

**Title: TR SOP-03: FTIR and Microspectrophotometry Instrument Verification****A. PURPOSE:**

For use when verifying that the FTIR and the microspectrophotometer are acceptable for use (e.g., for evidence in casework).

**B. RESPONSIBILITY:**

Analysts within the Chemistry Unit

**C. DEFINITIONS:**

FTIR – Fourier Transform Infrared Spectrophotometer

ATR – Attenuated Total Reflectance

NIST – National Institute of Standards and Technologies

PS – Polystyrene

UV-VIS-NIR – Ultraviolet-Visible-Near Infrared

QA/QC – Quality Assurance/Quality Control

**D. PROCEDURE:****FTIR**

An FTIR instrument will be validated/verified before it is used for casework. While the validation process may be considered calibration, no parameter changes will be made which will affect the accuracy of data. Validation is only necessary when the instrument is initially placed in service in order to show that the instrument is capable of successfully producing valid data and that such data can be used within casework. Verification is a process wherein the instrument is shown to be ready for analysis and is operating normally.

Use a NIST traceable polystyrene (PS) reference standard, when available. While some PS reference standards may have expiration dates, usage of these standards when they are beyond their expiration dates is allowable for validation/verification as long as comparative data from pre-expiration analyses has not significantly changed.

Any relevant documentation should be stored with the instrument or in a place readily accessible by analysts. Verification that the FTIR is working properly will be conducted prior to use of the instrument and should occur on the same day. Additional verifications can occur, if necessary.

A check mark in a designated box located on the Physical Evidence worksheet which will indicate that the verification check has been completed. A log book should be used to list all QA/QC data and instrument issues (e.g., repairs, services).

## 1. Analysis

The FTIR can be used in conjunction with a horizontal attenuated total reflectance (HATR) (aka. ATR) accessory or can be used with a microscope accessory. The ATR accessory allows for samples to be analyzed directly without sample preparation. The microscope accessory allows very small particles to be analyzed (e.g., paint).

The QA/QC will be completed on a monthly basis and will be conducted prior to the use of the instrument for casework. It may additionally be performed at the Examiner's discretion (e.g. after a power outage).

- a. An internal polystyrene (PS) reference is analyzed and all printouts should be placed into the instrument logbook.
- b. The energy of the light source may be monitored to verify that there has not been a significant energy decrease. Any energy decrease may indicate the need to replace the source. If the source is left unchanged it could result in loss of sample sensitivity.
- c. If the QA/QC check has problems or the instrument operability check fails, document the failure in the instrument log book and notify the appropriate Lead Examiner or Deputy Director.

## 2. Evaluation

The FTIR is a stable instrument and requires very little maintenance. The maintenance performed includes daily (as used) and 'as needed' items.

- a. Compare the polystyrene spectra to previously acquired spectra in order to confirm that there were no shifts or changes. Major differences indicate problems with the instrument or the accessory.
- b. If needed, compare spectrum to library spectra. Consult supervisor, or designee, if shifts in spectra, changes in spectra, or problems are observed.
- c. Print necessary spectra and place in the instrument's maintenance logbook.
- d. Change desiccant as needed. This is performed by opening the cover of the instrument removing the cage which contains the desiccant and placing fresh desiccant bags in the cage. The cage is then returned to the instrument and the software can be updated to show the change. If applicable, this can be done as part of the service contract by a service engineer.

## Microspectrophotometer

A microspectrophotometer will be validated/verified before it is used for casework. While the validation process may be considered calibration, no parameter changes will be made which will

affect the accuracy of data. Validation is only necessary when the instrument is initially placed in service in order to show that the instrument is capable of successfully producing valid data and that such data can be used within casework. Verification is a process wherein the instrument is shown to be ready for analysis and is operating normally.

Use a NIST traceable reference standard, when available. While some reference standards may have expiration dates, usage of these standards when they are beyond their expiration dates is allowable for validation/verification as long as comparative data from pre-expiration analyses has not significantly changed.

Any relevant documentation should be stored with the instrument or in a place readily accessible by analysts. Verification that the instrument is working properly will be conducted prior to use of the instrument and should occur on the same day. Additional verifications can occur, if necessary.

A check mark in a designated box located on the Physical Evidence worksheet which will indicate that the verification check has been completed. A log book should be used to list all quality assurance/quality control (QA/QC) data and instrument issues (e.g., repairs, services).

## 1. Analysis

The UV-visible-NIR microspectrophotometer (MSP) can be configured to measure the transmittance, absorbance, reflectance, polarization, fluorescence, and luminescence microspectra of materials using areas smaller than a micron. A MSP is capable of non-destructive and non-contact colorimetry and thin film thickness measurement.

A set of holmium oxide, didymium oxide, and neutral density filters (0.1OD, 0.5OD, 1.0OD) along with a quartz slide are used to perform a wavelength and a photometric calibration/accuracy check for the MSP.

- a. Perform the Performance Check by selecting “Auto Calibration – Transmission” from the “Tools” menu.
- b. Run Autoset Optimize, Dark Scan, and Reference Scan.
- c. Follow instrument prompts to perform the wavelength and photometric calibration.

## 2. Evaluation

- a. The instrument will indicate if all performance checks are within manufacturer’s pre-established limits.
- b. If all of the performance checks are within the manufacturer’s calibration limits, the instrument shall be deemed for use in casework. If it is not within those limits, the instrument shall not be used for casework.
- c. A report can be printed out to indicate a pass for the wavelength and photometric checks.
- d. If the instrument is not within the limits, ensure the filters are lying flat. Check Köhler illumination and repeat the performance check.

- e. Do not proceed with casework samples until all of the filter peak mark values are within an acceptable range of the NIST certified reference values (as determined by the computer).
- f. Notify an appropriate Lead Examiner, Deputy Director, or CRAIC technical support if values are out of the acceptable ranges as indicated by the calibration report.

**The following are applicable to all instruments:**

- User manuals should be kept either next to the instrument or in a place that is readily available to analysts.
- Analysts may refer to the manufacturer's user manual for instrument operating and maintenance instructions.
- Analysts should consult with the manufacturer's documents for guidance on the proper handling, use, and storage of reference standards and operability.
- Efforts will be made to ensure the safe handling, transport, storage, and use of reference standards in order to prevent contamination/deterioration and to protect the standard's integrity.
- Reference standards should be free from defects and should provide reliable data that can be used to measure instrument performance. If any question arises regarding the reliability or quality of a reference standard, analysts should consult either a Technical Lead or the appropriate Deputy Director.
- When an instrument doesn't pass verification (i.e., QA/QC), then casework data should not be generated from that instrument. If an instrument can't be fixed so that it satisfies verification, then that instrument should be taken out of service.
- An 'Out of Service' sign must be placed on all instruments taken out of service so other analysts will know not to use the instrument. The instrument must satisfactorily pass all QA/QC measures (i.e., be verified) prior to being placed back into service.
- When an instrument is out of service, appropriate measures should be performed in order to rectify problems and to get the instrument operational again (i.e., troubleshoot, contact Technical Lead/Deputy Director, or contact service engineer).
- Parameters for QA/QC testing should be recorded within the saved data file on the instrument.
- When an instrument is determined to be out of service, analysts may need to refer back to previously acceptable verification data to review for acceptability.

**E. References:**

Operating the Perkin-Elmer Spectrum 100 FT-IR Spectrophotometer, operator's manual.

Spectrum 100 FT-IR User's Guide; Perkin-Elmer, 2008

Spectrum 100 FT-IR Help Topics; Instrument Validation.

Spectrum 100 FT-IR Help Topics; Performing an instrument validation

CRAIC technologies User Manual

Clark's Isolation and Identification of Drugs in pharmaceuticals, body fluids, and post-mortem materials, The Pharmaceutical Society of Great Britain.

RETIRED

## Rev. #

## History

2

Title change. Significant format and verbiage changes throughout document. Removed 'Trace Section' from Purpose and Responsibility sections. Slightly modified definitions section. Changes made within Procedure section which made the procedure less strict and better to understand. Removed strict adherence to expiration date on reference standards. Manuals no longer are required to be directly next to instrument. Gave more guidance when instrument goes out of service. Added specific steps to operation and added evaluation criteria. Added MSP calibration/verification information.

RETIRE