

Measurement of the infrared absorption spectrum with an FT-IR spectrometer

**Miscellaneous Operating Procedure # 2519
NRMRL/AEMD**



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1 Scope and Application

This miscellaneous operating procedure (MOP) describes the procedure for measuring Fourier-transform infrared (FT-IR) absorbance spectra of membrane air filter samples on a FT-IR spectrometer. Light absorption measurements obtained by FT-IR can be used for a variety of applications such as quantifying the underlying composition of organic and inorganic functional groups containing molecular bonds with a dipole moment. This MOP will cover the basic procedures of the filter absorption measurement that will be common to any such application.

This MOP is intended for scientists, engineers, and technicians seeking to measure the absorption spectrum of a membrane air sampling filter. This MOP will describe the procedures necessary to make an absorption measurement from membrane air sampling filters. Sample preparation and collection procedures will be covered in separate project-specific quality assurance project plans (QAPPs).

2 Personnel

A table of responsible personnel is shown in Table 1.

Name	Role
Emily Y. Li	Analyst
Michael D. Hays	Principal Investigator
Richard Shores	Distributed Source & Buildings Branch Chief
Libby Nessley	Quality Assurance Manager
Robert S. Wright	Technical Services Team

Table 1: FT-IR spectrometer personnel.

Due to the complexity of the FT-IR spectroscopic method, new analysts must demonstrate proficiency before performing routine analyses. For example, a new analyst will be requested to duplicate FT-IR spectroscopic measurements already performed by an experienced analyst.

3 Method Summary

This method covers the procedures for measuring the absorption spectrum of a membrane air sampling filter with a TENSOR II Spectrometer (Bruker Corporation, Billerica, MA) equipped with a liquid nitrogen-cooled detector. Water vapor and CO_2 are purged from the sample compartment and instrument optics to minimize absorption bands of gas phase ucompounds in the aerosol spectra.

Samples are measured in transmission-mode, where the IR beam is directed through the sample. Absorbance (A) can be obtained by ratioing the measured extinction of radiation through the sample (I) by a reference value (I_0 , also called the “background”) and taking

the negative value of their base-10 logarithm (Eqn. 1). The absorbance across a range of IR beam wavenumbers is an absorbance spectrum using the most recent empty chamber spectrum as a reference, collected hourly. The quality of the absorbance spectrum depends on how accurately the background reflects the conditions of the sample scan, and therefore the background is acquired regularly. The total measurement time for one filter is approximately 5 minutes.

$$A = -\log_{10} \frac{I}{I_0} \quad (1)$$

4 Definitions

- Measuring device: TENSOR II FT-IR Spectrometer (Bruker Corporation, Billerica, MA).
- Sample holder: Square stainless steel box with a slot and lid, custom-built to hold each filter sample the same distance from the source.
- Wavenumber: the number of waves in one centimeter. Directly proportional to and used as a measure of the specified energy of the light source, and has the units of reciprocal centimeters (cm^{-1}).
- Detector: the liquid nitrogen-cooled mid-IR detector used for FTIR measurements.

5 Health and Safety Considerations

Laboratory safety practices should be in accordance with the sample and standard laboratory practices. Gloves, lab coat, and safety glasses should be used as necessary.

Liquid nitrogen, which is a cryogenic fluid, is used when setting up the FT-IR instrument. There are two groups of safety hazards associated with cryogenic liquid nitrogen: extreme cold and asphyxiation.

The TENSOR II FT-IR spectrometer is equipped with a laser diode that is classified as a laser class 1 product[2].

Safety measures to be taken with extreme cold, asphyxiation, and laser radiation hazards are detailed in Subsections 5.1-5.3.

5.1 Extreme cold hazard

Cryogenic liquids and vapors can produce effects on skin similar to a thermal burn. Brief exposures that would not affect skin on the hands can still damage delicate tissues such as the eyes. Prolonged breathing of extremely cold air may damage the lungs. A face shield must be worn when dispensing or transporting liquid nitrogen. The face shield must be used in conjunction with safety glasses or goggles.

Prolonged exposure of the skin or contact with cold surfaces can cause frostbite. Unprotected skin can stick to extremely cold metal, tearing the skin when pulled away. Even non-metallic materials are dangerous to touch at low temperatures. Thermally insulating gloves must be worn while handling liquid nitrogen.

When transporting liquid nitrogen, use a double-walled vessel, such as a glass dewar flask. In these vessels, the space between the two walls is a vacuum, which means a crack forming in the inner or outer wall may cause a sudden implosion. With cryogenic fluid inside, a crack may cause a sudden expansion of cryogenic gas that may explode the vessel. Handle such vessels carefully for personal safety and to prevent breakage.

5.2 Asphyxiation hazard

When liquid nitrogen forms a gas, the gas displaces oxygen in the air. When there is not enough oxygen, asphyxiation and death can occur. Oxygen deficiency is a serious hazard in enclosed or confined spaces. Small amounts of liquid nitrogen can evaporate into very large volumes of gas. For example, one liter of liquid nitrogen evaporates to 695 liters of nitrogen gas when warmed to room temperature (21°C). Do not dispense liquid nitrogen in enclosed or confined spaces.

5.3 Hazardous laser radiation

The TENSOR II spectrometer is classified by the International Electrotechnical Commission standard (IEC 60825-1:2007) as a laser class 1 product. According to this classification, laser radiation is not accessible for the user if the spectrometer is used as intended. Intended use is defined in Section 1.5 of the TENSOR II User Manual[2].

Inside the interferometer, the semiconductor vertical-cavity surface-emitting laser (VCSEL) diode emits invisible light with a wavelength of 850 nm at power output 2 mW. IEC 60825-1:2007 classifies this laser component as laser class 3B[2].

According to the TENSOR II User Manual, there may be potential risk of exposure to laser class 3B radiation in the cases of damaged spectrometer housing or unauthorized removal of the laser safety cover. **In these cases, switch off the spectrometer immediately.** Avoid any eye exposure to the laser radiation, and keep in mind that this laser radiation is invisible. The TENSOR II FT-IR power switch is located in the back of the spectrometer (see Figure 3 on page 8).

6 Limits of Application

Ambient temperature must be between 18°C and 35°C at a non-condensing humidity ($\leq 80\%$ relative humidity). See the TENSOR II User Manual[2] for more site requirements.

7 Equipment and Supplies

7.1 Instrumentation

1. [TENSOR II FTIR Spectrometer](#)
2. Computer with Windows operating system and [OPUS Spectroscopy Software](#) installed.

The TENSOR II FTIR Spectrometer (Figure 1) has a spectral range of mid-IR 8,000-340 cm^{-1} , a spectral resolution of better than 0.4 cm^{-1} , wave number accuracy of better than 0.01 cm^{-1} , photometric accuracy of better than 0.1% T, and scan velocities of 1.6 to 60 kHz. The MIR source is a U-shaped silicon carbide piece that emits mid-infrared light. The beamsplitter material is KBr, the detector used is a liquid nitrogen-cooled mercury cadmium telluride (MCT) detector. The laser is a semiconductor VCSEL diode outputting a wavelength of 850 nm at a power of 2 mW.

Technical details are available online at [Bruker.com/Products](#) and the specifications are available in Appendix A of the TENSOR II User Manual[2].

7.2 Installation

The TENSOR II is a benchtop FT-IR spectrometer capable of measuring light absorption over the IR wavelength range. Initial instrument installation was performed by the manufacturer; refer to the hardware manual (TENSOR II User Manual, saved as a PDF to the desktop of the computer installed with the TENSOR II FT-IR in lab E580A) for detailed instructions if the instrument needs to be connected to a new computer.

7.3 Maintenance

Components with a limited service lifetime are the IR source lamps and the desiccant cartridge.

The interferogram peak test and the energy test described in Section 9.2 on page 17 can be used to determine whether the IR source lamp needs to be replaced.

The desiccant cartridge in the TENSOR II interferometer should be replaced every 6 months[2]. Refer to the TENSOR II User Manual Section 6.3 Replacing the cartridge and regenerating the desiccant.

Refer to the TENSOR II User Manual for troubleshooting procedures and replacement part numbers.

The spectrometer sample compartment and sample holder should be kept clean.

7.4 Ancillary equipment: purge gas supply

An [ekom® DK50 PLUS/M MOBILE combination air compressor and dehumidifier](#) acts as the purge gas supply for the TENSOR II FT-IR Spectrometer.

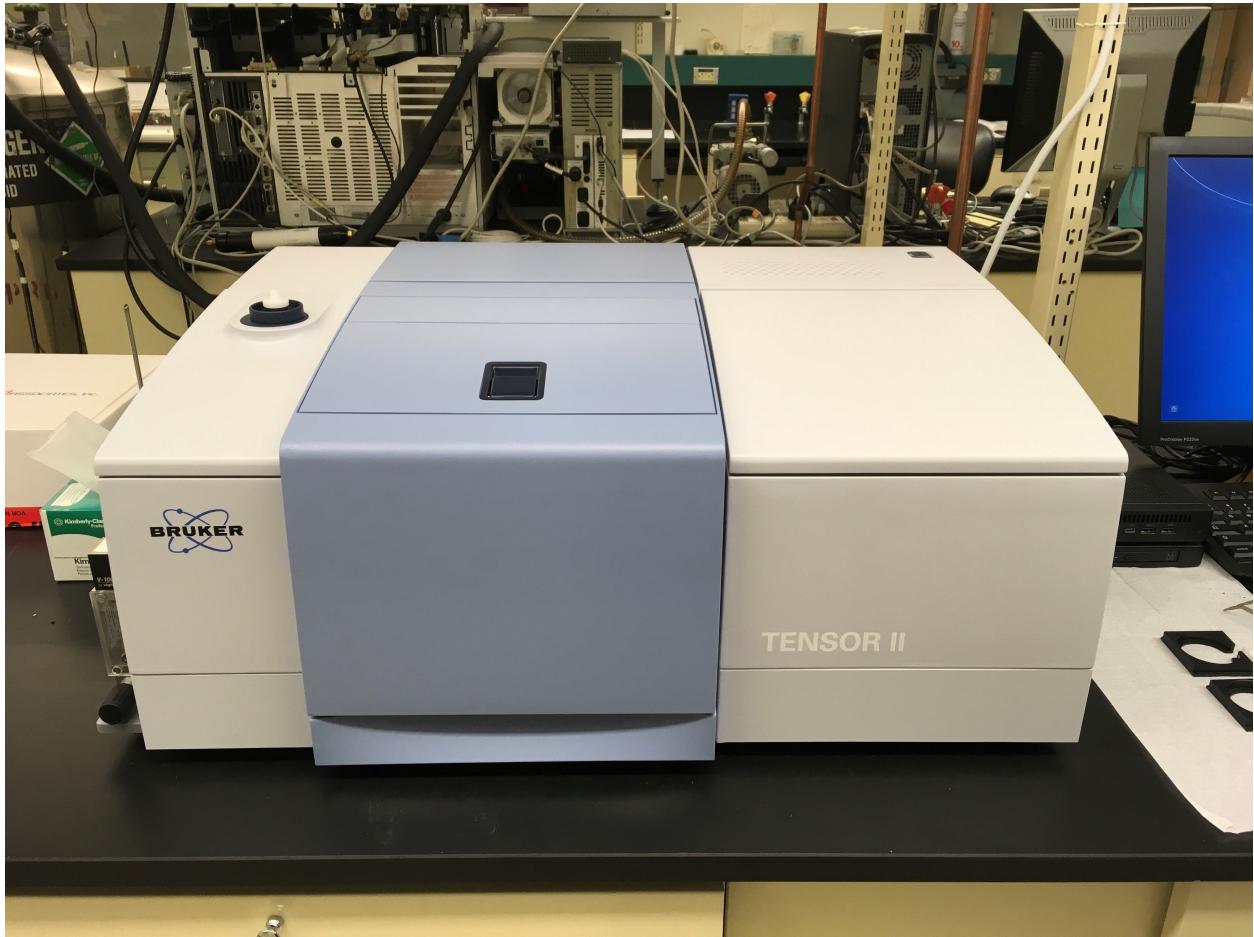


Figure 1: FT-IR instrument.

A [Vögtlin V-100 55 variable area flow meter](#) is used to control the purge gas flow rate. The datasheet PDF is available at https://www.voegtlin.com/data/139-2018_en.infoV100.pdf.

The purge gas supply is plumbed to the FT-IR spectrometer as shown in Figure 2.

Use the following specifications as stated by the TENSOR II User Manual to purge the optical bench and sample compartment in the TENSOR II FT-IR[2]:

- Dry air (with a dew point < -40°C , which corresponds to a degree of dryness of 128 ppm humidity)
- Oil-free and dust-free
- Maximum pressure: 2 bar (29 psi)
- Recommended sustained purge gas flow rate: 200 liters/hour



Figure 2: Back of the TENSOR II FT-IR, showing where the purge gas supply is plumbed into the spectrometer housing.

- The purge gas flow rate should not exceed 500 liters/hour. Too high a purge gas flow rate will both damage the optics and introduce acoustic noise into measurements.

7.5 Sample requirements

Sample collection will be covered by the separate quality assurance plan. This QAPP covers the measurement of filter samples.

Air filters used with this FT-IR measurement procedure must be Teflon or polytetrafluoroethylene (PTFE) membrane disc filters of circumference 25, 37, or 47 mm. The membrane pore sizes must be 1, 2, or 3 microns. Acceptable brands include Pall Corporation and MTL, LLC.

8 Measurement Procedures

Measurement procedures include preparing samples, preparing the spectrometer, preparing parameters and sample list files for routine measurement, cleanup, and shutting down.

8.1 Preparing the spectrometer for measurement

1. Make sure the Bruker TENSOR II FT-IR spectrometer (Figure 1) has been on for at least two hours.
2. If the TENSOR II FT-IR is off, turn it on using the switch in the back, indicated in Figure 3. Wait two hours.

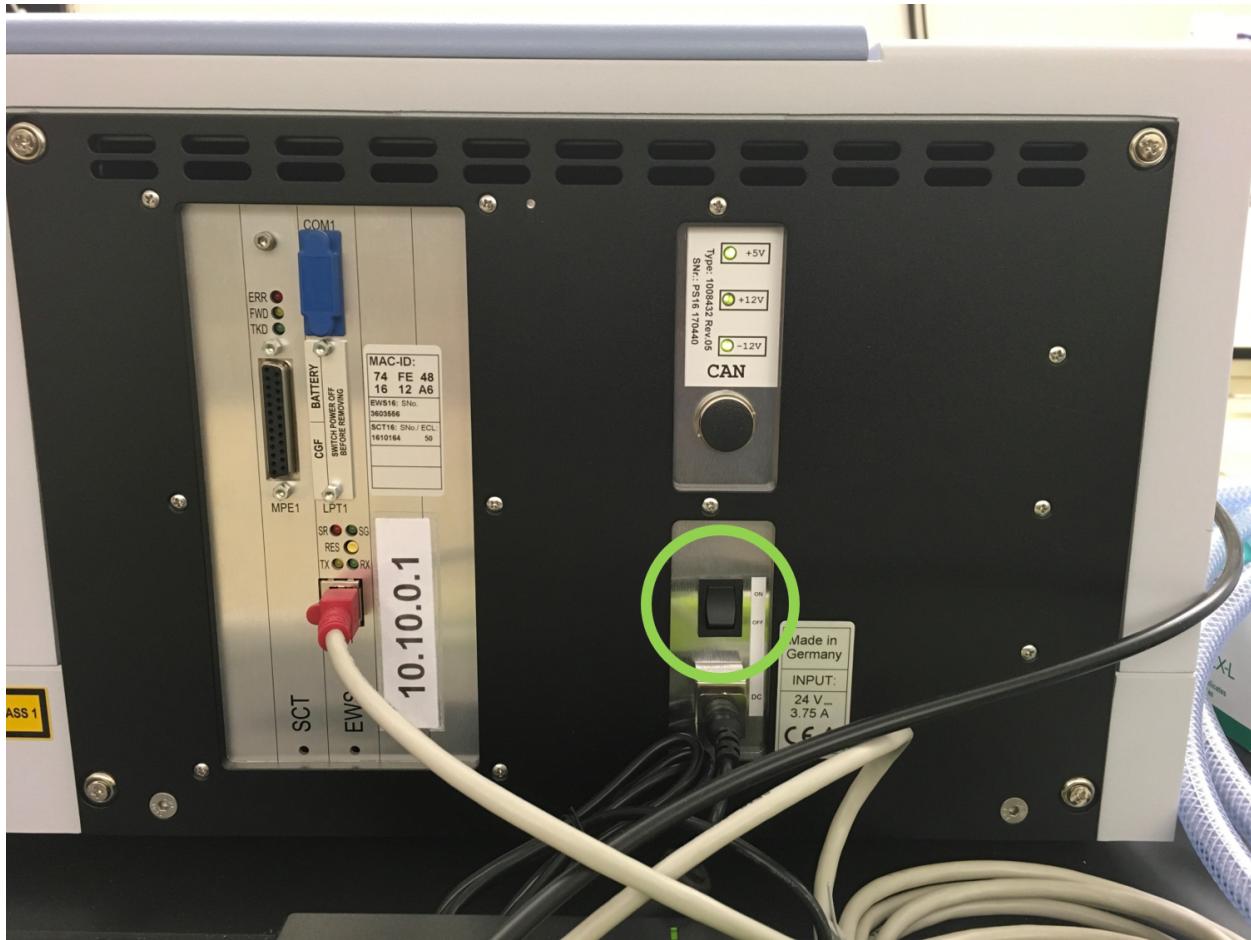


Figure 3: Back of the TENSOR II FT-IR, with the on/off switch indicated in a green circle.

3. Fill the liquid nitrogen dewar on the MCT detector (Figure 4). Follow the TENSOR II User Manual Section 5.10, “Cooling the MCT Detector.” It takes approximately 0.5

L of liquid nitrogen to fill the detector dewar. After filling the dewar, wait 5 minutes for the detector temperature to stabilize. A full liquid nitrogen dewar on the MCT detector will hold at operating temperature for 8 hours.

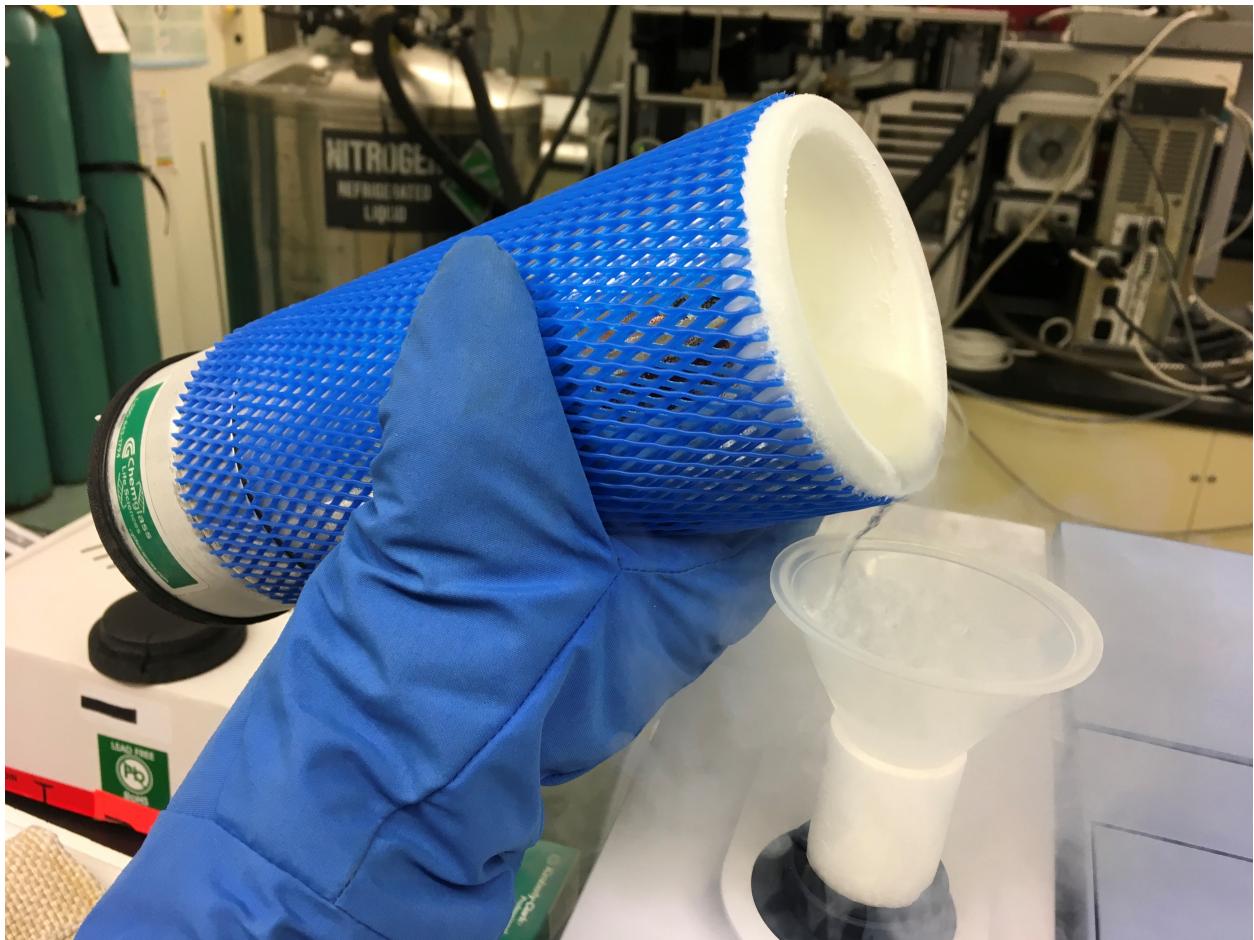


Figure 4: Liquid nitrogen pouring.

4. Double click the OPUS software icon on the desktop (see Figure 5).
5. Log in with the following credentials and workspace (see Figure 6):
 - User ID: **Administrator**
 - Password: **OPUS**
 - Assigned workspace: **EPA.ows**
6. Allow the software to run through all the system checks. The status circle in the bottom right corner of the software will be green if the system checks are all OK (Figure 7).



Figure 5: Desktop.

For example, the status circle will be red if the liquid nitrogen level in the detector's dewar is too low. Click the status circle to bring up a diagnostics window that will show which system checks need attention (Figure 8).

7. If the OPUS system checks do not pass, open the diagnostics window by clicking the status circle in the bottom right corner of the OPUS software screen. Follow the TENSOR II user manual to troubleshoot each issue. Common failures include not filling the liquid nitrogen dewar on the detector, or an expired performance test. More details on the instrument tests are available in Section 9 Quality Control on page 16.

8.2 Taking a routine measurement

1. List the desired sample names in the text file `Sample_list.txt`. Note that this can be any text file where each line is the desired name for each sample to be measured.
[REDACTED] The last line will be ignored by the measurement routine, so be sure to terminate the .txt file with a final filler line such as “`sample_x`” so that the last sample is included in measurements.
2. Save and close the notepad.
3. To start a routine measurement, click the “Background/Filter Scan” icon, which looks

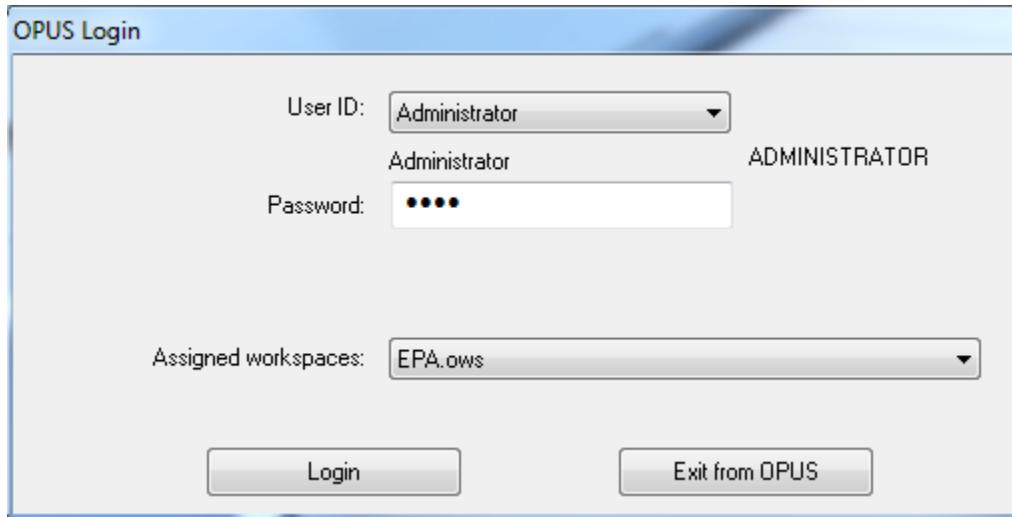


Figure 6: OPUS Login.

like a globe of planet earth in the toolbar at the top left of the workspace (see again Figure 7).

4. In the Measurement Parameters window (Figure 9), check that the following fields are filled out:

- XPM File:
 - Path: C:\Users\Public\Documents\Bruker\OPUS_7.5.18\XPM\
 - Name: BrukerXPM2.xpm
- Force to acquire new background in **60** minutes
- Wait time before acquiring background **0** seconds
- Wait time before measuring sample **180** seconds
- Text File Containing Sample Names
 - Path: C:\Temp
 - Name: Sample_list.txt

Entering 180 seconds into the field “Wait time before measuring sample” sets up the sample compartment to purge for 180 seconds, or for 3 minutes before starting each measurement.

5. Proceed by clicking the blue arrows.
6. The system will prompt the user to measure an empty background as a reference. Make sure the sample chamber is empty and closed, and then click OK to proceed.

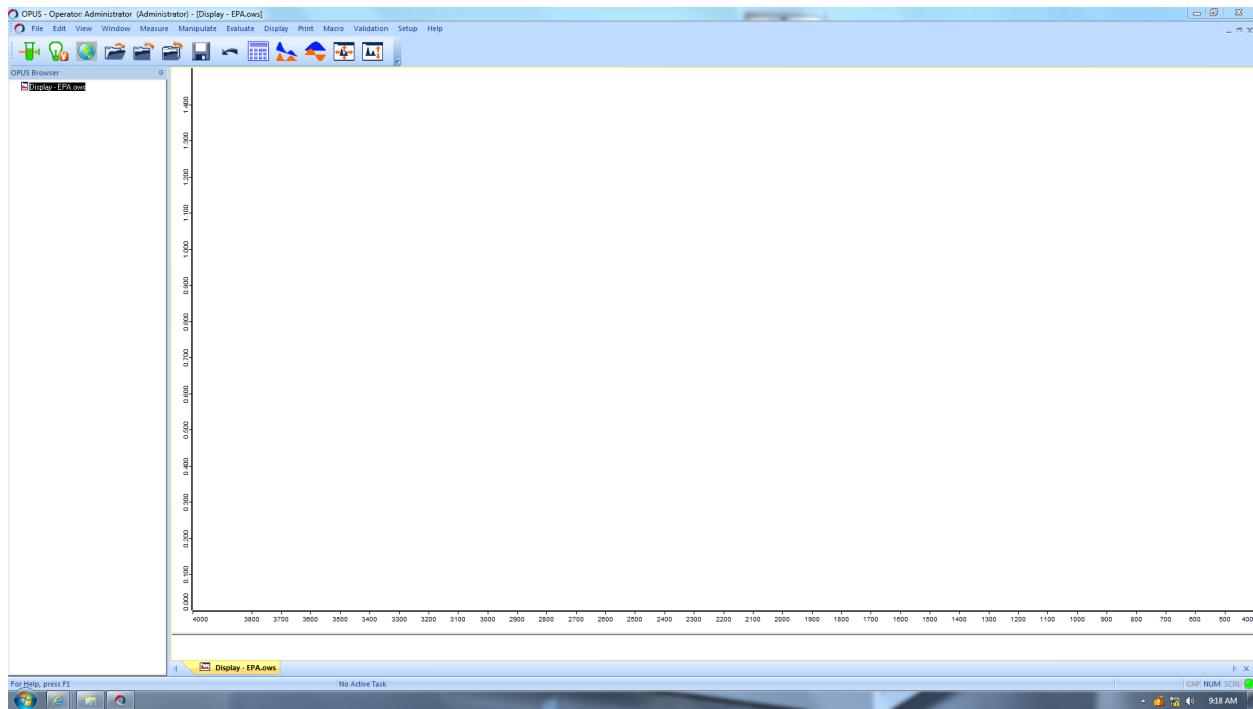


Figure 7: Starting screen for the OPUS software. The status circle in the bottom right corner should be green. If the status circle is red, click it to open a diagnostics window.

7. Wait for the measurement to be taken.
8. When the measurement is complete, the system will prompt you to insert a sample. First check the sample name and the corresponding filter label, then place the matching filter into the sample holder. The collection side of the filter should face the IR light source beam. Close the sample holder fully, and then click OK to proceed.
[REDACTED] The system will initiate the 180-second wait time for the sample measurement as soon as the user clicks OK, so to ensure a consistent purge time between each sample, be sure to only click OK after the sample filter is slotted into place inside the sample holder and the cover is fully shut.
9. Repeat steps 7 and 8 until the measurements for all the samples in the **Sample_list.txt** are complete. If the background measurement expires (which is scheduled to occur after 60 minutes of measurements), re-measure the empty chamber as needed.
10. Locate the measurement files in the directory C:\FTIR_Data. There will be two files saved for each sample measurement: a spreadsheet “.csv” and an OPUS file “.0”. If the **Sample_list.txt** included duplicate sample names, the second duplicate will have file extension “.1”, the third will have file extension “.2”, etc. The background measurement has automatically been subtracted from each sample measurement.

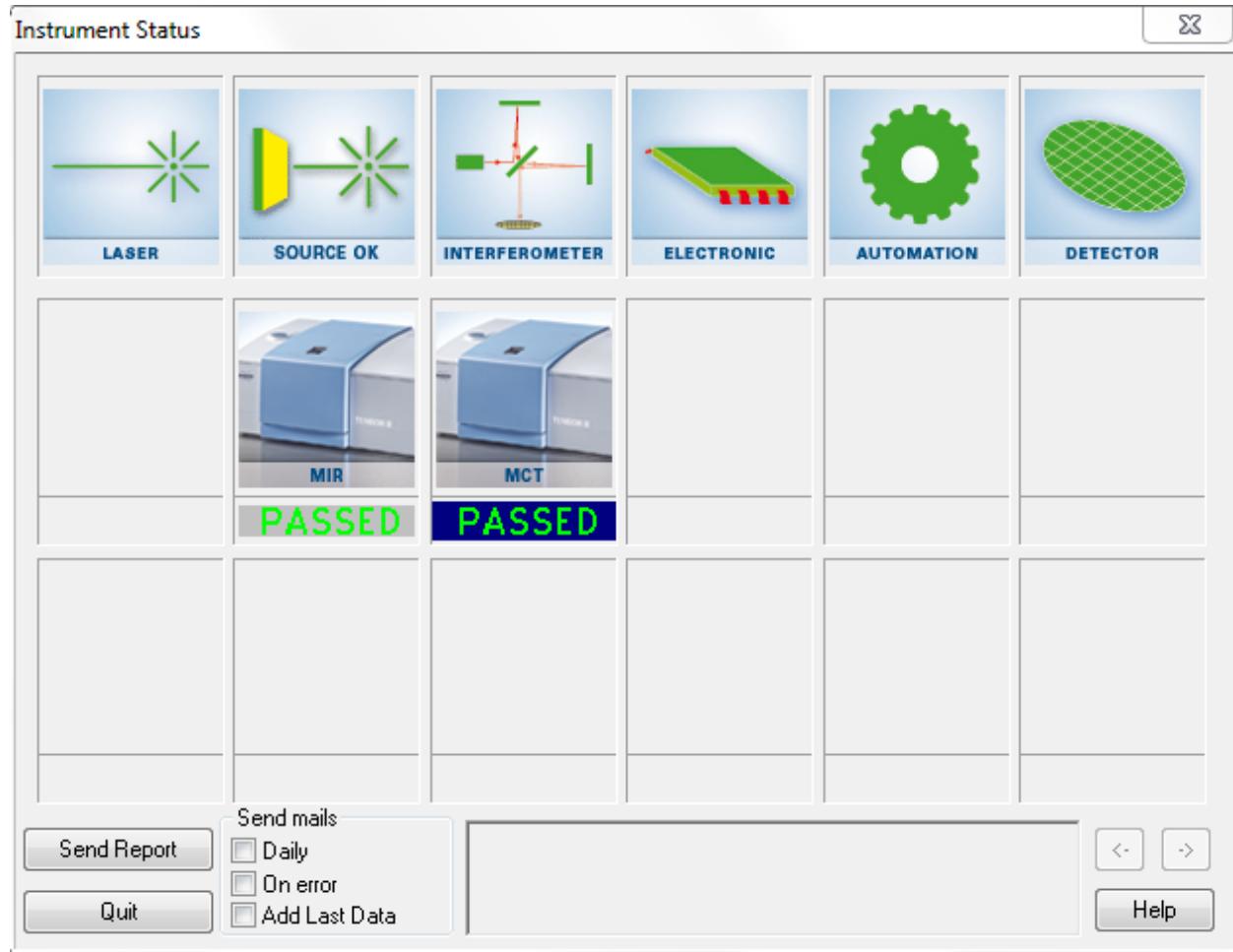


Figure 8: The OPUS diagnostics window for the TENSOR II FT-IR showing all instruments statuses and system checks OK.

11. Move all the sample files (both .csv and OPUS files) into a folder labeled [yyymmdd-xx], where xx is the experiment number for that day. For example, the third set of measurements taken on April 4, 2018 would be in the folder labeled “180404-03” (see Figure 10).

8.3 Taking screenshots in the OPUS software

To take screenshots of spectra in the OPUS software, select the OPUS window displaying the desired spectra and axes. Press Ctrl+C. Open Paint and press Ctrl+V. Save as a .png.

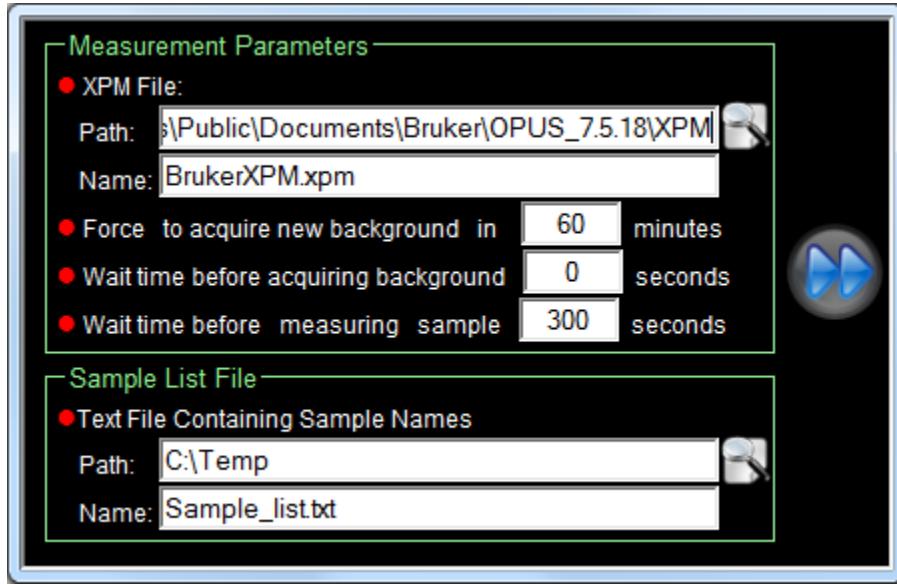


Figure 9: Parameters for routine measurement.

8.4 Shutting down

1. Save data as necessary as described in Steps 10-11 in Subsection 8.2 on page 10.
2. Close the OPUS software and any other associated open program by clicking the x in the top right-hand corner or clicking on the File menu and selecting Exit.
3. **Do not turn off the instrument.** As described in the TENSOR II User Manual, the instrument should be left on overnights and over the weekends. The TENSOR II spectrometer should only be switched off in case of a long time of nonuse.

8.5 Data description and calculations

The OPUS software saves both a .0 and a .csv file for each spectrum taken (see Figure 10). Data can be viewed directly in the OPUS software by importing .0 files.

The .csv data file consists of two columns of wavenumber and relative absorbance data.

8.6 Data analysis

FT-IR spectra of particle matter collected on Teflon membrane filters will be analyzed for functional group composition and for calibration of various chemical entities. A baseline correction is required prior to performing peak-fitting for functional group analysis. Typically smoothing splines are used to perform the baseline correction [1]. Peak-fitting for functional group characterization is described exhaustively in Takahama et al. 2013 [4]; similar methods will be applied here. Calibration for determination of a variety of chemical entities, e.g.

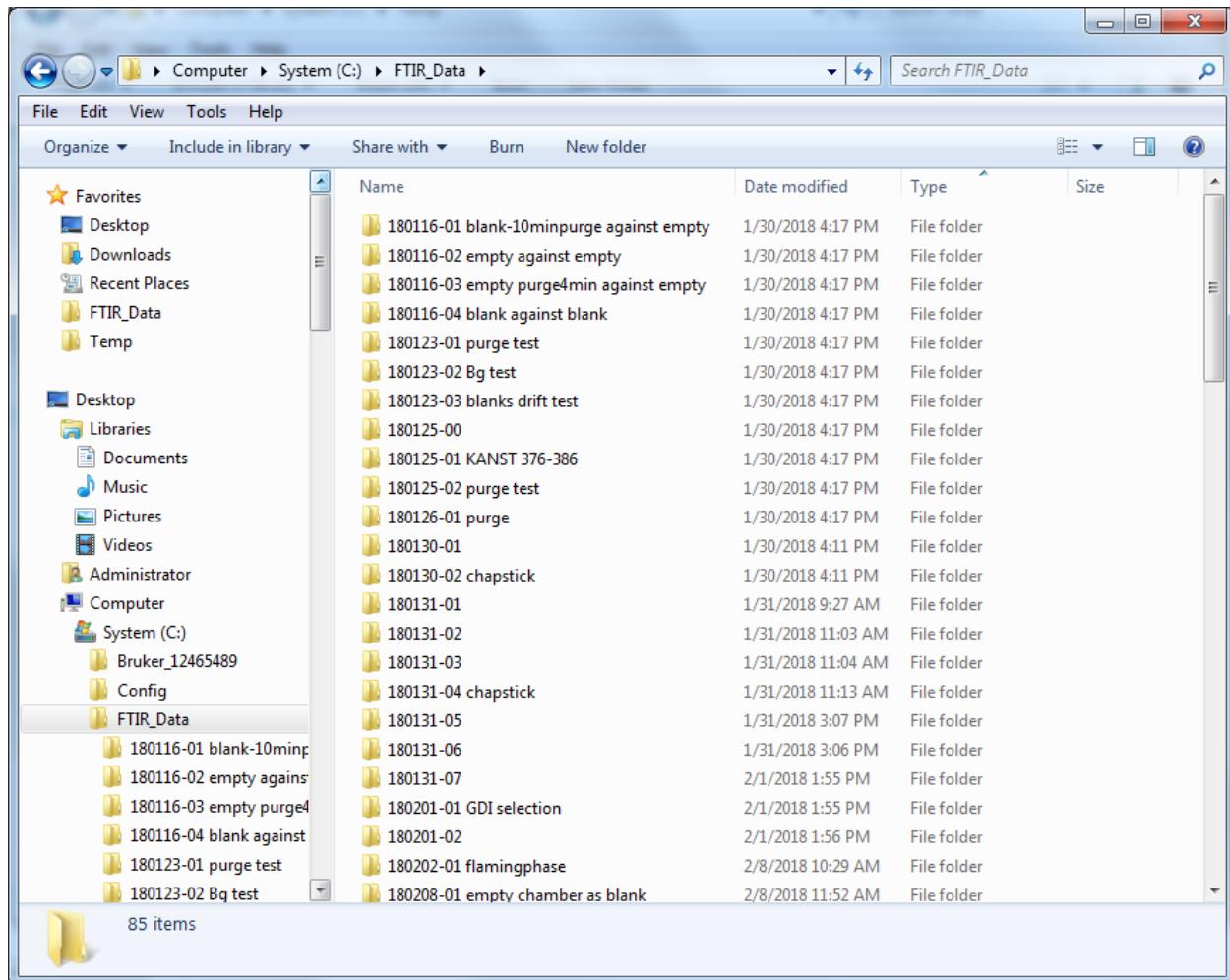


Figure 10: File directory for FT-IR measurements.

OC-EC, will be performed using a multivariate regression analysis as described earlier [3]. Baseline correction may also be performed prior to using FT-IR spectra for calibration. On-line chemometric tools developed for processing of FT-IR spectra are available at <http://airspec.epfl.ch/>.

Table 2 provides functional group characterization by wavenumbers.

Bond type	Corresponding wavenumbers (cm^{-1}) ^[4]
carbonyl CO	1720–1714
amine NH	1630–1620
carboxylic COH	3500–2400
ammonium NH	3400–2700
alcohol COH	3500–3377, 3371–3203
alkane CH	2932–2921, 2886–2882, 2855–2849, 2805–2800
alkene CH	3050
aromatic CH	2980

Table 2: Target functional group bond types and their associated FT-IR spectral wavenumber(s) or wavenumber range(s).

9 Quality Control

Monthly quality control checks are performed to monitor the comparability, precision and stability of the TENSOR II FT-IR instrument over time. The TENSOR II spectrometer is prepared with automated performance qualification (PQ) test routines for instrument validation^[2].

All details on PQ tests in this section are sourced from the OPUS software manual accessible via the Help menu of the OPUS software application.

The PQ test for the spectrometer can not be deactivated, and the maximum validity period of a PQ test is 30 days. When the PQ test validity period expires, the instrument status in the OPUS software turns yellow and indicates a warning that the reliability of the measurement results is not ensured.

The PQ test protocol includes the following tests. This document details each test in Section 9.2 below.

- Signal-to-noise test: This test verifies that the signal-to-noise ratio is better than a specified limit.
- Deviation from 100% line: This test verifies that the 100% line does not shift on the long term.
- Interferogram peak amplitude: This test verifies that the interferogram peak amplitude is not below a certain limit.
- Energy distribution test: This test verifies, by means of different methods, the total energy of the spectrometer and its different components.
- Ice band test: This test verifies the leak tightness of liquid nitrogen-cooled detectors.
- Wavenumber accuracy test / X-axis frequency calibration test: This test verifies that the frequency calibration of the instrument is correct.

- Photometric accuracy test / Y-axis reproducibility test: This test verifies that the photometric accuracy is within specified limits. The test is performed by using one or two filter(s) with different absorbing properties.
 - Glass Filter A Test
 - Glass Filter B Test (not available for the spectrometer referenced in this QAPP)

Upon delivery of the TENSOR II FT-IR spectrometer, Bruker set up the test channels necessary for the spectrometer and delivered accessories. If additional accessories are purchased in the future, new test channels will need to be set up. For instructions on setting new test channels or PQ tests in the OPUS software, see the TENSOR II User Manual[2] Section 5.11.4.

9.1 PQ Test Results

The PQ Test Protocol contains the PQ test results. The first page summarizes the individual test results and the overall PASSED or FAILED test result is shown (see Figs. 11-12). Its remaining pages list the acquired spectra and the used measurement parameter values for each individual test of the PQ test.

The PQ test protocol PDF file is saved automatically in the following directory: *Libraries \Documents \Public Documents \Bruker \OPUS-[current version] \Validation \Reports*. This report is automatically displayed immediately after the PQ test is finished.

9.2 Individual tests in the PQ test protocol

9.2.1 Signal to Noise Test

The signal-to-noise ratio test determines the sensitivity of the spectrometer by calculating the average signal-to-noise ratio of ten 100% spectra.

The S/N ratio is determined by collecting and analyzing a 100% spectrum. A 100% spectrum is the ratio of two successively acquired single-channel spectra with no sample in the sample compartment. The ratio of these two single-channel spectra is used to generate a transmission spectrum. The S/N ratio is calculated by the OPUS Signal-to-Noise Ratio command from the Evaluate menu, using peak-to-peak by means of the quadratic parabola fit option. In order to get a reliable result 10 spectra are measured (reference and sample). The S/N ratio is calculated separately for each spectrum, using the mean value of all 10 results.

9.2.2 Deviation from 100% Line Test

This test measures the maximum deviation of a 100% line within a larger frequency range. The average determined by 10 measurements must not exceed the predefined limit of 0.5, defined by the instrument manufacturer.

OVP - PQ Test Protocol



Company:	United States EPA		
Operator:	Administrator		
Instrument Type:	TENSOR II Sample Compartment RT-DLaTGS		
Optics Configuration:	Sample Compartment with: MIR, KBr, RT-DLaTGS [Internal]		
Accessory:	None		
Instrument Serial Number:	996		
Instrument Firmware Version:	5.000 1-0-0 Jan 27 2016		
OPUS/DB Version:	OPUS 7.5 Build: 7, 5, 18 / DB: 7,5,18,424		
Overall Test Result	PASSED		
Test expires:	6/8/2018, 9:36:03 AM (GMT-4)		
Test Date/Time:	5/9/2018, 9:36:03 AM (GMT-4)		
Test Spectra Path:	C:\Users\Public\Documents\Bruker\OPUS_7.5.18\Validation\Data\20180509\093603		
Date of last PQ Reference			
Measurement:	3/13/2018		
Comment:			
Signal to Noise Test ✓			
Minimum S/N(area 1):	3000	Measured S/N:	7142
100% Line Test ✓			
Maximum 100% Line Deviation:	0.5	Measured 100% Line Deviation:	0.20
Interferogram Peak Test ✓			
Minimum Amplitude[%] :	70	Measured Amplitude[%]:	94.8
Energy Test ✓			
Maximum allowed Value:	30	Measured Value:	5.5
Wavenumber Accuracy Test - Polystyrene (30.0 Deg. C, Peak is T. corr.) ✓			
Sample Material:	Polystyrene		
Specified Peak:	1601.45 cm ⁻¹	Maximum Deviation:	0.50 cm ⁻¹
Measured Peak:	1601.16 cm ⁻¹		
Corrected Peak:	1601.23 cm ⁻¹	Measured Deviation:	0.22 cm ⁻¹
Photometric Reproducibility Test - Glass Filter A ✓			
Maximum Deviation[%]:	0.8	Measured Deviation[%]:	0.04
Overall Test Result = PASSED ✓			
Date and Signature	Page 1 of 5	Date and Signature	Page Signature

Figure 11: First page of a PQ Test Protocol file for the room-temperature detector.

OVP - PQ Test Protocol



Company:	United States EPA		
Operator:	Administrator		
Instrument Type:	Tensor II Sample Compartment LN-MCT Mid		
Optics Configuration:	Sample Compartment with: MIR, KBr, LN-MCT Mid [Internal]		
Accessory:	None		
Instrument Serial Number:	996		
Instrument Firmware Version:	5.000 1-0-0 Jan 27 2016		
OPUS/DB Version:	OPUS 7.5 Build: 7, 5, 18 / DB: 7,5,18,424		
Overall Test Result	PASSED		
Test expires:	6/8/2018, 9:56:44 AM (GMT-4)		
Test Date/Time:	5/9/2018, 9:56:44 AM (GMT-4)		
Test Spectra Path:	C:\Users\Public\Documents\Bruker\OPUS_7.5.18\Validation\Data\20180509\095644		
Date of last PQ Reference			
Measurement:	3/16/2018		
Comment:			
Signal to Noise Test ✓			
Minimum S/N(area 1):	1000	Measured S/N:	3267
100% Line Test ✓			
Maximum 100% Line Deviation:	0.5	Measured 100% Line Deviation:	0.25
Interferogram Peak Test ✓			
Minimum Amplitude[%] :	70	Measured Amplitude[%]:	93.3
Energy Test ✓			
Maximum allowed Value:	30	Measured Value:	6.7
Ice Band Test ✓			
Maximum allowed Value:	50	Measured Value:	0.80
Wavenumber Accuracy Test - Polystyrene (30.0 Deg. C, Peak is T. corr.) ✓			
Sample Material:	Polystyrene		
Specified Peak:	1601.45 cm ⁻¹	Maximum Deviation:	0.50 cm ⁻¹
Measured Peak:	1601.28 cm ⁻¹		
Corrected Peak:	1601.35 cm ⁻¹	Measured Deviation:	0.10 cm ⁻¹
Photometric Reproducibility Test - Glass Filter A ✓			
Maximum Deviation[%]:	0.8	Measured Deviation[%]:	0.05
Overall Test Result = PASSED ✓			
Page 1 of 5		Page signature	
Date and Signature		Date and Signature	

Figure 12: First page of a PQ Test Protocol file for the liquid nitrogen-cooled detector.

9.2.3 Interferogram Peak Test

The interferogram peak amplitude test is a long-term stability test that compares the amplitude of a measured interferogram to that of the reference interferogram stored by the manufacturer. The amplitude of the reference interferogram corresponds to 100%. In the PQ test the same measurement is repeated and the interferogram amplitude of this test spectrum is compared to the interferogram amplitude of the reference interferogram. The amplitude of the test interferogram is indicated in the PQ test report relative to the reference value and must not fall below 70% of the reference interferogram.

9.2.4 Energy Distribution Test

The energy distribution test is a long-term stability test that compares the amplitude of a measured single-channel spectrum to that of a reference single-channel spectrum stored at installation. The integral over the total reference single-channel spectrum is set to 100%. If the power of the spectrometer source decreases, e.g., the distance between the two single-channel spectra will increase. Therefore, this test can be used to detect changes in the source power.

The decrease in the integral of the single-channel spectrum must not exceed 30% of the stored reference spectrum.

The reference spectra can be re-measured after instrument re-alignment or optical source changes.

9.2.5 Ice Band Test

If the vacuum of a liquid nitrogen-cooled detector decreases, the ice band caused by condensed water vapor is observable in the single-channel spectrum. The ice band test compares the integral in the ice band region ($3485\text{--}3050\text{ }cm^{-1}$) with the integral of the reference spectrum measured for the PQ test. The integral should not exceed a 50% difference from the manufacturer's reference. This test is part of the energy test.

9.2.6 X-Axis Frequency Calibration Test (Wavenumber Accuracy Test)

The x-axis frequency calibration test ensures that the frequency calibration of the instrument is within the specified limits.

If possible, water vapor is used to determine the wavenumber accuracy. Water vapor has an extremely narrow band, and allows the x-axis position to be measured to a very high degree of accuracy. The instrument uses a high-resolution setting to ensure that the water vapor band (at $1554.353\text{ }cm^{-1}$ for mid-IR) is completely resolved. The maximum deviation of the measured peak is $0.50\text{ }cm^{-1}$ from the specified band at $1554.353\text{ }cm^{-1}$.

It is also possible to measure the spectrum of the mounted filter, instead of water vapor, if the water vapor concentration is too low. In this case, a polystyrene standard sample can be used, with a specified peak at $1601.45\text{ }cm^{-1}$. The standard is on the internal validation unit.

9.2.7 Y-Axis Reproducibility Test (Photometric Accuracy)

The y-axis reproducibility test is a long-term stability test that compares the total intensity values of glass filter spectra to those of the reference spectra stored at installation.

A glass filter transmission spectrum is used to test the precision of the y-axis. The test spectra of glass filters are compared to the stored reference spectra by calculating the mean difference over a large spectral range ($4000\text{--}2000\text{ }cm^{-1}$ for mid-IR). The maximum deviation of the mean difference is 0.8%.

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