

Structural Study of Citric Esters by IR Spectroscopy

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Abstract—In this article, we determined by IR spectroscopy the composition of the following citric esters: ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1), n-propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1), and n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2). The absorption spectrum of ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) is between the wave numbers 3481cm⁻¹ and 829cm⁻¹. The absorption spectrum of n-propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) is between the wave numbers 3482cm⁻¹ and 829cm⁻¹. The absorption spectrum of n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (1) is between the wave numbers 3482cm⁻¹ and 829cm⁻¹. The absorption spectrum of n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl citrate (2) is between the wave numbers 3488cm⁻¹ and 829cm⁻¹. IR spectra for citric esters were obtained on a Jasco 430 FT-IR spectrophotometer with a resolution of 1 cm⁻¹. Which is valuable applications for the development of plasticizers and biodegradable polymers.

Keywords— Esters, citrus, spectroscopy, study.

I. INTRODUCTION

IR spectrometry involves studying the absorption of infrared (IR) radiation, which can cause transitional vibrations in the analyzed molecule. Molecules are composed of atoms joined by chemical bonds. The movement of atoms and chemical bonds causes characteristic vibrations to appear as a result of the absorption of IR radiation. There are two types of vibration: stretching and bending. Different functional groups present in a molecule absorb IR radiation at certain characteristic frequencies. Therefore, certain characteristic values are given for the identified peak. Thus, the IR spectrum of a certain substance can be considered as a fingerprint of it and can help identify it [1-8].

Infrared spectroscopy (IR spectroscopy or vibrational spectroscopy) is the estimation of the interaction of infrared radiation with matter by assimilation, emanation, or reflection. It is utilized to think about and distinguish chemical substances or utilitarian bunches in strong, fluid, or vaporous shapes. It can be utilized to characterize unused materials or recognize and confirm known and obscure tests. The strategy or procedure of infrared spectroscopy is conducted with an instrument called an infrared spectrometer (or spectrophotometer) which produces an infrared range. An IR range can be visualized in a chart of infrared light absorbance (or transmittance) on the vertical hub vs. recurrence, wavenumber or wavelength on the flat pivot. Ordinary units of wavenumber utilized in IR spectra are complementary centimeters, with the image cm⁻¹.

Units of IR wavelength are commonly given in micrometers (once in the past called "microns"), image μ m, which are related to the wavenumber in a corresponding way. A common research facility instrument that employments this

procedure could be a Fourier change infrared (FTIR) spectrometer.

Two-dimensional IR is additionally conceivable as talked about underneath. The infrared parcel of the electromagnetic range is ordinarily partitioned into three districts; the close-, mid- and far-infrared, named for their connection to the unmistakable range.

The higher-energy near-IR, roughly $14,000-4,000 \text{ cm}^{-1}$ (0.7–2.5 µm wavelength) can energize hint or combination modes of atomic vibrations. The mid-infrared, which is approximately $4,000-400 \text{ cm}^{-1}$ (2.5–25 µm), is primarily used to study the fundamental vibrations and the associated rotational-vibrational structure.

The far-infrared, around $400-10 \text{ cm}^{-1} (25-1,000 \text{ }\mu\text{m})$ has moo vitality and may be utilized for rotational spectroscopy and moo recurrence vibrations. The locale from 2–130 cm⁻¹, bordering the microwave locale, is considered the terahertz locale and may test intermolecular vibrations.[1] The names and classifications of these subregions are traditions, and are as it were freely based on the relative atomic or electromagnetic properties [8-19].

II. MATERIAL AND METHODS

IR spectra were obtained on a Jasco 430 FT-IR spectrophotometer. Samples were prepared in KBr pellets or in film between two KBr panes. A Jasco 430 FT-IR spectrophotometer with a resolution of 1 cm⁻¹ was used to monitor FT-IR reactions in temperature-controlled cuvettes with silicon panes that were 0.137 mm thick (fig.1).



Fig. 1. Jasco 430 FT-IR spectrophotometer

Synthesis of citric esters

Ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) are obtained by the synthesis of 0.5 moles of citric acid, 1.1 moles of ethyl alcohol and 0.008 moles of p-toluene sulfonic acid. In stage I at temperature = $100 - 105^{\circ}$ C, for 10 - 12 hours and toluene = 115 ml. In the second stage, 0.52 moles of 2-(pnonyl-phenoxy) ethanol are added at a temperature between 120° C - 135° C, for 8 - 10 hours.





n-Propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) are obtained by the synthesis of 0.5 moles of citric acid, 1.1 moles of n-propyl alcohol and 0.008 moles of p-toluene sulfonic acid. In stage I at temperature = $100 - 105^{\circ}$ C, for 10 - 12 hours and toluene = 115 ml. In the second stage, 0.52 moles of 2-(p-nonyl-phenoxy) ethanol are added at a temperature between 120° C - 135° C, for 8 - 10 hours.



n-Propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2) are obtained by the synthesis of 0.5 moles of citric acid, 0.65 moles of n-propyl alcohol and 0.005 moles of p-toluene sulfonic acid. In stage I at temperature = $100 - 105^{\circ}$ C, for 10 - 12 hours and toluene = 115 ml. In the second stage, 1.050

moles of 2-(p-nonyl-phenoxy) ethanol are added at a temperature between 120^{0} C - 135^{0} C, for 8 - 10 hours.



III. RESULTS AND DISCUSSION

In figures 2-4 the absorption spectra for the following compounds are presented: ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1), n-propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) and n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2) in this spectrum, the fundamental absorption bands, located in the spectral range from 3500 cm^{-1} to 829 cm^{-1} , are clearly evident.

Figure 2 presents the IR spectrum of ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1).



Fig. 2. Spectrul IR al citratului de etil (2) și de 2-(p-nonil-fenoxi) etil (1)

TABLE 1. Peak assignments in the FTIR spectrum of ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1)				
Wave number, cm ⁻¹	Functional grouping	Vibration mode		
3481	OH	a vera ge		
2960	CH ₃ alifatic	asymmetric		
2931	CH ₃ aromatic	asymmetric		
2872	CH ₃ aromatic	symmetric		
1741	C=O saturated esters	intensity		
1511	${\cal V}_{sch.arom}$ + δ CH _{intensity}	intensity		
1246	$\mathcal{V}^{s}_{\text{C-O(intensity)}} + \gamma^{as}_{Ar-C-CH_{2}(inensity)}$	intensity		
1186	Benzene 1,4,	intensity		
829	Benzene 1,4,	average		



As can be seen in the spectrum, 22 peaks appear for ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1). The most important ones are listed in Table 1.

Table 1 shows the peak assignments for ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1).

At the wavelength of 3481 cm⁻¹ the OH functional group corresponding to medie. At the wavelength of 2960 cm⁻¹ the CH₃ functional group corresponding to asymmetric alifatic appears. At the wavelength of 2931 cm⁻¹ and 2872 the CH₃ functional group corresponding to asymmetric aromatic appears. The C=O functional group associated with the intensity of saturated esters has a wavelength of 1741 cm⁻¹. The benzen di 1,4 functional group corresponding to intensity and average at the wavelength of 1186 and 829 cm⁻¹.

As can be seen in the spectrum, 22 peaks appear for npropyl citrate (2) and 2-(p-nonyl-phenoxy)ethyl (1). The most important ones are listed in Table 2.

Figure 3 shows the IR spectrum of n-propyl citrate (2) and 2-(p-nonyl-phenoxy)ethyl (1).

Table 2 shows the peak assignments for n-propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1).



Fig. 3. IR spectrum of n-propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1)

Wave number, cm ⁻¹	Functional grouping	Vibration mode
3482	OH	medium
2962	CH ₃ alifatic	asymmetric
2875	CH ₃ aromatic	asymmetric
1740	C=O saturated esters	intensity
1511	$v_{sch.arom}$ + δ CH _{intensity}	intensity
1248	$\mathcal{V}^{s}_{\text{C-O(intensity)}} + \gamma^{as}_{Ar-C-CH_{2}(inensity)}$	intensity
1186	Benzene di 1,4,	intensity
829	Benzene di 1,4,	average

At the wavelength of 3482 cm^{-1} the OH functional group corresponding to the medium. At the wavelength of 2962 cm^{-1} the CH₃ functional group corresponding to the asymmetric aliphatic appears. At the wavelength of 2875 cm^{-1} and 2872 the CH₃ functional group corresponding to the asymmetric aromatic appears. At the wavelength of 1740 cm^{-1} the C=O functional group corresponding to the intense saturated esters. At the wavelength of 1186 and 829 cm^{-1} the 1,4-benzene functional group corresponding to the intense and medium.

Figure 4 shows the IR spectrum of n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2).

As can be seen in the spectrum, 21 peaks appear for npropyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2). The most important ones are listed in Table 3.

At the wavelength of 3488 cm^{-1} the OH functional group corresponding to the average. At the wavelength of 2960 cm^{-1} the CH₃ functional group corresponding to the asymmetric aliphatic appears. At the wavelength of 2873 cm^{-1} and 2872the CH₃ functional group corresponding to the asymmetric aromatic appears. At the wavelength of 1742 cm^{-1} the C=O functional group corresponding to the intense saturated esters. At the wavelength of 1186 and 829 cm^{-1} the 1,4-benzene functional group corresponding to the intense and average.

Table 3 shows the assignment of the peaks for n-propyl

citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2).



Fig. 4. IR spectrum of n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl citrate (2)

 TABLE 3. Peak assignment in the FTIR spectrum of n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl citrate (2)

 Works number on 1

Wave number, cm-1	Functional grouping	Vibration mode
3488	OH	medium
2960	CH ₃ a lifatic	asymmetric
2873	CH ₃ aromatic	asymmetric
1742	C=O saturated esters	intensity
1511	${\cal V}_{sch.arom}$ + δ CH _{intensity}	intensity
1248	$V^{s}_{\text{C-O(intensity)}} + \gamma^{as}_{Ar-C-CH_{2}(inensity)}$	intensity
1186	Benzene di 1,4,	intensity
829	Benzene di 1,4,	average

IV. CONCLUSIONS

The spectra of the following citric esters: ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1), n-propyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) and n-propyl citrate (1) and 2-(p-nonyl-phenoxy) ethyl (2). The absorption spectra of ethyl citrate (2) and 2-(p-nonyl-phenoxy) ethyl (1) contain the following functional groups: CH₃, C=O, 1,4-benzene di. The spectra were determined with a Jasco 430 FT-IR spectrophotometer.

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