Infrared Absolute Calibrations Down to 10 fW in Low-Temperature Environments at NIST

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ABSTRACT

The Low Background Infrared (LBIR) facility at the National Institute of Standards and Technology (NIST) is responsible for absolute IR radiometric calibrations (SI traceable) in low-background temperature (below 80 K) environments. IR radiometric test hardware that needs to be operated in cryogenic environments is calibrated in cryogenic vacuum chambers maintained by the facility to create environments that simulate the low-temperature background of space. Transfer radiometers have also been developed to calibrate IR radiometric test hardware that is too large to ship to NIST from their own IR test facilities. The first generation transfer radiometer, the BXR, is a filter-based radiometer that utilizes an As-doped Si blocked impurity band detector, and can calibrate IR test chambers to a total uncertainty of less than 3 % (1 σ) at irradiance as low as 10^{-14} W/cm^2. The BXR has evaluated 9 chambers and the performance of a subset of these chambers will be discussed to a limited extent to demonstrate the need for calibrating IR test chambers. The second generation transfer radiometer, the MDXR, and new primary standards allowing absolute calibrations as low as 10^{-15} W/cm^2 are in the final stages of development. The MDXR will have all the functionality of the BXR and it will have a cryogenic Fourier transform spectrometer (FTS) for high resolution spectral capability. Performance specifications and test results from development activity on the new primary standards will be discussed.

Keywords: calibration, infrared, IR, traceability, low-background temperature, low power, LBIR, NIST

1. INTRODUCTION

The Low-Background Infrared (LBIR) Facility at the National Institute of Standards and Technology (NIST) began calibration services in 1989 to fulfil the national need for calibrations at IR wavelengths in background environments below 80 K [1]. The calibration capability is based on the ability to measure infrared power absolutely using Absolute Cryogenic Radiometers (ACRs) [2]. The need for calibrations in low-temperature environments is driven by a variety of factors that make standard ambient environment radiance and irradiance modelling unreliable when applied to low temperature, high vacuum environments. Such issues include, but are not limited to, large thermal gradients, high radiative power loads, and changes in material properties at low temperatures.

The LBIR facility has been calibrating blackbodies that operate in cryogenic environments since 1989. Details of the calibration methodology and selected calibrations are communicated elsewhere [3]. Blackbody sources delivered to the LBIR facility are calibrated directly against an ACR in a very simple optical configuration. This provides the shortest calibration chain for our customers to absolute standards, or SI units. For this service, the sources are integrated into low-background calibration chambers, the contact thermometers on the blackbody cavity are calibrated against the measured radiance temperature, and the sources are shipped back to the customer. In most cases, the source is then integrated back into an IR test chamber as part of a larger optical system used to calibrate remote sensors.

To improve the calibration dissemination capabilities of the LBIR facility a filter-based transfer radiometer, the BXR, was developed to calibrate low-background temperature IR calibration chambers that are too large to be transported to NIST. The BXR uses collection optics together with narrow band-pass filters and an arsenic-doped blocked impurity band (BIB) detector to measure the output of collimated sources in the IR test chambers [4]. The band-pass filters currently cover the spectral range from 2 µm to 15 µm with pass bands of width ranging from 1% to 3 % of the band center wavelength. The filters are interchangeable and can be selected to match customer requirements to a wavelength less than 32 µm, where the BIB detector responsivity drops to effectively zero. The BXR has a 7 cm diameter entrance aperture and is designed to calibrate collimated sources with a divergence up to 1 mrad (full cone). If test chambers do not have collimated outputs, fore-optics can be placed in front of the BXR to collimate the beam into the BXR. The BXR

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can calibrate IR test chambers to a total combined uncertainty of less than 3% (k = 1) and routinely achieves 2% Type A uncertainties (k = 1) at irradiances as low as 10^{-14} W/cm^2 within a 60 second integration time.

Despite the success of the calibration accuracy that can now be delivered to users of the LBIR facility, this can only be achieved at relatively high powers compared to what our customers require. For chamber calibrations, for example, direct traceability to an ACR is limited to the lowest power that can be measured by our current ACRs, which corresponds to a relatively high irradiance of 10^{-10} W/cm^2. This means that the ACRs currently in use are approximately 4 orders of magnitude less sensitive than required to calibrate the BXR at its lowest irradiance sensitivity.

2. CALIBRATION FACILITIES

The LBIR facility has two chambers designed to create low-temperature, space-like environments for calibration activities. The larger chamber has a working volume that is approximately 30 cm x 30 cm x 150 cm, which is accessible by two thermally stabilized working surfaces (Figure 1). The working volume is surrounded by a cylindrical cryo-shroud that is 50 cm in diameter. The entire environment, shroud and working surfaces can be cooled to below 20 K by a large He refrigerator system and brought to a vacuum of 1 x 10^{-7} Pa. To support experimental and calibration activity for the users of the LBIR facility there are over 200 shielded electrical feedthroughs (including 10 co-axial feedthroughs) and auxiliary cryogen feedthroughs for systems that require liquid He. The He refrigerator can cool a load of up to 100 W while maintaining an environment below 30 K. There is a slightly smaller calibration chamber that is similar in function for activities that require less volume. There is also an antechamber that can be mated to the larger calibration chamber through a 17.75 cm diameter cooled baffle tube which allows the two chambers to form a completely enclosed low background environment. The antechamber has a larger cylindrical test volume that is 55 cm in diameter and 45 cm in length and can support systems that have an output power of 1000 W or greater, depending on required test conditions.

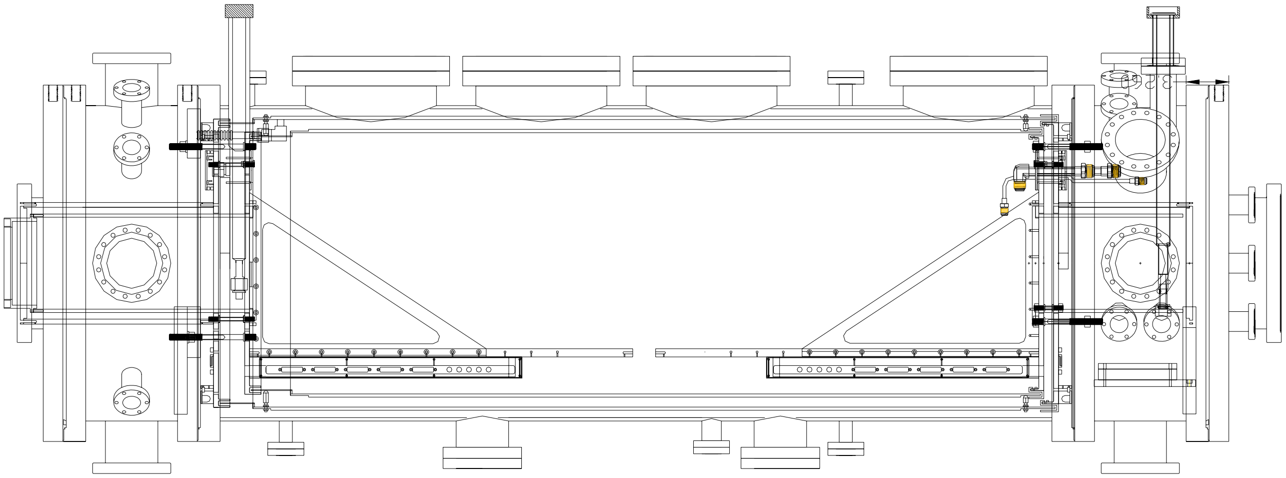

Fig. 1. Low-temperature calibration chamber at the LBIR Facility at NIST. Two roll out thermally stabilized working surfaces provide an enclosed working volume of 30 cm x 30 cm x 150 cm that can achieve background environments of below 20 K. There are over 200 shielded electrical feedthroughs (including 10 co-axial feedthroughs) and auxiliary cryogen feedthroughs for systems that require liquid He.

3. ABSOLUTE CRYOGENIC RADIOMETERS

The LBIR facility maintains a variety of ACRs that are designed for calibrations over different power ranges. The higher power variety, the series ACR I and ACR II, cover the range of 1 mW to 10 nW in total optical power at the ACR entrance apertures, which are 3 cm and 2.5 cm in diameter, respectively. These ACRs have a power threshold that is high enough to allow them to be compared with the NIST Primary Optical Watt Radiometer (POWR). This intercomparison is performed periodically and is used to validate the standard setting methodology used in the LBIR facility to create detector standards at much lower power levels where intercomparisons with other radiometers can not be made. The results of a recent intercomparison are shown in Table 1 and the details are described elsewhere [5]. In

Section 5, the development of an ACR designed to measure 1 pW of power with a combined uncertainty of ≈1 fW is discussed.

Table 1. Ratio of radiometric power measured by the Primary Optical Watt Radiometer to that measured by the high power ACRs maintained at the LBIR facility.

ACR Name	$R_{NatStand/ACR}$	Total Uncertainty
ACR Ia	0.999339	0.024%
ACR Ib	0.999130	0.030%
ACR IIa	0.999595	0.053%
ACR IIb	0.998765	0.029%

4. BLACKBODY AND IR TEST CHAMBER CALIBRATIONS

Blackbody sources are used for radiometric calibration activity because they are stable, reproducible, and their radiometric output can be modeled accurately. Ideally a blackbody has a large cavity that is uniform in temperature and an aperture that is integral to the cavity and whose area is a very small fraction of the surface area of the cavity. However, practical applications of blackbody sources often require the use of a relatively large blackbody opening and the use of a separate aperture wheel that is a finite distance from the blackbody cavity. If test requirements mandate the use of filter wheels, a chopper wheel or shutter, then the distance of the defining apertures from the blackbody cavity becomes even greater. For testing under cryogenic conditions there are also usually constraints on the size and total output power of a given source. The most commonly observed source of radiometric inaccuracy is the light-weighting of the blackbody cavity. This is most often done in order to make blackbody temperature change times shorter. With all of the performance requirements and design constraints incorporated into a blackbody source design, the radiometric power output of a typical blackbody source usually deviates significantly from what would be expected from contact thermometry and ideal Planckian performance.

Figure 2 shows the radiance calibration results for nine different blackbodies representing six distinct blackbody source designs. Blackbody source designs A through C used PRTs that were calibrated before they were mounted onto the blackbody cavity. The remaining designs used uncalibrated PRTs. The resistance measurement accuracy of all the temperature controllers was checked before and after each calibration. The expanded uncertainty at 300 K for the approximate confidence interval of 95 % (k = 2) for this set of nine blackbodies ranged from 0.24 K to 0.86 K. The feature to note in Figure 2 is that the radiance temperature of six out of nine of the blackbodies deviated by more than the uncertainty for the 95 % confidence level at some point in their calibrated temperature range. The data helps illustrate that testing requirements other than radiometric accuracy tend to degrade the performance of a blackbody source. The three Design A blackbodies showed the best calibration results and had characteristics typically associated with accurate blackbodies. The Design A blackbodies had large cavities with respect to the exit aperture size and were thermally massive (and thus slow to change in temperature). All of the other blackbodies tended to be lighter and smaller in design, favorable for trying to reduce power consumption and stabilizing time, but usually resulting in lower radiance temperature accuracy.

Once calibrated, blackbody sources are often installed into larger, cryogenic test chambers that are used to calibrate IR remote sensors. The optical systems in such test chambers often include components that can add significant uncertainty to the modeled chamber output irradiance that is presented to the sensor under test. Such components may include coated mirrors, windows or filters, beam splitters or combiners, and integrating spheres. With the accuracy requirements of remote sensing instruments becoming more stringent, the uncertainties of the output irradiance of the test chambers becomes unacceptable, and calibration of the chamber output becomes necessary. To meet this need for the calibration of infrared, low-temperature background test chambers the LBIR facility has been using a portable transfer radiometer, the BXR. It has been in service since 2001 and has calibrated a variety of test chambers. Figure 3 shows a subset of these chambers and the differences in their output performance. If the modeled output of these chambers agreed with the calibrated measurements of the BXR, all of the curves in Figure 3 would lie at 0% relative irradiance error. Instead, there is a 20% spread in the results. After calibration, however, each of these chamber models could be corrected and given traceability to an absolute scale. By measuring the chamber output irradiance over a variety of test conditions such as blackbody cavity temperature, blackbody source aperture size, and BXR filter wavelength, the components of the test chamber model contributing the largest amount of error can be identified and corrected. For most test chambers, the BXR can provide a calibration with traceability to an absolute scale with a combined uncertainty of less than 3% (k = 1).

Despite the success of the BXR, a major limitation is that absolute traceability can only be established directly to an ACR at relatively high irradiances. The lowest power ACRs currently in use can only provide an accuracy of about 1% at 10 nW of power. Below that, uncertainty grows inversely with power. This means that for the BXR, with a 7 cm diameter entrance aperture, direct traceability to an absolute standard can only be provided at irradiances of about 2.5×10^{-10} W/cm^2. As mentioned above, the BXR routinely achieves 2 % Type A ("random") uncertainties (k = 1) at irradiances as low as 10^{-14} W/cm^2 within a 60 second integration time. This means that traceability has to be extended about 4 orders of magnitude with arguments of detector linearity in order to provide calibrations at the lowest irradiances that the BXR is capable of measuring. This poses an unacceptable risk to several critical LBIR users.

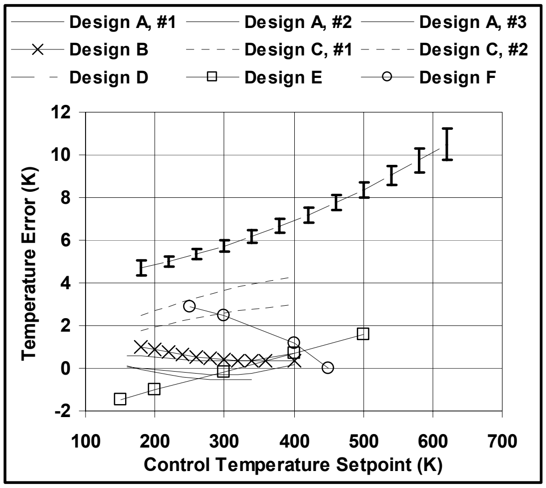

Fig. 2. Blackbody cavity radiance temperature calibration data. The temperature error is equal to the measured radiance temperature minus the contact temperature. The error bars shown for Design D are the expanded uncertainties at the estimated 95% confidence level (k = 2) and are larger than the others. The error bars for the others were removed for clarity.

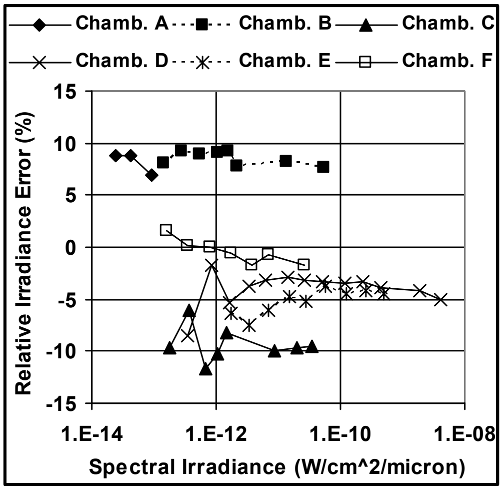

Fig. 3. The relative difference between measured and modeled IR test chamber output irradiance. The uncertainties in the data range from 2% to 5% (k = 1). Each of the data sets were measured with a blackbody temperature of 400 K through a narrow band filter centered on 10.6 µm.

Another limitation of the BXR is that it only has limited spectral capability through the use of its narrow band-pass filters. For this reason a second generation transfer radiometer, the MDXR, was developed that has within it a cryogenic Fourier transform spectrometer (FTS). In the MDXR, the sampled irradiance from the IR test chamber being calibrated can be diverted into one of two directions. In one path, the beam passes only through filters that perform a function similar to those in the BXR, thus assuring radiometric accuracy in at least those filter bands. In the other path, the beam passes through the FTS permitting a high resolution measurement of the relative spectral output of IR test chamber. Then, by combining these two data sets appropriately, a high resolution absolute spectral calibration of customer test chamber can be made. The beam splitter and compensator in the cryo-FTS are currently optimized for performance over the 4 µm to 14 µm spectral range; however, with changes of the beam splitter, compensator, and detector, the MDXR could spectrally characterize systems from the near IR out to the very far IR. In addition to calibrating the irradiance of collimated sources, the MDXR will also be able to make radiance measurements for calibrating flood or non-collimated sources.

5. FEMTOWATT CALIBRATION CAPABILITY DEVELOPMENT

To overcome the low power limitation of the current ACRs a high sensitivity “pW ACR” is being developed. It is expected to have a power range of 40 nW to 1 pW with a combined uncertainty that is no greater than 0.1% of the operating power. This represents a factor of 10,000 improvement of noise floor over the currently used ACRs. To achieve this level of noise performance, several changes in ACR fabrication practice are necessary. Two of the changes are relatively simple. First, the thermal link from the receiver cavity to the heat sink needs to be made much weaker so that fW scale changes in optical power into the receiver cavity result in a measurable change in temperature of the receiver cavity. Second, the receiver cavity needs to be made much smaller so that the time constant of the ACR remains at a manageable time of less than 1 minute. Fortunately, these two changes work well together. The very weak thermal link, which was changed from 200 K/mW to 40,000 K/mW, is also mechanically weak. However, the smaller receiver, with a 3 mm entrance aperture, is about 100 times less massive than the ones currently used. So the strength to weight ratio is approximately the same. These two changes are relatively simple, and have been realized in the laboratory on prototype assemblies.

The other factor of 50 in required performance improvement will come from improvements in the temperature sensor used on the receiver cavity. Previous ACRs have used commercially available germanium resistance thermometers. The new temperature sensors will employ a tin (Sn) superconducting transition element and a SQUID to sense the changes in the superconducting state of the Sn. Ignoring various sources of noise, the power measurement sensitivity achieved using this temperature sensing arrangement on an ACR is better than required, as shown in equation (1).

$$Noise\,Equivalent\,Power = \left(10^{-19}\,Tm^2\right)\left(\frac{2x10^{-2}\,K}{10^{-10}\,Tm^2}\right)\left(\frac{10^{-3}\,W}{40{,}000K}\right) = 5\times10^{-19}\,W \tag{1}$$

The first term is the flux sensitivity of the SQUID that we have measured. The second term is the ratio of the temperature transition width of our Sn sample to the flux change sensed by the SQUID (Figure 4). The third term is the thermal impedance of the thermal link that joins the receiver cavity to the heat sink. This level of power sensitivity will never be realized due to other sources of noise. The list of potential noise sources in terms of spectral noise power includes, but may not be limited to, background and signal photon noise, Johnson noise from the heater resistor on the receiver cavity, magnetic field noise on the SQUID sensing loop, and phonon noise in the thermal link. According to calculations, phonon noise from the thermal link poses the greatest challenge to reaching fW sensitivity, and the other sources of noise are expected to be smaller. Phonon noise associated with the thermal link was approximated using Equation (2), where G is dynamic thermal conductance, k is thermal conductivity and T_{HS} is heat sink temperature. [6]

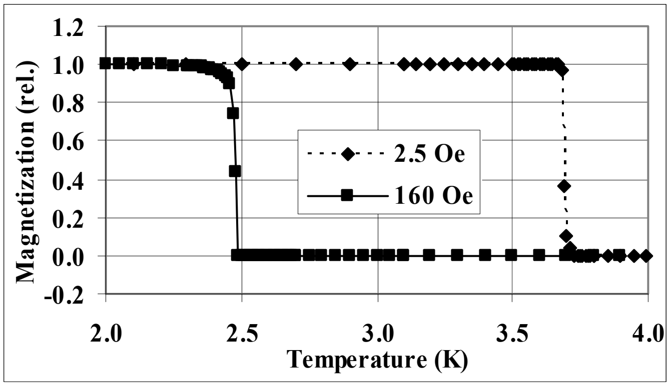

Fig. 4. The change in magnetization of a 1 mm diameter by 3 mm long piece of tin in magnetic fields of 2.5 and 160 Oersted. While the transition temperature shifted, the sharpness of the transition was not significantly changed. Thus, magnetic fields of different strengths can be used to tune the Sn transition edge sensor to operate at the temperature that is best for any particular power measurement within the 40 nW to 1 pW power measurement range, without loss of sensitivity.

$$PhononNoisePower = 4k_B G T^2 \frac{\left(\int_{T_{HS}}^{T} \left[\frac{tk(t)}{Tk(T)} \right]^2 dt \right)}{\left(\int_{T_{HS}}^{T} \left[\frac{k(t)}{k(T)} \right]^1 dt \right)} \tag{2}$$

A spectral noise density of about 2 fW is estimated assuming a 1 Hz band width, G = (40,000K/mW)$^{-1}$, a heat sink temperature of T_{HS} = 1.9 K, and a receiver cavity temperature of T = 2.0 K. The quotient containing the integrals of the thermal conductivity of the heat link, k(t), in the equation is approximately equal to unity. Based on the current state of materials development, the modeled noise performance and the capabilities of phase sensitive detection methods, it is anticipated that this proposed pW ACR shall be able to provide absolute traceability to power measurements at power levels as low as 1 pW with an approximate 1 fW noise floor.

A significant drawback of the pW ACR is that it can only be operated in a stable, 2 K background environment. Above 2 K, noise from background radiation significantly increases the overall noise floor of the instrument. This makes it difficult to realize the new pW power scale in a practical way because stable 2 K background environments are only easily created on small scales, such as inside liquid He cryostats. This does not lend itself well to the 15 K background, large scale calibration environments used to calibrate the BXR.

In order to overcome this problem, a transfer standard is being developed that can be calibrated by the pW ACR in the 2 K environment, and then be placed into the 15 K to 20 K environment of the LBIR calibration chambers for the calibration of the BXR. For this purpose a trap detector is being developed that will consist of two As-doped Si BIB detectors placed in a trapping configuration. The use of solid state detectors in trapping configurations as high quality transfer standards is common [5, 7]. BIB detectors will work well for this task because of their very high sensitivity, and their low-temperature operation. Their drop in responsivity at wavelengths above 30 μm makes their sensitivity to background temperatures below 20 K very weak; thus the contribution to noise from the background does not change significantly between the 2 K background of the pW ACR environment and the 20 K background of the LBIR calibration chambers. The first BIB trap to be developed will consist of two 10 mm x 7.5 mm BIB detectors facing each other at an angle of 14 degrees as shown in Figure 5a. The entrance aperture to the trap will be 3 mm in diameter and all photons entering into the trap within an f/4 full cone will, if not absorbed within the active layer of a detector, strike a detector active surface at least 7 times before striking a reflective wall inside the trap or escaping through the entrance aperture. Based on transmission and reflection measurements made on BIB detector material, and the designs of the BIB trap and the new large area detectors currently being fabricated, it is expected that the internal quantum efficiency of the BIB trap will be greater than 98% from about 3 μm to about 29 μm in wavelength. The modeled absorptivity at the entrance aperture of the BIB trap is shown in Figure 5b. The manufacturer of the new BIB detectors expects their internal quantum efficiency to between 0.99 and 1.00. Therefore the spectral quantum efficiency of the BIB Trap is expected to be the absorptivity of the BIB Trap multiplied by the expected internal quantum efficiency. The BIB detectors are in their final fabrication stages and are expected to be delivered by the end of July, 2008.

The BIB traps are expected to be useful in multiple applications. For use as a transfer standard, where noise performance and responsivity linearity are important, the optimization of the BIB trap performance can be controlled by temperature. When operated at about 5 K the noise equivalent power (NEP) of the BIB trap will be ≈1 fW and the signal from the detector will deviate from linearity by 0.1% at ≈30 pW. At a temperature of 16 K the BIB traps will have an NEP of ≈100 fW and the signal from the detector will deviate from linearity by 0.1% at ≈300 μW. Another very practical feature of the BIB trap is that it is very flat in spectral response. This will make it well-suited for spectral applications where a detector with a complicated spectral response would significantly increase measurement uncertainty. Therefore we anticipate using the BIB traps in the BXR and MDXR. The very flat spatial response of the BIB trap will also make the device very tolerant of systems with vibration. The BIB detectors that make up the trap are also very fast; these are expected to have a 3 dB roll-off of about 1 MHz at their warmer operating temperatures. This makes the BIB traps well-suited for Fourier transform spectroscopy applications. In addition, the BIB traps have practically no back reflected signal, which is a very practical feature for systems such as Fourier transform spectrometers and other systems with transmissive optics that can suffer from back reflections from detectors.

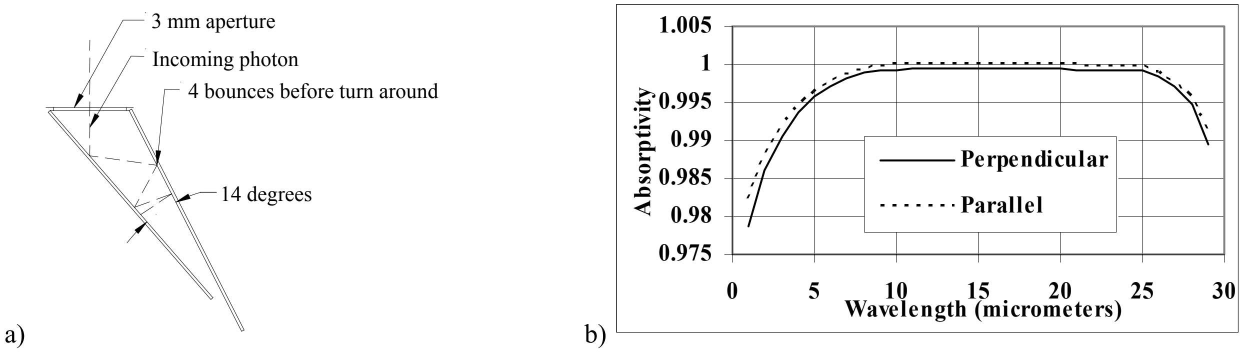

Fig. 5. a) Schematic representation of the first BIB trap under development. b) The modeled BIB Trap absorptivity for polarizations perpendicular and parallel to the plane formed by the detector surface normal and the incident ray. The expected detector internal quantum efficiency is between 0.99 and 1.00, so the spectral responsivity of the detector should be somewhere between 99% and 100% of the modeled BIB Trap absorptivity.

6. SUMMARY AND CONCLUSIONS

The need for radiometric calibration of IR cryogenic test equipment was reviewed. The cryogenic vacuum environment and typical radiometric test requirements make test hardware development constraints very stringent and thus the development of accurate radiometric sensor calibration systems very difficult. Therefore, for the most stringent accuracy requirements, radiometric calibration at the source output must be made.

The feasibility of pW absolute traceability in the IR with ≈1 fW noise floor was demonstrated. Prototypes of the most critical components of an ACR were constructed and tested, demonstrating that the power and noise floor requirements for absolute traceability are within practical reach. In addition, significant progress was made on a BIB trap detector that is necessary for the practical realization and dissemination of this new low-power calibration capability. Once completed, the BIB traps are expected to become easily distributed radiometric calibration standards of very high accuracy, and they should be useful in many spectroscopy applications in the 2 μm to 30 μm spectral range.

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