CHAPTER II

EXPERIMENT AND RESULTS

2.1 Instruments and Equipments

2.1.1 Infrared Spectra (IR)

The IR spectra were recorded on a Perkin-Elmer Model IR 718 Infrared Spectrophotometer. Solid samples were examined by incorporating the sample into a pellet of potassium bromide, while the liquid samples were examined neat on sodium chloride cell.

2.1.2 Mass Spectra (MS)

The mass spectra were obtained by a Jeol Mass Spectrometer Model JMS-DX-300/JMA 2000 at 70 eV.

2.1.3 Proton and Carbon-13 Nuclear Magnetic Resonance (^{1}H NMR and ^{13}C NMR)

The ^{1}H and ^{13}C NMR spectra were performed using a Jeol Fourier Transform NMR Spectrometer Model FX 90 Q. Tetramethylsilane (TMS) was used as an internal standard. The chemical shifts (δ) were given in ppm. down field from the TMS.

2.1.4 Elemental Analyses

The elemental analyses were made by using a Perkin Elmer CHNO Analyzer Model 240C.

2.2 Physical Seperation Techniques

2.2.1 Column chromatography (CC)[30]

Merck's silica gel 700-325 mesh ASTM and aluminium oxide active, neutral for column chromatography, were used as adsorbents.

Packing the column

Pre-adjusting the stopcock so that it was almost closed, but still allowed the solvent to drip through. Allowing approximately 0.5 cm. of solvent to remain in the column when the slurry of the silica gel was poured. After the bed had settled, the sample, dissolved in the solvent, was introduced when the solvent level was just above the bed in the column. Some adsorbent was usually added to the top of the column prior to the addition of the eluent.

Flash column chromatography

This method was similar to column chromatography, the adsorbent particles were smaller and an air pump was applied on the top of the column.

2.2.2 Thin-layer chromatography (TLC)[31]

The 0.25 mm. thin-layer chromatoplate was prepared in the following manner.

A mixture of silica gel (25 g.) and water (50 mL.) was stirred until it became slurry; it was then applied to glass plates (5x20 cm.) using a desaga spreader. After being dried at room temperature for an hour, the plates were heated at 125°C for

30 minutes, then cooled and stored in desiccator until used.

Two lines were drawn on each plate. One was 2.0 cm. from one edge; this line would be referred to as the base line. The other line was 14.0 cm. above and parallel to the first line called the upper line. The solution containing the compounds to be investigated was applied as small spots on the base line of the plate at 1.0 cm. interval. And after the solvent had evaporated, the plate was placed in a glass tank filled to a depth of 1.0 cm. with the eluting solvent. The eluting solvent moved up the plate immediately. As the solvent front had reached the upper line, the plate was removed from the tank; the solvent was allowed to evaporate, and the plate was detected with a suitable detector such as UV, I_2 and 25% sulfuric acid to reveal the compounds.

2.2.3 Preparative thin-layer chromatography (PTLC)[31]

PTLC plates were prepared in the same ways as described for TLC plates, but PTLC plates were coated thicker than the TLC ones.

The sample solution was applied as a narrow band about 2.0 cm. from the edge of the plate. The plate after being dried by the air current, was developed by the ascending method. The components were detected by viewing under the UV lamp and appeared as purple bands against the white background of the adsorbent. These bands were cut out, dispersed in appropiate solvent contained in a flask. After stirring the mixture for a few minutes, it was filtered by suction. The filtrate was concentrated and the residue was further purified or identified by an appropiate method.

2.3 Syntheses

2.3.1 Preparation of 5-ethoxycarbonyl-2-tetrahydrofuranone(41)[21]

To a suspension of L-glutamic acid (60.0 g., 0.408 mole) in water (160 mL.) and conc. hydrochloric acid (126 mL.) was added a solution of sodium nitrite (42 g. 0.608 mole) in water (90 mL.) during 3 hours under vigorous stirring at -5 to 0°C, and then the resulting clear solution was allowed to stand at room temperature overnight. The solvent was evaporated *in vacuo* to dryness below 50°C to give residue, which was shaken with ethyl acetate (300 mL.). The insoluble material was filtered off and washed with ethyl acetate. The filtrate and washings were combined, and dried over anhydrous sodium sulfate. Evaporation of the solvent *in vacuo* afforded 5-carboxy-2-tetrahydrofuranone(40a) as a pale yellow syrup (52.8 g.). A solution of this syrup (52.8 g.) and *p*-toluenesulfonic acid (1.3 g.) in ethanol (80 mL.) and benzene (200 mL.) was refluxed for 5 hours and then was distilled off under atmospheric pressure until the boiling point raised to 79°C. Chloroform (600 mL.) was added to the residue, and the whole was washed sucessively with water, 10% aqueous sodium carbonate solution, and water and dried over anhydrous sodium sulfate. The solvent was then removed and the residue was purified by flash column chromatography on silica gel with 1% methanol in dichloromethane as eluting solvent to give two compounds. The first eluent was pale yellow oil, identified as diethyl 2-hydroxypentanedioate. The second one was also pale

yellow oil, after distillation of this oil gave 5-ethoxycarbonyl-2-tetrahydrofuranone (41) as colorless liquid (49.5 g., 76.8 % yield) of b.p. 114-118°C/ 1 mm.(lit.[21], 135-140°C/10 mm.); R_f = 0.61 (ethanol:chloroform = 1:9 v/v).

IR ν_{max}^{neat} (cm^{-1}): 2980, 2930, 1790, 1740, 1180 (Fig. 1) as reported in lit.[21].

^1H NMR ($CDCl_3$) δ(ppm.): 1.32(t, 3H), 2.58(m, 4H), 4.27(q, 2H), 4.90(m, 1H) (Fig. 2) as reported in lit.[21].

^{13}C NMR ($CDCl_3$) δ(ppm.): 14.06(q), 25.80(t), 26.82(t), 61.85(t), 75.86(d), 170.87(s), 176.45(s) (Fig. 3)

2.3.2 Preparation of 5-hydroxymethyl-2-tetrahydrofuranone(42)[21]

To a stirred suspension of sodium borohydride (8.4 g., 0.222 mole) in ethanol (120 mL.) was added a solution of (41) (49.0 g., 0.310 mole) in ethanol (200 mL.) at 20 to 25°C, and the whole was stirred at room temperature for 1 hour. The mixture was adjusted to pH 3 with 30% aqueous hydrochloric acid solution. The resulting precipitate was filtered off, and the filtrate was evaporated in vacuo. Methanol was added to the residue, filtered and then evaporated the filtrate in vacuo. This process of methanol addition and evaporation was repeated 4 times, and then the residue was purified by flash column chromatography on silica gel with ethyl acetate as eluting solvent to give a pale yellow oil of 5-hydroxymethyl-2-tetrahydrofuranone(42) (19.1 g., 53.2 % yield); R_f = 0.47 (ethanol:chloroform = 1:9 v/v).

IR ν_{max}^{neat} (cm^{-1}): 3700-3100, 2950, 1760, 1200, 1065 (Fig. 4) as reported in lit.[21].

^1H NMR ($CDCl_3$) δ(ppm.): 2.18(m, 2H), 2.51(m, 2H), 3.26(s, 1H, exchanged with D_2O), 3.63(dd, J= 4.39, 12.69 Hz, 1H), 3.90(dd, J= 2.93, 12.69 Hz, 1H), 4.69(m, 1H) (Fig. 5)

^{13}C NMR ($CDCl_3$) δ(ppm.): 23.24(t), 28.71(t), 63.76(t), 81.53(d), 178.99(s) (Fig. 6)

2.3.3 Preparation of 5-tosyloxymethyl-2-tetrahydrofuranone(43)[20,22]

Powdered p-toluenesulfonyl chloride (52.8 g., 0.278 mole) was added in one portion to an ice-cooled and stirred solution of (42) (25.0 g., 0.216 mole) and N,N-dimethylaminopyridine (2.63 g., 0.022 mole) in dry pyridine (140 mL.). The mixture was stirred for 2 hours at 0 to 10°C. The solvent was removed in vacuo to dryness to give residue, which was solubilized in ethyl acetate. The ethyl acetate solution was washed with water and sodium chloride solution, dried over anhydrous magnesium sulfate and concentrated in vacuo. The crystalline residue was recrystallized from ethyl acetate-ether mixture to give 5-tosyloxymethyl-2-tetrahydrofuranone(43) as colorless needle (53.5 g., 91.9 %yield), m.p. 83.0-84.0°C (lit.[22], 85-86°C); R_f = 0.39 (chloroform); elemental analysis found: C 53.12 %, H 5.28 %. $C_{12}H_{14}O_5S$ requires: C 53.33 %, H 5.19 %.

IR ν_{max}^{KBr} (cm^{-1}): 3030, 2960, 1770, 1660, 1445, 1365, 1190, 1173, 815 (Fig. 7) as reported in lit.[22].

^{1}H NMR ($CDCl_3$) δ(ppm.): 2.10-2.62(m, 7H), 4.16(d, 2H), 4.65(m, 1H), 7.58(dd, J= 8.30, 28.08 Hz, 4H) (Fig. 8) as reported in lit.[22].

^{13}C NMR ($CDCl_3$) δ(ppm.): 21.26(q), 23.40(t), 27.85(t), 70.10(t), 75.68(d), 127.85(d), 130.07(d), 132.13(s), 145.40(s), 176.12(s) (Fig. 9)

2.3.4 Preparation of 5-azidomethyl-2-tetrahydrofuranone (44)[20]

To a stirred solution of (43) (36.0 g., 0.133 mole) in N,N-dimethylformamide (150 mL.) was added sodium azide (52 g., 0.800 mole), and the mixture was refluxed for 3 hours. After cooling, the solvent was removed in vacuo, the residue was shaked with chloroform (300 mL.) and filtered through a Celite pad, and the pad was washed with chloroform. The combined filtrate and washings was concentrated to give crude oil which was purified by column chromatography on silica gel using 20% acetone in hexane as the eluant to give a yellow-colored liquid of 5-azidomethyl-2-tetrahydrofuranone(44) (10.2g., 54.3 %yield); R_f = 0.62 (chloroform).

IR ν_{max}^{neat} (cm^{-1}): 2940, 2100, 1780, 1180 (Fig. 10) as reported in lit.[20].

^{1}H NMR ($CDCl_3$) δ(ppm.): 1.98-2.27(m, 4H),
3.44(dd, J= 4.88, 13.43 Hz, 1H),
3.65(dd, J= 3.91, 13.43 Hz, 1H),
4.66(m, 1H) (Fig. 11) as
reported in lit.[20].

^{13}C NMR ($CDCl_3$) δ(ppm.): 24.54(t), 26.78(t), 54.17(t),
78.23(d), 176.55(s) (Fig. 12)

2.3.5 **Preparation of 5-hydroxy-2-piperidone(45)[20]**

To a stirred solution of (44) (8.1 g., 0.057 mole) in dry methanol (250 mL.) was added 10% palladium on carbon and the mixture was stirred overnight under hydrogen atmosphere. The mixture was filtered, and the filtrate was evaporated to dryness in vacuo. The residue was purified by column chromatography on silica gel using 10% methanol in chloroform as the eluant to yield, after evaporation, 5-hydroxy-2-piperidone(45) (4.84 g., 73.2 %yield), which was recrystallized from a mixture of methanol and ether to give colorless prism product with m.p. 126.0-127.0°C (lit.[20], 124-125°C); R_f = 0.47 (methanol:chloroform = 1:4 v/v); elemental analysis found: C 51.74 %, H 7.95 %, N 12.07 %. $C_5H_9NO_2$ requires: C 52.17 %, H 7.82 %, N 12.17 %.

IR ν_{max}^{KBr} (cm^{-1}): 3280, 3200, 2960, 2920, 1635, 1500,
1290, 1090 (Fig. 13)

^{1}H NMR ($CDCl_3$) δ(ppm.): 1.89(t, 2H), 2.29(m, 2H),
3.26(m, 2H), 4.00(m, 1H),
4.87(s, 1H, exchanged with D_2O)
7.16(s, 1H, exchanged with D_2O)

(Fig. 14)

^{13}C NMR ($CDCl_3$) δ(ppm.): 27.68(t), 28.22(t), 48.43(t), 62.41(d), 171.14(s) (Fig. 15)

2.3.6 **Preparation of 5-methoxy-1-methyl-2-piperidone(47a)**[26]

After stirring potassium hydroxide (0.78 g., 13.9 mmole) in dimethyl sulfoxide (3 mL.) for 5 minutes at room temperature, powdered (45) (0.20 g., 1.7 mmole) was added, followed by methyl iodide (0.43 mL., 0.97 g., 6.8 mmole). Stirring was continued for 45 minutes, then the mixture was poured into water (10 mL.) and extracted with dichloromethane (3x20 mL.). The combined organic extracts were washed with water (2x10 mL.) and dried over anhydrous magnesium sulfate. The solvent was removed in vacuo to give a residue, after distillation (Kugelrohr apparatus, bath temperature 100 °C, pressure 2 mm.Hg) gave 5-methoxy-1-methyl-2-piperidone(47a) (0.16 g., 64.4 %yield); R_f = 0.65 (methanol:chloroform = 1:9 v/v).

IR ν_{max}^{neat} (cm^{-1}): 2940, 2815, 1630, 1100 (Fig. 16)

^{1}H NMR ($CDCl_3$) δ(ppm.): 1.94(t, 2H), 2.38(t, 2H), 2.93(s, 3H), 3.38(s, 5H), 3.62(m, 1H) (Fig. 17)

^{13}C NMR ($CDCl_3$) δ(ppm.): 25.19(t), 27.85(t), 34.62(q), 53.58(t), 56.07(q), 72.54(d), 169.40(s) (Fig. 18)

MS m/e (% rel int.): 143(M^+, 40.6), 128(58.7), 72(35.6), 58(100.0), 57(45.0), 55(18.9), 44(75.2), 42(36.2) (Fig. 19)

2.3.7 Preparation of 3-hydroxy-5-methoxy-1-methyl-2--piperidone(48a)[27]

The solution of base, lithium 2,2,6,6-tetramethylpiperidide(ca.3.84mmole), was generated by adding 1.38 M n-butyl lithium (2.78 mL., 3.84 mmole) to a stirred solution of 2,2,6,6-tetramethylpiperidine (0.69 mL., 0.57 g., 4.06 mmole) in dry tetrahydrofuran (13 mL.) under nitrogen atmosphere at -78°C which gave a yellow solution and stirring was continued at 0°C for 30 minutes. A solution of (47a) (0.25 g., 1.75 mmole) in dry tetrahydrofuran (7 mL.) was added at -78°C and stirred at 0°C for 1 hour. Oxygen gas was bubbled into the enolate anion solution for 1 hour at 0°C, then an aqueous solution of sodium sulfite (0.24 g., 1.93 mmole) was introduced at 0°C and the mixture was vigorously stirred for 15 minutes. The solvent was removed in vacuo at room temperature, the residue was shaken with dichloromethane. The insoluble material was filtered off and washed with dichloromethane. The filtrate and washings were combined and dried over anhydrous magnesium sulfate. Evaporation of the solvent in vacuo gave a residue which was first purified by PTLC using 4% methanol in chloroform as developing solvent. Distillation of this liquid (Kugelrohr apparatus, bath temperature 120°C, pressure 0.2 mm.Hg) afforded 3-hydroxy-5-methoxy-1-methyl-2-piperidone(48a) as a colorless liquid (0.055 g., 19.8 %yield), R_f = 0.55 (methanol:chloroform = 1:9 v/v)

IR ν_{max}^{neat} (cm^{-1}): 3420, 2950, 2900, 2840, 1650, 1100

(Fig. 20)

^{1}H NMR ($CDCl_3$) δ(ppm.): 1.84(m, 1H), 2.48(m, 1H), 2.94(d, 3H), 3.38(s, 5H), 3.67(m, 1H), 4.24(m, 1H), 4.50(s, br, 1H, exchanged with D_2O) (Fig. 21)

^{13}C NMR ($CDCl_3$) δ(ppm.): 32.34(t), 34.40(t), 34.62(q), 34.78(q), 53.09(t), 53.69(t), 56.18(q), 56.34(q), 64.03(d), 65.55(d), 71.56(d), 71.84(d), 172.11(s) (Fig. 22)

MS m/e (%rel int.): 159(M^+, 49.7), 144(9.3), 127(9.4), 102(20.4), 98(22.1), 87(16.3), 72(8.5), 71(10.2), 58(35.8), 44(100.0), 42(22.3), 41(15.5) (Fig. 23)

2.3.8 **Preparation of 2,4-dibromopentanedioic acid(50)[29]**

A mixture of pentanedioic acid (100 g., 0.75 mole) and thionyl chloride (140 mL., 229.6 g., 1.9 mole) was stirred and warmed until no more sulfur dioxide was generated. The product was heated at 100°C before a 60 watt lamp, while bromine (87 mL., 1.65 mole) was slowly added from a dropping funnel. The mixture was poured into formic acid (80 mL.). The precipitates which seperated from the formic acid solution were collected and washed with a small amount of chloroform and hexane, and was then extracted with boiling chloroform until the melting point of the undissolved portion was 160°C. The undissolved portion, on

recrystallising from a mixture of ether and chloroform, yielded meso-2,4-dibromopentanedioic acid(50) (54.30 g., 24.9 %yield) with m.p. 164.0-165.0°C (lit.29, 170°C); elemental analysis found: C 20.82 %, H 2.03 %. $C_5H_6Br_2O_4$ requires: C 20.71%, H 2.09%.

IR ν_{max}^{KBr} (cm^{-1}): 3300-2500, 1730, 1420, 1170 (Fig. 24)

^1H NMR ($CDCl_3$+DMSO-d_6) δ(ppm.):

2.58(t, 2H), 4.42(t, 2H),

12.40(s, br, 2H, exchanged with D_2O) (Fig. 25)

^{13}C NMR ($CDCl_3$+DMSO-d_6) δ(ppm.):

38.63(t), 44.69(d), 169.67(s)

(Fig. 26)

2.3.9 Preparation of 2,4-dihydroxypentanedioic acid(51)

Method A [29]

A solution of (50) (2.5 g., 8.68 mmole) in 2 N aqueous sodium carbonate solution (25 mL.) was refluxed for 1.5 hours, then acidified with 10% aqueous hydrochloric acid solution. The resulting solution was evaporated to dryness and the residue was extracted with hot acetone. The syrup, remaining after evaporation of acetone, solidified completely after a few days. This solid, after recrystallizing from acetone, yielded 2,4-dihydroxypentanedioic acid(51) as white crystal with m.p. 152.0-154.0°C (0.28 g., 19.7 %yield); elemental analysis found: C 36.56 %, H 4.91%, O 58.53%. $C_5H_8O_6$ requires: C 36.59 %, H 4.91 %, O 58.50 %.

IR ν_{max}^{KBr} (cm^{-1}): 3510, 3300-2500, 1735, 1685, 1220, 1100

(Fig. 27)

^{1}H NMR ($CDCl_3$+DMSO-d_6) δ(ppm.):

2.01(dd, 2H), 4.30(dd, 2H),

6.7-9.0(s, br, 4H, exchanged with

D_2O) (Fig. 28)

^{13}C NMR ($CDCl_3$+DMSO-d_6) δ(ppm.):

36.76(t), 64.55(d), 174.09(s)

(Fig. 29)

Method B

To a solution of (50) (40.0 g., 0.14 mole) in water was added powdered silver oxide (46.0 g., 0.17 mole), stirred until no more bromide ion was exchanged. The excess silver ion was precipitated with 10% aqueous hydrochloric acid. All the silver salts were filtered off, the filtrate was evaporated to dryness. The residue was recrystallized from acetone to yield 2,4-dihydroxypentanedioic acid(51) with m.p. 146.0-148.0°C (20.36 g., 89.4 %yield).

IR of this product was found to be identical with the IR spectrum of 2,4-dihydroxypentanedioic acid from method A.

2.3.10 **Preparation of 5-carboxy-3-hydroxy-2-tetrahydrofuranone(52)**

Method A[29]*

Powder of (51) (2.3 g., 14.0 mmole) was heated at 165°C (in an oil bath) for 5 minutes to form a colorless, very viscous gum. After solidifying to a crystalline substance, recrystallizing from ethyl acetate yielded 5-carboxy-3-hydroxy--2-tetrahydrofuranone(52) as white cluster needle with m.p. 153.0-155.0°C (1.54 g., 74.4 %yield); elemental analysis found C 40.87%, H 4.09 %, O 55.04 %. $C_5H_6O_3$ requires: C 41.07 %, H 4.11 %, O 54.82%.

IR ν_{max}^{KBr} (cm^{-1}): 3460, 3200-2500, 1800, 1742, 1420, 1250, 1188, 1130 (Fig. 30)

^1H NMR ($CDCl_3$+DMSO-d_6) δ(ppm.): 2.06(m, 1H), 2.78(m, 1H), 4.47(dd, J= 8.43, 10.01 Hz, 1H), 4.79(dd, J= 6.59, 10.01 Hz, 1H) (Fig. 31)

^{13}C NMR ($CDCl_3$+DMSO-d_6) δ(ppm.): 33.02(t), 65.20(d), 70.23(d), 168.89(s), 174.36(s) (Fig. 32)

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* Increasing the starting material, the designated product was decreased and yielded an amorphous, colorless solid, insoluble in ethyl acetate, as the major product.

Method B

A suspension of(51) (1.0 g., 6.1 mmole) in toluene and a catalytic amount of conc. hydrochloric acid was refluxed for 1.5 hours. A viscous, colorless gum seperated from the toluene. The solidified gum, recrystallizing from ethyl acetate afforded 5-carboxy-3-hydroxy-2-tetrahydrofuranone(52) as method A (0.3 g., 33.7 %yield).

2.3.11 **Preparation of 5-methoxycarbonyl-3-hydroxy-2-tetrahydrofuranone(53)**

Method A[21]

A solution of (52) (3.46 g., 23.7 mmole) and p-toluenesulfonic acid (0.08 g.) in methanol (5.2 mL.) and benzene (12 mL.) was refluxed for 5 hours and the solvent was evaporated in vacuo. The residue was purified by column chromatography on silica gel using chloroform as eluant to give a pale yellow liquid, identified as dimethyl 2,4-dihydroxypentane-dioate(53a) (3.13 g., 68.6 %yield).

IR ν_{max}^{neat} (cm^{-1}): 3400-3200, 2960, 1760-1720, 1440, 1100 (Fig. 33)

^{1}H NMR ($CDCl_3$) δ(ppm.): 2.18(t, 2H), 3.34(s, br, 1H, exchanged with D_2O), 3.79(s, 6H), 4.36(t, 2H) (Fig. 34)

^{13}C NMR ($CDCl_3$) δ(ppm.): 38.0, 52.5, 67.5, 175.1 (Fig. 35)

Method B

A solution of diazomethane in ether was introduced to a stirred solution of (52) (3.46 g., 23.7 mmole) in methanol at -78°C until a yellow solution was obtained. The solvent was removed in vacuo to yield a pale yellow liquid which gave the identical ^{1}H NMR spectrum with the dimethyl 2,4-dihydroxypentanedioate(53a) from method A.

2.3.12 **The spectroscopic data of odoram**

The spectroscopic data of odoram were

IR ν_{max}^{KBr} (cm^{-1}): 3260, 2980, 2940, 1620, 1210, 1080, 920 (Fig. 36)

^{1}H NMR (D_2O) δ(ppm.): 2.21(ddd, J=4.64, 10.92, 14.42 Hz, 1H), 2.50(dddd, J=1.96, 7.64, 14.42 Hz, 1H), 3.04(s, 3H), 3.18(ddd, J=1.96, 12.94 Hz, 1H), 3.95(dd, J = 4.63, 12.94 Hz, 1H), 4.19(dd, J = 7.60, 10.92 Hz, 1H), 4.63(m, 1H) (Fig. 37, 38)

^{13}C NMR (D_2O) δ(ppm.): 40.85(t), 45.72(q), 65.28(t), 72.05(d), 72.65(d), 175.42(s) (Fig. 39)

MS m/e (rel int.): 145(M^+,3.0), 101(9.6), 100(100.0), 82(35.0), 72(3.3), 44(10.1), 42(23.6) (Fig. 40)

Elemental Analysis found: C 49.93 %, H 7.62 %, N 9.79 %

$C_6H_{11}NO_3$ requires: C 49.66 %, H 7.59 %, N 9.66 %

2.3.13 **Methylation of odoram**

A mixture of odoram (0.10 g., 0.69 mmole) and sodium t-butoxide, in situ generated, in dry t-butanol was stirred at room temperature overnight, then excess amount of methyl iodide (1 mL., 2.28 g., 16.06 mmole) was added and stirring was continued overnight. The mixture was filtered. The insoluble material was extracted with methanol and filtered again. The methanol solution was evaporated in vacuo to give a residue, white precipitate (0.04 g.).

IR ν_{max}^{neat} (cm^{-1}): 3600-3200, 1630, 1400, 1200, 1050 (Fig. 41)

^{1}H NMR (D_2O) δ(ppm.): 2.43(m, 2H), 2.61(dd, J = 6.84, 10.25 Hz, 1H), 3.11(s, 3H), 3.40(s, 3H), 3.50(dd, J = 3.91, 12.94 Hz, 1H), 4.07(dd, J = 6.23, 12.94 Hz, 1H), 4.38(dd, J = 7.82, 10.26 Hz, 1H) (Fig. 42)

^{13}C NMR (D_2O) δ(ppm.): 38.57, 51.25, 56.88, 68.96, 76.55, 78.82, 173.14 (Fig. 43)

The other method for methylating odoram, using sodium metal in stead of sodium t-butoxide as described above, was also attempted. The methylated product was not obtained since the ^{1}H NMR spectrum of the product was identical with the ^{1}H NMR of odoram, the starting material.