NIR Analyzers and Accessories

Viscous Liquid Sampler

User Guide

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WARNING Avoid an explosion or fire hazard. This instrument or accessory is not designed for use in an explosive atmosphere.

Viscous Liquid Sampler

The Viscous Liquid Sampler (VLS) can be used with any Thermo Scientific integrating sphere sampling module or accessory to collect near-infrared transflection spectra from viscous liquids. The apparatus is designed to measure thick, concentrated samples such as honey and syrup through clear plastic packaging with little mess and easy cleanup.

Contents

Before reading this manual and using the VLS, please read the documentation that came with your integrating sphere sampling module or accessory.

Conventions Used in This Manual

This manual uses these conventions for providing safety and other special information:

CAUTION Indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

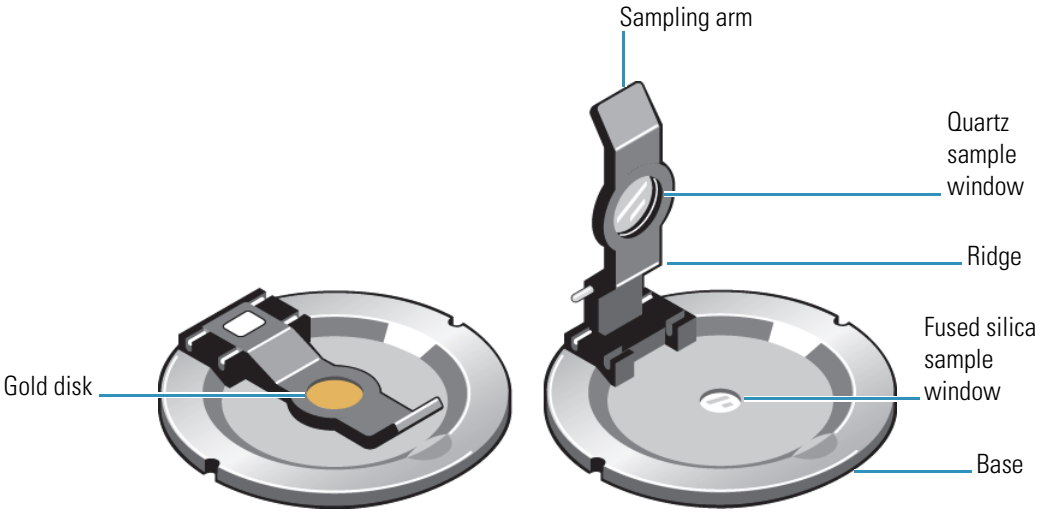
NOTICE Follow instructions with this label to avoid damaging the system hardware or losing data.

Note Contains helpful supplementary information.

Tip Provides helpful information that can make a task easier.

Important Features

Figure 1. Viscous Liquid Sampler features



The VLS has these important features:

- The **sample window** in the VLS **base** is fused silica.
- The **sampling arm** has a diffusely reflective **gold disk** mounted behind a **quartz sample window**. The disk reflects the near-infrared energy back through the sample to the detector for the integrating sphere.
- The **ridge** on the underside of the sampling arm determines the sample thickness. The arm is available in three sample thickness configurations (0.5 mm, 1.0 mm, and 2.0 mm) to accommodate a range of sample types and concentrations.

Operating Precautions

Before using the VLS, read these operating precautions to avoid damaging your equipment.

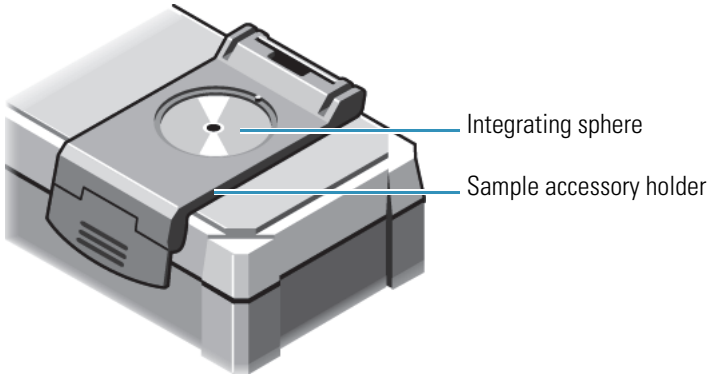
- **Do not touch the sample windows**. Oils from your fingers will affect the spectral data.
- **Do not pour liquids directly onto the windows**. The VLS is designed to sample viscous liquids that are contained in plastic bags.

- **Do not allow the sample windows come into contact with harsh solvents.** Although the seals around the windows are chemically resistant, harsh solvents may weaken them. To clean the windows, follow the instructions in "Cleaning the VLS."
- **Protect the sample windows from harsh, abrasive substances.** Although the window materials are durable, harsh, abrasive substances may scratch or crack them, affecting the spectral data.

Installation

The VLS fits in the sample accessory holder for the integrating sphere. The sample accessory holder may be included with your integrating sphere or it may be purchased separately. For more information, contact our representative in your area or use the information at the beginning of this document to contact us.

Figure 2. Sample accessory holder for the integrating sphere



Before you Begin

- **Make sure the sample windows are clean**, including the window on the integrating sphere and the two windows on the VLS. Refer to the user documentation for your integrating sphere and the "Cleaning the VLS" section of this guide for instructions and recommended cleaning agents.
- **Install the sample accessory holder** on the integrating sphere. For installation instructions, refer to the documentation that came with your integrating sphere.

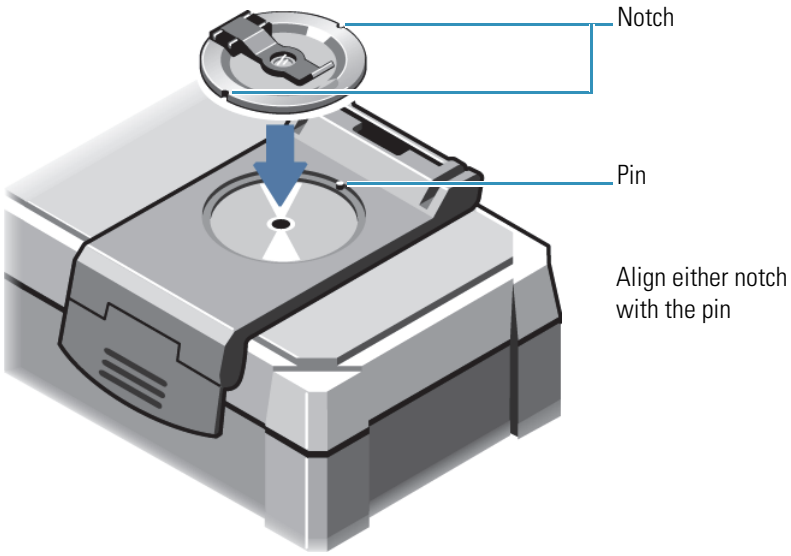
Installing the VLS

CAUTION Avoid eye hazard. Do not stare at the beam that exits the integrating sphere sampling window when the spectrometer is powered on and the Integrating Sphere beam path is selected.

❖ To install the VLS

Place the VLS in the sample accessory holder. Align one of the notches in the VLS base with the pin in the sample holder base to secure it. You can use either notch. One notch aligns the VLS with the sampling arm hinge on the left side; the other aligns it with the hinge on the right.

Figure 3. Installing the VLS



Inserting the Sampling Arm

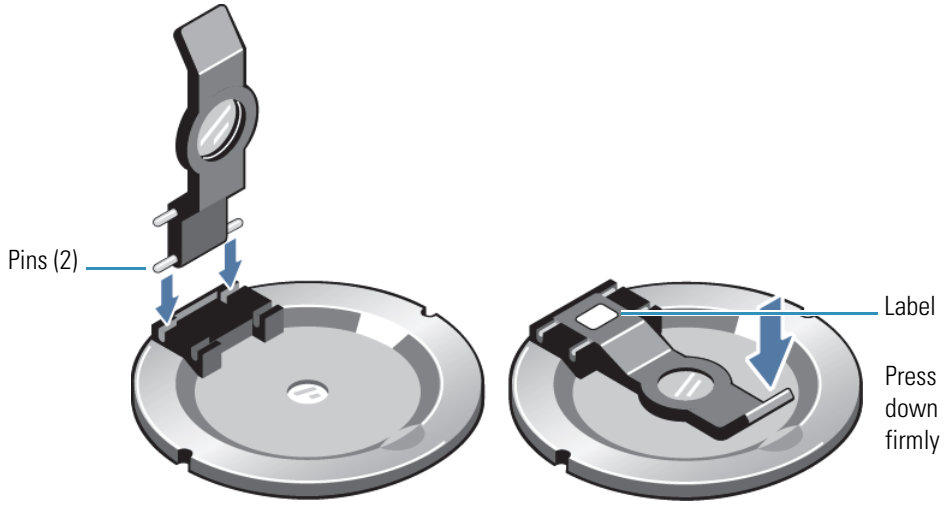
❖ To insert the sampling arm

1. Select a sampling arm that is appropriate for the sample material or concentration (see "Important Features" and "Compatible Samples" for details).

NOTICE Be careful not to touch the sample window when handling the VLS sampling arm.

2. Position the two bottom pins on the sampling arm above the long slot on the VLS base and press firmly on the arm so the pins snap into the slot.

Figure 4. Inserting the sampling arm



To engage the sampling arm (with or without a sample in place), press down firmly on the upturned end until the arm locks in the sampling position. When the arm is in this position, the distance between the two sample windows is fixed. The label attached to the sampling arm indicates the distance, or sample thickness, value for the experiment.

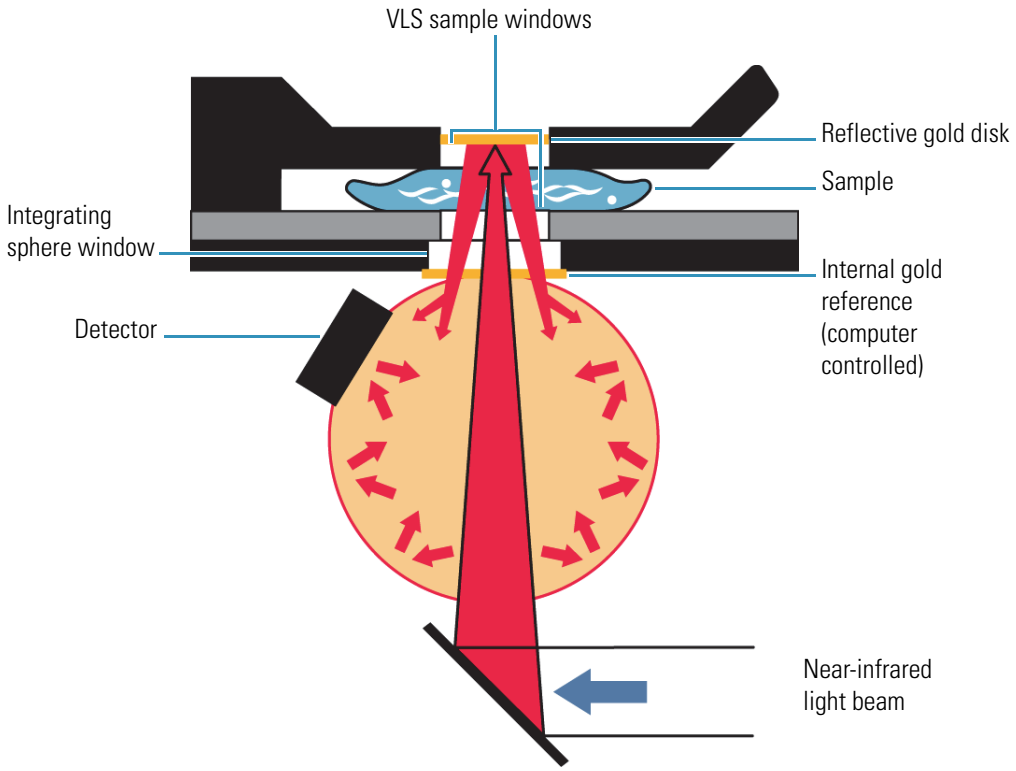
To raise the sampling arm, grasp the upturned end and pull it up firmly.

Note To remove the sampling arm from the base, first raise the sampling arm as described above. Then hold the end of the sampling arm and firmly press it right or left to free the pins from the slot.

Understanding Transflection Sampling

The VLS can be used for transflection experiments which measure the percentage of light reflected through a sample. During transflection data collection, the instrument directs the near-infrared beam into the integrating sphere, through the integrating sphere sample window and the sample window in the VLS base and into the sample, which absorbs specific frequencies. The light that passes through the sample is reflected off the gold disk, which sends the beam back through the sample to the detector in the integrating sphere.

Figure 5. Transflection sampling using the VLS



Compatible Samples

The VLS is designed to measure homogeneous viscous liquids. The samples can be light or dark in color but the material should be translucent (suspended or opaque liquids and powders reflect rather than transmit light and should be measured another way).

Samples are analyzed through clear, flexible packaging materials such as a clear polyethylene or other plastic bag. The sampling mechanism produces consistent compression with a fixed pathlength, which improves sampling accuracy and repeatability.

Sample Pathlength

The sample pathlength is determined by the sample thickness, which is defined by the size of the ridge on the sampling arm. For transflection analysis, the near-infrared energy passes through the sample and is then reflected through the sample a second time before it continues to the detector. As a result, the sample pathlength is equal to twice the sample thickness value.

The table below shows the sample thickness and pathlength value for each sampling arm.

Table 1. Available pathlength values for the VLS

Sampling arm	Sample thickness (mm)	Sample pathlength (mm)
0.5	0.5	1.0
1.0	1.0	2.0
2.0	2.0	4.0

As a general rule, use a sampling arm that produces a longer sample pathlength when measuring trace components in a sample mixture and samples that are light in color. If you want to measure dark or concentrated samples or bulk components in a sample mixture, choose an arm that gives a shorter pathlength.

Preparing Samples

The VLS is designed to measure samples through clear, flexible packaging materials such as a clear polyethylene or other plastic bag. Special 1x3 inch polyethylene bags are provided with the accessory for this purpose (contact Thermo Fisher Scientific to order more).

Start by filling the bag about one quarter full. Seal the bag, place it over the window in the VLS base and lower the sampling arm to test the volume. You need enough sample to adequately fill the space between the windows but not so much that the arm won't close.

Press firmly on the arm until it locks into the sampling position. If the arm will not lock, remove some sample from the bag and try again.

Note To improve accuracy and repeatability for repeated sampling with the VLS, fill each bag with the same amount of sample.

Collecting Data

You should be familiar with collecting data with your integrating sphere before you attempt data collection with the VLS. For more information, refer to the user guide that came with your integrating sphere.

Collecting Background Data

You can collect a background spectrum using the integrating sphere's internal gold reference or with an external reference, that is, through the VLS sample window. Refer to the user documentation that came with your integrating sphere for information about collecting data with the internal or an external reference.

When collecting a background with the internal reference, the software automatically moves the reference into the beam, collects the background data, and then moves the reference out of the beam. Before you collect a background spectrum through the VLS sample windows, remove any sample from the VLS, make sure the windows are clean (see "Cleaning the VLS") and then lower the VLS arm to the sampling position.

Collecting Sample Data

Prepare the sample as described in "Preparing Samples." To load the sample, center the bag that contains the sample over the VLS window. Press firmly on the arm until it locks in the sampling position. To improve the accuracy and repeatability of your data, wait 30 seconds before you start collecting the sample data to allow the liquid time to stabilize.

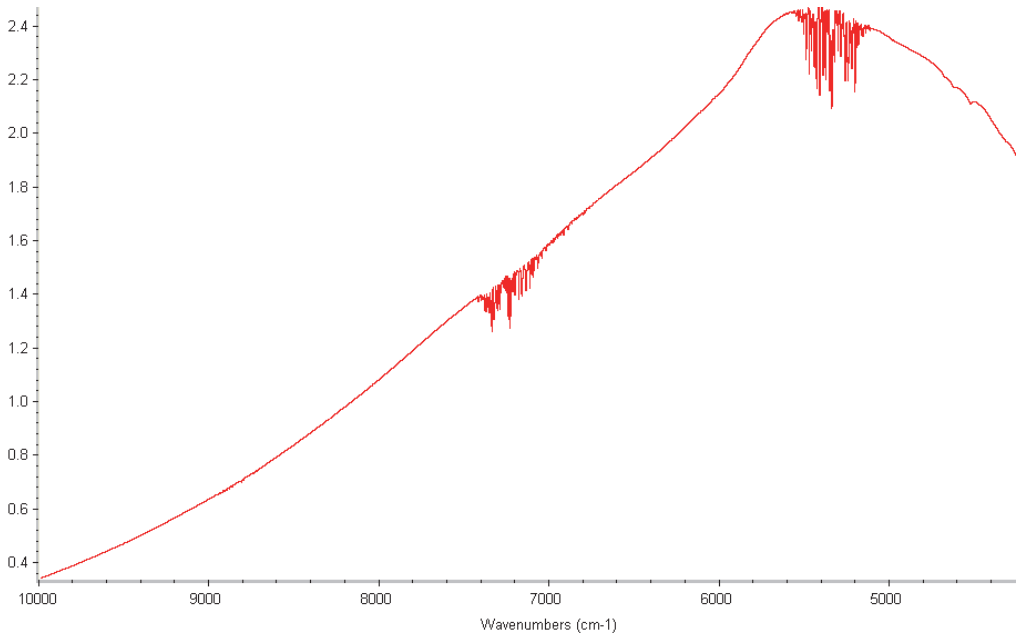
If the VLS windows collected any residue from the sample, follow the instructions for cleaning them in "Cleaning the VLS."

Typical Spectra

FT-NIR spectra produced by transflection can have unique characteristics. The spectra shown in this section are only examples of the kinds of results you may obtain. The actual spectra produced from your experiments may vary greatly, depending on the sample material and preparation.

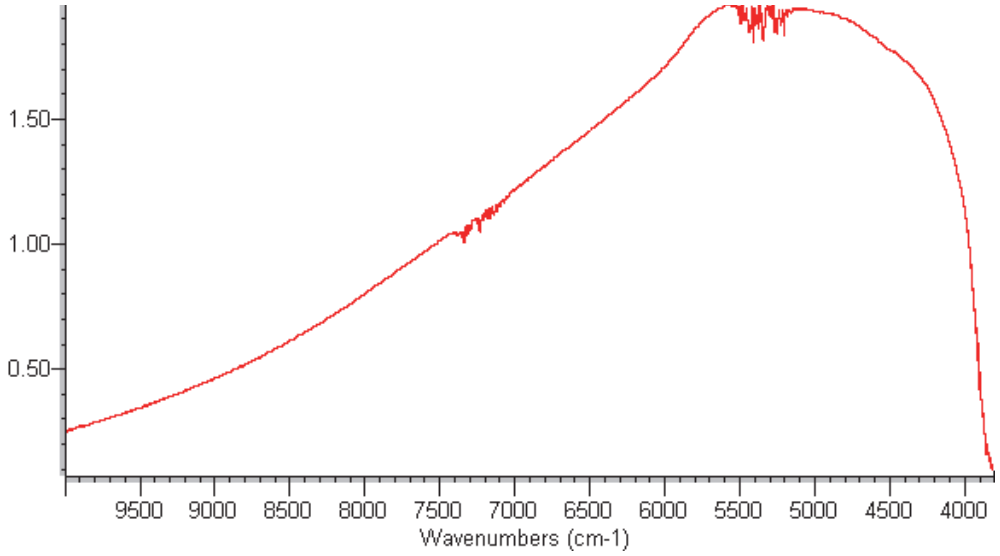
The illustration below shows a typical background spectrum taken through the VLS windows.

Figure 6. Typical background spectrum taken through the VLS windows



For convenience, you may prefer to take backgrounds with the integrating sphere's internal gold reference. A typical gold reference background spectrum should resemble the following:

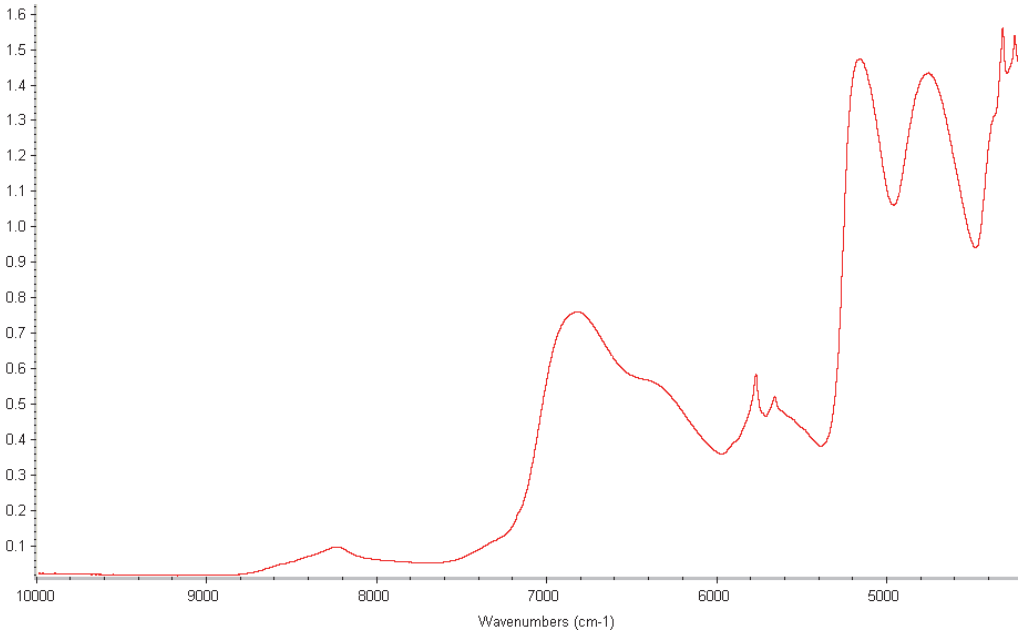
Figure 7. Typical gold reference background spectrum taken with the integrating sphere



If your background spectrum is not similar to one of the above spectra, or if it is atypical from previous background spectra, see "Troubleshooting."

The following is representative of a typical spectrum of a viscous liquid taken using an integrating sphere and the VLS.

Figure 8. Spectrum of sweetener taken with the VLS



Troubleshooting

If you encounter a problem with the VLS, before running any other diagnostics or deeming backgrounds or samples as "bad," you may first want to review the items discussed here.

Problems with Sample Spectra

If a sample spectrum you collected is significantly different from previously-collected samples or from the typical spectra described in this document, the problem may be one of these:

- Make sure the sample is centered over the VLS sample window and the arm is locked in the sampling position when collecting the sample spectrum.
- Make sure the software is set up to run an integrating sphere sample. Refer to your software and integrating sphere user documentation for more information.
- After you lock the VLS arm in the sampling position, always wait the same length of time for the sample material to stabilize before collecting data. Waiting 30 seconds is usually sufficient but very thick samples may need more time to stabilize. After you determine the optimum delay for your samples, be consistent from one sample to the next.

Problems with Background Spectra

If a background spectrum you collected is atypical from previously-collected backgrounds or from the typical spectra described in this document, the problem may be one of the following:

- If collecting a background through the VLS, make sure the:
  - VLS sampling arm is locked in the sampling position with no sample in place.
  - Integrating sphere and VLS sample windows are clean. Follow the instructions in "Cleaning the VLS" and in the user guide for your integrating sphere.

Cleaning the VLS

If residue accumulates on the VLS sampling arm or base, wipe it with a clean, soft cloth. You can dampen the cloth with a mild soap solution if necessary. Dry it with a clean, dry cloth.

Wipe both sides of the sample windows with a clean, soft lint-free cloth, a white paper napkin or a cotton swab. You can dampened the wipe with distilled water or isopropyl alcohol if necessary. Dry the windows with another clean wipe or a jet of air or allow them to air dry.

NOTICE Some chemicals including, acetone, chlorine, fluorine, and amyl alcohol can attack the epoxy seal around the sample windows. Do not allow these chemicals to come into contact with the windows.

Store the VLS in a dust-free enclosure when it is not in use.