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Analysis of Pharmaceutical Packaging with FTIR

This Standard Operating Procedure builds on 'Qualitative Analysis of Paracetamol with FTIR'

IRSpirit-X Series

Compact, Robust and Reliable Spectroscopic Technology - only 390mm wide!
Ideal for teaching where versatility to handling a broad range of compounds is required

Introduction

Packaging serves to protect, identify, and contain a product throughout storage and transportation until it is used. Consequently, the materials employed in pharmaceutical packaging need to have appropriate physical, chemical, and biological properties to maintain the product's integrity.

USP Chapter 661.1 outlines the standards for characterizing plastic materials utilized in pharmaceutical packaging. It mandates a series of biological, chemical, and physicochemical tests on plastics that will be in direct contact with medicinal products. One crucial aspect of these standards is measuring the infrared spectrum from 3800 to 650 cm^{-1}. In this experimental lab, you will be identifying various unknown pharmaceutical packages and identifying the specific type of plastics used by identifying characteristic peaks in the spectrum.

Materials

Tablet Packets	-	Harmful if ingested in quantity; irritant; reproductive effects
Isopropyl Alcohol	-	Harmful if ingested; irritant; reproductive effects

Safety

Avoid skin and eye contact with reagents by wearing a lab coat, gloves and safety glasses. Do not expose to a source of ignition. Avoid inhalation of IPA vapor.

Provided the recommended precautions are adopted, the risk to operators during this procedure is minimal.

Apparatus

IRSpirit-X FTIR Spectrometer

Various Tablet Packaging

Isopropyl Alcohol

IRSpirit-X Parameters

Measurement Mode:	% Transmittance
Apodisation:	Happ-Genzel
No. of Scans:	32
Resolution:	4cm^{-1}
Range (cm^{-1}):	4000-400

Preparation of samples:

Little preparation of sample is required for this experiment. A visual inspection to ensure each sample is clean on the inside and outside as both surfaces will need to be scanned.

Analysis of packaging:

1. Ensure the diamond puck is clean and free from contamination. If needed wet a clean tissue with IPA and clean the puck, allowing the solvent to evaporate.
2. Open *Spectrum* from *LabSolutions™ IR.*
3. Connect the IRSpirit to *Spectrum* using the **[Instrument]** Drop Down menu, clicking **[Connect]**.
4. Set method parameters as per *[IRSpirit-X Parameters].*
5. Insert your file name and sample name in the sample labelling section.
6. Select **[BKG Scan]** ensuring nothing is on the crystal.
7. Once completed, add a small amount of sample to cover the crystal and clamp the sample in place.
8. Select **[Sample Scan]** to produce a spectrum.
9. Repeat steps 4-8 for the rest of your samples.

Post-processing of spectra:

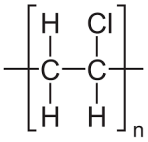
1. In the **[View]** window, select the **[Peak Pick]** option on the drop down list
2. In the **[Threshold]** box, input the %T peak threshold on the spectrum
3. Set **[Noise]** to 0.1 and press **[Calc]**
4. If you wish to select any additional peaks, check the **[Consider Manual]** box then select **[Add Peak]**
5. Use the crosshairs on the lower spectrum to add any additional peaks for characterisation.
6. Repeat steps 1-5 for the rest of your samples.
7. Use a functional group table to identify the characteristic peaks in each spectrum and identify the plastic type used in each pharmaceutical product

Model report for the identification of pharmaceutical packaging

Investigated packaging: Generic Paracetamol

Plastic type: Polyvinyl Chloride

Structure:



Sample: Own-store brand paracetamol

Paracetamol tablets containing 500 mg paracetamol per tablet

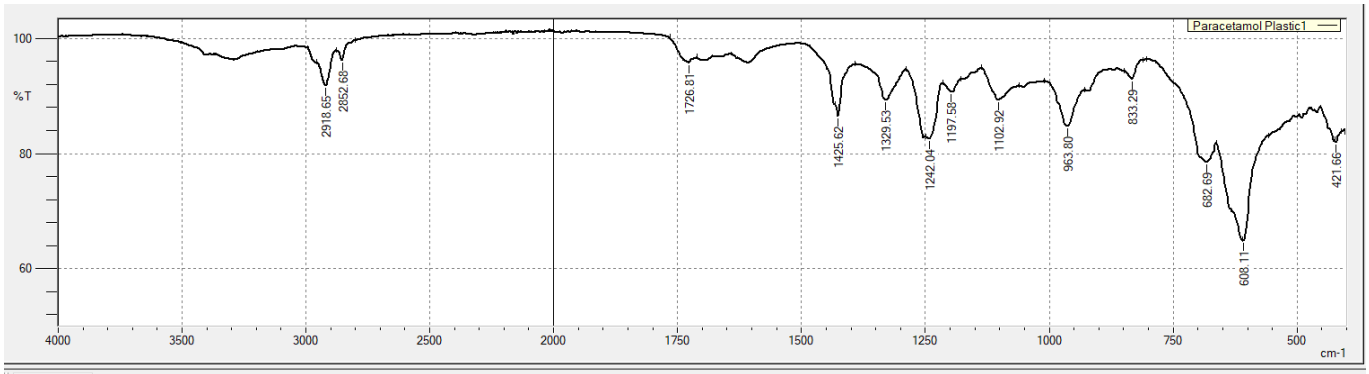
Results:

The sample packaging that was analyzed was scanned using an IRSpirit-TX with a QATR-S (Extended Range Diamond Puck) with the following parameters.

Table 1 Measurement Conditions

Measurement Mode	% Transmittance
Apodization	Blackman Harris
No. of Scans	32 scans
Resolution	4 cm^{-1}
Measurement Range cm^{-1}	4000 - 400

After the sample packaging was scanned, the [Peak Pick] function was performed to pick out the characteristic peaks in the spectrum, see below. After the peak picking was performed, a peak data table show peaks heights, %T values and peaks areas was automatically created.



Peak Pick Table | Point Pick Table | Pause Record

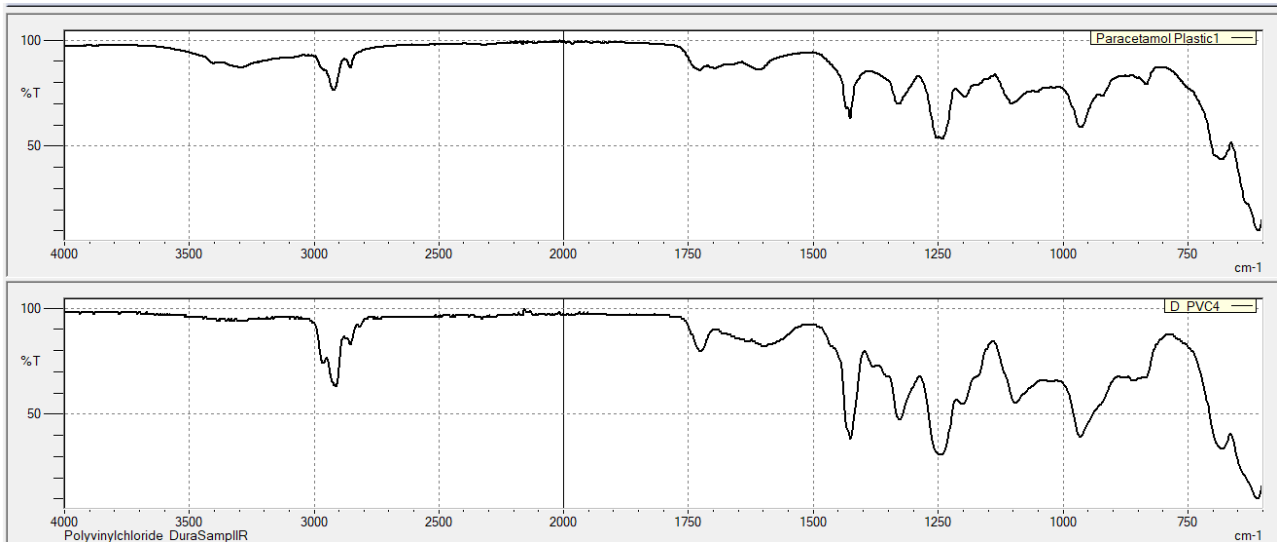
	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area	Area color	Comment
1	421.66	82.01	0.69	424.53	411.62	221.824	4.117	Auto	
2	608.11	64.84	1.20	610.98	557.91	1250.914	-124.002	Auto	
3	682.69	78.52	5.30	804.60	662.61	1626.002	70.991	Auto	
4	833.29	93.00	2.14	849.06	811.77	205.568	30.095	Auto	
5	963.80	84.76	6.96	1009.70	927.94	898.679	229.718	Auto	
6	1102.92	89.33	4.16	1137.34	1061.33	656.438	150.139	Auto	
7	1197.58	90.71	1.85	1214.79	1177.50	310.155	33.801	Auto	
8	1242.04	82.58	2.28	1249.21	1214.79	473.769	46.041	Auto	
9	1329.53	89.29	5.68	1391.20	1289.37	702.232	200.622	Auto	
10	1425.62	86.56	2.97	1431.36	1391.20	299.367	-20.261	Auto	
11	1726.81	95.78	2.04	1765.53	1711.03	147.052	68.418	Auto	
12	2852.68	96.23	2.31	2878.49	2799.61	134.977	44.351	Auto	
13	2918.65	91.88	5.00	2957.38	2878.49	423.500	178.640	Auto	

Now the peaks are labelled the correlate the wavenumber to a characteristic group table to identify the likely plastic component in the pharmaceutical packaging.

Wavenumber cm^{-1}	**Assignment**
2960	Stretch C-H of CHCl
2918	Stretch C-H of CH_2
1425	Wagging of CH_2
1329 and 1242	Wagging/Rotation C-H of CHCl
1102	Stretching C-C
963	Rocking CH_2
682 and 608	Stretching C-Cl

From the functional group assignment of the peaks present in the spectrum, it is likely that the plastic packaging is predominantly made from Polyvinyl Chloride (PVC).

After peak picking, a spectral search was performed to verify the packaging was PVC. Below are the results from the spectral search which verifies the packaging used in paracetamol blister packs is PVC.



	Score	Library	Name	
1	990	191 - ATR-Polymer2	D_PVC4	Polyvinylchloride DuraSamplIR

Conclusion:

A random packet of paracetamol from a selection of pharmaceutical packaging was selected for determination of the material used using manual peak identification and verifying the determination using the spectral search function. The spectral search show an excellent match with Polyvinyl Chloride, with a score of 990/1000, which verifies the determination by peak identification using the Peak Pick function in LabSolutions IR.