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PYROELECTRIC STANDARDS FOR SPECTRAL AND BROADBAND RESPONSIVITY MEASUREMENTS FROM 210 NM TO 3000 NM

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Abstract

Tunable-laser applied spectral measurements, for obtaining low uncertainty responsivity scales, are slow and expensive. Instead, recently developed pyroelectric detectors with stable organic-black coating have been used and calibrated for responsivity, based on spectral reflectance measurements of the coating. The reflectance measurements were performed on glass coated samples and also in-situ on the detector between 210 nm and 3 μm using monochromator- and Fourier-Transform-based setups. Since the coating absorbs twenty times more flux than what it reflects, the 3 % (k=2) reflectance measurement uncertainty produced about 0.15 % (k=2) absorptance uncertainty. The uncertainty for the relative response which is proportional to the absorptance has been determined from transmittance and reflectance measurements. An absolute tie point with similar or lower uncertainty converted the relative response into spectral irradiance or radiant power responsivity. This pyroelectric irradiance responsivity determination resulted in fast and accurate calibrations using regular monochromators. In addition to the spectral calibrations, broadband UV-to-SWIR responsivity measurements were performed by selecting the flat-response versions of the above pyroelectric detectors. These flat-response meters could be similarly used to lux meters that measure illuminance, the photometric equivalent of integrated irradiance.

Keywords: Pyroelectric standard, Spectral irradiance responsivity, Integrated irradiance, UV-to SW-IR calibrations, Pyroelectric spectral reflectance, Organic black coating, Spectral radiant power responsivity

1 Introduction

Previously, for reference calibrations, tunable laser used spectral responsivity measurements were applied to obtain low responsivity-uncertainty in the UV and IR [1]. The tunable laser applied method is time consuming and expensive.

In field-level broadband UV and IR applications, use of a lux-meter type instrument is needed to perform measurements fast, accurate, and inexpensive. In the UV, the filter corrected detectors produce large errors because the realization of the CIE standardized rectangular-shape responsivity functions (such as UV-A and UV-B) can be made only with large spectral mismatch. In practice, all function realizations in the UV are poor and different. The UV measurement errors could be decreased when the spectral distribution of the source was measured and spectral mismatch corrections were applied. These traditional methods are either slow and expensive or complicated. In the IR, broadband measurement method that gives uniform results with low uncertainty is not available.

The development of radiometric quality pyroelectric detectors with low noise-equivalent-power (NEP) opened a new era in pyroelectric based radiometric detector calibrations. The Gentec EO developed Model STEP-45 [2] pyroelectric hybrid detectors [3] have sturdy organic-black paint coatings and excellent long-term stability. They do not need a protecting or sealing window and have a typical NEP = 5 nW/Hz$^{1/2}$. This NEP is low enough to obtain high signal-to-noise ratios using traditional lamp-and-monochromator-based tunable light sources for low-uncertainty spectral radiant power responsivity measurements from the UV to the IR. Up until now, it was impossible or difficult to perform similar measurements with traditional pyroelectric detectors which have NEPs of 60 nW/Hz$^{1/2}$ to 100 nW/Hz$^{1/2}$ [4].

2 Evaluation of large organic-black coated samples

To determine the relative spectral response of the low-NEP pyroelectric hybrid detectors with low uncertainty, their organic-black coatings were evaluated. In the pyroelectric hybrid detectors, Model Pyromark 1200 paint coatings are deposited on the $LiTaO_3$ crystals. The detector sizes are either 5 mm or 9 mm. To obtain good signal-to-ratios in the evaluations (spectral measurements), 20 mm by 20 mm coatings of different thicknesses were deposited on thin glass plates. In these tests, the relationship of the coating thickness to the response change was analyzed. Figure 1 shows the measured spectral reflectance factors of the organic black coatings of 30 μm, 40 μm, and 50 μm thickness.

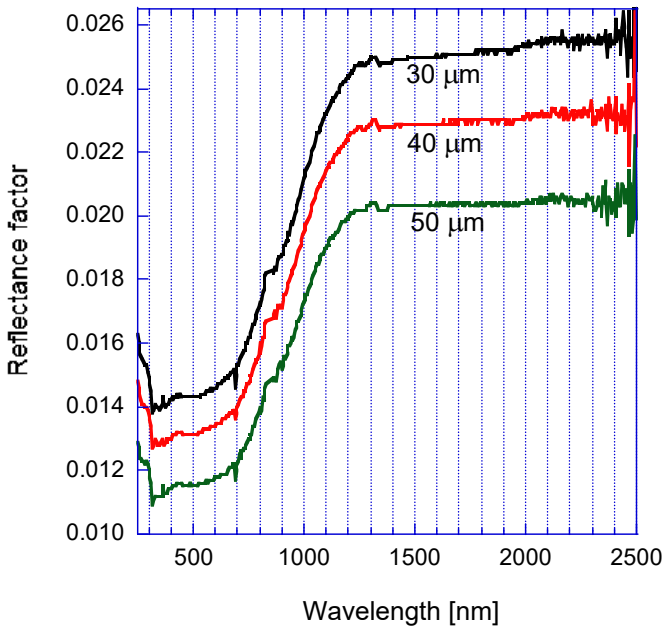

Figure 1 – Reflectance factor versus wavelength of organic-black coatings of 30 µm, 40 µm, and 50 µm thickness (see labels) in the wavelength range from 250 nm to 2500 nm.

The measurements were made using a commercial spectrophotometer fitted with an integrating sphere. Each sample was mounted on the sample port of the sphere, so that the incident beam illuminated the sample (at an 8-degree angle with respect to the sample normal) and the reflected flux was collected within the integrating sphere where it was detected. The reflectance factor for this total reflectance measurement is the ratio of reflected flux to incident flux relative to the ratio of a perfectly reflecting diffuser (NIST standard). The reflectance changed about 0.01 between 250 nm and 2500 nm and the reflectance was flat (constant) above 1200 nm when the thickness of the coating was at least 50 μm. The reflectance increased for coatings thinner than 50 μm. An increasing measurement noise can be seen above 2000 nm. The reproducibility of the measured reflectance factor values was 6 % and the reproducibility of thickness comparison was 3 %.

Figure 2 depicts the spectral diffuse transmittance of the organic black coatings. These measurements were collected by mounting each sample at the entrance port of the integrating sphere of the spectrophotometer. The incident beam illuminated the sample (at a 0-degree angle with respect to the sample normal) and the transmitted flux was collected within the integrating sphere where it was detected. The diffuse transmittance is the ratio of the transmitted flux to the incident flux. The figure shows that the thickness of the organic black coating must be close to 50 μm to get a negligible diffuse transmittance for the measured overall wavelength range.

Since the sum of the reflected flux R, the absorbed flux A, and the transmitted flux T is 1 (unity), the measurement equation can be written as:

$$R + A + T = 1 \tag{1}$$

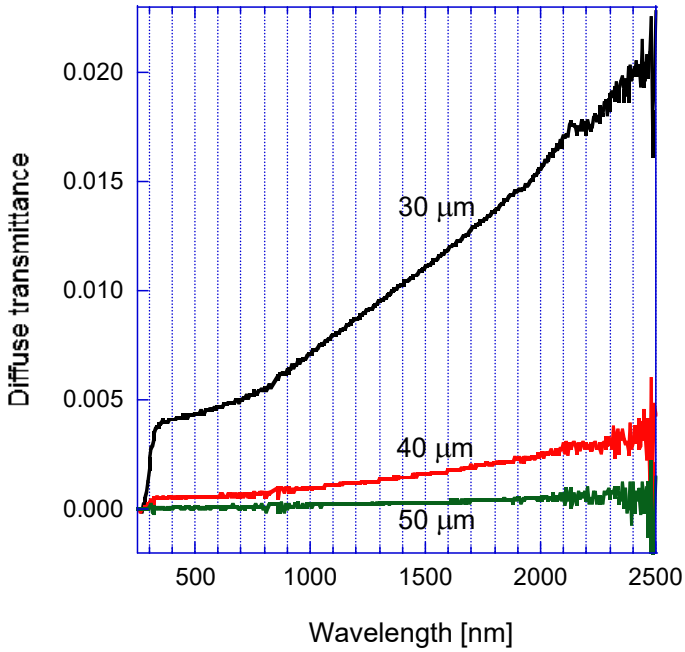

Figure 2 – Spectral diffuse transmittance for organic black coatings of 30 µm, 40 µm, and 50 µm thickness between 250 nm and 2500 nm.

When using the 50 μm thick organic black coating with the negligibly small transmittance, the measurement equation can be simplified:

$$A = 1 - R \tag{2}$$

Since the absorbed flux is much larger than the reflected flux, a percent level relative uncertainty in the reflectance measurements will result in a significantly smaller relative uncertainty for the absorbed flux.

The obtained *A* (called absorptance) is proportional to the relative response of the pyroelectric detector. However, Eq. 2 wouldn't be valid for thinner coatings. For thinner coatings, such as the traditionally applied 25 μm to 35 μm thick coatings, Eq. 1 shoud be used together with the measurement results shown in Figs. 1 and 2 to obtain the relative response of the pyroelectric detector with low uncertainty.

Although the wavelength dependent reflectance increase was about 0.01 at 1200 nm, the absorptance change according to Eq. 1 is ~1 % for the 0.98 absorptance of the 50 μm thick coating at a wavelength of 1200 nm or higher. Accordingly, a strong (e.g. 50 % in Fig. 1) wavelength dependent change in the reflectance factor produces a small (about 1 %) wavelength dependence in the absorptance. This uncertainty reduction is the main advantage of the here proposed reflectance-based spectral-response calibration method of organic black coated pyroelectric detectors.

3 In-situ pyroelectric response determination

After decreasing the beam size of the monochromator-based reflectance measurement facility, three 9 mm pyroelectric hybrid detectors were measured instead of using the large, 20 mm dummy samples applied on glass plates. Using these real detectors, the relative spectral response could be in-situ determined from spectral reflectance measurements with improved response uncertainty.

In addition to the above discussed organic black coating measurements on the "dummy" glass samples, spectral reflectance measurements were performed on real pyroelectric detectors. For all detectors the same coating type and standard (traditional) thickness of 25 μm to 35 μm were

used. In these measurements, the transmittance of the coatings in the real detectors could not be measured. Figure 4 shows the reflectance factors of the three real detectors where the $LiTaO_3$ crystals were coated with 25 μm to 35 μm thick black coatings. The graph also shows the reflectance factor obtained from the above discussed measurements of a glass (dummy) sample when it was coated with a 30 μm thick black paint. The thickness of the coatings in these spectral measurements, performed on the three real pyroelectric detectors and one coated-glass sample, was very similar. The reflectance was the smallest on the coated glass sample where the transmitted flux was the highest. As shown in Fig. 2, the transmittance in this wavelength interval was about 0.005. These measurements were made between 200 nm and 700 nm.

As shown in Fig. 5, the absorptance curves between 210 nm and 700 nm, were calculated from the spectral reflectance measurement results using Eq. 2. The curves are normalized at 660 nm where the absolute tie point is applied (see below). Here, the organic black coated detectors (made in 2019) are compared to the dummy coating of 30 μm thickness on a glass substrate (made in 2016). The graph shows that the spectral responses (proportional to 1-Reflectance) of the real detectors agree within 0.2 %. However, the response deviation from the dummy sample has a maximum of 0.5 % at 365 nm. The measurement results at 200 nm are not shown because the signal-to-noise ratio here was poor caused by the low reflectance of the standard diffuser.

The relative response ratios of two 9 mm detectors (#11 and #12) are shown in Fig. 3 relative to another 9 mm detector (#10) between 210 nm to 700 nm. Here, the deviations for real detectors are less than 0.2 %. When we compare the response calculated for the dummy sample using Eq. 2, a 0.996 response ratio was obtained in the UV which is 2-to-4 times worse than the ratio obtained for the 9 mm detectors. When the reflectance data of the dummy sample is used to create a pyroelectric detector standard, the 0.4 % difference should be taken into consideration. Based on the here discussed recently measured reflectance curves of the three real pyroelectric detectors, a response uncertainty at 365 nm of about 0.1 % (k=2) was obtained. This component for the relative response in the UV-VIS range should be included in the overall responsivity uncertainty. This additional component includes the uncertainty of the absorptance originating from the spectral reflectance measurement.

4 Pyroelectric irradiance responsivity standard

A 5 mm pyroelectric hybrid detector was calibrated based on the spectral reflectance measurement of the dummy sample. The 5 mm Model STEP-45 detector (from Gentec-EO USA) was purchased before the 9 mm versions were developed and acquired. The 5 mm detectors were too small to measure the reflectance directly. The estimated thickness of the organic black coating was ~30 μm. The spectral reflectance and absorptance for this detector was determined from the large (dummy) sample measurements as discussed in Section 2 above. Figure 6 shows the spectral irradiance responsivity of this pyroelectric detector between 250 nm and 2000 nm. From the relative response, which is proportional to the absorptance, the irradiance responsivity was determined. The uncertainty of the relative response at 365 nm, as discussed in Section 3 above, is 0.1 % (k=2). The absolute tie pont was created to obtain a reasonably low spectral irradiance responsivity uncertainty for the UV-A range from 320 nm to 400 nm. For the illustrated 250 nm to 2000 nm wavelength range in Fig. 6, the 2000 nm to 2500 nm interval was not used because of the oscillations shown in Figs. 1 and 2.

The tie point was obtained from a transfer standard detector. It was derived with direct comparison of the signal from the pyroelectric detector to a Si trap irradiance standard at a distance of 1843 mm. The irradiance responsivity uncertainty of this Si trap-detector is 0.1 % (k=2) and it was derived from the cryogenic radiometer in power measurement mode [1]. The area of the aperture was separately determined [1]. The tie point was used for the relative to absolute spectral responsivity conversion.

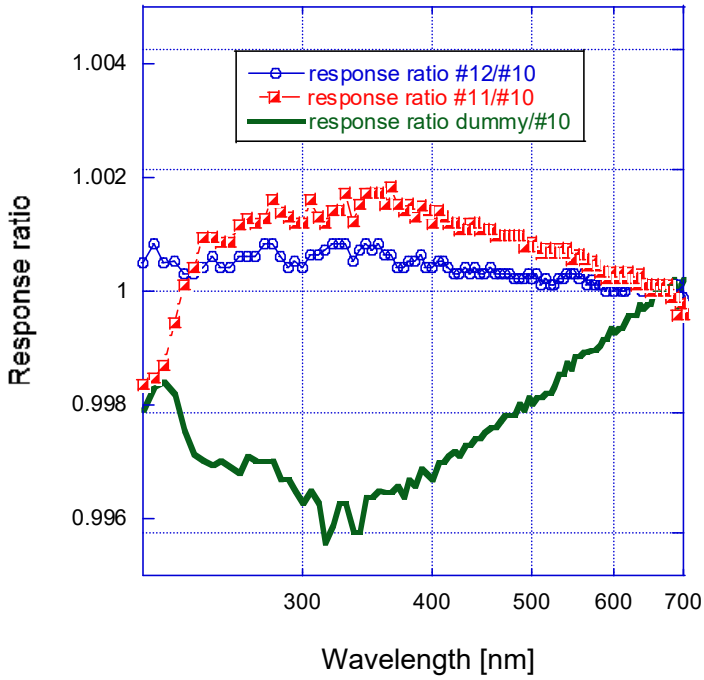

Figure 3 – Response ratios of two 9 mm detectors (#11 and #12) and a dummy coating (no detector) to another 9 mm detector (#10) from 210 nm to 700 nm.

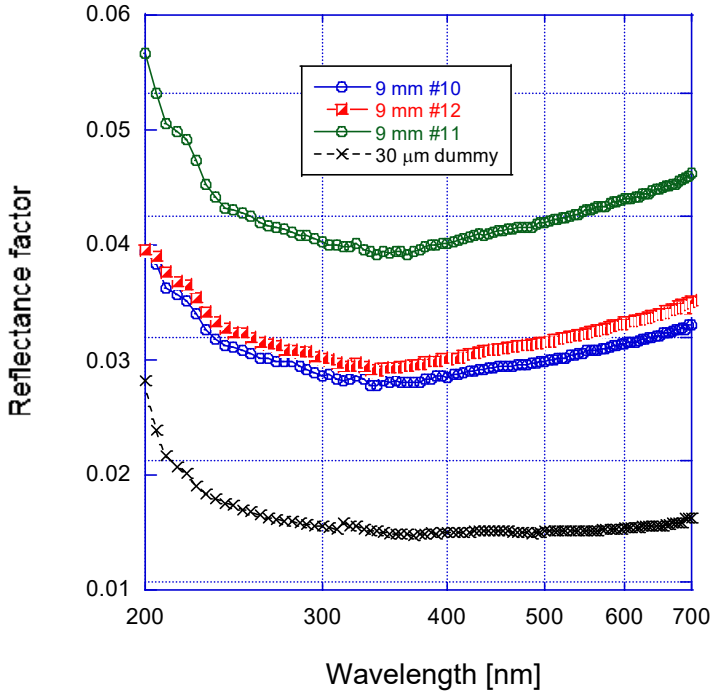

Figure 4 – Reflectance factors of 9 mm $LiTaO_3$ crystals coated with 25-35 μm thick organic black coating and a glass (dummy) sample coated with 30 μm thick coating of the same type.

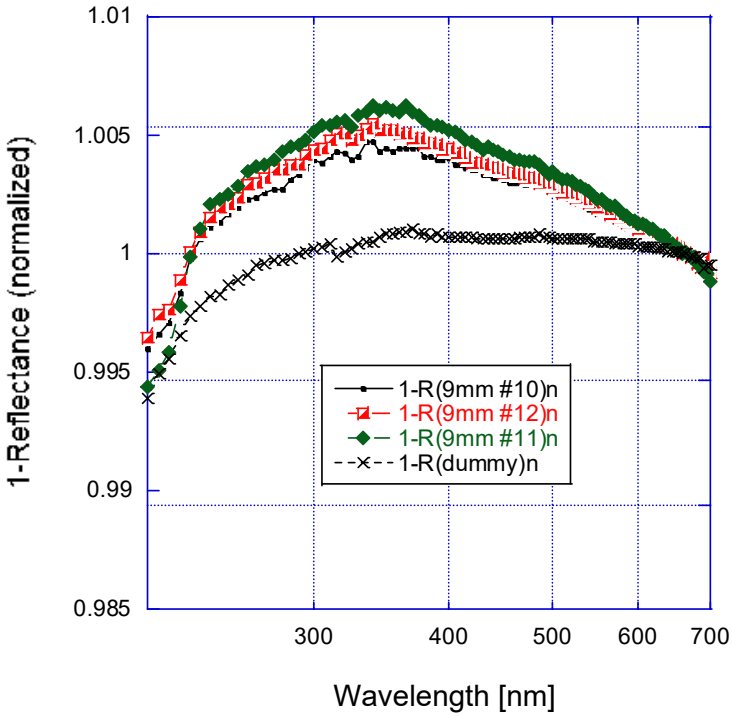

Figure 5 – Absorption (1-reflectance) curves between 210 nm and 700 nm (normalized at 660 nm) of the organic black coated 9 mm detectors (made in 2019) compared to the dummy coating of 30 μm thick on a glass substrate (made in 2016).

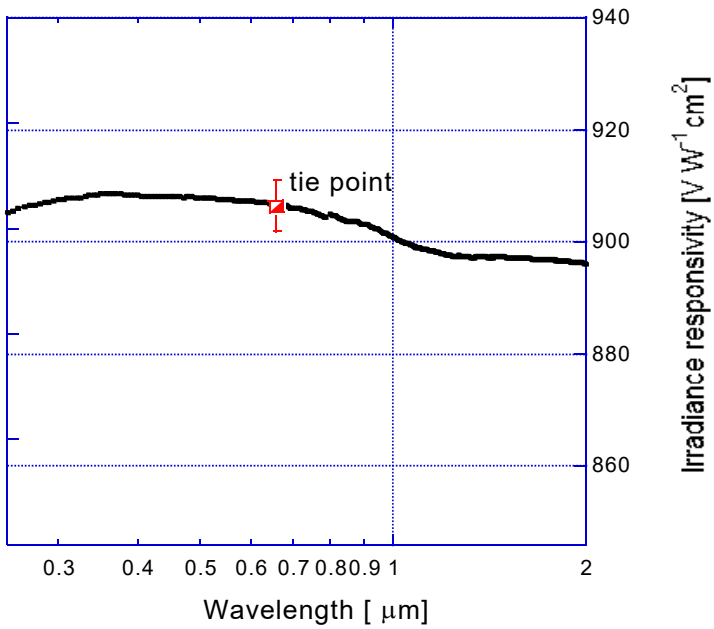

Figure 6 – Spectral irradiance responsivity of an organic-black-coated hybrid pyroelectric detector between 250 nm and 2000 nm. The tie point with its uncertainty bar is shown at 660 nm.

The tie point uncertainty budget with its resultant uncertainty of 0.5 % (*k*=2) is shown in Table 1. This 0.5 % (*k*=2) responsivity uncertainty together with the 0.4 % (*k*=2) uncertainty of the relative response (coming from difference between dummy sample and in-situ measurement of real detector), produce a 0.64 % (*k*=2) overall irradiance responsivity uncertainty that propagates for the 250 nm to 2500 nm wavelength range. This calibrated pyroelectric detector can be used

as a standard for spectral irradiance responsivity calibrations in the UV-to-SWIR range assuming that sufficient signal-to-noise ratio is available.

Table 1 – Responsivity-uncertainty budget of the organic-black coated pyroelectric detector at the 660 nm irradiance responsivity tie point.

Relative uncertainty components	**[%]**
$\Delta\lambda$	0.03
Distance	0.04
Target spot non-uniformity	0.10
Spectral response changes	0.18
Output signal ratio	0.10
Reference Si-trap	0.10
Combined (*k*=1)	**0.25**
Expanded (*k*=2)	**0.5**

The uncertainty components in Table 1 originate from distance, wavelength error, target spot spatial non-uniformity, and signal (ratio) measurement errors. To keep the uncertainties originating from distance measurement and target-spot irradiance-nonuniformity small, an increased separation was used between the LED irradiance source and the irradiance measuring detectors. Figure 7 shows the optical geometry for irradiance responsivity measurements using a collimated LED source peaking at 660 nm. This source was used only for irradiance responsivity transfer and it is not a standard. In this discussed example, the standard was a Si trap-detector with known power-responsivity and area of its front aperture.

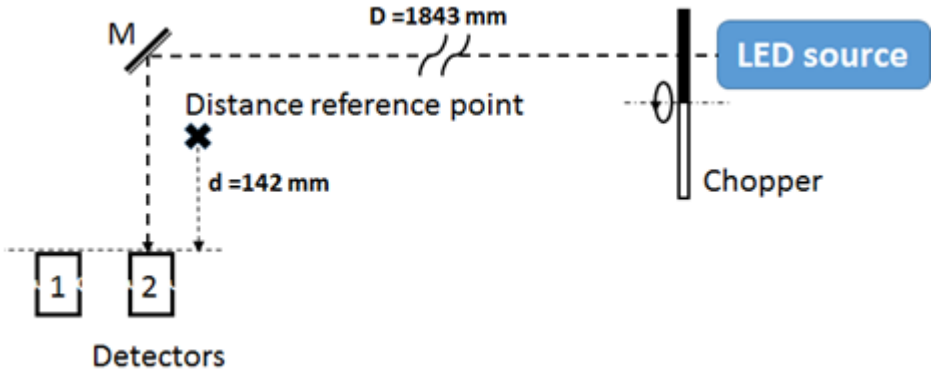

Figure 7 – Optical geometry for irradiance responsivity measurements using a collimated red LED (transfer but not standard) source.

For the required low-uncertainty distance measurement, an arbitrary distance-reference-point was used. The distance measurement error for the shown d = 142 mm distance is about 1 mm. However, when moving the LED source further away, the increased distance D = 1843 mm can be measured with a decreased relative error even if a simple tape measure is used. Detector 1 is the transfer standard Si-trap with the irradiance responsivity uncertainty of ~0.1 % (*k*=2). This irradiance responsivity is transferred to Detector 2 which is the above discussed pyroelectric hybrid detector. The long distance between the collimated LED source and each detector was needed to obtain a small enough uncertainty component for the distance measurement. The irradiance level could be within a large interval with a minimum value determined by the signal-to-noise ratio of the pyroelectric detector.

For power responsivity calibration of the above pyroelectric detector in the UV-to-SWIR, the tie point can be derived from the power-responsivity of the same Si trap-detector (where the above irradiance responsivity was derived from) also at 660 nm but both detectors are to be underfilled by the incident-beam originating from the 660 nm LED.

5 Power responsivity extension and validation to 3 μm

The measured reflectance and calculated absorptance curves of organic-black paint (of about 25 μm thickness) deposited on a thin glass plate, are shown in Fig. 8. The two curves labelled with Sample (purple and green) were measured using the commercial instrument described in Section 2 between 250 nm and 2500 nm. The curve labeled with R s Mean (blue) was measured using the Infrared Reference Integrating Sphere (IRIS) in the Fourier Transform Infrared Spectrophotometry (FTIS) Lab [5] from 800 nm to 3 μm. The deviation between the monochromator-based and the FT-based measurements in the SW-IR where the two curves overlap is about 0.25 %. The effect of this reflectance deviation on the relative response is small because the absorbed flux is twenty times higher than the measured reflected flux resulting in low absorptance uncertainties.

The relative response curve which is proportional to the spectral absorptance (shown in Fig. 8) needs a tie point with low power responsivity uncertainty such as 0.2 % (*k*=2). Since the purple Sample curve is the same absorptance as the one shown in Fig. 6, the 660 nm tie point can be used for the FT measured (R s Mean) absorptance (relative response) curve as well.

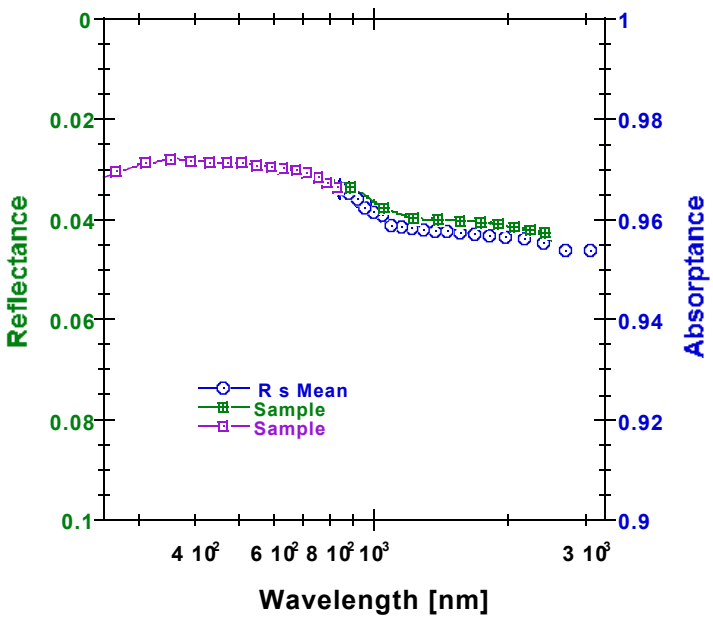

Figure 8 – Absorptance deviation obtained from monochromator (the curves labeled Sample from 250 nm to 2500 nm) and FT (R s Mean curve from 800 nm to 3 μm) measured spectral reflectance data of the same organic-black paint sample.

The expanded uncertainty of the absorptance of the organic-black coated (dummy) sample, as evaluated at the IRIS/FTIS facility, was 0.0015 between 1 μm and 3 μm. The close to 5 % spectral reflectance was measured with about 3 % (*k*=2) uncertainty. This uncertainty converts to about 0.15 % (*k*=2) for the ~20 times higher (~95 %) absorbed flux (absorptance).

The 9 mm #11 real pyroelectric detector was also measured in situ at the FTIS facility when the detector can was removed to avoid reflections. The thickness of the organic black coatings was about 30 μm on both the detector and the dummy sample. The measured spectral reflectance curves, normalized at 2 μm, are shown in Fig. 9. The noise caused by the poor signal-to-noise ratios at the short wavelength end was smoothed (filtered) out. The spectral variations, that contribute to the response are within a few percent between the two devices.

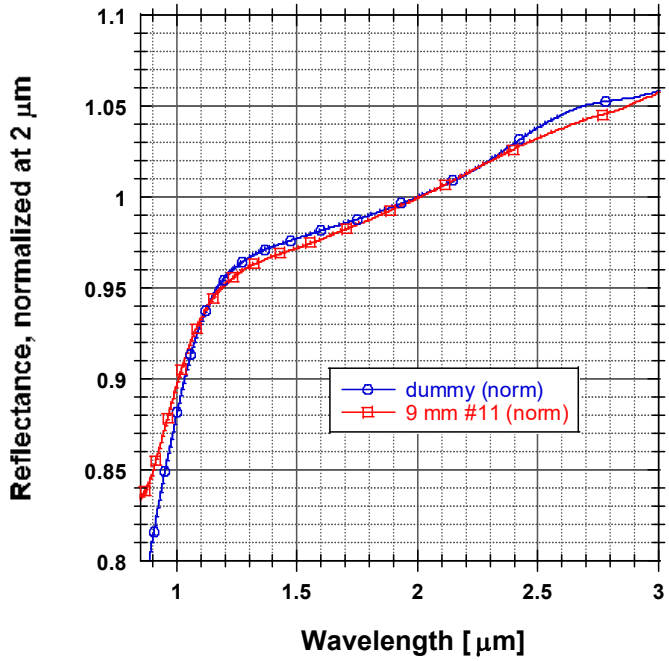

Figure 9 – Normalized spectral reflectance curves of a 9 mm pyroelectric detector and a large dummy sample both coated with organic black of 30 μm thickness

It is the main advantage of the here discussed calibration method that a relatively large reflectance measurement uncertainty could produce a much smaller (relative) response-uncertainty. Using the FT reflectance measurement method, the radiant power responsivity was determined to 3 μm.

The uncertainty of the power responsivity tie point can be similar to that of the 0.5 % (k=2) irradiance responsivity tie point discussed in Section 4 above. The dominant uncertainty component for power responsivity may originate from the spatial non-uniformity of response of the pyroelectric detector. In this example, an overall power responsivity uncertainty of close to 0.5 % (k=2) was obtained for the 800 nm to 3 μm range.

6 Broadband (integrated irradiance) applications

In addition to the above discussed spectral calibrations, broadband UV-to-SWIR irradiance measurements could be performed by selecting the flat-response versions of the above discussed irradiance measuring pyroelectric detectors. These flat-response pyroelectric radiometers can be similarly used to lux meters that measure illuminance, the photometric equivalent of the integrated irradiance.

The integrated irradiance [W/cm^2] when using the flat response pyroelectric radiometer is:

$$\bar{E} = \frac{i_{out}}{s} C$$

where i_{out} is the output current [A] of the pyroelectric radiometer, s is the constant responsivity [A cm^2/W], and C is the real (calibrated) correction factor for the lock-in reading. The theoretical value of C is 2.22.

Using this pyroelectric detector standard with the constant spectral irradiance responsivity, a standard source is not needed to measure integrated irradiance.

Flat-response pyroelectric radiometers have already been successfully used for integrated irradiance measurements of UV LEDs [6].

7 Conclusions

Organic black coated hybrid pyroelectric detectors have been developed and applied for low-uncertainty spectral responsivity calibrations and integrated irradiance measurements. This is the first time use of low-NEP pyroelectric detectors for irradiance measurements. The responsivity determinations are based on spectral reflectance measurements of the pyroelectric coatings. The applied low-NEP pyroelectric detectors can be used as radiometric standards even at the output of regular monochromators. Using these pyroelectric standards, responsivity uncertainties of close to 0.5 % (k=2) were achieved in the UV and IR. Using spectrally calibrated pyroelectric detectors of flat response, broadband measurements can be performed from UV to IR without using any source standard. The described pyroelectric radiometers can perform fast and accurate spectral responsivity and broadband measurements from 210 nm to 3 μm.

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[2] DISCLAIMER, c*ertain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by NIST, nor does imply that the equipment are necessarily the best available for the purpose*.

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