FREEZING PROCEDURE, THE KEY FOR A SUCCESSFUL FORMATION EVALUATION PROGRAM - HOW TO ENSURE CORE SAMPLES REMAIN FROZEN DURING THE ENTIRE PROCESSING AND TRANSPORTATION STAGE –

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ABSTRACT

Freezing is a preservation technique recommended for unconsolidated formations and is used to maintain core integrity during handling. Remote areas can often create challenges for successfully completing freezing operations. Appropriate design and equipment solutions together with a full operation plan are key factors in assuring that all eventualities are covered and the core is given the required freezing conditions. Inappropriate planning and equipment design will render the whole process obsolete and increases the potential for irreversible core damage. Lack of awareness in maintaining a constant core temperature is one of the most common mistakes made throughout the industry and the resulting change in rock properties brings into question the accuracy of core analysis data further down the chain.

This case study aims to highlight the various stages and precautions taken during the core handling process and provides details showing the full audit trail of the rock samples during operation. Extensive testing has resulted in the creation of specialized equipment and procedures using specialized corrugated freezing containers equipped with internal and external thermal sensors. The tests have been modelled to allow use in areas typically characterized by harsh environmental conditions in both their weather patterns and remoteness pushing freezing capability beyond the normal range of conditions. The purpose of this study is to increase the confidence of oil operators in using this new freezing technique as a reliable and comprehensive method in preserving quality core and by making transparent the whole freezing process from core acquisition at well site to arrival at the laboratory.

INTRODUCTION

From literature relating to freezing techniques over 10 years ago, freezing was seen as a controversial method [1] or that the effects remained unknown [2]. However, more recent studies portray freezing as a positive procedure in core stabilisation and preservation under the right circumstances. Freezing appears more under control and is becoming more widely recommended [3]. Over the last 12 months, numerous coring operations worldwide involving core freezing have been recorded (GOM, Offshore Brazil, Brunei, India and the UK/North Sea). Most operators using the freezing technique recognise that the method satisfies the basic requirements for effective core stabilisation as listed that core freezing:

1. Is safe to apply at the wellsite
2. Prevents fluid loss and core drying during the (short) period of time the core is set aside for analysis
3. Maintains the mechanical structure of the core
4. Protects the core during transportation to the laboratory
5. Provides easy handling at the laboratory: i.e., frozen core sections allow gamma-ray logging, CT scanning and sampling (slabbing, plugging)
6. Is comparative in the level of core protection provided and can be associated with other stabilization media (Foam, Lithotarge)

The Areas of Concerns

This study does not aim to discuss the side effects of freezing and the below summarizes some of the concerns raised by the industry as highlighted in some other technical papers.

- Freezing alters internal grain structure affecting both permeability and porosity in an unpredictable way [2]
- The effect of freezing is a function of brine saturation and salinity [4]
- Freezing core is inefficient in gas reservoirs where Sw is low
- Rapid freezing leads to high temperature differentials between the surface and the interior of samples, creating the buildup of pressure within these samples and, ultimately, fracturing [5]
- Dry ice involves high CO_2 concentration in air and may cause inhaling hazard.

EQUIPMENT AND TESTS

Equipment Specifications:

Full Length Freezing Equipment (Figure 1):

- 6 or 9m long, made of steel
- Up to 5 compartments
- Use of polyester foam on the external side as insulator to prevent cold burn on the outside of the basket
- Polyester lid or heavy duty tarpaulin to cover the basket during the freezing process

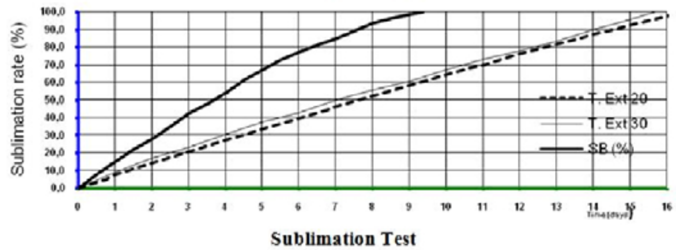
Figure 1

Shipping Container Specifications (Figure 2):

- Specification: weight: 64kg
- Internal dimensions: 1050x600x700mm
- Monitoring of external and internal temperature from +50 to -85° Celsius.
- Max loading capacity for 10 days trip without reload:
- Holds 9m of 5 ¼" core or 12m of 4" core

Figure 2

Probe Specifications:

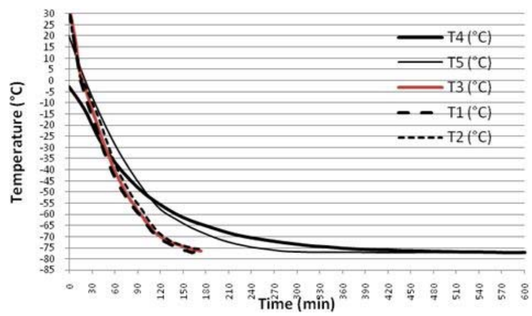


Type T (Copper–Constantan) thermocouples are suited for measurements with a range from −200 to +350 °C. They are often used as a differential measurement since only copper wire touches the probes. Both conductors are non-magnetic. Type T thermocouples have a sensitivity of about 43 µV/°C.

Configurations of Freezing Coring Operations

Test# / Country	In the Below Graph	Avg Day Temp. °C	Avg Night Temp. °C	Type of Dry ice	Volume Used
#1 / Brazil	T EXT 30°C	32 °C	25 °C	10 kg blocks	250kg
#2 / UK	T EXT 20°C	18 °C	10 °C	10 kg blocks	250kg
#3 / UK	SB%	18 °C	10 °C	6mm pellets	250kg

Test 1: 1m core within a fiberglass inner tube placed inside a compartment of the freezing basket. Dry ice pellets of 1.6mm and 6mm used to freeze the fully immersed sample within the pellets. Use of dry ice blocks not in contact with the sample to avoid fast thermal shock.



Description: The tube was fully immersed in 1.6mm pellets in order to freeze. Probes were placed at the center of the core at regular interval to measure the freezing cycle. 160 min has been required to freeze the sample to -75°C. Core was made-up with building sand only.

Interpretation: The freezing curve is constant.
The three probes placed along the center of the core show very similar temperature trends indicating uniform core freezing. The small size pellets of 1.6mm maximize surface contact with the liner guaranteeing uniform and the most rapid freezing.

On a parallel test we observed that the utilization of bigger pellets (6mm pellets) marginally decreased the freezing process due to a lower surface contact of dry ice with the tube. Using dry ice blocks with a lower surface contact with the liner in an attempt to avoid freezing certain areas faster than others, proved to be inefficient.

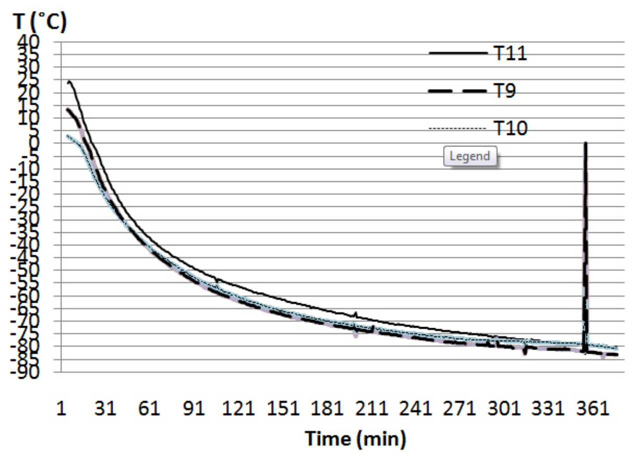
- T1 to T3: 1.6mm pellets: 180 min to freezing point
- T4: Dry Ice Block not in contact: over 400min to freezing point
- T5: 6mm pellets: 240 min to freezing point

Test 2: 1m of core placed in a freezing basket. 6mm pellets used and surrounding the sample. Test conducted with an aluminum and fiber glass inner tube with an external temperature of ~20°C.

Interpretation: Cores within aluminum liners freeze faster than cores within fiberglass liners due to the insulating nature of the tube.

- Aluminum Liner: 180 min to the freezing point
- Fiberglass Liner: 210 min to the freezing point

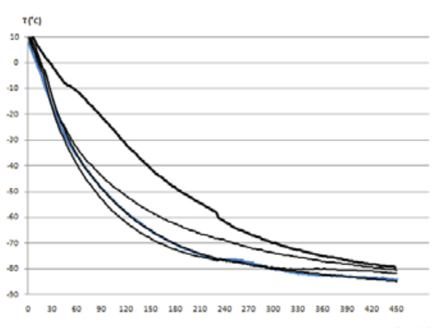
Test 3: Freezing full length core (aluminum inner tube of 6mm) in a basket. 6mm pellets used during the process and in direct contact with the tube. External temperature of 30° Celsius.



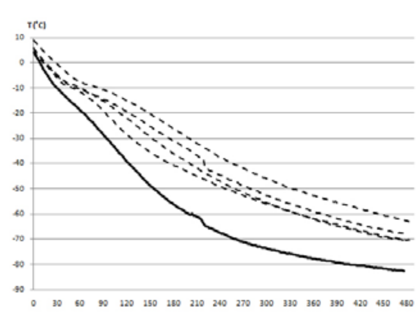
Description: T9, T10 and T11 are temperatures of the core centre. They have been placed at 1, 3 and 5m along the 6m aluminum liner. The core was unconsolidated sand saturated with oil.
The freezing point was reached in 280min.

Interpretation: In comparison with Test#2 where the outside temperature differs from 20°C to 30°C, we can observe a longer freezing time also, in part, due to the oily nature of the core.

Test 4: Full length freezing of 5 ¼" core. Core 1 and core 2 composed mainly of oily unconsolidated sand and shale. Use of 6mm pellets. Outside temperature of 10 degrees C.



Interpretation: Top of core#1 composed mainly of unconsolidated loose oily sand. Freezing point was achieved in 240 min. The last meter of core#1 plus the first meter of core#2, a mix of oily sand and shale, froze 330 min. The remainder of core#2, composed mainly of shale, required over 500 min to freeze. These examples show how the physical properties of the core (water/oil saturation, por., perm.) affect the time taken to freeze core. It is therefore useful to increase the interval of monitoring during freezing (recommended at 1 probe every meter) in order to monitor effectively and optimize efficiently, the freezing process.



RECOMMENDED OPERATION

Job Planning	**Call out**
1. Dry ice supplier selection with a lead time on manufacture to base following a pre-order notice. 2. Operational logistics in: a) Knowing the schedule of supply boats to rig b) Knowing the time required to reach the rig c) Being in close contact with rig logistics for the last minute call out of dry ice. d) Anticipating a need for last minute dry ice supplies in case of a drilling delay or extension in the coring program. 3. Coring Plan in knowing the depth of core point, trip out speed program, interval of coring.	1. Equipment mobilization: a) Certified Freezing cargo basket with probe and data logger b) Multipoint spreader bar with lifting sling c) Core lay down cradle d) Saw unit and spare parts e) Appropriate shipping container f) Thermal protective equipment 2. Quantity required: a) 50kg of pellets/m is required for full length freezing b) 250kg of blocks are required to guarantee 12 days shipping without refilling
Freezing process	
1. Full length freezing: a) Lay down the tube onto the cradle and, using a multi-point spreader bar, transfer the tube into one compartment of the freezing cargo basket keeping the sling on. b) Drill at regular intervals (every meter), holes into the center of the core using a drill stop to set the drill to half the core diameter c) Prior to transfer of the tube, add a layer of pellets to the bottom, then another layer on top of the tube to fully surround it. d) Cover the compartment with a lid or heavy tarpaulin to avoid fast melting due to adverse conditions as well as to confine the frost. e) Start monitoring the freezing using the multi-channel data logger. Variable freezing rates across an interval signifies variable formation within the tube. It is important, when taking into account coring parameters, to know when to stop the freezing. . The average freezing for 4” core is 280min whereas 5 ¼” core can take up to 360min	2. Further core processing: a) Reconnect the sling to the spreader bar and move the tube back to the core lay down cradle b) Using an air power driven saw, cut the core into meter sections. c) Return each section cut into the freezing basket in the remaining dry ice pellets. This avoids yet opening the freezing container containing the dry ice blocks. 3. Loading the shipping container: a) Create a layer of blocks at the bottom, then position each cut section carefully using thermal gloves. b) Add another layer of dry ice blocks across the top of the tubes. **c)** If the container is equipped with internal probes, start the recording.

CONCLUSIONS AND RECOMMENDATIONS

Freezing will be considered as an alternative to all other preservation/stabilization media when the core recovered is unconsolidated with no presence of annulus. Freezing is probably the most expensive technique; therefore optimum preparation is required in ensuring savings are maximized. As a result, applying this technique to wellsite operations requires the design of a rigorous and thorough core acquisition program taking into account all the logistics involved in freezing operations. Contrary to all other methods of stabilization and core processing, freezing involves a commitment of all parties from the operator initiating the order, the service provider in running the service, through to the laboratory in being prepared to receive and handle geological samples in frozen state.

Findings

- Full length freezing using the smallest type of available pellets is recommended. It optimizes core liner encapsulation and ensures the most uniform and efficient freezing.
- Pellets in general sublimate faster than dry ice block by 40 to 50%. Accurate planning/calculation are required to ensure safe arrival of the ice, on time without incurring inefficient losses.
- Aluminum liner freezes faster than fiber glass tube by 15%. This raises a potential requirement to investigate the freezing time against various rock types and in doing so the most appropriate tube may be selected prior to coring. A quick assessment of the geologic content will aid in ensuring the core is only left under freezing conditions for the time required to freeze the sections that require freezing. For example, if it takes 3 hours to effectively freeze oily sands and a 10 meter section is composed of 1m sand and 9 m shales, freezing only need continue up until the point that the sand is frozen, not the shale.
- Metal to reinforce the shipping container has an effect on the overall performance and increases the sublimation rate, especially under high external temperatures, by 20%.

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