

## **Performance Monitoring Protocol (QA/QC) for the Thermo Nicolet FTIRs**

### **1 Scope**

This document addresses the performance monitoring (QA/QC) of the Thermo Nicolet Fourier Transform Infrared (FTIR) bench spectrometer and various accessories. This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Drug chemistry, toxicology, paint, explosives (chemistry), fire debris, and Chemistry Unit general physical and chemical analysis.

### **2 Principle**

The Thermo Nicolet FTIR is a bench spectrometer that can be used in transmission mode or in conjunction with an accessory. The accessories include a Continuum microscope, a DuraSamplIR ATR (Attenuated Total Reflectance), a Golden Gate ATR, a Smart Orbit ATR, and an integrated iS50 ATR. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

### **3 Equipment/Materials/Reagents**

- a. Thermo Nicolet 6700 Nexus or iS50 FTIR, and Omnic Software (or equivalent)
- b. Microscope Accessories: Thermo Nicolet Continuum (or equivalent)
- c. ATR Accessories: SensIR DuraSamplIR, Golden Gate, Smart Orbit, integrated iS50 (or equivalent)
- d. Compressed nitrogen to purge instrument and microscope, if appropriate
- e. Liquid nitrogen (for microscopes)
- f. Dewar Flask (for microscopes) (supplied with the instrument by Thermo Nicolet)
- g. 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 Nicolet or equivalent)
- h. 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 Nicolet or equivalent)

## 4 Standards and Controls

### 4.1 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 Nicolet that they are replaced if showing signs of wear or if results have drifted.

## 5 Sampling or Sample Selection

Not applicable.

## 6 Procedures

### 6.1 Daily Checks

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

- a. Choose the appropriate bench and/or accessory. If using an ATR accessory, insert it into the bench (if applicable). If using a microscope accessory, cool the detector with liquid nitrogen.
- 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 of this protocol.
- 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 'Decision Criteria' section of this protocol. If the results are acceptable, print the spectrum.
- e. Save the spectrum to the QA/QC polystyrene standards folder.
- f. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." 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.

## 6.2 As Needed Checks

The following steps are to be performed as needed based on system performance, and can be performed more frequently if desired. If a problem is indicated by the failure of the 'Daily Checks', these steps can help to identify the cause of an instrument error. Indicate completion in the appropriate QA/QC log.

### 6.2.1 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) on the QA/QC Log. This value reflects the amount of signal (in terms of voltage) being detected.
- e. If the signal value has dropped significantly, the beamsplitter can be automatically adjusted to improve the beam voltage throughput. Refer to the manufacturer's instrument manuals for further instructions or contact appropriate instrument support personnel.

### 6.2.2 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. Save the spreadsheet data to the hard drive
- f. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, the IOSS Manager or appropriate instrument support personnel will determine the corrective maintenance to be performed.

## 7 Instrumental Conditions

### 7.1 ATR Accessory

Mode: Reflectance  
Number of scans: 32  
Resolution: 4  
Scan range: minimum 400 – maximum 4000  $\text{cm}^{-1}$

### 7.2 Microscope Accessory

Mode: Transmission  
Number of scans: 128  
Resolution: 4  
Scan range: 650-4000  $\text{cm}^{-1}$  for MCT/A  
475-4000  $\text{cm}^{-1}$  for MCT/B  
Objective and stage compensators: 0

## 8 Decision Criteria

### 8.1 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. The following values have been provided by Nicolet:

<u>Expected</u>	<u>Acceptable Range</u>
3025 $\text{cm}^{-1}$	3021 to 3029 $\text{cm}^{-1}$
1601 $\text{cm}^{-1}$	1597 to 1605 $\text{cm}^{-1}$
1028 $\text{cm}^{-1}$	1024 to 1032 $\text{cm}^{-1}$
906 $\text{cm}^{-1}$	902 to 910 $\text{cm}^{-1}$

### 8.2 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.

## 9 Calculations

Not applicable.

## 10 Measurement Uncertainty

Not applicable.

## 11 Limitations

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

## 12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis.

## 13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual*.

*FBI Laboratory Safety Manual*.

Rev. #	Issue Date	History
2	04/25/16	Updated instrumentation in sections 2 and 3 to remove model 560 and 670, and add the iS50 and integrated iS50 ATR. Changed Scan range in section 7.1 to allow for wider variable range.
3	10/04/18	Updated Section 1 Scope to include applicable disciplines/categories of testing. Updated heading in Section 5. Removed labeling requirement in Section 6.1. Added 'appropriate instrument support personnel' to Sections 6.2.1 e, 6.2.2 f, and 8.1. Updated 'Instrument Operation and Systems Support' in Section 13 and header.

**Approval**

Redacted - Signatures on File

Drug Chemistry/  
 General Chemistry  
 Technical Leader:

Date: 09/28/2018

Toxicology  
 Technical Leader:

Date: 09/28/2018

Paints and Polymers  
 Technical Leader:

Date: 09/28/2018

Fire Debris Technical  
 Leader:

09/28/2018

Explosives (Chemistry)  
 Technical Leader:

Date: 09/28/2018

IOSS Manager:

Date: 09/28/2018

Chemistry Unit Chief:

Date: 09/28/2018

**QA Approval**

Quality Manager:

ate: 09/28/2018