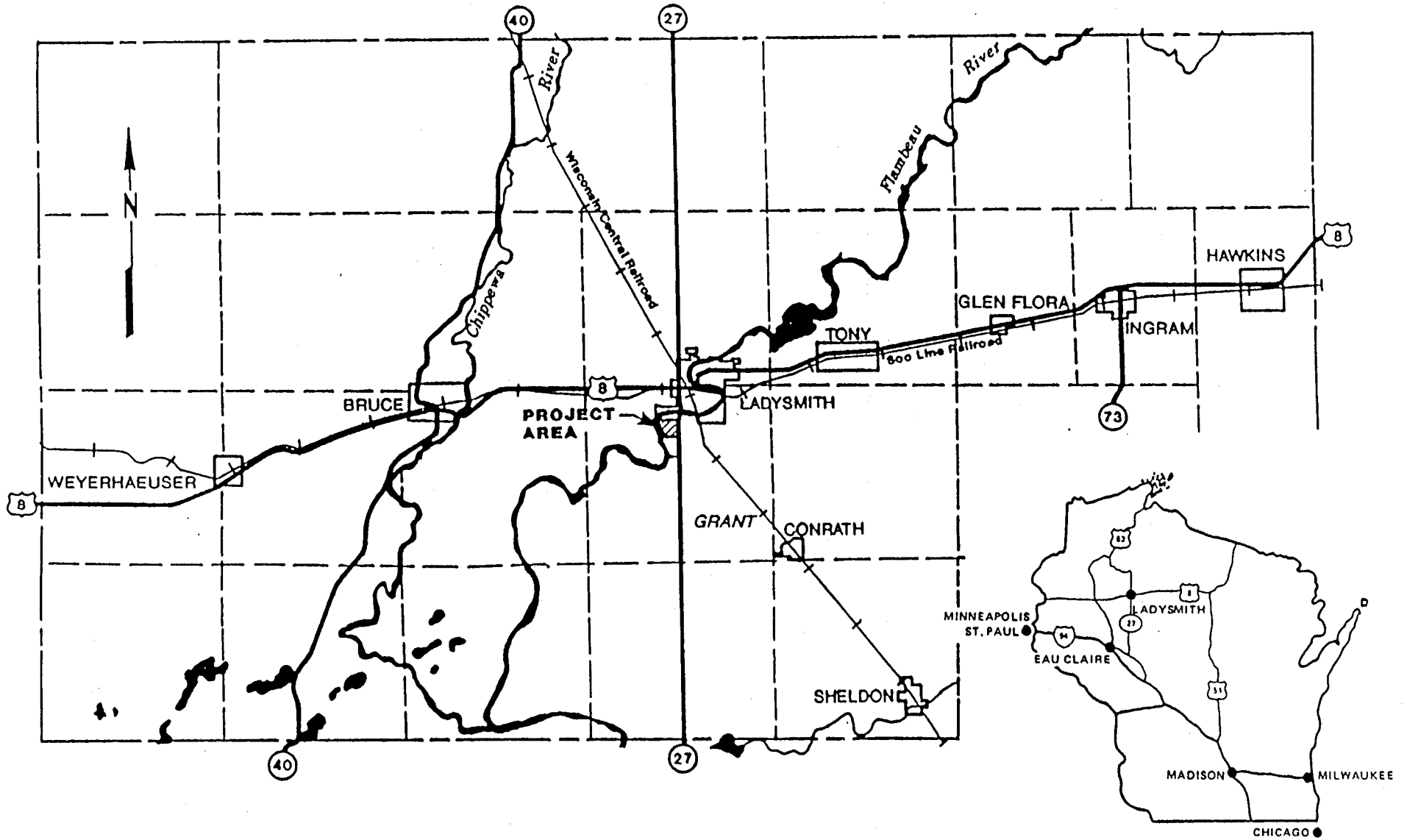


FIGURE 1
PROJECT LOCATION MAP

As shown on Drawing No. 120-C-027, the alignment for the slurry cutoff wall system is very similar to that developed for the preliminary design. The slurry soil-bentonite wall component commences at approximately the northwest corner of the pit, traverses slightly east of south and then doglegs to the southeast. This component is approximately 400 feet long. The slurry diaphragm wall component is located between the slurry soil-bentonite wall component and the pit and is orientated in a northwest to southeast direction. This system component is approximately 300 feet long, running approximately parallel to the southeast traversing leg of the slurry soil-bentonite wall. The two system components overlap approximately 140 feet. Total slurry cutoff wall system length is approximately 560 feet. As was originally envisioned, the system will be constructed over and through the flood control dyke.

Also, as shown on Drawing No. 120-C-027, a number of groundwater monitoring wells included in the project's groundwater monitoring program are located near the alignment of the slurry cutoff wall system. The only monitoring well that likely will be impacted by system construction is MW-1000. A final determination of potential impact on this and other monitoring wells will be made at the time the alignment of the system is surveyed in the field. Any wells to be impacted will be properly abandoned in accordance with NR 141.25 if the well will not be removed in its entirety by mine pit excavation. Replacement wells will be installed following discussions with WDNR personnel regarding location and construction specifications.

Construction techniques for both system components are similar in that trenches are excavated using a bentonite slurry to hold the hole open. In the case of the soil-bentonite component, the trench is backfilled with a soil-bentonite mixture. In the case of the diaphragm component, the trench is backfilled via a tremie pipe with concrete. Details regarding component construction are presented in Section 4 of this report. General information regarding slurry diaphragm wall construction is provided in Appendix B.

The slurry soil-bentonite cutoff wall component will be three feet thick and extend to and be keyed into the underlying bedrock, approximately 20 to 50 feet below the surface. Soil-bentonite backfill will be placed from the base of trench excavation to elevation 1087 which is two feet above the normal Flambeau River water.

The slurry concrete diaphragm wall component will consist of a two-foot thick reinforce concrete wall connected by tie rods to a deadman anchor. The diaphragm wall will extend to a depth of approximately eight to 25 feet below the ground surface. The reinforced concrete wall will extend from the base of trench excavation to approximately one foot above final elevation of the surrounding grade.

2 Slurry Cutoff Wall System Contractor

The Flambeau Project slurry cutoff wall system will be installed by Case International Company (Case), P.O.Box 40, Roselle, Illinois, 60172, one of the leading firms in slurry wall construction. Resumes of the key Case personnel who will be actively involved in this project are included in Appendix C. The resumes contain information regarding the background and experience of each person assigned to the project and a description of the project assignments with which they have been previously involved.

3 Project Organization

The Slurry Wall Contractor shall organize the necessary personnel to provide:

- Slurry Trench Specialist who has had a minimum of three years of proven and successful experience in slurry trench construction.
- Construction Quality Control Specialist for conducting field testing and control of the bentonite slurry, and documentation of field construction methods and testing.
- Soil-bentonite mix design by a registered professional Engineer in the State of Wisconsin.
- Experienced Operators and Construction Personnel familiar with the excavation and backfill of soil-bentonite cutoff walls.

The Flambeau Project Construction Manager will employ a third party Quality Assurance Contractor to perform:

- Testing, Observation and Construction Monitoring
- Construction Documentation for WDNR reporting requirements

4 Slurry Cutoff Wall System Construction

Construction of the slurry cutoff wall system involves site preparation, bentonite slurry preparation, trench excavation, trench backfilling and final site cleanup. This section of the report contains a description of these construction activities. Also included in this section is a listing of the equipment to be used during construction activities.

4.1 Site Preparation

Prior to start of construction of the slurry wall system, a construction corridor approximately 60 to 80 feet wide will be graded along the east side of the slurry wall system alignment. Topsoil will be removed from this area and temporarily stored nearby for replacement and revegetation following completion of construction. The construction platform will be graded to provide a two percent sloping work area with all drainage being directed away from the Flambeau River. A berm will be established along the river side of the construction corridor to prevent drainage from migrating in that direction.

In general, erosion control will be accomplished in accordance with the project's Surface Water Management Plan dated May 1991. This plan was approved by the WDNR on July 3, 1991 (file reference 2720). Specifics regarding erosion control techniques are described in the following two paragraphs.

A temporary diversion ditch will direct upstream tributary storm runoff away from the slurry wall system construction site. Temporary sedimentation ponds, straw bales, or silt fences will be installed along the ditch to remove suspended soils, as directed by the Construction Manager.

Storm water in the construction area will be directed to a temporary sump. The sump will be sized to handle runoff from a two-year 24-hour storm. Materials excavated during construction of this sump will be placed in the Type I Stockpile. A temporary shallow ditch will direct run-off water from the construction corridor to the temporary sump. Run-off water will enter the sump, settling of suspended solids will take place, and the clarified effluent will be pumped to the settling ponds. No direct discharge to the Flambeau River will take place from the construction area.

4.2 Bentonite Slurry

4.2.1 Bentonite Sources and Gradation

Bentonite, as supplied to the site prior to mixing, shall have properties suitable for the functions of acting as a stabilizing fluid during slurry wall construction and as a component of the soil-bentonite backfill. The bentonite shall be a high-swelling, pure, premium grade powdered, sodium cation base product consisting mainly of montmorillonite bentonite clay. The bentonite shall conform to American Petroleum Institute (API) Specification 13A. An excerpt of API specification 13B relating to bentonite is included in Appendix D.

Chemically-treated bentonite will not be permitted. A suitable bentonite brand, such as Hydrogel or Federal Jel "90" or approved equal will be selected after tests performed to determine that the viscosity range, the gel strength and filtration properties satisfy the values set forth in the design specifications. Test procedures are described in Appendix E. Bentonite will be pulverized with 80 percent or more passing the P200 sieve. Examples of potential bentonite suppliers are listed below. Bentonite product data are included in Appendix F.

- M.I. Drilling Fluids (615) 528-6438
- WYO-BEN, Inc. (406) 652-6351; 1-800-548-7055
- The Bentonite Corp. (303) 291-2943

4.2.2 Slurry Preparation

Bentonite slurry will be prepared by mixing bentonite and water in a continuous mixing batch plant. The mixed bentonite slurry will be allowed to hydrate in the designated slurry storage pond for at least eight hours. The storage pond and slurry mixing operation will be conducted on-site in the designated areas shown on Drawing 120-C-027. Mixing water will be obtained from the Flambeau River. Mixing water shall be clean, fresh, and free from oil, acid alkali, organic matter, or other deleterious substances. Density and marsh funnel tests (Appendix E) will be performed to control the production of the slurry. After hydration, the slurry will be delivered as needed by pump through a four-inch discharge hose to the trench. Viscosity adjustment of the slurry in the pond which exceeds the minimum requirements may be made as the slurry is pumped from the pond by thinning with water at the pump suction, or by recirculation and thinning.

4.3 Soil-Bentonite Wall

4.3.1 Soil-Bentonite Mix Design

Prior to the start of construction, a laboratory testing program, will be conducted to determine the proper blend of soil, borrow material, dry bentonite and bentonite slurry to achieve the required soil-bentonite permeability results. Compatibility testing with existing ground water will also be conducted. Sample batches will be mixed to the consistency achievable with field mixing equipment. Permeability tests will be conducted after consolidation of the samples. The laboratory tests that will be performed during the preconstruction testing program include:

- Grain size analysis of on-site materials.
- Atterberg Limits, water content and moisture-density (compaction curves).
- Permeability tests using water as the permeant.
- Slurry trench fluid mix testing to determine the percentage of bentonite required to seal the trench wall.

4.3.2 Soil-Bentonite Wall Construction

The slurry wall system will be constructed by using a back-hoe with a long boom extension. A trench will be excavated approximately three feet in width starting at the north end. Total length of the trench is expected to be approximately 400 feet. The trench will be initially excavated to bedrock and then lengthened along the alignment of the final wall. A mixture of seven percent bentonite and 93 percent water will be mixed in a mixer located within the corridor. The bentonite slurry will then be mixed within a hydration pond to allow hydration of the bentonite to occur and to meet the slurry mix specifications. The hydrated mixture will then be pumped into the trench as material is excavated. This mixture of seven percent bentonite acts as a form of "drilling mud" and effectively keeps the trench from caving during excavation. The excavated material will be placed on the work pad east of the trench for re-use as backfill in the finished slurry wall or removal to the Type I or Type II stockpile. Drainage will be directed to the temporary sump. The mix design test program will verify the percentage of bentonite slurry required to maintain the trench in the field.

As soon as the trench has been excavated to a sufficient length, backfilling will commence. Backfill material will consist of a mixture of select on-site material, and/or imported clay (depending on material quality), and approximately two percent to five percent bentonite as determined by the design mix testing program. The backfill will be spread within the construction corridor, adjacent to the trench.

The width and length of the backfill pile will vary depending on the amount of backfill required at the time. The height of the pile will be from one to two feet. The bentonite will be mixed with water in the mixer and sprayed on the backfill. The backfill material and bentonite will be mixed and blended by windrowing, disk harrowing, by bulldozing, or blading as the contractor best determines in the field in order to achieve a homogeneous consistency having a four-inch to six-inch slump as measured of ASTM C143. The mixed backfill will then be placed in the excavated trench at a rate compatible with the excavation rate. The backfill material will fall to the bottom of the trench and form an angle of repose of between 5H:1V to 10H:1V. This process will continue until the trench has been completely backfilled. During the final stage of backfilling, slurry from the trench will overflow to a temporary trench, and be directed to the sediment sump. A cap will then be placed on top of the soil-bentonite wall. The cap will consist of soil placed to a finished elevation of 1087. The remainder of the trench will be brought to grade with soil backfill as shown in Drawing No. 120-C-027.

Upon completion of the trench backfilling, the disturbed area will be topsoiled and revegetated except for a narrow access road approximately 30 feet to 40 feet wide immediately adjacent to the mine pit and those areas to be excavated within the pit perimeter. Further details describing the soil-bentonite slurry wall are contained in Appendix G (Slurry Wall Construction Specifications).

Major equipment to be used for construction of the slurry soil-bentonite wall include a bulldozer, extended boom back-hoe, a slurry batch plant for mixing bentonite, a forklift for handling pallets of bentonite, and one or two pick-up trucks. Supplies of bentonite, clay and backfill material will be stored within the construction corridor. Total time to prepare the area, construct the slurry wall, and revegetate the disturbed areas is expected to be approximately eight weeks.

4.4 Diaphragm Wall

4.4.1 Diaphragm Wall Design

The slurry diaphragm wall will consist of 19, two-foot thick reinforced concrete panels constructed in-situ (Appendix H). The panels will range in width from 12 feet at both ends of the wall to 18 feet in the center. Panel depth will range from eight feet at both ends of the wall to 25 feet in the center.

The diaphragm wall will be supported horizontally by a deadman/tie rod system. The deadman will be located approximately 25 feet behind the diaphragm wall and consist of a two-foot thick concrete beam constructed from the ground surface to a depth of 6.5 feet. Anchor rods will connect the deadman to the diaphragm wall. The deadman/tie rod system will be constructed behind panel Nos. 4 through 16 for a total length of approximately 212 feet. Calculations for sizing the deadman and spacing of the tie rods are also contained in Appendix H.

The concrete for the diaphragm walls will have a minimum cement content of 564 pounds per cubic yard. The slump of the concrete will be between six and nine inches for tremie placement. The concrete will have a minimum cylinder compression strength of 4,000 psi at 28 days. Panel reinforcement will be assembled in individual cages for inception into each excavated slurry-filled panel trench.

4.4.2 Diaphragm Wall Construction

The diaphragm wall will be constructed in accordance with the specifications in Appendix I. First, the site will be cleared as described in Section 4.1. A temporary guide wall will then be installed along the perimeter of the diaphragm wall. The guide wall is a lightly reinforced footing placed on both sides of the diaphragm wall alignment to depths of three to five feet. The guide wall serves to guide the slurry excavation tools during initial panel excavation as well as to stabilize the upper soil strata. With the guide walls in place, the panels are then excavated. The panel excavation sequence is to first dig a primary panel on either side of a secondary or closure panel. The panels are excavated under a full head of bentonite slurry with a specially designed eight to ten-ton slurry clamshell bucket. The bentonite slurry will be delivered by means of a slurry pump previously discussed in the bentonite slurry section of this report (Section 4.3.2).

Once the panel excavation is complete, the steel reinforcing cage will be placed in the excavation. V-joint forms known as bulkheads will be placed at each end of the primary panels to form interconnecting keys or joints between panels. Concrete is then placed into the bottom of each panel by a ten-to-12-inch diameter tremie pipe. As the concrete rises in the panel, slurry is displaced and pumped off at the top of the panel to storage tanks to be reused during future panel excavations. The tremie pipe is removed in ten-foot sections as the concrete rises the full height of the diaphragm wall. Within a few hours of the concrete pour, the V-joint forms are removed.

This entire procedure of excavating and concrete pouring is repeated again for the secondary or closure panels. The formed V-joints or keys provide both the vertical excavation guide and a keyway between the primary and secondary panels.

The final step to the diaphragm wall construction consists of construction of a cap beam along the length of the diaphragm wall.

Similarly to the soil-bentonite wall, upon completion of wall construction the disturbed area in the vicinity of the diaphragm wall will be topsoiled and revegetated except for a narrow access road approximately 30 feet to 40 feet wide immediately adjacent to the mine pit and those areas to be excavated within the pit perimeter.

Major equipment to be used for construction of the diaphragm wall include a bulldozer, a slurry batch plant for mixing bentonite, a fork lift for handling pallets of bentonite, one or two pick-up trucks, a specialized clamshell for excavation of the panels, and concrete trucks for delivery of the concrete. Supplies of bentonite and reinforcing steel bar will be stored within the construction corridor. Concrete will be supplied by a local concrete plant in ready-mix trucks. The total time to prepare the area, construct the diaphragm wall and revegetate disturbed areas is expected to take 8 weeks.

5 Monitoring Plan & Quality Assurance Plan

The Flambeau Project Construction Manager will provide independent quality assurance monitoring and testing during construction of the slurry wall. The third party Quality Assurance Contractor will work closely with the Slurry Cutoff Wall Contractor to document that construction is accomplished according to specification. The Contractor will maintain his own quality control program for slurry wall construction under the direction of a qualified Engineer. The Contractor shall provide all necessary equipment and personnel to test and determine that the mixing of the slurry and backfill and the construction of the slurry trench meet the requirements of the specifications. Testing by the Quality Assurance Contractor shall in no way relieve the Contractor of the responsibility of performing testing necessary to meet the construction requirements.

Tests to determine slurry properties shall be made in accordance with testing procedures described in American Petroleum Institute Recommended Practice 13B, Latest Edition, Standard Procedure for Testing Drilling Fluids shown in Appendix E.

5.1 Testing Methods and Procedures

The following is a description of the types of QA/QC tests and procedures which will be performed during the slurry mixing, trenching and backfilling stages of the slurry soil-bentonite and slurry diaphragm wall work.

5.1.1 Bentonite Slurry

During bentonite slurry mixing the following tests will be performed:

- Weighing of dry bentonite for mixing in the batch plant.
- Measuring volume of water added to batch plant mixer tanks by flow meter.
- Acidity of mixing water by pH meter, one test per 100,000 gals used at least monthly.
- Viscosity of slurry immediately after mixing by Marsh Funnel Viscometer, two tests per eight hours working shift.
- Unit weight of slurry immediately after mixing by mud balance.

While the slurry is in the hydration ponds, viscosity will be measured with time to determine the maximum value achieved; this indicates complete hydration. In addition, the pH and viscosity of the ponds will be monitored each day.

5.1.2 Slurry Soil-Bentonite Wall

After mixing, the soil-bentonite backfill will be placed into the excavated trench. The quality of the bentonite slurry in the excavation during slurry trenching will be closely monitored to control the viscosity and density which could impact slurry wall effectiveness.

The depth of the wall during trenching will be measured relative to the work platform elevation by physical soundings at 20-foot intervals. Verticality is assured by the sweeping action of the backhoe bucket during excavation.

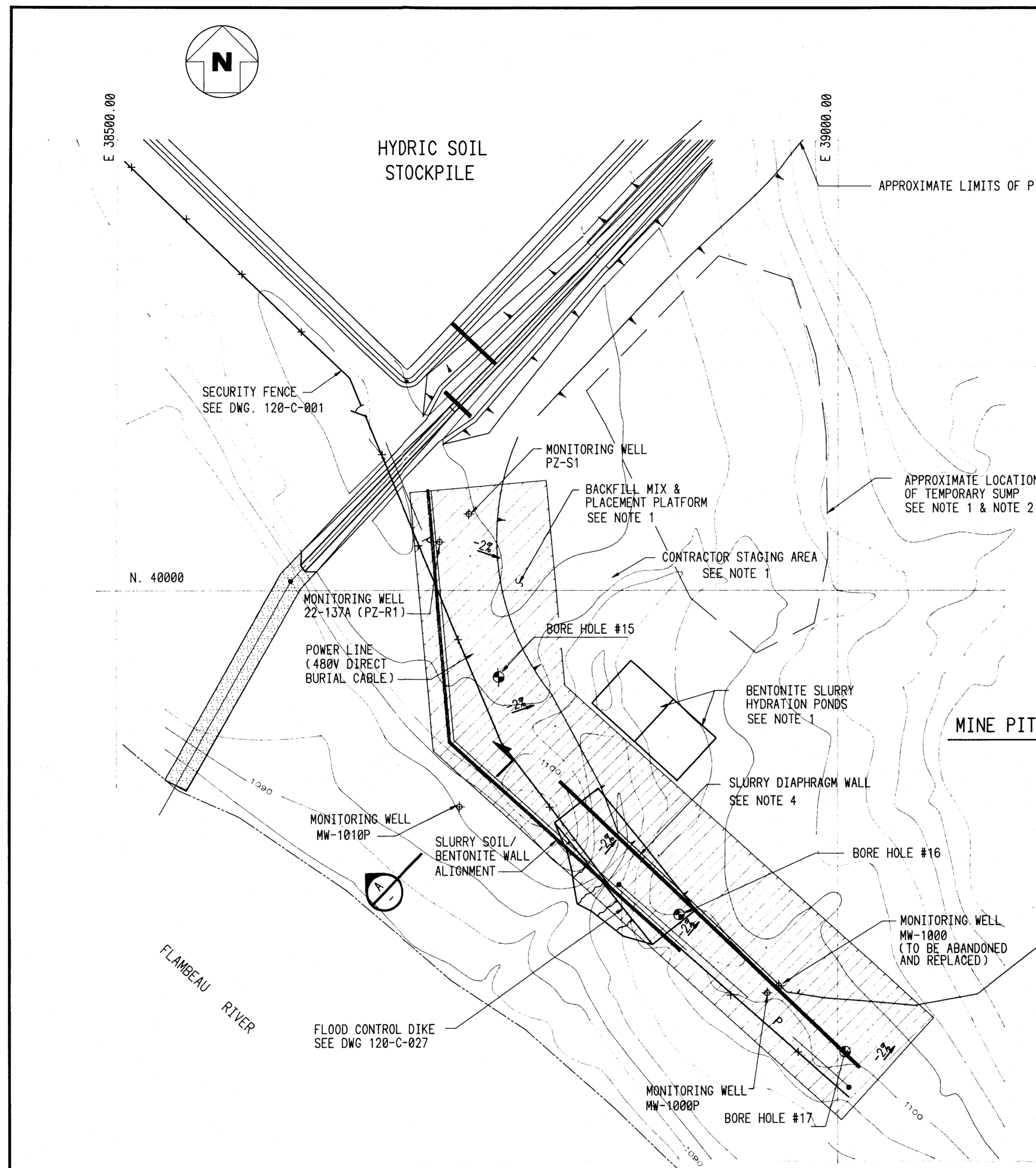
Water used for slurring mixing and make-up shall be tested at a rate of one test per 100,000 gallons used for TDS, hardness, and field pH. Soils from the base of the trench shall be tested at 50-foot intervals for grain size, analysis to the P200 sieve size, liquid limit, and plasticity index. Bentonite and admixed soil shall be tested at a rate of one test per 100 feet of wall for slump, one test per 1,000 cubic yards for permeability and unit weight, and grain size analysis.

5.1.3 Slurry Diaphragm Wall

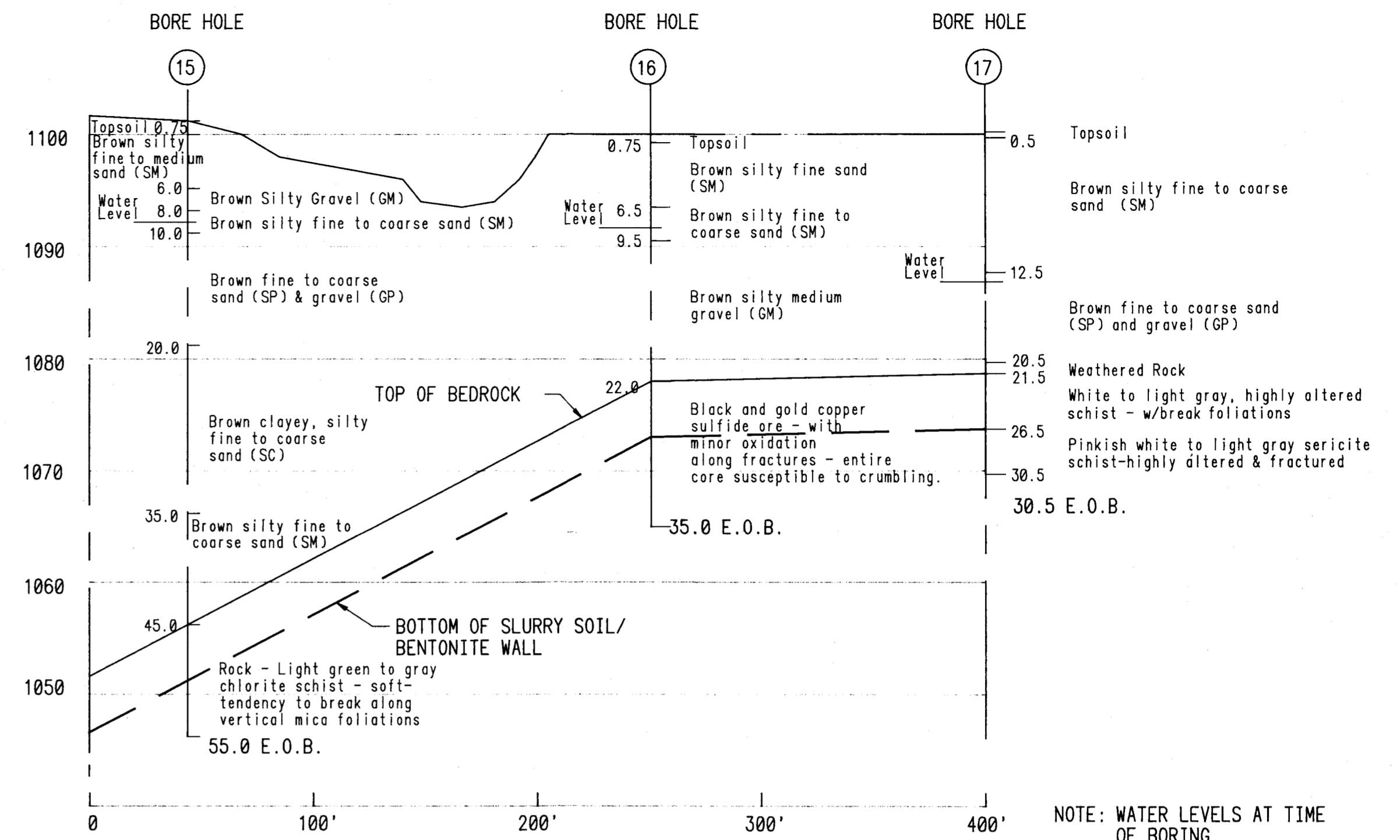
The quality of the bentonite slurry in the excavation during slurry diaphragm wall trenching will be closely monitored to control the viscosity and density which could impact slurry wall effectiveness.

The depth of the wall during trenching will be measured relative to the work platform elevation by physical soundings at 20-foot intervals. Verticality is assured by the guide wall installed as part of the construction process.

Water used for slurring mixing and makeup shall be tested at a rate of one test per 100,000 gallons used for TDS, hardness, and field pH. Two concrete cylinders will be made and tested in accordance to ASTM C192, C31, C39 and C42 for each panel of the diaphragm wall constructed. A slump test will be performed for each set of two cylinders made for the diaphragm wall concrete.

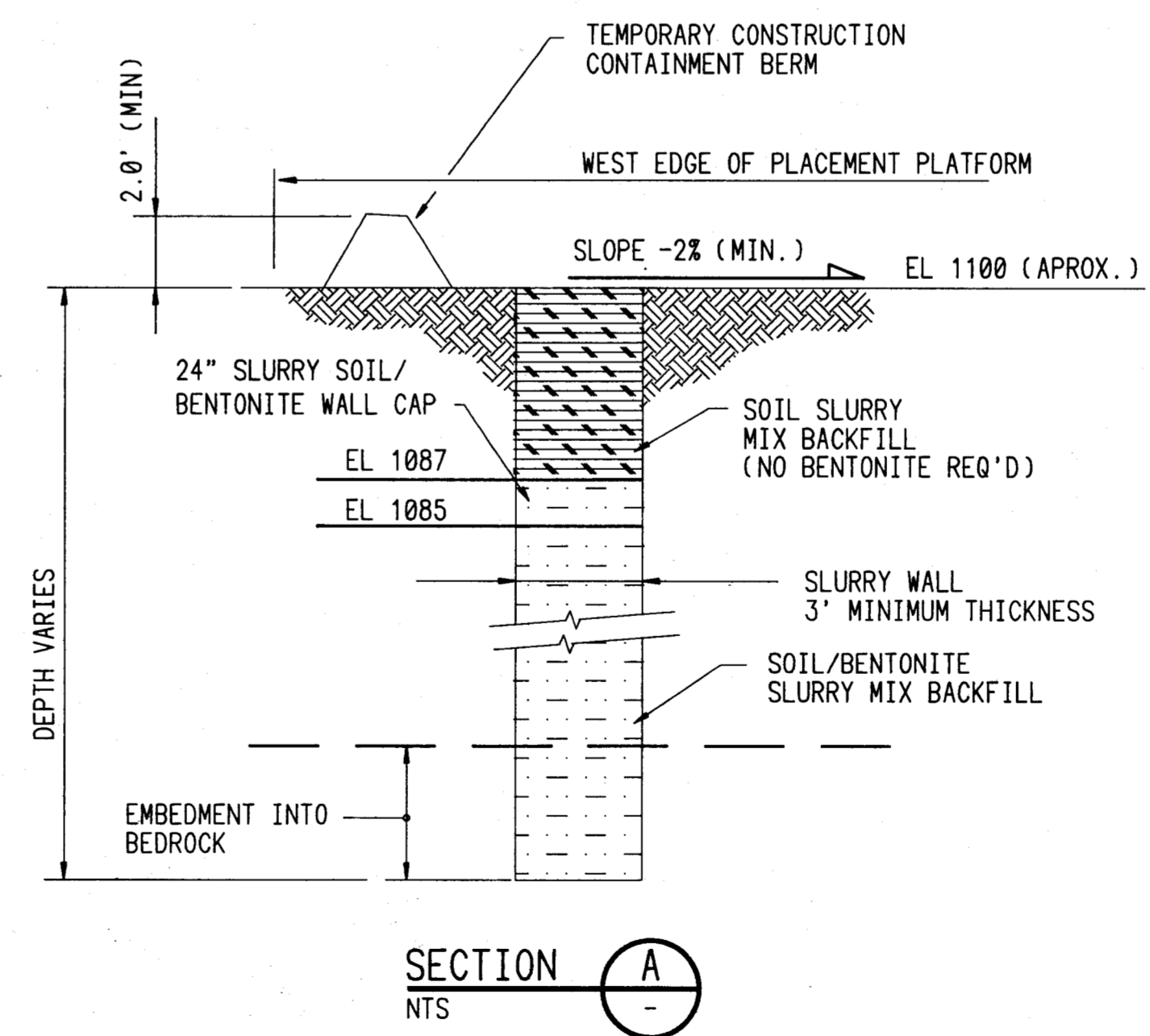


SLURRY SOIL/ BENTONITE WALL - SLURRY DIAPHRAGM WALL PLAN
SCALE: 1"=50'



BORING LOG PROFILE
SCALE: 1"=50' HORIZ.
1"=10' VERT.

- NOTES:
1. THE SHOWN LOCATION OF: SLURRY HYDRATION PONDS, CONTRACTOR STAGING AREA, TEMPORARY SUMP, AND THE BACKFILL MIX AND PLACEMENT PLATFORM ARE SUGGESTED LOCATIONS. FINAL LOCATIONS ARE TO BE BASED ON CONTRACTOR'S APPROVED DESIGN REQUIREMENTS AND THE CONSTRUCTION MANAGER'S SEDIMENT CONTROL AND SITE PLANS.
 2. THE SLURRY CUT OFF WALL SYSTEM CONSTRUCTION AREA SHALL BE ISOLATED FROM THE TRIBUTARY RUNOFF AREA BY DIVERSION DITCHES WITH TEMPORARY SUMPS AND DIKING AS REQUIRED AND WITH STRAW BALES OR SILT FENCES AS APPROVED BY THE CONSTRUCTION MANAGER.
 3. THE SLURRY SOIL/BENTONITE WALL LOCATION SHOWN IS APPROXIMATE AND SHOWN FOR BID PURPOSES ONLY. THE CONSTRUCTION MANAGER WILL FIELD LOCATE AND ADJUST ALIGNMENT AS REQUIRED TO MEET PERMIT REQUIREMENTS.
 4. THE 300' LONG SLURRY DIAPHRAGM WALL LOCATION SHOWN IS APPROXIMATE AND SHOWN FOR BID PURPOSES ONLY. THE CONSTRUCTION MANAGER WILL FIELD LOCATE AND ADJUST ALIGNMENT AS REQUIRED TO MEET PERMIT REQUIREMENTS.



SECTION A
NTS

NO	DATE	REVISIONS	BY	CHK'D	ENGR	ENGR	NO	DATE	REVISIONS
B	8-26-91	FOR APPROVAL	GLT				H	1/12/92	
D	3-10-92	REVISE PER CLIENT DESIGN DATA	GLT						
E	4-1-92	FOR QUOTE	GLT						
F	6-30-92		GLT						
G	7-7-92	MISC.	GLT						

Ford, Bacon & Davis Utah, Inc.
ENGINEERS - CONSTRUCTORS
375 CHIPETA WAY SALT LAKE CITY, UTAH

BY	CHK'D	ENGR	ENGR	ENGR	NO	DATE	REVISIONS

BY	CHK'D	ENGR	ENGR	ENGR	NO	DATE	REVISIONS

BY	CHK'D	ENGR	ENGR	ENGR	NO	DATE	REVISIONS

FLAMBEAU PROJECT
LADYSMITH, WISCONSIN

NO	DATE	REVISIONS

BY	CHK'D	ENGR	ENGR	ENGR	NO	DATE	REVISIONS

BY	CHK'D	ENGR	ENGR	ENGR	NO	DATE	REVISIONS



CFASBC

Appendix A

Structural Stability Calculations

3625 E. 42nd Strav.
Tucson, Arizona 85713 U.S.A.

July 15, 1992

Mr. Jim Hutchison
Foth & Van Dyke Associates
2737 South Ridge Road
Greenbay, WI 54304-9102

Re: Review of Overall Soil Slope Stability, Concrete Diaphragm,
and Slurry Wall Design, Flambeau Mining Company, Ladysmith,
Wisconsin.

Dear Mr. Hutchison:

This letter is to provide you with our opinion with respect to the stability of soil slope including the proposed concrete diaphragm and slurry wall. The purpose of the concrete wall is to retain the soils above bedrock in the Southwest Design Sector. These soils consist primarily of Glacial Till consisting of silty sands and gravels. A slurry wall will be constructed approximately 30 feet behind the concrete diaphragm wall.

The information used in the slope stability analyses presented in this letter were obtained from the following sources:

1. Call & Nicholas Inc.'s 1988 report, Flambeau Project Pit Slope Design.
2. Boring logs and soil test results from studies performed by Foth & Van Dyke in 1991.
3. Information about the site including geology, topography, and groundwater conditions obtained from Flambeau Mining Company.
4. Preliminary Diaphragm Wall Designs provided to Flambeau Mining Company by Case International Company, the contractor.
5. Conversations with Mr. Jeff Tygesen of Flambeau Mining Company, Mr. Zavis Zavodni of Kennecott, and Mr. Safdar Gill of STS Consultants, the designer of the wall.

Mr. Jim Hutchison
Page 2
July 15, 1992

To evaluate the stability of the slope we performed a slope stability analysis. A cross section (Figure 1), including the soil profile, through the concrete diaphragm wall was developed from the above listed information. A maximum exposed wall height of 20 feet was assumed as a conservative worst case condition. In the stability analysis, the water table between the tied back concrete diaphragm wall and the slurry wall is located approximately 12 feet below the surface. Based on the contractor's specifications, the slurry wall will be backfilled with a mixture of on or off site soils with a minimum of 4 to 5 percent bentonite clay. The stability analysis was conducted assuming a rigid tied back concrete wall based on Case International's design.

Material properties used in the slope stability analyses are as follows:

Material	Unit Weight (pcf)	Effective Angle of Friction (deg)	Effective Cohesion (psf)
Till	145	36	0
Saprolite	143	24	412
Bedrock	165	45	1000
Slurry Wall	90	28	0

The stability analyses were performed using UTEXAS, a slope stability analyses computer program. Spencer's method of slices was used to calculate the factor of safety. The results of the analyses are shown on Figures 1 and 2. Figure 1 shows the critical shear surface (Factor of Safety = 1.5) calculated by the computer program. Figure 2 shows a shear surface (Factor of Safety = 2.0) that extends through the slurry wall.

The stability analyses indicate that the most critical mechanism of instability is movement at the toe of the structural wall. We have reviewed the design method, soil parameters and overall stability of the wall. Both the design method and the soil parameters were selected conservatively, providing an additional margin of safety in the concrete wall design.

Mr. Jim Hutchison
Page 3
July 15, 1992

Based on the information we have on the site conditions, the design of the concrete diaphragm and slurry walls, and the above analyses, it is our opinion that the overall soil slope will be stable.

If you have any questions about the contents of our letter or the analyses performed, please contact us.

Sincerely,

CALL & NICHOLAS, INC.

George T. Lightwood

George T. Lightwood, P.E.
Mining Engineer

Enclosures: Figures 1 and 2

Flambeau - Overall Stability of Soil Slope
 Concrete Diaphragm Wall
 June 18

UTEXAS2

Input File

flam.in

Output File

flam.ou

Method Used

Spencer
 49 Slices

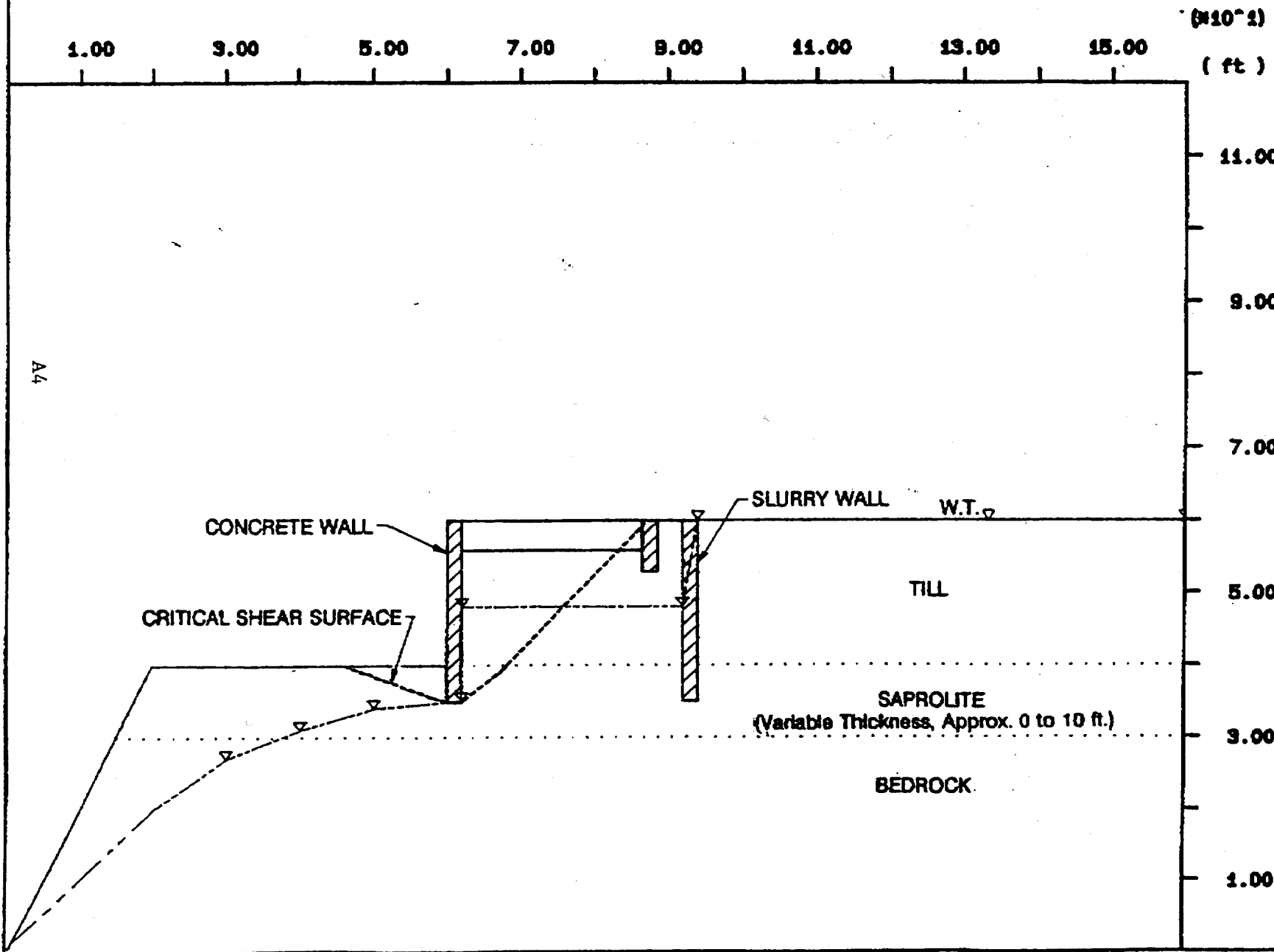
Calculated FS

1.5

flam.plt
 12: 57: 18
 08-20-1992

Call & Nicholas
 Tucson, Arizona

FIGURE 1



A4

Flambeau - Overall Stability of Soil Slope
 Concrete Diaphragm Wall
 June 18

UTEXAS2

Input File

flam1.in

Output File

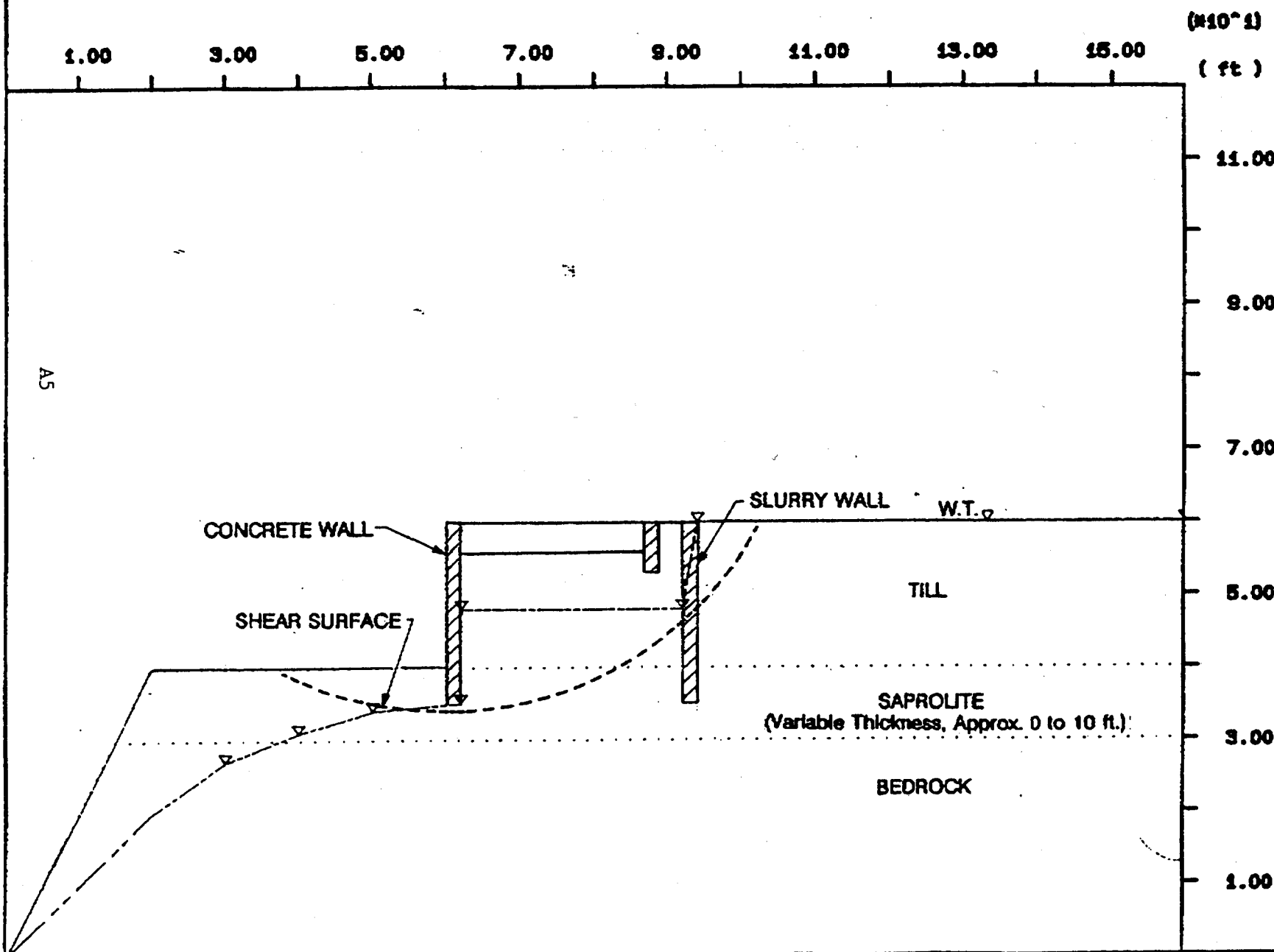
flam1.ou

Method Used

Spencer
 46 Slices

Calculated FS

2.0



flam1.plt
 12:28:16
 08-30-1992

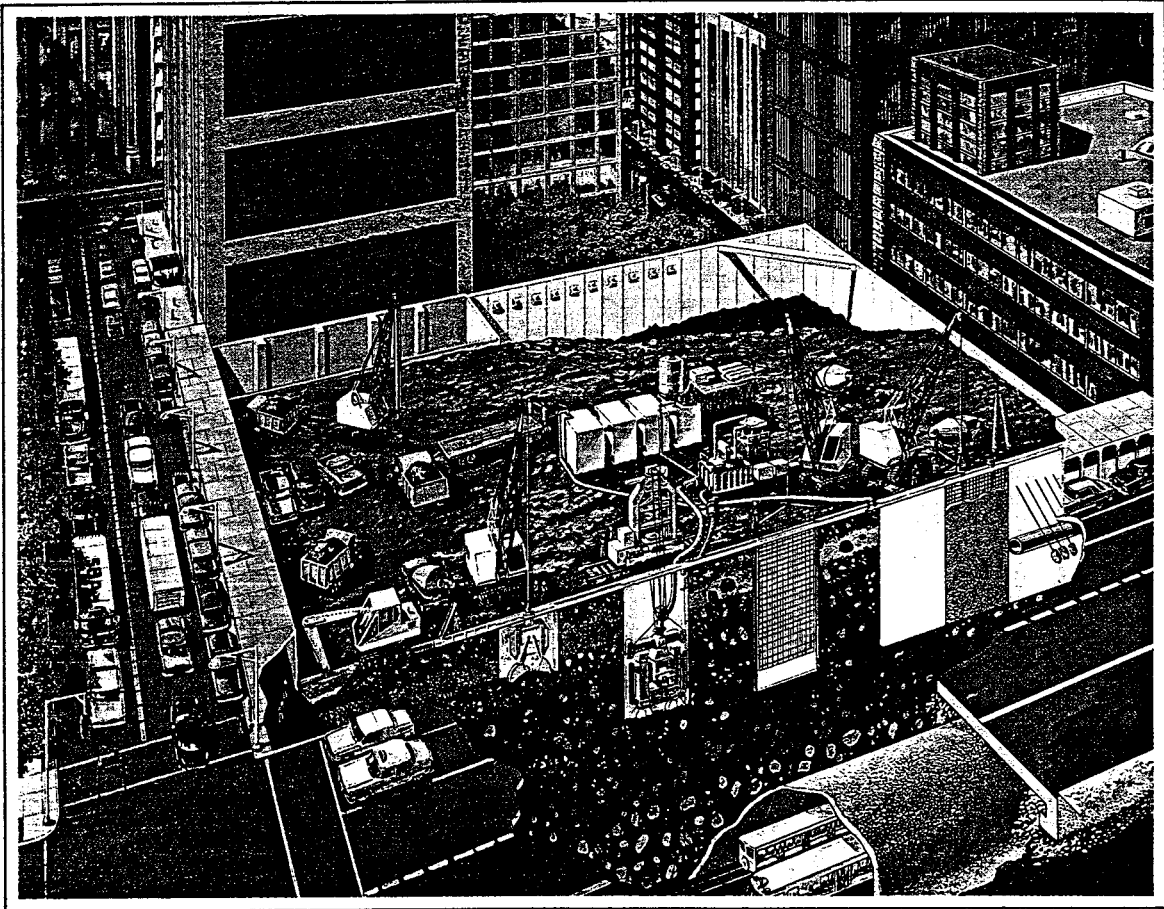
Cell & Nicholas
 Tucson, Arizona

FIGURE 2

Appendix B

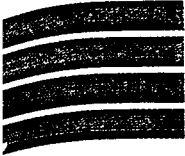
General Information Regarding Slurry Diaphragm Walls

Slurry Diaphragm Wall Construction Method

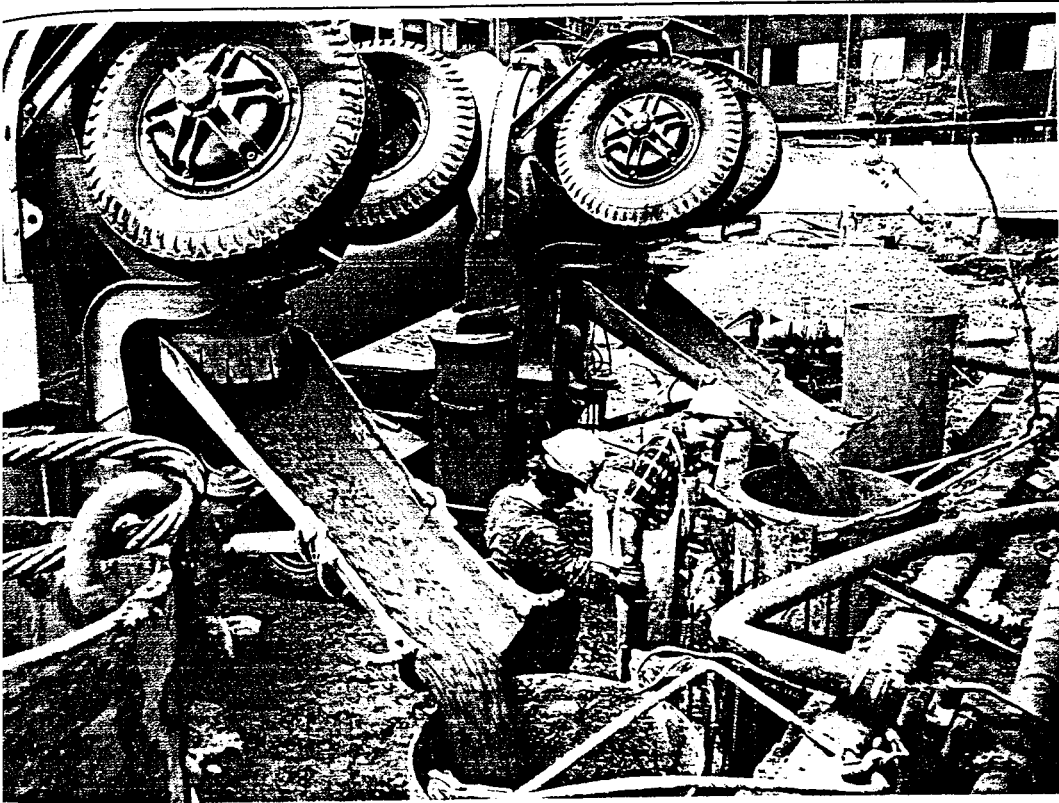
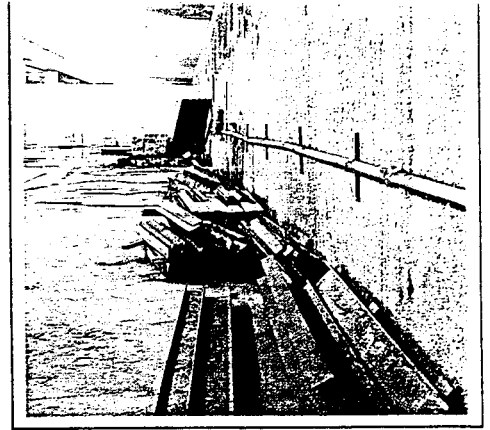


CASE
INTERNATIONAL COMPANY

A CASE GROUP COMPANY



Completed slurry diaphragm wall from the United Airlines Terminal Expansion Program, O'Hare International Airport, Chicago, Illinois.



Completed slurry diaphragm wall with tiebacks installed for the Wilshire-Midvale Building, Los Angeles, California.

Placement of concrete in primary panel using twin tremie pipe system.

Slurry clamshell shown excavating secondary panel.



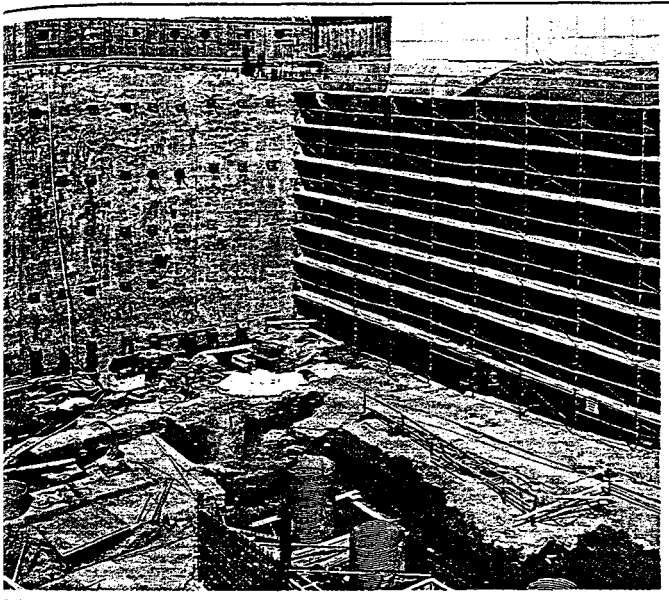
Slurry Diaphragm Walls...

an alternative construction method for difficult job site applications

Developed and applied successfully in Europe and Japan for many years, the slurry diaphragm wall construction method is used to solve difficult urban foundation problems. From a cost, technical and building schedule viewpoint, slurry diaphragm walls offer distinct advantages as an alternative to conventional "cut-and-cover" or other building construction methods. Slurry diaphragm walls are most effective and cost efficient, when applied to construction problems involving a high ground water table, unstable soils and restricted urban job site locations, such as job sites surrounded by adjacent buildings, streets or below ground subway tunnels and stations.

In many cases, conventional construction methods, such as sheeting and shoring, soldier piles and lagging or mass excavation, are either cost prohibitive or lack the technical capability to handle difficult earth retention, building underpinning or dewatering situations. For example, building sites with a water table close to the surface would require an extensive pumped dewatering system when using a conventional mass excavation building construction method. With a slurry diaphragm wall, the need for a dewatering system is significantly reduced due to the ability of the slurry diaphragm wall to reduce the volume of subsurface ground water entering the job site area.

In structural applications, a slurry diaphragm wall is principally used as a load bearing element or retaining wall. Further, on sites with closely adjacent buildings, conventional retaining wall construction techniques may create unacceptable, or unnecessary risks associated with adjacent ground movements. The slurry diaphragm wall technique, on the other hand, can reduce such risks without requiring *any high vibration* sheeting or pile driving activity.



▲ Placement of corner steel reinforcing cage.

TYPICAL APPLICATIONS

In the United States, the slurry diaphragm wall process has become quite popular in subway construction for both "cut-and-cover" tunnels and stations. Likewise, it has excellent application in large building construction on sites where adjacent buildings are creating lateral pressures, requiring a retaining wall for construction of the basement portion of the project.

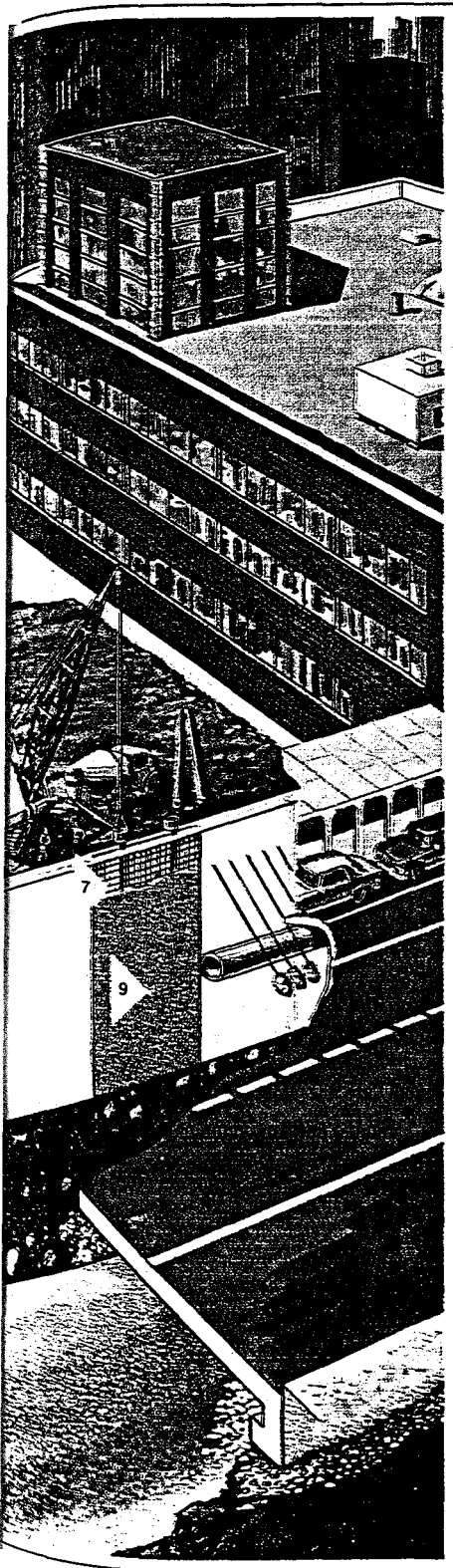
Based on cost efficiency and the ability to maintain the building schedule, slurry diaphragm walls are proving to be an ideal alternative to conventional construction methods. Costs for sheeting and shoring, soldier piles and lagging or mass excavation present prohibitive costs or may not be feasible in applications with high ground water tables, unstable or sandy soils or confined construction sites surrounded by adjacent buildings and high traffic volume streets. These are the projects which are ideally suited for the unique foundation capabilities inherent in slurry diaphragm walls.

ADVANTAGES

- ▲ Cost-effective alternative for use in highly saturated soils or high ground water applications, unstable soils and confined or restricted job sites.
- ▲ Eliminates the need for costly dewatering systems inherent to conventional construction methods.
- ▲ Helps keep construction schedules on time, and in some cases accelerates construction schedules.
- ▲ Diaphragm walls provide both temporary and permanent load bearing support.
- ▲ Lateral ground movements with diaphragm walls are minimal in comparison with other conventional construction techniques.
- ▲ Ground water seepage is controlled or cut-off.
- ▲ Diaphragm walls can eliminate the need for additional underpinning or otherwise protect adjacent structures from settlement or lateral movements.
- ▲ Diaphragm walls significantly reduce construction time and street disruptions.
- ▲ Slurry diaphragm wall construction methods are virtually vibration free, making surrounding soils less prone to soil movements.
- ▲ Allows construction of multiple storied basements, underground garages and underground machinery vaults, subways and underground transit station areas having a high ground water table.
- ▲ Allows owners, engineers and contractors to reevaluate building sites formerly considered unworkable due to site, soil or ground water conditions.

Slurry Diaphragm Wall

Construction Process Overview



1 SITE CLEARING: The first step in the diaphragm wall construction process is site clearing, near surface obstruction removal and utility relocation. As a general rule, pre-excavation of obstructions (such as old foundations), and utility relocation, will usually be more economical than to attempt to dig through such obstructions by slurry clam and chisel techniques.

2 GUIDEWALLS: Once the site is cleared, the second step is to install a temporary guide wall along the perimeter of the diaphragm wall. The guide wall is a lightly reinforced footing placed on both sides of the diaphragm wall alignment. The guide wall is three to five feet in depth and serves to guide the slurry excavation tools during initial panel excavation, as well as to stabilize the upper soil strata.

3 PANEL EXCAVATION: With the guide walls in place, the third step is to proceed with panel excavation. The panel excavation sequence is to first dig a primary panel on either side of a secondary or closure panel. The panels are excavated under a full head of bentonite slurry, with a specially designed slurry clamshell bucket. The weight of the clamshell bucket is 8-10 tons, with the width and length of the bucket jaws dependent on panel design dimensions.

4 SLURRY: During the excavation process, the slurry level in the panel is kept close to the top of the guide wall. The slurry, due to a combination of forces, including a positive hydrostatic head, the filter cake at the soil liquid interface point, and the migration and gelling of the slurry in the soil, stabilizes the soil surrounding the panel excavation...to any depth in any soil.

5 MIXING PLANT: The bentonite slurry is prepared in a high shear, centrifugal mixing system to assure full hydration of the bentonite and water slurry. The Case mixing system includes high-speed pumps, storage tanks, screening and desanding equipment to maintain and control slurry properties during all phases of the excavation operation.

6 REINFORCING CAGE/BULKHEADS: Once primary panel excavation is completed, the fourth step is to place a steel reinforcing cage in the panel. The cage may contain blockouts and special detailing for floor keyways, sleeves for tiebacks and utility connections. Circular joint pipes (also known as bulkheads) are placed at each end of the primary panels to form the interconnecting key or joints between panels. The slurry is desanded to reduce the in-panel density of the slurry fluid such that the slurry can be easily displaced by the concrete.

7 CONCRETE PLACEMENT: Concrete placement is the fifth step. Tremie pipes are placed in each panel. A tremie pipe is a ten to twelve inch diameter pipe with a funnel at the top. The pipe (in sections) is placed from the top to the bottom of each panel through the rebar cage. Concrete is introduced into the tremie pipe. The concrete flows out the bottom of the tremie pipe at the bottom of the panel. As the concrete rises in the panel, the slurry is displaced and pumped off at the top of the panel and sent to storage tanks to be reused during future panel excavations. The tremie pipe is removed in ten foot sections as the concrete level rises. Concrete placement by the tremie pour method continues until the design top elevation of the diaphragm wall is reached.

8 JOINT PIPES: The sixth step is to remove the circular joint pipes at either end of the primary panels. The joint or key formed by the removal of the joint pipe is concave in dimension. The joint pipes are pulled within a few hours of completion of concrete placement before the concrete sets making it impossible to pull the pipe.

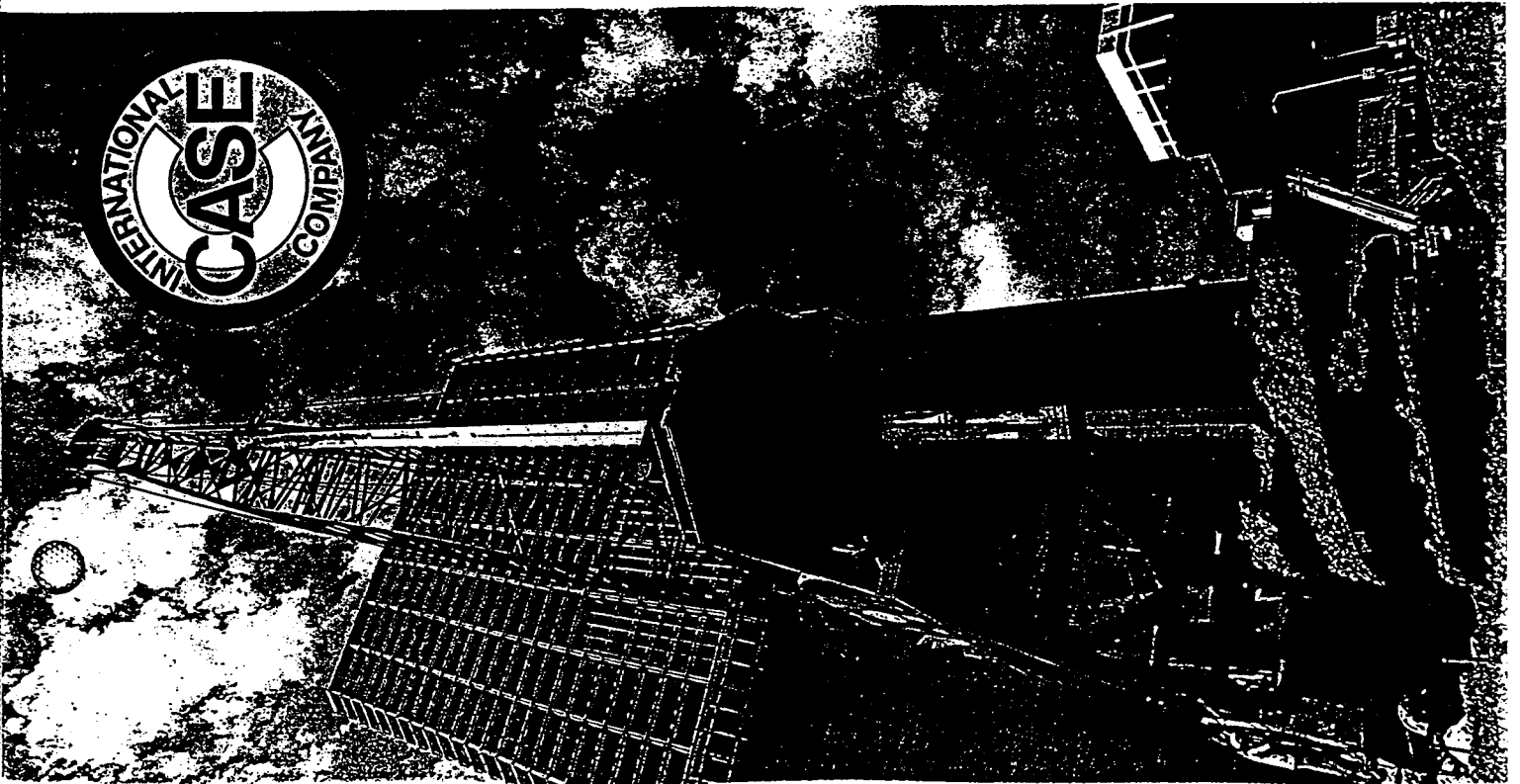
9 CLOSURE PANELS: The seventh step is to repeat the excavation, placement of rebar cage and tremie concreting process for the secondary or closure panel. The closure panel is the panel area between the two primary panels. The formed concave joints of the adjoining primary panels provide both the vertical excavation guide and, more importantly, the interconnecting joint key between primary and secondary panels. This sequence of excavating primary and then secondary panels is known as the alternating slurry panel construction process. It is repeated until the entire length of slurry diaphragm wall is completed.

10 GUIDE WALL REMOVAL & CAP BEAM: The last step in the slurry diaphragm wall process is to remove the guide wall and pour a cap beam. The cap beam helps to tie the panels together such that the overall foundation wall can best achieve a semi-rigid wall design.

Appendix C

Contractor Qualification

CASE INTERNATIONAL COMPANY
P.O. Box 40, Roselle, IL 60172



R E S U M E

NAME: Edward Jacobsen
Vice President

YEARS OF EXPERIENCE: 30

EDUCATION:

BS - Civil Engineering, 1959
University of Wisconsin

EXPERIENCE HISTORY:

1961 - 1982 Caisson Corporation
Northbrook, Illinois

1982 - Present Case International Company
Roselle, Illinois

ACCOMPLISHMENTS:

- Instrumental in diversifying company's activities into all areas of specialty underground construction, such as circulation drilling, rock drilling using roller bits and down-the-hole hammers, slurry walls, slurry cut-off trenches, driven piles, sheeting, shoring and tie-back anchors.
- Refined and improved bidding techniques, proposal formats and estimating procedures.
- Wrote, edited and assisted in production of 18 minute (16mm sound, color, some animation) movie showing sequenced installation of rock-bearing caissons.
- Developed cost-saving techniques for large-diameter shaft installations.
- Developed and negotiated "design-build" contracts for concrete slurry wall construction on 8 major building projects.
- Assisted in developing (now patented) system for self-contained circulation drilling in offshore/subsea conditions. This technique was subsequently applied to work performed for Dome Petroleum (Calgary, Alb.) in the Beaufort Sea.
- Established subsidiary company to do foundation work in the United Arab Emirates. Traveled extensively between U.S. and Persian Gulf area during time that foundation work was being performed in that area.

RESUME - EDWARD JACOBSEN

Page 2

- Negotiated offshore drilling project in Panama. Initiated use of specially designed collapsing jack-up platform to support drilling rig; project was located in remote, non-navigable portion of Chagres River.
- Conducted negotiations to establish foundation company in Lagos, Nigeria (General Foundation, Ltd.).
- Traveled to numerous foreign countries to visit job-sites and bid on foundation projects: Brazil, Honduras, Panama, Canada, Israel, Saudi Arabia, U.A.E.

MAJOR CONCRETE DIAPHRAGM WALL PROJECTS:

(Engineering, Estimating, Project Management Responsibility)

- | | |
|--|---------------|
| . Standard Oil Headquarters Building | - Chicago |
| . Health Science Building (Northwestern Univ.) | - Chicago |
| . Westinghouse Vocational High School | - Chicago |
| . One Magnificent Mile (Office & Condo) | - Chicago |
| . Wastewater Treatment Plant | - Hudson, WI |
| . One Westwood Office Building | - Los Angeles |
| . Wilshire-Westwood Office Building | - Los Angeles |

MEMBERSHIPS:

Illinois Society of Professional Engineers
National Society of Professional Engineers
American Society of Civil Engineers, Fellow
Contractors License: Utah, New Mexico
The Moles
Professional Engineer (P.E.) - State of Illinois
Served on Chicago Committee for High-Rise Buildings
Occasional guest lecturer at Chicago Loop College.

R E S U M E

NAME: Thomas Mushong
TITLE: General Superintendent
YEARS OF EXPERIENCE: 34

EXPERIENCE HISTORY:

1955 - 1964 Lake States Engineering Corporation
Des Plaines, Illinois
Operator, Field Superintendent

1965 - 1983 Caisson Corporation
Northbrook, Illinois
Field Superintendent, General Superintendent,
Vice President

1983 - Present Case International Company
Roselle, Illinois
Field Superintendent, General Superintendent.
Oversees the performance of all Field Superintendents.
Experienced in all phases of field operations, including
all types of caissons, driven piles, slurry trenches,
slurry walls and all other construction operations.

The following is a partial list of projects in which Thomas Mushong has participated in:

- Dome Petroleum, Beaufort Sea, Calgary, Alberta
Large diameter holes 18' X 100'
- Tennessee Tombigbee River, Columbus, Mississippi
Slurry cutoff trench 3' X 65' X 6500 L/F
- Deep Tunnel Project, Chicago, Illinois
Large diameter access shafts
- Transmission Line, Albany, New York
18 miles with rock foundations 9' diameter X 40' deep

RESUME - THOMAS MUSHONG

Page 2

Yellow Creek Nuclear Station, Iuka, Mississippi
Large diameter rock holes 8' diameter X 85' deep

Hudson River, Troy, New York
2' diameter rock holes, 20' rock sockets

State Office Building, Cleveland, Ohio
Deep caisson drilling 3'0" - 6'0" diameters X 200' deep

Dade County, Miami, Florida
Slurry drilling 4' diameter X 80' deep

Salt River Bridge, Phoenix, Arizona
Large diameter drilling 7'0" X 180' deep

Westwood Towers, Los Angeles, California
Large diameter drilling 6'0" X 170' deep for structural columns
61,000 S/F of 30" slurry diaphragm wall 100' deep

United Airlines, O'Hare Terminal Airport, Chicago, Illinois
3,500 L/F of 24" diaphragm wall

Browning Ferris Industries, Livingston, Louisiana
558,000 S/F soil/bentonite slurry trench

U.S. Army Corps of Engineers, Wilmington, North Carolina
477,000 S/F soil/bentonite slurry trench

Union Station/Main Shops & Yard, Los Angeles, California
286,000 S/F concrete diaphragm wall

Guest Quarters Hotel, Chicago, Illinois
41,700 S/F soil/bentonite slurry trench

U.S. Army Corps of Engineers, Lock & Dam #5, Shreveport, Louisiana
750,000 S/F soil/bentonite slurry trench

Shot Tower Station, Baltimore, Maryland
82,000 S/F concrete diaphragm wall

Sunnydale Facilities, San Francisco Clean Water Program
San Francisco, California
162,000 S/F concrete diaphragm wall

R E S U M E

THOMAS O'MALLEY
Project Manager

Years of Experience: 42

Education: 1948 St. Philip High School, Chicago, IL
1954-55 University of Illinois, Champaign-Urbana, IL

Experience: Mr. O'Malley has been involved in the foundation construction business since 1948 and came to Case International Company in 1954 as a Field Labor Foreman, responsible for labor coordination on caisson projects.

1981 - Present Case International Company
Roselle, Illinois

Serve as Project Manager for ongoing slurry wall projects. Estimating and researching slurry wall techniques and quality controlled methods of bentonite applications in cutoff and structural walls.

1969 - 1981 Gateway Contractors
Chicago, Illinois

Employed as a Structural & Reinforcing Ironworker.

1967 - 1969 American Bridge Company
Pittsburgh, Pennsylvania

Employed as a Structural Ironworker at the 1st National Bank, Chicago, Illinois.

1965 - 1967 Millgard Corporation
Livonia, Michigan

Opened Chicago office to represent them as Estimator and Project Manager.

1954 - 1965 Case Foundation Company
Roselle, Illinois

Employed as Labor Foreman. Appointed Job Superintendent over caisson installation in 1956. Appointed assistant to General Superintendent in 1962.

1953 - 1954 University of Illinois
Champaign-Urbana, Illinois

Completed 1 year in Civil Engineering.

1951 - 1953 Military Service.

1951 - 1948 United Engineers
McCook, Illinois

Employed as a laborer and was appointed Concrete Foreman after 6 months. Acquired a great deal of experience placing structural concrete in various building types as usually found at a chemical plant.

Forty-two years of combined field management experience and have attended welding, structural steel, reinforcing and post tensioned concrete courses to support field experiences.

R E S U M E

PAUL LECLERC

Assistant Project Superintendent

Education: 1977 Graduate, Elgin (Illinois) High School
1976-77 Elgin Community College

Experience: Mr. Leclerc has been involved in the foundation construction business since 1977 and came to Case International Company in 1983 as a Field Labor Foreman, responsible for labor coordination on caisson and slurry wall projects.

Appointed Assistant Project Superintendent 1985, specializing in major projects involving construction of slurry walls, slurry caissons, and slurry cut-off trenches.

Work History: 1977-1983 Caisson Corporation, Northbrook, Illinois
Labor Foreman for caisson and slurry wall projects.

Following are representative projects in which Mr. Leclerc has been involved:

Los Angeles Metro Rail Project Contracts A130 & A135
Los Angeles, CA

Concrete reinforced slurry wall 30" thick including 14 panels constructed under low overhead conditions and related guide walls and support system. Approximately 286,000 sq.ft. total wall area.

Salt River Project 230 KV Transmission Line
Pinnacle Peak to Papago Buttes
Phoenix, AZ

Large diameter caissons for Transmission Line structures along Salt River in Phoenix, AZ.

I-10 Salt River Bridge
Phoenix, AZ.

Large diameter caissons for Freeway Bridge replacement in Phoenix.

AT&T Corporate Center - Phase I
Chicago, IL.

Concrete reinforced slurry wall 30" wide, approximately 49,000 square feet with a maximum depth of 75 feet. Belled caissons 3 foot to 7.5 foot diameter shafts with 6 foot to 17.5 foot diameter bells, 90 feet deep.

RESUME - PAUL LECLERC

Page 2

Redwood Towers
Baltimore, MD

Twelve slurry caissons (8 interior and 4 exterior) ranging in diameter from 42 inches to 66 inches, 38 to 58 feet deep, installed from the roof of an existing 6-story parking structure.

Massachusetts General Hospital
Boston, Mass.

Deep slurry caissons with rock sockets.

Scholl Canyon Landfill
Spadra Landfill
Palos Verdes Landfill - Los Angeles, CA.
Los Angeles County Sanitation District

Cement/Bentonite slurry cut-off trenches for leachate barriers around landfills.

United Airlines Terminal Expansion
O'Hare Field - Chicago, IL.

Structural concrete slurry wall for below grade passenger service facilities. 30" thick wall, approximately 155,000 sq.ft. total wall area.

Sky Harbor International Airport - Terminal 4
Phoenix, AZ

380 large diameter drilled shafts by slurry displacement method.

New Waddell Dam
Phoenix, AZ

20" diameter drilled shaft secant wall in rock to 120 ft. depth, for water seepage below foundation of proposed earthfill dam.

7/91

R E S U M E

Jack Ross

25411 Columbia Bay Drive
Lake Villa, Illinois 60046

Education: High School
University of Illinois (1 year)

Experience: Mr. Ross has been involved in the foundation construction business since the late 1950's and was continuously employed with Case International Company for eight years and then worked an additional nineteen years with Case's major competitor, returning to Case International Company in 1983 when the competitor company was purchased by Case. With his vast knowledge in all areas of field operations and his experience in large-diameter drilling, off-shore drilling, slurry and circulation drilling, and slurry trench/wall construction, Mr. Ross has been called upon to superintend the more complicated, difficult and/or largest projects both domestic and foreign. Mr. Ross has served in all capacities of field management from shift supervisor, assistant superintendent, superintendent to general superintendent.

Following is a list of recent representative projects in which Mr. Ross has been involved:

1. Los Angeles Metro Rail Project Contracts A130 & A135 (Prime)
Los Angeles, California
Engineer: Southern California Rapid Transit District
General Contractor: Tutor-Saliba-Perin, JV

Concrete Reinforced Slurry Wall 14 Panels and Related Guidewalls
and Support System
286,000 sq.ft.
30" wide

2. Flagship Wharf, Building #197 (Subcontractor)
Charlestown Navy Yard, Charlestown, MA
General Contractor: Turner Construction Company

Concrete Reinforced Slurry Wall: 44,88 sq.ft.
30" wide
724 l.f.

3. Prudential Plaza (Subcontractor)

Chicago, IL

General Contractor: Turner Construction Company

44 Caissons

33 rock socketed and 11 hardpan
2'-3" to 10' dia. shafts with
5'-6" to 9'-6" dia. bells
80 ft. to 105 ft. deep.

Concrete Reinforced Slurry Wall:

30,000 sq. ft.
24" wide

4. AT&T Corporate Center - Phase I (Subcontractor)

Chicago, Illinois

General Contractor: Mayfair/Blount, Joint Venture

Concrete Reinforced Slurry Wall:

4900 Sq. Ft.
75 ft. deep

Belled Caissons:

2 ft. wide and 2.5 ft. wide
3 ft. to 7.5 ft. diameter shafts
6 ft. to 17.5 ft. diameter shafts
90 ft. deep

5. Maternal & Child Health Care Facility (Subcontractor)

The Evanston Hospital, Evanston, IL

General Contractor: Power Contracting & Engineering Corp.

Concrete Diaphragm Wall:

23,507 sq.ft.
40 to 70 ft. deep
30" wide

55 Caissons:

36" to 42" diameter shafts

Earth Retention System:

Steel sheet piling and related
bracing

6. Spadra Landfill Leachate Barrier System (Prime Contract)

Pomona, CA

Engineer: Los Angeles County Sanitation District

Cement/Bentonite Slurry Trench:

21,000 sq. ft.
2 ft. wide
15 to 80 ft. deep

7. Palos Verdes Landfill Leachate Barrier System (Prime Contract)
 Rolling Hills Estates, CA
 Engineer: Los Angeles County Sanitation District

Cement/Bentonite Slurry Trench: 45,000 sq.ft.
 2 ft. wide
 15 to 80 ft. deep

8. Scholl Canyon Landfill Leachate Barrier System (Prime Contractor)
 Pasadena, CA
 Engineer: Los Angeles County Sanitation District

Cement/Bentonite Slurry Trench: Collection System and Wells
 14,000 sq.ft.
 20 to 45 feet deep
 2 ft. deep

9. One Westwood Office Building (Subcontractor)
 Los Angeles, CA
 General Contractor: Turner Construction Company

Concrete Diaphragm Wall: 61,000 sq.ft.
 30" thick
 105 ft. deep

10. 7 World Trade Center (Subcontractor)
 New York City, NY
 Architect: Emery Roth & Sons

51 Caissons 30" to 36" diameter shafts
 70' deep
 80 tons per sq.ft.
 200 psi friction

11. One Magnificent Mile (Subcontractor)
 Chicago, IL
 General Contractor: Schal Associates

81 Caissons: 2'-6" to 7'-6" diameter shafts
 22' diameter bells
 75' deep
 25,000 psf bearing
 Concrete Diaphragm Wall: 45,000 sq. ft.
 52 ft. deep
 2 ft. wide

RESUME

NAME: Ken Metivier
Corporate Safety Director and Risk Manager

EDUCATION:

1943 Butler University - Indianapolis, Indiana
2 Years Undergraduate - Engineering

SAFETY TRAINING: Graduate, OSHA Training Institute - Rosemont, Illinois
Certified Construction Safety Training Instructor

Graduate, Associated General Contractors of America
- Washington, DC
Construction Supervisors Safety Training Program

PROFESSIONAL MEMBERSHIPS: American Society of Safety Engineers
Construction Safety Association of America
Past Member of Safety Committee - Builders Association of Chicago

WORK HISTORY AND EXPERIENCE:

1989 - Present The Case Group
Roselle, Illinois

Joined the parent company of J.H. Pomeroy & Co., Inc., Case International Company and Pomco Associates, Inc. as Corporate Safety Director/Risk Manager. Develop and implement safety programs for all operations including: general contracting; caisson drilling; slurry and diaphragm walls; pile driving and retention systems; manufacturing and erection of pre-cast concrete products. Provide technical support and assistance for safety to all management and supervisory personnel. Conduct on-site safety inspections, and safety training programs. Manage insurance programs.

1972 - 1989 Morse/Diesel, Inc.
Chicago, Illinois

Joined this national construction management firm as Safety Engineer on construction of the 110-story Sears Tower. Then as Vice President/Corporate Safety Director developed and implemented corporate safety programs for construction of major projects of nearly every type nationwide at an average annual volume of \$750 million. Supervised three regional safety directors to provide a complete range of safety services to management and supervisory personnel at 12 branch office locations.

RESUME

Ken Metivier

Corporate Safety Director and Risk Manager

Page 2

1970 - 1972 J.A. Jones Construction Company
Chicago, Illinois

Joined this national general contractor as Safety Manager on construction of a 45-story office building for C.N.A. Insurance Company in Chicago. Full time, on-site responsibility for implementation of safety program.

1959 - 1970 Harvey Wrecking Company
Chicago, Illinois

Joined this firm regarded as the foremost building demolition contractor in the Mid-West as a Superintendent. Responsibilities included on-site supervision of demolition operations, estimating, and salvage sales. Supervised demolition of hundreds of buildings ranging in type from single-family residences to major high-rise buildings in downtown Chicago, and dismantlement of large industrial plants.

1952 - 1959 Modern Age Construction Company
St. Louis, Missouri

Joined this local general contractor specializing in residential construction as a carpenter, and progressed to Carpenter Foreman, and Superintendent. Project types ranged from renovations to construction of residential subdivisions up to 100 homes.

1946 - 1952 United States Steel Supply Company
Chicago, Illinois and St. Louis, Missouri

Joined the Chicago headquarters office of this U.S. Steel Corp. subsidiary as a sales trainee. Then as sales representative in the St. Louis district office responsible for direct sales to industrial users of steel products in a 5 state sales territory.

Appendix D

API Specification 13A Specification for Drilling Fluid Materials

Specfication for Drilling-Fluid Materlals

API SPECIFICATION 13A (SPEC 13A)
FOURTEENTH EDITION, AUGUST 1, 1991

American Petroleum Institute
1220 L Street, Northwest
Washington, DC 20005



**SECTION 4
BENTONITE**

4.1 Description

a. Drilling grade bentonite is a naturally occurring clay containing the clay mineral smectite. It may also contain accessory minerals such as quartz, mica, feldspar, and calcite.

b. Drilling grade bentonite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 4.1 and represents the product produced and is controlled by the manufacturer.

**TABLE 4.1
BENTONITE PHYSICAL REQUIREMENTS**

Requirement	Specification
Suspension Properties	
Viscometer Dial Reading at 600 rpm	30, minimum
Yield Point/Plastic Viscosity Ratio	3, maximum
Filtrate Volume	15.0 cm ³ , maximum
Residue greater than 75 micrometers	4.0 wt percent, maximum
Moisture	10.0 wt percent, maximum

SUSPENSION PROPERTIES

4.2 Equipment

- a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)
- b. Balance: precision of ±0.01 g
- c. Mixer: 11,000 ±300 rpm under load with single sine-wave impeller approximately 1 inch (25 mm) in diameter (e.g., Multimixer Model 9B with 9B29X impeller blades or equivalent as shown in Fig. 2.1, mounted flash side up). The impeller shall be replaced when it weighs a minimum of 5.1 g. The original weight is about 5.5 g. New blades will be weighed prior to installation. Mixer speed under sample loading shall be determined and documented once every 90 days unless manufacturer has documented objective evidence to extend calibration time.
- d. Container: approximate dimensions — 7 inches (180 mm) deep, 3 1/4 inch (97 mm) ID top, 2 1/4 inch (76 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)
- e. Spatula
- f. Motor-Driven Direct Indicating Viscometer: as referenced in API Spec 18A, Specifications for Oil Well Drilling Fluid Materials, Section 1.8a.
- g. Filter press: as described in API Spec 18A, Specifications for Oil Well Drilling Fluid Materials, Section 1.8b.
- h. Graduated cylinder (TD): 500 ±5 cm³

i. Deionized or distilled water

j. Container: about 500-cm³ with lid

k. Timers: two interval, mechanical or electrical, precision of ±0.1 minute

l. Graduated cylinder (TD): 10 ±0.1 cm³

4.3 Procedure — 600 rpm Reading, Plastic Viscosity, Yield Point

a. Prepare a suspension of the bentonite. Add 22.5 ±0.01 g of clay (as received) to 350 ±5 cm³ of deionized water while stirring on the mixer.

b. After stirring five ±0.5 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any bentonite adhering to container walls. Be sure all bentonite clinging to the spatula is incorporated into the suspension.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

d. Age the bentonite suspension up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

e. After aging bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 ±0.5 minutes.

f. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C).

4.4 Calculation — Plastic Viscosity and Yield Point

$$\text{Plastic viscosity (cP)} = \left[\begin{matrix} 600 \text{ rpm} \\ \text{reading} \end{matrix} \right] - \left[\begin{matrix} 300 \text{ rpm} \\ \text{reading} \end{matrix} \right] \quad (a)$$

$$\text{Yield Point (lb/100 ft}^2) = \left[\begin{matrix} 300 \text{ rpm} \\ \text{reading} \end{matrix} \right] - \text{Plastic viscosity (b)}$$

$$\text{Yield point/Plastic viscosity Ratio} = \frac{\text{Yield point [Eq 4.4(b)]}}{\text{Plastic viscosity [Eq 4.4(a)]}} \quad (c)$$

Record calculated values for plastic viscosity, yield point, and yield point/plastic viscosity ratio.

4.5 Procedure — Filtrate Volume

a. Recombine all of the suspension as prepared and tested in Par. 4.3 and stir in container for 1 ±0.5 minute on the mixer.

b. Pour the suspension into filter press cell. Before adding suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. Pour the mud to within about 1/2 inch (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place filter cell in frame and close relief valve. Place a container under the drain tube.

c. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on cell at 100 ±5 psi. Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen, or helium.

d. At 7.5 ±0.1 minutes on the first timer remove the container and any adhering liquid on the drain tube and discard. Place the dry 10-cm³ graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

4.6 Calculation -- Filtrate Volume

a. Calculate the filtrate volume of the clay suspension as:

$$\text{Filtrate volume} = 2 \text{ times filtrate volume, cm}^3 \text{ collected between 7.5 and 30 minutes} \quad (a)$$

Record calculated filtrate volume.

RESIDUE GREATER THAN 75 MICROMETERS

4.7 Equipment

- a. Oven: regulated to 220 ±5°F (105 ±3°C)
- b. Sodium hexametaphosphate (CAS #10124-58-8)
- c. Balance: precision of ±0.01 g
- d. Mixer: 11,000 ±800 rpm under load with single sine-wave impeller approximately 1 inch (25 mm) in diameter (e.g., Multimixer Model 9B with 9B29X impeller blades or equivalent as shown in Fig. 21, mounted flash side up). The impeller shall be replaced when it weighs a minimum of 5.1 g. The original weight is about 5.5 g. New blades will be weighed prior to installation. Mixer speed under sample loading shall be determined and documented once every 90 days unless manufacturer has documented objective evidence to extend calibration time.
- e. Container: approximate dimensions -- 7 inches (180 mm) deep, 2 1/8 inch (87 mm) ID top, 3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)
- f. Spatula
- g. A 75-micrometer sieve: approximate dimensions -- 3 inch (76 mm) diameter and 2.5 inches (63 mm) from top of frame to wire cloth
- h. Spray nozzle: 1/2 TT body (Spraying Systems Company, No. TG 8.5 tip with 1/2 TT body or equivalent) attached to water line with 90 degree elbow as shown in Fig. 22
- i. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)
- j. Evaporating dish
- k. Wash bottle

4.8 Procedure

- a. Weigh 10 ±0.01 g of bentonite.
- b. Add the weighed bentonite sample to approximately 350 cm³ of water containing about 0.2 g of sodium hexametaphosphate.
- c. Stir suspension on the mixer for 30 ±1 minutes.
- d. Transfer the sample to the sieve. Use the wash bottle to remove all material from the container to sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ±7 kPa) from the spray nozzle for two minutes ±15 seconds. While washing, allow the elbow bend of the nozzle to rest on the rim of the sieve and move the spray of water repeatedly over the sample.
- e. Transfer the residue from the sieve to a tared evaporating dish.
- f. Dry the residue in the oven to a constant weight. Record residue weight and total drying time.

4.9 Calculation

$$\text{Residue, \% greater than 75 micrometers} = 100 \left(\frac{\text{Weight of residue, g}}{\text{Weight of sample, g}} \right) \quad (a)$$

Record calculated value.

MOISTURE

4.10 Equipment

- a. Oven: regulated to 220 ±5°F (105 ±3°C)
- b. Balance: precision of ±0.01 g
- c. Evaporating dish
- d. Spatula
- e. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant or equivalent

4.11 Procedure

- a. Weigh 10 ±0.01 g of bentonite sample into a tared evaporating dish.
- b. Dry sample in the oven to a constant weight. Record total drying time.
- c. Cool to room temperature in desiccator.
- d. Reweigh the evaporating dish containing the dry bentonite. Record weight.

4.12 Calculation

$$\text{Moisture, wt percent} = 100 \left[\frac{\text{wt of orig sample, g} - \text{wt dry sample, g}}{\text{wt orig sample, g}} \right] \quad (a)$$

Record calculated value.

*Correction factor to be determined and documented by manufacturer.

**SECTION 5
NONTREATED BENTONITE**

5.1 Description

a. Drilling grade nontreated bentonite is dried and ground, but not chemically-treated clay, composed principally of the mineral smectite. It may also contain accessory minerals such as quartz, mica, feldspar, and calcite.

b. Drilling grade nontreated bentonite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 5.1 and represents the product produced and is controlled by the manufacturer.

**TABLE 5.1
NONTREATED BENTONITE PHYSICAL
REQUIREMENTS**

Requirement	Specification
Suspension Properties	
Yield Point/Plastic Viscosity Ratio	1.5, maximum
Dispersed Plastic Viscosity	10 cp, minimum
Dispersed Filtrate Volume	12.5 cm ³ , maximum

SUSPENSION PROPERTIES

5.2 Equipment

a. Balance: precision of 0.01 g

b. Mixer: 11,000 ± 300 rpm under load with single sine-wave impeller approximately 1 inch (25 mm) in diameter (e.g., Multimixer Model 9B with 9B29X impeller blades or equivalent as shown in Fig. 2.1, mounted flash side up). The impeller shall be replaced when it weighs a minimum of 5.1 g. The original weight is about 5.5 g. New blades will be weighed prior to installation. Mixer speed under sample loading shall be determined and documented once every 90 days unless manufacturer has documented objective evidence to extend calibration time.

c. Container: approximate dimensions — 7 inches (180 mm) deep, 3 1/8 inch (97 mm) ID top, 2 1/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

d. Motor-Driven Direct Indicating Viscometer: as referenced in API Spec 18A, Specifications for Oil Well Drilling Fluid Materials, Section 1.8a.

e. Filter press: as referenced in API Spec 18A, Specifications for Oil Well Drilling Fluid Materials, Section 1.8b.

f. Spatula

g. Thermometer: 32-220 ± 1°F (0-105 ± 0.5°C)

h. Deionized or distilled water

i. Graduated cylinder (TD): 500 ± 5 cm³

j. Container: about 500-cm³ with lid

k. Syringe or pipette: 5 ± 0.01 cm³

l. Sodium hexametaphosphate (CAS #10124-56-8) solution: 10 ± 0.5% by wt

m. Timers: two interval, mechanical or electrical, precision of ± 0.1 minute

n. Graduated cylinder (TD): 10 ± 0.01 cm³

5.3 Procedure — Yield Point/Plastic Viscosity Ratio

a. Prepare a suspension of nontreated bentonite. Add 25 ± 0.01 g of clay (as received) to 350 ± 5 cm³ deionized water while stirring on the mixer.

b. After stirring five ± 0.5 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any bentonite adhering to container walls. Be sure all bentonite clinging to the spatula is incorporated into the suspension.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ± 1 minutes.

d. Age the bentonite suspension up to 16 hours in a sealed container at room temperature. Record storage temperature and storage duration.

e. After aging bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 ± 0.5 minutes.

f. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ± 2°F (25 ± 1°C).

5.4 Calculation — Plastic Viscosity and Yield Point

$$\text{Plastic viscosity (cP)} = \frac{600 \text{ rpm}}{\text{reading}} - \frac{300 \text{ rpm}}{\text{reading}} \quad (a)$$

$$\text{Yield point lb/100 ft}^2 = \frac{300 \text{ rpm}}{\text{reading}} - \text{Plastic viscosity} \quad (b)$$

$$\text{Yield point/Plastic viscosity Ratio} = \frac{\text{Yield point [Eq 5.4(b)]}}{\text{Plastic viscosity [Eq 5.4(a)]}} \quad (c)$$

Record calculated values for plastic viscosity, yield point, and yield point/plastic viscosity ratio.

5.5 Procedure — Dispersed Plastic Viscosity

a. Recombine all of the bentonite suspension as prepared and tested in Par. 5.3 and stir in container for 1 ± 0.5 minute on the mixer.

b. Add 5 ± 0.1 cm³ of a 10% solution of sodium hexametaphosphate to the suspension and stir for 3 ± 0.5 minutes on the mixer.

c. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ± 2°F (25 ± 1°C).

d. Calculate and record the dispersed plastic viscosity as per Par. 5.4.a.

5.6 Procedure — Dispersed Filtrate Volume

a. Recombine all of the suspension as prepared and tested in Par. 5.5 and stir in the container for 1 ± 0.5 minute on the mixer.

b. Pour the suspension into filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or

worn. Pour the suspension to within about ¼ inch (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place filter cell in frame and close relief valve. Place a container under the drain tube.

c. Set one timer for 7.5 ± 0.1 minutes and the second timer for 30 ± 0.1 minutes. Start both timers and adjust pressure on cell at 100 ± 5 psi. Both of these steps should be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen, or helium.

d. At 7.5 ± 0.1 minutes on the first timer remove the container and any adhering liquid on the drain tube and discard. Place dry 10-cm³ graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of its filtrate collected.

5.7 Calculation — Dispersed Filtrate Volume

a. Calculate the dispersed filtrate volume of the clay suspension as:

$$\text{Filtrate volume} = \frac{2 \text{ times filtrate volume, cm}^3 \text{ collected between 7.5 and 30 minutes}}{\text{}} \quad (a)$$

Record calculated filtrate volume.

CASE INTERNATIONAL CO.

of pages 6

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Appendix E

API (RP 13B)

**Recommended Practice-
Standard Procedure for Field Testing Drilling Fluids**

Recommended Practice Standard Procedure for Field Testing Drilling Fluids

RECOMMENDED PRACTICE 13B (RP 13B)
TWELFTH EDITION, SEPTEMBER 1, 1988

American Petroleum Institute
1220 L Street, Northwest
Washington, DC 20005



Issued by
AMERICAN PETROLEUM INSTITUTE
Production Department

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Note:

This edition supersedes the Eleventh Edition, dated May 1, 1985. This edition includes revisions adopted at the 1987 API Standardization Conference and subsequently approved by letter ballot.

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RECOMMENDED PRACTICE STANDARD PROCEDURE FOR FIELD TESTING DRILLING FLUIDS

Foreword

a. This recommended practice is under the jurisdiction of the API Committee on Standardization of Drilling Fluid Materials.

b. The purpose of this recommended practice is to provide standard procedures for the testing of liquid drilling fluids. It is not a detailed manual on mud control procedures. It should be remembered that the agitation history and temperature of testing have a profound effect on mud properties.

c. This recommended practice is organized to follow the tests as listed on the API Drilling Mud Report form (API RP 13G, Second Edition, May 1982). Additional tests are given in the Appendix of this recommended practice.

d. Metric "SI" unit equivalents have been included in

this publication in parentheses following the U.S. customary units.

e. Additional publications under jurisdiction of this committee: Spec 13A, Specification for Oil-Well Drilling Fluids Materials, covers specifications and test procedures for barite, weight material, bentonite, non-treated bentonite, attapulgite, sepiolite, starch and technical-grade low viscosity CMC.

Bul. 13C, Bulletin on Drilling Fluids Processing Equipment; Bull. 13D, Bulletin on the Rheology of Oil-Well Drilling Fluids; RE 13E, Recommended Practice for Shale Shaker Screen Cloth Designation; RP 13G, Recommended Practice for Drilling Mud Report Form; RP 13I, Recommended Practice for Laboratory Testing Drilling Fluids; RP 13H, Recommended Practice for Drilling Fluid Bioassays; RP 13J, Recommended Practice for Testing Heavy Brines; RP 13K, Recommended Practice for Chemical Analysis of Barite.

WATER BASE DRILLING FLUIDS

SECTION 1 MUD WEIGHT (DENSITY)

1.1 Description This test procedure is a method for determining the weight of a given volume of liquid. Mud weight may be expressed as pounds per gallon (lb/gal), pounds per cubic foot (lb/ft³), grams per cubic centimeter (g/cm³), or kilograms per cubic meter (kg/m³).

1.2 Equipment

a. Any instrument of sufficient accuracy to permit measurement within ±0.1 lb/gal (or 0.5 lb/ft³, 0.01 g/cm³, 10 kg/m³) may be used. The mud balance (Fig. 1.1 and 1.2) is the instrument generally used for mud weight determinations. The mud balance is designed such that the mud cup, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (Attachments for extending the range of the balance may be used when necessary.)

b. Thermometer: 32-220°F (0-105°C)

1.3 Procedure

a. The instrument base should be set on a flat, level surface.

b. Measure the temperature of the mud and record on the Drilling Mud Report form.

c. Fill the clean, dry cup with mud to be tested; put the cap on the filled mud cup and rotate the cap until it is firmly seated. Insure that some of the mud is expelled through the hole in the cap in order to free any trapped air or gas (see Appendix D for Air Removal).

d. Holding cap firmly on mud cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

e. Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the center line.

f. Read the mud weight at edge of the rider toward the mud cup. Make appropriate corrections when a range extender is used.

1.4 Procedure-Calibration The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/ft³ (1000 kg/m³) at 70 F (21 C). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

1.5 Calculation

a. Report the mud weight to the nearest 0.1 lb/gal or 0.5 lb/ft³ (0.01 g/cm³, 10 kg/m³).

b. To convert the reading to other units, use the following:

$$\text{Density} = \text{g/cm}^3 = \frac{\text{lb/ft}^3}{62.43} = \frac{\text{lb/gal}}{8.345} \quad (a)$$

$$\text{kg/m}^3 = (\text{lb/ft}^3) (16) = (\text{lb/gal}) (120) \quad (b)$$

$$\text{Mud gradient, } \frac{\text{psi}}{\text{ft}} = \frac{\text{lb/ft}^3}{144} \cdot \frac{\text{lb/gal}}{19.24} \cdot \text{or} \cdot \frac{\text{kg/m}^3}{2309} \quad (c)$$

TABLE 1.1
DENSITY CONVERSION

pounds per gallon (lb/gal)	pounds per cubic foot (lb/ft ³)	grams per cubic centimeter (g/cm ³)*	kilograms per cubic meter (kg/m ³)
6.5	48.6	0.78	780
7.0	52.4	0.84	840
7.5	56.1	0.90	900
8.0	59.8	0.96	960
8.3	62.3	1.00	1000
8.5	63.6	1.02	1020
9.0	67.3	1.08	1080
9.5	71.1	1.14	1140
10.0	74.8	1.20	1200
10.5	78.5	1.26	1260
11.0	82.3	1.32	1320
11.5	86.0	1.38	1380
12.0	89.8	1.44	1440
12.5	93.5	1.50	1500
13.0	97.2	1.56	1560
13.5	101.0	1.62	1620
14.0	104.7	1.68	1680
14.5	108.5	1.74	1740
15.0	112.5	1.80	1800
15.5	115.9	1.86	1860
16.0	119.7	1.92	1920
16.5	123.4	1.98	1980
17.0	127.2	2.04	2040
17.5	130.9	2.10	2100
18.0	134.6	2.16	2160
18.5	138.4	2.22	2220
19.0	142.1	2.28	2280
19.5	145.9	2.34	2340
20.0	149.6	2.40	2400
20.5	153.3	2.46	2460
21.0	157.1	2.52	2520
21.5	160.8	2.58	2580
22.0	164.6	2.64	2640
22.5	168.3	2.70	2700
23.0	172.1	2.76	2760
23.5	175.8	2.82	2820
24.0	179.5	2.88	2880

*Same as specific gravity (sg).

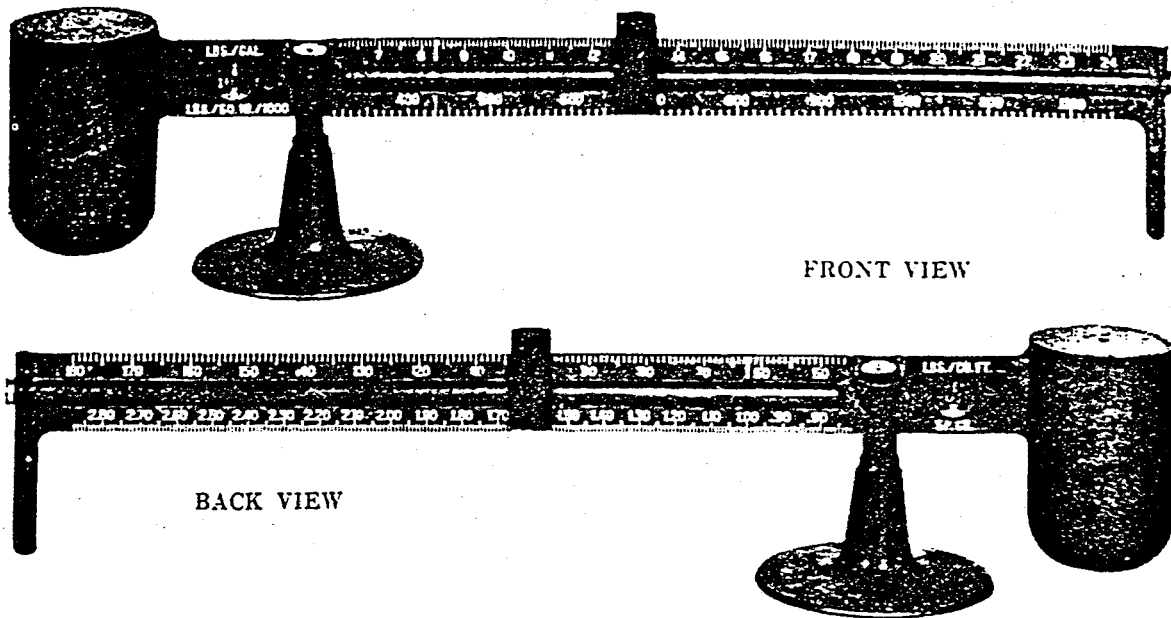


FIG. 1.1
MUD BALANCE

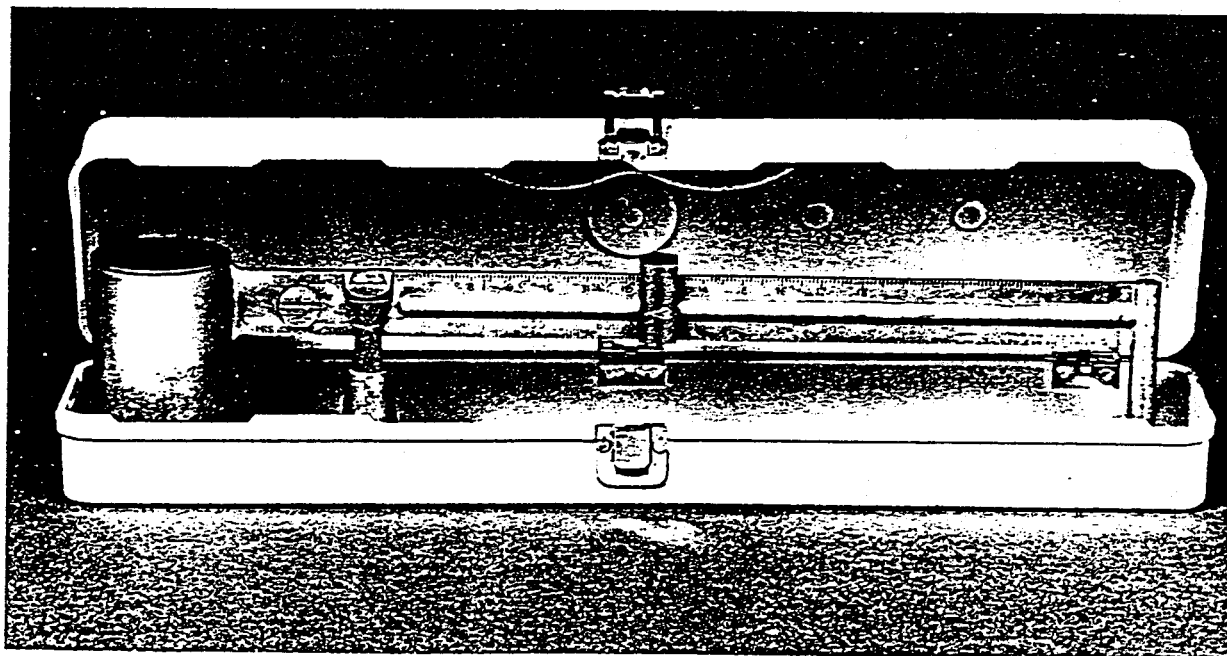


FIG. 1.2
MUD BALANCE AND CASE

SECTION 2 VISCOSITY AND GEL STRENGTH

2.1 Description

a. The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

1. Marsh funnel — a simple device for indication of viscosity on a routine basis.
2. Direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

b. Viscosity and gel strength are measurements that relate to the flow properties of muds. The study of deformation and flow of matter is rheology. An in-depth discussion of rheology is made in API Bulletin 13D: *The Rheology of Oil-Well Drilling Fluids*.

MARSH FUNNEL

2.2 Equipment A Marsh funnel (see Fig. 2.1) is calibrated to outflow one quart (946 cm³) of fresh water at a temperature of 70°F ± 5 (21°C ± 3) in 26 seconds ± 0.5. A graduated cup is used as a receiver.

a. Marsh funnel

Specifications

Funnel Cone

- Length 12.0 in. (305 mm)
- Diameter 6.0 in. (152 mm)
- Capacity to bottom of screen 1500 cm³

Orifice

- Length 2.0 in. (50.8 mm)
- Inside Diameter 3/16 in. (4.7 mm)

Screen

- Has 1/16 in. (1.6 mm) openings and is fixed at a level 3/4 in. (19.0 mm) below top of funnel.

b. Graduated cup: one-quart

c. Stopwatch

d. Thermometer: 32-220°F (0-105°C)

2.3 Procedure

a. Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.

b. Remove finger and start stopwatch. Measure the time for mud to fill to one-quart (946 cm³) mark of the cup.

c. Measure temperature of fluid in degrees F (C).

d. Report the time to nearest second as Marsh funnel viscosity. Report the temperature of fluid to nearest degree F (C).

DIRECT-INDICATING VISCOMETER

2.4 Equipment

a. Direct-indicating viscometers are rotational types of instruments powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity (rpm). The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob.

Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 rpm and 600 rpm.

Specifications — Direct-indicating viscometer

Rotor sleeve

- Inside diameter 1.450 in. (36.83 mm)
- Total length 3.425 in. (87.00 mm)
- Scribed line 2.30 in. (58.4 mm)
above the bottom of sleeve.

Two rows of 1/8 in. (3.18 mm) holes spaced 120 deg (2.09 radians) apart, around rotor sleeve just below scribed line.



FIG. 2.1
MARSH FUNNEL AND CUP

Bob

Diameter.....	1.358 in. (34.49 mm)
Cylinder length.....	1.496 in. (38.00 mm)
Bob is closed with a flat base and a tapered top.	
Torsion spring constant	
386 dyne-cm degree deflection	
Rotor speeds	
High speed.....	600 rpm
Low speed.....	300 rpm

The following are types of viscometers used to test drilling fluids:

1. Hand-cranked instrument (Fig. 2.2) has speeds of 300 rpm and 600 rpm. A knob on the hub of the speed-change lever is used to determine gel strength.
2. The 12-volt, motor driven instrument (Fig. 2.3) also has speeds of 300 and 600 rpm. A governor-release switch permits high shearing before measurement, and a knurled hand-wheel is used to determine gel strength.
3. The 115-volt instrument (Fig. 2.4) is powered by a two-speed synchronous motor to obtain speeds of 3, 6, 100, 200, 300 and 600 rpm. The 3-rpm speed is used to determine gel strength.
4. The variable speed 115-volt or 240-volt instrument (Fig. 2.5) is motor powered to obtain all speeds between 1 and 625 rpm. The 3-rpm speed is used to determine gel strength.

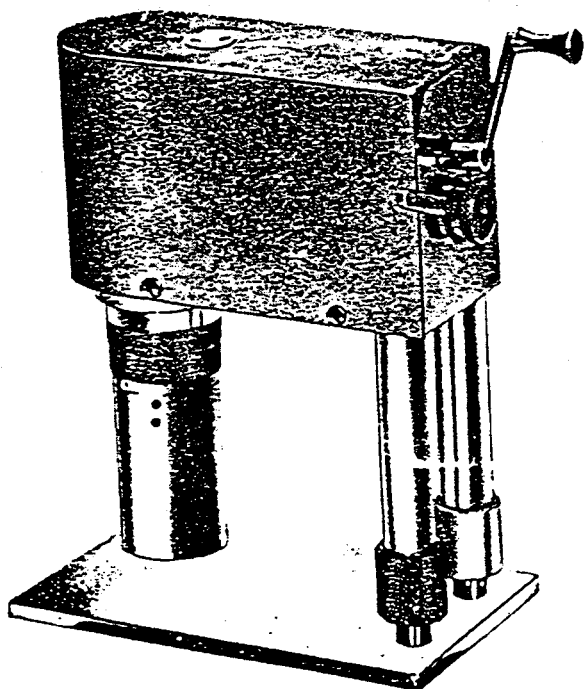


FIG. 2.2
HAND-CRANK VISCOMETER

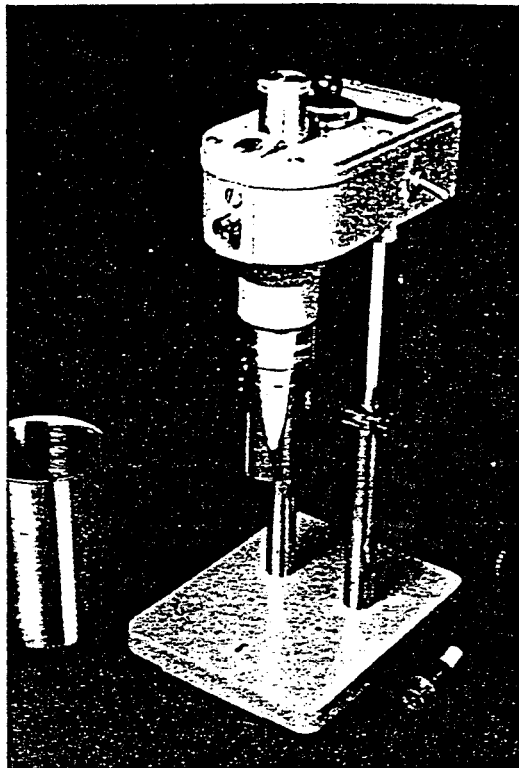


FIG. 2.3
12-VOLT MOTOR-DRIVEN VISCOMETER

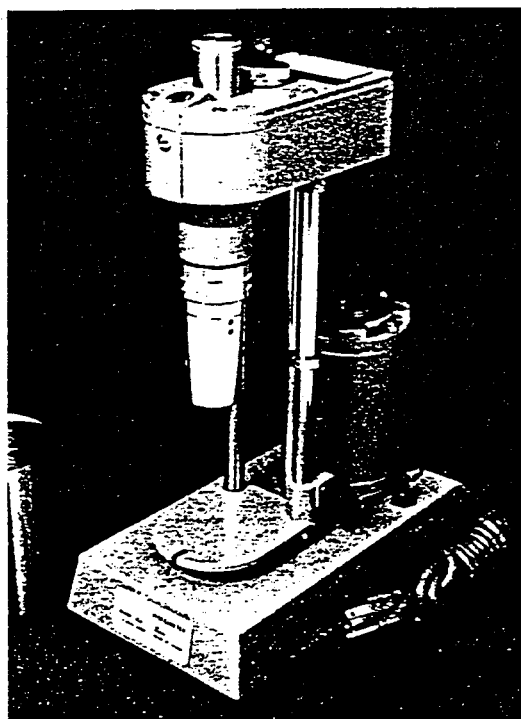
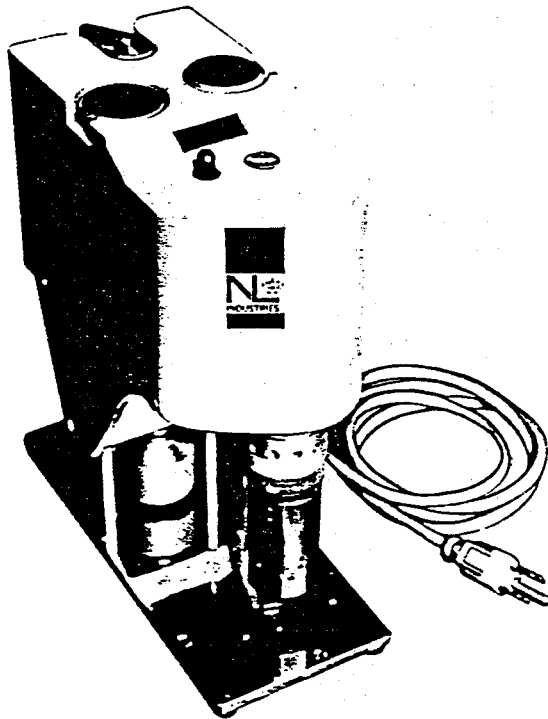


FIG. 2.4
115-VOLT MOTOR-DRIVEN VISCOMETER

- b. Stopwatch
- c. Suitable container, e.g., the cup provided with the viscometer.
- d. Thermometer: 32-220°F (0-105°C)

2.5 Procedure

CAUTION: *Maximum operating temperature is 200°F (93°C). If fluids above 200°F (93°C) are to be tested, a solid metal bob or a hollow metal bob, with a completely dry interior, should be used. Liquid trapped inside a hollow bob may vaporize when immersed in high temperature fluid and cause the bob to explode.*



a. Place a sample in container and immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay (within five minutes, if possible) and at a temperature as near as practical to that of the mud at the place of sampling (not to differ more than 10°F (6°C)). The place of sampling should be stated on the report.

b. Record the temperature of the sample.

c. With the sleeve rotating at 600 rpm, wait for dial reading to reach a steady value (the time required is dependent on the mud characteristics). Record the dial reading for 600 rpm.

d. Shift to 300 rpm and wait for dial reading to reach steady value. Record the dial reading for 300 rpm.

e. Stir drilling fluid sample for 10 seconds at high speed.

f. Allow drilling fluid sample to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a 3-rpm speed, the maximum reading attained after starting rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10 sec gel) in lb/100 ft² (Pa).

g. Restir the drilling fluid sample at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in Par. 2.5f and report the maximum reading as 10 minute gel in lb/100 ft² (Pa).

2.6 Calculation

$$\text{Plastic Viscosity, cp} = \left[\begin{matrix} 600 \text{ rpm} \\ \text{reading} \end{matrix} \right] - \left[\begin{matrix} 300 \text{ rpm} \\ \text{reading} \end{matrix} \right] \quad (a)$$

$$\text{Yield Point, lb/100 ft}^2 = \left[\begin{matrix} 300 \text{ rpm} \\ \text{reading} \end{matrix} \right] - \text{Plastic Viscosity} \quad (b)$$

$$\text{Apparent Viscosity, cp} = \frac{600 \text{ rpm reading}}{2} \quad (c)$$

SECTION 3 FILTRATION

3.1 Description

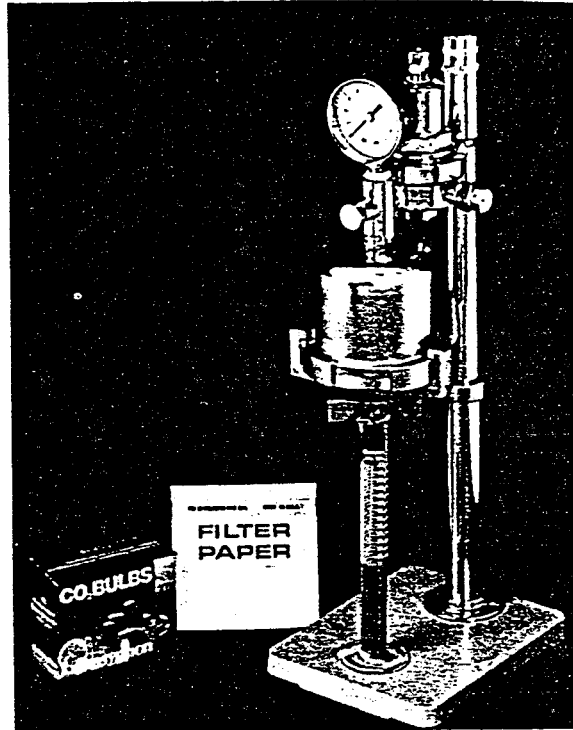
a. Measurement of the filtration and wall-cake-building characteristics of a mud are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate.

b. These characteristics are affected by the types and quantities of solids in the fluid and their physical and chemical interactions, which, in turn, are affected by temperature and pressure. Therefore, tests are run at both low-pressure, low-temperature and high-pressure/high-temperature, and each requires different equipment and techniques.

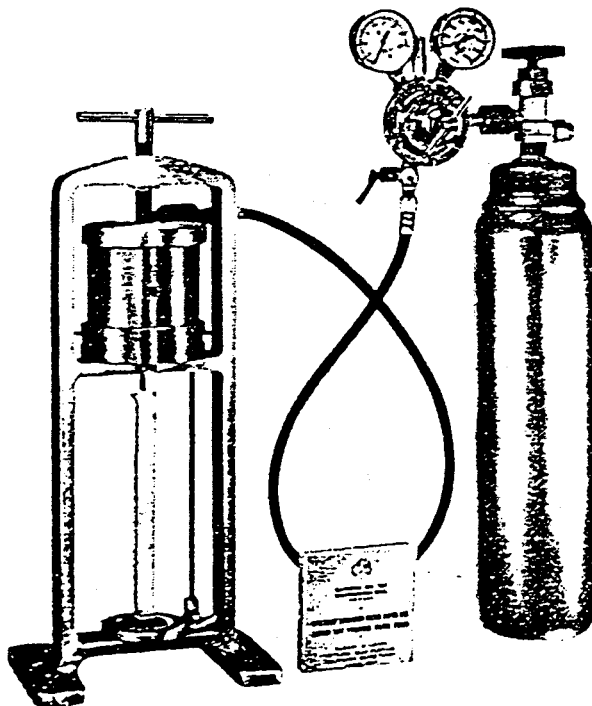
LOW-TEMPERATURE/LOW-PRESSURE TEST

3.2 Equipment — Low-Temperature/Low-Pressure Test

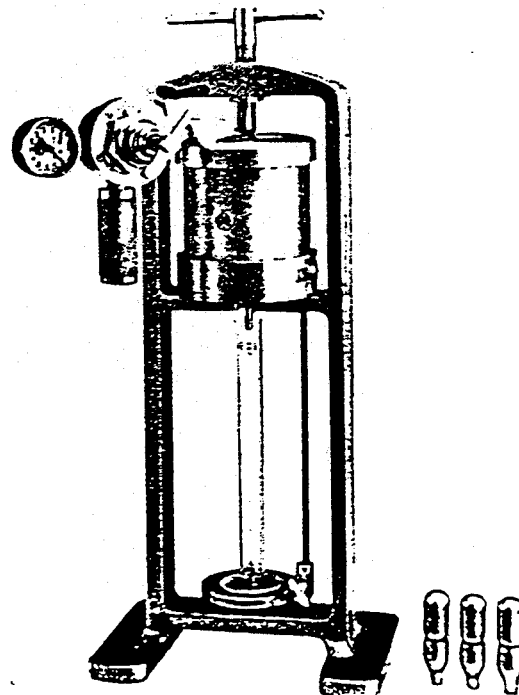
a. A filter press consists mainly of a cylindrical mud cell having an inside diameter of 3 in. (76.2 mm) and a height of at least 2.5 in. (64.0 mm). This chamber is made of materials resistant to strongly alkaline solutions, and is so fitted that a pressure medium can be conveniently admitted into, and bled from the top. Arrangement is also such that a sheet of 9 cm filter paper can be placed in the bottom of the chamber just above a suitable support. The filtration area is 7.1 in.² \pm 0.1 (4580 mm² \pm 60). Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets, and the entire assembly supported by a stand. Fig. 3.1 and 3.2 depict standard units by different manufacturers.



With CO₂ Cartridge Pressurization
FIG. 3.1
FILTER PRESS



With Nitrogen Pressurization



With CO₂ Cartridge Pressurization

FIG. 3.2
FILTER PRESS

Pressure can be applied with any nonhazardous fluid medium, either gas or liquid. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midget pressure cartridges or means for utilizing hydraulic pressure.

To obtain correlative results, one thickness of the proper 90-mm filter paper, Whatman No. 50, S&S No. 576, or equivalent, must be used.

NOTE: *The mini-press or half-area press does not directly correlate with the results obtained when using the standard sized press.*

b. Timer: 30-minute interval

c. Graduated cylinder (TC): 10-cm³ or 25-cm³

3.3 Procedure — Low-Temperature/Low-Pressure Test

a. Be sure each part of the cell, particularly the screen, is clean and dry, and that the gaskets are not distorted or worn. Pour the mud sample into the cell to within ½ in. (13 mm) to top (to minimize CO₂ contamination of filtrate), and complete the assembly with the filter paper in place.

b. Place a dry graduated cylinder under the drain tube to receive the filtrate. Close the relief valve and adjust the regulator so that a pressure of 100 psi ±5 (690 kPa ±35) is applied in 30 seconds or less. The test period begins at the time of pressure application.

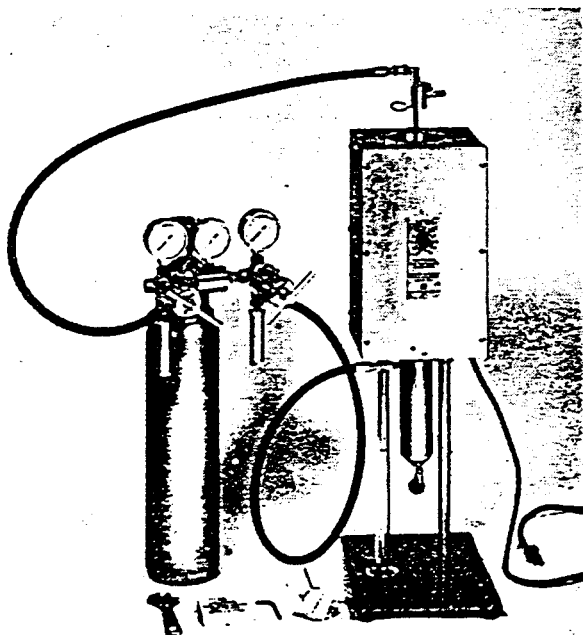


FIG. 3.3
HIGH TEMPERATURE FILTER PRESS

c. At the end of 30 minutes, measure the volume of filtrate. Shut off the flow through the pressure regulator and open the relief valve carefully. The time interval, if other than 30 minutes, will be reported.

d. Report the volume of filtrate in cubic centimeters (to 0.1 cm³) as the API filtrate, and the initial mud temperature in °F (°C). Save the filtrate for appropriate chemical testing.

e. Remove the cell from the frame, first making certain that all pressure has been relieved. Using extreme care to save the filter paper with a minimum of disturbance to the cake, disassemble the cell and discard the mud. Wash the filter cake on the paper with a gentle stream of water.

f. Measure and report the thickness of the filter cake to the nearest 1/32 in. (0.8 mm).

g. Although cake descriptions are subjective, such notations as hard, soft, tough, rubbery, firm, etc., may convey important information of cake quality.

HIGH-TEMPERATURE/HIGH-PRESSURE TEST

3.4 Equipment — High-Temperature/ High-Pressure Test

CAUTION: *All manufacturers' equipment is not capable of the same temperatures and pressures. Rigid adherence to manufacturers' recommendations as to sample volumes, temperatures, and pressures is essential. Failure to do so could result in serious injury.*

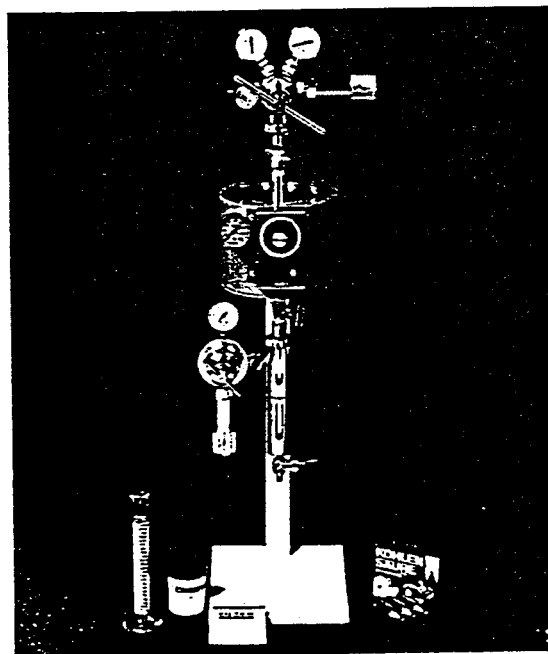


FIG. 3.4
HIGH TEMPERATURE FILTER PRESS

a. The high-temperature/high-pressure filter press consists of a controlled pressure source (CO₂ or nitrogen), regulators, a mud cell able to contain working pressures from 600 to 1300 psi, a system for heating the cell, a pressurized collection cell able to maintain proper back pressure (see Table 3.1) in order to prevent flashing or evaporation of the filtrate, and a suitable stand. Fig. 3.3 and 3.4 show available units. The mud cell has a thermometer well, oil-resistant gaskets, a support for the filter medium and a valve on the filtrate delivery tube to control flow from the cell. It may be necessary to replace the gaskets frequently.

b. Filter medium:

1. Whatman No. 50, or equivalent filter paper, for temperatures to 400°F (204°C).
2. Dynalloy X-5, or equivalent porous disc, for temperature above 400°F (204°C). A new disc is required for each test.

c. Timer: 30-minute interval

d. Thermometer: up to 500°F (260°C)

e. Graduated cylinder (TC): 25-cm³ or 50-cm³

f. High speed mixer

3.5 Procedure — Temperature to 300°F (149°C)

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain desired temperature.

b. Stir mud sample for 10 minutes with a high speed mixer. Install the filter paper and pour the mud sample into the mud cell, being careful not to fill closer than ½ in. (13 mm) from the top to allow for expansion.

c. Complete the assembly of the cell and, with both top and bottom valves closed, place it in the heating jacket. Transfer the thermometer to the well in the mud cell.

d. Connect the high-pressure collection cell to the bottom valve and lock in place.

e. Connect a regulated pressure source to the top valve and collection cell, and lock in place.

f. Keeping the valves closed, adjust top and bottom regulators to 100 psi (690 kPa). Open the top valve, applying 100 psi (690 kPa) to the mud. Maintain this pressure until the desired temperature is stabilized. Time of heating of the sample in the filter cell should never exceed a total of one hour.

g. When the sample reaches the selected temperature, increase the pressure of the top pressure unit to 600 psi (4140 kPa) and open the bottom valve to start filtration. Collect the filtrate for 30 minutes, maintaining the selected temperature within ± 5°F (± 3°C). If back pressure rises above 100 psi (690 kPa) during the

test, cautiously reduce the pressure by drawing off a portion of the filtrate. Record the total volume, temperature, pressure and time.

h. The filtrate volume should be corrected to a filter area of 7.1 in.² (4580 mm²). If the filter area is 3.5 in.² (2258 mm²), double the filtrate volume and report.

i. At the end of test, close top and bottom valves on mud cell. Bleed pressure from the regulators.

CAUTION: *Pressure in the mud cell will still be approximately 500 psi (3450 kPa). Keep cell upright and cool to room temperature before disassembling. Bleed pressure from cell before disassembling.*

3.6 Procedure — Temperature Above 300°F (149°C)

CAUTION: *Not all manufacturers' equipment can be used above 300°F (149°C). Know the pressure/temperature rating of equipment in use. Failure to do so could result in serious injury.*

Testing at high temperature and high pressure will call for added safety precautions. All pressure cells should be equipped with manual relief valves. Heating jackets should be equipped with both an overheat safety fuse and thermostatic cutoff. Vapor pressure of the liquid phase of muds becomes an increasingly critical design factor as test temperatures are raised. Water vapor pressures at various temperatures are shown in Table 3.1.

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain the correct temperature.

b. Stir mud sample for 10 minutes with a high speed mixer. Install the proper filter medium (Par. 3.4b). Pour the mud sample into the mud cell, being careful not to fill the cell closer than 1.5 in. (38 mm) from the top to allow for expansion.

c. Complete the assembly of the cell, and with top and bottom valves closed, place the mud cell in the heating jacket. Transfer the thermometer to the well in the mud cell.

d. Connect the high-pressure collection cell to the bottom valve, and lock in place.

e. Connect the regulated pressure source to the top valve and the collection cell, and lock in place.

f. With top and bottom valves closed, apply the recommended back pressure (Table 3.1) for the test temperature to both top and bottom. Open the top valve, applying the same pressure to the mud while heating. Maintain this pressure until the test temperature is reached and stabilized.

g. When the temperature of the sample reaches the test temperature, increase the pressure on the top by 500 psi (3450 kPa) over the back pressure being held.

and open the bottom valve to begin filtration. Collect the filtrate for 30 minutes, holding the test temperature within $\pm 5^{\circ}\text{F}$ ($\pm 3^{\circ}\text{C}$) and maintaining the proper back pressure. If the back pressure should begin to rise, it can be reduced by cautiously drawing off a small portion of the filtrate. Time of heating of the sample in the filter cell should never exceed a total of one hour.

h. After the test period close both top and bottom valves on the pressure cell and bleed pressure from the regulators. Allow a minimum of 5 minutes for the fil-

trate to cool to avoid vaporizing, then cautiously drain and record the total volume. Also record the temperature, pressures, and time. Be sure to allow sufficient time for all the filtrate to drain from the receiver.

CAUTION: *Pressure inside the filter cell will still be approximately 500 psi (3450 kPa). Keep cell upright and cool to room temperature before disassembly. Bleed pressure from cell before disassembling. Failure to do so could result in serious injury.*

**TABLE 3.1
RECOMMENDED MINIMUM BACK PRESSURE**

Test Temperature		Vapor Pressure		Minimum Back Pressure	
$^{\circ}\text{F}$	$^{\circ}\text{C}$	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
..... Limit of "Normal" Field Testing					
*350	177	135	932	160	1104
*400	204	247	1704	275	1898
*450	232	422	2912	450	3105

*Do not exceed equipment manufacturers' recommendations for maximum temperatures, pressures, and volumes.

SECTION 4 WATER, OIL AND SOLIDS

4.1 Description. The retort instrument provides a means for separating and measuring the volumes of water, oil and solids contained in a sample of water base mud. In the retort, a known volume of a whole water mud sample is heated to vaporize the liquid components which are then condensed and collected in a graduated receiver. Liquid volumes are determined directly from reading the oil and water phases in the receiver. Total volume of solids (suspended and dissolved) is obtained by difference (total sample volume — liquid volume). Calculations are necessary to determine the volume of suspended solids since any dissolved solids will be retained in the retort. The relative volumes of low gravity solids and weighting material can also be calculated. Knowledge of the solids concentration and composition is considered basic to rheology and filtration control in water base muds.

4.2 Equipment

a. Retort instrument (Fig. 4.1 and 4.2)

Retorts of two sizes (10-cm³ and 20-cm³) are commonly available. Specifications of these retorts are:

1. Sample cup

	Sample Cup Size	
	10-cm ³	20-cm ³
Total volume	10-cm ³	20-cm ³
Precision	±0.05 cm ³	±0.10 cm ³

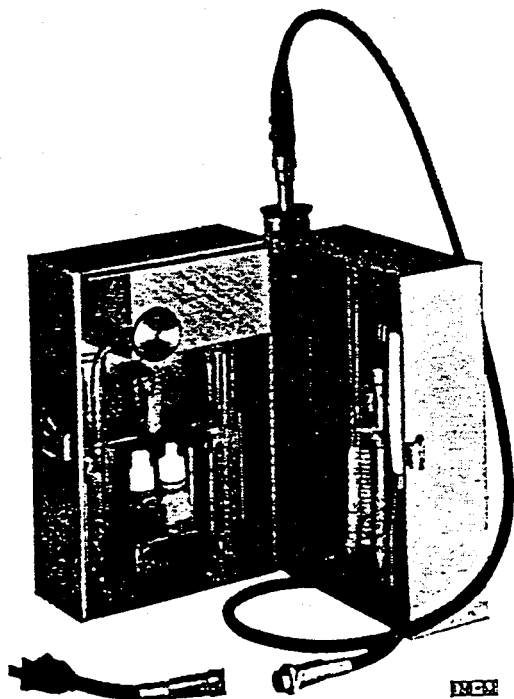


FIG. 4.1
RETORT FOR LIQUID AND SOLID CONTENT

2. Liquid condenser — sufficient mass to cool the oil and water vapors below their vaporization temperature prior to leaving the condenser.
3. Heating element — sufficient wattage to raise the temperature of the sample above the vaporization point of the liquid components within 15 minutes without causing solids boil-over.
4. Temperature control — a temperature control is desirable. It should be capable of limiting the temperature of the retort to 930°F ± 70 (500°C ± 20).

b. Liquid receiver

1. Graduated cylinder, or tube
2. Material — transparent and inert to oil, water, salt solution and temperatures up to 90°F (32°C).
3. Specifications of graduation marks:

	Graduate Size	
	10-cm ³	20-cm ³
Precision	±0.10 cm ³	±0.20 cm ³
Graduation	0.10 cm ³	0.20 cm ³
Calibration method for marks	TC (to contain at 20°C)	
Scale to read	cm ³ and/or volume percent	

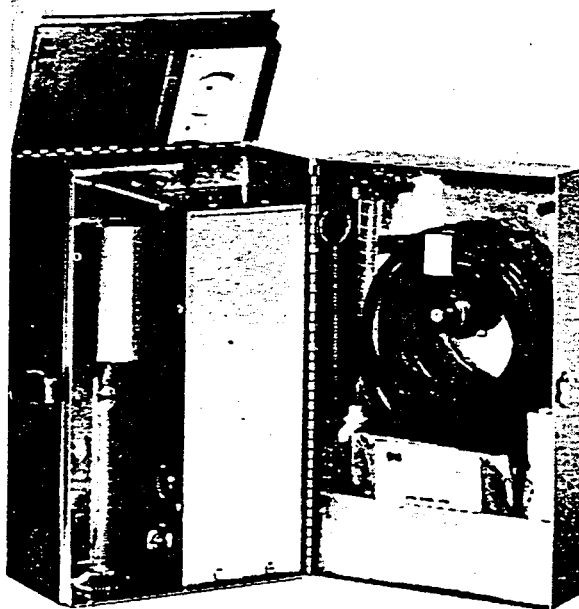


FIG. 4.2
RETORT FOR LIQUID AND SOLID CONTENT

c. Fine steel wool (i.e., No. 000)

NOTE: "Liquid Steel Wool" is not recommended.

d. High temperature silicone grease. (Used for a thread seal and a lubricant.)

e. Pipe cleaners

f. Putty knife or spatula with blade shaped to fit the inside dimensions of the sample cup of the retort.

g. Defoaming agent

4.3 Procedure

NOTE: Procedure will vary slightly depending on type of retort used. See manufacturer's instructions for complete procedure.

a. Be sure the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use.

1. The inside of the sample cup and lid must be thoroughly cleaned prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool.

2. The condenser passage should also be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser can decrease condensation efficiency and cause erroneous liquid readings in a test.

b. Collect a representative sample of water mud and allow it to cool to approximately 80°F (26°C). Screen the sample through the 12-mesh screen on the Marsh Funnel to remove lost circulation material, large cuttings or debris.

c. If mud sample contains gas or air, add 2 to 3 drops of defoaming agent to about 300 cm³ of mud and stir slowly for 2 to 3 minutes to release gases.

d. Lubricate the threads on the sample cup and condenser tube with a light coating of silicone grease. This prevents vapor loss through the threads and also facilitates disassembly of the equipment and clean-up at the end of the test.

e. Lightly pack a ring of steel wool into the chamber above the sample cup. Use only enough steel wool to prevent boil over of solids into the liquid receiver. (This is determined from experience.)

f. Fill the retort sample cup with gas-free water mud, from Par. 4.3c.

g. Carefully place the lid on the sample cup and allow an overflow of the sample through the hole in the lid to insure that the correct volume of sample is in the cup.

h. With the lid held tightly in place, wipe the overflow from the sample cup and lid. Be sure that the

sample cup threads are still covered with silicone grease after wiping, and that the hole in the lid is not plugged.

i. Screw the retort cup onto the retort chamber with its condenser.

j. Place a clean, dry, liquid receiver under the condenser discharge tube.

k. Heat the retort and observe the liquid falling from the condenser. Continue heating for ten minutes beyond the time that no more condensate is being collected.

l. Remove the liquid receiver from the retort. Notice if solids are in the liquid which was recovered. If so, whole mud has boiled over from the sample cup and the test must be repeated from Par. 4.3a.

m. Read the volumes of water and oil in liquid receiver after it has cooled to ambient temperature. Record the volumes (or volume percentage) of water and oil collected.

4.4 Calculation

Using the measured volumes of oil and water and the volume of the original whole mud sample (10-cm³ or 20-cm³), calculate the volume percent water, oil and total solids in the mud.

Volume percent water:

$$V_w = \frac{100 (\text{volume of water, cm}^3)}{\text{volume of sample, cm}^3} \quad (a)$$

Volume percent oil:

$$V_o = \frac{100 (\text{volume of oil, cm}^3)}{\text{volume of sample, cm}^3} \quad (b)$$

Volume percent retort solids:

$$V_s = 100 - (V_w + V_o) \quad (c)$$

NOTE: The volume percent retort solids above is only the difference between water plus oil, and the total sample volume (10-cm³ or 20-cm³). This difference is both suspended solids (weighting material and low gravity) and dissolved materials (e.g., salt). This volume percent retort solids is the suspended solids only if the mud is an untreated, fresh water mud.

b. Additional calculations are required to find the volume percent suspended solids and relate them to the relative volumes of low gravity solids and weighting material. To make these calculations, an accurate mud weight and chloride concentration are needed.

$$V_{ss} = V_s - V_w \frac{C_s}{1.680.000 - 1.21 C_s} \quad (d)$$

where:

V_{ss} = volume percent suspended solids

C_s = chloride concentration, mg/L

Volume percent low gravity solids are calculated as:

$$V_{lg} = \frac{1}{(\rho_b - \rho_{lg})} [100\rho_f + (\rho_b - \rho_f)V_{ss} - 12W_m - (\rho_f - \rho_o)V_o] \quad (e)$$

where:

- V_{lg} = volume percent low gravity solids
 W_m = mud weight, lb/gal
 ρ_f = density of filtrate, g/cm³
 $(\rho_f = 1 + 0.00000109 C_s)$ based on sodium chloride (f)
 ρ_b = density of weighting material, g/cm³
 ρ_{lg} = density of low gravity solids, g/cm³
 (use 2.6 if unknown)
 ρ_o = density of oil, g/cm³ (use 0.84 if unknown)

Volume percent weighting material (V_b) is calculated as:

$$V_b = V_{ss} - V_{lg} \quad (g)$$

Concentration of low gravity solids, weighting material and suspended solids can be calculated as:

$$C_{lg} = 3.49 (\rho_{lg}) (V_{lg}) \quad (h)$$

$$C_b = 3.49 (\rho_b) (V_b) \quad (i)$$

$$C_{ss} = C_{lg} + C_b \quad (j)$$

where:

- C_{lg} = low gravity solids concentration, lb/bbl
 C_b = weighting material concentration, lb/bbl
 C_{ss} = suspended solids concentration, lb/bbl

SECTION 5 SAND

5.1 Description The sand content of mud is the volume percent of particles larger than 74 microns. It is measured by a sand-screen set (see Fig. 5.1).

5.2 Equipment

- a. 200-mesh sieve, 2.5 in. (63.5 mm) in diameter
- b. Funnel to fit sieve
- c. Glass measuring tube marked for the volume of mud to be added. The tube is graduated from 0 to 20 percent in order to read directly the percentage of sand.

5.3 Procedure

- a. Fill the glass measuring tube with mud to the "mud" mark. Add water to the next mark. Close the mouth of the tube and shake vigorously.
- b. Pour the mixture onto the clean, wet screen. Discard the liquid passing through the screen. Add more water to the tube, shake, and again pour onto the screen. Repeat until the tube is clean. Wash the sand retained on the screen to free it of any remaining mud.
- c. Put the funnel upside down over the top of the sieve. Slowly invert the assembly and insert the tip of the funnel into the mouth of the glass tube. Wash the sand into the tube by playing a fine spray of water through the screen. Allow the sand to settle. From the graduations on the tube, read the volume percent of the sand.
- d. Report the sand content of the mud in volume percent. Report the source of the mud sample, i.e., above shaker, suction pit, etc. Coarse solids other than sand will be retained on the screen (e.g., lost circulation material) and the presence of such solids should be noted.



FIG. 5.1
SAND-CONTENT SET

SECTION 6 METHYLENE BLUE CAPACITY (TENTATIVE)

METHYLENE BLUE CAPACITY OF DRILLING FLUIDS

6.1 Description

a. The methylene blue capacity of a drilling fluid is an indication of the amount of reactive clays (bentonite and/or drill solids) present as determined by the methylene blue test (MBT). The methylene blue capacity provides an estimate of the total cation exchange capacity (CEC) of the drilling fluid solids. Methylene blue capacity and cation exchange capacity are not necessarily equivalent, the former normally being somewhat less than the actual cation exchange capacity.

b. Methylene blue solution is added to a sample of drilling fluid (which has been treated with hydrogen peroxide and acidified) until saturation is noted by formation of a dye "halo" around a drop of solids suspension placed on filter paper. Variations of the procedure used on the drilling fluid can be performed on drill solids and commercial bentonite to allow an estimate of the amount of each type of solid present in the fluid (see API RP 13I Section 9).

c. Drilling fluids frequently contain substances in addition to reactive clays that adsorb methylene blue. Pretreatment with hydrogen peroxide (see Par. 6.3b) is intended to remove the effect of organic materials such as lignosulfonates, lignites, cellulosic polymers, polyacrylates, etc.

6.2 Equipment

a. Methylene blue solution: 3.20 g Reagent grade methylene blue ($C_{16}H_{18}N_3SCl$)/L (1 cm^3 = 0.01 milliequivalent)

NOTE: The moisture content of Reagent grade methylene blue must be determined each time the solution is prepared. Dry a 1.000 gram portion of methylene blue to a constant weight at 200°F ± 5 (93°C ± 3). Make the appropriate correction in the weight of methylene blue to be taken to prepare the solution as follows:

$$\text{Weight of sample to be taken, g} = \frac{3.20}{\text{weight of dried sample}} \quad (a)$$

- b. Hydrogen peroxide: 3 percent solution
- c. Dilute sulfuric acid: approximately 5N
- d. Syringe (TD): 2.5- cm^3 or 3- cm^3
- e. Erlenmeyer flask: 250- cm^3
- f. Burette (TD): 10- cm^3 , micropipette: 0.5- cm^3 , or graduated pipette: 1- cm^3
- g. Graduated cylinder (TD): 50- cm^3
- h. Stirring rod
- i. Hot plate
- j. Whatman No. 1 filter paper, or equivalent

6.3 Procedure

a. Add 2.0 cm^3 of drilling fluid (or suitable volume of drilling fluid to require from 2-10 cm^3 of methylene blue solution) to 10 cm^3 of water in the Erlenmeyer flask. To assure that exactly 2.0 cm^3 is being added, use the following procedure:

1. The syringe should have a capacity of more than 2 cm^3 , generally 2.5 or 3 cm^3 . By using a larger syringe, it is not necessary to remove the air trapped in the syringe.
2. The air or gas entrained in the drilling fluid must be removed. (See Appendix D.) Stir the drilling fluid to break the gel and quickly draw the drilling fluid into the syringe. Then slowly discharge the syringe back into the drilling fluid keeping the tip submerged.
3. Again draw the drilling fluid into the syringe until the end of the plunger is at the last graduation on the syringe (e.g., at the 3 cm^3 line on a 3 cm^3 syringe).
4. Deliver 2.0 cm^3 of drilling fluid by pushing the plunger until the end of the plunger is exactly 2 cm^3 from the last graduation on the syringe. Thus in a 3 cm^3 syringe, it would be at the 1 cm^3 line.

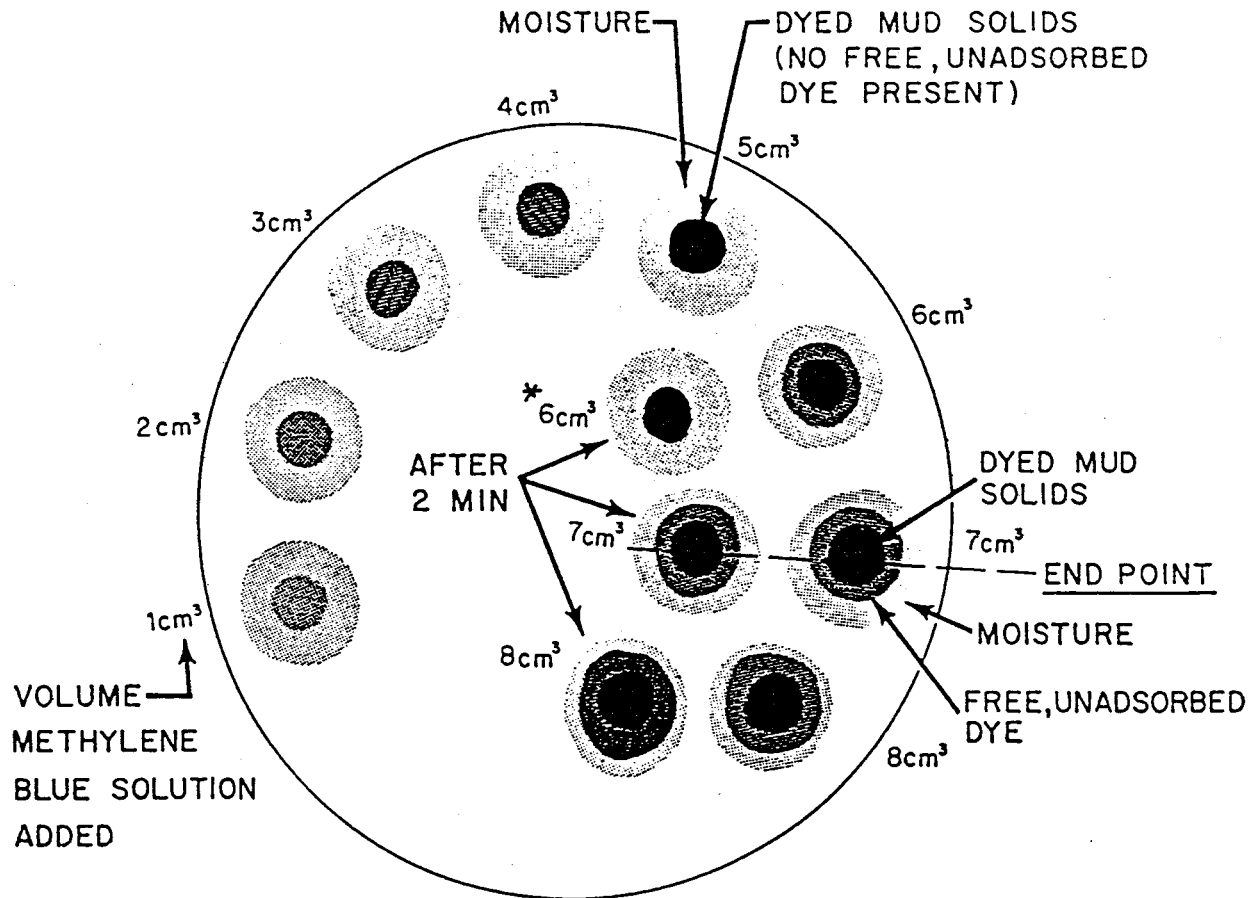
b. Add 15 cm^3 of 3 percent hydrogen peroxide and 0.5 cm^3 of sulfuric acid. Boil gently for 10 minutes, but do not allow to boil to dryness. Dilute to about 50 cm^3 with water.

c. Add methylene blue to the flask in increments of 0.5 cm^3 . If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous testing, larger increments (1-2 cm^3) can be used at the beginning of the titration. After each addition of methylene blue solution, swirl the contents of the flask for about 30 seconds. While the solids are still suspended, remove one drop of liquid with the stirring rod and place the drop on the filter paper. The initial endpoint of the titration is reached when dye appears as a blue ring surrounding the dyed solids as shown in Fig. 6.1.

d. When the blue tint spreading from the spot is detected, shake the flask an additional 2 minutes and place another drop on the filter paper. If the blue ring is again evident, the final endpoint has been reached. If the blue ring does not appear, continue as before (Par. 6.3c) until a drop taken after 2 minutes shows the blue tint.

6.4 Calculation. Report the methylene blue capacity (MBT) of the drilling fluid, calculated as follows:

$$\text{Methylene blue capacity, } cm^3/cm^3 = \frac{\text{methylene blue, } cm^3}{\text{drilling fluid, } cm^3} \quad (a)$$



*Note: Free dye detected immediately after adding sixth cm^3 is adsorbed after two minutes and indicates that end point has not quite been reached.

FIG. 6.1
SPOT TESTS FOR END POINT OF METHYLENE BLUE TITRATION

Alternately, the methylene blue capacity can be reported as lb/bbl bentonite equivalent (based on bentonite with a cation exchange capacity of 70 meq/100 g) calculated as follows:

$$\text{Bentonite equivalent, lb/bbl} = \frac{5 \text{ (methylene blue, cm}^3\text{)}}{\text{drilling fluid, cm}^3} \quad (b)$$

$$\text{Bentonite equivalent, kg/m}^3\text{g} = \frac{2.85 \text{ (bentonite equivalent, lb/bbl)}}{\text{drilling fluid, cm}^3} \quad (c)$$

NOTE: The lb/bbl bentonite equivalent from (b or c) is not equal to the amount of commercial bentonite in the drilling fluid. Reactive clays in the drill solids contribute to this quantity as well as commercial bentonite. See API RP 13I Section 9 for additional information on estimating the amount of commercial bentonite and drill solids present.

SECTION 7

pH

7.1 Description

a. Field measurement of drilling fluid (or filtrate) pH and adjustments to the pH are fundamental to drilling fluid control. Clay interactions, solubility of various components and contaminants, and effectiveness of additives are all dependent on pH, as is the control of acidic and sulfide corrosion processes.

b. The term "pH" denotes the negative logarithm of the hydrogen ion, H^+ , activity in aqueous solutions (activity and concentration are equal only in dilute solutions):

$$pH = -\log [H^+]$$

For pure water at 75°F (24°C) the hydrogen ion activity [H^+] is 10^{-7} moles/liter and $pH = 7$. This system is termed "neutral" because the hydroxyl ion activity [OH^-] is also 10^{-7} moles/liter. In aqueous systems at 24°C the ion product, $[H^+] \times [OH^-]$, is 10^{-14} (a constant). Consequently, an increase in H^+ denotes a like decrease in OH^- . A change in pH of one unit indicates a ten-fold change in both $[H^+]$ and $[OH^-]$. Solutions with pH less than 7 are termed "acidic" and those with pH greater than 7 are termed "basic" or "alkaline."

c. The recommended method for pH measurement of drilling fluid is with a glass electrode pH meter. This method is accurate and gives reliable pH values, being free of interferences if a high quality electrode system is used with a properly designed instrument. Rugged pH instruments are available that automatically temperature compensate the slope and are preferred over the manually adjusted instruments.

NOTE: Color matching pH paper and sticks are used for field pH measurements, but are not the methods recommended. These methods are reliable only in very simple water muds. Mud solids, dissolved salts and chemicals, and dark-colored liquids cause serious errors in pH paper values. Readability is normally about 0.5 pH unit.

7.2 Equipment

a. pH meter: millivolt range potentiometer calibrated to show pH units for measuring the potential between a glass-membrane electrode and a standard "reference" electrode. The instrument is (preferred) to be water, shock, and corrosion-resistant and portable. Specifications are:

1. pH range: 0 to 14
2. Electronics type: solid state (preferred)
3. Power Source: batteries (preferred)
4. Operating temperature range: 32-150°F (0-66°C)
5. Readout: digital (preferred)

6. Resolution: 0.1 pH unit

7. Accuracy: ± 0.1 pH unit

8. Repeatability: 0.1 pH unit

9. Adjustments

- (a) "temperature" compensation of electrode system
- (b) "slope" of electrode system (preferred)
- (c) "calibration" setting of readout. (Instrument with the above internal temperature compensation is preferred).

b. Electrode system: A combination system of a glass electrode for sensing H^+ ions and a standard voltage reference electrode, constructed as a single electrode (preferred). Body of this probe should be constructed of durable material. A flat-end probe is preferred for better protection and easier cleaning of the electrode. Waterproof connection to the meter is recommended. Specifications are:

1. Glass pH electrode response range: 0 to 14 pH unit
2. Electrodes: a glass electrode and a silver/silver chloride electrode in combination, having a ceramic or a plastic single or double junction.
NOTE: Use double junction electrode for measuring liquids containing sulfide or bromide ion to avoid damaging (silver) reference electrode system.
3. Electrolyte in reference electrode: KCl gel
4. Glass composition: suitable for low sodium ion error.
5. Sodium ion error: at $pH = 13$ or at 0.1 mole Na^+ ion, an error less than 0.1 pH unit.

c. Buffer solutions: three solutions to calibrate and set slope of pH meter prior to sample measurement.

1. $pH = 4.0$: potassium hydrogen phthalate at 0.05 molar in water. Gives 4.01 pH at 75°F (24°C).
2. $pH = 7.0$: potassium dihydrogen phosphate at 0.02066 molar and disodium hydrogen phosphate at 0.02934 molar in water. Gives 7.00 pH at 75°F (24°C).
3. $pH = 10.0$: sodium carbonate at 0.025 molar and sodium bicarbonate at 0.025 molar in water. Gives 10.01 pH at 75°F (24°C).

NOTE: Buffers may be obtained from supply houses as pre-made solution, dry-powder packages, or a given formula, but must duplicate

National Bureau of Standards primary or secondary buffers. Shelf life of all buffers not to exceed six months before disposal. Date of preparation of buffer should be shown on bottles used in the field. Bottles should be kept tightly stoppered.

- d. Distilled or deionized water: in spray bottle
- e. Soft tissues: to blot electrodes
- f. Thermometer: glass. 32-220°F (0-150°C)
- g. Accessory equipment:
 - 1. Soft-bristle test tube brush: to clean electrode
 - 2. Mild liquid detergent: Ivory[®], or equivalent
 - 3. Electrode storage vial: to keep electrodes moist
 - 4. Sodium Hydroxide: 0.1 molar (approximately): to recondition electrode.
 - 5. Hydrochloric Acid: 0.1 molar (approximately): to recondition electrode.
 - 6. Ammonium Bifluoride: 10% solution (approximately): to recondition electrode.
CAUTION: *This is a strong and toxic acid.*
 - 7. Hydrofluoric acid: ACS reagent grade.
CAUTION: *This is a strong acid.*

7.3 Procedure — pH Measurement

a. Obtain sample of fluid to be tested. Allow it to reach 75°F ± 5 (24°C ± 3).

b. Allow buffer solution to also reach the same temperature as the fluid to be tested.

NOTE: *For accurate pH measurement the test fluid, buffer solution, and reference electrode must all be at the sample temperature. The pH of the buffer solution indicated on the container label is the correct pH only at 75°F (24°C). If attempting to calibrate at another temperature, the actual pH of the buffer at this temperature must be used. Tables of buffer pH values at various temperatures are available from the suppliers and should be used in the calibration procedure.*

- c. Clean electrodes by washing with distilled water and blot dry.
- d. Place probe into pH 7.0 buffer.
- e. Turn on meter; wait 60 seconds for reading to stabilize. (See Par. 7.4a if meter reading is not stable.)
- f. Measure temperature of pH 7 buffer solution.
- g. Set this temperature on "temperature" knob.
- h. Set meter reading to "7.0" using "calibration" knob.
- i. Rinse probe with distilled water and blot dry.

j. Repeat operations in Par. 7.3f through 7.3i using either pH 4.0 or pH 10.0 buffer. Use pH 4.0 if "acidic" sample, or pH 10.0 if "alkaline" sample is to be tested. Set meter to number "4.0" or "10.0" respectively, using "slope" adjustment knob. (If no "slope" knob exists, use the "temperature" knob to set "4.0" or "10.0" on meter.)

k. Check the meter with pH 7 buffer again. If it has changed, reset to "7.0" with "calibration" knob. Repeat Par. 7.3f through 7.3k. If meter does not calibrate properly, recondition or replace electrodes as given in Par. 7.4a through 7.4f.

NOTE: *Discard and do not reuse the sample of buffer solutions used in calibration. Meter should be fully calibrated every day, Par. 7.3b through 7.3k, using two buffers. Check with pH 7 buffer every three hours.*

l. If meter calibrates properly, rinse electrode with distilled water and blot dry. Place electrode in sample to be tested and stir gently. Allow 60 to 90 seconds for reading to stabilize.

m. Record sample pH to nearest 0.1 pH unit and the temperature of sample tested on Drilling Mud Report form. (Indicate whether mud or filtrate was tested.)

n. Carefully clean the electrode in preparation for next usage. Store in vial of pH 7 buffer. NEVER let the probe tip become dry.

o. Turn meter off and close cover to protect instrument. Avoid storing instrument at extreme temperatures. (Below 32°F (0°C) or above 120°F (49°C)).

7.4 Procedure — Care of Electrode

a. Cleaning the electrode will be necessary periodically, especially if oil or clay particles coat the face of the glass electrode or the porous frit of the reference electrode. Clean electrode with a soft-bristle brush and a mild detergent.

b. Reconditioning the electrode may be necessary if plugging becomes severe, as indicated by slow response, drifting of readings, or if "slope" and "calibration" cannot be mutually set.

c. Recondition by soaking electrode for 10 minutes in 0.1 M HCl followed by rinsing in water and soaking for 10 minutes in 0.1 M NaOH and rinsing again.

d. Check electrode for response by performing calibration Par. 7.3b through 7.3k.

e. If electrode continues to perform poorly, soak electrode for two minutes only in 10% NH₄F·HF solution. (CAUTION: *This is strong and toxic acid.*) Repeat Par. 7.3b through 7.3k to check for calibration capability.

f. Replace electrode system if above steps fail to recondition it.

SECTION 8 CHEMICAL ANALYSIS

ALKALINITY AND LIME CONTENT

8.1 Description

a. Alkalinity can be considered as the acid-neutralizing power of a substance. In drilling fluid testing, alkalinity measurements can be made on either the whole mud (designated with a subscript m) or on the filtrate (designated with a subscript f). The data collected from the alkalinity test can also be used to estimate the concentrations of hydroxyl (OH^-), carbonate (CO_3^{2-}) and bicarbonate (HCO_3^-) ions in the drilling fluid.

b. Knowledge of the mud and filtrate alkalinities is important in many drilling operations to insure proper control of the mud chemistry. Mud additives, particularly some deflocculants, require an alkaline environment to function properly. The source and nature of the alkalinity is often as important as the fact that some alkalinity exists. Alkalinity arising from hydroxyl ions is generally accepted as being beneficial while alkalinities resulting from carbonates and/or bicarbonates may have adverse effects on the mud performance.

c. The ions that are primarily responsible for filtrate alkalinities are the hydroxyl (OH^-), carbonate (CO_3^{2-}), and bicarbonate (HCO_3^-) ions. It is important to realize that the carbonate species can change from one form to another form by changing the solution pH. The interpretation of filtrate alkalinities involves calculating differences between the titration values obtained by the following procedures. It is for this reason that special attention to accurate measurement of the various reagents is important in all steps of the procedure. In addition it is important to realize that the following calculations are only estimates of the concentrations of the reported ionic species based on theoretical chemical equilibrium reactions.

d. The composition of mud filtrates is often so complex that the interpretation of alkalinities in terms of estimated ionic components may be misleading. Any particular alkalinity value represents all of the ions which will react with the acid in the pH range over which that particular value was tested. Inorganic ions which may contribute to the alkalinity, in addition to the hydroxyl, carbonate, and bicarbonate ions, are: borates, silicates, sulfides, and phosphates. Perhaps more serious in drilling fluids, the anionic organic thinners, filtrate reducers, and their degradation products may contribute to a large portion of the alkalinity value as well as masking the endpoint color change. These organic materials make a particularly large contribution to the M_f alkalinity and thus render the test highly inaccurate in muds treated with organic thinners. However, for simple bentonite base mud systems containing no organic thinners the P_f and M_f alkalinities can be used as guidelines to determine both the presence of carbonate/bicarbonate contamination and the treatment necessary to alleviate the contamination. If organic thinners are present in large amounts, the

P_f/M_f test is suspect and the P_1/P_2 method should be used.

8.2 Equipment

a. Sulfuric acid solution: standardized 0.02 Normal (N/50)

b. Phenolphthalein indicator solution: 1 g/100 cm³ of a 50% alcohol/water solution

c. Methyl orange indicator solution: 0.1 g/100 cm³ of water

d. pH Meter (optional)

NOTE: *pH meter is more accurate than indicator solution.*

e. Titration vessel: 100-150-cm³, preferably white

f. Serological (graduated) pipettes (TD): one 1-cm³ and one 10-cm³

g. Volumetric pipette (TD): 1-cm³

h. Hypodermic syringe (TD): 1-cm³

i. Stirring rod

8.3 Procedure — Filtrate Alkalinity: P_f , M_f

a. Measure one or more cm³ of filtrate into the titration vessel. Add two or more drops of the phenolphthalein indicator solution. If the indicator turns pink, add 0.02 normal (N/50) sulfuric acid, drop by drop from the graduated pipette, while stirring, until the pink color just disappears. If the sample is so colored that the indicator color change is masked, the endpoint can be taken when the pH drops to 8.3, as measured with a pH meter. (Refer to Section 7 for proper pH measurement.)

b. Report the phenolphthalein alkalinity of the filtrate, P_f , as the number of cm³ of 0.02 normal acid required per cm³ of filtrate.

c. To the sample which has been titrated to the P_f endpoint, add two or three drops of methyl orange indicator solution. Add the standard acid drop by drop from the pipette, while stirring, until the color of the indicator changes from yellow to pink. The endpoint can also be taken when the pH of the sample drops to 4.3 as measured by a pH meter. (Refer to Section 7 for proper pH measurement.)

d. Report the methyl orange alkalinity of the filtrate, M_f , as the total cm³ of 0.02 normal acid per cm³ of filtrate required to reach the methyl orange endpoint (including that amount required for the P_f endpoint.)

8.4 Procedure — Mud Alkalinity: P_m

a. Measure 1.0 cm³ of mud into the titration vessel using a syringe or volumetric pipette. Dilute the mud sample with 25-50 cm³ of distilled water. Add 4-5 drops of phenolphthalein indicator solution and, while stirring, titrate rapidly with 0.02 normal (N/50) standard sulfuric acid solution until the pink color disappears. If

the endpoint color change cannot be seen, it can be taken when the pH drops to 8.3 measured by a pH meter. (Refer to Section 7 for proper pH measurement.)

NOTE: If cement contamination is suspected, the titration must be performed as rapidly as possible and the endpoint reported as the first disappearance of the pink color.

b. Report the phenolphthalein alkalinity of the mud, P_m , as the number of cm³ of 0.02 normal (N/50) acid required per cm³ of mud.

8.5 Calculation — P_f , M_f The concentrations of hydroxyl, carbonate, and bicarbonate ions can be estimated as follows:

TABLE 8.1
CONCENTRATIONS, mg/L

	OH ⁻	CO ₃ ⁻²	HCO ₃ ⁻
$P_f = 0$	0	0	1220M _f
$2P_f < M_f$	0	1200P _f	1220 (M _f - 2P _f)
$2P_f = M_f$	0	1200P _f	0
$2P_f > M_f$	340 (2P _f - M _f)	1200 (M _f - P _f)	0
$P_f = M_f$	340M _f	0	0

8.6 Procedure — Estimated Lime Content

a. Determine the P_f and P_m of the filtrate and mud as described in Par. 8.3 and 8.4.

b. Determine the volume fraction of water in the mud, F_w , using the value for volume percent water from the liquid and solids determination (Section 4) in the following equation:

$$F_w = \frac{\text{Volume percent water}}{100} \quad (a)$$

8.7 Calculation — Estimated Lime Content. Report the lime content of the mud in pounds per barrel (lb/bbl) from the following equation:

$$\text{Estimated Lime, lb/bbl} = 0.26 (P_m - F_w P_f) \quad (a)$$

$$\text{Estimated Lime, kg/m}^3 = 0.742 (P_m - F_w P_f) \quad (b)$$

ALTERNATE ALKALINITY METHOD

8.8 Description The P1/P2 back-titration method was mainly developed in an attempt to overcome the limitations of the P_f/M_f alkalinity method. (Par. 8.1 through 8.5.) The P1/P2 method also has limitations. A comparison of the generally accepted advantages and disadvantages of the two alkalinity measurements are listed in Table 8.2.

8.9 Equipment — P1/P2 Alkalinity Method

a. Hydrochloric acid solution: standardized 0.02 normal (N/50)

b. Sodium hydroxide solution: 0.1 normal (N/10).

c. Barium chloride solution: 10 percent, neutralized to pH 7 with NaOH.

d. Phenolphthalein indicator solution: 1 g/100 cm³ of a 50% alcohol-water solution.

e. Deionized water

TABLE 8.2
FILTRATE ALKALINITY METHODS
COMPARISON

Method	Advantage	Disadvantage
P_f/M_f	a. Traditional method b. 2 titrations, 1 sample	a. Interference in the M_f titration b. Bicarbonate result normally too high
P1/P2	a. Eliminates interference in M_f titrations	a. 3 titrations with 3 samples b. Caustic measurement critical c. Uses a toxic material (BaCl ₂)

NOTE: The total carbonate concentration in a drilling fluid can also be determined by use of the Garrett Gas Train as described in the Appendix A, Carbonate.

f. pH paper strips: 6-12 range

g. pH meter, optional (as discussed in Section 7)

h. Titration vessel: 100-150-cm³, preferably white

i. Volumetric pipette (TD): one 1-cm³ and one 2-cm³

j. Burette (TD): automatic student type, 25-cm³

k. Graduated cylinders (TC): one 25-cm³ and one 5-cm³ or 10-cm³

l. Stirring rod

8.10 Procedure — P1/P2 Alkalinity Method

a. Determine the P_f alkalinity as described in Par. 8.2, 8.3a and 8.3b.

b. Using a volumetric pipette, measure 1.0 cm³ of filtrate into the titration vessel. Add 25 cm³ of deionized water to the titration vessel.

c. Using a volumetric pipette, add 2.0 cm³ of 0.1 normal (N/10) sodium hydroxide solution and stir well. Measure the pH with the high range pH paper (or pH meter). If the pH is 11.4 or greater, proceed to Par. 8.10d. If the pH is less than 11.4 add 2.0 cm³ more of 0.1 normal sodium hydroxide solution, then proceed to Par. 8.10d.

NOTE: Exact measurement of the sodium hydroxide is necessary to avoid serious errors.

d. Using the small graduated cylinder, measure 3 cm³ of barium chloride solution and add to the titration vessel. Add 2-4 drops of phenolphthalein indicator solution while stirring.

CAUTION: Do not use your mouth to pipette the barium chloride solution. IT IS EXTREMELY POISONOUS.

e. Immediately titrate the mixture with the standard 0.02 normal hydrochloric acid to the first disappearance of the pink color (or to a pH of 8.3 with a pH meter). The color may reappear after a short time: do not continue the titration.

f. Report the alternate alkalinity, P1, as the cm^3 of 0.02 normal acid to reach the phenolphthalein endpoint.

g. Determine the blank alkalinity, P2. Omit the filtrate, but otherwise repeat the procedure described in Par. 8.10b through 8.10f for determining P1, using *exactly* the same quantities of water and reagents in preparing the sample.

h. Report the blank alkalinity, P2, as the cm^3 of 0.02 normal acid needed to titrate the reagent mixture to the phenolphthalein endpoint.

8.11 Calculation — P1/P2 Alkalinity Method

a. The procedure given in the previous paragraphs is intended to reduce the major interferences in the P_f/M_f alkalinity test, and thus provide a better estimate of hydroxyl, carbonate, and bicarbonate concentrations. Calculating these concentrations does not make them true values; the composition is theoretical, based on water chemistry carbonate equilibriums.

b. Within the limitations outlined the various ionic concentrations can be calculated as follows:

When $P1 > P2$

$$\text{OH}^-, \text{mg/L} = 340(P1 - P2) \quad (a)$$

$$\text{CO}_3^{-2}, \text{mg/L} = 1200[P_f - (P1 - P2)] \quad (b)$$

When $P1 < P2$

$$\text{HCO}_3^-, \text{mg/L} = 1220(P2 - P1) \quad (c)$$

$$\text{CO}_3^{-2}, \text{mg/L} = 1200P_f \quad (d)$$

CHLORIDE

8.12 Description The chloride test measures the chloride-ion concentration in the mud filtrate.

8.13 Equipment

a. Silver nitrate solution: containing 4.7910 g/L (equivalent to 0.001 g chloride-ion/ cm^3), stored in an amber or opaque bottle.

b. Potassium chromate indicator solution: 5 g/100 cm^3 of water.

c. Sulfuric or nitric acid solution: standardized 0.02 normal (N/50).

d. Phenolphthalein indicator solution: 1 g/100 cm^3 of 50% alcohol/water solution.

e. Calcium carbonate: precipitated, chemically pure grade.

f. Distilled water.

g. Serological (graduated) pipettes (TD): one 1- cm^3 and one 10- cm^3 .

h. Titration vessel: 100-150- cm^3 , preferably white.

i. Stirring rod.

8.14 Procedure

a. Measure one or more cm^3 of filtrate into the titration vessel. Add 2-3 drops of phenolphthalein solution. If the indicator turns pink, add acid drop by drop from pipette, while stirring, until the color is discharged. If

the filtrate is deeply colored, add an additional 2 cm^3 of 0.02 normal (N/50) sulfuric or nitric acid and stir. Then add 1 g of calcium carbonate and stir.

b. Add 25-50 cm^3 of distilled water and 5-10 drops of potassium chromate solution. Stir continuously, while adding standard silver nitrate solution drop by drop from the pipette, until the color changes from yellow to orange-red and persists for 30 seconds. Record the number of cm^3 of silver nitrate solution required to reach the endpoint. If over 10 cm^3 of silver nitrate solution are used, repeat the test with a smaller sample of filtrate.

NOTE: If the chloride-ion concentration of the filtrate exceeds 10,000 mg/L, a silver nitrate solution equivalent to 0.01 g chloride-ion per cm^3 may be used. The factor 1,000 in Equation 8.15 (a) is then changed to 10,000.

TABLE 8.3
CONVERSION OF MILLIGRAMS PER LITER
CHLORIDE TO WEIGHT PERCENT SALT (NaCl)
OR PARTS PER MILLION SALT
SOLUTION TEMPERATURE AT 68°F (20°C)

Milligrams Per Liter Chloride	Weight Percent Salt	Milligrams Per Liter Salt	Parts Per Million Salt
3.040	0.5	5.020	5.000
6.100	1	10.050	10.000
12.300	2	20.250	20.000
18.600	3	30.700	30.000
24.900	4	41.100	40.000
31.599	5	52.000	50.000
37.900	6	62.500	60.000
44.200	7	73.000	70.000
51.200	8	84.500	80.000
57.600	9	95.000	90.000
64.900	10	107.100	100.000
71.800	11	118.500	110.000
79.000	12	130.300	120.000
86.100	13	142.000	130.000
93.400	14	154.100	140.000
100.900	15	166.500	150.000
108.200	16	178.600	160.000
115.800	17	191.000	170.000
123.500	18	203.700	180.000
131.200	19	216.500	190.000
139.200	20	229.600	200.000
147.300	21	256.100	210.000
155.200	22	256.100	220.000
163.600	23	270.000	230.000
169.400	24	279.500	240.000
171.700	25	283.300	250.000
188.700	26	311.300	260.000

NOTE: It is quite common for many laboratories analyzing water samples to report milligrams per liter of salt as parts per million. If it is known that an analysis is reported in this way, the milligram per liter salt column should be used in converting to percent salt rather than the parts per million salt column.

8.15 Calculation Report the chloride-ion concentration of the filtrate in mg/L, calculated as follows:

$$\text{Chloride, mg/L} = \frac{1000 (\text{silver nitrate, cm}^3)}{\text{filtrate sample, cm}^3} \quad (\text{a})$$

To convert units:

$$\text{Chloride, ppm} = \frac{\text{Chloride, mg/L}}{\text{Specific Gravity of Filtrate}} \quad (\text{b})$$

$$\text{Salt (NaCl), mg/L} = (1.65) (\text{Chloride, mg/L}) \quad (\text{c})$$

Refer to Table 4.1 for Filtrate Specific Gravity.
Refer to Table 8.3 for conversion of % salt in salt water to mg/L or ppm.

TOTAL HARDNESS AS CALCIUM

8.16 Description The hardness of water or mud filtrate is due primarily to the presence of calcium and magnesium ions. When EDTA (ethylenediaminetetraacetic acid or its salt) is added to the water, it combines both with the calcium and magnesium and the endpoint is determined with a suitable indicator. The total hardness of the water is expressed as mg/L calcium. An endpoint obscured by dark components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

8.17 Equipment

a. EDTA solution (Versenate): 0.01 molar; disodium (ethylenediamine) tetraacetate dihydrate standardized (1 cm³ = 1000 mg/L CaCO₃, 1 cm³ = 400 mg/L Ca⁻²)

b. Buffer solution: 67.5 g ammonium chloride and 570 cm³ ammonium hydroxide (15N) diluted to 1000 cm³ with distilled water.

c. Hardness indicator solution: 1 g/L "Calmagite" or equivalent, 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid in distilled water.

d. Acetic acid: glacial (CAUTION: Avoid skin contact).

e. Titration vessel: 150-cm³ beaker

f. Serological (graduated) pipettes (TD): one 5-cm³ and one 10-cm³

g. Volumetric pipettes (TD): one 1-cm³, one 2-cm³, and one 5-cm³

h. Hot plate (required if filtrate colored)

i. Masking agent 1:1:2 mixture by volume of triethanolamine: tetraethylenepentamine: water (optional)

j. pH paper strip

k. Sodium hypochlorite solution: 5.25% sodium hypochlorite in deionized water (i.e., Clorox® or equivalent)

CAUTION: Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure the sodium hypochlorite is fresh as it will deteriorate with time.

l. Deionized or distilled water

NOTE: The deionized water and sodium hypochlorite solution should be tested for hardness by using 50.0 cm³ of the deionized water in Par. 8.18f, and 10 cm³ of the hypochlorite in Par. 8.18b, without the test sample, and continuing with Par. 8.18g and 8.18h. If the procedure is then repeated with the test sample utilizing 50.0 cm³ of the deionized water and 10 cm³ hypochlorite in Par. 8.18f, 8.18g, and 8.18 h, the hardness of the test sample can be determined by subtracting the hardness of the deionized water and hypochlorite.

8.18 Procedure

a. Measure 1.0 cm³ or more of sample into a 150-cm³ beaker. (If filtrate is clear, or is only lightly colored, omit steps 8.18b through 8.18e).

b. Add 10 cm³ hypochlorite (Clorox® or equivalent) and mix.

c. Add 1 cm³ glacial acetic acid and mix.

d. Boil the sample for five minutes. Maintain the sample volume by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required.

CAUTION: Work in an adequately ventilated area.

e. Cool the sample and wash the sides of the beaker with deionized water.

f. Dilute the sample to 50 cm³ with deionized water. Add about 2 cm³ of hardness buffer and swirl to mix.

NOTE: The presence of soluble iron may interfere with the endpoint determination. Should this be suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume, respectively) has proven to be a suitable masking agent. One cm³ of the mixture is used per titration.

g. Add sufficient hardness indicator (2-6 drops) and mix. A wine-red color will develop if calcium and/or magnesium is present.

h. While stirring, titrate with EDTA solution to the proper endpoint. Calcium indicators will produce a red to blue change. The endpoint is best described as that point where additional EDTA produces no further red to blue color change. The EDTA volume will be used in the calculation in Par. 8.19.

8.19 Calculation

$$\text{Total hardness as calcium, mg/L} = \frac{400 (\text{EDTA volume, cm}^3)}{\text{volume sample, cm}^3} \quad (\text{a})$$

OIL BASE DRILLING FLUIDS

SECTION 1
MUD WEIGHT (DENSITY)

1.1 Description This test procedure is a method for determining the weight of a given volume of liquid. Mud weight may be expressed as pounds per gallon (lb/gal), pounds per cubic foot (lb/ft³), grams per cubic centimeter (g/cm³), or kilograms per cubic meter (kg/m³).

1.2 Equipment

a. Any instrument of sufficient accuracy to permit measurement within ± 0.1 lb/gal (or 0.5 lb/ft³, 0.01 g/cm³, 10 kg/m³) may be used. The mud balance (Fig. 1.1 and 1.2) is the instrument generally used for mud weight determinations. The mud balance is designed such that the mud cup, at one end of the beam, is balanced by a fixed counterweight at the other end and a sliding rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (Attachments for extending the range of the balance may be used when necessary.)

b. Thermometer: 32-220°F (0-105°C)

1.3 Procedure

a. The instrument base should be set on a flat, level surface.

b. Measure the temperature of the mud and record on the Drilling Mud Report form.

c. Fill the clean, dry cup with mud to be tested; put the cap on the filled mud cup and rotate the cap until it is firmly seated. Insure that some of the mud is expelled through the hole in the cap in order to free any trapped air or gas (see Appendix D for Air Removal).

d. Holding cap firmly on mud cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

e. Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the center line.

f. Read the mud weight at edge of the rider toward the mud cup. Make appropriate corrections when a range extender is used.

1.4 Procedure — Calibration The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/ft³ (1000 kg/m³) at 70 F (21 C). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

1.5 Calculation

a. Report the mud weight to the nearest 0.1 lb/gal or 0.5 lb/ft³ (0.01 g/cm³, 10 kg/m³).

b. To convert the reading to other units, use the following:

$$\text{Specific gravity} = \frac{\text{g/cm}^3}{62.43} = \frac{\text{lb/ft}^3}{8.345} = \frac{\text{lb/gal}}{8.345} \quad (\text{a})$$

$$\text{kg/m}^3 = (\text{lb/ft}^3)(16) = (\text{lb/gal})(120) \quad (\text{b})$$

$$\text{Mud gradient, } \frac{\text{lb/ft}^3}{144} = \frac{\text{lb/gal}}{19.24} \text{ or } \frac{\text{kg/m}^3}{2309} \quad (\text{c})$$

TABLE 1.1
DENSITY CONVERSION

pounds per gallon (lb/gal)	pounds per cubic foot (lb/ft ³)	grams per cubic centimeter (g/cm ³)*	kilograms per cubic meter (kg/m ³)
6.5	48.6	0.78	780
7.0	52.4	0.84	840
7.5	56.1	0.90	900
8.0	59.8	0.96	960
8.3	62.3	1.00	1000
8.5	63.6	1.02	1020
9.0	67.3	1.08	1080
9.5	71.1	1.14	1140
10.0	74.8	1.20	1200
10.5	78.5	1.26	1260
11.0	82.3	1.32	1320
11.5	86.0	1.38	1380
12.0	89.8	1.44	1440
12.5	93.5	1.50	1500
13.0	97.2	1.56	1560
13.5	101.0	1.62	1620
14.0	104.7	1.68	1680
14.5	108.5	1.74	1740
15.0	112.2	1.80	1800
15.5	115.9	1.86	1860
16.0	119.7	1.92	1920
16.5	123.4	1.98	1980
17.0	127.2	2.04	2040
17.5	130.9	2.10	2100
18.0	134.6	2.16	2160
18.5	138.4	2.22	2220
19.0	142.1	2.28	2280
19.5	145.9	2.34	2340
20.0	149.6	2.40	2400
20.5	153.3	2.46	2460
21.0	157.1	2.52	2520
21.5	160.8	2.58	2580
22.0	164.6	2.64	2640
22.5	168.3	2.70	2700
23.0	172.1	2.76	2760
23.5	175.8	2.82	2820
24.0	179.5	2.88	2880

*Same as specific gravity (sg).

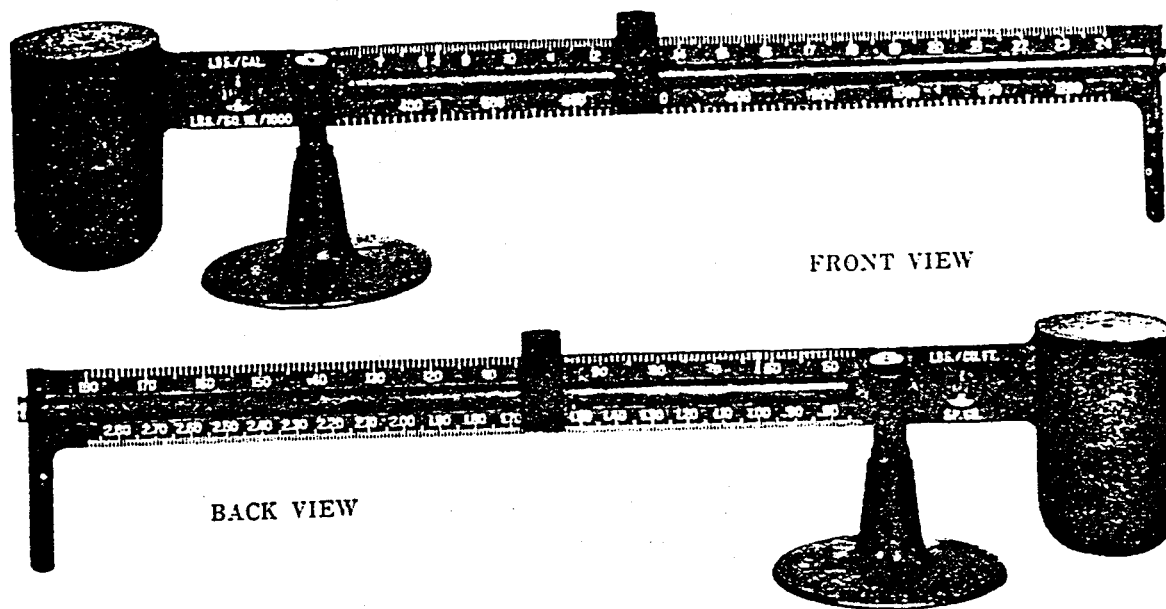


FIG. 1.1
MUD BALANCE

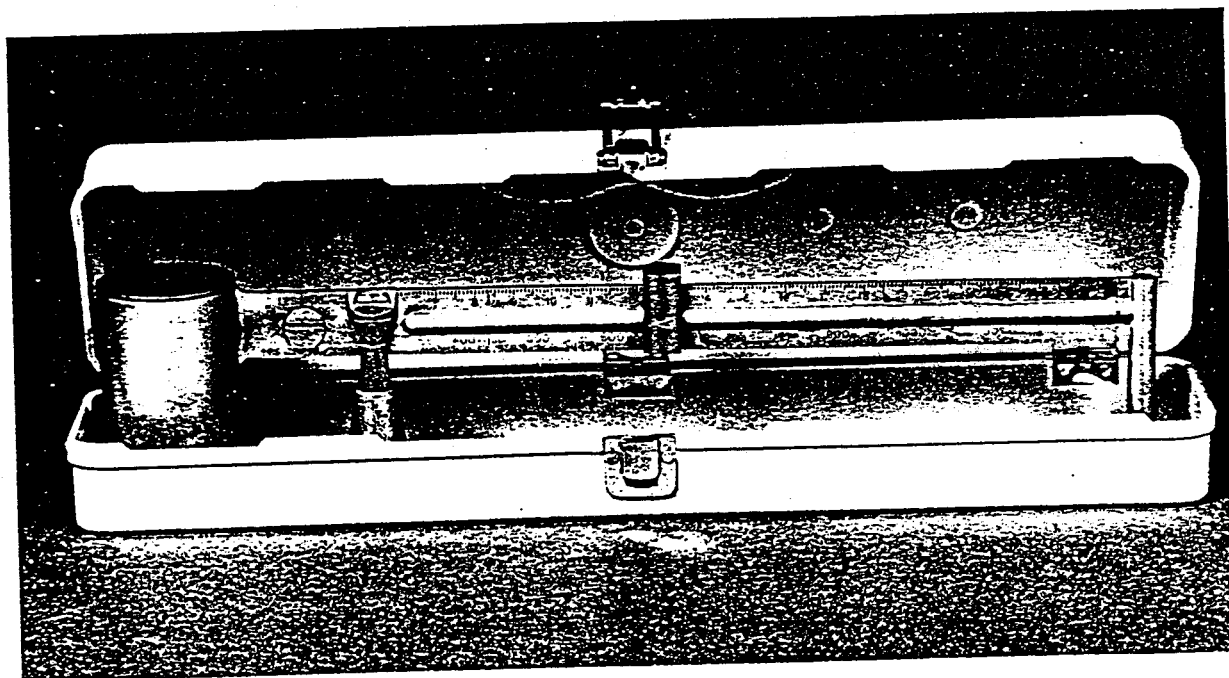


FIG. 1.2
MUD BALANCE AND CASE

SECTION 2 ELECTRICAL STABILITY

2.1 Description

a. The relative stability of a water-in-oil emulsion is indicated by the dielectric breakdown voltage at which the emulsion becomes conductive.

b. This measurement is made using a pair of accurately and permanently spaced electrode plates which are immersed in the fluid sample. A DC power source (battery) is used to provide an AC voltage which is applied to the electrode at a constant rate of voltage increase. The voltage at which the emulsion becomes conductive is indicated by current flow between the electrode plates, completing the circuit and illuminating an indicator bulb or similar device.

NOTE: *Temperature has an effect on the electrical stability, and the measurement should be made at 120°F ± 5 (50°C ± 2).*

2.2 Equipment

a. Electrical Stability Meter

1. Specifications:

0-2000 volts range, with 0-1500 volts optimal
330-350 Hertz, operating frequency
61 microamps, instantaneous electric current at emulsion breakdown.

2. Electrode, spaced 0.061 in. (1.59 mm)

b. Thermometer: 32-220°F (0-105°C).

2.3 Procedure

a. Place sample, which has been screened of particles greater than 12 mesh (Marsh funnel), in container and hand stir with electrode for 30 seconds.

b. Adjust the sample to a temperature of 120°F ± 5 (50°C ± 2). Record temperature on Drilling Mud Report form.

c. Immerse the electrode into the fluid, making certain that the fluid covers the electrode surfaces and that the electrode does not touch the sides or bottom of the container.

d. Depress the Power Source button and hold during the entire test. Do not move the electrode during measurement.

e. Starting from a zero reading, increase the voltage by moving dial in clockwise direction. The rate of increase should be about 100-200 volts per second. Continue increasing voltage until indicator light illuminates.

f. Note dial reading and return dial to zero.

g. Clean electrode thoroughly by passing a paper towel between the electrode plates.

h. To determine repeatability, a duplicate test should be made. Restir the sample for 30 seconds and repeat Par. 2.3c through Par. 2.3f.

2.4 Calculation

Electrical Stability, = 2 (Dial Reading, volts) (a)
volts

A maximum deviation of ±5% is allowed in the results obtained in Par. 2.4. Example: An initial Emulsion Stability of 900 volts, the repeated test should range between 855-945 volts (5% of 900 volts = 45 volts).

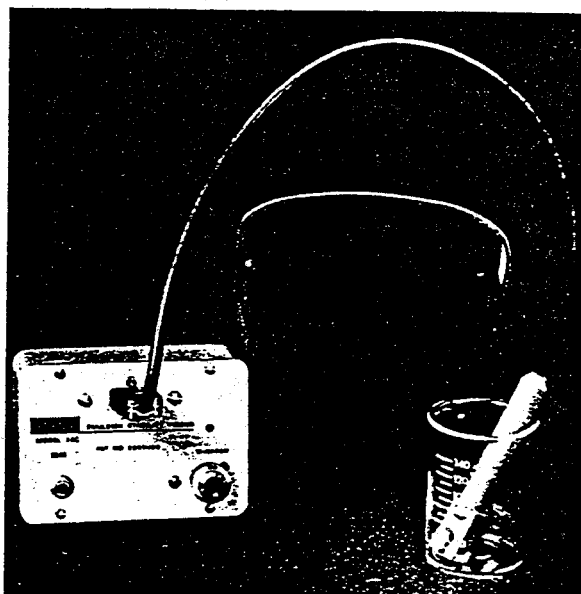


FIG. 2.1
EMULSION TESTER

SECTION 3 WATER, OIL AND SOLIDS

3.1 Description

a. The retort instrument provides a means for separating and measuring the volumes of oil and water and of estimating the solids contained in a sample of oil base or invert emulsion oil mud. In the retort, a known volume of a whole oil mud sample is heated to vaporize the liquid components which are then condensed and collected in a special-shaped graduated receiver. The liquid volumes are measured after centrifugation. Liquid volumes are determined directly from reading the oil and water phases in the receiver. Volumes of solids (suspended and dissolved) are estimated by difference (total sample volume - liquid volume). However, due to dissolved material such as salt in the water and loss by thermal decomposition of heavy organic solids in the retort cup, this estimated solid volume can be erroneously high.

b. The primary purpose for performing the retort test is to determine the water content of the oil mud system. Water content is fundamental to proper control of oil mud composition and mud properties. Percent water is also used in calculations of results from other tests, such as salinity of the aqueous phase of an oil mud. The retort data also measures the percent oil. Knowledge of oil and water content, or o/w ratio, are fundamental to compositional control related to rheol-

ogy, density and filtration. Solids content of oil muds, although not as accurate as in the water mud retort test, can be estimated from retort data and is useful in monitoring solids buildup and solids control.

3.2 Equipment

a. Retort instrument (Fig. 3.1 and 3.2)
Retorts of two sizes (10-cm³ and 20-cm³) are commonly available. Specifications of these retorts are:

1. Sample cup

Total volume	Sample Cup Size	
	10-cm ³	20-cm ³
Precision	±0.05 cm ³	±0.10 cm ³

2. Liquid condenser — sufficient mass to cool the oil and water vapors below their vaporization temperature prior to leaving the condenser.

3. Heating element — sufficient wattage to raise the temperature of the sample above the vaporization point of the liquid components within 15 minutes without causing solids boil-over.

4. Temperature control — a temperature control is desirable. It should be capable of limiting the temperature of the retort to 930°F ± 70 (500°C ± 20).

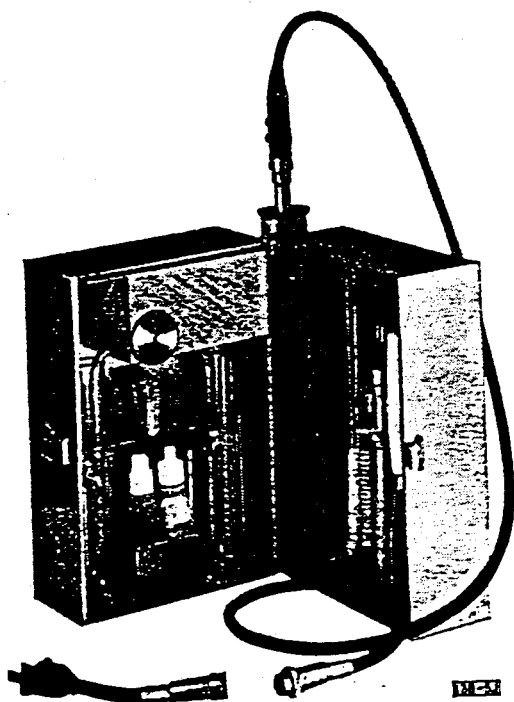


FIG. 3.1
RETORT FOR LIQUID AND SOLID CONTENT

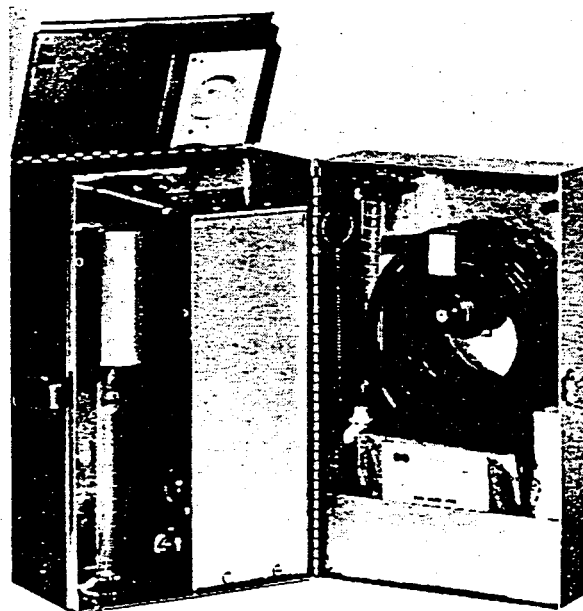


FIG. 3.2
RETORT FOR LIQUID AND SOLID CONTENT

b. Liquid receiver

1. Kolmer-type clinical centrifuge tube (Fig. 3.3)
2. Material — Pyrex or equivalent glass, transparent and inert to oil, water, salt solution and temperatures up to 90°F (32°C)
3. Specifications of graduation marks:

Total Volume	Centrifuge Tube Size	
	10-cm ³	20-cm ³
0 to 5% volume	±0.02 cm ³	±0.04 cm ³
5 to 20% volume	±0.04 cm ³	±0.10 cm ³
20 to 100% volume	±0.10 cm ³	±0.20 cm ³
Frequency of graduation marks:		
0 to 40% volume	±0.10 cm ³	±0.20 cm ³
40 to 100% volume	±0.20 cm ³	±0.20 cm ³

Calibration method for marks TC (to contain at 20°C)
Scale to read cm³ and/or volume percent

c. Hand cranked or electric centrifuge which is capable of holding at least two liquid receivers and having a speed of at least 1800 rpm.

d. Fine steel wool (i.e., No. 000)

NOTE: "Liquid Steel Wool" is not recommended.

e. High temperature silicone grease. (Used for a thread seal and a lubricant.)

f. Pipe cleaners

g. Putty knife or spatula with blade shaped to fit the inside dimensions of the sample cup of the retort.

3.3 Procedure

NOTE: Procedure will vary slightly depending on type of retort used. See manufacturer's instructions for complete procedure.

a. Be sure the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use.

1. The inside of the sample cup and lid must be thoroughly cleaned prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool.
2. The condenser passage should also be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser can decrease condensation efficiency and cause erroneous liquid readings in a test.

b. Collect a representative sample of oil mud and allow it to cool to approximately 80°F (26°C). Screen the sample through the 12-mesh screen on the Marsh Funnel to remove lost circulation material, large cuttings or debris.

c. If mud sample contains gas or air, stir slowly for 2 to 3 minutes to release gases.

d. Lubricate the threads on the sample cup and condenser tube with a light coating of silicone grease. This prevents vapor loss through the threads and also facilitates disassembly of the equipment and cleanup at the end of the test.

e. Lightly pack a ring of steel wool into the chamber above the sample cup. Use only enough steel wool to prevent boil over of solids into the liquid receiver. (This is determined from experience.)

f. Fill the retort sample cup with gas-free oil mud, from Par. 3.3c.

g. Carefully place the lid on the sample cup and allow an overflow of the sample through the hole in the lid to insure that the correct volume of sample is in the cup.

h. With the lid held tightly in place, wipe the overflow from the sample cup and lid. Be sure that the sample cup threads are still covered with silicone grease after wiping, and that the hole in the lid is not plugged.

i. Screw the retort cup onto the retort chamber with its condenser.

j. Place a clean, dry, liquid receiver under the condenser discharge tube.

k. Heat the retort and observe the liquid falling from the condenser. Continue heating for ten minutes beyond the time that no more condensate is being collected. (This may be as long as 45 minutes total time for some tests.)

l. Remove the liquid receiver from the retort. Note the total volume of liquid recovered (oil plus water). Notice if solids are in the liquid which was recovered. If so, whole mud has boiled over from the sample cup and the test must be repeated from Par. 3.3a.

m. Place the liquid-receiver tube into one of the tube holders of the centrifuge rotor head.

n. Select an empty liquid receiver tube and fill it with water to a weight approximately equal to the receiver tube from the test. (This second tube will be used as counterbalance in the centrifuge.)

o. Place the counterbalance tube into the centrifuge tube holder on the opposite side from the retort sample tube.

p. Spin the tubes in the centrifuge for at least 2 minutes at approximately 1800 rpm. (See Potassium test procedure in API RP 13B, Appendix A for instruction on operating the hand cranked centrifuge.) If an emulsion band exists between the oil and water layers after spinning, warm the tube to about 120°F (49°C) before centrifuging again.

q. Read the volumes of oil and water in liquid receiver after it has cooled to 75°F ± 25 (24°C ± 14). Record the volumes (or volume percentage) of water and oil collected.

3.4 Calculation

Using the measured volumes of oil and water and the volume of the original whole mud sample (10-cm³ or 20-cm³), calculate the volume percent water and oil and estimate the volume percent solids in the mud.

Volume percent water:

$$V_w = \frac{100 (\text{volume of water, cm}^3)}{\text{volume of sample, cm}^3} \quad (a)$$

Volume percent oil:

$$V_o = \frac{100 (\text{volume of oil, cm}^3)}{\text{volume of sample, cm}^3} \quad (b)$$

Volume percent solids:

(estimate only)

$$V_s = 100 - (V_w + V_o) \quad (c)$$

NOTE: The calculated percent solids will overestimate the volume of suspended solids by an amount equal to the volume of dissolved salt plus the thermally decomposed organic material in the oil mud sample. This creates a positive error in retort testing because some materials (e.g., inorganic salts) will be retained in the retort and decomposed organics lost as a gas from the condenser. Normally no attempt is made to correct for loss due to degraded organic materials in oil mud retort analysis but a salinity correction can be made using mathematical relationships available from most oil mud suppliers.

SECTION 4 FIELD TESTING OF OIL MUD VISCOSITY AND GEL STRENGTH

4.1 Description

a. The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

1. Marsh Funnel — a simple device for indication of viscosity on a routine basis
2. Direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

b. Viscosity and gel strength are measurements that relate to the flow properties of muds. The study of deformation and flow of matter is rheology. An in-depth statement of rheology is made in API Bulletin 13D: The Rheology of Oil-Well Drilling Fluids.

MARSH FUNNEL

4.2 Equipment. A Marsh Funnel (see Fig. 4.1) is calibrated to outflow one quart (946 cm³) of fresh water at a temperature of 70°F ± 5 (21°C ± 3) in 26 seconds ± 0.5. A graduated cup is used as a receiver.

a. Marsh Funnel

Specifications

Funnel Cone

Length	12.0 in. (305 mm)
Diameter	6.0 in. (152 mm)
Capacity to bottom of screen	1500 cm ³

Orifice

Length	2.0 in. (50.8 mm)
Inside Diameter	$\frac{7}{16}$ in. (4.7 mm)

Screen

12 mesh
Has $\frac{1}{16}$ in. (1.6 mm) openings and is fixed at a level $\frac{3}{4}$ in. (19.0 mm) below top of funnel.

b. Graduated cup: one-quart

c. Stopwatch

d. Thermometer: 32-220°F ± 2 (0-105°C ± 1)

4.3 Procedure

a. Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.

b. Move finger covering orifice and start stopwatch. Measure the time for mud to fill to one-quart (946 cm³) mark of the cup.

c. Measure temperature of fluid in degrees F (C).

d. Report the time to nearest second as Marsh Funnel viscosity. Report the temperature of fluid to nearest degree F (C).

DIRECT-INDICATING VISCOMETER

4.4 Equipment

a. Direct-indicating viscometers are rotational types of instruments powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity (rpm). The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob. Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 rpm and 600 rpm.

Specifications — Direct-Indicating Viscometer

Rotor Sleeve

Inside diameter	1.450 in. (36.83 mm)
Total length	3.425 in. (87.00 mm)
Scribed line	2.30 in. (58.4 mm)
above the bottom of sleeve. Two rows of $\frac{1}{8}$ in. (3.18 mm) holes spaced 120 deg (2.09 radians) apart, around rotor sleeve just below scribed line.	

Bob

Diameter	1.358 in. (34.49 mm)
Cylinder length	1.496 in. (38.00 mm)
Bob is closed with a flat base and a tapered top.	

Torsion Spring Constant

386 dyne-cm/degree deflection

Rotor Speeds

High speed	600 rpm
Low speed	300 rpm

The following are types of viscometers used to test drilling fluids:

1. Hand-cranked instrument (Fig. 4.2) has speeds of 300 rpm and 600 rpm. A knob on the hub of the speed-change lever is used to determine gel strength.
2. The 12-volt, motor driven instrument (Fig. 4.3) also has speeds of 300 and 600 rpm. A governor-release switch permits high shearing before measurement, and a knurled hand-wheel is used to determine gel strength.
3. The 115-volt instrument (Fig. 4.4) is powered by a two-speed synchronous motor to obtain speeds of 3, 6, 100, 200, 300 and 600 rpm. The 3-rpm speed is used to determine gel strength.
4. The variable speed 115-volt or 240-volt instrument (Fig. 4.5) is motor powered to obtain all

speeds between 1 and 625 rpm. The 3-rpm speed is used to determine gel strength.

b. Thermostatically controlled viscometer cup: (Figs. 4.6 and 4.7).

c. Stopwatch

d. Thermometer: $32\text{-}220^{\circ}\text{F} \pm 2$ ($0\text{-}105^{\circ}\text{C} \pm 1$)

4.5 Procedure

CAUTION: *Maximum operating temperature is 200°F (93°C). If fluids above 200°F (93°C) are to be tested, a solid metal bob or a hollow metal bob, with a completely dry interior, should be used. Liquid trapped inside a hollow bob may vaporize when immersed in high temperature fluid and cause the bob to explode.*

a. Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume in the cup for the displacement of the viscometer bob and sleeve. The bob and sleeve will displace approximately 100 cm³ of drilling fluid. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of sampling. Test should be made at either $120^{\circ}\text{F} \pm 2$ ($50^{\circ}\text{C} \pm 1$) or $150^{\circ}\text{F} \pm 2$ ($65^{\circ}\text{C} \pm 1$). The place of sampling should be stated on the report.

b. Heat or cool the sample to the selected temperature. Intermittent or constant shear at the 600 rpm speed should be used to stir the sample while heating or cooling to obtain a uniform sample temperature. After the cup temperature has reached the selected temperature, immerse the thermometer into the sample and continue stirring until the sample reaches the selected temperature. Record the temperature of the sample.

c. With the sleeve rotating at 600 rpm, wait for dial reading to reach a steady value (the time required is dependent on the mud characteristics). Record the dial reading for 600 rpm.

d. Shift to 300 rpm and wait for dial reading to reach steady value. Record the dial reading for 300 rpm.

e. Stir drilling fluid sample for 10 seconds at high speed.

f. Allow mud to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum

reading is the initial gel strength. For instruments having a 3-rpm speed, the maximum reading attained after rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10 sec gel) in lb/100 ft² (Pa).

g. Restir the mud at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in Par. 3.5f and report the maximum reading as 10 minute gel in lb/100 ft² (Pa).

4.6 Calculation

$$\text{Plastic Viscosity, cp} = \left[\begin{matrix} 600 \text{ rpm} \\ \text{reading} \end{matrix} \right] - \left[\begin{matrix} 300 \text{ rpm} \\ \text{reading} \end{matrix} \right] \tag{a}$$

$$\text{Yield Point, lb/100 ft}^2 = \left[\begin{matrix} 300 \text{ rpm} \\ \text{reading} \end{matrix} \right] - \text{Plastic Viscosity} \tag{b}$$

$$\text{Apparent Viscosity, cp} = \frac{600 \text{ rpm reading}}{2} \tag{c}$$



FIG. 4.1
MARSH FUNNEL AND CUP

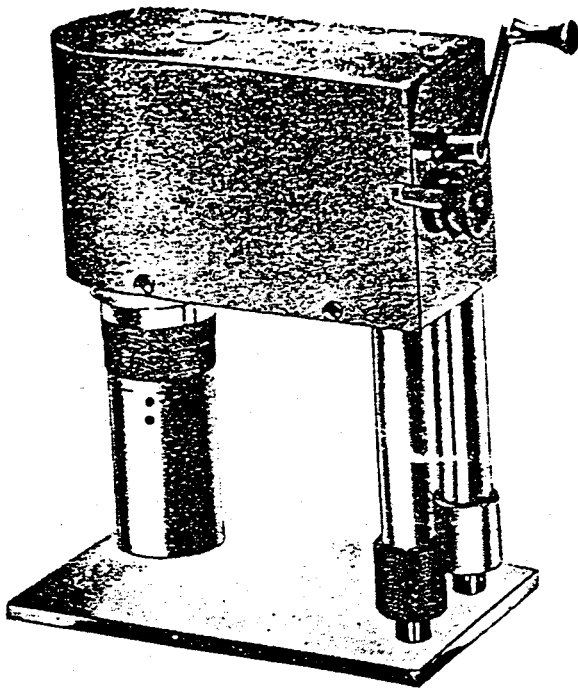


FIG. 4.2
BAROID-FANN HAND-CRANK
VISCOMETER

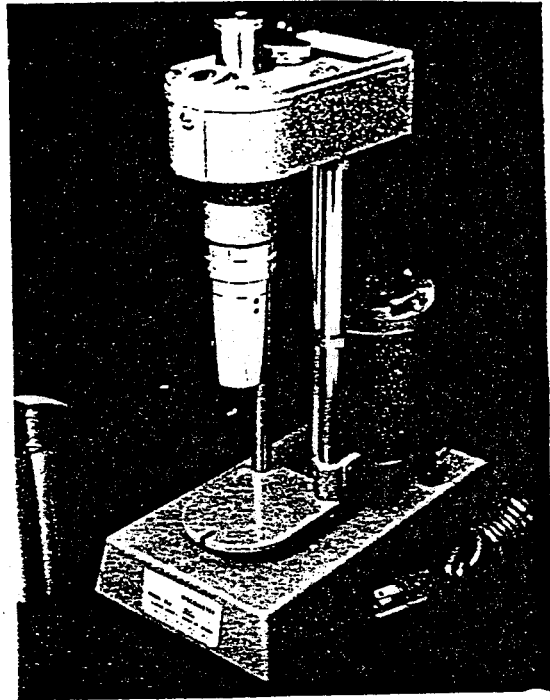


FIG. 4.4
FANN 35 115-VOLT MOTOR-DRIVEN
VISCOMETER

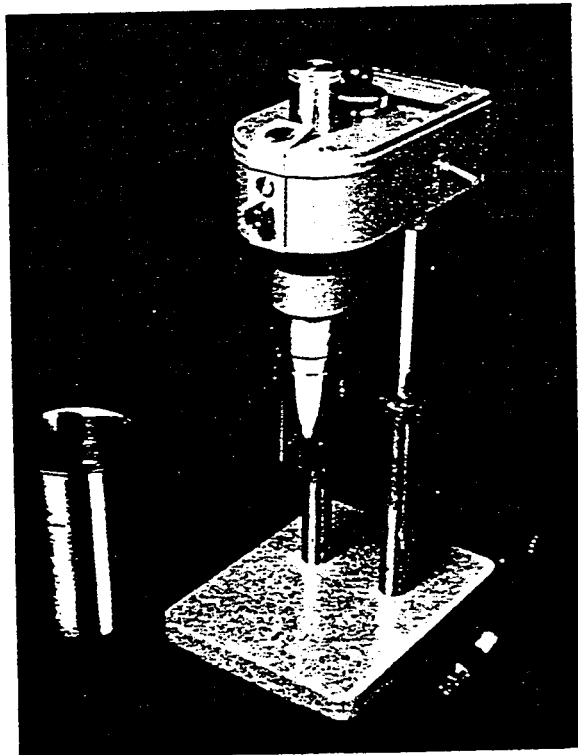


FIG. 4.3
FANN 34C 12-VOLT
VISCOMETER

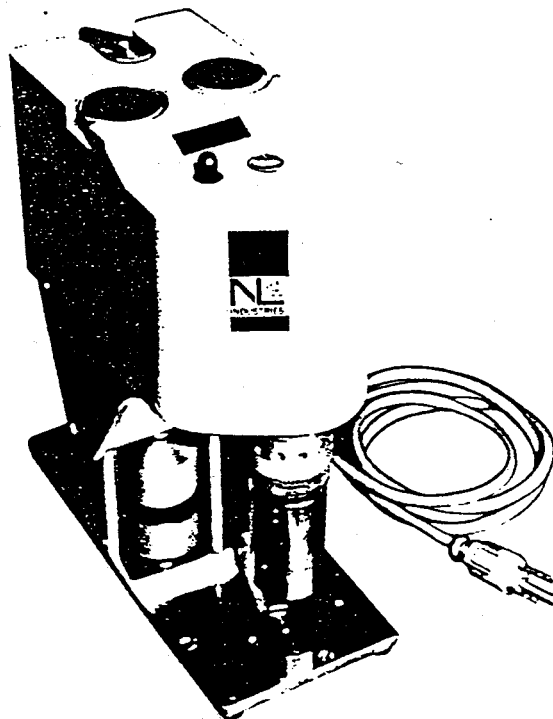


FIG. 4.5
BAROID VARIABLE SPEED VISCOMETER

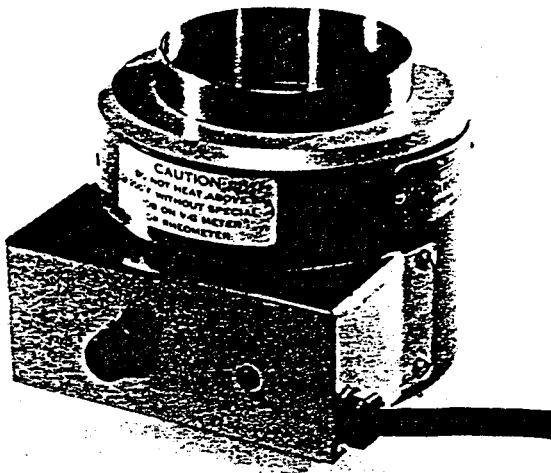


FIG. 4.6
BAROID HEATED JACKET WITH CUP

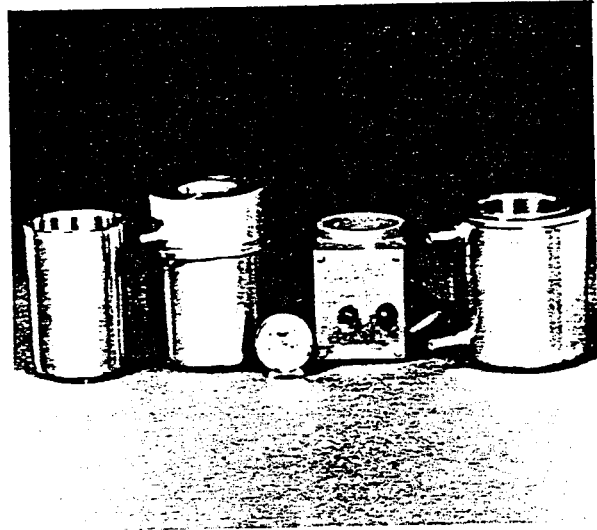


FIG. 4.7
FANN THERMOCUP

SECTION 5 CHEMICAL ANALYSIS OF OIL MUDS

5.1 Description

a. The whole mud alkalinity test procedure is a titration method which measures the volume of standard acid required to react with the alkaline (basic) materials in an oil mud sample. The alkalinity value is used to calculate the pounds per barrel unreacted "excess" lime in an oil mud. Excess alkaline materials, such as lime, help to stabilize the emulsion and also neutralize carbon dioxide or hydrogen sulfide acidic gases.

b. The whole mud chloride test procedure is a titration method which measures the volume of standard silver nitrate required to react with the chloride (and other halide) ions to form insoluble silver chloride (or silver halide) salts. The test procedures can utilize the same sample as the alkalinity test provided the sample is acidic (pH below 7.0). The chloride value reported in the whole mud can be assigned to the water phase up to the saturation point. The water soluble salt concentration is related to the effectiveness of an oil mud in controlling shale through the "aqueous phase activity" concept. The water phase salinity value is also needed to adjust the retort water value to obtain a corrected solids content for the oil mud.

5.2 Equipment

a. Solvent: 50/50 volumetric mixture of xylene and isopropanol (technical grade or better)
CAUTION: XYLENE — flammable, vapor harmful

b. Titration vessel: pint jar or 400-cm³ beaker

c. Phenolphthalein indicator solution: 1 g/100 cm³ of 50% isopropanol/water solution

d. Sulfuric acid solution: standardization 0.1 normal (N/10)

e. Potassium chromate indicator solution: 5 g/100 cm³ of water

f. Silver nitrate reagent: containing 47.91 g/L (equivalent to 0.01 g chloride-ion/cm³) (0.282 N), stored in an amber or opaque bottle

g. Distilled or deionized water

h. Disposable syringe: one 5-cm³

i. Graduated cylinder (TC): one 25-cm³

j. Serological (graduated) pipettes (TD): two 1-cm³ and two 10-cm³

NOTE: One pair of pipettes for sulfuric acid and one pair for silver nitrate

k. Magnetic stirrer with 1.5-inch (38-mm) stirring bar (coated)

5.3 Procedure — Whole Mud Alkalinity

a. Add 100 cm³ of 50/50 xylene/isopropanol solvent to a 400-cm³ beaker or pint jar.

b. Fill a 5-cm³ syringe with whole mud to past the 3 cm³ mark.

c. Displace 2.0 cm³ of whole mud into the beaker or pint jar.

d. Swirl the oil mud and solvent until the mixture is homogenous.

e. Add 200 cm³ of distilled (or deionized) water.

f. Add 15 drops of phenolphthalein indicator solution.

g. While stirring rapidly with a magnetic stirrer, slowly titrate with 0.1 N sulfuric acid until pink color just disappears. Continue stirring and if no pink color reappears within one minute, stop stirring.

NOTE: It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to visually see the indicator in the aqueous phase.

h. Let sample stand for five minutes. If no pink color reappears, the end point has been reached. If a pink color returns titrate a second time with sulfuric acid. If pink color returns, titrate a third time. If after the third titration, pink color returns, call this the end point.

i. Use the cm³ of 0.1 N sulfuric acid solution required to reach the end point in Par. 5.3h to calculate the whole mud alkalinity.

5.4 Calculation — Whole Mud Alkalinity

$$\begin{aligned} \text{Alkalinity (whole mud), } P_{om} &= \frac{0.1 \text{ N sulfuric acid, cm}^3}{\text{mud sample, cm}^3} \\ &= \frac{0.1 \text{ N sulfuric acid, cm}^3}{2.0 \text{ cm}^3} \quad (a) \end{aligned}$$

5.5 Procedure — Whole Mud Chloride

a. Perform the Alkalinity procedures in Par. 5.3a through h.

NOTE: Make sure the mixture to be titrated for chloride is acidic (below pH 7.0) by adding 1-2 drops of 0.1 N sulfuric acid.

b. Add 10-15 drops potassium chromate indicator solution.

c. While stirring rapidly with a magnetic stirrer, slowly titrate with 0.282 N silver nitrate reagent until a

salmon pink color remains stable for at least one minute.

NOTES: 1) Additional potassium chromate indicator solution may be required during titration process.

2) It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to visually see the indicator in the aqueous phase.

d. Use the cm^3 of 0.282 N silver nitrate reagent required to reach the end point in Par. 5.5 c to calculate the whole mud chloride.

5.6 Calculation — Whole Mud Chloride

$$\begin{aligned} \text{Chloride (whole mud). } Cl_{\text{wm}} &= \frac{\text{Cl. mg}}{\text{oil mud sample. L}} \\ &= \frac{10.000 (0.282 \text{ N silver nitrate. cm}^3)}{2.0 \text{ cm}^3} \quad (\text{b}) \end{aligned}$$

APPENDIX A

CHEMICAL ANALYSIS OF WATER BASE DRILLING FLUIDS

CALCIUM

A.1 Description When EDTA (ethylenediaminetetraacetic acid or its salt) is added to water or mud filtrate containing both calcium and magnesium, it combines first with calcium. Calcium can be determined with EDTA when the pH of the sample is sufficiently high, so that magnesium is precipitated as the hydroxide, and an indicator specific for calcium is used. Several indicators will give color changes when all of the calcium has been complexed by EDTA at a pH of 12-13. An end point obscured by dark organic components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

A.2 Equipment

a. EDTA solution (Versenate): 0.01 molar. EDTA: disodium (ethylenediamine) tetraacetate dihydrate standardized (1 cm³ = 1000 mg/L CaCO₃, 1 cm³ = 400 mg/L calcium).

b. Calcium buffer solution: 1N sodium hydroxide (NaOH)

c. Calcium indicator: Calver® II or hydroxy naphthol blue

d. Acetic acid: glacial

CAUTION: *Avoid skin contact.*

e. Titration vessel: 150-cm³ beaker

f. Serological (graduated) pipettes (TD): two 10-cm³ and one 1-cm³

g. Volumetric pipettes (TD): one 1-cm³, one 2-cm³, and one 5-cm³

h. Hot plate (required if filtrate colored)

i. Masking agent: 1:1:2 mixture by volume of triethanolamine: tetraethylenepentamine: deionized water (optional)

j. pH paper

k. Graduated cylinder (TC): 50-cm³

l. Sodium hypochlorite solution: 5.25% sodium hypochlorite in deionized water (i.e., Clorox®, or equivalent).

CAUTION: *Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure sodium hypochlorite is fresh as it will deteriorate with time.*

m. Deionized or distilled water

NOTE: *The deionized water and sodium hypochlorite solution should be tested for calcium by using 50.0 cm³ of the deionized water in Par. A.3f and 10 cm³ of the hypochlorite in Par. A.3h, without the test sample, and continuing with Par. A.3g and A.3h. If the procedure is then repeated with the test sample utilizing 50.0 cm³ of the deionized water and 10 cm³ of the hypochlorite in Par. A.3f, A.3g, and A.3h, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and hypochlorite.*

A.3 Procedure

a. With a volumetric pipette, add 1.0 cm³ or more of sample to a 150-cm³ beaker. This sample volume will be used in the calculation shown in A.4. If filtrate is colorless or is only slightly colored, omit steps A.3b through A.3e.

b. With serological pipette, add 10 cm³ hypochlorite solution and mix.

c. With serological pipette, add 1 cm³ glacial acetic acid and mix.

d. Boil the sample for five minutes. Maintain the sample by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required. A sufficiently boiled sample will show a pH of 5.0.

CAUTION: *Work in an adequately ventilated area.*

e. Cool the sample and wash the sides of the beaker with deionized water.

f. Dilute the sample to approximately 50 cm³ with deionized water. Add 10-15 cm³ of NaOH buffer solution, or sufficient NaOH to produce of pH of 12-13.

NOTE: *The presence of soluble iron may interfere with the endpoint determination. Should this be suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume, respectively) is a suitable masking agent. Add 1.0 cm³ of the mixture after A.3f.*

g. Add sufficient calcium indicator (0.1-0.2 g) to produce a pink to wine-red color if calcium is present. Too much indicator will obscure the endpoint.

NOTE: *The addition of several drops of methyl orange along with the calcium indicator may improve the visibility of the endpoint.*

h. While stirring, titrate with standard EDTA to the proper endpoint. Calcium indicators will produce a red to blue change. The endpoint is best described as that point where additional EDTA produces no further red to blue color change. The EDTA volume will be used in the calculation in Par. A.4.

A.4 Calculation

$$\text{Calcium, mg/L} = \frac{400 (\text{EDTA volume, cm}^3)}{\text{volume sample, cm}^3} \quad (a)$$

MAGNESIUM

A.5 Description The magnesium content of the mud filtrate can be calculated by subtracting the calcium ion content from the total hardness. This gives the magnesium content in terms of calcium which is converted to magnesium by multiplying the value by the ratio of atomic weights (24.3/40 = 0.6).

A.6 Procedure

- a. Determine the total hardness as calcium (Par. 8.18 through 8.21).
- b. Determine the calcium content (Par. A.1 through A.4).

A.7 Calculation

$$\text{Magnesium, mg/L} = (0.6) \frac{(\text{Total Hardness, mg/L} - \text{Calcium, mg/L})}{\text{mg/L}} \quad (\text{a})$$

CALCIUM SULFATE

A.8 Description The calcium sulfate content of mud is determined by using the EDTA method as described in Par. A.1 through A.4 to determine the total calcium in a mud filtrate and the whole mud. The total and undissolved calcium sulfate contents of the mud can then be calculated.

A.9 Equipment

a. EDTA solution (Versenate): 0.01 molar EDTA: disodium (ethylenediamine)tetraacetate dihydrate standardized. (1 cm³ = 1000 mg/L CaCO₃, 1 cm³ = 400 mg/L Ca⁻²)

b. Buffer solution: 1N sodium hydroxide (NaOH)

c. Calcium indicator: Calver® II or equivalent, or hydroxy naphthol blue

d. Acetic acid: glacial

CAUTION: Avoid skin contact.

e. Titration vessel: 150-cm³ beaker

f. Serological (graduated) pipettes (TD): two 10-cm³ and one 1-cm³

g. Volumetric pipettes (TD): one 1-cm³, one 2-cm³, one 5-cm³, one 10-cm³

h. Hot plate (required, if filtrate colored)

i. Masking agent: 1:1:2 mixture by volume of triethanolamine:tetraethylenepentamine:deionized water (optional)

j. pH paper

k. Graduated cylinder (TC): 50-cm³

l. Mud retort

m. Sodium hypochlorite solution: 5.25% sodium hypochlorite in deionized water (i.e., Clorox® or equivalent)

CAUTION: Many brands contain calcium hypochlorite or oralic acid and should not be used. Be sure sodium hypochlorite is fresh as it will deteriorate with time.

n. Deionized or distilled water

NOTE: The deionized water and sodium hypochlorite solution should be tested for calcium by using 10.0 cm³ of the deionized water in Par. A.10a and 10 cm³ of the hypochlorite in Par. A.10a, without the test sample, and continuing with Par. A.10a through A.10c. If the procedure is then repeated with the test sample utilizing 10.0 cm³ of the deionized water and 10 cm³ of the sodium

hypochlorite in Par. A.10a, A.10b, and A.10c, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and sodium hypochlorite.

A.10 Procedure

a. Add 5 cm³ of whole mud to 245 cm³ of deionized water. Stir the mixture for 15 minutes and filter through a standard API filter press. Collect only clear filtrate. Into a 150-cm³ beaker, add 10 cm³ of clear filtrate with the 10-cm³ volumetric pipette and titrate to the EDTA endpoint as described in Par. A.1 through A.4. Call this volume of EDTA V_t.

b. Titrate 1 cm³ of the original mud filtrate (obtained as described in Par. 3.1 through 3.3) to the EDTA endpoint. Call this volume of EDTA V_f.

c. Retort the mud as described in WATER BASE DRILLING FLUIDS Section 4. Determine the volume fraction of water in the mud, F_w, by using the value for volume percent water from the liquid and solids determination and the following equation:

$$F_w = \frac{\text{Volume percent water}}{100} \quad (\text{a})$$

A.11 Calculation

a. The calcium sulfate content of the mud in lb/bbl is calculated by using the following equation:

$$\text{Total calcium sulfate, lb/bbl} = 2.38 (V_t) \quad (\text{a})$$

b. The (excess) undissolved calcium sulfate content of the mud in lb/bbl may be calculated by using the subsequent equation:

$$\text{Excess calcium sulfate, lb/bbl} = 2.38 (V_t) - 0.48 (V_f F_w) \quad (\text{b})$$

FORMALDEHYDE

A.12 Description This procedure is for the determination of paraformaldehyde content of drilling fluid. Sodium sulfite is reacted with a filtrate sample (neutralized to a phenolphthalein endpoint) which is then titrated with the acid to the phenolphthalein endpoint again. A blank must be run to subtract the contribution to alkalinity attributable to sodium sulfite. The difference in two titrations is the amount of paraformaldehyde present in the drilling fluid.

A.13 Equipment

a. Phenolphthalein indicator: 1 g/100 cm³ of 50% alcohol/water

b. Sodium hydroxide solution: 0.02 normal (N/50)

c. Sulfuric acid solution: 0.02 normal (N/50)

d. Sodium sulfite solution: 4 g/100 cm³ of distilled water

NOTE: This solution deteriorates rapidly. If older than 30 days, it should be replaced with a fresh solution.

e. Titration vessel: a test tube or a casserole

- f. Serological (graduated) pipette (TD): 10-cm³
 g. Volumetric pipettes (TD): one 1-cm³ and one 3-cm³

A.14 Procedure

a. Pipette 3 cm³ of the mud filtrate into the casserole or test tube. Add 2 drops of phenolphthalein indicator solution. If the sample remains colorless, add sodium hydroxide solution drop by drop, with agitation, until a faint pink color develops. Then add sulfuric acid drop by drop to dispel the color.

b. If, upon the first addition of phenolphthalein, the filtrate becomes colored, add sulfuric acid dropwise until the color is just dispelled.

c. To the neutralized filtrate, add 1 cm³ of sodium sulfite solution: a red color will develop.

d. After approximately 30 seconds, titrate with sulfuric acid until the sample is a very faint pink. Record the amount of acid used in cm³. This is the amount of sulfuric acid which will be used for the calculation, and noted as V_f .

e. Repeat the foregoing Par. A.14c through A.14d using distilled water instead of mud filtrate. Record the amount of acid used in cm³. This will be used for the calculation in A.15, and noted as V_b .

A.15 Calculation

$$\text{Formaldehyde content,} = 0.07 (V_f - V_b) \quad (\text{a})$$

lb/bbl

SULFIDE

A.16 Description

a. The concentration of soluble sulfides in a drilling fluid can be determined by this method. Soluble sulfides include H₂S and the sulfide (S⁻²) and bisulfide (HS⁻) ions. Mud filtrate is acidified in a Garrett Gas Train, converting all sulfides to H₂S which is evolved by bubbling an inert carrier gas through the sample. The Gas Train separates the gas from the liquid. The gas stream is passed through a Dräger tube which responds to H₂S by darkening along its length. The darkened length is proportional to the total sulfide in the mud filtrate. The low range Dräger tube turns from white to brownish-black and the high range Dräger tube turns from pale blue to jet-black. No common mud contaminant will cause these color changes.

b. Lead-acetate paper disks can be accommodated in the Garrett Gas Train to determine the presence or absence of sulfide. If the presence of sulfide is indicated by darkening of the lead-acetate paper, a Dräger tube should be used for quantitative analysis.

A.17 Equipment

a. The Garrett Gas Train apparatus, as shown in Fig. A.1, consists of: a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball

flowmeter and a Dräger tube. Specifications of Garrett Gas Train:

Body

Chamber 1

Depth 3.54 in. (90 mm)
 Diameter 1.52 in. (38 mm)

Chambers 2 & 3

Depth 3.54 in. (90 mm)
 Diameter 1.18 in. (30 mm)

Passages between Chambers:

Diameter 0.08 in. (2.0 mm)

Material:

Lucite® or equivalent transparent material or glass which is inert to acid, sulfides, and hydrogen sulfide gas.

Dispersion Tube

Stem:

Diameter 0.315 in. (8.0 mm)
 Length approx. 5.9 in. (150 mm)

Dispersion Frit (Bell-shaped ASTM 4-5.5 Fine)

Diameter 1.18 in. (30 mm)
 Pyrex® or equivalent glass

Flow Meter: Floating ball type preferred, capable of measuring 300 cm³ per minute of CO₂ gas.

Flexible Tubing: Type inert to hydrogen sulfide and carrier gas. Latex rubber or Tygon® plastic or equivalent is preferred.

Fittings and Rigid Tubing: Type inert to hydrogen sulfide and acid.

b. Carrier gas: type inert to hydrogen sulfide, acid, and Dräger tube reagents. Nitrogen is preferred but carbon dioxide is acceptable. (Avoid air or other oxygen-containing gases.)

c. Dräger H₂S Analysis Tubes:

- (1) Low range: marked H₂S 100/a (No. CH-291-01)
- (2) High range: marked H₂S 0.2%/A (No. CH-281-01)

d. Lead-acetate paper disk (see Note Par. A.18o)

e. Sulfuric acid: approximately 5N, reagent grade

f. Octanol defoamer in a dropper bottle

g. Hypodermic syringes: one 10-cm³ (for acid), and one 2.5-cm³, one 5-cm³ and one 10-cm³ (for sample)

h. Hypodermic needles: two 1.5 in. (38 mm) 21-gauge needles

A.18 Procedure

a. Be sure the gas train is clean, dry and on a level surface, with the top removed.

NOTE: Moisture in the train can cause the ball in the flowmeter to float erratically and may affect the accuracy of the Dräger tube reading.

- b. Add 20 cm³ of deionized water to Chamber 1.
- c. Add 5 drops octanol defoamer to Chamber 1.

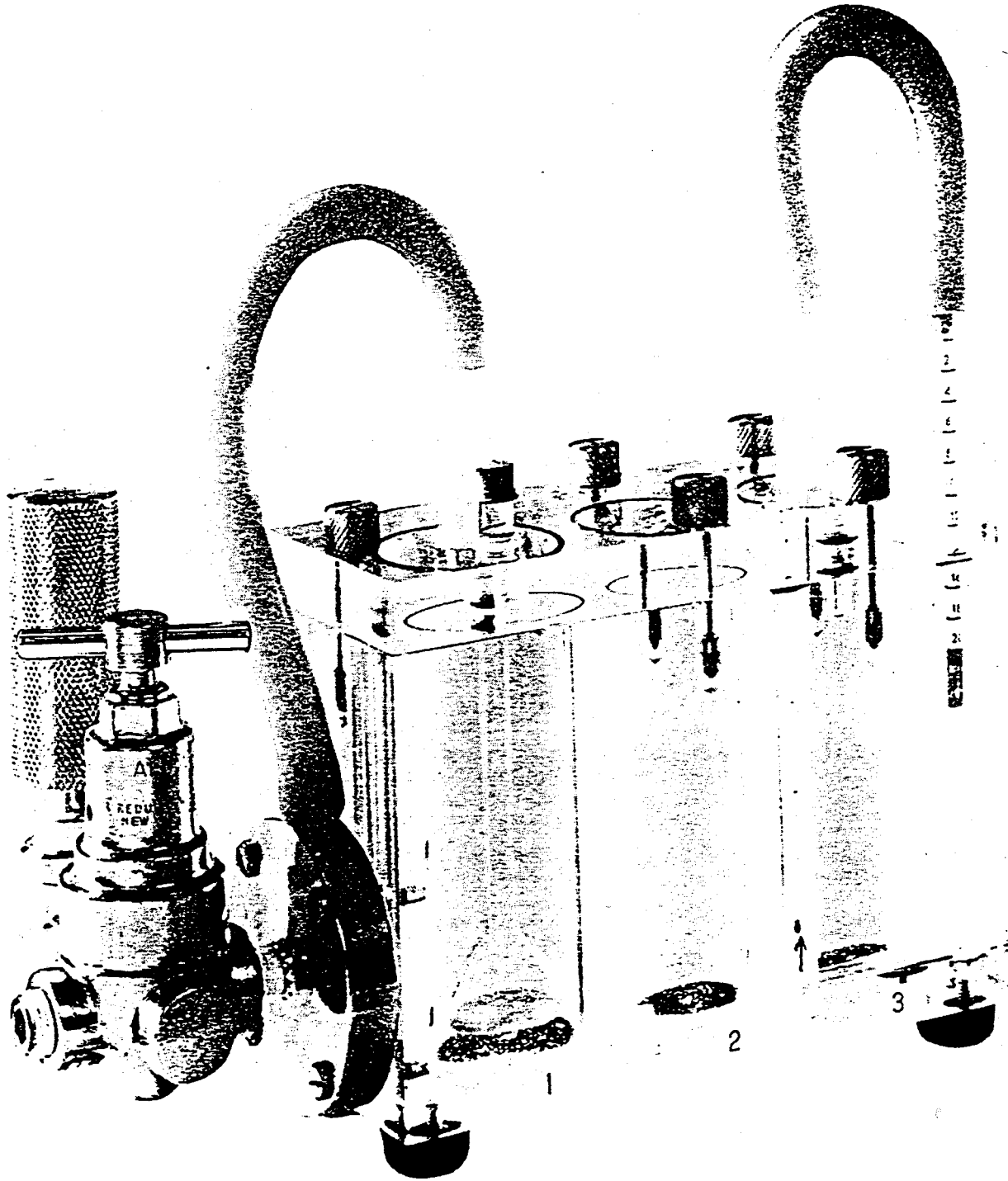


FIG. A.1
ANALYSIS OF SOLUBLE SULFIDES

d. See Table A.1 for sample volume and type of Dräger tube required for the expected sulfide range. Select the proper type Dräger tube. Break the tip from each end of the tube.

e. Install the Dräger tube with the arrow pointing downward into the bored receptacle. Likewise, install the flowmeter tube with the word TOP upward. Be sure o-rings seal around the body of each tube.

TABLE A.1
DRÄGER TUBE IDENTIFICATION, SAMPLE VOLUMES AND TUBE FACTORS TO BE USED FOR VARIOUS SULFIDE RANGES

Sulfide Range (mg/L)	Sample Volume (cm ³)	Dräger Tube I.D. (See Tube Body)	Tube Factor (See A.19a)
1.2 - 24	10.0	H ₂ S 100/a	12
1.5 - 48	5.0		
4.8 - 96	2.5		
60 - 1020	10.0		
120 - 2040	5.0	H ₂ S 0.2%/A	600*
240 - 4080	2.5		

*"Tube Factor" 600 is based on a "Batch Factor" (stenciled on box) of 0.40. For another "Batch Factor" (as stenciled on box), a corrected "Tube Factor" should be calculated:

$$\text{Correct Tube Factor} = \frac{600 (\text{Batch Factor})}{0.40} \quad (\text{a})$$

f. Install the top on the gas train and hand tighten all screws evenly to seal the o-rings.

g. With the regulator backed off, connect the carrier gas to the dispersion tube of Chamber 1 using flexible tubing. If CO₂ cartridge is used, install and puncture cartridge and connect to dispersion tube as shown in Fig. A.1.

h. Attach the flexible tubing from Chamber 3 outlet to the Dräger tube, as shown in Fig. A.1.

NOTE: Use only latex rubber or inert plastic tubing. Do not clamp flexible tubing; unclamped tubing provides pressure relief in the event of over pressurization.

i. Adjust the dispersion tube in Chamber 1 to approximately ¼ in. (5 mm) above the bottom.

j. Gently flow carrier gas for a 30 second period to purge air from the system. Check for leaks. Shut off the carrier gas.

k. Collect a sufficient volume of solids-free filtrate for analysis. (If a low concentration of soluble sulfides is to be detected, a large volume of filtrate is required. Use Table A.1 as a guide.)

l. Inject a measured volume of the solids-free filtrate sample into Chamber 1 through the rubber septum, using a hypodermic syringe and needle.

m. Slowly inject 10 cm³ sulfuric acid solution into Chamber 1 through rubber septum using the hypodermic syringe and needle.

n. Immediately restart the carrier gas flow. The flow rate should be maintained between 200-400 cm³ per minute.

NOTE: One CO₂ cartridge should provide about 15-20 minutes of flow at this rate.

o. Observe changes in appearance of the Dräger tube. Note and record the maximum darkened length (in units marked on the tube) before the front starts to smear. Continue flowing for a total of 15 minutes although the front may attain a diffuse and feathery coloration. In the high-range tube an orange color (caused by SO₂) may appear ahead of the black front if sulfites are present in the sample. The orange SO₂ region should be ignored when recording darkened length.

NOTE: For best Dräger tube accuracy, the "Darkened Length" should fill more than half the tube's length, therefore the filtrate "Sample Volume" must be carefully selected.

p. A lead-acetate paper disk fitted under the o-ring of Chamber 3 can be substituted for the Dräger tube in the Gas Train. The lead-acetate paper will show qualitatively the presence or absence of sulfides in the sample. A dark discoloration of the paper is a positive indication of sulfides. After the positive indication, the Dräger tube should be used on a separate sample for quantitative analysis.

q. To clean the gas train remove the flexible tubing and remove the top. Take Dräger tube and flow-meter out of the receptacles and plug the holes with stoppers to keep them dry. Wash out the chambers with warm water and mild detergent, using a soft brush. Use a pipe cleaner to clean the passages between the chambers. Wash, rinse and blow out the dispersion tube with a dry gas. Rinse the unit with deionized water and allow to drain dry.

A.19 Calculation

a. Using the measured Sample Volume, the Dräger tube's maximum Darkened Length and the Tube Factor from Table A.1, calculate sulfide in the sample:

$$\text{Sulfide, mg/L} = \frac{(\text{Darkened Length}^*) (\text{Tube Factor})}{(\text{Sample Volume, cm}^3)} \quad (\text{a})$$

*In units marked on the tube

CARBONATE

A.20 Description The concentration of soluble carbonates in a drilling fluid filtrate can be determined by this method. Total soluble carbonates include CO₃ and the carbonate (CO₃⁻²) and bicarbonate (HCO₃⁻) ions. Mud filtrate is acidified in a Garrett Gas Train, converting all carbonates to CO₂, which is then evolved by bubbling an inert carrier gas through the sample. The Gas Train separates the gas from the liquid. The gas stream is collected in a one liter gas bag (to allow CO₂ to mix uniformly) and subsequently drawn through a Dräger tube at a fixed flow rate. The Dräger tube responds to CO₂ by progressively staining purple along its length. A reaction between CO₂ and a hydrazine

chemical causes a crystal violet indicator to turn purple. The stain length is proportional to the total carbonate concentration in the filtrate.

A.21 Equipment

a. Garrett Gas Train apparatus, as shown in Fig. A.2, consists of: a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flowmeter and a Dräger tube. Specifications of Garrett Gas Train:

Body

Chamber 1
 Depth 3.54 in. (90 mm)
 Diameter 1.52 in. (38 mm)

Chambers 2 & 3
 Depth 3.54 in. (90 mm)
 Diameter 1.18 in. (30 mm)

Passages between Chambers:
 Diameter 0.08 in. (2.0 mm)

Material:

Lucite® or equivalent transparent material or glass which is inert to acid, carbonates, and carbon dioxide gas.

Dispersion Tube

Stem:

Diameter 0.315 in. (8.0 mm)
 Length approximately 5.9 in. (150 mm)

Dispersion Frit: (bell-shaped ASTM 4-5.5 Fine)
 Diameter 1.18 in. (30 mm)

Pyrex® or equivalent glass

Flexible Tubing: Type inert to carbon dioxide and carrier gas. Latex rubber or Tygon® plastic or equivalent is preferred.

Fittings and Rigid Tubing: type inert to carbon dioxide and acid.

b. Carrier gas: nitrogen (N₂) bottle with low-pressure regulator (preferred), or N₂O gas cartridges (e.g., WHIPPETS #561241 by Walter Kidde and Co. Inc., Belleville, N.J.) or equivalent.

c. Dräger CO₂ analysis tube: marked CO₂ 0.01%/a (No. CH-308-01).

d. Dräger one-liter ALCOTEST gas bag No. 7626425 or equivalent.

e. Dräger MULTIGAS DETECTOR hand-operated vacuum pump, Model 31, or equivalent.

f. Stopcock (2-way bore), 8 mm glass with Teflon® plug.

g. Sulfuric Acid: approximately 5N, reagent grade.

h. Octanol defoamer in a dropper bottle.

i. Hypodermic syringes: one 10-cm³ (for acid) and one 1.0-cm³, one 5-cm³ and one 10-cm³ (for sample).

j. Hypodermic needles: two 1.5-inch (38 mm) 21-gauge needles.

NOTE: Nitrogen is preferred over N₂O as the carrier gas. Because N₂O cools upon expansion and chills the diaphragm in the regulator, prolonged N₂O flow will cause the regulator to perform erratically.

A.22 Procedure

a. Be sure that the gas train is clean, dry and on a level surface, with the top removed.

NOTE: If CO₂ has been used as the carrier gas in the previous test (i.e., sulfide analysis), the regulator, tubing and dispersion tube should be purged with carrier gas at this time.

b. Add 20 cm³ deionized water to Chamber 1.

c. Add 5 drops of octanol-defoamer to Chamber 1.

d. Install the top on the gas train and hand-tighten evenly to seal all o-rings.

e. Adjust the dispersion tube to approximately ¼ in. (5 mm) off bottom.

f. With regulator backed off, connect carrier gas supply to glass dispersion tube of Chamber 1 using flexible tubing.

g. Flow carrier gas through train for one minute to purge air from the system. Check for leaks in gas train unit.

h. Fully collapse the gas bag and simultaneously check the system for leaks. To do this, connect the gas bag and stopcock to hand pump, as shown in Fig. A.3. (Use a discarded Dräger tube as connection and start with the bag essentially empty.) Fully depress and release the hand pump. When the bag is completely empty and free of leaks, the pump will remain depressed for several minutes. If leakage is detected, check the pump and all connections. To check the pump alone, insert a sealed Dräger tube into the pump opening and depress bellows. It will remain depressed if pump does not leak.

i. With the bag fully collapsed, install flexible tubing from the stopcock and bag onto the outlet of Chamber 3, as seen in Fig. A.2.

j. Inject a measured volume of solids-free filtrate into Chamber 1 through septum with a hypodermic syringe and needle. See Table A.2.

TABLE A.2
DRÄGER TUBE IDENTIFICATION, SAMPLE VOLUMES AND TUBE FACTOR TO BE USED FOR VARIOUS CARBONATE RANGES

Carbonate Range (mg/L)	Sample Volume (cm ³)	Dräger Tube Identification (see Tube Body)	Tube Factor (see A.23a)
25 - 750	10.0		
50 - 1500	5.0	CO ₂ 0.01%/a	25,000
250 - 7500	1.0		

k. Slowly inject 10 cm³ sulfuric acid solution into Chamber 1 through rubber septum using a clean syringe and needle. Gently shake the gas train to mix acid with sample in Chamber 1.

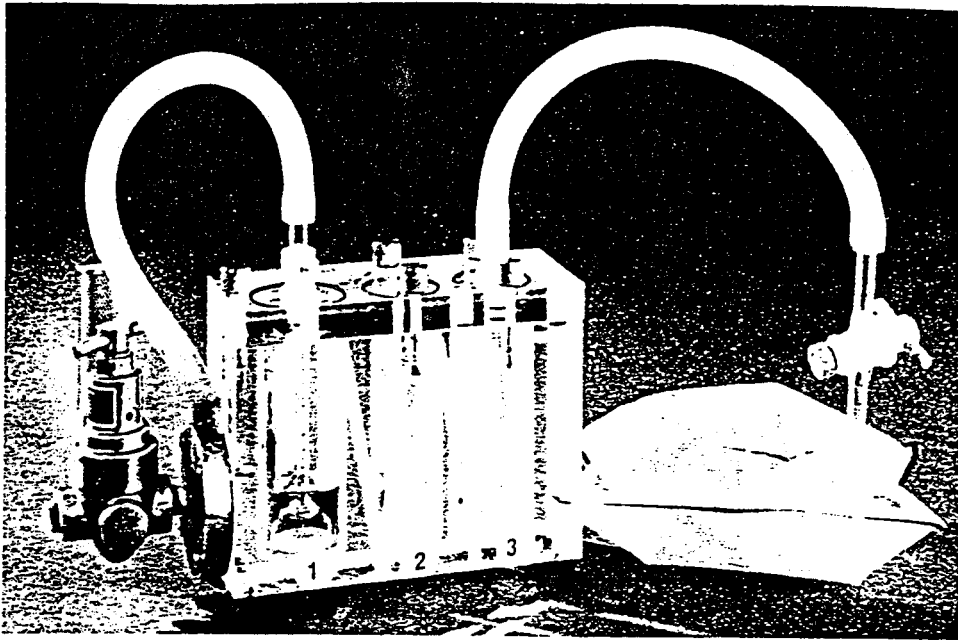


FIG. A.2
Step 1 — Freeing Carbonates as CO₂ Gas

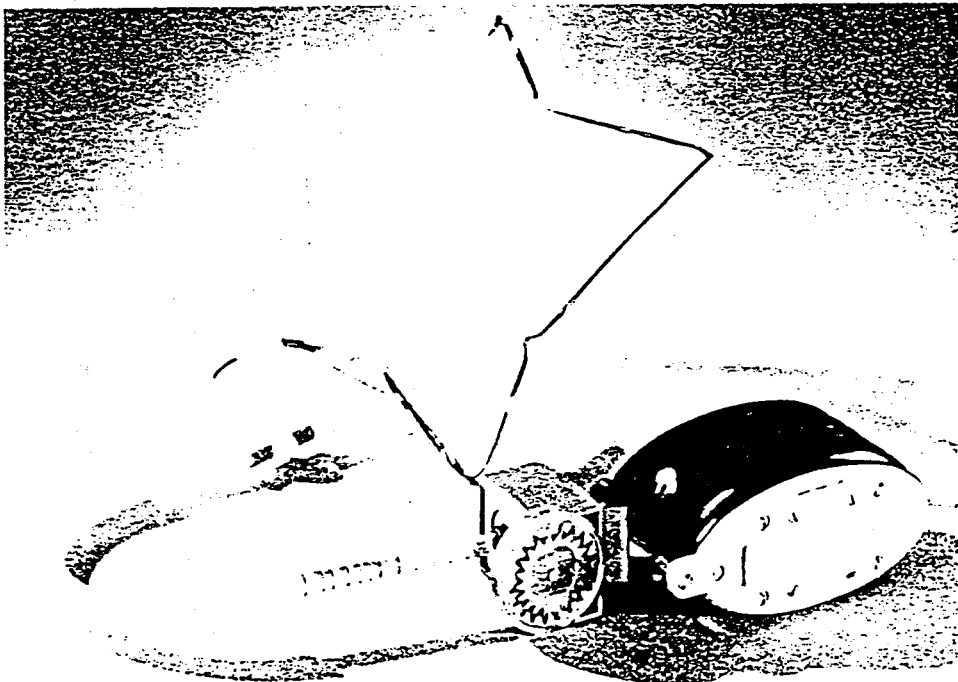


FIG. A.3
Step 2 — Analyzing CO₂ with Dräger Tube
ANALYSIS OF SOLUBLE CARBONATE

l. Open the stopcock on the gas bag. Restart gas flow and allow gas bag to fill steadily during a 10 minute interval. When bag is firm to the touch (do not burst it) shut off flow and close the stopcock. Immediately proceed to next step.

m. Break the tip off each end of the Dräger tube.

n. Remove the tubing from Chamber 3 outlet and reinstall it onto the upstream end of the Dräger tube. (Observe that an arrow on the tube indicates gas flow direction.) Attach Dräger hand pump to downstream end of Dräger tube, as shown in Fig. A.3.

o. Open the stopcock on the bag. With steady hand-pressure fully depress the hand pump. Release pump so that gas flows out of the bag and through the Dräger tube. Operate the pump and count the strokes until the bag is empty. (Ten strokes should empty the bag. More than ten strokes indicates leakage has occurred and test results will not be correct.)

p. Observe a purple stain on the Dräger tube if CO_2 is present in the gas bag and record the stain length in units marked on the Dräger tube. (Include the faint blue tinge in the purple stain length reading.)

NOTE: For best Dräger tube accuracy, the "Stain Length" should fill more than half the tube length, therefore "Sample Volume" must be carefully selected.

q. To clean the gas train remove the flexible tubing and remove the top. Wash out the chambers with warm water and mild detergent, using a brush. Use a pipe cleaner to clean the passages between chambers. Wash, rinse and then blow out the dispersion tube with dry gas. Rinse the unit with deionized water and allow to drain dry. Be sure to periodically replace the disposable gas bag to avoid leaks and contamination in the bag. (Bag replacement is suggested after 10 analyses.)

A.23 Calculation

a. Using the measured "Sample Volume," the Dräger tube "Stain Length" and "Tube Factor" of 25,000 (see Table A.2), calculate total soluble carbonates ($\text{CO}_2 + \text{CO}_3^{-2} + \text{HCO}_3^{-}$) in the filtrate sample using equation:

$$\text{Carbonate, mg/L} = \frac{25,000 \cdot (\text{Stain Length})}{\text{Sample Volume, cm}^3} \quad (\text{a})$$

POTASSIUM

A.24 Description Potassium ion is used in drilling fluids to aid in the stabilization of shales and to control swelling clays. The accurate determination of the potassium ion content is necessary to control the properties of the drilling fluid. This procedure is used to measure the potassium ion content in mud filtrates at levels above 5000 mg/L or 3.5 lb/bbl KCl. Potassium is precipitated in a centrifuge tube as the perchlorate salt, and then the precipitate volume is measured. The potassium ion content is read from a prepared standard curve.

A.25 Equipment

a. Sodium perchlorate solution: 150.0 g $\text{NaClO}_4/100 \text{ cm}^3$ distilled water.

NOTE: Sodium and potassium perchlorates are explosive in the dry state if heated or if in contact with organic reducing agents. The perchlorates are not hazardous if kept wet. They will decompose harmlessly in water.

b. Standard potassium chloride solution: 14.0 g KCl made up to 100 cm^3 with deionized or distilled water

c. Centrifuge, horizontal-swing rotor head (manual or electric) capable of producing approximately 1800 rpm (see Fig. A.4)

NOTE: A fairly constant 1800 rpm can be obtained with a manual centrifuge as follows:

1. Determine the number of revolutions of the rotor per each turn of the crank; i.e., move the crank very slowly and count the number of revolutions of the rotor head during one turn of the crank. For example, 15 revolutions of the rotor per one turn of the crank.
2. Calculate the number of crank turns required to obtain 1800 revolutions of the rotor head. In the example, to obtain 1800 revolutions of the head would require 120 turns of the crank ($1800/15$). Thus the crank must be turned 120 times in one minute to obtain the rate of 1800 rpm. At this rate, in 5 seconds the handle must be turned 10 times ($120/60$) (5). By counting the crank turns in 5 seconds and adjusting the rate to obtain the required number of turns, a constant 1800 rpm should be obtained in 15-20 seconds. The interval used to adjust to the 1800 rpm should be included in the centrifuge time of the sample.

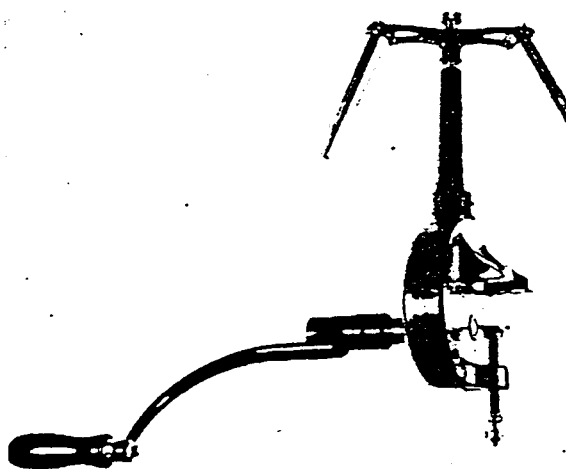


FIG. A.4
MANUAL CENTRIFUGE
WITH HORIZONTAL-SWING ROTOR HEAD

d. Clinical centrifuge tube: 10-cm³ Kolmer type (do not substitute), e.g., Corning #8360 (see Fig. A.5)

e. Volumetric pipettes (TD): one 0.5-cm³, one 1.5-cm³, one 2.5-cm³, and one 3-cm³

f. Hypodermic syringe or serological (graduated) pipette (TD): 10-cm³

g. Distilled or deionized water

A.26 Procedure — Standard Calibration Curve

A standard calibration curve is required for each type of centrifuge. A minimum of three points (3.5, 10.5 and 17.5 lb/bbl KCl) is required to obtain an accurate graph.

a. Samples can be prepared by using the standard potassium chloride solution (0.5 cm³ of standard potassium chloride solution is equivalent to 3.5 lb/bbl KCl). To obtain 3.5, 10.5, and 17.5 lb/bbl KCl, use 0.5, 1.5 and 2.5 cm³ of the standard potassium chloride solution, respectively.

b. Dilute the sample to the 7.0 cm³ mark with distilled water and agitate.

c. Add 3.0 cm³ of standard sodium perchlorate solution. (DO NOT AGITATE.)

d. Centrifuge at a constant speed (approximately 1800 rpm) for one minute and read the precipitate volume immediately.

CAUTION: Counterbalance the centrifuge tube with another tube and liquid of the same weight.

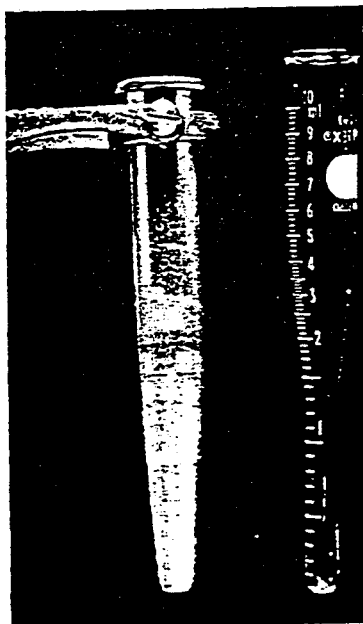


FIG. A.5
10-cm³ CLINICAL CENTRIFUGE TUBE
KOLMER TYPE

e. Clean the centrifuge tube immediately after use to facilitate ease of cleaning.

f. Plot the volume of precipitate (cm³) versus potassium chloride content (lb/bbl) on rectangular graph paper as shown in Fig. A.6.

A.27 Procedure — Sample Testing

a. Measure the appropriate volume of filtrate into the centrifuge tube (see Table A.3 for range).

b. Dilute to 7.0 cm³ if necessary with distilled water, and agitate.

c. Add 3.0 cm³ of standard sodium perchlorate solution. (DO NOT AGITATE.) If potassium is present, precipitation occurs at once.

d. Centrifuge at constant speed (approximately 1800 rpm) for one minute. Read the precipitate volume immediately and record.

NOTE: Counterbalance the centrifuge tube with another tube and liquid of the same weight.

e. Add 2-3 drops of the sodium perchlorate solution to the tube. If precipitate still forms the total amount of potassium was not measured. (See Table A.3 and use the next smaller filtrate volume and repeat Par. A.27a through A.27e.)

f. Determine the potassium chloride concentration by comparing the precipitate volume measured with the standard calibration curve as prepared in Par. A.26. Report the potassium concentration as lb/bbl KCl (kg/m³). The potassium concentration may also be reported as mg/L potassium ion. If the filtrate potassium chloride concentration from the standard calibration curve exceeds an 18 lb/bbl reading, accuracy of the results is reduced. For more accurate results, use the next smaller filtrate volume as noted in Table A.3, and repeat A.27a through A.27e.

A.28 Calculation

$$\text{KCl in filtrate, lb/bbl} = \frac{7}{\text{cm}^3 \text{ filtrate used}} \times (\text{value from standard curve}) \quad (\text{a})$$

$$\text{K}^+ \text{ in filtrate, mg/L} = 1500 (\text{KCl in filtrate, lb/bbl}) \quad (\text{b})$$

TABLE A.3
FILTRATE VOLUMES TO BE USED AT
VARIOUS KCl CONCENTRATIONS

Concentration Range		Filtrate Volume to Use
KCl (lb/bbl)	K ⁺ (mg/L)	(cm ³)
3.5-18	5,250- 27,000	7.0
18-35	27,000- 52,500	3.5
35-70	52,500-105,000	2.0
over 70	over 105,000	1.0

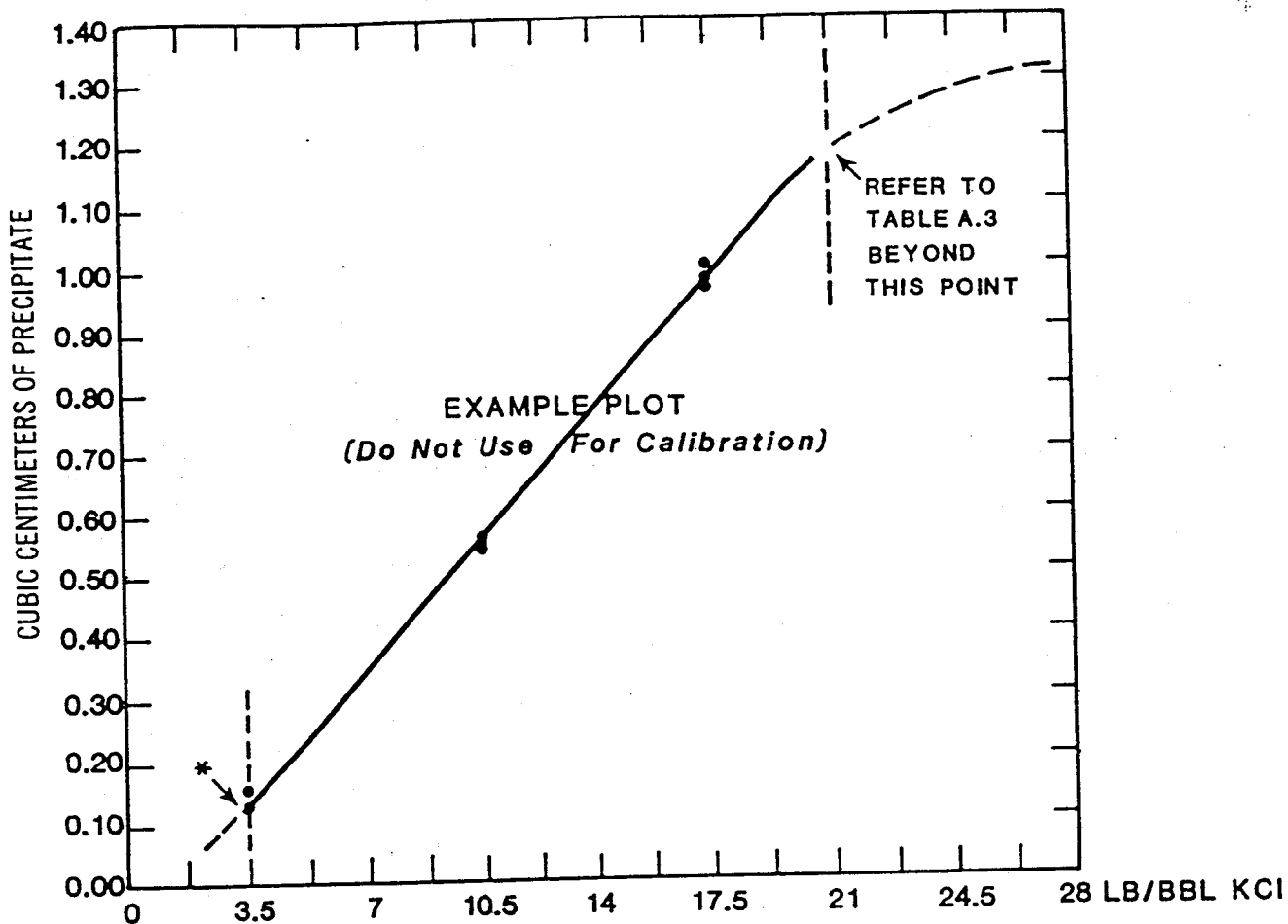


FIG A.6
 EXAMPLE PLOT
 CALIBRATION CURVE FOR POTASSIUM CHLORIDE

*Note: Do not use this procedure for concentrations less than 3.5 lb/bbl of KCl

POTASSIUM BELOW 5000 mg/L (Tentative)

A.29 Description This procedure is used to measure potassium ion content in mud filtrates at levels below 5000 mg/L. Potassium ion is precipitated as the tetraphenylborate salt by adding an excess of standard sodium tetraphenylborate (STPB) solution. The unreacted STPB is then determined by titration with a quaternary ammonium salt (QAS), hexadecyltrimethyl ammonium bromide, using bromophenol blue as an indicator. The endpoint is a purple-blue to light blue color change. The potassium ion concentration in the sample is calculated by subtracting the amount of unreacted STPB from the amount of STPB originally added to the sample.

A.30 Equipment

a. Standard sodium tetraphenylborate solution (STPB): 8.754 g in 800 cm³ deionized water. Add 10-12 g aluminum hydroxide, stir 10 minutes and filter. Add 2 cm³ of 20% NaOH solution to the filtrate and dilute to one liter with deionized water.

b. Quaternary ammonium salt solution (QAS): 1.165 g hexadecyltrimethyl ammonium bromide/500 cm³ deionized water

c. Sodium hydroxide solution: 20 g/80 cm³ deionized water

d. Bromophenol blue indicator: 0.04 g tetrabromophenolsulfonphthalein/3 cm³ 0.1N NaOH. Dilute to 100 cm³ with deionized water.

e. Deionized or distilled water

f. Serological (graduated) pipettes (TD): one 2-cm³ graduated in 0.01 cm³ subdivisions, two 5-cm³, and two 10-cm³

g. Graduated cylinders: two 25-cm³ (TD) and two 100-cm³ (TC)

h. Beakers: two 250-cm³

i. Funnel

j. Filter paper

A.31 Procedure

a. Place the proper amount of filtrate into a 100-cm³ graduated cylinder, using Table A.4 to determine sample size. Be sure to use a pipette to measure the amount of filtrate.

b. Add 4 cm³ of 20% NaOH solution (measured with a 5-cm³ pipette), 25 cm³ of STPB solution (measured with a 25-cm³ graduated cylinder), and enough deionized water to bring the level of the solution to 100 cm³ mark.

TABLE A.4
FILTRATE VOLUMES TO BE USED AT LOW
KCl CONCENTRATIONS

Concentration Range		Filtrate Volume to Use
KCl (lb/bbl)	K ⁺ (mg/L)	(cm ³)
0.2-1.3	250- 2,000	10.0
1.3-2.7	2,000- 4,000	5.0
2.7-6.7	4,000-10,000	2.0

c. Mix and allow to stand ten minutes.

d. Filter into a 100-cm³ graduated cylinder. If the filtrate is cloudy, the solution must be refiltered.

e. Transfer 25 cm³ of the above filtrate (measured with a 25-cm³ graduated cylinder) into a 250-cm³ beaker.

f. Add 10-15 drops of bromophenol blue indicator.

g. Titrate with QAS solution until color changes from purple-blue to light blue.

NOTE: It is important to check the concentration of QAS solution versus the STPB solution at monthly intervals. To determine the equivalent QAS, dilute 2 cm³ of the STPB solution in a titration vessel with 50 cm³ deionized water. Add 1 cm³ of 20% NaOH solution and 10-20 drops of the bromophenol blue indicator. Titrate with the QAS solution until color changes from purple-blue to light blue.

$$\text{Ratio of QAS to STPB} = \frac{\text{QAS, cm}^3}{2} \quad (\text{a})$$

If the ratio is other than 4.0 ± 0.5 , calculate a correction factor to be used in the calculation of mg/L K⁺.

$$\text{Correction factor (CF)} = \frac{8}{\text{QAS, cm}^3} \quad (\text{b})$$

A.32 Calculation

$$\text{K}^+ \text{ in filtrate, } = \frac{1000 (25 - \text{QAS, cm}^3)}{\text{mud filtrate, cm}^3} \quad (\text{a})$$

If correction factor is necessary:

$$\text{K}^+ \text{ in filtrate, } = 1000 \left[\frac{25 - (\text{CF})(\text{QAS, cm}^3)}{\text{mud filtrate, cm}^3} \right] \quad (\text{b})$$

$$\text{KCl in filtrate, } = \frac{(\text{K}^+ \text{ in filtrate, mg/L})}{1500} \quad (\text{c})$$

APPENDIX B SHEAR STRENGTH MEASUREMENT USING SHEAROMETER TUBE

B.1 Description

a. Experience has shown that some drilling muds tend to develop excessive shear strength under static conditions, especially at elevated temperatures. Excessive shear strength results in high pump pressures to "break circulation," and may therefore result in loss of circulation. High shear strength may also cause difficulties in logging, perforating, and other "down hole" operations.

b. The following technique can be used to determine the tendency and estimate the extent to which the mud will develop excessive shear strength. This shear strength measurement is normally made on a static heat-aged mud sample. Aging temperatures are therefore selected to be near the estimated bottom-hole temperature of the well. Aging cells or vessels meeting the pressure and temperature requirements for the test are required.

B.2 Equipment

a. Stainless steel shearometer tube

Length 3.5 in. (89 mm)
 Outside diameter 1.4 in. (36 mm)
 Wall thickness 0.008 in. (0.2 mm)

NOTE: A slight outside taper on the bottom of the shear tube has been found to improve reproducibility of the test results.

- b. Platform for weights
- c. Set of gram weights
- d. Ruler, in.

B.3 Procedure

a. The shear tube and platform are placed and balanced carefully on the surface of the aged sample cooled to room temperature. If may be necessary to shift the weights on the platform to assure the tube's initial penetration into the mud is vertical. If a crust develops on the heat aged sample, this crust should be gently broken before placing the shear tube in place for test.

b. Sufficient gram weights are placed carefully on the platform to start the downward movement of the shear tube. Unless too much weight is added, the tube will stop its downward travel at the point where the shear strength of the aged mud against the surface of the tube is sufficient to support the applied weight (see Fig. B.1). It is desirable to submerge at least one half the length of the tube.

c. Record the total weight in grams which includes the platform and weights. Measure the portion of the tube submerged in the fluid in inches. The length of the tube submerged can be most accurately determined by measuring the length of the nonsubmerged portion while the tube is at its maximum penetration depth. A small ruler held at the mud surface and alongside the tube will facilitate this measurement. The length of the tube minus the exposed length equals the submerged portion.

B.4 Calculation

$$\text{Shear strength (S), lb/100 ft}^2 = \frac{3.61 (Z + W)}{L} - 0.256 A \quad (a)$$

- Where:
- S = shear strength, lb/100 ft²
 - Z = weight of shear tube, g
 - W = total shear weight, g
(sum of platform and weights)
 - L = submerged length of shear tube, in.
 - A = mud weight, lb/gal

Reference: Watkins, T. E. and Nelson, M. D., "High Temperature Gelation of Drilling Fluids," *AIME Transactions* Vol. 193, 1953, pp. 213-218.

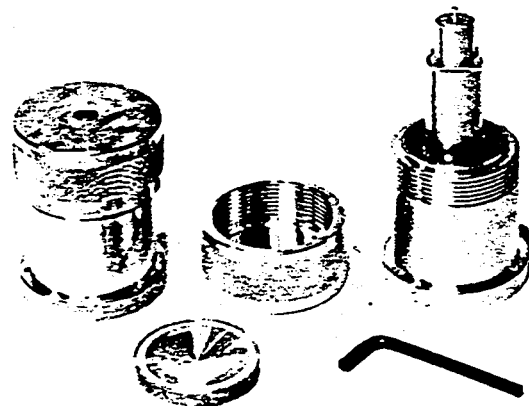


FIG. B.1
SHEAR STRENGTH APPARATUS

APPENDIX C RESISTIVITY

C.1 Description Control of the resistivity of a mud and mud filtrate may be desirable to better evaluate formation characteristics from electric logs.

C.2 Equipment

a. Direct reading resistivity meter or similar resistivity meter. Follow manufacturer's instructions for current source, calibration, measurement and calculations (see Fig. C.1 and C.2).

b. Calibrated resistivity cell.

c. Thermometer: 32-220°F (0-105°C)

C.3 Procedure

a. Fill the clean, dry resistivity cell with freshly stirred mud or mud filtrate.

CAUTION: No air or gas should be entrained in sample.

b. Connect cell to meter.

c. Measure resistance in ohm-meters (direct reading) or ohms (not direct reading). Meter or manufacturer's instructions will indicate type of reading.

d. Measure the temperature of sample to nearest degree F (C).

e. Clean the cell. Scrub with brush and detergent if necessary. Rinse with distilled water and allow to dry.

C.4 Calculation

a. Report the mud resistivity R_m or filtrate resistivity R_{mf} in ohm-meters to nearest 0.01 ohm-meter.

b. Report the sample temperature in degrees F (C). If reading is in ohms, convert to ohm-meters by:

$$\text{Resistance, } = (R, \text{ ohms}) (\text{cell constant, } m^2/m) \quad (a) \\ \text{ohm-meter}$$

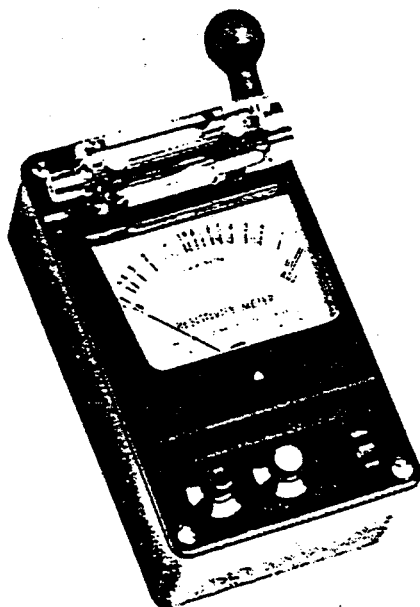


FIG. C.1
DIRECT-READING RESISTIVITY METER

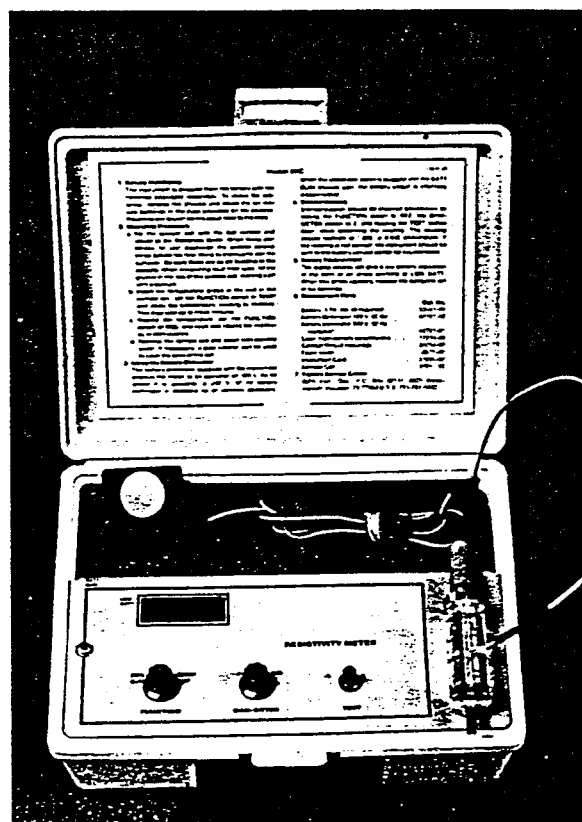


FIG. C.2
DIRECT-READING RESISTIVITY METER

APPENDIX D PROCEDURE FOR REMOVAL OF AIR OR GAS FROM MUD PRIOR TO TESTING

D.1 Description The majority of drilling muds require no special equipment to remove entrained air or gas prior to testing. Usually, gentle agitation together with a few drops of an appropriate defoamer are all that is necessary. Stirring with a spatula or pouring back and forth is sufficient in most cases. When a mud is encountered that retains air or gas after the preceding steps have been taken, the following procedure can be taken to deaerate the mud.

NOTE: *If mud density is the only property desired, the Pressurized Fluid Density Balance described in API Spec 10: API Specification for Materials and Testing for Well Cements may be used. This unit is illustrated in Fig. C.1.*

D.2 Equipment A device is necessary which can be evacuated. The equipment shown in Fig. D.1 is available. Follow the manufacturer's operating instructions.

D.3 Procedure

- a. Fill clean, dry reservoir about one-half full with the air-cut mud.
- b. Add several drops of defoamer to the mud surface.
- c. Insert stirrer and cap; cover with gasketed lid.
- d. Affix vacuum line from the pump to the instrument and hold about 5 inches-of-mercury vacuum.
- e. Increase vacuum to 25-27 inches of mercury and proceed according to the manufacturer's instructions.
- f. When mud has been deaerated, partially relieve vacuum to about 15-10 inches-of-mercury vacuum and observe mud for air bubbles.

g. If deaeration is not sufficient, repeat D.3e and D.3f until air is removed.

h. With cylinder on end, relieve vacuum completely and remove mud sample for testing.

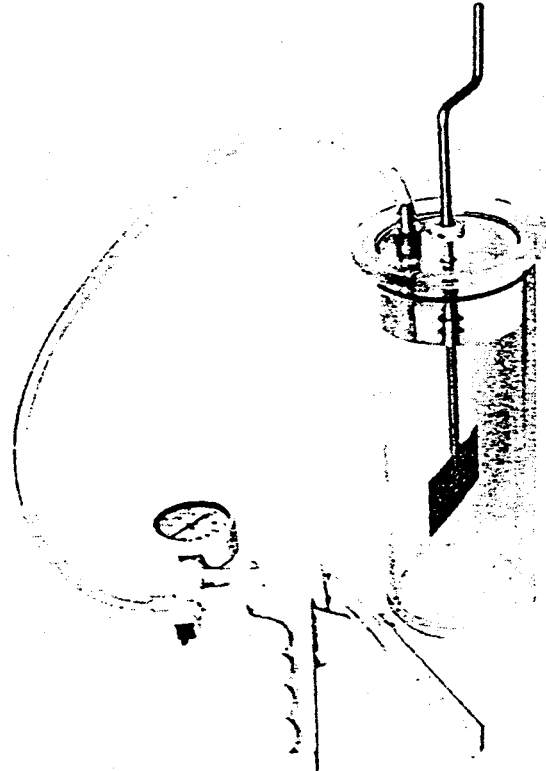


FIG. D.1
INSTRUMENT FOR AIR OR GAS REMOVAL

APPENDIX E DRILL PIPE CORROSION RING COUPON

E.1 Description

a. The placement of corrosion test rings in the drill string is one of the more common techniques used to evaluate the corrosiveness of drilling fluid environments on the drill string and other steel equipment. Removal and examination of these rings after a period of exposure downhole can be highly informative as to the corrosiveness of the drilling fluid as well as to the type of corrosion encountered. An examination of scales and pits on the exposed rings gives clues as to the cause of the corrosion, thus aiding in choosing proper remedial action.

b. The ring technique is specifically designed for detection of the type of corrosion characterized by metal loss whether it be localized pitting or generalized attack. The test ring is not designed to give information relating to hydrogen embrittlement, stress corrosion cracking, or other forms of fracture formation except in the manner in which pitting may relate to these failures.

E.2 Equipment

a. Ring construction: The ring-type drill string corrosion coupon, or corrosion ring, should be machined to fit in the tool box recess, at the end of the pin, and should have a bore the same as that of the tool joint to minimize turbulence.

b. Ring composition: To avoid galvanic corrosion, the ring should be made from steel identical to that of the tool joint in which it is placed. Such a requirement is impractical and use of a steel that is similar in chemical composition, such as AISI 4130, is recommended.

NOTE: *If steel other than AISI 4130 is used, it should be identified on the report form.*

The rings are normally cut from tubes that have not been quenched and tempered. The similarity in composition of the 4130 steel and the tool joint should be adequate to minimize galvanic effects and provide useful data.

c. Ring marking: The rings should be stenciled with a serial number for permanent identification.

d. Ring preparation: The rings should be scrubbed with a stiff fiber bristle brush and detergent solution, rinsed with clean water and with anhydrous acetone or methanol. Allow to dry, weigh to nearest milligram, and record this weight on the report form. Store the ring in a dry container, such as a desiccator, to prevent corrosion. The corrosion rings should be shipped to the field in sealed envelopes or wrappers to minimize atmospheric corrosion.

E.3 Procedure

a. Drill pipe corrosion rings should be kept in the drill string for a minimum of 40 hours. A normal time for exposure is 100 hours. Exposure periods of less than

40 hours should not be used because initial corrosion rates may be unusually high and can give misleading data. The ring is usually placed in the tool joint at the top of the first stand above the drill collars and can be left in the drill string for more than one bit run. An additional ring can be placed in the Kelly saver sub to monitor corrosion at that point. Care should be taken to ensure that the box recess is clean to prevent interference with proper make-up of the joint and to avoid damage to the ring. In some instances specially manufactured subs have been used for the ring placement in the string. During installation the ring should be handled with clean, dry gloves.

b. The drill pipe corrosion coupon form should be filled out completely. Each form should have a space for ring material, drilling fluid properties, type of corrosion, location of ring in the drill string, initial weight, time, depth in, depth out, ring number, color of scale, and any other information of significance in the specific test. The form may be printed on a mailing envelope for the ring or on a separate form to be enclosed with the ring.

c. The drilling fluid residue should be removed from the coupon by wiping with a cloth when the ring is pulled from the drill string. The ring should be examined for severity of corrosion or mechanical damage. If severe corrosion is evident, the cause of the corrosion should be determined promptly so remedial action can be taken. Following visual observation, immediately coat the coupon with oil or other available grease. (Do not use thread compound for this purpose.) Place in a plastic bag and then in the mailing envelope.

d. Before proceeding with a quantitative evaluation of corrosion of the ring, the ring should be rinsed with suitable solvent, such as acetone or petroleum ether, to remove the oil applied to the ring on location. Prior to cleaning for weighing, a spot test should be made for corrosion by-products and mineral scale. For example, the surface can be examined qualitatively for sulfides by the acid arsenite test (see Corrosion of Oil- and Gas-Well Equipment, API, Dallas (1958)). The rings should be cleaned with a detergent solution and a stiff fiber bristle brush. It may be necessary to dip the ring for 5-10 seconds in inhibited 10-15% hydrochloric acid one or more times to remove corrosion products. The ring should be scrubbed with detergent solution after each acid dip. Rinse thoroughly with clean water and then with anhydrous acetone or methanol. Allow to dry prior to weighing. Very abrasive materials or strong, uninhibited acids should not be used. An ultrasonic bath can be useful in cleaning the rings.

e. After the preweighed drill pipe corrosion coupon has been properly cleaned and the corrosion film and type of attack noted, the ring should be reweighed to nearest milligram and the weight loss determined. If

significant loss of metal due to mechanical damage is evident, it should be noted and taken into consideration in evaluation of the ring. The corrosion rate may be reported as kilograms per square meter per year, pounds per square foot per year, or mils per year. Formulas for calculating for corrosion rate are given in Par. E.4.

f. Comments on Visual Examination

a. If visual corrosion is evident, it will normally be detectable as pitting corrosion. Uniform attack or general corrosion can best be determined by a weight loss measurement. Mechanical damage to the ring will most often be evidenced by cuts or dents on the outer surfaces of the ring. In some cases, the ring will exhibit a series of dents and worn spots, indicating considerable movement of the ring in the box recess.

b. In assessing the magnitude of the corrosion rates as calculated from weight loss measurements, it should be remembered that the rate is also influenced by the erosive effects of the drilling fluid. Since the bore of the ring is exposed to the mud pumped down the drill pipe, the loss of metal includes that removed by erosion as well as from corrosion. Loss from erosion can be substantial when the drilling fluid contains a high concentration of sand.

c. Examination of the ring may reveal a few deep pits with a relatively low weight loss. This condition would indicate a rather severe corrosion problem even though the calculated corrosion rate would be considered low.

E.4 Calculation

$$\begin{aligned}
 \text{a. kg/m}^2/\text{yr} &= \left[\frac{\text{wt. loss. mg}}{1,000,000} \right] \left[\frac{10,000}{\text{area. cm}^2} \right] \left[\frac{365}{\text{days exposure}} \right] \quad (\text{a}) \\
 &= \frac{87.60 (\text{wt. loss. mg})}{\text{area. cm}^2 (\text{hours exposed}^{**})} \quad (\text{b})
 \end{aligned}$$

$$= \frac{13.58 (\text{wt. loss. mg})}{\text{area. in.}^2 (\text{hours exposed}^{**})} \quad (\text{c})$$

$$\text{b. lb/ft}^2/\text{yr} = \left[\frac{\text{wt. loss. mg}}{453.600} \right] \left[\frac{144}{\text{area. in.}^2} \right] \left[\frac{365}{\text{days exposure}} \right] \quad (\text{d})$$

$$= \frac{2.781 (\text{wt. loss. mg})}{\text{area. in.}^2 (\text{hours exposed}^{**})} \quad (\text{e})$$

$$\text{c. mils/yr} = \frac{\text{wt. loss. mg}}{\left[\frac{16,387}{\text{cm}^3/\text{in.}^3} \right] \left[\frac{(\text{specific gravity.})}{\text{g/cm}^3} \right] \left[\frac{(\text{area.})}{\text{in.}^2} \right] \left[\frac{(\text{year.})}{\text{days}/365} \right]} \quad (\text{f})$$

For steel coupons with a specific gravity of 7.86, the formula can be reduced to:

$$\text{mils/yr} = \frac{68.33 (\text{wt. loss. mg})}{\text{area. in.}^2 (\text{hours exposed}^{**})} \quad (\text{g})$$

*Total surface area of the ring is used in these calculations.

**Time used based on total time in the drill string.

d. Following are the conversion rates between the various units for steel coupons (specific gravity 7.86):

- mils/yr = 24.62 (lb/ft²/yr) (h)
- mils/yr = 5.03 (kg/m²/yr) (i)
- lb/ft²/yr = 0.04 (mils/yr) (j)
- lb/ft²/yr = 0.20 (kg/m²/yr) (k)
- kg/m²/yr = 0.20 (mils/yr) (l)
- kg/m²/yr = 4.90 (lb/ft²/yr) (m)

e. Corrosion rings available from drilling fluid service companies or corrosion test laboratories are generally supplied with a multiplication factor which includes the metal density and surface area of the ring. Thus, the weight loss divided by the exposure time need only be multiplied by the supplied factor to obtain the corrosion rate.

APPENDIX F
METRIC "SI" UNIT CONVERSION TABLE

Property	Traditional Unit	Recommended SI Unit	Symbol	Conv. Factor	Example
Multiply By:					
Mechanical Properties					
Depth	feet	metre	m	0.3048	10,000 ft = 3048 m
Hole Diameter	inch	millimetre	mm	25.4	12¼ in. = 311 mm
Pipe Diameter	inch	millimetre	mm	25.4	4½ in. = 114 mm
Bit Size	inch	millimetre	mm	25.4	12¼ in. = 311 mm
Weight on Bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary Table Speed	rpm	*revolutions per minute	r/min	1	45 rpm = 45 r/min
Nozzle Size	1/32 inch	millimetre	mm	0.79	10/32 in. = 7.9 mm
Nozzle Velocity	feet/sec	metre/sec	m/s	0.3048	400 ft/sec = 122 m/s
Drilling Rate	feet/hour	*metre/hour	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels	cubic metre	m ³	0.159	3000 bbl = 477 m ³
Liner Size	inch	millimetre	mm	25.4	6½ in. = 165 mm
Rod Diameter	inch	millimetre	mm	25.4	2¼ in. = 57 mm
Stroke length	inch	millimetre	mm	25.4	16 in = 406 mm
Pump Output	barrel/minute	*cubic metre/min	m ³ /min	0.159	8.5 bbl/min = 1.35 m ³ /min
	gallon/minute	*cubic metre/min	m ³ /min	.00378	357 gpm = 1.35 m ³ /min
Pump Pressure	pound/sq inch	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular Velocity	feet/min	*metre/minute	m/min	0.3048	200 ft/min = 61 m/min
Slip Velocity	feet/min	*metre/minute	m/min	0.3048	20 ft/min = 6.1 m/min
				(°F-32) 1.8	80°F = 27°C
Temperature	degree Fahrenheit	degree Celsius	°C	1.8	
	degree centigrade	degree Celsius	°C	1.0	
Funnel Viscosity	seconds/quart	seconds/litre	s/L	1.0	Units cannot normally be converted
	seconds/1000 cm ³	seconds/litre	s/L	1.0	
Mud Weight	pounds/gallon	kilogram/cubic metre	kg/m ³	120	10 lb/gal = 1200 kg/m ³
	pounds/ft ³	kilogram/cubic metre	kg/m ³	16	74.8 lb/ft ³ = 1200 kg/m ³
Pressure Gradient	psi/ft	kilopascal/metre	kPa/m	22.6	0.52 psi/ft = 11.8 kPa/m
Hydrostatic Head	psi	kilopascal	kPa	6.9	4000 psi = 27,600 kPa
Shear Stress	lbf/100 ft ²	pascal	Pa	0.48	20 lb/100 ft ² = 960 Pa
	dynes/cm ²	pascal	Pa	0.100	10 dynes/cm ² = 1.0 Pa
Shear Rate	reciprocal second	reciprocal second	s ⁻¹	1.0	
Apparent, Plastic & Effective Viscosity	centipoise	*centipoise	cP		
Yield Point	lbf/100 ft ²	pascal	Pa	0.48	15 lb/100 ft ² = 7.2 Pa
Gel Strength	lbf/100 ft ²	pascal	Pa	0.48	3 lb/100 ft ² = 1.44 Pa
Dial Reading	lbf/100 ft ²	pascal	Pa	0.51	dial reading - 10 = 5.1 Pa
Standard V-G Meter	1.065				
Power Law Constants					
n	dimensionless				
K	dynes s ⁿ /cm ²	millipascal seconds ⁿ /square centimetre	mPa.s ⁿ /cm ²	100	10 dynes s ⁿ /cm ² = 100 mPa s ⁿ /cm ²
	lbf s ⁿ /100 ft ²	millipascal seconds ⁿ /square centimetre	mPa.s ⁿ /cm ²	479	1.2 lb s ⁿ /100 ft ² = 575 mPa s ⁿ /cm ²
API Filtrate	cubic centimeter/30 min	*cubic centimetre/30 min	cm ³ /30 min		
Filter Cake	1/32 inch	millimetre	mm	0.8	3/32 in. = 2.4 mm
Sand Content)					
Solids Content)	Volume percent	cubic metre/cubic metre	m ³ /m ³	0.01	10% = 0.1 m ³ /m ³
Oil Content)					
Water Content)					
Particle Size	micron	micrometre	um	1.0	
Chemical Properties					
Ionic Concentration	grains/gallon	*milligram/litre	mg/L	17.1	500 grains/gal = 8600 mg/L
	parts/million	*milligram/litre	mg/L	x specific gravity	100,000 ppm of NaCl x 1.0707 = 107,070 mg/L
Alkalinity	cubic centimetre/cubic centimetre	cubic centimetre/cubic centimetre	cm ³ /cm ³		
P_T, M_T, P₁, P₂					
MBT					
Methylene Blue Content	cubic centimetre/cubic centimetre	cubic centimetre/cubic centimetre	cm ³ /cm ³		
Miscellaneous					
Additive Concentration	pound/barrel	kilogram/cubic metre	kg/m ³	2.85	10 lb/bbl = 28.5 kg/m ³
Corrosion Rate	lb/ft ² /yr	kilogram/square metre/year	kg/m ² /yr	4.9	0.9 lb/ft ² /year = 4.3 kg/m ² /year
	mils/year	millimetre/year	mm/a	.0254	200 mils/year = 5.08 mm/a
Bentonite Yield	barrels/US ton	*cubic metre/metric tonne	m ³ /t	0.175	100 bbl/ton = 17.5 m ³ /t
Hydraulic HP	horsepower	kilowatt	kw	0.746	600 hhp = 450 kw
Screen Size					
Mesh	openings/inch	openings/centimetre	openings/cm	0.254	100 mesh = 25.4 openings/cm
Aperture	micron	micrometre	um	1	
Open Area	area percent	square metre/square metre	m ² /m ²	0.01	30% = 0.3 m ² /m ²
Weight of Drill Pipe	pound/foot	kilogram/metre	kg/m	1.49	19.5 lb/ft = 29.1 kg/m
Oil Muds					
Oil Content)	volume percent	cubic metre/cubic metre	m ³ /m ³	0.01	10% = 0.1 m ³ /m ³
Water Content)					
Emulsion Stability	volts	volts	V		
Activity	Dimensionless				
Salinity	ppm	*milligrams/litre	mg/L	specific gravity	250,000 ppm of CaCl ₂ x 1.24 = 310,000 mg/L
				(°F-32) 1.8	150°F = 66°C
Aniline Point	degree Fahrenheit	degree Celsius	°C	1.8	
					*Denotes acceptable deviation from API 2564 preferred unit

Appendix F

Bentonite Product Data

FEDERAL JEL "90"

description

FEDERAL JEL sodium bentonite is made from the finest blends of western montmorillonite clays. It is designed to meet exacting customer specifications. FEDERAL JEL "90" sodium bentonite is a consistent, high quality material which has proven successful in a variety of civil engineering applications.

applications

*SLURRY TRENCH CUTOFF WALL
 CEMENT/BENTONITE CUTOFF WALL
 SLURRY TRENCH BACKFILL ADDITIVE*

advantages

... Meets or Exceeds A.P.I. 13A Specification

**typical
 chemical
 analysis**

SiO ₂	61.3% to 64.0%	K ₂ O	0.4%
Al ₂ O ₃	19.8%	Na ₂ O	2.2%
Fe ₂ O ₃	3.9%	TiO ₂	0.1%
MgO	1.3%	Trace Elements	3.2%
CaO	0.6%	H ₂ O (Crystal)	7.2%

**physical
 properties**

	<u>Typical Analysis</u>	<u>A.P.I. 13A Specification</u>
Fann 600 Reading	35.0 cps	30.0 cps min
Fann 300 Reading	25.0 cps	-
Plastic Viscosity	10.0 cps	-
Yield Point	3 x plastic vis max	3 x plastic vis max
Apparent Viscosity	17.5 cps	-
Filtrate	13.5 ml	15.0 ml max
Dry Screen Analysis (% minus U.S. 200 Mesh)	80.0%	-
Wet Screen Analysis (% Retained on U.S. 200 Mesh)	2.5-3.0%	4.0% Max
Moisture at time of shipment	7.0-9.0%	10.0% max
pH	9.0-10.0	

packaging

Bulk Carload/Truckload Quantities
 100- & 50-lb Multiwall Kraft Bags
 4,000-lb Bulk Bags

HYDROGEL

TRADE NAME HYDROGEL®

CHEMICAL NAME Sodium montmorillonite

DESCRIPTION AND USE A premium grade, high quality sodium bentonite for use in all types of slurry trenching.

CHEMICAL ANALYSIS				
	Silica (SiO ₂)	60.34%	Soda (Na ₂ O)	2.34%
Alumina (Al ₂ O ₃)	19.28	Magnesia (MgO)	1.67	
Ferric Oxide (Fe ₂ O ₃)	3.48	Potash (K ₂ O)	.10	
Titanium Dioxide (TiO ₂)	.22	Other	.07	
Lime (CaO)	.38	Water (H ₂ O)	7.75	
L.O.I.	4.37			

BARREL YIELD One ton of HYDROGEL will yield a minimum of 92 bbls of 15cps. mud when mixed with potable water.

PRODUCT SPECIFICATIONS	A.P.I.	TYPICAL
	SPECIFICATION 13-A, Sec. 3-1980	HYDROGEL
Viscometer Reading at 600 R.P.M.	30 Min.	39 ± 5
Water Loss	15.0 cc Max.	13.5 ± 1
% Thru 200 Mesh Screen	—	80 ± 2
Wet Screen Analysis		
Residue on U.S. Sieve No. 200	4.0% Max.	3.0 ± .5
% Moisture	10.0% Max.	7 ± 1
pH	—	9.1 ± .4
Gel Strength - 10 Sec.	—	7 ± 1
Gel Strength - 10 Min.	—	18 ± 2
Plastic Viscosity	—	14 ± 2
Yield Point, 1b/100ft. ²	3 x P.V. Max.	16 ± 4

Viscosity, yield point, water loss and pH are determined on the basis of suspension of 22.5 grams of bentonite in 350 ml of distilled water.

PACKAGING 100# multi-wall paper bags or bulk

SHIPPING Truck, rail (Export information available on request)

Why Choose HYDROGEL®?

HYDROGEL — a high swelling Wyoming bentonite is ideally suited for all types of slurry trenching. Ordinary clays swell one to two times their dry volume upon hydration. HYDROGEL swells twelve to sixteen times its dry volume.

When HYDROGEL and the soil backfill are mixed and returned to the trench, this unique swelling action literally "plugs up" the voids between the soil particles to effectively stop fluid seepage.

DAMS

Slurry trenches are used extensively to stop seepage through or beneath earthen dams, coffer dams, and levees.

DEWATERING

A slurry trench is an excellent method to process slurry. Since sheet piling is eliminated, a significant cost saving is realized.

Appendix G

Slurry Soil-Bentonite Wall Construction Specification

SPECIFICATION
FOR
SLURRY CUTOFF WALL (TRENCH)

1.0 GENERAL

1.1 WORK INCLUDED

The work covered by this section of the specifications consists of furnishing all plant, labor, equipment, and materials and performing all operations in connection with constructing an impermeable slurry cutoff wall in accordance with the drawings and these specifications. The slurry cutoff wall shall basically consist of a soil/bentonite slurry cutoff trench excavated through the various subsurface strata to the approximate depths shown on the drawings or as designated by the Engineer.

1.2 QUALIFICATIONS FOR SLURRY CUTOFF TRENCH CONSTRUCTION

The Contractor shall submit evidence that he or his subcontractor is competent in slurry trench construction. The Contractor shall ensure that he or his subcontractor has sufficient competent personnel to carry out the operations specified and such personnel shall have experience in this type of construction. In particular, a slurry trench specialist employed by the Contractor shall be used to control the composition, mixing, placing, cleaning, and maintaining of the slurry. Credentials of the slurry trench Contractor and slurry trench specialist shall be submitted to the Engineer prior to the start of work. If the slurry trench Contractor or slurry trench specialist is not considered properly qualified, he will be rejected, and if the slurry trench specialist is rejected, the Contractor shall provide other properly qualified personnel at the same bid price. Evidence of competency shall be a minimum of five years experience in slurry trench construction and quality control and at least one successfully completed slurry trench project of a similar size and complexity as this project.

The above information, along with appropriate reference shall be furnished to the Engineer on proposed key personnel prior to mobilization for the slurry cutoff trench construction.

2.0 PRODUCTS

2.1 BENTONITE

Premium grade of sodium bentonite complying with the requirements of API Specification 13A. The bentonite shall not contain any additives used in oil well drilling.

2.2 BENTONITE SLURRY

Slurry mixture containing a minimum of 6 percent bentonite by weight in clean water. The viscosity of the bentonite slurry shall have a minimum Marsh Funnel Value of 40, after hydration for a minimum of eight (8) hours, when tested in accordance with Marsh Funnel Procedure 2.3 in API Specification RP-13B.

2.3 WATER

Water Quality. The water shall be free from oil, acid, alkali, organic matter, or other deleterious substances that prevent the hydration of bentonite, complying with the requirements listed in Quality Control Standards. It is the responsibility of the Contractor that the resulting slurry from the water used shall always meet the specified permeability standards in the specification.

2.4 ADDITIVES

Additives shall not be used without the prior written approval of the Engineer.

2.5 BACKFILL

Materials for backfilling the slurry cutoff wall may be obtained from excavation for the slurry cutoff trench, on-site borrow areas, and/or from off-site materials provided by the Owner. The materials shall be thoroughly blended with a minimum of 2% bentonite (by dry weight).

At the time of placement, the backfill materials shall have a slump of 4 to 6 inches.

3.0 EXECUTION

3.1 GENERAL

Excavation shall be carried to full depth at the point where excavation is started and then the full depth of cut shall be carried along the line of the trench. Excavation shall proceed in a continuous fashion from the starting point to the finishing point. Slurry shall be introduced into the trench at the time trenching is begun and shall be continuously maintained in the trench during excavation and until the backfilling is completed. The Contractor shall maintain the stability of the excavated trench at all times for its full depth.

Upon completion of any segment of trench, care shall be exercised to ensure that the excavation face shall have a uniform slope no steeper than 1:1. The slope shall be free from undercutting or hollows capable of trapping slurry or inhibiting slurry displacement by the backfill. When keying one segment of trench into an already completed segment, a minimum overlap of 20 feet shall be provided at the bottom of the trench.

3.2 EQUIPMENT

- a. Trench Excavation. Excavation of the slurry cutoff trench shall be accomplished by use of any suitable earth-moving equipment or combination thereof such as a backhoe and/or clamshell so that the required width trench, as shown on the drawings, can be carried to its final depth of cut continuously along the trench line.
- b. Slurry Batching Plant. The slurry batching plant shall include the necessary equipment including a mixer capable of producing a colloidal suspension of bentonite in water, pumps, valves, hoses, supply lines, and all other equipment as required to adequately supply slurry to the trench. Storage ponds shall be provided to store initially mixed slurry to allow hydration and to serve as reserve in cases where substantial slurry loss from the trench may occur. The slurry shall be agitated or recirculated in the storage ponds as required to maintain a homogeneous mix. All slurry for use in the trench shall be prepared using a suitable mixer. No slurry is to be made in the trench. Mixing of water and bentonite shall continue until bentonite particles are hydrated and the resulting slurry is homogeneous.
- c. Backfilling, Mixing and Placing. Equipment for mixing and placing backfill shall consist of suitable type of earth moving, blending, or grading equipment, such as bulldozers, disc harrows or blade graders that are capable of thoroughly mixing the backfill materials into a homogeneous paste having the required properties and placing the material in the trench as hereinafter specified.

3.3 EXCAVATION PROCEDURES

- a. General. The slurry cutoff trench shall be excavated by the slurry method of excavation. Excavation shall be carried to the full depth shown on the drawings or as modified by the Engineer immediately at the point where excavation is started. The entire depth of excavation shall then be carried along the trench line. The face of the trench excavation shall not precede the toe of the backfill slopes by more than 300 feet or less than 100 feet. The slurry cutoff trench shall be constructed without undue interruption until it is complete.

A minimum overlap length of five feet shall be made at any corner to obtain the continuous full depth through the entire length of each side of the trench, unless the equipment used produces vertical end walls.

- b. Slurry. Slurry shall be introduced into the trench at the time excavation begins. The level of the slurry in the open trench shall be maintained a minimum of 5 feet above the static water level as well as being maintained within 3 feet of the top of the working surface at all times. Dilution of slurry by surface waters shall be prevented. The slurry shall be maintained at all times in a condition which meets the requirements set forth in paragraph 4.0, Quality Control and subparagraphs thereof.

c. Stockpiling. Material excavated from the slurry trenches that are suitable for the backfill mixture may be stockpiled for subsequent processing. All such stockpiles shall be located so as not to damage the trench wall.

d. Spoil. Material excavated from the slurry trench that is not suitable for the backfill mixture or is in excess to the Contractor's needs shall be placed in a designated area.

3.4 BACKFILLING OF SLURRY CUTOFF TRENCH

a. Backfill Blending. Blending shall be performed in such a manner as to produce the required gradation and shall consist only of suitable material as specified under paragraph 2.5 BACKFILL. This material, when mixed with the slurry to attain the specified slump and gradation, shall constitute the backfill used to fill the slurry trench. Excess slurry which may drain away from the blending operations shall be allowed to drain back into the trench. The backfill material shall be thoroughly blended into a homogeneous mass, free from large lumps or pockets of fines, sand or gravel. Occasional lumps of up to six inches in their largest dimensions will be permitted. The backfill material, just prior to placing, shall have a slump of four to six inches. If two or more materials are to be blended to obtain the required backfill gradation, the coarser grained material shall be placed first, then the finer grained material mixed into it.

b. Backfill Placement. The backfill shall be placed continuously from the beginning of the trench, in the direction of the excavation, to the end of the trench. Placing operations shall proceed in such fashion that the surface of the backfill below the slurry shall follow a reasonably smooth grade and shall not have hollows which may trap pockets of slurry during subsequent backfilling. Free dropping of backfill material through the slurry will not be permitted.

Initial backfill shall be placed by lowering it to the bottom of the trench with the equipment bucket until the surface of the backfill area rises to the level of the working platform at the end of the trench. Additional backfill may then be placed in such manner that the backfill enters the trench by sliding down the forward face of the previously placed backfill. To accomplish this, sufficient backfill shall be piled on the edge of the existing to cause a slump and sliding action on the face of the in-place backfill. The backfill shall not be dropped or deposited in any manner that will cause segregation.

An acceptable substitute for the initial placing of backfill by clamshell bucket shall be to commence excavation with a lead-in trench. The lead-in trench shall have a minimum slope of 2:1 (horizontal to vertical) to the initial point of the design trench bottom. Backfill placement shall commence at the point where the lead-in trench intersects the ground surface following the provision of this specification.

c. Backfilling, Blending and Placement During Cold Weather. No blending or placing of backfill shall be performed when the air temperature is below 20 degrees Fahrenheit or when the backfill material is freezing.

d. Cleanup. After completion of backfilling operations, all remaining excavated material and slurry shall be removed from the working surfaces. All materials containing the bentonite slurry shall be removed and disposed of within designated areas.

4.0 QUALITY CONTROL

4.1 GENERAL

The Contractor shall be responsible for quality control testing and reporting for the slurry and slurry trench backfill and shall obtain samples for testing. The following table summarizes the required Quality Control standards applicable to this project:

QUALITY CONTROL STANDARDS

<u>Item</u>	<u>Feature Examined</u>	<u>Standard</u>	<u>Frequency of Test</u>	<u>Specification</u>
A. MATERIALS: Bentonite	Grade of Bentonite	API spec.13A Section 4,7, 8,9	1 per lot (or shipment)	Manufacturer's certificate of compliance and supporting test data
Water	pH	API RP 13B, Section 7	1 per water source, and per 100,000 gallons used, and at least monthly	7 ± 1
	Total Hardness	API RP 13B, Section 8		≤ 50 ppm
	Total Dissolved Solids	API RP 13B, Section 4		≤ 500 ppm

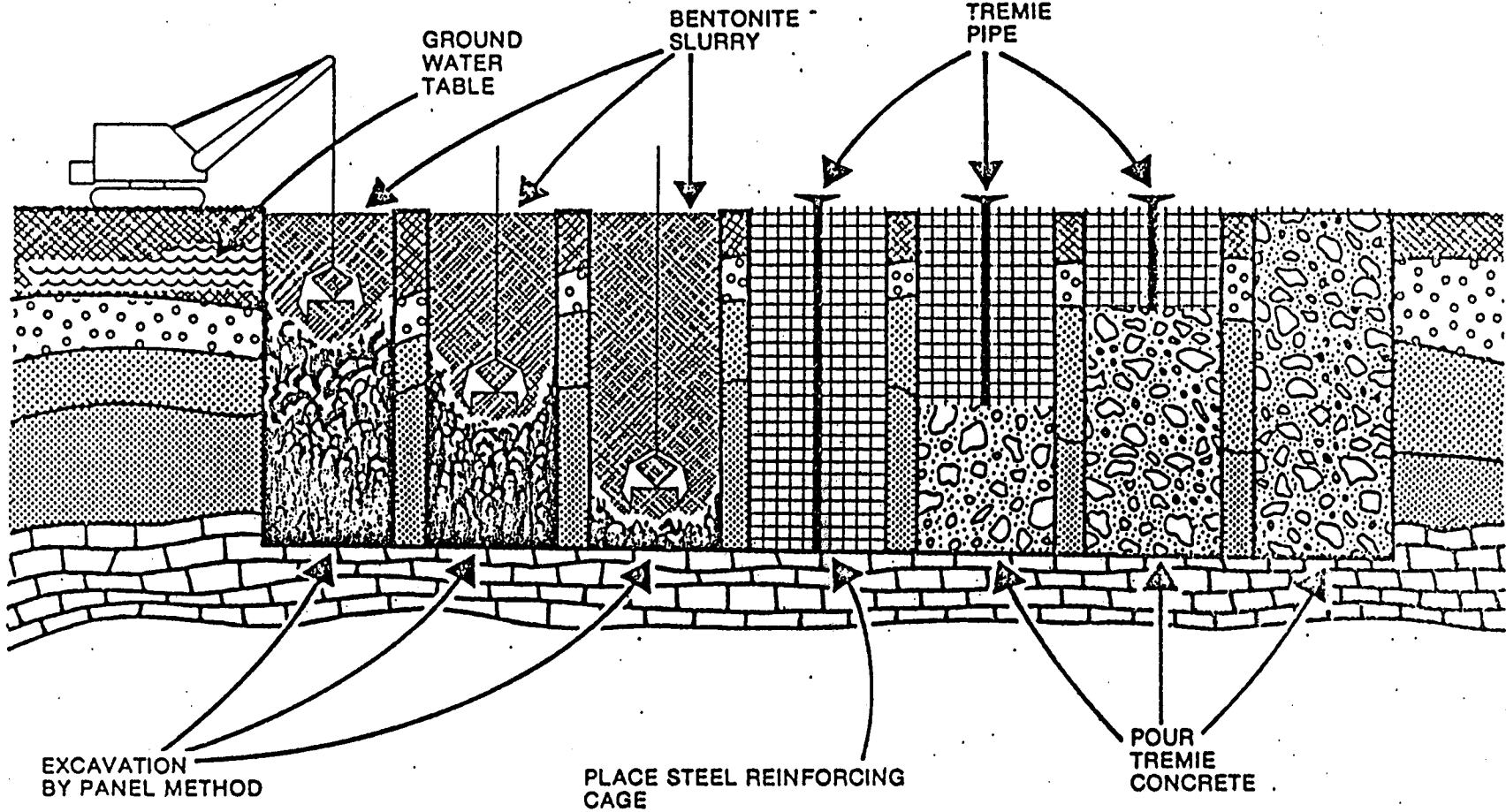
<u>Item</u>	<u>Feature Examined</u>	<u>Standard</u>	<u>Frequency of Test</u>	<u>Specification</u>	
B. TRENCH EXCAVATION	Continuity of width & alignment	————	1 horizontal taped measurement per 50 linear feet	Within 6" of alignment and width, as shown on the plans	
	Continuity of depth	————	1 vertical taped measurement per 50 linear feet	Within 6" of depth, as shown on the plans	
	Keying into foundation	————	Continued visual monitoring of cuttings, verified by vertical depth sounding, as above		
C. SLURRY Hydration Pond	Viscosity	API RP 13B, Section 4	2 per 8-hour working shift	40-50 seconds by Marsh Funnel	
	Filtrate Loss	API RP 13B, Section 3	1 per 3 consecutive working days or fraction thereof	≤ 30cc in 30 minutes at 100 psi	
	Trench	Unit Weight	API RP 13B, Section 1	2 per 200 linear feet of trench	64.5 - 85.0 pcf
		Filtrate Loss	API RP 13B, Section 3	1 per 200 linear feet of trench	≤ 25cc in 30 minutes at 100 psi
D. BACKFILL MIX	Permeability	Corp of Engineers EM1110-2-1906 Appendix VII	1 per 1000 c.y.	Coefficient k of $\leq 1 \times 10^{-7}$ cm/s	

<u>Item</u>	<u>Feature Examined</u>	<u>Standard</u>	<u>Frequency of Test</u>	<u>Specification</u>
	Slump	ASTM C143	1 per 100 l.f. and at least one daily	4 - 6 inches
	Unit Weight	Corp of Engineers EM1110-2-1906 Appendix II	1 per 1000 c.y.	≥ 80 pcf, and at least 15 pcf greater than slurry

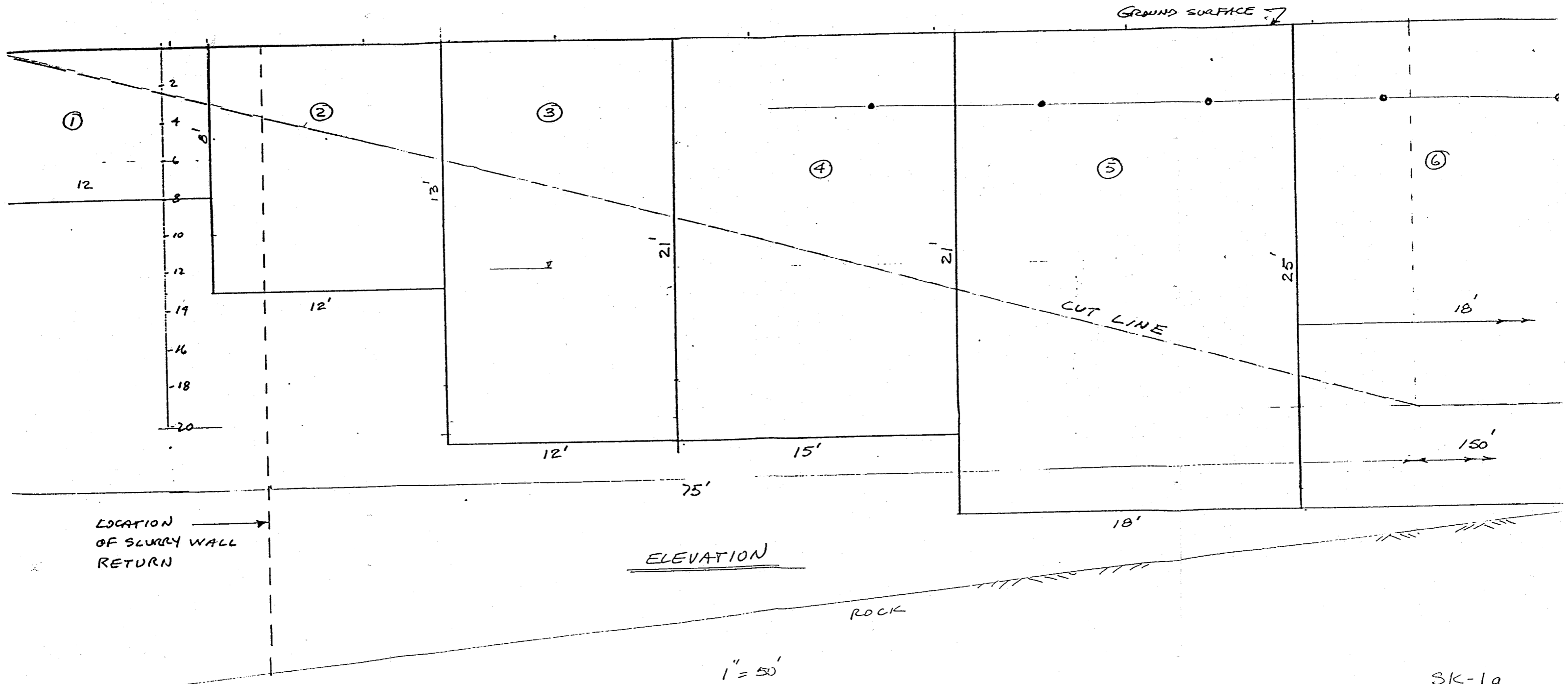
Appendix H

Slurry Concrete Diaphragm Wall Design Information

DIFFERENT PHASES OF CONSTRUCTION OF A SLURRY DIAPHRAGM WALL



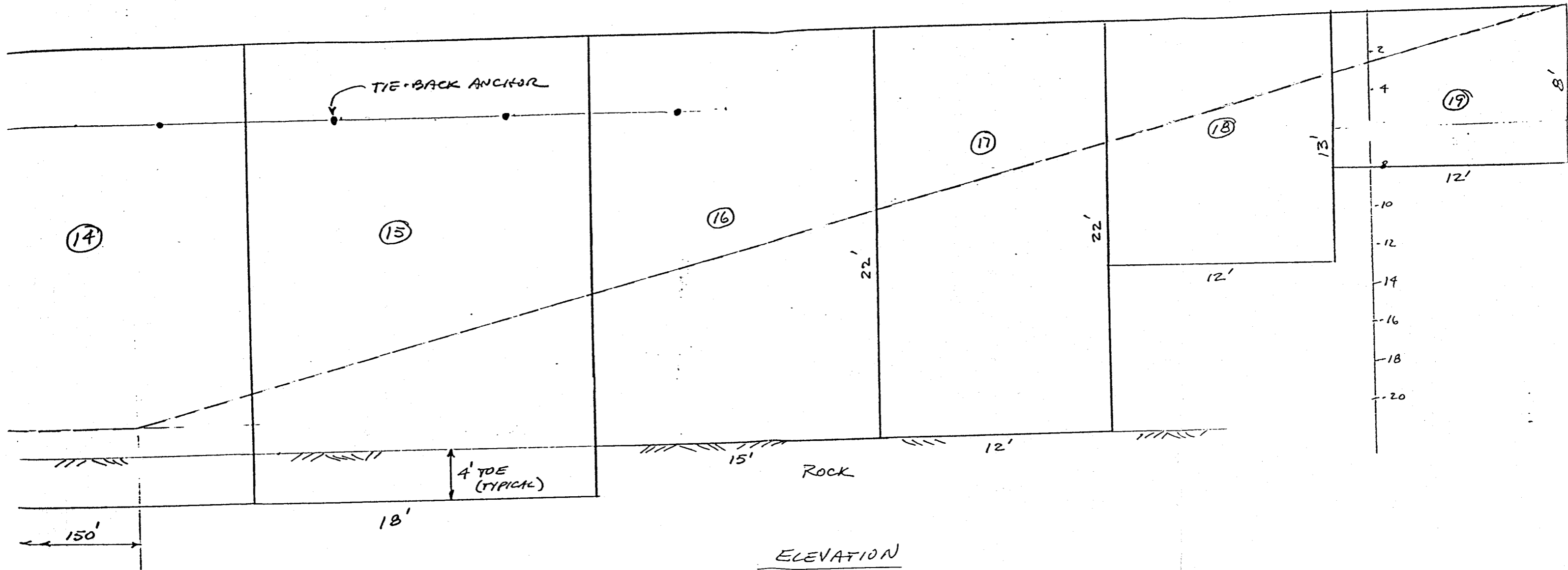
KENNECOTT FLAMBEAU PROJECT
 CONCRETE DIAPHRAGM WALL

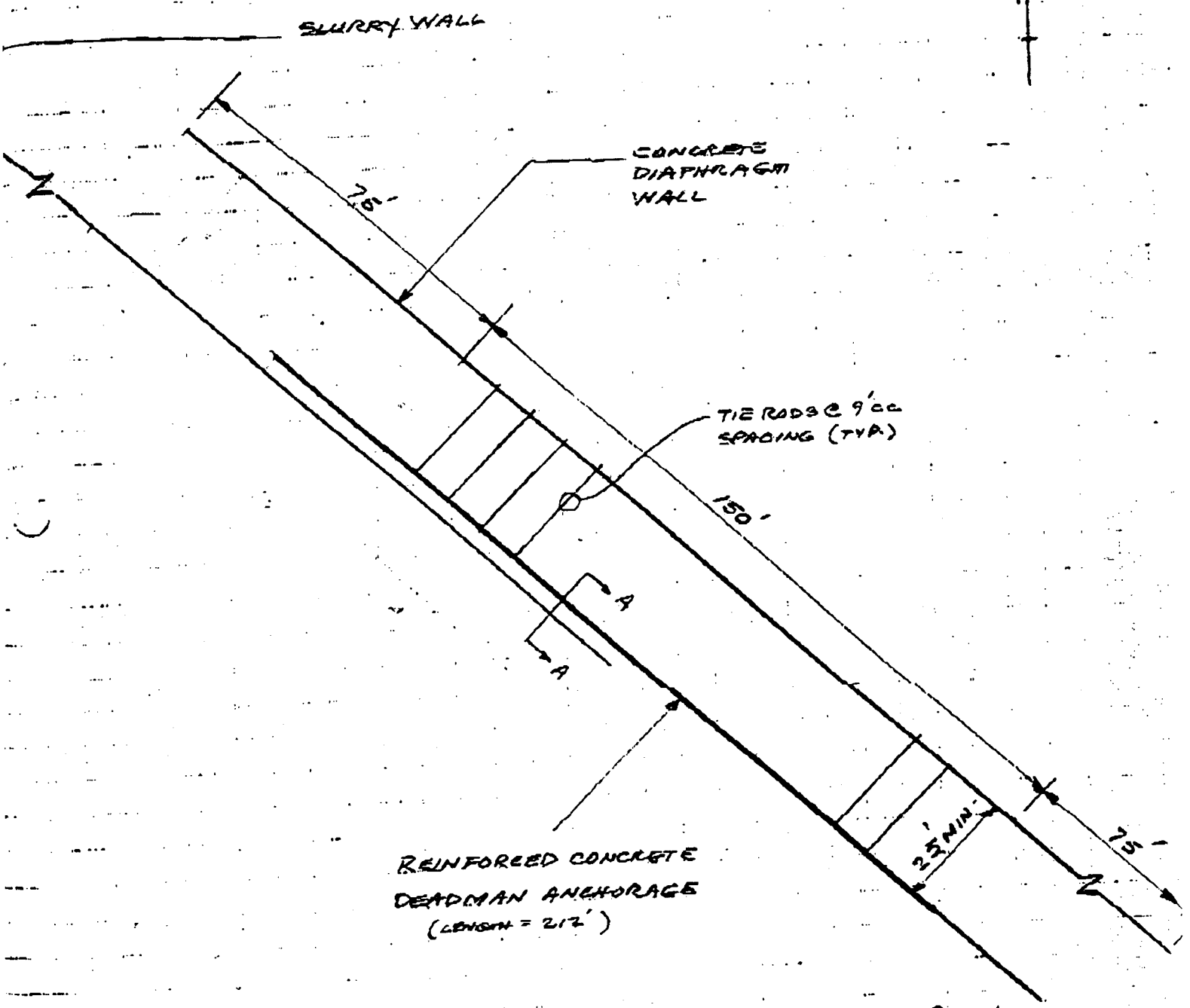
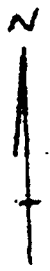


LOCATION OF SLURRY WALL RETURN

ELEVATION

1" = 50'





SLURRY WALL / CONCRETE DIAPHRAGM WALL PLAN
[1" = 30']

SK-2

REV. 6-19-92

FLAMBEAU PROJECT
LAD./SMITH, KLS.

CASE INTERNATIONAL CO.

SK-3
6-18-92

[1" = 2']

SECTION A-A



Flambeau Mine - Diaphragm Wall Design

①

Excavation Depth

= 20'

Soil Conditions

0 - 20' silty sand med. to dense: ± sand & gravel lenses

22' - 35' copper sulfide ore rock fractured

RQD = 2-4% Recovery 90-95%

Estimated Soil Parameters

$\gamma = 125 \text{ pcf}$

$\phi = 30^\circ$

$k_a = 1/3$

water table @ 12'

Lateral Pressure

Average Surface Surcharge = 300 pcf

$P_{at 0'} = 1/3 \times 300 = 100 \text{ pcf}$

$P_{at 12'} = 1/3 \times (300 + 125 \times 12) = 1800/3 = 600 \text{ pcf}$

$P_{at 22'} = 1/3 \times (300 + 125 \times 12 + (3 \times 10)) = 2430/3 = 810 \text{ pcf} \div 62.4 \times 10 = 524 \text{ pcf}$

Reaction at Tie Level

$$P \approx \frac{L}{18} \times \left\{ 100 \times \frac{20^2}{2} + 500 \times \frac{12^2}{2} + \left(\frac{12}{3} + 10 \right) \times 500 \times \frac{10^2}{2} + 210 \times \frac{10^2}{2} \right\}$$

$$= \frac{1}{18} \times \{ 20000 + 42000 + 35000 + 3500 + 10500 \}$$

$$= \frac{105200}{18}$$

$$= 5844 \text{ lb/ft}$$

$$T_{LR} = \left(\frac{100 \times 20}{2} + \frac{600 \times 12}{2} + \frac{600 \times 10}{2} \right) \times 10$$

$$+ 624 \times \frac{10}{2} - 5844$$

$$= 4200 + 7050 + 3120 - 5844$$

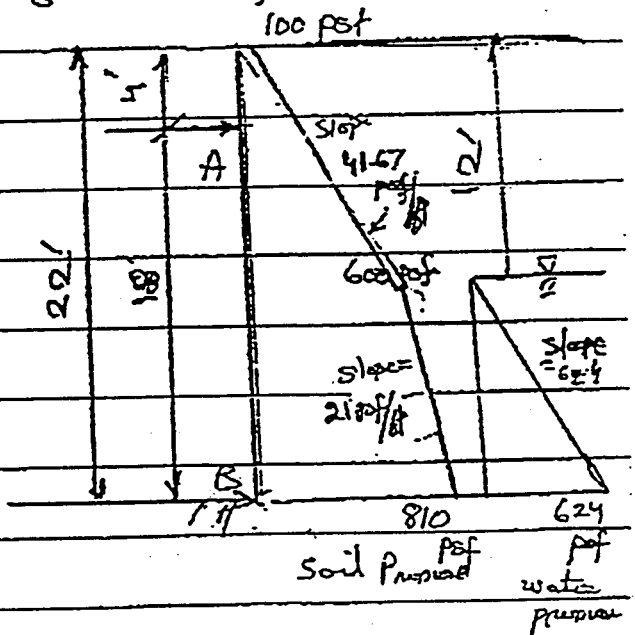
$$= 8526 \text{ lb/ft}$$

B.M. in the wall

Zero Shear is at 14.3 ft from top (by trial)

$$V = 5844 - 100 \times 12 = 4644$$

$$= 11.67 \times 12 \times \frac{12}{2} - 600 \times 2.3 = (21 + 62.4) \times 2.3 \times \frac{2.3}{2}$$



Max B.M.

$$= 5844 \times (14.3 - 4) - 100 \times \frac{14.3^2}{2} - 500 \times \frac{12}{2} \times \left(\frac{14.3}{3} + 2.3\right) - 600 \times \frac{2.3^2}{2}$$

$$- (62.4 + 21) \times \frac{2.3^2}{6}$$

$$= 60193 - 10224 - 18900 - 1587 - 73 = 29408 \text{ lbm-ft}$$

$$= 29.4 \text{ k-ft}$$

Reinf. Design

24" wall

d = 24 - 3 - 1/2 = 20.5"

A_s = (29.4 * 12) / (24 * 0.87 * 20.5) = 0.82 D"

Major use # 8 @ 10" O.F.

A_s = 0.95 D" I.F.

6 @ 10" O.F.

A_s = 0.53 D" O.F.

Horiz # 5 @ 12"

Anchorage

Tie back load = 5844 lb/ft

Spy 6.0 k/ft

Using Concrete deadman:

phi = 30, delta = 10, k_p = 4.1

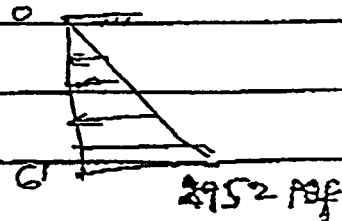
P_p = 120 * 4.1 = 492 psf/ft

Try 6' deep deadman wall

k * PH = 4.1 * 120 * 6 = 2952

Total = 2952 * 6 = 8856 lb/ft

FS = 8856 / 5844 = 1.51 O.K.



Tie rods 1 7/8" dia upset to 2 1/2" rods or 2" dia

Non Threaded area 2.50"

Allowable load = 2.5 * 24 = 60k

Use Tie back spacing = 9' 9 * 6 = 54k

Bit in deadman = 6 * 9^2 / 10 = 48.6 k-ft

H6 d = 24" - 3 - 1/2 = 20.5"

A_s = 1.86 D" use 6-# 5 @ 12" = 1.86 D" O.F.

Conditions of excavation depth of 12

Active Pressure $P_a = k_a (\gamma H + \text{Surcharge})$

$P_a @ 0' = \frac{1}{3} \times (0 + 300) = 100 \text{ psf}$

$P_a @ 12' = \frac{1}{3} \times (125 \times 12 + 300) = \frac{1800}{3} = 600 \text{ psf}$

$P_a @ 15' = \frac{1}{3} \times (125 \times 12 + 63 \times 3 + 300) = \frac{1}{3} \times 1959 = 653 \text{ psf}$

$P_a @ 22' = \frac{1}{3} \times (125 \times 12 + 63 \times 10 + 300) = \frac{1}{3} \times 2430 = 810 \text{ psf}$

Water Pressure

$P_w @ 12' = 0$

$P_w @ 15' = 62.4 \times 3 = 187 \text{ psf}$

$P_w @ 22' = 62.4 \times 10 = 624 \text{ psf}$

Passive Pressure: $P_p = k_p \gamma H$

Soil inside the cut will be drained

Using $\phi = 32^\circ$: $\delta = 15^\circ$: $k_p = 5.5$

$P_p @ 15' = 0$

$P_p @ 22' = 5.5 \times 125 \times 7 = 4812 \text{ psf}$

Reaction at Tie level:

let x be the depth below

cut level for equilibrium

$\sum \tau = 0$ Moment about T = 0

$100 \times 12 \times \left(\frac{12}{2} - 4\right) + 500 \times \frac{12}{2} \times \left(\frac{24}{3} - 4\right)$

$+ 600 \times 3 \times \left(\frac{3}{2} + 8\right)$

$+ (63 + 187) \times \frac{3}{2} \times \left(\frac{3}{2} + 8\right)$

$+ (63 + 187) \times x \times \left(\frac{x}{2} + 11\right)$

$- (657.5 - 21 - 624) \times x \times \frac{x}{2} \times \left(\frac{x}{2} + 11\right) = 0$

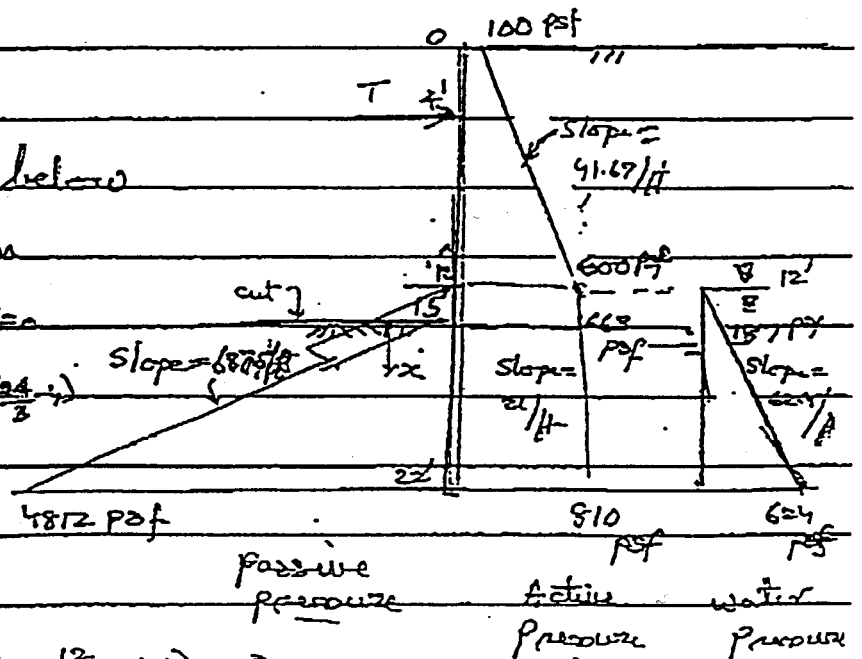
$2400 + 18000 + 3750 + 17100 + 4.25x^2 + 9350x - 2067x^2 - 3322.6x = 0$

or $201.37x^3 + 2897.6x^2 - 9350x - 35250 = 0$

By trial $x = 4.5$

H7

$18250 + 58676 - 42075 - 20050 = -299 \approx 0$



(4)

$$\sum F_x = 0$$

$$\begin{aligned} & \left(\frac{100 + 600}{2} \right) \times 12 + \left(\frac{600 - 662}{2} \right) \times 2 + 187 \times \frac{3}{2} = (697.5 - 21 - 62.4) \times 4.5 \times \frac{5}{2} \\ & = 662 \times 4.5 = 4200 + 1894.5 = 6116.5 + 2983.5 \\ & = 2962 \text{ lb/ft} \end{aligned}$$

$$\text{Design reaction} = 2962 \times 1.25 = 3702 \text{ lb/ft} \quad \text{say } 3.7 \text{ k/ft}$$

B.M. in the Wall:

Zero shear is at 9.7 ft from top (by trial)

$$\begin{aligned} V &= 2962 - 100 \times 9.7 = 41.67 \times 9.7 \times \frac{9.7}{2} = 2962 - 970 = 1960 \\ &= 32 \text{ lb} \end{aligned}$$

$$\begin{aligned} \text{Max BM} &= 2962 \times (9.7 - 4) - 100 \times \frac{9.7^2}{2} = \frac{9.7^3}{6} \times 41.67 \\ &= 16883 - 4704 = 6359 \\ &= 5840 \text{ lb-ft} \end{aligned}$$

Reinf. Required

$$A_s = \frac{M}{f_s j d} = \frac{5840 \times 12}{24000 \times 0.87 \times 20.5} = 0.170''$$

$$\text{Min } A_s = 0.0033 \times 24 \times 12 = 0.950''$$

$$\text{Use } \# 8 @ 10'' \text{ T.E. } A_s = 0.950'' \quad \text{O.K.}$$

$$\# 6 @ 10'' \text{ O.F. } A_s = 0.530''$$

$$\text{HORIZ: steel } \# 5 @ 12'' \text{ S.F. } A_s = 0.620'' \quad \text{O.K.}$$

$$\text{Required temperature steel} = 0.002 \times 24 \times 12 = 0.576''$$

Tie Embedment:

$$\text{Minimum required for equilibrium} = 4.5'$$

$$\text{Provided tie to rock level} = 7' \quad \text{O.K.}$$

Tie back: Design reaction = 3.7 k/ft

$$\begin{aligned} \text{Capacity of 6' deep deadman with F.S. } \frac{1.5}{1.5} &= \frac{5865}{1.5} = 5910 \text{ k/ft} \\ \text{if use 5' deadman available } f_p &= \frac{1}{2} \times 4.1 \times 120 \times 5 = 6150 \text{ lb/ft} \end{aligned}$$

$$\text{F.S.} = \frac{6.15}{3.7} = 1.66 \quad \text{O.K.} \quad \text{H8} \quad \text{May use 5' deadman depth.}$$

Cantilever Wall Section:

Try cantilever height of 10'

Active Pressures:

Active pressures are same as for the tied back wall.

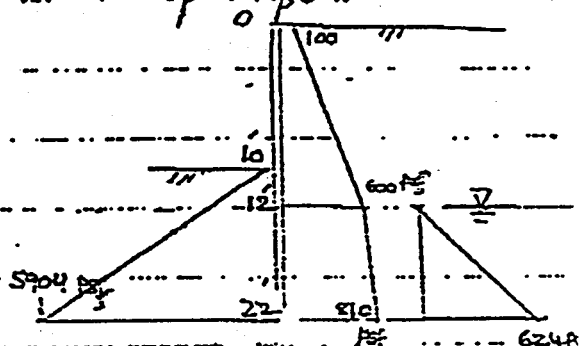
Passive Pressure:

Soil in the interior will be drained.

Using $\phi = 30^\circ$ & $\delta = 10^\circ$: $K_p = 4.1$ $P_p = K_p \gamma H^2$

$P_p @ 10' = 0$

$P_p @ 22' = 4.1 \times 120 \times 12$
 $= 5904 \text{ psf}$



Stability of the Wall:

Overturning moment about the toe

$= 100 \times \frac{22^2}{2} + 500 \times \frac{12}{2} \times (\frac{12}{3} + 10) + 500 \times \frac{10^2}{2} = 210 \times \frac{2}{6} + 624 \times \frac{2}{6}$
 $= 24200 + 42000 + 25000 + 3500 = 104400$

$= 105100 \text{ lbs-ft}$

Resisting moment $= 5904 \times \frac{12}{2} \times \frac{12}{3}$
 $= 141696 \text{ lbs-ft}$

F.S. against overturning $= \frac{141696}{105100} = 1.35$

B.M. in the Wall:

Zero shear occurs at 15 feet from top (by trial)

$V = (\frac{100+600}{2}) \times 12 + 600 \times 3.08 + (21+62.4) \times \frac{3.08^2}{2} - 492 \times \frac{5.08^2}{2}$
 $= 4200 + 1848 + 395 = 6348 = 0$

Max B.M $= 100 \times \frac{15.08^2}{2} + 500 \times \frac{12}{2} \times (\frac{12}{3} + 3.08) + 500 \times \frac{3.08^2}{2}$
 $+ (21 + 62.4) \times \frac{3.08^2}{6} - 492 \times \frac{5.08^2}{6}$
 $= 11370 + 21240 + 2372 + 406 = 10750$
 $= 24638 \text{ lbs-ft} = 24.64 \text{ k-ft}$

Reinforcement $R_{reqd} = A_s = \frac{M}{f_s j d} = \frac{24.64 \times 12}{24 \times .87 \times 20.5} = 0.69 \text{ D}''$

$\# 7 @ 10''$
 $\# 6 @ 10''$

$A_s = 0.72 \text{ D}''$
I.F.



STS Consultants Ltd.
CALCULATION SHEET

PROJECT Flambeau Mine, Lady Smith, Wis

STS JOB NO.

SUBJECT Prelim. design of slurry wall

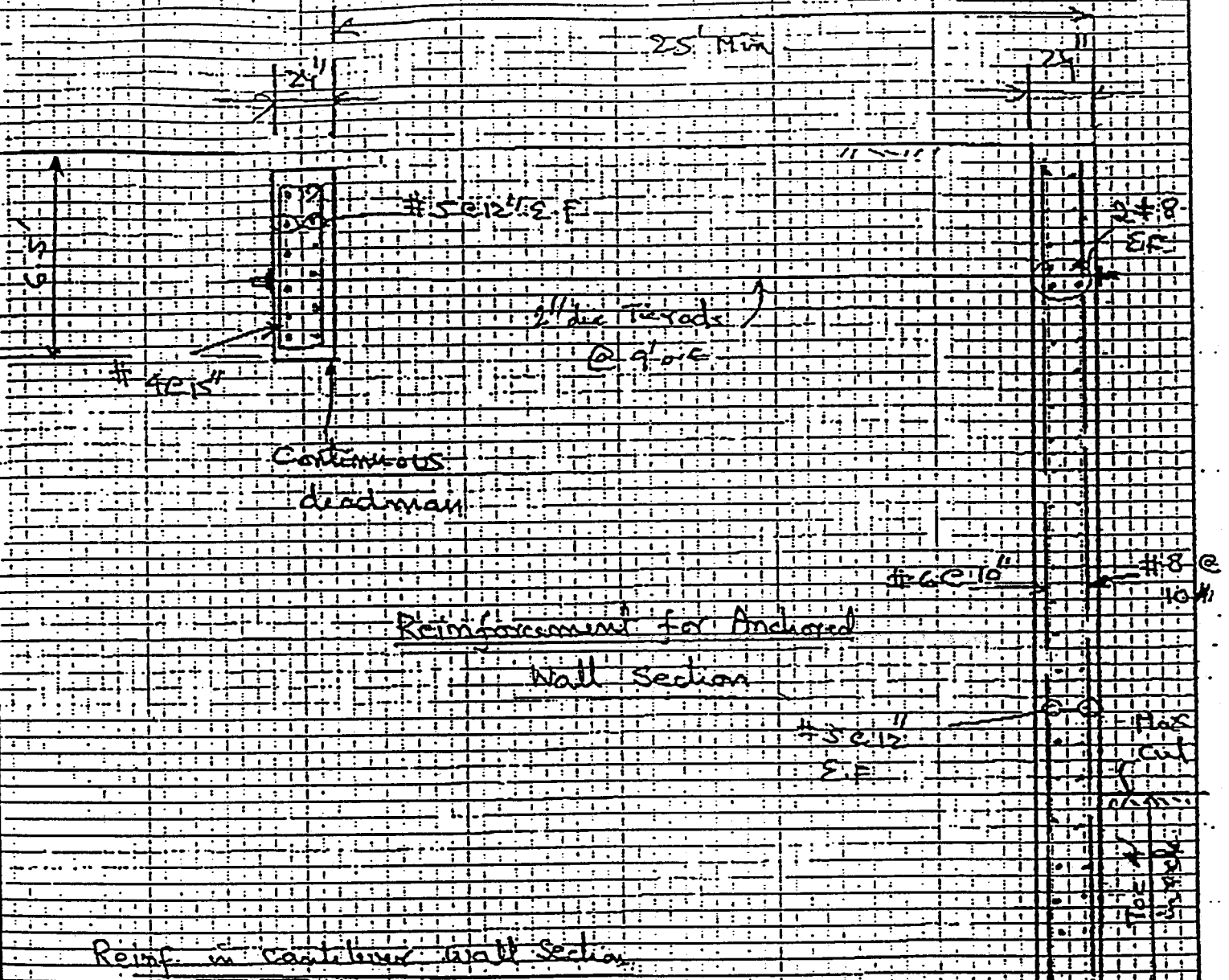
CALC. NO.

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DATE 4-21-92 BY SAG

CHECKED BY

REV. NO.



Reinf. in continuous wall section

Vertical # 7 @ 10' o.c.

6 @ 10" I.F.

Horizontal # 5 @ 12" E.F.

Slurry Wall

Appendix I

Slurry Concrete Diaphragm Wall Construction Specification

SECTION 00000 - SLURRY (CONCRETE DIAPHRAGM) WALLS

PART 1 - GENERAL

1.01 DESCRIPTION

- A. This Section specifies materials and workmanship requirements for the construction of diaphragm walls at locations shown on the Contract Drawings.
- B. Related work specified elsewhere:
 - 1. Earthwork
 - 2. Concrete Work

1.02 QUALITY CONTROL

- A. Comply with the applicable reference standards as specified herein.
- B. Qualifications. All work required under this Section shall only be performed by a Contractor experienced with bentonite slurry trench, diaphragm wall method of construction. Such experience shall have been obtained within the preceding five years on projects of similar nature, complexity and scope as to the subject work. The Contractor's experience record shall be submitted to the Engineer in writing for review, verification and approval.
- C. Tolerances
 - 1. Guide walls tolerances shall be as specified in Section 3.01.B.6.
 - 2. Diaphragm walls tolerances shall be as specified in Section 3.01.B.6.
 - 3. Reinforcing steel positioning tolerances shall be as specified in Section 3.01.B.6.
- D. Bentonite Certificate. With each shipment of bentonite delivered to the site, provide the Engineer with a certificate from the manufacturer of the bentonite, stating from which consignment the material delivered to the site has been taken.

- E. Control Testing of Bentonite Slurry. Perform control testing of bentonite slurry, using suitable apparatus, to determine the following parameters:
1. Freshly Mixed Bentonite Slurry. Measure density of freshly-mixed slurry as a check on the quality of slurry being formed.
 2. Bentonite Slurry Inside Trench Excavation. Perform tests specified in Section 3.02B to verify that properties of the slurry inside trench excavations are within the specified limits. Take samples from various locations within the trench.
 3. Bentonite Slurry in Trench Prior to Placing Concrete. Immediately prior to placing concrete in any wall panel, take a sample of the slurry 6-12 inches from the bottom of the trench, and test it for density. Modify or replace the slurry in the trench if the density of the sample is found to exceed 1.10 g/ml (68.7 lbs/ft³). Do not place any concrete in the trench until the density of the slurry at the trench bottom has been corrected and not to exceed 1.10 g/ml (68.7 lbs/ft³). Additional tests, such as measurement of sand content and fluid loss, shall be performed if requested by the Engineer.
 4. Maintain daily job records of tests.

1.03

SUBMITTALS

- A. Submittals shall be made as specified herein.
- B. Samples of the bentonite to be used by the Contractor, together with manufacturer's detailed test reports shall be submitted to the Engineer for approval before its use.
- C. Certificates for each consignment of all materials to be incorporated in the slurry shall be obtained from the suppliers and submitted to the Engineer. These shall state the grade and quality of the material in the consignment, and the results of all tests carried out thereon.

D. Construction Records:

1. The following records shall be kept for each wall panel and such records shall be available for inspection by the Engineer at any time:
 - (a) Panel Number;
 - (b) Date and time of start of panel excavation;
 - (c) Date and time of finish of panel excavation;
 - (d) Details of any obstruction encountered and the time spent in dealing with obstructions;
 - (e) Date and time of start and completion of cage placement;
 - (f) Date and time of start and completion of panel concreting;
 - (g) Length, width and depth of panel from top of guide wall level;
 - (h) Volumes of normal mix concrete including volume of concrete used at time of any interruptions in concrete supply where these exceed 15 minutes;
 - (i) Cut-off level of concrete below top of guide wall level;
 - (j) Concrete test cylinder markings, date and results obtained on testing;
 - (k) Details of cage type for reinforcement;
 - (l) A graph of calculated and measured concrete volumes with depth.

PART 2 - PRODUCTS

2.01 MATERIALS

A. Reinforcement

1. Reinforcement shall comply with the design drawings.

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2. Cages shall not entrap slurry during construction. The reinforcement shall be adequately fixed to avoid displacement and to maintain the minimum specified cover during concreting.
3. Welding of reinforcement shall be subject to the approval of the Engineer and conform to the relevant provisions and requirements of AWS D1.4 "Structural Welding Code for Reinforcing Steel".

B. Concrete

1. Concrete for diaphragm walls shall have a minimum cement content of 564 lbs/cy where the concrete is being placed by tremie methods.
2. Slump of the concrete shall be between 6 and 9 inches. The mix shall flow easily within the tremie pipe and be designed to produce a dense impervious concrete. Such concrete shall have a minimum cylinder strength of 4000 psi at 28 days.

C. Bentonite

1. Bentonite for use in the wall support slurry shall be in accordance with the latest edition of American Petroleum Institute Standard API-RP-13A.
2. Bentonite shall be stored in dry conditions. Particular care shall be taken with bulk storage to prevent balling together of bentonite powder or deterioration of properties due to dampness. A suitable design of hopper cone and bentonite feeding device shall be submitted for approval by the Engineer.
3. Composition: The bentonite shall be the high swelling sodium base bentonite consisting mainly of the clay mineral montmorillonite.

4. Purity:

Montmorillonite content: 90 percent minimum
Native sediments: 10 percent maximum

5. Chemical Composition:

Sodium montmorillonite: 60 percent minimum

Calcium and Magnesium
montmorillonite: 40 percent maximum

6. Sizing: Pulverized bentonite shall be ground to a fineness such that 80 percent minimum passes a US No. 200 mesh sieve in dry form.

D. Water

1. Water used for dispersion of bentonite shall be as nearly neutral in pH as practicable. Care shall be taken to use only fresh water when mixing bentonite.

E. Alternative Materials

1. The Contractor may submit proposals for additional or alternative ingredients and additives to the wall support slurry specified above. Samples and full details including the supplier and manufacturer of such ingredients shall be submitted. Any such materials shall not detrimentally affect the ability of the slurry to support wall excavation or have any detrimental effect upon concreting, or formed concrete or subsequent disposal of contaminated slurry. Ingredients to counteract loss of slurry to the surrounding strata shall be deemed covered by this Article.

PART 3 - EXECUTION

3.01 CONSTRUCTION

A. Excavation

1. Contractor shall provide all checking instrumentation necessary for the close and continuous checking of the movements of adjacent structures, foundations, services, and underground constructions.
2. Contractor shall ensure that his operations are conducted in such a manner as to minimize any spillage of slurry over the site.

3. Should an unknown obstruction be encountered during excavation, the Contractor shall immediately inform the Engineer and shall obtain approval from the Engineer for the method to be employed in removing the obstruction.
4. Bentonite powder shall be mixed thoroughly into the water by a suitable high shearing action mixer until all lumps have been broken up and dispersed within the mix. The proportion of bentonite to be included in the slurry shall take into account the geological properties of the soil such that the slurry shall be sufficient to maintain the stability of the excavation in the ground conditions encountered. The density and head of slurry above ground water shall together be such as to ensure the stability of the trench excavations throughout its entire depth.
5. Contaminated slurry, not suitable for re-use, shall be removed off site and disposed of in designated disposal areas in accordance with local state and federal environmental requirements.

B. Concrete Construction

1. Guide walls shall be constructed to establish the wall location and thickness. The top of the guide wall shall be not less than 4 feet above any standing ground water level and the wall shall extend to such a depth as will maintain stability of the upper soils during the panel excavation unless otherwise required or approved by the Engineer.
2. Before tremie concreting, the Contractor shall use inspection devices in the presence of the Engineer, to demonstrate that the breach has been completely excavated and that settling cuttings and excavated material have been removed. Remove concrete from previous concreting operation which has flowed into the trench.
3. All diaphragm walls shall be concreted to the following top levels:
 - (a) Underside of roof slab.

- (b) Where the top level of the wall is below the top of the guide wall, the concrete shall be brought up to a level at least one foot above the design level shown on the Drawings. This additional height of wall shall be cut back to the design level when the face of the wall is exposed.
4. Any bentonite remaining in the trench after concreting shall be displaced using sand or suitable material. Such backfill shall be brought up to the top of the guide walls.
5. Construction shall be carried out in accordance with the following tolerances:
- (a) The minimum distance between guide walls shall be the specified diaphragm wall thickness plus 1 inch and the maximum distance shall be the diaphragm wall width plus 2 inches.
- The guide walls shall be supported, as necessary, to maintain these tolerances during construction.
- (b) The plane of the wall face to be exposed shall be vertical to within a tolerance of 1 in 100. In addition to this tolerance, 5 inches shall be allowed for local protrusions beyond the wall face, resulting from irregularities in the ground.
- (c) The tolerances in positioning reinforcement shall be as follows:
- Longitudinal tolerance at cage head measured along the trench ± 2 inches.
- Vertical tolerance at cage head in relation to top of guide wall ± 2 inches.
- (d) A minimum cover to reinforcement of 3 inches shall be maintained at all levels.
- (e) Tolerances may be aggregated only to the extent that they do not exceed 6 inches.

C. Tremied Concrete

1. Start placement of tremie concrete in excavated panels within two hours after placing reinforcement cage. Proceed continuously until completion of panel concreting. If these time limits are exceeded. Remove reinforcement cage, clean, and reinstall.
2. The placing of concrete by tremie pipe shall be performed such that the discharge end of the tremie pipe is kept a minimum of 10 feet below the surface of the tremie concrete at all times except at the start of concreting. This shall be confirmed during concreting by soundings taken to the top of the concrete.
3. Contractor shall ensure that an adequate supply of concrete to the tremie is available at all times so that placement is continuous. The number of tremie tubes per panel shall be approved by the Engineer.
4. Where more than one tremie pipe is employed during concrete pouring to any one panel, the charging of concrete into the tubes shall be arranged so that it is evenly distributed between the tubes and so that no differential head exists at the concrete/slurry interface over the length of the panel. This level shall be confirmed by soundings taken during the concrete pour.

D. Panel Jointing

1. Panel jointing, inserted before placement of concrete in the panel, shall be clean and have a smooth regular surface. Where panel jointing is inserted in sections, adequate joint connections shall be provided to ensure verticality of the complete unit.
2. The extraction of panel jointing shall be carried out at such a time and in such a manner that no damage is caused to the concrete placed against them, or to the adjacent soil and structures.

00000 - 8

E. Joints

1. Where concrete is cast against previously completed wall panels, the previously formed concrete joint shall be cleaned so that adhering foreign substances are removed before the joint is cast. The joints between diaphragm wall panels shall be essentially watertight. When the joint is exposed upon subsequent excavation, the Contractor shall repair any joints which show active leakage of water.

F. Disposal of Slurry

1. Contractor shall be responsible for the disposal of used bentonite or contaminated slurry not suitable for re-use. The Contractor shall take adequate precautions during use of bentonite, transportation to and from work site, to avoid pollution or hazard to the general public. Slurry disposal shall be in accordance with local, state, and federal laws.

3.02

TESTS

A. Concrete Testing

1. Test cylinders shall be made and tested in accordance with ASTM C192, C31, C39 and C42. Two test cylinders shall be taken for each panel constructed. Cylinders shall have the same marks as the wall panel numbers and shall be sub-marked within each panel set.

B. Bentonite Testing

1. The test equipment employed shall in general be as specified in American Petroleum Institute Standard API-RP-13B. Additional or alternative equipment may be employed with the approval of the Engineer.
2. Freshly mixed bentonite shall be tested for density using a mud balance, and viscosity on a Marsh funnel.
3. Density measurements shall take into account the moisture content of the bentonite powder.

4. Samples shall be obtained from the panels during excavation and immediately before concreting.
5. pH shall be monitored during the addition of additives.
6. The range of values for slurry properties shall conform to that given in the following table unless otherwise agreed with the Engineer:

Marsh Funnel viscosity (second)	30	-	45
Apparent Viscosity (Centipoise)	15	-	21
Plastic Viscosity (Centipoise)	10	-	15
Density (lb/cu.ft.) (in use)	64	-	72
Sand Content (in use) (before concreting)	15 percent maximum 2 percent maximum		
pH	8.5	-	11.5

7. Density and viscosity shall be measured together with sand content from a sample taken at the base of the trench immediately before concreting. A pH meter shall be used for determination of pH values.

3.03 CORRECTIVE MEASURES

- A. If during the general excavation it is detected that the tolerances specified in Article 3.01.B.6 have been exceeded, the Contractor shall submit for approval by the Engineer his proposals for remedying the defects.