CHAPTER IV

CONCLUSION

3-Hydroxy-5-methoxy-1-methyl-2-piperidone(48), an isomer of odoram, was synthesized starting from L-glutamic acid(40). The synthesis was carried out by deamination and cyclization of L-glutamic acid to form 5-ethoxycarbonyl-2-tetrahydrofuranone (41), a lactone containing a carboxylic acid group. The carboxylic acid group of (41) was converted to alcohol: 5-hydroxymethyl-2-tetrahydrofuranone (42), tosylate: 5-tosyloxy-methyl-2-tetrahydrofuranone(43), and azide: 5-azidomethyl-2-tetrahydrofuranone(44), respectively. Catalytic hydrogenation of the azido lactone (44) gave a six-membered ring lactam, 5-hydroxy-2-piperidone(45), then methylation and α-hydroxylation yielded 3-hydroxy-5-methoxy-1-methyl-2-piperidone(48a) of which the configuration of C-3 (the mixture of $R_{(equatorial)}$- and $S_{(axial)}$-configuration) and C-5 ($S_{(equatorial)}$-configuration) were not identical with ones of odoram which the configuration of C-3 was $R_{(equatorial)}$- and C-5 was $R_{(axial)}$-configuration. Therefore, the confirmation of the configuration of odoram could not be achieved by the synthesis of 3-hydroxy-5-methoxy-1-methyl-2-piperidone(48)

The synthesis of odoram from other starting material namely pentanedioic acid(49) was also attempted. The α-bromination and further hydroxylation yielded 2,4-dihydroxy-

pentanedioic acid(51). Lactonization of hydroxy acid (51) gave lactone, 5-carboxy-3-hydroxy-2-tetrahydrofuranone(52), but methylation of the carboxy lactone(52) caused ring-fission. Therefore odoram and/or its derivative could not be prepared by the method starting from pentanedioic acid.

Some new compounds were synthesized from the reaction routes proposed, i.e. 5-methoxy-1-methyl-2-piperidone (47a) and 3-hydroxy-5-methoxy-1-methyl-2-piperidone (48a), and their spectral data were assigned. The specral data of some known compounds which were not previously reported, i.e. the ^{13}C NMR spectra of each compound, the spectral data of compound (45), (50), (52), and (53a), were also assigned.

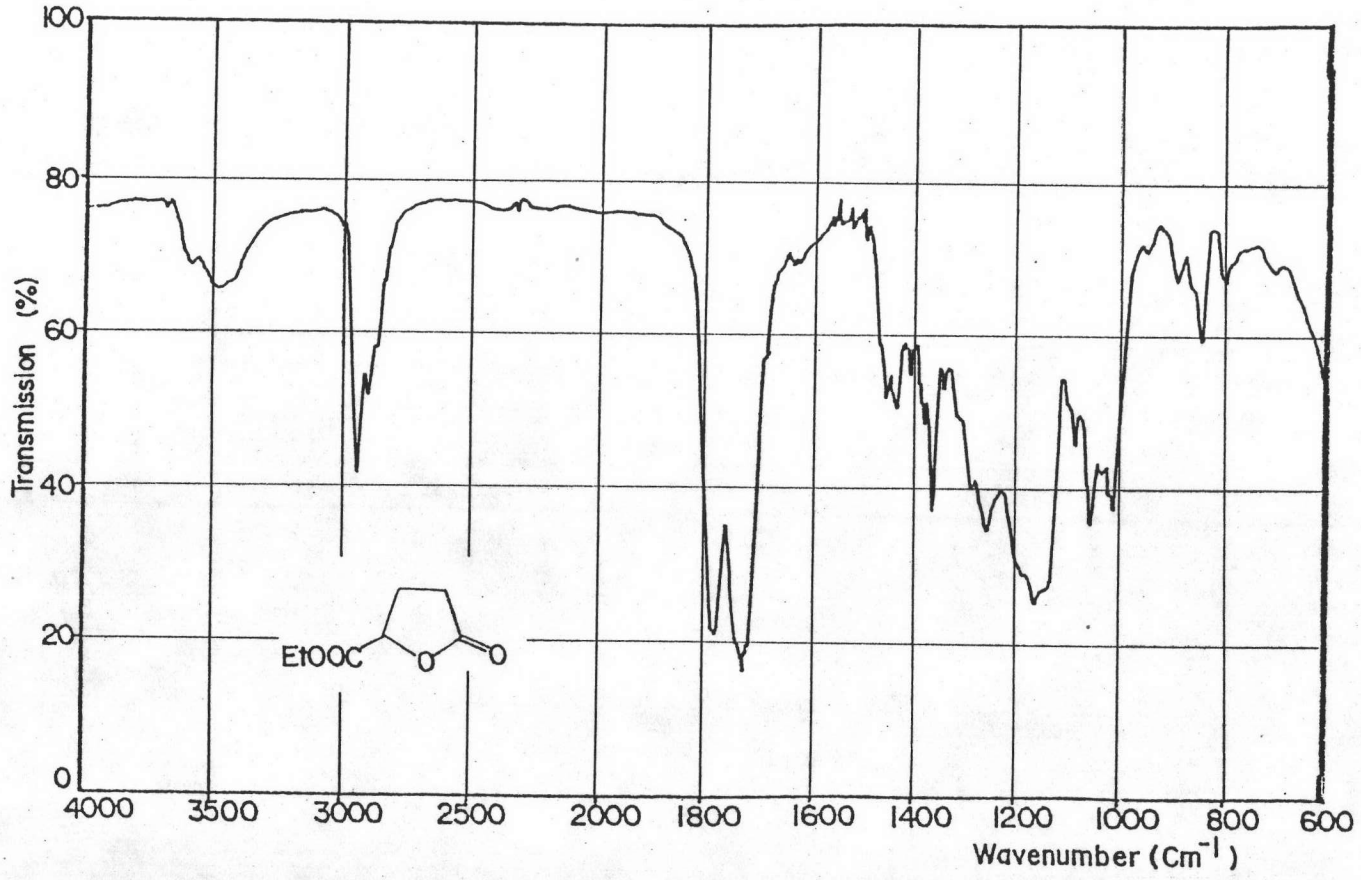

Figure 1 The IR spectrum of 5-ethoxycarbonyl-2-tetrahydrofuranone(41)

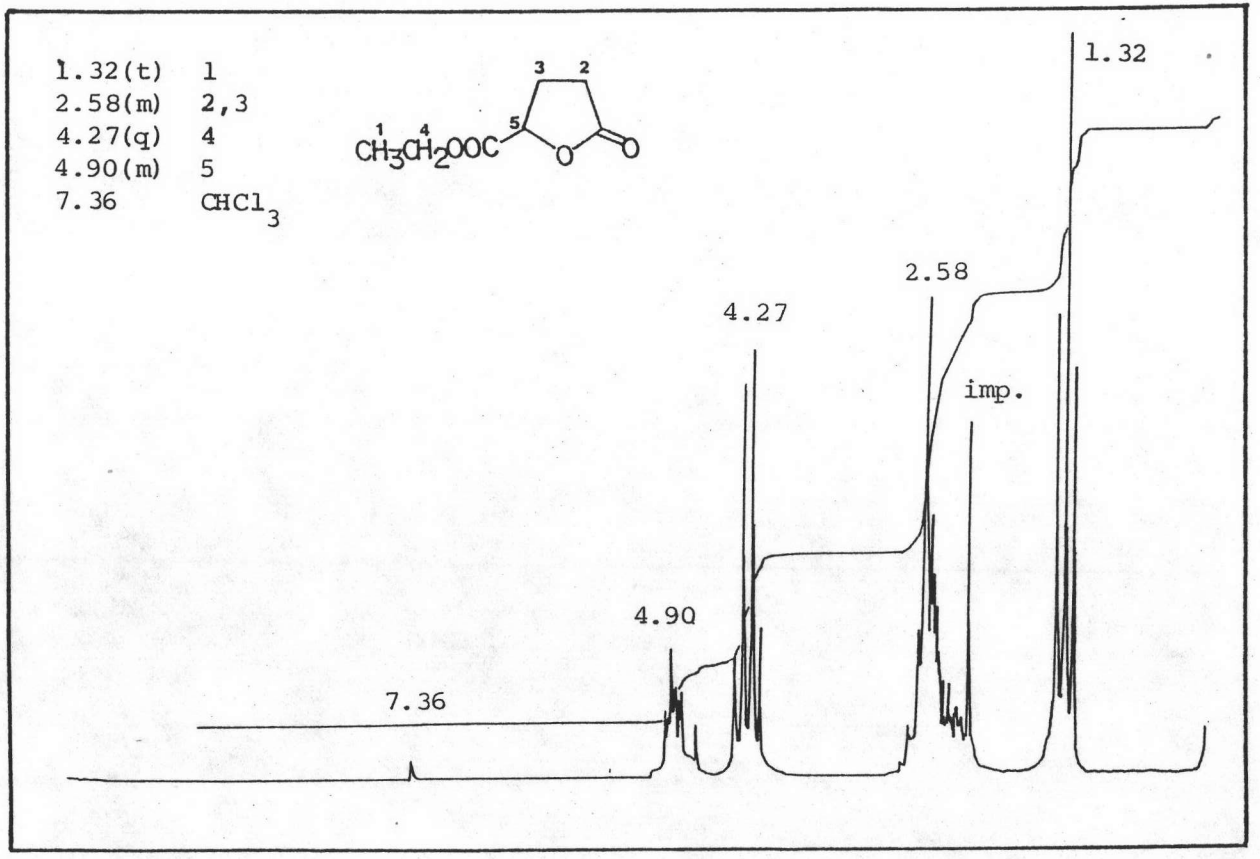

Figure 2 The ^1H NMR spectrum of 5-ethoxycarbonyl-2-tetrahydrofuranone (41)

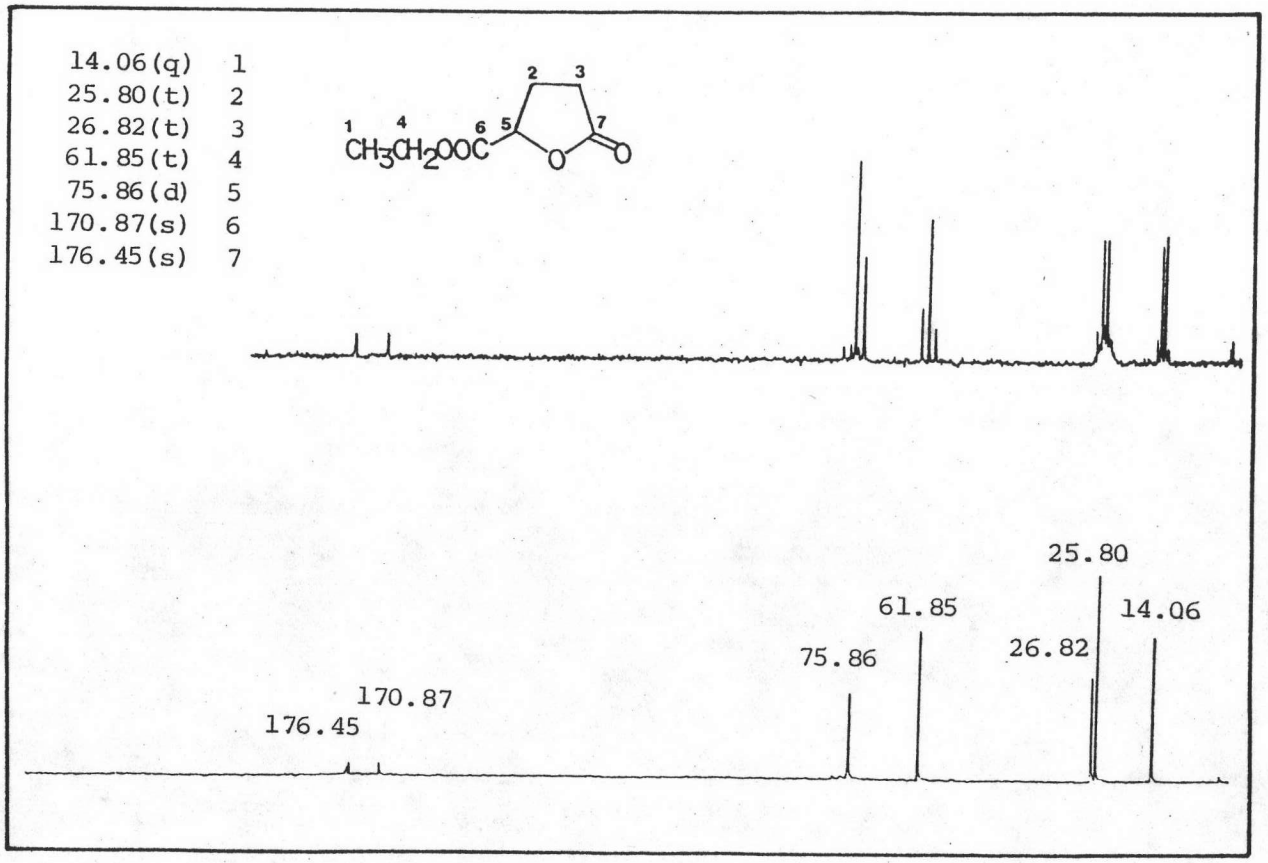

Figure 3 The ^{13}C NMR spectrum of 5-ethoxycarbonyl-2-tetrahydrofuranone (41)

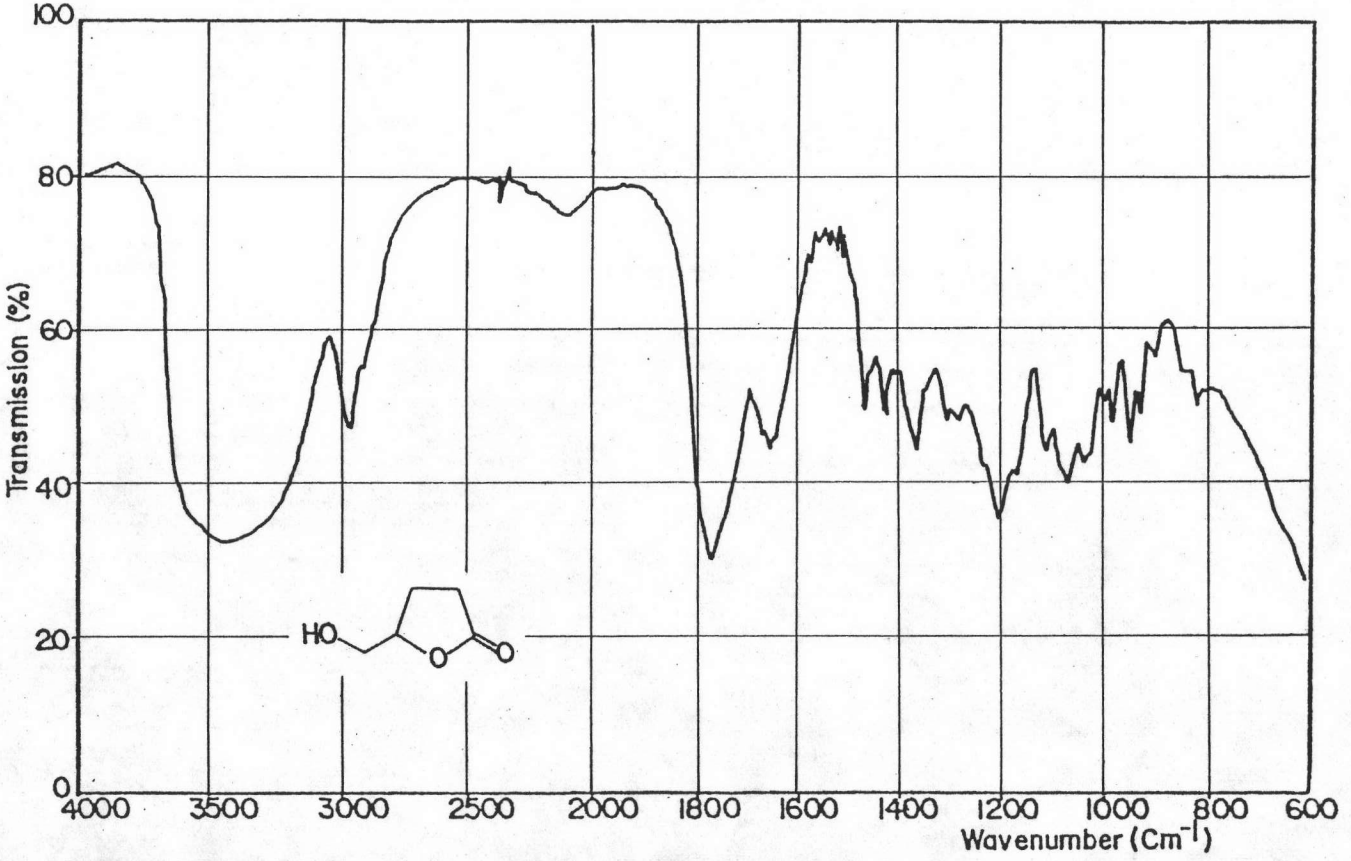

Figure 4 The IR spectrum of 5-hydroxymethyl-2-tetrahydrofuranone(42)

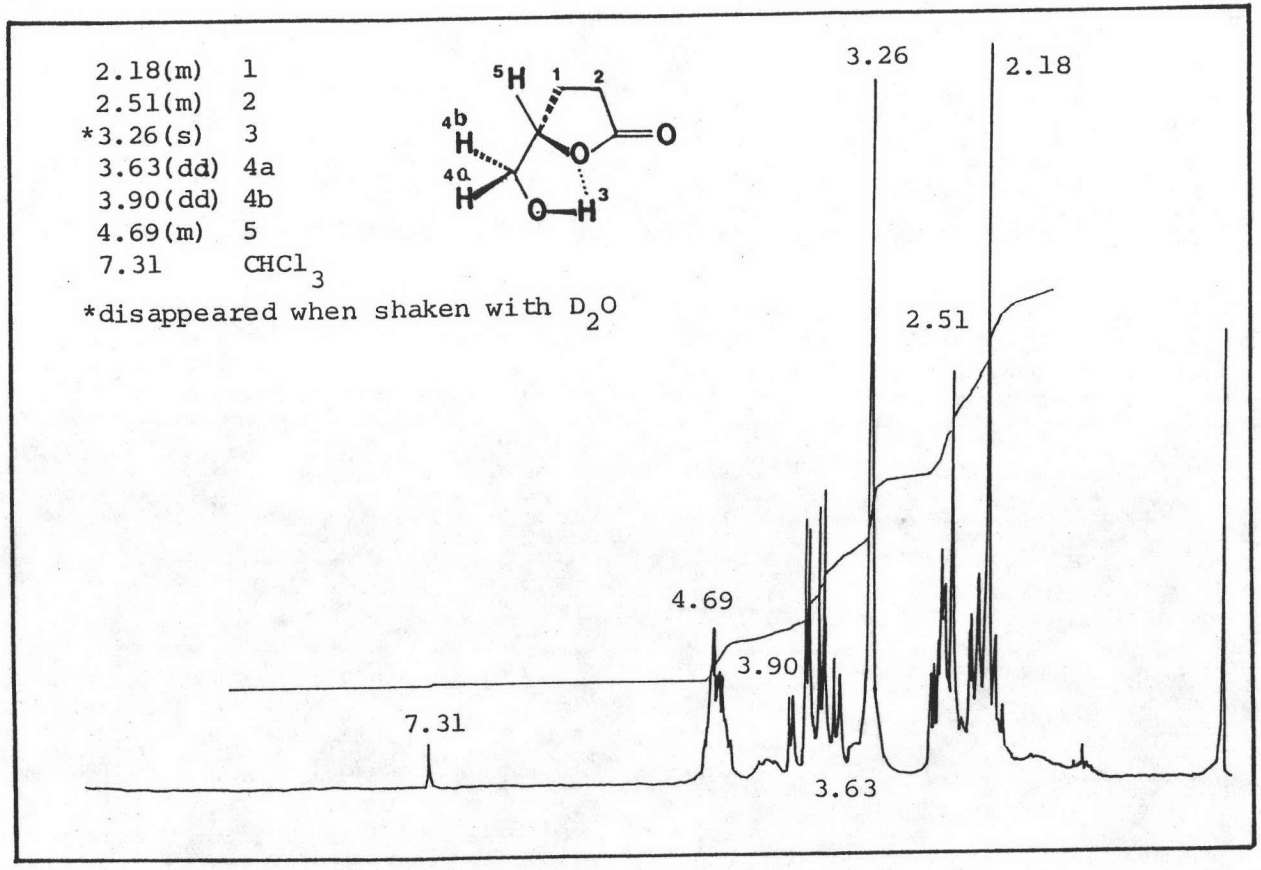

Figure 5 The ^1H NMR spectrum of 5-hydroxymethyl-2-tetrahydrofuranone (42)

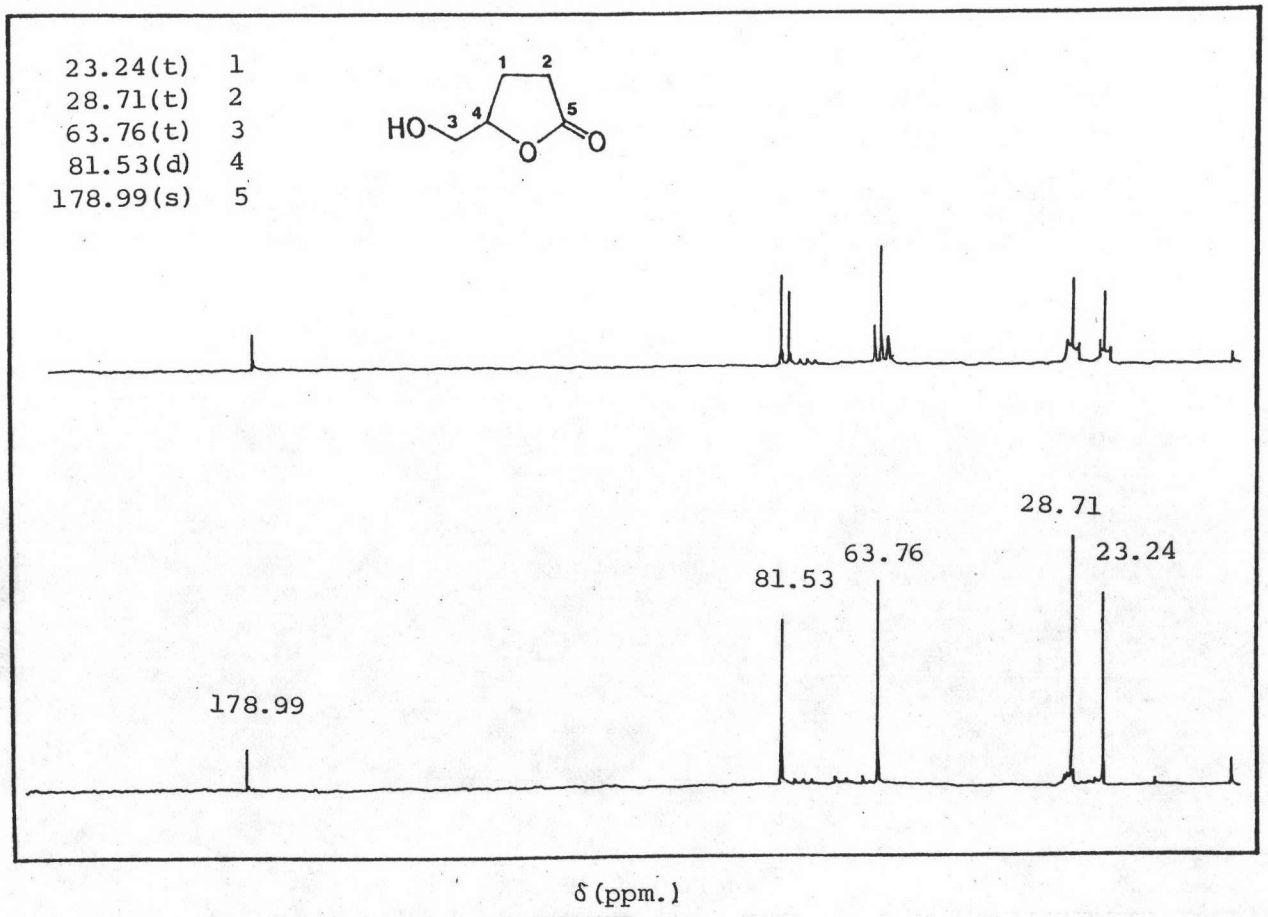

Figure 6 The ^{13}C NMR spectrum of 5-hydroxymethyl-2-tetrahydrofuranone (42)

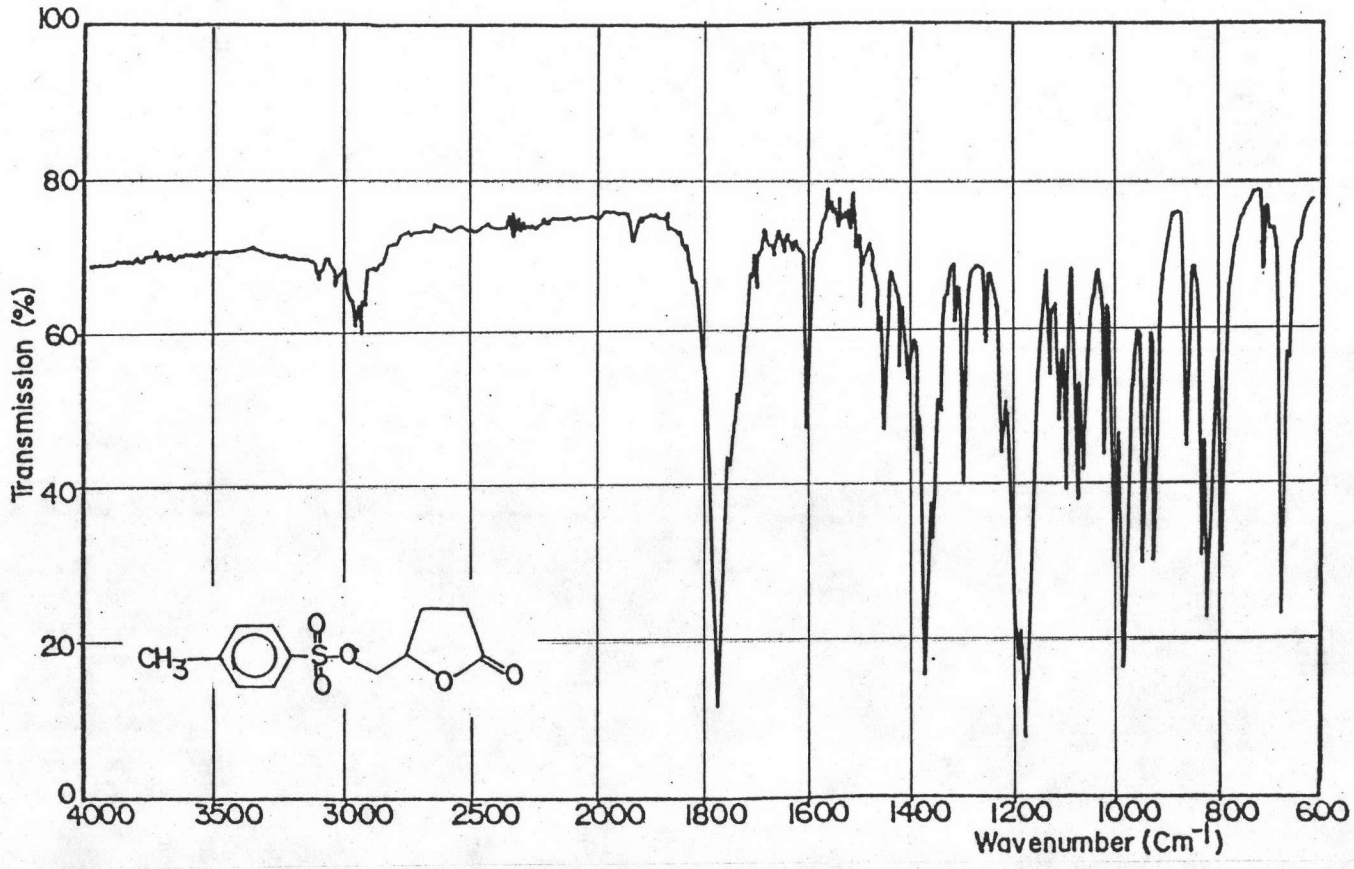

Figure 7 The IR spectrum of 5-tosyloxymethyl-2-tetrahydrofuranone(43)

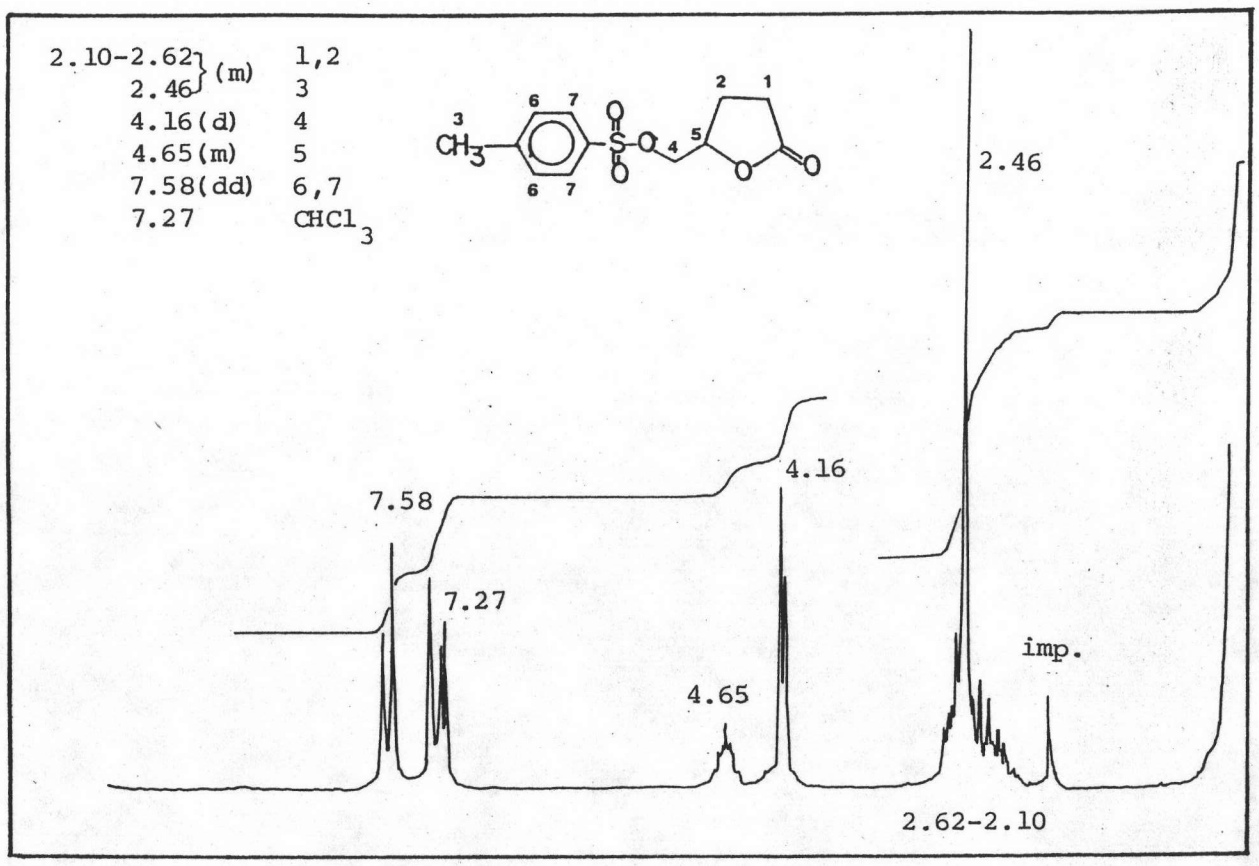

Figure 8 The ^{1}H NMR spectrum of 5-tosyloxymethyl-2-tetrahydrofuranone (43)

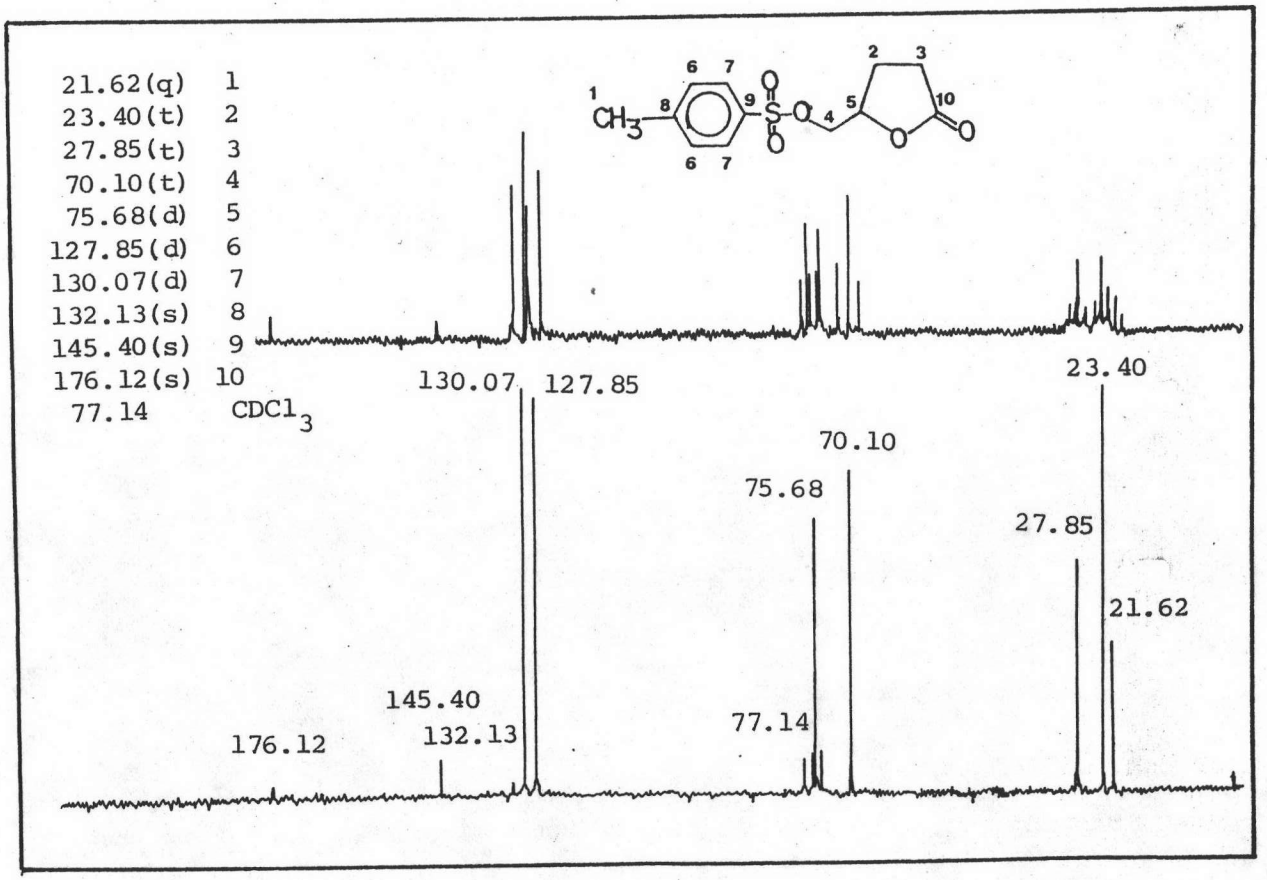

Figure 9 The ^{13}C NMR spectrum of 5-tosyloxymethyl-2-tetrahydrofuranone (43)

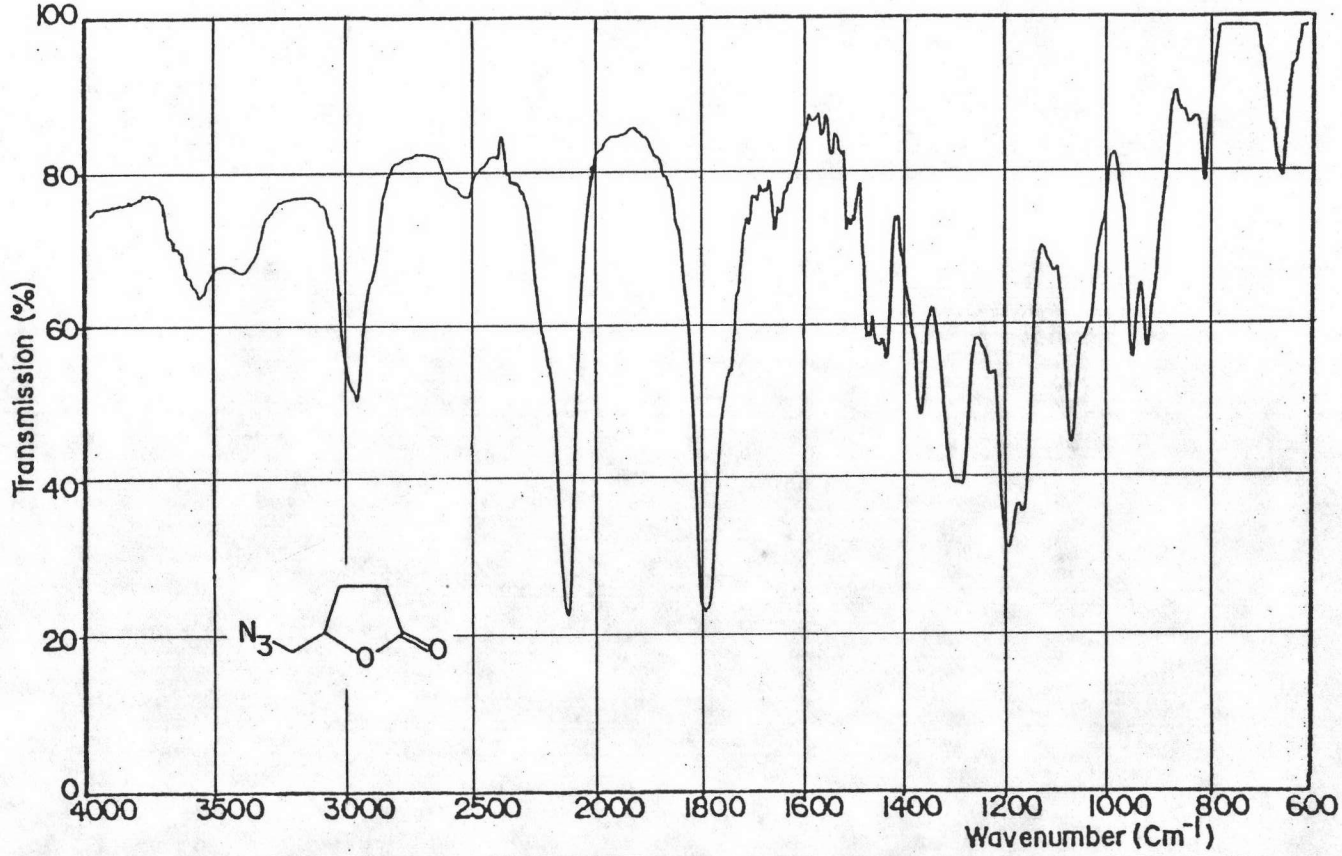

Figure 10 The IR spectrum of 5-azidomethyl-2-tetrahydrofuranone(44)

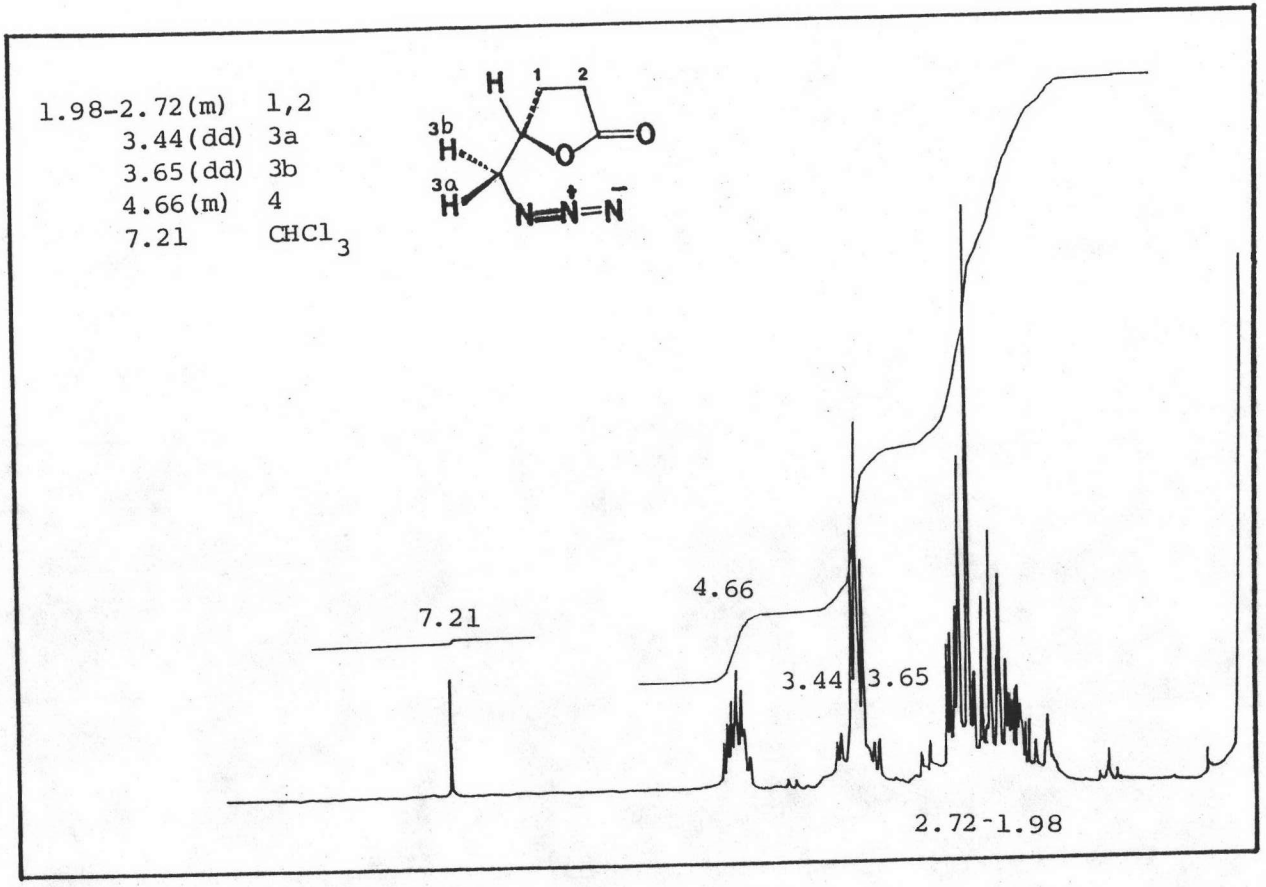

Figure 11 The ^{1}H NMR spectrum of 5-azidomethyl-2-tetrahydrofuranone(44)

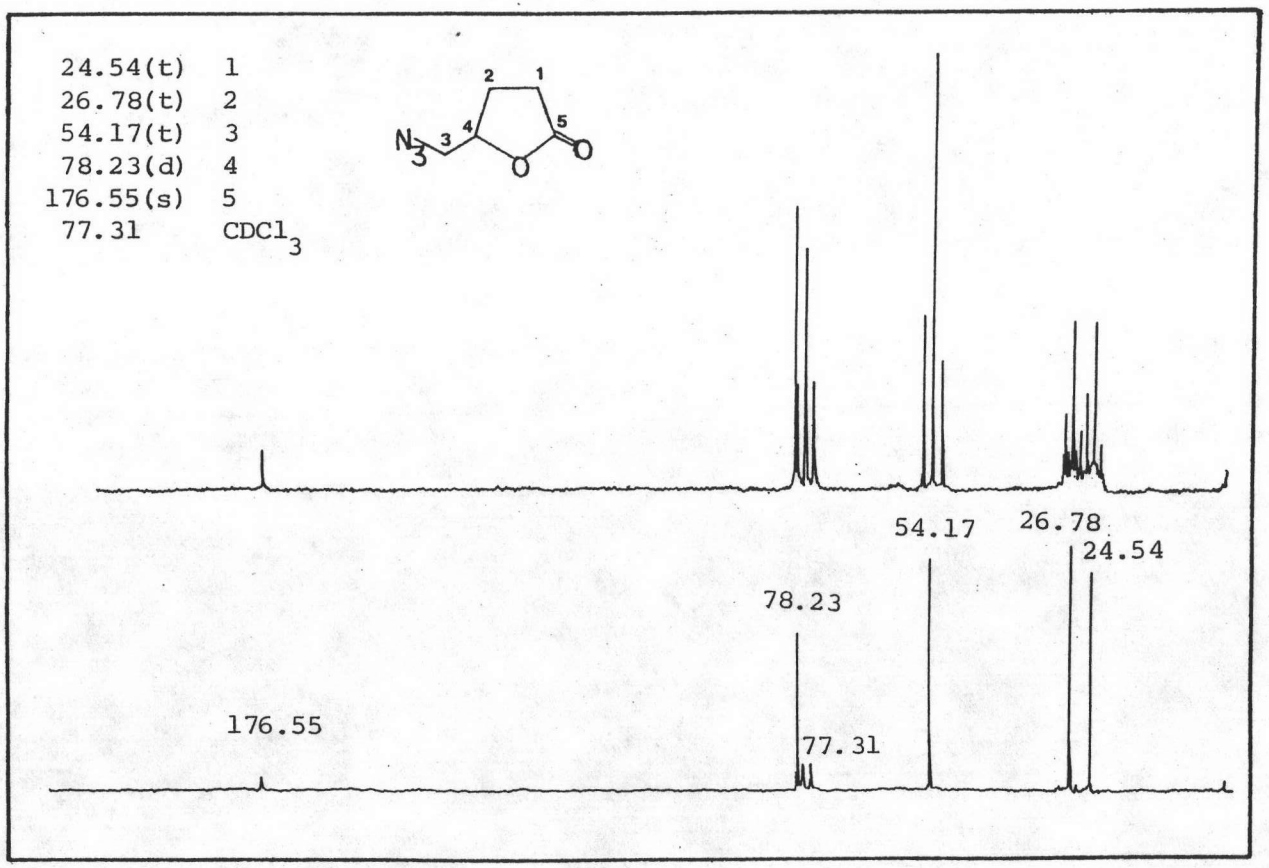

Figure 12 The ^{13}C NMR spectrum of 5-azidomethyl-2-tetrahydrofuranone (44)

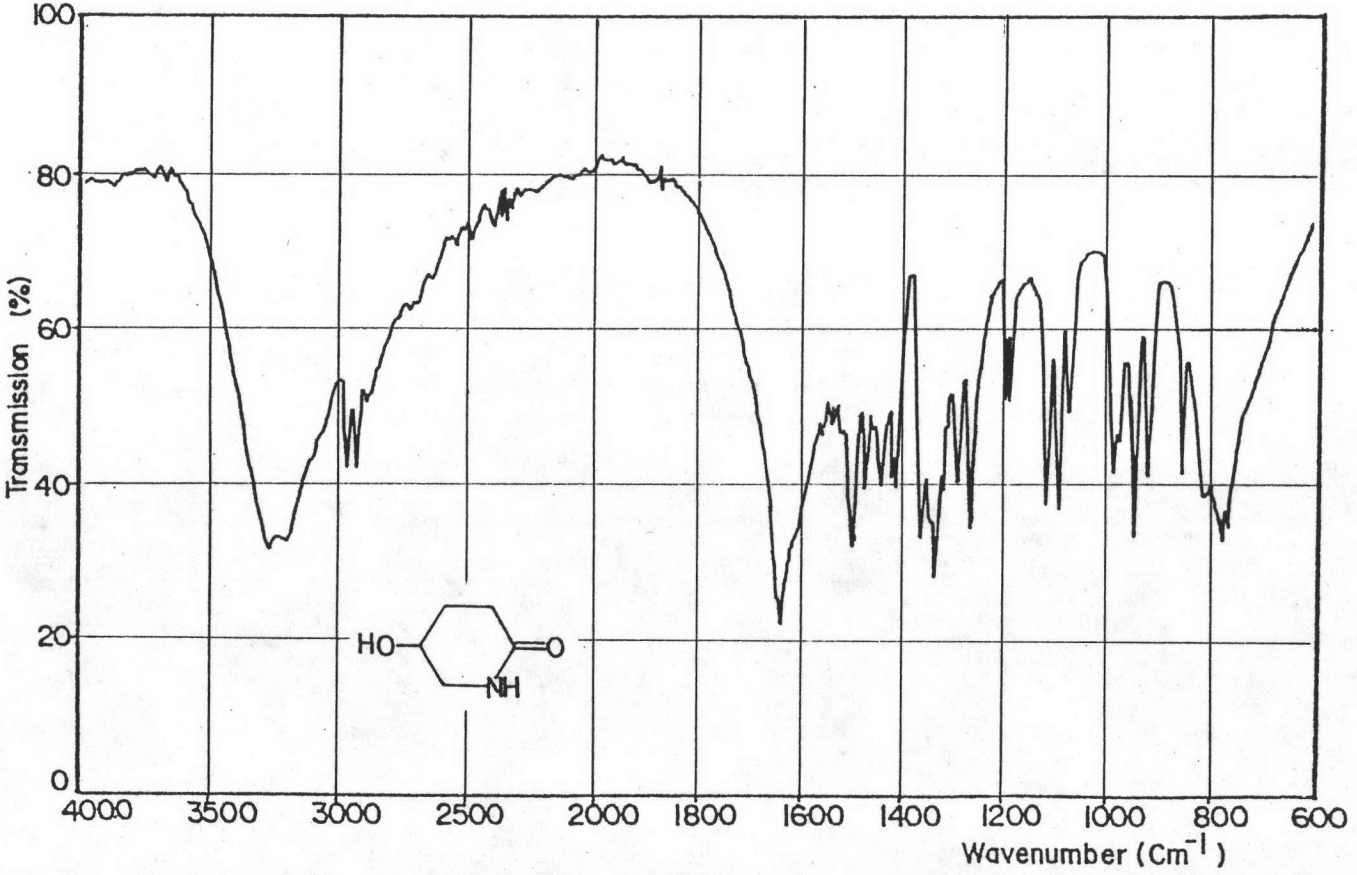

Figure 13 The IR spectrum of 5-hydroxy-2-piperidone(45)

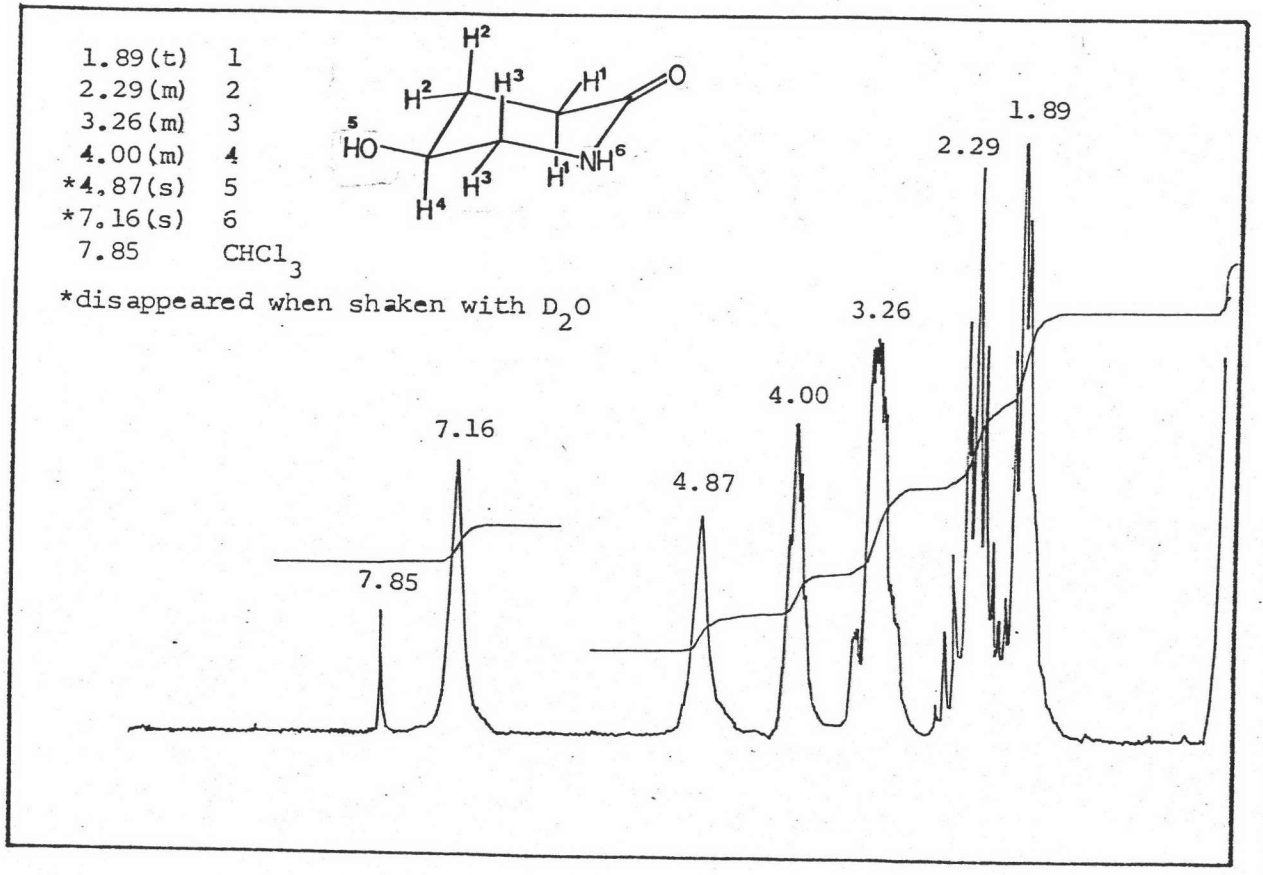

Figure 14 The ^{1}H NMR spectrum of 5-hydroxy-2-piperidone(45)

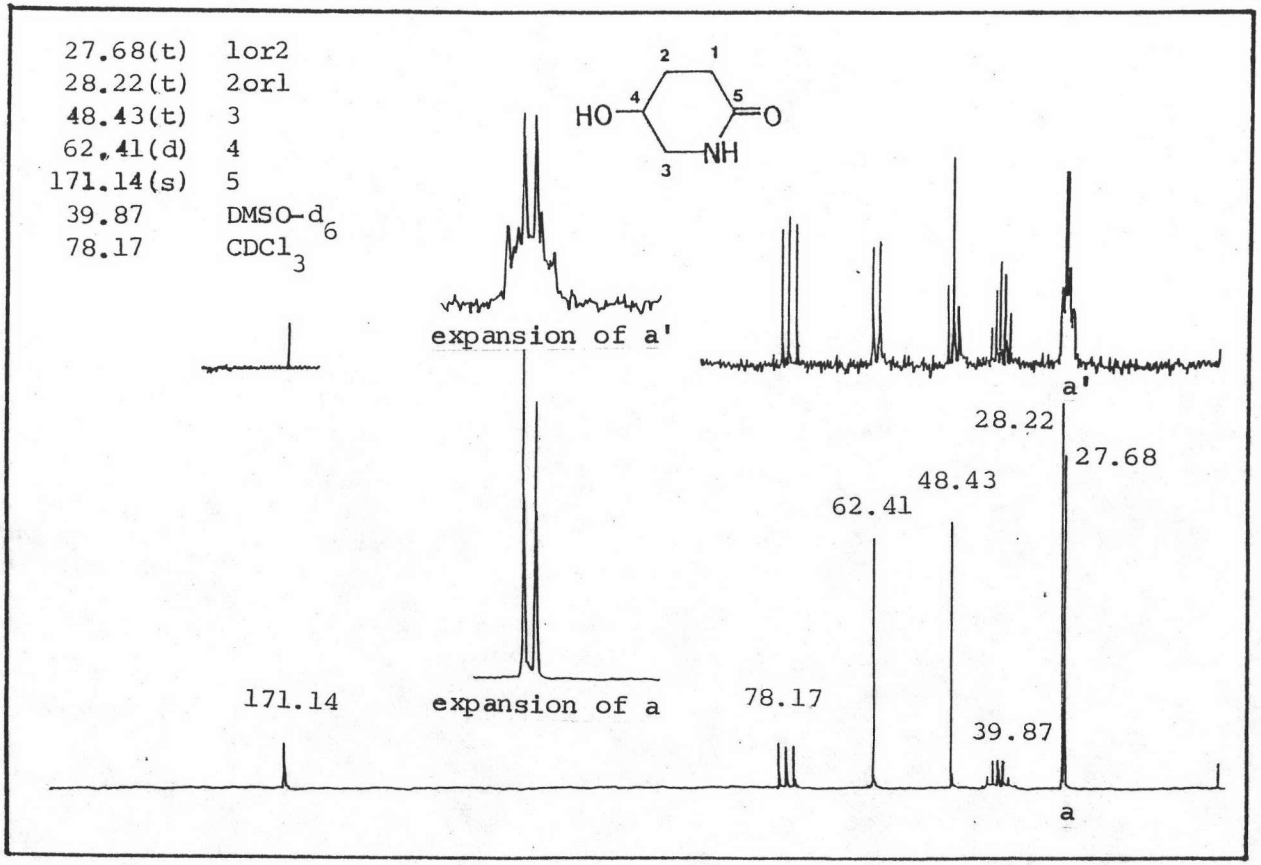

Figure 15 The ^{13}C NMR spectrum of 5-hydroxy-2-piperidone(45)

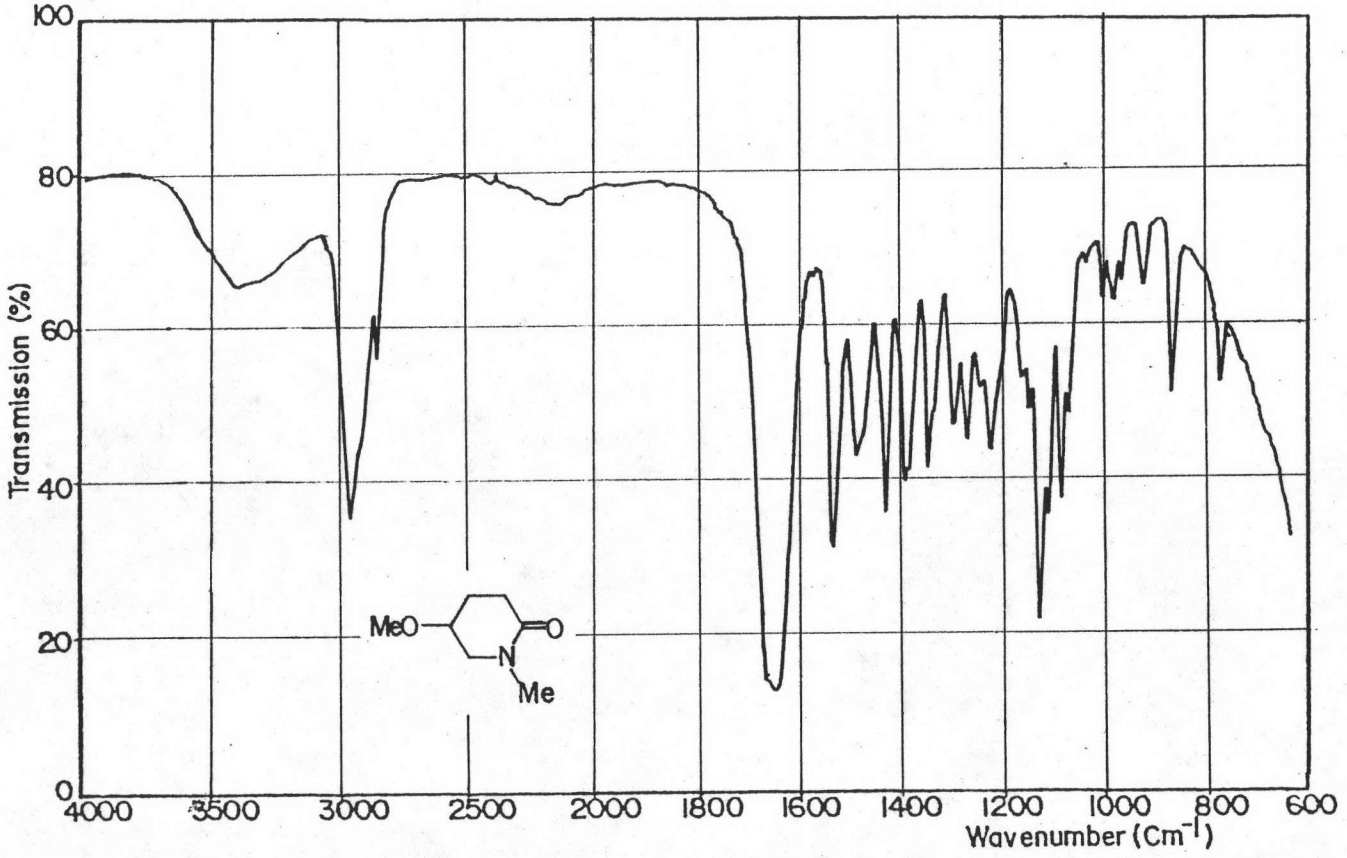

Figure 16 The IR spectrum of 5-methoxy-1-methyl-2-piperidone(47a)

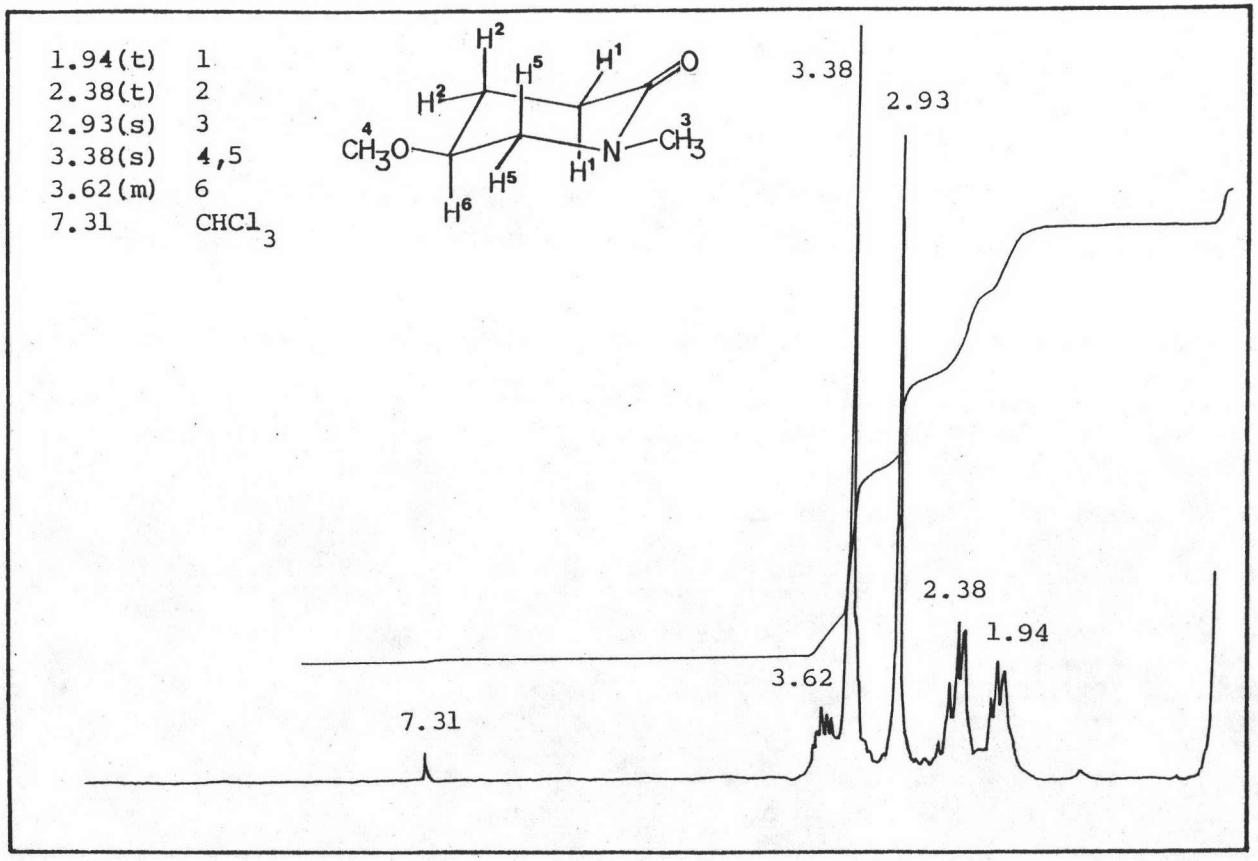

Figure 17 The ^{1}H NMR spectrum of 5-methoxy-1-methyl-2-piperidone(47a)

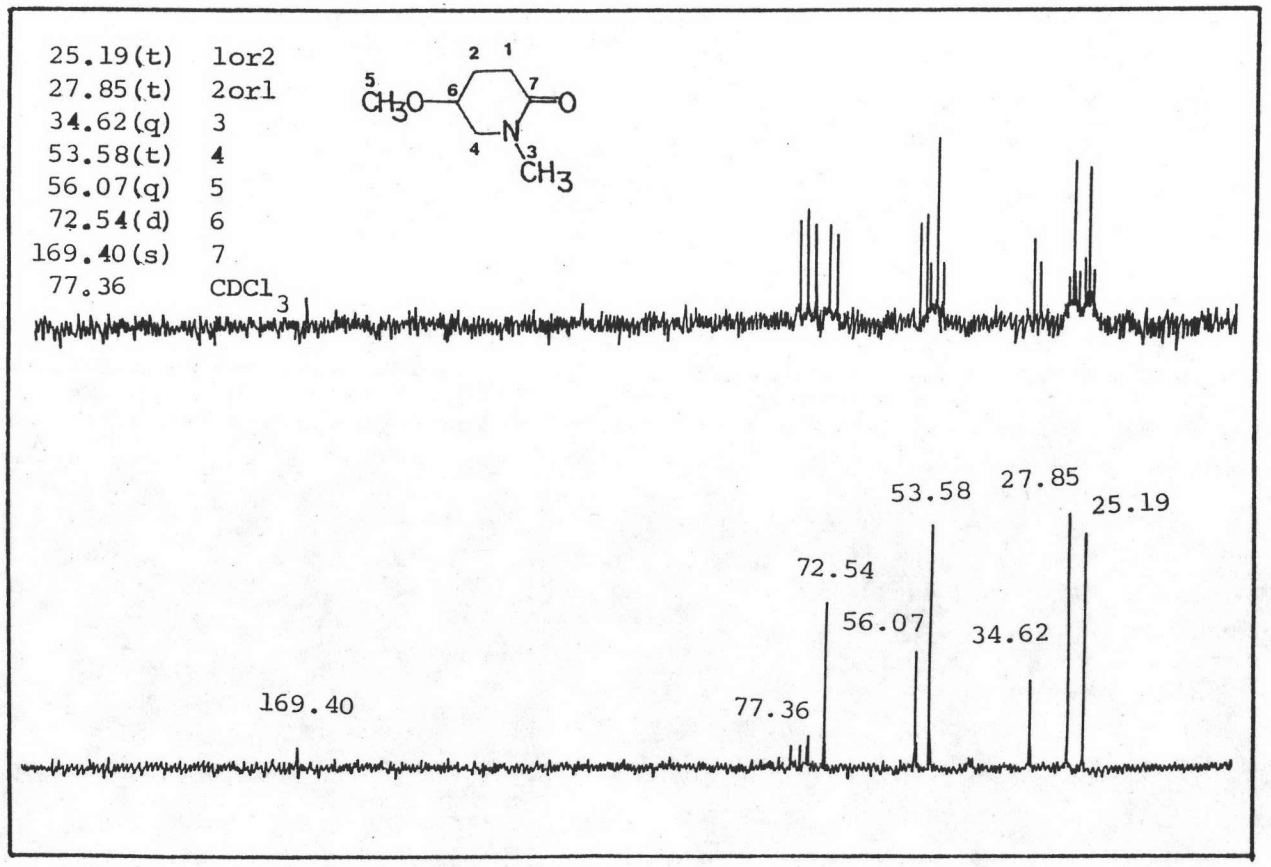

Figure 18 The ^{13}C NMR spectrum of 5-methoxy-1-methyl-2-piperidone(47a)

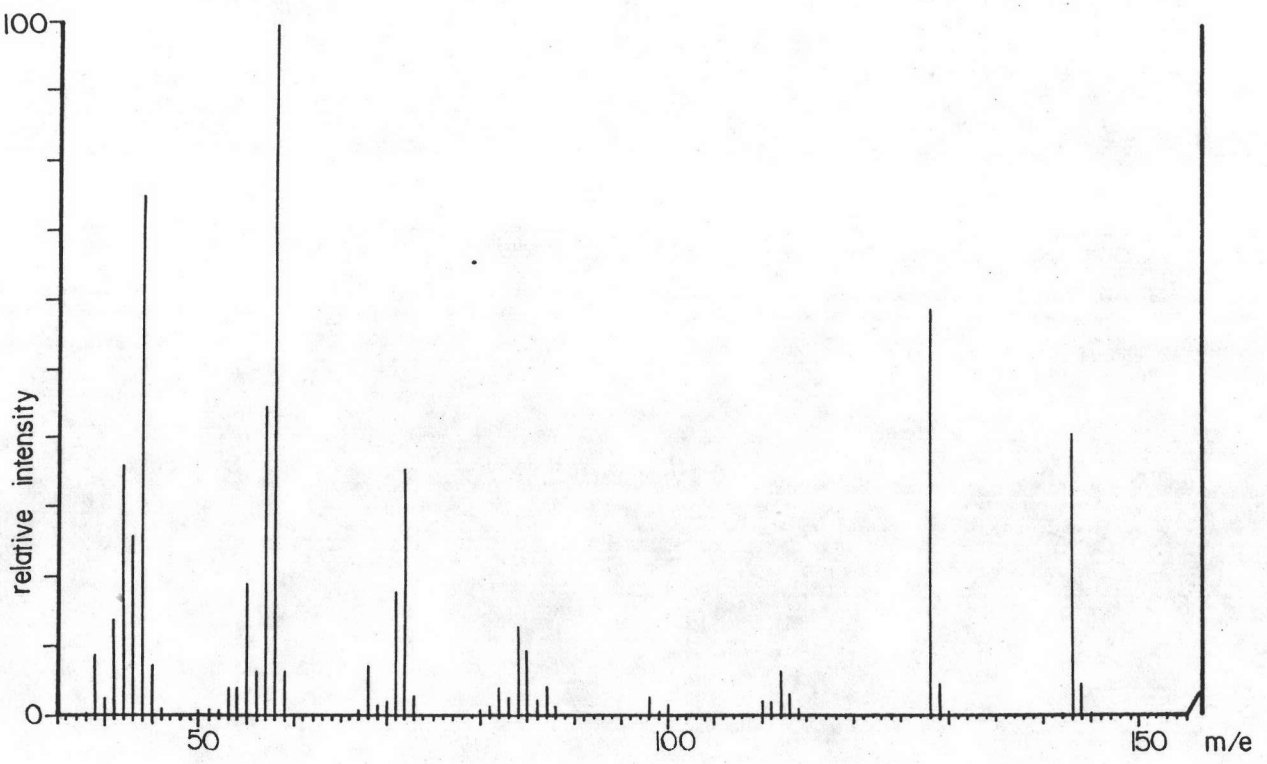

Figure 19 The mass spectrum of 5-methoxy-1-methyl-2-piperidone(47a)

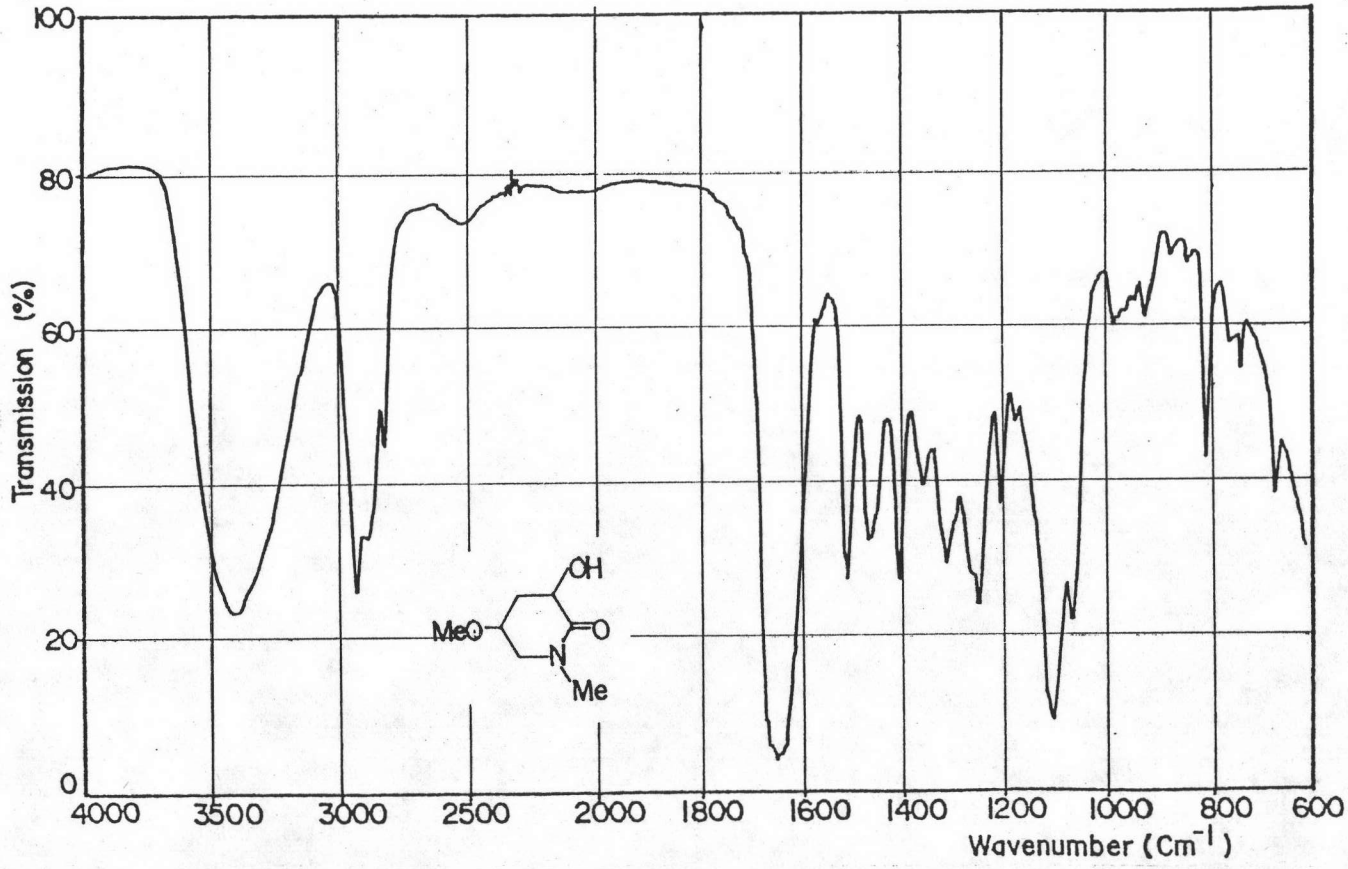

Figure 20 The IR spectrum of 3-hydroxy-5-methoxy-1-methyl-2-piperidone (48a)

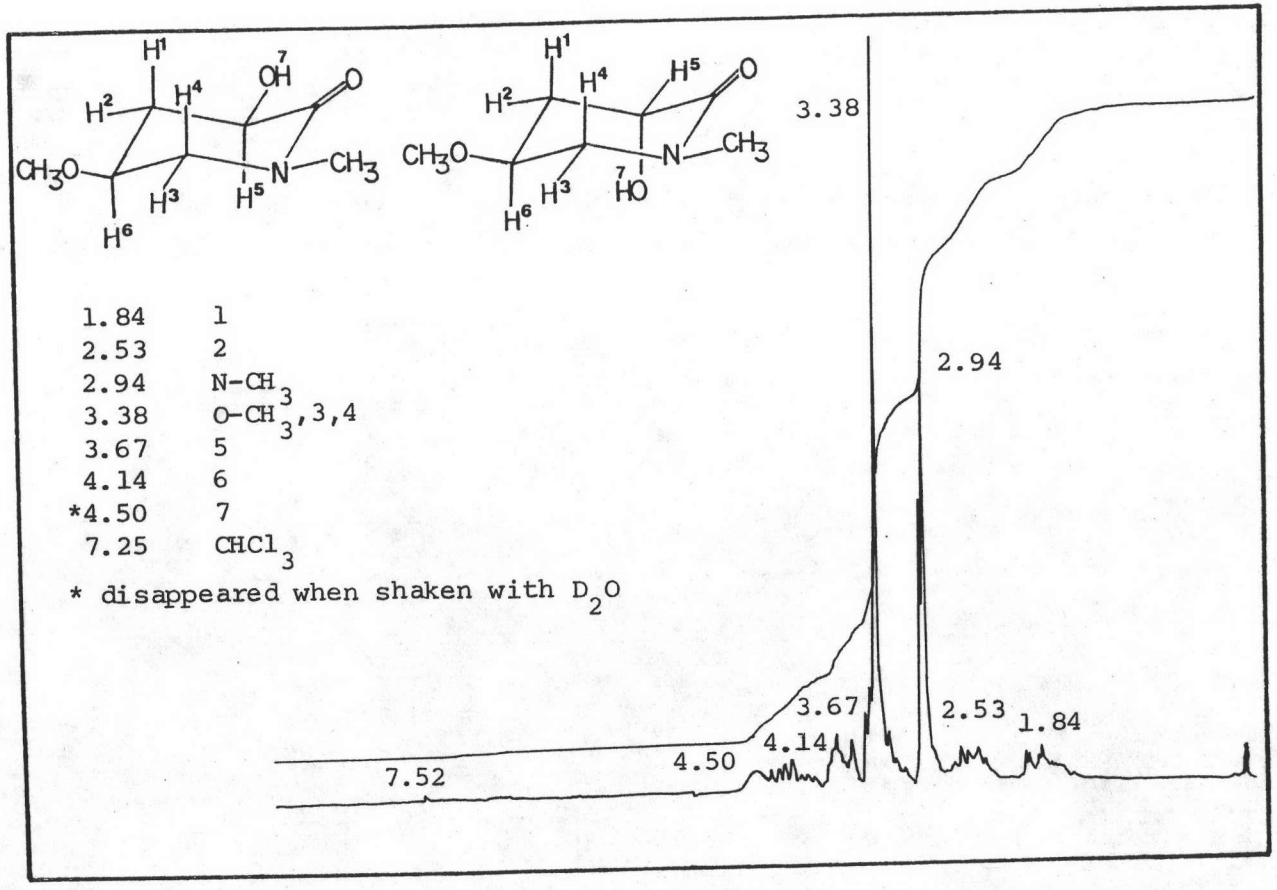

Figure 21 The ^{1}H NMR spectrum of 3-hydroxy-5-methoxy-1-methyl-2-piperidone(<u>48a</u>)

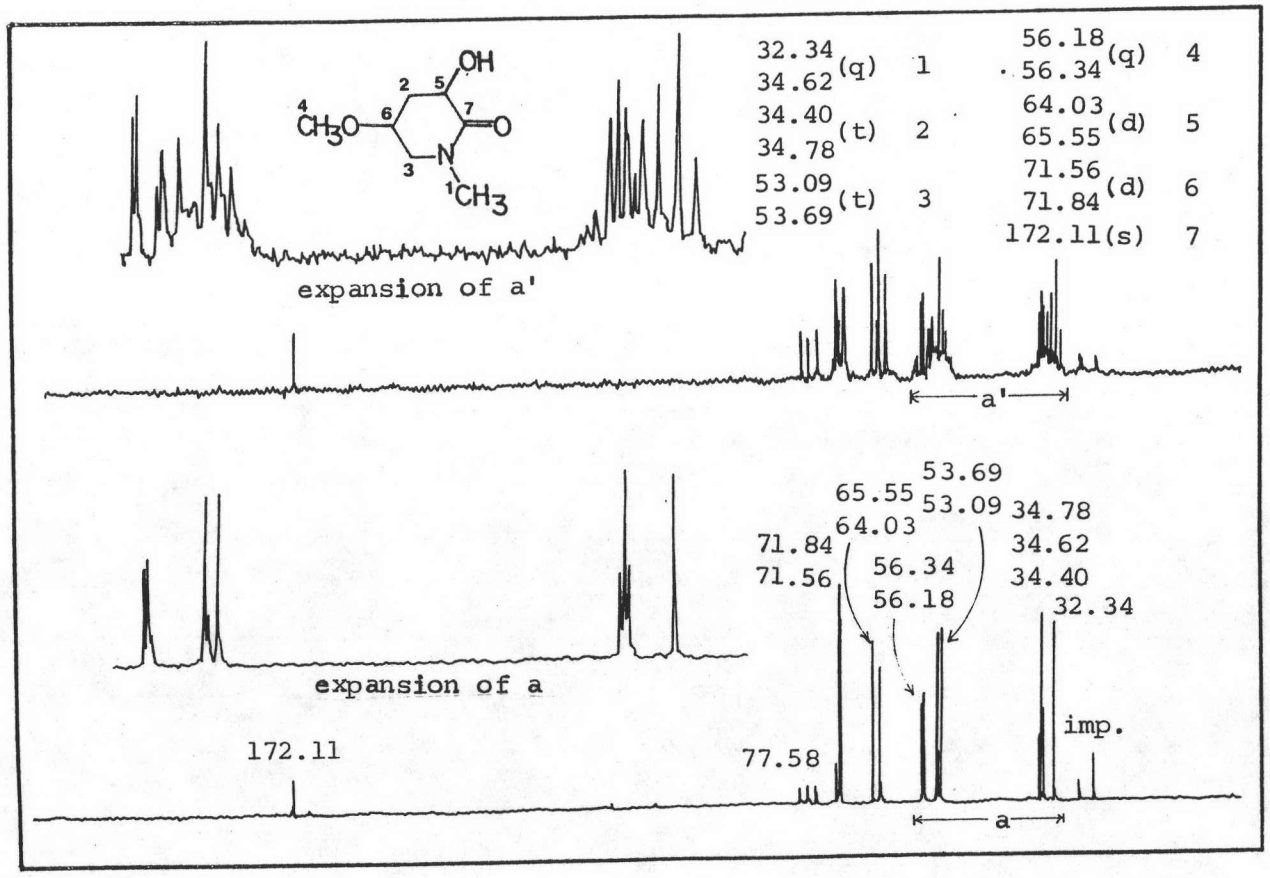

Figure 22 The ^{13}C NMR spectrum of 3-hydroxy-5-methoxy-1-methyl-2-piperidone(48a)

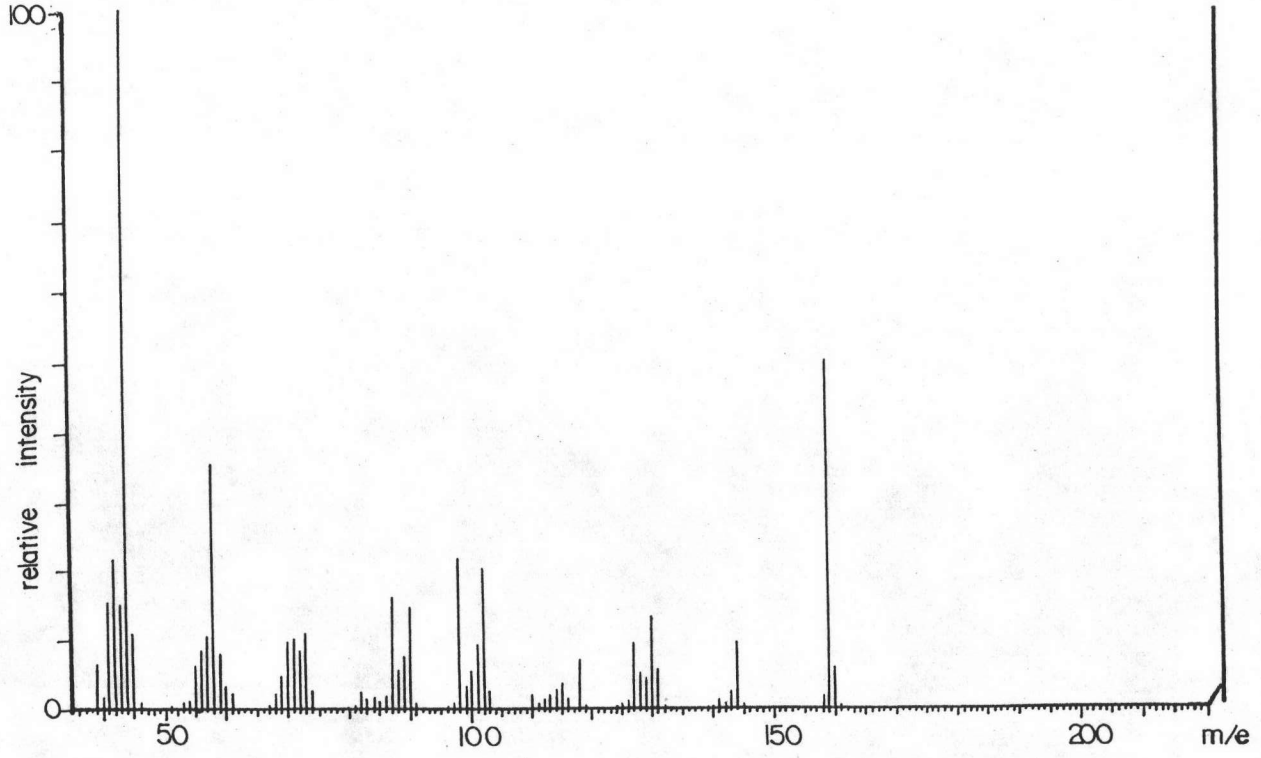

Figure 23 The mass spectrum of 3-hydroxy-5-methoxy-1-methyl-2-piperidone(48a)

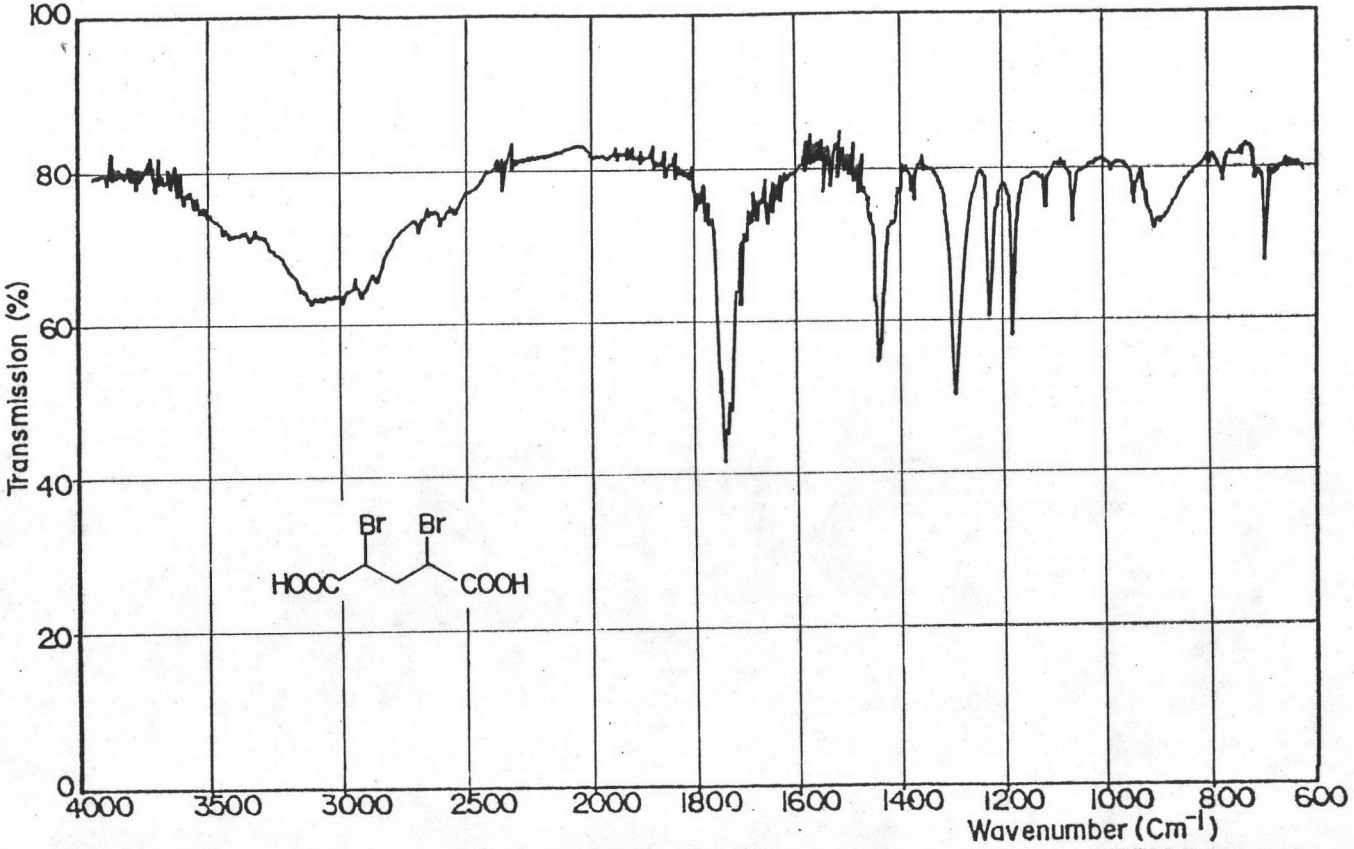

Figure 24 The IR spectrum of 2,4-dibromopentanedioic acid(50)

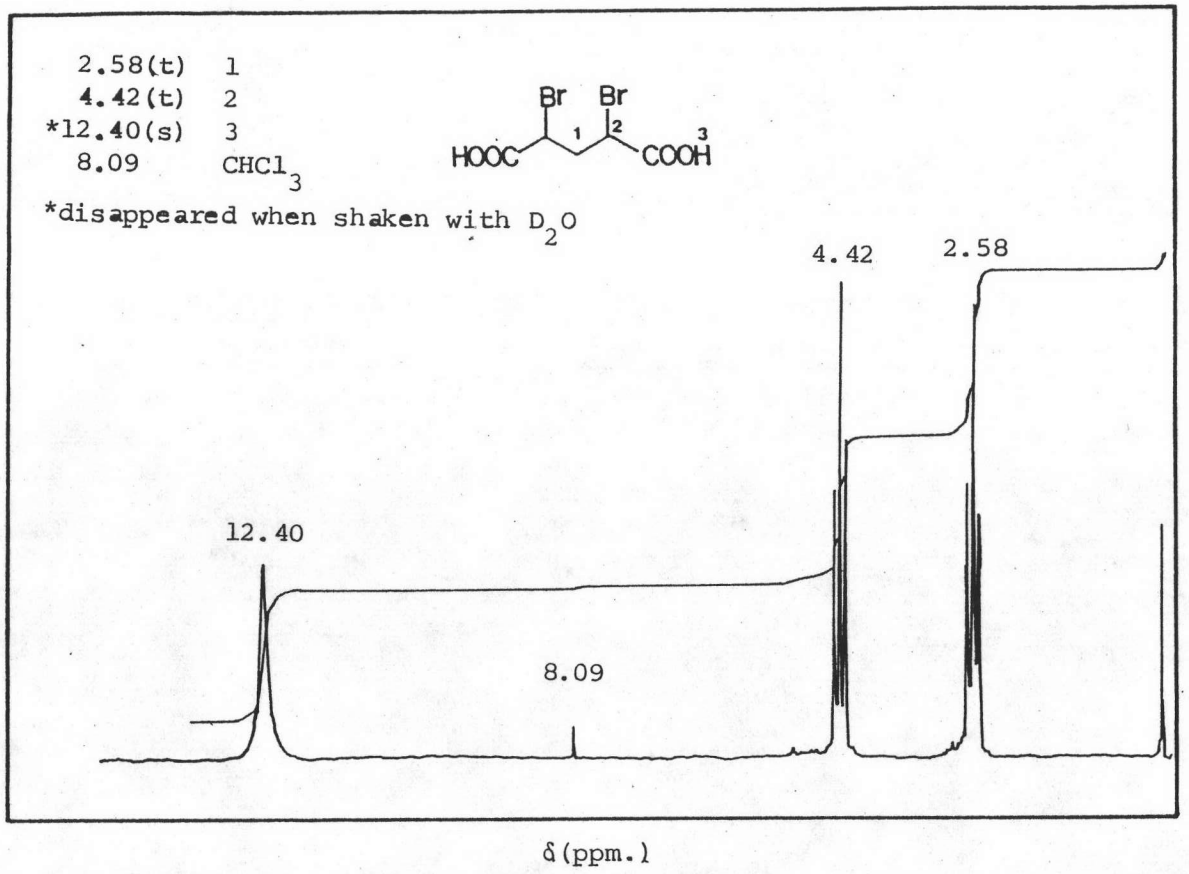

Figure 25 The ^1H NMR spectrum of 2,4-dibromopentanedioic acid(50)

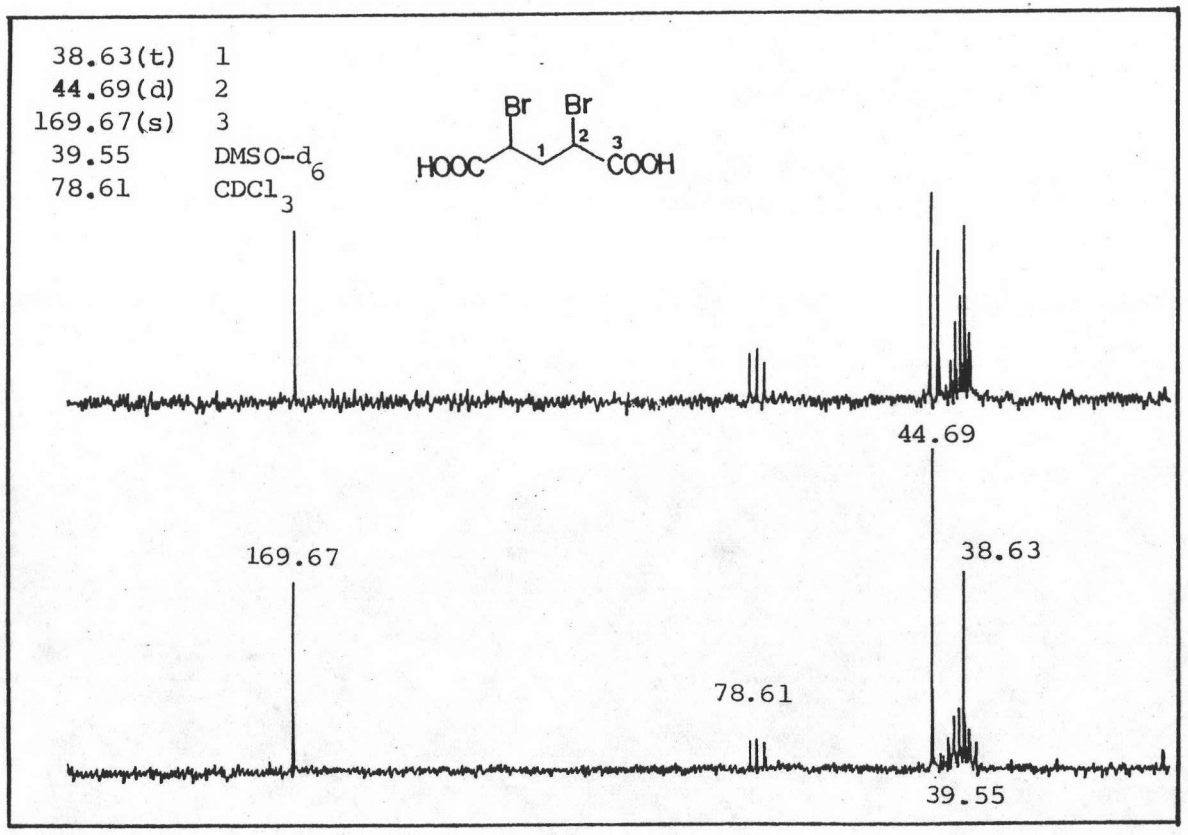

Figure 26 The ^{13}C NMR spectrum of 2,4-dibromopentanedioic acid(50)

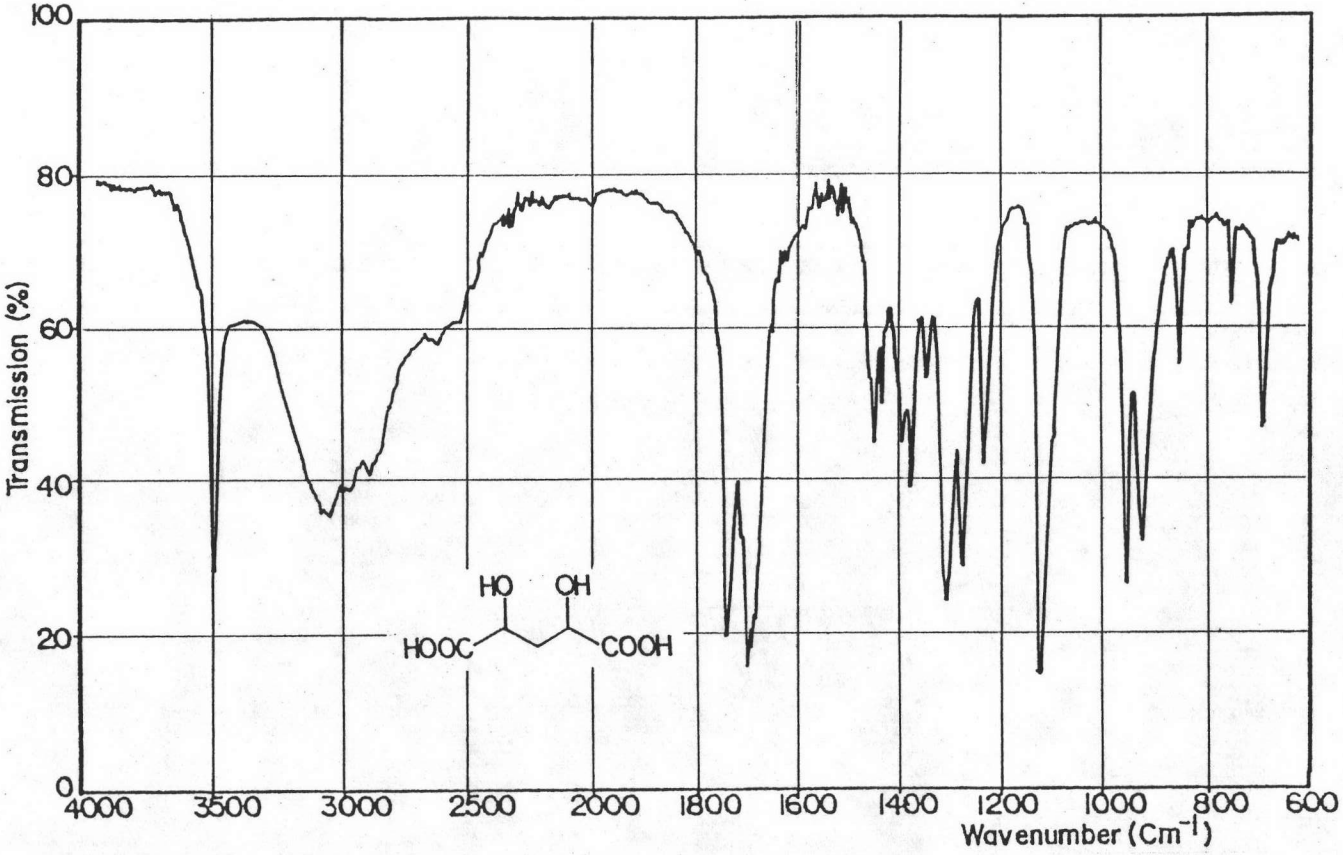

Figure 27 The IR spectrum of 2,4-dihydroxypentanedioic acid(51)

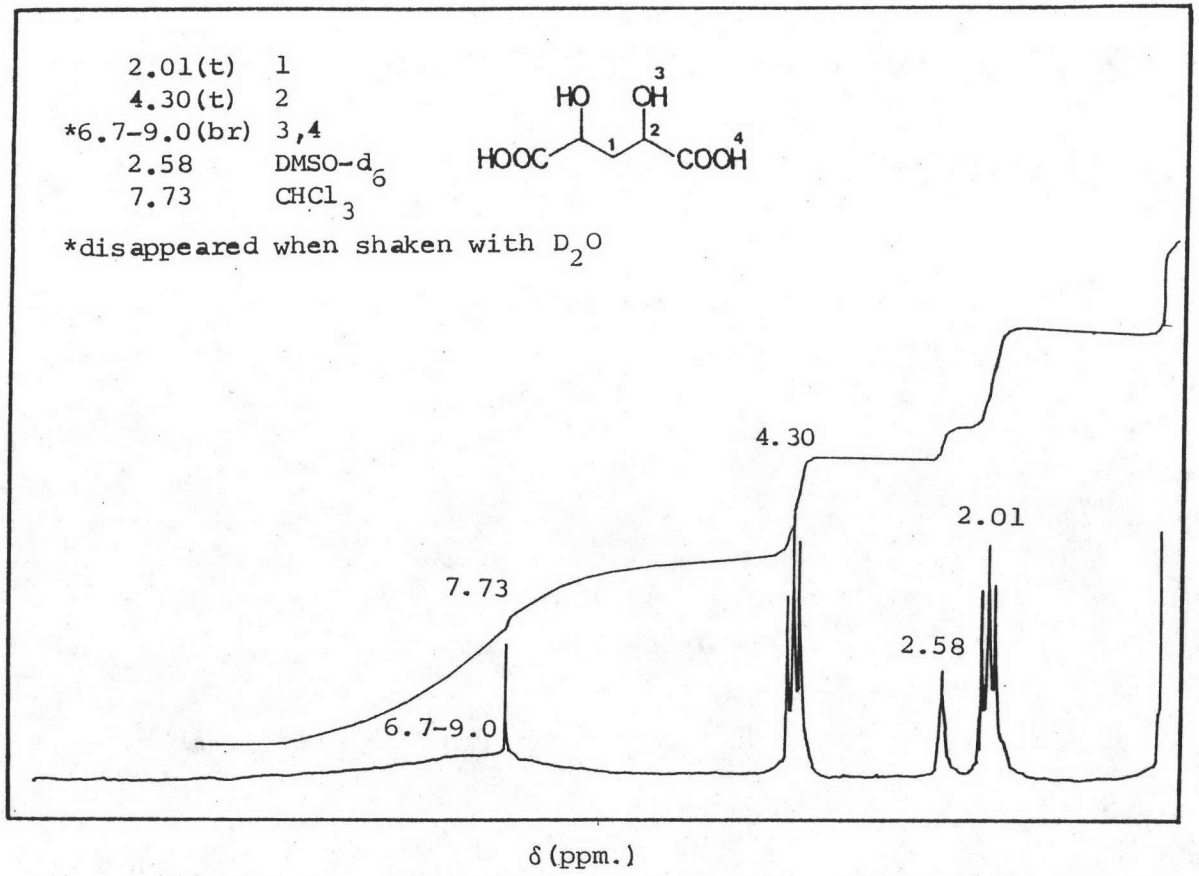

Figure 28 The ^{1}H NMR spectrum of 2,4-dihydroxypentanedioic acid(51)

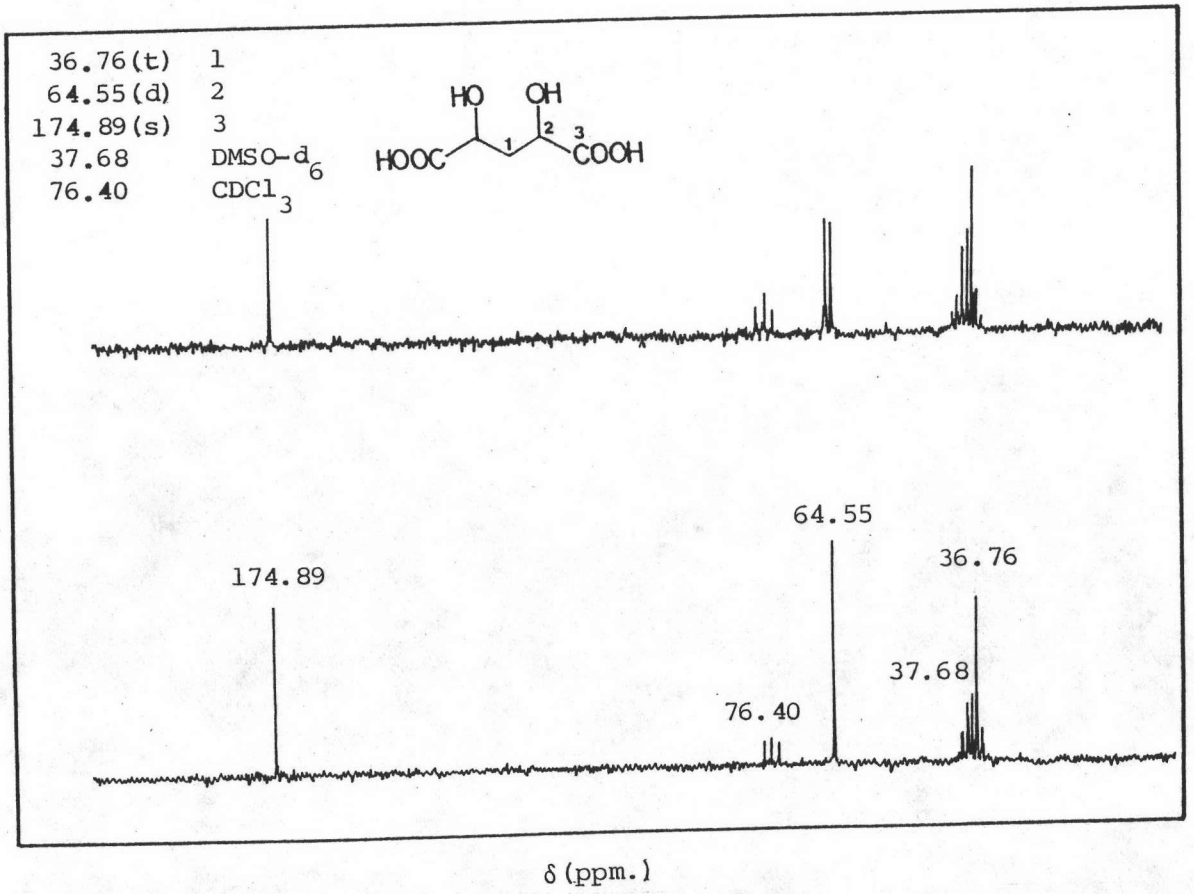

Figure 29 The ^{13}C NMR spectrum of 2,4-dihydroxypentanedioic acid(51)

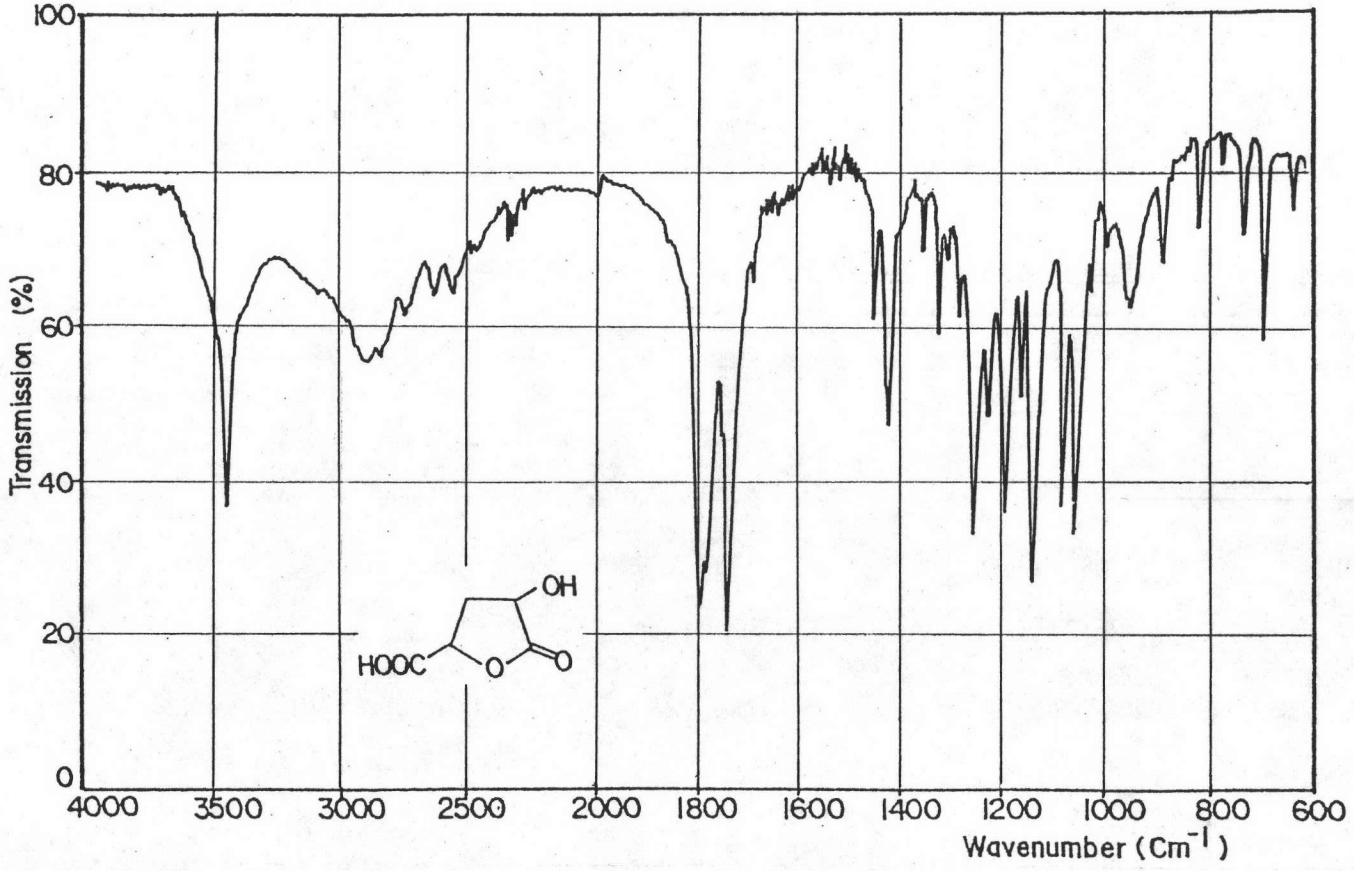

Figure 30 The IR spectrum of 5-carboxy-3-hydroxy-2-tetrahydrofuranone (52)

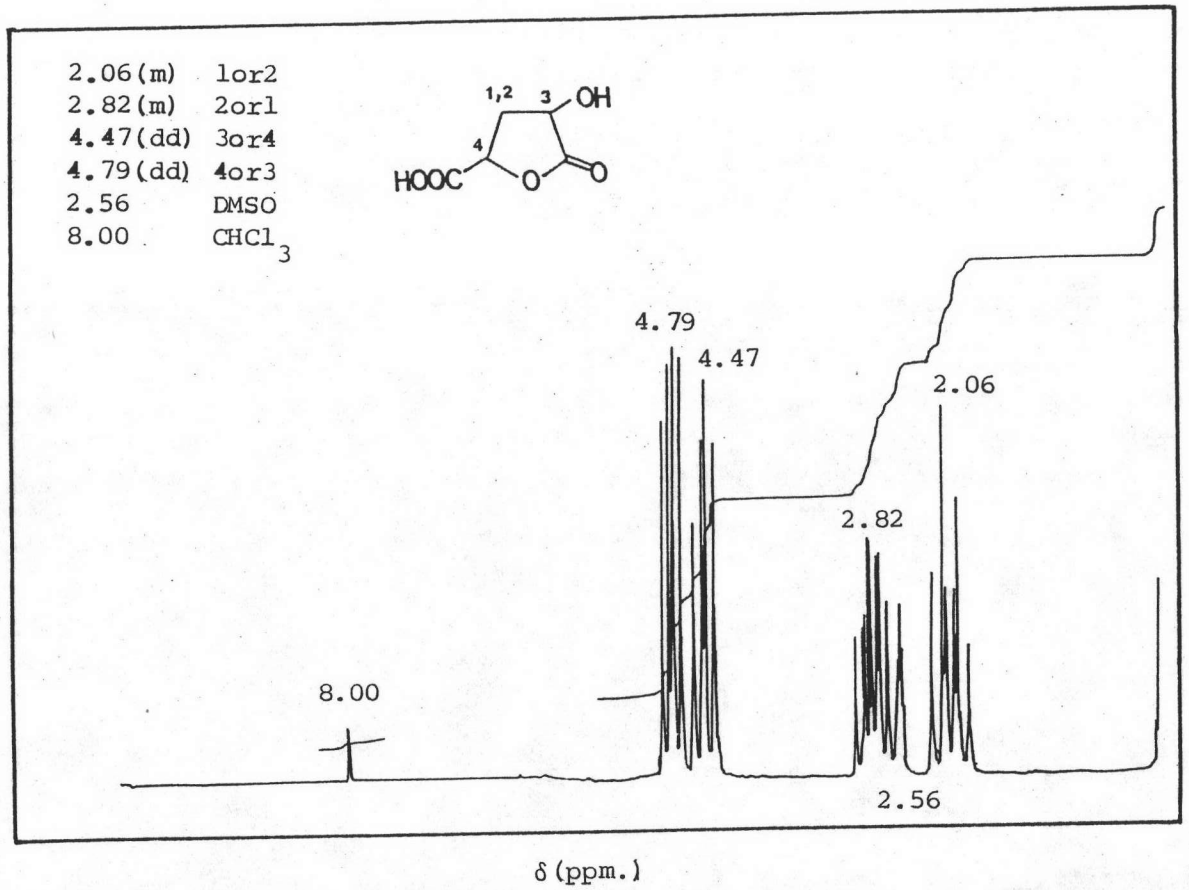

Figure 31 The ^1H NMR spectrum of 5-carboxy-3-hydroxy-2-tetrahydrofuranone(52)

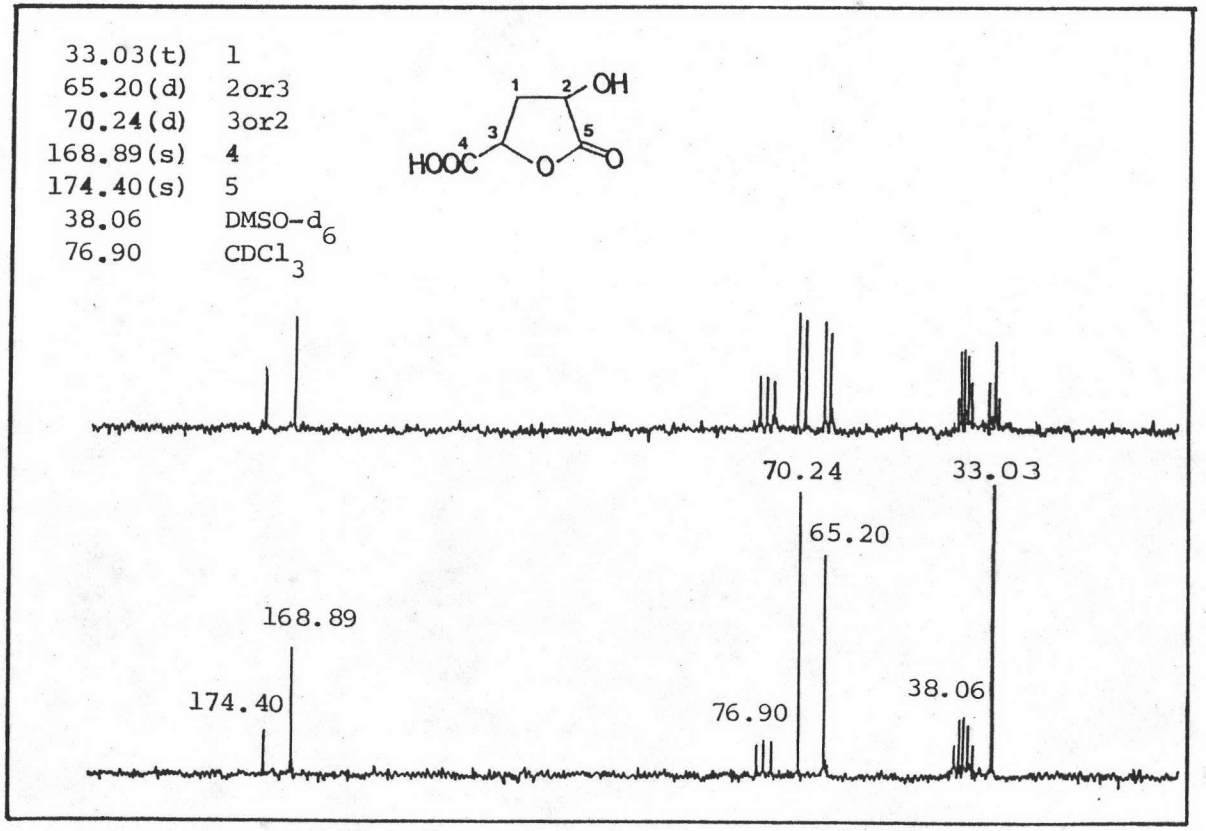

Figure 32 The ^{13}C NMR spectrum of 5-carboxy-3-hydroxy-2-tetrahydrofuranone(52)

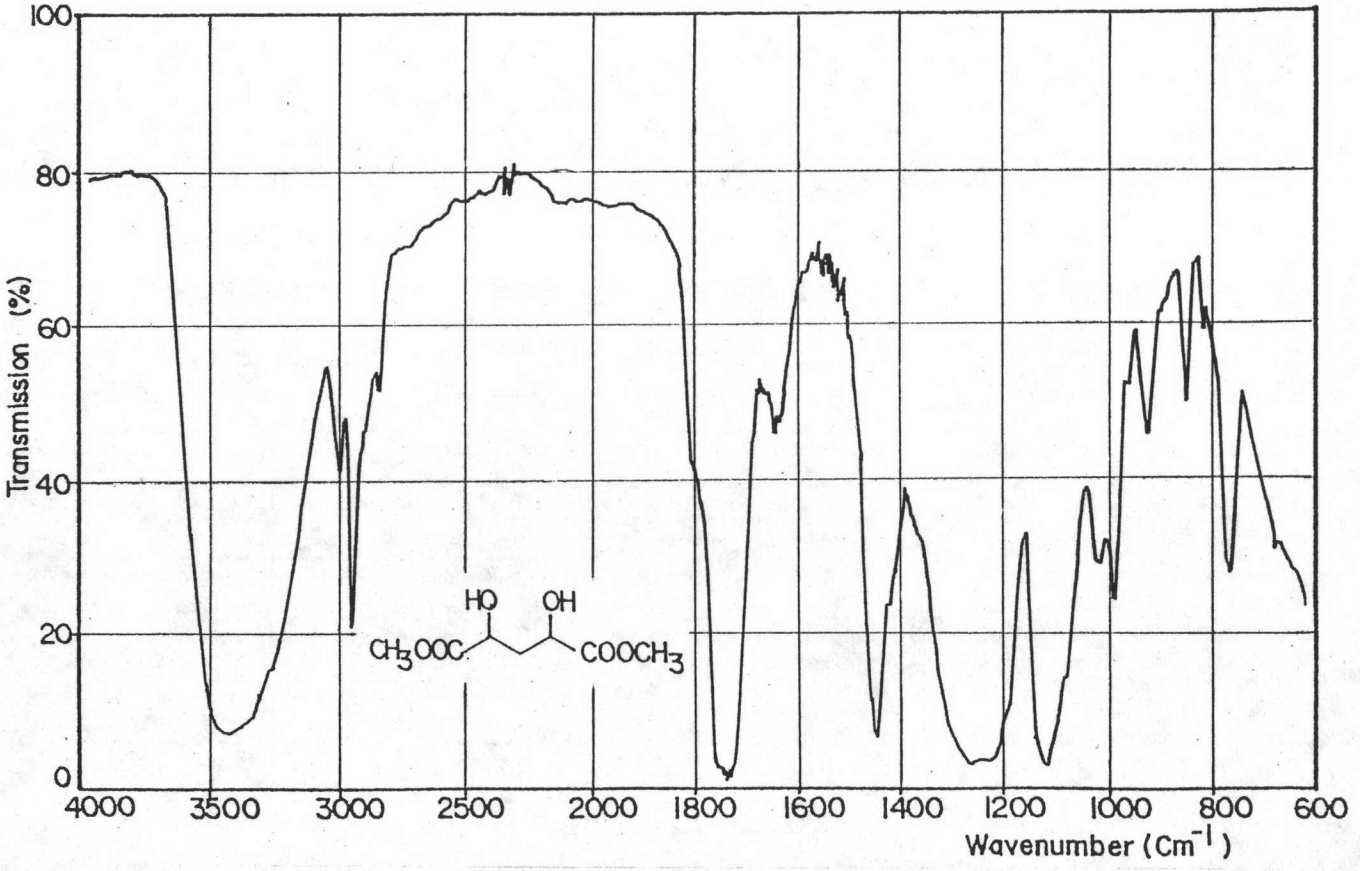

Figure 33 The IR spectrum of dimethyl 2,4-dihydroxypentanedioate(53a)

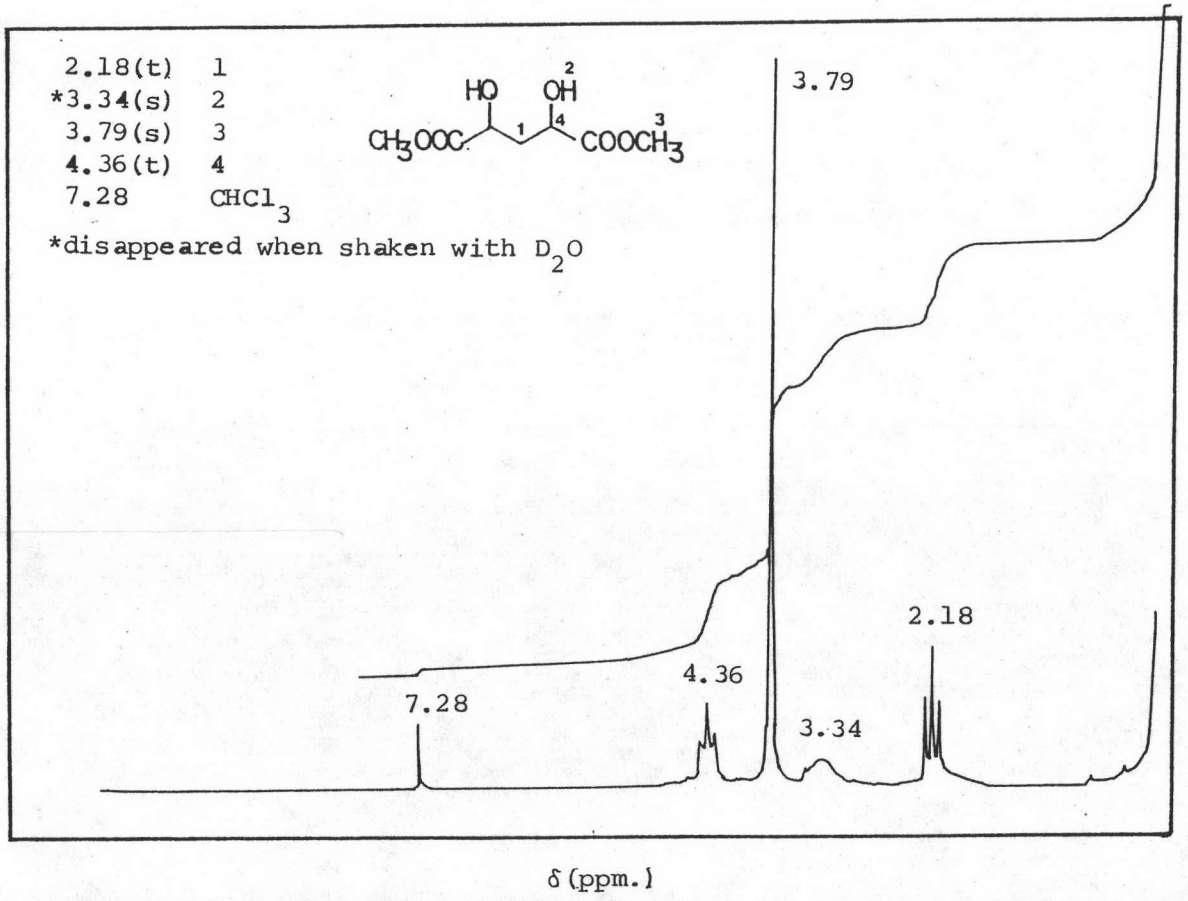

Figure 34 The ^1H NMR spectrum of dimethyl 2,4-dihydroxypentanedioate (53a)

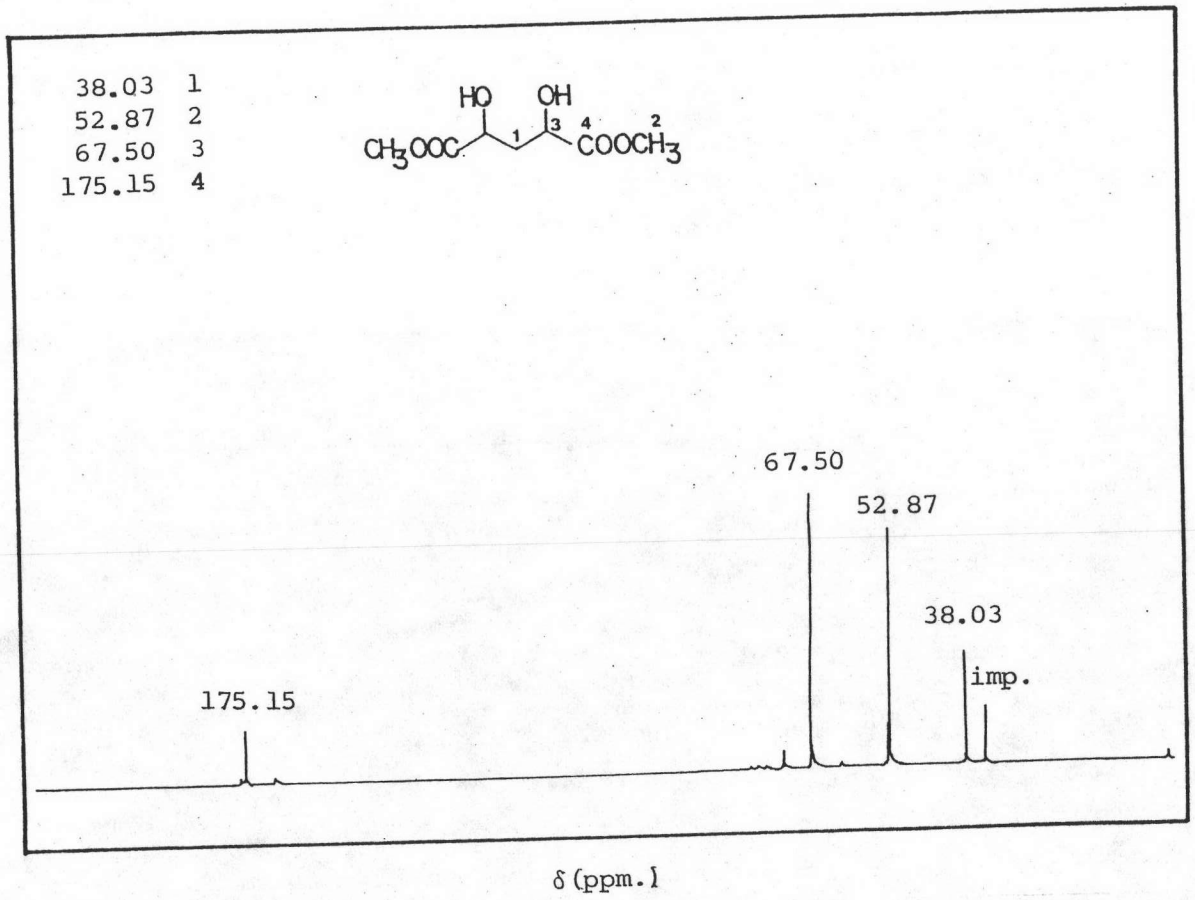

Figure 35 The ^{13}C NMR spectrum of dimethyl 2,4-dihydroxypentanedioate (53a)

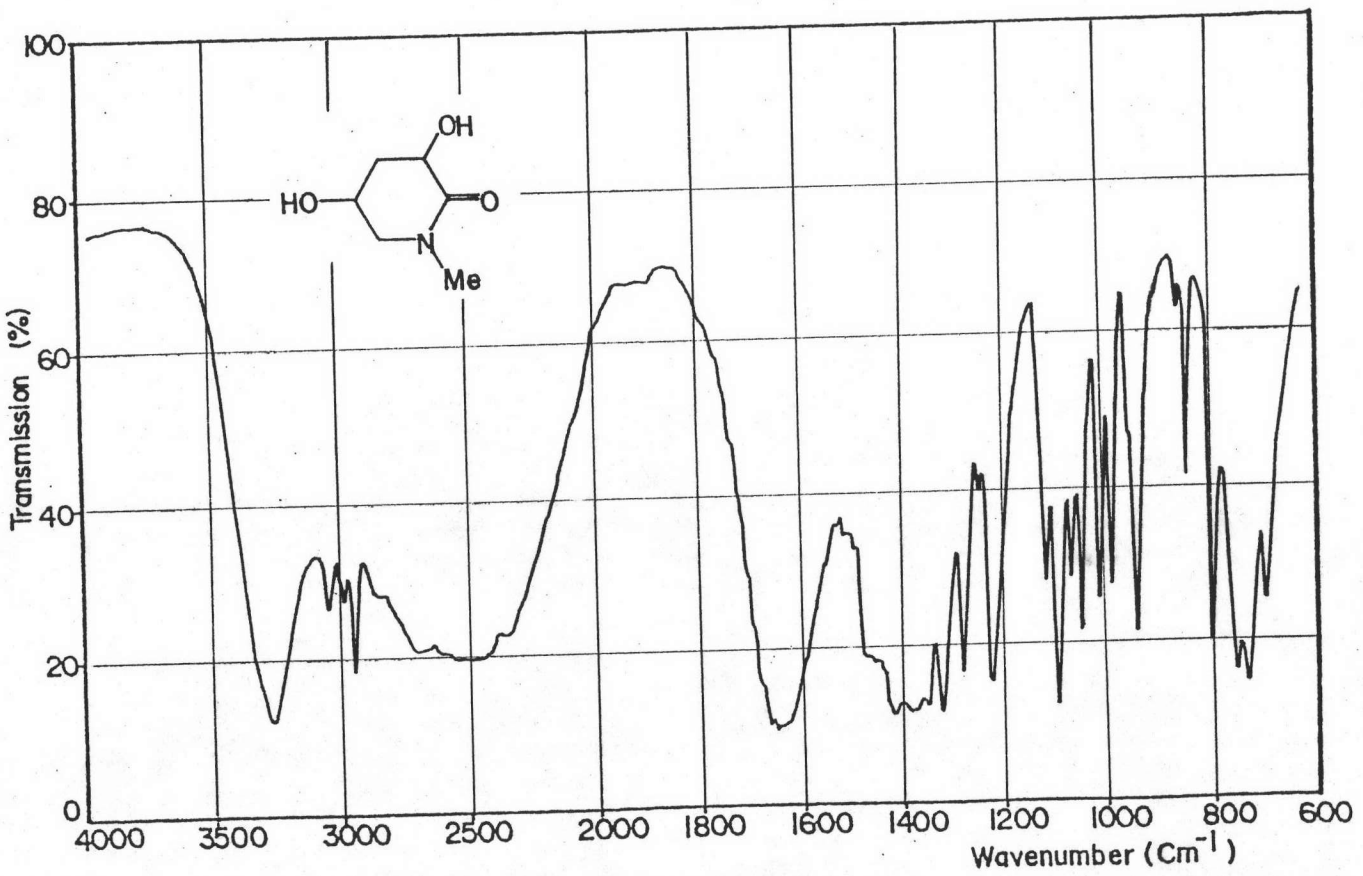

Figure 36 The IR spectrum of odoram

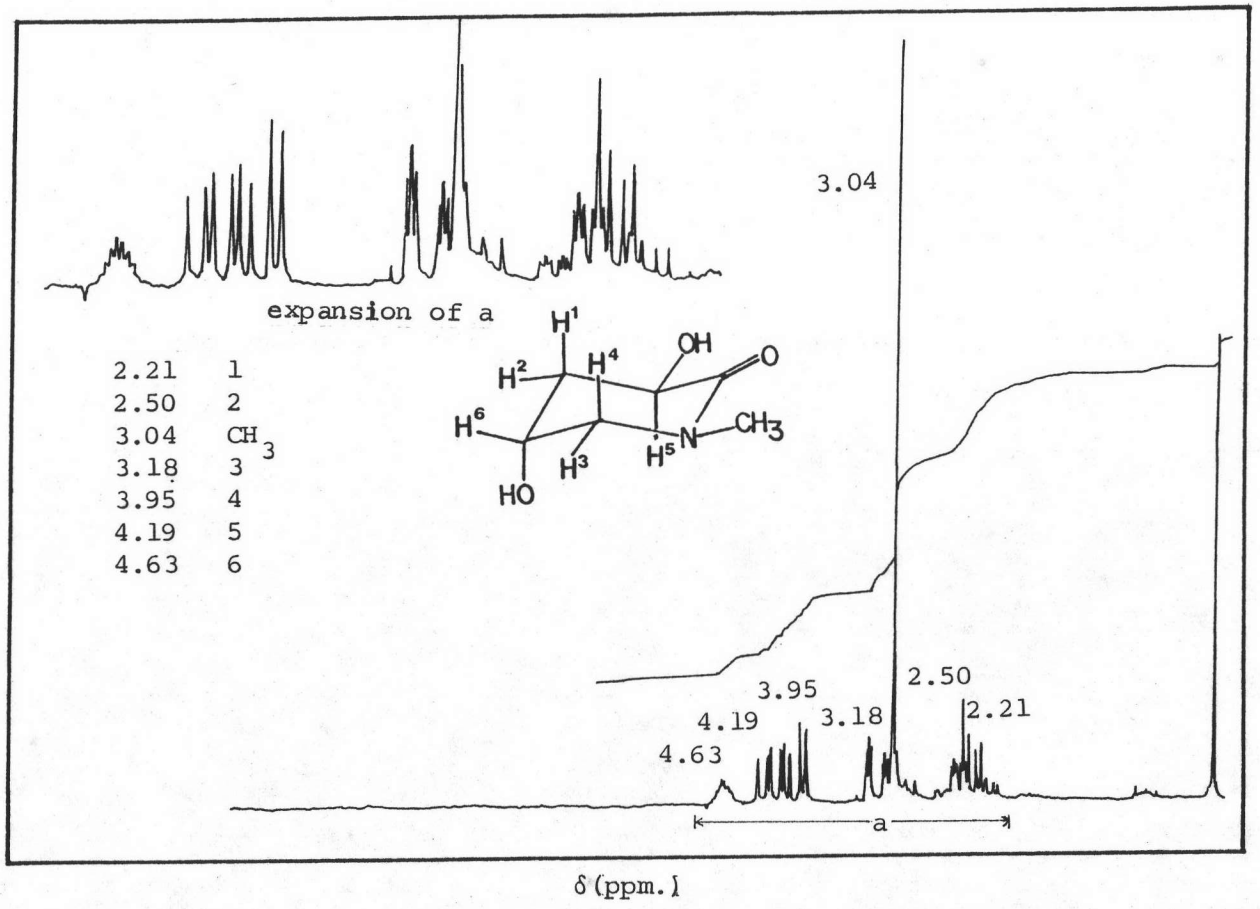

Figure 37 The ^{1}H NMR spectrum of odoram

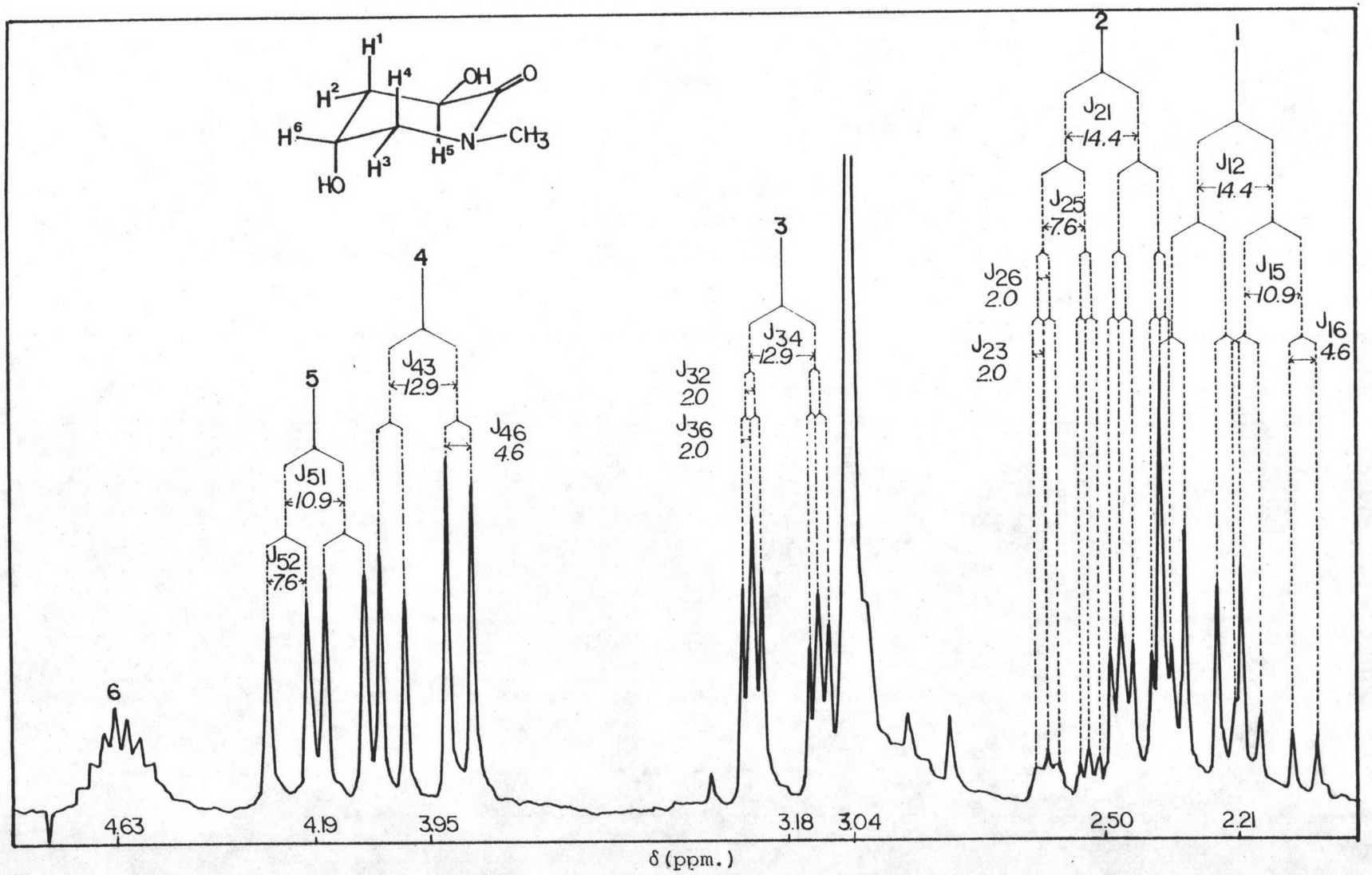

Figure 38 The ^{1}H NMR spectrum of odoram shows the protons coupling and the coupling constants

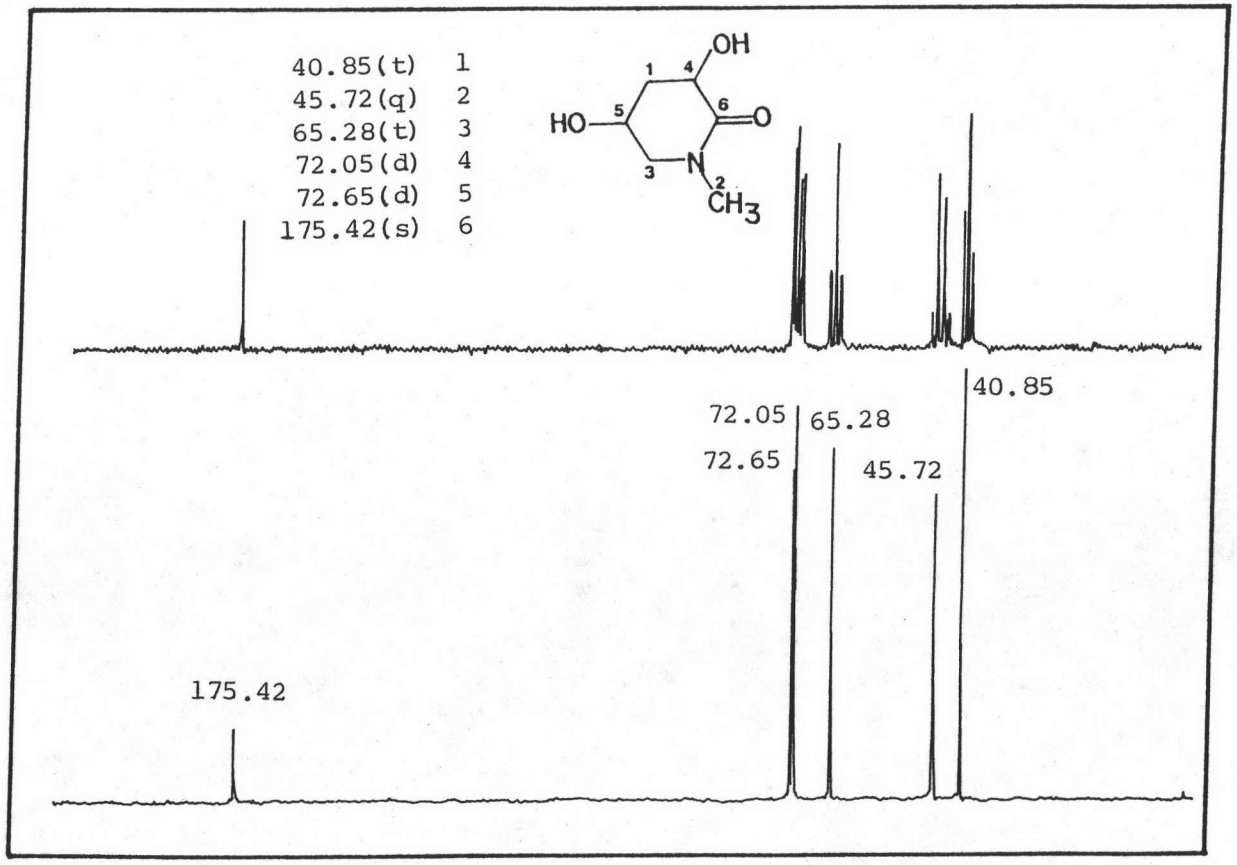

Figure 39 The ^{13}C NMR spectrum of odoram

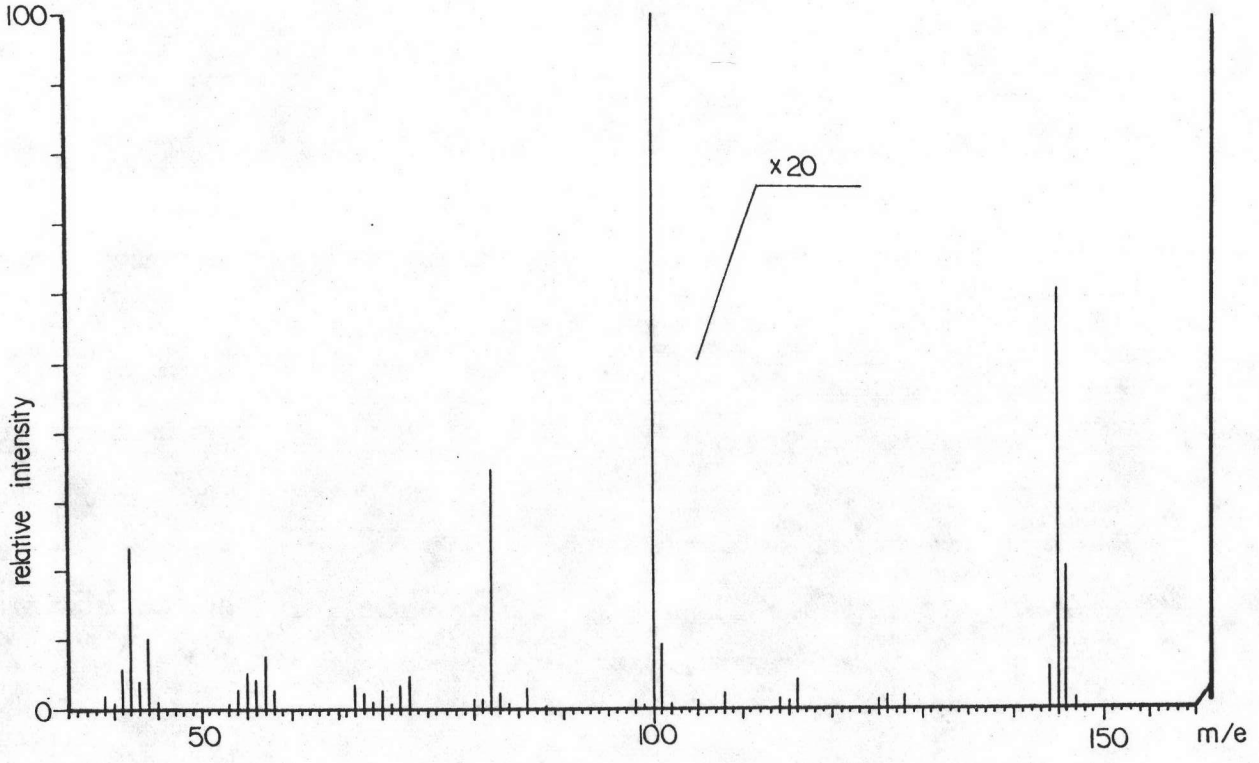

Figure 40 The mass spectrum of odoram

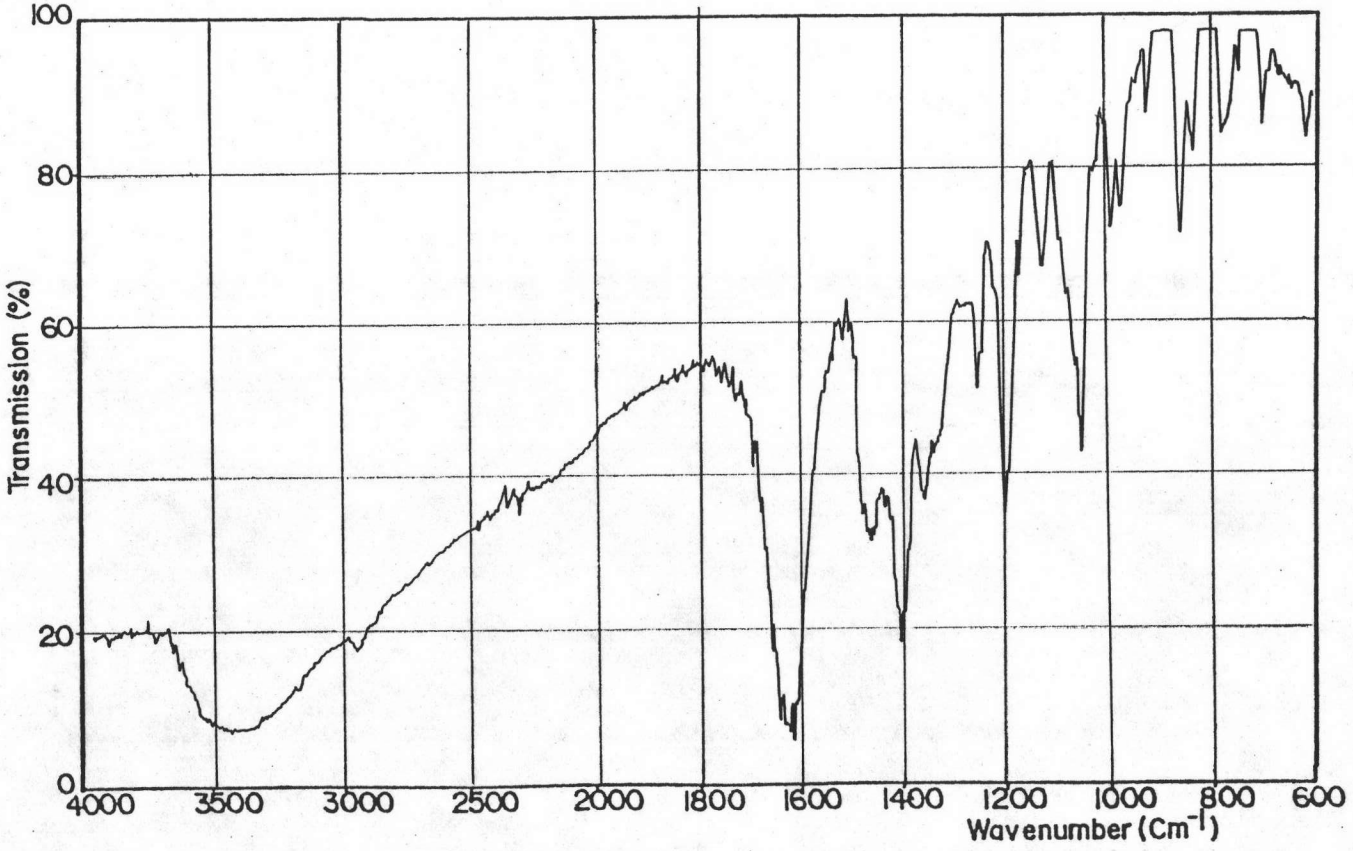

Figure 41 The IR spectrum of methylated odoram

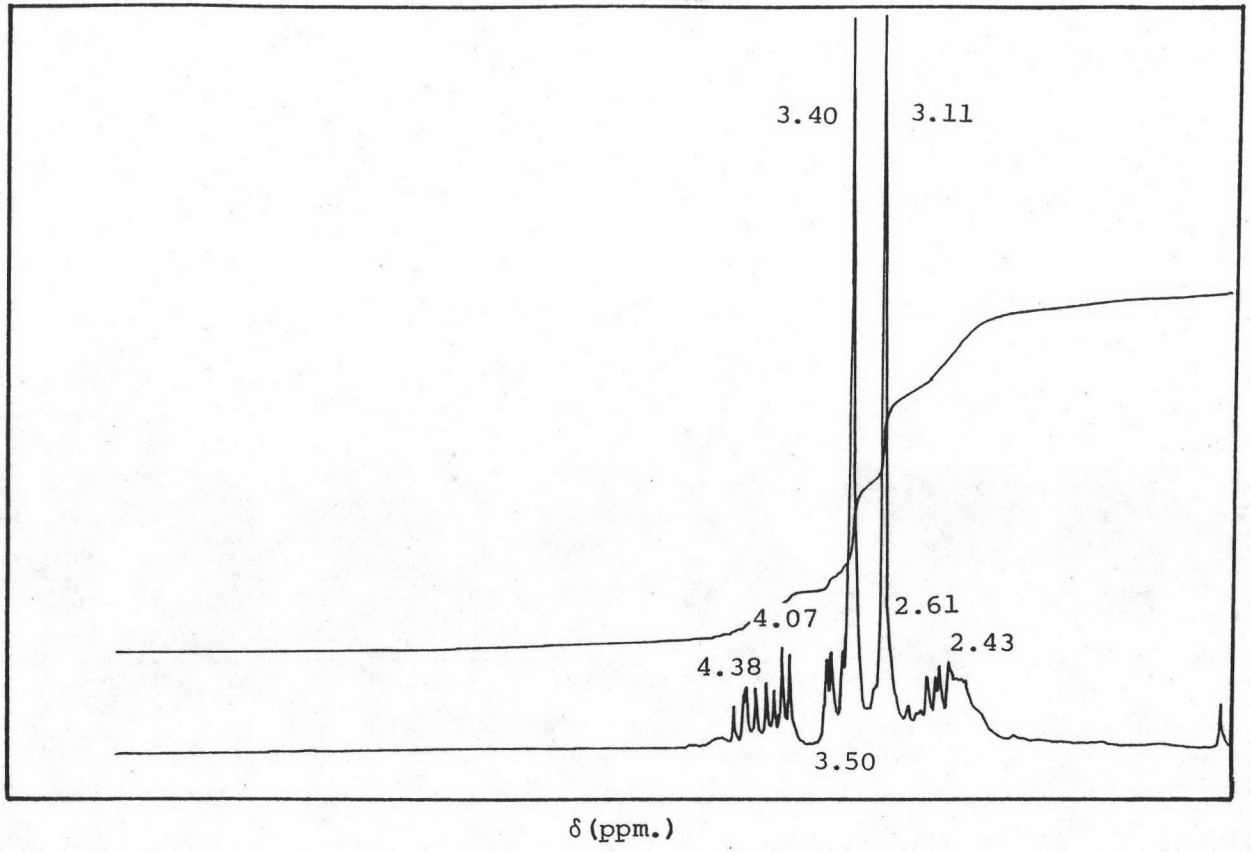

Figure 42 The ^{1}H NMR spectrum of methylated odoram

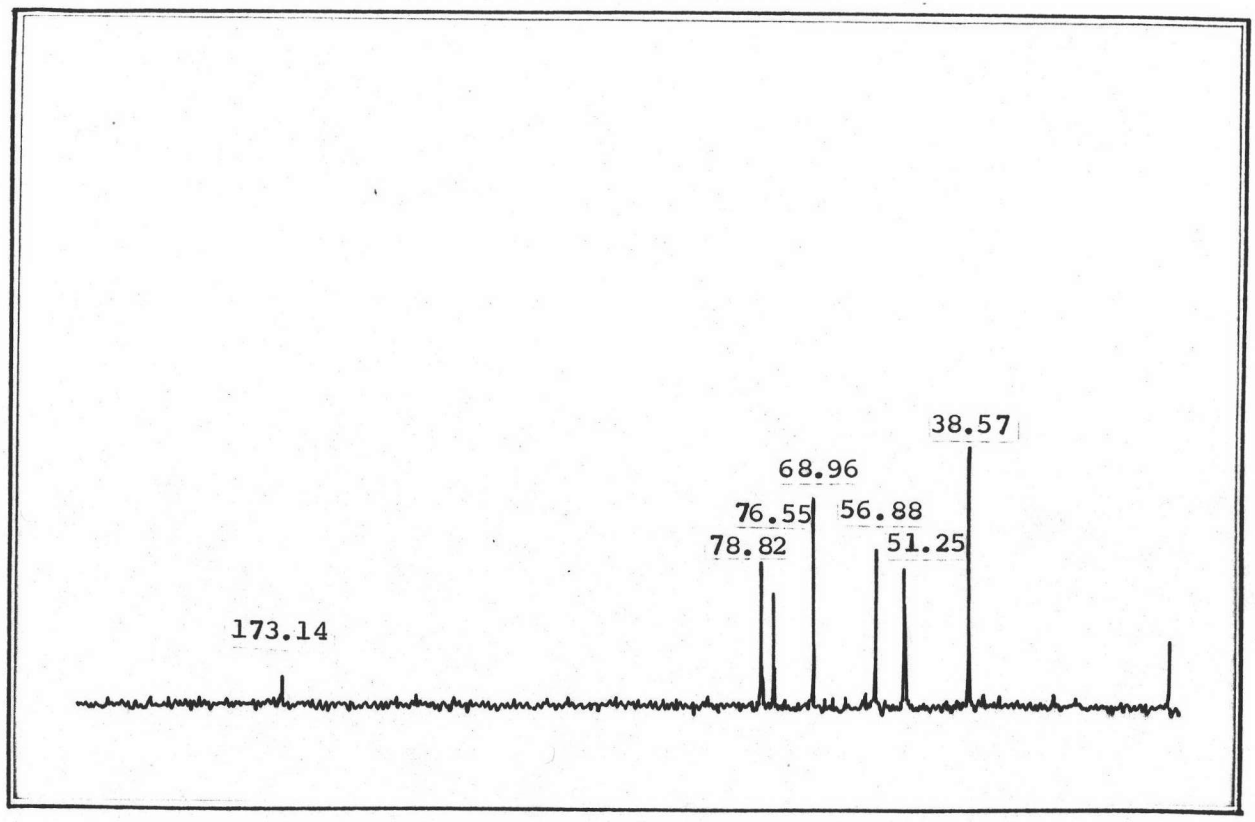

Figure 43 The ^{13}C NMR spectrum of methylated odoram