DEPARTMENT OF ECOLOGY
State of Washington

Standard Operating Procedure PTP005, Version 1.1

Thermo Scientific Nicolet iS5 FTIR Spectrometer

February 2023
Publication 23-03-201
[Recertified 2021]

Purpose of this Document

The Washington State Department of Ecology develops Standard Operating Procedures (SOPs) to document agency practices related to sampling, field and laboratory analysis, and other aspects of the agency's technical operations.

Publication Information

This SOP is available on the Department of Ecology's website at https://apps.ecology.wa.gov/publications/SummaryPages/2303201.html.

Ecology's Activity Tracker Code for this SOP is 22-007.

***Recommended citation*:**
Tuladhar, P. 2023. Standard Operating Procedure PTP005, Version 1.1: Thermo Scientific Nicolet iS5 FTIR Spectrometer. Publication 23-03-201. Washington State Department of Ecology, Olympia. https://apps.ecology.wa.gov/publications/SummaryPages/2303201.html. [Recertified 2021.]

Contact Information

For more information contact:

Publications Coordinator
Environmental Assessment Program
Washington State Department of Ecology
P.O. Box 47600
Olympia, WA 98504-7600

Phone: (360) 407-6764

Washington State Department of Ecology – https://ecology.wa.gov

- Headquarters, Olympia 360-407-6000
- Northwest Regional Office, Bellevue 425-649-7000
- Southwest Regional Office, Olympia 360-407-6300
- Central Regional Office, Union Gap 509-575-2490
- Eastern Regional Office, Spokane 509-329-3400

Original Author – Prajwol Tuladhar
Date – 07/19/2019

Original Reviewer – Chrissy Wiseman, Ken Nelson, Kari Trumbull
Date – 10/14/2019

Current Author – Prajwol Tuladhar
Date – 03/04/2021

Current Reviewer – Chrissy Wiseman, Ken Nelson, Kari Trumbull
Date – 04/16/2021

QA Approval – Arati Kaza, Ecology Quality Assurance Officer

Recertification Date – 07/28/2021

SIGNATURES AVAILABLE UPON REQUEST

Please note that the Washington State Department of Ecology's Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical and administrative experts. Their primary purpose is for internal Ecology use, although sampling and administrative SOPs may have a wider utility. Our SOPs do not supplant official published methods. Distribution of these SOPs does not constitute an endorsement of a particular procedure or method.

Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by the Department of Ecology.

Although Ecology follows the SOP in most instances, there may be instances in which the Ecology uses an alternative methodology, procedure, or process.

SOP Revision History

Revision Date	Revision History	Summary of changes	Sections	Reviser(s)
04/16/2021	1.1	Document Flow	All	Nelson, K., Trumbull, K., Wiseman, C.,

1.0 Purpose and Scope

1.1 This document serves as the Product Testing (PT) Standard Operating Procedure (SOP) for the operation of the Thermo Scientific Nicolet iS5 Fourier Transform Infrared Spectrometer (FTIR) utilizing the attenuated total reflectance (ATR) accessory.

2.0 Applicability

2.1 The SOP details the operational methods and parameters for data collection using the FTIR instrument. This SOP also includes methods for instrument safety, training, verification, quality assurance, and basic maintenance/troubleshooting of the FTIR as a screening tool. The ability of the instrument in determining the presence of these chemicals are dependent on the availability of the standards or spectra.

2.2 Product testing XRF screening data are collected to help select samples for further confirmatory laboratory analysis. This method is designed as a rapid screening tool, and is not accredited.

3.0 Definitions

3.1 Absorption –The transfer of energy from an electromagnetic radiation to molecules that are present in the path of the radiation.

3.2 Attenuated Total Reflectance (ATR) – A sampling technique used in conjunction with infrared spectroscopy which enables samples to be examined directly in the solid or liquid state without further preparation.

3.3 ATR Correction – A correction applied to a spectrum obtained using the ATR diamond crystal accessory that accounts for absorption band shifts and variation in depth of penetration. This allows for the acquired spectrum to be compared against the spectra collected using standard transmission techniques.

3.4 Background spectrum – A signal-beam spectrum captured without a sample in place. This spectrum captures the characteristics of the environment of the spectrometer, including the source, detector, and atmospheric conditions.

3.5 Component – Individual piece or part of a product containing different colors, functions, and/or material (For example, a sweater may be comprised of several components such as, the woven fabric, a button, and a zipper).

3.6 Component Sample – Component identified to be screened as a sample using the FTIR. Components cataloged in the PTDB receive an alpha-numeric identification (ID) that is used as the FTIR component ID (e.g. TG-1-1-1).

3.7 Fourier Transform – The mathematical operation used to convert an interferogram that contains data in a time domain to a single beam spectrum that contains data in a frequency domain in order to reveal the response at all frequencies within a spectral range.

3.8 Fourier Transform Infrared Spectroscopy – A spectroscopic technique that uses an interferometer to collect data and a Fourier transform to digitally process the data.

3.9 Infrared Radiation – A region of the electromagnetic spectrum extending from approximately 10 to 14000 cm-1. The Far Infrared extends from 400 to 10 cm-1, the Mid Infrared extends from 400 and 4000 cm-1, and the Near Infrared extends from 4000 to 14000 cm-1.

3.10 Infrared Spectroscopy – A spectroscopic technique used to identify molecular structure using electromagnetic radiation in the mid-infrared region. A sample placed in the path of an infrared beam absorbs some of the radiation and transmits the rest. Absorption of this infrared radiation is a function of the vibrational or rotational energy of molecular structures present in the sample. Since, different molecular structures absorb at different frequencies each molecular structure has a unique molecular fingerprint which can be used to identify chemical compositions of a sample.

3.11 Initial Demonstration of Capability (IDC) –IDC is the process of documenting a new users' ability to operate the FTIR using the ATR accessory. A new operator must be able to collect a complete set of spectra with the instrument under minimal supervision and complete the Initial Demonstration of capability (IDC) Form for FTIR. The analysis consists of running a performance verification check, collecting spectrum of a check sample, and five random samples using the ATR accessory. A copy of the completed IDC form with appropriate signatures should be stored in the FTIR Performance Verification (PV) and IDC Records Log Book in the PT Room. The trainee should also keep a copy for their own records.

3.12 Interferogram – An electronic recording of an optical interference pattern that plots infrared intensity versus the optical path difference.

3.13 Interferometer – An optical device used to measure all the infrared frequencies simultaneously. It produces an interferogram that holds information on the rotational and vibrational frequencies of a particular molecular structure which is translated to a spectrum using the Fourier Transform

3.14 Spectrum – In chemistry, a spectrum refers to the characteristic wavelength of electromagnetic radiation that is emitted or absorbed by an object or substance, atom or a molecule.

3.15 Wavelength – A unique span of measured electromagnetic energy from crest to crest. The shorter the wavelength the more energy it contains.

3.16 Wavenumber – The number of waves that fit in a centimeter, the inverse of a wavelength. Wavenumbers are expressed in cm-1 and are proportional to energy. They are most often represented in the X-axis of a spectrum.

4.0 Personnel Qualifications/Responsibilities

4.1 Staff must receive training and perform a successful Initial Demonstration of Capability (IDC) prior to operating the FTIR. A qualified operator will provide the training. A qualified operator is a permanent employee that has been trained to operate the FTIR by another qualified operator.

4.2 Employees may also receive training through qualified Thermo technicians and or other formal training opportunities to become a qualified operator.

4.3 All operators should review the FTIR Nicolet iS5- Getting Started, Fast Facts Sheet, and Site and Safety information booklets available in the Product Testing (PT) Preparation Room (OL-21).

4.4 For new operator training, the overseeing qualified operator will be responsible for supplying the new operator with the following:

4.4.1 Initial Demonstration of capability (IDC) Form for FTIR

4.4.2 A copy of this SOP

4.4.3 Hands on training

4.4.4 Documents listed in Section 4.3

4.4.5 Administering IDC analysis

4.4.6 Continued oversight and review of the operator's first several FTIR operation sessions.

4.4.7 The qualified operator will also provide a signature to sign off on the IDC Form for FTIR when complete.

4.5 The completed training documentation shall be submitted to the PT Management for a final signature and record retention. A copy will be stored in the PT Room in the FTIR Performance Verification (PV) and IDC records Logbook. The trainee should also keep a copy for their own records.

4.6 Upon completion of the training with a successful IDC, and when all appropriate signatures are obtained, the operator is qualified to collect data using the FTIR.

4.7 IDC must be performed initially for every new operator. Ongoing demonstration will be performed for each current operators at the recertification of this SOP, when there is a change to the procedure, and at deployment of a new instrument. Use the IDC Form for FTIR to document on-going demonstration of capability.

4.8 Personnel Responsibilities:

4.8.1 Always operate the FTIR in accordance with this SOP. The SOP is based on the guidance established in accordance with manufacturer's instructions and recommendations.

4.8.2 All Ecology health and safety guidelines described in Section 9.0 must be followed as specified.

4.8.3 The PT Sample Collection and Processing SOP should be followed for the tool cleaning protocols, and deconstruction and preparation of component samples. This SOP which is specific to preparing component samples for XRF screening is also applicable for preparing component samples for screening with the FTIR.

4.8.4 Study specific Quality Assurance Project Plans (QAPPs) shall be reviewed for additional product deconstruction, preparation, and analysis procedures prior to the beginning of screening component samples.

4.8.5 The FTIR operator will be responsible for verifying the completeness and correctness of data acquisition in accordance with this SOP and any additional guidance pre-established in study-specific QAPPs. Errors made during data collection (e.g., sample ID typos, incomplete scans) are recorded in the FTIR Error Log during the data collection session.

4.8.6 All corrections made to the file mentioned in section 4.8.5 will be documented as a case narrative (word document) to be saved to the PTDB as an attachment. The file should be saved with a standard filename format that says "Project Name FTIR case narrative" and has the analysis date. (Project Name FTIR Case Narrative-mm-dd-yyyy). (For Example: ProjectNameFTIRCaseNarrative10-21-2019)

4.8.7 The project manager (PM) will inspect datasets and case narratives uploaded to the Product Testing Database (PTDB) to verify accuracy, completeness, and adherence to the specific study criteria.

5.0 Equipment, Reagents, and Supplies

5.1 Thermo Scientific Nicolet iS5 FTIR (S/N: ASB1502454)

5.2 iTR-iD5 ATR accessory with high pressure tower/clamp (Thermo P/N 869-128600)

5.3 Solid (nylon) press tip specific for use with the diamond crystal (Thermo P/N 869-128600)

5.4 Diamond iTR-iD5 3-Bounce ATR crystal (Pike P/N: 025-2114) or Diamond iTR-iD5 Hastelloy Diamond Plate (single-bounce ATR crystal; Thermo Item # 869-129400)

5.5 OMNIC software and Adobe Acrobat Reader

5.6 Lab samples, referred to as CHK SMP 1, and CHK SMP 2 (previously analyzed in the lab for known amounts of ortho-phthalate analytes)

5.7 Crystal cleaning agents: 24% ethanol (recommended)

5.8 De-ionized water (DIW) in squirt bottles.

5.9 Kim Wipes, or other optical grade wipes

5.10 Nitrile gloves

5.11 Forceps

5.12 Glass pipettes and pipette bulbs for transferring liquid samples.

5.13 FTIR Instrument Log (Maintenance Records)

5.14 IDC and FTIR Performance Verification Record Log (Record of IDC and Performance Verification (PV) Tests)

5.15 FTIR Daily Log (Record of samples scanned)

5.16 FTIR Error Log (Record of errors during screening)

6.0 Summary of Procedure

6.1 Spectra Collection Pre-Checks:

6.1.1 The humidity level inside the instrument must be monitored routinely to maintain proper operation of the instrument. A desiccant cartridge located in the back of the instrument maintains the humidity level of the instrument. The operator should check the desiccant indicator and document the desiccant indicator color on the FTIR Daily Log before running a screening session on the instrument.

6.1.2 Note: Replace the desiccant when the humidity indicator turns pink (can be light pink or almost white) or the software displays a message that the internal humidity is above 50%. Also replace the paper humidity indicator each time the desiccant is replaced. Any time the desiccator is replaced it should be recorded in the FTIR Instrument Log.

6.1.3 A blue light on the front panel of the instrument indicates that it is ON. For an instrument that is turned off, turn it on using the toggle switch at the back of instrument. The instrument should warm up for a minimum of 30 minutes prior to operation.

6.1.4 Some wireless devices may affect instrument performance. Move all wireless devices at least 2.0 m (6.5 ft.) away from the instrument.

6.1.5 Refer to Appendix A to get acquainted with the different parts of the instrument. The instrument is equipped with interchangeable accessories to run the spectrometer in transmission (iD1) and Attenuated Total Reflectance (iD5) mode. For procedures outlined in this SOP, only the iD5 ATR accessory is used.

6.1.6 The product testing prep room has two interchangeable diamond crystals that can be used with the iD5 ATR accessory: the single-bounce diamond and the triple-bounce diamond.

6.1.7 The single-bounce crystal is more durable and is recommended for use with any type of materials. Ideal materials that can be analyzed with the single-bounce crystal are homogeneous solid samples (rigid or flexible) such as laminates, plastics, rubbers, powders, as well as free flowing or viscous liquids.

6.1.8 The triple-bounce crystal is more sensitive (can generate signals at lower concentrations) but is recommended only for use with homogeneous bendable solids and free flowing liquids. The manufacturer recommends using only the plastic press tip with the triple-bounce crystal.

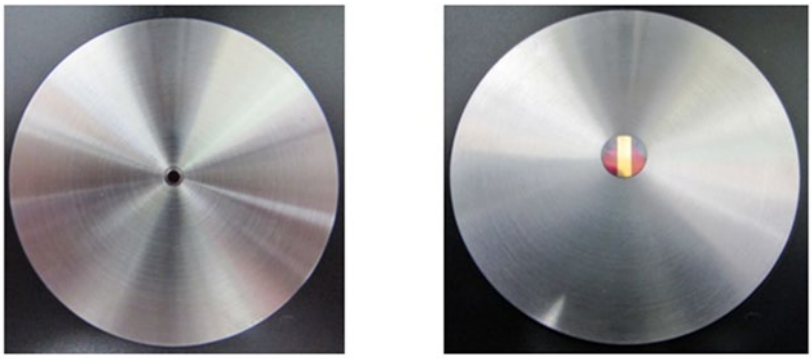


Figure 1. The single-bounce crystal plate with a small sampling aperture (Left) & the triple-bounce crystal plate with a larger aperture and a gold line across the center. (Right)

6.1.9 It is recommended that abrasive liquids and any other liquids (viscous or free flowing) that cannot be cleaned off with an alcohol-based solvent not be analyzed with either crystal.

6.1.10 The project manager may choose to use one or both crystals depending on the type of materials being analyzed in a study. For more information on choosing a crystal refer to videos available in the "reference links" sections of the "Screening Instruments" page on the Product Testing SharePoint Site.

6.1.11 At the adjoining computer, log on with your User Name and password, then open the "OMNIC" icon on the Desktop. The software works from any user ID.

6.1.12 Check the System Status by clicking on the System Performance Verification shield icon in the upper right corner of the Omnic software interface.

6.1.13 If the System Status shield is green move on to the next step. If the System Status shield is yellow it may indicate that the system identified a minor problem, which should be viewed to determine the possible effects on the data collected. Follow the instructions on the System Status Overview which will identify the issues to be addressed. If the System Status shield is red, it warns that the system is out of specification, or a subsystem failed. It may also indicate that a Performance Verification (PV) test may be required.

6.1.14 Please refer to Appendix B for checking system status and running a PV test. Requirement for running a PV test is auto-programmed to be triggered at the beginning of each month.

6.1.15 Prior to any data collection using the ATR, the surface of the crystal should be cleaned.

6.1.16 For initial cleaning and cleaning between samples, dampen a Kim wipe with a small amount of 24% ethanol, and then gently wipe the surface of circular crystal and surrounding sample platform from top to bottom or left to right. (Never scrub the surface of the diamond crystal in a circular motion.)

6.1.17 Note: Acetone must never be used on the diamond ATR. Use of incompatible solvents, or samples containing such solvents, will damage/delaminate the ATR crystal. Delamination of the crystal by a solvent is not covered under the manufacturer warranty.

6.1.18 To clean the solid/plastic press tip, gently pat the underside with wetted Kim Wipe.

6.1.19 Even though it is highly unlikely that contamination will pass or be detected during testing of solid materials on the FTIR from the press tip, it is best practice to clean the press tip in between each sample.

6.1.20 Note: While running the FTIR in ATR mode with the triple-bounce crystal, the pressure clamp should not be used without a plastic tip. It may damage the ATR crystal. The pressure clamp is not necessary when running liquid samples.

6.2 Experiment Parameters Setup:

6.2.1 Conduct the following steps before every scanning session to ensure correct setup for data collection:

6.2.2 For plastics screening and phthalates analysis the default FTIR operating configuration is set as “Phthalate Analyzer” and the default experiment is “Phthalates and Plastics Screening.” The analyst should verify that the correct experiment is loaded by checking the experiment heading located at the top of the OMNIC screen.

6.2.3 To set scan parameters: Collect – Experiment set up—Collect.

6.2.4 No. of scans default is 16. If different, set it to 16.

6.2.5 Resolution: 4 is fine. If small amplitude noise is a problem in your spectra, try increasing the number to 6 or more.

6.2.6 Final format: Absorbance

6.2.7 Correction: None

6.2.8 Check the box for “Preview data collection,” the other three boxes should be unchecked.

6.2.9 File Handling: Uncheck box for “save automatically.” Verify that the “save interferogram” box is checked.

6.2.10 Background Handling: Choose “Collect background after 120 minutes.” This directs the software to generate a prompt for obtaining a new background at intervals of 120 minutes. Make sure to collect a new background when the prompt occurs.

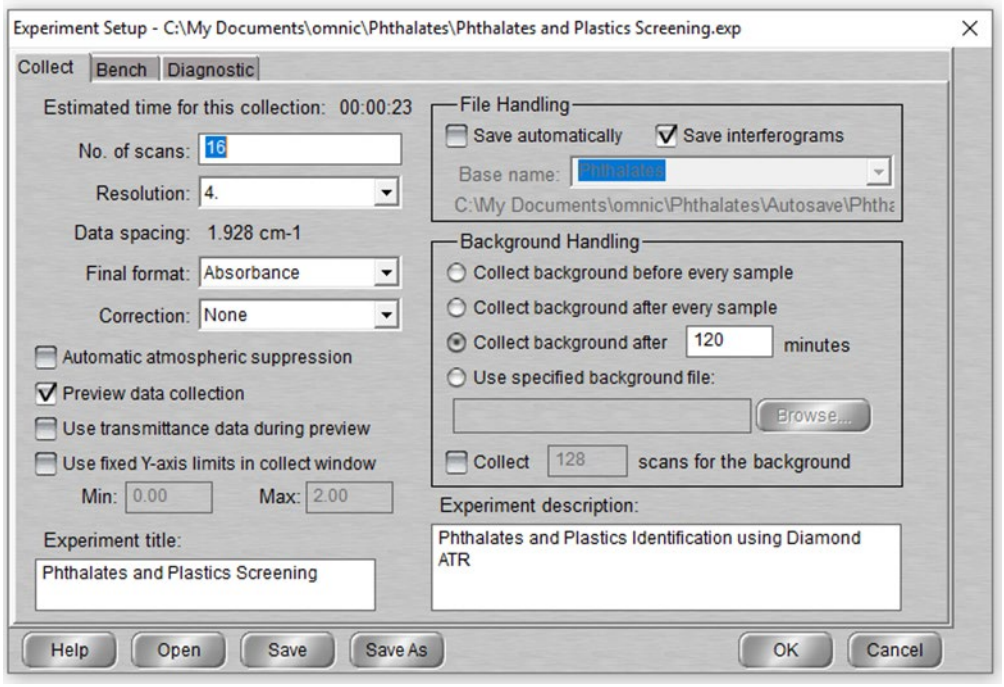


Figure 2. Omnic experiment setup screen for setting up data collection parameters

6.2.11 To check beam, click the bench tab (if window is closed then reopen by 'Collect' – Experiment set up – Bench): If something is wrong with the beam, an error message will pop up. Call the Thermo rep for troubleshooting when this occurs. For ATR, click peak to peak button to verify the height of the interferogram which should be between 6 and 8.

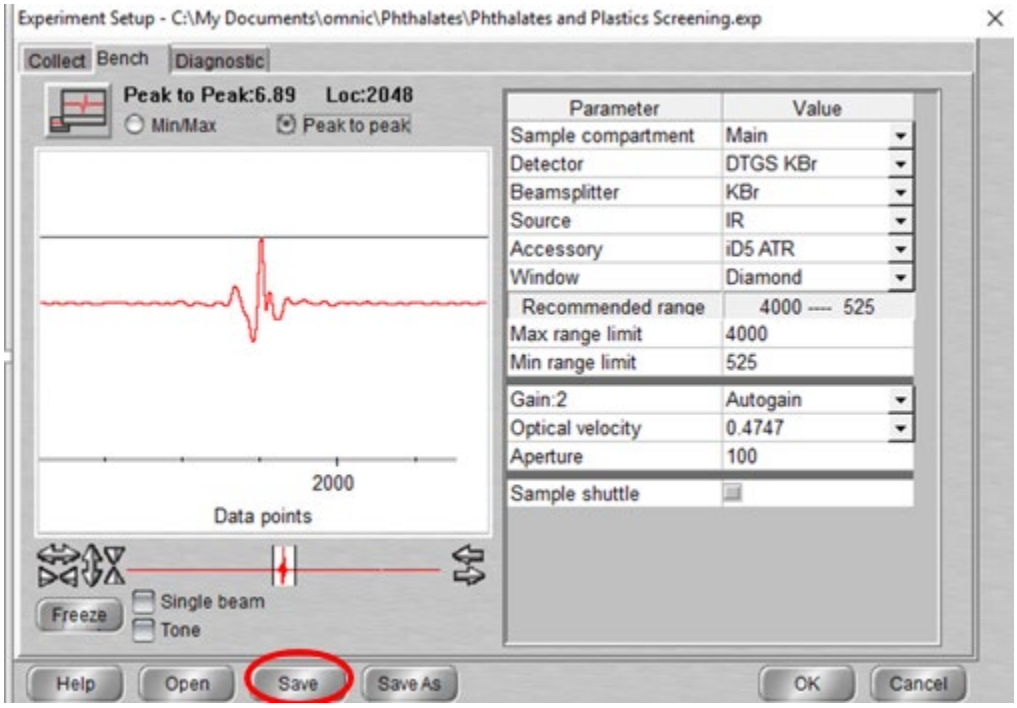


Figure 3. Omnic bench tab screen to check peak to peak height

6.2.12 Click “Save” button to ensure that any changes made have been saved to the experiment setup, and then click OK to exit.

6.3 Background Collection:

6.3.1 Collection of a background signal is essential for the instrument to generate a spectrum for a sample screened. Make sure that the crystal surface is clean and dry before collecting a background. The pressure clamp needs to be loosened to ensure that the press tip is not touching the crystal surface when the background is being collected. Move the solid press tip away from the sample plate.

6.3.2 Note: Collect a new background at the beginning of each session. A new background should be collected at an interval of 120 minutes. (Follow system prompt.)

6.3.3 Click on either one of Col Bkg icons, and press OK when prompted by the dialog box to proceed with the background collection.

Figure 4. Omnic menu screen with the collect background button highlighted

6.3.4 A preview screen with the background is displayed on the screen. Hit “Start Collection” (in the upper right corner) to start the background collection process.

Figure 5. Omnic screen start collection button to begin data collection

6.3.5 After the 16 scans have been completed, a dialog box will appear; select “No.” It is not necessary to save the background for it to auto correct.

Figure 6. Add to window popup

6.3.6 Note: The OMNIC Specta software ratios the collected background to the sample spectrum to get rid of the absorption curve from the diamond and it also zeroes out any water vapor and CO2 in the sample spectrum.

6.3.7 Note: Water vapor and CO2 peaks will start appearing in your sample spectra as the concentrations in the air can fluctuate near the spectrometer and, could affect the background. Water in particular can interfere with the background spectra, for more details see excerpt from the Pre-Webinar Training Guide 3 - "When do you need a new background" provided by Ecology Center (Appendix D).

6.4 Choosing the correct library setup:

6.4.1 Before screening samples ensure that the correct spectral libraries have been selected. Depending on the class of chemicals that are to be identified different libraries can be selected. The optimum selection of libraries for identifying plastic class and running the DI water blank and phthalate check samples have been predetermined. The PM may provide instructions on a different library selection depending on a particular study.

6.4.2 For plastic class identification:

6.4.2.1 Analyze – Library Setup –Select "Polymer class identification" from the "Available search libraries and groups" table on the left and click Add>>. A list of selected Polymer Libraries is then displayed in the Search libraries and groups table. Ensure the following libraries are displayed on the right table:

6.4.2.1.1 HR Aldrich Alcohols and Phenols

6.4.2.1.2 HR Hummel Polymer and Additives

6.4.2.1.3 HR Polymer Additives and Plasticizers

6.4.2.1.4 HR Specta Polymers and Plasticizers by ATR

6.4.2.1.5 HR Specta Polymers and Plasticizers by ATR-Corrected

6.4.2.1.6 Hummel Polymer Sample Library

6.4.2.1.7 SRL PVC Plasticizer Analysis

6.4.2.2 Remove any other libraries by selecting from the table on the right and clicking <<Remove. Click OK to exit the library setup screen.

6.4.3 For collecting Di water blank spectrum and running phthalate check samples:

6.4.3.1 Click Analyze – Library Setup –Select "Phthalates analysis" from the "Available search libraries and groups" table on the left and click Add>>. A list of selected plasticizer and additive libraries are then displayed in the Search libraries and groups table. Ensure the following libraries are displayed on the right table:

6.4.3.1.1 HR Aldrich Alcohols and Phenols

6.4.3.1.2 HR Polymer Additives and Plasticizers

6.4.3.1.3 Hummel Polymer Sample Library

6.4.3.1.4 SRL PVC Plasticizer Analysis

6.4.3.2 Remove any other libraries by selecting from the table on the right and clicking <<Remove. Click OK to exit the library setup screen.

6.5 Collecting a DI water blank spectrum and running phthalate check samples:

6.5.1 Prior to running any unknown samples, a check verification of the instrument is conducted by assessing the instruments' ability to identify the class of ortho-phthalates using commercially available standards or lab samples that have known amounts of ortho-phthalates.

6.5.2 *Note: A De-ionized (DI) water blank is collected each time before running a set of check verification samples.

6.5.3 Running a check verification consists of completing two different tasks:

6.5.3.1 At first, the previously analyzed lab samples (CHK SMP 1 and CHK SMP 2 available in the Product Testing Prep Room ROL-21) with a known concentration of an ortho-phthalate are scanned using the FTIR. The specific peaks associated with these ortho-phthalates are then identified as being present. Information on commonly known ortho-phthalate peaks can be found in Appendix C.

6.5.3.2 Next the scanned spectrum of the CHK SMP1 and CHK SMP 2 are compared to other spectra in a commercial library to obtain a positive match for the known lab sample ortho-phthalate. The scan will also most likely identify a positive match for other ortho-phthalates. Since the screening instrument is to be used to identify only the presence or absence of ortho-phthalates, all identified matches should have a match % of greater than 60%.

6.5.4 *Disclaimer: Previously analyzed laboratory samples (CHK SMP1 and CHK SMP 2) used in the process for verifying the instrument's ability to identify the class of ortho-phthalates were analyzed using methods that have been updated by the WA State Accreditation Board. These samples are used to roughly demonstrate the instrument's ability to screen for phthalates and are not to be considered as standards.

6.5.5 Print the FTIR Sample Check Verification Log available in the Product Testing SharePoint site. This log is to be filled out with information obtained from both Check Verification Samples.

6.5.6 Before collecting the spectrum of the DI water blank and running phthalate check samples ensure that the correct spectral libraries have been selected.

6.5.6.1 Click Analyze – Library Setup –Select "Phthalates analysis" from the "Available search libraries and groups" table on the left and click Add>>. A list of selected plasticizer and additive libraries are then displayed in the Search libraries and groups table. Ensure the following libraries are displayed on the right table:

6.5.6.1.1 HR Aldrich Alcohols and Phenols

6.5.6.1.2 HR Polymer Additives and Plasticizers

6.5.6.1.3 Hummel Polymer Sample Library

6.5.6.1.4 SRL PVC Plasticizer Analysis

6.5.6.2 Remove any other libraries by selecting from the table on the right and clicking <<Remove. Click OK to exit the library setup screen.

6.5.7 After the background (see section 6.3) has been collected click on Col Smp icon to begin collecting the spectrum.

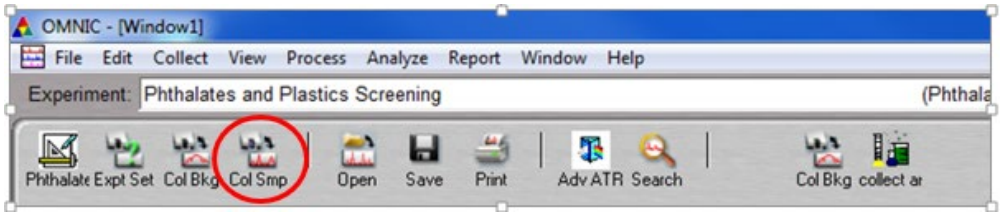


Figure 7. Omnic menu screen with the collect sample button highlighted

6.5.8 A prompt for entering the spectrum name appears. Enter the sample name “DI water Blank” in front of the time date stamp, and Press OK.

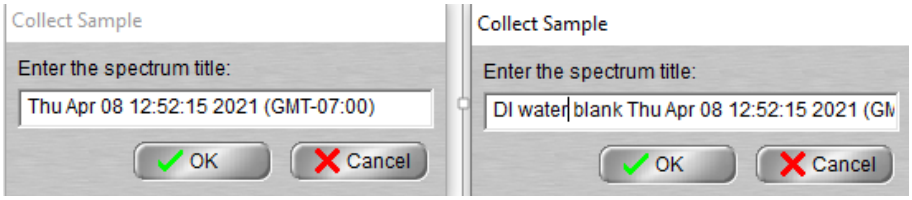


Figure 8. Omnic popup screen for entering spectrum name

6.5.9 A prompt appears “Please prepare to collect the sample spectrum.” Click OK to proceed.

6.5.10 A preview screen with a flat baseline should appear from 4000 to approximately 650 wavenumbers. If the ATR crystal is not clean the baseline may appear to have peaks. In case of unwanted peaks, clean the crystal and sample platform with 24% ethanol and wait for it to air dry.

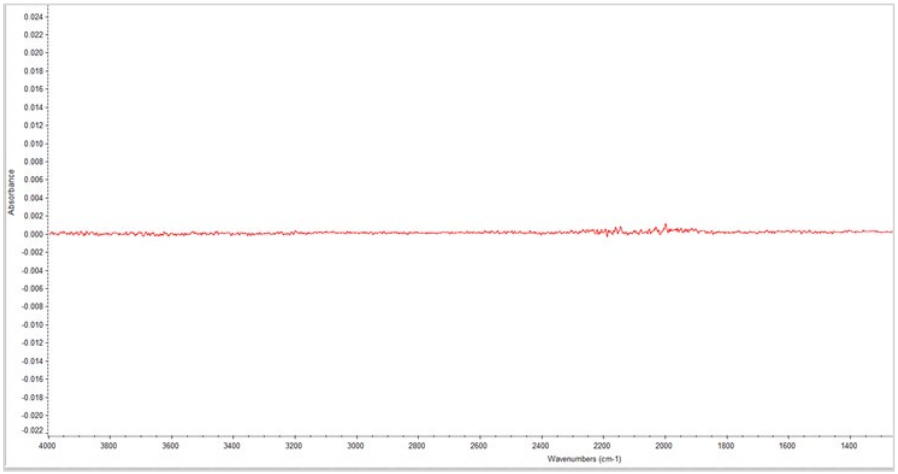


Figure 9. Omnic data collection preview screen

6.5.11 Once a flat baseline is achieved, squirt a few drops of DI water on the crystal surface, and press start collection button in upper right corner.

6.5.12 Note: The solid/plastic press tip is not necessary for liquid samples.

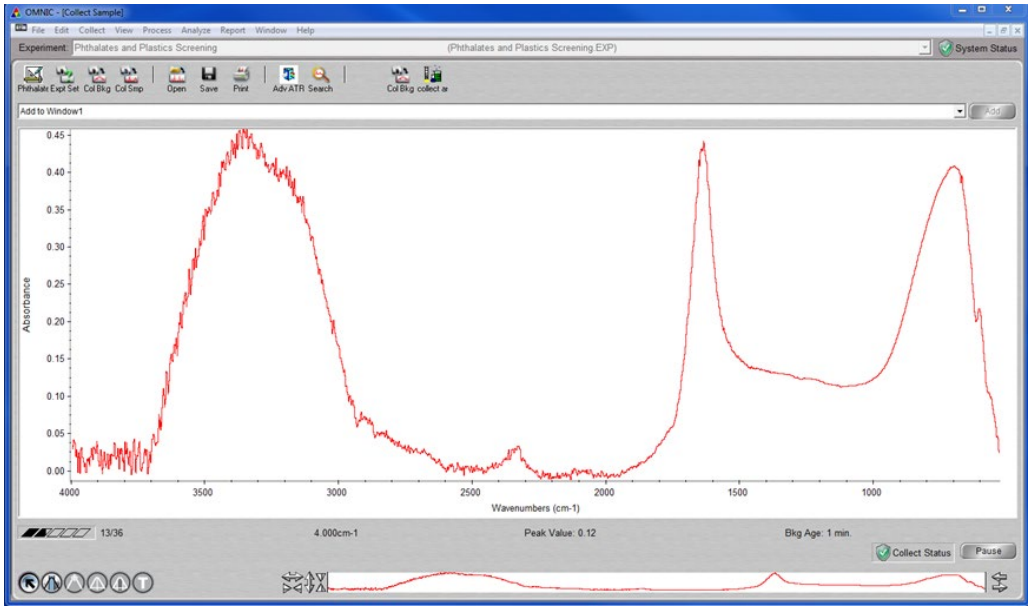


Figure 10. Omnic data collection screen during data collection

6.5.13 After scanning is completed choose "Yes Add to Window" option on the popup that appears.

6.5.14 A prompt may appear asking for a window label. Click OK to automatically choose the default window label. (window 1, window 2….etc)

6.5.15 Correct the baseline of the spectrum by clicking on the Process Tab and choosing the Automatic Baseline Correct option.

6.5.16 A new baseline corrected spectrum highlighted in red is added to the current window.

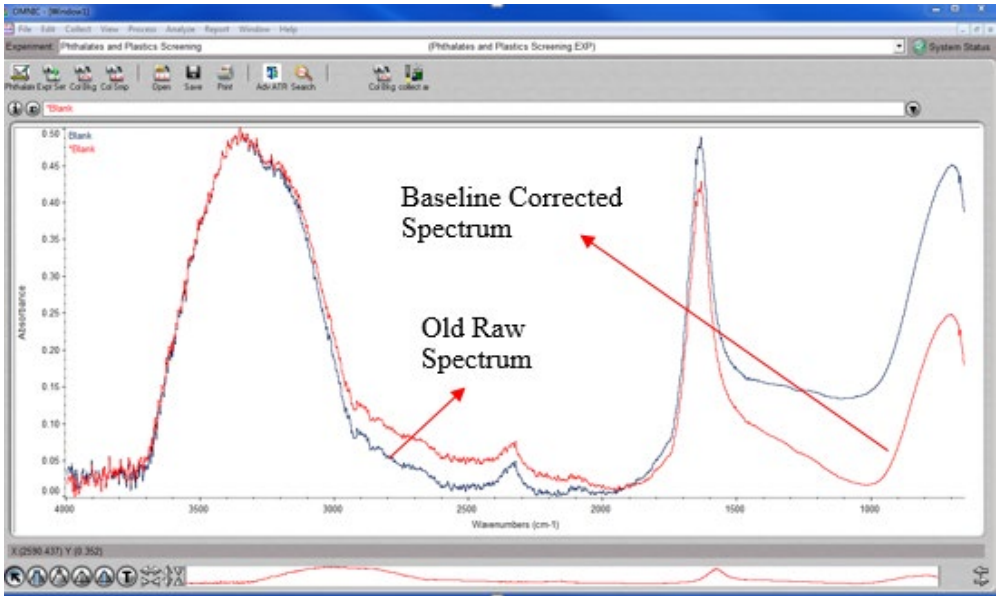


Figure 11. Omnic data collection screen with a new baseline corrected spectrum

6.5.17 Hide the old raw spectrum (spectrum without the corrected baseline) from the current window by first clicking on it to highlight it in red, and then pressing CTRL+H.

6.5.18 Run the spectral match by pressing on the Analyze Tab and clicking on the Search button.

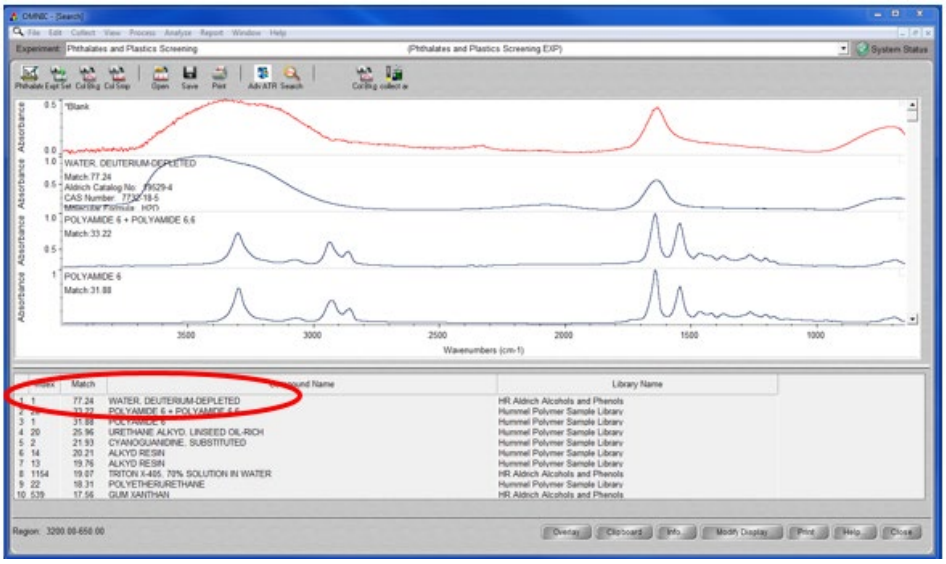


Figure 12. Omnic spectral database search results screen

6.5.19 Once the result is obtained verify that water (H_2O) Deuterium-Depleted is identified in the results and has the highest match % (at minimum, greater than 60%). Also identify that there are no false phthalate matches.

6.5.20 Save a digital copy of the spectrum with identified matches as a PDF by clicking on the print button and choosing the Adobe PDF option.

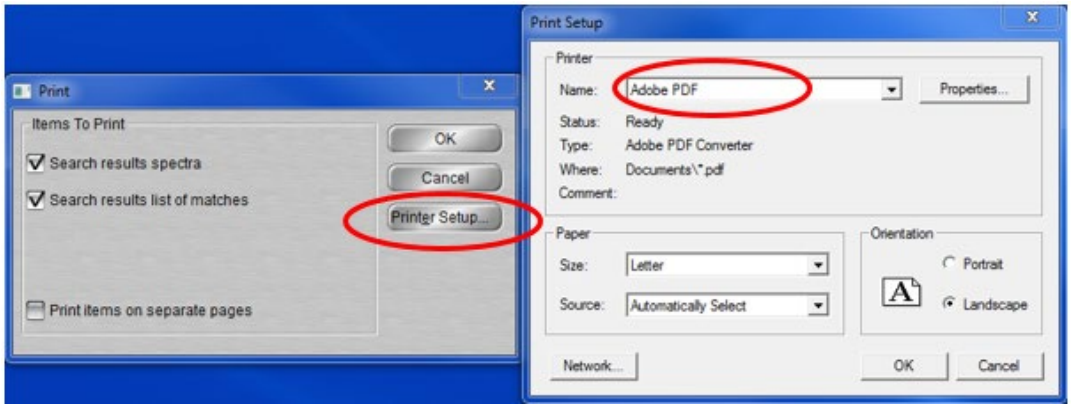


Figure 13. Using the print to PDF function in Omnic

6.5.21 Note: Using the Save function in the software does not save any analysis results such as labeled peaks or library comparisons. Print to PDF option works best for saving the spectrum with results.

6.5.22 Save the PDF file with a standard filename that includes the date, sample ID. (mmddyyyy Sample ID). For example (10102019 DI Water Blank)

6.5.23 The file should be saved in a FTIR folder, in the specific study folder created by the PM in F: drive. If there is no FTIR folder under the specific study create a new one. F:\EAP\TSU\ProductTesting\SpecificStudyName\FTIR

6.5.24 Once the PDF has been saved print a hard copy for records.

6.5.25 Save the .SPA file by clicking on the File tab located on the top left side of the screen and choosing “Save As.” Use the same filename format and location mentioned above to save the .SPA file in the same study specific folder.

6.5.26 Once the DI water blank has been screened, gently wipe the DI water off the crystal surface using a Kim wipe. Dampen a Kim wipe with a small amount of 24% ethanol and gently wipe the surface of circular crystal and surrounding sample platform from top to bottom or left to right. (Never scrub the surface of the diamond crystal in a circular motion)

6.5.27 Close the result screen by clicking the **Close** button on the bottom right of the screen.

Figure 14. Highlighted close button at the bottom of the library match screen

6.5.28 After closing the results or library match screen, the processing window displays the original spectrum and all other spectra obtained (Baseline corrected, and ATR corrected). Just close this window to start a new sample. Make sure to choose the correct close button as shown in figure below. Choosing the wrong close button may terminate the OMNIC program.

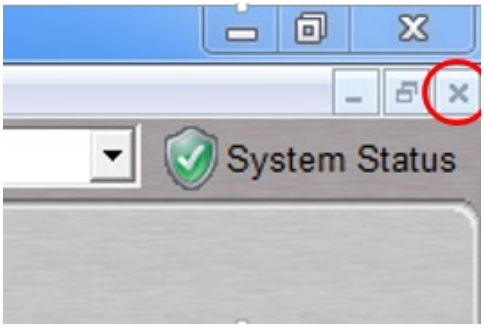


Figure 15. Close icon for closing the current data collection screen

6.5.29 Click on Col Smp button to begin collecting the spectrum for the 1st check verification sample.

6.5.30 A prompt for entering the spectrum name appears. Enter the check sample name “CHK SMP 1” in front of the time date stamp and Press OK.

6.5.31 A prompt appears “Please prepare to collect the sample spectrum.” Click Ok to proceed.

6.5.32 A preview screen with a flat baseline should appear from 4000 to approximately 650 wavenumbers. If the ATR crystal is not clean the baseline may appear to have peaks. In case of unwanted peaks, clean the crystal and sample platform with 24% ethanol and wait for it to air dry.

6.5.33 Once a flat baseline is achieved, use dedicated tweezers (tweezers should be cleaned with Kim Wipes wetted with 24% ethanol in between each use) to carefully place the 1st check sample CHK SMP 1 on the crystal surface and tighten the pressure clamp. Best results are obtained when the sample is pressed flat against the crystal. Once the sample is pressed flat on the surface the pressure tower needs to be tightened till a click is heard or felt.

6.5.34 Note: Be careful to keep an eye on the solid press/plastic tip while tightening. To ensure that the plastic tip does not break, it should rest flat on the surface of the material and stay in a horizontal alignment as much as possible when tightening.

6.5.35 Press start collection button in upper right corner to begin scanning.

6.5.36 After scanning is completed choose "Yes Add to Window" option on the popup that appears.

6.5.37 A prompt may appear asking for a window label. Click OK to automatically choose the default window label. (Window 1, window 2…, etc.)

6.5.38 Correct the baseline of the spectrum by clicking on the Process Tab and choosing the Automatic Baseline Correct option.

6.5.39 A new baseline corrected spectrum highlighted (*is corrected spectrum) in red is added to the current window.

6.5.40 Hide the old raw spectrum (spectrum without the corrected baseline) from the current window by first clicking on it to highlight it in red, and then pressing CTRL+H.

6.5.41 Click on the Analyze Tab and select 'Find Peaks' button to label the peaks in the spectrum. A black line runs through the middle of the screen and labels all the peaks above it with their respective wavenumbers. Click at the bottom of the spectrum window above the Y-axis to move the line to the bottom in order to label smaller peaks.

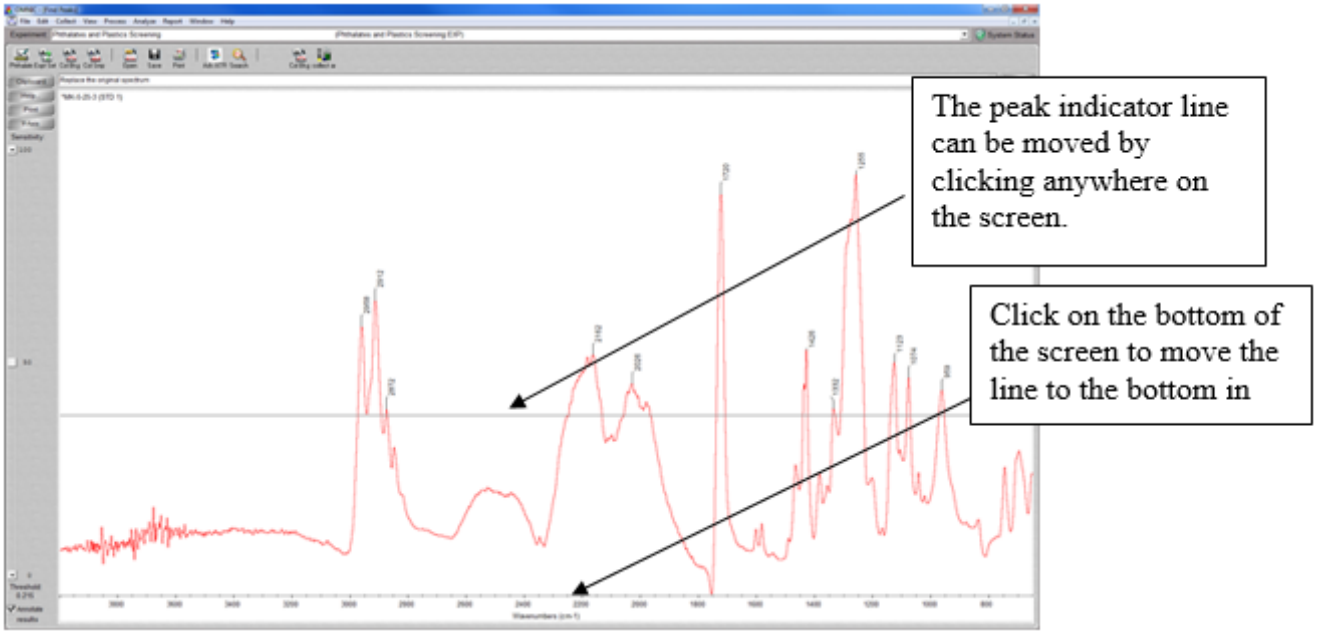


Figure 16. Using the find peaks function to analyze the spectrum

6.5.42 Check to verify that all the peaks on the check verification log table below have been identified. Some desired peaks on the check verification log table that do not have a peak label, (most often peak at 1595 wavenumber) can be added by increasing the threshold on the sensitivity bar located to the left of the screen.

Wavenumber (cm-1)	Measured Wavenumbers CHK STD 1	Measured Wavenumbers CHK STD 2	Result
Peak at 1725 ± 5			
Peak at 1595 ± 5			
Peak at 1580 ± 5			
Peak at 1120 ± 5			
Peak at 1070 ± 5			
Peak at 740 ± 5			
Diisononyl phthalate >60%			
Di(2-ethylhexyl) phthalate >60%			

Table 1. Check verification log

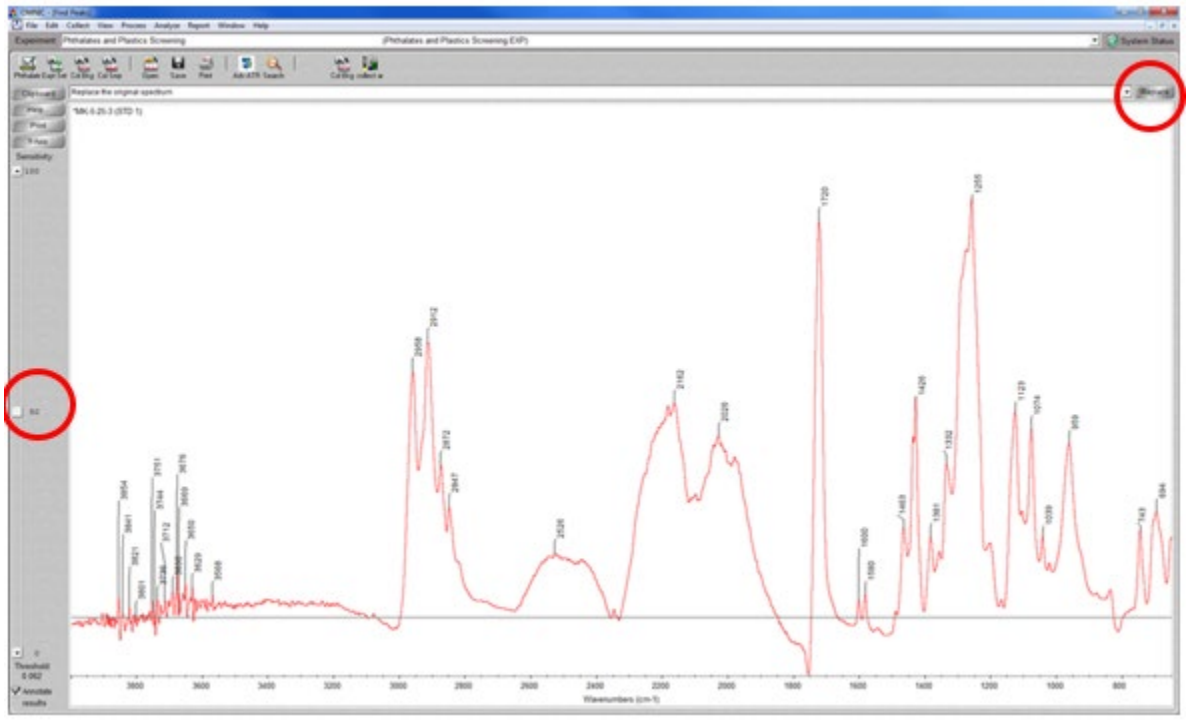


Figure 17. Increasing the sensitivity to find lower peaks in the spectrum, and use replace button to add the labeled spectrum to the Omnic data collection screen

6.5.43 Once all the desired peaks have been labeled, click on the Replace button located on the top right corner of the OMNIC window to add the processed spectrum with identified peaks to the current window.

6.5.44 Adjust the window to include only the wavenumbers associated with phthalate peaks by pressing CTRL+D and choosing the start of the X-axis limit at 1800 wavenumbers.

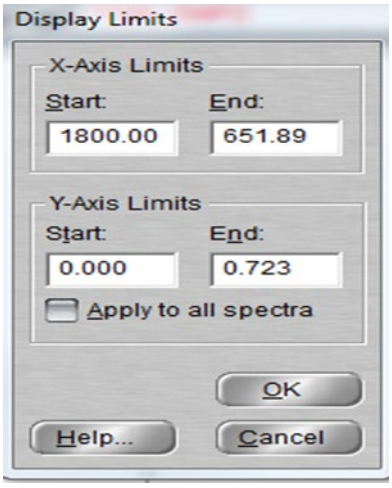


Figure 18. Popup for adjusting the X-axis view scale

6.5.45 Save a digital copy of the spectrum with identified peaks as a PDF by clicking on the print button and choosing the Adobe PDF option.

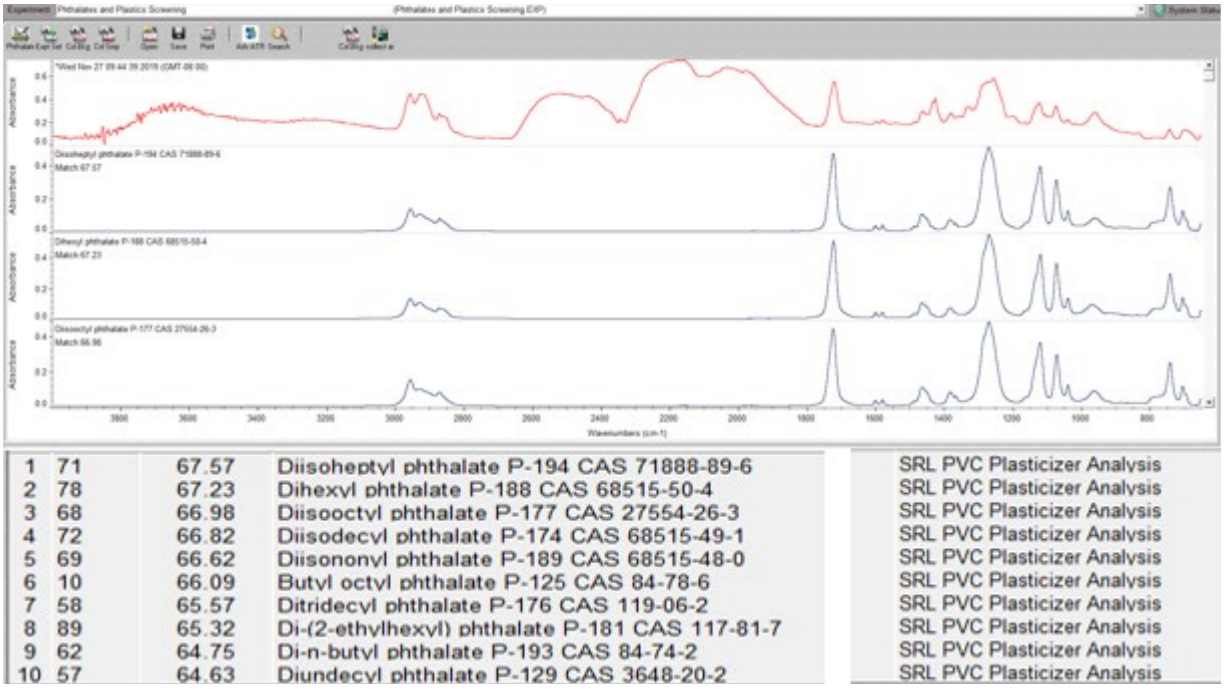
6.5.46 Save the PDF file with a standard filename that includes the date, sample ID, task description (mmddyyyy SAMPLE ID TASK DESCRIPTION) For example (10102019 CHK SMP1 FIND PEAKS)

6.5.47 The file should be saved in a FTIR folder, in the specific study folder created by the PM in F: drive. If there is no FTIR folder under the specific study create a new one. F:\EAP\TSU\ProductTesting\SpecificStudyName\FTIR

6.5.48 Also, save the .SPA file by clicking on the File tab located on the top left side of the screen and choosing “Save As.” Use the filename format (mmddyyyy Sample ID) to save the .SPA file in the same study specific folder. For example (04202021 CHK SMP1)

6.5.49 Print a copy and fill out the measured wavenumbers for CHK SMP 1 in the FTIR Sample Check Verification Log.

6.5.50 The next step is to compare the spectrum of the collected check verification sample against the spectra available in commercial spectral library databases. Run the spectral match by pressing on the Analyze Tab and clicking on the Search button.



1	71	67.57	Diisoheptyl phthalate P-194 CAS 71888-89-6	SRL PVC Plasticizer Analysis
2	78	67.23	Dihexyl phthalate P-188 CAS 68515-50-4	SRL PVC Plasticizer Analysis
3	68	66.98	Diisooctyl phthalate P-177 CAS 27554-26-3	SRL PVC Plasticizer Analysis
4	72	66.82	Diisodecyl phthalate P-174 CAS 68515-49-1	SRL PVC Plasticizer Analysis
5	69	66.62	Diisononyl phthalate P-189 CAS 68515-48-0	SRL PVC Plasticizer Analysis
6	10	66.09	Butyl octyl phthalate P-125 CAS 84-78-6	SRL PVC Plasticizer Analysis
7	58	65.57	Ditridecyl phthalate P-176 CAS 119-06-2	SRL PVC Plasticizer Analysis
8	89	65.32	Di-(2-ethylhexyl) phthalate P-181 CAS 117-81-7	SRL PVC Plasticizer Analysis
9	62	64.75	Di-n-butyl phthalate P-193 CAS 84-74-2	SRL PVC Plasticizer Analysis
10	57	64.63	Diundecyl phthalate P-129 CAS 3648-20-2	SRL PVC Plasticizer Analysis

Figure 19. Omnic spectral database search results

6.5.51 Once the result is obtained verify that the Diisononyl phthalate is identified in the results at a match % of greater than 60%, and all other ortho-phthalates identified also have a match of greater than 60% for CHK SMP1.

6.5.52 Note: If the search results does not find a match for the desired phthalates, go back to the library setup, and delete all the libraries from the “search libraries and groups” and then reselect the “phthalates analysis” group before running the search again.

6.5.53 In the Sample Check Verification Log fill out the measured wavenumber column for Diisononyl phthalate with the match % obtained. If the match % obtained is greater than 60% write pass on the results column. If the match obtained is less than 60% the sample check verification has failed. In case of failure remove the sample, clean the sample surface, and rescan a different surface on the check sample.

6.5.54 Click the print button to save a digital copy of the results obtained. Click the printer setup button on the popup screen and set the printer name to Adobe PDF. Press OK on both the popups. Navigate to the designated FTIR folder mentioned above in the F: drive under the specific study and save with a standard filename such as 10092019 CHKSMP1 library match. Then print a physical copy to be filed.

6.5.55 Remove CHK SMP 1 from the sample holder and clean the crystal surface and the solid/plastic press tip with alcohol and wipes. Wait till the surface is completely dry before proceeding with CHK SMP 2.

6.5.56 Close the result screen by clicking the Close button on the bottom right of the screen.

6.5.57 After closing the results screen, the processing window displays the original spectrum and all other spectra obtained (Baseline corrected). Just close this window to start a new sample.

6.5.58 Repeat the steps of collecting a sample spectrum, correcting the baseline, and finding peaks with the lab sample CHK SMP 2. Save a digital copy of the spectrum with a standard filename such as 10092019 CHKSMP2 Find Peaks and save it to the FTIR folder under the specific study in F: drive.

6.5.59 After a digital copy has been saved, print a copy, and fill out the table in the Sample Check Verification Log for Analysis of Phthalates with measured wavenumbers for CHK SMP 2.

6.5.60 Also, save the .SPA file by clicking on the File tab located on the top left side of the screen and choosing "Save As." Use the filename format (mmddyyyy Sample ID) to save the .SPA file in the same study specific folder. For example (04202021 CHK SMP2)

6.5.61 Repeat the process for finding library matches. Verify that Di (2-ethylhexyl) phthalate is present and all other ortho-phthalates identified are at >60% for CHK SMP 2.

6.5.62 In the Sample Check Verification Log fill out the measured wavenumber column for Di (2-ethylhexyl) phthalate with the match % obtained. If the match % obtained is greater than 60% write pass on the results column. If the match obtained is less than 60% the sample check verification has failed. In case of failure remove the sample, clean the sample surface, and rescan a different surface on the check sample.

6.5.63 Click the print button to save a digital copy of the results obtained. Click the printer setup button on the popup screen and set the printer name to Adobe PDF. Press OK on both the popups. Navigate to the designated FTIR folder mentioned above in the F: drive under the specific study and save with a standard filename such as 10092019 CHKSMP2 library match. Then print a physical copy to be filed.

6.5.64 A completed sample check verification packet should be filed in the Data Entry and Review Checklist folder under the associated study. It must have a filled out sample check verification log, spectra with labeled peaks for both check samples and, spectral library comparison results for the blank and both of the check verification samples.

6.5.65 Once the sample check verification process has been completed for both check samples with satisfactory results (pass in all categories), the analyst can proceed to screening of unknown samples.

6.6 Sample Screening for plastic class identification, plasticizers and additives, and other organic contaminants:

6.6.1 Extensive preparation is not necessary for analyzing a material (sample), however size reduction may be necessary to allow for the pressure tower to be used in pressing the sample to the crystal surface. Follow procedures outlined in Product Testing's Consumer Products Sample Collection and Processing SOP for processing the samples for screening by XRF. The sample processing method for screening is identical for both instruments.

6.6.2 Best results are obtained when the sample is pressed flat against the crystal. Once the sample is pressed flat on the surface the pressure tower needs to be tightened till a click is heard or felt.

6.6.3 Note: Be careful to keep an eye on the plastic tip while tightening. To ensure that the tip does not break, it should rest flat on the surface of the material and stay in a horizontal alignment as much as possible when tightening.

6.6.4 Use extreme caution when screening liquid samples. Do NOT screen liquids that may need acetone or strong acids and bases for cleanup. Any samples suspected of having these types of compounds should not be screened using the FTIR.

6.6.5 For thick viscous samples such as nail polish and paints that can be desiccated, it is best to dry it on a glass surface or XRF sample cup in a fume hood to convert it into a solid chip before analyzing it on the FTIR crystal.

6.6.6 Follow steps in section 6.4 to determine the correct library setup before scanning.

6.6.7 Clean the crystal surface prior to screening each sample to eliminate any cross contamination between samples.

6.6.8 Col Smp icon to begin collecting the spectrum.

6.6.9 A prompt for entering the spectrum name appears. Enter the sample name “Component ID” in front of the time date stamp and Press OK.

6.6.10 A prompt appears “Please prepare to collect the sample spectrum.” Click Ok to proceed.

6.6.11 A preview screen with a flat baseline should appear from 4000 to approximately 650 wavenumbers. If the ATR crystal is not clean the baseline may appear to have peaks. In case of unwanted peaks, clean the crystal and sample platform with 24% ethanol and wait for it to air dry.

6.6.12 Once a flat baseline is achieved, use dedicated tweezers (tweezers or non-phthalate gloves should be cleaned with Kim Wipes wetted with 24% ethanol in between each use) to carefully place the desired sample on the crystal surface and tighten the pressure clamp. Best results are obtained when the sample is pressed flat against the crystal. Once the sample is pressed flat on the surface the pressure tower needs to be tightened till a click is heard or felt.

6.6.13 Note: When using the plastic tip with the triple-bounce crystal be careful to keep an eye on the plastic tip while tightening. To ensure that the tip does not break, it should rest flat on the surface of the material and stay in a horizontal alignment as much as possible when tightening.

6.6.14 Press the “start collection button” in upper right corner to begin scanning.

6.6.15 After scanning is completed choose “Yes Add to Window” option on the popup that appears.

6.6.16 A prompt may appear asking for a window label. Click OK to automatically choose the default window label. (Window 1, window 2….etc)

6.6.17 Correct the baseline of the spectrum by clicking on the Process Tab and choosing the Automatic Baseline Correct option.

6.6.18 A new baseline corrected spectrum highlighted in red is added to the current window.

6.6.19 Hide the old raw spectrum (spectrum without the corrected baseline) from the current window by first clicking on it to highlight it in red, and then pressing CTRL+H.

6.6.20 Run the spectral match by pressing on the Analyze Tab and clicking on the Search button.

6.6.21 Close the window and remove the sample from the instrument. Clean the surface and begin the process with a different sample.

6.6.22 Save a digital copy of the spectrum with identified matches as a PDF by clicking on the print button, and choosing the Adobe PDF option.

6.6.23 Save the PDF file with a standard filename that includes the date, sample ID. (mmddyyyy Sample ID) For example (10102019 DI Water Blank)

6.6.24 The file should be saved in a FTIR folder, in the specific study folder created by the PM in F: drive. If there is no FTIR folder under the specific study create a new one. F:\EAP\TSU\ProductTesting\SpecificStudyName\FTIR

6.6.25 Once the PDF has been saved print a hard copy for records.

6.6.26 Save the .SPA file by clicking on the File tab located on the top left side of the screen and choosing "Save As." Use the same filename format and location mentioned above to save the .SPA file in the same study specific folder

6.6.27 Once all the samples have been screened all PDF files of individuals scans are to be combined into one document and uploaded into the study page in the PTDB. The combined PDF document is saved as "FTIR screening results for Study Name"

6.6.28 Note: PTDB limits the size of the files that can be uploaded at 20Mb. Break down combined PDF documents that exceed the size limit into multiple documents before uploading. PM's may also choose to compile PDFs based on a certain criteria, and upload these as additional files.

6.6.29 Optional: A separate word document with a case narrative from the notes recorded in the FTIR Error Log and any other observations noted by the analyst is also uploaded to the PTDB.

6.6.30 Please refer to Product Testing's SOP for Data Entry and Data Entry Quality Assurance for steps on how to upload documents to the PTDB.

7.0 Records Management

7.1 All records and logs mentioned below are to be stored in the Product Testing Prep Room. (ROL-21)

7.1.1 FTIR Instrument Log (Maintenance Records)

7.1.2 IDC and FTIR Performance Verification Record Log (Record of IDC and Performance Verification (PV) Tests)

7.1.3 FTIR Daily Log (Record of samples scanned)

7.1.4 FTIR Error Log (Record of errors during screening)

7.2 Other records as specified in the sections within this SOP

8.0 Quality Control and Quality Assurance

8.1 Instrument Verification:

8.1.1 A monthly Performance Verification Test based on ASTM E1421 standard practice ensures that the spectrometer is in good working condition and can be used to screen samples.

8.1.2 The Performance Verification Test aligns the spectrometer, verifies the laser frequency, checks the energy throughput, and uses a NIST-traceable polystyrene film located inside the spectrometer to confirm wavenumber accuracy.

8.1.3 The table below identifies the wavenumbers at which the performance verification confirms the wavenumber accuracy for an internal 1.5 mil polystyrene sample.

Wavenumbers (cm^{-1})	High Limit	Low Limit	Measured	Result
Peak at 3060	3061	3059	3059.126	Pass
Peak at 1601.2	1602.2	1600.2	1600.802	Pass
Peak at 1028.3	1029.3	1027.3	1028.244	Pass

Table 2. Wavenumber accuracy for internal polystyrene standard

8.1.4 An example copy of the whole performance verification page that is printed and filed in the FTIR performance verification folder in the prep room at the beginning of each month is available at Product testing SharePoint site.

8.1.5 In case of Performance Verification Test failure, refer to manufacturer recommendations available in the "Nicolet iS5 Spectrometer Fast Facts Sheet" available in the Department of Ecology, Sample Prep Room (OL-21).

8.2 Sample Check Verification for FTIR analysis:

8.2.1 Sample check verification is to be conducted at least once every day before using the instrument for screening.

8.2.2 Sample check verification first verifies the wavenumber accuracy by checking for the presence of specific peaks associated with ortho-phthalates. The presence of the peaks at ± 5 wavenumbers is acceptable and is labeled as a passing result.

8.2.3 Then, the spectrum of the check verification samples are matched to the spectral library for positive identification of phthalates. A positive match of greater than 60% for all identified phthalates is considered to be within the desired standard.

8.2.4 Note: Make sure that the spectral library that is chosen for matching the spectra is consistent in every search.

8.2.5 *Disclaimer: Previously analyzed laboratory samples (CHK SMP1 and CHK SMP 2) used in the process for verifying the instrument's ability to identify the class of ortho-phthalates were analyzed using methods that have been updated by the WA State Accreditation Board. These samples are used to roughly demonstrate the instrument's ability to screen for phthalates, and are not to be considered as standards.

8.2.6 The method listed in this SOP can be used to screen for the presence of phthalates only when the presence is greater than 10%. Therefore, the results obtained cannot be the only determining factor in selecting the product for further analysis.

8.3 Data Collection:

8.3.1 Collection of a background signal is essential for the instrument to generate a spectrum for a sample screened. Make sure that the crystal surface is clean and dry before collecting a background. The pressure clamp needs to be loosened to ensure that the plastic tip is not touching the crystal surface when the background is being collected.

8.3.2 Any errors or abnormal incidents such as errors in sample ID, incomplete scans or bad spectrum due to improper tightening of the pressure clamps etc. should be recorded in the FTIR Error Log book.

8.3.3 Make sure to clean the surface and the plastic tip in between samples. Allow time for the surfaces to dry off before placing another sample.

8.3.4 Use forceps or tweezers to replace samples in and out the crystal surface to reduce the need for changing gloves after each sample. Make sure to clean the forceps with Kim wipes and alcohol in between samples.

8.4 Data Upload:

8.4.1 Once all the samples have been screened all PDF files of individuals scans are to be combined into one document using the Acrobat Reader and uploaded into the study page in the PTDB. The combined PDF document is saved as "FTIR screening results for Study Name"

8.4.2 Please refer to Product Testing's SOP for Data Entry and Data Entry Quality Assurance for steps on how to upload documents to the study page of PTDB.

8.4.3 Optional A separate word document with a case narrative from the notes recorded in the FTIR Error Log and any other observations noted by the analyst is also uploaded to the study page of the PTDB.

8.4.4 The project manager (PM) will inspect datasets uploaded to the Product Testing Database (PTDB) to verify accuracy, completeness, and adherence to the specific study criteria.

9.0 Safety

9.1 Where QAPP specific preparation procedures require the use of the hazardous materials the Ecology Chemical Hygiene Plan and Hazardous Material Handling Plan (Ecology, 2018) which includes the laboratory Safety Orientation, Job-Specific Orientation and Chemical Safety Procedures must be reviewed. The standard Operating Procedures in Section 16 of the Chemical Hygiene Plan and Hazardous Material Handling Plan for handling chemicals must be followed.

9.2 Protocols for sample preparation safety is discussed in Product Testing Sample Collection and Processing Standard Operating Procedure, EAP PTP001.

9.3 Laser Safety:

9.3.1 The instrument uses an 850 nm diode laser that emits radiation invisible to the naked eye. A protective housing covers the instrument and >80% of the laser light is lost as it passes through the instrument optics. Exposure to reflective laser radiation is less than 200 µW during normal use and maintenance. Never stare into the laser beam or at its bright reflection.

9.4 Instrument Safety:

9.4.1 The FTIR is generally left on, even when not in use. The FTIR should not be covered or pushed up against the wall; internally generated heat is vented at the back of the instrument and care should be taken to not obstruct this vent.

9.4.2 Long-term stability of the instrument improves with the length of the time the instrument is on, so turning it off is not necessary.

9.4.3 The instrument is sealed and desiccated to help prevent damage to the optics or other internal components. Check the desiccant indicator located at the back of the instrument before every run to check for the need to change desiccant.

9.4.4 Do NOT use acetone or strong acids and bases for cleaning the crystal surface. Any samples suspected of having these compounds should not be screened using the FTIR.

10.0 References

10.1 ASTM, 2015. ASTM E1421-99 2015(e1) Standard Practice for Describing and Measuring Performance for Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests. ASTM International, West Conshohocken, PA: https://www.astm.org/e1421-99r21.html

10.2 Ecology, 2018. Chemical Hygiene Plan and Hazardous Management Plan. Olympia, WA.

10.3 Ecology, 2015. 15-04-021 HWTR Product Sampling Procedure. Olympia, WA

10.4 Thermo, 2013. BR50555_E 05/13M Introduction to Fourier Transform Infrared Spectroscopy: http://assets.thermofisher.com/TFS-Assets/MSD/brochures/introduction-fourier-transform-infrared-spectroscopy-br50555.pdf

10.5 Thermo Scientific. FTIR Glossary: https://tools.thermofisher.com/content/sfs/brochures/FTIR-Glossary.pdf

10.6 Thermo, 2009. Thermo Fisher 269-249300 Revision A. OMNIC Specta User Guide (User's Guide)

10.7 Wiseman, C. 2018. Standard Operating Procedure for Consumer Product Sample Collection and Processing, Version 2.0 Document No. PTP001. Publication 18-04-018. Washington State Department of Ecology, Olympia, WA.

10.8 Wiseman, C. 2018. Product Testing Database Standard Operating Procedure for Data Entry Quality Assurance, Version 2.0 Document No. PTP002. Publication 19-04-015. Washington State Department of Ecology, Olympia, WA.

Appendix A

Familiarization with the Nicolet iS5 FTIR Spectrometer

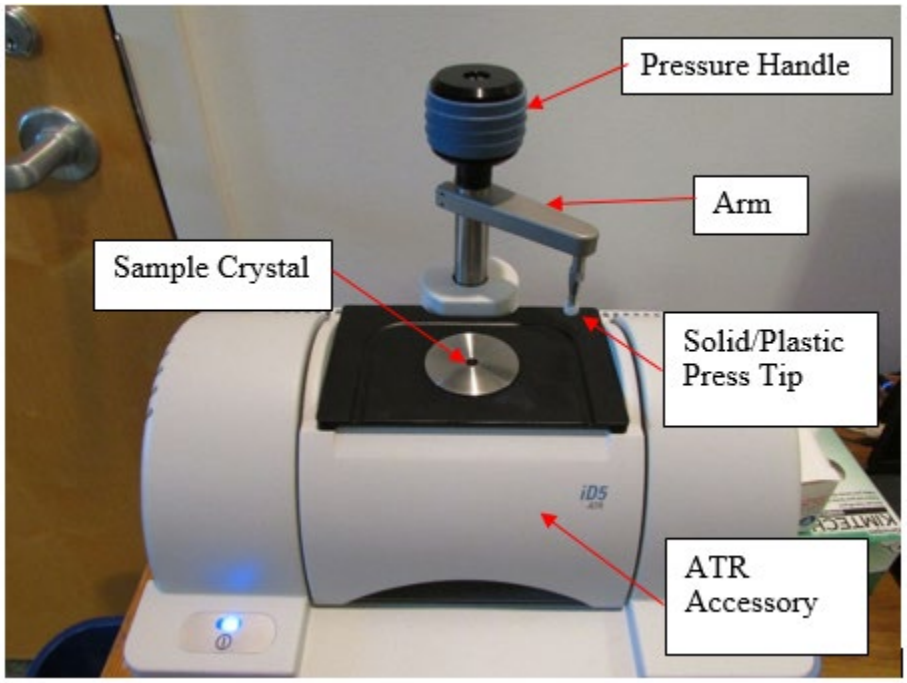


Figure 20. Nicolet iS5 spectrometer

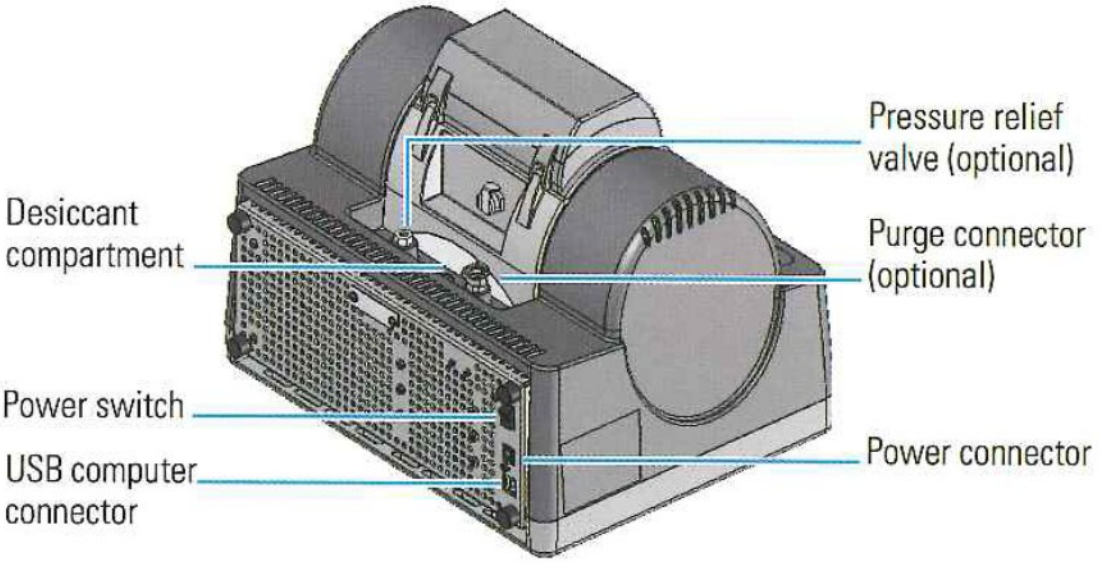


Figure 21. Spectrometer features

Appendix B

Identifying System Status and running a Performance Verification Test

At the adjoining computer, log on with your User Name and password, then open the "OMNIC" icon on the Desktop. The software works from any user ID. Collected data can be stored on the computer in any user-defined study-specific F: drive folder. For product testing studies a specific FTIR folder is created under each study in F: drive.

After opening the OMNIC software, check the System Status by reviewing the System Performance Verification shield icon in the upper right corner of the Omnic software interface.

 A green icon indicates that the status is good and no action is needed.

 A yellow icon indicates there is a problem detected but it is not serious and corrective action may be required. Any minor issues should be checked to determine if it has any effect on the quality of the data.

 A red icon indicates that the system is out of specification or a subsystem failed. Most often it indicates that the last PV test did not meet specifications, or a monthly PV test has not been performed.

Follow steps below for running the Performance Verification test:

1. Click on the red system status icon which opens the following dialog box.

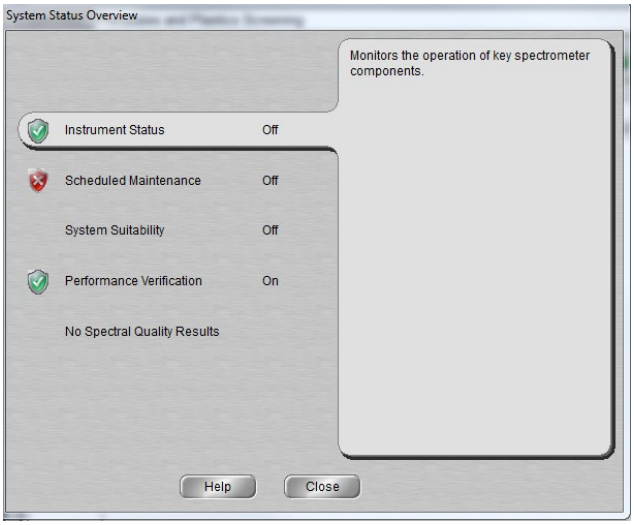


Figure 22. System Status Overview

2. Click the Performance Verification button. This displays the last date when a PV test was run and the date after which it expired.

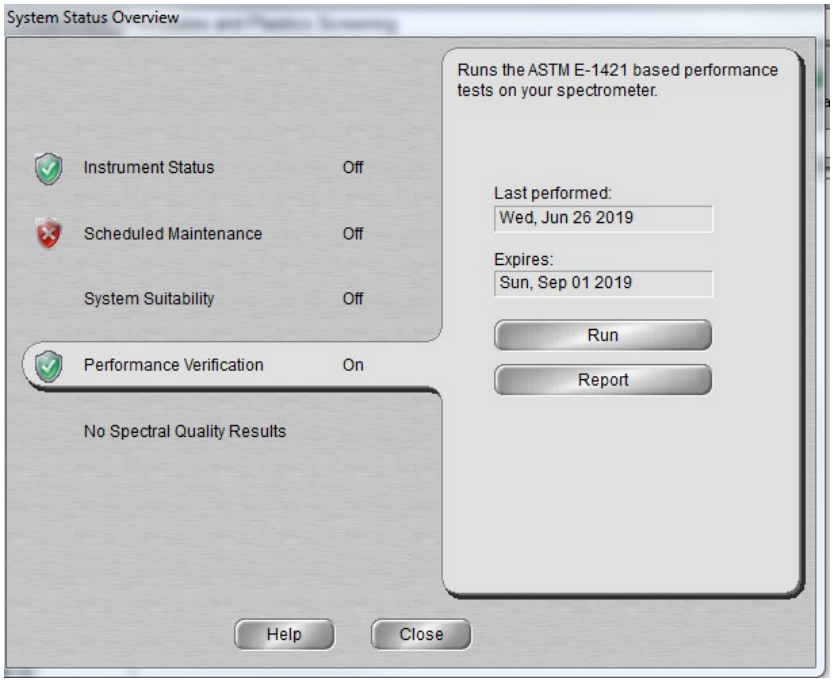


Figure 23. Performance Verification

3. Click Run. On clicking the run button Omnic prompts (Laser Verification) you to remove the accessory in the sample compartment. Carefully remove the iD5 ATR accessory and place it on clean Kim Wipes. To remove the accessory, carefully grasp it by the handles and lift straight up. (as shown below) Click OK on the new prompt that appears on removing the iD5 ATR accessory. Next, Click OK on the Laser Verification prompt.
4. Note: Make sure there is always an accessory installed on the instrument. When not using an accessory, store it in a dust-free environment, such as in a box inside the FTIR cabinet in the Product Testing Prep Room.


Figure 24. Removing an accessory

5. The procedure aligns the spectrometer, verifies the laser frequency, checks the energy throughput, and uses a NIST-traceable polystyrene film located inside the spectrometer to confirm wavenumber accuracy. The PV test is based on ASTM E1421 standard practice and takes about 6 minutes to complete.
6. The performance verification process goes through a series of windows and messages detailing each step. You may hear the motor rotate the internal polystyrene sample in and out of position while the system measures the polystyrene spectrum.

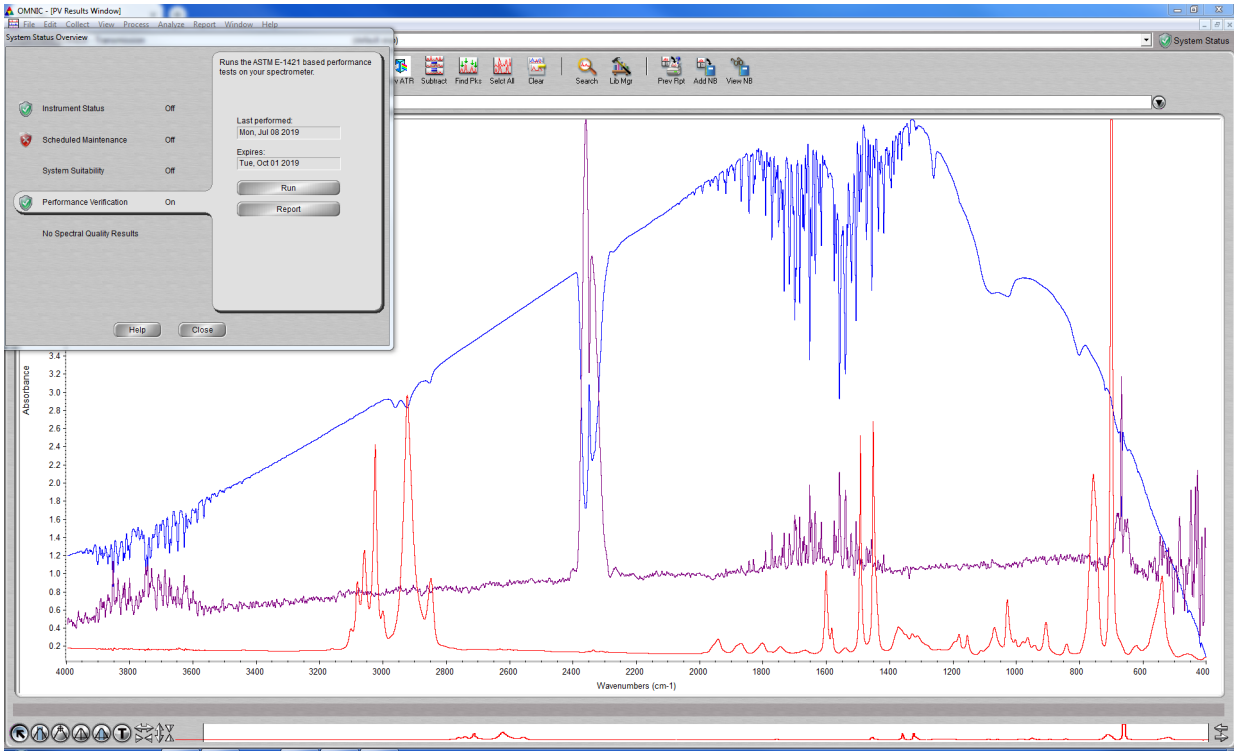


Figure 25. Performance verification complete screen

7. After the performance verification has been completed print the report by clicking on Report button then select print.
8. The FTIR analyst should sign and date the **performed by** portion of the Performance Verification Test Log and add it to the FTIR Performance Verification Log Book.
9. Once the report is printed the System Status icon should turn **Green**. Close all windows and insert the iD5 ATR accessory to begin using the equipment for running sample verification and screening samples.
10. Once the iD5 accessory has been installed a prompt appears on the screen which specifies the accessory installed and the experiment loaded. Click Ok to run a performance test that verifies that the accessory has been inserted correctly.
11. A prompt comfirms the accessory passed the performance test.
12. Return to Procedure.

Appendix C

Standard peaks for identifying ortho-phthalates

1. Given below is a spectrum of dioctyl phthalate (DOP), also known as di (2-ethyl hexyl) phthalate:

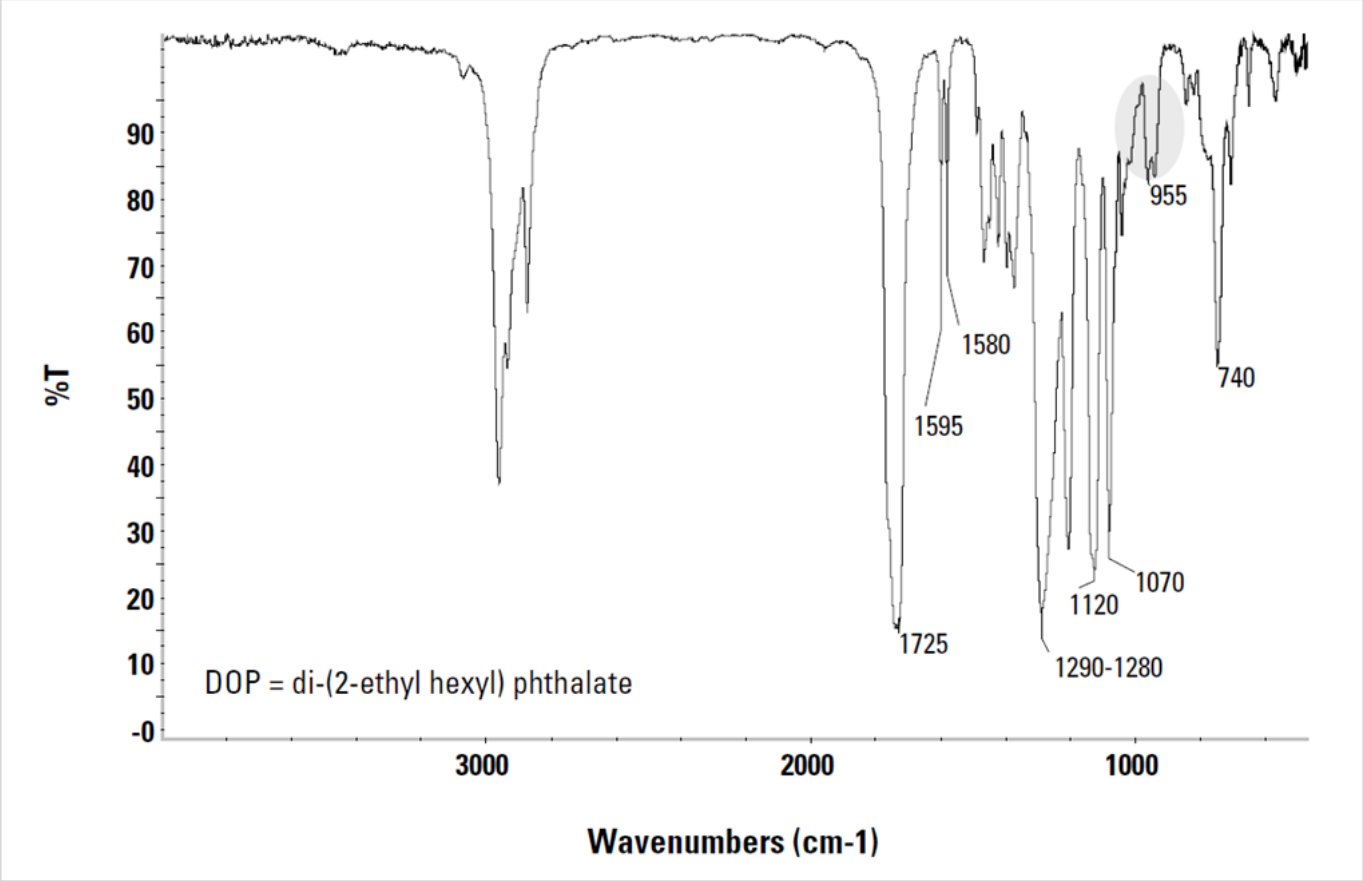


Figure 26. IR spectrum of DOP with characteristic ortho-phthalate peaks

2. It is characterized by the following bands that are characteristic of all ortho-phthalates.

Vibration modes	Wavenumber (cm^{-1})	Comment
$V_{C=O}$	1725	Very intense
V^1_{ring} V^2_{ring}	1595 1580	Weak doublet band, sharp and of nearly equal intensity
V^a_{C-O-C}	1280-1290	Very intense Split
V^s_{C-O-C}	1120	Intense
	1070	Intense, Sharp
$\omega_{4H\ adjacent}$	740	Medium, Sharp

Table 3. Information on the wavenumbers associated with characteristic ortho-phthalate

Laboratory measured verification sample information

3. The table below provides information on the samples with known amounts of ortho-phthalates, being used to verify the ability of the instrument to detect the presence of ortho-phthalates. These samples also set the current limit of detection (LOD) of the instrument to 13%.

Sample Component ID	Extraction method used	Analysis Method Used	Most Significant Phthalate Identified	Analysis Value (mg/Kg)	Analysis Reporting Limit (mg/Kg)	Analysis Lab
MK-5-25-3 CHK SMP 1	CPSC-CH-C1001-09.3	SW8270 C	Diisononyl phthalate	130,000	10,000	BSK Associates Analytical Laboratory
SL-1-29-1 CHK SMP 2	SW3546	SW8270 D	Di (2 ethyl-hexyl) phthalate	160,000	2500	Dept. of Ecology Manchester Environmental Lab

Table 4. Check verification samples

Appendix D

When do you need a new background?
The CO_2 and humidity levels in the air near the spectrometer will change over time. This will cause absorption features, negative or positive, to show up in your sample spectrum because the sample spectrum is being ratioed to a background that had slightly different levels. If you start seeing CO_2 peaks around half the height of the main peaks in your samples, you should take a new background. Water vapor is typically more disruptive. So, if you see water vapor peaks that are taller than perhaps 5% of the tallest peak in your spectrum, you should get a new background.
See Figure 27, where CO_2 is much too strong and water vapor is making a mess of the important 1400-1800 region. You can collect a new background anytime--you don't have to wait for the reminder.

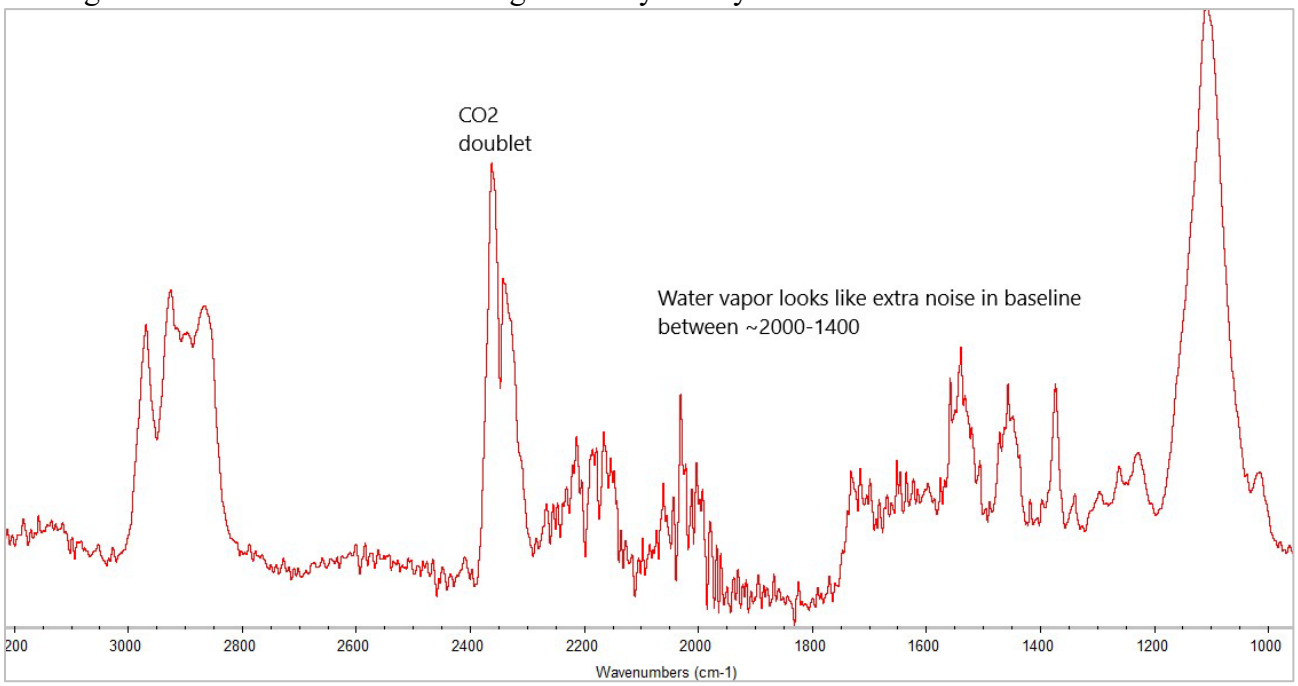


Figure 27. Example of a polyurethane sample that needs to be redone because it needs a new background.

Figure 28 shows the same sample with a proper background.

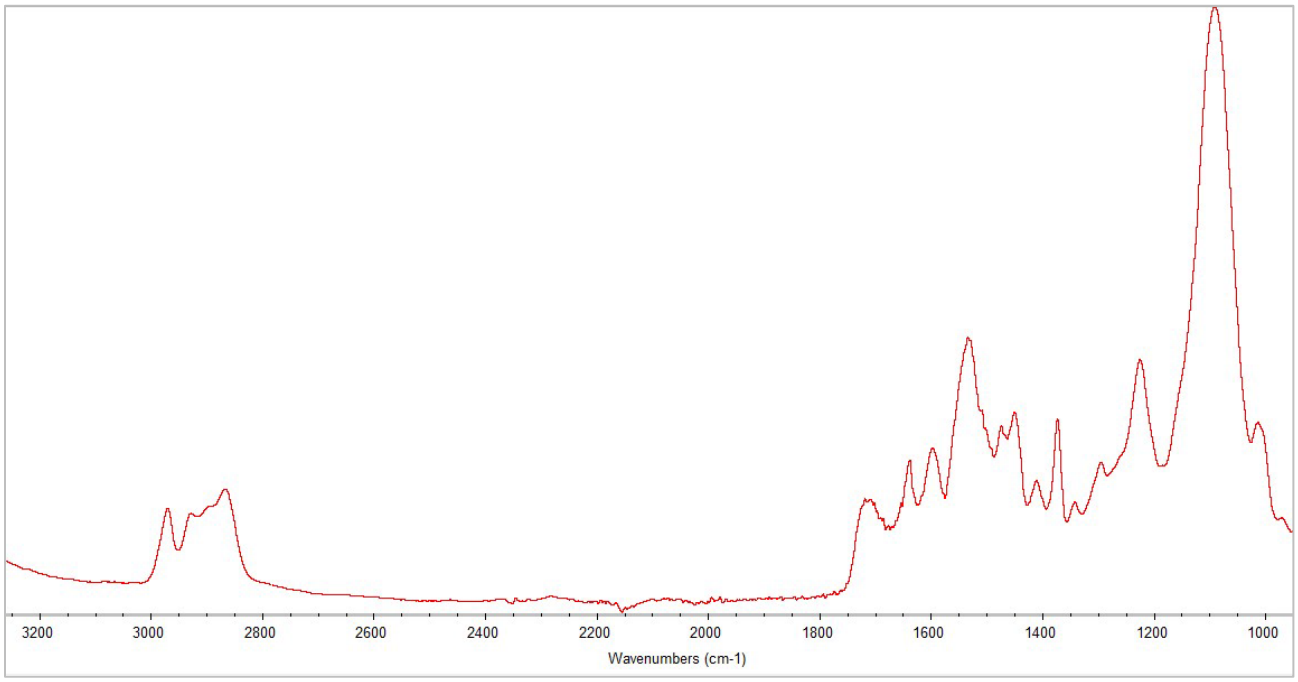


Figure 28. Polyurethane sample with appropriate background. Note the smooth baseline.