



SPM Solver PRO Instruction Manual

Conducting Measurements in Liquid

NT-MDT Co., Zelenograd Research Institute of Physical Problems 124460, Moscow, Russia tel.: +7(095) 535-03-05 fax: +7(095) 535-64-10 e-mail: spm@ntmdt.ru http://www.ntmdt.com



Windows Control Program

- **Notice:** A delivery set of your device may differ from the set described in the given manual. For more detailed information refer to the specification of your contract.
- **Copyright:** No part of this manual for no purposes may be reproduced or transmitted in any form and by any means, electronic or mechanical, including photocopying and video-recording, without a written permission from the NT-MDT company.

GENERAL SAFETY MEASURES

• Observe safety measures for operation with electrical installations. Prior to begin operation with the device, provide its grounding. Prior to connect or disconnect the connectors, turn off the device. Connecting or disconnecting of the connectors in the process of work may lead to damage of the electrical circuit and death of the device. Warning label is located on the back panel of electronics block.



• Observe safety measures for operation with instruments having laser radiation source. Do not stare into beam. Warning label is located on the measuring head.



- Do not disassemble any part of the device! Disassembling of the product is permitted only for experts certified with the NT-MDT company.
- Do not connect additional devices to the instrument without advice from NT-MDT specialists.
- Protect the device from powerful mechanical effects.
- Protect the device from the effect of high temperatures and liquids ingestion.
- When transporting, provide packaging of the device which eliminates damage in the process of transportation.

Conducting Measurements in Liquid. Instruction Manual

Table of Contents

1.	SCA	NNING	MEASURING HEAD FOR OPERATION IN LIQUID	4
	1.1. 1.2.	BASIC INFORMATION		
		Design		
		1.2.1.	Probe Holder for Operation in Liquid	7
		1.2.2.	Probe Holder for Operation in Air	8
2.	LIQ	UID CE	LLS	10
3.	PRE	PARAT	ION FOR MEASUREMENTS IN DROP	14
	3.1.	PROBE	INSTALLATION	14
	3.2.	PROBE	HOLDER INSTALLATION	15
	3.3.	SAMPL	E INSTALLATION	16
	3.4.	ADJUS'	TMENT OF THE IENGTH OF MEASURING HEAD THREADED SUPPORTS	16
	3.5.	LASER	BEAM ALIGNMENT ONTO THE CANTILEVER	18
	3.6.	MEASU	JRING HEAD INSTALLATION	21
4.	PRE	PARAT	ION FOR OPERATION WITH THE LIQUID CELL	22
	4.1.	MEASU	JRING HEAD PREPARATION	23
		4.1.1.	Diaphragm Installation	
		4.1.2.	Probe Installation	24
		4.1.3.	Probe Holder Installation	
		4.1.4.	Laser Beam Aligning onto the Cantilever	
	4.2.	PREPA	RATION OF LIQUID CELL	
		4.2.1.	Sample Installation	
		4.2.2.	Initial Approach	
		4.2.3.	Assembling the Cell and its Installation on the Approach unit	
	4.3.	MEASU	JRING HEAD INSTALLATION	
5.	ADJ	USTING	G THE SYSTEM FOR DETECTING THE CANTILEVER DEFLECTION	
	5.1.		TING WITH THE OPTICAL VIEWING SYSTEM	
	5.2.	ADJUS	TING WITHOUT USING THE OOPTICAL VIEWING SYSTEM	34
6.	INST	TALLA]	TION OF A PROTECTIVE HOOD	
7.	FEA	TURES	OF CONDUCTING THE MEASUREMENTS IN LIQUID	
	7.1.	CONTA	.ст AFM	
	7.2.	SEMICONTACT AFM		
		7.2.1.	Approach	
		7.2.2.	Setting the Piezodriver Working Frequency	
		7.2.3.	Preparation for Scanning	
		7.2.4.	Scanning	
		7.2.5.	Saving the Data	
		7.2.6.	Finishing the Operation	

AT	TACE	IMENT					
1.	SEM	SEMICONTACT AFM. SETTING THE WORKING FREQUENCY IN					
	AUT	OMATIC MODE					
	1.1.	SETTING THE PIEZODRIVER WORKING FREQUENCY					
	1.2.	Approach					
	1.3.	PREPARATION FOR SCANNING					

1. Scanning Measuring Head for Operation in Liquid

1.1. Basic Information

Scanning measuring heads models SFC050L and SFC100L (Fig. 1-1) are designed for conducting measurements in liquid using basic AFM techniques. Exchangeable probe holders also enable the heads to be used for measurements in air and in gaseous media.



a) SFC050L

b) SFC100L

Fig. 1-1. Scanning measuring heads for liquid operation

Two types of measurements are available with the scanning measuring heads for operation in liquid:

- in a drop;
- in a liquid cell.

The scanning measuring head can also function as a standalone unit. In this case the sample is secured on a laboratory desk and the head is placed above the sample or just on to of it, thus enabling the observation of samples of arbitrary dimensions.

The scanning measuring heads models SFC050L and SFC100L are intended for measurements using the following modes and techniques:

- Contact AFM;
- Semicontact AFM;
- Lateral Force Imaging;
- Adhesion Force Imaging;
- Force Modulation Mode;
- Phase Imaging Mode;
- AFM Litography.

Specifications

Scanning measuring head SFC050L

Table 1-1

Parameter	Value
Scanning range	50x50x5 μm
XY linearity error	0.15%
Z linearity error	1%
RMS noise on XY in 200 Hz band	≤0.2 nm
RMS noise on Z in 1000 Hz band	<0.1 nm
Error in determining XY linear dimensions	± 1.2%
Sample size	up to Ø100x15 mm
Sample mass	up to 300 g
Dimensions	113x132x113 mm
Mass	0.8 kg
Laser wavelength	650 nm
Liquid depth	0.5÷7 mm

Scanning measuring head SFC100L

Table 1-2

Parameter	Value
Scanning range	100x100x10 μm
XY linearity error:	
in fast-scanning direction	0.4 %
in slow-scanning direction	0.8 %
RMS noise on XY in 100 Hz band	0.02 nm
RMS noise on Z in 1000 Hz band	<0.1 nm
Error in determining XY linear dimensions	5%
Sample size	up to Ø100x15 mm
Sample mass	up to 300 g
Dimensions	113x132x113 mm
Mass	0.8 kg
Laser wavelength	650 nm
Liquid depth	0.5÷7 mm

1.2. Design

The main components of the measuring head are shown in Fig. 1-2.

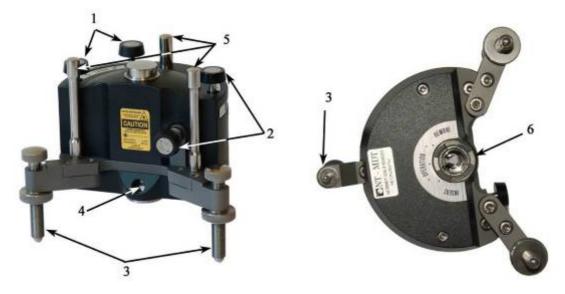


Fig. 1-2. Main components of the scanning measuring head 1 – laser adjustment screws; 2 – photodiode adjustment screws; 3 – threaded supports; 4 – mirror; 5 – props; 6 – probe holder seat

Scanning measuring heads for operation in liquid are supplied with exchangeable probe holders of the two types:

- for operation in liquid (<u>Fig. 1-3</u>);
- for operation in air (Fig. 1-4).



Fig. 1-3. Exchangeable probe holder for operation in liquid



Fig. 1-4. Exchangeable probe holder for operation in air

The holder is fixed on the measuring head by magnets (Fig. 1-5). Four ferromagnetic sectors are glued to the holder from below (Fig. 1-6). To avoid shocks when mounting the holder on the measuring head, the holder is first positioned so that the ferromagnetic inserts fall between the magnets. Then the holder is turned until the inserts coincide with the magnets, thus providing magnetic clamping to the scanner tube.



Fig. 1-5. Magnets for fixing the probe holder

Fig. 1-6. Probe holder, view from below. The arrows show the location of the ferromagnetic sectors

1.2.1. Probe Holder for Operation in Liquid

Fig. 1-7 shows the probe holder for operation in liquid. Its design allows to conduct measurements in liquid as well as in air and in gaseous media (when using the liquid cell).

The sketch of the probe holder is given in <u>Fig. 1-8</u>. It consists of disk 1 with the base on which glass pedestal 2 is mounted. The probe is fixed on the pedestal 2 by means of removable clip 3, the lower end of which is inserted in slot 4 in the base.



Fig. 1-7. Probe holder for operation in liquid

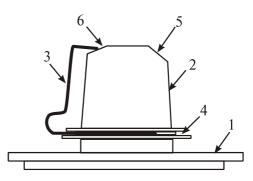


Fig. 1-8. Main parts of the probe holder for operation in liquid
1 – disk with the base; 2 – glass pedestal;
3 – removable clip; 4 – slot;
5 – beveled side for installing the probe for operation in air; 6 – beveled side for installing the probe for operation in liquid

The glass pedestal has two beveled sides 5 and 6 in its upper part serving as seats for the probe. The tilting angles of the sides differ, side 6 with the smaller angle being intended for the installation of the probe for operation in liquid, while side 5 with the larger angle is meant for operation in air and in gaseous media.

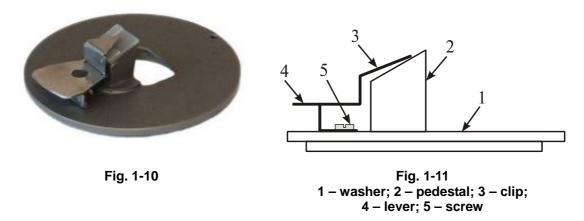
To install the clip take it with your fingers near its bend and insert the lower part of the clip into slot 4 in the base, see Fig. 1-9.



Fig. 1-9

1.2.2. Probe Holder for Operation in Air

The probe holder for operation in air is shown in <u>Fig. 1-10</u>. The main parts of the holder design are represented schematically in <u>Fig. 1-11</u>. It consists of disk 1, pedestal 2 and clip 3 with lever 4. The clip is fixed on the disk with screw 5. Four ferromagnetic sectors are glued to the disk from below to provide magnetic clamping to the measuring head scanner tube.



The preparation procedure for using the probe holder in air is similar to that for scanning measuring heads (see "Solver PRO Instruction Manual") except for the installation of the probe.

Probe Installation

- 1. Put the holder on the desk.
- 2. Using tweezers take a probe from the case and bring in to the holder. Do not turn the chip over as the probes are placed in the case with their tips pointing upwards.

- 3. Press the clip lever while keeping the holder, as shown for example in Fig. 1-12. Install the probe and release the clip.
- 4. If required, adjust the probe position using the tweezers.



Fig. 1-12. Probe installation



Fig. 1-13. The probe installed in the holder

Probe holder installation

1. Bring the holder to its seat on the head. The holder shall be rotated 45° relative to its working position and the positioning marker (spot) on its ring shall coincide with **INSERT** marker on the lower part of the measuring head housing, see Fig. 1-14.







Fig. 1-15. Probe holder working position

- 2. Secure the holder in its seat. The holder shall rest on the magnetic support evenly and without skew.
- 3. Turn the holder ring clockwise until the positioning marker on the ring coincides with **OPERATION** marker on the lower part of the measuring head housing, Fig. 1-15. This is the holder working position.

Removal of the probe holder from the measuring head

To remove the holder turn it 45° clockwise in **REMOVE** position and take the holder from its seat on the measuring head.

2. Liquid Cells

Designation

Liquid cell MP3LC (Fig. 2-1) is used together with scanning measuring heads SFC050L and SFC100L. It is designed for conducting measurements in liquid and in gaseous media.



Fig. 2-1. Liquid cell MP3LC

The cell is made of chemically neutral materials (silicon rubber, titan, Teflon, polycrystalline sapphire and stainless steel) that are resistant to the solutions of acids, such as H_2SO_4 and HCl. Limitations on the liquid (gas) are determined by the materials contacting with this liquid or gas.

Specifications

Table 2-1

Parameter	Value	
RMS noise on Z with SFC050L and SFC100L	<0.1 nm	
Sample size	up to 15x15x3 mm	
Volume of liquid	4 ml	
Cell dimensions	Ø70x24 mm	
Material	Titan and Teflon	
Cell mass	160 g	

Components



Fig. 2-2 shows the components required for the operation of MP3LC liquid cell.

Fig. 2-2. Components of the liquid cell 1 – cell; 2 – diaphragm; 3 – cell holder; 4 – fitting tool; 5 – cover

The components of the liquid cell include:

- cell 1 itself with the outlets for feeding liquid (or gas);
- diaphragm 2 that is put on the probe holder, sealing the cell from the top;
- cell holder 3 on a unified carriage for fixing on the universal stage;
- fitting tool 4 required for mounting the holder with the diaphragm on the measuring head;
- cover 5 for closing the cell during transportation and storage.

For measurements with the liquid cell, diaphragm 2 is put on the special probe holder as shown in Fig. 2-3 (the description of the holder see in par. 1.2.1 on page 7) The diaphragm is made of silicon rubber and is fixed in metal ring 4.

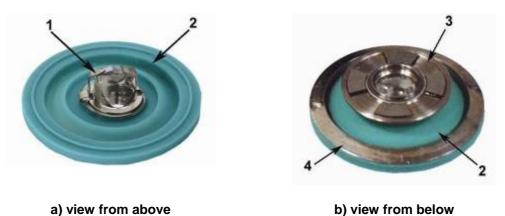


Fig. 2-3. Probe holder inserted into the diaphragm 1 – probe holder; 2 – diaphragm; 3 – probe holder washer; 4 – ring

To prevent the diaphragm from damage while fixing the holder on the measuring head the fitting tool shall be used (see item 4 in <u>Fig. 2-2</u>) This fitting tool has two right sides for aligning the holder in set-up and working positions.

Design

Fig. 2-4 show the parts forming the cell.



Fig. 2-4. Parts of MP3LC cell 1 – base; 2 – flange with clamping device; 3 – substrate; 4 – Teflon ring with an o-ring; 5 – fit ring; 6 – outlets for feeding liquid; 7 – cover

A polycrystalline sapphire substrate 3, <u>Fig. 2-4</u>, with two clips for fixing the sample is supplied with the cell.

The flange design includes a clamping device in the form of two levers 1, see Fig. 2-5. The levers are drawn apart to secure the diaphragm on the Teflon ring and then they are joint again, clamping the diaphragm firmly to the ring on the edge and sealing the cell.



Fig. 2-5. 1 – clamping levers

The cell is supplied assembled but it has to be dismantled for washing.

The outlets with tubes for feeding liquid allow to change the liquid in the cell without removing it from the approach unit of the instrument. The cell is sealed, so the liquid (gas) can be pumped through the cell by feeding it under pressure through one outlet while simultaneously pumping it out from the other one.

During the measurements the liquid contacts the Teflon ring that is chemically neutral to corrosive media, the polycrystalline sapphire substrate, the clips made of stainless steel, the probe, the glass pedestal and the probe clip, also made of stainless steel. Moreover, it should be noted that humidity inside the cell reaches 100% and the liquid vapour can react with the silicon rubber diaphragm.

and

ATTENTION! Use liquids (gases) chemically neutral to silicon rubber, glass, polycrystalline sapphire, stainless steel and the probe material.

3. Preparation for Measurements in Drop

The instrument is supposed to be assembled in the configuration required by the user and all relative connections are assumed to be made (see Solver PRO Instruction Manual. Chapter 2. "Initial set-up of the instrument"):

- 1. The interface card is installed.
- 2. The software is set up.
- 3. The connection of the instrument electromechanical parts is made.
- 4. The electromechanical configuration for "scanning by probe" is performed:
 - the measuring head is connected to the approach unit;
 - the sample holder is mounted in the positioning system of the approach unit;
 - the Closed-Loop Scanner Equivalent is connected (if available).
- 5. The scanner calibration parameters are loaded.
- 6. The Closed-Loop Scanner Equivalent (if available) or the scanner with sensors is prepared for operation.

The basic operations performed when preparing for the measurements in drop include:

- 1. Probe Installation (see i. <u>3.1</u> on page <u>14</u>).
- 2. Probe Holder Installation (see i. <u>3.2</u> on page <u>15</u>).
- 3. Sample Installation (see i. <u>3.3</u> on page <u>16</u>).
- 4. Adjustment of the length of Measuring Head Threaded Supports (see i. <u>3.4</u> on page <u>16</u>) (accomplished during the initial set-up of the measuring head).
- 5. Laser Beam Alignment onto the Cantilever (see i. <u>3.5</u> on page <u>18</u>).
- 6. Measuring Head Installation (see i. <u>3.6</u> on page <u>21</u>).

3.1. Probe Installation

To install the probe:

- 1. Place the probe holder on a smooth surface.
- 2. Using tweezers take a probe from the set. So not turn the chip over as the probes are placed with their tips pointing upwards.
- 3. To fix the probe use the special wrench. Clench the clip with the wrench staple, see <u>Fig. 3-1</u>. By slightly pressing the wrench handle unbend the clip and place the probe on the beveled side of the pedestal under the clip. The length of the chip part projecting above the polished surface of the pedestal shall be about 0.8 mm, i.e. the end of the chip with the cantilever shall be positioned above the center of the upper polished side of the pedestal.



Fig. 3-1. Probe installation. The arrow shows the direction of force application

4. Release the clip and remove the wrench.

3.2. Probe Holder Installation

1. Bring the holder to its seat on the measuring head. The holder shall be rotated 45° relative to its working position and the positioning marker (spot) on its ring shall coincide with **INSERT** marker on the lower part of the measuring head housing (Fig. 3-2).



Fig. 3-2

- 2. Secure the holder in its seat. The holder shall rest on the magnetic support evenly and without skew.
- 3. Turn the holder ring clockwise until the positioning marker on the ring coincides with **OPERATION** marker on the lower part of the measuring head housing, <u>Fig. 3-3</u>. This is the holder working position.



Fig. 3-3. Probe holder working position

3.3. Sample Installation

Fix the sample on a standard sapphire substrate 24x19x0.5 mm.

Mount the substrate with the sample on the sample stage, see <u>Fig. 3-4</u>. Push the substrate under the clips from the side with two balls.

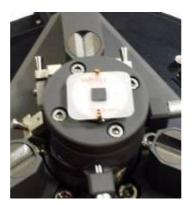


Fig. 3-4

3.4. Adjustment of the length of Measuring Head Threaded Supports



NOTE. This procedure is accomplished at the initial set-up of the measuring head or when changing the cell.

The length of the measuring head threaded supports shall be sufficient to provide at least 2-3 mm gap between the sample and the probe when fixing the measuring head on the universal stage. To reduce undesired tilting during scanning the measuring head base surface shall be parallel to that of the sample stage (providing the object to be observed is nearly parallel-sided.)

To adjust the threaded supports length proceed as follows:

- 1. Place the substrate with the sample on the sample stage under the clips.
- 2. Turning the manual approach screw counter clockwise move the stage with the sample to its lowermost position.
- 3. Check the distance between the sample and the probe holder:
 - a. Put the measuring head front supports fixed by the lock-nuts in their seats 1 (see Fig. 3-5) without letting down the rear support.





- b. Looking from a side, carefully lower the rear support of the measuring head. If the gap between the holder and the sample is less than 2 mm then remove the measuring head.
- c. Loosen the lock-nuts and unscrew the threaded supports on the required length.
- 4. Place the measuring head on the universal stage. The gap between the sample surface and the probe holder shall be at least 2-3 mm.
- 5. Verify that the measuring head base is parallel to the sample stage surface:
 - a. Measure the distance between the universal stage surface and the measuring head base near each of the tree supports.
 - b. Adjust the length of the supports so that these distances are equal. A ruler with onemillimeter-point scale would provide the adjustment accuracy of 0.5 mm which usually suffices.
 - c. Fix the lock-nuts on the front supports.

This finishes the adjustment of the measuring head supports length.

3.5. Laser Beam Alignment onto the Cantilever

- 1. Power on the computer and start the control program.
- 2. Turn on the Scanning Probe Microscope controller.
- 3. The laser switching-on and switching-off is controlled by **K** button (to the right on the main parameters bar.) The laser is automatically switched on upon starting the program.
- 4. Take the measuring head and lift it about 10-15 cm above a sheet of paper using the latter as a screen for observing the shape of the laser spot (Fig. 3-6).

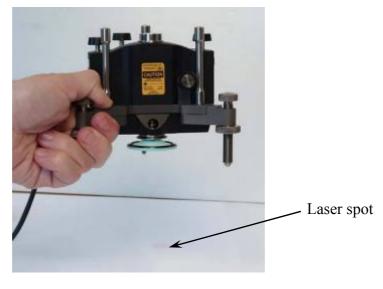
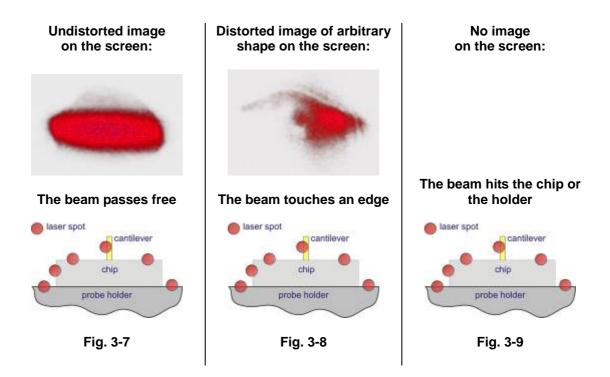


Fig. 3-6

Three are three possibilities here:

- a. Undistorted image of the laser spot is observed on the screen (Fig. 3-7). This means that the laser beam hits neither the cantilever nor the probe tip.
- b. The image of the laser spot is distorted. The beam partly hits against some part of the design or the cantilever (Fig. 3-8). It should be noted that quite a variety of images is possible (as, for instance, in Fig. 3-8).
- c. No image of the spot is seen on the screen. This means that the laser beam hits either the chip (Fig. 3-9) or the probe holder and cannot leave the measuring head.



Moreover, the design of the measuring head allows to visually examine the cantilever and the surrounding parts thus enabling the determination of the approximate location of the laser beam.

To align the laser beam onto the cantilever the following steps are required:

1. Rotating screw 2 (Fig. 3-10) achieve the undistorted shape of the laser beam. Generally, the laser spot would appear in position 1 (see Fig. 3-11).



Fig. 3-10 1, 2 – laser adjustment screws; 3, 4 – photodiode adjustment screws

2. Rotating screw 2 move the beam at the right angle to the front edge of the chip (1→2 in Fig. 3-11) until the laser spot becomes distorted again.

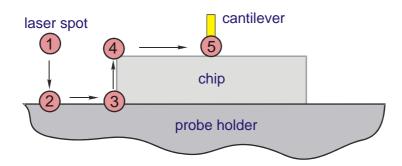


Fig. 3-11. Moving the laser spot in general case

- 3. Rotating screw 1 move the beam parallel to the front edge of the chip. The two alternatives are possible:
 - a. The laser spot moves along the edge of the holder $(2\rightarrow 3)$: in this case the spot disappears when it hits the chip;
 - b. The laser spot moves along the edge of the chip $(4\rightarrow 5)$: in this event, when the beam hits the cantilever an interference pattern is observed (Fig. 3-12). Now the laser beam is at the base of the cantilever. Move it towards the cantilever tip. The laser beam alignment onto the cantilever is complete.
- 4. When the spot disappears rotate screw 2 moving the laser beam towards the end face of the chip (3→4) until the laser spot reappears. Now the laser beam is at the edge of the chip (position 4).
- 5. Use screw 1 to move the beam along the front edge of the chip $(4\rightarrow 5)$ until an interference pattern is observed (Fig. 3-12). Now the laser beam is at the cantilever base.
- 6. Move the laser beam towards the cantilever tip.



Fig. 3-12. Image of the spot when the laser beam hits the cantilever

The laser beam aligned, put the measuring head on the desk with the threaded supports up.

3.6. Measuring Head Installation

- 1. Turning the manual approach screw counter clockwise, move the stage with the sample to its lowermost position.
- 2. Place the measuring head so that the threaded supports rest on the seats of the universal stage as shown in Fig. 3-13.



Fig. 3-13. The measuring head is installed in the approach unit

- 3. Adjust the sample location in XY plane. Use the micrometer screws of the positioning device to place the cantilever above the area to be observed.
- 4. Bring the sample to the cantilever at a distance of 0.5–1 mm by turning the manual approach screw clockwise.
- 5. Remove the measuring head from the approach unit.
- 6. Drip a few drops of liquid on the sample. The height of the drop shall be at least 0.5 mm above the sample surface. After that remount the measuring head.

The preparation for measurements in drop is complete. Proceed with Capter 5 "Adjusting the System for Detecting the Cantilever Deflection" on page 32.

4. Preparation for Operation with the Liquid Cell

The instrument is supposed to be assembled in the configuration required by the user and all relative connections are assumed to be made (see Solver PRO Instruction Manual):

- 1. The interface card is installed.
- 2. The software is set up.
- 3. The connection of the instrument electromechanical parts is made.
- 4. The electromechanical configuration for "scanning by probe" is performed:
 - the measuring head is connected to the approach unit;
 - the Closed-Loop Scanner Equivalent is connected (if available).
- 5. The scanner calibration parameters are loaded.
- 6. The Closed-Loop Scanner Equivalent (if available) or the scanner with sensors is prepared for operation.

The preparation of the instrument for working with the liquid cell generally includes the following stages:

- 1. Measuring Head Preparation (see i. 4.1 on page 23):
 - a. Diaphragm Installation (see i. <u>4.1.1</u> on page <u>23</u>);
 - b. Probe Installation (see i. <u>4.1.2</u> on page <u>24</u>);
 - c. Probe Holder Installation (see i. <u>4.1.3</u> on page <u>25</u>);
 - d. Laser Beam Aligning onto the Cantilever (see i. <u>4.1.4</u> on page <u>27</u>).
- 2. Preparation of Liquid Cell (see i. <u>4.2</u> on page <u>27</u>):
 - a. Sample Installation (see i. <u>4.2.1</u> on page <u>27</u>);
 - b. Initial Approach (see i. <u>4.2.2</u> on page <u>28</u>);
 - c. Assembling the Cell and its Installation on the Approach unit (see i. 4.2.3 on page 30).
- 3. Measuring Head Installation (see i. 4.3 on page 31).

4.1. Measuring Head Preparation

4.1.1. Diaphragm Installation

1. Remove the clip from the holder. To do this, take the clip with tweezers near the bend and pull it lengthwise along the base until the lower part of the clip releases from the guide slot in the base, Fig. 4-1.



Fig. 4-1. Clip removal

2. Take the diaphragm and turn it so that the side with the ring faces downwards to the holder. Put the diaphragm on the holder, with the glass pedestal passing through the hole in the diaphragm, see <u>Fig. 4-2</u>.



Fig. 4-2. Diaphragm installation



Fig. 4-3. Diaphragm fixed on the holder

- 3. Slightly stretching the diaphragm first from one side then from the other put it under the base flange, Fig. 4-3.
- 4. Wipe the holder with a napless tissue moistened in alcohol.

5. Mount the clip, <u>Fig. 4-4</u>. Recall that for operation in liquid the clip shall be inserted from the side with the smaller tilting angle relative to the horizontal plane, while the side with the larger tilting angle is designated for operation in gaseous media.



Fig. 4-4. Clip mounted



NOTE. In order for the diaphragm to last longer avoid putting in on and off too often.

4.1.2. Probe Installation

- 1. Place the probe holder on a smooth surface.
- 2. To fix the probe use the special wrench. Clench the clip with the wrench staple, Fig. 4-5.



Fig. 4-5

- 3. Using tweezers take a probe from the set. So not turn the chip over as the probes are placed with their tips pointing upwards.
- 4. By slightly pressing the wrench handle (see Fig. 4-6) unbend the clip and place the probe on the beveled side of the pedestal under the clip. The length of the chip part projecting above the polished surface of the pedestal shall be about 0.8 mm, i.e. the end of the chip with the cantilever shall be positioned above the center of the upper polished side of the pedestal.

ATTENTION! To avoid deformation of the clip unbend it only on the minimum angle required.

(ad)



Fig. 4-6. Probe installation. The arrow shows the direction of force application

5. Release the clip and remove the wrench.

4.1.3. Probe Holder Installation

In order to prevent the diaphragm from damage, a special fitting tool was designed for fixing the holder on the scanning measuring head for liquid operation.

Holder installation procedure:

1. Insert the holder in the fitting tool (Fig. 4-7) so that the clip goes into its slot.



Fig. 4-7

 Bring the holder to its seat on the head, holding the diaphragm with your fingers, see <u>Fig. 4-8</u>. The holder shall be rotated 45° relative to its working position, so that the positioning marker on its ring is located above **INSERT** arrow on the scale of the head.



Fig. 4-8

3. When the marker coincides with **INSERT** arrow, lower the holder into its seat, <u>Fig. 4-9</u>. The set ring on the lower part of the holder disk shall enter the scanner tip tube. The holder shall rest on the magnetic support evenly and without skew.







- 4. Turn the fitting tool with the holder 45 degrees clockwise. The right sides of the fitting tool shall be at the right angle to the front surface of the measuring head housing, see <u>Fig. 4-10</u>. This is the working position of the holder in which the measurements are performed.
- 5. Remove the fitting tool, <u>Fig. 4-11</u>.

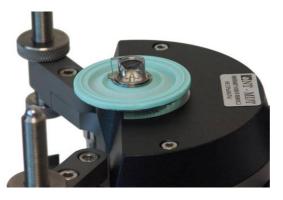


Fig. 4-11. The probe holder mounted on the measuring head

4.1.4. Laser Beam Aligning onto the Cantilever

The procedure for aligning the laser beam onto the cantilever is similar to that performed when preparing the measuring head for measurements in drop (see. i. 3.5 "Laser Beam Alignment onto the Cantilever" on page <u>18</u>).

4.2. Preparation of Liquid Cell

The preparation of liquid cell for operation includes the following stages:

- 1. Sample Installation (see i. <u>4.2.1</u> on page <u>27</u>).
- 2. Initial Approach (see i. <u>4.2.2</u> on page <u>28</u>).
- 3. Assembling the Cell and its Installation on the Approach unit (see i. <u>4.2.3</u> on page <u>30</u>).

When the optical viewing system is used stage 2 can be skipped. In this case the initial approach is accomplished during the adjustment of the the detection system.

4.2.1. Sample Installation

1. Fix the sample with the clips either directly to the substrate or using an adapter substrate, Fig. 4-12.



Fig. 4-12

2. Place the substrate with the sample on the cell base, Fig. 4-13.



Fig. 4-13

NOTE. When it is desirable to expose the sample to air for as short time as possible, an auxiliary plate of the same thickness can be mounted instead of the sample. After the probe vertical position has been adjusted, this auxiliary plate can be removed and the sample can be installed just before flooding the cell.

4.2.2. Initial Approach

1. Unbend the spring stops, <u>Fig. 4-14</u>, and put the cell holder into the positioning device, see <u>Fig. 4-15</u>.



Fig. 4-14



Fig. 4-15

2. Place the cell base on the holder, see Fig. 4-16.



Fig. 4-16

- 3. Turning the manual approach screw counter clockwise, move the cell base to its lowermost position.
- 4. During the initial installation of the measuring head and the cell and when changing the cell, the length of the measuring head supports shall be adjusted to provide the gap between the probe and the sample surface of at least 2-3 mm. The adjustment

procedure is described in par. 3.4 "Adjustment of the length of Measuring Head Threaded Supports" on page <u>16</u>. If this procedure was accomplished earlier and there is confidence that the supports length has not changed, the procedure can be skipped.

- 5. Fix the measuring head on the universal stage, see <u>Fig. 4-17</u>. If required, use the positioning device screws to align the head in the horizontal plane so that the diaphragm is in the center of the base (i.e. the base and the diaphragm are coaxial). Ensure that the sample and the probe are horizontal. The probe shall be located above the sample area to be observed. If necessary, remove the measuring head and change the sample position.
- 6. Looking from a side, bring the sample to the probe at a distance of 1-2 mm using the approach knob, see Fig. 4-18.
- 7. Remove the measuring head from the approach unit.
- 8. Remove the cell base from the approach unit.



Fig. 4-17



Fig. 4-18

4.2.3. Assembling the Cell and its Installation on the Approach unit

1. Mount the Teflon ring atop the cell substrate so that the pins in the base get into the corresponding holes in the ring, Fig. 4-19.



Fig. 4-19





- 2. Place the flange on the Teflon ring and put the fit ring on top of it. Screw up the fit ring to clamp the Teflon ring to the cell base, Fig. 4-20.
- 3. Fix the tubes with outlets for feeding liquid or gas, Fig. 4-21.



Fig. 4-21

4. Mount the liquid cell on the holder and draw apart the clamping levers, Fig. 4-22.

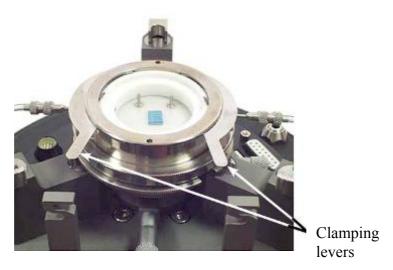


Fig. 4-22. Liquid cell on the approach unit

5. Flood the cell.

4.3. Measuring Head Installation

During the initial installation of the measuring head and the cell and when changing the cell the length of the measuring head supports shall be adjusted to provide the gap between the probe and the sample surface of at least 2-3 mm. The adjustment procedure is described in par. 3.4 "Adjustment of the length of Measuring Head Threaded Supports" on p. 16.

1. Mount the measuring head, with the threaded supports resting on their seats in the universal stage, Fig. 4-23.



NOTE. To avoid formation of air bubbles between the cantilever and the holder pedestal during immersion, it is recommended to drip some amount of liquid on the holder with the probe.



Fig. 4-23. Measuring head installation

2. Adjust the cell location in XY plane. Use the micrometer screws of the positioning device to place it exactly below the diaphragm.

5. Adjusting the System for Detecting the Cantilever Deflection

5.1. Adjusting with the Optical Viewing System

The adjustment of the detection system is best performed using the optical viewing system.

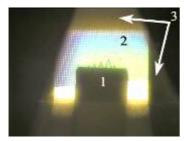
Z

NOTE: When using the optical viewing system the length of the measuring head supports shall be adjusted so that, when installed on the sample stage, the distance between the stage surface and the probe is about 4 mm.

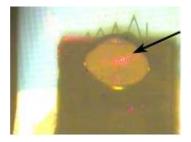
Proceed as follows:

- 1. Prepare the optical viewing system for operation as specified in "Optical viewing system" book.
- 2. Focus the videomicroscope on the cantilever.
- 3. Turning the approach knob and monitoring the sample position on the display (Fig. 5-1) bring the sample to the probe so that the edge of the glass pedestal with the probe dip in the liquid.

If bubbles appear (see Fig. 5-1 b), remove the measuring head and carefully wipe the liquid off the probe and its holder using a napless tissue. Remount the measuring head on the universal stage.



a) probe in the air; 1 – probe; 2 – sample; 3 – chamfered sides of the glass pedestal



b) the probe submerged in the liquid; the arrow shows an air bubble



c) the probe submerged in the liquid; air bubbles are absent

Fig. 5-1. The image on the display of the optical viewing system during approach

4. Bring the sample to the probe at the distance of 0.5-1 mm by carefully turning the approach knob until the sample surface comes to focus.

5. Align the laser beam onto the cantilever using screws 1 and 2 (Fig. 5-2).



Fig. 5-2. Scanning measuring head 1, 2 – laser adjustment screws; 2, 3 – photodiode adjustment screws

Further, precise alignment of the laser beam onto the cantilever tip shall be performed based upon the magnitude of the total photodiode signal. This procedure is implemented in the control program:

- 1. Open Aiming window (Aiming tab on the main operations panel).
- 2. Carefully turning XY screws 1 and 2 for tuning the adjustment unit, achieve the maximum value of the **Laser** signal (15-20 nA).



NOTE. If there is no signal on the photodiode indicator but the laser beam is aligned onto the cantilever, as could be seen on the optical viewing system display, then the photodiode might be in a position where the laser beam misses it. If this is the case, rotate the photodiode adjustment screws until the signal appears.

3. Rotate photodiode adjustment screws 3 and 4 to center the laser spot on the photodiode indicator. The values of **DFL** and **LF** signals shall be close to zero while the magnitude of the total Laser signal shall remain rather large.



NOTE. If the **Laser** signal decreases to zero when turning any of the screws then it means that the laser beam hits the photodiode edge. In this event the corresponding screw shall be turned in the opposite direction.

5.2. Adjusting without Using the Ooptical Viewing System

After lowering the cantilever in liquid adjust the detection system using the readings of the photodiode signal indicator.

Adjustment procedure:

- 1. Open **Aiming** window.
- 2. Turn screw 2 (see Fig. 5-2) $10-25^{\circ}$ counter-clockwise (this causes the laser spot to move towards the chip) until some Laser signal appears.
- 3. Then turn screw 1 **counter-clockwise** while observing the variation in the **Laser** signal. A criterion for hitting the cantilever is the presence of maximum in Laser signal when turning screw 1. In case of a triangular cantilever the laser beam hits one of its arms.
- 4. Turn photodiode adjustment screws 3, 4 to achieve the maximum increase in Laser signal.



NOTE. When adjusting the photodiode position it is important to make sure that turning the photodiode vertical displacement screw causes the vertical motion of the laser beam and similarly for the horizontal screw. If the laser beam moves arbitrarily and **Laser** signal decreases to zero it means that the laser beam hits the photodiode edge. In this event the corresponding screw shall be turned in the opposite direction.

- 5. Move the laser beam in the direction away from the chip by alternately turning the adjustment screws. Tune on the maximum value of **Laser** signal (15-20 nA).
- 6. Turn photodiode adjustment screws 3 and 4 (Fig. 5-2) center the laser on the photodiode indicator. The values of **DFL** and **LF** signals shall be close to zero while the magnitude of the total **Laser** signal shall remain fairly large.

For triangular cantilever it is recommended to check whether the laser beam hits the tip of the triangle or one of its arms. To do this:

- 1. Shift the laser beam closer to the chip without changing the laser horizontal position in order to reduce **Laser** signal approximately twice. The laser adjusted correctly, the laser beam now passes between the cantilever arms reflecting to some extent from both of them.
- 2. When turning the laser horizontal displacement screw left and right two maxima of **Laser** signal shall be observed corresponding to the two arms of the triangular cantilever.
- 3. To return the laser beam to the triangle tip position the laser beam between the arms to obtain minimum Laser signal and then move the laser beam from the chip to the triangular tip.

A criterion for the right alignment of the laser beam onto the triangular cantilever tip is that the **Laser** signal decreases upon turning the laser adjustment screws.

When using the procedure described above for multy-beam probes the laser beam is likely to be aligned onto an outer beam. For more precise laser beam positioning use the optical viewing system. Moreover, the laser beam can be aligned onto the required probe beam by moving the former along the front edge of the chip and noting the number of maxima in the **Laser** signal, each of which corresponds to an arm of the triangular cantilever or to a rectangular cantilever.

6. Installation of a Protective Hood

It is recommended to work with a protective hood in the following cases:

- a. if it is necessary to obtain a high resolution image in XY plane or along Z axis;
- b. when carrying out temperature measurements;
- c. for protection against temperature differences;
- d. for reduction of acoustic noise level.

For installation of a protective hood execute the following actions:

- 1. Install the protective hood on the approach unit.
- 2. Ground the hood connecting the ground wire of the approach unit with grounding jack of the hood. (Fig. 6-1).



Fig. 6-1. The protective hood is installed on the approach unit 1 – grounding jack

After the installation of the protective hood turn on the Vibration Isolation System, see the Vibration Isolation System Manual for more details.

7. Features of Conducting the Measurements in Liquid

7.1. Contact AFM

In contact AFM mode of operation all the procedures are similar to performing the measurements in air (see Solver PRO Instruction Manual, section "Contact Atomic Force Microscopy").

7.2. Semicontact AFM

Nitride cantilevers with in-liquid resonance frequency in the range 10÷20 kHz are recommended for use with liquid cells.

As opposed to the cantilever resonance curve in air which contains only one working peak, the curve in liquid features a multitude of peaks. Some of them are suitable for operation while the others are not. The working frequency shall be chosen correctly in order to ensure successful operation.

We suggest two procedures for choosing the working frequency: one is manual and the other is automated.

When using cantilevers of a new type for the first time the manual adjustment of the working frequency is preferable. This manual procedure (described below) allows finding the working frequencies for cantilevers of practically any type.

Subsequently, the automatic adjustment can be used (see the <u>Attachment</u> on page <u>48</u>) provided it has been verified that the resulting frequencies are the same.

The basic steps for the preparation of the instrument and performing the measurements of the piezodriver working frequency in the manual mode are:

- 1. Approach (see i. <u>7.2.1</u> on page <u>38</u>).
- 2. Setting the Piezodriver Working Frequency (see i. 7.2.2 on page 40).
- 3. Preparation for Scanning (see i. <u>7.2.3</u> on page <u>44</u>):
 - a. Selection of working point (Set Point);
 - b. Setting of the working level of the feedback gain (FB Gain).
- 4. Scanning (see i. $\underline{7.2.4}$ on page $\underline{46}$).
- 5. Saving the Data (see i. $\underline{7.2.5}$ on page $\underline{47}$).
- 6. Finishing the Operation (see i. 7.2.6 on page 47).

7.2.1. Approach

When applying semicontact microscopy techniques for operation in liquid the approach procedure for contact-mode measurements should be used.

Contact-mode approach procedure:

1. Select **Contact** in the menu for choosing the electronic configuration of the instrument on the main parameters bar (Fig. 7-1).

<u>F</u> ile Vie	w Settings	Tools	Help	Contact	-	E FB	FB Gain 1,000	DFL	-	-0,326	Set Point 2,00
1			A10	1 Proven		100	17 A	17			

Fig. 7-1

2. Switch to Approach window (Approach tab on the main operation panel) (Fig. 7-2).

🖫 Data 🛛 🛃 Aiming	Resonance	Approach Scan	
Stop Landing	Backward 📢 One S	tep	Way = 0,0 Jum 💌
Auto SetPoint	Forward IN One S	itep Fast Moving 0,35 mm	🚀 Settings

Fig. 7-2

- 3. Check that **Auto SetPoint** button responsible for the automatic adjustment of the **Set Point** parameter is pressed (<u>Fig. 7-2</u>).
- 4. Start the approach procedure by clicking **Landing** button.

Watch the **DFL** signal changes in the oscilloscope window during the approach procedure, while monitoring the progress of the scanner protraction indicator. Wait till the procedure is completed.

If the approach parameters are set correctly, the procedure is completed after some time, leading to the following (see Fig. 7-3):

- DFL signal increases to the value of Set Point parameter with the feedback maintaining Z-direction scanner in the position where DFL signal equals Set Point with the position corresponding approximately to one half of the scanner protraction range;
- the length of the indicator's color bar decreases and stops somewhere in the middle (pos. 1 in Fig. 7-3);
- the step motor stops;
- the increase of DFL signal to the value of Set Point parameter is represented on DFL (t) trace on the oscilloscope (Fig. 7-3).



- the message "...Approach Done." will appear in the journal (pos. 2 in Fig. 7-3).

Fig. 7-3. Completing of approach procedure 1 – indicator of scanner protraction; 2 – journal

A detailed description of the operations performed by the program during the approach is given in Solver PRO Instruction Manual section "Semicontact atomic-force microscopy".

7.2.2. Setting the Piezodriver Working Frequency

The procedure for setting the piezodriver working frequency is:

- 1. Turn off the feedback (The button on the main parameters bar).
- 2. Select **SemiContact** in the menu for choosing the electronic configuration of the instrument on the main parameters bar (Fig. 7-4).

Help	SemiContact 두	₽ E FE	B FB Gain 1,00)0 Mag
🗎 🗰 Re	Contact	bach	🗏 Scan	↓ ↓ Curves
	 SemiContact 			-
€ 🖉 🤊	Tunnel Current Custom	i	NT-MD1	File(*.mdt)

Fig. 7-4

- 3. Switch to Resonance window (Resonance tab on the main operation panel) (Fig. 7-5).
- 4. In the synchronous detector settings panel select the frequency range **Pass Band** <55kHz.

ြီးစာ Data	E Aiming	Resonance 🛛 🐺 4	Approach 🛛 🗮 Scan 🛛 🕂	Curves 🛃 Litho
▶ Run		KHz Options kHz Auto peak find kHz Auto peak find	Generator Frequency 150,000 kHz Amplitude 0,20 ∨× 1 ▼	Lock-In Low Pass Gain Harm 3 kHz v 10,00 1 \$
Generator outp Response sigr		Average 5 Point N 1000	Phase 0,00 ⁰	Input : DFL ▼ × 1 ▼ Pass Band <55kHz ▼

Fig. 7-5

- 5. Set the range for measuring the frequency response of the cantilever oscillation amplitude (input boxes **From** and **To**) so that it contains the cantilever resonance frequency. It should be noted that the cantilever resonance frequency in liquid is 5-6 smaller than that in air. The recommended values are **From**=5 kHz, **To**=50 kHz.
- 6. In **Options** panel (<u>Fig. 7-6</u>) uncheck the option for automatic setting the resonance frequency (**Auto peak find**).



Fig. 7-6

- 7. Set the maximum amplitude of the generator output signal (Amplitude parameter):
 - a. On **Generator** panel select the gain for **Amplitude** parameter equal to 10 (Fig. 7-7).

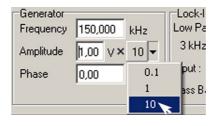


Fig. 7-7

b. Double-click in **Amplitude** input box and, using the drop-down box that appears, (Fig. 7-8) increase the value of **Amplitude** to its maximum.

Run	From	5,000	kHz	Contraction of Contraction	tions Auto pe	ak find	0250	nerator		000	Hz	Lock		ain H	larm					
nun	To	50,000	kHz	1	1		Am	plitude	1,00	v×	1 🕶	3 kH	lz 🔻 10	,00 1	\$					
																				1,00
	1	1	0.15	1	0.25	0.3	0.35	0.4	0.45	0.5	0.55	0.6	0,65	0.7	1	0.8	0.85	0.9	0,95	



8. Click **Run** button (on the left on the control bar) to obtain the frequency response of the cantilever oscillations amplitude (**Mag** signal). As can be seen in <u>Fig. 7-9</u> the cantilever resonance curve in liquid contains numerous peaks.

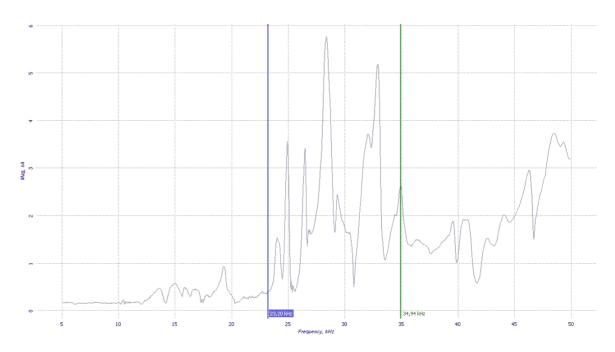


Fig. 7-9

- 9. Select a range with the highest resonance peaks and input a new range for plotting the frequency response of **Mag** signal. This can be done by two ways:
 - by changing the values of **From** and **To** on the control bar;
 - using the markers that appear on the plot (see Fig. 7-9).
- 10. Click **Run** button to plot the resonance curve for the selected range (Fig. 7-10).

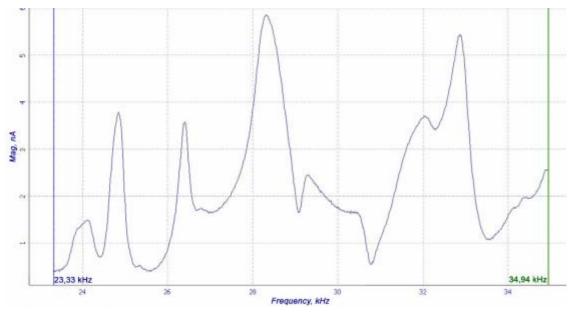


Fig. 7-10. Cantilever resonance curve in the free state

11. In the main parameters bar set **DFL** as a feedback input signal (Fig. 7-11).

ATTENTION! Before switching the signal applied to the feedback input make sure that the feedback is off (

² FB	FB Gain 1,00	DFL 🔻	0,354	
oroach 🧧	📕 Scan	,	Mag RMS	
Generator Frequency Amplitude	150,000 k	сн 1_	 DFL Phase Mag*Sin(t) 	iain H. 0,00 1

Fig. 7-11

- 12. Enter a value of **Set Point** parameter that exceeds the current **DFL** value by 1.5-2 nA.
- 13. Click to turn the feedback on. The piezotube will advance by half of its length, as can be seen on the piezotube extension indicator, and the cantilever will approach the sample surface.

and)

14. Click **Run** button to repeat the resonance curve plotting procedure for the same range but with the feedback on. (Fig. 7-12).

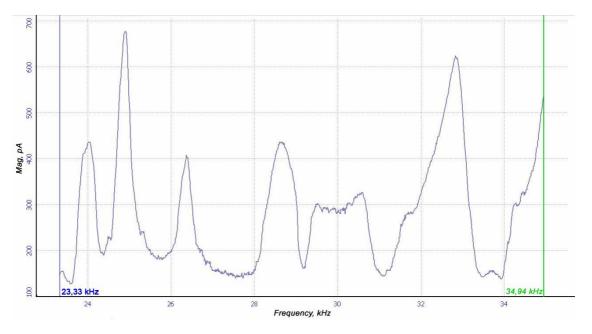


Fig. 7-12. Resonance curve for the cantilever propped to the sample surface

Compare the resonance curve corresponding to free cantilever with that for the propped cantilever. Some of the resonance peaks decreased significantly, others remained practically the same but some increased.

Any frequency at which the cantilever oscillation amplitude increases when the cantilever is propped can be chosen for the measurements. Select a peak which best meets the following two requirements:

- the cantilever oscillation amplitude is the largest when the feedback is off;
- with the feedback turned on (when the cantilever is propped against the sample surface), the drop in the cantilever oscillation amplitude is most pronounced.
- 15. Turn off the feedback (🛱 🕫 button).
- 16. Replot the cantilever resonance peak in the free state (Run button).
- 17. On the plot obtained find the selected peak. Set the piezodriver resonance frequency (**Frequency** parameter) equal to that of the peak:
 - a. In **Frequency** parameter input box on **Generator** panel enter the value corresponding to the maximum of the selected peak. At this a marker will appear on the plot (see Fig. 7-13);
 - b. Moving the marker (with the mouse left button pressed) set the frequency value related to the maximum **Mag** signal for the selected peak. **Frequency** input box will display a value at the current marker position.

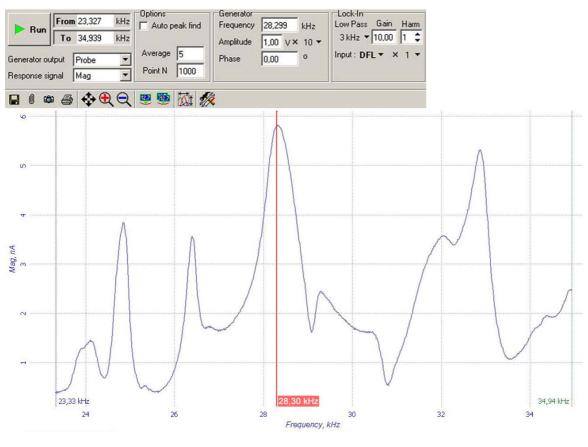


Fig. 7-13



NOTE. The resonance peak may have a flat top. In this case decrease the value of the **Amplitude** parameter.

Setting the piezodriver working frequency is complete.

7.2.3. Preparation for Scanning

Selection of working point (Set Point)

1. On the main parameters bar select **Mag** as a feedback input signal (before switching make sure that the feedback is off).





- 2. Input the value of Set Point equal to approximately half of the Mag signal value.
- 3. Reduce the **FB Gain** value to 0.3-0.5 in order to avoid excitation after turning on the feedback.
- 4. Switch to Approach window (Approach tab on the main operations panel).

5. Turn on the feedback (the feedback is on, **Mag** signal will fall to the level of **Set Point** (Fig. 7-15).

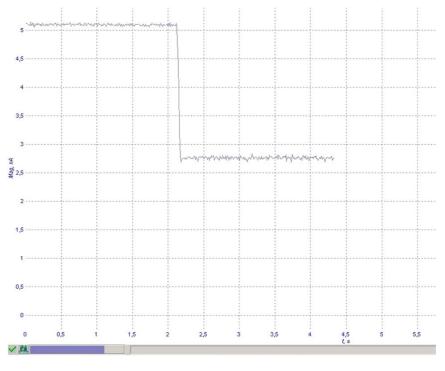


Fig. 7-15

6. Verify that the probe was brought to contact with the sample surface by changing the value of **Set Point** in the range $\pm 20\%$ using the scroll bar and monitoring the piezotube extension on the corresponding indicator. Minor variations in **Set Point** shall not result in any piezotube displacement.

Otherwise, repeat the approach procedure for a smaller value of Set Point:

- 1) Reduce **Set Point** by a factor of 0.5-0.7;
- 2) Deactivate Auto SetPoint;
- 3) Start the approach procedure by clicking Landing button.

Once the approach is finished recheck the piezotube behavior with variations in **Set Point**.

Setting the working level of the feedback gain

- 1. Double click with the mouse left button in **FB Gain** input box located on the main operations bar. Using the scroll bar that appears increase **FB Gain** while monitoring the **Mag** signal level on the software oscilloscope (in **Approach** window).
- 2. Determine **FB Gain** value at which excitation starts.

The beginning of excitation is detected by a sudden rise in the Mag signal a.c. component (Fig. 7-16).

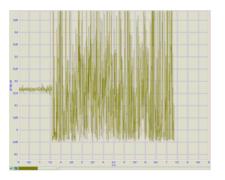


Fig. 7-16. Generation

3. Set **FB** Gain equal to 0.6-0.7 of the value at which excitation starts.

If the excitation is still present after decreasing **FB** Gain to 0.1 - 0.2 try setting the piezodriver frequency on another peak. Also, the excitation may result from poor fixing of the sample or the cell.

7.2.4. Scanning

Setting up the scanner parameters (selection of SPM mode, scan region and scanning speed) and the measuring procedures are similar to those in air. See Solver PRO Instruction Manual for details.

Given below is the sequence of the basic procedures performed when starting the scan process:

- 1. Switching to **Scan** window.
- 2. Selection of SPM mode.
- 3. Selection of scan region and the scanning speed.
- 4. Starting scanning (**Run** button).

7.2.5. Saving the Data

Once the sample surface is scanned, the resulting surface image is stored in RAM.

To save the obtained data to a hard disk perform the following:

- 1. Choose **File** \rightarrow **Save** in the Main menu panel;
- 2. Enter the filename in the dialog window and click **Save** button. The data will be saved in the file with ***.mdt** extension. A separate frame corresponds to each scanned image of a surface. Indexing of frames corresponds to the order of their acquisition.

7.2.6. Finishing the Operation

Closing the control program

- 1. Switch to Approach window.
- 2. Retract the sample from the probe at approximately 2-3 mm (**Fast** button for **Backward** on the control bar).

Aiming	Resonance	Approach	📕 Scan	Curves
▶ Landing	Backward	Step	oving 0,35 mm	Way = 0,0

Fig. 7-17

- 3. Turn off the feedback (TFB button).
- 4. Turn off the SPM controller.
- 5. Turn off the Vibration Isolation system.
- 6. Close the control program.

Operations to be performed after finishing the work with the cell

After using, disassemble the cell and rinse it in a solution of a surfactant (in proportion of 1 ml surfactant to 100 ml of distilled water). Use a syringe to wash the outlets with the tubes. Then, rinse the components of the cell, the outlets and the tubes in distilled water. Blow out the cell with warm air. It is also recommended to dry the dismantled cell in a thermostat at a temperature below 80°C.



ATTENTION! Before drying the cell in the thermostat detach the tubes from the outlets. Do not dry the tubes in the thermostat. It is recommended to wear rubber gloves during the whole washing procedure.

After drying reassemble the cell, close it with the cover and bring together the levers on the flange.

Attachment

1. Semicontact AFM. Setting the Working Frequency in Automatic Mode

Nitride cantilevers with in-liquid resonance frequency in the range $10\div 20$ kHz are recommended for use with the open liquid cell.

Before using the procedure for setting the piezodriver working frequency in the automatic mode it is recommended to compare the resonance frequency values determined in both modes for a chosen cantilever type. If the results agree then the automatic procedure should be used for the cantilevers of the given type.

Main stages of preparation to measurements when the piezodriver working frequency is set in automated mode include:

- 1. Setting the Piezodriver Working Frequency (see i. <u>1.1</u> on page <u>49</u>).
- 2. Approach (see i. <u>1.2</u> on page <u>51</u>).
- 3. Preparation for Scanning (see i. <u>1.3</u> on page <u>53</u>):
 - a. correction of the working frequency;
 - b. selection of Set Point;
 - c. setting **FB Gain**.

Scan parameters settings (selection of SPM technique, scan area and scan speed) and scanning itself are similar to that in air. See Solver PRO Instruction Manual for details

1.1. Setting the Piezodriver Working Frequency

- 1. Prior to determining the resonance frequency in the automatic mode the probe shall be manually brought to the sample surface at a distance of 1-2 mm. The optical viewing system can be used for this purpose. With the videomicroscope focused on the cantilever, proceed with manually approaching the sample to the probe until the sample surface comes to focus.
- 2. Switch the instrument in the semicontact AFM measuring mode by selecting **SemiContact** (Fig. 1-1) in the menu for choosing the electronic configuration of the instrument on the main parameters bar.

Help	SemiContact	F	₽ E FB	FB G	iain 1,0	00 Mag
Re Re	Contact		bach	æ	Scan	ĴV _{Curves}
	 SemiContact 					-
ջ ⊠ ե	Tunnel Current Custom	K	i		NT-MD	T File(*.mdt)

Fig. 1-1

3. In the main menu select subsequently Tools → Script → Scripts → Res_Liq_Find (Fig. 1-2).

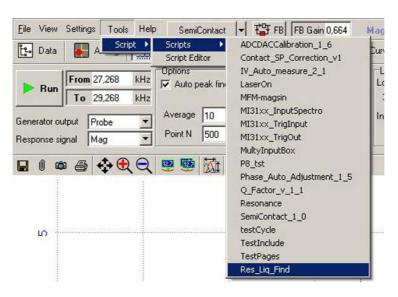
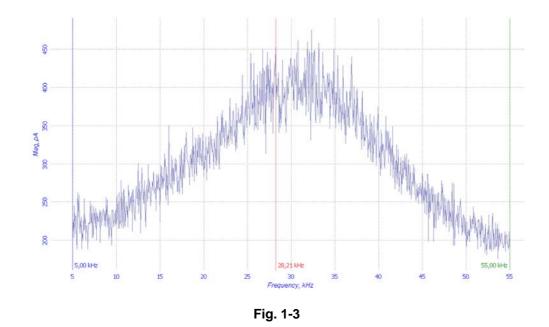


Fig. 1-2

After selecting **Res_Liq_Find** the working frequency will be automatically adjusted.

As the result of this procedure:



- cantilever thermal noise will be measured (Fig. 1-3).

- the frequency response of the cantilever oscillation amplitude (Mag signal) will be
- obtained in the range corresponding to the maximum of thermal noise;
 the frequency response of Mag signal will be plotted near the maximum (Fig. 1-4);
- the piezodriver working frequency will be adjusted to a peak optimal for the operation in the semicontact mode.

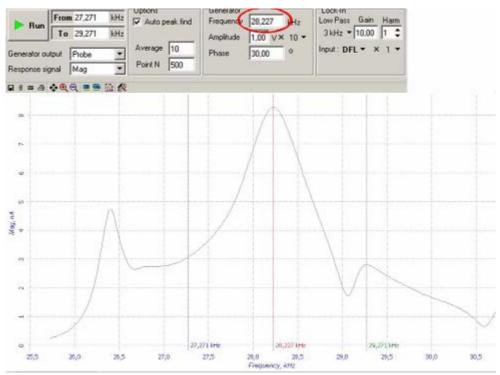


Fig. 1-4

1.2. Approach

1. Switch to Approach window (Approach tab on the main operation panel) (Fig. 1-5).

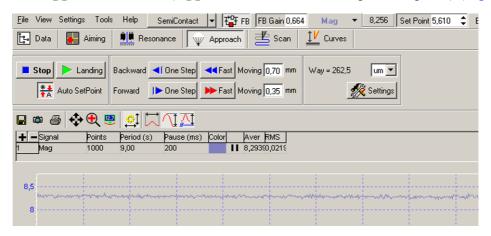


Fig. 1-5

- 2. Check that the option for the automatic setting of **Set Point** parameter is activated. **Auto SetPoint** button shall be pressed as shown in Fig. 1-5.
- 3. Click Landing button to start the approach procedure.

During approach, monitor the changes of **Mag** signal in the oscilloscope's screen as well as the state of the scanner extension indicator and wait for the conclusion of the approaching process.

After a while, if the tuning of the approaching parameters was correct, the approaching process will be completed and the system will perform the following:

- the Mag signal will be reduce down to the value of Set Point parameter, the feedback will keep the Z-scanner in the position where Mag signal is equal to Set Point, moreover that position of the scanner will be approximately one half of the scanner extension range;
- the length of the indicator colored strip will be reduced and would be in some intermediate position;
- the step motor will stop;
- the Mag(t) signal trace in the oscilloscope will display a step-down of the Mag signal to the value of Set Point parameter (Fig. 1-7, Fig. 1-8);
- The message "... Approach Done." will appear in the journal (Fig. 1-6).

Stop Landing Backward I One St *** Auto SetPoint Forward I> One St	P Fast Moving 0,35 mm		17:34.20 Remote complete. 17:34.22 Remote complete. 17:34.59 Remote complete. 17:35.01 Remote complete. 17:35.28 StopZ is reached. Approach Done.					

Fig. 1-6

A detailed description of the operations performed by the program during the approach is given in Solver PRO Instruction Manual section "Semicontact atomic-force microscopy".

A criterion for finishing the approach, i.e. for the contact between the probe and the sample surface, is the sudden drop of the **Mag** signal value (a nearly vertical line in Fig. 1-7).

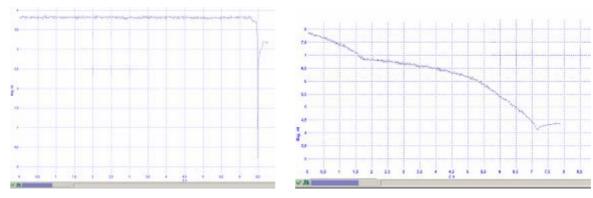


Fig. 1-7. Mag(t) function in case of a successful approach of the probe to the sample surface

Fig. 1-8. Mag(t) function for the case when the approach procedure was incomplete

The absence of a sharp dip on the plot, as in the example shown in $\underline{Fig. 1-8}$, is the evidence that the approach was incomplete. In this event the value of **Set Point** shall be decreased:

- 1. Switch to Resonance window.
- 2. Turn off the feedback (The button on the main parameters bar).
- 3. Click **Run** button to plot the resonance curve.
- 4. Using the marker fine-tune the piezodriver working frequency.
- 5. Switch to Approach window.
- 6. Uncheck Auto SetPoint.
- 7. Decrease **Set Point** by a factor of 0.5-0.7 (at this the scanner shall advance on its full length).
- 8. Start the approach procedure by clicking Landing button.

Once the approach procedure in liquid is finished, it is recommended to verify that the probe has actually approached the sample:

- 1. Double-click in **Set Point** signal input box.
- 2. Using the scroll bar that appears change the value of **Set Point** in the range $\pm 0.5-1$ nA, while observing the piezotube extension by the indicator.

The indicator shall not change its position. Otherwise, the approach shall be performed with a smaller value of **Set Point**.

1.3. Preparation for Scanning

Correction of the piezodriver working frequency

Near the surface the shape of the resonance peak can change hence after finishing the approach the piezodriver working frequency shall be corrected:

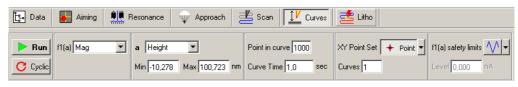
- 1. Switch to **Resonance** (**Resonance** tab on the main operations panel).
- 2. Turn off the feedback (FB button on the main parameters panel).
- 3. Click **Run** button to plot the resonance curve.
- 4. Using the marker correct the piezodriver working frequency.

Selecting the working point (Set Point)

The working point for scanning shall be chosen 0.8-0.9 of the cantilever free-oscillations amplitude measured after the approach.

The amplitude curve shall be measured (Mag(z) function) in order to determine the maximum amplitude of Mag signal:

- 1. Switch to Curves window (Curves tab on the main operations panel).
- 2. In the control panel of **Curves** window (<u>Fig. 1-9</u>) select **Mag** for the signal to be measured (**f1(a)** parameter) and **Height** signal as its argument (**a** parameter).





- 3. Set the height variation range (Min and Max parameters) from -10 nm to +100 nm.
- 4. Using the cursor select a point in the scanning region where **Mag** (z) function is to be measured.
- 5. Click **Run** button. Once the measurements are finished an additional window appears displaying the measured response.

6. Carefully adjust the height variation range to obtain a curve similar to that in Fig. 1-10.

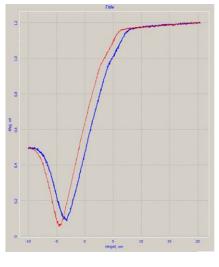


Fig. 1-10

7. Assign **SetPoint** a value 0.8 - 0.9 of the maximum **Mag** signal amplitude.

A typical value for the maximum Mag signal amplitude is within the range of 5-10 nm.

Setting the working level of FB Gain

Set the feedback gain as described in i. 7.2.3 on page 46.

If the excitation is still present after decreasing **FB** Gain to $0.1\div0.2$ try setting the piezodriver frequency on another peak. Also, the excitation may result from poor fixing of the sample or the cell.

Setting up the scanning parameters and scanning are similar to those described in i. $\underline{7.2}$ "Semicontact AFM" on page $\underline{37}$.