3rd IR-RF Workshop

27.02.2018 Leipzig

Practical guide to IR-RF spectrometry

Dirk Mittelstraß
Currently master student physics at TU Dresden
Former employee at:
- Freiberg Instruments (2007 – 2014)
- AG Geomorphology, JLU Gießen (2014 - 2015)

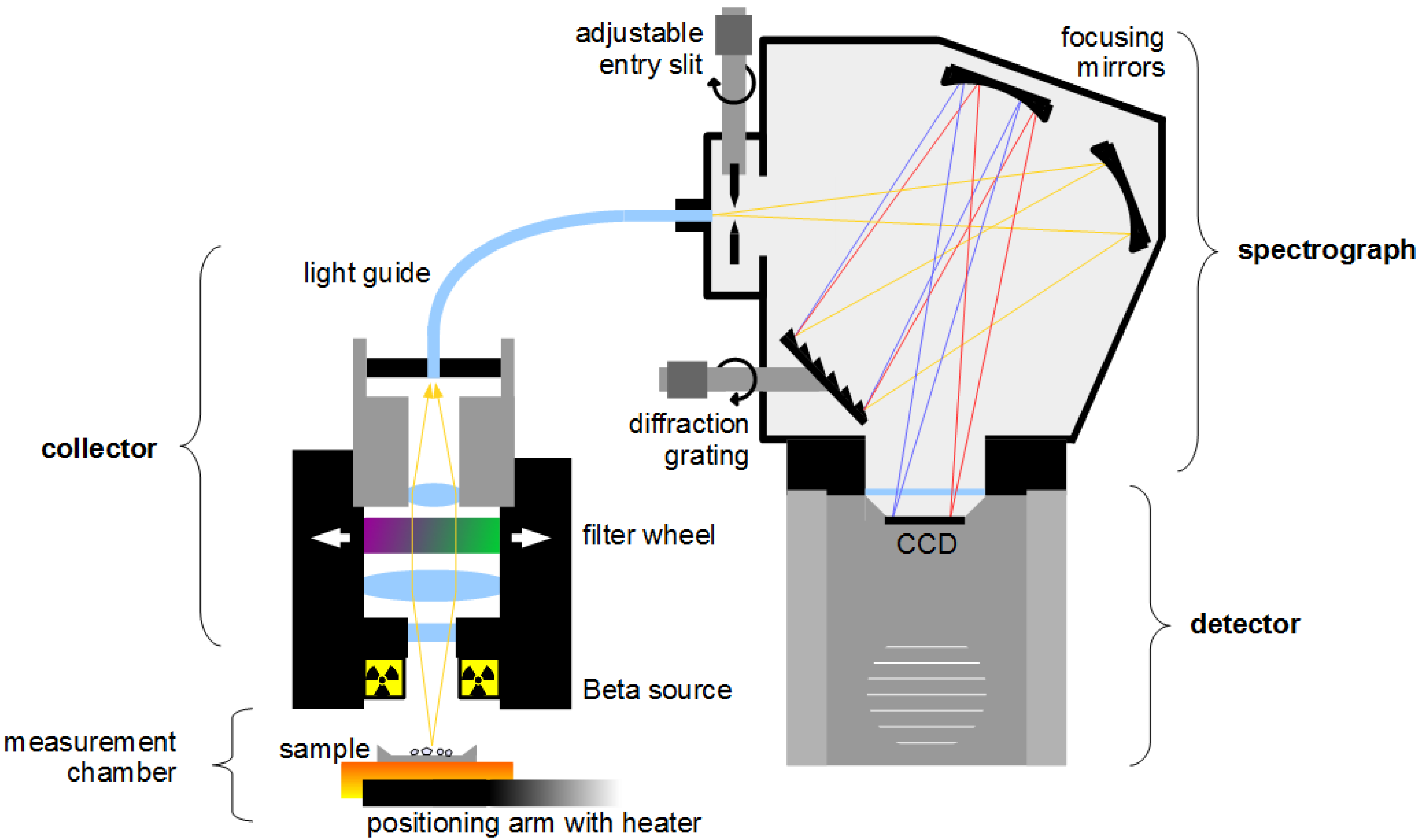
mittelstrassdirk@gmx.de

Practical guide to IR-RF spectrometry

1. Luminescence spectrometry build-up
2. Simulated feldspar spectrum
3. From luminescence emission to raw data
    1. Light collection
        - Collecting efficiency
        - Coupling efficiency
        - Stem effect
    2. Spectrograph
        - Diffraction efficiency
        - Signals higher order
        - Spectral coverage
    3. Camera
        - Quantum efficiency
        - Cosmic rays
        - Noise and offset
    4. Comparison Before and After
4. From raw data to luminescence emission
    1. Outlier removal & smoothing
    2. Background substraction
    3. Spectral response function
    4. Comparison Real and Corrected

1. Spectrometry build-up

lexsyg research Gießen



2. Simulated feldspar spectrum

Assuming five feldspar emissions, identified in nearly all of 35 various mineral feldspar samples by Trautmann (1999)*

No.	Peak (eV)	σ (eV)	Peak (nm)	σ (nm)
1	3.3	0.25	376	29
2	2.9	0.3	428	44
3	2.2	0.2	564	51
4	1.7	0.11	729	47
5	1.42	0.07	873	43

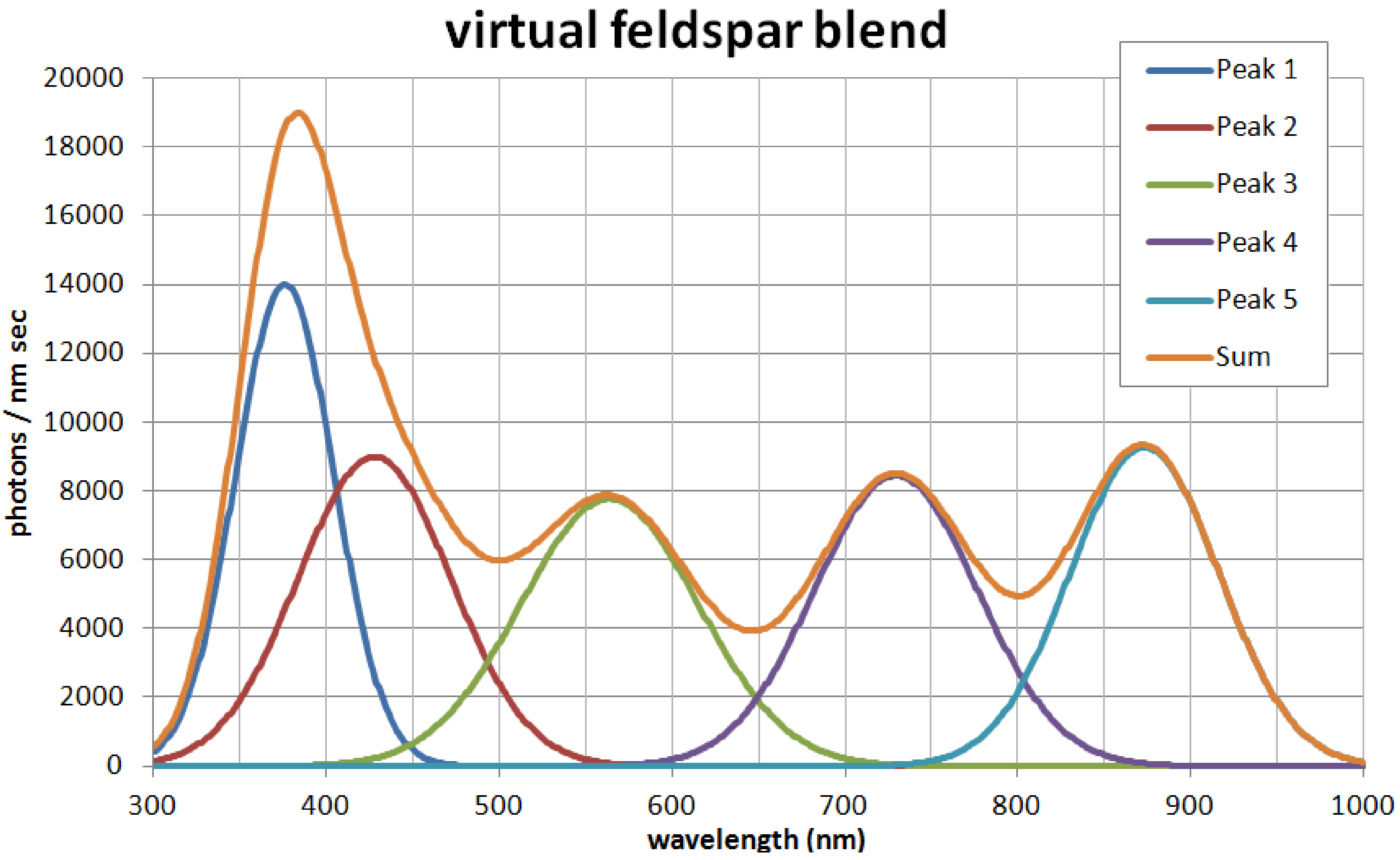
Assuming steady and equal photon flux and gaussian distribution for all emissions:

$$\phi(\lambda) = \frac{\phi_{emission}}{\sqrt{2\pi\sigma^2}} \mathrm{e}^{-\frac{(\lambda-\lambda_{max})^2}{2\sigma^2}} \quad \text{with} \quad \phi_{emission} = 10^6 \text{ photons/sec}$$

Result is a *virtual feldspar blend* spectrum

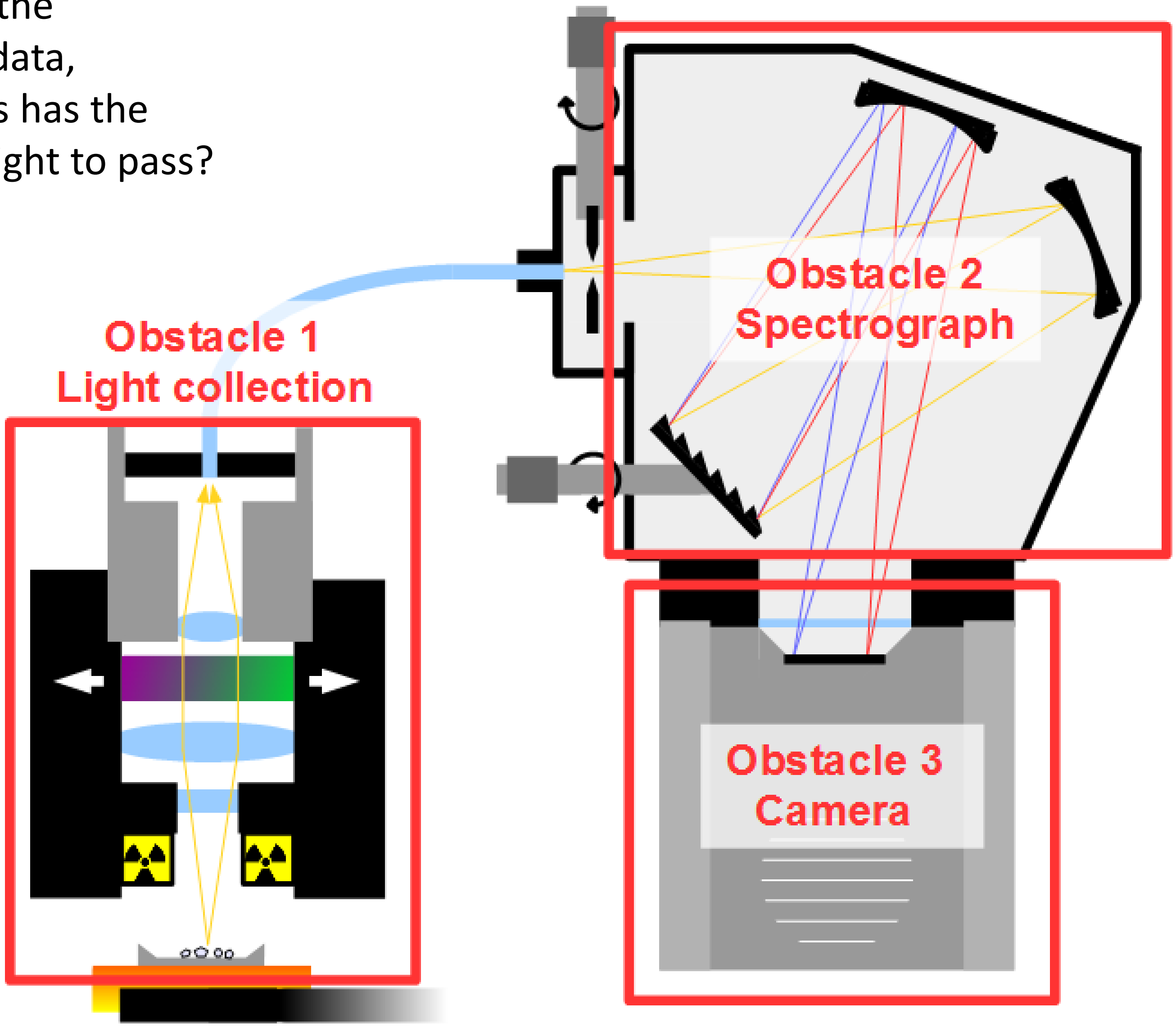
Reference:
Trautmann T., PhD thesis, 1999: Radiolumineszenzuntersuchungen an Feldspat. TU Freiberg
Data can be found at table A3 (note: Trautmann confused sigma and FWHM)

2. Simulated feldspar spectrum



3. From emission to raw data

When forming the measurement data, which obstacles has the luminescence light to pass?



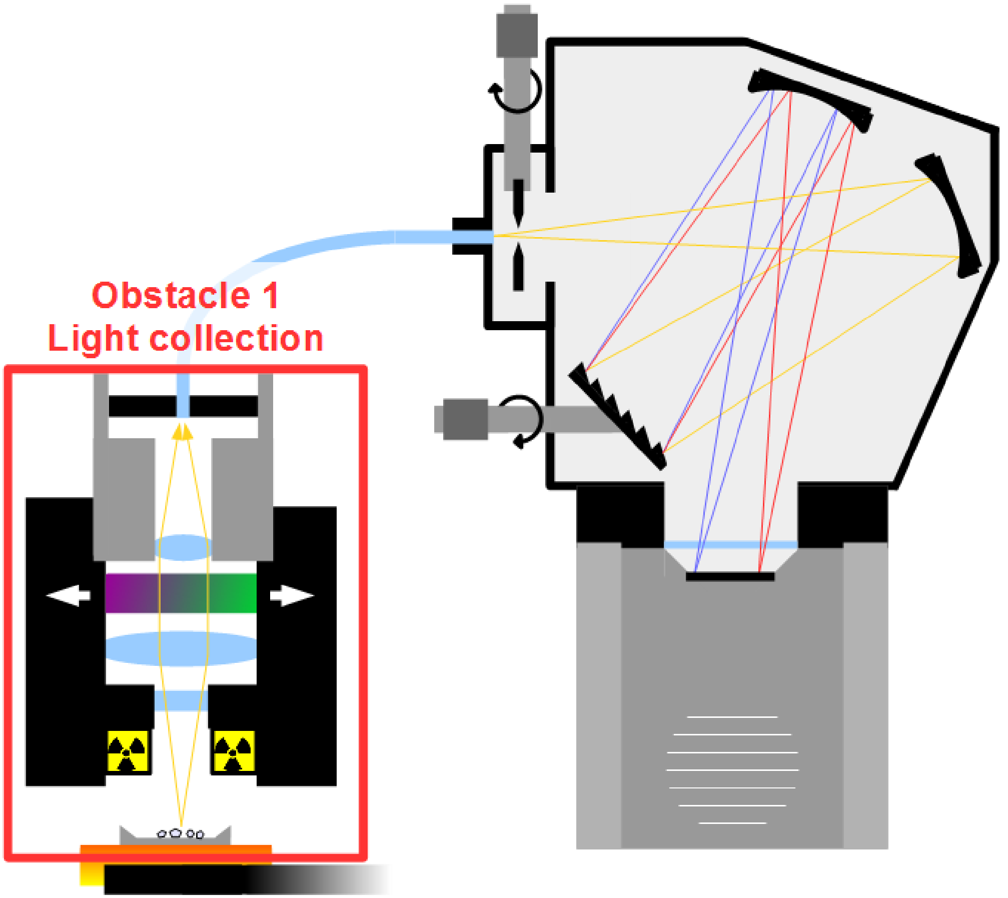
3.1. Obstacle 1: Light collection

Issues

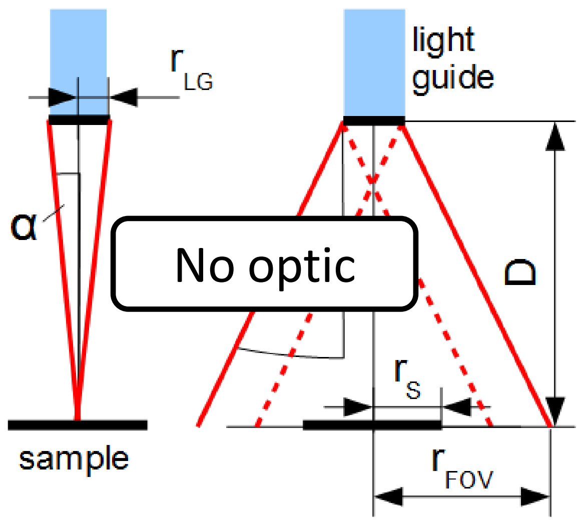
1. Light collecting efficiency of (no) optic
2. Coupling efficiency light guide
3. Cherenkov light & radiouminescence of glass elements (stem effect)

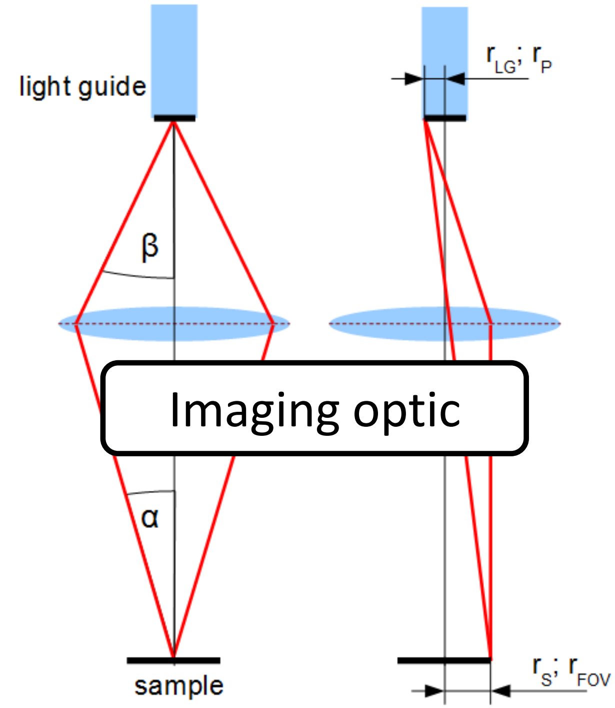
Ignored here

- Chromatic abberation
- Optical abberations
- Filter transmission
- Light guide transmission
- Lens/safety glass transmission
- Glass surface reflectance
- Radio- or Photoluminescence of mechanical elements



3.1.1. Collecting efficiency of (no) optic





<u>Three set-up parameters…</u>

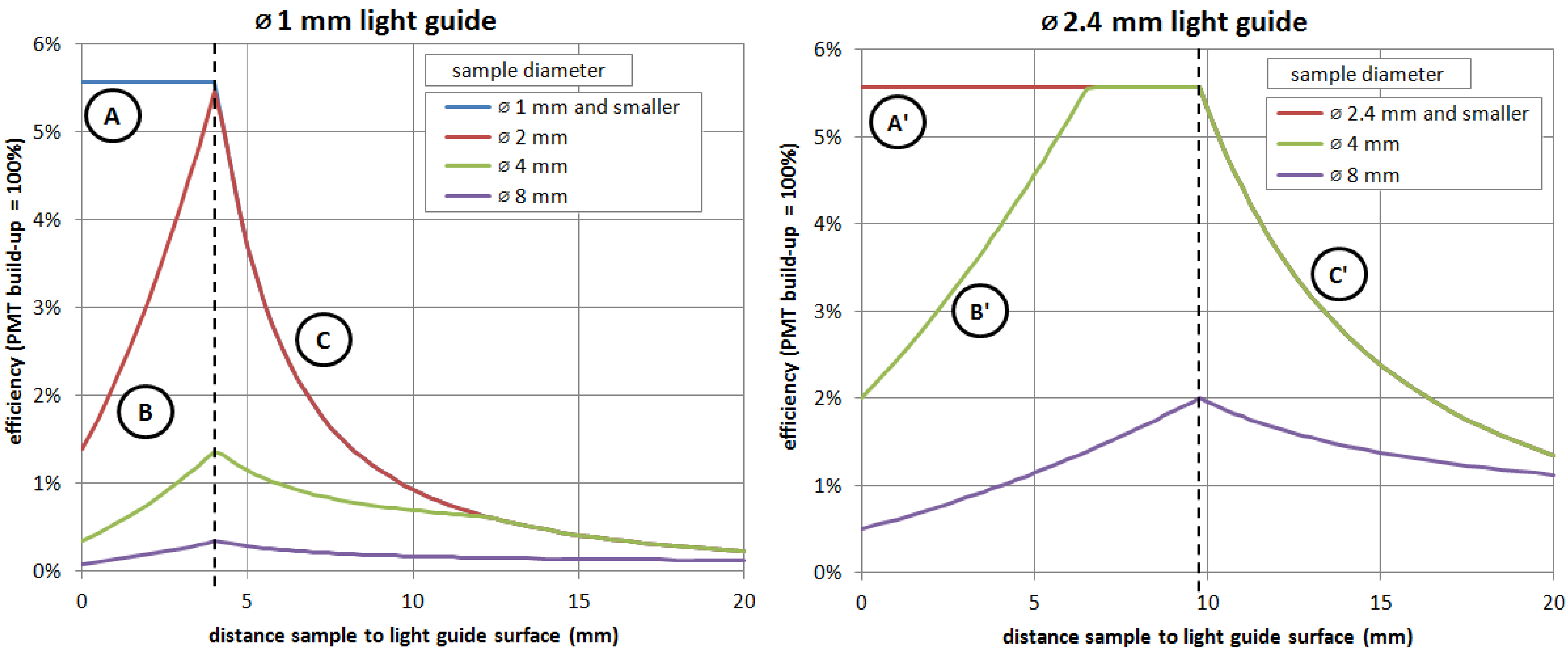
Acceptance angle spectrograph:	angle ↑, efficiency ↑
Light guide thickness:	diameter ↑, efficiency ↑
Sample size:	diameter ↑, efficiency per mm^2 ↓

<u>… lead to one optimization parameter:</u>

No optic:	**Distance sample to light guide**
Imaging optic:	**Lateral magnification**

3.1.1. Collecting efficiency of (no) optic

Case: **No optic**



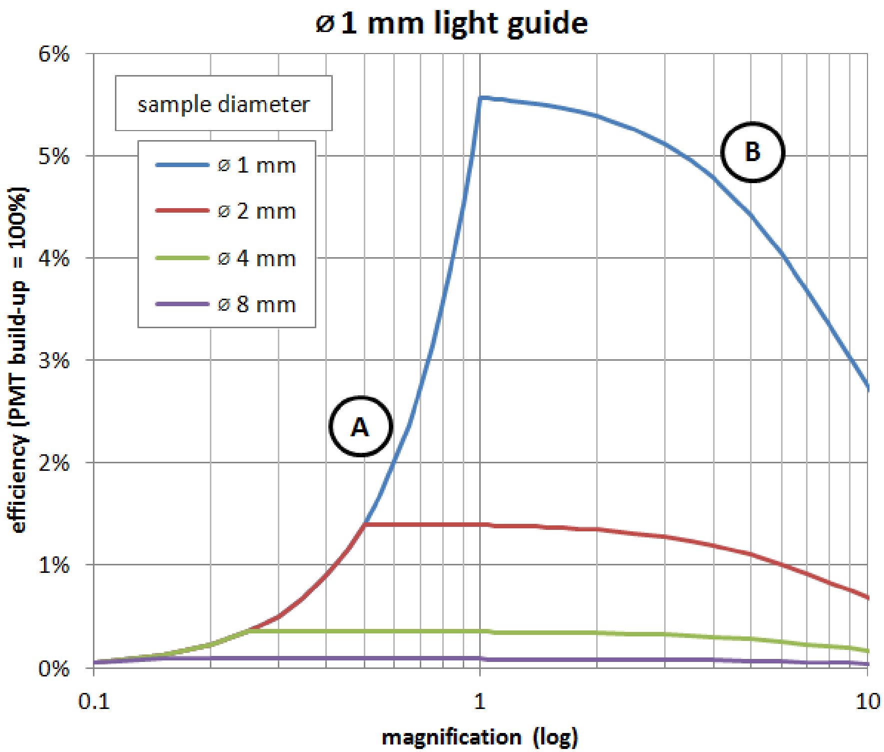
Rule of thumb:

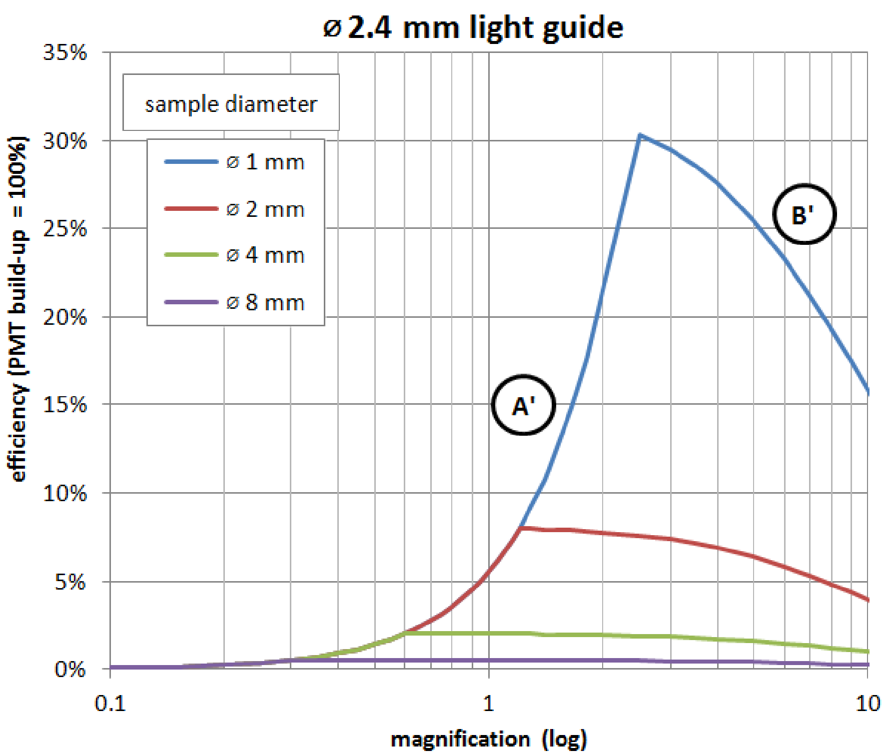
best distance value ≈ 4x light guide diameter

best sample size ≈ 2x light guide diameter

3.1.1. Collecting efficiency of (no) optic

Case: **Imaging optic**





Rule of thumb:

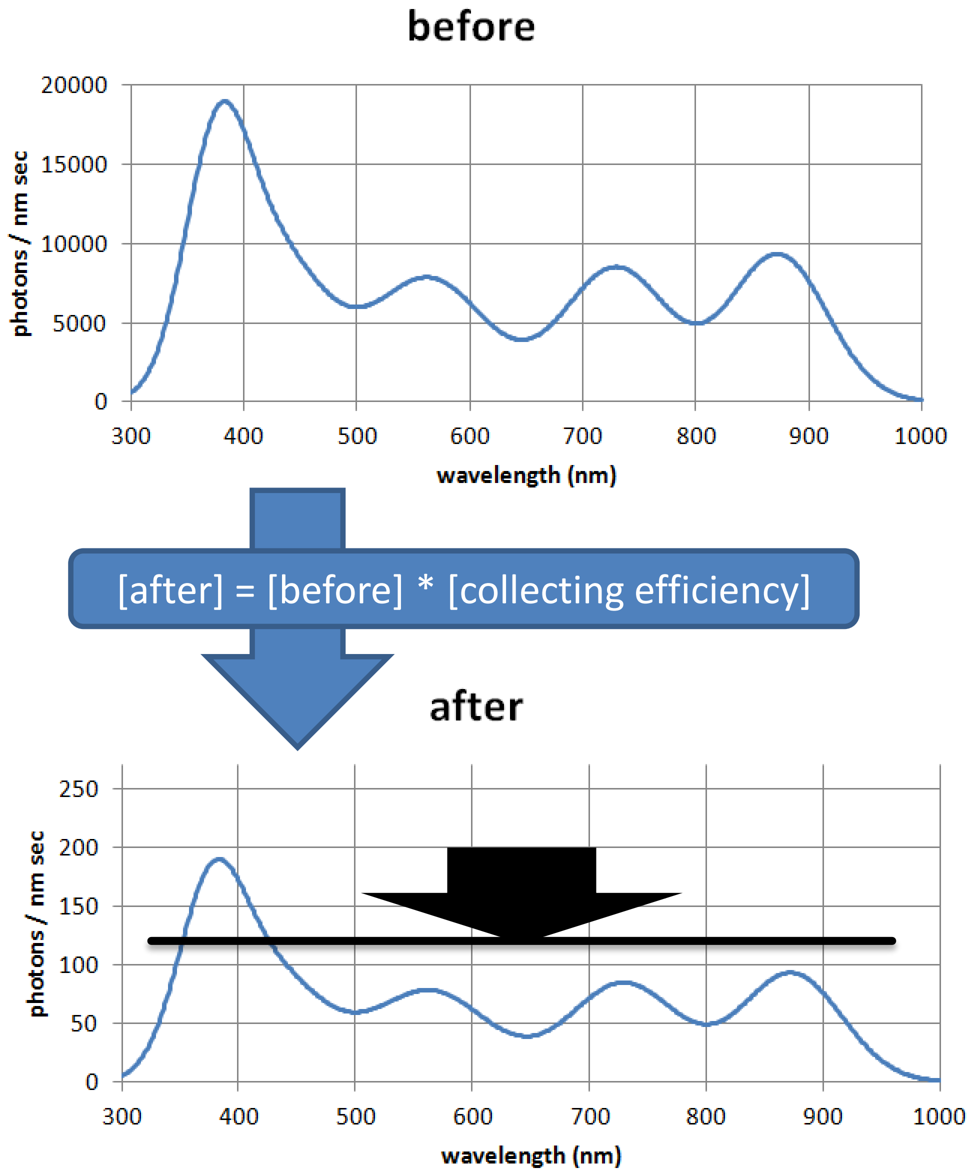
best magnification value ≈ 1x

best sample size ≈ 1x light guide diameter

3.1.1. Collecting efficiency of (no) optic

<u>best case scenario:</u>

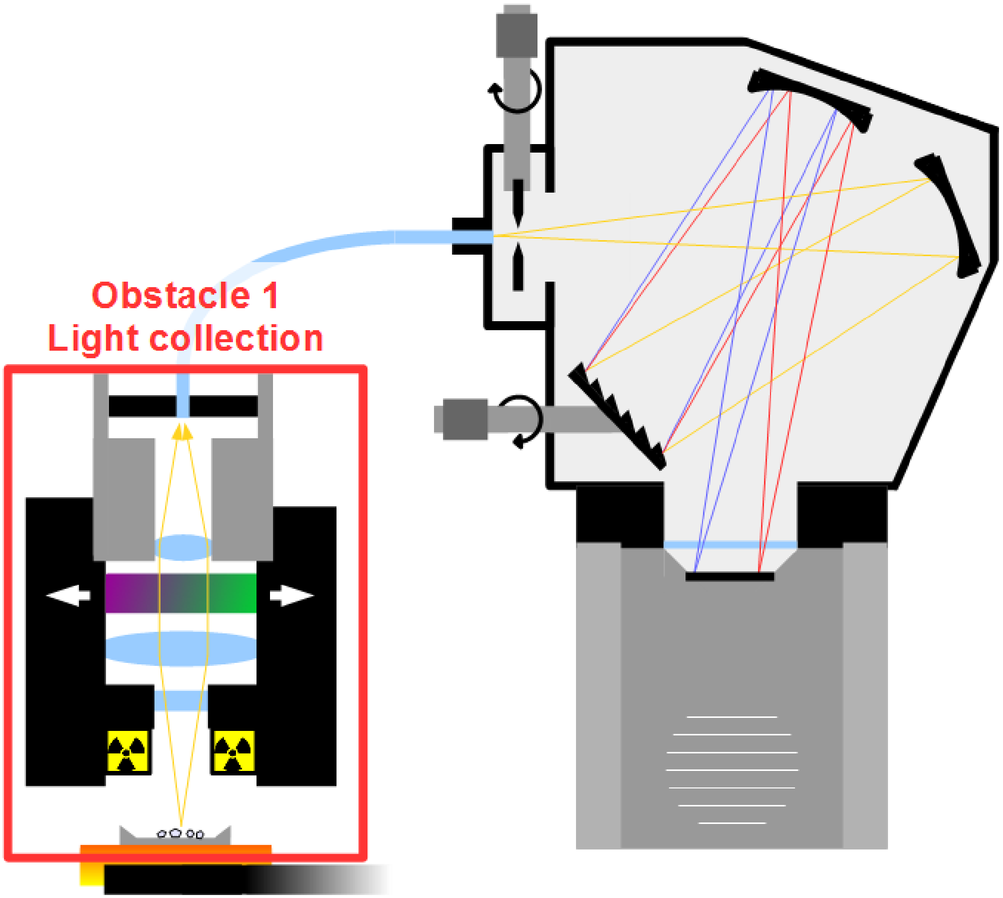
~ 1 % of luminescence light is collected



3.1. Obstacle 1: Light collecting

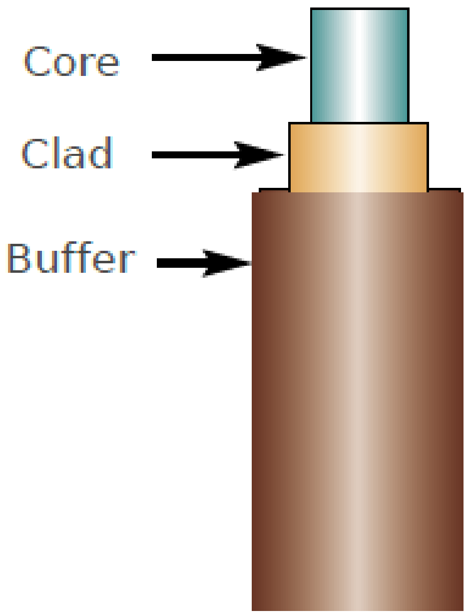
Issues

1. ~~Light collecting efficiency of (no) optic~~ → loss of > 99% of signal
2. **Coupling efficiency light guide**
3. Cherenkov light & radiouminescence of glass elements (STEM effect)

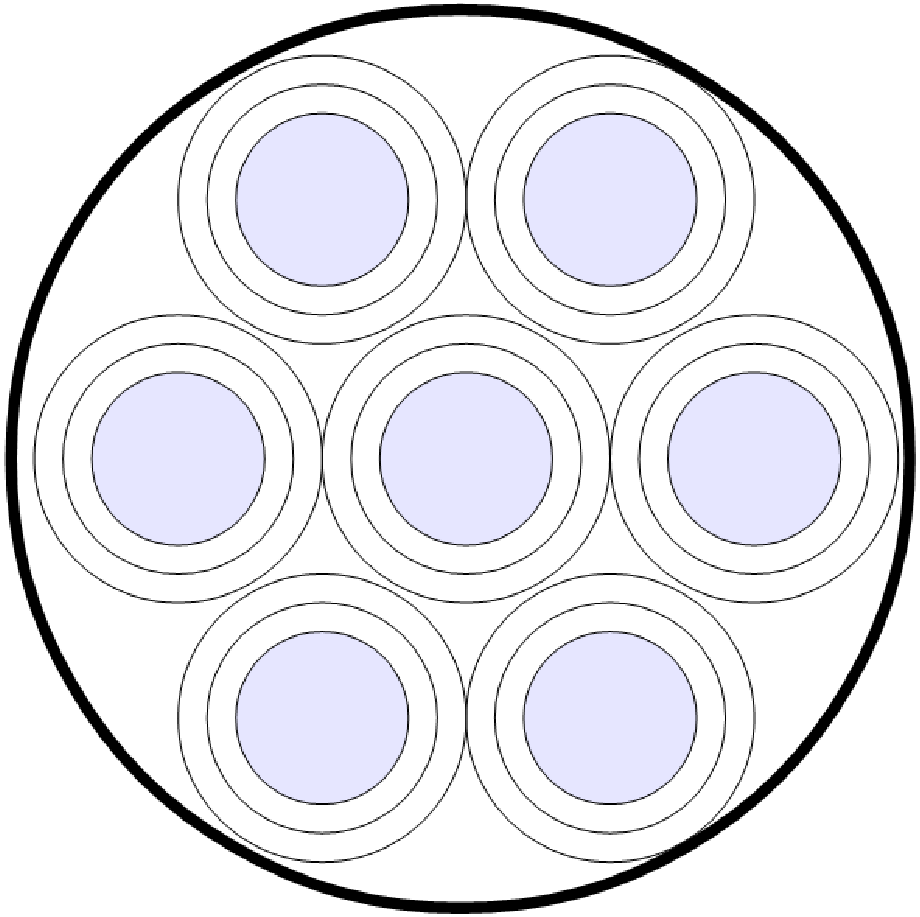


3.1.2. Coupling efficiency

Depending on core-to-fibre ratio, **50 – 70 %** of light guide surface is sensitive to light



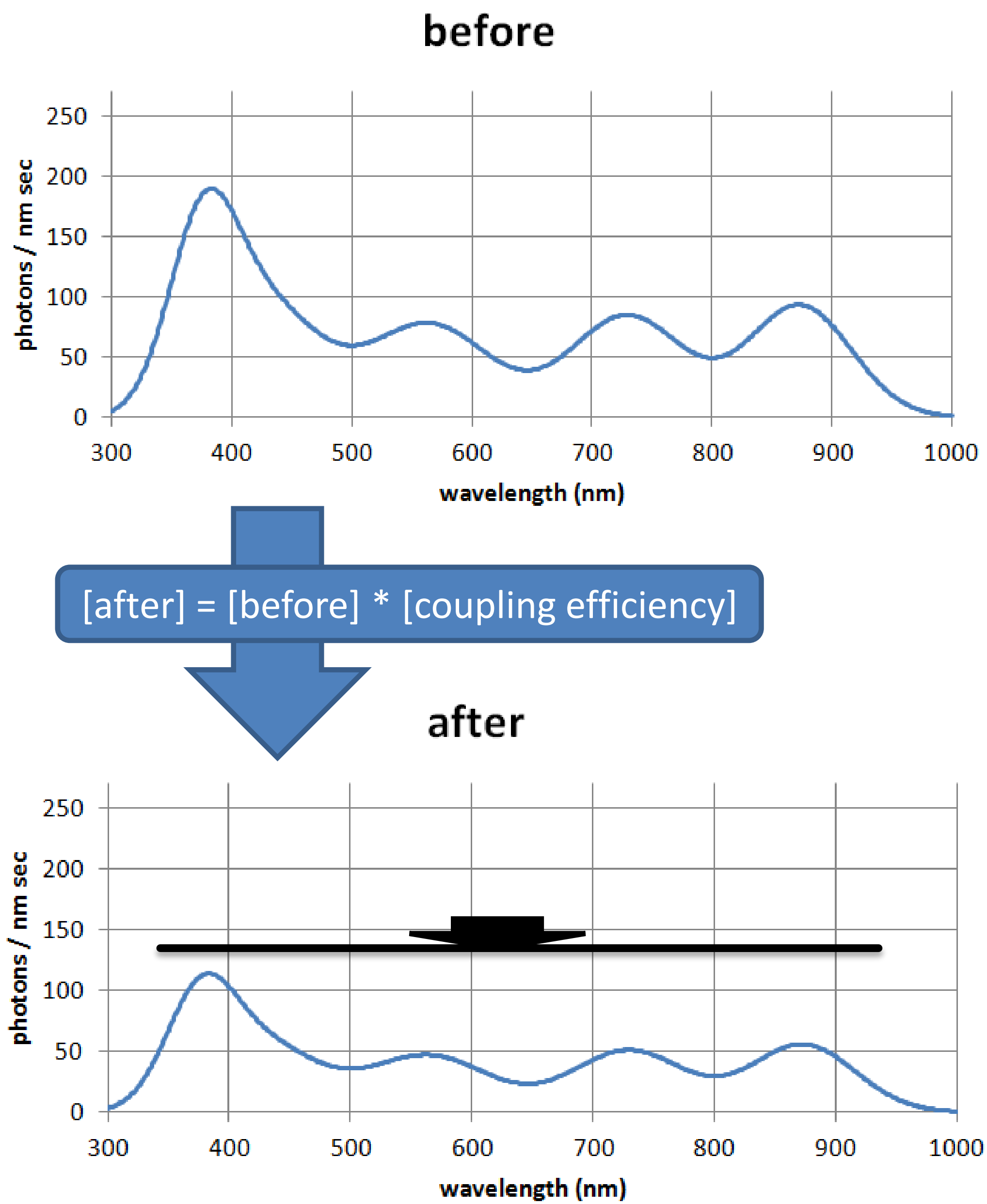
Light guide fibre sheme
(from MOLEX Ultra Low –OH data sheet)



Light guide section with 6 fibres
(usually ~ 100 fibres)

3.1.2. Coupling efficiency

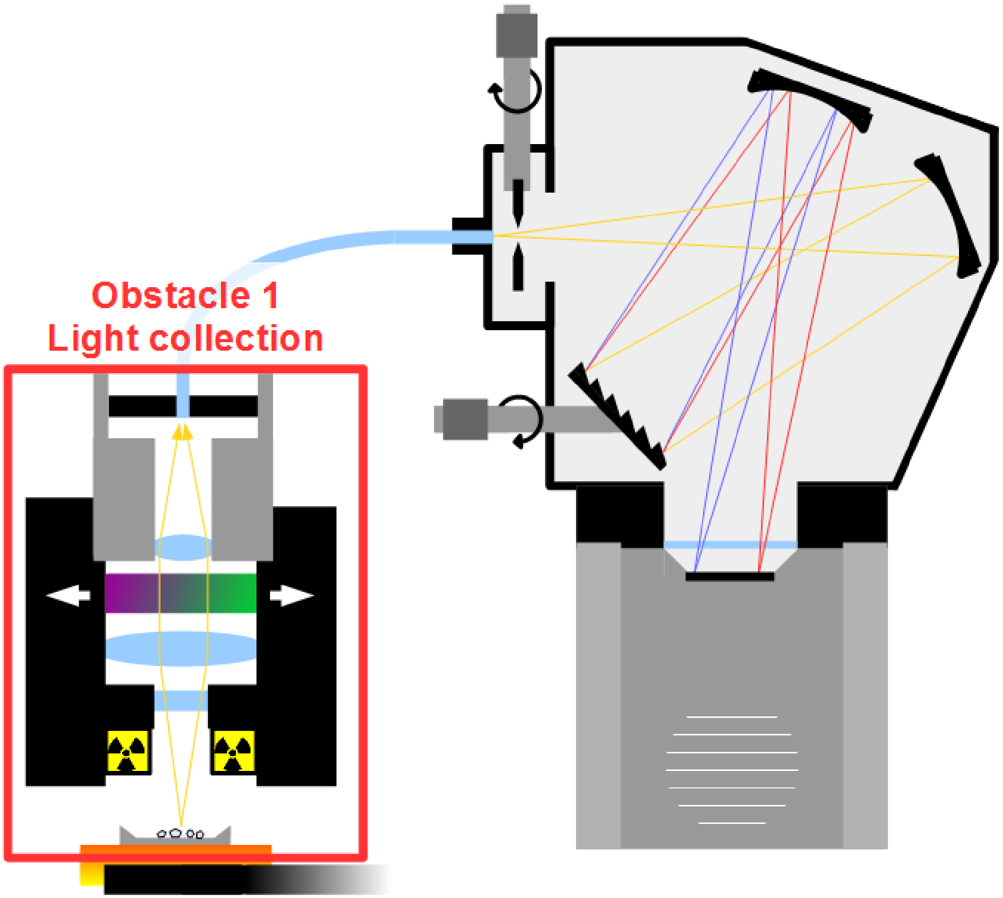
~ 60 % of collected light
is coupled into light guide



3.1. Obstacle 1: Light collecting

Issues

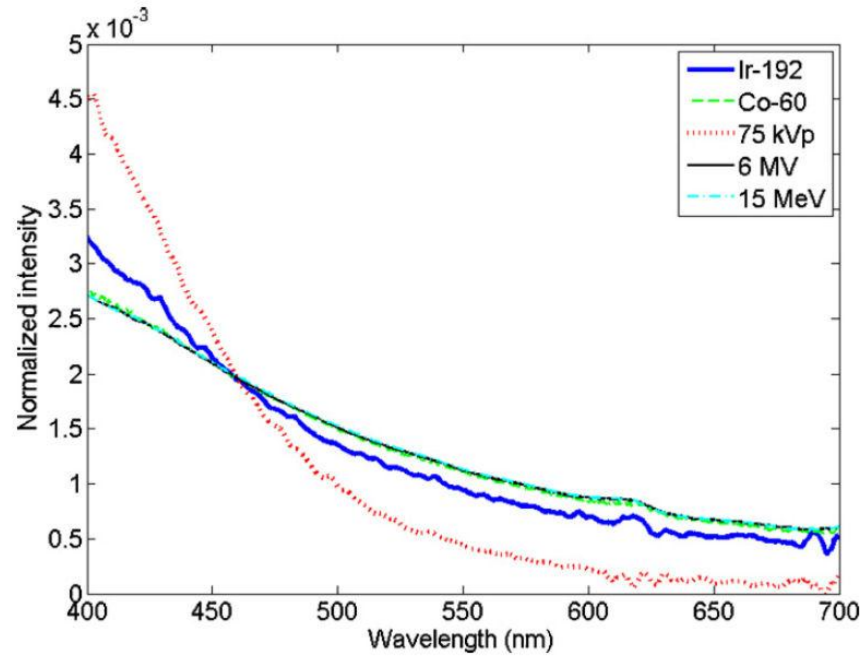
1. ~~Light collecting efficiency of (no) optic~~ → loss of > 99% of signal
2. ~~Coupling efficiency light guide~~ → loss of ~30% of signal
3. **Cherenkov light & radiouminescence of glass elements (stem effect)**



3.1.3. stem effect

Cherenkov radiation & radioluminescence of glass elements close to the irradiation source cause an additional signal background, probably dominated by a blue component

- known in medical dosimetry, see Yukihara & McKeever (2011) page 249
- **Not investigated in Risö/lexsyg RF systems so far**



Some stem effect spectra of a plexiglas light guide, Therriault-Proulx et al. (2013), figure 2

References:

Therriault-Proulx, F., Beaulieu, L., Archambault, L., Beddar, S., 2013. On the nature of the light produced within PMMA optical light guides in scintillation fiber-optic dosimetry. Physics in Medicine and Biology

Yukihara, E.G., McKeever, S.W.S., 2011. Optically stimulated luminescence: fundamentals and applications. Wiley, Chichester, West Sussex.

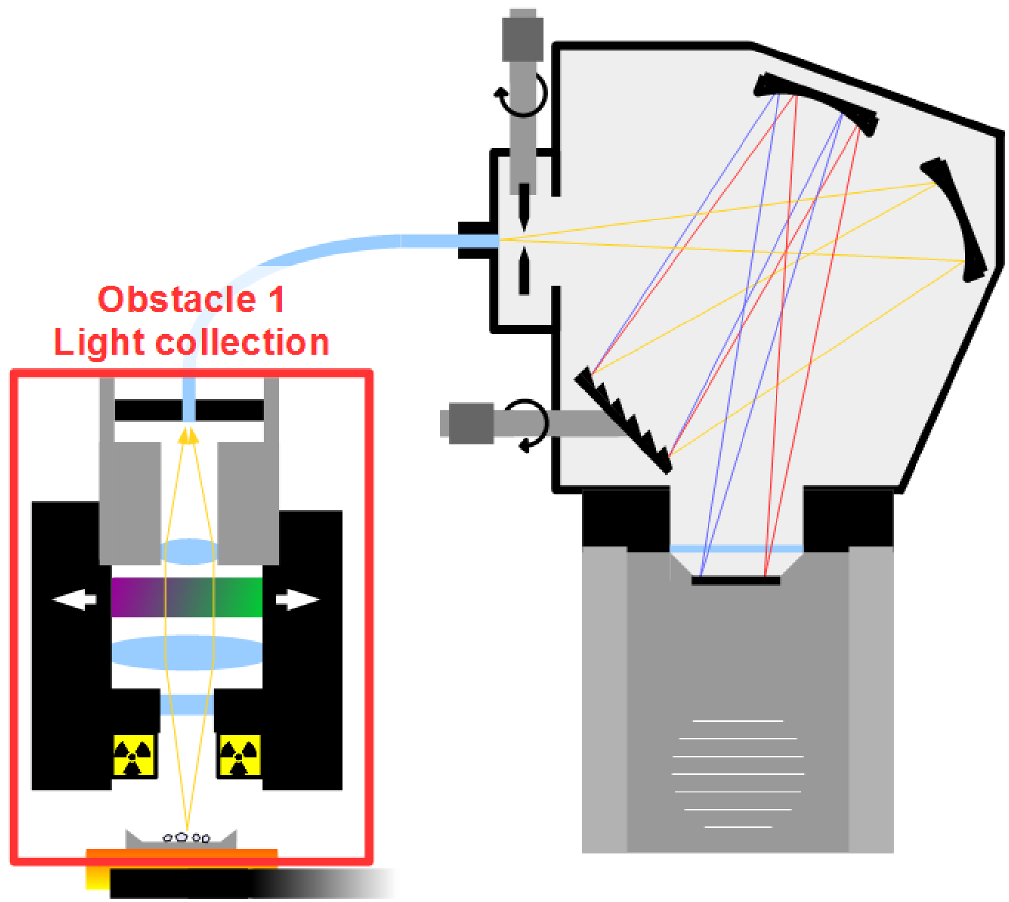
What is Cherenkov radiation?

blue light caused by electric ‚supersonic' shock waves of high energy electrons (> ~200keV) in a medium.

3.1. Obstacle 1: Light collecting

Issues

1. ~~Light collecting efficiency of (no) optic~~
   → loss of > 99% of signal
2. ~~Coupling efficiency light guide~~
   → loss of ~30% of signal
3. ~~Cherenkov light & radiouminescenc of glass elements (stem effect)~~
   → some extra signal background



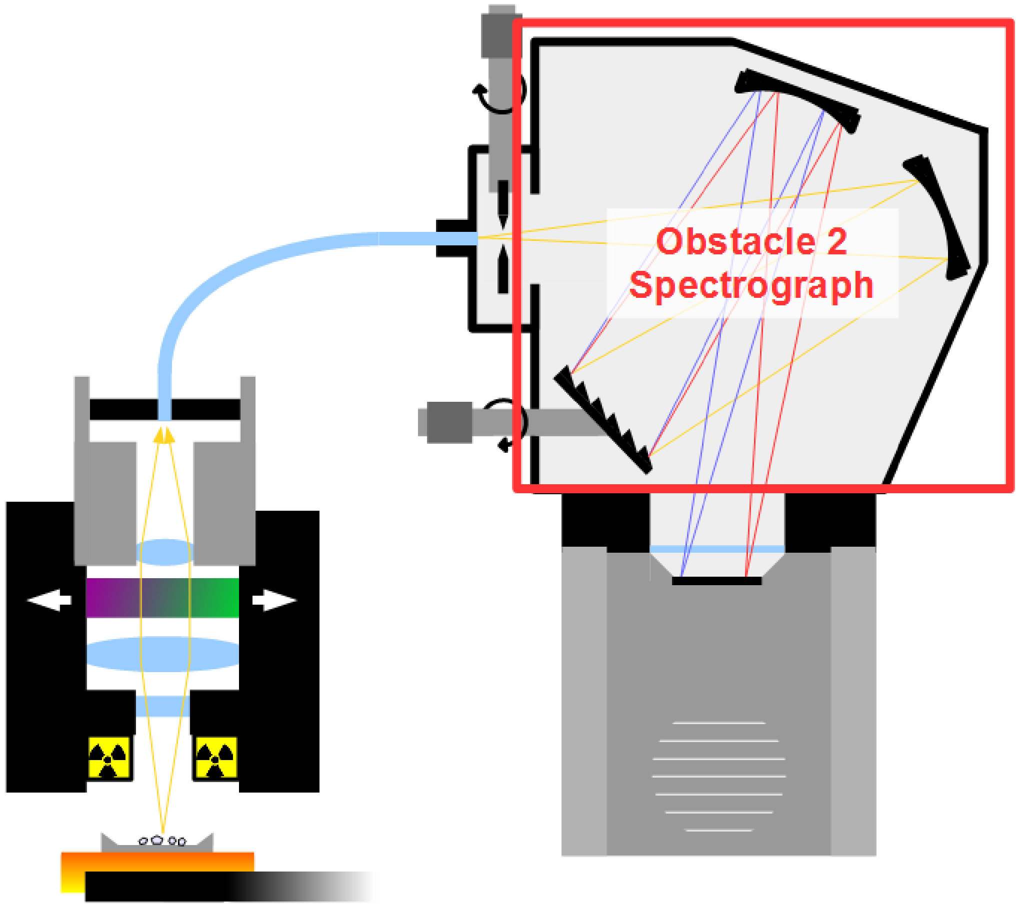
3.2. Obstacle 2: Spectrograph

<u>Issues</u>

1. Diffraction efficiency
2. Signals of higher order
3. Spectral coverage

<u>Ignored here</u>

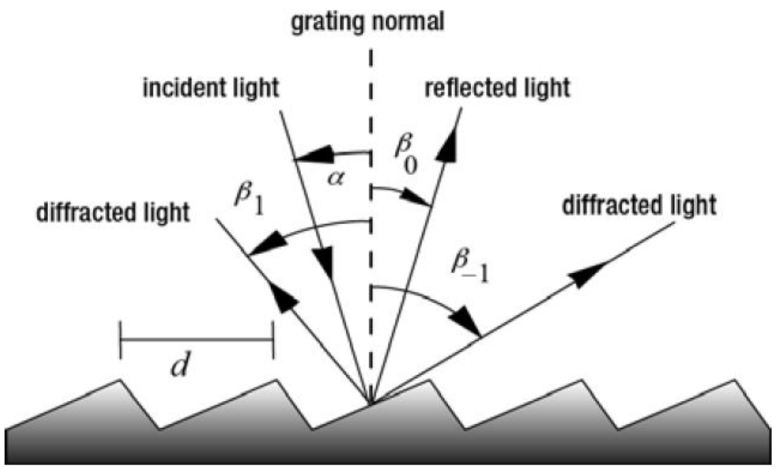
- Slit projection
- Polarization dependence
- Adjustment dependence
- Stray light
- Mirror reflectance
- Mirror degration



3.2.1. Diffraction efficiency

Wavelength seperation by interference effects at a grooved mirror (= grating)

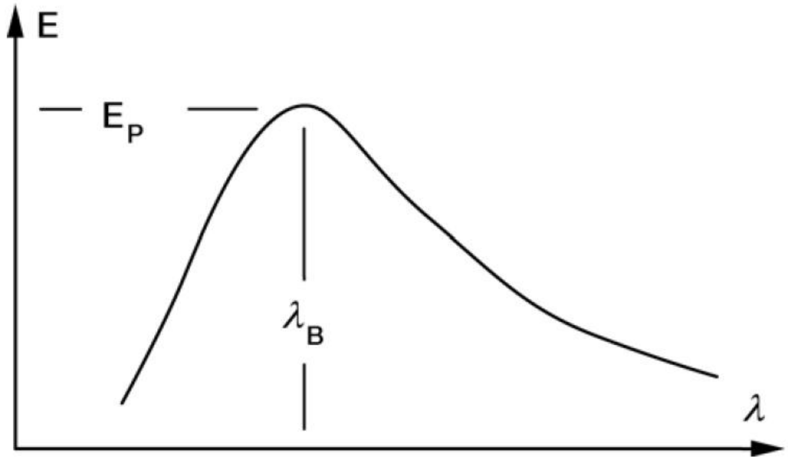
Groove shape and inclination is designed for maximum efficiency at a specfic **blaze wavelength λ_B**



Diffraction by a plane grating
Palmer & Loewen (2014) figure 2-1

<u>Rule of thumb:</u>

efficiency at …
Blaze wavelength ≈ 80 %
Half blaze wavelength ≈ 20 %
Double blaze wavelength ≈ 20 %

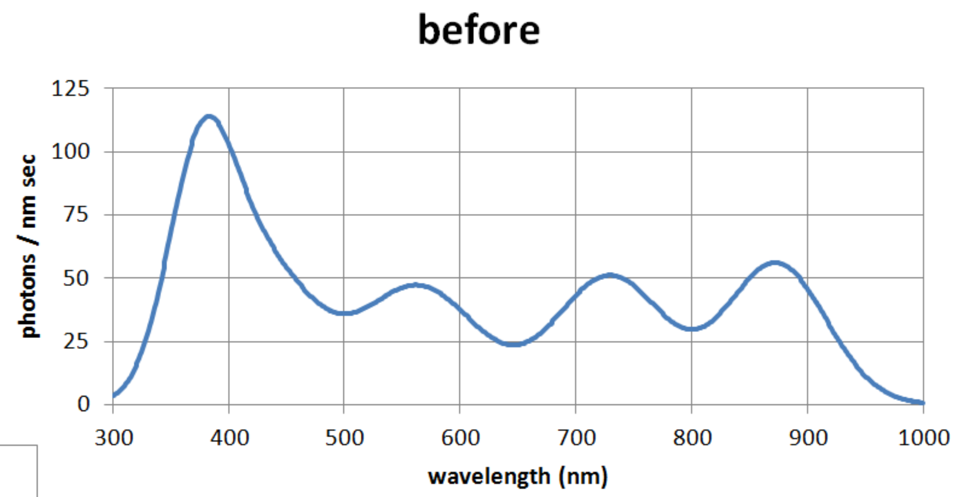


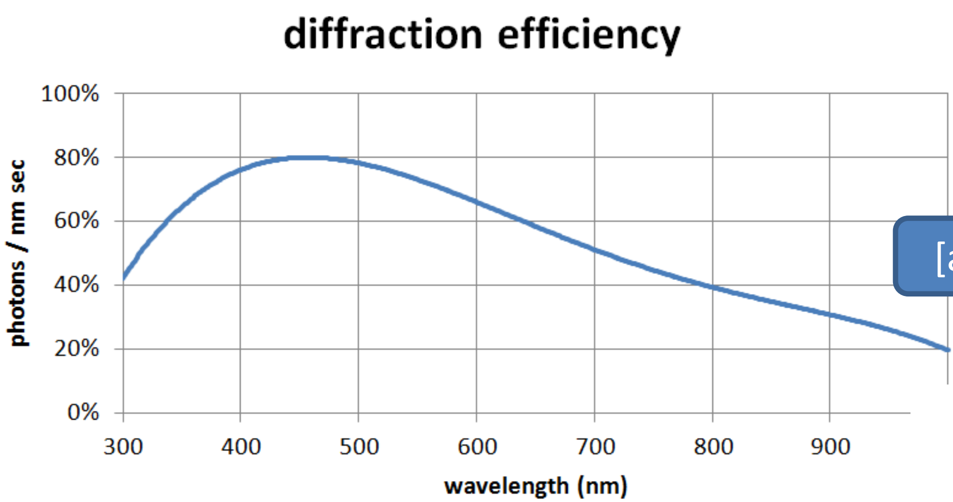
Shematic efficiency over wavelength spectrum
Palmer & Loewen (2014) figure 9-1

Reference:
Palmer, C., Loewen, E., 2014. Diffraction Grating Handbook, 7. ed. Richardson Gratings.

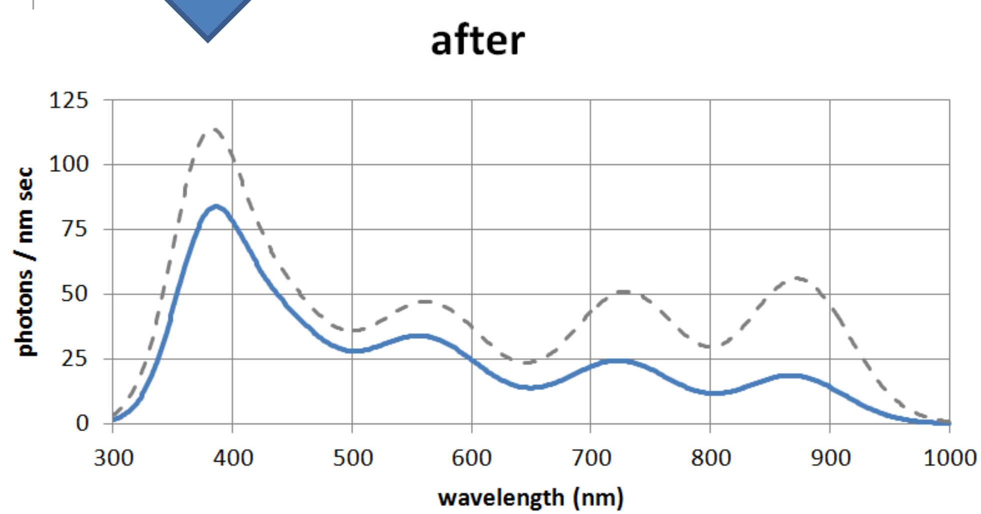
3.2.1. Diffraction efficiency

Standard grating
in lexsyg research: „300/**500**“
→ <u>Blaze wavelength = 500 nm</u>





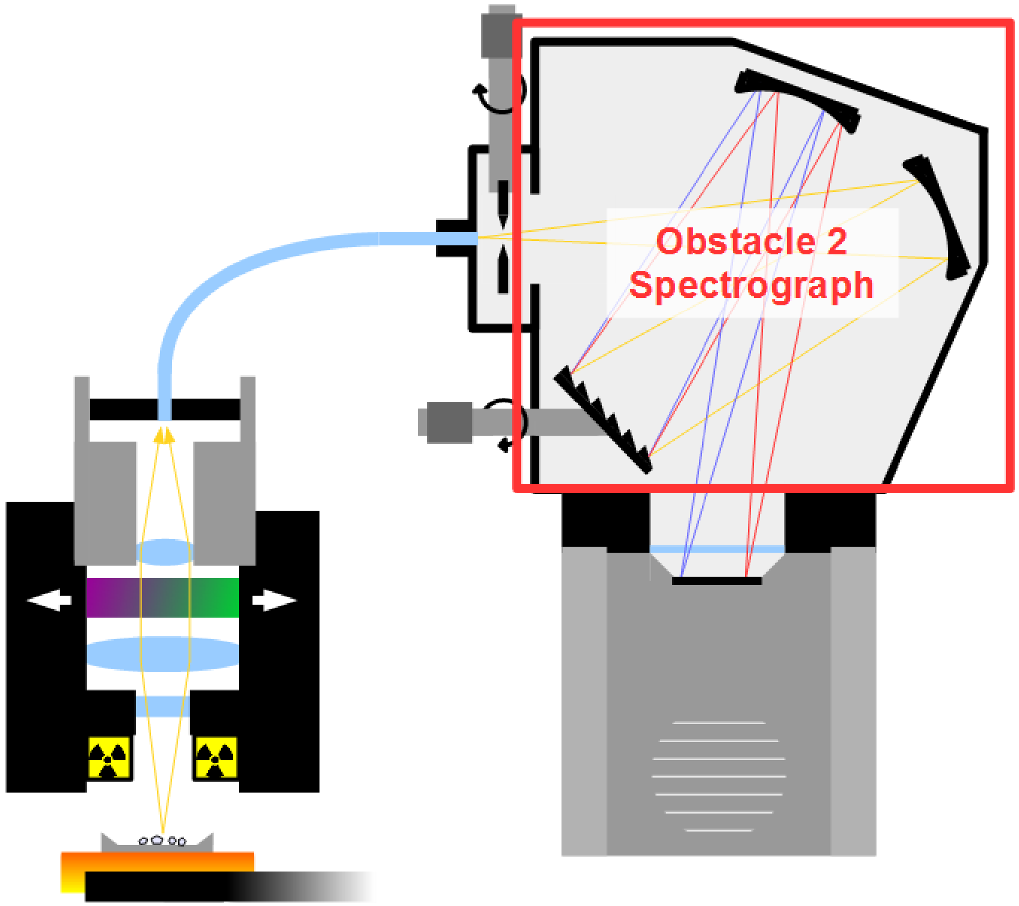
[after] = [before] * [diffraction efficiency]



3.2. Obstacle 2: Spectrograph

Issues

1. ~~Diffraction efficiency~~ → up to 80 % signal loss, towards the borders of the specified wavelength range
2. **Signals of higher order**
3. Spectral coverage



3.2.2. Diffraction order

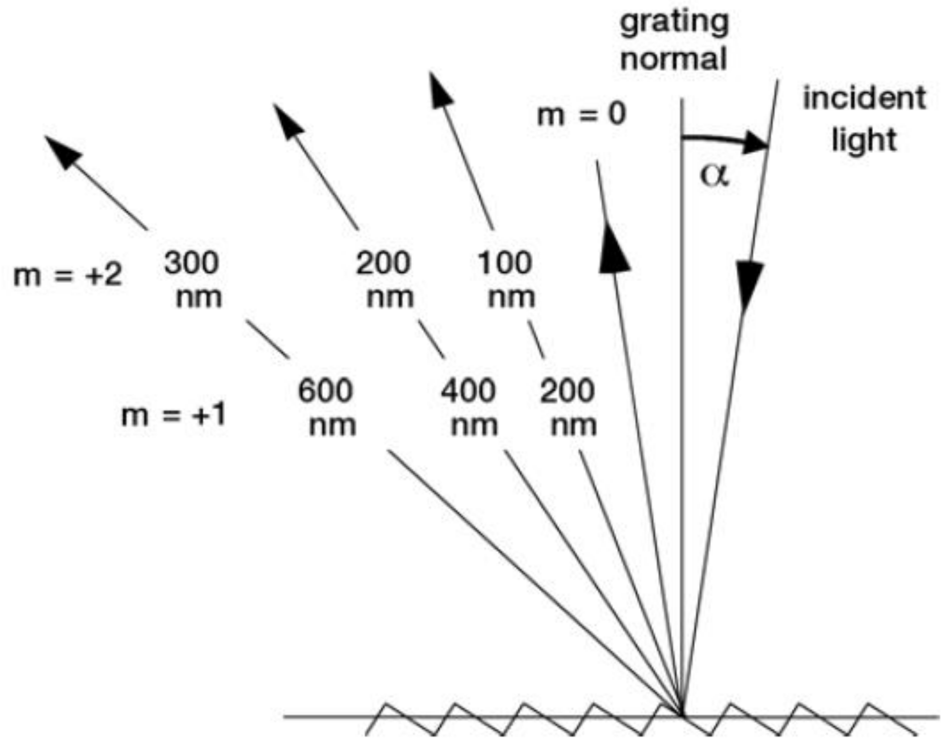
Gratings diffract light into multiple orders, which lead to overlapping spectra

Rule of thumb:

Any signal appears again, at twice the wavelength, with a few % intensity

Solution: Longpass interference filters block signal light with shorter wavelengths than of interest

Warning: A grating inefficient in first order diffraction at a certain wavelength, may be efficient in second (or higher) order diffraction

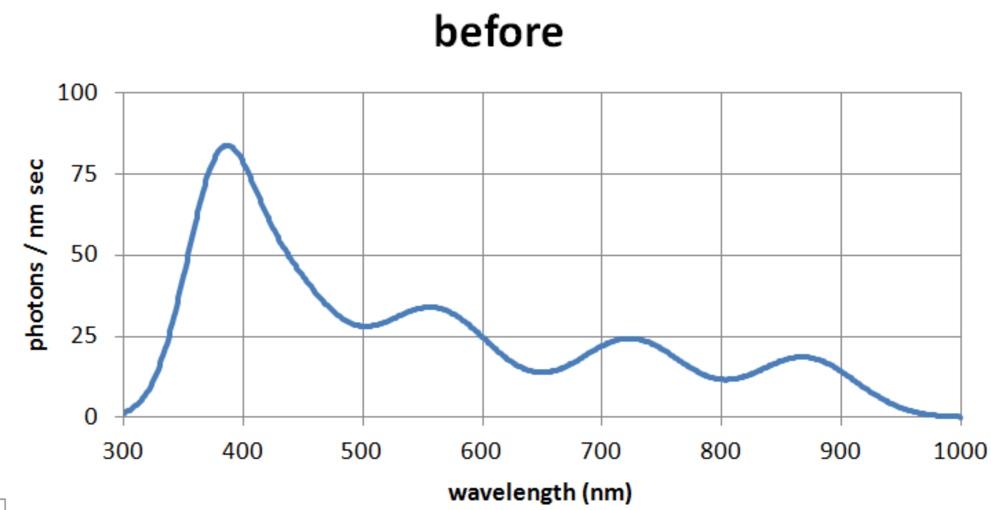


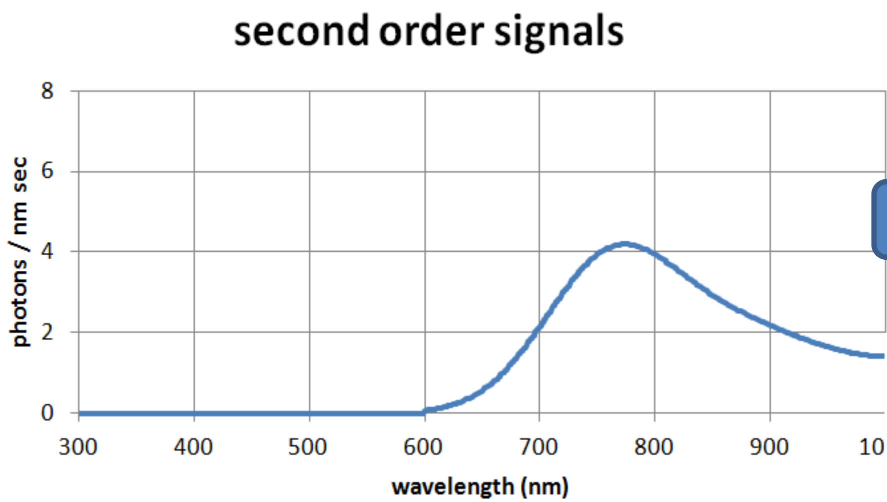
Overlapping spectral orders
Palmer & Loewen (2014) figure 2-5

Reference:
Palmer, C., Loewen, E., 2014. Diffraction Grating Handbook, 7. ed. Richardson Gratings.

3.2.2. Diffraction order

Rough (over-)estimation here:
Second order signals have 10 % intensity (integral value) or 5 % signal height of first order signals

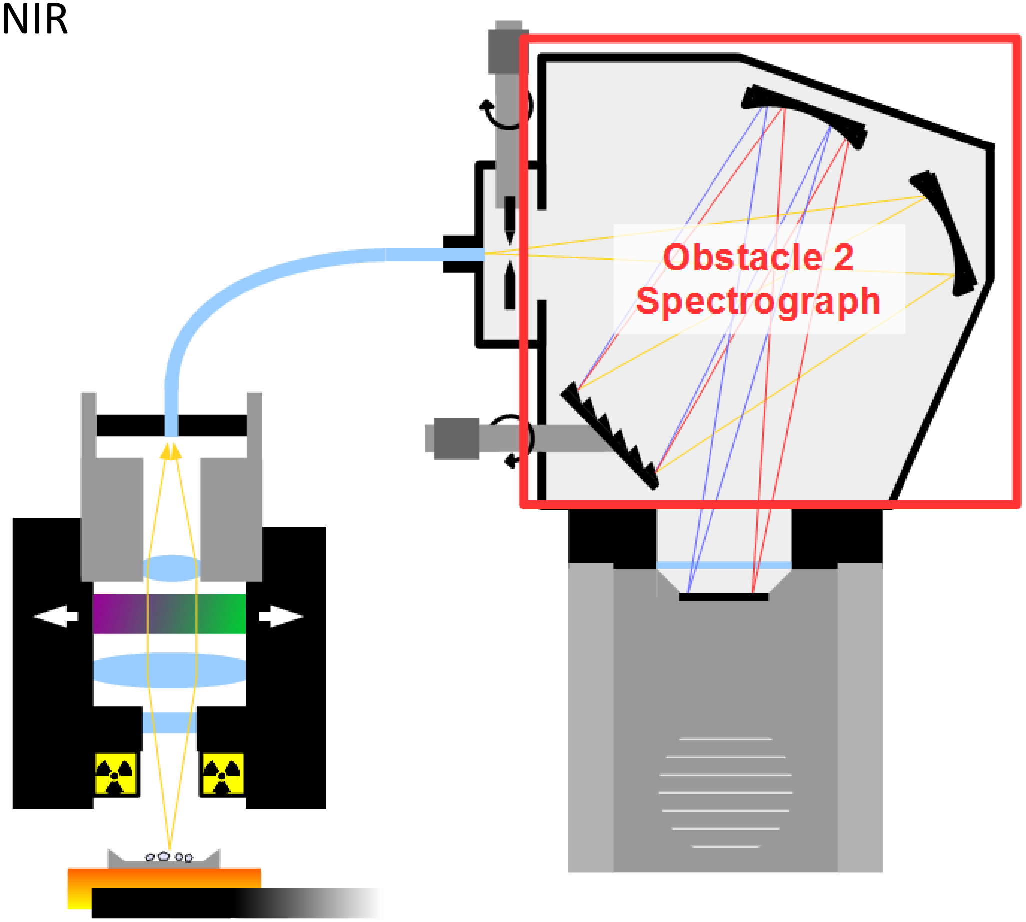




3.2. Obstacle 2: Spectrograph

Issues

1. ~~Diffraction efficiency~~ → up to 80 % signal loss, towards the borders of the specified wavelength range
2. ~~Signals of higher order~~
   → may add ‚ghost' signals in red-NIR
3. **Spectral coverage**



3.2.3. Groove density

Groove density (l/mm)

Grating type: $\boxed{300}$/$\boxed{500}$

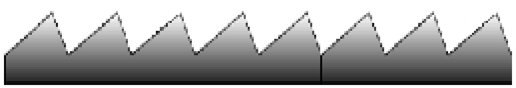
Blaze wavelength (nm)

The groove densitiy (ruling) determines the angular spreading of the spectrum and therefore the **spectral coverage**

Rule of thumb:

$$\Delta\lambda \approx 10^6 \frac{W_D}{f \cdot G}$$

$\Delta\lambda$	spectral coverage (nm)
W_D	width CCD chip (mm) usually: $W_D \approx 26$ mm
f	focal length spectrograph Andor SR-163: $f = 163$ mm
G	groove density (lines/mm)

Grating type	**Ruling sheme**	**Spectral coverage**	**Signal-to-noise ratio***	**Resolution****
600/X		~ 265 nm	~ 70 %	~ 8 nm
300/X		~ 530 nm	100 %	~ 16 nm
150/X		~ 1060 nm	~ 140 %	~ 32 nm

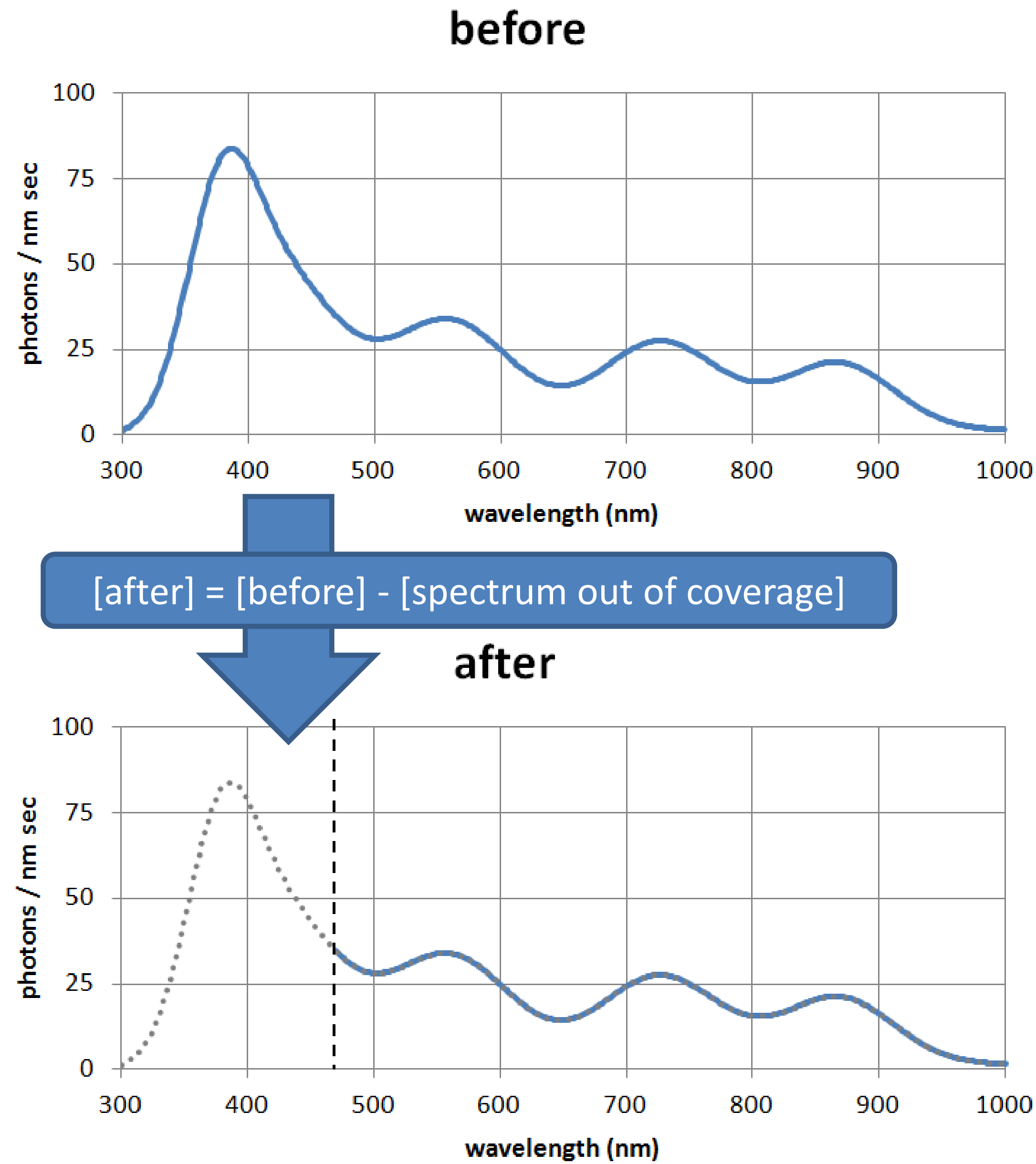
***per CCD pixel column of raw data**

****slit projection dependent; Here: lexsyg Gießen at fully opened slit**

3.2.3. Groove density

First order signals out of spectral coverage range become invisible, but their second order signals remain

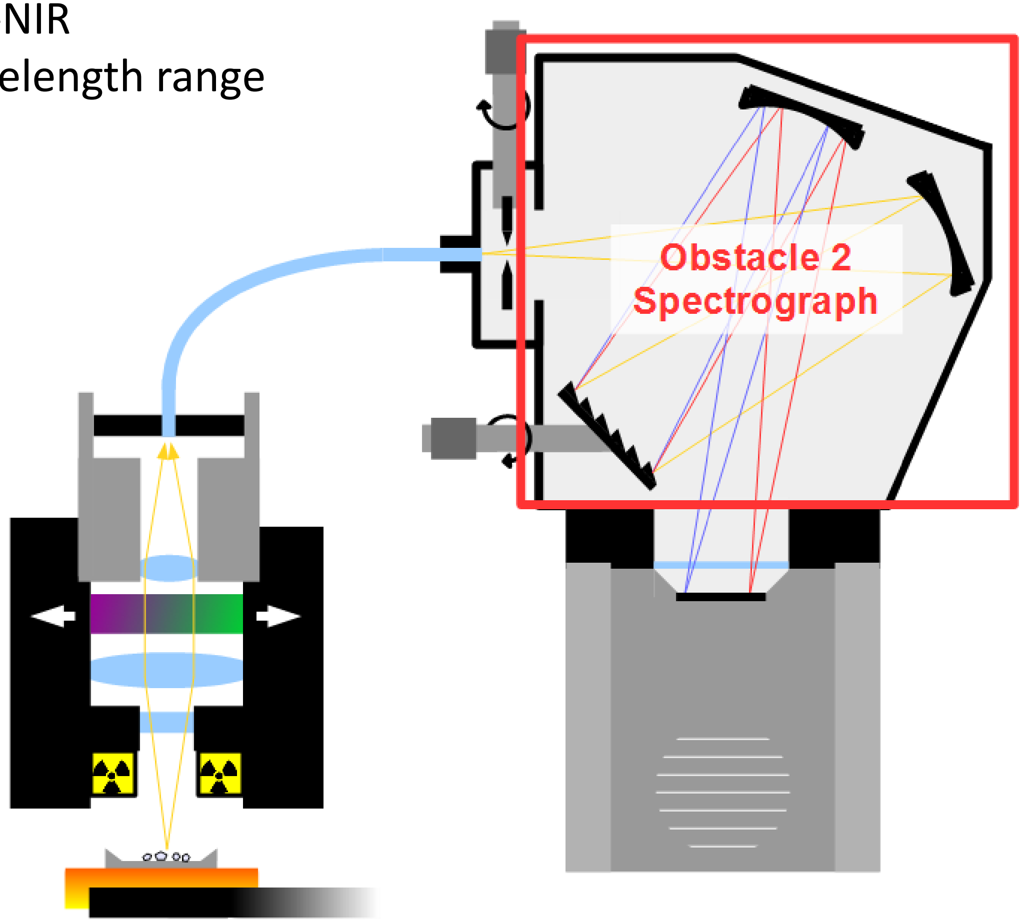
Example: 300/500 grating sets wavelength range to 530 nm; spectrograph is adjusted to 470 - 1000 nm



3.2. Obstacle 2: Spectrograph

Issues

1. ~~Diffraction efficiency~~ → up to 80 % signal loss, towards the borders of the specified wavelength range
2. ~~Signals of higher order~~
   → may add a ‚ghost' signal in red-NIR
3. ~~Spectral coverage~~ → restricts wavelength range



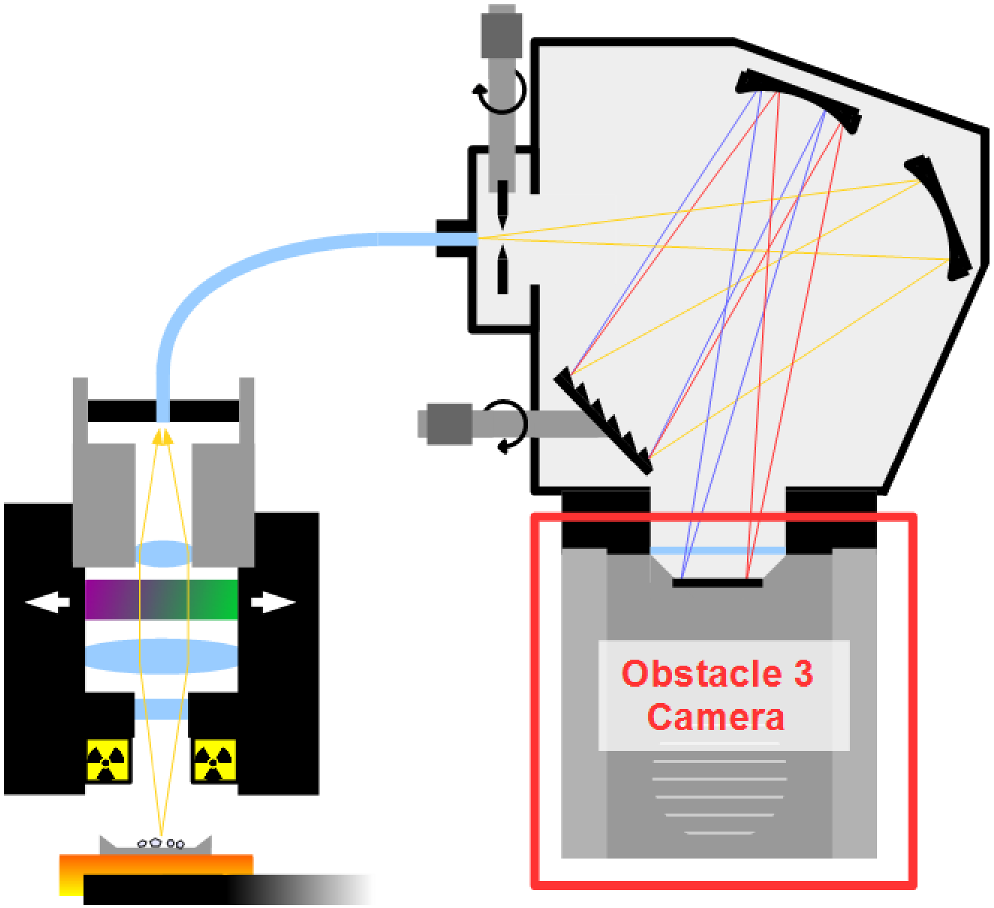
3.3. Obstacle 3: Camera

Issues

1. **Quantum efficiency**
2. ‚Cosmic‘ rays
3. Noise and offset

Ignored here

- Etaloning
- Setting dependence
- Gain & conversion rate
- Digitization
- Clock induced charge noise
- Detector dead time

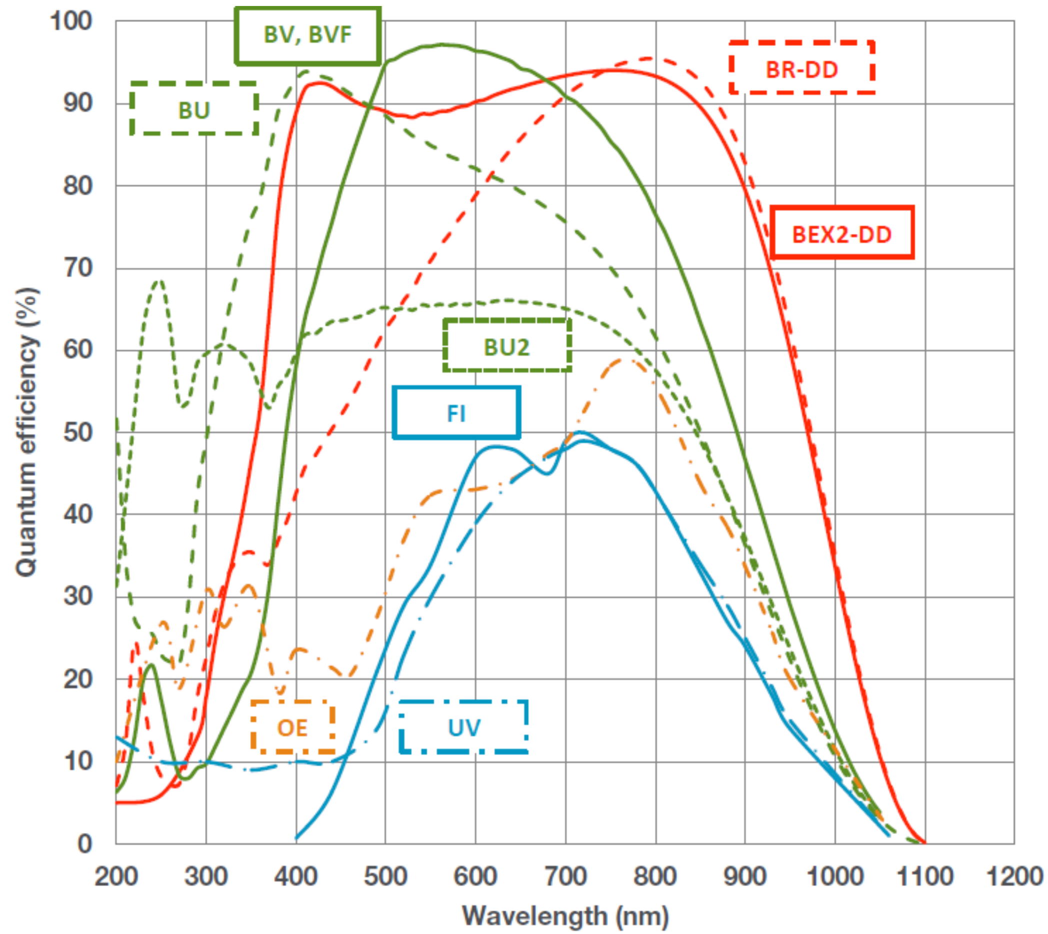


3.3.1. Quantum efficiency

Not all incoming photons at the CCD chip are converted into measureable **photoelectrons**

Quantum efficiency relies on CCD chip parameters:

- Back-illuminated or front-illuminated or open electrode?
- UV-enhanced (coated)?
- Deep depletion?

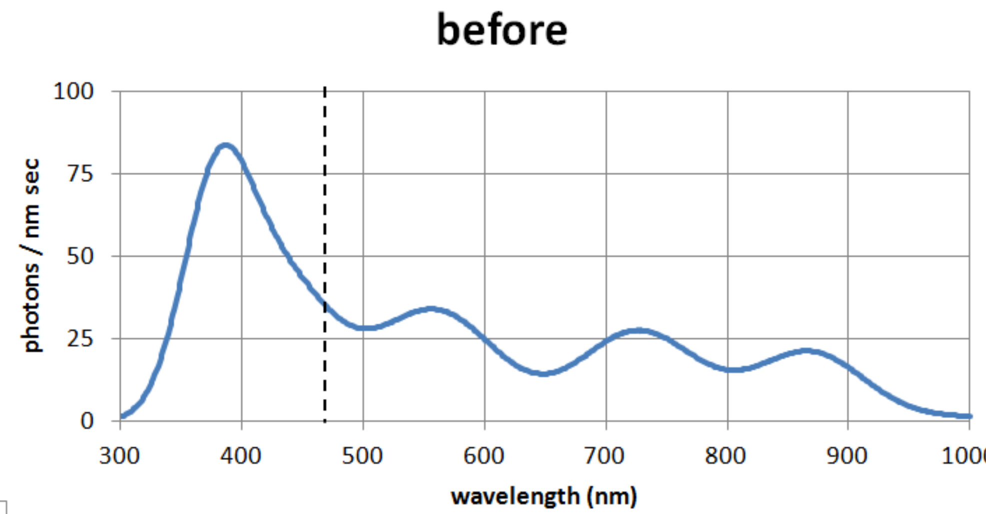


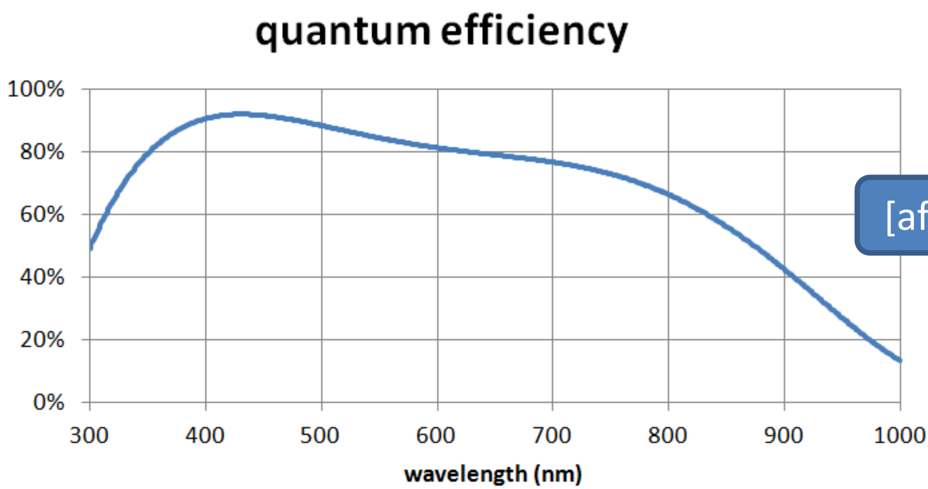
Quantum efficiency spectra of CCD chips available for Andor Newton camera
Source: Andor Newton specifications (08/2015)

3.3.1. Quantum efficiency

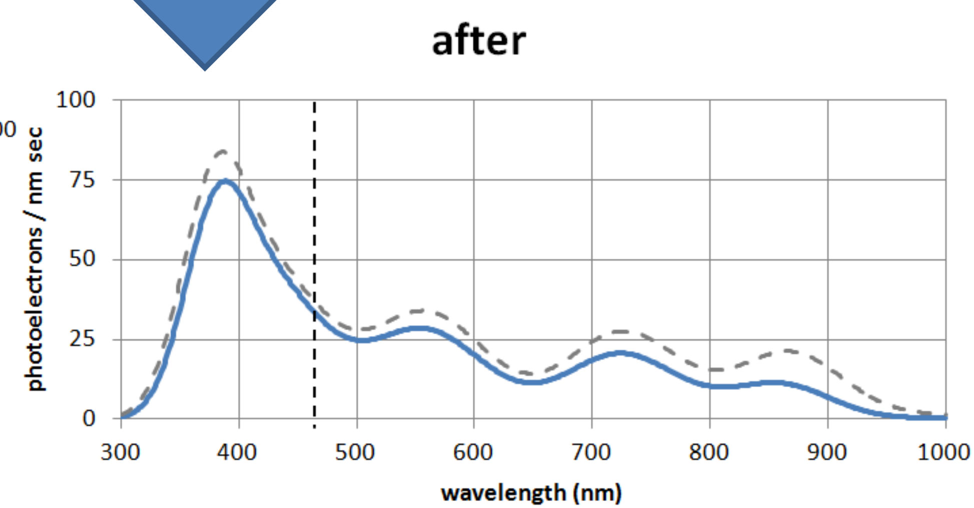
QE curve here:
Andor Newton DU920P-BU (Gießen)

Note: second order signals apply to their (first order) wavelength QE, not to their position on the detector





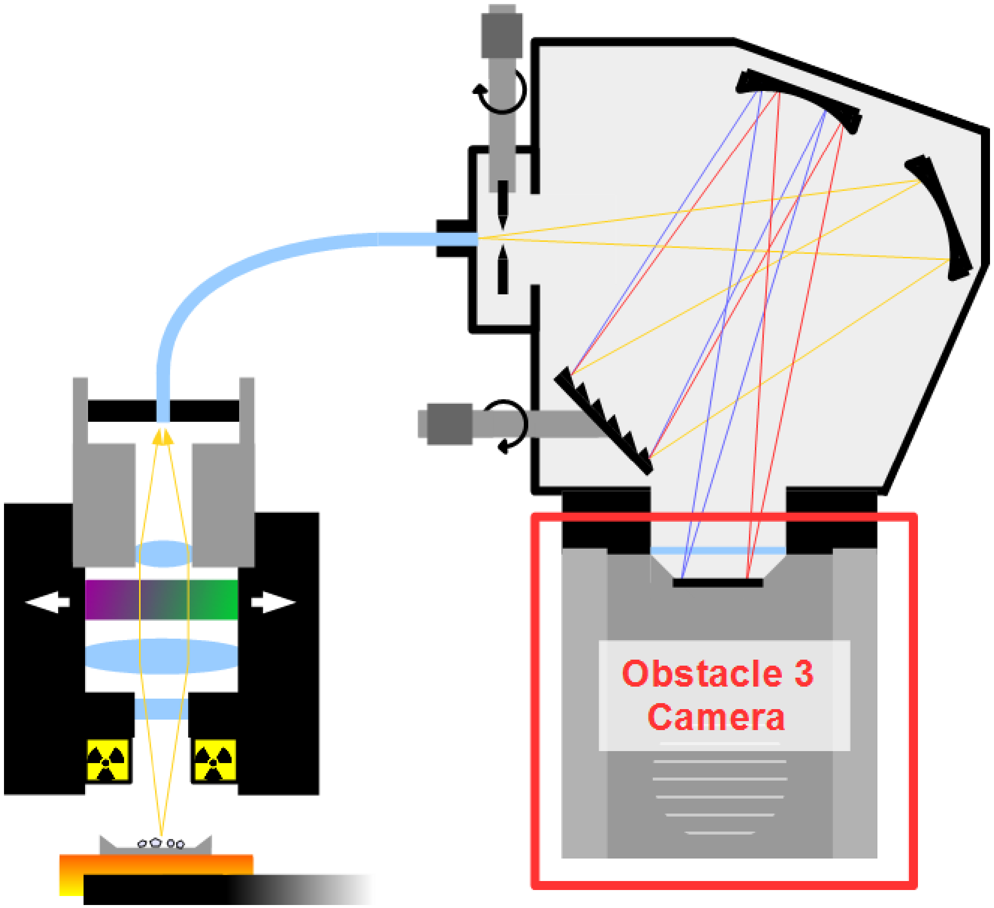
[after] = [before] * [quantum efficiency]



3.3. Obstacle 3: Camera

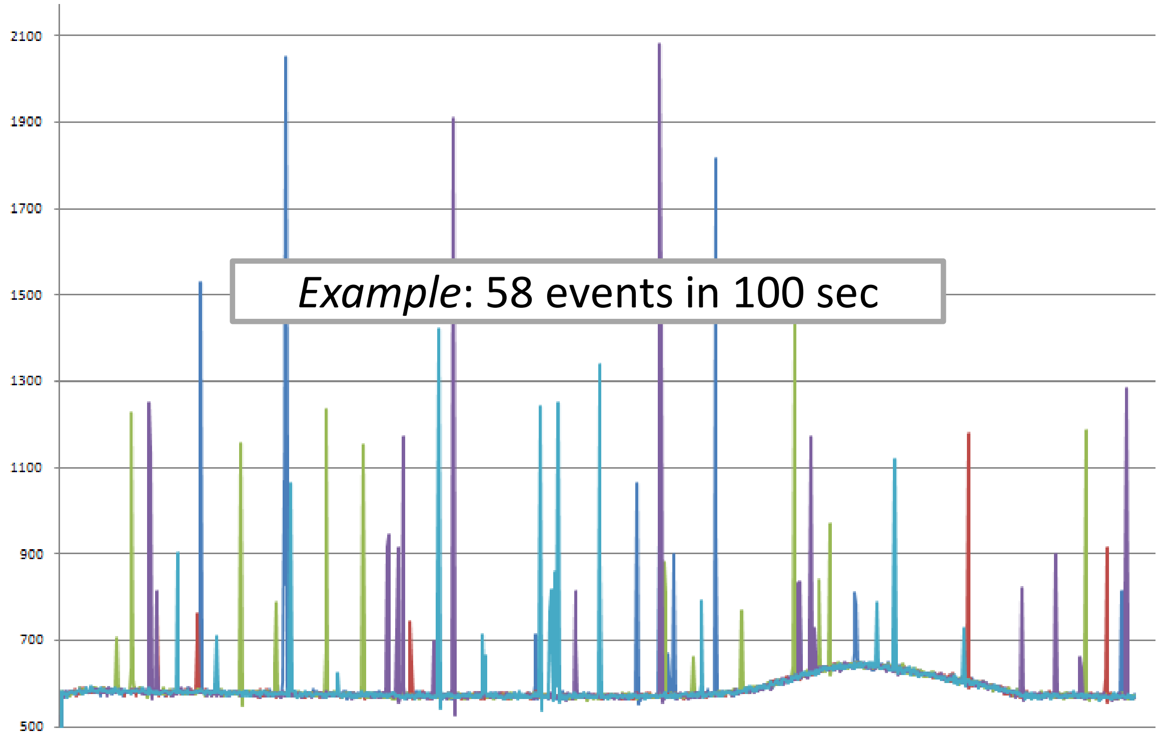
Issues

1. ~~Quantum efficiency~~ → wavelength dependent signal loss
2. **‚Cosmic‘ rays**
3. Noise and offset



3.3.2. Cosmic rays

‚Cosmic' ray events at RF are (mostly) caused by β- and γ-impacts at the CCD chip



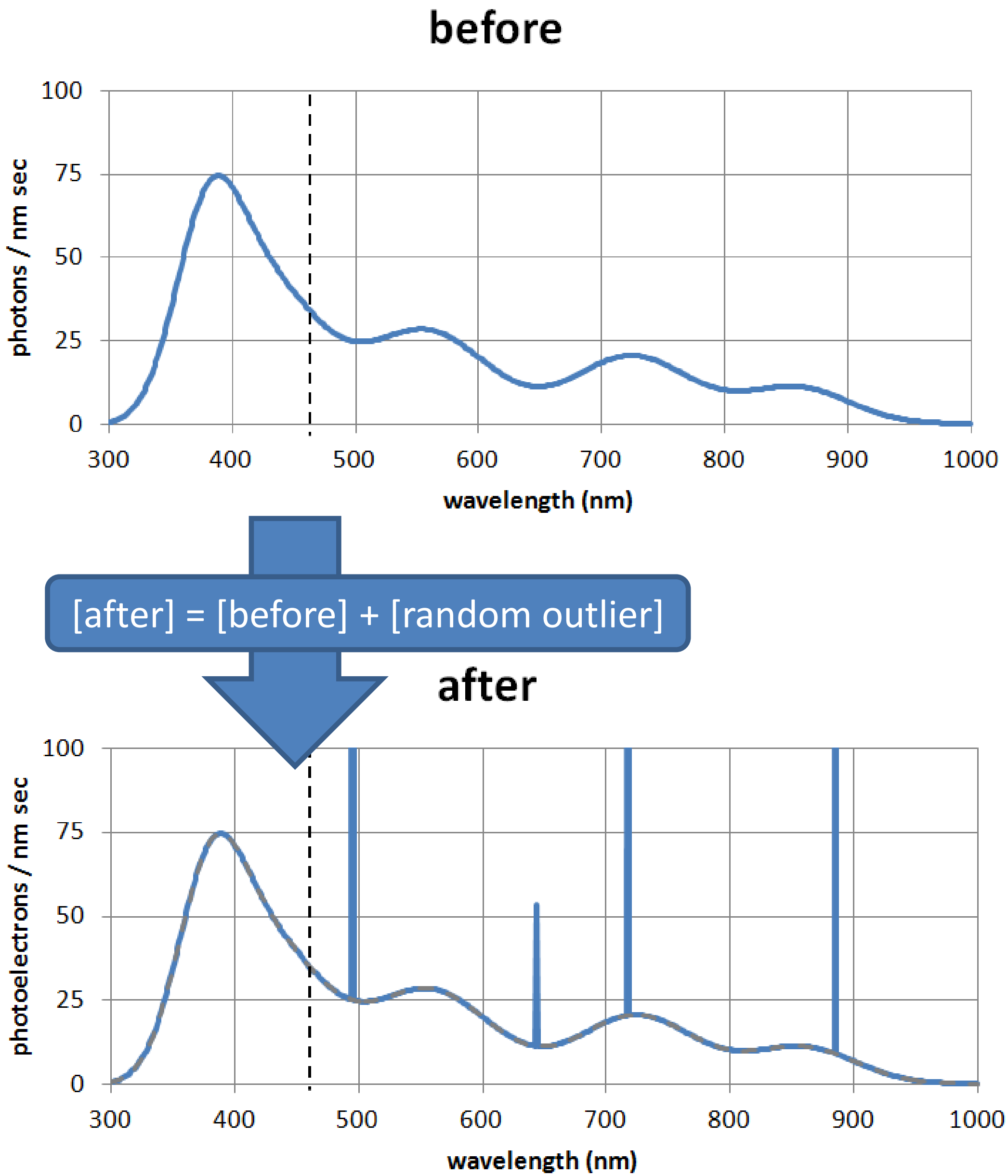
Five IR-RF spectra, 20 sec exposure time each.
Taken 2014 with lexsyg Gießen

<u>Rules of thumb:</u>

- Mostly, just one or two datapoints per event are affected
- Shielding or increased distance β- source to CCD decreases event rate

3.3.2. Cosmic rays

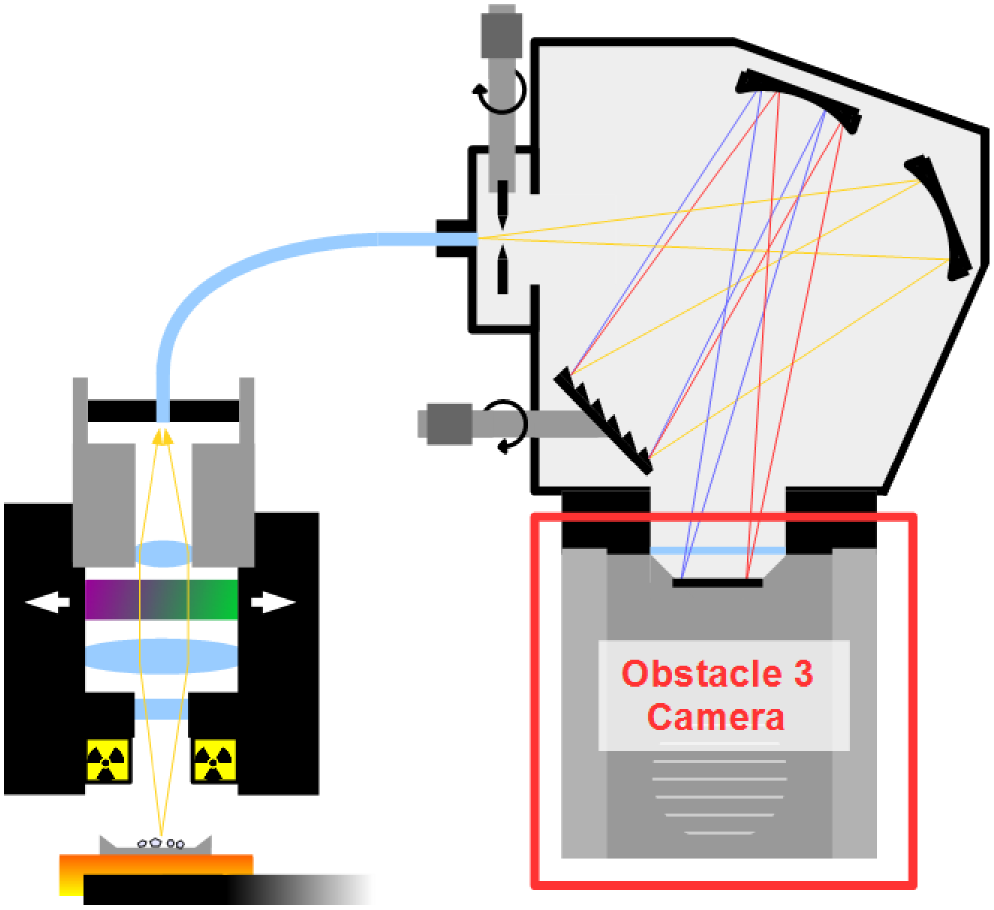
We add 4 random events:



3.3. Obstacle 3: Camera

Issues

~~1. Quantum efficiency~~ → wavelength dependent signal loss
~~2. ‚Cosmic‘ rays~~ → some random sharp peaks are added
3. Noise and offset



3.3.3. Noise and offset

Camera noise:
Standard deviation of one single superpixel*:

$$\sigma = \sqrt{\sigma_{shot}^2 + \sigma_{dark}^2 + \sigma_{read}^2}$$

*Superpixel: Collection of pixels read out at once
**Ratio superpixel value/data value (‚counts‘) depends on *Gain* and *Conversion factor*

Sorts of camera noise:

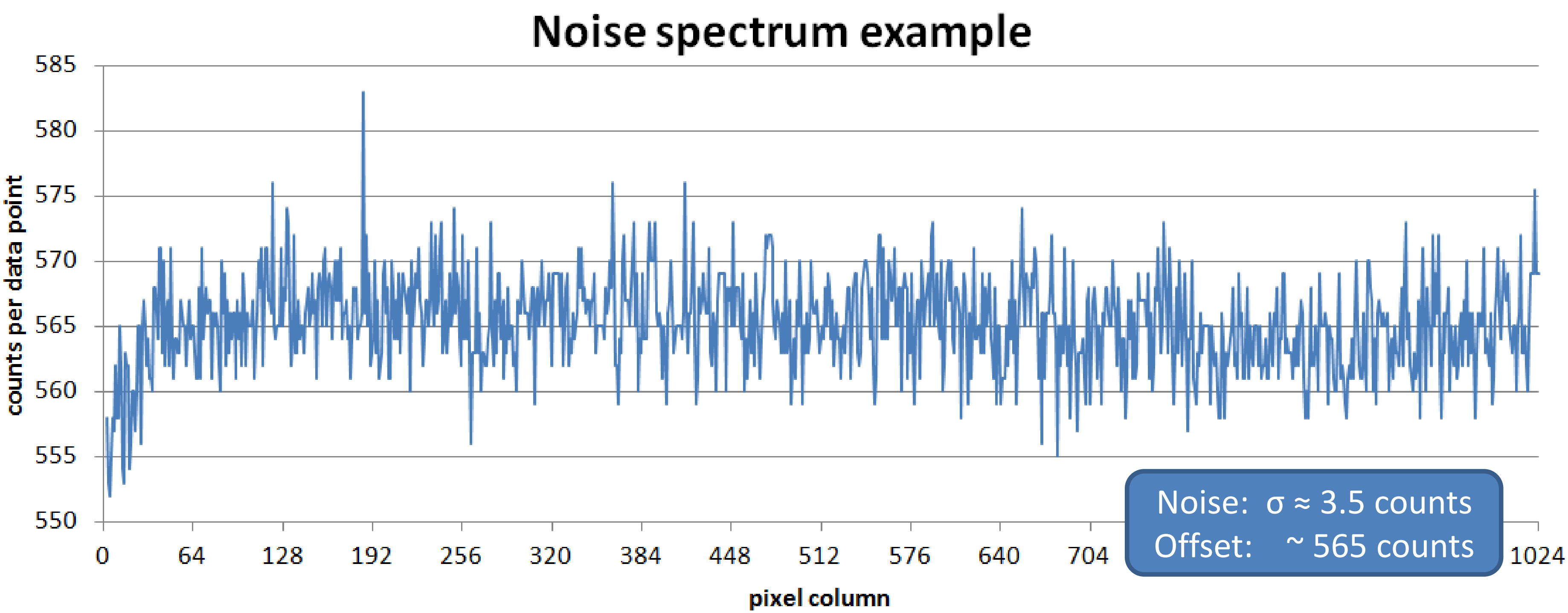
- **Shot noise**
  Statistical uncertainty of number of photoelectrons located in one superpixel
- **Dark current noise**
  Statistical event of the appearance of a thermal electron in one pixel
- **Read out noise**
  Electronic noise added in the event of superpixel read out

Noise type	Formula*	Depend on	Example value**
Shot	$\sigma_{shot} = \sqrt{I}$	signal height *I*	-
Dark	$\sigma_{dark} = \sqrt{N\,\phi_{dark}\Delta t}$	chip temperature, superpixel size *N*, exposure time *Δt*	$0.9\ e^-$ (Δt = 1 sec) $3.9\ e^-$ (Δt = 20 sec)
Read	values see: *certificate of performance*	camera type & setting	~ $4\ e^-$

*** *I* – number of e- per superpixel; *N* – number of pixels per superpixel (usually N = 255 = one column);**
φ_{dark} - dark current in e-/pixel sec, highly chip temperature dependent
****Camera Gießen (Andor Newton DU920P-BU) at -70°C chip temperature, 50 kHz read out rate, default settings**

3.3.3. Noise and offset

Camera noise is approximately Gauss distributed
To avoid negative data values an **offset value** is added
This offset value depends on the camera settings

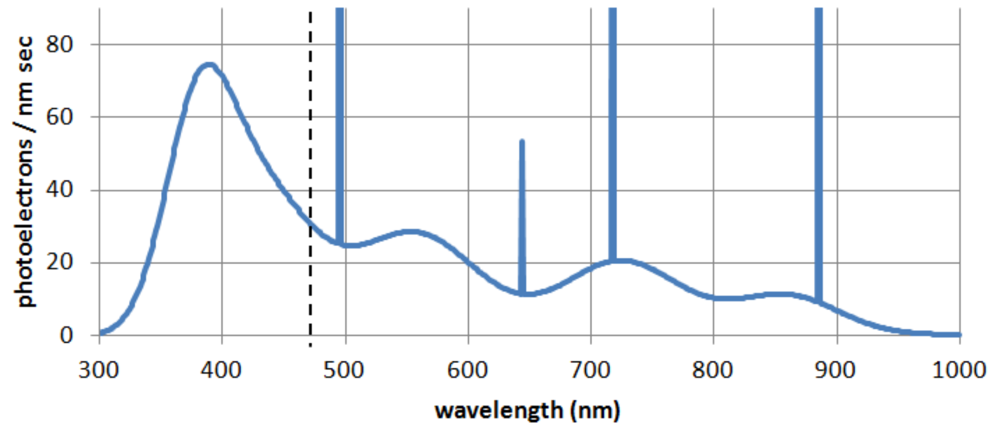


Taken with camera Gießen (Andor Newton DU920P-BU) at closed shutter, 20 sec exposure time, -70°C chip temperature, 50 kHz read out rate, default settings; Data cleaned for cosmic rays

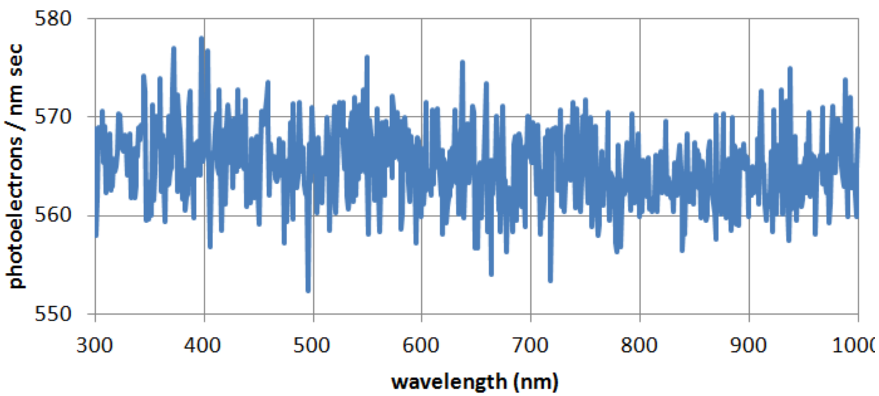
3.3.3. Noise and offset

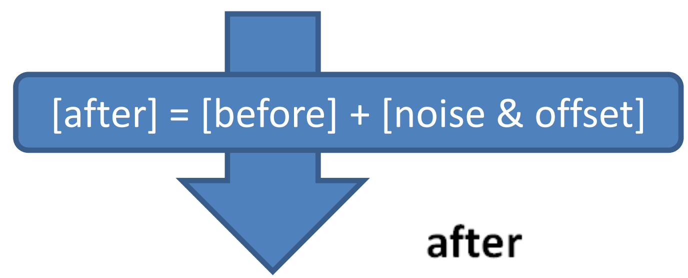
Adding noise spectrum from last slide plus shot noise

before

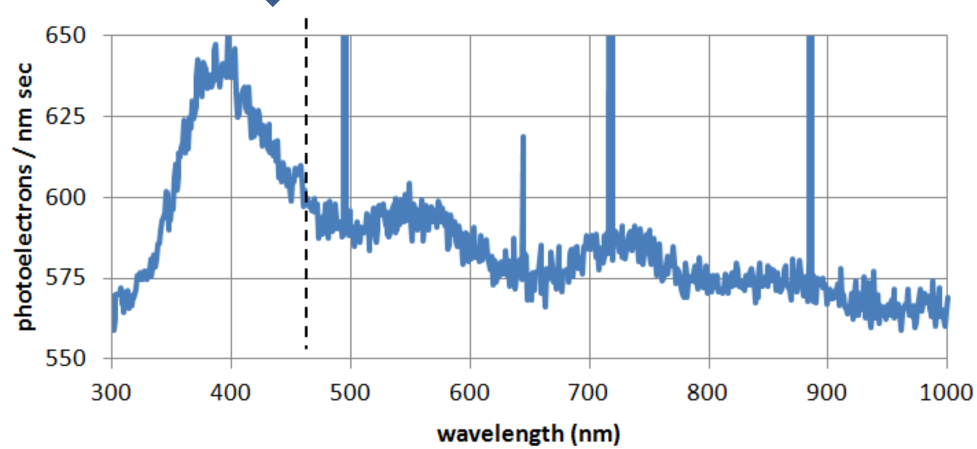


noise and offset





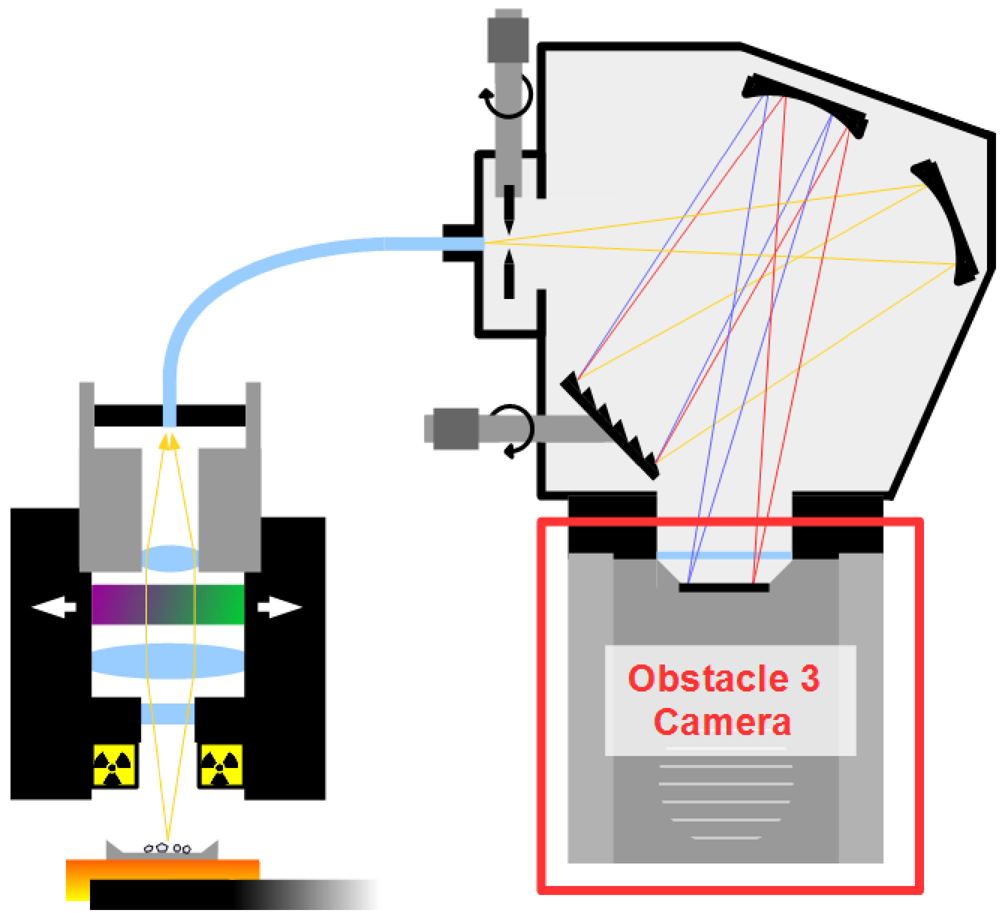
after



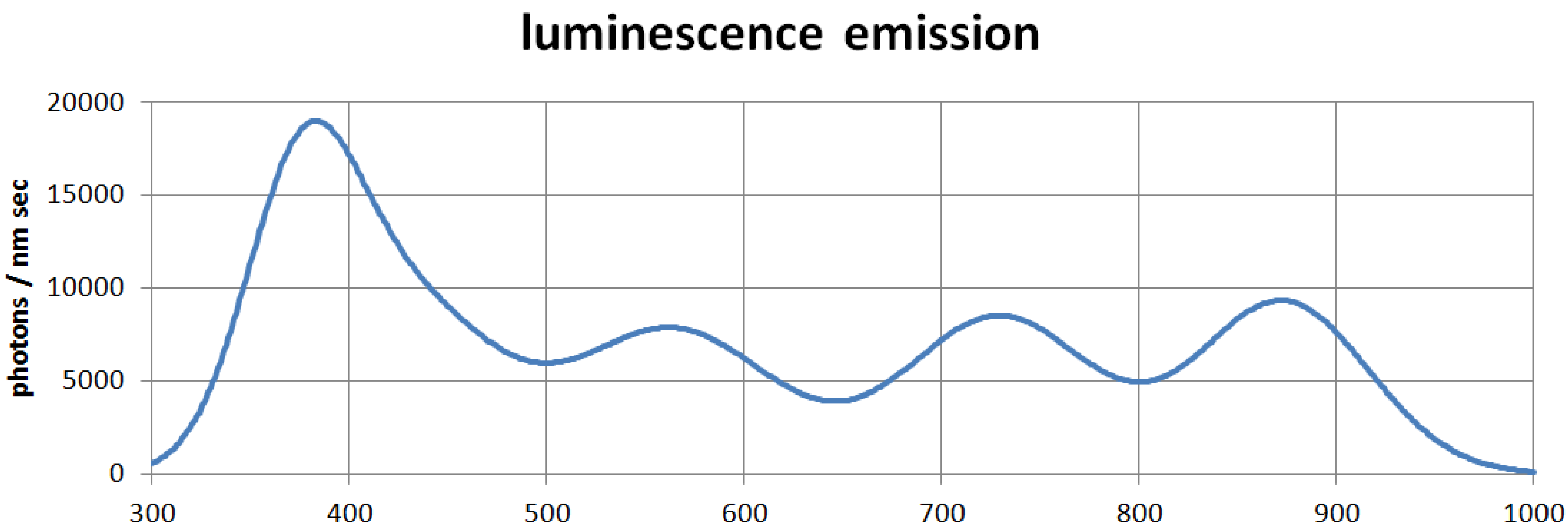
3.3. Obstacle 3: Camera

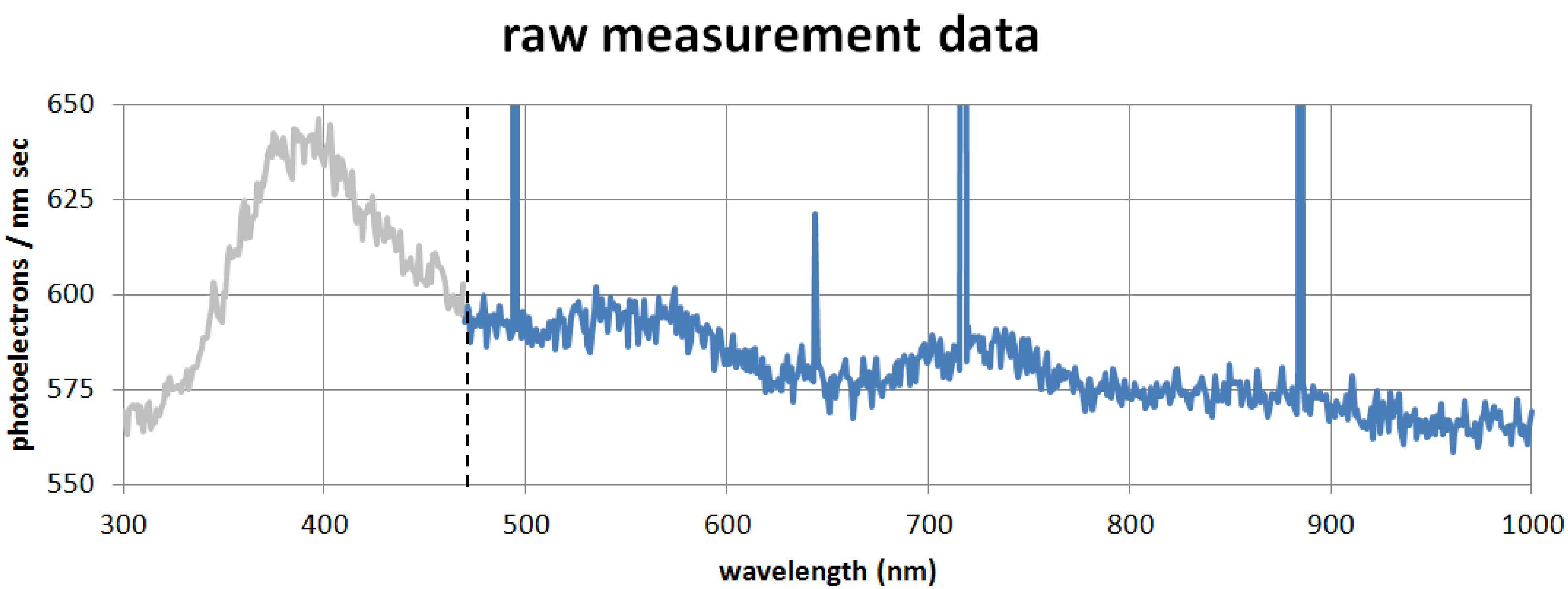
Issues

1. ~~Quantum efficiency~~ → wavelength dependent signal loss
2. ~~‚Cosmic‘ rays~~ → some random sharp peaks are added
3. ~~Noise and offset~~ → may hide weak peaks



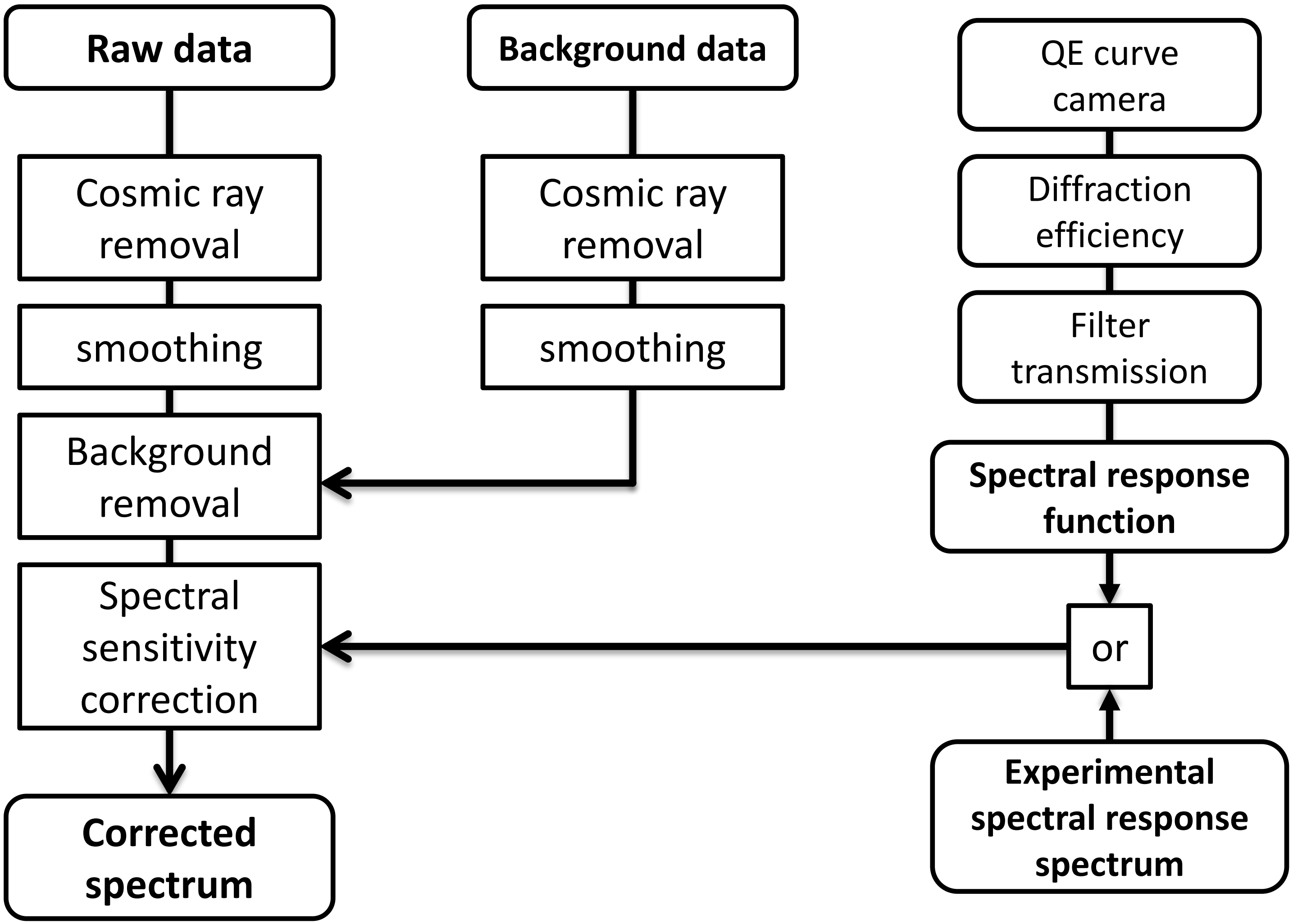
3.4. Comparison Before and After





4. From raw data to luminescence emission

How to transform the distorted measured spectrum back to an adequate luminescence emission spectrum?



4.1. Cosmic ray removal

Ways of removing outlier

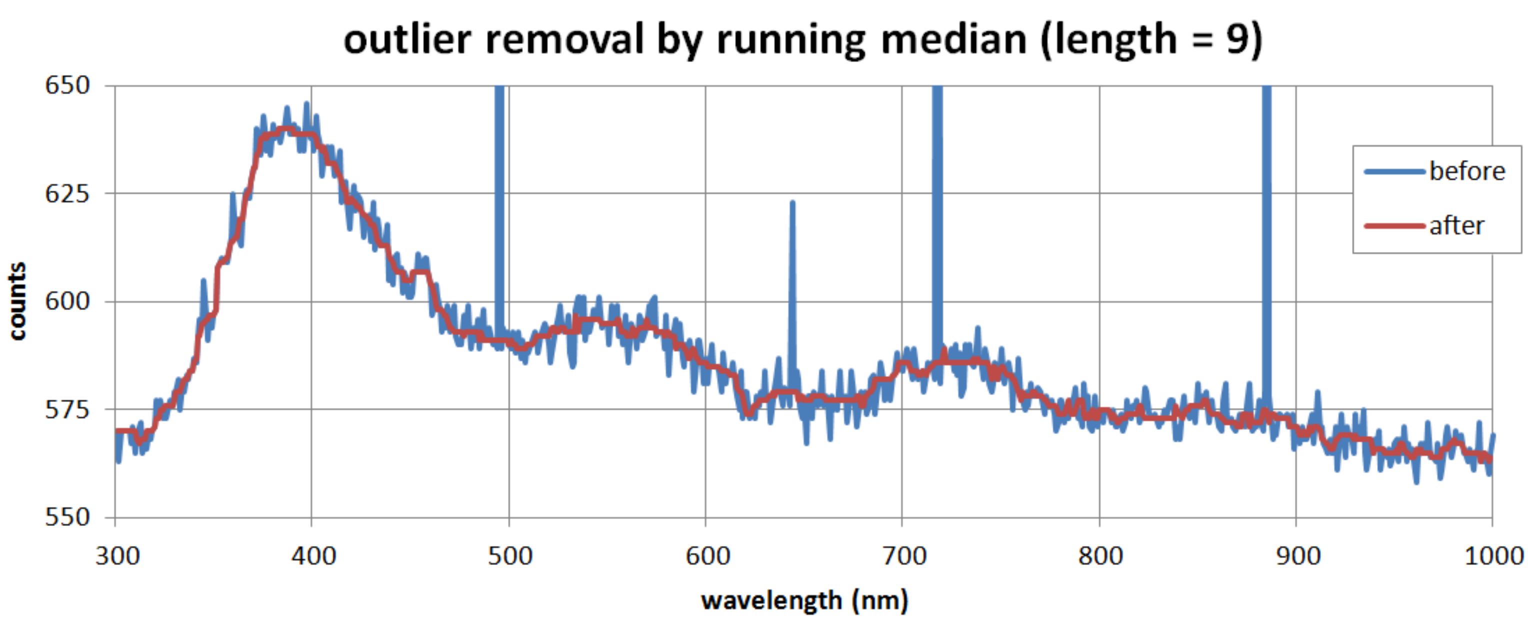
- Iterative histogramm based removal (Pych 2003)
  available in R luminescence package

- differential threshold triggered median
  deletes outlier but keeps the noise if noise analysis for setting optimization is wished

Excel example code: Column C = raw data; C11 = Cell of interest

```
=IF(OR(ABS(C11-C10)>[Threshold];ABS(C11-12)>[Threshold]);MEDIAN(C7:C15);C11)
```

- running median



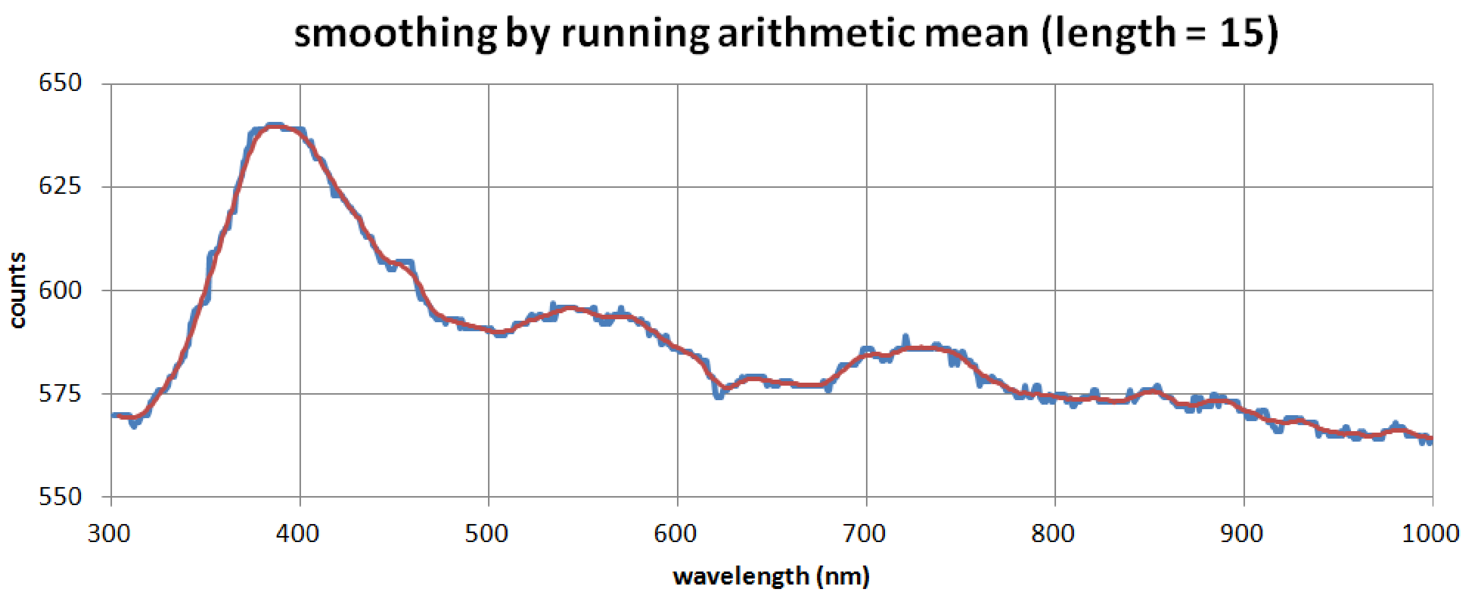
4.2. Smoothing

Ways of data smoothing

- Pixel binning by Hardware
  Sums signals without increasing read out noise (almost). Best way of increasing signal-to-noise ratio, but increases impact of cosmic rays. **Recommended at short exposure times**

- Pixel binning by Software
  2 Pixels summed up = 2x signal but only $2^{0.5}$x noise → 40 % SNR win

- Running mean
  Similar as software binning but may flatten peaks if length to high. **Attention**: Some running mean algorithms shift peaks. (Excel running mean fitting)

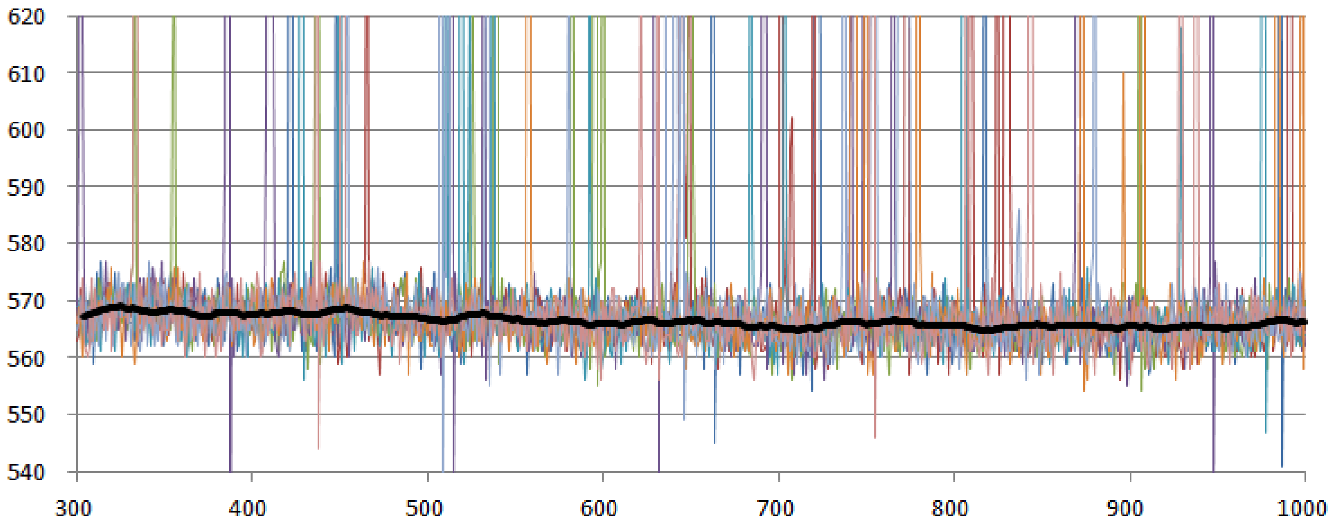


4.3. Background substraction

Background substraction removes camera offset and stem effect

How to obtain a noise-free background spectrum?

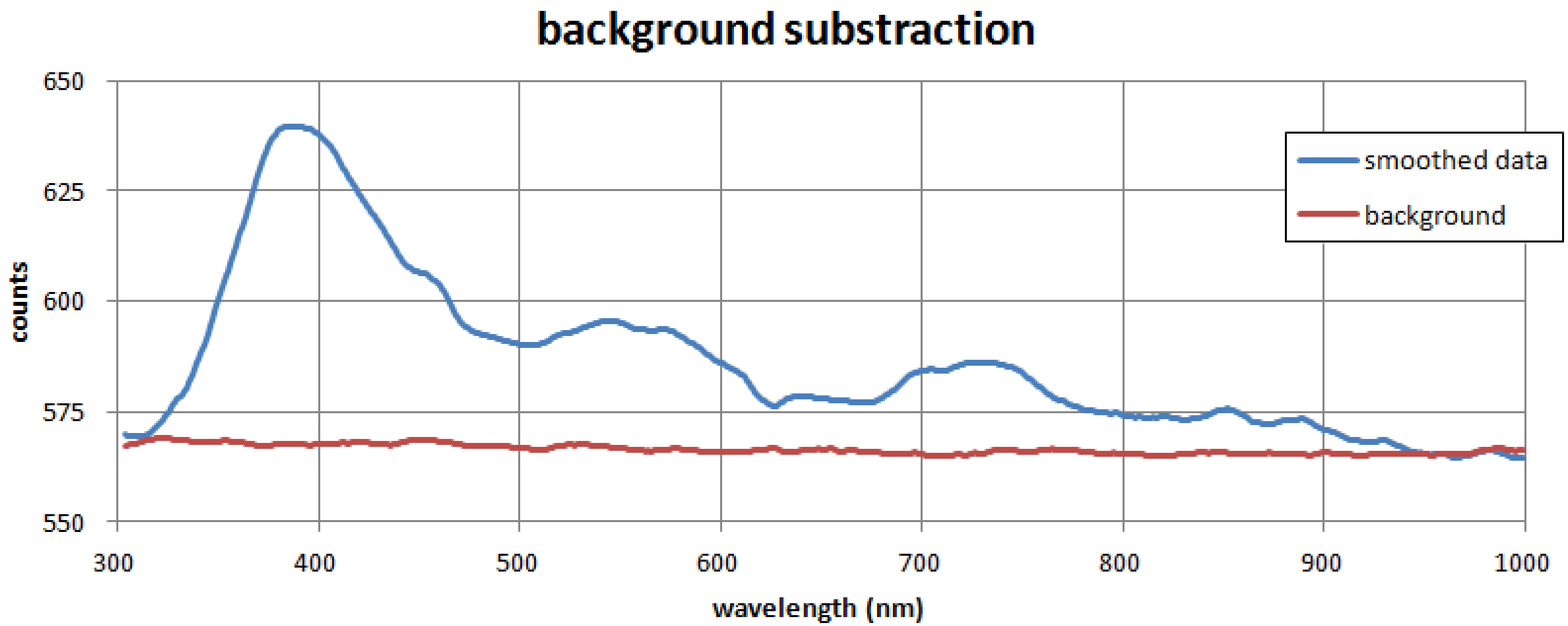
1. Repeat sequence with empty aliquot. Especially camera settings must be exactly the same
2. Outlier removal of obtained spectrum collection by applying running median in direction of wavelength (length = 5 or higher) AND time (length = 6 or higher)
3. Smoothing of every spectrum by running mean (length as for RF data or higher)
4. Check spectrum collection for peak shifting with time. (don‘t worry about static peaks)
   If there is peak shifting → You may have an contaminated aliquot or measurement chamber or a serious problem with the sequence or the setup
5. Create one single mean spectrum from collection = **Background**

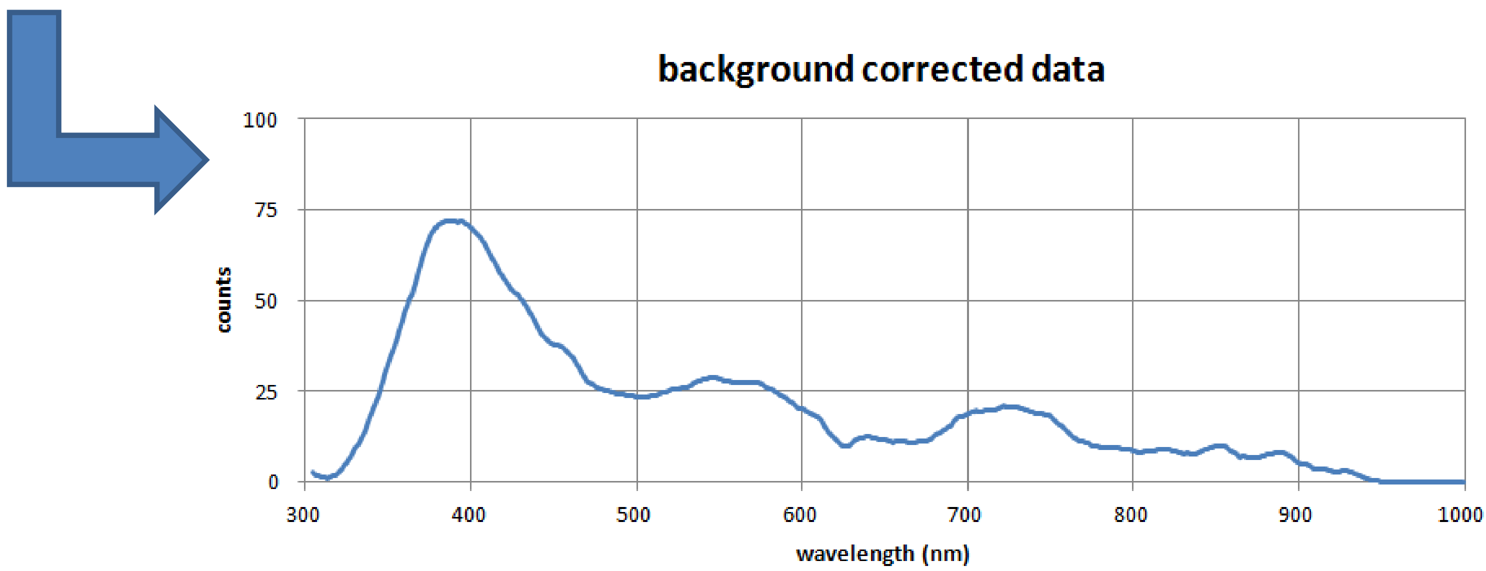


Example data:
Just 8 background spectra with closed beta source availabe

Background creation:
1. Created Running median spectrum (length@time = 8; length@wavelength = 3)
2. Smoothed resulting spectrum (length = 15)

4.3. Background substraction





4.4. Spectral response correction

A **spectral response function** [SRF] describes the wavelength dependend attenuation of the ‚real' luminescence spectrum [LUM] which leads to the measured spectrum [MEAS]:

$$[\mathrm{MEAS}] = [\mathrm{SRF}] \cdot [\mathrm{LUM}]$$

The spectral response function is also the product of all **spectral transfer functions** [STF] which take an effect on the luminescence signal:

$$[\mathrm{SRF}] = [\mathrm{STF}_1] \cdot [\mathrm{STF}_2] \cdot [\mathrm{STF}_3] \cdot \ldots$$

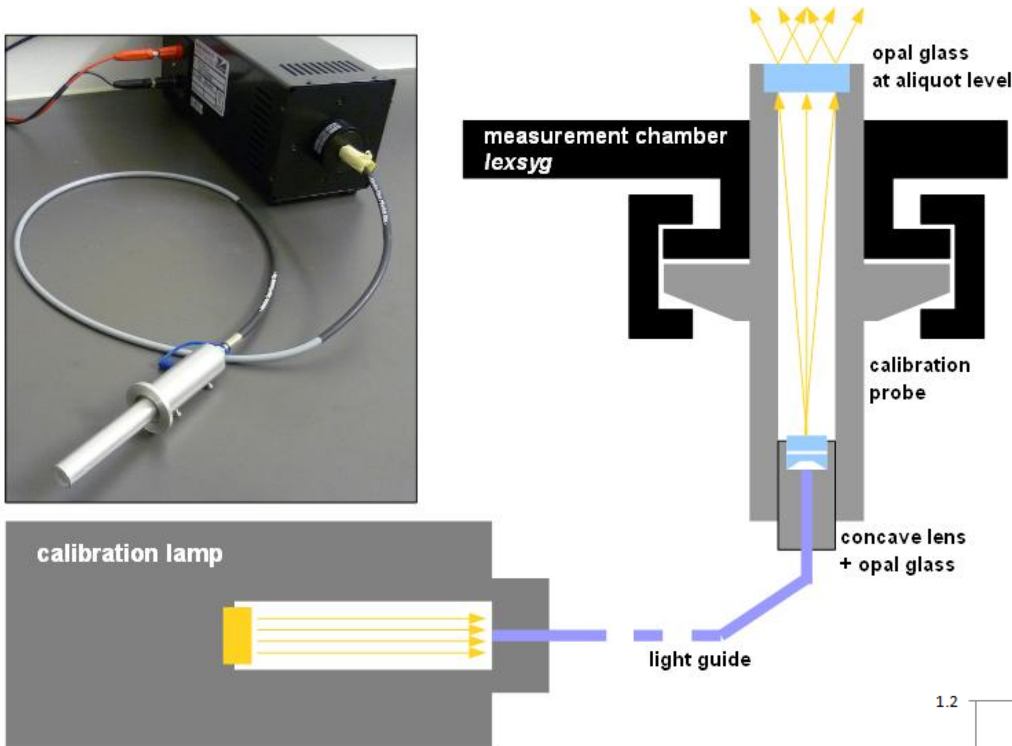
Spectral transfer functions are called **reflectance** or **transmittance** or **efficiency** depending on the considered element

<u>Two ways to get a SRF:</u>

1. Multiplying as many spectral transfer functions as available from data sheets
2. Obtaining it experimentally by measuring an exactly known spectrum [LUM*] and using the relation …

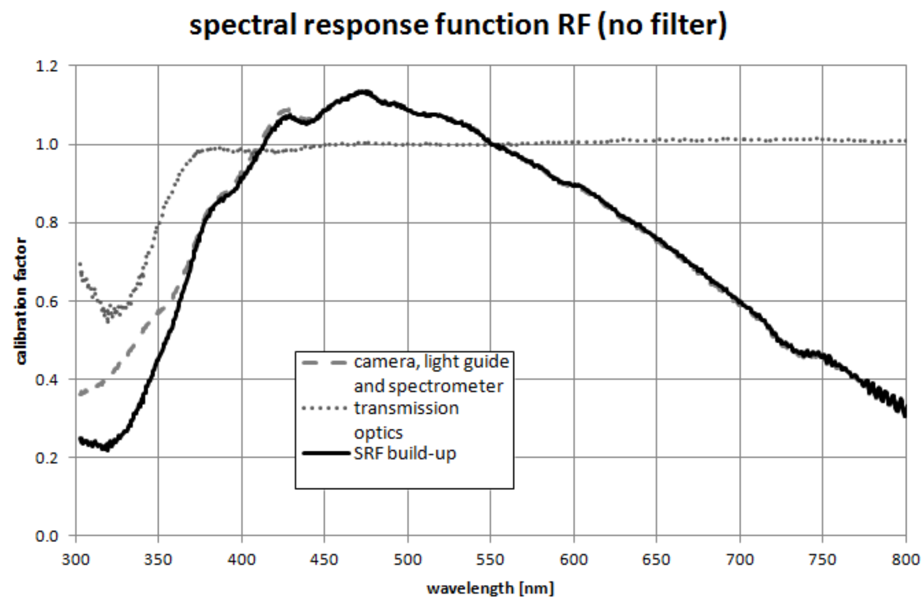
$$[\mathrm{SRF}] = \frac{[\mathrm{MEAS}]}{[\mathrm{LUM^*}]}$$

4.4. Spectral response correction



SRF measurement kit for lexsyg research systems, available for lending at Freiberg Instruments

Developed in coorperation with S. Kreutzer and the AG Geomorphology, Gießen



4.4. Spectral response correction

Getting the spectral response function from **data sheets**:

Primary STFs

- Quantum efficiency of the camera
- Diffraction efficiency of the grating
- Transmittance of every applied filter

Secondary STFs

- Transmittance of the light guide, lenses and safety glasses
- Reflectance of the spectrographs mirrors
- Reflectance of glass surfaces
- Transmittance of AR-coatings (AR-coatings work like bandpass filters)

In Vis-NIR secondary STFs can be ignored usually, but below ~ 400 nm most elements transmittance and reflectance decrease rapidly

Practical issue

How to convert the data sheet spectrum into the same data point interval as the measurement?

- ***R luminescence:*** `apply_EfficiencyCorrection` does this by interpolation with `approx`
- ***Excel***: Polynomal fitting of the datasheet values and rebuilding the STF as column related to the measurements data x-Axis. Eventually the STF of an element has to be seperated to multiple wavelength ranges with a specific polynom each

4.4. Spectral response correction

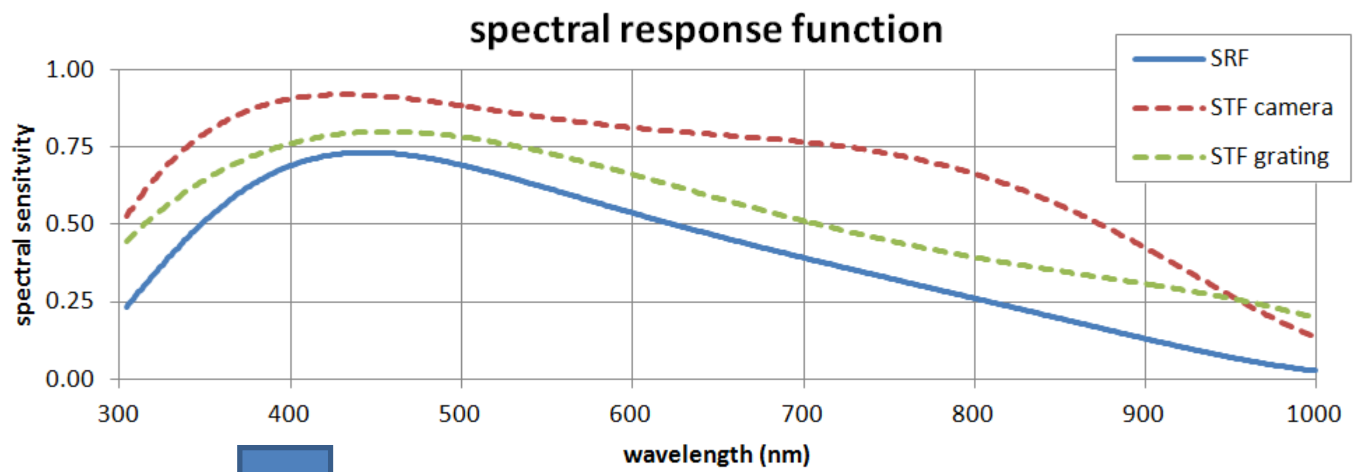
Example data:

<u>STF camera:</u>

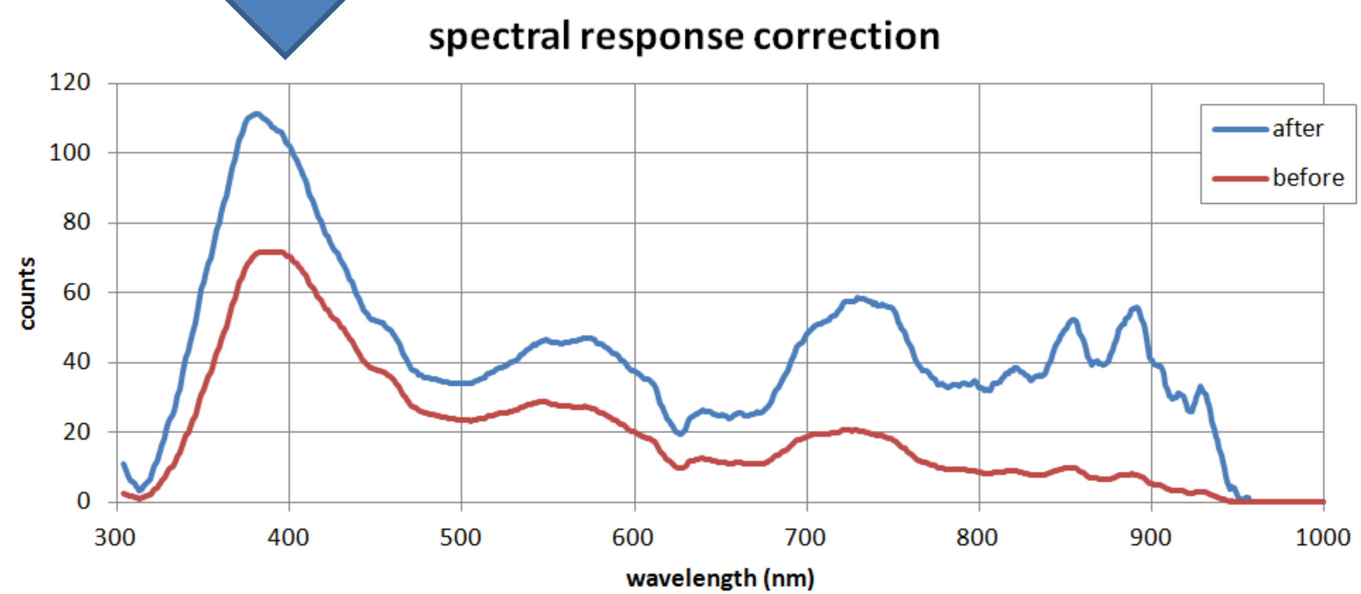
1. CSV data from LOT (trader) by request
2. One polynom 5th grade

<u>STF grating:</u>

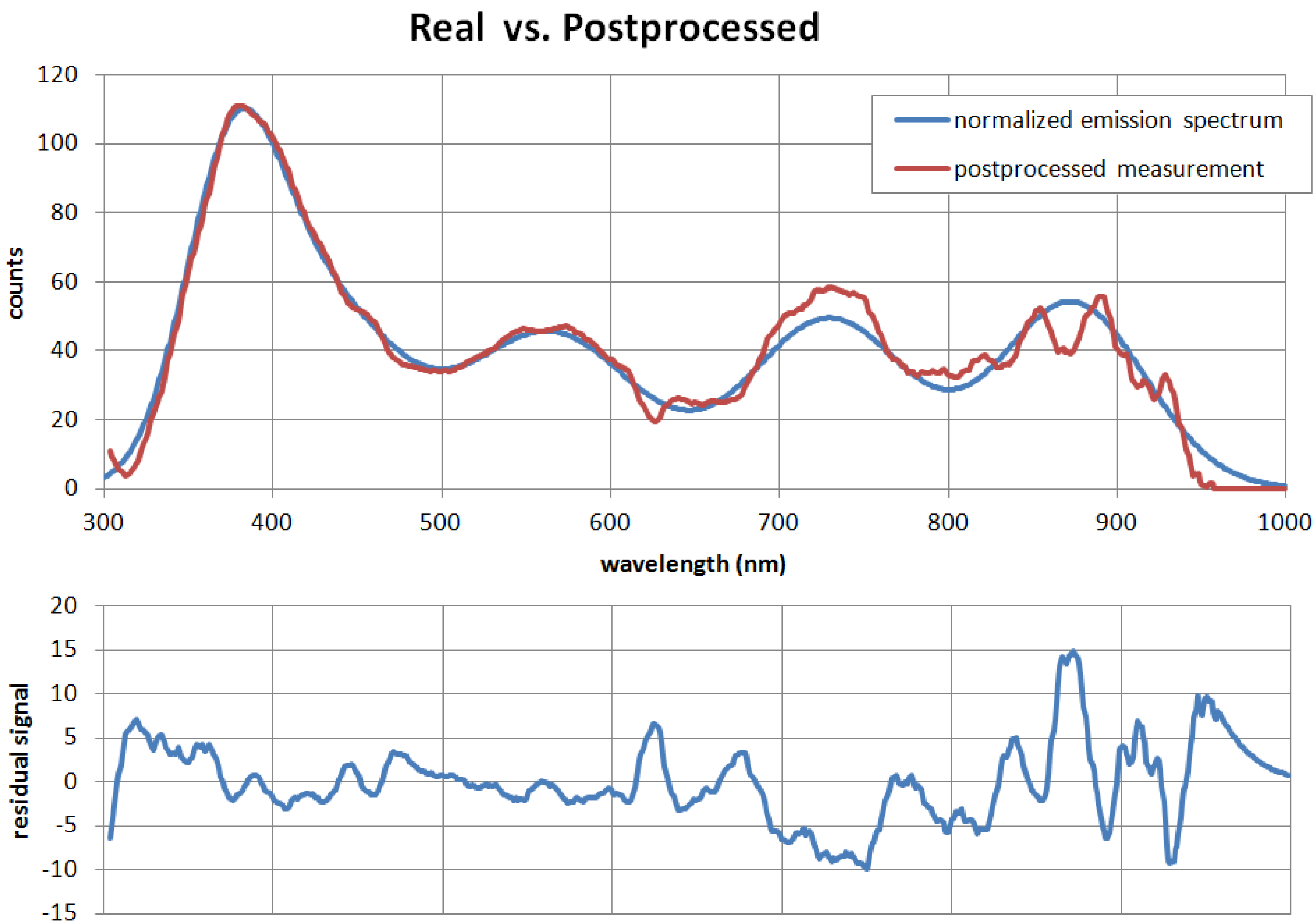
1. PDF datasheet from Richardson grating (manufacturer)
2. Mixed polarization values transcribted ‚over-the-thump'
3. From 800 nm to 1000 nm approximated
4. One polynom 4th grade



[after] = [before] / [SRF] = [before] / ([STF camera] * [STF grating])



4.5. Comparison Real and Corrected



Dominating residual cause: Low frequency part of statistical noise, amplified by low spectral sensitivity at NIR

Thank you for your attention

For the slides and the Excel sheets, send me an short email to:
mittelstrassdirk@gmx.de

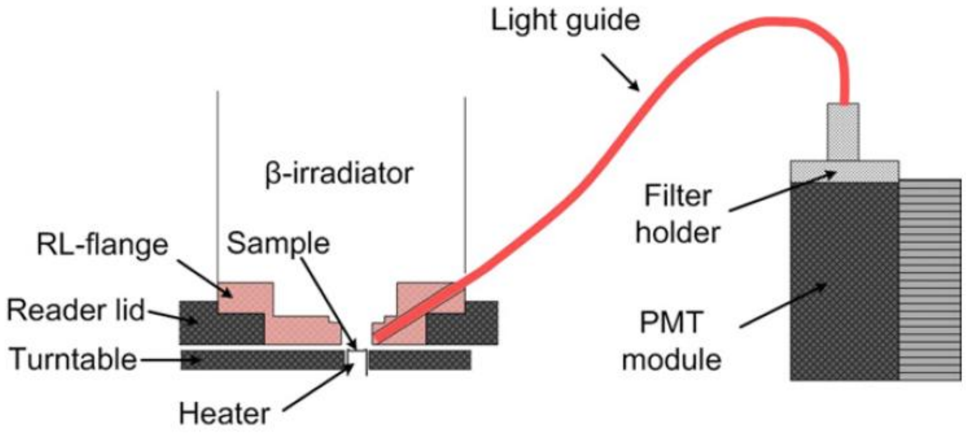
Recommended lecture:
Wang, Y., Townsend, P.D., 2013. Potential problems in collection and data processing of luminescence signals. Journal of Luminescence 142, 202–211.
https://doi.org/10.1016/j.jlumin.2013.03.052

O'Haver, T., 2017. A Pragmatic Introduction to Signal Processing.
pdf at researchgate.net

Palmer, C., Loewen, E., 2014. Diffraction Grating Handbook, 7. ed. Richardson Gratings.
pdf at researchgate.net

1. Spectrometry build-ups

Risö Reader / lexsyg research Xray



Reference:
Lapp, T., Jain, M., Thomsen, K.J., Murray, A.S., Buylaert, J.-P., 2012. New luminescence measurement facilities in retrospective dosimetry. Radiation Measurements 47, 803–808.