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:

The FTIR is 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.

1. QA/QC
    a. Perform the QA/QC check on the FTIR daily – that is, on the day it is to be used for sample analysis.
    b. Perform the following tests using the OMNIC Software:
        i. System Suitability
        ii. ValPro Qualification: Internal-PHEUR

iii. ValPro Qualification: ATR-PHEUR

c. Analyze a polystyrene (PS) reference and benzocaine standard.
   i. 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.
   ii. If benzocaine is unavailable an alternative standard may be used. Consult an appropriate Unit Lead (or higher).
d. Monitor the Source Voltage to verify that there has not been a significant energy decrease.
   i. 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.
e. Monitor the Interferogram Voltage (peak-to-peak sum)
f. Document all QA/QC checks in the maintenance log and save printouts in the logbook or electronically.
g. If the QA/QC check has problems or the instrument operability fails, document the failure on the maintenance log and notify the appropriate Unit Lead (or higher).

2. Sample Analysis
   a. Clean the ATR platform with methanol (or other appropriate solvent) to remove any materials.
   b. Collect a background spectrum before acquiring the sample spectrum.
   c. Collect the sample spectrum.
      i. Do not apply pressure directly to the crystal without a sample in place.
      ii. If the sample spectrum reflects poor absorbance, it is recommended to rerun the sample. This could be an indication of carryover or not enough sample being placed on the crystal.
   d. Save all data (saving interferogram data is recommended, but optional).
   e. Perform data analysis (e.g., library searching, spectral comparisons, spectral subtractions)
   f. At the completion of the sample set, collect a sodium chloride blank.
      i. The spectrum for the blank should not contain any significant extraneous peaks.

3. Interpretation of Data
   a. The following peak positions should be present for Cocaine form determination and the spectrum should be marked accordingly:

Compound	**Expected Peak Position (cm^{-1})**
Cocaine Free Base	1275, 1700, 1106, 1728, 710
Cocaine HCl	1712, 1730, 1276, 1230 (side peak), 732

Note: Peaks are listed in order of magnitude of absorbance; may vary from sample to sample.

b. Spectra should compare favorably to a reference standard.
   i. As long as there have been no major changes to the instrument, reference standards only need to be analyzed on a weekly basis. It is recommended that this take place before samples are analyzed.
   ii. When comparing spectra, it is best to overlay the spectra in absorbance mode as opposed to transmittance mode.

F. Maintenance:

The FTIR is a very stable instrument and requires very little maintenance.

1. Change desiccant as needed.
   a. 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.
   b. The cage is then returned to the instrument and the software can be updated to show the change.
2. Alignment

   To be performed if there is an issue with wavenumber accuracy.

   a. Remove the accessory from the sample compartment.
   b. In OMNIC, click on the "Collect" menu and the "Experiment Setup" command.
   c. Select the "Bench" tab in the "Experiment Setup" window.
   d. In the table on the right, set the following parameters:

Gain	1.0
Optical Velocity	0.4747
Aperture	100

   e. Select the "Diagnostic" tab.
   f. Click the "Align" button below the white box.
   g. Return the accessory to the sample compartment.
   h. The alignment is complete, rerun the instrument ValPro test and the Smart iTX-PHEUR test.

3. Reseat the Pressure Tower

   To be performed when the pressure tower will not lower the tip when the knob is turned.

   a. Remove the accessory from the sample compartment.

b. Turn the accessory on its side or upside down, being careful so that the crystal plate does not fall off.
c. On the underside of the accessory there is a hole that is directly below the pressure tower, and in the hole, the user will notice a screw. Using an appropriate sized flat blade screwdriver turn the screw counterclockwise while at the same time turning the pressure tower knob clockwise. This should reset the pressure tower and allow the tip to be lowered.
d. Return the accessory to the sample compartment.

G. References:

Recommended methods for the Identification and Analysis of Cocaine in Seized Materials. 2012. United Nations Office on Drugs and Crime. (page 35)

ThermoFisher Scientific Maintenance Guide (located in “Quality” folder within Qualtrax)