20.0 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

20.1 Application
Fourier Transform Infrared Spectroscopy (FTIR) is a confirmatory test used in the analysis of legally significant substances. FTIR will generate a unique spectrum in the infrared region of 400-4000 wave numbers for liquid, solid, or vapor samples. The samples must be relatively pure to obtain the best results.

20.2 Equipment
The TBI FCU currently utilizes FTIR instruments manufactured by Thermo Fisher Scientific for the qualitative analysis of legally significant substances. The hardware systems are controlled by proprietary Nicolet OMNIC software packages. While versions of this software vary on each instrument, they all have the ability to search library databases for spectral comparisons.

Mortars, pestles, and hand presses are necessary for potassium bromide (KBr) pellet preparation. KBr must be FTIR quality and will be stored in a desiccator or low temperature oven to prevent moisture accumulation.

Liquid cells, salt plates, or an Attenuated Total Reflectance (ATR) sampling accessory may be used in lieu of pellet preparation.

20.3 Standards
Traceable polystyrene and/or glass standards provided by the manufacturer will be used for performance verification.

20.4 Method
Samples can be extracted to remove any potential extraneous compounds before any FTIR analysis method is performed.

20.4.1 KBr Pellets
A small amount of dried, anhydrous KBr is mixed with a very small aliquot of the sample. The sample is thoroughly ground until a fine powder is obtained. Samples that are dissolved in a solvent can be dried directly on KBr in a mortar before making a pellet. The mixture is then transferred to a die set. The opening or hole in the die must be filled completely before the sample is compressed. The sample should be pressed completely to ensure a flat pellet. The prepared sample is placed in the sample chamber and analyzed.

20.4.2 Attenuated Total Reflectance (ATR)
This technique requires very little sample preparation. The instrument window should be completely covered with the sample and properly compressed. Liquid samples can be analyzed by placing them directly on the ATR window. An accessory cap is available to cover the liquid to prevent or reduce evaporation for volatile compounds. Extremely corrosive liquids should not be placed on the ATR accessory.
20.4.3 Gas Cells

Highly volatile samples can be analyzed in a gas cell apparatus that contains clear KBr windows on each end and an opening for sample addition.

Please refer to the FTIR, iS10 FTIR with Smart iTR Accessory, and FTIR iS10 with Golden Gate Accessory Operating Instructions found in the Lab Documentation folder in Ensur.

20.5 Quality Assurance

Running daily primary standards for making spectral comparisons to unknown samples is not required since all primary standards and samples are run using the same instrument spectral parameters. The IR source can deteriorate over time and will eventually need replacement, but the spectra generated from a new source will not differ from the previously collected spectra.

The parameters affecting data acquisition will vary based on the sample preparation method. Therefore, the background, sample and standard spectra must be collected under the same parameters for the specific preparation method for a valid comparison. Consult the instrument logbook for these parameters.

**KBr Method background collection:**
When using the KBr method, a KBr pellet will be analyzed daily at a minimum on each instrument used to show that the KBr is free of contaminants. This spectrum will be retained in each case record for any FTIR using that daily KBr preparation.

The analyst will look for unusual peaks or indications of moisture in the KBr before using it for sample analysis.

**ATR Method background collection:**
The ATR window will be cleaned and examined before a new sample is placed on it. A background spectrum will be collected prior to each sample analysis.

20.6 Performance Verification and Acceptance Criteria

20.6.1 Performance Verification

Monthly instrument performance verification will be conducted using the OMNIC validation program. Other internal diagnostic software will be used to monitor the source and detector if available.

Wavelength placement, noise level, and energy (energy ratio or peak-to-peak) must be monitored. The TBI FCU may choose to monitor additional parameters for diagnostic purposes.

Results will be compared to manufacturer specifications or to previous results from that instrument. Tolerances for each piece of equipment will be specified in the instrument’s maintenance and verification notebook according to the manufacturer.

The diagnostic software will generate an alignment value for the instrument. This value should be higher than three (3) at a max gain of one (1). Any values less than three (3) may indicate that the source should be replaced.
The detector signal should be checked on a regular basis to ensure that the interferogram is producing an adequate signal. The system software will show the acceptable range for signal strength.

20.6.2 Acceptance Criteria

The spectrometer’s proprietary software will generate pass/fail results for the following tests:

- Signal to Noise (if applicable)
- Noise Performance
- Val-Pro or Val-Q qualification

If any of these tests produce failed results, the internal diagnostics will be repeated. The instrument will be removed from service if it fails the repeated diagnostic, and the unit supervisor will be notified.

Refer to Appendix F for maintenance requirements and intervals.

20.7 Criteria for Initial Evaluation

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

20.8 Interpretation

Direct peak-to-peak comparison between the primary reference standard and the sample must be made for confirmation. Spectral data must be compared to a primary reference standard that has been analyzed using the same sample preparation method (see section 20.4) and spectral parameters.

Spectral differences between samples and standards are usually due to the presence of additional compounds in the sample. Concentration differences between a sample and the standard should also be considered when determining a spectral match for KBr preparations.

A spectrum that may not have sufficient data for compound identification may be used to determine the salt form. Adequate salt form data must be present to justify the determination. Another confirmatory test must be performed in these instances.