

FCS18 - SOP for Operating and Maintaining Spectrum Two Fourier Transform Infrared Spectroscopy (FT-IR) Instrument

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1. Scope

- 1.1. This document establishes the procedures for preventative maintenance and quality control that apply to instrumental analysis of the Fourier Transform Infrared Spectroscopy (FT-IR) instrument. The purpose of these maintenance and quality control (QC) procedures is to ensure that instruments are working properly and are free of contaminants before processing casework.

2. Background

- 2.1. To establish a procedure for regular instrument maintenance and QC to ensure quality and accuracy of reported casework results.

3. Safety

- 3.1. Reagent Toxicity: Personnel should refer to the appropriate SDS for solvents and reagents used during analysis for any specific safety requirements.
 - 3.1.1. For a complete review of required Health and Safety regulations of the PHL, see DOM13 DFS Health and Safety Manual
- 3.2. Protective Equipment: Personnel should wear personal protective equipment (PPE) including: lab coat, gloves, and safety goggles when carrying out standard operating procedures.

- 3.2.1. Wear vinyl or nitrile gloves when handling these chemicals to prevent absorption through the skin. If any chemicals are spilled onto gloves, discard gloves into hazardous waste.
- 3.3. Training: Formal training in use of instruments and software is necessary.
- 3.4. Personal Hygiene: Universal Precautions must be followed. Care should be taken when handling instrument, chemicals or any biological specimen. Routine use of gloves and proper hand washing should be practiced.
 - 3.4.1. Refer to DOM13 – Health and Safety Manual.
- 3.5. Disposal of Waste: Waste materials must be disposed of in compliance with laboratory, Federal, state, and local regulations. Solvents and reagents should always be disposed of in an appropriate container clearly marked for waste products and temporarily stored in a chemical fume hood.
 - 3.5.1. Consult DFS Safety Officer for proper procedures.

4. Materials Required

- 4.1. Reagent Grade Methanol (MeOH) or better
- 4.2. Binder/Folder for Standard results, or electronic equivalent
- 4.3. Quarterly/Weekly Maintenance Logbook, or electronic equivalent

5. Standards and Controls

- 5.1. Polystyrene Standard (Thermo Scientific, VALPRO ATR Standard Set 840166700, or equivalent)
- 5.2. Procaine Standard (VWR, TCP1961-25G, or equivalent)
- 5.3. Caffeine Standard (VWR, BT127310-100G, or equivalent)

6. Calibration

- 6.1. The FT-IR instrument is calibrated by the Vendor during the annual Preventative Maintenance (PM).

7. Procedures

- 7.1. General Maintenance
 - 7.1.1. Preventative Maintenance Schedule

Preventative maintenance will be performed by the supplier as per their recommended schedule (yearly).

7.1.2. Quality Control Procedures

- 7.1.2.1. A background spectrum will be obtained immediately prior to each sample.
- 7.1.2.2. A negative control (blank run) will be run prior to each sample using the same parameters as the sample analysis. The blank must be free of discernible peaks to be acceptable.
- 7.1.2.3. Sample run printouts shall be accompanied by the previous blank run's printout to ensure the system was free of contaminants prior to the run of interest.
- 7.1.2.4. Only one sample will be run on FT-IR at a time to ensure that the correct sample is run and to avoid contamination.
- 7.1.2.5. The Attenuated Total Reflectance (ATR) diamond and anvil will be wiped clean with methanol after each sample or standard run.

7.1.3. Standard Library Searches

- 7.1.3.1. Verified standards will be added to the FT-IR Standards Library as they are received and their spectra will be used as the basis for a successful match.
- 7.1.3.2. The analyst will confirm a library match through visual inspection of the sample spectrum against the standard spectrum.
- 7.1.3.3. To be conclusively identified, a sample spectrum must match a verified standard spectrum and must pass a visual inspection against the standard spectrum.
- 7.1.3.4. A copy of the matching search results and the blank run immediately before it shall be included with the printout of the sample.
- 7.1.3.5. All data will be saved in order to be reviewed later.

7.2. Weekly Maintenance Procedure

- 7.2.1. Weekly Maintenance must be carried out every week on the instrument used for casework and must be performed prior to any other casework. If only one instrument is used for casework, only the instrument being

used must undergo this weekly maintenance. Weekly maintenance must be performed only if the instrument is in use that week.

7.2.2. Monday is considered the start of a new week for weekly maintenance purposes.

7.2.2.1. Instrument Ready Check

7.2.2.1.1. Clean instrument using methanol prior to initiating the Ready check.

7.2.2.1.2. Select Run Selected from the top panel (top right).

7.2.2.1.3. Click Scan

7.2.2.1.4. If the Ready Checks passed, print Report and file in the FT-IR Logbook.

7.2.2.2. Procaine Search Procedure

7.2.2.2.1. Ensure the sample window is clear on the ATR.

7.2.2.2.2. Click Load Setup and select FCU 16 Scan method.

7.2.2.2.3. Click Background without lowering the anvil.

7.2.2.2.4. Next, click Sample Table (left panel) and under Sample ID name the standard with identifying information for the day's checks preceded by "Blank" and enter Analyst name in the Description field. (Do this before each analysis).

7.2.2.2.5. After entering sample information into the Sample Table, collect spectrum of your blank by clicking Scan with the anvil lowered. Adjust force gauge accordingly and click Scan again to collect sample.

7.2.2.2.6. Ensure the spectrum is saved and print a copy of the blank for the Weekly FT-IR Logbook.

7.2.2.2.7. Enter appropriate information into Sample Table, then place Procaine Hydrochloride standard on the ATR sample window.

7.2.2.2.8. Click Scanalyze (Scan and Search) with the anvil lowered. Adjust force gauge accordingly and click Scan again to collect sample.

7.2.2.2.9. Ensure the spectrum is saved.

7.2.2.2.10. If the procaine standard matches the standard in the search library, print the search results and file it and its blank in the Weekly FT-IR Logbook.

7.2.2.3. Another substance may be used for this procedure upon approval from the Unit Manager and will be documented on the maintenance logbook.

7.3. Control Chart Maintenance

7.3.1. As appropriate, the significant parameters for the identification of procaine (or other substance used for quality control) will be recorded in the laboratory control chart for FT-IR. Critical pieces of information include: (1) peak width and (2) position of characteristic peaks.

7.4. Quarterly Maintenance Procedure

7.4.1. Quarterly Maintenance shall be carried out every three months on the instrument used for casework and must be performed prior to any other casework. If only one instrument is used for casework, only the instrument being used must undergo this monthly maintenance. Quarterly maintenance must be performed only if the instrument is in use during the covered time period.

Quarterly maintenance includes an Instrument Ready Check, Instrument Verification, and Procaine Standard Check.

7.4.2. Instrument Verification (FT-IR)

7.4.2.1. Clean instrument analysis surfaces (ATR) using methanol prior to initiating Instrument Verification check.

7.4.2.2. Select Instrument Verification from the top panel (top right).

7.4.2.3. Click Scan

7.4.2.4. If the Instrument Verification passed, print Report and file in the FT-IR Logbook.

7.4.3. Instrument Ready Check

7.4.3.1. Ready Check (ATR)

7.4.3.1.1. Clean instrument analysis surfaces (ATR) using methanol prior to initiating the Ready check.

- 7.4.3.1.2. Select Run Selected from the top panel (top right).
- 7.4.3.1.3. Click Scan
- 7.4.3.1.4. If the Polystyrene check passes, print Report and file in the FT-IR Logbook.
- 7.4.3.1.5. If the Polystyrene check does not pass, clean the ATR and repeat. If this does not resolve the issue, contact the Lead Chemist and ensure the instrument is properly marked to prevent use until the system is found to be in conformance, e.g., with a sign marked "Instrument Not in Use," etc.

7.4.3.2. Caffeine or Procaine Hydrochloride Check (whichever is used)

- 7.4.3.2.1. Clean the ATR using methanol prior to initiating the Caffeine or Procaine Check.
- 7.4.3.2.2. Ensure the sample window is clear on the ATR.
- 7.4.3.2.3. Click Load Setup and select FCU 16 Scan method.
- 7.4.3.2.4. Click Background without lowering the anvil.
- 7.4.3.2.5. Next, click Sample Table (left panel) and under Sample ID name the standard with identifying information for the day's checks preceded by "Blank" and enter Analyst name in the Description field. (Do this before each analysis).
- 7.4.3.2.6. After entering sample information into the Sample Table, collect spectrum of your blank by clicking Scan with the anvil lowered. Adjust force gauge accordingly and click Scan again to collect sample.
- 7.4.3.2.7. Ensure the spectrum is saved and print a copy of the blank for the Weekly FT-IR Logbook.
- 7.4.3.2.8. Enter appropriate information into Sample Table, then place Procaine Hydrochloride standard on the ATR sample window.
- 7.4.3.2.9. Click Scanalyze (Scan and Compare) with the anvil lowered. Adjust force gauge accordingly and click Scan again to collect sample.
- 7.4.3.2.10. Ensure the spectrum is saved.

7.4.3.2.11. If the procaine standard matches the standard in the search library, print the search results and file it and its blank in the Weekly FT-IR Logbook.

7.4.3.2.12. If the procaine standard does not match, clean the ATR, take a new standard sample and repeat the check. If this does not resolve the issue, contact the Lead Chemist and ensure the instrument is properly marked to prevent use until the system is found to be in conformance, e.g., with a sign marked "Instrument Not in Use," etc.

7.5. Sample Run Procedure

7.5.1. Ensure the sample window is clear on the ATR.

7.5.2. Click Load Setup and select FCU 16 Scan method.

7.5.3. Click Background without lowering the anvil.

7.5.4. Next, click Sample Table (left panel) and under Sample ID name the blank with identifying case information preceded by "Blank" and enter Analyst name in the Description field. (Do this before each analysis).

7.5.5. After entering sample information into the Sample Table, collect spectrum of your blank by clicking Scan with the anvil lowered. Adjust force gauge accordingly and click Scan again to collect sample.

7.5.6. Ensure the spectrum is saved and print/retain it with casework.

7.5.7. Before analyzing your sample, enter appropriate information into the Sample Table, then place sample on the ATR sample window.

7.5.8. Click Scanalyze (Scan and Compare) with the anvil lowered. Adjust force gauge accordingly and click Scan again to collect sample.

7.5.9. Ensure the spectrum is saved.

7.5.10. Print the search results and retain it with casework.

7.5.11. Perform additional extraction and rerun the sample, as deemed necessary by the analyst (record in case notes).

7.6. Sample acceptance Criteria

Performance criteria are assessed using the following acceptance criteria and interpretation parameters:

- 7.6.1. Library searches can be used to provide useful information pertaining to the identity of a compound but should not be used as a replacement for analyst verification of the overall appearance and the presence and location of major spectral peaks when making an identification.
- 7.6.2. If used for identification, results from library searches must be printed and retained with sample spectra.
- 7.6.3. The infrared spectrum of the majority of controlled substances and other substances routinely identified is specific to a single compound and may be used for structural identification.

Table 3. Fourier Transform InfraRed Spectroscopy (FT-IR) Acceptance Criteria

	Acceptance Criteria	Detail
FT-IR PARAMETERS	Major peaks match relative height	As evaluated by the analyst; ideally within 20%.
	Position of major peaks	As evaluated by the analyst; ideally within 5cm ⁻¹ .
	Peak width match	As evaluated by the analyst; ideally within 20%.

8. Sampling

8.1. N/A

9. Calculations

9.1. N/A

10. Uncertainty of Measurement

10.1. N/A

11. Limitations

11.1. N/A

12. Documentation

12.1. FT-IR Maintenance Logbook

13. References

13.1. DFS Departmental Operations Manuals (current revisions).

13.2. Forensic Chemistry Unit SOPs (current revisions)

13.3. Controlled Substances Standard Operating Procedures; Comparative and Analytical Division, Houston Forensic Science Center (Document ID 2923, July 3, 2017)