

Standard Operating Procedure

Bruker Alpha-P ATR FTIR (Diamond-ATR)

1. Introduction

1.1. Purpose

To outline the procedure for the infrared (IR) analysis of a course-provided or course-produced sample with the help of a Bruker Alpha-P Attenuated Total Reflectance (ATR) Fourier Transform InfraRed spectrometer (FTIR).

1.2. Scope

Applicable to undergraduate and graduate students enrolled in courses within the Department of Physical and Environmental Sciences. This document may also be used as a template for research users within the Department of Physical and Environmental Sciences.

1.3. Responsibility

1.3.1. TRACES Users

- 1.3.1.1. All Users must obtain training with TRACES Staff prior to system operation. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols.
- 1.3.1.2. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.
- 1.3.1.3. User must ensure that FTIR is fully operational before commencing use of the instrument.
- 1.3.1.4. Instrument time must be booked by the User via the online booking system prior to system operation.

1.3.2. TRACES Staff

- 1.3.2.1. It is the responsibility of TRACES Staff to provide introductory and, if required, subsequent training to all users.

1.4. Accountability

Principal Investigator

1.5. Emergency Contacts

1. Emergency Fire/Police/Ambulance:911
2. UofT Police:416-978-2222
3. TRACES Lab Manager 416-7239

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2. Safety

IMPORTANT:

- Most solvents are flammables
- Many samples and materials requiring analysis are toxic and dangerous
 - Take the appropriate precautions
- Seek assistance from TRACES Staff if you have any concerns

3. Referenced Documents

- 3.1. Bruker OPUS Spectroscopy Software ver.7 ed.2011
- 3.2. Bruker Alpha User Manual ed.2 (p/n1005541)
- 3.3. Bruker Attenuated Total Reflectance (ATR) AN#79 (BOPT-4000352-01)
- 3.4. Perkin Elmer Attenuated Total Reflectance (ATR) TN:007024B_01
- 3.5. <http://www.utsc.utoronto.ca/~traceslab/PDFs/Principles%20of%20ATR.pdf>
- 3.6. <http://www.utsc.utoronto.ca/~traceslab/PDFs/FTIR%20Theory.pdf>

4. Chemicals & Supplies

- 4.1. Bruker Alpha-P ATR FTIR (diamond-ATR)
- 4.2. Isopropanol or Ethanol
- 4.3. Kimwipes
- 4.4. Course-provided or Course-produced samples ONLY
 - 4.4.1. **Use of this instrument for purposes other than laid out by the course instructor will result in severe penalty and academic offense.**

5. Personal Protective Equipment

- 5.1. Nitrile/Nylon Gloves
- 5.2. Laboratory Coat/Jacket
- 5.3. Safety Glasses

6. COVID-19 Related Safety Precaution

6.1. Do not enter if you have one of the following symptoms:

- 6.1.1. cough
- 6.1.2. fever
- 6.1.3. difficulty breathing
- 6.1.4. pneumonia in both lungs
- 6.1.5. travelled outside the country in the last 14 days

6.2. Cleaning and Sanitizing Hands**6.3. General Laboratory Practice during COVID-19**

- 6.3.1. You MUST work >2m from others. The use of adjacent instruments less than 2m is suspended at this time. Please schedule your analysis appropriately.

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7. Safety and Electronic Equipment Concern

7.1. Chemical Safety

- 7.1.1. Read and become acquainted with the SDS of all the chemicals you will be using and/or exposed to during the lab period -including the alcohols for cleaning.
- 7.1.2. Dispose of the chemical waste and chemicals-soaked paper in the designated containers.

7.2. Electronic Safety

- 7.2.1. Please refer to the manufacturer's recommendations and warning label.

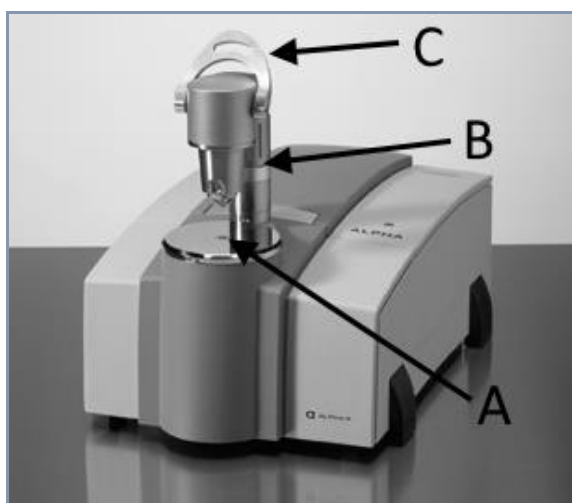


Figure 1

8. Acquiring FTIR Spectra

8.1. Before Commencing work

- Obtain a bottle of alcohol and several paper towels. Kimwipes should also be available near the FTIR.
- Clean the keyboard, mouse, GLOVES, and any area you will be exposed to (monitor excluded) with the solvent-soaked paper towels.
- DO NOT spray directly onto surfaces.
- Discard ALL the paper towels (whether they were used or not) into the designated waste container.
- Wait 5 minutes before commencing work.

8.2. Before Commencing Analysis

- 8.2.1. DO NOT PLACE SAMPLE onto CRYSTAL(A); keep anvil (C) in the up position.
- 8.2.2. ENSURE THAT THE ATR CRYSTAL (A) IS CLEAN OF ANY MATERIAL.
- 8.2.3. Clean the crystal with the alcohol and let it dry.

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8.3. Starting the Software (if not already done)

- 8.3.1. Click the OPUS icon (select OPUS 7.0 or 7.5).
- 8.3.2. Username & Password (please ask TA/TRACES Staff).
- 8.3.3. Allow the Alpha-P to run diagnostic test.
- 8.3.4. Anvil (C) remain in the up position during background runs.
- 8.3.5. When finished you will be prompted to 'CONTINUE' or 'RUN Background'.
- 8.3.6. Run 'Background' once completed, press 'CONTINUE'.

8.4. Setting up FTIR

8.4.1. Analysis flow follows the ICONS from LEFT to RIGHT



8.4.2. Click on the Measurement icon



- 8.4.3. Start Background Measurement; keep anvil (C) in the up position.
- 8.4.4. The green bar indicates the progress of the measurement (sample and background).
- 8.4.5. Enter Sample Name once the buttons are no longer grayed out.

8.5. Sample Loading

8.5.1. Liquid Samples

- 8.5.1.1. Introduce the liquid sample (1 drop) on to the ATR crystal (A) keep anvil (C) in the up position

8.5.2. Solid Samples

- 8.5.2.1. Place the solid sample so that it covers the ATR crystal (A). Gently press down on the anvil (C) till it make contact the sample. If necessary, rotate the anvil arm (B) to ensure full contact is made with the crystal. In most cases, solid samples that are ground to a finer powder, result in sharper FTIR spectra.

8.6. FTIR Sample Analysis

- 8.6.1. Select **Start Sample Measurement**
- 8.6.2. The green bar indicates the progress of the measurement

8.7. FTIR Troubleshooting

- 8.7.1. Why do I have no spectra?
 - 8.7.1.1. Did you select the Measure Background button by mistake?
- 8.7.2. Why are my spectra peaks large and wide in shape?
 - 8.7.2.1. Reduce the amount of sample or dilute (speak to your TA) your sample.
- 8.7.3. Why are some peaks not in my spectra but in my partners?
 - 8.7.3.1. Did the sample make good contact with the crystal?

9. Evaluating FTIR Spectra

9.1. If the sample requires Baseline Correction click the icon



9.2. Click on the icon



- 9.2.1. Right-click mouse and to choose Single Peak Pick for manual selection.

9.3. Click on the icon



9.4. Click on the icon



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9.5. Remove your sample and clean the surrounding area and the ATR CRYSTAL (A) BEFORE departing with an alcohol. Place all waste into the appropriate waste bottle.

10. Post FTIR Analysis

10.1.1. Obtain a bottle of alcohol and several paper towels. Kimwipes should also be available near the FTIR.

10.1.2. Clean the keyboard, mouse, GLOVES, and any area you will be exposed to (monitor excluded) with the solvent-soaked paper towels.

10.1.3. DO NOT spray directly onto surfaces.

10.1.4. Discard ALL the paper towels (whether they were used or not) into the designated waste container.

10.1.5. Wait 5 minutes before next users can use the area and instrument.

***The TRACES Manager will provide further details during hands-on training.**