Preliminary report on LBNL 2011 inter-laboratory comparison for laboratories submitting specular data to the IGDB

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1 Introduction

Laboratories that submit data to the International Glazings Database (IGDB) have to participate in an inter-laboratory comparison (ILC) every four years. This a procedure that allow both contributors and database maintainers to confirm that the measurement capabilities of the laboratories are of high quality. All laminate and applied film samples are manufactured using the same batch of clear glass to allow for an investigation of the accuracy in the Optics 5 laminate deconstruction process.

The IGDB contains optical information in the wavelength region between 300-2500 nm where transmittance as well as reflectance for both the front and the back surface is recorded. In addition to that emissivity, obtained through measurement of reflectance between 5 and 25 μm, is recorded for both the front and back surface.

The goal for submitters is to pass within the tolerances dictaded by NFRC document 302 which states that transmittances should be within 1% and reflectance/-emissivity withing 2%. As an organizing entity LBNL aims to educate and help submitters troubleshoot any issues that give rise to systematic errors.

The ILC is a living ILC and does not necessarily contain the first result submitted by a lab. As errors are found submitters are encouraged to correct procedures or update equipment so that they are allowed to submit data to the IGDB. The risk of this practice is that if any of the recommended solutions introduces new systematic errors that will start to influence the average. Therefore this report tries to highlight the recommendations made so that they can be challenged.

This preliminary report only includes submissions received before September 26th, a total of 29 instruments used (some participants have multiple instruments they use to characterize the same box).

2 Samples

The ILC was a parallel ILC, i.e. all participants gets their own set of samples. This has proven valuable in the past for the participants since they can go back and remeasure their samples after e.g moving or modifying their measurement equipment.

2.1 Selection committee

Mike Rubin organized a sample selection committee consisting of Dave Hasins, PPG; Jordan Lagerman, Cardinal; Jason Theios, Guardian; Bob Curtin, AGC; Dave Duly, NSG; Dan Wacek, Viracon; Raghu Padiyath, 3M; Brija Nand, Southwall; Julia Schimmelpenningh, Solutia.

2.2 Specular sample selection

A total of five samples were selected. PPG produced a clear low-iron glass as well as said glass coated with a low-e coating. All glass used in the ILC was taken from the same production run. Solutia created laminates using the uncoated and coated samples. 3M applied a reflecting film to to the clear substrate for the final sample.

1. 6 mm Starphire
2. 6 mm Starphire coated with triple silver Solarban 70XL
3. 2 pieces of sample 1 laminated with Solutia Saflex 0.76 mm R series PVB.
4. Sample 1 and sample 2 laminated with Solutia Saflex 0.76 mm R series PVB.

5. Sample 1 with applied film

A total of 48 boxes were sent out in the initial round, another 52 were kept at LBNL to allow for inclusion of laboratories to submit to the IGDB.

2.3 Sample variation

Transmittance measurements of each sample was carried out at 550 nm to give an indication of the sample variation. The variation from the average is shown in figure 1. The uncoated samples had the lowest variation but very few samples were within 0.5% absolute of the mean value.

The measured variation was as obtained at LBNL, the samples where packaged, shipped, and cleaned by the recipient before they measured it with their instrument.

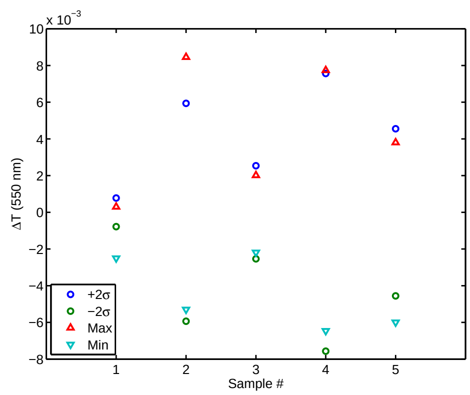

Figure 1: Statistics of the absolute variation of transmittance measured at 550 nm for the five different samples.

3 Solar optical range, 300–2500 nm

3.1 Instruments and detectors used

A clear majority of the submitters use Perkin-Elmer Lambda 900/950 instruments fitted with a 150 mm integrating sphere. The low number of other instrument types limits the ability to draw conclusions from the results. A breakdown is shown in figure 2 on the next pagea).

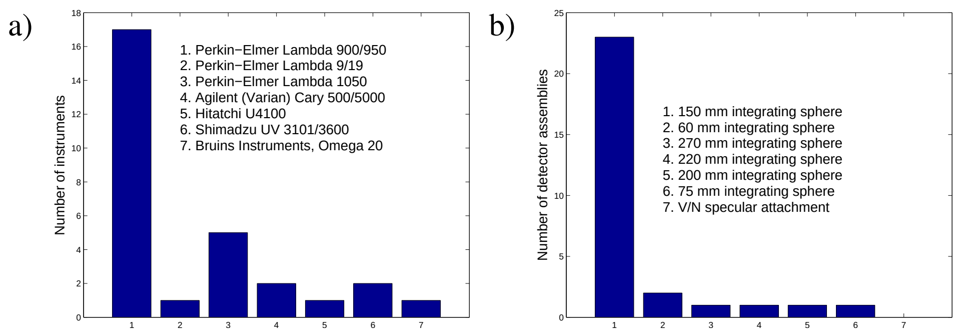

Figure 2: a)Distribution of instruments among the participants. b) Distribution of detector systems used.

The typical detector combination is a photo multiplier tube (PMT) for the visible range and a lead sulfide (PbS) detector for the NIR. The Lambda 1050 instruments feature and indium gallium arsenide (InGaAs) detector instead. All participants had an integrating sphere, the diameter distribution is shown in figure 2b).

3.2 Example of results

This preliminary report does not have graphs of all results, but an example is shown in figure 3 on the next page. Even the two clear outliers are within the 2% tolerance dictated by NFRC. They do not however pass the requirement to be physical, the high reflectance is above 1, or contain absorption artefacts (one result has been multiplied with the reflectance of Spectralon).

3.3 Example of corrected mistakes

This section highlights some of the more confounding problems that show up repeatedly but can be hard to replicate on different instruments.

3.3.1 Discontinuity at grating change

These spectrophotometers are built to cover two wavelength ranges and mechanical alignment of detectors, gratings, and light sources is an engineering problem that is part of the challenge of building these intstruments.

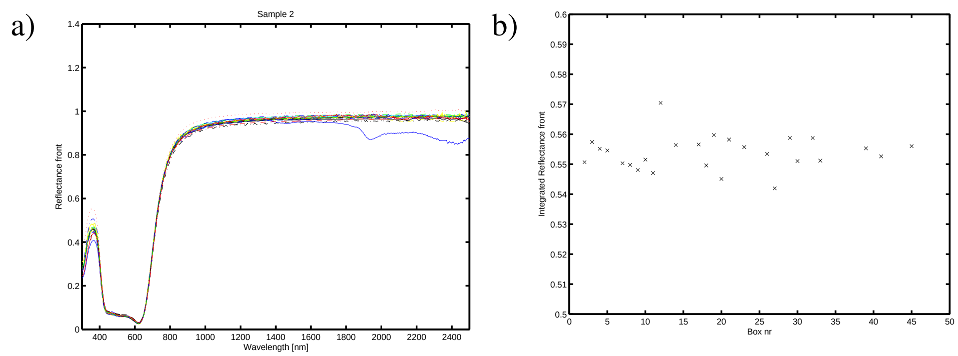

Figure 3: a) Film-side reflectance of sample #2. b) Integrated solar value for the data shown in a).

Example of a couple of different instrument results are shown in figure 4 on the following pagea) A step of .02 indicates that you have no room for sample variation if you want to stay within .02 tolerance. Smaller steps are unsightly and could create problems for calculation of optical constants or when deconstructing an applied film or a laminate.

The step shown in figure 4 on the next pageb) was reduced by using a fixed slit width in NIR rather than the default servo setting. It also mattered what the ratio of slit width between the two gratings, best results were obtained when the ratio matched the ratio between the number of grooves per mm for the gratings. This keeps the light spot the same size.

The gratings also have a strong polarizing effect, if the instrument is not fitted with a depolarizer and the sample is polarized there is a possibility that there will be a discontinuity here as well.

3.3.2 Absorption artefacts in NIR

Sample #2 has an exposed metal coating that is highly reflective in NIR. The flat shape of the reflectance for the coated side makes it easy to spot any absorption artefacts in that range. An example of the effect is shown in figure 5 on page 7 from a metal coated sample used in the ILC 2007, sample #2 in this ILC has similar properties but very few submissions showed this effect so far this year.

It is hard to repeat this effect but a theory for how this happens is suggested. The submissions in figure 5 on page 7 all used a diffuse reference and a Spectralon integrating sphere. In theory this should give the reflectance value assuming the

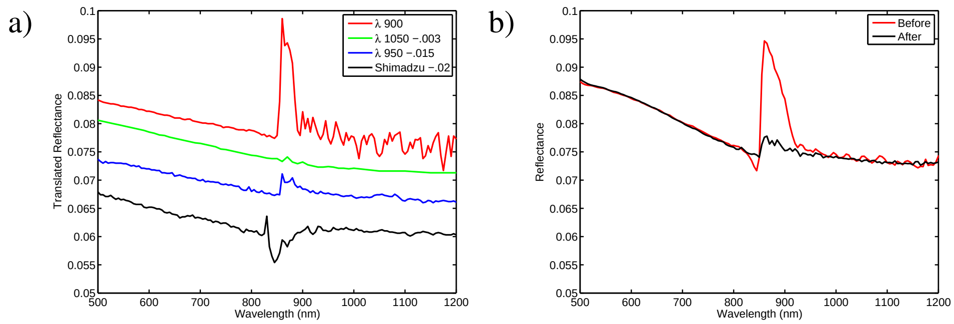

Figure 4: a) Example of different glass reflectance measurement of sample #1, values have been shifted laterally to clearer show the discontinuities. b) Example from a measurement in the ILC conducted in 2007.

detector response is the same for light incident on the reference sample and the specular port[1]. These two sphere locations are both baffled and not directly in the detector field of view and in those cases the most plausible explanation would be that the reference and the port have degraded differently. Some submitters tried to clean their reference samples but without any improvement. The only way they could get accurate results was to use a specular reference mirror.

3.3.3 Inter-reflections between sample and optical system not controlled

When inserting the sample for transmittance measurement the sample reflectance will interact with the optical system generating the beam (such as lenses and polarizers).

Some commercial instruments, e.g. the Lambda 950, have a ”sample compartment” where the sample can be mounted in a chamber with small quartz windows a fair distance from the samples. If the sample is inserted at normal or very close to normal angle of incidence the reflection from the sample will be reflected from the first quartz window and add to the total recorded transmittance. In theory this can occur with the sample at the sphere port as well if there optical system is perfectly aligned.

The magnitude if this error can be calculated and is not very large, but it becomes noticable when comparing highly transparent glass.

[1]It is common, but not necessary that an integrating sphere has a specular port, if none is present it is the sphere wall at the spot where the specular reflection first interacts with the sphere that has to have the same detector response as the reference sample

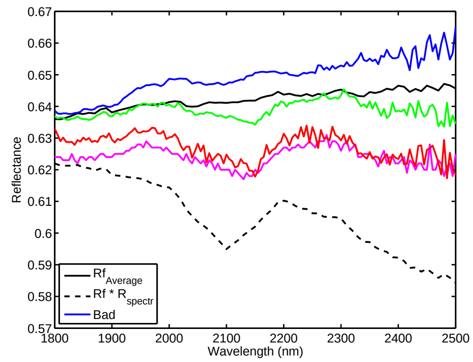

Figure 5: Average reflectance of a metal coated glass subtrate and that value multiplied with the reflectance of Spectralon contrasted against submissions with absorption artefacts.

4 Infrared range, 5–25μm

4.1 Instruments used

The IR instrument market is more diverse the solar optical instrument market and that shows in the range of instruments used.

4.2 Example of results

Measured reflectance for sample #2 is shown in figure 6 on page 9. For the glass part it is clear that some submitters fail to resolve the peak at 9.5μm due to a step that was larger than the dictated at least one value per μm.

The film side measurements show some agreement around .98, but there is a significant spread. The outlier at .96 has some step at 15μm that is not seen in their measurement of the glass side. Further investigation has to be carried out to

5 List of Participants

An autogenerated list based on the submitters information in the *boxXXinfo.txt* that was included in the submission is show in table 1. The list is not sorted by box number but it is not organized to avoid that occuring through coinicence.

Institute	**Contact**
AGC Glass Company North America	BOb Curtin
AGC Glass Japan/Asia Pacific	Sigetosi Hirasima
AGC glass Europe	Ingrid Marenne
Berlin Institute of Technology	Stefan Gramm
CEPT, Navrangpura, Ahmedabad.	Dr. Vinod Patel
CSG Holding Co., Ltd.	Chengde Huang
China Bluilding Material Test & Certification Center (ctc)	Wu,Jie
Euroglas	Martin Daams
Fraunhofer Institute for Solar Energy Systems	Helen Rose Wilson
HanGlas Gunsan R&D Centre	Choi, Junbo
INTERPANE Entwicklungs- und Beratungsesellschaft	Karl Häuser
Lawrence Berkeley National Laboratory	Jacob C. Jonsson
Madico, Inc	Andy Hayes
Madico, Inc., St. Petersburg, FL	David Harney
Optical Data Associates, LLC	Michael R Jacobson
PFG Building Glass	Rahab Bopape
PPG Industries	Nathaniel Hazelton Dave Haskins
Pilkington Weiherhammer Laboratory	Dr. Joachim Bretschneider
SHANGHAI YAOHUA PILKINGTON GLASS CO.,LTD	Sun Dahai
Saint-Gobain Glass CRDC	Michel PICHON
Solar Energy Research Institute of Singapore (SERIS)	Teo Wei-Boon
Solutia Perforamnce Films	Beth Lawless-Coale
Southwall Technologies	Brija Nand
Stazione Sperimentale del Vetro	Antonio Daneo
Viracon	Dan Wacek
arcon Flachglas-Veredlung GmbH & Co.KG	Carsten Ruppe

Table 1: Autogenerated table from what participant wrote in the boxnninfo.txt file. Not listed in box number order.

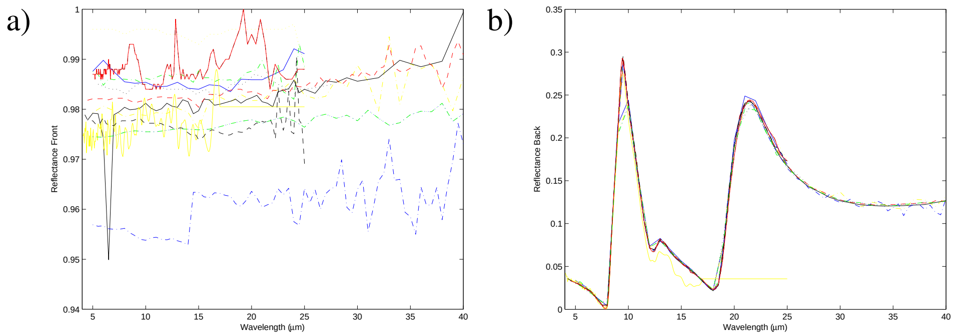

Figure 6: a) Reflectance of the metal coating of sample #2 b) Reflectance of the glass side of sample #2.

6 Conclusions

Based on this preliminary report it is clear that the state of submitters is in general very healthy, almost all measurements are within the tolerances set by NFRC.

It is the ambition of LBNL to work with ISO and ASTM standards groups to improve on the language in standards to make it easier for new submitters to find information in the right place on how to carry out good measurements, and if possible prove that the tolerances could be decreased.

7 Future work

The final report is expected to be presented at the end of October 2011. Investigations of the infrared reflectance measurements will be conducted and a full presentation of the integrated results, such as show in figure 3 on page 5b) will be included. Please contact the author with further comments and questions that you would like to have answered.