A NEW STANDARD FOR CALIBRATION OF HIGH TEMPERATURE EMISSIVITY: LABORATORY INTERCALIBRATION AT PEL OF DLR AND ALEC OF BROWN UNIVERSITY. A. Maturilli[1], K. L. Donaldson Hanna[2], J. Helbert[1], C. P. Pieters[2], [1]Institute for Planetary Research, German Aerospace Center DLR, (Rutherfordstr. 2, 12489 Berlin-Adlershof, Germany, alessandro.maturilli@dlr.de), [2]Department of Geological Sciences, Brown University, Providence, RI 02912, USA.

Introduction: Emissivity or emittance is a non directly measurable characteristic of each material. There are several methods to derive it, using a direct or an indirect formula for retrieval. Whichever method is used, a reference body (often called a "blackbody" in the thermal infrared range) needs to be measured. Many good, black paints are available on the market, most have peak emissivity values around 0.99 with little variation across the infrared spectral range (1 to 100 µm) we usually observe. Unfortunately, these paints are chemical products that can not withstand temperatures around 600 K for more than few minutes.

At the Planetary Emissivity Laboratory (PEL) of the German Aerospace Center (DLR) in Berlin, we have spent the last few years investigating materials that could act as a good "blackbody" under challeging conditions. We need a reference body that: (1) has a very high emissivity, (2) has an emissivity spectrum nearly flat across the 1 to 100 µm spectral region, (3) does not outgas and its emissivity spectrum does not change in shape when heated in vacuum above 700K. Such high temperatures are needed to simulate the Mercury surface conditions and to get a good signal close to 1 µm, where Venus' atmosphere is transparent to the planet's emitted radiation. The only material fulfilling these criteria is blast furnace slag, a residue from metal production. We present in this paper a joint calibration/evaluation campaign between the PEL and RELAB laboratories to define the spectral characteristics of 2 blast furnace slags.

The PEL set-up: The PEL is equipped with a Bruker Vertex 80V instrument, coupled to an evacuable high temperature emissivity chamber and an older Bruker IFS 88 attached to a purged low/moderate temperature emissivity chamber. The two instruments can work independently and in parallel as they do not share any crucial device [1, 2].

The Bruker Vertex 80V FTIR spectrometer itself can be operated under vacuum conditions to remove atmospheric features from the spectra. To cover the spectral range from 1 to 100 µm, a liquid-nitrogen cooled MCT (1-16 µm) and a room temperature DTGS (16-100 µm) and two beamsplitter, a KBr and a Mylar Multilayer, are used. However, the system DTGS+Multilayer is usually operated under its full capability, since it allows to measure spectra until 300 µm. The spectrometer is coupled to a planetary simulation chamber, that can be evacuated so that the full optical path from the sample to the detector is free of any influence by atmospheric gases. The chamber has an automatic sample transport system which allows maintaining the vacuum while changing the samples, and an heating system, based on the principle of electromegnetic induction, allowing to heat up the samples to temperatures above 1000 K in minutes [3].

The same two FTIR spectrometers are used for complimentary biconical reflectance and transmittance measurements across the 0.4 to 100 µm spectral range.

The ALEC set-up: The Asteroid and Lunar Environment Chamber (ALEC) is the latest addition to Brown University's Reflectance Experiment Laboratory (RELAB) [4]. ALEC is a vacuum chamber that can reach pressures of $<10^{-4}$ mbar and has an internal rotation stage that enables the measurement of six samples and two blackbodies without breaking vacuum [5]. Sample cups can be heated in one of three configurations: (1) from below using heaters embedded in the base of the sample cup, (2) from above using a halogen radiant heat source through a quartz window to replicate solar-style heating, or (3) both from below and above. Both sample cups and blackbodies are monitored by temperature controllers allowing accurate temperature control and monitoring. ALEC is attached to RELAB's Thermo Nicolet Nexus 870 Fourier Transform Infrared (FTIR) spectrometer through a potassium bromide (KBr) emission port window. A KBr beamsplitter and a deuterated triglycine sulfate and thermoelectrically cooled (DTGS-TEC) detector allow collection of laboratory emissivity spectra at a resolution of 2 cm^{-1} over the 400 - 7400 cm^{-1} (1.4 – 25 µm) spectral range. The spectrometer is purged with dry air to remove particulates, water vapor, and CO_2. Additionally, the same dry air line can be used to backfill the environment chamber to any desired pressure.

Experiment description: The goal of this joint experiment was to characterize two slag materials (both 63 – 125 µm particle size fractions) as reference bodies and to check their influences on similar measurements taken at the two laboratories. In addition to the two slag samples, a bytownite sample of the 0-25 µm particle size fraction from RELAB and the PlanetX sample (a mixture of minerals simulating Mercury's surface of the 0-25 µm particle size fraction) from PEL were measured. Reflectance spectra of all the materials have been measured in both labs. Furthermore, all the materials have been measured in a purged atmosphere

(1000 mbar) and in vacuum (1 mbar) at 375 and 405 K in both laboratories. Reflectance measurements of the two slags were converted to emittance using the formula E=1-R (Kirchhoff's law). Emissivities of bytownite and PlanetX have been calibrated against the two slags, correcting for the retrieved emissivity spectra. Figure 1 shows the reflectance spectra of each slag material measured in PEL and ALEC. Since the two slags come from PEL, we used their ID number to distinguish them. A 0.1 offset has been added to sample 000001203 for better visualization.

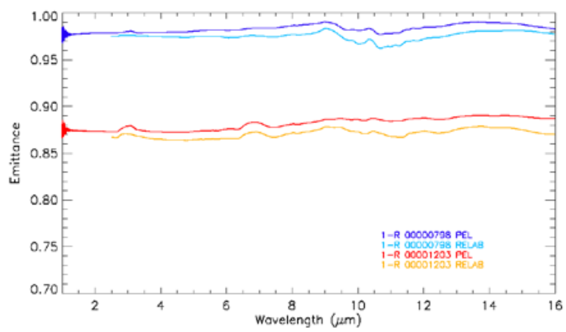

Figure 1. Reflectance spectra of the two slags taken at PEL and in ALEC, sample 00001203 is offset for clarity.

Both slags have very flat spectra, peaking at 0.99 emissivity and do not have deep absorption bands; however their spectra look slightly different and it is important to record those peculiarities for a better calibration of emission data. It is worth to note that both labaratories delivered very close results (within 1% difference), the small discrepancies between spectra may arise from slight differences in sample preparation, illumination angles and/or viewing geometry.

A bytownite sample (PEL ID 00000777) was provided from Brown University as a comparison material. It was measured at PEL and ALEC at 1000 mbar (under purged atmosphere) at 375 and 405 K (100 and 135 °C). The two slags were measured under exactly the same conditions, so that emissivity could be calibrated with either slag sample. Figure 2 show the results of this experiment. The ALEC spectra clearly show that both slags provide the same excellent quality for calibration and the difference between the two slags is indistinguishable in the resultant spectra. At 1000 mbar the results from the 2 laboratories are very similar, however below 8 μm they start to diverge. This could be due to different sensitivities for the 2 detectors, different radiative contribution from the surroundings, or difference in sample cup depths. The slight trend in slope between 8 and 16 μm can be easily explained with small differences in the temperature the different samples reached.

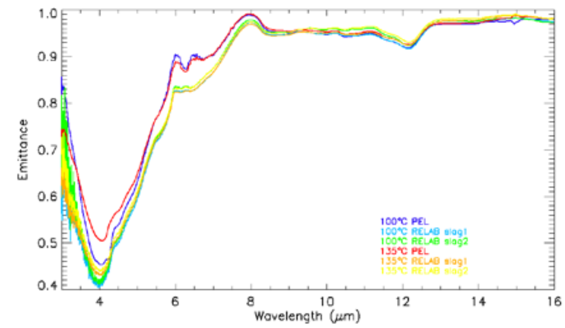

Figure 2. Emissivity spectra of a bytownite sample measured at PEL and in ALEC, taken at 1000 mbar for two temperatures.

Measurements in vacuum (1 mbar) for a PlanetX sample (PEL ID 00000779) at 375 and 405 K were simultaneously measured at PEL and ALEC. The larger differences in the results (also they departure from the ideal 1-R case) can be explained from the thermal gradients produced in the cups used in the different laboratories. These results highlight how important is to proceed investigating in this field as well as the importance for cross-laboratory studies. Below 5 μm the PEL spectra show a "fake" drastic increase in emissivity, arising from a huge raise of the contribution from cup bottom or ring.

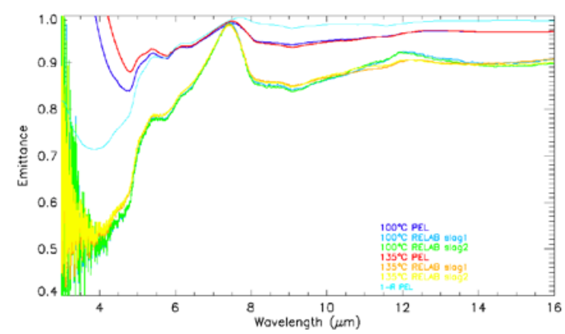

Figure 3. Emissivity spectra of a PlanetX sample measured at PEL and ALEC, taken at 1 mbar for two temperatures.

Summary: The slag samples under examination at PEL and RELAB are very good reference bodies for emissivity measurements. At room pressure both labs produce very comparable results on reference materials. Under vacuum (1 mbar) conditions, the differences in laboratory set-ups may produce very different calibrated emissivity spectra.

References: [1] A. Maturilli, J. Helbert et al. (2006), *PSS 54*, 1057–1064. [2] A. Maturilli, J. Helbert, et al. (2008), *PSS 56*, 420–425. [3] Helbert, J. and Maturilli, A. (2009), *EPSL 285*, 347–354. [4] Pieters C. M. and Hiroi T. (2004) *LPS XXXV*, Abstract #1720. [5] K. L. Donaldson Hanna, et al. (2012), *LPS XLIII*, Abstract #2241.