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Appraisal on spectral methods in chemical research

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Abstract : *In this paper we discussed about the basic fundamentals of IR, proton NMR, C13 NMR, scope and need of usage of spectral methods with their data and some examples. In the process of modern drug research, the new methods and technologies which can detect drug molecules' chemical composition, structure and interaction with biomolecules are always the key scientific problems people care about. Spectra (including IR, UV and NMR) are the most common analytical methods, of which NMR can obtain detailed parameter about the nucleus of organic molecules through researching the laws of nuclear transition in the impact of surrounding chemical environment. The latest developments in vibrational spectroscopic tools used to determine the functional groups present in various chemical compounds. Various concepts for the enhancement of Infrared spectroscopic techniques, including the principles of Attenuated Total Reflection Infrared (ATR-IR), (phase-modulated) Infrared Reflection Absorption Spectroscopy (IRRAS/PM-IRRAS), and Surface Enhanced Infrared Reflection Absorption Spectroscopy (SEIRAS).*

Keywords*: IR, NMR, Spectroscopy, Research*

INTRODUCTION

In the process of modern drug research, the new methods and technologies which can detect drug molecules' chemical composition, structure and interaction with biomolecules are always the key scientific problems people care about. Spectra (including IR, UV and NMR) are the most common analytical methods, of which NMR can obtain detailed parameter about the nucleus of organic molecules through researching the laws of nuclear transition in the impact of surrounding chemical environment. The parameter contains rich information about the chemical composition, structure and interaction with other molecules of organic molecules. In many complex environments, such as liquid, solid or gas state, even biological in situ environment, NMR can provide molecules' chemical composition, atomic-resolution three-dimensional structure, information of interaction with each other and dynamic process. In recent years, the applications of nuclear magnetic resonance spectrum in drug research and development are more and more widespread.

Infrared spectroscopy (IR spectroscopy or Vibrational Spectroscopy)

Infrared spectroscopy (IR spectroscopy or Vibrational Spectroscopy) is the spectroscopy that deals with the infrared region of the electromagnetic spectrum, that is light with a longer wavelength and lower frequency than visible light. It covers a range of techniques, mostly based on absorption spectroscopy. As with all spectroscopic techniques, it can be used to identify and study chemicals. For a given sample which may be solid,

liquid, or gaseous, the method or technique of infrared spectroscopy uses an instrument called an infrared spectrometer (or spectrophotometer) to produce an infrared spectrum. A basic IR spectrum is essentially a graph of infrared light absorbance (or transmittance) on the vertical axis vs. frequency or wavelength on the horizontal axis. Typical units of frequency used in IR spectra are reciprocal centimeters (sometimes called wave numbers), with the symbol cm^{-1}. Units of IR wavelength are commonly given in micrometers (formerly called "microns"), symbol μm, which are related to wave numbers in a reciprocal way. A common laboratory instrument that uses this technique is a Fourier transform infrared (FTIR) spectrometer.

The term "infra red" covers the range of the electromagnetic spectrum between 0.78 and 1000 mm. In the context of infra red spectroscopy, wavelength is measured in "wavenumbers", which have the units cm^{-1}.

wavenumber = 1 / wavelength in centimeters. It is useful to divide the infra red region into three sections; *near*, *mid* and *far* infra red;

Region	**Wavelength range** (mm)	**Wavenumber range** (cm^{-1})
Near	0.78 - 2.5	12800 - 4000
Middle	2.5 - 50	4000 - 200
Far	50 -1000	200 - 10

The most useful I.R. region lies between 4000 - 670cm^{-1}. (25–1000 μm)

Characteristic IR Absorption Frequencies of Organic Functional Groups			
Functional Group	**Type of Vibration**	**Characteristic Absorptions (cm-1)**	**Intensity**
Alcohol			
O-H	(stretch, H-bonded)	3200-3600	strong, broad
O-H	(stretch, free)	3500-3700	strong, sharp
C-O	(stretch)	1050-1150	strong
Alkane			
C-H	stretch	2850-3000	strong
-C-H	bending	1350-1480	variable
Alkene			
=C-H	stretch	3010-3100	medium
=C-H	bending	675-1000	strong
C=C	stretch	1620-1680	variable
Alkyl			

Halide			
C-F	stretch	1000-1400	strong
C-Cl	stretch	600-800	strong
C-Br	stretch	500-600	strong
C-I	stretch	500	strong
Alkyne			
C-H	stretch	3300	strong,sharp
-C≡C-	stretch	2100-2260	variable, not present in symmetrical alkynes
Amine			
N-H	stretch	3300-3500	medium (primary amines have two bands; secondary have one band, often very weak)
C-N	stretch	1080-1360	medium-weak
N-H	bending	1600	medium
Aromatic			
C-H	stretch	3000-3100	medium
C=C	stretch	1400-1600	medium-weak, multiple bands
Analysis of C-H out-of-plane bending can often distinguish substitution patterns			
Carbonyl	Detailed Information on Carbonyl IR		
C=O	stretch	1670-1820	strong
(conjugation moves absorptions to lower wave numbers)			
Ether			
C-O	stretch	1000-1300 (1070-1150)	strong
Nitrile			
CN	stretch	2210-2260	medium
Nitro			
N-O	stretch	1515-1560 & 1345-1385	strong, two bands

IR Absorption Frequencies of Functional Groups Containing a Carbonyl (C=O)			
Functional Group	**Type of Vibration**	**Characteristic Absorptions (cm-1)**	**Intensity**
Carbonyl			
C=O	stretch	1670-1820	strong
(conjugation moves absorptions to lower wave numbers)			
Acid			
C=O	stretch	1700-1725	strong
O-H	stretch	2500-3300	strong, very broad
C-O	stretch	1210-1320	strong

C-13 NMR spectroscopy

The major isotope of carbon (^{12}C) has no spin, this option seems unrealistic. Fortunately, 1.1% of elemental carbon is the ^{13}C isotope, which has a spin I = 1/2, so in principle it should be possible to conduct a carbon nmr experiment. It is worth noting here, that if much higher abundances of ^{13}C were naturally present in all carbon compounds, proton nmr would become much more complicated due to large one-bond coupling of ^{13}C and ^{1}H.

Many obstacles needed to be overcome before carbon nmr emerged as a routine tool :

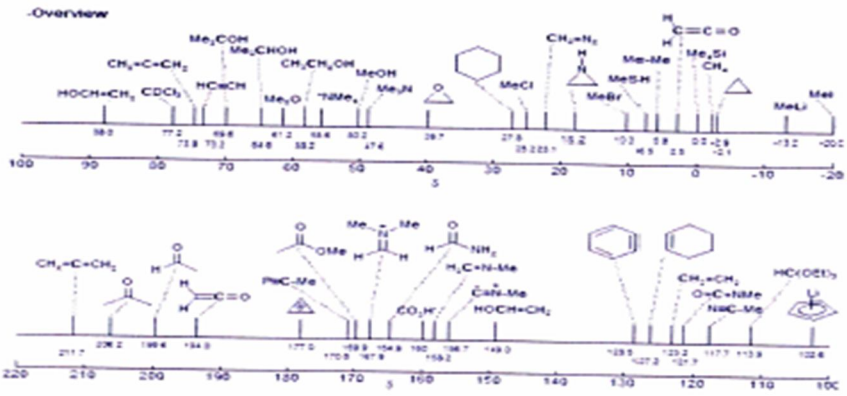
i) As noted, the abundance of ^{13}C in a sample is very low (1.1%), so higher sample concentrations are needed.

ii) The ^{13}C nucleus is over fifty times less sensitive than a proton in the nmr experiment, adding to the previous difficulty.

iii) Hydrogen atoms bonded to a ^{13}C atom split its nmr signal by 130 to 270 Hz, further complicating the nmr spectrum.

The most important operational technique that has led to successful and routine ^{13}C nmr spectroscopy is the use of high-field pulse technology coupled with broad-band heteronuclear decoupling of all protons. The results of repeated pulse sequences are accumulated to provide improved signal strength. Also, for reasons that go beyond the present treatment, the decoupling irradiation enhances the sensitivity of carbon nuclei bonded to hydrogen. When acquired in this manner, the carbon nmr spectrum of a compound displays a single sharp signal for each structurally distinct carbon atom in a molecule (remember, the proton couplings have been removed).

C-13 Chemical Shifts



Reference

1.http://chemwiki.ucdavis.edu/Physical_Chemistry/Spectroscopy/Vibrational_Spectroscopy/Infrared_Spectroscopy/

2. http://www2.ups.edu/faculty/hanson/Spectroscopy/IR/IRfrequencies.html

3. http://www.chem.wisc.edu/areas/reich/handouts/nmr-h/hdata.html

4. http://www.chem.ucalgary.ca/courses/351/Carey5th/Ch13/ch13-cnmr-2.html