



Abbreviated Standard Operating Protocol for Scanning Analysis of Discarded Drug Packaging Detritus from the Massachusetts Drug Supply Data Stream (MADDS)



# Acknowledgements and Further Information

This Standard Operating Protocol for Scanning Analysis of Discarded Drug Packaging

Detritus was developed by the Massachusetts Drug Supply Data Stream (MADDS). Parts of this

protocol were adapted from drug checking resources made available by the British Columbia

Centre on Substance Use (BCCSU) and images from the OPUS software for scanning and

analysis (www.bccsu.ca/wp-content/uploads/2019/03/BCCSU-Technician-Manual-March-

<u>2019.pdf</u>). For more information, please visit our website at: <u>streetcheck.org</u>. To get in touch

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## 1.0 Data Entry and Management

Data entry is managed through the StreetCheck app where users are able to record details and view results related to collected drug samples. The link to the MA tenant StreetCheck Portal is: <u>https://ma.streetcheck.org/Account/SignIn</u>

\*For other state tenants, please delete "ma" and add the abbreviation for your state into the link.

For direction on how to use StreetCheck to enter/view a sample, please refer to the Standard Operating Protocol for StreetCheck which can be found at the following link: <u>https://www.info.streetcheck.org/community-drug</u>



## 2.0 Equipment and Supplies Required

Below is the supply list for the scanning and analysis of drug packaging.

#### 2.1 Supplies for Scanning

- Bruker (HP) laptop and charger
- FTIR spectrometer and case
- Disposable nitrile gloves
- Stainless steel spatula
- Stainless steel scissors
- Disposable plastic medicine cups
- Kimwipes
- Aluminum foil
- Isopropyl alcohol (in wash bottle)
- Tap water (in a wash bottle)
- Deterra bags
- Naloxone kit

- BTNX Fentanyl Test Strips
- BTNX Benzodiazepine Test Strips
- BTNX Xylazine Test Strip
- pH strips
- Stamps
- Small plastic bags (2x2)
- Glassine bags
- Bubble wrap
- Small disposable scoops
- Sharpie
- Magnifying glass
- Mortar and pestle



Figure 1: Toolbox with necessary equipment



Figure 2: FTIR in carrying case

#### Supplies to Send Samples for Additional Testing:

- Envelopes for confirmatory lab
  - Mylar bags (black: DrugsData, blue: CFSRE, red: RIH)

- Small Plastic Bag with Additional Label (Lab Code)
- Glassine Bags (CFSRE)
- Sample Submission Paper Form (DrugsData only)
- Envelope for CFSRE
  - Blue Mylar bag with Additional Label (Lab Code)
  - o Glassine Bag

## 2.1.1 DrugsData Analytical Sample Submission Form

- Leave the first entry box at the top of the form blank as DrugsData will fill this in.
- DrugsData Sample Code will already be on the sheet.
- <u>Your Sample ID</u>: Record the sample ID found on the sample baggie and/or in StreetCheck or a case file.
- <u>Date Acquired</u>: Record the date the sample was obtained or initially tested. (Format should be Jan 01 2023).
- <u>Sample Name</u>: Record what the sample is known as / the street name (e.g., dope, crack, ketamine, etc.).
- <u>Source Location</u>: Report the city and state the sample was collected. Write out the full name of the city, abbreviate the state, and separate with a comma.
- <u>Appearance</u>: Provide a brief but precise description of the sample including color and form of the sample (e.g., green pill (part), white powder (chunky), brown residue, etc.).
- <u>Sold As</u>: If known, report what the sample was said to be at time of purchase (e.g. cocaine, heroin, meth, etc.).
- Expected to Be: If known, report what the user/participant expects is actually in the sample.
- The <u>Experience Notes</u>: If known, report any details regarding the user/participant's experience while under the influence of the substance. If the sample is associated with a fatal or non-fatal overdose, record "adverse health event occurred." Whatever you write in this box will be posted publicly. Use discretion.
- The <u>Observations</u>: section is the place to note any initial testing results.
  - FTIR: Note your analysis of the spectra library reference matches that you believe to be in the sample. If you are not doing analysis in advance of mailing, leave blank.
  - HPVX/MX908: Leave blank.
  - BTNX Fentanyl Strip: Record the results of the BTNX Fentanyl Test Strip at 5mL (positive/negative).
  - BTNX Benzo Strip: Record the results of the BTNX Benzodiazepine Test Strip at 5mL (positive/negative).
  - Other: This can be left blank. However, you can record any outstanding observations regarding the sample.

DrugsData Sample	Code:				
Payment Code:	202005-	BST-QWE (40 analyses)			
<ul> <li>ONE SAMPLE per I</li> <li>Put 1–50 mg in sma</li> <li>Then put clear sam</li> <li>Can be filled out dig</li> <li>Responses are tran</li> </ul>	BLACK MYLAR BAG & ( all clear sample baggie w ple baggie inside protec gitally as PDF, but Samp sscribed, writing will not	DNE FORM for EACH SAMPLE. with Drugsdata Sample Code matching above. tive black mylar ziplock (NO LIQUIDS'). le and Payment Codes must be entered above. appear online. • Avoid Cross-contamination.			
Your Sample ID:					
Date Acquired:		(format: Sep 04 2019)			
Sample Name:	8	(What it's known as: dope, crack, cocaine)			
Source Location:	×	(city and state, online)			
Appearance:		(off-white powder, brown tar, clear crystals)			
Sold As:		(heroin, dope, cocaine, ketamine)			
Expected to Be:	¢	(What user suspects it might contain: cuts, substitutes			
Experience Notes:					
Observations:	FTIR				
	HPMS / MX908				
	BTNX - Fentanyl Strip				
	BTNX - Benzo Strip				
	Other				

DrugsData Analytical Sample Submission Form

Figure 3. DrugsData Analytical Sample Submission Form

#### 2.1.2 Sending a Sample to DrugsData Lab

- 1. Using your dental tool, place one scoop / ~1mg of sample into the small, clear baggie labeled with the DrugsData lab code.
- 2. Place the small clear baggie into the black mylar bag and seal it.
- Place the black mylar bag into the big white envelope addressed to D.D.L. in Sacramento, California. Seal and put all white envelopes in the mail with proper postage, or package them all together in a box to be mailed.

#### 2.1.3 Sending a Sample to CFSRE Lab

- Using your dental tool, place one scoop / ~1mg of sample into the small, clear plastic baggie. Write the sample ID on the bag as well in Sharpie.
- If you have a substantial amount of sample and are interested in receiving quantification results from CFSRE, add ~5-10 mg (two scoops) of sample to the glassine bag, or send along the original glassine bag.
- 3. Place the small clear baggie or the glassine bag into the blue mylar bag labeled with the laboratory code and seal it.

4. Place the blue mylar bag into the big white envelope addressed to Alex at CFSRE in Willow Grove, Pennsylvania. Seal and put all white envelopes in the mail with proper postage, or package them all together in a box to be mailed.

### 2.1.4 Sending a Sample to Rhode Island Hospital (RIH) Lab

- Using your dental tool, place one scoop / ~1mg of sample into the small, clear baggie labeled with the RIH lab code. Write the sample ID on the baggie as well in Sharpie.
- 2. Place the small RIH clear baggie into the red mylar bag and seal it.
- 3. Place the red mylar bag into the big white envelope addressed to Adina at RIH Toxicology in Providence, Rhode Island. Seal and put all white envelopes in the mail with proper postage, or package them all together in a box to be mailed.



Figure 4. Lab Color-Coordinated Mylar Bags.

#### 2.2 How to set up for analysis:

#### **Supplies for FTIR Analysis**

- Bruker (HP) Laptop and charger
- Secondary laptop and charger and/or iPad/tablet and charger

#### 2.2.1 Personal Protective Equipment (PPE)

All study staff will take appropriate precautions when handling materials and sample specimens. The PPE listed below serve as preventative measures when handling unknown substances to reduce the risk of exposure. They also serve to prevent unintentional cross-contamination of samples.

- The following PPE must be worn at all times when handling materials for sample scanning:
  - Disposable nitrile gloves
  - Closed toed shoes
  - High quality face mask

• When conducting sample measurement and scanning, operational technicians must wear nitrile gloves and hands should be washed before and after administering services. Gloves should be changed on a regular basis (every 30 to 60 minutes), whenever they come into contact with a substance, or if they tear. Always remove gloves and wash hands before touching your face, touching door knobs, using the restroom, eating, drinking, or leaving the sample scanning area.

#### 2.2.2 Location Requirements

- A lab bench (or table) of approximately 4' by 2'
- Access to a sink with running water
- Trash Can
- Plugs (electricity source)



## 3.0 Fourier Transform Infrared Spectroscopy (FTIR)

The below protocol is modified from the BCCSU Drug Checking Program Operational Technician Manual for use on the Bruker ALPHA-II spectrometer equipped with the platinum attenuated total reflectance (ATR) module (which has a diamond ATR crystal), operated with OPUS software.

#### 3.1 Spectrometer Setup:

- 1. Remove the spectrometer module from the box and place it on the table. Use care to not touch any of the interior components of the device.
- 2. Remove the sampling module from the box. Using the side rails on the spectrometer module, slide the sampling module into place. Open the latch by firmly pressing the silver rectangular button located on top of the spectrometer module.
- 3. When the sampling module is in the proper position, you will hear a click as the rectangular button becomes level with the spectrometer module (Figure 4, left). If the rectangular button is not level with the spectrometer module (i.e., the rectangular button does not fully raise), press firmly down on the rectangular button and try again (Figure 5, right).



Figure 5. Bruker Alpha Assembly (Incorrect)



Figure 6. Bruker Alpha Assembly(Correct)

4. Insert the power supply cable to the power port on the back of the spectrometer module (Figure 6). Be sure to connect the device to a reliable power source. **Any disruption in the power supply will cause the spectrometer to restart.** 



Figure 7. Bruker Power Supply

- 5. Once the FTIR is plugged in, a warm-up phase, lasting approximately 10 minutes, will begin. If the spectrometer is unplugged in the middle of testing a sample, the spectrometer will automatically restart and the warm-up phase will repeat.
- 6. Connect the laptop to the spectrometer by inserting the network cable into the back of the spectrometer module, and the Ethernet port on the laptop. Open the OPUS program (DrugID Wizard) on the laptop desktop by clicking this icon:



Figure 8. OPUS DrugID Icon

 A small window will pop up. The default username and password are the same for all spectrometers and are set up when the spectrometer is delivered and installed by Bruker.

User ID.	Default	~
	Default	ADMINISTRATOR
Password		
Assigned workspaces	Drug_Identification.ows	
Assigned workspaces:	Drug_Identification.ows	

Figure 9. OPUS Login

8. Once logged into the system, a countdown will be visible in the task bar (the bottommost border of the OPUS window). When a task is running, the bar will be green.

OPUS - Operator Default (Administrator) - [Doplay - Drug.]dentification.ovs]		-	0 3
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ante ante atos atos atos atos atos atos atos atos	800	600	400
🐑 🔛 Display - Doug Mentification.com			×
Background Waiting for instrument ready 373.7s - Source			
Manuar Mathematical Control Control Control of Mathematical Control Co		NUMBER OF T	

Figure 10. Opus Measurement Window

- After the warm-up phase is complete, the spectrometer will run a 'performance test.' Do not touch the diamond ATR crystal during the performance test. If the diamond ATR crystal is touched, it could become damaged or disrupt the calibration.
- 10. Next, under validation, select RUN OVP Test, select PQ test, click run. This takes an additional ten minutes. A PQ test is required each time the FTIR is moved or set up in a new location. OQ test is not required.
- 11. When the warm up and validation is complete, the status light in the bottom right corner of the OPUS window will turn green, as will the light on the top of the spectrometer module. The FTIR is now ready to begin. **Do not move forward with scanning if the status light is not green.**

mp are		
	CDI C	

Figure 11. Opus Task Status

#### 3.2 Measuring a Sample

- 1. Before each sample is tested, ensure that the diamond ATR crystal is clean, in the raised position, and click the large "Measure Background" button on the left-hand side of the window.
  - a. This scan will take approximately 30 seconds. Once this procedure is complete, the large "Measure Background" button (Figure 9) on the left will change to "Measure Sample" (Figure 10).



Figure 12. OPUS Measure Background

Measure Sample

Figure 13. OPUS Measure Sample

- 2. Click "Measure Sample". You will be prompted to enter information about the sample you are running. You will notice on this screen the fields will appear auto-populated with information about the last measured sample. Delete that in order to put in new sample information.
- 3. Enter the sample name (sample ID), sample form (e.g. white powder) and operator initials and click OK.

Please insert sample information				
Sample name	105281817JD			
Sample form	heroin			
Operator	JD			
C.\Users\Public\Documents\Bruke	\DPUS_7.8.44\DrugID	ок		

Figure 14. OPUS Sample Information

- 4. The new window displays a live reading of the sample (i.e. signal viewer). You are now ready to prep your sample.
- 5. Using a sterilized metal spatula (dental tool), scrape the sample from the packaging.
  - a. It may be easiest to collect the substance into one corner of the bag or to collect it near the opening of the bag.



Figure 15. Sample Prep from Baggie

6. Add the sample to the crystal so that the square is fully covered, and add a pre-prepared foil hat (Figure 15) on top of the substance before lowering the anvil. Make sure you hear the anvil click into place.



Figure 16. Foil Hat

7. To maximize the signal, the test substance and foil hat should be directly on the square crystal and the anvil should have the red dot directly in the center of the alignment viewer (see below). Adjust placement of the anvil, and/or sample accordingly.



*Figure 17. Proper Alignment of Bruker Anvil* 

- 8. Take note of the peak heights by looking at the scale on the left. Try to get the signal as high as possible, ideally over 0.300. If it is under 0.200, readjust the sample on the sensor and try again.
  - a. If the signal viewer (see below) is not turned on, go to Measure → Setup Measurement Parameters → Display and check the box next to "Display single scans before measurement".



Figure 18. OPUS Measurement Parameters

When you have maximized the signal for the sample, click "Start Measurement". The task bar will turn green — this shows the progress of the scan. The reading will take about 30 seconds. When the reading is finished, OPUS will automatically show the spectrum and search the libraries for matching samples. (Do not touch the anvil/sample AT ALL until the sample is completely measured).

- 9. Once the sample is scanned, remove the foil hat and set aside for fentanyl/benzo/xylazine test strip analysis by adding the sample to a plastic cup for the test strip analysis. The remaining sample will be carefully transferred into a 2" x 2" plastic glassine bag for lab testing and any remaining sample will be disposed of in a Deterra bag.
- 10. Following sample scanning, all Bruker surfaces and tools shall be cleaned with all least 3 alcohol pads and dried with a Kim wipe. (Can also use isopropanol to clean but do not spray directly on Bruker, spray on Kim wipe and then wipe the Bruker)
- 11. Any materials exposed to the substances during this process (including gloves, alcohol pads, Kim wipes, etc.) shall be disposed of per protocol in hazardous waste bins or the Deterra drug disposal system.

### Abbreviated FTIR Steps:



- 1. Attach Power and Ethernet Cables  $(\sim 2 \text{ min})$
- 2. Allow Instrument to Warm Up  $(\sim 10/15 \text{ min})$
- 3. Clean Instrument w/ Isopropanol sprayed onto Kimwipe not FTIR (~1 min)
- 4. OVP (Opus Validation Program) QC Test (~5 min)
- 5. Scan Background (~1 min)
- 6. Load Sample, Place Tinfoil Hat, Rotate Anvil Over, Bring Anvil Down, Ensure Red Circle is in the Center, Press Scan Sample (~5 min)
- 7. Unload Scan and Press Next Sample (~5 min)
- 8. Move Anvil Over, Clean Instrument w/ Isopropanol as above (~1 min)
- 9. Scan Background (~1 min)
- 10. Repeat with Next Sample! (~10 min)

## 3.3 Analyzing a Sample

A spectrum search is the function in OPUS that detects the components of an unknown spectrum. OPUS will match the unknown spectrum you have just generated to reference spectra in selected libraries. Libraries are made up of hundreds of spectra of pharmaceuticals, street drugs, common adulterants, and other substances.

 After a spectrum is generated, OPUS will usually run a Spectrum Search automatically. If this does not occur, this can be done manually: click "Evaluate" > "Spectrum Search". You can also use the shortcut button in the toolbox on the right-hand side. The icon is a magnifying glass.



#### Figure 19. Spectrum Search Icon

- 2. Once this search is performed, a window will open with three tabs:
  - Spectrum Search: In this tab, you can select which sections of the spectrum to use for the search. To start, ensure that "Use file limits" is selected. This setting uses the entire measured spectrum.
  - Search Parameters: In this tab, you can select the search algorithm to use. When performing basic analysis, use Spectrum Correlation, Vector Normalization, 1st Derivative.
  - Select Libraries: In this tab, you can select the reference libraries for the spectrum search. If it is your first time running a drug sample, ensure that the SWGDRUG, TICTAC ATR-FTIR, and BCCSU libraries are selected at a minimum.

Spectrum Search		
Spectrum Search	Search Parameters	Select Ubraries

#### Figure 20. Spectrum Search Input Window (Parameters and Library)

- 3. After selecting the relevant options, select "Search Library".
  - The spectral matches will be listed by descending Hit Quality (1000 represents a perfect fit). "Hit Quality" refers to OPUS' prediction of what the substance is based on in comparison to reference spectra found within selected libraries.
  - Samples that contain more than one component are referred to as mixtures. Mixtures must be analyzed by subtracting the individual spectral components until there are no residual peaks present (i.e., it has returned to the baseline measurement from the background scan).
  - To subtract components, hover over the spectral match, right-click and select "Auto-Subtract" > "New Search". This subtracts the selected reference spectrum from the measured spectrum, thereby revealing the peaks in the measured spectrum not accounted for by the first reference hit. Note that it may be necessary to perform more than one subtraction (with different reference spectra) to best identify all of the components in a mixture.





- If there is a section of the subtracted spectrum with a large dip or other subtraction issue, you can use the "Spectrum Search" tab of the "Spectrum Search" function window to restrict the search area, excluding that region. Right click to add or remove new regions and drag the width to fit accordingly.
- Note: If there are peaks remaining that you think could be more than noise (i.e. they could be a signal from an unknown compound), yet do not match any existing hits, try using different libraries. The PHARMA libraries are very large and therefore slow to run but do contain substances that are not accounted for in the smaller libraries.
- Control which window you are viewing using the tabs at the bottom. Use the 'x' button on the right side to close the present window.

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- 4. Mixture Analysis Steps and Benefits
  - Mixture analysis is a great tool to use after completing spectral subtraction.
     Mixture analysis is helpful to see if it corroborates the substances identified via other techniques.
  - Select "Mixture Analysis" under the "Search Parameters" tab when performing a Spectrum Search and adjust the number of expected components in the sample (minimum 2, maximum 10).
    - Do not perform Mixture Analysis if only one substance was detected through the initial spectral search.

- OPUS will generate a composite spectrum dependent on the number of expected components. OPUS creates the composite spectrum to be as similar as possible to the sample spectrum by combining library references. The composite spectrum is created by weighing library references to best match the sample spectrum. The weight for each sample indicates the impact on the composite spectrum and is provided as a percentage make-up.
- If the composite spectrum is not a good match to the sample spectrum (e.g. the composite spectrum is missing multiple peaks that are present in the sample spectrum) re-run the mixture analysis with more expected substances.
- When Mixture Analysis is performed, OPUS also provides a residual spectrum. The residual spectrum is the equivalent to an auto-subtraction. The sample spectrum "subtracts" the composite spectrum and the difference is the residual spectrum.
- The residual spectrum is a useful tool to identify any peaks that remain unaccounted for by the composite spectrum.
- 5. Mixture Analysis Limitations
  - Please note that there are many limitations of Mixture Analysis and the results should be considered in consideration with other techniques.
  - Mixture analysis will not work well on all samples. If you have attempted the mixture analysis with multiple expected components and no composite spectra match well to the sample spectrum, do not record the mixture analysis results.
  - Mixture analysis results should only be recorded when the composite spectrum appears to be a good match and provides reasonable results.
  - Frequently, mixture analysis will provide substances that were not identified via the previous methods. OPUS selects the library references that best match the composite spectrum and will frequently provide substances that we do not see in the drug supply.
    - Mixture analysis will not provide an accurate Fentanyl HCl value if it was not detected through spectral subtraction (BCCSU, 2021).
  - The weights/ratios provided by mixture analysis are not representative of quantification of substances in the sample. Previous studies have shown that the quantification/weight provided by mixture analysis can vary GREATLY. Weights provided from mixture analysis can be recorded but are not precise.

- In samples where fentanyl was confirmed with laboratory testing to be below 20% concentration, mixture analysis on FTIR failed to detect fentanyl 34% of the time (Tobias, 2021, as cited in BCCSU Fentanyl Quantification and Messaging SOP, 2021).
- Fentanyl analogues are not interchangeable when attempting to quantify using FT-IR Mixture Analysis. Do not add the values returned for multiple analogues to give an overall percentage of fentanyl present.

6. When the interpretation is complete, unload the spectrum from OPUS by clicking "File" > "Unload All Files", or use the shortcut on the toolbox along the top.



Figure 23. Unloading a File

• You can view which files are loaded into OPUS by viewing the OPUS Browser. Hover your cursor over OPUS Browser in the top left of the OPUS window, below the taskbar.



#### Figure 24. How to View Uploaded Files

 When you perform subtractions and spectrum searches, the new subtracted spectrum will be loaded into the OPUS browser. To perform a spectrum search on a specific spectrum, ensure the correct one is highlighted in the OPUS Browser (i.e. the AB block). This is important if you have multiple spectra open in the same window.

7. After unloading the spectra from OPUS, the software is ready to measure a new sample. Click the large button on the left-hand side of the screen that reads "Next Sample".

## 3.4 Bruker Maintenance and Troubleshooting

As with any machinery, the Bruker will require some regular maintenance and upkeep to avoid major malfunctions.

To keep the Bruker running at its best, the Operational Technician on site will perform the Performance Qualification (PQ) test weekly or when the spectrometer is moved to a new site. The PQ test evaluates the performance and function of the spectrometer and should serve as a regular check-up on the machine.

The Operational Qualification (OQ) test is used to check the current state of the instrument performance. OQ tests are intended to be performed after major repairs, exchange of optical components that influence instrument performance, and annual maintenance. The Operational Technician will complete this test when performing these tasks.

In the case of machine error, the operational technician shall follow the process below:

- 1. Clean and restart the machine to see if error recurs.
- 2. If error persists, the Operational Technician on site shall contact the Research Coordinator, On-site Manager and Principal Investigator to discuss and attempt to remedy the problem.
- If the error is not able to be remedied through the study team, the Operational Technician on site will contact Bruker Customer Service at 978-439-9899 or info.bopt.us@bruker.com. The reference number for our BRANDEIS Bruker is 1 263 1415.

#### 3.5 Data Capture

All FTIR spectra will be saved in their original form on the password protected study laptop, and will only be accessible to study staff through the password protected OPUS software.

If a file is accidentally saved in a modified form, the operational technician on site will follow the below process (adapted from BCCSU) to recover the original file.

1. With the file loaded into OPUS, open the "Spectrum Calculator" from the "Manipulate" menu in the toolbar.

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7	8	9	•			
4	5	6	-	٤		>
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Figure 25. Spectrum Calculator

- 2. Clear the input window by clicking the " $\leftarrow$ " button.
- 3. Drag the SSC block from the OPUS browser into the input window. Select "divide" (/), then drag the RSC block from the OPUS browser into the input window.
- 4. Click "equals" (=).

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PUS Browser	Spe	ctrum Calculator		×
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	4000	Ext	Cancel	Help

Figure 26. Spectrum Calculation Example

a. This calculation generates a transmission spectrum block. For drug checking, we deal with absorbance spectra, which is the inverse of transmission. To convert from transmission to absorbance:

- i. Select the TR Block in the OPUS browser.
- ii. In the "Manipulate" menu in the toolbar, open "AB  $\leftrightarrow$  TR Conversion"

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Figure 27. AB  $\leftrightarrow$  TR Conversion Window

- iii. Select "Automatic".
- iv. Click "Convert".
- 5. You can now save the corrected file under the original file name. Select "Save" in OPUS. Remember that to perform analysis, the AB Block in OPUS Browser must be selected.



4.0 BTNX Test Strips

Please note that we are providing a procedure/directions on using BTNX supplied test strips. This is in no way an endorsement of their product; however this product is currently used by the state of Massachusetts in its prevention programming. These directions are applicable only to test strips sold by this provider and directions will differ if a test strip is produced/sourced from another vendor. Please review package inserts for instructions with other manufacturers and indicate their use in StreetCheck.

The below protocol was modified from the BCCSU Drug Checking Program Operational Technician Manual.

## 4.1 BTNX Fentanyl test strips

- 1. After completing the FTIR scan, a small portion (a few visible grains) of the remaining sample will be scraped from the foil hat using a stainless-steel spatula (dental tool) or taken from the sample bag and added to a labeled medicine cup.
- 2. Dilute the solution to 5mL and gently swirl the mixture to thoroughly dissolve the substance.
- 3. When conducting test strips on residue samples or cooker/cotton samples, add 1 mL of water directly to the cooker with residue, and perform the test strip at a 1mg/1mL dilution. One mg of sample is approximately the equivalent of the size of the tip of a pencil.



Figure 28. Visualize one (1) milligram of sample.

- 4. Remove the BTNX Rapid Response<sup>™</sup> Fentanyl immunoassay strip from the pouch and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 5. Holding the strip from the solid blue end, dip the white end of the strip into the liquid in the sample cup, being careful not to submerge past the blue solid line. For 5 mL dilution you may momentarily rest the strip in the cup.
- 6. When the liquid wicks up the strip to the top of the white portion, (typically about 10-15 seconds), remove the strip from the sample cup and place on a non-absorbent surface or across the top of the cup to allow the test to fully develop.
- 7. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative.
- 8. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands. See the image below for guidance in interpretation of the results.
- 9. If the test results are positive, add another 25mL of water to the cup so that the total volume is 30mL. Repeat steps 3-7 with 30 mL of water only if the 5mL test was positive.



One coloured band (top only) = Fentanyl Positive Two Colour Bands = Fentanyl Negative

Figure 29. Interpreting BTNX Test Strip Results

#### 4.2 BTNX Benzodiazepine Test Strips

- 1. Pulverize your suspected Benzodiazepine pill using a mortar and pestle into a fine powder.
- 2. Add ~1 mg of sample to a labeled medicine cup using a dental tool or microscoop.
- 3. Add 5 mL of water to the medicine cup and gently swirl to dissolve the mixture.
- 4. Remove the BTNX Rapid Response<sup>™</sup> Fentanyl immunoassay strip from the pouch and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 5. Holding the strip from the solid blue end, dip the white end of the strip into the liquid in the sample cup, being careful not to submerge past the blue solid line. For 5 mL dilution you may momentarily rest the strip in the cup.
- 6. When the liquid wicks up the strip to the top of the white portion, (typically about 10-15 seconds), remove the strip from the sample cup and place on a non-absorbent surface or across the top of the cup to allow the test to fully develop.
- 7. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative.
- 8. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands.

#### 4.3 Xylazine Test Strips

The presence of xylazine in the drug supply has increased significantly over the last couple of years. Xylazine test strips are currently being developed and piloted.

#### 4.3.1 BTNX Xylazine Test Strips

Procedure for powder/crystal/rock:

\*Please note that for this protocol we need 5 mg of sample for non-residue samples.\*

- 1. Pulverize crystal and rocks samples via a mortar and pestle into a fine powder prior to adding to your labeled measuring cup.
- 2. Use a small black microscoop to add ~5mg of your sample to your labeled medicine cup.



Figure 30. Visualize five (5) mg of sample.

- 3. Dilute the solution to 5mL and gently swirl the mixture to thoroughly dissolve the substance.
- 4. Remove the BTNX Rapid Response<sup>™</sup> Xylazine immunoassay strip from the pouch and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 5. Holding the strip from the solid red end, dip the white end of the strip into the liquid in the sample cup, being careful not to submerge past the red solid line. For 5 mL dilution you may momentarily rest the strip in the cup.
- 6. When the liquid wicks up the strip to the top of the white portion, (typically about 10-15 seconds), remove the strip from the sample cup and place on a non-absorbent surface or across the top of the cup to allow the test to fully develop.
- 7. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands. See the image below for guidance in interpretation of the results.



Figure 31. Interpretation of Xylazine Test Strip Results.

8. If a sample produces an unexpected XTS positive (donated as non-opioid but shows positive or the triplicate testing results in inconsistent results), dilute to 30 mL (by adding 25 mL of water), and perform XTS at 30 mL dilution.

Procedure for cooker/residue samples (1mg/1mL dilution)

- 1. Add 1 mg of your residue sample to a medicine cup using your spatula/dental tool to scoop just enough sample to cover the tip of your tool.
- 2. Using your 1 mL disposable syringe, add 1 mL of water to the medicine cup or directly to the cooker with residue. Note to gently rotate the cooker such that the water travels up the sides of the cooker.
- 3. Remove the BTNX Rapid Response<sup>™</sup> Xylazine immunoassay strip from the pouch and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 4. Holding the strip from the solid red end, dip the white end of the strip into the liquid in the sample cup, being careful not to submerge past the red solid line.
- 5. When the liquid wicks up the strip to the top of the white portion, (typically about 10-15 seconds), remove the strip from the sample cup and place on a non-absorbent surface or across the top of the cup to allow the test to fully develop.
- 6. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands.
- If a sample produces an unexpected XTS positive (donated as non-opioid but shows positive or the triplicate testing results in inconsistent results), dilute to 5 mL (by adding 4 mL of water to the cooker), and perform XTS at 5 mL dilution.

#### 4.3.2 Cassette Style Xylazine Test Strips

- 1. A small portion (a few visible grains) of the remaining sample should be scraped from the foil hat using a stainless-steel spatula (dental tool) or taken from the sample bag and added to a labeled medicine cup.
- 2. Approximately 1 mL of water should be added to the medicine cup and the mixture should be gently swirled to thoroughly dissolve the substance.
- 3. Remove the xylazine test strip from the packaging, lay it on a flat surface, and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 4. Obtain a plastic Disposable Graduated Transfer Pipette. Squeeze the top of the pipette letting the air out before placing it in the solution to prevent splashing. Release your grip to fill the pipette with the solution.
- 5. Add ~3 drops of solution into the specimen well on the test strip.
- 6. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative.
- 7. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands.
- 8. For a positive result, one line should be in the control line region (C). For a negative result, two lines appear. One line should be in the control line region (C), and another line should be in the test line region (T).

## 4.4 Testing Cannabis Using an FTS

- 1. To 0.15 g of cannabis (about 1 rounded teaspoon) in a medicine cup, add 5 mL of water and gently swirl. Let the water/cannabis solution sit for about 1 minute.
- 2. In the meantime, remove the BTNX Rapid Response<sup>™</sup> Fentanyl immunoassay strip from the pouch and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 3. Holding the strip from the solid blue end, dip the white end of the strip into the liquid in the sample cup, being careful not to submerge past the blue line.
- 4. When the liquid wicks up the strip to the top of the white portion, (typically about 10-15 seconds), remove the strip from the sample cup and place on a non-absorbent surface or across the top of the cup to allow the test to fully develop.
- 5. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands.



Figure 32. Direction for Testing Cannabis Using a FTS

## 4.5 Testing Blotter Paper Using a FTS

- 1. Add the blotter paper sample to a medicine cup. Add 5 mL of water and gently swirl. Let the solution sit for about 1 minute.
- 2. In the meantime, remove the BTNX Rapid Response<sup>™</sup> Fentanyl immunoassay strip from the pouch and visually check for any obvious defects. If the package is ripped or torn, discard the test strip.
- 3. Holding the strip from the solid blue end, dip the white end of the strip into the liquid in the sample cup, being careful not to submerge past the blue line.
- 4. When the liquid wicks up the strip to the top of the white portion, (typically about 10-15 seconds), remove the strip from the sample cup and place on a non-absorbent surface or across the top of the cup to allow the test to fully develop.
- 5. Once the liquid has wicked through the entire test region, make an initial reading to determine if the sample is positive or negative. Visually examine the strip under bright, direct lighting in order to avoid missing fainter bands.

\*Note: You may send a Cannabis or Blotter paper sample for confirmatory testing only if it meets one or more of the following criteria:

- FTS result is positive.
- Adverse health event has occurred.
- If the participant has tested positive on a drug test for another substance when they've only used Cannabis or Blotter Paper.
- FTIR analysis results in an unexpected active substance.
- Suspected to have been contaminated with another substance: Fentanyl, Synthetic Cannabinoids (K2, spice, etc.), Cocaine, etc.



# 5.0 Off-Site Laboratory Testing

After each same

sufficient leftover sample, all remaining sample material will be placed into a separate mylar pouch bag, labeled following the form's instructions, and placed into a pre-addressed, prestamped mailing envelope for validation at the Drugs Data Laboratory (Drugsdata.org). The packages can be mailed from any mailbox or mailing center.

#### 5.1 Mailing Guidance

When sending samples in a box or padded envelope, you can put multiple closed envelopes in the package. The padded envelope does not need to be sent only containing the cooker. Similarly, the box does not need to be sent only containing the pipe. As long as items are packaged and labeled per our protocol, other samples can be placed in the box/padded envelope as well.

If you are going to send more than one inflexible item (cooker, pipe, container), use a padded envelope or box. If you are mailing more than 5 cookers or other containers, use a box.

Form of the original sample /original packaging	What to mail it in	How to mail	Notes
Baggie	Envelope (not padded)	One stamp is sufficient Can mail in blue box or at post office	
Container (Plastic Medicinal Vial, small metal container, etc.)	Padded Envelope	Place all other samples from that day in the padded envelope. Seal, address, bring to post office and mail at service desk.	It is OK to add multiple items to a padded envelope! Typically, do not send more than 2-4 hard items or, if heavier, shift to a box.
1 Cooker	Envelope with cooker wrapped in bubble wrap	Seal and bring to the post office to get weighed and mailed.	Not a recommended procedure.
More than 1 cooker	Padded Envelope or Box	Place all other samples from that day in the padded envelope. Seal, address, bring to post office and mail at service desk.	It is OK to add multiple items to the padded envelope! Typically, do not send more than 5 items in a padded envelope. If items are heavier or if you have multiple cookers ( <u>&gt;</u> 5), use a box

Pipe/Stem	Box or Padded Envelope	Place all other samples from that day in the box. Seal with packing tape, address, bring to post office and mail at service desk.	It is OK to add multiple items to the box!
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## 6.0 General FTIR + OPUS Facts/Points

Designate the relative concentration of each substance as major, minor, trace, or unknown. The relative ratio for each substance is based on the relative heights of the peaks compared to one another.

\*\*\*If you would like another technician to review the spectrum, please contact Abby or Cole with the sample ID. (abbyedelmann@brandeis.edu)[colejarczyk@brandeis.edu]\*\*\*

Major\*

- The substance appears to contribute substantially to the overall composition of the sample.
- The substance has clear peaks and are the tallest peaks within the spectrum.
- Substances in major amounts in a sample likely make up more than ~50% of the overall sample composition.

Minor\*

- The substance appears to contribute notably to the overall composition of the sample, but not as substantially as a major component.
- The substance has clearly identifiable peaks but are not the tallest peaks within the spectrum.
- Substances in minor amounts in a sample likely make up 10-50% composition of the sample.

Trace\*

- The substance is barely visible on the original sample spectrum.
- Substances in trace amounts in a sample likely make up 5-10% composition of the sample and is near the limit of detection (LOD)\*\*.
- There are indications of the substance but it is very hard to determine. Typically a substance with very small peaks but are visible above the noise of the baseline.

Unknown

- The technician is NOT confident in the presence of the substance in the sample.
- Unknown refers to the technician being uncertain if the compound is in the sample at all. There are indications of the substance in the spectrum but the technician has doubts if the substance is actually present.
- The uncertainty in the identification can be due to the substance being near the LOD\*\*, noise inhibition, swamping of identification from other substances, etc.

\*The technician is confident in the presence of the substance based on the FTIR spectrum. \*\*Limit of Detection (LOD) for the Bruker ALPHA II is ~5%. Substances under ~5% composition in a sample will not be detectable on FTIR.

It is possible and commonly seen that more than one substance in a sample will have overlapping peaks at the same location which will result in **stacking**, causing the peaks to increase in height.

**Shifting** of peaks can occur in a spectra where the relative height of peaks will match the reference but not the location.

#### Library Usefulness

Use	Library	Path	Status	Entries	Description
	ATR-LIB-PHARMA-1-472-2.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	5782	ATR-FTIR Pharmaceuticals Library, Vol.
	ATR-LIB-PHARMA-2-472-2.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	3654	ATR-FTIR Pharmaceuticals Library, Vol.
	ATR-LIB-PHARMA-3-472-2.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	51	ATR-FTIR Pharmaceuticals Library, Vol.
	ATR-LIB-PHARMA-4-472-2.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	618	ATR-FTIR Pharmaceuticals Library, Vol.
	BCCSU FTIR-ATR Library 2022-	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	95	BCCSU ATR-FTIR Library for use in dru
	BPAD.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	234	Bruker Optics ATR-Polymer Library
	Demolib.s01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	350	General Library IR
	RAMDEMO.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	246	Bruker Raman Demolibrary
V	RM08272019.S01	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	624	SWGDRUG, August 27, 2019
	SR.IDX	C:\Users\Public\Documents\Bruker\OPU	$\checkmark$	200	Demo Database

At a minimum, BCCSU and SWGDRUG libraries should be selected. TICTAC and Georgia Crime Lab libraries can be useful to add if you're having trouble identifying a substance with solely two libraries. Select PHARMA libraries when analyzing a pill. The number of entries column lists the number of references in each library, the more entries, the longer the library search will take. When searching multiple libraries at once, it is more than likely the same reference will come up as a match multiple times. Naming of the references across libraries are usually the same except for small differences (ex: Lactose, alpha-Lactose, D-(+)-LACTOSE POWDER).

Spectrum Search		×
Spectrum Search Search Parameters Select Libraries		
Search algorithm		
O Standard	⊖ Mixture analysis	
<ul> <li>Spectrum correlation</li> <li>Normalization method</li> </ul>		
Vector normalization	~	
Derivative 1st derivative	~	
O Use existing peak tables from selected spectra		
Minimum hit quality: 100 Maximum number of hits:		
Search Library	Cancel	Help

#### Spectrum Search Default Settings

