Supplementary Information (SI) for RSC Mechanochemistry.
This journal is © The Royal Society of Chemistry 2026

Supporting Information

Solvent-Free Continuous Flow Reduction of Aldehydes and Ketones via Mechanochemistry in a Screw Reactor

Mukesh Purohit [ab], Jagannath Padhi[ab] and Amol A. kulkarni [*ab]

a) Chemical Engineering & Process Development, CSIR-National Chemical Laboratory Pune, 411008, India. E-mail: aa.kulkarni@ncl.res.in

b) Academy of Scientific and Innovative Research (AcSIR), Ghaziabad, Uttar pradesh 201002, India.

Table of Contents

Content	Page #
General Methods	S3
General Reaction Procedure	S3
Experimental setup Reduction of aldehyde and ketone by using Screw Reactor	S4
Details of the Reaction Setup	S4
IR Characterization Data	S7
1H NMR and 13C NMR Spectra of Compounds (Characterization Data)	S10
1H NMR and 13C NMR Spectra of Compounds (Spectroscopic Data)	S14

General Methods

All reagents and solvents used in this study were of commercial grade and were utilized without further purification. The reactions were conducted in a single-screw Teflon reactor at room temperature. Reaction progress was monitored via thin-layer chromatography (TLC) using Merck silica gel 60-F254 plates (0.25 mm thickness), with detection carried out under UV light. Yields reported corresponding to isolated, spectroscopically pure compounds. 1H and 13C NMR spectra were obtained using a Bruker instrument operating at 400 MHz, 500 MHz and 100 or 125 MHz respectively, with tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in parts per million (ppm, δ) respectively in CDCl3 and DMSO-d6 solutions if not otherwise specified; chemical shifts (δ) are given in ppm. The 1H and 13C{1H} NMR spectra are referenced to residual solvent signals (CDCl3: δ H = 7.26 ppm, δ C = 77.2 ppm; DMSO-d6: δ H = 2.50 ppm, δ C = 39.5 ppm). NMR data are presented in the following format: chemical shift (δ), multiplicity [s = singlet, d = doublet, t = triplet, m = multiplet, b = broad], coupling constant (J, Hz), and integration values.

General experimental procedure Note:

Upon completion of the reaction, as verified by thin-layer chromatography (TLC), the residual product present in the grooves of the screw reactor was extracted using the appropriate solvents utilized during the reaction. This was followed by vacuum evaporation to minimize potential product yield loss.

General procedure for continuous flow Reduction of aldehyde and ketone functional group by mechanochemical approach.

Benzaldehyde (1) (0.047 mol, one equivalent) was introduced at a flow rate of 2.40 ml/min from dosing unit 1. In contrast, sodium borohydride (0.047 mol, one equivalent) was fed at a flow rate of 0.88 g/min from solid dosing unit 2 into a screw reactor rotating at a speed of 50 rpm, maintained at room temperature. The total residence time for the formation of alcohol and its derivatives ranged from 1 to 10 minutes. A solid reaction mass was collected and analyzed using thin-layer chromatography (TLC) with a solvent system of ethyl acetate and petroleum ether in a ratio of 5:95 to 20:80 to monitor the formation of the desired products. The reaction mixture was quenched with water (4 ml), diluted with EtOAc (8 ml) and washed with brine (2

ml). The combined organic layers were dried over anhydrous Na_2SO_4 (2g) and concentrated in vacuo. For ketone reductions, boric acid (30 mol%) was co-fed with the ketone substrate and sodium borohydride into the screw reactor, where it functions as a Lewis acid catalyst. The reaction was performed under ambient atmosphere. The extrudate was subsequently quenched with saturated aqueous sodium bicarbonate to neutralize residual acid and decompose any unreacted sodium borohydride. TLC analysis confirmed the complete conversion of the starting material . Finally, the product alcohols were extracted using ethyl acetate. The TLC analysis indicated complete product formation, achieving 100% conversion. All products were characterized using ^{1}H and ^{13}C NMR spectroscopic techniques, and their molecular masses were confirmed by gas chromatography-mass spectrometry (GC-MS).

General experimental setup for Reduction of aldehyde and ketone by using screw reactor:

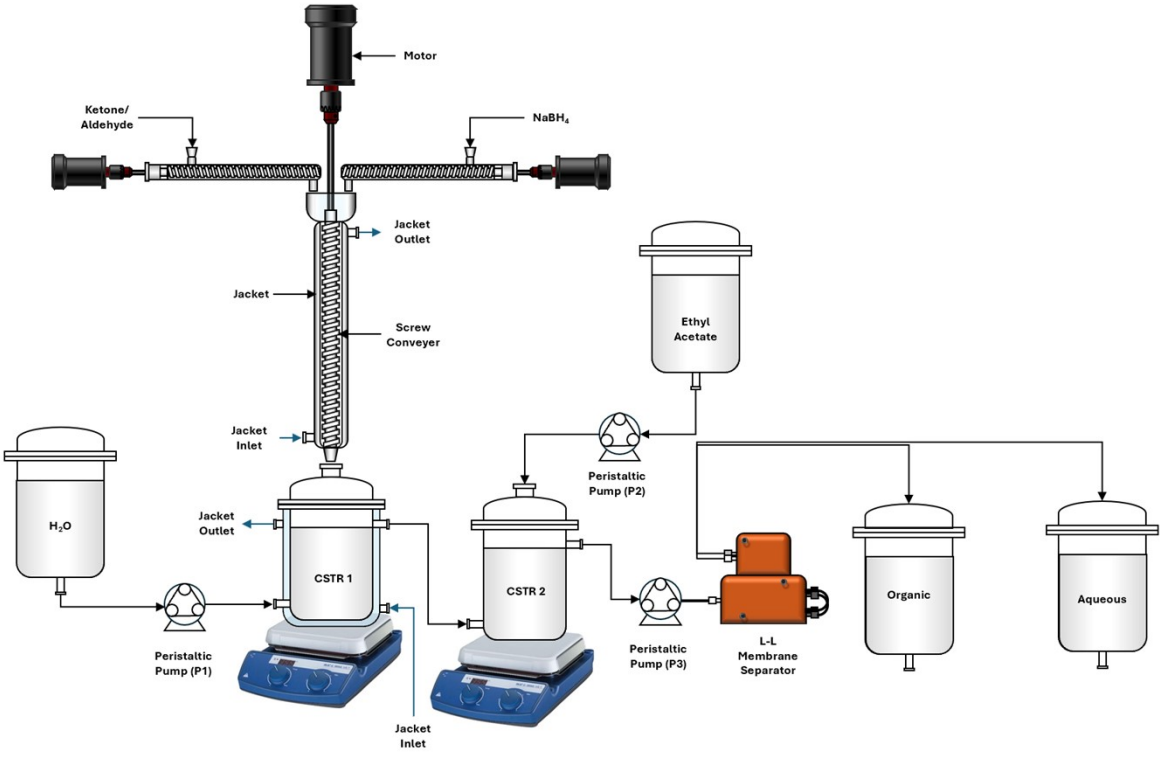


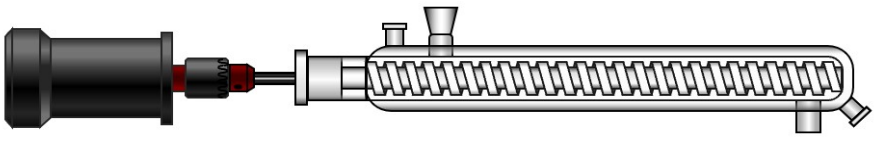
Figure 1: Reaction set-up for continuous flow reduction of aldehyde and ketone

Details of the reaction setup:

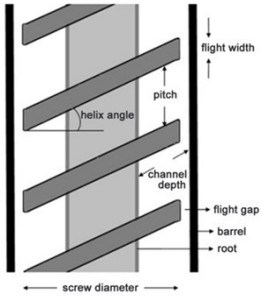
A jacketed single-screw reactor (glass-Teflon) has been used for continuous flow mechanochemical reduction of aldehyde and ketone functional group using Sodium borohydride. We have purchased a glass-Teflon reactor. which allows us to monitor the

changes occurring visibly during the course of the reaction. The extruder was fabricated by M/s Alpro Pvt. Ltd., Pune (India), and the vertical alignment for the screw reactor (having a glass jacket with a has a 39.6 mm outer diameter and 18 mm inner diameter with a 315 mm long PTFE screw having a diameter of 17.8 mm. This leaves a gap of only 0.2 mm between the jacket wall and the screw threads, the screw reactor, The screw has a pitch of 5 mm, a thread depth of 3 mm, and a total thread flight of 2.52 m. The reactor volume is 50 ml.as shown in Figure 1 The residence time was controlled using the rotation speed of the screw and flow rate of liquid reactant, controlled using a precision motor (Remi, India). The screw reactor parameters can be tuned to optimize the process for the reduction of the aldehyde functional group with good to excellent yield with short residence time, including the screw profile, feed rate, and screw speed. After completion of the reaction in the screw reactor, the reaction mixture containing benzyl alkoxide was discharged directly into a continuous stirred tank reactor (CSTR-1). receiving the stream from the screw reactor outlet while water was simultaneously introduced via pump P-1 in (CSTR-1) to quench the alkoxide intermediate. Continuous stirring ensures efficient mixing and complete quenching of the reaction mass. The quenched mixture exits the CSTR through an overflow outlet and is continuously transferred to CSTR-2, where ethyl acetate is added (P-2). This mixture is then pumped by pump P-3 to a liquid–liquid membrane separator for downstream phase separation, resulting in distinct organic and aqueous streams. Consequently, downstream processing proceeds continuously without interrupting the operation of the screw reactor.

Aligning the screw with a different angle



Horizontal position → **θ = 0°**



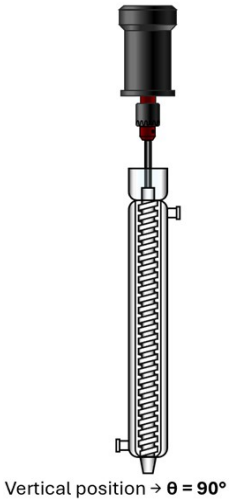


Figure 2: Screw reactor alignment

Experimental Setup

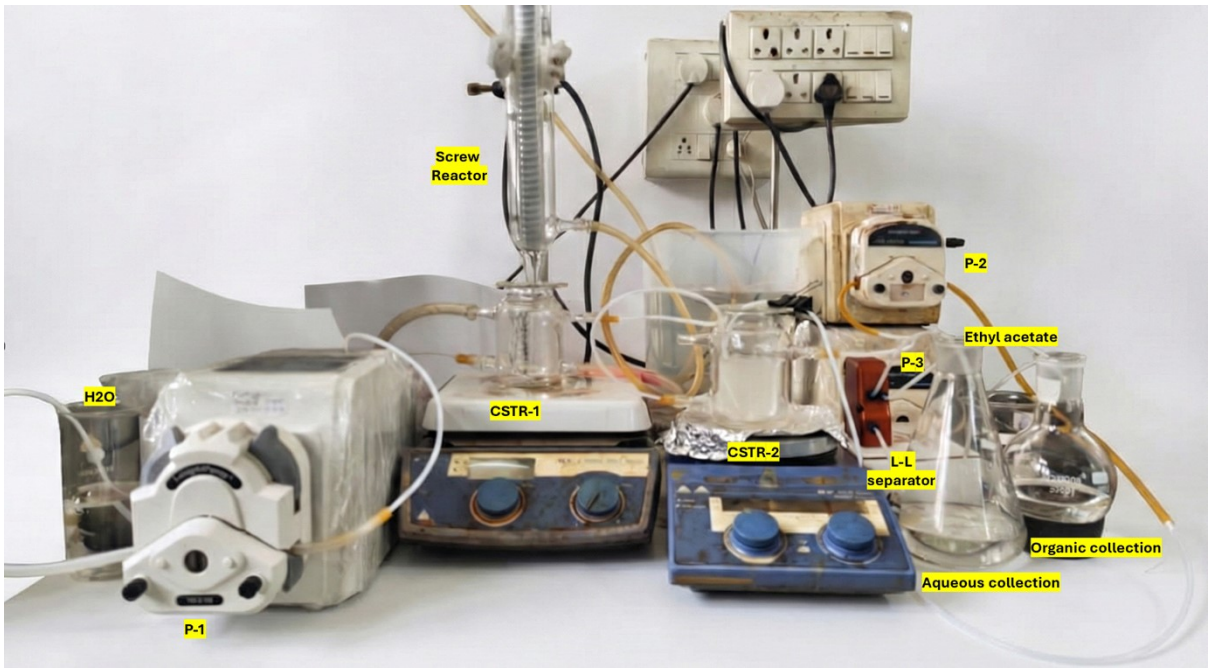
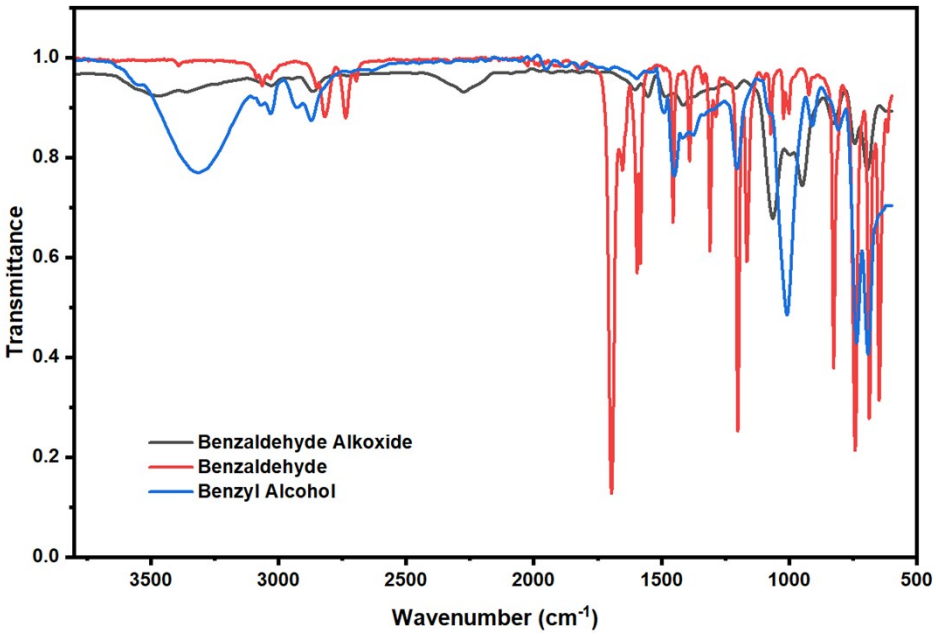


Figure 3: Experimental setup during reaction

IR Data:



In this study, Samples were collected directly from the reactor at defined residence times and immediately quenched by the addition of a small volume of water to protonate alkoxide or borate intermediates formed during the reduction. The quenched samples were then extracted with ethyl acetate, and the extracts were analyzed by TLC. This procedure enabled reliable detection of free alcohol products. we have included IR spectra comparing the starting material (benzaldehyde), the alkoxide intermediate sampled directly from the screw reactor, and the final benzyl alcohol product obtained after aqueous quenching. The IR spectrum of

benzaldehyde shows a strong C=O stretching band near 1700 cm^{-1}, characteristic of the aldehyde group. In the alkoxide intermediate spectrum collected from the screw reactor, this C=O band is significantly diminished or absent, indicating consumption of the aldehyde during reduction. Furthermore, no broad O–H stretch (~3350 cm^{-1}) is observed at this stage, consistent with the intermediate existing as an alkoxide rather than free alcohol After quenching, the IR of the product shows the appearance of a broad O–H stretch band near 3300 cm^{-1}, confirming the formation of benzyl alcohol. The absence of the C=O band further demonstrates the reaction's completion.

Continuous 2-h operation of the reaction:

The scaled 50 mL screw reactor was operated continuously for 2 h under optimized conditions to evaluate operational stability. Benzaldehyde was continuously fed at a flow rate of 16.15 mL min^{-1}, while sodium borohydride was introduced at 5.9 g min^{-1} to maintain the required stoichiometric ratio. During this period, consistent conversion (≈99%) was maintained without observable fouling, clogging, or performance decline. Based on experimentally measured substrate throughput and isolated yield, approximately 2.06 kg of benzyl alcohol was produced during the 2-hour continuous operation,

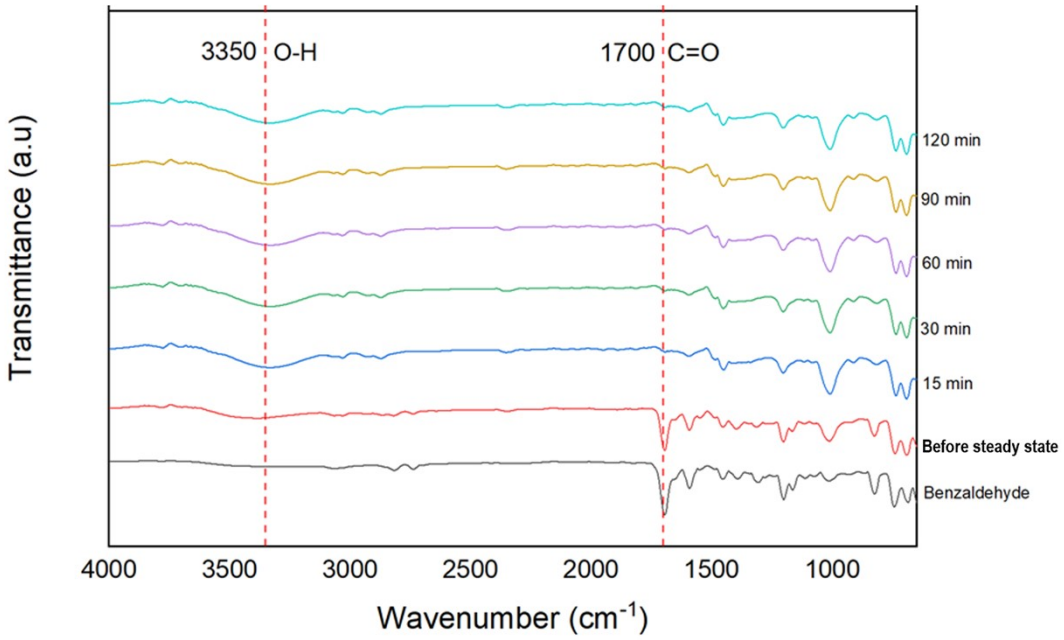
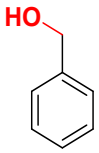


Figure 4: Time-Dependent ATR-IR Spectra for Steady-State Conversion of Benzaldehyde during 2 h Continuous Processing

Spectral data of the synthesized compounds

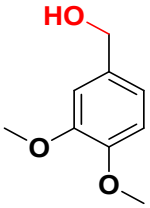
Characterization Data:

(2a) Benzyl alcohol



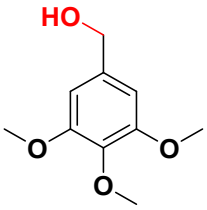
^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 4.52 (d, J=5.88 Hz, 2 H) 5.17 - 5.26 (t, J=5.43Hz, 1 H) 7.18 - 7.27 (m, 1 H, Ar-H) 7.27 - 7.37 (m, 4 H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6) δ ppm 63.47, 126.91, 127.10, 128.51, 143.01. GC-MS m/z 108.2

(2b) (3,4-Dimethoxyphenyl) methanol



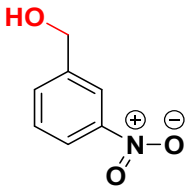
^{1}H NMR (400 MHz, CHLOROFORM-d) δ ppm 3.76 (s, 3 H) 3.74 (s, 3 H) 4.24 (br. s., 1 H) 4.48 (s, 2 H) 6.70 - 6.86 (m, 3 H). ^{13}C NMR (101 MHz, CHLOROFORM-d) δ ppm 55.48, 55.63, 64.25, 110.34, 110.98, 119.11, 133.85, 148.04, 148.71. GC-MS m/z 153.0

(2c) (3,4,5-Trimethoxyphenyl) methanol



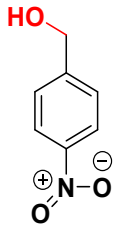
^{1}H NMR (400 MHz, CHLOROFORM-d) δ ppm 3.69 - 3.83 (m, 9 H) 4.49 - 4.56 (m, 2 H) 6.51 - 6.59 (m, 2 H). ^{13}C NMR (101 MHz, CHLOROFORM-d) δ ppm 55.68, 60.50, 64.40, 103.45, 136.54, 137.20, 152.89 GC-MS m/z 198.1

(2d) (3-Nitrophenyl) methanol



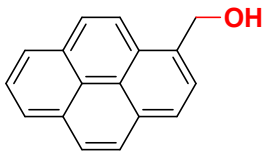
^{1}H NMR (400 MHz, CHLOROFORM-*d*) δ ppm 4.79 (s, 2 H) 7.51 (t, *J*=7.88 Hz, 1 H) 7.67 (d, *J*=7.50 Hz, 1 H) 8.09 (d, *J*=8.00 Hz, 1 H) 8.20 (s, 1 H). ^{13}C NMR (101 MHz, CHLOROFORM-*d*) δ ppm 63.80, 121.43, 122.40, 129.43, 132.70, 142.97, 148.30. GC-MS m/z 153.1

(2e) (4-Nitrophenyl) methanol



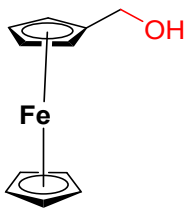
^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 4.64 (d, *J*=5.75 Hz, 2 H) 5.53 (t, *J*=5.69 Hz, 1 H) 7.56 - 7.62 (m, 2 H) 8.17 - 8.23 (m, 2 H). ^{13}C NMR (101 MHz, DMSO-d_6) δ ppm 62.46, 123.76, 127.50, 146.78, 151.26 Gc-MS m/z 153.0

(2f) 1-Pyrenemethanol



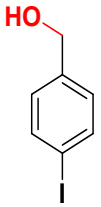
^{1}H NMR (400 MHz, CHLOROFORM-*d*) δ ppm 5.42 (d, *J*=4.88 Hz, 2 H) 7.98 - 8.09 (m, 4 H) 8.12 - 8.24 (m, 4 H) 8.39 (d, *J*=9.26 Hz, 1 H). ^{13}C NMR (101 MHz, CHLOROFORM-*d*) δ ppm 63.91, 123.03, 124.74, 125.00, 125.29, 125.32, 126.02, 126.08, 127.40, 127.50, 127.95, 128.85, 130.80, 131.27, 131.32, 133.79. GC-MS m/z 232.1

(2g) Ferrocenemethanol



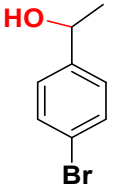
^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 4.09 (t, *J*=1.81 Hz, 2 H) 4.11 - 4.14 (m, 5 H) 4.17 - 4.22 (m, 4 H) 4.71 (t, *J*=5.75 Hz, 1 H). ^{13}C NMR (101 MHz, DMSO-d_6) δ ppm 59.61, 67.86, 68.54, 68.61, 88.86 GC-MS m/z 216.0

(2h) 4-Iodo Benzyl Alcohol



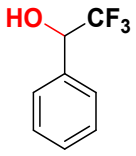
^{1}H NMR (400MHz, CHLOROFORM-d) δ = 7.71 - 7.65 (m, 2 H), 7.13 - 7.08 (m, 2 H), 4.63 (d, *J* = 5.6 Hz, 2 H), 1.80 (t, *J* = 5.8 Hz, 1 H), ^{13}C NMR (101MHz, CHLOROFORM-d) δ ppm 140.4, 137.6, 128.8, 93.0, 64.7 GC-MS m/z 234.1

(2i) 1-(4-Bromophenyl) ethanol



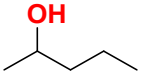
^{1}H NMR (400 MHz, CHLOROFORM-*d*) δ ppm 1.21 (d, *J*=6.50 Hz, 3 H) 3.72 (br. s., 1 H) 4.55 (q, *J*=6.59 Hz, 1 H) 6.98 (m, *J*=8.50 Hz, 2 H) 7.25 (m, *J*=8.50 Hz, 2 H). ^{13}C NMR (101 MHz, CHLOROFORM-*d*) d ppm 25.19, 69.46, 121.02, 127.27, 131.46, 144.82 GC-MS m/z 202.2

(2j) 2,2,2-Trifluoro-1-phenyl ethanol



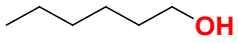
^{1}H NMR (400 MHz, CHLOROFORM-*d*) δ ppm 3.04 (br. s., 1 H) 4.85 (q, *J*=6.75 Hz, 1 H) 7.26 - 7.32 (m, 3 H) 7.32 - 7.39 (m, 2 H) ^{13}C NMR (101MHz, DMSO-d_6) δ ppm = 136.4, 129.2, 128.6, 128.1, 126.9, 124.1, 121.3, 71.5 19F NMR (377 MHz, CHLOROFORM-d) δ ppm -78.36 (s, 1 F) GC-MS m/z 177.1

(2k) 2-Pentanol



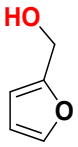
^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 0.80 - 0.91 (m, 3 H) 1.02 (d, *J*=6.13 Hz, 3 H) 1.19 - 1.40 (m, 4 H) 3.51 - 3.63 (m, 1 H) 4.28 (dd, *J*=4.75, 1.00 Hz, 1 H) ^{13}C NMR (101 MHz, DMSO-d_6) δ ppm 14.48,19.00, 24.05,41.80, 65.93

(2l) Hexanol



^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 0.82 - 0.93 (m, 3 H) 1.18 - 1.35 (m, 6 H) 1.35 - 1.47 (m, 2 H) 3.34 - 3.43 (m, 2 H) 4.33 (s, 1 H) ^{13}C NMR (101 MHz, DMSO-d_6) δ ppm 14.28, 22.64, 25.67, 32.99, 39.31, 61.21

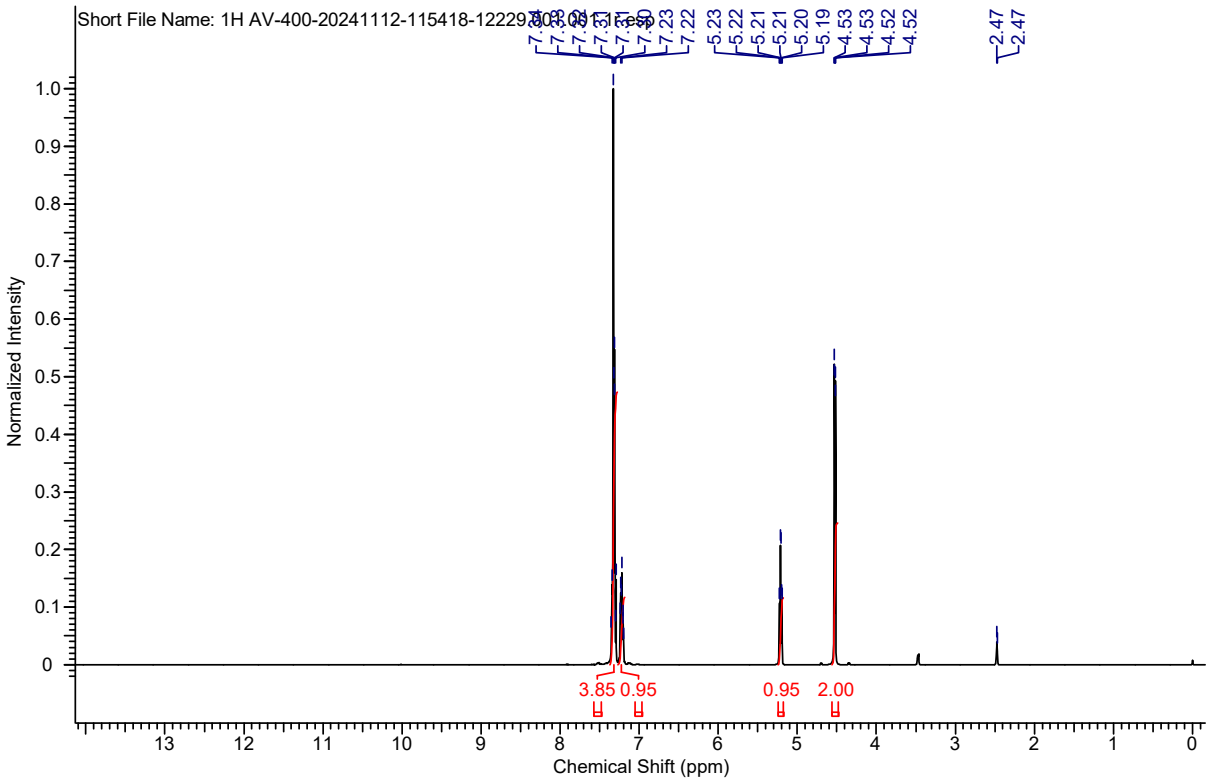
(2M) Furfural alcohol



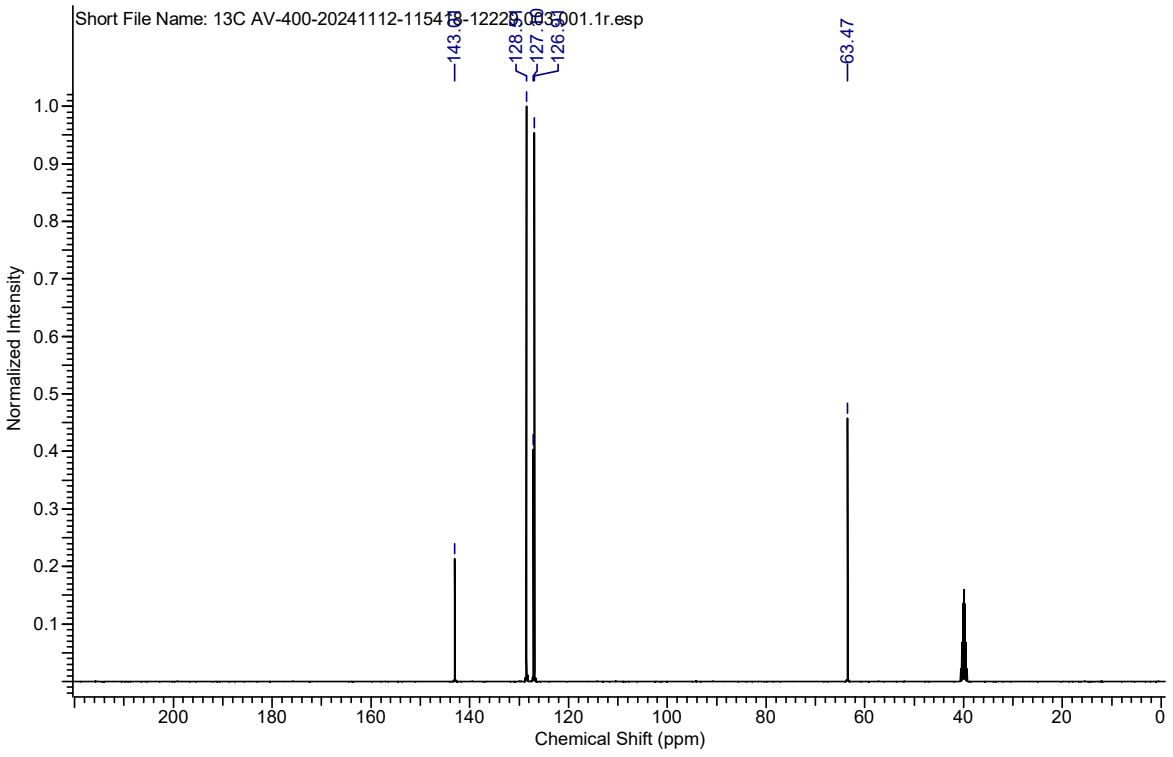
^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 4.33 - 4.45 (m, 3 H) 5.14 - 5.23 (m, 1 H) 6.22 - 6.32 (m, 1 H) 6.34 - 6.42 (m, 1 H) 7.52 - 7.61 (m, 1 H). ^{13}C NMR (101 MHz, DMSO-d_6) δ ppm 56.06, 107.30, 110.73, 142.52, 155.87

Spectroscopic Data:

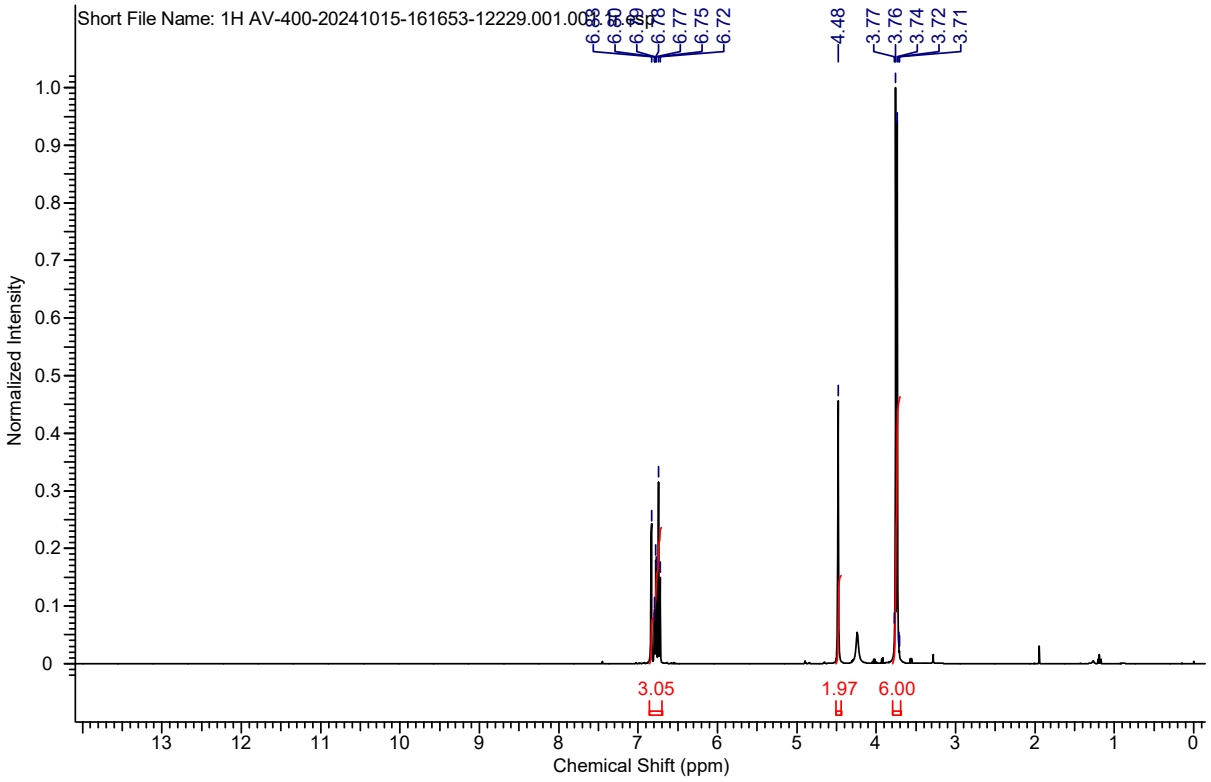
1H NMR - Benzyl alcohol :-(2a)



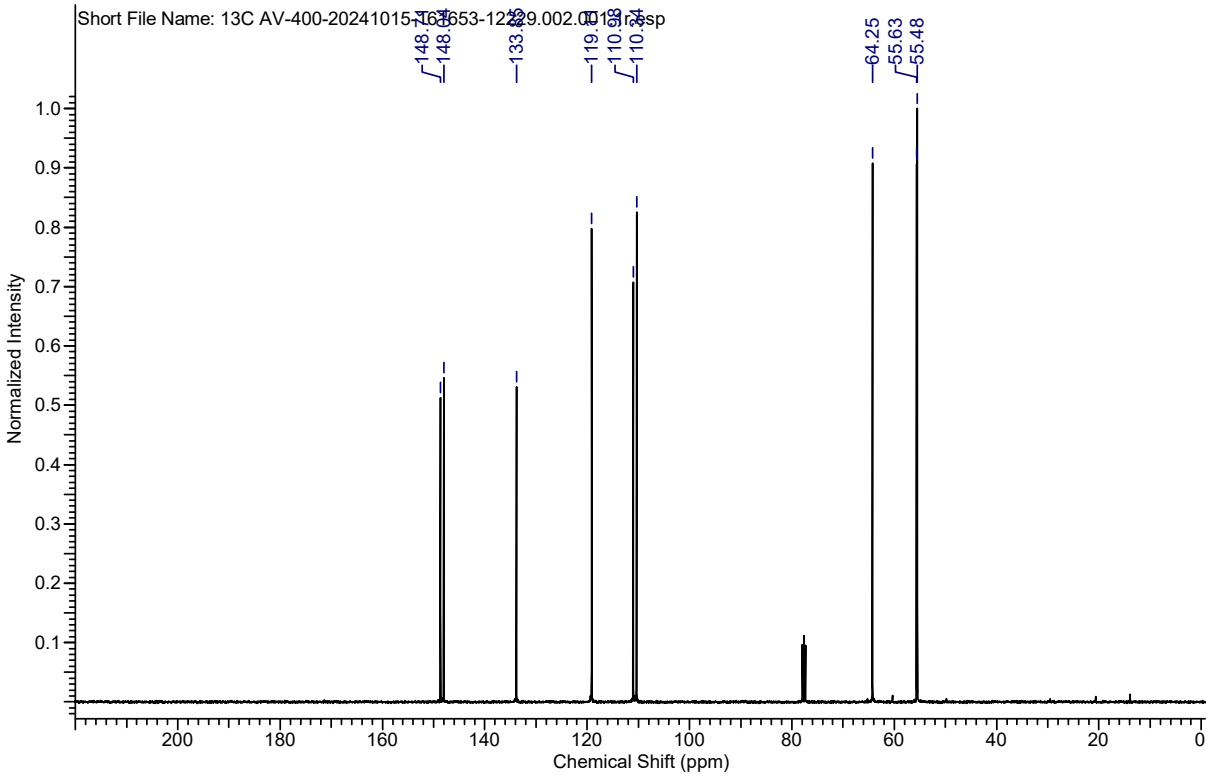
13C NMR - Benzyl alcohol :-(2a)



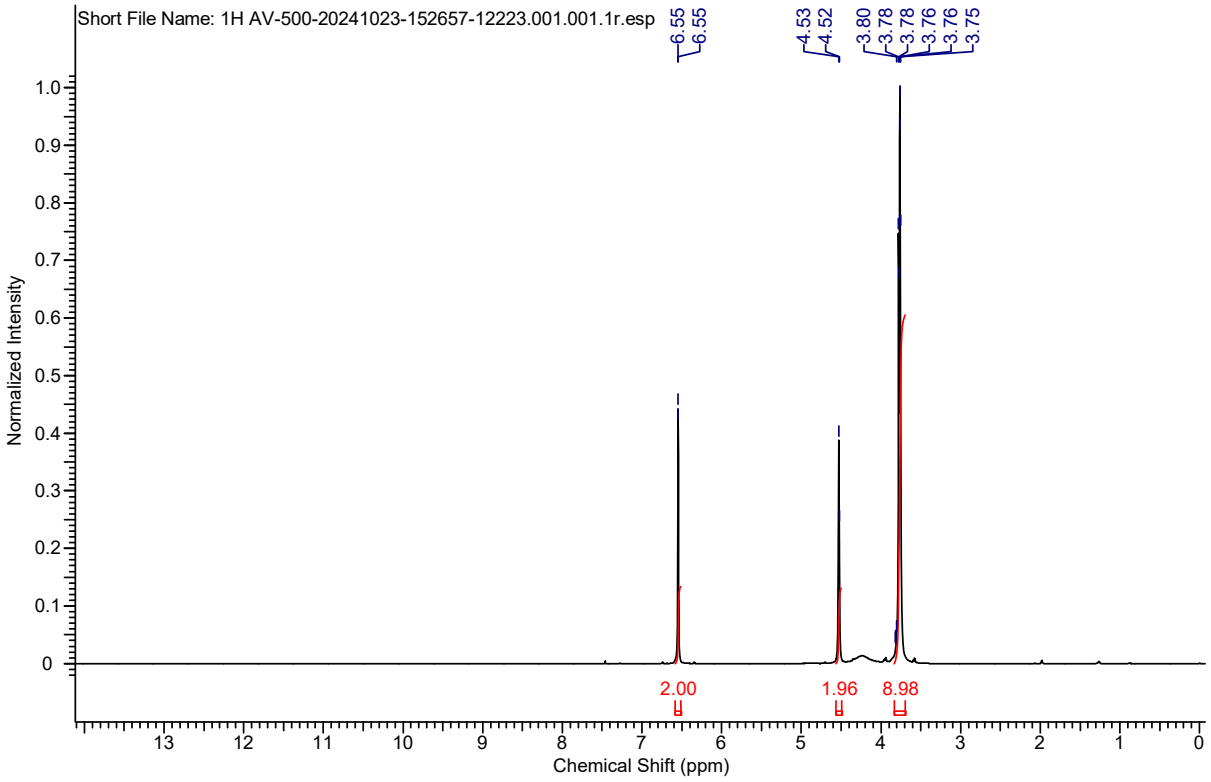
1H NMR - (3,4-Dimethoxyphenyl)methanol :- (2b)



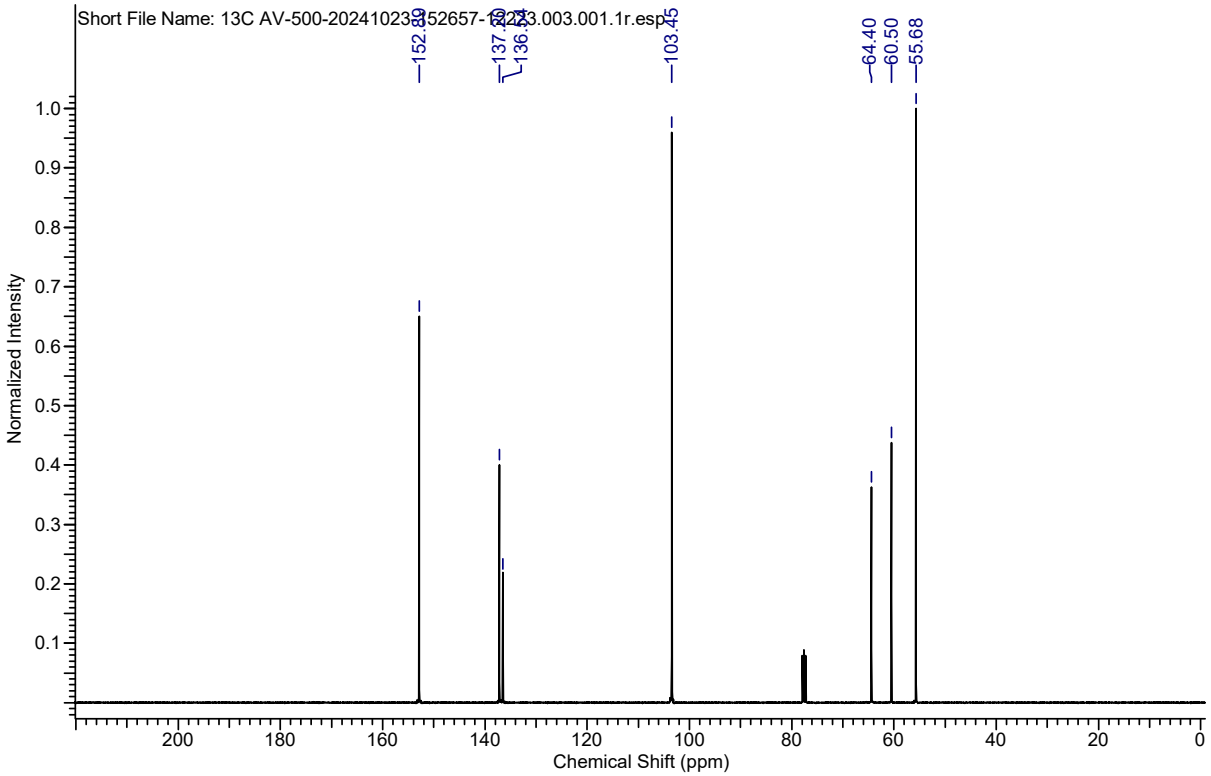
13C NMR - (3,4-Dimethoxyphenyl)methanol :- (2b)



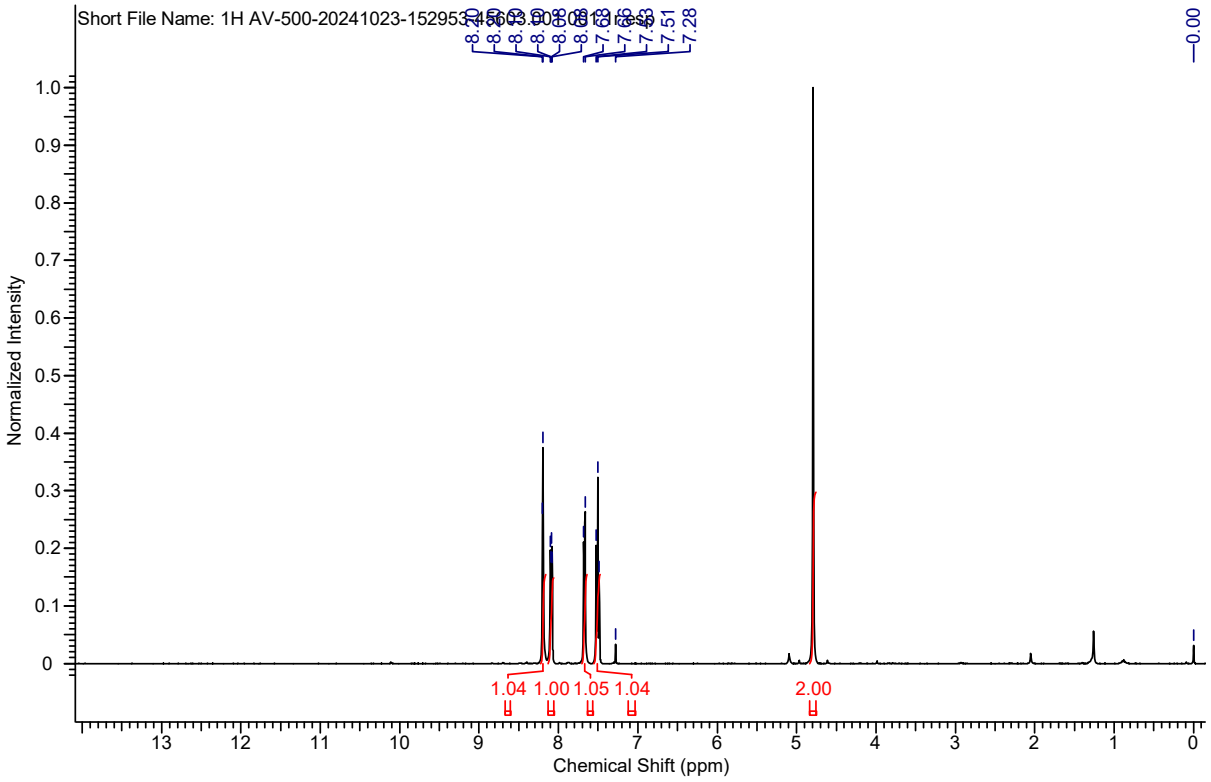
1H NMR - (3,4,5-Trimethoxyphenyl)methanol :- (2c)



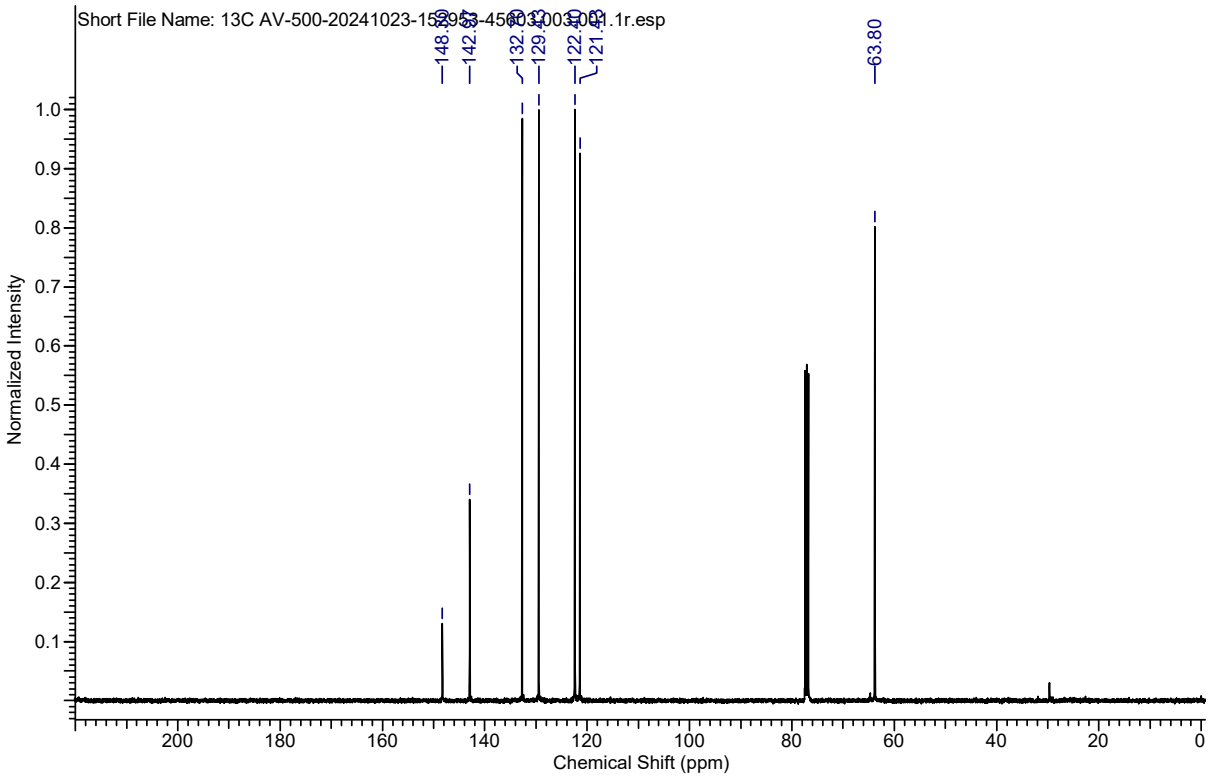
13C NMR- Of (3,4,5-Trimethoxyphenyl)methanol :- (2c)



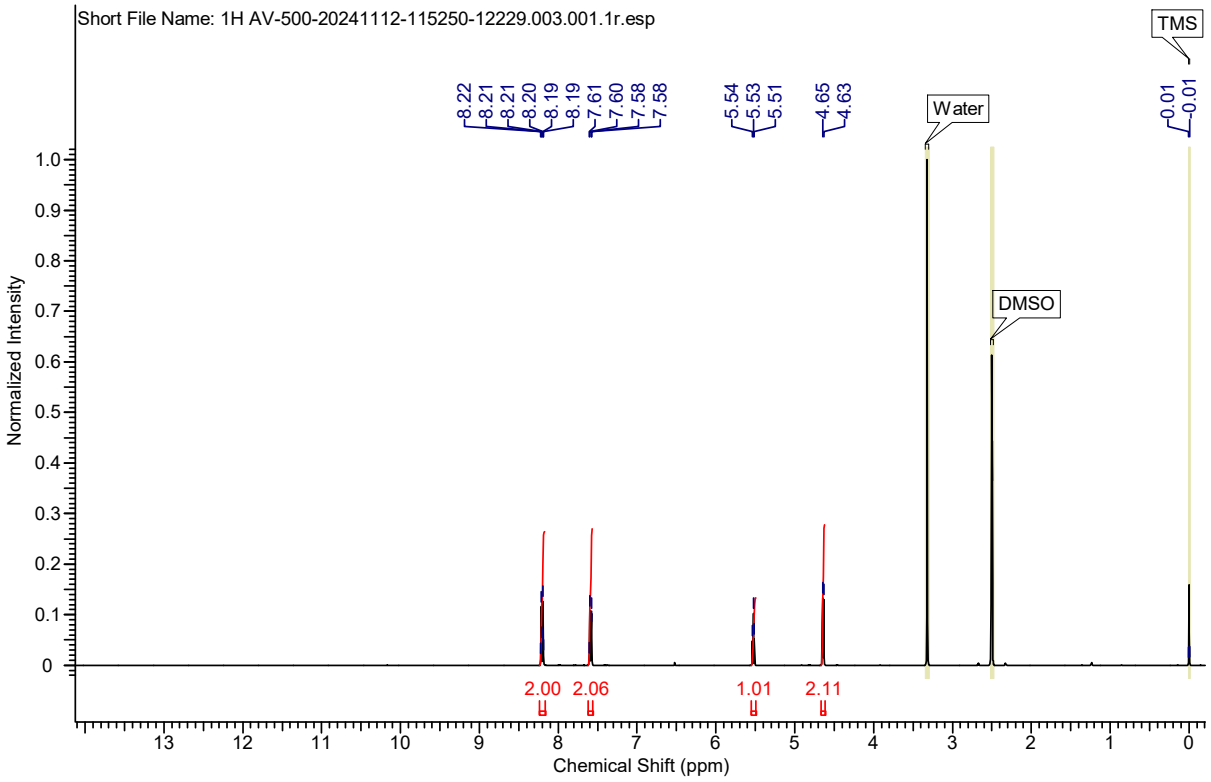
1H NMR - (3-Nitrophenyl)methanol :- (2d)



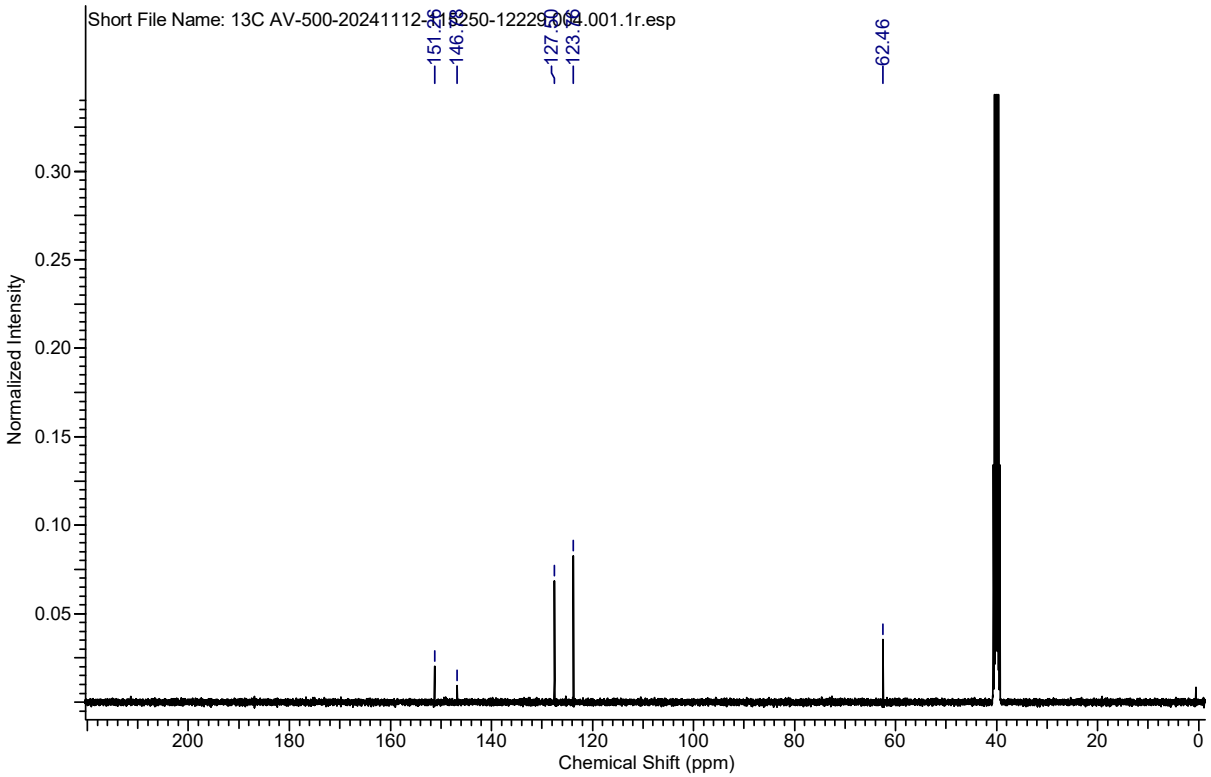
13C NMR - (3-Nitrophenyl)methanol :- (2d)



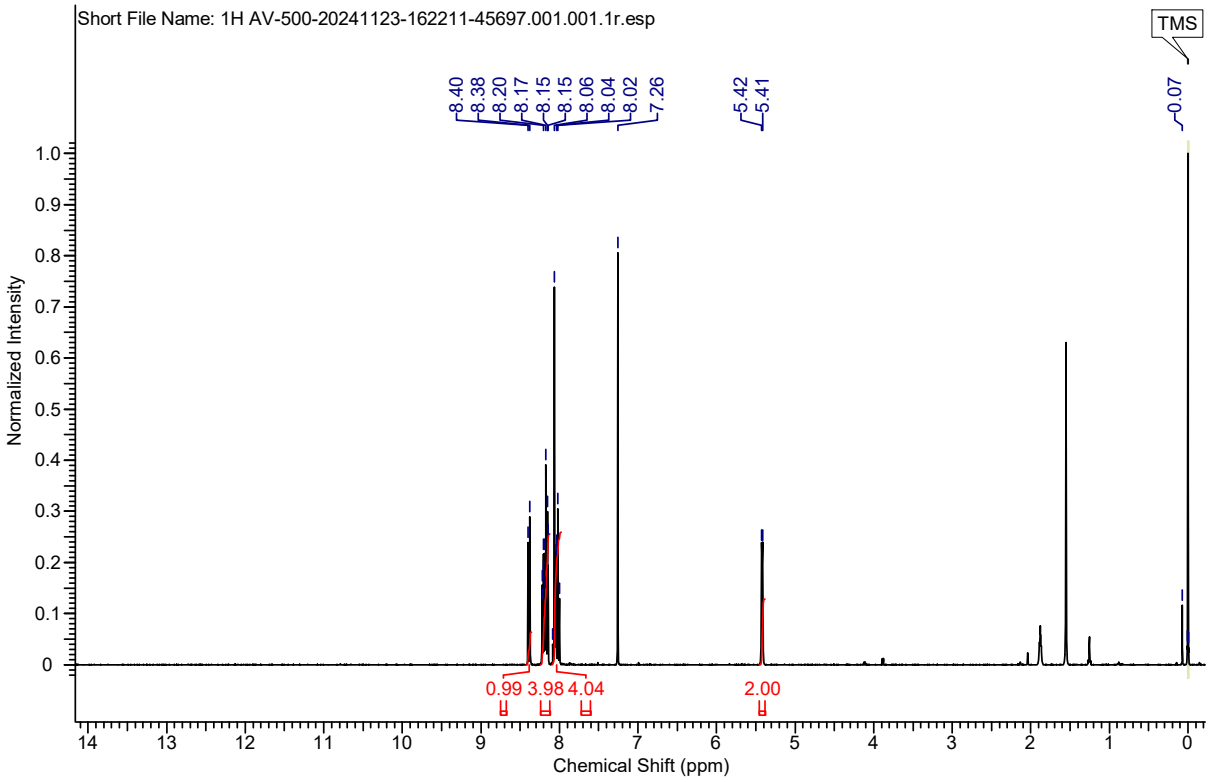
1H NMR – (4-Nitrophenyl)methanol:-(2e)



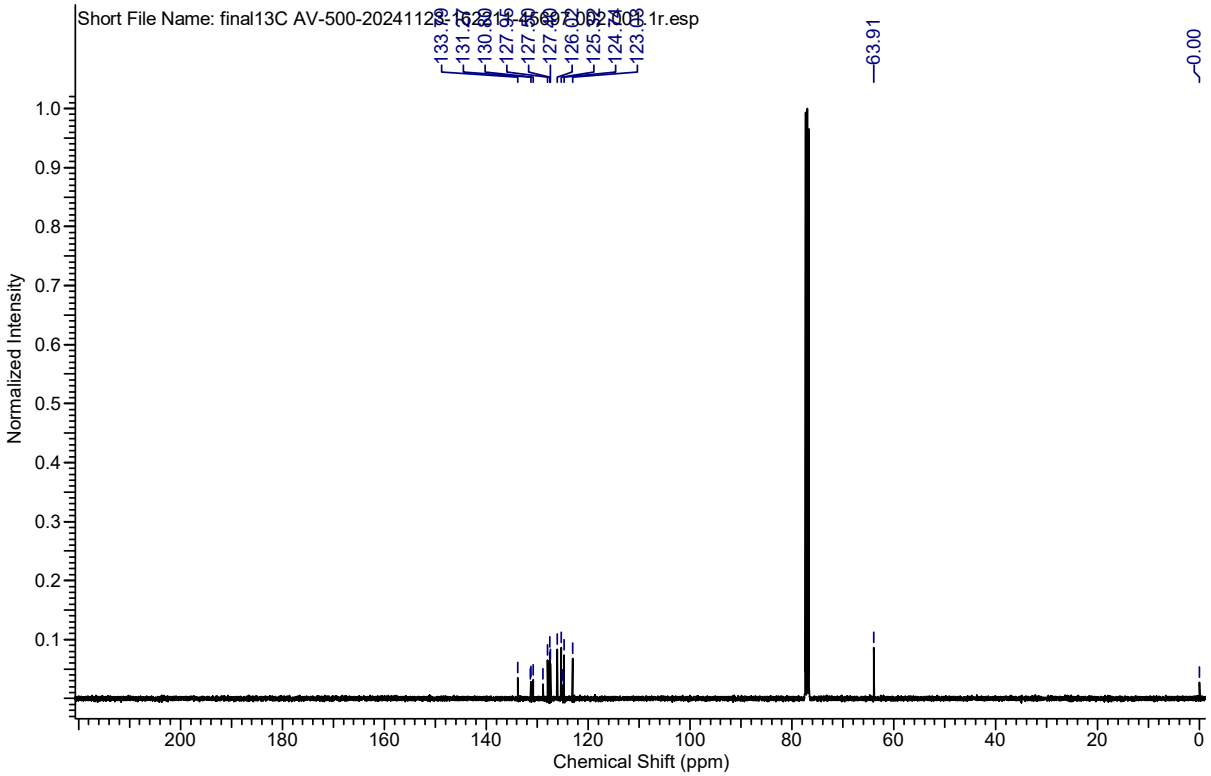
13C NMR - (4-Nitrophenyl)methanol :- (2e)



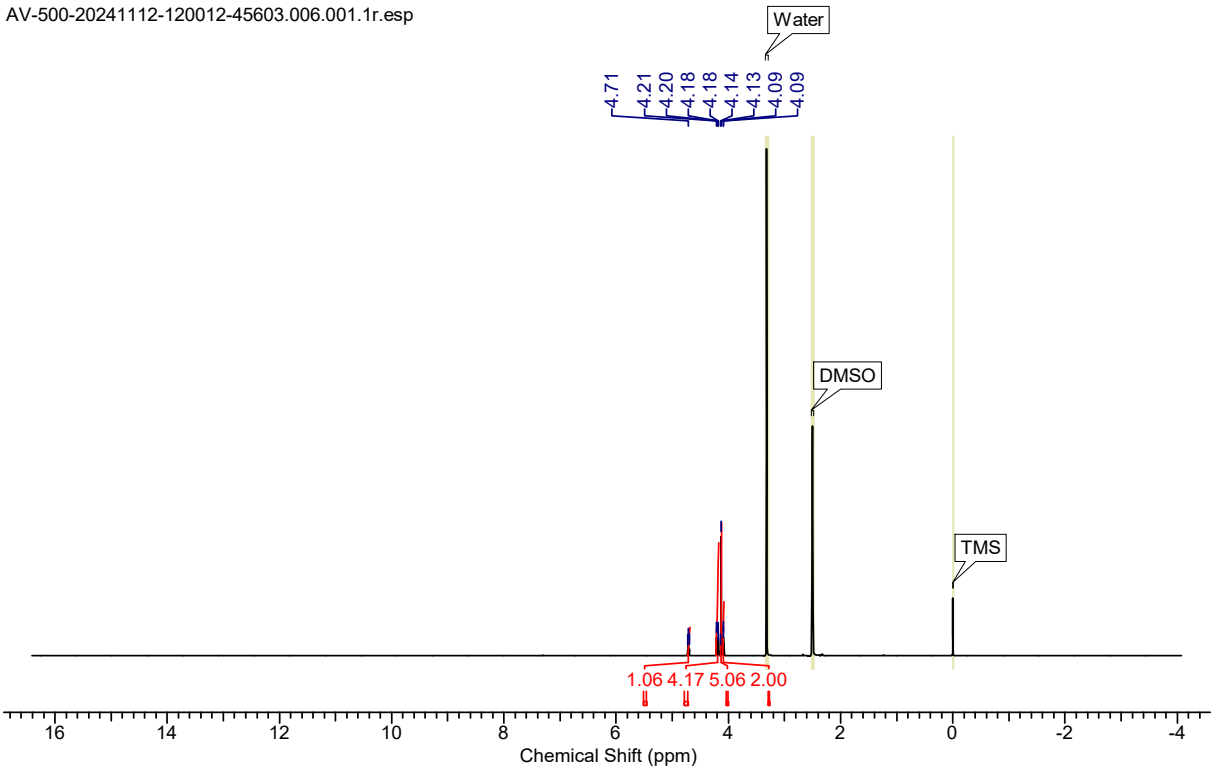
1H NMR - 1-Pyrenemethanol :- (2f)



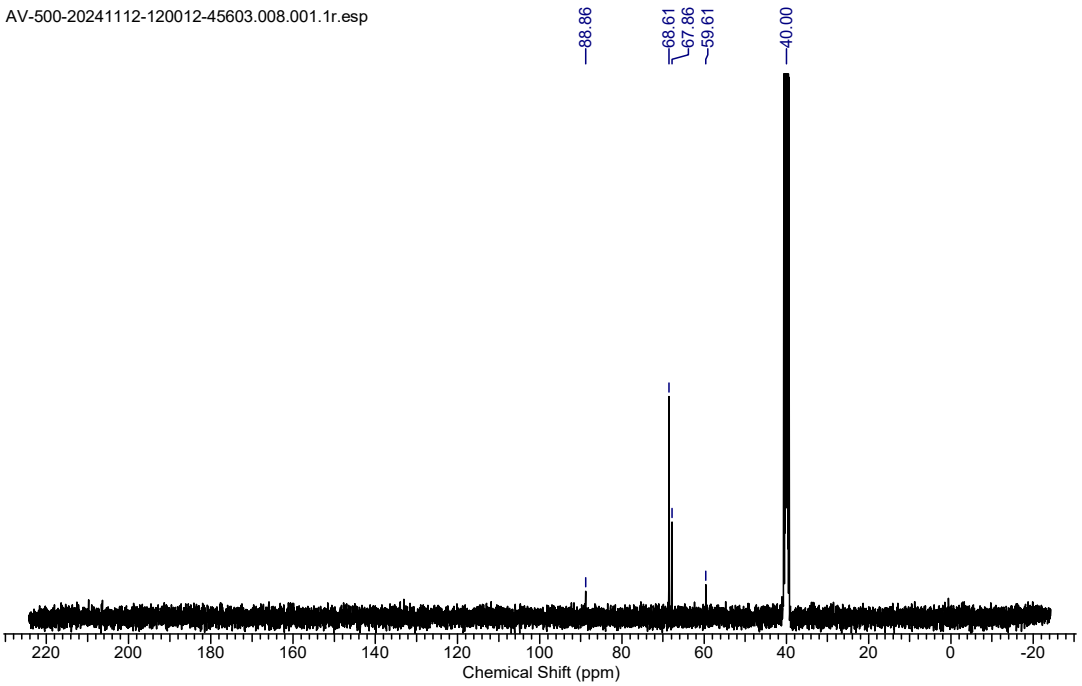
13C NMR - 1-Pyrenemethanol:- (2f)



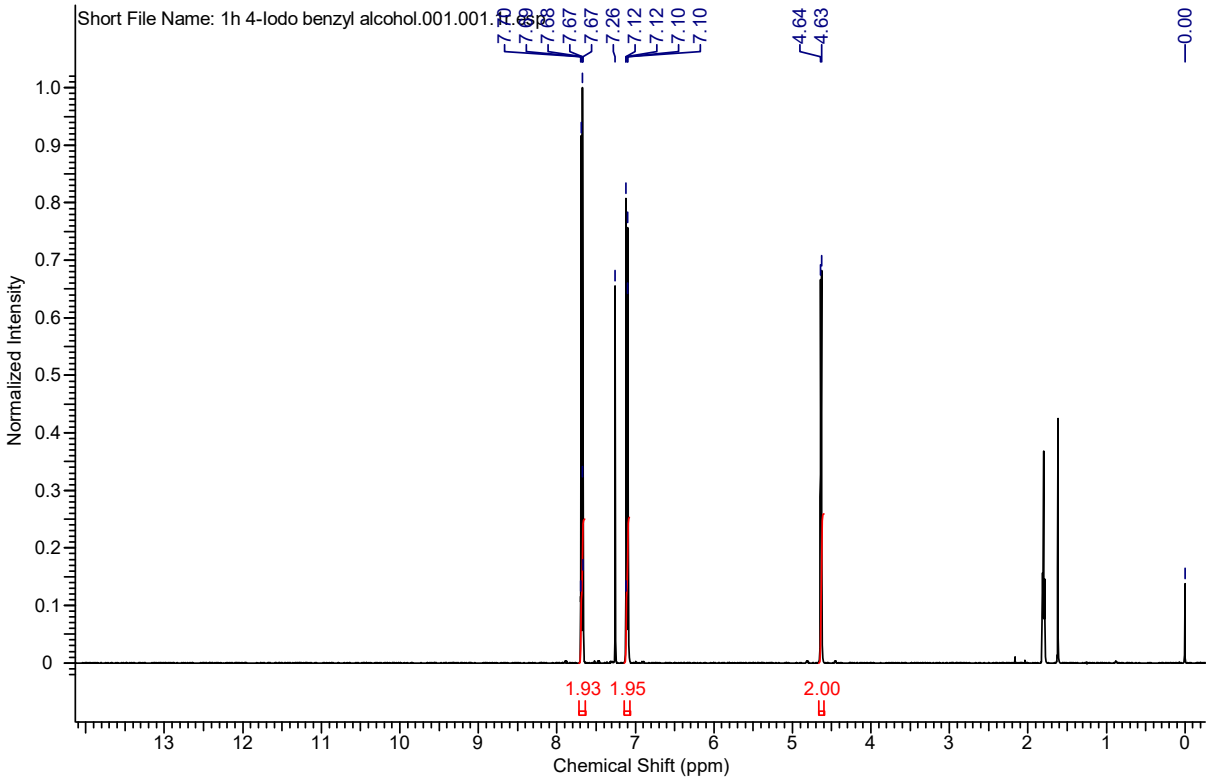
1H NMR – Ferrocenemethanol :- (2g)



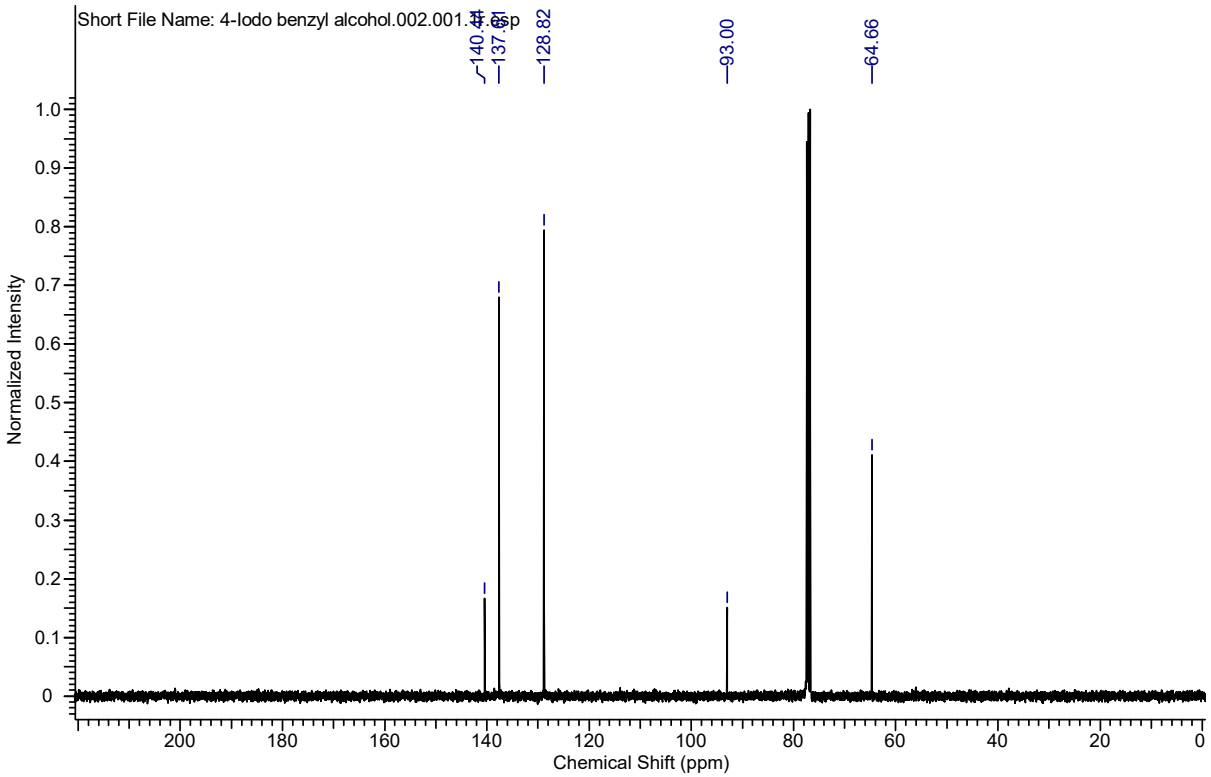
13C NMR – Ferrocenemethanol :- (2g)



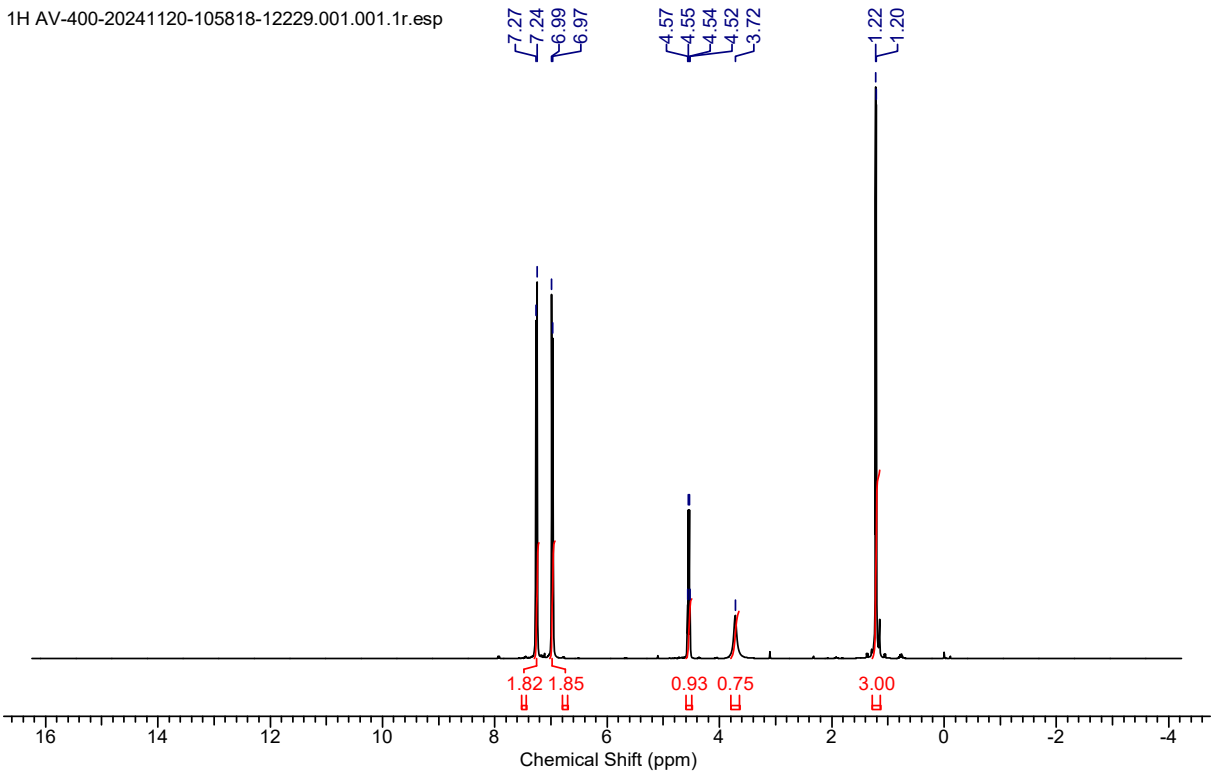
1H NMR – 4-Iodo benzyl alcohol :- (2h)



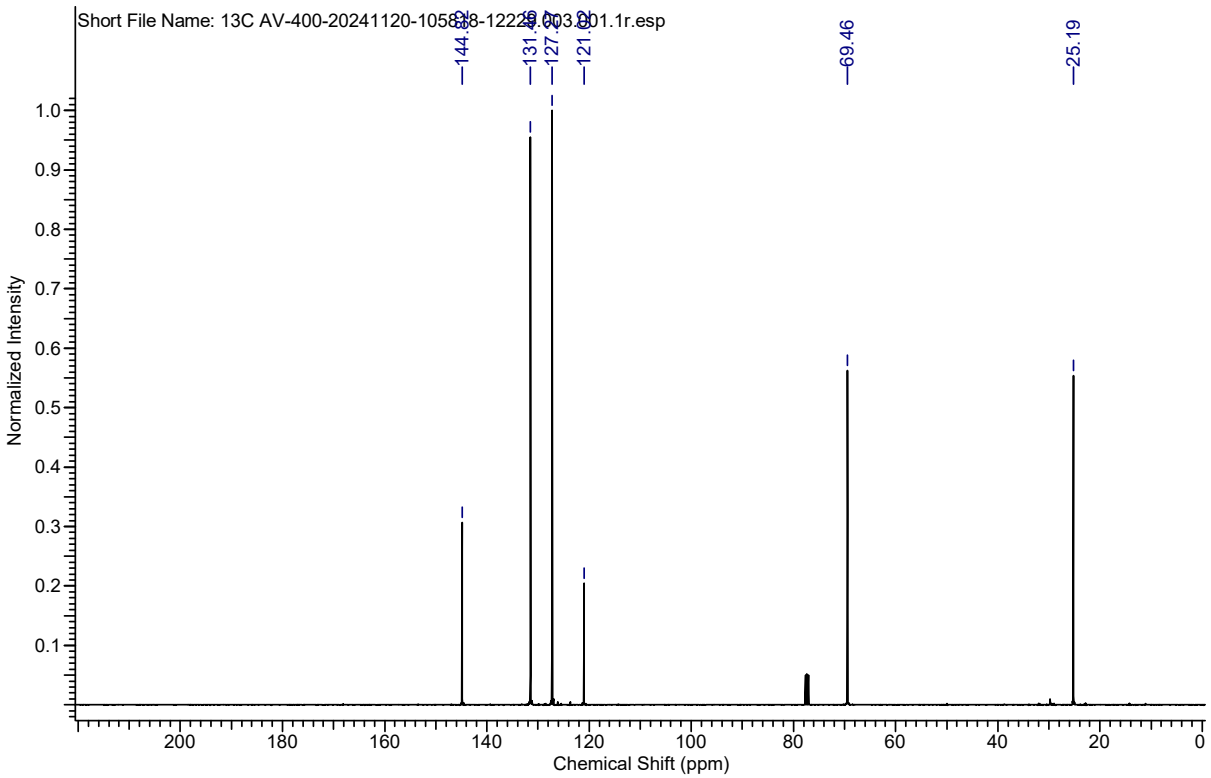
13C NMR – 4-Iodo benzyl alcohol :- (2h)



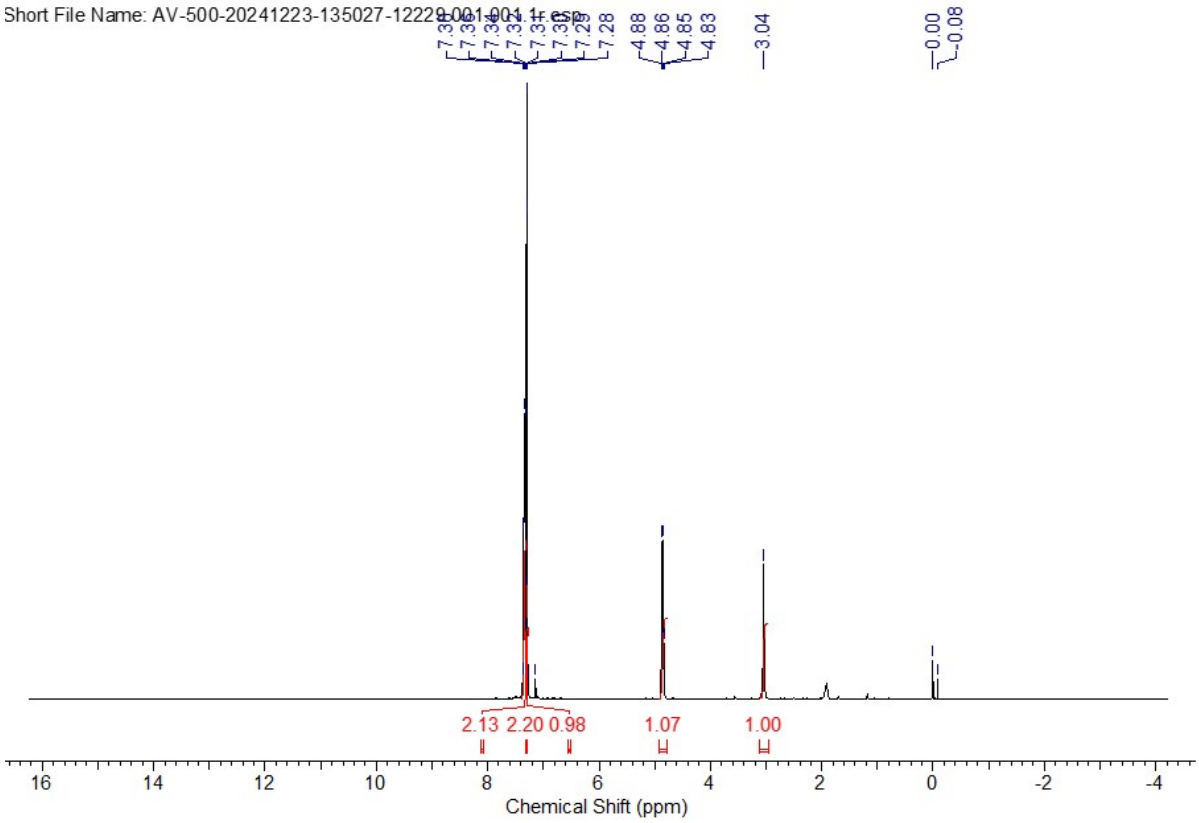
1H NMR - 1-(4-Bromophenyl)ethanol:- (2i)



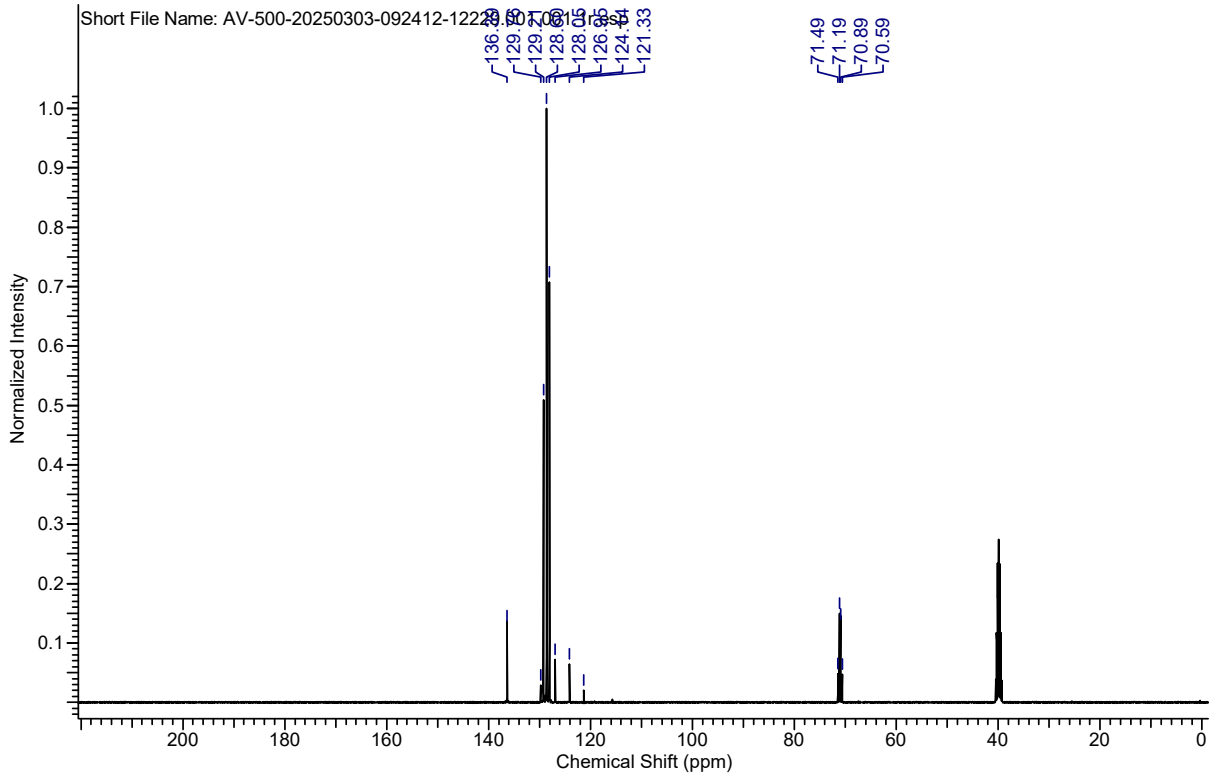
13C NMR - 1-(4-Bromophenyl)ethanol:- (2i)



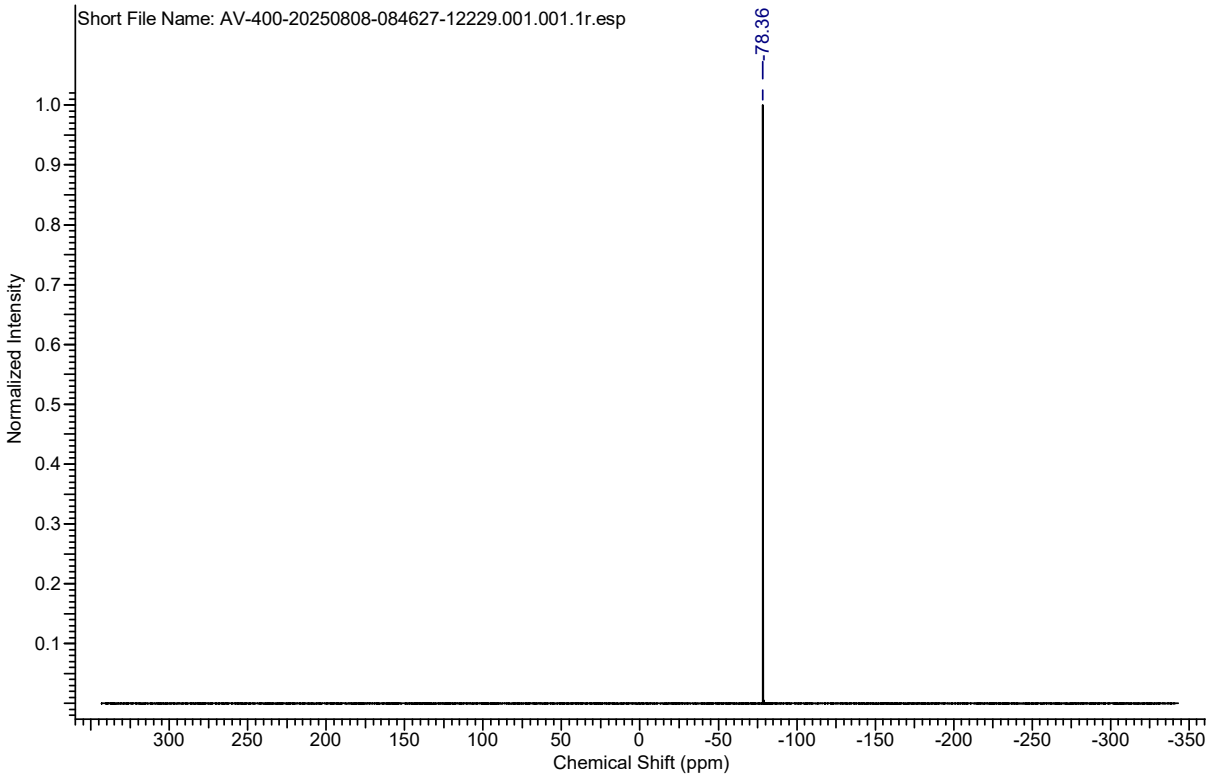
1H NMR - 2,2,2-Trifluoro-1-phenylethanol :- (2j)



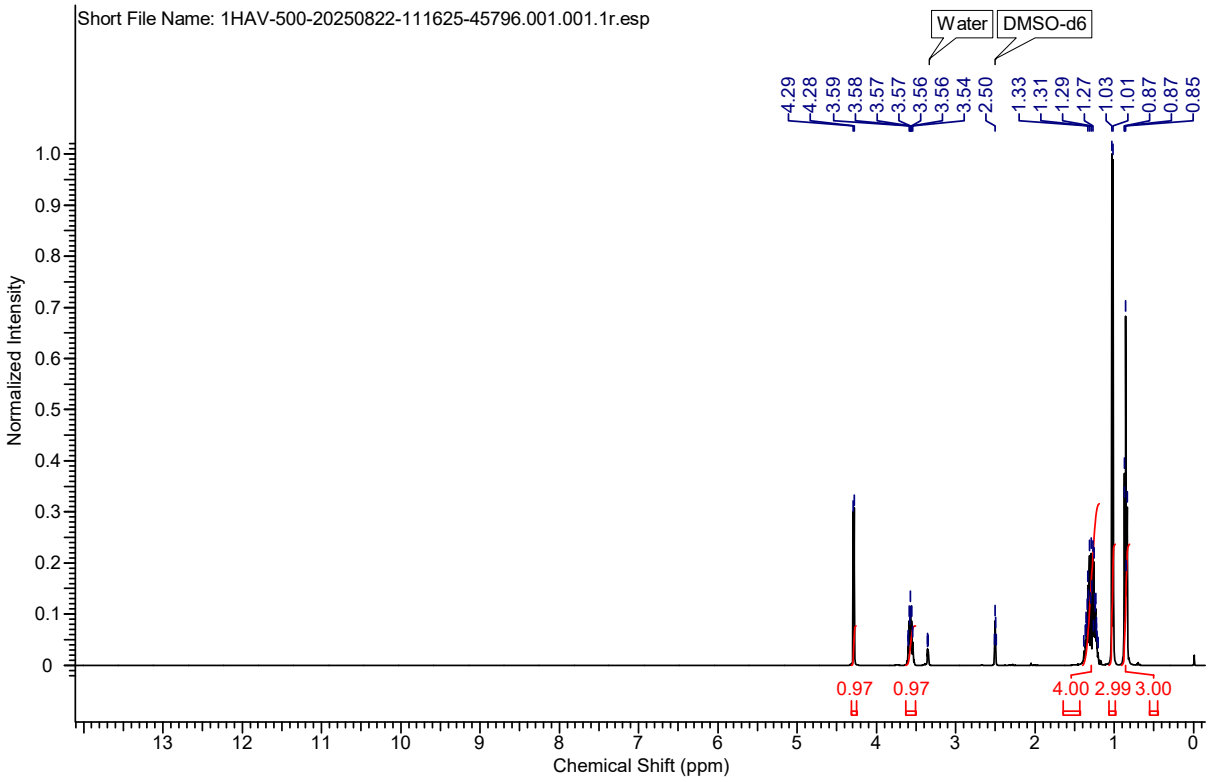
13C NMR - 2,2,2-Trifluoro-1-phenylethanol :- (2j)



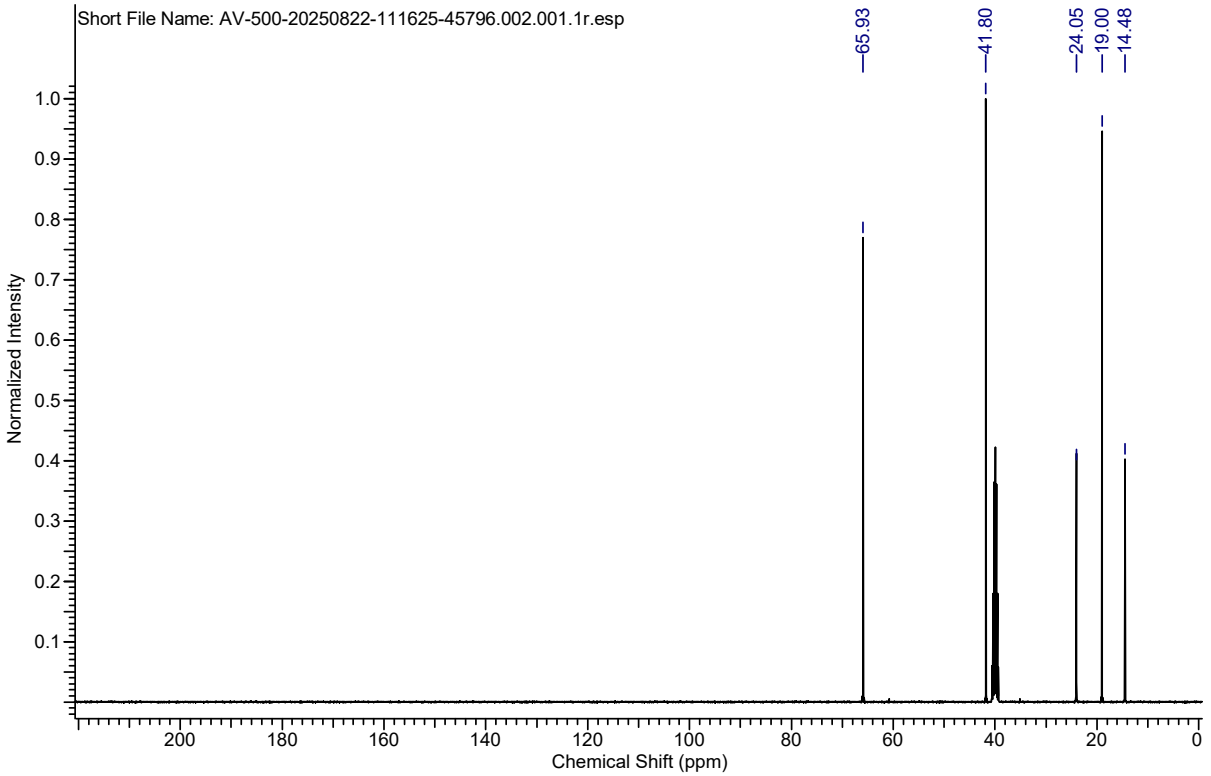
19F NMR - 2,2,2-Trifluoro-1-phenylethanol :- (2j)



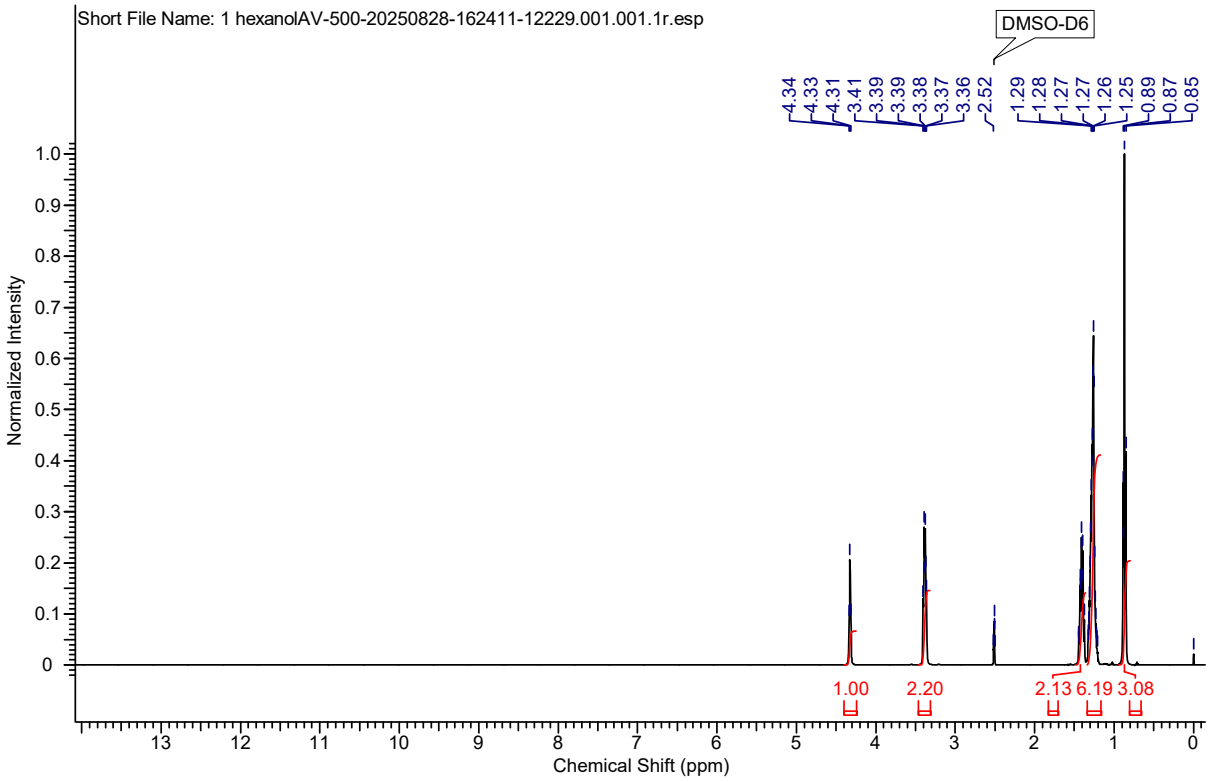
1H NMR of 2-Pentanol:- (2k)



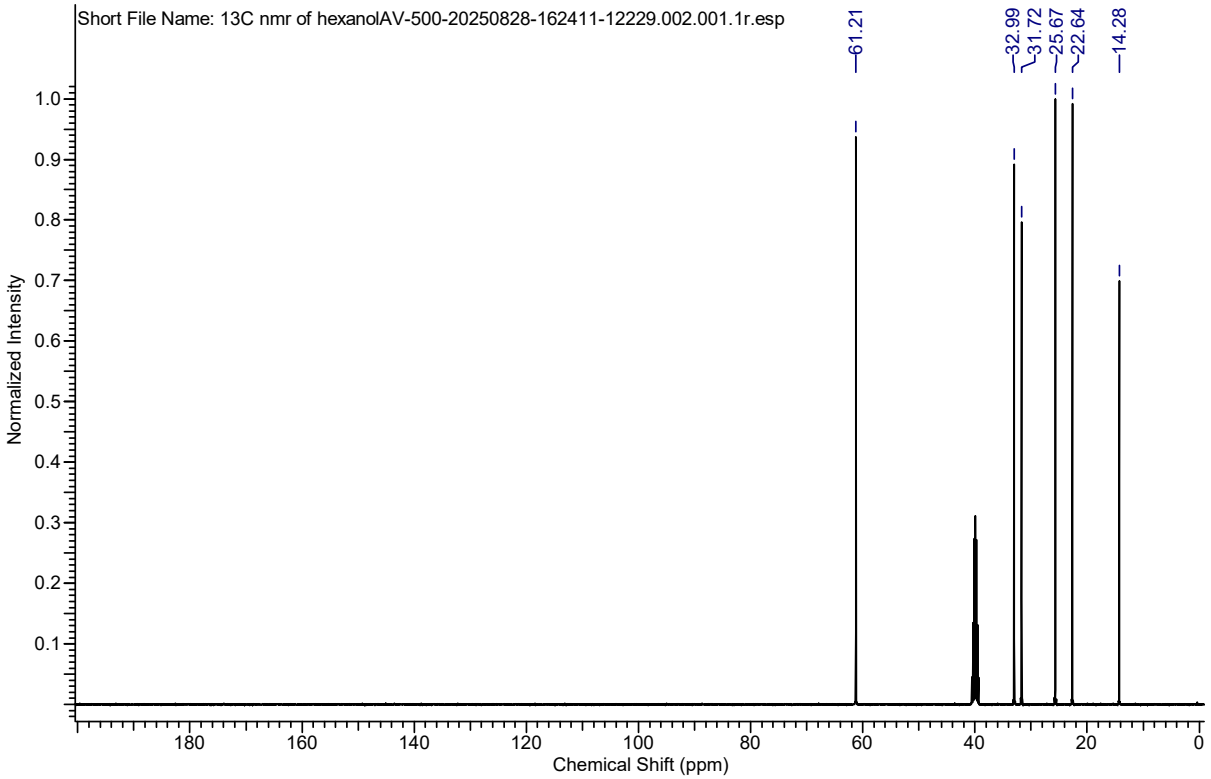
13C NMR of 2-Pentanol:- (2k)



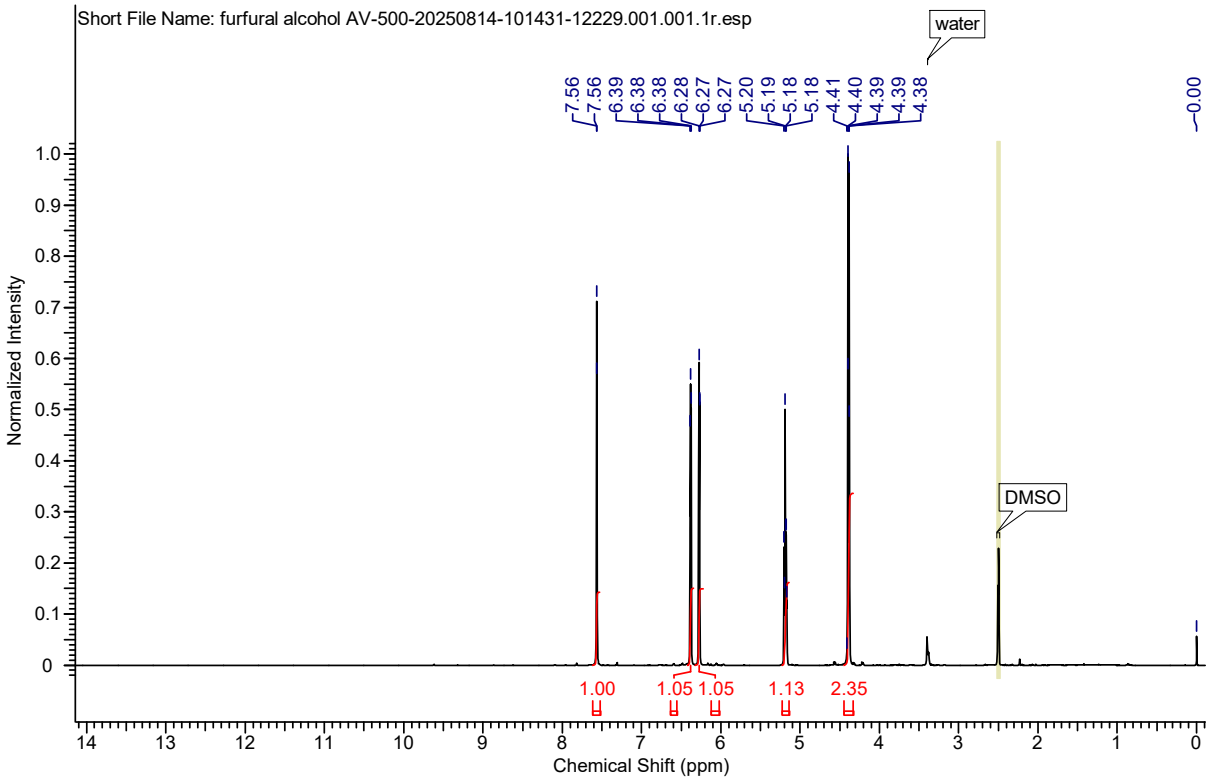
1H NMR of Hexanol:- (2L)



13C NMR of Hexanol :- (2L)



1H NMR Furfural alcohol:- (2M)



13C NMR Furfural alcohol:- (2M)