

AFM Contact Configuration

This document describes the principles of AFM Contact Measurements followed by the WITec Control software configuration for AFM Contact Measurements. The beam path and the step by step alignment procedure can be found in Sections 3 and 4.

1 Principles of Atomic Force Microscopy (AFM)

Since the invention of atomic force microscopy (AFM) in 1986 by Binning, Quate and Gerber [G.Binning, C.F.Quate and Ch.Gerber; Phys.Rev.Lett. 56, 930 (1986)], AFM has rapidly developed into a powerful and invaluable surface analysis technique on micro- and nanoscales and even on atomic and molecular scales. Using AFM, it is possible to image surfaces in real-space with a resolution down to the level of molecular structures. In addition to the imaging of small topographic features on surfaces, AFM is also used to image additional surface properties such as adhesion, stiffness, magnetic properties, conductivity and many more.

The operating principle of an AFM is rather simple. A probe is scanned over a sample and various interactions between tip and sample are used as feedback mechanisms to trace the surface topography (Fig. 1).

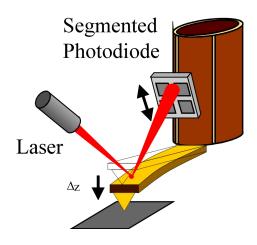


Fig. 1: Principle of AFM operation.



At the free end of a cantilever (typically 100 to $200\mu m$ long) a sharp tip (less than 10 nm across) is mounted. This tip is brought into contact with the sample while the repulsive force between tip and sample bends the cantilever. The bending of the cantilever is measured using a highly focused beam deflection system as shown in Fig. 1. By keeping the bending of the cantilever constant, a constant force is applied to the sample while scanning the tip across the surface. The alpha300/alpha500/alpha700 microscopes are sample scanning systems, where the cantilever remains at a constant position and the sample is scanned precisely underneath it. A detailed description of the scanner can be found in the al-pha300/alpha500/alpha700 system description. This setup has the advantage of fixed optical beam-paths, which eliminates the requirement of tracting the beam deflection system. The vertical movement of the scanner follows the surface profile and is recorded as the surface topography.

Several forces typically contribute to the bending of the AFM cantilever. The force most commonly associated with scanning force microscopy is the interatomic van der Waals force. The dependence of the van der Waals force upon the distance between tip and sample is described by a Lennard Jones potential V, which can be written as

$$V_{sample}(z) = Az^{-12} - Bz^{-6}. (1)$$

Here z denotes the tip sample distance and A and B are interaction parameters. As the tip is approaching the surface, attractive forces act between the tip and sample before repulsive forces start to dominate. In an AFM, the tip is attached to a flexible cantilever which is subject to Hook's law

$$V_{cantilever}(z) = k \frac{(z - z_0)^2}{2},$$
(2)

where k is the spring constant of the cantilever and z_0 is the tip-sample distance for an unbent cantilever. An example of a resulting force distance curve of this coupled system is shown in Fig. 2.



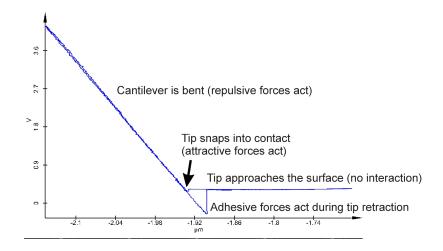


Fig. 2: Force distance curve in an AFM measurement.

These curves contain all tip-sample interactions, enabling the mapping of material properties on the nanometer scale.

Over the past decade, a large number of additional AFM imaging modes were developed for a variety of applications. AFM can be used on any kind of sample. Therefore the number of publications in materials science, life science, and related industries has increased tremendously since its invention.

1.1 AFM Contact Mode

The operating principle of an AFM in contact mode is described above. The feedback parameter in this mode is the bending of the cantilever, which ensures a constant force between tip and sample. The up and down movement of the scan stage is recorded as surface topography. Any deviation from the constant bending of the cantilever, measured on the photo-detector, is represented in the deflection image. These images highlight the edges of various topographic levels.

Different mechanical properties of the surface can be evaluated from the friction images. In this case the torsion of the cantilever is recorded. The operating principle of friction mode is shown in Fig. 3.



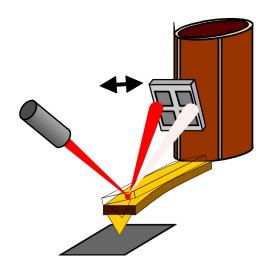


Fig. 3: Principle of AFM friction measurements.

1.2 Cantilever choice

In this imaging mode, the tip is always in contact with the surface and the applied forces between tip and sample strongly depend on the spring constant of the cantilever. Not only the controlled vertical force, but lateral forces also act between tip and sample as the tip traces the surface topography. These lateral forces might lead to the dragging of particles weakly bound to the substrate, thus resulting in blurred images. Therefore, for AFM contact mode measurements, cantilevers with spring constants below 1N/m are recommended.

1.3 Cantilever approach

Before any approach in contact mode, the top minus bottom (T-B) and the left minus right (L-R) signal as recorded by the quadrant photo diode need to be adjusted to approximately zero, indicating the zero bending position of the cantilever. The setpoint is selected at a higher value to set the bending of the cantilever, which is proportional to the interaction force between tip and sample while scanning.

The force distance curve shown in Fig. 4 displays the initial T-B value and the range within which the setpoint should be selected for contact mode measurements. The feedback loop maintains a constant bending of the cantilever at the selected setpoint.



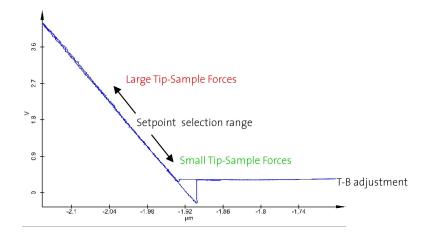


Fig. 4: Typical force-distance curve showing the setpoint selection for AFM contact mode measurements.

2 WITec Control AFM Contact configuration description

This description is intended to be used in combination with the WITec Control manual. The WITec Control manual contains the description of the full functionality of WITec Control, whereas this section indicates only which functionalities are included in this configuration. Please refer to the WITec Control manual for further details. The configuration-specific speed buttons, as well as the layout of the Control Window, will also be illustrated below.

The AFM Contact configuration is used to perform AFM contact mode measurements. During an AFM contact mode measurement, data can be acquired using the following sequencers:

- Image Scan: acquisition of AFM contact mode images,
- Line Scan: acquisition of force-distance curves along a line,
- Distance Curve: acquisition of force-distance curves at the current position.

The characteristics of the AFM contact configuration are described in the following section. The typical layout of the AFM contact configuration is shown in Fig. 5.

2.1 Speed Buttons

The main menu contains, in addition to the standard WITec Project speed buttons, several speed buttons which provide quick access to microscope and controller



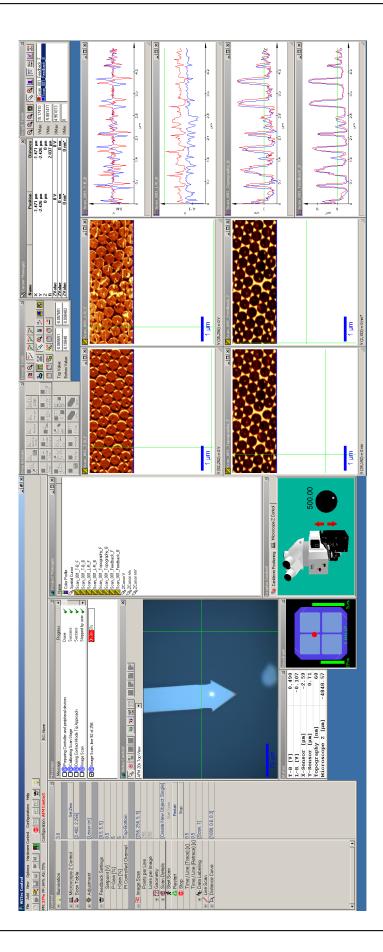


Fig. 5: Typical layout of the AFM contact configuration.



functions used in this configuration. A short description of these speed buttons is given below.

Stop 🌚

This speed button is used to stop any sequencer.

Start Scan 🞏

With this button, the *Image Scan* sequencer is started.

Tip Approach 💹

This button is used to start the *Tip Approach* sequencer.

Retract Tip 👱

A mouse-click on this speed button causes the tip to retract from the sample by the distance defined in the *Microscope Z Control* device under the parameter *Retract Distance* $[\mu m]$.

P-I Control

Clicking with the mouse on this speed button will open the *Feedback Settings* in the Control Window.

Illumination 🙀

With this speed button, the white light illumination can be switched on and off. The brightness of the illumination is defined in the Control Window using the illumination device.

2.2 Control Window

The Control Window described in Chapter 3 of the WITec Control manual is customized for AFM contact mode measurements. The reduced tree structure of the Control Window in the AFM contact configuration is shown in Fig. 6. Devices and sequencers are grouped in this configuration based on functionality. Each device and sequencer contains only the list of parameters which are used during an AFM contact mode measurement. The following devices and sequencers are accessible in the AFM contact configuration.

2.2.1 Setup and Control

In this section, the software controls for the hardware components (called devices in WITec Control) and the software controls for data acquisition processes (called sequencers) are listed and briefly described. These controls are used for microscope setup and measurement control.



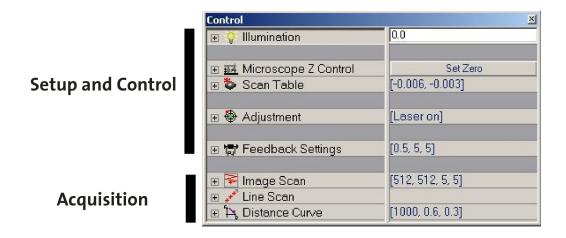


Fig. 6: Control Window for the AFM contact configuration.

Illumination 🐺

This device is used to control the white light illumination of the tip/sample video image. A detailed description of the parameters used to control the white light illumination is given in the WITec Control manual Section 3.4.2. The illumination speed button located in the main menu uses the brightness parameter set here.

Microscope Z Control

The microscope Z control is part of the scan table device (WITec Control manual Section 3.4.3). In this configuration, the Move Mode is automatically set to Z for Feedback, allowing the control of the Z-axis of the scan table through the PI controller. Therefore, Microscope Z control refers only to the movement of the microscope Z-stage.

HINT The microscope Z-stage can be controlled via the remote control (see WITec Control manual Section 3.4.1), the Graphic Control Window (see WITec Control manual Chapter 8) or using the parameters Speed, Move Up 🚹, or *Move Down* Usited in the Control Window.

In addition, the microscope Z-stage is used for the safe tip approach (WITec Control manual Section 3.5.8). All parameters of the tip approach sequencer are preset to appropriate values to guarantee a safe tip approach in this configuration.

HINT The speed button from the main menu (**★**) can be used for safe tip approach.

The Retract Distance $[\mu m]$ parameter defines the distance of the upward



movement of the microscope Z-stage if the *Retract Tip* button (<u>t</u>) is activated.

Scan Table 💺

In this configuration, the manual movement of the scan table is reduced to a planar movement (X-Y) to allow precise positioning within the scan-range. For a detailed description of the scan table, please see also the WITec Control manual Section 3.4.3.

Adjustment 🐠

All devices and sequencers required for the cantilever alignment are summarized in this section. These include controls of the beam deflection laser (see the WITec Control manual Section 3.4.5) and the cantilever positioning (see the WITec Control manual Section 3.4.7). Additionally, the control elements for the computer guided alignment of the system can be found here (see WITec Control manual Section 3.5.10 for a detailed description of these elements).

Feedback Settings 🖃

The feedback settings provide access to the parameters used in conjunction with the PI control of the scan table Z axis. A detailed description of the parameters is given in the WITec Control manual Section 3.4.5.

2.2.2 Acquisition

The sequencers used to acquire data in this configuration are listed and briefly described below.

Image Scan 📴

The parameters used to perform an image scan are described in WITec Control manual Section 3.5.2. In this configuration, all parameters required to perform an image scan in AFM contact mode are listed.

Line Scan 📝

The parameters used to perform a line scan are described in WITec Control manual Section 3.5.3. In standard AFM contact mode, force distance curves can be recorded along a line. The parameters used to record each force distance curve are the actual parameters defined in the distance curve sequencers (see below).

Distance Curve 🔼

The parameters used to acquire a distance curve are described in WITec Control manual Section 3.5.7. In this configuration, only those parameters required to acquire a force distance curve are listed.



2.3 Data Sources and Status Window

The data sources (WITec Control manual Section 3.6) used during an AFM contact mode measurement are summarized in Tab. 1 together with the default data labeling of the acquired channels. The data sources used for adjustment and control of the measurement are displayed in the Status Window (WITec Control manual Chapter 5) or the Quadrant Window (WITec Control manual Chapter 7).

Channel	Image	Distance	Channel	Unit	Display
	Scan	Curve	Caption		
T-B	✓	✓	T-B	[V]	Status Window
L-R	✓		L-R	[V]	Status Window
Sum					Quadrant Window
X-Sensor				$[\mu m]$	Status Window
Y-Sensor				$[\mu m]$	Status Window
Z-Sensor	✓	✓	Topography	[nm]	Status Window
					Quadrant Window
Feedback	✓		Feedback	$[nm^*]$	
Microscope Z				$[\mu m]$	Status Window

Table 1: Output channels and their sequencers.

2.4 Quadrant Window

The Quadrant Window is used for the alignment and display of the beam deflection system. A detailed description of this window is given in the WITec Control manual Chapter 7.

In AFM Contact Mode, the Quadrant Window shows the position of the beam deflection laser on the four quadrant photo diode as the red spot on the display (see Fig. 7).



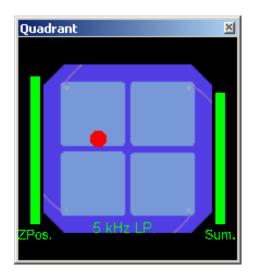


Fig. 7: The Quadrant Window in AFM Contact Mode



3 Beam Path

In this section, the beam path is illustrated schematically. The parts common to all instruments are displayed in dark grey. Parts not common to all instruments, but necessary for the described measurement mode are indicated in blue. Optional parts which may be present with the instrument, but are neither common to all instruments nor are necessary for the described measurement mode are displayed in light grey. The parts indicated are listed below the figure where an *alpha300* system is shown exemplarily. Detailed descriptions of the parts indicated can be found in the *alpha300/alpha500/alpha700* system description. The beam path is represented in red in Fig. 8.

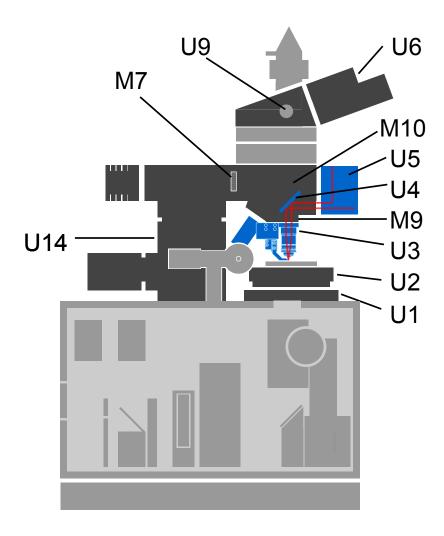


Fig. 8: Schematic illustration of the beam path for AFM.



U1 XY positioner

U2 Scan stage

U3 Objective turret with objectives including the inertial drive and the AFM tip

U4 Dichroic mirror

U5 Beam deflection unit

U6 Binocular tube with ocular camera

U9 Pushrod

U14 Microscope Z stage with stepper motor

M7 Field stop diaphragm

M9 Objective turret

M10 Reflector slider

4 Step by step alignment

The following listing describes the alignment of the *alpha300/alpha500/alpha700* system in order to obtain an AFM contact mode image, to perform a line scan or to capture a force distance curve. The first steps describe the focusing of the microscope on the sample using white light illumination followed by the cantilever alignment procedure. The automatic tip approach is described thereafter.

Parts of the alignment procedure are computer guided through the adjustment procedure. The procedure can be started using the corresponding Start Adjustment button located in the adjustment menu. The computer guided steps of the alignment procedure below are marked with a yellow bar on the left.

- 1. Switch on the alphaControl. This is usually done using the switch on the multi-plug.
- 2. Power up the computer and start WITec Control . Select the AFM contact mode from the Configurations-menu.
- 3. Mount the sample (e.g. the Aluminum Latex projection pattern included with the instrument) on the scan stage.
- 4. Remove the magnetically fixed cantilever arm from the inertial drive of the SPM objective (see the *alpha300/alpha500/alpha700* system description for details).



- 5. Rotate the microscope turret until the SPM objective is in the working position.
- 6. Push in the pushrod (U9) of the sliding prism and direct the beam to the eyepiece color video camera (U6).
- 7. Move the reflector slider (M10) to the illumination position (M11; see the alpha300/alpha500/alpha700 system description for details). The beam splitter is mounted in the left position of the three position reflector slider. Therefore, move the reflector slider to the right.
- 8. Adjust the illumination to the required level using the illumination menu item in the Control Window.
- 9. Observe the image of the eyepiece color video camera on the computer monitor using the Video Control Window in the WITec Control software. Using the drop down menu in the Video Control Window, select the appropriate view (top view with the correct objective).
- 10. Focus on the surface of your sample with the Microscope Z stage (U14). This can be done in three different ways:
 - Using the Z microscope control in the Graphic Control Window. Clicking on the arrows will start the movement in the indicated direction and using the virtual potentiometer, the speed can be adjusted from 0.01 to 500 $\mu m/s$.
 - Using the remote control with the Z Microscope selected as the controlled device. Using the +Z and -Z buttons, the Zfocusing-stage can be moved up or down and the potentiometer allows the selection of speeds between 0.01 and 500 $\mu m/s$.

HINT The remote control needs to be activated if the controlled device has changed or if it was idle for more than two minutes. To activate the remote control turn the potentiometer fully anticlockwise.

• Using the Move Up and Move Down buttons, which can be found in the Microscope Z Control menu of the Control Window. The speed can be adjusted between the minimum and maximum values using the corresponding field.

If possible, move the objective initially away from the sample to avoid a collision between the objective and the sample.

It can sometimes be very difficult to focus on flat and clean surfaces. The field stop diaphragm (M7) can help to overcome these difficulties as outlined in the following. To focus, close the field stop diaphragm (M7) to a value of 1-3. Make sure to move in the 50:50 beam splitter (M11) using the reflector slider (M10) to illuminate the sample. Approach the sample until the edge of the field stop appears focused. At this point, the sample is also in focus. This is due to the



fact that the field stop is positioned at the back focal plane of the objective. If the field stop is not in the middle of the field of view, move it to this position with the centering screws (M7; see the *alpha300/alpha500/alpha700* system description for details).

- 11. Move the sample to the area of interest using the XY positioner (U1).
- 12. Reset the position of the Microscope Z Stage (by pressing the Set Zero button in the Microscope Z Control menu).
- 13. Move the Microscope Z stage upwards several millimeters by using one of the controls described in point 10.
- 14. Attach an AFM-cantilever to the cantilever arm and mount it to the inertial drive.
- 15. Select the start adjustment sequencer () from the Control Window, which is located in the adjustment menu (). This will guide you through the alignment procedure. The requested user input is displayed in the Message Window. The following steps (indicated by the yellow bar on the left) are automatically requested and/or performed by the software.
- 16. Rotate the SPM objective into the working position and position the cantilever centrally and in focus in the top view video image. A coarse positioning (until the cantilever is somewhat visible in the video image) can be performed manually by gently moving the bottom part of the inertial drive in X, Y and Z. Once the cantilever is visible within the video image, the inertial drive should be used to position the cantilever correctly. The inertial drive can be driven in three different ways:
 - Using the cantilever positioning control in the Graphic Control Window. Clicking on the arrows will start the movement in the indicated direction and using the virtual potentiometer, the speed can be adjusted from 1 to 1500.
 - Using the remote control with cantilever position selected as the controlled device. Using the corresponding buttons, the cantilever can be moved in all three directions and the potentiometer allows the selection of speeds between 1 and 1500.
 - Using the |X+|, |X-|, |Y+|, |Y-|, |Z+| and |Z-| buttons, which can be found in the Adjustment⇒Cantilever Positioning menu of the Control Window. The speed can be adjusted between the minimum and maximum values using the corresponding field.

The cantilever is in its correct position once the beam deflection laser is in a position as shown in the video control image of Fig. 5. If the beam deflection laser is not visible try reducing the illumination brightness.



HINT

If the cantilever is angled significantly relative to the video image, check the position of the video camera. It should be oriented so that the connecting cable is pointing horizontally to the right. If the camera seems to be rotated significantly, this should be adjusted. Note that the video calibrations will be lost if the camera is rotated.

If the video camera seems approximately correctly oriented and the cantilever still shows a significant tilt in the video image (more than approximately 10°), the cantilever should be re-mounted.

17. Using the T-B and L-R adjustment screws on the front of the beam deflection unit, center the reflected beam deflection laser on the four quadrant photo diode. Use the Quadrant Window in the WITec Control software (see WITec Control manual Chapter 7) to observe the changes as you turn the screws. The red spot indicates the position of the beam deflection laser on the diode.

If it is on one of the edges of the field of view, observe the Σ -signal on the right hand side of the Quadrant Window. Turning the screw in the wrong direction will cause the sum signal to decrease whereas it will maximize if the screw is turned in the correct direction and the red spot will then move toward the center of the screen. If the Σ -signal is below 3 V, the bar representing the Σ -signal will turn red, which indicates that the Σ -signal is critically low.

The correct position has been reached when the red spot is in the center of the Quadrant Window (note that the display is non-linear and more sensitive toward the center), the T-B signal as indicated in the Status Window (see WITec Control manual Chapter 5) is $0\pm0.05\,\text{V}$ and the L-R signal is $0\pm0.1\,\text{V}$. The bar on the left hand side of the Quadrant Window (indicating the Z-position of the scan stage) should be fully extended at this point.

- 18. The feedback settings are preset upon loading the configuration. However, they should be checked before starting the approach. The recommended settings are a setpoint of 0.5 V, a proportional gain of 5.0 and an integral gain of 5.0.
- 19. Move the microscope down using the microscope Z stage (as described in step 10) until the cantilever is close to the sample, but be careful to not hit the sample.
- 20. Check the position of the beam deflection laser in the Quadrant Window again and correct it if necessary.
- 21. Press the Auto-Approach speed button (1) or select the Start Approach button from the Microscope Z Control menu. The Message Window (see WITec Control manual Chapter 6) displays the message: *Doing Contact-Mode Tip Approach*; *Moving fast* in the initial stages of the Auto-Approach.



22. As soon as the tip is in contact with the surface, the message in the Message Window changes to: *Doing Contact-Mode Tip Approach*; *Moving slowly*, the T-B signal in the Status Window will show 1.0 V (if that is the setpoint selected) and the red spot in the Quadrant Window will have moved up. If you hear a noise from the stage during contact, the feedback is too aggressive. In this case, reduce the I and P-gains until the noise stops. The scan table will now start to retract, which can be seen in a decrease of the height of the bar on the left hand side of the Quadrant Window and from the topography value in the Status Window. Once it has reached its middle position, the approach was successful and a corresponding message will be displayed in the Message Window.

The automatic approach may fail and/or be interrupted if one of the following problems occur. The solutions for the respective problems are indicated by the blue bars in the following.

- The approach is interrupted by the user clicking on the button. In the Message Window, the message *Tip Approach interrupted by user* is displayed.
- Approach again.
- ullet Instability of the sum signal. If the sum-signal increases by 10% or decreases by 20%, the automatic approach is interrupted. In the Message Window, the message *Dangerous Sum-Signal while approaching* is displayed.
 - Check the alignment of the beam deflection system and try the automatic approach again.
- No contact was registered after the microscope moved 2 mm down. In the Message Window, the message *Tip Approach stopped. Drove 2 mm down.* is displayed.
 - Check the alignment of the beam deflection system as well as the Micoscope Z position and approach again.
- The setpoint has been altered by the user during the approach. In the Message Window, the message *Tip Approach interrupted by user* is displayed.
- Restart the automatic approach using the desired setpoint.
- 23. The measurement parameters in the image scan (or line scan or distance curve) menu of the Control Window should be adjusted next (see WITec Control manual Section 3.5.2 (or 3.5.3 or 3.5.7) for a description of the individual



parameters).

If the (X) and (Y) positions indicated in the Scan Table menu of the Control Window differ from the position entered as Center (X) and Center (Y) in the image scan menu, the scan table will move to a different position before starting the scan. This might then lead to data acquisition at the wrong position/area. Use the Center at Current Pos. button in the image scan menu of the Control Window to avoid this displacement.

HINT

Typical values for an image scan in this mode are:

Points per line: 256-512 Lines per image: 256-512

Width: 2-30 μm Height: 2-30 μm Time per line: 0.5-2 s

24. You may now start your measurement.