

Lab Report 1 Final - OM

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FT-IR SPECTRAL ANALYSIS.

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I. Abstract

This report presents the use of FT-IR spectroscopy with a PerkinElmer Spectrum IR ATR system to identify four unknown organic samples. Consistent cleaning, contact pressure, and scanning parameters ensured reproducible, high-quality spectra. Each sample was analysed using 15 background and 15 sample scans, and results were verified through both Spectrum IR library matching and manual interpretation. The compounds were confirmed as guanidine nitrate, acetaminophen, ascorbic acid, and ethyl-3-nitrobenzoate, with confidence levels between 95–99 %. The study highlights how precise methodology and expert analysis underpin reliable FT-IR identification.

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1. Introduction

Fourier Transform Infrared (FT-IR) spectroscopy identifies chemical compounds by detecting characteristic vibrational frequencies of molecular bonds. Each functional group absorbs infrared radiation at specific wavenumbers (cm^{-1}), producing a unique spectral fingerprint. Using Attenuated Total Reflectance (ATR) enables direct surface analysis of solid samples without complex preparation [1].

This experiment analysed four unknown organic materials—representative of both energetic and pharmaceutical compounds—to obtain clear, reproducible spectra. Results were interpreted through Spectrum IR's automated library matching and manual verification by comparing functional group frequencies. Emphasis was placed on reliable methodology, ensuring consistent contact pressure, optical cleanliness, and baseline stability to achieve dependable results.

2. Methodology

2.1. Safety and Handling

All work was completed wearing nitrile gloves and safety glasses to prevent skin contact or sample contamination. Samples A, B, and D were disposed of via general waste, while Sample C (ascorbic acid) was disposed of in contamination waste due to mild acidity. Work was carried out on a stable, vibration-free bench to ensure measurement consistency. Maintaining a clean environment was essential, as residue on optical surfaces can lead to false absorbances and spectral distortion.

2.2. Equipment and Operating parameters

Component	Specification / Setting
ATR-FTIR Spectrometer	PerkinElmer Spectrum IR (Diamond ATR crystal)
Spectral range	4000 – 250 cm^{-1}
Background scans	15 (equal to sample scans)
Sample scans	15 (co-added for improved S/N)
Resolution	$\approx 4 \text{ cm}^{-1}$ (instrument default)
Apodization	Happ-Genzel (default)
ATR correction	Not applied – avoids refractive over-scaling
Baseline correction	Not applied – sample-only measurement
Output format	CSV and native Spectrum IR for library analysis

Table 1 – Equipment & Parameters Table [2]

Averaging 15 scans per measurement improved the signal-to-noise ratio by $\sqrt{15} \approx 3.9$, ensuring consistent intensity without altering relative absorbances [3].

2.3. Measurement Procedure

2.3.1. Follow the manufacturer's manual for start-up, stabilisation, and pre-run checks, including background verification and method setup [2]. These

parameters were pre-configured by the laboratory technician and confirmed before sample measurement.

2.3.2. Sample placement and contact control: Each powdered sample was placed directly on the metal base plate of the ATR module, ensuring a thin, even layer fully covering the contact area beneath the diamond tip [4]. The diamond crystal, located in the ratchet-controlled pressure arm, was then carefully lowered until the ratchet clicked, providing uniform torque and reproducible pressure across all runs. Correct placement and pressure were essential, as the penetration depth of the evanescent field depends directly on intimate contact between the diamond and the powder surface.

2.3.3. Spectral acquisition: Each sample was scanned 15 times across the range *averaged* 4000–250 cm^{-1} , and the spectra were co-added to reduce random noise. The live energy trace was observed to ensure stable signal intensity; any fluctuations indicated poor contact or uneven sample spread and prompted reseating of the material.

2.3.4. Data storage and documentation: Each spectrum was saved as Sample_[ID].csv and annotated with sample appearance, contact torque, and scan quality notes in the laboratory logbook.

- 2.3.5. Post-run cleaning and validation: Residual material was removed from the base plate using dry tissue, followed by wiping both the plate and the diamond tip in the ratchet arm with IPA. A blank scan was then collected to confirm the baseline was flat before continuing (the operator sometimes decided not to carry out an extra blank scan, this could have reduced the fidelity of future scans).
- 2.3.6. Verification and validation: After collection, each spectrum was compared to the Spectrum IR library database. The library match (typically 95–99 % confidence) provided an initial identification, which was then manually confirmed by locating at least three diagnostic peaks corresponding to expected molecular vibrations. This ensured that each match was supported by physical interpretation rather than software prediction alone.  2
- 2.3.7. Rationale: Pressure consistency (from ratchet mechanism), and post-scan cleaning directly control the accuracy and repeatability of ATR-FTIR measurements. Inconsistent contact can reduce peak intensity, while contamination causes false signals in the fingerprint region. The standardised approach used here ensured valid, comparable spectra across all samples.

2.4. Quality control and troubleshooting

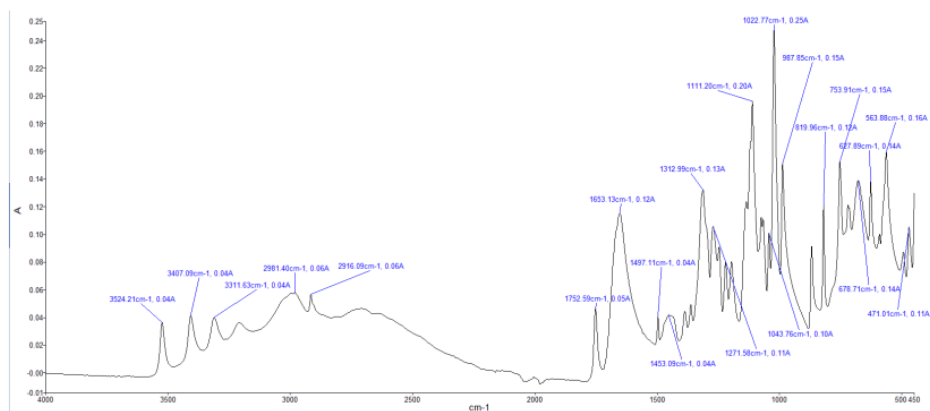
- Background scans taken before each session removed atmospheric H₂O and CO₂ interference [5].
- No baseline correction preserved authentic absorption profiles (can shift band positions) [6].
- Constant contact pressure maintained uniform penetration depth (Ratchet Mechanism).
- IPA cleaning and blanks prevented carry-over contamination.
- Peak reproducibility within $\pm 5 \text{ cm}^{-1}$ and S/N > 100:1 confirmed measurement stability.

3. Results

3.1. Spectral Overview

All four spectra were sharp and well-resolved, confirming proper cleaning and consistent contact during acquisition. Spectrum IR produced confidence matches between 95 % and 99 %, which were verified manually by identifying diagnostic absorption bands characteristic of each compound [7]. 

3.2. Sample A – Guanidine Nitrate



Sample A exhibited broad N–H stretching absorptions from 3524–3311 cm^{-1} , characteristic of the guanidinium cation ($\text{NH}_2\text{--C(=NH)--NH}_2^+$). Weaker C–H stretches at 2981 and 2916 cm^{-1} likely resulted from minor organic residues. Strong NO_2 asymmetric and symmetric stretches at 1653 and 1313 cm^{-1} confirmed the nitrate anion, while C–N stretches between 1111–1023 cm^{-1} indicated the guanidine framework. Minor peaks below 820 cm^{-1} were associated with N–H wagging and lattice vibrations [8]. The pattern produced a 95.2 % match and was manually confirmed as guanidine nitrate [9].

3.3. Sample B – Acetaminophen

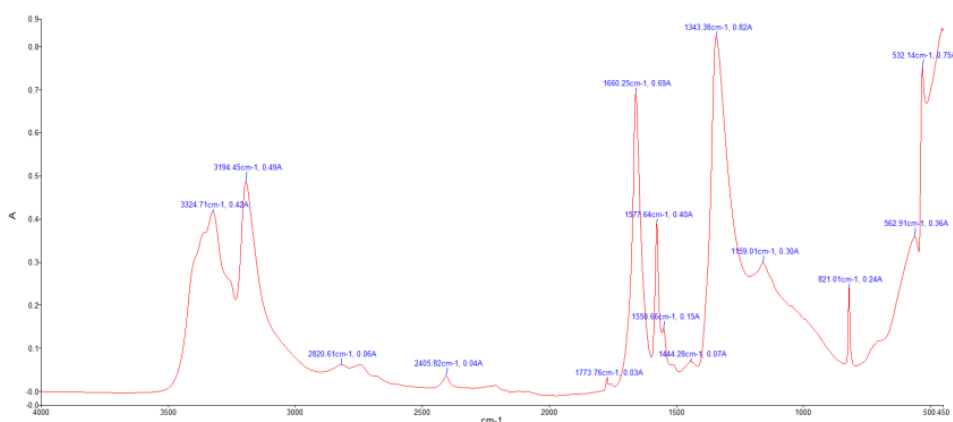


Figure 2 – Sample B – Acetaminophen

Sample B showed a broad band between 3325–3194 cm^{-1} from overlapping O–H (phenolic) and N–H (amide) stretches. A strong C=O stretch at 1660 cm^{-1} identified the amide carbonyl, while 1578 cm^{-1} represented C=C stretching of the aromatic ring. Bands at 1343 cm^{-1} (C–N) and 1159 cm^{-1} (C–O) confirmed amide and phenolic linkages, while 821 and 563 cm^{-1} indicated out-of-plane aromatic C–H bending, consistent with para substitution [8].

Although early numerical output emphasised low-frequency peaks ($< 500 \text{ cm}^{-1}$), these were later identified to be minor background artefacts. Minor intensity differences were attributed to slight background mismatch or over-packing. The 99.4 % Spectrum IR match verified acetaminophen as the correct identity [9].

3.4. Sample C – Ascorbic Acid

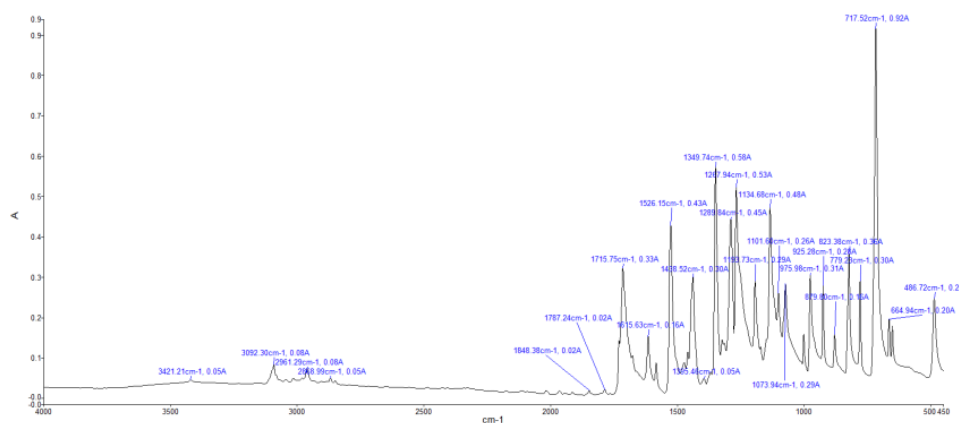


Figure 3 – Sample C – Ascorbic Acid

Sample C exhibited a broad O–H stretch centred at 3421 cm^{-1} , consistent with multiple hydroxyl groups in the enediol-lactone structure of ascorbic acid. Several C–H stretches appeared between $3092\text{--}2869\text{ cm}^{-1}$, and a strong C=O absorption at 1716 cm^{-1} indicated a lactone carbonyl. The 1526 cm^{-1} band represented O–H bending, while multiple C–O stretches between $1349\text{--}1100\text{ cm}^{-1}$ originated from alcohol and ether linkages [8]. A sharp 717 cm^{-1} peak corresponded to C–H wagging in the lactone ring. The library match provided a 95.2 % confidence match, confirming the compound as ascorbic acid [9].

3.5. Sample D – Ethyl-3-Nitrobenzoate

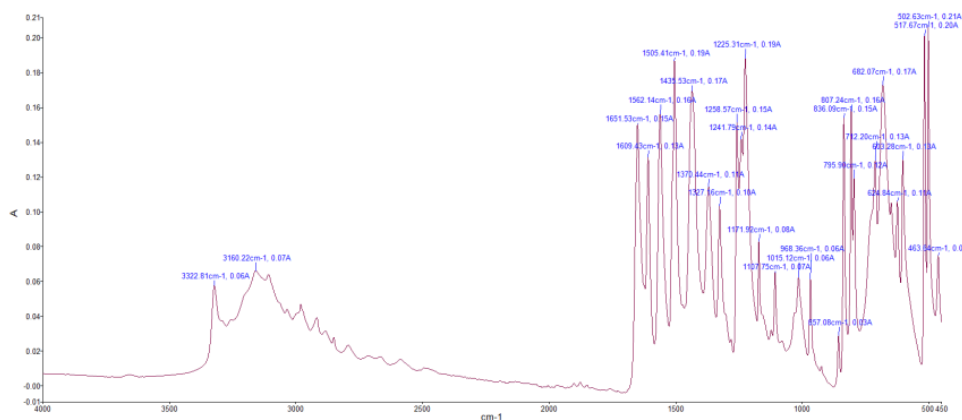


Figure 4 – Sample D – Ethyl-3-Nitrobenzoate

Sample D displayed clear aromatic C–H stretches between $3323\text{--}3160\text{ cm}^{-1}$ and a strong C=O band at 1721 cm^{-1} , typical of an ester carbonyl. Paired NO₂ asymmetric (1609 cm^{-1}) and symmetric (1351 cm^{-1}) bands confirmed a nitro substituent on the aromatic ring. Peaks at 1459 cm^{-1} (CH₂ scissoring) and 1371 cm^{-1} (CH₃ deformation) showed the presence of both CH₂ and CH₃ groups, confirming an ethyl ester rather than a methyl analogue. The C–O stretch at 1225 cm^{-1} and ring deformations between $807\text{--}603\text{ cm}^{-1}$ further supported this structure [8].

The software identified the spectrum as methyl-3-nitrobenzoate (99.2 % match), but manual inspection of the CH₂ scissoring feature confirmed the true compound as ethyl-3-nitrobenzoate—a misclassification resulting from the software library lacking the ethyl reference [9].

4. Conclusion

The ATR-FTIR analysis produced clear, reproducible spectra for all samples. Consistent cleaning, controlled pressure, and equal scan parameters ensured data accuracy. Each compound was identified using Spectrum IR's library (95–99 % confidence) and confirmed manually through functional-group analysis:

- Sample A: Guanidine nitrate – amine and nitrate absorptions.
- Sample B: Acetaminophen – phenolic O–H and amide C=O.
- Sample C: Ascorbic acid – lactone C=O and hydroxyl groups.
- Sample D: Ethyl-3-nitrobenzoate – ester carbonyl and nitro bands with ethyl confirmation.

The experiment demonstrates how procedure, consistent contact control, and manual verification underpins reliable FT-IR characterisation.

Word Count: Not including Executive Summary, Table of Contents, Headers and Figures – 1086 Words

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FINAL GRADE

82/100

GENERAL COMMENTS

Engagement with Literature 13/15

You make strong and appropriate use of literature, including vendor documentation, academic FT-IR resources, and high-quality spectral references. IEEE formatting is consistent but misses REF numbers. A few analytical points (e.g., explanation of spectral shifts, methyl-ethyl mismatch) could be more explicitly supported with citations, but overall your literature engagement is very good.

Knowledge and Understanding 17/20

You demonstrate a very strong understanding of FT-IR principles, ATR operation, and functional-group behaviour. Vibrational modes are mentioned and their importance is recognised. You correctly identify the limitations of the library and provide detailed structural reasoning. The only issue is that the introduction does not fully emphasise the true aim of the practical, developing a reproducible FT-IR method. Functional-group identification directly on the spectra would improve readability and support interpretation.

Cognitive and Intellectual Skills 17/20

Your spectral analysis is excellent. You critically examine mismatches, explain why the library misidentified methyl-3-nitrobenzoate, and use multiple diagnostic peaks effectively. Interpretation is selective and well justified. A deeper fingerprint-region analysis and slightly more discussion of peak-shape/intensity would improve this even further, but your reasoning is consistently strong.

Practical Skills 21/30

Your methodology is very detailed, with correctly stated spectral parameters, cleaning steps, apodisation, scan counts, and good attention to reproducibility. However, several required FT-IR parameters are still missing, including: atmospheric suppression, baseline/smoothing settings, ATR correction, scan speed, detector type and beam splitter, explicit QC criteria (background acceptance, S/N, re-run triggers), sample-handling detail (mass, grinding, coverage). These omissions prevent the method from being a fully complete SOP, which was the main aim of the practical.

Transferable Skills 14/15

A very well-presented report with excellent structure, clear diagrams, and polished writing. Figures and tables are consistently labelled. Minor

issues with brevity in the method section, but overall highly professional.

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Text Comment. averaged



Comment 1

have you done it?

PAGE 8



Comment 2

excellent approach

PAGE 9



Comment 3

what database did you use? our showed ~50% match with methyl 3-nitrobenzoate.

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Comment 4

what software specifically have

you used? our database found 50% match only

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is it a book?

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