FCS08 - SOP for Operating and Maintaining Nicolet iS50 Fourier Transform Infrared Spectroscopy (FT-IR) Instruments

Table of Contents

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). 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 routine and preventative 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 and authorization 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 – DFS 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 (See DOM13 – Health & Safety Manual).

4. Materials Required

4.1. Reagent Grade Methanol (MeOH) or better

4.2. Polystyrene and Caffeine or Procaine Standards

4.3. Binder/Folder for Standard results, or electronic equivalent

4.4. 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. Not applicable

7. Procedures

7.1. General Maintenance

7.1.1. Preventative Maintenance Schedule

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

7.1.2. Quality Control Procedures

7.1.2.1. A background spectrum shall be obtained immediately prior to each sample.

7.1.2.2. A negative control (blank run) shall 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 shall 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 ATR diamond and anvil shall be wiped clean with methanol after each sample or standard run.

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 shall 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. Procaine Search Procedure

- Ensure the sample window is clear on the Attenuated Total Reflectance (ATR).
- Ensure the 16 Scans experiment is selected.
- Click Collect Background without lowering the anvil.
- Click Collect Sample with the anvil lowered. Name standard with identifying information for the day's checks preceded by "Blank"
- Click "OK" and wait for sample collection.
- Save the spectrum as the title name.
- Place Procaine Hydrochloride standard on the ATR sample window.
- Click Collect Sample with the anvil lowered. Name standard with identifying information for the day's checks.
- Click "OK" and wait for sample collection.

- Save the spectrum as the title name.
- Click “Search” to search the standard library.
- If the procaine standard matches the standard in the search library, print the search results using the report template and file it and its blank in the Weekly FT-IR Logbook.

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

7.3. Control Chart Maintenance

7.3.1. As appropriate, the significant parameters appropriate for the identification of procaine (or other substance used for quality control) shall be recorded in the laboratory control chart for FT-IR. Critical pieces of information include peak width and 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 shall undergo this quarterly maintenance. Quarterly maintenance must be performed only if the instrument is in use during the covered time period.

Quarterly maintenance includes Suitability Checks and a Val Pro Qualification.

7.4.2. Suitability Check Procedure

7.4.2.1. Polystyrene Check

- Clean instrument using methanol prior to initiating the Polystyrene Check.
- Select Polystyrene Check from the Experiment pull-down menu.
- Select the System Status. Then select System Suitability. Then Select Run.
- Place the Polystyrene standard on the accessory and tighten the anvil.
- Select OK once added onto the accessory plate.
- Pop-up window will appear once completed. Select Report.
- If the Polystyrene check passed, print Report and file in the FT-IR Logbook.

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

- Clean the ATR using methanol prior to initiating the Caffeine or Procaine Check.
- Select Caffeine or Procaine Check from the Experiment pull-down menu.
- Select the System Status. Then select System Suitability. Then Select Run.
- Place the standard on the accessory and tighten the anvil.
- Select OK once added onto the accessory plate.
- Pop-up window will appear once completed. Select Report.
- If the check passed, print Report and file in the FT-IR Logbook.

7.4.3. Val Pro Qualification

7.4.3.1. Wipe instrument plate with methanol prior to initiating the Val Pro qualification.

7.4.3.2. Select Analyze on the top toolbar. Then Click on Val Pro Qualification

7.4.3.3. Select a Qualification test from the pull down menu.

Nicolet iS50 system with integrated ATR and built-in DTGS-EP

7.4.3.4. Select OK to collect BACKGROUND

7.4.3.5. When indicated, place the polystyrene standard on the accessory and tighten the anvil. Then select OK.

If qualification failed, clean instrument and polystyrene accessory and restart the Val Pro Qualification.

7.4.3.6. Print Report and file into the FT-IR Logbook.

7.5. Sample Run Procedure

7.5.1. Ensure the sample window is clear on the Attenuated Total Reflectance (ATR).

7.5.2. Ensure the 16 Scans experiment is selected.

7.5.3. Click Collect Background without lowering the anvil.

7.5.4. Click Collect Sample with the anvil lowered. Name standard with identifying information for the case (the lab and item number, generally) preceded by “Blank”

7.5.5. Click “OK” and wait for sample collection.

7.5.6. Save the spectrum and print using the report template.

7.5.7. Place a small amount of sample on the ATR diamond.

7.5.8. Click Collect Sample with the anvil lowered. Name standard with identifying information for the case (the lab and item number, generally).

7.5.9. Click “OK” and wait for sample collection.

7.5.10. Save the spectrum.

7.5.11. Click “Search” to search the standard library.

7.5.12. Print the search results using the report template and retain it with casework.

7.5.13. Perform additional extraction and rerun the sample, if desired.

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	As evaluated by the analyst;

	peaks	ideally within $5cm^{-1}$.
	Peak width match	As evaluated by the analyst; ideally within 20%.

7.7. Standard Library Searches

7.7.1. Verified standards shall be added to the FT-IR Standards Library as they are received and their spectra shall be used as the basis for a successful match.

7.7.2. The analyst shall confirm a library match through visual inspection of the sample spectrum against the standard spectrum.

7.7.3. To be conclusively identified, a sample spectrum must match a verified standard spectrum and must pass a visual inspection against the standard spectrum.

7.7.4. A copy of the matching search results and the blank run immediately before it shall be included with the printout of the sample.

7.7.5. All data shall be saved in order to be reviewed later.

8. Sampling

8.1. Not applicable

9. Calculations

9.1. Not applicable

10. Uncertainty of Measurement

10.1. Not applicable

11. Limitations

11.1. Not applicable

12. Documentation

12.1. Maintenance Logbooks and Control Charts

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)