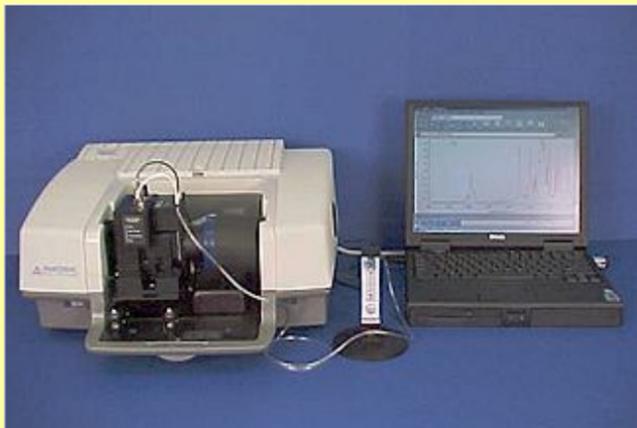


PAC 300 Photoacoustic



**Thermo Scientific FT-IR spectrometer with
PAC 300 Photoacoustic E.S.P. accessory installed**

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What you will learn

This tutorial introduces the PAC 300 Photoacoustic E.S.P. accessory from Thermo Fisher Scientific. The PAC 300 can be used with Thermo Scientific's Nicolet™ Series FT-IR spectrometers.

Run this tutorial to learn the basics of photoacoustic sampling with your spectrometer. It explains step-by-step how to install the PAC 300 in the spectrometer sample compartment and how to use the accessory to analyze solids, semi-solids, and powders, including samples with layers or compositional gradients. You will also learn how to properly maintain and store the accessory.

Exit

Contents

Thermo
SCIENTIFIC

Print

Next

Contents

Click a unit name to move to the beginning of that unit.

Unit _____



PAC 300 Photoacoustic E.S.P.



Product Features



Product Specifications



Product Components



Operating Precautions



OMNIC Software



System Performance

Unit _____



Installing the PAC 300



Measuring the Background



Installing a Sample



Measuring the Sample



Cleaning Up



Turning Off the PAC 300



Removing the PAC 300



Where to Go Next



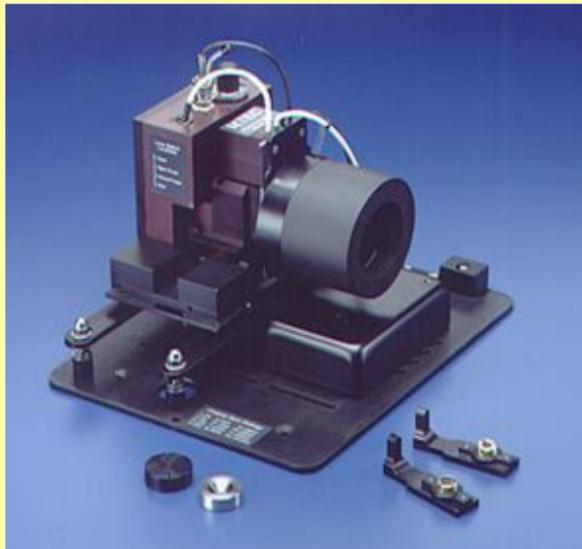
Home

Thermo
SCIENTIFIC



Return

Product Features



PAC 300 Photoacoustic E.S.P. accessory

PAC 300 features

The PAC 300 features:

- Purged sample chamber.
- Wide spectral range (UV to far-IR, depending on window material).
- Less than 10 seconds per sample for qualitative analysis.
- Handles solid, semi-solid, powder and liquid samples with little preparation.
- Allows variable sampling depth.
- Step-scan mode.
- Optional Multi-Sampler (for sequential DRIFTS, PAS, and transmission sampling).
- Optional Micro-Sampler (for analyzing single particles and fibers).



Home



Contents

Thermo
SCIENTIFIC

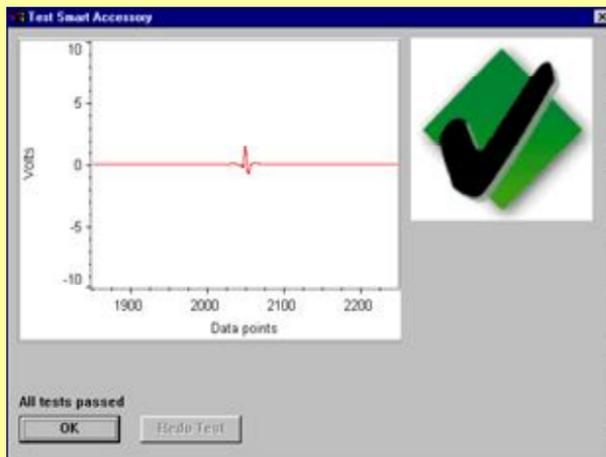


Back



Next

Product Features



Routine performance checks let you focus on your work—not on the instrument

E.S.P. accessory features

The PAC 300 is part of Thermo Scientific's family of E.S.P. accessories for FT-IR spectrometers. All E.S.P. accessories offer the following features:

- Standard baseplate mounted.
- Automatic recognition.
- Automatic experiment setup.
- Automatic performance checking.
- Multi-media tutorials.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Product Specifications



PAC 300 specifications

The PAC 300 Photoacoustic E.S.P. accessory with the standard KBr window features:

Spectral range: 20,000 - 400 cm^{-1}

Sampling depth: Can range from less than 1 micrometer to over 100 micrometers

Useful pH range: 1 - 14

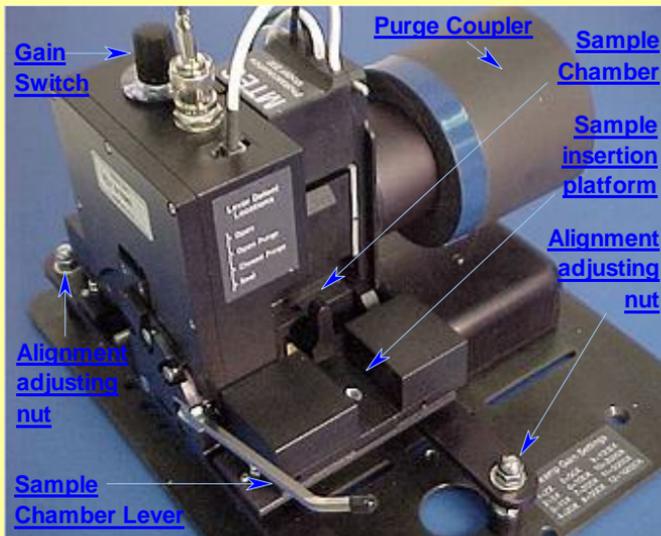
Sampling area: 5 mm diameter

Maximum sample size: 10 mm diameter X 8 mm high

[Click here](#) for information on optional window materials.

Product Components

For information on a PAC 300 component, click the component name in the picture below.



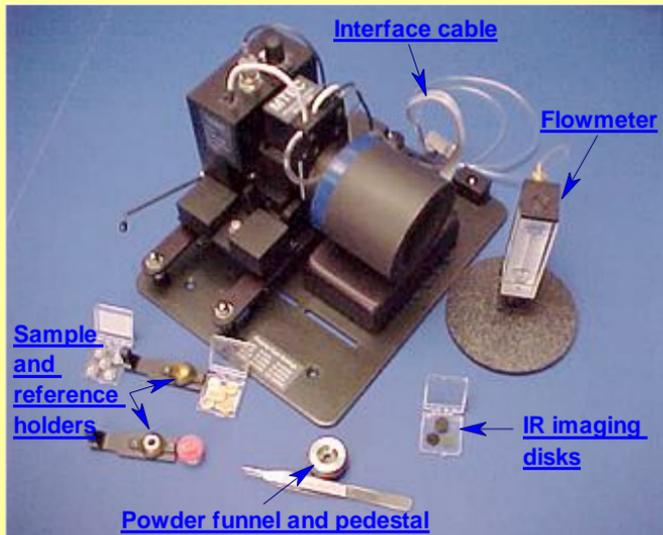
PAC 300 Photoacoustic E.S.P. accessory

Main components of the PAC 300

The PAC 300 uses pressure sensing technology to gather information about a sample material. This unique spectroscopy technique can be applied to many kinds of samples, including solids, semi-solids, powders and liquids. The beam from the IR spectrometer interacts with the sample material the same way it does in an IR transmission analysis. However, the PAC 300 measures heat-induced pressure oscillations produced by the characteristic absorptions of the sample. The sample is placed in a sample holder or an optional sampling head which is then inserted into the sample chamber for analysis. The sample chamber lever seals the sample in the chamber.

Product Components

For information on a PAC 300 component, click the component name in the picture below.



PAC 300 standard components

Accessories for the PAC 300

The PAC 300 includes sample and reference holders for running solid, semi-solid, powder and liquid samples. Optional [Multi-Sampler](#) and [Micro-Sampler](#) sampling heads are also available. The sample holder or head determines how the PAC 300 senses the interaction between the infrared beam and the sample.

Additional cups, spacers, pedestal, funnel and other tools are provided with the PAC 300 to make loading samples quick and easy. A flowmeter is also included for monitoring the flow rate of the helium gas used to purge the sample chamber.



Home

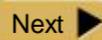


Contents

Thermo
SCIENTIFIC



Back



Next

Operating Precautions



Helium
flowmeter



Caution

Always place the sample chamber lever in the sealed position while you open the helium tank valve or adjust the gas flow. This protects the microphone from damage if a gas surge occurs.



Purging the sample chamber

The PAC 300 sample chamber must be purged with helium. The helium is needed to conduct the heat oscillations produced by the sample's characteristic absorptions of infrared energy. The heat oscillations cause thermal expansion-driven pressure oscillations, which can be read by the photoacoustic detector.

Accidental gas surges can damage the microphone in the photoacoustic detector. To protect the microphone from damage, set the sample chamber lever to Seal before opening the helium regulator valve to purge the sample chamber or adjusting the helium flow.



Home

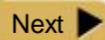


Contents

Thermo
SCIENTIFIC



Back



Next

Operating Precautions



Caution

Always set the sample chamber lever to Sealed when you finish running samples for the day. This protects the photoacoustic detector and prevents moisture from entering the sample chamber.



Protecting the photoacoustic detector

To avoid contaminating the detector when analyzing fine powder samples, reduce or shut off the helium gas flow when moving these samples into or out of the sampling area. See the section on installing powder samples in this tutorial for recommended techniques.

When you are finished running samples with the PAC 300, set the sample chamber lever to Open Purge and wait a minute or two for the sample chamber to completely fill with helium. Then set the lever to Sealed. This protects the photoacoustic detector and prevents moisture from entering the sample chamber.



Home



Contents

Thermo
SCIENTIFIC

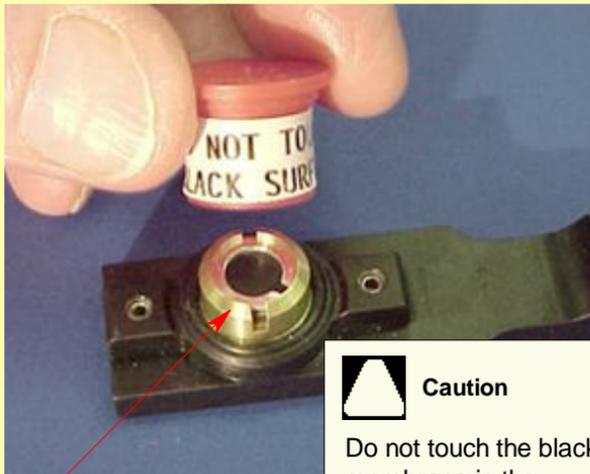


Back



Next

Operating Precautions



Carbon black reference sample



Caution

Do not touch the black membrane in the carbon black reference or blow on it with a gas jet. The membrane is delicate and may break.

Handling the carbon black reference

The carbon black reference sample is used to run the accessory performance test described later in this tutorial. The reference has a delicate, carbon black-coated membrane to absorb infrared radiation and efficiently transfer the absorption-created heat to the sample chamber atmosphere.

Do not touch the membrane or blow on it with a gas jet. The membrane's requirements for low thermal mass and high signal generation efficiency make it very thin and, thus, vulnerable to damage.

When you finish using the carbon black reference, remove it from the PAC 300 and replace the cap.



Home



Contents

Thermo
SCIENTIFIC



Back



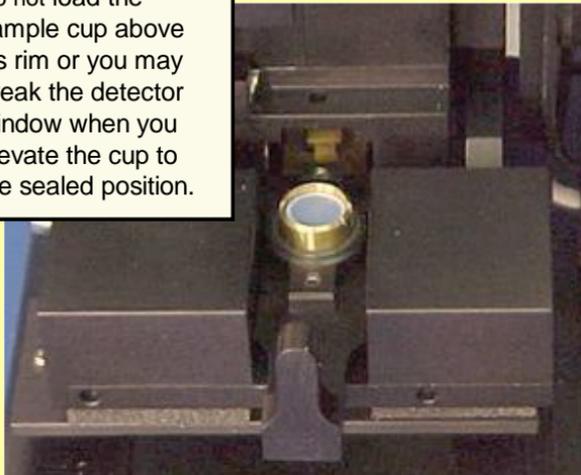
Next

Operating Precautions



Caution

Do not load the sample cup above its rim or you may break the detector window when you elevate the cup to the sealed position.



Sample cup with sample positioned approximately 1 mm below the cup's rim.

Loading a sample cup

The sample cup has an assortment of stainless-steel inner cups and brass and aluminum spacers to displace extra gas volume. Do not load the sample cup above its rim.

If you overfill the sample cup, it may break the detector window when you elevate the cup to the sealed position.



Home

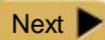


Contents

Thermo
SCIENTIFIC



Back



Next

OMNIC Software

How to start the OMNIC software:

The methods you can use to start OMNIC depend on the version of Windows you are using.

[Click here](#) to learn how to start OMNIC.

For information on other methods of starting applications and more detailed instructions on using Windows features, see your Windows documentation.

After you start OMNIC, the [OMNIC window](#) appears on the display.

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Windows is a trademark of Microsoft Corporation.

Starting the OMNIC software

Thermo Scientific's OMNIC™ Software is an advanced software package for FT-IR spectroscopy that you can run using Windows®.

With OMNIC you can perform a wide range of tasks, from collecting infrared spectra to performing quantitative analysis. The commands needed to collect and process spectra are conveniently arranged in menus and can also be entered from the keyboard.

For instructions on starting OMNIC, refer to the box at the left.



Home



Contents

Thermo
SCIENTIFIC

◀ Back

Next ▶

System Performance



Thermo Scientific FT-IR spectrometer

Checking spectrometer performance

Run the performance test on your spectrometer at least once a week to verify your system's performance without an accessory installed.

See the documentation that came with your spectrometer for instructions or [click here](#) to learn how to check individual spectrometer components.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to install the PAC 300:

1. Prepare the spectrometer for installation of the PAC 300 sampling accessory.

Make sure the spectrometer is [turned on](#).

If the sample compartment cover is attached to the spectrometer housing, [remove the cover](#).

If you have a Nicolet Series spectrometer, [install the side wall purge adapter](#) on the beam entry port (adapter not required for Magna-IR spectrometers).

If you were using any of the connectors at the back of the sample compartment, disconnect the cables.

[Remove the snap-in baseplate](#) or any other accessories installed inside the sample compartment.

Installing the PAC 300

If the spectrometer performance results are acceptable, follow the instructions at the left to install the PAC 300 E.S.P. accessory in the sample compartment of your spectrometer.

The accessory is mounted on an E.S.P. baseplate that fits in the spectrometer sample compartment. The optical components are mounted in a sealed unit, which connects to the purge system in your IR spectrometer.



Home

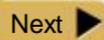


Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to install the PAC 300 (continued):

2. Insert the PAC 300 into the spectrometer sample compartment.

Hold the PAC 300 accessory on either side of the sample chamber. Rest the accessory on the floor of the spectrometer sample compartment, but don't lock the snap-in baseplate onto the alignment pins.



Steps to install the PAC 300

Prepare spectrometer

Insert PAC 300

Connect interface cable

Position baseplate on pins

Secure purge coupler



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to install the PAC 300 (continued):

3. Connect the interface cable.

If you are installing the PAC 300 in a Nicolet Series spectrometer, use the interface cable with the 15-pin D connector on one end and a plastic clip connector on the other end.

Connect one end of the interface cable to the connector in the side wall of the spectrometer sample compartment. Attach the other end of the cable to the pre-amplifier on the PAC 300 baseplate as shown in the picture at the right.



Steps to install the PAC 300

Prepare spectrometer

Insert PAC 300

Connect interface cable

Position baseplate on pins

Secure purge coupler



Home

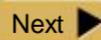


Contents

Thermo
SCIENTIFIC



Back

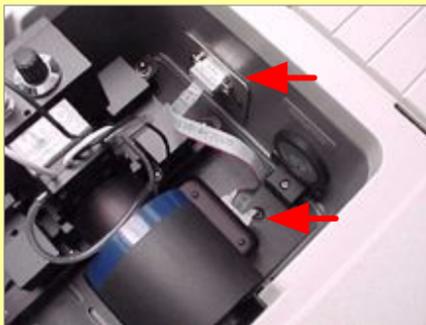


Next

Installing the PAC 300

How to install the PAC 300 (continued):

If you are installing the PAC 300 in a Magna-IR spectrometer, use the interface cable with the 9-pin D connector on one end and plastic clip connector on the other.



4. Adjust the PAC 300 baseplate so it fits snugly over the alignment pins on the sample compartment floor.

Steps to install the PAC 300

Prepare spectrometer

Insert PAC 300

Connect interface cable

Position baseplate on pins

Secure purge coupler



Home



Contents

Thermo
SCIENTIFIC



Back

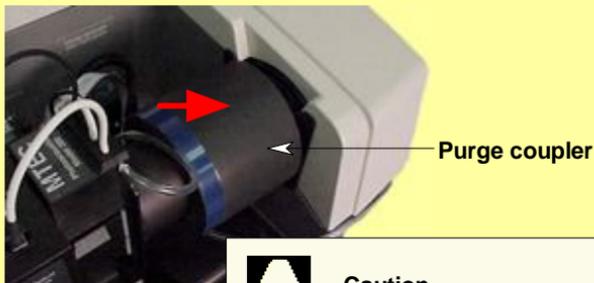


Next

Installing the PAC 300

How to install the PAC 300 (continued):

- Slide the purge coupler so that it fits snugly against the side wall of the spectrometer sample compartment.



Caution

Make sure the plastic shipping cover and desiccant packet have been removed from the purge coupler tube before connecting the tube to the spectrometer.

Steps to install the PAC 300

Prepare spectrometer

Insert PAC 300

Connect interface cable

Position baseplate on pins

Secure purge coupler



Home

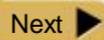


Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to connect the helium purge lines:

1. Make sure the helium gas valve and regulator are closed and the PAC 300 sample chamber is sealed by moving the lever all the way down.



2. Use a length of 1/4-inch inner diameter plastic tubing to connect your helium gas source to the flowmeter inlet (bottom connector).
3. Attach one end of the small diameter purge tube (provided) to the flowmeter outlet (top connector).



Connecting the helium purge lines

The PAC 300 requires a source of helium to purge the sample chamber. The helium environment is needed to generate an adequate signal for the photoacoustic detector. The helium must be zero grade or higher purity so it is free of moisture.

Connect the flowmeter provided with the accessory to a pressure regulator on the helium gas cylinder. Connect the flowmeter outlet to the PAC 300 using the tubing provided. See the box at the left for instructions. DO NOT install an on/off valve between the regulator and the PAC 300.

See Purging the Sample Chamber for instructions on setting the gas flow.



Home



Contents

Thermo
SCIENTIFIC



Back

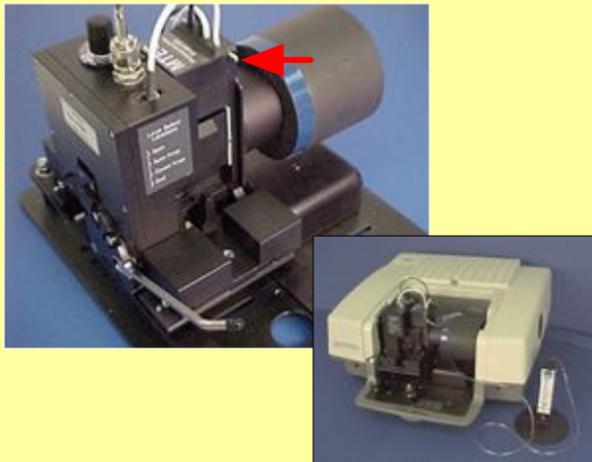


Next

Installing the PAC 300

How to connect the helium purge lines (continued):

- Slide the other end of the purge tube over the stainless-steel purge connector on the PAC 300 accessory.



Connecting the helium purge lines

Seal sample chamber

Connect flowmeter to helium source

Connect flowmeter to PAC 300



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to purge the PAC 300 sample chamber:

1. Make sure the PAC 300 sample chamber is sealed by lowering the sample chamber lever.



Caution

Always place the sample chamber lever in the sealed position while you open the helium tank valve or adjust the gas flow. This protects the microphone from damage due to an accidental gas surge.

2. Make sure the regulator on the helium gas source is closed.

Purging the PAC 300 sample chamber

After you install the PAC 300 accessory for the first time, the accessory must be purged for two hours to establish a helium atmosphere in the microphone's capillary system. To purge the sample chamber, set the sample chamber lever to Seal and then adjust the helium flow to 5 cc/s. Use the pressure regulator on the helium cylinder to regulate the gas flow. Do not install an on/off valve between the regulator and the PAC 300. Once the flow has stabilized, move the sample chamber lever to Closed Purge. See the box at the left for instructions.

Repeat the two hour purge if the sample chamber was left unsealed (not recommended).



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to purge the PAC 300 sample chamber (continued):

3. Open the main valve on the helium gas cylinder.
4. Gradually open the regulator valve until the flowmeter reads approximately 5 cc per second.
5. Set the sample chamber lever to Closed Purge.
6. Wait 2 hours before you begin collecting sample data.

This allows the helium to flow through the sample chamber and fill the microphone's capillary system.

7. When two hours has passed, set the sample chamber lever to Seal.

Purging the PAC 300 sample chamber

Set sample chamber lever to Seal

Close helium regulator

Open main helium valve

Gradually open helium regulator until flow is approximately 5 cc per second

Set sample chamber lever to Closed Purge

Wait 2 hours

Set sample chamber lever to Seal



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to install the carbon black reference:

1. Set the PAC 300 Gain control to 3.



2. Set the helium flow rate to 10 cc/s.



3. Set the sample chamber lever to Open.



Installing the carbon black reference

The carbon black reference sample is used to check the PAC 300 accessory's performance. The carbon black reference must be installed in the PAC 300 sampling area before you start the performance test. Follow the instructions at the left to install the carbon black reference sample in the PAC 300 sampling area.

Use the standard PAC 300 configuration (without the optional multi- or micro-sampler) to run the performance test.



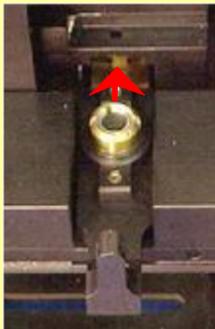
Thermo
SCIENTIFIC



Installing the PAC 300

How to install the carbon black reference (continued):

4. Place the carbon black reference sample on the sample insertion platform and slide it into the PAC 300 as far as it will go.



5. Set the sample chamber lever to Seal.

Installing the carbon black reference

Set gain

Set helium flow rate

Set sample chamber lever to Open

Install carbon black reference

Set sample chamber lever to Seal

Installing the PAC 300

How to select the default experiment:

After you install an E.S.P. accessory, its name and the title of its associated experiment are displayed in a dialog box.



Opening an experiment

When you install an E.S.P. accessory, the system automatically opens the experiment files that are associated with the accessory.

Each experiment file contains a complete set of parameters, which have already been optimized for collecting data with the accessory. There is no need for you to set the parameters individually.



Home



Contents

Thermo
SCIENTIFIC



Back

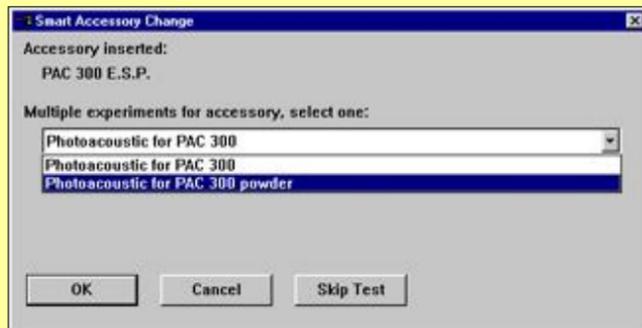


Next

Installing the PAC 300

How to select from a list of experiments:

If multiple experiments exist for the accessory, a list box is provided so you can select the experiment you want to open.



Click the arrow in the list box to see the titles of the experiments for the currently installed accessory.

Click a title to select the experiment.

Steps to open an experiment

Select the default experiment

Select from a list of experiments

Consider the background option



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

How to set the background option:

If you collected a background spectrum before installing your PAC 300 accessory, the following option appears in the Experiment Change dialog box.

Use current background

This option lets you associate the current background with the new experiment.

Leave the check box blank if you plan to collect a new background (recommended). The current background spectrum will be deleted.

When you are finished selecting the experiment and setting the background option, choose OK.

Steps to open an experiment

Select the default experiment

Select from a list of experiments

Consider the background option



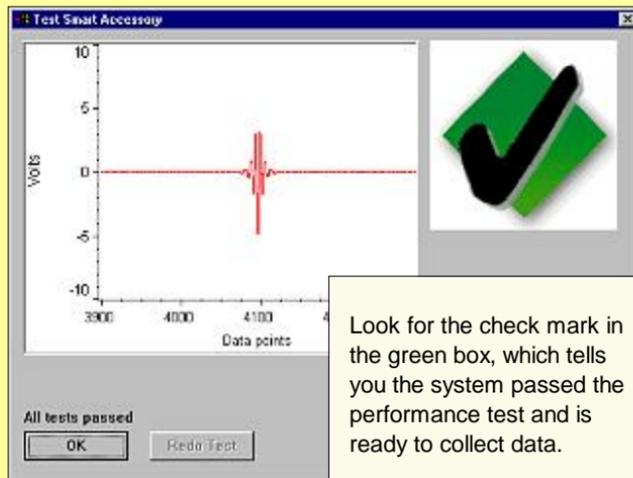
Thermo
SCIENTIFIC



Installing the PAC 300

Performance test passed:

The performance test starts automatically when you are finished opening an experiment. When the test is completed, a dialog box shows you the results.



Look for the check mark in the green box, which tells you the system passed the performance test and is ready to collect data.

Running the performance test

After an E.S.P. accessory is installed, the system automatically runs a series of diagnostic tests to evaluate its performance with the new accessory.

The [spectral quality checking](#) features of your OMNIC software specify the criteria used for the performance test.

The performance diagnostics continue working in the background while the accessory is installed, ensuring high quality spectra time after time.



Home

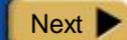


Contents

Thermo
SCIENTIFIC



Back

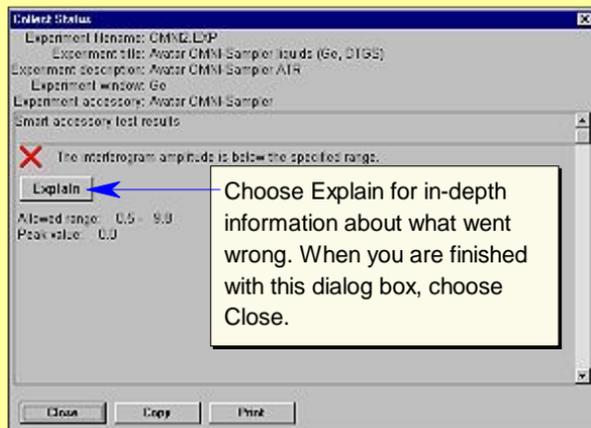


Next

Installing the PAC 300

Performance test failed:

If the system does not meet the performance criteria (it won't if you didn't install the carbon black reference sample), a message appears along with instructions on how to verify and fix the problem.



Steps to run the performance test

Performance test passed

Performance test failed

Rerun performance test



Home

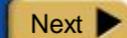


Contents

Thermo
SCIENTIFIC



Back

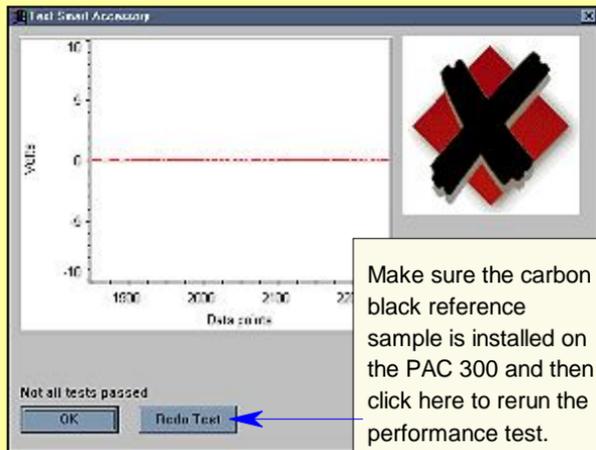


Next

Installing the PAC 300

How to rerun the performance test:

The next screen indicates that a problem occurred with the performance test and allows you to restart the test. Follow the instructions to correct the problem. Then choose Redo Test to rerun the performance test.



Steps to run the performance test

Performance test passed

Performance test failed

Rerun performance test



Home

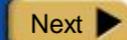


Contents

Thermo
SCIENTIFIC



Back



Next

Installing the PAC 300

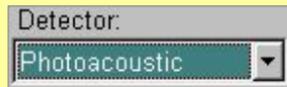
How to select the photoacoustic accessory:

1. From the OMNIC Collect menu, choose Experiment Setup.

The Experiment Setup dialog box is displayed.

2. Click the Bench tab.

3. Set the Detector list box to "Photoacoustic."



The image shows a screenshot of a software dialog box. At the top, the label "Detector:" is followed by a dropdown menu. The dropdown menu is open, and the word "Photoacoustic" is selected and displayed in the menu's text area. A small downward-pointing arrow is visible on the right side of the dropdown box.

4. Click the Save button at the bottom of the Experiment Setup dialog box.

Selecting the photoacoustic accessory

In order for the OMNIC software to operate properly, you must select the PAC 300 accessory in the current experiment the first time you use it. See the instructions at the left to learn how to set the Detector parameter in the current experiment.

You must repeat these steps for each new experiment you create for the PAC 300 Photoacoustic accessory.



Home



Contents

Thermo
SCIENTIFIC

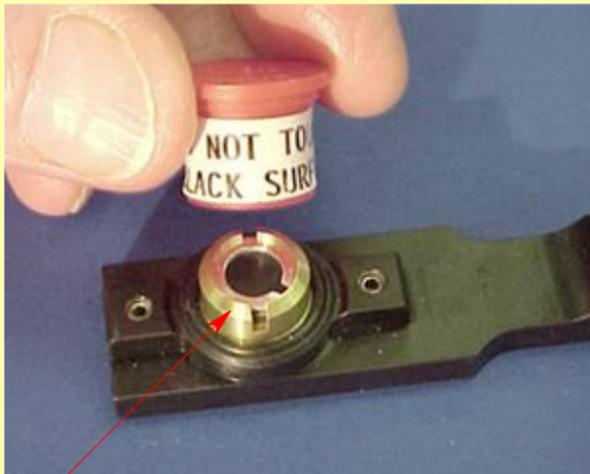


Back



Next

Measuring the Background



**Carbon black
reference sample**

What is a background?

A background spectrum is needed to process the sample data to an infrared spectrum. The background is a reference spectrum which accounts for the unique optics of the PAC 300 accessory and the spectrometer. Each sample spectrum is ratioed against a background so that the final spectrum is free of these features.

The carbon black reference sample is used for standard photoacoustic background measurements.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Measuring the Background

How to collect a background spectrum:

1. Set the helium flow rate to 10 cc/s.
2. If the helium gas flow was off, wait 1 minute to purge any air from the gas line.



3. Set the sample chamber lever to Open.
4. Place the carbon black reference sample on the sample insertion platform and slide it into the PAC 300 as far as it will go.



Collecting the background data

Use the carbon black reference to measure the background when collecting photoacoustic spectra. This is true whether you are collecting photoacoustic spectra with the standard configuration or with the optional multi- or micro-sampler. Backgrounds are typically measured with a mirror velocity of 0.16 cm/s and a PAC 300 gain setting of 3. [Click here](#) for tips on collecting background data using other experimental conditions.

Use the [Collect Background](#) command in the OMNIC Collect menu or the Collect Background button on the toolbar to start background data collection. See the box at the left for details.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Measuring the Background

How to collect a background spectrum (continued):

5. Seal the sample chamber by rotating the sample chamber lever directly to the sealed position.



6. If the spectrometer Velocity parameter in the current OMNIC experiment is set to 0.16 cm/s, set the PAC 300 amplifier Gain to 3.



[Click here](#) for recommended Gain settings when using other velocities. See your OMNIC on-line help for information on setting Velocity and other parameters in OMNIC experiments.

Steps to collect the background data

Set helium flow rate

Wait 1 minute

Set sample chamber lever to Open

Install carbon black reference sample

Set sample chamber lever to Seal

Set Gain

Collect background spectrum



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Measuring the Background

How to collect a background spectrum (continued):

7. Collect a background spectrum.

Use the Collect Background command in the OMNIC Collect menu or click the Collect Background button on the toolbar, if it is displayed.



Collect Background button

The quality of the sample spectra you collect will be enhanced if the sample spectra are processed with an appropriate background. [Click here](#) for tips on collecting representative backgrounds.

Steps to collect the background data

Set helium flow rate

Wait 1 minute

Set sample chamber lever to Open

Install carbon black reference sample

Set sample chamber lever to Seal

Set Gain

Collect background spectrum



Home



Contents

Thermo
SCIENTIFIC



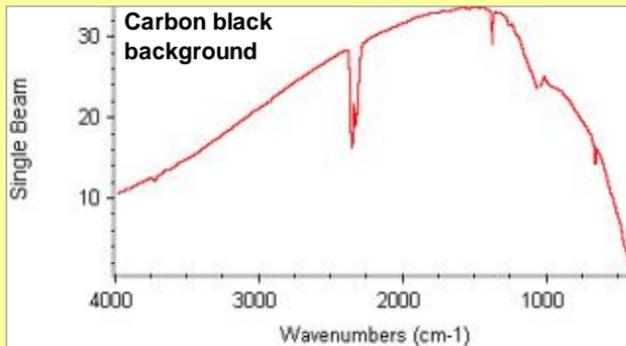
Back



Next

Measuring the Background

Here is what a background spectrum of the carbon black reference looks like.



**Photoacoustic background spectrum
collected from carbon black reference sample**

Background spectrum

The new background spectrum remains in memory and is selected as the current background. It will be used to process all of the sample spectra you collect until you replace it by collecting another background.



Home



Contents

Thermo
SCIENTIFIC



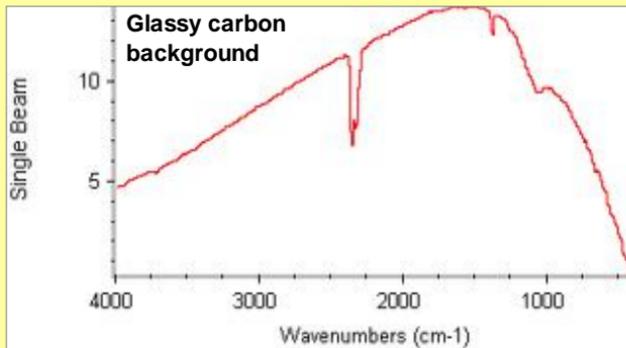
Back



Next

Measuring the Background

This illustration shows a background spectrum collected using the glassy carbon-phase reference. This reference is used for step-scan FT-IR measurements. (The step-scan feature is available on Magna-IR 860, Nexus 870 and Nicolet 8700 spectrometers only.)



Photoacoustic background spectrum collected using the glassy carbon-phase reference

Background spectrum

The new background spectrum remains in memory and is selected as the current background. It will be used to process all of the sample spectra you collect until you replace it by collecting another background.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Measuring the Background

When to collect a new background:

If any of the following is true, you should immediately remeasure the background and use the new background to process your sample data.

- You changed a component in your spectrometer or sampling accessory.
- You changed one of the Collect, Bench or Advanced settings in the selected experiment (except Gain, Final Format, Number Of Scans or Correction).
- You see a change in the amount of water or carbon dioxide bands in the IR spectra of your samples.
- You see an unexpected change in the spectral baseline.
- The quality of your spectral data is reduced (more noise or spurious peaks in the spectrum).

When to collect a new background

The background data used to process each sample measurement to an IR spectrum must be measured under exactly the same conditions as the sample. For best results, we recommend collecting a background spectrum before each sample.

If you use the same sampling technique and instrument settings to analyze all of your samples, you can use the same background to process multiple samples. However, we recommend collecting a new background at least once every two hours. If any of the conditions described at the left are true, remeasure the background immediately.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing a Sample



Installing a sample

When background collection is completed, you are ready to insert the sample. Samples should be positioned between 1 and 2 mm below the rim of the brass sample cup. The space allows a gas layer to form above the sample, which is needed to generate the photoacoustic signal. Use the spacers to fill excess cup volume that would reduce signal strength.

You can place samples directly in the brass sample cup or use one of the stainless-steel cups provided with the accessory. The stainless-steel cups come in two sizes to accommodate various sized samples. They also allow you to prepare additional samples while the previous sample is being analyzed.



Home



Contents

Thermo
SCIENTIFIC



Back



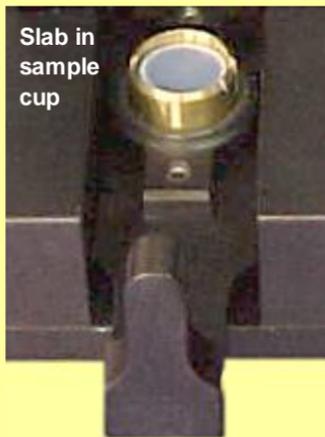
Next

Installing a Sample

How to install a solid sample:

1. Add spacers, if needed, to the brass sample cup.
2. Load the sample.

If you are analyzing a flat sample, place the sample directly on the spacers. Make sure you have enough spacers to raise the sample to the proper height (1 to 2 mm below the rim).



Installing a solid sample

If the sample is flat, you can usually load it directly in the brass sample cup. Add two or three of the spacers below the sample to raise it to the proper height (1 to 2 mm below the rim). When analyzing pellets, we recommend using a stainless-steel sample cup (you may need to use fewer spacers to keep the sample below the 1 mm maximum).

If the sample evolves water vapor when heated by the infrared beam, we recommend adding a magnesium perchlorate desiccant layer below the sample. The desiccant reduces vapor concentration in the sample chamber as samples outgas. Vapors produce interfering spectral peaks. See the box at the left for instructions.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing a Sample

How to install a solid sample (continued):

If the sample evolves vapor when heated, add a layer of desiccant below the sample as shown below.



- Fill a large stainless-steel sample cup with desiccant and place the desiccant in the bottom of the brass sample cup.

- Place a notched spacer over the desiccant.

- Place the sample in a cup and place the cup on top of the desiccant and spacer.



Steps to install a solid sample

Add spacers, if needed

Load sample

Adjust flow rate

Set sample chamber lever to Open

Insert sample holder

Set gain

Set sample chamber lever to Open
Purge, if necessary, to clear any vapors

Set sample chamber lever to Seal



Home

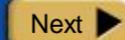


Contents

Thermo
SCIENTIFIC



Back



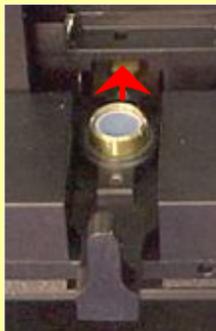
Next

Installing a Sample

How to install a solid sample (continued):

3. Set the helium flow rate to 10 cc/s.

4. Set the sample chamber lever to Open.



5. Place the sample holder on the sample insertion platform and slide the holder into the PAC 300 as far as it will go.

Steps to install a solid sample

Add spacers, if needed

Load sample

Adjust flow rate

Set sample chamber lever to Open

Insert sample holder

Set gain

Set sample chamber lever to Open

Purge, if necessary, to clear any vapors

Set sample chamber lever to Seal



Home

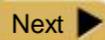


Contents

Thermo
SCIENTIFIC



Back



Next

Installing a Sample

How to install a solid sample (continued):

6. If the spectrometer Velocity parameter in the current OMNIC experiment is set to 0.16 cm/s, set the PAC 300 amplifier Gain to 3.



[Click here](#) for recommended Gain settings when using other velocities. See your OMNIC on-line help for information on setting Velocity and other parameters in OMNIC experiments.

7. If the sample evolves vapor when heated by the infrared beam, set the sample chamber lever to Open Purge and wait for the vapors to clear.

You may need to experiment with the time required to completely clear the vapors before continuing with the analysis.

Steps to install a solid sample

Add spacers, if needed

Load sample

Adjust flow rate

Set sample chamber lever to Open

Insert sample holder

Set gain

Set sample chamber lever to Open Purge, if necessary, to clear any vapors

Set sample chamber lever to Seal



Home



Contents

Thermo
SCIENTIFIC

◀ Back

Next ▶

Installing a Sample

How to install a solid sample (continued):

8. Set the sample chamber lever to Seal.



Caution

If the lever sticks, do not force it. Jiggle the sample holder to line up the sample cup with the bore of the sample chamber.

Steps to install a solid sample

Add spacers, if needed

Load sample

Adjust flow rate

Set sample chamber lever to Open

Insert sample holder

Set gain

Set sample chamber lever to Open

Purge, if necessary, to clear any vapors

Set sample chamber lever to Seal



Home

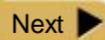


Contents

Thermo
SCIENTIFIC



Back



Next

Installing a Sample

How to install a powder sample:

1. Fill the sample cup.



Small cup filled with powder in sample holder with spacer above cup

Funnel used on sample holder



Funnel used on pedestal



Loading powder samples using funnel and spatula

Installing a powder sample

Use the sample holder with a stainless-steel cup to measure powder samples. Use the pedestal and a funnel to load the powder. Load enough powder to cover the bottom of the cup.

You must be careful not to let the purge gas blow the powder from the sample cup when loading and unloading powder samples. To prevent this from occurring, turn off the gas flow (or pinch the gas line), insert the sample and set the sample chamber lever to Sealed. Then turn on the gas flow, move the lever to Closed Purge and wait until the chamber fills with helium. Move the lever back to Sealed before collecting data. Remember to turn off the gas flow again while you remove the sample.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing a Sample

How to install a powder sample (continued):

2. Set the sample chamber lever to Open.

In this position the purge gas will flow through the sample chamber



3. Turn off the gas flow (or pinch the gas line) to prevent it from blowing the powder from the cup.



4. Place the sample holder on the sample insertion platform and slide it into the PAC 300 as far as it will go.

Steps to install a powder sample

Fill sample cup

Set sample chamber lever to Open

Turn off gas flow

Insert sample

Set sample chamber lever to Seal

Resume gas flow

Set sample chamber lever to Closed
Purge

Set sample chamber lever to Seal

Set gain



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Installing a Sample

How to install a powder sample (continued):

Establish a helium atmosphere in the sample chamber without blowing gas directly over the powder sample:



Seal sample chamber by rotating lever directly to Seal.

6. Turn gas back on

Resume flow of purge gas by unpinching line or turning gas back on.



7. Close Purge

Purge sample chamber by rotating lever to Closed Purge.



8. Seal

Seal sample chamber by rotating lever back to Seal.

Steps to install a powder sample

Fill sample cup

Set sample chamber lever to Open

Turn off gas flow

Insert sample

Set sample chamber lever to Seal

Resume gas flow

Set sample chamber lever to Closed Purge

Set sample chamber lever to Seal

Set gain



Thermo
SCIENTIFIC



Installing a Sample

How to install a powder sample (continued):

9. If the spectrometer Velocity parameter in the current OMNIC experiment is set to 0.16 cm/s, set the PAC 300 amplifier Gain to 3.



[Click here](#) for recommended Gain settings when using other velocities. See your OMNIC on-line help for information on setting Velocity and other parameters in OMNIC experiments.

Steps to install a powder sample

Fill sample cup

Set sample chamber lever to Open

Turn off gas flow

Insert sample

Set sample chamber lever to Seal

Resume gas flow

Set sample chamber lever to Closed
Purge

Set sample chamber lever to Seal

Set gain



Home

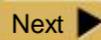


Contents

Thermo
SCIENTIFIC



Back



Next

Measuring the Sample

How to collect a sample spectrum:

Use the Collect Sample command in the OMNIC Collect menu or click the Collect Sample button on the toolbar, if it is displayed, to start sample collection.



Collect Sample button

Measuring the sample

Once the sample is sealed in the PAC 300, you are ready to collect the sample data. The sample measurement will show how the energy you started with was reduced, due to the lower absorption and higher thermal mass of the sample relative to the reference.

Use the [Collect Sample](#) command in the OMNIC Collect menu to start data collection. Then follow the instructions displayed on your screen to measure the sample. The instructions that appear will differ depending on how the [Background Handling](#) and other parameters and options are set in OMNIC and in the selected experiment.



Home



Contents

Thermo
SCIENTIFIC



Back

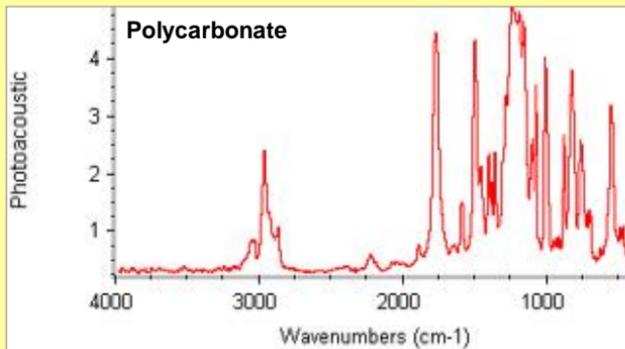


Next

Measuring the Sample

How to collect a sample spectrum (continued):

This is a spectrum of polycarbonate measured as a slab. A carbon black background spectrum was used to ratio the sample spectrum.



Photoacoustic spectrum of polycarbonate

Sample spectrum

When Photoacoustic is selected as the [final format](#) of your sample spectra—that's how it's set up in the standard PAC 300 experiment file—OMNIC collects a few scans and then calculates and displays a photoacoustic spectrum. The spectrum is updated as new data are collected.

When the system has finished collecting the number of scans specified in the experiment, the final spectrum is displayed in a window.

The final spectrum shows only the sample's photoacoustic absorbance spectrum (the background absorbance has been removed).



Home



Contents

Thermo
SCIENTIFIC



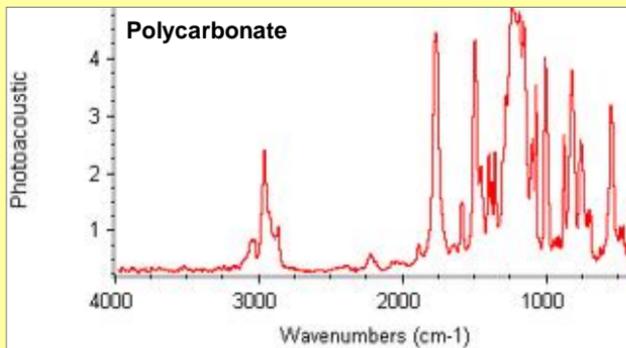
Back



Next

Measuring the Sample

Here is a spectrum of polycarbonate. The spectrum is shown in photoacoustic units, which are arbitrary absorbance units.



Photoacoustic spectrum of polycarbonate

Typical photoacoustic spectrum

A photoacoustic spectrum is similar to an absorbance spectrum measured by transmission in that the locations and intensities of spectral bands are unique to a particular sample. [Differences in the shapes and intensities of bands](#) are primarily due to differences in the effective sample thickness for the two measurements. The physical sample thickness is the effective sample thickness for transmission sampling whereas, for photoacoustic sampling, the effective thickness, L , is approximated by $\sqrt{D/\pi f}$ where f is the IR beam modulation frequency and D is the sample's thermal diffusivity. Strong bands in photoacoustic spectra usually exhibit truncation due to the size of L but spectra can still be used for library search and quantitative analyses.



Home



Contents

Thermo
SCIENTIFIC

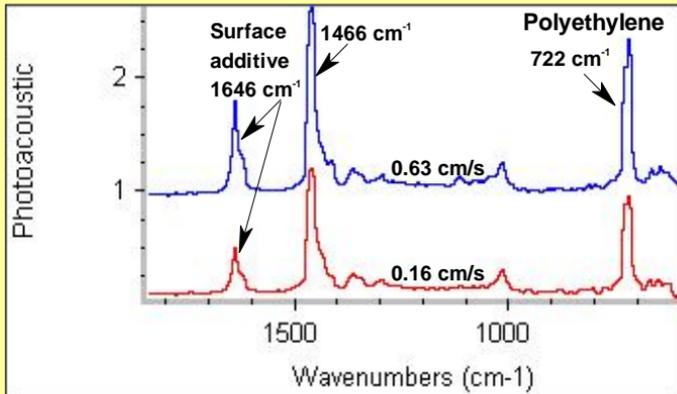


Back



Next

Measuring the Sample



Surface segregating additive revealed by varying the photoacoustic sampling depth

Variable sampling depth

The sampling depth, L , of a photoacoustic measurement is approximated by the expression $L = \sqrt{(D/\pi V \nu)}$, where D = thermal diffusivity, V is the spectrometer mirror velocity, and ν the wavenumber of a point of interest in a spectrum. A value $D = 10^{-3} \text{ cm}^2/\text{sec}$ is a good approximation for most polymers. Samples with gradient or layered composition can be studied by [measuring spectra as a function of increasing velocity](#) in order to sample closer to the surface. The spectra shown at the left indicate the presence of a surface segregating additive due to the increased size of the band at 1646 cm^{-1} . The bottom spectrum was measured using a velocity setting of 0.16 cm/s . The top spectrum was measured at 0.63 cm/s .



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Cleaning Up



Cleaning the sample cup

Clean the outer brass sample cup and the inner sample cup, if one was used, after you finish measuring each sample.

You can blow the cups clean with a jet of dry air or nitrogen. If thorough cleaning is needed, wipe the cup with a cotton swab moistened with ethanol. If you clean the holder with ethanol, allow enough time for the ethanol to evaporate completely. To ensure complete evaporation, dry the sample cup with clean, dry air or nitrogen.

When the cup is clean and dry, you are ready to load the next sample.



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Turning Off the PAC 300

How to turn off the PAC 300 accessory:

- 1. Remove any sample holder from the sample chamber.**
- 2. Place fresh desiccant in a clean, dry sample cup and place a notched spacer over the desiccant.**
- 3. Insert the sample holder with the desiccant into the PAC 300 sample chamber.**
- 4. Wait 10 seconds to allow the sample chamber to completely purge with helium, and then set the sample chamber lever to Seal.**
- 5. Turn off the regulator and main valve on the helium cylinder.**

Turning off the PAC 300

When you are finished running samples with the PAC 300 (or at the end of the day), follow the steps in the box at the left to purge and seal the sample chamber. A purged sample chamber protects the detector microphone and other internal components from moisture and other contaminants found in room air. It also shortens the time required to set up the accessory the next time you want to use it.

Place small parts in a common location to avoid losing them.



Home



Contents

Thermo
SCIENTIFIC



Back

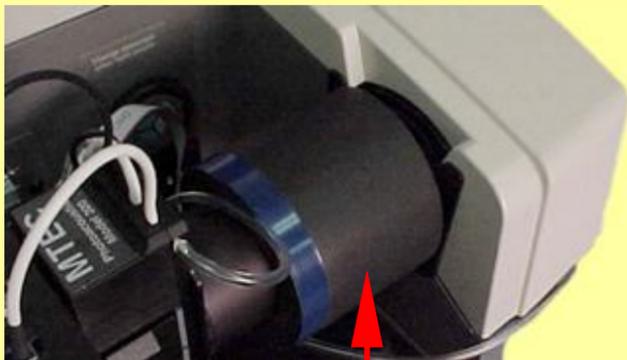


Next

Removing the PAC 300

How to remove your accessory:

1. Slide the purge coupler away from the side wall of the spectrometer sample compartment.



Purge coupler

Removing the PAC 300

Before removing the PAC 300 from the spectrometer sample compartment, follow the instructions in the previous section to purge and seal the sample chamber. A purged sample chamber protects sensitive internal components and shortens the setup time.

You can remove the PAC 300 accessory as easily as you installed it. When not in use, your accessory and its associated components should be stored in a dry, dust-free environment such as a cabinet or box.



Home

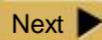


Contents

Thermo
SCIENTIFIC



Back

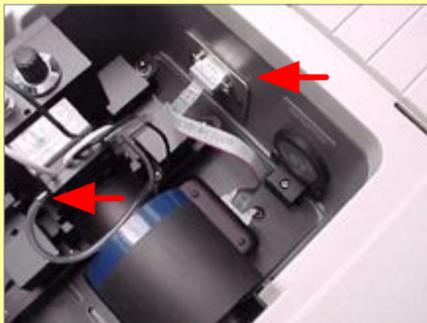


Next

Removing the PAC 300

How to remove your accessory (continued):

2. Disconnect the interface cable from the spectrometer sample compartment.



3. Make sure the regulator and main valve on the helium gas cylinder are off and then disconnect the purge line.

Steps to remove the PAC 300

Slide purge coupler away from side wall

Disconnect cable

Disconnect purge line

Remove accessory

Select experiment for next analysis



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Removing the PAC 300

How to remove your accessory (continued):

4. Grasp the PAC 300 on either side of the sample chamber and gently lift the accessory out of the spectrometer sample compartment.



Steps to remove the PAC 300

Slide purge coupler away from side wall

Disconnect cable

Disconnect purge line

Remove accessory

Select experiment for next analysis



Home

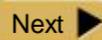


Contents

Thermo
SCIENTIFIC



Back



Next

Removing the PAC 300

How to remove your accessory (continued):

5. Open an experiment.

After you remove an E.S.P. accessory, a dialog box appears allowing you to select the experiment for your next analysis.



Click the arrow in the list box to see the titles of the experiments available in OMNIC.

Click a title to open the experiment.

Steps to remove the PAC 300

Slide purge coupler away from side wall

Disconnect cable

Disconnect purge line

Remove accessory

Select experiment for next analysis



Home



Contents

Thermo
SCIENTIFIC



Back



Next

Where To Go Next



For more information

Your OMNIC software includes many kinds of on-line documentation to give you easy access to useful information.

If you are interested in learning more about FT-IR spectrometers and how they work, see the Getting Started and Learning OMNIC options in the OMNIC Help menu. For detailed information about your spectrometer and OMNIC software, choose the OMNIC Help Topics or the Spectrometer Help topics option in the OMNIC Help menu.

If you need information on another sampling technique or accessory, open Sampling Techniques from the OMNIC Help menu and then choose the technique or accessory name.

