

FTIR Spectrometer Performance Monitoring and Maintenance

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FTIR Spectrometer Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Thermo Fourier Transform Infrared (FTIR) bench spectrometer and various accessories. The Thermo FTIR is a bench spectrometer that can be used in transmission mode or in conjunction with an accessory. The accessories include a Continuum microscope and Attenuated Total Reflectance (ATR) sampling devices. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/subdisciplines: Explosives Chemistry, General Chemistry, Seized Drugs, and Paints and Polymers.

3 EQUIPMENT

- Instrumentation
 - Thermo 6700, iS5, or iS50 FTIR and Omnic Software (or equivalent)
 - Thermo Continuum Microscope accessory (or equivalent)
 - Golden Gate, Smart Orbit or integrated ATR accessories (or equivalent)
- Materials
 - Polystyrene Standards - 1.5 mil and 3.0 matte-finish films mounted on a card, or mounted in a metal frame, or as internal standards within the bench (Thermo or equivalent)
 - Slide containing a metal disk with a 100 micron pinhole, an open hole approximately 11 mm in diameter, and a 14 mm diameter gold mirror (for microscopes) (Thermo or equivalent)
 - Compressed nitrogen to purge instrument and microscope (if appropriate)
 - Liquid nitrogen (for cooling microscope detectors)
 - Dewar Flask (Thermo or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

- Performance Standards

The polystyrene films are used to assess operating performance, wavenumber assignment, and continued integrity of the system. The polystyrene standards used for this procedure require no preparation and do not expire. It is recommended by Thermo that they are replaced if showing signs of wear or if results have drifted.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily. Enter information in the appropriate log for tracking purposes.

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- A. Choose the appropriate bench and/or accessory. If using an ATR or transmission accessory, insert it into the bench (if applicable). If using a microscope accessory, cool the detector with liquid nitrogen using a Dewar flask.
- B. Load a method that is appropriate for the bench/accessory and mode of detection (ATR or %T) being used. Verify all parameters using the 'Instrumental Conditions' section.
- C. Collect an ambient background spectrum and sample spectrum of the polystyrene standard.
- D. Use 'Find Peaks' to label the major peak apexes. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the spectrum.
- E. Save the file to the appropriate polystyrene standards folder.
- F. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, align the bench/accessory and re-analyze the polystyrene. If the results are still poor or failing, contact appropriate instrument support personnel.

5.2 As Needed Checks

The following procedure will be performed as needed based on performance. Enter the information in the appropriate log to indicate completion.

5.2.1 Interferogram Signal Evaluation

- A. Select the method (mode of detection) 'Bench %T' or equivalent.
- B. Clear the sample compartment of any material which would impede the IR beam.
- C. Monitor the interferogram signal under a gain setting of one (1.0).
- D. Record the peak-to-peak voltage of the interferogram (which is the sum of the absolute minimum and maximum peak values) in the appropriate log. This value reflects the amount of signal (in terms of voltage) being detected.
- E. If the signal value has dropped significantly, the optical velocity of the beamsplitter can be automatically adjusted to improve the beam voltage throughput by the appropriate instrument support personnel.

5.2.2 Bench Evaluation

- A. Initialize the appropriate system validation/qualification program (ex. Val-Q/ValPro) from within Omnic.
- B. Open the appropriate bench.csv file (if applicable).
- C. Start the validation. The Nexus 6700 and iS50 will internally supply the polystyrene standards.
- D. Evaluate the validation report. It will specify if all tests pass or if any fail. If any fail, align the bench and repeat. When the results are acceptable, print the report. Printing the spectral data is optional.
- E. Ensure that the results are saved to the data system.
- F. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, the appropriate instrument support personnel will determine the corrective maintenance to be performed.

6 INSTRUMENTAL CONDITIONS

6.1 Bench

Mode:	Transmission or Reflectance
Number of Scans:	32
Resolution:	4
Scan Range:	400 (min) - 4000 cm^{-1} (max)

6.2 ATR Accessory

Mode:	Reflectance
Number of Scans:	32
Resolution:	4
Scan Range:	400 (min) - 4000 cm^{-1} (max)

6.3 Microscope Accessory

Mode:	Transmission
Number of Scans:	128
Resolution:	4
Scan Range (MCT/A):	650 - 4000 cm^{-1}
Scan Range (MCT/B):	475 - 4000 cm^{-1}

7 ACCEPTANCE CRITERIA

7.1 Polystyrene

The Polystyrene spectrum is acceptable if the following four peaks are within a $\pm 4 \text{ cm}^{-1}$ window of the expected wavenumber. If values lie outside the specified range, align the bench/accessory and re-analyze the polystyrene. If the results are still poor or failing, contact appropriate instrument support personnel.

Expected (cm^{-1}):	Acceptable Range (cm^{-1}):
3025	3021 to 3029
1601	1597 to 1605
1028	1024 to 1032
906	902 to 910

7.2 Validation Report

The Validation Report generated will indicate whether the obtained values lie within the ranges specified by the manufacturer and provide a 'pass' or 'fail' result. All tests should pass. If any fail, contact appropriate instrument support personnel.

8 LIMITATIONS

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

9 SAFETY

FTIR instruments contain a laser source which can be hazardous and must be used following best practices for eye safety and other potential hazards. Use caution and avoid direct eye contact with the laser beam or its reflection.

Liquid nitrogen is extremely cold and therefore potentially hazardous. When filling the detector Dewar, avoid liquid nitrogen skin contact. Wear protective gloves and follow standard laboratory safety practices.

10 REVISION HISTORY

Revision	Issued	Changes
04	07/15/2022	Revised to match new format requirements. Section 6- Added 'Bench' conditions. No other substantive changes to content.
05	11/15/2022	Section 2- Updated Scope to remove location. No other substantive changes to content.