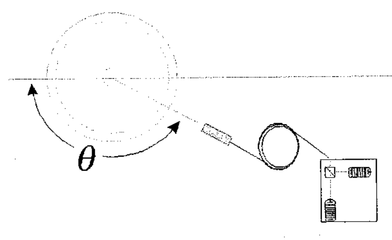
ALV / DLS / SLS - 5000 System

Compact Goniometer System

Adjustment Manual V.2.2. 03/98

(WinAlign-Version)

Compact Goniometer



$$\mathbf{I}_S \propto \left| \int e^{i\mathbf{r}(\mathbf{k}_i - \mathbf{k}_j)}\, d\mathbf{r} \right|^2$$

Content

Nomenclature:

CDN	Centricity Definition Needle
CGS	Compact Goniometer System
DNAU	Double Needle Adjustment Unit
IMV	Index Matching Vat
LHDN	Laserbeam Height Definition Needle
LSE	Light Scattering Electronics
ODS	Optical Detection System
SO-SIPD	Small Outline-Single Photon Detector
X,Y,Z	Cartesian System of Coordinates, X - axis in direction of laser beam propagation (180°-0°) Y - axis in the rotation plane of the goniometer, perpendicular to X-axis Z - height axis
$\Theta_{Y,Z}$	tilt angle with respect to the Y and Z - axis.

1.0 Installation of the Compact Goniometer System, Preparations

1.1 Installation of the Mechanical Components

The following modules of the Compact Goniometer System (CGS) are mounted onto the optical table/breadboard or aluminium-profile table according to the positions schematically drawn in Fig. 1:

- Laser Beam Folding/Adjustment Unit I, or
- Laser Beam Folding/Adjustment Unit II for use of two separated lasers (optional, instead of Unit I),
- Laser mounted on an appropriate stand,
- Monitor-Diode/Attenuator Unit,
- ALV-125 standard goniometer with cell housing (remove the "Click-Stop-Shutter" at the 180° position of the cell housing, remove the beam stop or beam stop/beam folding housing (= part of the 0°-diode, optional) at the 0° position of the cell housing),
- Projection optics system, optionally with 90°- or 270°- beam folding unit

All modules of the CGS which mechanically obstruct free turning of the arm of the rotary table between 0° and 180°(e.g. beam protection tube) or optically interfere with the free laser beam pass (e.g. beam splitter plate, Laser Beam Optimisation Unit) have **not** yet to be mounted.

1.2 Installation of the Electronic Components

Attention! *Prior to connect or disconnect any cable to or from the ALV/LSE unit it is mandatory to switch off the power. Otherwise damage of electronic components of the ALV/LSE unit may occur caused by undefined current peaks.*

1.2.1 ALV/LSE-5000

The ALV/LSE-5000 unit usually contains the following modules:

- ALV-3014 with RS-232 asynchronous serial port
- ALV-3017 Stepper Motor and Encoder Controller with Temperature Input/Control
- ALV-3018 Monitor Diode Input and Temperature Input/Control.

For detailed description please refer to the manual ALV-3014 Bus Controller for ALV-Light Scattering Electronics.
Connect the RS-232 cable from the computer to the ALV-3014 module according to the instructions "Functional Test of RS-232C...".
Connect the special cable for the stepper motor, encoder and limit switches of the goniometer to the socket MOTOR of the ALV-3017 module, the cable(s) from the monitor diode(s) to the ALV-3018 module and the cable from the ALV-3027/Pt-100 unit to the ALV-3017 module (TEM0 socket).

1.2.1 ALV/LSE-5001

The ALV/LSE-5001 unit contains the RS-232 asynchronous serial port for communication with the computer and the stepper motor and encoder controller logic. Laser beam intensity measurement via the monitor diode and temperature measurement using the ALV-3027/Pt-100 unit are performed directly by the ALV-5000/E/WIN Correlator Board.

Connect the RS-232 cable from the computer to the ALV/LSE-5001 unit according to the instructions "Functional Test of RS-232C...".

Connect the special cable for the stepper motor, encoder and limit switches of the goniometer to the socket MOTOR of the ALV/LSE-5001.
A special cable is used to connect the monitor diode and the ALV-3027/Pt-100 unit to the ALV-5000/E/WIN Correlator Board.

1.3 Installation of the Software

Insert the CD-ROM containing the ALV software and following the instructions to install the ALV-5000/E WINDOWS or DOS software and the ALV/WIN-Alignment software in order to operate and align the ALV-Compact Goniometer System.
The ALV/WIN-Alignment software is a program under MS-WINDOWS for semi-automatic adjustment and for optimisation of the laser beam path. This program supports the following steps of the alignment procedure:

- Alignment of the cell housing with respect to the axis of rotation of the rotary table;
- Alignment of the index-matching vat with respect to the axis of rotation of the rotary table, independent of the alignment of the cell housing.
- Alignment of the laser beam with respect to the plane of rotation of the goniometer rotary arm

The program requires the use of either the ALV/LSE-5000 or 5001 unit.

1.4 Installation of the Optical Components

1.4.1 General Handling of Optical Components

All optical components used in the ALV-Compact Goniometer System are highly precise manufactured parts with polished and if necessary multi-layer antireflection coated surfaces. To guarantee optimum performance all optical surfaces must be clean from dust and other contamination (e.g., traces of oil or fat, finger prints etc.), otherwise the Gaussian profile of the laser beam is distorted and/or additional stray light occurs which distorts the static light scattering measurements.

Attention! *Never touch lenses and laser mirrors with bare hands,* ***always*** *protect optical surfaces by use of lens-paper!! In case optical elements have to be hold by hand use sufficient layers (min. of 3 layers) of lens paper between surface and finger skin.*

Use only special lens cleaning paper (we recommend to use KODAK lens cleaning paper) and pure organic solvents, having a high vapour pressure, such as methyl ethyl ketone (2-butanone), acetone or isopropanole. The solvents must be of p.A. quality and should be fresh destilled. Methyl ethyl ketone starts to become a yellowish colouring with time due to radical formation resulting in high molecular impurities. Acetone as well as isopropanole must be free of water. ALV company recommends to use methyl ethyl ketone.

Never use any surfactant type glass cleaning agents (e.g., surfactants, metal-organic complex forming agents), such agents remove the multilayer coating from the optical surfaces.

The lens paper can be used loosely wadded to remove dust from the surface. To remove oil films and other contamination put a sheet of lens paper on the optical surface, then moisten it using a pipette with a drop of the organic solvent and slowly drag the lens paper along the surface. Be careful to remove the solvent with the impurities completely from the surface by dragging the lens paper.

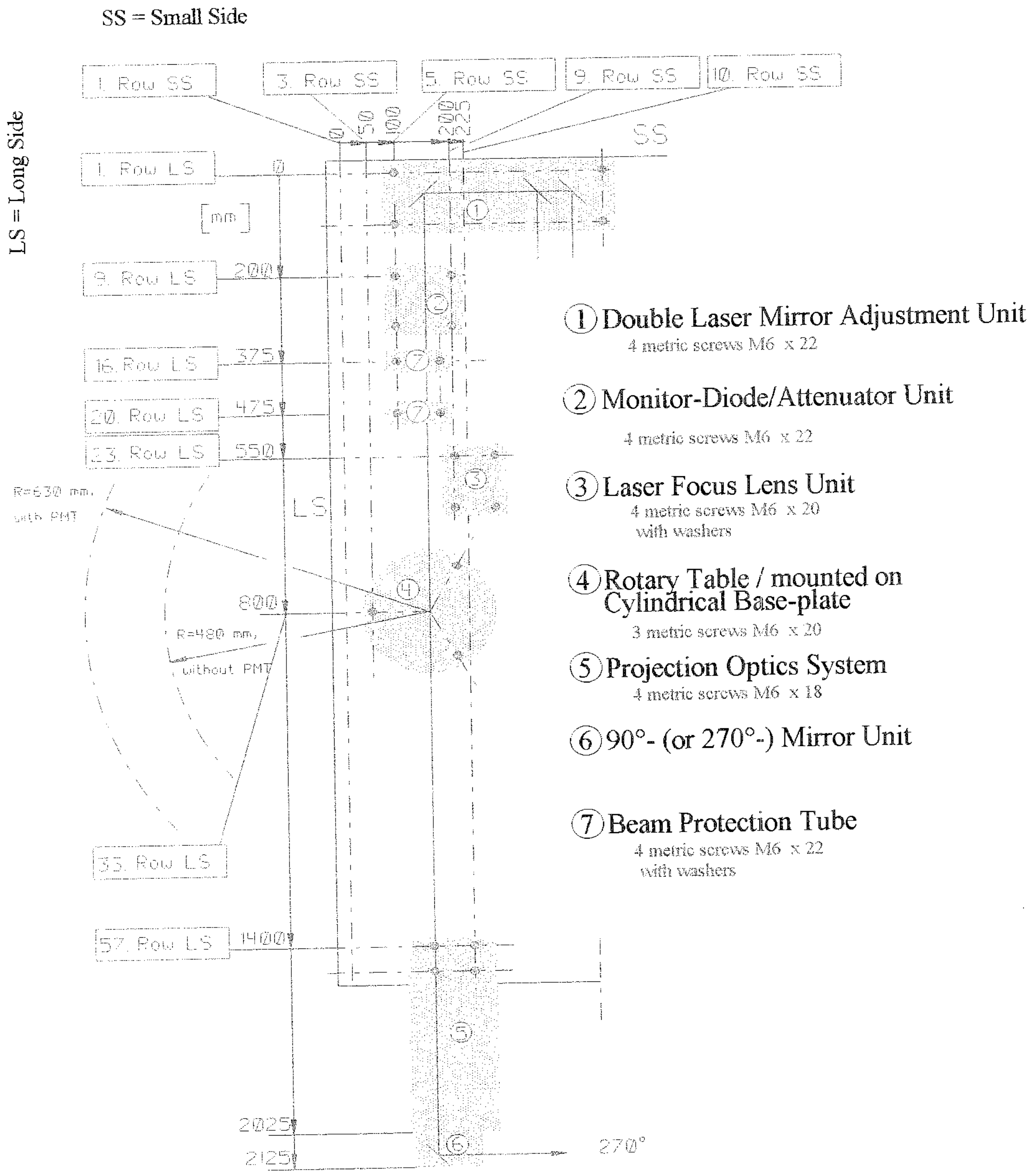


Figure 1a:
ALV / DLS/SLS-5000P
2F/2F Pinhole/Lens Compact Goniometer System

Screw-down positions of the opto-mechanical modules/units on top of the optical table/breadboard with standard size:

600 mm B x 1800 mm L x 58 mm H
and
25 x 25 mm grid spacing with metric threads of M6

Figure on following page shows - similar to figure 1a - :

ALV / DLS/SLS-5000F

Fiber Compact Goniometer System

using

ALV / STATIC and DYNAMIC Enhancer

and

Laser Focussing Lens Unit

Screw-down positions of the opto-mechanical modules/units on top of the optical table/breadboard with standard size:

600 mm B x 1800 mm L x 58 mm H

and

25 x 25 mm grid spacing with metric threads of M6

1.4.2 Installation of Optical Components

- Mount the laser mirrors (mirrors with ∅ = 25 mm are mounted in adapters with ∅ = 50 mm) in to the gimbals of the Laser Beam Folding/Adjustment Unit I or II, respectively.
- Mount the projection lens (FL = 50,8 mm, ∅ = 50 mm) in to the gimbal of the Projection Optics System; mount the projection mirror in to the gimbal for the 90°/270°- mirror unit of the Projection Optics System, if purchased as part of delivery.

1.5 Preparations

- The CGS has two limit switches to prevent the rotary arm of the goniometer to move to angular ranges which are not accessible during measurement mode due to mechanical obstructions (Focus Lens Unit and beam protection tubes at angles larger than 155°) or which should not be accessed for safety reasons (low angular range, to avoid overexposure of light to the photomultiplier unit due to large forward scattering intensities, reflexes and stray light from the cuvette etc.). During alignment of the CGS deactivate the limit switches to have access to the whole angular range between 0° and 180°, which provides the highest accuracy for mechanical alignment and is mandatory to align the laser beam position using the Double Needle Adjustment Unit (DNAU). The limit switches are based on two Hall-effect switches, each of them is activated by a limit positioner, which is a small iron plate and positioned in the groove at the rotor part of the rotary table. The limit positioners are fixed using a M3 hex socket screw (1.5 mm hex key) and their positions correspond to the lowest and highest angle, accessible to the goniometer rotary arm during routine measurements (factory installation low angle ≅ 14°, high angle ≅ 151°). The limit switches are mounted in a compartment and positioned at the rear side of the goniometer/rotary table. They can either be deactivated using a Limit Switch Shorting Adapter or by moving the whole compartment containing the limit switches downwards. In case a Limit Switch Shorting Adapter is available, unplug the cable from the compartment. The cable plug is fixed positioned in the socket (as part of the compartment) by a click-stop mechanism with spring load. The spring firms the outer oversized locking part of the cable-plug, and this part must be pulled off in the direction from the socket in order to release the cable-plug. **Attention!** Never rotate the cable plug while it is inserted in the socket, and, never drag directly the cable, you will otherwise shear or pull off the wires of the cable from their appropriate pins - hence, when this has appeared, then the limit switches are constantly off (for safety reasons the logic is chosen to be active if the signal is off), blocking the motor from turning. The cable plug is fixed in a socket with click stop position which is only released if the outer ring of the plug is pulled out first. Then the Limit Switch Shorting Adapter is connected to the cable plug. If no Limit Switch Shorting Adapter is available the whole compartment has to be moved down do deactivate the limit switches, first slightly loosening the two Allen-head screws holding the unit and then move it down by approx. 3-5 mm. The limit positioners remain at their positions in the groove at the rotor part of the rotary table.

After completion of the adjustment of the laser beam, either the Limit Switch Shorting Adapter has to be removed and the cable plug to be inserted into the socket or the compartment with the limit switches has to be lifted up. In the later case make certain that the gap between the limit positioners and the limit switches has a width of approx. 1.5 mm. We recommend to use a card board sheet with this thickness to check for this width and to perform a test of the limit switches by trying to access angles beyond the limits set by the limit positioners. Finally do not forget to fasten the two Allen head screws again!

During execution of the ALV/WIN-Alignment program, all entries of readings of micrometers or dial / feeler gauges are made as decimal numbers with unit mm, e. g. for a micrometer reading of 17.436 (mm).

Be warned (!) that all micrometers move only 500 μm = 0.5 mm per turn (360°). Readings may therefore be difficult, e.g. a reading of 12.460 is easily misinterpreted as a reading of 12.960.

2.0 Basic Adjustment Principles

The axis of rotation, respectively the level of rotation - which corresponds to the scattering plane - of the rotary table is the main reference for the entire goniometer system. All relevant modules or units have to be adjusted to this reference; these units are:

1. the cell housing with the cuvette holder, and
2. the index matching vat, and finally - not being a unit -
3. the laser beam has to be adjusted appropriate parallel in height to the rotary level and to pass the rotary axis symmetrically.

The exact position of the cuvette is most critical for static light scattering measurements using cylindrical cuvettes with an outer diameter of 10 mm, the positioning of the cuvette becomes less critical using cuvettes with larger diameter due to lower curvature of the cuvette surface; therefore alignment is performed inserting the cuvette holder for cuvettes with 10 mm outer diameter in to the conical bore of the upper heat exchanger. To ensure exact repositioning of the cuvette holder in the conical bore of the upper heat exchanger the cuvette holder has a line-marking on its top which must be positioned to the small line-marker on the upper rim of the upper heat exchanger (at 270° position).

The first step of the adjustment is to position the cell housing and with this the cuvette holder. The adjustment aim is to position the cell housing appropriately until the axis of rotation of the rotary table coincides with the symmetry axis of the cuvette in the cuvette holder. To achieve utmost precision the Calibration Disk as part of the system is inserted in to the cuvette holder instead of a cuvette. This Calibration Disk is machined in master quality, respectively with such an high accuracy, that at the position of the tip of the dial gauge the level of rotation (PLANE) deviates less than ± 2 μm from a plane perpendicular oriented to the Calibration Disk axis **A** and the perpendicularly oriented level (SHIFT) is parallel to the same axis within ± 2 μm.

To adjust the cell housing to its final position, using the Calibration Disk and the Double Dial Gauge Unit (Fig. 5), requires - on request of the computer - to enter via keyboard the readings of the appropriate dial gauge to the ALV/WIN-Alignment program, and to continue with the next step of the adjustment according to the displayed instructions. The relevant adjustment is completed when the readings of the appropriate dial gauge show finally only variations within 1/2 digit from a mean value reading of the dial gauge, while the rotary arm advances from 0° - 180° or vice versa; **please note:** a reading of 1/2 digit on the dial gauge corresponds to an absolute measurement of **5 μm**.

Please, keep in mind a human hair equals in average to 60 μm in diameter.

However, a repositioning of the Calibration Disk in the cuvette holder is only accurate to less then 20 µm with respect to the rotational axis, and is a consequence of the sum of all tolerances of the machined accuracy's of the units involved. But, the above stated accuracy of mechanical alignment of the cell housing guarantees the necessary alignment precision.

The exact position of the cuvette holder as part of the cell housing is not only essential for the actual measurement once the system is operative, but, moreover it is mandatory during the adjustment procedure of the laser beam. The cuvette holder accepts the Centricity Definition Needle (CDN), which defines with its tip of the needle the center of symmetry of the detected volume

The laser beam alignment is based on the use of diffraction patterns, which occur if precisely conical machined needle tips are inserted into the laser beam. Provided the laser beam profile is of Gaussian type, the diffraction pattern is symmetrically if the needle tip is positioned symmetrical to the laser beam. Three different needle adjustment tools are part of the standard delivery:

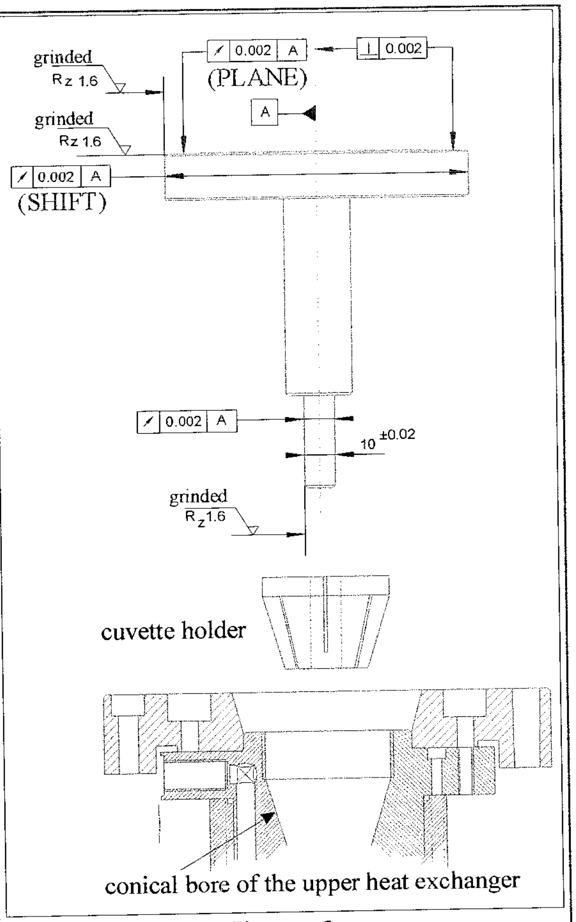


Figure 2: Calibration Disk

- the Centricity Definition Needle (CDN), to be inserted into the cuvette holder,
- the Height Definition Needle (LHDN), to be inserted instead of the cuvette holder into the conical bore of the upper heat exchanger,
- The Double Needle Adjustment Unit (DNAU), to be mounted on the rotary arm of the rotary table, consisting of two needles, perpendicular oriented to each other and each mounted on a micrometer controlled ball bearing sliding stage.

The DNAU allows defined positioning of the relevant needle in either HORIZONTAL position or VERTICAL height, and, can therefore either be used to measure the actual beam position ,or, after successful adjustment of the laser beam was achieved they can be used for a defined re-positioning of each needle by setting the relevant micrometer to the appropriate reading.

As the DNAU travels with the movement of the rotary table to an exact value of $\Delta = 180.000° \pm 3/1000°$, the position of both needles define a straight line, crossing the axis of rotation of the goniometer perpendicular. Together with the CDN and LHDN an adjustment of the laser beam can be achieved within an accuracy of a few arc seconds with respect to the tilt angle out of the scattering plane and of 10 - 20 µm lateral deviation from symmetrical crossing of the axis of rotation.

3.0 Adjustment of the Cell Housing

3.1 Description of the Cell Housing

A drawing of the cell housing is shown in Figure 3.

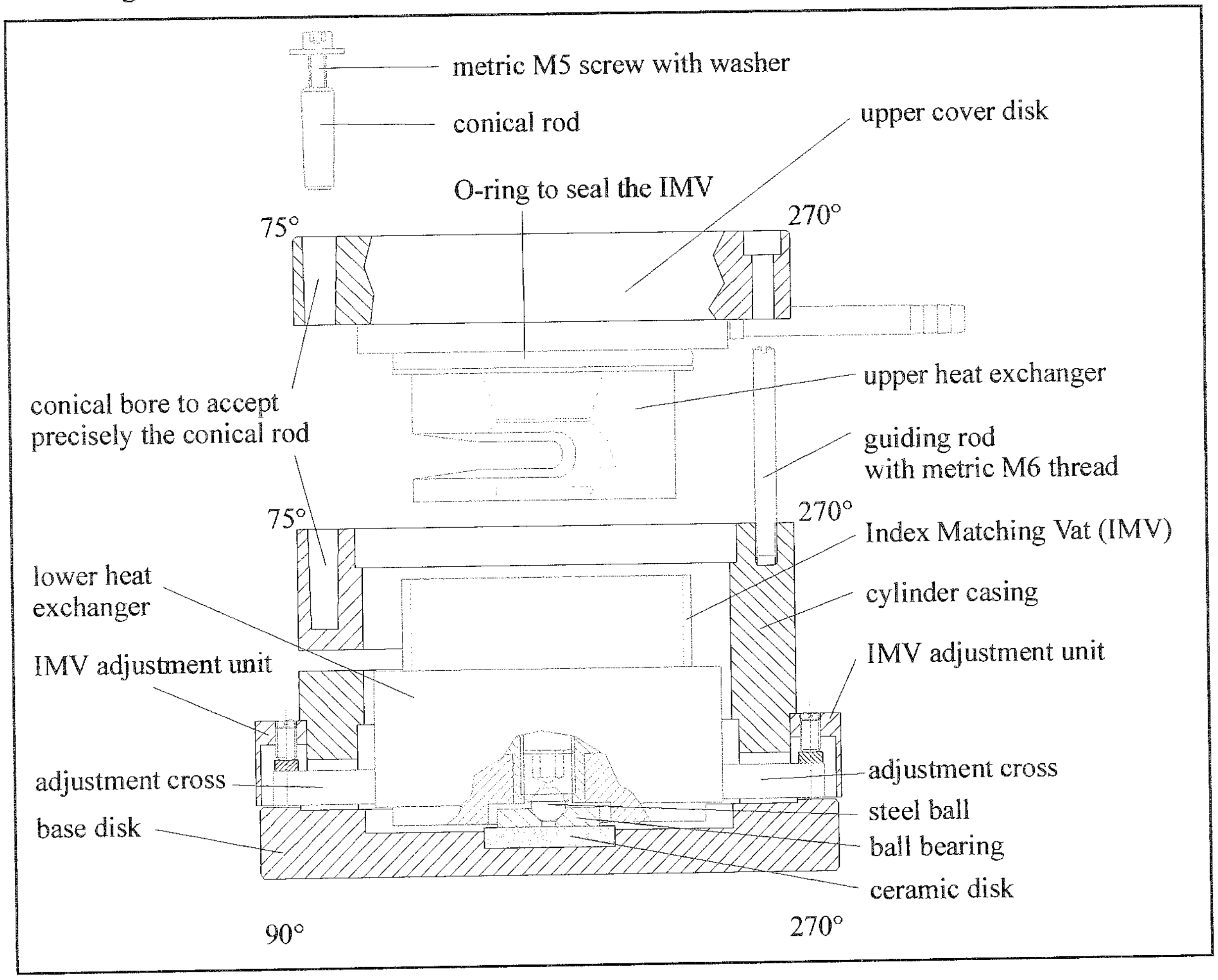
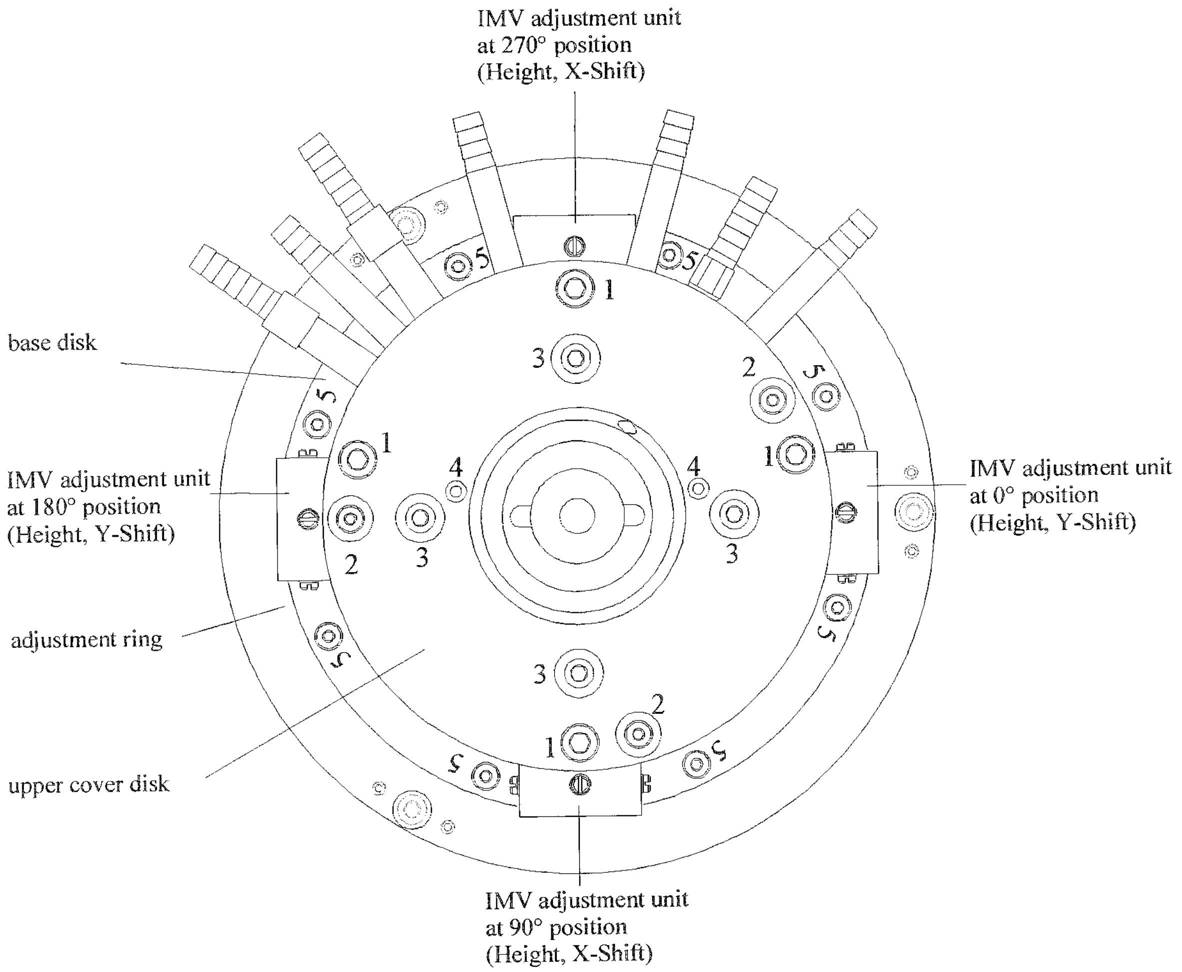


Figure 3:
Section view of the cell housing

The cell housing consists of a base disk, a cylinder casing, the lower heat exchanger with provisions to hold the Index Matching Vat (IMV) and with an adjustment cross to position the IMV, and the upper cover disk with the upper heat exchanger. The base disk of the cell housing is mounted on an adjustment ring (Figure 4). The adjustment ring rests on a separate disk which is fix mounted on the central stand of the goniometer (see Fig. 8).

The upper heat exchanger is mounted on the bottom of the upper cover disk and fastened using 4 off metric M5 screws. The upper cover disk is precisely positioned to the cylinder casing using 3 off conical rods placed in corresponding conical bores in the cover disk and the cylinder casing. The conical rods have a metric M5 internal thread with a metric M5 screw inserted together with a washer. The washer rests on the cover disk and by clockwise turning of the metric M5 screw the conical rod is lifted and can afterwards be removed. Attention! Prior to insert the conical rods unscrew the metric M5 screws such an amount which allows the conical rods to be placed in their final position.

Additionally the cover disk is fastened using 4 off metric M6 screws. To remove the cover disk each of these screws is replaced by a guiding rod with metric M6 thread to avoid any touch between the upper heat exchanger unit and the Index Matching Vat while lifting the cover disk.



1 ... 4 off metric M6 screws
2 ... 3 off conical rods
3 ... 4 off metric M5 screws to fasten the upper heat exchanger (never loosen these screws)
4 ... 2 off bores to accept Pt-100 temperature probe in stainless steel case
5 ... 8 off metric M4 screws to fasten the cell housing on the adjustment ring

Figure 4:
Top view of the cell housing mounted on the adjustment ring

The lower heat exchanger has at the bottom an adjustment cross; its ends are positioned in the corresponding IMV adjustment units. The lower heat exchanger is connected at the center of its bottom to the base disk of the cell housing only via a ball. The ball rests on a ball bearing of hardened stainless steel which can freely move on a ceramic disc which is inserted in the base disk of the cell housing (see Figure 3).

3.2 Preparations

- Insert the Calibration Disk with its ∅ = 10 mm rod into the cuvette holder bore, then tighten the screw ring positioned overhead the cuvette holder. The vertical dial gauge of the Double Dial Gauge Unit is positioned in a spring load mechanism and can be fixed by fasten the knurled knob. If the knob is loosen, the spring mechanism shifts the vertical dial gauge upwards to ensure that the dial gauge is not damaged while mounting the unit. Prior to mounting the Double Dial Gauge Unit on the post of the rotary arm of the goniometer, check the knurled knob to be loosen and if necessary loosen it. Mount the Double Dial Gauge Unit on the post and turn the until the ball tip of the upper dial gauge (PLANE) is placed in the center of the machined track surface (the ball tip is then positioned close to default setting of the program = 45 mm from the rotary axis of the rotary table) (see Figure 5). Fix the unit at this position using the two Allen screws at the end of the unit near the mounting post. Move the vertical dial gauge downwards until a reading of approx. 1.5 to 2.5 mm is set. Fix the dial gauge by fastening the knurled knob.

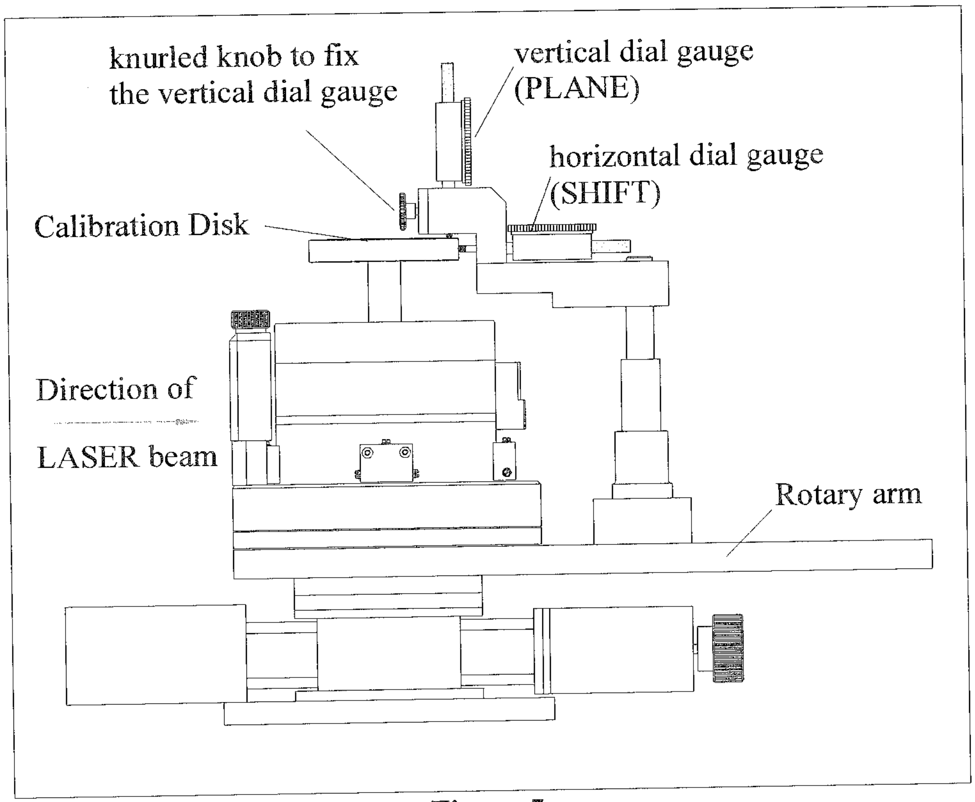


Figure 5:
Calibration Disk and Double Dial Gauges Unit

3.3 Adjustment of the Cell Housing In-Plane with the Rotary Level of the Rotary Table

The level of rotation of the Calibration disk can be adjusted using the height adjustment screws located at the 0°-, 120°- and 240°-position of the adjustment plate of the cell housing. Each adjustment unit consists of two off set screws and one off locking screw centred between the set screws.

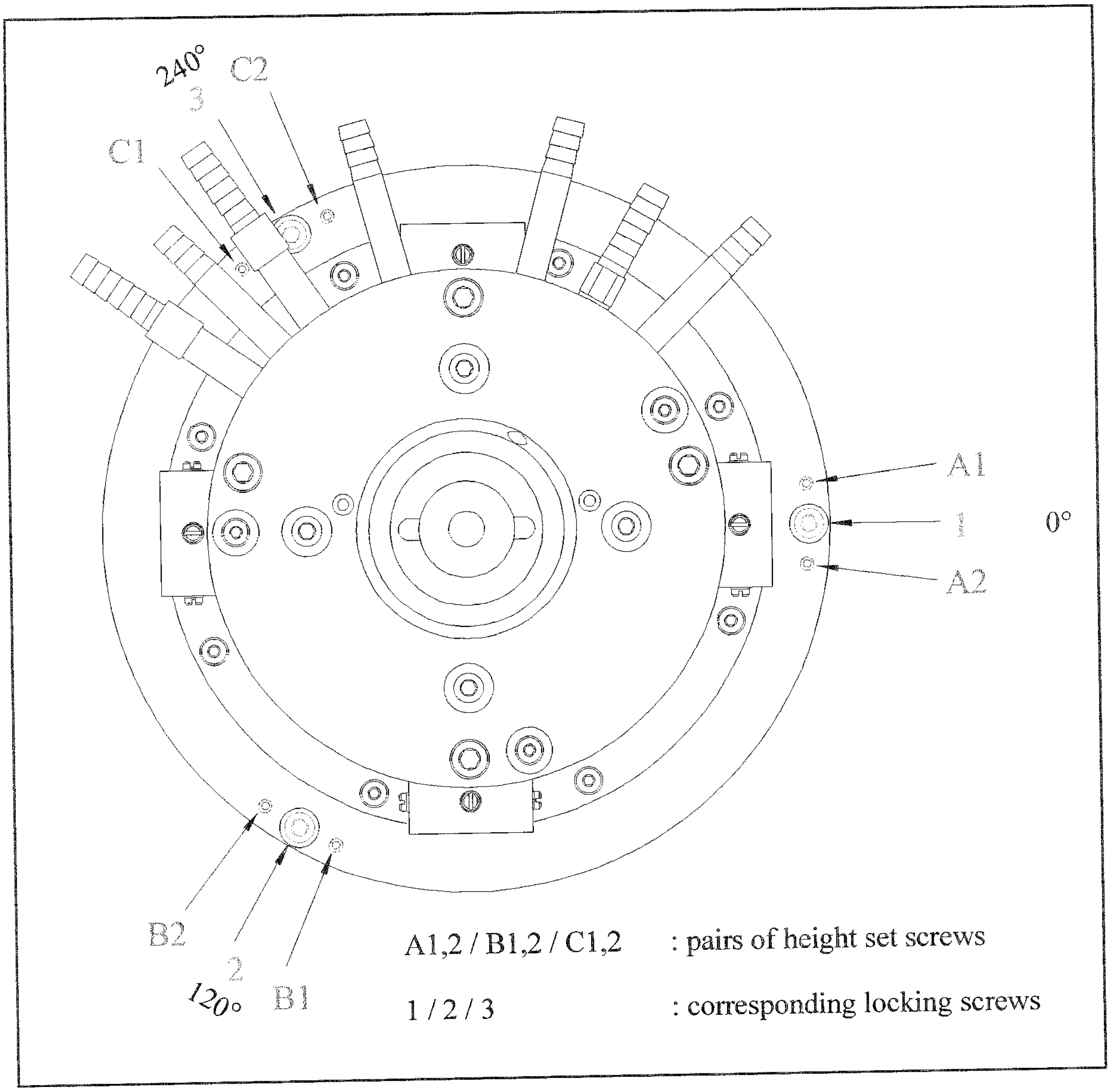


Figure 6:
Adjustment ring with cell housing (screws for PLANE adjustment)

A sectional view of a particular height adjustment unit is shown in Fig. 7. The ball tips of the set screws rest on a plate made of hardened stainless steel, respectively. During height adjustment it must be ensured to adjust both set screws of a particular height adjustment unit such, that the ball tips exert equal pressure on the steel plates. Only in this case no torque or tension is applied to the base plate of the cell housing while the counter screws are fastened. Only this procedure guarantees a long term stability of the adjustment of the cell housing, independent of the measurement temperature set within the specified range.

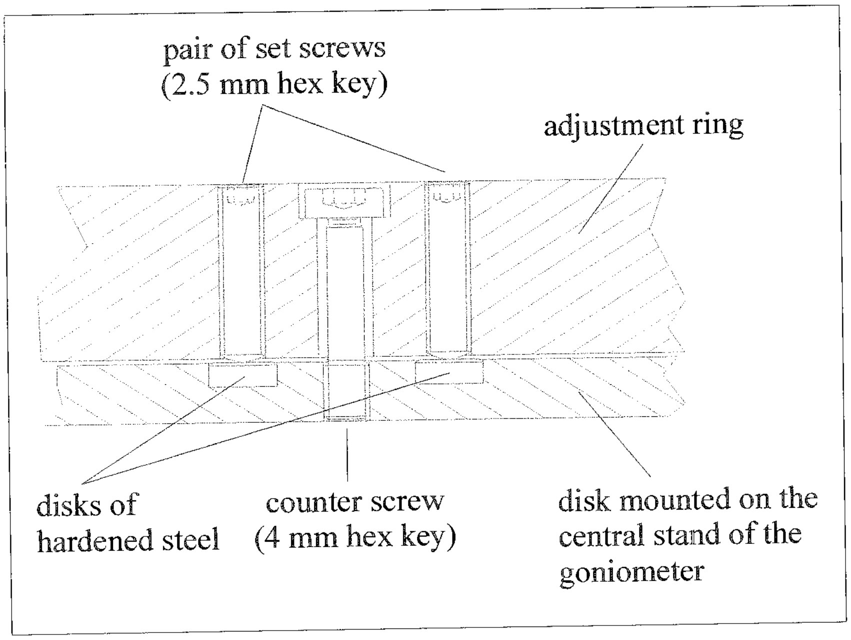


Figure 7:
Sectional view of set and counter screws for height adjustment

- To start the adjustment of the cell housing choose Plane from the Cell Housing Adjustment window of the ALV/WIN-Alignment program.
- Follow the instructions of the program and enter the required readings of the vertical dial gauge.

3.4 Adjustment of the Cell Housing Coaxial to the Rotary Axis of the Rotary Table

The adjustment of the cell housing coaxial to the rotary axis of the rotary table is achieved by positioning the cylindrical base-plate of the cell housing in the X/Y-plane using two adjustment units in 0°-180° and 90°-270° direction (see Fig. 8). The adjustment units allow for orthogonal and not coupled adjustment in the X- and Y-direction. Each of the adjustment units consists of 2 off set screws, one spring, and 2 off locking screws opposite to the set screws and the spring. Prior to start the SHIFT adjustment the locking screws have to be loosened. That pair of locking screws positioned in the present direction of adjustment (screws at 0° position for X-direction, screws at 90° position for Y-direction) should be loosened several turns. That pair of locking screw positioned perpendicular to the present direction of adjustment (screws at 90° position for X-direction, screws at 0° position for Y-direction) acts at the same time as support for the base plate to ensure tilt free motion and must therefore still have to be in gentle touch (free of force) with the surface of the base plate.

Position of the adjustment units at the cylindrical base-plate of cell housing (s. Fig. 8):

X-direction: set screw pair in 180°, locking screw pair in 0°;

Y-direction: set screw pair in 270°, locking screw pair in 90°.

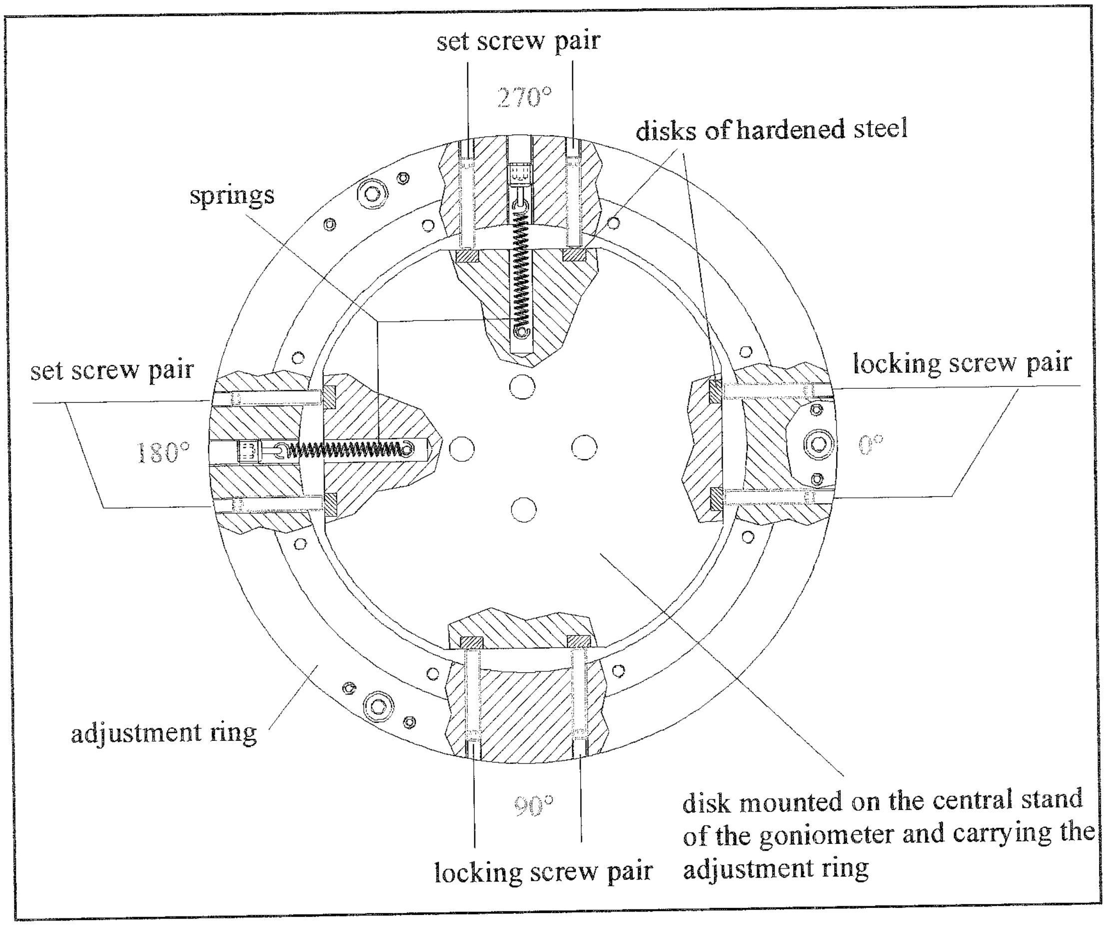


Figure 8:
Sectional view of the adjustment ring with X/Y adjustment units (SHIFT adjustment)

Prior to start the "SHIFT" adjustment loosen the 3 off locking screws (!) of the height adjustment units of the cell housing (PLANE) to allow for shifting the adjustable ring.

- To start the adjustment of the cell housing choose Shift from the Cell Housing Adjustment window of the ALV/WIN-Alignment program.
- Follow the instructions of the program and enter the required readings of the horizontal dial gauge.

A final verification of the reading values of the VERTICAL and HORIZONTAL dial gauges while turning the rotary arm of the rotary table from 0° to 180° terminates the adjustment of the cell housing if the reading value of both dial gauges are constant within the required accuracy of ± 5 µm. Otherwise repeat the adjustment of the cell housing.

Remove the Calibration Disk. Loosen the knurled knob, lift up the vertical dial gauge, loosen the screws which are fixing the Double Dial Gauge Unit, turn the unit approx. By 45° and remove it.

4.0 Adjustment of the Index Matching Vat

4.1 Basic Principle

The index-matching vat (IMV) is positioned inside the lower heat exchanger unit and hold by a clamp with a set spring load torque (see Figure 9). The holding clamp is released by tightening the metric M4 socket set screw, which is positioned in the cell housing case below the hose coupling for the ventilation of the outer surface of the IMV cylinder. To fix the IMV loosen (!) the M4 socket set screw of the holding clamp using a 2mm hex-key.

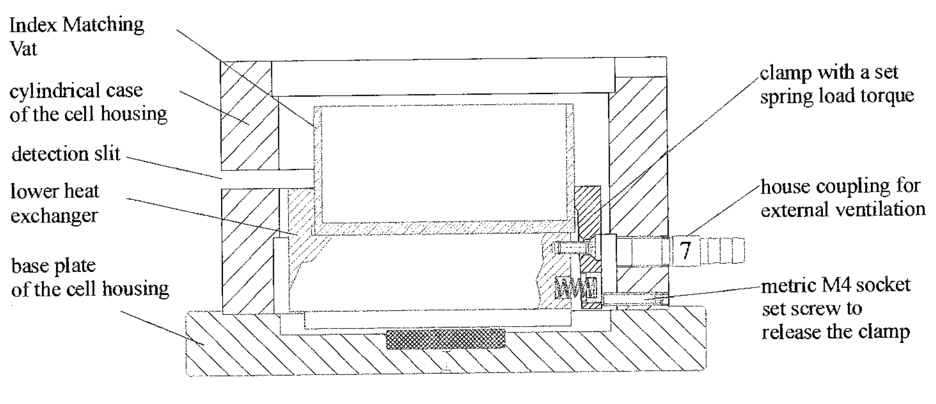


Figure 9:
Sectional view (section plane in 120°/300° direction) of the Index Matching Vat and cell housing without upper cylindrical cover disk

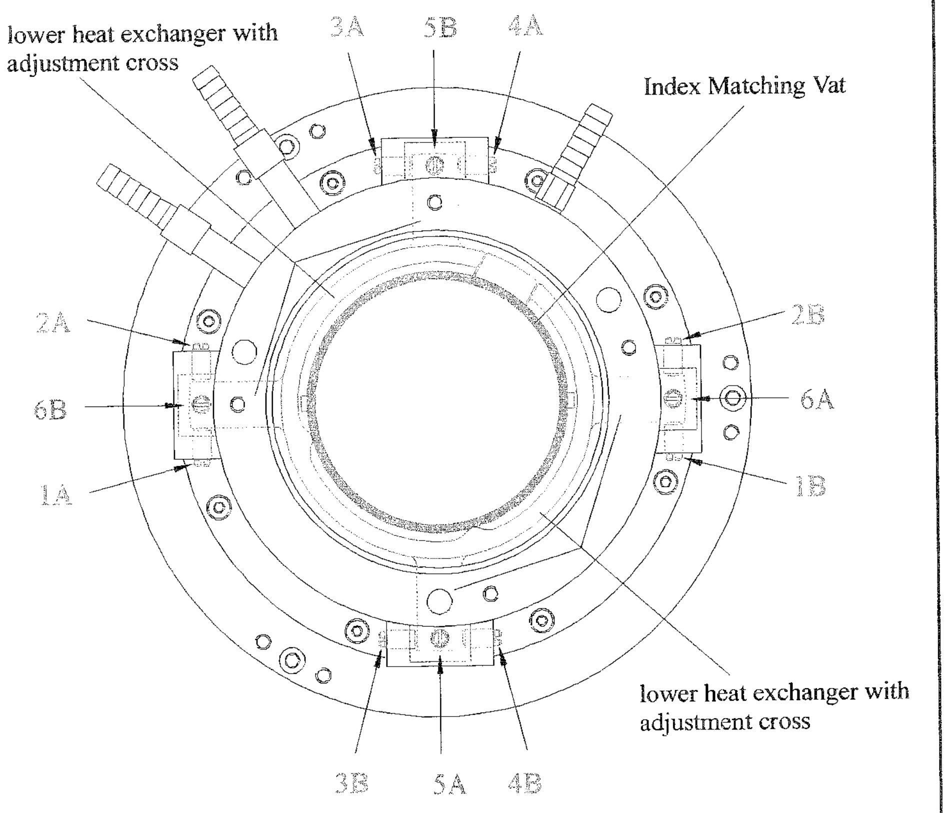
The lower heat exchanger is worked out at the bottom in form of an adjustment cross. Each of the 4 ends of the adjustment cross are hold in an adjustment unit.

This allows for an uncoupled positioning of the adjustment cross in height (PLANE, i.e., adjustment in-plane with the rotary level of the rotary table) and in the X/Y plane (SHIFT, i.e., adjustment of the coaxial coincidence of the axis of rotation of the rotary table with the symmetry axis of the IMV). The X/Y position is set by an appropriate horizontally positioned set/lock screw pairs at each end of the cross and the height is set by 1 off vertically positioned height set screw at each end (see Figure 10).

The 4 off height set screws together are pressing the adjustment cross against the ball connecting the lower heat exchanger to the cell housing If the pressure on to the adjustment cross applied by the height set screws is too low the heat exchanger is not properly fixed and changing the temperature of the cell housing might lead to changes in the position of the heat exchanger and hence to changes in the position of the IMV; if the pressure is too high the adjustment units of the IMV are damaged!

ALV company specifically notes that all warranty or liability claims of the customer are lost, if the adjustment elements are improperly treated !

We therefore explicitly recommend to tighten the adjustment screws of the IMV adjustment cross ***only*** *using a* ***torque-screwdriver****! The torque applied to each of the screws must not exceed* ***0,25 Nm = 25 Ncm !***



SHIFT:

1A,B / 2A,B	: set/lock screw pair for shift in X-direction (0°-180°)
3A,B / 4A,B	: set/lock screw pair for shift in Y-direction (90°-270°)

PLANE:

5A/5B	: height set/lock screw pair (90°)
6A/6B	: height set/lock screw pair (0°)

Figure 10:
Top view on the IMV and cell housing without upper cylindrical cover disk

The IMV adjustment tool consists of a holder with one dial gauge (VERTICAL) and one feeler gauge (HORIZONTAL); both gauges are mounted on a plate which can be tilted along a rotary axis. The plate with the two gauges is lifted upwards in case the two knurled screws are not locked by a built in spring mechanism, and in contrary the plate can be hold down and be locked in the measuring position by tightening the two knurled screws. Fig. 12 shows the working position of the IMV adjustment tool, mounted on the post of the rotary arm.

4.2 Preparations

The IMV is an highly accurate optical device, in fact it is a cylindrical lens, with polished surfaces and multilayer coated flat windows. Handling of the IMV requires the same precautions as handling of lenses and mirrors. ***Never*** *touch the IMV with bare hands,* ***always*** *protect the surfaces by use of lens-paper. Clean the IMV using an appropriate solvent (e.g., methyl ethyl ketone (2-butanone) or toluene) with the same careful treatment as required for lenses etc.. Never use any surfactant type glass cleaning agents (e.g., surfactants, metal-organic complex forming agents), that agents remove the multilayer coating from the windows.*

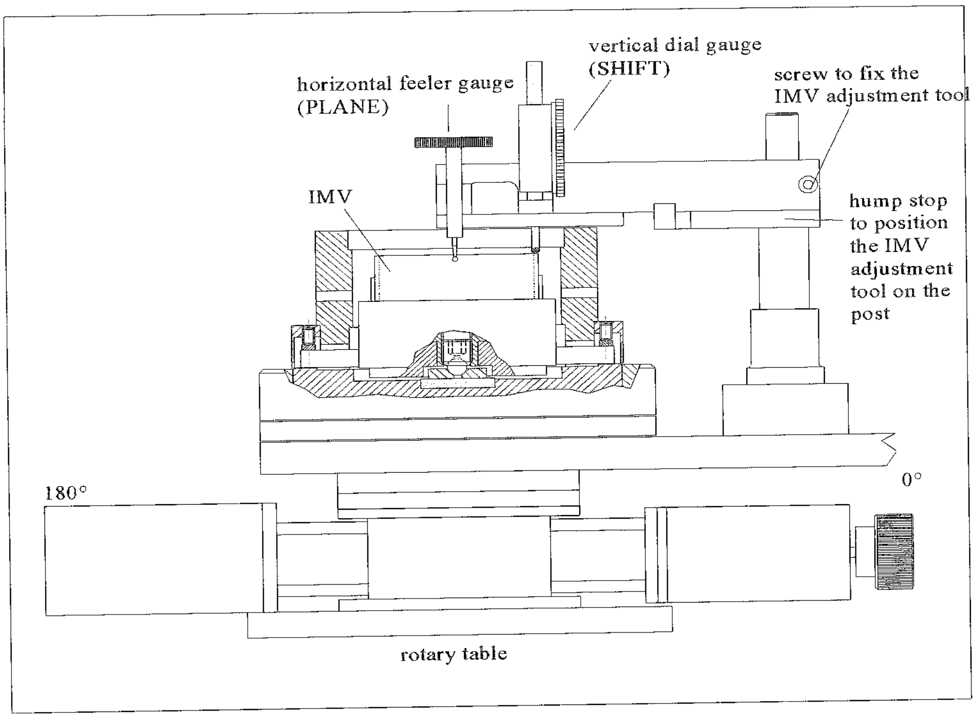


Figure 11:
Working position of the IMV adjustment tool

- Loosen the 3 off conical rods with a metric M5 internal thread by clockwise turning of a metric M5 screw inserted together with a washer in the internal thread. Remove the conical rods. Loosen and remove the 4 metric M6 screws. Then remove the upper cylindrical cover disk with the upper heat exchanger and place the IMV in the holder of the lower heat exchanger, such that:
  - the "ALV" logo of the IMV is directed toward the detection slit of the cell housing,
  - the outer cylinder of the IMV is hold in position against the 2 off polished rods in the holder, and the front window of the IMV is positioned in 180°, towards the laser.

  Fasten the IMV by loosening the screw of the holding clamp.
- Make certain the two knurled screws of the IMV adjustment tool are loose and the plate with the two gauges is lifted upwards, respectively tilted approximately 45° (see Figure 12, position 1). Provided the plate with a hump stop is already fixed positioned on the post of the rotary arm, then position the IMV adjustment tool on to the post of the rotary arm in the appropriate clamp hole of the unit and let it rest on top of the plate with a hump stop. Then turn the IMV adjustment tool until it locks against the hump stop, which is the defined position at which the IMV adjustment tool should be fixed positioned by tightening the M5 screw, that is supposed to clamp it to the post.
- Carefully tilt the plate with the two gauges downwards to the IMV, watch out for both dial gauge tips and make certain that: (see Figure 12)
  - the tip of the feeler gauge (HORIZONTAL) under all circumstances moves <u>inside</u> the vat, and does not (yet) touch the inner surface of the vat cylinder. If however during the downwards tilting it clearly appears the tip of the feeler gauge will either contact the rim of the vat cylinder, or even moves outside the vat, then immediately interrupt the downwards tilting. Don't hesitate to turn the feeler gauge into the appropriate position, the tip can easily be turned in it's support/friction clutch.

- the tip of the dial gauge (VERTICAL) is positioned in the center of the rim of the vat cylinder,
- **both** dial gauges are finally appropriate positioned to accept deviations of the reading value from the mean value in both directions.

- Finally lock the two knurled screws (see Figure 12, position 2).

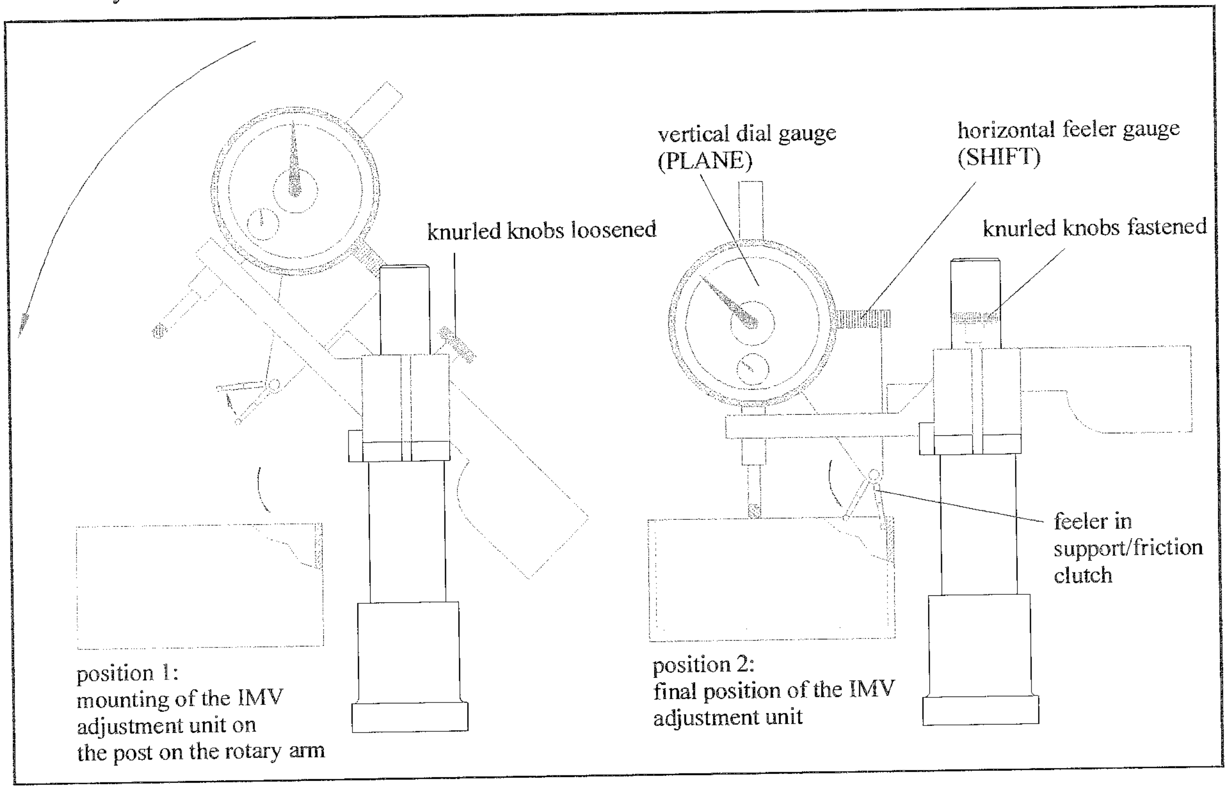


Figure 12:
Positioning of the IMV adjustment tool

4.3 Adjustment of the IMV In-Plane with the Rotary Level of the Rotary Table

- To start the adjustment of the cell housing choose Plane from the IMV Adjustment window of the ALV/WIN-Alignment program.
- Follow the instructions of the program and enter the required readings of the vertical dial gauge.
- Set the appropriate adjustment screws as indicated by the program.
  - PLANE adjustment of the IMV ***using a torque screwdriver***, set to a torque of 0.25 Nm:

    Loosen the height set screw of the adjustment unit that is indicated by the program. Turn the opposite height set screw clockwise controlled by reading the dial gauge until the reading is advanced by approx. 5-10 µm beyond the desired reading value; while screwing clockwise this screw it may be necessary to loosen the screw opposite to this one, in order to allow for the required movement, which may otherwise be blocked. Intermittently tighten both height set screws using the set torque and starting with that screw that has been loosened before until the desired reading value of the dial gauge is received. Verify that all screws are tighten with the same torque.

  - PLANE adjustment of the IMV ***without using a torque screwdriver***

    ⇒ Loosen the height set screw of the adjustment unit that is indicated by the program.

⇒ Then turn the opposite height set screw clockwise controlled by reading the dial gauge until the reading is advanced by 10 µm beyond the desired reading value; while screwing clockwise this screw it may be necessary to loosen the screw opposite to this one, in order to allow for the required movement of 10 µm, which may otherwise be blocked.

⇒ Up to this point one aim of the adjustment is to proceed with movements of the adjustment cross without remaining torque, in order to achieve a clear defined positioning, from which the final positioning will be adjusted and a definite amount of torque will be affixed.

⇒ The final positioning is started using that height set screw which is opposite to that one used to set the reading of the dial gauge. Turn this height set screw clockwise controlled by reading the dial gauge until the reading is advanced by 5 µm beyond the desired reading value

⇒ Finally turn the previously used height set screw clockwise until the reading value of the dial gauge is set exactly to the desired value.

⇒ This procedure ensures a reproducible torque and a proper tightening of the adjustment cross.

- Repeat the PLANE adjustment until the required accuracy is achieved.

4.4 *Adjustment of the IMV Coaxial to the Rotary Axis of the Rotary Table*

- To start the adjustment of the cell housing choose Shift from the IMV Adjustment window of the ALV/WIN-Alignment program.
- Follow the instructions of the program and enter the required readings of the horizontal feeler dial gauge.
- Set the appropriate adjustment screws as indicated by the program.
  - SHIFT adjustment of the IMV ***using a torque screwdriver***, set to a torque of 0.25 Nm:

    Slightly loosen the horizontally positioned screws of the appropriate adjustment unit that are indicated by the program. The loosened screws acts further as lock screws. Intermittently turn both set screws to the same amount, and loosen the locking screws if the set screws cannot be turned further with the applied torque of the screwdriver. Repeat this procedure until the desired reading value of the dial gauge is received and tighten the lock screws. Verify that all screws are tighten with the same torque.

  - SHIFT adjustment of the IMV ***without using a torque screwdriver***:

    The procedure is the same as described in the previous chapter. Make certain to turn the corresponding screws of opposite adjustment units by the same amount and to loosen the lock screws by turning the same amount. Finally tighten the lock screws and verify that all screws are tighten with approximately the same torque as applied for the height set screws.

- Repeat the SHIFT adjustment until the required accuracy is achieved.
- A final verification of the reading values of the VERTICAL and HORIZONTAL dial gauges while turning the rotary arm of the rotary table from 0° to 180° terminates the adjustment of the IMV if the reading values of the dial gauges and the feeler gauge are constant within the required accuracy of ± 5 µm. Otherwise repeat the adjustment of the IMV.

- Remove the IMV adjustment tool from the post, release the holding clamp by tightening the metric M4 screw and take the IMV out of the cell housing.
- To proceed on further with the alignment of the laser beam the IMV has to be removed again. The IMV can be repositioned within the accuracy of the adjustment if it is ensured that the contact area between the bottom of the vat and the adjustment cross is clean and free of dust contamination.

4.5 Installation of Heat Exchanger Liquid Circulation and External Filtering Hoses

The Figures 9 and 13 show the hose couplings of the cell housing for connecting the hoses of the heat exchanger liquid to and from the waterbath thermostat circulator, as well as for connecting the hoses to an external filtering device of the index matching liquid.

Circulation of organic solvents (e.g. toluene) results in electrostatic charging of the components due to the low electrical conductivity of the solvents, therefore a careful electrical "grounding" of all parts (including the inner parts of the hoses and all other non-conducting devices used, e.g., heat exchanger made from glass) of the external filtering device is required to avoid the existing danger of explosion.

Position:

Upper cylindrical cover plate with upper heat exchanger:	hose coupling 1 - 4, 8
Cylindrical case of cell housing with lower heat exchanger:	hose coupling 5 - 7

Purpose:

External filtering of the index matching liquid:	hose coupling 1 and 4
heat exchanger liquid from the waterbath thermostat:	hose coupling 2
heat exchanger liquid to the waterbath thermostat:	hose coupling 3
heat exchanger liquid from upper to lower heat exchanger	hose coupling 5 and 6
for ventilation of the outer surface of the IMV cylinder:	hose coupling 7 (Figure 9)
overflow of index matching liquid:	hose coupling 8

The heat exchanger liquid circulates via the hoses from the waterbath thermostat circulator into the hose coupling 2 of the upper heat exchanger, then out of hose coupling 3 via a short hose (tube) to hose coupling 5 of the lower heat exchanger, and then out of hose coupling 6 back to the waterbath thermostat circulator. This direction of flow of the heat exchanger liquid, from the thermostat circulator into the upper heat exchanger and via the connection of the couplings 3 and 5 into the lower heat exchanger and then back into the waterbath thermostat circulator, suppresses convection due to gravity in the cuvette at temperatures of the heat exchanger liquid higher than room temperature.

When attaching tubes on to the hose couplings one has to be aware that force will be added to the cell housing, which off-sets the adjustment of the cell housing, even if compensated by an external counter force - one should expect the adjustment as provided exactly ***within ± 5 µm*** *cannot withstand external force. Therefore applying too much force to the hose couplings may almost certainly change the position of the cell housing, when attaching the tubes to or removing them from the hose coupling.*

Be advised to act most carefully using the lowest force possible whenever any action is required to the cell housing or any part of, that may change its optimum adjustment. In any *case a control measurement to ensure the correct positioning of the cell housing is required by repeating the steps described under chapter* ***3.0****.*

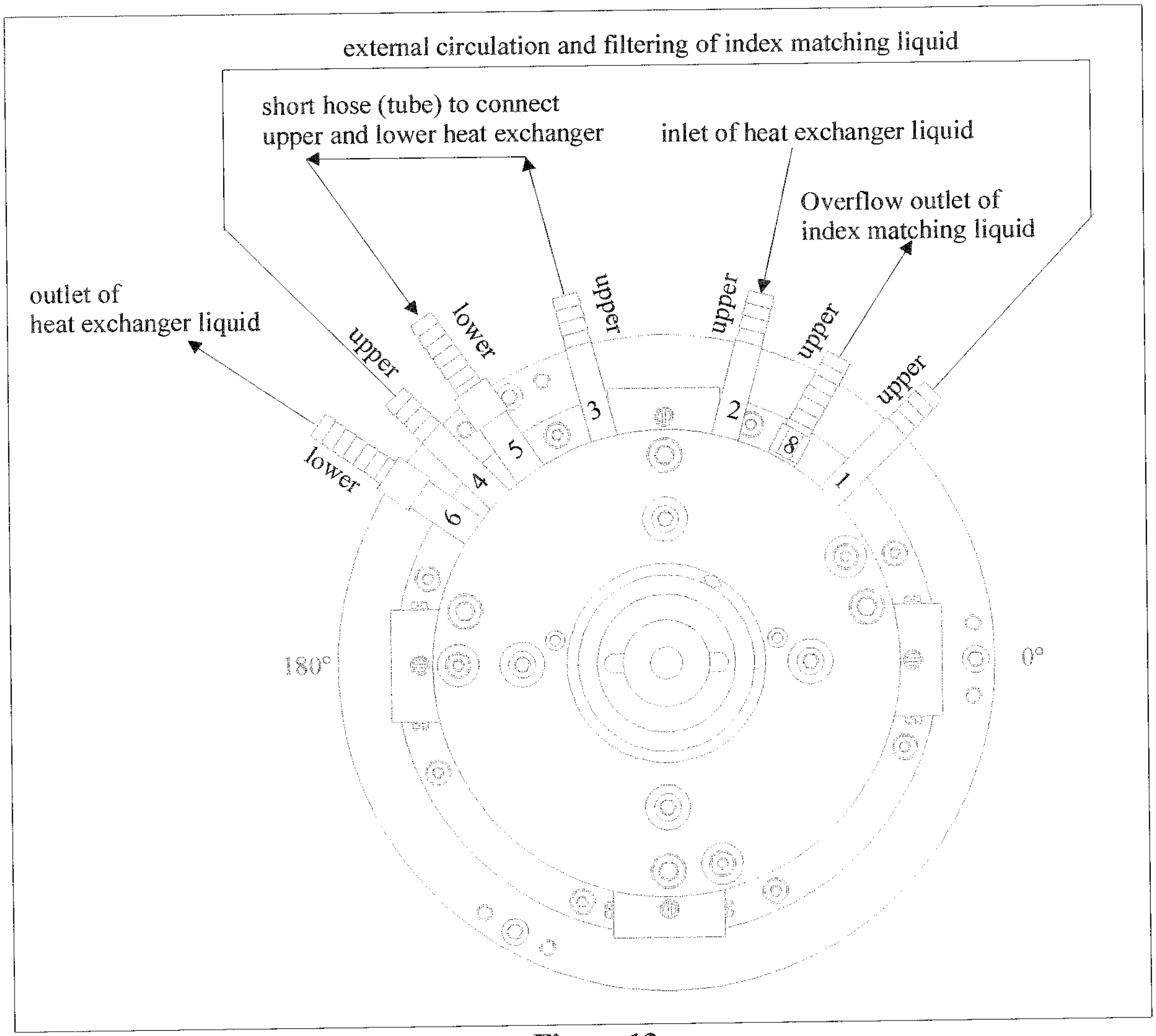


Figure 13:
Top view on the cell housing with hose couplings

To ensure safe operation it is mandatory to use appropriate clamps, worm driven hose clips, tubing clips or steel-band clips with hex-screw to fasten the approx. 7 mm inner diameter tubes to the hose coupling of the cell housing.

Attention! *It is mandatory to use the common measures for stress relief of the tubes to avoid steady strain to the cell housing, which may otherwise lead to a gradually mechanical displacement of the cell housing.*

5.0 Adjustment of the Laser Beam

5.1 Remarks

The following part of the adjustment procedure require the operation of the laser without the beam protection units of the compact goniometer systems. Therefore uncontrolled reflected or scattered light of high intensity may arise and may lead to damages or even irreversible damages of human eyes.

The compact goniometer as being a system used for scientific research purposes can not be built or operated as a system housed in a totally closed compartment!

Adjustment of the laser beam to optimum position requires operation under open beam condition, and using the appropriate adjustment tools.

Only a person ***CERTIFIED*** *to operate the laser in accordance to the safety regulations legally and otherwise in effect in your country can be assigned to perform the adjustment of the laser beam. A person* ***CERTIFIED*** *to operate the laser and assigned by the appropriate Official (an Official in charge and in power to assign the operation of the laser to such a certified person) to operate the laser, is solely and fully responsible for the operation of the laser.*

Depending on the laser classification differently drastical regulations apply; the appropriate laser safety label on the laser specifies the CLASS of the laser.

Strictly follow all regulations and instructions, and use laser goggles

Take all required precautions! *Before opening the laser shutter it is mandatory required to ensure that:*

- *the laser beam will not hit other surfaces than the ones the person in charge has chosen or positioned appropriately by control of his good judgement,*
- *the laser cannot hit OTHER reflective surfaces, or obstacles producing uncontrolled reflections,*
- *the laser cannot produce parasitic reflection elsewhere.*
- *the laser is operated with absolute minimum power the experiment, or any adjustment action allow.*

5.2 Pre-alignment of the Laser Beam

- An iris is mounted in an adjustable holder, which is mounted on a black anodized protection tube, that is positioned at the laser beam entrance of the Laserbeam Folding/Adjustment Unit. Position the laser on the laser stand starting with fully opened iris (!) in HORIZONTAL direction, until the laser beam hits the center of the first laser mirror (S_1). Successively reduce the aperture of the iris and position the laser to pass through the aperture while hitting S_1 at its center. If the laser/laser stand has a height adjustment, by the same procedure position the laser beam also in VERTICAL direction to the centre of S_1. Lock the laser and the laser stand in this position.

- Adjust the stop position of the iris, i.e. the smallest accessible aperture diameter of the iris without diffraction of the laser beam (set by a stop screw at the iris holder).

- Position the micrometer controlled sliding-stage of the second laser mirror (S_2) to a reading value of the micrometer of 7.500 mm. Align the mirror S_1 by tilting and turning the mirror holder using the appropriate screws of the gimbal to reflect the laser beam in the center of the mirror S_2.

*The lower (right) adjustment screw turns the laser beam in HORIZONTAL direction, the upper (left) adjustment screw tilts the beam in the VERTICAL direction (see Figure 14); both adjustments are **not** coupled! This applies to **all** gimbals !*

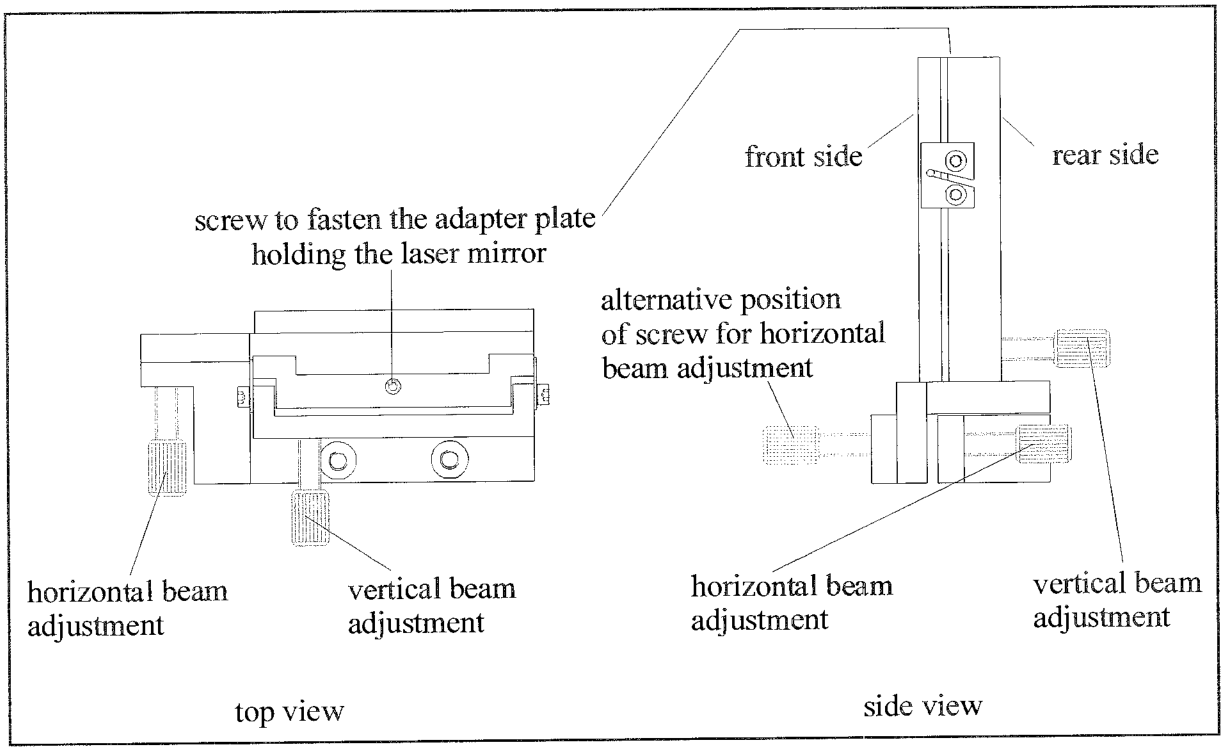


Figure 14:
Top and side view of the gimbal holder

- Open both iris apertures mounted on the Monitor Diode/Attenuator Unit. Position mirror S_2 (only using the adjustment screws of the gimbal of S_2, **not** the micrometer controlled sliding stage of S_2) and if necessary the vertical adjustment of the mirror S_1 until the beam passes both iris apertures and the cell housing (the beam stop at 0° position is removed). Successively reduce the aperture diameter of the iris directed toward the cell housing and position the laser beam symmetrically to this iris.

5.3 Alignment of Projection Optics System

The final procedure to adjust the laser beam position requires the use of the adjustment apertures, the Centricity Definition Needle (CDN), the Laserbeam Height Definition Needle (LHDN) and the Double Needle Adjustment Unit (DNAU). These tools are used to estimate the position of the laser beam by the diffraction pattern caused by the needles (as obstacles) in a laser beam with Gaussian beam profile. The accuracy of positioning of the needle depends strongly on the laser beam diameter and it generally holds true, that the thinner the beam diameter the more accurately the needle can be positioned in the center of the laser beam. The needle is correctly positioned to the laser beam if a symmetric diffraction pattern is observed, i.e., if both minima neighbouring the maximum of 0. order are in respect to height and intensity (darkness) identical. This applies also to maxima and minima of higher order. To watch the symmetry of the diffraction pattern, a magnified image of the laser beam is projected in the far field. The projection lens used is mounted in a gimbal holder which allows for adjustment of Y/Z position and tilting. The gimbal holder is mounted on a dovetail-sliding stage for a reproducible positioning, which allows a fast change between a magnified and a non-magnified far field projection of the laser beam.

To keep the aberration effect of the magnifying lens to a minimum the lens must be exactly adjusted perpendicular and also coaxially to the laser beam entering this lens. Therefore first remove the projection lens with the dovetail-sliding stage from the beam path (**Use laser goggles!**). Then align the crosshairs of the projection screen to the beam either by moving the sheet of paper or by using the projection mirrors of the (optional) 90°- or 270°-beam folding unit.

Projection screen: at least a 500 X 500 mm paper with a visible crosshairs with scales on all axis'.

Move the projection lens back into the beam path using the dovetail-sliding stage and adjust the Y/Z position of the projection lens using the two micrometer-controlled sliding stages until the magnified laser beam is centred to the crosshairs on the projection screen.

Finally correct the tilting of the projection lens using the two knobs at the front side of the gimbal. Tilt the lens until the interference fringes, which can easily be watched on the cell housing, are symmetrically positioned to the laser beam.

Check the proper position of the projection lens always after an adjustment step of the laser beam since any angular adjustment of the laser beam also displaces the beam entry into the lens. If necessary correct the position.

5.4 Check of the Beam Profile and the Polarisation Plane of the Laser

Observe the far field projection of the magnified laser beam with as well as without the laser goggles, when **only** the projection lens is placed in the beam path. Make certain that no apertures cause diffraction of the laser beam. The laser can be used with a CGS **only** when the following observations are true without exception:

- the laser must operate in TEM_{00} mode,
- the laser beam must have a Gaussian (circular) intensity profile,
- **no** deformations or shadings of the (circular) beam profile as well as in the circular diffraction pattern are observed.

Position a calibrated polarisation filter in the laser beam path to determine the polarisation plane of the laser. The laser can only be used in the CGS if the polarisation plane deviates from the vertical direction less than ± **1,5°** !

The laser must be VERTICALLY polarised to be used in the Compact Goniometer System !

5.5 Adjustment of the Laser Beam using the adjustment apertures

Further laser beam alignment is achieved using the CDN and LHDN and the adjustment apertures of the Detection Optics System:

⇒ *Fiber Optical Detection System:* adjustment aperture (black anodized disk with outer diameter of 25 mm and an aperture diameter of 2.5 mm) inserted in to the Fiber Adjustment Holder front side (toward the cell housing).

⇒ *ODS-125 (fixed pinholes):* two adjustment apertures positioned in the front and rear plate of the ODS-housing.

⇒ *ODS-125 II (variable pinholes):* one adjustment aperture inserted in the lens adjustment unit and one adjustment aperture screwed in the P_1 pinhole holder

- Insert the CDN and turn S_2 until a symmetric diffraction pattern is observed on the screen.
- Turn the rotary arm of the rotary table to the 0° position and turn S_1 and S_2 until the diffraction pattern of the CDN passes symmetrically through the adjustment aperture of the Optical Detection Unit. Turn the arm to 180° position and check the beam position and correct it if necessary.
- For the final adjustment of the laser beam mount the Double Needle Adjustment Unit on the end of the rotary arm of the goniometer and fix the unit with the 2 off metric M6 Allen screws. Remove all adjustment apertures.

5.6 Final Adjustment of the Laser Beam

The final adjustment is performed using the superimposed diffraction pattern of two needles, which is an extremely sensitive procedure to align the laser beam position. Please note, that the resolution of the diffraction pattern of each needle is a function of the beam diameter at the position of the needle tip, divergence, distance to the projection lens and overall distance to the screen. Therefore, the needle at 180° has the best resolution, which can immediately be seen from the diffraction pattern.

5.6.1 Horizontal Position

To adjust the laser beam use only the ALV/WIN Alignment buttons 'GOTO 0' or 'GOTO 180' to turn the rotary arm. These commands ensure to overcome and eliminate the backlash of the rotary table. The final accuracy of the angular position is better than 1/3000°.

- Insert the CDN and adjust the laser beam until a symmetric diffraction pattern is observed on the projection screen.

Due to the high intensity of the laser light reflected from the needles
never look at the needles, not even with laser goggles !

- Enter the Beam Adjustment window, start the Horizontal alignment and follow the instructions of the program.
- The final reading value of the micrometer is stored by the program to verify the alignment later on.

5.6.2 Laser Beam Height

- Insert the LHDN and adjust the laser beam until a symmetric diffraction pattern is observed on the projection screen.

- Enter the Beam Adjustment window, start the Horizontal alignment and follow the instructions of the program.

The adjustment of the laser beam in VERTICAL direction (i.e. in-plane with the rotary level of the rotary table) __at__ a given height, defined by the Laserbeam Height Definition Needle (LHDN) can only be achieved by an adjustment of the tilt of both mirrors S_1 __and__ S_2:
Tilting of mirror S_1 changes the height at which the laser beam hits the mirror S_2 and tilting of the mirror S_2 allows for adjustment of the laser beam in-plane with the rotary level at this height.

- The final reading value of the micrometer is stored by the program to verify the alignment later on.

5.7 *Installation of the Beam Splitter Plate and the Monitor Diode*

- The monitor photodiode is a four quadrant photodiode used to measure beam intensity and position. Therefore a small part of the laser beam is reflected from a beam splitter plate, expanded using an appropriate lens and adjusted symmetrically to the four quadrants. The monitor diode measures very sensitive the beam position and indicates directly a misalignment of the laser beam. It is also very helpful for re-alignment purposes provided the position of the lens has not been changed after a perfect alignment of the laser beam was achieved.

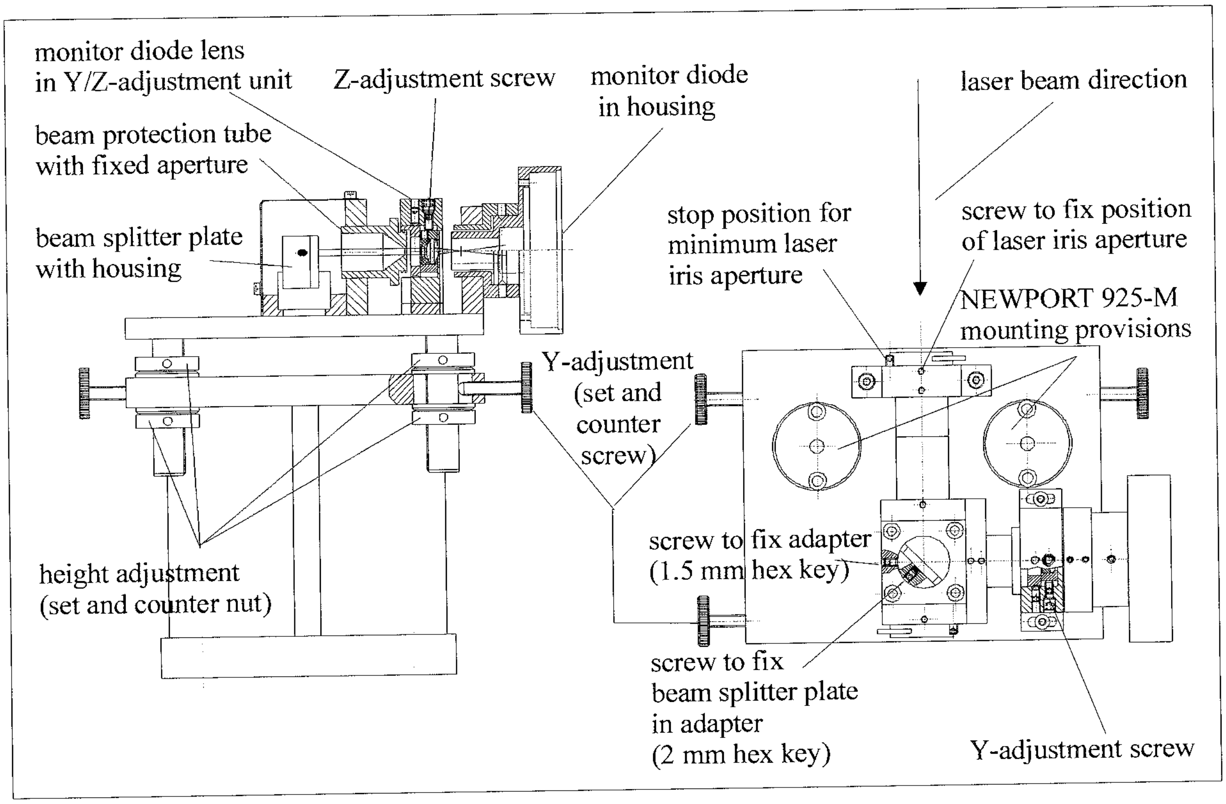


Figure 15:
Monitor Diode/Attenuator Adjustment Unit

- The beam splitter plate and monitor photodiode are positioned in the Monitor Diode/Attenuator Unit (see fig 15). The unit allows for adjustment in Y/Z position and also for slight tilt corrections. Adjustment of the Monitor Diode/Attenuator Unit is performed by positioning the two iris apertures as part of the unit symmetrically to the beam. Without the beam splitter plate only that iris aperture directed toward the cell housing has to be adjusted.

- Prior to insert the beam splitter plate the monitor diode housing is removed from the 4-rod holding system. The lens remains inserted in the Y/Z adjustable holder.
- The beam splitter plate is inserted in the slit of the cylindrical holder and fixed by a metric screw M4 (2 mm hex key), positioned perpendicular to the slit. The cylindrical holder itself rests in the base plate of the beam splitter housing and is fixed by a metric screw M3 (1.5 mm hex key). Loosing this screw allows for rotation of the cylindrical holder with beam splitter plate to position it such that the reflected beam is centred on to the fixed aperture. The position of the beam with respect to the fixed aperture can easily be obtained on a screen appropriately positioned in the path of the reflected beam approx. 15 cm behind the expanding monitor diode lens. The second reflected beam (originating from the second surface of the beam splitter plate) is blocked by the fixed aperture, and therefore not visible on the screen. Ensure to use for monitoring the reflected beam from the surface of the beam splitter plate directed toward the laser and to block the beam originating from the surface directed toward the cell housing.
- Inserting the beam splitter plate results in a parallel horizontal shift of the beam position which is corrected using the sliding stage of the mirror S_2. The correct beam position is checked using the CDN and vertical needle of the DNAU (the horizontal micrometer must be set to the recorded value). After correction of the beam position a slight rotation of the position of the holder with the beam splitter plate is necessary for optimum positioning of the reflected beam with respect to the aperture. Finally, a check and if necessary correction of the laser beam position has to be performed using the CDN and vertical needle of the DNAU.
- Check and if necessary correct the position of the iris aperture which is directed toward the laser to be symmetrically to the laser beam. Attention! Adjusting the iris aperture affects the position of the beam splitter plate and the position of the second iris aperture directed toward the cell housing. A subsequent rotation of the beam splitter plate and adjustment of the second iris aperture may be necessary. A final check of laser beam position is necessary in case the beam splitter plate has to be repositioned.
- The monitor diode housing is mounted to the 4-rod holding system and fixed using the appropriate screws and the beam is centred to the photoactive area of the diode by shifting the lens horizontally and vertically (Y/Z) to the beam direction. The monitor diode output is displayed by the ALV-5000 program (type 'dio' and press enter). In case of symmetrical illumination of the photoactive area equal readings are observed for the four diode channels.

5.8 Installation of the Laserbeam Optimisation Unit (only Fiber ODS)

- Insert the lens in the Laserbeam Optimisation Unit. The achromatic lens has to be inserted with the biconvex side towards the laser. Depending on the focal length of the lens used mount the unit in the appropriate distance to the cell housing at which the waist point of the focused laser beam is observed at the center of the cell housing (distance is about 20% larger than the nominal focal length of the lens and depends on beam divergence).
- Insert the CDN and correct the beam displacement by shifting the lens in Y direction using the appropriate screw of the Laserbeam Optimisation Unit until a symmetric diffraction pattern is observed.

- Insert the LHDN and correct the beam displacement by shifting the lens in Z direction using the appropriate screw of the Laserbeam Optimisation Unit until a symmetric diffraction pattern is observed.
- Cross-check again for beam displacement using the CDN. Check the position of the lens and correct it if necessary.

6.0 Optical Detection System

Attention: *Use only the the ALV/WIN Alignment buttons 'GOTO 0' or 'GOTO 180' to turn the rotary arm. (confer 5.4)*

6.1 Adjustment of the Fiber Optical Detection System

Prior to starting the installation of the Fiber and the SO-SIPD carefully read the instructions "Handling and Characteristics of Fibers with Integrated Collimation-Detection-Optics" and "Safe Operation. of ALV/SO-SIPD/DUAL Photomultiplier Detection Unit".

Make certain to attenuate the laser beam intensity below the 'safe level' of 2mW at 633 nm, respectively 1 mW at 488 nm.

- Turn the rotary arm to the 0° position. Adjust the Y/Z position such that the laser beam passes symmetrically through the center bore of the adapter plate that accepts the optical fiber.

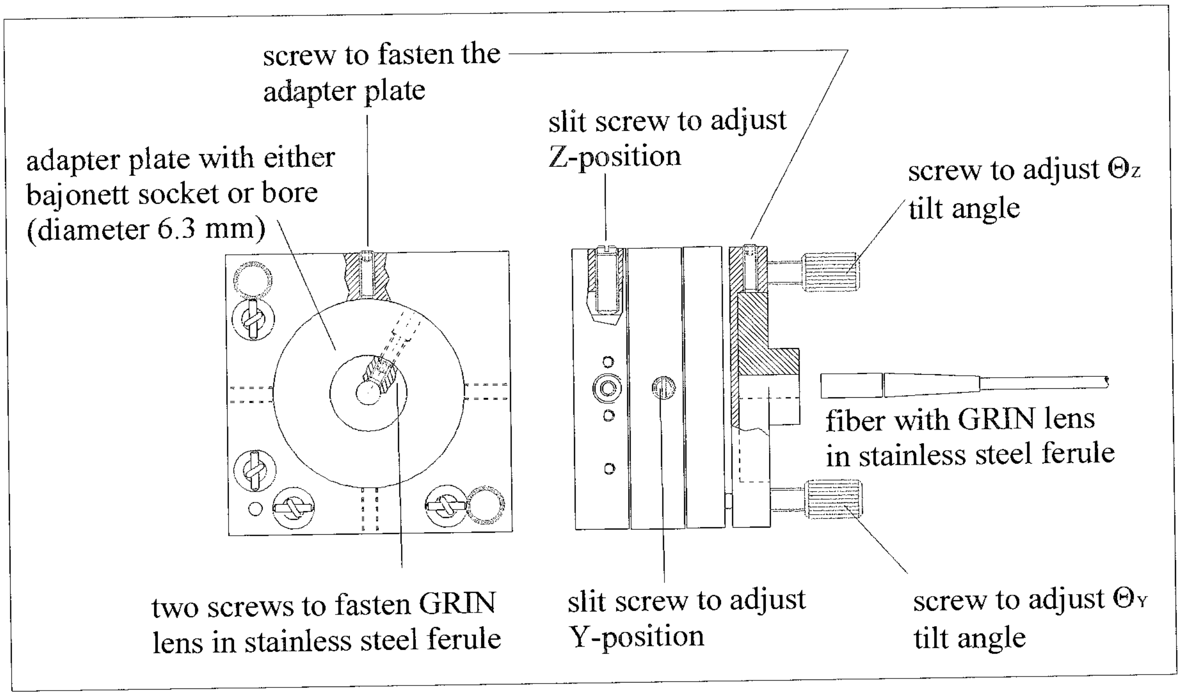


Figure 16:
Fiber Adjustment Holder

- Connect the detection fiber (either bayonet cable plug or GRIN lens in stainless steel ferule) to the Fiber Adjustment Holder (see Figure 15). Adjust the $\Theta_{Y/Z}$ tilt angles until light is transmitted through the fiber. Once transmission of light is observed the intensity is maximised by final adjustment of the Y/Z position and the $\Theta_{Y/Z}$ tilt angles.

- Turn the rotary arm to approximately 60°.
- **Activate the Hall-limit switches.**
- Mount the SO-SIPD/DUAL Photomultiplier Detection Unit on the rotary arm and fix the unit from the bottom of the rotary arm using 2 off metric Allen screws.
- Connect the SMA or FC connector of the fiber to the SMA or FC socket of the SO-SIPD, respectively.
- Connect the SO-SIPD unit via a LEMO/LEMO low power supply cable to the LSE-5000 or to the separate power supply and via two of BNC/BNC cable to the ALV-5000/E Correlator board.

6.2 Adjustment of the Pinhole/Lens/Pinhole Optical Detection System

6.2.1 Position of pinholes and detection lens

The holder for the detection lens and the P_1- and P_2-pinhole holder are positioned according to the 2F/2F-configuration:

a) distance from the axis of rotation of the goniometer to the holder of the detection lens equals 2F = 156 mm with respect to the mounting position of the lens (center).

b) the P_1-pinhole holder is positioned as close as possible to the end of the detection lens holder, but without contact between each other.

c) distance from the axis of rotation of the goniometer to the P_2-pinhole holder equals 4F = 312 mm with respect to the mounting position of the pinhole.

6.2.2. Adjustment of the ODS 125 (fixed pinholes)

- Mount the P_2-holder and fix its position by inserting the two off conical rods. Tighten the 4 off M6 screws.
- Turn the rotary arm to 0° position. Adjust the pinhole in y/z position to obtain a symmetric diffraction pattern, which is coaxial to the position of the naked beam. Turn the rotary arm to 180° and check the pinhole position. If deviations occur the beam position is not adjusted to the required precision and the beam adjustment must be repeated.
- Mount the detection lens holder containing the detection lens with the biconvex side directed to the cell housing. Insert the CDN and shift the lens holder horizontally to obtain a symmetrical diffraction pattern of the needle through the pinhole on the screen. Please note, that the diffraction pattern varies very strong with the depth of the needle tip in the beam profile. Move the needle up and down and watch the diffraction pattern. Insert the LHDN and repeat the vertical alignment of the lens. Remove the LHDN. In case no LHDN is available adjustment is performed to obtain a symmetrical diffraction pattern of the pinhole.
- Block the laser beam behind the lens and tilt the lens holder using the two screws on the front side of the holder (1.5 mm hex key) until the interference fringes watched on the iris aperture in front of the laser are coaxial to the laser beam. Check again the Y/Z position of the lens using the CDN and LHDN.
- Mark the new center position of the pinhole diffraction pattern on the screen. Due to the lens centricity error this position may deviate from the position of the naked beam.
- If the LHDN is available insert pinhole P_1 and adjust the Y/Z position to obtain symmetrical diffraction patterns from the CDN and LHDN on the screen. If the LHDN is not available

remove the P_2 holder and adjust the pinhole to obtain a symmetric pinhole diffraction pattern at the newly marked center position of the P_2 diffraction pattern. Mount the P_2 holder and check the diffraction pattern of the CDN again.

- Close the ODS housing using the appropriate cover plates and insert the apertures in the front and end plate of the housing.

6.2.3. Adjustment of the ODS 125 II (variable pinholes)

The pinhole holder consists of a sliding stage which can precisely be positioned at three different stops. At two stops the pinholes are positioned into the beam and the third stop allows unhindered beam transmission. This allows the pinholes to be aligned to the beam independent from each other. The third stop can only be accessed while the knurled knob on top of the holder is pulled up. The pinholes are mounted inside a cylindrical adjustment unit which is screwed in to the sliding part of the holder. The pinholes can be shifted in Y/Z direction using the two screws of the adjustment unit. **Attention!** The adjustment screws are directed 45° off the Y/Z axis, i.e., you have to turn both adjustment screws simultaneously the same amount in the same direction (clock- or counter-clockwise) to adjust the pinholes in Z-direction (vertical) and to turn both screws simultaneously the same amount in opposite direction to adjust the pinholes in Y-direction (horizontal).

- Turn the rotary arm to 0° and adjusted both pinholes of the P_2-holder to obtain a symmetric diffraction pattern on the screen. Turn the rotary arm to 180° and check the position of both pinholes. If deviations occur the beam position is not adjusted to the required precision and the beam adjustment must be repeated.
- The lens is inserted in the lens holder with the biconvex side directed to the cell housing and fixed by mounting the Delrin ring to the lens holder.
- Insert the CDN and shift the lens holder horizontally to obtain a symmetrical diffraction pattern of the needle through the pinhole on the screen. Please note, that the diffraction pattern varies very strong with the depth of the needle tip in the beam profile. Move the needle up and down and watch the diffraction pattern. Insert the LHDN and repeat the vertical alignment of the lens. Remove the LHDN. In case no LHDN is available adjustment is performed to obtain a symmetrical diffraction pattern of the pinhole.
- Block the laser beam behind the lens and tilt the lens holder using the two screws on the front side of the holder (1.5 mm hex key) until the interference fringes watched on the iris aperture in front of the laser are coaxial to the laser beam. Check again and correct if necessary the Y/Z position of the lens using the CDN and LHDN.
- Mark the new center position of the pinhole diffraction pattern on the screen. Due to the lens centricity error this position may deviate from the position of the naked beam.
- Move the P_2-sliding stage in that position where the beam passes unhindered. Adjust the 2 off P_1-pinholes to obtain symmetric diffraction pattern at the newly marked center position of the P_2 diffraction pattern.
- Check the position of both pairs of P_1- and P_2-pinholes using the CDN and LHDN.

7.0 Mounting and Adjustment of the ALV/STATIC and DYNAMIC Enhancer Unit and the Optical Optimization Unit

The ALV / STATIC and DYNAMIC Enhancer was developed by ALV Company to optimize GRIN lens fiber optical detection of scattered light for SLS and DLS measurements. Use of the ALV / STATIC and DYNAMIC Enhancer for SLS and DLS measurements results in optimum performance of GRIN lens fiber optical detection with suppression of additional slow term fluctuations of the transmitted light intensity due to physical fiber properties. Additionally an achromatic lens is integrated part of the Fiber Adjustment Unit, provided the Goniometer System is already equipped with the Fiber Adjustment Unit including the Optical Optimization Unit. This unit allows an optimum match of the size of the detected and illuminated volume in the sample under Gaussian laser beam versus Gaussian detection considerations.

7.1 Adjustment of the ALV / STATIC and DYNAMIC Enhancer

The ALV / STATIC and DYNAMIC Enhancer replaces the standard GRIN Lens/Fiber for detection of light and installation is performed according to the procedure described in chapter **6.1 Adjustment of the Fiber Optical Detection System** *of the* **Adjustment Manual V.2.2. 03/98** *of the ALV / Compact Goniometer System.*

*Make certain to attenuate the laser beam intensity below the 'safe level' of 2mW at 633 nm, respectively 1 mW at 488 nm for direct coupling of laser light into an optical fiber (see "Handling and Characteristics of Fibers with Integrated Collimation-Detection-Optics"). Make certain that the fiber output is **not** connected to the ALV / SO-SIPD (see "Safe Operation of ALV/SO-SIPD/DUAL Photomultiplier Detection Unit").*

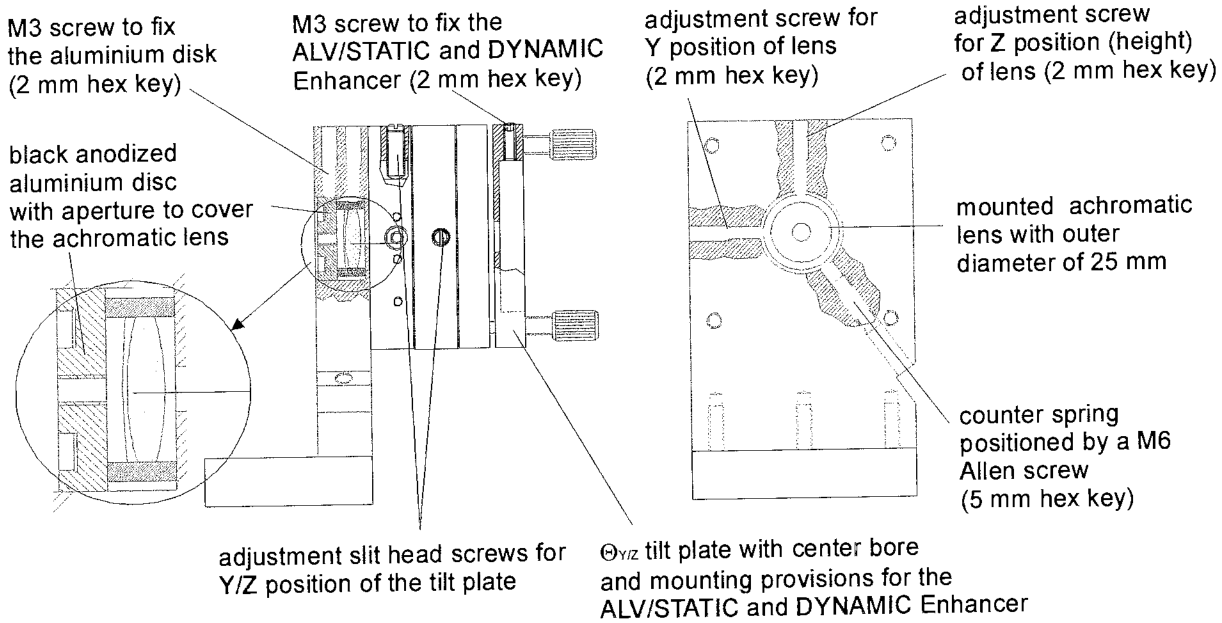


Figure 17:
Fiber Adjustment Unit with integrated Optical Optimization Unit

- Turn the rotary arm to the 0° position.
- The ALV / STATIC and DYNAMIC Enhancer has not be mounted to the Fiber Adjustment holder to allow the laser beam to pass freely through the center bores of holder. Adjust the Y/Z position of the tilt plate of the Fiber Adjustment Holder such that the laser beam passes symmetrically through the center bore of the tilt plate.
- Mount the ALV / STATIC and DYNAMIC Enhancer and fix the unit using the appropriate screws.
- Adjust the $\Theta_{Y/Z}$ tilt angles until light is transmitted through the fiber. Once transmission of light is observed at the ALV / STATIC and DYNAMIC Enhancer fiber output the intensity is maximized by final adjustment of the Y/Z position and the $\Theta_{Y/Z}$ tilt angles.

7.2 Mounting and Adjustment of the Optical Optimization Unit

The achromatic lens of the Optical Optimization Unit has to be mounted after a test measurement of the Sine(Theta) behaviour of a STANDARD measurement has been completed satisfactory according to chapter **9.0 Test of the adjustment: Sine (Θ) measurement of a Toluene sample** of the **Adjustment Manual V.2.2. 03/98** of the ALV / Compact Goniometer System. Provided the ALV / STATIC and DYNAMIC Enhancer detection unit is adjusted to its optimum position the following steps have to be performed to install the Optical Optimization Unit:

- Attenuate the laser beam intensity below the 'safe level' of 2 mW at 633 nm, respectively 1 mW at 488 nm for direct coupling of laser light into an optical fiber (see "Handling and Characteristics of Fibers with Integrated Collimation-Detection-Optics"). Disconnect the ALV / STATIC and DYNAMIC Enhancer fiber output from the ALV / SO-SIPD (see "Safe Operation of ALV/SO-SIPD/DUAL Photomultiplier Detection Unit").
- Turn the rotary arm to the 0° position.
- Insert the achromatic lens at the position indicated in figure 1. Attention! The lens has to be positioned with the biconvex side towards the ALV / STATIC and DYNAMIC Enhancer unit. The position of lens curvatures is engraved on the lens mounting.
- Insert the black anodized aluminium disc in front of the lens and fix it with the appropriate screw.
- Adjust the Y/Z position of the lens using the appropriate screws to obtain maximum light transmission through the ALV / STATIC and DYNAMIC Enhancer.
- Turn the rotary arm to the 90° position. Connect the ALV / STATIC and DYNAMIC Enhancer fiber output to the ALV / SO-SIPD input.
- Finally a slight adjustment of the height position of the lens may be required to obtain maximum intensity of scattered light at 90°.

8.0 Mounting of the Laser Focus Lens unit and PMT (pinhole set-up only)

- The Laser Focus Lens Unit with the laser focus lens (FL = 200 mm) inserted is mounted on the optical table at the marked position. If the laser focus lens position is not marked, mount the unit such that the distance between the lens and the center of rotation of the goniometer is 220 mm. The unit is roughly oriented to obtain the backreflected light spot centered to the iris aperture in front of the laser and fixed using the four M6 Allen screws.
- Turn the rotary arm to about 60° position. Insert the CDN and adjust the Y-position of the focus lens to obtain a symmetric diffraction pattern on the projection screen. Insert the LHDN and adjust the Z-position of the focus lens to obtain a symmetric diffraction pattern on the projection screen. If the LHDN is not available turn the rotary arm to 0° position and adjust the height of the focus lens to obtain maximum transmission through the optical detection system.
- Turn the rotary arm to approximately 60°. **Activate the Hall-limit switches.**
- *ODS-125:* Mount the photomultiplier adjustment plate together with the photomultiplier and its housing on the rear side of the P2 holder using the four metric M3 screws.
- *ODS-125 II:* Mount the Click-stop shutter housing together with the photomultiplier and the adjustment plate on the ODS ground plate.

9.0 Mounting of the IMV

- Close the laser shutter.
- Loosen the 3 off conical rods with a metric M5 internal thread on top of the cell housing by clockwise turning of a metric M5 screw inserted together with a washer in the internal thread. Remove the guiding rods. Loosen and remove the 4 metric M6 screws. Then remove the upper cylindrical cover disk with the upper heat exchanger and place the IMV(carefully cleaned) in the holder of the lower heat exchanger, such that:
  - the "ALV" logo of the IMV is directed toward the detection slit of the cell housing,
  - the outer cylinder of the IMV is hold in position against the 2 off polished rods in the holder, and the front window of the IMV is positioned in 180°, towards the laser.
- Open the laser and block the laser beam inside the vat. Slightly turn the vat until the back-reflected light spot from the front window is perfectly centered to the laser beam. Watch the back-reflected light spot for a rough positioning on the iris aperture of the Monitor Diode/Attenuator Unit and for final positioning on the aperture in front of the laser .
- Fasten the IMV by loosening the metric M4 socket set screw of the holding clamp while watching the back-reflected light spot on the aperture in front of the laser. If its position is changed while fastening the vat repeat the whole procedure of positioning of the vat.
- Prior to mount the upper cylindrical cover disk of the cell housing with the upper heat exchanger screw the 4 off guiding rods with metric M6 thread into the appropriate bores on the upper rim of the cylindrical case of the cell housing. Hold the upper cylindrical cover disk horizontally on top of the 4 off guiding rods in that position which corresponds to its final position. At this position the guiding rods fit into the appropriate bores of the upper cylindrical cover disk. Slowly lower the upper cylindrical cover disk - without tilting it - until it rests with the O-ring made of teflon on the upper rim of the vat. Insert the 3 off conical rods. Make certain that the metric M5 screws are turned counter-clockwise for such an amount that allows the conical rods to fit properly into the bores.

Attention !! If a conical rod does not fit into the bore, **do not try to position it by applying pressure on top of it, because this could lead to a change in the position of the vat.** Instead lift up the cover disk a few millimeters and turn it slightly to the left and right until a position is found where all conical rods directly fit in the appropriate bores. Slowly lower the upper cylindrical cover disk at this position.
After giving a slight blow on the top of the 3 off conical rods they are fixed and the upper cylindrical cover disk cannot be removed by hand. Replace the 4 off guiding rods by the metric M6 screws and tighten the screws crosswise; in case of the standard cell housing until the upper cylindrical cover disk rests directly on the upper rim of the cell housing cylinder and in case of the high temperature cell housing until a torque of 35 Ncm is applied to the screws.
Block the laser beam inside the vat by inserting the CDN and watch the back-reflected light spot at the aperture in front of the laser beam. If it is not centered to the laser beam, the whole procedure of orientation of IMV has to be repeated.

- Remove the CDN and mount the beam stop housing to the cylindrical case of the cell housing at 0° position.

10.0 Test of the adjustment: Sine (Θ) measurement of a Toluene sample

10.1 General remarks about inserting cuvettes in to the cuvette holder

The following precaution !! is required when inserting cylindrical cuvettes for the first time in to a cuvette holder made of black PTFE as part of the ALV / Cell housing.

The hereafter listed steps are required, because the inner cylindrical bore of the cuvette holders made of black PTFE are machined with great precision to exactly the mean diameter with very stringent tolerances. However, the cuvette holders can accept even those cuvettes with different diameters that are within the specified diameter range, e.g. the cuvette holder for cylindrical cuvettes is machined with nominal mean diameters to accept cylindrical cuvettes within the range of:

20.0mm cuvette holder: **19.70 $^{0/-0.05}$ mm to 20.03 $^{0/-0.05}$ mm**

10.0mm cuvette holder: **9.90 $^{0/-0.05}$ mm to 10.10 $^{0/-0.05}$ mm**

Please perform the following steps **prior** to inserting the cylindrical cuvette holder in to the conical bore of the upper heat exchanger of the ALV / Cell housing:

1. use a cylindrical cuvette of appropriate diameter corresponding to the diameter bore in the relevant cuvette holder.

2. use a cylindrical cuvette holder with an appropriate inner diameter bore corresponding to the diameter of the cuvette, as indicated under para 1).
DO NOT INSERT THIS CUVETTE HOLDER yet in to the conical bore of the upper heat exchanger of the ALV / Cell housing.

3. have the cuvette holder positioned on a plane surface and then try most carefully to insert the cuvette in to the inner bore of the cuvette holder. The inner diameter of the cylindrical bore is machined to the nominal mean diameter and consequently cylindrical cuvettes with outer diameters at the lower end of the diameter range may quite easily be inserted, but those with outer diameters at the upper limit of the accepted diameter range may NOT be

easily inserted. Those cuvettes with the outer diameter at the upper limit will be used to open up the cuvette holder for the first time to the upper diameter limit - this will NOT work, if the cuvette holder is already positioned in the conical bore of the upper heat exchanger of the ALV / Cell housing !!

4. when inserting a cylindrical cuvette in to the cuvette holder with a known outer diameter at the upper limit, or a noticeable resistance when pushed in to the bore, the cuvette must be inserted slowly straight forward pushed (perpendicular to the cuvette holder, NOT TILTED) - by all means avoid any turning of the cuvette when pushed in to the cuvette holder. Please take the cuvette out again and repeat the inserting by the same procedure several times until the cuvette holder begins to accept this cuvette easily when inserted. NEVER TURN THE CUVETTE WITH A „"SCREW-IN"-MOVEMENT.
5. after step 4) insert the cuvette holder in to the conical bore of the upper heat exchanger of the ALV / Cell housing and use the appropriate screw-tool (made of stainless steel) that goes on top of the cuvette holder, but ONLY GENTLY screw this part in so it just touches the cuvette holder at the top.
6. repeat all steps as described above. If the cuvette cannot be easily inserted and provided the cuvette is partially already positioned in the cuvette holder (in consequence this means one has tried by this action to widen the outer conical diameter of the cuvette holder, which would react with an upwards movement, but is blocked by the upper screw-tool) and should at the same time give room for an expansion of the inner diameter for the cuvette, this can ONLY be achieved, if the upper screw-tool has been SLIGHTLY UNSCREWED and the cuvette then be pulled out of the cuvette holder by an upwards movement.
7. try again as described under para 6) and proceed until the cuvette can easily be inserted - to achieve this the upper screw-tool may have therefor be step-by-step several times SLIGHTLY UNSCREWED.

AS A RULE FROM THE BEGINNING ON:

*NEVER UNSCREW THE UPPER SCREW-TOOL **TOO** MUCH.*

10.2 Preparations

- Fill the Index Matching Vat with approx. 50 ml purified and dust free toluene (p.a. quality) or another index matching fluid. Choose a filling procedure which avoids the formation of air bubbles. Watching the vat illuminated by the laser beam through the slit of the cell housing air bubbles and the surface level of the index matching fluid must not be seen. This is mandatory to perform static light scattering measurements.
- Insert a quartz cuvette (∅ = 10 mm) filled with toluene in the cuvette holder.

*The toluene in the cuvette and in the IMV **must** be dust free!*

- *Fiber ODS only:* Turn the rotary arm to 90° position and adjust the height (Z-position) of the Fiber Adjustment holder until maximum intensity is obtained. The correction of the height depends on the actual diameter of the laser beam, but should usually not exceed a quarter turn of the corresponding height adjustment screw (see fig. 15).

- *pinhole ODS only:* Turn the rotary arm to 90° position and adjust first the vertical and then the horizontal position of the photomultiplier tube to obtain maximum count rate. The adjustment is performed using the appropriate screw and counter-screw pairs of the adjustment plate of the photomultiplier unit.

10.3 Performing the measurement

Start the program ALV5000/E/WIN and perform a STANDARD measurement following the instructions given in the "A Guide to perform Simultaneous Dynamic & Static Light Scattering Measurements using the ALV-5000/E/WIN Software". Use the following recommended parameters:

- Number of runs: 3,
- Duration of single measurement (*Duration*),
  The duration is dependent on the scattering intensity of the illuminated sample and a function of laser power and/or its attenuation. A guideline for sufficient statistically secured result is a total counting of 100,000 counts, and the duration is simply a function of the count rate.
- Starting angle (≤ 15°), ending angle (= 150°) and step width (= 1°).

- The angular dependence of intensity corrected for the size of the scattering volume (i.e., intensity multiplied by Sine(Θ)) is displayed in an appropriate graphic window.
- The measured Sine(Θ) dependency for toluene gives a first order information under nearly ideal optical conditions, as all the involved refractive indices are nearly of the same value. A most crucial test is of course to insert a cylindrical cuvette of 10 mm diameter, filled with a liquid of an refractive index as low as equivalent to water. Now the optical conditions are dominated by astigmatism and spherical aberration, mainly caused by the refractive index difference at the path steps where the laser beam enters and leaves the liquid; here we have a very similar situation as with a cylindrical lens and a cylindrical mirror. Furthermore the angular dependency of Sine(Θ) is under this condition strongly influenced as dependent on the laser beam diameter and the remaining divergence on its path length within the liquid in the cuvette. This will then result in excess of detected intensities at lower and higher angles.
- **In case of proper adjustment of all mechanical and optical units of the compact goniometer system, the values for the Sine(Θ) measurement should not deviate from the mean value of all measured angles more than ± 2 % for the angular range of 25° - 140° (see Figure 17). Provided the beam diameter in the cuvette equals approximately 120 - 150 µm with a Rayleigh length of at least 40 mm the Sine(Θ) measurement can be aligned to an accuracy of ± 1 % for the angular range of 20° - 150°. This is mandatory to perform static light measurements with acceptable accuracy.**

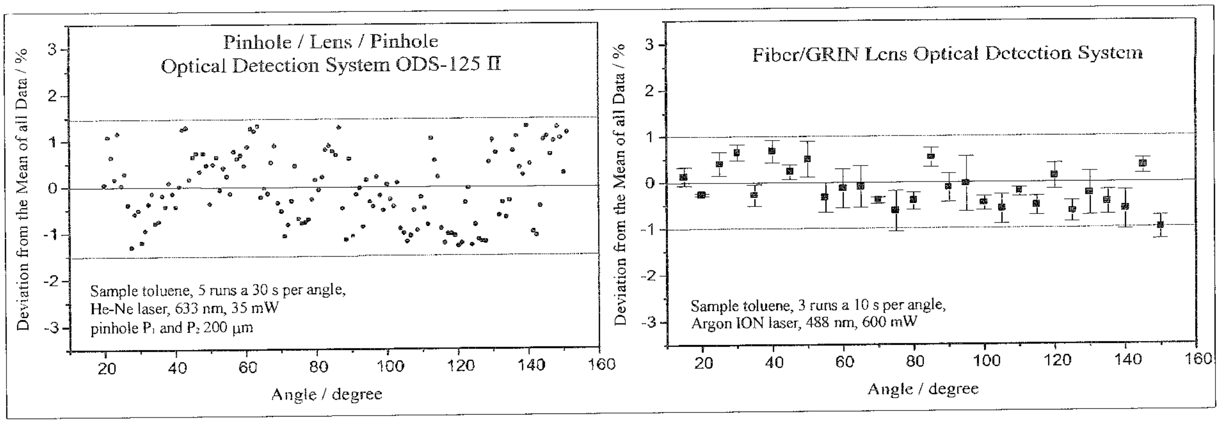


Figure 18:
Test of alignment by measurement of deviation from Sine(Theta)

- Measurement of Sine(Θ) dependency will show the additional influence of the refractive index of the liquid in the cylindrical cuvette (water; volume correction based on Herman & Levinson).
- If the standard deviation of a measurement performed with 3 runs is larger than 0,7% (possibly due to dust particles in the scattering volume during a single measurement) the measurement for this angle has to be neglected and/or repeated, respectively.
- *Fiber Optical Detection System:* A fine tuning of the height of the focus lens and of the Z position and the Θ_Z tilt angle of the Fiber Adjustment Holder should improve the Sine(Θ) measurement to the desired accuracy.
- *Pinhole/Lens/Pinhole Optical Detection System:* A fine tuning of the height of the focus lens should improve the Sine(Θ) measurement to the desired accuracy.

11.0 Re-alignment of the ALV-5000 Compact Goniometer System

The need for re-alignment of the Compact Goniometer System occurs only if measurements of toluene as standard show **systematic** deviations from the Sine (Theta) dependency larger than 2-5 % (depending on the required accuracy of static light scattering measurements).

Prior to start a re-alignment one should try to analyse the cause of misalignment. The following rules apply:

1. Check the laser beam position using the monitor diode. Deviations less then 10 - 20% per quadrant from the mean value are still within the limit (this corresponds to a length of the green vector line within the white circle in the diode display of the program ALV5000).
2. Remove the beam stop and check the laser beam position in the far field versus the position of the naked beam marked after the alignment performed at last.

In the case both checks result in a still perfect alignment of the laser beam there are two possible reasons for the deviations from the Sine (Theta) law:

- The cell housing is no longer adjusted to the axis and plane of rotation of the goniometer. Solution: An adjustment of the cell housing must be performed. This can be done according to chapter 3 in a limited angular range of 15° until 150° without removing any unit from the system.
- The detection unit is misaligned for some reasons. In the case of Fiber/GRIN lens detection a re-alignment can simply be performed using the primary laser beam after unplugging the fiber from the photomultiplier unit.
  Attention! Prior to unplugging the fiber the voltage supply of the photomultiplier unit must be switched off or unplugged and after unplugging the fiber the input of the photomultiplier unit must be closed using the appropriate cap.
  The rotary arm is moved to the 0° position and the fiber is adjusted to maximum transmission. A re-alignment of the pinhole set-up requires to remove the photomultiplier unit, the Laser Focus Lens and the IMV and is performed according to par. 6.2.

11.1 Re-alignment of the Laser Beam

In case the laser beam deviates significantly from its position found in the previous alignment a re-alignment of the laser beam is necessary.

- First remove the index matching fluid out off the IMV. Replace the 4 off metric M6 screws from the upper cylindrical cover disk by the 4 off guiding rods with metric M6 thread. Loosen the 3 off conical rods by screwing the metric M5 screws clockwise and remove the rods. Slowly lift the upper cylindrical cover disk and remove it. Loosen the IMV by fastening the screw of the holding clamp positioned below the house coupling for external ventilation (see fig. 9), remove the IMV and store it at a safe place. Mount the upper cylindrical cover disk and fit it with the 3 off conical guiding rods. Remove the 4 off guiding rods with metric M6 thread.
- *Fiber ODS:* Remove the Laser Beam Optimization Unit and unplug the optical Fiber from the Fiber Adjustment Holder as well as from the photomultiplier unit. **Attention! Prior to unplugging the fiber the voltage supply of the photomultiplier unit must be switched off or unplugged and after unplugging the fiber the input of the photomultiplier unit must be closed using the appropriate cap.** If a Fiber/GRIN Lens detection system is used mark the position of the adapter plate containing the stainless steel ferule end of the fiber and remove the adapter plate. Thus, inserting the adapter plate with the fiber end in the

marked position results usually in transmission of light and the adjustment of the fiber becomes much easier.

Pinhole/Lens/Pinhole ODS: Remove the Laser Focus Lens Unit. If the Double Needle Adjustment Unit is fix mounted on the optical table (e.g., as part of the Projection Optics System) the logged settings of the micrometers can be used to realign the beam. Otherwise the Photomultiplier Unit and the Detection Lens (ODS 125 II) and additionally the pinholes P_1 and P_2 (ODS 125) have to be removed and after re-alignment of the laser beam to be adjusted according to par. 6.2.

- Check and, if necessary, correct the alignment of the cell housing (see chapter 3.0).
- Provided the laser beam position is not completely off, which is in general only the case if the laser or the laser tube has been changed, the alignment of the laser beam is performed according to par. 5.6 using the logged values of the setting of the micrometers of the DNAU. Otherwise the alignment has to be started according to par. 5.2 and 5.5. Additionally the display of the monitor diodes (window Diode Adjust of the ALV-5000/E/WIN software) and the marked position of the naked laser beam on the screen in the far field can be used to restore the old laser beam position.
- For the Pinhole/Lens/Pinhole ODS it is recommended although not necessarily required to check the adjustment of the pinholes. This check can only be performed with the photomultiplier unit removed according to par. 6.2.

Proceed with par. 5.8 and 6.1 (Fiber ODS) or chap. 7.0 (Pinhole/Lens/Pinhole ODS) and complete the alignment according to chap. 8.0 and 9.0.

List of Figures

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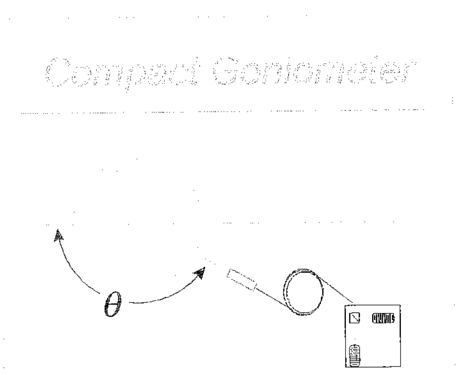
ALV / DLS / SLS - 5000 System

Compact Goniometer System

A Guide to perform
Simultaneous Dynamic & Static Light Scattering Measurements
using the ALV-5000/E/WIN Software



$$\mathbf{I}_S \propto \left| \int e^{i\mathbf{r}(\mathbf{k}_i - \mathbf{k}_f)} \, d\mathbf{r} \right|^2$$

Content

1. General Notes about Simultaneous Static and Dynamic Light Scattering Measurements

1.1 Introduction

The ALV / DLS / SLS - 5000 Compact Goniometer System is designed to perform Dynamic (DLS) and Static Light Scattering (SLS) data simultaneously.

Two different data file systems are used in the ALV-5000/E/WIN software. The correlation function and count rate trace data together with all relevant sample parameters can be stored as TAB separated ASCII values, and, the angular and concentration dependent normalized mean values of the scattering intensity as well as diffusion coefficients derived by a Cumulant analysis (up to third order) of the correlation functions are stored in a file with a special binary record format (DILS file format).

An analysis of SLS/DLS data stored in DILS file format easily be performed using the ALV/Static & Dynamic Fit and Plot program by creating ZIMM, BERRY and GUINIER plots, plots of diffusion coefficients versus q^2 and form factor plots.

1.2 Measurement of Laser Beam Intensity and Sample Temperature

The ALV / DLS / SLS - 5000 System Compact Goniometer System provides with the possibility to measure the laser beam intensity using a monitor diode and the sample temperature using a Pt-100 temperature probe inserted in the index matching fluid.

Both information is vital for high quality Static Light Scattering measurements and once the appropriate settings are performed in the "Option Setup" after each restart of the ALV-5000/E/WIN software the measurements are initialized automatically.

Performing Static Light Scattering without monitoring of the laser beam intensity results in a limitation of the precision of these measurements due to the laser intensity fluctuation, which can be as high as +/- 3% for long time measurements.

Temperature measurement is necessary for correcting the Rayleigh Ratios for the STANDARD, but usually the absolute influence is significantly smaller than that of laser intensity fluctuation. Please remember, that the ALV/Pt-100 probes used for temperature measurement have an absolute precision of +/- 0.2 K within the range of 263.17 ... 373.17 K and higher absolute precision requires special calibration by the user. The relative precision of the temperature measurement is about +/- 0.01 K.

1.3 Measurement of the standard sample (STANDARD Mode)

The measurement of a STANDARD sample with known Rayleigh Ratio is the most critical point in Static Light Scattering, which defines the limit of accuracy for estimation of the absolute value of the scattering ratio of an unknown sample.

All goniometer systems are in general not adjusted to give absolute values of measured scattering intensities, but relative only. To perform measurements of absolute values of static light scattering intensities the optical efficiency of a measuring system is calibrated by measurement of a STANDARD sample with a-priori known Rayleigh Ratio.

A common STANDARD used is toluene, due to its very well tabulated values of the Rayleigh Ratios and existing interpolation formula to correct the Rayleigh Ratios for different wavelengths of the laser and the actual sample temperature.

But, about 30% of the light scattered by toluene is due to depolarised light scattering. This fact has to be considered because the ALV Compact Goniometer System offers for SLS/DLS measurements the following two set-up's of detection systems:

1. **Standard set-up (VU set-up):** vertical polarised laser light and non-polarised detection (either fiber or pinhole detection), and
2. **Optional set-up (VV set-up):** vertical polarised laser light and vertical polarised detection (e.g., using a Glan-Thompson prism).

The values of the Rayleigh Ratio for toluene differ in both cases by about 30% and depending on the actual set-up the correct value has to be selected for calculation (see chapter 2.2.3).

If the count rate is very low, an usual situation for the STANDARD measurement (since no scattering particles are within the sample, but scattering is just due density fluctuations within the fluid), the precision of the measurement is shot noise limited, that is the finite number of photons detected with the total measurement time is the major noise source for this measurement.

A good approximation to this noise contribution is the standard Gaussian approximation, i.e.,

$$3\sigma = \pm 3 \frac{\sqrt{N}}{N}$$

with N being the total number of photons detected. That means, a 1 kHz count rate requires about 30 s of total measurement time for a 1% precision and the three sigma approximation (99,5 % of all measurements are within this error region).

The STANDARD measurement should be performed with highest possible precision, and, in order to check the current mechanical and optical alignment conditions with very small angular steps (use 1° steps if possible). The precision machined mechanics of ALV-goniometer systems ensures that these measured STANDARD values remain within the limits measured for very long times, usually more than 6 months, provided the ambient temperature of the room is kept at the set temperature within ± 1°C. However, to perform SLS measurements with highest precision it is recommended to perform a STANDARD measurement prior to the measurement of a sample series at that angles, where the sample series will be measured.

1.4 Measurement of the solvent sample (SOLVENT Mode)

SLS measurements are often performed on dilute solutions, where the excess scattering ratio is used to estimate molecular weights, radius of gyration and other relevant physical parameters. Therefore, the knowledge of the scattering ratio of the solvent is required to obtain the excess scattering ratio.
If the STANDARD and SOLVENT sample are identical (both toluene, for example), the file obtained from the STANDARD measurement can also be used as the SOLVENT file. Otherwise a measurement of the scattering intensities of the solvent is required, and the same remarks regarding measurement conditions as for the STANDARD measurement apply.

For further data evaluation using the SOLVENT data, only the scattering ratio and refractive index of the solvent are of interest, all other parameters (Rayleigh Ratio etc.) stored in the file are ignored in further calculations.

1.5 Measurement of the solution samples (SOLUTION Mode)

In SOLUTION mode angular dependent values of the scattering intensity of a solution are measured and absolute scattering ratios or excess scattering ratios are calculated based on the STANDARD and SOLVENT measurement. Furthermore, apparent diffusion coefficients can be obtained from Cumulant analysis up to third order and stored together with the scattering ratios of the solution.
Results of successive measurement of sample series with different concentrations can be appended to one file.

In general, the measurement conditions in SOLUTION mode are similar to that of the STANDARD or SOLVENT mode, but one must be take care of the following:

- Depending on the size of the scattering particles the scattering intensities may vary with the scattering angle and the intensity is increasing with smaller angles, thus a dynamics of more than a factor of 10^4 in the intensity can be detected over the entire angular range (e.g., Mie scattering regime).
  Adjustment of the laser beam intensity at the lowest measurement angle to such a scattering intensity not exceeding the safe intensity level of the actually used photomultiplier ensures proper operation and avoids possible damage of the photomultiplier due to intensity overload. Furthermore, the adjustment has to be made so that a level of laser beam intensity is chosen that still guarantees a linear relation between photon density and count rate.
- AutoScaling should be switched on (see chapter 2.1).
- The measurement duration of a single run depends on the observed largest correlation time t_c and is in most cases defined by diffusion noise (or whatever process responsible for the observed correlation) rather than photon noise. Typically, the fluctuation of the light scattered due to the particle diffusion becomes as large as several 10 ms at low angles and measurement precision may require total measurement times of several 10 s, even if shot noise considerations only may lead to much smaller measurement times. A rough estimation of the diffusion noise can be obtained from the following relation:

$$3\sigma_D \cong \pm 12 \sqrt{\frac{t_c}{duration}}$$

2. Performing Simultaneous SLS and DLS Measurements

2.1 Setting of General Parameters

Prior to start a measurement in STANDARD, SOLVENT or SOLUTION mode using the QuickSet "General DLS and SLS" the following parameters should be checked for correct setting (for more information please refer to the on-line help of the ALV-5000/E/WIN software:

1. Option Setup (Menu Setup):
   communication port for communication with the LSE-unit,
   temperature and monitor diode measurement,
   Pseudo-cross mode selected, if ALV/SO-SIPD used for detection,
   count rate alert (750 kHz for EMI PMT's, 5000 kHz for ALV/SO-SIPD)

2. ALV-5000/E Setup (Menu Setup):
   correlation mode, (AUTO, SINGLE or CROSS)
   Autoscaling (off in STANDARD and SOLVENT mode, on in SOLUTION mode),

3. Sample Description (Menu Sample):
   sample name,
   refractive index and viscosity of solvent or sample res.,
   laser wavelength,

4. AutoSave Option (Menu File)
   to store the individual correlation functions for further data analysis if required (only in SOLUTION mode).

2.2 The QuickSet "General DLS and SLS" (Menu QuickSet, Dynamic / Static Light Scattering

2.2.1 General remarks

The QuickSet "General DLS and SLS" has to be used to perform any angular dependent SLS and/or DLS measurements.
All parameters necessary to perform a measurement are set in 3 steps, the first is to set *Measurement Control parameters*, the second to check the *Static & Dynamic LS Options* and the third to define the *Measurement Type* and to enter the appropriate filename to store data. Once the parameters are set a measurement can be started by clicking the *Start Measurement* button.
If the check box "Generate measurement protocol" is set the window *Static/Dynamic Light Scattering Data* containing a table of the measured quantities and a plot of the calculated scattering ratio vs. angle is automatically opened to display the results of the measurement. Clicking the *Stop Measurement* button at the bottom of this window interrupts the actual measurement after finishing the actual run.

After completion of a particular measurement or in case of interrupting a measurement a new measurement can only be started using the QuickSet "General DLS and SLS" again. After starting a new measurement the user is asked either to clear all previous results in the protocol table of the window *Static/Dynamic Light Scattering Data* or to append the new data. Up to 500 measurements can be displayed in the table, this corresponds, e.g., to a measurement of 10 different samples or concentrations at 50 scattering angles. In STANDARD and SOLVENT mode data are **not** appended to the files, instead old data in the files are overwritten. Therefore, to proceed with a STANDARD or SOLVENT measurement after interrupting the measurement a new filename must be entered and the data can only be merged into one file after completion of the measurement using the MS-WINDOWS® Editor.

2.2.2 Step1: Measurement Control

Up to 6 ranges with particular settings for minimum and maximum angle, angular step increase, number of runs per angle and duration of a single run can be defined. At least 1 range must be entered into the table using the button *Accept this Set* to enable starting a measurement via the button *Start Measurement*.

For further data treatment it is recommended to use the same settings of minimum, maximum angle and angular step increase for corresponding measurement in STANDARD, SOLVENT and SOLUTION mode, although it is not strictly required.
A Dust Filter can be enabled to repeat a measurement at a particular angle if the standard deviation of the measured mean count rates of all runs at this particular angle exceeds the value set.

2.2.3 Step2: Static & Dynamic LS Options

The following options can be enabled:

- *Measure and correct for detector dark counts*:
  Measurement and correction of detector dark counts should be performed if the count rate of the sample at 90° scattering angle (usually in STANDARD or SOLVENT mode) is not larger than a factor of 100 compared to the dark count rate of the photon detection unit. If the option is enabled, a 30 s measurement of the dark count is performed prior to start the angular dependent measurement and the measured mean dark count rate is then subtracted from the measured mean count rate of each single run.

  The default setting of this option is disabled.

- *Use Toluene Rayleigh Ratios as standard:*
  If this option is enabled the program assumes toluene be used as standard sample and the Rayleigh Ratio RR_{VU} of toluene is extrapolated for the laser wavelength set and temperature measured assuming a non-polarized detection (VU set-up). Furthermore, the refractive index set in the corresponding input line is used as refractive index of the standard, independent on the refractive index set in the *Menu Sample*.
  Provided a polarized detection (VV set-up) is used the sub-option *Compute Rayleigh Ratio for VV-Scattering* must be enabled to use the correct Rayleigh Ratio RR_{VV} instead of RR_{VU}.
  If the option is disabled the Rayleigh Ratio of the standard must be entered in the corresponding input line. In this case the refractive index specified in the *Menu Sample* is used as refractive index of the standard sample.

  The default setting of this option is enabled and use of RR_{VU} for non-polarized detection. This option affects only measurements in STANDARD mode.

- *Compute and store Cumulant Analysis:*
  If this option is enabled a Cumulant analysis up to third order is performed with the correlation function of each single run. The mean apparent diffusion coefficient and their standard deviations are stored and the mean apparent hydrodynamic radius calculated from the Cumulant analysis of first order is displayed in the measurement protocol. The last channel that is included in the Cumulant analysis is selected using a data cut off defined in percentageof the intercept of the correlation function (default value is 10% which is a reasonable value for a narrow unimodal distribution of correlation times).

  The default setting is to enable this option. Calculations are only performed for measurements in SOLUTION mode, i.e., it is not necessary to disable this option in STANDARD and SOLVENT mode explicitely.

- Generate a measurement protocol:
  If this option is enabled the window *Static/Dynamic Light Scattering Data* is opened after the measurement is started using the *Start Measurement* button.

  The default setting is to enable the option.

2.2.4 Step3: Measurement Type

In this step the measurement mode is selected and the appropriate file names are entered. Filenames can either be entered via keyboard after selecting the appropriate input using the mouse cursor or selected from the standard file dialog after clicking on the folder icon positioned right from the input line.
If STANDARD or SOLVENT mode is selected only the filename for the standard or solvent file has to be entered. Default filenames are *Standard.tol* and *Solvent.tol*, but any other filenames and extensions are accepted by the program.
In SOLUTION mode a standard file name must be entered that contains valid data of a STANDARD measurement. If either no solvent file name is entered or the option *Measure without solvent information/file* is enabled the calculations are performed without subtracting the scattering ratio of the solvent from the scattering ratio of the solution. A valid file name must be entered prior starting the measurement, no default file name is given but in order to be able to import the file into the ALV/Static & Dynamic Fit and Plot program the file

extension *.sta should always be used. Furthermore, the values for the concentration and the refractive index increment have to be entered. In SOLUTION mode only data may be appended to an existing file.

2.2.5 Measurement Protocol and further data analysis

Depending on the measurement mode different entries are displayed in the table of the measurement protocol. For a detailed description please refer to the on-line help of the ALV-5000/E/WIN software. All data displayed in the table can be exported via the clipboard into other worksheet oriented programs for further data evaluation and plotting.

Using the ALV/Static & Dynamic Fit and Plot program all data stored in standard, solvent and solution files can be plotted and further analyzed. For more details please refer to the on-line help of this program.

Appendix

A: Reflection Correction of the Measured Scattering Ratio

Correction of reflection effects on the measurement of absolute scattering ratios using cylindrical cuvettes

Light passing through an transition between two dielectric materials having different dielectric constants, i.e., refractive indices, is partially reflected. For perpendicular incident light and isotropic dielectric materials the reflection coefficient $r = I_{refl}/I_0$ is independent on polarisation and can be obtained from the Fresnel equation for perpendicular incidence of light:

$$r = (n_1-n_2)^2 / (n_1+n_2)^2 \quad (1)$$

with n_1 and n_2 being the refractive index of each material. Equation (1) is valid within the required precision even for deviation from perpendicular incidence up to 10° which allows for separation of reflection and refraction effects. Refraction effects are in general considered by the Herman & Levinson correction.

Samples of reflection coefficients for various refractive index differences are given below.

approx. value of sample refractive index	sample fluid	reflection coefficient at transition of fluid to SUPRASIL cuvette (n = 1.47)	reflection coefficient at transition of fluid to borosilicate glass (n = 1.52)
1.5	toluene	0.01 %	< 0.01 %
1.4	THF	0.06 %	0.17 %
1.33	water	0.25 %	0.44 %
1.0	air	3.6 %	4.25 %

Due to the non-linear dependence of the reflection coefficient from the refractive index difference a remarkable amount of reflected light is only observed for refractive index differences larger 5-10 %.

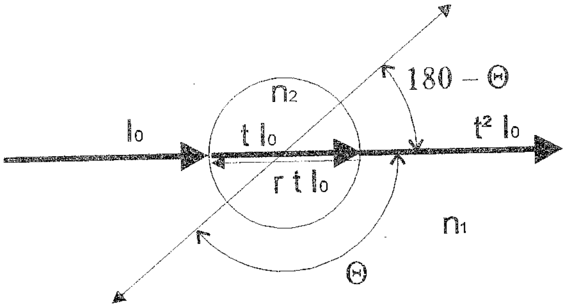
In order to minimise the effects of cuvette curvature (refraction) on the laser beam the standard procedure for SLS measurement is to transform the stronger curvature of the sample cuvette into an appropriately reduced curvature by using an Index Matching Vat (IMV) of an increased wider diameter, filled with a fluid of a refractive index as close as possible to refractive index of the cuvette. Additionally the use of the IMV almost completely reduces the reflection effects at the outer surface of the cuvette.

ALV Goniometer Systems include a precisely machined IMV made from SUPRASIL quartz glass having two plan parallel windows at 0° and 180° position to allow non-refracted passing of the laser beam through the IMV. Additionally the entrance and exit window for the illuminating laser beam are broadband multi-layer antireflection coated at the outer surfaces to minimise reflection of the laser beam below 0.2 % per surface.

Furthermore, the upper part of the heat exchanger holds the sample cuvette in position and blocks mechanically the rear side of the cylindrical wall of the IMV and, therefore, no reflection of scattered light at this glass/air transition occurs. In this case there is only the glass/air transition in the detection path that lowers the transmission of scattered light by a factor of 0.96 due to reflection, but independent of the type of sample and sample cell inserted into the IMV. This transition loss must not be included into the reflection correction, because it is already included in the STANDARD measurement used to calculate absolute scattering ratios.

In the following we restrict ourselves to use of an index matching fluid with a refractive index differing no more than 0.03 from refractive index of the cuvette used, which is a mandatory condition to perform SLS measurements over a wide angular range. In this case only the sample/cuvette interface may produce reflected light. All other interfaces have negligible reflection coefficients. Furthermore, we consider only first order reflections because higher order reflections have intensities below the noise level.

The following drawing shows an idealised ray tracing of a laser beam through a cuvette with refractive index n_1 that contains a liquid with refractive index n_2.



I_0 is the primary laser beam intensity (monitored using the ALV / Monitor Diode) and each time either the laser beam or the scattered light passes the interface between fluid and cuvette reflection occurs reducing the transmitted beam intensity by the factor $t = 1 - r$ (assuming no measurable energy loss due to absorption and scattering at the transition and in the fluid).

The detected intensity of the scattered light at the angle Θ consists of 4 contributions:

I. light scattered from the primary laser beam at a scattering angle Θ,

II. light scattered from the primary laser beam at a scattering angle 180°- Θ and then reflected to the observation angle Θ,

III. light scattered from the reflected part of the primary laser beam at a scattering angle 180°- Θ,

IV. light scattered from the reflected part of the primary laser beam at a scattering angle Θ and then reflected to the observation angle Θ; this will be neglected as being a second order reflection that is in any case by orders of magnitude lower than contribution I.

The contribution II cannot be neglected due to the cylinder lens effect of the cuvette, resulting in collection of scattered light from a similar or even larger size of the scattering volume compared to contribution III.

The contributions to the measured intensity of scattered light can be calculated according to the following formulas:

I. $$I_1 = \left(\frac{R_\Theta}{\sin(\Theta)} \; tI_0 \right) t$$

II. $$I_2 = r \left(\frac{R_{180°-\Theta}}{\sin(180° - \Theta)/k'} \; tI_0 \right) t$$

III. $$I_3 = \left(\frac{R_{180°-\Theta}}{\sin(180° - \Theta)} \; r\,(tI_0) \right) t$$

where R_Θ resp. $R_{180°-\Theta}$ is the normalised scattering ratio of the fluid at the angle Θ resp. 180°- Θ. The factor k' accounts for the change of the scattering volume actually contributing to the detected light due to the reflection at the rare side of the cuvette. In general k' depends on the diameter of the cuvette, the refractive index of the sample and the geometrical parameter of the detected volume and, therefore, on the scattering angle Θ,. Although, this factor is difficult to calculate one can show, that using a cuvette with 20 mm diameter, a sample with $n_2 = 1.33$ and an optical detection system with an cylindrical geometry of the detection volume having a diameter of 0.8 mm, $k' \cong 1$ at 90° and at maximum $k' \cong 1.2$ at 150° assuming homogeneous illumination and even less assuming Gaussian intensity distribution of the laser beam and the detected light. Therefore, $k' = 1$ is assumed for further calculations

Summing and sorting equations I.-III. using the identity $\sin(\Theta) = \sin(180° - \Theta)$ results in the following equation for the normalised measured intensity of scattered light:

$$R_\Theta^{meas} = \frac{I_\Theta^{meas}}{I_0} \sin(\Theta) = R_\Theta \; t^2 + R_{180°-\Theta} \; 2\,t^2\,r \qquad (2)$$

For a Rayleigh scattering sample $R_\Theta = R_{180°-\Theta}$ yields and equation (2) reduces to

$$R_\Theta^{meas} = R_\Theta \; t^2\,(1+2\,r) \cong R_\Theta \qquad (3)$$

where the relation $t^2(1+2\,r) \cong 1$ yields within much better than 1 % even for r = 4 %.

2.3 *Data Processing*

The following calculations are performed during execution of the QuickSet "General DLS and SLS":

1. *in STANDARD, SOLVENT and SOLUTION mode:*

   - calculation of the scattering vector
     (λ is the laser wavelength, n is the refractive index of standard or solvent/solution sample)

$$q = \frac{4 \pi n}{\lambda} \text{Sine}\left(\frac{\Theta}{2}\right)$$

   - normalization of the measured mean count rate for the scattering volume (Sine(Θ)) and the laser beam intensity measured using the monitor diode (I_{mon})

$$\text{Ratio}_{\text{Standard/Solvent}} = \frac{CR_{\text{Standard/Solvent}} \, \text{Sine}(\Theta)}{I_{mon}}$$

$$\text{Ratio}_{\text{Solution}} = \frac{CR_{\text{Solution}} \, \text{Sine}(\Theta)}{I_{mon}}$$

2. *in SOLUTION mode:*

   - calculation of the optical constant K (dn/dc is the refractive index increment of the solution, NA is the Avogadro number)

$$K = \frac{4 \pi^2 \left(\frac{dn}{dc} n_{\text{Solvent}}\right)^2}{N_A \lambda^4}$$

   - calculation of the scattering ratio R taking into account the Herman & Levinson correction factor for the scattering volume due to refraction effects for samples with refractive index different to the refractive index of the standard sample (J.J. Hermans, S. Levinson, J.Opt.Soc.Am., 41, 1033 (1951)), (RR_{Standard} is the Rayleigh Ratio of the standard sample)

$$R = \frac{R_{\text{Solution}} - R_{\text{Solvent}}}{R_{\text{Standard}}} RR_{\text{Standard}} \left(\frac{n_{\text{Solvent}}}{n_{\text{Standard}}}\right)^2$$

   - calculation of Kc/R (c is the concentration of the solution)
   - calculation of the apparent hydrodynamic radius according to the Stokes/Einstein relation (if Cumulant analysis is enabled), (T is the absolute temperature, k is Boltzmann' constant, η is the viscosity of the solvent and Γ the correlation rate obtained from the Cumulant analysis of first order)

$$\text{Rad} = \frac{kT}{6 \pi \eta \Gamma} q^2$$

In general a correction of the measured scattering ratio at the angle Θ for contributions of reflected light can be performed based on the measurement of the scattering ratio at the counter angle 180° - Θ according to the following formula:

$$R_\Theta = R_\Theta^{meas} \left(\frac{1-2\,r\;R_{180°-\Theta}^{meas} / R_\Theta^{meas}}{t^2} \right) \qquad (4)$$

It is evident from equation (4) that only for $R_\Theta << R_{180-\Theta}$ a remarkable contribution of reflected light is observed that requires a correction of the measured scattering ratio. Therefore, a correction of reflection effects is only necessary for samples that show large angular dependencies for the values of the scattering ratio (depending on the value of r a variation of more than 1 order of magnitude should be obtained), and, furthermore, the correction is only required for angles where the lower scattering ratio is detected compared to the detected value at the counter angle. The restriction that a correction of reflection effects is only possible at angles where the scattered intensity at the counter angle can be measured is usually not a limit, because in most cases the correction has to be performed at large angles where a measurement at the counter angle is no problem.

Conclusion:

A reflection correction is not required for systems showing Rayleigh Scattering behaviour, i.e., STANDARD and SOLVENT measurements are not affected by reflection effects.

A correction is only required for systems showing a strong angular dependence of the scattering ratio and an appropriate routine to correct the reflection effect using equation (4) is included in the ALV / Static & Dynamic Fit and Plot program.

B: ALV / STATIC and DYNAMIC Enhancer

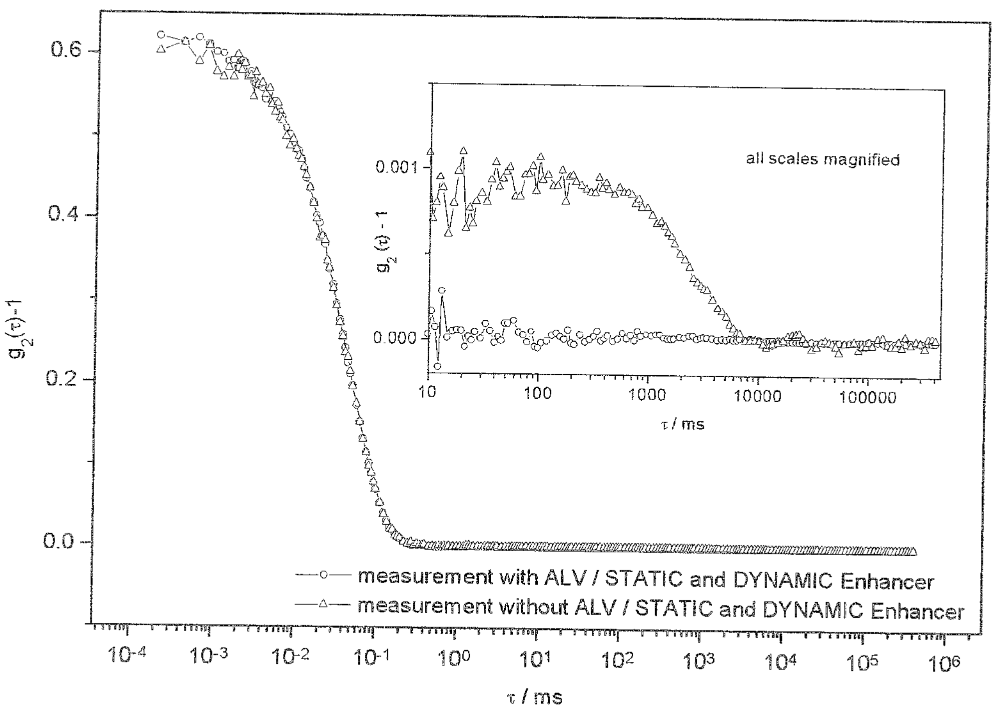
What is the ALV / STATIC and DYNAMIC Enhancer?

The ALV / STATIC and DYNAMIC Enhancer was developed by ALV company to optimize GRIN lens fiber optical detection of scattered light for SLS and DLS measurements. Use of the ALV / STATIC and DYNAMIC Enhancer for SLS and DLS measurements results in optimum performance of GRIN lens fiber optical detection with no additional slow term fluctuations of the transmitted light intensity due to physical fiber properties. The following measurements demonstrate the superior performance of the ALV / STATIC and DYNAMIC Enhancer.

Laser: frequency doubled Nd:YAG laser, Single Longitudinal Mode (TEM_{00}) $\lambda = 532$ nm, $P = 50$ mW,

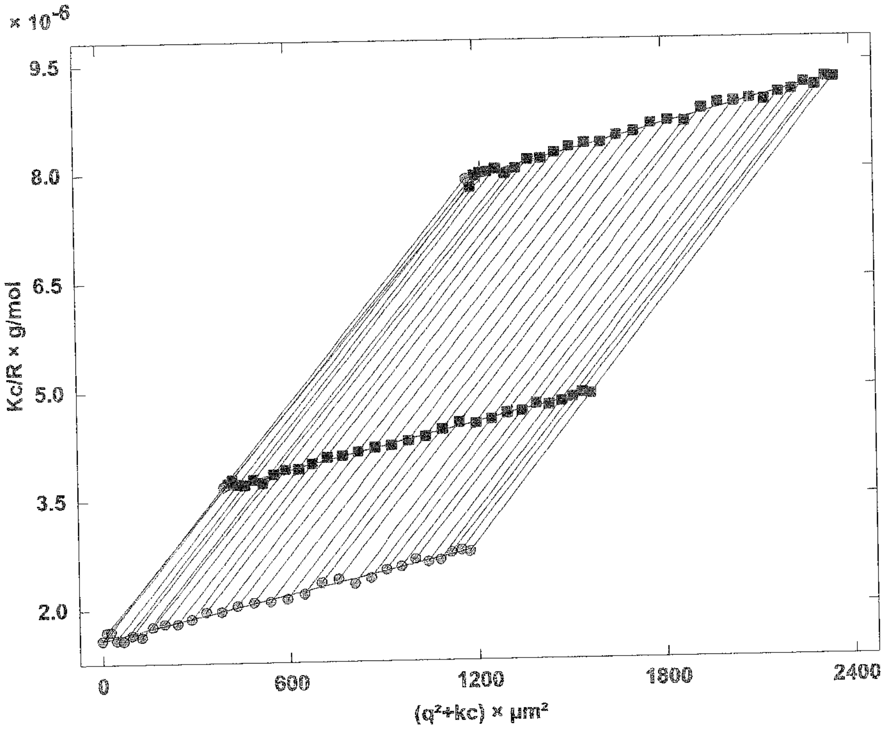
Detection: ALV/SO-SIPD DUAL photomultiplier / Pseudo-Cross correlation

DLS measurement conditions: Polystyrene in toluene, $c = 1$ g/l, 1 run a 1200 s, $\Theta = 90°$

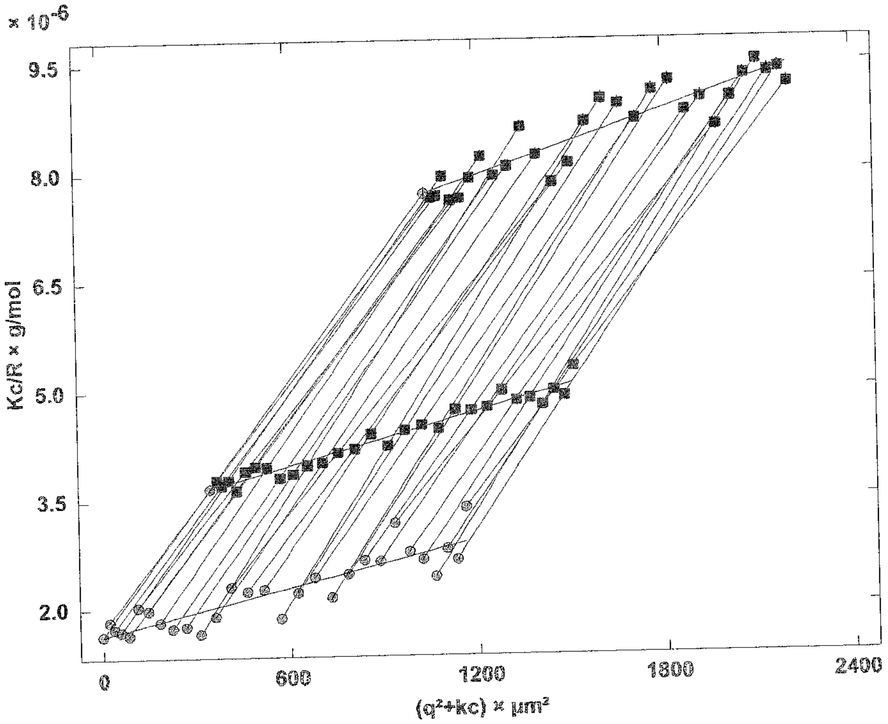


SLS measurement conditions: 3 runs a 20 s, Θ starting from 15° to 150°, step 5°,

detection with optical fiber with ALV / STATIC and DYNAMIC Enhancer:



detection with optical fiber without ALV / STATIC and DYNAMIC Enhancer:



Zimm analysis of the measured data:

without use of ALV / STATIC and DYNAMIC Enhancer:

File	Conc/(g/dm³)	Mw(app)/(g/mol)	<S²>(app)/µm²	Rg(app)/nm
Conc.=0	0.0000	614600	2.000e-3	44
PS1.dat	1.0000	273800	1.005e-3	31
PS1.dat	3.0000	129800	5.850e-4	24

Mw(c): 614600 g/mol (± 0 %) Mw(q^2): 614600 g/mol (± 4.72 %)
Rg: 44.72 nm (± 5.76 %) A2: 1.01e-6 mol dm^3/g^2 (± 0 %)

with use of ALV / STATIC and DYNAMIC Enhancer:

File	Conc/(g/dm³)	Mw(app)/(g/mol)	<S²>(app)/µm²	Rg(app)/nm
Conc.=0	0.0000	633800	1.933e-3	43
PS2.sta	1.0000	271600	8.701e-4	29
PS2.sta	3.0000	126700	4.449e-4	21

Mw(c): 633800 g/mol (± 0 %) Mw(q^2): 633800 g/mol (± 1.01 %)
Rg: 43.97 nm (± 1.26 %) A2: 1.05e-6 mol dm^3/g^2 (± 0 %)

Conclusion:

From the plots shown and the results of the Zimm analysis it is evident, that the use of the ALV / STATIC and DYNAMIC Enhancer decreases drastically the statistical variance of the measured mean intensity of the scattered light, which in turn reduces the error of estimation of physically relevant quantities as e.g., molecular mass, radius of gyration etc.. Furthermore, a reduction of correlated noise distribution to the baseline of the correlation function of more than one order of magnitude is achieved.