IARPA MORGOTH'S CROWN: Preparation Materials for Sieved Samples

Contents

Preparation Methodology for Sieved Samples

The sieved sample coupons were fabricated for the IARPA "MORGOTH'S CROWN" Prize challenge to investigate the effect of particle size on the diffuse reflectance spectrum at infrared wavelengths. The Prize Challenge was designed within the constraints of relevant analyte materials (featuring typical absorbance features in the mid-IR), typical substrate parameters, (rough and smooth, reflective, transmissive, and intermediate, and exhibiting their own characteristic spectral features) and realistic particle size distributions (log normal). Particles with diameters of 1 to 40 microns are relevant for fingerprint transfers and also adhere persistently to many surfaces after a spill or aerosol deposition. This size range may exhibit anomalous spectral features due to interference resonances where the particle diameter matches the wavelength of light. This size range will also exhibit complex mixing of the analyte and substrate spectral features due to the fact that the diameter may exceed the absorption depth at some wavelengths, but transmit other wavelengths through to the substrate.

To achieve these objectives, the analytes of interest were ground into fine particulate powders using standard mortar and pestle methods. The resulting particles were sifted through a sieve screen and allowed to disperse by gravity randomly onto the substrate surface below. Due to the small particle diameters, once resting on the surface they are held in place by van der Walls forces stronger than gravity and they remain fixed unless disturbed by a relatively strong force. To study the effects of particle size, two different sieve mesh sizes were used. The smaller mesh provided 20 μm openings, and the larger mesh provided 38 μm openings. The resulting particle size distributions were measured on each sample coupon using a high magnification optical microscope. In both cases, the mass loading was selected to provide realistic surface "fill factors" where the particles cover a small fraction of the surface area with a minimal number of "clusters" (where two or more particles touch each other).

The results of optical microscopy for each sample coupon are included in the available Prize Challenge metadata. While the precise particle size distributions are unique to each coupon, they can be grouped according to which mesh size was used for sieving. For the sieve with 20 μm openings, the particles form an approximately log normal size distribution centered around a median value of ≈12 μm with a standard deviation of ≈6 μm. For the 38 μm mesh, the particles form an approximately log normal size distribution centered around a median value of ≈24 μm with a standard deviation of ≈12 μm. The metadata provided include other parameters of interest, including the histogram of observed effective particle diameters, the "fill factor" (the percentage of the substrate surface area covered by all of the particles –about 1%), and the total number of particles counted in the microscope image. Such information could provide valuable input to physical models used to replicate, simulate, or fit to the observed diffuse infrared reflectance spectra from the sieved sample coupons.