All data taken at Pacific Northwest National Laboratory (PNNL)

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## SAMPLE CONDITIONS & PHYSICAL PROPERTIES

| | |
|---|---|
| Chemical name | Sand: mostly quartz with hematite and defect-rutile impurities |
| Chemical formula | $SiO_2$ along with $Fe_2O_3$ and $TiO_2$ impurities |
| Synonyms | n/a |
| CAS number | n/a |
| Location of field sample | n/a |
| History of sample | Heated to 760 °C for ~ 2 hrs on 9/21/1990 |
| Molecular Weight | n/a |
| Melting Point | n/a |
| Boiling Point | n/a |
| Density (20 °C) | n/a |
| Hardness, Mohs scale | n/a |
| Crystallography: | |
|     Cell dimension | a = Å  b = Å  c = Å |
|     Crystal system | |
|     H-M symbol (point gr) | |
|     Space group | |
|     H-M symbol (space gr) | |
|     Crystal habit | |
| Color | Various colors of tan, pink, and orange |
| Diaphaneity | Translucent to sub-translucent |
| Particle size | $417 \pm 186\,\mu m$ |
| Particle size assessment | Optical microscopy |
| Supplier | n/a |
| Stated purity | n/a |
| Date packed | 31 August 2016     Weight: 3.494 grams |
| Synthesis method | n/a |
| Synthesis reference | n/a |
| Texture | Hard, sub-round to angular granules |
| Physical state | Solid |
| Surface roughness | n/a |
| Elemental composition | n/a |
| Isotopic composition | n/a |
| Moisture content | n/a |
| Temperature of sample | $25 \pm 2$ °C |
| Substrate | n/a |

## INSTRUMENT PARAMETERS

### Tensor 37 FT-IR manufactured by Bruker Optics

| | |
|---|---|
| External diffuse reflectance accessory | A 562-G integrating sphere |
| Sphere diameter | 75 mm |
| Angle to normal incidence | 14.8° |
| Sphere opening diameter | 19 mm (entrance port) |
| Spectral range | 7,500 to 600 cm$^{-1}$ saved; 7500 to 600 cm$^{-1}$ reported |
| Beamsplitter | Ge on KBr |
| Detector (dia. Det. Port in sphere) | 2×2 mm, 60° field of view MCT (550; 0.9); 1 cm |
| Apodization function | Blackman-Harris 3-term |
| Aperture | 6 mm |
| Coadded scans | 2048 |
| Scanner speed | 40 kHz |
| Switch gain on | 512 points |
| Low pass filter | Open |
| Scan technique | double-sided, forward-backward |
| Non-linear correction | On |
| High and low folding limit | 15800.54-0.00 cm$^{-1}$ |
| Phase resolution | 32.00 |
| Phase correction mode | Mertz |
| Zerofilling | 4× |
| Wavenumber accuracy | ± 0.4 cm$^{-1}$ |
| Spectral resolution | 4 cm$^{-1}$ |
| Accuracy verification | 10/28/2015 |
| Wavelength vetted on: | ICL polystyrene standard #0009-7394-0025A, thin film |
| Reflectance: | ±2% using SRS reflectance standards 50-010-DH27B-4878 |



Figure 1: The Bruker 562-G integrating sphere (a) and Tensor 37 (b)
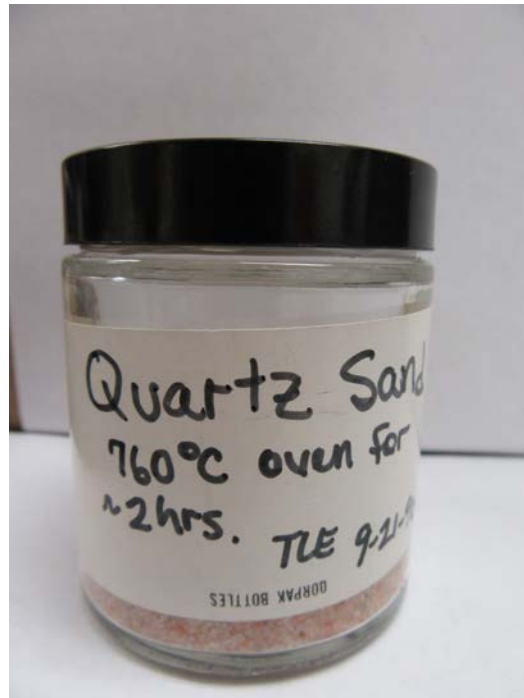
**Photographs of sample Sand**



Figure 2:  Sand in container.



Figure 3: Sand loaded in IR sample cup.

**Optical microscopy —**

A Keyence VHX-1000 digital microscope with 16-bit resolution is used to provide photomicrographs of the various samples and particle sizes. Software included with the microscope differentiates the brightness and colors in the image and extracts the bright objects to produce a binary image. The software assumes all adjacent bright points are part of the same object then calculates the area for each of these objects. The area (A) is used to calculate the mean particle diameter (d) by assuming the particles are spherical and using the relationship $d=(4*A/\pi)1/2$. Although the assumption of spherical particles is clearly not always valid, this procedure provides a reasonable estimate of the mean particle size.
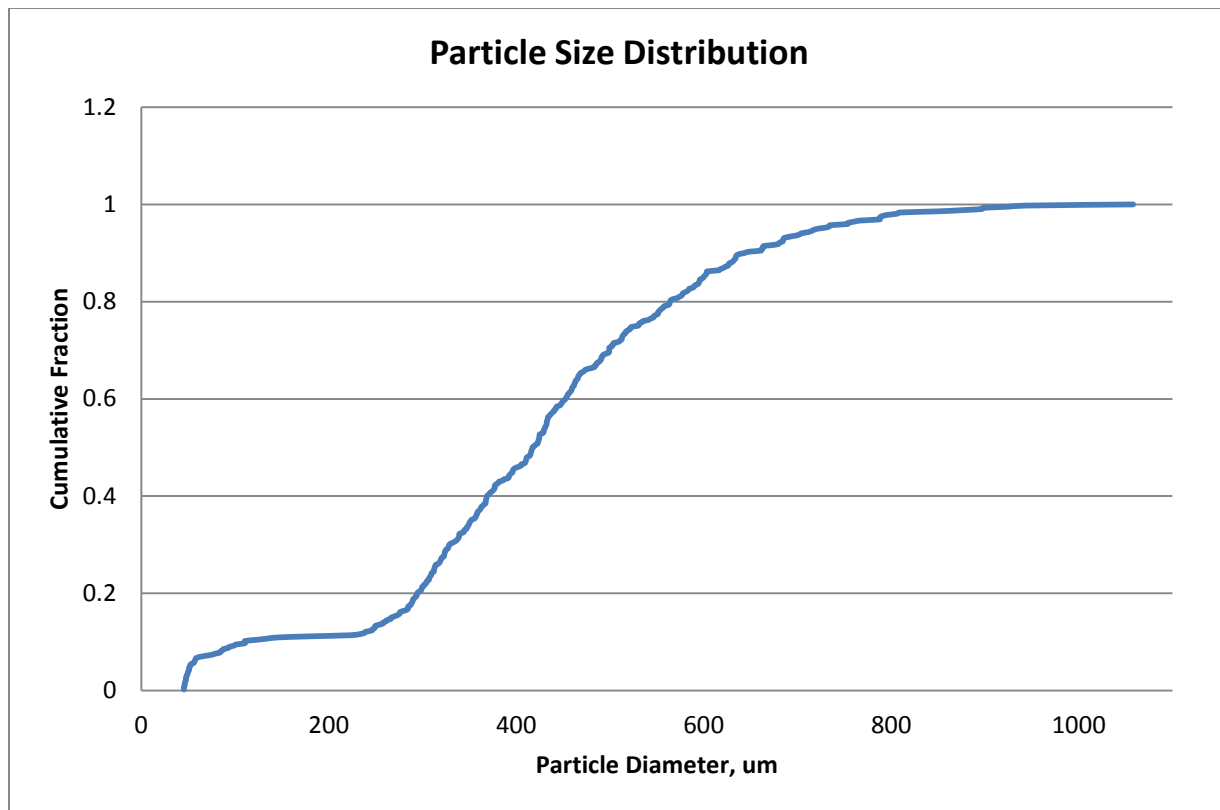


Figure 4:  Photomicrograph of Sand.

Figure 5: Particle size distribution of Sand.

## ANCILLARY METHODS TO CONFIRM IDENTITY/PURITY:

A primary analytical method used to quantify or confirm the composition of the crystalline solid samples is x-ray diffraction (XRD). This technique involves the interpretation of x-ray diffraction patterns to determine the relative positions of atoms within a crystalline solid. While structural information obtained from XRD can be used to identify the composition of a solid there are several limitations to this technique including: a) limited ability to characterize amorphous solids, b) identification can only be performed for compounds that have been structurally characterized, c) some compounds are chemically unique but structurally similar which confounds identification based on structure alone, d) there are large uncertainties (~5%) when quantifying mixtures and trace impurities (<5%) often go undetected.

Quartz grains with various colors were present in the sample and an optical microscope is used to help select grains that are strongly colored.  A smaller quartz grain was selected for analysis that had a surface coating of a red powder to maximize the volume fraction of impurity. The diffraction pattern is overwhelmingly quartz, but near the background, hematite is clearly observed (see Figure 6). The hematite peaks are broader than the quartz peaks consistent with a finer crystalline size. The microXRD shows peaks that are more than 200 times smaller than the largest quartz peak.

In addition to the quartz grains, a few small very dark grains are present that look quite different.  The diffraction pattern for these grains shows the major phase is rutile, in addition to quartz. Since Titanium fluoresces with the x-rays, a huge background is observed making identification difficult. Rutile is naturally white so the black color could be caused by an impurity or substitution/defects in the rutile lattice.  Since hematite is also present, $Fe^{3+}$ may substitute for $Ti^{4+}$ and create oxygen vacancies.
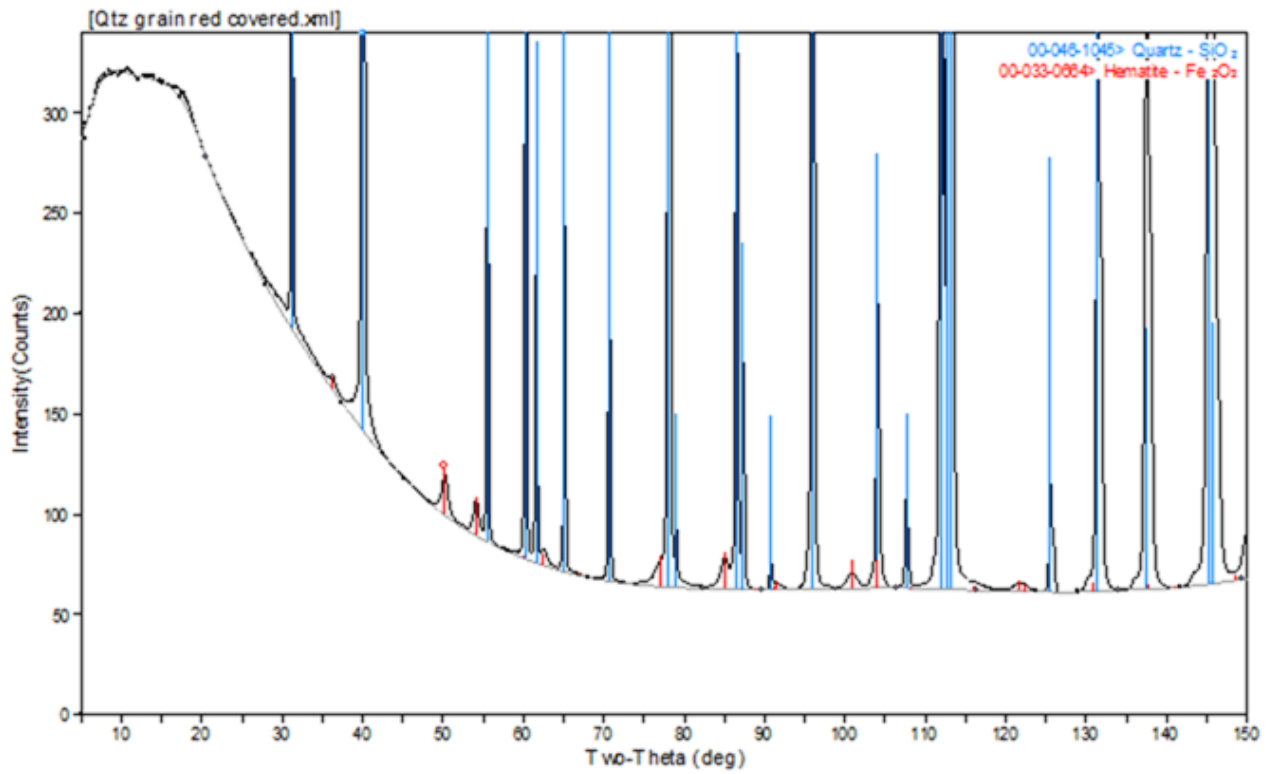
Figure 6: X-ray Diffraction Pattern of Sand.