

3rd. TRI-NATIONAL WORKSHOP ON STANDARDS FOR NANOTECHNOLOGIES

# Diffraction-Grids Calibration at *CENAM*

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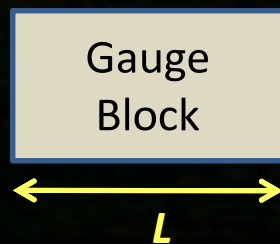
# Calibration

- Operation that, under specified conditions, in a first step, establishes the relationship between **the quantity values with measurement uncertainties** provided by measurement standards and corresponding **indications** with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a **measurement result** from an indication.

## In other words...

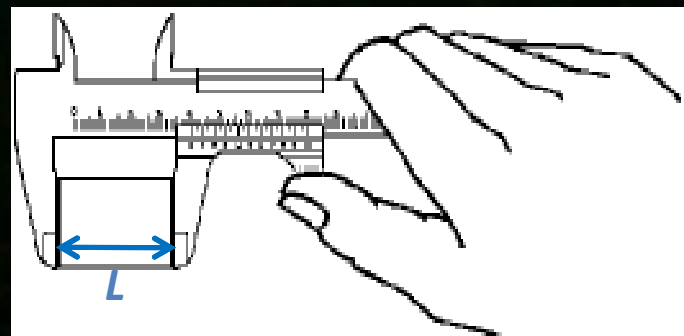
- **Calibration (of an instrument or standard).**- Comparison of the measurement results (of an instrument or a standard) to those of a hierarchically superior instrument or standard. In this operation a calibration uncertainty has to be established (considering the uncertainty of the reference).

Superior standard



$$L \pm U_{GB}$$

Instrument under calibration



$$L (+ Corr) \pm U_{cal}$$

$$U_{cal} > U_{GB}$$

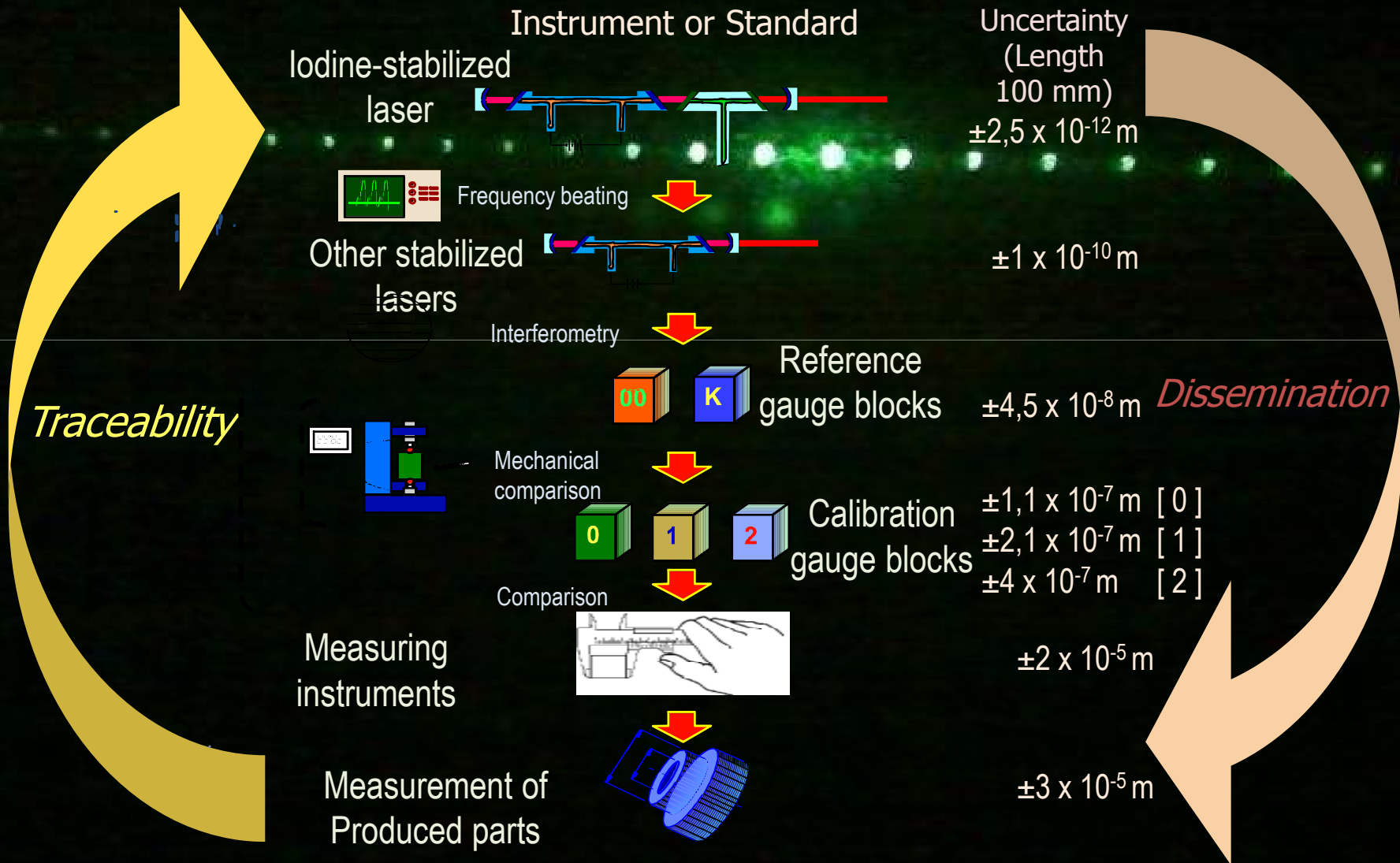


# Traceability

- Property of a measurement result whereby the result may be related to a reference through a documented unbroken chain of **calibrations**, each contributing to the **measurement uncertainty**.

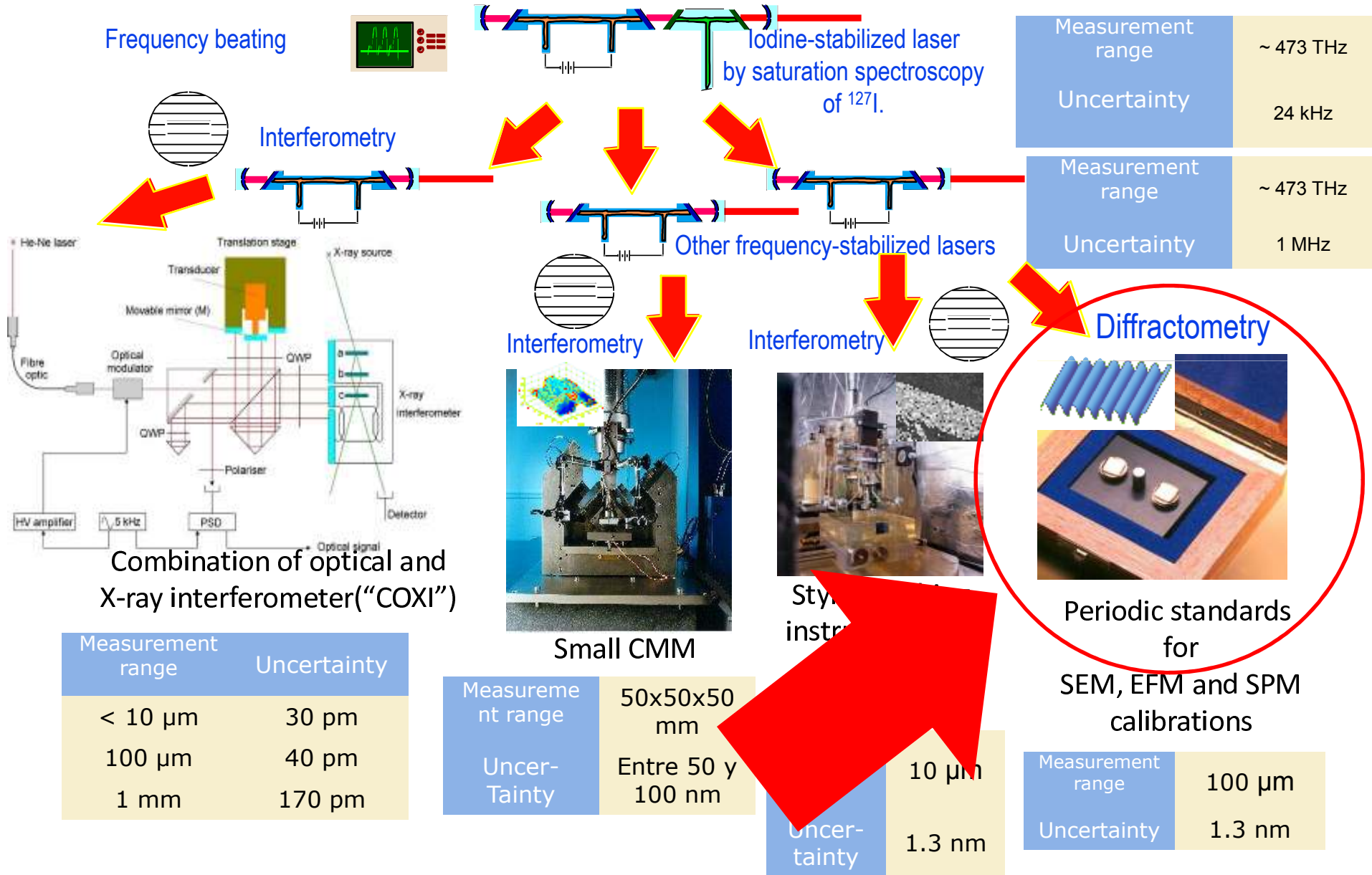
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# Dissemination of the Unit of Length through an Unbroken Chain of Calibrations



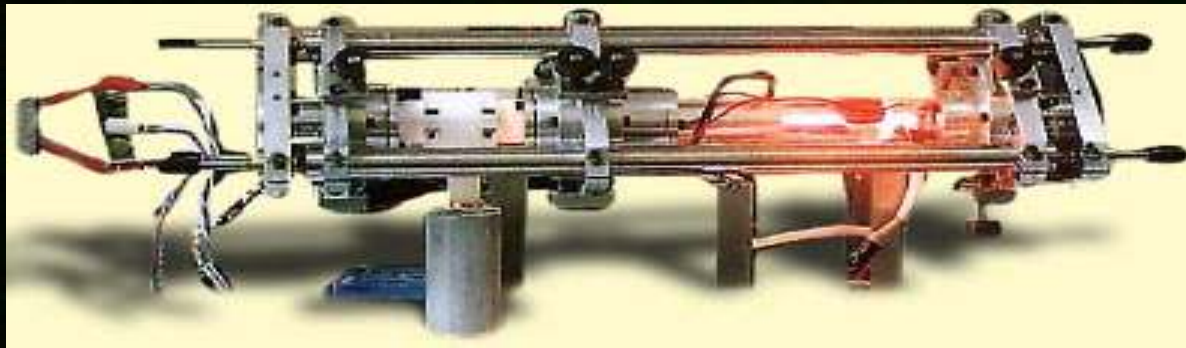
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# Traceability and dissemination of the unit of length in some Nano-applications



# Definition of the length unit

- The metre is the length of the path travelled by light in vacuum during a time interval of  $1/299\,792\,458$  of a second.



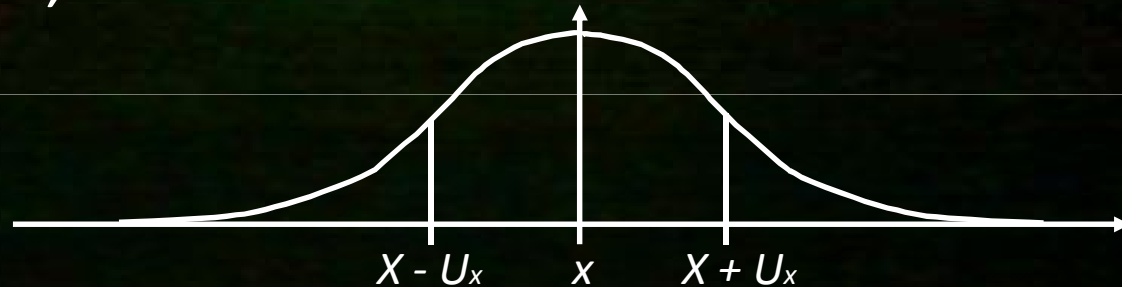
$$\lambda = \frac{c}{f}$$

- Realization at CENAM: He-Ne Iodine-stabilized laser at  $\sim 633$  nm.

# Measurement uncertainty

- Non-negative parameter characterizing the dispersion of the **quantity values** being attributed to a **measurand**, based on the information used.

$$x \pm U_x$$

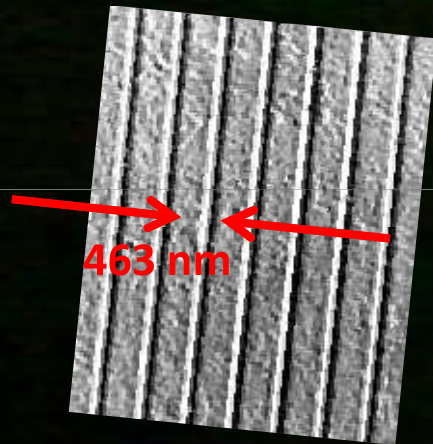


**In other words...**

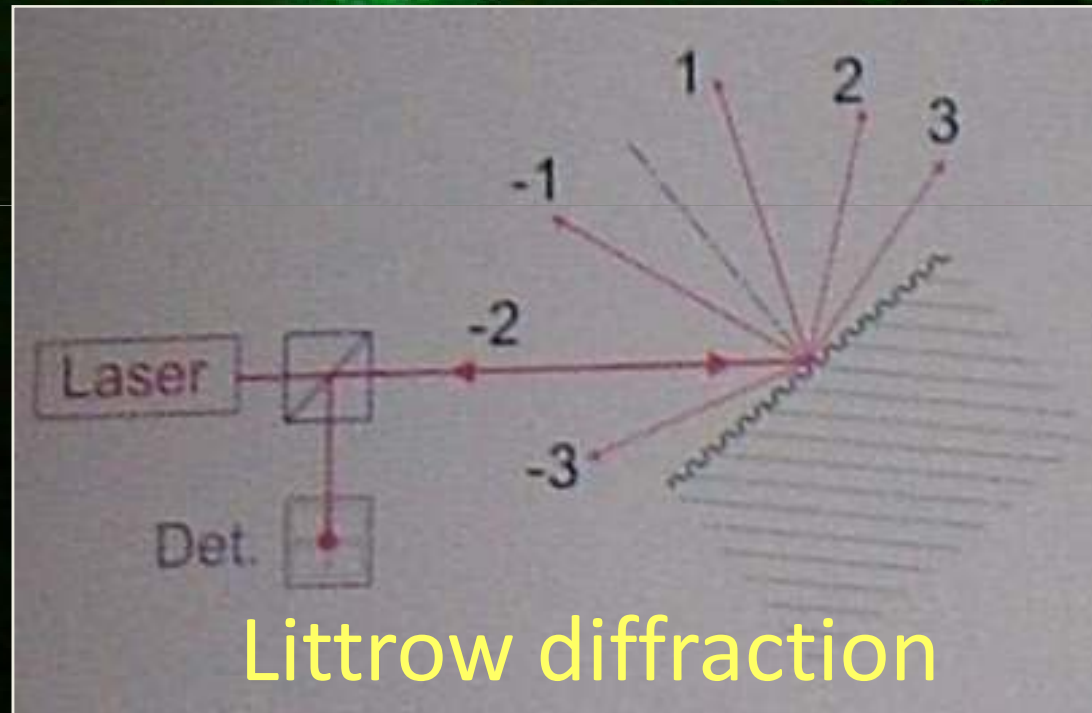
- It is an **interval** ( $U_x$ ) around the **best estimate** of a **measurand** ( $x$ ) where the **true value** of the measurand should lie with a specified **confidence level**.



# Diffractometer principle



Example of a commercial grating:  
2,160 lines/mm





# Diffraction principle

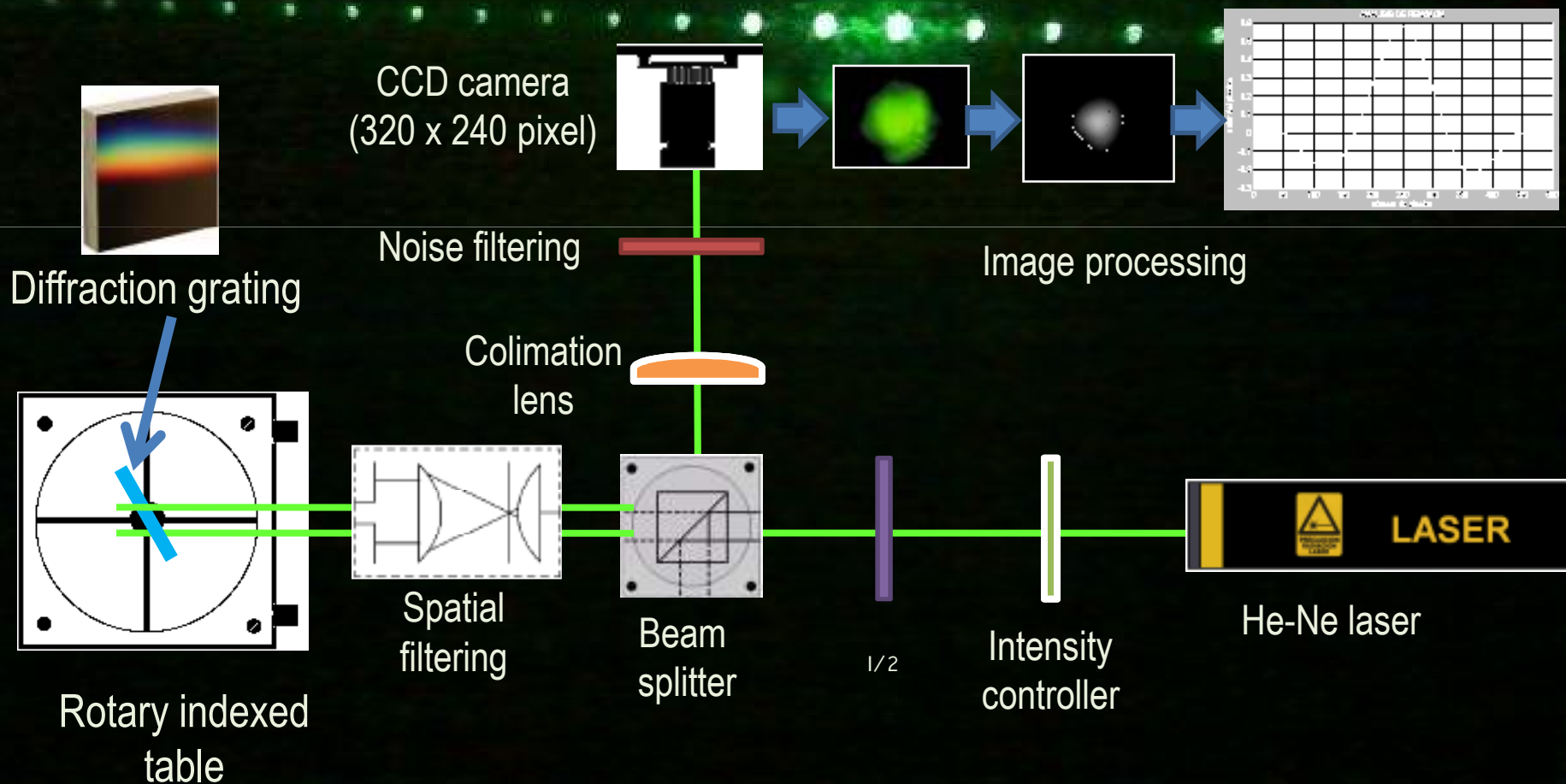
$$p = \frac{m\lambda}{2 \sin \theta}$$

$m$ , diffraction order  
( $m = 1, 2, 3\dots$ ) ;

$\lambda$ , Light wavelength

$\theta$ , measured angle

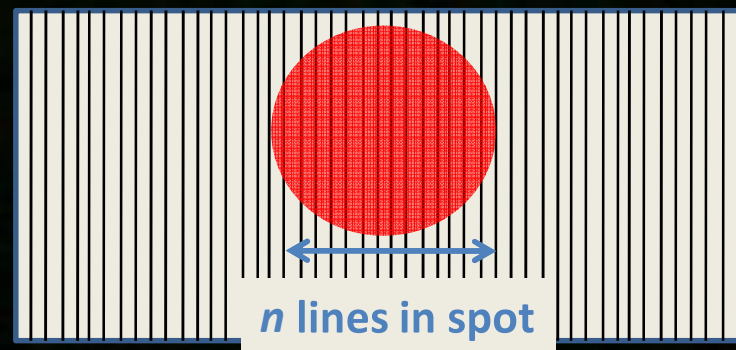
# Measurement set-up





## Some considerations...

- **Calibration** of a **bundle of lines** (between 2 000 and 15 000 depending on the grating and the laser beam spot size) whereas the grating is used to **measure between individual lines**.





## Some considerations...

- Smallest pitch that can be calibrated **limited by** half of the **wavelength** applied (318 nm red, 272 nm green).

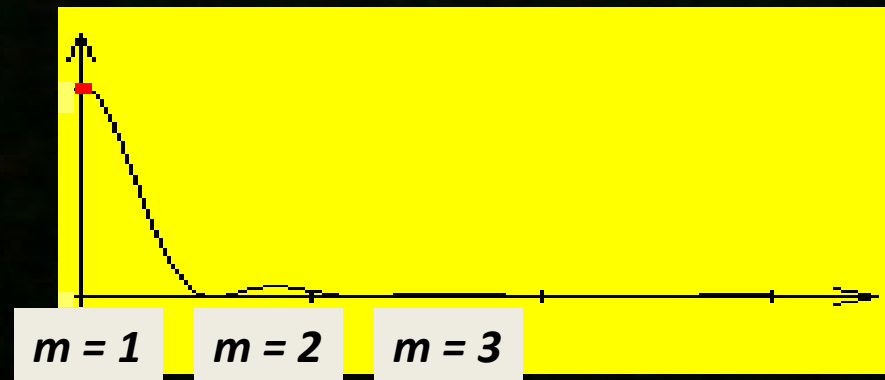
$$p = \frac{\lambda}{2}$$

And only one order of diffraction ( $m = 1$ ) may be observed under this condition.



## Some considerations...

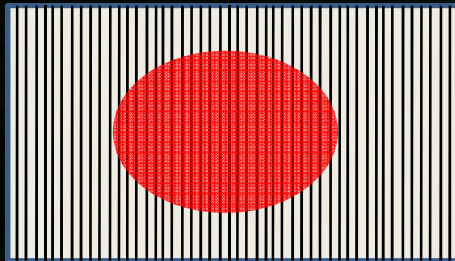
- Number of diffraction orders that can be measured limited by the decreasing intensity of light with diffraction order (only the two first orders are usually measured).



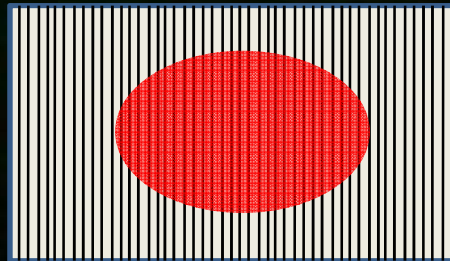


## Some considerations...

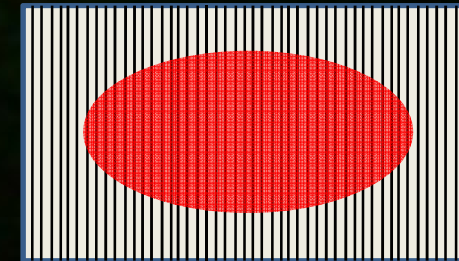
- Number of lines in the averaging spot is a function of the Littrow condition angle.



$n_1$  lines on spot  
Smaller angle



$n_2$  lines on spot



$n_3$  lines on spot  
Larger angle

$$n_1 < n_2 < n_3$$



# Uncertainty of measurement

$$U_p = 2 \sqrt{\left(\frac{\partial p}{\partial \lambda}\right)^2 u_\lambda^2 + \left(\frac{\partial p}{\partial \theta}\right)^2 u_\theta^2}$$

Where:

$$\frac{\partial p}{\partial \lambda} = \frac{m}{2 \operatorname{sen} \theta}$$

$$\frac{\partial p}{\partial \theta} = - \frac{m \lambda \cos \theta}{2 \operatorname{sen}^2 \theta}$$





# Uncertainty of measurement

40  $\mu\text{m}$   
grating  
 $\theta = 0,40^\circ$

| Contrib.  | Type of Unc. | Uncertainty Value                 | Sensitivity Value              | Unc. Cont.                        | Variance                             | %   |
|-----------|--------------|-----------------------------------|--------------------------------|-----------------------------------|--------------------------------------|-----|
| $\lambda$ | B            | $5,44 \times 10^{-7} \mu\text{m}$ | 71,6                           | $1,51 \times 10^{-5} \mu\text{m}$ | $3,78 \times 10^{-10} \mu\text{m}^2$ | 0   |
| $\theta$  | A            | $10'' (4,85 \times 10^{-5})$      | $5,57 \times 10^3 \mu\text{m}$ | $2,70 \times 10^{-1} \mu\text{m}$ | $7,29 \times 10^{-2} \mu\text{m}^2$  | 100 |

$\Rightarrow p = (39,91 \pm 0,54) \mu\text{m}$

|         |                                     |     |
|---------|-------------------------------------|-----|
| $u^2 =$ | $7,29 \times 10^{-2} \mu\text{m}^2$ | 100 |
| $u =$   | $2,70 \times 10^{-1} \mu\text{m}$   |     |
| $U =$   | $0,54 \mu\text{m}$                  |     |

1,66  $\mu\text{m}$   
grating  
 $\theta = 9,65^\circ$

| Contrib.  | Type of Unc. | Uncertainty Value                 | Sensitivity Value  | Unc. Cont.                        | Variance                             | %   |
|-----------|--------------|-----------------------------------|--------------------|-----------------------------------|--------------------------------------|-----|
| $\lambda$ | B            | $5,44 \times 10^{-7} \mu\text{m}$ | 2,98               | $1,62 \times 10^{-6} \mu\text{m}$ | $2,62 \times 10^{-12} \mu\text{m}^2$ | 0   |
| $\theta$  | A            | $10'' (4,85 \times 10^{-5})$      | $9,53 \mu\text{m}$ | $4,62 \times 10^{-4} \mu\text{m}$ | $2,14 \times 10^{-7} \mu\text{m}^2$  | 100 |

$\Rightarrow p = (1,65016 \pm 0,00092) \mu\text{m}$

|         |                                     |     |
|---------|-------------------------------------|-----|
| $u^2 =$ | $2,14 \times 10^{-7} \mu\text{m}^2$ | 100 |
| $u =$   | $4,62 \times 10^{-4} \mu\text{m}$   |     |
| $U =$   | $0,00094 \mu\text{m}$               |     |

# Working prototype





# Conclusions

- Diffraction gratings may be calibrated by a simple set-up of diffractometer.
- The light source frequency stability has negligible impact in the quality of the measurement. An un-stabilized laser may be used.
- The measurement of angle is almost the only source of uncertainty.
- Angle measurement with an uncertainty of  $10''$  seems to be enough.
- Uncertainty is a function of angle and angles larger than  $10^\circ$  are desirable to keep uncertainty low. To be able to get adequate diffraction angles to have a set of different wavelengths available is recommended.
- It is also recommended to measure higher diffraction angles to get larger angles to measure as long as the intensity of the signal is enough.
- The present prototype and experiments will lead to the design of a compact low cost diffractometer to calibrate diffraction gratings. It is the continuation of this project.