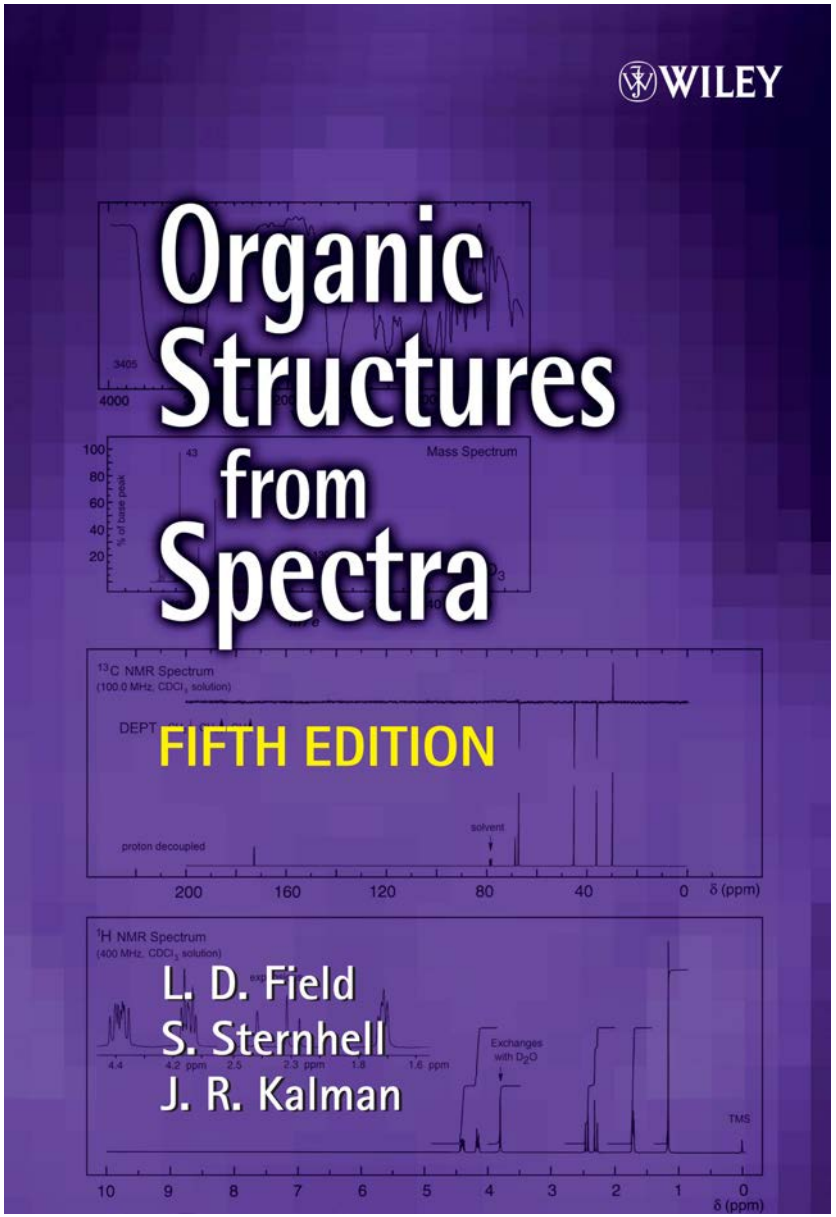
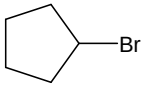
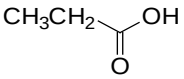
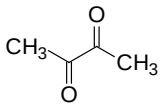
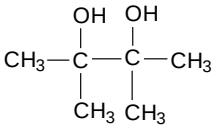
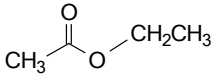
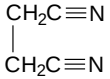
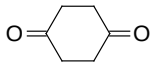
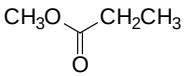
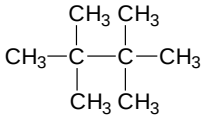
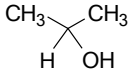
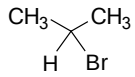
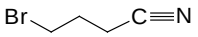
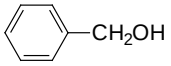
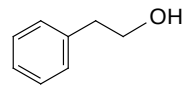
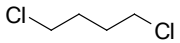
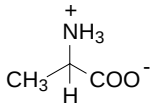
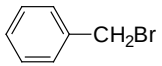
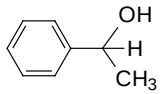
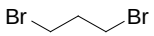
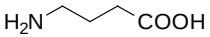
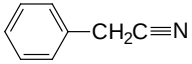
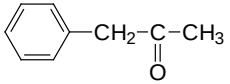
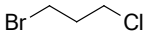
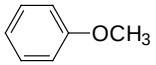
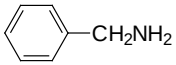
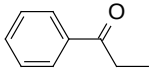


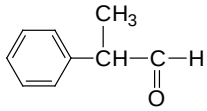
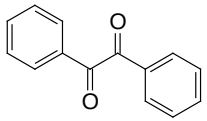
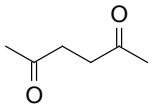
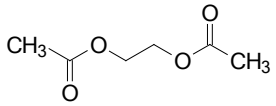
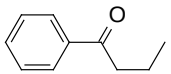
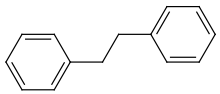
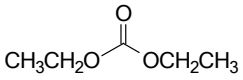
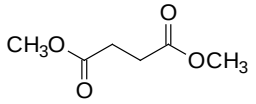
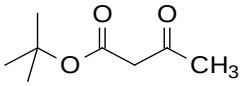
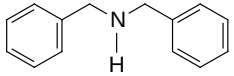
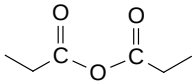
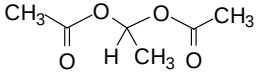
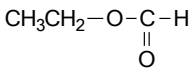
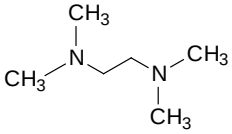
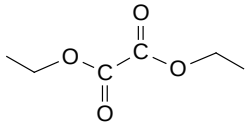
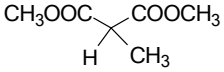
Solutions Manual

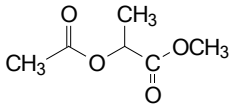
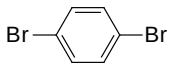
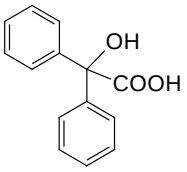
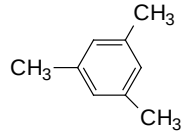
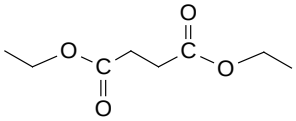
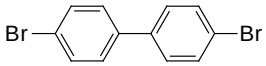
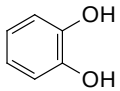
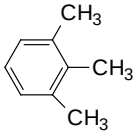
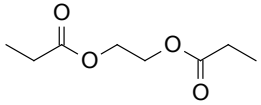
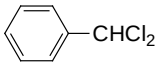
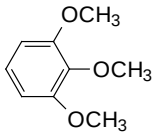
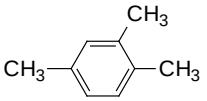
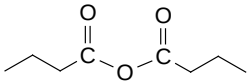
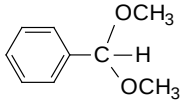
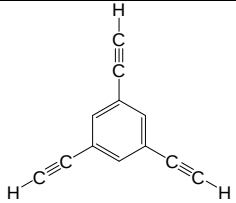
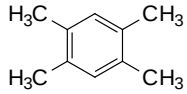
ORGANIC STRUCTURES FROM SPECTRA – 5th EDITION

L D Field, S Sternhell and J R Kalman

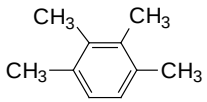
1  2-butanone C_4H_8O	**5** $BrCH_2-CH_2Br$ 1,2-dibromoethane $C_2H_4Br_2$	**9**  cyclopentane C_5H_{10}	**13**  bromocyclopentane C_5H_9Br
2  propionic acid $C_3H_6O_2$	**6**  1,2-butanedione (biacetyl) $C_4H_6O_2$	**10**  pinacol $C_6H_{14}O_2$	**14** CH_3CH_2-I iodoethane C_2H_5I
3  ethyl acetate $C_4H_8O_2$	**7**  succinonitrile $C_4H_4N_2$	**11**  1,4-cyclohexanedione $C_6H_8O_2$	**15** Cl_2CH-CH_3 1,1-dichloroethane $C_2H_4Cl_2$
4  methyl propionate $C_4H_8O_2$	**8**  2,2,3,3-tetramethylbutane C_8H_{18}	**12**  cyclopentanone $C_5H_8O_2$	**16**  2-propanol C_3H_8O

17  2-bromopropane C_3H_7Br	**21**  4-bromobutyronitrile C_4H_6NBr	**25**  benzyl alcohol C_7H_8O	**29**  2-phenylethanol $C_8H_{10}O$
18  1,4-dichlorobutane $C_4H_8Cl_2$	**22**  alanine $C_3H_7NO_2$	**26**  benzyl bromide C_7H_7Br	**30**  1-phenylethanol $C_8H_{10}O$
19  1,3-dibromopropane $C_3H_6Br_2$	**23**  4-aminobutyric acid $C_4H_9NO_2$	**27**  benzyl cyanide C_8H_7N	**31**  benzyl methyl ketone $C_9H_{10}O$
20  1-bromo-3-chloropropane C_3H_6BrCl	**24**  anisole C_7H_8O	**28**  benzylamine C_7H_9N	**32**  propiophenone $C_9H_{10}O$

33  2-phenylpropionaldehyde $C_9H_{10}O$	**37**  benzil $C_{14}H_{10}O_2$	**41**  2,5-hexanedione $C_6H_{10}O_2$	**45**  ethylene glycol diacetate $C_6H_{10}O_4$
34  butyrophenone $C_{10}H_{12}O$	**38**  1,2-diphenylethane $C_{14}H_{14}$	**42**  diethyl carbonate $C_5H_{10}O_3$	**46**  dimethyl succinate $C_6H_{10}O_4$
35  *t*-butyl acetoacetate $C_8H_{14}O_3$	**39**  dibenzylamine $C_{14}H_{15}N$	**43**  propionic anhydride $C_6H_{10}O_3$	**47**  1,1-diacetoxyethane $C_6H_{10}O_4$
36  ethyl formate $C_3H_6O_2$	**40**  *N,N,N,N*-tetramethyl-1,2-ethanediamine $C_6H_{16}N_2$	**44**  diethyl oxalate $C_6H_{10}O_4$	**48**  dimethyl methylmalonate $C_6H_{10}O_4$

49  methyl acetyllactate $C_6H_{10}O_4$	**53**  1,4-dibromobenzene $C_6H_4Br_2$	**57**  benzilic acid $C_{14}H_{12}O_3$	**61**  mesitylene C_9H_{12}
50  diethyl succinate $C_8H_{14}O_4$	**54**  4,4'-dibromobiphenyl $C_{12}H_8Br_2$	**58**  catechol $C_6H_6O_2$	**62**  1,2,3-trimethylbenzene C_9H_{12}
51  ethylene glycol dipropionate $C_8H_{14}O_4$	**55**  α,α-dichlorotoluene $C_7H_6Cl_2$	**59**  1,2,3-trimethoxybenzene $C_9H_{12}O_3$	**63**  1,2,4-trimethylbenzene C_9H_{12}
52  butyric anhydride $C_8H_{14}O_3$	**56**  benzaldehyde dimethylacetal $C_9H_{12}O_2$	**60**  1,3,5-triethynylbenzene $C_{12}H_6$	**64**  durene $C_{10}H_{14}$

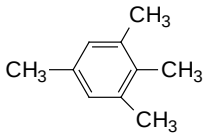
65



1,2,3,4-tetramethylbenzene

$C_{10}H_{14}$

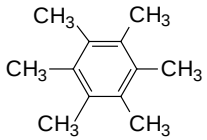
66



1,2,3,5-tetramethylbenzene

$C_{10}H_{14}$

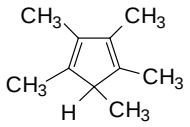
67



hexamethylbenzene

$C_{12}H_{18}$

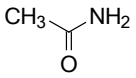
68



1,2,3,4,5,-pentamethylcyclopentadiene

$C_{10}H_{16}$

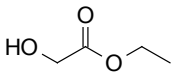
69



acetamide

C_2H_5NO

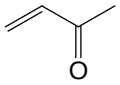
70



ethyl glycolate

$C_4H_8O_3$

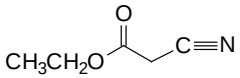
71



methyl vinyl ketone

C_4H_6O

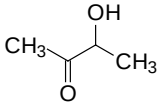
72



ethyl cyanoacetate

$C_5H_7NO_2$

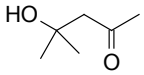
73



3-hydroxybutanone
(acetoin)

$C_4H_8O_2$

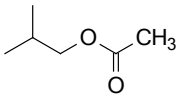
74



4-hydroxy-4-methyl-2-pentanone

$C_6H_{12}O_2$

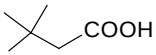
75



isobutyl acetate

$C_6H_{12}O_2$

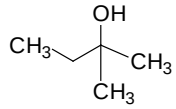
76



3,3-dimethylbutyric acid

$C_6H_{12}O_2$

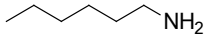
77



2-methyl-2-butanol
(*t*-amyl alcohol)

$C_5H_{12}O$

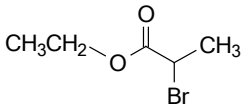
78



hexylamine

$C_6H_{15}N$

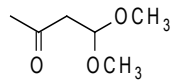
79



ethyl 2-bromopropionate

$C_5H_9O_2Br$

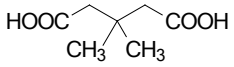
80



4,4-dimethoxy-2-butanone

$C_6H_{12}O_3$

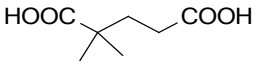
81



3,3-dimethylglutaric acid

$C_7H_{12}O_4$

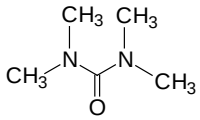
82



2,2-dimethylglutaric acid

$C_7H_{12}O_4$

83



tetramethylurea

$C_5H_{12}N_2O$

84

1,3-dioxane

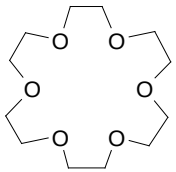
$C_4H_8O_2$

85

1,4-dioxane

$C_4H_8O_2$

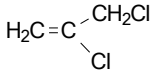
86



18-crown-6

$C_{12}H_{24}O_6$

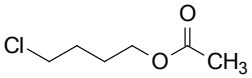
87



2,3-dichloropropene

$C_3H_4Cl_2$

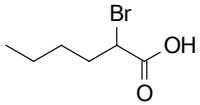
88



4-chlorobutyl acetate

$C_6H_{11}O_2Cl$

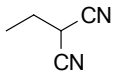
89



2-bromohexanoic acid

$C_6H_{11}O_2Br$

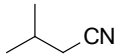
90



2-ethylmalononitrile

$C_5H_6N_2$

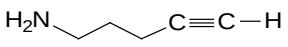
91



3-methylbutyronitrile

C_5H_9N

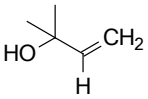
92



5-amino-1-pentyne

C_5H_9N

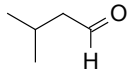
93



2-methylbut-3-en-2-ol

$C_5H_{10}O$

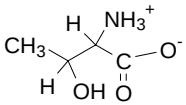
94



3-methylbutyraldehyde

$C_5H_{10}O$

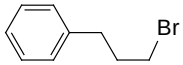
95



threonine

$C_4H_9NO_3$

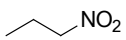
96



1-bromo-3-phenylpropane

$C_9H_{11}Br$

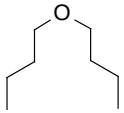
97



1-nitropropane

$C_3H_7NO_2$

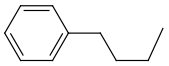
98



dibutyl ether

$C_8H_{18}O$

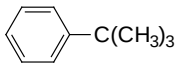
99



butylbenzene

$C_{10}H_{14}$

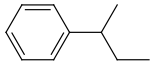
100



t-butylbenzene

$C_{10}H_{14}$

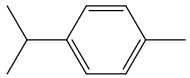
101



sec-butylbenzene

$C_{10}H_{14}$

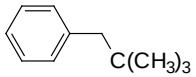
102



cymene

$C_{10}H_{14}$

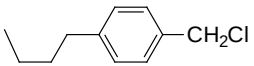
103



neopentylbenzene

$C_{11}H_{16}$

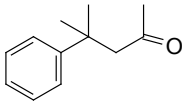
104



4-(*n*-butyl)-α-chlorotoluene

$C_{11}H_{15}Cl$

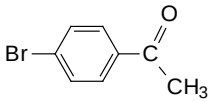
105



4-methyl-4-phenyl-2-pentanone

$C_{12}H_{16}O$

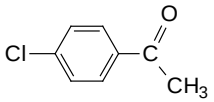
106



p-bromoacetophenone

C_8H_7OBr

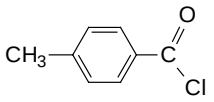
107



p-chloroacetophenone

C_8H_7OCl

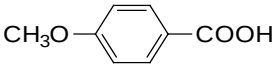
108



p-toluoyl chloride

C_8H_7OCl

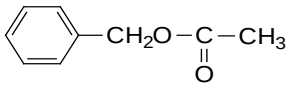
109



p-anisic acid

$C_8H_8O_3$

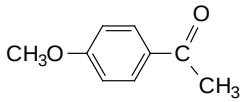
110



benzyl acetate

$C_9H_{10}O_2$

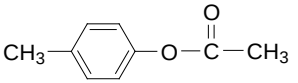
111



4-methoxyacetophenone

$C_9H_{10}O_2$

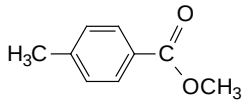
112



p-cresyl acetate

$C_9H_{10}O_2$

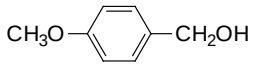
113



methyl *p*-toluate

$C_9H_{10}O_2$

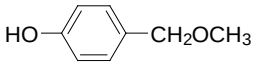
114



p-methoxybenzyl alcohol

$C_8H_{10}O_2$

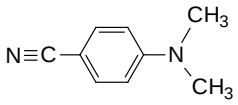
115



4-methoxymethylphenol

$C_8H_{10}O_2$

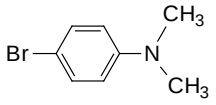
116



4-dimethylaminobenzonitrile

$C_9H_{10}N_2$

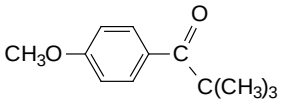
117



p-bromo-*N*,*N*-dimethylaniline

$C_8H_{10}NBr$

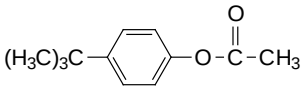
118



p-anisyl *t*-butyl ketone

$C_{12}H_{16}O_2$

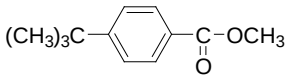
119



4-*t*-butylphenyl acetate

$C_{12}H_{16}O_2$

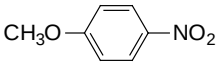
120



methyl 4-*t*-butylbenzoate

$C_{12}H_{16}O_2$

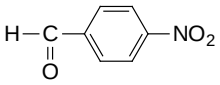
121



p-nitroanisole

$C_7H_7NO_3$

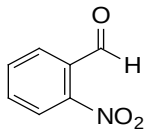
122



p-nitrobenzaldehyde

$C_7H_5NO_3$

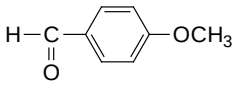
123



o-nitrobenzaldehyde

$C_7H_5NO_3$

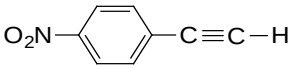
124



p-anisaldehyde

$C_8H_8O_2$

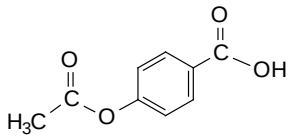
125



4-nitrophenylacetylene

$C_8H_5NO_2$

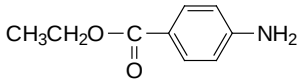
126



4-acetoxybenzoic acid

$C_9H_8O_4$

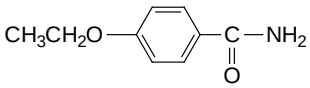
127



ethyl *p*-aminobenzoate

$C_9H_{11}NO_2$

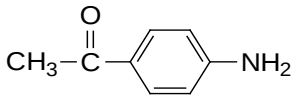
128



p-ethoxybenzamide

$C_9H_{11}NO_2$

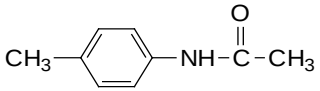
129



4-aminoacetophenone

C_8H_9NO

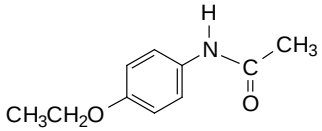
130



4-methylacetanilide

$C_9H_{11}NO$

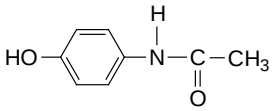
131



p-ethoxyacetanilide (phenacetin)

$C_{10}H_{13}NO_2$

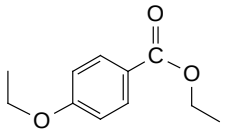
132



p-hydroxyacetanilide (paracetamol)

$C_8H_9NO_2$

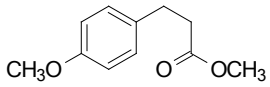
133



ethyl *p*-ethoxybenzoate

$C_{11}H_{14}O_3$

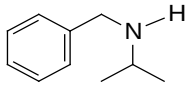
134



methyl (*p*-methoxyphenyl)-propionate

$C_{11}H_{14}O_3$

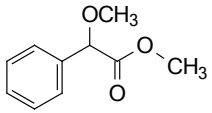
135



N-isopropylbenzylamine

$C_{10}H_{15}N$

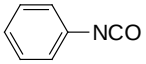
136



methyl 2-methoxy-2-phenylacetate

$C_{10}H_{12}O_3$

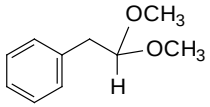
137



phenyl isocyanate

C_7H_5NO

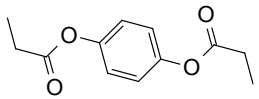
138



phenylacetaldehyde dimethyl acetal

$C_{10}H_{14}O_2$

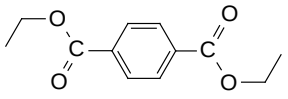
139



hydroquinone dipropionate

$C_{12}H_{14}O_4$

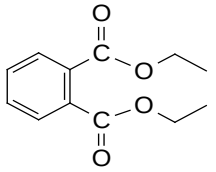
140



diethyl terephthalate

$C_{12}H_{14}O_4$

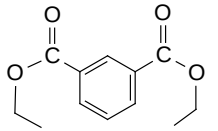
141



diethyl *o*-phthalate

$C_{12}H_{14}O_4$

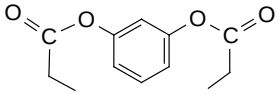
142



diethyl isophthalate

$C_{12}H_{14}O_4$

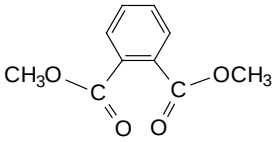
143



1,3-dihydroxyphenyl dipropionate

$C_{12}H_{14}O_4$

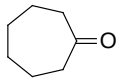
144



dimethyl *o*-phthalate

$C_{10}H_{10}O_4$

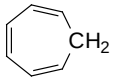
145



cycloheptanone

$C_7H_{12}O$

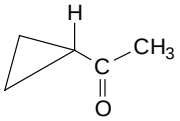
146



cycloheptatriene

C_7H_8

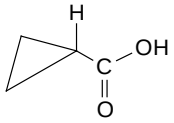
147



cyclopropyl methyl ketone

C_5H_8O

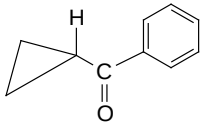
148



cyclopropane carboxylic acid

$C_4H_6O_2$

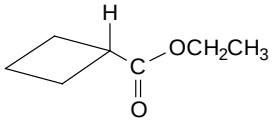
149



cyclopropyl phenyl ketone

$C_{10}H_{10}O$

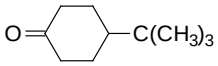
150



ethyl cyclobutane-carboxylate

$C_7H_{12}O_2$

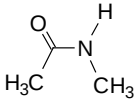
151



4-*t*-butylcyclohexanone

$C_{10}H_{18}O$

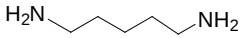
152



N-methylacetamide

C_3H_7NO

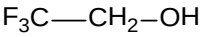
153



1,5-diaminopentane

$C_5H_{14}N_2$

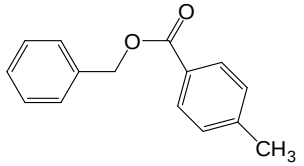
154



2,2,2-trifluoroethanol

$C_2H_3F_3O$

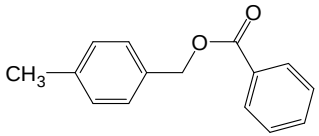
155



benzyl toluate

$C_{15}H_{14}O_2$

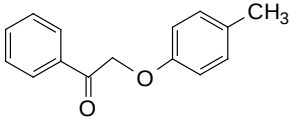
156



4-methylbenzyl benzoate

$C_{15}H_{14}O_2$

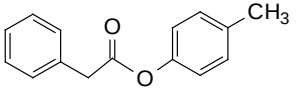
157



(*p*-cresyl)methyl phenyl ketone

$C_{15}H_{14}O_2$

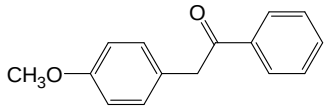
158



p-cresyl phenylacetate

$C_{15}H_{14}O_2$

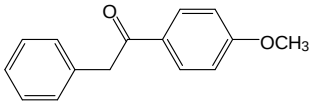
159



p-methoxybenzyl phenyl ketone

$C_{15}H_{14}O_2$

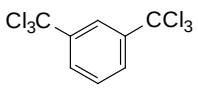
160



benzyl 4-methoxyphenyl ketone

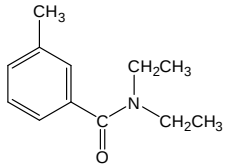
$C_{15}H_{14}O_2$

161



1,3-bis(trichloromethyl)-benzene
$C_8H_4Cl_6$

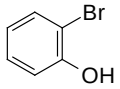
162



N,N-diethyl-*m*-toluamide

$C_{12}H_{17}NO$

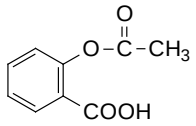
163



2-bromophenol

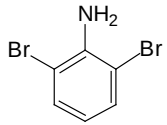
C_6H_5OBr

164



acetylsalicylic acid (aspirin)
$C_9H_8O_4$

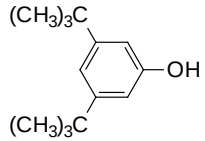
165



2,6-dibromoaniline

$C_6H_5NBr_2$

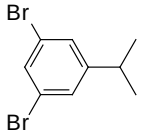
166



3,5-di-*t*-butylphenol

$C_{14}H_{22}O$

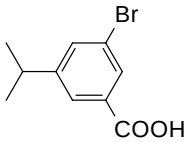
167



3,5-dibromocumene

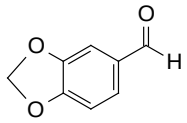
$C_9H_{10}Br_2$

168



3-bromo-5-isopropylbenzoic acid
$C_{10}H_{11}O_2Br$

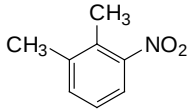
169



piperonal

$C_8H_6O_3$

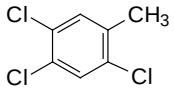
170



3-nitro-*o*-xylene

$C_8H_9NO_2$

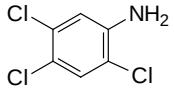
171



2,4,5-trichlorotoluene

$C_7H_5Cl_3$

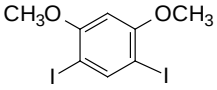
172



2,4,5-trichloroaniline

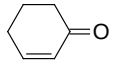
$C_6H_4NCl_3$

173



4,6-diiodo-1,3-dimethoxybenzene
$C_8H_8O_2I_2$

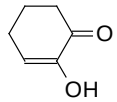
174



2-cyclohexene-1-one

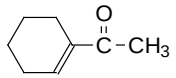
C_6H_8O

175



2-hydroxycyclohex-1-en-3-one
$C_6H_8O_2$

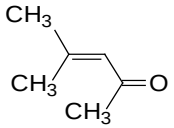
176



1-acetyl-1-cylohexene

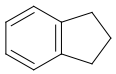
$C_8H_{12}O$

177



4-methylpent-3-en-2-one
(mesityl oxide)
$C_6H_{10}O$

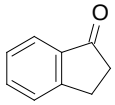
178



indane

C_9H_{10}

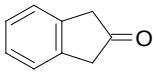
179



1-indanone

C_9H_8O

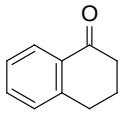
180



2-indanone

C_9H_8O

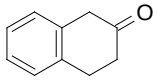
181



α-tetralone

$C_{10}H_{10}O$

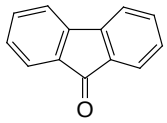
182



β-tetralone

$C_{10}H_{10}O$

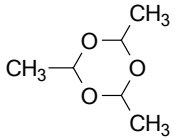
183



fluorenone

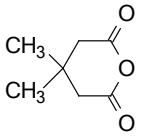
$C_{13}H_8O$

184



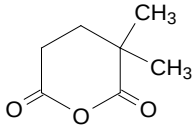
2,4,6-trimethyl-
1,3,5-trioxane
$C_6H_{12}O_3$

185



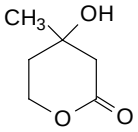
3,3-dimethylglutaric
anhydride
$C_7H_{10}O_3$

186



2,2-dimethylglutaric
anhydride
$C_7H_{10}O_3$

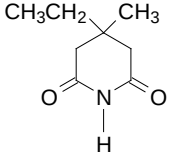
187



mevalonic lactone

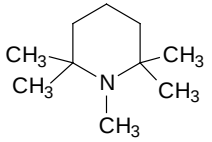
$C_6H_{10}O_3$

188



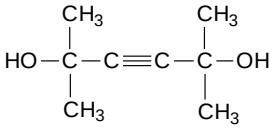
4-ethyl-4-methyl-2,6-
piperidinedione
$C_8H_{13}NO_2$

189



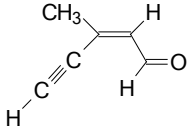
1,2,2,6,6-pentamethyl-
piperidine
$C_{10}H_{21}N$

190



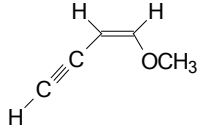
2,5-dimethyl-3-hexyne-
2,5-diol
$C_8H_{14}O_2$

191



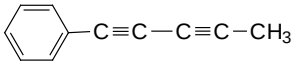
(*Z*)-3-methylpent-2-
en-4-ynal
C_6H_6O

192



(*Z*)-1-methoxybut-
1-en-4-yne
C_5H_6O

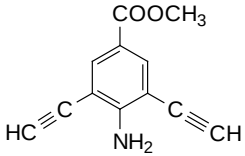
193



1-phenyl-1,3-pentadiyne

$C_{11}H_8$

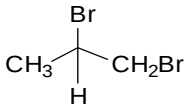
194



methyl 4-amino-3,5-diethynylbenzoate

$C_{12}H_9NO_2$

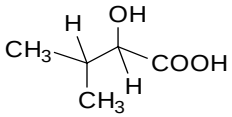
195



1,2-dibromopropane

$C_3H_6Br_2$

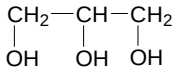
196



2-hydroxy-3-methylbutyric acid

$C_5H_{10}O_3$

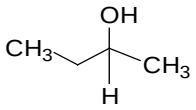
197



glycerol

$C_3H_8O_3$

198



2-butanol

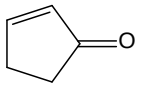
$C_4H_{10}O$

199

cyclopentene

C_5H_8

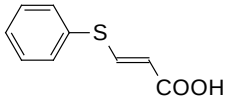
200



2-cyclopentenone

C_5H_6O

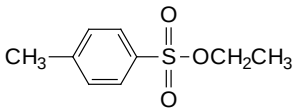
201



(*E*)-3-(phenylthio)acrylic acid

$C_9H_8O_2S$

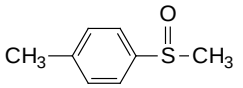
202



ethyl *p*-toluenesulfonate

$C_9H_{12}O_3S$

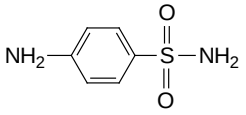
203



p-tolyl methyl sulfoxide

$C_8H_{10}OS$

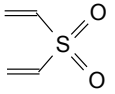
204



p-amino-benzenesulfonamide

$C_6H_8N_2O_2S$

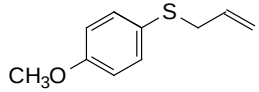
205



divinyl sulfone

$C_4H_6O_2S$

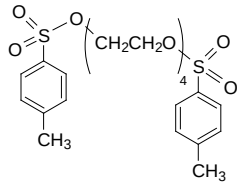
206



allyl *p*-anisyl thioether

$C_{10}H_{12}OS$

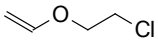
207



tetraethylene glycol ditosylate

$C_{22}H_{30}O_9S_2$

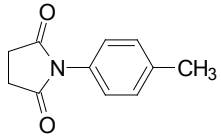
208



vinyl 2-chloroethyl ether

C_4H_7OCl

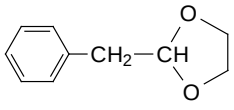
209



N-(*p*-tolyl)succinimide

$C_{11}H_{11}NO_2$

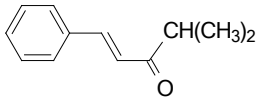
210



phenylacetaldeyde ethylene glycol acetal

$C_{10}H_{12}O_2$

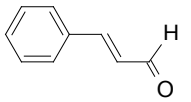
211



(*E*)-1-phenyl-4-methyl-1-penten-3-one

$C_{12}H_{14}O$

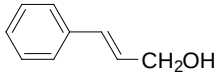
212



cinnamaldehyde

C_9H_8O

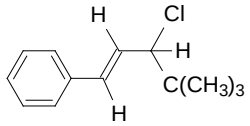
213



cinnamyl alcohol

$C_9H_{10}O$

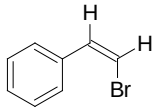
214



(*E*)-3-chloro-4,4-dimethyl-1-phenyl-1-pentene

$C_{13}H_{17}Cl$

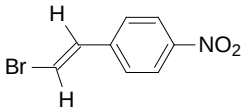
215



(*Z*)-β-bromostyrene

C_8H_7Br

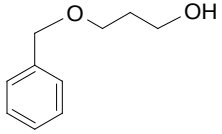
216



(*E*)-*p*-nitro-β-bromostyrene

$C_8H_6BrNO_2$

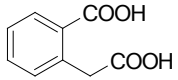
217



3-benzyloxy-1-propanol

$C_{10}H_{14}O_2$

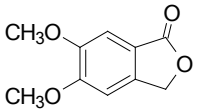
218



homophthallic acid

$C_9H_8O_4$

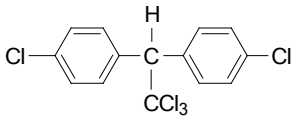
219



5,6-dimethoxy-2-coumaranone

$C_{10}H_{10}O_4$

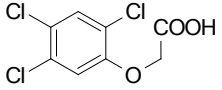
220



1,1-di-(*p*-chlorophenyl)-2,2,2-trichloroethane (DDT)

$C_{14}H_9Cl_5$

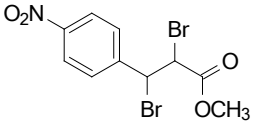
221



2,4,5-trichlorophenoxy-acetic acid (2,4,5-T)

$C_8H_5O_3Cl_3$

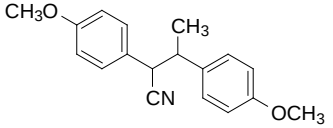
222



methyl 2,3-dibromo-3-(*p*-nitrophenyl)propionate

$C_{10}H_9NO_4Br_2$

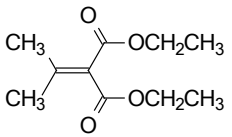
223



2,3-di-(*p*-anisyl)butyronitrile

$C_{18}H_{19}NO_2$

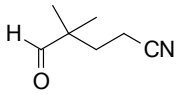
224



diethyl isopropylidene-malonate

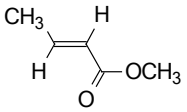
$C_{10}H_{16}O_4$

225



4-cyano-2,2-dimethylbutyraldehyde
$C_7H_{11}NO$

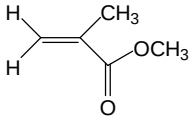
226



methyl (*E*)-3-methylacrylate

$C_5H_8O_2$

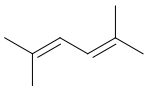
227



methyl crotonate

$C_5H_8O_2$

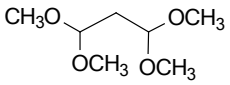
228



2,5-dimethyl-2,4-hexadiene

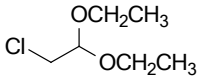
C_8H_{14}

229



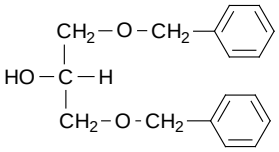
malonaldehyde bis(dimethyl acetal)
$C_7H_{16}O_4$

230



2-chloroacetaldehyde diethylacetal
$C_6H_{13}O_2Cl$

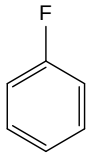
231



1,3-dibenzylglycerol

$C_{17}H_{20}O_3$

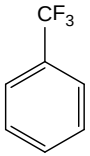
232



fluorobenzene

C_6H_5F

233



benzotrifluoride

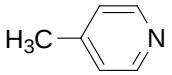
$C_7H_5F_3$

234

pyridine

C_5H_5N

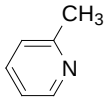
235



4-picoline

C_6H_7N

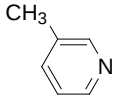
236



2-picoline

C_6H_7N

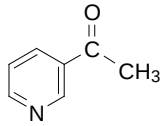
237



3-picoline

C_6H_7N

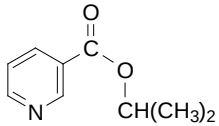
238



3-acetylpyridine

C_7H_7NO

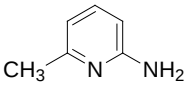
239



isopropyl nicotinate

$C_9H_{11}NO_2$

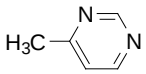
240



2-methyl-6-aminopyridine

$C_6H_8N_2$

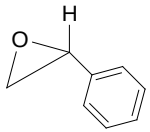
241



4-methylpyrimidine

$C_5H_6N_2$

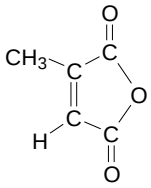
242



styrene epoxide

C_8H_8O

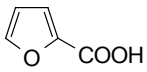
243



citraconic anhydride

$C_5H_4O_3$

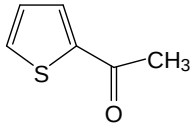
244



2-furoic acid

$C_5H_4O_3$

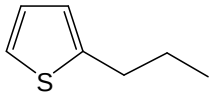
245



2-acetylthiophene

C_6H_6OS

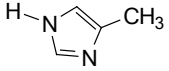
246



2-propylthiophene

$C_7H_{10}S$

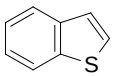
247



4-methylimidazole

$C_4H_6N_2$

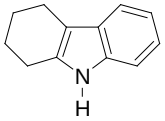
248



benzothiophene

C_8H_6S

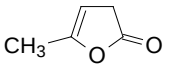
249



2,3,4,9-tetrahydrocarbazole

$C_{12}H_{13}N$

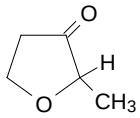
250



α-angelicalactone

$C_5H_6O_2$

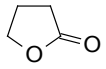
251



2-methyl-tetrahydrofuran-3-one

$C_5H_8O_2$

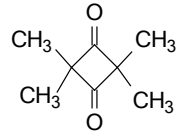
252



γ-butyrolactone

$C_4H_6O_2$

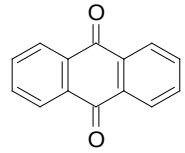
253



tetramethyl-1,3-cyclobutanedione

$C_8H_{12}O_2$

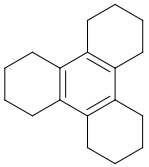
254



anthraquinone

$C_{14}H_8O_2$

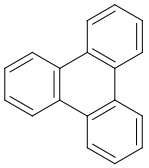
255



dodecahydrotriphenylene

$C_{18}H_{24}$

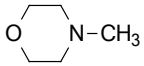
256



triphenylene

$C_{18}H_{12}$

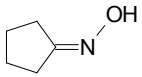
257



N-methylmorpholine

$C_5H_{11}NO$

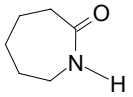
258



cyclopentanone oxime

C_5H_9NO

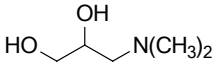
259



ε-caprolactam

$C_6H_{11}NO$

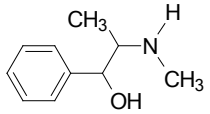
260



N,N-dimethyl-2,3-dihydroxy-1-propylamine

$C_5H_{13}NO_2$

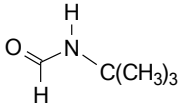
261



pseudoephedrine

$C_{10}H_{15}NO$

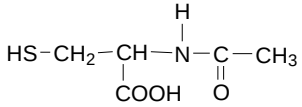
262



t-butylformamide

$C_5H_{11}NO$

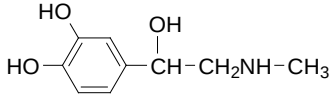
263



N-acetylcysteine

$C_5H_9NO_3S$

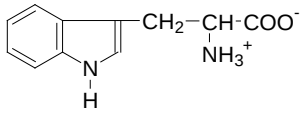
264



adrenalin

$C_9H_{13}NO_3$

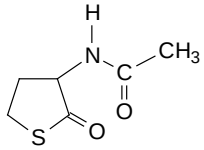
265



tryptophan

$C_{11}H_{12}N_2O_2$

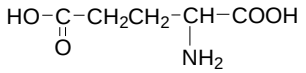
266



N-acetylhomocysteine thiolactone

$C_6H_9NO_2S$

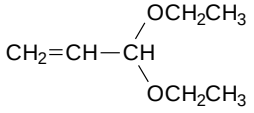
267



glutamic acid

$C_5H_9NO_4$

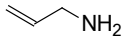
268



acrolein diethyl acetal

$C_7H_{14}O_2$

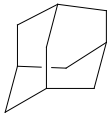
269



allylamine

C_3H_7N

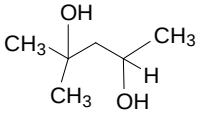
270



adamantane

$C_{10}H_{16}$

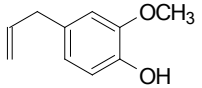
271



2-methyl-2,4-pentanediol

$C_6H_{14}O_2$

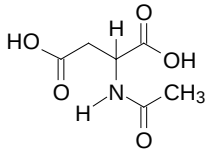
272



eugenol

$C_{10}H_{12}O_2$

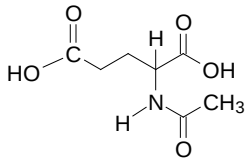
273



N-acetylaspartic acid

$C_6H_9NO_5$

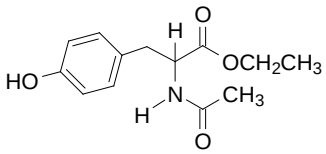
274



N-acetylglutamic acid

$C_7H_{11}NO_5$

275



N-acetyltyrosine ethyl ester

$C_{13}H_{17}NO_4$

276

2,5-dihydrofuran

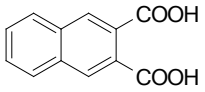
C_4H_6O

277

2,3-dihydrofuran

C_4H_6O

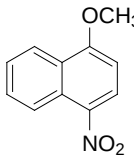
278



2,3-naphthalene-dicarboxylic acid

$C_{12}H_8O_4$

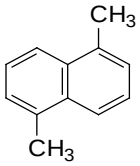
279



1-methoxy-4-nitro-naphthalene

$C_{11}H_9NO_3$

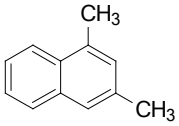
280



1,5-dimethylnaphthalene

$C_{12}H_{12}$

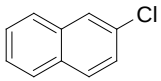
281



1,3-dimethylnaphthalene

$C_{12}H_{12}$

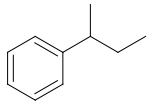
282



2-chloronaphthalene

$C_{10}H_7Cl$

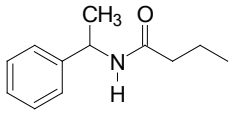
283



sec-butylbenzene

$C_{10}H_{14}$

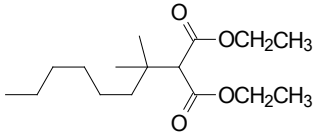
284



N-(1-methyl-1-phenylethyl)-butyramide

$C_{12}H_{17}NO$

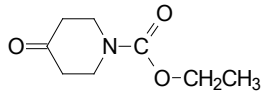
285



diethyl 2-(1,1-dimethylheptyl)malonate

$C_{16}H_{30}O_4$

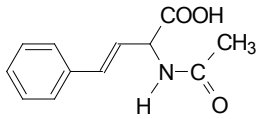
286



ethyl 4-piperidone-*N*-carboxylate

$C_8H_{13}NO_3$

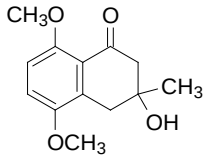
287



N-acetyl-2-amino-4-phenyl-(*E*)-but-2-enoic acid

$C_{12}H_{13}NO_3$

288



3-hydroxy-3-methyl-5,8-dimethoxy-1-coumarinone

$C_{13}H_{16}O_4$

Chapter 9.2 – The Analysis of Mixtures

Problem 289

Compound	Mole %
ethanol	57
bromoethane	43

Problem 290

Compound	Mole %
benzene	15
diethyl ether	46
dichloromethane	39

Problem 291

Compound	Mole %
benzene	24
ethyl acetate	59
dioxane	17

Problem 292

Compound	Mole %
ethanol	41
bromoethane	59

Problem 293

Compound	Mole %
benzene	13
diethyl ether	22
dichloromethane	65

Problem 294

Compound	Mole %
benzene	23
ethyl acetate	51
dioxane	26

Problem 295

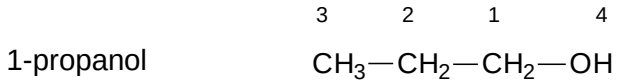
Compound	Mole %
fluorene	75
fluorenone	25

Problem 296

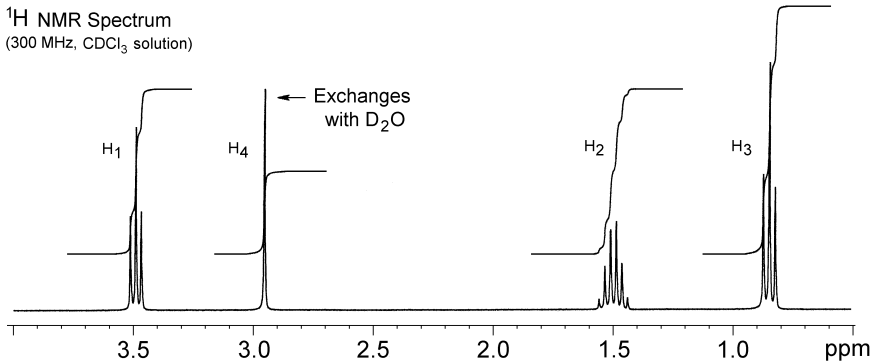
Compound	Mole %
4-nitroanisole	38
2-nitroanisole	62

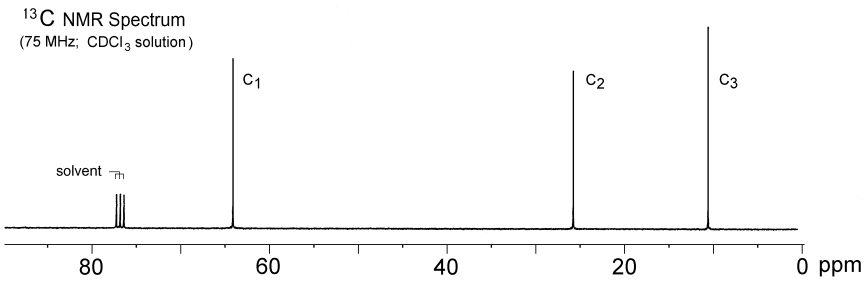
Chapter 9.3 – Problems in 2D NMR

Problem 297 1-propanol

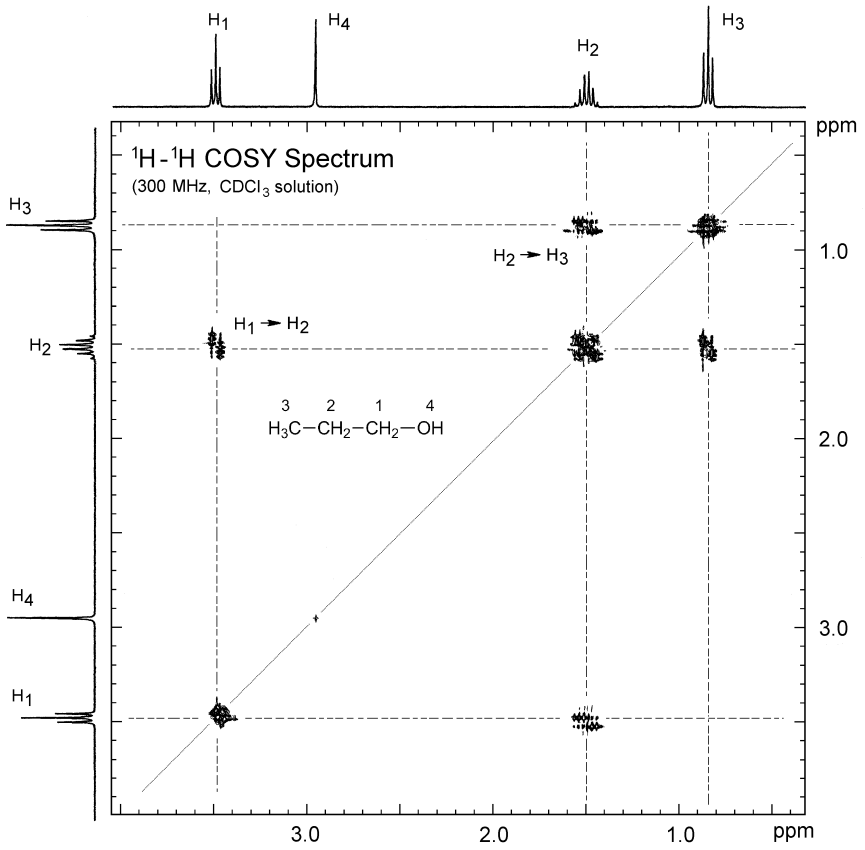


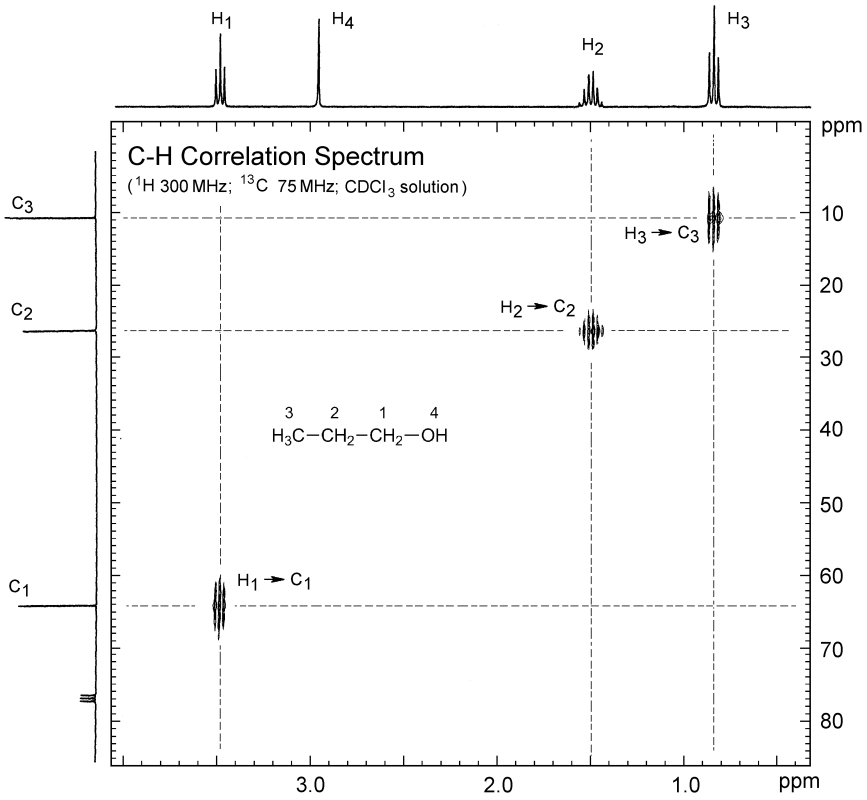
1. The exchangeable proton is the alcohol proton H4.
2. The low-field resonance in ^{1}H spectrum (at δ 3.49 ppm) is consistent with deshielding by electronegative oxygen, identifying this as H1, the -CH_2- group bound directly to the alcohol oxygen.
3. The COSY spectrum has a diagonal and the presence of the off-diagonal peaks indicates those pairs of protons which are coupled to each other.
4. Note also that the COSY spectrum is symmetrical so only one section (either above the diagonal or below the diagonal) needs to be analysed.
5. Having identified H1, the ^{1}H-^{1}H COSY allows step-wise assignment of the other protons: H1 (δ 3.49 ppm) correlates to H2 (δ 1.50 ppm); H2 correlates to H3 (δ 0.85 ppm).
6. Once the ^{1}H resonances have been assigned, the C-H correlation spectrum easily assigns the carbon spectrum. H1 (δ 3.49 ppm) correlates to C1 (δ 64.0 ppm) and so forth to identify C2 (δ 25.5 ppm) and C3 (δ 9.9 ppm).





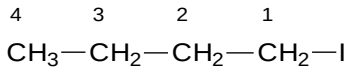
Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
H1	3.49	**C1**	64.1
H2	1.50	**C2**	26.3
H3	0.85	**C3**	10.6
H4	2.95		



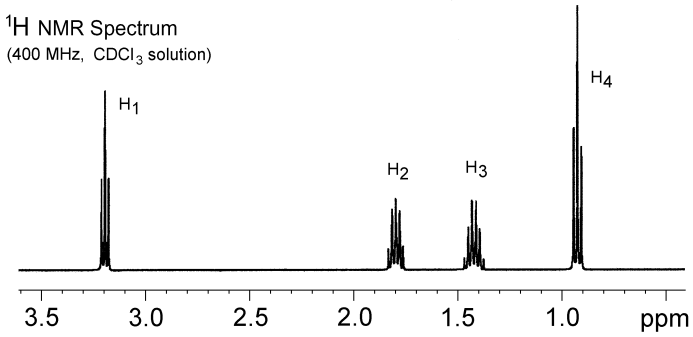


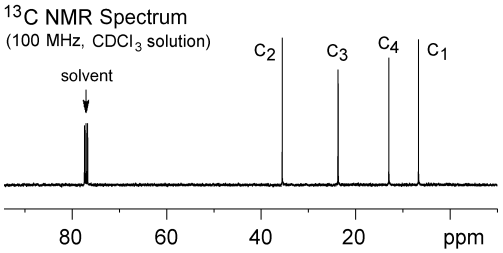
Problem 298 1-iodobutane

1-iodobutane

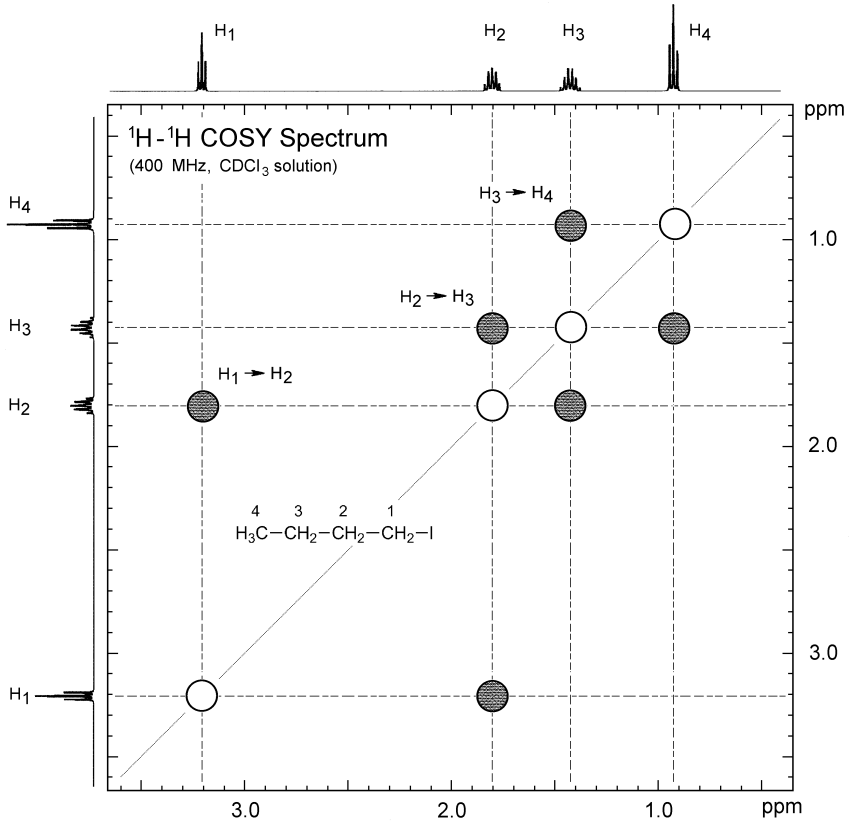


1. The ^{1}H and ^{13}C assignments are provided.

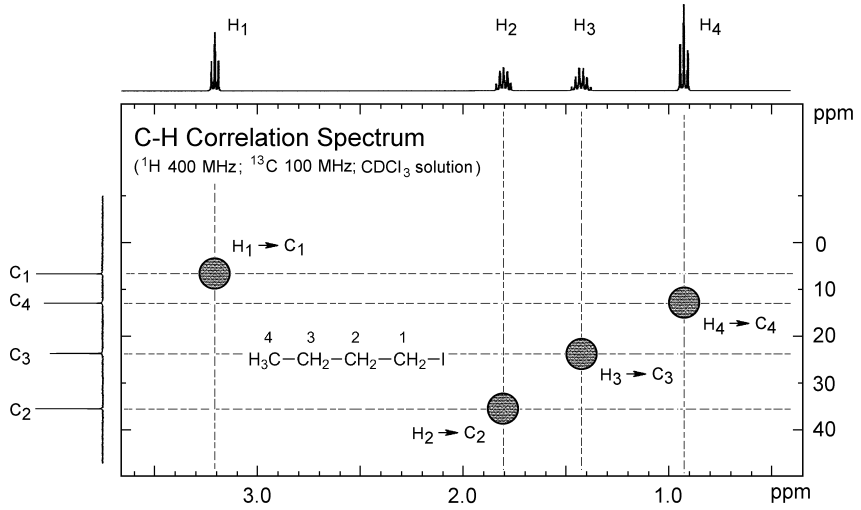




2. The COSY spectrum has a diagonal and the off-diagonal peaks indicate those pairs of protons which are coupled to each other.
3. Note also that the COSY spectrum is symmetrical so only one section (either above the diagonal or below the diagonal) needs to be analysed.
4. In the ^{1}H-^{1}H COSY spectrum, diagonal peaks will be observed at the frequency of each proton resonance.
5. 3 correlations would be expected:
   a. between H1 (δ 3.20 ppm) and H2 (δ 1.80 ppm);
   b. between H2 (δ 1.80 ppm) and H3 (δ 1.42 ppm);
   c. between H3 (δ 1.42 ppm) and H4 (δ 0.94 ppm).

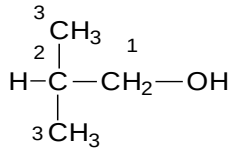


8. 4 correlations would be expected in the C-H correlation spectrum:
   a. between H1 (δ 3.20 ppm) and C1 (δ 6.7 ppm);
   b. between H2 (δ 1.80 ppm) and C2 (δ 35.5 ppm);
   c. between H3 (δ 1.42 ppm) and C3 (δ 23.6 ppm); and
   d. between H4 (δ 0.93 ppm) and C4 (δ 13.0 ppm).

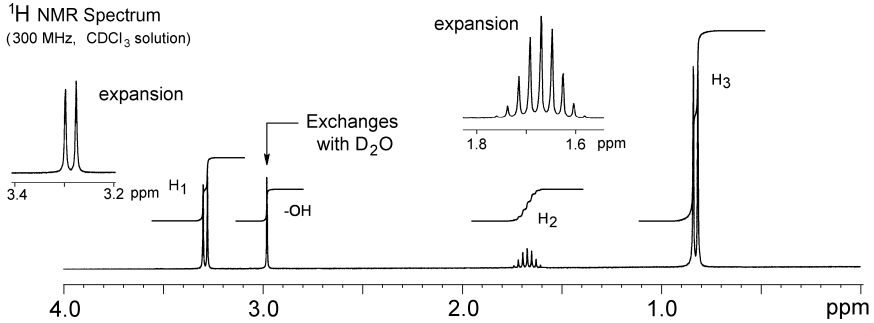


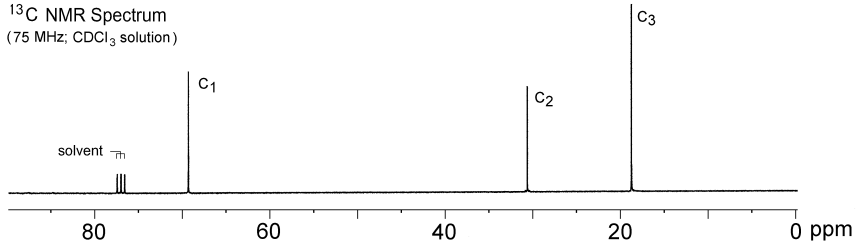
Problem 299 isobutanol

isobutanol

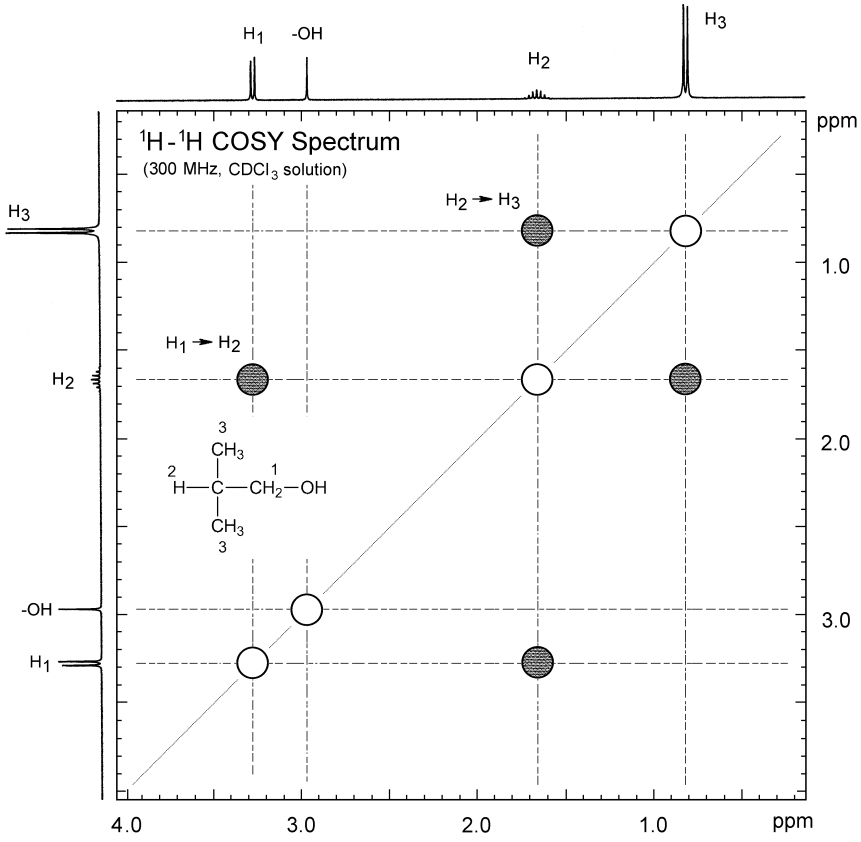


1. The ^{1}H and ^{13}C assignments are provided.

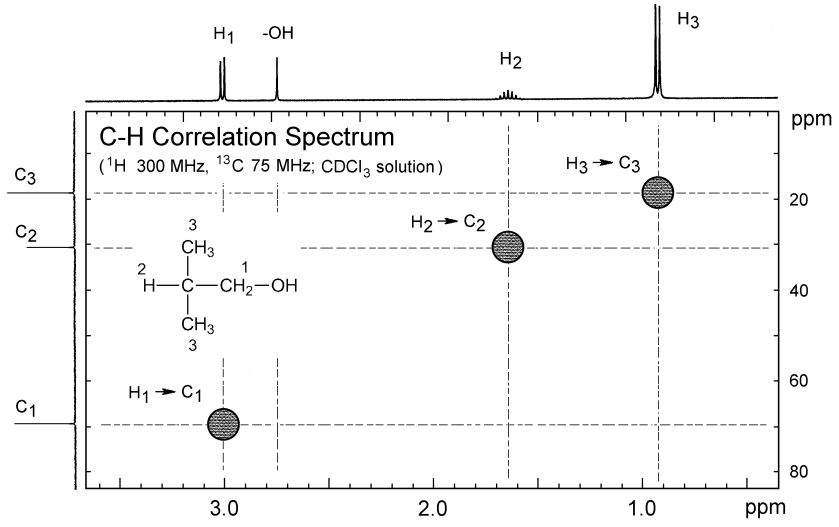




2. The COSY spectrum has a diagonal and the off-diagonal peaks indicate those pairs of protons which are coupled to each other.
3. Note also that the COSY spectrum is symmetrical so only one section (either above the diagonal or below the diagonal) needs to be analysed.
4. In the ^{1}H-^{1}H COSY spectrum, diagonal peaks will be observed at the frequency of each proton resonance, including the –OH resonance.
5. 2 correlations would be expected in the COSY spectrum:
    a. between: H1 (δ 3.28 ppm) and H2 (δ 1.68 ppm);
    b. between H2 (δ 1.68 ppm) and H3 (δ 0.83 ppm),

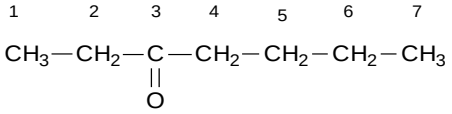


6. 3 correlations would be expected in the C-H correlation spectrum:
   a. between H1 (δ 3.28 ppm) and C1 (δ 69.3 ppm);
   b. between H2 (δ 1.68 ppm) and C2 (δ 30.7 ppm);
   c. between H3 (δ 0.83 ppm) and C3 (δ 18.7 ppm).
   d. The alcohol proton will not show any correlations in a C-H Correlation Spectrum since it has no directly bound C.

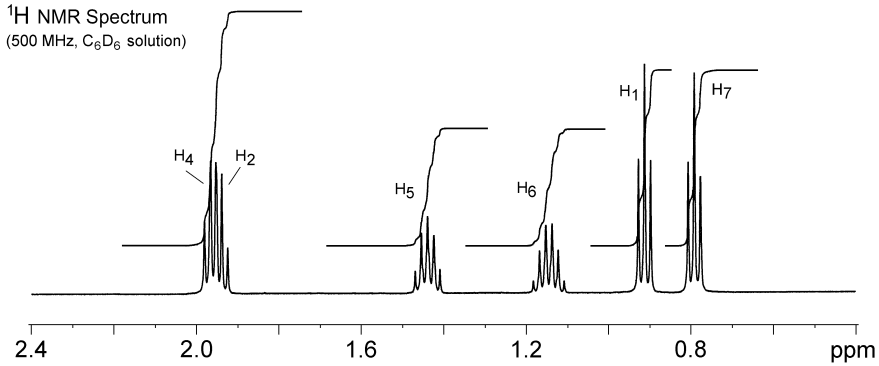


Problem 300

3-heptanone



Proton	Chemical Shift (δ) in ppm
H1	0.91
H2	1.94
H4	1.97
H5	1.44
H6	1.14
H7	0.79

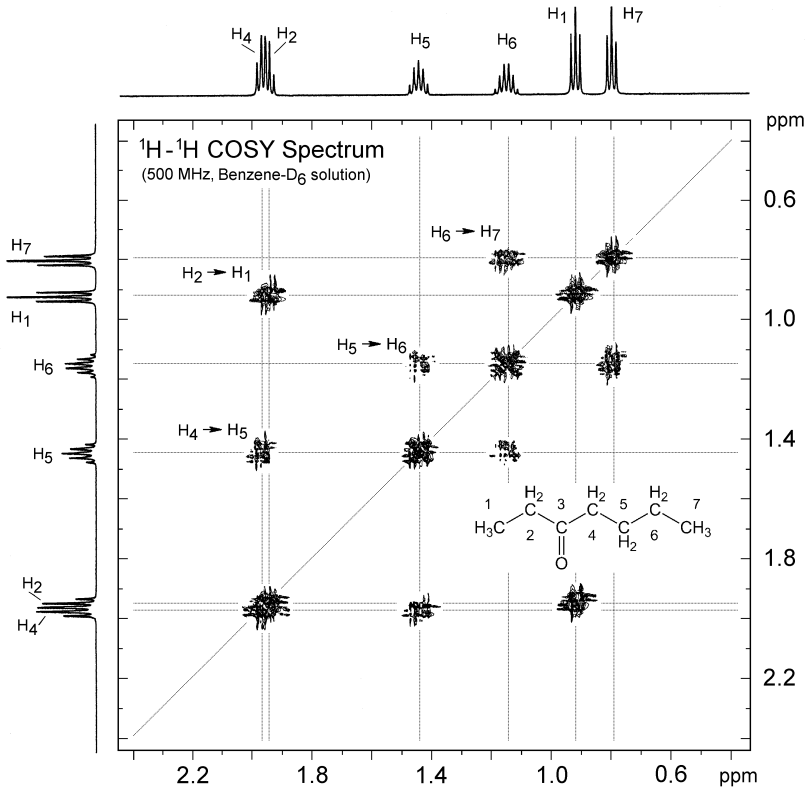


1. From the 1D spectrum there are clearly 2 CH_3 resonances (at δ 0.79 ppm and δ 1.14 ppm) and 4 CH_2 resonances.
2. There is overlap between the CH_2 resonances near δ 1.95 ppm.

3. In the COSY spectrum, it is easier to begin at the high-field end of the spectrum *i.e.* with the 2 CH_3 resonances.

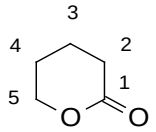
4. The -CH_3 resonance at δ 0.79 ppm correlates to the resonance at δ 1.14 ppm, which in turn correlates to the resonance at δ 1.44 ppm. Careful inspection shows that the resonance at δ 1.44 ppm also correlates to the resonance at δ 1.97 ppm. Clearly this is a spin system with 4 sets of different protons and this assigns H7 (δ 0.79 ppm); H6 (δ 1.14 ppm); H5 (δ 1.44 ppm) and H4 (δ 1.97 ppm).

5. The second -CH_3 resonance (at δ 0.91 ppm) correlates to the resonance at δ 1.94 ppm, therefore identifying H1 (δ 0.91 ppm) and H2 (δ 1.94 ppm).



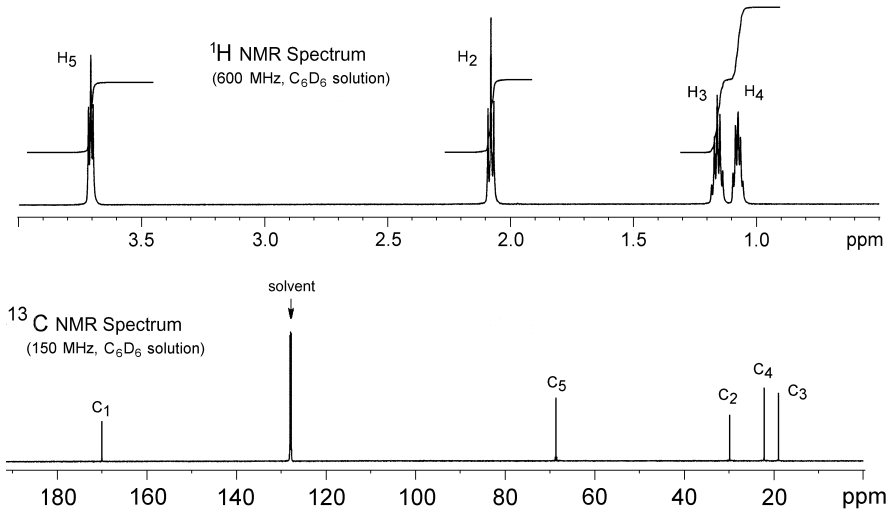
Problem 301

δ-valerolactone



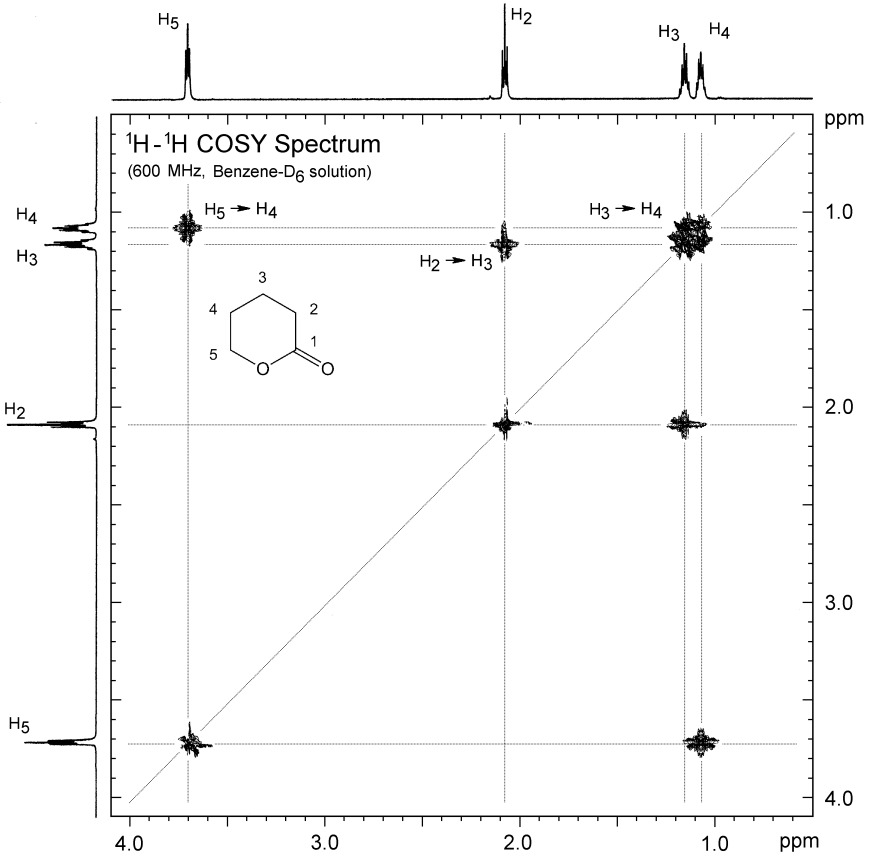
1. There are 4 multiplets in the ^{1}H NMR spectrum.

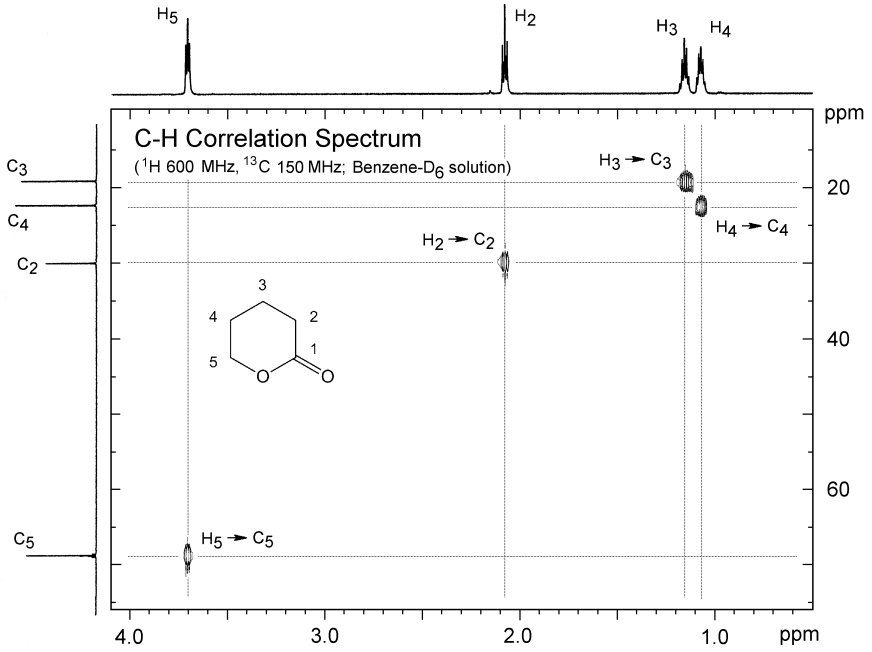
2. The low-field resonance in ^{1}H spectrum (at δ 3.71 ppm) is consistent with deshielding by electronegative oxygen, identifying this as H5.



3. Once H5 has been identified, the COSY spectrum is used to assign the other resonances sequentially. H5 correlates to H4 (δ 1.08 ppm); H4 correlates to H3 (δ 1.16 ppm); H3 correlates to H2 (δ 2.08 ppm).

4. Once the proton assignments are known, the C-H Correlation Spectrum easily identifies the protonated carbons. H5 correlates to C5 (δ 68.8 ppm); H4 correlates to C4 (δ 22.9 ppm); H3 correlates to C3 (δ 19.0 ppm); and H2 correlates to C2 (δ□29.9 ppm).
5. The carbonyl signal (no attached protons) is identified by its chemical shift (δ 170.0 ppm).



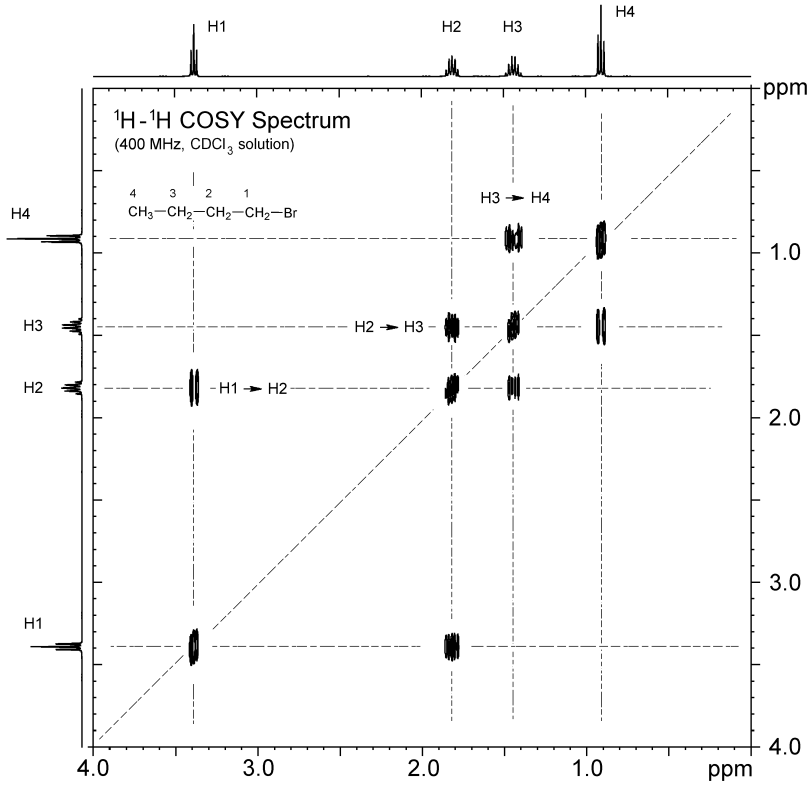


Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
		C1	170.0
H2	2.08	**C2**	29.9
H3	1.16	**C3**	19.0
H4	1.08	**C4**	22.2
H5	3.71	**C5**	68.8

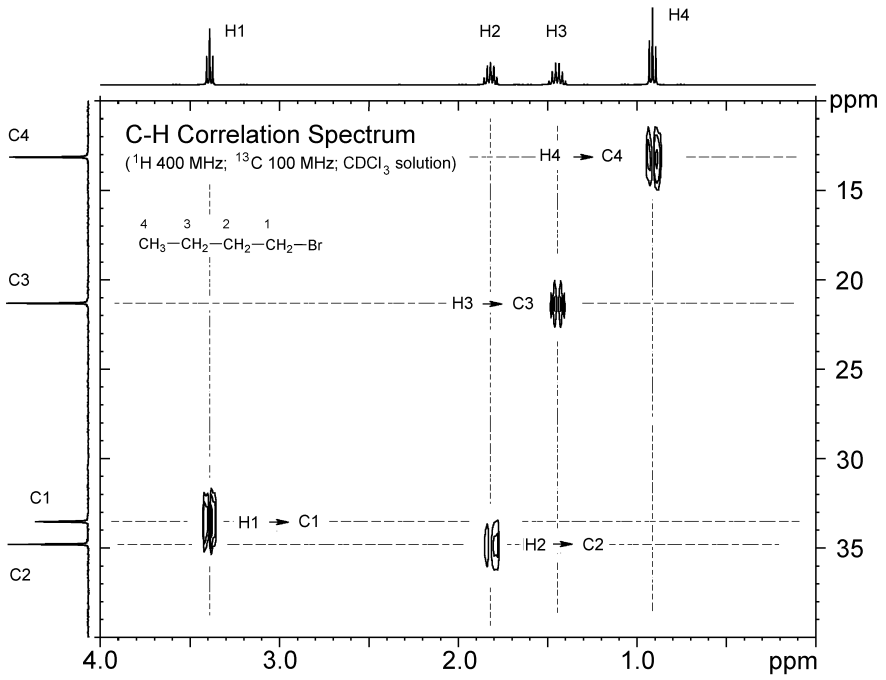
Problem 302

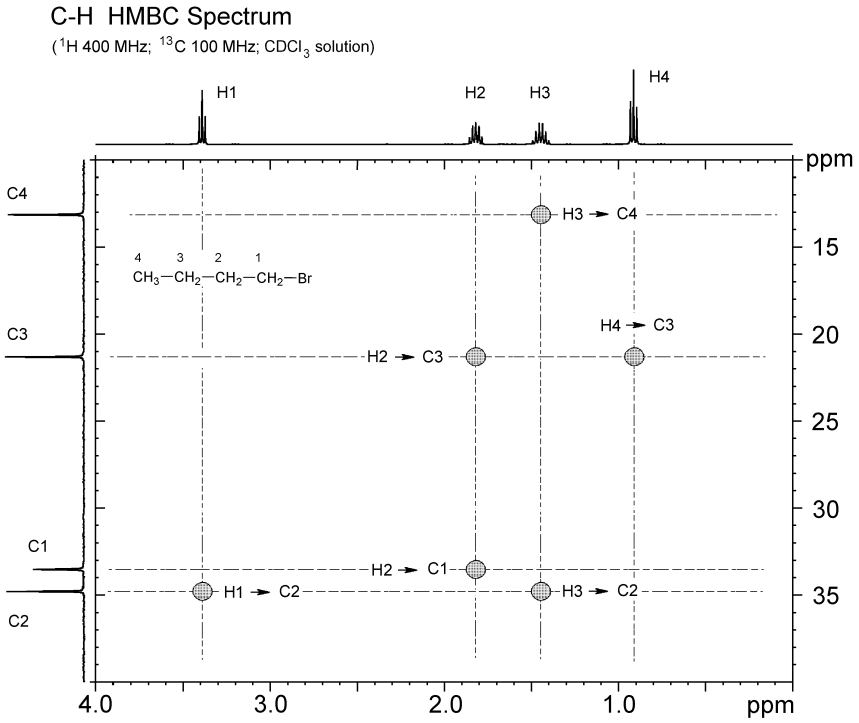
1-bromobutane $\overset{4}{CH_3}—\overset{3}{CH_2}—\overset{2}{CH_2}—\overset{1}{CH_2}—Br$

Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
H1	3.39	**C1**	33.4
H2	1.82	**C2**	34.7
H3	1.45	**C3**	21.4
H4	0.91	**C4**	13.2



1. The 1D spectra show 4 clear ^{13}C resonances and 4 ^{1}H resonances. There is no apparent overlap on any of the signals.
2. H1 and H4 are clearly the two triplets. H1 must be the 2-proton ^{1}H resonance at lowest field (δ 3.39) because of the Br substituent.
3. In the COSY spectrum, the resonance at δ 1.82 correlates to the resonance for H1 so the resonance at δ 1.82 must be H2. Likewise in stepwise fashion H3 and H4 can be assigned sequentially from the COSY spectrum.
4. Once the ^{1}H spectrum has been assigned, the ^{13}C resonances can be assigned directly from the C-H Correlation Spectrum.

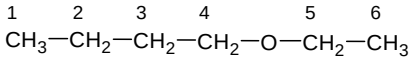




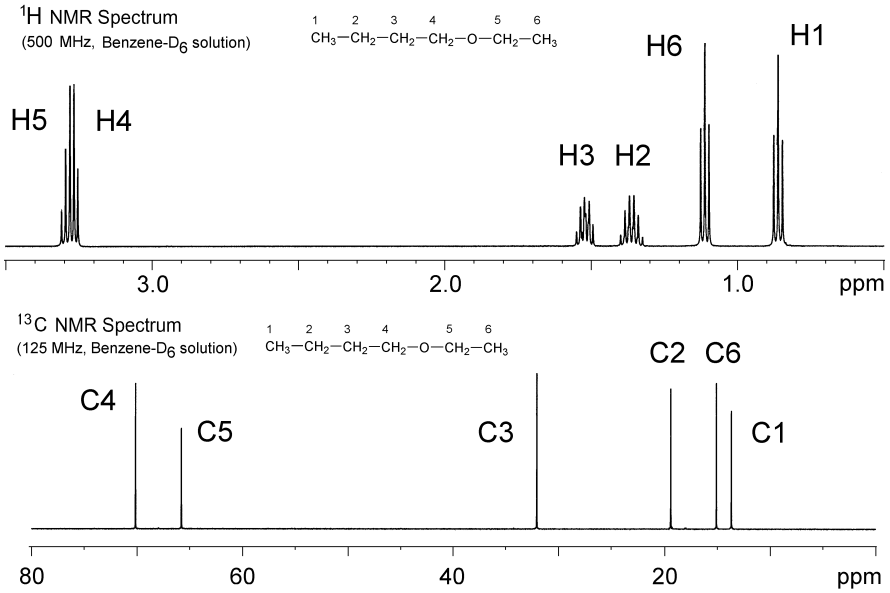
5. In the HMBC spectrum remember that in aliphatic systems, the interaction ($^2J_{C\text{-}H}$) is the strongest so aliphatic protons correlate strongly to the next carbon along an alkyl chain.

Problem 303

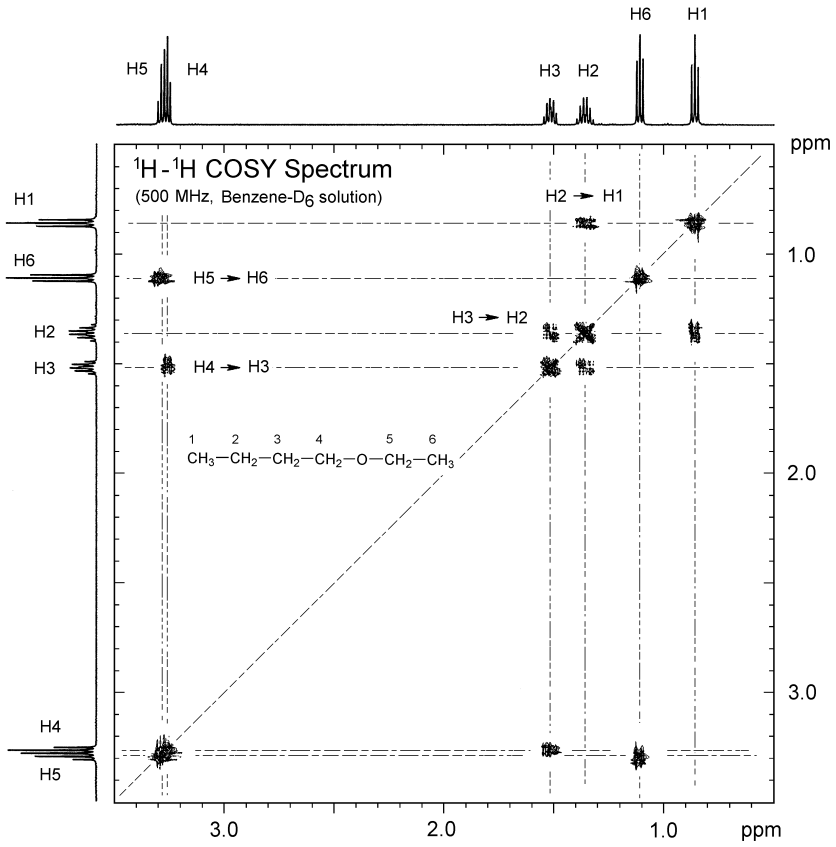
butyl ethyl ether

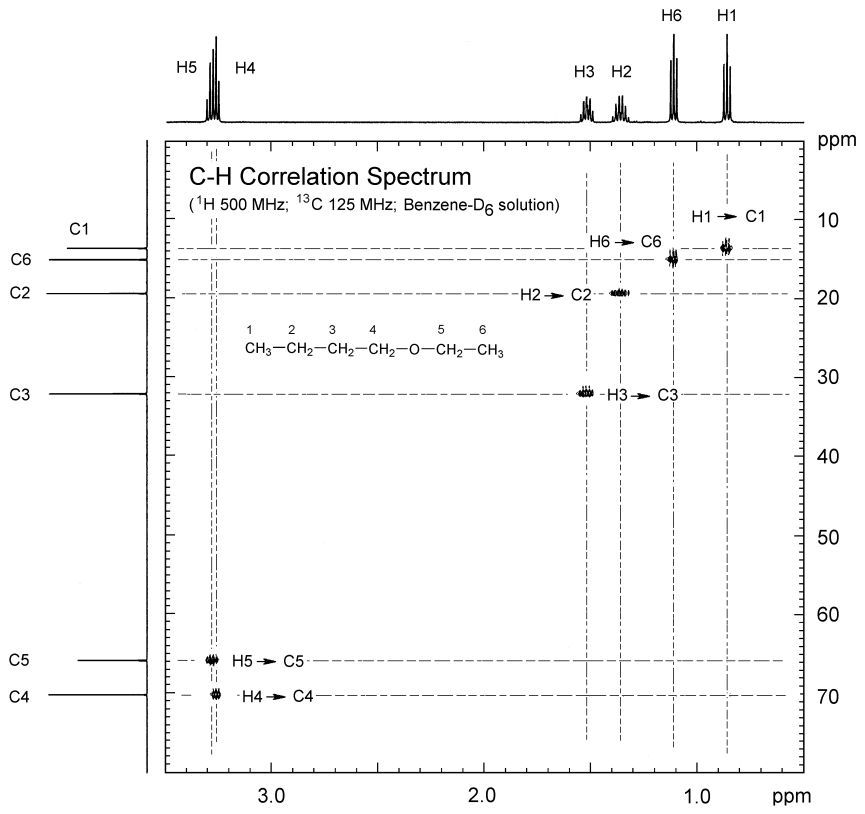


Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
H1	0.87	**C1**	13.5
H2	1.36	**C2**	19.4
H3	1.52	**C3**	32.1
H4	3.27	**C4**	70.1
H5	3.29	**C5**	66.0
H6	1.11	**C6**	15.0

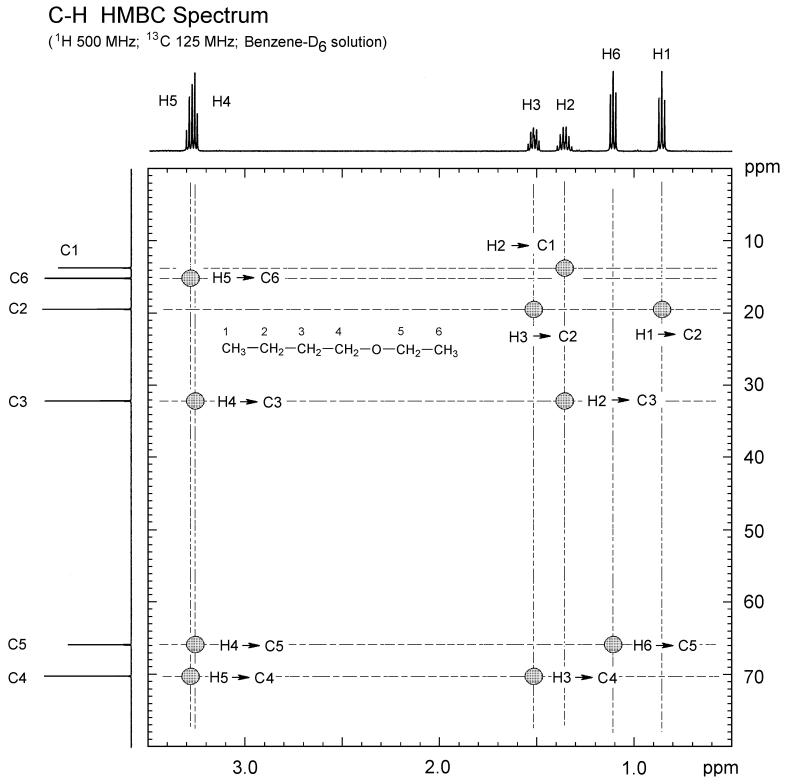


1. The 1D spectra show 6 clear ^{13}C resonances and 6 ^{1}H resonances. There is overlap between the two low-field resonances at δ 3.29 and δ 3.27 however the pattern of a quartet at δ 3.29 and a triplet (δ 3.27) can be seen from a close examination of the multiplet pattern.
2. H1 and H6 are clearly the two 3-proton triplets at high-field. H2 and H3 are the multiplets in the mid-range of the spectrum.
3. In the COSY spectrum, the resonance at δ 0.87 correlates to resonance at δ 1.36 which in turn correlates to the resonance at δ 1.52. The fact that there are at least 3 separate sets of coupled spins in this spin system identifies these as protons at 1, 2 & 3. The other protons are then identified sequentially.
4. Once the ^{1}H spectrum has been assigned, the ^{13}C resonances can be assigned directly from the C-H correlation spectrum.



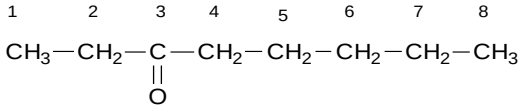


5. In the HMBC spectrum remember that: *(i)* in aliphatic systems the interaction ($^2J_{C\text{-}H}$) is the strongest so aliphatic protons correlate strongly to the next carbon along an alky chain; and *(ii)* in the HMBC spectrum, it is usual to see the correlation across a heteroatom ($^3J_{H\text{-}C\text{-}X\text{-}C}$) (where X is O, S or N) or $^4J_{H\text{-}C\text{-}(CO)\text{-}X\text{-}C}$ across a functional group like –COO- or –CONH-.



Problem 304

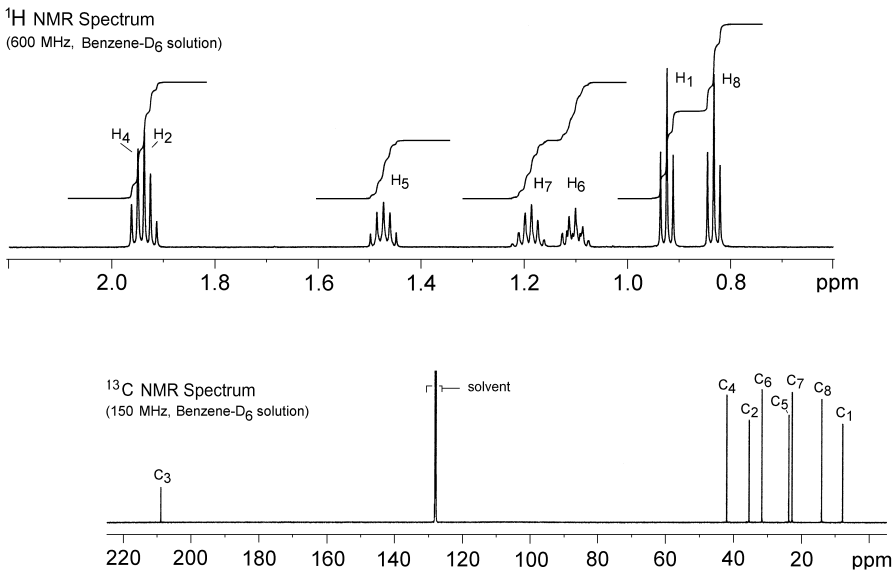
3-octanone



Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
H1	0.92	**C1**	7.8
H2	1.92	**C2**	35.4
		C3	209.0
H4	1.94	**C4**	42.1
H5	1.47	**C5**	23.7
H6	1.11	**C6**	31.7
H7	1.19	**C7**	22.7
H8	0.82	**C8**	14.0

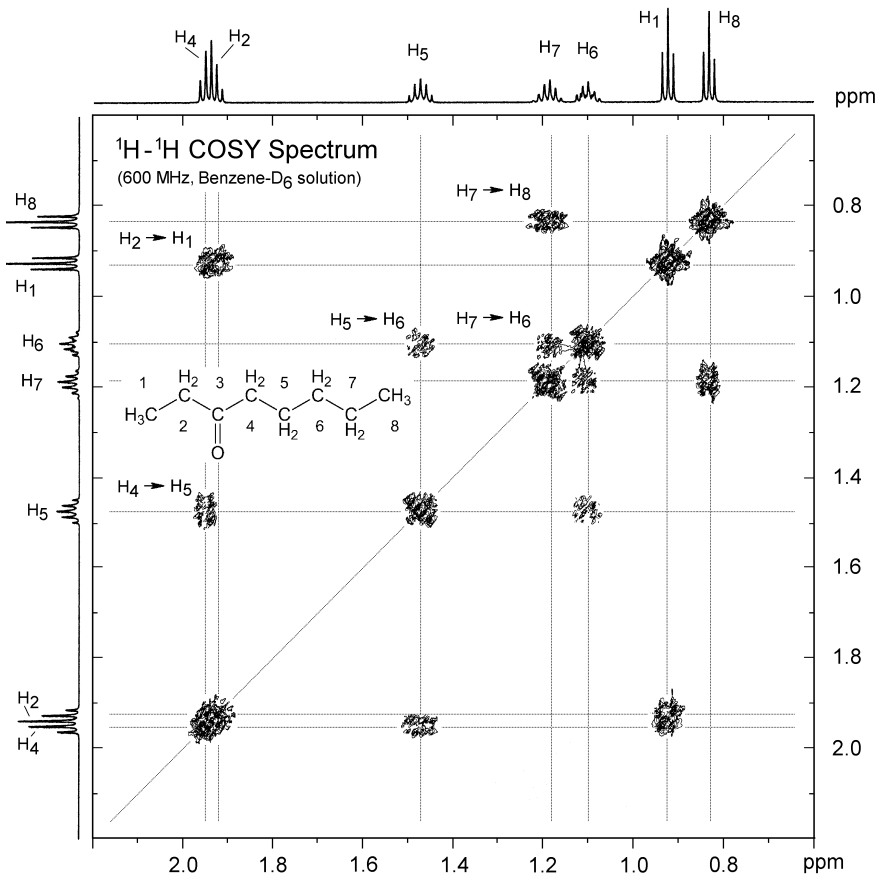
1. The 1D spectra show 8 ^{13}C resonances and 6 ^{1}H resonances. In the ^{1}H spectrum there are two 3-proton triplets corresponding to the 2 terminal $-CH_3$ groups. There are 5 x $-CH_2-$ groups and there is overlap between the 2 low-field resonances at δ 1.92 and δ 1.94.

2.



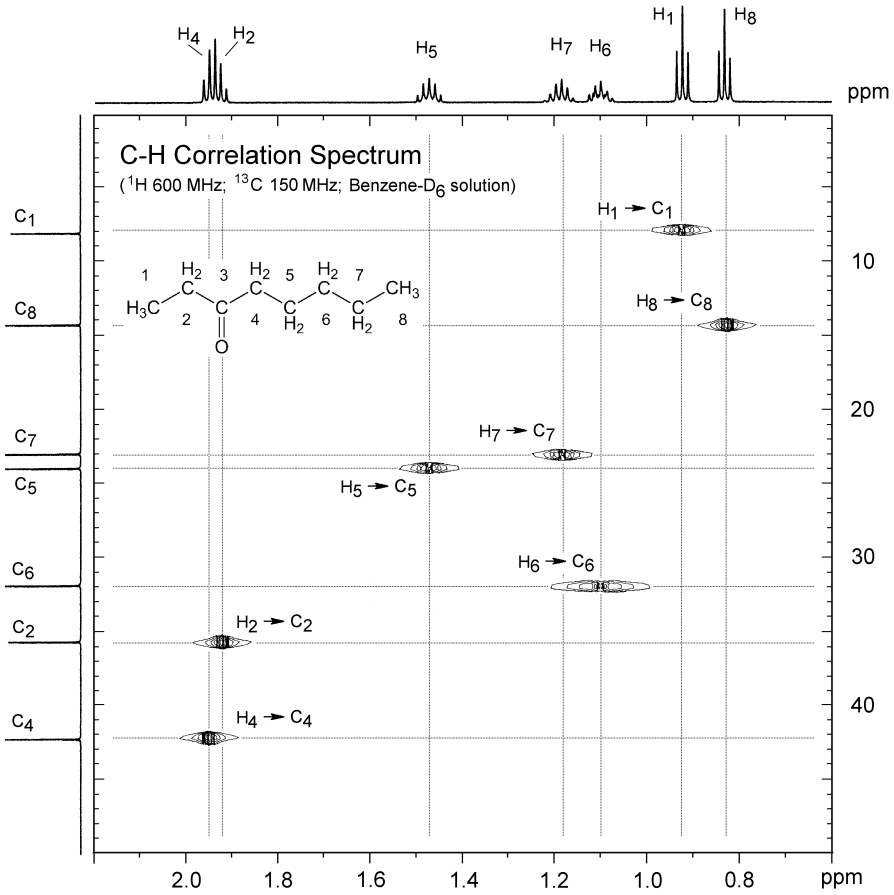
3. In analysing the COSY spectrum, it is easier to begin at the high-field end of the spectrum. The two high-field resonances are the -CH_3 groups.

4. In the COSY, the resonance at δ 0.82 ppm correlates to the resonance at δ 1.11 ppm, which in turn correlates to the resonance at δ 1.47 ppm, and then to the resonance δ 1.94 ppm. The fact that there are at least 5 separate sets of coupled spins in this spin system identifies these as protons at 4, 5, 6, 7 & 8. The assignment is H8 (δ 0.82 ppm); H7 (δ 1.19 ppm); H6 (δ 1.11 ppm); H5 (δ 1.47 ppm) and H4 (δ 1.94 ppm). H1 (δ 0.92 ppm) and H2 (δ 1.92 ppm) comprise the remaining spin system.



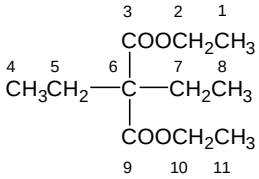
5. Once the proton spectrum is assigned, the protonated carbons are assigned from the C-H correlation spectrum. H1 correlates to C1 (δ 7.8 ppm); H2 correlates to C2 (δ 35.4 ppm); H4 correlates to C4 (δ 42.1 ppm); H5 correlates to C5 (δ 23.7 ppm); H6 correlates to C6 (δ 31.7 ppm); H7 correlates to C7 (δ 22.7 ppm); and H8 correlates to C8 (δ 14.0 ppm).

6. The carbonyl signal (no attached protons) is identified by its chemical shift (δ 209.0 ppm).



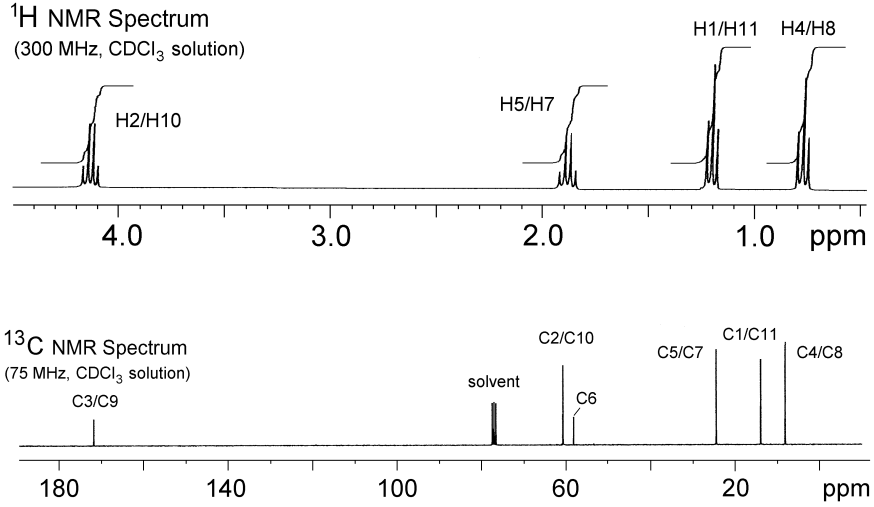
Problem 305

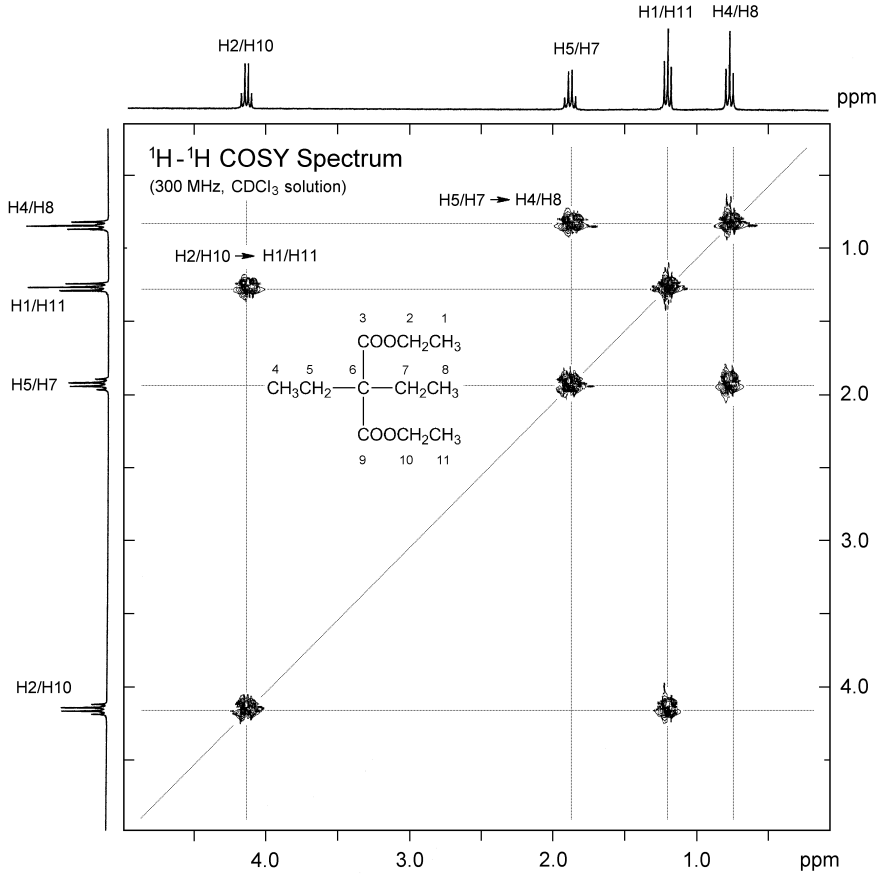
diethyl diethylmalonate



Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
H1	1.19	**C1**	14.0
H2	4.13	**C2**	60.8
		C3	171.9
H4	0.76	**C4**	8.1
H5	1.88	**C5**	24.5
		C6	58.0
H7	1.88	**C7**	24.5
H8	0.76	**C8**	8.1
		C9	171.9
H10	4.13	**C10**	60.8
H11	1.19	**C11**	14.0

1. Note that this molecule is highly symmetric, C1 is equivalent to C11; C2 is equivalent to C10; C3 is equivalent to C9; C4 is equivalent to C8; C5 is equivalent to C7. C6 is unique.
2. The proton spectrum contains two 3-proton triplets that must correspond to the $-CH_3$ groups and two 2-proton quartets that must correspond to the $-CH_2-$ groups.



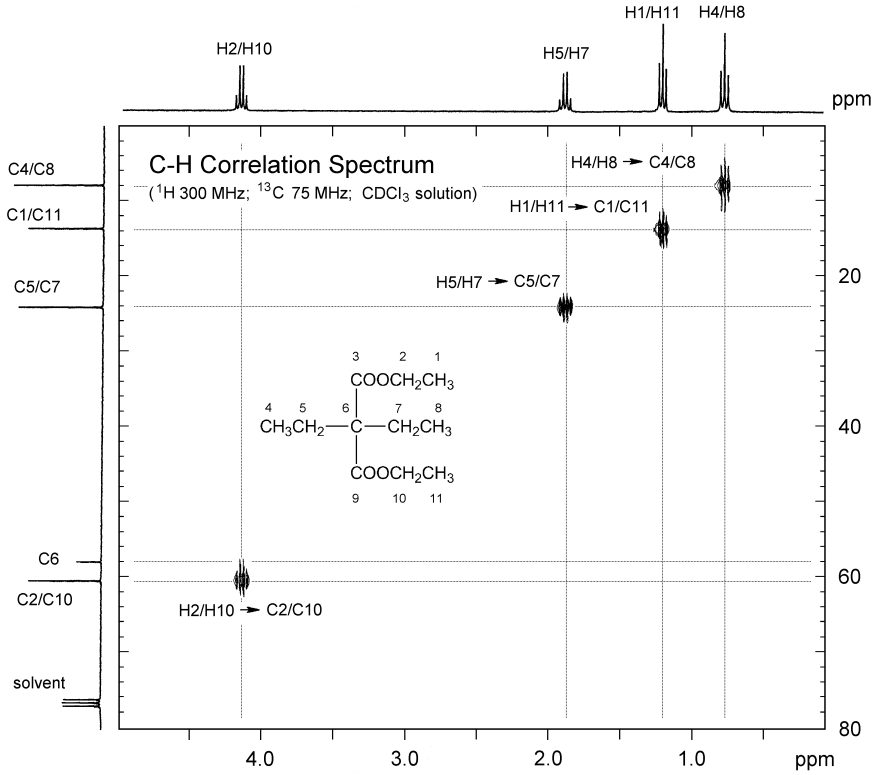


3. The low-field resonance in the ^{1}H spectrum (at δ 4.13 ppm) is consistent with deshielding by the electronegative –O-, so this must correspond to H2/H10.

4. In the COSY spectrum, H2/H10 correlates to the triplet signal at δ 1.19 ppm so this is clearly H1/H11. The other two assignments in the proton spectrum are then obvious – H4/H8 at δ 0.76 ppm which correlates to H5/H7 at δ 1.88 ppm.

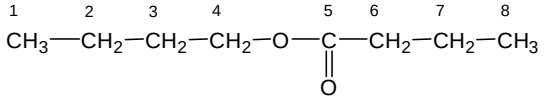
5. Once the proton spectrum is assigned, the protonated carbons are assigned from the C-H correlation spectrum. H1/H11 correlates to C1/C11 (δ 14.0 ppm); H2/H10 correlates to C2/C10 (δ 60.8 ppm); H4/H8 correlates to C4/C8 (δ 8.1 ppm); H5/H7 correlates to C5/C7 (δ 24.5 ppm).

6. The carbonyl signal (C3/C9 no attached protons) is identified by its chemical shift (δ 171.9 ppm). C6 has no attached protons, and is identified by an absence of a correlation in the C-H correlation spectrum (δ 58.0 ppm).



Problem 306 butyl butyrate

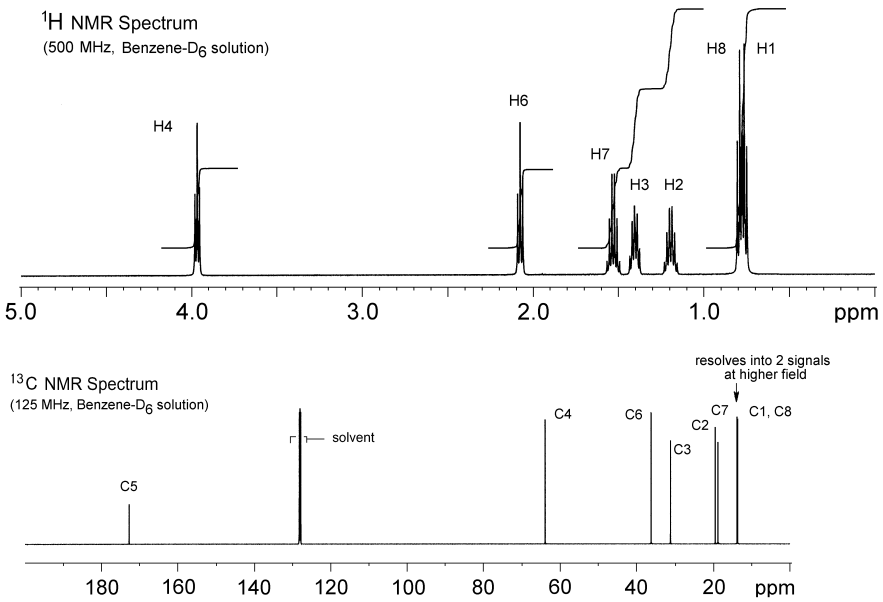
butyl butyrate



Proton	Chemical Shift (δ) in ppm	Carbon	Chemical Shift (δ) in ppm
H1	0.75	**C1**	13.9
H2	1.19	**C2**	19.5
H3	1.40	**C3**	31.2
H4	3.97	**C4**	64.0
		C5	172.8
H6	2.08	**C6**	36.2
H7	1.52	**C7**	19.0
H8	0.79	**C8**	13.9

1. The proton spectrum contains 7 multiplets with two 3-proton triplets overlapped at the high field end of the spectrum. 5 –CH_2- resonances are well dispersed with no overlap.

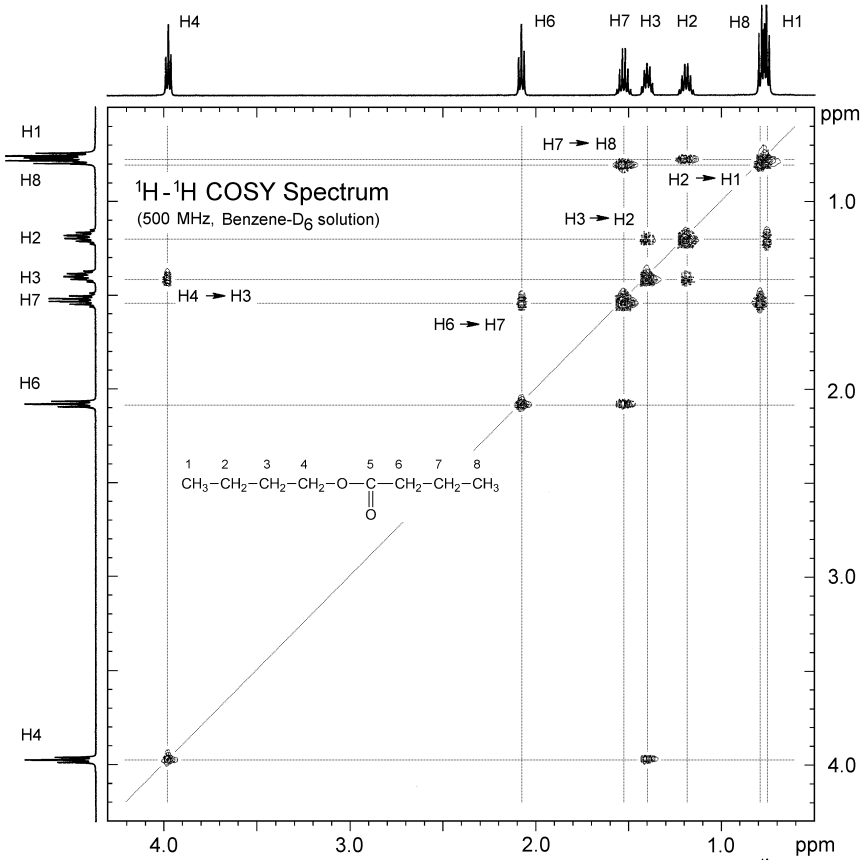
2. The low-field resonance (at δ 3.97 ppm) is consistent with deshielding by electronegative oxygen, identifying this resonance as H4.



3. Once you have identified one proton in the COSY spectrum, the other protons can be identified sequentially. H4 correlates to H3 (δ 1.40 ppm); H3 correlates to H2 (δ 1.19 ppm); H2 correlates to H1 (δ 0.75 ppm).

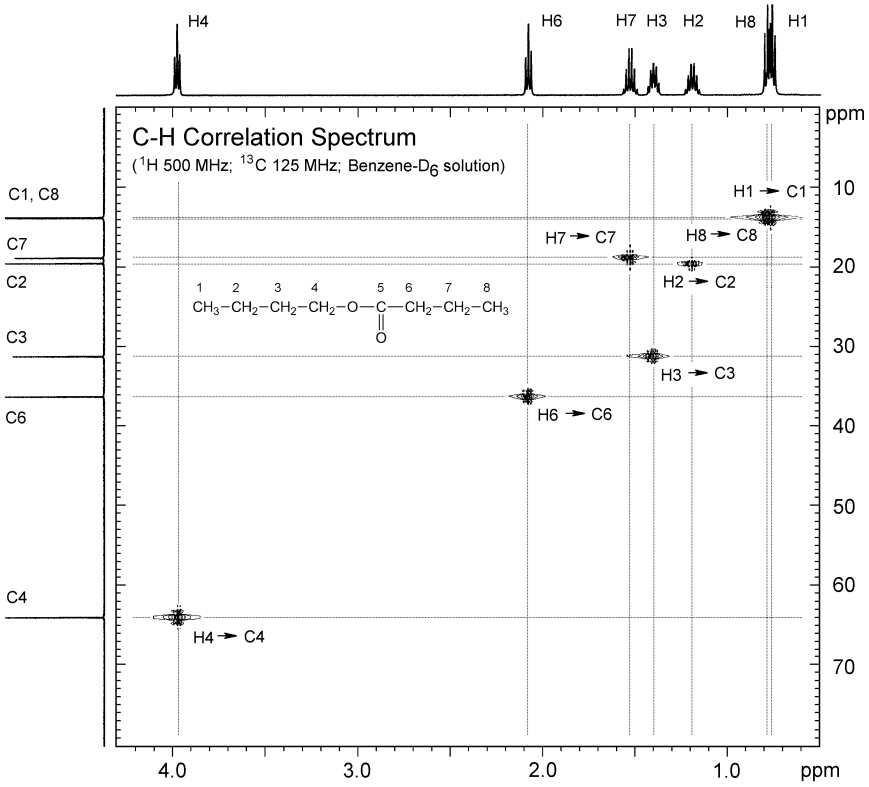
4. This leaves three unidentified resonances at δ 2.08, 1.52 and 0.79 ppm.

5. The chemical shift and multiplicity of the signal at δ 2.08 ppm assign it as H6 and the other protons in the spin system can be assigned sequentially. H6 correlates to H7 (δ 1.52 ppm) and H7 correlates to H8 (δ 0.79 ppm).



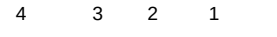
6. Once the proton spectrum is assigned, the protonated carbons are assigned from the C-H correlation spectrum. H1 correlates to C1 (δ 13.9 ppm); H2 correlates to C2 (δ 19.5 ppm); H3 correlates to C3 (δ 31.2 ppm); H4 correlates to C4 (δ 64.0 ppm); H6 correlates to C6 (δ 36.2 ppm); H7 correlates to C7 (δ 19.0 ppm); H8 correlates to C8 (δ 13.9 ppm).

7. The carbonyl signal C5 (no attached protons) is assigned by its chemical shift (δ 172.8 ppm).

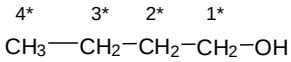


Problem 307

1-iodobutane

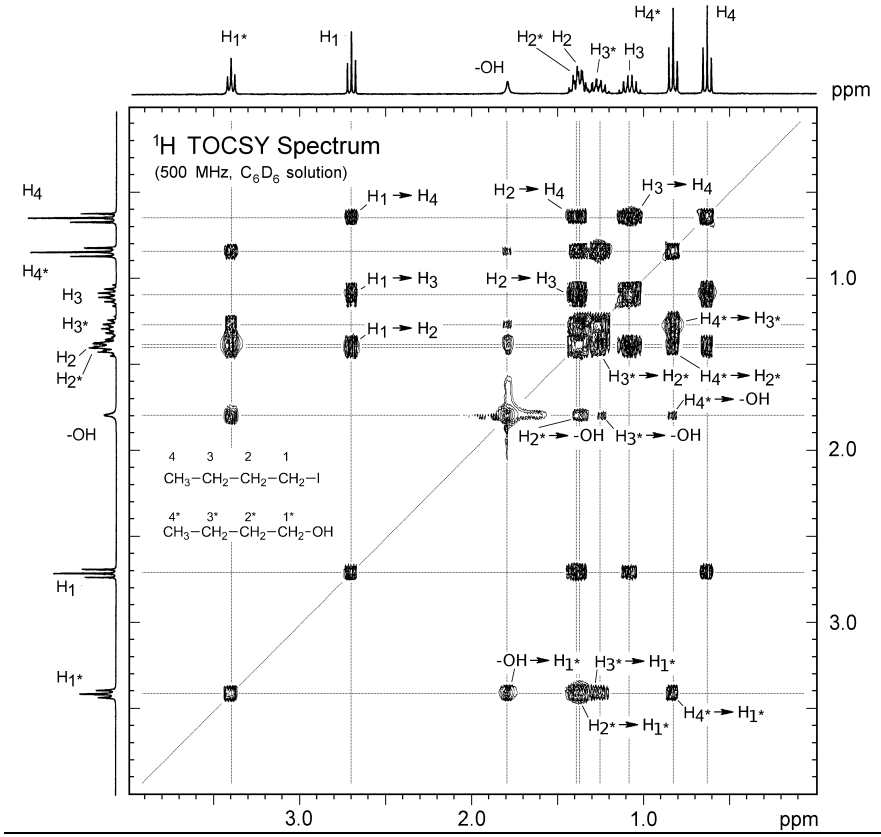


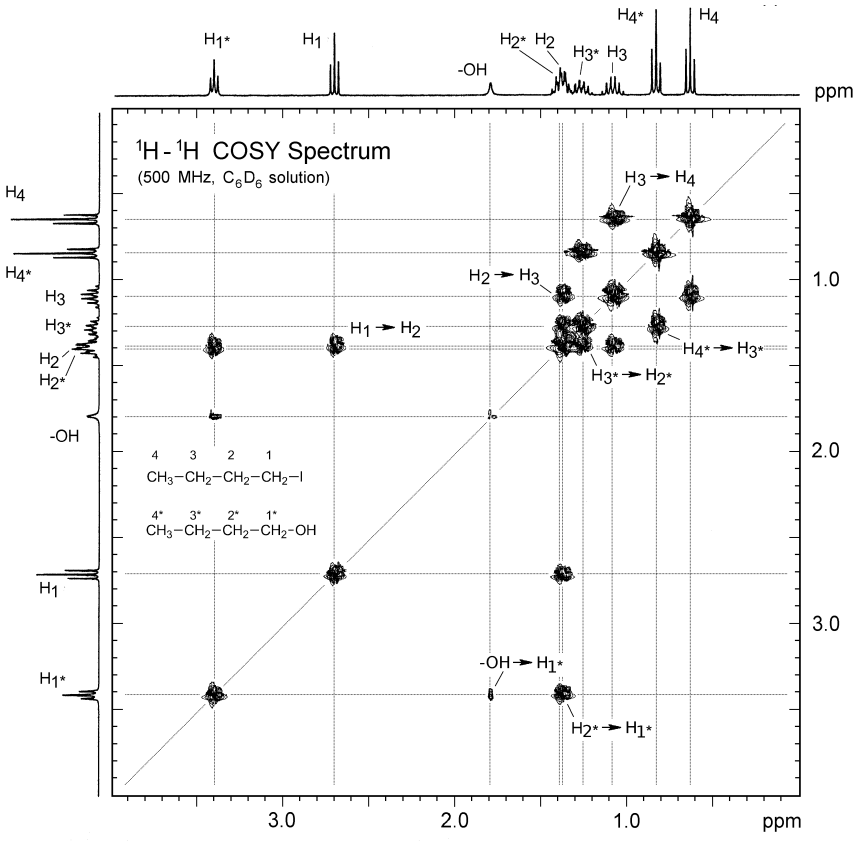
1-butanol



1. The low-field resonance in the ^{1}H NMR spectrum (at δ 3.4 ppm) is consistent with deshielding by electronegative oxygen, identifying this as being H1* of 1-butanol.
2. The TOCSY spectrum allows identification of all of the resonances in a coupled spin system – H1* correlates to cross-peaks at δ 1.8, δ 1.4, δ 1.3 and δ 0.85 ppm so these are all the resonances of the butanol spin system.
3. Likewise, the TOCSY spectrum allows correlation of the resonance at δ 2.7 ppm (H1) to peaks at δ 1.4, δ 1.1 and δ 0.65 ppm, identifying these as the resonances due to 1-iodobutane.
4. The ^{1}H-^{1}H COSY spectrum allows stepwise identification of the 1-butanol resonances. H1* (δ 3.4 ppm) correlates to -OH (δ 1.8 ppm) and to H2* (δ 1.4 ppm); H2* correlates to H3* (δ 1.3 ppm); and H3* correlates to H4* (δ 0.85 ppm).
5. The resonances for 1-iodobutane may also be identified step-wise from the COSY spectrum: H1 (δ 2.7 ppm) correlates to H2 (δ 1.4 ppm); H2 correlates to H3 (δ 1.1 ppm); and H3 correlates to H4 (δ 0.65 ppm).

1-iodobutane	^{1}H Chemical Shift (δ) in ppm	1-butanol	^{1}H Chemical Shift (δ) in ppm
H1	2.70	**H1***	3.41
H2	1.40	**H2***	1.39
H3	1.08	**H3***	1.27
H4	0.64	**H4***	0.84
		-OH	1.95

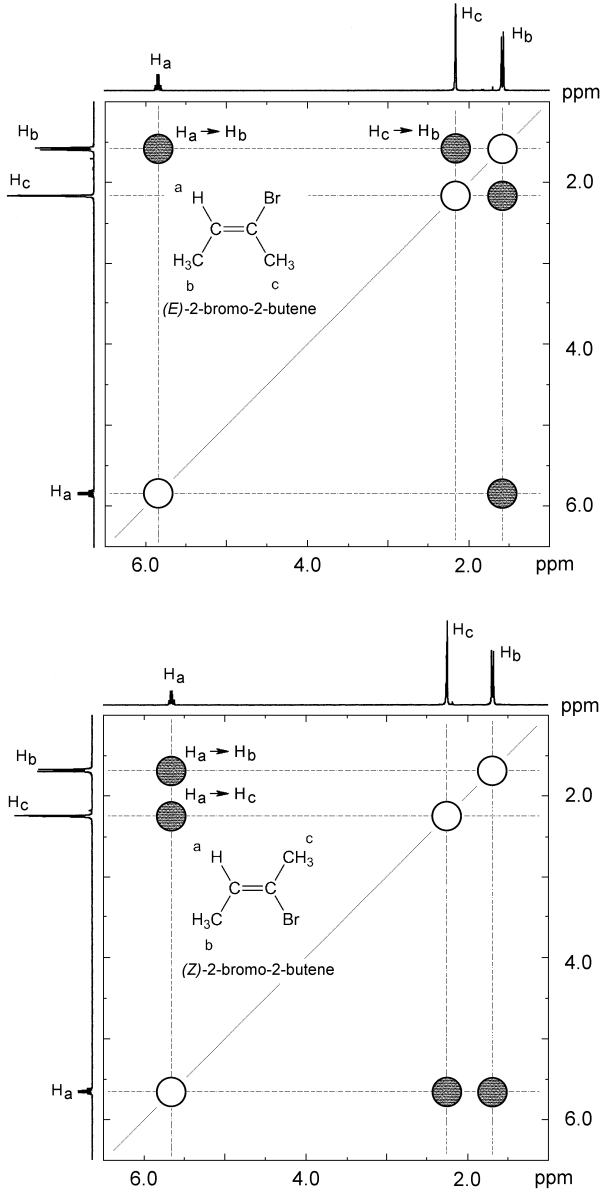




Problem 308

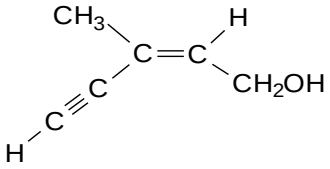
(*E*)- and (*Z*)-2-butene

1. Each of the spin systems contains 3 different resonances (one C-H and two $-CH_3$ signals).
2. The NOESY spectrum has peaks on the diagonal at the frequencies of each of the resonances in the spectrum so each spectrum will contain three diagonal peaks.
3. NOESY spectra show cross-peaks (off-diagonal peaks) at positions where a proton whose resonance appears on the F2 axis is close in space to another whose resonance appears on the F1 axis.
4. For (*E*)-2-bromo-2-butene, the two $-CH_3$ groups (Hb and Hc) are *cis* to each other and hence close in space. The H of the C-H group (Ha) is geminal to one methyl group (Hb) (and hence the protons are close in space). Two cross-peaks would be expected, one between Ha and Hb, and one between Hb and Hc.
5. Note also that the NOESY spectrum is symmetrical, so only one section (either above the diagonal or below the diagonal) needs to be analysed.
6. For (*Z*)-2-bromo-2-butene, one $-CH_3$ group (Hc) is *cis* to the H of the C-H group (Ha) and hence close in space. The other $-CH_3$ group (Hb) is geminal to the H of the C-H group (Ha) and so these two groups are close in space. Two cross-peaks would be expected, one between Ha and Hb, and one between Ha and Hc.

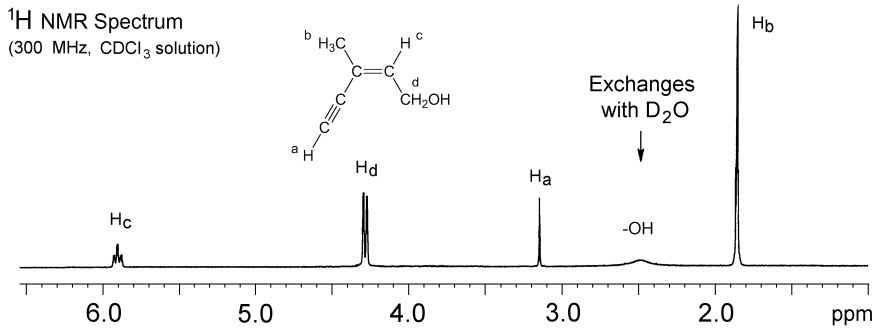


Problem 309

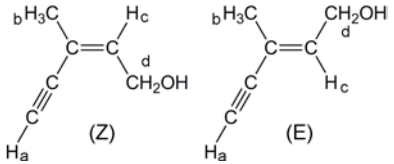
(*Z*)-3-methyl-2-penten-4-ynol



1. The 5 resonances in the ^{1}H NMR spectrum can be assigned by inspection.

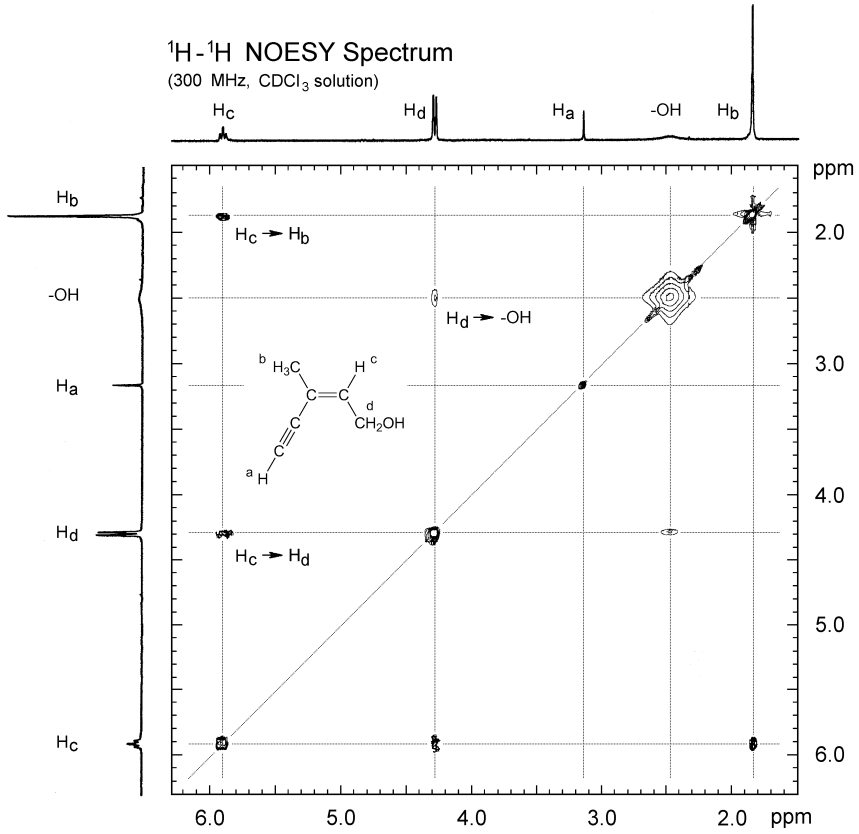


2. There are two possible isomers of 3-methylpent-2-en-4-yn-1-ol:

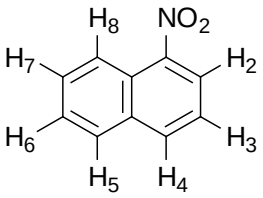


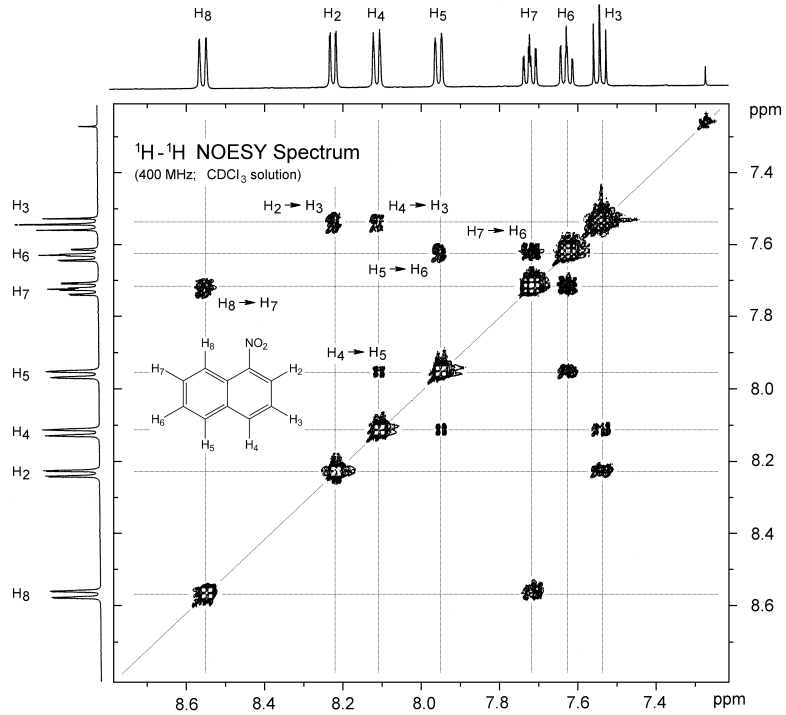
3. There is a clear correlation between Hc and Hb in the NOESY spectrum and this places the H of the =C-H group (Hc) and the methyl group (Hb) close together in space and on the same side of the double bond.

4. The compound must be the *Z*-isomer, *(Z)*-3-methyl-2-penten-4-ynol



Problem 310

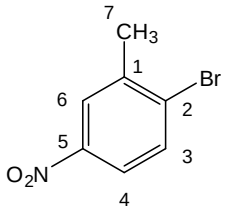
1-nitronaphthalene	
Proton	**Chemical Shift (δ) in ppm**
H2	8.22
H3	7.53
H4	8.10
H5	7.95
H6	7.62
H7	7.71
H8	**8.56**



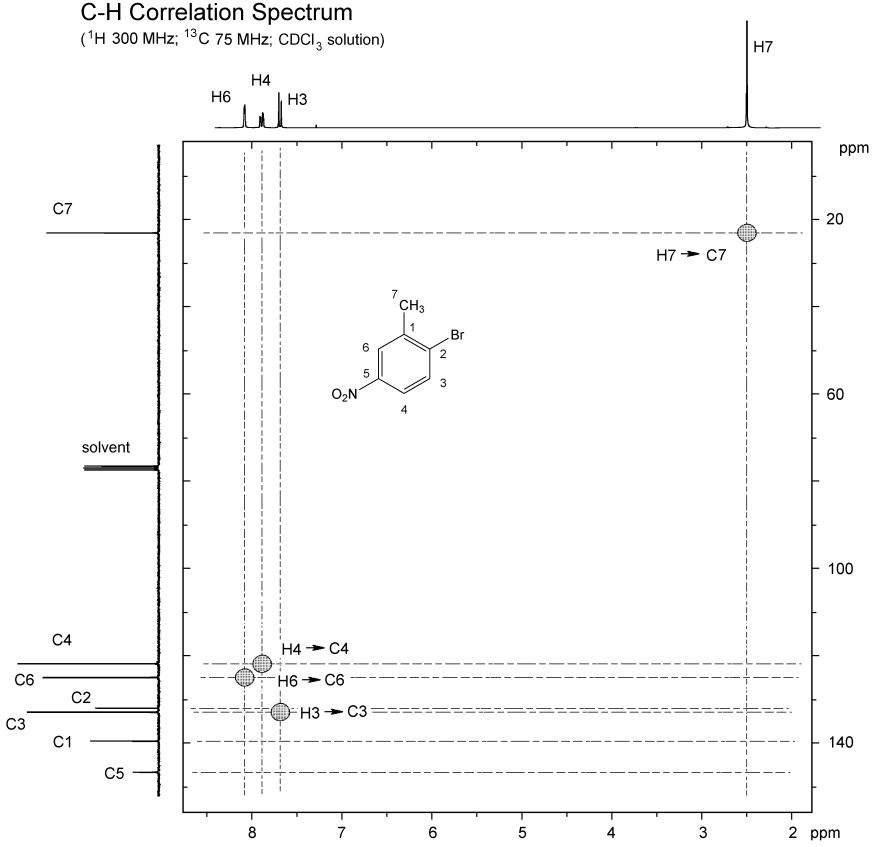
1. Given the chemical shift of H8 (δ 8.56 ppm), the remaining protons can be identified sequentially. H8 correlates to H7; H7 correlates to H6 and so forth until all of the protons in the spin system have been identified.

Problem 311

2-bromo-5-nitrotoluene

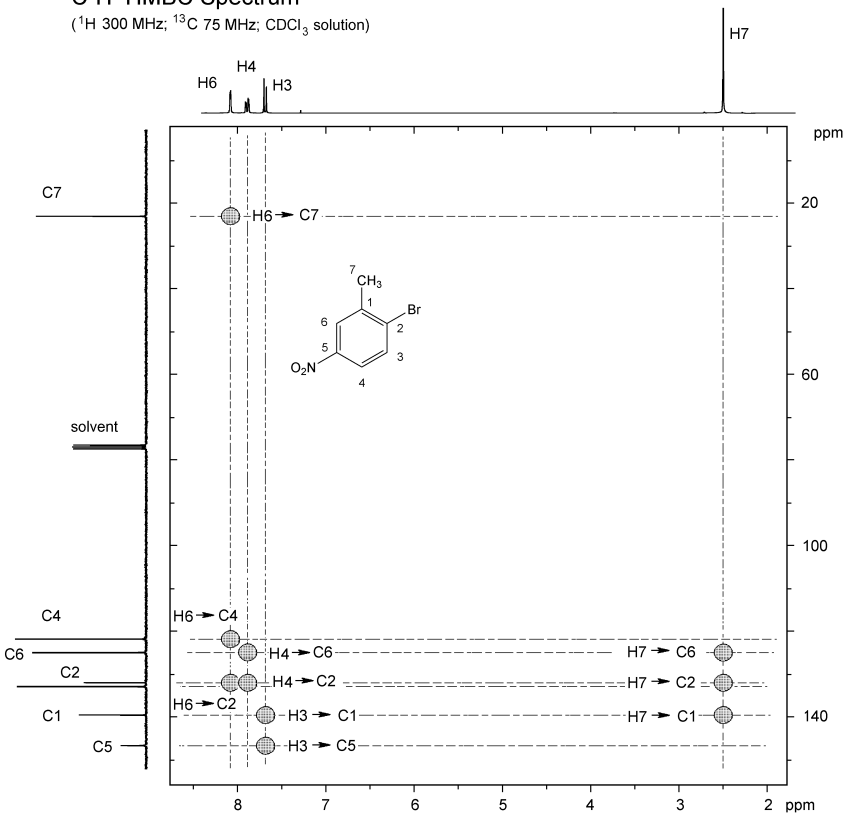


C-H Correlation Spectrum
(^{1}H 300 MHz; ^{13}C 75 MHz; $CDCl_3$ solution)



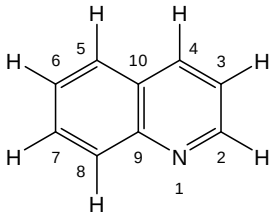
1. In the HMBC spectrum remember that:
   i. the benzylic protons will correlate strongly to the *ipso* carbon and to the *ortho* protons on the aromatic ring;
   ii. conversely the protons *ortho* to the $-CH_3$ group will correlate to the benzylic carbon; and
   iii. in aromatic spin systems the 1-3 interaction ($^3J_{C-H}$) is the strongest so aromatic protons correlate most strongly to the carbons which are *meta* to them.

C-H HMBC Spectrum
(^{1}H 300 MHz; ^{13}C 75 MHz; $CDCl_3$ solution)

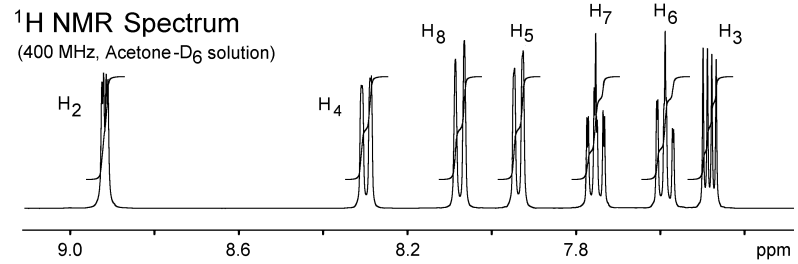


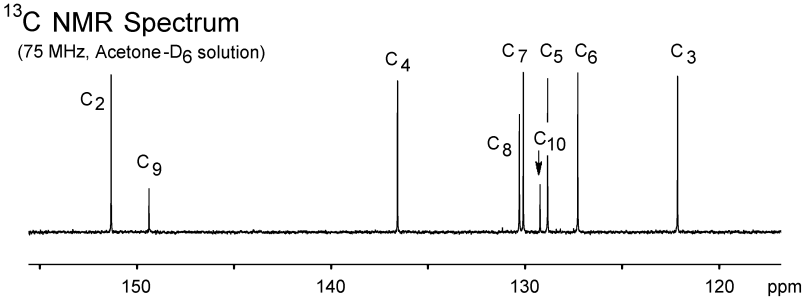
Problem 312

quinoline

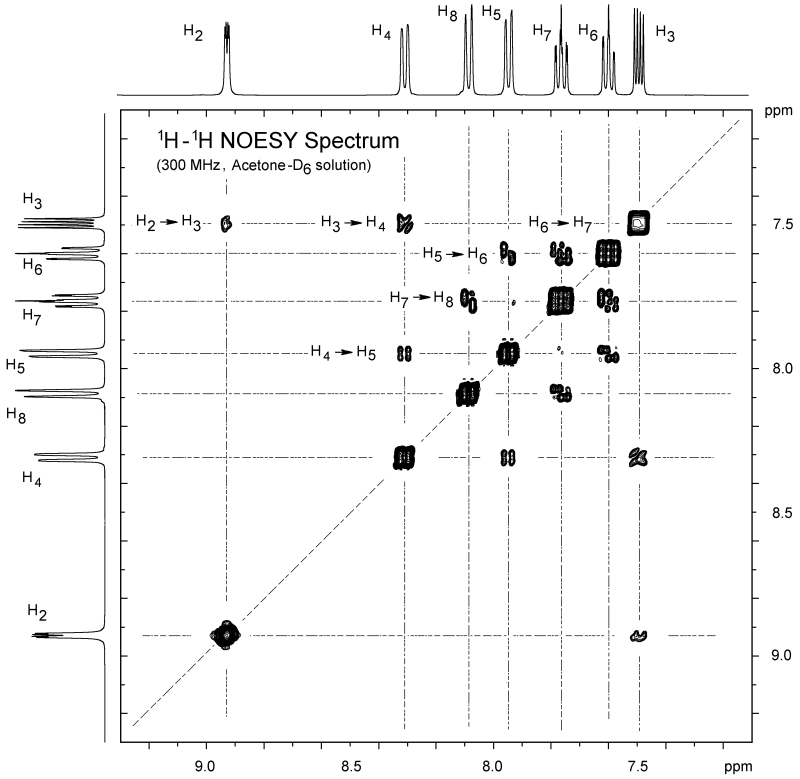


1. 1D spectra show that there are 7 clearly visible protons (no overlapping signals) and 9 clearly visible carbons (no overlapping signals).
2. The ^{13}C signals of lower intensity are probably the quaternary carbons.
3. The two "apparent triplets" (actually doublets-of-doublets) in the ^{1}H NMR spectrum must be H6 and H7 since these each have 2 *ortho* protons.

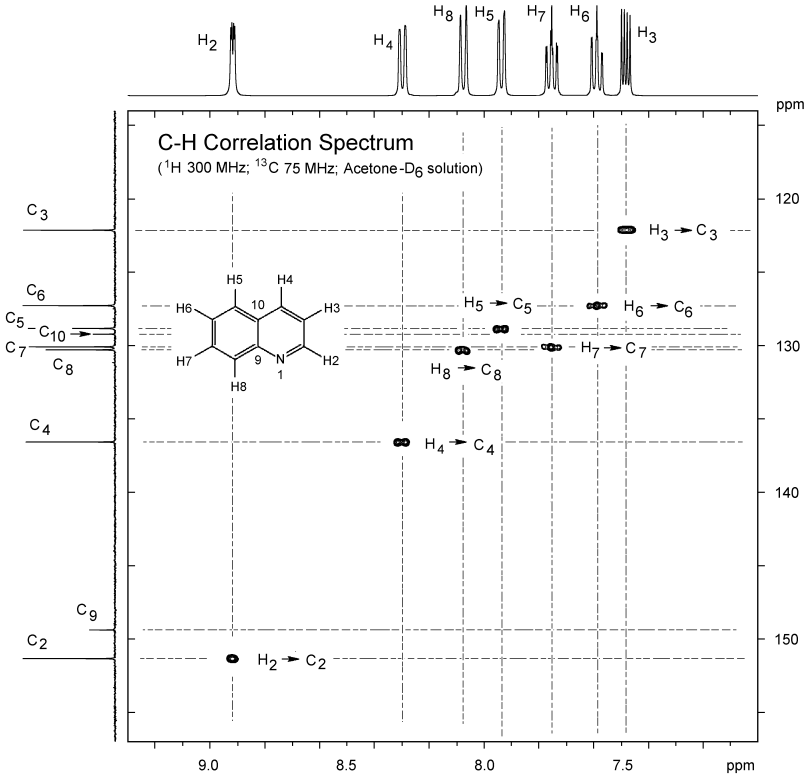




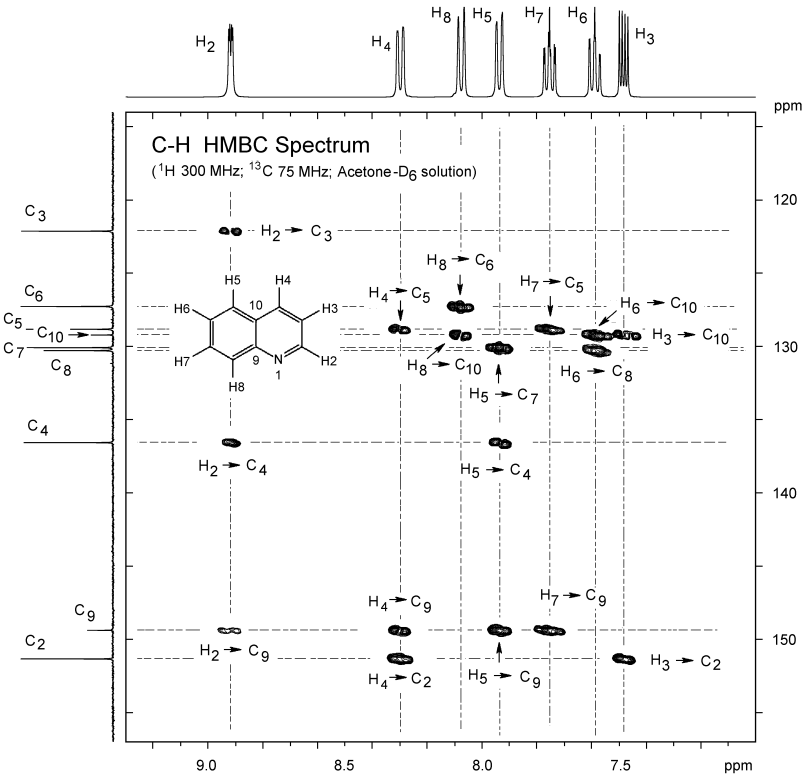
4. From the NOESY spectrum, all signals have two correlations except for the signals at δ 8.92 and δ 8.08. These must be H8 and H2 since every other proton has 2 near neighbours.
5. If you make the assumption that the proton at δ 8.92 is H8, you find that the protons which would be H7 and H6 have the wrong multiplicity in the 1D spectrum to be part of a regular aromatic spin system. The proton at δ 8.08 must be H8.
6. Once you have identified one proton in the NOESY, the other protons can be identified sequentially. H8 correlates to H7; H7 correlates to H6 and so forth until all of the protons in the spin system are identified.



7. Once the ^{1}H spectrum has been assigned, the C-H Correlation spectrum easily identifies the carbon to which each proton is correlated.
8. This assigns all of the protonated carbons and leaves only the non-protonated carbon resonances at δ 149.4 and δ 129.2 to be assigned.



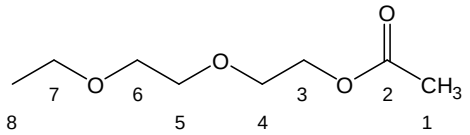
9. The HMBC spectrum is used to assign the remaining 2 carbon resonances.
10. Remember that, in aromatic systems, the 3-bond coupling $^{3}J_{H\text{-}C}$ is typically the larger long-range coupling and gives rise to the strongest cross peaks.
11. The carbon at δ 149.4 correlates to H2, H4, H5 and H7 and this must be C9. The carbon at δ 129.2 correlates to H3, H6 and H8 and this must be C10. All of the other cross peaks in the HMBC are reasonable.



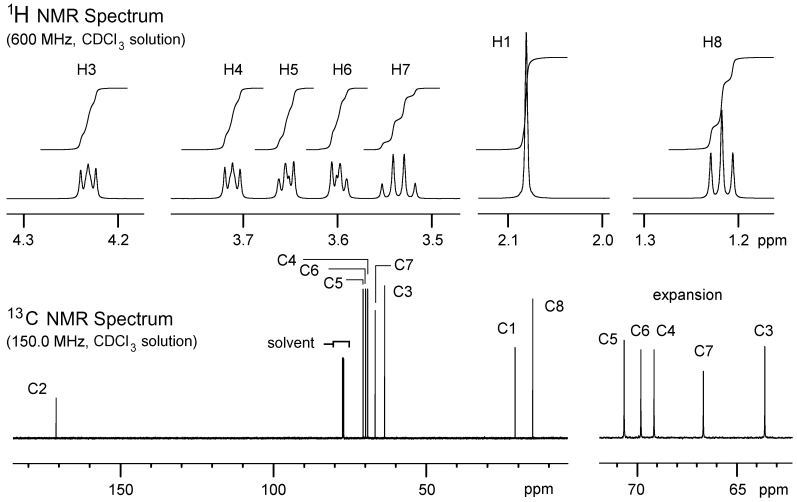
Proton	^{1}H Chemical Shift (δ) in ppm	Carbon	^{13}C Chemical Shift (δ) in ppm
H2	8.92	C2	151.3
H3	7.48	C3	122.1
H4	8.30	C4	136.6
H5	7.94	C5	128.8
H6	7.59	C6	127.3
H7	7.75	C7	130.1
H8	8.08	C8	130.3
		C9	149.4
		C10	129.2

Problem 313

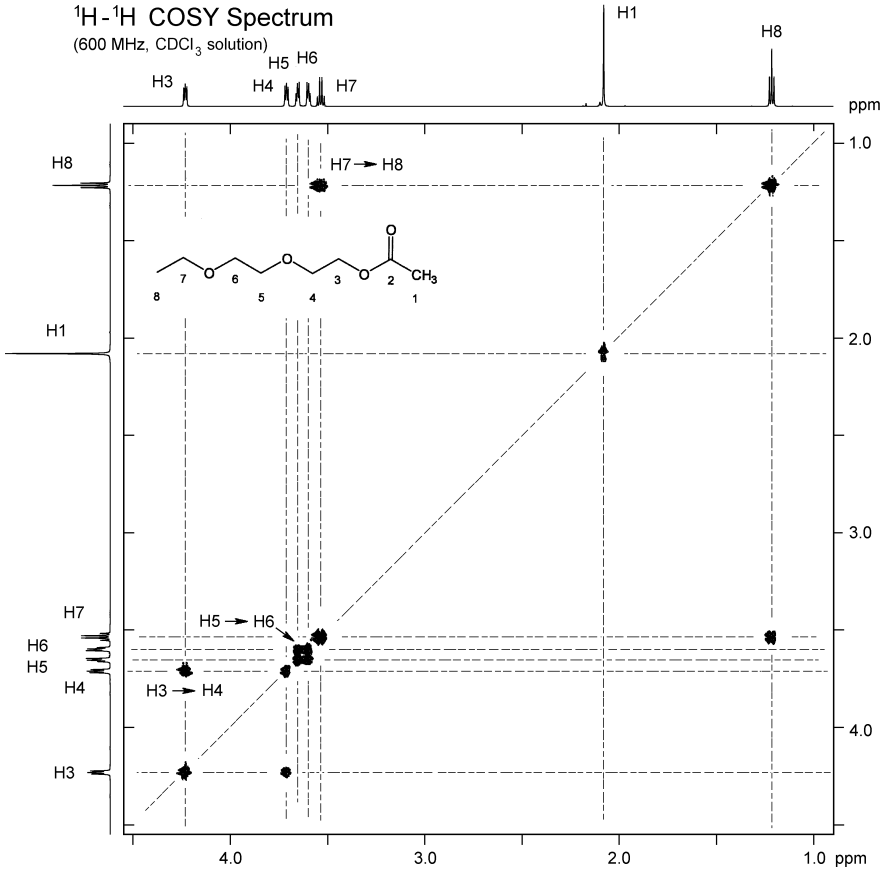
Diethyleneglycol ethyl ether acetate

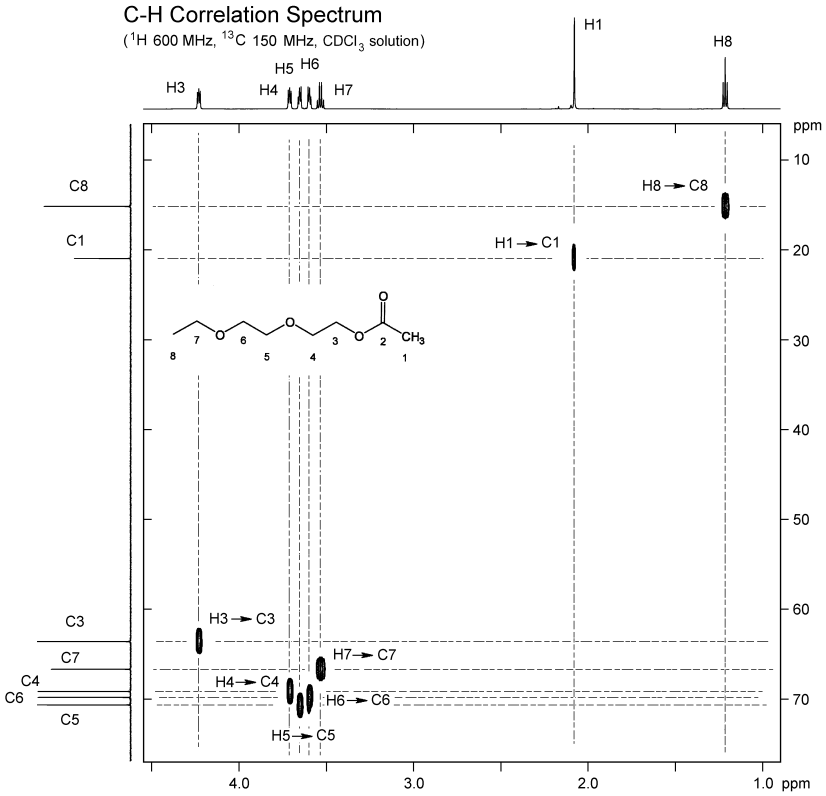


1. 1D spectra establish that there are 7 clearly visible proton signals (no overlapping signals). The integrals indicate 5 x $-CH_2-$ groups and 2 x $-CH_3$ groups consistent with the structure. The quartet at δ 3.54 and the triplet at δ 1.22 clearly belong to H7 and H8 respectively. The 3-proton singlet at δ 2.09 ppm corresponds to H1.
2. There are 8 clearly visible carbons (no overlapping signals) but there is some crowding between 60 and 75 ppm. The carbonyl resonance is clearly the signal at low field (δ 171.0).



3. From the COSY spectrum, the correlation between H7 and H8 is confirmed. The correlation between two pairs of protons H3 to H4 and H5 to H6 is clear but it is not possible to actually assign any of the protons.

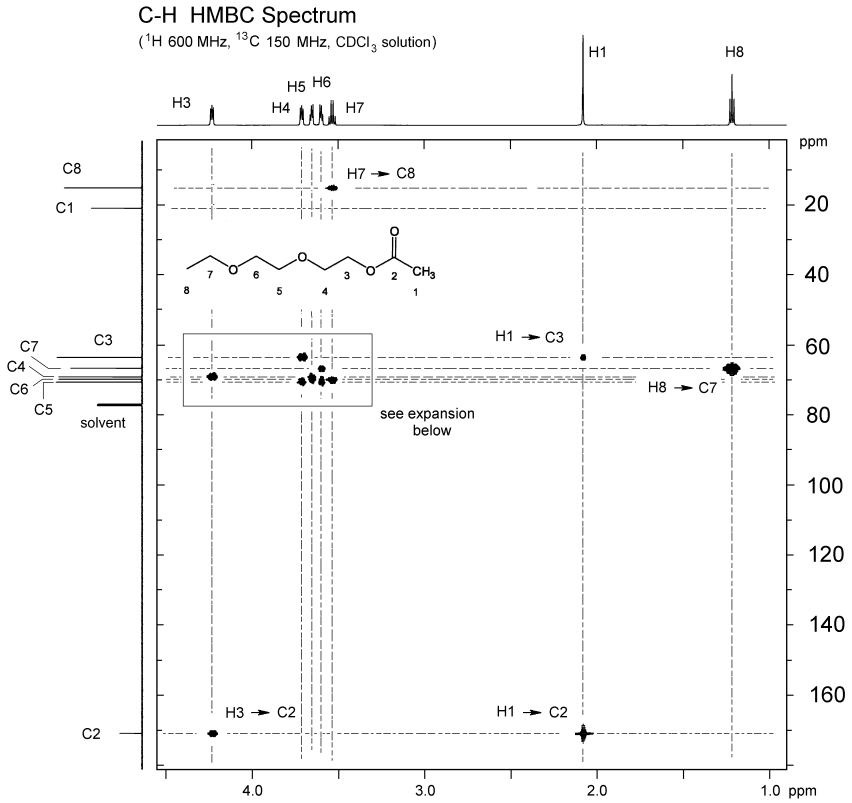




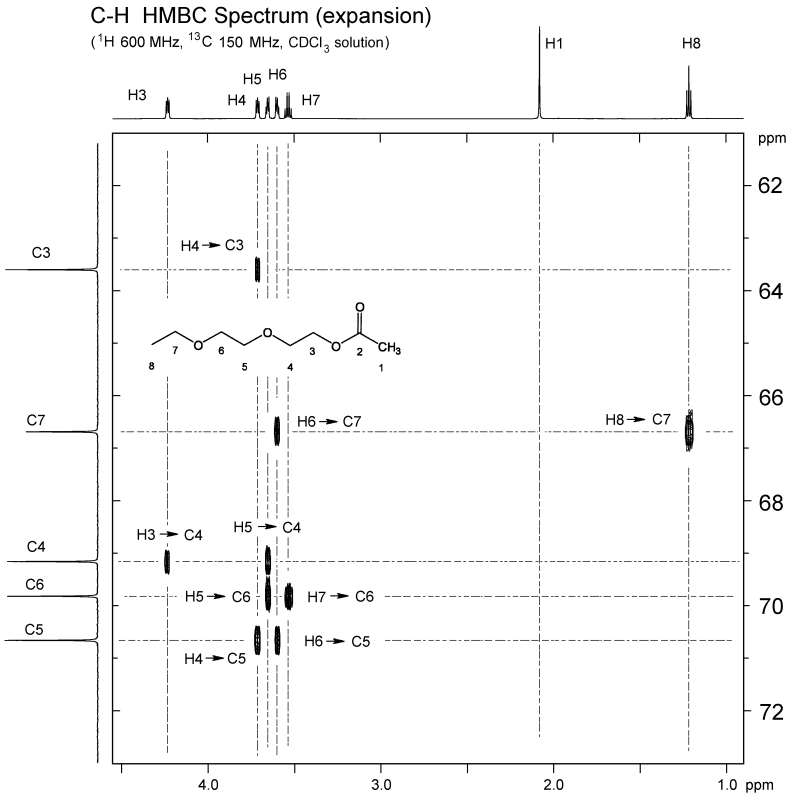
4. From the C-H Correlation spectrum, one can identify C7 and C8 by correlation to their respective protons however it is not possible to assign any of the other protonated carbons.

5. From the HMBC Spectrum, H1 correlates to C2 but also to the resonance at δ 63.6 and this identifies this signal as C3.

6. From the C-H Correlation spectrum C3 identifies H3 as the resonance at δ 4.23 and the COSY spectrum then identifies H4 as the resonance at δ 3.71 and this in turn identifies C4 in the C-H Correlation spectrum.



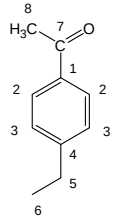
7. From the HMBC spectrum (expansion below), H4 correlates to C3 but also to C5 at δ 70.8.
8. C5 then identifies H5 in the C-H Correlation spectrum which in turn identifies H6 in the COSY spectrum which then identifies C6 in the C-H Correlation.
9. The HMBC spectrum correlates H6 to C5 but also to C7 which we have already identified.



Proton	^{1}H Chemical Shift (δ) in ppm	Carbon	^{13}C Chemical Shift (δ) in ppm
H1	2.08	C1	21.0
		C2	171.0
H3	4.23	C3	63.6
H4	3.71	C4	69.2
H5	3.66	C5	70.8
H6	3.60	C6	69.8
H7	3.54	C7	66.7
H8	1.22	C8	15.2

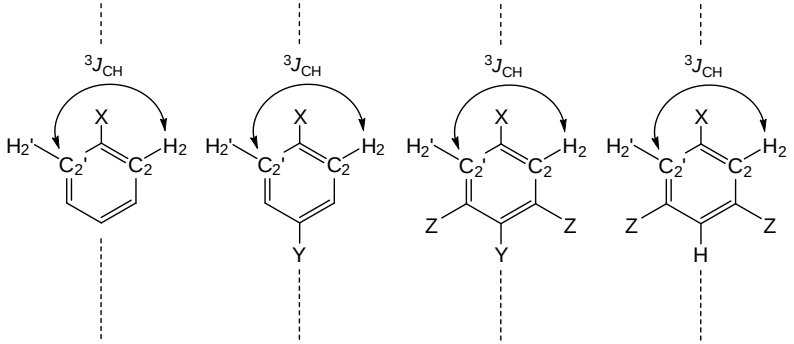
Problem 314

4-ethylacetophenone



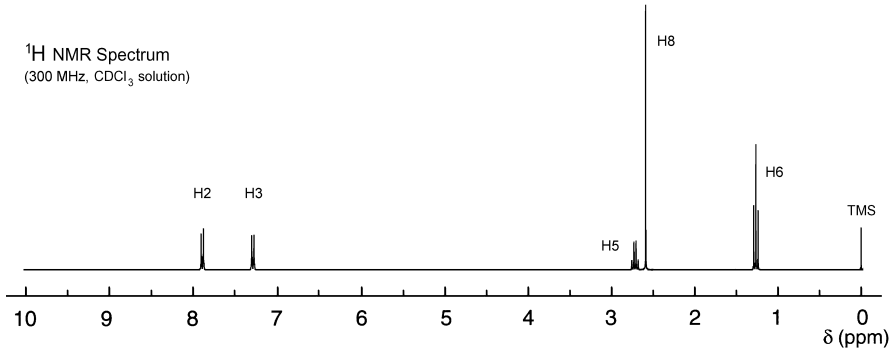
1. 1D spectra establish that the compound is a ketone from the ^{13}C where the carbonyl signal has no attached protons and from the absence of an aldehyde signal in the ^{1}H spectrum.
2. The ^{1}H & ^{13}C spectra show the presence of an ethyl group (CH_3CH_2-) & a methyl group (CH_3-).
3. It is a 1,4-disubstituted benzene from the pattern of signals in the aromatic region of the ^{1}H spectrum and from the symmetry that is evident in the ^{13}C spectrum.
4. This means that answer must be isomer **C** (4-methylpropiophenone) or **F** (4-ethylacetophenone).
5. The C-H Correlation Spectrum establishes which carbons correspond to the ethyl substituent (C5 at δ 29.2 ppm and C6 at δ 15.3 ppm) and to the isolated methyl group (C8 at δ 26.8 ppm).
6. The HMBC spectrum establishes that the methyl group is attached to the carbonyl carbon (H8 → C7) and that the CH_2 group of the ethyl group is attached to the aromatic ring (H5 → C4 & H5 → C2) and this identifies the answer as compound **F** (4-ethylacetophenone).

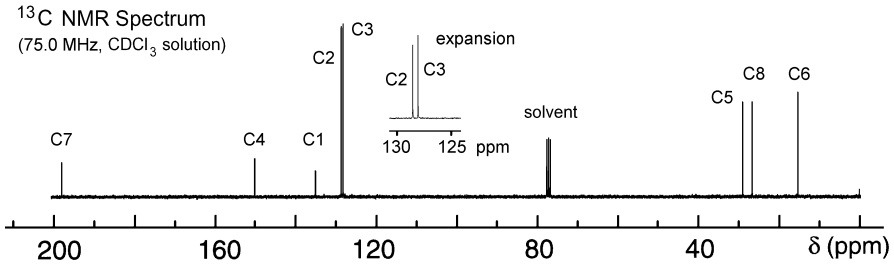
7. Note also that in the HMBC spectrum for this compound, there are strong correlations between H2 and C2, and between H3 and C3. While these appear to be 1-bond correlations, in *para*-disubstituted benzenes and in monosubstituted benzenes, or in 1,3,5- or 1,3,4,5-tetrasubstituted benzenes where there is a mirror plane of symmetry through the aromatic ring, these apparent 1-bond correlations arise from the $^{3}J_{H\text{-}C}$ interaction (H2 → C2') of a proton with the carbon which is *meta* to it.

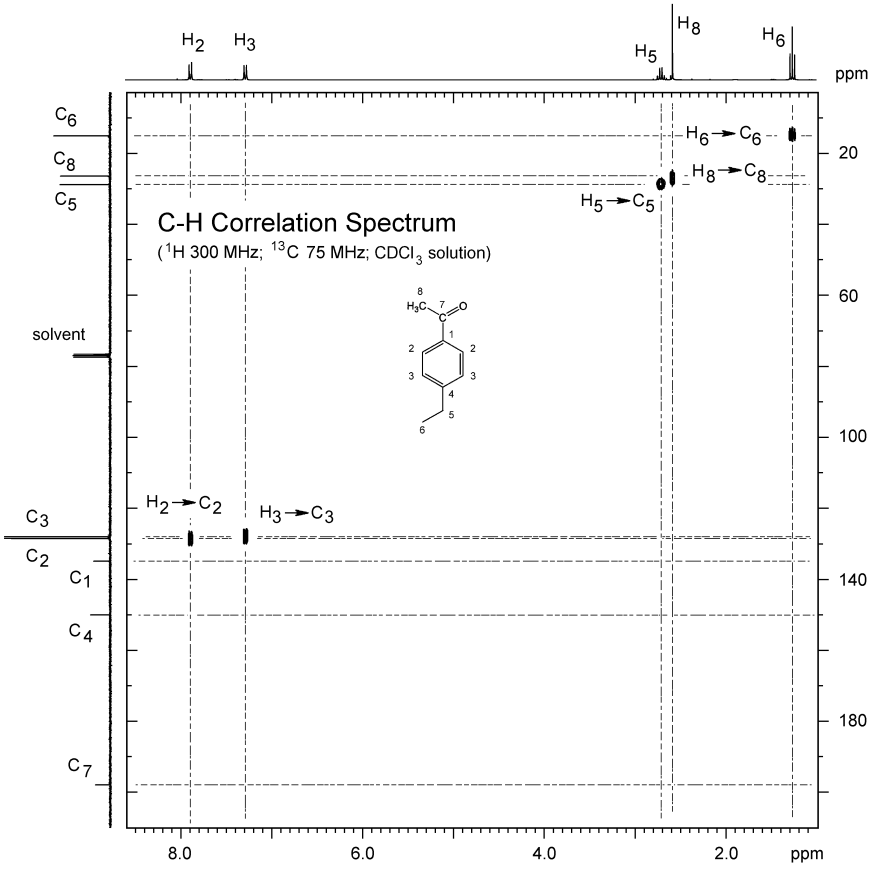


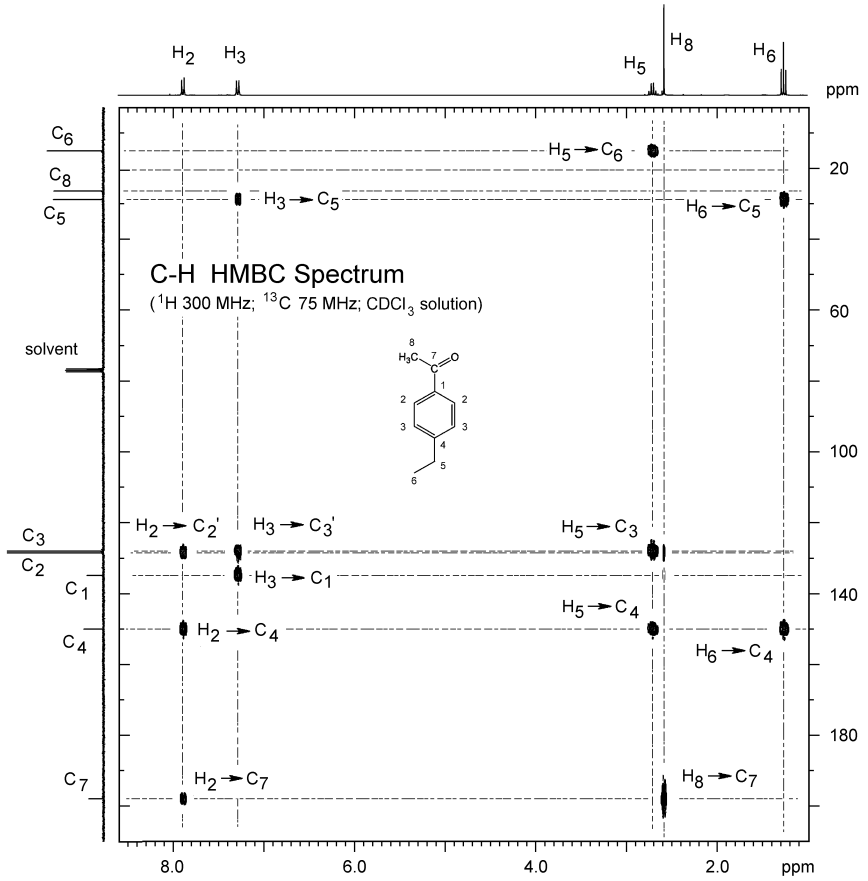
8. The ^{1}H and ^{13}C assignments follow by inspection and are consistent with the structure.

Proton	^{1}H Chemical Shift (δ) in ppm	Carbon	^{13}C Chemical Shift (δ) in ppm
		C1	135.0
H2	7.9	C2	128.9
H3	7.3	C3	128.3
		C4	150.1
H5	2.7	C5	29.2
H6	1.3	C6	15.3
		C7	198.1
H8	2.6	C8	26.8



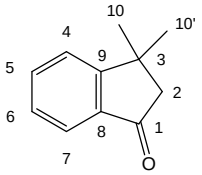




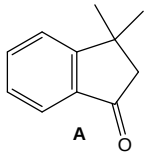


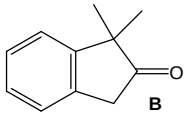
Problem 315

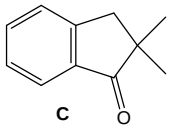
3,3-dimethylindanone



1. 1D spectra establish that the compound is a dimethylindanone but it is not easy to distinguish between 3 of the isomers (**A-C**):

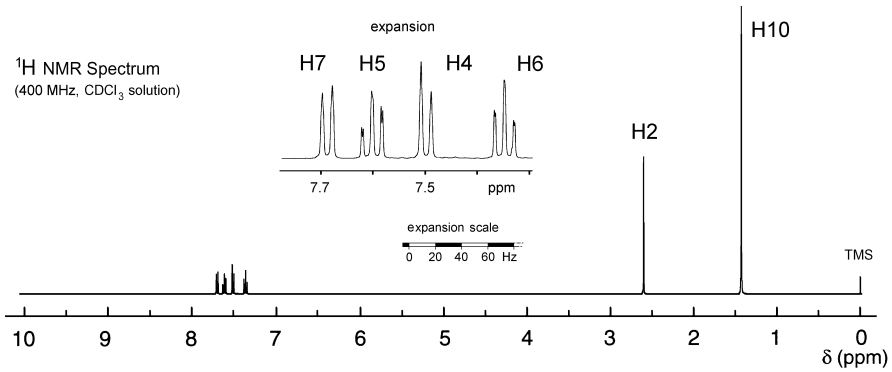


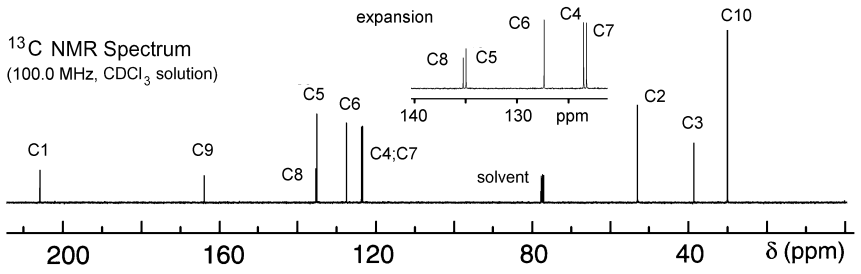


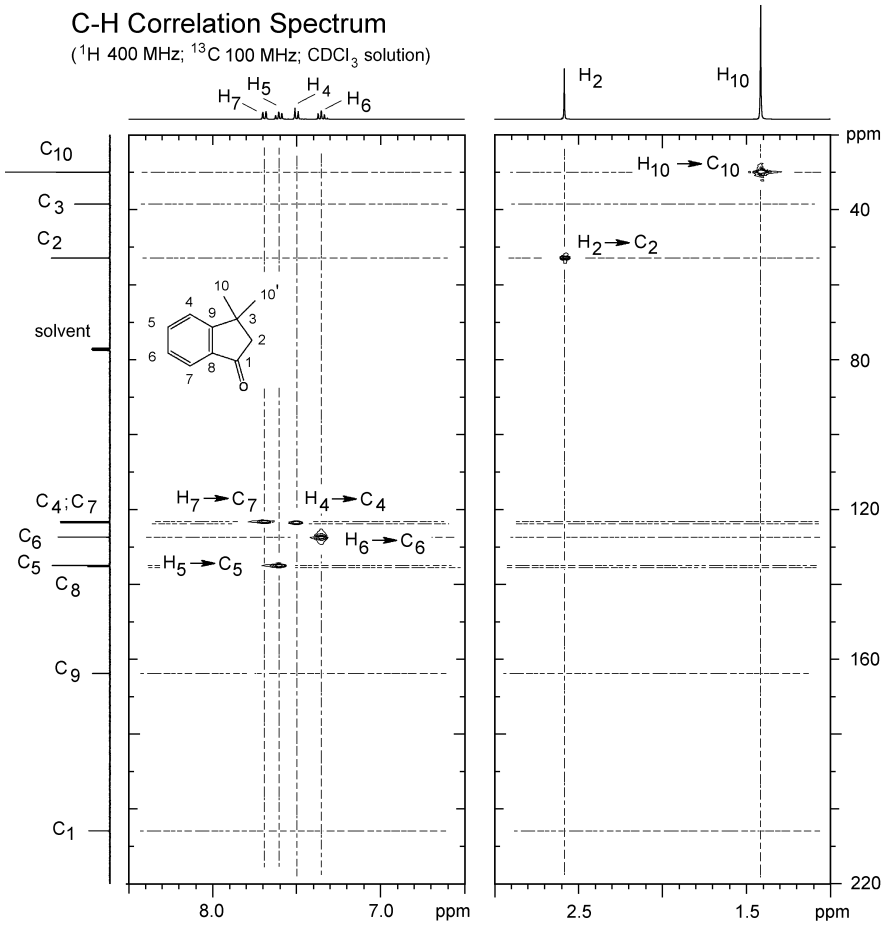


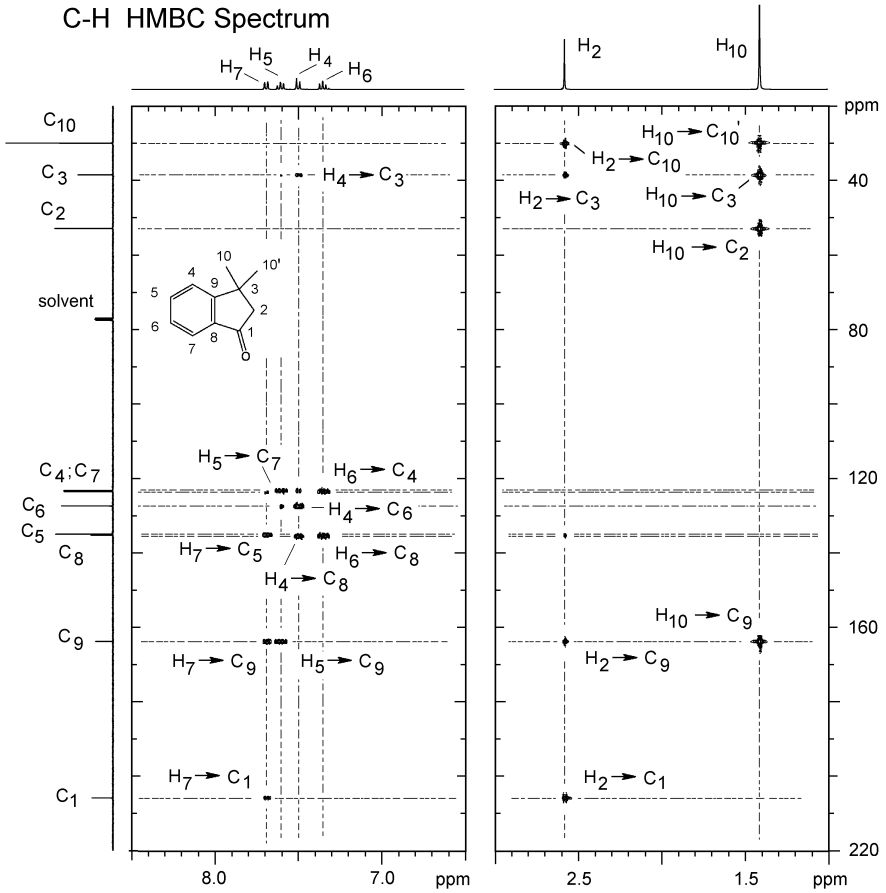
2. In the ^{1}H spectrum, the assignment of the –CH_2- group (C2 at δ 2.58 ppm) and the -CH_3 groups (C10 at δ 1.41 ppm) are obvious (by integration).
3. In the ^{13}C spectrum, the assignment of the carbonyl group is obvious from its chemical shift (C1 at δ 205.8 ppm). The DEPT experiment and the C-H correlation experiment identifies C2 at δ 52.9 ppm.
4. The HMBC spectrum establishes that the -CH_2- group is adjacent to C3 (H2 → C3) and also to the carbonyl group (H2 → C1). The carbonyl carbon is correlated to H7 in the aromatic ring (H7→ C1). The carbonyl is in a position which is adjacent to the aromatic ring. There are no correlations from H2 to the protonated carbons in the aromatic ring
5. The compound is 3,3-dimethylindanone (Isomer A).
6. Once H7 is assigned, the proton and carbon assignments then follow sequentially. All other HMBC correlations are reasonable for 3,3-dimethylindanone.

Proton	^{1}H Chemical Shift (δ) in ppm	Carbon	^{13}C Chemical Shift (δ) in ppm
		C1	205.8
H2	2.58	C2	52.9
		C3	38.5
H4	7.42	C4	123.5
H5	7.60	C5	134.9
H6	7.35	C6	127.4
H7	7.68	C7	123.2
		C8	135.2
		C9	163.8
H10	1.41	C10	29.9

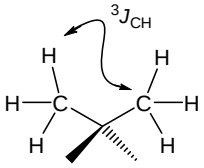






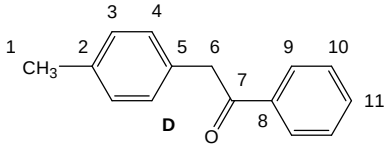


7. Note also that in the HMBC for this compound, there is a strong correlation between H10 and C10'. While this appears to be a 1-bond correlation, in *t*-butyl groups, isopropyl groups or in compounds with a *gem*-dimethyl group, the apparent 1-bond correlation arises from the $^{3}J_{H\text{-}C}$ interaction of the protons of one of the methyl groups with the chemically equivalent carbon which is 3 bonds away.



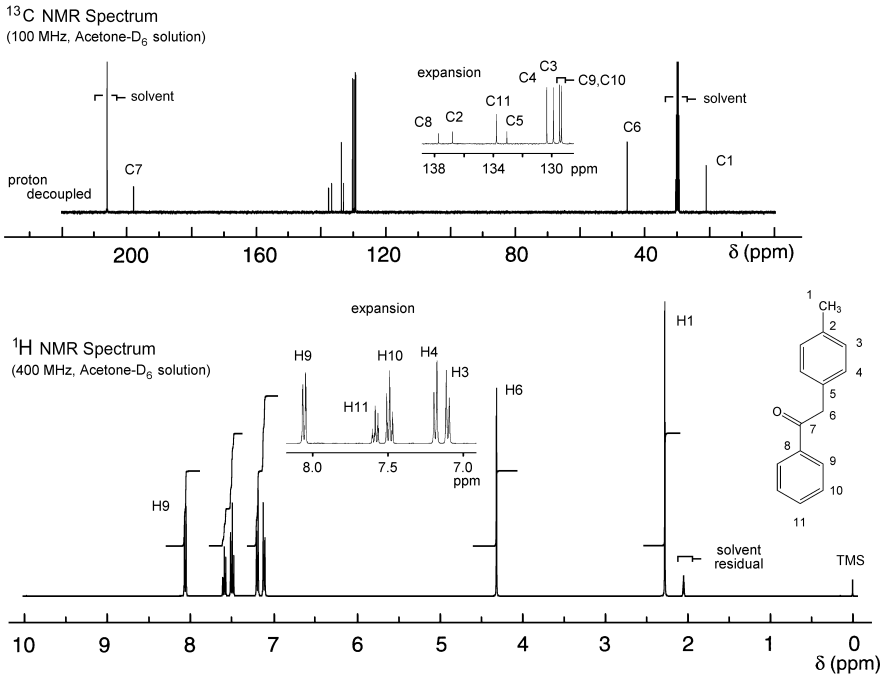
Problem 316

4-methylbenzyl phenyl ketone



1. 1D spectra establish that the compound has a -CH_3 group at δ 2.28 ppm and a –CH_2- group (H6 at δ 4.32 ppm).
2. The pattern of resonances in the expansion of the ^{1}H aromatic region of the spectrum shows 9 aromatic protons. There are obviously 4 protons on a *para*-disubstituted ring (H3 and H4 at δ 7.13 and 7.21 ppm) but one can't readily distinguish which is H3 and which is H4. There are 5 protons on a monosubstituted aromatic ring (H9 at δ 8.08 ppm as a 2-proton signal with the superficial appearance of a doublet resulting from one large *ortho* splitting; H10 at δ 7.51 ppm as a two-proton signal with the superficial appearance of a triplet resulting from two large *ortho* splittings; and H11 at δ 7.61 ppm as a 1-proton signal with the superficial appearance of a triplet resulting from two large *ortho* splitting).
3. Ignoring the solvent resonances (singlet at δ 205.9 and multiplet δ 29.9 ppm), the ^{13}C spectrum shows an obvious carbonyl (at δ 197.8 ppm) and resonances for the aliphatic –CH_2- (C6 at 45.4 ppm) and –CH_3 (C1 at δ 21.0 ppm). There are 3 substituted (*i.e.* non-protonated) aromatic carbons in the spectrum at δ 137.7, 136.8, 133.1 ppm and 5 protonated carbons at δ 133.8, 130.3, 129.99, 129.94, 129.93 ppm.

Proton	^{1}H Chemical Shift (δ) in ppm	Carbon	^{13}C Chemical Shift (δ) in ppm
H1	2.28	C1	21.0
		C2	136.8
H3	7.13	C3	129.93
H4	7.21	C4	129.94
		C5	133.1
H6	4.32	C6	45.4
		C7	197.8
		C8	137.7
H9	8.08	C9	130.3 or 129.99
H10	7.51	C10	130.3 or 129.99
H11	7.61	C11	133.8



4. In the expansion of the CH Correlation Spectrum, it is apparent that H11 (δ 7.61 ppm) correlates to C11 (δ 133.8 ppm) and that the H9 and H10 correlate to the carbons at δ 130.3 and 129.99 ppm) but it is difficult to assign them individually. Likewise the protons on the *para*-disubstituted aromatic ring (H3 and H4) correlate to carbons at 129.94 and 129.93 ppm) but it is not possible to assign the carbons further.
5. In the CH HMBC Spectrum, the carbonyl group (C7 at δ 197.8 ppm) correlates to both H9 (δ 8.08 ppm) and to H6 (δ 4.32 ppm). The carbonyl group is attached to the monosubstituted ring and this is only consistent with isomer D.
6. To complete the assignments – the methyl carbon (H1 at δ 2.28 ppm) correlates to the high field aromatic proton (δ 7.13 ppm) which confirms this proton as H3. Likewise the –CH_2-

carbon correlates to the other aromatic protons of the *para*-disubstituted ring (d 7.21 ppm) and confirms these protons as H4.

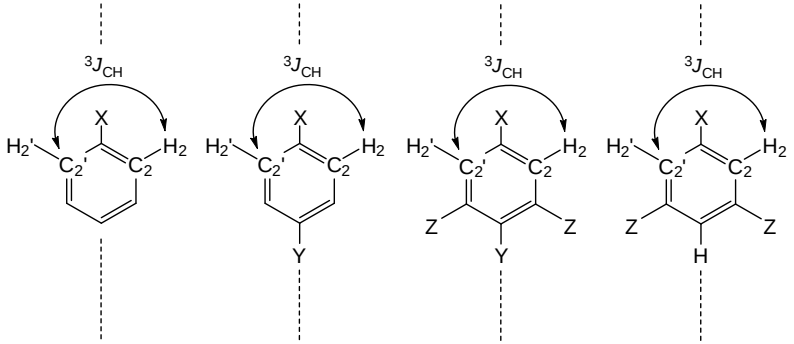
7. The $-CH_2-$ protons H6 at δ 4.32 ppm, correlate to one of the unprotonated carbons at δ 133.1 confirming this as C5.

8. The $-CH_3$ protons H1 at δ 2.28 ppm, correlate to one of the unprotonated carbons at δ 136.8 confirming this carbon as C2.

12. The protons identified as H10 at δ 7.51 ppm, correlate to one of the unprotonated carbons at δ 137.7 confirming this carbon as C8.

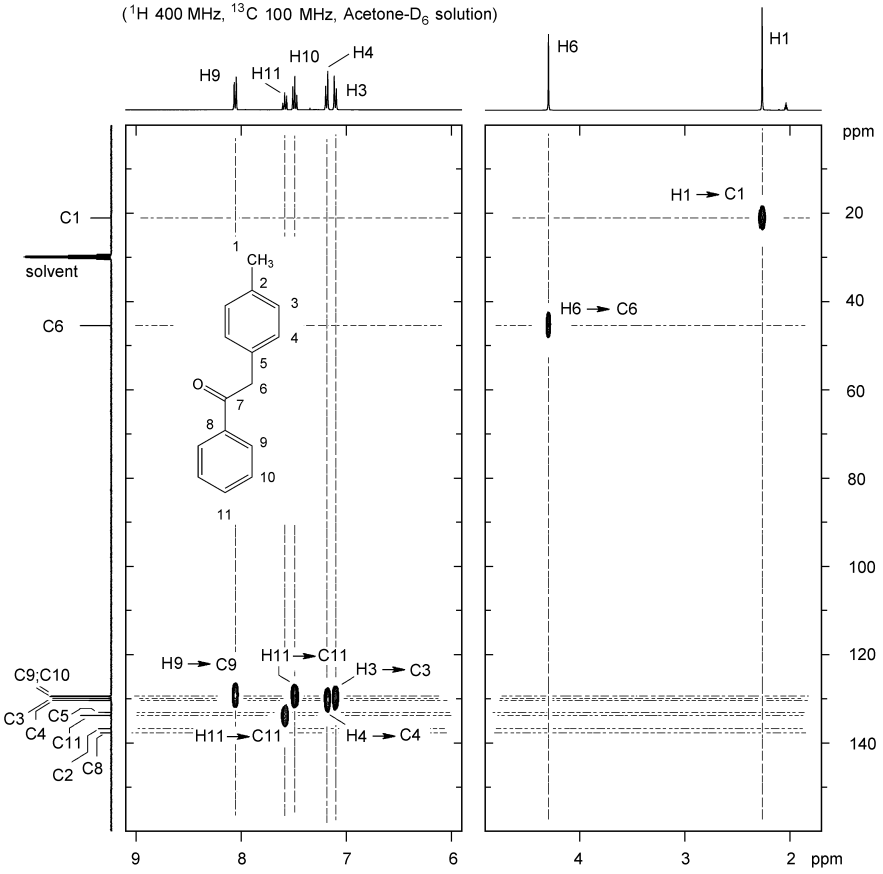
13. Remember that, in aromatic systems, the 3-bond coupling $^3J_{H\text{-}C}$ is typically the larger long-range coupling and gives rise to the strongest cross peaks.

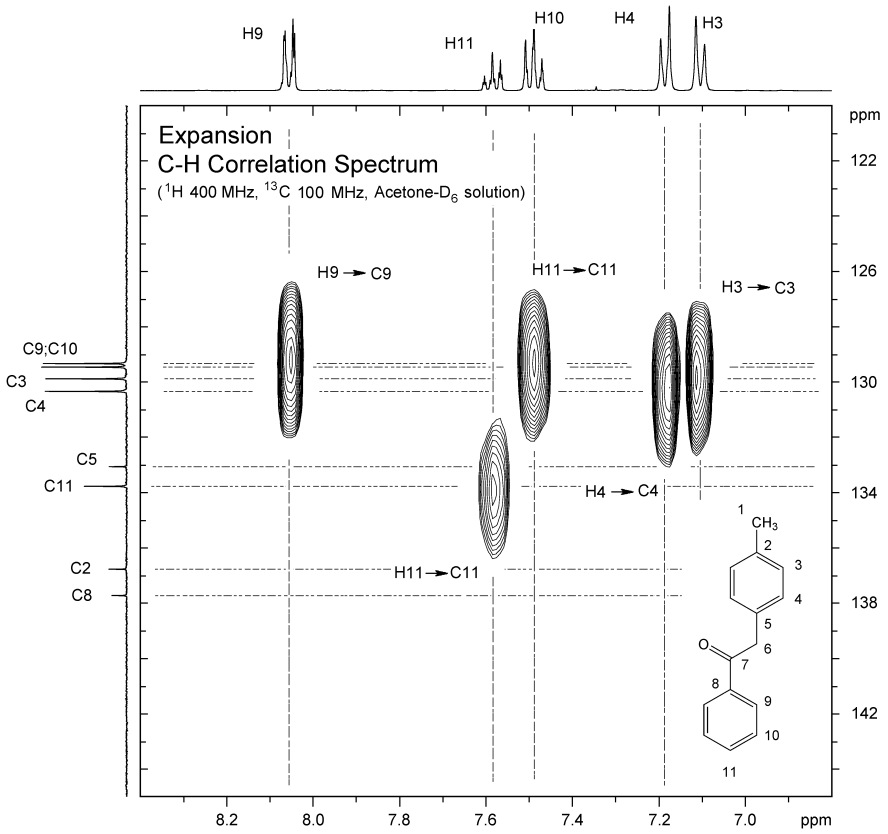
14. Note also that in the HMBC for this compound, there are strong correlations between H3 and C3', H4 and C4', H9 and C9' and between H10 and C10'. While these appear to be 1-bond correlations, in *para*-disubstituted benzenes and in monosubstituted benzenes, or in 1,3,5- or 1,3,4,5-tetrasubstituted benzenes where there is a mirror plane of symmetry through the aromatic ring, these apparent 1-bond correlations arise from the $^3J_{H\text{-}C}$ interaction (H2 → C2') of a proton with the carbon which is *meta* to it.

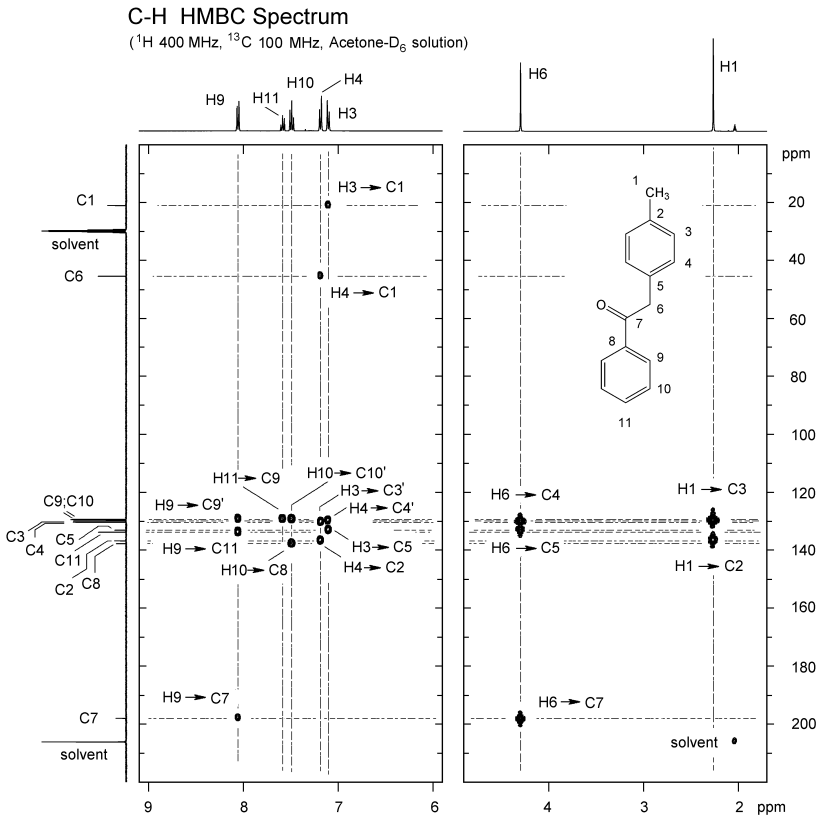


15. C9 and C10 have resonances which are close together (δ 129.94 and δ 129.93 ppm) and it is not possible to assign these resonances unambiguously from the spectra.

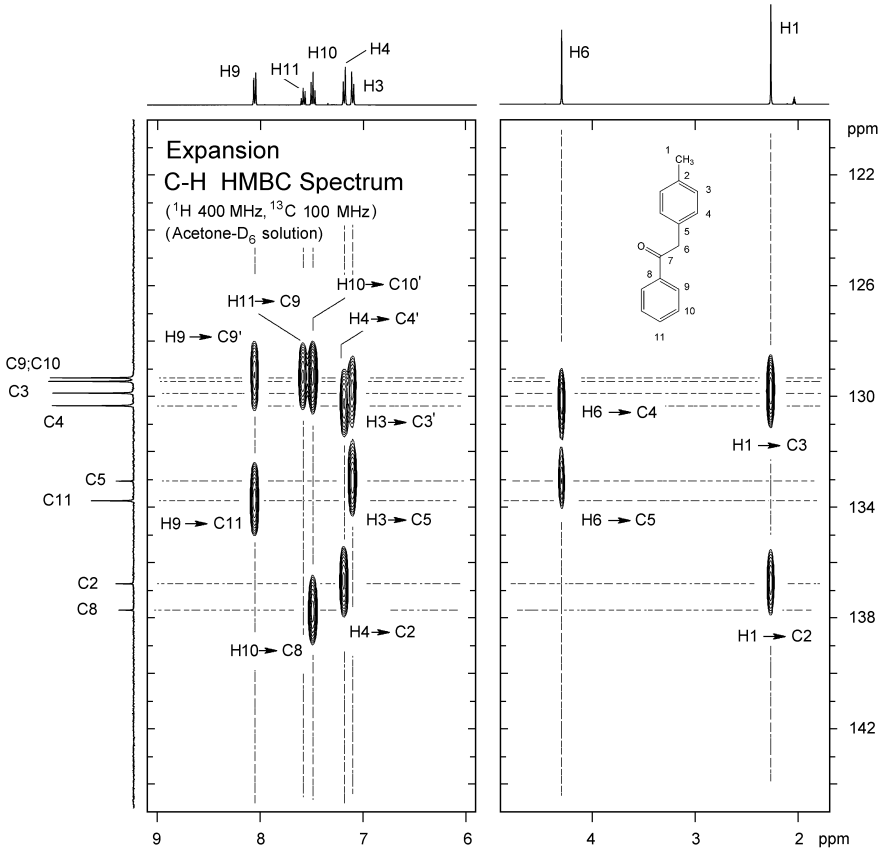
C-H Correlation Spectrum
(^{1}H 400 MHz, ^{13}C 100 MHz, Acetone-D_6 solution)



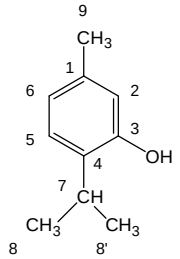




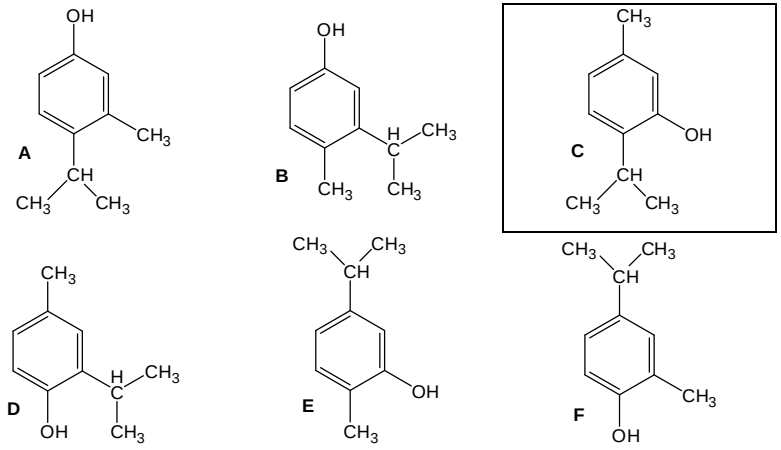
Problem 317



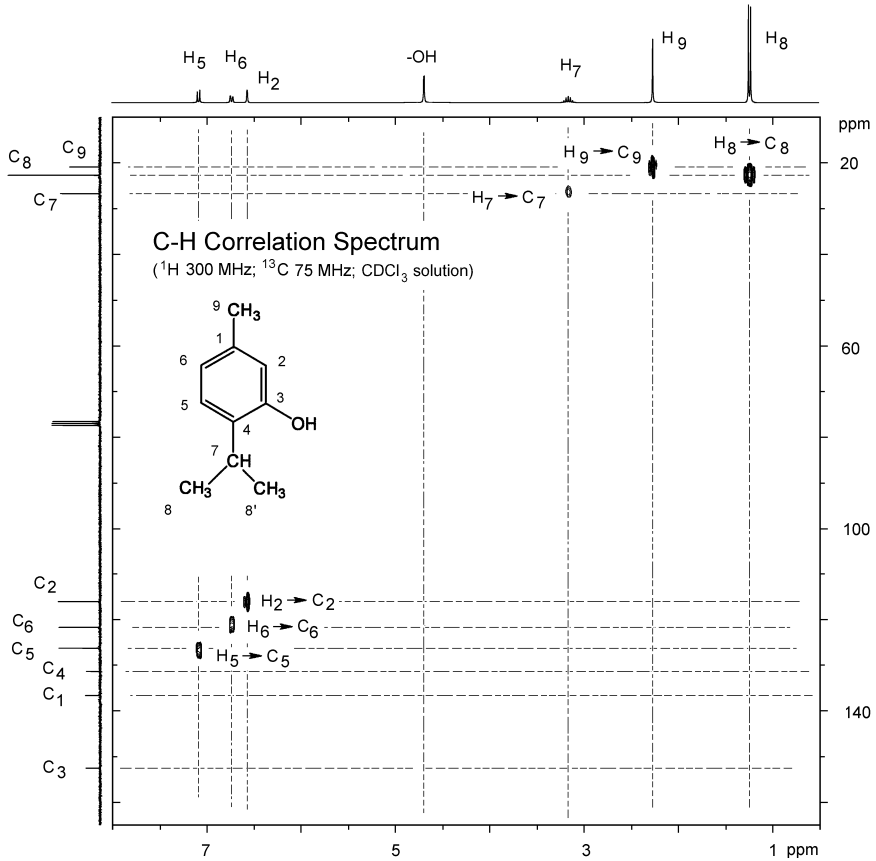
thymol



1. 1D spectra establish that the compound is a trisubstituted benzene with an –OH substituent, a –CH_3 substituent and an isopropyl substituent.
2. The coupling pattern in the expansion of the aromatic region of the ^{1}H spectrum establishes that the substituents are in positions 1,3 & 4 on the aromatic ring and that H2, H5 and H6 are at δ 6.6, 7.1 & 6.7 respectively, however it is difficult to establish which substituents are where.
3. For a 1,3,4 trisubstituted benzene, there are 6 possible isomers.



4. The C-H Correlation Spectrum easily identifies the protonated carbons. The aliphatic carbons C9, C8 & C7 are at δ 21, 22 & 28 ppm respectively. The protonated aromatic carbons C2, C6 & C5 are at δ 116, 121 & 126 ppm respectively.



5. In the HMBC, the methyl protons (H9) correlate to C2 & C6 and also to the signal at δ 138 – so this identifies the carbon to which the methyl is attached.

6. **Remember** that, in aromatic systems, the 3-bond coupling $^{3}J_{H\text{-}C}$ is typically the larger long-range coupling and gives rise to the strongest cross peaks.

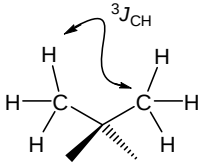
7. In the HMBC, the resonance at δ 138 also correlates to H5 which places it in a 1,3-relationship with H5 and eliminates isomers **B** and **E**.

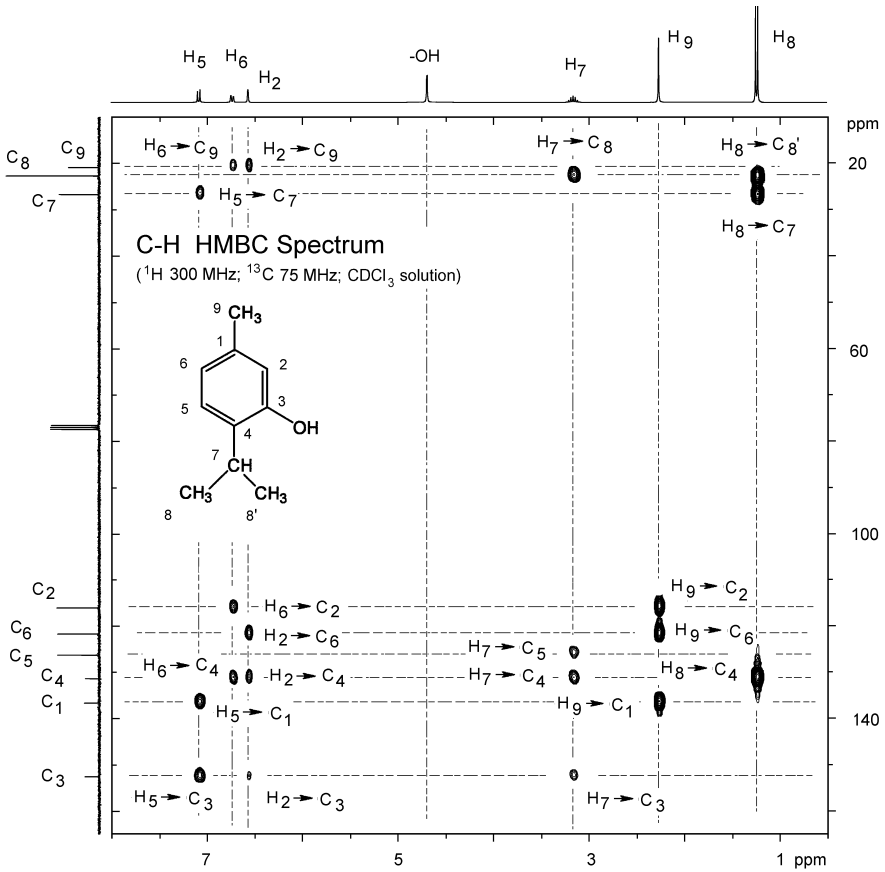
8. In the HMBC, the protons at H6 and H2 correlate to C4 which identifies it as the signal at δ 132. This leaves the signal at δ 152 ppm as C3.

9. In the HMBC, the methane proton (H7) correlates to (C9) correlate to C8 and also to C3 & C4 and C5. This places the isopropyl group at C4 and leaves the –OH group at C3.

10. The fact that C3 is the aromatic signal at lowest field is consistent with the fact that it bears the –OH substituent. In the HMBC, C3 also correlates strongly to H5 which is consistent with its 1,3-relationship with H5.

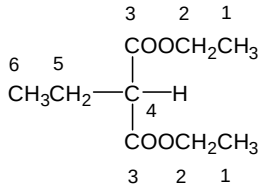
11. Note also that in the HMBC for this compound, there is a strong correlation between H8 and C8'. While this appears to be a 1-bond correlation, in *t*-butyl groups, isopropyl groups or in compounds with a *gem*-dimethyl group, the apparent 1-bond correlation arises from the $^{3}J_{H\text{-}C}$ interaction of the protons of one of the methyl groups with the chemically equivalent carbon which is 3 bonds away.



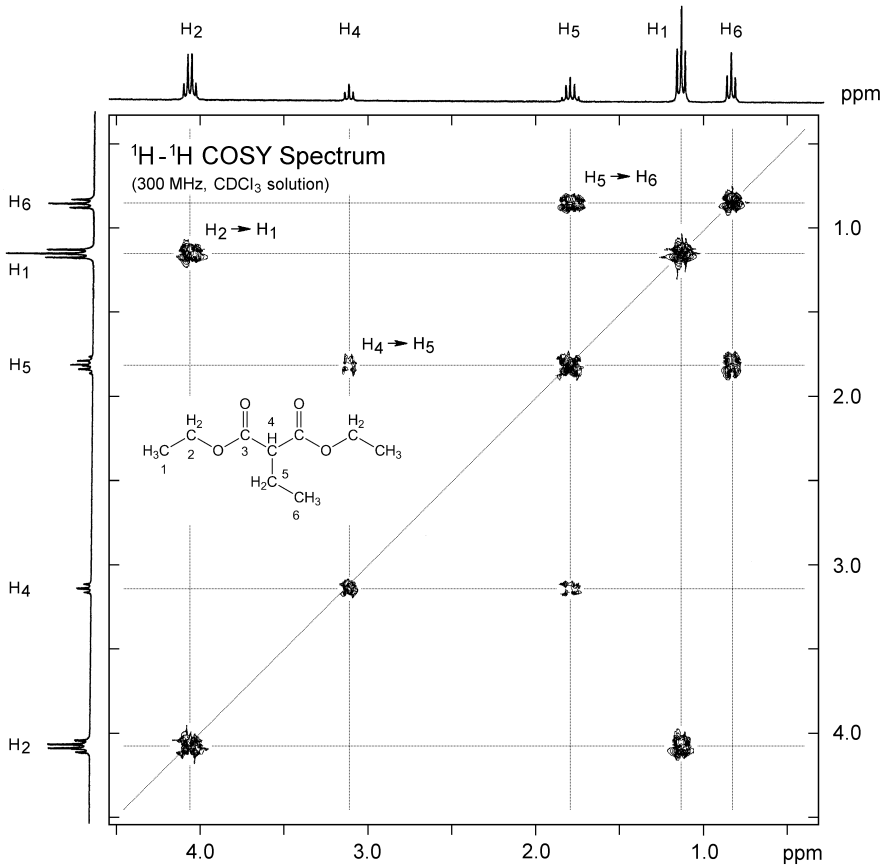


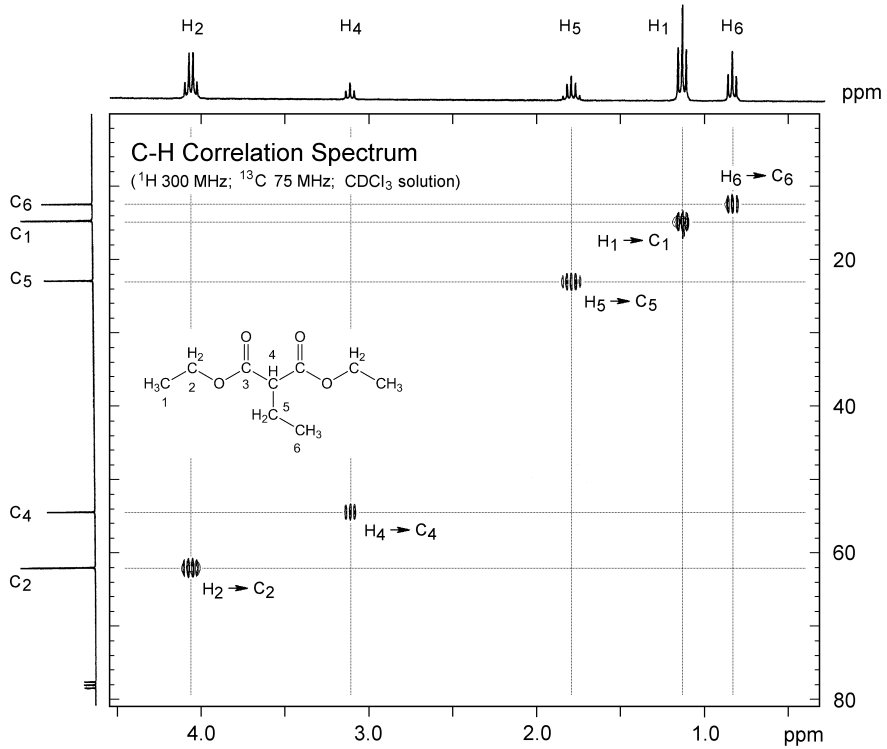
Problem 318

diethyl ethylmalonate



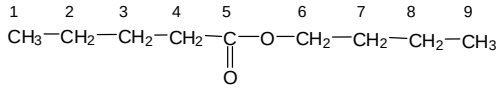
1. The IR Spectrum indicates a carbonyl functional group. The 1D NMR spectra establish this as an ester (chemical shift in ^{13}C spectrum, absence of exchangeable protons in ^{1}H spectrum).
2. There is clearly some symmetry in this compound because there are only 6 x ^{13}C resonances for the 9 carbons in the molecular formula.
3. The low-field resonance in ^{1}H spectrum (at δ 4.1 ppm, integration 4 protons) is consistent with deshielding by electronegative oxygen, identifying this as 2 x $-CH_2-$ groups (H2) bound directly to the ester oxygen.
4. The H2 resonance correlates with its carbon signal C2 (at δ 61 ppm) in the C-H correlation spectrum. The DEPT spectrum, also confirms this as a $-CH_2-$ resonance.
5. ^{1}H-^{1}H COSY shows that H2 is correlated with H1 at δ 1.15 ppm (integration 6 protons), establishing the presence of two chemically equivalent ethyl esters.
6. The resonance at δ 3.1 ppm in the ^{1}H spectrum (integration 1 proton) is correlated to the resonance at δ 1.85 ppm (integration 2 protons). Further, this resonance correlates to the resonance at δ 0.9 ppm (integration 3 protons), establishing the presence of a $-CHCH_2CH_3$ fragment.
7. By connecting the known pieces, the structure is identified as diethyl ethylmalonate.



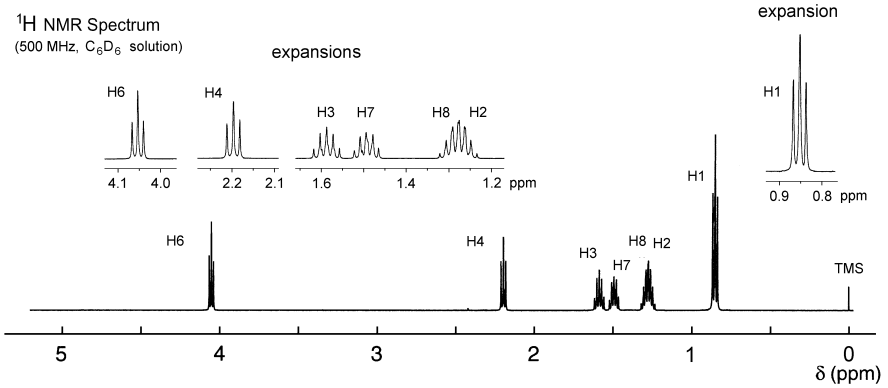


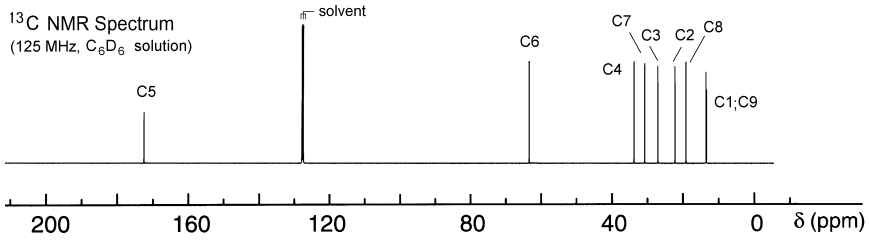
Problem 319

butyl valerate



1. The IR spectrum indicates a carbonyl functional group. The 1D NMR spectra establish this as an ester (chemical shift in ^{13}C spectrum, absence of exchangeable protons in ^{1}H spectrum).
2. It is clear from the C-H Correlation Spectrum that the signal near δ 1.25 ppm is two overlapping -CH_2- resonances, while the signal at δ 0.85 is two overlapping -CH_3 signals.

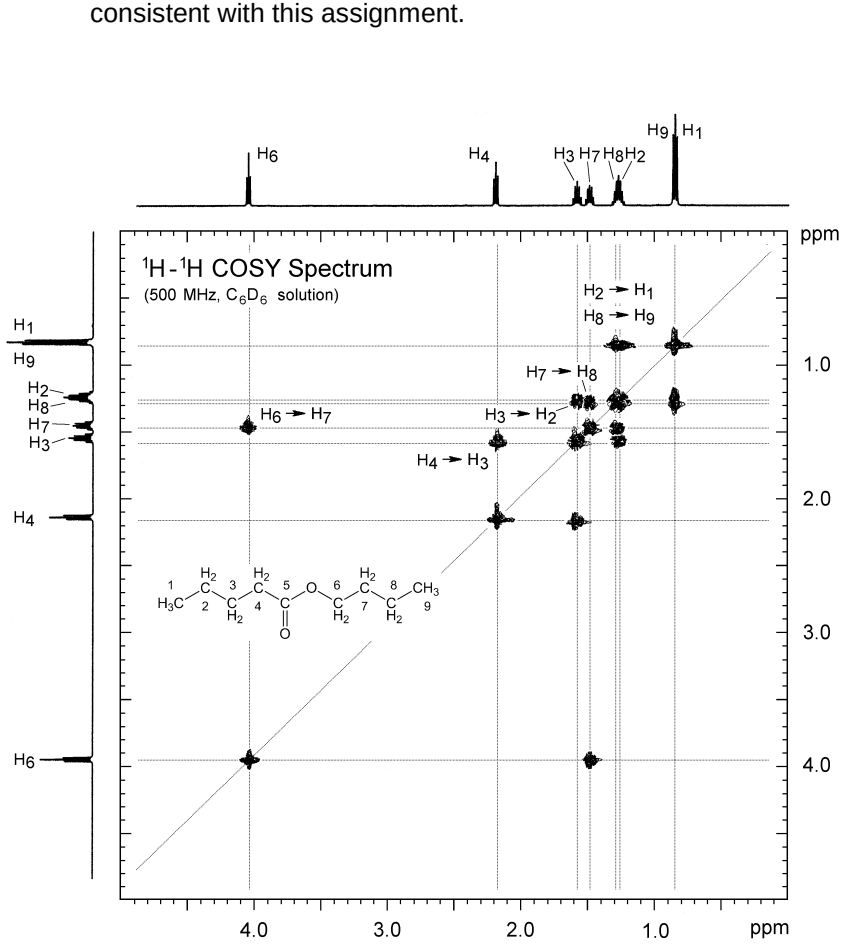


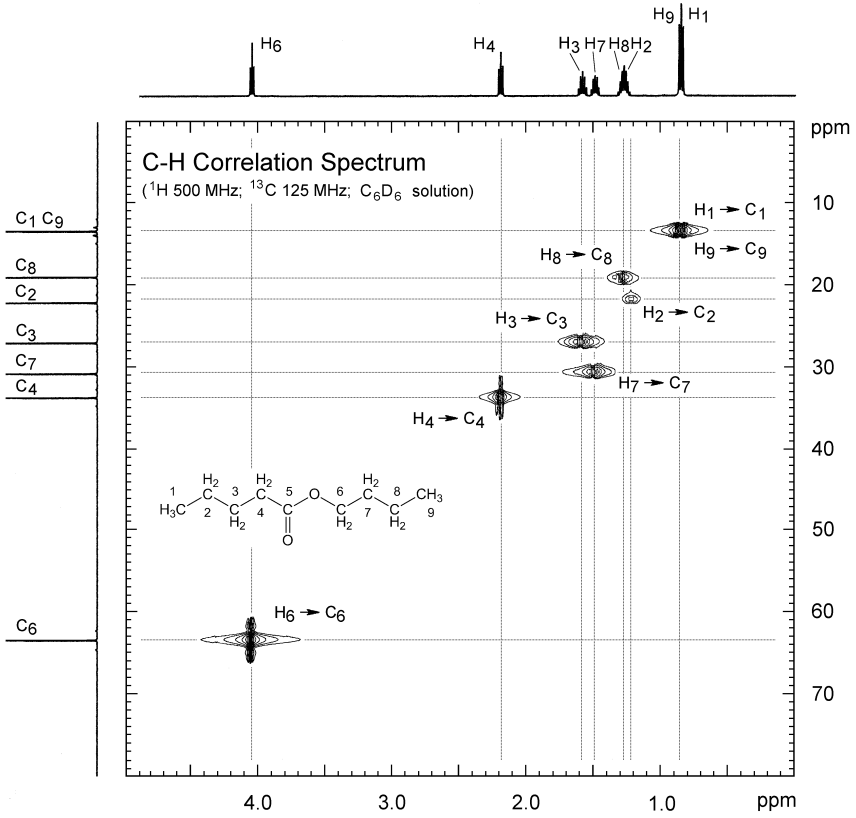


Proton	1H Chemical Shift (δ) in ppm	Carbon	13C Chemical Shift (δ) in ppm
H1	0.85	C1	13.3
H2	1.27	C2	22.1
H3	1.58	C3	27.0
H4	2.20	C4	33.6
		C5	172.4
H6	4.05	C6	63.4
H7	1.49	C7	30.7
H8	1.28	C8	19.0
H9	0.85	C9	13.3

3. The low-field resonance in the ^{1}H NMR spectrum (at δ 4.05 ppm, integration 2) is consistent with deshielding by electronegative oxygen, and this must be H6, bound directly to the ester oxygen.
4. Once H6 has been assigned, the ^{1}H-^{1}H COSY spectrum is used to trace the remaining parts of the butyl spin system: H6 correlates to H7 (δ 1.49 ppm); H7 correlates to H8 (δ 1.28 ppm, overlapping signal), and H8 correlates to the terminal methyl group H9 (δ 0.85 ppm), giving the -$C(=O)OCH_2CH_2CH_2CH_3$ fragment.
5. A second spin system may also be identified using the COSY spectrum. The -CH_2- resonance at δ 2.20 ppm (H4) correlates to H3 (δ 1.58 ppm); H3 correlates to H2 (δ 1.27 ppm, overlapping signal), H2 correlates to the terminal methyl group H1 (δ 0.85 ppm). This gives the remaining fragment as -$CH_2CH_2CH_2CH_3$.
6. Assembling the two parts identifies the compound as $CH_3CH_2CH_2CH_2C(=O)OCH_2CH_2CH_2CH_3$ – butyl valerate.

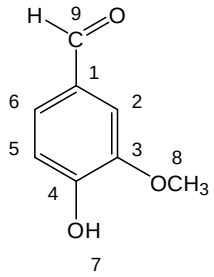
3. The remaining cross peaks in the C-H Correlation Spectrum are consistent with this assignment.

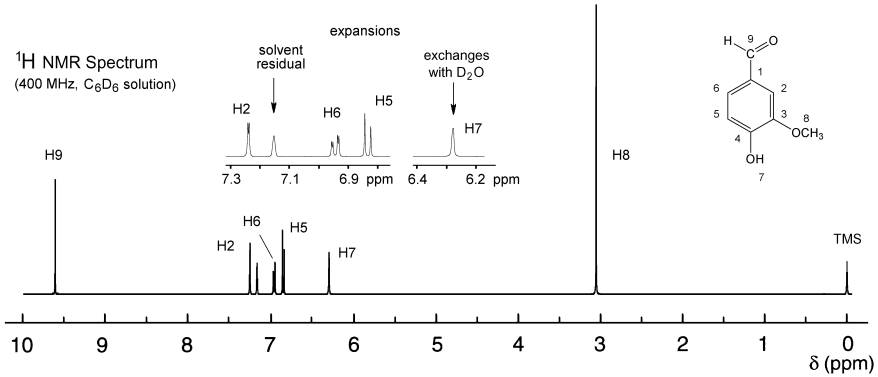




Problem 320

vanillin

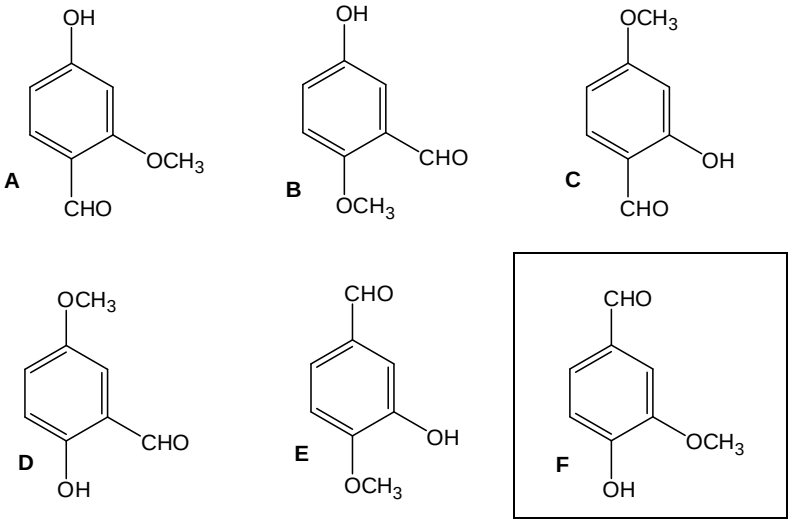




1. The functional groups are apparent from an analysis of the 1D spectra. The aldehyde group is obvious from the ^{13}C spectrum (C9 at δ 190.3) and the DEPT spectrum which indicates that the carbonyl has one attached proton.
2. The aldehyde proton (H9) is obvious in the ^{1}H NMR spectrum as a 1-proton resonance at δ 9.60 ppm. There is also a carbonyl stretch in the IR at 1670 cm^{-1} consistent with an aldehyde conjugated to an aromatic ring.
3. The methoxy group is a characteristic 3-proton singlet (H8 at δ 3.04 ppm) in the ^{1}H NMR spectrum.
4. The OH signal is a 1-proton exchangeable signal (H7 at δ 6.28 ppm and this is consistent with the strong –OH stretch in the IR (3184 cm^{-1}).
5. The aromatic substitution pattern (substituents at positions 1,3 & 4) is established from the coupling constants measured from the expansion of the aromatic spin system.
6. The aromatic protons can be assigned by inspection. H2 (δ 7.24 ppm) has no large *ortho* couplings so appears as narrow doublet due to the *meta* coupling ($^{4}J_{H2\text{-}H6}$); H5 (δ 6.83 ppm) appears as a doublet with a relatively large splitting since it has only one large *ortho* coupling $^{3}J_{H5\text{-}H6}$) and the *para* coupling to H2 ($^{5}J_{H2\text{-}H5}$) is too small to be resolved; H6 (δ 6.94 ppm) appears as a doublet of doublets and has a large *ortho* coupling ($^{3}J_{H5\text{-}H6}$) and a medium *meta* coupling ($^{4}J_{H2\text{-}H5}$).

Proton	1H Chemical Shift (δ) in ppm
H2	7.24
H5	6.83
H6	6.94
H7	6.28
H8	3.04
H9	9.60

7. For a 1,3,4 trisubstituted benzene, there are 6 possible isomers.

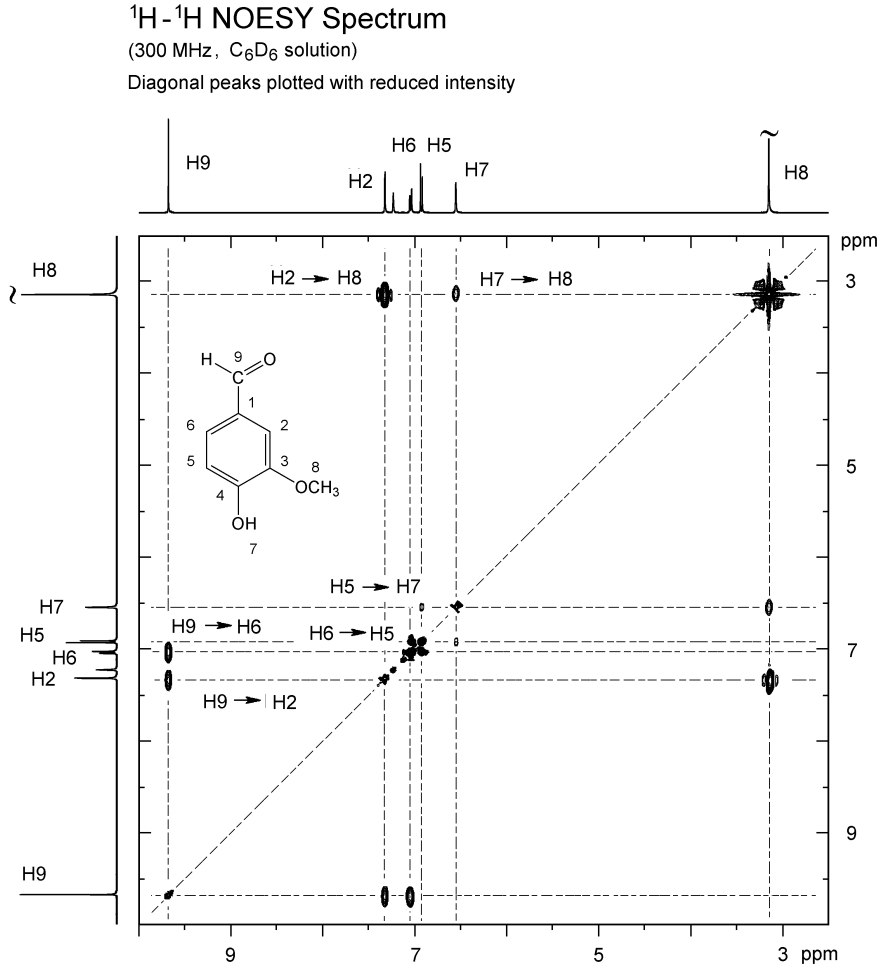


8. NOESY spectra show cross-peaks (off-diagonal peaks) at positions where a proton whose resonance appears on the F2 axis is close in space to another whose resonance appears on the F1 axis.

9. Note also that the NOESY spectrum is symmetrical, so only one section (either above the diagonal or below the diagonal) needs to be analysed.

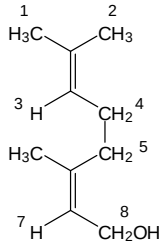
10. The NOESY spectrum shows that the –CHO proton (H10) has 2 aromatic protons which are near neighbours and these must be *ortho* protons. This places the –CHO group at position 1 in the 1,3,4-substitution pattern and eliminates the isomers A, B, C & D.

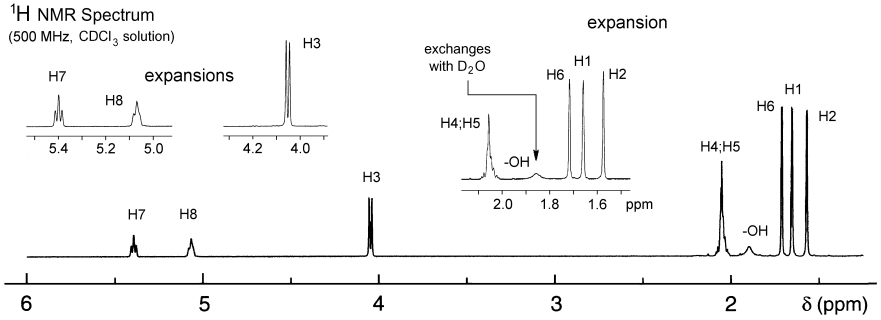
11. The $-OCH_3$ group has a strong cross peak to H2 and a weaker cross peak to H7 (the –OH proton) and this places the $-OCH_3$ substituent at position 3. By default the –OH substituent is at position 4 and <u>isomer F is the only isomer consistent with the spectral information</u>.



Problem 321

nerol

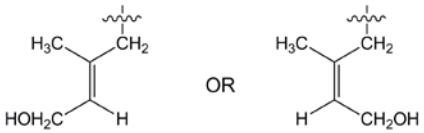




Proton	^{1}H Chemical Shift (δ) in ppm
H1	1.65
H2	1.57
H3	5.07
H4	2.04
H5	2.04
H6	1.71
H7	5.39
H8	4.04
-OH	1.90

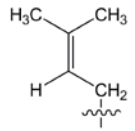
1. There are no aromatic protons – this is an aliphatic compound.
2. The ^{1}H NMR must account for the 18 protons in the molecular formula. There are 3 x 3-proton singlets at the high-field end of the spectrum and these must be 3 isolated methyl groups (*i.e.* with no large coupling from adjacent protons).
3. There is 1 exchangeable proton in the ^{1}H NMR and this must be an –OH resonance and this is confirmed by the IR spectrum (strong broad absorption at 3327 cm^{-1}).
4. There are 2 –C-H multiplets between 5 and 5.5 ppm in the proton spectrum and these are vinylic protons. Neither of the vinylic protons has a large splitting (>10Hz) so the vinylic protons are not *cis* or *trans* to each other on the same double bond.
5. There are four resonances at low field in the ^{13}C spectrum, confirming the presence of two alkene units. Two of the vinylic carbons are –CH groups from the DEPT and this is consistent with the vinylic region of the ^{1}H NMR spectrum. None of the vinylic carbons are =CH$_2$ groups. Given the fact that the vinylic protons show no strong coupling to each other, this means that there must be two independent trisubstituted double bonds.
6. There is a -CH$_2$- resonance (H8) at δ 4.05 ppm in the ^{1}H spectrum and a -CH$_2$- carbon at δ 58 ppm in the ^{13}C characteristic of a -CH$_2$OH unit.
7. The remaining two carbon atoms are aliphatic -CH$_2$- units (H4 & H5) whose proton resonances overlap near δ 2.04 ppm. This accounts for all of the proton and carbon resonances and carbon resonances.
8. In the COSY spectrum, the -CH$_2$O- protons (H8) are coupled to a vinyl proton (at δ 5.39 ppm), also, H8 appears as a doublet with a coupling of about 7 Hz and this is typical $^3J_{HH}$ for vinyl protons coupling to a "geminal" substituent – *i.e.* =C(H)(CH$_2$OH). The vinylic proton also appears as a broadened triplet, consistent with coupling the –CH$_2$- group (H8).

9. In the COSY spectrum, the vinyl proton at δ 5.4 ppm also shows coupling to one -CH_3 group (H6 at δ 1.71 ppm) and to one of the -CH_2- substituents (H5 at δ 2.04 ppm). These are both long-range couplings, too small to be resolved readily in the 1D proton NMR. The substituents on one of the double bonds are established as -CH_3, -CH_2-, H and –CH_2-OH and one double bond must be:

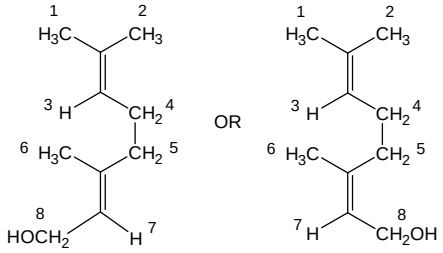


10. In the COSY spectrum, the vinyl proton at δ 5.05 ppm couples to a -CH_2- (H4 at δ 2.04 ppm) and two singlet -CH_3 resonances. If either methyl group were in a position *gem* to the vinyl proton, it would exhibit spin coupling of the order of 7 Hz, however in this case, all methyl resonances appear as (at best) broadened singlets.

11. The second trisubstituted double bond must be:



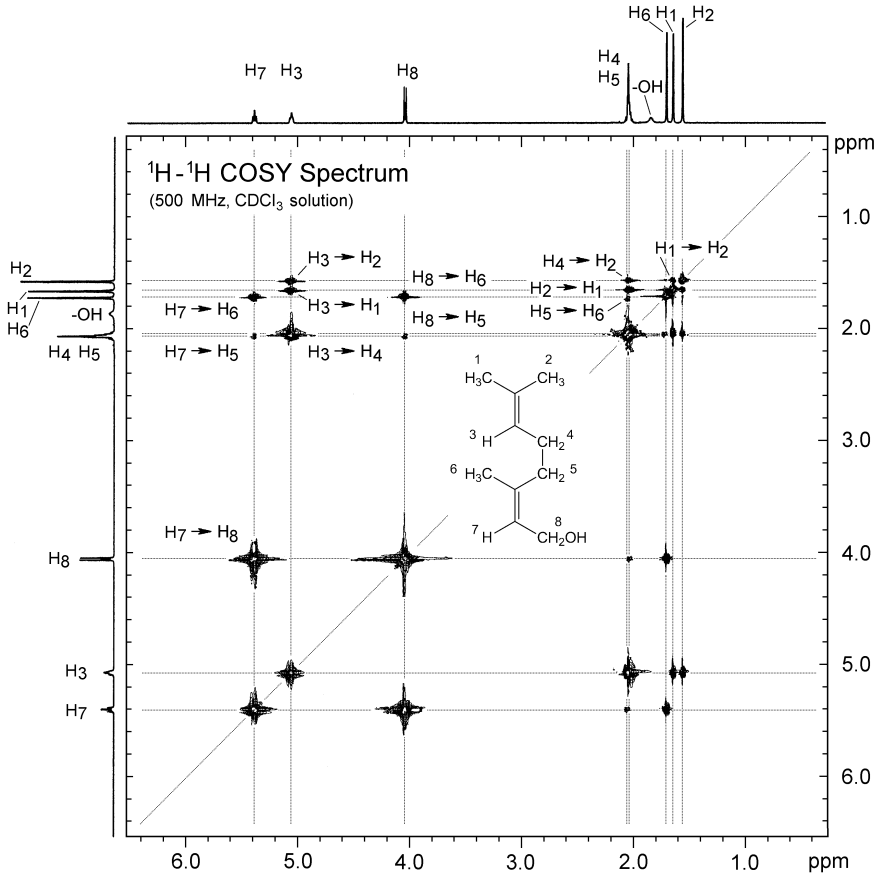
12. Joining the parts gives two possible isomers:

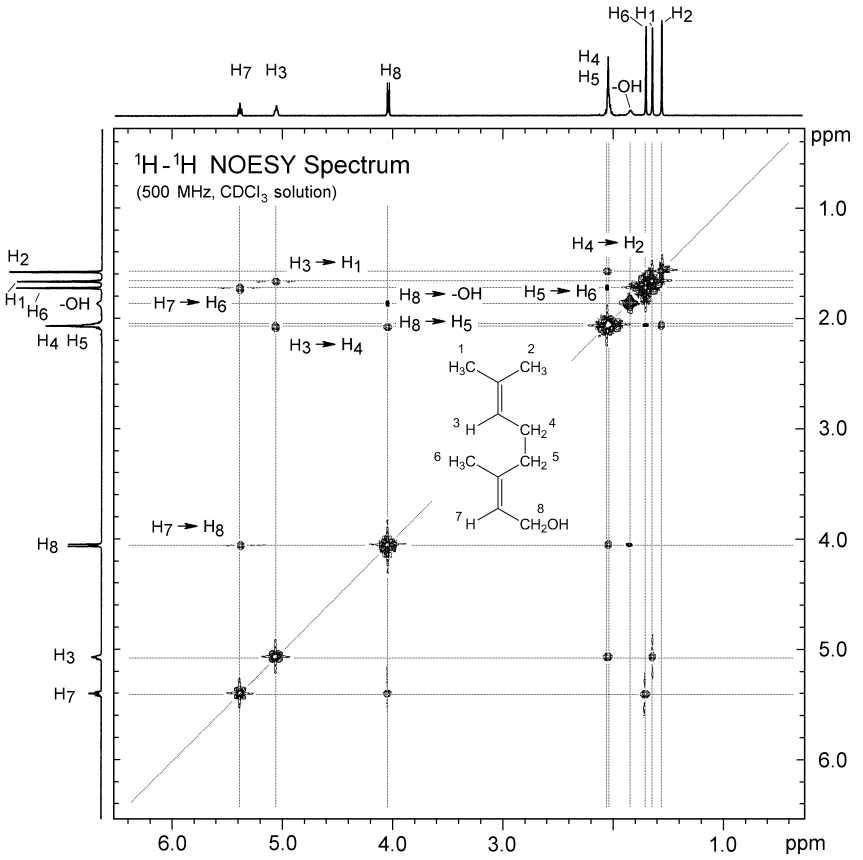


Isomer A Isomer B

13. In the ^{1}H-^{1}H NOESY spectrum, there is a correlation between H8 (δ 4.04 ppm) and the –CH_2- group H5 (δ 2.04 ppm). There is also a correlation between the vinyl proton H7 (δ 5.39 ppm) and the methyl group H6 (δ 1.71 ppm) so this identifies the compound as Isomer B (nerol).

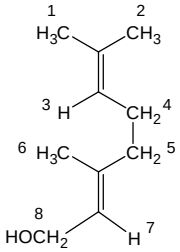
14. Other correlations in both the COSY spectrum and the NOESY spectrum are consistent with this structure.





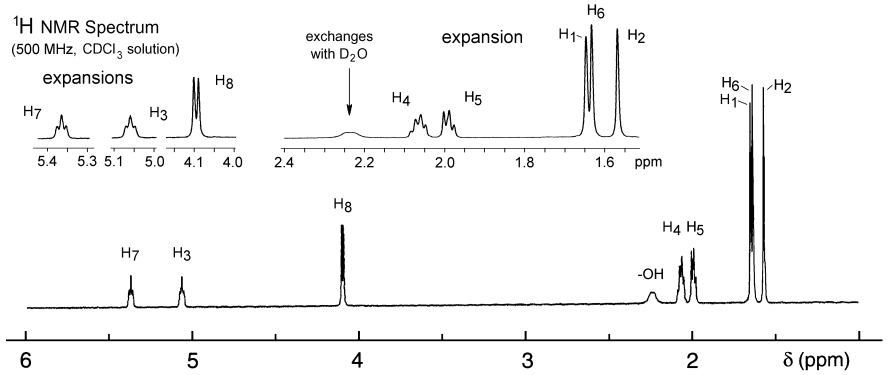
Problem 322

geraniol



This problem is very similar to Problem 323.

1. There are no aromatic protons – this is an aliphatic compound.
2. The ^{1}H NMR must account for the 18 protons in the molecular formula. There are 3 x 3-proton singlets at the high-field end of the spectrum and these must be 3 isolated methyl groups (*i.e.* with no large coupling from adjacent protons).
3. There is 1 exchangeable proton in the ^{1}H NMR and this must be an –OH resonance and this is confirmed by the IR spectrum (strong broad absorption at 3330 cm^{-1}).

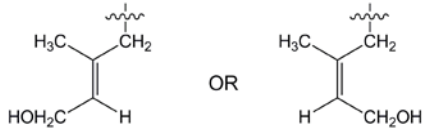


Proton	1H Chemical Shift (δ) in ppm
H1	1.64
H2	1.56
H3	5.06
H4	2.05
H5	1.98
H6	1.63
H7	5.36
H8	4.09
-OH	2.17

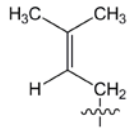
4. There are 2 –C-H multiplets between 5 and 5.5 ppm in the proton spectrum and these are vinylic protons (H3 and H7). Neither of the vinylic protons has a large splitting (>10Hz) so the vinylic protons are not *cis* or *trans* to each other on the same double bond.
5. There are four resonances at low field in the ^{13}C spectrum, confirming the presence of two alkene units. Two of the vinylic carbons are –CH groups from the DEPT and this is consistent with the vinylic region of the ^{1}H NMR spectrum. None of the vinylic carbons are =CH_2 groups. Given the fact that the vinylic protons show no strong coupling to each other, this means that there must be two independent trisubstituted double bonds.
6. There is a -CH_2- resonance (H8) at δ 4.09 ppm in the ^{1}H spectrum and a -CH_2- carbon at approximately δ 59 ppm in the ^{13}C characteristic of a -CH_2OH unit.
7. The remaining two carbon atoms are aliphatic -CH_2- units (H4 at δ 2.05 ppm & H5 at δ 1.98 ppm). This accounts for all of the proton and carbon resonances and carbon resonances.

8. In the COSY spectrum, the -CH_2O- protons (H8) are coupled to a vinyl proton (H7 at δ 5.36 ppm), also, H8 appears as a doublet with a coupling of about 7 Hz and this is typical $^3J_{HH}$ for vinyl protons coupling to a "geminal" substituent – *i.e.* =C(H)(CH_2OH). The vinylic proton also appears as a broadened triplet, consistent with coupling the –CH_2- group (H8).

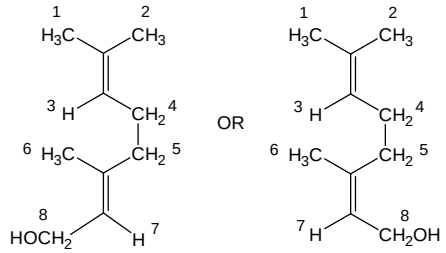
9. In the COSY spectrum, the vinyl proton at δ 5.36 ppm (H7) also couples to one -CH_3 (H6 at δ 1.63 pm) and a -CH_2- group (H5 at δ 1.98 ppm). These are both long-range couplings, too small to be resolved readily in the 1D proton NMR. The substituents on one of the double bonds are established as –CH_3, -CH_2-, H and –CH_2-OH and one double bond must be:



10. In the COSY spectrum, the vinyl proton at δ 5.06 ppm couples to a -CH_2- (H4 at δ 2.05 ppm) and two singlet -CH_3 resonances. If either methyl group were in a position *gem* to the vinyl proton, it would exhibit spin coupling of the order of 7 Hz, however in this case, all methyl resonances appear as (at best) broadened singlets. The second trisubstituted double bond must be:



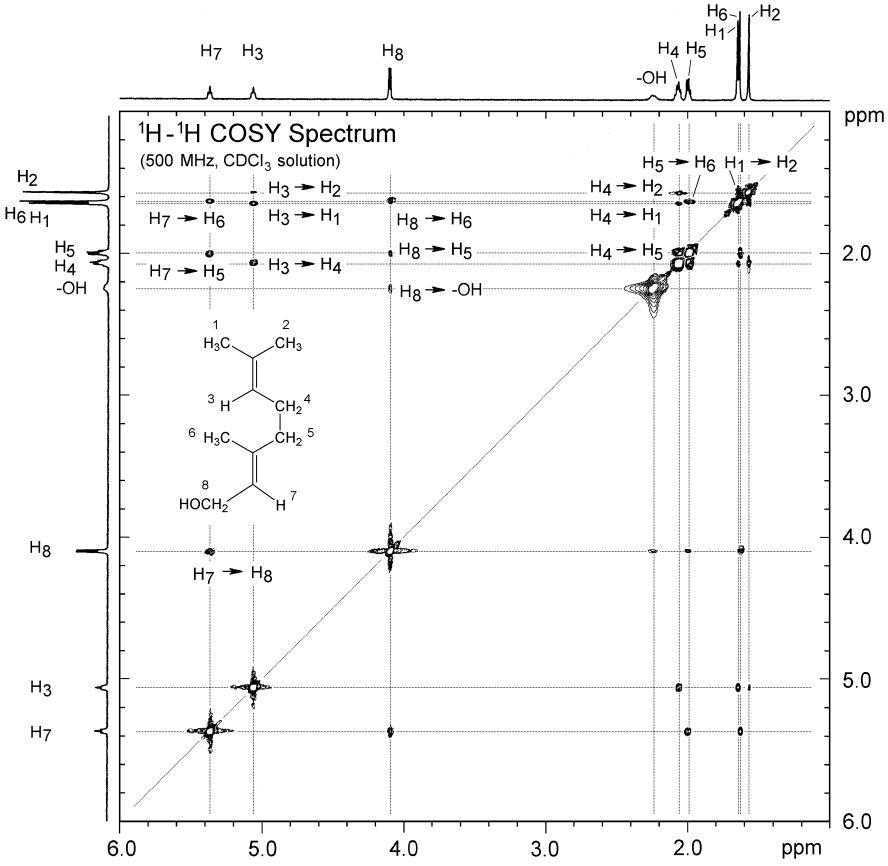
11. Joining the parts gives two possible isomers:

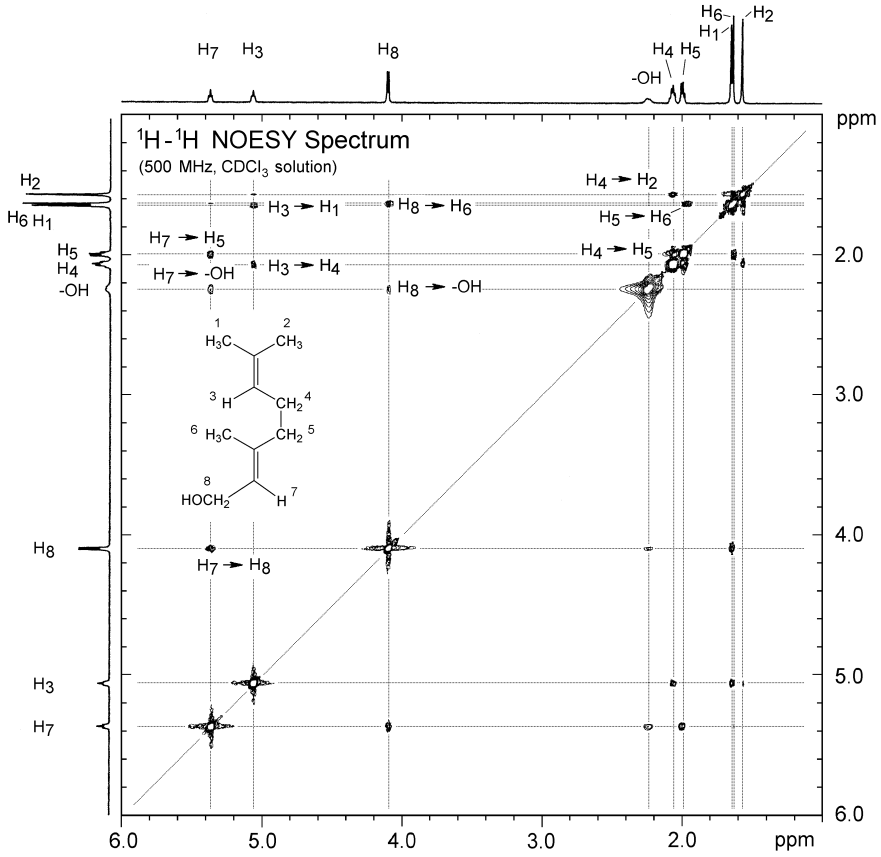


Isomer A Isomer B

15. In the ^{1}H-^{1}H NOESY spectrum, there is a correlation between the -CH2- group (H8 δ 4.09 ppm) and the –CH_3 group (H6 at δ 1.63 ppm). There is also a correlation between the vinyl proton H7 (δ 5.36 ppm) and the –CH_2- group H5 (δ 1.98 ppm) so this identifies the compound the compound as Isomer A (geraniol).

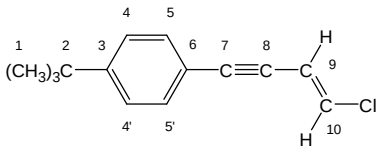
16. Other correlations in both the COSY spectrum and the NOESY spectrum are consistent with this structure.



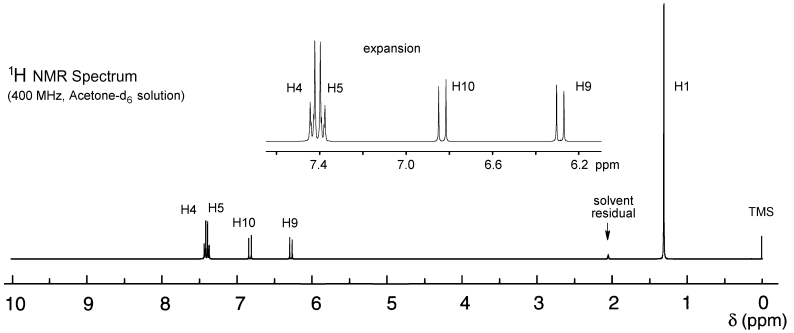


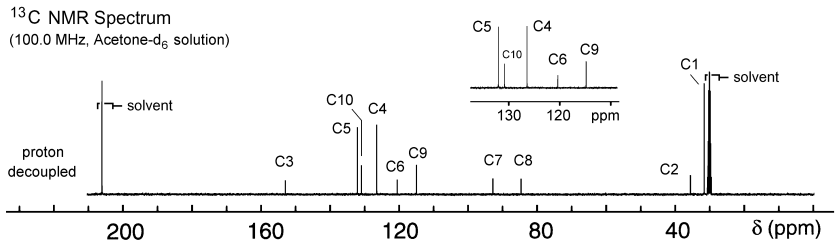
Problem 323

E-1-chloro-4-(4-*t*-butylphenyl)but-1-en-3-yne



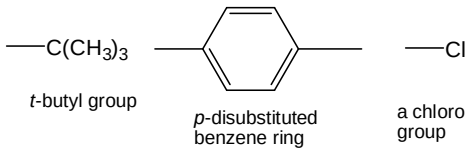
1. There are 4 aromatic protons and the symmetrical pattern in the expansion of the aromatic region is characteristic of a *p*-disubstituted benzene.
2. There are two protons in the vinylic region between 6 and 7 ppm. These are clearly coupled to each other and the coupling is about 14 Hz which is characteristic of protons which are *trans* to each other across a double bond.
3. The ^{1}H NMR spectrum also shows a strong 9-proton singlet at δ 1.31 ppm and this probably a *t*-butyl group.
4. This accounts for all of the protons in the spectrum.
5. The ^{13}C spectrum shows 10 resonances including 5 quaternary carbons (*i.e.* carbons with no attached protons). The DEPT spectrum shows that all of the protonated carbons are either C-H or -CH_3 groups.
6. The DEPT spectrum also shows a strong -CH_3 group (at δ 31.3 ppm) and a quaternary carbon (at δ 35.4 ppm) in the aliphatic region, consistent with the presence of a *t*-butyl group. Two of the quaternary carbons in the aromatic region must belong to the substituted carbons in the aromatic ring.
7. The IR spectrum shows a peak at 2204 cm^{-1} and apart from the chlorine, there are no heteroatoms in the structure. The compound must contain a C≡C group. There are two quaternary carbons between 80 and 100 ppm and these would be consistent with a non-terminal C≡C group.





Proton	^{1}H Chemical Shift (δ) in ppm	Carbon	^{13}C Chemical Shift (δ) in ppm
H1	1.31	C1	31.3
		C2	35.4
		C3	152.9
H4	7.44	C4	126.4
H5	7.39	C5	132.0
		C6	120.4
		C7	92.7
		C8	84.5
H9	6.28	C9	114.8
H10	6.83	C10	130.8

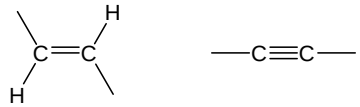
8. So we can identify a number of fragments:



t-butyl group

p-disubstituted benzene ring

a chloro group

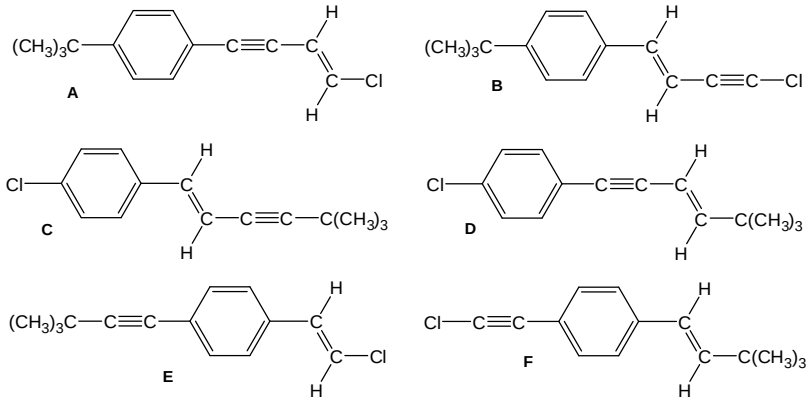


a *trans*-disubstuted vinyl group

a non-terminal alkyne

9. Taken together, these satisfy the molecular formula without any additional functional groups.

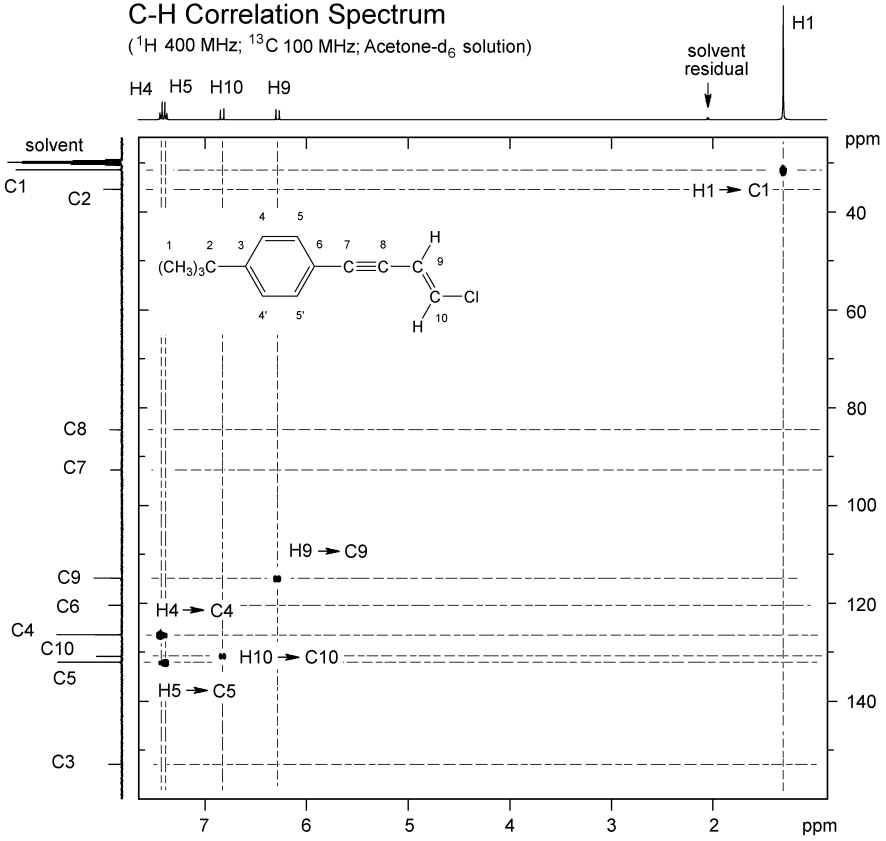
10. There are 6 possible isomers that incorporate all of these fragments:



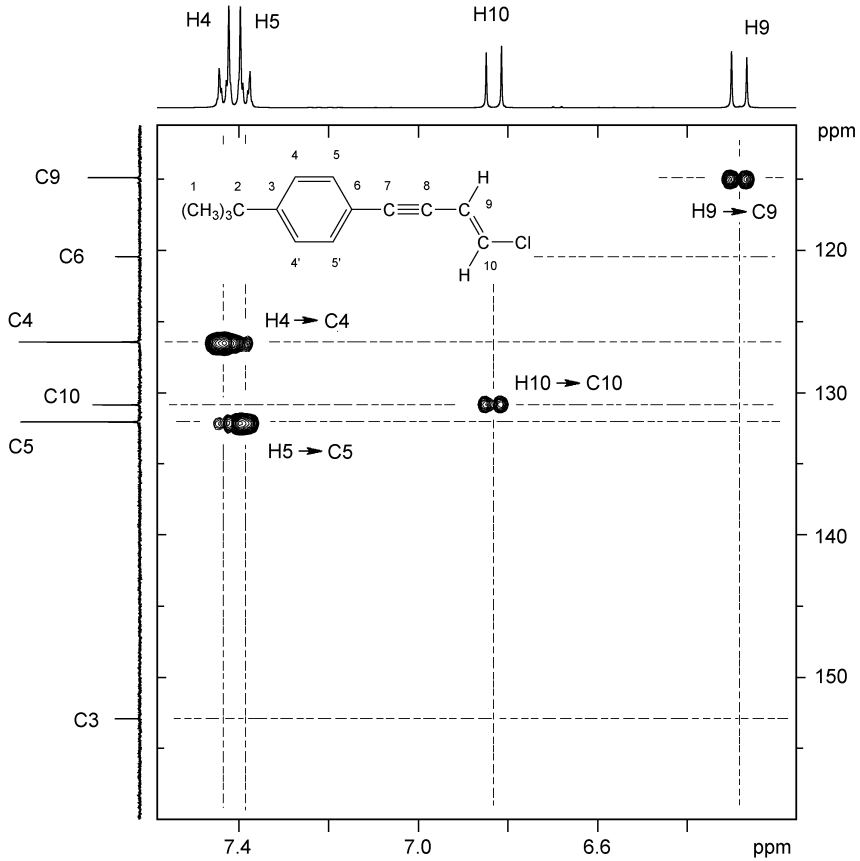
11. The fragmentation pattern in the mass spectrum shows no strong fragments which would distinguish these possible isomers.

12. The C-H Correlation Spectrum identifies which aromatic protons (δ 7.44 and 7.39 ppm) correlate to the aromatic carbons (δ 7.44 and 7.39 ppm (δ 126.4 and 132.0 ppm) respectively, but it is not possible to determine which proton is H4 and which is H5.

13. Likewise the C-H Correlation Spectrum identifies which vinylic protons (δ 6.28 and 6.83 ppm) correlate to the vinylic carbons (δ 114.8 and 130.8 ppm) respectively, but it is not possible to determine which proton is H9 and which is H10.



C-H Correlation Spectrum (expansion)
(^{1}H 400 MHz; ^{13}C 100 MHz; Acetone-d_6 solution)



14. The HMBC spectrum shows a clear correlation between the protons of the *t*-butyl group and one of the aromatic carbons (H1 correlates to C3) and one of the aromatic protons correlates to the quaternary carbon of the *t*-butyl group (H5 correlates to C7). The *t*-butyl group must be directly attached to the aromatic ring and this immediately eliminates isomers **C**, **D**, **E** and **F**.

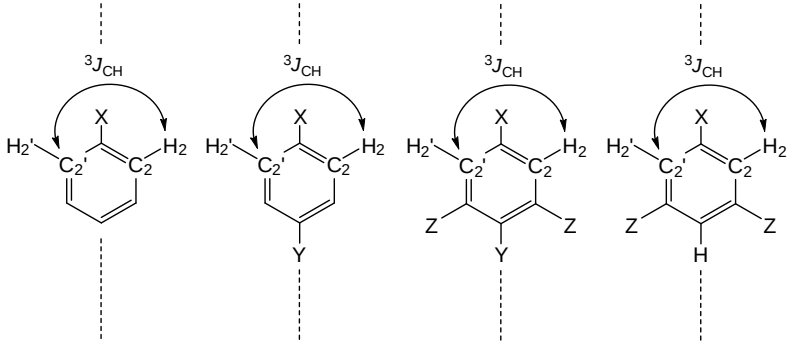
15. Likewise one of the aromatic protons correlates to one of the alkyne carbons (H5 – C7) and this indicates that the alkyne is the other substituent on the aromatic ring. Isomer **A** is the only isomer consistent with the HMBC.

16. The other correlations in the HMBC are consistent with this structure.

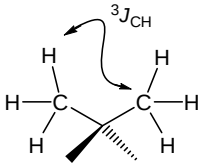
17. **Remember** that, in aromatic systems, the 3-bond coupling $^3J_{H\text{-}C}$ is typically the larger long-range coupling and gives rise to the strongest cross peaks.

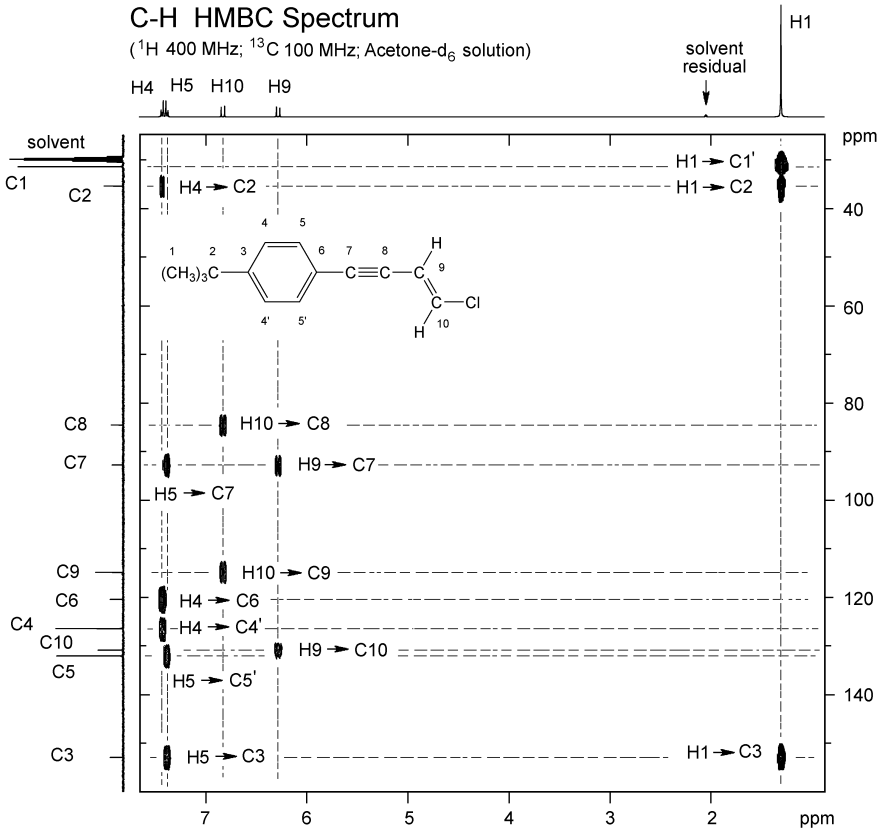
18. Likewise in conjugated non-aromatic systems, the 3-bond coupling $^3J_{H\text{-}C}$ is typically a strong long-range coupling and there is a strong correlation between H10 and C8 and also between H9 and C7 consistent with Isomer **A** as the correct structure.

19. Note also that in the HMBC for this compound, there are strong correlations between H4 and C4' and between H5 and C5'. While these appear to be 1-bond correlations, in *para*-disubstituted benzenes and in monosubstituted benzenes, or in 1,3,5- or 1,3,4,5-tetrasubstituted benzenes where there is a mirror plane of symmetry through the aromatic ring, these apparent 1-bond correlations arise from the $^3J_{H\text{-}C}$ interaction (H2 → C2') of a proton with the carbon which is *meta* to it.

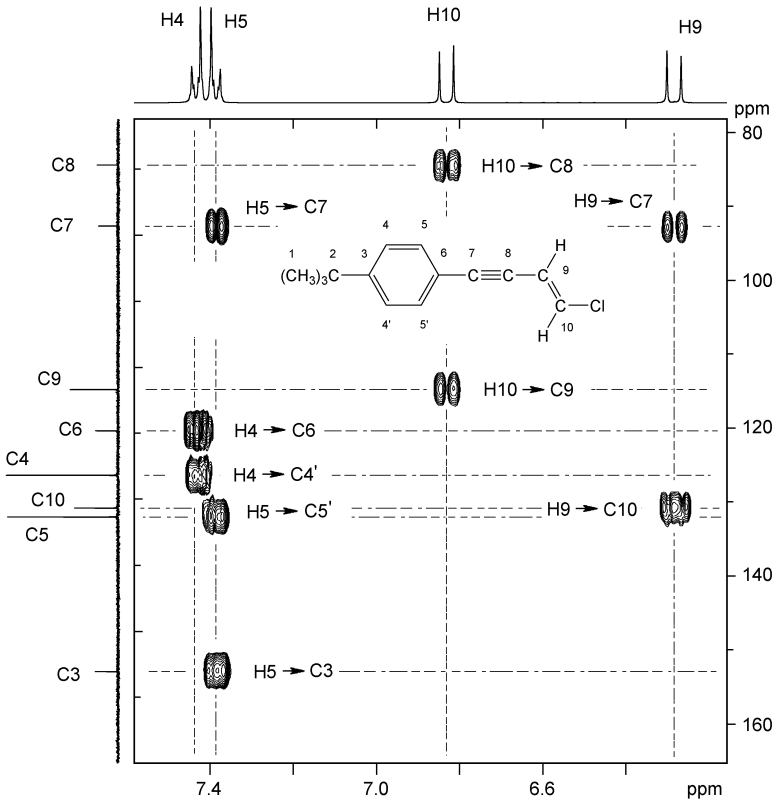


20. Note also that in the HMBC for this compound, there is a strong correlation between H1 and C1'. Again while this appears to be a 1-bond correlation, in *t*-butyl groups, isopropyl groups or in compounds with a *gem*-dimethyl group, the apparent 1-bond correlation arises from the $^3J_{H\text{-}C}$ interaction of the protons of one of the methyl groups with the chemically equivalent carbon which is 3 bonds away.



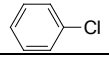
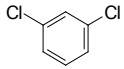
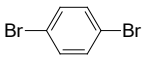
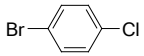
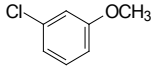
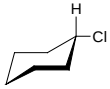


C-H HMBC Spectrum (expansion)
(^{1}H 400 MHz; ^{13}C 100 MHz; Acetone-d_6 solution)

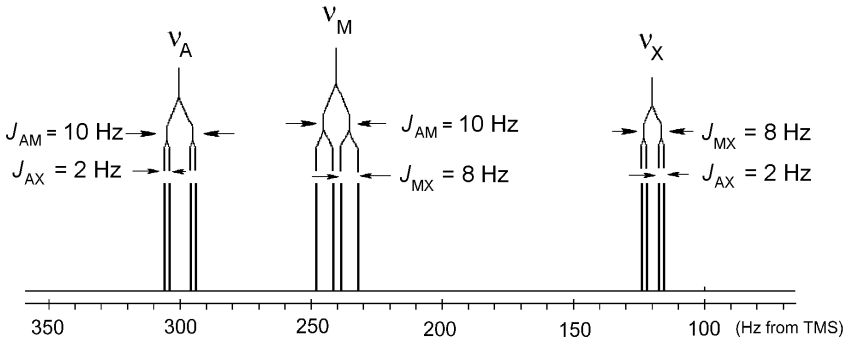


Chapter 9.4 – Analysis of NMR Spectra

Problem 324

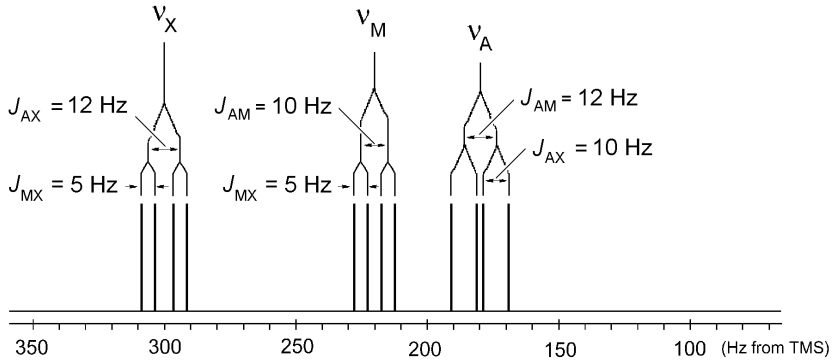
Structure		Number of ^{1}H environments	Number of ^{13}C environments
$CH_3-CO-CH_2CH_2CH_3$		4	5
$CH_3CH_2-CO-CH_2CH_3$		2	3
$CH_2=CHCH_2CH_3$		5	4
cis- $CH_3CH=CHCH_3$		2	2
trans- $CH_3CH=CHCH_3$		2	2
		1	1
		3	4
		2	3
		3	4
		1	2
		2	4
		5	7
	**slow chair-chair**	2	1
	**fast chair-chair**	1	1
	***rigid***	7	4

Problem 325



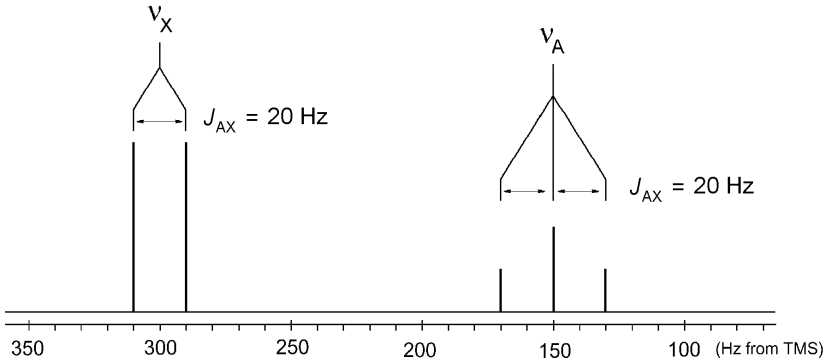
Chemical Shifts δ_A = 300 Hz / 60 MHz = 5.0 ppm
δ_M = 240 Hz / 60 MHz = 4.0 ppm
δ_X = 120 Hz / 60 MHz = 2.0 ppm

Problem 326



Chemical Shifts δ_A = 180 Hz / 200 MHz = 0.90 ppm
δ_M = 220 Hz / 200 MHz = 1.10 ppm
δ_X = 300 Hz / 200 MHz = 1.50 ppm

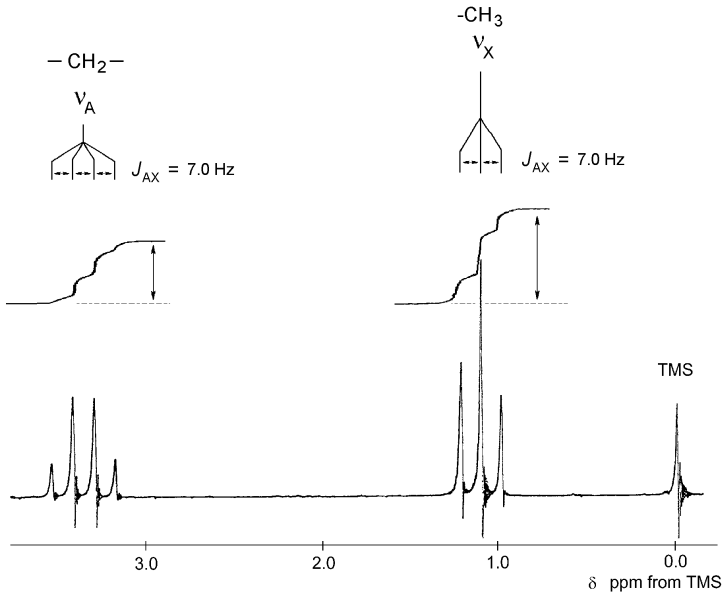
Problem 327



Chemical Shifts δ_A = 150 Hz / 400 MHz = 0.375 ppm
δ_X = 300 Hz / 400 MHz = 0.750 ppm

Problem 328

Spin System A_2X_3



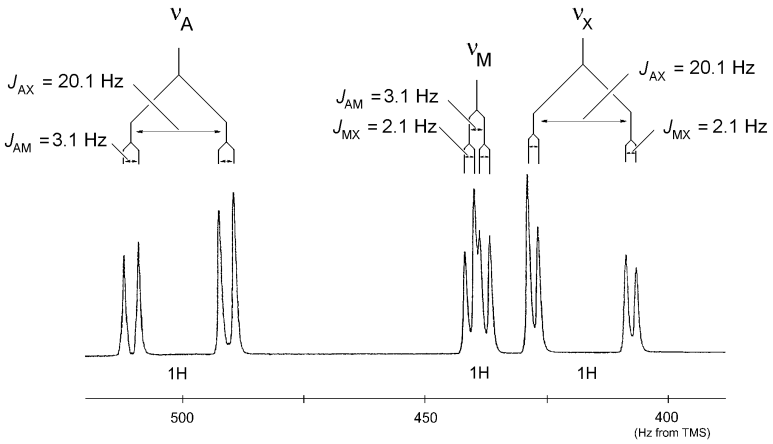
Chemical Shifts δ_A = 3.36 ppm = 3.36 x 60 = 202 Hz from TMS
δ_X = 1.11 ppm = 1.11 x 60 = 67 Hz from TMS

1st Order Analysis $\Delta\nu_{AX} = \nu_A - \nu_X$ = 202 - 67 = 135 Hz

$\Delta\nu_{AX} / J_{AX}$ = 135 / 7 = 19.3

This ratio is much greater than 3 so a 1st order analysis is justified.

Problem 329 **Spin System** AMX



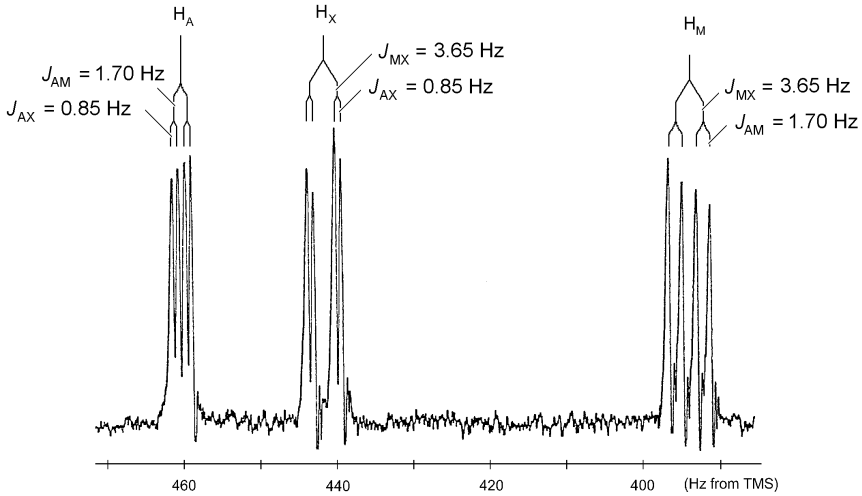
Chemical Shifts δ_A = 501 Hz / 100 MHz = 5.01 ppm
δ_M = 439 Hz / 100 MHz = 4.39 ppm
δ_X = 408 Hz / 100 MHz = 4.08 ppm

Coupling constants J_{AM} = 3.1 Hz
J_{AX} = 20.1 Hz
J_{MX} = 1.1 Hz

1st Order Analysis $\Delta\nu_{AM} = \nu_A - \nu_M$ = 501 - 439 = 62 Hz
$\Delta\nu_{AX} = \nu_A - \nu_X$ = 501 - 408 = 93 Hz
$\Delta\nu_{MX} = \nu_M - \nu_X$ = 439 - 408 = 31Hz
$\Delta\nu_{AM} / J_{AM}$ = 62 / 3.1 = 20.0
$\Delta\nu_{AX} / J_{AX}$ = 93 / 20.1 = 4.6
$\Delta\nu_{MX} / J_{MX}$ = 31 / 2.1 = 14.7

All ratios are greater than 3 so a 1st order analysis is justified.

Problem 330 **Spin System** AMX



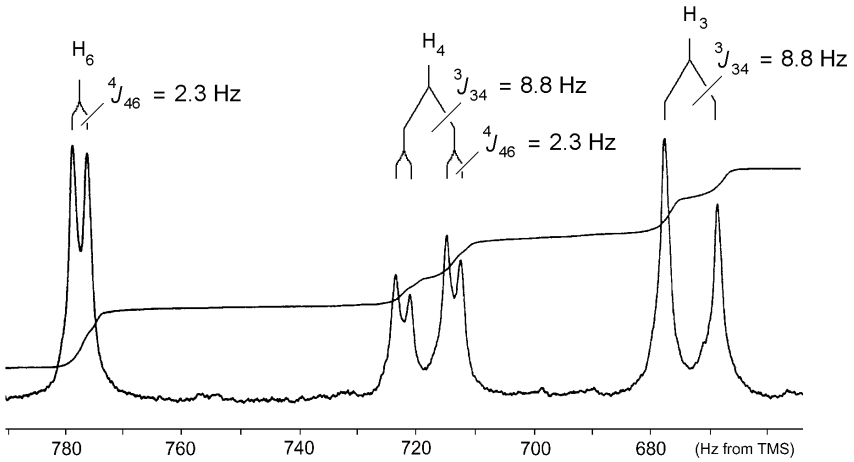
Chemical Shifts δ_A = 460 Hz / 60 MHz = 7.67 ppm
δ_X = 442 Hz / 60 MHz = 7.37 ppm
δ_M = 394 Hz / 60 MHz = 6.57ppm

Coupling constants J_{AM} = 1.70 Hz
J_{AX} = 0.85 Hz
J_{MX} = 3.65 Hz

1st Order Analysis $\Delta\nu_{AM} = \nu_A - \nu_M$ = 460 - 394 = 66 Hz
$\Delta\nu_{AX} = \nu_A - \nu_X$ = 460 - 442 = 18 Hz
$\Delta\nu_{MX} = \nu_M - \nu_X$ = 442 - 394 = 48 Hz
$\Delta\nu_{AM} / J_{AM}$ = 66 / 1.7 = 38.8
$\Delta\nu_{AX} / J_{AX}$ = 18 / 0.85 = 21.2
$\Delta\nu_{MX} / J_{MX}$ = 48 / 3.65 = 13.2

All ratios are greater than 3 so a 1st order analysis is justified.

Problem 331 **Spin System** AMX



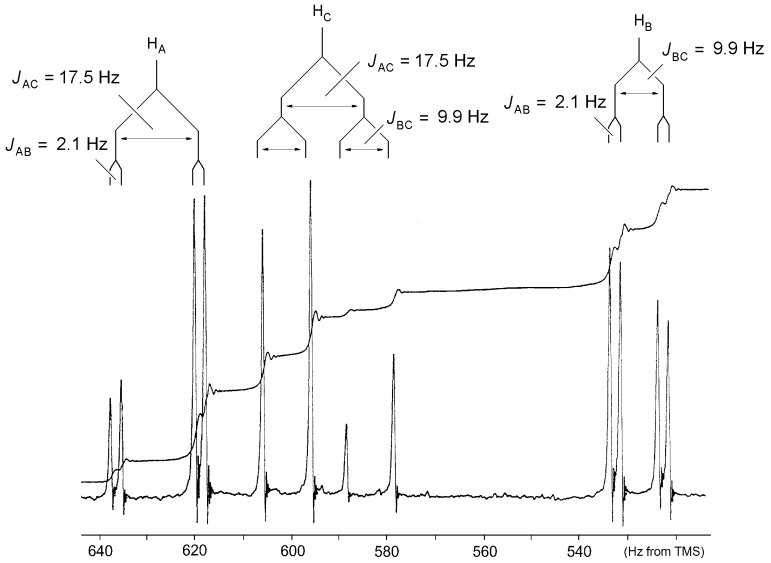
Chemical Shifts δ_3 = 673 Hz / 100 MHz = 6.73 ppm
δ_4 = 719 Hz / 100 MHz = 7.19 ppm
δ_6 = 777 Hz / 100 MHz = 7.77ppm

Coupling constants $^3J_{34}$ = 8.8 Hz
$^4J_{46}$ = 2.3 Hz
$^5J_{36}$ not resolved (< 1Hz)

1st Order Analysis $\Delta\nu_{34} = \nu_4 - \nu_3 = 719 - 673 = 46$ Hz
$\Delta\nu_{46} = \nu_6 - \nu_4 = 777 - 719 = 58$ Hz
$\Delta\nu_{36} = \nu_6 - \nu_3 = 777 - 673 = 104$ Hz
$\Delta\nu_{34} / J_{34} = 46 / 8.8 = 5.2$
$\Delta\nu_{46} / J_{46} = 58 / 2.3 = 25.2$
$\Delta\nu_{36} / J_{36} = 104 / <1 = >104$

All ratios are greater than 3 so a 1st order analysis is justified.

Problem 332 **Spin System** AMX



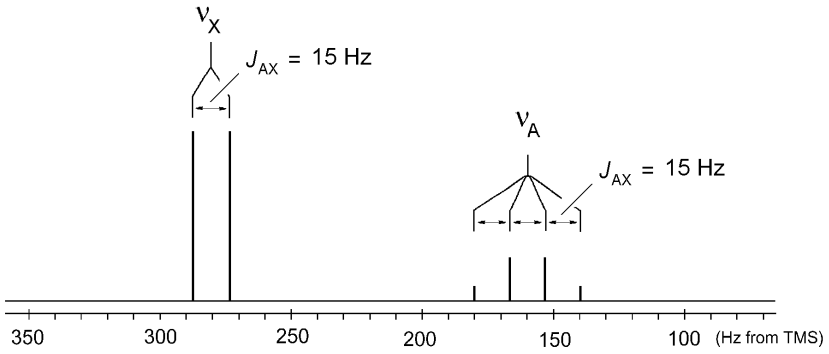
Chemical Shifts δ_A = 628 Hz / 100 MHz = 6.28 ppm
δ_B = 527 Hz / 100 MHz = 5.27 ppm
δ_C = 592 Hz / 100 MHz = 5.92 ppm

Coupling constants J_{AB} = 2.1 Hz; J_{AC} = 17.5 Hz; J_{BC} = 9.9 Hz

1st Order Analysis $\Delta\nu_{AB} = \nu_A - \nu_B = 628 - 527 = 101$ Hz
$\Delta\nu_{AC} = \nu_A - \nu_C = 628 - 592 = 36$ Hz
$\Delta\nu_{BC} = \nu_C - \nu_B = 592 - 527 = 65$ Hz
$\Delta\nu_{AB} / J_{AB} = 101 / 2.1 = 48.1$
$\Delta\nu_{AC} / J_{AC} = 36 / 17.5 = 2.1$
$\Delta\nu_{BC} / J_{BC} = 65 / 9.9 = 6.6$

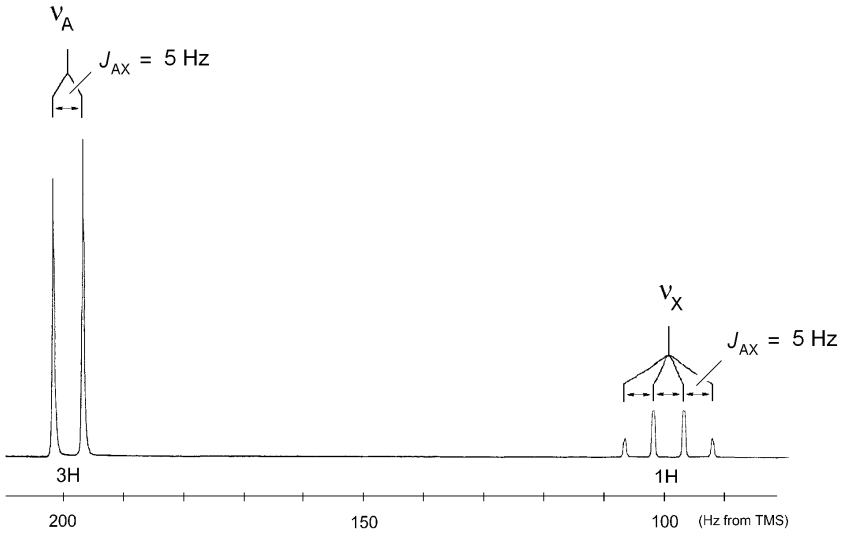
2 out of 3 ratios are greater than 3 so this is borderline 1st order. The main deviation from 1st order is that intensities are severely distorted - a 1st order spectrum would have all lines of equal intensity. J_{AC} = 17.5 Hz indicates that H_A and H_C must be *trans*. J_{BC} = 9.9 Hz indicates H_A and H_C are *cis*.

Problem 333 **Spin System** AX_3



Chemical Shifts δ_A = 160 Hz / 60 MHz = 2.67 ppm
δ_X = 280 Hz / 60 MHz = 4.67 ppm

Problem 334 **Spin System** AX_3



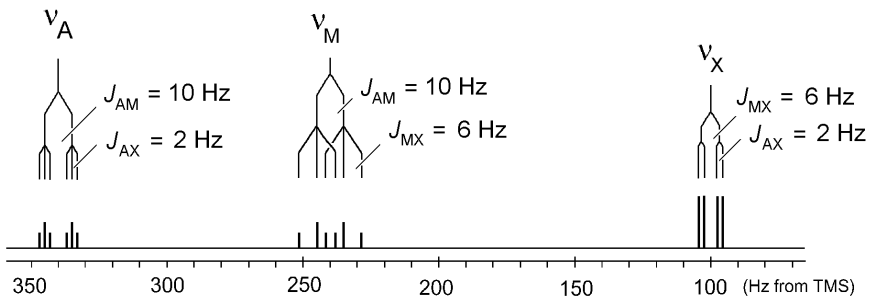
Chemical Shifts δ_A = 199 Hz / 100 MHz = 1.99 ppm
δ_X = 99 Hz / 100 MHz = 0.99 ppm

Coupling constants J_{AX} = 5 Hz

1st Order Analysis $\Delta\nu_{AX} = \nu_A - \nu_X$ = 199 - 99 = 100 Hz
$\Delta\nu_{AX} / J_{AX}$ = 100 / 5 = 20.0

$\Delta\nu_{AX} / J_{AX}$ is much greater than 3 so a 1st order analysis is justified.

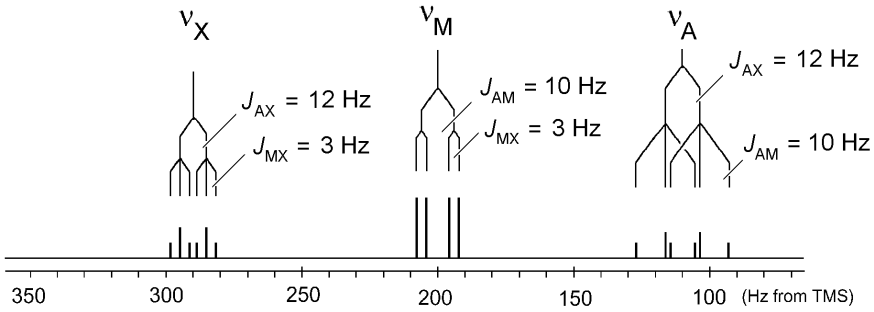
Problem 335 **4 Spin System** AMX_2



Chemical Shifts

δ_A = 340 Hz / 60 MHz = 5.67 ppm
δ_M = 240 Hz / 60 MHz = 4.00 ppm
δ_X = 100 Hz / 60 MHz = 1.67 ppm

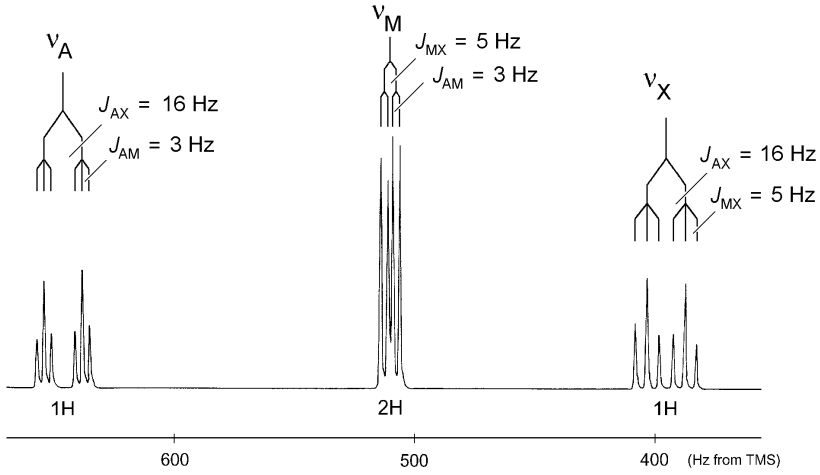
Problem 336 **4 Spin System** AM_2X



Chemical Shifts

δ_A = 110 Hz / 60 MHz = 1.83ppm
δ_M = 200 Hz / 60 MHz = 3.33 ppm
δ_X = 290 Hz / 60 MHz = 4.83 ppm

Problem 337 4 Spin System AM_2X



Chemical Shifts δ_A = 646 Hz / 100 MHz = 6.46 ppm
δ_M = 510 Hz / 100 MHz = 5.10 ppm
δ_X = 395 Hz / 100 MHz = 3.95 ppm

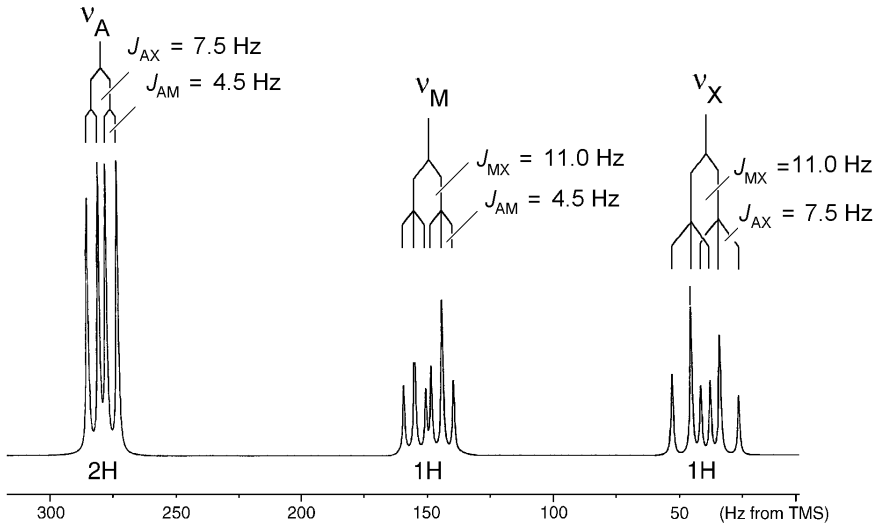
Coupling constants J_{AM} = 3 Hz; J_{AX} = 16 Hz; J_{MX} = 5 Hz;

1st Order Analysis $\Delta\nu_{AX} = \nu_A - \nu_X$ = 646 - 395 = 251 Hz
$\Delta\nu_{AM} = \nu_A - \nu_M$ = 646 - 510 = 136 Hz
$\Delta\nu_{MX} = \nu_M - \nu_X$ = 510 - 395 = 115 Hz

$\Delta\nu_{AX} / J_{AX}$ = 251 / 16 = 15.7
$\Delta\nu_{AM} / J_{AM}$ = 136 / 3 = 45.3
$\Delta\nu_{MX} / J_{MX}$ = 115 / 5 = 23.0

All ratios are significantly greater than 3 so a 1st order analysis is justified.

Problem 338 4 Spin System A_2MX



Chemical Shifts δ_A = 279 Hz / 100 MHz = 2.79 ppm
δ_M = 149 Hz / 100 MHz = 1.49 ppm
δ_X = 39 Hz / 100 MHz = 0.39 ppm

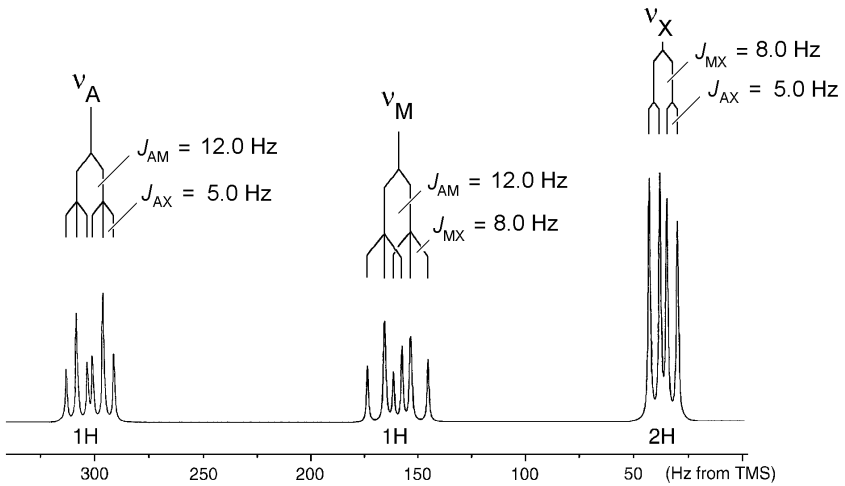
Coupling constants J_{AM} = 4.5 Hz; J_{AX} = 7.5 Hz; J_{MX} = 11.0 Hz;

1st Order Analysis $\Delta\nu_{AX} = \nu_A - \nu_X$ = 279 - 39 = 240 Hz
$\Delta\nu_{AM} = \nu_A - \nu_M$ = 279 - 149 = 130 Hz
$\Delta\nu_{MX} = \nu_M - \nu_X$ = 149 - 39 = 110 Hz

$\Delta\nu_{AX} / J_{AX}$ = 240 / 7.5 = 32.0
$\Delta\nu_{AM} / J_{AM}$ = 130 / 4.5 = 28.9
$\Delta\nu_{MX} / J_{MX}$ = 110 / 11 = 10.0

All ratios are significantly greater than 3 so a 1st order analysis is justified.

Problem 339 4 Spin System AMX_2



Chemical Shifts δ_A = 302 Hz / 100 MHz = 3.02 ppm
δ_M = 160 Hz / 100 MHz = 1.60 ppm
δ_X = 37 Hz / 100 MHz = 0.37 ppm

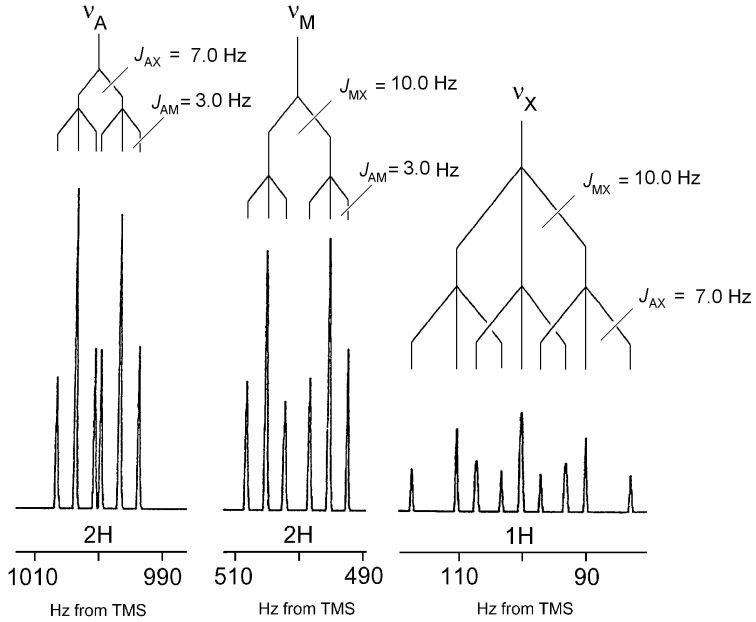
Coupling constants J_{AM} = 12.0 Hz; J_{AX} = 5.0 Hz; J_{MX} = 8.0 Hz;

1st Order Analysis $\Delta\nu_{AX} = \nu_A - \nu_X = 302 - 37 = 265$ Hz
$\Delta\nu_{AM} = \nu_A - \nu_M = 302 - 160 = 142$ Hz
$\Delta\nu_{MX} = \nu_M - \nu_X = 160 - 37 = 123$ Hz

$\Delta\nu_{AX} / J_{AX} = 265 / 5.0 = 53.0$
$\Delta\nu_{AM} / J_{AM} = 142 / 12.0 = 11.8$
$\Delta\nu_{MX} / J_{MX} = 123 / 8.0 = 15.4$

All ratios are significantly greater than 3 so a 1st order analysis is justified.

Problem 340 5 Spin System A_2M_2X



Chemical Shifts δ_A = 1000 Hz / 200 MHz = 5.0 ppm
δ_M = 500 Hz / 200 MHz = 2.5 ppm
δ_X = 100 Hz / 200 MHz = 0.5 ppm

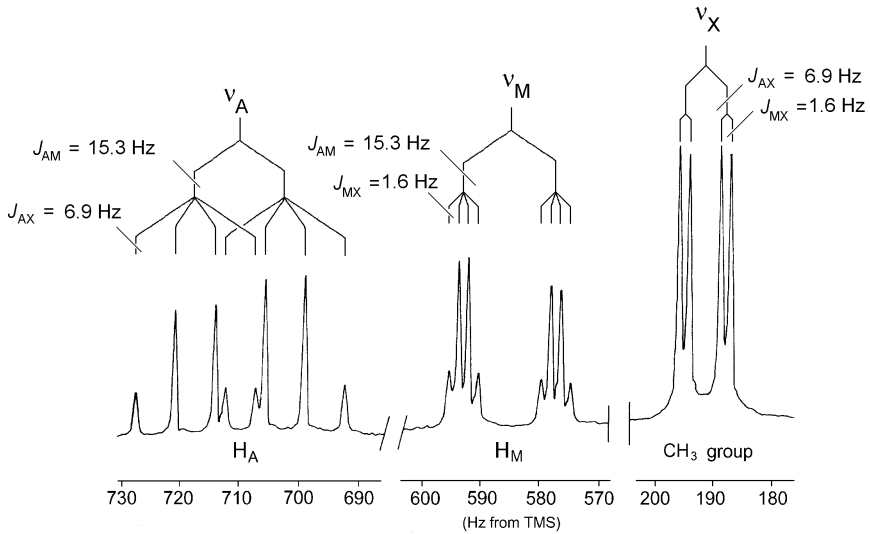
Coupling constants J_{AM} = 3.0 Hz; J_{AX} = 7.0 Hz; J_{MX} = 10.0 Hz;

1st Order Analysis $\Delta\nu_{AX} = \nu_A - \nu_X = 1000 - 100 = 900$ Hz
$\Delta\nu_{AM} = \nu_A - \nu_M = 1000 - 500 = 500$ Hz
$\Delta\nu_{MX} = \nu_M - \nu_X = 500 - 100 = 400$ Hz

$\Delta\nu_{AX} / J_{AX} = 900 / 7.0 = 128.6$
$\Delta\nu_{AM} / J_{AM} = 500 / 3.0 = 166.7$
$\Delta\nu_{MX} / J_{MX} = 400 / 10.0 = 40.0$

All ratios are significantly greater than 3 so a 1st order analysis is justified.

Problem 341 5 Spin System AMX_3



Chemical Shifts
δ_A = 710 Hz / 100 MHz = 7.10 ppm
δ_M = 585 Hz / 100 MHz = 5.85 ppm
δ_X = 192 Hz / 100 MHz = 1.92 ppm

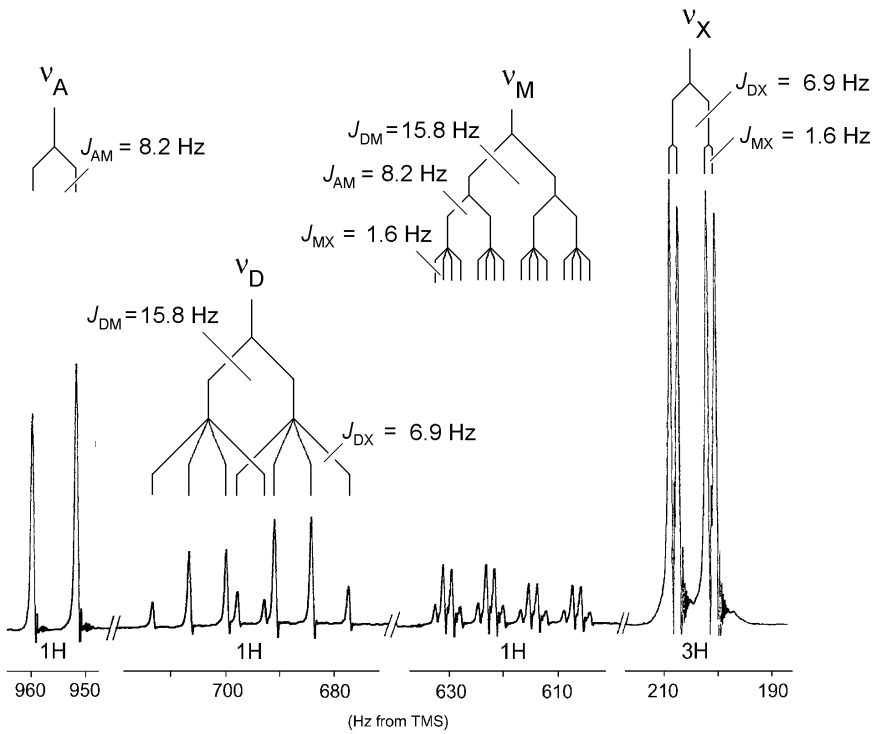
Coupling constants J_{AM} = 15.3 Hz; J_{AX} = 6.9 Hz; J_{MX} = 1.6 Hz;

1st Order Analysis
$\Delta\nu_{AX} = \nu_A - \nu_X$ = 710 - 192 = 518 Hz
$\Delta\nu_{AM} = \nu_A - \nu_M$ = 710 - 585 = 125 Hz
$\Delta\nu_{MX} = \nu_M - \nu_X$ = 585 - 192 = 393 Hz

$\Delta\nu_{AX} / J_{AX}$ = 518 / 6.9 = 84.7
$\Delta\nu_{AM} / J_{AM}$ = 125 / 15.3 = 8.2
$\Delta\nu_{MX} / J_{MX}$ = 393 / 1.6 = 245.6

All ratios are significantly greater than 3 so a 1st order analysis is justified. J_{AM} = 15.3 Hz is typical of a coupling between vinylic protons which are *trans* to each other (see Section 5.7)

Problem 342 5 Spin System $ADMX_3$



Chemical Shifts
δ_A = 956 Hz / 100 MHz = 9.56 ppm;
δ_D = 695 Hz / 100 MHz = 6.95 ppm;
δ_M = 619 Hz / 100 MHz = 6.19 ppm;
δ_X = 205 Hz / 100 MHz = 2.05 ppm;

Coupling constants J_{AD} = < 1 Hz; J_{AM} = 8.2 Hz; J_{AX} = < 1 Hz;
J_{DM} = 15.8 Hz; J_{DX} = 6.9 Hz; J_{MX} = 1.6Hz;

1st Order Analysis

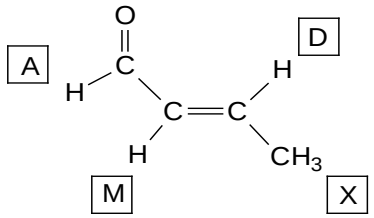
$\Delta\nu_{AD} = \nu_A - \nu_D = 956 - 695 = 261$ Hz
$\Delta\nu_{AM} = \nu_A - \nu_M = 956 - 619 = 337$ Hz
$\Delta\nu_{AX} = \nu_A - \nu_X = 956 - 205 = 751$ Hz
$\Delta\nu_{DM} = \nu_D - \nu_M = 695 - 619 = 76$ Hz
$\Delta\nu_{DX} = \nu_D - \nu_X = 695 - 205 = 490$ Hz
$\Delta\nu_{MX} = \nu_M - \nu_X = 619 - 205 = 414$ Hz

$\Delta\nu_{AD} / J_{AD} = 261 / <1 = >261$
$\Delta\nu_{AM} / J_{AM} = 337 / 8.2 = 41.1$
$\Delta\nu_{AX} / J_{AX} = 751 / <1 = >751$
$\Delta\nu_{DM} / J_{DM} = 76 / 15.8 = 4.8$
$\Delta\nu_{DX} / J_{DX} = 490 / 6.9 = 71.0$
$\Delta\nu_{MX} / J_{MX} = 414 / 1.6 = 258.8$

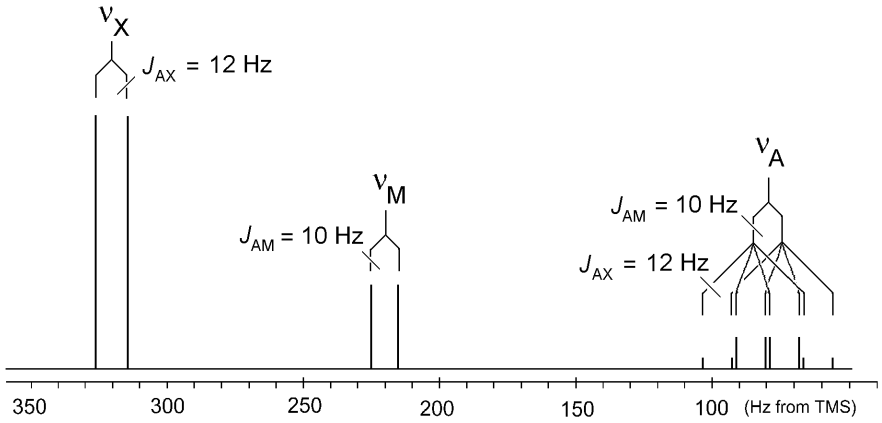
All ratios are significantly greater than 3 so a 1st order analysis is justified.

The critical coupling constant is $J_{DM} = 15.8$ Hz which is typical of a coupling between vinylic protons which are *trans* to each other (see Section 5.7).

The compound is:



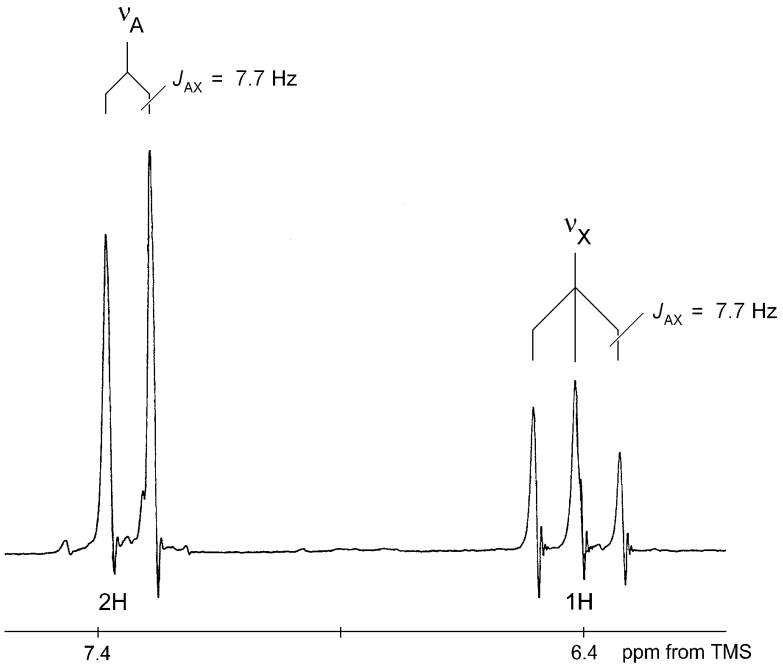
Problem 343 **5 Spin System** AMX_3



Chemical Shifts

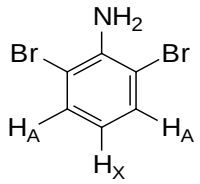
$\delta_A = 80$ Hz / 60 MHz = 1.33 ppm
$\delta_M = 220$ Hz / 60 MHz = 3.67 ppm
$\delta_X = 320$ Hz / 60 MHz = 5.33 ppm

Problem 344 **3 Spin System** A_2X



Of the 6 isomeric anilines, only compounds **4** and **6** have the correct symmetry to give a spectrum with only two chemical shifts in the aromatic region, in the ratio 2:1.

Both **4** and **6** would give A_2X spin systems. The measured coupling constant is 7.7 Hz which is in the range for protons which are *ortho* to each other. Compound **4** is the correct answer.



Problem 345

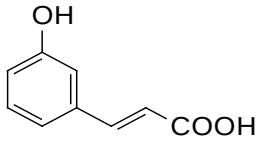
The spectrum is obtained after D_2O exchange so the carboxylic acid and phenolic protons will not be present and the spectrum only contains the aromatic and vinylic protons.

The spectrum shows 6 distinct resonances therefore compounds **5** and **6** can be eliminated because they would each have only 4 resonances (on symmetry grounds).

The proton at about δ 7.1 shows no large coupling (> 7 Hz), this means that it has no protons *ortho* to it. This eliminates compounds **1** and **2** since all protons in these compounds will have at least one large *ortho* coupling.

Compounds **3** and **4** differ by the stereochemistry at the double bond. The proton at δ 6.4 is clearly one of the vinylic protons and it is coupled to the other vinylic proton at δ 7.6. The coupling constant is 16 Hz and this is characteristic of vinylic protons which are *trans* to each other.

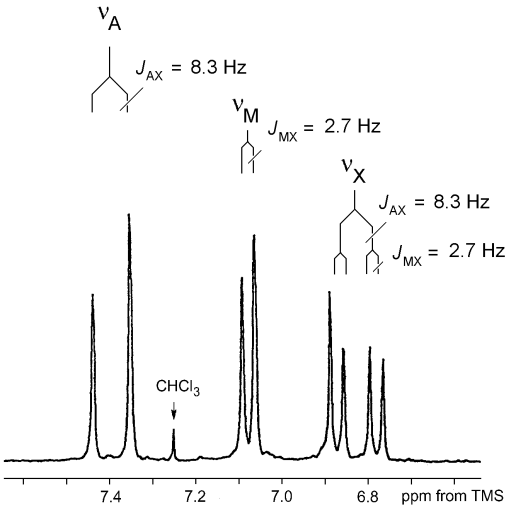
The correct answer is compound **3**.

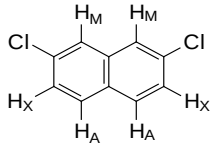


Problem 346

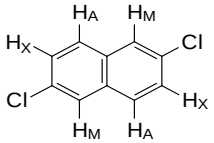
All of the protons in the ^{1}H spectrum 1,5-dichloronaphthalene have protons which are *ortho* to them. This means that every proton must have at least one large (>7 Hz) *ortho* coupling. The spectrum has one proton (at δ 7.1) which has only a small coupling so this cannot be the spectrum of 1,5-dichloronaphthalene.

The spectrum is an AMX spectrum with couplings between A and X of about 8.3 Hz (typical of an *ortho* coupling) and coupling between M and X of about 2.7 Hz (typical of a *meta* coupling). Two possible structures are given below.





2,7-dichloronaphthalene



2,6-dichloronaphthalene

Notes and Errata Edition 5

Problem 46 the DEPT spectrum has been incorrectly phased by exactly 180 degrees. The resonance near 52 ppm should be phased upwards (-CH_3) and the resonance near 29 ppm should be phased downwards (-CH_2-).

Problem 222 the molecular formula should be $C_{10}H_9NO_4Br_2$ instead of $C_8H_9NO_4Br_2$.

Problem 244 the scale on the high-field expansion should be 6.58 and 6.56 ppm rather than 7.58 and 7.56 ppm.

Editions 1-5: Cross-referenced Master list

Edition 5	Edition 4	Edition 3	Edition 2	Edition 1	Name	Formula
1	1	1	2	1	2-butanone	C4H8O
2	2		1		propionic acid	C3H6O2
3	3	2	3		ethyl acetate	C4H8O2
4	4	3			methyl propionate	C4H8O2
5	5	4	29		1,2-dibromoethane	C2H4Br2
6	6				2,3-butanedione (biacetyl)	C4H6O2
7	7	5			succinonitrile	C4H4N2
8	8	6	22		2,2,3,3-tetramethylbutane	C8H18
9					cyclopentane	C5H10
10	9	7			pinacol	C6H14O2
11	10	8			1,4-cyclohexanedione	C6H8O2
12	11	9			cyclopentanone	C5H8O
13					bromocyclopentane	C5H9Br
14	12	10	4	2	iodoethane	C2H5I
15	13	11	5		1,1-dichloroethane	C2H4Cl2
16	14	12	6		2-propanol	C3H8O
17	15	13	7		2-bromopropane	C3H7Br
18	16	14	58		1,4-dichlorobutane	C4H8Cl2
19	17	15			1,3-dibromopropane	C3H6Br2
20	18	16	59		1-bromo-3-chloropropane	C3H6ClBr
21	19				4-bromobutyronitrile	C4H6BrN
22	20	18			alanine	C3H7NO2
23	21	19			4-aminobutyric acid	C4H9NO2
24	22				anisole	C7H8O
25	23				benzyl alcohol	C7H8O
26	24	26	15	9	benzyl bromide	C7H7Br
27	25	27			benzyl cyanide	C8H7N
28	26				benzylamine	C7H9N
29	27	23	13	6	2-phenylethanol	C8H10O
30	28	24	14	7	1-phenylethanol	C8H10O
31	29	20	9	3	benzyl methyl ketone	C9H10O
32	30	21	10	4	phenyl ethyl ketone	C9H10O
33	31	104	61		2-phenylpropionaldehyde	C9H10O
34	32	22	11		butyrophenone	C10H12O
35	33				t-butyl acetoacetate	C8H14O3
36	34	25	12	5	ethyl formate	C3H6O2
37	35				benzil	C14H10O2
38	36			11	1,2-diphenylethane	C14H14
39	37	28			dibenzylamine	C14H15N
40	38	30			tetramethyl ethylenediamine	C6H16N2
41	39	31	27	12	2,5-hexanedione	C6H10O2
42	40	32	19		diethyl carbonate	C5H10O3
43	41	33	20		propionic anhydride	C6H10O3
44	42	34	24		diethyl oxalate	C6H10O4
45	43	35	28		ethylene glycol diacetate	C6H10O4
46	44				dimethyl succinate	C6H10O4
47	45	52	55	20	1,1-diacetoxyethane	C6H10O4
48	46	53	56		dimethyl methylmalonate	C6H10O4
49	47	67	73		methyl acetyllactate	C6H10O4
50	48	36	25	15	diethyl succinate	C8H14O4
51	49	37	26		ethylene glycol dipropionate	C8H14O4
52	50	38	21	17	butyric anhydride	C8H14O3
53	51	39	30	16	1,4-dibromobenzene	C6H4Br2
54	52	40	31		4,4'-dibromobiphenyl	C12H8Br2
55	53	41	18		a,a-dichlorotoluene	C7H6Cl2
56	54	42			benzaldehyde dimethylacetal	C9H12O2
57	55				benzilic acid	C14H12O3
58	56				catechol	C6H6O2
59	57	43			1,2,3-trimethoxybenzene	C9H12O3
60					1,3,5-triethynylbenzene	C12H6
61	58	44	32	14	mesitylene	C9H12
62	59	45			1,2,3-trimethylbenzene	C9H12

63	60	46			1,2,4-trimethylbenzene	C9H12
64	61	47	33	18	durene	C10H14
65	62	48	34		1,2,3,4-tetramethylbenzene	C10H14
66	63	49			1,2,3,5-tetramethylbenzene	C10H14
67	64	50	35		hexamethylbenzene	C12H18
68	65	51			1,2,3,4,5,-pentamethylcyclopentadiene	C10H16
69	66	54	17	8	acetamide	C2H5NO
70	67	55	69		ethyl glycolate	C4H8O3
71					methyl vinyl ketone	C4H6O
72	68		72	27	ethyl cyanoacetate	C5H7NO2
73	69		68		3-hydroxy-2-butanone (acetoin)	C4H8O2
74	70	56	70	26	4-hydroxy-4-methyl-2-pentanone	C6H12O2
75	71	175	113	57	isobutyl acetate	C6H12O2
76	72				3,3-dimethylbutyric acid	C6H12O2
77	73		78		2-methyl-2-butanol (t-amyl alcohol)	C5H12O
78	74	64	80	33	hexylamine	C6H15N
79	75	57	71		ethyl 2-bromopropionate	C5H9BrO2
80	76	58	76	28	4,4,-dimethoxy-2-butanone	C6H12O3
81	77	59	37		3,3-dimethylglutaric acid	C7H12O4
82	78	70	102	54	2,2-dimethylglutaric acid	C7H12O4
83	79	60	77	31	tetramethylurea	C5H12N2O
84	80	62			1,3-dioxane	C4H8O2
85	81	61			1,4-dioxane	C4H8O2
86	82	219			18-crown-6	C12H24O6
87	83	65	81	35	2,3-dichloropropene	C3H4Cl2
88	84	66	82	37	4-chlorobutyl acetate	C6H11ClO2
89	85	68	83	39	2-bromohexanoic acid	C6H11BrO2
90	86	69	91	48	2-ethylmalononitrile	C5H6N2
91	87	144	118	61	3-methylbutanenitrile	C5H9N
92	88	17			5-amino-1-pentyne	C5H9N
93	89	71	115	59	2-methylbut-3-en-2-ol	C5H10O
94	261	236	152	98	3-methylbutyraldehyde	C5H10O
95	90	72			threonine	C4H9NO3
96	91	73	16	13	1-bromo-3-phenylpropane	C9H11Br
97	92	74	43	22	1-nitropropane	C3H7NO2
98	93	75			dibutyl ether	C8H18O
99	94	76	44	29	butylbenzene	C10H14
100	95	77	45		t-butylbenzene	C10H14
101	96	78			sec-butylbenzene	C10H14
102					p-cymene	C10H14
103	97	79	46		neopentylbenzene	C11H16
104	98	80	47		4-(*n*-butyl)-α-chlorotoluene	C11H15Cl
105	99	107	74		4-methyl-4-phenyl-2-pentanone	C12H16O
106	100	81			p-bromoacetophenone	C8H7BrO
107	101	82	38	19	p-chloroacetophenone	C8H7ClO
108	102	83			p-toluyl chloride	C8H7OCl
109	103				p-anisic acid	C8H8O3
110	104				benzyl acetate	C9H10O2
111	105	84	50	38	4-methoxyacetophenone	C9H10O2
112	106	85			p-cresyl acetate	C9H10O2
113	107	86			methyl *p*-toluate	C9H10O2
114	108	87	42		p-methoxybenzyl alcohol	C8H10O2
115	109	88			4-methoxymethylphenol	C8H10O2
116	110	89	40		4-dimethylaminobenzonitrile	C9H10N2
117	111	90	41		p-bromo-*N,N*-dimethylaniline	C8H10BrN
118	112	91	39		p-anisyl *t*-butyl ketone	C12H16O2
119	113	92			4-*t*-butylphenyl acetate	C12H16O2
120	114	93			methyl 4-*t*-butylbenzoate	C12H16O2
121	115	94			p-nitroanisole	C7H7NO3
122	116	95	48	30	p-nitrobenzaldehyde	C7H5NO3
123	117	95			2-nitrobenzaldehyde	C7H5NO3
124	118	96			4-methoxybenzaldehyde	C8H8O2
125	119		49		4-nitrophenylacetylene	C8H5NO2
126	120	97			4-acetoxybenzoic acid	C9H8O4
127	121	98	51	40	ethyl *p*-aminobenzoate	C9H11NO2
128	122	99	52		p-ethoxybenzamide	C9H11NO2
129	123				4-aminoacetophenone	C8H9NO
130	124				4-methylacetanilide	C8H11NO
131	125	100	53		phenacetin	C10H13NO2
132	126	101			paracetamol	C8H9NO2
133	127	102	54		ethyl-*p*-ethoxybenzoate	C11H14O3
134	128	182	98	34	methyl (*p*-methoxyphenyl)propionate	C11H14O3

135	129	103	57	21	N-isopropylbenzylamine	C10H15N
136	130	105	62	23	methyl 2-methoxy-2-phenylacetate	C10H12O3
137	131	106	114	58	phenyl isocyanate	C7H5NO
138	132	108	75		phenylacetaldehyde dimethyl acetal	C10H14O2
139	133	109	63		hydroquinone dipropionate	C12H14O4
140	134	110	64	24	diethyl terephthalate	C12H14O4
141	135	111	65	25	diethyl *o*-phthalate	C12H14O4
142	136	113	66		diethyl isophthalate	C12H14O4
143	137	114			1,3-dihydroxyphenyl dipropionate	C12H14O4
144	138	112			dimethyl *o*-phthalate	C10H10O4
145	139	115			cycloheptanone	C7H12O
146	140	116			cycloheptatriene	C7H8
147	141	117			cyclopropyl methyl ketone	C5H8O
148	142	118			cyclopropane carboxylic acid	C4H6O2
149	143	119		86	cyclopropyl phenyl ketone	C10H10O
150	144	120			ethyl cyclobutanecarboxylate	C7H12O2
151	145	121			4-*t*-butylcyclohexanone	C10H18O
152	146	122	60		N-methylacetamide	C3H7NO
153	148	124			1,5-diaminopentane	C5H14N2
154					2,2,2-trifluoroethanol	C2H3F3O
155					benzyl toluate	C15H14O2
156					4-methlybenzyl benzoate	C15H14O2
157	150	126	85	42	(*p*-cresyl)methyl phenyl ketone	C15H14O2
158	151	127	86	43	p-cresyl phenylacetate	C15H14O2
159					4-methoxybenzyl phenyl ketone	C15H14O2
160					benzyl 4-methoxyphenyl ketone	C15H14O2
161	152	128	67		1,3-bis(trichloromethyl)benzene	C8H4Cl6
162	153	129			N,N-diethyl-*m*-toluamide	C12H17NO
163	154	130	105		2-bromophenol	C6H5BrO
164	155	131	156	102	acetylsalicylic acid	C9H8O4
165	156	132	103	55	2,6-dibromoaniline	C6H5Br2N
166	157	133	104	56	3,5-di-*t*-butylphenol	C14H22O
167	158	134	106		3,5-dibromocumene	C9H10Br2
168	159	135	108		3-bromo-5-isopropylbenzoic acid	C10H11BrO2
169	160	136	107	62	piperonal	C8H6O3
170	161	137	109		3-nitro-*o*-xylene	C8H9NO2
171	162	138	110		2,4,5-trichlorotoluene	C7H5Cl3
172	163	139	111		2,4,5-trichloroaniline	C6H4Cl3N
173	164	140	112	107	4,6-diiodo-1,3-dimethoxybenzene	C8H8I2O2
174	165	141			2-cyclohexene-1-one	C6H8O
175	166	142			2-hydroxycyclohex-1-en-3-one	C6H8O2
176	167	143			1-acetyl-1-cylohexene	C8H12O
177	168		92	70	4-methylpent-3-en-2-one (mesityl oxide)	C6H10O
178	169	146			indane	C9H10
179	171	148			1-indanone	C9H8O
180	172	149			2-indanone	C9H8O
181	173	150			1-tetralone	C10H10O
182	174	151			β-tetralone	C10H10O
183	176	153			fluorenone	C13H8O
184	177	154	138	80	2,4,6-trimethyl-1,3,5-trioxane	C6H12O3
185	178	155	88	46	3,3-dimethylglutaric anhydride	C7H10O3
186	179	156			2,2-dimethylglutaric anhydride	C7H10O3
187	180	157			mevalonic lactone	C6H10O3
188	181	158			4-ethyl-4-methyl-2,6-piperidinedione;	C8H13NO2
189	182	159			1,2,2,6,6-pentamethylpiperidine	C10H21N
190	183	160			2,5-dimethyl-3-hexyne-2,5-diol	C8H14O2
191	184	161	134		(*Z*)-3-methyl-pent-2-en-4-ynal	C6H6O
192	185	162	135	77	(*Z*)-1-methoxybut-1-en-4-yne	C5H6O
193	187	164	137		1-phenyl-1,3-pentadiyne	C11H8
194	188	165			Methyl 4-amino-3,5-diethynylbenzoate	C12H9NO2
195	189				1,2-dibromoproane	C3H6Br2
196	190	166			2-hydroxy-3-methylbutyric acid	C5H10O3
197	191	145			glycerol	C3H8O3
198	192	63	79	32	2-butanol	C4H10O
199					cyclopentene	C5H8
200					2-cyclopentenone	C5H6O
201	194	167	120	69	(*E*)-3-(phenylthio)acrylic acid	C9H8SO2
202	195	168	121	64	ethyl *p*-toluenesulfonate	C9H12O3S
203	196	169	122	71	p-tolyl methyl sulfoxide	C8H10OS
204	197	170	139	81	p-aminobenzenesulfonamide (sulfanilamide)	C6H8N2O2S
205	198	171	158	104	divinyl sulfone	C4H6O2S
206	199	172	159	105	allyl *p*-anisyl thioether	C10H12OS

207	200	173			Tetraethylene glycol ditosylate	C22H30O9S2
208	201	174	155	101	vinyl 2-chloroethyl ether	C4H7OCl
209	202	176	162		N-(*p*-tolyl)succinimide	C11H11NO2
210	203	177			phenylacetaldeyde ethylene glycol acetal	C10H12O2
211	204	178	94	51	(*E*)-1-phenyl-4-methyl-1-penten-3-one	C12H14O
212	205	179	95	52	cinnamaldehyde	C9H8O
213	206	180	96		cinnamyl alcohol	C9H10O
214	207	181	97	79	(*E*)-3-chloro-4,4-dimethyl-1-phenyl-1-pentene	C13H17Cl
215	208	183	99		cis-β-bromostyrene	C8H7Br
216	209	184	100		trans-*p*-nitro-β-bromostyrene	C8H6BrNO2
217	210	185			3-benzyloxy-1-propanol	C10H14O2
218	211	186	142		homophthallic acid	C9H8O4
219	212	187	141		5,6-dimethoxy-2-coumaranone	C10H10O4
220	213	188	143	84	1,1-di (p-chlorophenyl)-2,2,2-trichloroethane (DDT)	C14H9Cl5
221	214	189	151	96	2,4,5-trichlorophenoxyacetic acid	C8H5Cl3O3
222	215	190	154	100	methyl 2,3-dibromo-3-(*p*-nitrophenyl)propionate	C10H9NO4Br2
223	216	191	172		2,3-di-(*p*-anisyl)butyronitrile	C18H19NO2
224	217	192	90	73	diethyl isopropylidenemalonate	C10H16O4
225	218	193	93	50	4-cyano-2,2-dimethylbutyraldehyde	C7H11NO
226	219	194	147	90	methyl (*E*)-3-methylacrylate	C5H8O2
227					methyl crotonate (methyl 2-methylacrylate)	C5H8O2
228	220	195	160	106	2,5-dimethyl-2,4-hexadiene	C8H14
229	221	196	163	109	malonaldehyde dimethyl acetal	C7H16O4
230	222	197	164	110	2-chloroacetaldehyde diethylacetal	C6H13ClO2
231	223				1,3-dibenzylglycerol	C17H20O3
232					fluorobenzene	C6H5F
233					benzotrifluoride	C7H5F3
234	224	198	124		pyridine	C5H5N
235	225	199	125	36	4-picoline	C6H7N
236	226	200	126		2-picoline	C6H7N
237	227	201	127		3-picoline	C6H7N
238	228	202			3-acetylpyridine	C7H7NO
239	229	203	145	88	isopropyl nicotinate	C9H11NO2
240	230	204	146	89	2-methyl-6-aminopyridine	C6H8N2
241	231	205	128		4-methylpyrimidine	C5H6N2
242	232	206	130	72	styrene epoxide	C8H8O
243	233	217		65	citraconic anhydride	C5H4O3
244	234	207	123	66	2-furoic acid	C5H4O3
245					2-acetylthiophene	C6H6OS
246					2-propylthiophene	C7H10S
247	238	211	129		4-methylimidazole	C4H6N2
248	239	212			benzothiophene	C8H6S
249	240	213			2,3,4,9-tetrahydrocarbazole	C12H13N
250	241	214	116		α-angelicalactone	C5H6O2
251	242	215			2-methyltetrahydrofuran-3-one	C5H8O2
252	243	216	117	60	butyrolactone	C4H6O2
253	244	218	89	95	tetramethyl-1,3-cyclobutanedione	C8H12O2
254	246	221			anthraquinone	C14H8O2
255	247	222	149	92	dodecahydrotriphenylene	C18H24
256	248	223	150		triphenylene	C18H12
257	249	224	161	108	N-methylmorpholine	C5H11NO
258	250	225	166	112	cyclopentanone oxime	C5H9NO
259	252	227	165	111	ε-caprolactam	C6H11NO
260	253	228	167	113	N,N-dimethyl-2,3-dihydroxy-1-propylamine	C5H13NO2
261	254	229	168		pseudoephedrine	C10H15NO
262	255	230	157		t-butylformamide	C5H11NO
263	256	231			N-acetylcysteine	C5H9NO3S
264	257	232			adrenalin	C9H13NO3
265	258	233			tryptophan	C11H12N2O2
266	259	234			N-acetylhomocysteine thiolactone	C6H9NO2S
267	260	235			glutamic acid	C5H9NO4
268	262	239			acrolein diethyl acetal	C7H14O2
269	263	237	153	99	allylamine	C3H7N
270	264	238			adamantane	C10H16
271	265	240			2-methyl-2,4-pentanediol	C6H14O2
272	266	241	169		eugenol	C10H12O2
273	268	242			N-acetylaspartic acid	C6H9NO5
274	269	243			N-acetylglutamic acid	C7H11NO5
275	270	244			N-acetyltyrosine ethyl ester	C13H17NO4
276	271	245	170		2,5-dihydrofuran	C4H6O
277	272	246	171	114	2,3-dihydrofuran	C4H6O
278	273	247	173		2,3-naphthalenedicarboxylic acid	C12H8O4

279	274	250	176	115	1-methoxy-4-nitronaphthalene	C11H9NO3
280	275			94	1,5-dimethylnaphthalene	C12H12
281	276	248	174		1,3-dimethylnaphthalene	C12H12
282	277	249	175		2-chloronaphthalene	C10H7Cl
	147	123			1,4-diaminobutane	C4H12N2
	149	125	84	41	benzyl benzoate	C14H12O2
	170	147	140	82	3,3-dimethylindanone	C11H12O
	175	152	87	44	9-methylfluorene	C14H12
	186	163	136	97	3-methyl-1-phenylpent-1-yn-3-ol	C12H14O
	193	29			dibenzyl sulfoxide	C14H14SO
	235	208	133	76	2-furyl *t*-butyl ketone	C9H12O2
	236	209	131	74	2,4-dinitrothiophene	C4H2N2O4S
	237	210	132	75	2-(5-nitrothienyl) isopropyl ketone	C8H9NO3S
	245	220	148	91	octahydroanthracene	C14H18
	251	226			cyclohexanone oxime	C6H11NO
			8		2-bromoisobutyric acid	C4H7O2Br
			36		acetone dimethyl acetal	C5H12O2
			101	53	diethyl ethylmalonate	C9H16O4
			119	63	1-methoxy-2-(chloromethoxy)ethane	C4H9OCl
			144	87	2-(2-hydroxyethyl)-pyridine	C7H9O2Cl
				10	2-chloro-2-methylbutane	C5H11Cl
				45	α-bromostyrene	C8H7Br
				47	α-methylstyrene	C9H10
				49	1,1-diphenyl-1,4-butanediol	C16H18O2
				67	3,5-di-t-butylcatechol	C12H21O2
				68	3,5-di-t-butyl-1,2-benzoquinone	C12H19O2
				78	N-methyl-*N*-(2-hydroxyethyl)ethanolamine	C5H13O2N
				83	N-*p*-tolylurea	C8H10ON2
				85	phenylisothiocyanate	C7H5NS
				93	2,7-dimethylnaphthalene	C12H12
				103	N-methyl-1-methyl-2-hydroxy-2-phenylethanolamine	C10H15ON
283	278	251	177		sec-butylbenzene	C10H14
284	279	252	178		N-(1-methyl-1-phenylethyl)-butyramide	C12H17NO
285	280	253	179		diethyl 2-(1,1-dimethylheptyl)malonate	C16H30O4
286	281	254	180		ethyl 4-piperidone-*N*-carboxylate	C8H13NO3
287	282	255	181		N-acetyl-2-amino-4-phenyl-(E)-but-2-enoic acid	C12H13NO3
288	283	256			3-hydroxy-3-methyl-5,8-dimethoxy-1-coumarinone	C13H16O4
289	284				Mixture 1H - ethanol and bromoethane	
290	285				Mixture 1H - benzene, DCM and diethyl ether	
291	286				Mixture 1H - benzene, ethyl acetate and dioxane	
292	287				Mixture 13C - ethanol and bromoethane	
293	288				Mixture 13C - benzene, DCM and diethyl ether	
294	289				Mixture 13C - benzene, ethyl acetate and dioxane	
295	290				Fluorene + Fluorenone	
296	291				Mixture of o/p nitroanisole	
297	292				1-propanol - COSY & HSQC - determine 1H & 13C assignments	C3H8O
298	293				1-iodobutane - predict the COSY & HSQC	C4H9I
299	294				isobutanol - predict the COSY & HSQC	C4H10O
300	295				3-heptanone - COSY - determine 1H assignments	C7H14O
301	296				delta-valerolactone - COSY & HSQC - determine 1H & 13C assignments	C5H8O2
302	297				1-bromobutane - COSY & HSQC - determine 1H & 13C assignments; predict the HSQC	C4H9Br
303	300				butyl ethyl ether - COSY & HSQC - determine 1H & 13C assignments; predict the HMBC	C6H14O
304	298				3-octanone - COSY & HSQC - determine 1H & 13C assignments	C8H16O
305	299				diethyl diethylmalonate - COSY & HSQC - determine 1H & 13C assignments	C11H20O4
306	301				butyl butyrate - COSY & HSQC - determine 1H & 13C assignments	C8H16O2
307	302				mixture 1-iodobutane & 1-butanol - COSY & TOCSY - determine 1H assignments	C4H9I/C4H10O
308	303				E & Z-2-bromo-2-butene - predict the NOESY	C4H7Br
309	304				3-methylpent-2-en-4-yn-1-ol - NOESY - determine stereochemistry	C6H8O
310	305				1-nitronaphthalene - NOESY - determine 1H assignments	C10H7NO2

311					2-bromo-5-nitrotoluene - aromatic compound with benzylic - predict the HSQC & HMBC	C7H6BrNO2
312					quinoline - NOESY, HSQC & HMBC - determine 1H & 13C assignments	C9H7N
313					diethyleneglycol ethyl ether acetate - COSY, HSQC & HMBC - determine 1H & 13C assignments	C8H16O4
314					4-ethylacetophenone - 1D NMR data, HSQC & HMBC - identify the correct compound from a series	C10H12O
315					3,3-dimethylindanone - 1D spectral data, HSQC & HMBC -identify the compound	C11H12O
316					4-methylbenzyl phenyl ketone - 1D NMR data, HSQC & HMBC - identify the correct compound from a series	C15H14O
317					thymol - 1D NMR data, HSQC & HMBC - identify the compound	C10H14O
318	306				diethyl ethylmalonate - 1D spectral data, COSY & HSQC - identify compound	C9H16O4
319	307				butyl valerate - 1D spectral data, COSY & HSQC - identify compound	C9H18O2
320					vanillin - 1D NMR data, NOESY - identify the compound	C8H8O3
321	308				nerol - 1D spectral data, COSY & NOESY - identify compound & stereochemistry	C10H18O
322	309				geraniol - 1D spectral data, COSY & NOESY - identify compound & stereochemistry	C10H18O
323					E -1-chloro-4-(4-t-butylphenyl)but-1-en-3-yne - 1D NMR data, HSQC & HMBC - identify the compound	C14H15Cl
324	310	257	182		symmetry - predict number of chemically non-equivalent nuclei	
325	311			122	draw schematic spectrum AMX from shifts and coupling constants	
326	312	270	193		draw schematic spectrum AMX from shifts and coupling constants	
327	313	271			draw schematic spectrum AX2 from shifts and coupling constants	
328	314	258	183	127	Analyse 60 MHz spectrum of diethyl ether	
329	315	259	184	117	Analyse 100 MHz 3-spin AMX system	
330	316	260	185	116	Analyse 60 MHz 3-spin AMX system 2-furoic acid	
331	317	261	186	128	Analyse 100 MHz 3-spin AMX system 2-amino-5-chlorobenzoic acid	
332	318a	262a	187	121a	Analyse 100 MHz 3-spin AMX system methyl acrylate	
332b	318b	262b	187b	121b	Analyse 100 MHz 3-spin AMX system methyl acrylate - simulation	
333	319			124	draw schematic spectrum AX3 from shifts and coupling constants	
334	320	263	188	118	Analyse 100 MHz 4-spin AX3 system	
335	321	272	194	126	draw schematic spectrum AMX2 from shifts and coupling constants	
336	322	273	195	123	draw schematic spectrum AM2X from shifts and coupling constants	
337	323	264	189	119	Analyse 100 MHz 4-spin AM2X system	
338	324	265			Analyse 100 MHz 4-spin A2MX system	
339	325	266			Analyse 100 MHz 4-spin AMX2 system	
340	326	267	190		Analyse 200 MHz 5-spin A2M2X system	
341	327	268	191	120	100 MHz 5-spin AMX3 system - crotonic acid	
342	328	269	192	129	Analyse 100 MHz 6-spin ADMX3 system - unknown aldehyde	
343	329	274	196/7		draw schematic spectrum AMX3 from shifts and coupling constants	
344	330	275	198	130	identify the isomer of dibromoaniline from coupling constants	
345	331	276	199	131	identify the isomer of hydroxycinnamic acid from coupling constants	
346	332	277	192		identify the isomer of dichloronaphthalene from coupling constants	
				125	draw schematic 60 MHz spectrum A2MX from shifts and coupling constants	