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Quality Manual of Reference Spectrometer Laboratory

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1. Table of contents

2. Definition

2.1. Scope

This quality manual describes the principle and operation of the reference spectrometer used to realise the national scale of regular spectral transmittance at the Metrology Research Institute (MRI). The procedures for characterisation of the instrument and calibration of the filters are described. This equipment is also used for the realisation the relative scale of diffuse spectral reflectance, the unit of spectral responsivity, and the unit of regular spectral reflectance for which the procedures of calibrations are given in separate manuals.

Chapter 5.4 of this document also describes the traceability of the regular spectral transmittance measurements performed with a transfer standard spectrometer. The latter has been characterised using similar procedures and reference materials as for the reference spectrometer.

2.2. Object and field of application

Filters of general material: Regular spectral transmittance for wavelengths between 220 nm and 1700 nm. (The scope of CMC is for wavelengths between 240 nm and 1700 nm)

Useful information can be obtained about the properties of many transparent or translucent materials by measuring the regular spectral transmittance of optical radiation through the filter media. Most applications are in radiometric, photometric, and colour measurements where different types of filters are used for calibration purposes. The reference instrument is well characterised and the combined standard uncertainty can be as low as 0,0003 transmittance units (k=1) at the visible wavelengths of the spectrum.

2.3. Features

The reference spectrometer has been characterised for a measurement beam with a wavelength between 240 nm and 1700 nm. The configuration of the reference spectrometer for transmittance measurements includes the following features:

a) Automated instrument providing simultaneous measurement of several filters and scanning of the selected wavelength settings

b) Reflecting optics

c) Collimated single-beam design allowing the determination of various uncertainty components

d) Detector unit, including an averaging sphere and a silicon photodiode, which minimises the beam-displacement error caused by insertion of imperfect filters, the error due to interreflections between the filter and the detector, and the spatial responsivity errors of the detector.

2.4. Regular spectral transmittance

Transmission of radiation through a medium, without a change in the wavelength, is regular if the direction of the passing radiation does not change.

The regular spectral transmittance

[*] T of a given medium is the ratio of the transmitted spectral radiant flux $\Phi_{\lambda\lambda,\mathrm{T}}$ to the incident spectral radiant flux Φ_{λ}

$$T(\lambda,\theta_1) = \frac{\Phi_{\lambda,T}}{\Phi_{\lambda}}\,, \tag{1.1}$$

where λ is the wavelength of the radiation and θ_1 is the angle of incidence. The transmittance varies as the physical parameters of the filter and/or the properties of the incident light change. Variations in the incident light beam can be due to changes in polarisation, degree of collimation or convergence, and coherence of the radiation.

To examine the above-mentioned dependencies, let us assume, that the incident light is a perfectly monochromatic, plane-polarised, plane wave of wavelength λ. The filter is isotropic, homogenous, and forms a plane parallel slab of infinite extent and of thickness t. The initial and final dielectric media are the same and have an index of refraction of n_1. Let the complex index of refraction of the slab medium be $\hat{n}_2 = n_2(1+ik_2)$, where k_2 is the attenuation coefficient.

Based on the laws of reflection and refraction and by including the effect of multiple reflections between the slab surfaces, the regular spectral transmittance of the slab is given by

$$T(\lambda,\theta_1) = \left| \frac{(1-\hat{r}^2)e^{i\beta}}{1-\hat{r}^2 e^{2i\beta}} \right|^2 , \tag{1.2}$$

where $\hat{r}$ is the complex Fresnel coefficient of the amplitude reflectance at the first boundary, and β is defined as

$$\beta = \left(\frac{2\pi}{\lambda}\right) t\,\hat{n}_2 \cos\hat{\theta}_2 . \tag{1.3}$$

The complex angle $\hat{\theta}_2$ is given by Snell's law as

$$\sin\hat{\theta}_2 = \frac{n_1 \sin\theta_1}{\hat{n}_2}. \tag{1.4}$$

For s-polarisation the coefficient of the amplitude reflectance is,

[*] K.D. Mielenz, *Physical Parameters in High-Accuracy Spectrophotometry*, NBS Special publication 378, 81-93 (1973).

$$\hat{r} = \hat{r}_s(\theta_1) = \frac{n_1 \cos\theta_1 - \hat{n}_2 \cos\hat{\theta}_2}{n_1 \cos\theta_1 + \hat{n}_2 \cos\hat{\theta}_2} \tag{1.5}$$

and for p-polarisation,

$$\hat{r} = \hat{r}_p(\theta_1) = \frac{\hat{n}_2 \cos\theta_1 - n_1 \cos\hat{\theta}_2}{\hat{n}_2 \cos\theta_1 + n_1 \cos\hat{\theta}_2} \,. \tag{1.6}$$

The transmittance is a complicated function of the angle of incidence, the polarisation state of light, and the thickness of the filter. When the angle of incidence deviates from $\theta_1 = 0$ the internal transmittance will decrease due to the increased optical path length. The effect of polarisation is due to different reflectance values for different polarisation states of the light, as indicated in Eq. (1.5) and Eq. (1.6), and it causes further angular variations of the transmittance.

The dependence of the transmittance on the thickness *t* of the slab is seen from Eq. (1.3). For very thin filters the *t/λ* term gives rise to interference effects, which cause the transmittance to oscillate rapidly as the wavelength is varied. The effects of interference observed in practice are generally low for imperfect filters and for incoherent light. However, this effect is not negligible when discussing accuracy of a few parts in 10^4. For minimal effect of interference the filter should be relatively thick (>1mm) and the instrument bandwidth should be larger than 1 nm. *

Measuring the transmittance at normal incidence eliminates the effects of light polarisation. Therefore, if the effects of interference are negligible one can define an average transmittance τ as

$$\tau \equiv \overline{T(\lambda,0)} = \frac{(1-\rho)^2 \tau_i}{1-\rho^2 \tau_i^2} \,, \tag{1.7}$$

where

$$\rho = \left|\hat{r}_s(0)\right|^2 = \left|\hat{r}_p(0)\right|^2 = \left|\frac{n_1 - \hat{n}_2}{n_1 + \hat{n}_2}\right|^2 , \tag{1.8}$$

is the intensity reflectance of the filter surfaces for normal incidence for both states of polarisation, and

$$\tau_i = e^{-4\pi n_2 k_2 t/\lambda} \tag{1.9}$$

is the internal transmittance of the filter. This average transmittance $\tau = \overline{T(\lambda,0)}$ is a function of the filter parameters n_2, k_2, and *t* only and it is called the "true" regular spectral transmittance of a given filter.

3. Equipment

3.1. Description of setups

3.1.1. Equipment used in operation of the reference spectrometer

A. Light sources and input optics (see Figure 1.)

1) Quartz-tungsten-halogen (QTH) lamp (OSRAM HLX 64642 or HLX 64610) with lamp power supply Hewlett-Packard, HP-IB 6654A or Elekro-Atomatik GmbH, EA 7030-100, for operations in the wavelength range 350 – 1700 nm.

2) Xenon arc lamp, L2273 with power supply, C4246-01 and starter C4251 Hamamatsu, for operations in the wavelength range 250 – 500 nm.

3) Deuterium arc lamp, L5498-50, with power supply, C4545 Hamamatsu, for operation in the wavelength range 220 – 320 nm.

4) Spherical and cylindrical concave mirrors (Teknofokus, custom design)

5) Order-sorting filters are inserted manually for wavelength range 1501 –1700 nm, and automatically using a Filter-wheel for wavelength ranges 280–350 nm, 351 – 600 nm, 601 –870 nm, and 871 – 1500 nm. An open beam position of the filter-wheel is used for the wavelength settings shorter than 280 nm.

B. Monochromator and output optics

1) Grating monochromator (MC) and control unit (MRP-23, Russia)

2) Filter-wheel (Oriel) containing an opaque position and order-sorting filters for wavelength ranges 530.4 nm, 860.4 nm

2) Off-axis parabolic mirror (Oriel 45347)

3) Iris, limiting apertures, stands, and holders (built at MRI)

C. Data acquisition and control system

1) Interface cards to parallel printer connector of the computer (built at MRI)

2) Computer IBM/PC AT compatible

3) A computer program written in NI LabView v.5.1 (or higher) software development environment, the measurements are controlled by RefSpecV3 - version 2013.12.03, Custom codes based on RefSpecV3 are used for major customers needs.

3.1.2. Equipment used for calibration of wavelength scale

A. Radiation sources and input optics

1) Low-pressure Hg lamp (Oriel 6035),

2) Low-pressure Krypton lamp (Oriel 6031),

3) Power supply for low-pressure lamps (Oriel 6061),

4) 632,82-nm He-Ne laser (Thorlabs HRP005S, 0.5 mW),
5) Spherical and cylindrical concave mirrors, as in Sec. 3.1.1.A.4.

B. Monochromator and output optics

i) Same as in Sec. 3.1.1.B.

ii) McCrone wavelength standard (McCrone Scientific Ltd, UK) provides absorption lines at air wavelengths of 353.9 nm, 527.2 nm, 531.3 nm, 569.3 nm, 588.45 nm 684.5 nm, 743.9 nm, 748.2 nm, 795.4 nm, and their corresponding wavelengths for second-order diffraction of the spectrometer.

iii) Holmium oxide glass wavelength standard (Hellma GmbH & Co., Germany).

C. Data acquisition and control system

i) Same as in Sec. 3.1.1.C.

ii) Digital multimeter HP 3458A or HP 34401A or HP 34410A

3.1.3. Equipment used in transmittance measurements

The configuration of the reference spectrometer for transmittance measurements is shown in Figure 1 and the operation parameters are given in Table 1. The equipment include the followings:

1) Same as in reference spectrometer Sec. 3.1.1.
2) Linear translator stages, Physik-Instrumente, Newmark Systems Inc. and filter-holder unit built at MRI,
3) Iris, limiting apertures, stands, and holders (built at MRI)
4) Averaging sphere (built at MRI and coated at Labsphere, USA),
5) Detector with silicon-photodiode (Centronics OSD100-7CQ, 10 mm x 10 mm) including a preamplifier mounted on the averaging sphere, for wavelength range 240 – 1000 nm,
6) Detector with InGaAs-photodiode (10 mm x 10 mm Telcom devices 35D10M) including a preamplifier mounted on the averaging sphere, for wavelength range 850 – 1700 nm,
7) Power supply (15 V Signaali) for detectors listed above,
8) Trap detectors, MRI 9806 and MRI 9605 built at Aalto, with three 18 mm x 18 mm silicon photodiodes used with a calibrated external amplifier (Vinculum SP042 or Stanford Research SR570 or Keithley 6485) for calibrations of transmittance below 0.01 and requiring small beam size (<4 mm), for wavelengths range 240 – 440 nm,

C. Data acquisition and control system

1) Digital multimeter HP 3458A or HP 34401A or HP 34410A
2) Same as in Sec. 3.1.1.C.

Table 1. Operation parameters and features of the instrument in transmittance measurements.

	Wavelength range (nm)	Bandwidth (nm)	Beam diameter (mm)	Light source	Collimation	High-order rejection	Detection system
1.	UV 220-330	0.5-2.8	3-10	Deuterium lamp	<1°	—	Si-trap detector & amplifier
2.	UV/Visible 260-450	0.5-2.8	3-10	Xenon arc lamp	<1°	Filter	Si-trap detector & amplifier
3.	Visible 390-800	0.3-2.8	2-14	Quartz-halogen lamp	~ 0.5°	Filter	Averaging sphere and Si-photodiode
4.	NIR 800-1700	0.5-5.7	3-14	Quartz-halogen lamp	<1°	Filter	Averaging sphere and InGaAs-photodiode

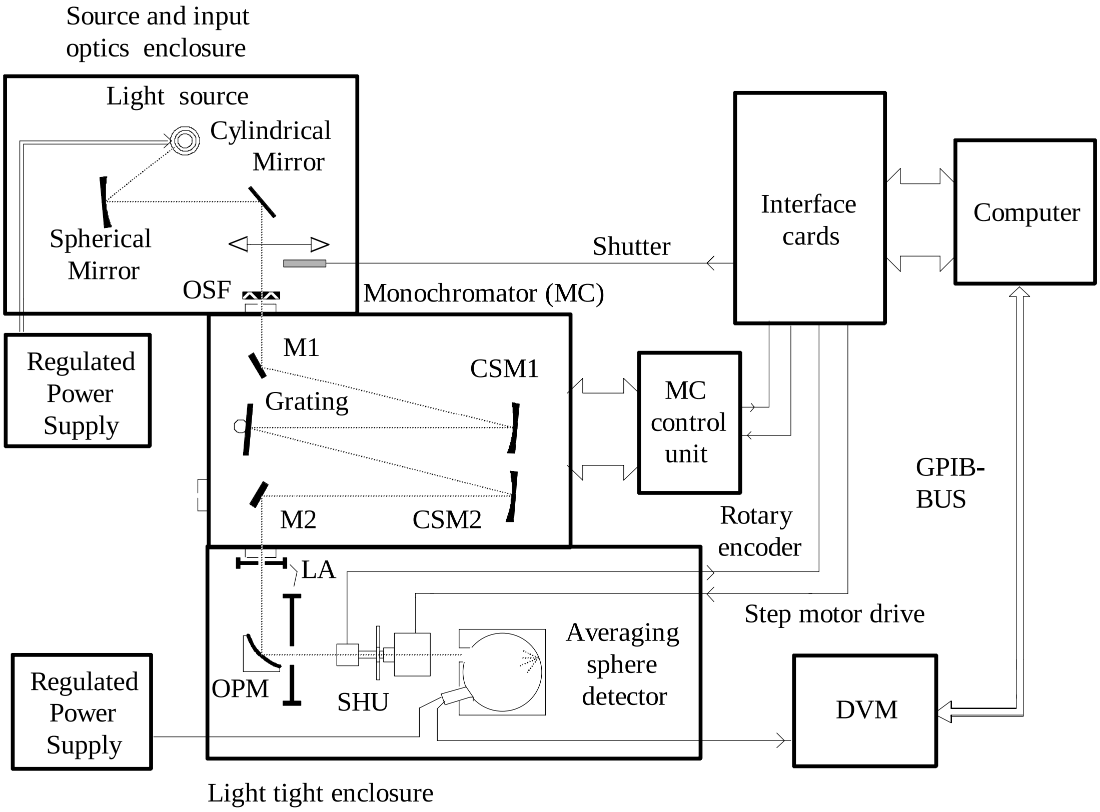

Figure 1. Schematic of spectrometer for transmittance measurements. OSF, order-sorting filter; M1, M2, flat mirrors; CSM1, CSM2, collimating spherical mirrors; LA, limiting apertures, OPM, off-axis parabolic mirror; SHU, filter-holder unit; DVM, digital volt meter.

3.2. Calibration requirements

3.2.1. Maintenance of the equipment

- The light source has a limited lifetime. The sum of time periods of the operation of the source is recorded in hours in a A4 paper posted on the input box of the Reference Spectrometer. The source is replaced with a new one when the sum is about 1000 hours for the halogen lamp, 2000 hours for the Xenon lamp, and 5000 hours for the Deuterium lamp.
- The filter-holder unit is checked biannually and calibrated if necessary
- The averaging sphere coating is visually checked biannually and throughput should be measured every 7 years and is renewed if necessary.
- The photodetector is tested for linearity in 5 years interval by using reference filters.
- Wavelength scale of the monochromator is checked after major modifications of the monochromator, e.g. replacement of gratings.
- The multimeters and amplifiers are calibrated according to Calibration Schedule of Aalto/MRI.

The information on the electronics can be found in publications [†] and [‡]. More information about handling the instrument and performing measurements are given in the Operating Instructions for Reference Spectrometer.

3.2.2. Responsible Persons

Branch manager and deputy are defined in the Quality Management System. Persons authorised to do calibrations are defined in Annex A.

[†] A. Äijälä, M.Sc. Thesis, *Harmaasuotimen Transmittanssin Mittauslaitteisto*, TKK, 1992.

[‡] F. Manoochehri, Licentiate Thesis, *High Precision Spectrometer for Transmittance Measurements*, Helsinki University of Technology, 1993.

4. Measurement traceability

Traceability of transmittance measurements

The accuracy of the regular spectral transmittance is not defined by a fundamental standard, but by the definition given in Sec. 2.4 and the calibration of instruments used during the measurements. The accuracy of the measurements has been verified by measuring a known reference material and by carrying out international intercomparisons between laboratories using different designs of reference spectrometers [§].

The wavelength scale is traceable to known transitions in gases (Hg, Kr, HeNe, HeCd). The absorption wavelengths of the McCrone and Holmium Oxide wavelength standards are not fundamental. They should not be used to establish traceability, but they are useful in checking the consistency of subsequent wavelength calibrations.

In order to characterise and calibrate various parameters in the operation of our reference spectrometer we investigated and controlled their effects on the measurements. During the calibration we utilised well-known methods and materials whose validity has been proven. In the following section the uncertainty components of transmittance measurements are estimated from the evaluation of the instrument parameters.

[§] F. Manoochehri, *High-Accuracy Spectrometer for Applications in Photometry and Radiometry*, Doctors thesis, Helsinki University of Technology, 115 p. (1998).

5. Calibration and measurement procedures including validation methods

5.1. Procedure for calibration of the wavelength scale

Wavelength calibration has been performed by means of emission lines from spectral lamps and laser radiation sources listed in Table 2.

The intensity data for each spectral line were recorded at small wavelength steps of about 0.003 nm with a narrow instrument band pass of less than 0.4 nm. The positions of the line centres were estimated by using a procedure based on the symmetry of the line shape. The wavelength calibration data were obtained in terms of the number of the step motor driving pulses required to bring the grating of the monochromator to the line centre position from a selected starting point. These numbers are least squares fitted to a quadratic polynomial whose argument is the wavelength. A typical linear coefficient given by the fit is -300.1 ± 0.5 driving pulses per nm, and the coefficient of the second-order term is 0.0002 ±0.0003 pulses/nm^2. The residual deviations between measured and fitted wavelengths are shown in Figure 2. The reproducibility of the wavelength calibration procedure is better than ±0.03 nm

Table 2. Wavelengths of atomic emission lines in air used for wavelength calibrations.

	Source	Wavelength (nm)		Source	Wavelength (nm)
1.	Hg lamp	253.65	9.	Helium-Cadmium laser	324.91
2.		296.73	10.		441.88
3.		404.66	11.	Helium-Neon lasers	543.35
4.		435.83	12.		632.82
5.	*	507.30			
6.		546.07			
7.	*	809.31			
8.	*	871.67			

* Lines measured at wavelengths corresponding to the second-order diffraction.

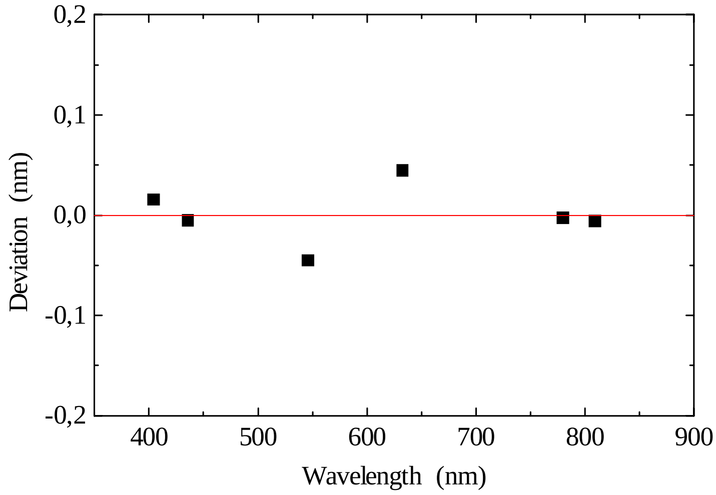

Figure 2. Residual deviations from the quadratic polynomial fit applied to the wavelength calibration data.

We operate the reference spectrometer with switching of gratings to cover the wavelength regions in ultra violet, visible, and near infrared. After such a switching, the wavelength scale is verified by scanning the spectral absorption peaks of known wavelength standard materials such as Holmium oxide doped glass filter and McCrown glass filter (See Sec. 3.1.2.B).

5.2. Transmittance measurement method and procedures

The spectrometer can measure regular spectral transmittances automatically. The spectrum of the lamp is scanned by the monochromator and the transmittance of one or several filters is measured at the desired wavelengths. The emerging beam from the monochromator during the scanning is directed into the averaging sphere through the filter-holder unit. The intensity of the beam is converted to a voltage signal, which is measured by a calibrated digital voltmeter (DVM). The DVM is read by a microcomputer via a GPIB-488 bus. Simultaneously, the computer controls the operations of the filter-holder unit, the shutter, and the monochromator via the corresponding interface and driver units. The slit width setting of 0.02 to 2.2 mm and beam-diameter selection of 2 to 15 mm are handled manually, but these settings are seldom changed. The measurement procedures are generally straightforward. Measurements involving unusual filter sizes, however, require special attention.

Following filter insertion and alignment, the measurements are performed according to the settings supplied to the computer control program. To eliminate the effects of the small drifts of the instrument, the transmittance measurements are performed in a time-symmetrical sequence. After the wavelength is selected, each set of measurements begins with measuring the detector signal for an empty filter holder in order to initialise the DVM for the full-scale reading. Depending on the number of filters to be measured, the following series of readings are taken:

one filter,

m times {forward pass: I_b, I_0, I_{S1}, I_{S1}, I_0; reverse pass: I_0, I_{S1}, I_{S1}, I_0, I_b},

two filters,

m times {forward pass: I_b, I_0, I_{S1}, I_{S1}, I_0, I_{S2}, I_{S2}, I_0;

reverse pass: I_0, I_{S2}, I_{S2}, I_0, I_{S1}, I_{S1}, I_0, I_b}, etc.,

where forward pass and reverse pass involve rotation of the filter wheel in the clockwise and the counter clockwise direction, respectively, when viewed in the beam direction. Symbols I_b and I_0 denote the signal readings for the detector dark current and for the beam intensity through an empty filter holder. Symbols I_{S1} and I_{S2} are signal readings for the transmitted beam through filter 1 and filter 2, respectively, and m is the number of passes chosen by the operator. Two readings are taken for each individual filter for the sake of preserving the time symmetry of the detector exposure resulting in the mean value $\bar{I}_{S1}$, $\bar{I}_{S2}$, etc. The signal-reading data are processed by taking the average value of closest I_b and I_0 readings on both side of I_{sj} readings, giving mean values $\bar{I}_b$ and $\bar{I}_0$. Then the transmittance T_j is computed for each filter according to

$$T_j = \frac{\bar{I}_{sj} - \bar{I}_b}{\bar{I}_0 - \bar{I}_b}$$

where j is 1, 2; etc. The corresponding filter transmittance values for the forward and reverse pass are then averaged over the 2m passes to obtain $\bar{T}_j$. The standard deviation corresponding to the T_j values is also calculated.

5.3. Automation software

The functions of the instrument are controlled by a LabVIEW software running on a PC. The latest implementation of the control software is “RefSpecV3”. The software is archived in the quality manual folder under “\spphotom\Software\RevSpecV3 - version YYYY.MM.DD”, such that versions are labelled with a date of latest modification. Current version is “RefSpecV3 - version 2013.12.03”. The program controls the operations of the monochromator, filter wheel and linear translators, and takes readings from the DVM. The program utilizes queued state-machine logic, such that all measurement automation is pre-programmed into a queue, which is then performed in sequence.

Previous version of the control software is “MAIN_Reference_spectrometer”, which can be still used, but it is not actively maintained. The latest version is archived in the folder “\spphotom\Software\MAIN_Reference_spectrometer Folder 7.2.2012”. This program has several pre-set measurement methods for commonly used types of measurements on the reference spectrometer.

5.4. Transfer standard spectrometer for regular transmittance

5.4.1. Instrument description **

The instrument (PE900) is a double monochromator, double beam spectrometer with a wavelength range of 185-3300 nm. The bandpass can be selected within 0.05 to 5.00 nm in UV/VIS range and within 0.2 to 20 nm in NIR range. A series of user manuals and tutorials are available in the laboratory and in electronic form manufacturer's Internet library. An Instruction Manual and the manufacturer's users guide of PE900 are archived in the quality manual folder under "\spphotom\.." PE900 spectrometer in our laboratory has been characterised for operation with the parameters given in Table 3.

Table 3. Operation parameters of the PE900 spectrometer in transmittance measurements.

	Wavelength range (nm)	Band-width (nm)	Beam size (mm x mm)	Light source	Colli-mation	High-order rejection	Detection system
1.	UV 220-330	0.5 - 5	2 x 4 – 5 x 12	Deuterium lamp	< 3°	—	Photomultiplier-tube detector
2.	Visible 330-870	0.3 - 5	1 x 3 – 5 x 12	Quartz-halogen lamp	~ 3°	Filter	Photomultiplier-tube detector
3.	NIR 870-1700	1 - 20	2 x 5 – 9 x 12	Quartz-halogen lamp	< 3°	Filter	TE-cooled PbS detector

5.4.2. Instrument calibration

Similar procedures were applied for characterisation of the PE900 spectrometer as for the Reference spectrometer. The level of stray light in the beam is negligible. The effects of beam displacement, non-uniformity, and interreflections are filter dependent and are taken into account during the uncertainty analysis. In the following subsections several other instrument parameters are explained.

5.4.2.1. Wavelength scale and bandpass

After a 30-minute warm-up period, running the software (Start-up check) tests the wavelength scale. Instructions are given in the user manual on how to proceed, if the test fails. The wavelength scale and bandpass are verified twice a year by scanning the spectral absorption peaks of known wavelength standard materials such as Holmium oxide doped glass filter and McCrown glass filter (See Sec. 3.1.2.B).

** Lambda 900 UV/VIS/VIS spectrometer, Perkin-Elmer Corporation, USA.

5.4.2.2.Linearity

The linearity of the detectors of PE900 has been checked with a set of five reference filters measured with the Reference spectrometer at 300 nm, 750 nm, and 1300 nm. The nominal transmittance of the filters is 0.05, 0.25, 0.5, 0.7, and 0.92.

5.4.2.3.Stability

If the temperature remains stable the wavelength scale is not altered, but for the base-line a drift of 0.1% has been observed in the NIR wavelength regions in less than one hour. Usually the measuring time is less than 15 minutes.

5.4.2.4.Beam polarisation

The degree of polarisation of the instrument beam was determined in the same manner as described in Sec. 7.1. For this purpose the transmittance of a Glan-Taylor polarising prism was measured over the spectral range from 240 to 1700 nm using the sample beam. The calculated degree of polarisation P, varied from 0.06 to 0.75.

6. Handling of calibration items

- Calibrated items are not to be cleaned without permission from customer.
- If samples are cleaned calibration results are given before and after the cleaning.
- Filters are cleaned according to [[††]] with permission from branch manager

[††] ORIEL, *Guide to Cleaning Research Optics*, (Tutorial text).

7. Uncertainty budgets

7.1. Uncertainty components of transmittance measurements

Table 4 summarises the uncertainty components for high-quality non-fluorescent, absorption neutral density filters.

Table 4. Uncertainty components (1σ) for regular spectral transmittance measurements of neutral density filters at visible wavelengths for transmittance levels of higher than 10%. The uncertainty is given in transmittance units.

	Component	Standard uncertainty / 10^{-4}
1.	Wavelength bias	1.0
2.	Detector linearity	0.8
3.	System drift and noise (m=9)	2.0
4.	Beam displacement	1.0
5.	Polarisation	0.1
6.	Stray light	0.5
7.	Interreflections	0.3
8.	Combined standard uncertainty (Root-sum-of-squares)	3.0

m : number of measurement passes (see Sec. 5.2)

Wavelength scale and band pass

The wavelength scale of the monochromator is checked periodically to ensure the best attainable wavelength accuracy. The uncertainty of the wavelength scale is $\Delta\lambda$=0.06 nm which causes a transmittance uncertainty of $\Delta T = \Delta\lambda\ (dT/d\lambda)$. This uncertainty depends on the filter and is estimated to be smaller than 1×10^{-4} transmittance units for neutral density filters whose change of transmittance per unit wavelength is about $dT/d\lambda = 10^{-3}$ nm^{-1}.

The nominal instrument band pass is calculated as the product of the reciprocal linear dispersion (1.3 nm/mm) and the slit width of the monochromator. In routine measurements a band pass of 1.5 nm is chosen. It has been checked that the band pass of the monochromator has no effect on the wavelength calibration

Detector linearity

The working principle of the present spectrometer is based on measuring ratios of light intensities at each selected wavelength. Any deviation from linearity will cause an error in the measured transmittance. The linearity of the detection system has been studied by applying a modified version of the "ac-dc" method[‡‡]. The method is based on determining the ac response of the detector at several dc operating points. The photodetector response function is defined as $U(I) = g \cdot I[1 - e(I)]$, where *U* is the photodetector output voltage, *I* is the incoming optical power, *g* is the constant for a linear response, and *e(I)* is the relative deviation from linearity. The linearity tests are performed by applying a sinusoidal optical signal at a fixed amplitude to the detector in combination with a dc optical signal at different power levels. The maximum level of the dc signal is chosen in such a way that the dc component of the detector output signal is approximately equal to 1V as in transmittance measurements. The ac-signal amplitude imposes the minimum dc signal. The optical signals are obtained from two light-emitting diodes of wavelength 840 nm. The ac component of the detector output signal due to the ac optical signal is 200 mV (rms). The result of the tests gives an upper limit of $|e(I)| \leq 3 \times 10^{-4}$ for the relative deviation from linearity in the lowest order approximation (see Figure 3). The effect of the non linearity of the detector is estimated by assuming a first-order deviation from linearity that corresponds to a maximum transmittance uncertainty of $\Delta T = 8 \times 10^{-5}$ calculated from $\Delta T = T(1-T) \cdot e(I)$.

Stability of source and detection system

For single-beam spectrometers the stability of the light source is important. Simultaneous measurements of the voltage across the QTH-lamp terminals and the detector output voltage proved that there were fluctuations in the source and that the detector signal closely followed these fluctuations. However, these fluctuations are of random nature and can be considered as noise. An integration time of one second was found to give a smooth detector output signal. Temperature variations may also cause fluctuations in the throughput due to thermal expansion of the mechanical and optical components. This effect is avoided by using a chimney-like structure in the input optics enclosure that guides the heat away from the instrument.

The total system drift was measured for several wavelength settings. The detector signal was monitored every minute for a period of 60 minutes with a 5-second integration time. The result of a typical measurement is shown in Figure 4 where the data are fitted to a linear function. The slope of -1.7×10^{-5} volts per minute is considered to be due to a systematic drift, and the residuals give a measure of the random noise component whose relative standard deviation is approximately 0.07%. The system drift introduces a measurement uncertainty of smaller than 3×10^{-5} due to the time-symmetrical procedure of the measurements (see Sec. 5.2). The standard deviation of the transmittance measurements is about 2×10^{-4} for 9 readings. This is in agreement with the effect of fluctuations in the beam intensity as shown in Figure 4.

‡‡ R.G. Frehlich, "Estimation of the non linearity of a photodetector", Appl. Opt. 31, 5926-5929 (1992).

Beam displacement

To determine that how far an incident beam in the averaging sphere can be displaced without a considerable change in the detector response the following test was performed. The sphere was mounted on a micro positioner, that moved in a plane normal to the direction of the incoming beam of light. A 543.5-nm He-Ne laser beam was directed through a 2-mm-diameter aperture in front of the entrance port. Then, for small displacement steps of the sphere, observations were made of the fractional departures of the detector signal from the value measured when the beam was centred onto the entrance port. The average deviation was 0.01% for a 0.2-mm beam-displacement, which is considered as the worst case in the transmittance measurements*. The uncertainty caused by the beam-displacement in the averaging sphere depends on the filter and is less than 1×10^{-4}.

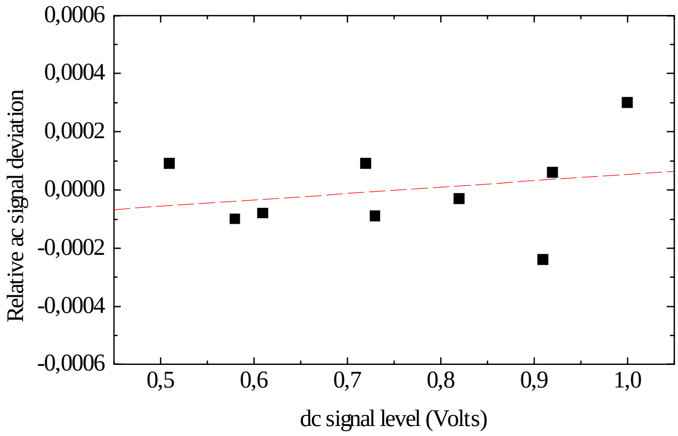

Figure 3. Relative deviation of the detection system from linearity obtained by observing the response of the detector to an ac signal at different dc signal levels.

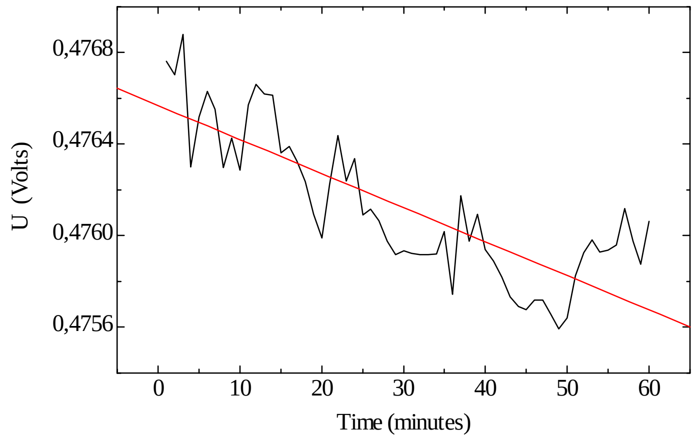

Figure 4. Drift of the detector output signal U with time. The total system drift is 0.003% per minute calculated from the linear fit (dashed line). The relative standard deviation of the measured values is 0.07%.

Instrument polarisation

High-quality neutral density filters should not exhibit detectable polarisation effects at normal incidence. For oblique angles of incidence, the transmittance will, however, depend on the plane of polarisation according to Fresnel's laws.

In the present instrument the angle of incidence of the beam on the filter is less than 1 degree due to the convergence of the beam. In order to estimate the transmittance error caused by the beam convergence and its polarisation effects, the degree of polarisation of the instrument output beam was determined. For this purpose the transmittance of a Glan-Taylor polarising prism was measured over the spectral range from 375 to 800 nm in two runs. First, the prism was placed in the filter-holder in such a way that its transmission axis was vertical. The measured transmittance is denoted as T_v. In the second run the prism was rotated by 90 degrees and the measured transmittance is denoted as T_h. The same procedure was applied for the wavelength range 240 – 380 nm. The degree of polarisation P, calculated as

$$P = \left| \frac{T_v - T_h}{T_v + T_h} \right|$$

varied from 0.06 to 0.26. Using the Fresnel formulae, calculations show that for a non absorbing glass filter with a refractive index of 1.5, the maximum transmittance error caused by the value P=0.3 and an angle of incidence of 1 degree is less than 1×10^{-5}. The effects of polarisation and obliquity of the beam or misalignment of the filter should not contribute by more than 1×10^{-5} to the total measurement uncertainty.

Stray light

Stray light effects in the measurements are caused by unwanted radiation present in the beam and the light scattered from various components in the measurement compartment. In order to estimate the level of radiation having a different wavelength from that of the beam, the transmittance of a number of Schott colour glass filters of 3-mm thickness was measured at 10 nm intervals. For the RG 665 filter with a sharp cut-off wavelength of 665 nm, the results are 10^{-4} and 10^{-5} transmittance units for wavelengths ranging from 380 nm to 450 nm and from 460 nm to 600 nm, respectively. The transmittance is less than 10^{-5} for a green filter (Oriel 59070) and for a black filter (Oriel 59562) for wavelengths longer than 650 nm.

In order to magnify the effect of the light scattered by the components (specially the limiting apertures) in the output-optics enclosure, transmittance measurements were performed with an aluminium-mirror as the filter. An average result of 7×10^{-6} units of transmittance was obtained throughout the operating spectral region. The uncertainty due to stray light also depends on the filter and can be estimated to be 5×10^{-5} when all stray light is transmitted by the filter.

Interreflections

One of the disadvantages of focused beam spectrometers is the relatively large optical error caused by inter reflections between the lenses and the filter. In the present instrument, use of an off-axis mirror eliminates such an error to a large extent by using a collimated light beam with normal incidence on the filter.

For the case of interreflections between the monochromator exit slit and the filter, the error introduced mostly depends on the properties of the filter. This error is reduced by the structure of the exit slit in such a way that it reflects the back-reflected light from the filter and the parabolic mirror to another direction and away from the mirror. Part of this inter reflection component can be considered as stray light error.

The inter reflections between the interior of the averaging sphere and the filter are also negligible due to the diffuse nature of reflections inside the sphere and the small dimensions of the entrance port dimension relative to the total area of the sphere. To give an estimate of the latter effect, suppose a filter with a 10% specular reflectivity is placed in the beam at a distance of 200 mm from the sphere. The maximum fraction of the transmitted beam reflected back from the sphere to the filter and finally back to the sphere through the 22 mm entrance port is less than 3×10^{-5}.

Beam uniformity

The uniformity of the beam at the position of the filter was investigated by recording the detector signal corresponding to the intensity of individual small spots in the beam. For this purpose a 3-mm circular aperture was mounted in front of the entrance port of the averaging sphere which was located in the position of the filter-holder unit. Moving the sphere in the horizontal and vertical directions in steps of 2 mm scanned the beam. With a rectangular shape (18 mm × 20 mm), the uniformity of the beam was better in the vertical than in the horizontal direction. The maximum variation was 8% along the 18-mm vertical dimension of the beam. In the horizontal direction the maximum variation was about 20%. Usually, the beam is made circular in shape by passing it through an aperture with a diameter of 15 mm. The intensity roll-off at the edge of the beam is about 10% of the maximum. The beam uniformity was found to have similar profiles at different wavelengths (450 nm; 550 nm; 650 nm) and different slit widths (0.7 mm; 1.2 mm; 2 mm).

The effect of the beam nonuniformity is most significant when the filter inhomogeneity is larger than 1%. The uncertainty introduced by this effect should be determined separately for each filter with different profile of inhomogeneity.

7.1.1. Measurement of the reference material

It has been possible to verify the measurement accuracy of our instrument by measuring a sample of UV grade synthetic fused silica whose refractive index is known[§§] with an uncertainty of 1×10^{-5}. The refractive index is homogeneous and stable with respect to time and temperature. The measured sample of this reference material has a thickness of 3 mm, a diameter of 25 mm, flatness of $\lambda/10$ on both sides, and parallel surfaces to within 10 arc-sec. The results of a measurement using a 1.5 nm band pass over the spectral range from 400 nm to 700 nm are shown in Figure 5. The measured transmittance values are in agreement with the calculated values to within 5 parts in 10^4.

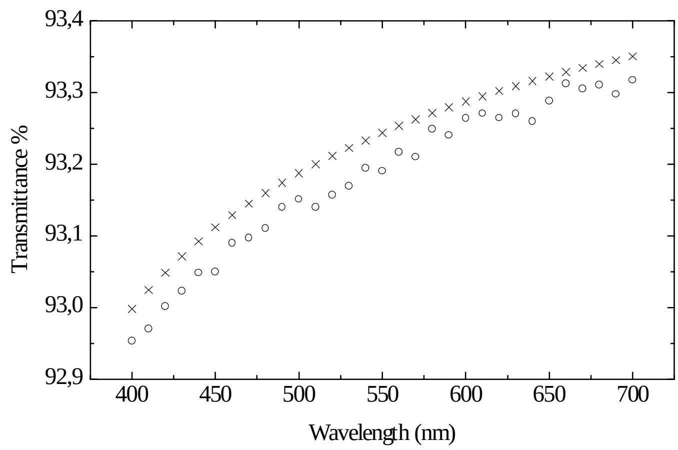

Figure 5. Measured spectral transmittance of the reference material (◦) and the corresponding calculated values (×) determined from the refractive index.

[§§] Dynasil corporation of America, NJ, Catalog 702-B and I.H. Malitson, "Interspecimen comparison of the refractive index of Fused Silica", Journal of the Optical Society 55, 1205-1209 (1965).

7.2. Measurement ranges and best measurement capabilities

7.2.1. Reference spectrometer

The best measurement capability of the reference spectrometer for regular spectral transmittance has been given in Publication [3]. Table 5 summarises the measurement range 0f 0.001 – 0.999 and the corresponding uncertainty budgets applied for customer calibrations. In addition, filter induced uncertainties such as material homogeneity are added as sum of squares to the measurement standard uncertainty. The latter is usually the major component for uncertainty in measurement of transmittance lower than 0.001. The expanded uncertainty in measurement of 0.0001 – 0.001 transmittance, ranges from 0.5% to 2%.

7.2.2. Transfer standard spectrometer (PE900)

Table 6 summarises the measurement ranges and the corresponding uncertainty budgets applied for customer calibrations. In addition, filter induced uncertainty such as material homogeneity and high reflectance is added as sum of squares to the measurement standard uncertainty. The expanded uncertainty in measurement of 0.0001 – 0.001 transmittance, ranges from 0.5% to 5%.

Table 5. Uncertainty components in regular transmittance measurements for neutral-density and for interference filters with a nominal transmittance of 0.001 - 0.999 at UV, visible, and NIR wavelengths. The numbers are absolute values and valid in the wavelengths. The numbers are absolute values and valid in the wavelength range of 240 - 1700 nm.

	Bandwidth: 0.5 nm – 2.0 nm	Standard uncertainty in transmittance / %					
	Component	Neutral-density filters			10-nm interference filters		
		UV	Visible	NIR	UV	Visible	NIR
1.	Wavelength scale	0.020	0.004	0.002	0.100	0.050	0.100
2.	Detector linearity	0.026	0.016	0.020	0.026	0.016	0.020
3.	Stray light	0.010	0.004	0.008	0.030	0.004	0.010
4.	System drift and noise	0.050	0.020	0.030	0.050	0.020	0.030
5.	Beam displacement	0.036	0.020	0.020	0.036	0.020	0.020
6.	Interreflections	0.016	0.034	0.034	0.016	0.034	0.034
7.	Temperature	0.010	0.004	0.004	0.060	0.030	0.030
8.	Square root of sum of squares	0.07	0.05	0.05	0.14	0.08	0.12

9.	Expanded uncertainty (k=2)	0.15	0.10	0.11	0.28	0.15	0.24

Table 6. Uncertainty components in regular transmittance measurements for absorption neutral-density and for interference filters with a nominal transmittance of 0.1 - 1.0 at UV, visible, and NIR wavelengths. The numbers are absolute values and valid in the wave-lengths. The numbers are absolute values and valid in the wavelength range of 220 - 1700 nm.

	Bandwidth: 0.5 nm – 2.0 nm	Standard uncertainty in transmittance / %					
	Component	Neutral-density filters			20-nm interference filters		
		UV	Visible	NIR	UV	Visible	NIR
1.	Transmittance of ref-erence filters	0.07	0.05	0.05	0.14	0.08	0.12
2.	Detector linearity	0.04	0.04	0.09	0.04	0.04	0.09
3.	System drift and noise	0.05	0.02	0.06	0.05	0.02	0.06
4.	Beam polarisation*	0.03	0.07	0.03	0.03	0.07	0.03
5.	Interreflections	0.04	0.02	0.02	0.09	0.08	0.07
6.	Square root of sum of squares	0.11	0.10	0.12	0.18	0.14	0.18
7.	Expanded uncertainty (k=2)	0.21	0.20	0.25	0.36	0.28	0.36

*The filters are measured in two perpendicular orientations to average the effect of beam geometry and polarisation where necessary.

7.2.3. Absorbance measurements and uncertainty determination

The regular spectral transmittance T and absorbance A of a neutral density filter meas-ured are measured by the PE900 Transfer Standard Spectrometer. The absorbance val-ues A are related to regular transmittance T as

$$A = -\log_{10} T.$$

UV WinLab v6.0 software of PE900 is used to collect measured data and to find wave-lengths of absorbance peaks for the case of rare-earth doped-glass 'wavelength stand-ard' filters.

Table 7 summarises the corresponding uncertainty budgets of Absorbance measure-ments applied for customer calibrations.

The uncertainty components for absorbance value are determined from those of spectral transmittance as u_A=0.4343 × $\Delta T/T$, where $\Delta T/T$ is the relative uncertainty of transmittance measurements .

If $A = -\log_{10} T$, then $T=10^{-A}$ or $T=(0.1)^A$

To observe the change of transmittance as a function of absorbance, we have $\Delta T/\Delta A=(0.1)^A \ln(0.1) = T \ln(0.1)$.

After rearranging then,

$\Delta T/T = \ln(0.1)\,\Delta A$

$\Delta A = 1/\ln(0.1)\,\Delta T/T = 0.4343 \times \Delta T/T.$

Table 7. Uncertainty components in Absorbance measurements for absorption neutral-density and for interference filters with a nominal absorbance of 0.02 - 1.0 at UV, visible, and NIR wavelengths. The numbers are absolute values and valid in the wavelength range of 220 - 1700 nm.

	Bandwidth: 0.5 nm – 2.0 nm	Standard uncertainty in Absorbance					
	Component	Neutral-density filters			20-nm interference filters		
		UV	Visible	NIR	UV	Visible	NIR
8.	Transmittance of reference filters	0.0003	0.0002	0.0002	0.0006	0.0003	0.0005
9.	Detector linearity	0.0002	0.0002	0.0004	0.0002	0.0002	0.0004
10.	System drift and noise	0.0002	0.0001	0.0003	0.0002	0.0001	0.0003
11.	Beam polarisation*	0.0001	0.0003	0.0001	0.0001	0.0003	0.0001
12.	Interreflections	0.0002	0.0001	0.0001	0.0004	0.0003	0.0003
13.	Square root of sum of squares	0.0005	0.0004	0.0005	0.0008	0.0006	0.0008
14.	Expanded uncertainty (k=2)	0.0009	0.0009	0.0011	0.0016	0.0012	0.0016

*The filters are measured in two perpendicular orientations to average the effect of beam geometry and polarisation where necessary.

8. Accommodation and environmental conditions

Measurements are usually done in the Reference spectrometer laboratory that is located in room 1560 in the 1st floor of the department of School of Electrical and communications engineering building (Maarintie 8). In Figure 6 the locations of the equipment of the spectrometer in the laboratory are shown. This laboratory is a clean room. Instructions for using such rooms have been given in ***.

During the calibrations:

- The Clean Zone -aggregate should be on, to prevent dust.
- Temperature should be monitored.
- Humidity should be monitored.

Humidity and temperature values during the calibrations are written to calibration certificates.

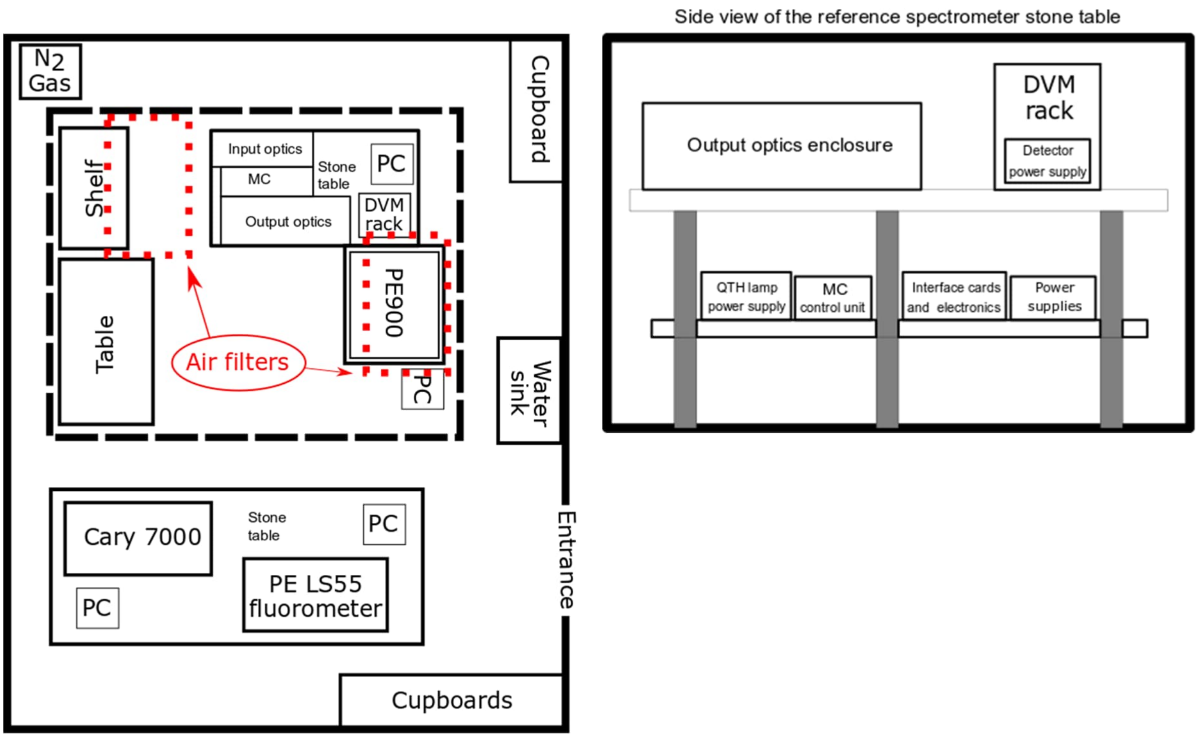

Figure 6. Approximate location of the equipment in the Reference Spectrometer Laboratory (not in scale).

*** *Clean room instructions / Puhdastilaohjeet*, MRI publication.

9. Field calibrations

Not applicable.

10. Control data

The measurement data coming from calibrations or development of equipment are archived in the computers of the reference and transfer standard spectrometer. The measurement notes (date, set up, raw data) are written down and the analysed measurement data are stored in chronological order. The related computer data files are also stored in the computers of the responsible persons under the directory called “Calibrations”. The records are arranged in the following manner:

- Regular measurement records are kept in folders under the title of the spectral region (UV, VIS, NIR) in which the measurements are done.
- International comparison records are kept in their respective folders.
- Calibration records are kept in folders under the title of ‘’Transmittance calibrations and certificates’’.
- Maintenance records of the equipment are written in a chronological order to a notebook labelled “Calibrations and measurements with reference spectrometer” and it is kept in the Reference spectrometer laboratory.

11. Certificates

Calibration certificates are handled according to publication in Annex C of quality system[†††].

In brief, each calibration has a certificate with a unique running number as in the following pattern; T-R 1, T-R 2, T-R 3, etc. It includes the method of calibration, traceability, uncertainty, measurements, and results. The calibration certificates are stored at the archive of the Metrology Research Institute.

[†††] *Instructions on writing calibration certificates*, MRI document.

12. Intercomparisons

CCPR-K6 International comparison of regular spectral transmittance

The key comparison was on spectral regular transmittance measurements in the wavelength region from 380 nm to 1000 nm. The measurements for this comparison were carried out by HUT in 2000. The report is available from the web site of the BIPM with the link as:

http://kcdb.bipm.org/AppendixB/KCDB_ApB_info.asp?cmp_idy=492

or from the Reference: [Metrologia, 2009, 46, Tech. Suppl., 02002, CCPR-K6 Technical Protocol]

Other comparison measurements performed with the Reference spectrometer are reported in publications [‡‡‡, §§§].

[‡‡‡] F. Manoochehri, E. Ikonen, and L. Liedquist, Comparison measurements on regular spectral transmittance, *Color Research and Application* 21 (1996) 440-447.

[§§§] J. F. Verrill, "Intercomparison of spectrophotometric measurements of regular transmittance" National Physical Laboratory report COEM XXX, UK (1998).

13. Publications

1. A. Äijälä, M.Sc. Thesis, "Harmaasuotimen Transmittanssin Mittauslaitteisto", TKK, 1992.
2. F. Manoochehri, Licentiate Thesis, "High Precision Spectrometer for Transmittance Measurements", Helsinki University of Technology, 1993.
3. F. Manoochehri, “High-Accuracy Spectrometer for Applications in Photometry and Radiometry”, Doctors thesis, Helsinki University of Technology, 115 p. (1998).
4. ORIEL, *Guide to Cleaning Research Optics*, (Tutorial text).
5. F. Manoochehri, E. Ikonen, and L. Liedquist, Comparison measurements on regular spectral transmittance, *Color Research and Application* 21 (1996) 440-447.
6. J. F. Verrill, “Intercomparison of spectrophotometric measurements of regular transmittance” National Physical Laboratory report COEM XXX, UK (1998)
7. F. Manoochehri and E. Ikonen, "High-accuracy spectrometer for measurement of regular spectral transmittance", Appl. Opt. 34, 3686-3692(1995).
8. P. Kärhä *et al.*, ''Optical power and transmittance measurements and their use in detector-based realization of the luminous intensity scale'', Opt. Eng. 34, 2611-2618(1995).
9. F. Manoocheri, Operating Instructions for Reference Spectrometer, ''a short note on steps to be taken for spectral transmittance measurements'', Quality Manual of Metrology Research Institute, (1994).