

All data taken at Pacific Northwest National Laboratory (PNNL)

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SAMPLE CONDITIONS & PHYSICAL PROPERTIES

| | |
|--------------------------|---|
| Chemical name | Malonic acid |
| Chemical formula | C ₃ H ₄ O ₄ |
| Synonyms | Propanedioic acid; Methanedicarboxylic acid |
| CAS number | 141-82-2 |
| Location of field sample | n/a |
| History of sample | n/a |
| Molecular Weight | 104.06 g/mol |
| Melting Point | 132 - 135 °C |
| Boiling Point | 263.5 °C |
| Density (20° C) | 1.600 g/cm ³ |
| 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 | White |
| Diaphaneity | Sub-translucent to opaque |
| Particle size | 70 ± 32 µm |
| Particle size assessment | Optical microscopy |
| Supplier | Aldrich |
| Stated purity | ≥ 99.95% |
| Date packed | 25 July 2016 Weight: 2.123 grams |
| Synthesis method | n/a |
| Synthesis reference | n/a |
| Texture | Irregularly shaped flakes/crystals/needles and powder |
| Physical state | Solid |
| Surface roughness | n/a |
| Elemental composition | n/a |
| Isotopic composition | n/a |
| Moisture content | n/a |
| Temperature of sample | 25 ± 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 | $\pm 0.4 \text{ cm}^{-1}$ |
| Spectral resolution | 4 cm^{-1} |
| Accuracy verification | 10/28/2015 |
| Wavelength vetted on: | ICL polystyrene standard #0009-7394-0025A, thin film |
| Reflectance: | $\pm 2\%$ using SRS reflectance standards 50-010-DH27B-4878 |



Figure 1: The Bruker 562-G integrating sphere (a) and Tensor 37 (b)

Photographs of sample Malonic acid



Figure 2: Malonic acid in Aldrich container.



Figure 3: Malonic acid loaded in IR sample cup.

PARTICLE SIZE PREPARATION AND CHARACTERIZATION

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 $d = \sqrt{4A/\pi}$. Although the assumption of spherical particles is clearly not always valid, this procedure provides a reasonable estimate of the mean particle size.

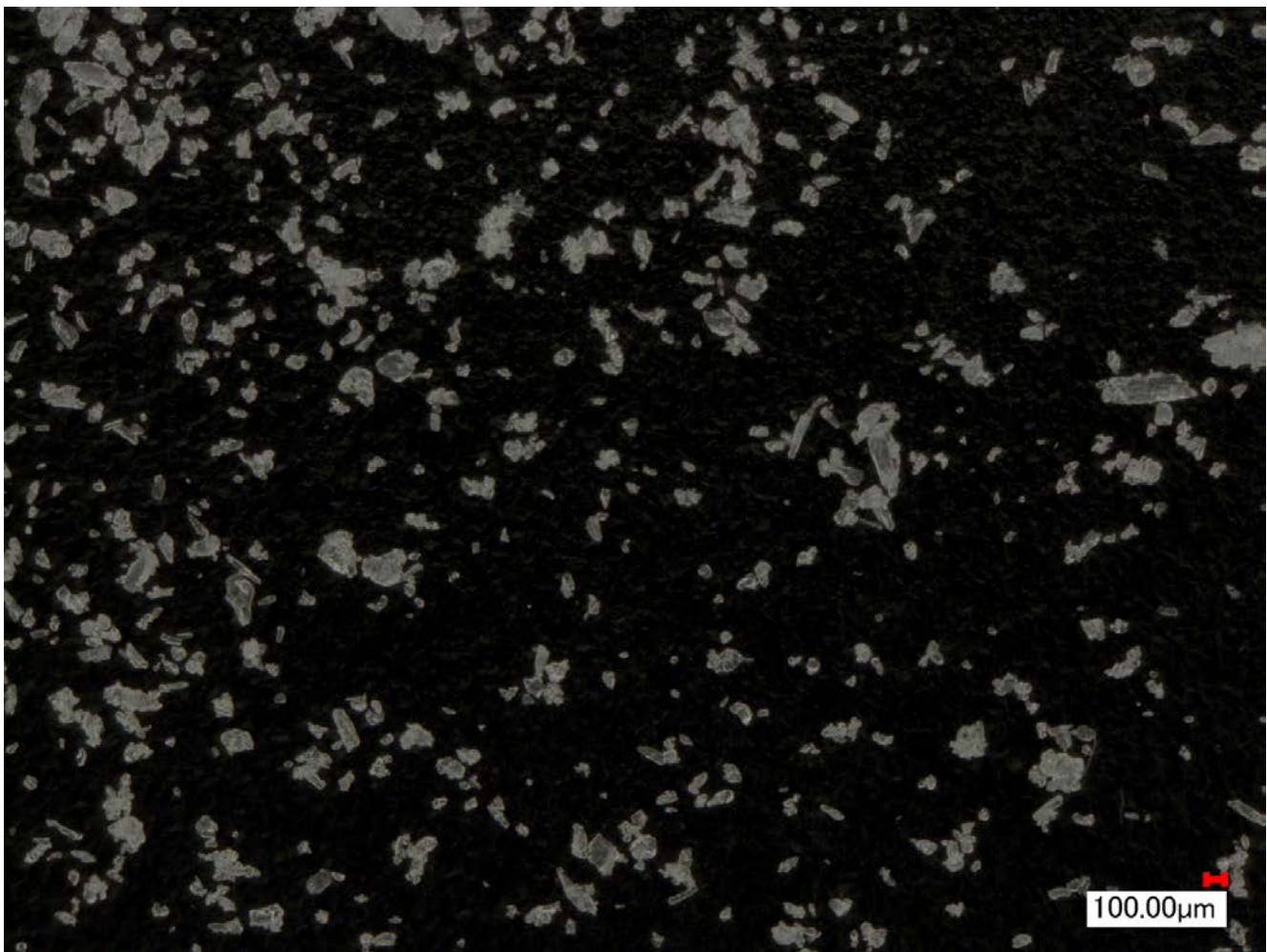


Figure 4: Photomicrograph of Malonic acid.

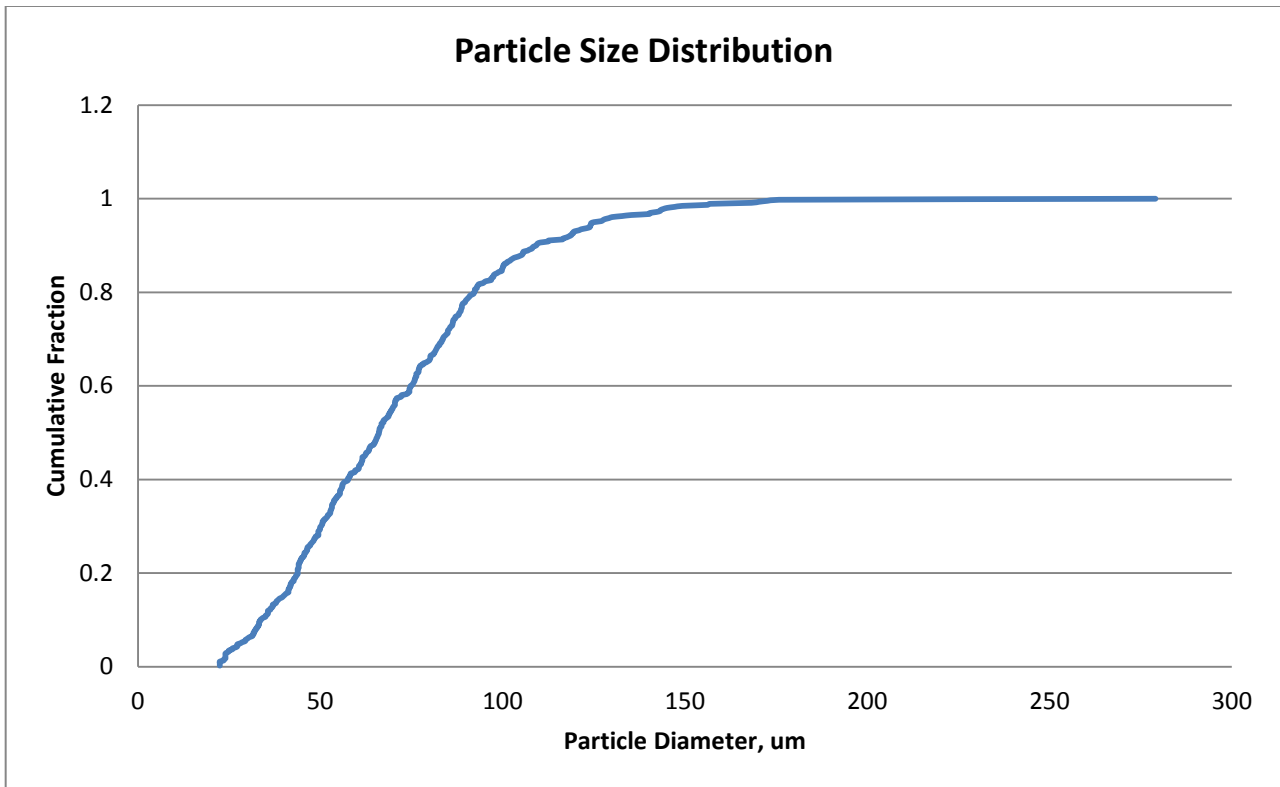


Figure 5: Particle size distribution of Malonic acid.