

A. Purpose:

The primary use of the Fourier-Transform Infrared spectrophotometer (FTIR) in this procedure is for the identification of drugs and controlled substances. It is particularly useful in differentiating the different forms of cocaine (i.e., cocaine base and cocaine hydrochloride). The FTIR instrument can be used in the identification of a variety of solid, liquid, and gas samples (e.g., nitrous oxide). Analysis of drugs and other compounds by FTIR is considered a structurally elucidating confirmatory technique.

The FTIR is used in conjunction with the gas chromatograph/mass spectrometer (GC/MS) in order to identify cocaine in samples. The use of water solubility testing and FTIR analysis can be used to differentiate the different forms of cocaine.

Historically the distinction of cocaine free base (CFB) from cocaine salt form (CSF), or a mixture of the two forms, was important prior to 2005. This was because the criteria weights and associated penalties associated with the possession of these drugs were set at 0.5 grams for CFB and 28.35 grams (1 ounce) for CSF. Currently the criteria weights and associated penalty changes are set at 0.5 ounces for both forms of the drug, so differentiation of the two forms is not always necessary.

B. Safety:

The FTIR utilizes a laser which should not be directly viewed. As with any electrical device there is a chance of electrical shock if not handled properly. Do not perform maintenance on this instrument unless trained to do so. A laboratory coat will be worn while working with the FTIR instrument while analyzing casework samples.

C. Responsibility:

All analysts examining samples for the presence of drugs, controlled substances, or other chemicals.

D. Materials and Equipment:

Fourier-transform infrared (FTIR) spectrophotometer (Perkin-Elmer or equivalent)

Horizontal Attenuated Total Reflectance (ATR) Accessory (Pike, KRS-5, ZnSe, or equivalent)

Benzocaine (Reagent grade)

Polystyrene (Certified Reference Material (CRM) or equivalent)

E. Procedure:

1. Analysis: Using ATR:

The FTIR is used in conjunction with an HATR (aka. ATR) accessory. The ATR accessory allows for samples to be analyzed directly without sample preparation. Other FTIR accessories (e.g., microscope, gas cell) may be used for casework if they have been validated.

- a. Perform the QA/QC check on the FTIR daily – that is, on the day it is to be used for sample analysis. A polystyrene (PS) reference and benzocaine standard are analyzed and all printouts should be placed into the instrument logbook.
- b. The energy of the light source should be monitored to verify that there has not been a significant energy decrease. Any energy decrease may indicate the need to replace the source. If the source is left unchanged it could result in loss of sample sensitivity.
- c. If the QA/QC check has problems or the instrument operability fails, document the failure in the instrument log book and notify the section supervisor, or designee.
- d. Clean the ATR platform with methanol (or other appropriate solvent) to remove any materials.
- e. Collect a background spectrum before acquiring a sample spectrum. A background spectrum can be saved and used if multiple samples will be analyzed during a relatively short time period. If desired, a background spectrum can also be collected directly after sample collection instead of before sample acquisition.
- f. Thorough cleaning of the ATR accessory usually results in not carryover from sample to sample. If necessary, collect a blank spectrum in between casework samples. The blank ensures that the sampling platform is clean and void of materials.
- g. Where applicable, collect positive control samples after unknown samples have been analyzed.
- h. Save all data
- i. Perform data analysis (e.g., library searching, spectral comparisons, spectral subtractions)
- j. Interpretation of Data:

- i. Cocaine form determination:

Cocaine freebase: $\sim 2945\text{cm}^{-1}$, $\sim 1734\text{cm}^{-1}$ (doublet peak), $\sim 1706\text{cm}^{-1}$, $\sim 712\text{cm}^{-1}$.

Cocaine salt form: similar to CFB, however there will be an additional peak at $\sim 730\text{cm}^{-1}$.

Additionally for CSF, slight shifts can occur at the 2945 cm^{-1} , 1734 cm^{-1} , and 1706cm^{-1} ranges.

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- ii. Spectra should compare favorably (whether determining the form of cocaine or whether helping determine the identity of a drug).
- iii. While library searches are helpful, it is the responsibility of the analyst to make the final determination as to what drug or chemical was detected.
- iv. When comparing spectra it is best to overlay the spectra in absorbance mode as opposed to transmittance mode.

2. Analysis: Using Gas Cell

The gas cell is a chamber which allows a sample that is in a gaseous state to be sampled and analyzed on the FTIR (e.g., nitrous oxide)

- a. Remove the HATR and attach the gas cell accessory to the FTIR. The gas cell is a closed glass cylinder with 2 stop cocks. These are utilized to fill and purge the cell.
- b. Collect a background spectra using the glass cell itself.
- c. Analyze a blank using the gas cell and ensure the cell is contamination-free.
- d. Purge the gas cell with unknown gas by opening both stop cocks (so that it is an open system) for an adequate amount of time. Stop the purging process and close the stopcocks. Analyze the cell.
- e. Purge the cell with air and ensure that it is contamination-free.
- f. If there is only one sample, analyze the sample in duplicate. If there are 2 or more items and they all indicate that they are of the same type of gas, then analyze 2 random samples (e.g., two indistinguishable gas cylinders).
- g. Analyze a positive control.

F. Evaluation:

The FTIR is a very stable instrument and requires very little maintenance. The maintenance performed includes daily (as used), and as needed items.

- 1. Day of use: analyze a PS reference standard and a benzocaine standard
- 2. Compare the spectra to previously acquired spectra to confirm that there are no shifts or changes. Major differences indicate problems with the instrument.
- 3. If needed, compare spectra to library spectrum. Consult supervisor, or designee, if shifts in spectra, changes in spectra, or problems are observed.
- 4. Print necessary spectra and place in the instrument's maintenance logbook.

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5. Change desiccant as needed. This is performed by opening the cover of the instrument removing the cage which contains the desiccant and placing fresh desiccant bags in the cage. The cage is then returned to the instrument and the software can be updated to show the change.
6. Adjust HATR as needed. This should only be performed by appropriately trained personnel and is needed when there's a drop in the energy level or after replacing the HATR (Pike). The Pike alignment can be adjusted to maximize the energy levels of the light source.

G. References:

Operating the Perkin-Elmer Spectrum 100 FT-IR Spectrophotometer, operator's manual.

Spectrum 100 FT-IR User's Guide; Perkin-Elmer, 2008

Spectrum 100 FT-IR Help Topics; Instrument Validation.

Spectrum 100 FT-IR Help Topics; Performing an instrument validation

Clark's Isolation and Identification of Drugs in pharmaceuticals, body fluids, and post-mortem materials, The Pharmaceutical Society of Great Britain.

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3	Insert Effective Date	General formatting changes throughout document. Changed the primary use of the instrument from cocaine form differentiation to chemical, drug, and controlled substance identification.

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