

Supplementary Material

Diels-Alder reactions with 1,1-Diethoxybut-3-yn-2-one and some 1,1-diethoxy-5-hydroxyalk-3-yn-2-ones and their acetates

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Flash-column chromatography gradient elution

Flash-column chromatography (FC) was performed using a Reveleris X" Flash System Silica Gel from Fluka Analytical (230–400 mesh) and eluting with a gradient of ethyl acetate in a mixture of hexanes for in total 16.5 min. The gradient applied can be summarized as follows:

| Time span per mixture (min) | Ethyl acetate (% v/v) in hexanes |
|-----------------------------|----------------------------------|
| 0.0 – 1.5 | 0 |
| 1.5 – 2.5 | 1 |
| 2.5 – 4.0 | 2 |
| 4.0 – 9.5 | 3 |
| 9.5 – 16.5 | 10 |

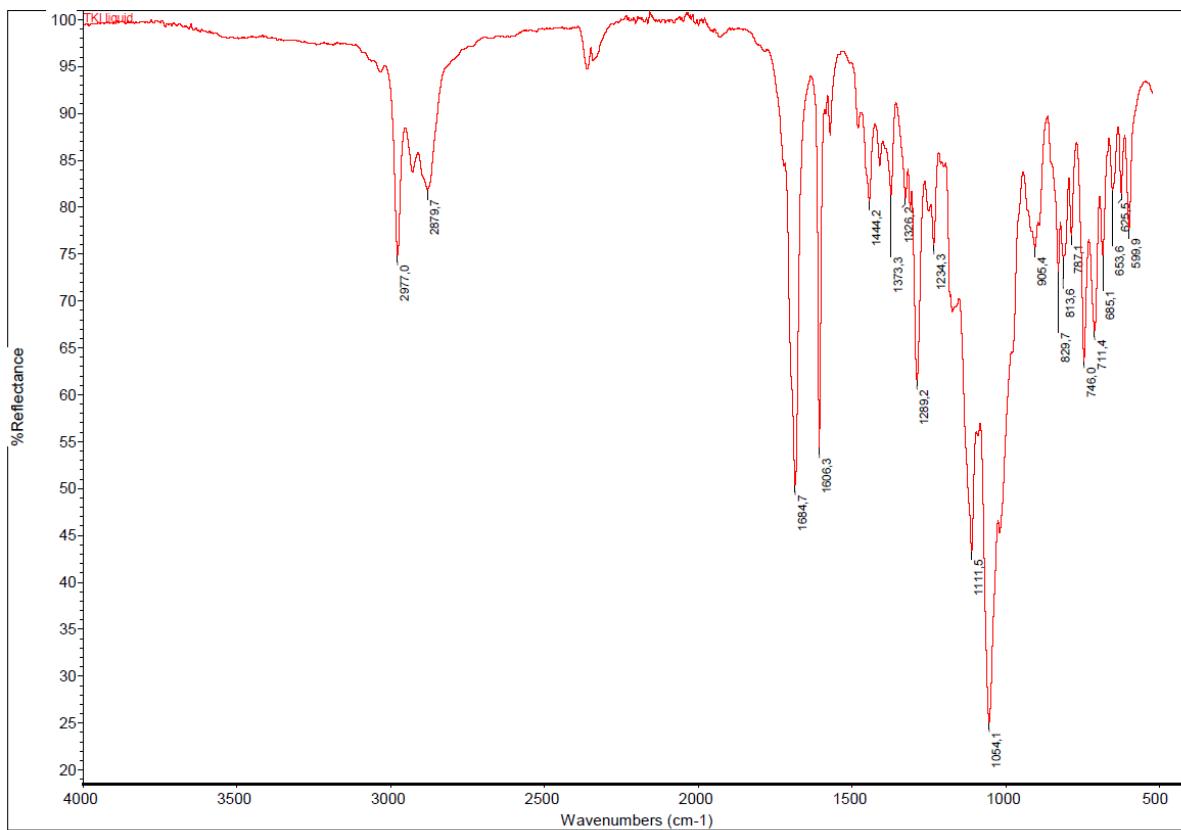
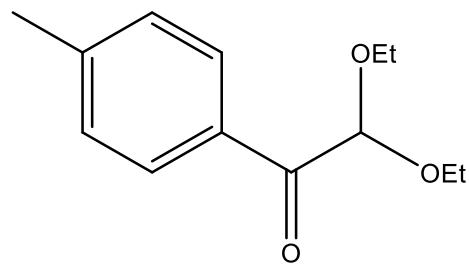
2,2-Diethoxy-1-(4-tolyl)ethanone (**3a**)

Figure 1. IR spectrum of 2,2-diethoxy-1-(4-tolyl)ethanone

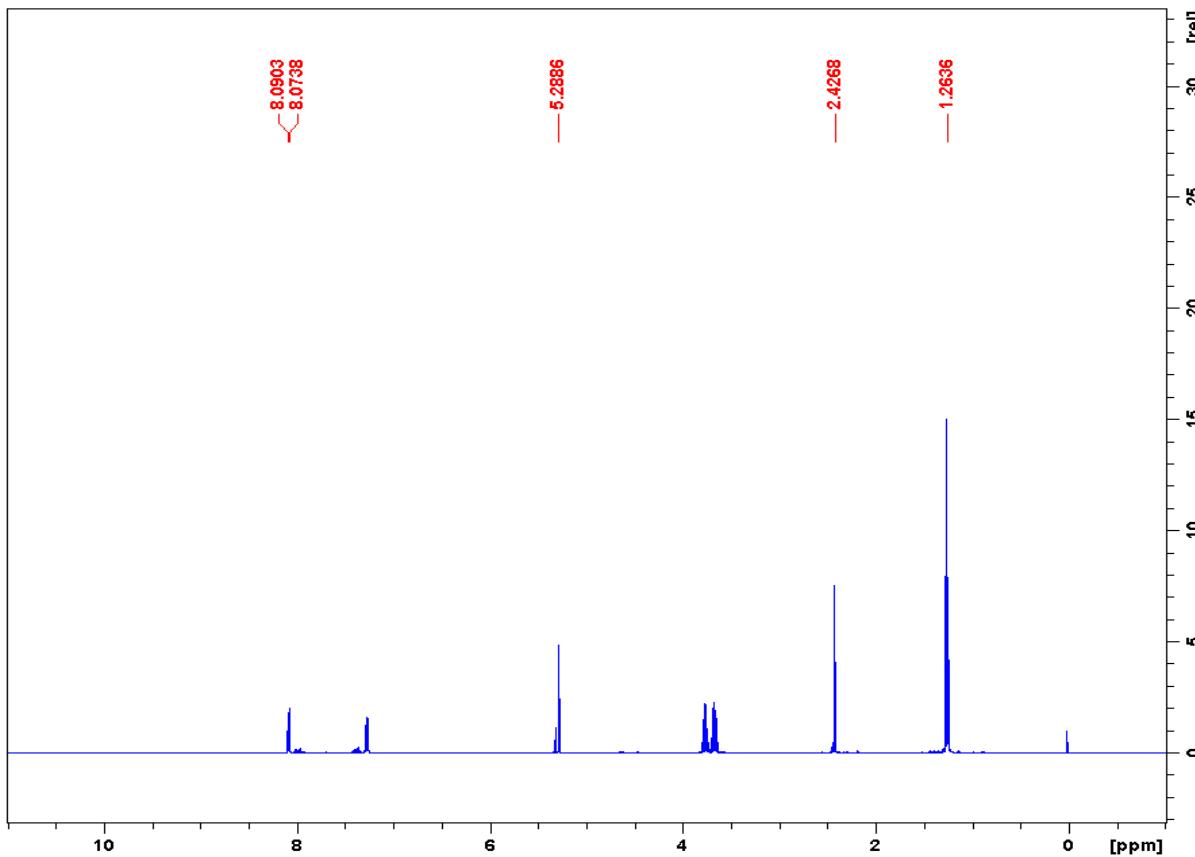


Figure 2: ^1H -NMR spectrum of 2,2-diethoxy-1-(4-tolyl)ethanone

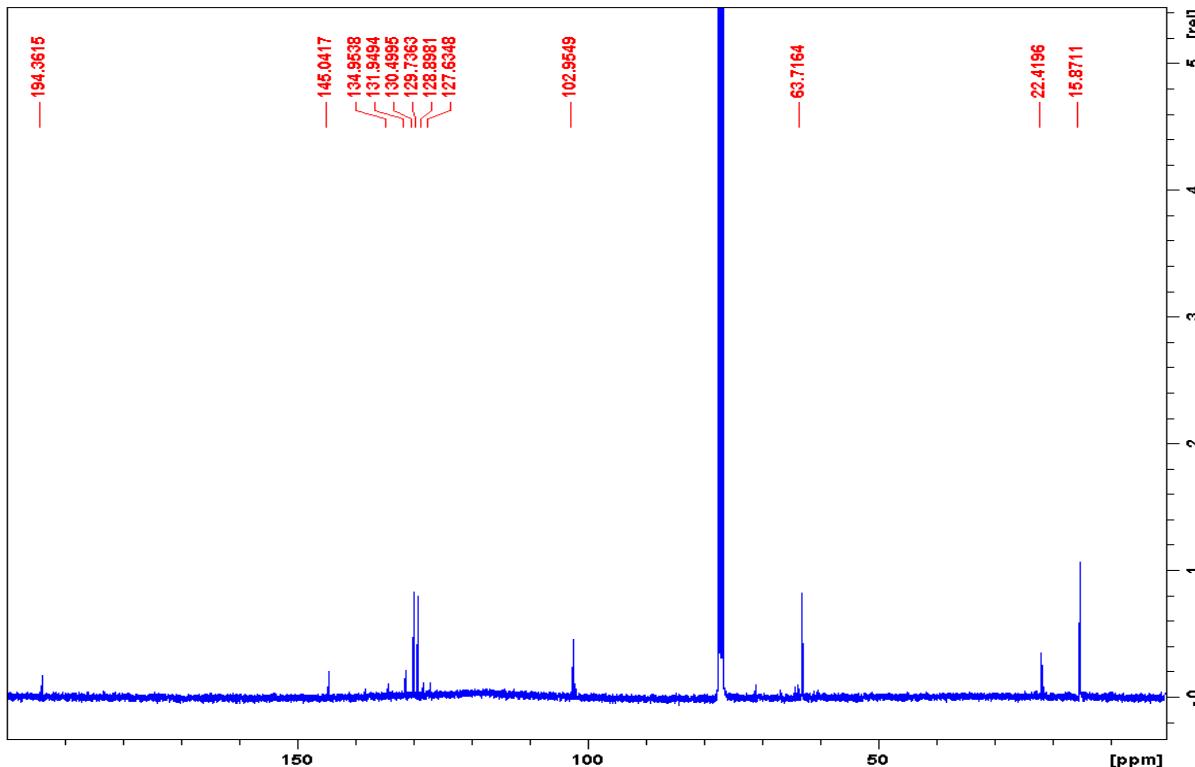
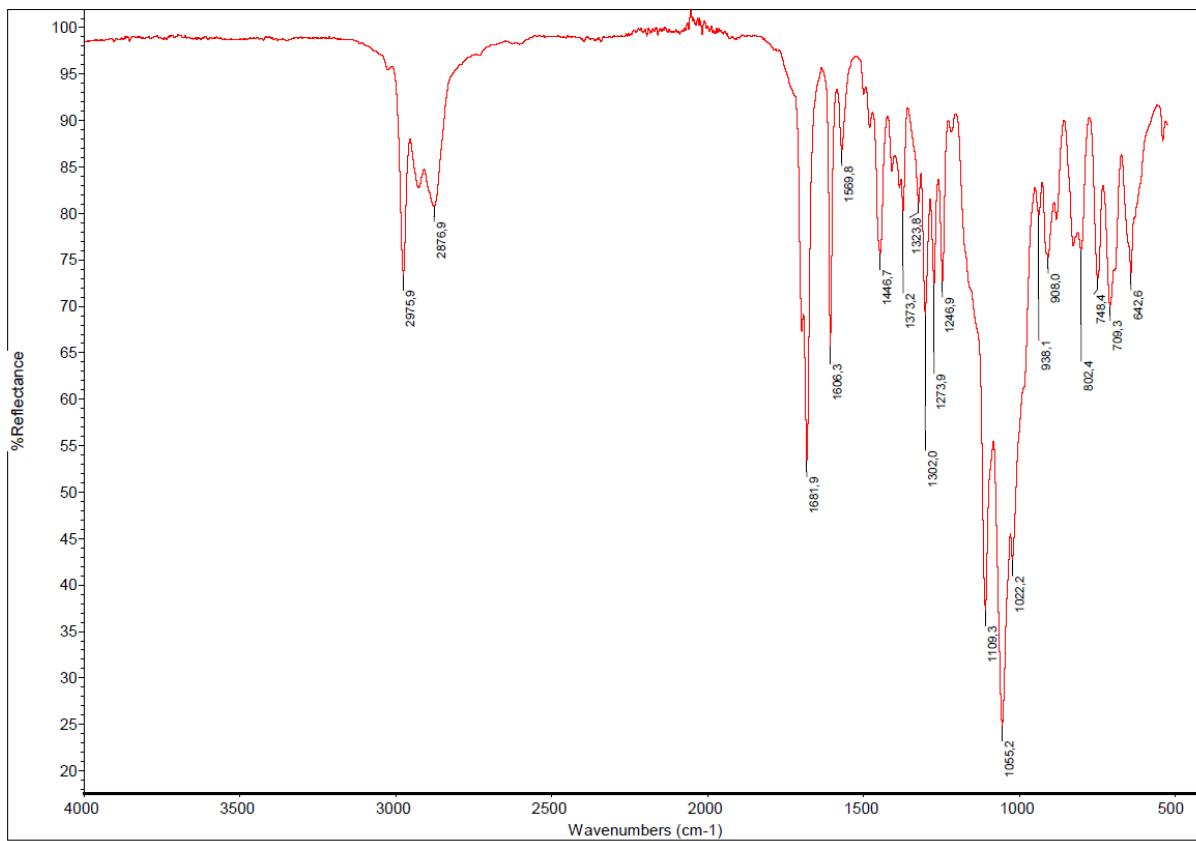
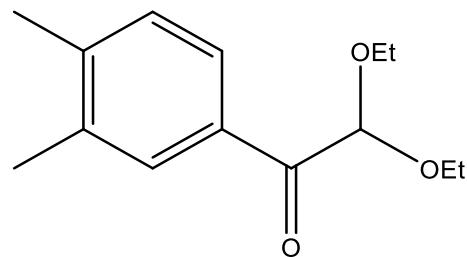


Figure 3 ^{13}C -NMR spectrum of 2,2-diethoxy-1-(4-tolyl)ethanone

2,2-Diethoxy-1-(3,4-dimethylphenyl)ethanone (3b**)****Figure 4** IR spectrum of 2,2-diethoxy-1-(3,4-dimethylphenyl)ethanone

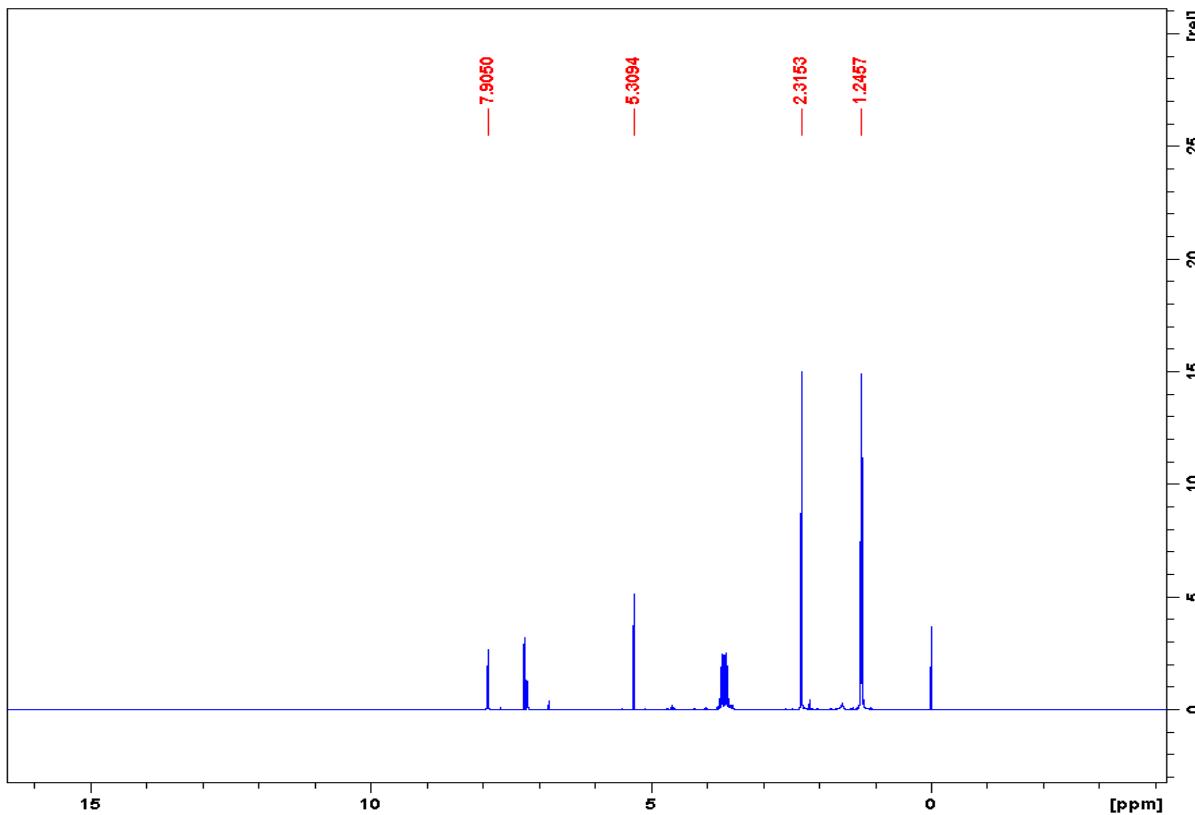


Figure 5 ^1H -NMR spectrum of 2,2-diethoxy-1-(3,4-dimethylphenyl)ethanone

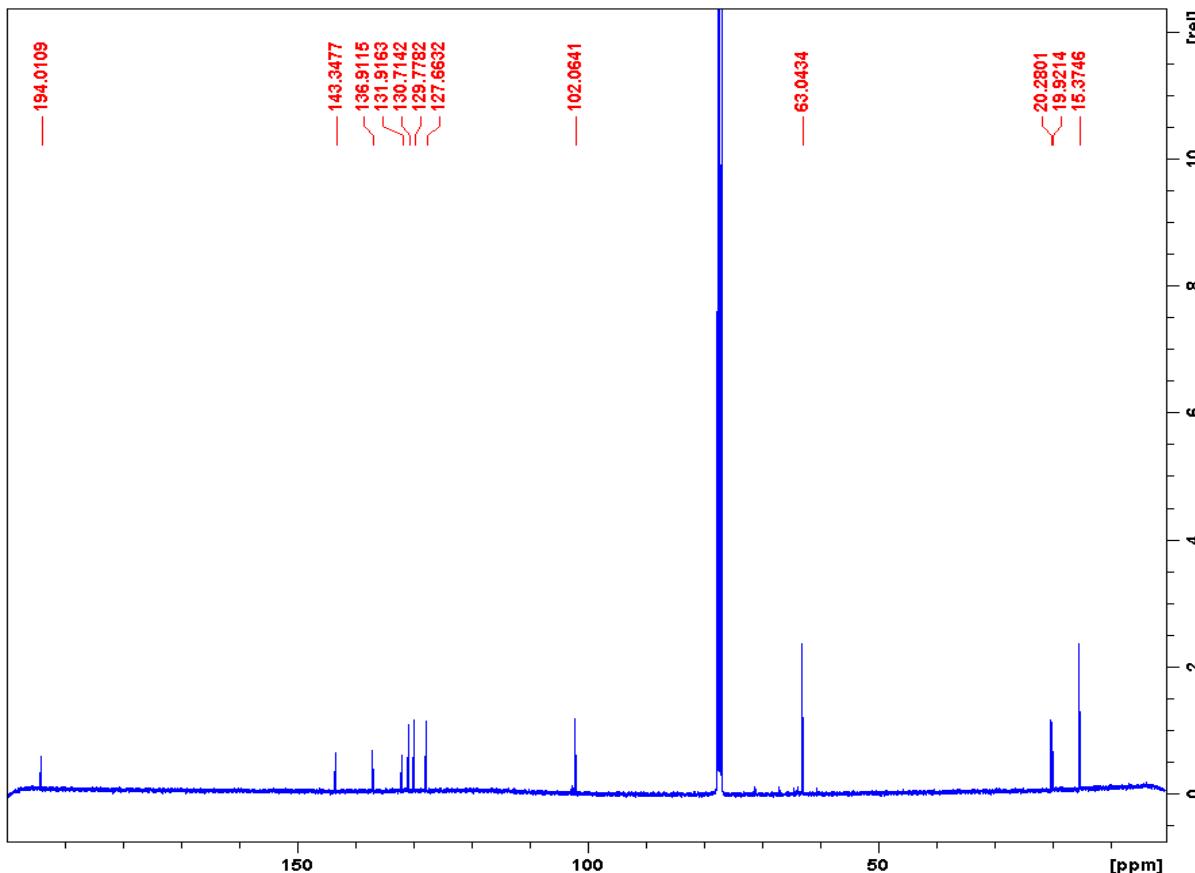


Figure 6 ^{13}C -NMR spectrum of 2,2-diethoxy-1-(3,4-dimethylphenyl)ethanone

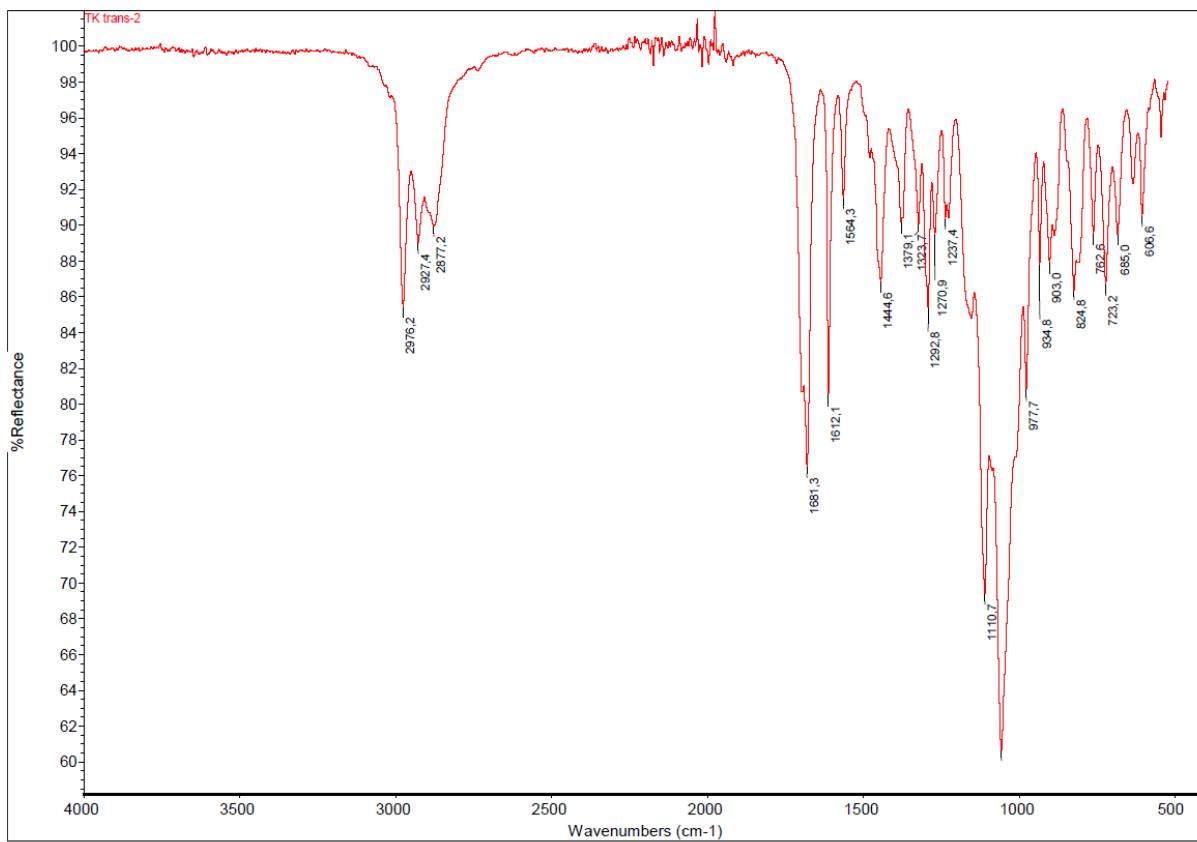
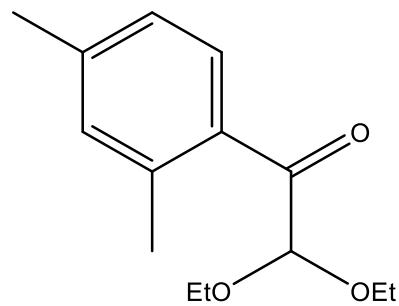
2,2-Diethoxy-1-(2,4-dimethylphenyl)ethanone (**3c**)

Figure 7 IR spectrum of 2,2-diethoxy-1-(2,4-dimethylphenyl)ethanone

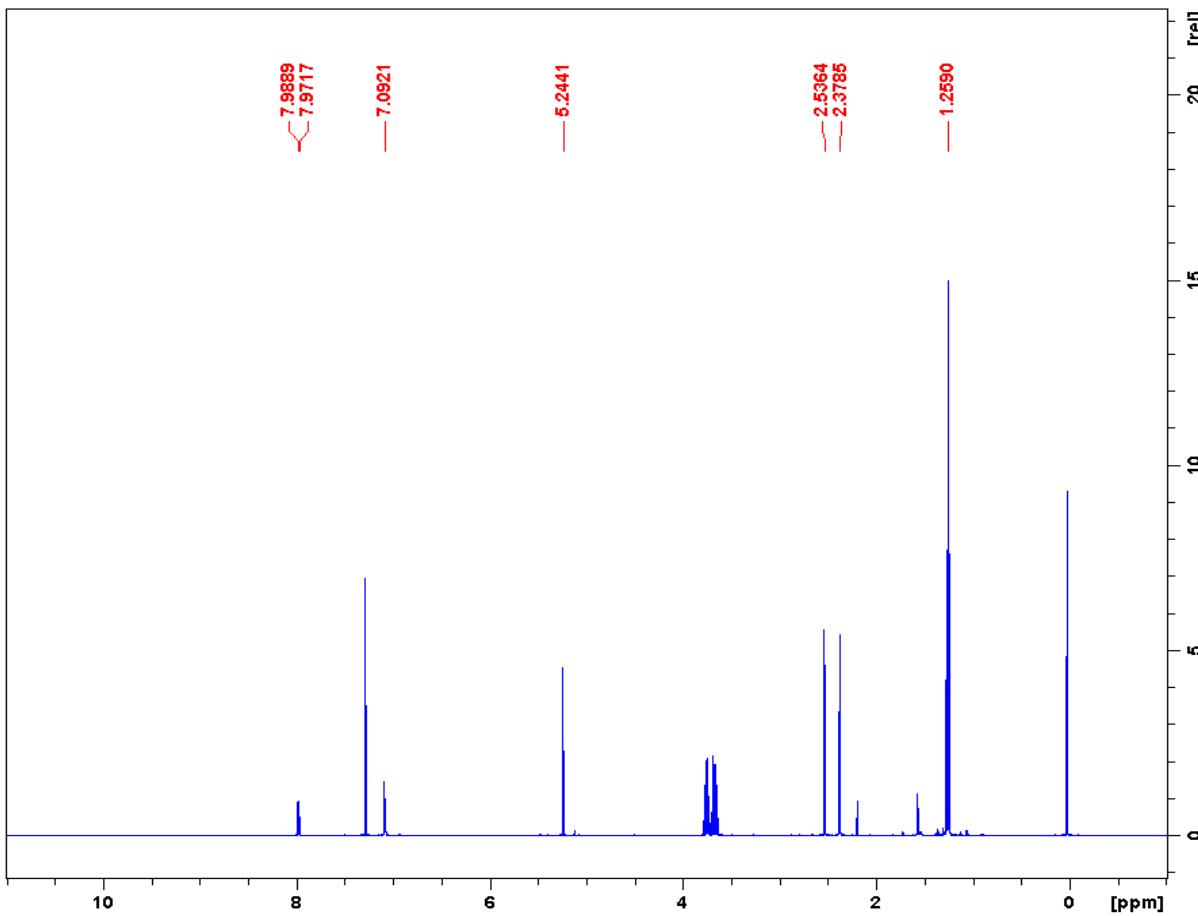


Figure 8 ¹H-NMR spectrum of 2,2-diethoxy-1-(2,4-dimethylphenyl)ethanone

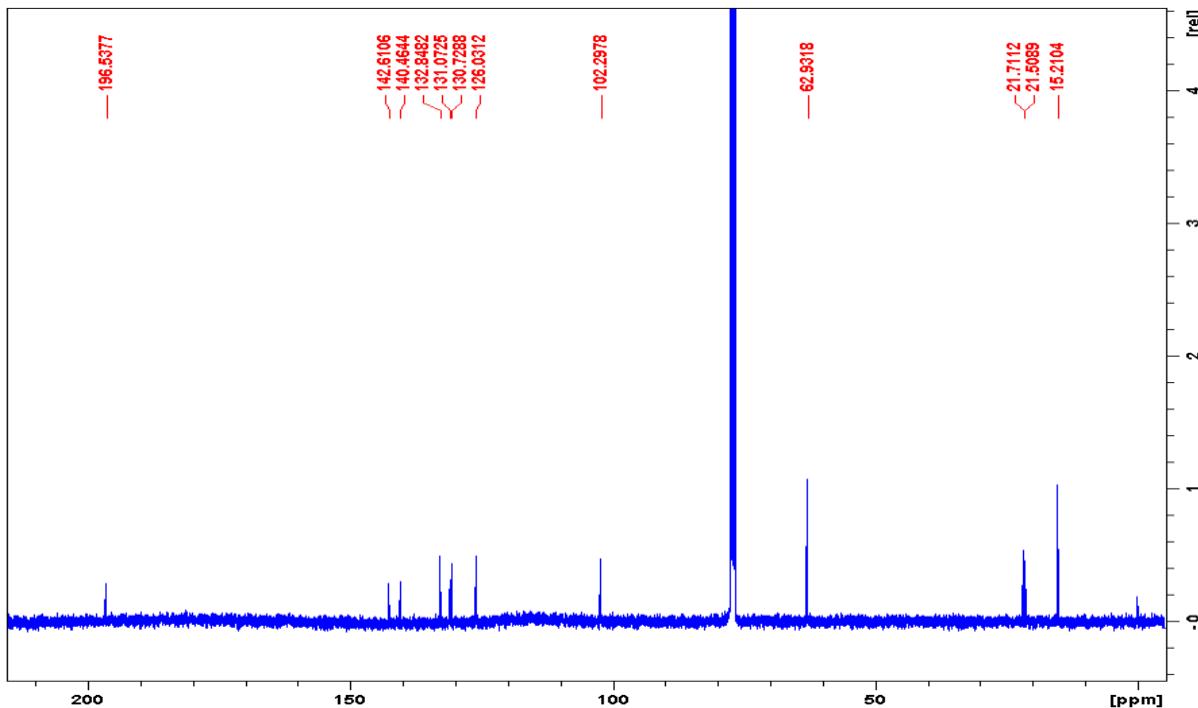


Figure 9 ¹³C-NMR spectrum of 2,2-diethoxy-1-(2,4-dimethylphenyl)ethanone

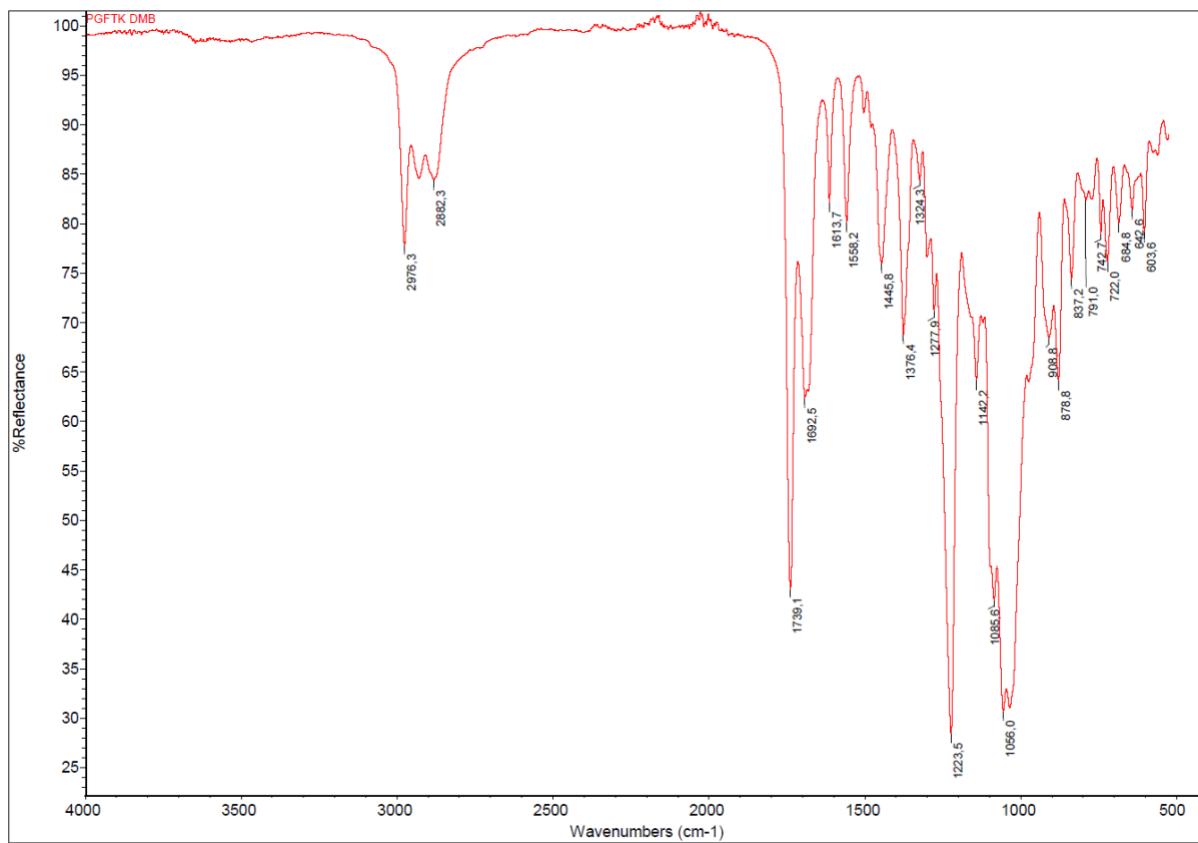
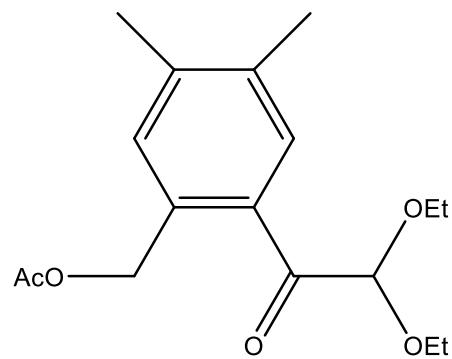
(2-Diethoxyacetyl-4,5-dimethylphenyl)methyl acetate (**7a**)

Figure 10 IR spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)methyl acetate

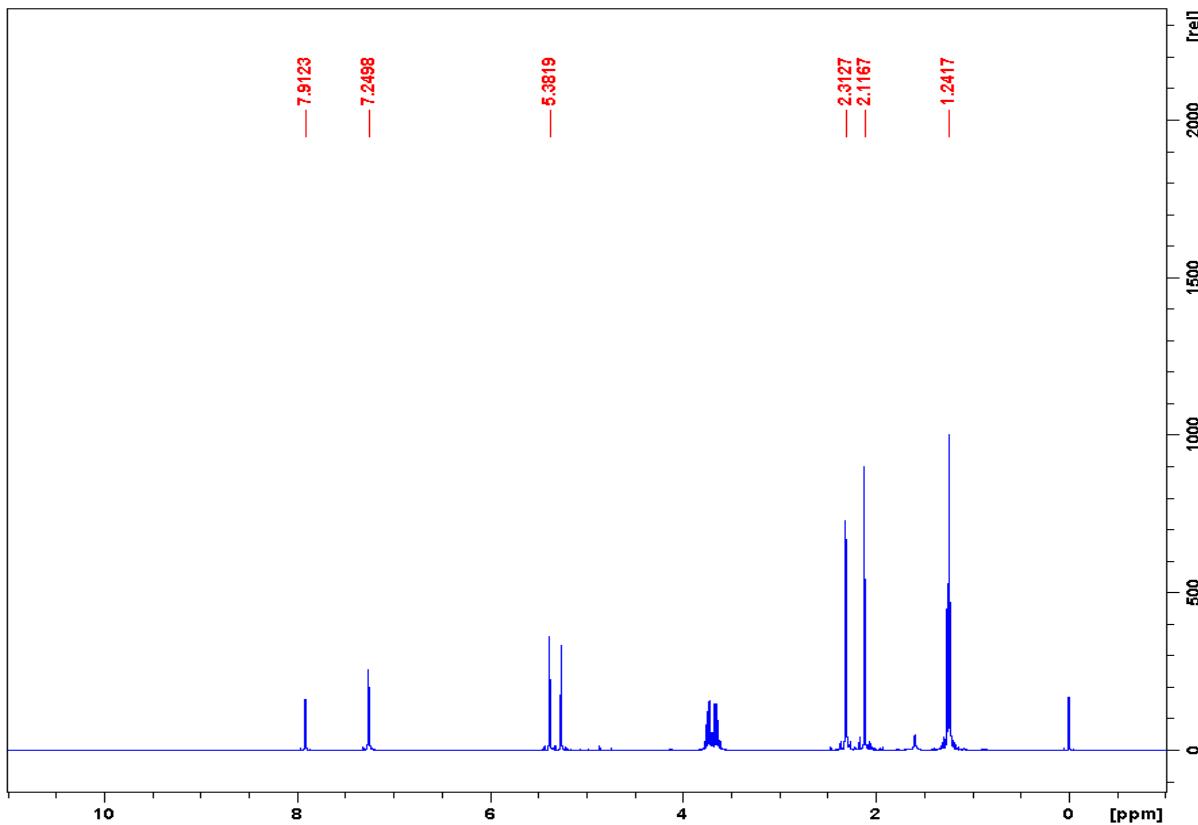


Figure 11 ¹H-NMR spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)methyl acetate

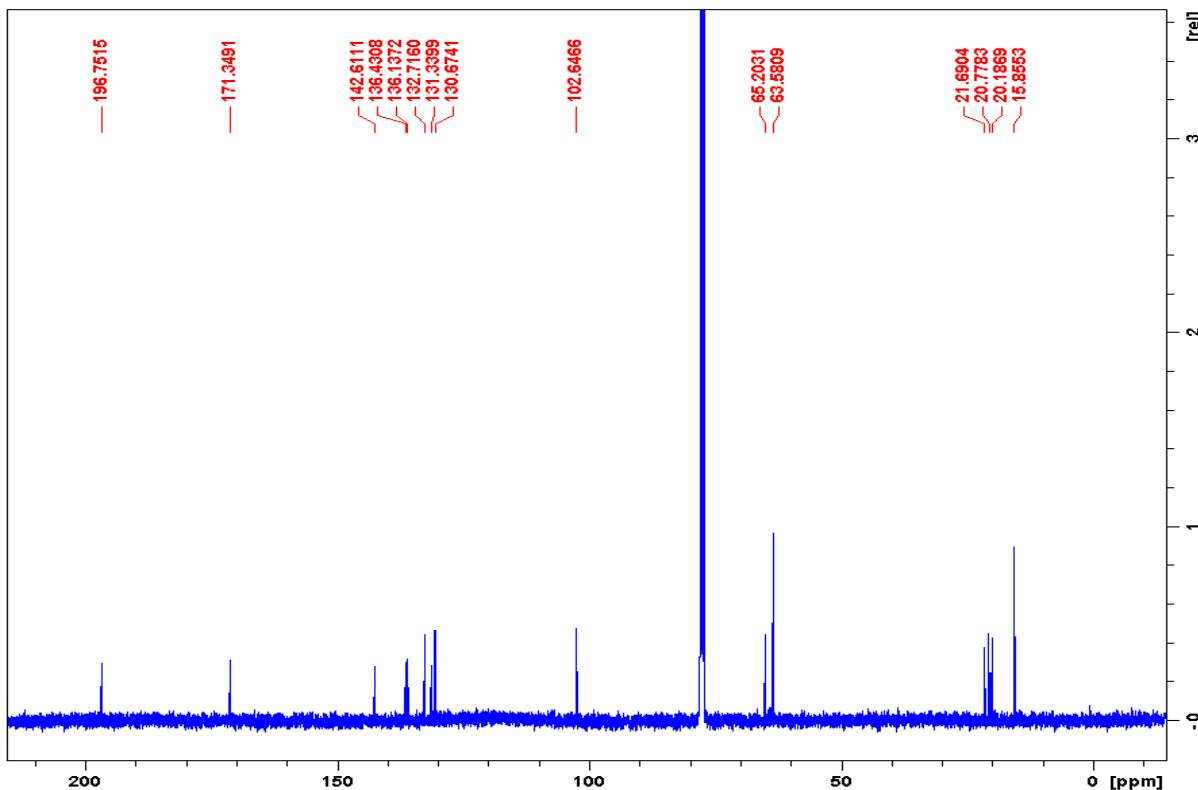


Figure 12 ¹³C-NMR of (2-diethoxyacetyl-4,5-dimethylphenyl)methyl acetate

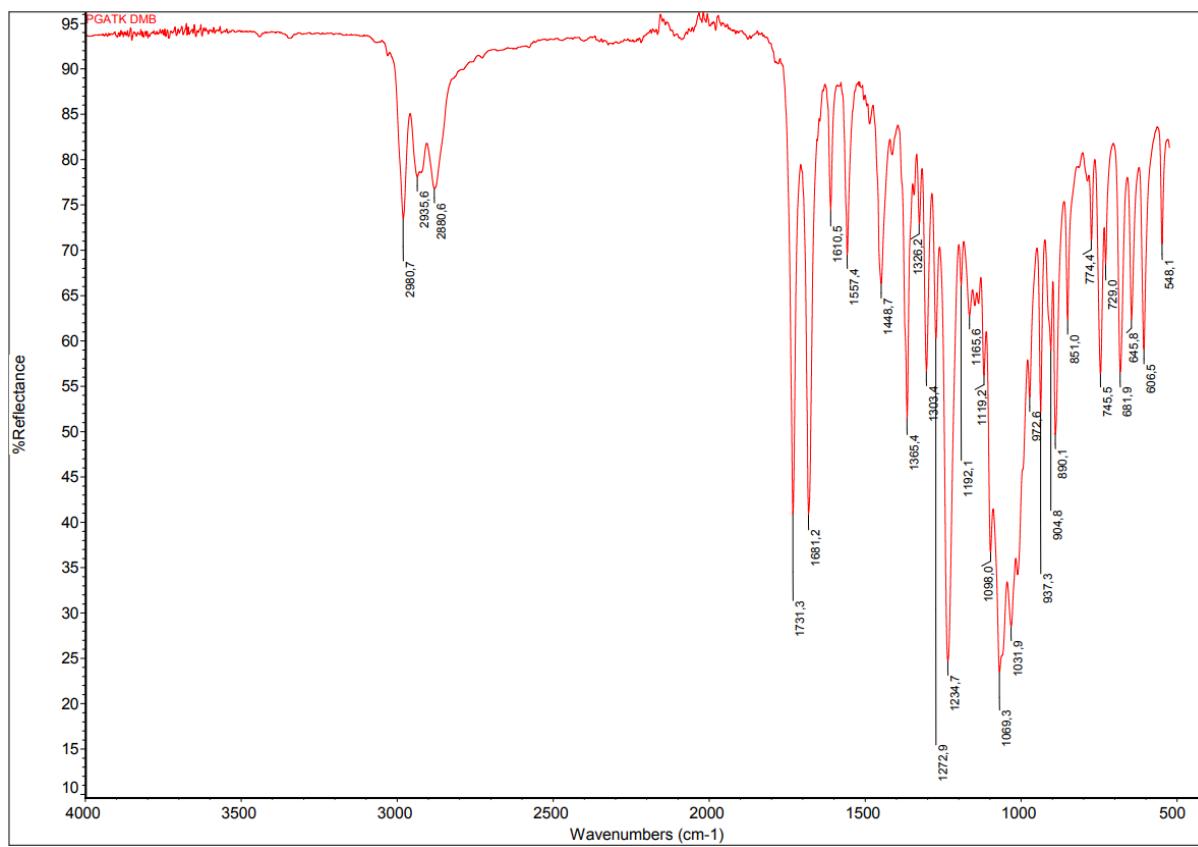
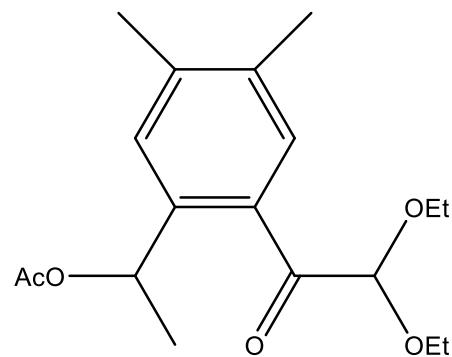
1-(2-Diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate (**7b**)

Figure 13 IR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate

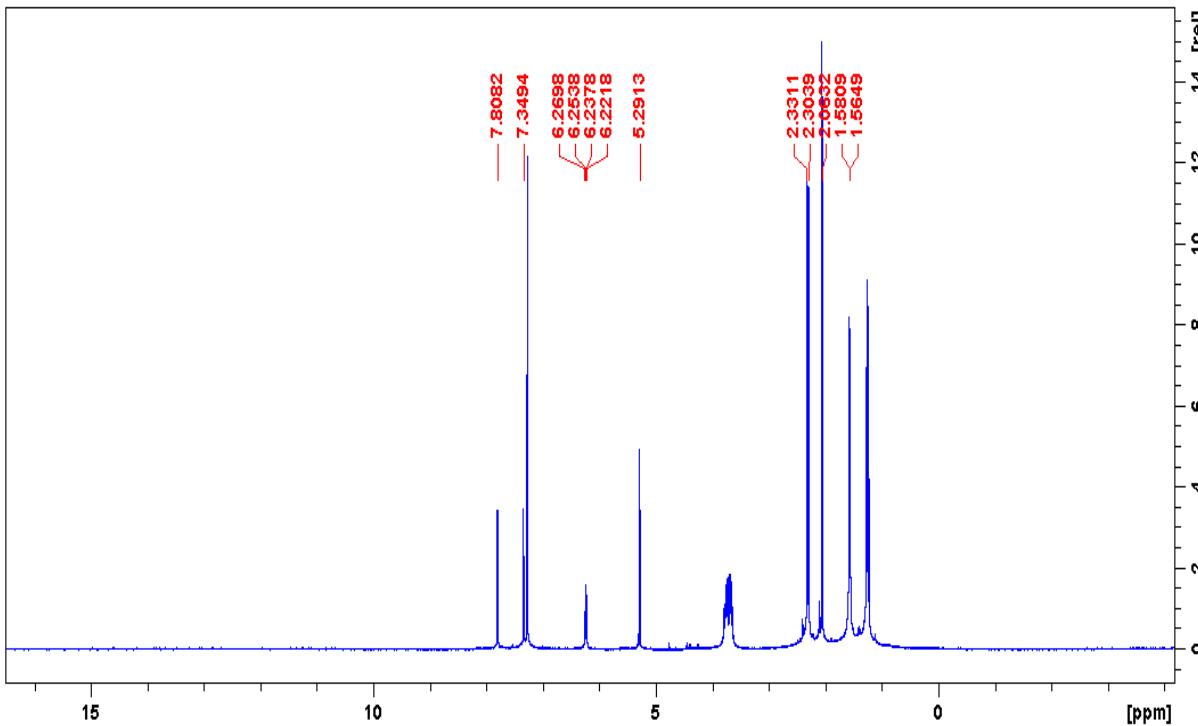


Figure 14 ¹H-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate

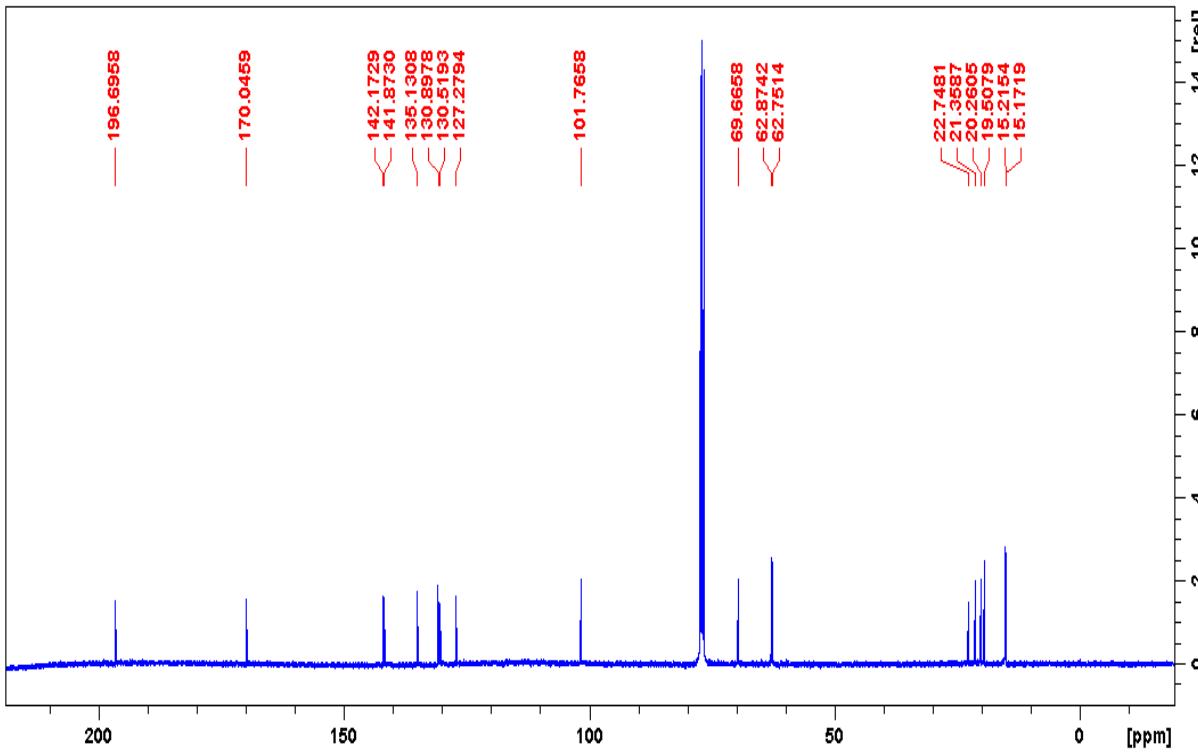


Figure 15 ¹³C-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate

1-(2-Diethoxyacetyl-4,5-dimethylphenyl)octyl acetate (**7c**)

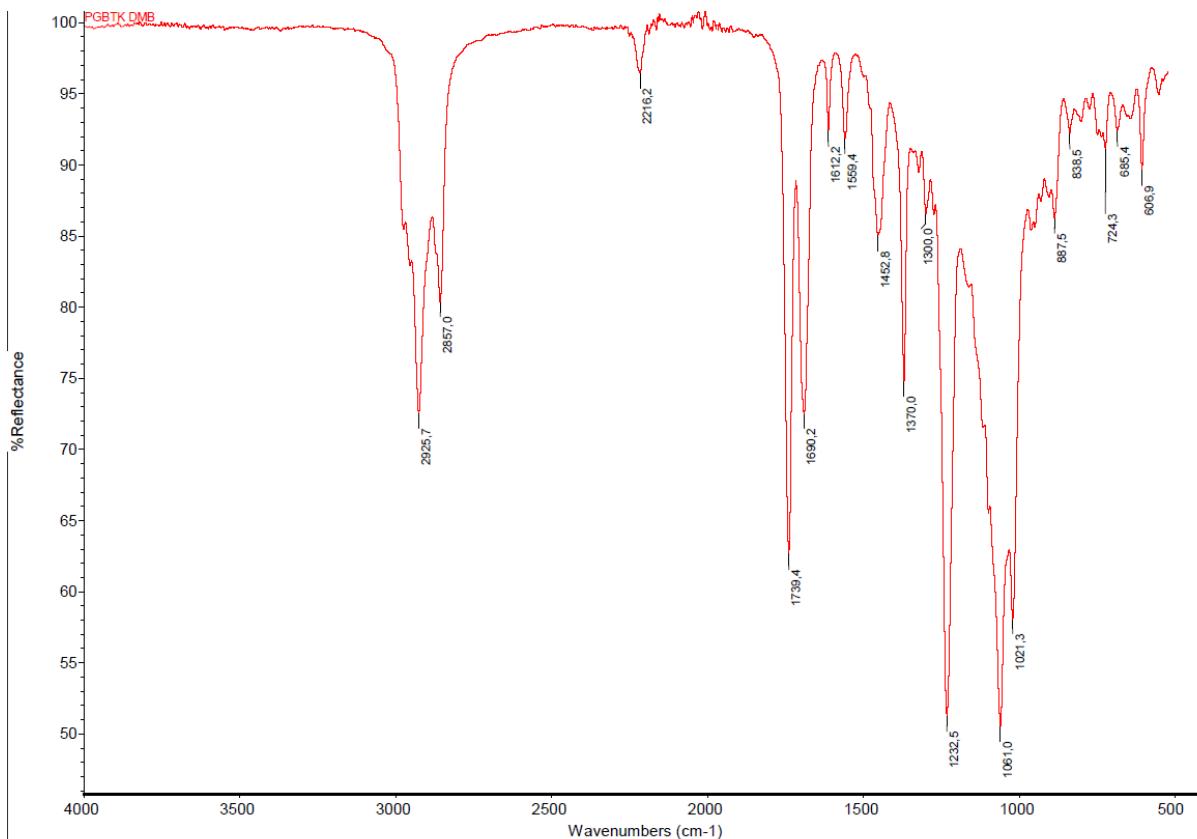
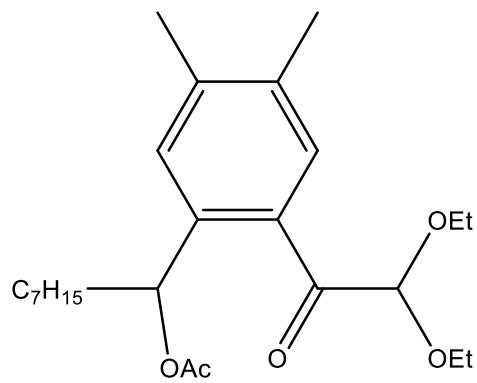


Figure 16 IR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)octyl acetate

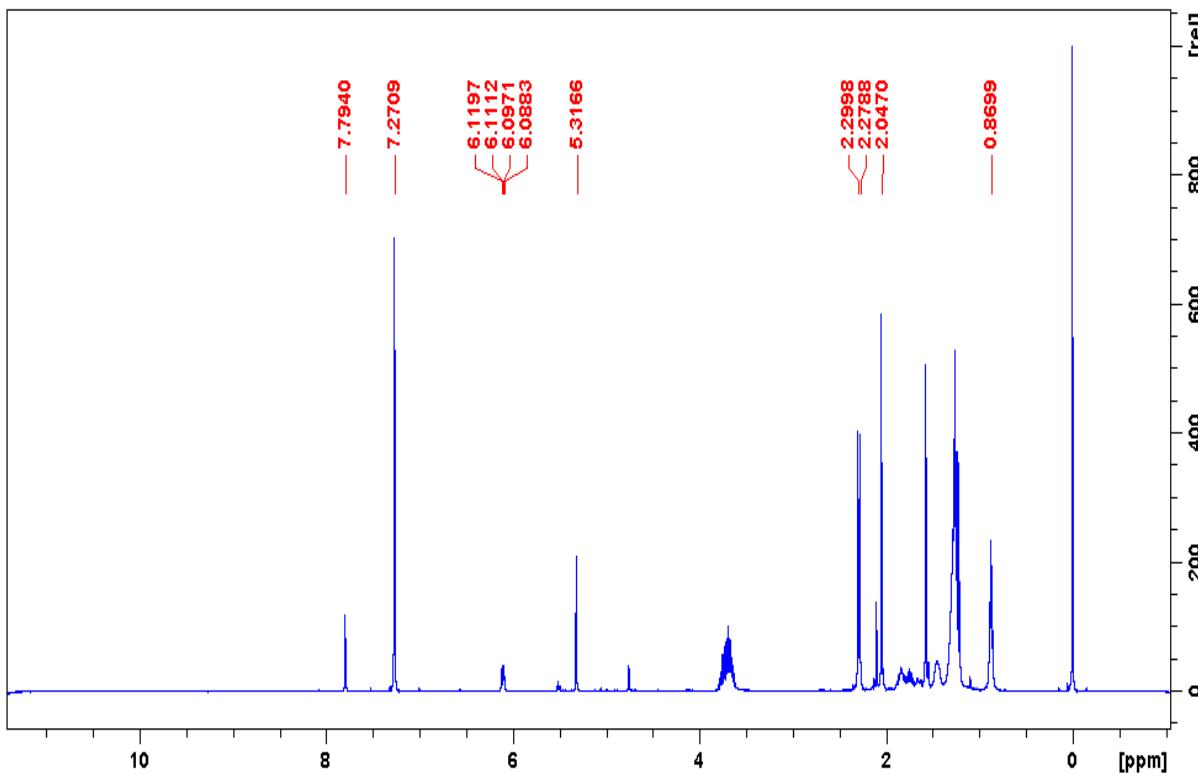


Figure 17 ¹H-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)octyl acetate

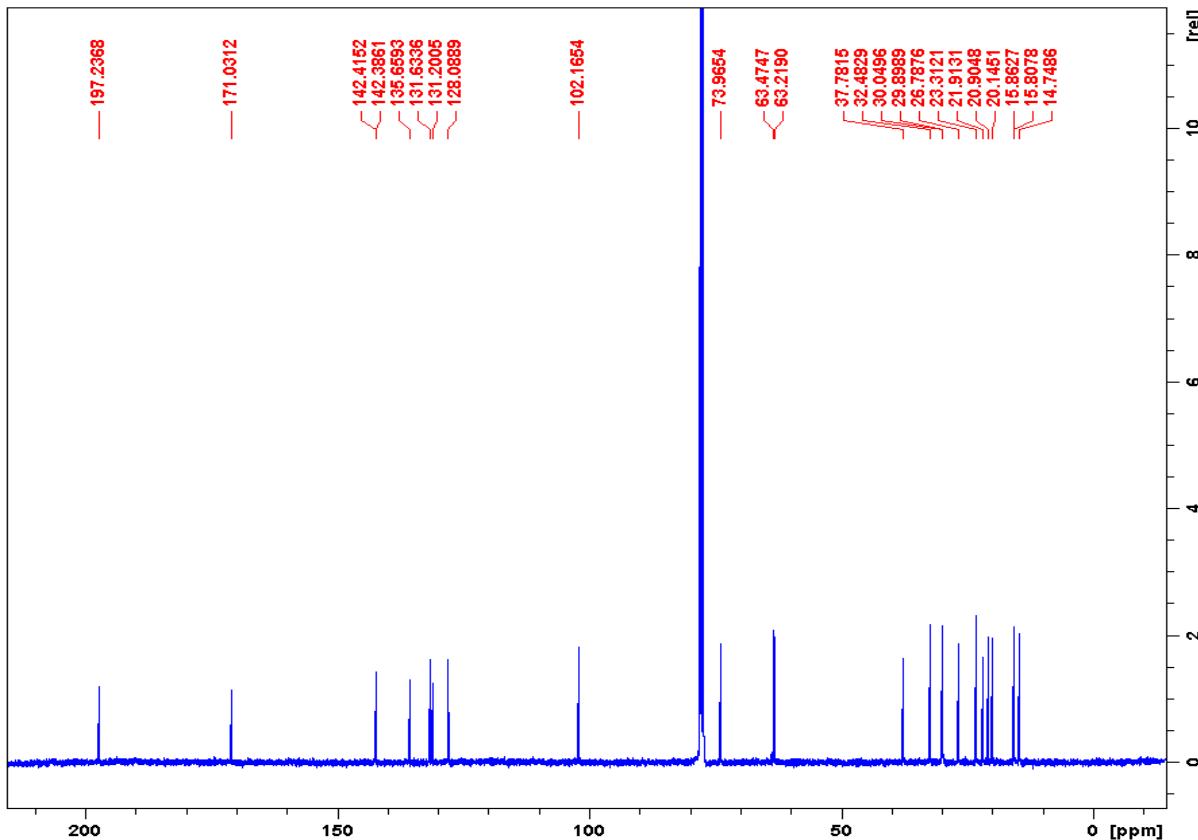


Figure 18 ¹³C-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)octyl acetate

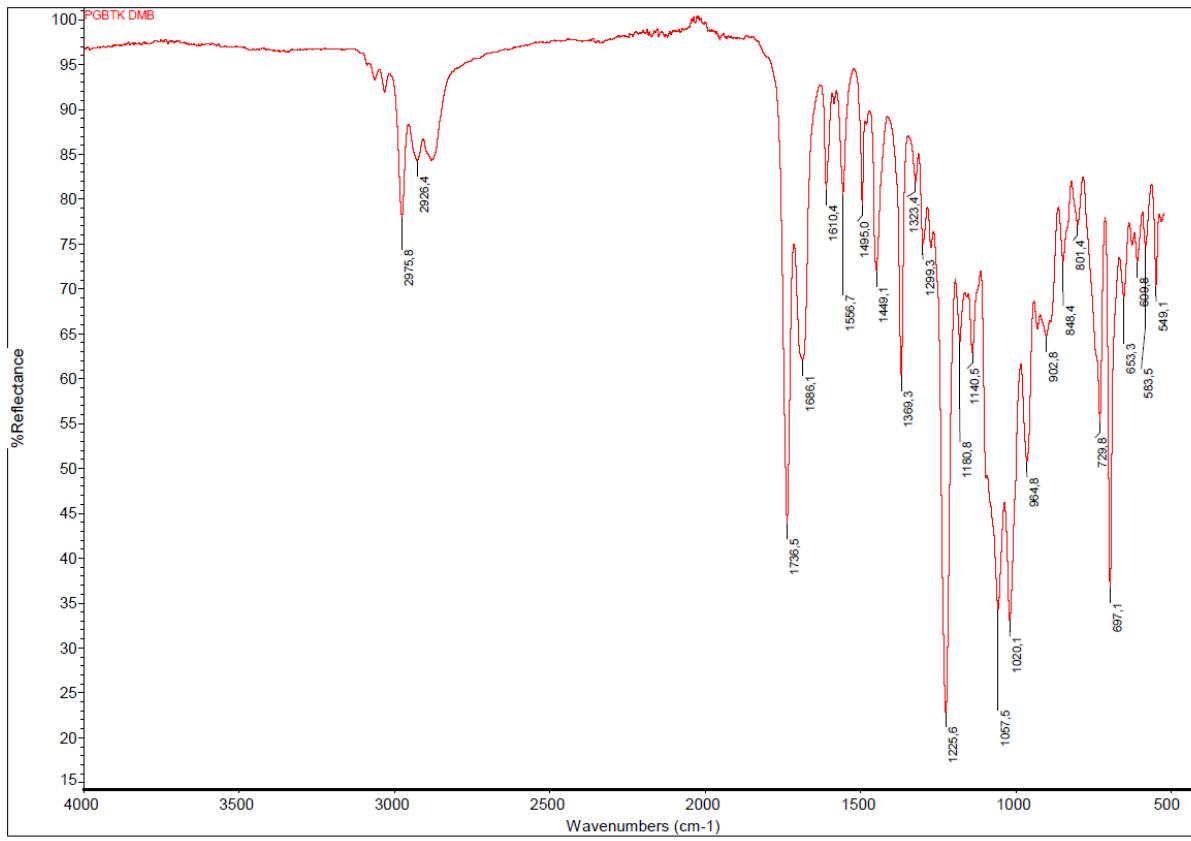
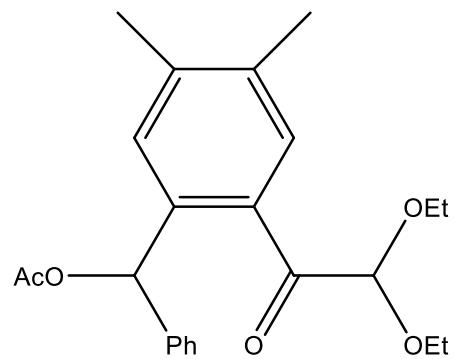
(2-Diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate (**7d**)

Figure 19 IR spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate

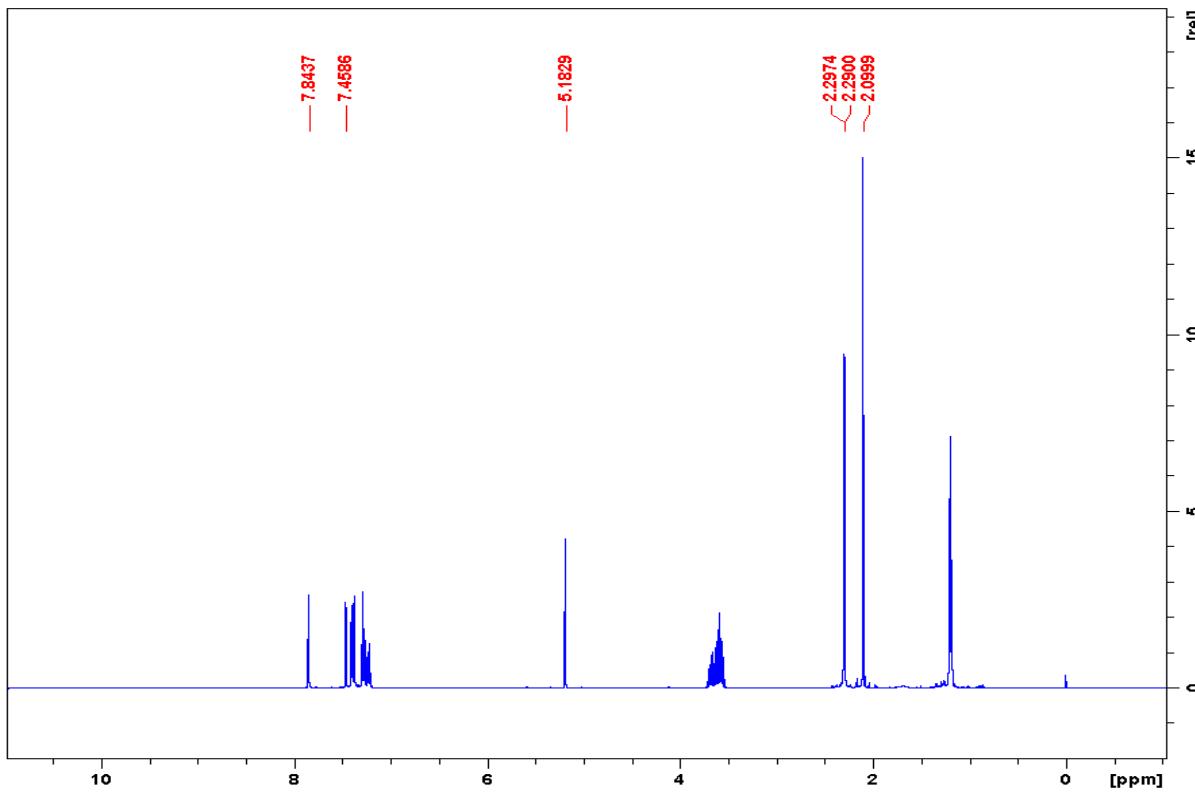


Figure 20 ^1H -spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate

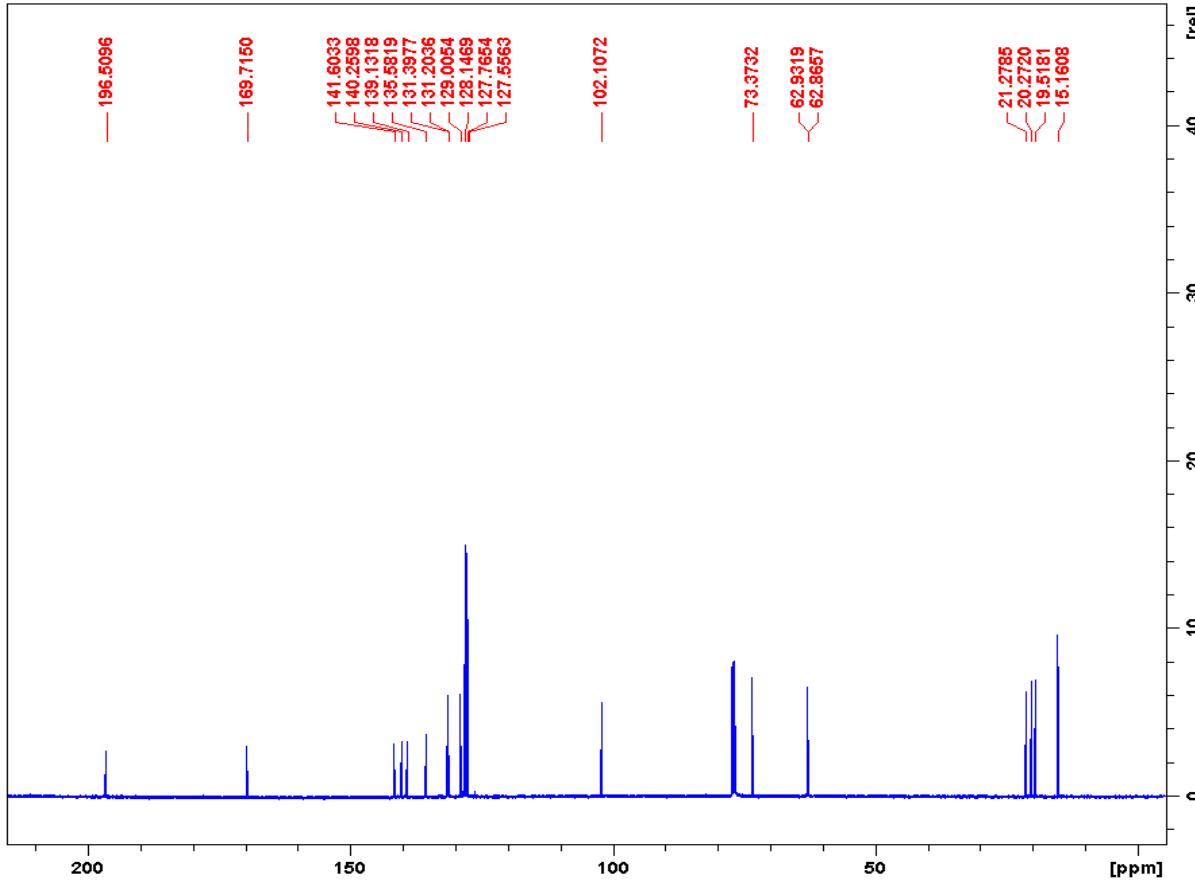


Figure 21 ^{13}C -spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate