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Simulation Tool to Determine Presence of Impurities in an Organic Compound Using NMR Spectroscopy

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Abstract: ***The popularity of using NMR Spectroscopy in elucidation of organic compounds, medical pharmacology and natural products has driven development of an array of NMR Spectral analysis of tools and databases. NMR Spectroscopy determines the physical and chemical properties of atoms or the molecules in which they are contained. It relies on the nuclear magnetic resonance and can provide detailed information about the structure, dynamics, reaction state, and chemical environment of molecules. The physical and chemical properties can be determined through NMR spectra. NMR Spectra is the set of characteristic peaks of a compound which are unique, well-resolved, and analytically track able. The paper outlines the development of simulation tool to obtain the NMR Spectra of the compound resulted after a reaction is performed. The position of the peaks is obtained through chemical shift of that compound. We compare this resultant NMR Spectra with theoretical characteristic peaks of that compound to determine the presence of impurities.***

Keywords: NMR Spectrum, Chemical shift, Fourier transform NMR

1. Introduction

Nuclear Magnetic Resonance Spectroscopy or most commonly known as NMR spectroscopy is a technique based on the absorption of electromagnetic radiation in the radio frequency region 4 to 900 MHz by the nuclei of the atoms.
NMR is a property of the nucleus of an atom concerned with what is known as spin of a nucleus[1].

The spin of the nucleus generates the magnetic field. When there is no external magnetic field, the nuclear spins are in the random direction. When an external magnetic field is applied the electrons align themselves either in the direction of magnetic field or opposite to it. If an external magnetic field is applied, energy is transferred from ground state to excited state. When spin returns, the absorbed radio frequency energy is emitted at the same frequency level. This emitted radio frequency gives the NMR spectrum of the nucleus. [3] It is given by the formula,

$$v = \frac{\gamma B}{2\pi} \quad (1)$$

NMR spectrum is the plot of intensity of NMR signals VS magnetic field (frequency) in reference to TMS (tetramethylsilane).

Different nuclei absorb the electromagnetic radiation at different wavelengths. These nuclei will resonate at different frequencies depending on their chemical and electronic environment. The position and pattern of the spectra gives the information about the chemical environment. Hence to identify the presence of impurities we compare the standard spectral data with the peaks obtained for the given substance.

Due to the presence of impurities, there are variations observed in the NMR spectrum of the same nucleus. This happens because of the variation in the electron distribution. This variation is called chemical shift[2]. Chemical shift is the resonant frequency relative to the standard in a magnetic field. Chemical shift is obtained by [3],

Chemical shift,

$$\partial = \frac{\text{frequency of sample-reference frequency}}{\text{spectrometer frequency}} * 10^6 \text{ppm} \quad (2)$$

Different compounds would give different chemical shifts even if they are of same kind of nucleus.

The objective of our tool is to provide a method to determine the presence of impurities in a given compound by comparing the chemical shift value and the spectral data with the theoretical chemical shift value and spectral data of that compound. Depending on this comparison, the result is deduced.

2. Method

The flow chart shows the procedure to determine the presence of impurities and possible purifying mechanisms in the given substance using the simulation tool. First, the compound, Fourier transform parameters, compound

frequency and NMR spectrometer frequency has to be given as input to the simulation tool. After the calculation of chemical shifts, spectral data of the sample input and theoretical data is displayed on the monitor screen. The spectral lines of theoretical data and sample are compared. After comparison, a conclusion is drawn regarding the presence of impurities. If the spectral lines are same then we can say "NO IMPURITIES" otherwise we say "IMPURITIES PRESENT". If impurities are present, then different possible purifying mechanisms for the compound are predicted with respect to the data that is stored in the database.

3. Observation

An example can be considered to observe how the peaks can be obtained

3.1 Case 1) Ethyl Bromide(CH_3CH_2Br)

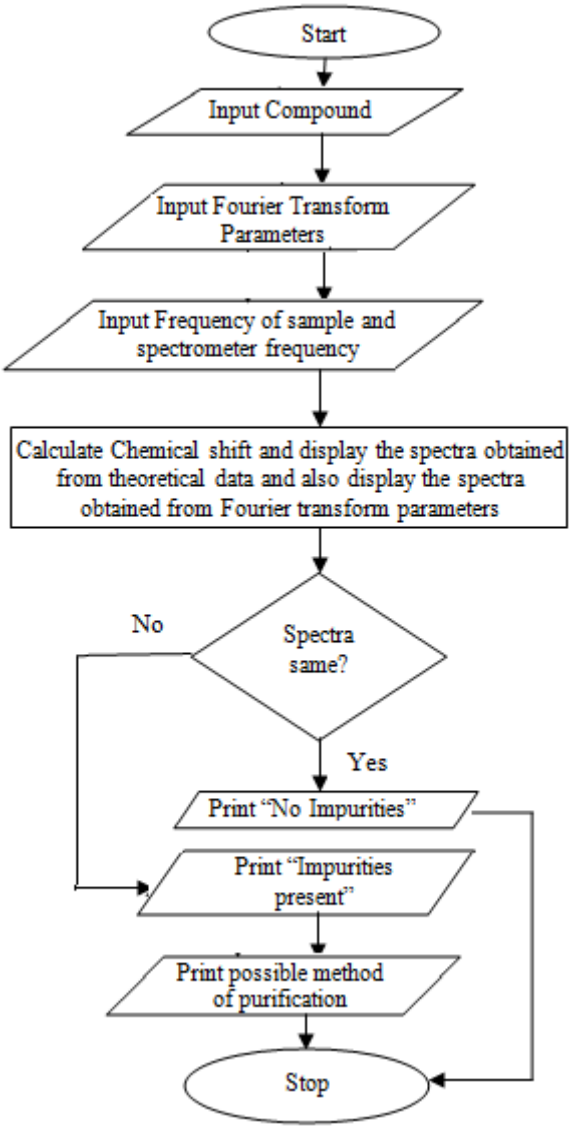

Figure 1: NMR Spectra of Ethyl Bromide

In this compound, we have two different types of chemical environment. The number of peaks can be observed by the N+1 rule. Here N is the number of neighbours of the atom[4].

Let the two types of hydrogen be a and b- type.
N value for a-type hydrogen = 3
Therefore, number of peaks for a-type hydrogen is 4
Similarly, N value for b-type hydrogen = 2
Therefore, number of peaks for b-type hydrogen is 3
The peaks of Ethyl bromide can be obtained as shown above in Figure 1.

3.2 Case 2) Ethanol (CH_3CH_2OH)

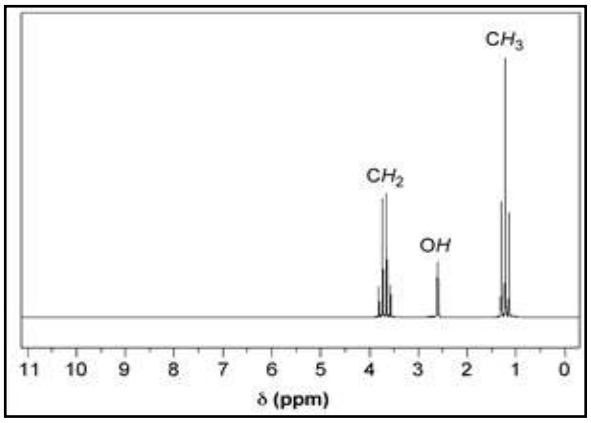

Figure 2: NMR Spectra of Ethanol

In this compound, we have three different types of hydrogen. Let the types of hydrogen be a, b and c-type.
N value for a- type hydrogen = 3
Number of peaks of a-type hydrogen is 4
N value for b- type hydrogen = 1
Number of peaks for b- type hydrogen is 2
N value for c- type hydrogen = 2
Number of peaks for c- type hydrogen is 3
The peaks of Ethanol are then obtained as shown above in Figure 2.

4. Applications

1) For the impurity profile of pharmaceuticals: Various regulatory authorities such as the International Conference on Harmonization (ICH), the United States Food and Drug administration (FDA), and the Canadian Drug and Health Agency (CDHA) are emphasizing on the purity requirements and the identification of impurities in Active Pharmaceutical Ingredients (APIs) [5].
2) NMR chemical shifts of trace impurities present in industrially preferred solvents used in process and green chemistry
3) Agrochemical development and production

5. Acknowledgement

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