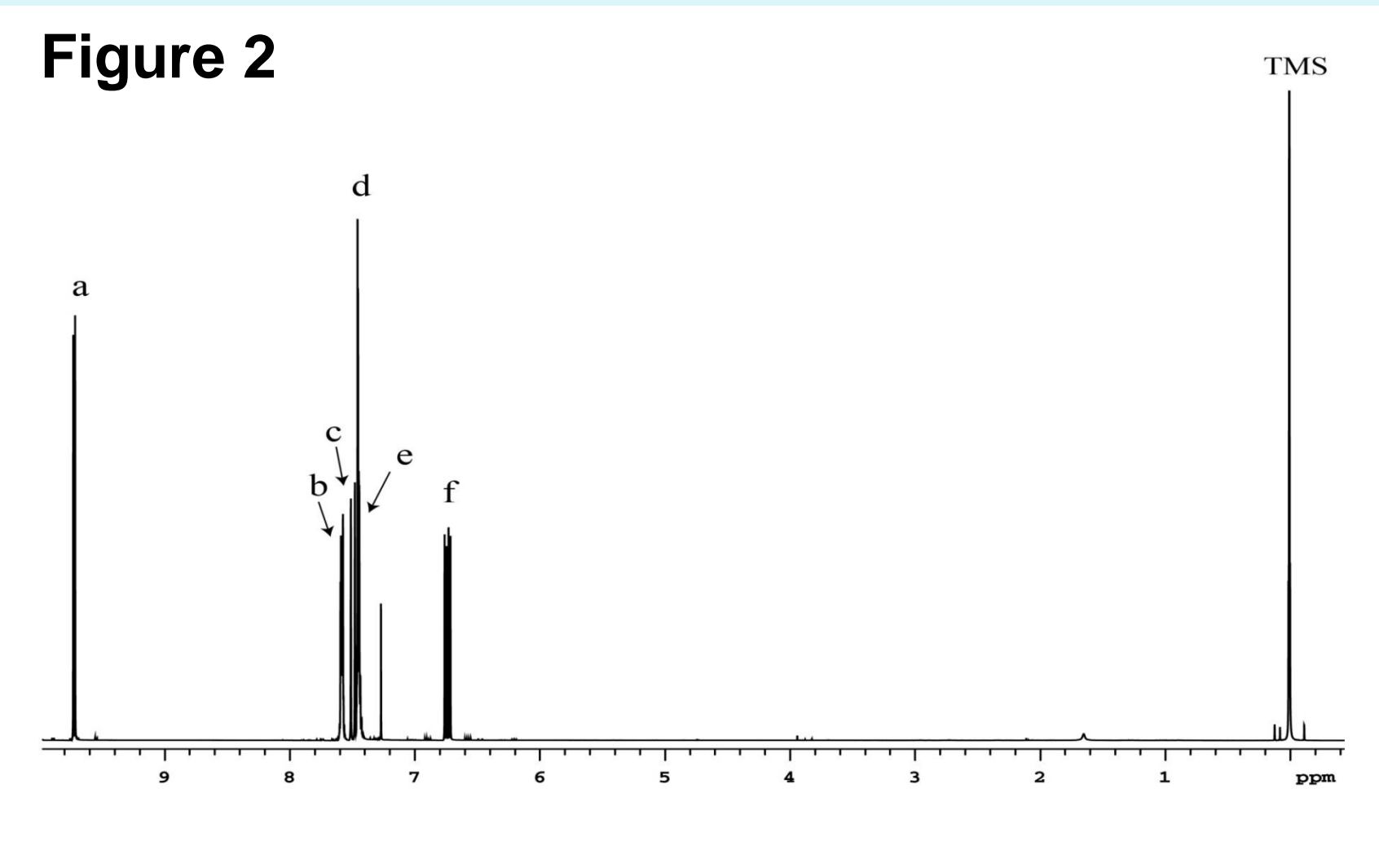


Cinnamaldehyde is the primary organic molecule that gives cinnamon its characteristic scent and zest. This compound is composed of a monosubstituted benzene ring, a trans-alkene, and an aldehyde functional group.

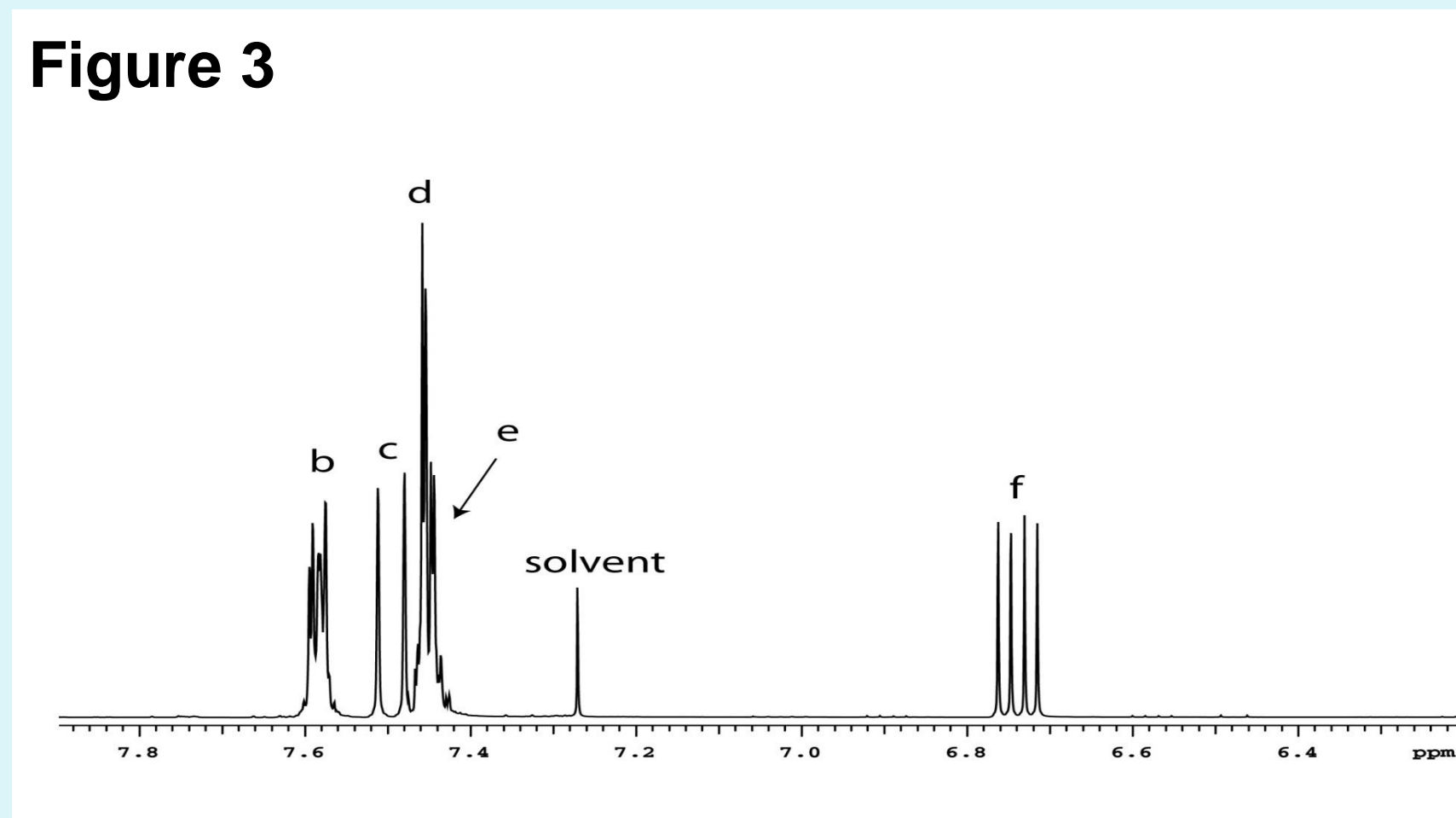
The molecular formula and molecular weight of cinnamaldehyde is  $C_9H_8O$  and 132.16g/mol, respectively. The structure of cinnamaldehyde is shown in Figure 1.

## $^1H$ 1D NMR Spectrum

The  $^1H$  1D NMR spectrum of cinnamaldehyde is shown in Figure 2. The spectrum data was recorded at 500 MHz ( $^1H$ ) with a spectral width of 6038.65 Hz and 8192 complex points. The solvent peak ( $CDCl_3$ ) appears at 7.27 ppm. Resonance HA is the most downfield doublet appearing at 9.73 ppm.



The chemical shift of HA is indicative of the aldehyde proton at C1. Figure 3 is an expanded view of the aromatic and alkene region



The molecule has two alkene protons that are trans to each other. In order to distinguish between the two, splitting patterns were

used. H3 would be split into a doublet by the proton bound to C2, whereas H2 would be split into a doublet of doublets due to the presence of the two surrounding nonequivalent protons at C3 and C1. Resonance F at 6.74 ppm is a doublet of doublets (15.732 Hz, 7.538 Hz) and is assigned to H2.

## $^1H$ NMR Assignments (ppm)

H1	9.73
H2	6.74
H3	7.48
H5/H9	7.58
H6/H8	7.46
H7	7.45

The remaining alkene proton, HC, is assigned to H3, a doublet ( $J=15.974Hz$ ) found at 7.48 ppm. The aromatic region has a line of symmetry running through the carbons labeled 4 and 7, giving rise to three unique aromatic proton and carbon resonances. In this molecule, C5 and C9, C6 and C8, are considered to be chemically equivalent. Definitive assignment of the remaining protons requires 2D NMR experimentation in order to resolve the remaining three protons.

## NOESY NMR Spectrum

Figure 4 shows the aromatic/alkene region of a NOESY experiment. The NOESY was ran at 500 MHz ( $^1H$ ) with a spectral width of 6038.65 Hz and a MixN time of 500ms. The completely conjugated structure of cinnamaldehyde gives some rigidity to the molecule and places H5 and H2, resonance HF, close enough to experience the Nuclear Overhauser Effect, NOE.

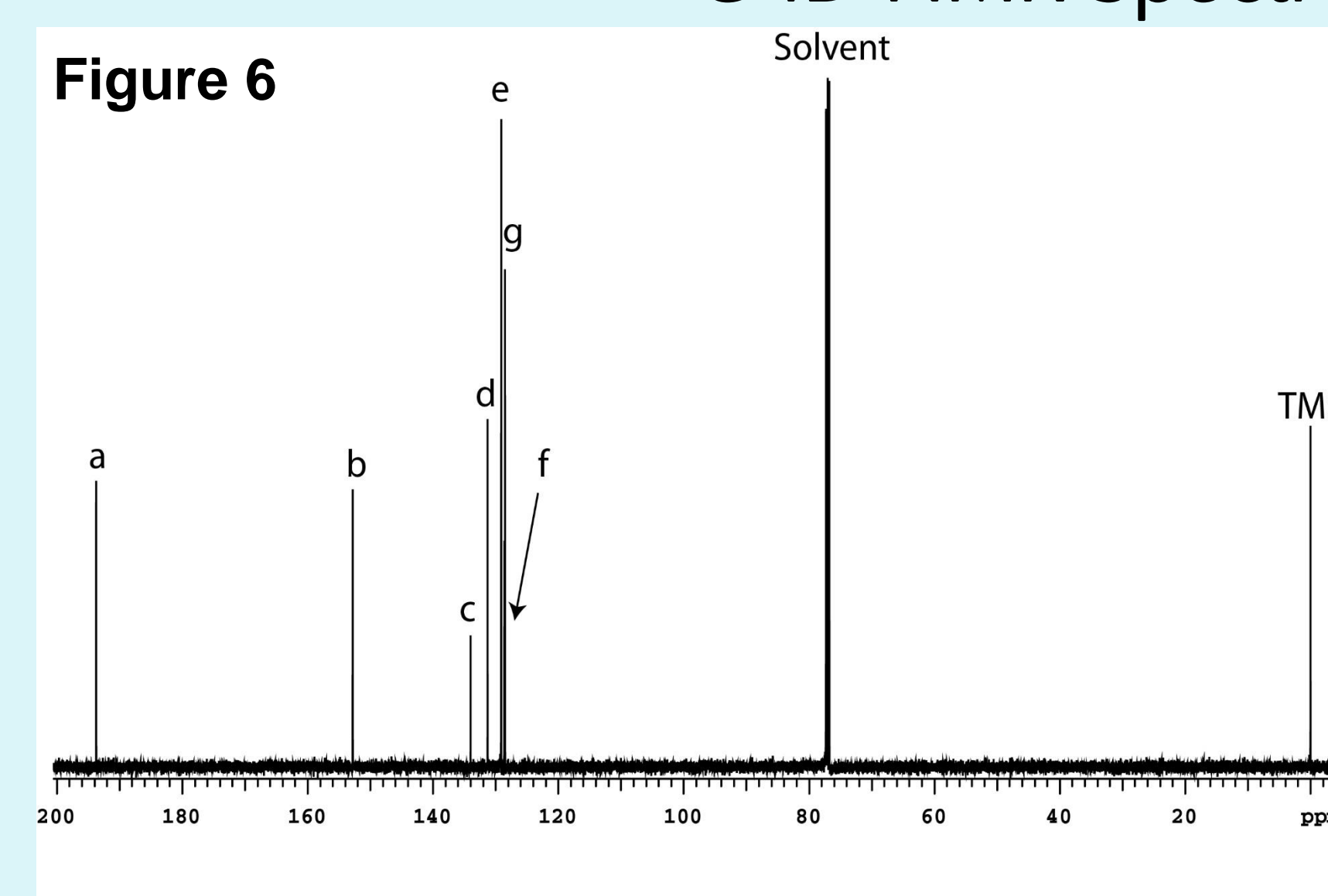
By inspecting the crosspeaks, it is observed that H2 is only coupled to resonance HB. Therefore, resonance HB, the doublet, belongs to H5/H9.

## gCOSY NMR Spectrum

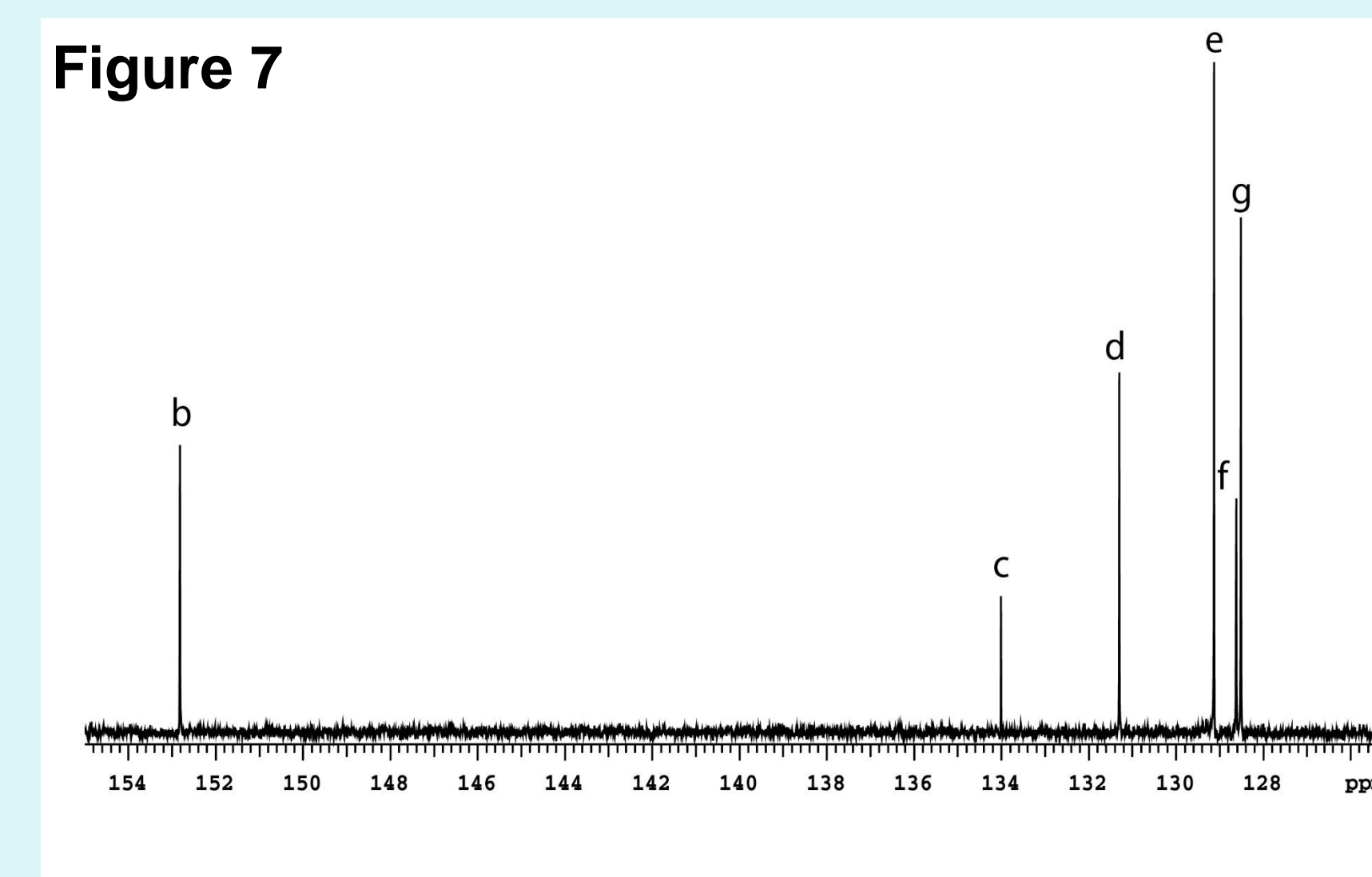
The two remaining unresolved aromatic protons will be deciphered using a gCOSY experiment. Figure 5 represents the aromatic region of a gCOSY spectrum that was recorded at 500 MHz ( $^1H$ ) with a spectral width of 6038.65 Hz and 1024 complex points. We'll start by inspecting the cross peak resonance that belongs to H5/H9.

H5/H9, resonance HB, is only coupled to H6/H8; Thus, H6/H8 is resonance HD. By deduction we are able to assign resonance HE to H7. However, the gCOSY also shows that resonances HE and HD are coupled to each other providing further evidence. All of the protons have been assigned

## $^{13}C$ 1D NMR Spectrum



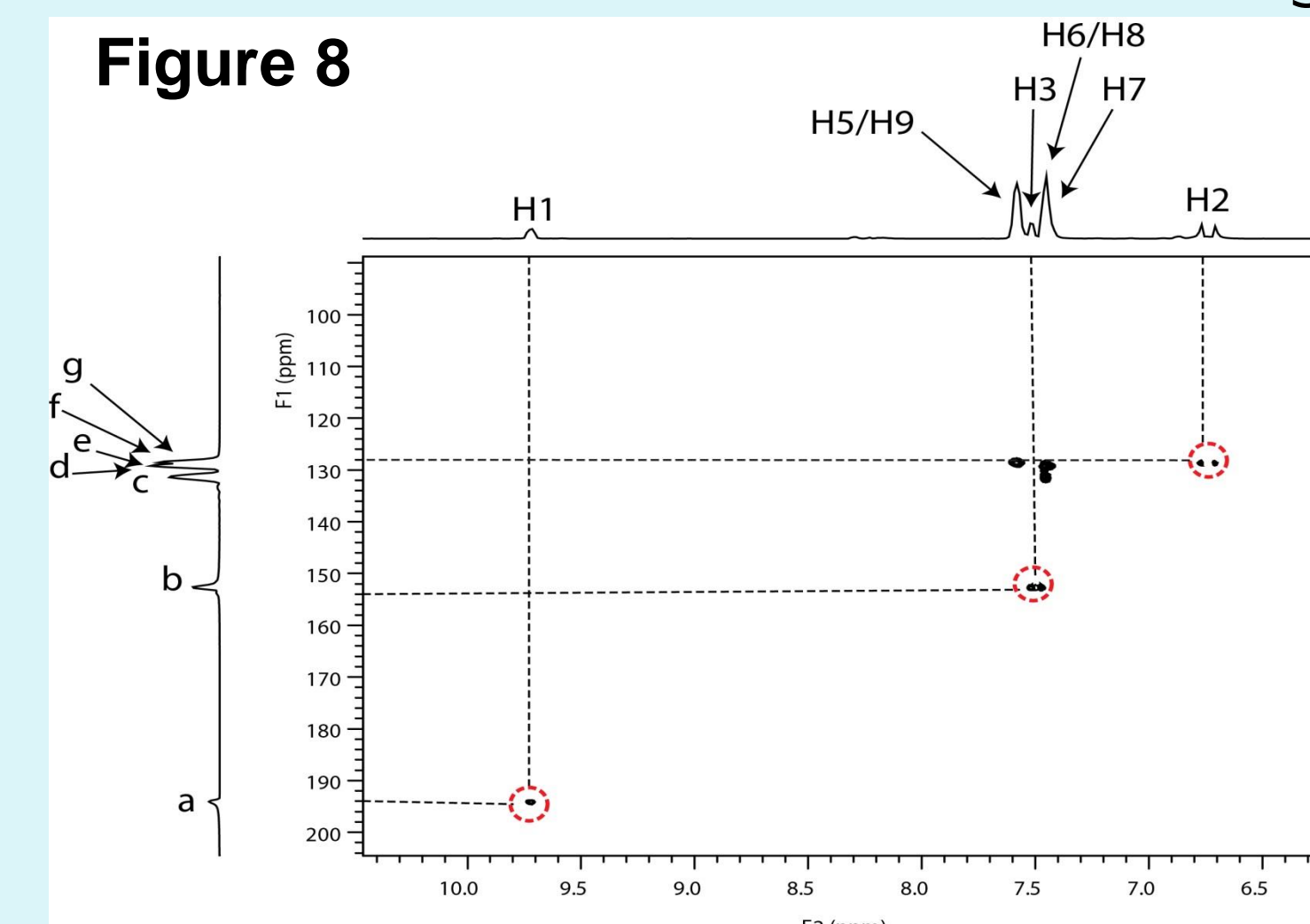
The  $^{13}C$  spectrum of cinnamaldehyde is shown in Figure 6. The spectrum was recorded at 500 MHz ( $^1H$ ) with a spectral width of 28409.1 Hz. Resonance CA, the most downfield resonance, belongs to C1, the carbonyl carbon. The three peaks at 77.3 belong to the solvent,  $CDCl_3$ . Figure 7 represents the aromatic region between 127 ppm and 153 ppm. This also includes the alkene carbons that are part of the conjugated system.



## gHMQC NMR Spectrum

Since all of the proton assignments have been made, a 2-Dimensional gHMQC will help resolve the peaks that pertain to the aromatic/alkene region. The gHMQC spectrum was recorded at 500 MHz ( $^1H$ ) with a spectral width of 6038.65 Hz in the direct dimension and 28409.1 Hz in the indirect dimension. Figure 8 and 9 show the direct associations between the proton and carbon nuclei.

Consequently, CG correlates with H5/H9 and is therefore C5. CB correlates with H3 and is therefore C3, leading to CD being C2, CE being C6/C8, and CF being C7. By

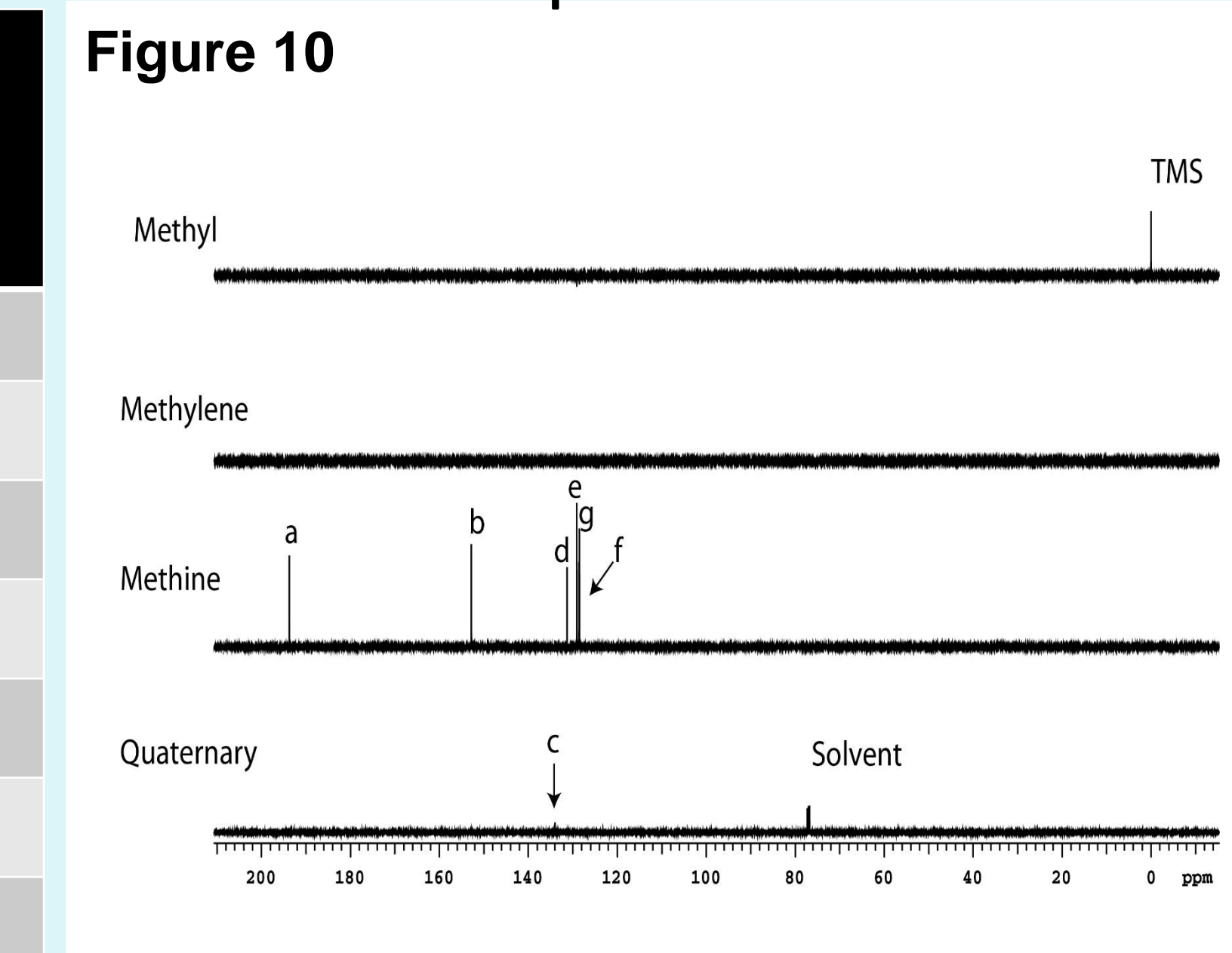


process of elimination, we can go ahead and assign peak CC to C4. Confirmation of these assignments is accomplished by careful consideration of the results of the DEPT experiment, Figure 10.

## $^{13}C$ DEPT NMR 1D Spectrum

**Figure 10**

$^{13}C$ NMR Assignments (ppm)	Category
C1	193.75
C2	131.30
C3	152.82
C4	134.01
C5	129.13
C6	128.62
C7	128.52



The  $^{13}C$  DEPT spectrum was recorded at 500 MHz with a spectral width of 28409.1 Hz and a relax delay of 5s. The  $^{13}C$  DEPT shows the hybridization of the carbons pertaining to cinnamaldehyde. All but one carbon is  $sp^2$  hybridized, tertiary carbons. We can assign resonance CC to atomic position C4 located at 134 ppm.

## Results

By careful analysis of the 1D and 2D spectra of all of the protons and carbons have been assigned. We were able to assign some protons directly based on the fine structure of the  $^1H$ -1D spectrum. The assignment ambiguities were resolved using the NOESY and gCOSY experiments. Based on the assigned protons, assignment of the directly bound carbons was carried out using a gHMQC experiment. The DEPT, which provides derived sub-spectral analysis of the  $1D-^{13}C$  spectrum, provided key confirmation of resonance assignments.

## References

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