**1.0 Purpose** - This procedure specifies the required elements for the performance verifications, quality control checks, and use of *Perkin Elmer* Fourier Transform Infrared Spectrophotometer with Universal Attenuated Total Reflectance (ATR) Sampling Accessories.

**2.0 Scope** - This procedure applies to the infrared spectrophotometer used in the Drug Chemistry section of the Pitt County Sheriff's Office Forensic Services Unit.

**3.0 Definitions**

- **Performance verification -** The initial confirmation of the reliability of a previously or externally validated method or instrument.
- **Quality control (QC) check -** Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
- **Reference Material** – Material sufficiently homogeneous and stable, with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

**4.0 Equipment, Materials, and Reagents**

**4.1 Equipment**

- *Perkin Elmer* Fourier Transform Infrared Spectrophotometer with Universal Attenuated Total Reflectance (ATR) Sampling Accessory

**4.2 Materials and Reagents**

- Traceable Reference Material (TRM) Polystyrene film
- TRM Polystyrene film standard spectra
- Printer
- Spatula
- Methanol or other suitable organic solvent (ACS grade or higher)
- Water

**5.0 Procedure**

**5.1 Standards and Controls**

**5.1.1 Naming of Instrument Files**

**5.1.1.1** Instrument files used for documenting performance verifications and monthly/yearly QC checks shall be named with at least the instrument identifier, year/month/day and a description (abbreviations are acceptable).

**5.1.1.2** Instrument files published to a casefile for blanks, sample scans, and spectral subtractions shall be named with at least the case number, item number and a description (abbreviations are acceptable).

**5.1.1.3** If multiple spectral subtractions are performed on the same sample and published to the case record, each resulting scan shall be named as above, with a unique identifier for each subtraction.

**5.1.2 Saving of Instrument Files**

**5.1.2.1** Instrument files described above shall be saved on the instrument hard drive, at least until they are archived.

**5.1.2.2** Instrument files described above shall be placed in a file named with the instrument identifier, year, and month in which they were collected, and then archived to the Pitt County Sheriff's Office external hard drive attached to the instrument.

**5.1.3 Operating parameters** – The following operating parameters shall be used in the Drug Chemistry Section:

- Accumulations – four scans
- Resolution 4.00 $cm^{-1}$
- Range 4000.00 to 650.00 $cm^{-1}$
- $CO_2/H_2O$ Correction "On"
- Diamond/Zinc Selenide crystal – one bounce
- Threshold for interpolated peaks – 1.00 %T

**5.1.4 Negative control**

**5.1.4.1** Perform a background spectrum upon instrument start up, and at the beginning of each day the instrument is in use. Additional background spectrums may be obtained as needed.

**5.1.4.2** Perform a blank (clean sample path) in the same manner as the sample to be analyzed.

**5.1.4.3** An acceptable blank spectrum does not exhibit extraneous peaks indicative of contamination.

**5.1.4.4** If the blank is contaminated, clean the crystal again and repeat the blank until no contamination is present.

**5.1.4.5** An acceptable blank (clean sample path) shall be obtained between each new sample scan obtained.

**5.1.5 Positive control - Monthly QC Check**

**5.1.5.1** A Chemist shall obtain a polystyrene scan monthly to ensure proper functioning, and record the completion of the Monthly QC Check.

**5.1.5.2** The internal polystyrene may be used for the Monthly QC Check.

**5.1.5.2.1** Name the instrument file according to the instructions above, and label the resulting peaks utilizing instrument software.

**5.1.5.2.2** Label the scan with notations for internal polystyrene and Chemist's initials and date.

**5.1.5.2.3** Print the labeled scan as a .pdf file and save on the instrument hard drive.

**5.1.5.2.4** Copy the .pdf file to the Document Management (DM).

**5.1.5.2.5** After evaluation of data (see below) record completion in the instrument log for QC checks.

**5.1.5.3** Evaluate the designated wave numbers from the Monthly QC check data and compare to the Traceable Reference Material polystyrene film (traceable to the NIST Standard Reference Material (SRM) 1921b.

**Traceable Polystyrene Reference Material Parameters**

3060 $cm^{-1}$ (+/- 1 $cm^{-1}$)
2850 $cm^{-1}$ (+/- 1 $cm^{-1}$)
1943 $cm^{-1}$ (+/- 1 $cm^{-1}$)
1601 $cm^{-1}$ (+/- 1 $cm^{-1}$)
1583 $cm^{-1}$ (+/- 1 $cm^{-1}$)
1154 $cm^{-1}$ (+/- 1 $cm^{-1}$)
1028 $cm^{-1}$ (+/- 1 $cm^{-1}$)

**5.1.5.4** The allowable variance from the certified values shall be within the data interval parameters of the instrument (+/- 1 $cm^{-1}$). If the results are outside these specifications, the instrument shall be removed from casework immediately and the following shall be done:

- Place an "Out of service" sign on the front of the instrument and enter the Out of Service status in the logbook.
- Notify the Section Technical Leader so he/she can call the service engineer to schedule an on-site assessment and document in the instrument log.

### 5.1.6 Yearly Internal Polystyrene QC Check

**5.1.6.1** A scan of a Traceable Reference Material polystyrene film shall be collected yearly for each instrument with the KBR accessory in place, followed by the collection of a scan of the internal polystyrene with the ATR attachment in place.

**5.1.6.2** The instrument file and .pdf scans generated for the yearly QC Check shall be handled according to the specifications listed above for the monthly QC check, except for resetting the instrument expiration date, which has been designated for use as a monthly reminder.

### 5.1.7 Initial Performance Verification for New Instrument Set Up

**5.1.7.1** New FTIR instruments shall be installed by a certified engineer according to the manufacturer's guidelines, and procedure specifications.

**5.1.7.2** External (and internal) polystyrene scans shall be obtained according to the procedure for Monthly QC Check listed above.

**5.1.7.3** Scans from at least three controlled substance primary standards shall be obtained (e.g., methamphetamine, cocaine base, and heroin). Other controlled substances may be used depending on the availability of standards. The data obtained shall be reviewed by the Technical Leader and found to be substantially the same as the library standard for that compound.

**5.1.7.4** The .pdf files generated during the performance verification shall be filed in DM by the Technical Leader to document set up of the new instrument.

**5.1.7.5** If the polystyrene checks are acceptable, and the controlled substance standard spectra are found to be substantially the same as the library standard for that compound, the instrument shall be released for casework. A new entry for the instrument shall be made in DM prior to use in casework. The new entry shall include:

- The manufacturer's serial number for the FTIR and the ATR attachment.
- The unique section identifier for the new instrument.
- Statement by the Technical Leader that the instrument has been released for casework.
- A notation to reflect the date the initial performance validation was completed.

## 5.2 Suggested Maintenance Schedule

**5.2.1** Yearly preventive maintenance shall be performed by an approved vendor.

**5.2.2** Desiccants shall be changed at six month intervals, or sooner when needed if external indicators begin to change color.

**5.2.3** Record completion of maintenance and repairs, the date and identity of the person performing the work in the instrument log for Maintenance/Repairs. The instrument log shall be kept in a notebook near the instrument.

**5.2.4** Document the maintenance or repair by listing the name of the vendor/person performing the work, and the work performed on the instrument. Any documentation associated with a service call shall be recorded in DM.

### 5.3 Shutdown/Startup

- The power switch to the infrared instrument shall be left ON at all times to ensure the optics stay warm and excess moisture does not build up in the instrument.
- The software and computer may be shut down at the end of each business day.
- Each time the software is restarted, a background and an acceptable blank (clean sample path) shall be performed.
- When an IR has been placed out of service for an extended period of time, document the status by entering it as "Out of Service" in the instrument log.
- A successful QC Check (as outlined above) is required following an "Out of Service" status, and routine maintenance when the instrument has been out of direct control of the laboratory. Document the QC Check by adding an entry in the logbook when the instrument is returned to casework.
- Chemists shall examine the effect(s), if any, of a malfunction on analysis results and implement the Procedure for Corrective Action as required.

### 5.4 Application of Procedure on Evidence

#### 5.4.1 Solid samples using the ATR

**5.4.1.1** Clean the ATR sampling accessory crystal using water or an organic solvent. Ensure the crystal is completely dry.

**5.4.1.2** Perform a background scan at least daily and additional backgrounds as needed (e.g., when atmospheric conditions warrant.)

**5.4.1.3** Perform the negative control check as described above.

**5.4.1.4** Print the results of the blank (clean sample path) for the master case file. The blank scan may be ATR and baseline corrected before it is printed for the master case file.

**5.4.1.5** Place approximately 1 milligram of sample evenly onto the ATR crystal.

**5.4.1.6** Apply force using the ATR force arm to ensure good contact between the sample and the surface of the crystal.

**5.4.1.7** Scan to acquire data.

**5.4.1.8** Data can now be processed using ATR and auto baseline corrections.

### 5.4.2 Liquid samples using the ATR

**5.4.2.1** Clean the ATR sampling accessory crystal using water or an organic solvent. Ensure that the crystal is completely dry.

**5.4.2.2** Perform the negative control check as described above.

**5.4.2.3** Apply enough liquid sample to cover the ATR crystal, leaving the pressure arm unengaged.

**5.4.2.4** Scan to acquire data.

**5.4.2.5** Data can now be processed in the same manner as solid samples. (See above)

### 5.4.3 Criteria for Initial Evaluation:

**5.4.3.1** The spectral peaks obtained from an unknown shall be evaluated prior to comparison to ensure they are of sufficient intensity to make an accurate comparison to known reference standards or published spectral data.

**5.4.3.1.1** Any peaks generated that are below 100% transmission (for FTIR) and /or 100% reflectance (for ATR) will be considered of sufficient intensity and will be valid for further comparison.

### 5.4.4 Identification:

**5.4.4.1** If the chemist, based on his/her training and experience, determines that the spectrum of the controlled substance does not correspond to the reference standard due to the presence of other substances in the mixture, the controlled substance shall be separated from the mixture and an IR spectrum obtained of the isolated controlled substance.

**5.4.4.2** A known impurity within a mixture containing a controlled substance may also be subtracted from the IR spectrum by using the "difference" function of the FTIR. A printout of the straight material before any spectral subtractions are performed shall be required for the master case file.

**5.4.4.3** When using FTIR as the primary structural elucidation technique:

**5.4.4.3.1** The overall sample spectral pattern (relative peak intensities and wavenumbers) shall correspond to that of the reference spectrum.

**5.4.4.3.2** The observed wavenumbers for six prominent and well-defined signals in the sample spectrum between 2000 $cm^{-1}$ to 650 $cm^{-1}$ shall be within +/- 2 $cm^{-1}$ of those in the reference spectrum.

- NOTE: This correspondence shall be demonstrated by displaying these measured wavenumbers on each sample spectrum and the reference spectrum.

**5.4.4.3.3** The sample spectral pattern between 4000 $cm^{-1}$ and 2000 $cm^{-1}$ corresponds to that of the reference spectrum.

**5.4.4.3.4** No prominent extraneous signals are observed in the sample spectrum.

**5.4.4.4** When using FTIR to differentiate cocaine base from cocaine hydrochloride or another salt form, the areas of the spectrum which are different between cocaine base and cocaine hydrochloride shall be clear. Other areas may have interfering peaks present that do not mask the "salt form" identity.

**5.4.5** **Documentation:** Print the properly labeled data generated by the FTIR/ATR instrument to a .pdf file and import to the master case file.

**5.4.6** The reference standard used for identification, and any reference standards used for spectral subtraction(s) shall also be imported to the master case file if a positive identification of a controlled substance is made.

**5.4.6.1** If the reference standard is from a Laboratory generated collection, the unique identifier shall be included in the master case file.

**5.4.6.2** If the reference standard is from a published source, a citation for the source shall be included in the master case file.

## 5.5 Reporting

**5.5.1** When a sample's infrared spectrum indicates a mixture of a controlled substance(s) and non-controlled substance(s), the ratio may be evaluated based on the training and experience of the chemist. If the overwhelming majority of the sample is indicated to be non-controlled, then the reported results shall indicate that the material contains the controlled substance(s).

**5.5.1.1** Suggested Examples:
Item 1:
Material containing Cocaine – Schedule II.

Net weight of material – 2.51 (+/- 0.0X) grams (confidence level 99.7%).

**5.6 Sampling -** See Technical Procedure for Sampling.

## 6.0 Limitations

**6.1** Generally, infrared spectra cannot distinguish between optical isomers.

**6.2** Compounds may exist in different crystal forms which may produce unique spectra. (Mannitol is an example of one compound that exhibits these polymorphic characteristics.)

**6.3** Due caution shall be exercised when using the similarity index generated by the library search algorithm. The chemist shall evaluate the data and not singularly rely on the computer software index.

## 7.0 Safety - Do not over tighten the force gauge.

## 8.0 References

Moffat, A.C., et al, ed. *Clarke's Analysis of Drugs and Poisons.* 4th Edition. London: Pharmaceutical Press, 2011.

Moffat, A.C., et al., ed. *Clarke's Isolation and Identification of Drugs*. 2nd Edition. London: Pharmaceutical Press, 1986.

Mills, III, Terry and J. Conrad Roberson. *Instrumental Data for Drug Analysis*. 2nd Edition. CRC Press,Inc.: Volumes 1-5, 1993.

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Sliverstein, Robert M., et al. *Spectrometric Identification of Organic Compounds*. 5th Edition. New York Wiley, 1991.

Keller, Roger. *The Sigma Library of FT-IR Spectra*. 1st Edition. Missouri: Sigma Chemical Company, Volumes 1 and 2, 1986.

Pouchert, Charles J. *The Aldrich Library of Infrared Spectra*. Aldrich Chemical Company: 1981.

ASTM Standard E-1252, 2013, "Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis." ASTM International: West Conshohocken PA, 2013, www.astm.org.

ASTM Standard E334-01, Reapproved 2013, "Standard Practice for General Techniques of Infrared Microanalysis." ASTM International: ASTM International: West Conshohocken PA, 2019, www.astm.org.

## 9.0 Records

- Initial Performance Verification of new instruments in DM
- Traceable Polystyrene Film Infrared Spectrum in DM
- Maintenance and QC Checks in Instrument log, and archived in DM
- Spectra .pdf data imported to master case files

| REVISION HISTORY | | |
|---|---|---|
| **CURRENT VERSION** | **EFFECTIVE DATE** | **SUMMARY OF CHANGES** |
| 1 | 2017/11/14 | Original Document. |
| 2 | 2018/04/01 | **5.1.2.2** – Removed compressed zip file requirement for archiving.<br>**Entire document** - changed case file to master case file, updated "Illicit Drugs" to "Drug Chemistry" section |
| 3 | 2018/10/22 | **Entire document –** Updated all references to Document Management System (DM).<br>**5.1.3 –** Updated "Illicit Drugs" to "Drug Chemistry".<br>**5.1.5.2.4** – Replaced Records Management System with DM.<br>**5.1.5.3 and 5.1.5.4 –** Changed Data Interval to +/- 1.<br>**5.1.7.5 –** Updated wording for acceptable criteria to release instrument for casework.<br>**5.4.1.9 –** Removed repeated "master."<br>**5.5.1.1 –** Updated suggested examples for reporting "material containing…." |
| 4 | 2019/04/02 | **5.1.3** – Added "Accumulations – four scans" to operating parameters. Range of data collection changed to 4000-650 $cm^{-1}$ instead of 4000-550 $cm^{-1}$<br>**5.1.5.2.2** – Removed references to external polystyrene<br>**5.1.5.4** – "resolution" changed to "data interval parameters"<br>**5.4.1.8** – Data processing instructions rewritten to reflect new software changes.<br>**Original 5.4.1.11** – Deleted statement reference comparison to known standard.<br>**5.4.1.9 –** Statement added to clarify evaluation of the unknown to identify characteristics suitable for comparison.<br>**5.4.1.10** to **5.4.1.13 –** Moved and changed the order – see **5.4.5**<br>**Original 5.4.2.6 (now 5.4.3.1)** – Changed "compare favorably" to "correspond"<br>**Original 5.4.2.8** – Removed statement reference "compare favorably."<br>**5.4.3** – Edited requirements for identification and printing of resulting data.<br>**5.4.4** – Created new "Documentation" section and moved **original 5.4.1.11 and 5.4.1.13** to here.<br>**References** – Added Clarke's Analysis of Drugs and Poisons, 4th Edition, and added ASTM E334-01.<br>**9.0** – Removed "instrument files" duplication |
| 5 | 2019/04/17 | **Revision History for Version 4 -** Made corrections to **5.4.3** and **5.4.3.3** |

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|---|---|---|
| | | **5.1.3** – Added parameters for threshold of interpolated peak (1.00%T).<br>**5.4.3** – Added new section "Criteria for Initial Evaluation"<br>**5.4.3.1** – Moved **Original 5.4.3** and reworded<br>**5.4.3.1.1** – Added clarification for meaning of sufficient intensity.<br>**5.4.4.3.2 -** Clarified peak labeling requirements for identification. |
| 6 | 2019/05/22 | **Header** – Changed "Instruments" to "Drug Chemistry" and edited title and Technical Leader designation.<br>**5.1.5.3** – Added reference to NIST SRM1921b traceability. Edited chart to reflect whole number values and added all TRM values for comparison, and removed references to Perkin Elmer and Nicolet brand instruments. |
| 7 | 2020/01/15 | **Materials and Reagents –** Added ACS grade or higher to organic solvents.<br>**References** – Updated ASTM E1252 to more current version. |