Technical Procedure for Infrared Spectroscopy

1.0 Purpose - This procedure specifies the required elements for the performance check and use of Fourier Transform Infrared Spectrophotometers.

2.0 Scope - This procedure applies to all infrared spectrophotometers used in the Drug Chemistry Sections of the State Crime Laboratory.

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

- 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 (see Certified Polystyrene in Section Files)
- Printer/ink cartridge/paper and/or Print2PDF capability with instrument network
- Spatula
- Methanol or other suitable organic solvent
- Water

5.0 Procedure

5.1 Standards and Controls

5.1.1 Negative control

- Perform a contamination check in the same manner as the sample to be analyzed.
- If the check fails, clean the crystal again and repeat the contamination check until no contamination is present.
- A clean contamination check shall be obtained between each sample scan obtained.

5.1.2 Monthly QC Check

5.1.2.1 A Forensic Scientist shall obtain a polystyrene scan monthly for each instrument to ensure proper functioning.

5.1.2.2 The internal polystyrene or external TRM polystyrene may be used for the Monthly QC Check.

5.1.2.2.1 Label the scan with notations for internal or external (TRM) polystyrene (include serial number if TRM polystyrene was used) and analyst's initials and date. Print the data using a hard copy, or Print2PDF software.

5.1.2.3 Evaluate the designated wave numbers from the Monthly QC check data and compare to the Traceable Reference Material polystyrene film. Refer to the chart below for parameters.

Traceable Polystyrene Reference Material Parameters
***Perkin Elmer and Nicolet 380 FTIR**

3082.XX cm^{-1} (+/- 4 cm^{-1})
3060.XX cm^{-1} (+/- 4 cm^{-1})
1601.XX cm^{-1} (+/- 4 cm^{-1})
1583.XX cm^{-1} (+/- 4 cm^{-1})
1028.XX cm^{-1} (+/- 4 cm^{-1})

***Refer to current certificate for Polystyrene Standard Reference Material for full peak wave numbers.**

5.1.2.4 The allowable variance from the certified values shall be within +/- the resolution of the instrument. 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.
- Notify the Section IR Key Operator so he/she can call the Service Engineer to schedule an on-site assessment.

5.1.2.5 The monthly polystyrene scan shall be filed by the Forensic Scientist who collected the data. Records shall be maintained by the IR Key Operator.

- Record completion in the instrument log for QC checks. (See Monthly QC Check Log in Logbook and see Section files for printable version.)

5.1.3 Yearly Internal Polystyrene QC Check (Applicable only to Perkin Elmer FT-IRs)

5.1.3.1 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. This shall be performed by the Section IR Key Operator or designee.

5.1.3.2 Print both scans according to the specifications for the monthly QC check.

5.1.3.3 The Section IR Key Operator is responsible for evaluating the data according to the specifications for the monthly QC checks. The allowable variance from the certified value for the peaks appearing at the listed wave

numbers shall be within +/- the resolution of the instrument. 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.
- Notify the Section IR Key Operator so he/she can call the service engineer to schedule an on-site assessment.

5.1.3.4 This data shall be filed and maintained by the Section IR Key Operator.

- Record completion in the instrument log for QC checks. (See Instrument Log for Monthly/Yearly QC Checks in logbook, and see Section Files for printable version.)

5.1.4 Performance Verification for New Instrument Set Up

5.1.4.1 New FT-IR instruments shall be installed by a certified engineer according to the manufacturer's guidelines.

5.1.4.2 Internal (if applicable) and external polystyrene scans shall be obtained according to the procedure for Yearly Internal Polystyrene Verification listed above.

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

5.1.4.4 The data shall be filed and maintained by the Section IR Key Operator to document set up of the new instrument.

5.1.4.5 If the polystyrene checks are acceptable, and the controlled substance standard spectra match the respective library entries, the instrument shall be released for casework. A new entry for the instrument shall be made in the Resource Manager section of FA prior to use in casework. The new entry shall include:

- The manufacturer's serial number.
- The unique section identifier for the new instrument. Infrareds are numbered in numerical order with the notation "FTIR" in front of the number.
- A notation under "Verification Date" to reflect the date the performance verification was completed.

5.2 Suggested Maintenance Schedule

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

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

5.3 Record completion of maintenance and repairs, the date and identity of person performing the work in the instrument log for Maintenance/Repairs. (See FTIR Maintenance Log in Section files for printable version.) The instrument log shall be kept in a notebook near the instrument. If converted to electronic format, the maintenance/repair records shall be maintained by the IR Key Operator and stored in section files.

5.4 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 clean contamination check shall be performed.
- When an IR has been placed out of service (e.g., maintenance, malfunction, leaving the direct control of the Laboratory), correct operation shall be demonstrated by a performance verification following the procedure for a Monthly QC Check outlined above.
- Laboratory personnel shall examine the effect(s), if any, of a malfunction on analysis results and implement the Procedure for Corrective Action as required.

5.5 Application of Procedure on Evidence

5.5.1 Solid samples using the ATR Method

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

5.5.1.2 Perform a background scan at least daily, and additional backgrounds as needed. (For example, when instrument is restarted after power down or when atmospheric conditions warrant.)

5.5.1.3 Perform the negative control check as described above in **5.1**.

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

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

5.5.1.6 Scan to acquire data.

5.5.1.7 Data can now be processed. The following operating parameters shall be used in the Drug Chemistry Section:

- Resolution 4.00 cm^{-1}
- Range 4000.00 to 550.00 cm^{-1}
- CO_2/H_2O Correction "On"
- Diamond/Zinc Selenide crystal – one bounce

5.5.1.7.1 "ATRNoPrint" Macro:

- Delete
- Scan sample (4 times)
- ATR Correction, 0.00
- Auto baseline correction, 4000.00-550.00

5.5.1.8 Spectral subtractions may be used as a means to remove diluents from the IR of controlled substances. If this technique is used, a printout of the straight material before any spectral subtractions are performed shall be required for the FA case record.

5.5.1.9 Print the data generated by the FT-IR instrument for the FA case record. Label the generated data with the case number, item number, date, and instrument identifier, for inclusion in the FA case record. If a hard copy is obtained, it shall be labeled with initials and date before being scanned into the FA case record.

5.5.1.9.1 If more than one scan is obtained from the same sample, labels shall be affixed noting the identity of the scan, such as an extraction, solvent wash, or spectral subtraction. .

5.5.1.10 Compare the completed scan to a known reference standard.

5.5.1.11 The reference standard shall also be included in the FA case file if a positive identification of a controlled substance is made.

5.5.2 Liquid samples using the ATR Method

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

5.5.2.2 Perform the negative control check as described above in **5.1.**

5.5.2.3 Apply enough liquid sample to cover the ATR crystal.

5.5.2.4 Scan to acquire data.

5.5.2.5 Data can now be processed. The following operating parameters shall be used in the Drug Chemistry Section:

- Resolution 4.00 cm^{-1}
- Range 4000.00 to 550.00 cm^{-1}
- CO_2/H_2O Correction "On"
- Diamond/Zinc Selenide crystal – one bounce

5.5.2.5.1 "ATRNoPrint" Macro:

- Delete

- Scan sample (4 times)
- ATR Correction, 0.00
- Auto baseline correction, 4000.00-550.00

5.5.2.6 Print the data generated, labeled with at least the case number, item number, date, and instrument identifier, for inclusion in the FA case record.

5.5.2.6.1 If more than one scan is obtained from the same sample, labels shall be affixed noting the identity of the scan, such as an extraction or solvent wash.

5.5.2.7 Compare the completed scan to a known reference standard.

5.5.2.8 The reference standard shall also be included in the FA case file if a positive identification of a controlled substance is made.

5.5.2.9 **Identification:** If the Forensic Scientist, based on his/her training and experience, determines that the spectrum of the controlled substance does not compare favorably 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.

5.5.2.9.1 A known impurity within a mixture containing a controlled substance can also be subtracted from the IR spectrum by using the "difference" function of the FT-IR.

5.5.2.9.2 An IR spectrum of a controlled substance shall compare favorably to the IR spectrum of a known reference standard before an identification is confirmed.

5.5.2.10 When using FT-IR as the primary structural elucidation technique, the sample spectrum shall compare favorably with a spectrum of a known standard in both its overall appearance and in the presence and location of the peaks.

5.5.2.11 When using FT-IR 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.

5.6 Sampling - See Drug Chemistry Section Administrative Procedure for Sampling.

5.7 Calculations - N/A

5.8 Uncertainty of Measurement - N/A

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 Forensic Scientist 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 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.

Mills, III, Terry, et al. *Instrumental Data for Drug Analysis*. 3rd Edition. CRC Press, Inc.: Volumes 6-7, 1996.

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, 2002, "Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis." ASTM International: West Conshohocken PA, 2002, www.astm.org.

9.0 Records

- Monthly/yearly polystyrene checks
- Traceable Polystyrene Film Infrared Spectrum
- Instrument log for monthly and yearly polystyrene checks.
- Data generated from case work – included in the FA case record.
- Maintenance Log

10.0 Attachments – N/A

Revision History

Effective Date	Version Number	Reason
09/17/2012	1	Original Document Technical Procedure F-01 converted to ISO standards. Instrument log added for monthly/yearly QC checks. Print2PDF new option for storage of polystyrene data. Instructions added for collection of polystyrene data and for desiccant changes. KBr Methods rescinded. They may be reinstated by Drug Chemistry Section FSM.
02/15/2013	2	Scope – changed to include Raleigh, Triad and Western Laboratories. **5.1.2.2** Removed operating instructions for Monthly QC check. **5.1.2.2.1** Add date to scan. Remove operating instructions for software. **5.1.2.4** List allowable variance for each instrument type. **5.1.3** Only applies to Perkin Elmer FT-IRs. Nicolet FT-IRs have no internal polystyrene. **5.1.4.2** Internal Polystyrene only applicable to Perkin Elmer FT-IRs. **5.2** Remove instructions for desiccant change. Corrected typo on "desiccant." **5.5.2.9.1** Formatting error corrected on quotation marks. **5.5.2.10** Removed the word "major."
03/08/2013	3	Purpose – Remove reference to Perkin-Elmer to accommodate equipment in all three laboratories. **5.1.2.4** – Removed reference to +/- 0.50 cm^{-1} for Perkin-Elmer instruments so allowable variance is +/- 1.0 cm^{-1} for all instruments. **5.1.3.3** – Changed reference to +/- 1.0 cm^{-1} instead of 0.5 cm^{-1} for yearly polystyrene checks.
05/03/2013	4	**5.5.2.9** - Changed wording for substantially **5.5.2.9.2** - Changed wording for substantially comparable.
05/10/2013	5	**5.1.2.3, 5.1.2.4, 5.1.3.3** – Revised parameters for Monthly QC check to match resolution of the instruments. **5.5.1.7, 5.5.2.5** – Amended instrument resolution to match instrument parameters. **5.6** – Revised name of Sampling Plan from Technical to Administrative Procedure.
11/15/2013	6	Added issuing authority to header.