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Authors

Jonsson, Jacob C.
Rubin, Michael

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NFRC Interlaboratory Comparison on Optical Properties

Jacob C. Jonsson and Michael Rubin
Windows and Daylighting Group
Lawrence Berkeley National Laboratory
Berkeley, CA 94709

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Introduction

As part of the NFRC rating process, optical data on glazing materials is combined with other information to calculate various properties of a window product. The administrative procedure for gathering such optical data is governed by NFRC 302¹, which in turn refers to NFRC 300² and NFRC 301³ for the technical procedures by which the optical properties are determined in the solar and infrared ranges, respectively. In practice, the data is compiled by the Lawrence Berkeley National Laboratory (LBNL) and becomes part of the International Glazing Database (IGDB).

NFRC 302 specifies that submitters of optical data or their representatives must participate in a “round robin” or ILC. Often, manufacturers of glazing materials have the optical equipment necessary to perform their own measurements. NFRC 302 allows manufacturers to submit their own measured data subject to a set of checks including peer review to ensure the accuracy of such data. In some cases the glazing manufacturer does not have the required equipment and so may choose to send the samples to a test laboratory. In other cases the manufacturer of the final product such as a laminate may ask a component supplier, often a glass manufacturer, to perform the measurements for them. In such cases the “representative” must have qualified by participating in the ILC. An ILC is only required every four years and it would be unfair to expect new product submitters to wait so long. Therefore, two interpretations are made on occasion: (1) a new data submitter does not have to wait for the next ILC if they submit a set of samples with their first dataset for comparison at LBNL (a mini ILC), or (2) if they have participated in an ILC conducted by some other reputable independent organization.

What does it mean to successfully participate in the ILC? It would be nice to be able to go to the statement of error in the relevant measurement standards for a simple answer. Unfortunately, the current statements of precision and accuracy are not adequate or not

¹ NFRC 302-2004: Verification Program for Optical Spectral Data. http://nfr.org/documents/NFRC_302-2004.pdf

² NFRC 30-2004-E0A1 (January 2007): Test Method for Determining the Solar Optical Properties of Glazing Materials and Systems. http://nfr.org/documents/NFRC_300-2004-E0A1.pdf

³ NFRC 301-2004-E0A1 (January 2007): Standard Test Method for Emittance of Specular Surfaces using Spectrometric Methods. http://nfr.org/documents/NFRC_301-2004_E0A1.pdf

clear as discussed below. Instead this ILC will help to redefine the expected and allowable errors in our standards. This is an opportune time for such introspection because NFRC is currently leading the effort to renew two ASTM optical property standards. The findings of this ILC will support that effort. The lapsed ASTM standards will be renewed through NFRC initiatives and participation of NFRC members on ASTM committees. Every effort will be made to harmonize our standards with international standards.

In the solar range, NFRC 300 refers to ASTM E903 (currently discontinued) for measurement practice. This venerable standard gives a lengthy discussion of possible sources of error. There is much of interest in that discussion, but also some ambiguity. We can ignore the errors mentioned in E903 resulting from a simplified selected ordinate calculation, because NFRC uses an accurate weighted-average calculation. In any case participants do not perform these calculations themselves; they submit only raw spectral data. We can also discount “errors” discussed in E903 produced by using a different solar spectral irradiance in the calculation than the one that exists locally; NFRC ratings are relative and all participants are equally affected by such errors. That still leaves us with a measurement error as large as ± 0.02 as estimated by ASTM E903. Such a large error would be unacceptable and we should be able to do better.

For the emittance, NFRC 301 simply says to report the values to three decimal places. The usual interpretation of such a bare statement of error is that we know the answer to ± 0.001 , which we certainly do not. A recent ILC conducted by the European Thermes project stated that the spread in the emittance of the test samples was no better than 0.005. To achieve this result, the participants were instructed to ignore their usual procedures and follow a narrowly prescribed sequence of measurements. It is unlikely that we will do better than Thermes for the foreseeable future. One of the outcomes of the Thermes project is supposed to be a revision of CEN and ISO standards on emittance. This is important because, with FTIRs taking the place of dispersives, existing infrared standards such as NFRC 301 will become obsolete.

The main purpose of this ILC is to evaluate the current ability of data submitters to make accurate measurements by following NFRC 300 (and ASTM 903) and NFRC 301. In this way we will find out whether and how our standards need to be improved. Unlike the Thermes ILC, however, our immediate goal is not to test new procedures. That will come in the followup to the ILC as we work with the participants to make improvements to their process and to rewrite our standards.

Measurement Procedure

Each participating laboratory received two sets of samples: one for the solar range and the other for the thermal infrared. One set of samples was measured in sequences by each lab. The samples come from previous ILCs originating in Europe. It may prove useful and interesting to compare our results with an extensive database of previous results. Also it will assist us in the elusive quest for international harmonization. One disadvantage is that at least one of the coated samples suffered degradation over the years

which we will take in to account. Others such as the uncoated glass should be quite unchanged. The samples are of various types, but variety in application and and composition are much less important than variety in level of transmission and reflection for our purposes. Table 1 and Table 2 identify the samples for the solar and infrared tests, respectively:

Table 1. Solar test samples.

Sample ID	Name	Description
H.8.1	Antelior Argent	Pyrolytic TiO ₂
H.8.2	Amiran	Antireflection coated glass
H.8.3	Diamond glass	Uncoated clear float
H.8.4	CoolLite SKN	Double-Ag low-e
H.8.5	Planitherm Futur	Single-Ag solar-control low-e

Table 2. Emittance test samples.

Sample ID	Name	Description
S01	Planitherm Std	Single-Ag low-e
S02	SS-108	SS/SiN medium-e
S03	Mirror SGR	Ag/SiN low-e
S04	Ecologique	SnO ₂ :F, low-e
S05	SKN-165B	Double-Ag low-e
S06	Planilux	Clear float, Sn side

Our immediate purpose is to assess current measurement practice. It was known in advance that the participants were following procedures that had a high degree of variability although technically within the fairly loose guidelines of NFRC 300 and 301. We did not wish to fix too many of the measurement parameters because there are legitimate reasons for some of this variability. For example, a scan speed might be chosen so that the noise level in the spectrum was minimized which would depend on the age and model of the spectrometer. As long as acceptable accuracy can be achieved we do not wish to overspecify. The participants were instructed to follow their usual measurement procedure as performed for a submission to the IGDB following the guidelines of NFRC 300 and NFRC 301. Thus, each participant measured the transmittance and the reflectance from each side of the solar-range sample set and the reflectance (emittance) from the coated side of the infrared sample set. In some cases the participants did not have an infrared spectrometer. This does not necessarily disqualify them for submitting data because, for example, they may make only laminates which are encapsulated in glass. Glass is highly absorbing in the infrared so whatever coatings or polymers may be inside do not contribute to the surface properties. The emittance of glass is a standardized value and so no measurement of emittance is needed in this case.

Instrumentation for the solar range consists of a so-called “uv-vis-nir spectrophotometer” which is a highly automated dispersive type of instrument with multiple sources, detectors, gratings and filters. Although redesigned instruments are produced every decade or so, it is not uncommon for a laboratory to keep their instrument for 30 years or more. The most popular series of instruments in the Perkin-Elmer Lambda 9/19/900/950 as seen in

Table 3. Seven out of 13 instruments in this ILC are of the most modern Lambda 900/950 variety. Two instruments are Varian Cary 500s which are considered to be of comparable quality to the Lambda 950 and operate on very similar principles. All instruments in Table 3 are equipped with an integrating spheres made by Labsphere with a diameter of 150 mm except in one case. Despite the relative uniformity of instrumentation there is wide latitude in our standards to set scan parameters such as scan speed and slit width. Note that most participants use the default fixed slit of 2 nm in the visible (not necessarily the best choice), but a wide range of NIR sensitivities (variable slit program) in the infrared. The scan speed also varies widely. Many opt for a slow speed of about 240 nm/min which is probably the best choice if you don't need higher sample throughput. Most use calibrated reference mirrors from reputable sources, but some could not even provide complete information about those mirrors. This is a very important point and will be discussed in terms of the results below.

Instrumentation for the thermal infrared has changed significantly over the years and not for the better. First, notice in

Table 4 that there is a much wider variety of instrument make and model. Much less is known about the relative quality of these instruments than is known about the solar spectrometers. In the past Perkin Elmer was also the most popular maker of infrared dispersive spectrometers which operated on principles not dissimilar to the solar spectrometers. They are no longer manufactured due to the superiority of Fourier-transform infrared spectrometers (FTIRs) for chemical spectroscopists who far outnumber those desiring to measure radiometric properties with accuracy. For our purposes FTIRs suffer from at least two severe disadvantages: First, FTIRs are single-beam instruments which means that source instability or other factors can quickly cause the baseline to drift. Second, the beamsplitter, which is the heart of the interferometer, is a transmitting element. In order to extend the range beyond 25 microns a special beamsplitter must be used made of hygroscopic CsI. The instrument must be purged constantly not only to avoid atmospheric absorption bands during measurement but also to protect the beamsplitter from irreversible damage. Only half of the participants are venturing beyond 25 microns despite the fact that significant energy exists beyond this point in the blackbody energy spectrum. The entire purpose of the Thermes project is to find better ways to deal with this new reality that has been thrust upon us.

Table 3. Test equipment and parameters for the solar optical range.

Lab	Spectrometer	Sphere	Slit (nm)	Scan Speed (nm/min)	Sensitivity/ Gain	Integration Time (sec)	Reference
1	Lambda 19	Labsphere RSA-PE-19 150mm	2	240	3		NIST AI 2 nd 2023
2	Cary 500	DRA-CA-5500	2	600/2400			Spectralon
3	Lambda 950	Labsphere 150mm					Labsphere AI 1 st
4	Lambda 9/19	DRTA 9A	2	240 (T) /480 (R)	1		AI
5	Lambda 900	RSA	2	267 (T) / 530 (R)	2	0.033/0.033	AI
6	Lambda 19	Labsphere RSA-PE-19 150 mm		480			NIST AI 2 nd – working AI first
7	Lambda 900	Labsphere	2		1	1	4 High index glasses
8	Cary 500			188		.033	
9	Lambda 900	Labsphere PELA-1050 60mm with VN 8deg.	5	300/300	4	.88/.96	Absolute VN 8deg PELA 6008
10	Cary 500E	Labsphere 150mm					
11	Lambda 950	Labsphere 150mm 8°	5	822(t)/650 (R)	5	.24/.36	Spectralon SRS-99-010 calibrated by NIST AI 2nd
12	Lambda 900						
13	Lambda 900	Labsphere 150mm	5	833 /937	5	.24/.28	NIST AI 2 nd – working AI 2nd
14	Lambda 19	Labsphere RSA-PE-19	4	480	3		“calibrated data from Labsphere”

Table 4. Test equipment and parameters for measurement of thermal emittance.

Lab	Spectrometer	Type	Reflection Accessory	Angle	Resolution (cm-1)	Scans	Reference	Purge	Max. λ (μm)
1	PE 983G	D	3x condenser		11.5	1	NPL 1 st Al	no	40
2	-	-	-	-	-	-	-	-	-
3	Nicolet Magna 550	F				5	NPL 1 st Al	Dry air	25
4	Matson Galaxy 5030	F	Pike	10		32	1 st Al	N2	50
5	-	-	-	-	-	-	-	-	-
6	PE Paragon 1000	F		16	4		NPL 1 st Al	N2	40
7	Bruker Tensor 27	F	A510/Q	8	4	7	Al - calibration for generic Al	N2	25
8	-	-	-	-	-	-	-	-	-
9	Nicolet 6700	F	Harrick VR1-VWA-12	12		64x2	Absolute	N2	45
10	Nicolet 560	F	Infragold Sphere				InfraGold IRS-94-010		25
11	Nicolet Magna 750	F	Barnes/SpectraTech M-134	11	4	128	NPL 1 st Al	N2	25
12	-	-	-	-	-	-	-	-	-
13	-	-	-	-	-	-	-	-	-

Results and Analysis

Solar

Looking first at the raw spectral data in Figures 1-5 for each of the 5 samples we see some typical features. The visible wavelengths have relatively smooth curves while in the infrared, especially near the high-wavelength limit, the curves tend to be more noisy. This is a known consequence of the types of source and detectors used in standard instruments. If we were to make a statistical analysis of the data at each point we could calculate standard deviations but this would not be entirely meaningful or useful. These points are all connected through the physical phenomena of resonance and thus we expect the curves to be smooth and data points to be dependent on their neighbors. In any case, when the visible and solar average quantities are calculated any noise is effectively smoothed out. Random error in the form of noisy spectra then is not the main contributor to the spread in the results. Furthermore, all of the instruments used in this ILC are of the double-beam type which means that instrument drift is not a major factor either. The main sources of error will undoubtedly be systematic error or inaccuracy.

The contention that systematic error dominates is borne out in several ways that are perhaps more clear in the solar average results of figures 6-10. Look for example at Sample 2 and Sample 3. These samples are symmetric, sample 2 being an antireflected glass coated on both sides and sample 3 being a piece of low-iron glass. The reflectance measured from each side of a symmetric sample amounts to two measurements of the same sample in sequence with the sample removed from and then returned to the compartment. This high repeatability is quite common for most labs as known from prior experience. Always high on the list of suspects is the use of different, poorly calibrated or deteriorated reference mirrors in reflectance mode. Similarly, there are several types of errors possible in correcting the raw data for the reflectance of the standard reference material. This source of error does not exist for transmittance where the reference is always the air in the unobstructed reference beam. The generally better behaved transmittance measurements bear out the suspicion that the reference is a significant problem for reflectance. There are many other possible sources of systematic error in both reflection and transmission such as misaligned samples or port plugs.

Many systematic trends can be spotted in the spectra of Figures 1-5. The region of the spectrum in which they occur, their proximity to sharp features and programmed component changes in the instrument give us clues to their cause. Many other systematic trends cannot be understood without more information from the laboratory in question. Scanning the summary graphs by sample in Figures 6-10 immediately shows that some labs always measure too high and others too low. The participants should look especially at the summary graphs for their particular laboratory. Although not identified by name in this report each participant will know their laboratory number. It is also instructive to look at the summary graphs by measured property in Figures 24-29. There is no particular reason to believe that reflectance from the coated side should incur a different level of error than reflectance from the uncoated side, at least for samples of moderate

thickness. There is reason to believe that the transmittance should have a tighter distribution for reasons discussed above and that is apparently true from the evidence of Figures 24-29. If the problems with reference materials and correction procedures are addressed, there is reason to expect that reflectance can be measured with the same confidence as transmittance and we will strive to achieve that goal.

Summary Table 5 is a gross simplification of all the data presented in this report. Furthermore, calculating the standard deviation of a collection of nonrandom data is not correct procedure. It would be more meaningful to look at the full spread of data as shown in Figures 24-29 when assessing the performance of the participant group. When considering what our error expectations should be, however, it is probably better to look at a number on the order of or less than the standard deviation. If some labs can achieve this level of performance, it is logical to assume that the outliers can be improved to this level since all use equipment of identical or similar quality. From this point of view we should be trying to achieve errors in transmittance of no more than a few tenths of a percent and in reflectance perhaps twice as much, say half a percent.

Table 5. Summary of errors for the solar range.

Sample	1	2	3	4	5
TsolMean	63.2457	82.1277	88.8447	40.9195	48.7503
TsolStd	0.3101	0.3884	0.8173	0.3456	0.2338
R1solMean	24.5373	9.9710	8.2257	38.4898	34.2059
R1solStd	1.1479	0.5446	0.6136	1.0412	2.4198
R2solMean	20.7849	10.0096	8.2203	26.3115	25.3619
R2solStd	0.7734	0.5398	0.5955	0.6795	0.6407

Emittance

As in the solar spectrum we first look at the detailed reflectance spectra in Figures 30-35. At first glance we see that some spectra have extreme problems. One lab has a glitch at 5 microns as well as a spectral shift. Another lab has a fall off in reflectance at high wavelengths. Some spectra are taken at very low resolution which is not necessarily a problem for average values but the peak shapes are not smooth. Noise is not as apparent as in some of the solar spectra, because FTIRs can be set to scan as many times as necessary to reduce noise, but combined with baseline drift the spectra don't quite flatten out. Nevertheless with proper use of reference mirrors to set the baseline most labs manage to approach the same value of reflectance at least for high-reflectance (low-emittance) coatings whose spectra are very flat. Almost all labs use the required mirror calibrated by the National Physical Laboratory (NPL).

Systematic errors are easier to spot looking at the summary graphs. A scan of Figures 36-41 again shows that some labs are consistently lower (or higher) for each sample. We plot two values of normal emittance: one value that is averaged only to 25 microns beyond which some labs cannot go, and another value averaged to the maximum wavelength of

the submitted data which some labs provide to 40 or 50 microns. The differences between the two values is generally quite small, at least compared to the variations among laboratories. Again, each laboratory should look at their own values which are summarized in Figures 42-49. Perhaps the best overall way to assess the spread in the data is in Figures 50-53 by property. Table 6 boils down the data from Figures 50-53 still further.

Table 6. Summary of errors for emittance data.

Sample	1	2	3	4	5	6
E_5-25 Mean	89.7618	60.7541	97.6756	82.8465	96.8743	10.2179
E_5-25 Stdev	0.7360	2.2097	1.4686	1.7366	0.4769	0.5939
E_lmax Mean	89.8346	60.9160	97.7574	83.0195	96.9297	10.3549
E_lmax Stdev	0.7215	2.2334	1.4194	1.8002	0.4060	0.6850

The numbers here are well out of the range where we would like to be. This is not surprising given the rapid and unfavorable changes in available instrumentation while our standards remain static. The Thermes project hypothesized that two factors were chiefly responsible for the deterioration in measurement accuracy with FTIRs: nonuniformity in the use of reference mirrors and instrument stability. Therefore they conducted an ILS which was an experiment to see how these two factors, if controlled, would improve the results. In our case the reference mirror is not a major factor because there is only one place to get a traceable reference mirror, i.e., NPL and we specified that in our NFRC 301 standards long ago. Almost all of our participants use this mirror. The other factor, stability, is a serious problem to us, which Thermes addressed by designing a sequence of measurements including frequent recalibration of the baseline.

Conclusions and Recommendations

It has been argued that accuracy on the order of 1% for optical properties is more than adequate for comparing the energy performance of fenestration products considering the higher levels of uncertainty in other factors that go into the final determination. There are some cases in which a higher accuracy is desired, say a few tenths of a percent, for calculations with laminates and applied films that involve deconstruction of the glazing. We are not currently achieving either of these levels consistently, except perhaps in solar transmittance, but other ILCs and fundamental considerations indicate that it is possible. We should be able to make rapid improvement by the following steps:

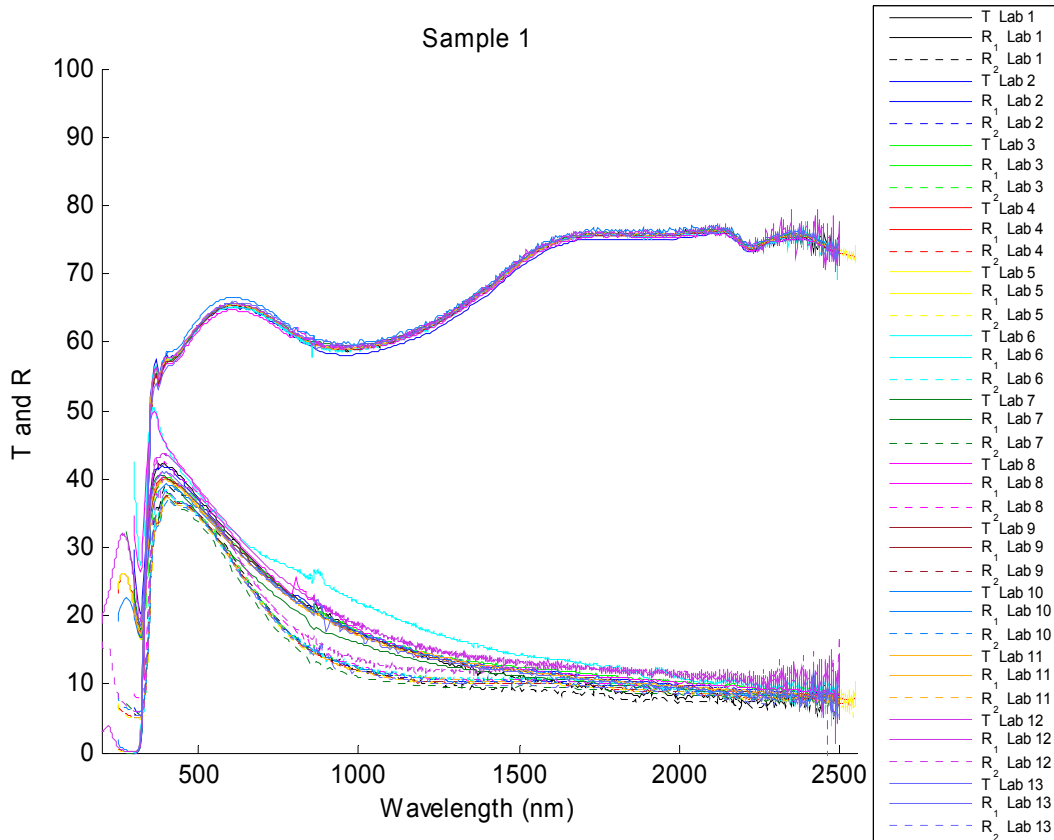
1. Discussions will be held with each lab to identify specific sources of error. Replacement of reference mirrors, review of baseline correction procedures, and better choices for scan parameters should result in significant improvement in a matter of weeks.
2. A rapid follow-up ILC using simultaneous uniform samples will verify progress.
3. A 1-2 day workshop will be held at LBNL in the early summer for all participants along the lines of a previous successful workshop. Invited guests will include a representative of Thermes and a Perkin-Elmer and/or Varian applications specialist.
4. Revise our standards based on the workshop outcomes and in harmony with ISO and CEN standards. For the infrared ideally we would adopt the new CEN standard which will be based on the Thermes recommendations.

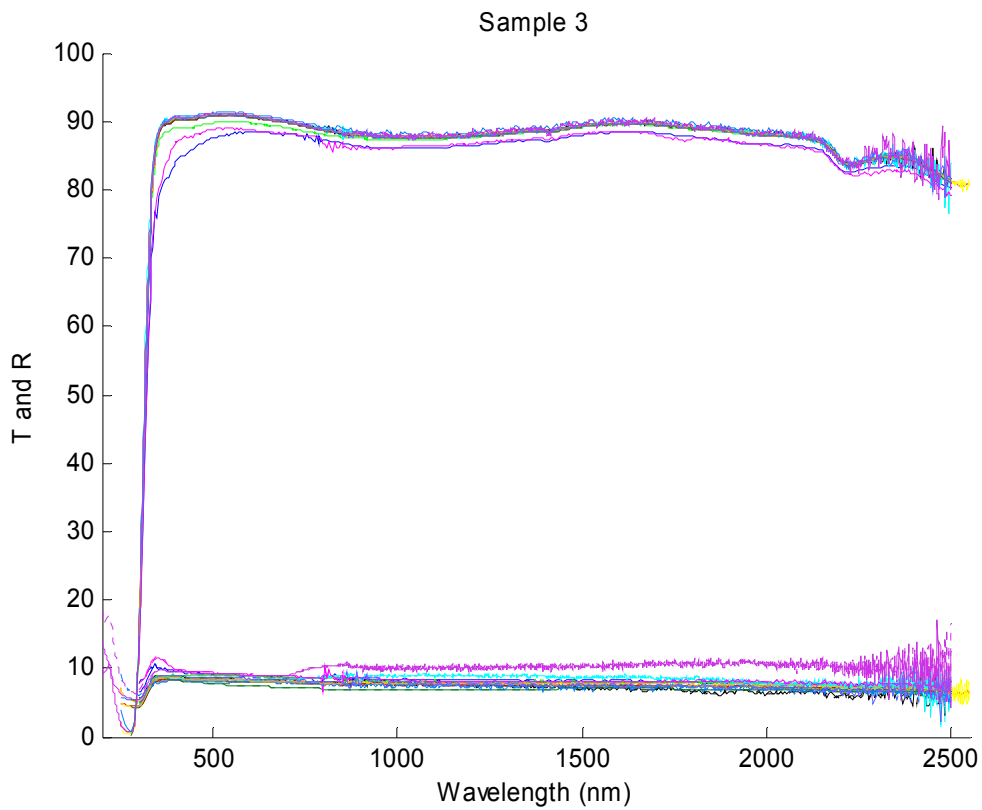
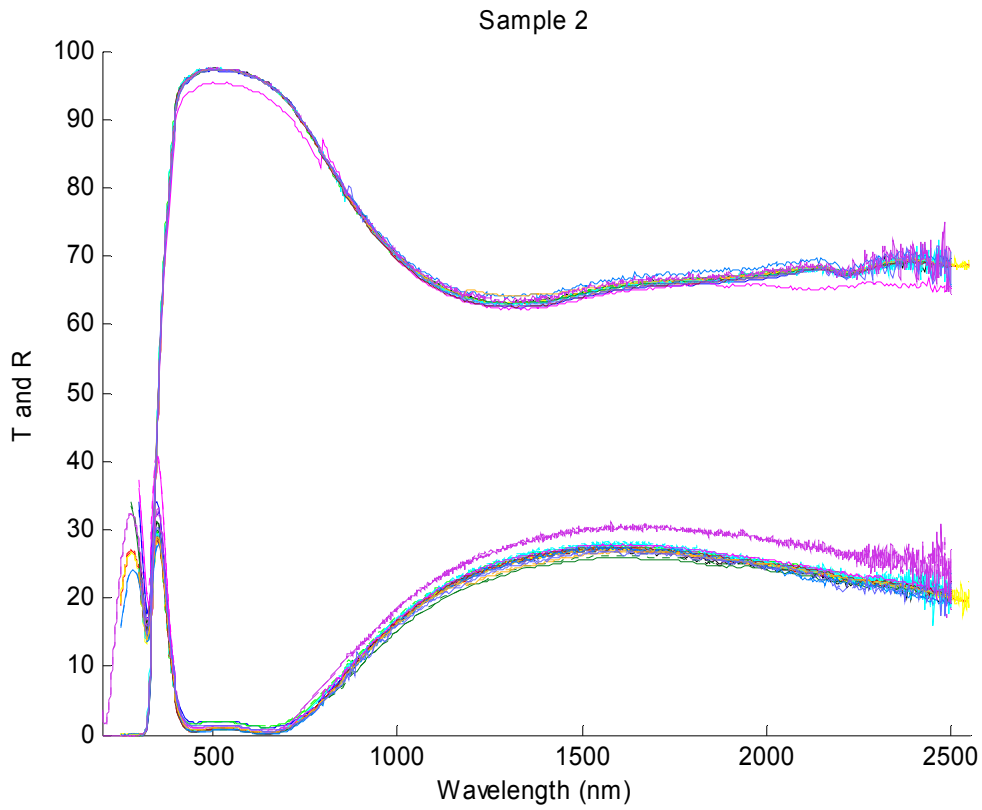
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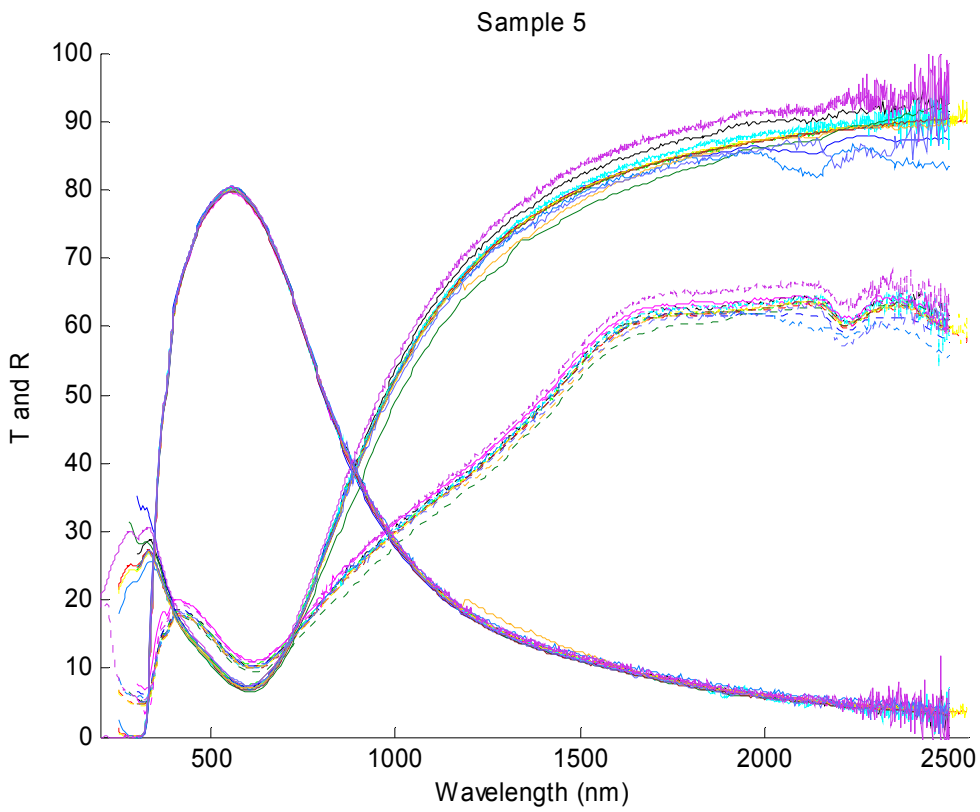
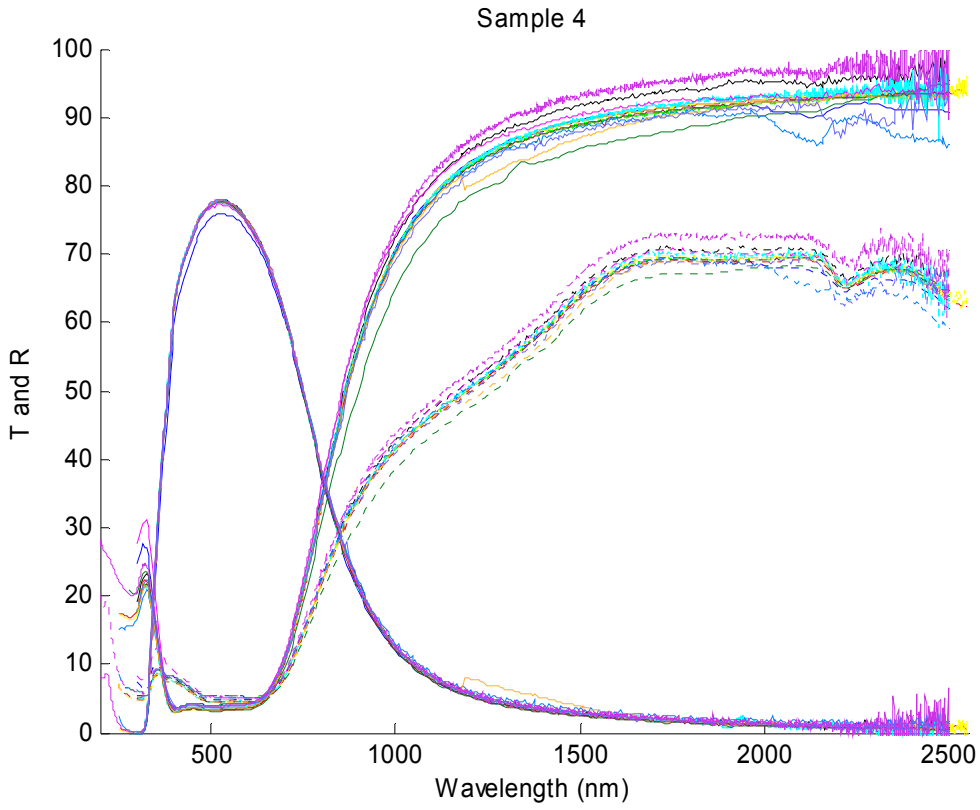
This work was supported by the Assistant Secretary for Energy Efficiency and Renewable Energy, Building Technologies Program, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

Solar-Range Figures

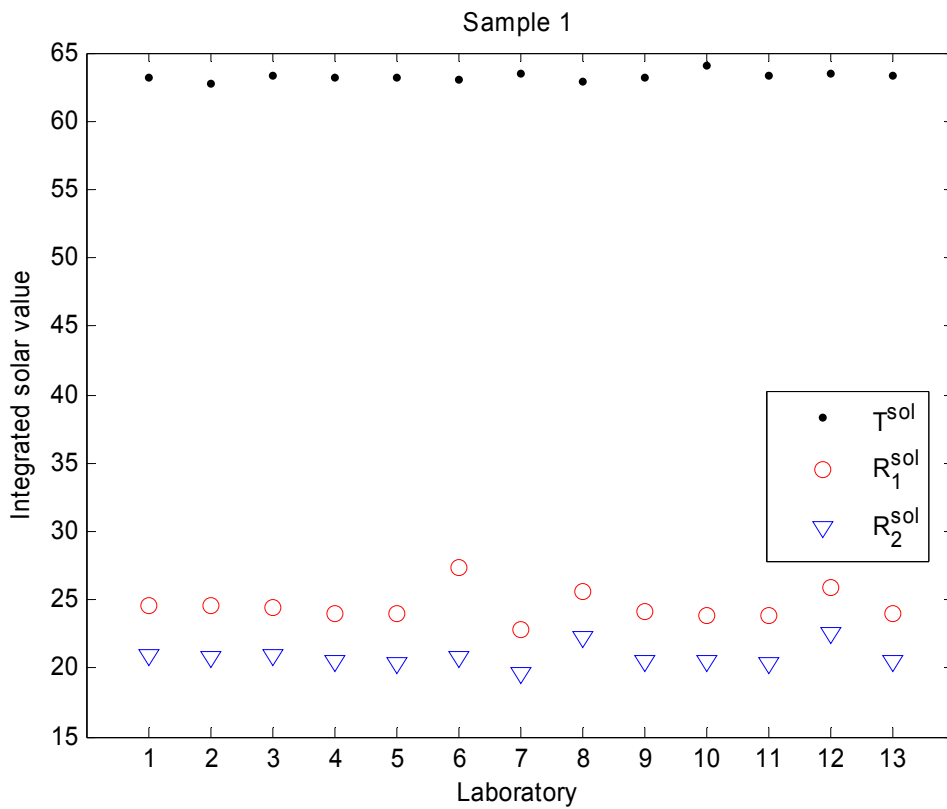
Figures 1-5. Each figure represents one sample. For each laboratory the transmittance and reflectance spectra are plotted over the solar range. All three spectra for each laboratory are plotted in the same color as shown in the key below. Reflectance from the second side is plotted as a dotted line of the same color to avoid confusion in cases where the reflectance is of the same order from each side.

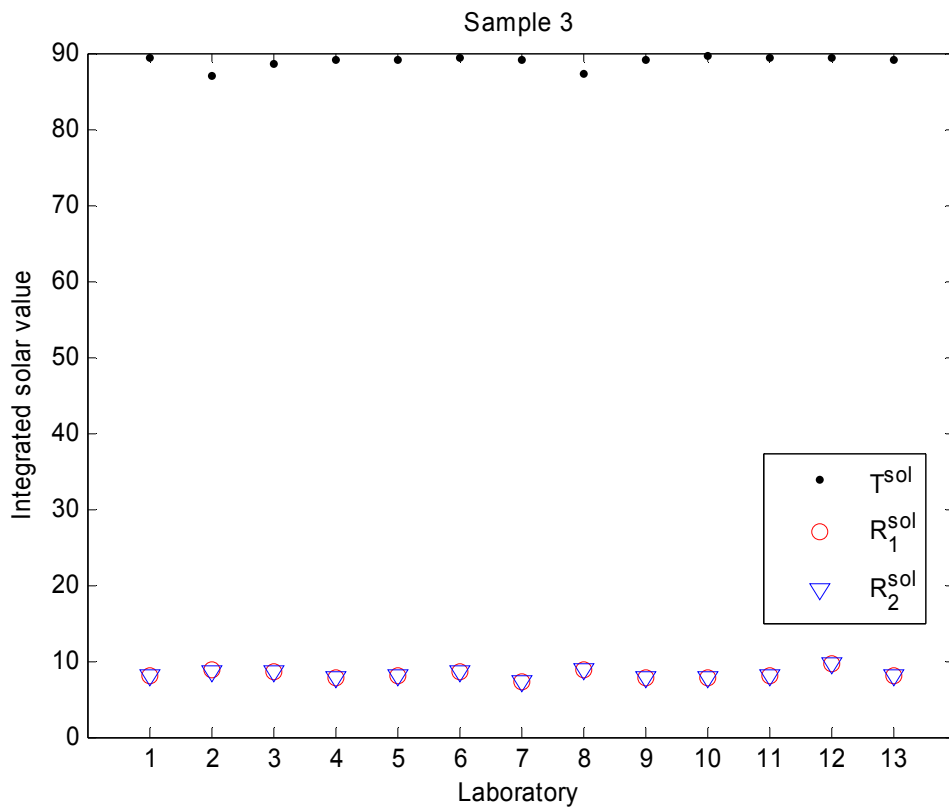
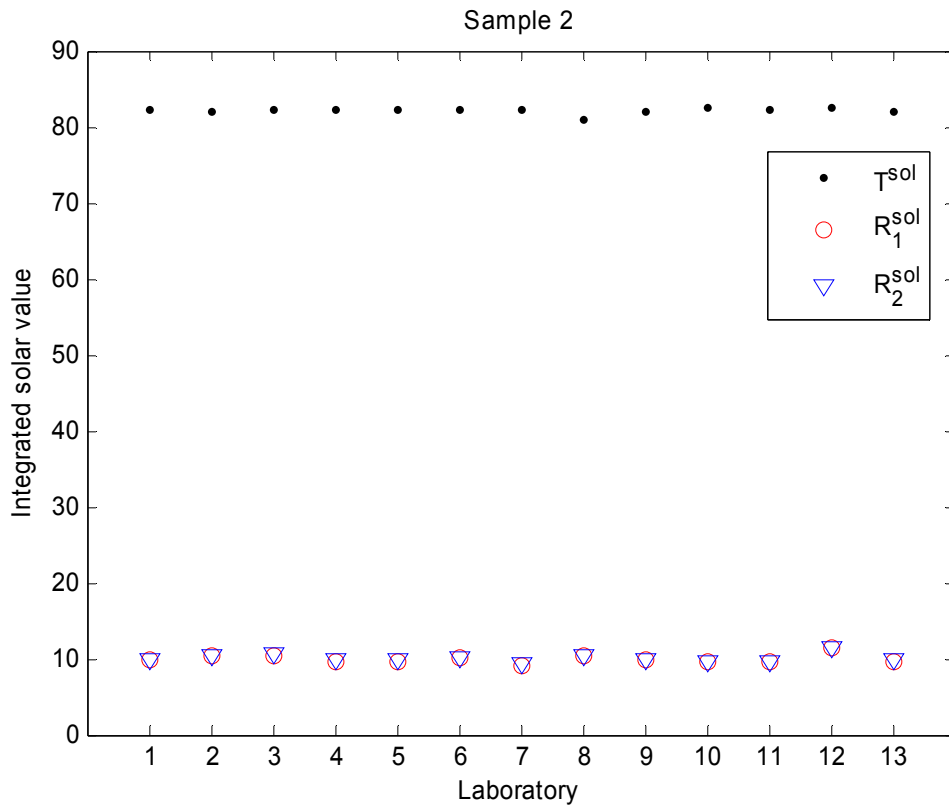


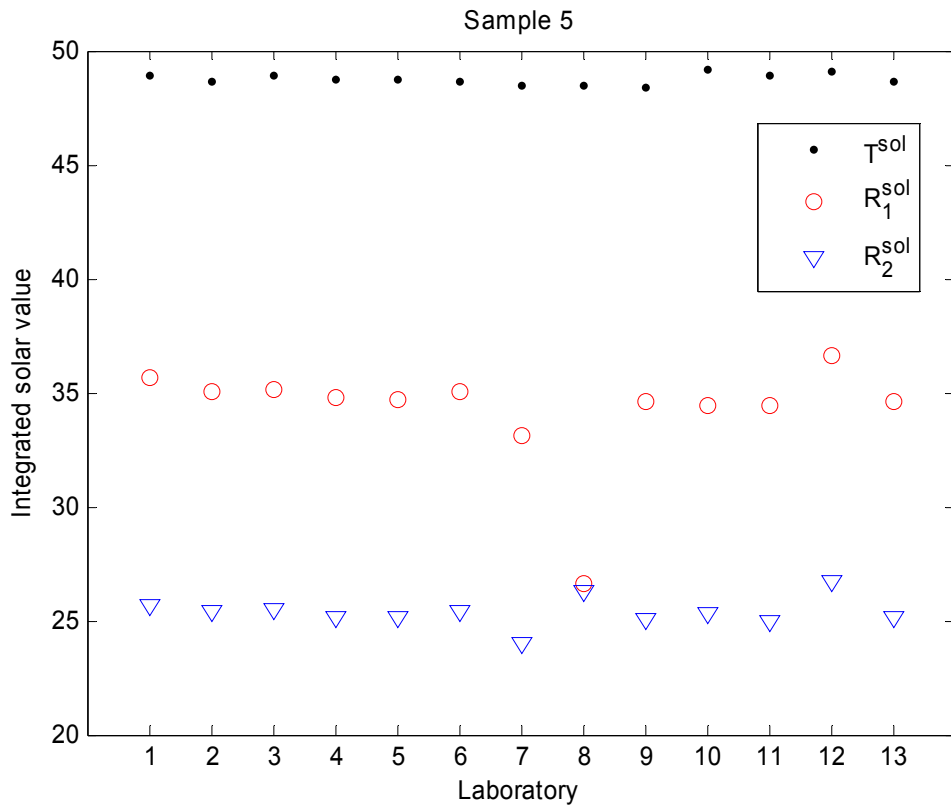
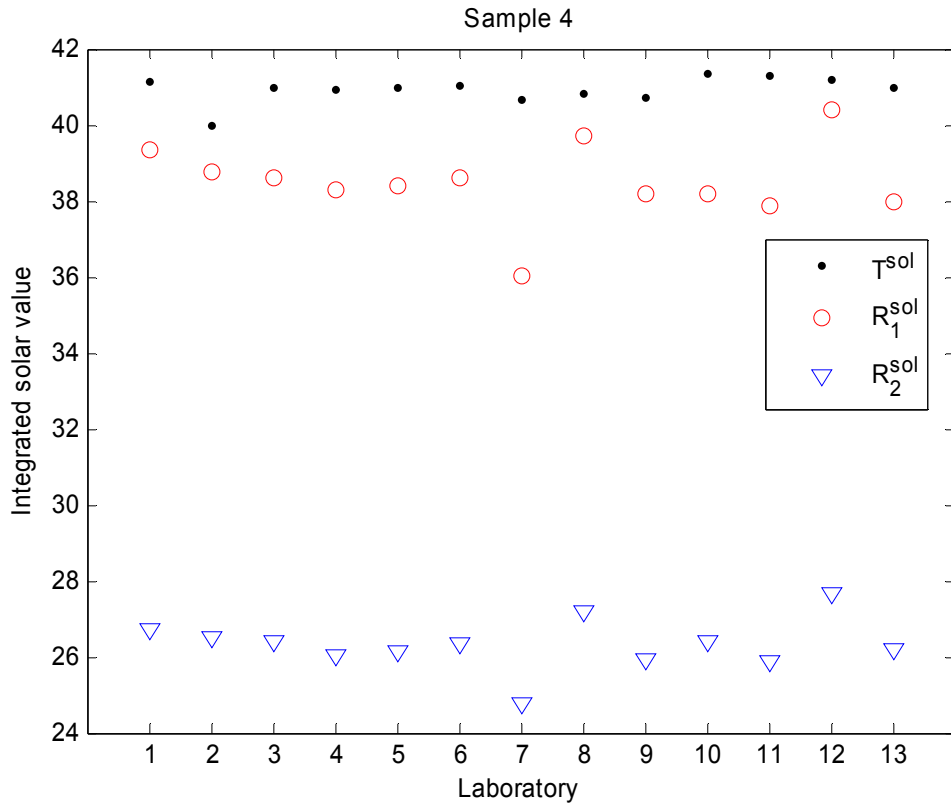




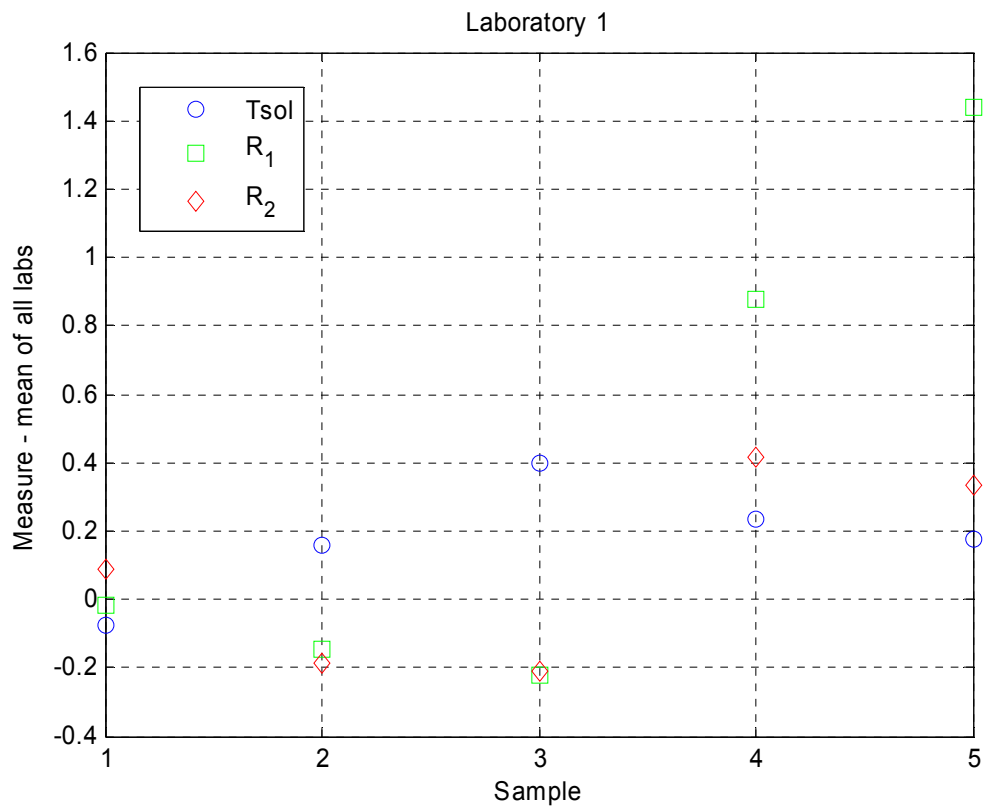
Figures 6-10. Each figure represents one sample and shows for each laboratory the weighted average solar transmittance and reflectance from side 1 and side 2. The average values were calculated using a standard procedure by LBNL from the raw data provided by each laboratory so that the differences are due only to the measured values not to the calculation procedure. Open symbols are used for reflectance so that for symmetric samples (sample 2 and sample 3) the symbols do not overlap.

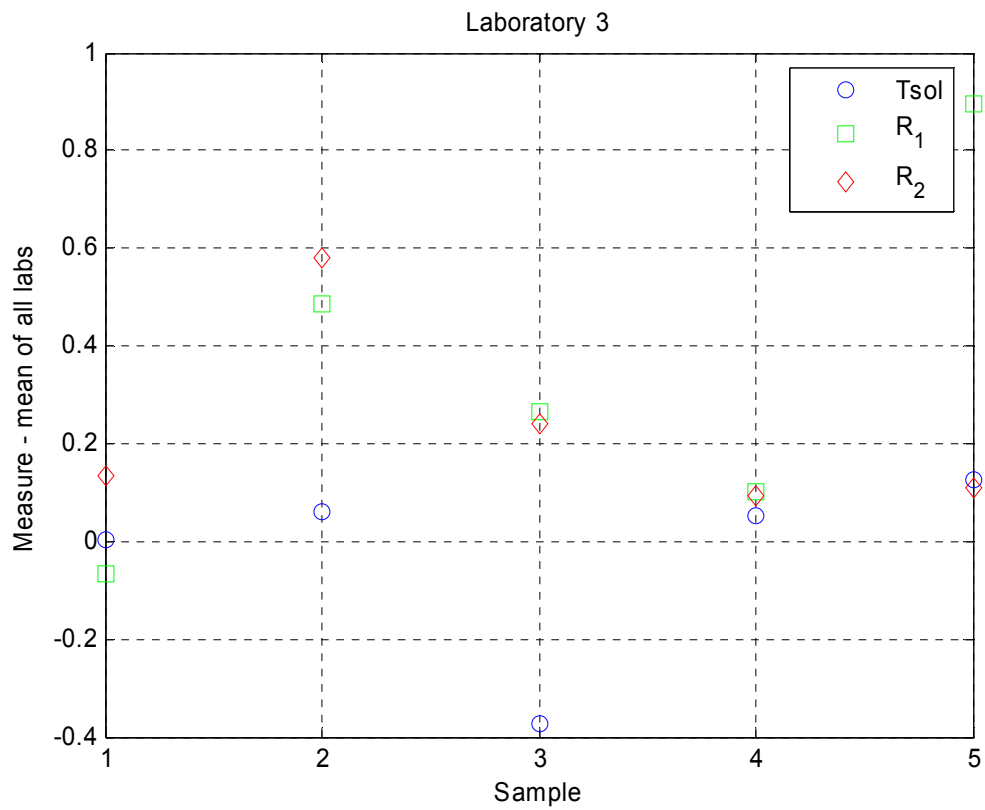
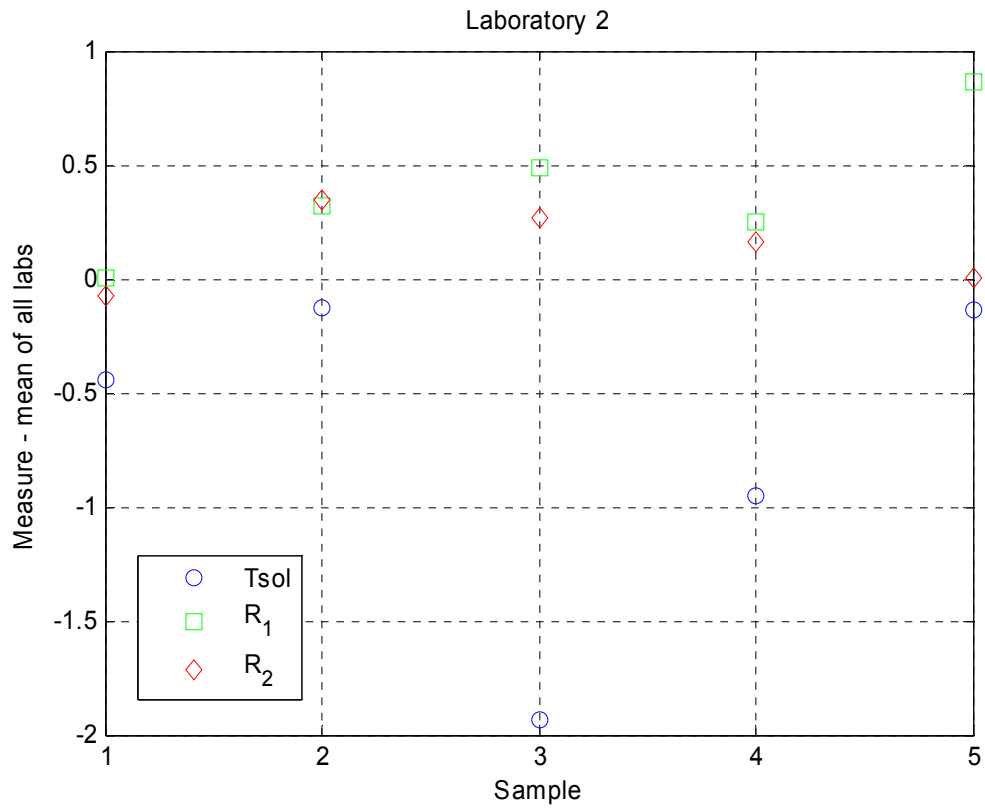


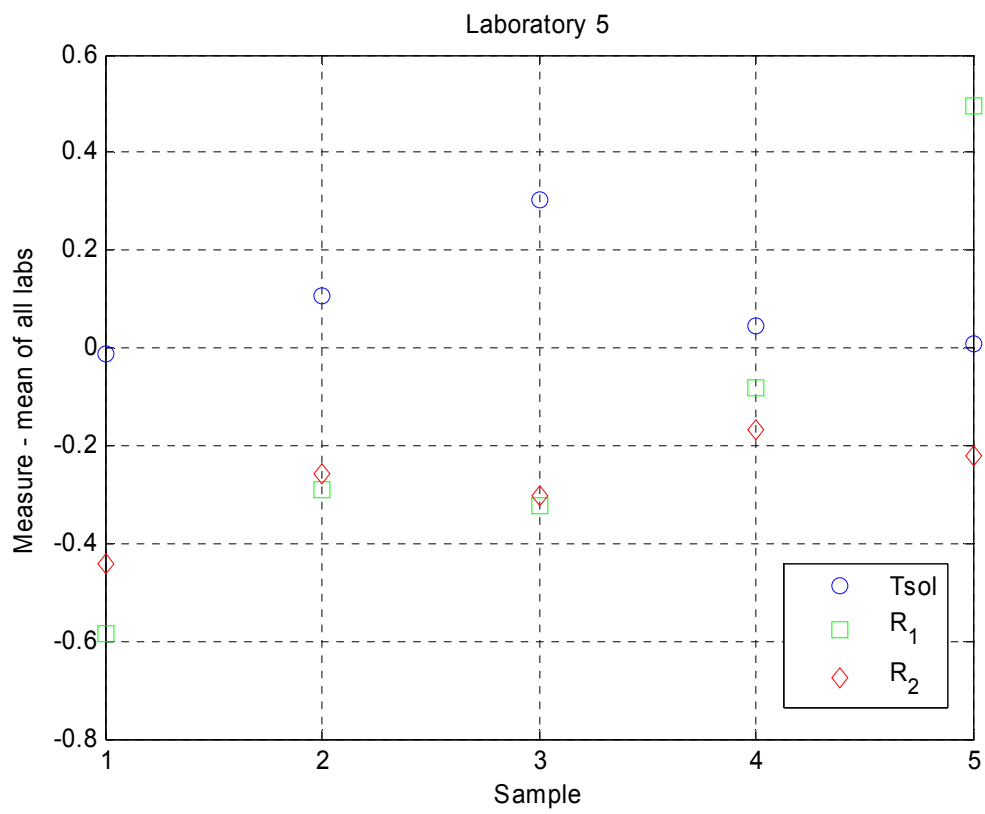
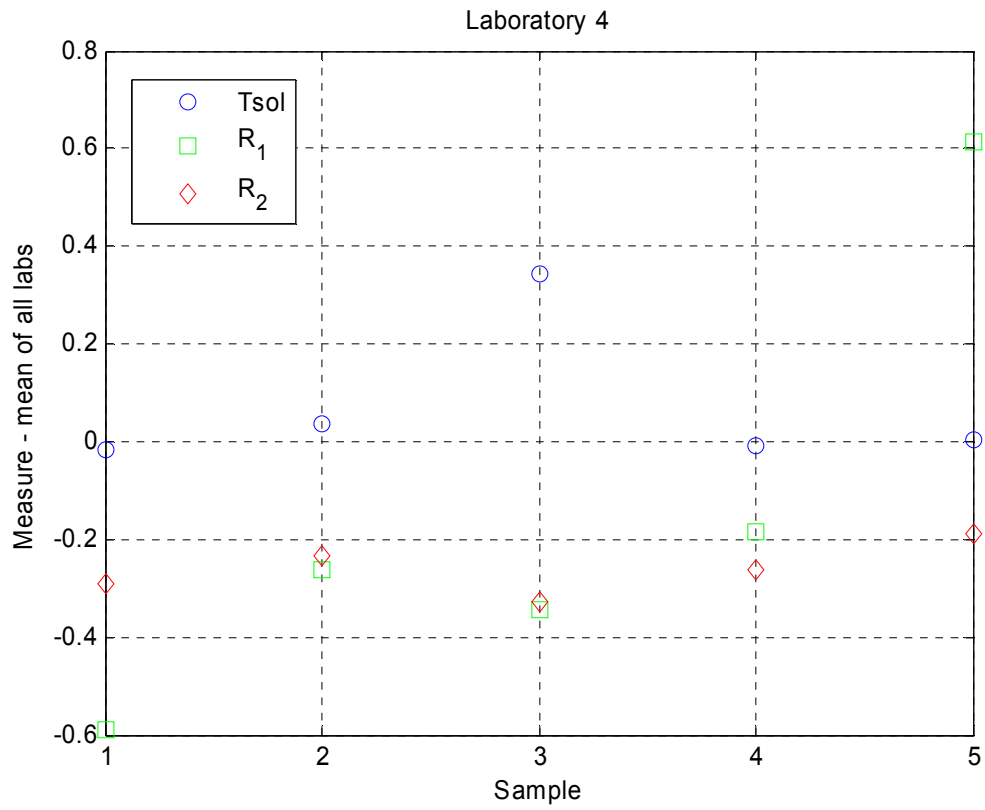


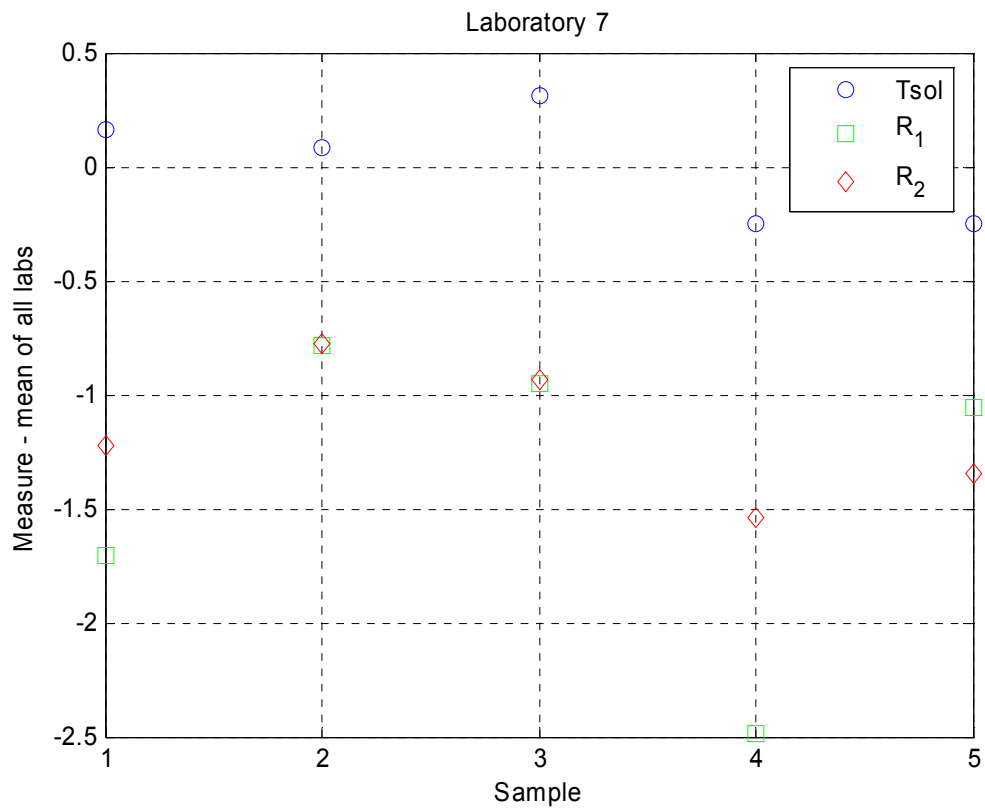
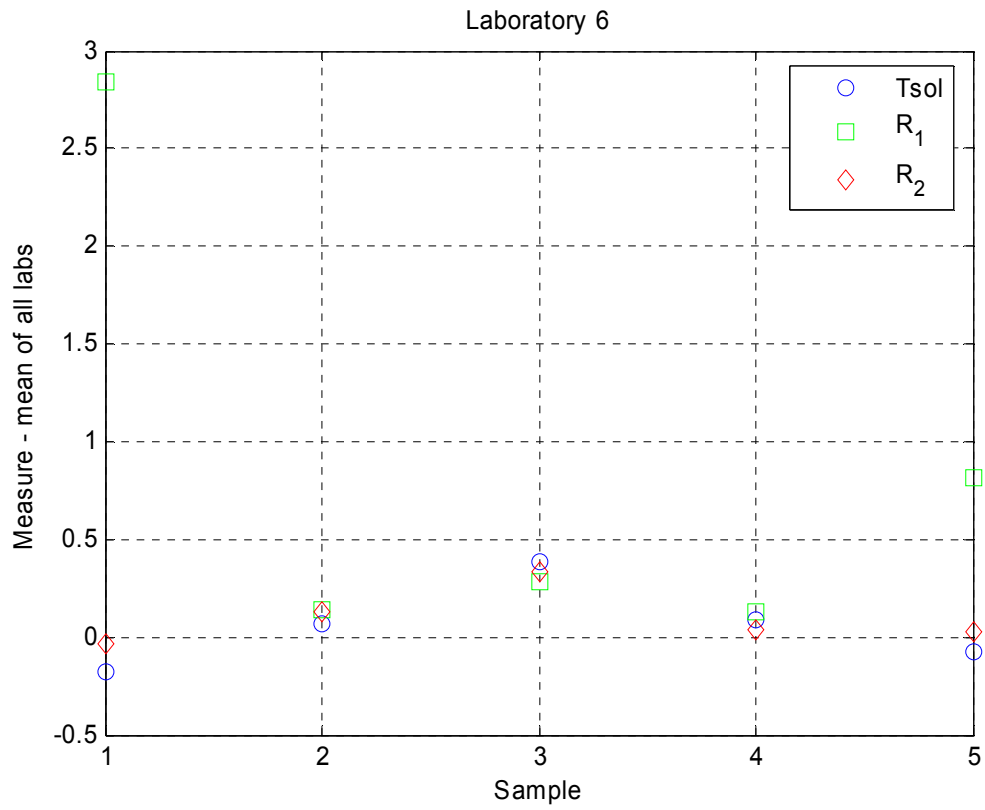


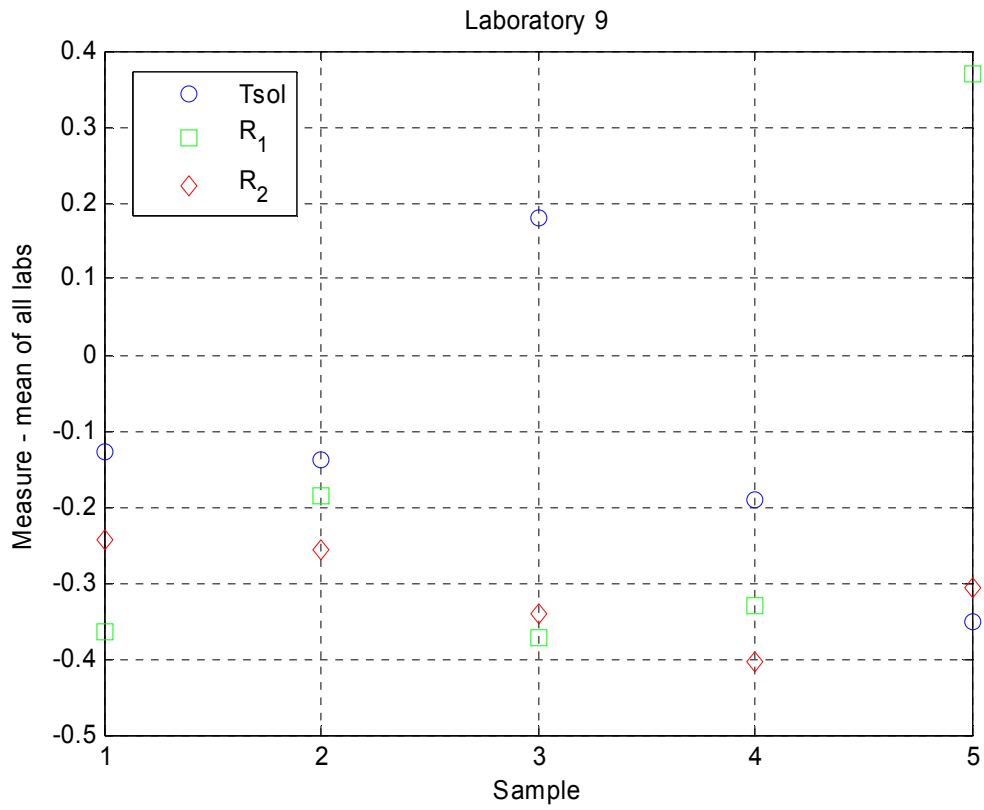
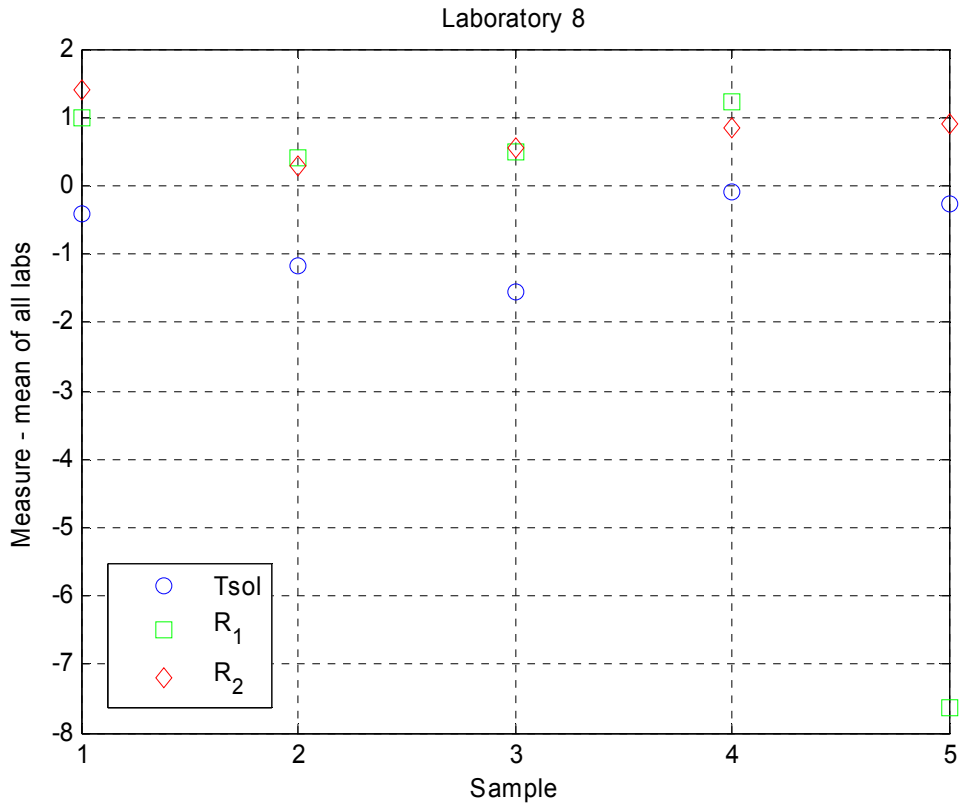
Figures 11-23. Each figure summarizes the results in the solar spectrum for one laboratory. The three solar properties for each sample are plotted as their deviation from the mean value for all laboratories.

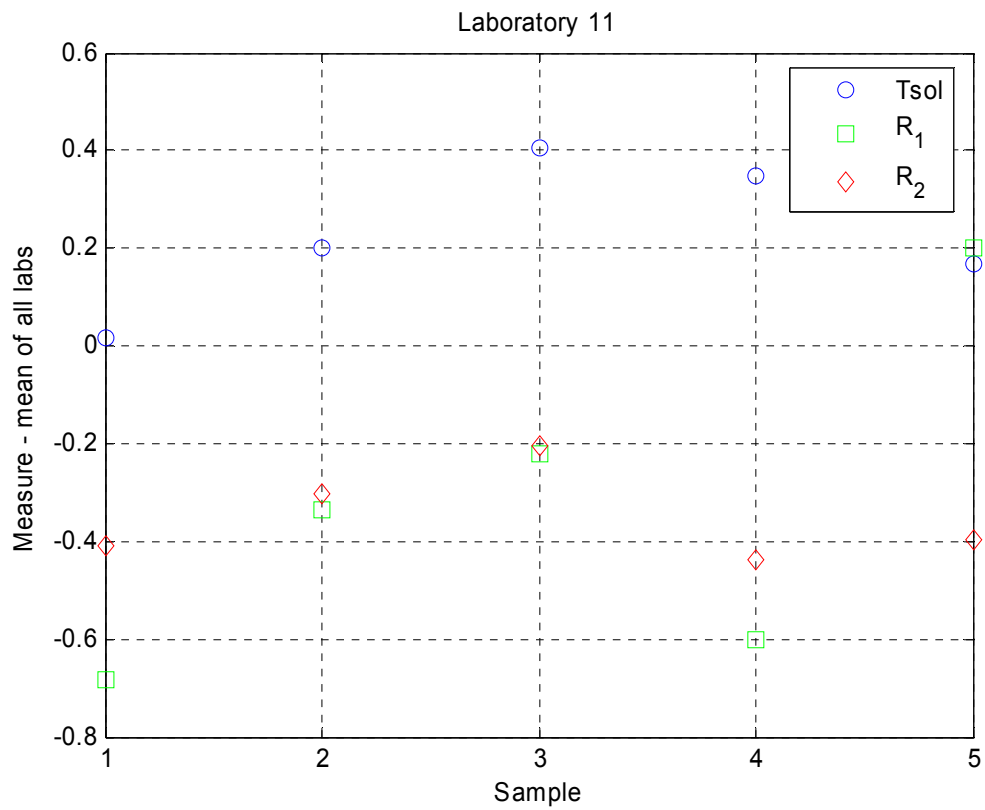
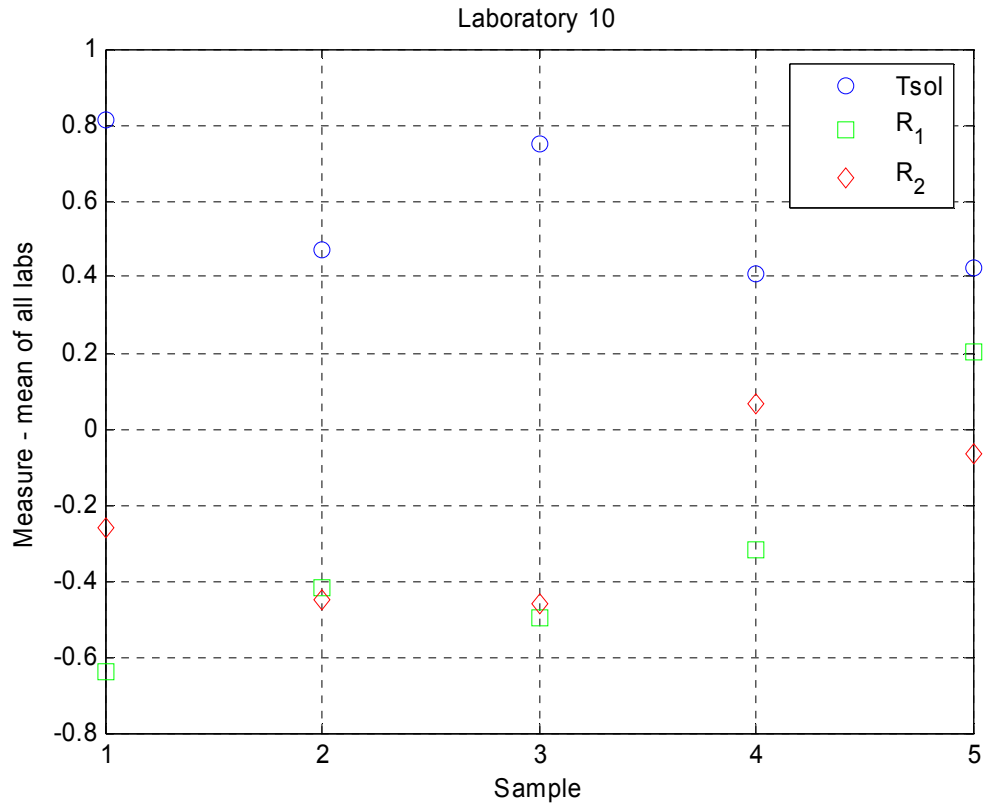


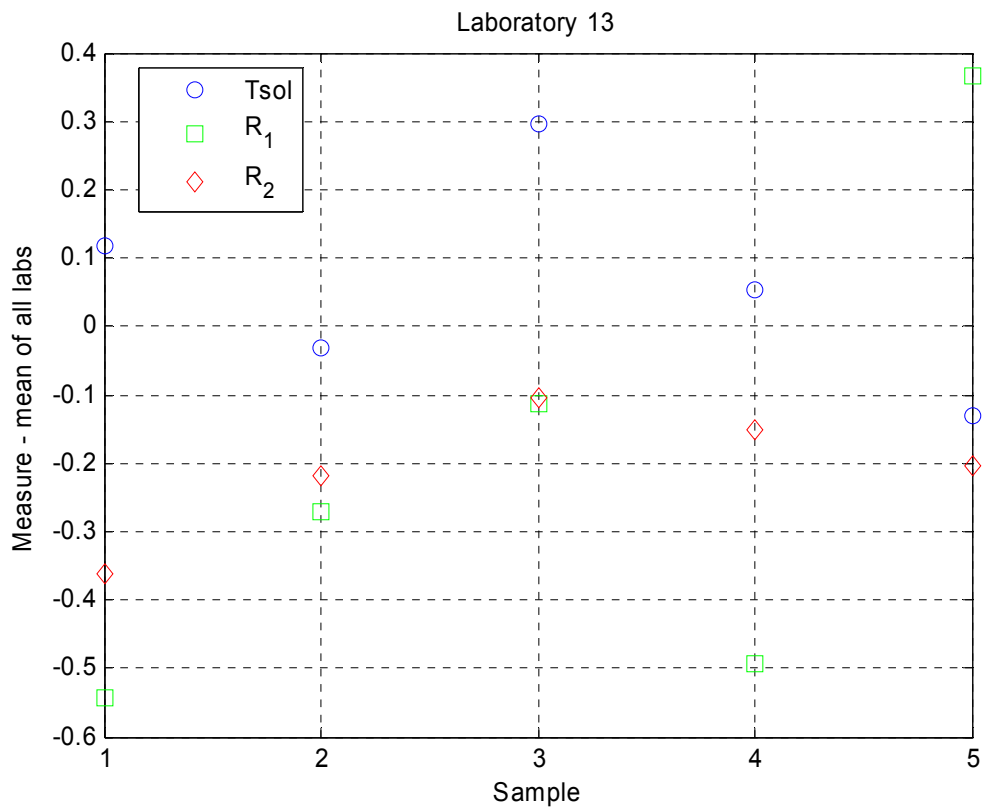
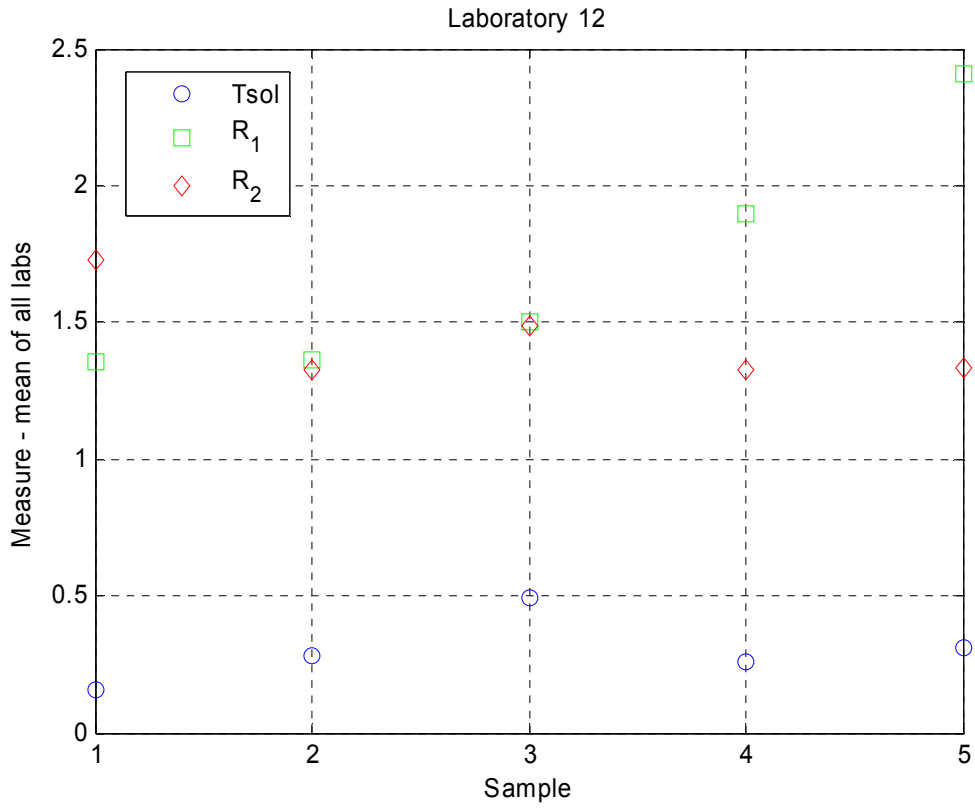




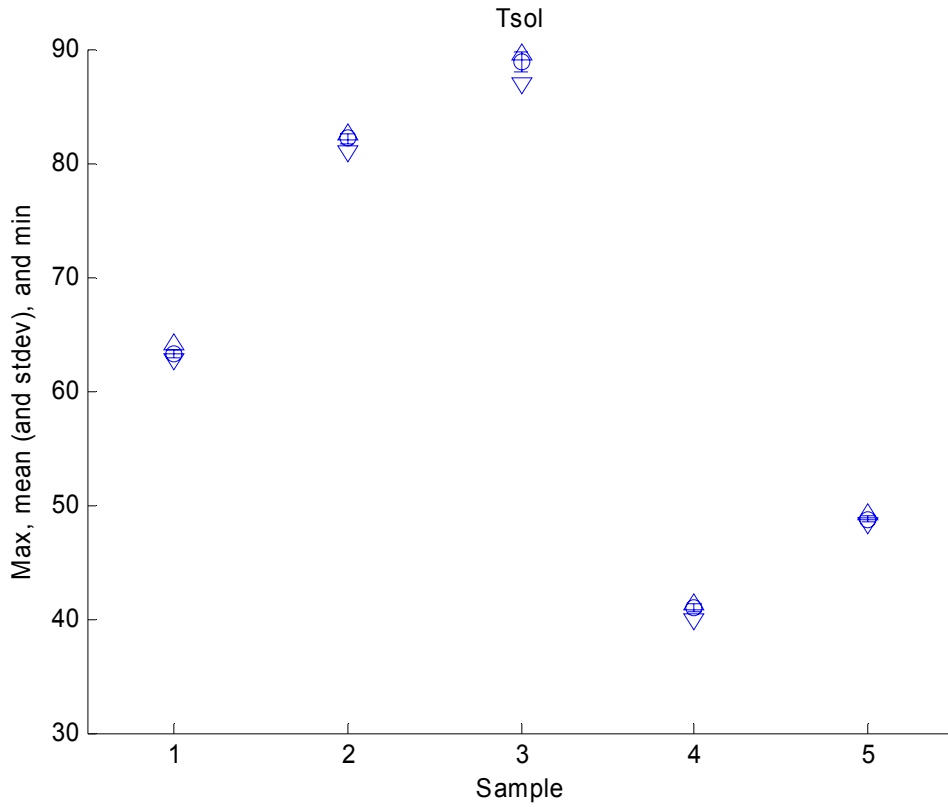


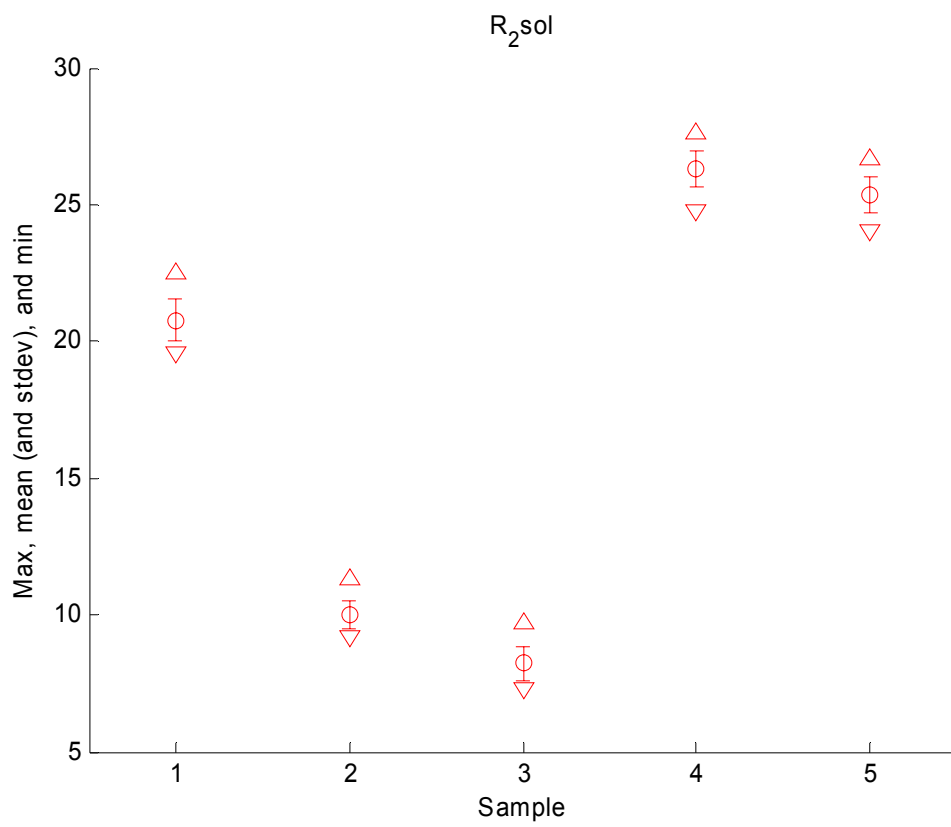
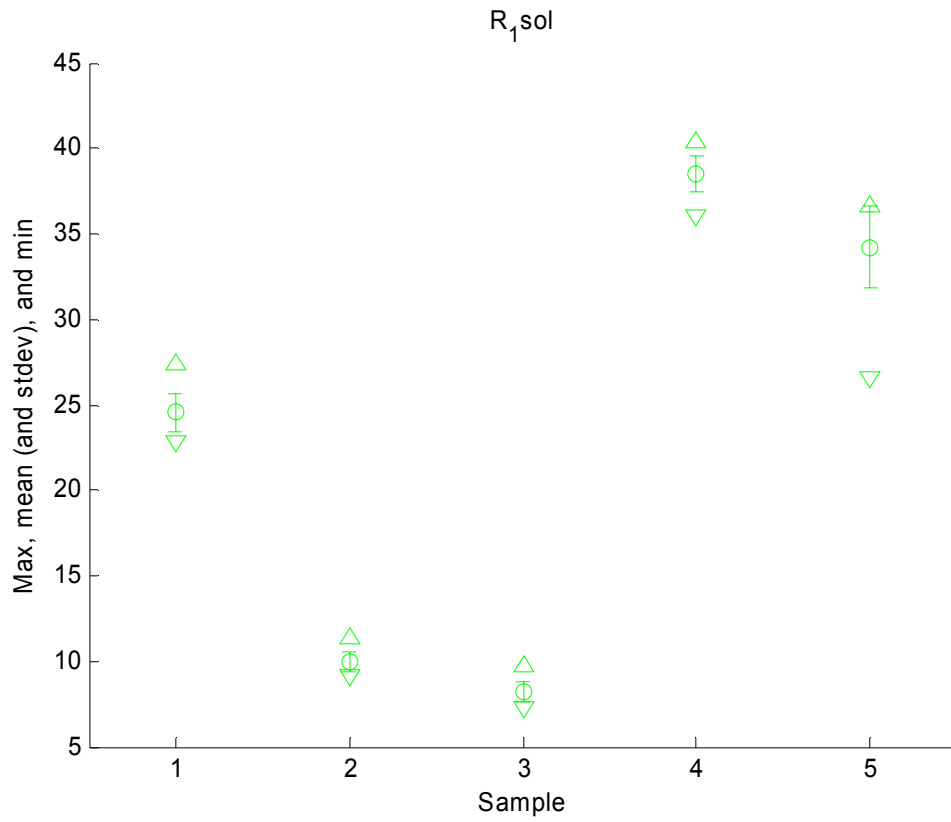


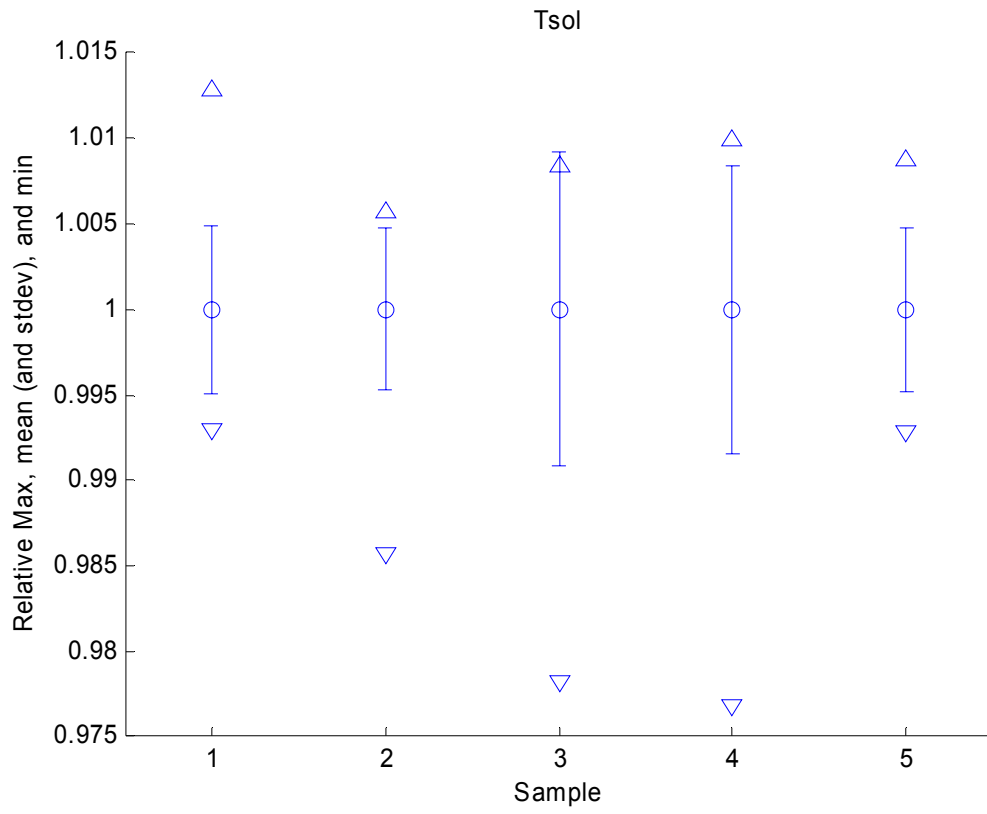


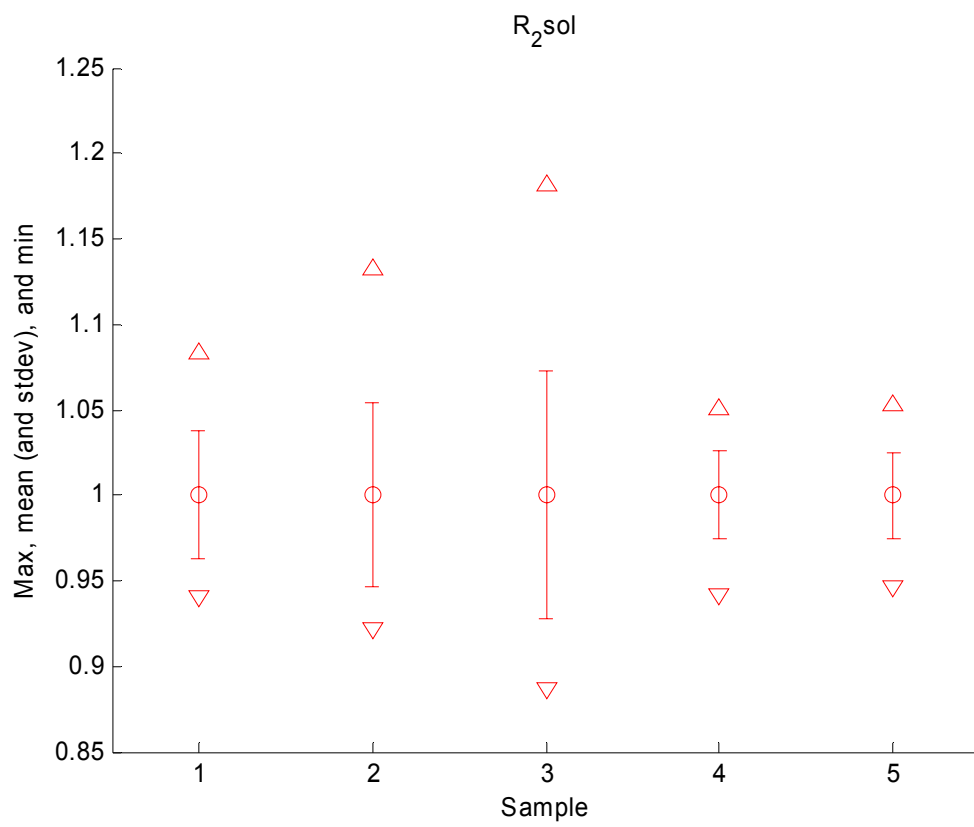
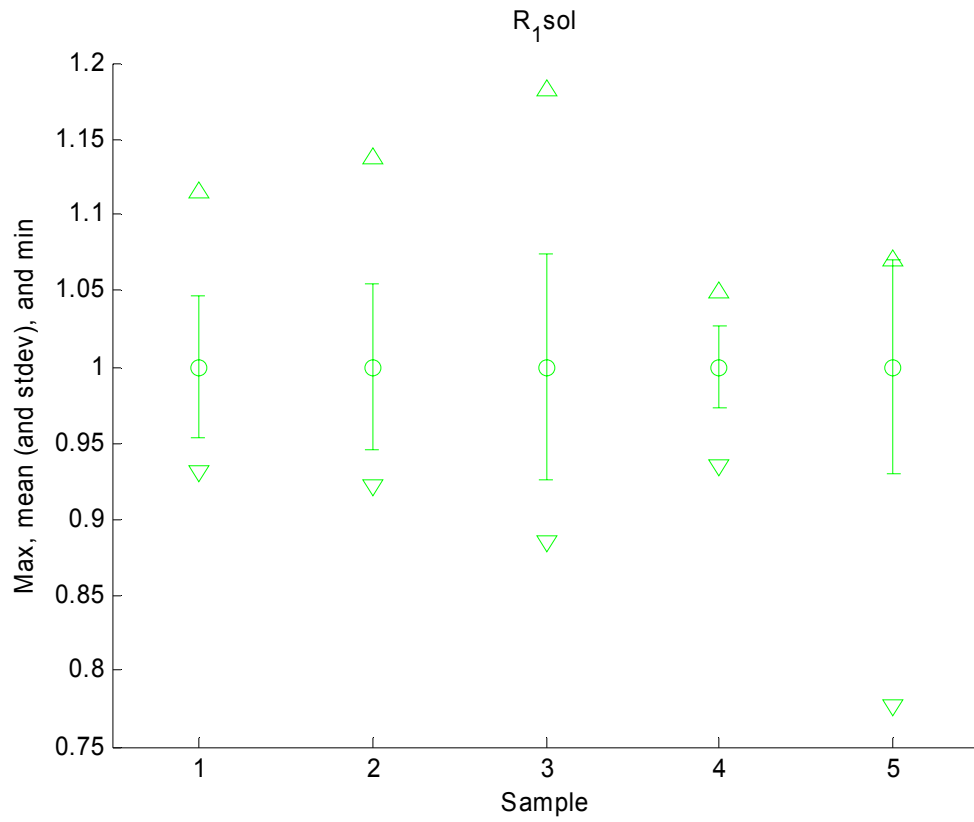


Figures 24-29. Each pair of graphs represents a given solar property, T, R1 and R2. The first graph in the pair give the maximum, minimum and standard deviation over all labs for each sample. The second graph in each pair presents the same values normalized to the mean value.



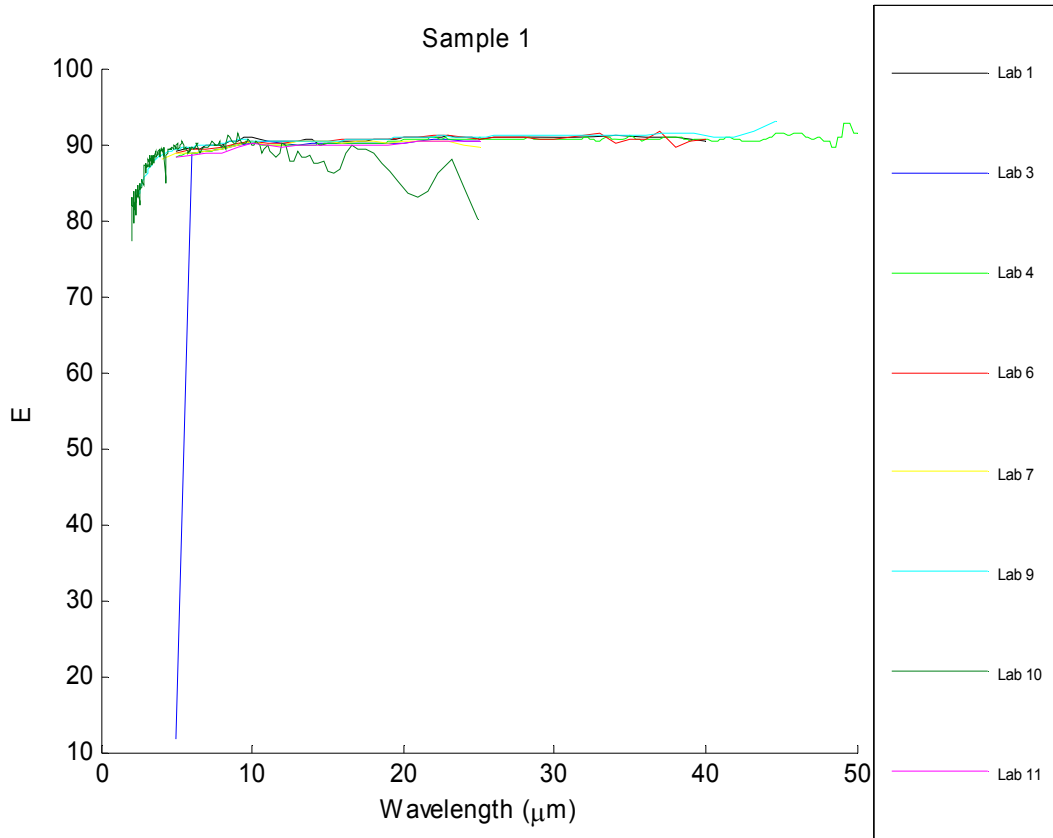


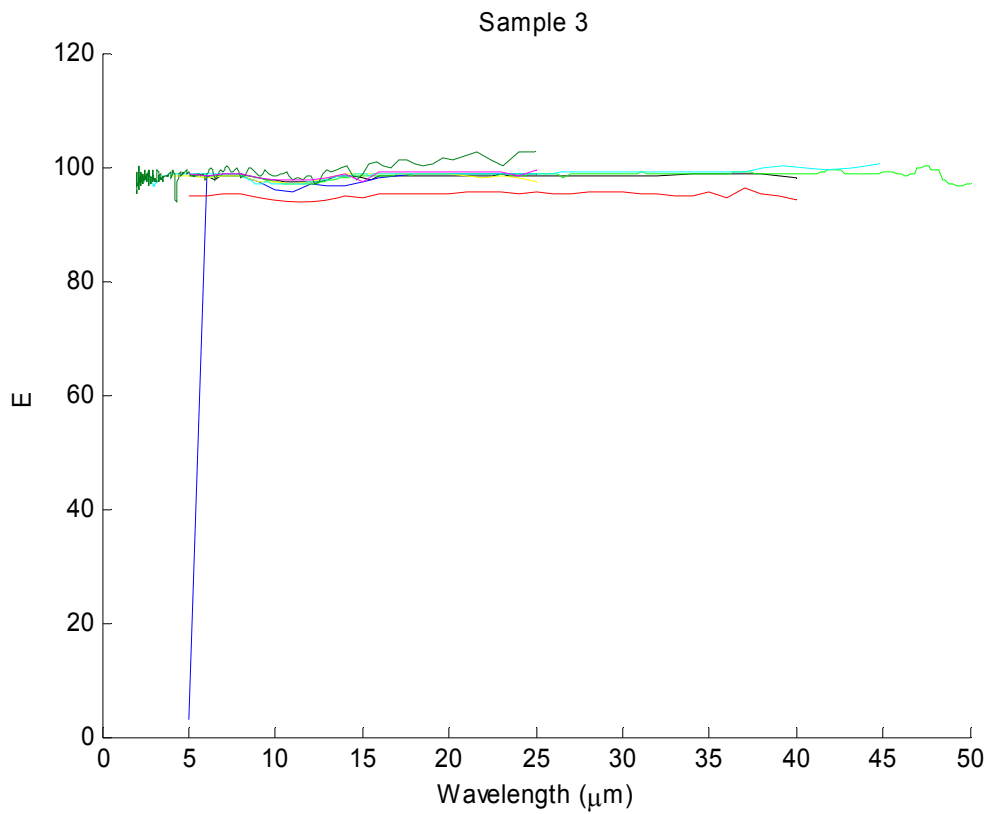
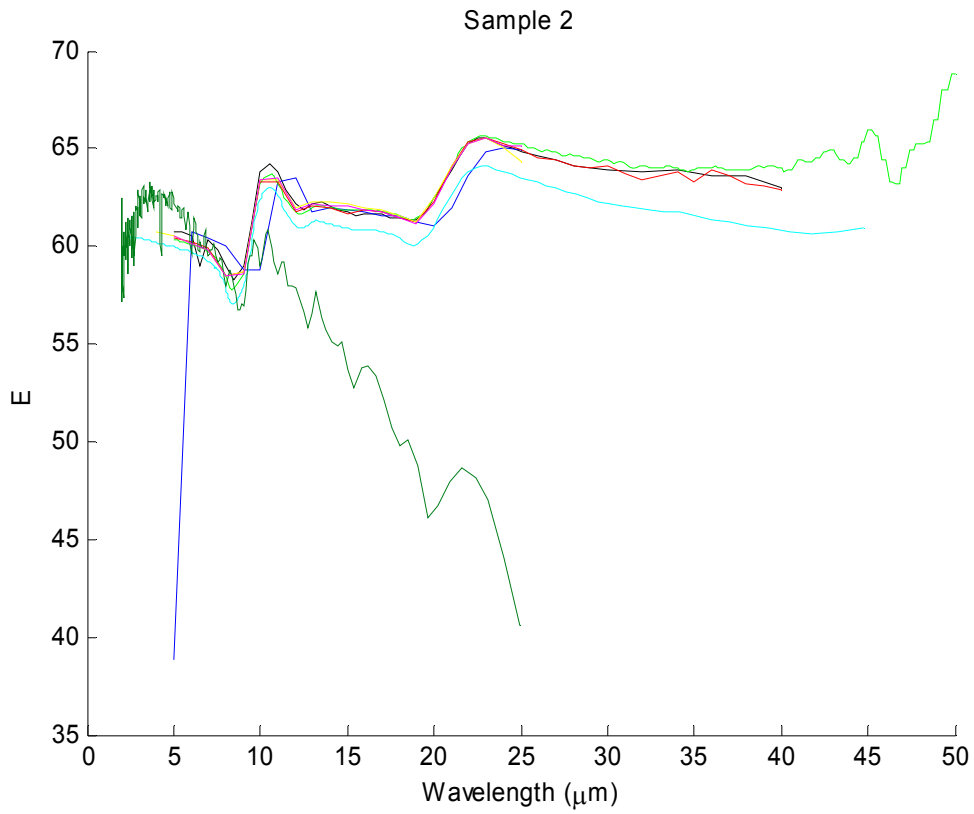


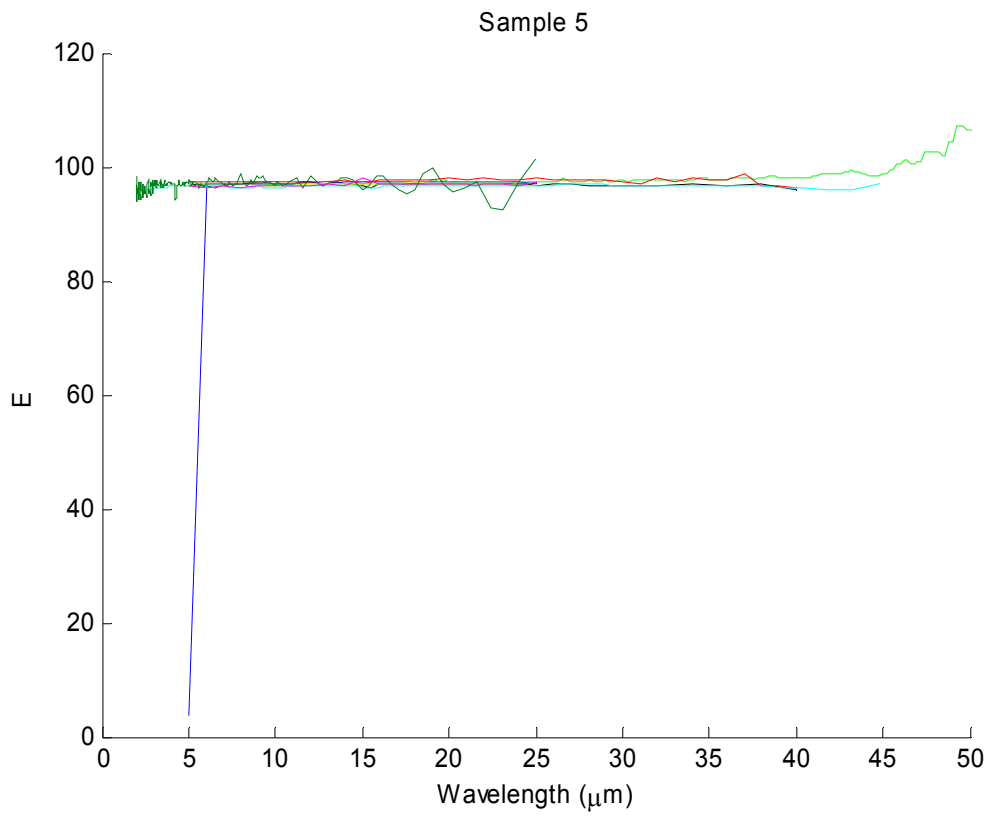
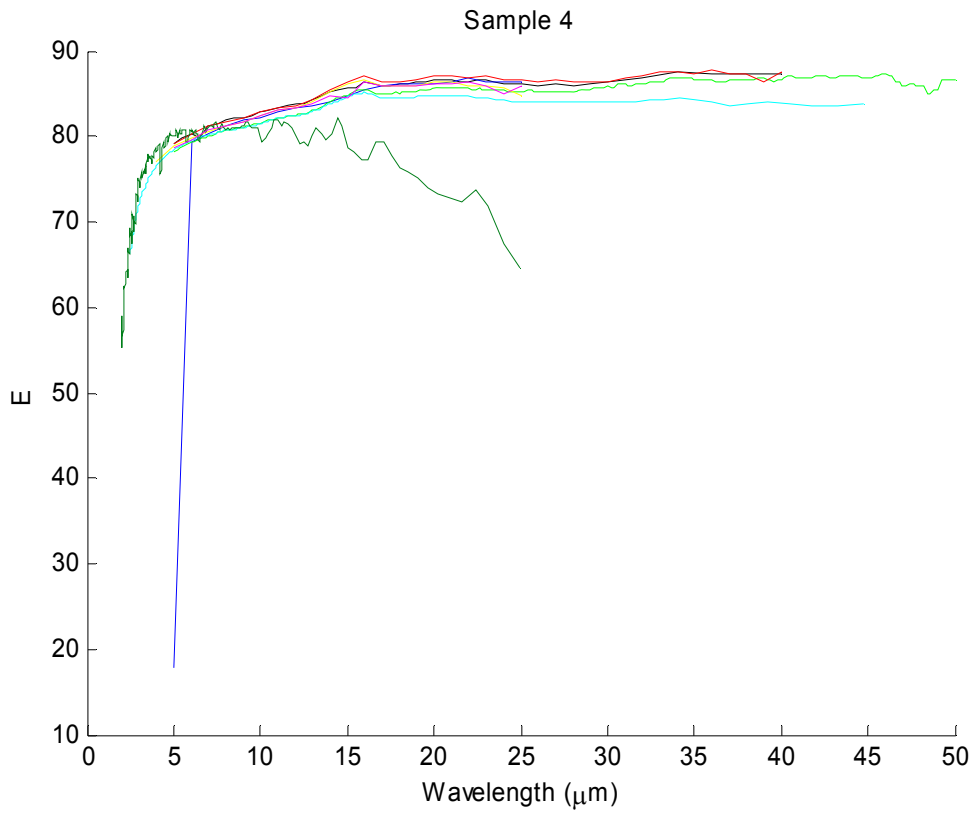


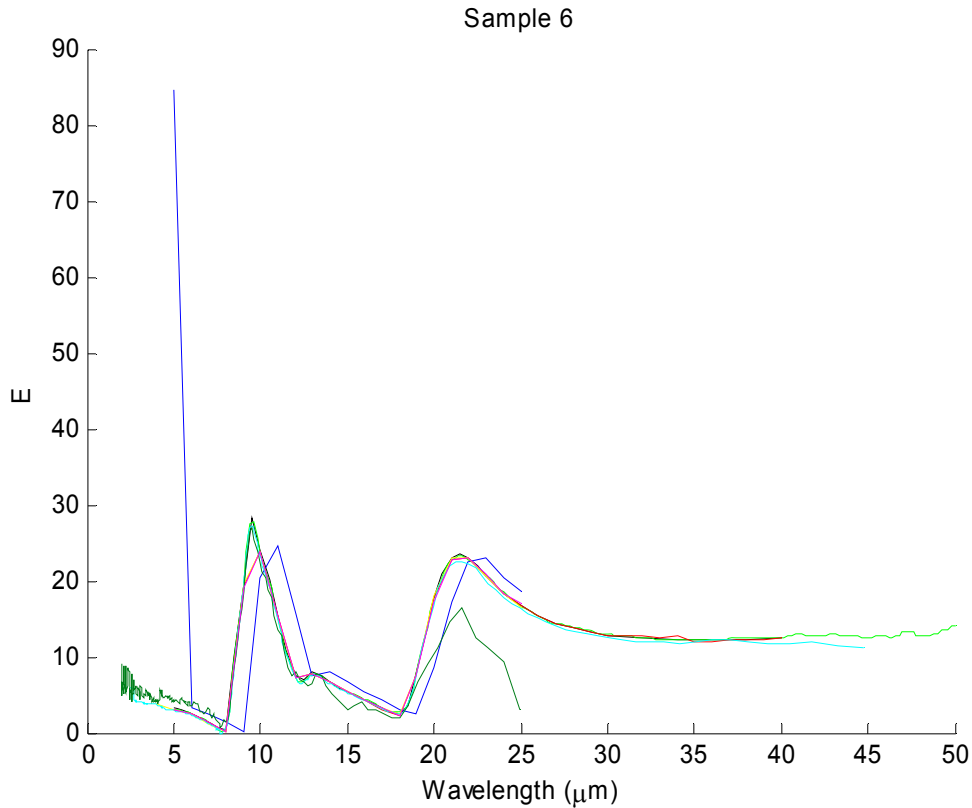
Infrared-Range (Emittance) Figures

Figures 30-35. Each figure represents one sample. For each laboratory the reflectance spectrum is plotted over the thermal-infrared range. The spectra for each laboratory are plotted in the color shown in the key below.

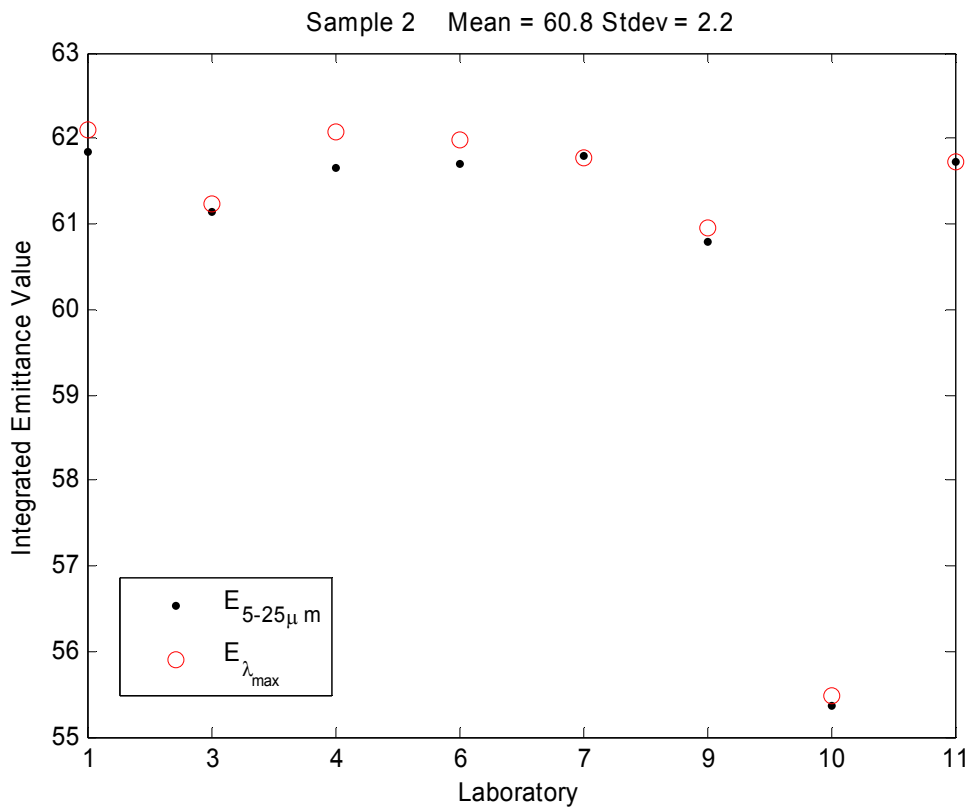
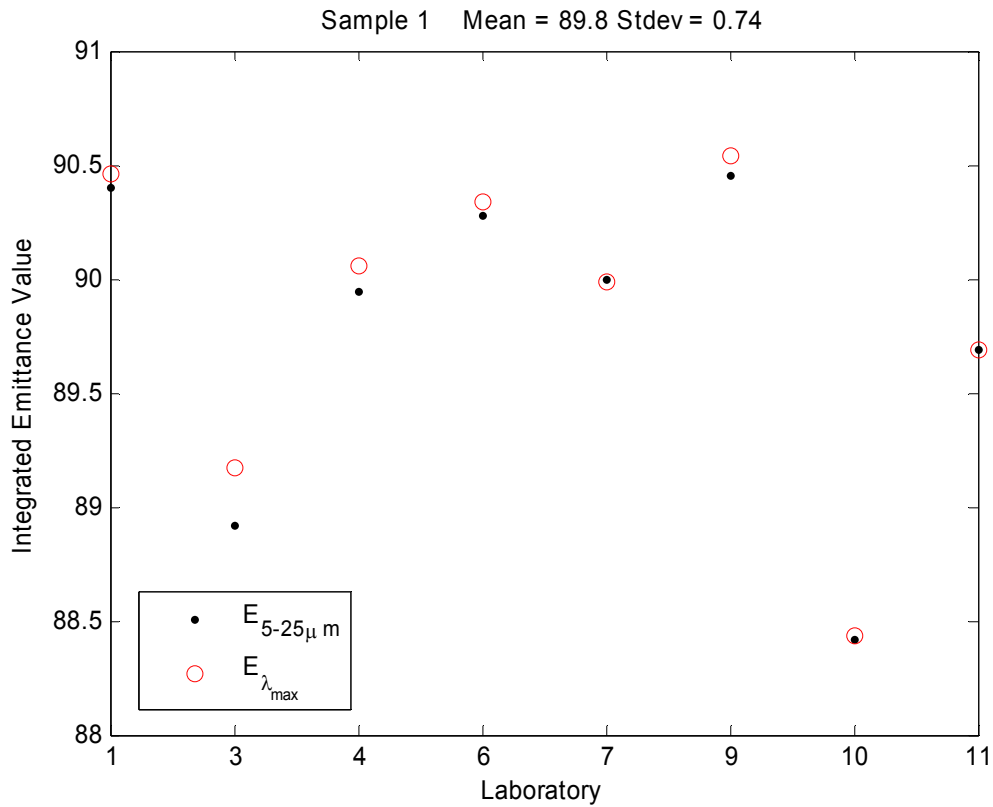


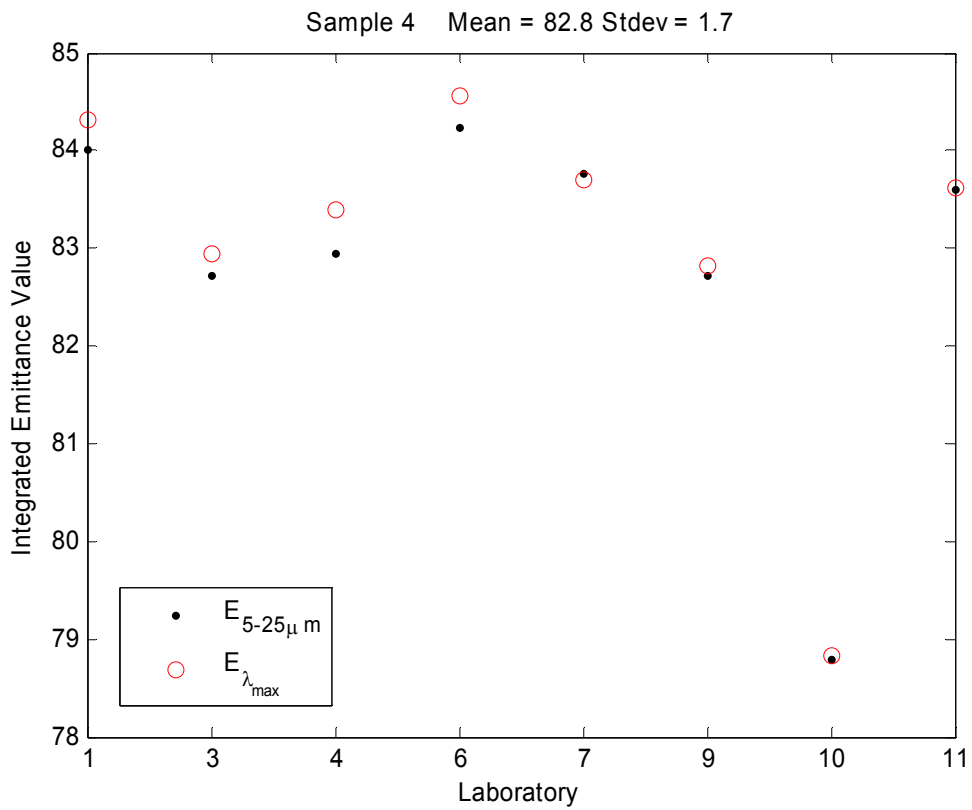
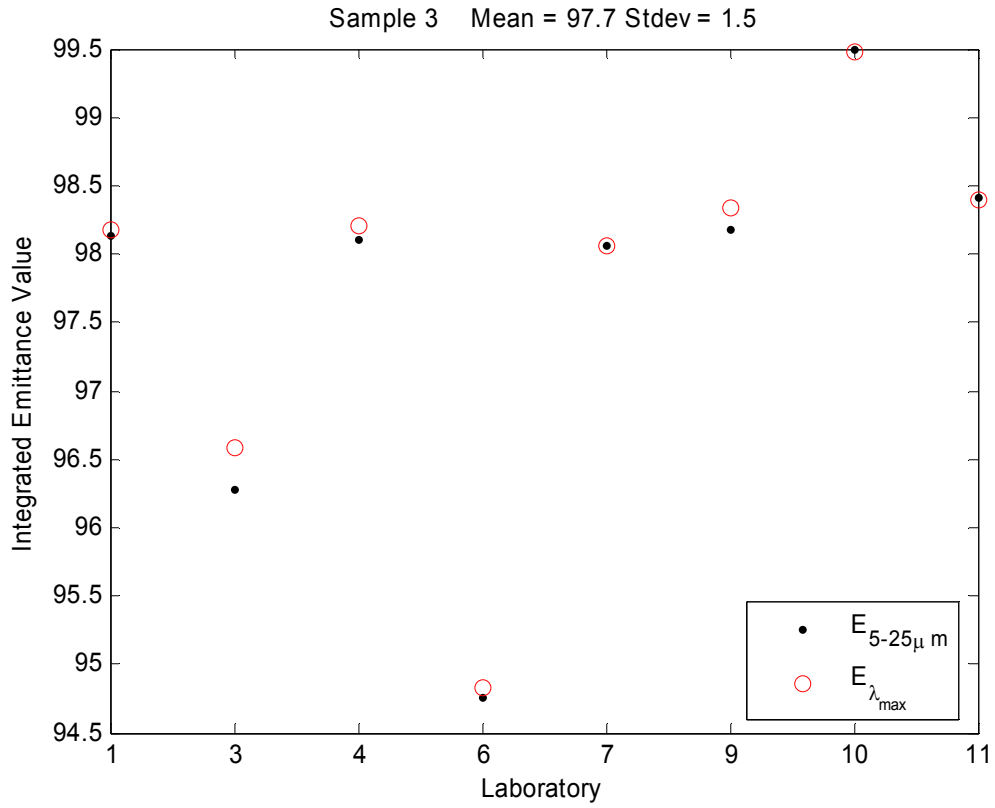


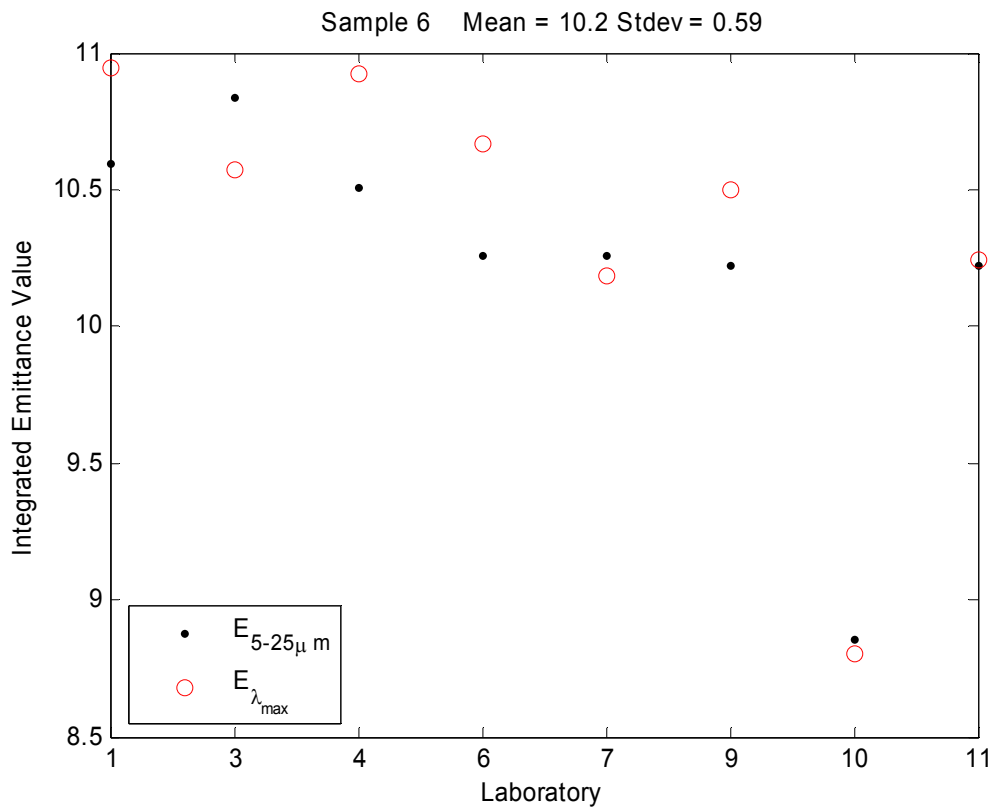
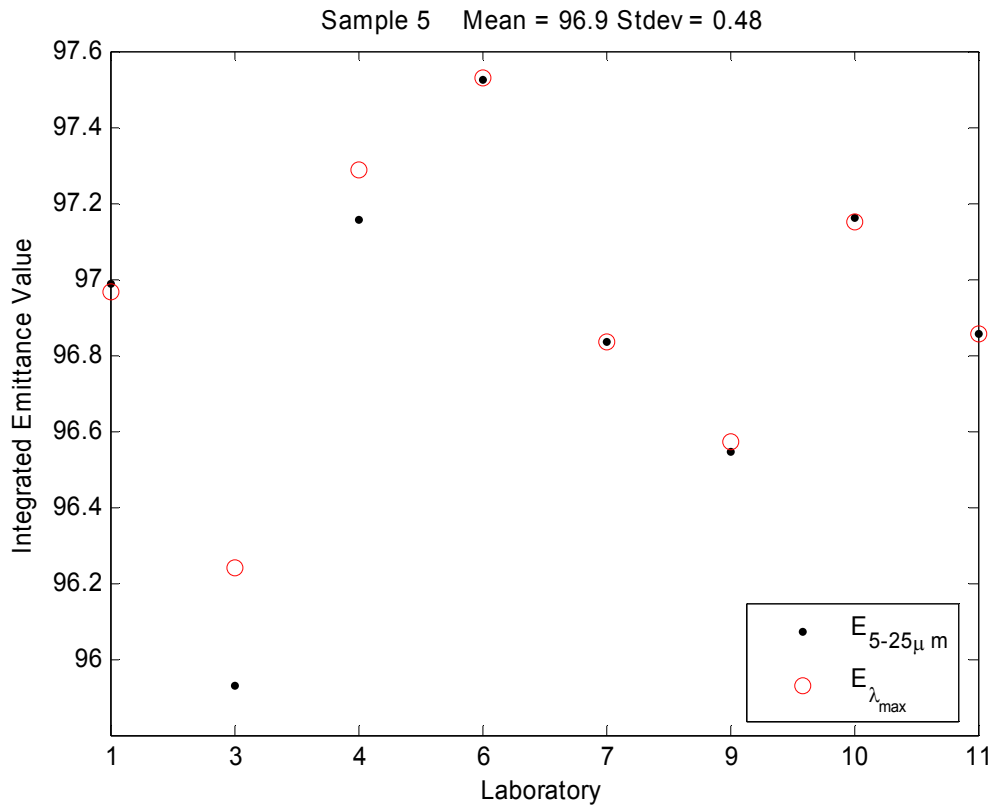




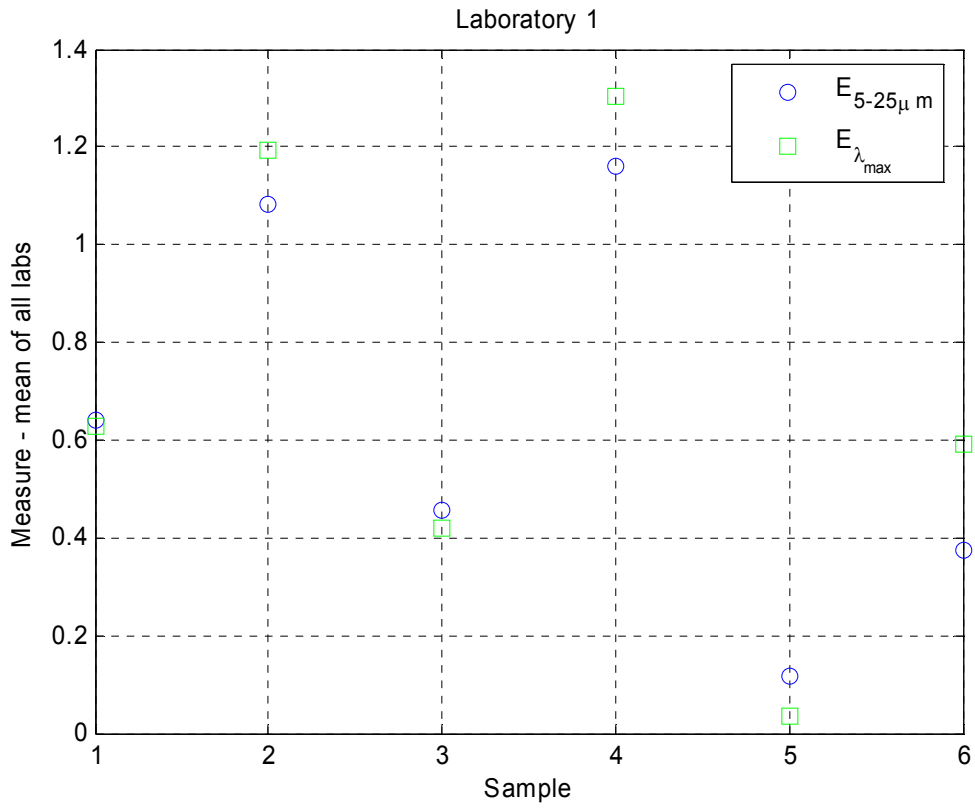
Figures 36-41. Each figure represents one sample and shows for each laboratory the weighted average normal emittance from side 1, which is the coated side in the case of coated samples. The average values were calculated using a standard procedure by LBNL from the raw data provided by each laboratory so that the differences are due only to the measured values not to the calculation procedure. Two values are plotted: (1) the emittance averaged over the minimum required range of 5-25 microns and (2) the emittance averaged from over the full range provided which varies from laboratory to laboratory. Open symbols are used for the full-range average so that symbols do not overlap.

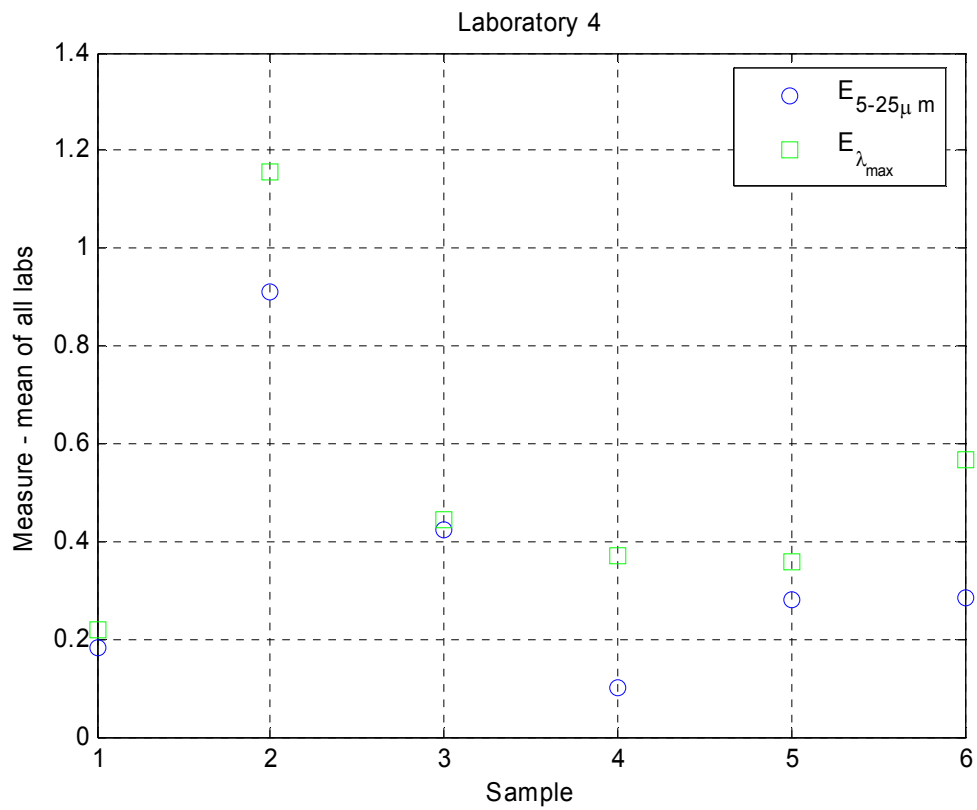
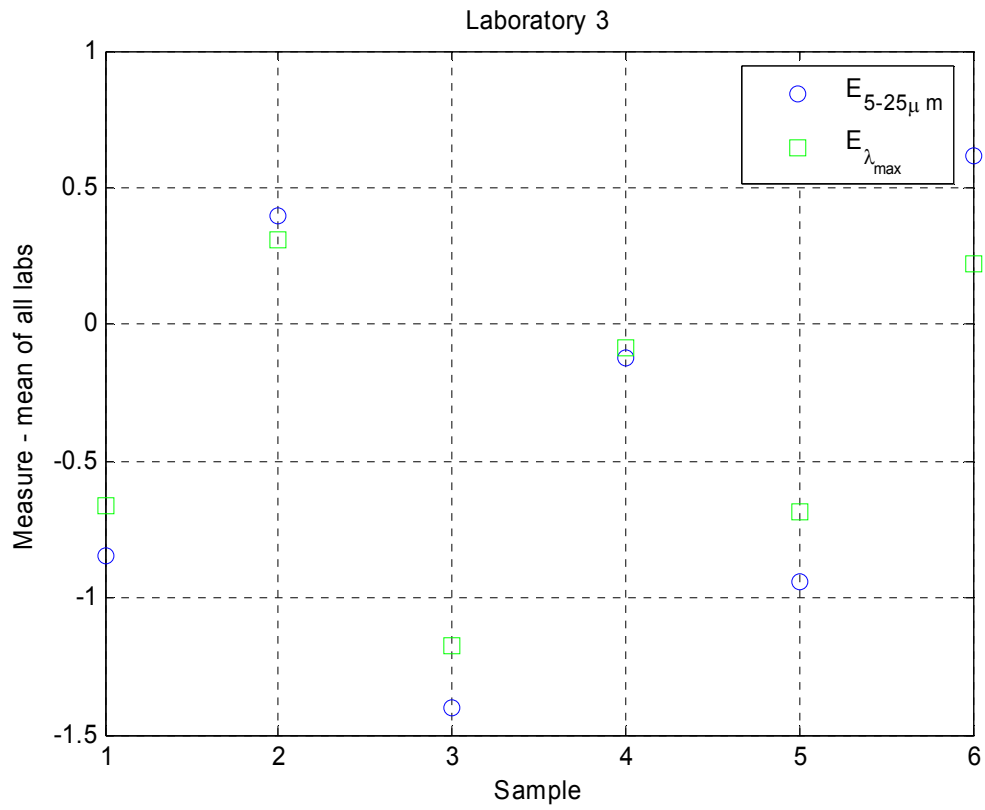


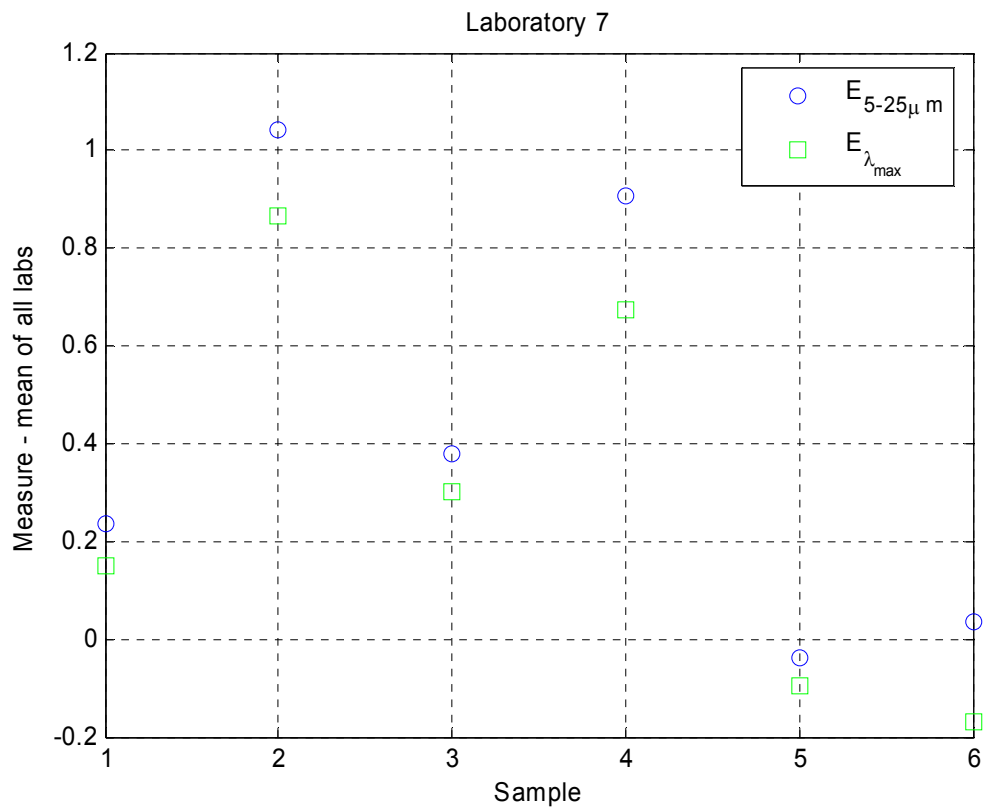
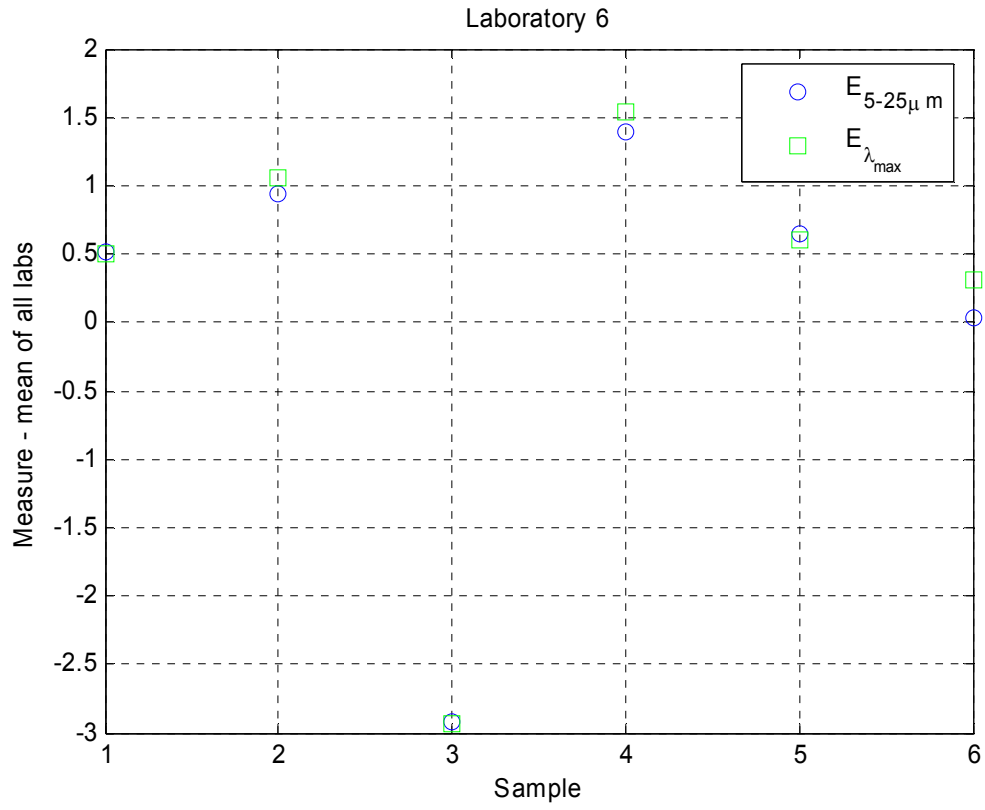


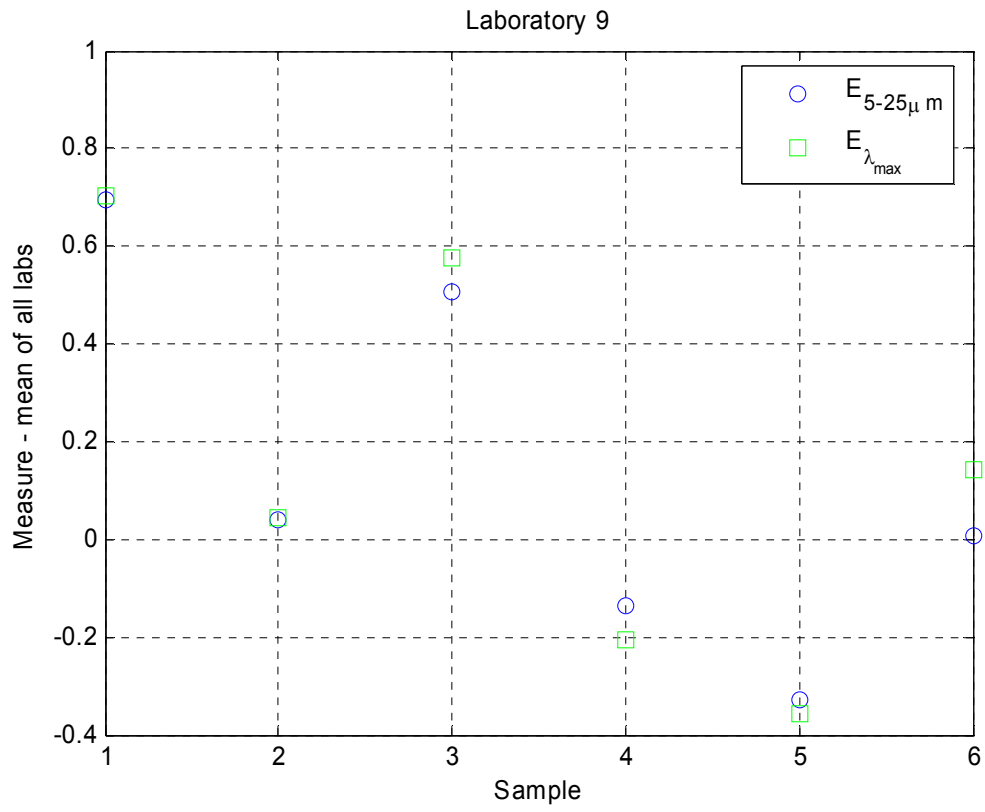


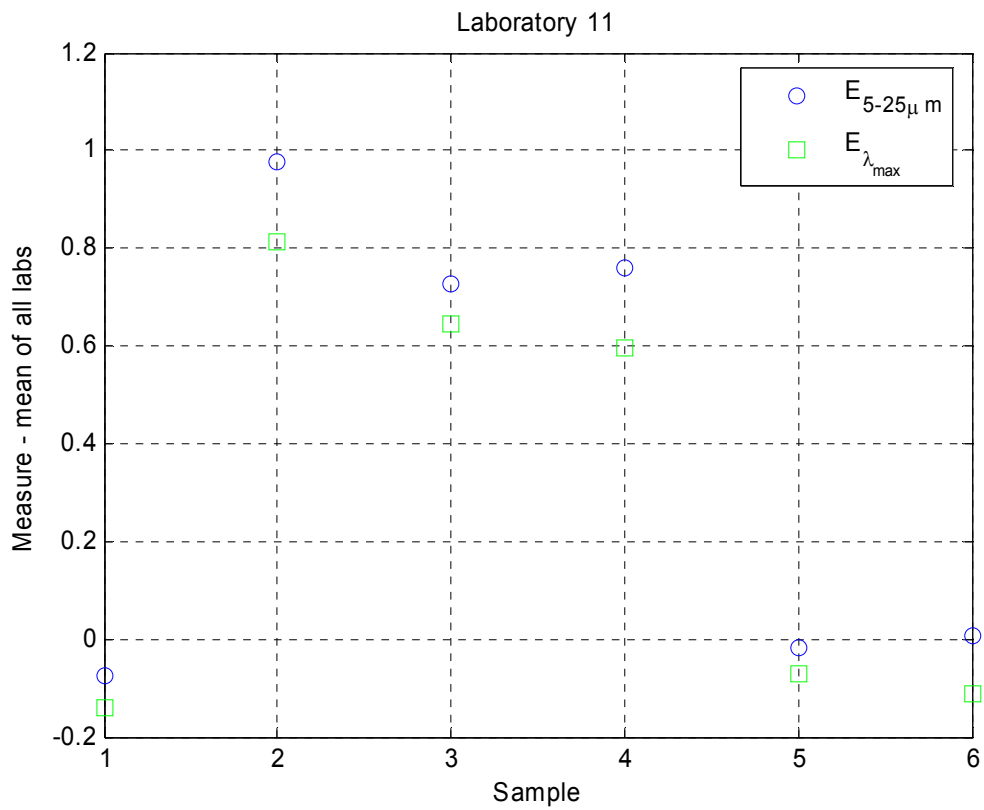
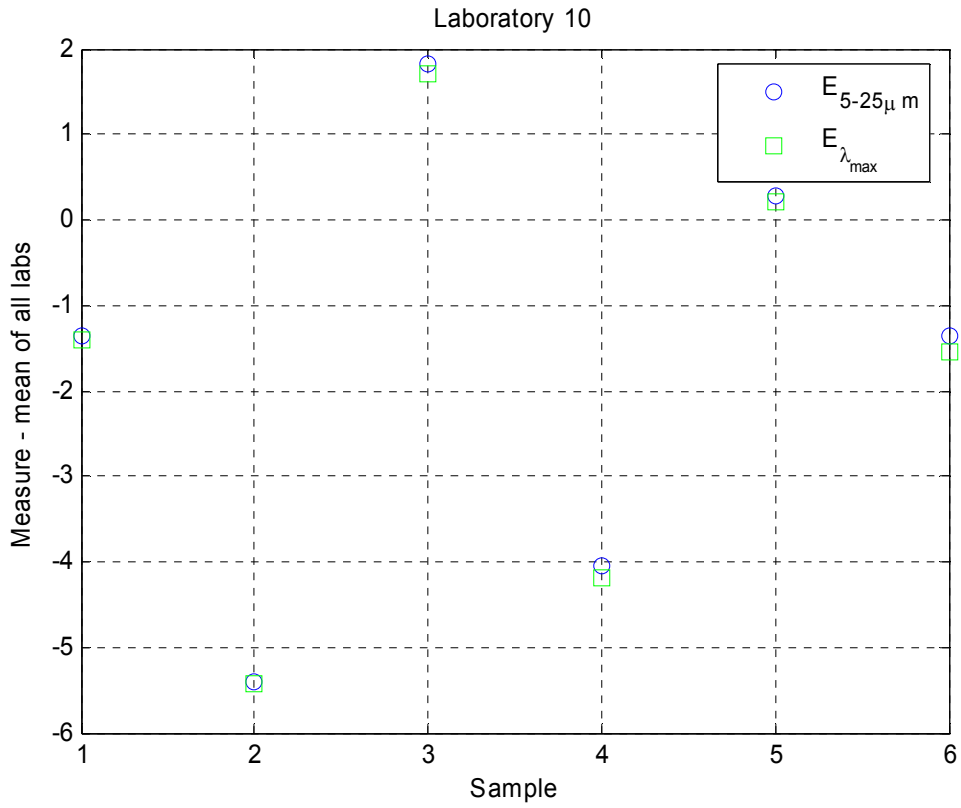
Figures 42-49. Each figure summarizes the results in the infrared spectrum for one laboratory. The emittance from 5-25 microns for each sample is plotted as its deviation from the mean value for all laboratories.











Figures 50-53. Each pair of graphs represents one type of emittance property: the average to 25 microns and the average to the maximum measured wavelength. The first graph in the pair gives the maximum, minimum and standard deviation for the particular property represented by the graph over all labs and for each sample. The second graph in each pair presents the same values normalized to the mean value.

