Title

LBNL 2011 Inter-Laboratory Comparison for Laboratories Submitting Specular Data to the International Glazings Database (IGDB)

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LBNL 2011 Inter-Laboratory Comparison for Laboratories Submitting Specular Data to the International Glazings Database (IGDB)

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2012

DISCLAIMER

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. 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 dictated 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 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 was 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 Selection committee

Mike Rubin, previously employed at LBNL, organized a sample selection committee consisting of Dave Haskins, 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. This selection has been retained for the present ILC.

2.2 Specular sample selection

A total of five samples were selected from three companies, PPG, Solutia, and 3M. PPG produced a clear low-iron glass and also clear low-iron 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. To summarize:

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

A total of 50 boxes were sent out in the initial round, another 50 were kept at LBNL to allow for future 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, 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.

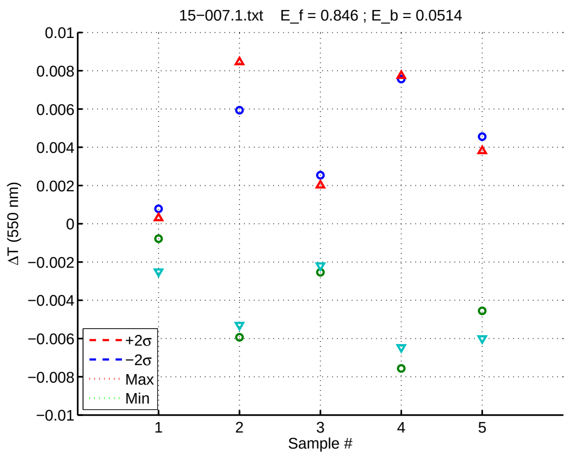


Figure 1: Statistics of the absolute variation of transmittance measured at 550 nm for the five different samples.

The variation of the clear glass samples and the clear clear laminates was very 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 conclusion from looking at this data is that it is of little benefit to force the manufacturers to measure more than one sample.

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

3 Solar optical range, 300–2500 nm

3.1 Instruments and detectors used

A majority of the ILC participants used 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 2a).

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).

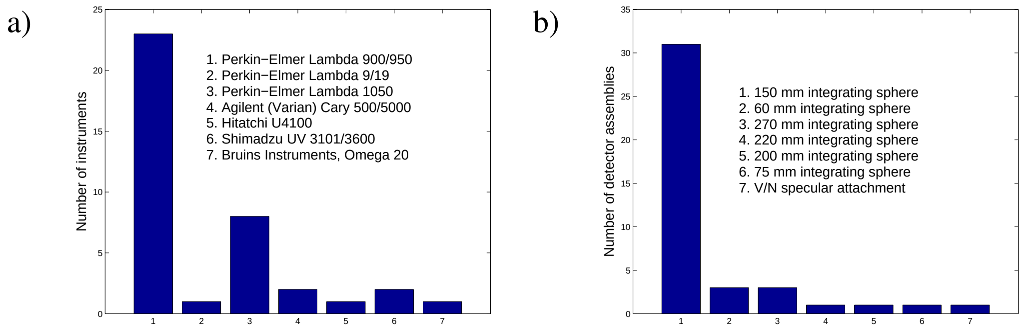


Figure 2: a) Distribution of instruments among the participants. b) Distribution of detector systems used.

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 Trends

With access to large data sets like this is is interesting to see if there are any trends that can be quantified.

3.2.1 Effects of large wavelength steps when measuring applied films

LBNL requires steps of 50 nm or shorter 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.

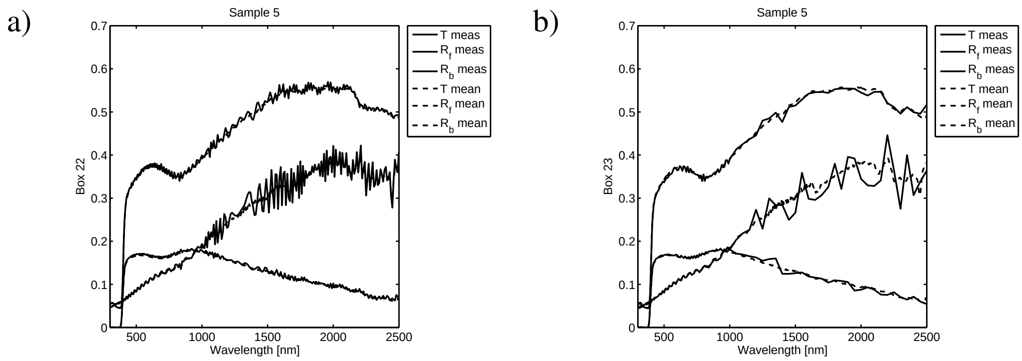


Figure 3: Data for the applied film sample. a) Data presented with 5 nm steps. b) Data presented with 50 nm steps.

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 sensible to degradation.

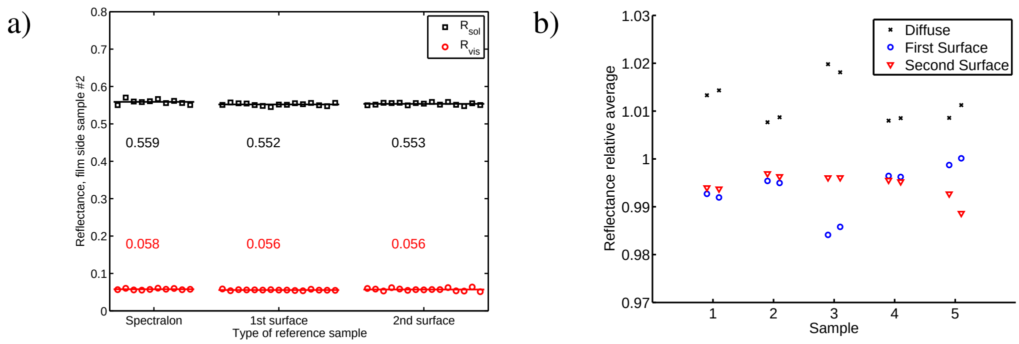


Figure 4: Integrated reflectance grouped for kind of reference sample. The average value for each group is written next to the curve. The diffuse Spectralon group has a slightly higher average than the other two. a) Film-side reflectance of sample #2 showing the individual measurements for each participant.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

3.3 Example of corrected results

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 instruments.

Example of a couple of different instrument results are shown in figure 5a) 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 5b) 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.

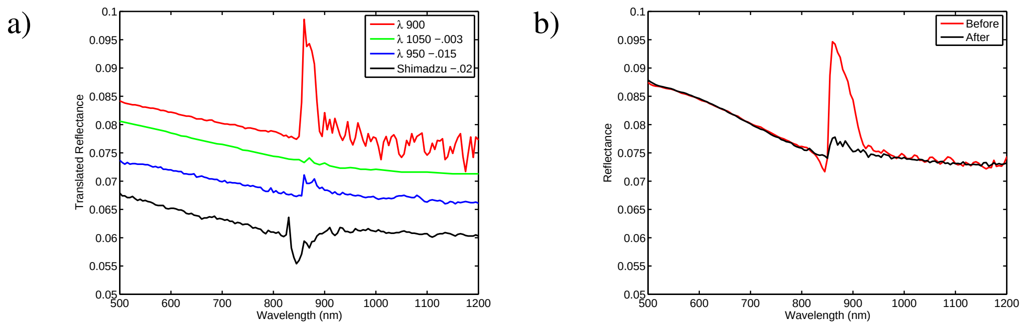


Figure 5: a) Example of different glass reflectance measurement of sample #1, values have been shifted laterally to clearer show the discontinuities. b) Example from a measurement in the ILC conducted in 2007.

3.3.2 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 6 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 examplified using data from 2007.

It is hard to repeat this effect but a theory for how this happens is suggested. The submissions in figure 6 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 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.

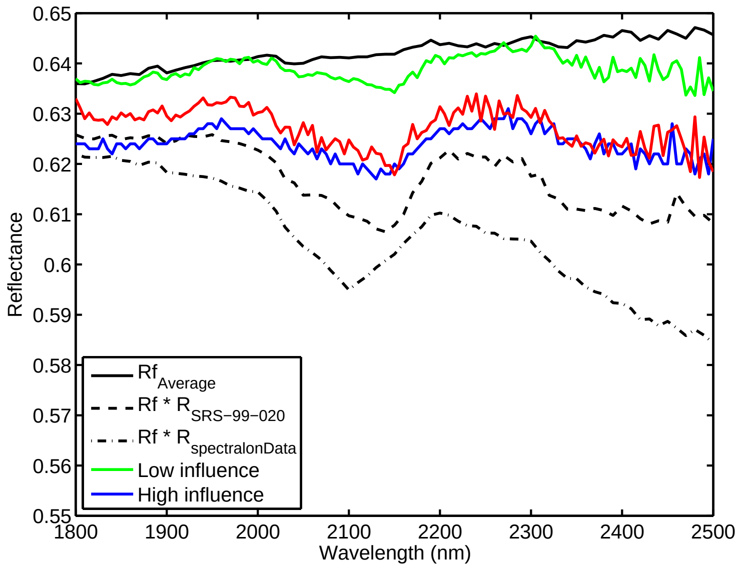


Figure 6: Average reflectance of a metal coated glass substrate and that value multiplied with the reflectance of Spectralon contrasted against submissions with absorption artifacts.

[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

4 Thermal infrared range, 5–25μm

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 7. The THERMES project[2, 3, 4] did thorough comparisons between dispersive and FTIR instruments and those have not been repeated here since there were only three dispersive instruments in the whole test.

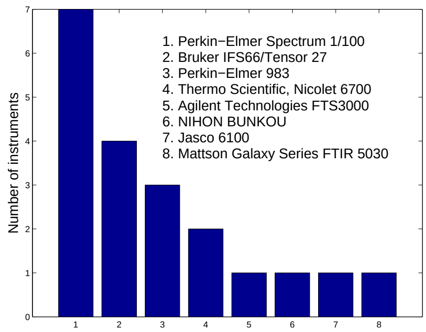


Figure 7: Distribution of instruments used to measure reflectance between $5\mu m$ and $25\mu m$ for calculation of emissivity.

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 5 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 8 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].

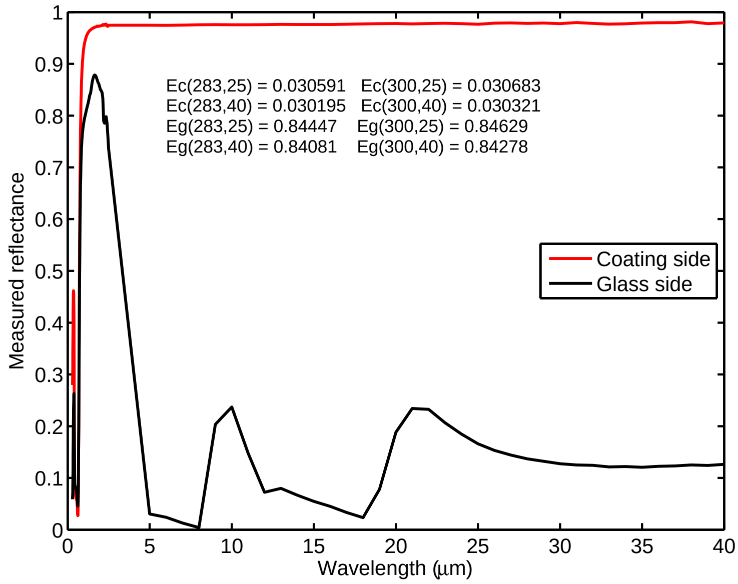


Figure 8: Spectral reflectance measured and hemispherical emissivity calculated for two temperatures, 283 K and 300 K, and using two different upper boundaries for the calculation. The calculation was carried out for both the glass side (E_g-values) and the coated side (E_c-values) of the sample.

4.3 Measurements

Out of the five samples, there were only two surfaces that were not uncoated glass, those were the low-e coating of sample 2 and the applied film of sample 5. By measuring glass emissivity 8 times the laboratories got good information about how the repeatability of the instrument was. An example of such a result is shown in figure 9.

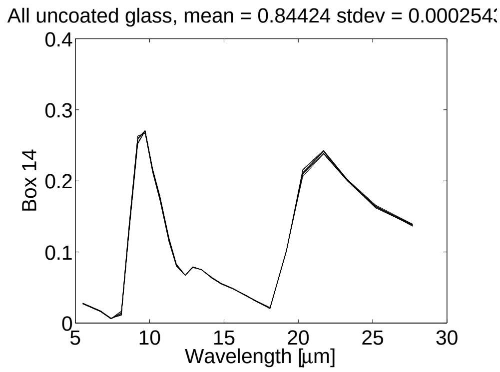


Figure 9: Example of submitter number 14's reflectance measurement of the 8 uncoated glass surfaces all show together in one graph to demonstrate the instrument variation.

In addition to the glass reflectance the low-e and applied film coatings were both graphed individually. Examples of such measurements are shown in figure 10.

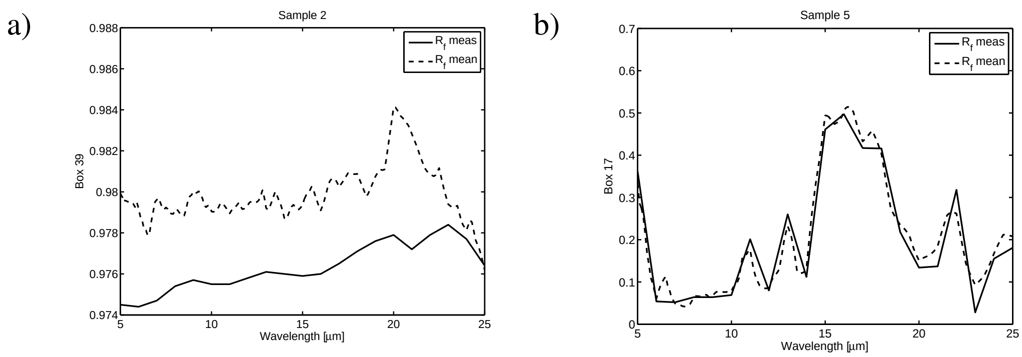


Figure 10: Example of submitted IR reflectance of the low-e coating and the applied film in a) and b), respectively. $R_f meas$ is the submitter's measured value of the film/coated surface and it is compared to the average of all submitters measured values $R_f mean$. The index $_f$ indicates film side rather than glass 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 emisssivity

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

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

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

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

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 $(1.4387 \times 10^4 mumK)$

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 to measure the hemispherical value it is calculated using empricial expressions[7].

For uncoated substrates the expression is:

$$\varepsilon_h = 0.1569\varepsilon_n + 3.7669\varepsilon_n^2 - 5.4398\varepsilon_n^3 + 2.47333\varepsilon_n^4 \tag{3}$$

where ε_n is the normal emissivity calculated using equation 1.

For coated substrates the expression is:

$$\varepsilon_h = 1.3217\varepsilon_n - 1.8766\varepsilon_n^2 + 4.6586\varepsilon_n^3 - 5.8349\varepsilon_n^4 + 2.7406\varepsilon^5. \tag{4}$$

4.4.3 Calculated emissivities for samples 1–5

All calculated emissivity values are presented in appendix C. The average value for the low-e coating was 0.024. The average value for the the applied film was 0.77.

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 3.3.2 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 adviced 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.

Two changes to the IGDB submission procedure are suggested as a result of this ILC and they are both considered to be straight-forward:

1. Switch to require narrower steps that 50 nm in the near infra-red. This is demonstrated in section 3.2.1 on the applied film data. Most instruments already measure this data so it is a small change for submitters.

2. Require emissivity to be submitted as the measured direct IR reflectance. This improves the quality of the database since it allows users to calculate emissivity according the their standard of choice. The submission of self-calculated emissivity data seemed to be the largest problem in section 4. Also, having spectral data uncorrected allows users of the data to perform their own calculations which is useful for as long as there are multiple standard ways of doing these calculations.

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

This work was supported by the Assistant Secretary for Energy Efficiency and Renewable Energy, Building Technologies Program of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

All participants contributed significant time in measuring all sample properties.

References

[1] W. Möller, K. Nikolaus, and A. Höpe, "Degradation of the diffuse reflectance of spectralon under low-level irradiation," *Metrologia* **40**, pp. 212–215, 2003.

[2] "THERMES, thermal emissivity of energy-saving coatings on glass - preservation of the measuremnt infrastructure of the glazing industry," tech. rep., EU Growth Programme Contract G6RD-CT-2001-00658, 2001.

[3] K. Gelin, A. Roos, P. van Nijnatten, and F. Geotti-Bianchini, "Thermal emittance of coated glazing - simulation versus measurement," *Optical Materials* **27**, pp. 705–712, 2005.

[4] P. van Nijnatten, M. Hutchins, A. Roos, F. Geotti-Bianchini, P. Polato, C. Anderson, F. Olive, M. Köhl, R. Spragg, and P. Turner, "Thermal emissivity of energy-saving coatings on glass: The THERMES project," in *4th International Conference on Coatings on Glass*, 2002. Braunschweig, Germany.

[5] National Fenestration Rating Council, "NFRC301: Standard test method fo emittance of specular surfaces using spectrometric measurements," 2010.

[6] European Committee for Standardization, "EN673: Glass in building - determination of thermal transmittance (u value) - calculation method," 1997.

[7] M. Rubin, D. Arasteh, and J. Hartmann, "A correlation between normal and hemispherical emissivity of low-emissivity coatings on glass," *Int. Comm. Heat Mass Transfer* **14**, pp. 561–565, 1987.

Appendix

A List of Participants

An auto-generated 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.

Institute	**Contact**
3M Company	Raghu Padiyath
AGC Glass Company North America	BOb Curtin
AGC Glass Japan/Asia Pacific	Sigetosi Hirasima
AGC glass Europe	Ingrid Marenne
Arcon Flachglas-Veredlung GmbH & Co.KG	Carsten Ruppe
Berlin Institute of Technology	Stefan Gramm
CEPT, Navrangpura, Ahmedabad.	Dr. Vinod Patel
CSG Holding Co., Ltd.	Chengde Huang
Cardinal Glass Industries	Jordan Lagerman
China Bluilding Material Test & Certification Center	Wu,Jie
DuPont P&IP Glass Laminating Solutions R&D	Stephanie H. Lott
Euroglas	Martin Daams
Fraunhofer Institute for Solar Energy Systems	Helen Rose Wilson
Guardian Industries Corp	Jason Theios
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
Saint-Gobain Solar Gard LLC	JON MITCHELL
Solar Energy Research Institute of Singapore (SERIS)	Teo Wei-Boon
Solutia Inc.	Julia Schimmelpenningh
Solutia Perforamnce Films	Beth Lawless-Coale
Sonnergy Limited	Michael G Hutchins
Southwall Technologies	Brija Nand
Southwall europe GmbH	Gunnar Spitzer
Stazione Sperimentale del Vetro	Antonio Daneo
The Ångstrom Laboratory, Uppsala University	Arne Roos
Viracon	Dan Wacek

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

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

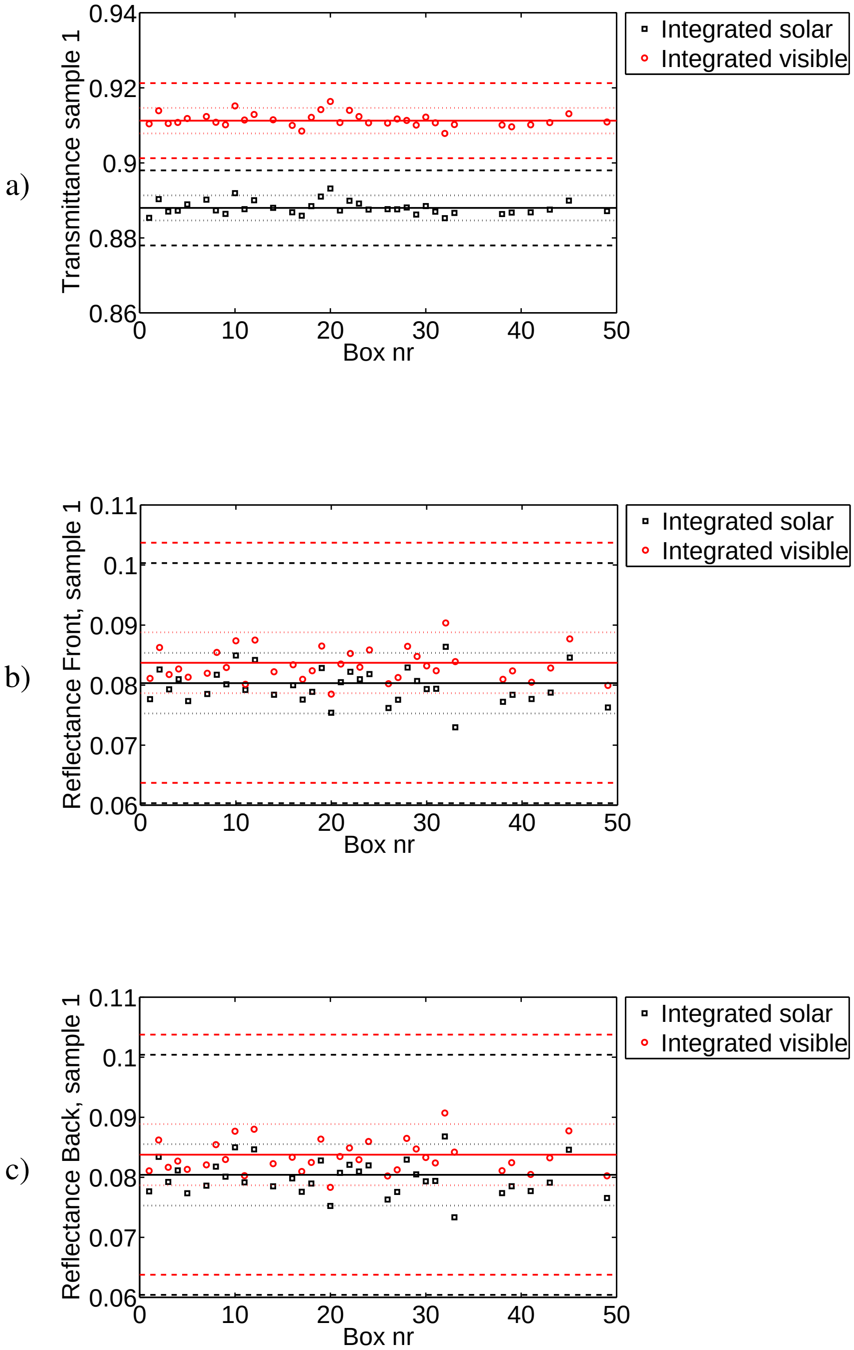


Figure 11: Integrated solar and visible optical properties for sample 1. a) Transmittance, b) front reflectance, and c) back reflectance.

B.2 Sample #2

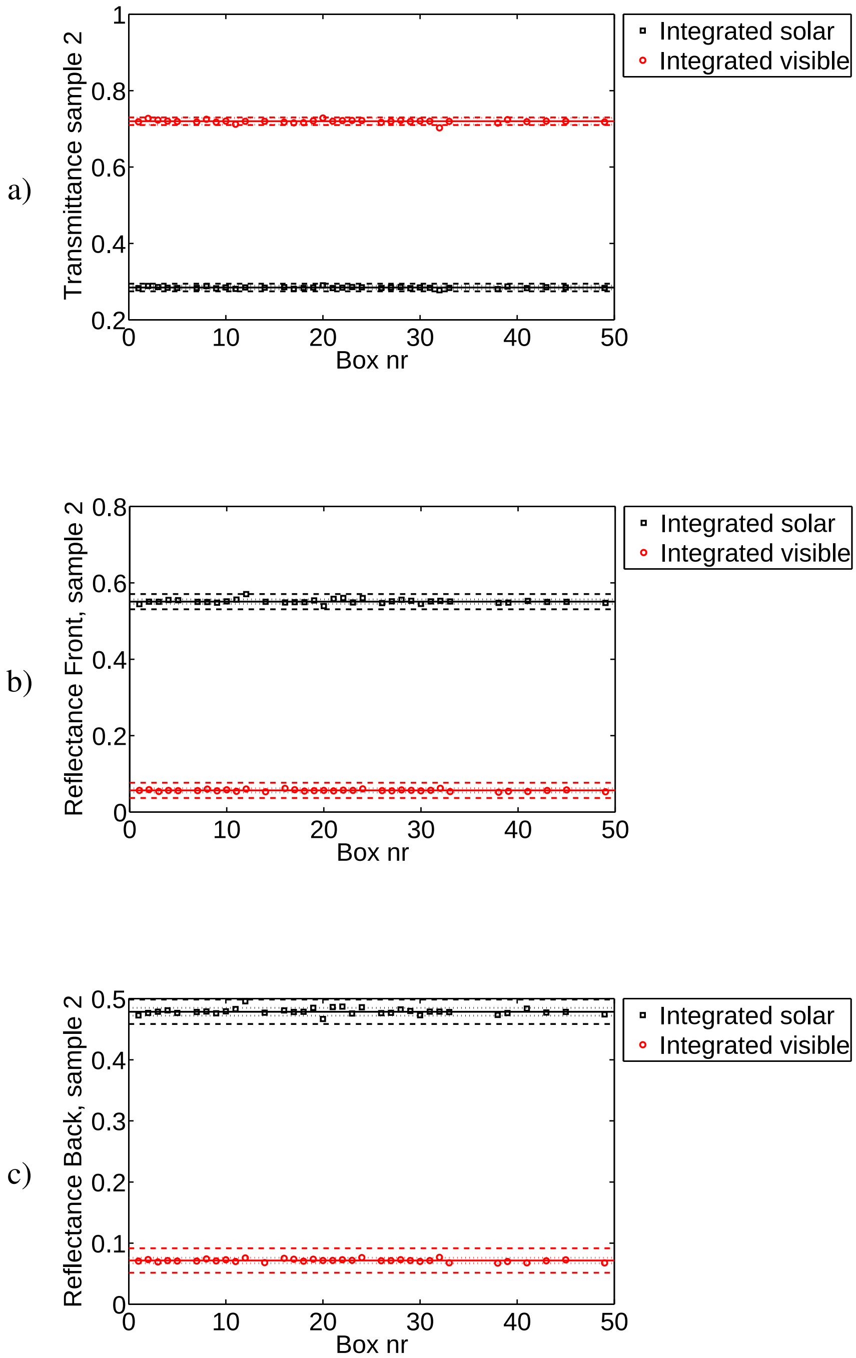


Figure 12: Integrated solar and visible optical properties for sample 2. a) Transmittance, b) front reflectance, and c) back reflectance.

B.3 Sample #3

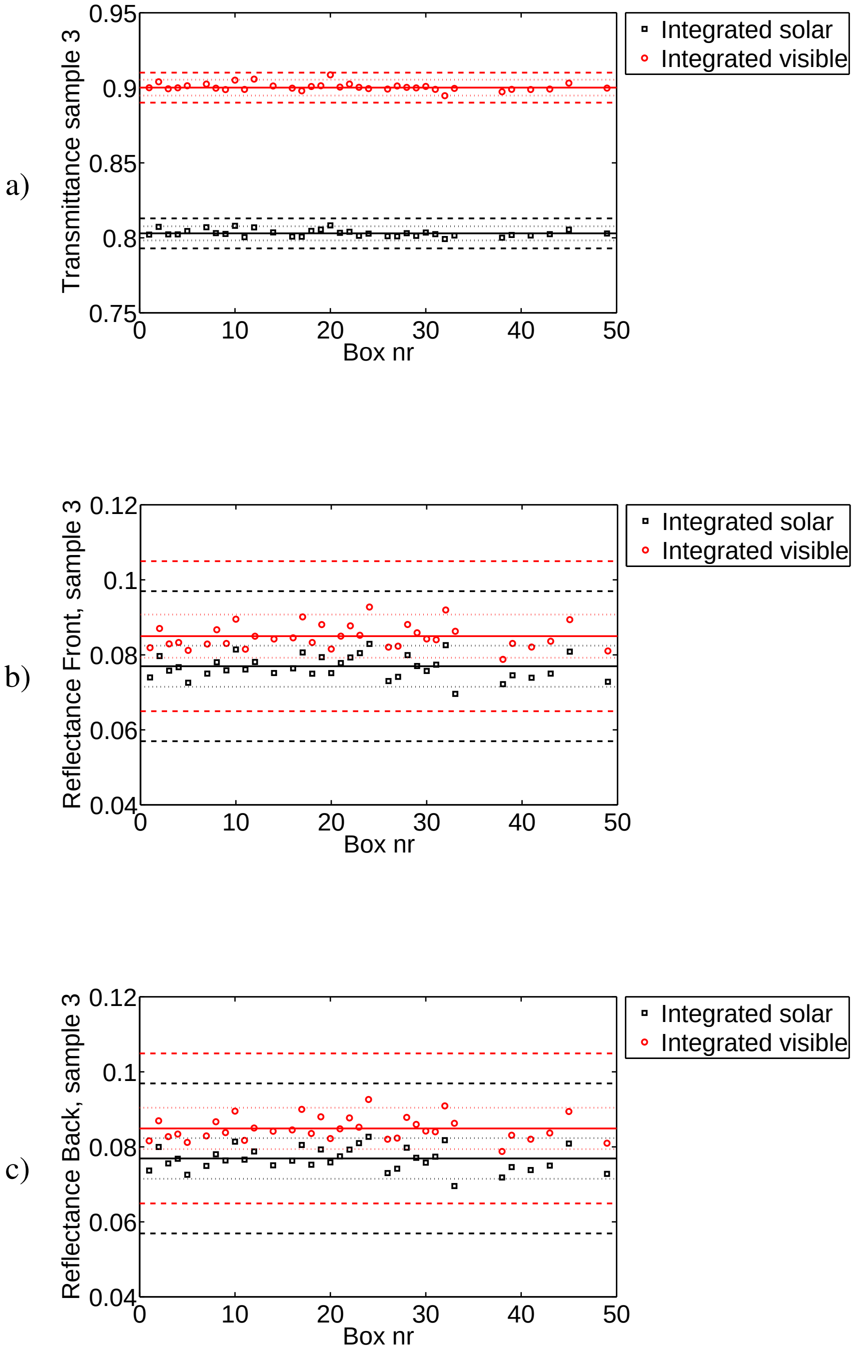


Figure 13: Integrated solar and visible optical properties for sample 3. a) Transmittance, b) front reflectance, and c) back reflectance.

B.4 Sample #4

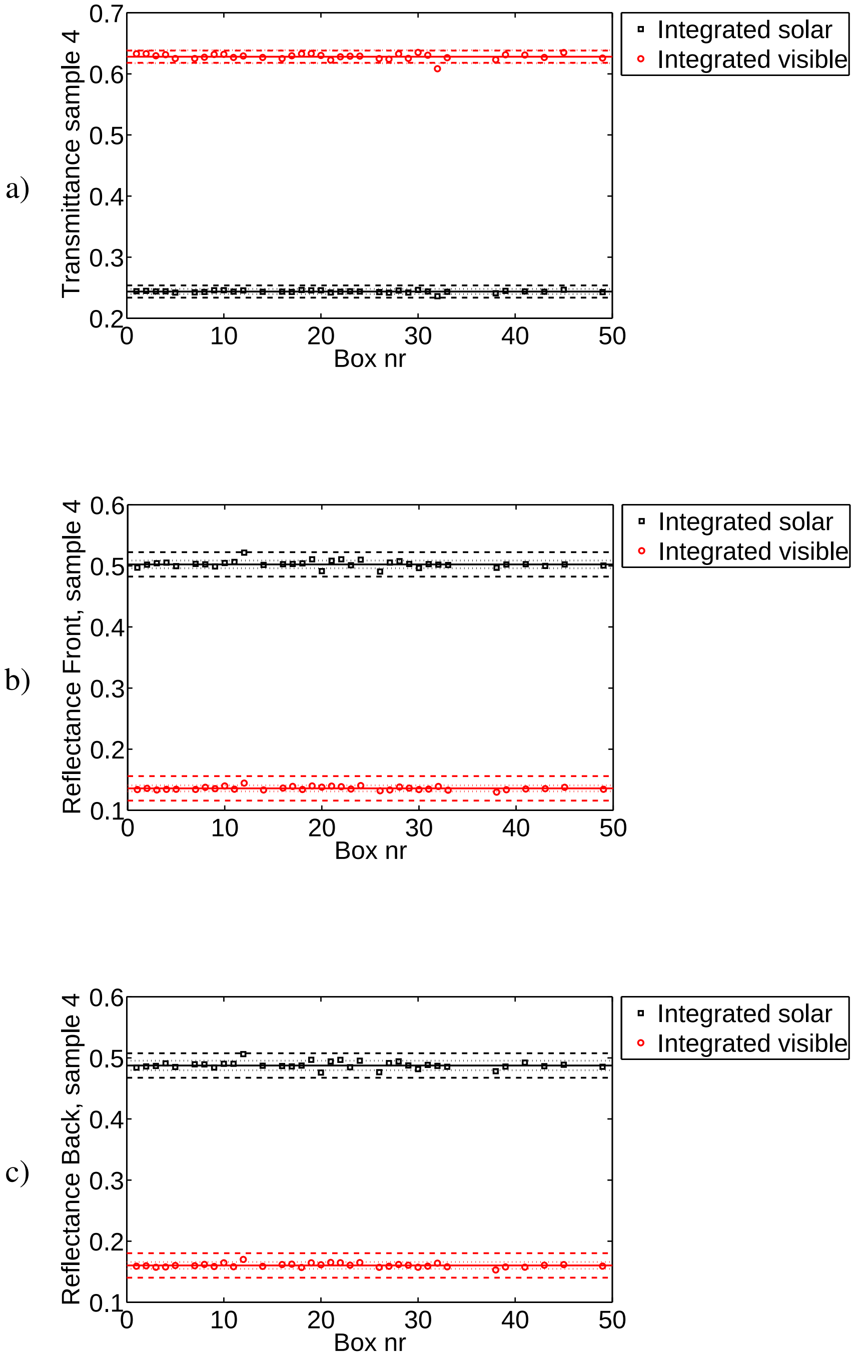
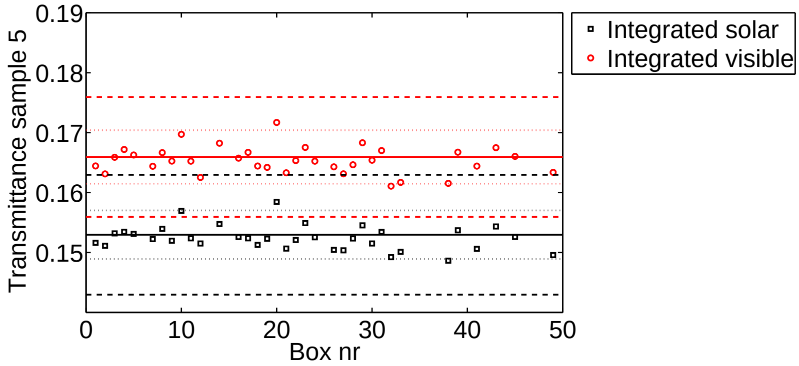


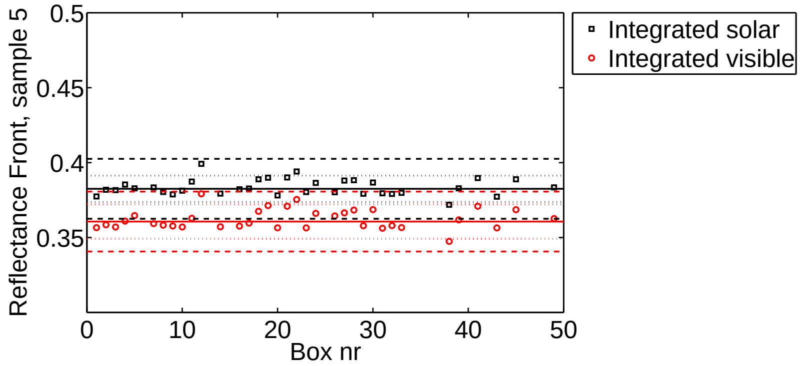
Figure 14: Integrated solar and visible optical properties for sample 4. a) Transmittance, b) front reflectance, and c) back reflectance.

B.5 Sample #5

a)



b)



c)

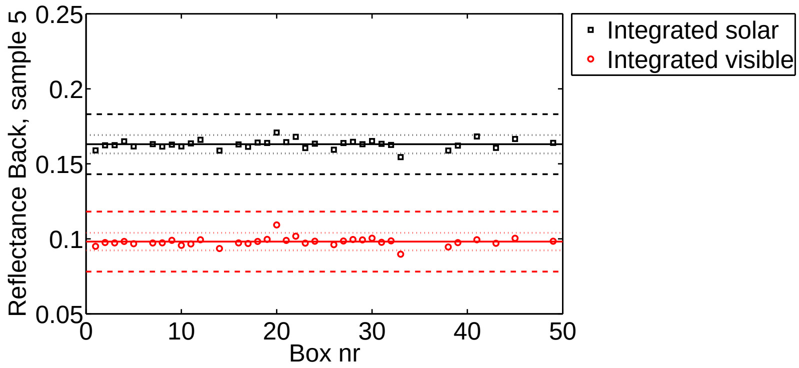


Figure 15: Integrated solar and visible optical properties for sample 5. a) Transmittance, b) front reflectance, and c) back reflectance.

C Graphs for all IR measurements

The graphs in this section shows the calculated emissivity for the participants that submitted spectral IR data and submitted emissivity values for the participants that did not do so. 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 (.02 for emissivity).

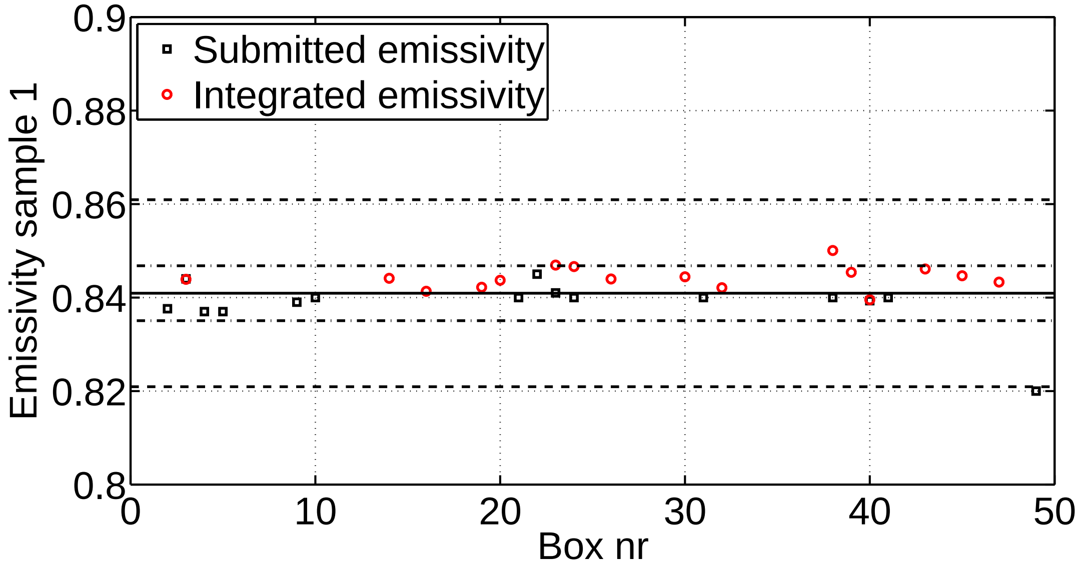


Figure 16: Calculated emissivity of uncoated glass surface of sample 1.

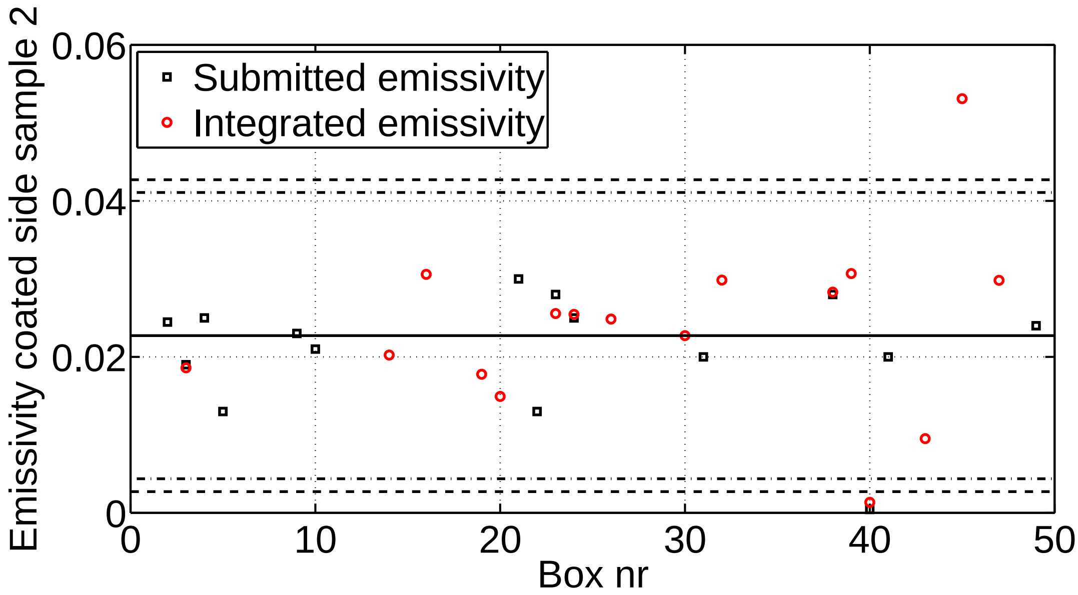


Figure 17: Calculated emissivity of coated surface of sample 2.

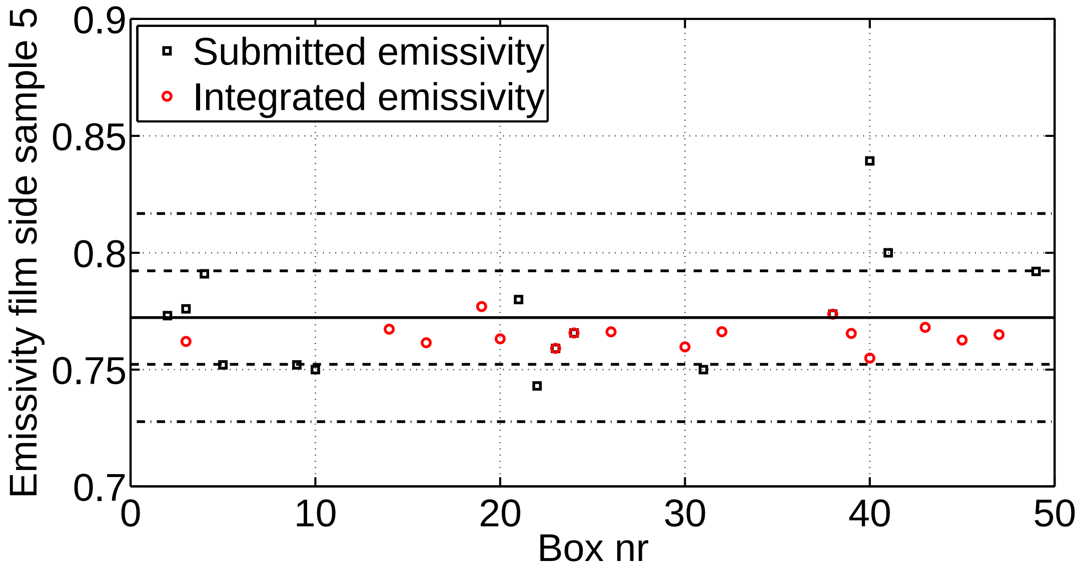


Figure 18: Calculated emissivity of coated surface of sample 2.