Electronic Supplementary Material (ESI) for Green Chemistry.

Supplemental Information

Antimony(V) Catalyzed Acetalisation of Aldehydes: An Efficient, Solvent-Free, and Recyclable Process

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2.) Procedure for testing recyclability of $[\mathbf{1}]^+$ S32

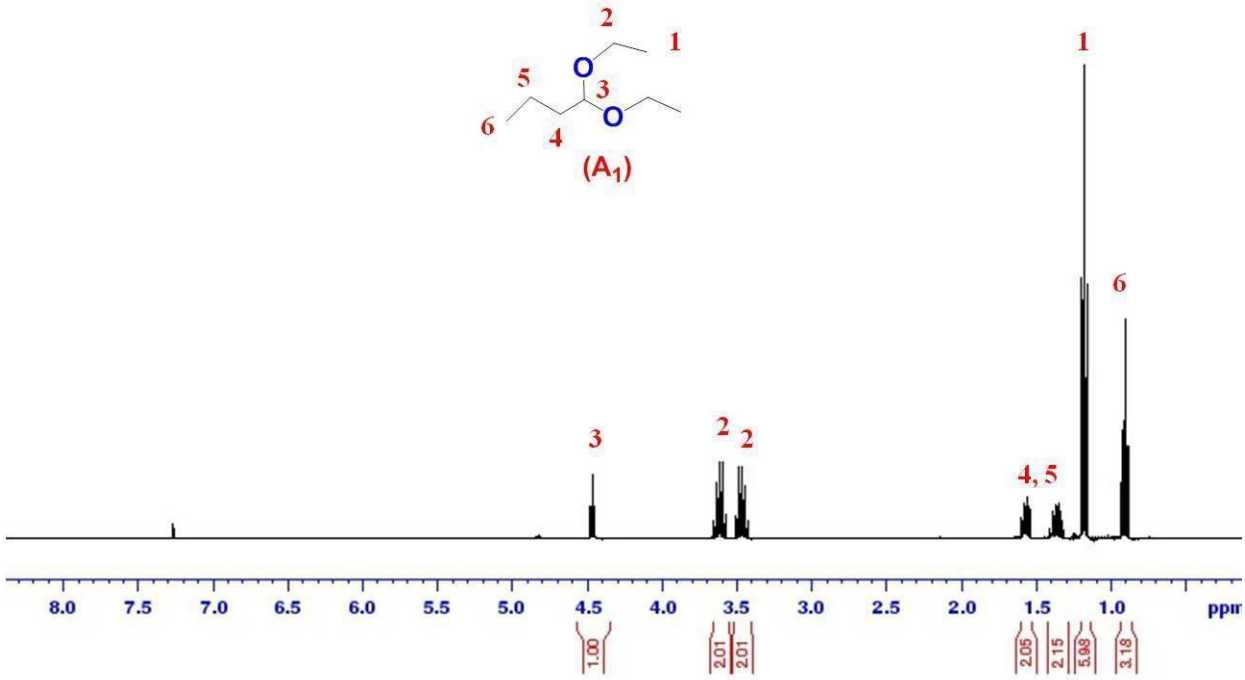

Figure S1: ^{1}H NMR spectroscopy of isolated butyraldehyde diethylacetal (A_1) in CDCl3

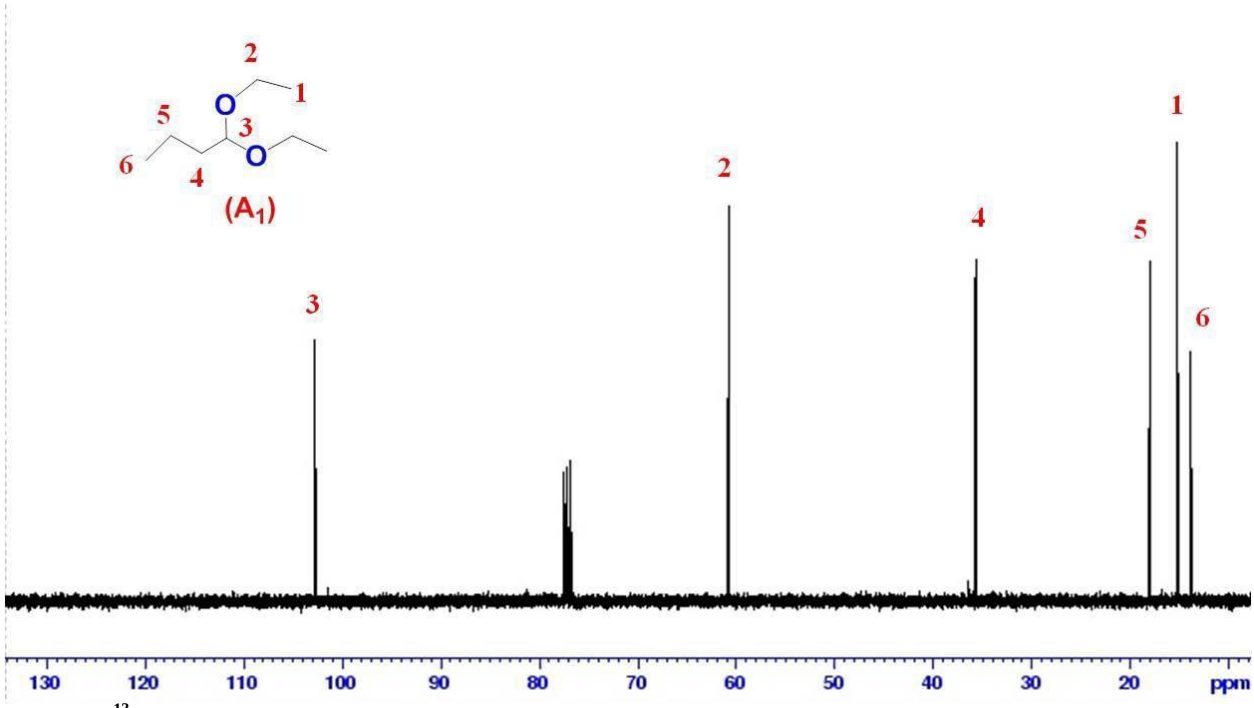

Figure S2: ^{13}H NMR spectroscopy of isolated butyraldehyde diethylacetal (A1) in $CDCl_3$

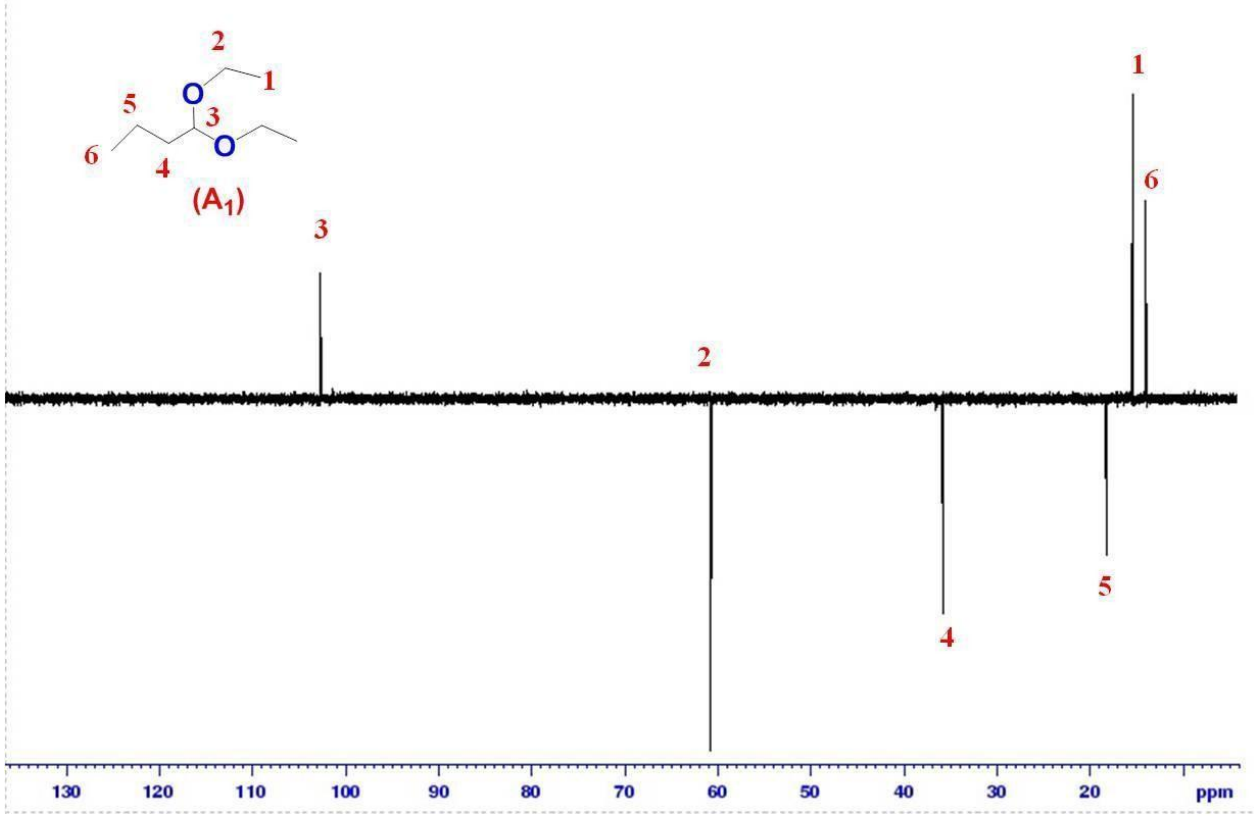

Figure S3: Dept 135 NMR spectroscopy of isolated butyraldehyde diethylacetal (A1) in $CDCl_3$

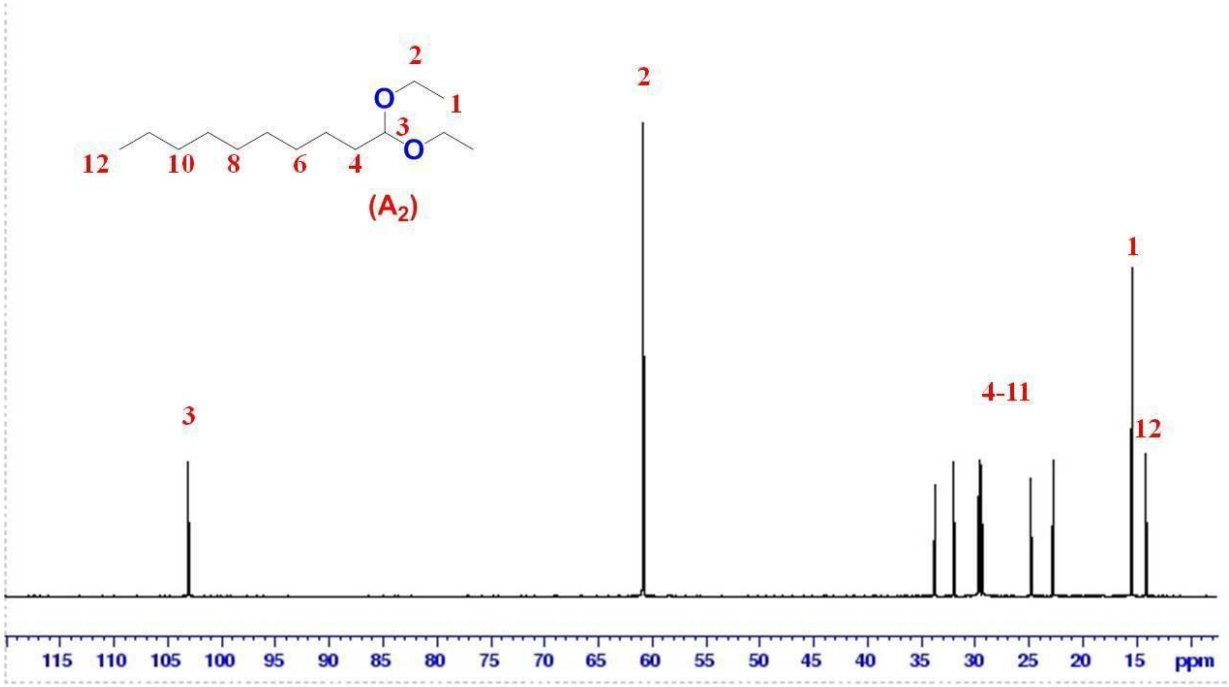

Figure S4: ^{13}C NMR Spectroscopy of isolated decanal diethylacetal (A2) in $CDCl_3$

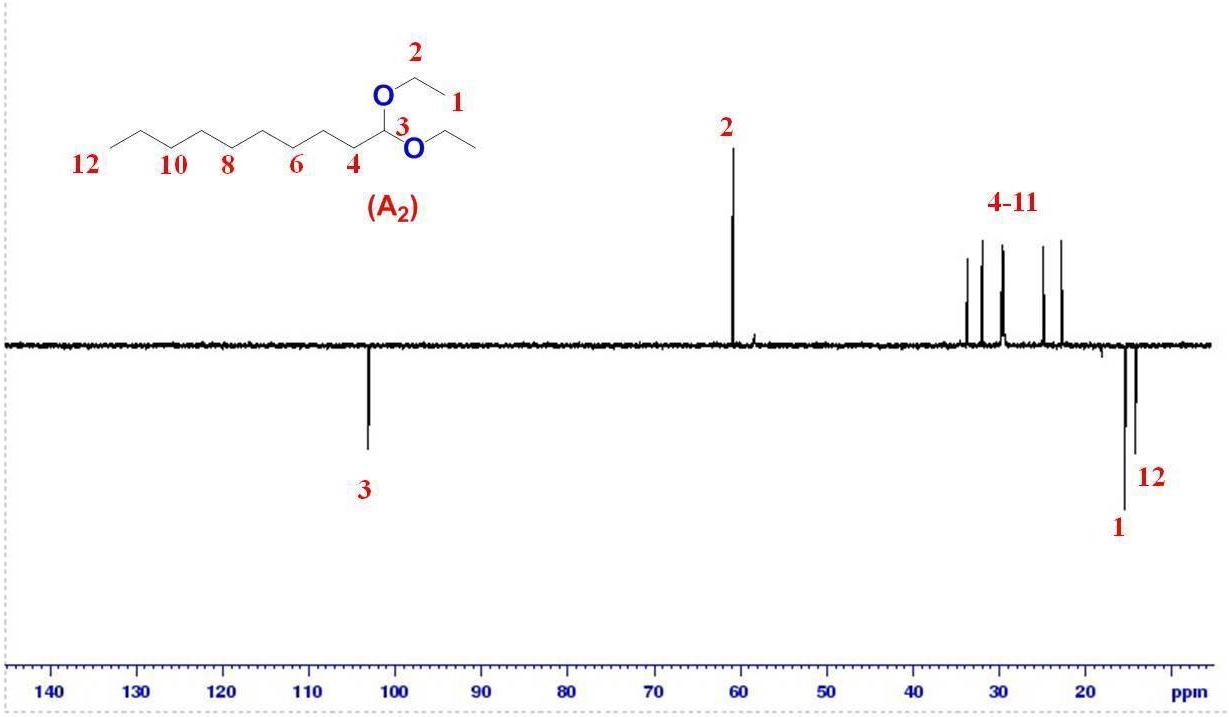

Figure S5: Dept 135 NMR Spectroscopy of isolated decanal diethylacetal (A2) in $CDCl_3$

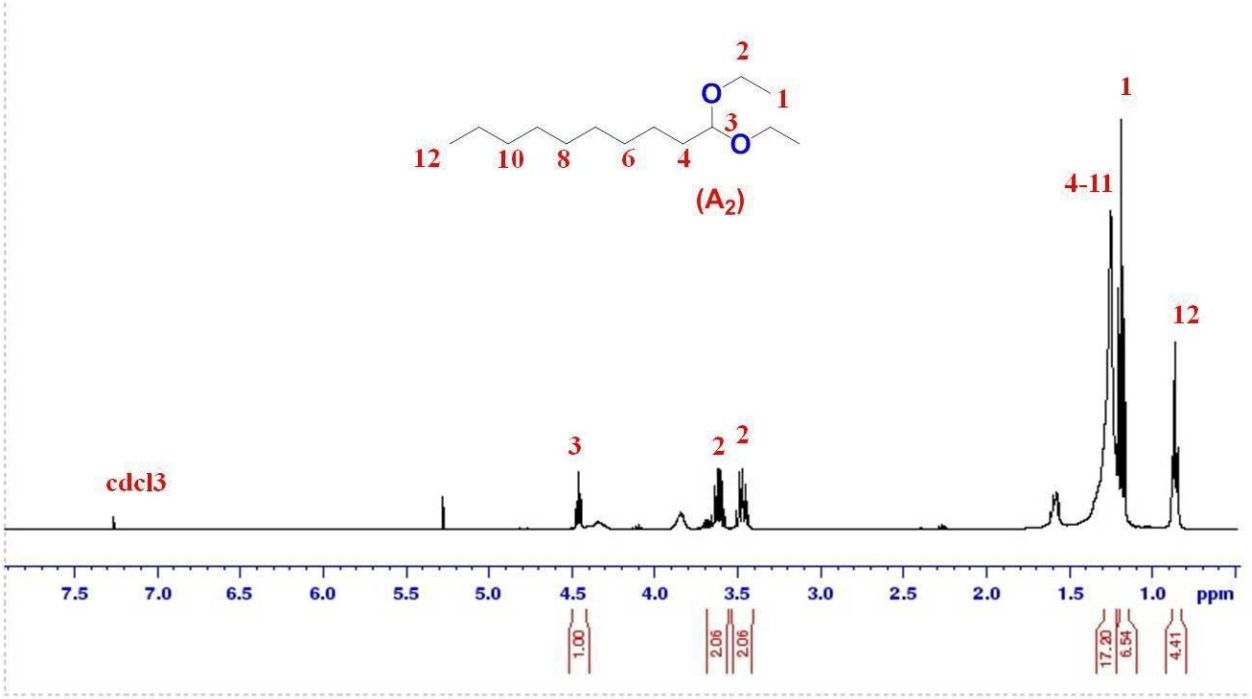

Figure S6: ^{1}H NMR Spectroscopy of isolated decanal diethylacetal (A_2) in $CDCl_3$

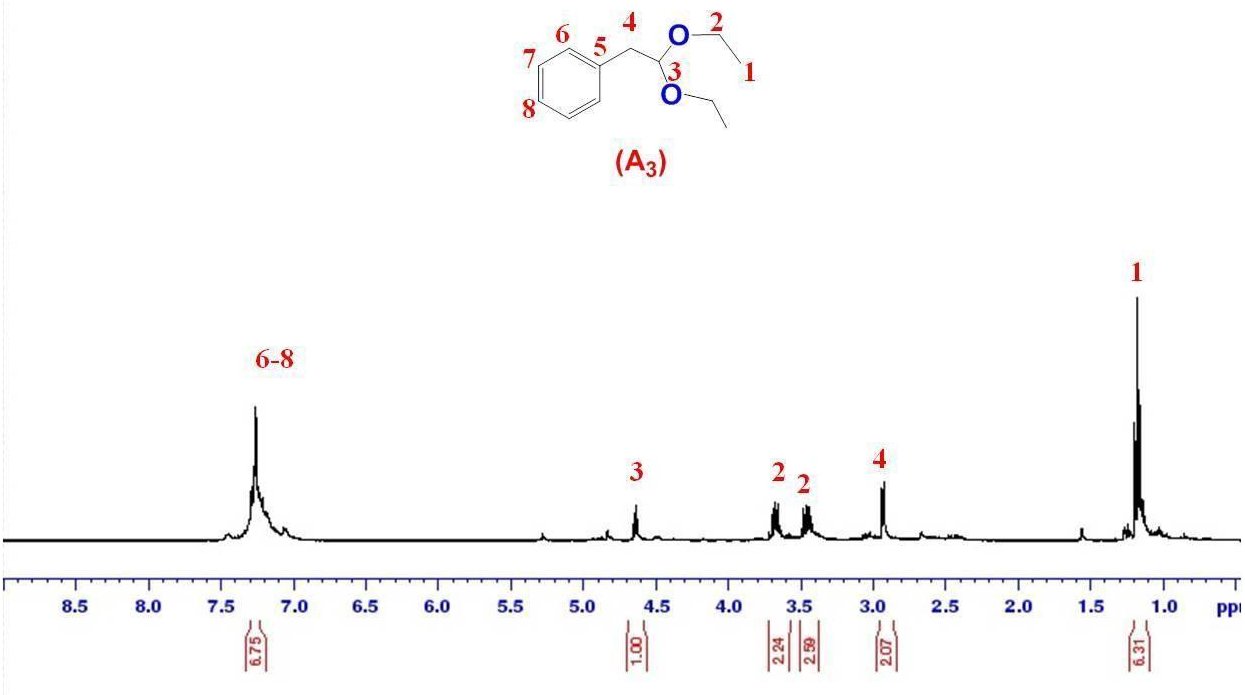

Figure S7: ^{1}H NMR Spectroscopy of isolated 2-phenylethanaldehyde diethylacetal (A_3) in $CDCl_3$

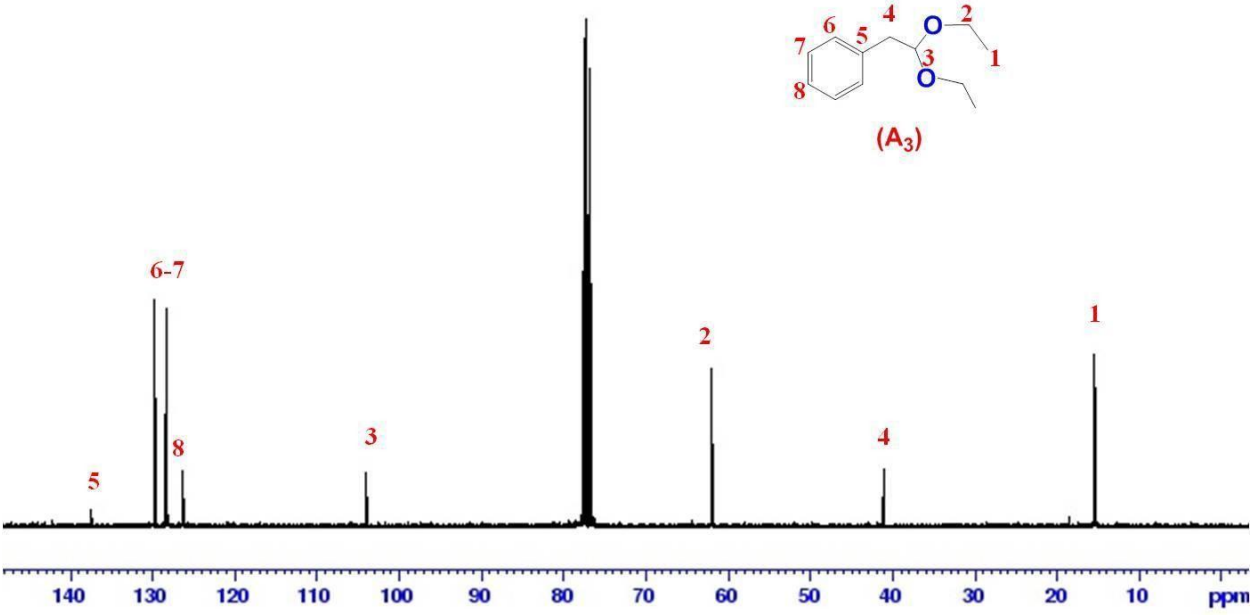

Figure S8: ^{13}C NMR Spectroscopy of isolated 2-phenylethanaldehyde diethylacetal (A3) in $CDCl_3$

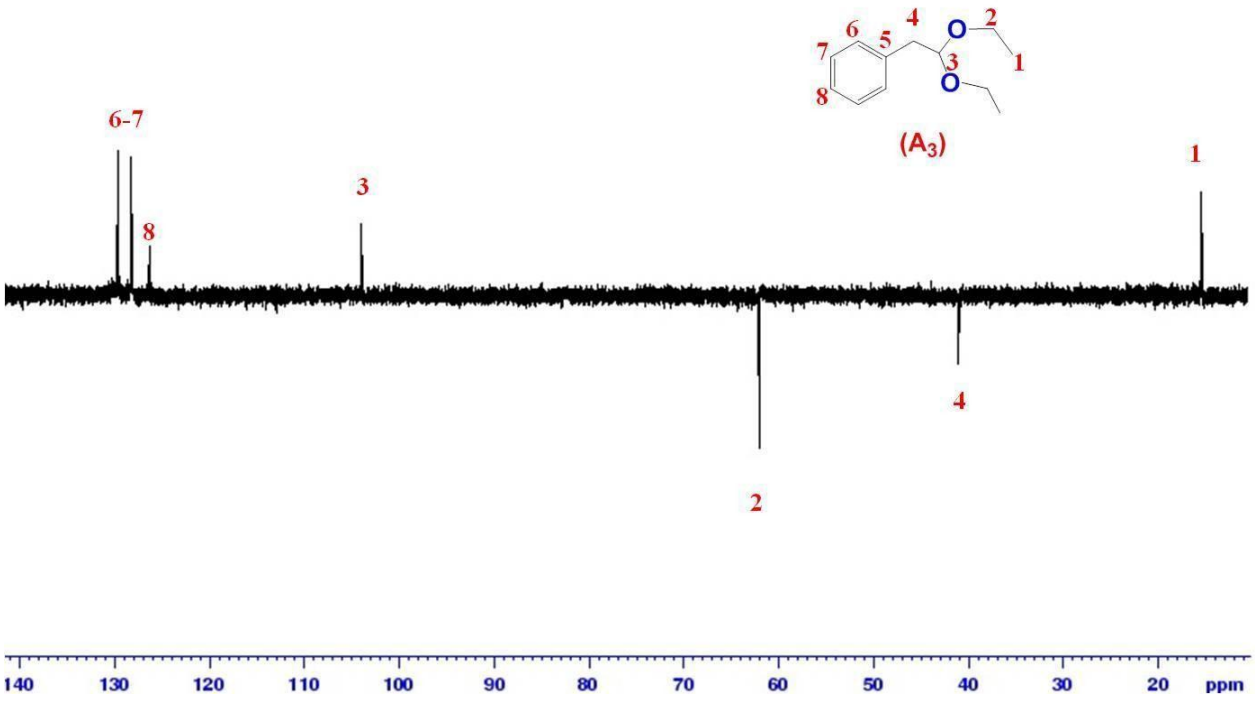

Figure S9: Dept 135 NMR Spectroscopy of isolated 2-phenylethanaldehyde diethylacetal (A_3) in $CDCl_3$

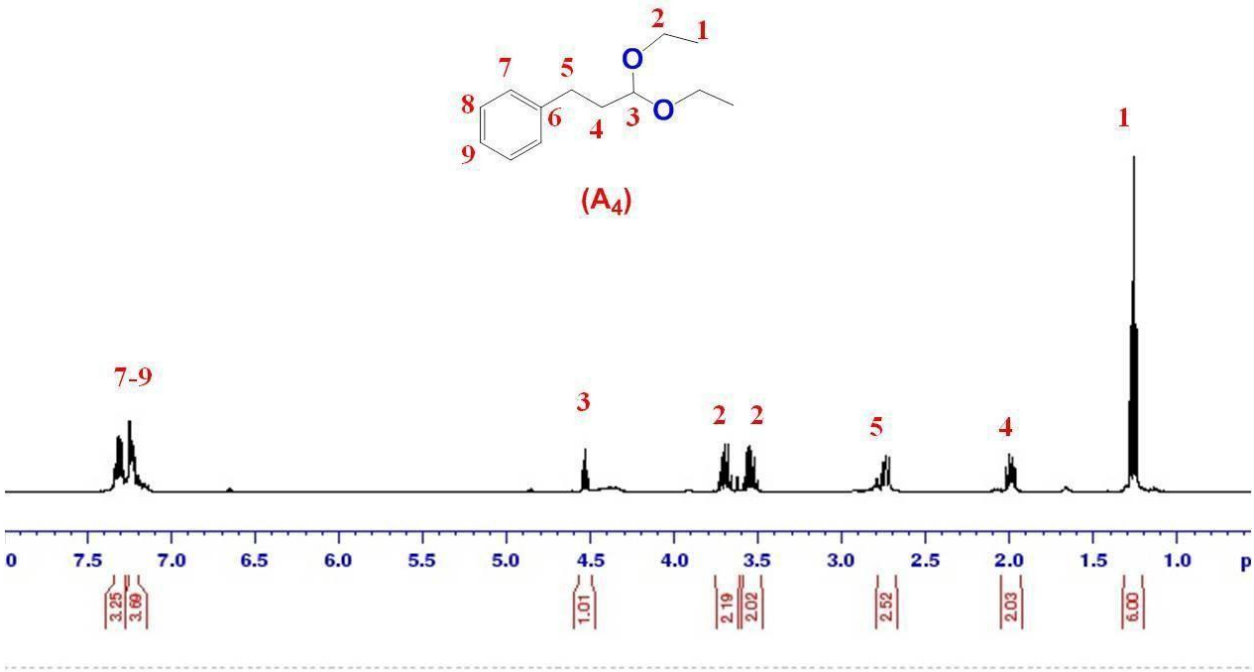

Figure S10: ^1H NMR Spectroscopy of isolated 3-phenylpropionaldehyde diethylacetal (A_4) in $CDCl_3$

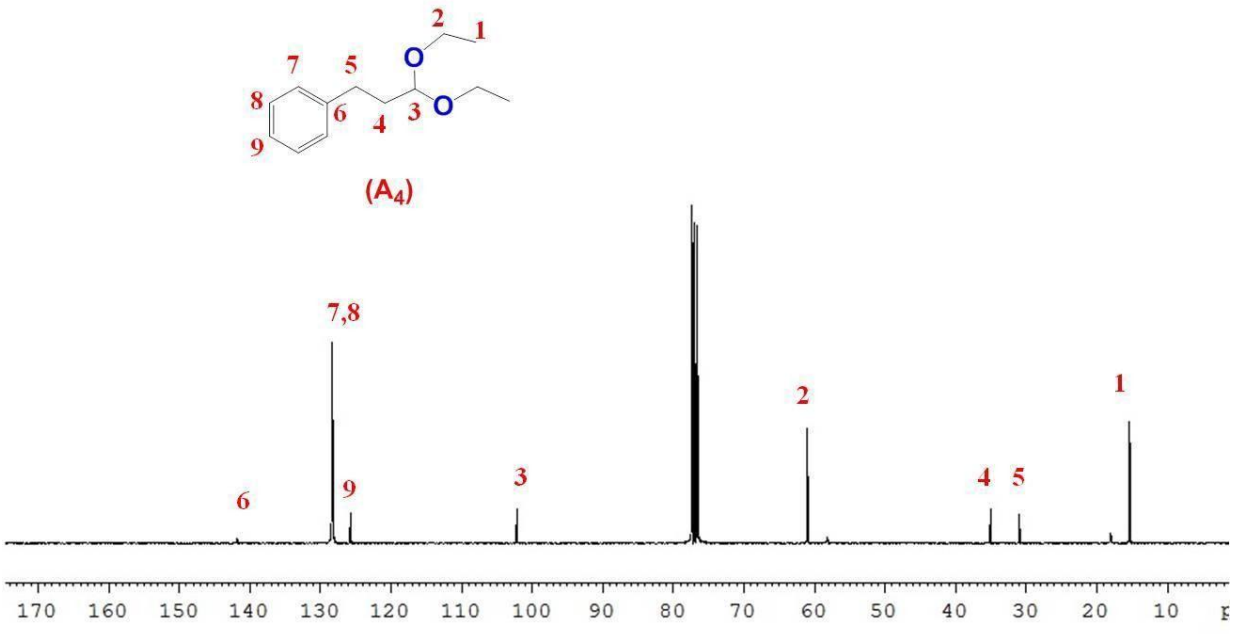

Figure S11: ^{13}C NMR Spectroscopy of isolated 3-phenylpropionaldehyde diethylacetal (A_4) in $CDCl_3$

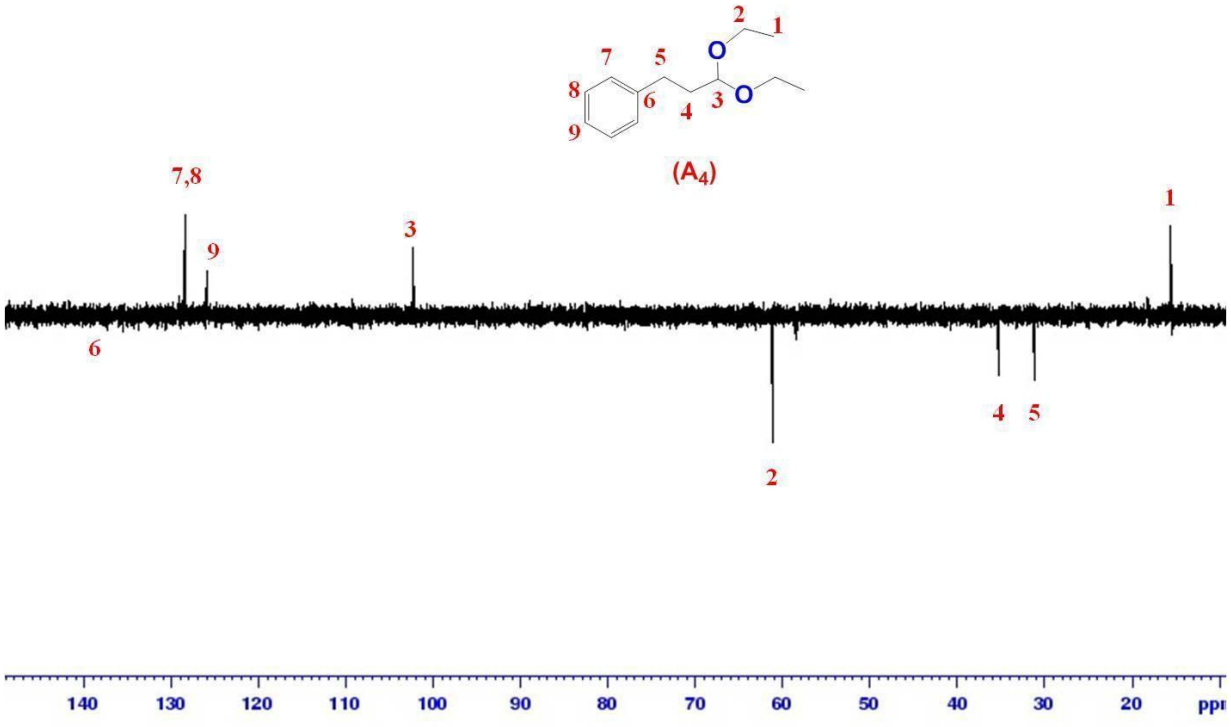

Figure S12: Dept 135 NMR Spectroscopy of isolated 3-phenylpropionaldehyde diethylacetal (A_4) in $CDCl_3$

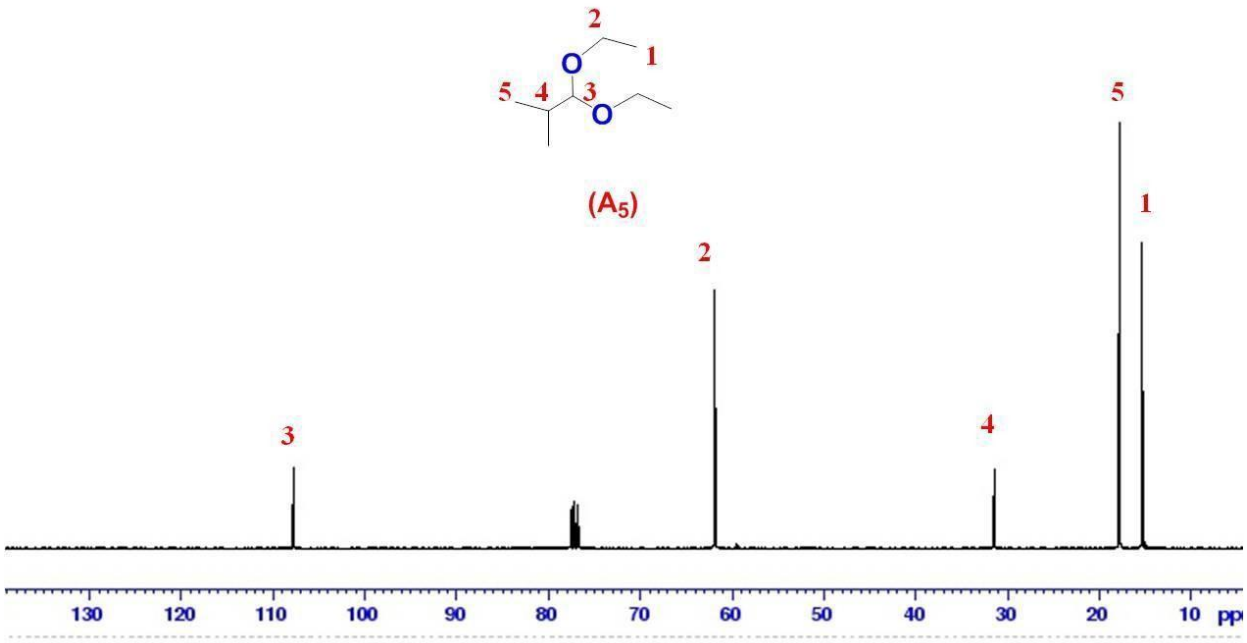

Figure S13: ^{13}C NMR Spectroscopy of isolated isobutyraldehyde diethylacetal (A_5) in $CDCl_3$

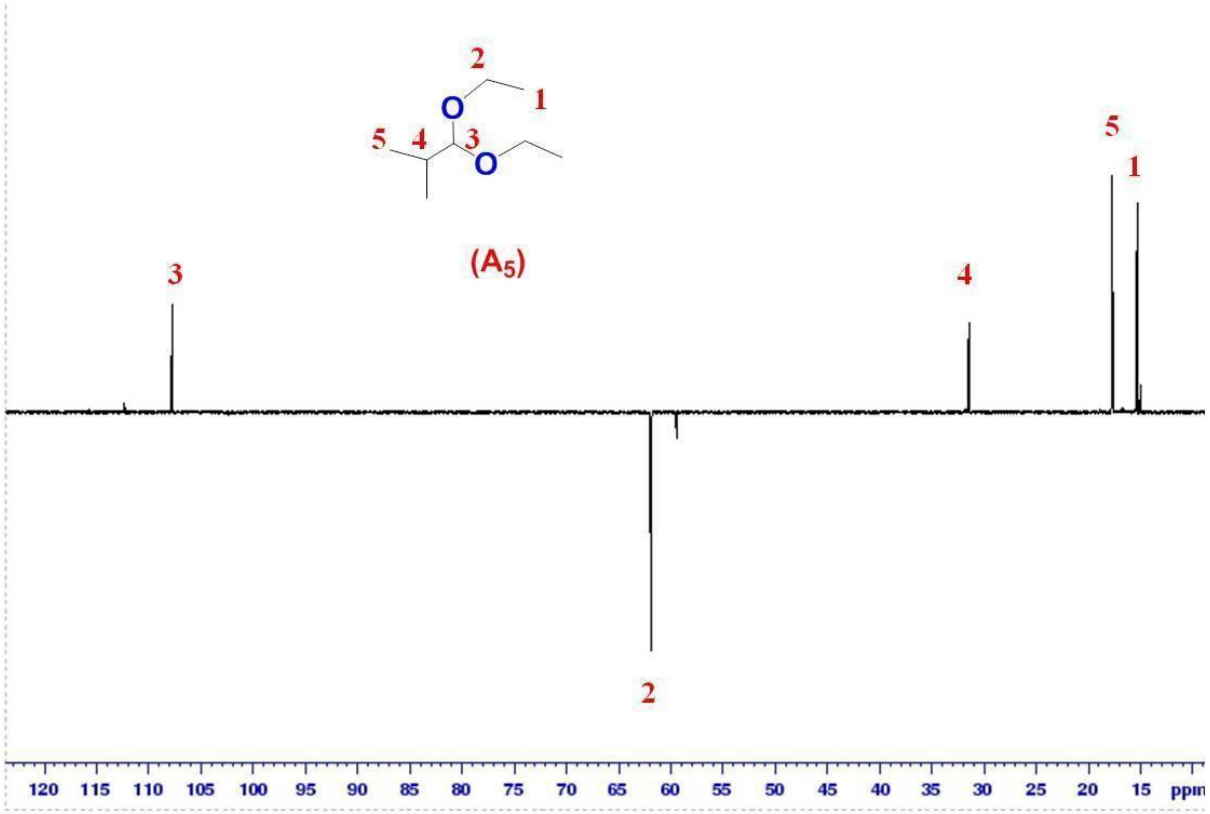

Figure S14: Dept 135 NMR Spectroscopy of isolated isobutyraldehyde diethylacetal (A_5) in $CDCl_3$

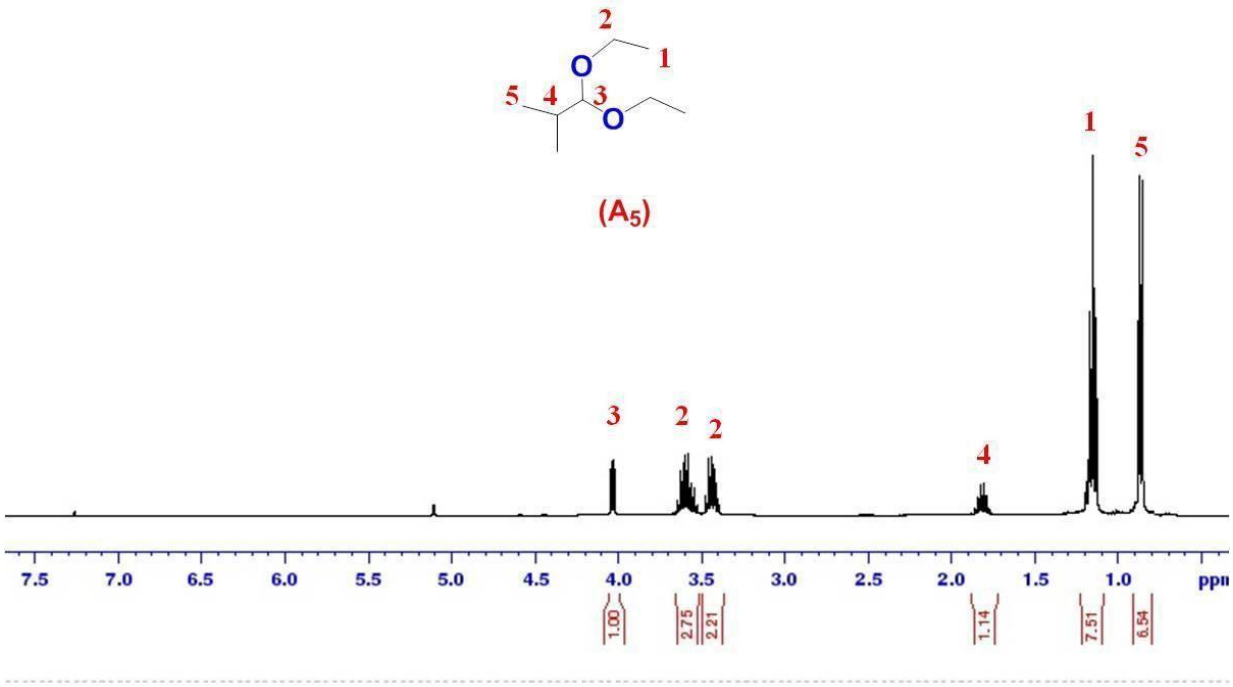

Figure S15: ^{1}HNMR Spectroscopy of isolated isobutyraldehyde diethylacetal (A_5) in $CDCl_3$

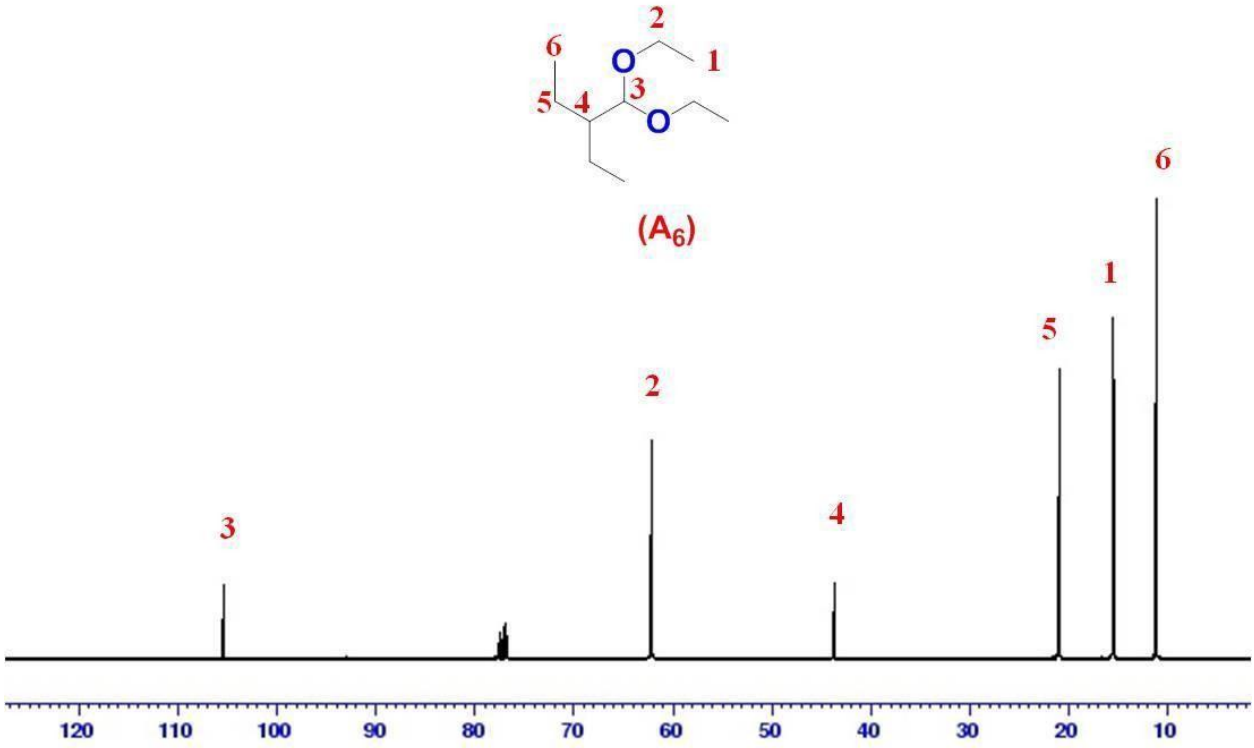

Figure S16: ^{13}C NMR Spectroscopy of isolated butyraldehyde 2-ethyl diethylacetal (A_6) in $CDCl_3$

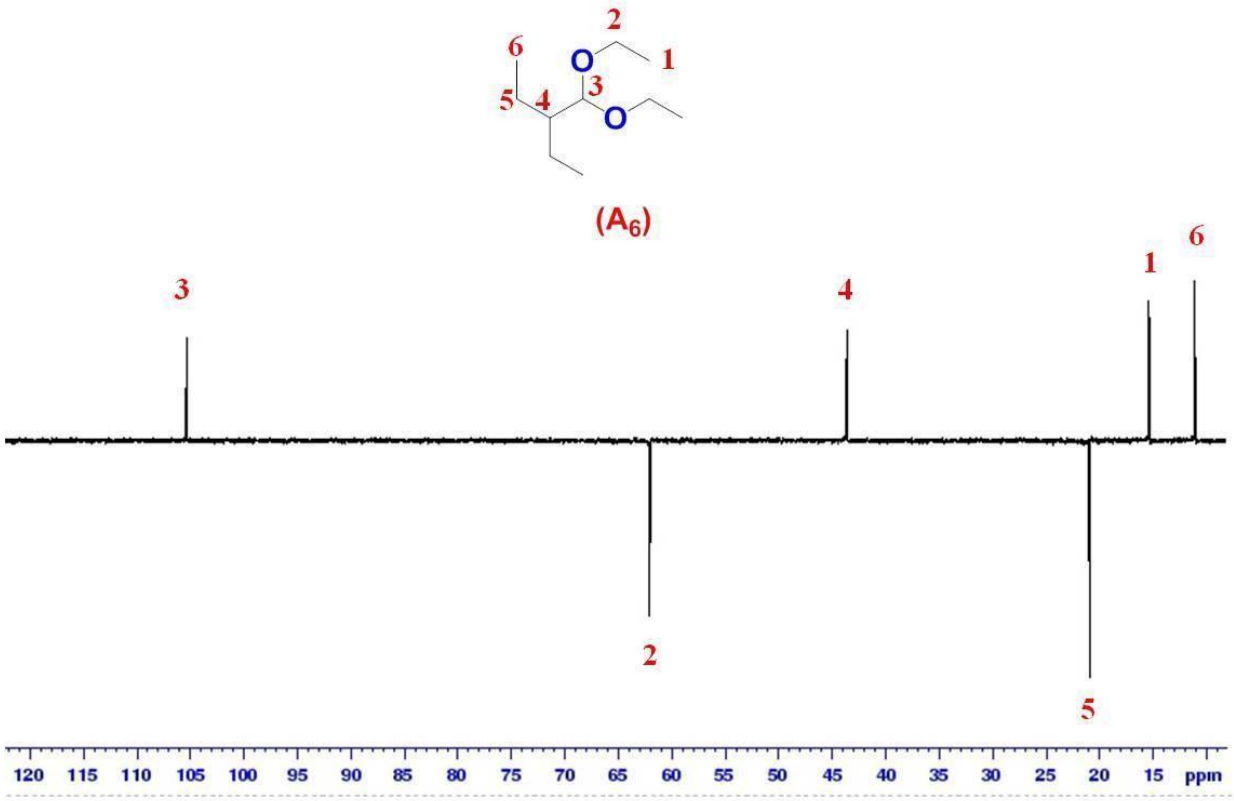

Figure S17: Dept 135 NMR Spectroscopy of isolated butyraldehyde 2-ethyl diethylacetal (A_6) in $CDCl_3$

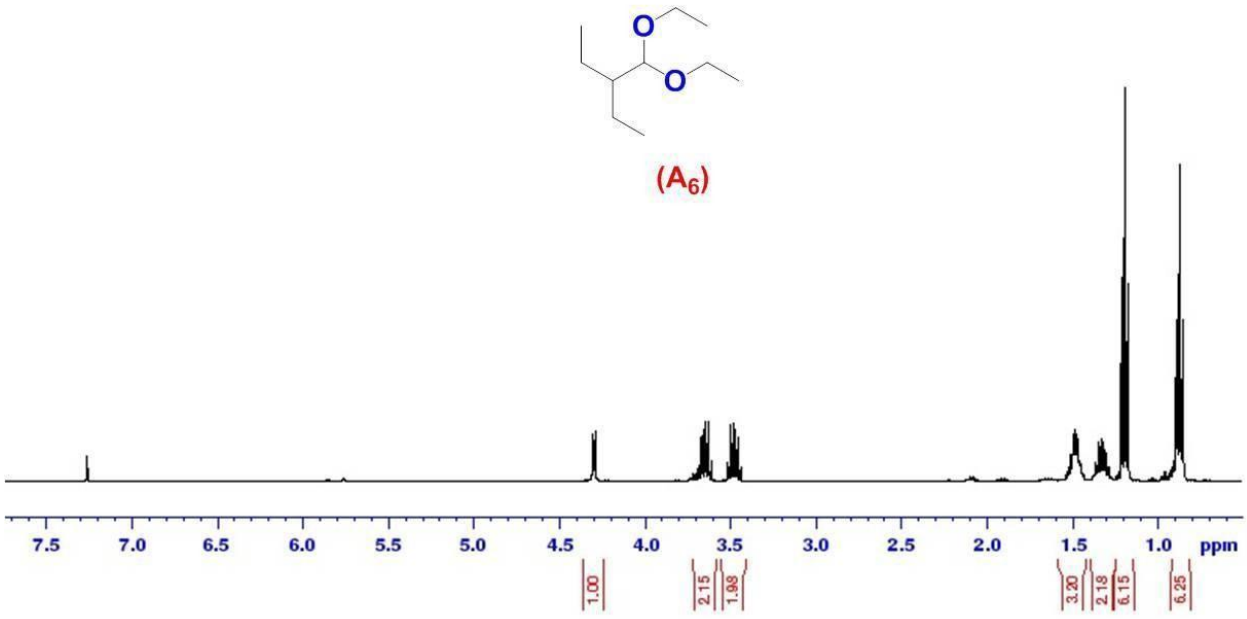

Figure S18: ^{1}H NMR Spectroscopy of isolated butyraldehyde 2-ethyl diethylacetal (A_6) in $CDCl_3$

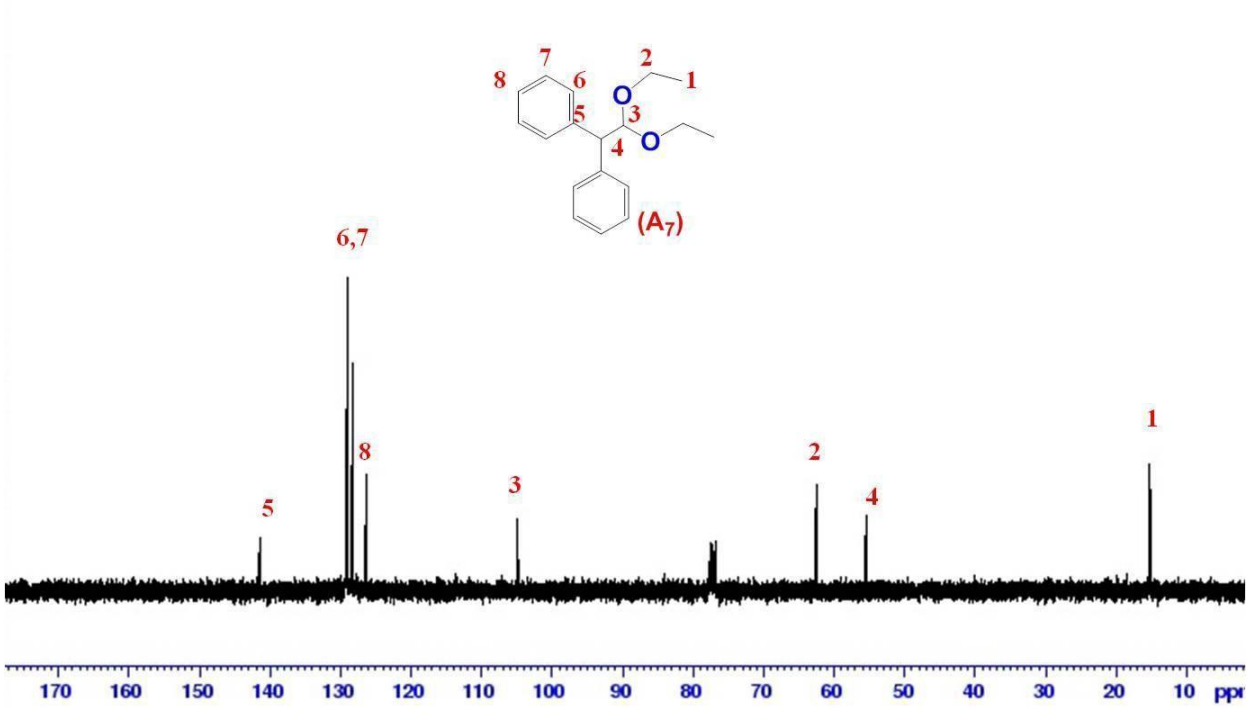

Figure S19: ^{13}C NMR Spectroscopy of isolated 2,2 dipheylacetaldehyde diethylacetal (A_7) in $CDCl_3$

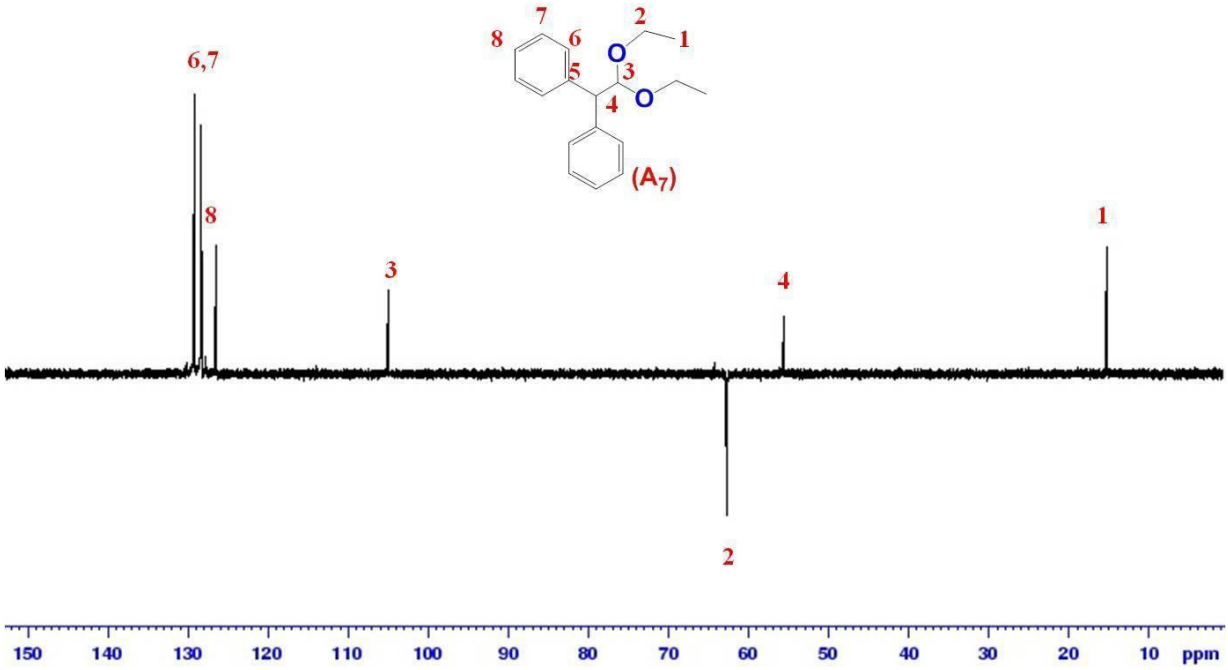

Figure S20: Dept 135 NMR Spectroscopy of isolated 2,2 dipheylacetaldehyde diethylacetal (A_7) in $CDCl_3$

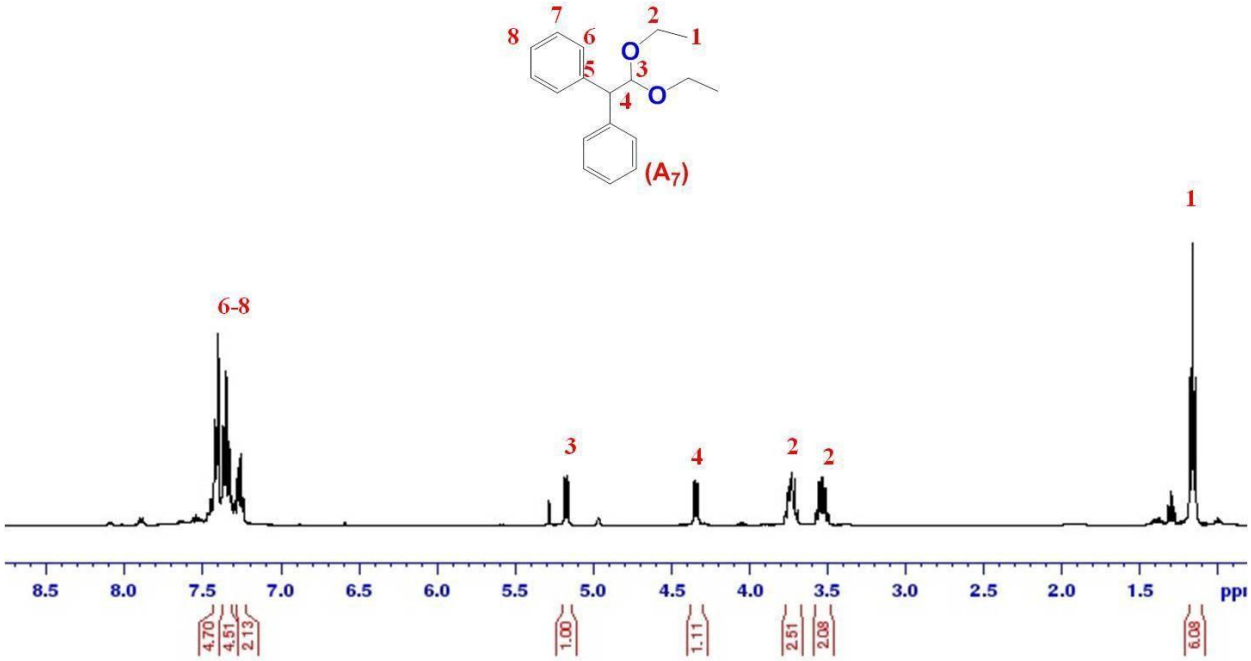

Figure S21: ^{1}H NMR Spectroscopy of isolated 2,2 dipheylacetaldehyde diethylacetal (A_7) in $CDCl_3$

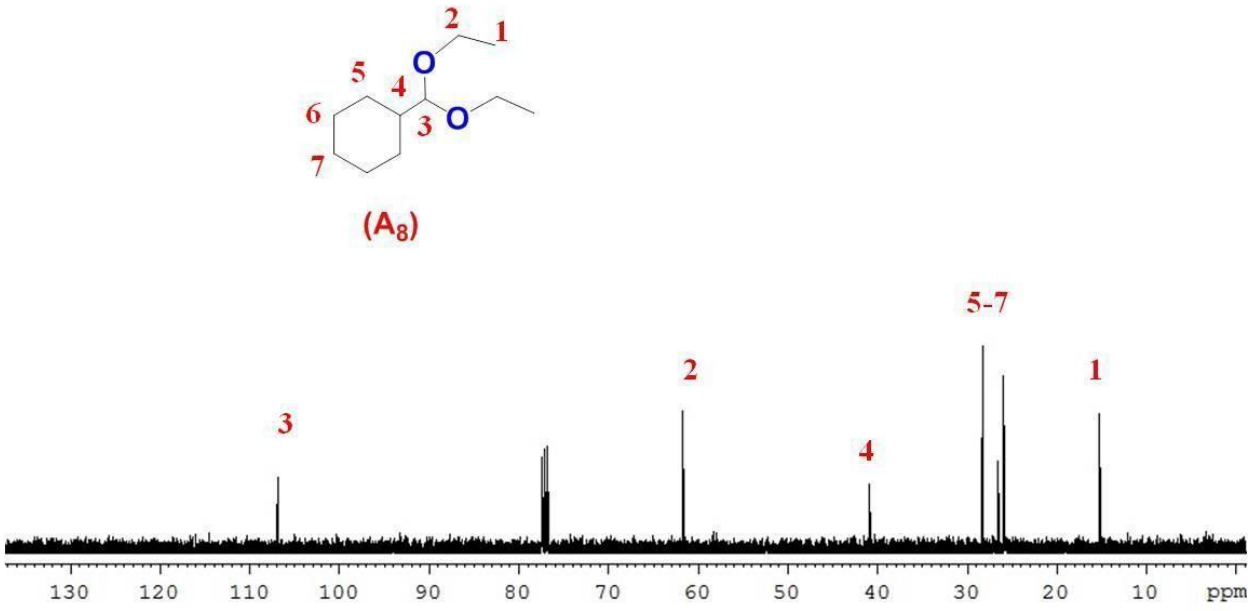

Figure S22: ^{13}C NMR Spectroscopy isolated cyclohexanecarboxaldehyde diethylacetal (A_8)

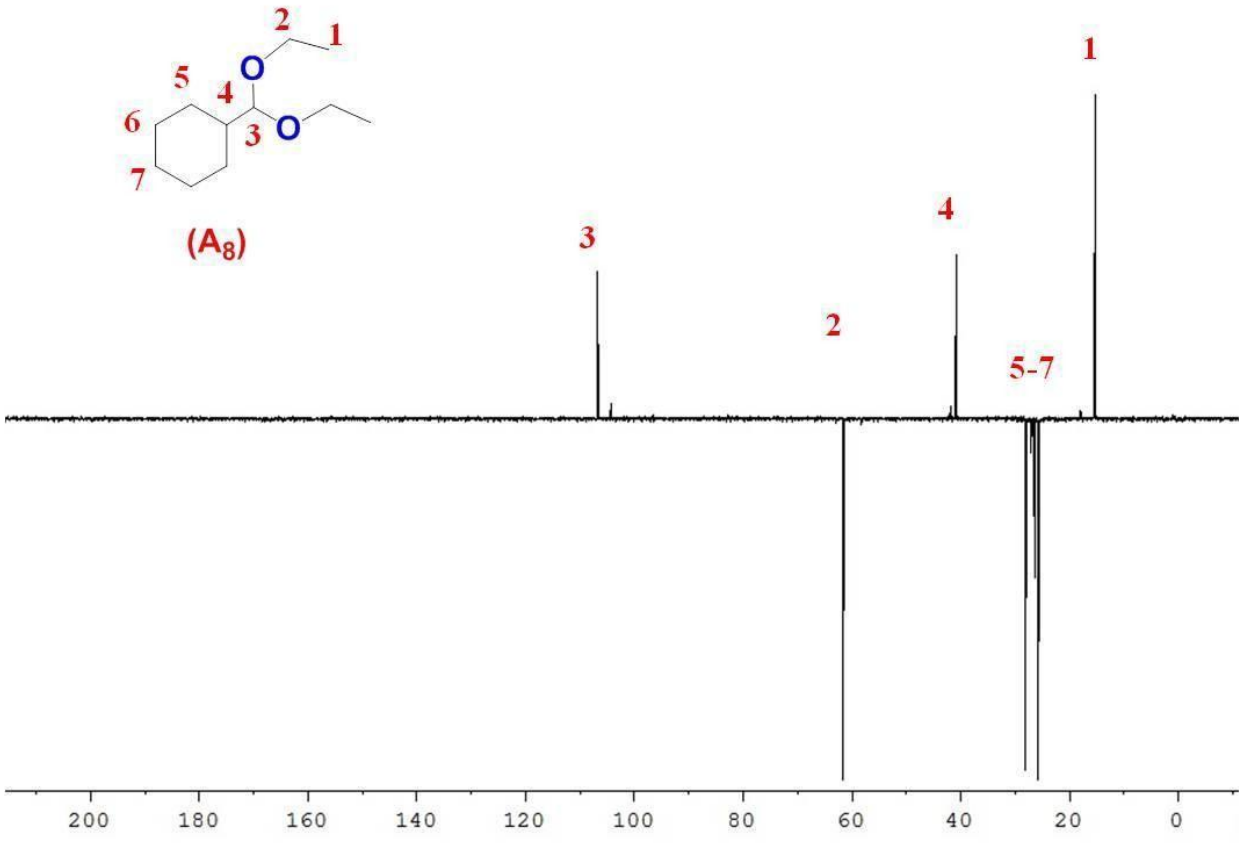

Figure S23: Dept 135 NMR Spectroscopy isolated cyclohexanecarboxaldehyde diethylacetal (A_8) in $CDCl_3$

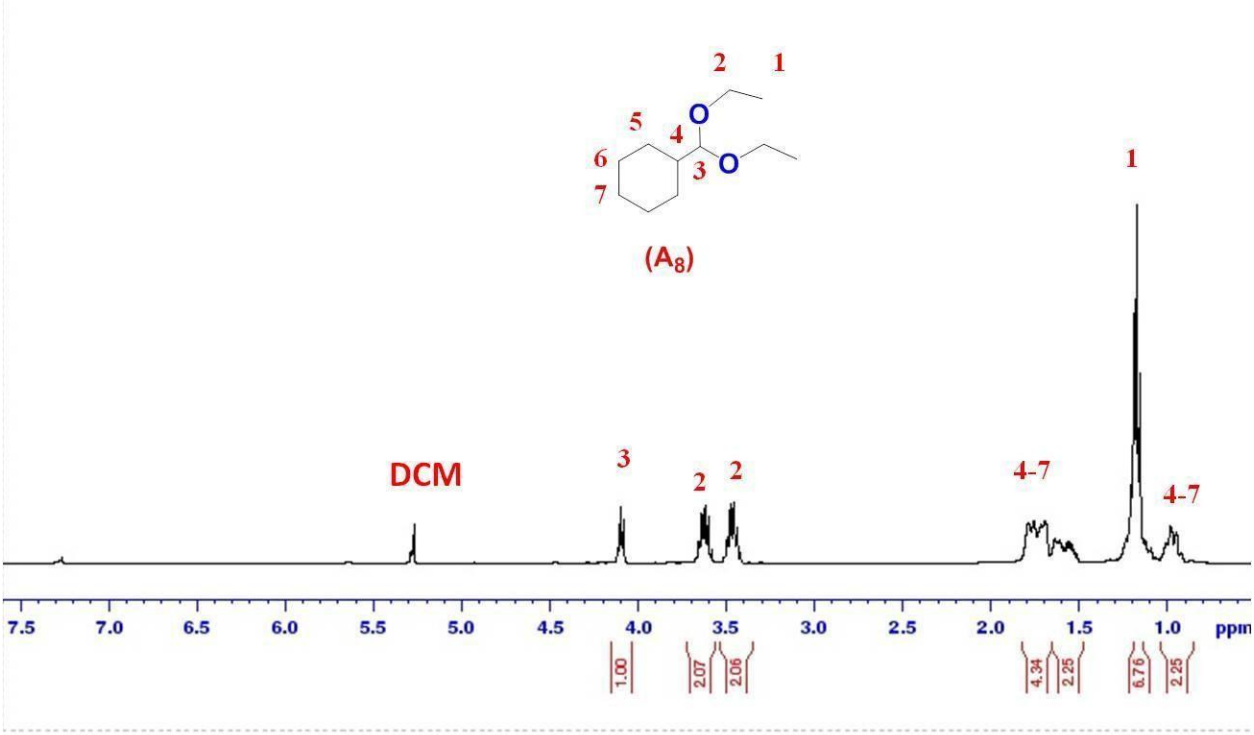

Figure S24: ^{1}H NMR Spectroscopy isolated cyclohexanecarboxaldehyde diethylacetal (A_8) in $CDCl_3$

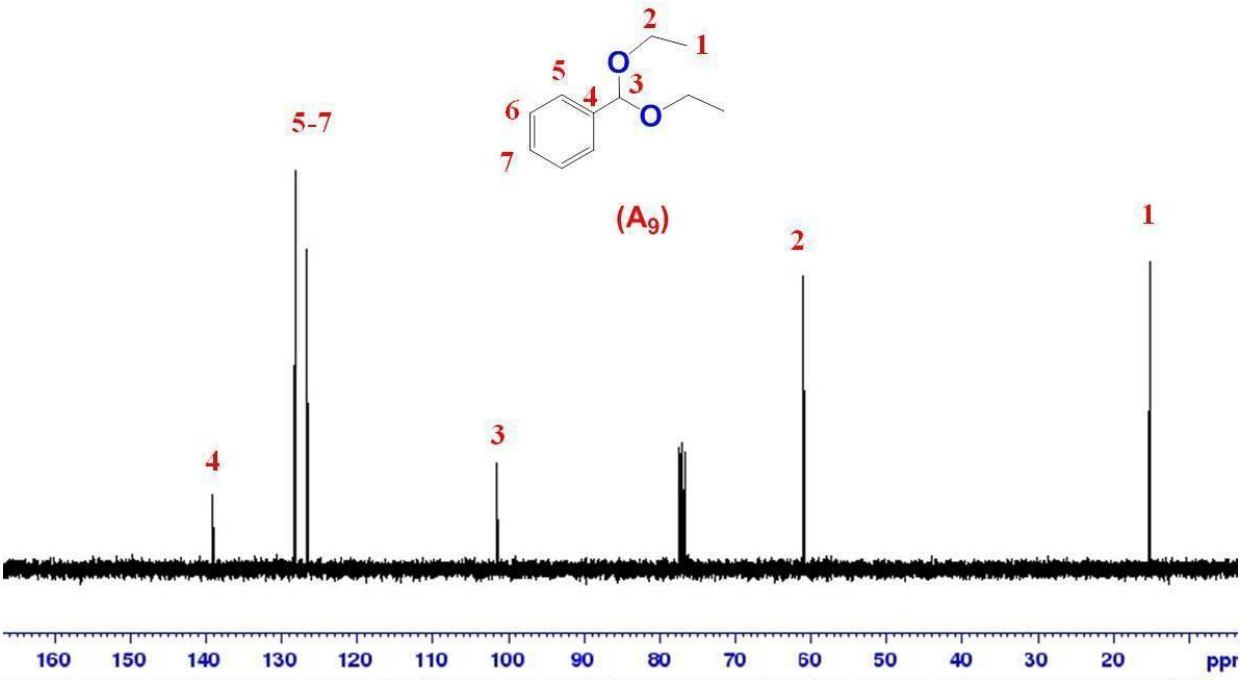

Figure S25: ^{13}C NMR Spectroscopy of isolated benzaldehyde diethylacetal (A_9) in $CDCL_3$

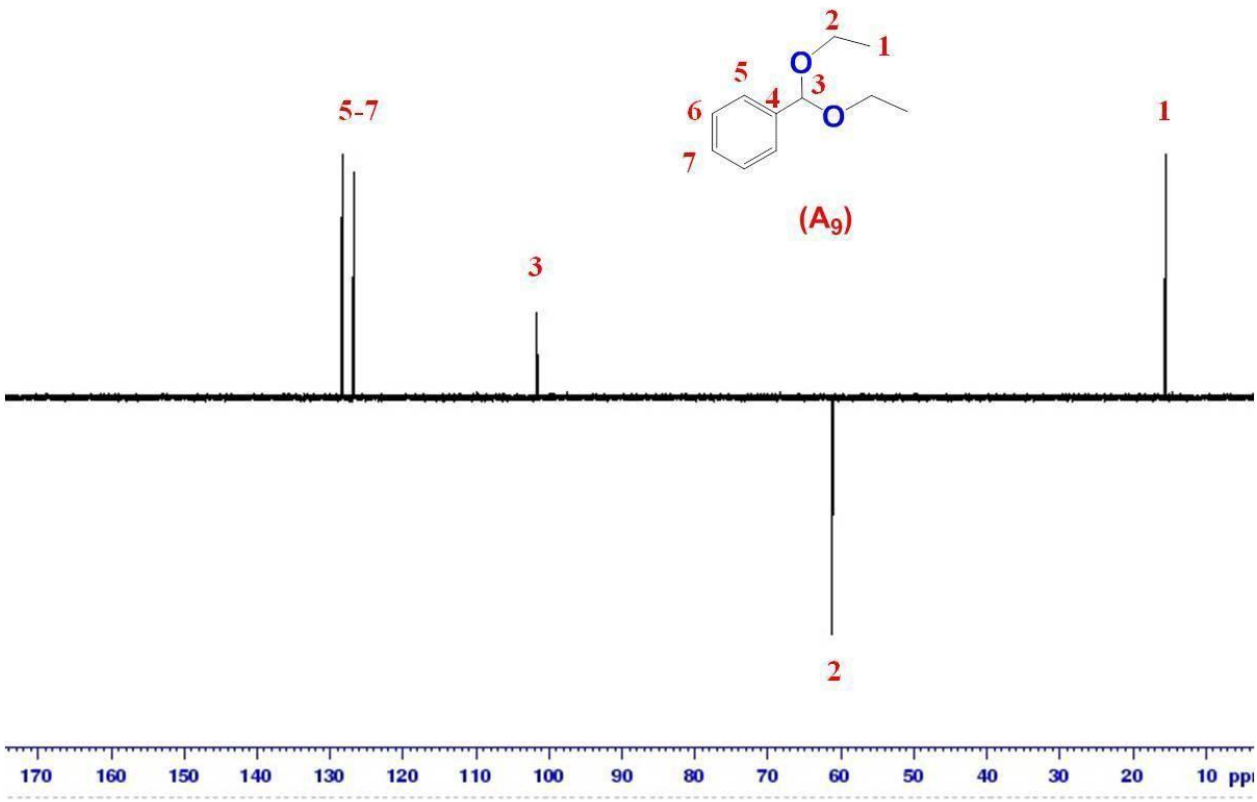

Figure S26: Dept 135 NMR Spectroscopy of isolated benzaldehyde diethylacetal (A_9) in $CDCL_3$

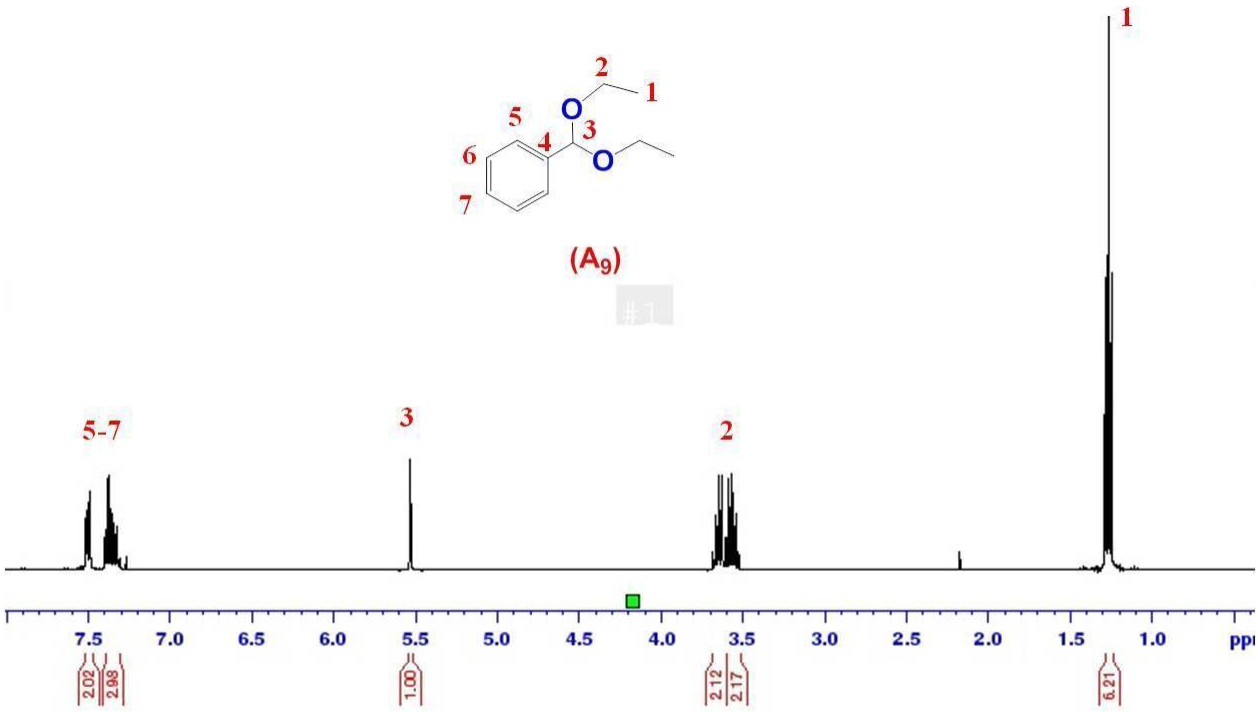

Figure S27: ^{1}H NMR Spectroscopy of isolated benzaldehyde diethylacetal (A_9) in $CDCL_3$

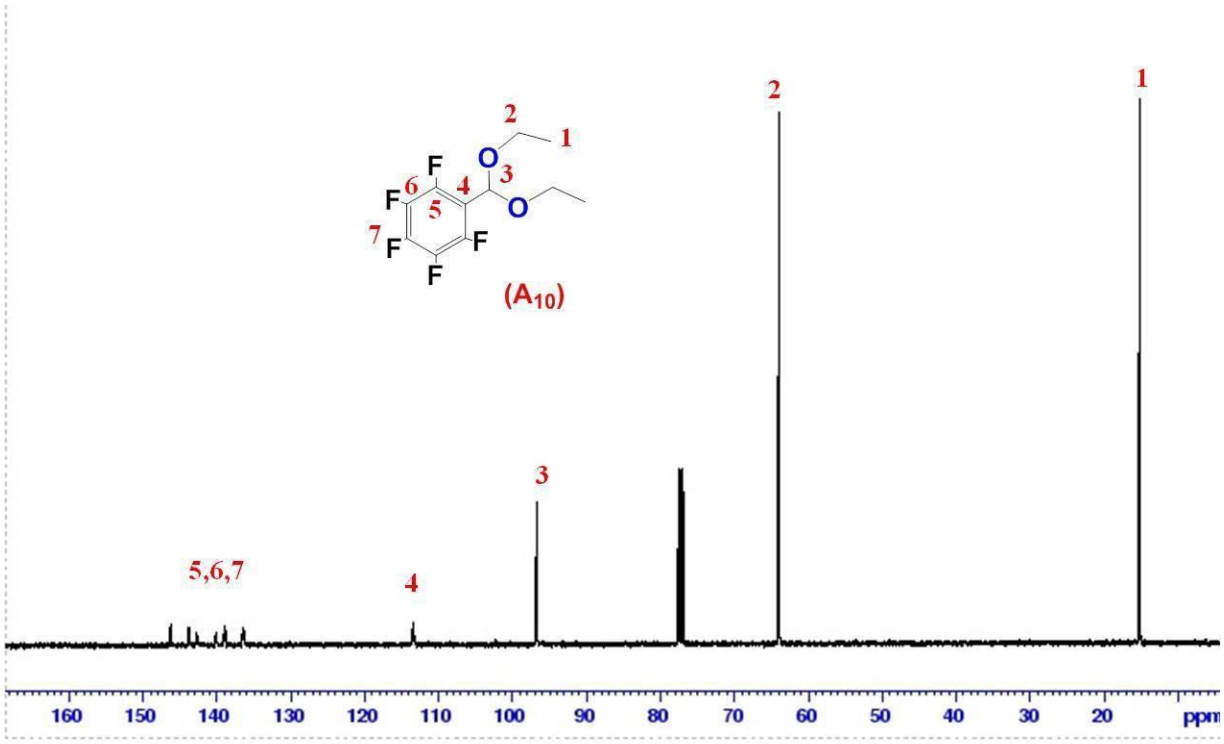

Figure S28: ^{13}C NMR Spectroscopy of new isolated 2,3,4,5,6 pentafluorobenzaldehyde diethylacetal (A_{10}) in $CDCl_3$

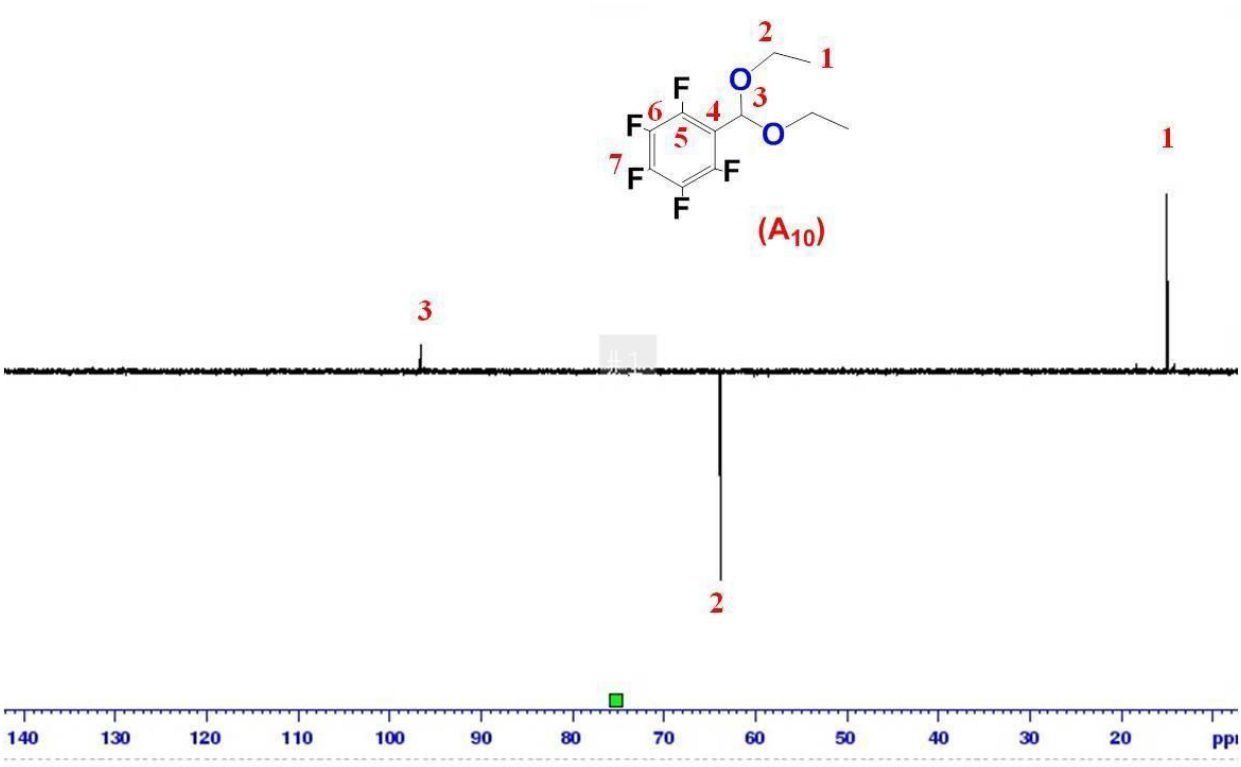

Figure S29: Dept 135 NMR Spectroscopy of new isolated 2,3,4,5,6 pentafluorobenzaldehyde diethylacetal (A_{10}) in $CDCl_3$

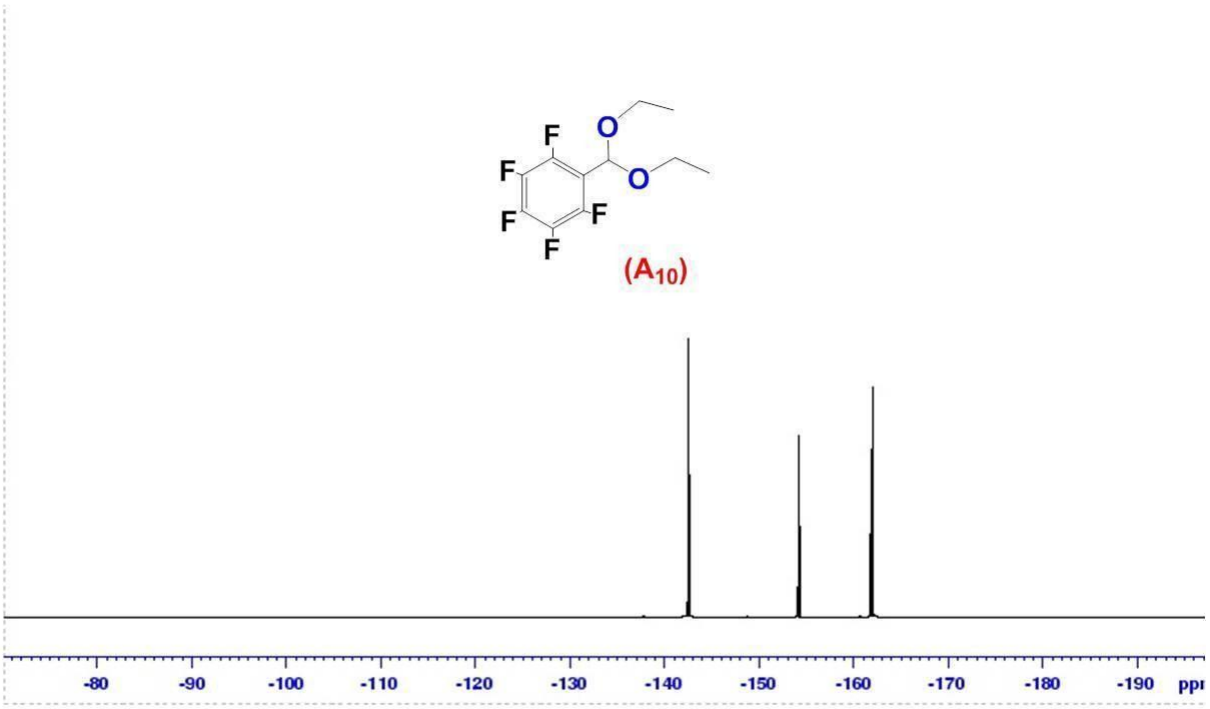

Figure S30: ^{19}F NMR Spectroscopy of new isolated 2,3,4,5,6 pentafluorobenzaldehyde diethylacetal (A_{10}) in $CDCl_3$

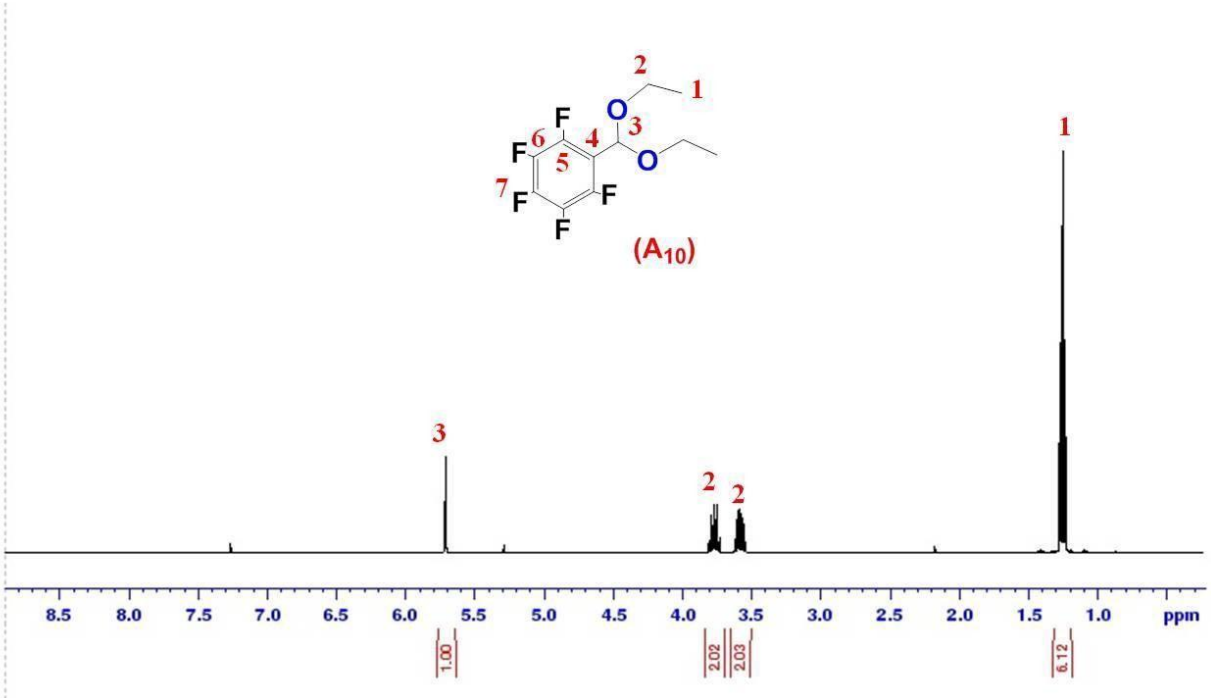

Figure S31: ^{1}H NMR Spectroscopy of new isolated 2,3,4,5,6 pentafluorobenzaldehyde diethylacetal (A_{10}) in $CDCl_3$

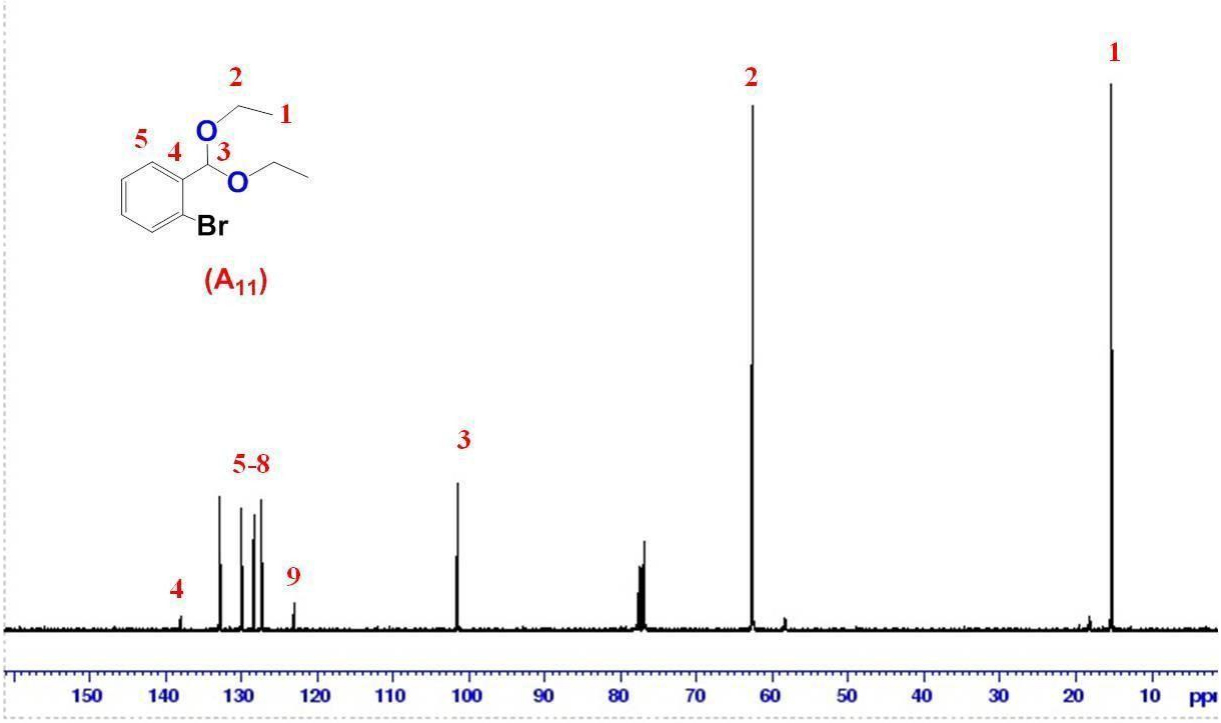

Figure S32: ^{13}C NMR Spectroscopy of isolated 2-bromobenzaldehydediethylacetal (A_{11}) in $CDCl_3$

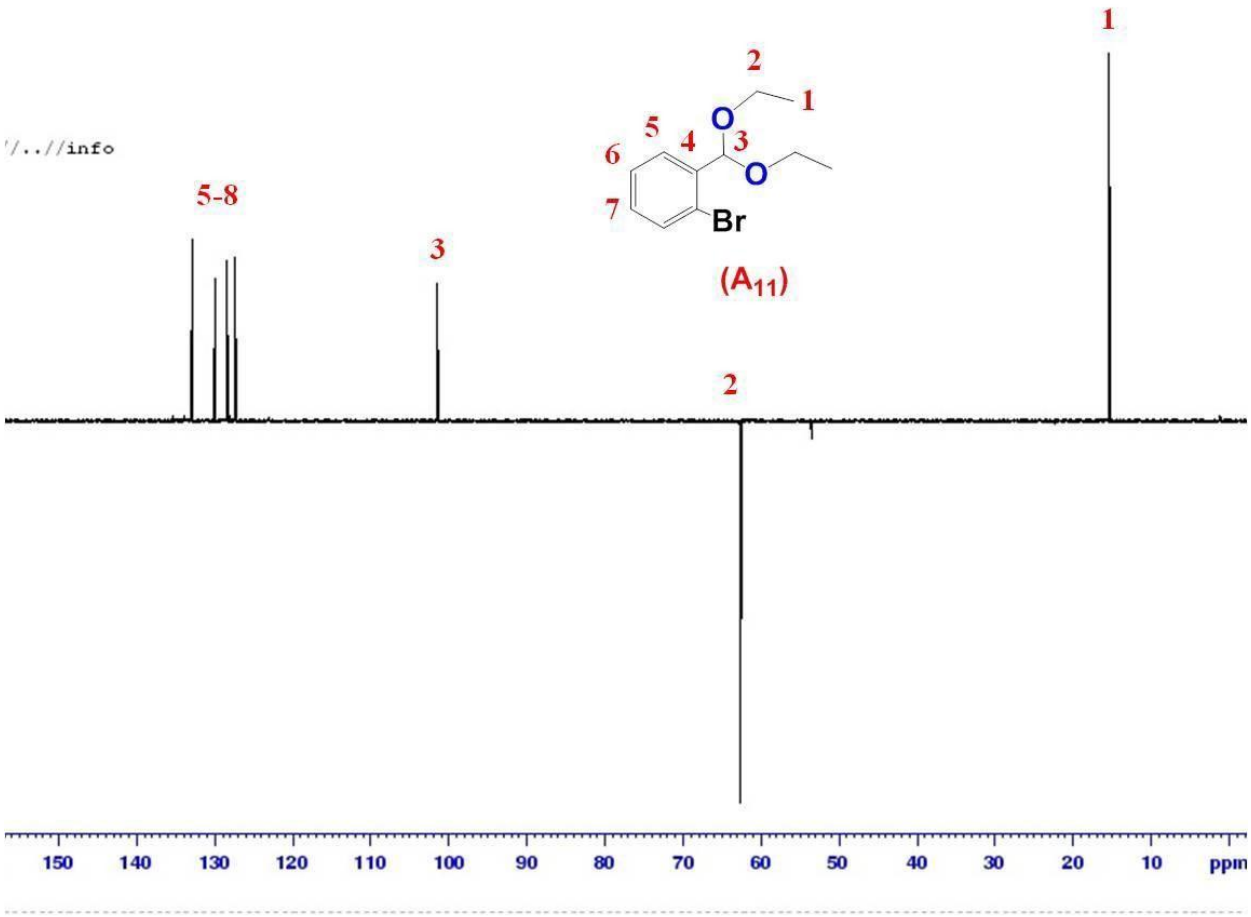

Figure S33: Dept 135 NMR Spectroscopy of isolated 2-bromobenzaldehydediethylacetal (A_{11}) in $CDCl_3$

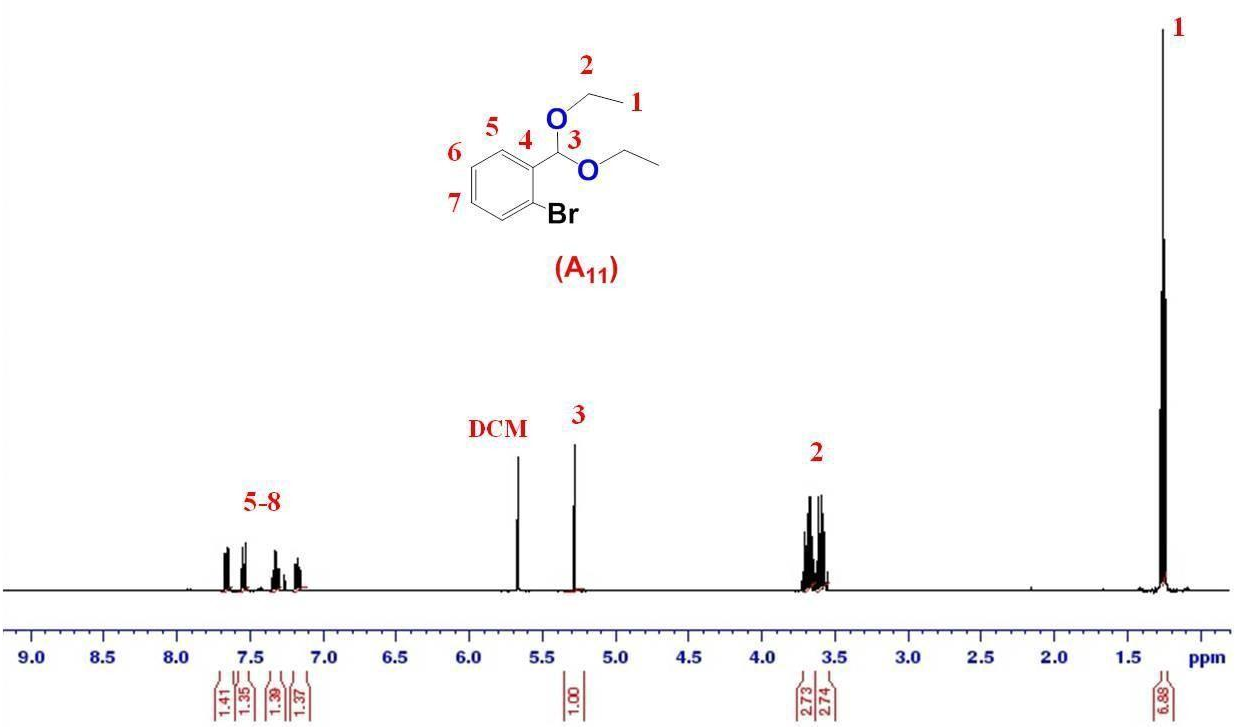

Figure S34: ^{1}H NMR Spectroscopy of isolated 2-bromobenzaldehydediethylacetal (A_{11}) in $CDCl_3$

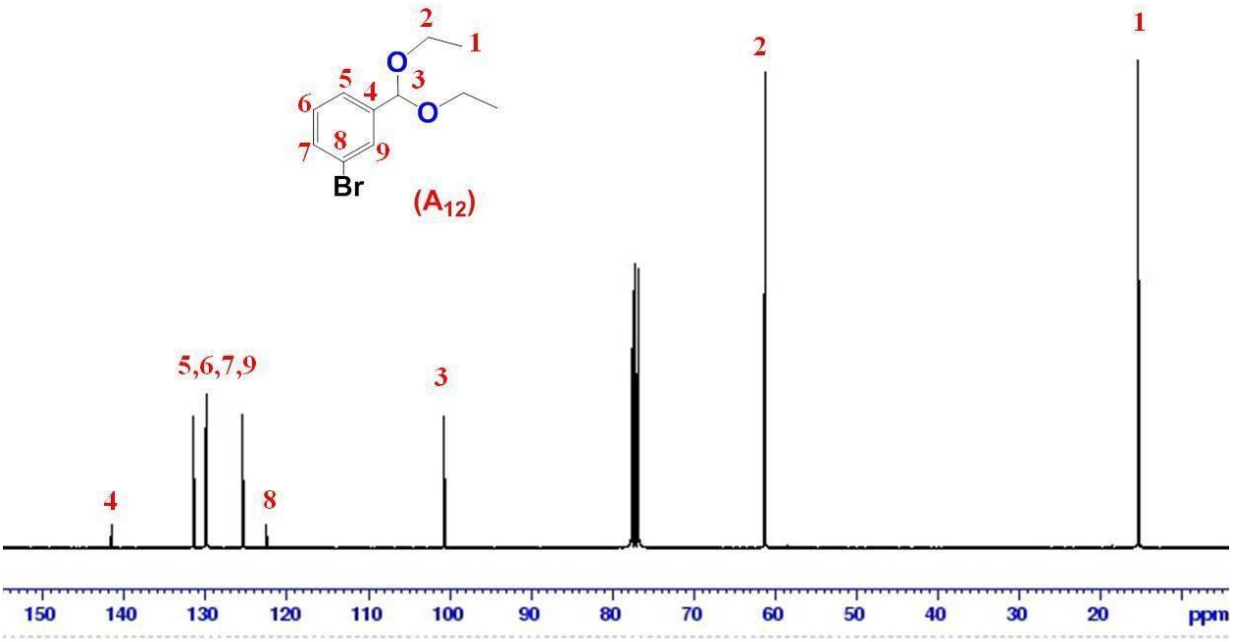

Figure S35: ^{13}C NMR Spectroscopy of isolated 3-bromobenzaldehyde diethylacetal (A_{12}) in $CDCl_3$

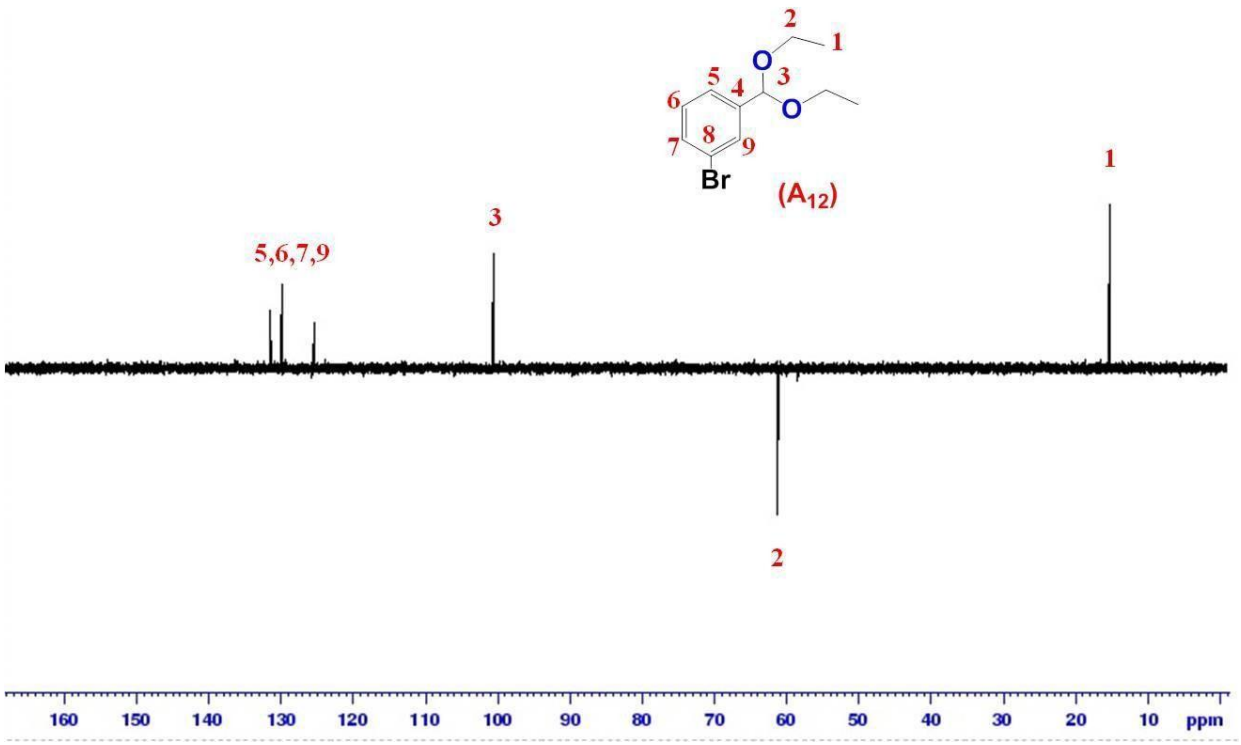

Figure S36: Dept 135 NMR Spectroscopy of isolated 3-bromobenzaldehyde diethylacetal (A_{12}) in $CDCl_3$

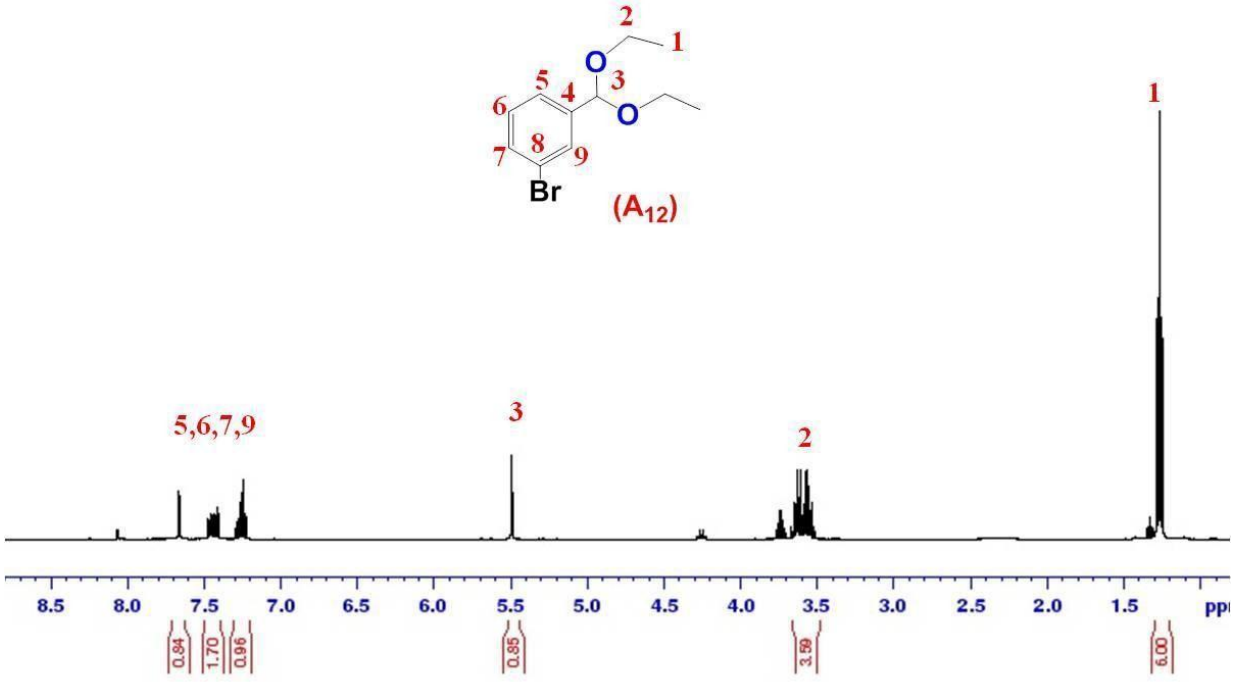

Figure S37: ^{1}H NMR Spectroscopy of isolated 3-bromobenzaldehyde diethylacetal (A_{12}) in $CDCl_3$

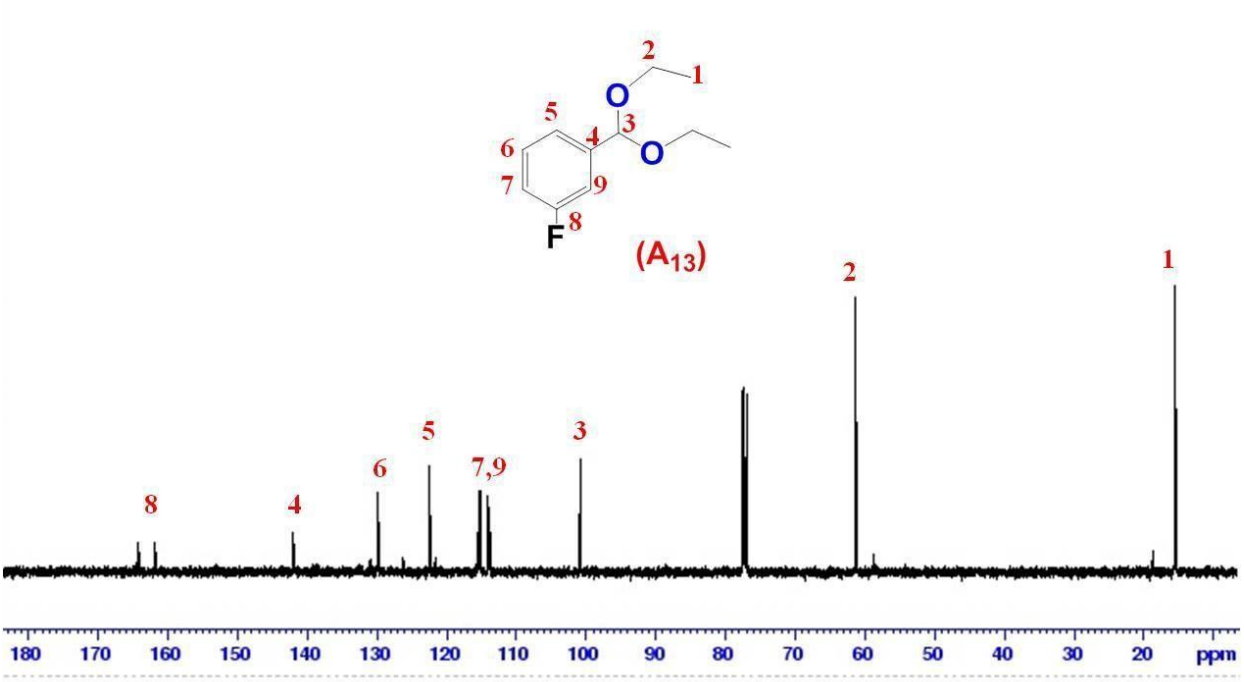

Figure S38: ^{13}C NMR Spectroscopy of isolated 3-fluorobenzaldehyde diethylacetal (A_{13}) in $CDCl_3$

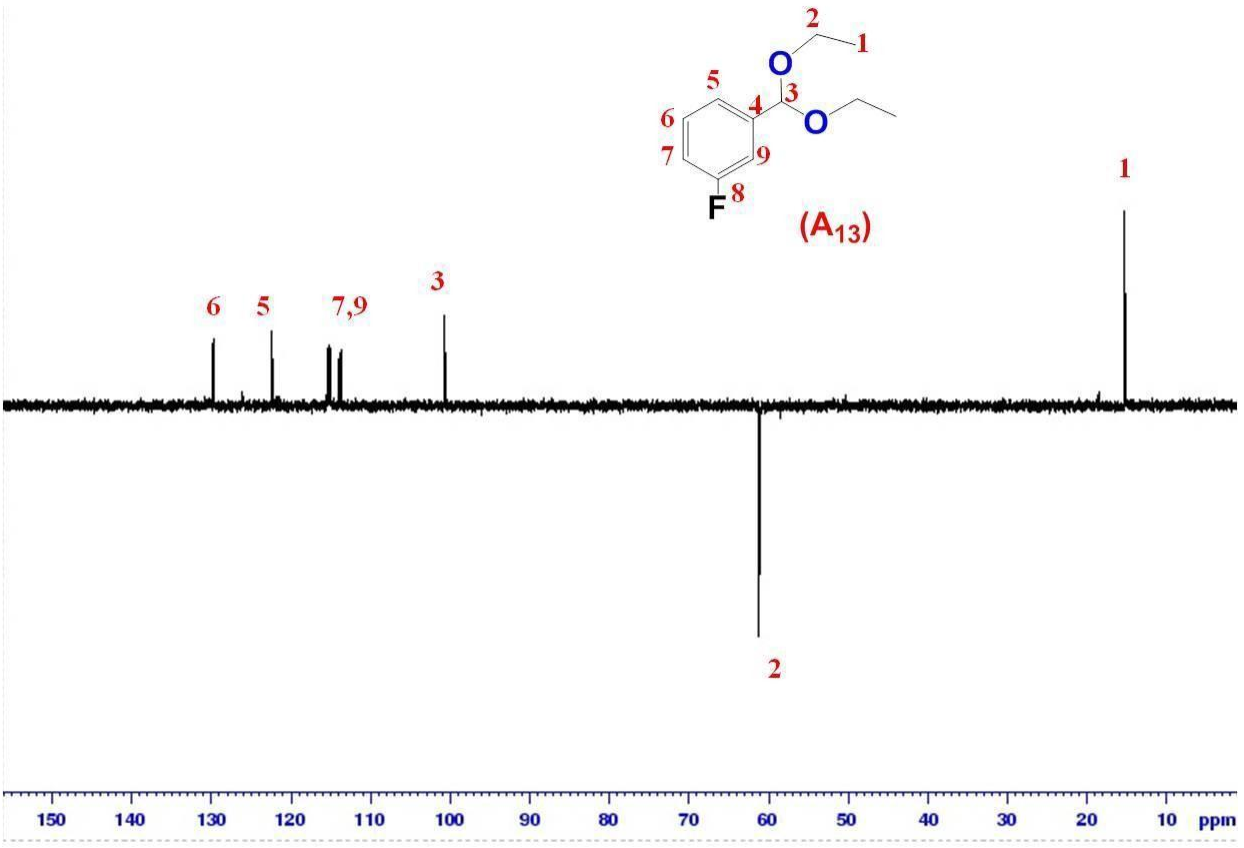

Figure S39: Dept 135 NMR Spectroscopy of isolated 3-fluorobenzaldehyde diethylacetal (A_{13}) in $CDCl_3$

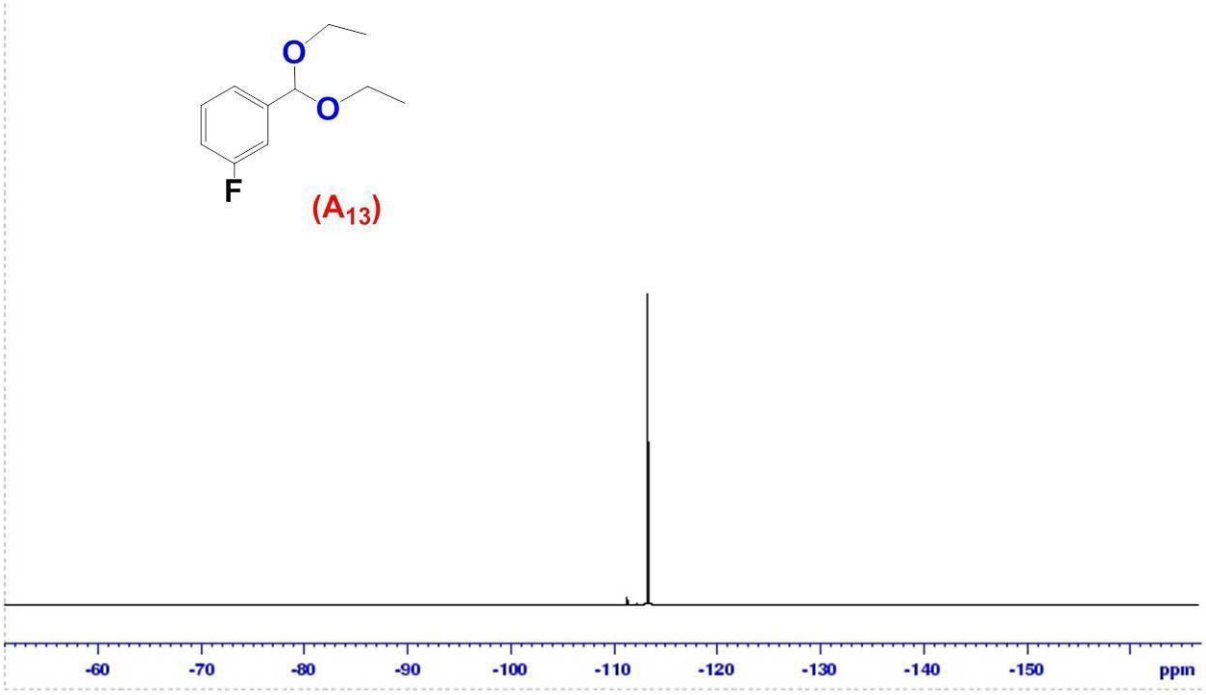

Figure S40: ^{19}F NMR Spectroscopy of isolated 3-fluorobenzaldehyde diethylacetal (A_{13}) in $CDCl_3$

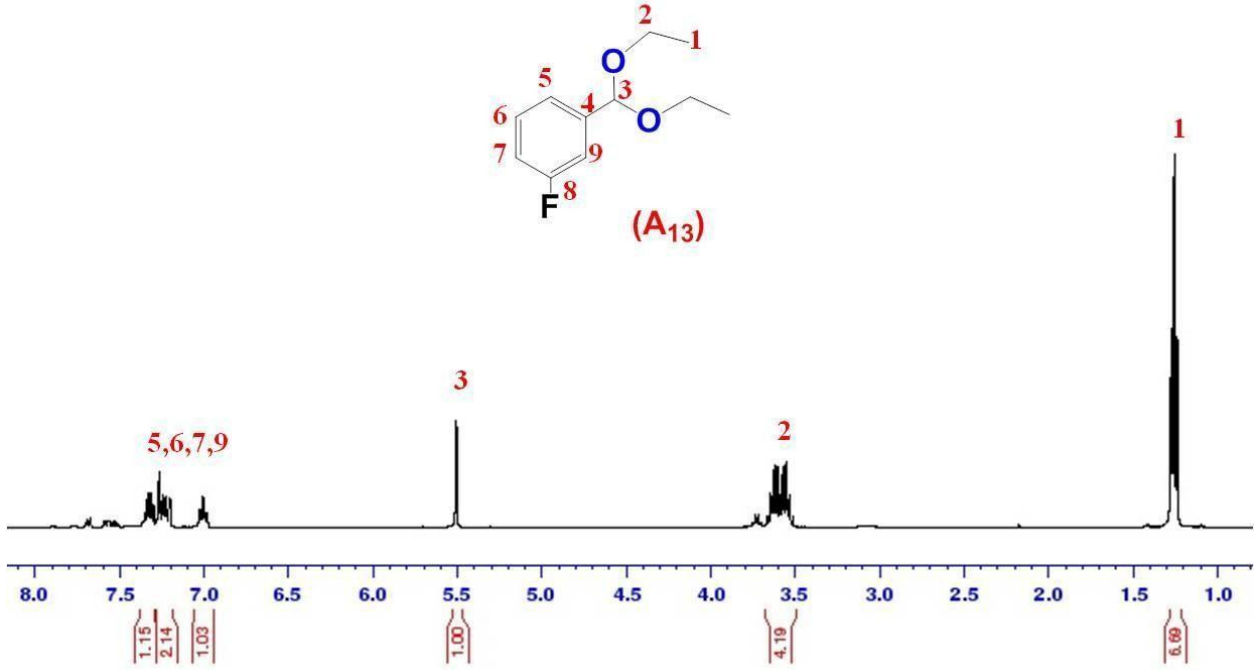

Figure S41: ^{1}H NMR Spectroscopy of isolated 3-fluorobenzaldehyde diethylacetal (A_{13}) in $CDCl_3$

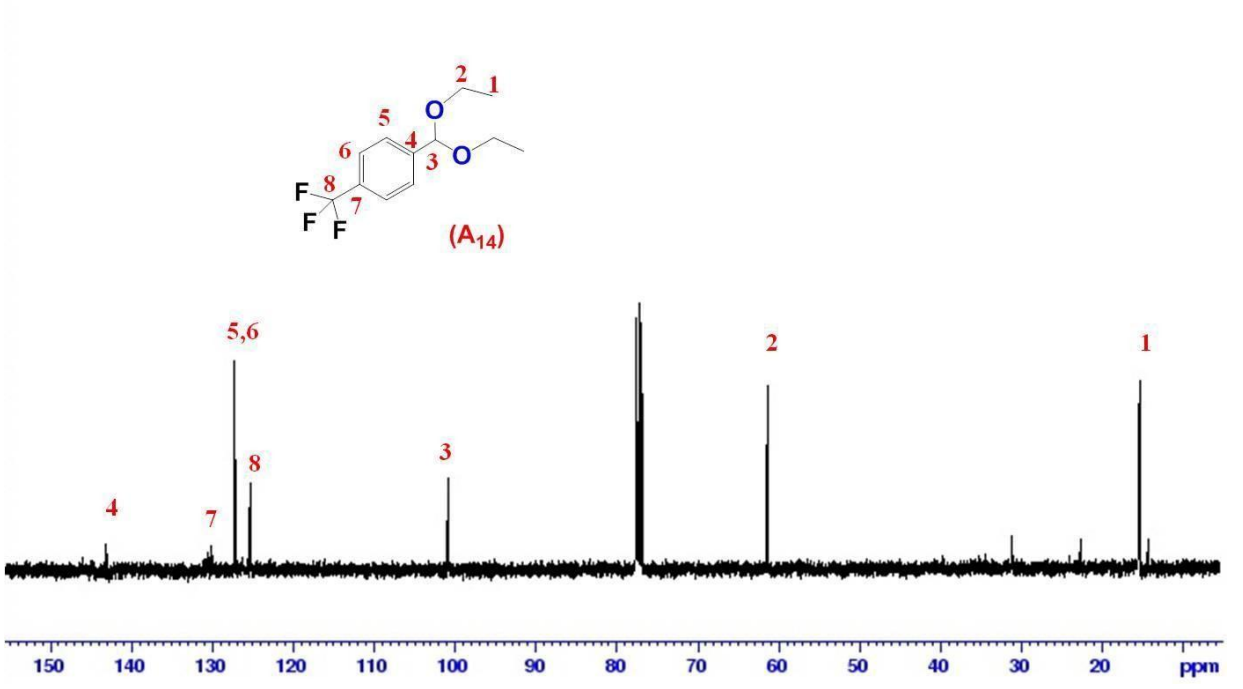

Figure S42: ^{13}C NMR Spectroscopy of isolated 4-trifluorobenzaldehyde diethylacetal (A_{14}) in $CDCl_3$

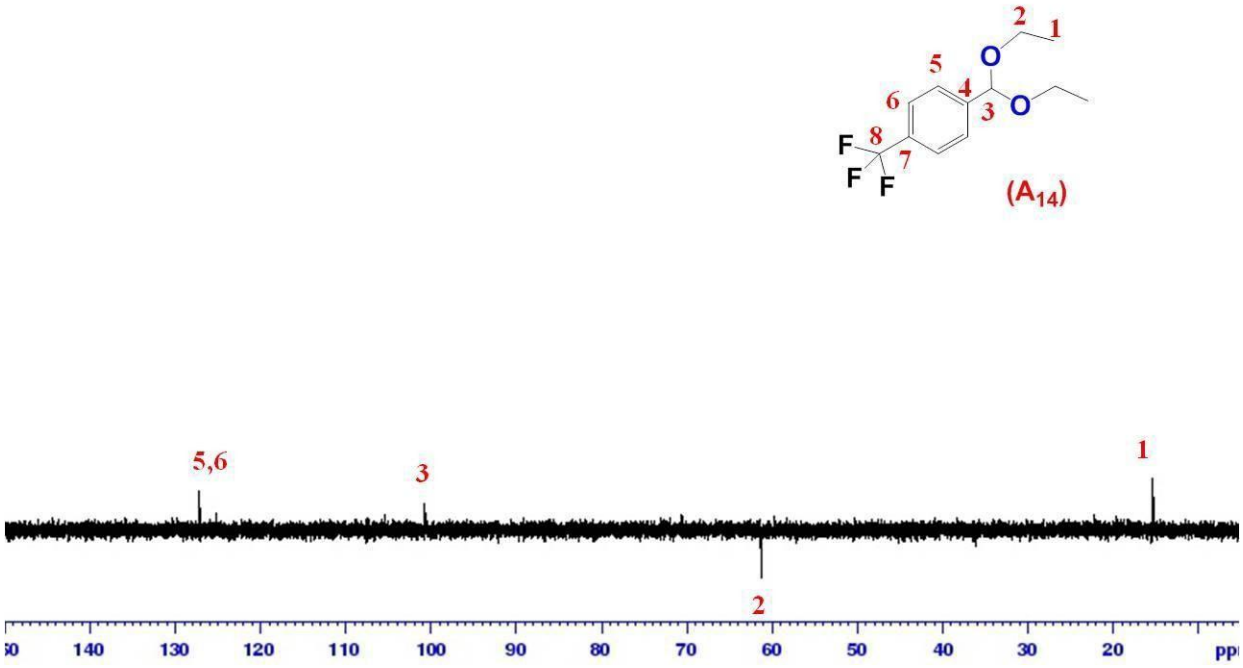

Figure S43: Dept 135 NMR Spectroscopy of isolated 4-trifluorobenzaldehyde diethylacetal (A_{14}) in $CDCl_3$

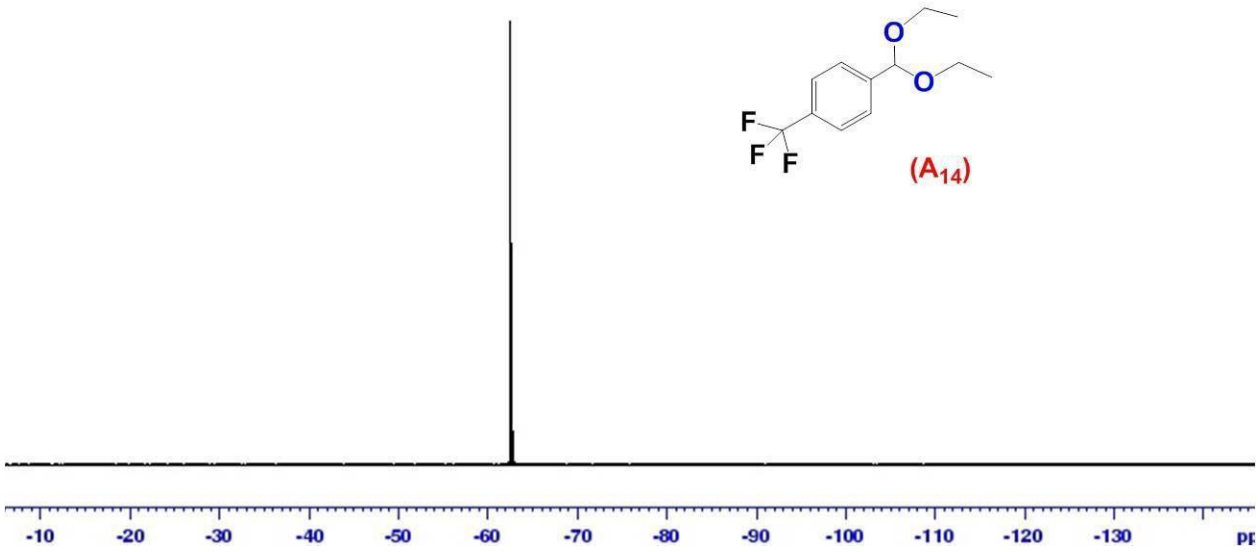

Figure S44: ^{19}F NMR Spectroscopy of isolated 4-trifluorobenzaldehyde diethylacetal (A_{14}) in $CDCl_3$

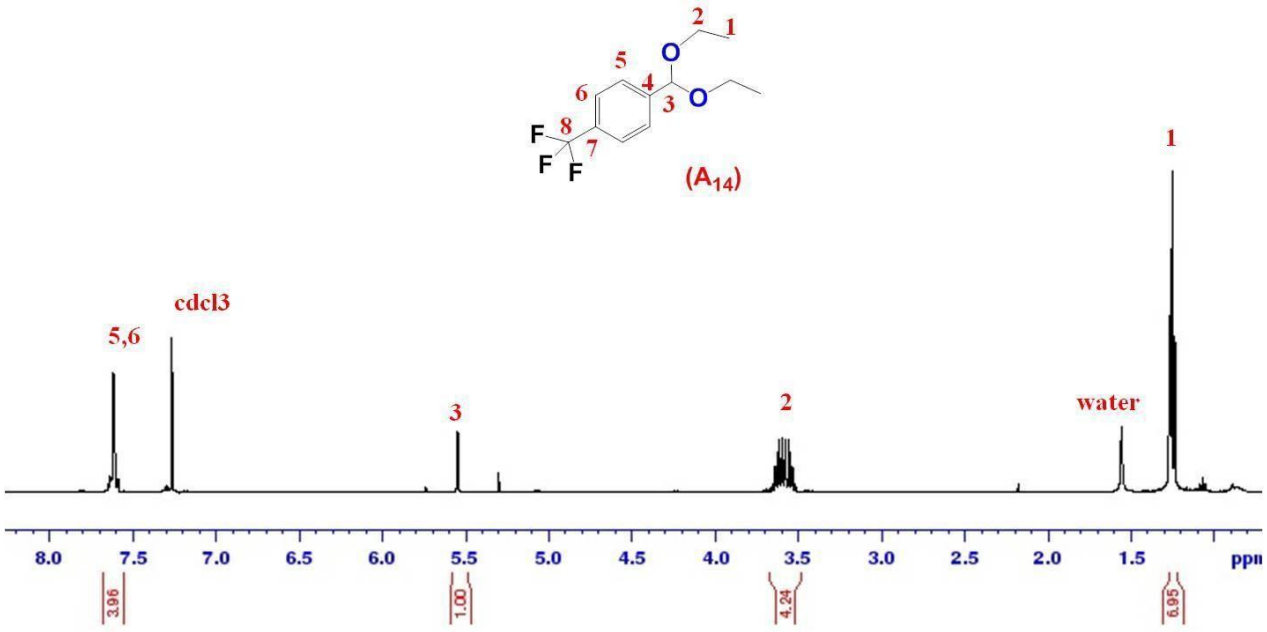

Figure S45: ^{1}H NMR Spectroscopy of isolated 4-trifluorobenzaldehyde diethylacetal (A_{14}) in $CDCl_3$

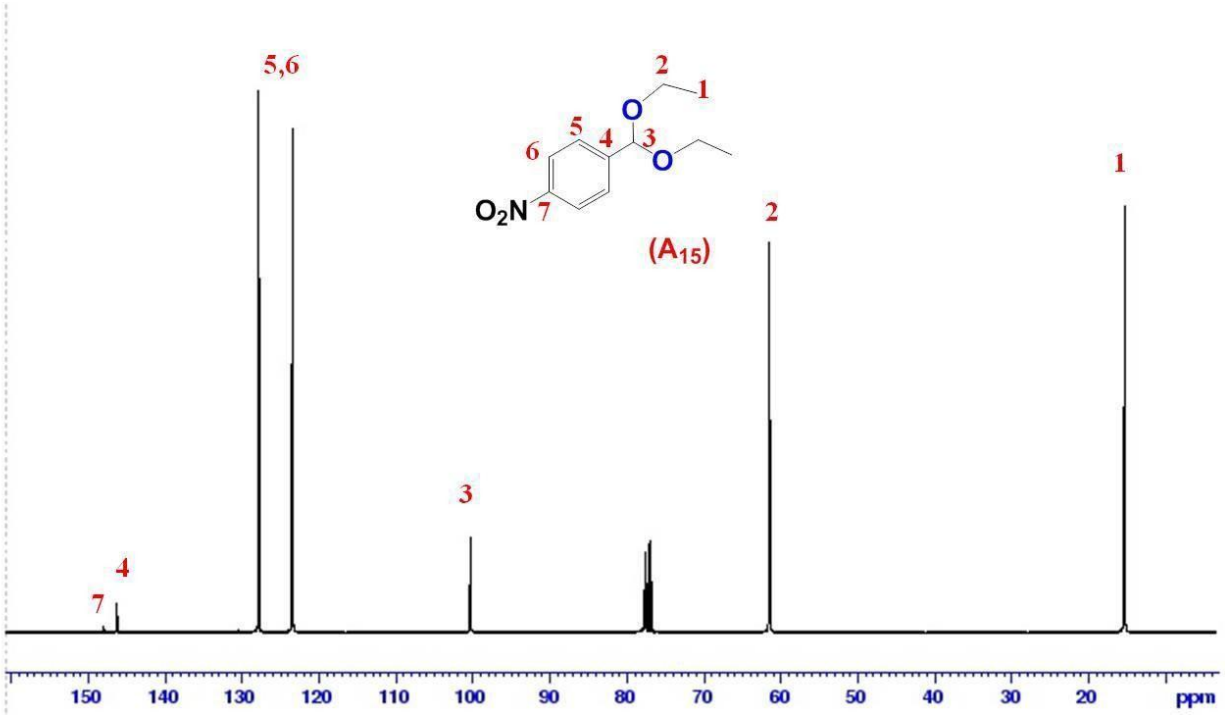

Figure S46: ^{13}C NMR Spectroscopy of isolated 4-nitrobenzaldehyde diethylacetal (A_{15}) in $CDCl_3$

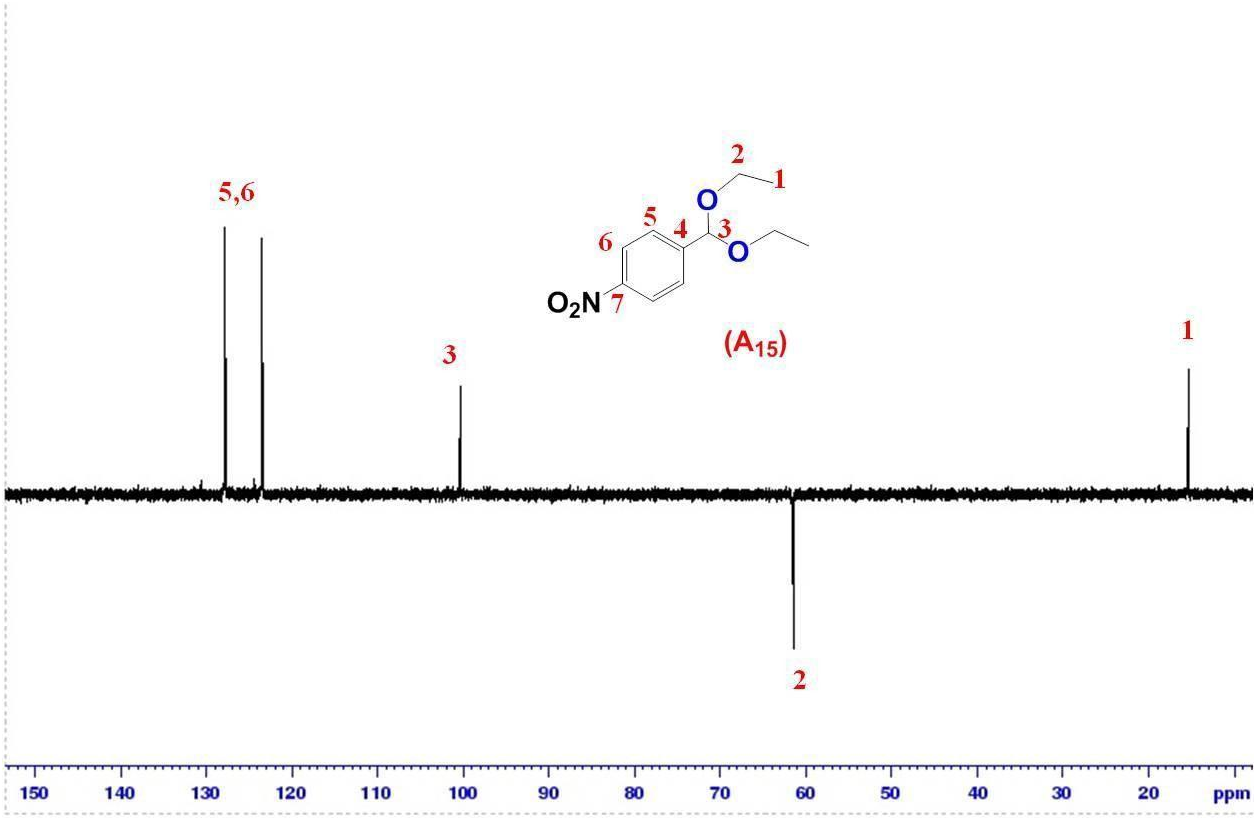

Figure S47: Dept 135 NMR Spectroscopy of isolated 4-nitrobenzaldehyde diethylacetal (A_{15}) in $CDCl_3$

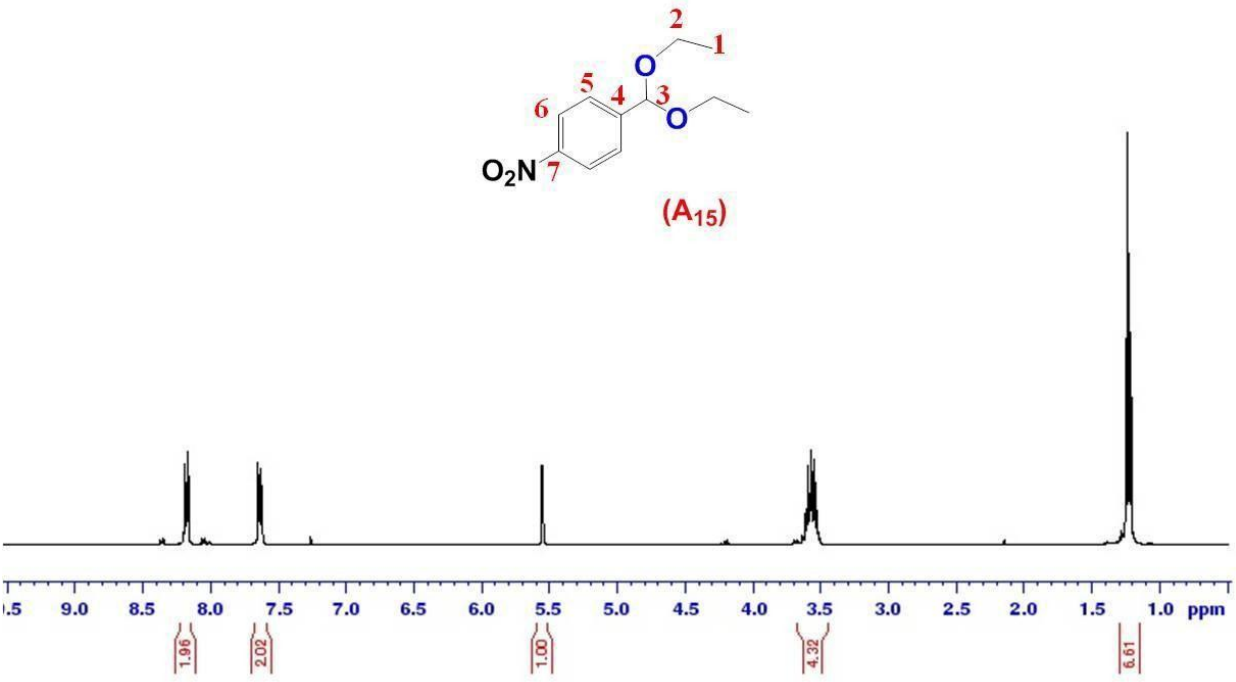

Figure S48: ^{1}H NMR Spectroscopy of isolated 4-nitrobenzaldehyde diethylacetal (A_{15}) in $CDCl_3$

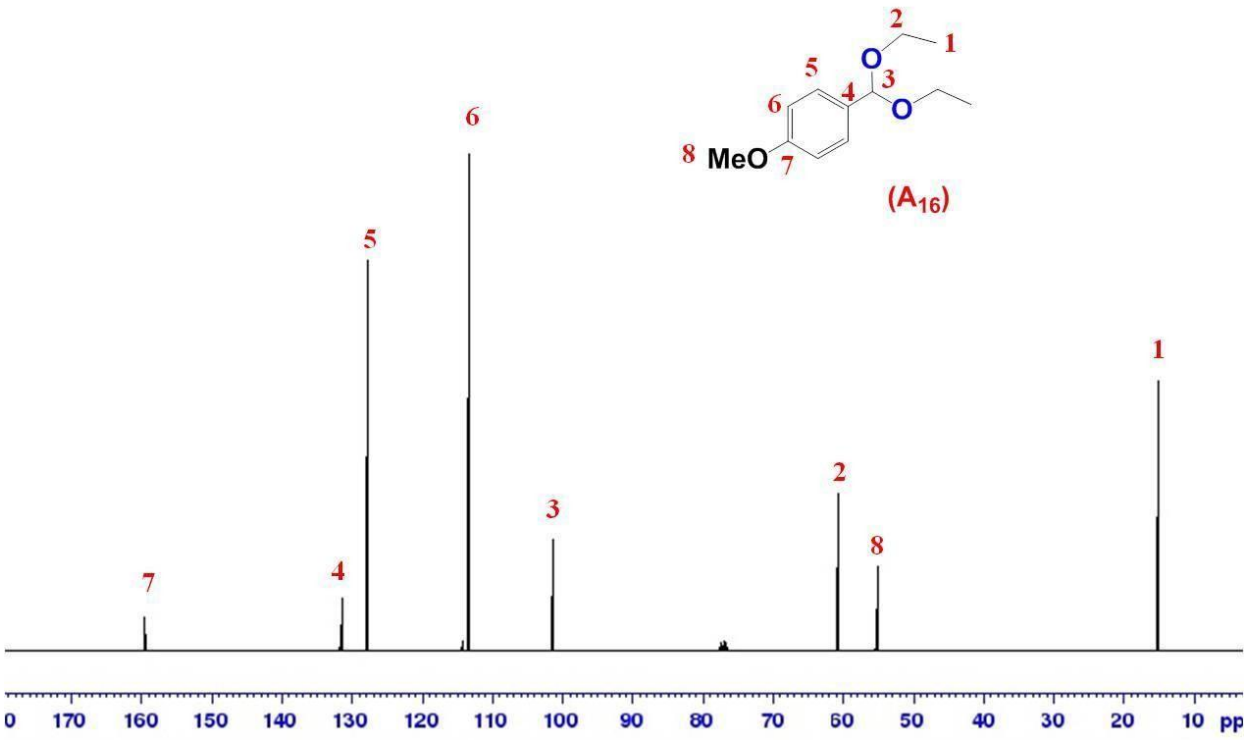

Figure S49: ^{13}C NMR Spectroscopy of isolated 4-methoxybenzaldehydediethylacetal (A_{16}) in $CDCl_3$.

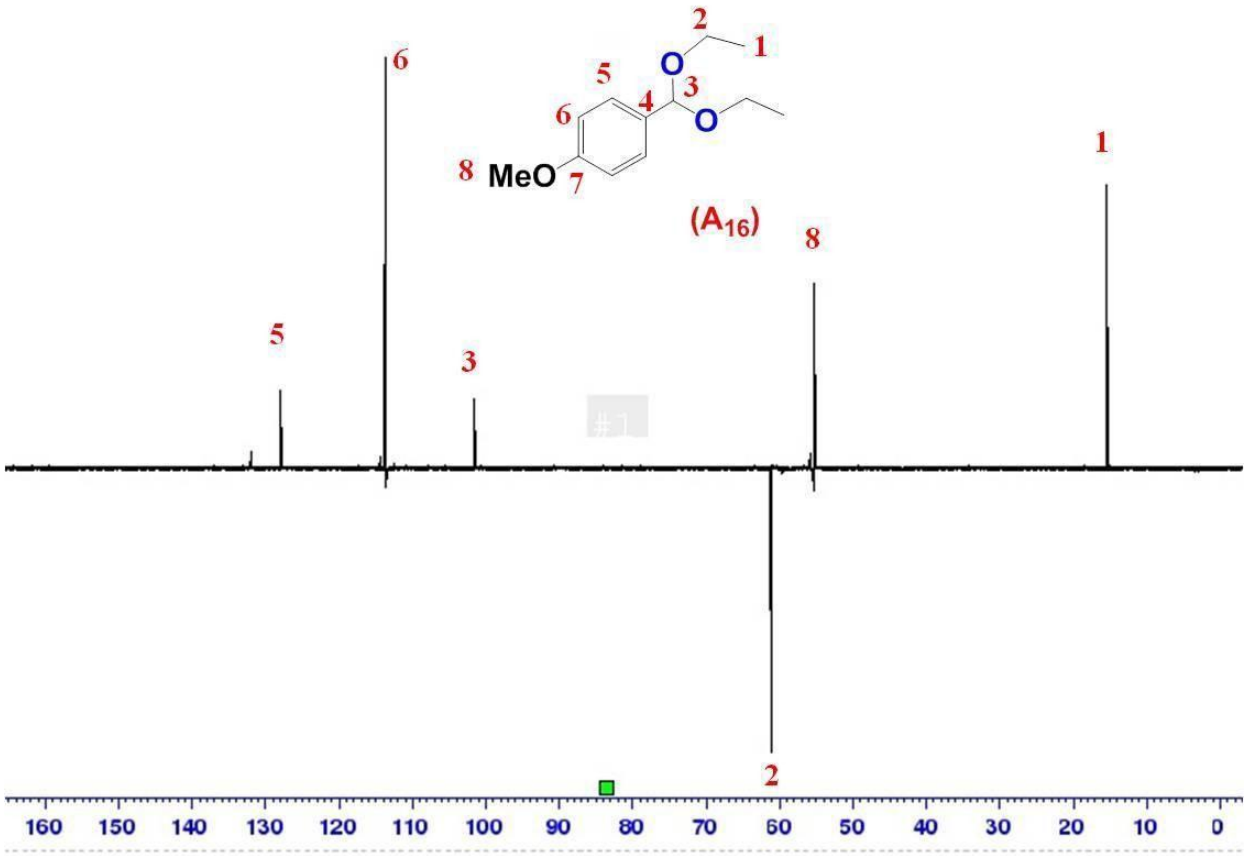

Figure S50: Dept 135 NMR Spectroscopy of isolated 4-methoxybenzaldehydediethylacetal (A_{16}) in $CDCl_3$.

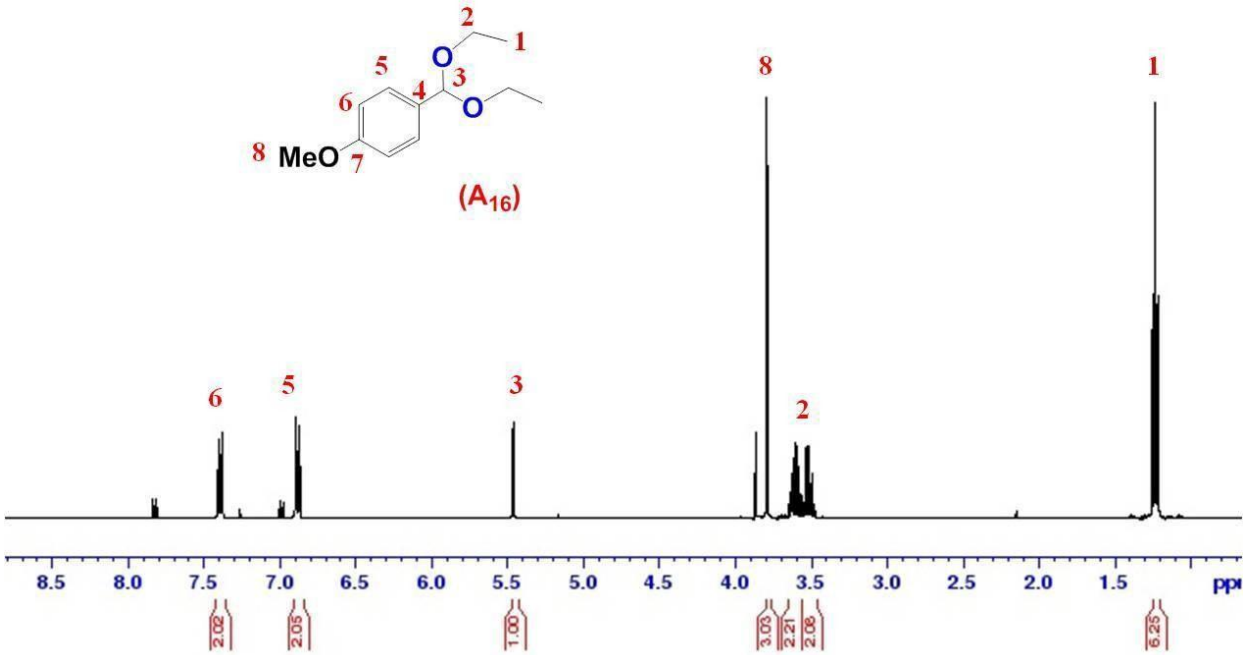

Figure S51: ^{1}H NMR Spectroscopy of isolated 4-methoxybenzaldehydediethylacetal (A_{16}) in $CDCl_3$.

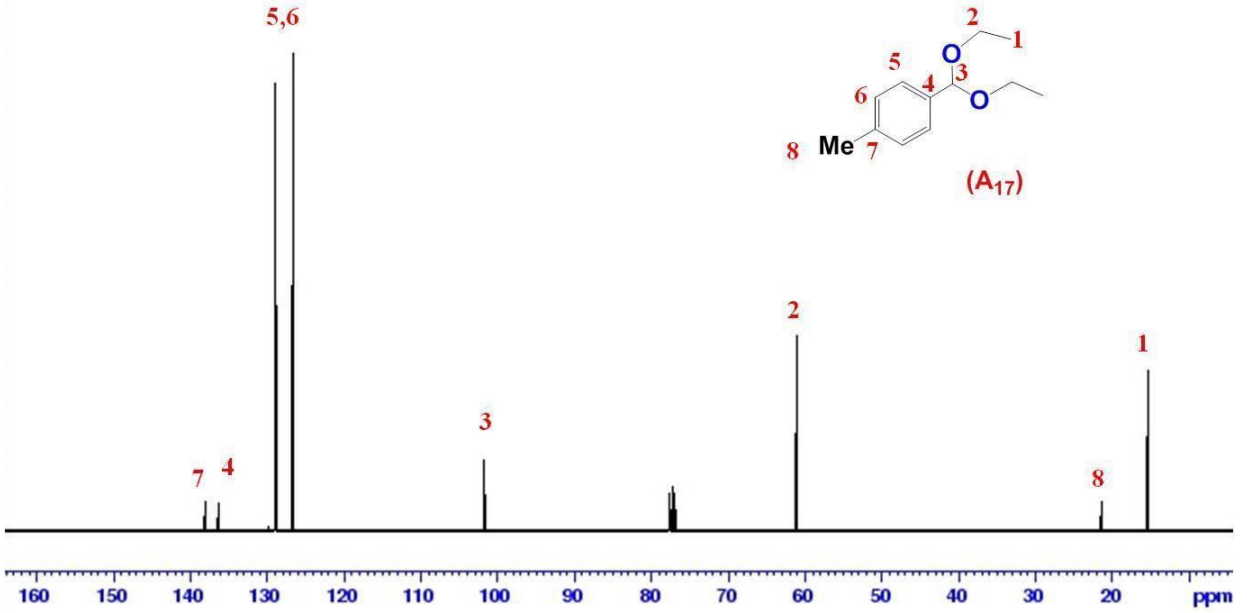

Figure S52: ^{13}C NMR Spectroscopy of isolated 4-methylbenzaldehyde diethylacetal (A_{17}) in $CDCl_3$.

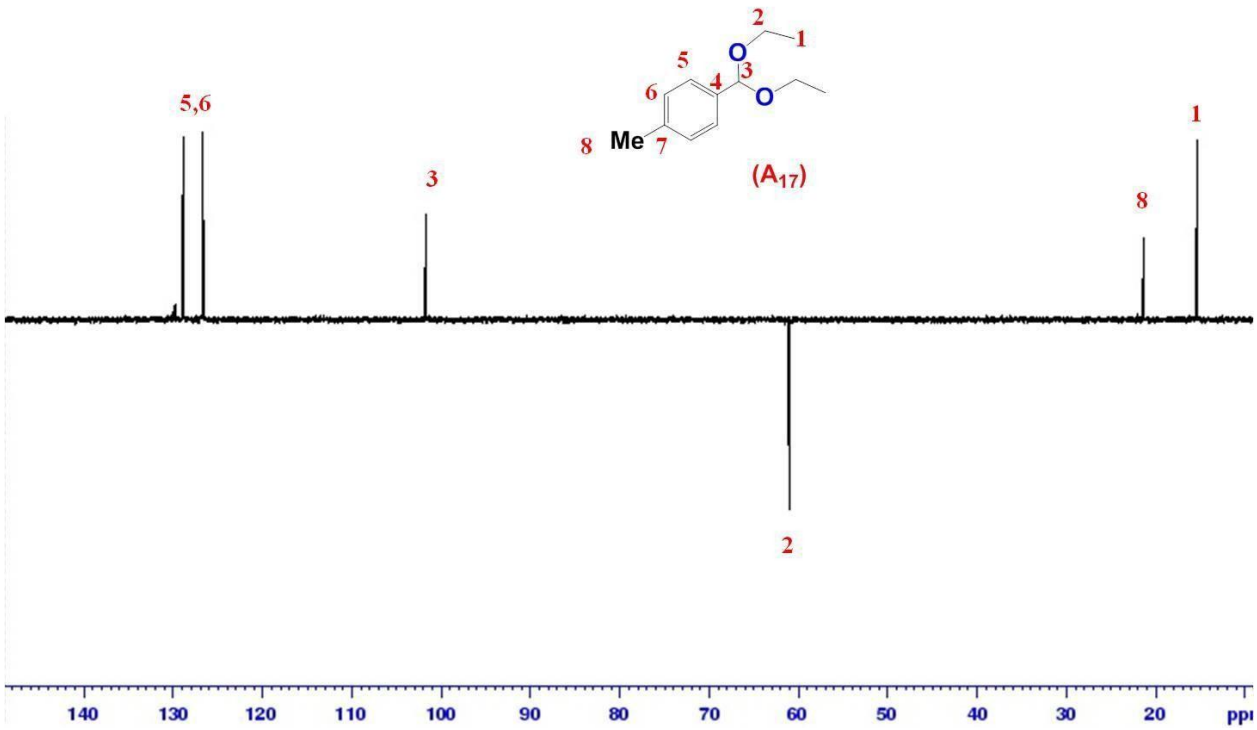

Figure S53: Dept 135 NMR Spectroscopy of isolated 4-methylbenzaldehyde diethylacetal (A_{17}) in $CDCl_3$.

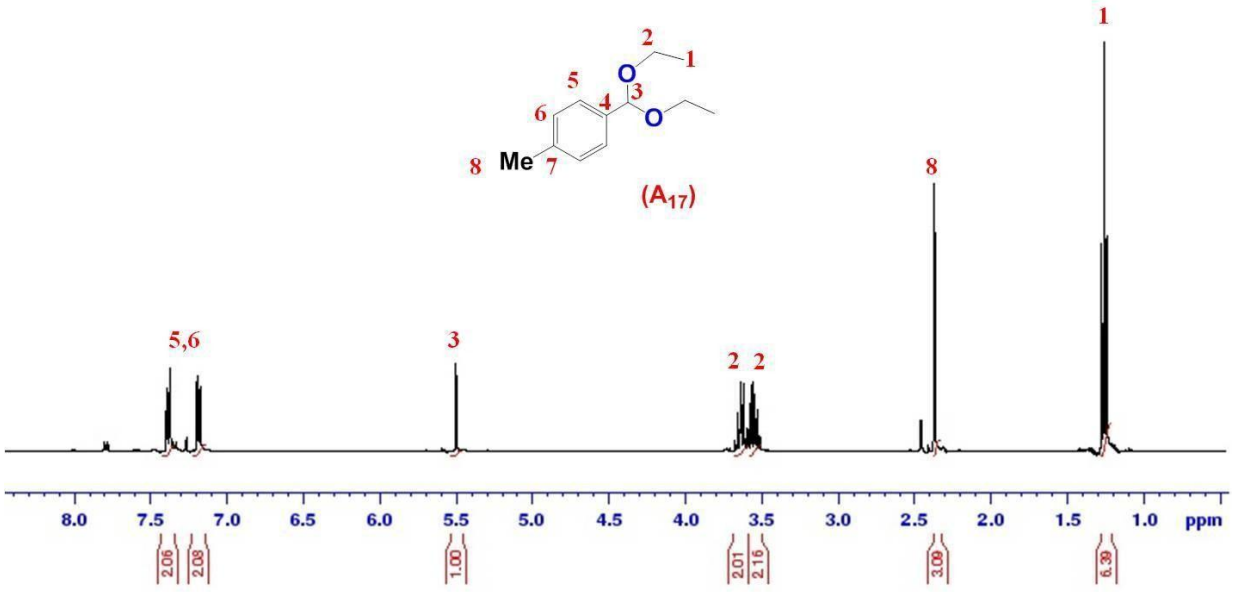

Figure S54: ^{1}H NMR Spectroscopy of isolated 4-methylbenzaldehyde diethylacetal (A_{17}) in $CDCl_3$.

Procedure for testing the recyclability of [1]+ using 2-ethylbutanal as a model substrate and TES as an ethoxide source. (All reactions were monitored by ^{13}C NMR).

Procedure:

In a NMR tube 0.1mol % of [**1**][OTf] was added initially, and then 1 mol triethoxysilane (TES) was added dropwise directly to the stibonium salt to ensure complete dissolutione. After that 100 mg (1 mmol) of the 2-ethylbutanal was added dropwise over this mixture and was stirred and monitored by ^{13}C NMR spectroscopy in $CDCl_3$. After 1h we confirmed that all the aldehyde was consumed, and the reaction was purified according to the main text in the manuscript. The resulting compound was then characterized by ^{13}C NMR in $CDCl_3$, and was labeled Run 1. The residue containing the catalyst after distillation of the acetal was then recycled by adding more reagents (TES and aldehyde). We found that the acetalisation could be performed 5 times without a noticeable drop in activity of the catalyst (see Figure S55 below).

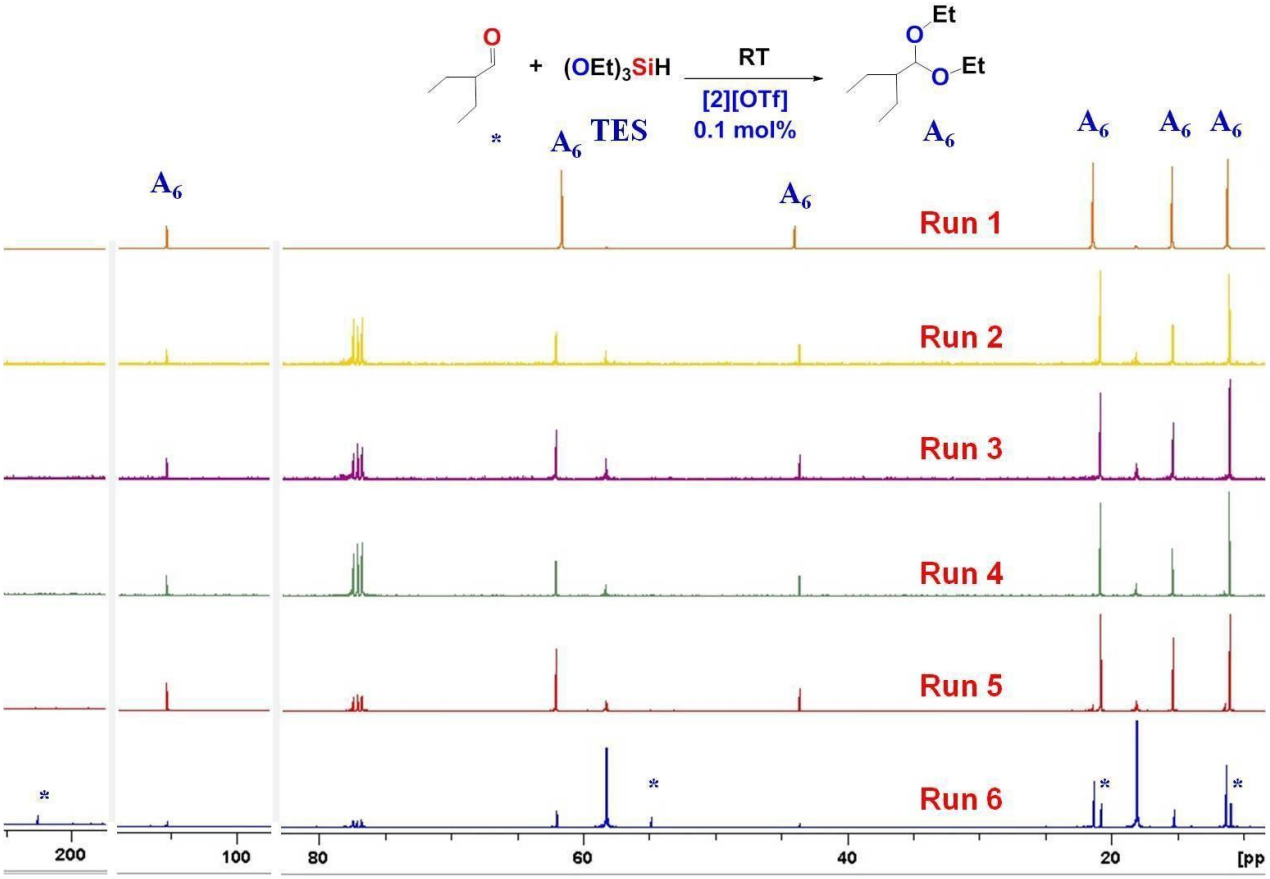

Figure S55: Stacked ^{13}C NMR spectra ($CDCl_3$) demonstrating that the catalytic activity of [1]+ could be recycled for up to 5 cycles without significant loss in activity.