

Infrared Spectrophotometry; Some of the Pitfalls

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Abstract

Spectrophotometry is a relatively straightforward technique compared to, for example, infrared spectroradiometry because the former does not require the definition of a radiometric zero, nor does it require the use of a standard. Two of the most important requirements of spectrophotometry are for the detection system to be stable and to have a linear response over the range of incident radiant powers used for a particular measurement. However, recent work by the author has shown that the identification of an infrared detector with a responsivity which does not drift with time and has a linear response is far from straightforward. The detector linearity requirement is particularly difficult to satisfy in the case of spectrophotometers based on Fourier Transform (FT) spectrometers because at the vicinity of the “zero path difference” point, the photodetector receives the full spectral output of the source and is therefore likely to run into a non-linear regime. Infrared radiometrists should also be aware of a number of phenomena which give rise to changes/drifts in the responsivity of infrared detectors and which can result in serious errors in spectrophotometric measurements. This paper emphasizes the very limited range over which the responsivity of some infrared detectors is linear and summarises a number of phenomena which give rise to large drifts in the responsivity of infrared detectors. The paper also highlights how important it is to have an infrared detector with a wide linearity range when developing spectrophotometers utilising FT spectrometers.

1. Introduction

The quality of a measurement is governed by the combined uncertainty associated with that particular measurement. This assumes that a rigorous uncertainty budget has been developed which includes all components that contribute to the total uncertainty of the measurement. Recently NPL has reported, in a series of publications, a number of phenomena which give rise to drifts in the responsivity of infrared detectors [1-5]. NPL has also reported the linearity of response characteristics of a number of widely used infrared detectors and showed that the range over which their responsivity is linear is very limited [6-8]. This means that a number of new uncertainty contributions must be introduced in the uncertainty budget which will cause the combined uncertainty of infrared spectrophotometric measurements to increase.

The purpose of this paper is to summarise the non-linear response and drifts in the responsivity of infrared detectors and highlight how they can give rise to serious uncertainty contributions in infrared spectrophotometric measurements. It is important to stress that there are a host of other phenomena such as atmospheric absorption [1], the spatial uniformity of response of infrared detectors [9] etc which can also contribute to the combined uncertainty of infrared spectrophotometric measurements. However, these will not be considered in this paper.

2. Uncertainties due to the non-linear response of infrared detectors

A photodetection system is linear if its output is directly proportional to the level of incident radiation. The linearity of the response of infrared detectors is a seriously neglected phenomenon and the linearity of photodetection systems is often assumed rather than demonstrated.

Until recently, there were no serious scientific studies/publications dealing with the linearity characteristics of infrared detectors, despite their widespread use.

Radiometrists have adopted the “linearity factor” as a means of quantifying the linearity characteristics of photodetectors [6]. The linearity factor $L(V_{A+B})$ of a photodetector for an output of $(V_A+V_B)/2$ is given by:

$$L(V_{A+B}) = \frac{V_{A+B}}{(V_A + V_B)} \dots\dots\dots(1)$$

where V_A , V_B and V_{A+B} represent the zero-corrected signals from the photodetector when apertures A, B and A+B were open, respectively, with V_A set to be approximately equal to V_B [6]. For a linear detector the value of $L(V_{A+B})$ will be equal to unity. Where $L(V_{A+B})$ deviates from unity, its value can be used to estimate correction factors to apply for different detector output signals to transfer the absolute responsivity calibration from one level to another [10]. Another useful parameter in defining the non-linearity of a detector is the *deviation from linearity* or *non-linearity* which is given by $1 - L(V_{A+B})$.

Spectrophotometry involves the measurement of the radiant power of a beam of optical radiation with and without an artifact inserted into the beam. The transmittance of the artifact is defined as the ratio of these two measurements. The deviation from linearity in the response of the photodetection system is, arguably, the parameter contributing the largest uncertainty component in infrared spectrophotometric measurements.

The author was the first to report absolute linearity measurements of the response of some infrared detectors. So far the non-linearity characteristics of Photo Conductive (PC) and Photo Voltaic (PV) HgCdTe detectors [6], PbS detectors [7] and PbSe detectors [8] has been reported. PC HgCdTe, PbS and PbSe detectors are intrinsic photoconductors [11]. This means that they offer high D^* values [11] at relatively high operating temperatures which, in the case of PbS and PbSe as well as in some HgCdTe applications, can be attained by thermoelectric cooling, hence their widespread use. PC and PV HgCdTe detectors are widely used in infrared spectrophotometry in the 5 μm to 26 μm wavelength region. PbS was until recently the detector of choice for most detection applications in the 1.6 μm to 3 μm wavelength region while PbSe detectors are widely used in photodetection applications in the 2 μm to 5 μm wavelength range [11].

Recent measurements by the author have shown that the non-linearity of HgCdTe, PbS and PbSe detectors cannot be quantified in terms of the voltage output or indeed in terms of the radiant power incident on the active areas of these detectors [6]. Instead, it was shown that in the case of PC HgCdTe and PC PbSe detectors, it is the photon irradiance which defines the linearity factor of these detectors [6, 8]. In the case of PC PbS detectors the parameter of interest was shown to be the spectral irradiance [7] which suggests that another effect such as the heating effect of the incident radiation

may have a significant role in the non-linear behaviour of PbS detectors. What is particularly worrying to users of these detectors is that their non-linearity is significant even for modest levels of incident irradiance. In the case of the PbS detector, for example, a deviation from linearity of 1% was measured for an incident radiant power of 1 nW concentrated in an area of 1 mm² [7]. The author expects that some PbS detectors currently being used in commercially available equipment are illuminated with higher levels of spectral irradiance and are therefore likely to be operating in a non-linear regime.

Fig. 1 shows a plot of the linearity factor of a PbS detector versus output voltage under different illumination conditions (plots corresponding to different beam diameters and wavelength of the incident beam). It is obvious that the non-linearity cannot possibly be correlated to the output voltage without additional information. Fig. 2 shows the corresponding data for a PbSe detector [8]. Again, the deviation from linearity cannot be directly correlated to the radiant power incident on the detector. It is therefore clear that attempts to correct for the effect of the non-linear response of these detectors based on voltage output or even incident radiant power are bound to fail. The wavelength of the incident radiation as well as the area of the detector being illuminated has to be taken into consideration.

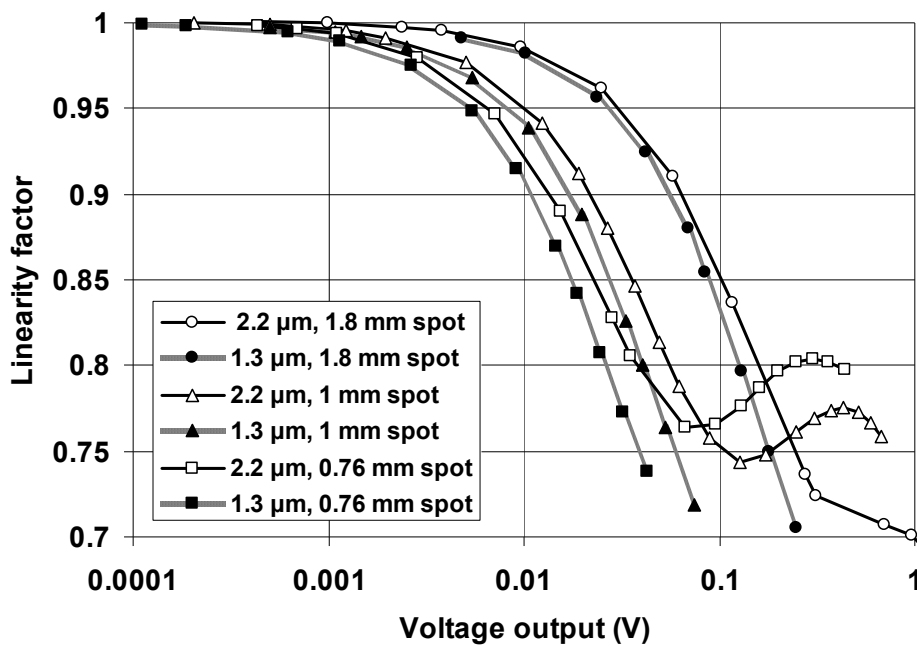


Fig. 1. Linearity factor of a Hamamatsu PbS detector as a function of the output voltage at 1.3 μm and 2.2 μm for 0.76 mm, 1 mm and 1.8 mm diameter spots illuminating the active area of the detector [7].

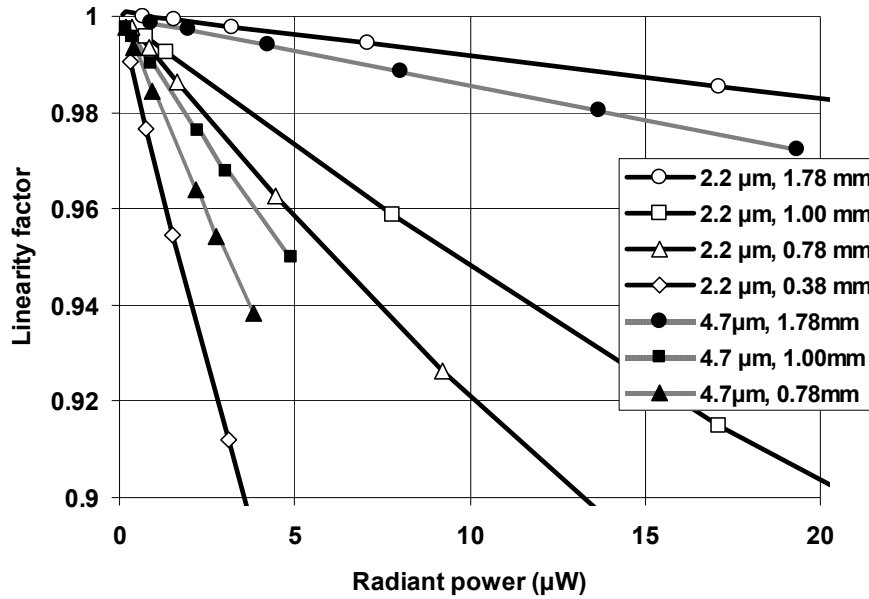


Fig. 2. Linearity factor of the Judson PbSe detector as a function of the incident radiant power at 2.2 μm and 4.7 μm wavelength, for different spot diameters [8].

In the case of the PC HgCdTe detectors, the non-linear behaviour can be explained on the basis of the electron-hole recombination (Auger recombination) which is the dominant loss mechanism of photo-generated charge carriers in this type of photodetector. The density of the photo-generated charge carriers is proportional to the photon irradiance. However, at higher carrier densities, there is a higher probability of electron-hole recombination (Auger recombination) and therefore, as the photon irradiance increases, the carrier lifetime decreases and therefore the non-linearity increases.

3. Implications of the detector non-linearity in FT spectrophotometry

Infrared spectrophotometry is now mostly being done using FT spectrometers because they offer significant advantages compared to monochromator-based spectrophotometers [12]. An FT spectrometer is basically a Michelson interferometer where the optical path difference between the passive arms can be varied. The radiant power reaching the detector changes with path difference, resulting in the acquisition of the interference function or “interferogram”. The voltage output of the photodetector and hence the radiant power incident on it can be recovered by performing a Fourier Transform on the interferogram. However, if the interferogram is not a true representation of the radiant power incident on the detector due to (for example) the response of the photodetector being non-linear, then the spectrum generated by the Fourier Transform process of the interferogram will not be a true representation of the spectral power reaching the photodetector.

How can the interferogram fail to be a true representation of the radiant power incident on the photodetector? Fig. 3 shows a typical interferogram obtained from an infrared FT spectrometer. It is characterised by small variations in the output except for some very large variations near the zero path difference between the two interferometer arms. At this position, the photodetector “sees” the full radiant output of the source captured within the entendue/throughput [13] of the instrument. This is in contrast to a monochromator-based spectrophotometer where the output at different wavelengths is

observed sequentially. The wavelength bandwidth of a monochromator-based system is typically 1% of the working wavelength so the strength of the optical radiation incident on the photodetector is (typically) two orders of magnitude lower than the signal corresponding to the peak of an FT interferogram. The author has observed significant non-linearities in radiometric measurements completed using monochromator-based systems [7]. Since the photodetectors in FT-based spectrophotometers are illuminated with values of radiant power which are two orders of magnitude higher than in a monochromator-based system, it is safe to assume that spectrophotometric measurements based on FT spectrometers can suffer from the effects of detector non-linearity, unless precautions have been taken to prevent this from happening.

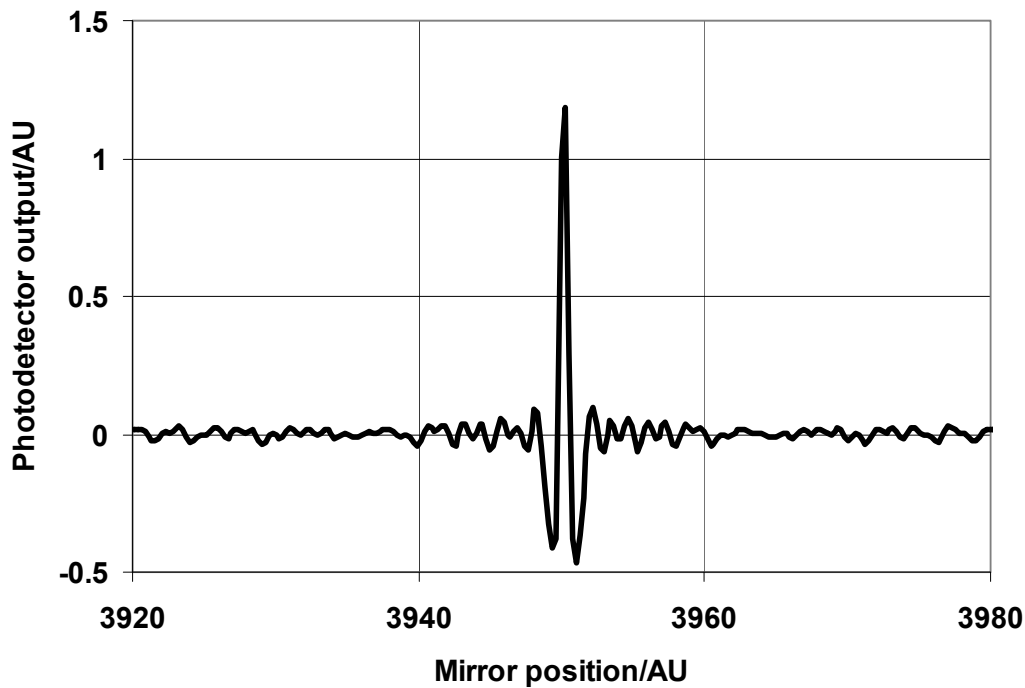


Fig.
3. FT interferogram demonstrating the higher output signal observed in the vicinity of the “zero path length difference” between the two interferometer arms. (courtesy of C. J. Chunnillall, NPL)

Errors due to detector non-linearity in radiant power measurements using FT spectrometers can be in excess of 30% [14, 15]. FT instrument manufacturers are aware of these potential sources of error and market methods of correcting the errors which can arise due to the non-linear response of infrared detectors. One method relies on special preamplifier electronics to overcome the effects of detector non-linearities [16]. Others market software which promise to achieve the same goal [17]. However, a correction method based on the electrical gain of a pre-amplifier assumes that the detector non-linearity can be characterised in terms of the voltage output, something which is contradicted by data published recently [6 - 8]. A correction method based on software algorithms also assumes that the conditions with which the photodetector is illuminated are known, which, again, is not necessarily the case.

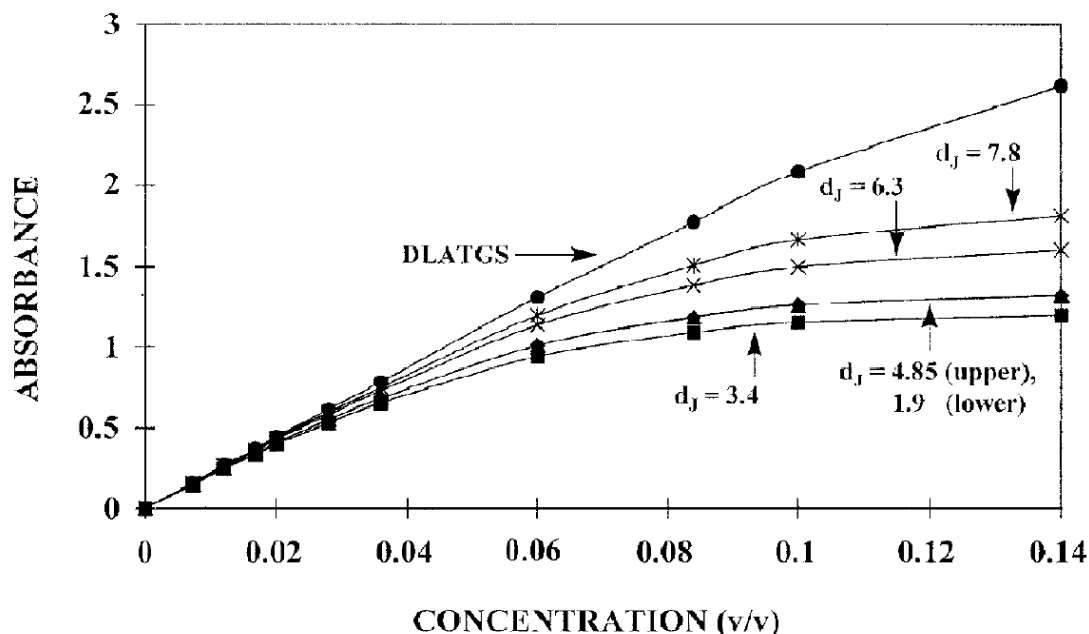


Fig. 4. The absorbance of a particular absorption band does not vary linearly with the concentration of t-butyl ether in CCl_4 . Deviation from Beer's law is caused by the detector non-linearity (figure reproduced from reference 18).

Fig. 4, reproduced from reference 18, shows how the absorbance of the $3.38\ \mu\text{m}$ t-butyl ether absorption band changes as the concentration of t-butyl ether in CCl_4 increases. These measurements were performed using an FT spectrophotometer utilising a HgCdTe and a DLATGS pyroelectric detector. If Beer's law was obeyed, then all the plots in Fig. 4 should have been straight lines. Figure 4 shows that the plots exhibit strong deviations from linearity, suggesting that Beer's law is not obeyed. In fact Griffiths and co-workers showed [18] that these deviations from linearity are caused by the non-linear response of the photodetectors used. Plots using HgCdTe detectors exhibit much more severe excursions from Beer's law compared to the corresponding plots acquired using DLATGS detectors. This is to be expected because the linearity characteristics of HgCdTe detectors are considerably poorer compared to those of DLATGS pyroelectric detectors. Fig. 4 shows that the concentration of t-butyl ether in CCl_4 will be estimated incorrectly even for concentrations as low as 2% and under certain conditions, the measured absorbance will never exceed unity irrespective of the concentration of t-butyl ether in CCl_4 . This demonstrates the importance of the detector linearity in FT spectrometer based spectrophotometers. An extensive discussion on the practical implications of the photodetector non-linearity on the accuracy of photometric measurements acquired using FT spectrometers can be found elsewhere [18, 19].

4. Uncertainties due to drifts in the responsivity of infrared detectors

Some researches will argue that problems due to detector non-linearities can be eliminated from FT-based spectrophotometric measurements by choosing detectors such as InSb detectors which are believed to have a linear response. Unfortunately the response of InSb detectors is limited to wavelengths shorter than $5.3\ \mu\text{m}$ and HgCdTe detectors remain (by far) the most widely used photon detectors capable of detecting wavelengths longer than $5\ \mu\text{m}$ at relatively high operating temperatures (77 K).

Furthermore, the range over which the response of InSb detectors is linear has also been shown to be restricted [20]. Finally the responsivity of InSb detectors, like all cooled detectors, has been shown to drift with time and this effect will also introduce uncertainties in infrared spectrophotometric measurements. A brief summary of this behaviour is presented below.

The performance of infrared photodetectors benefits greatly from cooling the photodetectors to cryogenic temperatures [11]. For this reason, the vast majority of infrared photodetectors are sold mounted in evacuated Dewars where they can be cooled to temperatures below 80 K using cryo-coolants such as liquid Nitrogen or liquid Helium. Work at NPL has shown that moisture can also go past the O-rings sealing the Dewar window and evacuation port [21]. Once the Dewar is cooled, the Dewar cold finger (which includes the infrared detector) acts like a cryo-pump so that the moisture deposits as a thin film of ice on all cold parts of the Dewar, including the surface of the infrared detector. Ice is known to have a number of strong absorption bands in the infrared, so the film of ice deposited on the detector preferentially absorbs wavelengths coinciding with the ice absorption bands [1, 4]. This causes the responsivity of the infrared detector at wavelengths coinciding with the ice absorption bands to decrease with time, causing drifts of many percent per minute at wavelengths around the 3.1 μm absorption band [1], as can be seen from Fig. 5. Significant drifts have also been observed around 12 μm where another strong ice absorption band is present [1, 3, 4]. Fig. 5 shows that the responsivity of this detector decreases by 30% over the period of 12 minutes and provides an indication of the magnitude of the expected drift. It should also provide a warning to users of cooled infrared detectors and demand that infrared spectrophotometric measurements utilising such detectors must include corrections as well as uncertainty contributions to take into account the consequences of this phenomenon.

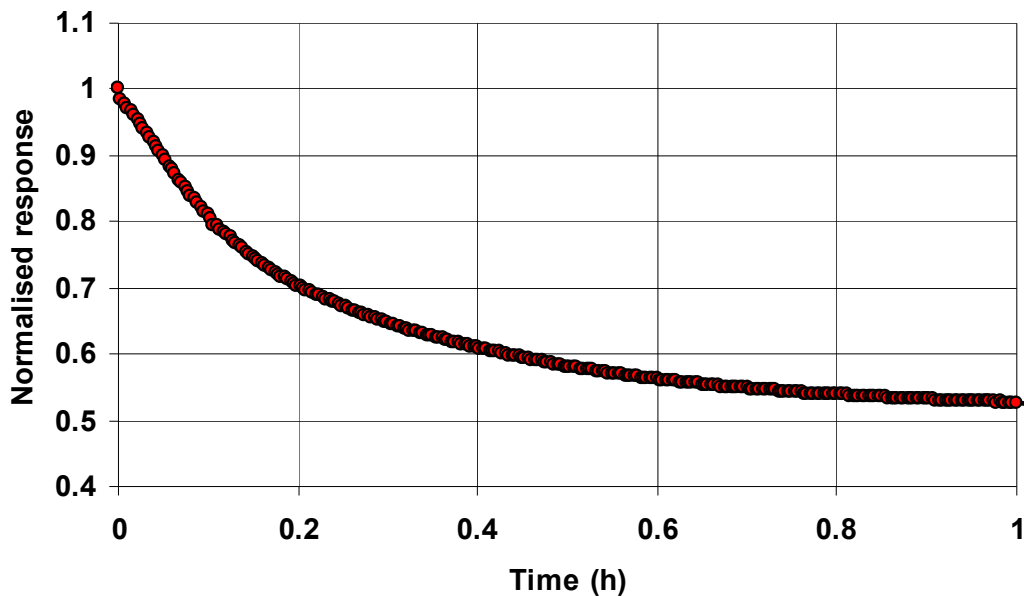


Fig. 5. Normalised response of an InSb detector monitoring radiation at 3.1 μm , immediately after cooling to 77 K.

However, drifts are not restricted to wavelengths coinciding with the absorption bands of ice. Ice has strong dielectric properties and when it is formed on the active area of photodetectors, it interacts with the multi-layer dielectric anti-reflection coatings deposited on the surface of the photodetectors. This causes the transmission of the anti-reflection coatings to change and thus the detector responsivities to drift with time [2, 3]. This effect was observed in InSb detectors [2], HgCdTe detectors [3] as well as in silicon detectors (in the visible!) cooled to -40°C . This implies that the responsivity of a cryogenically cooled detector can be expected to drift for all wavelengths where the detector has a finite responsivity.

The introduction of cold filters restricting the range of wavelengths reaching cryogenically cooled infrared photon detectors and blocking some of the thermal background radiation, results in a reduction in the total noise power at the output of the detector [11]. This provides a well-established technique for improving the detector Noise Equivalent Power (NEP) and specific detectivity (D^*) of infrared detectors over specific wavelength ranges [11]. The cold bandpass filters are mounted on the cold shield of the detector so any moisture inside the Dewar vacuum deposits on the bandpass filter rather than the detector which it shields. The bandpass filter is nothing more than a stack of dielectric layers designed to have a specified transmission profile. The deposition of a film of ice a few nanometres thick, interacts with the dielectric structure of the filter and can alter its transmission characteristics, causing the response of the filter/detector combination to change. The response of the filter/detector combination was observed to decrease at some wavelengths and to increase at others due to the deposition of ice. Numerical modelling and a dedicated experiment have confirmed that the deposition of a thin film of ice on the dielectric bandpass filter is the cause of the observed drifts [4].

In the absence of alternative good quality detectors, InSb detectors with low-pass filters mounted on their cold shields are used extensively in the $1.6\text{ }\mu\text{m}$ to $2.6\text{ }\mu\text{m}$ wavelength range. The filters are nothing more than glass plates which transmit wavelengths below about $3\text{ }\mu\text{m}$ but block radiation of longer wavelength. Because they are cold (they are mounted on the cold shield of the detectors) they block the thermal background radiation of wavelength longer than $3\text{ }\mu\text{m}$. When moisture is present in the Dewar vacuum, it deposits on the cold glass plate and it behaves like a thin film of oil on the surface of water. This means that as the thickness of the film of ice grows, the transmission goes through maxima and minima [6]. The response of the filter/detector combination exhibits an identical behaviour as can be seen in Fig. 6. The “oscillations” are “fast” at first as there is a lot of moisture in the Dewar vacuum. As the moisture is deposited as ice, it is gradually depleted, so the deposition rate decreases, causing the frequency of the “oscillations” to reduce, as seen in Fig. 6.

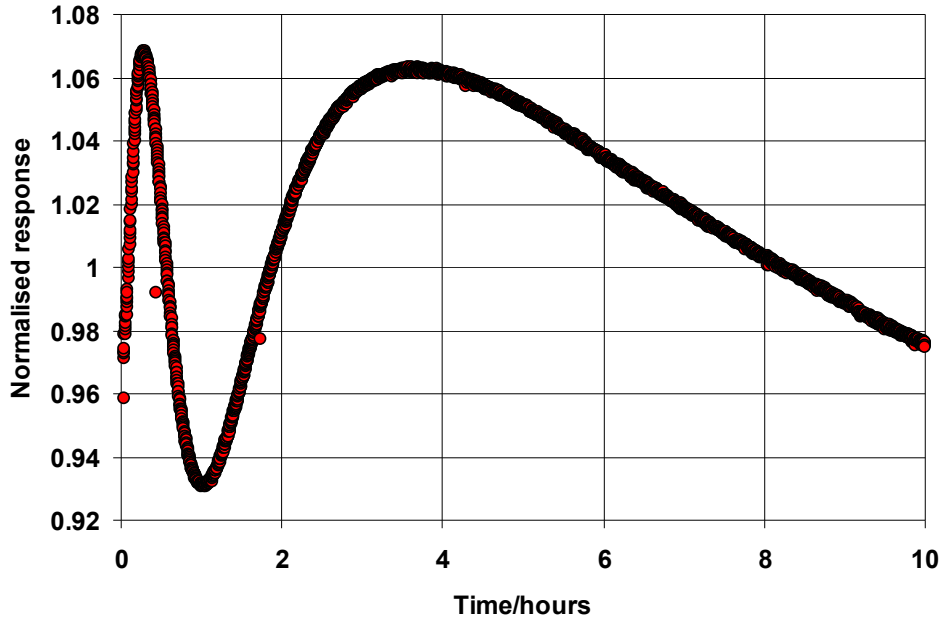


Fig. 6. Normalised response of an InSb filtered detector monitoring radiation at 1.55 μm , immediately after cooling to 77 K

Fig. 6 shows that the responsivity of this detector “oscillates” with a peak to peak amplitude of approximately 14% and confirms that it can be an important uncertainty contribution in infrared spectrophotometric measurements.

The drifts in the responsivity of cryogenically cooled detector are temporarily eliminated after evacuation and baking of the Dewar at around 60 °C. It must be stressed that the elimination of these drifts is only temporary. Drifts slowly reappear and they grow in magnitude as moisture enters the Dewar vacuum past the O-ring seals.

Spectrophotometric measurements are based on the measurement of the radiant power of a beam with and without the artefact inserted in the beam. These two measurements can only be done sequentially and the period between these two measurements can vary from a few seconds to many hours. In some field measurements, a measurement of the reference (artefact out of the beam) is recorded at the beginning and end of the day whereas all measurements during the day include the artefact in the beam. The work described above indicates that such measurements can be seriously flawed due to the drifts which can occur in the responsivity of infrared detectors. The extent of the problem depends on the wavelength range of the measurements, the type of detector used and on the type of Dewar in which the detector is housed [21].

5. Uncertainties due to drifts in the thermal background

Yet another effect which can give rise to significant drifts in the responsivity of infrared detectors such as HgCdTe detectors has recently been reported [22]. The author has shown that the responsivity of these detectors depends strongly on the unmodulated (background) infrared spectral irradiance and therefore on the temperature of objects in the FOV of these detectors. This effect was largest in PC HgCdTe detectors [22] and was shown to arise due to the non-linearity of response characteristics of these detectors. The incident radiation generates free charge carriers. Auger recombination is

a significant loss mechanism and this loss mechanism is known to rise with the increasing density of charge carriers. This leads to the observed high non-linearity in the response of HgCdTe detectors. Note that both the modulated radiation being detected as well as the thermal background radiation can produce free charge carriers which contribute to the non-linearity. The contribution due to the thermal background is rejected by the phase sensitive detection utilised, but its contribution to the non-linearity of response of the detector cannot be avoided. The irradiance due to the thermal background was estimated to be over an order of magnitude greater than the irradiance due to the modulated radiation being detected [22]. Thus the thermal background is the dominant source of free charge carriers and is, therefore, the main component of the observed non-linearity in the response of infrared HgCdTe detectors. This phenomenon is expected to provide a practical limit to the accuracy of radiometric measurements using not only HgCdTe detectors, but also other detectors whose linearity is a function of the thermal background [22]. Fig. 7 shows the normalised response of a PC HgCdTe detector at $4.7\ \mu\text{m}$ versus time as the temperature of the FOV was raised in steps of approximately $3\ ^\circ\text{C}$. Also plotted in the same figure are the values of the temperature of the field of view. The responsivity of HgCdTe detectors was shown to decrease by approximately 0.7% per $^\circ\text{C}$ increase in the background (ambient) temperature, but the exact value of this change is governed by many factors such as the background temperature, the FOV of the photodetector and detector spectral responsivity [22]. Observed drifts in the calibration of infrared radiometers can easily be assigned to this phenomenon. This means that the accuracy of infrared spectrophotometric measurements utilising cooled photon detectors such as HgCdTe detectors will be affected by this phenomenon. The uncertainty contribution due to this effect must, therefore, be estimated and included in the uncertainty budget of the relevant measurement.

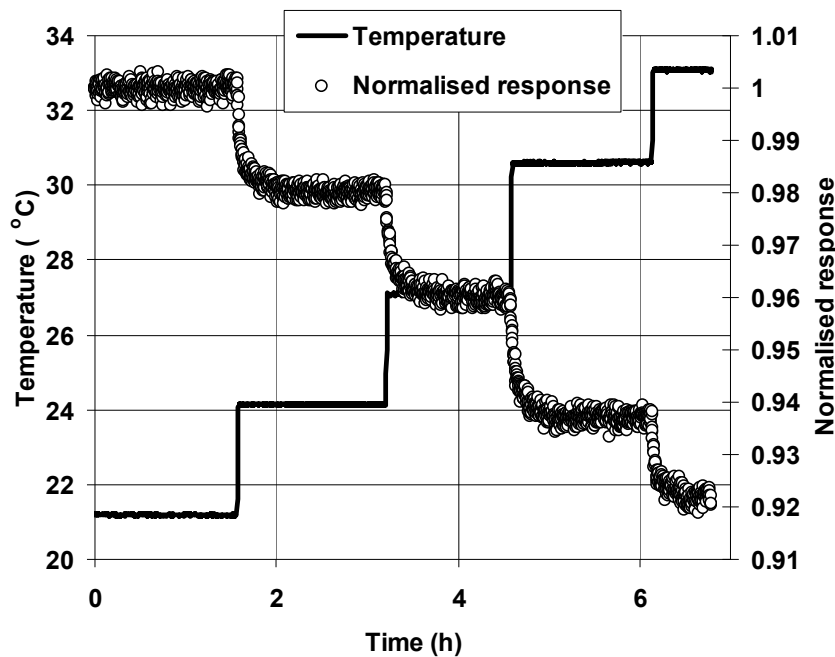


Fig. 7. Normalised response of the CMT9 detector at $4.7\ \mu\text{m}$ versus time as the temperature of the FOV was raised in steps of approximately $3\ ^\circ\text{C}$. Also plotted in the same figure are the values of the temperature (reproduced from reference 22).

6. Conclusions

The main requirement of spectrophotometry is that the detection system has to have a linear response over the range of incident radiant powers used for a particular measurement. A photodetection system whose spectral responsivity does not drift is also highly desirable. This paper shows that the identification of an infrared detector with a linear response is not straightforward. Investigation of the linearity characteristics of three types of infrared detectors has shown their responsivity to be highly non-linear even for relatively low levels of radiant power incident on these detectors. In the case of spectrophotometers based on Fourier Transform (FT) spectrometers the problems are likely to be significantly worse because in the vicinity of the “zero path difference” point, the detector receives the full spectral output of the source captured within the instrument etendue, thus increasing the likelihood that the detector operates in its non-linear regime. The responsivity of cooled infrared detectors was also shown to drift significantly with time, thus requiring this drift to be quantified and included as a correction and/or as an extra uncertainty component. Finally another phenomenon is highlighted which can also generate apparent drifts in the responsivity of infrared detectors. The origin of this source of drift is changes in the thermal background signal incident on infrared detectors.

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