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Instruction Manual on Using Gonioreflectometer Setup for Spectral Bidirectional Radiance Factor and Diffuse Reflectance Measurements

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2. Definition

2.1. Scope

This instruction manual describes principles and operation of the gonioreflectometer setup at the Metrology Research Institute (MRI) that is used to perform absolute measurements of diffuse reflectance factors. Procedures for the characterization of the instrument and calibration of diffuse reflectance standards are given. This equipment can be used for measuring spectral bidirectional reflectance distribution function (BRDF) and spectral bidirectional radiance factors of non-fluorescent opaque reflecting materials. It can also be used for the measurement of fluorescent opaque reflecting materials at wavelengths outside their spectral range of excitation or emission.

2.2. Object and field of application

Gonioreflectometer: setup for measuring radiance and reflectance factors.

Reflectance factor: ratio of the reflectance of a test sample to that of the perfect reflecting diffuser under standardized geometrical conditions, such as incidence at 0° and collection of the diffuse component or the reflected flux (0°:d), or incidence at 8° and collection over the whole hemisphere (8°:t), or incidence at 0° and collection at 45° (0°:45°).

Radiance factor: ratio of the reflectance of a test sample to that of the perfect reflecting diffuser under in-plane geometrical conditions, such as incidence at 0° and collection of the reflected flux within a solid angle of 0.002 steradians at angles in the range of ±10° to ±85°. *Reflected Radiance Factor* is also defined as the product of BRDF and π steradians.

2.3. Features

The gonioreflectometer setup consists of a light source and a one-axis goniometric detection systems [1]. The setup is mounted on an optical table in the gonioreflectometer lab of the MRI. The instrument is designed to perform angle-resolved spectrophotometric measurements. In the case of normal incidence of the beam, smallest possible viewing angle is 10° while for the incidence angles ≥ 10° it is 0°. The incidence angle can be adjusted to any value within +90° to -90°. By integrating the measured angular distribution of the reflected radiant flux, hemispherical reflectance factors can be derived, such as 0°:d or 8°:t. The diffuse reflectance factors from the range of 0.05 – 1.0 can be determined over the wavelengths from 300 to 1650 nm.

[1] S. Nevas, F. Manoocheri, and E. Ikonen, “Gonioreflectometer for measuring spectral diffuse reflectance,” *Applied Optics* **43**, 6391-6399 (2004).

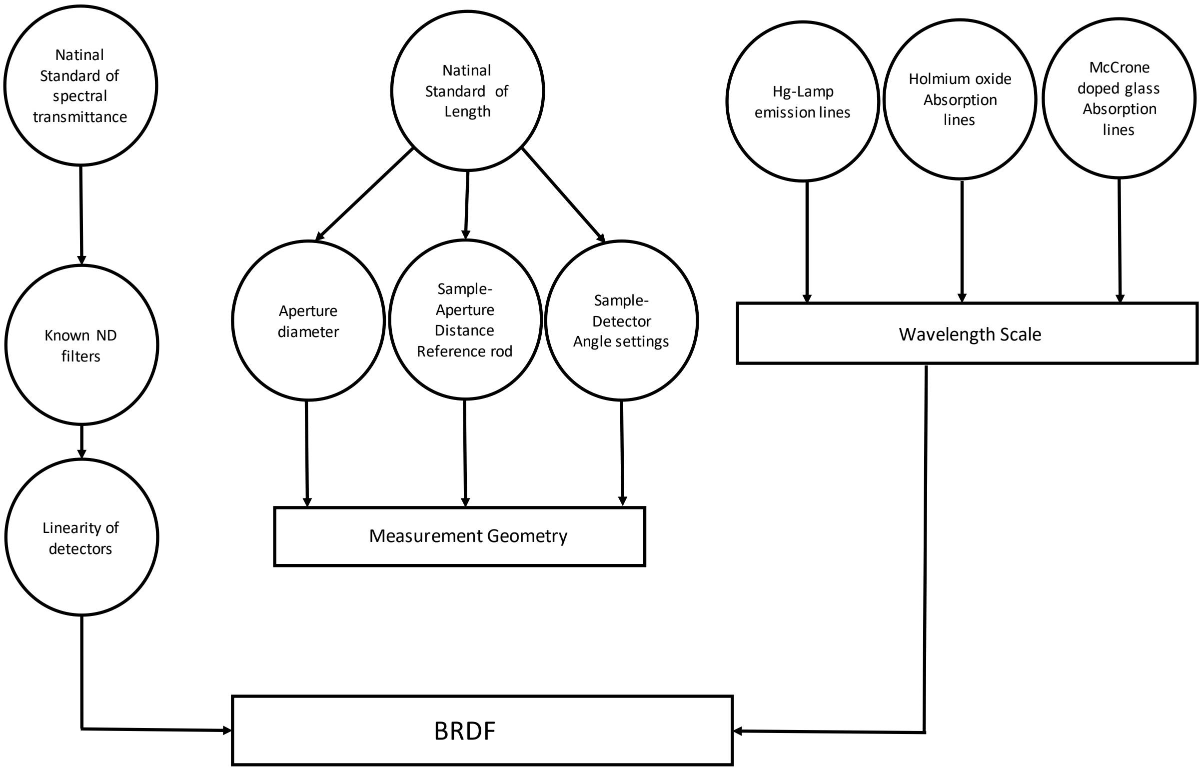
2.4. Principles and Definitions

The hemispherical reflectance factors are derived from the measured distribution of the reflected flux as a function of the polar angle. The distribution of the flux is obtained from the detector signal readings, known area of the detector aperture and known distance between the aperture and the reflecting surface / axis of rotation. The principles and definitions necessary for deriving the diffuse reflectance factors when using the gonioreflectometer setup are described with more details in ref. [1].

3. Traceability Chain

The hemispherical reflectance factors are determined from the measured angular distribution of the reflected radiant flux i.e. from the Bidirectional Reflectance Distribution Function (BRDF) or spectral bidirectional radiance factors. The flow chart below shows the traceability chain for the BRDF measurement results.

Bidirectional Reflectance Distribution Function (BRDF) Traceability chain



4. Equipment

4.1. Gonioreflectometer setup

The gonioreflectometer consists of a double monochromator-based light source system, a one-axis goniometric receiver system, two digital voltage meters (DVMs), and a control computer. A schematic representation of the source and the receiver systems of the gonioreflectometer setup is shown in Figure 1. The components utilized in the measurement setup are listed in Table 1.

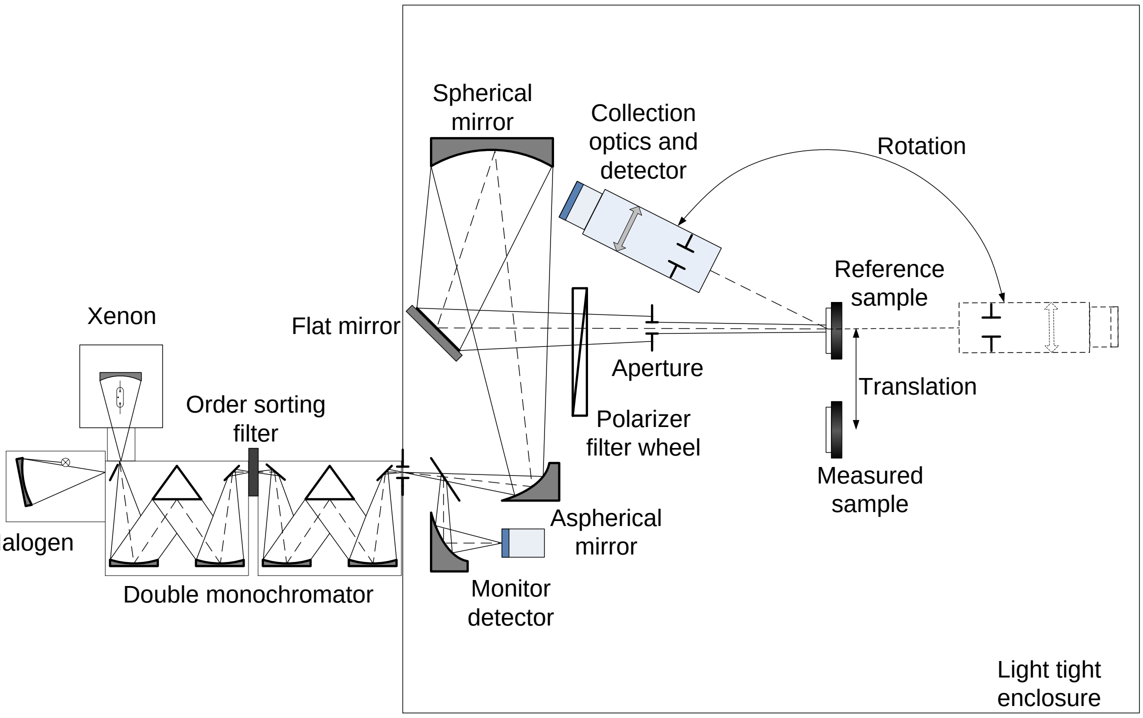


Figure 1. Schematics of the gonioreflectometer setup.

Table 1. Equipment used in the gonioreflectometer setup.

	Description	Quantity	Identification
A.	**Light source system**		
1.	Double monochromator	1	Bentham DTMc 300
2.	Quartz-tungsten-halogen (QTH) lamp with power supply	1	OSRAM, 50W 12V; Elekro-Automatik GmbH, EA 7030-100
3.	Xenon lamp	1	
4.	Spherical concave mirror	1	Teknofokus, custom design, ∅ 100 mm, F = 125 mm
5.	Off-axis parabolic mirror	1	Melles Griot POA017

6.	Flat mirror	1	Melles Griot 02MFG019/028
7.	Monitor detector	1	Silicon diode connected to a preamplifier made at MRI
8.	Beam splitter	1	50x50 plate of C7059 optical glass
9.	Polarizer	1	Two sets of polarizer sheets in 4 positions of a filter wheel Or Melles Griot Glan-Taylor prism, attached to PI M-038.DG turntable
10.	Adjustable iris	3	Thorlabs

B. Goniometric Detection system

1.	Detector turntable with the controller	1	HUBER GmbH one-circle goniometer 420 and 9012 controller
2.	Linear translator with the controller	1	Physik Instrumente (PI) GmbH & Co. M-415.PD with power supply, C-862.00 / Mercury II Palm-Top DC-Motor-Controller
3.	Sample turntable	1	PI M-038, operated manually
4.	Sample-holder unit	1	Designed and made at MRI
5.	Si detector with	1	Hamamatsu S1337 photodiode, Connected to a SRS amplifier
6.	InGaAs detector		Hamamatsu xxxx photodiode
7.	calibrated transimpedance amplifier with PC control gain		Stanford Research Systems SR570s
8.	Detector optics package	1	Two SM2L30 Thorlabs lens tubes, ∅ 25 mm black anodised Al aperture, Thorlabs LA4545 fused-silica plano-convex Lens
9.	Tools for setting the distance between the detector and the sample surface	1	Calibrated 498-mm long rod of Al, caliper, specially designed plate for the sample holder and cover for the detector tube

C. Control and Data Acquisition

1.	Digital multimeter	2	HP 3458A or Agilent, HP 34401A or HP 34410A
2.	Control computer	1	PC with GPIB card and 2 RS-232 ports, OS MS WinXP
3.	Software		NI LabView v.5.1 (or higher) software development environment, the measurements are controlled by DReflect_scan_asym.vi within Measurement_control_newMC1.llb library

4.2. Maintenance of the equipment

- The equipment used in the gonioreflectometer setup is not supposed to be removed from the setup. This does not apply to widely used equipment, such as DVMs and motor controllers.
- The quartz envelope of QTH lamp is not supposed to be touched with bare fingers. The lamp is operated in limited current mode. The lamp 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 QTH lamp box of the Gonioreflectometer. The source is replaced with a new one when the sum is about 1000 hours for the halogen lamp and 2000 hours for the Xenon lamp (Xe lamp power supply keeps track for elapsed working time).
- The time period of the operation of the source is recorded in hours and it is replaced with a new one after being used for ~90% of its average lifetime.
- The monochromator is checked for proper operation each time before starting the measurements. The wavelength calibration is renewed upon a demand. When not in use it should be kept switched off.
- The mirrors and other optical components in the source system should be handled with extreme care in order to avoid contamination or damage. When necessary, cleaning can be arranged, which should be carried out following the guidelines for safe cleaning of optics [2].
- The detector turntable and the linear translator are checked for proper operation before being used and a repair arranged if necessary. Care must be taken to lubricate the turn table with a dedicated grease on a timely basis every 5 years.
- The photodetector is tested for linearity every third year. This can be done by e.g. using reference neutral-density grey filters.

[2] "Instructions for cleaning optical filters", *Oriel publication*.

- Edge of the detector aperture is not allowed to be touched by any means.
- The calibrated aluminum rod used for setting the detector distance should be stored in a safe place with the plastic protective caps on both ends. When handling, care should be exercised to avoid mechanical damage.
- The digital multimeters are auto calibrated every day before the start of the measurements. Calibration of the multimeters is performed according to the calibration schedule of MRI.

5. Uncertainty components

The uncertainty components for the measurements of spectral diffuse reflectance using the gonioreflectometer setup are described in ref. [1]. Table 3 summarizes best measurement capabilities for measurements of high-quality non-fluorescent samples with the nominal bidirectional radiance factor (BRF) in the range of 0.01 to 50.

For rare cases of BRF from 50 to 5000, the uncertainty can be up to 50% and depends on the quality of the sample surface.

Table 3. List of standard uncertainty components for the gonioreflectometer setup and their effect on the relative uncertainty of BRF measurements. (VIS: 500-800 nm, NIR: (800-1000 nm)

Source of uncertainty	**Standard uncertainty**		**Uncertainty in Radiance factor/ %**	
	VIS	NIR	VIS	NIR
Measurement noise	0.29%	0.10%	0.29	0.10
Instrument stability*	0.13%	0.12%	0.13	0.12
Wavelength* **	0.1 nm	0.2 nm	0.15	0.26
Straylight (isochromatic)	0.14%	0.04%	0.14	0.04
Solid angle (due to sample-detector distance and detector aperture diameter)**	0.15 mm 2 μm	0.15 mm 2 μm	0.06 0.02	0.06 0.02
Detector nonlinearity	0.04%	0.09%	0.07	0.13
Spatial nonuniformity***	0.10%	0.06%	0.1	0.06
Illumination and viewing angles*	0.10°	0.15°	0.02-0.3	0.04-0.4
Polarization***	0.10 %	0.10%	0.1	0.1
Combined standard uncertainty			0.41-0.51	0.36-0.53

* This uncertainty depends on the viewing zenith angle

** This uncertainty depends on the sample mounting method

*** This uncertainty depends on the sample

6. Measurement ranges and best measurement capabilities

Table 2 summarizes best measurement capabilities for measurements of high-quality non-fluorescent samples with the nominal 0/d reflectance factor values from the range of 0.05 – 1.0. The uncertainty estimation has been divided into two wavelength regions because of the varying signal/noise ratio. In addition, sample induced uncertainties such as material homogeneity are to be quadratically added to the given uncertainties.

Table 2. Uncertainty components in 0/d reflectance factor measurements for high quality samples with nominal reflectances of 1.0 - 0.05 over UV, visible and NIR wavelengths. The numbers are relative percent-values.

	Uncertainty Component		Relative Standard Uncertainty (%)	
			360-440 nm	440-830 nm
	Reflectance level 1.0-0.50			
1.	Signal noise		0.20	0.04
2.	Instrument stability		0.02	0.02
3.	Aperture-to-sample distance		0.06	0.06
4.	Aperture diameter		0.02	0.02
5.	Wavelength		0.02	0.01
6.	Stray light	isochromatic	0.08	0.08
		heterochromatic	0.01	0.01
7.	Detector nonlinearity		0.04	0.04
8.	Spatial non-uniformity		0.10	0.10
9.	Illumination and viewing angles		0.01	0.01
10.	Polarization		0.05	0.05
11.	Combined uncertainty		0.26	0.16
12.	Expanded uncertainty (k = 2)		0.52	0.32
	Uncertainty Component		Relative Standard Uncertainty (%)	
			360-440 nm	440-830 nm
	Reflectance level 0.50-0.25			
1.	Signal noise		0.40	0.10
2.	Instrument stability		0.02	0.02
3.	Aperture-to-sample distance		0.06	0.06
4.	Aperture diameter		0.02	0.02

5.	Wavelength		0.02	0.01
6.	Stray light:	isochromatic	0.08	0.08
		heterochromatic	0.01	0.01
7.	Detector nonlinearity		0.04	0.04
8.	Spatial non-uniformity		0.10	0.10
9.	Illumination and viewing angles		0.01	0.01
10.	Polarization		0.05	0.05
11.	Combined uncertainty		0.43	0.19
12.	Expanded uncertainty (k = 2)		0.86	0.38

	Uncertainty Component		Relative Standard Uncertainty (%)	
			360-440 nm	440-830 nm
	Reflectance level 0.25-0.05			
1.	Signal noise		0.75	0.50
2.	Instrument stability		0.02	0.02
3.	Aperture-to-sample distance		0.06	0.06
4.	Aperture diameter		0.02	0.02
5.	Wavelength		0.02	0.01
6.	Stray light:	isochromatic	0.08	0.08
		heterochromatic	0.01	0.01
7.	Detector nonlinearity		0.04	0.04
8.	Spatial non-uniformity		0.10	0.10
9.	Illumination and viewing angles		0.01	0.01
10.	Polarization		0.05	0.05
11.	Combined uncertainty		0.77	0.52
12.	Expanded uncertainty (k = 2)		1.54	1.04

7. Calibration methods and procedures

7.1. Measurement method

The hemispherical reflectance factors are determined from the measured angular distribution of the reflected radiant flux. The measurements are performed over the polar angles only. More information of the measurement method is provided in [1]. Procedure for calculating the hemispherical reflectance factor values is built in the LabView program that controls the measurements. Alternatively, the raw measurement data can be post processed with the help of a Mathlab code that is in a file called GUI.m and gonio file_opener.m.

7.2. Procedures for preparing the gonioreflectometer setup for the measurements

Before the measurements can be started, both source and receiver systems of the gonioreflectometer have to be set up, aligned, and calibrated.

The optical components at the input and output slits of the double monochromator (DMC) are set up and aligned according to the schematic representation of Figure 1. The light from the QTH lamp is collected by a spherical mirror and coupled into the input slit of the DMC assembly [1]. The positioning of the lamp and the mirror at the input slit is depicted in Figure 2. Under the depicted configuration, the image of the lamp filament at the DMC input slit is magnified by a factor of two and the F/# of the input optics is matched to that of the DMC.

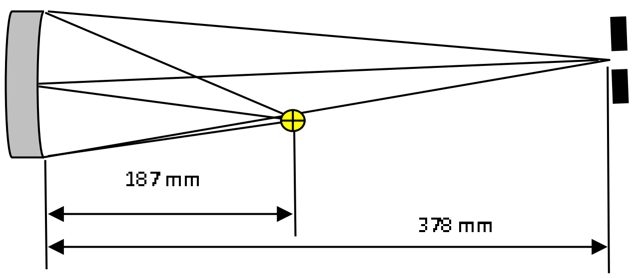


Figure 2. Coupling the light from QTH lamp into the DMC.

The configuration of the output optics as shown in Figure 1 is slightly different from what is described in ref. [1]. In the present setup, the spherical mirror and one flat mirror are absent. This is due to changes in the DMC, which made it astigmatism free. Hence the astigmatism correction became unnecessary and some of the optical components became redundant. However, the purpose of the remaining optical components is the same as described in [1] and the guidelines for their alignment are valid.

Procedures for calibrating the wavelength scale of the instrument are described in [1]. The additional equipment needed for the wavelength calibration is the same as in [3]. The result of the wavelength calibrations is expressed in the form of wavelength-correction polynomials determined separately for each of the gratings in use. The polynomial formulas are then input to the DMC control VIs within the Measurement_control_newMC1.llb.

The receiver system is aligned in a series of consecutive steps that are discussed in [1]. This instruction manual supplements the description with some important practical details. As mentioned in [1], the alignments are carried out with the help of a two beam alignment laser. It is fixed on the optical table just after the flat mirror that is used for steering the beam towards the receiver system (Figure 1).

The first step in the alignment procedure is to adjust the beam to be in the plane that is at the height of the DMC exit slit and parallel to the surface of the optical table (Figure 3). The plane of rotation by the detector and the sample are also parallel to the surface of the optical table.

For the next step, finding the rotational axis of the detector turntable and making the beam to intercept it, the following approach should be used.

- Remove optical detector from big turntable and place a Thorlabs post with a hole or iris instead.
- Adjust two-beam laser so that one line goes through all optics and monochromator (it should be set to laser wavelength) up to light source filament and other line of the laser goes to the hole of post or iris.
- Move turntable 180 degrees and check again whether laser line goes through the post/iris hole. If not adjust laser again so that it goes through illumination optical system on one side and hole on the other. The displacement of the beam from the centre of the iris opening at the new position is an indication of how much and to which direction the laser should be moved (Figure 4).
- Move turntable back to 0 position. Check if the beam passes through the hole again. Repeat the procedure of laser adjustment until laser goes trough hole of post or iris at 0 and 180 positions. When it is, the laser goes through rotational axis of the gonioreflectometer. Move turntable using TTposit.vi

[3] Quality manual of reference spectrometer laboratory.

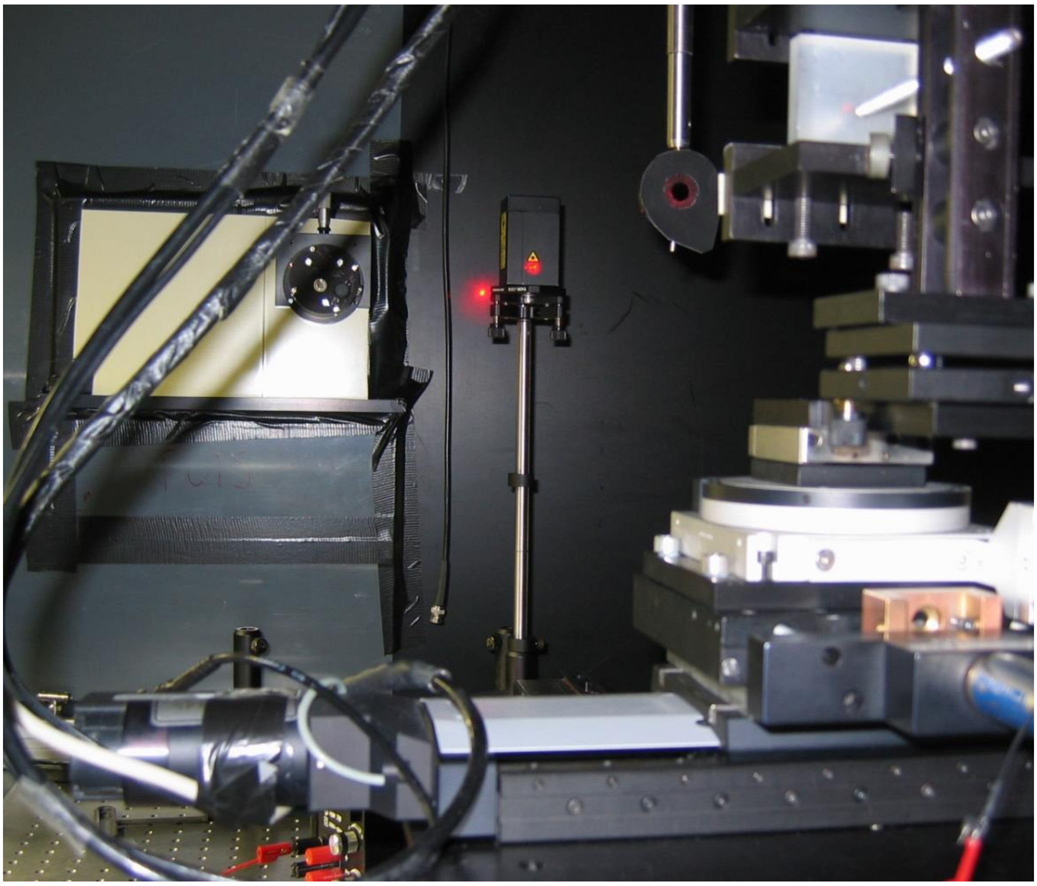


Figure 3. An alignment laser is used for the alignment of the receiver system.

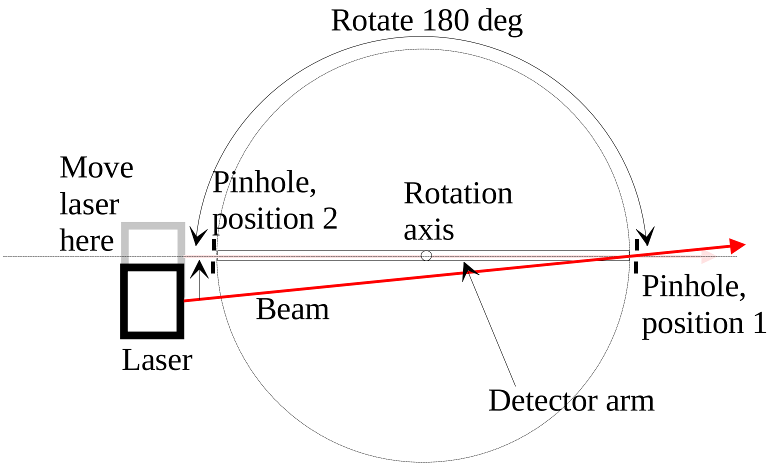


Figure 4. Finding the axis of rotation of the detector turntable with the help of an alignment laser and a small aperture opening fixed on the detector arm.

The following steps in the receiver alignment procedure are aimed at aligning the rotational axis of the second turntable (sample) to be lying in the plane of the sample front surface and making both the sample and the detector axis of rotation to be collinear. For that purpose, a dummy sample with cross hair markings on its surface is fixed at the sample holder. The concept of the alignment procedure is based on tracking the displacement of the beam spot position on the sample surface with respect to the reference mark while the sample is rotated both clockwise and counterclockwise.

In the first move, align the dummy sample such that the laser beam is incident at normal angle on the center of the cross hair marking. Now, if the sample is rotated four scenarios are possible. In the ideal case, the center of the illuminated spot on the sample surface does not change its position with respect to the reference mark no matter how much and to which direction the sample is rotated. This would mean that the alignment is already achieved. The other three scenarios are depicted in Figure 5: 1) the rotational axis of the sample turntable lies in the sample front surface but it is away from the incident beam (and from the rotational axis of the detector turntable); 2) the rotational axis of the sample turntable is away from the sample front surface but it lies in the plane formed by the incident beam and the rotational axis of the detector turntable); 3) the rotational axis of the sample turntable is away both from the sample front surface and from the laser beam (and also from the rotational axis of the detector turntable). As it is demonstrated in Figure 5, rotation of the sample by $\pm\alpha$ causes a displacement between the reference mark and the beam spot on the sample surface that is denoted by *A* and *B*, respectively. Obviously, observing the sign and the size of *A* and *B* allows deducing the type and the amount for the misalignment among the rotational axis, sample surface, and the optical axis (laser beam).

Hence one should proceed as follows. Turn the sample by e.g. +80° and note the sign and the magnitude for the displacement *A*. Then turn the sample by -80° and note the sign and the magnitude for the displacement *B*. Compare *A* and *B* and refer to Figure 5. Depending on the situation, use the PI linear translator to move the sample turntable sideways with respect to the beam and/or the small translator with an adjustment knob to adjust the position of the sample holder along the direction of the laser beam such that *A* and *B* get smaller. Readjust the position of the dummy sample. Iterate the sequence until both *A* and *B* converge at zero.

PI translator is controlled by MR_LT.vi (move relative from current position), GoHomeLT.vi (move to reference home position), and DefHomeLT.vi (define current position as home). For the alignment purposes, however, it may be more convenient to adjust the position of the translator manually and then use the DefHomeLT.vi to mark the adjusted position as the reference home position.

The next action to be taken is to adjust the position of the rotational axis of the sample turntable along the optical axis (laser beam) such that it is at the position of the rotational axis of the detector turntable. The required degree of freedom for this adjustment is provided by the horizontal slide (Oriel) on which the sample turntable is fixed. The measure for the necessary adjustment to be applied is again a displacement between the beam spot and the reference mark on the dummy sample. This time, however, the laser is fixed on the detector arm and the laser itself is rotated rather than the sample.

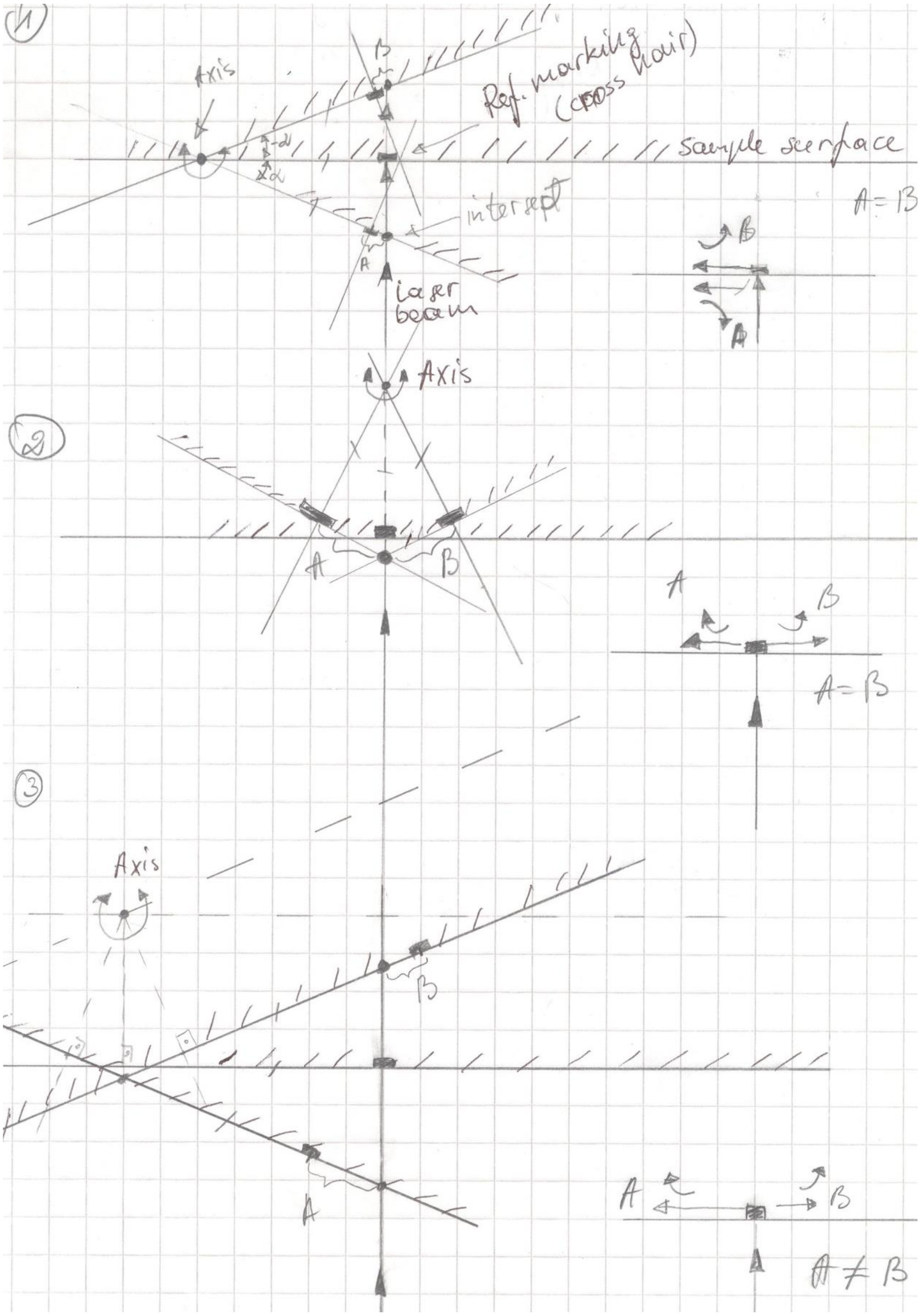


Figure 5. Three types of misalignment among sample, rotational axis, and incident beam. Rotation of the sample causes displacement between the beam spot and the reference mark on the sample surface.

The purpose of the final stage in the alignment procedure is to define the distance between the detector aperture and the sample plane. For this purpose, the alignment laser, a calibrated-length aluminum rod, a specially designed cover for the detector tube and a special plate for the sample holder are used. First of all, the detector package is mounted on the cantilever at the reference position of the detector turntable. The detector tube is aligned such that the optical axis goes through its center. The part of the tube containing the lens and the aperture is removed and the cover is put on the remaining part of the tube. The plate is put into the sample holder and aligned such that the laser beam (optical axis) is incident at zero degree on a small hole at its centre. Then the detector is rotated 180° to face the sample. The aluminum rod is put in between the sample holder and the detector (Figure 6). One end of the rod slides into a clearance in the plate. The other end is matched by a clearance in the detector tube cover. The distance setting is carried out by releasing the fixing screws on the detector rail carrier and carefully pushing the detector towards the sample until the detector cover just touches the aluminum rod. When the distance is set, the removed part of the detector tube is put back. The distance between the detector aperture and the sample is calculated from the known length of the aluminum rod and that of the removable part of the detector tube, the measured distance between the aperture and the tube edge (caliper can be used for that purpose), the thickness of the tube cover, the thickness of the plate in the sample holder, and the thickness of the front part of the sample holder.

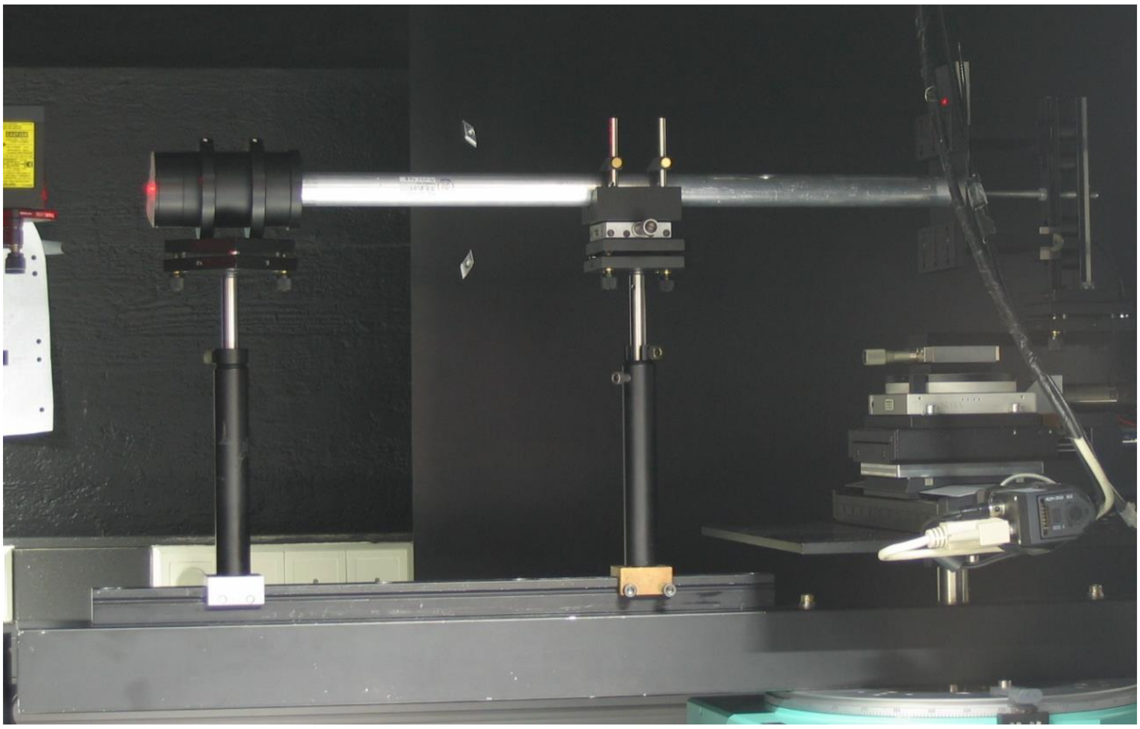


Figure 6. Defining the distance between the detector and the sample with the help of a calibrated-length aluminum rod.

8. Laboratory accommodation and environment

The gonioreflectometer laboratory is located in room 1572 in the first floor of the department of Electrical and Communications Engineering. The laboratory is a clean room. Instructions for using the clean rooms have been given in [4].

During the calibrations, the ambient temperature and the relative humidity should be monitored. Temperature and relative humidity values during the calibrations are written to calibration certificates.

[4] P.Kärhä, "Clean room instructions / Puhdastilaohjeet", *MRI publication*.

9. Records

The measurement data coming from the calibrations and development of the equipment is archived. The measurement notes (comments on the measurement setup etc.) are written down to measurement data file (the measurement control program has a place where to type in the comments) and/or a dedicated notebook. The raw data files are stored in the control computer and backed-up in responsible person's PC.

10. Certificates

Calibration certificates are handled according to [5]. In the calibration certificates included are:

- Ambient temperature and relative humidity,
- Source of traceability,
- Spectral diffuse reflectance values at the measurement wavelengths and uncertainty estimates.

[5] *Instructions on writing calibration certificates*.