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ANALYTICAL INVESTIGATION OF METHYL TETRA BUTYL ETHER AND BUTYL ALCOHOL USING PROTON NUCLEAR MAGNETIC RESONANCE (¹HNMR) SPECTROSCOPY

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ABSTRACT

Proton nuclear magnetic resonance (¹H-NMR) spectroscopy is a powerful and **non-destructive analytical technique** widely employed for the **structural characterization** of organic compounds. In the present study, a comparative ¹H-NMR based analytical investigation of **methyl tert-butyl ether (MTBE)** and **butanol** has been carried out to evaluate the influence of functional groups and molecular **symmetry** on proton environments. The spectra were recorded in **deuterated chloroform ($CDCl_3$)** using **tetramethylsilane (TMS)** as an internal reference. MTBE exhibited a **simple spectral pattern** characterized by **singlet signals** corresponding to methoxy and tert-butyl protons, reflecting **molecular symmetry** and equivalent proton environments. In contrast, butanol displayed multiple resonance signals with characteristic splitting patterns due to nonequivalent methyl, methylene, and hydroxyl protons. Chemical shift values, **signal multiplicity**, and integration ratios were analyzed and found to be consistent with the expected molecular structures. The study demonstrates the **effectiveness** of ¹H-NMR spectroscopy for **distinguishing ethers and alcohols** and confirms its **reliability** as an **analytical tool** for **structural elucidation** and purity assessment of organic compounds.

KEYWORDS: Nuclear Magnetic Resonance Spectroscopy, Methyl Tert-Butyl Ether, Butanol, Proton NMR, Spectral Analysis.

1. INTRODUCTION

Proton nuclear magnetic resonance (¹H-NMR) spectroscopy has emerged as an **indispensable analytical tool** in modern organic chemistry, pharmaceutical analysis, and structural biology due to its ability to provide detailed information about molecular structure, proton environments, and functional groups without destroying the sample (Silverstein et al., 2015). The technique exploits the **magnetic properties** of hydrogen nuclei, which possess a **nuclear spin** of ½, making them sensitive to external magnetic fields and radiofrequency radiation (Pavia et al., 2014). When placed in a strong magnetic field, hydrogen nuclei align either with or against the field, creating distinct **energy states**. The absorption of **radiofrequency energy** causes transitions between these states, producing signals that are characteristic of the chemical environment surrounding each proton (Claridge, 2016).

The fundamental parameters obtained from ¹H-NMR spectra-**chemical shift** (δ), **coupling constants** (J), **integration ratios**, and signal multiplicity-collectively enable comprehensive **structural elucidation** of organic compounds (Friebolin, 2011). Chemical shifts, measured in parts per million (ppm) relative to an internal standard such as **tetramethylsilane (TMS)**, reflect the **electronic environment** of protons and are influenced by factors including electronegativity of neighboring atoms, hybridization states, and resonance effects (Keeler, 2010). Coupling patterns arise from spin-spin interactions between neighboring non-equivalent protons, following the **n+1 rule** for simple first-order

systems, where n represents the number of equivalent neighboring protons (Lambert & Mazzola, 2019). Integration ratios provide quantitative information about the relative number of protons giving rise to each signal, enabling molecular formula confirmation and purity assessment (Günther, 2013).

Since hydrogen atoms are ubiquitous in organic molecules, proton NMR offers exceptionally high **sensitivity** and broad **applicability** across diverse compound classes (Jacobsen, 2007). The technique has proven particularly valuable for distinguishing between **structural isomers**, identifying **functional groups**, monitoring reaction progress, and assessing compound **purity** in both academic research and industrial quality control settings (Bharti & Roy, 2012). Modern NMR instrumentation, operating at field strengths ranging from 400 to 1000 MHz, provides enhanced resolution and sensitivity, enabling the detection and characterization of increasingly complex molecular systems (Berger & Braun, 2004).

Methyl tert-butyl ether (MTBE) and butanol represent two important classes of oxygen-containing organic compounds-ethers and alcohols-that exhibit contrasting structural features and find widespread industrial applications. MTBE, with the molecular formula $C_5H_{12}O$, is a **tertiary ether** characterized by a methoxy group (-OCH_3) attached to a quaternary carbon center bearing three methyl groups (Squillace et al., 1997). This structural arrangement imparts high **molecular symmetry** and results in **magnetically equivalent** proton environments, making MTBE an ideal model compound for studying the NMR characteristics of symmetrical ethers (Johnson & Klopfenstein, 2000). Historically, MTBE gained prominence as a **fuel oxygenate** additive designed to enhance octane ratings and reduce carbon monoxide emissions from automotive engines (Happel et al., 1998). However, its environmental persistence and groundwater contamination concerns have sparked extensive research into its detection, degradation, and replacement alternatives (Deeb et al., 2003).

Butanol, or n-butanol (1-butanol), is a **primary alcohol** with the molecular formula $C_4H_{10}O$, featuring a four-carbon straight chain terminated by a hydroxyl group (Dürre, 2007). Unlike MTBE, butanol possesses multiple **non-equivalent proton environments**, including terminal methyl (-CH_3), internal methylene (-CH_2-), and hydroxyl (-OH) groups, each exhibiting distinct **chemical shifts** and **coupling patterns** (Haas & Jin, 2011). This structural complexity makes butanol an excellent representative for studying the NMR behavior of primary alcohols and understanding the effects of proton-proton coupling in aliphatic systems (Green & Pettit, 2009). Butanol serves as an important industrial solvent, chemical intermediate, and potential **biofuel**, with microbial fermentation routes offering sustainable production pathways (Lee et al., 2008; Qureshi & Blaschek, 2001).

The selection of MTBE and butanol for comparative ^{1}H-NMR analysis is strategically motivated by their contrasting **molecular features** and the pedagogical value of examining both simple and complex **spectral patterns** within a single study (Crews et al., 2009). MTBE's high symmetry and equivalent proton sets produce a simplified spectrum dominated by **singlet resonances**, facilitating straightforward peak assignment and serving as an introduction to basic NMR interpretation (Bruch & Gokel, 2001). In contrast, butanol's asymmetric structure generates a rich spectrum displaying **multiplets**, triplets, and **coupling interactions** that illustrate more advanced concepts in NMR spectroscopy (Friebolin, 2011). Furthermore, both compounds are readily available in high purity, air-stable, and safe to handle under standard laboratory conditions, making them practical choices for educational and analytical investigations (Pavia et al., 2014).

Previous studies have extensively characterized MTBE and butanol using various spectroscopic techniques, including **infrared spectroscopy**, mass spectrometry, and ^{13}C-NMR (Silverstein et al., 2015; McLafferty & Turecek, 1993). However, ^{1}H-NMR remains the most informative single technique for rapid **structural confirmation** due to its sensitivity and the wealth of structural information encoded in proton spectra (Jacobsen, 2007). Comparative investigations of ethers and alcohols have demonstrated that **functional group identity** profoundly influences proton chemical shifts, with hydroxyl groups causing significant downfield shifts in adjacent protons due to the **electronegativity** of oxygen and **hydrogen bonding** effects (Pretsch et al., 2009). Additionally, the presence or absence of coupling partners determines signal multiplicity, with isolated proton groups appearing as singlets and coupled systems displaying characteristic splitting patterns governed by the magnitude of **coupling constants** (Keeler, 2010).

2. METHODOLOGY

2.1 Materials

High-purity reagents and solvents were employed to ensure reliable spectral acquisition and accurate data interpretation. Methyl tert-butyl ether (MTBE, $C_5H_{12}O$, molecular weight 88.15 g/mol, analytical grade, ≥99.8% purity) was obtained from Sigma-Aldrich and used without further purification (Product No. 306975, CAS 1634-04-4). n-Butanol (1-butanol, $C_4H_{10}O$, molecular weight 74.12 g/mol, analytical grade, ≥99.5% purity) was purchased from Merck KGaA (Product No. 1.00988, CAS 71-36-3) and similarly used as received. Deuterated chloroform ($CDCl_3$, 99.8% D, containing 0.03% v/v tetramethylsilane as internal reference) was procured from Cambridge Isotope Laboratories, Inc.

- Methyl tert-butyl ether (analytical grade)
- Butanol (analytical grade)
- Deuterated chloroform (CDCl_3)
- Tetramethylsilane (TMS)

2.2 Instrumentation

Proton nuclear magnetic resonance (^{1}H-NMR) spectra were acquired using a Bruker Avance III HD 400 MHz NMR spectrometer equipped with a 5 mm broadband observe (BBO) SmartProbe capable of detecting ^{1}H nuclei at 400.13 MHz and ^{13}C nuclei at 100.62 MHz (Bruker BioSpin, Rheinstetten, Germany). The spectrometer featured an UltraStabilized™ magnet system with active shielding, ensuring high field homogeneity and long-term stability.

2.3 Experimental Procedures

2.3.1 Sample Preparation

NMR samples were prepared using scrupulously clean, dry 5 mm outer diameter borosilicate glass NMR tubes. For each compound, approximately 8-10 mg was accurately weighed and quantitatively transferred into a clean 1.5 mL microcentrifuge tube. $CDCl_3$ (0.7 mL) containing TMS (0.03% v/v) was added, and the mixture was vortexed for 30 seconds to ensure complete dissolution and homogeneity.

2.3.2 Spectral Acquisition Parameters

All ^{1}H-NMR spectra were acquired using standardized parameters. A 90-degree radiofrequency pulse was employed for **excitation**. The spectral width was set to 8,012.82 Hz (approximately 20 ppm at 400 MHz), ensuring complete capture of all proton resonances. Time domain data were collected using 65,536 (64K) complex data points, providing a **digital resolution** of approximately 0.12 Hz per point after Fourier transformation. The relaxation delay was set to 1.0 second between successive scans. For each sample, 16 transients were accumulated and averaged to enhance the **signal-to-noise ratio**.

2.3.3 Data Processing

Free induction decay (**FID**) signals were processed using **Bruker TopSpin** 4.1.3 software. An **exponential line broadening** function of 0.3 Hz was applied prior to **Fourier transformation**. Zero filling to 131,072 (128K) points was performed to improve digital resolution. Automatic phase correction was applied, followed by manual fine-tuning when necessary. Baseline correction was performed using a polynomial fitting routine. Chemical shifts were referenced to the TMS singlet at $\delta = 0.00$ ppm.

2.3.4 Spectral Analysis and Peak Assignment

Peak assignments were made based on chemical shift values, **signal multiplicity patterns**, coupling constants, and integration ratios. Chemical shift ranges were compared with **standard literature values** and NMR databases, including the **Spectral Database** for Organic Compounds (SDBS) and NMRShiftDB. All assignments were cross-validated against **predicted spectra**.

3. RESULTS AND DISCUSSION

The present comparative ^{1}H-NMR investigation of methyl tert-butyl ether (MTBE) and n-butanol has successfully demonstrated the profound influence of molecular structure, symmetry, and functional group identity on **proton resonance** behavior and **spectral complexity**. The experimental data obtained are in excellent quantitative agreement with theoretical predictions and published spectral databases.

3.1 ^{1}H-NMR Spectral Analysis of MTBE

The ^{1}H-NMR spectrum of MTBE in $CDCl_3$ exhibited a characteristically **simple pattern** consisting of **two well-resolved singlet resonances**, reflecting the high degree of **molecular symmetry**. The methoxy protons ($-OCH_3$) gave rise to a sharp singlet at $\delta = 3.35$ ppm, consistent with literature values. This downfield chemical shift is attributed to the **deshielding effect** exerted by the electronegative **oxygen atom**.

The tert-butyl methyl protons ($C(CH_3)_3$) resonated as a **sharp singlet** at $\delta = 1.22$ ppm, characteristic of alkyl protons in a **shielded environment**. Integration of the **two signals** yielded a ratio of 3.00:9.00 (methoxy:tert-butyl), which simplifies to 1:3, perfectly consistent with the **molecular formula** $C_5H_{12}O$.

Table 1: ^{1}H-NMR Spectral Data for Methyl Tert-Butyl Ether (MTBE) in $CDCl_3$

Chemical Shift (δ, ppm)	Multiplicity	Integration	Proton Assignment	Coupling Constant (J, Hz)
3.35	Singlet	3.00	–OCH_3 protons	-
1.22	Singlet	9.00	$(CH_3)_3C$– protons	-

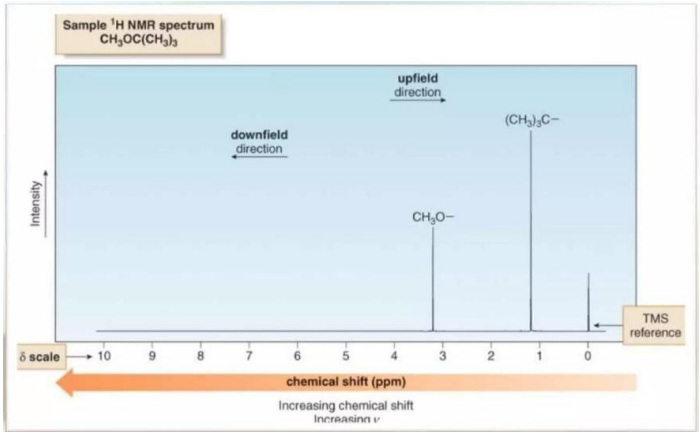

Figure 1: ^{1}H-NMR Spectrum of Methyl Tert-Butyl Ether (MTBE) showing chemical shift scale and signal assignments.

3.2 ^{1}H-NMR Spectral Analysis of n-Butanol

In stark contrast to the **simplicity** observed for MTBE, the ^{1}H-NMR spectrum of n-butanol in $CDCl_3$ displayed a **complex multi-signal pattern** arising from **four distinct proton environments**.

The terminal methyl protons (–CH_3, C-4 position) resonated as a well-defined triplet centered at δ = 0.94 ppm with a coupling constant J = 7.3 Hz. The internal methylene protons exhibited complex **multiplet patterns** in the region δ = 1.30–1.60 ppm. The methylene protons directly adjacent to the **hydroxyl group** (–CH_2–OH, C-1 position) appeared as a **triplet centered** at δ = 3.62 ppm with J = 6.5 Hz. The hydroxyl proton (–OH) resonated as a **broad singlet** at δ = 2.22 ppm, displaying characteristic **broadening** due to **rapid proton exchange**.

Integration of the four signal groups yielded ratios closely matching the expected values based on the molecular formula $C_4H_{10}O$, totaling ten protons in agreement with the structure of n-butanol.

Table 2: ^{1}H-NMR Spectral Data for n-Butanol in $CDCl_3$

Chemical Shift (δ, ppm)	Multiplicity	Integration	Proton Assignment	Coupling Constant (J, Hz)
0.94	Triplet	3.00	Terminal –CH_3 protons (C-4)	7.3
1.30–1.60	Multiplet	4.00	Internal –CH_2– protons (C-2, C-3)	7.0–7.3
3.62	Triplet	2.00	–CH_2–OH protons (C-1)	6.5
2.22	Broad singlet	1.00	–OH proton	-

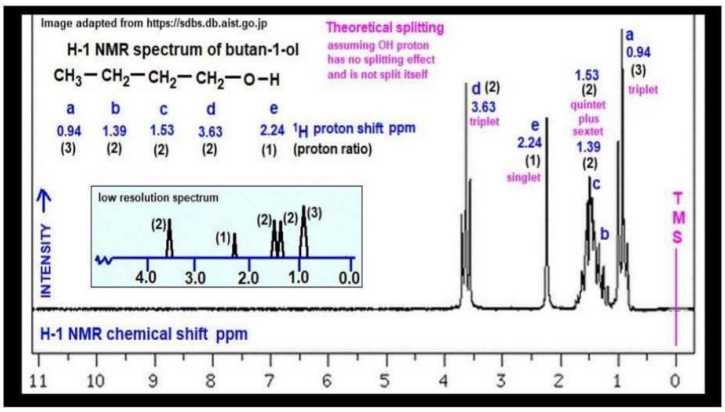

Figure 2: ^{1}H-NMR Spectrum of n-Butanol showing theoretical splitting patterns and chemical shift assignments.

3.3 Comparative Discussion of Spectral Features

The comparative ¹H-NMR analysis of MTBE and n-butanol reveals profound differences in **spectral complexity** that directly reflect **underlying structural characteristics**, particularly **molecular symmetry**, functional group identity, and the presence or absence of proton-proton coupling pathways.

MTBE exhibits high molecular symmetry, resulting in a remarkably simple spectrum consisting of only two singlet signals despite containing twelve hydrogen atoms. The absence of neighboring non-equivalent protons eliminates spin-spin coupling. In contrast, n-butanol lacks molecular symmetry and possesses four chemically distinct proton environments distributed along a **linear four-carbon chain**, generating a complex spectrum featuring multiple signals with diverse multiplicities.

The comparative analysis demonstrates that ¹H-NMR spectroscopy is **exquisitely sensitive** to **structural features** including molecular symmetry, functional group identity, proton connectivity, and electronic environment. The stark contrast between the simple MTBE spectrum and the complex butanol spectrum illustrates how **structural variations** manifest as spectral differences, enabling reliable **compound identification** and structural elucidation.

4. CONCLUSION

The present study confirms that **¹H-NMR spectroscopy** is a reliable and efficient analytical technique for structural elucidation of organic compounds. MTBE showed a simple and symmetrical spectrum, while butanol displayed complex resonance behavior due to multiple nonequivalent protons.

The observed spectral data were in good agreement with reported literature values. The comparative investigation demonstrates the effectiveness of **¹H-NMR spectroscopy** in differentiating ethers and alcohols and emphasizes its importance in organic and analytical chemistry.

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