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 and liquid.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 analytes of interest.

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 (ThermoNicolet or equivalent)

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

Benzocaine (Reagent grade)

Polystyrene (ThermoNicolet or equivalent)

E. Procedure:

1. Analysis: Using ATR:

   The FTIR used in conjunction with a 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 source voltage 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.

   f. Where applicable, collect positive control samples after unknown samples have been analyzed.

   g. Save all data (saving interferogram data is recommended, but optional)

   h. Perform data analysis (e.g., library searching, spectral comparisons, spectral subtractions)

   i. Interpretation of Data:

      i. Cocaine form determination:

      Cocaine freebase (CFB): ~2945cm^{-1}, ~1734cm^{-1} (doublet peak), ~1706cm^{-1} ,~712cm^{-1}.

      Cocaine salt form (CSF): similar to CFB, however there will be an additional peak at ~730cm^{-1}. Additionally for CSF, slight shifts can occur at the 2945cm^{-1}, 1734cm^{-1}, and 1706cm^{-1} ranges.

      ii. Spectra should compare favorably to an in-house standard or reference library spectra for the identified analyte

      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 stopcocks (these are utilized to fill and purge the cell).
   b. Collect a background spectra using the glass cell without sample.
   c. Purge the cell with air (or $N_{2(g)}$).
   d. Analyze a blank using the gas cell and ensure the cell is contamination-free.
   e. 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.
   f. Perform an analysis.
   g. Purge the cell with air (or $N_{2(g)}$) and ensure that it is contamination-free.
   h. Use a sampling plan similar to other types of drug analyses (i.e., if only one sample then analyze 2x ; if all evidence is indistinguishable, sample only two (2) pieces of evidence and stop if results are same).
   i. Analyze positive control(s) in a similar manner.

F. Maintenance:

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 standard and a benzocaine standard
2. Compare the spectra to previously acquired spectra to confirm that there were no shifts or changes. Major differences indicate problems with the instrument or the accessory.
3. If needed, compare spectra to library spectra. 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.
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. If applicable, this can be done as part of the service contract by an Perkin-Elmer Engineer.

6. Adjust HATR as needed. This should only be performed by appropriately trained personnel and as needed when there's a drop in the energy level or after replacing the HATR. The HATR alignment can be adjusted to maximize the energy levels of the light source.

G. References:

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