Supplementary Information for

Synthesis of Biginelli dihydropyrimidinone derivatives with various substituents on aluminum-planted mesoporous silica catalyst

Hiroaki Murata, Haruro Ishitani, Masakazu Iwamoto*

Chemical Resources Laboratory, Tokyo Institute of Technology, 4259-R1-5 Nagatsuta, Midori-ku, Yokohama, 226-8503, Japan

iwamoto@res.titech.ac.jp

Table of Contents

1. Analytical data of Al-planted MCM-41s

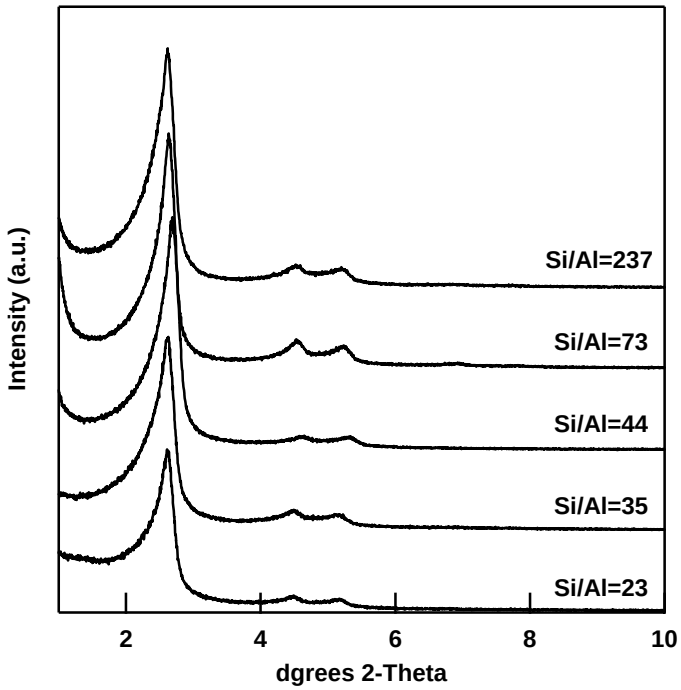


Fig. S1. XRD of (Al-) M41s

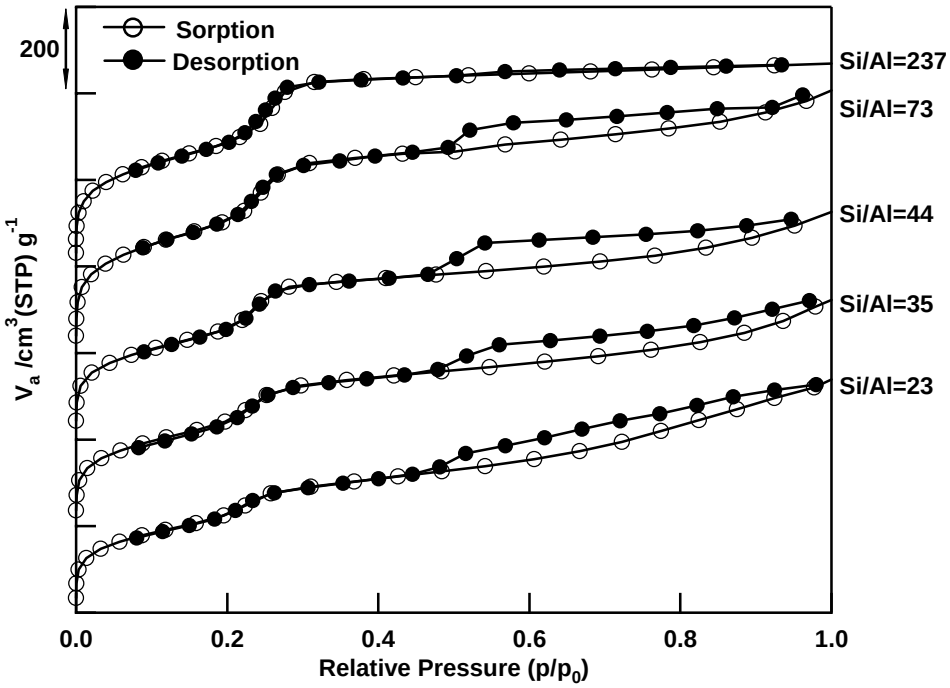
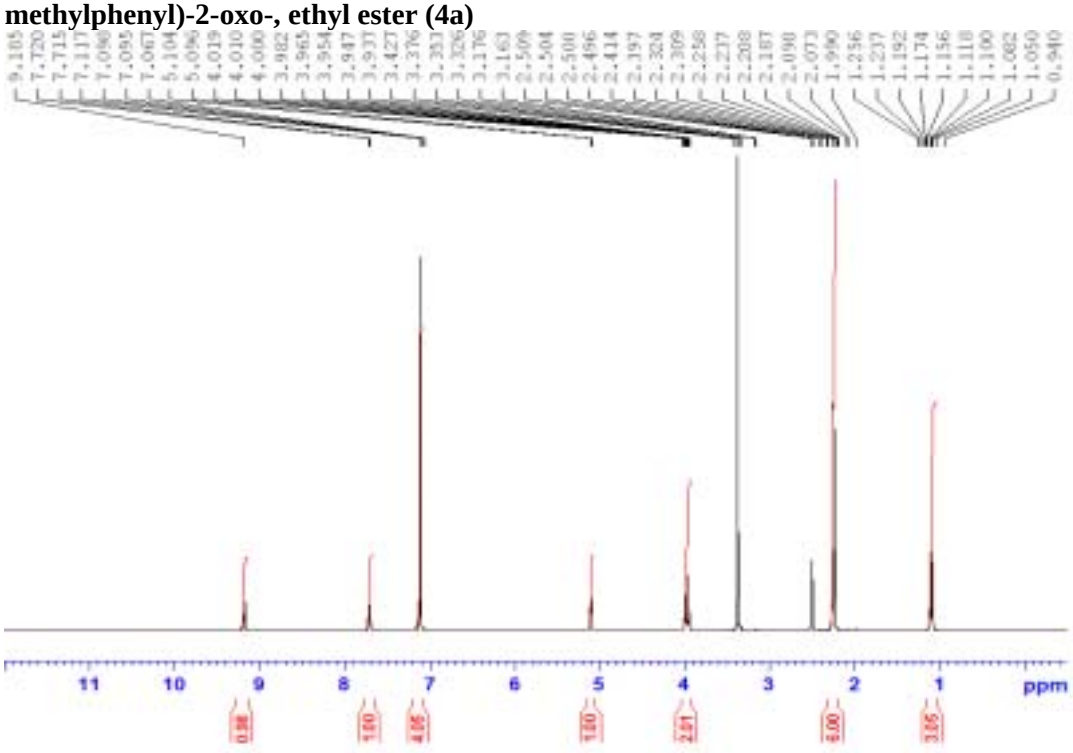
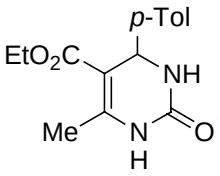


Fig. S2. N_2 sorption and desorption isotherms of (Al-) M41s

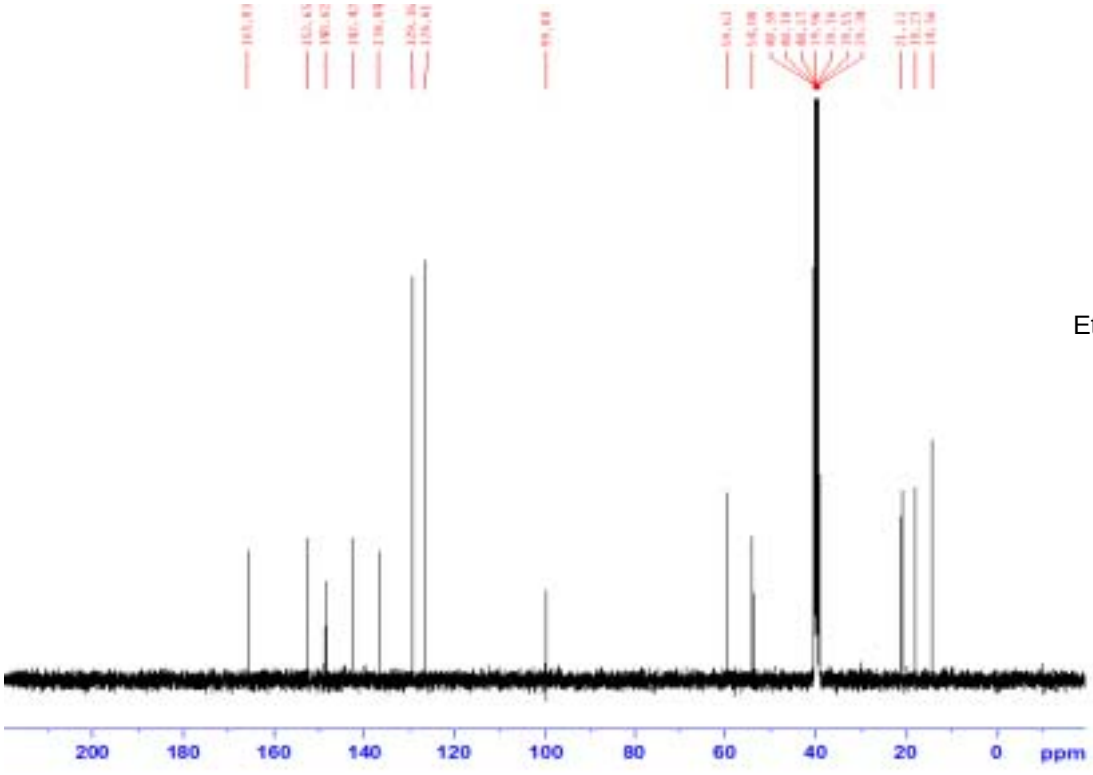
2. ^{1}H and ^{13}C NMR spectra of 4a-4q, 4s-4aa, and 5a

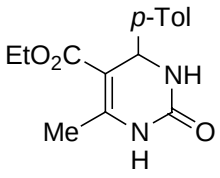
Fig. S3. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-methyl-4-(4-methylphenyl)-2-oxo-, ethyl ester (4a)





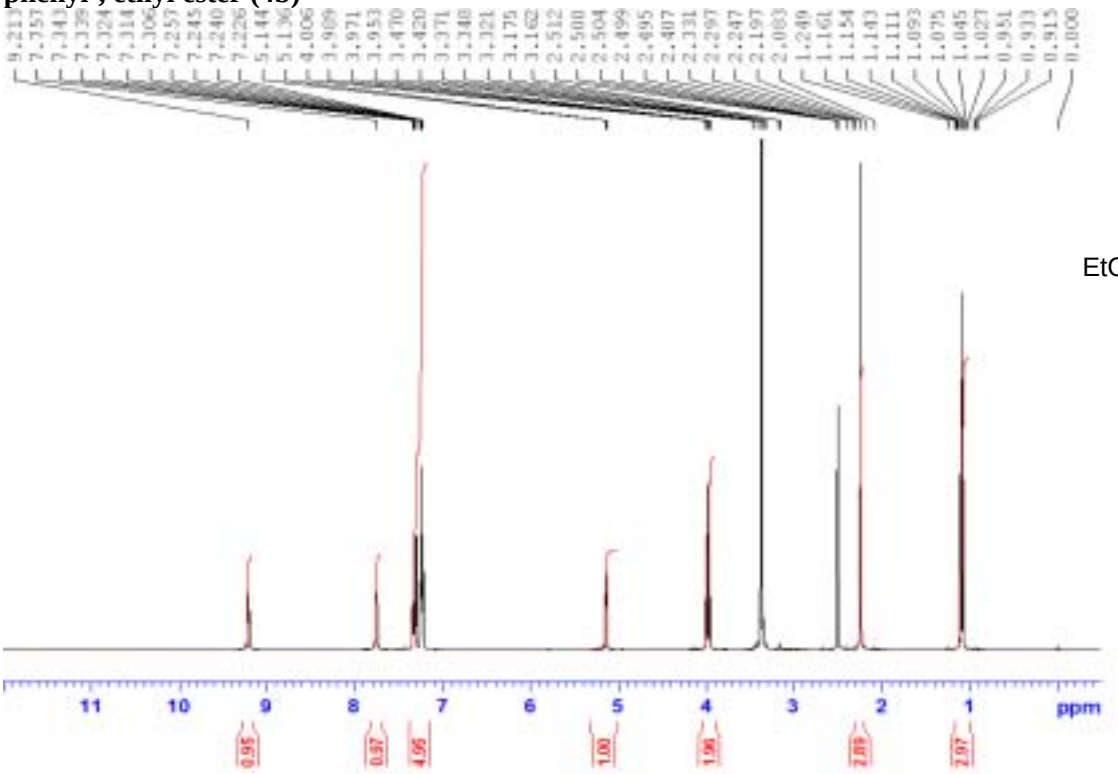
4a

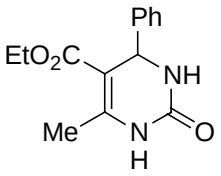




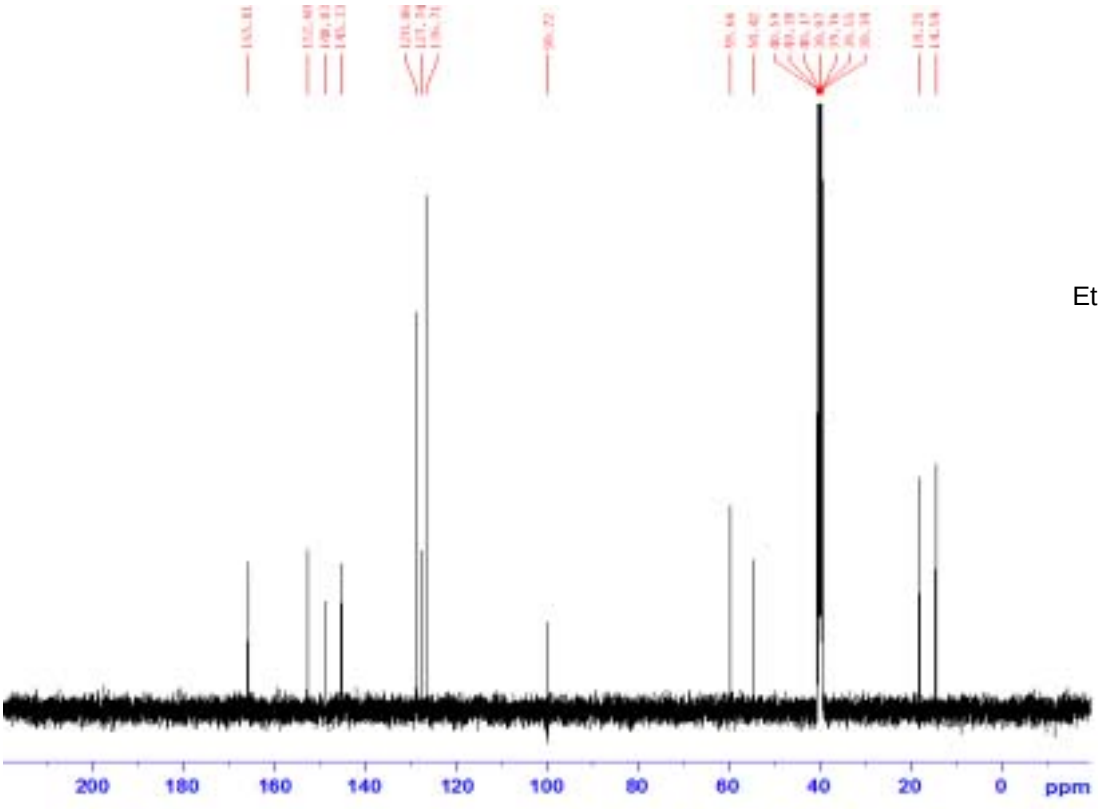
4a

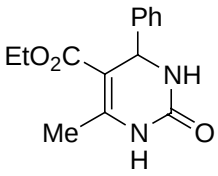
Fig. S4. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-phenyl-, ethyl ester (4b)





4b





4b

Fig. S5. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-1,6-dimethyl-4-(4-methylphenyl)-2-oxo-, ethyl ester (4c)

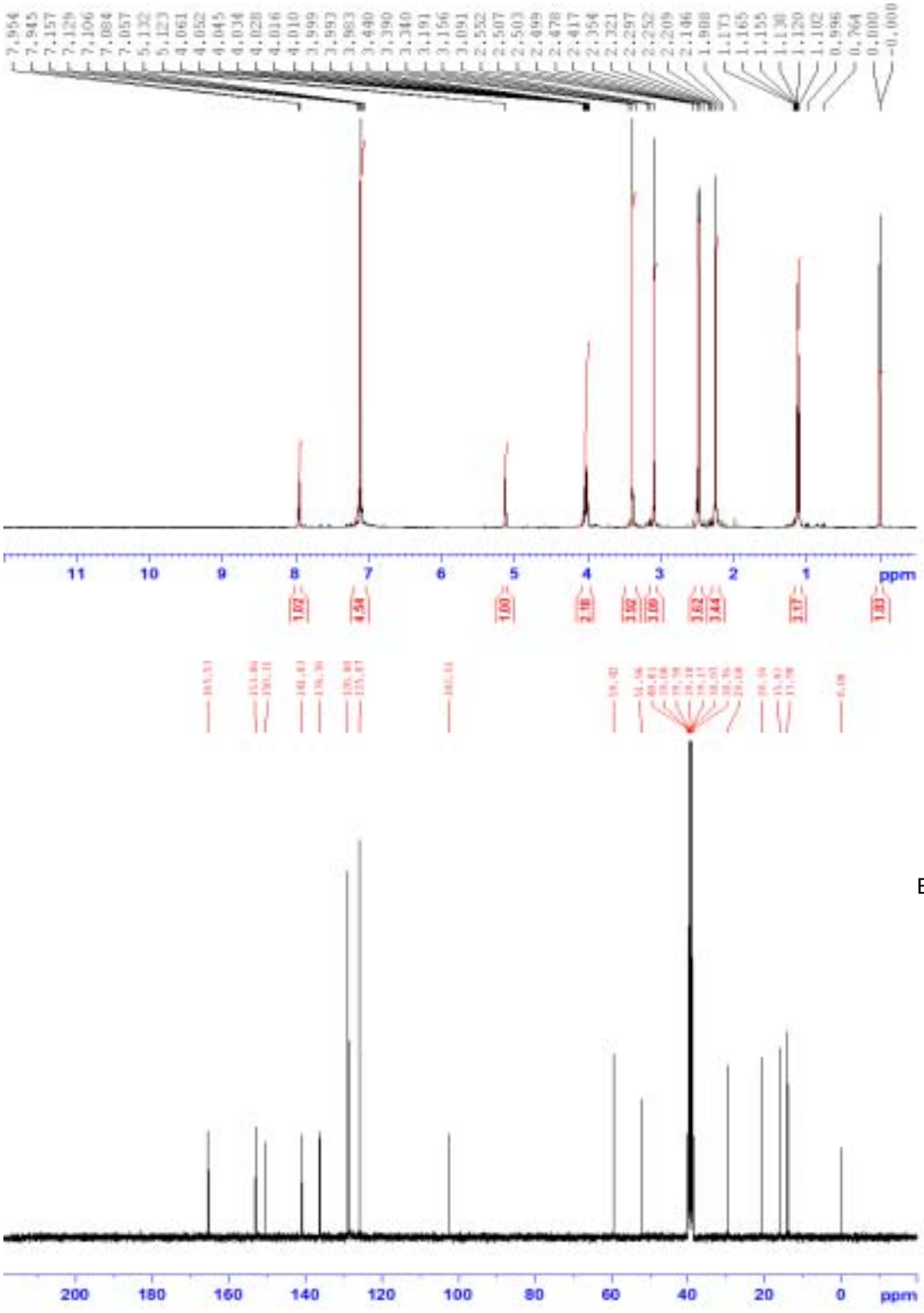


Fig. S6. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-methyl-4-(4-methylphenyl)-2-thioxo-, ethyl ester (4d)

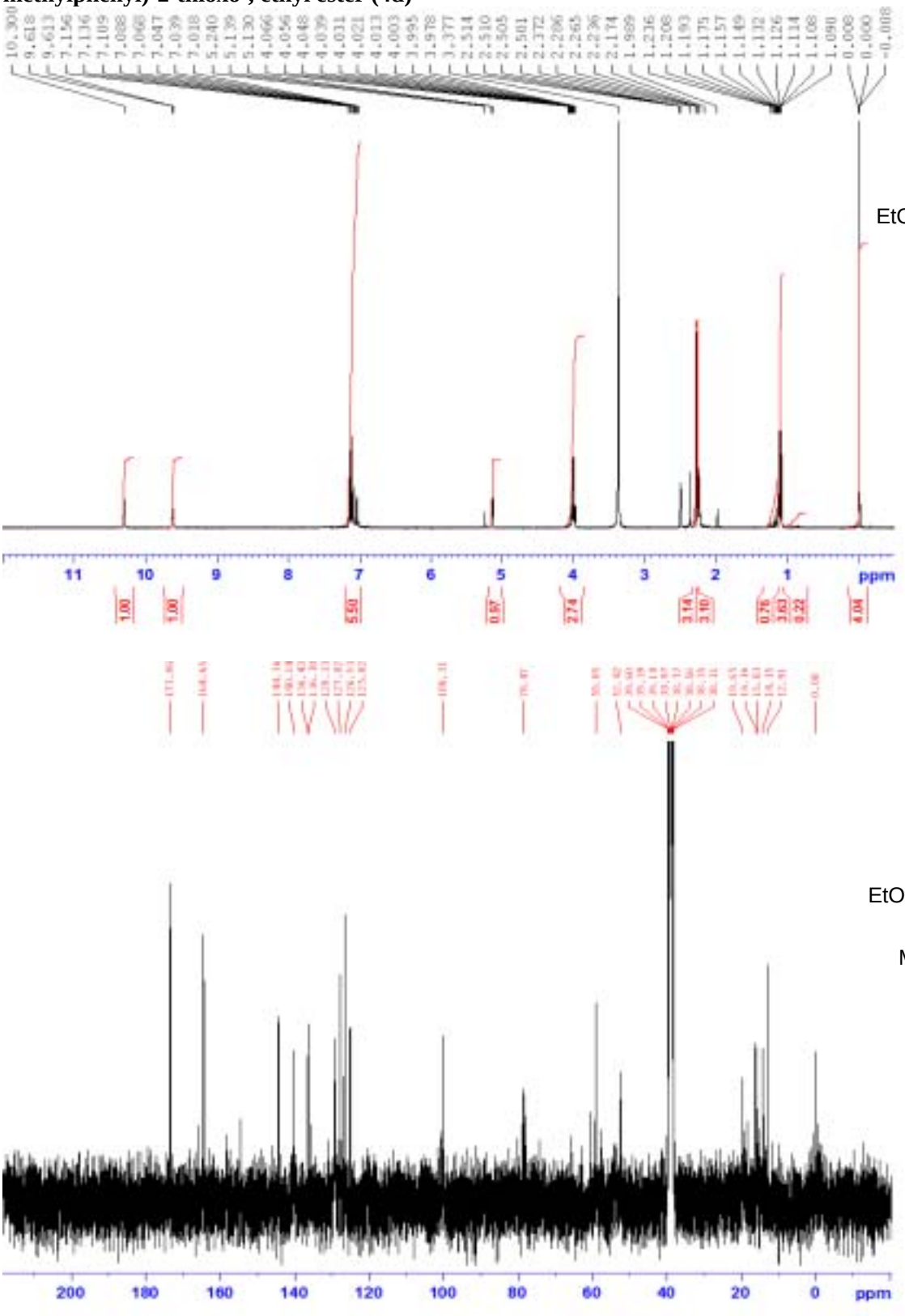


Fig. S7. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-chlorophenyl)-6-methyl-2-oxo-, ethyl ester (4e)

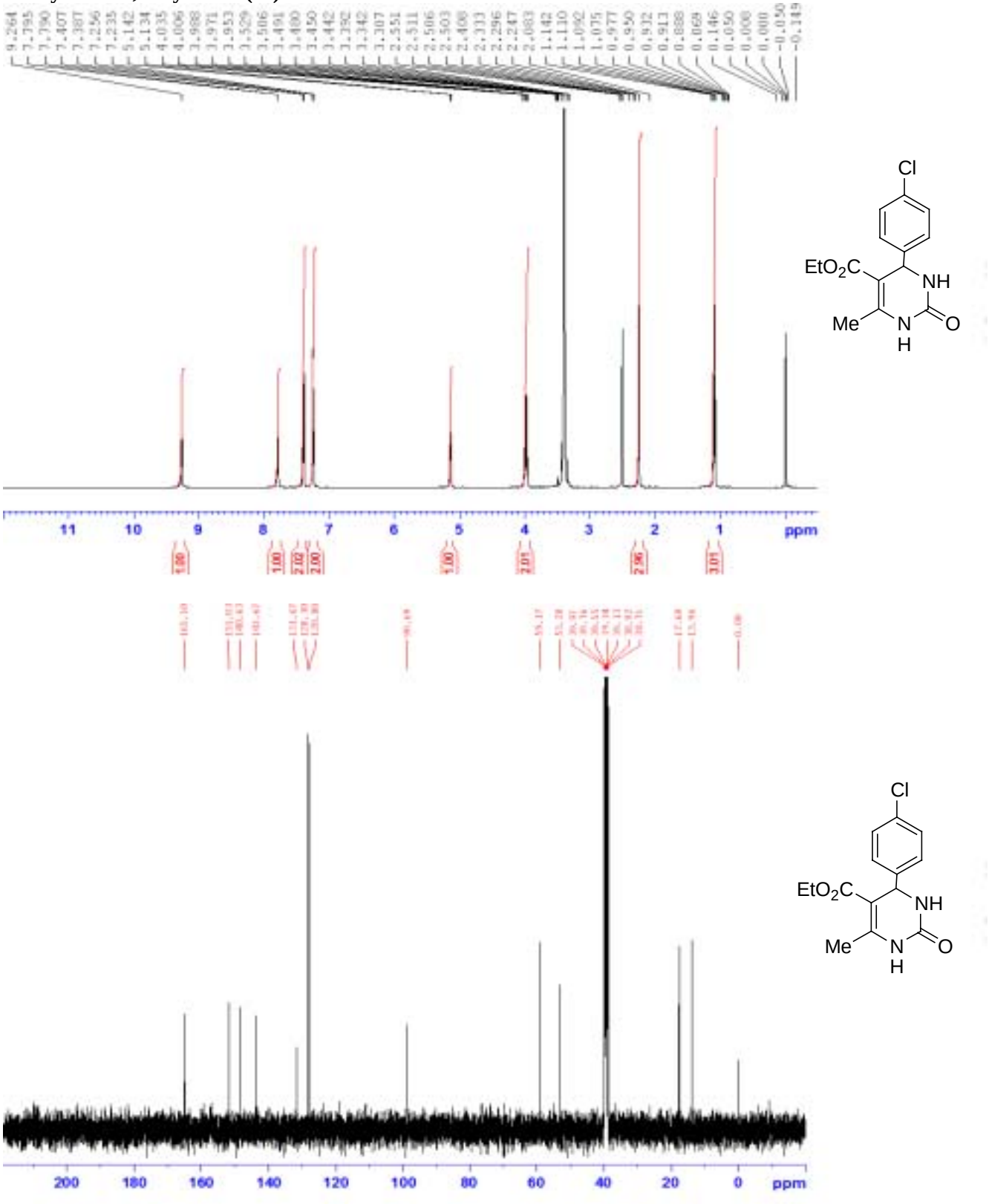
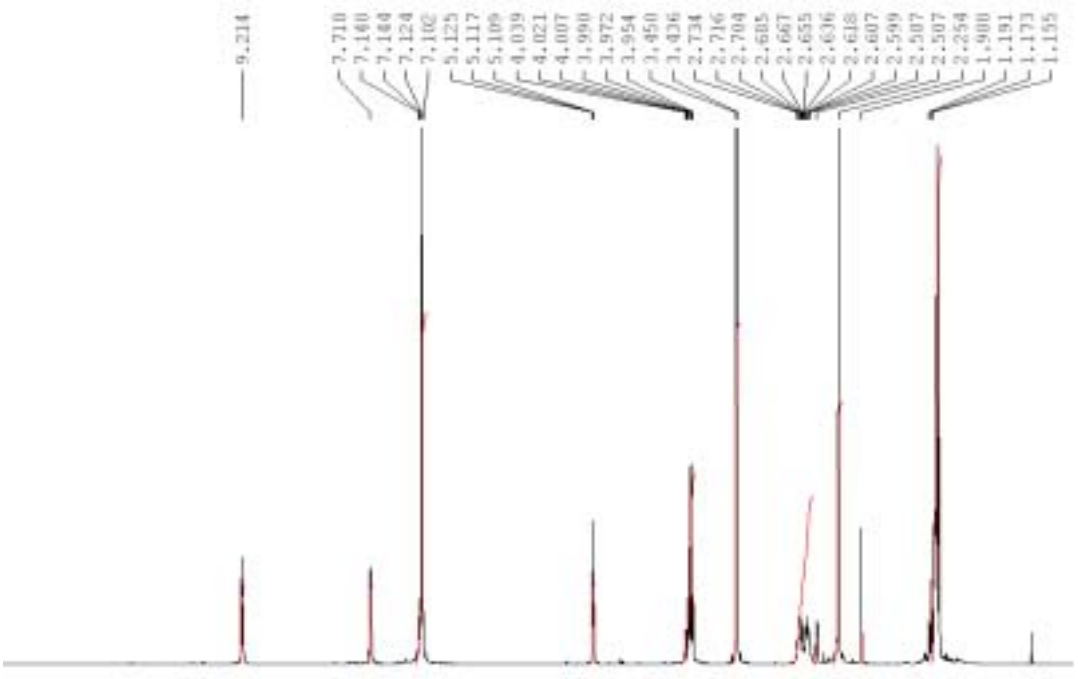
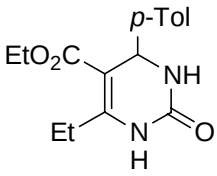
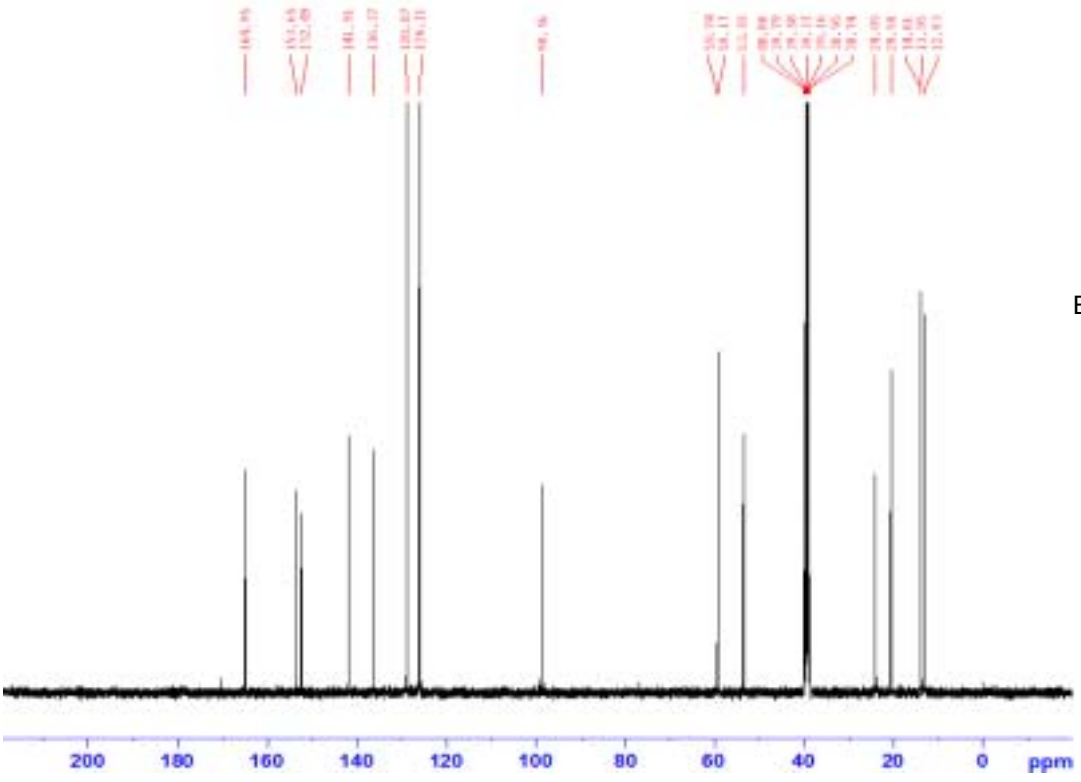


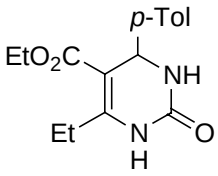
Fig. S8. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-ethyl-4-(4-methylphenyl)-2-oxo-, ethyl ester (4f)





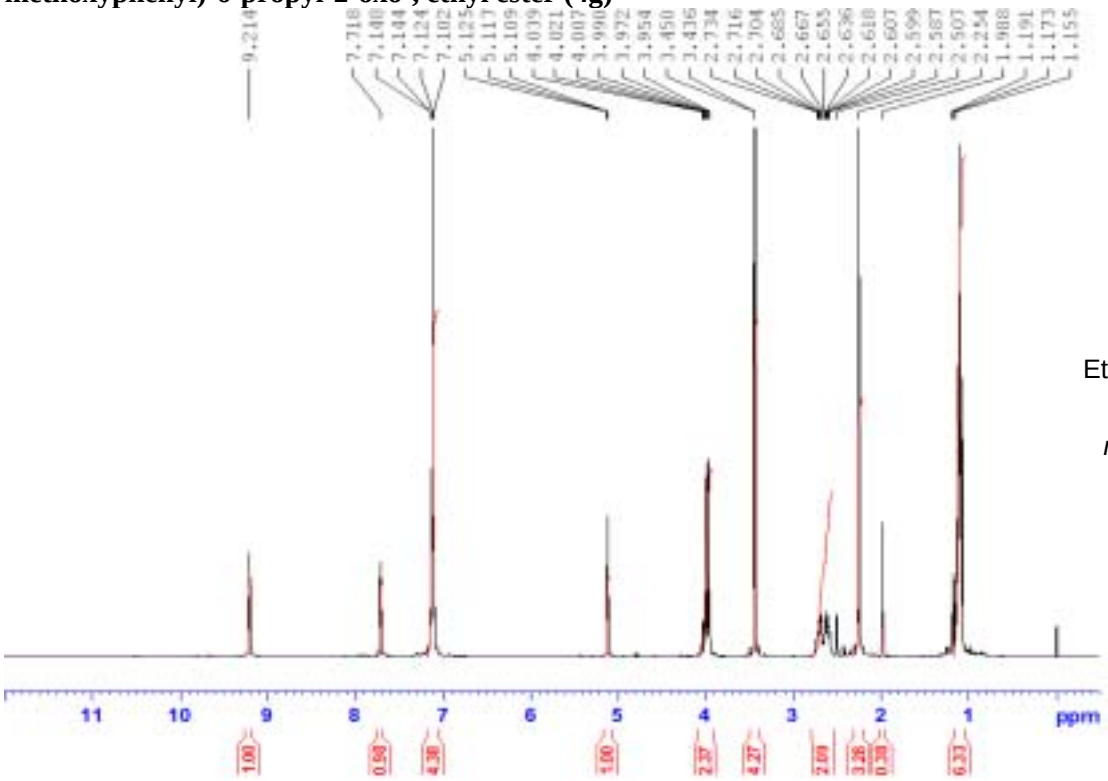
4f

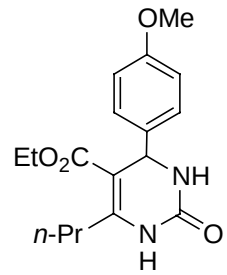


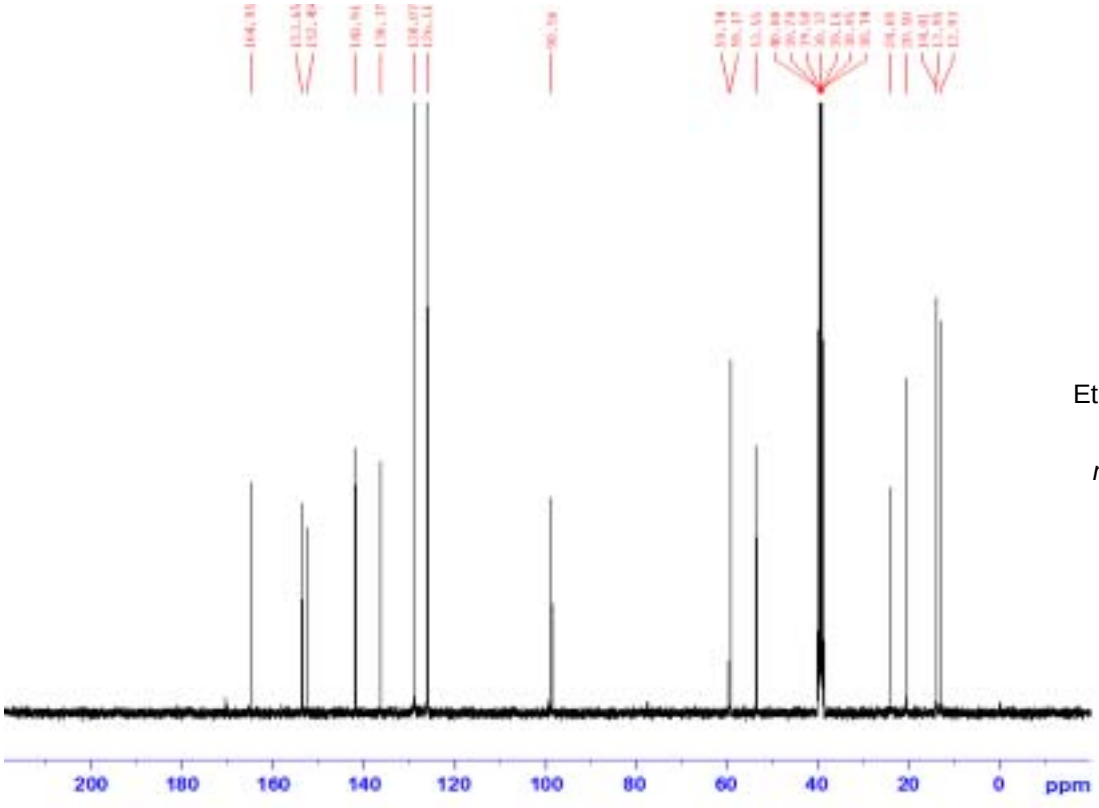


4f

Fig. S9. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methoxyphenyl)-6-propyl-2-oxo-, ethyl ester (4g)







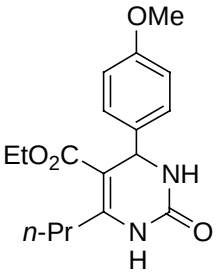


Fig. S10. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methoxyphenyl)-6-propyl-2-thioxo-, ethyl ester (4h)

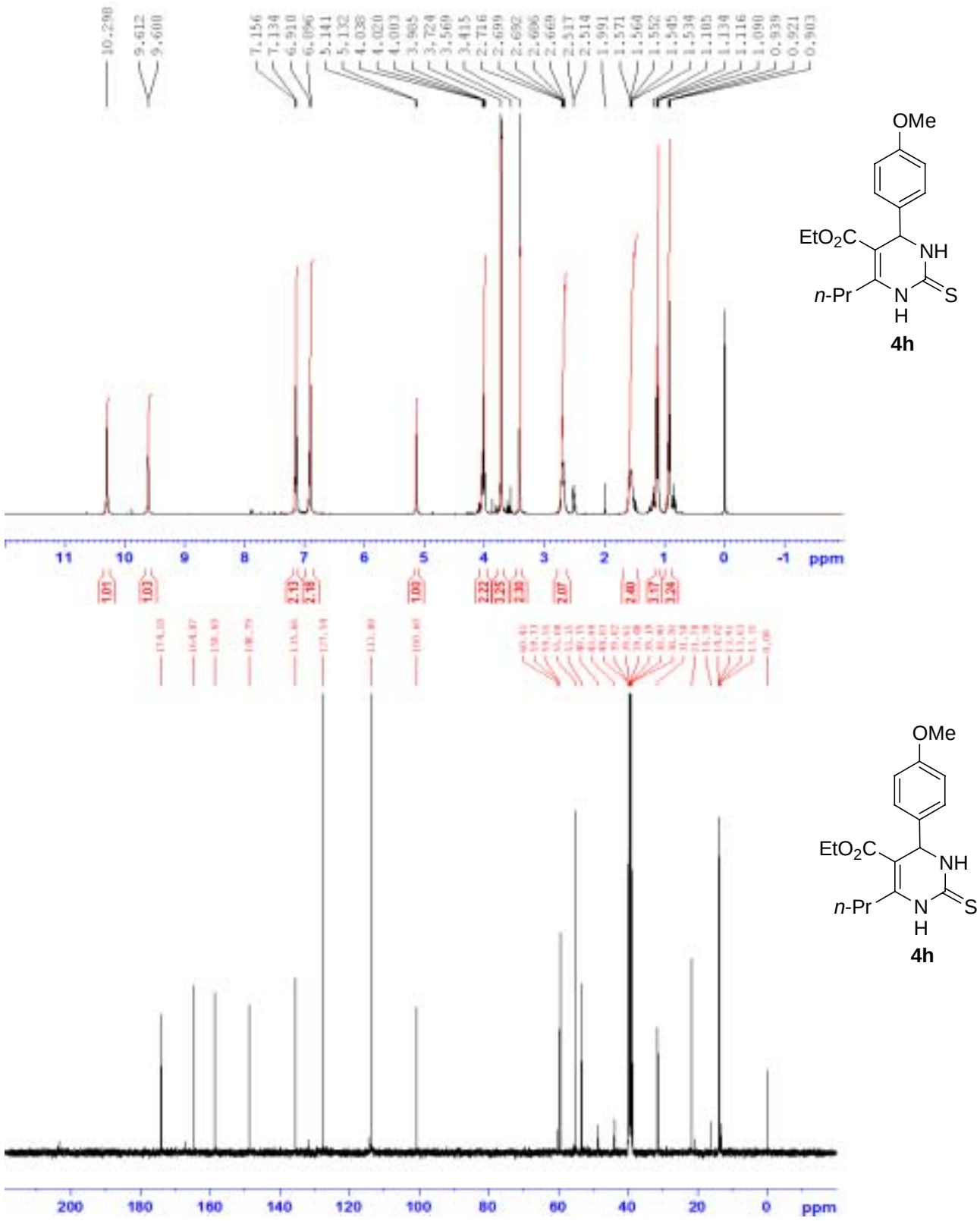
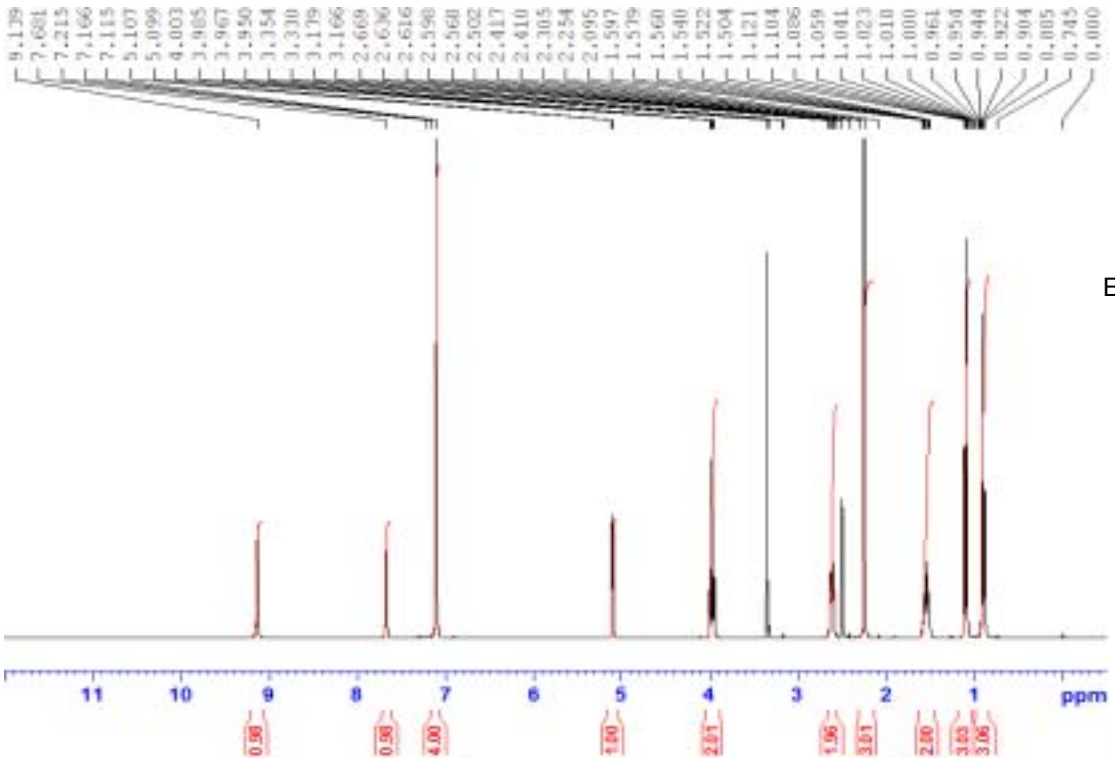
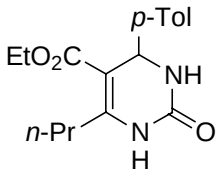
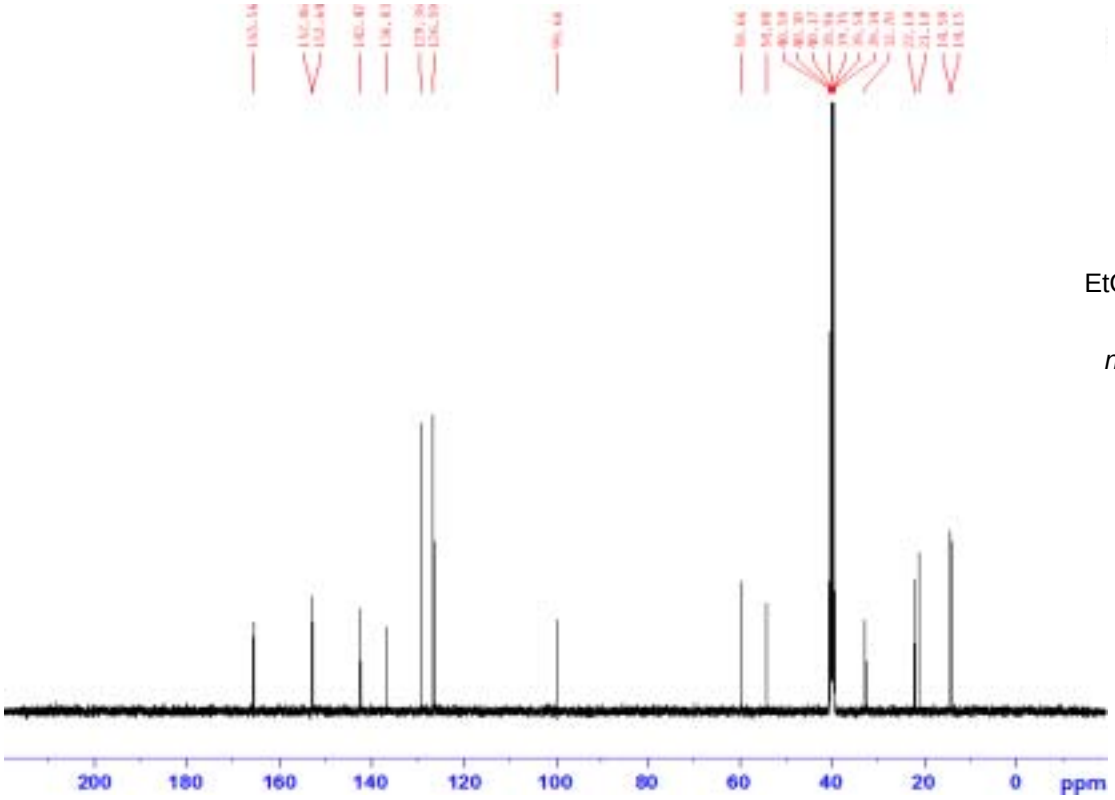


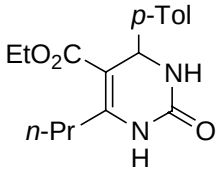
Fig. S11. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro -4-(4-methylphenyl)-2-oxo-6-propyl-, ethyl ester (4i)





4i





4i

Fig. S12. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-1-methyl-4-(4-methylphenyl)-2-oxo-6-propyl-, ethyl ester (4j)

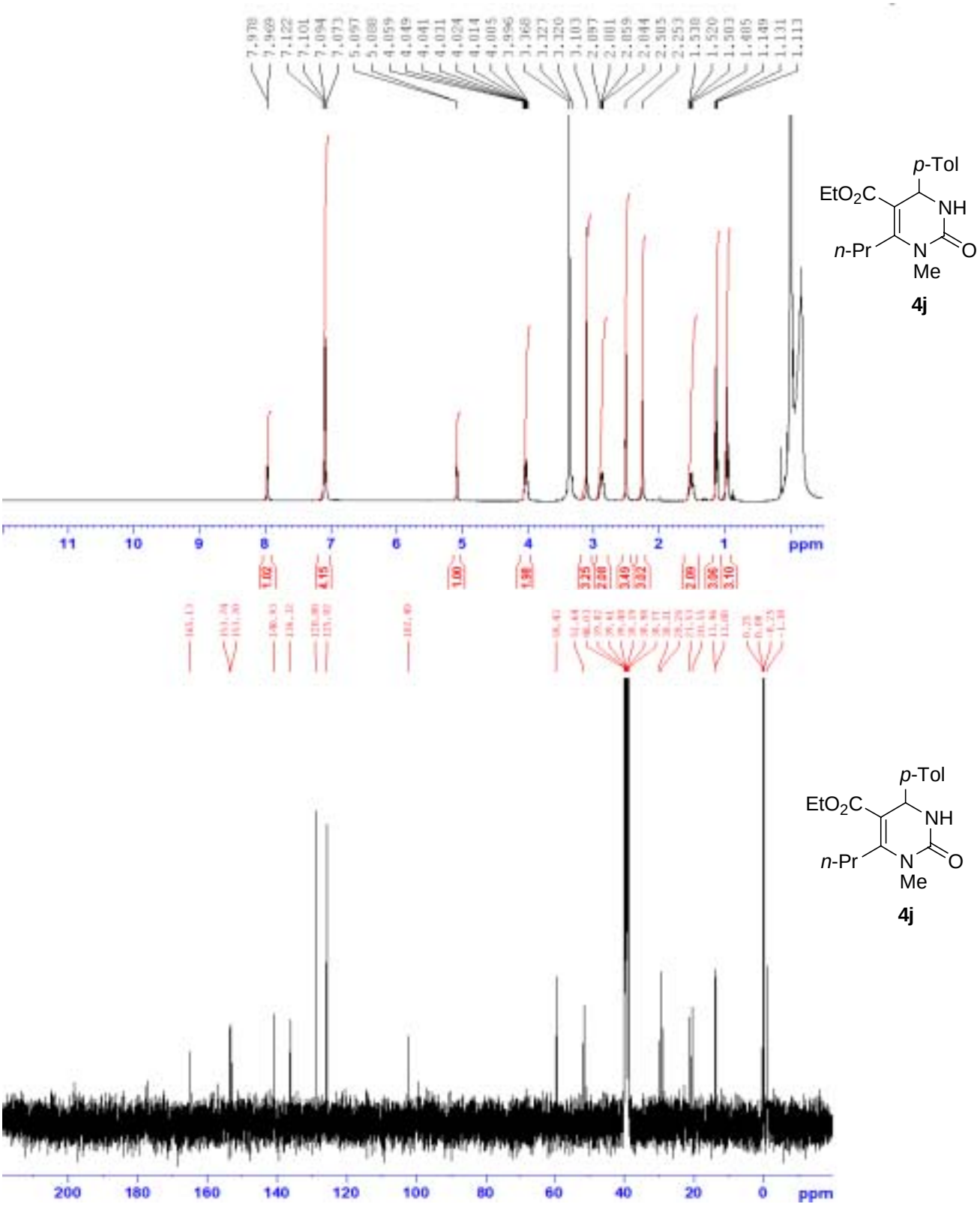
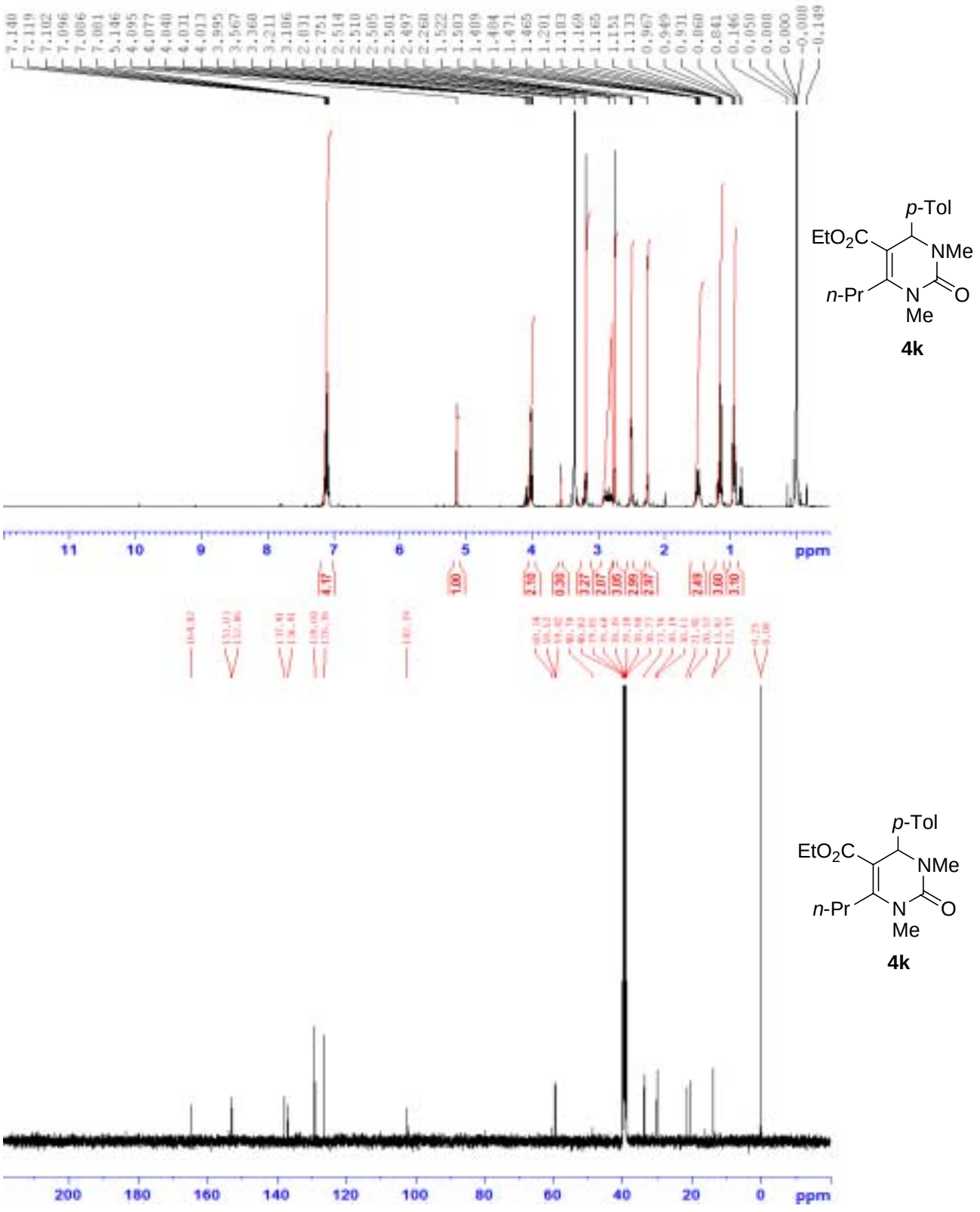
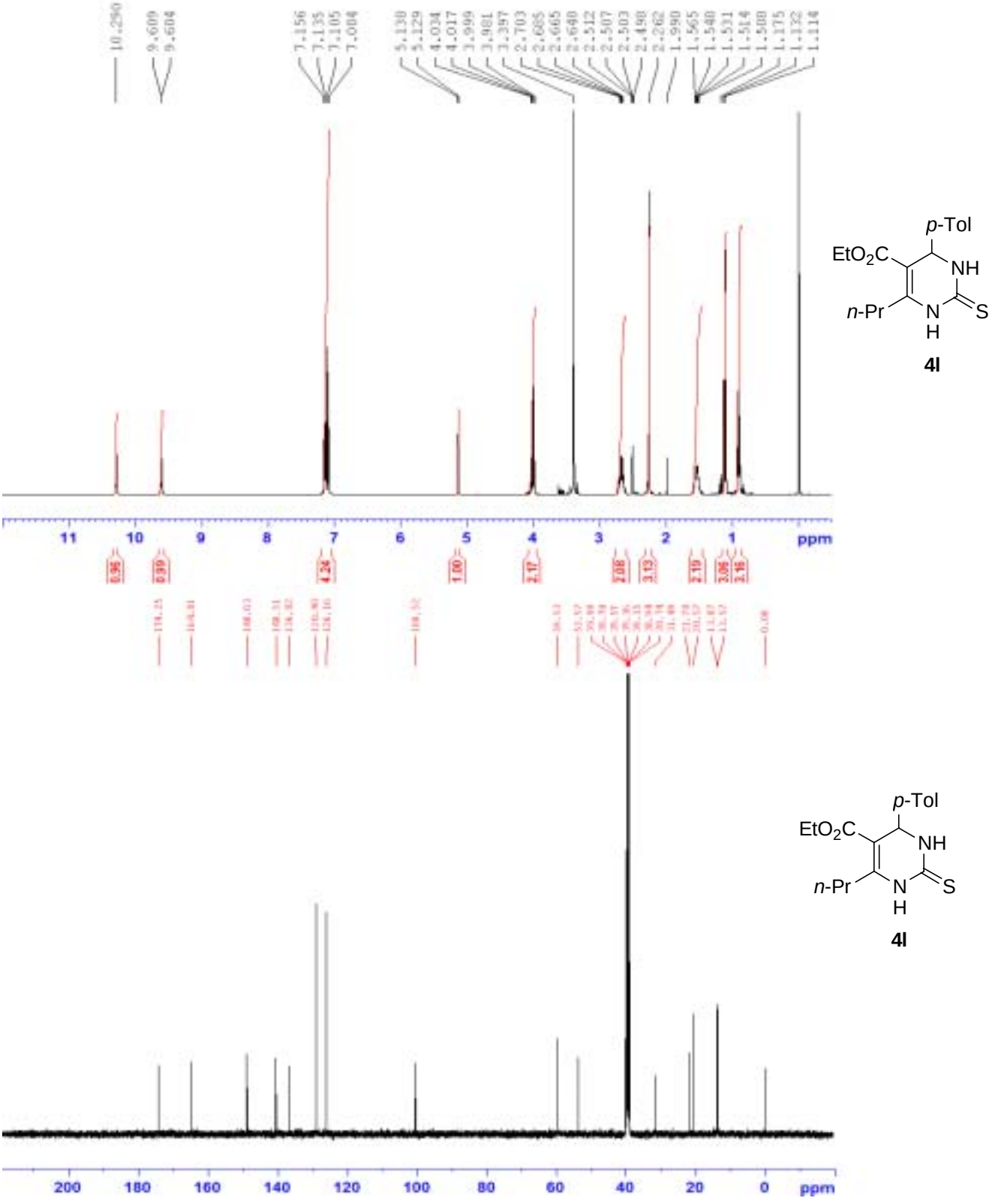


Fig. S13. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-1,3-dimethyl-4-(4-methylphenyl)-2-oxo-6-propyl-, ethyl ester (4k)



5

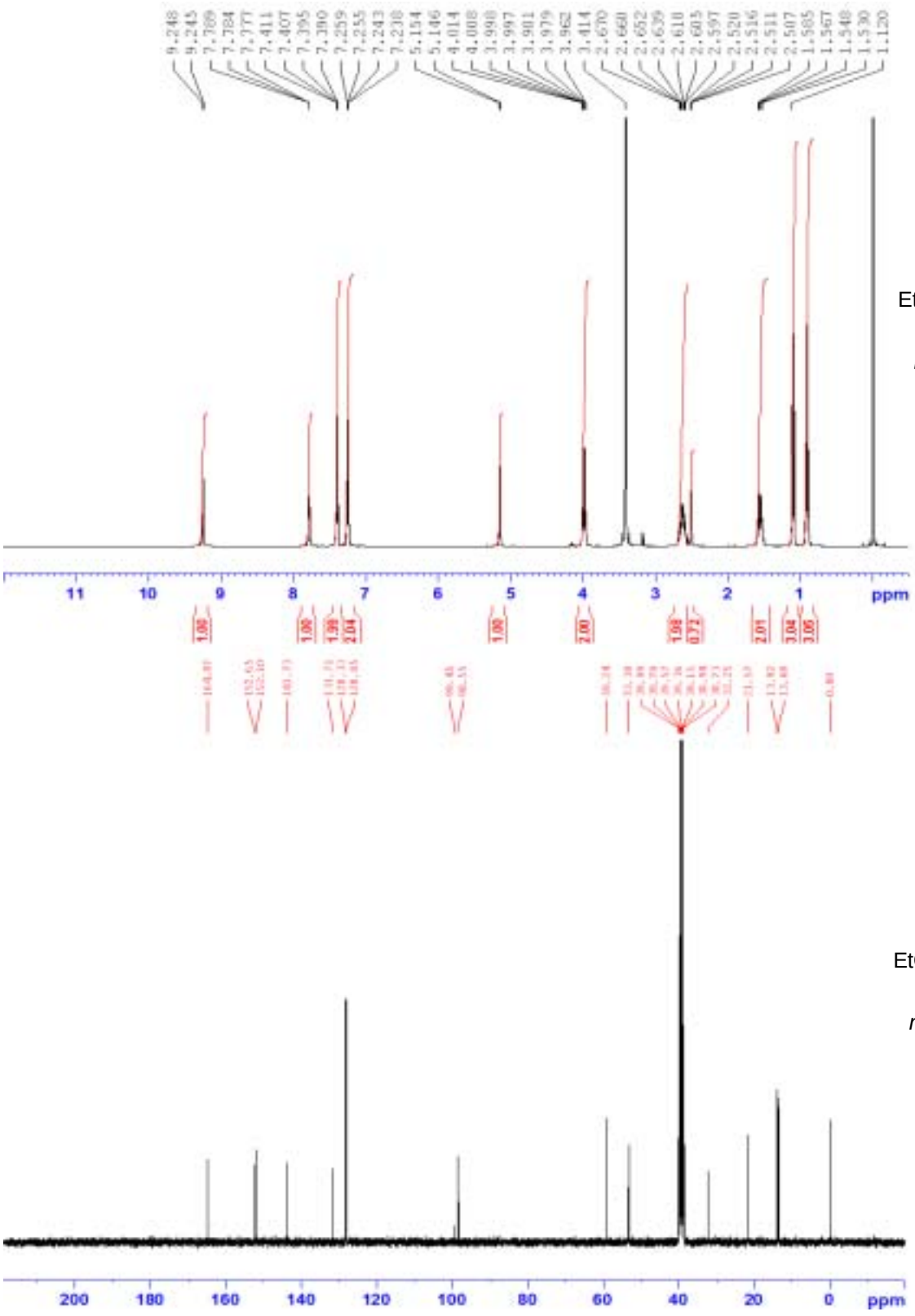
Fig. S14. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methylphenyl)-6-propyl-2-thioxo-, ethyl ester (4l)

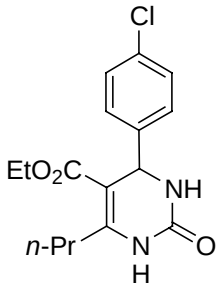


4l

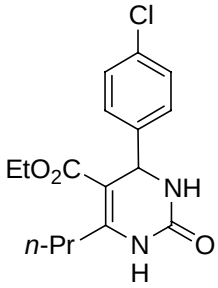
4l

Fig. S15. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-chlorophenyl)-2-oxo-6-propyl-, ethyl ester (4m)





4m



4m

Fig. S16. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-chlorophenyl)-6-propyl-2-thioxo-, ethyl ester (4n)

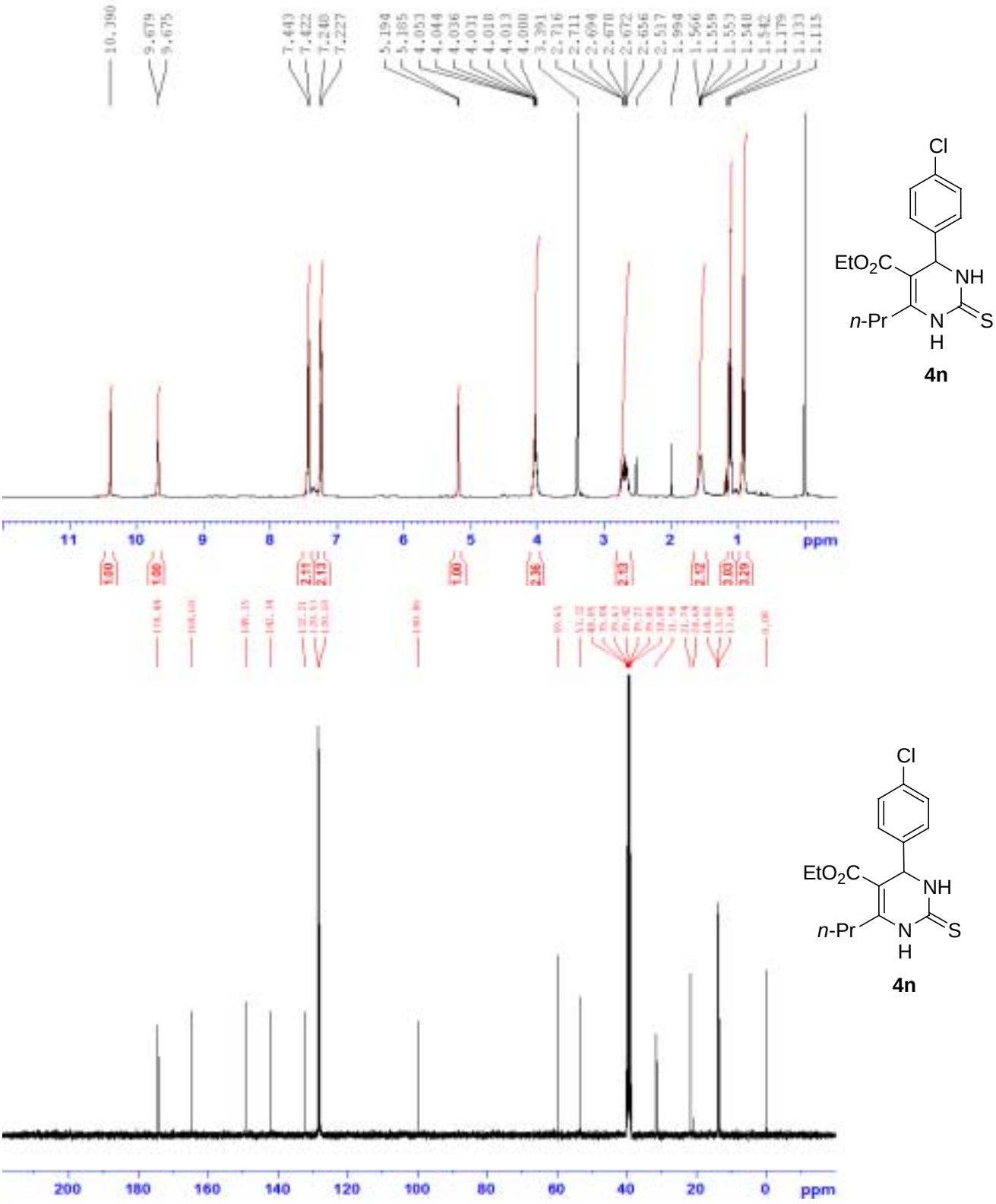
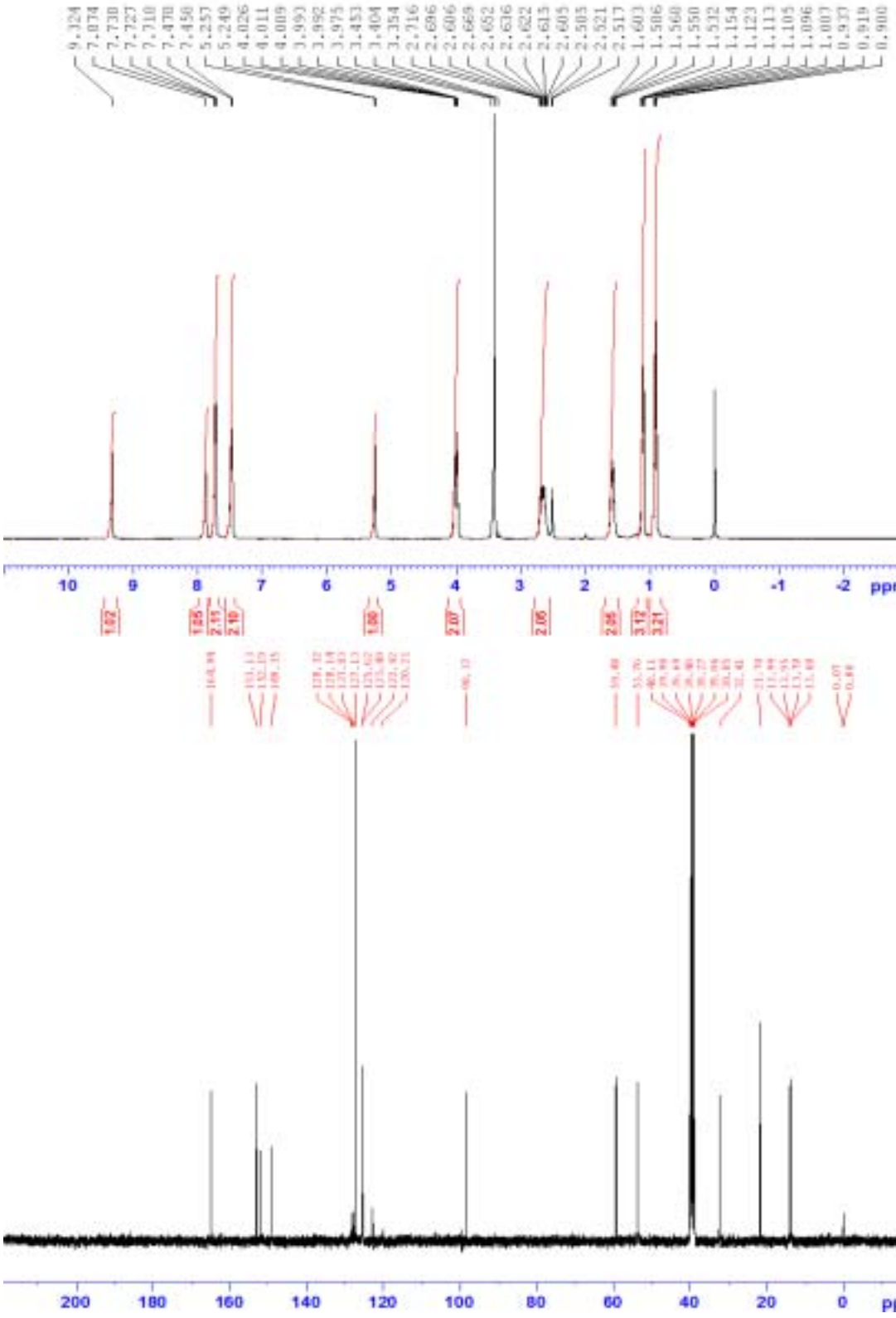
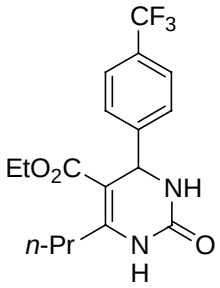
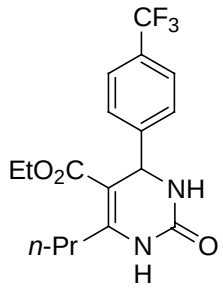


Fig. S17. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-[(4-trifuluoromethyl)-phenyl]-6-propyl-2-oxo-, ethyl ester (4o)



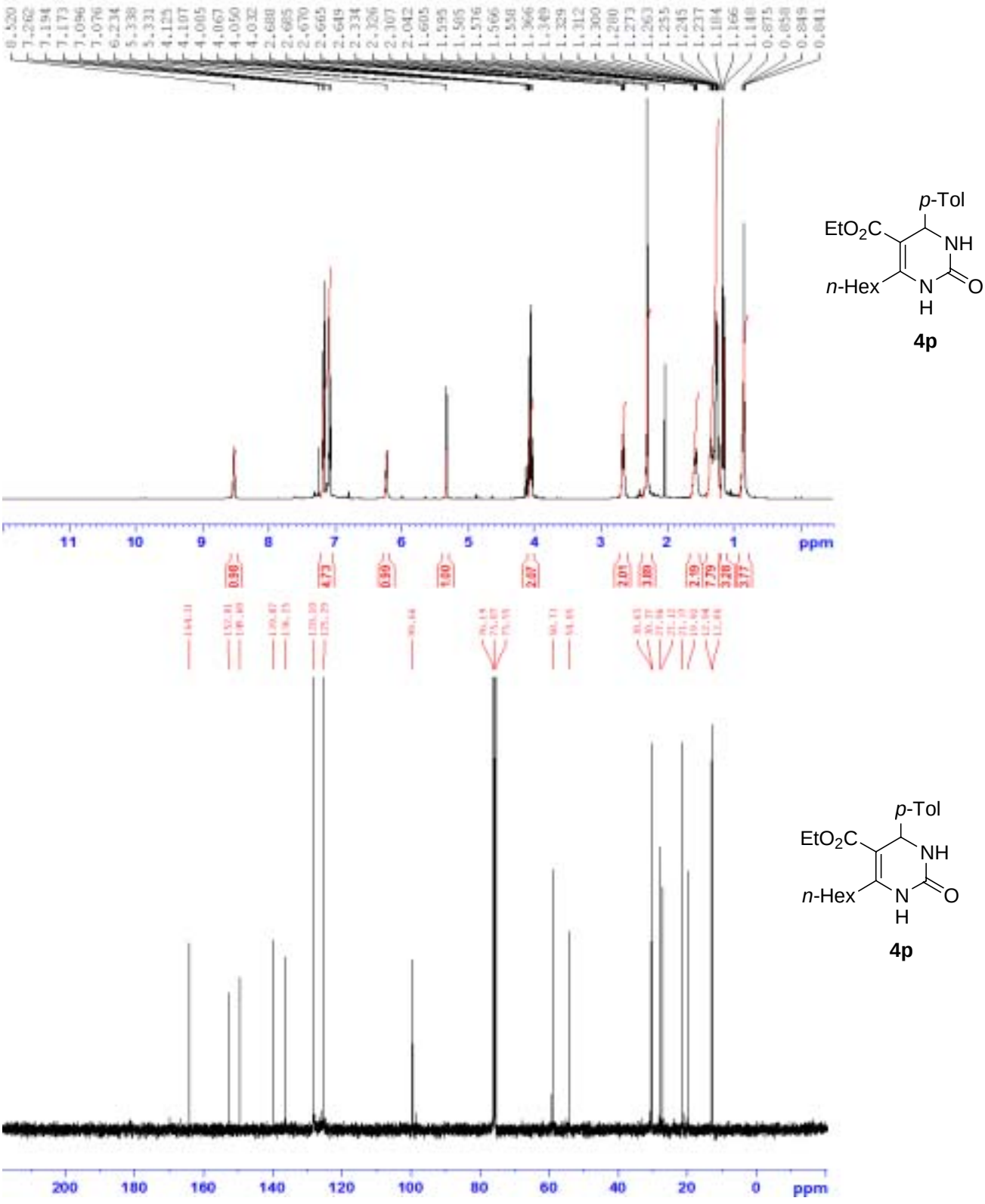


4o



4o

Fig. S18. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-hexyl-4-(4-methylphenyl)-2-oxo-, ethyl ester (4p)



5

Fig. S19. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-phenyl-6-(2-phenylethyl)-2-oxo-, ethyl ester (4q)

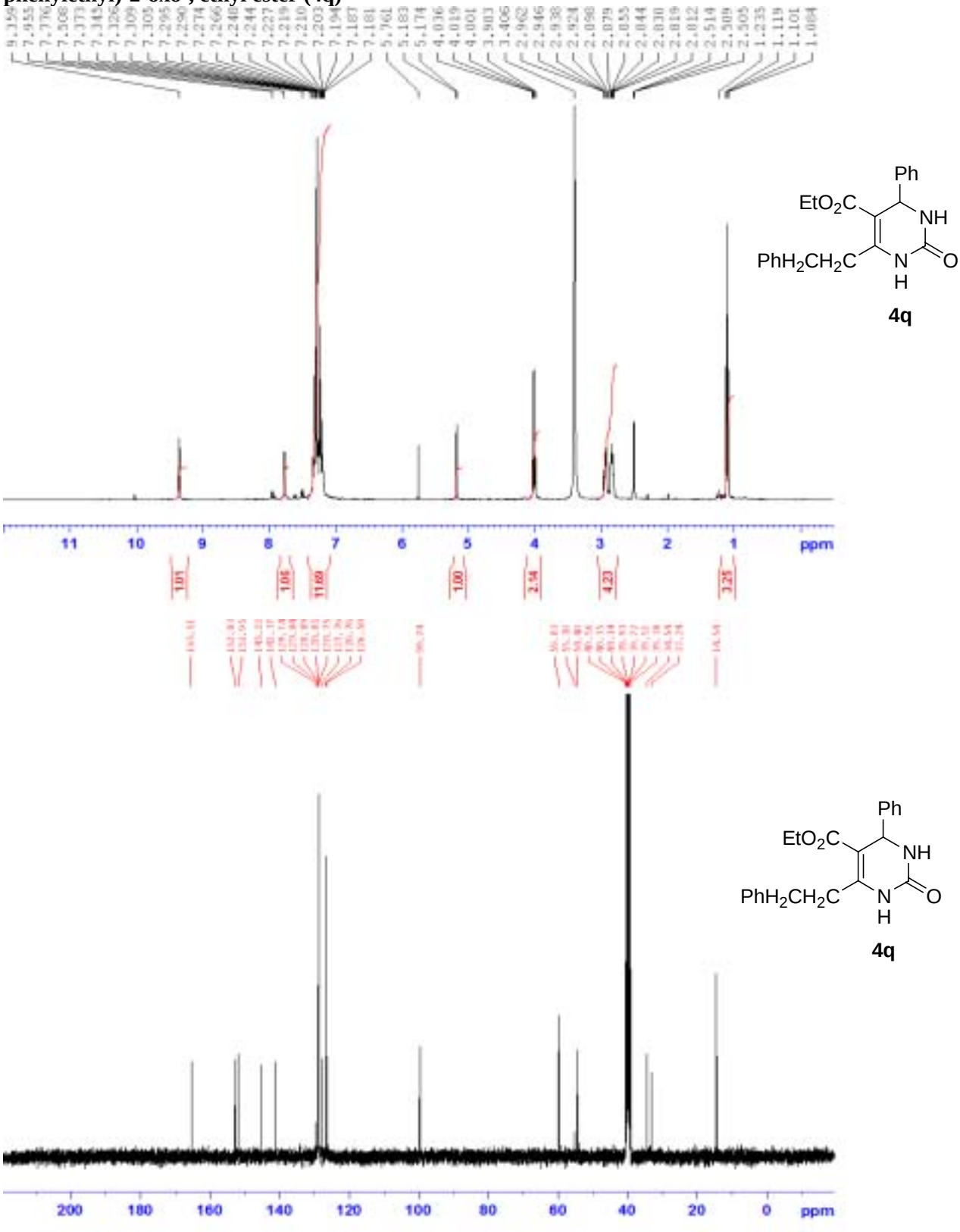


Fig. S20. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-cyclohexyl-4-(4-methylphenyl)-2-oxo-, ethyl ester (4s)

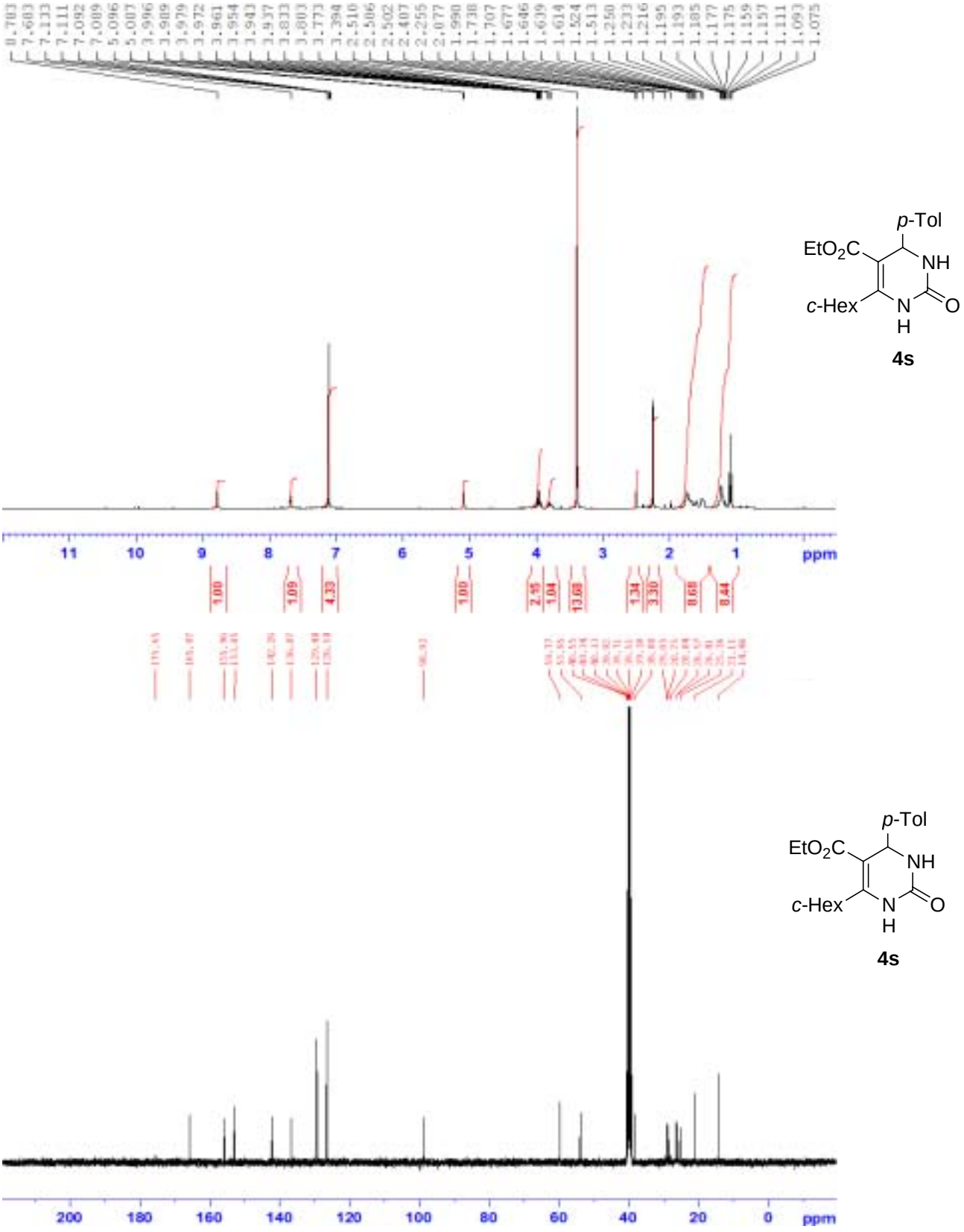
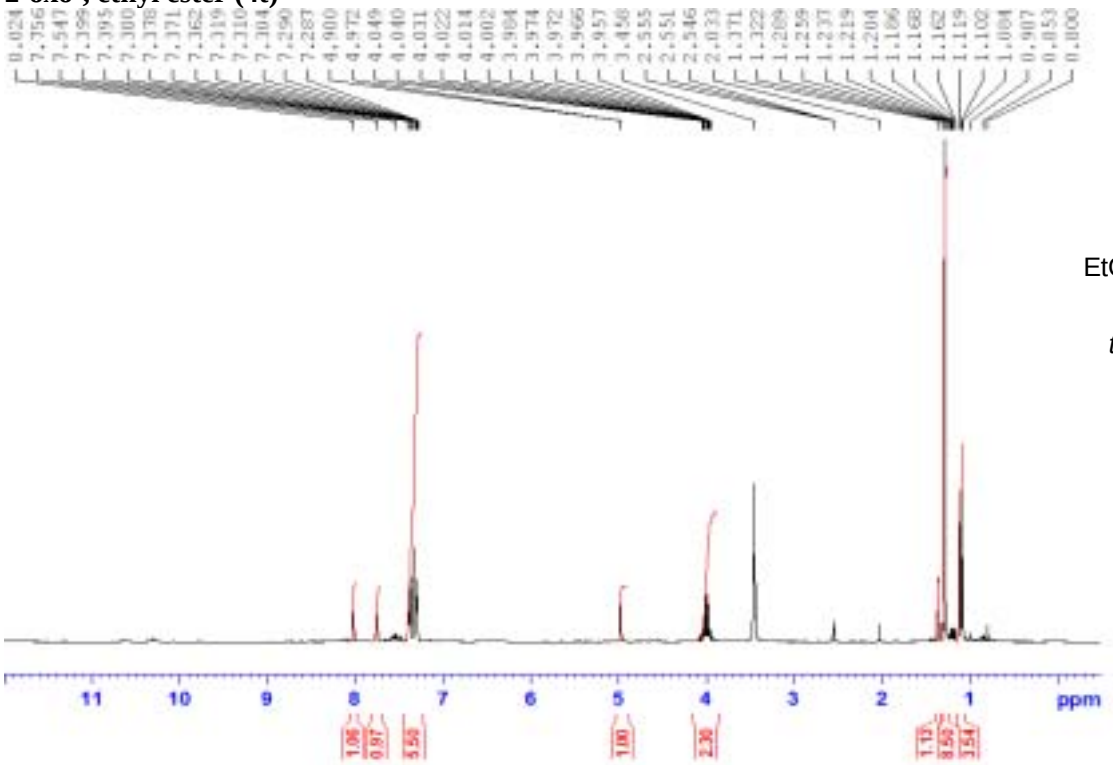
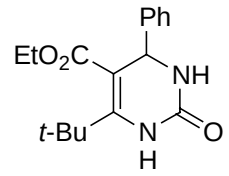
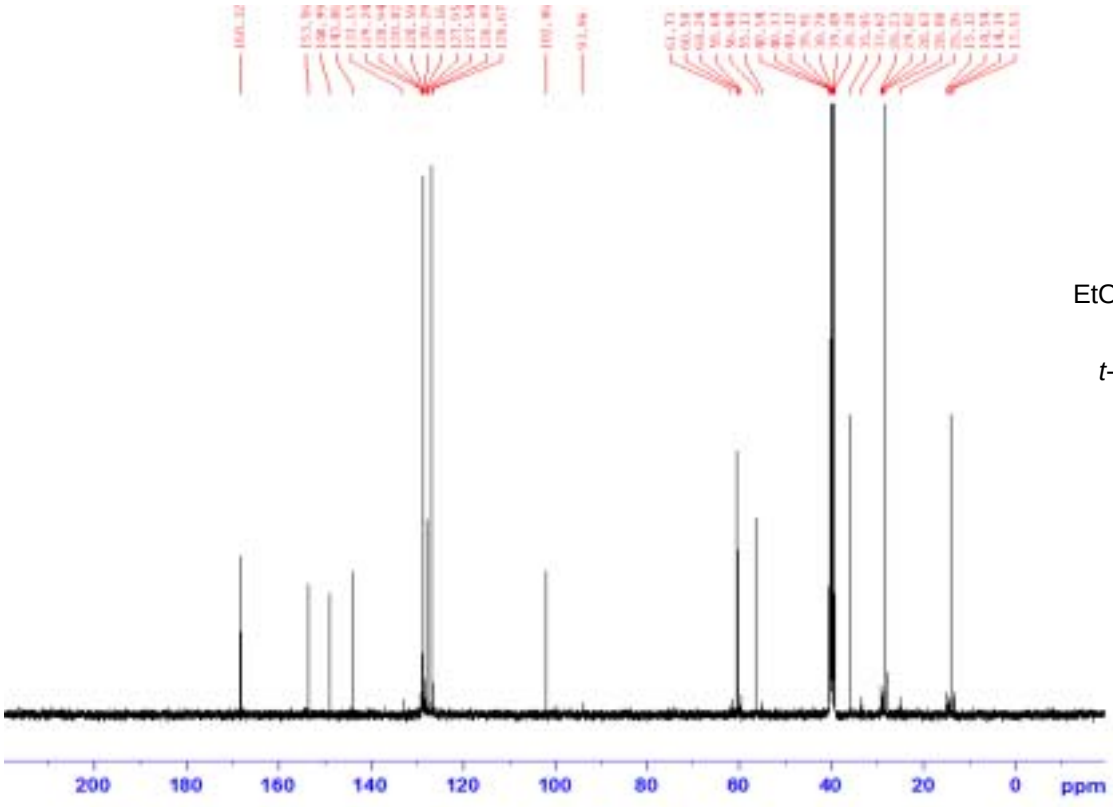


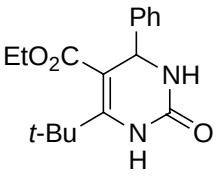
Fig. S21. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-*t*-butyl-4-phenyl-2-oxo-, ethyl ester (4t)





4t





4t

Fig. S22. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-(1-heptenyl)-4-(4-methylphenyl)-2-oxo-, ethyl ester (4u)

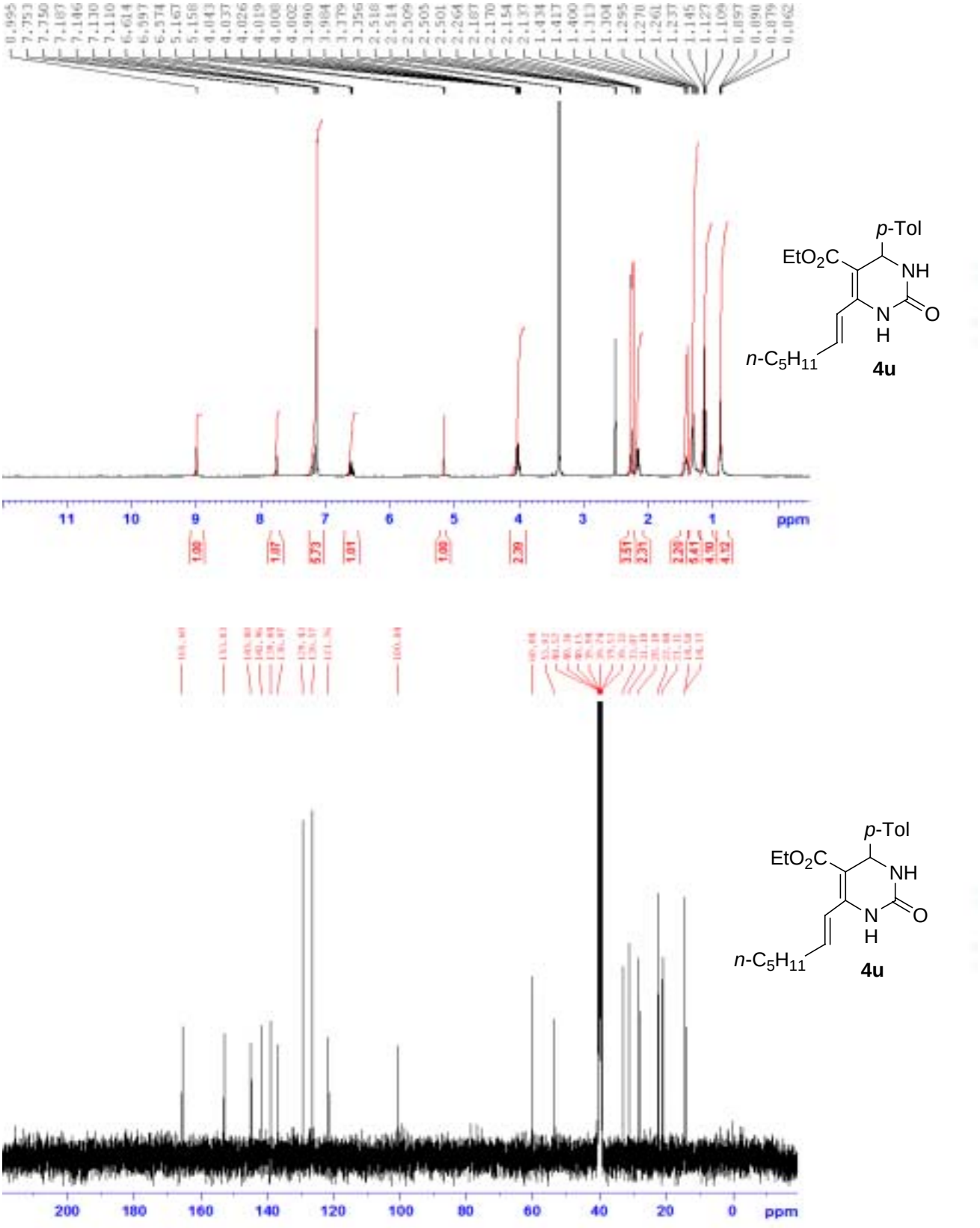


Fig. S23. ^{1}H and ^{13}C NMR spectra of 5-pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-6-cyclohexyl-4-(4-methylphenyl)-2-oxo-, ethyl ester (4v)

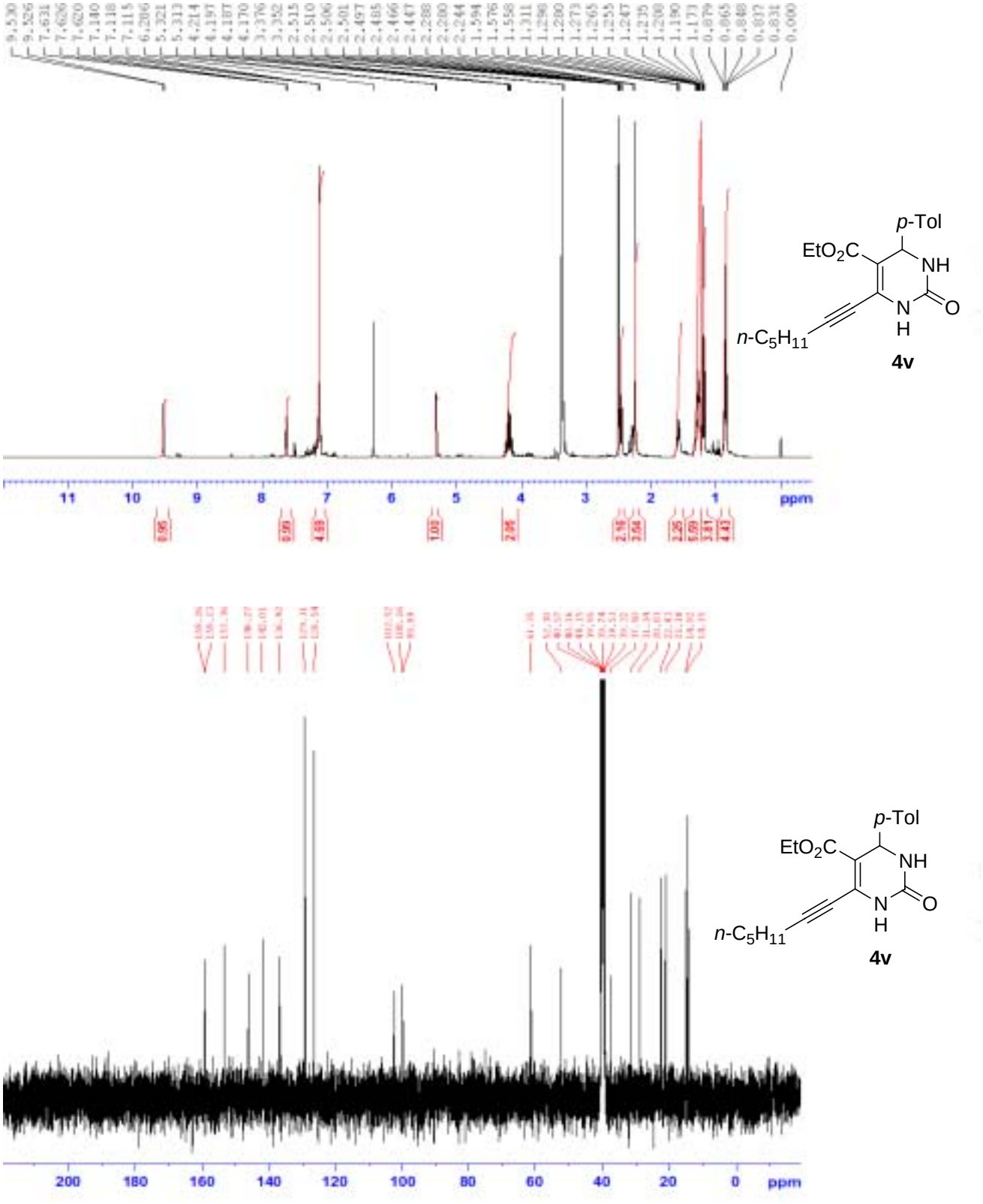
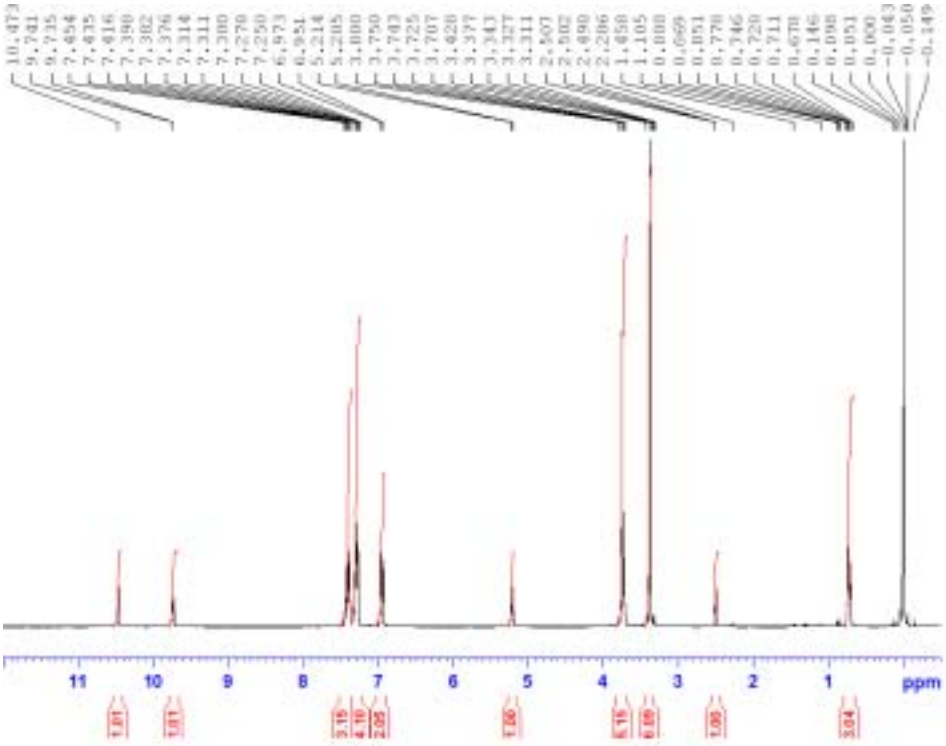
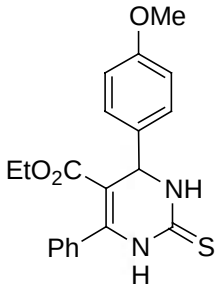
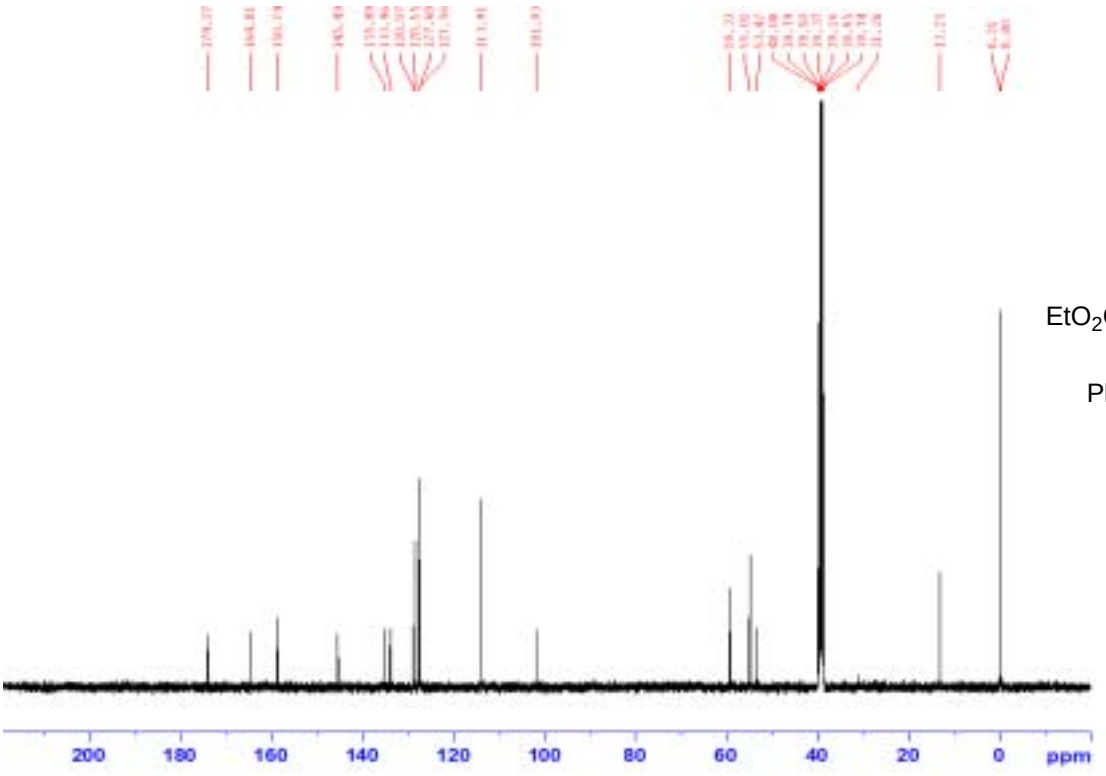


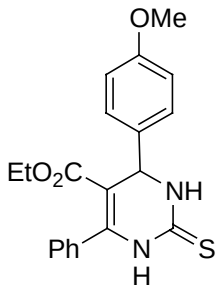
Fig. S24. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methoxyphenyl)-6-phenyl-2-thioxo-, ethyl ester (4w)





4w





4w

Fig. S25. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methylphenyl)-2-oxo-6-phenyl-, ethyl ester (4x)

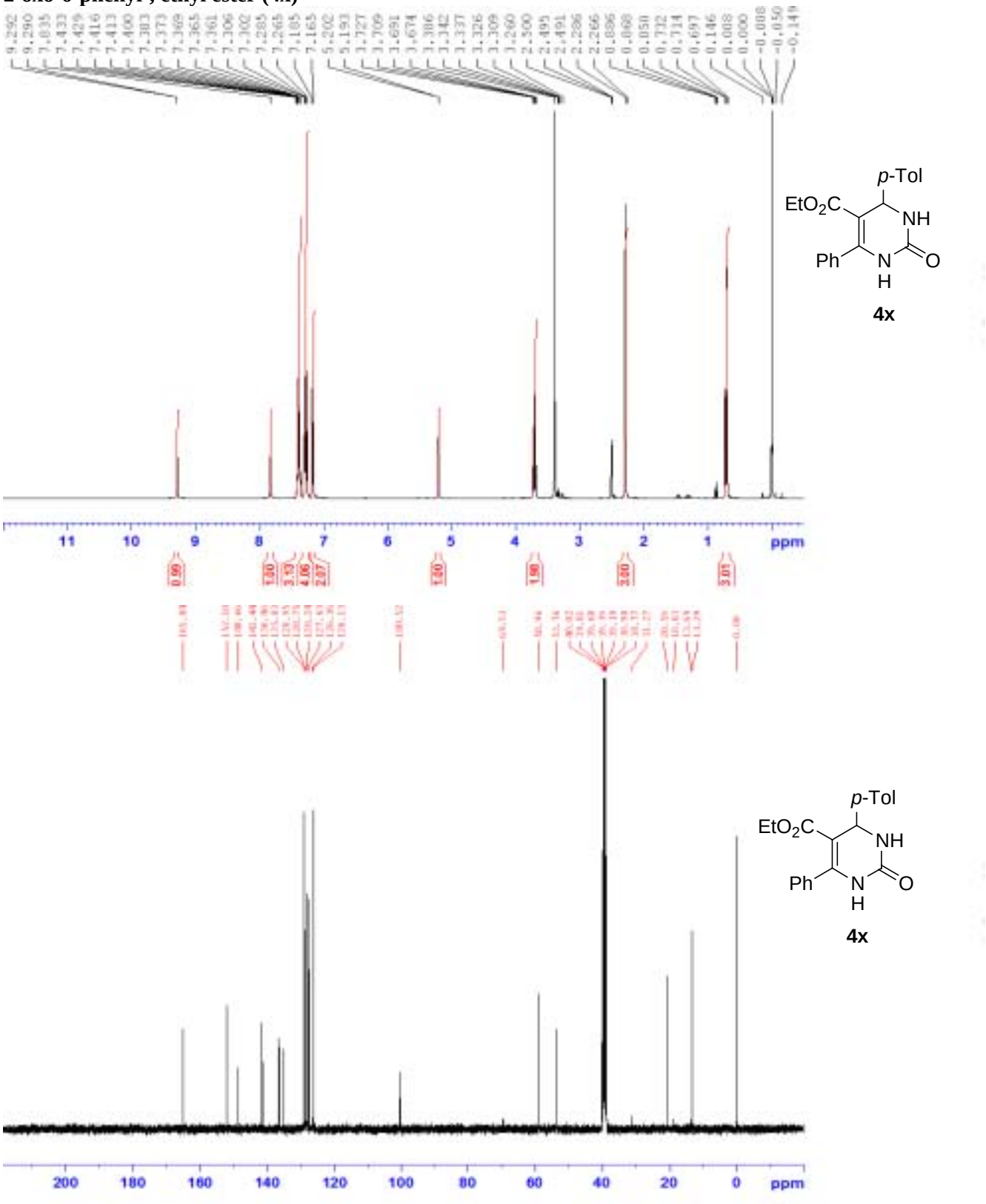


Fig. S26. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methylphenyl)-6-phenyl-2-thioxo-, ethyl ester (4y)

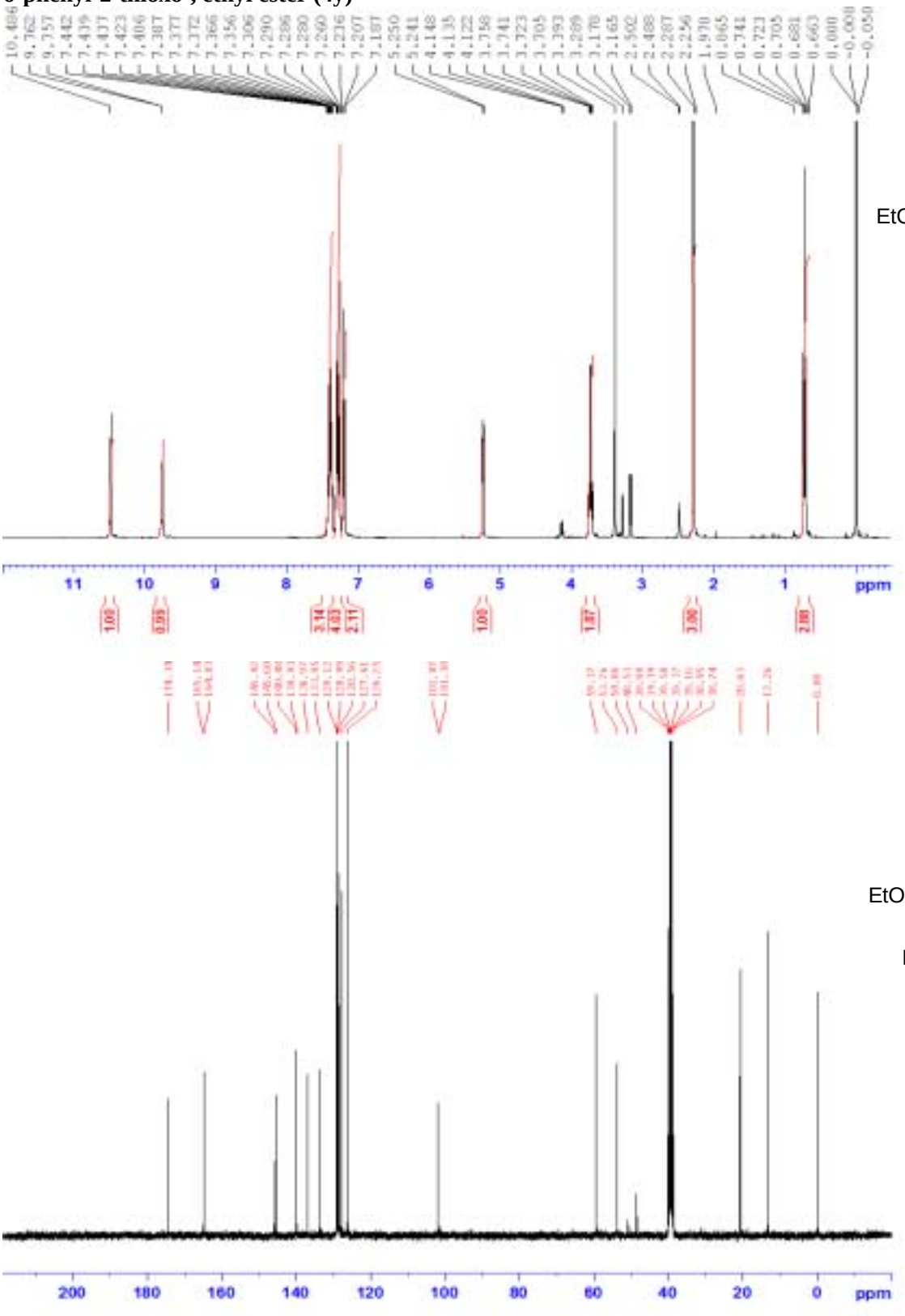
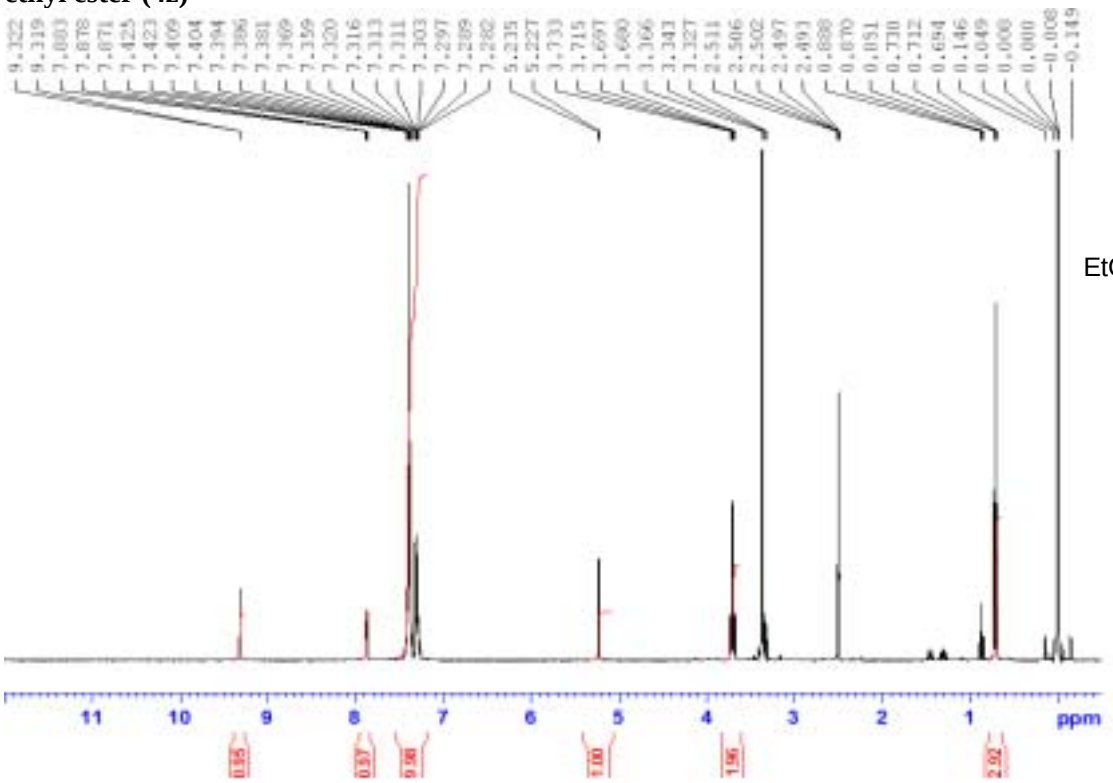
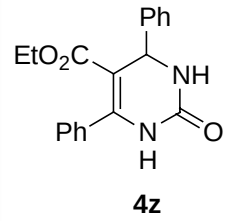
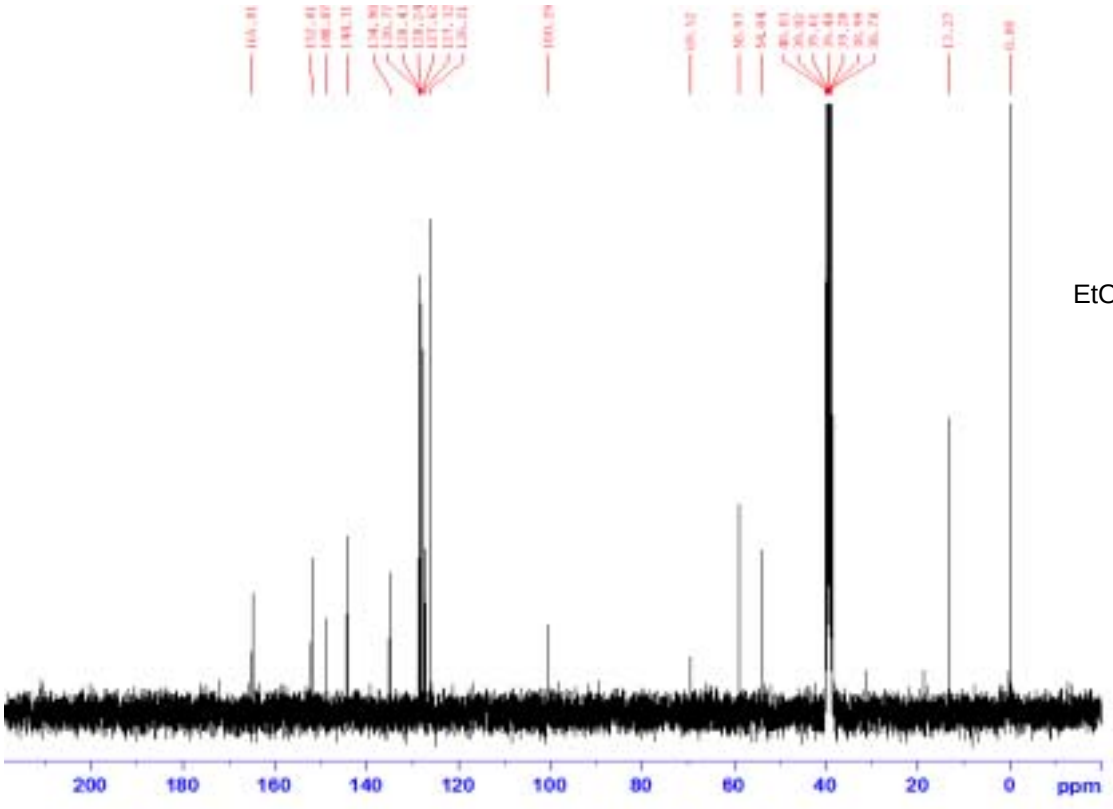


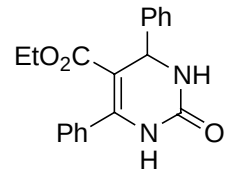
Fig. S27. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-2-oxo-4,6-diphenyl-, ethyl ester (4z)





4z





4z

Fig. S28. ^{1}H and ^{13}C NMR spectra of 5-Pyrimidinecarboxylic acid, 1,2,3,4-tetrahydro-4-(4-methoxyphenyl)-6-phenyl-2-thioxo-, ethyl ester (4aa)

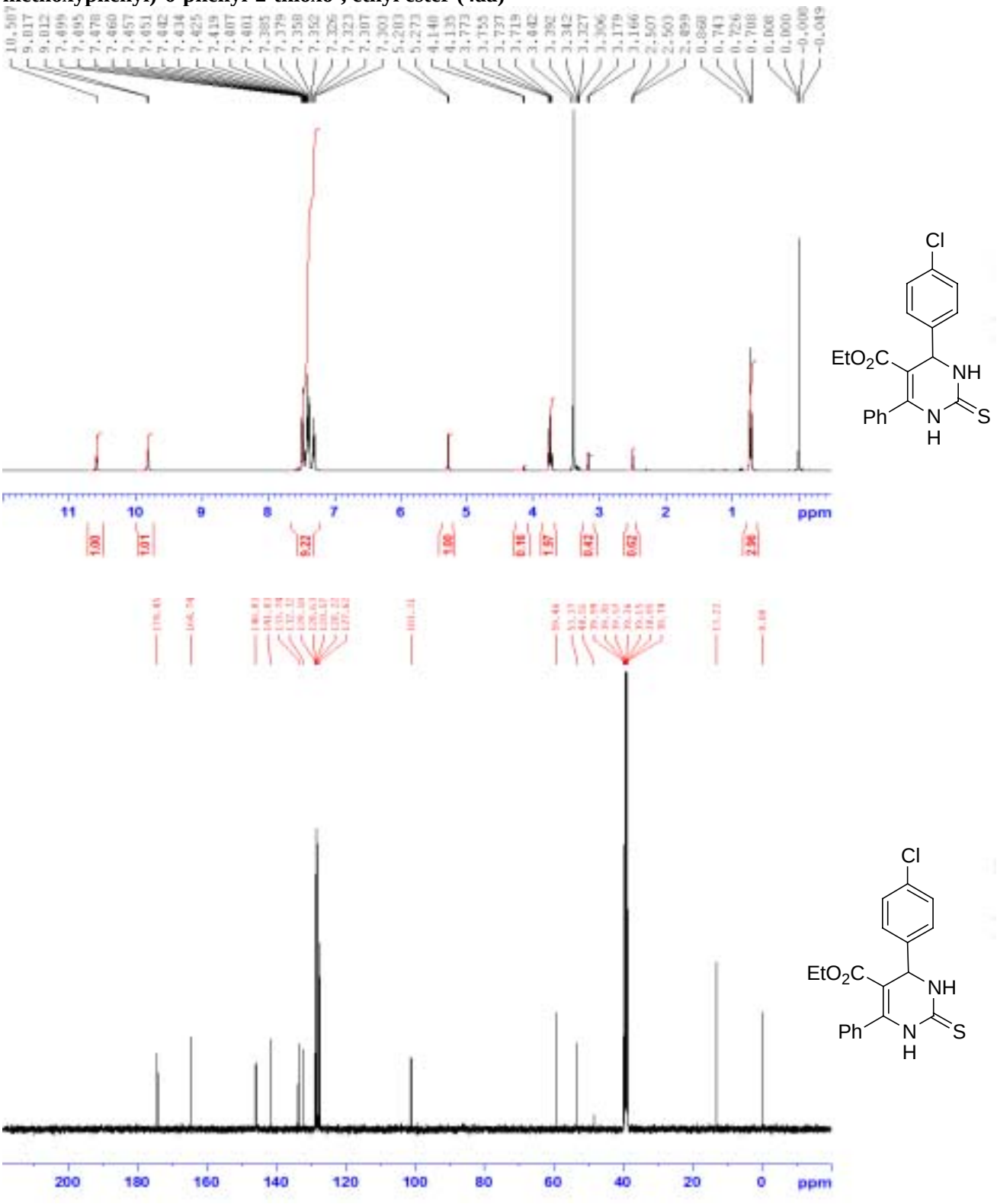


Figure S29. ^{1}H and ^{13}C NMR spectra of Hexanoic acid, 2-[(4-methylphenyl)methylene]-3-oxo-, ethyl ester (*Ca. 55/45 E/Z* mixture) (5a)