

# Testing of Heavy Brines

API RECOMMENDED PRACTICE 13J  
SECOND EDITION, MARCH 1996



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**Exploration and Production Department**

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## CONTENTS

	Page
1 SCOPE .....	1
2 REFERENCES .....	1
3 DEFINITIONS AND ABBREVIATIONS .....	1
3.1 Definitions .....	1
3.2 Abbreviations .....	1
4 REQUIREMENTS .....	1
4.1 Quality Control Instructions .....	1
4.2 Records Retention .....	1
5 CALIBRATION .....	1
5.1 Coverage .....	1
5.2 Equipment and Reagents .....	2
5.3 Calibration Intervals .....	2
5.4 Calibration Procedure—Thermometers .....	2
5.5 Calibration Procedure—Sieves, 75 Micrometers .....	3
5.6 Calibration Procedure—Hydrometers .....	3
6 DENSITY .....	5
6.1 Description .....	5
6.2 Mud Balance .....	5
6.3 Hydrometer .....	6
6.4 Calculation .....	6
7 BRINE CRYSTALLIZATION TEMPERATURE .....	7
7.1 Description .....	7
7.2 Equipment .....	9
7.3 Procedure .....	10
7.4 Calculation .....	12
8 BRINE CLARITY .....	12
8.1 Description .....	12
8.2 Laboratory Procedure—Preparation of Calibration Curve .....	13
8.3 Field Testing .....	15
Figures	
1—Calibration Curve for 75-Micrometer Sieve .....	4
2—Atmospheric Fluid Density Balance .....	7
3—Typical Pressurized Fluid Density Balance Diagram .....	8
4—Density Correction Versus Temperature .....	8
5—Temperature Curve for CaCl <sub>2</sub> Solutions .....	9
6—Representative Cooling Curve for a Brine .....	9
7—Typical Setup for Temperature Determination .....	10
8—Electronic Cooling System .....	11
9—Representative Turbidimeter .....	14
10—Example Calibration Curve (Range 0–1000 mg/L) .....	15
11—Example Calibration Curve (Range 0–2000 mg/L) .....	16
Tables	
1—Correction Values for 75-Micrometer Sieves .....	4
2—Hydrometer Calibration Example Data Sheet and Calculation .....	5
3—Dilution of Stock Solution .....	12

## Testing of Heavy Brines

### 1 Scope

This American Petroleum Institute standard covers the physical properties and test procedures for heavy brines manufactured for use in oil and gas well completion and workover fluids. This document is intended for the use of manufacturers of heavy brines.

### 2 References

The most recent editions of the following standards contain provisions which, through reference in this text, constitute provisions of this standard.

#### API

- RP 13B-1 *Recommended Practice Standard Procedure for Field Testing of Water-Based Drilling Fluids*

#### ASTM<sup>1</sup>

- D422 *Standard Test Method for Particle-Size Analysis of Soils*  
 E-11 *Standard Specification for Wire-Cloth Sieves for Testing Purpose*  
 E77 *Standard Test Method for Inspection and Verification of Thermometers*  
 E177 *Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods*

### 3 Definitions and Abbreviations

#### 3.1 DEFINITIONS

For the purposes of this standard, the following definitions apply:

**3.1.1 ACS reagent grade:** American Chemical Society (ACS) grade chemical meeting purity standards as specified by the ACS.

**3.1.2 flash side:** Side containing residue ("flash") from stamping; also, the side with concave indentations.

#### 3.2 ABBREVIATIONS

- ACS American Chemical Society  
 API American Petroleum Institute  
 ASTM American Society of Testing Materials  
 C<sub>r</sub> correction factor

- CAS chemical Abstracting Service  
 cm<sup>3</sup> cubic centimeters  
 D<sub>70</sub> corrected density at 70°F, lb/gal  
 D<sub>m</sub> measured density, lb/gal  
 D<sub>1</sub> density measured at lower temperature, lb/gal  
 D<sub>2</sub> density measured at upper temperature, lb/gal  
 °C degrees centigrade  
 °F degrees Fahrenheit  
 g gram  
 L liter  
 lb/gal pounds per gallon  
 mg milligram  
 NIST National Institute of Standards and Technology  
 NTU nephelometer turbidity units  
 rpm revolutions per minute  
 SG specific gravity  
 T<sub>m</sub> temperature at which density was measured, °F  
 T<sub>1</sub> lower temperature  
 T<sub>2</sub> upper temperature  
 TC to contain  
 TD to deliver  
 μm micrometer

### 4 Requirements

#### 4.1 QUALITY CONTROL INSTRUCTIONS

All quality control work shall be controlled by manufacturer's documented instructions, which include appropriate methodology and quantitative acceptance criteria.

#### 4.2 RECORDS RETENTION

All records specified in this document shall be maintained for a minimum of five years from the date of preparation.

### 5 Calibration

#### 5.1 COVERAGE

This section covers calibration procedures and calibration intervals for laboratory equipment and reagents specified. For laboratory items not listed, the manufacturer shall develop procedures where deemed appropriate.

**5.1.1** The manufacturer shall control, calibrate, verify, and maintain the laboratory equipment and reagents used in this standard for measuring product conformance.

**5.1.2** The manufacturer shall maintain and use laboratory equipment and reagents in a manner such that measurement uncertainty is known and meets required measurement capability.

<sup>1</sup>American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.

**5.1.3** The manufacturer shall document and maintain calibration procedures including details of laboratory equipment and reagent type, identification number, frequency of checks, acceptance criteria, and corrective action to be taken when results are unsatisfactory.

**5.1.4** The manufacturer shall establish and document responsibility for administration of the calibration program and responsibility for corrective action.

**5.1.5** The manufacturer shall document and maintain calibration records for laboratory equipment and reagents; shall periodically review these records for trends, sudden shifts, or other signals of approaching malfunction; and shall identify each item with a suitable indicator or approved identification record to show calibration status.

## 5.2 EQUIPMENT AND REAGENTS

**5.2.1 Volumetric glassware:** Laboratory volumetric glassware used for testing, including flasks and pipettes, are usually calibrated by the supplier. Manufacturers of products to this standard shall document evidence of glassware calibration prior to use. Supplier certification is acceptable. Calibration may be checked gravimetrically. Periodic recalibration is not required.

**5.2.2 Laboratory thermometers:** The manufacturer shall calibrate all laboratory thermometers used in measuring product conformance against a secondary reference thermometer. The secondary reference thermometer must show evidence of calibration as performed against NIST-certified master instruments in accordance with the procedures outlined by ASTM E77 and NBS (NIST) Monograph 150.

**5.2.3 Laboratory balances:** The manufacturer shall calibrate laboratory balances periodically in the range of use with NIST Class P, Grade 3 or better weights and shall service and adjust balances whenever calibration indicates a problem.

**5.2.4 Sieves:** Manufacturers shall calibrate 200-mesh (74-micrometer) sieves using API Test Calibration Barite with established values for residue retained.

**5.2.5 Hydrometers:** The manufacturer shall calibrate each hydrometer with the known density fluids.

**5.2.6 Mixers (Multimixer Model 9B with 9B29X impeller blades or equivalent):** The manufacturer shall verify that all spindles rotate at 11,500 revolutions per minute  $\pm 300$  revolutions per minute under no load with one spindle operating. Each spindle will be fitted with a single sine-wave impeller approximately 1 inch (25 millimeters) in diameter mounted flash side up. New impellers shall be weighed prior to installation, with weight and date recorded.

**5.2.7 Chemicals and solutions:** Must meet ACS reagent grade if available.

**5.2.8 Deionized (or distilled) water:** Manufacturer shall develop, document, and implement a method to determine hardness of water. The water shall not be used if hardness is indicated.

**5.2.9 API Test Calibration Materials:** Manufacturer shall test API Test Calibration Barite for properties listed with their Certificates of Analysis.

## 5.3 CALIBRATION INTERVALS

**5.3.1** Any instrument subjected to movement that will affect its calibration shall be recalibrated prior to use.

**5.3.2 Thermometers:** Calibrate each thermometer before it is put into service. After calibration, mark each thermometer with an identifying number that ties it to its corresponding correction chart. Check calibration annually against the secondary reference thermometer.

**5.3.3 Laboratory balances:** Calibrate each balance prior to being put into service. Check calibration at least once per month for six months, then at least once per six months if required measurements capability is being maintained. If not, service and recalibrate, then check at least once per month until required measurement capability is maintained for six months, then once per six months.

**5.3.4 Sieves:** Calibrate each sieve (where required; refer to 5.2.4) prior to being put into service. Check calibration at least once per 40 tests. After calibration, mark each sieve with an identifying number that ties it to its correction record. Since sieve calibration will change with use, maintain an up-to-date correction record.

**5.3.5 Hydrometers:** Calibrate each hydrometer prior to being put into service. After calibration, note and record each hydrometer identifying number that ties it to its correction chart. Periodic recalibration is not required.

**5.3.6 Mixers (for example, Multimixer 9B with 9B29X impellers or equivalent):** Check and record mixer spindle speed at least once every 90 days to ensure operation within the prescribed rpm range, using a phototachometer or similar device. Remove, clean, dry, and weigh each impeller blade in service at least once every 90 days. Record weight and replace blades when weight drops below 90 percent of its original value.

**5.3.7 Deionized (or distilled) water:** Manufacturer shall determine hardness of water whenever a new batch of water is prepared or purchased, or whenever deionized cartridges are replaced.

## 5.4 CALIBRATION PROCEDURE—THERMOMETERS

**5.4.1** Place thermometer to be calibrated side-by-side with secondary reference thermometer into a constant-tempera-

ture water bath (or suitable container of 1 gallon or more, filled with water, on a counter in a constant-temperature room) and allow to equilibrate for at least 1 hour.

**5.4.2** Read both thermometers and record readings.

**5.4.3** Repeat readings throughout at least a 1-hour interval to obtain a minimum of four readings.

**5.4.4** Calculate the average and the range of readings for each thermometer. The difference between the range of readings for each thermometer shall not exceed 0.2°F (0.1°C), or the smallest scale division on the thermometer being calibrated.

**5.4.5** Calculate average deviation of thermometer reading from secondary reference thermometer reading. Calculate and document correction for each thermometer.

## 5.5 CALIBRATION PROCEDURE—SIEVES, 74 MICROMETERS

**5.5.1** Obtain a 74-micrometer sieve with a *certified centerline value*.

**5.5.2** Take at least three samples of approximately 50 grams dry API Test Calibration Barite (TCB).

**5.5.3** Test each of the samples per Specification 13A, Section 7.9 using the certified sieve described in 5.5.1.

**5.5.4** Calculate percent retained for each sample by:

$$\% \text{ Residue, } R = 100 \frac{\text{Weight of residue, g}}{\text{Weight of sample, g}} \quad (1)$$

**5.5.5** Calculate average percent retains,  $S$ , of test calibration material on certified sieve:

$$S = \frac{R_1 + R_2 + R_3 \dots + R_N}{N} \quad (2)$$

Where:

$R_1 + R_2 + R_3 \dots + R_N$  = total of individual test results.

$N$  = number of samples tested.

Note: Individual sample values must agree within  $\pm 0.2$  of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat.

**5.5.6** From the intersection of percent retained and certified sieve opening size on the Sieve Calibration Graph (Figure 1), determine the calibration line (A, B, C, etc.) for use with the specific container of test calibration material. Record this and identify the container with this value.

**5.5.7** Repeat 5.5.2 through 5.5.4, except substitute the working sieve to be calibrated from the certified sieve.

**5.5.8** Calculate average percent retains,  $R_s$ , of test calibration material on work sieve:

$$R_s = \frac{R_1 + R_2 + R_3 \dots + R_N}{N} \quad (3)$$

Note: Individual sample values must agree within  $\pm 0.2$  of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat, beginning at 5.5.7.

**5.5.9** From Sieve Calibration Graph (Figure 1) determine the working sieve opening size to the nearest whole value from intersection of percent retained and the calibration line from 5.5.6 above. Record the working sieve opening size and identify the calibrated sieve and test calibration material container.

**5.5.10** Determine correction value ( $C$ ) for working sieve from Table 1. Record this value and identify it with the calibrated sieve and specified test calibration material container.

Note: The sieve correction value obtained from Table 1, as specified, is a number to be added to the retained value obtained on a test sample. (Negative values are subtracted.)

**5.5.10.1** Example of barite sieve correction value determination:

Certified Sieve Size = 73  $\mu\text{m}$

Test Calibration Barite Average Percent Retained on Certified Sieve,  $S = 2.0\%$

Calibration Line = F

Test Calibration Barite Average Percent Retained on Working Sieve,  $R_s = 1.3\%$

Working Sieve Size = 78  $\mu\text{m}$

Correction Value (from Table 1),  $C = +0.4\%$

**5.5.10.2** Example of sieve correction application:

Sieve Correction Value,  $C = +0.4\%$

Test Sample Retained,  $R_s = 2.8\%$

Corrected Test Sample Retained,  $R_c = 2.8 + 0.4 = 3.2\%$

## 5.6 CALIBRATION PROCEDURE—HYDROMETERS

**5.6.1** Calibrate each hydrometer to be used using the same concentration dispersant solution as used in the test, at temperatures spanning the anticipated test temperatures, and by reading the top rather than the bottom of the meniscus. Calibrate each hydrometer using the procedure below.

**5.6.2** Prepare 1 liter of dispersant solution as follows:

**5.6.2.1** Place 125 cubic centimeters  $\pm 2$  cubic centimeters (127 grams  $\pm 2$  grams) of dispersant solution into a 1-liter volumetric flask.

**5.6.2.2** Dilute to the 100 cubic centimeter mark with deionized water. Mix thoroughly.

**5.6.3** Place the dispersant solution in a sedimentation cylinder. Then place the cylinder in a constant-temperature



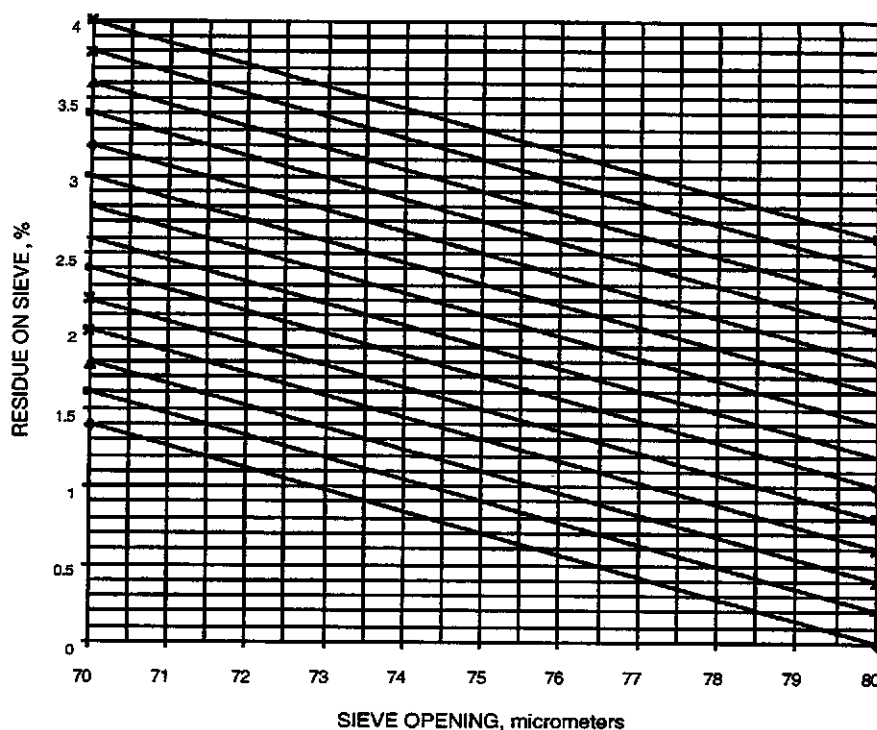


Figure 1—Calibration Curve for 75-Micrometer Sieve

bath. Set bath temperature to the lowest expected temperature for any actual test. Allow to reach equilibrium  $\pm 0.5^{\circ}\text{F}$  ( $\pm 0.2^{\circ}\text{C}$ ). Insert hydrometer to be calibrated and wait at least 5 minutes for the hydrometers and solution to reach bath temperature.

Table 1—Correction Values for 75-Micrometer Sieves<sup>a</sup>

Working Sieve Size Average Opening, <sup>c</sup> mm	Correction Size <sup>b</sup>
70	-0.7
71	-0.6
72	-0.4
73	-0.3
74	-0.1
75	0
76	+0.1
77	+0.3
78	+0.4
79	+0.6
80	+0.7

Note 1: These correction values are valid between 0 and 4 percent retained on sieve.

Note 2: Correction values are rounded to the nearest 0.1.

<sup>a</sup>ASTM Sieve specification allows  $\pm 5$  micrometers variation.

<sup>b</sup>Value to be added to test result of sample tested on sieve to convert results to equivalent 75 micrometers (Note: Negative values are subtracted).

<sup>c</sup>Determined from Sieve Calibration Graph (Figure 1).

5.6.4 Take a hydrometer reading at the top of the meniscus formed by the stem, and take a thermometer reading. Repeat readings at least 5 minutes apart so as to obtain a minimum of four readings each.

5.6.5 Calculate the average hydrometer reading and designate as  $R1$ . Calculate the average temperature reading and designate as  $t1$ .

5.6.6 Repeat 5.6.3 through 5.6.4, except set bath temperature to highest expected test temperature, calculate average hydrometer and temperature readings, and designate these readings as  $R2$  and  $t2$ .

5.6.7 Calculate the hydrometer correction curve slope,  $Mc$ , as follows:

$$Mc = \frac{1000 (R1 - R2)}{(t2 - t1)} \quad (4)$$

Where:

$R1$  = average hydrometer reading at lower temperature.

$R2$  = average hydrometer reading at higher temperature.

$t1$  = average temperature reading at lower temperature.

$t2$  = average temperature reading at higher temperature.

Note: Temperature may be measured in either  $^{\circ}\text{F}$  or  $^{\circ}\text{C}$  so long as all measurements and calculations are consistent in units.

**5.6.8** Calculate the hydrometer correction curve intercept,  $B_c$ , as follows:

$$B_c = (M_c \times r_1) + [(R_1 - 1) \times 1000] \quad (5)$$

Where:

$M_c$  = hydrometer correction curve slope.

$r_1$  = thermometer reading at lower temperature.

$R_1$  = hydrometer reading at lower temperature.

**5.6.9** Record  $M_c$ ,  $B_c$ , and hydrometer serial number in permanent calibration record. See Table 2.

## 6 Density

### 6.1 DESCRIPTION

**6.1.1** The composition of a brine (salt type and concentration) determines its density. The density of brine, in turn, determines the hydrostatic pressure that a column of brine exerts downhole.

**6.1.2** The density of a brine is the mass of brine per unit of volume. Density is generally reported as pounds mass per gallon or grams per cubic centimeter. Density may also be expressed in terms of specific gravity or pressure gradient. Specific gravity is the ratio of the weight of a specific volume of a material when compared to an equal volume of a reference material at the same temperature. For brines, the reference material is water. Pressure gradient is the hydrostatic pressure exerted by the fluid per unit of length (that is, pounds per square inch per foot).

**6.1.3** The reference temperature for density reporting of heavy brines is 70°F (21.1°C). Brine density is affected by temperature and decreases with increasing temperature. The amount of decrease will depend on the brine composition. See 6.3.3.

Table 2—Hydrometer Calibration Example  
Data Sheet and Calculation

Hydrometer Serial No.	Temperature °C (°F)	Reading (R)	Corr. Curve Slope (M <sub>c</sub> )	Corr. Curve Intercept (B <sub>c</sub> )
xxxx	$r_1 = 26.0$	$R_1 = 1.0017$	0.2600	8.46
	$r_2 = 31.0$	$R_2 = 1.0004$		

Certified by: \_\_\_\_\_

Date: \_\_\_\_\_

Using Equation 4:

$$M_c = \frac{1.0017 - 1.0004 \times 1000}{31.0 - 26.0} = 0.2600$$

Using Equation 5:

$$B_c = (0.2600 \times 26.0) + (1.0017 - 1)(1000) = 8.46$$

**6.1.4** The measurement of the density of brines will be affected by entrapped gasses. If gas entrainment is a problem, it is recommended that the procedures outlined in API Recommended Practice 13B-1, Appendix D be used. These procedures use a pressurized fluid density balance and/or de-aerator to aid in the density measurement.

**6.1.5** Any instrument of sufficient accuracy to permit measurement to within  $\pm 0.05$  pounds per gallon (0.005 grams per cubic centimeter) and reportable to within  $\pm 0.1$  pounds per gallon (0.01 grams per cubic centimeter) may be used. Generally this will require the use of hydrometers for fluid density measurements. An atmospheric or pressurized mud balance may be used, but lacks sufficient accuracy to meet the requirements of this section. See 6.2.1 through 6.2.3.

### 6.2 MUD BALANCE

#### 6.2.1 Equipment

**6.2.1.1** The atmospheric mud balance (Figure 2) is a simple counterweight instrument. The weight of the mud cup filled with brine is balanced by a fixed counterweight and rider that is free to move on a graduated scale. A level bubble is mounted at the pivot point on the beam. Attachments are available to extend the range of the balance.

**6.2.1.2** The atmospheric and pressurized mud balances (Figure 3) are useful for determining density trends (increases or decreases) in the field, but is not sufficiently accurate for absolute determination of brine density. For purposes of absolute density determination, the use of a set of calibrated hydrometers is recommended over a mud balance.

#### 6.2.2 Procedure

**6.2.2.1** The mud balance base shall be set up approximately level.

**6.2.2.2** Fill the clean, dry cup with the brine to be tested. Put on and rotate the cap until firmly seated. Make sure some of the brine is expelled through the hole in the cap to free trapped air or gas.

**6.2.2.3** Wash or wipe the brine from the outside of the cup.

**6.2.2.4** Place the beam on the support and balance it by moving the rider along the graduated scale. The beam is horizontal when the bubble is under the center line.

**6.2.2.5** Read the density at the side of the rider toward the knife edge. Make appropriate corrections when a range extender is used.

#### 6.2.3 Procedure—Calibration

This instrument shall be calibrated frequently with fresh water. Fresh water when measured at 70°F (21.1°C) shall give a reading of 8.33 pounds per gallon (0.998 grams per cubic centimeter).

## 6.3 HYDROMETER

### 6.3.1 Equipment

**6.3.1.1** A set of glass hydrometers covering the range of specific gravities encountered in heavy brine applications. Graduations shall be in specific gravity units (dimensionless) not greater than 0.002.

Note: 1: The hydrometer is a weighted bulb with a graduated stem. The depth to which the hydrometer sinks in a fluid is determined by the density of the fluid. The specific gravity is read directly from the graduated stem. Any oil present in the sample will interfere with the determination; therefore, only freshly filtered samples should be used.

Note: 2: Although hydrometers with built-in thermometers may be used, it is recommended that the sample be brought to 70°F (21.1°C) to avoid conversion errors.

**6.3.1.2** Cylindrical sample container of either clear glass or plastic. For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 1 inch (2.54 centimeters) greater than the outside diameter of the hydrometer used. The height of the cylinder shall be such that the hydrometer floats in the sample with at least 1 inch (2.54 centimeters) clearance between the bottom of the hydrometer and the bottom of the cylinder.

**6.3.1.3** Thermometer: 32–220°F ± 2°F (0–105°C ± 1°C).

Note: Temperature of the brine at measurement should be 70°F (21.1°C). If any other temperature must be used due to local conditions, appropriate corrections shall be made.

### 6.3.2 Procedure

**6.3.2.1** Pour the sample into the clean cylinder without splashing so as to avoid formation of air bubbles. Remove any air bubbles adhering to the surface of the brine by touching them with a piece of clean filter paper.

**6.3.2.2** Select a location that is free from wind and air currents.

**6.3.2.3** Place the hydrometer vertically in the brine contained by the sample container.

**6.3.2.4** After the hydrometer has come to rest and floats freely away from the walls of the cylinder, read the specific gravity as the point at which the surface of the sample apparently cuts the hydrometer scale.

**6.3.2.5** Determine the temperature of the sample just after reading the hydrometer.

### 6.3.3 Procedure—Correction to 70°F (21.1°C)

**6.3.3.1** The measured density of the brine should be corrected to 70°F (21.1°C) if the measurement was taken at any other temperature. When density is reported in pounds per gallon, the following formulas can be used for this correction:

$$D_{70} = D_m + (T_m - 70) \times C_f \quad (6)$$

$$C_f = (-5.9659 \times 10^{-4}) + (8.341 \times 10^{-4}) \times D_m - (6.904 \times 10^{-5}) \times D_m^2 + (2.1795 \times 10^{-6}) \times D_m^3 \quad (7)$$

Where:

$D_{70}$  = corrected density at 70°F, lb/gal.

$D_m$  = measured density, lb/gal.

$T_m$  = temperature at which density was measured, °F.

$C_f$  = correction factor.

**6.3.3.2** The correction factor for a particular fluid can also be calculated by measuring the density of the brine at two or more widely separated temperatures. The calculated correction factor is then substituted into Equation 6. The formula for this calculation is:

$$C_f = \frac{(D_1 - D_2)}{(T_2 - T_1)} \quad (8)$$

Where:

$C_f$  = correction factor.

$D_1$  = density measured at lower temperature, lb/gal.

$D_2$  = density measured at upper temperature, lb/gal.

$T_1$  = lower temperature.

$T_2$  = upper temperature.

**6.3.3.3** An additional field method for correction of brine density is to develop a curve comparing the measured brine density at various temperatures. The curve may then be used for density conversion back to 70°F (21.1°C). Figure 4 is an example of one such brine correction curve. Note that this curve will be specific for the particular brine being tested and cannot be applied to any other fluid, nor can it be applied to the fluid after its composition has been altered (that is, dilution, increase in density).

Regardless of the method selected, care must be taken to accurately measure the density and the temperature of the brine. Minor variations in either of these measurements will lead to errors in the calculations or graphs used for density correction.

Note: These methods are to be used to correct the density measured at surface and are not intended for use to calculate the required density downhole nor the effective hydrostatic pressure exerted by a column of the fluid.

### 6.3.4 Procedure—Calibration

The set of hydrometers in use may be calibrated with a series of brines of known density.

## 6.4 CALCULATION

**6.4.1** Report the brine density to the nearest 0.1 pound per gallon (0.01 gram per cubic centimeter).

**6.4.2** To convert the density reading to other units, use the following conversions:

$$\text{Density, g/cm}^3 = \frac{\text{lb/gal}}{8.345} \quad (9)$$

$$= \frac{\text{lb/ft}^3}{62.43}$$

$$\text{Mud Gradient, psi/ft} = \frac{\text{lb/ft}^3}{144} \quad (10)$$

$$= \frac{\text{lb/gal}}{19.24}$$

$$= \frac{\text{g/cm}^3}{2.31}$$

$$\text{Density, g/cm}^3 = \text{Specific Gravity} \times \rho_w \quad (11)$$

Where:  $\rho_w = 0.998$ , density of water at 70°F (21.1°C).

## 7 Brine Crystallization Temperature

### 7.1 DESCRIPTION

**7.1.1** The recommended practice in this section describes a method for the measurement of crystallization temperatures of brines used in completion, workover, and drilling operations.

**7.1.2** Because of the nature of the brine cooling curve, three different crystallization temperatures are often quoted for the same brine. The following defines the three crystallization temperatures, discusses the practical significance of

each crystallization temperature, and explains the difference between crystallization of salt crystals from a brine and the freezing of water from a brine.

**7.1.3** The actual crystallization temperature of a brine is that temperature at which a solid will begin to form out of solution if given sufficient time and proper nucleating conditions. The solid may be either salt or freshwater ice.

**7.1.4** As brines are normally formulated, the crystallization temperature is the temperature at which the brine is saturated with one or more salts that it contains. At this crystallization temperature, the least soluble salt's solubility is exceeded and crystallizes. Cooling the brine below the crystallization temperature results in additional formation of salt crystals. Users of brines normally specify a crystallization temperature near or below the lower anticipated temperature to prevent crystallization of the brine.

**7.1.5** Precipitation of salt crystals in brines at or below the crystallization temperature can lead to a number of problems. Rapid plugging of filtration units can occur. If salt crystals settle in the tank, the density of the brine pumped downhole may be insufficient to control formation pressures. As additional crystals form, brine viscosity may increase. The viscosity can become so high that the brine appears to be a frozen solid. This can cause line plugging, fluid in tanks becoming solid, and pump seizure. Heating the brine redissolves the crystals with no permanent change in the brine properties.

**7.1.6** Since salt crystals have a smaller specific volume than the brine, the brine will not expand in volume during crystallization, and there will not be any ruptured lines, valves, or pump heads, which can occur when water freezes.

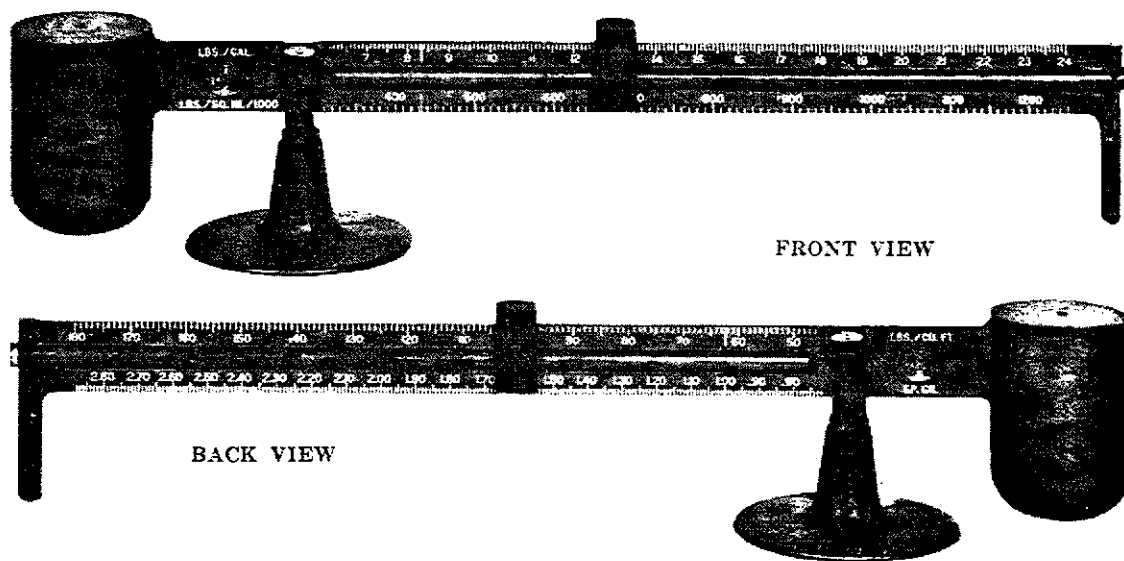


Figure 2—Atmospheric Fluid Density Balance

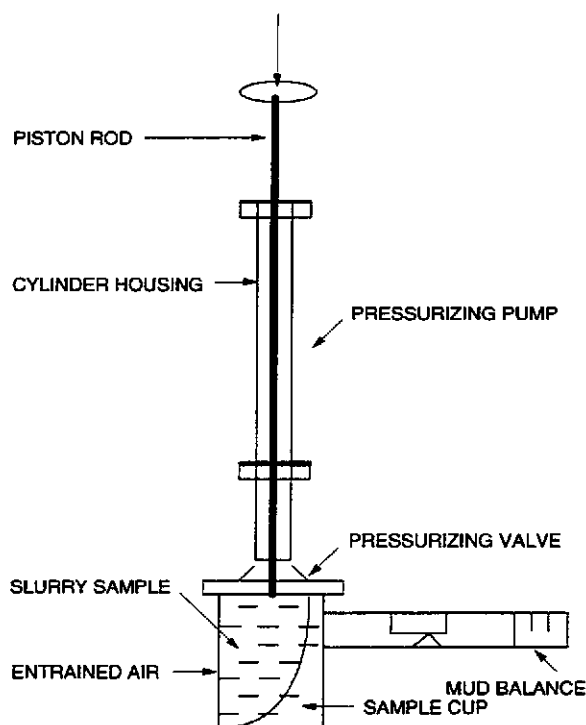


Figure 3—Typical Pressurized Fluid Density Balance Diagram

**7.1.7** The crystallization temperature of a brine at a given density can be varied by adjusting the concentrations and composition of the salts in the brine. Consequently, brines of a given density may be formulated with numerous crystallization temperatures. Generally the lower crystallization temperature brines will be more costly. Design of a brine with an excessively low crystallization temperature can significantly increase the cost of the brine. A lower cost brine with an excessively high crystallization temperature can increase costs due to problems caused by crystallization of the fluid in the pumps, lines, filtration units, and tanks.

**7.1.8** With dilute brines (for example, seawater) the salt in the water depresses the crystallization temperature or the freezing point of the brine. The temperature at which fresh-water ice will form is reduced by the salts.

**7.1.9** Figure 5 shows the crystallization temperature of calcium chloride solutions as a function of concentration. The crystallization temperature of the brine decreases to as low as  $-59^{\circ}\text{F}$  ( $-51^{\circ}\text{C}$ ) at a concentration of 30 percent calcium chloride (10.8 pounds per gallon, 1.29 specific gravity). In this portion of the curve, temperatures below the crystallization temperature result in the formation of fresh-water ice, producing a brine with a higher salt concentration.

**7.1.10** At concentrations above 30 percent calcium chloride, the crystallization temperature of the brine begins to in-

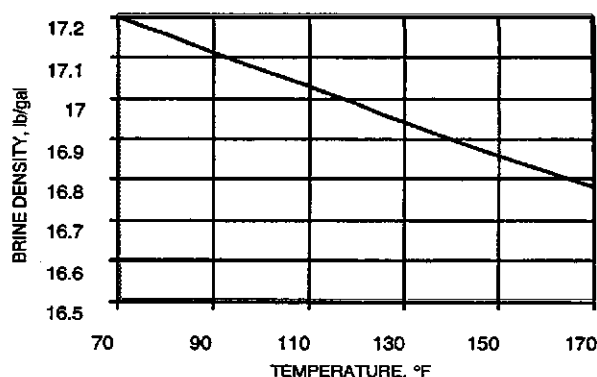


Figure 4—Density Corrections Versus Temperature

crease. This portion of the curve represents the solubility limit of calcium chloride in water. The crystallization temperature increases with increasing calcium chloride concentration.

**7.1.11** Another way of evaluating the right-hand portion of the curve in Figure 5 is that as the temperature of the brine is decreased, the calcium chloride becomes less soluble. Calcium chloride precipitates out of solution, resulting in a brine with a reduced salt concentration and lower density.

**7.1.12** All experimental methods for measuring the crystallization temperature of a brine involve alternately cooling and heating the brine. Figure 6 is a representative cooling curve for a brine. Measured temperature of the brine is plotted with time while the brine is alternately cooled and heated. Three experimental measurements of crystallization temperature are identified on Figure 6. These values for crystallization temperature are defined as follows:

- a. **FCTA (First Crystal to Appear):** The temperature corresponding to a minimum in a plot of temperature during cooling, or the temperature at which visible crystals start to form. FCTA will generally include some supercooling effect (cooling below the actual crystallization temperature).
- b. **TCT (True Crystallization Temperature):** In a plot of temperature during cooling cycle, the maximum temperature reached following the supercooling minimum, or the inflection point in cases with no supercooling. If there is no supercooling, TCT will equal FCTA.
- c. **LCTD (Last Crystal to Dissolve):** In a heating cycle, the temperature at which crystals disappear, or the inflection point on the heating curve.

**7.1.13** TCT is the measured crystallization temperature nearest the temperature at which a brine will crystallize in pumps, lines, filtration units, and tanks. LCTD is the measured crystallization temperature nearest the temperature at which crystals that have formed will redissolve.

**7.1.14** The accuracy of the crystallization temperature testing method depends on several factors, most importantly

the supercooling control. Supercooling may be minimized by slow cooling rates and nucleation of crystallization with selected solid surfaces.

**7.1.15** When a brine is cooled below its actual crystallization temperature, crystals may not form spontaneously due to lack of nucleation sites. This is the supercooling effect. The use of selected solid surfaces can initiate the nucleation of crystallization. Barium oxide, barium hydroxide, calcium carbonate, and bentonite have been found to be effective nucleators for brines. Only a very small amount is required to reduce the amount of supercooling.

**7.1.16** As crystals start to form at the FCTA, the heat released by the crystallization process increases the temperature of the brine. At the same time, the formation of the solids lowers the concentration of salt in the remaining brine. The temperature stops increasing when it reaches the crystallization temperature of the fluid remaining at that point, not the crystallization temperature of the original sample.

**7.1.17** The maximum temperature following supercooling is the actual crystallization temperature of the brine remaining at that point, not the TCT of the original sample. If significant supercooling occurs (that is, TCT exceeds FCTA by 5°F [3°C] or more), the measurements should be repeated at a slower cooling rate. Investigators have found that samples that supercool on the first cycle often do not supercool on subsequent cycles.

**7.1.18** The heat of dissolution is responsible for the inflection point at LCDT that can be observed as a crystallized

solution is warmed. As the brine is heated, crystals absorb thermal energy in dissolving, thus slowing the heating rate of the solution. When all the crystals have redissolved at LCDT, the brine will warm more rapidly.

**7.1.19** When measuring the crystallization temperature of brines, one must be aware that minor components may precipitate from solution before the TCT. For example, both sodium chloride and potassium chloride are less soluble than calcium chloride, calcium bromide, or zinc bromide. If a brine is contaminated with sodium chloride, the small amount of sodium chloride may crystallize and turn the brine cloudy at a temperature much higher than the TCT. If the sodium chloride is truly a minor component, it would not produce the rig problems mentioned above. Reformulation of the brine to reduce the faulty TCT could prove costly.

**7.1.20** In summary, the best measure of the crystallization temperature of a brine is the TCT. This measured crystallization temperature best represents the temperature at which crystals will precipitate from a brine. FCTA and LCDT may also be specified. FCTA will generally be lower than TCT, and LCDT will generally be higher than TCT. The difference between FCTA and TCT represents the degree of supercooling, and if this difference exceeds 5°F (3°C), repeat the measurements at a slower cooling rate.

## 7.2 EQUIPMENT

A typical crystallization temperature measurement setup is shown in Figure 7. The following equipment is recommended:

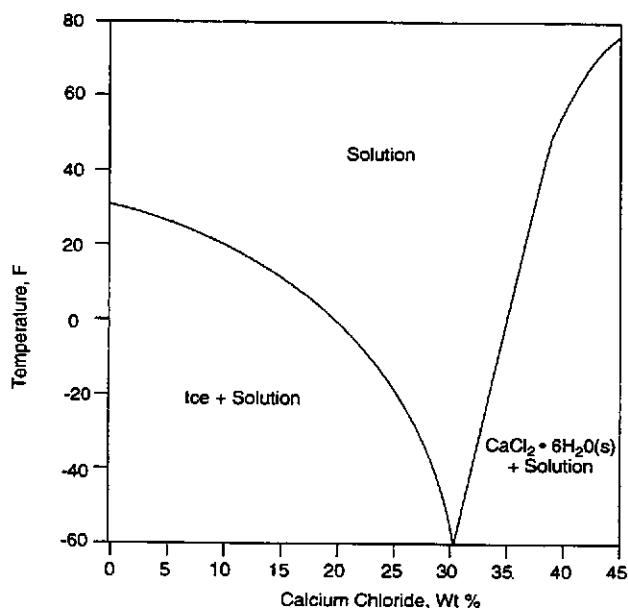


Figure 5—Temperature Curve for  $\text{CaCl}_2$  Solutions

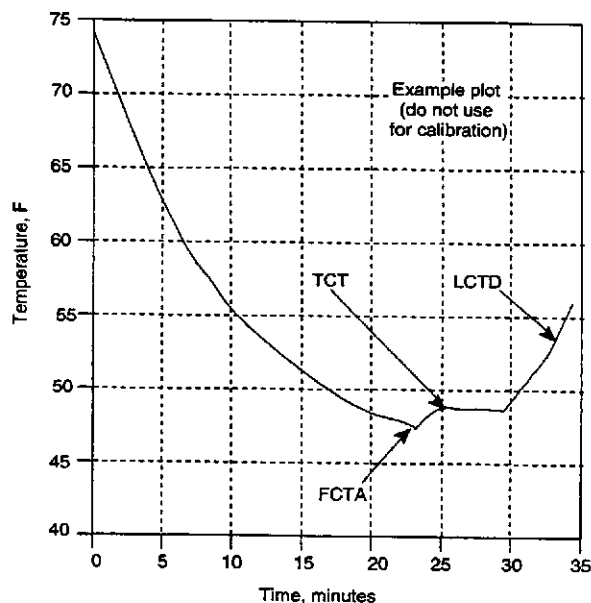


Figure 6—Representative Cooling Curve for a Brine

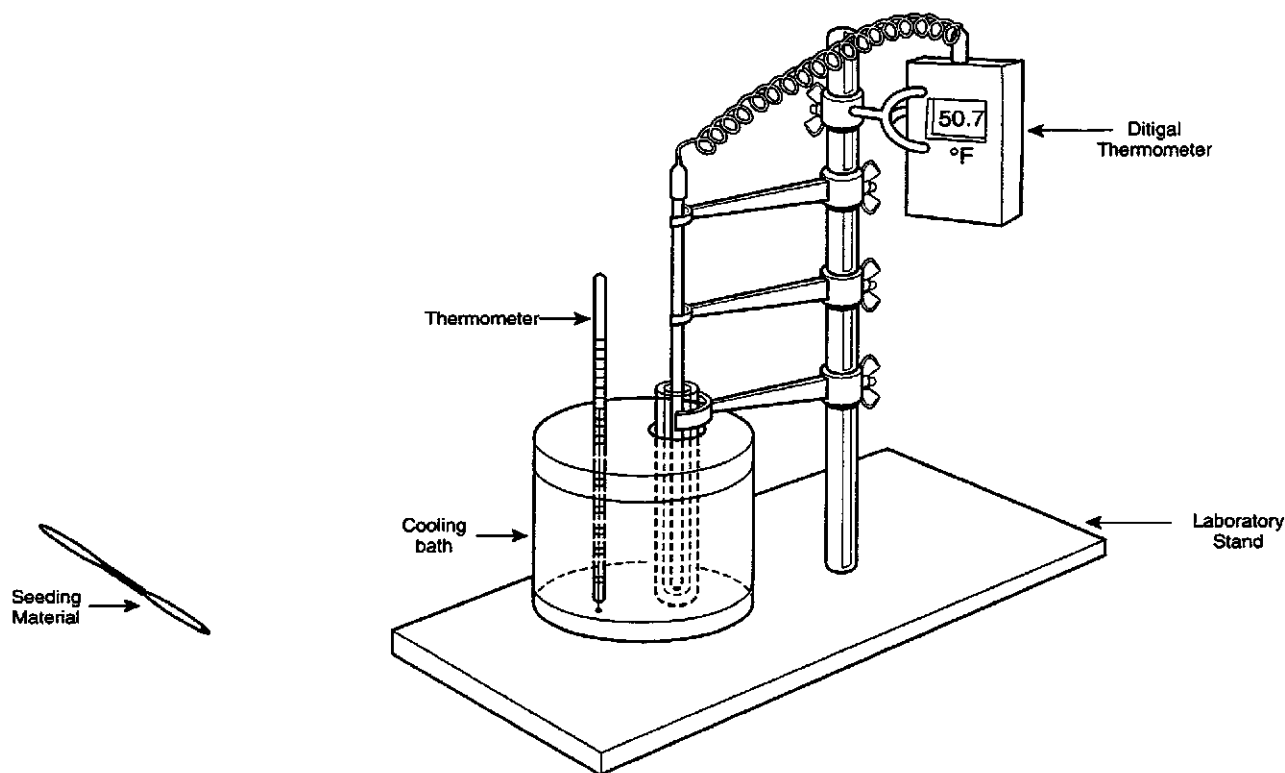


Figure 7—Typical Setup for Temperature Determination

### 7.2.1 Digital thermometer with thermistor probe. Specifications:

Indicating range:  $-50$  to  $+100^{\circ}\text{F}$  ( $-45$  to  $38^{\circ}\text{C}$ ) minimum

Resolution:  $0.1^{\circ}\text{F}$  ( $0.05^{\circ}\text{C}$ )

**7.2.2** Sample container of sufficient volume to hold at least 25 cubic centimeters. (An example container is a  $25 \times 150$  millimeter test tube that will fit inside a  $20 \times 150$  millimeter tube.)

**7.2.3** Cooling bath: to limit supercooling. Cool the sample slowly. The cooling bath should be no more than  $20^{\circ}\text{F}$  ( $11^{\circ}\text{C}$ ) below the FCTA temperature and ideally use a cooling rate of less than  $1^{\circ}\text{F}$  ( $0.5^{\circ}\text{C}$ ) per minute. This may be achieved through the use of an electronic cooling system or through the use of one or more of the following:

- Ice/water slurry (50/50 volume per volume ice and water). The temperature of the bath will be  $32^{\circ}\text{F}$  ( $0^{\circ}\text{C}$ ). Use if FCTA temperature is above  $35$  to  $40^{\circ}\text{F}$  ( $2$  to  $5^{\circ}\text{C}$ ).
- Ice/saltwater slurry (50/50 volume per volume ice and saltwater; NaCl solution should contain 30 grams NaCl in 90 cubic centimeters water). Temperature of the bath will be

approximately  $5$  to  $10^{\circ}\text{F}$  ( $-15$  to  $-12^{\circ}\text{C}$ ). Use if the FCTA temperature is approximately  $10$  to  $40^{\circ}\text{F}$  ( $-12$  to  $4^{\circ}\text{C}$ ).

c. Antifreeze/water slurry (50/50 volume per volume antifreeze/water mixture). Cool the bath by placing the container in a larger container with a dry ice/acetone bath. Cool to a temperature approximately  $15^{\circ}\text{F}$  ( $9^{\circ}\text{C}$ ) below the FCTA temperature.

d. Ice/ $\text{CaCl}_2$  slurry (50/50 volume per volume crushed ice and  $\text{CaCl}_2$  solution.  $\text{CaCl}_2$  solution should contain 50 grams  $\text{CaCl}_2$  in 50 grams water): The temperature of the bath will approach  $-40^{\circ}\text{F}$  ( $-40^{\circ}\text{C}$ ) as the ice melts.

**7.2.4 Seeding material:** 0.1 percent by weight of an inert, insoluble nucleating agent (see 7.1.15). Use approximately 0.03 grams, equivalent to a few grains of salt, for 25 cubic centimeters of brine.

## 7.3 PROCEDURE

Note: Alternatively, when using an electronic cooling system, follow the manufacturer's recommended test procedure. See Figure 8.

**7.3.1** Measure approximately 25 cubic centimeters of the brine to be tested into the sample container. Add the nucleating agent to the sample. Place the sample container inside



Figure 8—Electronic Cooling System



an outer container and place both in the cooling bath with the thermometer probe inserted in the brine.

Note: The outer container will help to slow the cooling rate of the sample.

**7.3.2** Stir the brine and record the temperature decrease of the sample. The temperature of the brine will decrease steadily until crystals begin to form. Record the minimum temperature reached before crystallization as the FCTA temperature.

**7.3.3** Record the maximum temperature achieved immediately after crystallization has occurred as the TCT. The temperature should stabilize briefly (10 to 20 seconds). If the temperature does not stabilize, or if large amounts of supercooling are evident (TCT minus FCTA is greater than 5°F [3°C]), the test should be repeated using a cooling system at a warmer initial temperature.

**7.3.4** Discontinue cooling the sample by removing the sample container from the bath or reducing the temperature setting of the cooling plate, and allow the sample to warm. Watch for the dissolution of crystals formed during the cooling cycle. When all of the crystals have dissolved, record the temperature as the LCTD temperature.

**7.3.5** Repeat the procedure at least three times using the same sample. Once an estimation of TCT is known, the sample may be cooled rapidly to within 10°F (6°C) above the TCT.

**7.3.6** Record the average of at least three tests. Do not include any data where supercooling of the sample was evident.

## 7.4 CALCULATION

Report the temperatures for FCTA, TCT, and LCTD.

## 8 Brine Clarity

### 8.1 DESCRIPTION

**8.1.1** Clarity is a relative expression referring to the turbidity of a brine due to the presence of suspended insoluble or immiscible particulate matter.

**8.1.2** This procedure concerns standard methods for the measurement of brine turbidity and the use of turbidity measurements with a solids calibration curve to estimate solids concentration of a representative sample of clear brine. Concentration, particle size distribution, and type of solids may affect the utility of the brine. Minimum and/or maximum limits on contaminating solids is left to the supplier and user.

Note: No conclusions on the effect of the particle size of the solids on formation damage are intended to be drawn or implied from any details of this procedure.

**8.1.3** Brine sampling is complicated because of the possible presence of large particles of contaminants; the instabil-

ity of certain dissolved ions, such as iron; and the overall influence of a variety of minerals, materials, and residues on brine quality. Fluids weighted with calcium carbonate or other weighting agents are excluded from this procedure. Readings may also be affected by colored additives, such as some corrosion inhibitors.

**8.1.4** Brine clarity is important in the following determinations: formation damage potential; isolation of a source of contamination in the course of manufacture, transport, storage, or wellsite use; or providing a basis for which specifications or limits may be placed on the concentration and/or types of solids found in water or brines.

**8.1.5** The primary objective of the test is to allow onsite estimation of solids content by measuring brine turbidity. Turbidity is not a direct measure of concentration of suspended solid particles but a measure of the scattering effect such particles have on light. Since turbidity readings depend on particle size, size distribution, particle shape, particle refractive index, brine refractive index, and other factors in addition to particle concentration, any estimated correlation of turbidity to weight concentration of suspended solids can be done only if a calibration curve is generated. This calibration curve is determined in the laboratory using samples of solids that are expected to contact the brine during the wellsite use and the actual brine composition to be used on the well.

**8.1.6** This test method is a compromise to accommodate measurement at the wellsite. Direct gravimetric measurement of particle solids concentration is more accurate, but gravimetric analysis onsite is impractical. Onsite measurement of turbidity with a portable nephelometer is quick and easy. Preplanning is required to generate a calibration curve in the laboratory with wellbore solids. This calibration curve method introduces some inaccuracy because of the assumption that the suspended solids variables remain constant and that only the concentration changes. Field testing has shown that this turbidity method may be a useful way to evaluate brine quality. It is most often used to decide when to stop filtering a brine after displacement.

**8.1.7** The procedure recommended here will only place a relative value on the presence or absence of solids in a particular sample. It should also be cautioned that in transferring brines from one tank to another, or perhaps sampling during the course of several downhole displacements, the turbidity and type of solids will probably vary significantly

Table 3—Dilution of Stock Solution

Dilution Ratio Nonfiltered Fluid	Stock Solution, cm <sup>3</sup>	Completion Brine, cm <sup>3</sup>
0/100	0	100
25/75	25	75
50/50	50	50
75/25	75	25
100/0	100	0

from one sample to another. It is extremely important to be aware of the actual event that a particular sample represents.

Note: Coarse matter greater than 200 mesh (74 micrometers) is excluded as a turbidity factor. This procedure may not be applicable in the presence of fluid loss additives or thickeners. In some cases, NTU (nephelometer turbidity unit) values will be affected by color.

## 8.2 LABORATORY PROCEDURE— PREPARATION OF CALIBRATION CURVE

### 8.2.1 Equipment

- a. Balance: analytical laboratory, precision of 0.0001 gram.
- b. Petri dishes: eleven.
- c. Oven: drying, regulated to 220°F (105°C).
- d. Desiccator.
- e. Volumetric flasks (TC): one 1000-cubic centimeters and ten 100-cubic centimeters.
- f. Pipette (TD): one 25-cubic centimeters.
- g. Filter disks: 47 millimeters, 1.2 micrometers.
- h. Forceps.
- i. Vacuum filtration device: funnel to accommodate 47-millimeter filter disks, funnel clamp, 1000-cubic centimeter vacuum flask, and fritted disks.
- j. Vacuum pump.
- k. Deionized or distilled water.
- l. Wellbore solids: 500-cubic centimeter sample from well to be completed. Solids may be obtained from the shale shaker, mud pit, bottom of transport tank, or other appropriate place. Pass the wellbore solids through a 200-mesh (74-micrometers) sieve (that is, sand content screen) to remove the coarsest particles. The remaining sample normally contains enough mud, barite, drill solids, rust, and so forth, to give sufficient concentration of particles less than 74 micrometers in diameter to produce a useful calibration curve.
- m. Completion brine: 2000-cubic centimeter (filtered through a 1.2-micrometer filter) sample of the clear brine that will be used for completion.
- n. Turbidimeter: The turbidimeter shall be of the nephelometer type and measure scattered light using two photodiodes positioned at an angle of 90 degrees to the incident light beam. The source of light shall be a tungsten filament bulb operating at a color temperature of 2000–3000 Kelvin. The light path shall be perpendicular to the axis of the sample tube. The model of turbidimeter used in the field should be the same model used in the laboratory to generate the calibration curve. Different models give different turbidity readings. See Figure 9 for representative model.
- o. Toluene solvent, or acceptable alternative.
- p. API Sand Content Test Kit. See API Recommended Practice 13B-1, Section 5.

### 8.2.2 Procedure—Solids Content

**8.2.2.1** Shake the wellbore solids sample, as received (see 8.2.1, Item l), until the sample is homogeneous.

**8.2.2.2** Weigh a small amount of the wellbore solids sample into a preweighed petri dish. Record the weight of the sample.

**8.2.2.3** Dry in oven regulated to 220°F (105°C) until no further loss in weight is observed.

**8.2.2.4** Cool in desiccator. Reweigh the petri dish, and record the weight of the dried sample.

### 8.2.3 Calculation—Solids Content

$$\text{Solids Content, \%} = \frac{100 (\text{Dried sample weight, g})}{\text{As received sample weight, g}} \quad (12)$$

### 8.2.4 Procedure—Calibration

**8.2.4.1** Make a stock solution (nonfiltered) using 1 gram of the wellbore solids sample (see 8.2.1, Item l) as dry solids. Fill the remainder volume with the completion brine sample (see 8.2.1, Item m) in the 1000-cubic centimeter volumetric flask.

**8.2.4.2** Using this stock solution and the completion brine sample from 8.2.1, Item m, make the following dilutions in the 100-cubic centimeter volumetric flasks. Make duplicates of each dilution.

**8.2.4.3** Calibrate the nephelometer according to manufacturer's standards. For most nephelometers, the sample size is 20 cubic centimeters.

**8.2.4.4** Measure turbidity (NTU) of each dilution sample and record.

**8.2.4.5** Weigh ten predried 1.2-micrometer filter disks and record weight of each disk ( $W_0$ ) in grams.

**8.2.4.6** Place one disk into the filter equipment.

**8.2.4.7** Pour 100 cubic centimeters of distilled water through the filter assembly to prewet the disk.

**8.2.4.8** Pour 100 cubic centimeters of dilution sample from 8.2.4.2 through disk at 20 cubic centimeters per time and record the volume ( $V_p$ ) in cubic centimeters when no more volume will pass through.

Note: If the sample contains high concentrations of solids (over 1000 milligrams per liter), it may be difficult to filter all of the 100 cubic centimeters. In such cases, pass as much volume as possible and note the volume ( $V_p$ ).

**8.2.4.9** Pour 500 cubic centimeters of distilled water through the disk to dissolve soluble salts.

Note: If hydrocarbons are present in the completion brine sample, rinse with 500 cubic centimeters filtered toluene, or acceptable alternate.

**8.2.4.10** Remove the disk from filter equipment and place in preweighed petri dish.

**8.2.4.11** Dry at 220°F (105°C) for at least 1 hour, place in desiccator for 30 minutes, and reweigh. Record the weight of the dried filter disk and sample ( $W_p$ ) in grams.



Figure 9—Representative Turbidimeter

## 8.2.5 Calculation—Calibration Curve

### 8.2.5.1

$$\text{Solids Concentration, mg/L} = \frac{(W_t - W_o) \times 10^6}{V_t} \quad (13)$$

Where:

$W_t$  = weight of filter disc and sample, g.

$W_o$  = weight of filter disc, g.

$V_t$  = volume of dilution sample through disc, cm<sup>3</sup>.

**8.2.5.2 Calibration curve:** Plot NTU readings from 8.2.4.4 against milligrams per liter. The completed graph is to be used to monitor changes in NTU value for a particular field application. Examples of graphs are shown in Figures 10 and 11.

## 8.3 FIELD TESTING

### 8.3.1 Equipment

**8.3.1.1 Graduated cylinder:** one 100-cubic centimeter.

**8.3.1.2 U.S. 200-mesh (74-micrometer) screen:** woven sieve or API Sand Content set.

Note: Screens must be in good condition, with no blocked openings or tears in screen cloth.

**8.3.1.3 Sample bottles:** six to eight 500-cubic centimeters and six to eight 100-cubic centimeters.

**8.3.1.4 Nephelometer,** see 8.2.1, Item n.

**8.3.1.5 Calibration curve** from 8.2.5.

### 8.3.2 Procedure—Sampling Points for Brine

**8.3.2.1 Mixing plant:** fully circulated sample from the low-pressure line (no surface sampling).

**8.3.2.2 Transport truck:** flow at exit valve for about one-half of the load volume, and then sample.

**8.3.2.3 Transport supply boat:** thief a sample before off-loading, or as in 8.3.2.2 above.

**8.3.2.4 Rigsite specific tank:** sample at low-pressure line, if available.

**8.3.2.5 Rigsite flow line.**

**8.3.2.6 Rigsite filtration sample.**

### 8.3.3 Procedure—Rigsite Turbidity Test

**8.3.3.1** Collect 100 cubic centimeters of representative sample at the locations of interest (see 8.3.2.1 through 8.3.2.6).

**8.3.3.2** Pour the 100 cubic centimeters through a 200-mesh (74-micrometer) screen into a 100-cubic centimeter graduated cylinder.

**8.3.3.3** Allow to stand for 2 to 5 minutes to allow solids to settle or float. If no large solids are present, this step is omitted.

Note: Filtering and allowing the sample to stand will give an indication of particle size and concentration of particles present.

**8.3.3.4** Remove the floating solids. Pour the remaining sample containing dissolved and suspended solids in a sample bottle, being careful not to transfer coarse solids that have settled.

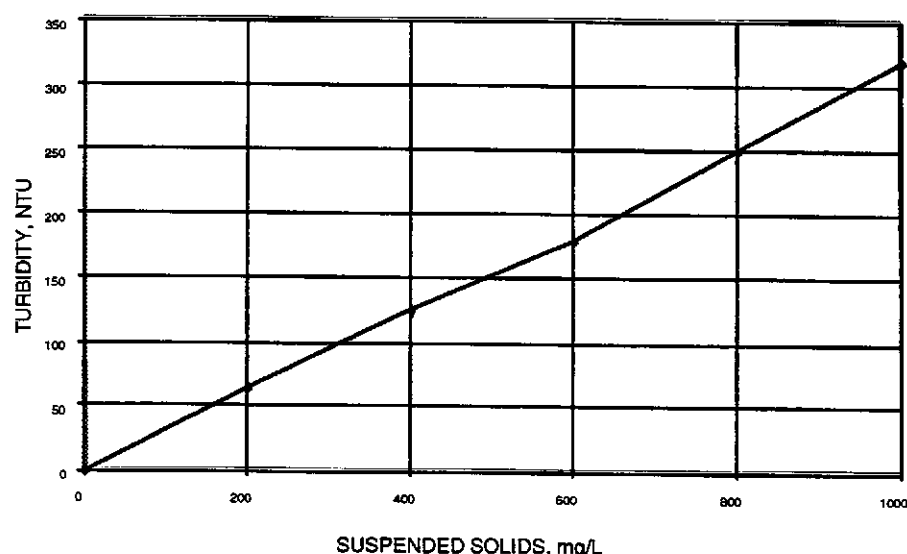


Figure 10—Example Calibration Curve  
(Range 0–1000 mg/L)  
EXAMPLE ONLY—DO NOT USE FOR CALIBRATION

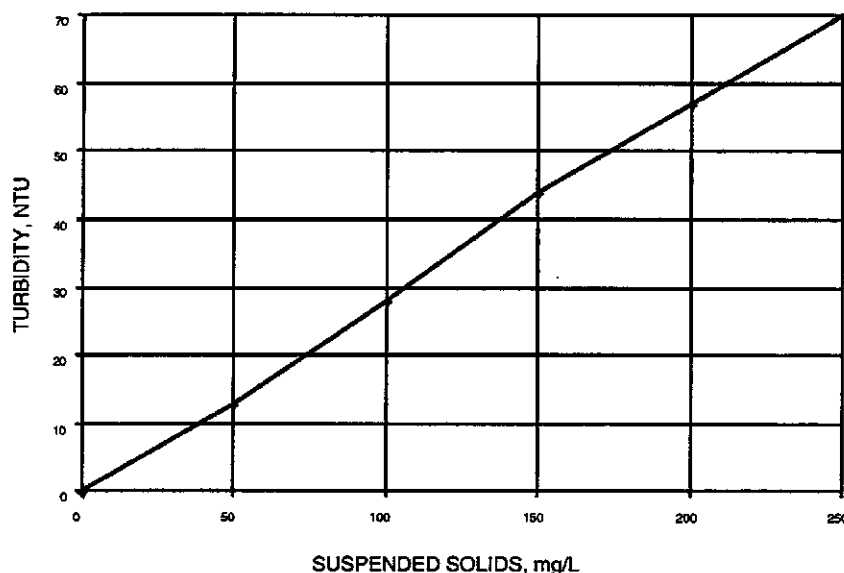


Figure 11—Example Calibration Curve  
(Range 0–2000 mg/L)

EXAMPLE ONLY—DO NOT USE FOR CALIBRATION

**8.3.3.5** Calibrate the nephelometer using manufacturer's standards. For most nephelometers, the sample size is 20 cubic centimeters.

**8.3.3.6** Measure the turbidity of collected samples (see 8.3.3.1). Record the NTUs for each sample.

#### 8.3.4 Calculation—Rigsite Turbidity Test

Compare the NTUs to milligrams per liter using the calibration curve from 8.2.5.2. In this way an estimate of solids content is achieved from turbidity measurements.

#### 8.3.5 On-Location Use of NTU Data

**8.3.5.1** The absolute NTU value of a fluid is specific to each fluid and can ONLY be used for comparison values to

the same fluid. An NTU reading of 30 is specific to an individual fluid and individual solids. For example, a 12.5 pounds per gallon fluid and a 17.2 pounds per gallon fluid may both have an NTU reading of 30, but the milligrams per liter of solids in each fluid may be vastly different. The absolute NTU reading is dependent on fluid color, solids distribution, air entrainment, and so forth.

**8.3.5.2** The field use of this procedure is recommended as a guide only and may be used for the determination of trends rather than absolute values. For example, during filtration on location, when the NTU values are no longer falling, no further gains in fluid quality will be made without making some change to the filtration process.

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