

THE AMERICAN MUSEUM OF NATURAL HISTORY MINERAL LIBRARY FOR SPECTROSCOPIC STANDARDS. A. Nissinboim¹, D. S. Ebel², G. E. Harlow², J. S. Boesenberg², K. M. Sherman², E. R. Lewis², T. N. Brusentsova³, R. E. Peale³, C. M. Lisse⁴, C. A. Hibbitts⁴. ¹Dept. of Geology, Brooklyn College, 2900 Bedford Ave., Brooklyn NY 11210 (a.nissinboim@gmail.com); ²Department of Earth and Planetary Sciences, American Museum of Natural History, 79th Street at Central Park West, New York, New York 10024 (debel@amnh.org, gharlow@amnh.org, bosenbrg@amnh.org, kc2262@gmail.com, erlewis@uchicago.edu); ³Dept. of Physics, University of Central Florida, Orlando FL 32816 (peale@mail.ucf.edu, tbrusentova@mail.ucf.edu); ⁴Applied Physics Laboratory, The Johns Hopkins University (Carey.Lisse@jhuapl.edu, Karl.Hibbitts@jhuapl.edu).

Introduction: The American Museum of Natural History Department of Earth and Planetary Sciences has developed a library of mineral standards for astronomical spectroscopy. Samples are a subset of existing collections within the Museum. The initial motivation for the development and curation of this collection was to obtain new transmission spectra at infrared wavelengths in support of the Herschel space telescope observatory launched in May 2009. The Herschel photo-detector array camera and spectrometer (PACS) has longer wave coverage (48-175 cm^{-1}) than the Spitzer telescope. High-quality transmittance spectra of very finely ground ($< 2 \mu\text{m}$), well-characterized mineral samples are necessary to interpret PACS spectra.

Fine-grained mineral dust in astrophysical environments is of fundamental importance in understanding a myriad of processes. This dust goes through an entire lifecycle of change from “birth” until “adulthood” in the interstellar medium and stellar environments. The minerals that have been analyzed so far include many that have been identified in planetary nebulae, supernova remnants, circumstellar disks, comets [1-3], Stardust mission samples (comet Wild 2), observations of the Deep Impact mission using the Spitzer telescope [4], stratospheric interplanetary dust (IDP), the Moon, Mars, asteroids [5], and meteorites, or which are expected theoretically from condensation models [6-7].

It is our perception that many libraries of spectra contain data on minerals specimens which are not curated as part of a long-term (e.g. 100+ years) collection. For this study, each highly characterized specimen is digitally documented in a database that we can reasonably anticipate will be publicly available over decades spanning the careers of individual researchers. The comprehensive methodology used in characterizing each sample subject to spectroscopic measurement is critical in building a database of re-analyzable standards. We anticipate that these standards will aid future discoveries through better interpretation of astronomical spectra.

Samples: Though we have a very broad and growing library of mineral standards, we have focused efforts on minerals for which existing data is sparse. These minerals groups include the carbonates, sulfides,

and phyllosilicates, particularly clay minerals. For each mineral class, we have analyzed endmembers, compositions that lie in between endmembers (i.e., on solid solution series, e.g., Fo_{75} olivine, $\text{Mg}_{1.5}\text{Fe}_{0.5}\text{SiO}_4$), and other species covering wide compositional ranges.

Methodology: To develop the library of mineral standards for transmission infrared astronomical spectroscopy, a series of tests and procedures were undertaken on each sample. In order to determine that a specimen is suitable as a standard for this project, the crystallographic parameters, exact chemical composition, degree of homogeneity, microstructure (e.g., exsolution), and size-distribution must be tightly constrained and documented. Once this is done, the sample is prepared in polyethylene pellets for spectroscopic measurements.

Sample Selection. Samples are chosen from the vast, comprehensive mineral collection at the AMNH. Macroscopic features are inspected to infer the likelihood of mineralogical and chemical homogeneity (e.g., clear, colorless quartz with no impurities and/or inclusions would be ideal, because colored quartz indicates that there is an elemental impurity or radiation damage in the crystal structure). Each sample is documented with a macroscopic image (photograph) including name, collection number, and locality.

Sample Grain Separation. After selecting a sample and deciding which sub-volume of that sample would most fit the project goals, we remove the sub-volume of interest and delicately crush it into pieces ranging from 1mm –10mm in size. These pieces are examined under the stereo microscope to observe their microscopic mineralogical features. We note the presence of any microscopic impurities (zoning, other phase exsolution, inclusions) and remove heterogeneous grains from the rest of the sample by handpicking wherever possible. When necessary, crushed samples are further cleaned to remove impurities, using techniques that include but are not limited to magnetic separation and acid baths. Microphotographs are taken after this step, and added to the database.

X-ray Single Crystal Diffraction. For each sample, a single crystal is analyzed in the Rigaku DMAX/Rapid single crystal diffractometer facility at AMNH. The results are cross-referenced with a digital

database and the mineral identity and major element chemical composition are determined. This is of high importance because many minerals sold by dealers, or even those in museum collections, are named too broadly (or wrongly) and specific phases and species are often poorly documented.

Electron Microprobe. Multiple macroscopic grains (1 to 3 mm) from each sample are prepared for electron microprobe (EMP) analysis on a Cameca SX100 electron microprobe. Grain mounts are carbon-coated epoxy pellets usually containing 5-6 grains of minerals of the same class/subclass per mount. The grains are analyzed using wavelength dispersive spectrometry (WDS) and a well-characterized suite of EMP analysis standards. Their exact chemical compositions are determined using mineralogical software developed by G. Harlow, from the EMP results. Back-scattered electron (BSE) imaging is used to assess areas of impurities in the grains and both WDS and energy dispersive spectrometry (EDS) are used to determine the chemical composition of impurity phases present, if any. At this point, samples lacking a high level of homogeneity are eliminated from the study. Each sample in the database has an associated BSE image along with complete cation distribution information based on EMP analysis.

Sample Comminution. In order to yield the least amount of light scattering, leading to the most accurate results for transmission IR spectroscopy at short wavelengths, our specimens must be of nearly one micron average grain size [8]. We begin the grinding process by manually crushing each sample in a mortar and pestle and then running the small grains through a 0.5 mm sieve. Once the sample is fully crushed to this level, the fine grains are placed inside a container for the McCrone MicroMill device, filled with 48 agate or corundum cylinders (depending on the sample hardness). Also placed in the container is a small amount (carefully calibrated) of cyclohexane or mineral spirits to perform slurry grinding. Slurry grinding is performed in the McCrone MicroMill for 15 to 30 minutes per sample, depending on hardness. The cylinders in the container produce line contact blows and planar shearing on the grains inside (as opposed to the random contact blows of conventional ball milling). These techniques along with the slurry grinding are preferred because they reduce defects in the crystal structures, cause no chemical degradation, relatively little sample loss, and a high consistency of the product. Samples susceptible to oxidation (e.g., pyrrhotite $Fe_{1-x}S$) are ground and pelletized in a dry N_2 -purged glove bag.

X-ray Powder Diffraction. The mineral powders resulting from MicroMill processing are analyzed in a Phillips PW-1710 automated powder diffractometer, to

determine whether samples have been affected by the grinding process. The analysis is done on a large quantity of grains (since they are of micron sizes). The resulting identity is a perfect representation of the standard material produced to this point.

Size Analysis. Each powder is imaged in a Hitachi S-4700 field-emission scanning electron microscope (FE-SEM). Samples are mounted on conductive carbon tape in a way that avoids size selectivity. Stokes-precipitation sorting and SEM analysis at UCF yield size peaks at $\sim 2\mu m$, with 90% below $4\mu m$, prior to spectra measurements. From sample powder images, the distribution of grain sizes in each sample is determined. These images are also documented in the digital database. Samples that are too coarse are ground again.

Future Directions: This database will become available in a searchable web-based format using open source languages, as portions are published (e.g., Brusentsova et al., *American Mineralogist*, in review). We have a strong interest in extending the scope of our research, to explore a wider range of grain sizes, mineral combinations, spectral wavelengths, and temperature effects. This would extend the application of the mineral library to spectroscopic techniques useful in determining the mineralogy of materials observed by planetary missions, such as MESSENGER, Dawn, and LRO. Beyond transmission infrared laboratory astrophysics, the AMNH mineral library for spectroscopic standards is a new resource for reflectance infrared laboratory planetary astronomy and remote sensing, applicable to planetary, asteroidal, and lunar missions. The AMNH is committed to curation of these standards and accompanying data (including spectra), as building blocks of the infrastructure of present and future discovery.

References: [1] Brownlee D. et al. (2006) *Science* 314: 1711-1717. [2] Zolensky M.E. et al. (2006) *Science* 314: 1735-1739. [3] L. P. Keller et al (2006), *Science*, 314, 1728-1731. [4] C. M. Lisse et al (2006), *Science*, 313, 635. [5] Bell J.F. III (2002) *Icarus* 155, 119-144. [6] Ebel D. S. (2000), *J. Geophysical Res.* (Space Physics), 105, 10363-10370. [7] Ebel D. S. (2006) In *Meteorites and the Early Solar System II* (D. Lauretta et al., eds.) U.Arizona, p253-277, plates 7-10. [8] Coleman P. B. (1993), *Practical Sampling Techniques for Infrared Analysis* (CRC, New York).

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