International Journal of Multidisciplinary Education and Research
www.multieducationjournal.com
ISSN: 2455-4588
Received: 17-01-2026, Accepted: 16-02-2026, Published: 03-03-2026
Volume 11, Issue 1, 2026, Page No. 48-51

Structural study of adipic esters by ^{1}H-NMR nuclear magnetic resonance spectroscopy

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Abstract
In this article, we have determined the structure of adipic esters by nuclear magnetic resonance spectroscopy using a Bruker spectrometer. We have synthesized in two steps the following adipic esters: isotridecyl adipate and 2-(p-nonyl-phenoxy) ethyl, isotridecyl adipate and 2-(o-sec-butyl-phenoxy) ethyl and isotridecyl adipate and 2-phenoxy ethyl. These adipic esters have the following functional groups: RCH_3, R_2CH_2, C=C–CH_3, HC–COOH, ArOH and Ar-H.

Keywords: Ester, ^{1}H-NMR, structure, adipic

Introduction

Proton nuclear magnetic resonance (proton NMR, hydrogen-1 NMR, or ^{1}H NMR) is the application of nuclear magnetic resonance in NMR spectroscopy with respect to hydrogen-1 nuclei within the molecules of a substance, in order to determine the structure of its molecules. In samples where natural hydrogen (H) is used, practically all the hydrogen consists of the isotope 1H (hydrogen-1; i.e. having a proton for a nucleus).

Simple NMR spectra are recorded in solution, and solvent protons must not be allowed to interfere. Deuterated (deuterium = 2H, often symbolized as D) solvents especially for use in NMR are preferred, e.g. deuterated water, D_2O, deuterated acetone, $(CD_3)2CO$, deuterated methanol, CD_3OD, deuterated dimethyl sulfoxide, $(CD_3)2SO$, and deuterated chloroform, $CDCl_3$. However, a solvent without hydrogen, such as carbon tetrachloride, CCl_4 or carbon disulfide, CS_2, may also be used [1, 5].

Historically, deuterated solvents were supplied with a small amount (typically 0.1%) of tetramethylsilane (TMS) as an internal standard for referencing the chemical shifts of each analyte proton. TMS is a tetrahedral molecule, with all protons being chemically equivalent, giving one single signal, used to define a chemical shift = 0 ppm. It is volatile, making sample recovery easy as well. Modern spectrometers are able to reference spectra based on the residual proton in the solvent (e.g. the $CHCl_3$, 0.01% in 99.99% $CDCl_3$). Deuterated solvents are now commonly supplied without TMS.

Deuterated solvents permit the use of deuterium frequency-field lock (also known as deuterium lock or field lock) to offset the effect of the natural drift of the NMR's magnetic field B_0. In order to provide deuterium lock, the NMR constantly monitors the deuterium signal resonance frequency from the solvent and makes changes to the B_0 to keep the resonance frequency constant. Additionally, the deuterium signal may be used to accurately define 0 ppm as the resonant frequency of the lock solvent and the difference between the lock solvent and 0 ppm (TMS) are well known.

Proton NMR spectra of most organic compounds are characterized by chemical shifts in the range +14 to -4 ppm and by spin–spin coupling between protons. The integration curve for each proton reflects the abundance of the individual protons.

Simple molecules have simple spectra. The spectrum of ethyl chloride consists of a triplet at 1.5 ppm and a quartet at 3.5 ppm in a 3:2 ratio. The spectrum of benzene consists of a single peak at 7.2 ppm due to the diamagnetic ring current.

Together with carbon-13 NMR, proton NMR is a powerful tool for molecular structure characterization.

Chemical shift values, symbolized by δ, are not precise, but typical – they are to be therefore regarded mainly as a reference. Deviations are in ±0.2 ppm range, sometimes more. The exact value of chemical shift depends on molecular structure and the solvent, temperature, magnetic field in which the spectrum is being recorded and other neighboring functional groups. Hydrogen nuclei are sensitive to the hybridization of the atom to which the hydrogen atom is attached and to electronic effects. Nuclei tend to be deshielded by groups that withdraw electron density. Deshielded nuclei resonate at higher δ values, whereas shielded nuclei resonate at lower δ values. Examples of electron withdrawing substituents are −OH, −OCOR, −OR, $-NO_2$ and halogens.

These cause a downfield shift of approximately 2–4 ppm for H atoms on Cα (an aliphatic C atom directly bonded to the substituent in question) and of less than 1–2 ppm for H atoms on Cβ (an aliphatic C atom bonded to Cα). Carbonyl groups, olefinic fragments and aromatic rings contribute sp2 hybridized carbon atoms to an aliphatic chain. This causes a downfield shift of 1–2 ppm at Cα.

Note that labile protons (−OH, $-NH_2$, −SH) have no characteristic chemical shift. However, such resonances can be identified by the disappearance of a peak when reacted with D_2O, as deuterium will replace a protium atom. This method is called a D_2O shake. Acidic protons may also be suppressed when a solvent containing acidic deuterium ions (e.g. methanol-d4) is used. An alternate method for identifying protons that are not attached to carbons is the heteronuclear single quantum coherence (HSQC) experiment, which correlates protons and carbons that are one bond away from each other. A hydrogen that is not attached to a carbon can be identified because it does not have a crosspeak in the HSQC spectrum [6, 12].

Material and Methods

Structure Determination

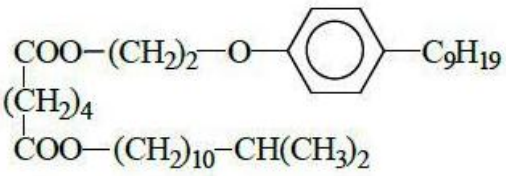
Nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a 200 MHz Bruker spectrometer. Deuterated

chloroform $CDCl_3$ (99.8% atom. %D) + 0.05% TMS (v/v) or DMSO-d6 (99.9% atom. %D) + 0.05% TMS (v/v) was used [252, 253]. Chemical shift values (δ) in 1H-NMR spectra are expressed in ppm (for $CDCl_3$ δ = 7.27 ppm), and coupling constants (J) are expressed in Hz. The abbreviations used for the multiplicity of signals are: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet) [13, 19].

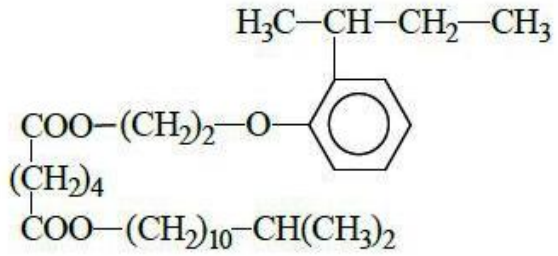
Fig 1: Bruker 200 MHz spectrometer

Synthesis of adipic esters

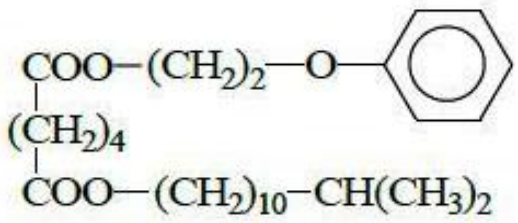
Isotridecyl adipate and 2-(p-nonyl-phenoxy) ethyl is obtained by synthesizing one mole of adipic acid with 1 mole of 2-(p-nonyl-phenoxy) ethanol and 0.035 moles of p-toluene sulfonic acid. In stage I: temperature 115 - 145^{0}C, time 5 - 7 hours, toluene 175 ml. In the second stage, one mole of isotridecanol is added at a temperature between 120^{0}C - 165^{0}C, time 5 - 7 hours.



Isotridecyl adipate and 2-(o-sec-butyl-phenoxy) ethyl is obtained by synthesizing 1.15 moles of adipic acid with 1.15 moles of 2-(p-sec-butyl-phenoxy) ethanol and 0.035 moles of p-toluene sulfonic acid. In stage I: temperature 115 - 145^{0}C, time 5 - 7 hours, toluene 175 ml. In the second stage, 1.15 of isotridecanol is added at a temperature between 120^{0}C - 165^{0}C, time 5 - 7 hours.



Isotridecyl adipate and 2-phenoxy ethyl are obtained in the first stage from 1.3 moles of adipic acid, 2 phenoxy ethanol 1.3 moles and 0.04 moles of p-toluene sulfonic acid. . In stage I: temperature 115 - 145^{0}C, time 5 - 7 hours, toluene 175 ml. In the second stage, 1.3 moles of isotridecanol are added at a temperature between 120^{0}C - 165^{0}C, time 5 - 7 hours.



Results and discussion

Figure 2 shows the spectrum of isotridecyl adipate and 2-(p-nonyl-phenoxy)ethyl.

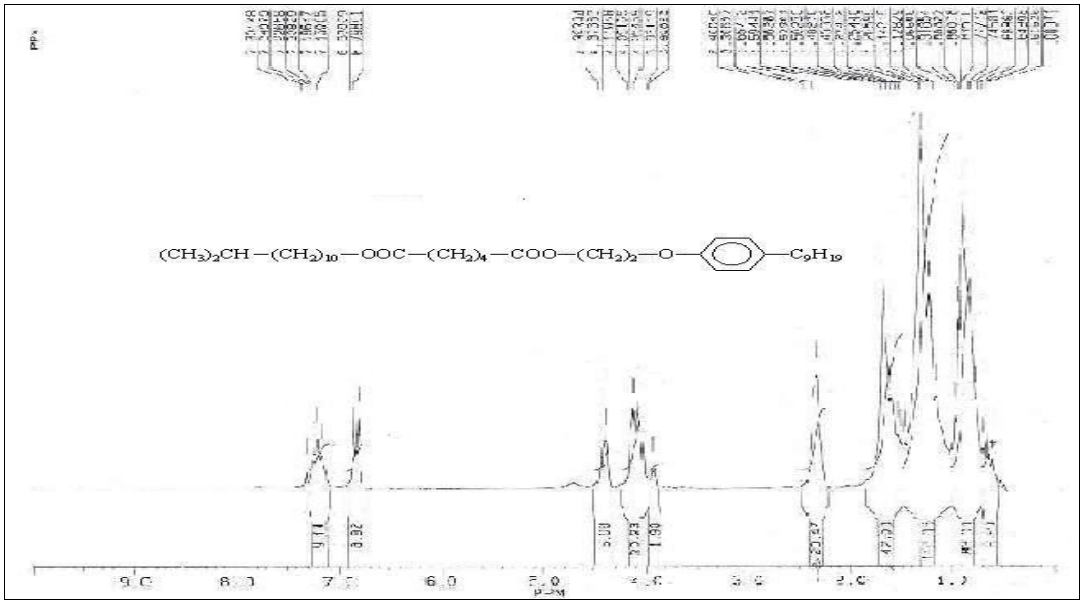


Fig 2: Spectrum of isotridecyl adipate and 2-(p-nonyl-phenoxy) ethyl

Table 1 shows the correspondence of peaks for isotridecyl adipate and 2-(p-nonyl-phenoxy) ethyl.

Table 1: Characteristic Proton NMR Shifts

Type of proton	Type of compound	Chemical shift range, ppm
RCH_3	1˚ aliphatic	0,83-0,96 (multiplet)
R2CH2	2˚ aliphatic	1,27 (multiplet)
C=C–CH3	allylic	1,66 (multiplet)
HC–COOH	acids	2,30-2,40 (multiplet)
ArOH	phenolic	4,05-4,11 (multiplet)
ArOH	phenolic	4,38 (multiplet)
Ar–H	aromatic	6,81 (dublet)
Ar–H	aromatic	7,20 (multiplet)

Figure 3 shows the spectrum of isotridecyl adipate and 2-(o-sec-butyl-phenoxy) ethyl.

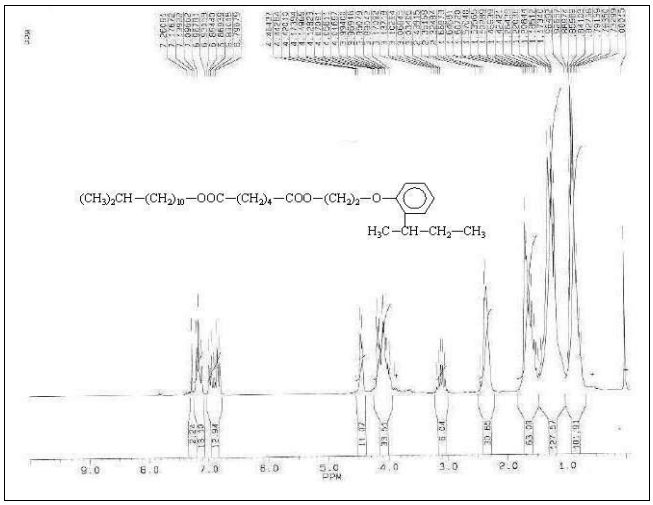


Fig 3: Spectrum of isotridecyl adipate and 2-(o-sec-butyl-phenoxy) ethyl

Table 2 shows the correspondence of the peaks for isotridecyl adipate and 2-(o-sec-butyl-phenoxy) ethyl.

Table 2: Characteristic Proton NMR Shifts

Type of proton	Type of compound	Chemical shift range, ppm
RCH_3	1˚ aliphatic	0,84-0,96 (multiplet)
R2CH2	2˚ aliphatic	1,17-1,22 (multiplet)
C=C–CH3	allylic	1,66 (multiplet)
HC–COOH	acids	2,32-2,43 (multiplet)
Ar–C–H	benzylic	3,03-3,17(multiplet)
ArOH	phenolic	4,05-4,12 (multiplet)
ArOH	phenolic	4,44 (multiplet)
Ar–H	aromatic	6,79-7,96 (multiplet)
Ar–H	aromatic	7,09-7,26 (multiplet)

Table 3 shows the correspondence of peaks for isotridecyl adipate and 2-phenoxy ethyl.

Table 3: Characteristic Proton NMR Shifts

Type of proton	Type of compound	Chemical shift range, ppm
RCH_3	1˚ aliphatic	0,85-0,96 (multiplet)
R2CH2	2˚ aliphatic	1,26 (multiplet)
C=C–CH3	allylic	1-42-1,69 (multiplet)
HC–COOH	acids	2,32-2,43 (multiplet)
ArOH	phenolic	4,05-4,12 (multiplet)
ArOH	phenolic	4,38 (multiplet)
Ar–H	aromatic	6,88-6,98 (multiplet)
Ar–H	aromatic	7,23-7,31 (multiplet)

Figure 4 shows the spectrum of isotridecyl adipate and 2-phenoxy ethyl.

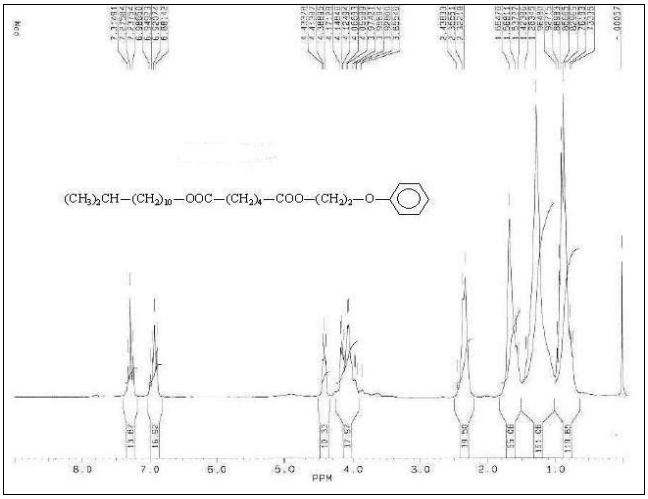


Fig 4: Spectrum of isotridecyl adipate and 2-phenoxy ethyl

Conclusions

The following adipic esters: isotridecyl adipate and 2-(p-nonyl-phenoxy) ethyl, isotridecyl adipate and 2-(o-sec-butyl-phenoxy) ethyl and isotridecyl adipate and 2-phenoxy ethyl have in their composition the following functional groups: RCH_3, R_2CH_2, C=C–CH_3, HC–COOH, ArOH and Ar-H and were determined with the Bruker spectrometer.

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