



**QUALIFYING FLUID & PROPPANT  
PERFORMANCE®**

**Minnesota Sand, LLC**

**Mr. Richard Frick  
(507) 458-2023**

**Raw Sand: 40/70 Cut**

**ISO 13503-2 Sand Analysis**

**RFA #100-11-11-13-29-C**

**November 23, 2011**



**Information:**

Sample Origin:	UPS Delivery
Sales Contact:	Brandon White
Report Generated by:	Kirk Decker
Lab Technician:	Brandon White

**Background:**

November 11, 2011 two unprocessed sand samples were received from Mr. Richard Frick, **Minnesota Sand, LLC** for the purpose of qualifying the material according to **ISO 13503-2** International Standards. Following review of the composite sieve, the “**40/70 Cut Sand**” is tested for all ISO 13503-2 tests. The request is verified via telephone from Mr. Richard Frick, Minnesota Sand, LLC and Brandon White, PropTester.

**Color Analysis – Proppant Test Data:**

Proppant test results are referenced against ISO 13503-2 standards and available public data. Classification by color or numerical variance does not imply a level of performance. However, coloring of standard and public data does indicate a specific range of variance of sample test results. The numerical ranges are typical of data variance between laboratories that participate in ISO round robin or performance (e.g. conductivity) evaluation. When limits (e.g. > or <) are used, then only green or red will apply.



## Conclusions / Comments:

### Minnesota Sand, LLC 40/70 Cut Sand

- The **Minnesota Sand, LLC 40/70 Cut Sand** meets all ISO 13503-2 Standard requirements for a 40/70 mesh frac grade sand proppant. **Figure 1 & Photomicrograph 1**
- **Particle Size Distribution** of the **Minnesota Sand, LLC 40/70 Cut Sand** is > 90% in size with a **Median particle Diameter (MPD)** of 0.307 mm. This sieve distribution varies somewhat from that of 40/70 commercially available frac sand public data. **Figures 1 & 2**
- ISO Standard **Acid Solubility** allows no more than 3.0% solubility in 12/3 HCl/FL for 40/70 mesh sand. **Minnesota Sand, LLC 40/70 Cut Sand** at 1.2% solubility in this acid blend meets the ISO Standard. This sample Acid Solubility exceeds that of 40/70 frac sand public data. **Figure 1**
- The **ISO Crush** requires that crush properties of a proppant are evaluated at incremental pressures until fines generated are  $\leq 10\%$  by weight. For the **Minnesota Sand, LLC 40/70 Cut Sand** the crush test was halted @ 12,000 psi with the sample producing an average of 8.82% fines. According to ISO 13503-2, **Minnesota Sand, LLC 40/70 Cut Sand** would be classified *12k proppant*. **Figure 1**
- **pH of Water Extract** test measures the pH of water with proppant to determine compatibility with frac fluid systems. The pH of this **Minnesota Sand, LLC 40/70 Cut Sand** at 8.4 is benign and if representative should not impact frac fluid chemistry. **Figure 1**



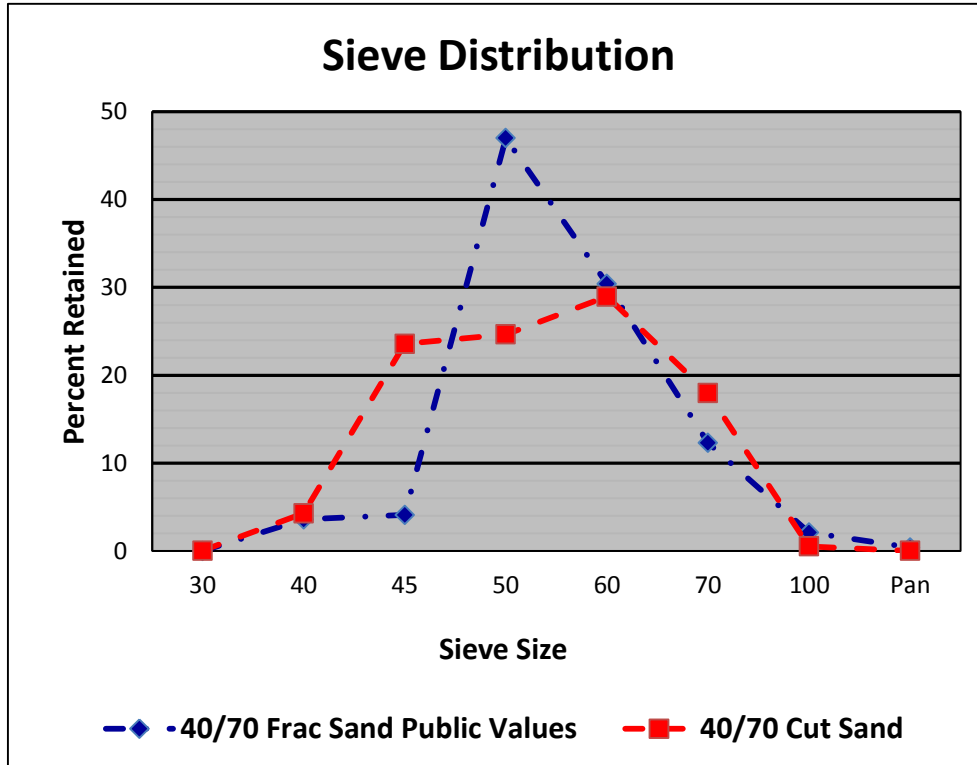
**Figure 1- Proppant Test Data:**  
**Minnesota Sand, LLC 40/70 Cut Sand**

<b>Quick Chek ✓</b>		<b>ISO 13503-2</b>	<b>40/70 Frac Sand Public Values</b>	<b>40/70 Cut Sand</b>	
Turbidity (NTU)		≤ 250	36	9	
Krumbein Shape Factors					
Roundness		≥ 0.6		0.8	
Sphericity		≥ 0.6		0.6	
Clusters (%)		≤ 1.0		0	
Bulk Density (g/cm <sup>3</sup> )				1.53	
Bulk Density (lb/ft <sup>3</sup> )				95.74	
Specific Gravity			2.65	2.63	
Particle Size Distribution, mm		Mesh size			
	0.600	30	≤ 0.1	0	0.0
	0.425	40		3.6	4.3
	0.355	45		4.1	23.6
	0.300	50		47.0	24.6
	0.250	60		30.4	29.0
	0.212	70		12.3	18.0
	0.150	100		2.1	0.5
	<0.150	Pan	≤ 1.0	0.4	0.0
	Total			100	100.0
	% In Size		≥ 90	93.8	95.1
Mean Particle Diameter, mm				0.317	
Median Particle Diameter (MPD), mm				0.307	
Solubility in 12/3 HCL/HF for 0.5 HR @ 150°F (% Weight Loss)		≤ 3.0	0.4	1.2	
pH of Water Extract – Test Fluid				7.80	
pH of Water Extract – Test Fluid & proppant				8.40	
Settling Rate (ft/min)				16.72	
<b>Crush Chek ✓</b>					
ISO Crush Analysis (% Fines) 4lb/ft <sup>2</sup> @ 12,000 psi		≤ 10		8.82	
ISO Crush Analysis (% Fines) 4lb/ft <sup>2</sup> @ 13,000 psi		≤ 10		10.63	
<b>Sample Correlation +/-0 to +/-8.999% Variance From ISO or Published Data</b>		<b>Sample Correlation +/-9 to +/-25% Variance From ISO or Published Data</b>		<b>Sample Correlation -25&gt; X &gt; +25% Variance From ISO or Published Data</b>	



**Figure 2- Sieve Distribution:**  
**Minnesota Sand, LLC *40/70 Cut Sand***

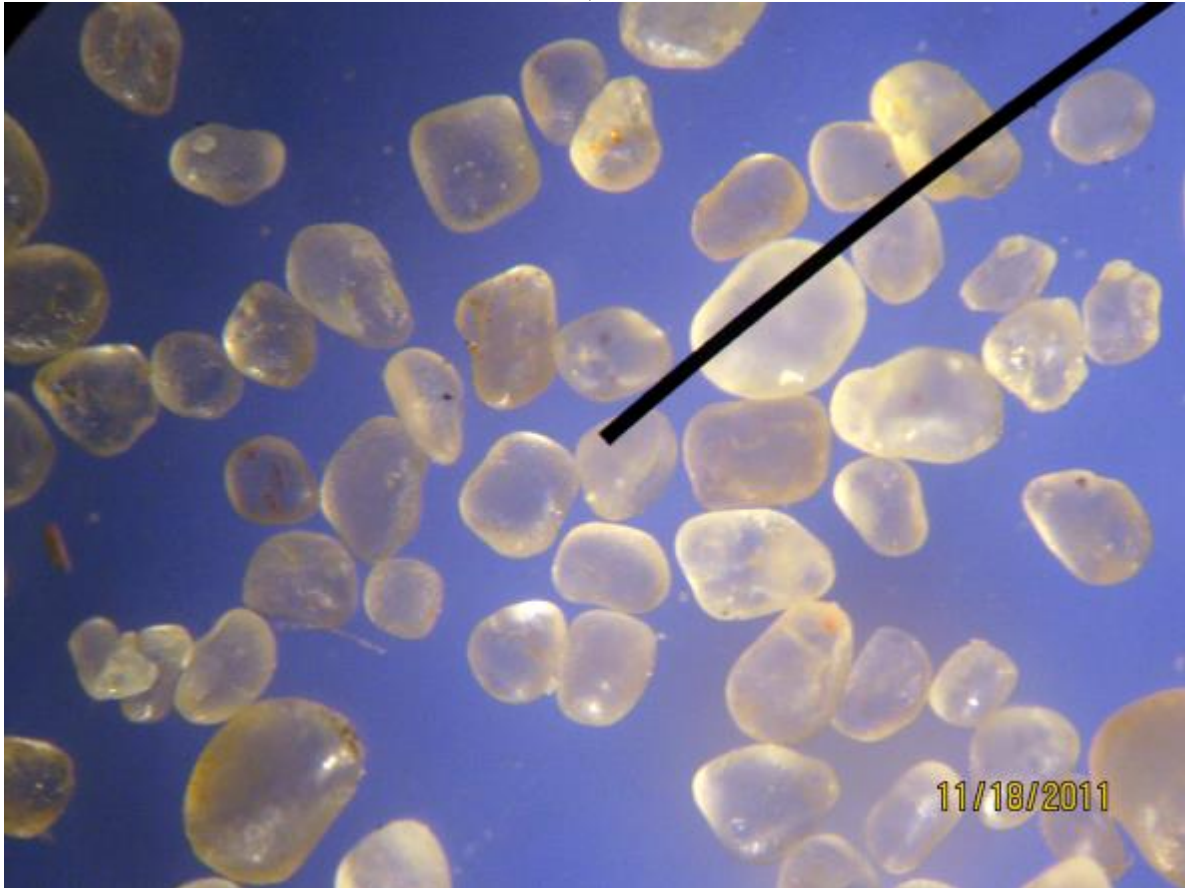
Sieve Size	40/70 Frac Sand Public Values	40/70 Cut Sand
30	0	0.0
40	3.6	4.3
45	4.1	23.6
50	47	24.6
60	30.4	29.0
70	12.3	18.0
100	2.1	0.5
Pan	0.4	0.0





Minnesota Sand, LLC 40/70 Cut Sand RFA# 100-11-11-13-29-C

**Photomicrograph 1**  
**Minnesota Sand, LLC *40/70 Cut Sand***





## **Testing Definitions & Descriptions**

**Turbidity** – A measure to determine the levels of dust, silt, suspended clay, or finely divided inorganic matter levels in fracturing proppants. High turbidity reflects improper proppant manufacturing and/or handling practices. The more often and more aggressively a proppant is handled, the higher the turbidity. Offloading pressures exceeding manufacturer guidelines can have a detrimental effect on the proppant performance. Produced dust can consume oxidative breakers, alter fracturing fluid pH, and/or interfere with crosslinker mechanisms. As a result, higher chemical loadings may be required to control fracturing fluid rheological properties and performance. If fluid rheology is altered, then designed or modeled fracture geometry and conductivity will be altered. A change in conductivity directly correlates to reservoir flow rate.

**Krumbein Shape Factors** – determines proppant roundness and sphericity. Grain roundness is a measure of the relative sharpness of grain corners, or of grain curvature. Particle sphericity is a measure of how closely a proppant particle approaches the shape of a sphere. Charts developed by Krumbein and Sloss in 1963 are the most widely used method of determining shape factors.

**Clusters** – Proppant grains should consist of single, well-rounded particles. During the mining and manufacturing process of proppants, grains can attach to one another causing a cluster. It is recommended by ISO 13503-2 that clusters be limited to less than 1% to be considered suitable for fracturing proppants.

**Bulk Density** – A dry test to gain an estimation of the weight of proppant that will fill a unit volume, and includes both proppant and porosity void volume. This is used to determine the weight of a proppant needed to fill a fracture or a storage tank.

**Specific Gravity** – Also called Apparent Density, it includes internal porosity of a particle as part of its volume. It is measured with a low viscosity fluid that wets the particle surface.

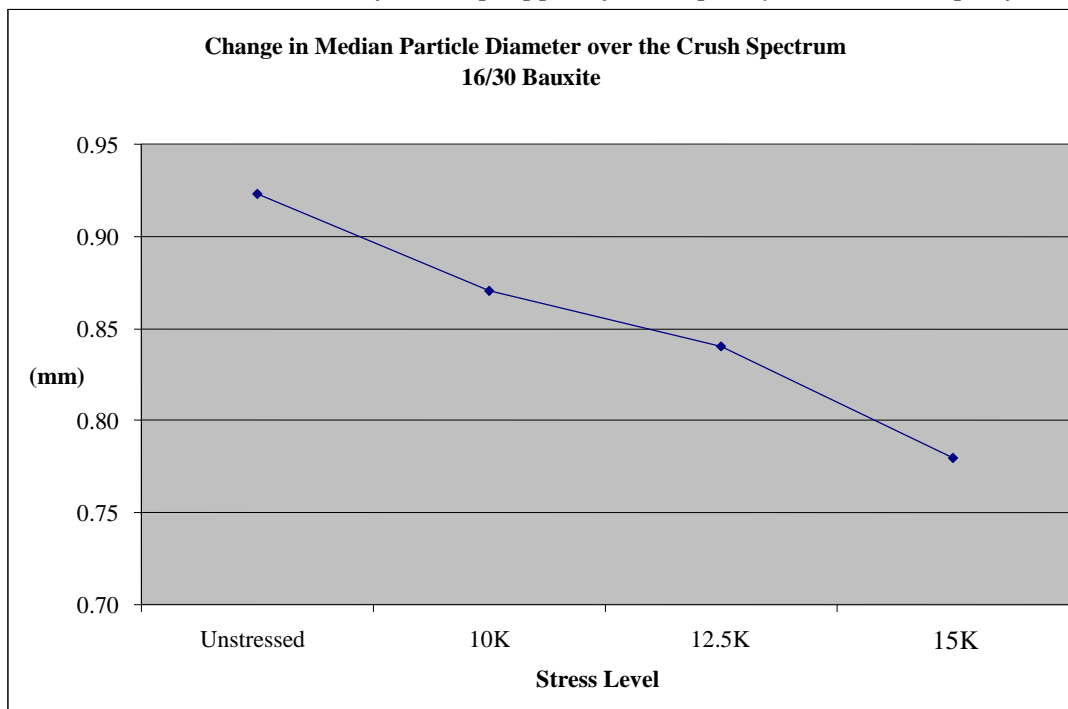
**Sieve Analysis: Particle Size Distribution & Median Particle Diameter** – Also called a sieve analysis, this test determines the particle size distribution of a proppant sample. Calibrated sieves are stacked according to ISO 13503-2 recommended practices and loaded with a pre-measured amount of proppant. The stack is placed in a Ro-Tap sieve shaker for 10 minutes and then the amount on each sieve is measured and a percent by weight is calculated on each sieve. A minimum of 90 % of the tested proppant sample should fall between the designated sieve sizes. Not over 0.1% of the total tested sample should be larger than the first sieve size and not over 1.0% should fall on the pan. The in-size percent, mean particle diameter, and median particle diameter are calculated which relates directly to propped fracture flow capacity and reservoir productivity.



## Testing Definitions & Descriptions

**API / ISO Crush Test** – The **API** test is useful for comparing proppant crush resistance and overall strength under varying stresses. A proppant is exposed to varying stress levels and the amount of fines is calculated and compared to manufacturer specifications. Studies by Coulter & Wells (e.g. SPE JPT, June 1972, pp. 643-650) have demonstrated that as little as 5% added fines can reduce propped fracture conductivity by 50%. The **ISO** test classifies a proppant according to the stress at which  $\leq 10\%$  fines are generated; for example an ISO 7k proppant would produce  $\leq 10\%$  fines at 7000 psi.

A **PT Crush Profile** (see example below) can show graphically how median particle diameter (MPD) can vary with changes in closure stress. Unlike the ISO crush test, the PT Crush Profile uses the entire proppant sample for crushing at each stress, the sample is then sieved to determine particle distribution, and MPD is then calculated. A change in MPD directly correlates to flow capacity and reservoir productivity. *This test, ordered separately, provides a more realistic view of initial proppant flow capacity at reservoir specific stresses.*



**Acid Solubility** – The solubility of a proppant in 12-3 hydrochloric-hydrofluoric acid (HCL-HF) is an indication of the amount of undesirable contaminates. Exposing a proppant (specifically gravel pack/frac pack materials) may result in dissolution of part of the proppant, deterioration in propping capabilities, and a reduction in fracture conductivity in the zone contacted by such acid. The loss of fracture conductivity near the wellbore may cause a dramatic reduction in well productivity, as has been demonstrated by Raymond and Binder (JPT, January 1967, Pgs. 120-130).





## **Testing Definitions & Descriptions**

**Resin Content/Loss on Ignition (LOI)** – This test determines the resin content remaining on the proppant. Resin content is a direct function of the proppants strength and its ability to encapsulate the substrate when exposed to high stress levels. By reducing fines generation and migration, the proppant pack remains clean, allowing maximum well production.

**Resin Coating Efficiency** – Used to determine the percent of uncoated grains in a resin coated proppant sample.

**Unconfined Compressive Strength (UCS)** – Grain-to-grain bonding at specific temperatures over time will develop bond strength that can be measured by using a UCS test. This test directly reflects the proppants ability to bond downhole in order to reduce embedment and control proppant flowback. By reducing embedment and keeping the available proppant in place, fracture width can be maximized.

**pH of Water Extract** – This test reflects the potential chemical impact of a proppant on fracturing fluid pH. Processing or manufacturing of a proppant can leave residues, or ‘free phenol’ in the case of resin coated proppants, which can interfere with polymer hydration rates, cross-linking mechanisms, etc. These effects if detected can usually be remedied by increasing buffering capacity, but if undetected can alter fracturing fluid rheology, change fracture geometry, and impact propped fracture conductivity. A change in conductivity directly correlates to reservoir production rate.



## TEST PROCEDURES

**PropTester®** & ISO test procedures were applied in this Request for Analysis (RFA)

### **Quick Chek**

Turbidity  
 Microscopic Exam  
     Krumbein Shape Factors  
     Clusters  
     Photomicrographs  
 Bulk Density  
 Specific Gravity  
 Sieve Analysis  
 Particle Size Distribution  
 Mean Particle Diameter  
 Median Particle Diameter (MPD)

### Procedures

**ISO 13503-2**  
**ISO 13503-2**  
  
**ISO 13503-2**  
**ISO 13503-2**  
**ISO 13503-2**  
**ISO 13503-2**  
**ISO 13503-2**  
**PropTester®**

### **Crush Chek**

API Crush Test  
 ISO Crush Test  
 PT Crush Profile

### Procedures

**API RP 56/58/60**  
**ISO 13503-2**  
**PropTester®**

### **Res Chek**

% Resin Content, LOI  
 Coating Efficiency %  
 Unconfined Compressive Strength (UCS)  
 pH of Water Extract

### Procedures

**PropTester®**  
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**PropTester®**

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