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Instruction Manual on Using Perkin-Elmer Lambda 900 spectrometer for Regular Spectral Transmittance and Absorbance Measurements

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2. Definition

2.1. Scope

This instruction manual describes principles and operation of the Perkin-Elmer Lambda 900 (PE900) transfer standard spectrometer setup at the Metrology Research Institute (MRI) that is used to perform transmittance and absorbance measurements. Procedures for the characterization of the instrument and calibration of transmittance standards are also given.

2.2. Object and field of application

PE900 spectrometer: setup for measuring transmittance and absorbance.

Regular spectral transmittance: The regular spectral transmittance T of a given medium is the ratio of the transmitted spectral radiant flux $\Phi_{\lambda\lambda,T}$ to the incident spectral radiant flux Φ_{λ}:

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

Regular spectral absorbance: The absorbance values A are related to regular transmittance T as

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

2.3. Features

Transfer standard spectrometer setup consists of Perkin-Elmer Lambda 900 device thoroughly described in ref. [1]. Setup is located in spectrometer laboratory of the MRI. The instrument is designed to perform regular transmittance and absorbance spectrophotometric measurements by comparing intensity of a beam transmitted through the medium to a reference beam transmitted through the free space (air).

2.4. Principles and definitions

Regular transmittance and absorbance are derived from monochromatic light fluxes transmitted through the medium under investigation and free space (air). Fluxes are measured by photosensitive detectors corresponding to wavelength of monochromatic light and compared to each other according to principles and definitions described in ref. [2]

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[1] Perkin Elmer Lambda 900 Users Guide

[2] Quality Manual of Reference Spectrometer Laboratory

3. Equipment

3.1. Instrument description

The instrument (Perkin-Elmer Lambda 900) 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. The setup and its maintenance are thoroughly described in ref. [1].

3.2. Measurement ranges and best measurement capabilities

PE900 spectrometer in our laboratory has been characterised for operation with the parameters given in Error! Reference source not found..

Table 1. 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°	—	Photomulti-plier-tube de-tector
2.	Visible 330-870	0.3 - 5	1 x 3 – 5 x 12	Quartz-halogen lamp	~ 3°	Filter	Photomulti-plier-tube de-tector
3.	NIR 870-1700	1 - 20	2 x 5 – 9 x 12	Quartz-halogen lamp	< 3°	Filter	TE-cooled PbS detector

4. Traceability Chain

4.1. Photometric scale

PE900 photometric calibration is traceable to reference spectrometer via several neutral density filters calibrated every 2 years according to the "Calsched" as described in ref. [2].

4.2. 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 ref. [2] Sec. Error! Reference source not found..B).

4.3. Linearity of the detectors

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.

4.4. 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.

4.5. Beam polarization

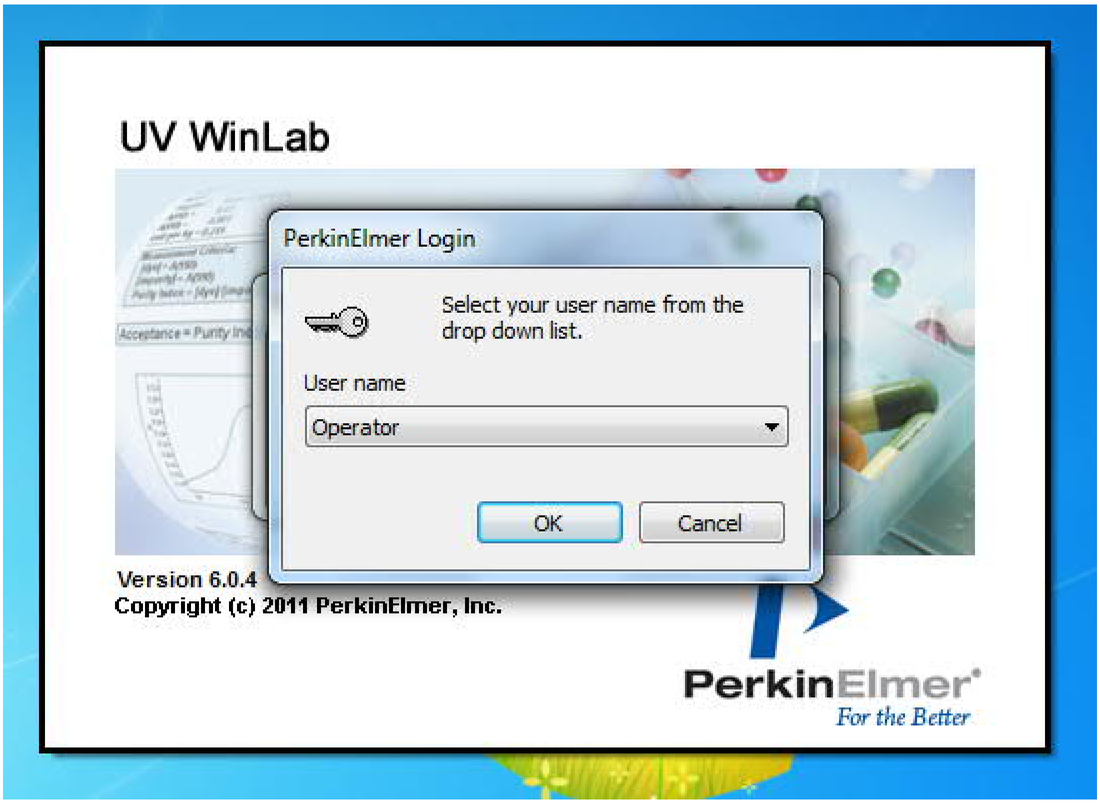
The degree of polarisation of the instrument beam was determined in the same manner as described in ref. [2] Sec. Error! Reference source not found.. For this purpose the transmittance of a Glan-Taylor polarising prism was measured over the spectral range from 240 to 1700 nm using the sample beam. The calculated degree of polarisation P, varied from 0.06 to 0.75.

5. Calibration methods and procedures

5.1. Measurement procedure

1. Turn on the device and start the program from the desktop

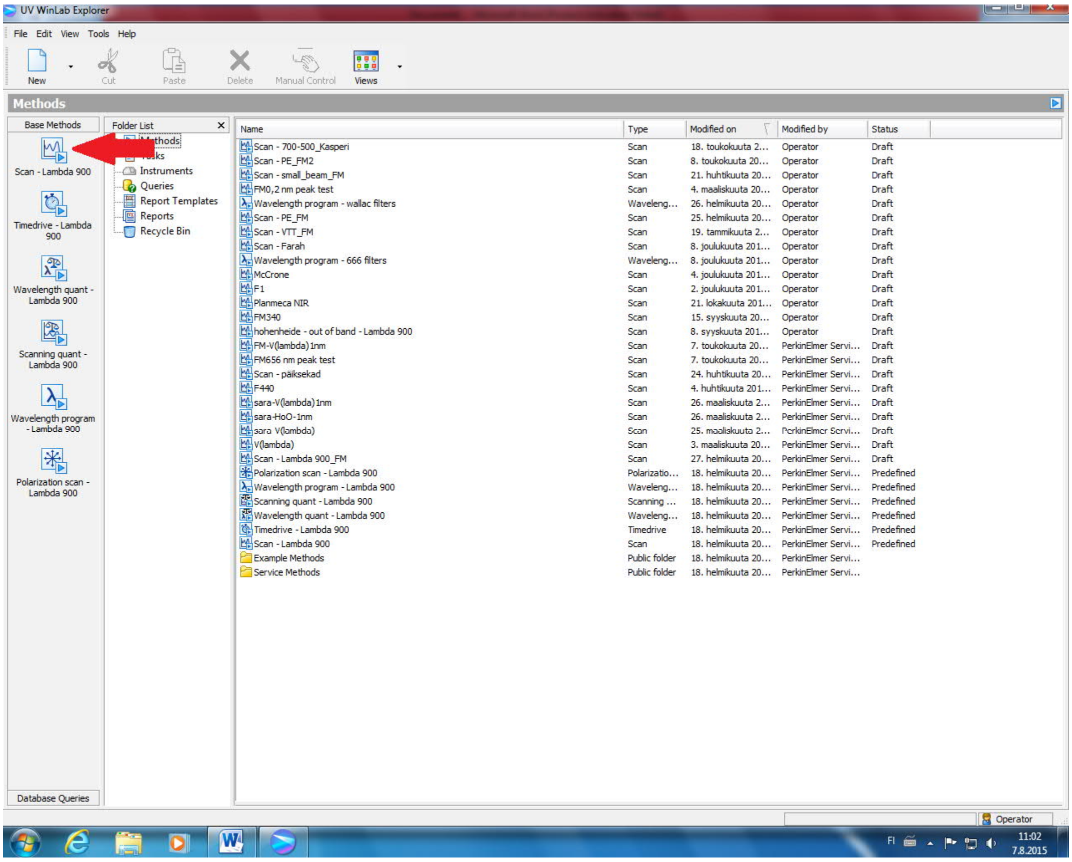
2. Choose your user name. 'Operator' is typically used



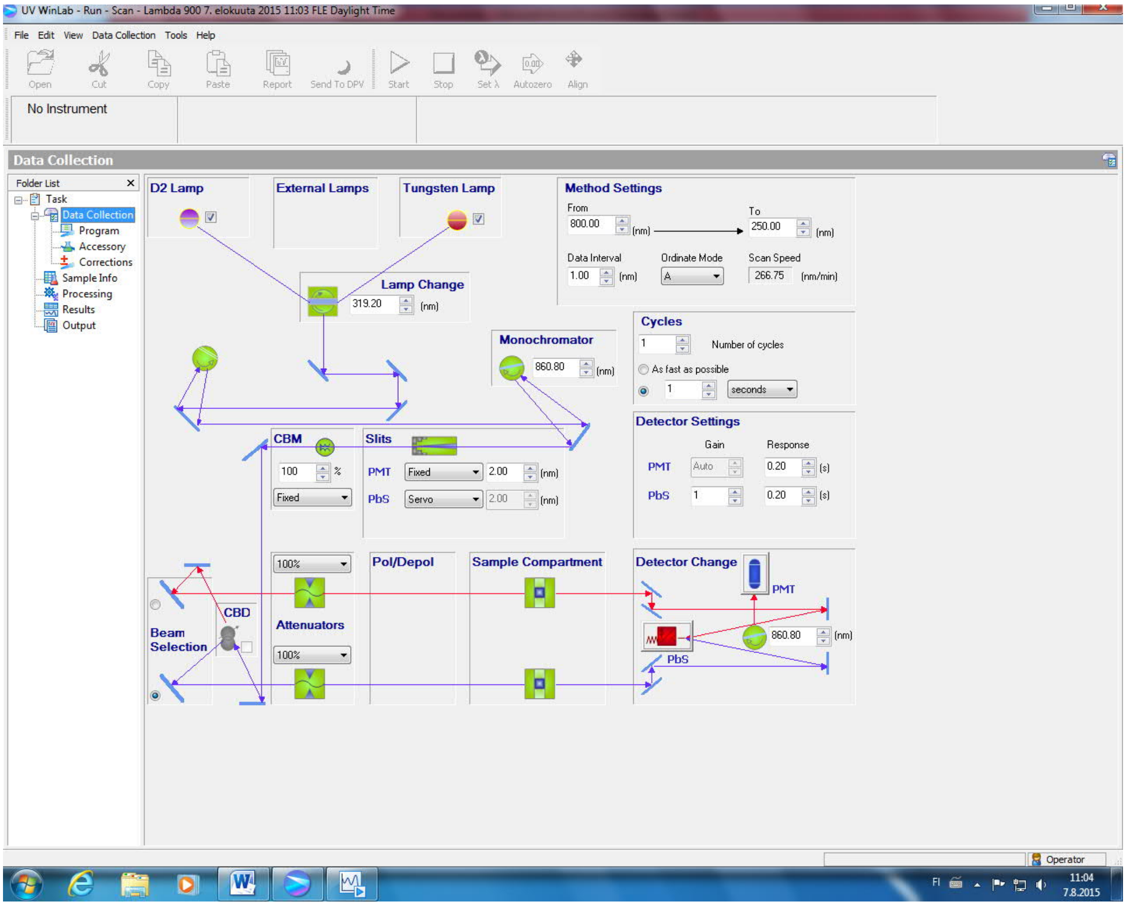
3. Turn on the PE900, the spectrometer will take some time before it is ready for use.

4. Choose either existing method, which are listed in the middle, or click “Scan – Lambda 900”.

   Scan method scans a range of wavelengths with chosen interval. Other methods are also available. For example, wavelength program method is used to measure discreet wavelengths chosen by the user.



5. General settings

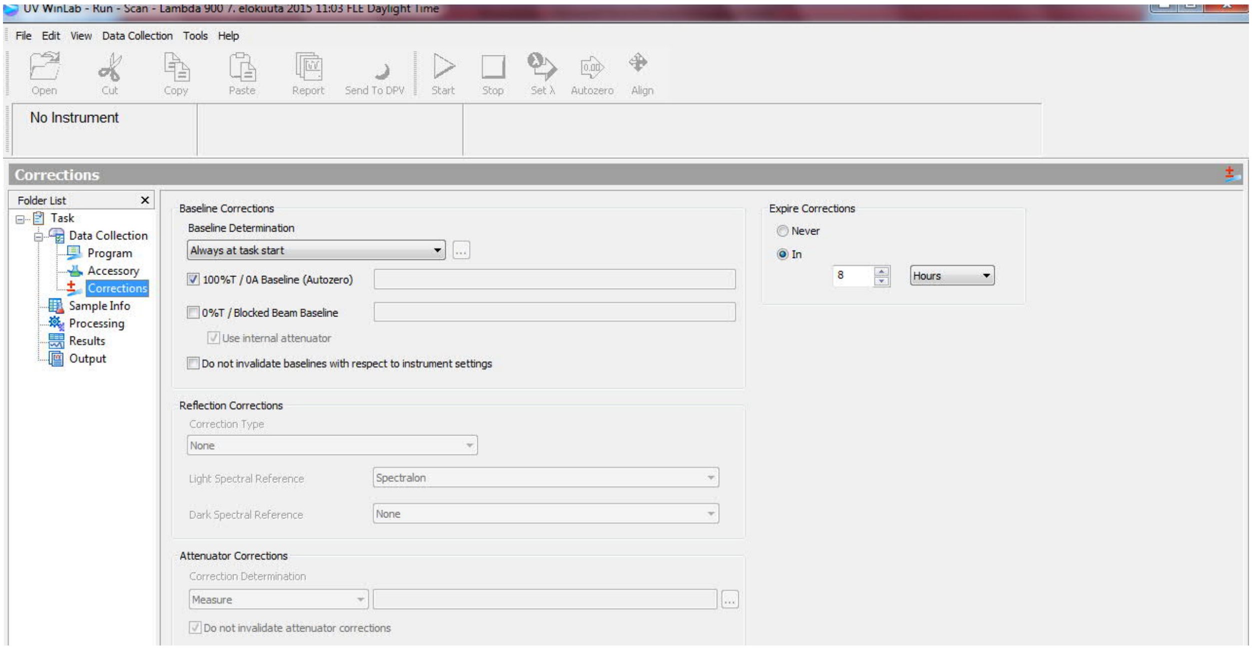


After the task is chosen, new window opens. Select Data Collection from the menu on the left side of the window to access general settings.

a. Method settings (Left upper coner)
   i. Set your wavelength range, start form the longer wavelenght (e.g. from 600 to 500)
   ii. Choose data interval (1 second is often suffcient)
   iii. Set the ordinate mode to %T
b. Detector settings
   i. Choose desired integration time by changing ‘Response’ setting
c. Slits
   i. Set the width of the slit
d. Beam selection
   i. Choose which beam passes through your sample (the rear beam is often prefered)

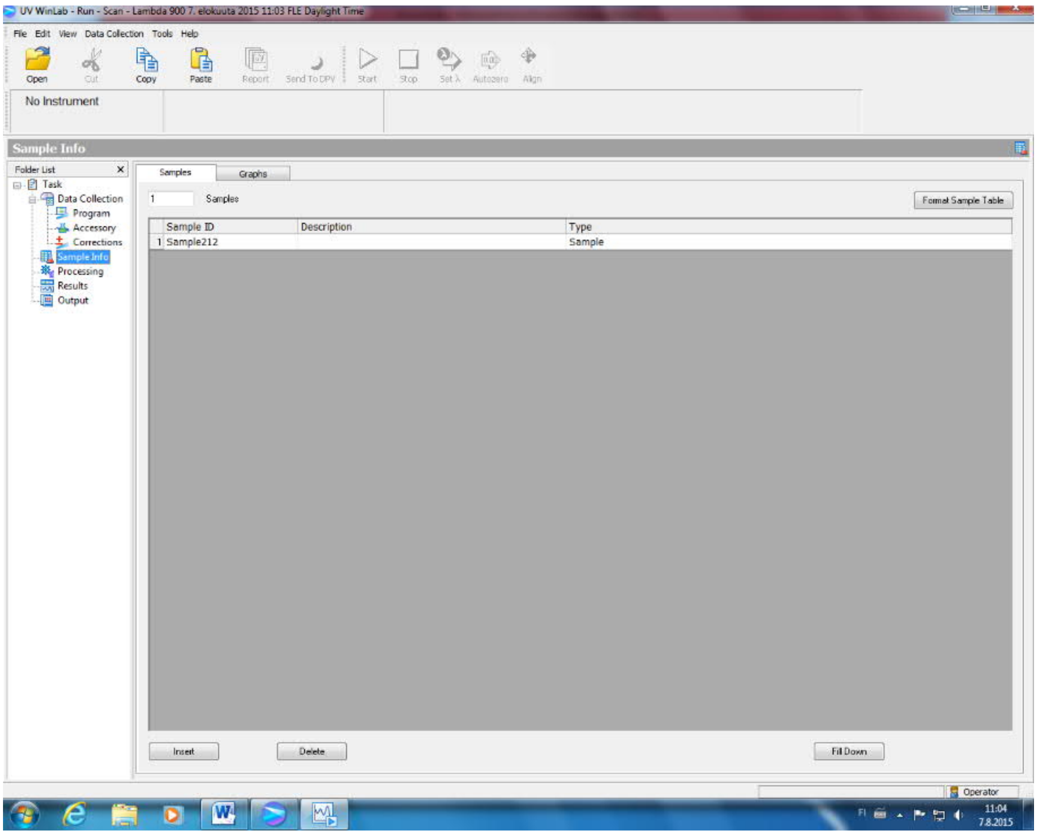
Other settings can also be modfied according to the requirements of your measurement.

6. Corrections tab

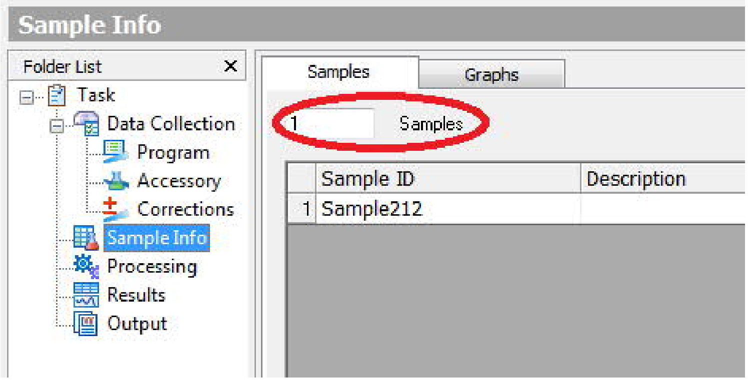


a. Tick ‘100%T / 0A Baseline (Autozero)’
b. Tick ‘0%T /Blocked Beam Baseline’

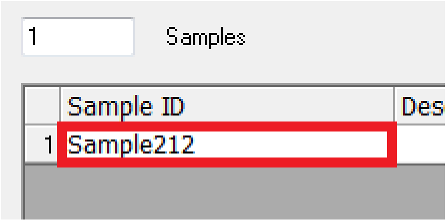
7. Samples tab



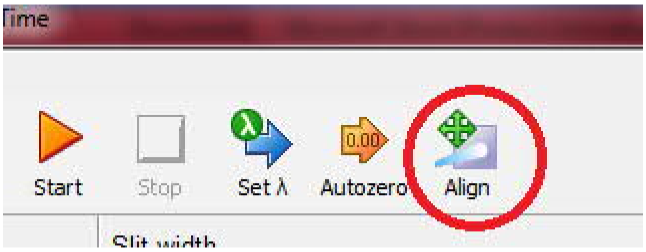
a. Set the number of samples you want to measure.



b. You can rename your samples by clicking the cell which contains the current name (here Sample212) and typing the desired name.

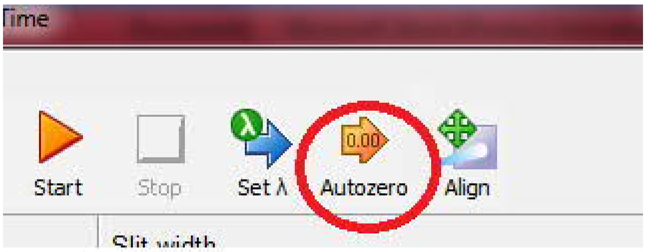


8. Aligning

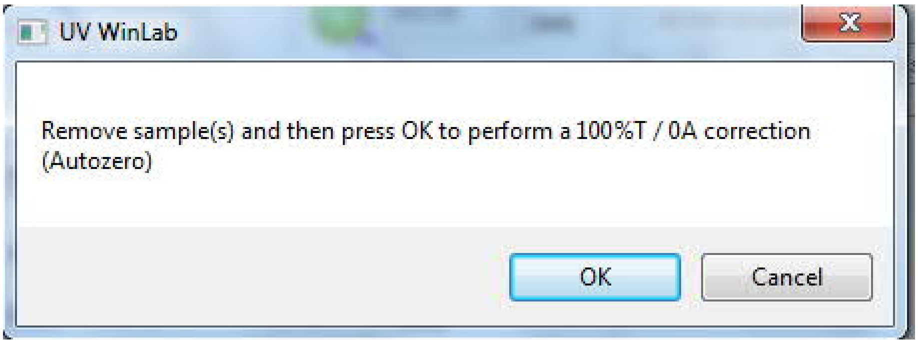


a. You can make sure that your sample is placed properly by using 'Align'.
b. NOTICE! When you press 'Align' and the instrument produces white light. It is recommended to cover the hole through which the beam is aimed to protect the detector from saturation. The hole can be easily covered with a sheet of paper.
c. When you are done with your aligning, press 'Align' button again and wait until the white light is gone. Then, remove the cover you used.

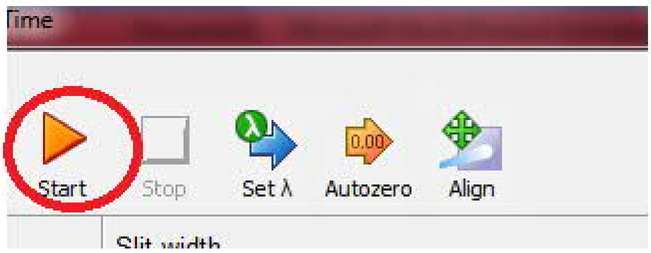
9. Autozero



a. Press ‘Autozero’.
b. Remember to remove everything that might be blocking the beams and click ‘OK’.

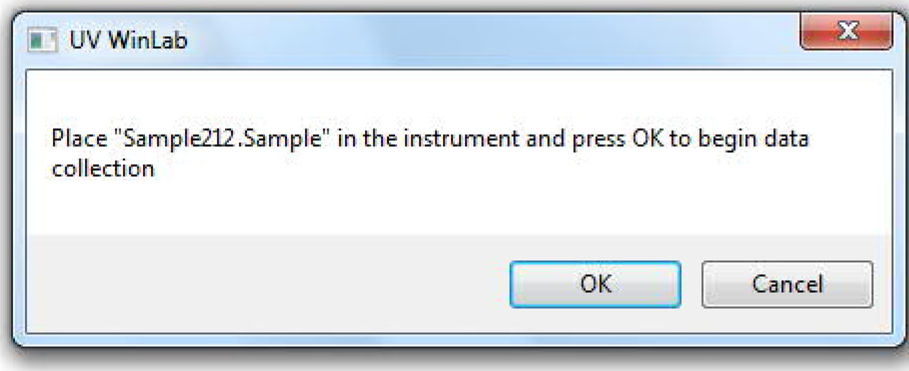


10. Starting the measurement



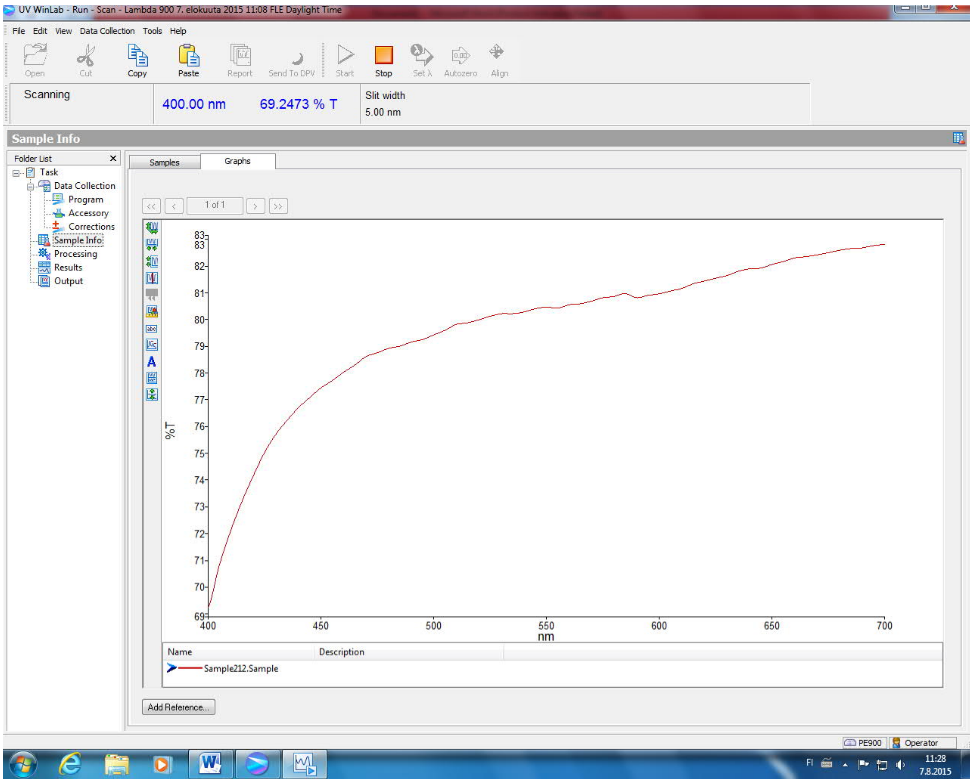
a. Press ‘Start’.

b. Insert your sample and press 'OK'.



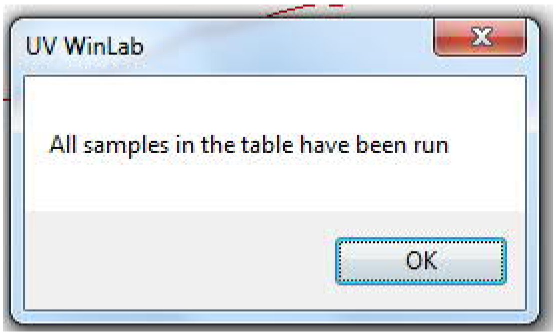
11. During the measurement

a. Results are shown in a graph.



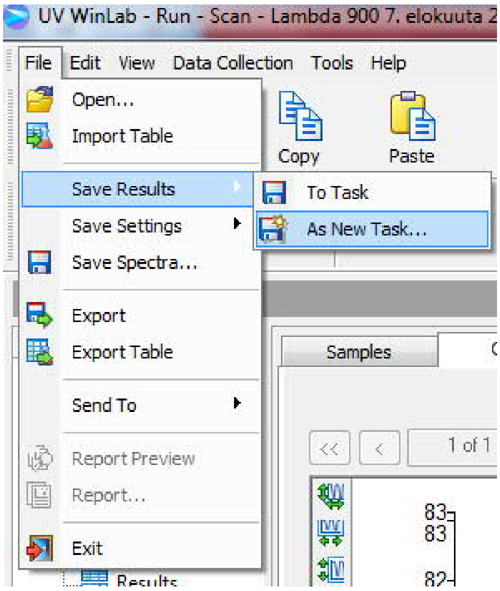
b. If you have several samples, the program will tell you when to insert the next one.

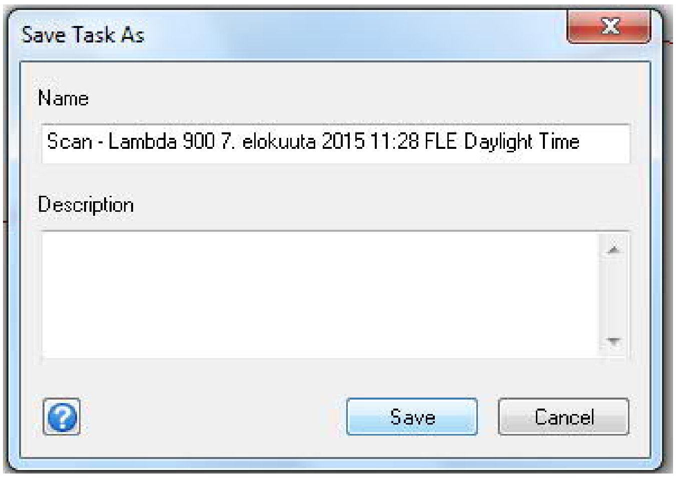
c. When all samples have been measured, the following message is shown, press 'OK'.



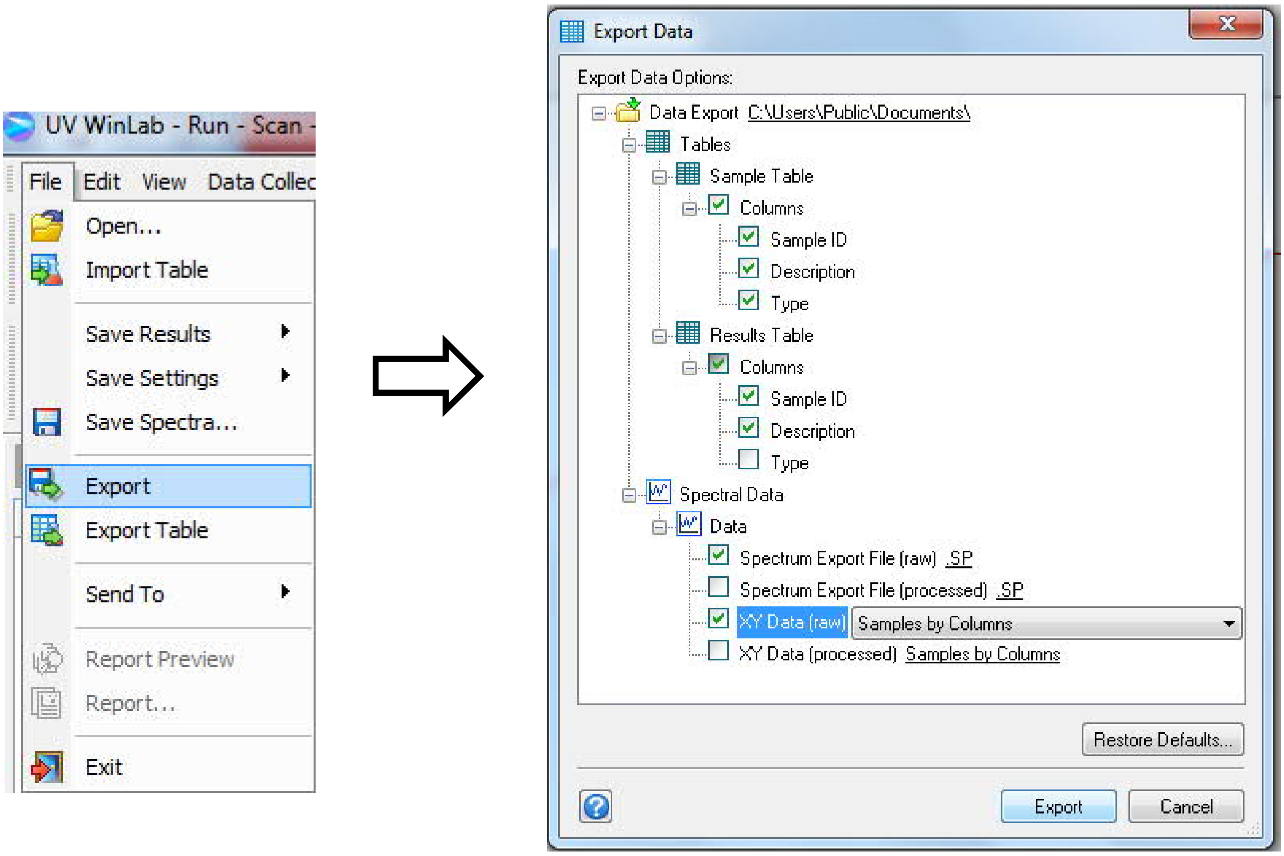
12. Saving the results

a. First, save the data to a task.

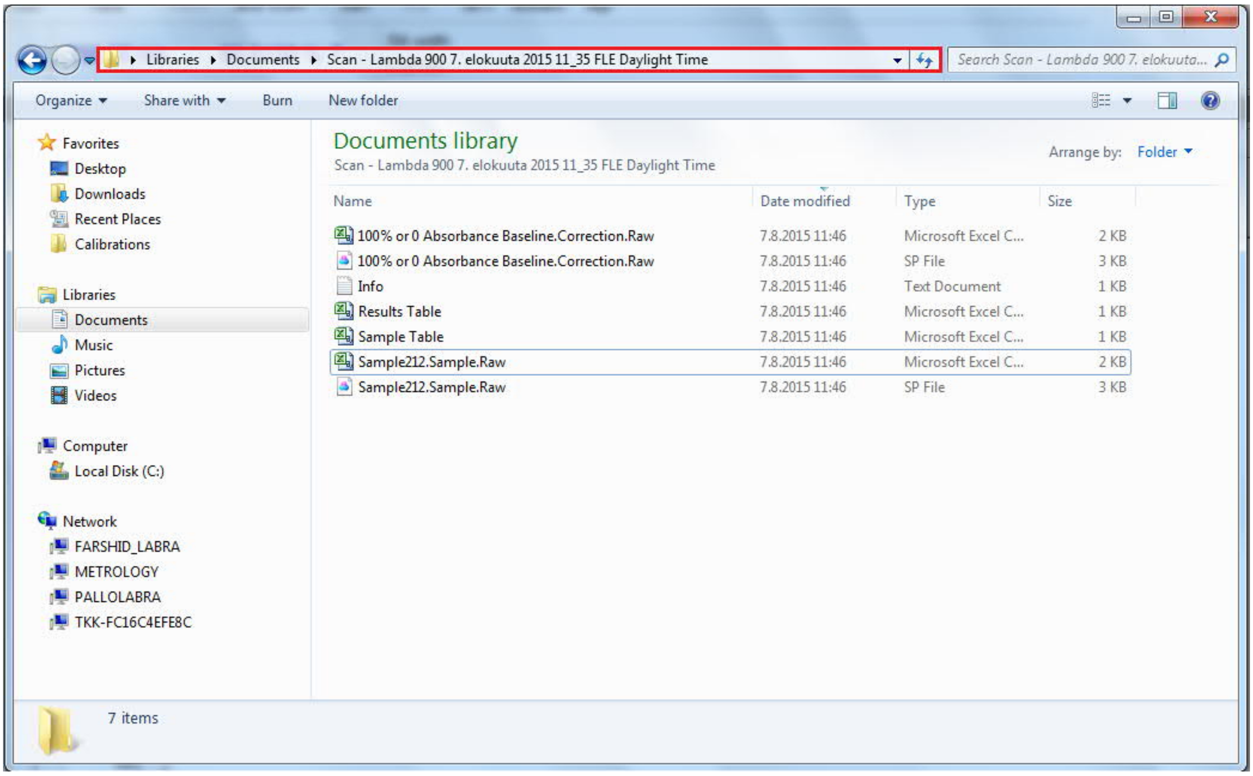




b. Next, click ‘Export’ to export the results data and select the type of data you need.



c. The data folder with the task name should now be found at the documents folder.



6. Handling calibrations 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 [3] with permission from branch manager

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

7. Uncertainty components

7.1. Regular transmittance

Table 2. 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 wavelengths. The numbers are absolute values and valid in the wavelength range of 220 - 1700 nm. 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 2. 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 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 transmittance / %					
	Component	Neutral-density filters			20-nm interference filters		
		UV	Visible	NIR	UV	Visible	NIR
1.	Transmittance of reference 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. Regular absorbance

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

The uncertainty components for absorbance value are determined from those of spectral transmittance as u_A=0.4343 × ΔT/T, where ΔT/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 3 summarises the corresponding uncertainty budgets of Absorbance measurements applied for customer calibrations.

Table 3. 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. Laboratory accommodation and environment

Laboratory accommodation and environment is the same as for the reference spectrometer and is described in ref. [2] Sec. 8.

9. Field calibrations

Not applicable.

10. Control data

Maintenance records of the equipment are written in a chronological order to a notebook labelled "PE900 Spectrometer Data notebook" and it is kept in the Reference spectrometer laboratory.

11. Certificates

Calibration certificates are handled according to [4]. In the calibration certificates included are:

- Ambient temperature and relative humidity,
- Source of traceability,
- Regular transmittance and/or absorbance values at the measurement wavelengths and uncertainty estimates.
- Comparison with previously recorded transmittance and absorbance values if data on articles under investigation is available.

[4] Instructions on writing calibration certificates.

12. Intercomparisons

13. Publications