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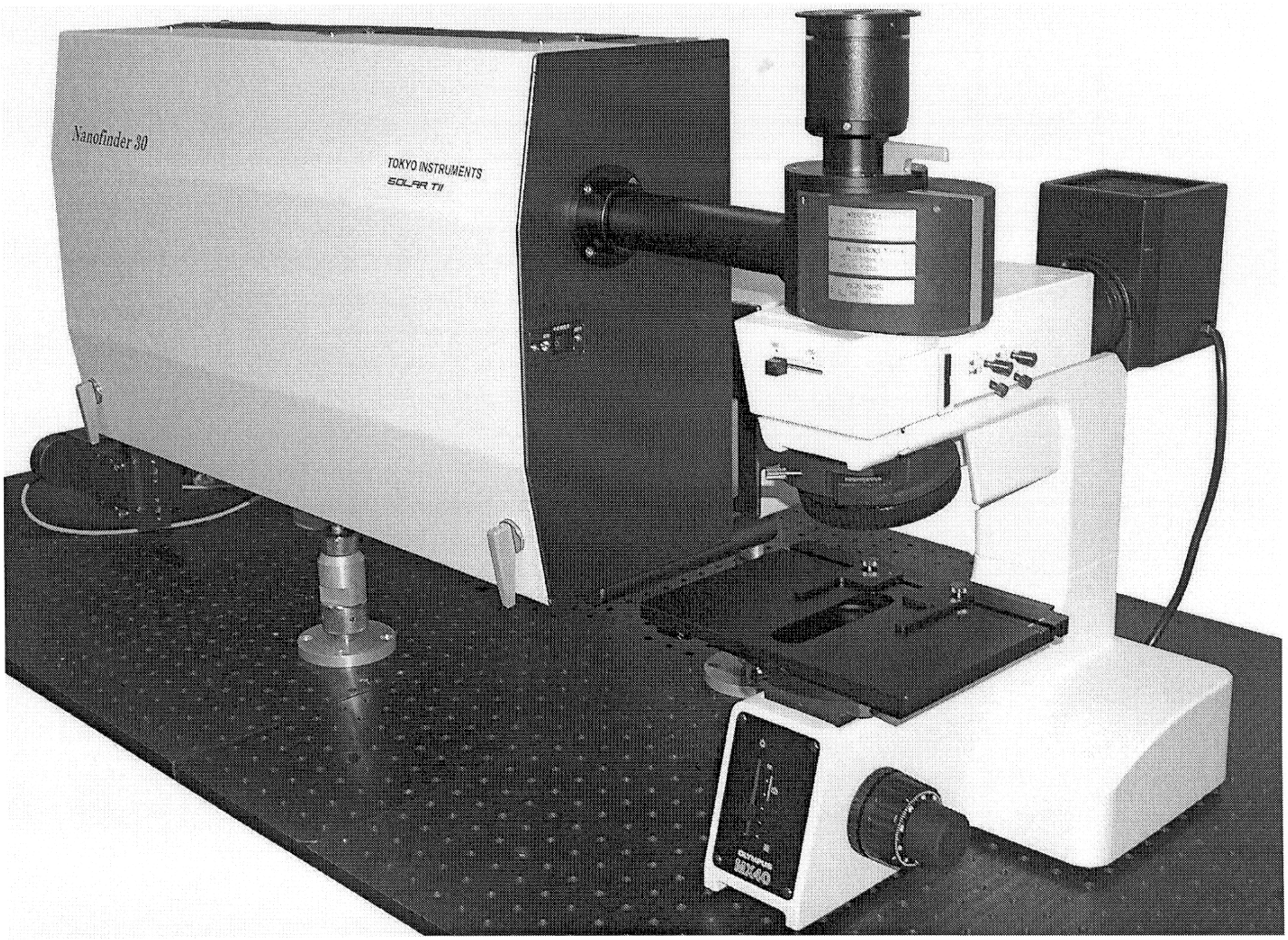

NANOFINDER-S

User's Manual

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1. INTRODUCTION

The 3-dimensional Raman Microscopy System *NANOFINDER-S* is an advanced nanotechnology instrument for micro/nanometer scale spectroscopy and imaging.

Key features

- ✓ 3-dimensional Raman and luminescent confocal imaging of an object with high spatial and spectral resolution
- ✓ 3-dimensional confocal topography via use of a laser microscope
- ✓ 3-dimensional imaging and chemical analysis - simultaneously
- ✓ analysis of objects having a complex structure
- ✓ detection of defects and stresses in semiconductors
- ✓ determination of ingredients in a pharmaceutical substance
- ✓ investigation of biological substances
- ✓ analyses in legal medicine
- ✓ nanotech studies
- ✓ environmental exploration and high-sensitivity detection of single molecules

The modular structure of the system, a wide range of choice for lasers and microscopes, utilization of automatic three-position units for operation with three lasers, availability of switching or running simultaneously three lasers with automatic tuning of mechano-optical units to provide maximum efficiency, quick determination of spatial distribution of a material with high spatial and spectral resolution with simultaneous construction of images by a laser confocal microscope or Raman / photoluminescent confocal microscope – all these advantages make the system flexible and capable of solving a variety of tasks.

The *NANOFINDER-S* is designed to be operated in compliance with the following ambient requirements:

■ ambient temperature	from +18°C to +22 °C
■ relative humidity	80% (max.) at 20 °C
■ atmospheric pressure	from 96 to 104 kPa

2. NANOFINDER-S STRUCTURAL ORGANIZATION

NANOFINDER-S has a modular design.

The principal construction units of the *NANOFINDER-S* are an optical-mechanical unit, a microscope, a laser, a scanning system and a detection system. The *NANOFINDER-S* is located on a vibration-proof optical bench.

2.1. Optical- mechanical unit (OMU)

The optical-mechanical unit is a system consisting of optics and mechanics sectors, power supplies and a control unit to control the mechanisms for OMU. The ports to connect a microscope, to deliver laser radiation, to attach a spectrometer or other equipment are also provided in the OMU.

2.2. Microscope

Either Inverted or Normal microscope with various microscope objectives (100x, 60x, 40x) are used.

2.3. Laser

It can be either a continuous or pulsed laser.

A variety of UV to IR lasers emitting at 244, 390, 442, 473, 488, 514, 532, 543, 633, 783 and 1064 nm can be used. The configuration of the *NANOFINDER-S* permits one, two or three lasers to be employed. This allows a system suitable for implementation of various tasks to be designed.

2.4. Scanning system

XY- piezo scanner
XY-galvanic scanner
Z-piezo scanner

Two options are offered for 3D scanning . Each of the option has its own advantages. Selection in favour of any of them can be made depending on a particular task.

- XY-piezo scanner + Z-piezo scanner;
- XY-galvanic scanner + Z-piezo scanner.

2.5. Detection system

- Standard Spectrometer

Model MS 5004i Imaging Monochromator/Spectrograph having a focal length of 520 mm with a 4-grating turret and two output ports to mount several types of detectors: PMT, MCP/PMT or APD and ICCD camera with cooling.

- Double monochromators, Echelles or other user-supplied spectrometers.
- PMT unit with interference filters

3. Optical-mechanical unit (OMU): structure and assignment of components

Fig.1 shows the OMU optic schematic diagram.

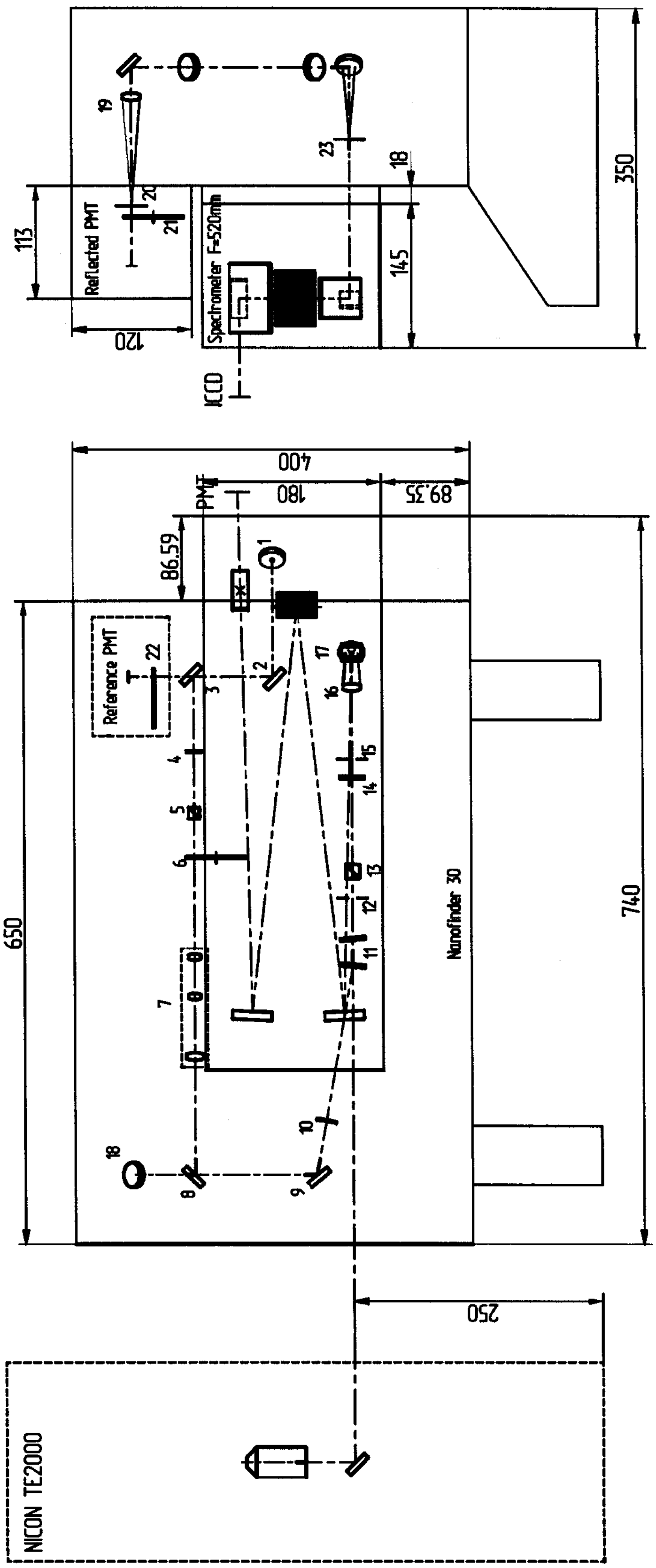


Fig.1 OMU optic schematic diagram (piezo-type, inverted microscope NIKON TE2000)

1 – beamsplitter; 2,3,8,9,18 – laser mirrors; 4 – plasma line filter; 5,13 – polarizer (Glan-Taylor prism); 6,21,22 – ND-filters; 7 – vario beamexpander; 10 – half wave plate; 11 – Edge or Notch filter (3-positioning); 12,15 – XY-adjusted irises; 14 – band-pass filters; 16,19 – objective XYZ-motorized; 17 – metal mirror; 20,23 – crossed slits (pinhole)

3.1. Delivery of laser radiation

3.1.1. Single Input port – this is used to deliver radiation emitted by one laser. The input port is equipped with a shutter that can be brought to any of the two positions - "Open" or "Closed".

3.1.2. Double input port - this is used to deliver radiation from two lasers.

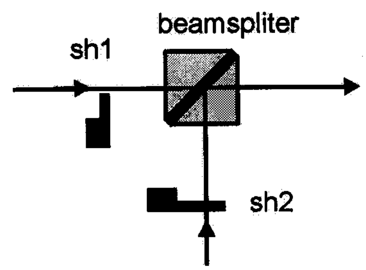


Fig. 2. Double input port

It has two input ports . Each port mounts a shutter (Sh1, Sh2). Beam splitter 1 (Fig.1) is used to deliver radiation from two lasers.

For operation with three lasers, one more beam splitter and one more shutter (Sh3) have to be used.

3.1.3. Laser radiation can be also delivered via fiber optics.

3.2. Plasma line rejection filter

In addition to the main laser line, gas lasers can also emit light at other wavelengths. Such light may arise, for instance, due to non-laser transitions in gas plasma. Although such lines are considerably weaker that the main laser line, their presence in a Raman spectrum is unwanted as they may introduce distortions or extra noise to a spectrum. Before the laser beam hits a sample, the laser radiation will become filtered by a plasma line rejection (PLR) filter (4, Fig.1) from the unwanted light. The filter is mounted in a holder to allow angular adjustment.

The specifications of a plasma rejection filter for the 488 nm-laser are given below as an example:

Central wavelength	488 + 0.4/-0.3 nm
Size	16 nm
Aperture diameter	> 6 nm
Half width	3±0.6 nm
Transmission	> 73%
Blocking	OD>5 (from UV to 900 nm)
Wave front distortion	< L/4 at 488 nm
Flatness	< L/4 at 488 nm
Parallelism	<30 arc.sec.

3.3. ND filter

In some cases an extremely intense radiation may cause damage to samples or give rise to increased background. To allow attenuation or continuous adjustment of laser radiation energy, circular variable ND - filters are installed in Excitation, Reference and Reflection channels (6, 22, 21, Fig.1).

The filter optical density can be continuously adjusted from 0 to 3.1.

Automatic control of ND filter is also provided.

3.4. Bandpass filters

A six-position filter wheel for interference filters can be also installed in the detection channel (14, Fig.1). Filter switching is effected automatically.

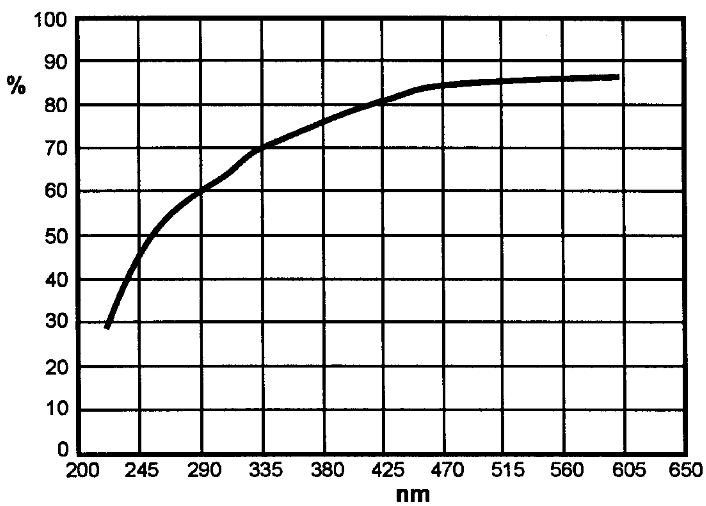
3.5. Polarizers

Polarizers to analyze the polarization of Raman stray or luminescent light are mounted in the excitation and detection channels. This provides helpful information for analysis of molecules. Thus, for instance, the internal viscosity of membranes and the effect of the membrane composition on their phase transitions were revealed in terms of the anisotropy of membrane-related luminophores. In many cases the polarization spectra are considerably more sensitive to small changes in the chemical composition of a compound than its absorption or emission luminescent spectra. This feature is taken into account when solving the problems related to the effect of various factors on the structure and chemical composition of molecules. Polarization spectra are also helpful in studying the multi-component luminescent light.

In the excitation channel, the polarizer position is set manually (15, Fig.1). In the detection channel, the polarizer position is controlled automatically (13, Fig.1). A Glan-Taylor prism is used as a polarizer here.

The specifications of a Glan-Taylor prism are given below as an example:

Wavelength range	350 to 2300 nm
Material	calcite
Polarization value	$<1\times10^{-5}$
Aperture diameter	13 mm
Angular field	7.7°



transmission at 350 nm	>70%
transmission at 500 nm	~85%
max. transmission	>85%

Fig. 3. Typical plot for Glan -Taylor prism transmission

3.6. Half-wave plate

A half-wave plate is used for rotation of the polarization of laser radiation. Rotation of polarization to within 90° is provided.

A three-position holder for three half-wave plates with automatic position switching and half-wave tuning for the selected laser is installed in the system (10, Fig.1)

3.7. Beam expander

The beam expander is a telescope to adjust the diameter of the laser beam with maintaining the beam collimating property.

3.7.1. Beam expander optic schematic diagram

The optic schematic of the beam expander uses the configuration of a Galilean telescope. The focal length f_I' of ocular I, consisting of two units, ranges from *–12,5 mm* to *–50 mm*. The distance *d* between ocular I and objective lens II is here adjusted so that with all displacements of the units, the points of rear F_I' and front F_{II}' focuses of the objective lens always remain aligned. The focal length of the objective lens is $f_{II}' = 90 мм$.

Fig.4 shows the diagram of the beam expander optic schematic.

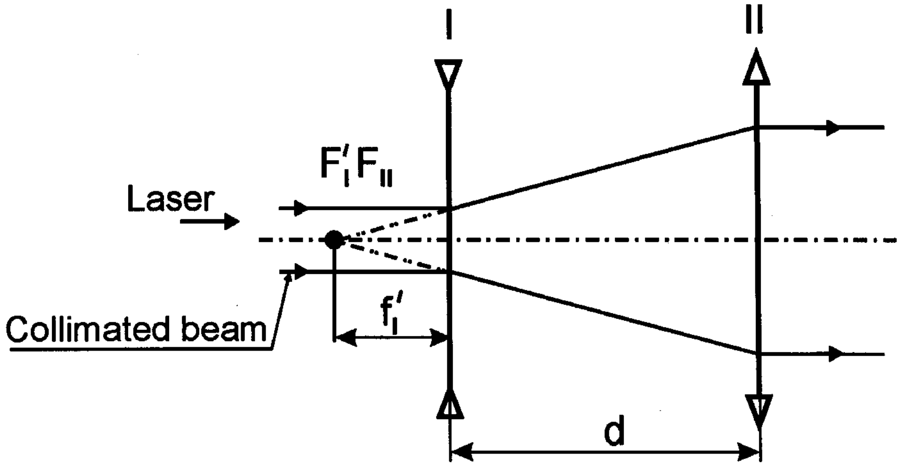


Fig. 4. The beam path in the telescope with a variable magnification factor (beam expander)

3.7.2. Beam expander design

The beam expander is formed by optic components such as a movable ocular part, an immovable ocular part and an objective. The magnification factor of the beam expander is determined by the position of the movable ocular part and that of the objective with respect to the position of the immovable ocular part. These positions are set with use of two step-motor drives.

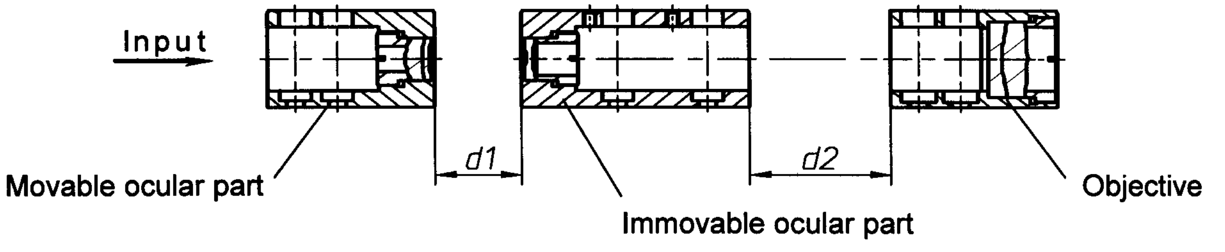


Fig. 5

Below follows a table containing the telescope magnification factors and the required number of steps for two drives.

Telescope magnification factor	***d1*,mm**	**Number of steps. *N1***	***d2*, mm**	**Number of steps. *N2***
1.78	11.8	549	21.4	1179
2.02	11.3	891	23.0	2242
2.27	10.8	1234	24.2	3070
2.51	10.3	1577	25.1	3736
2.76	9.8	1920	25.9	4283
3.00	9.3	2263	26.6	4738
3.26	8.8	2606	27.2	5128
3.50	8.3	2949	27.7	5462
3.75	7.8	3291	28.2	5751
3.98	7.3	3634	28.5	6005
4.24	6.8	3977	28.8	6229
4.48	6.3	4320	29.1	6429
4.74	5.8	4663	29.4	6608
5.00	5.3	5006	29.6	6769
5.27	4.8	5349	29.8	6915
5.46	4.3	5691	30.0	7048
5.71	3.8	6034	30.2	7170
5.95	3.3	63.77	30.3	7281
6.20	2.8	6720	30.5	7384
6.45	2.3	7063	30.6	7478
6.71	1.8	7406	30.7	7566
6.94	1.3	7749	30.8	7648
7.19	0.8	8091	31.0	7723

3.8. Edge or Notch filters

These are designed to ensure two functions: to reflect efficiently the exciting laser radiation and to separate Raman or luminescent light from Raleigh stray light.

A Notch filter is only used for operation in the anti-Stokes region. The disadvantage of a Notch filter is the poor quality of the reflected laser beam as, for instance, the presence of focused satellite spots around the main laser spot. Therefore in all other cases it is advisable to use an Edge filter which has no such disadvantage.

Specifications of an Edge filter for the 488 nm laser wavelength are presented below as an example.

Size	dia. 25+0/-0.1 mm
Thickness	3.5±0.1 mm
Optical aperture	dia.>/= 21 mm
Flatness	L/2 at 488 nm
Parallelism	</= 30 arc.s
Wave front distortion	L/4 at 488 nm
Transmission (average)	85% from 500 to 750 nm
Blocking	>/= OD5 from UV to 488 nm

To provide simultaneous operation with three lasers, a three-position positioner for Notch and Edge filters is used in the microscope (11, Fig.1). Filter switching is effected automatically according to the excitation laser wavelength.

3.9. Three-coordinate automatic positioner for objective

To focus Raman or luminescent light in the Registration channel and sample-scattered or laser light in the Reflection channel, objectives 16, 19 (Fig.1) are installed, respectively. The three-coordinate (X,Y,Z) automatic positioner provides fast and fine adjustment of the objective's position , focusing the light into a pinhole. The travel range of the objective along the X,Y coordinates with a 0.7 μm step is ±1.5 mm, while that along the Z axis is ±3 mm.

3.10. Pinhole

To ensure a confocal image both in the modes of a laser confocal microscope and Raman/ luminescence confocal microscope, a pinhole has to be used. An automatic double-crossed slit (20,23, Fig.1) is employed for a pinhole. This allows continuous adjustment of the pinhole size from 0 to 1.5 μm along the two coordinates simultaneously.

Pinhole specifications

Width	0 to1.5 mm
Height	0 to 1.5 mm
Step size	0.5 μm
Parallelism	±1 μm
Reading accuracy	±10 μm

Switching between the confocal and non-confocal modes is effected easily by the adjustment of the pinhole size.

3.11. SERS-option

One-or two-photon fluorescence excitation methods are extensively used at present to explore molecules. SERS is a very helpful method as it allows a Raman signal produced by molecules coupled to nanometric metallic structures to be strongly amplified. The amplification mechanism has both the electromagnetic and chemical nature. However to date the amplification mechanism has not been quantitatively described fully.

SERS signals were detected from the substances adsorbed in the surface layers of metals such as Ag, Au, Cu, Li, Na, K, Rb, Ni, Ti, Co, Al, Pd, Pt, Rh, Cd, Ga, In, and also oxides: ZnO, Ag_2O_3, TiO_2, NiO, Fe_3O_4 and other compounds such as AgCl, AgBr, AgI, CdS, GaP. A vibrational SERS spectrum contains plenty of information about the structure of molecules. But because the vibration time of relaxation is shorter than its electronic time, the number of Raman photons that can be emitted per unit time by a molecule under saturation is considerably larger than the number of fluorescent photons. This allows the detection time of a molecule to be minimized. Besides, molecules under study do not suffer from photodecomposition as the excitation energy is not in resonance with the molecular transitions.

The extension of SERS investigations will allow to discover new compounds or to explain the mechanism of the SERS effect and also may prove to be very helpful in practical applications.

The SERS telescope with a magnification factor of -1^x uses the Kepler configuration.

Fig.7 shows the optic schematic diagram for the SERS option.

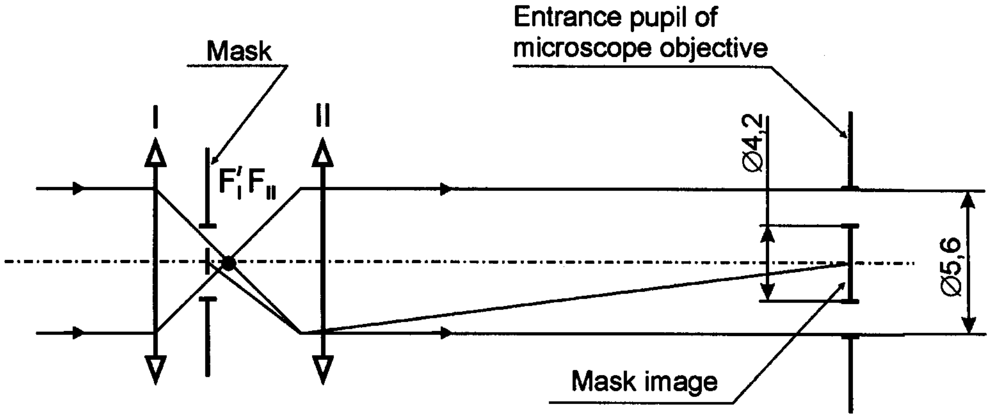


Fig. 7. SERS-option Optic Schematic Diagram

Objective lens II with focal length $f' = 41$ mm is projecting a 0.19 mm-dia -mask into the pupil of the objective with a linear magnification of -22^x.

4. Monochromator/Spectrograph MS 5004i

MS 5004i specifications (grating - 1200 l/mm)

System	
Configuration	vertical
Ports	1 input, 2 output
F/number (input)	9.8
Focal length	520 mm
Stray light	$1x10^{-5}$ (20 nm from 633 nm laser line)
Flat field	25mmx10mm
Gratings	
Grating mounts	4 -position turret
Grating rotation	Optical center @ grating face
Grating size	40x55x10 mm
Grating selection repeatability (image vertical position)	±0.049 mm
Wavelength	
Grating rotation range	0 to 1440 nm
Reciprocal dispersion (λ=600 nm)	1.529 nm/mm
Wavelength resolution*	0.03 nm
Wavelength accuracy	± 0.045 nm
Wavelength repeatability	± 0.011 nm
Wavelength step size (aver)	$1.68x10^{-3}$ nm

* This parameter was obtained with a 14μm pixel size CCD

Motorized Exit Slits	
- slit width control	motor(micrometer) driven slit assembly
- slit width	variable from 0 to 2.0 mm
- parallelism	±1 μm
- accuracy	±10 μm
- micrometer screw reading accuracy	2 μm
- step size	0.5μm

Table and plot of wavelength dependence of dispersion for 1200 l/mm grating

λ,nm	*nm/mm*	*λ,nm*	*nm/mm*
200	1,591	750	1,477
250	1,589	800	1,456
300	1,585	850	1,433
350	1,579	900	1,408
400	1,572	950	1,380
450	1,564	1000	1,349
500	1,554	1050	1,316
550	1,542	1100	1,280
600	1,529	1150	1,240
650	1,513	1200	1,196
700	1,496		

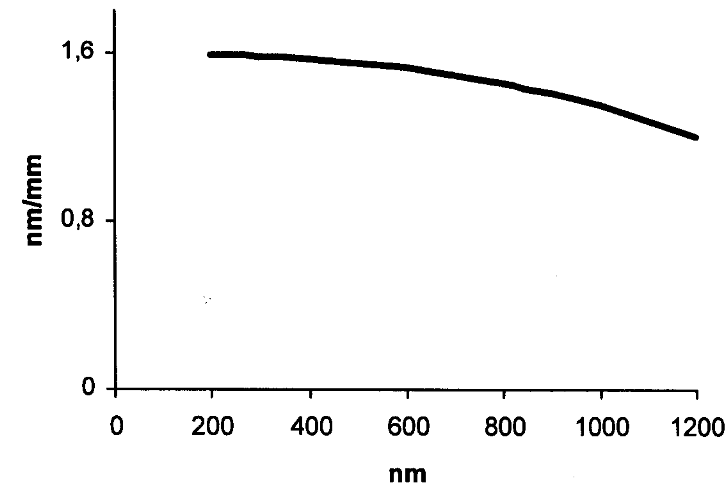


Fig. 6.

To provide the required spectral region and spectral resolution a variety of diffraction gratings (ruled or holographic) with different number of grooves from 100 l/mm to 3600 l/mm are offered.

One of the advantages of the MS 5004i design is the availability of using Echelle gratings ensuring high spectral resolution.

5. Reference unit

To account for the intensity drift of laser radiation, the Reference unit is used. The Reference unit is made as a separate module and can be easily mounted on the optical-mechanical unit. The Reference unit comprises a detector and a ND filter unit with a filter of variable density to allow continuous automatic adjustment of the laser radiation energy.

6. Reflection unit

The Reflection unit provides the system operation in the mode of a laser confocal microscope. It is fabricated as a separate module and can be easily mounted on the optical-mechanical unit.

The Reflection unit comprises:

- a three-coordinate automatic objective positioner with an objective;
- a double-crossed pinhole with continuous automatic adjustment;
- ND-filter unit with a variable-density filter;
- a PMT

7. Description of optic schematic for galvanic mirrors unit

With the "XY-galvanic scanner + Z-piezo scanner" version , galvanic mirrors are used as scanning components for scanning along the XY-axes.

Fig.8 shows the optic schematic diagram for the galvanic mirrors unit.

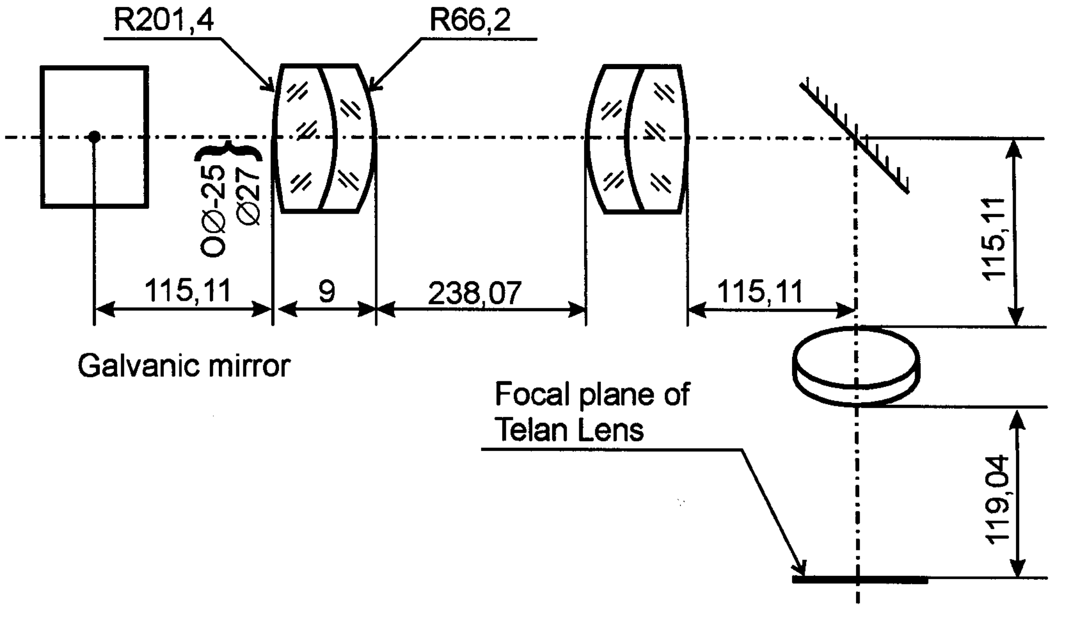


Fig. 8. Optic schematic diagram for galvanic mirrors unit

The optic schematic is formed by two galvanic and three turning mirrors and also by three achromatic lenses.

The galvanic mirrors ensure scanning of an object by a laser beam along the X and Y axes. The lenses (F – 120 mm) are used to obtain the required linear dimensions of the scan field and to align the field of view and the pupil of the microscope with those of the *NANOFINDER-S.*

8. Description of optic schematic for different modes of *NANOFINDER-S* operation (See Fig. 1)

8.1. Excitation optics sector

Depending on the shutter's position, the laser beam can either be reflected by beam splitter (1) or travel though the latter. Laser mirrors (2,3) direct the beam to beam expander (7). A small part of the radiation passes through mirror (3) in the Reference unit and is used as reference radiation. Before the laser beam hits beam expander (7), it will have to travel through plasma line rejection filter (4), polarizer (5) and ND-filter (6). The beam expander allows adjustment of the laser beam diameter with maintaining the beam's collimating property. Laser mirrors (8,9) route the laser beam through half-wave plate (10) to beam splitter unit (11). The laser beam reflected by Edge or Notch filter is directed to the microscope and is then focused to the sample with a microscope objective.

8.2. Laser confocal microscope mode

The sample-scattered laser radiation is being collimated by a microscope objective to be further routed back. After being reflected by Edge or Notch filter (11), the beam passes through half-wave plate (10) and is then reflected by laser mirror (9) to laser mirror (8). A small part of the radiation passes through mirror (8) in the Reflection unit. Objective (19) focuses the beam into pinhole (20). If necessary , the light is attenuated by ND-filter (21) to hit then a PMT.

8.3. Raman/luminescent microscope mode

Fluorescent and/or Raman light, emitted by excited molecules, is collimated by a microscope objective. The light travels through Edge/Notch filters (11), polarizer (13) and is then focused by objective (16) to pinhole (23) mounted on the input of the MS 5004 Monochromator/Spectrograph. One of the gratings (selected according to the required spectral region and spectral resolution) directs the light, depending on the position of the Spectrograph's exit mirror, either to a CCD or to a PMT. To build up a

Raman/ luminescent image of an object, the operational wavelength region is detected on a CCD, or it is determined by the size of the Monochromator's exit slit, if a PMT is in use.

If a mirror is installed in the Monochromator instead of a grating, the required wavelength region is then selected with use of interference filter (14).

Fig.9 shows the appearance of NANOFINDER-S in the following configuration:
- piezo – type with Standard Spectrometer MS 5004i;
- double input port;
- single-position unit for connection with an Inverted microscope (NIKON TE2000).

Fig.10 presents the location of the construction units inside the Optical-Mechanical Unit (the version for normal microscope).

9. PRE-OPERATION AND OPERATION PROCEDURES

9.1. After mounting all of the *NANOFINDER-S* system's components on the vibration-proof optical bench, carry out cabling attachment as per Fig. 11 using the cables from Standard Equipment.

9.2. Couple the communication connector of an external PC to *NANOFINDER-S* Interface connector "RS-232C" using Interface cable from Standard Equipment.

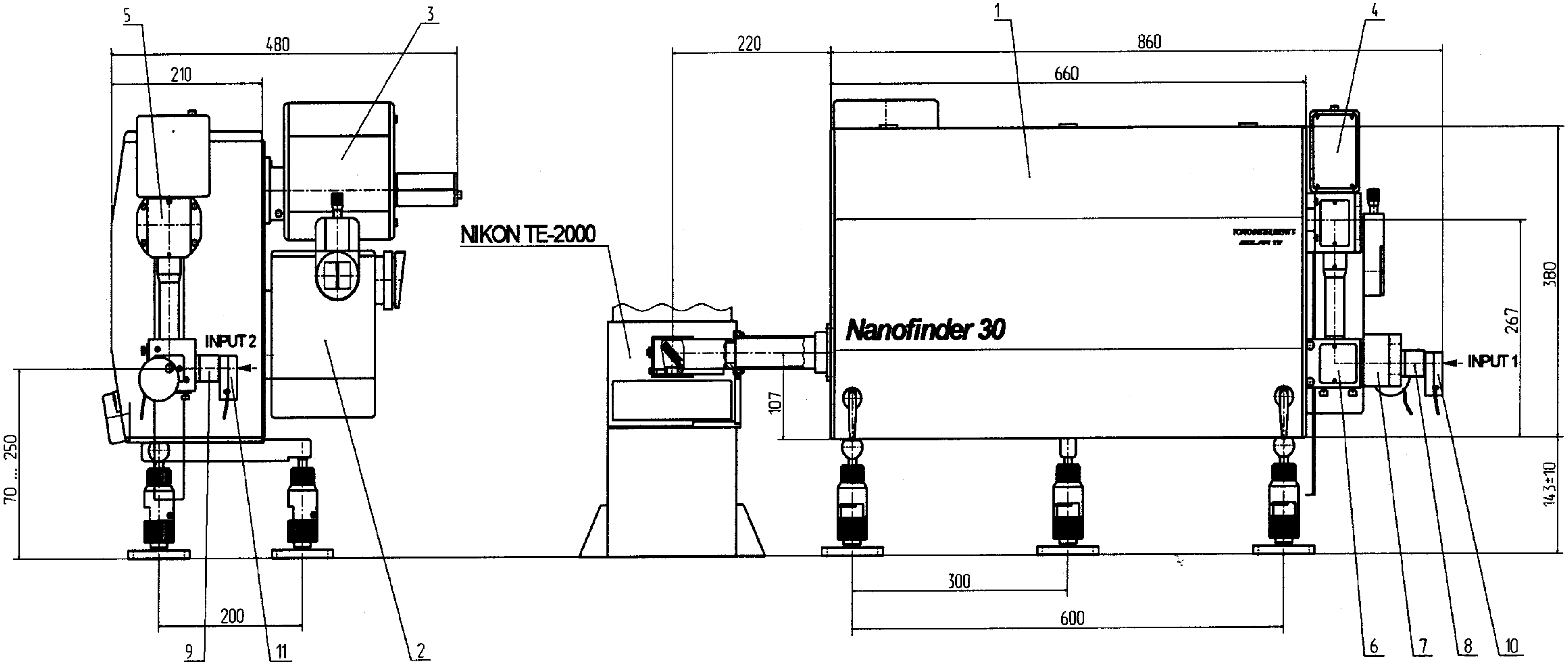
9.3. Bring Power Supply Adapter mains switch to the OFF position and connect Power Supply Adapter to the 100-240 VAC, 47-63 Hz line using the mains cable from Standard Equipment.

NOTE: Cable attachment must be performed with** NANOFINDER-S **and PC being completely powered off!

9.4. Move the Power Supply Adapter mains switch to the ON position. Be sure its indicator-lamp has come on and a single audible sound has been produced to evidence the *NANOFINDER-S* normal operation. Since then *NANOFINDER-S* is ready for operation.

9.5. The *NANOFINDER-S* is a completely automated device. It can be operated from a PC with the use of Control Program that comes in a CD together with the instrument.

PC configuration.
- IBM compatible.
- Windows 95/98/ME/NT/2000/XP operating system.



1 - Optical-mechanical unit. 2 - Spectrometer. 3 - Reflected PMT. 4 - Reference PMT. 5,6 - Turning mirrors units. 7 - Beamcombiner unit. 8,9 - Plasma line filter units. 10,11 - Shutters

Fig. 9. *NANOFINDER-S* appearance schematic representation

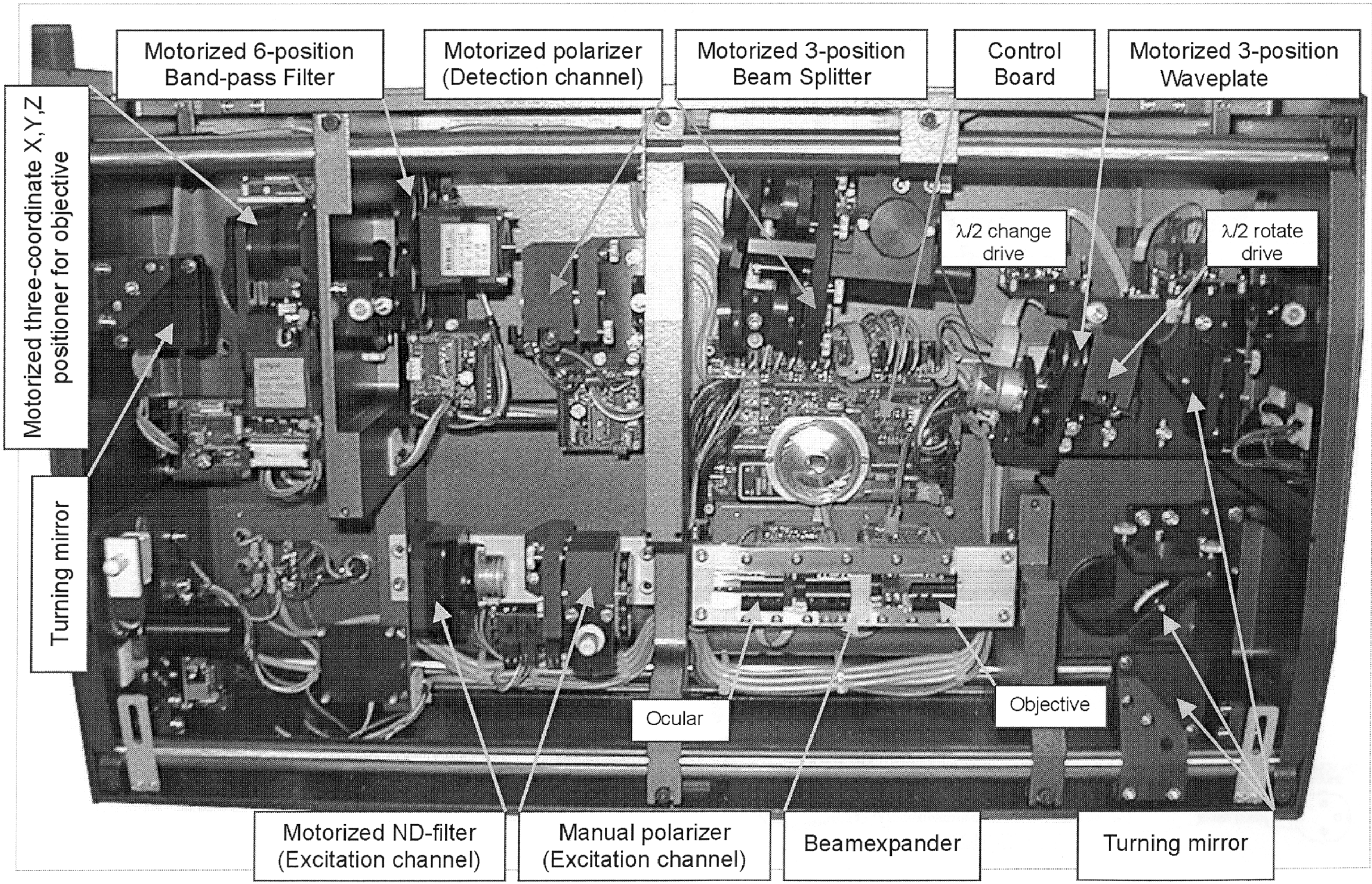

Motorized 6-position Band-pass Filter
Motorized polarizer (Detection channel)
Motorized 3-position Beam Splitter
Control Board
Motorized 3-position Waveplate
Motorized three-coordinate X,Y,Z positioner for objective
λ/2 change drive
λ/2 rotate drive
Turning mirror
Ocular
Objective
Motorized ND-filter (Excitation channel)
Manual polarizer (Excitation channel)
Beamexpander
Turning mirror

Fig. 10

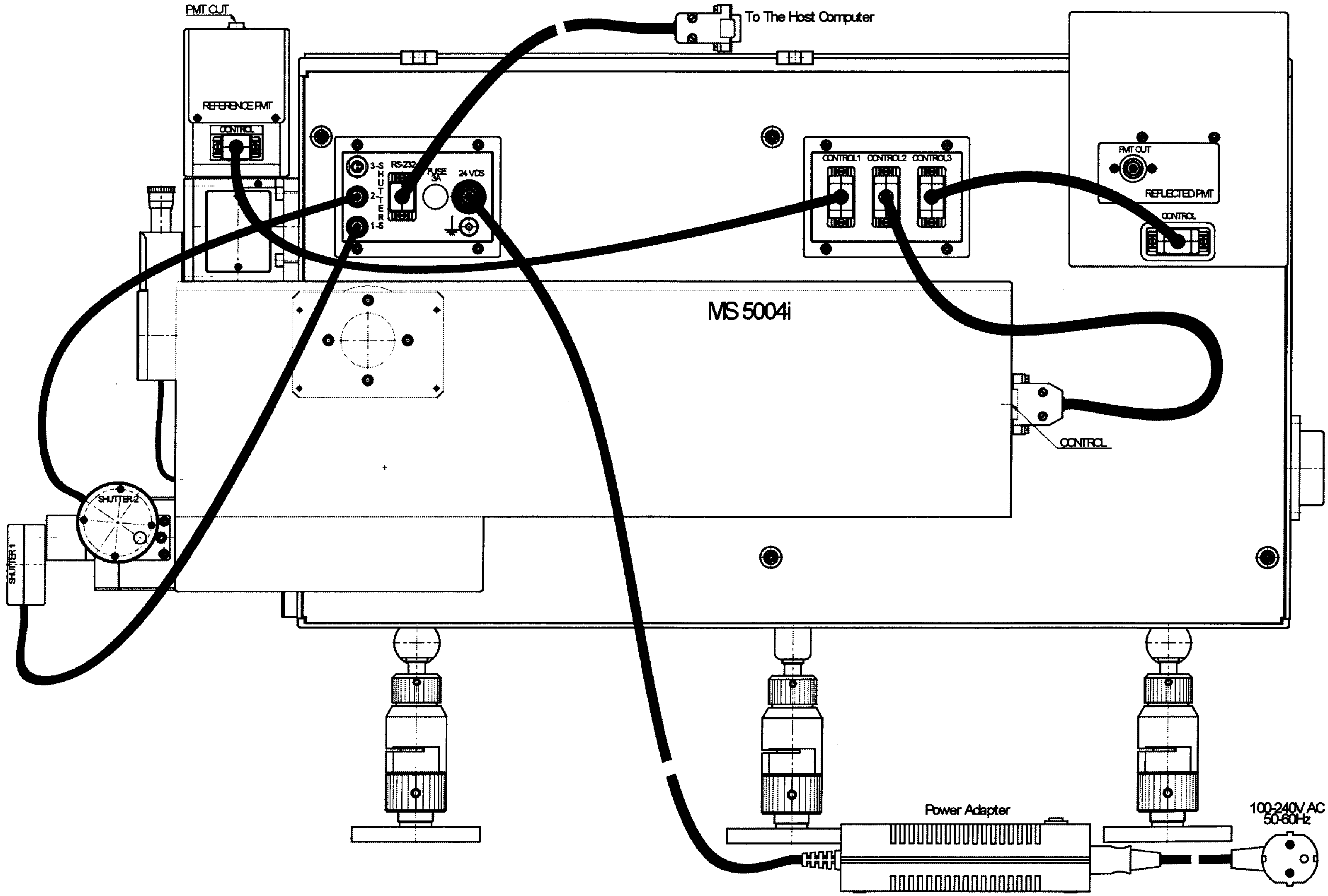


Fig. 11

NANOFINDER-S **Cabling**