Laboratory experiment: The Lambert-Beer-Law and Characteristics of Optical Filters

Measuring basic optical properties using a Spectrophotometer (Lambda 1050)

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1 Introduction

1.1 Preliminaries

Before carrying out the experiments, read these instructions thoroughly. With regard to the preliminary talk and the execution of the experiment, it is mandatory to have understood the basic terms of optics and the main principles of the spectrophotometer used. Please check beforehand that you know about the main goals of the experiments. In any case discuss all the work thoroughly with your supervisor. If you are not properly prepared there is no reason to do any of the following lab work. As all of the equipments used in the lab are really expensive, you are urged to work with care and always to ensure that you know what you are doing. If you do not know, please ask the supervisor first in any case!

1.2 General knowledge

You should be able to explain and deal with the following keywords during the preliminary talk and the lab work.

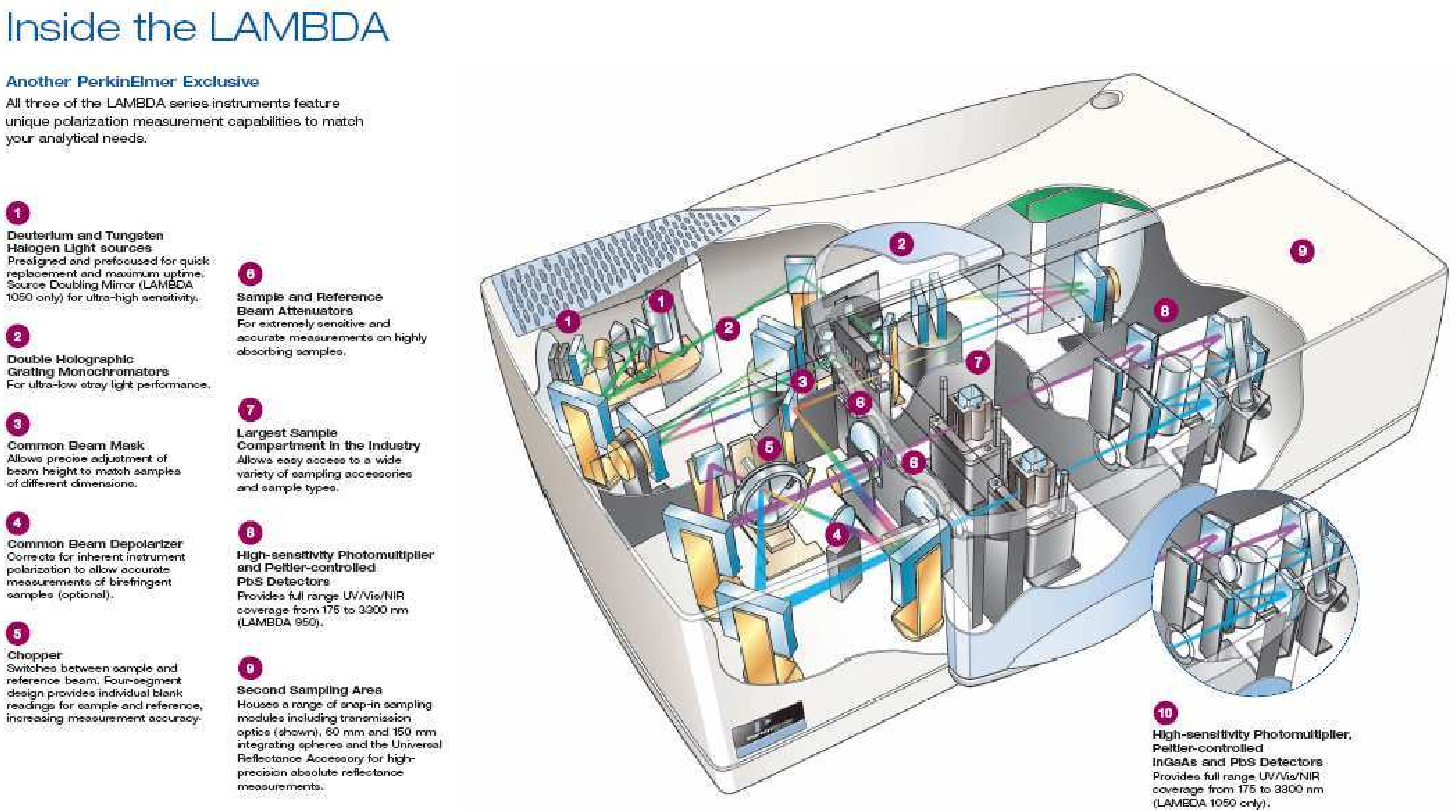
1.2.1 Basic terms of optics

1. Transmittance [Unit]
2. Absorbance [Unit]
3. Spectral bandwidth, definition for a spectrophotometer
4. Nyquist sampling theorem
5. Signal to noise ratio

1.2.2 Spectrophotometer Lambda 1050 from Perkin Elmer

1. Principle of the photoelectric effect
2. Basic functionality of the detectors in the Lambda 1050:
    - Photomultiplier, spectral sensitivity, semi conductive material used
    - InGaAs-Detector, spectral sensitivity, semi conductive material used
    - PbS-Detector, spectral sensitivity, semi conductive material used
    - See the following reference for more details [1]
3. Integration time of the detectors
4. Optical setup, double monochromator
5. Function and use of the chopper wheel
6. Light sources, spectral range
7. See the following reference for more details [2] and [3]

The Lambda 1050 is shown in principle on the following page in Figure 1.1. The software used during the lab work will be explained to you.



Inside the LAMBDA

Another PerkinElmer Exclusive

All three of the LAMBDA series instruments feature unique polarization measurement capabilities to match your analytical needs.

① **Deuterium and Tungsten Halogen Light sources**
Prealigned and prefocused for quick replacement and maximum uptime. Source Doubling Mirror (LAMBDA 1050 only) for ultra-high sensitivity.

② **Double Holographic Grating Monochromators**
For ultra-low stray light performance.

③ **Common Beam Mask**
Allows precise adjustment of beam height to match samples of different dimensions.

④ **Common Beam Depolarizer**
Corrects for inherent instrument polarization to allow accurate measurements of birefringent samples (optional).

⑤ **Chopper**
Switches between sample and reference beam. Four-segment design provides individual blank readings for sample and reference, increasing measurement accuracy.

⑥ **Sample and Reference Beam Attenuators**
For extremely sensitive and accurate measurements on highly absorbing samples.

⑦ **Largest Sample Compartment in the Industry**
Allows easy access to a wide variety of sampling accessories and sample types.

⑧ **High-sensitivity Photomultiplier and Peltier-controlled PbS Detectors**
Provides full range UV/Vis/NIR coverage from 175 to 3300 nm (LAMBDA 950).

⑨ **Second Sampling Area**
Houses a range of snap-in sampling modules including transmission optics (shown), 60 mm and 150 mm integrating spheres and the Universal Reflectance Accessory for high-precision absolute reflectance measurements.

⑩ **High-sensitivity Photomultiplier, Peltier-controlled InGaAs and PbS Detectors**
Provides full range UV/Vis/NIR coverage from 175 to 3300 nm (LAMBDA 1050 only).

Figure 1.1: Schematic drawing of the main features inside the spectrophotometer Lambda 1050 from PerkinElmer. For more information please read the Hardware Guide of PerkinElmer for High-Performance Lambda Spectrometers

1.2.3 The Lambert-Beer Law and the Extinction in Photometry

The absorption of light traveling trough any material can be describe by the Lambert-Beer-Law.

$$I = I_0 \ e^{(-\epsilon x)} \qquad \text{Lambert-Beer-Law} \tag{1.1}$$

with ϵ = Extinction coefficient, I_0 XXX, I XXX and x is.

If the interested substance is mixed or dissolved in some liquid, the extinction coefficient is proportional to the concentration:

$$\epsilon = \epsilon' \cdot c \tag{1.2}$$

$$I = I_0 \ e^{(-\epsilon' \cdot c \ x)} \tag{1.3}$$

with ϵ' = Molar extinction coefficient and c = Concentration of the substance.

Using this law it is possible to determine the molar extinction coefficient of many materials:

$$E' = lg \ \left(\frac{I}{I_0}\right) = -\epsilon' \cdot c \cdot x \tag{1.4}$$

The Lambert-Beer Law is only valid with some preconditions. What are these? Think about the best way of determining the molar extinction coefficient ϵ'

1.2.4 Optical Density (OD)

For a given wavelength, optical density is an expression of the transmittance of an optical element.

- Optical density is expressed by $log_{10}\left(\frac{1}{T}\right)$ where T is transmittance.
- The higher the optical density, the lower the transmittance.
- Optical density times 10 is equal to transmission loss expressed in decibels, e.g. an optical density of $0,3$ corresponds to a transmission loss of 3 dB.

1.2.5 Optical Filters

Optical filters are widely used to transmit only some parts of the whole spectrum, which is needed for the experiment. Three main categories of filter types are available:

- Shortpass filters
- Bandpass filters
- Longpass filters

The manufacturing processes of these filters can vary due to usage, costs and precision. The main manufacturing processes are named below. You should be able to explain the main techniques used and some advantages and disadvantages of these processes:

- Color glass filters
- Dielectric bandpass filters
- Dichroic color filters

One property of optical filters is the cut-on and cut-off slope. The cut-on slope is defined as follows,

$$Slope \ \% = \frac{\lambda_{0.3} - \lambda_4}{\lambda_4} \times \ 100 \tag{1.5}$$

and the cut-off slope as

$$Slope \ \% = \frac{\lambda_4 - \lambda_{0.3}}{\lambda_4} \times \ 100 \tag{1.6}$$

where λ_n is the wavelength, where the transmittance is only n % of the maximum transmittance.

2 The experimental work

2.1 Preparation

2.1.1 Chemical components and equipment

- Copper(2)-sulfate (Chemical formula: $CuSO_4$; Exact mass: 158.88; Molecular weight: 159.61 $\frac{g}{mol}$), powder. It is also known, dissolved in water, as ”Fehling Solution A” for the chemical estimation of sugar.

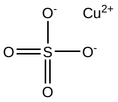


Figure 2.1: Chemical structure of Copper(2)-sulfate used in the Lambert-Beer measurements.

- Fehling Solution A, concentration of $CuSO_4$ unknown.
- Distilled water

Please dispose any of these chemical compounds in the proper waste canister!!

For the preparation you will work with the following lab-equipment:

- Magnetic stirrer
- Precision balance, and weighting paper
- Lab beaker and graduated cylinder
- Pipettes and cuvettes

2.1.2 The Copper(2)-sulfate solutions

The preparation of the all Copper(2)-sulfate solutions is most important for the accuracy of your results. Please work always in a clear and clean way. The first solution should contain Copper(2)-sulfate in a concentration of $1,00\ \frac{mol}{l}$. Do not produce more than 10 to 20 ml of thit solution. To weight the right amount of $CuSO_4$ use the precision balance with the weighting paper. Fold the paper once or twice to get it handy. Thoroughly measure the distilled water in the graduated cylinder and the water in the beaker on the magnetic stirrer. Slowly add the Copper(2)-sulfate powder and ensure that all powder is dissolved. Stir for a few minutes.
For verifying the Lambert-Beer law and to measure the normalization curve you will need the following concentration levels, each in one independent cuvette:

Cuvette	Concentration of $CuSO_4$	Volume $CuSO_4$ Solution	Volume Water
[-]	$[\frac{mol}{l}]$	[µl]	[µl]
1	1	1000	0
2	0.9	900	100
3	0.8	800	200
4	0.7	700	300
5	0.6	600	400
6	0.5	500	500
7	0.4	400	600
8	0.3	300	700
9	0.2	200	800
10	0.1	100	900
11	0	0	1000

2.1.3 The Spectrophotometer LAMBDA1050

This spectrophotometer has a value of about 100 000.- €. In any case before you "'try"' to do something, which you are not sure about: STOP IT :-). Please ask your supervisor beforehand. Due to the old fashioned RS232 communication between the LAMBDA1050 and the PC, there exists a specific order how to switch on and start the operating program UVWinLab:

- Switch on the PC and log yourself in. Password and user name are written on the keyboard.
- Switch on the LAMBDA1050 using the small green button on the far right top side. Now the spectrophotometer will initialize all the system components, which will take about 5 minutes.
- Start the UVWinLab program using the shortcut on the desktop of the PC.
  Login: LAMBDA1050 Password: sehnix123!
- The device is ready for the measurements

If you still get a message with a server error, you may have been too fast trough the above steps. Please ask the supervisor for help.

For the Lambert-Beer-Law measurements you will need the two cuvette holders installed in the sample compartment. To ensure a proper optical pathway trough the cuvette, you should always check using the zero order light, that the light spot is positioned in the cuvette center. By adjusting the CBM (Common Beam Mask) you can resize the height of the light spot. By adjusting the optical slit width you can resize the width. The neutral density filters and color filters will be mounted using the 30 mm micro cage system holder. You will have to adjust both beams again, according to the sample properties.

2.2 Verifying the Lambert-Beer Law

The following parameters should be used for these measurements:

- Wavelength range 400 $nm \rightarrow$ 800 nm
- 100% correction. Please correct all data to distilled water
- Integration time of the detector as much as necessary to get no noise in the signal
- Beam depolarizer "on"
- Use only the halogen lamp, therefor the lamp change $<$ 400 nm

Measure all $CuSO_4$ samples, the distilled water at last, to get a control sample.

Decide which wavelength is best to be used for the following calculations in your report. Discuss this matter with the supervisor. Perform the calculation using formula 1.4. What kind of graph do you expect, if the Lambert-Beer law is fulfilled? Do the calcuations at three different wavelength and compare the results.

2.3 Determining the concentration of a unknown Fehling solution

The Fehling solution from Roth chemicals has got an unknown concentration of $CuSO_4$. Determine the actual concentration of this chemical in $\left[\frac{mol}{l}\right]$ by using the measured results from above.

2.4 Absorptive neutral density filters

Measure three filters over a wide range of the VIS-spectra of about 200 $nm \rightarrow$ 800 nm. Use the internal attenuators to adjust the reference and signal channels to a dynamic range, which gives you a minimum of noise. Thus resulting in a wider spectral range, where the filters can be properly measured. The attenuators can be adjusted to the following values:

$$\begin{aligned} 100\% &\rightarrow OD\ 0 \\ 1\% &\rightarrow OD\ 2 \\ 0,1\% &\rightarrow OD\ 3 \\ 0,0\% &\rightarrow OD\ \ > 3 \end{aligned}$$

Discuss the use of this technique with the supervisor and, if possible, think of reasons why this technique is used for high optical densities and does not work with low optical densities. Determine the optical densities of three out of five neutral density filters, numbered from 1 to 5.

2.5 Bandpass, short pass and long pass filters

Measure all filters over a wide range of the VIS-spectra of about 200 $nm \rightarrow$ 1200 nm. Decide at which wavelength the detector and monochromator change , as well as the lamp change should occur. Determine the cut-off and cut-on slopes according to formula 1.5 and 1.6 of each of the filters.

3 Report and Data processing

To be able to write a proper report of these experiments the following principles should be taken into account during the work:

- The lab work has to be completely documented. Write down all parameters of each sample, measurement etc. that you are adjusting. Parameters like integration time, slit width, references, 100% references are quickly changed and if not written down, often not recoverable after the next measurement.
- Check already during your work, if the measured data does make sense.
- Properly name your samples in the data.

Some hints and recommendations for your report:

- There is no minimum or maximum length of your report, write as much as you think it is necessary.
- The report should contain all necessary raw and processed data including descriptions on the way they were retrieved. (Only graphs, not data listing.)
- Stick to clear and scientific wording.
- All graphs in the report need the right axis descriptions and have to be mentioned in the text and/or interpretation.
- Do not cite Wikipedia, we have scientific references, which can be used.

Some technical and data processing hints:

- Most preferred is a report written in LaTeX. MS Word is also ok.
- To prepare nice looking and simple graphs you can use the freeware GnuPlot.
- Please backup your measured data and the report to avoid reworking parts of the lab :-)

Bibliography

[1] Ralph W. Engstrom. *Photomultiplier Handbook : theory design application.* Lancaster, 1980. Literaturverz. S. 176.

[2] Casimer M. DeCusatis, editor. *Handbook of applied photometry.* American Institute of Physics, AIP Pr., [Woodbury, NY], 1998.

[3] Sune Svanberg. *Atomic and molecular spectroscopy : basic aspects and practical applications; with 14 tables.* Springer series on atomic, optical, and plasma physics ; 6. Springer, Berlin, 3., rev. and enl. ed. edition, 2001. Pp. : DM 198.00.