

ACDA6020 Lab Report 1.

Identification of organic materials using FT-IR

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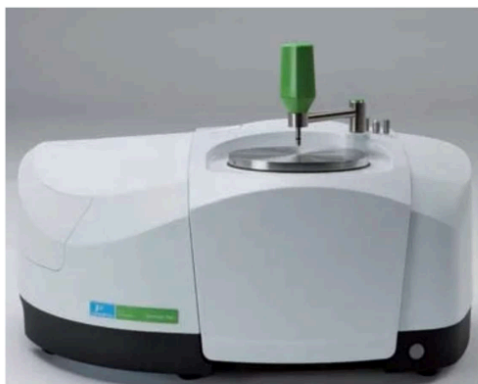


Figure 1 Perkin-Elmer Spectrum 2 [13]

LAB REPORT #1

Identification of organic materials using FT-IR

ABSTRACT

The use of FTIR in the identification of unknown compounds, their functional groups and how these functional groups of compounds may be used as precursors for the production of explosives.

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ACDA6020

Table of Contents

1. Introduction	2
1.1 Aim	2
1.2 Hypothesis	2
2. Theory	2
2.1 Application	3
3. Materials and Equipment	3
4. Method	3
5. Results	4
.....	5
5. Discussion	6
6. Conclusion	6
Bibliography	7

1. Introduction

To demonstrate the capabilities of equipment and the effectiveness of the spectroscopic methods this equipment uses.

1.1 Aim

The aim of this activity is to confirm the identity and chemical functionality of a given sample to clear the sample for disposal using equipment that utilises spectroscopic methods. For this activity FT-IR spectroscopy was used. There were a variety of samples labelled A-D being investigated, there was a list of known compounds given but the labelling of the samples did not correlate to the known list.

1.2 Hypothesis

Based on previous use of FT-IR spectroscopy it was predicted that the spectra output from the software could need further analysing due to various factors however the functional group of the compound will be easier to distinguish. From ^{first} assumptions when looking at the samples it was predicted that sample D is Acetaminophen due to its visible colour and texture. However, samples A-C would need further analysis to get initial identification and then further confirmation.

2. Theory

FTIR or Fourier Transform Infrared spectroscopy, fundamental technology relies on a molecule's atomic vibration, this molecule will only absorb specific frequencies of infrared light. This absorbed radiation into the molecule is then able to be detected and classified by FTIR because different molecules have different infrared spectrum [1].

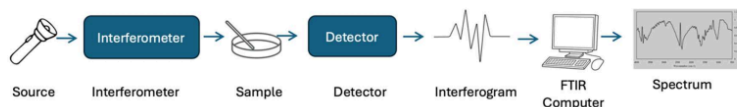


Figure 2 Simplified FTIR processing

As seen in figure 2 a simplified processing order performed by the FTIR and the output the 2 detectors for this process are both before and after the sample. As part of this process and how the detector can 'see' comes down to the medium surrounding the sample. This can either be potassium bromide, or a crystal. Diamond is usually used, but both methods work in a different way and have both have positives and negatives to each.

Potassium Bromide (KBr)  2

KBr is used as a carrier due to its 100% transmittance in the IR region [2]. Samples are ground and mixed with a specified particle size of KBr and then pressed into a pellet of a specified diameter. This is known as the diffuse reflection method [3]. This method can provide more background against legacy reference libraries as well as requiring a smaller sample size than ATR [4]. This method requires extensive sample preparation, including grinding, mesh filtering, pellet pressing, expensive equipment to perform the preparation, prepping in a vacuum, the pellets must stay water free or under a vacuum as the water present in the sample can peak at 3200 cm^{-1} , which overlaps with functional group peaks and could impact results [2].

Diamond (ATR)

FTIR instruments often use diamond for their Attenuated Total Reflection crystals, this is due to the diamonds mechanical properties of being both hard and durable as well as diamond having a high refractive index allowing for high quality spectral measurements it also boasts minimal sample preparation [5]. In its simplest form ATR works in this case by reflecting an IR beam off the surface of a crystal, in this case a diamond from the underside and interacts with the sample, this sample of a lower refractive index than the diamond is placed onto the surface of the diamond. Recording the intensity of the reflected light before and after sample placement produces the characteristic infrared spectrum of the sample [6] [7].  3

2.1 Application

There is a wide variety of applications for FTIR, specifically within OME throughout all stages of its lifecycle from initial identification, raw material and precursor purity to trace analysis post blast as well as identifying stabiliser depletion in propellants indicating chemical breakdown that can be used to either extend shelf life or determine disposal [8] *excellent reference to life cycle of explosives*

3. Materials and Equipment

Noted hazards associated with this task included that sample C, the test sample was an oxidiser.

The waste routes were considered and followed general waste disposal for any generated waste from this task.

Personal Protective Equipment (PPE) required for this task:

Lab coat

Nitrile gloves

Safety glasses.

Perkin-Elmer spectrum 2.

ATR Type: diamond

Cleaning solvent: IPA

Other consumables:

Blue roll

Spatula

4. Method

1. Put on PPE
2. Lift the arm of the FTIR and twist away from the sample plate
3. Remove the foot from the arm
4. Clean the foot that has been removed
5. Clean the sample plate, paying particular attention to the diamond ATR.
6. Place the foot back on the arm
7. Place a small amount of sample onto the centre of the plate, on the diamond.
8. Twist the arm back over to the centre of the plate and lower the foot.

9. Use the ratchet to ensure contact between sample and ATR diamond at the right pressure (one click).

10. Ensure cleaning solvent and percentage used have been input into the software.

11. Input the following settings onto the computer software:

Scans: 15
Background scans: 15
Resolution: 4
Acquisition parameters: Strong

12. Click go on the software to initiate scanning.

13. The first output given by the software is a preliminary result

14. Click go again using the same button to run the 15 scans.

15. Once the 15 scans & 15 background scans are complete the software displays the spectra result.

16. Run the results against the library to get results and confidences.

N.B- It is important to note that the software has an inbuilt function to auto pick and label what it deems as the strongest peaks. Avoid using this function as this is often wrong.

17. The result table from the library reference is often the most accurate with confidences found in percentages.

5. Results

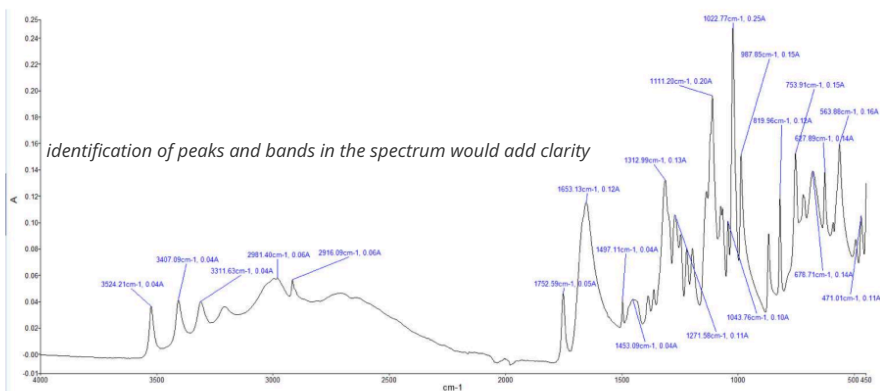


Figure 3 Sample A Spectra Result

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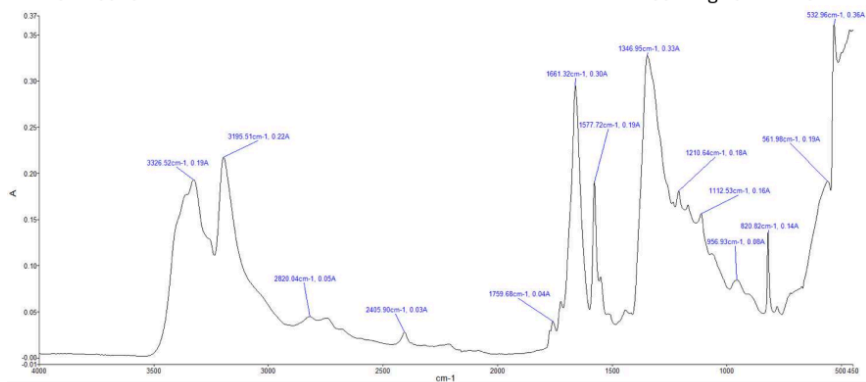


Figure 4 Sample B Spectra Result

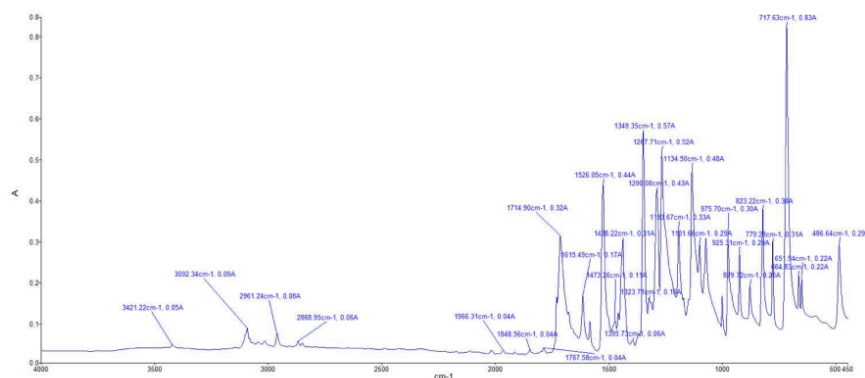


Figure 5 Sample C Spectra Result

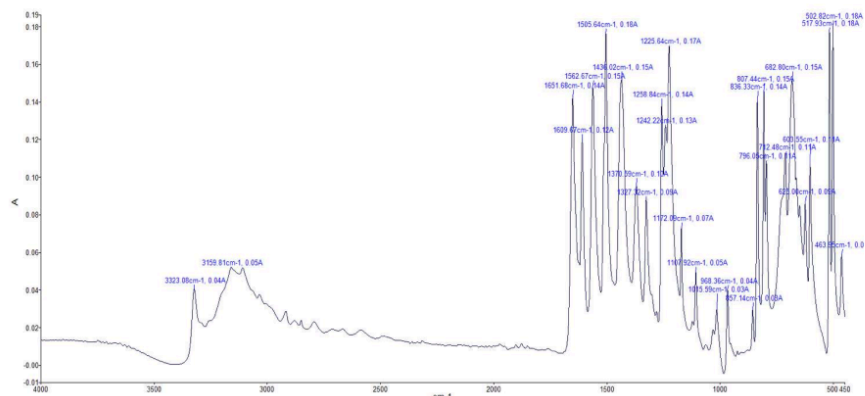


Figure 6 Sample D Spectra Result

5. Discussion

The functional group for each spectra result can be found in the wavenumber region of 1500-4000cm⁻¹.

It can be seen in figure 3, the spectra output from sample A the peaks from the functional group region indicate from the list of given examples that sample A is Ascorbic acid. This is due to the absorption regions for ascorbic acid:

Key absorption regions	Spectra result as seen in figure 3
Hydroxyl groups O-H. 3200-3600cm ⁻¹	3525 cm ⁻¹
Stretching vibrations of C-H bonds 3100-2840 cm ⁻¹	2981 cm ⁻¹
Carbonyl group C=O 1750-1760 cm ⁻¹	1752 cm ⁻¹

[9] [10]

This comparison was also performed on samples B and D comparing the peaks in spectra to the key absorption regions for guanidine and acetaminophen indicating that sample B, as seen in figure 4 is guanidine and sample D, as seen in figure 5 indicating acetaminophen.

Figure 6, sample C is methyl-3-nitrobenzoate. The output from the FTIR reference library when compared noted that this compound was ethyl-4-nitrobenzoate with 52% confidence, this incorrect labelling could be for a variety of factors including not having methyl-3-nitrobenzoate in the library, and so the closest compound in the library that best matched is what was given, it could have also been due to a contaminated sample, and rarely it could also be the IR light interacting with the sample and the resulting breakdown products [11].  4

6. Conclusion

Some of the functional groups of these materials could indicate their use in explosives, however this isn't always practical, or there are better ways to produce the molecules needed. For example, methyl-3-nitrobenzoate could go through redox to remove an oxygen molecule to form OCH₃, but in reality a common method to produce OCH₃  5 would use Williamson ether synthesis [12] this would also be the case for acetaminophen the removal of an oxygen molecule could produce an explosive precursor but again there are more practical ways to produce these. Guanidine nitrate along with nitric acid are precursors to produce nitroguanidine [13] a well-known propellant. There has been some research into the use of ascorbic acid in black powder substitutes as well as patents online for new propellant compositions consisting of potassium nitrate and ascorbic acid.

Using FTIR to identify the compounds  6 work well, the process is quick but is only as accurate as the reference library, using the confidence percentages works well to try and navigate some of the incorrect labelling, however when repeating this next time, a recommendation of using Raman spectroscopy to get conformation would be useful.

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GRADEMARK REPORT

FINAL GRADE

61 /100

GENERAL COMMENTS

Engagement with Literature 10/15

A good list of cited sources, but the literature engagement could be better and some statements (e.g., IR “breakdown products”) are incorrect. Some references are appropriate, but several peak assignments lack citation. IEEE formatting is inconsistent. More scholarly FT-IR references are needed.

Knowledge and Understanding 13/20

You show some understanding of FT-IR concepts and functional groups, but the theoretical framework is weak. Vibrational modes are mentioned as a foundation of FT-IR spectrum. The introduction focuses on colour/texture assumptions and a “hypothesis”, which are not relevant to the aim of this practical. Identifying functional groups on spectra would improve clarity.

Cognitive and Intellectual Skills 12/20

You correctly identify the samples, but the reasoning is very descriptive. No comparison with literature values is provided, and the interpretation lacks critical depth.

There is systematic functional-group analysis for each sample, and a clear comparison table is used. Sample C is interpreted correctly despite the library mismatch. There is no fingerprint-region analysis or literature comparison, but the reasoning quality is adequate.

Practical Skills 14/30

This is where the report is weakest. The method is far from the required SOP-level detail. Critical FT-IR parameters are missing, including: apodisation, baseline/smoothing settings, ATR correction, atmospheric suppression, scan speed, detector type, beam splitter, and IR source, sample-handling details, QC criteria (background quality, S/N threshold, reproducibility checks). Several statements are unclear or inaccurate (“ensure cleaning solvent is input in the software”), and the method cannot be reproduced by another analyst. OME relevance addressed.

Transferable Skills 12/15

The report reads well, with figures and structures included. Formatting is generally consistent but the writing sometimes uses informal phrasing. Good graphical representation of theory and results

Text Comment. first

Strikethrough.



Comment 2

The next two paragraphs are not very relevant to your report;

KBr pellet discussion is too detailed for this context and distracts from the *ATR methodology* you were required to develop



Comment 3

no mention of **vibrational modes**

Text Comment. excellent reference to life cycle of explosives

Text Comment. identification of peaks and bands in the spectrum would add clarity



Comment 4

No fingerprint-region discussion and also FT-IR is undestructive method so breakdown products must be of different origin



Comment 5

van tehse processes occur spontaneously?



Comment 6

or the knowledge and spectra-reading skills of the operator :-)



incorrect authors' names

To correctly list the authors' names when using Word referencing, click the 'Edit' button in the Author section and enter the first name. Then click 'Add', enter the next name, and click 'Add' again. When all names have been entered, click 'OK'.



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