

Part No. Description

33055.....	Flashlight
52600.....	Calcium Carbonate Powder, 2 oz.
53344.....	Ethylene Glycol, 2 oz.
60016.....	Probe Holder
60105.....	Glass Beaker, 50 ml
E1053.....	Instructions for Brine Crystallization
L3855.....	Insulation
L3979.....	Tygon Tubing, 1/4 X 1/16
L8865.....	Print Paper for Thermometer, (5 Rolls/PKG)
N5257.....	Magnetic Stirring Bar, 3/8 X 1 in.
N5282.....	Magnetic Cold Plate Stirrer, 115V
N8203.....	General Purpose Probe for Thermometer
N8231.....	Electronic Printing Thermometer

INTRODUCTION

This procedure describes a field and laboratory method for measurement of crystallization temperature of high-density oil field brines used in completion, workover and drilling operations.

A detailed discussion of brine crystallization and the relationships of the measured parameters is found in section 2 of API publication RP 13J, Recommended Practice for Testing Heavy Brines (June 1, 1986). API defines the values for crystallization temperature as follows:

FCTA (First Crystal to Appear): During the cooling cycle, the temperature at which visible crystals start to form. FCTA will generally include some supercooling effect (cooling below actual crystallization temperature).

TCT (True Crystallization Temperature): During the cooling cycle, the maximum temperature reached following the supercooling minimum, or the inflection point in cases with no supercooling.

LCTD (Last Crystal to Dissolve): In the heating cycle, the temperature at which crystals disappear, or the inflection point on the heating curve.

PROCEDURE

1. Equipment Setup

Using a suitable length of Tygon tubing, connect either one of the two tubing nipples on the back of the cold plate to a tap water source. Connect another length of tubing to the other nipple and place the free tubing end in a sink drain. Turn on the water and maintain a minimum flow of about 1 liter (1 quart) per minute.

Fann Instrument Company
P. O. Box 4350
Houston, Texas, U.S.A. 77210
Telephone: (281) 871-4482
Toll Free: (800)347-0450
Fax: (281) 871-4358

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Place about 25 ml of test sample and a pinch of nucleating agent in the beaker. Put the magnetic stir bar in the beaker. Put 3 or 4 drops of ethylene glycol on the center of the aluminum cooling plate and put the beaker on the plate. The ethylene glycol will improve the heat transfer from the beaker to the cold plate. Place the insulating jacket around the beaker.

Use some suitable means to secure the temperature probe so that at least ¼ inch of the probe tip is immersed in the test fluid. Place the temperature/printing unit so that the test sample and the unit can both be conveniently viewed. Position the high intensity light next to the cold plate and direct the light into the beaker to illuminate the test fluid. Flip the toggle switch on the cold plate to turn the unit on. Set the temperature control knob to 0%. Set the stirrer control between 30 and 50%, sufficient to create a vortex above, but not extending down to, the stir bar.

CAUTION

WHEN USED ON THE COOL POSITION, THE COLD PLATE WILL REACH A TEMPERATURE APPROXIMATELY 40°C BELOW THAT OF THE TAP WATER, e.g. 10°C TAP WATER GENERATES A -30°C COLD PLATE.

BEWARE OF FROSTBITE

2. Printing Thermometer Setup

Procedure for loading paper, batteries, printer testing, setting the clock and calendar, and the functions of the various keys are found in the operating instructions included with Electronic Thermometer.

To turn the thermometer on, press the power switch on the left side to the unit. The unit powers up in the centigrade format; press the centigrade/fahrenheit select key to read in fahrenheit. The temperature being displayed can be printed manually or at one of 5 preselected intervals. The intervals are 3 seconds, 30 seconds, 1 minute, 30 minutes, and 60 minutes. To select the desired printing interval, press the SET key. The selected interval will be displayed next to the word SET in the lower left of the display.

Each time the SET key is pressed the next interval is selected until you return, after seven presses, to the non-printing mode.

For this procedure, select either the 30 second or 1 minute interval. The 1 minute interval is preferred to conserve paper. To start printing at the selected interval, press the GO key. When the GO key is pressed, the word SET disappears and the symbol P appears. The thermometer will print the time and temperature at the selected interval until the STOP key is pressed. The unit can be made to print the displayed temperature at any time by pressing the PRT key. If the PRT key is pressed while the unit is running in a selected printing mode, an asterisk (*) will appear at the beginning of the printout line.

MEASUREMENT OF CRYSTALLIZATION POINT

Press the GO key to start printing the temperature at the selected interval. Set the cold plate temperature control to 100%. Observe the test sample as it is cooled. When crystals first appear in the sample (FCTA), press the PRT key to record the temperature. Watch the temperature readout on the thermometer. In most cases, the temperature will quickly rise to some maximum level (due to heat of crystallization) before resuming the downward trend. Continue pressing the PRT key every 2 to 3 seconds during the temperature rise to capture and record the maximum temperature attained. The maximum temperature recorded will be the TCT. When the temperature begins to decrease again after reaching the maximum, allow the temperature to fall 2 or 3 degrees while the thermometer records the temperature at the selected interval. After the temperature has fallen a few degrees, set the cold plate temperature control back to 0% and press the FEED key on the thermometer. This serves to place a blank space on the printout paper to separate the cooling from the warming cycle. Continue observing the crystallized sample as it is warmed. As the sample is warmed, the crystals will begin going back into solution. At the moment when the last crystal appears to dissolve, press the PRT key.

This temperature is the LCTD. Press the STOP key to exit the interval printing mode.

The above procedure should be repeated until consistent, repeatable ($\pm 2^\circ\text{F}$) results are obtained for TCT. This usually requires 3 or more test cycles. The temperature control settings suggested in the above procedure produce the maximum cooling and warming rates.

After the initial test cycle where a preliminary TCT has been determined, the cooling rate can be decreased and supercooling can be minimized by setting the temperature control to achieve a temperature 5 to 10 degrees below the expected TCT. Between test cycles, allow the sample to warm to a minimum of 10 and a maximum of 15 degrees above the LCTD before beginning another cooling cycle.

For more information, please contact us:

[ExpotechUSA](#)
[10700 Rockley Road](#)
[Houston, Texas 77099](#)
[USA](#)

[281-496-0900 \[voice\]](#)

[281-496-0400 \[fax\]](#)

E-mail: sales@expotechusa.com

Website: www.ExpotechUSA.com