

Brine Crystallization Kit Instruction Manual



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Instrument No. 208720

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Fann Instrument Company

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1 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 API RP 13J, Recommended Practice for Testing Heavy Brines. 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.

2 Safety

Safe laboratory practices and procedures should be observed while operating the Brine Crystallization Kit.

When using propylene glycol, review the MSDS for safe handling, storage, and disposal information.

3 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.

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 propylene glycol on the center of the aluminum cooling plate and put the beaker on the plate. The propylene 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.



Beware of frostbite. When the cold plate is used on the cool position, its temperature will reach approximately 40°C below that of the tap water (e.g., tap water generates a -30°C cold plate.)

4 Printing Thermometer Setup

The 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 the electronic thermometer.

Become familiar with the keys that perform the following functions:

- Start or Go
- Print
- Advance paper
- Stop or Exit

5 Crystallization Point Measurement



Identify the keys that you will use for operating the printing thermometer. See Section 4 of this manual.

1. Press the appropriate key to start printing the temperature at the selected interval.
2. Set the cold plate temperature control to 100%.
3. Observe the test sample as it is cooled.
4. When crystals first appear in the sample (FCTA), press the print 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.
5. Continue pressing the print 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.
6. 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.
7. After the temperature has fallen a few degrees, set the cold plate temperature control back to 0%.
8. Then advance the paper in the printing thermometer to leave a blank space on the printout paper to separate the cooling from the warming cycle.
9. Continue observing the crystallized sample as it is warmed. As the sample is warmed, the crystals will begin going back into solution.
10. At the moment when the last crystal appears to dissolve, press the print key. This temperature is the LCTD.
11. 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 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.

6 Parts List

Table 6-1 Brine Crystallization Kit, PN 208720

Part No.	Description
204232	Instruction Manual
204795	Insulation
205623	Tygon® Tubing, 1/4 x 1/16
205807	Print Paper for Thermometer (5 rolls/pkg)
206000	Magnetic Stirring Bar, 3/8 X 1 in.
206014	Magnetic Cold Plate Stirrer, 115V
206045	General Purpose Probe for Thermometer
206048	Electronic Printing Thermometer
208431	Flashlight
208721	Probe Holder
209940	Calcium Carbonate Powder, 2 oz
210011	Glass Beaker, 50 ml
102142310	Propylene Glycol

7 Warranty and Returns

7.1 Warranty

Fann Instrument Company warrants its products to be free from defects in material and workmanship for a period of 12 months from the time of shipment. If repair or adjustment is necessary, and has not been the result of abuse or misuse within the twelve-month period, please return, freight prepaid, and correction of the defect will be made without charge.

Out of warranty products will be repaired for a nominal charge.

Please refer to the accompanying warranty statement enclosed with the product.

7.2 Returns

For your protection, items being returned must be carefully packed to prevent damage in shipment and insured against possible damage or loss. Fann will not be responsible for damage resulting from careless or insufficient packing.

Before returning items for any reason, authorization must be obtained from Fann Instrument Company. When applying for authorization, please include information regarding the reason the items are to be returned.

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