User Guide

Semicon Process Refractometer
PR-33-S

VAISALA

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1. About this document

1.1 Version information

This document provides instructions for installing, using and maintaining the Vaisala K-PATENTS® in-line refractometer PR-33-S.

Table 1   Document versions (English)

Document code	Date	Description
IM-EN-PR33S-B	November 2021	Verification chapter added. Manual updated to new format and template. Minor changes to content.
IM-EN-PR33S-A	September 2019	First version.

1.2 Documentation conventions

WARNING! Warning alerts you to a serious hazard. If you do not read and follow instructions carefully at this point, there is a risk of injury or even death.

CAUTION! Caution warns you of a potential hazard. If you do not read and follow instructions carefully at this point, the product could be damaged or important data could be lost.

Note highlights important information on using the product.

Tip gives information for using the product more efficiently.

Lists tools needed to perform the task.

Indicates that you need to take some notes during the task.

1.3 Trademarks

Vaisala® and K-PATENTS® are registered trademarks of Vaisala Oyj.

Linux® is a registered trademark of Linus Torvalds.

Windows® is either a registered trademark or trademark of Microsoft Corporation in the United States and other countries.

All other product or company names that may be mentioned in this publication are trade names, trademarks, or registered trademarks of their respective owners.

2. Product overview

2.1 Vaisala Semicon Process Refractometer

The in-line refractometer PR-33-S measures the refractive index n_D and the temperature of the process medium. The concentration of the process liquid is calculated from these values when the composition of the process medium is known.

The output values of the refractometer are transmitted through its Ethernet connection by using a UDP/IP protocol. For more information, see Ethernet connection (page 9). An optional mA output unit is also available.
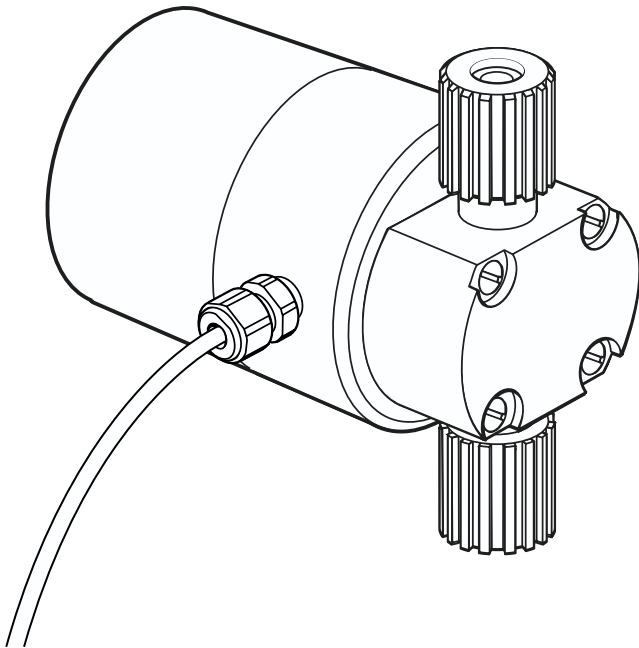
Figure 1   In-line refractometer PR-33-S

2.2 Safety

This product has been tested for safety. Note the following precautions:

> ⚠️ **WARNING!** Only licensed experts may install electrical components. They must adhere to local and state legislation and regulations.

> ⚠️ **WARNING!** Only trained service personnel of Vaisala and its representatives may service the refractometers. Servicing must be done according to separate instructions defined by Vaisala and must be reported to Vaisala.

The process medium may be hot or otherwise hazardous. Use shields and protective clothing adequate for the process medium. Do not rely on avoidance of contact with the process medium.

> 🥽 Wear protective eyewear.

> 🧤 Wear protective gloves.

Precautions when removing a refractometer from the process line:

- Check that the process line is depressurized and drained.
- Loosen the flow cell screws cautiously. Be prepared to tighten again.
- Make sure you are clear of any possible spillage and you have a clear emergency escape path.

3. Ethernet connection

The Ethernet connection enables data download from a PR-33 refractometer to a computer. Any type of computer (for example, PC, Mac, PDA, or mainframe) with a compatible network connection can be configured to view and download data from the refractometer.

You can configure and monitor the refractometer without special software by using a standard web browser. Communication protocol (page 43) provides all the specifications necessary to write a data acquisition program.

3.1 Ethernet specification

The refractometer is designed to be connected to a network with PoE (Power over Ethernet, IEEE-802.3af) modeA support, as it receives its operating power through the Ethernet. The refractometer is equipped with an integrated Ethernet cable, 10 m (33 ft) long. The cable can be connected to any standard RJ45 Ethernet equipment. For additional length you can use a UTP joint adapter/coupler with a standard Ethernet cable, length up to 50 m (164 ft). The maximum communication speed of the refractometer is 100 Mbit/s (Ethernet 100BASE-T).

In the simplest form the network consists of a refractometer, a computer, and a PoE switch. The following figure shows this configuration.

PR-33 | PoE switch | Laptop
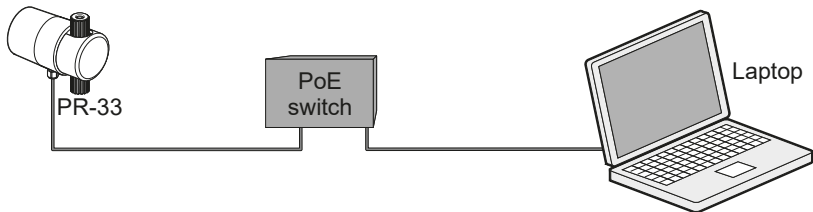
Figure 2   Simple network configuration

Several refractometers can be connected to the same Ethernet network. The refractometers also have an automatic function to detect the polarity of the network so that the network may utilize either cross-over or straight interconnecting cables.

The following figure shows an example of how to connect 3 refractometers to an existing LAN with no PoE functionality. A single PoE switch is enough in this case.
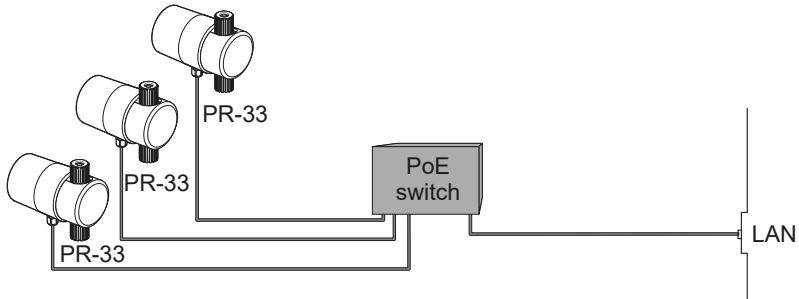
Figure 3   Three refractometers in same network

It is possible to use a WLAN access point to decrease the number of cables as shown in the following figure.
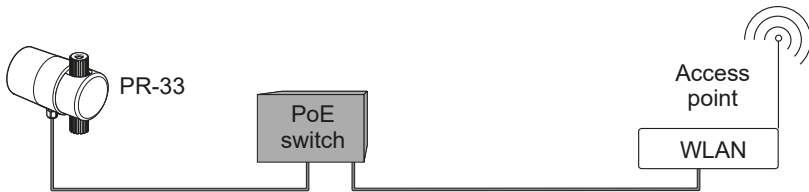
Figure 4   Connecting refractometer(s) using wireless

The maximum distance of a single Ethernet connection is 100 m (328 ft), including 1 joint adapter/coupler. If longer distances are required, you can use a fiber link to extend the range, as shown in the following figure.
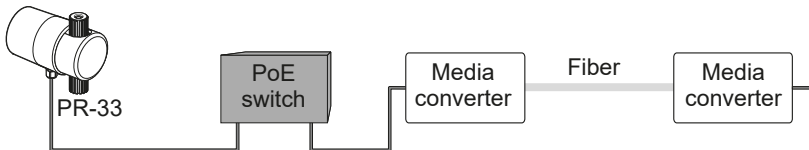
Figure 5   Using fiber link to connect refractometer(s)

3.2 Connection settings

The Ethernet connection settings for the refractometer consist of the following:

- IP settings
- Network configuration

3.2.1 IP settings for refractometer

All refractometers are shipped with the factory default IP address of 169.254.23.33.

This address belongs to the Zeroconf addresses (as defined in IETF standard RFC 3927) so that it can easily be reached from a stand-alone computer as shown in Figure 2 (page 9), usually without changing the network settings of the computer.

This address remains in the refractometer even after a different IP address has been set. The refractometer answers in the address that is first called up after startup.

> **i** If there is more than one PR-33 in the same network, this address cannot be used. See Configuring refractometer network (page 12).

Change the IP address of the refractometer through the instrument homepage. See Instrument homepage (page 17).

3.2.2 IP settings for stand-alone computer

When a computer with automatic IP settings (DHCP enabled) is turned on in a network with only the refractometer, the computer automatically obtains the IP address 169.254.x.x. In this case the factory default address of the refractometer can be used for connecting without any further changes in settings. If this does not work, make sure that the WLAN (Wireless network connection) is not active on the computer that is connected to the refractometer. If the WLAN is active, the computer's Ethernet connection may not function as expected. Obtaining the 169.254.x.x. address may take up to a minute.

If you still have difficulties connecting to the refractometer, check the computer's IP address by opening the command window (command prompt) and by typing the command `ipconfig` (press **ENTER** to give the command). In Mac OS X and Linux the same command is called `ifconfig`. The information given includes the computer's IP address. If the address does not start with 169.254, configure the IP address of the computer manually to 169.254.23.34, netmask 255.255.0.0.

For further troubleshooting, see Troubleshooting connection (page 15).

The following figure shows an example IP configuration for a stand-alone laptop when connected to the refractometer. The laptop wireless (WLAN) is turned off.
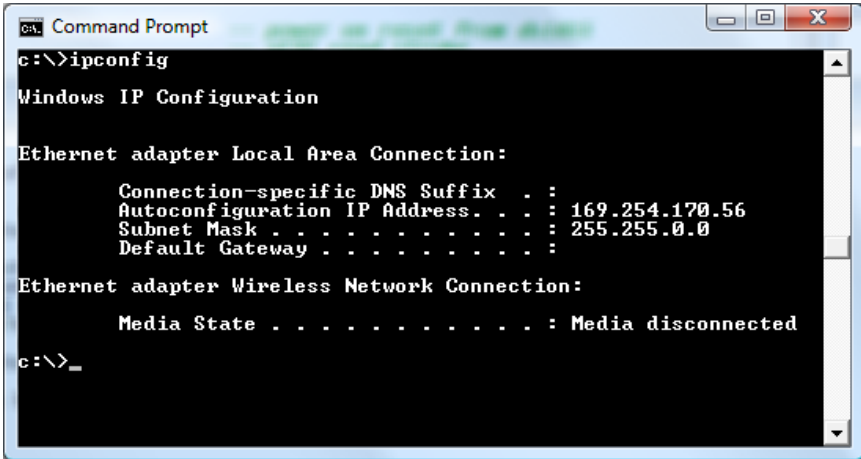
Figure 6   Typical stand-alone IP configuration

ℹ The connection does not work if the computer and the refractometer have exactly
   the same IP address.

When you have configured the network settings of the refractometer (and/or the computer)
according to the instructions above, you can proceed to test the connection as instructed in
Testing Ethernet connection (page 13).

3.2.3 Configuring refractometer network

If you have more than 1 refractometer in a network the refractometer IP addresses have to be
configured, as the factory default will not work.

If you are connecting the refractometer to a factory network, consult the network
administrator for the correct settings.

If the network is a stand-alone network with only PR-33 refractometers and 1 or more
computers with no connection to any other network, you can select the IP addresses. For
example, you can number the instruments so that they all have 192.168.33.x addresses, so
every computer and instrument has a different number x between 1 - 254. The subnet mask (or
netmask) is in this case 255.255.255.0, as shown in the following figure.

IP = 192.168.33.1   IP = 192.168.33.2   IP = 192.168.33.3

IP = 192.168.33.100
netmask = 255.255.255.0
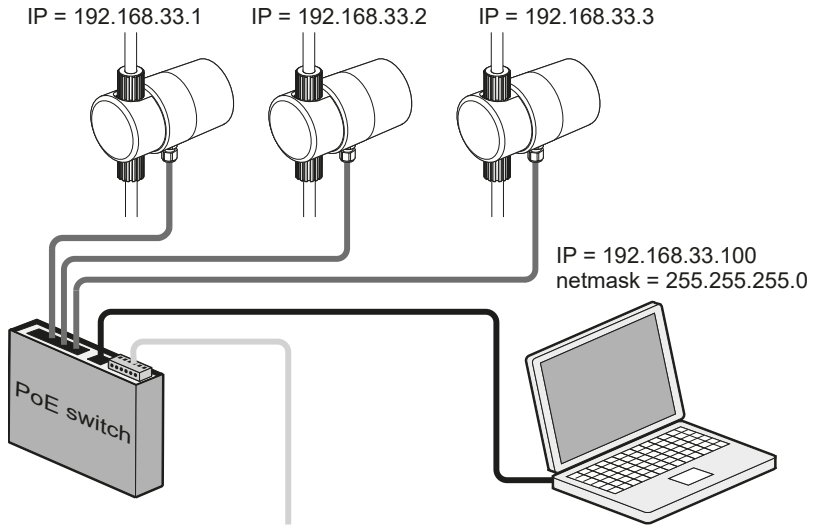
Figure 7   PR-33 refractometer network

ℹ️ There are no settings for subnet mask, default gateway or name servers in PR-33, as these settings are not required.

3.3 Testing Ethernet connection

When you connect the refractometer to a powered PoE (Power over Ethernet) switch, the indicator lights light up. The following figure shows an example of the indicators on a PoE switch. One indicator shows that the instrument is drawing power, another indicator (next to the Ethernet connector) shows the connection status. In the figure, a 100 Mbit/s connection is formed between 2 instruments at the switch ports 1 and 3 (1 refractometer for each port), and there is a PoE device connected in these ports, 1 and 3.

ℹ️ The actual position and function of the lights depend on the switch.
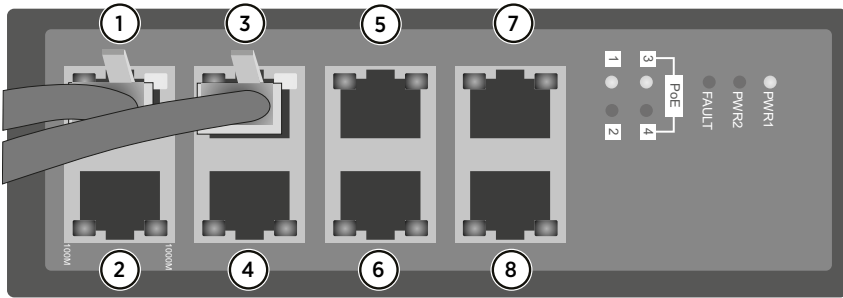
Figure 8   Example PoE switch indicator lights

It is typical that, for example, an 8-port PoE switch only has 4 ports supplying power.

If you keep the refractometer in a dimly-lit space, the flashing of the processor indicator lights is visible on the instrument cover. If the lights are flashing, the instrument receives power from the PoE connection.
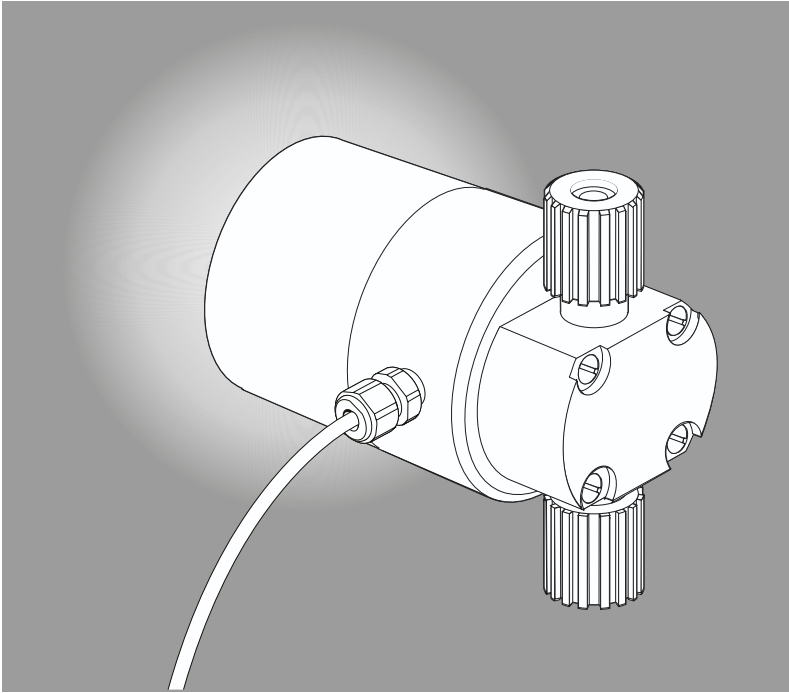
Figure 9   Process indicator lights in refractometer

Once the refractometer is powered up, you can reach it from any correctly-configured computer connected to the same Ethernet network by typing the IP address of the instrument in a web browser. For more information, see Instrument homepage (page 17).

> The factory-default IP address of the refractometer is 169.254.23.33. This address always responds. For more information, see IP settings for refractometer (page 11).

3.3.1 Troubleshooting connection

If you are unable to reach the instrument through the network, check the following:

- The instrument receives power; there is a faint flashing light on the cover of the instrument as shown in Figure 9 (page 15), or the PoE switch indicates **power OK** as shown in Figure 8 (page 14).
- The network settings of the computer are compatible with those of the instrument, see IP settings for stand-alone computer (page 11).
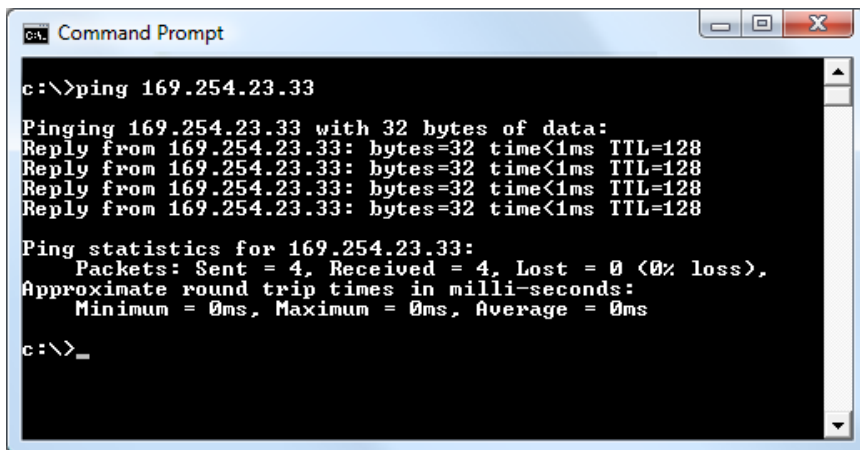
- If you try to reach the instrument at IP address 169.254.23.33, check that there is only one PR-33 in the same network, as otherwise there is an address conflict.
- Check that the software firewall of the computer does not block the connections.

A useful test to determine whether the problem is in network settings is to set up a small network. Perform the following steps:

- Set up a network of only one refractometer, PoE switch, and a computer as shown in Figure 2 (page 9).
- Check that the computer has suitable network settings and that its WLAN connection is turned off. For more information, see IP settings for stand-alone computer (page 11).
- Use the ping utility of the computer to try to reach the refractometer.

The ping utility is available in Windows systems by using the Command Prompt (usually found in the Accessories; or open **Run**, type **cmd** in the empty line and press **ENTER** to open Command Prompt).

To use ping go to the command interface, type the name of the command and the IP address you want to check and press **ENTER**. If the Ethernet connection is physically working and the address given to ping is correct, the refractometer answers the ping and returns any data packets sent to it.

Figure 10   Pinging address 169.254.23.33

For more information, see Instrument homepage (page 17).

4. Instrument homepage

Every PR-33 refractometer has a built-in web server with the instrument homepage. The homepage offers facilities to configure, monitor, diagnose and verify the instrument.

Once there is a functional Ethernet connection between the instrument and the computer, the homepage can be opened by typing the instrument's IP address to the address bar of a web browser. The recommendation is to use Firefox 2.0 or newer, but most functionality is available with any modern web browser.

Opening the instrument homepage

1. Establish a working Ethernet connection (see above) to the refractometer.
2. Start your preferred web browser (for example Mozilla Firefox, Internet Explorer, Safari, Chrome or Opera).
3. The address (URL) of the instrument homepage is the refractometer's IP address, for a factory set refractometer it is **`http://192.168.23.33/`**. Give the address to the browser just like you'd enter any other address (for example, **`http://www.vaisala.com/`**)
4. Wait until the homepage loads. This may take a few seconds.
   The page loads as shown in Figure 11 (page 18); the exact look of the page depends on your browser and screen settings, so slight variation can be expected.
5. Use the links in the link bar on the left side of the page to find more extensive information on the instrument.

> ℹ️ In order for the web pages to function as designed, JavaScript support must be enabled in the browser.

4.1 Main page

Once the instrument homepage is loaded, the most important information is visible on the **Main** page. This page shows the measurement values, serial number and the tag of the instrument.
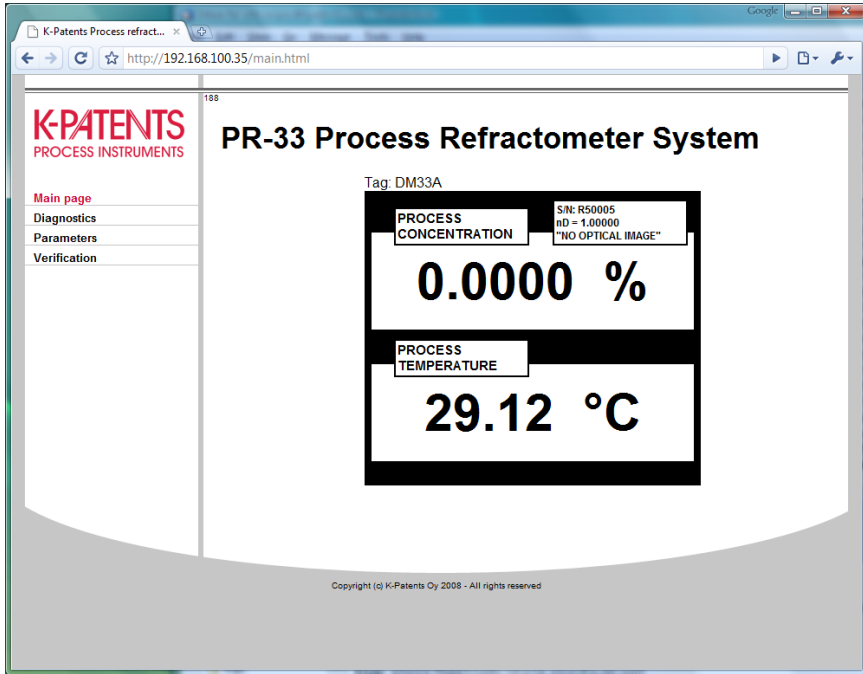
Figure 11   Main page (refractometer not in process)

4.2 Diagnostics

On the **Diagnostics** page you can see the diagnostic values produced by the refractometer. There is also a link to the optical image. The diagnostic values are updated live, but the optical image is only loaded on request.
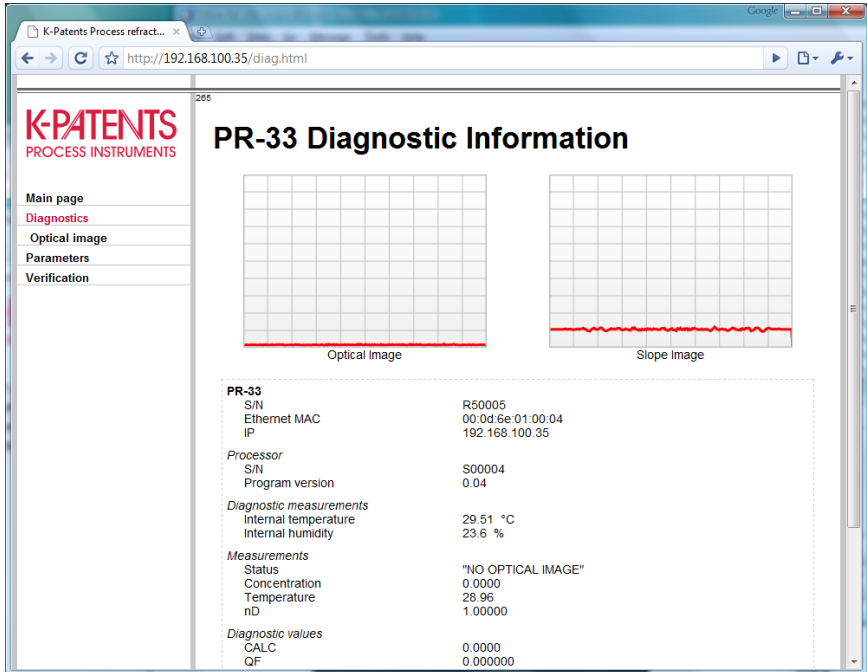
Figure 12   Diagnostics page

4.3 Parameters

You can change all the functional parameters of the refractometer on the **Parameters** page of the instrument. Type new parameters into the input fields.

Figure 13   Parameters page

Once the parameter editing is done, pressing the **Submit changes** button sends the parameters to the instrument after asking for a confirmation. Updating the parameters can take a few seconds.

> If you change the IP address of the instrument, you must enter the new address to the address bar of the browser, as the refractometer no longer exists at the old address.

4.4 Verification

Perform instrument verification on the **Verification** page. For more information on the verification procedure, see Refractometer verification (page 35).

Instrument verification

K-PATENTS®
PROCESS INSTRUMENTS

Main page
Diagnostics
Parameters
Verification
Verification report

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The instrument verification can be carried out by using standard refractive index liquids (available from K-Patents). The procedure consists of measuring a number of these liquids one-by-one and verifying the reading of the instrument.

Before starting the verification, please read through the following checklist:

- The verification requires the instrument to be removed from the process and placed onto a table prism pointing up.
- Depending on the instrument model, a sample holder may be needed.
- To carry out an acceptable verification, at least three R.I. standard liquids have to be measured successfully. The verification is valid only within the range of these liquids.
- The instrument must reach a stable temperature before verification. This temperature must be between 20 °C and 30 °C. The nominal temperature for the liquids is 25 °C, but there is an automatic temperature compensation which enables a wider temperature range.

To carry out the verification, do the following for each standard R.I. liquid:

1. Make sure the prism (and sample holder) is clean and dry before placing the liquid on the prism.
2. Keep an eye on the optical image; it will help in determining the sufficient amount of liquid on the prism; if there is too little liquid, the shape of the optical image will change.
3. Once the liquid is on the prism, click the "New verification point" button. A progress indicator will show the progress. Once the point is measured, the measured values are reported.
4. Clean the prism and repeat the procedure for each standard liquid.

You may remove a failed point by clicking the "Remove" button on the line. If you use the same liquid several times, only the newest result will be recorded.

Once you have carried out the measurement for all points, click the "Save verification" button. This will save the verification results into the instrument and show the verification report. The latest verification report can always

Figure 14   Verification page

5. In-line refractometer

5.1 Refractometer mounting

Choose the refractometer mounting location with care to ensure reliable readings from the process.

5.1.1 Choosing refractometer mounting location

The mounting location needs to be such that sediments or gas bubbles cannot accumulate by the refractometer. Good flow velocity is essential in keeping the prism clean.

⚠️ **CAUTION!** If the process pipe vibrates, support the pipe. A vibrating pipe might damage the in-line refractometer mounted on it.

Do not expose the refractometer cover to high temperature radiation. In most cases, draft and natural convection provide sufficient air cooling if the air gets to flow freely around the refractometer head.

5.1.2 PR-33-S refractometer installation
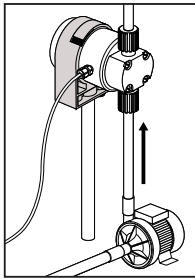
MOUNTING RECOMMENDATIONS

REFRACTOMETER SUPPORT - VERTICAL FLOW
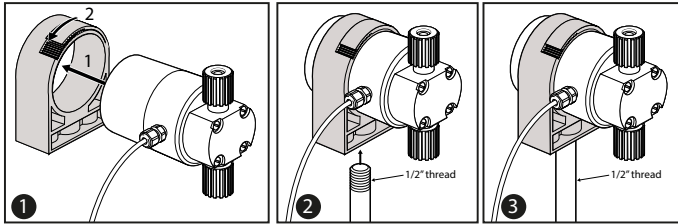
REFRACTOMETER SUPPORT - HORIZONTAL FLOW
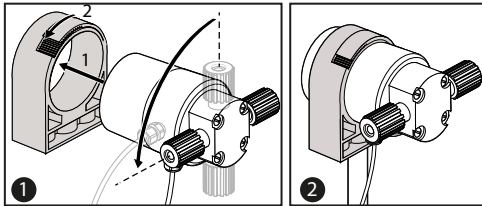
FLOW CELL ASSEMBLY/DISASSEMBLY   MOUNTING COVER ASSEMBLY
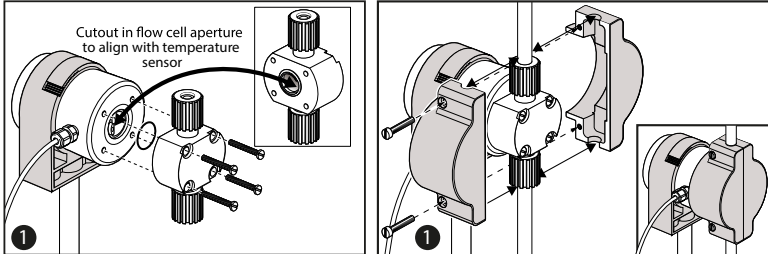
Figure 15   PR-33-S installation
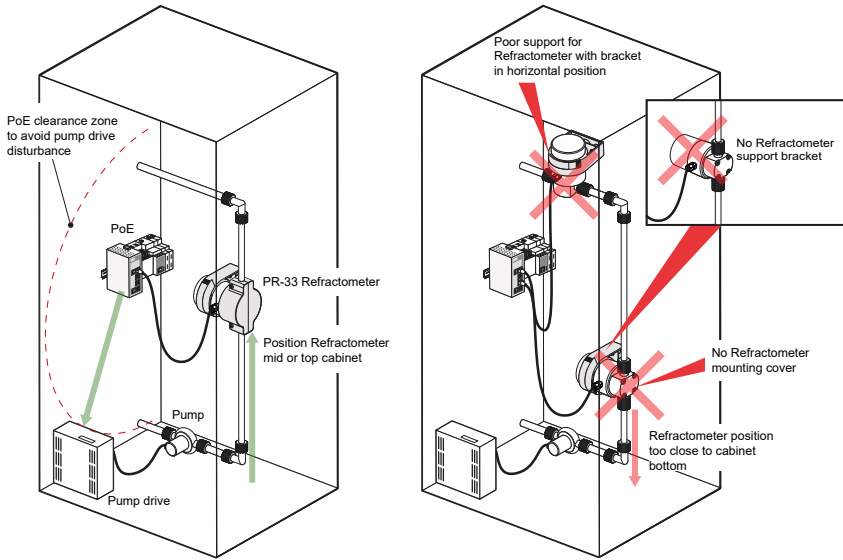
Figure 16   Mounting in cabinet

5.1.3 Pipe mounting checklist

Most Vaisala K-PATENTS® in-line refractometer models are mounted in a pipe. The recommended minimum flow velocity is 1.5 m/s (5 ft/s). The diameter and form of the pipe and the process temperature all affect the measurement and need to be taken into account.

1. If the process pipe diameter varies, select the *position with the smallest diameter* (and accordingly highest velocity). Then the prism stays clean better.
2. If the refractometer is used in a feedback control loop, *make the time lag short*. For example, when a dilution valve is controlled, mount the refractometer close to the dilution point. However, make sure complete mixing has occurred at mounting location.
3. If the temperature varies along the process pipe, select the *position with the highest process temperature*. This minimizes the risk of coating, because higher temperature means higher solubility and also lower viscosity.
4. Often the *position with the highest process pressure* (after the pump and before the valve) has favorable flow conditions without sedimentation or air trapping risks.
5. The refractometer should be conveniently accessible for service.

5.1.4 Mounting cover

Mounting covers come in the following sizes:

- Small mounting cover for 6.35 mm (0.25 in) and 12.70 mm (0.50 in) flow cells
- Large mounting cover for 19.05 mm (0.75 in) and 25.4 mm (1.00 in) lines

Fit the 2 halves of the mounting cover over the flow cell and secure with the screws provided.
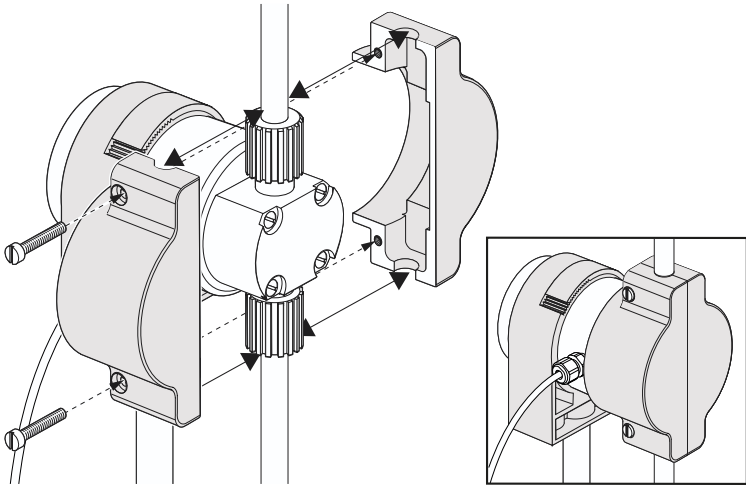
Figure 17   Mounting cover assembly

5.2 Connecting refractometer

Connect the refractometer to a PoE switch. Make sure you connect the refractometer to a powered port, some switches have both PoE ports and normal Ethernet ports.

Connect your laptop or control system to the switch. You can also use a FC-11 Field Communicator.

PR-33-S refractometer
with Ethernet cable
IP: 169.254.23.33

PoE switch

Computer with
Ethernet and
browser

PR-33-S refractometer
with Ethernet cable

PoE switch

Control
system
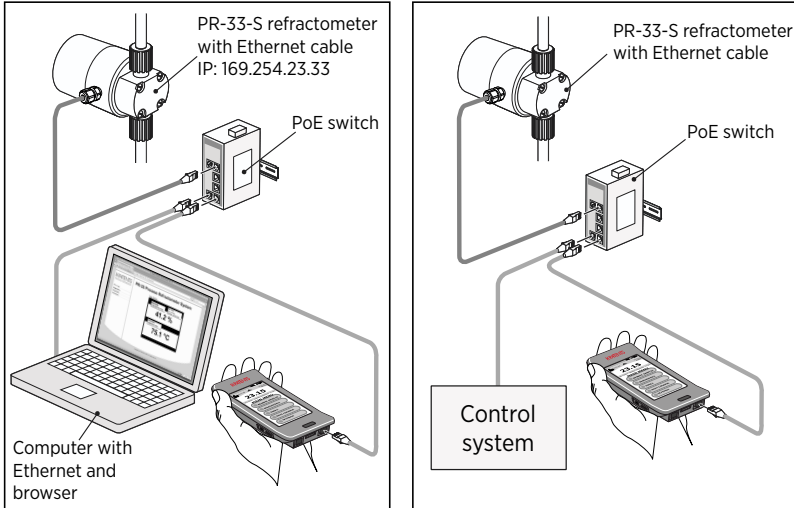
Figure 18   Ethernet connections

5.3 Connecting with Fieldbus converter

The Vaisala K-PATENTS® Fieldbus converter allows the connection to Fieldbus and industrial
Ethernet. For more information, see the Fieldbus converter manual.

> The converter can only send data. It is not possible, for example, to change the
> parameters through the converter.

PR-33-S
Refractometer
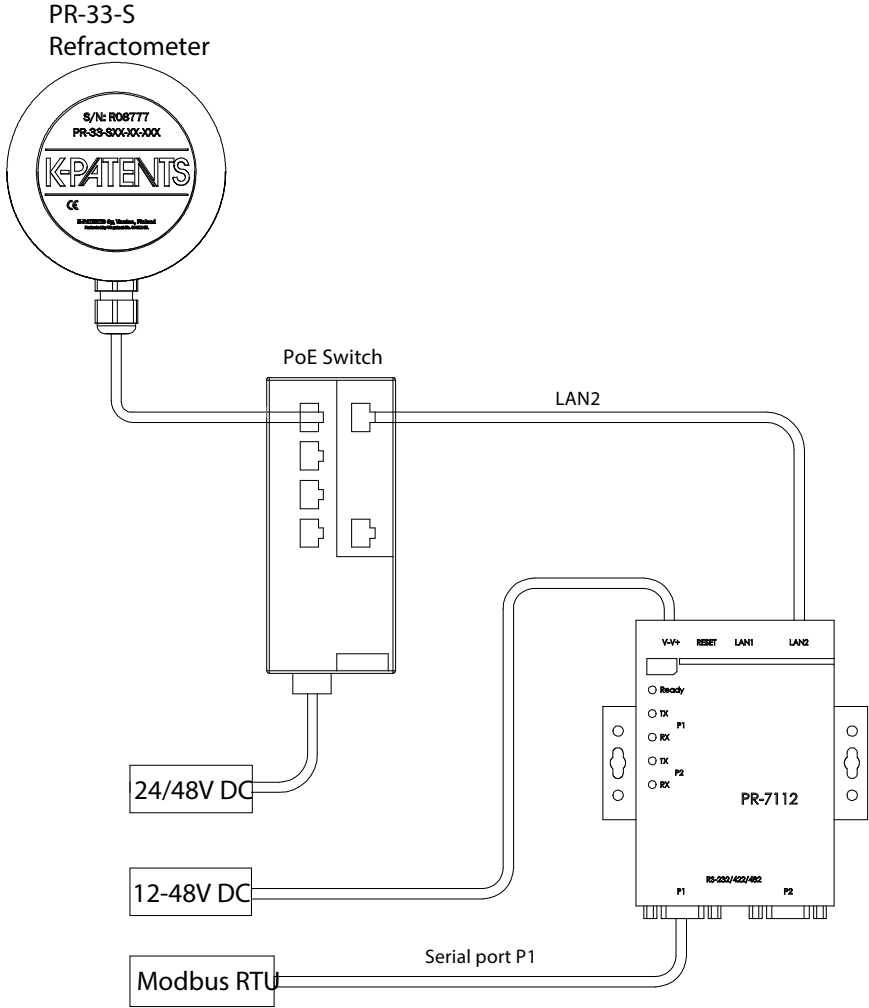
Figure 19   Connecting to Modbus RTU

PR-33-S
Refractometer
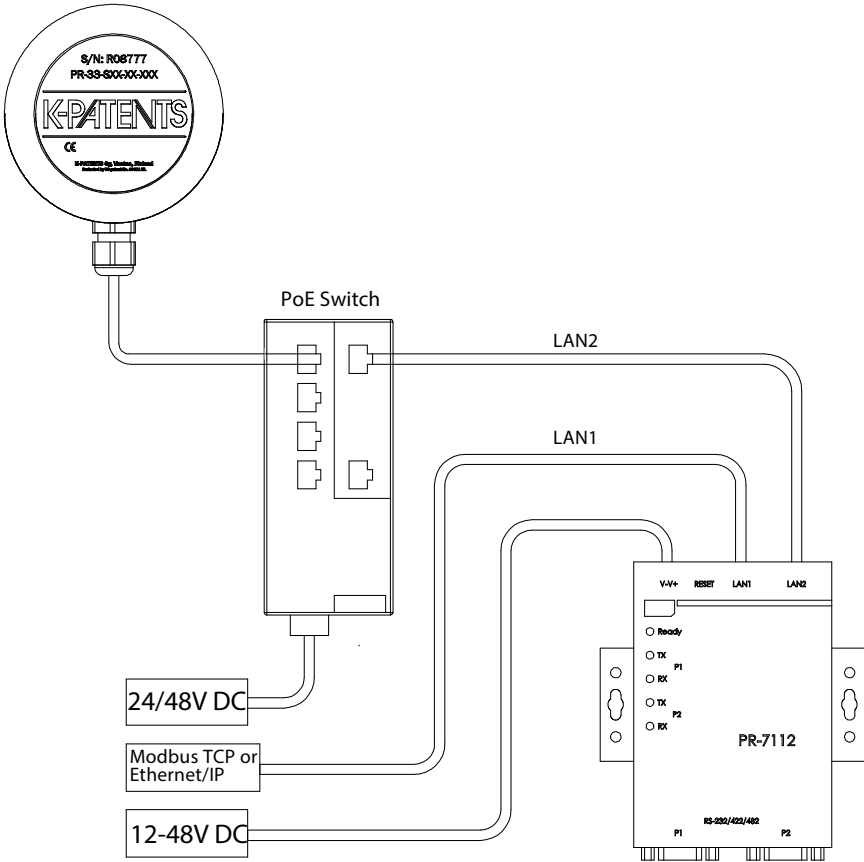
Figure 20   Connecting to Modbus TCP

6. Startup and use

6.1 Startup

The refractometer startup procedure includes the following checks:
- Initial check
- Calibration check

6.1.1 Initial check

Connect the refractometer to a PoE enabled network, and check that the instrument powers up properly. For more informaiton, see Testing Ethernet connection (page 13). Use a web browser to open the instrument homepage andcheck that the serial number of the instrument corresponds to that on the instrument nameplate. If you have difficulties connecting to the refractometer, see Troubleshooting connection (page 15).

6.1.2 Calibration check

Wait until normal process conditions occur. The concentration reading is precalibrated at delivery and a copy of the calibration certificate is shipped with the refractometer. If the diagnostic message is **Normal operation** but the concentration reading does not agree with the laboratory results, see Calibrating concentration measurement (page 31).

6.2 Viewing refractometer status

The **Main** page of the instrument homepage shows the basic information on the measurements, see Main page (page 17). The **Diagnostics** page shows more information, see Diagnostics (page 18).

The measurement result is calculated from the refractive index (n_D) and process temperature (T) values. Both of these values are available on the **Main** page.

In addition to these measurements, the refractometer monitors its internal temperature and humidity, which are both available on the **Diagnostics** page. The internal temperature should be below +55 °C (+131 °F), and the humidity should be below 30 %.

7. Configuration and calibration

7.1 Configuring refractometer

In PR-33, all parameter changes are made with a web browser through the **Parameters** page, see Parameters (page 19).

7.1.1 Signal damping

The system provides the possibility to enter signal damping to diminish the influence of process noise. The damping is applied to the CONC value (and thus the output signal).

The PR-33 offers two types of signal damping. **Exponential (standard) damping** works for most processes and is the standard choice for slow and continuous processes. However, if the process has fast step changes, **linear (fast) damping** gives shorter settling time.

The damping time is set separately. What the damping time means in practice depends on the damping type:

- With *exponential damping*, the damping time is the time it takes for the concentration measurement to reach half of its final value at a step change. For example, if the concentration changes from 50 % to 60 % and damping time is 10 s, it takes 10 seconds for the refractometer to display concentration 55 %. A damping time of 5 … 15 s seems to work best in most cases.
- With *linear damping* (fast damping), the output is the average of the signal during damping time. After a step change the signal rises linearly and reaches the final value after the damping time.

The following figures show how the damping time affects the measurement.

> ℹ️ The factory setting for damping in PR-33-S is **5 s linear**. Avoid overdamping, the signal should not be made insensitive.
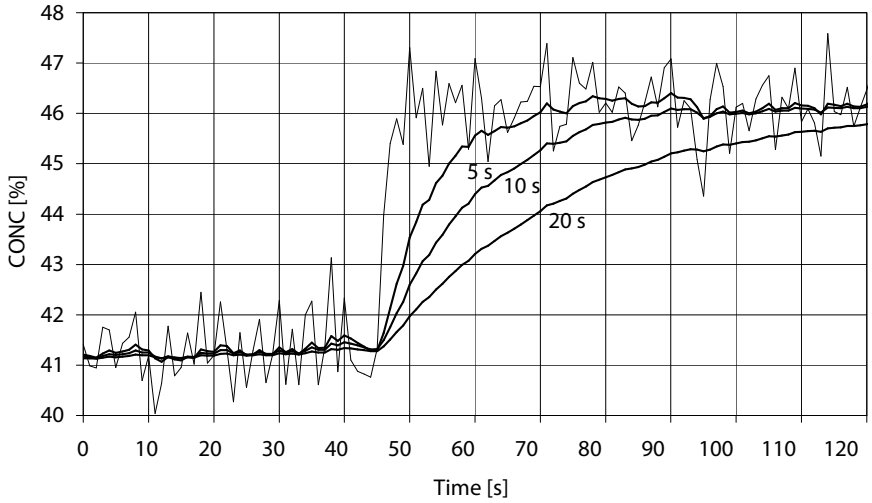
Figure 21   Exponential damping
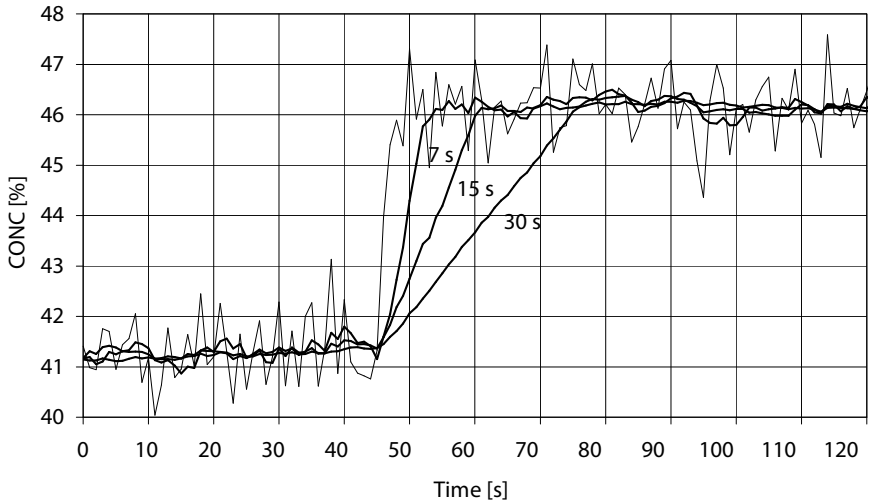
Figure 22   Linear damping

7.2 Calibrating concentration measurement

The concentration calibration of the in-line refractometer PR-33 is organized in 6 layers.

1.

| CCD | Pt-1000 |

2.

| n_D | TEMP |

3.

| CHEMICAL CURVE |

CALC

4.

| FIELD CALIBRATION |

5.

| DAMPING |

CONC

6.

| ETHERNET |
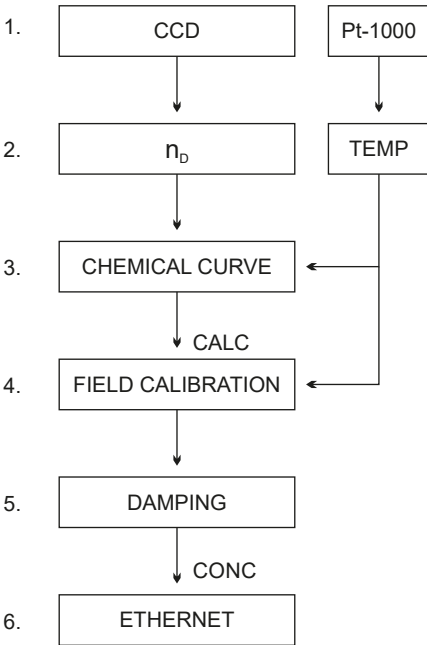
Figure 23   Calibration layers

1   The information from the CCD element and the Pt- 1000 temperature element. The position of the shadow edge is described by a number called CCD and scaled from 0 to 100 %.

2   The refractometer calibration: The actual refractive index n_D is calculated from the CCD value. The process temperature is calculated from the Pt-1000 resistance. The refractometer output is n_D and temperature TEMP in Centigrade.
    The calibration process ensures same n_D measurement performance for all PR-33-S refractometers, making them interchangeable. The calibration of each refractometer can be verified using standard refractive index liquids.

3   The chemical curve: The refractometer calculates the Brix value based on n_D and TEMP. The result is a temperature compensated calculated concentration value CALC.

4   Field calibration: Adjustment of the calculated concentration value CALC may be required to compensate for some process conditions or to fit the measurement to the laboratory results.

5   Damping.

6   Output signal: The output signal is transmitted through the Ethernet connection.

The calibration of each refractometer can be verified using standard refractive index liquids.

Field calibration determines the appropriate adjustment to CALC. The adjusted concentration is called CONC. If there is no adjustment, CALC and CONC are equal. Thus the chemical curve is kept intact as a firm base for the calculation, the adjustment is merely additional terms.

For more information on damping, see Signal damping (page 30).

> By adding an optional mA output unit, the Ethernet signal can be converted into mA output.

7.2.1 Chemical curve

The chemical curve is the theoretical concentration curve based on n_D and TEMP. It is defined by a set of 16 parameters.

C_{00}	C_{01}	C_{02}	C_{03}
C_{10}	C_{11}	C_{12}	C_{13}
C_{20}	C_{21}	C_{22}	C_{23}
C_{30}	C_{31}	C_{32}	C_{33}

A chemical curve is specific to the given process medium, for example, sucrose or sodium hydroxide. The set of parameters is given by Vaisala and should not be altered, except in case of changing to another process medium.

Vaisala provides a field calibration service that adapts the calibration to the factory laboratory determinations based on the data supplied. The field calibration procedure should be made under normal process conditions using standard laboratory determinations of sample concentration.

Record the calibrating data on the Refractometer field calibration form (page 50), also available by emailing a request to helpdesk@vaisala.com. Email the completed field calibration form either to helpdesk@vaisala.com or your local representative. A computer analysis of the data is made at Vaisala and optimal calibration parameters is sent to be entered in the system.

For a complete report, 10 to 15 valid data points are needed. A data point is of use for calibration only when the diagnostic message is **Normal operation**. Each data point consists of:

- LAB%: Sample concentration determined by the user
- CALC: Calculated concentration value
- T: Process temperature measurement in Centigrade
- nD: Actual refractive index n_D
- CONC: Measurement in concentration units, the large size number

In addition to the calibration data, write down the refractometer serial number.

Accurate calibration is only achieved if the sample is taken correctly. Pay special attention to following details:

- The sampling valve and the refractometer should be installed close to each other in the process.

> ⚠️ **WARNING!** Wear protective clothing appropriate to your process when operating the sampling valve and handling the sample.

- Run the sample before starting to collect data points to avoid sampling old process liquid that has remained in the sampling valve.

- Read the values CALC, T(emp), nD and CONC at exactly the same time with sampling.
- Use a tight container for the sample to avoid evaporation.

⚠️ **CAUTION!** Offline calibration using process liquid very seldom gives reliable results, as problems are caused by:
- Low flow which makes sample to form an unrepresentative film on the prism.
- Sample evaporation at high temperature or undissolved solids at low temperature giving deviations from laboratory determinations.
- An ageing sample which is not representative.
- Outside light reaching the prism.

Always make calibration using the process liquid *in-line*.

7.2.2 Direct BIAS adjustment

The concentration measurement value can also be directly adjusted by changing the field adjustment parameter f00.

The value of the bias parameter f00 is added to the concentration value:

NEW CONC = OLD CONC + f00.

8. Instrument verification

8.1 Refractometer verification

The refractometer calibration is verified using a set of standard refractive index liquids. In order to perform a valid verification, at least three liquids need to be used. The verification is valid only within the refractive index range defined by these three liquids.

The instrument automatically recognizes the following refractive index standard liquids. The values are stated at +25 °C (+77 °F):

- 1.3200
- 1.3300
- 1.3400
- 1.3500
- 1.3600
- 1.3700
- 1.3800
- 1.3900
- 1.4000
- 1.4100
- 1.4200
- 1.4300
- 1.4400
- 1.4500
- 1.4600
- 1.4700
- 1.4800
- 1.4900
- 1.5000
- 1.5100
- 1.5200

The accuracy of the certified standard refractive index liquids is ± 0.0002 and they can be traced back to NIST standards #1823 and #1823 II. As the specified accuracy of the refractometer is ± 0.0002, then the representative level is the sum of the two accuracy specifications, that is ± 0.0004.

Vaisala provides a set of standard R.I. liquids, PR-2300, containing five selected liquids (1.3300, 1.3700, 1.4200, 1.4700, 1.5200). The set can be ordered from Vaisala. Other liquids are available on request.

8.1.1 Handling R.I. liquids

Use gloves and safety glasses or goggles. Make sure the ventilation is good, local ventilation is preferable. Review the safety instructions and the MSDS shipped with the liquids (valid inside R.I. range 1.30-1.57, safety markings valid in EU/EEA areas). Do not put tissues or liquid bottles in household waste, dispose of waste according to local regulations for chemical waste.

8.2 Verification procedure

Perform instrument verification using the web interface. On the instrument homepage, go to the **Verification** page. The standard liquids are placed on the prism as instructed by the instructions on the screen. Once the liquid has settled, sample is measured by clicking the **New verification point** button.

The liquids are automatically detected, and the quality of the measurement is constantly monitored throughout the process. If the same liquid is measured several times, the new result replaces the older one. To remove a single measurement point, click the **Remove** button.

The verification result displays on the page. Reloading the page removes all points. After measuring a sufficient number of points the verification can be saved by clicking the **Save verification** button.

In order to avoid verification errors, please make sure:

- The temperature has settled, i.e., the refractometer is in the ambient temperature
- The temperature of the refractometer is between +20 °C (+68 °F) and +30 °C (+86 °F)
- The prism is cleaned properly before placing the sample
- The test liquid wets the prism properly

Check the temperature of the refractometer by following the temperature measurement on the **Verification** page. The temperature should be constant.

Figure 24   Verification page

You can pour the sample R.I. liquid into the prism notch. Make sure to cover the liquid so that light does not get into the sample.

The optical image displayed on the page helps determining whether the prism is wetted completely. The image should have a sharp corner as in the figure above. The position of the corner depends on the refractive index. A soft image may indicate improper cleaning of the prism. If there is not enough liquid on the prism, the image tends to flatten. If the image changes its shape during the measurement of a single liquid, redo the measurement.

After measuring a sample, remove the sample and clean the prism. The prism can be cleaned by soft tissue and ethanol or other suitable solvent. Repeat the procedure (cleaning, replacing the sample, measuring) for each n_D liquid. In case you perform the procedure more than once for a single sample, the latest measurement will replace earlier measurements.

The table on the verification page keeps a real time record of the points measured and the status (pass/fail) of the verification. If you have a failed point, either remove it or remeasure it.

Once all liquids have been measured, save the verification by clicking the **Save verification** button. The button is only available after a sufficient number of points has been measured.

The limit for acceptance is that all measurements must be within ± 0.0004 of the nominal values.

> ℹ The refractometer verification concerns only the refractive index n_D measurement. The calculation of concentration from n_D and process temperature TEMP is not included.

8.3 Verification report

The last saved verification data can be accessed by clicking the Verification report link on the left panel. This verification report includes all measurement data and the PASS/FAIL status of the verification. The report can be printed out or saved as a reference for the quality system.

PR-33 verification

The latest saved verification is shown in this report. The verification is valid within the range of standard refractive index liquids used in the verification process. If traceable index of refraction liquids are used in the process, a successful verification proves the standard traceablity of the instrument.

Instrument S/N: **R11502**
Verified at: **15:25:27 20-Mar-2018**

K-PATENTS
PROCESS INSTRUMENTS

Main page
Diagnostics
Parameters
Verification
Verification report

| STANDARD LIQUID | | MEASUREMENT | | | VERIFICATION RESULT | |
@ 25°C	@ T	T	nD	CCD	nD error	Status
1.34	1.339217	27.32	1.339192	83.465	0.000025	PASS
1.37	1.369189	27.37	1.369097	68.023	0.000092	PASS
1.41	1.409031	27.37	1.409202	52.878	0.000171	PASS
1.52	1.519018	27.41	1.519127	15.263	0.000109	PASS

Verification result: **Verification successful (1.34 .. 1.52)**

Figure 25   Verification report

8.4 Corrective action

If the verification is not passed, first check that the prism is absolutely clean before a standard liquid is applied. Make sure the standard liquids are in good condition and not past their expiration date. Also, inspect the prism surface, checking that it is flat and shiny without any scratches, digs or deposition on it.

Repeat the verification procedure. If the verification still fails, print out the verification report, send the data to helpdesk@vaisala.com or your nearest representative, and wait for further instructions.

9. Regular maintenance

9.1 Preventive O-ring replacement in EKC265 application

The amine-based chemical solvent EKC265 is formulated to remove post-etch residue from the wafer. The Semicon Process Refractometer is widely used to monitor the water content of the solvent. Field studies and experience have shown that the O-ring between the refractometer instrument body and flow cell has limited lifetime in EKC265. The current standard O-ring material is a perfluoroelastomer of Kalrez 6375UP, manufactured by Dupont, and this material has shown swelling after 6 months in use.

Preventive maintenance replacement of the O-ring PR-9252 every 6 months will ensure equipment reliability and prevent O-ring failure.

O-ring PR-9252
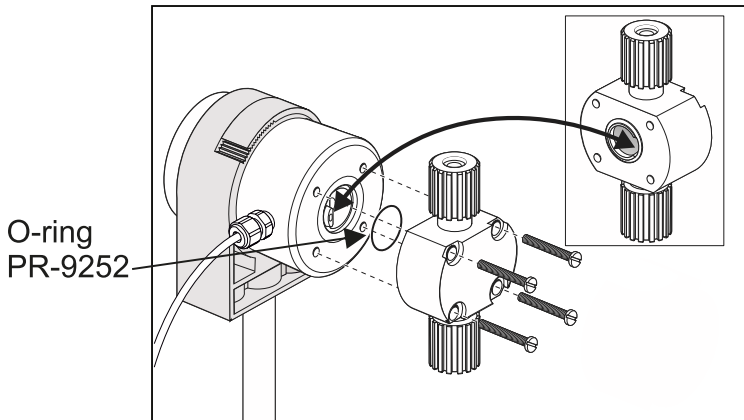
Figure 26   Replacing O-ring

10. Troubleshooting

10.1 Diagnostic message priorities

> 💡 The messages are listed in descending order of priority. For example, if both **NO OPTICAL IMAGE** and **TEMP MEASUREMENT FAULT** are activated, only **NO OPTICAL IMAGE** displays.

- **OUTSIDE LIGHT ERROR**
- **NO OPTICAL IMAGE**
- **TEMP MEASUREMENT FAULT**
- **HIGH SENSOR HUMIDITY**
- **HIGH SENSOR TEMP**
- **NO SAMPLE**
- **PRISM COATED**
- **OUTSIDE LIGHT TO PRISM**
- **LOW IMAGE QUALITY**
- **Normal operation**

The following sections list the diagnostic messages, their causes and corrective actions.

10.2 Hardware

The following table describes the hardware messages and their related actions.

Table 3   Hardware messages

Message	Description	Corrective action
HIGH SENSOR HUMIDITY	Tells that humidity measured inside the instrument exceeds 60 % relative humidity. The reason may be moisture leaking in through prism seal or the cover being open.	Contact Vaisala.
HIGH SENSOR TEMP	The temperature inside the instrument exceeds +65 °C (+150 °F). To read this temperature, go to the **Diagnostics** page. For more information, see Diagnostics (page 18).	See Choosing refractometer mounting location (page 22).

10.3 Measurement

The following table describes the measurement messages and their related actions.

Table 4   Measurement messages

Message	Description	Corrective action
OUTSIDE LIGHT ERROR or **OUTSIDE LIGHT TO PRISM**	The measurement is not possible or is disturbed because outside light reaches the camera.	Identify the light source and block the light from getting to the prism at the refractometer tip. The amount of outside light can be seen at **BGlight** on the **Diagnostics** page.
NO OPTICAL IMAGE	The prism is heavily coated.	Remove the refractometer from line and clean prism manually.
	There is moisture condensation in the refractometer.	See Hardware (page 40).
	The refractometer temperature is too high.	
	The light source is faulty. When the refractometer is removed from the process, the yellow flashing light can be seen through the prism. ⓘ The light is only visible at an oblique angle.	Check the LED value on the **Diagnostics** page. For more information, see Diagnostics (page 18). If the value is clearly below 100, LED fault is not likely. If the value is above 100, contact Vaisala.
	There are negative spikes in the optical image. The probable cause is dust or fingerprints on the CCD window.	Contact Vaisala.
	The CCD card in the refractometer is faulty.	
PRISM COATED	The optical surface of the prism is coated by the process medium or impurities in the process medium.	Remove the refractometer from line and clean prism manually. If the problem is recurrent, consider improving the flow conditions. For more information, see Choosing refractometer mounting location (page 22).
LOW IMAGE QUALITY	The most likely cause for this message is coating on the prism. There still is an optical image available, but the measurement quality may not be optimal.	
NO SAMPLE	The operation of the equipment is OK but there is no process liquid on the prism. In some cases this message may also be caused by coating on the prism.	

Message	Description	Corrective action
TEMP MEASUREMENT FAULT	Indicates faulty temperature element.	Contact Vaisala. ℹ️ A difference to some other process temperature measurement is not a fault. The refractometer shows the true temperature of the prism surface.
Concentration drift during **Normal operation**	For drift upward or downwards to zero, cause is likely the prism coating.	Remove the refractometer from line and clean prism manually. If the problem is recurrent, consider improving the flow conditions. For more information, see Choosing refractometer mounting location (page 22).

11. Ethernet protocol specification

The main purpose of the Ethernet connection is to collect measurement data from the instrument. For this data acquisition, you'll need to have suitable software on your computer. You can program a data acquisition program yourself following the specifications in the following sections.

For examples and ready-made applications, contact Vaisala.

11.1 Communication protocol

The communications protocol is based on **UDP/IP** to **port 50023**. It is a client/server protocol, where the refractometer is the server and so only sends information when the client (your computer) requests it. The server answers all requests within 100 ms.

11.1.1 Request format

Client to server communication is the requests sent from your computer to the refractometer in binary format. The request packets contain the following binary data (all integers are in the network order, MSB first):

- 32-bit integer: packet number
- 32-bit integer: request ID
- (any): request data (depends on the request)
- (any): fill-in data

> ℹ The maximum size of the message is 1472 octets (bytes).

The **packet number** is echoed back by the refractometer, but not processed in any way. The packet numbers do not have to be sequential, any 32-bit value is valid.

The **request ID** is a 32-bit value that identifies the requested function, for example refractometer information. See Request-response pair specification (page 45).

The **request data** consists of 0 to 1464 octets of additional data associated with the request.

The **fill-in data** can be used to increase the number of octets in a message. Any number of NULL characters (0x00) may be added to the end of the request as long as the total size of the message does not exceed the maximum of 1472 octets. This may be useful, for example, if the client implementation uses fixed-length packets.

11.1.2 Response format

The instrument sends response data in ASCII format. With the exception of the packet number, the data is human-readable. The data structure is as follows:

- Packet number (32-bit integer)

- zero or more lines of ASCII (text) keys and values associated with these keys (for example, temperature key and process temperature in Celsius)

The **packet number** is echoed back without change. The client (software on computer) can use the packet number to check the response against the packet number of the request.

The **message text** consists of lines of text, each line a single key (of one word) and its value or values. The values are separated from the key by an equal sign (=) and multiple values are separated by a comma. White space (space or tabulator) is allowed anywhere except within a single value or key name.

If the response consists of a character string, it is enclosed in double quotes ("). For example, all of the following are valid message text lines:

```
ok
```

```
temp = 23.45
```

```
headhum = 13.32
```

```
LEDcnt = 8341
```

```
ChemCurve = 1.234, 3.21, 0.00, 4.37, 1.11, 0.00002, 2.1345
```

```
StatusMessage = "Normal Operation"
```

> ℹ All the key identifiers (see Section 8.2 for additional information) are case-insensitive. However, the recommendation is that they are written as in this specifi- cation.

The server (refractometer) may send the response keys in any order. It sends the mandatory keys of the specific request, marked with an asterisk in Request-response pair specification (page 45), but it may omit any other keys. The server may also send keys that are not specified in this document, but the client (computer) may ignore them.

11.1.3 Request and response errors

When the server (refractometer) detects an error, it responds with an error message. For more information, see Error message specification (page 46). An error message can be caused, for example, by an unknown request or inability to collect data for the mandatory keys of a response.

11.2 Request-response pair specification

The following table describes the query messages, that is the request-response pairs, used for data collection using the Ethernet connection.

> ℹ️ Response keys that are always sent are preceded by an asterisk (*).

Table 5   Request-response pair specification

Specification	Description	Request ID	Request data	Response key
NULL message	Included in query messages for debugging purposes as it can be used to check whether the server is listening. The message gives a high-level 'ping' functionality.	0x00000000	(none)	• IP: IP address • MAC: Ethernet MAC address
Protocol version	Responded to with a value representing the server (refractometer) protocol version.	0x00000001	(none)	*Version: integer, the server protocol version (currently 3)
Refractometer information	Gives the basic information of the refractometer.	0x00000003	0x00000000: always zero	• *SensorSerial: string, refractometer serial number • *SProcSerial: string, processor card serial number • *SensorVersion: string, version number of the refractometer software

Specification	Description	Request ID	Request data	Response key
Measurement results	Gives the measured and calculated measurement values from the refractometer.	0x00000004	0x00000000: always zero	• Status: string, refractometer status message • PTraw: integer, PT1000 value • LED: float, LED value • RHsens: float, internal humidity • nD: float, calculated n_D value • CONC: float, final concentration value • Tsens: float, internal temperature • T: float, process temperature • CCD: float, image shadow edge • CALC: float, calculated concentration value • QF: float, quality factor • BGlight: integer, background light

11.3 Error message specification

If the server (refractometer) does not recognize the request or cannot fulfill it, it responds with an error message. The error message has the following keys:

- *Error: integer, error code 0x00000000: Unknown request
- *Error: integer, error code 0x00000001: Invalid request (request recognized, invalid request data)
- *Error: integer, error code 0x00000002: Invalid sensor number
- ErrorMsg: string, error details

There may also be error-dependent extra keys. Other error codes may be returned. 0x00000003 is handled as an unknown request. Codes with higher numbers refer to internal errors; contact Vaisala for more information.

12. **Principle of measurement**

The Vaisala K-PATENTS® in-line refractometer determines the refractive index n_D of the process solution. It measures the critical angle of refraction using a yellow LED light source with the same wavelength (589 nm) as the sodium D line (hence n_D). Light from the light source (L) in the following figure is directed to the interface between the prism (P) and the process medium (S). Two of the prism surfaces (M) act as mirrors bending the light rays so that they meet the interface at different angles.
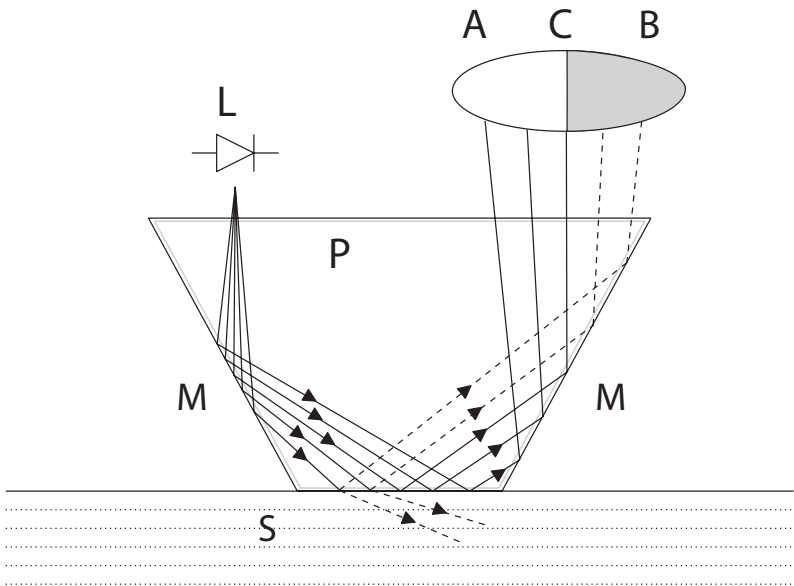
Figure 27   Refractometer principle

The reflected rays of light form an image (ACB), where (C) is the position of the critical angle ray. The rays at (A) are totally internally reflected at the process interface, the rays at (B) are partially reflected and partially refracted into the process solution. In this way the optical image is divided into a light area (A) and a dark area (B). The position of the shadow edge (C) indicates the value of the critical angle. The refractive index n_D can then be determined from this position.

The refractive index n_D changes with the process solution concentration and temperature. For most solutions the refractive index increases when the concentration increases. At higher temperatures the refractive index is smaller than at lower temperatures. From this follows that the optical image changes with the process solution concentration as shown in the following figure. The color of the solution, gas bubbles or undissolved particles do not affect the position of the shadow edge (C).

A   C B   A C   B
Low concentration   High concentration
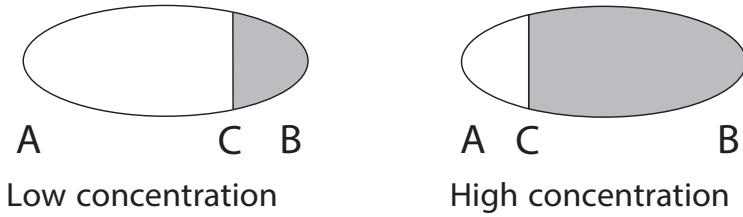
Figure 28   Optical images

The position of the shadow edge is measured digitally using a CCD element and is converted to a refractive index value n_D by a processor inside the instrument. This value is used together with the measured process temperature to calculate the concentration.
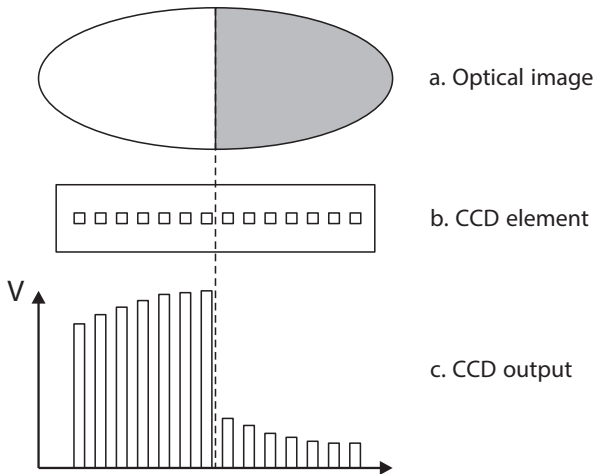

a. Optical image

b. CCD element

c. CCD output

Figure 29   Optical image detection

Appendix A. EU declaration of conformity

VAISALA   **2019-09-01H/JAMO**   1 (1)

EU DECLARATION OF CONFORMITY

Manufacturer: Vaisala Oyj

Mail address: P.O. Box 26, FI-00421 Helsinki, Finland
Street Address: Vanha Nurmijärventie 21, Vantaa, Finland

This declaration of conformity is issued under the sole responsibility of the manufacturer.

Object of the declaration:

K-Patents Process Refractometer PR-33 series

The object of the declaration described above is in conformity with Directives:

RoHS Directive (2011/65/EU)
EMC Directive (2014/30/EU)

The conformity is declared using the following standards:

EN 50581:2012 Technical documentation for the assessment of electrical and electronic products with respect to the restriction of hazardous substances

EN 61010-1:2010 Safety requirements for electrical equipment for measurement, control and laboratory use – Part 1: General requirements

EN 61326-1:2013 Electrical equipment for measurement, control and laboratory use – EMC requirements – intended for use in industrial locations

Signed for and on behalf of Vaisala Oyj, in Vantaa, on 1st September 2019

Jukka Lyömiö
Standards and Approvals Manager

Appendix B. Refractometer field calibration form

Fill in this form and email it to helpdesk@vaisala.com or to your local service representative.

Refractometer serial no:

Refractometer model:

Customer:

Address:

Email:

Sample description:

Solvent (water/other):

Laboratory method:

Date:

Data collected by:

Sample no.	LAB%	CALC	T	n_D	CONC

Warranty

For standard warranty terms and conditions, see www.vaisala.com/warranty.
Please observe that any such warranty may not be valid in case of damage due to normal wear and tear, exceptional operating conditions, negligent handling or installation, or unauthorized modifications. Please see the applicable supply contract or Conditions of Sale for details of the warranty for each product.

Technical support

Contact Vaisala technical support at helpdesk@vaisala.com. Provide at least the following supporting information as applicable:
• Product name, model, and serial number
• Software/Firmware version
• Name and location of the installation site
• Name and contact information of a technical person who can provide further information on the problem

For more information, see www.vaisala.com/support.

Recycling

Recycle all applicable material.

Follow the statutory regulations for disposing of the product and packaging.

VAISALA