

*Approved by Director: Dr. Guy Vallaro***A. PURPOSE:**

The primary use of the Fourier-Transform Infrared Spectrophotometer (FTIR) in the Controlled Substance Unit is for the identification of drugs. It is particularly useful in the differentiation of the free base form of cocaine from cocaine hydrochloride. This instrument can be used in the identification of a variety of solid, liquid, and gas samples (e.g., nitrous oxide). Analysis of unknown compounds by FTIR is considered a structurally elucidating confirmatory technique..

Most important to the CS Unit is the ability of the FTIR to distinguish cocaine salt form (CSF) from the cocaine free base form (CFB). The FTIR is used in conjunction with the gas chromatogram/mass spectrometer (GC/MS) in order to identify cocaine in samples. The combination of water solubility testing and FTIR is used to differentiate the forms of cocaine.

Historically the distinction of CFB from CSF (or a mixture of the two forms) was important prior to 2005 since the criteria weights and associated penalties associated with the possession of cocaine were set at 0.5g 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.

**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 caseworking samples on the FTIR.

**C. RESPONSIBILITY:**

All analysts assigned to the CS Unit are responsible for adhering to this SOP.

**D. DEFINITIONS:**

FTIR: Fourier-Transform Infrared Spectrophotometry

HATR: Horizontal Attenuated Total Reflectance

CSF: cocaine salt form (i.e., cocaine hydrochloride)

CFB: cocaine free base

GC/MS: gas chromatography/mass spectrometry

PS: polystyrene

QA/QC: Quality Assurance/Quality Control

**E. PROCEDURE:**

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**State of Connecticut Department of Emergency Services and Public Protection  
Division of Scientific Services**

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The CS Unit utilizes a Perkin-Elmer FTIR (or equivalent) with an HATR accessory. The ATR accessory allows for samples to be analyzed directly without sample preparation. Other FTIR accessories may be used for casework if they have been validated.

A QA/QC check is performed on the FTIR on a daily and on a monthly basis. A polystyrene (PS) reference standard is analyzed on a daily basis and compared to previous spectra for determination of instrument readiness. All printouts should be placed into the instrument logbook.

A PS reference standard, a Schott's glass standard, a blank, and a background are analyzed on a monthly basis. All printouts should be placed in the instrument log book. 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.

If any component of the validation fails, document the failure in the instrument log book and contact the section Supervisor, or designee, in order to follow-up on the issue. Prior to analyzing samples a background spectrum is acquired.

- i. Prior to each analysis the sampling platform is cleaned with methanol (or other appropriate solvent) to remove any materials.
- ii. Prior to each sample being analyzed a blank spectrum should be analyzed. The blank ensures that the sampling platform is clean and void of materials.

b. Interpretation of Data:

Cocaine form determination:

- (a) Cocaine freebase (CFB):  $\sim 2945\text{cm}^{-1}$ ,  $\sim 1734\text{cm}^{-1}$  (doublet peak),  $\sim 1706\text{cm}^{-1}$ ,  $\sim 712\text{cm}^{-1}$ .
- (b) Cocaine salt form (CSF): similar for CFB, however there will be an additional peak at  $\sim 730\text{cm}^{-1}$ . Additionally for CSF, slight shifts can occur at the  $2945\text{ cm}^{-1}$ ,  $1734\text{ cm}^{-1}$ , and  $1706\text{cm}^{-1}$  ranges.

Spectra should compare favorably to either cocaine base or cocaine hydrochloride in order to differentiate the cocaine forms of sample. While library searches are helpful, it is the responsibility of the analyst to make the final determination.

1.

Analysis: Using Gas Cell

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- c. 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)

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. Collect a background spectrum using the glass cell itself.

Analyze a blank using the gas cell and ensure the cell is contamination-free.

Purge the gas cell with unknown gas by opening both stop cocks (so that it is an open system) and analyze the cell.

- (a) Purge the cell with air and ensure that it is contamination-free.
- (b) The sample should be run in duplicate if there is only one item or 2 individual items run if there are 2 or more identical items (i.e. 2 or more like gas cylinders)
- (c) Run a nitrous oxide standard as a positive control.

#### **F. INSTRUMENT MAINTENANCE:**

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

- a. Day of use: analyze a PS reference standard
- b. Monthly a check is performed as described under the Controls section. Verify that the instrument is working consistently from month to month.
  - i. Polystyrene and Schott NG11 Glass Reference Standards; these two standards are used to show the consistent working condition of the instrument.
    - (a) Run the standards per the instruments operator's manual.
    - (b) Interpretation: Compare the spectra of the reference standard to previously run standards to confirm that there are no shifts in the spectra. Major differences indicate problems with the instrument. Consult with your supervisor, or designee, and they will determine whether to contact the instrument manufacturer.
    - (c) Print necessary spectra and place in the instrument's maintenance logbook.

Interpretation: Compare the spectra to previously analyzed standards. If there are major shifts in the spectra then consult your supervisor, or designee. The supervisor will determine if the instrument manufacturer

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- c. Bi-annually: the desiccant is changed. 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 is updated to show the change.
- d. As Needed: HATR adjustment – This 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.

While monitoring the energy level, adjust the two screws on the HATR to obtain the maximum energy level. Record this level in the maintenance log.

G. REFERENCES;

- a. Operating the Perkin-Elmer Spectrum 1000 FT-IR Spectrophotometer, operator's manual.
- b. Spectrum BX FT-IR User's Guide; Perkin-Elmer, 1998
- c. Spectrum BX FT-IR Help Topics; Instrument Validation.
- d. Spectrum BX FT-IR Help Topics; Performing an instrument validation
- e. Clark's Isolation and Identification of Drugs in pharmaceuticals, body fluids, and post-mortem materials, The Pharmaceutical Society of Great Britain.