Technical Procedure for Infrared Spectroscopy

1.0 Purpose – This procedure specifies the required elements for the calibration and use of the Perkin-Elmer Fourier Transform Infrared Spectrophotometer.

2.0 Scope – This procedure applies to all infrared spectrophotometers used in the Trace Unit.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

- Fourier Transform Infrared Spectrophotometer
- Microscope accessory
- Universal Attenuated Total Reflectance (ATR) sampling accessory
- Printer or Print2PDF capability
- Traceable polystyrene film standard
- KBr discs
- Pellet holder
- Microcompression cell with diamond windows
- Probes
- Methanol (or other organic solvent)
- Water
- O-ring

5.0 Procedure

5.1 Monthly Performance Check

5.1.1 A spectrum of a traceable polystyrene standard shall be obtained monthly from each instrument to ensure proper functioning (performance check). The following instrument parameters shall be used:

- Scans = 16
- Resolution = 4 cm^{-1}
- Gain = 1.0
- Range = at least 4000-450 cm^{-1}

5.1.2 The spectrum with peaks labeled to two decimal places shall be electronically archived.

5.1.3 The allowable variance from the certified value for band #14 located at 3082.22 is +/- the resolution of the instrument. Each time a new standard is acquired, this value shall be adjusted. If the target peak is outside of the allowable variance, perform a wavelength calibration.

5.1.3.1 Wavelength calibration

5.1.3.1.1 In the Spectrum software, go to the "wavelength calibration" section and enter the expected and observed band positions.

5.1.3.1.2 Rerun the traceable polystyrene standard and check the positions of the known bands. If they are now within the allowable variance, the instrument shall remain in service.

5.1.3.1.3 If they are still not within the allowable variance, the instrument shall be immediately removed from casework and maintenance shall be performed. Once the known bands of the traceable polystyrene standard are within the allowable variance, the instrument shall be returned to service.

5.1.4 Record the date and results of the monthly performance check in the IR logbook next to the instrument.

5.2 Performance Verification for New Instrument Set-Up

5.2.1 New FTIR instruments shall be installed by a certified engineer according to the manufacturer's guidelines.

5.2.2 A spectrum of a traceable polystyrene standard shall be obtained using the procedure for Monthly Performance Check **(5.1)**.

5.2.3 Spectra from ten (10) typical trace evidence samples shall be analyzed using the new instrument and shall be compared to the same ten samples analyzed using a previously validated instrument. The data obtained shall be reviewed by the FTIR Key Operator to determine if they are substantially the same.

5.2.4 If the target peak in the traceable polystyrene standard is within acceptable limits, and if the ten trace samples from the new instrument and the previously validated instrument are substantially the same, then the instrument shall be approved for use in casework.

5.3 Casework Analysis

5.3.1 The spectra of questioned samples are compared to the spectra of other samples and/or to the spectra of known standards.

5.3.2 Sample preparation techniques are dependent on the type of sample to be analyzed. The most frequently used sample preparation techniques are listed below.

5.3.3 Analysis of Samples using the Bench ATR

5.3.3.1 Clean the ATR sampling accessory crystal using water or an organic solvent, such as methanol. Ensure the crystal is completely dry.

5.3.3.2 Acquire a background scan.

5.3.3.3 Place sample onto the ATR crystal.

5.3.3.3.1 If the sample is in a liquid form, place a drop onto the crystal. An O-ring may be used to contain the sample.

5.3.3.3.2 If the sample is in a solid form, place a small amount of the sample onto the crystal and apply force using the ATR force arm to ensure good contact between the sample and the surface of the crystal.

5.3.3.4 Scan to acquire data using a minimum of 4 scans with a scan range of 4000-650 cm^{-1}.

5.3.3.5 Between each sample, clean the crystal with water or an organic solvent, such as methanol, and perform a contamination check to ensure that the crystal has been properly cleaned. If the contamination check fails, repeat the cleaning procedure until a successful contamination check is obtained.

5.3.4 Analysis of Samples using an IR Microscope

5.3.4.1 Flatten a sample and place onto a clean KBr disc.

5.3.4.2 Place the disc on the stage of the microscope and focus on the sample.

5.3.4.3 Adjust the apertures to fit the sample and mark the sample.

5.3.4.4 Move off the sample and choose a clean area on the KBr disc to select and mark the background position.

5.3.4.5 Scan to acquire data using a minimum of 4 scans with a scan range of 4000-600 cm^{-1}.

5.3.5 Analysis of Samples using a Microcompression Cell with Diamond Windows

5.3.5.1 Clean the diamond windows before and after analysis with a solvent such as methanol.

5.3.5.2 Place the sample between the windows and hand-tighten the cell to compress the sample.

5.3.5.3 Place the cell on the microscope accessory and focus on the sample.

5.3.5.4 Adjust the apertures to fit the sample and mark the sample.

5.3.5.5 Move off the sample and choose a clean area on the diamond cell, select and mark the background position.

5.3.5.6 Scan to acquire data using a minimum of 4 scans with a scan range of 4000-600 cm^{-1}.

5.4 Maintenance

5.4.1 Desiccant shall be changed every six (6) months at a minimum.

5.4.2 A qualified service engineer shall perform preventative maintenance on the instrument annually.

5.5 Standards and Controls

5.5.1 The traceable polystyrene standard shall be either replaced or recertified prior to its expiration date.

5.5.2 There are no special storage requirements for the polystyrene standard.

5.6 Sampling and Sample Selection – N/A

5.7 Calculations – N/A

5.8 Uncertainty of Measurement – N/A

6.0 Limitations - The optimum wavenumber ranges for the instruments' accessories are as follows:

- Bench: 7800-350 cm^{-1}
- Universal ATR: 7800-650 cm^{-1}
- Microscope Single Point Detector: 7800-600 cm^{-1}
- Microscope Array Detector: 7800-720 cm^{-1}
- Microscope ATR: 5500-600 cm^{-1}

7.0 Safety

7.1 Note all warning labels on the body of the instrument (e.g., desiccant, beamsplitter, and laser radiation warnings.)

7.2 Liquid nitrogen can burn skin and eyes. Care shall be exercised when filling the Dewar.

7.3 Be aware of pressure build up in the Dewar. High pressure can forcefully propel a funnel or detector cap upward from the Dewar. Allow sufficient time for the nitrogen overflow to dissipate and the pressure to equalize before replacing the Dewar cap.

8.0 References

8.1 ASTM / SWG Guidelines

ASTM Standard E1252, 1998 (2002), "Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis." ASTM International, West Conshohocken, PA, 2002.

ASTM Standard E1610, 2002 (2008), "Standard Guide for Forensic Paint Analysis and Comparison." ASTM International, West Conshohocken, PA, 2008.

ASTM Standard E334, 2001 (2007), "Standard Practice for General Techniques of Infrared Microanalysis." ASTM International, West Conshohocken, PA, 2007.

ASTM Standard E2224, 2002, "Standard Guide for Forensic Analysis of Fibers by Infrared Spectroscopy." ASTM International, West Conshohocken, PA, 2002.

Scientific Working Group for Materials Analysis (SWGMAT), "Forensic Fiber Examination Guidelines." *Forensic Science Communications* 1.1 (1999).

Scientific Working Group for Materials Analysis (SWGMAT), "Forensic Paint Examination and Comparison Guidelines." *Forensic Science Communications* 1.2 (1999).

Scientific Working Group for Materials Analysis (SWGMAT). "Guideline for the Forensic Examination of Pressure-Sensitive Tapes." Forensic *Science Communications* 10.4 (2008).

8.2 Books

Caddy, B., ed. *Forensic Examination of Glass and Paint*. New York: Taylor & Francis, 2001. Chapter 10: Use of Infrared Spectroscopy for the Characterisation of Paint Fragments.

Humecki, J.J., ed. *Practical Guide to Infrared Microspectroscopy*. New York: Marcel Dekker, Inc. 1995.

Robertson, J. and M. Grieve, eds. *Forensic Examination of Fibres.* 2nd Ed. London: Taylor and Francis, 1999. Chapter 8: Infrared Microspectroscopy of Fibres.

8.3 Training Materials

FBI Laboratory. *Forensic Analysis of Paints* workshop held in Quantico, VA, April 26-30, 2010.

Ryland, S. "Paint Binder Classification by Infrared Spectrometry and Pyrolysis Gas Chromatography." SAFS workshop, Spring 1991.

9.0 Records

- Performance Check Log
- Maintenance Log
- Request for Instrumental Examination of Evidence

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
02/01/2013	2	Request for Instrumental Examination of Evidence was added as a record.
05/10/2013	3	5.1.3 – changed allowable variance
09/30/2013	4	6.0 – corrected limitations on Bench and Microscope Array Detector
10/18/2013	5	Added issuing authority to header
08/29/2014	6	Updated header to Physical Evidence Section – Trace Unit, issuing authority to Physical Evidence Section Forensic Scientist Manager. Updated all references in procedure from Trace Evidence Section to Trace Unit. Added wavenumber scan ranges to 5.3.3.4, 5.3.4.5, 5.3.5.6
12/11/2015	7	Added Methanol (or other organic solvent), Water, and O-rings to 4.0; added wording in 5.3.3.5, changed wording in 5.3.4.1; added "and Sample Selection" to 5.6