Laser techniques for high accuracy spectrometric measurements of parallel-sided samples.

Jessica Cheung, Eric Usadi and Christopher Chunnilall

National Physical Laboratory, Hampton Road, Teddington, TW11 0LW

Abstract

A laser technique for measuring the reflectance of small cross-sections (~ 100 μm) of samples with parallel-sided surfaces is described. The measurement of the reflectance or transmittance of parallel-sided samples can suffer from inter-reflections. This paper describes how a knife-edge can be used to remove interference effects and the considerations that must be taken into account in order to determine the uncertainty of the measurement. The overall uncertainty in the reflectance measurement of a low reflectance (0.14%) surface is 0.014% (k=1).

Introduction

The measurement of transmittance and reflectance of small samples can be very challenging. By small we mean samples of cross-sectional dimensions < 1 cm and thickness on the order of mm and less. Two factors that make such samples challenging to characterise are:

- Small beam cross-section required to characterise sample can lead to non-linear effects in the detector;
- Inter-reflections between parallel-sided surfaces.

The sample may be a thin window used on a cryostat, crystals, scintillators, or small optical components such as lenses and filters. We describe the characterisation of a non-linear crystal which generates correlated photons used in the measurement of the quantum efficiency of photon counting detectors [1, 2]. Absorption in the non-linear crystal itself can prevent the correlated photons from reaching the detectors. It is therefore essential to measure these losses, which in turn require measurement of the crystal transmittance and the reflectances at the crystal boundaries. There are the complications that the two surfaces of the crystal have different anti-reflection coatings and the correlated photons are produced at an angle to the crystal surface. The requirement was to ascertain whether the loss due to the crystal could be measured with an uncertainty of less than 0.1%.

One transmittance measurement technique that can be used to avoid the effects of inter-reflections is through the use of a mode-locked laser. Hartree et al. [3] demonstrated the improved transmittance measurements of a glass plate (3 mm thick) using a mode-locked laser with pulse width 300 μm long.

The correlated photon application requires measurement of each face of the crystal in order to take account of the anti-reflection coatings of this particular crystal. This paper describes an experimental set up to measure the transmittance and reflectance of such a crystal. In particular, the paper demonstrates that it is possible to remove inter-reflection effects through the use of a knife-edge. In this way, it was possible to measure the front surface reflectance of each surface of the crystal.

Experimental details

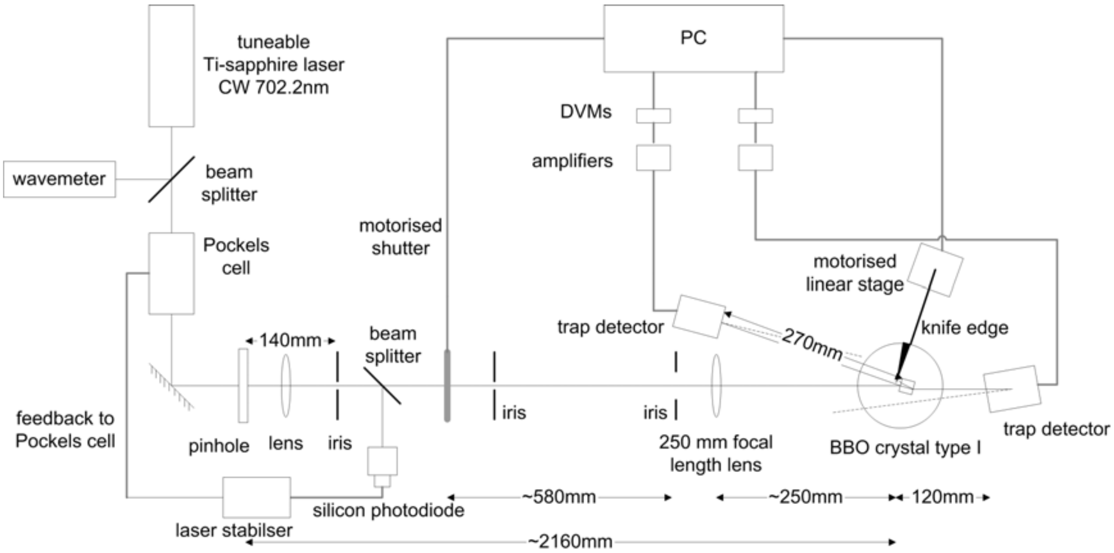


Fig. 1. Schematic of measurement set up for reflectance and transmittance measurements from BBO crystal.

Fig. 1 shows the experimental set up. The sample is a BBO (beta-barium borate) non-linear crystal, see Fig. 2, polished face area (7 mm x 7 mm), thickness 5 mm. The crystal has been anti-reflection coated on both surfaces S1 and S2.

S1: AR coat (Broad band: 345 – 370 nm)
S2: AR coat (Broad band: 345 – 370 nm and 680 – 800 nm)

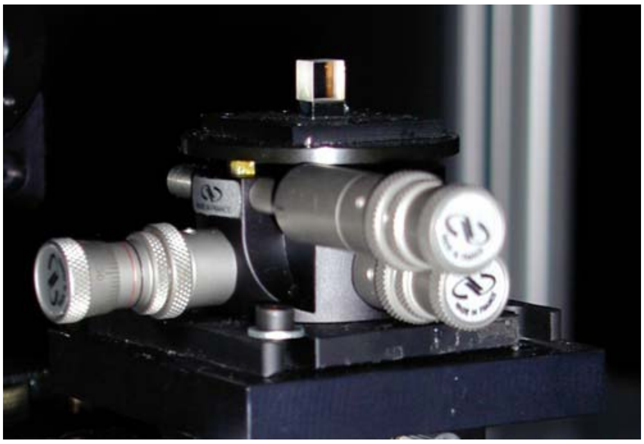


Fig. 2. Photograph of crystal

Experiment

The following sections describe the alignment of the laser, crystal, knife-edge and detectors and the measurements necessary to assess the uncertainties of the system.

Ti-sapphire laser

A Ti-sapphire laser (Fig. 1) was used to produce high power monochromatic radiation at 702.2 nm. The laser power was measured to have a standard deviation of 0.0026% over a period of 1.5 hours. A beam splitter was used to pick off part of the beam to monitor the wavelength. The Pockels cell was used to stabilise the intensity of the laser using the feedback from the silicon photodiode that sits just after the spatial filter assembly. The spatial filter was used to clean up the beam and the resultant image is an Airy disc. The iris was opened up such that it only allows the bright central fringe to pass through. A long focal length lens was then used to produce a beam waist of 100 µm diameter. A CCD camera mounted in place of the crystal was translated along the optic axis to assess the size of the beam waist. The measurements indicated that the beam waist was within the upper and lower limits set by the calculation below.

Laser beam waist

In order for the front and rear face reflections from the crystal to be spatially distinct, the laser beam diameter must be smaller than the separation between these two reflected beams. For a Gaussian beam with $1/e^2$ intensity radius of x, 99.85% of the total beam energy is contained within a radius of 1.8 x, and 99.97% within a radius of 2.0 x. Therefore to measure the single face reflectance to better than the 0.1% level requires that each beam can be measured over a radius of $2x$, and that therefore the separation, h, between the beams needs to satisfy:

$$h \geq 4x_0 \qquad 1$$

Here x_0 is the beam waist, i.e. the radius at the narrowest point in the beam. The value of h is determined from simple ray tracing of a ray incident at a particular angle on a parallel face crystal.

A further restriction comes from considering that the second face reflectance will diverge into the path of the first face reflectance unless the Rayleigh range (i.e. the distance from the waist over which the beam radius expands by $\sqrt{2}$) is longer than one return trip of the beam through the crystal. If λ is the wavelength, n is the crystal refractive index, and β is the angle of refraction within the crystal, then $2nL/\cos\beta$ is the optical length of this return trip. If Z_R is the Rayleigh range, then this condition requires that

$$Z_R = \pi \cdot x_0^{\,2} / \lambda \;\geq\; 2nL/\cos\beta \qquad 2$$

Combining Equations (1) and (2) yields the acceptable beam waist range:

$$h/4 \geq x_0 \geq \sqrt{2n\lambda L / \pi \cos\beta} \qquad 3$$

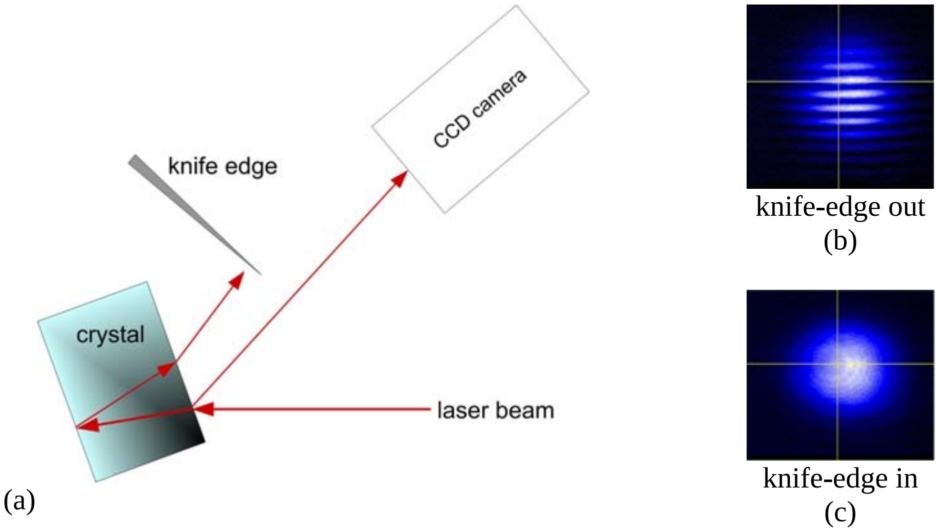
In our setup, where the angle of incidence is 6° and $n_o = 1.666$, equation (3) requires the beam waist to be between 61µm and 157µm. This shows that there is scope to trade off beam size and divergence.

The crystal

The crystal was mounted on a linear stage on top of a rotation stage so that the first surface was above the centre of the rotation of the rotation stage. The rotation stage and crystal were turned through 90° and the linear stage was adjusted until the beam was just skimming across the front face. This positioned the first surface above the centre of rotation. Small rotations away from normal shows that the laser beam does not move across the crystal surface, demonstrating that the first surface is indeed above the centre of rotation. Another linear stage at the bottom of the whole assembly moves the crystal in and out of the beam. All the measurements are carried out with the crystal rotated such that the incident beam is at 6° to the normal (this is the angle of interest with respect to the quantum efficiency measurement application described in the introduction).

Knife-edge

Before mounting the trap detector for reflectance a CCD camera is first mounted in its place. This is used to aid alignment of the knife-edge. As the crystal is tilted a small amount of the transmitted beam will be further reflected at the second surface (see Fig. 3). It is possible to see both of these spots if the incident beam itself is around 100 μm and the CCD camera is used to check beam size. The reflected spots diverge the further away they are from the crystal and this will result in an interference pattern. The knife-edge is mounted on a motorised linear stage (resolution 1 μm) and set close to the crystal surface where the spots are still seen to be separate. The knife-edge is used to block the spot reflected off the second surface and the CCD is used to assess how far the knife-edge must be moved in order to completely block the back reflection. The motorised linear stage aids the alignment process. LabVIEW software that controls the CCD and stage is used to collect the data. The stage is zeroed when the knife-edge blocks the second beam. The knife-edge is a scalpel blade that has been sprayed with matt black paint to reduce scatter effects.



knife-edge out
(b)

knife-edge in
(c)

Fig. 3. (a): Schematic of knife-edge measurements, (b) image of reflectance on CCD camera showing interference between beams reflected from both surfaces of the crystal. As the knife edge moves in to block the spot reflected from the furthest surface the fringes gradually disappear until it is totally blocked, as the image shows is (c). (He-Ne light at 633 nm was used for (b) and (c) since the front and back surface reflectances were similar, leading to high visibility fringes).

Mounting the trap detectors

The trap detectors were of the NPL three-element design using Hamamatsu S1337-1010 photodiodes. The trap detector was positioned such that the beam was sitting in its plateau region. All trap detectors suffer a small amount of reflectance loss (~ 0.3% of incident beam [4]). This reflected beam can aid alignment but once the trap is aligned so that the beam is normal the trap must be tilted slightly away from normal so that the reflected spot does not fall back on the sample. The trap detector should not be rotated more than 5° from the normal position, but to be on the safe side, not more than 1°. This is so that the reflected beam does not hit the trap housing. The trap detector can suffer from small polarisation effects, depending on the trap assembly. However, so long as the orientation does not change between measurements this does not matter. A small score was made on the trap detector aluminium housing to register the orientation.

Correction factor between the two traps

Each trap detector and its trans-impedance amplifier were taken as a single unit. One unit was set to measure the reflected beam and the other the incident and transmitted beam. In order to determine the correction necessary between readings taken from these two traps, each unit was set up to measure the incident beam (I_o). Three sets of consecutive data for each trap were taken, i.e. trap A would be mounted, 5 measurements made, then trap B would be mounted, 5 measurements taken, then trap A mounted again and so forth. In both cases the data was corrected for background radiation.

All the data collected on the trap measuring reflectance were divided by this correction factor. The correction factor ('reflectance' trap response ÷ 'transmittance' trap response) is 1.0024 ± 0.0031. The uncertainty in this value incorporates the spatial non-uniformity of the trap detectors and the laser instability.

Trap uniformity

A trap detector should have a response non-uniformity of less than 0.02% for a 4 mm diameter beam over ±2 mm about the centre [4]. In these measurements both the reflected and transmitted beams had diameters of about 2 mm. The non-uniformity of each trap was measured using the reflected and transmitted beams respectively.

Laser stability

The stability of the laser was measured using the one of the traps. Over one hour, a periodic trend is clearly seen, with a period of 15 minutes. This coincides with the air conditioning cycle in that lab. The measurement could be improved by building an enclosure around the measurement area. Figure 4 shows that the standard deviation of the data points is 0.000026. The measurement sequence for the crystal takes about 20 minutes in total.

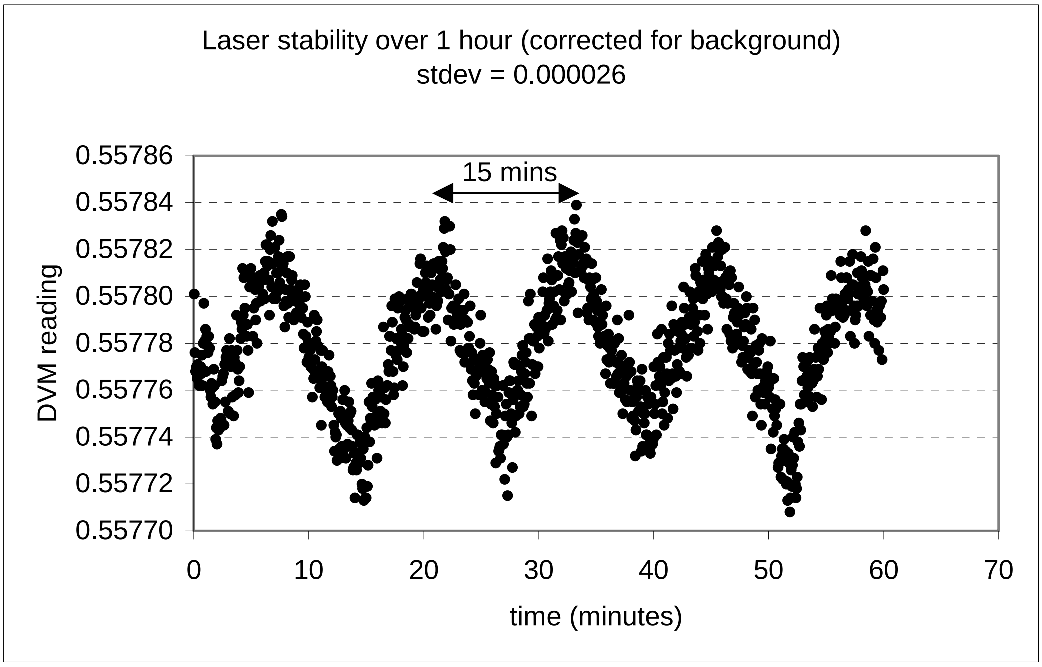


Fig. 4. Laser stability over 1 hour

Measurement procedure for crystal

Once aligned the following measurements were made:

Trap detector in the transmittance position:
I_0 = Straight through beam
I_{Tj} = Transmittance measurement with crystal in the beam, laser incident on side j.

Trap detector in the reflectance position:
I_{R2tot} = Reflectances off surfaces 2 and 1 of crystal (knife edge is out);
I_{R2} = Reflectance off surface 2 of crystal with knife edge blocking back reflection;
I_{R1tot} = Reflectances off surfaces 1 and 2 of crystal (knife edge is out);
I_{R1} = Reflectance off surface 1 of crystal with knife edge blocking back reflection;

Five measurements, each being the average of 50 readings on the DVM were taken on both traps. The knife-edge would then be moved in to block the second reflected spot and another 5 measurements taken. This would be repeated. The crystal was also rotated and realigned so that the reflectance from the surface 1 can be measured as well with the knife-edge in and out of the beam. All measurements were dark corrected.

Ideally, the measurements should agree with equation 4:

$$I_0 = I_{T\,j} + I_{R\,j\,tot} + A + S \qquad 4$$

where A is the absorption in the crystal and S accounts for any losses due to scatter at each crystal surface and within the crystal which propagates outside the capture region of the trap detectors.

Results

The following equations were used to calculate the reflectance and transmittance. I_R is the measured reflected signal, I_t is the measured transmitted signal and I_0 is the measured signal of the straight through beam. Table 1 summarises the results of the measurements.

$$Reflectance = \frac{I_R}{I_0.C_{correction\ factor}} \qquad 5$$

$$Transmittance = \frac{I_t}{I_0} \qquad 6$$

$C_{correction\ factor}$ = Ratio of measurements of I_0 of reflectance trap relative to transmittance trap 7

Table 1. Uncertainties

	mean value	stdev	d	final value	unc (k=1)
R trap signal	0.557855	0.000229	1.0	0.557855	0.001675
R trap uniformity	-0.039843	0.000205	1.7		
T trap signal	0.556540	0.000066	1.0	0.556540	0.000438
T trap uniformity	-0.514174	0.000693	1.7		
laser stability	0.557785	0.000022	1.7		
trap correction factor				1.002364	0.003112
I1_tot	0.925873	0.000073	1.0	0.925873	0.000724
I2_tot	0.926204	0.000071	1.0	0.926204	0.000724
R2	0.001403	0.000002	1.0	0.001403	0.000005
R2_tot	0.072438	0.000080	1.0	0.072438	0.000239
R1	0.071085	0.000082	1.0	0.071085	0.000235
R1_tot	0.072240	0.000082	1.0	0.072240	0.000239

The signal values and stand deviations are calculated from the series of repeat measurements. The final uncertainty for the trap signals are the combination of the signal, uniformity and laser stability standard deviations. These uncertainties then combine to give the uncertainty in the trap correction factor. The uncertainties for the transmittances I1_tot and I2_tot are the combination of the signal and trap uniformity standard deviations. Any effects due to laser drift are taken care of by the signal standard deviations. The uncertainties for the reflectances are the combination of the signal standard deviations and the trap correction factor. The two traps are triggered virtually simultaneously, so there are no effects due to laser drift. Any variation in moving the knife edge in and out are taken care of by the signal standard deviations. Subsequent to carrying out the measurements it was found that the ‘R’ trap was close to the edge of its plateau region, which explains the higher than expected non uniformity of this trap.

Other uncertainty components that were considered are summarised in Table 2.

Table 2. Other uncertainties that were also considered

Uncertainty	**Comments**
Beam position on trap	Trap detectors are insensitive to position as long as the signal beam sits on the response plateau and is not a strongly divergent beam
Angle of incidence into trap detectors	During the series of measurements the angle of incidence did not change as the traps were not moved. There may be a small difference between the angle at which the reflectance trap was compared to the transmittance trap and the angle at which it was used to measure reflectance. However, traps are insensitive to angle of incidence so long as the beam sits on the plateau.
Speckle	Laser speckle is only an issue if the beam profile overfills the detector
Detector linearity	Trap was set far away enough so that non-linearities due to tight focussing would not be an issue, the beam diameter was 2mm
Laser pointing drift	Accounted for in detector uniformities
DVM non-linearity	Accounted for in the ratio calculations, and only relevant in the reflectance calculations
Polarisation of incident beam	Trap detectors were not moved within their mounts and therefore always at the same orientation with respect to the beam

From Table 1, it can be seen that R2 << R2_tot, whereas R1 ≈ R1_tot. This is to be expected, given that surface 2 is AR coated at 702 nm, but surface 1 is not. The value of R2 cannot be taken as being due solely to the first surface reflectance, since the intensity of the back surface reflectance is ~ 50 times more intense, and therefore the wings of the back surface reflectance will significantly overlap with the front surface reflectance when the knife edge is in. However, for the application of this crystal we are particularly interested in the reflectance losses at surface 2 as this boundary sits between the point of downconversion and the detector.

If we consider the data for the laser incident on face 1,

$$R1 = r_1 \qquad (8)$$

$$R1_{tot} = r_1 + \sum_{2}^{\infty} R1_j = r_1 + \frac{t^2(1-r_1)^2 r_2}{1-t^2 r_1 r_2} \qquad (9)$$

where

$$R1_j = t^2(1-r_1)^2 r_2 \left(t^2 r_1 r_2\right)^{j-1} \qquad (10)$$

and r_1, r_2, and t are the reflectances at surfaces 1 and 2, and the single pass internal transmittance

Similarly,

$$T_{tot} = \frac{t(1-r_1)(1-r_2)}{1-t^2 r_1 r_2} \tag{11}$$

We make the following approximations:

(i) since r_1 ~ 0.07 and r_2 ~ 0.0014, we shall truncate the expression for R_{tot} after j=2, since the ratio $t^2 r_1 r_2$ ~ t^2 (0.0001);

(ii) taking r_2 ~ 0 leads to T_{tot} ~ t (1-r_1).

Hence

$$R1_{tot} = r_1 + (1-r_1)^2 t^2 r_2 = r_1 + T_{tot}{}^2 r_2 \tag{12}$$

i.e

$$r_2 = \frac{R1_{tot} - r_1}{T_{tot}{}^2} \tag{13}$$

Since we have confidence in the data obtained from surface 1, we can use (13) to estimate r_2. The difference term in the numerator is likely to lead to a significant increase in the relative uncertainty of the result.

As a consistency check, we can then use the estimated value for r2 to calculate $R2_{tot}$ using (14) which is derived in a similar manner to (12):

$$R2_{tot} = r_2 + (1-r_2)^2 t^2 r_1 = r_2 + (1-r_2)^2 \frac{T_{tot}{}^2}{(1-r_1)^2} r_1 \tag{14}$$

From equations (13) and (14) we find:

r_2 = 0.00135 ± 0.00014 (k=1)
R2_tot = 0.0718 ± 0.0026 (k=1)

where account has been taken of the correlations in the trap correction factor uncertainty.

The calculated value of R2_tot compares very well with the experimentally measured value of 0.07244 ± 0.00024 (k=1). The crystal suffers from a non-uniformity of 0.8% of the mean transmittance in its central 2 mm x 2 mm area, which is believed to be due to the AR coatings. While all measurements – reflectance and transmittance – on one face of the crystal were taken with the laser beam incident on the same spot (within the repeatability of the measurement sequence), all that can be said about the full set of measurements is that the laser was set incident as close to the centre of either side of the crystal as could be judged by eye, and therefore there is the likelihood that the measurements taken on opposite sides of the crystal sample slightly different sections of the crystal.

We observe that these measurements can in principle lead to a set of over-determined equations, and that therefore a fuller statistical analysis may lead to more consistent results.

Conclusions

Transmittance and reflectance measurements from small parallel-sided samples suffer from interference effects and, coupled with anti-symmetric AR coatings it may be necessary to measure the reflectance of each surface individually. The crystal studied in this paper is such a sample. Knowledge of the reflectance losses at each surface was required with a target uncertainty of better than 0.1%.

This paper demonstrates how it is possible to use a knife-edge to block the reflectance off the second surface of the crystal through monitoring the interference fringes on a CCD camera. Measurement considerations necessary for analysing the uncertainties have been discussed. This technique complements other techniques which have been developed to carry out spectrometric measurements on small samples [3, 5].

The sampled area of the crystal was of the order of 100 μm in diameter, and the overall uncertainty in calculated reflectance 0.0014 of surface 2 was 0.00014, while the uncertainty in the measured reflectance 0.00711 of surface 1 was 0.00024. These values represent an initial exploration of the technique, and further improvements may be feasible.

Acknowledgements

This work was funded by the UK DTI National Measurement Directorate's Programme on Optical Radiation. The authors would like to thank W S Hartree and J Wang for their helpful advice.

References

1. Cheung, J.Y., C.J. Chunnilall, and J. Wang. "Radiometric applications of correlated photon metrology". *Proceedings of SPIE*, **5551**, p. 220-230, 2004
2. Migdall, A.L., et al. "Absolute quantum efficiency measurements using correlated photons". *Metrologia*,. **32**, 479-483, 1995/96
3. Hartree, W.S., E. Theocharous, and N.P. Fox. "A wavelength tunable, quasi-cw laser source for high accuracy spectrometric measurement in the 200 nm to 500 nm region". *Proc. SPIE*,. **4826**, p. 104-112, 2003 (in press).
4. Fox, N.P. "Trap detectors and their properties". *Metrologia*, **28**, p. 197-202, 1991
5. Cheung, J.Y., et al. "High accuracy dual lens transmittance measurements". 2007. In preparation.

This work was supported by the National Measurement System Policy Unit of the Department of Trade and Industry.