



TECHNOLOGY FACILITY FOR RESERVOIR DEVELOPMENT AND OPTIMISATION

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ISO 13503-2 SUITE
BASELINE FRACTURE CONDUCTIVITY &
PERMEABILITY
16/30 CERAMIC PROPPANT
SUPPLIED BY TKZ PROPPANTS

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SUMMARY

A sample of 16/30 proppant was provided for testing by TKZ Proppants.

Testing was carried out in accordance with the ISO 13503-2 suite of tests, covering crush resistance, acid solubility, bulk density, specific gravity, sphericity and roundness, turbidity and sieve analysis.

The baseline conductivity and permeability tests of the sample were performed with a loading of 2lb/ft², at closure stresses of 2, 4, 6, 8 and 10kpsi, at 250°F and each maintained for 50 hours between Ohio sandstone.

A summary of results obtained from the ISO 13503-2 suite of tests is tabulated overleaf. Long term baseline conductivity results are presented in the main report.

Photomicrographs are given in Appendix A.

| Parameters | | 16/30 |
|-----------------------------------|-------|---------------|
| Crush Test (psi) | 7500 | 6.3 ± 0.0% |
| | 10000 | 9.2 ± 0.6% |
| Sieve Analysis (90% 16/30) | | 99.8 ± 0.0 |
| Sieve Analysis (0.1% -40) | | 0.0 ± 0.0 |
| Sieve Analysis (1% +12) | | 0.0 ± 0.0 |
| Mean Size (mm) | | 0.808 ± 0.000 |
| Turbidity (FTU) | | 40 ± 1 |
| Sphericity | | 0.9 ± 0.1 |
| Roundness | | 0.8 ± 0.1 |
| Bulk Density (g/cm ³) | | 1.77 ± 0.00 |
| Specific Gravity | | 3.11 ± 0.01 |
| Acid Solubility (%) | | 4.6 ± 0.0 |

Table i: Summary of ISO 13503-2 Test Suite on Proppant Sample

1. EXPERIMENTAL PROCEDURES

1.1 Specific Gravity

A dry density bottle, with stopper, is weighed (W_1). Approximately 30g of proppant is added and the bottle re-weighed (W_2). Mineral oil is added to cover the proppant and fill the bottle 3/4 full. The bottle is vacuumed to an absolute pressure of less than 30 mbar for approximately thirty minutes.

More oil is added to fill the bottle. The stopper is then placed in the neck. Excess oil is wiped off, being careful not to suck oil through the capillary in the stopper. The full bottle is weighed (W_3). The proppant and oil are disposed of and the bottle refilled with mineral oil only. The stopper is placed in the neck and the bottle dried and weighed again (W_4). This procedure is repeated.

The Specific Gravity is then given by,

$$(W_2 - W_1) / ((W_4 - W_3) + (W_2 - W_1))$$

1.2 ISO 13503-2 Methods

The following tests were performed in exact accordance with the ISO 13503-2 procedures. The relevant sections are as follows:

| Section | Test/Procedure |
|----------------|--|
| 5.2 | Sample Splitting |
| 6 | Sieve Analysis |
| 7 | Sphericity and Roundness |
| 8 | Acid Solubility (tests run using NH_4HF_2 as source) |
| 9 | Turbidity |
| 10.3 | Bulk Density |
| 11 | Crush Resistance (with pluviator)* |

Table 1: ISO Procedures

*Crush testing was carried out at pressures of 7500 and 10000psi.

1.3 Baseline Fracture Conductivity & Permeability

The Fracture Conductivity Cells allow for samples of proppant of various loading to be subjected to closure stress and temperature over extended time. Fluids are flowed through the pack and from differential pressure measurements the flow capacity of the pack can be determined. A schematic of the experimental set-up is given below. The cell is essentially a modified 10 square inch API conductivity cell.

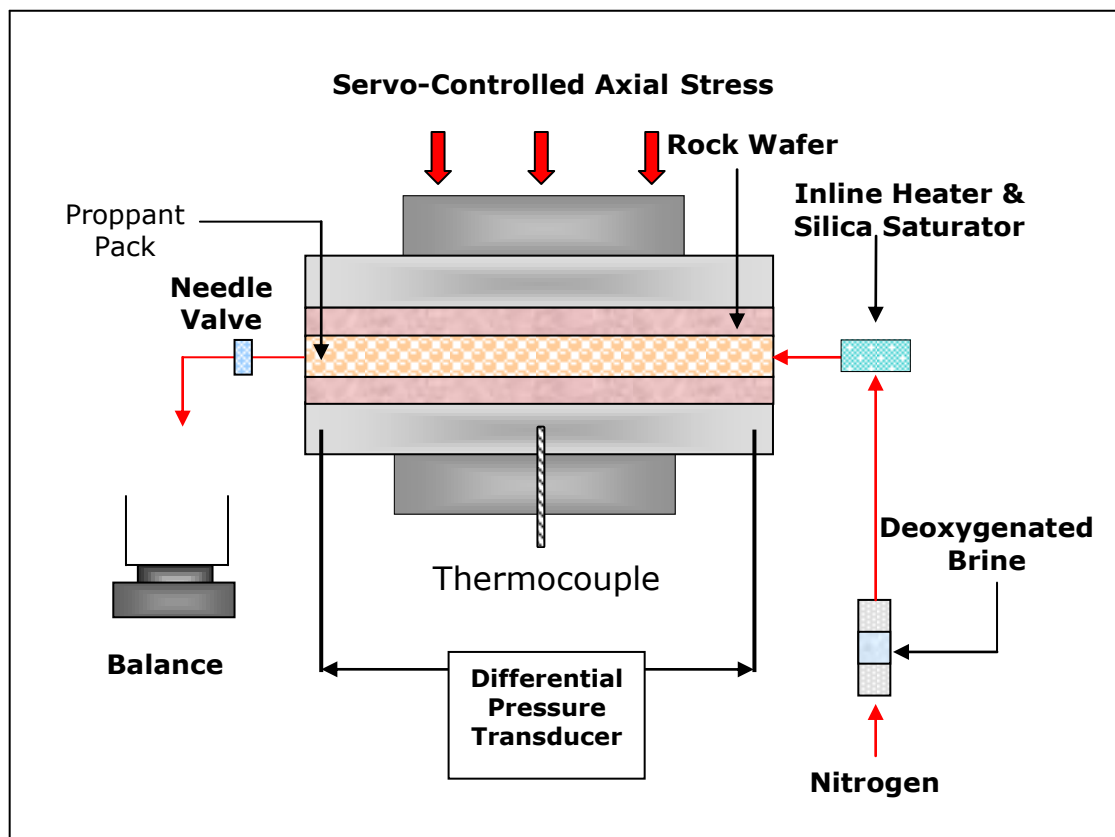


Figure 2: Configuration of Conductivity Cell

The test procedure is as follows:

1. Outcrop rocks are selected. For these tests, Ohio sandstone was used. Ohio sandstone has a static elastic modulus of approximately 4 million-psi and a permeability of 0.1mD. Wafers of thickness 9.5mm are machined to 0.05mm precision and one rock is placed in the cell.
2. The selected proppant is sample split and weighed out. Sample splitting ensures that a representative sample is achieved in terms of its particle size distribution.
3. In cell 1, the proppant is placed into the cell and levelled. The top rock is then inserted.
4. Heated steel platens provide the correct temperature simulation for the test. A thermocouple inserted in the middle port of the cell wall records the temperature of the pack. A servo-controlled loading ram provides the closure stress.
5. The cells were initially set at 80°F and 1000psi. The cells were then heated to 250°F and held for 15 hours at 1000psi before being ramped to 2000psi over 10 minutes. After 50 hours a set of measurements was made before the stress was ramped to 4000psi (total time: 115 hours).
6. Further measurements were made at 6000, 8000 and 10000psi, corresponding to a total time of 265 hours.

Conductivity measurements were made using the following procedure:

- i. A 70mbar full range differential pressure transducer is activated by closing the bypass valve and opening the low pressure line valve (the second valve is there to prevent fluid flow bypassing the cell itself while the d.p. bypass is open).
- ii. Flow is generated and maintained by a piston pump located downstream of the cell. The pump can control flow rates down to $10^{-5}\text{cm}^3/\text{min}$ with a resolution of 2mm^3 . Fluid is flowed at rates as specified by the ISO procedure. When the differential pressure appears to be stable, data is logged every second for a 3 minute interval. The output from the differential pressure transducer is fed to a data logger 5 digit resolution multi-meter.
- iii. The mean value of the differential pressure is retrieved from the multi-meter together with the peak high and low values. If the difference between the high and low values is greater than the 5% of the mean, the data is disregarded.
- iv. Temperature is recorded from the inline thermocouple at the start and end of the flow test period. If the temperature variation is greater than 0.5 degrees K the test is disregarded. Viscosity of the fluid is obtained from using the measured temperature and viscosity tables. For brine at 100psi, no pressure correction is made. The density of brine at elevated temperature is obtained from these tables.
- v. At least three permeability determinations are made at each stage. The standard deviation of the determined permeabilities should be less than 1% of the mean value for the test sequence to be considered acceptable.
- vi. At the end of the permeability testing, the widths of each of the four corners of the cell are determined using vernier callipers, to 0.01mm resolution.

2. RESULTS

2.1 Sieve Analysis

The result of the sieve analysis is tabulated below:

| ASTM Mesh | 16/30 | | | |
|-------------------|-----------------------------|-----------------------------|------------------------|-----------------------|
| | % Proppant (A) by weight | % Proppant (B) by weight | % Average by weight | Standard Deviation |
| +12 | 0.00 | 0.00 | 0.0 | 0.0 |
| -12 +16 | 0.00 | 0.00 | 0.0 | 0.0 |
| -16 +18 | 2.14 | 2.22 | 2.2 | 0.1 |
| -18 +20 | 26.58 | 26.84 | 26.7 | 0.2 |
| -20 +25 | 57.87 | 57.50 | 57.7 | 0.3 |
| -25 +30 | 13.22 | 13.23 | 13.2 | 0.0 |
| -30 +40 | 0.19 | 0.21 | 0.2 | 0.0 |
| -40 | 0.00 | 0.00 | 0.0 | 0.0 |
| Total | 100.0 | 100.0 | 100.0 | |
| %16/30 | 99.8 | 99.8 | 99.8 | 0.0 |
| Mean Size (mm) | 0.808 | 0.809 | 0.808 | 0.000 |

Table 2: Sieve Analysis

2.2 Bulk Density & Specific Gravity

The results of the bulk density & specific gravity are tabulated as follows:

| Sample No. | Bulk Density (g/cm ³) | Standard Deviation (g/cm ³) | Specific Gravity | Standard Deviation |
|------------|-----------------------------------|---|------------------|--------------------|
| 16/30 | 1.77 | 0.00 | 3.11 | 0.01 |

Table 3: Bulk Density & Specific Gravity: Results

2.3 Acid Solubility

The results of the Acid Solubility are tabulated below:

| Sample No. | Test No. | % Loss, by Mass | Average % Loss, by Mass | Standard Deviation % Loss, by Mass |
|------------|----------|-----------------|-------------------------|------------------------------------|
| 16/30 | 1 | 4.592 | 4.6 | 0.0 |
| | 2 | 4.591 | | |

Table 4: Acid Solubility Results

2.4 Sphericity & Roundness

| 16/30 | | |
|-------------------------|-------------------------------------|-------------------------------------|
| Sample Reference | Sphericity | Roundness |
| 1 | 0.9 | 0.8 |
| 2 | 0.7 | 0.8 |
| 3 | 0.9 | 0.8 |
| 4 | 0.9 | 0.8 |
| 5 | 0.7 | 0.8 |
| 6 | 0.9 | 0.9 |
| 7 | 0.9 | 0.9 |
| 8 | 0.9 | 0.8 |
| 9 | 0.7 | 0.8 |
| 10 | 0.9 | 0.9 |
| 11 | 0.9 | 0.8 |
| 12 | 0.9 | 0.8 |
| 13 | 0.9 | 0.8 |
| 14 | 0.9 | 0.8 |
| 15 | 0.9 | 0.8 |
| 16 | 0.9 | 0.8 |
| 17 | 0.7 | 0.8 |
| 18 | 0.9 | 0.9 |
| 19 | 0.9 | 0.8 |
| 20 | 0.9 | 0.8 |
| | Mean = 0.9 | Mean = 0.8 |
| | Standard Deviation = 0.1 | Standard Deviation = 0.1 |

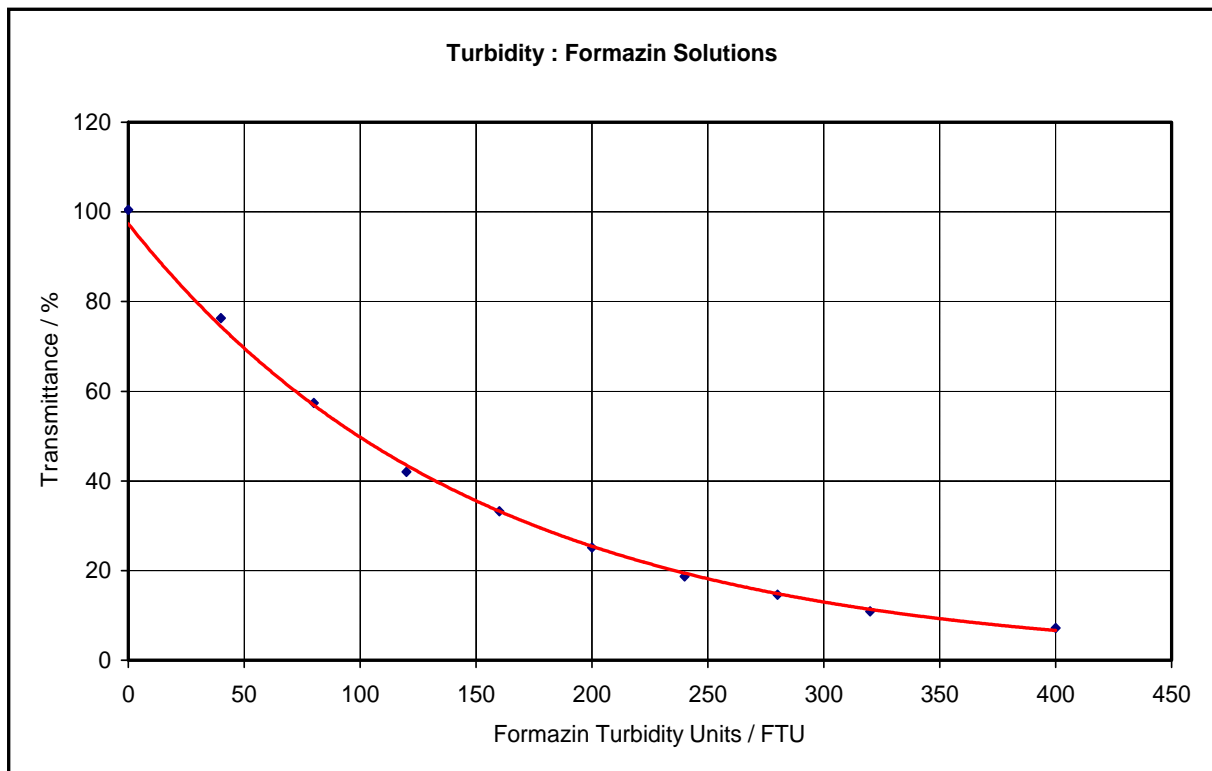
Table 5: Sphericity & Roundness

2.5 Turbidity

The following table and graph show the spectrophotometer calibration.

| Solution/FTU | Transmittance/% |
|--------------|-----------------|
| 0 | 100.5 |
| 40 | 76.3 |
| 80 | 57.4 |
| 120 | 42.0 |
| 160 | 33.2 |
| 200 | 25.1 |
| 240 | 18.7 |
| 280 | 14.6 |
| 320 | 10.9 |
| 400 | 7.2 |

Table 6: Turbidity Calibration



| Sample No. | Measured Sample Transmittance (%) | Turbidity (FTU) | Mean Turbidity (FTU) | Standard Deviation (FTU) |
|------------|-----------------------------------|-----------------|----------------------|--------------------------|
| 16/30 | 75.9 | 41 | 40 | 1 |
| | 76.6 | 39.5 | | |

Table 7: Turbidity Results

2.6 Crush Resistance

The results of the Crush Resistance tests are tabulated below:

| Stress (psi) | 16/30 | | |
|--------------|------------------|--------------------------|---------------------------------------|
| | % fines, by mass | Average % fines, by mass | Standard Deviation (% fines, by mass) |
| 7500 | 6.33 | 6.3 | 0.0 |
| | 6.33 | | |
| 10000 | 8.82 | 9.2 | 0.6 |
| | 9.67 | | |

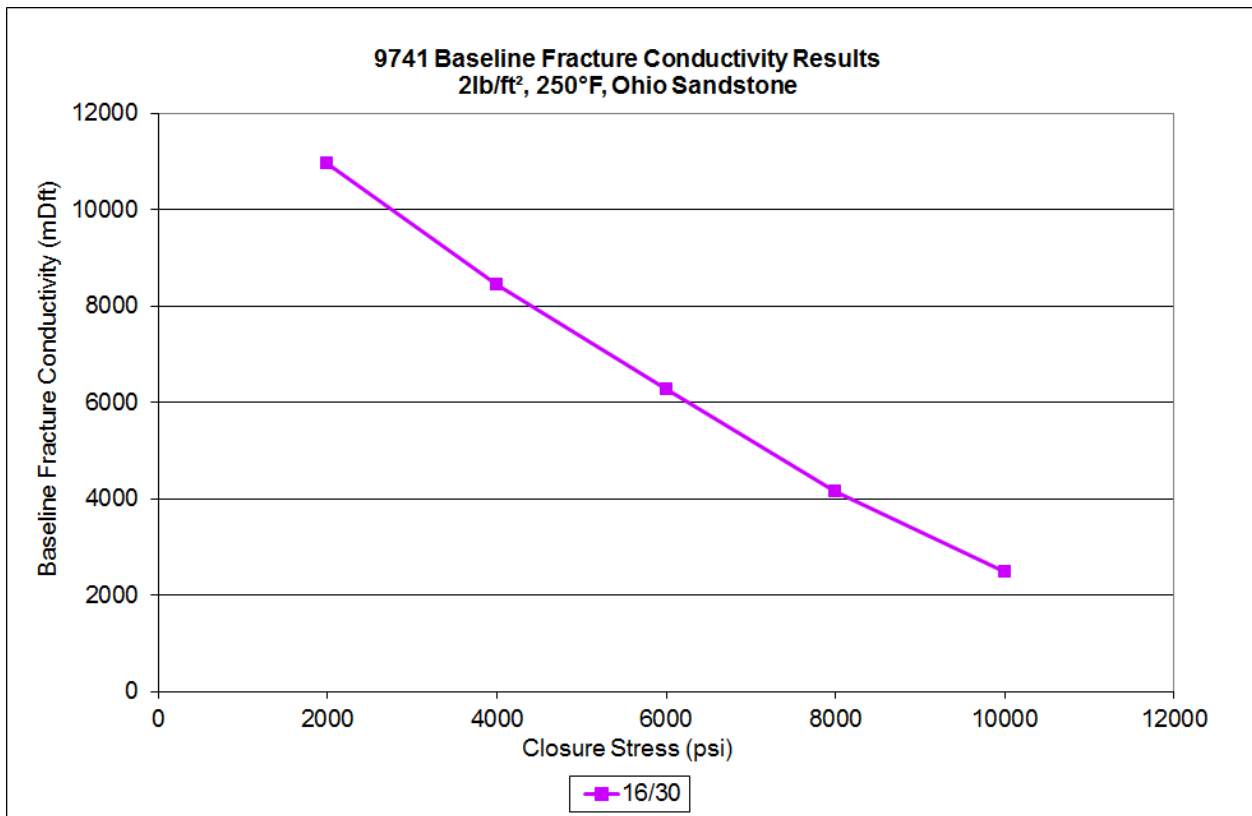
Table 8: Crush Resistance Results

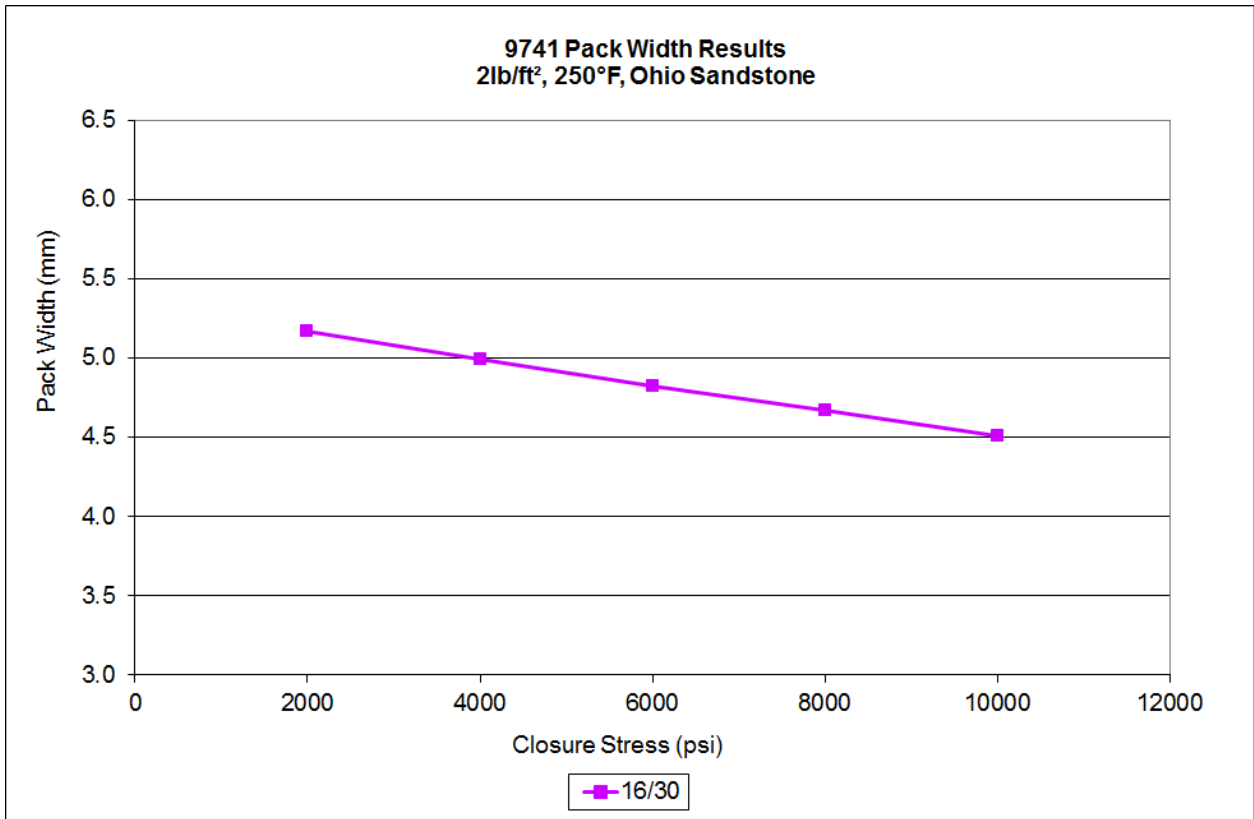
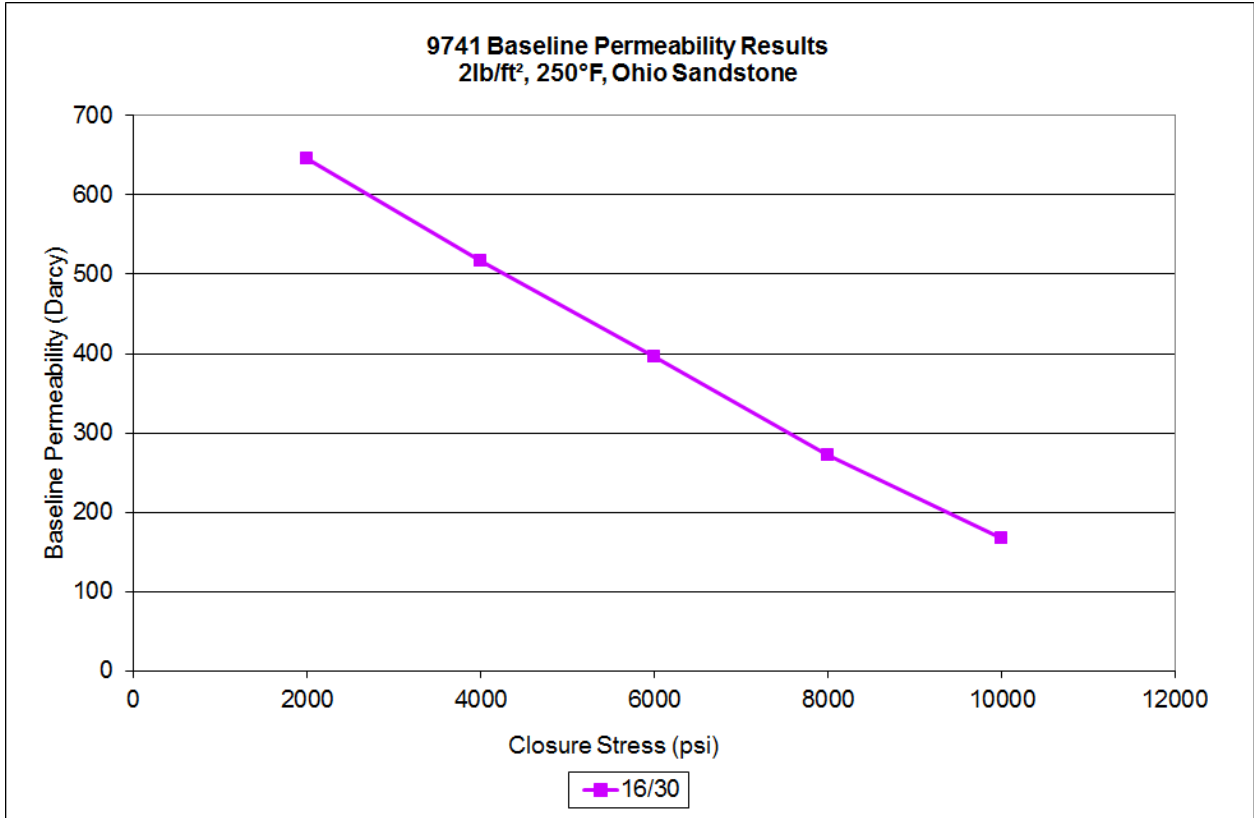
2.7 Baseline Fracture Conductivity & Permeability

The results of the baseline fracture conductivity; permeability and fracture widths are tabulated and plotted below.

| Closure Stress (psi) | 16/30 | | |
|----------------------|---------------------------------------|-------------------------------|-----------------|
| | Baseline Fracture Conductivity (mDft) | Baseline Permeability (Darcy) | Pack Width (mm) |
| 2000 | 10950 | 646 | 5.17 |
| 4000 | 8450 | 516 | 4.99 |
| 6000 | 6257 | 396 | 4.82 |
| 8000 | 4161 | 272 | 4.67 |
| 10000 | 2470 | 167 | 4.51 |

Table 9: Baseline Conductivity Results, 2lb/ft², 250°F, Ohio Sandstone



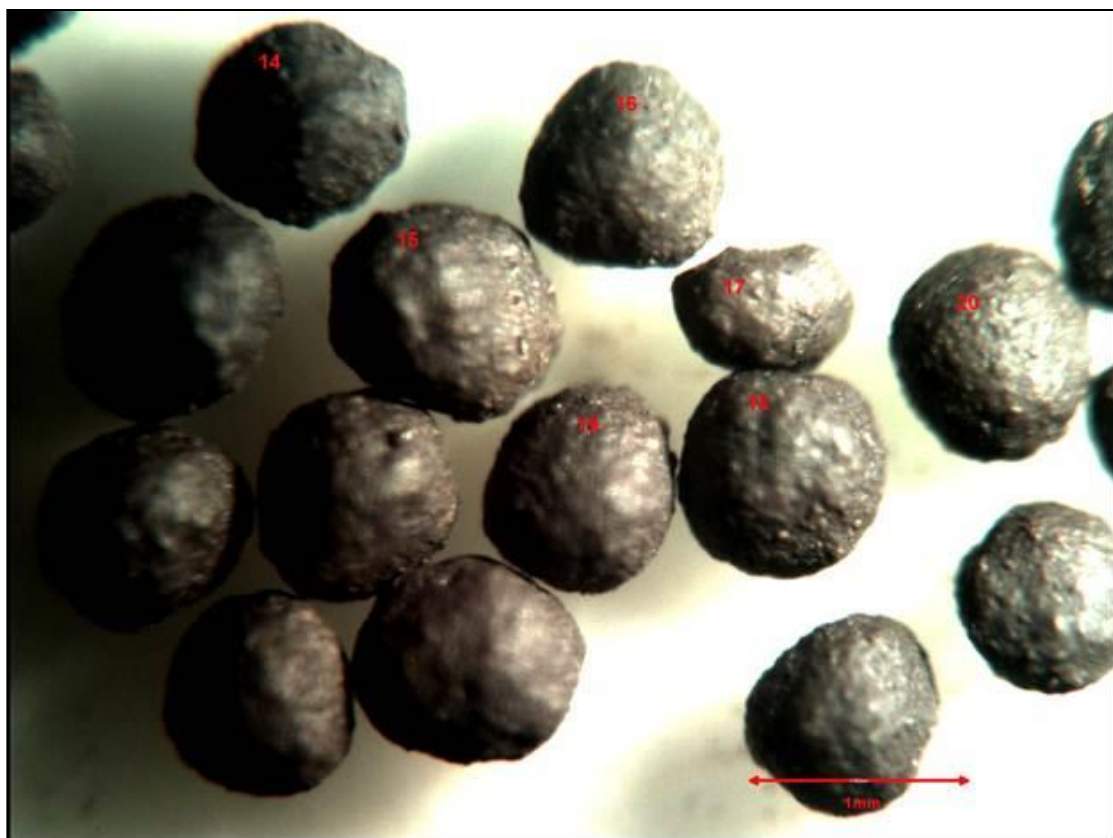


APPENDIX A

Proppant Photomicrographs



Proppant Sample 16/30 (a)



Proppant Sample 16/30 (b)