Performance Monitoring Protocol for FT-IR Systems

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1 INTRODUCTION

This document addresses the quality assurance/quality control (QA/QC) performance monitoring of the Fourier Transform Infrared (FT-IR) spectrometer systems. Identification of generic polymeric group and classification of polymeric sub-group is conducted using the FT-IR system(s) identified below or an equivalent or better system. The performance of the system(s) must be monitored to verify that the instrument is producing reliable and reproducible results.

2 SCOPE

This document applies to the Fourier Transform Infrared (FT-IR) spectrometer systems utilized by personnel in the Trace Evidence Unit (TEU) and Scientific and Biometrics Analysis Unit - Trace (SBAU-Trace).

3 EQUIPMENT

- Fourier Transform Infrared (FT- IR) Spectrometer with Microscope Accessory
- Omnic Series Software (e.g., Omnic, Picta)
- Liquid nitrogen
- Dewar flask
- Polystyrene standard (1.5mil)
- Pinhole slide

4 STANDARDS AND CONTROLS

4.1 Performance Standard

A. A 1.5mil polystyrene standard is analyzed as the performance standard to assess operating performance, wavenumber assignment, and continued integrity of the system.
    1. The polystyrene standard used for this procedure requires no preparation.
    2. It is recommended by Thermo Scientific that the standard be replaced if showing signs of wear or if analysis of the standard does not produce the expected results.
    3. Newly acquired polystyrene standards that are accompanied by a statement of traceability to a NIST sample require no further testing before use.
    4. Non-serialized polystyrene standards that are not accompanied by a statement of traceability to a NIST sample require a comparative analysis with a traceable polystyrene standard.
    5. This can be accomplished by comparison of spectral data to a spectrum obtained from a traceable, serialized polystyrene standard.
    6. The newly acquired polystyrene standard must also meet the Decision Criteria outlined in Section 5.1.F.

7. If the newly acquired standard meets the aforementioned requirements, it can be used as a daily performance standard. If it does not meet the requirements, it will not be used as a daily performance standard.
8. Spectral data produced during the acceptance process will be maintained on the instrument computer

5 PROCEDURE

5.1 Daily Performance Check

The following steps are to be performed each day the instrument is to be used before beginning casework analysis. The appropriate information will be recorded in the instrument log.

A. Cool the detector by filling the internal Dewar with liquid nitrogen.
B. Load the appropriate microscope transmission method, and verify the following parameters:
    - Mode = Transmission
    - Number of scans = 128
    - Resolution = 4
    - Scan range = 650-4000cm^{-1} (wavenumbers)
C. Collect a sample spectrum of the polystyrene standard followed by a background spectrum of air (empty stage).
D. Use “Find Peaks” to label the major peaks.
E. Evaluate the results.
F. The results of the polystyrene spectral analysis are acceptable if the following four peaks are within +/- 4 cm^{-1} of the expected values (ASTM 1421-99 (2015)).

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

G. Save the spectrum of the polystyrene standard in the appropriate electronic folder.
H. If the results are not acceptable, the polystyrene standard will be re-analyzed.
    1. If the results are still not acceptable, an out-of-service sign will be placed on the instrument, the appropriate log entry made, and the Technical Leader (TL) or SBAU - Instrumentation Operations group, contacted. The TL or SBAU - Instrumentation Operations group, will be responsible for ensuring the instrument is brought back into service. Any adjustments made will be recorded in the appropriate log.

5.2 FT-IR Microscope Accessory Troubleshooting

The following evaluations may be performed as needed to help troubleshoot poor instrument performance or malfunction. Evaluation results will be recorded in the affected instrument's log when appropriate.

5.2.1 Evaluation of the Bench Interferogram Signal

A. Load the "Transmission ESP" method.
B. On the "Bench" tab of "Experiment Setup", monitor the interferogram signal under a gain of one (1.0).
C. Record the peak-to-peak voltage of the interferogram in the instrument log. This value reflects the voltage being detected.
D. If the signal value has dropped significantly from the previous evaluation, the beamsplitter can be automatically adjusted to improve the beam voltage throughput. Refer to the manufacturer's instrument manuals for further instructions.

5.2.2 Evaluation of the FT-IR Microscope Accessory Interferogram Signal

A. Cool the detector with liquid nitrogen.
B. Load the microscope transmission method.
C. Set the objective and stage compensators to zero.
D. Align and focus the microscope using the 100 micron pinhole slide.
E. Monitor the interferogram signal under a gain setting of 1.0 on the Continuμm.
F. Record the peak-to-peak voltage of the interferogram in the instrument log.

6 LIMITATIONS

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

7 SAFETY

Standard precautions for the handling of liquid nitrogen to include lab coat, goggles, and cryogenic gloves will be taken. Personal protective equipment (at a minimum, a laboratory coat and gloves) will be used when handling any chemical. Refer to the FBI Laboratory Safety Manual for the proper handling and disposal of all chemicals. No specific hazards are associated with the microscopy techniques performed. Universal precautions will be followed.

8 REFERENCES

ASTM 1421-99 (2015), Standard Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests, American Society for Testing and Materials, West Conshohocken, PA

FBI Laboratory Safety Manual, (current version)

9 REVISION HISTORY

Revision	Issue Date	Changes
05	2/3/2020	Updated SBAU-Trace group name throughout. Updated in Section 5.1 e. Rearranged Decision Criteria to 5.1 g. Changed all reference to TEU Property Manager to TL.
06	1/28/2022	Reformatted entire document and rearranged for clarity. Removed Bench Evaluation and reference to additional Performance Standards.