

LBL 2015 inter-laboratory
comparison for laboratories submitting
specular data to the IGDB

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Abstract

This report covers the process and result of the LBNL run inter-laboratory comparison (ILC) between measurement laboratories characterizing the solar optical properties of glass and glazing products. An important part of this process is to educate and improve the results from laboratories that suffer from systematic errors in their results. Collecting both common and rare problems in this document is meant to serve as help for troubled spectroscopists.

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 is a procedure that allow both contributors and database maintainers to confirm that the measurement capabilities of the laboratories are of high quality.

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, emissivity values for both the front and back surface are obtained through measurement of reflectance between 5 μm and 25 μm .

The goal for submitters is to pass within the tolerances dictated by NFRC document 302 which states that transmittances should be within 1% and reflectance/emissivity within 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 this will start to influence the average. Therefore this report tries to highlight the recommendations made so that they can be challenged.

2 Samples

The ILC is a parallel ILC, i.e. all participants get 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 moving or modifying their measurement equipment.

2.1 Specular sample selection

A total of three samples were selected from three companies, Viracon, Eastman, and Guardian. A clear glass is stable and should have minimum sample variation and be stable over time, a thick laminate was included this year to verify measurement of samples in the 12 mm range, and a low-e coating is used to look at reflectance variations and emissivity. To summarize:

1. 6 mm clear monolithic glass supplied by Viracon
2. Safelex SG41 Solar laminate on 6 mm clear substrates supplied by Eastman
3. Double silver low-e on 6 mm clear supplied by Guardian

In the 2011 ILC approximately 45 boxes were sent out initially and the total number grew to 70 over the 4 years. For the 2015 we prepared 80 boxes and send out 48 boxes initially. The remaining boxes are kept at LBNL for future inclusion of participants.

2.2 Sample variation

Transmittance measurements of each sample were carried out at 550 nm to give an indication of the sample variation, this was done at LBNL before samples were shipped out. The transmittance was measured for 20 seconds with the signal sampled every second, typical variation in reading over 20 seconds was ± 0.0002 . The difference between samples and the average was calculated by subtracting the mean from each measured value. The extreme values as well as two times the standard deviation is shown in figure 1.

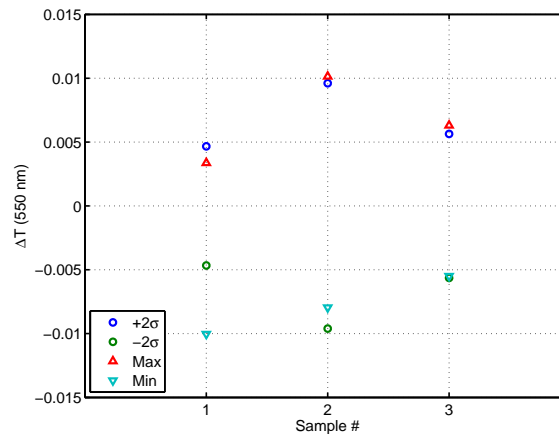


Figure 1: Statistics of the absolute variation of transmittance measured at 550 nm for the different samples.

The variation of the glass samples were small. All in all, more than 90% of the samples were within 0.005 absolute difference from the mean value. The outliers among the low-e coatings is believed to be due to defects from shipping and handling the samples. The information about the premeasured value is not shared with each box or used to scale the result submitted from the participants. It is instead seen as a test to verify that the product is not something completely different and as a measure of if the tolerance set by NFRC can handle minor sample variations as well as measurement variations. The conclusion from looking at this data is that it is of little benefit to force the ILC participants to measure more than one sample.

After the variation had been measured at LBNL, the samples were packaged, shipped, and upon reception cleaned by the recipient before they measured it with their instrument.

3 Solar optical range, 300–2500 nm

Graphs with all the results are presented in details in Appendix B. The coated sample yielded initial results that were outside the tolerance for two submitter which led to a modification of their process which resulted in agreement with the average.

3.1 Instruments and detectors used

A majority of the ILC participants used Perkin-Elmer Lambda 900/950/1050 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 2a). Instruments with zero occurrences were included in the graph for one of two reasons, either it was present in a past ILC or its user did not submit data before this report was finalized. This is not an attempt to list all possible instruments available.

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 an indium gallium arsenide (InGaAs) detector instead. All participants had an integrating sphere, the diameter distribution is shown in figure 2b).

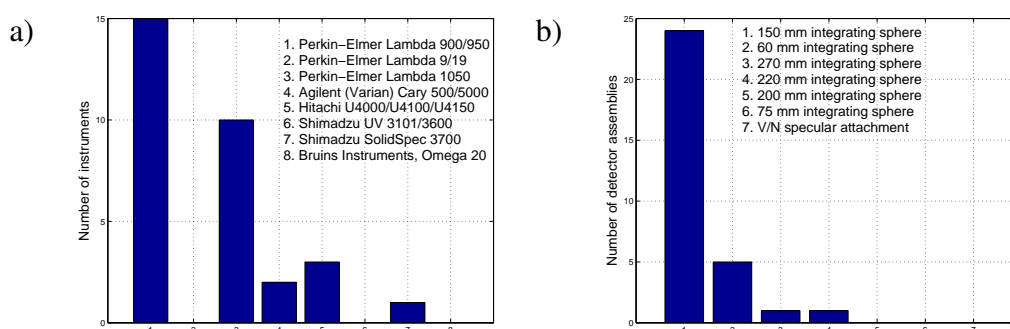


Figure 2: a) Distribution of instruments among the participants. b) Distribution of detector systems used.

With such a dominance of a few detector systems and instruments it is impossible to confidently say that the other instruments and detectors are performing better or worse. No error was tied to a single brand or detector type.

3.2 Past and recent outcomes

This section collects past and present conclusions that can be shown using data sets of this type. The goal is to explain some of the decisions made regarding to the procedures and be instructional about how different measurement methods might influence the result. This section also collects information and conclusions reached in past ILCs to contain it in the latest document and update it with the current state of measurement rules.

3.2.1 Effects of large wavelength steps when measuring applied films

LBNL used to allow steps up to 50 nm for data at wavelengths longer than 1000 nm. The consequences of using the longest step length is shown in figure 3b); with very narrow interference fringes it is more or less random what value is reported in the range from high to low.

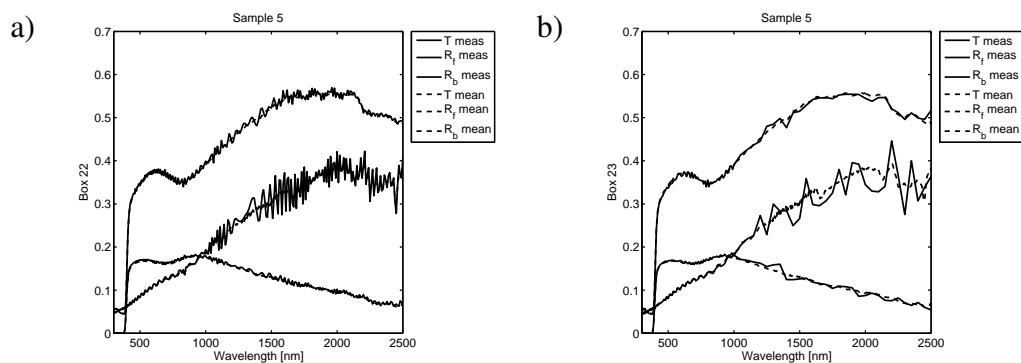


Figure 3: Data for the 2011 applied film sample. a) Data presented with 5 nm steps. b) Data presented with 50 nm steps.

There are two ways to avoid this, the practical way is to measure at shorter steps, as shown in figure 3a), which makes it less probable that streaks of high or low values will skew the integrated values.

The second way is to adjust the bandwidth of light used to illuminate the sample. The grating of a spectrophotometer in practice produces a distribution of wavelengths and the bandwidth of this is controlled by a slit in the optical system. This will create an average over multiple wavelengths which creates a smoother curve. While not an accurate representation of the interference fringes, it will produce accurate results for integrated values.

3.2.2 Diffuse versus specular reference

Integrating sphere theory suggests that using a diffuse reference sample of the same material as the sphere wall will give you an absolute reflectance measurement for specular samples. This requires that the detector response is identical for light incident on the specular port and the reflectance sample position. Since commercial integrating spheres are not ideal spheres it is not obvious that it would give the same result as when using a specular reference mirror. Data from this ILC can be used to compare results using diffuse standards, first surface mirrors, and second surface mirrors.

The specular mirrors have been divided between first, or front, surface mirrors and second surface mirrors. For the first surface mirrors the mirror film is exposed to air and will be in direct contact with the instrument. Even though some of these mirrors are protected with a surface coating they are sensitive to scratching which can occur when mounting against the sphere wall. The second surface mirrors have the mirror film sealed on the back of a transparent substrate. This protection results in a slightly lower reflectance but makes the mirror less susceptible to degradation.

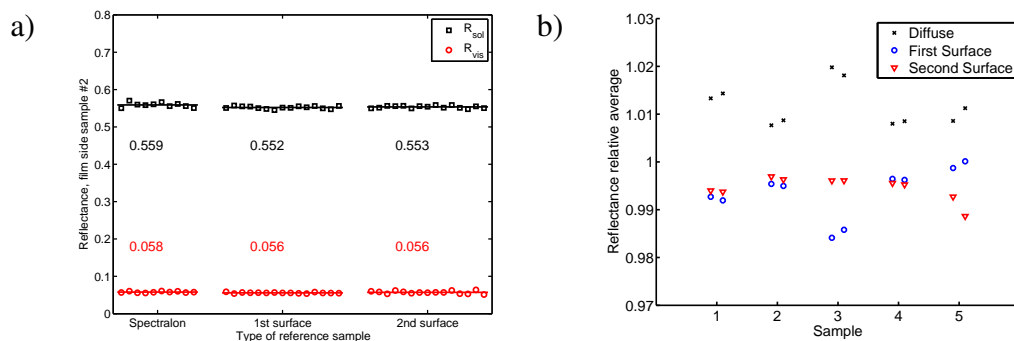


Figure 4: Integrated reflectance grouped for kind of reference sample. The average value for each group is written next to the curve. The diffuse Spectralon group has a slightly higher average than the other two. a) Film-side reflectance of sample #2 showing the individual measurements for each participant. b) Showing the reflectance relative the average for that sample for all measured reflectances. The two values for each sample are front and back reflectance.

The reflectance measured is graphed versus the type of reference used in figure 4. The metal coating of sample #2 is shown in figure 4a) and the solar reflectance is slightly higher, about .005 or 1% relative, on average but the visible reflectance is seemingly independent of reference sample. In figure 4b) the average of each group is graphed divided by the average for all groups. It shows that for all 10 measured reflectances, counting front and back of the five samples, the data submitted using a Spectralon reference is consistently higher than average and the specular mirrors are lower.

One way to get a value that is too high is if the reference sample has a lower reflectance than it is supposed to. In the case of a specular reference mirror that happens if the surface

has a lower reflectance than its certificate. In the case of a diffuse reference sample it happens if the Spectralon reference has a lower reflectance than the specular port. By lower reflectance in this case it is not only necessary to consider the actual reflectance of the material but also the response from the detector in the integrating sphere. So the sphere geometry coupled with the scattering distribution of the material, both the reference and the specular port, could play a role in any deviation from the true value.

It has been shown that Spectralon reflectance decreases with time even if the material is kept in the dark[1]. One possible hypothesis is that the Spectralon reference deteriorates faster than the specular port due to handling and that this gives rise to a systematically too high measured reflectance. Another possibility is that the detector response is different for light scattered from the specular port and the sample port.

Without a definite way to insure that the Spectralon absorption bands do not start to influence the result, it is highly recommended that a second surface mirror is used.

3.3 Example of corrected results

This section highlights some of the systematic errors that have occurred and suggested methods how to fix them. Some of these show up repeatedly but can be hard to replicate on different instruments.

3.3.1 Misaligned grating

The correlation between the mechanical position of the monochromator diffraction grating and the recorded wavelength of each measurement point has to be calibrated. Typically this is done using a sample with very sharp absorption peaks, e.g. a Holmium Perchlorate solution, or an emission light source, e.g. a Deuterium lamp. The instrument software correlates its reading of the grating position with the known position of the peaks.

The grating usually does not lose its position but, e.g. if it is moved and gets bumped or if dust is building up in the mechanical system, it is a good idea to run a calibration. Some software suggests that you do this with a fixed frequency which is not a bad idea.

There are some ways to spot if the grating is out of alignment without running the calibration. One is to look at sample properties with a significant derivative in grating change region. An example of this is shown in figure 5. This does not tell you which grating is out of alignment, but it signals that something is wrong. More about discontinuities in section 3.3.2. On the other hand, if both are the same amount out of sync there might be no discontinuity so this is not a sufficient test to say that the instrument is aligned, only a way to spot that it is not.

Some Perkin-Elmer instruments have a 0 nm wavelength setting where the grating is parallel to the beam to let it pass through as white light (also called alignment mode). If the calibration is sufficiently off this can result in the grating fully or partially blocking the beam resulting in no visible beam.

3.3.2 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 instruments.

Example of a couple of different instrument results are shown in figure 6a) A step of .02 indicates that you have no room for sample variation if you want to stay within .02

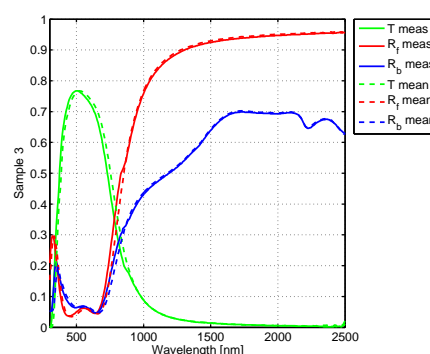


Figure 5: The slopes of the measured data are offset with respect to each other.

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 6b) was reduced by using a fixed slit width in NIR rather than the default servo setting. The ratio of slit width between the two gratings also mattered, 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.

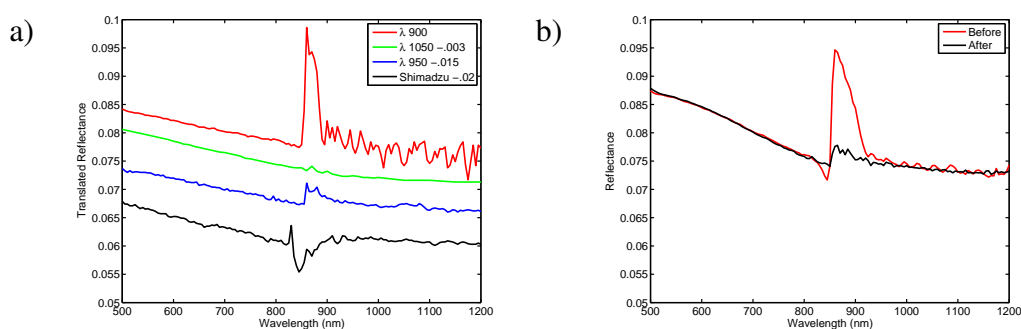


Figure 6: a) Example of different glass reflectance measurement of sample #1, values have been shifted laterally (the legend shows which instrument from top to bottom) to more clearly show the discontinuities for the different samples. b) Example from a measurement in the ILC conducted in 2007 where adjustments almost completely removed the discontinuity.

3.3.3 Absorption artifacts 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 artifacts in that range. An example of the effect is shown in figure 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 which is why it is exemplified using data from 2007.

It is hard to repeat this effect but a theory for how this happens is suggested. The submissions in figure 7 all used a diffuse reference and a Spectralon integrating sphere. In theory this should give the reflectance value assuming the detector response is the same for light incident on the reference sample and the specular port¹. 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.

¹It 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

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.

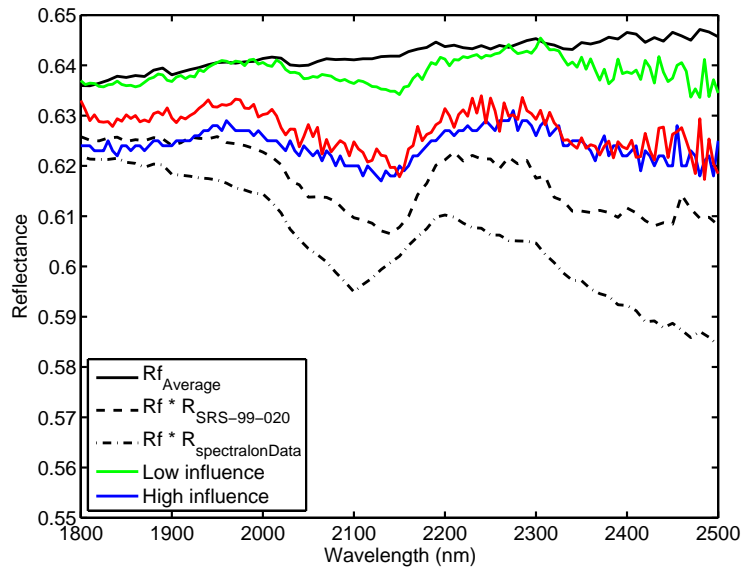


Figure 7: Average reflectance of a metal coated glass substrate and that value multiplied with the reflectance of Spectralon contrasted against submissions with absorption artifacts.

4 Thermal infrared range, 5–25 μm

Graphs with all the results are presented in details in Appendix C. The coated sample yielded initial results that were outside the tolerance for one submitter which led to a modification of their process which made the new result fall into the tolerance.

4.1 Instruments used

The IR instrument market is more diverse than the solar optical instrument market and that is seen in the range of instruments used presented in figure 8. Instruments with zero occurrences were included in the graph for one of two reasons, either it was present in a past ILC or its user did not submit data before this report was finalized. This is not an attempt to list all possible instruments available.

The THERMES project[2, 3, 4] did thorough comparisons between dispersive and FTIR instruments and those have not been repeated here since there were too few dispersive instruments in this data set.

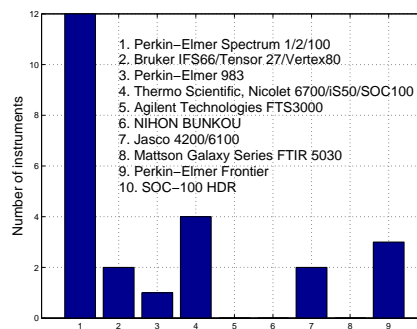


Figure 8: Distribution of instruments used to measure reflectance between 5 μm and 25 μm for calculation of emissivity.

There was a call for submission using emissometer type instruments but only two boxes were measured using those. The results from those two boxes were good but without a larger set of participants it is optimistic to draw any conclusions.

4.2 Emissivity calculations

The IGDB contains information about the emissivity in the infrared range. To obtain this value reflectance is measured and since the samples are opaque in the infrared wavelength region so the absorption is equal to one minus the reflectance. The spectral absorption is weighted using a 300 K black body curve according to NFRC 301[5]. This temperature is the default in the LBNL OPTICS/WINDOW programs. The IGDB allows submissions where the submitter has calculated the emissivity instead of submitting the measured data.

The calculation of emissivity is not always carried out in this way. The European standard EN673[6] uses a temperature of 283K instead of 300K. A room-temperature blackbody emits about 17% of the total energy at longer wavelengths than $25\mu m$, if the region is extended to $40\mu m$ a different value can be obtained for some materials. The difference in calculated emissivity for low-e coatings is very small though as there is next to no variation in reflectance beyond $25\mu m$. The numerical differences are shown in figure 9 for a single data file from this ILC. The reason to not measure beyond $25\mu m$ is purely practical in that for a long time it was impossible to purchase a new IR spectrophotometer that could measure longer wavelengths.

The conclusions to draw is that even though the differences are not large it could lead to rounding differently depending on how the emissivity was calculated.

All the emissivity values are shown in appendix C and in those graphs it is also possible to see which values were submitted spectra and which were submitted as calculated values.

In addition to the choice of black body temperature there is also a transformation from the direct emissivity (which is measured) to the hemispherical emissivity which is the reported property. This is carried out in accordance to NFRC 301[5].

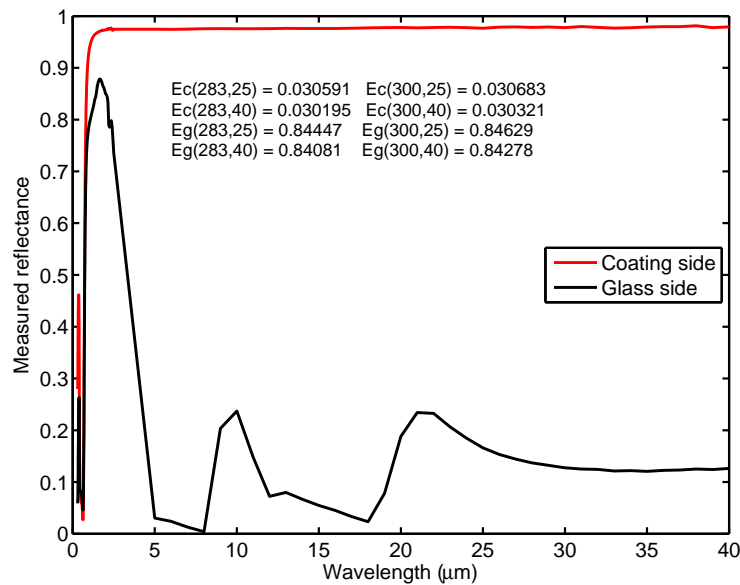


Figure 9: Spectral reflectance measured and hemispherical emissivity calculated for two temperatures, 283 K and 300 K, and using two different upper boundaries for the calculation. The calculation was carried out for both the glass side (E_g -values) and the coated side (E_c -values) of the sample.

4.3 Measurements

Out of the three samples, there was only one surface that was coated glass: the low-e coating of sample 3. By measuring glass emissivity 5 times the laboratories got good information about the instrument repeatability, and possibly drift. An example of such a result is shown in figure 10.

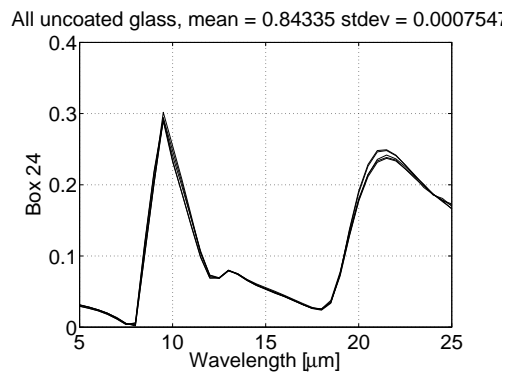


Figure 10: Example of submitter number 24's reflectance measurement of the 5 uncoated glass surfaces shown together in one graph to demonstrate the instrument variation.

In addition to the glass reflectance the low-e coating was graphed individually. Examples of that measurement is shown in figure 11.

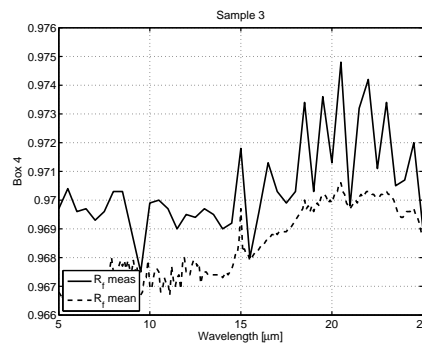


Figure 11: Example of submitted IR reflectance of the low-e coating. $R_f meas$ is the submitter's measured value of the film/coated surface and it is compared to the average of all submitters measured values $R_f mean$. The index f indicates film side.

4.4 Calculation of hemispherical emissivity

A two step process is used to calculate the hemispherical emissivity from the near normal IR reflectance measurement measured.

4.4.1 Calculation of normal emissivity

The normal emissivity is calculated by integrating the measured reflectance, $R(\lambda)$, weighted with the black-body emissivity spectrum of a 300 K body, $E_b(\lambda)$, according to

$$\varepsilon_n = \frac{\int_{5\mu m}^{25\mu m} (1 - R(\lambda)) E_b(\lambda) d\lambda}{\int_{5\mu m}^{25\mu m} E_b(\lambda) d\lambda}, \quad (1)$$

where $E_b(\lambda)$ is calculated according to

$$E_b(\lambda) = \frac{C_1}{\lambda^5 (\varepsilon^{C_2/\lambda T})}, \quad (2)$$

where the emitted black-body radiation, $E_b(\lambda)$, is given by

C_1 Planck's first constant ($3.743 \times 10^8 W \mu m^4 / m^2$)

C_2 Planck's second constant ($1.4387 \times 10^4 m \mu m K$)

T temperature (K)

λ wavelength (μm).

4.4.2 Conversion from normal to hemispherical emissivity

The hemispherical emissivity, rather than the normal emissivity, is the property used in thermal calculations. Rather than measure the hemispherical value it is calculated using empirical expressions[7].

For uncoated substrates the expression is:

$$\varepsilon_h = 0.1569\varepsilon_n + 3.7669\varepsilon_n^2 - 5.4398\varepsilon_n^3 + 2.47333\varepsilon_n^4 \quad (3)$$

where ε_n is the normal emissivity calculated using equation 1.

For coated substrates the expression is:

$$\varepsilon_h = 1.3217\varepsilon_n - 1.8766\varepsilon_n^2 + 4.6586\varepsilon_n^3 - 5.8349\varepsilon_n^4 + 2.7406\varepsilon_n^5. \quad (4)$$

4.4.3 Calculated emissivities for samples 1-3

All calculated emissivity values are presented in appendix C. The average emissivity for the low-e coating was 0.024.

5 Conclusions

This report indicates that the state of the participants measurements is in general very healthy, almost all measurements are within the tolerances set by NFRC.

Results shown in sections 3.3.3 and 3.2.2 indicate that a specular reference mirror is preferred compared to a diffuse reference. Since the method of using a Spectralon reference is theoretically sound and works well for some submitters it is still allowed, however, submitters are strongly advised to switch to using a a calibrated specular mirror instead. This topic has been brought up for discussion in the ASTM E903 committee but not resolved.

No changes to the IGDB submission procedure are suggested as an outcome from this ILC. The current methods give a good agreement between participants and the tolerance set in NFRC 302 is a sensible limit to define outliers.

It is the intent 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.

6 Acknowledgement

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Viracon, Eastman, and Guardian assisted with providing the samples that were used.

All participants contributed significant time in measuring all sample properties.

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Appendix

A List of Participants

Institute	Contact
3M	Raghu Padiyath
Aestec Testing Lab	Victor Sia
AGC Glass Co. North America	Cathy Thomas
AGC Glass Co. Japan/Asia Pacific	Sigetosi Hirasima
Beijing Jinjing Wisdom Co.Ltd	Huang Yali
Cardinal Glass	Jordan Lagerman
Centre For Research and Development Foundation	Yashkumar Shukla
Centro Brasileiro de Eficiência Energética em Edificações	Saulo Guths
China Building Material Test & Certification Center (CTC)	Wu Jie
China Southen Glass Holding Co., Ltd.	Chengde Huang
Eastman Chemical	Gunnar Spitzer
Eastman Chemical	Brija Nand
Eastman Chemical (China) Co.,Ltd	Albert Wang
Eastman Chemical Company	Jeff Skaza
Erickson International	Justin Shjarback
Facade Materials Testing Laboratory, OTM Solutions Pte Ltd	Chen Fangzhi
Fraunhofer Institute for Solar Energy Systems	Helen Rose Wilson
Guardian Europe S.a.r.l.	Jos Linden
Guardian Industries (North America)	Jason Theios
Hankuk Glass Industries Inc.	Dong-young, Park
INTERPANE Entwicklungs- und Beratungsgesellschaft	Karl Häuser
Lawrence Berkeley National Laboratory	Jacob C. Jonsson
Madico Inc.	Jesse Manship
Norwegian University of Science and Technology (NTNU)	Tao Gao
NSG (Pilkington)	James Farmer
NSG R&D Japan	Isao Muraguchi
Optical Data Associates, LLC	Michael R Jacobson
Optics & Radiometry Laboratory, UNSW	Brian Cheng
PFG Building Glass	Rahab Bopape
PPG Industries	Michael Buchanan
PT Asahimas Flat Glass Tbk	Gamal Trio Putra
Pilkington Glass Russia	Oleg Maksyuta
SAGE Electrochromics Inc.	Mark Burdis
Saint Gobain Glass CRDC	Matthieu Milan
Saint-Gobain Solar Gard	Jon Mitchell
Sanam Glass company	Suresh Sam
Sonnergy Limited	Michael G Hutchins
SYP GLASS GROUP CO.,LTD	Sun Dahai

Table 1: Autogenerated table from what participant wrote in the *boxnninfo.txt* file. It is not sorted after box number.

B Graphs for all UV/Vis/NIR measurements

The graphs on following pages all show integrated solar and visible optical properties for each sample. The individual markers (squares and circles) show reported values, dotted lines show plus and minus two times the calculated standard deviation for that property, and finally dashed lines show limits imposed by NFRC 302 (.01 for transmittance and .02 for reflectance).

B.1 Sample #1

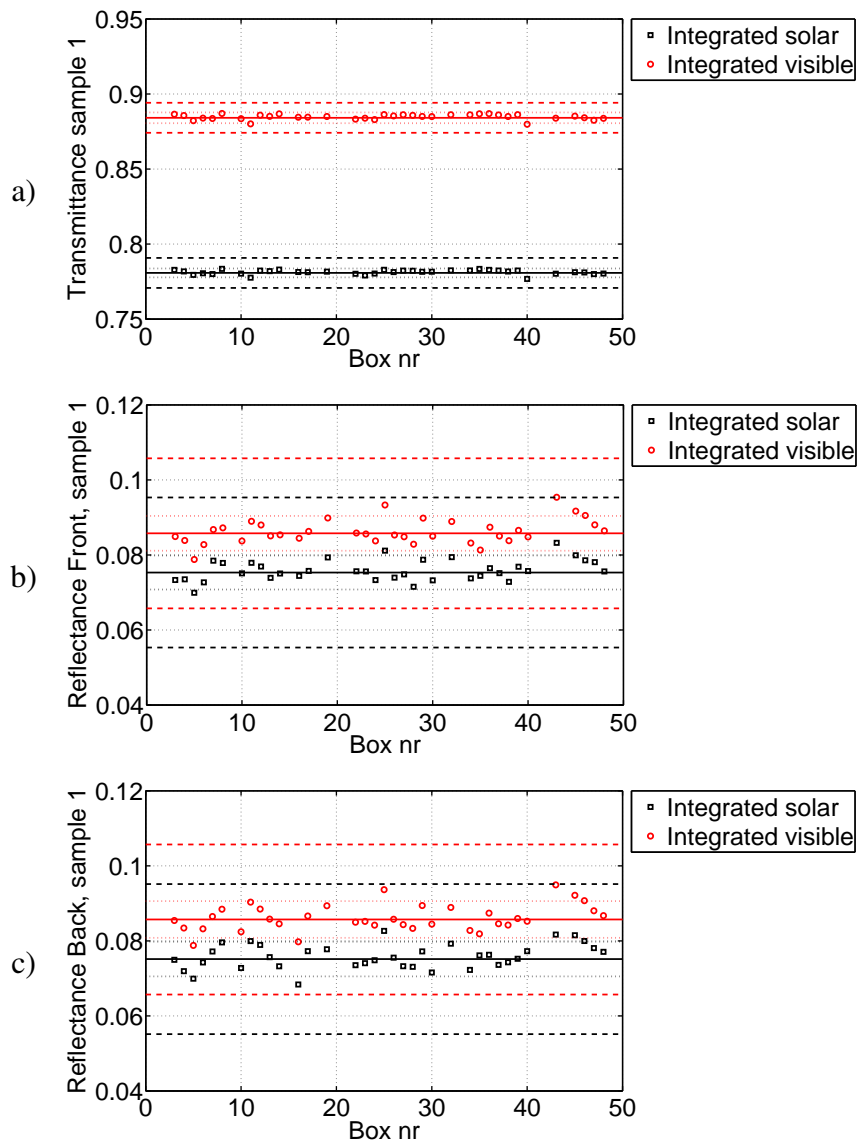


Figure 12: Integrated solar and visible optical properties for sample 1. a) Transmittance, b) front reflectance, and c) back reflectance.

B.2 Sample #2

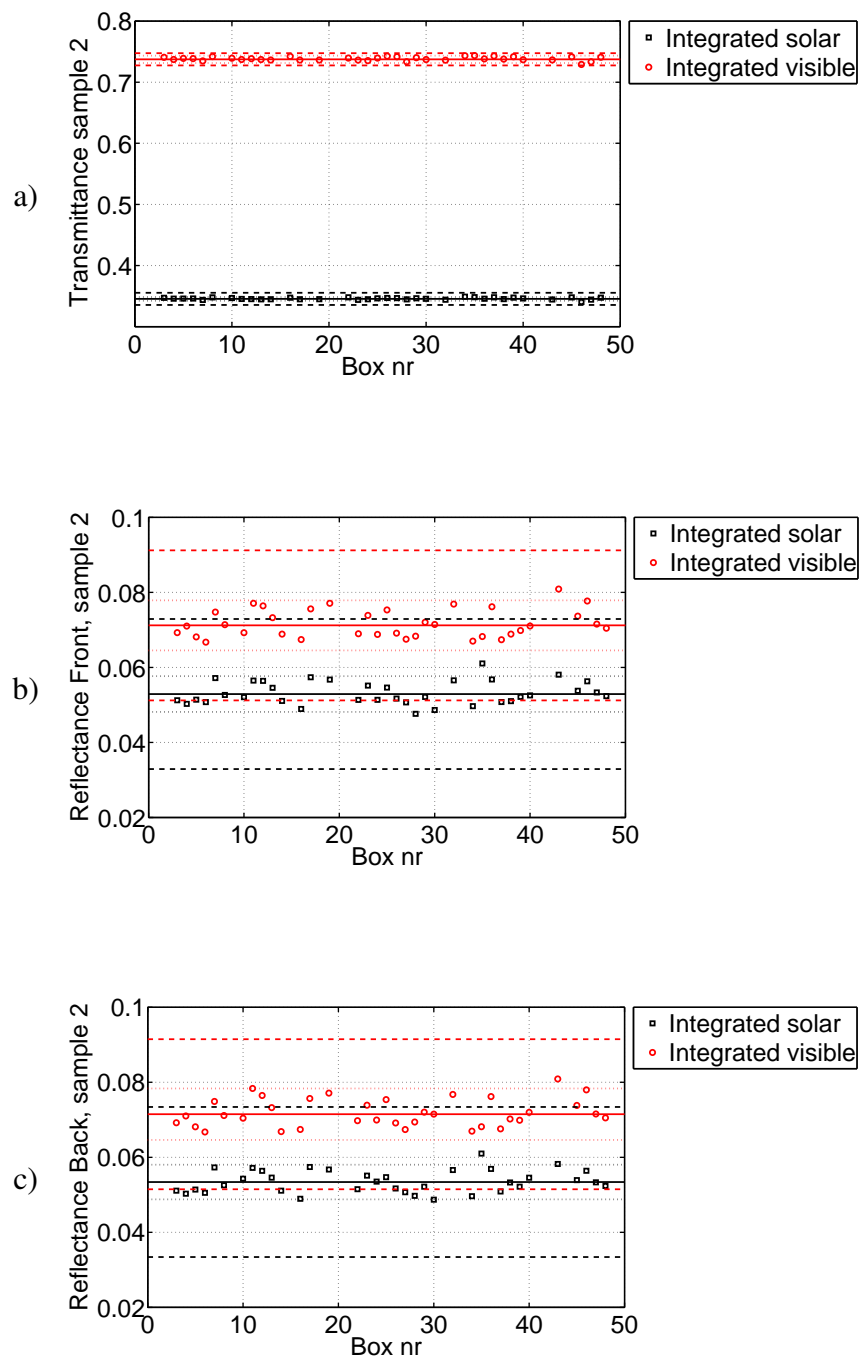


Figure 13: Integrated solar and visible optical properties for sample 2. a) Transmittance, b) front reflectance, and c) back reflectance.

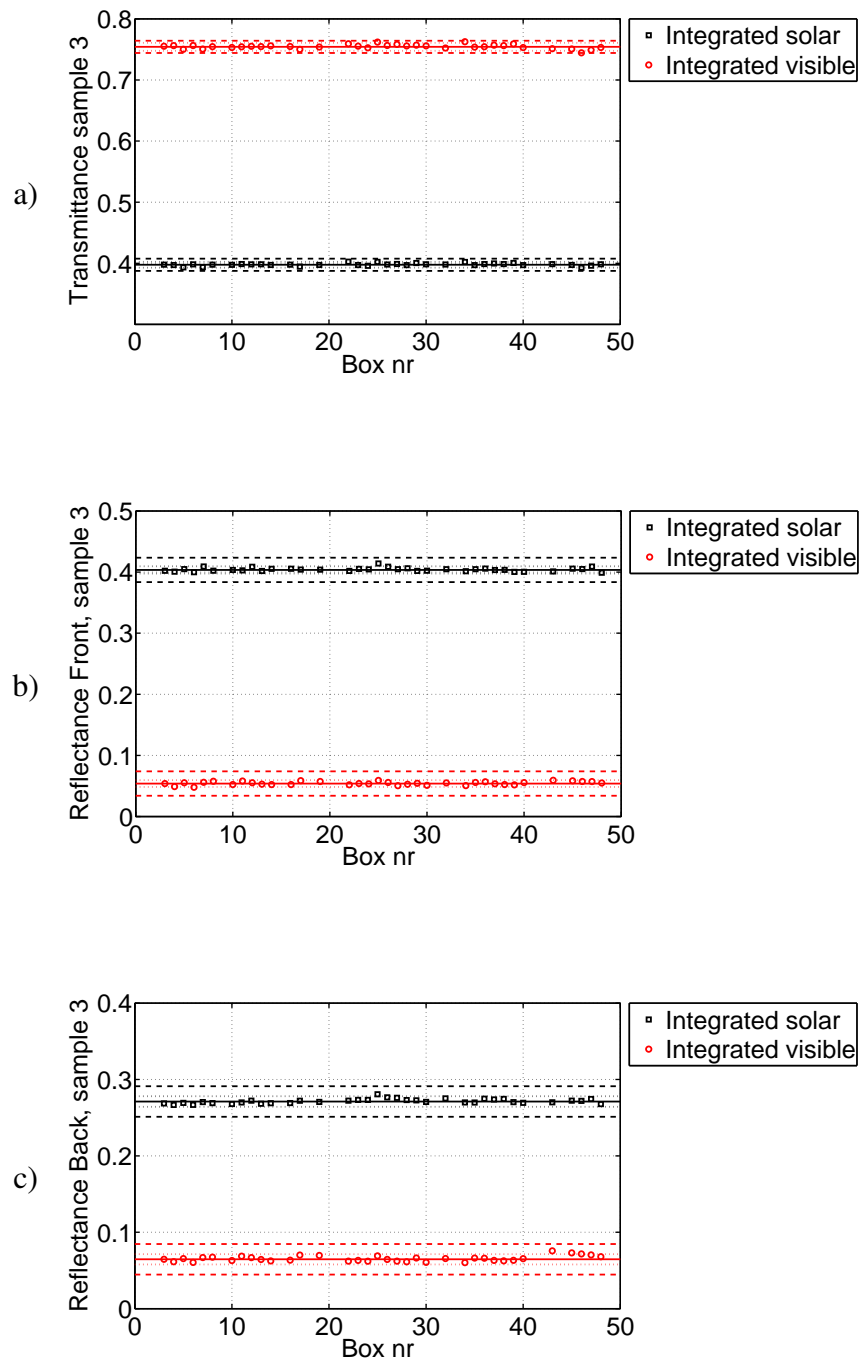
B.3 Sample #3

Figure 14: Integrated solar and visible optical properties for sample 3. a) Transmittance, b) front reflectance, and c) back reflectance.

C Graphs for all IR measurements

The graphs in this section shows the calculated emissivity according to NFRC 301. Only one of the five uncoated glass surfaces are shown. The individual markers show reported values, dotted lines show plus and minus two times the calculated standard deviation for that property, and finally dashed lines show limits imposed by NFRC 302 (.02 for emissivity).

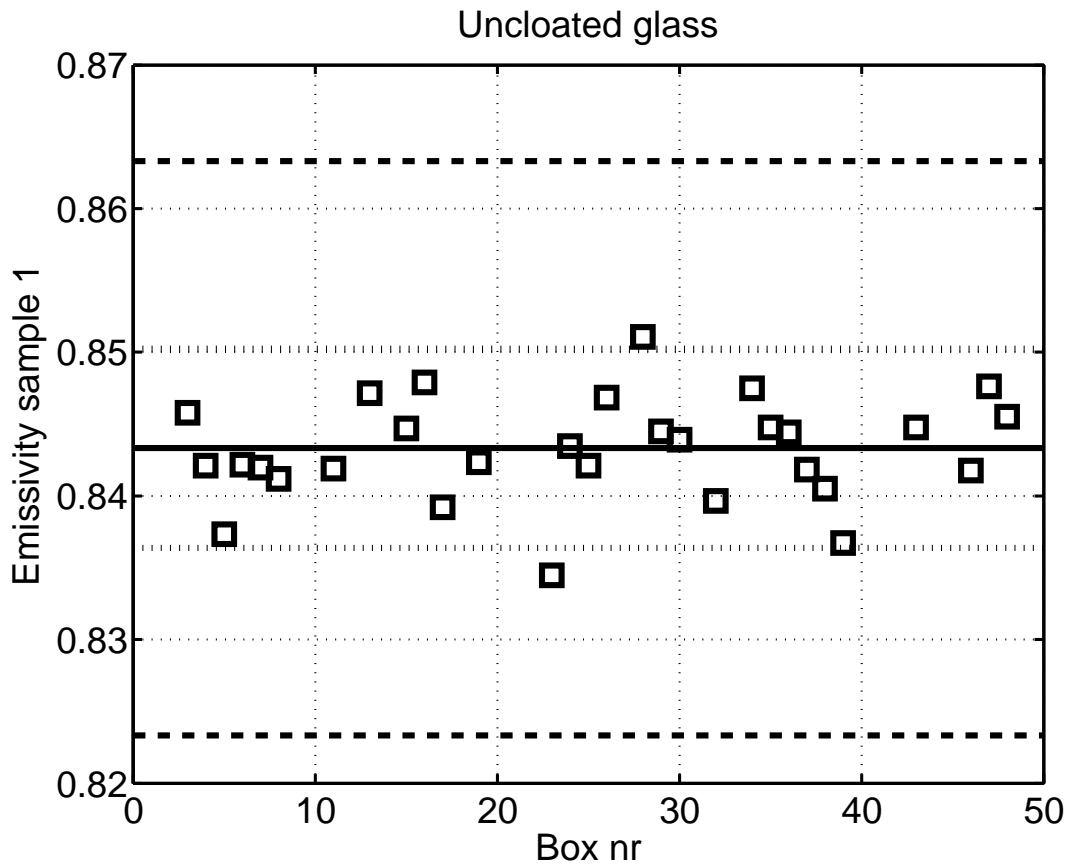


Figure 15: Calculated emissivity of uncoated glass surface of sample 1.

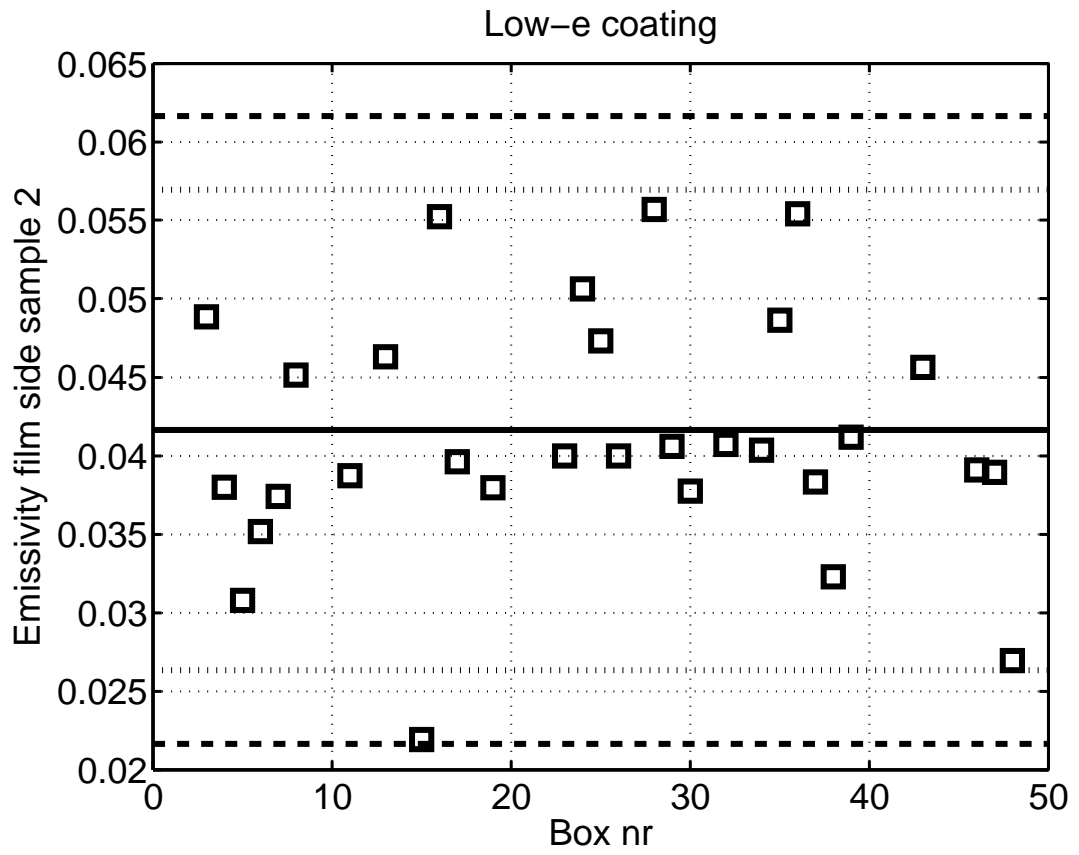


Figure 16: Calculated emissivity of coated surface of sample 3.