Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Materials:

A facile synthesis of vicinal *cis*-diols from olefins catalyzed by in situ generated Mn_xO_y nanoaggregates

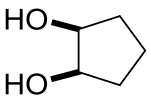
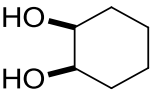
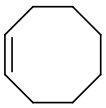
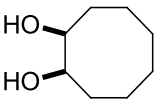
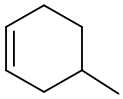
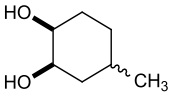
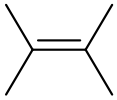
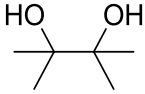
Diğdem DALMIZRAK[a], Haydar GÖKSU[a,b], Mehmet Serdar GÜLTEKİN*[a]

E-mail: gultekin@atauni.edu.tr;

Generally Procedure: To a magnetically stirred solution of $KMnO_4$ (5.0 mmol 0.79g) in water:acetone (1:2, 15 mL) was added olefin (10 mmol) in 5 mL water:aceton (1:2) and then H_2O_2 (30 mmol, 36%, 2.80 mL) was slowly added in mixture partially for an hour in nitrogen atmosphere at 0°C. After completion of the addition, the solution was stirred additional 1 hour. The reaction was monitored by TLC and approximately all of the reaction was finished at 2 h. The ethyl acetate was added to reaction mixture and this mixture was washed with water and dried over sodium sulfate. After removal of the solvent, the residue was determined by comparison of the NMR data with those of the corresponding visinal *cis*-diols. [5]

H_2O_2 ***concentration determination procedure:*** To a magnetically stirred solution of $KMnO_4$ (0.5 eq., 5.0 mmol 0.79g) in water:acetone (1:2, 15 mL) was added olefin (10 mmol) in 5 mL water:aceton (1:2) and then H_2O_2 [36%, (2.0 eq., 20.0 mmol, 1.86 mL) or (3.0 eq., 30.0 mmol 2.80 mL) or (3.5 eq., 35.0 mmol 3.27 mL) or (4.0 eq., 40.0mmol, 3.72 mL)] was slowly added (in different reaction vessels) in mixture partially for an hour in nitrogen atmosphere at 0°C, according to **table S1**. After completion of the addition, the solution was stirred additional 1 hour. The reaction was monitored by TLC and approximately all of the reaction was finished at 2 h. The ethyl acetate was added to reaction mixture and this mixture was washed with water and dried over sodium sulfate. After removal of the solvent, the residue was determined with the NMR data.

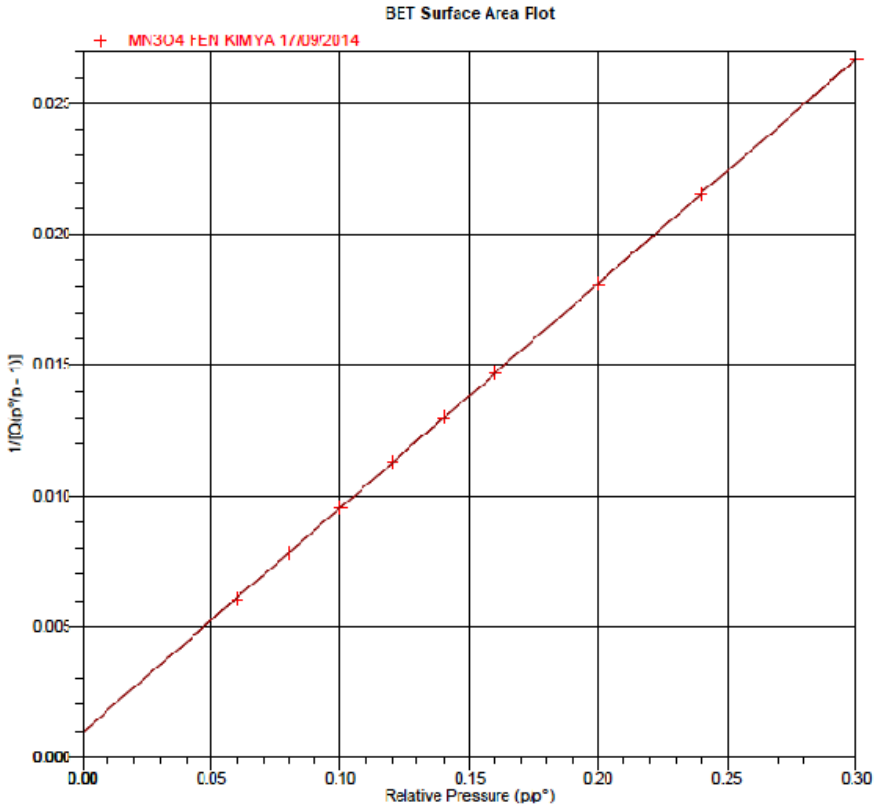
Table S1. H_2O_2 concentration was optimized by 0.5 eq. $KMnO_4$ (5.0 mmol).

entry	***Olefin***	***Visinal cis-diol***	***Yields (%)***			
			2eq	***3eq***	***3,5eq***	***4eq***
1	 **1**	 **2**	74	**90**	85	82
2	 **3**	 **4**	67	**82**	65	63
3	 **7**	 **8**	72	**79**	75	71
4	 **13**	 syn/anti(4:6) **14a,b**	56	**72**	63	50
5	 **25**	 **26**	70	**66**	53	49

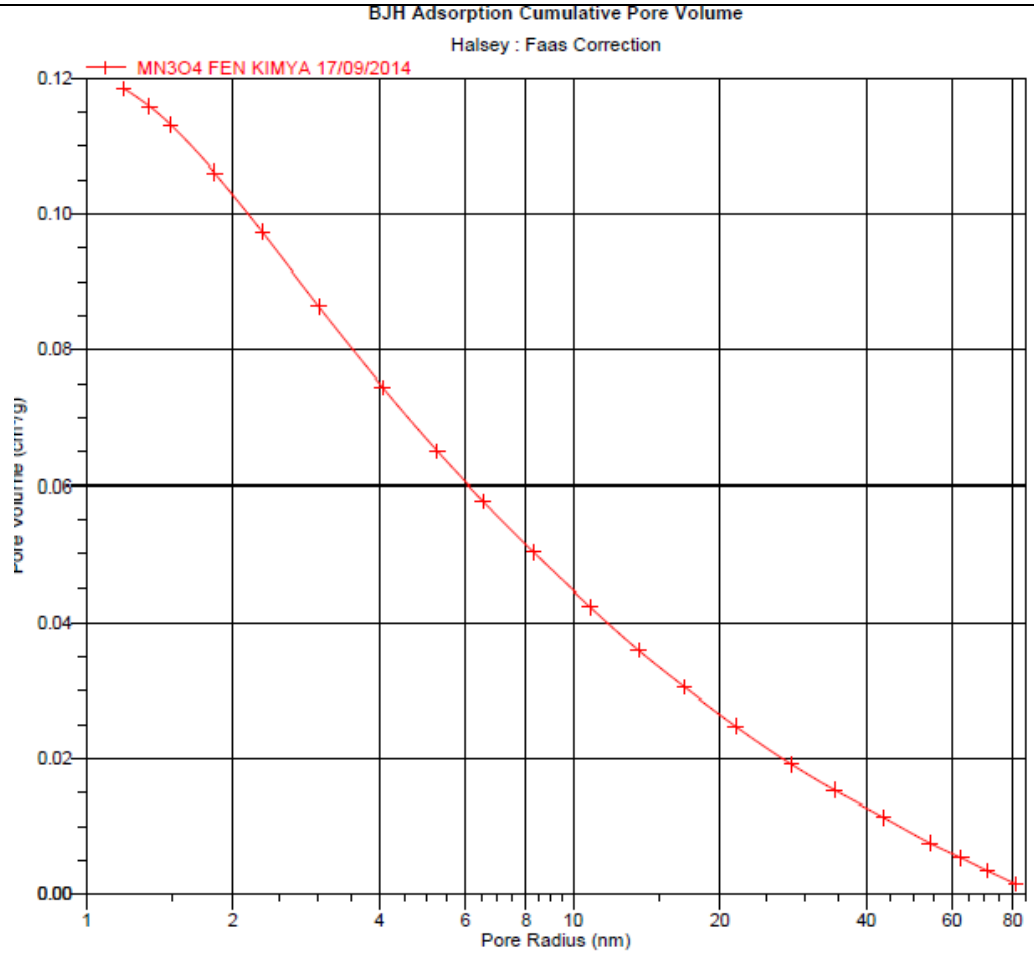
Reaction conditions: All reactions were performed in aqueous acetone (20.0 mL, water– acetone= 1: 2 v/v) using 5.0 mmol $KMnO_4$, 10.0 mmol olefin and H_2O_2 in N_2 atm. at 0°C.

The dihydroxylation reaction with MnO_2-H_2O_2 , Mn_3O_4-H_2O_2 , in situ generated Mn_xO_y-H_2O_2 and some olefins procedure:

To a magnetically stirred solution of manganes oxide derivative [(5.0 mmol, 0.44g MnO_2) or (5.0 mmol, 1.15g Mn_3O_4) or (0.4 g Mn_xO_y)] in water:acetone (1:2, 15 mL) was added olefin (10 mmol) in 5 mL water:aceton (1:2) and then H_2O_2 [(30.0 mmol, 36%, 2.80 mL) or (10.0 mmol, 36%, 0.94 mL)] was slowly added in mixture partially for an hour in nitrogen atmosphere at 0°C, according to **table S2**. After completion of the addition, the solution was stirred additional 1 hour. The reaction was monitored by TLC and approximately all of the reaction was finished at 2 h. The ethyl acetate was added to reaction mixture and this mixture was washed with water and dried over sodium sulfate. After removal of the solvent, the residue was determined with NMR data.



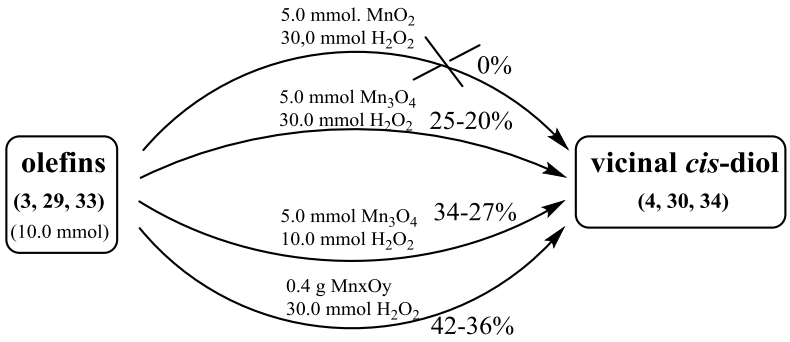
Relative Pressure (p/p°)	Statistical Thickness (nm)	Quantity Adsorbed (cm³/g STP)
0.060068341	0.33383	10.5605
0.080060604	0.35177	11.1297
0.100077439	0.36789	11.6303
0.120055811	0.38282	12.0897
0.140066309	0.39699	12.5202
0.160060722	0.41062	12.9477
0.200017674	0.43690	13.8010
0.240080448	0.46263	14.6668
0.299991431	0.50121	16.0621
0.350004027	0.53437	17.3590
0.449990321	0.60612	20.3575
0.550011638	0.69026	24.0440

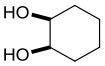
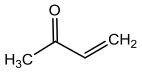
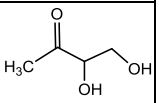
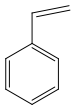
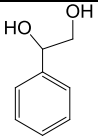


Summary Report
Surface Area Single point surface area at p/p° = 0.300101832: 127.8538 m²/g BET Surface Area: 129.5626 m²/g Langmuir Surface Area: 203.0529 m²/g t-Plot Micropore Area: 19.1249 m²/g t-Plot External Surface Area: 110.4377 m²/g BJH Adsorption cumulative surface area of pores between 0.8500 nm and 150.0000 nm radius: 117.355 m²/g
Pore Volume Single point adsorption total pore volume of pores less than 61.3398 nm radius at p/p° = 0.983963825: 0.262256 cm³/g Single point desorption total pore volume of pores less than 1.4996 nm radius at p/p° = 0.348834999: 3.507639 cm³/g t-Plot micropore volume: 0.008844 cm³/g BJH Adsorption cumulative volume of pores between 0.8500 nm and 150.0000 nm radius: 0.270170 cm³/g
Pore Size Adsorption average pore width (4V/A by BET): 8.09666 nm Desorption average pore width (4V/A by BET): 108.29171 nm BJH Adsorption average pore radius (2V/A): 4.6043 nm

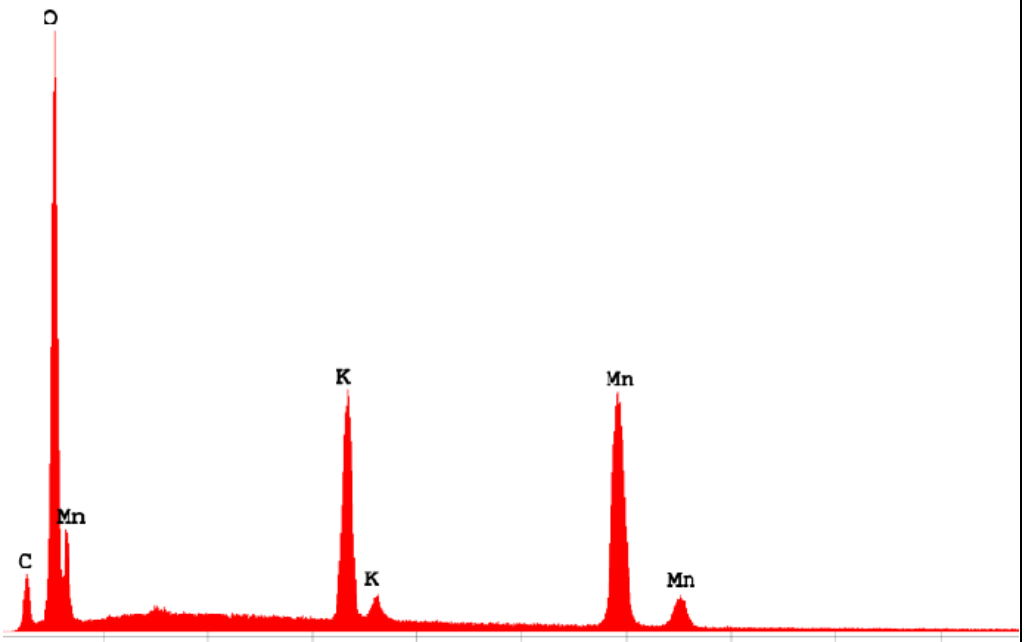
FS-1: (A) BET graphics **(B)** BJT graphics of the solid material recovered at the end of oxidation reactions.

Table S2. The oxidation reaction of cyclohexene **3,** but-3-en-2-one **29** and styrene **33** with MnO_2-H_2O_2, Mn_3O_4-H_2O_2 and in situ generated Mn_xO_y-H_2O_2.



		Yields (%)				
entry	***Olefin* (10.0 mmol)**	***5.0 mmol MnO_2 / 30.0 mmol H_2O_2***	***5.0 mmol Mn_3O_4 / 30.0 mmol H_2O_2***	***5.0 mmol Mn_3O_4 / 10.0 mmol H_2O_2***	***0.4 g Mn_xO_y / 30.0 mmol H_2O_2***	***Visinal cis-diol***
1	 **3**	No reaction	25	34	42	 **4**
2	 **29**	No reaction	22	30	40	 **30**
3	 **33**	No reaction	20	27	36	 **34**

Reaction conditions: All reactions were performed in aqueous acetone (20.0 mL, water–acetone= 1: 2 v/v) using manganese oxide derivatives, olefins and H_2O_2 in N_2 atm. at 0ºC.

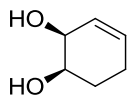


EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt. %	At. %	K-Ratio	Z	A	F
C K	4.35	10.38	0.0145	1.1317	0.2949	1.0005
O K	29.05	51.99	0.1183	1.1098	0.3663	1.0015
K K	13.79	10.10	0.1360	1.0031	0.9659	1.0177
MnK	52.81	27.53	0.4793	0.9118	0.9953	1.0000

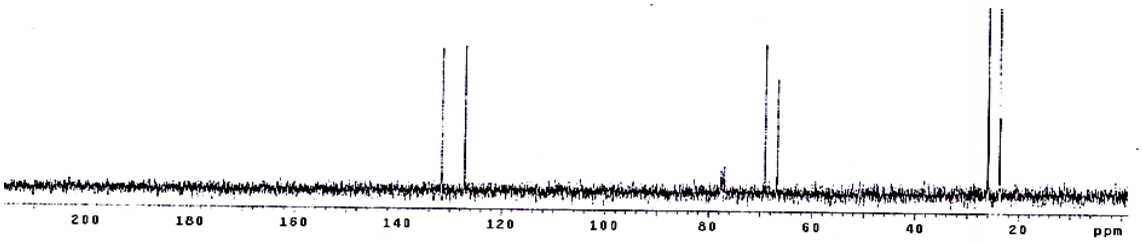
FS-2: EDX graphics of the solid material recovered at the end of oxidation reactions.

^{1}H-and ^{13}C-NMR SPECTRA

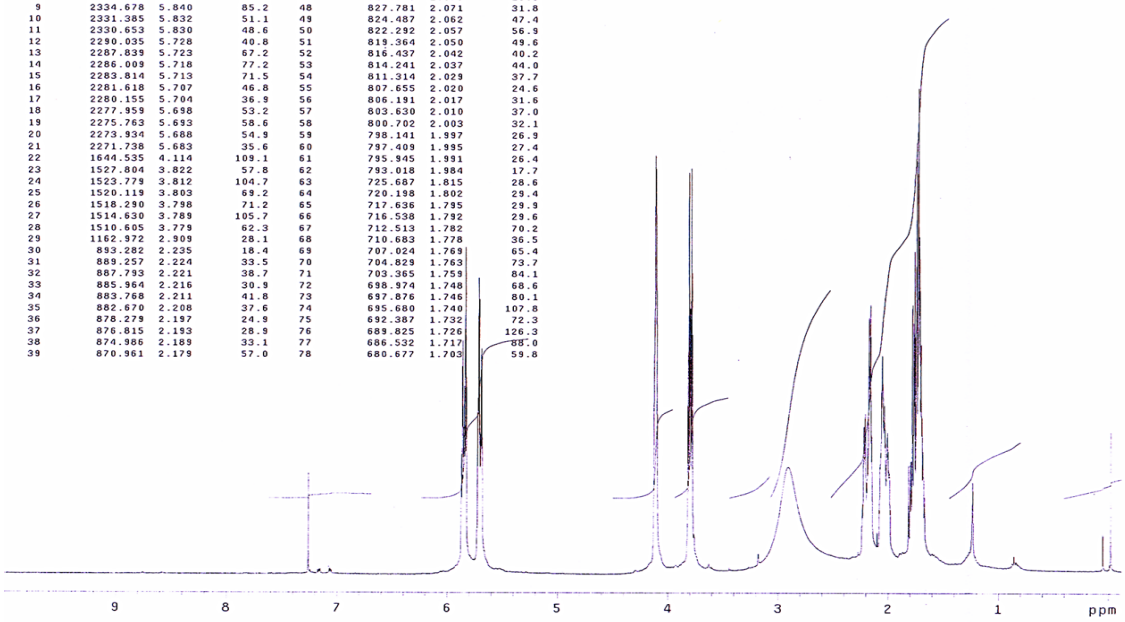


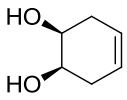
^{1}H-NMR (400MHz, $CDCl_3$, ppm): δ=5.9(2H, m), 5.8(1H, m), 4.1(brs) 3.9(2H, m), 2.1-2.2(4H m), **^{13}C-NMR (100MHz, $CDCl_3$, ppm):** δ=131.5, 127.2, 69.1, 66.7, 26.0, 23.8

INDEX	FREQUENCY	PPM	HEIGHT
1	13222.033	131.536	37.8
2	12782.134	127.160	38.6
3	6946.131	69.102	35.5
4	6705.560	66.709	30.3
5	2614.035	26.005	49.7
6	2389.044	23.767	49.6



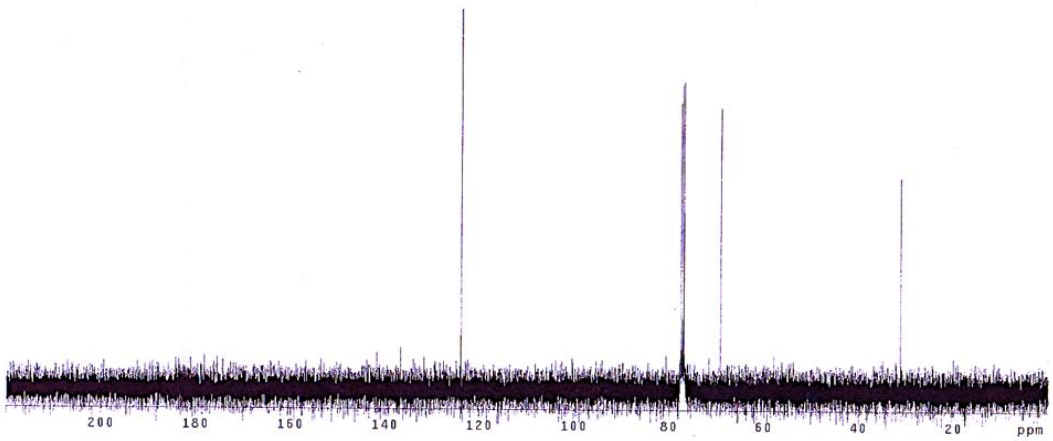
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2902.600	7.261	26.5	40	869.497	2.175	66.8	79	677.384	1.694	40.7
2	2348.583	5.875	32.0	41	867.301	2.170	51.7	80	673.725	1.685	32.1
3	2347.851	5.873	32.9	42	865.472	2.165	70.2	81	667.870	1.671	20.3
4	2344.558	5.865	60.8	43	864.008	2.161	61.9	82	492.955	1.233	24.1
5	2341.265	5.857	38.4	44	859.983	2.151	36.4	83	-8.368	-0.021	37.2
6	2340.533	5.855	38.1	45	858.519	2.148	25.4				
7	2338.703	5.850	48.9	46	832.538	2.083	19.8				
8	2337.971	5.848	48.4	47	829.976	2.076	28.8				
9	2334.678	5.840	85.2	48	827.781	2.071	31.8				
10	2331.385	5.832	51.1	49	824.487	2.062	47.4				
11	2330.653	5.830	48.6	50	822.292	2.057	56.9				
12	2290.035	5.728	40.8	51	819.364	2.050	49.6				
13	2287.839	5.723	67.2	52	816.437	2.042	40.2				
14	2286.009	5.718	77.2	53	814.241	2.037	44.0				
15	2283.814	5.713	71.5	54	811.314	2.029	37.7				
16	2281.618	5.707	46.8	55	807.655	2.020	24.6				
17	2280.155	5.704	36.9	56	806.191	2.017	31.6				
18	2277.959	5.698	53.2	57	803.630	2.010	37.0				
19	2275.763	5.693	58.6	58	800.702	2.003	32.1				
20	2273.934	5.688	54.9	59	798.141	1.997	26.9				
21	2271.738	5.683	35.6	60	797.409	1.995	27.4				
22	1644.535	4.114	109.1	61	795.945	1.991	26.4				
23	1527.804	3.822	57.8	62	793.018	1.984	17.7				
24	1523.779	3.812	104.7	63	725.687	1.815	28.6				
25	1520.119	3.803	69.2	64	720.198	1.802	29.4				
26	1518.290	3.798	71.2	65	717.636	1.795	29.9				
27	1514.630	3.789	105.7	66	716.538	1.792	29.6				
28	1510.605	3.779	62.3	67	712.513	1.782	70.2				
29	1162.972	2.909	28.1	68	710.683	1.778	36.5				
30	893.282	2.235	18.4	69	707.024	1.769	65.4				
31	889.257	2.224	33.5	70	704.829	1.763	73.7				
32	887.793	2.221	38.7	71	703.365	1.759	84.1				
33	885.964	2.216	30.9	72	698.974	1.748	68.6				
34	883.768	2.211	41.8	73	697.876	1.746	80.1				
35	882.670	2.208	37.6	74	695.680	1.740	107.8				
36	878.279	2.197	24.9	75	692.387	1.732	72.3				
37	876.815	2.193	28.9	76	689.825	1.726	126.3				
38	874.986	2.189	33.1	77	686.532	1.717	88.0				
39	870.961	2.179	57.0	78	680.677	1.703	59.8				



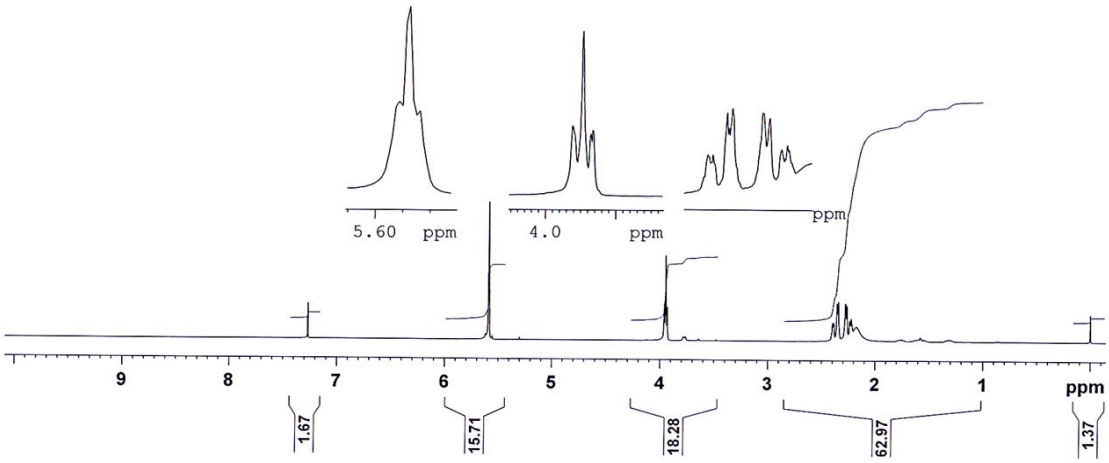


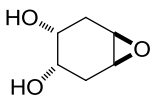
^{1}H-NMR (400MHz, $CDCl_3$, ppm): δ= 5.6 (2H, brs), 3.9 (2H, td, *J*=1.4, 5.7), 2.3(4H, m), 1.8 (2H, (OH) brs),**^{13}C-NMR (100MHz, $CDCl_3$, ppm):** δ=123.9, 69.1, 31.2

INDEX	FREQUENCY	PPM	HEIGHT
1	12454.958	123.905	97.4
2	7795.687	77.553	73.6
3	7763.611	77.234	78.1
4	7731.535	76.915	78.9
5	6946.131	69.102	72.3
6	3137.790	31.216	54.6



Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	5.5912	2237.2069	1240618.14
2	5.5880	2235.9265	2270933.52
3	5.5843	2234.4460	1057531.20
4	3.9613	1585.0350	659194.73
5	3.9472	1579.3932	1448252.95
6	3.9357	1574.7917	550434.79
7	3.9325	1573.5113	617865.09
8	2.4052	962.3927	151360.84
9	2.3973	959.2317	340120.49
10	2.3898	956.2307	277329.91
11	2.3858	954.6302	338585.65
12	2.3552	942.3862	648069.80
13	2.3510	940.7056	566864.38
14	2.3435	937.7047	728667.61
15	2.2770	911.0960	694184.70
16	2.2622	905.1741	626746.05
17	2.2512	900.7727	146002.50
18	2.2356	894.5306	385601.52
19	2.2279	891.4496	309980.70
20	2.2235	889.6891	407935.99
21	2.2196	888.1286	363321.80
22	2.2136	885.7278	270910.38
23	2.1719	869.0424	269311.80

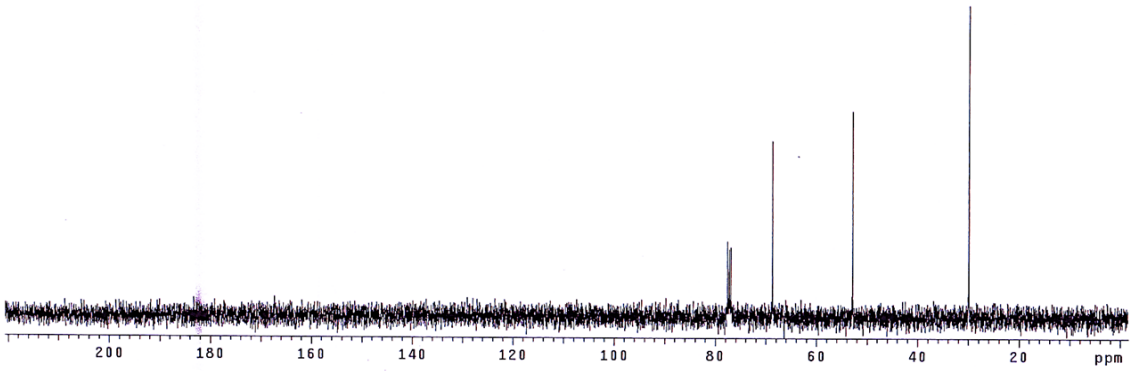




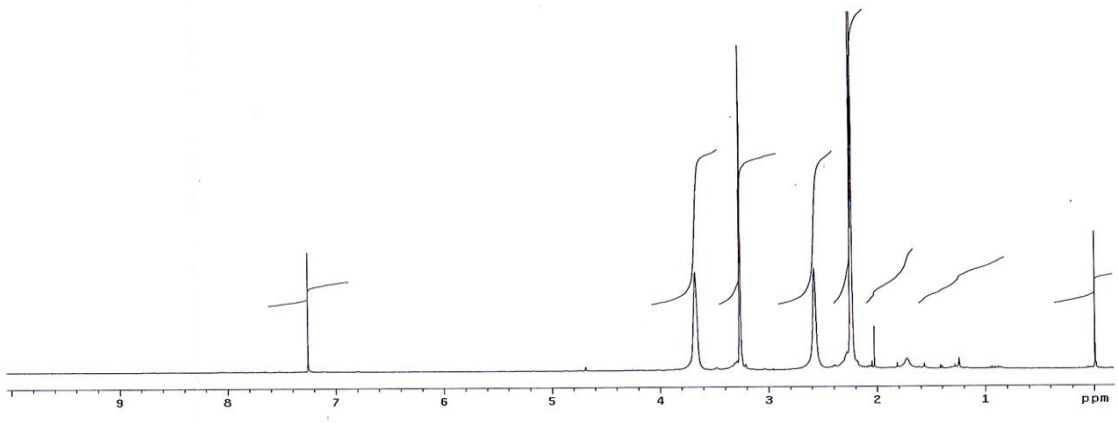
^{1}H-NMR (400MHz, $CDCl_3$, ppm): δ=3.67(2H br s), 3.26(2H t) 2.6(2H brs), 2.2(4H dt)

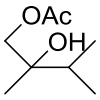
^{13}C-NMR (100MHz, $CDCl_3$, ppm): δ=68.7, 52.8, 29.9

INDEX	FREQUENCY	PPM	HEIGHT
1	6903.973	68.682	40.5
2	5307.046	52.796	47.5
3	3000.780	29.853	71.8



INDEX	FREQUENCY	PPM	HEIGHT
1	2901.503	7.258	35.9
2	1467.425	3.671	29.7
3	1303.855	3.262	75.9
4	1302.757	3.259	97.6
5	1301.659	3.256	80.6
6	1028.310	2.572	30.6
7	908.285	2.272	5.6
8	896.210	2.242	107.5
9	894.746	2.238	94.5
10	891.087	2.229	95.0
11	889.988	2.226	107.4
12	888.525	2.223	83.0
13	808.387	2.022	13.2
14	-5.074	-0.013	41.3

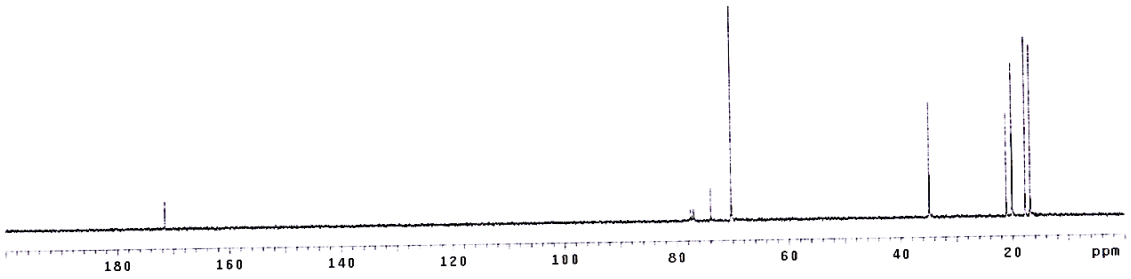




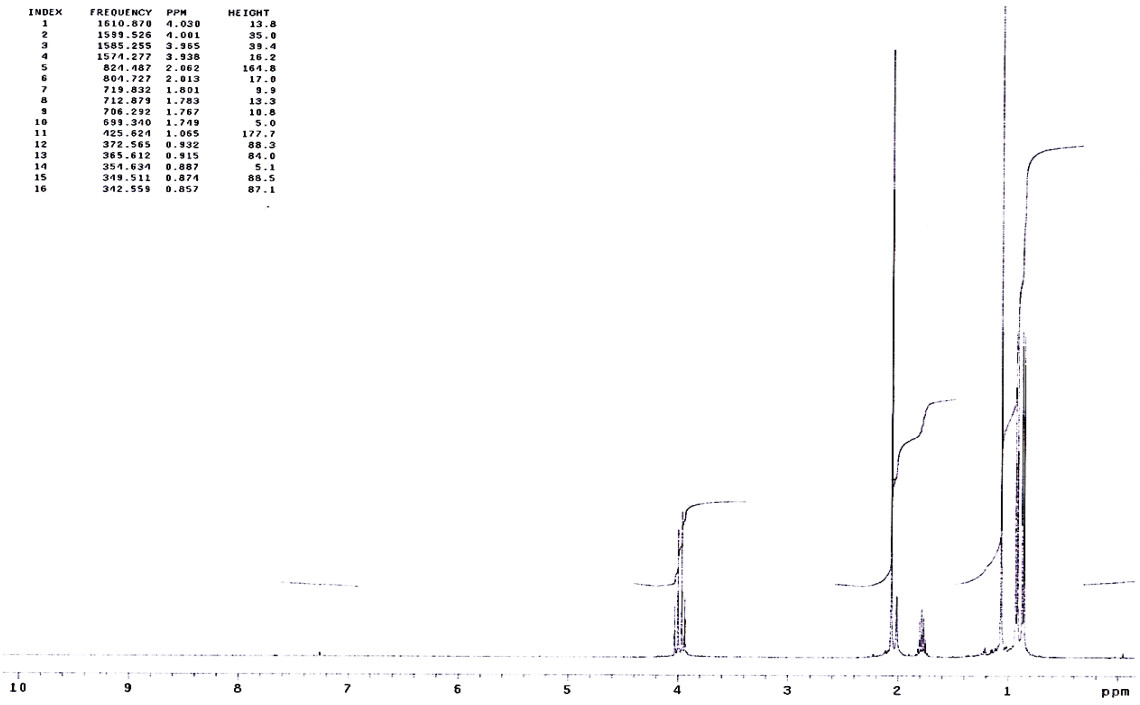
^{1}H-NMR (400MHz, $CDCl_3$, ppm): δ=3.92(1H, q, *J*=10.98), 2.27(1H, brs), 2.0(3H, s), 1.09(1H, dd, *J*=7.2), 0.86(3H, d *J*=6.95), 0.8(3H, d, *J*=6.95)

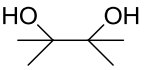
^{13}C-NMR (100MHz, $CDCl_3$, ppm): δ=171.4, 73.82, 70.2, 34.73, 21.04, 20.06, 17.73, 16.79

INDEX	FREQUENCY	PPM	HEIGHT
1	17233.848	171.447	6.6
2	7432.065	73.936	8.0
3	7065.080	70.285	52.2
4	3503.570	34.854	28.1
5	2120.318	21.093	25.3
6	2015.556	20.051	37.4
7	1783.089	17.739	43.6
8	1693.060	16.843	41.9



INDEX	FREQUENCY	PPM	HEIGHT
1	1610.870	4.030	13.8
2	1599.526	4.001	35.0
3	1585.255	3.965	39.4
4	1574.277	3.938	16.2
5	821.487	2.062	164.8
6	804.727	2.013	17.0
7	719.832	1.801	9.9
8	712.879	1.783	13.3
9	706.292	1.767	10.8
10	699.340	1.749	5.0
11	425.624	1.065	177.7
12	372.565	0.932	88.3
13	365.612	0.915	84.0
14	354.634	0.887	5.1
15	349.511	0.874	88.5
16	342.559	0.857	87.1

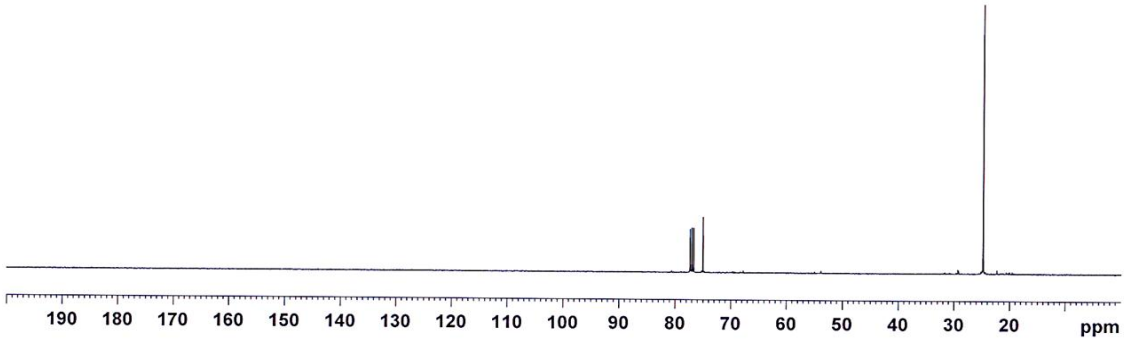




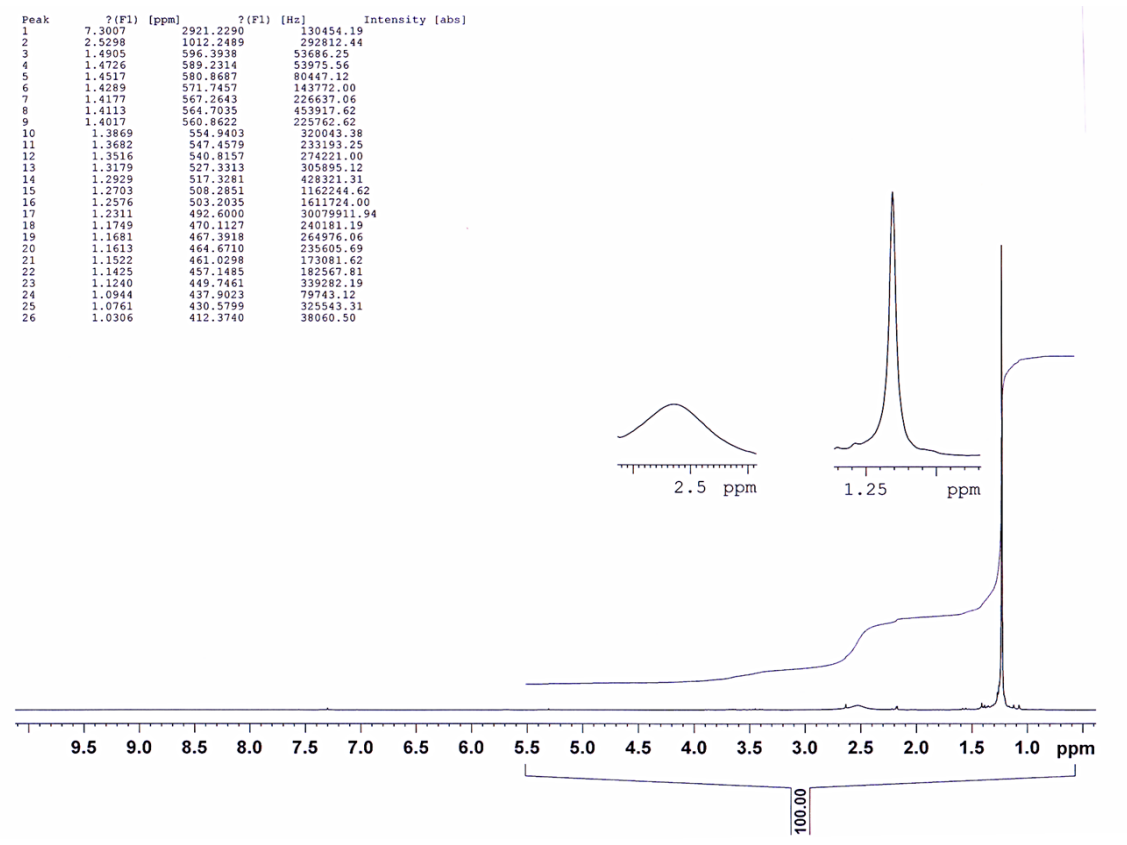
^{1}H-NMR (400MHz, $CDCl_3$, ppm): δ=1.38-1.31(12H, s), 2.5(1H, brs)

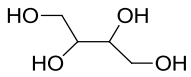
^{13}C-NMR (100MHz, $CDCl_3$, ppm): δ=75.26, 24.8

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	80.6628	8115.7077	77593.88
2	77.3619	7783.5950	1892688.25
3	77.0444	7751.6504	2097800.00
4	76.7259	7719.6053	1728848.78
5	75.2583	7571.9460	92484.56
6	75.0524	7551.2298	2599003.44
7	74.8439	7530.2520	65571.16
8	69.6165	7004.3088	61293.50
9	67.8020	6821.7470	70397.03
10	55.0019	5533.8935	65616.22
11	53.8665	5419.6577	109743.31
12	31.6946	3188.8815	65832.06
13	30.8608	3104.9905	53208.25
14	29.6450	2982.6655	55533.53
15	29.3532	2953.3067	202464.25
16	29.2253	2940.4384	144949.59
17	25.2387	2539.3355	98568.50
18	25.1680	2532.2222	121481.34
19	25.0800	2523.3682	124987.44
20	24.9839	2513.6994	179224.47
21	24.7984	2495.0357	9781810.34
22	24.6529	2480.3965	114153.34
23	24.5929	2474.3598	114246.31
24	24.5225	2467.2766	88568.19
25	24.3752	2452.4564	74700.28
26	22.3102	2244.6910	175542.69
27	20.6542	2078.0763	86666.34
28	20.0993	2022.2462	80449.16
29	20.0350	2015.7768	85562.78
30	19.5767	1969.6660	60636.69



Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.3007	2921.2290	130454.19
2	2.5298	1012.2489	292812.44
3	1.4905	596.3938	53686.25
4	1.4726	589.2314	53975.56
5	1.4517	580.8687	80447.12
6	1.4289	571.7457	143772.00
7	1.4177	567.2643	226637.06
8	1.4113	564.7035	453917.62
9	1.4017	560.8622	225762.62
10	1.3869	554.9403	320043.38
11	1.3682	547.4579	233193.25
12	1.3516	540.8157	274221.00
13	1.3179	527.3313	305895.12
14	1.2929	517.3281	428321.31
15	1.2703	508.2851	1162244.62
16	1.2576	503.2035	1611724.00
17	1.2311	492.6000	30079911.94
18	1.1749	470.1127	240181.19
19	1.1681	467.3918	264976.06
20	1.1613	464.6710	235605.69
21	1.1522	461.0298	173081.62
22	1.1425	457.1485	182567.81
23	1.1240	449.7461	339282.19
24	1.0944	437.9023	79743.12
25	1.0761	430.5799	325543.31
26	1.0306	412.3740	38060.50

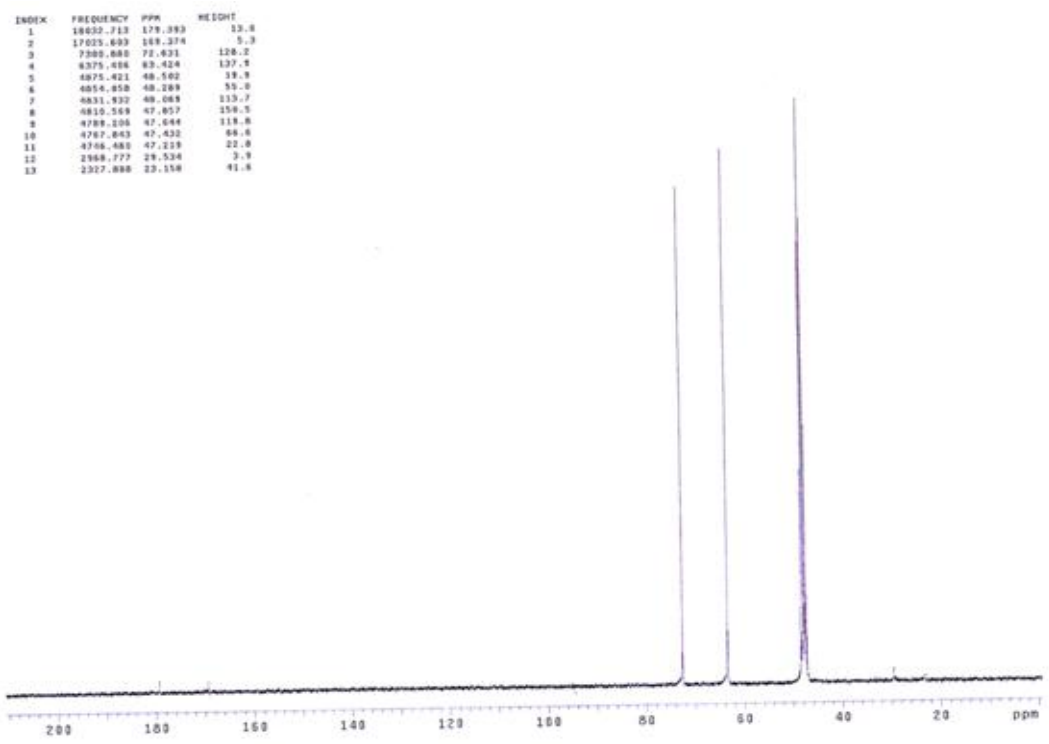




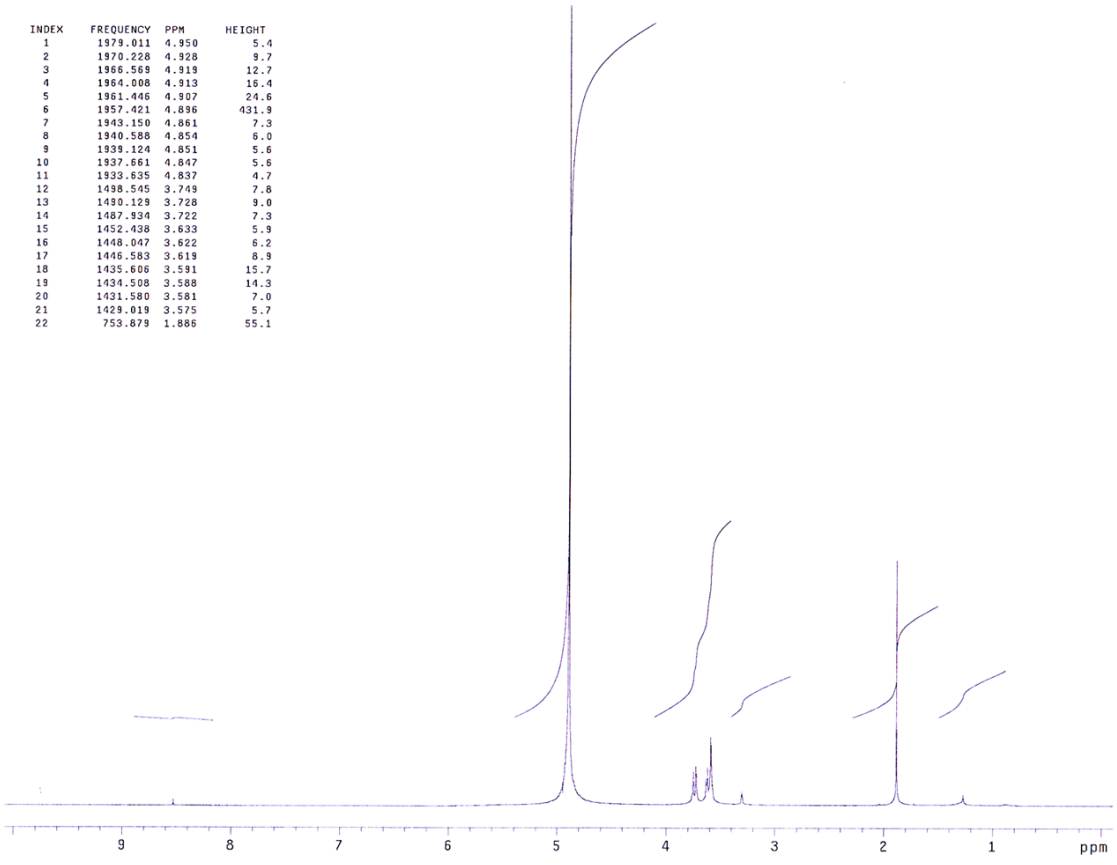
^{1}H-NMR (400 MHz, $CDCl_3$ pmm): δ= 3.75-3.72(2H, m), 3.63-3.57(4H, m)

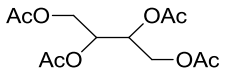
^{13}C-NMR (100MHz, $CDCI_3$, ppm): δ=72.63, 63.42,

INDEX	FREQUENCY	PPM	HEIGHT
1	18032.713	179.393	13.8
2	17025.693	169.374	5.3
3	7380.680	73.431	128.2
4	6375.686	63.424	137.9
5	4875.421	48.502	19.9
6	4854.858	48.289	55.0
7	4831.932	48.069	113.7
8	4810.569	47.857	150.5
9	4788.106	47.644	119.8
10	4767.843	47.432	66.6
11	4746.480	47.219	22.8
12	2968.777	29.534	3.9
13	2327.888	23.158	41.6



INDEX	FREQUENCY	PPM	HEIGHT
1	1979.011	4.950	5.4
2	1970.228	4.928	9.7
3	1966.569	4.919	12.7
4	1964.008	4.913	16.4
5	1961.446	4.907	24.6
6	1957.421	4.896	431.8
7	1943.150	4.861	7.3
8	1940.588	4.854	6.0
9	1939.124	4.851	5.6
10	1937.661	4.847	5.6
11	1933.635	4.837	4.7
12	1498.545	3.749	7.8
13	1490.129	3.728	9.0
14	1487.934	3.722	7.3
15	1452.438	3.633	5.9
16	1448.047	3.622	6.2
17	1446.583	3.619	8.9
18	1435.606	3.591	15.7
19	1434.508	3.588	14.3
20	1431.580	3.581	7.0
21	1429.019	3.575	5.7
22	753.879	1.886	55.1

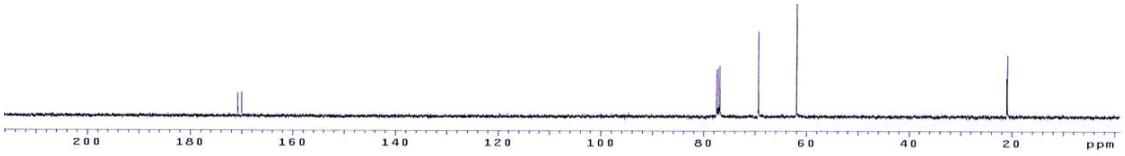




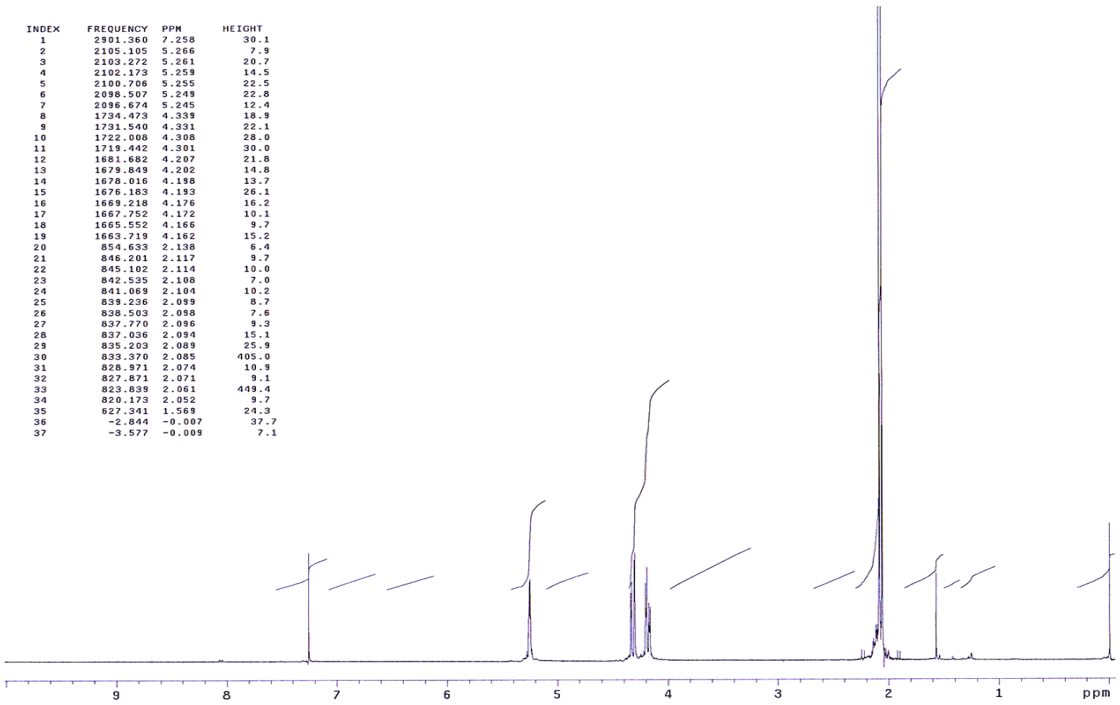
^{1}H-NMR (400 MHz, $CDCl_3$ pmm): δ=5.26-5.24(2H, brs), 4.34-4.20(2H, m), 4.20-4.16(2H, m) 2.085(6H, s), 2.061(6H, s)

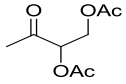
^{13}C-NMR (100MHz, $CDCI_3$, ppm): δ=170.7, 69.36, 61.97, 20.88

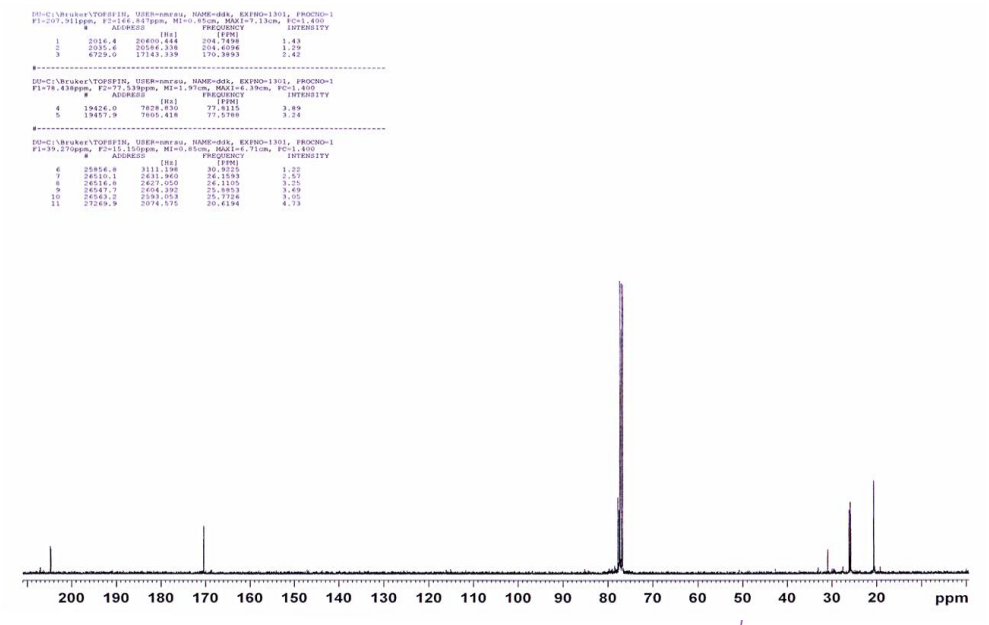
INDEX	FREQUENCY	PPM	HEIGHT
1	17162.892	170.741	7.7
2	17088.885	170.005	8.0
3	7794.473	77.541	15.7
4	7762.428	77.223	16.2
5	7730.384	76.904	17.0
6	6972.762	69.367	28.5
7	6229.636	61.974	37.6
8	2112.689	21.018	12.9
9	2099.719	20.889	20.6

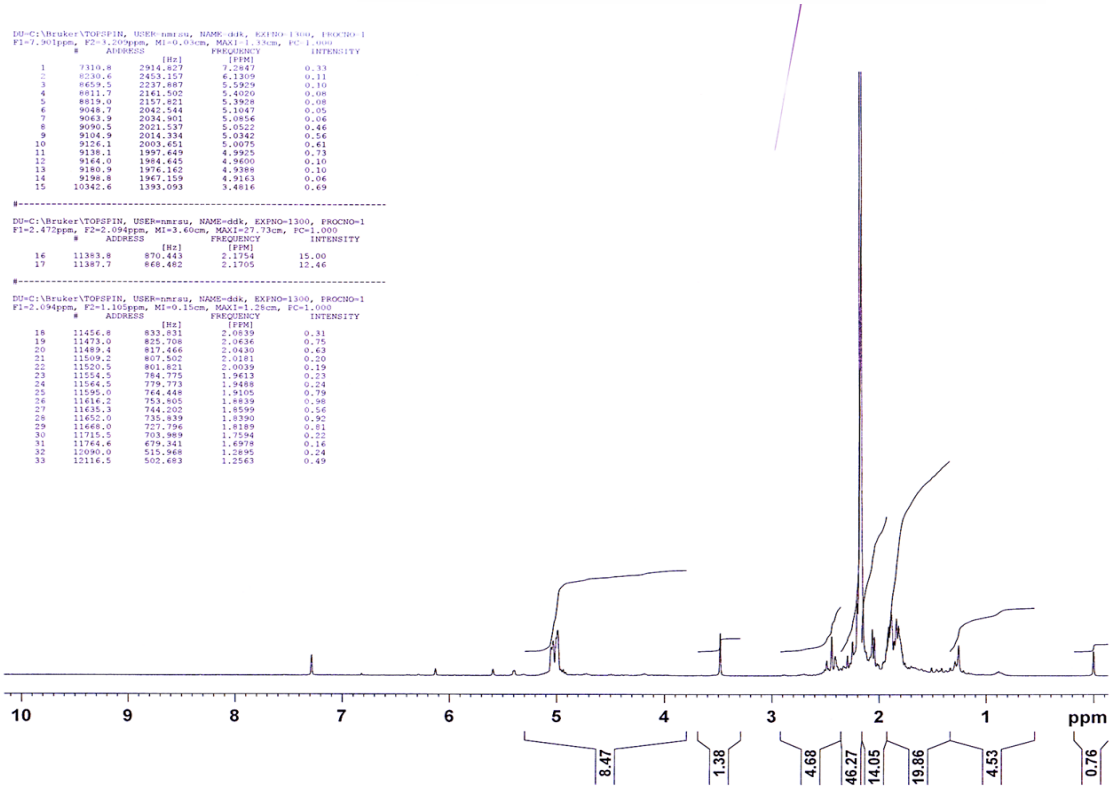


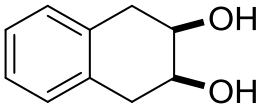
INDEX	FREQUENCY	PPM	HEIGHT
1	2901.360	7.258	30.1
2	2105.105	5.266	7.9
3	2103.272	5.261	20.7
4	2102.173	5.259	14.5
5	2100.706	5.255	22.5
6	2098.507	5.249	22.8
7	2096.674	5.245	12.4
8	1734.473	4.339	18.9
9	1731.540	4.331	22.1
10	1722.008	4.308	28.0
11	1719.442	4.301	30.0
12	1681.682	4.207	21.8
13	1679.849	4.202	14.8
14	1678.016	4.198	13.7
15	1676.183	4.193	26.1
16	1669.218	4.176	16.2
17	1667.752	4.172	10.1
18	1665.552	4.166	9.7
19	1663.719	4.162	15.2
20	854.633	2.138	6.4
21	846.201	2.117	9.7
22	845.102	2.114	10.0
23	842.535	2.108	7.0
24	841.069	2.104	10.2
25	839.236	2.099	8.7
26	838.503	2.098	7.6
27	837.770	2.096	9.3
28	837.036	2.094	15.1
29	835.203	2.089	25.9
30	833.370	2.085	405.0
31	828.971	2.074	10.9
32	827.871	2.071	9.1
33	823.839	2.061	449.4
34	820.173	2.052	9.7
35	627.341	1.569	24.3
36	-2.844	-0.007	37.7
37	-3.577	-0.009	7.1



	**^{1}H-NMR (400 MHz, $CDCl_3$, ppm):** δ=4.90-4.99(3H, m), 2.13 (3H, s), 2.1(3H, s), 1.98-1,71(3H, m),**^{13}C-NMR (100MHz, $CDCI_3$, ppm):** δ=204.95, 170.59(2), 77.8, 48.18, 26.8, 23.6, 20.82.



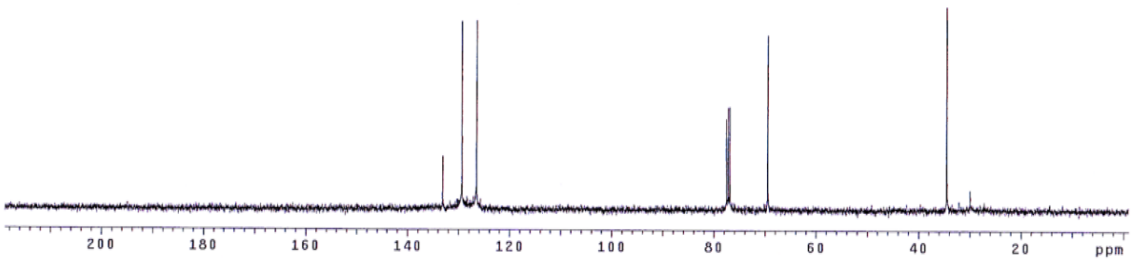




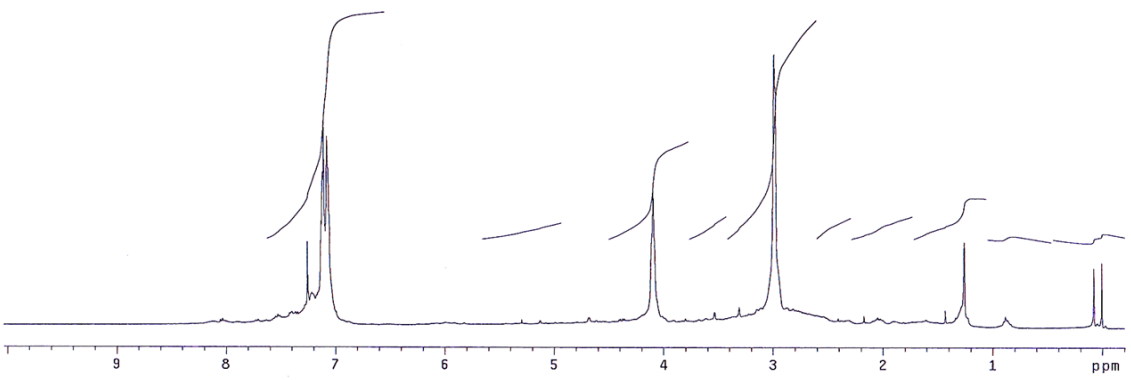
^{1}H-NMR (400 MHz, $CDCl_3$ ppm): δ=7.1-7.08 (5H, m), 4.1 (2H, br s)

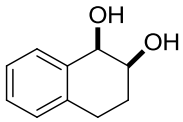
^{13}C-NMR (100MHz, $CDCl_3$ ppm): δ=133.1, 129.4, 126.5, 69.5, 34.5

INDEX	FREQUENCY	PPM	HEIGHT
1	13381.649	133.124	12.5
2	13002.456	129.352	44.1
3	12715.582	126.498	44.3
4	7798.288	77.579	21.5
5	7766.243	77.261	24.3
6	7734.199	76.942	24.3
7	6986.495	69.503	41.0
8	3470.000	34.520	47.8



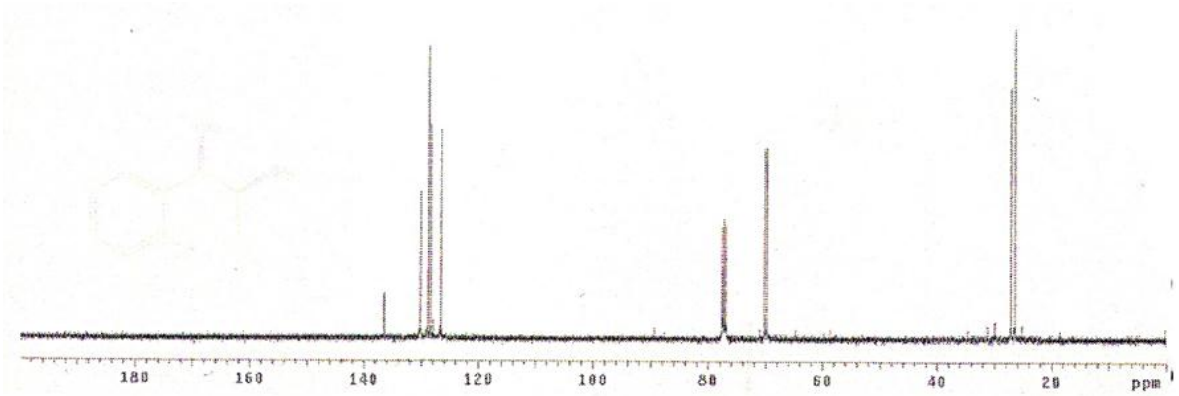
INDEX	FREQUENCY	PPM	HEIGHT
1	2901.869	7.259	19.4
2	2852.102	7.135	24.1
3	2846.979	7.122	35.3
4	2843.320	7.113	45.3
5	2831.244	7.082	43.2
6	2827.219	7.072	36.2
7	1637.948	4.097	31.1
8	1196.638	2.993	62.1
9	1192.612	2.983	54.6
10	503.201	1.259	19.4
11	30.787	0.077	13.4
12	2.244	0.006	14.7

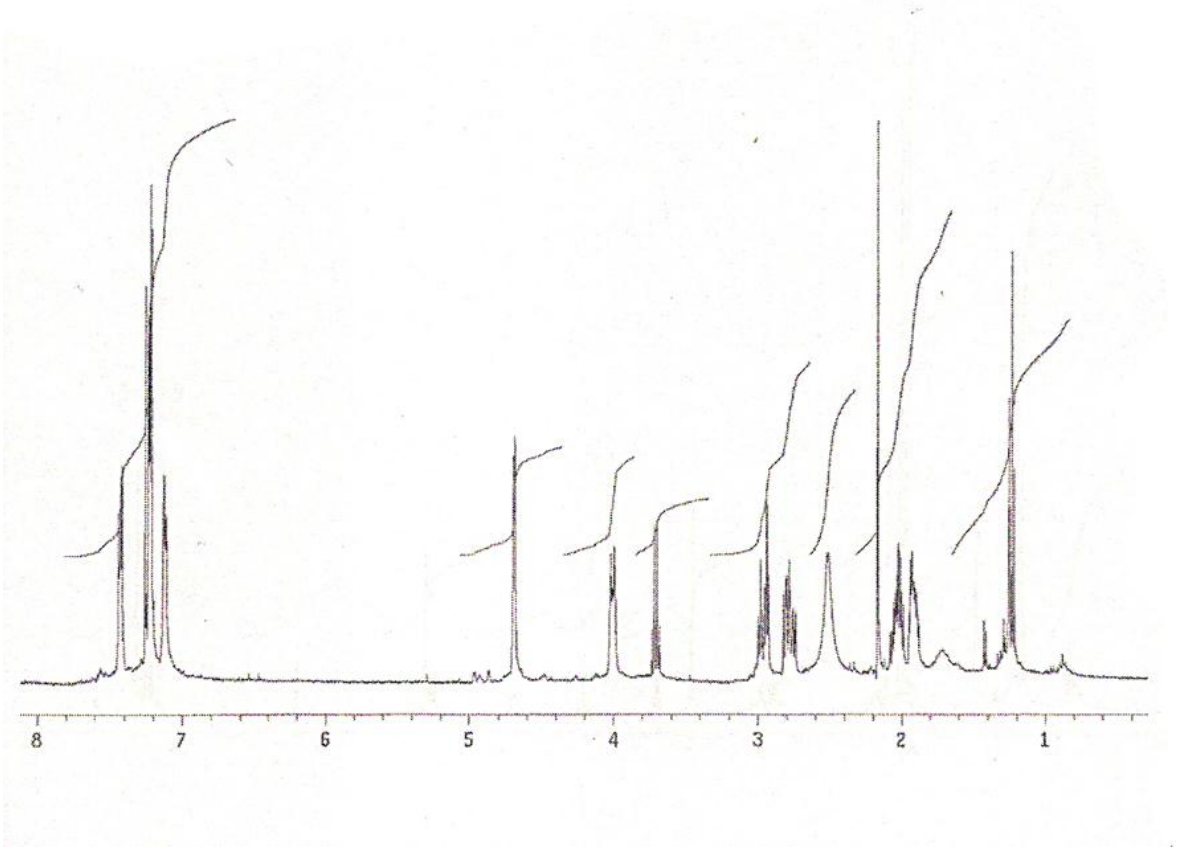


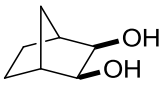


^{1}H-NMR (400MHz, $CDCI_3$, ppm): δ= 7.44-7.11(4Hm), 4.7(1H, d, *J=3.3* Hz), 4.0(1H,dt, *J*=9.9, 3.3Hz), 2.96-2.92(1H dt, *J*=17.2, 5.5Hz), 2.82-2.74(1H, ddd, *J*=17.2, 9.9, 5.5 Hz), 2.5(2H, (2OH) br s), 2.08-1.98(1H, m), 1.95-1.88(1H, m)

^{13}C-NMR (100MHz, $CDCl_3$, ppm): δ=136.6, 136.4, 130.1, 128.8, 128.4, 126.7, 70.2, 69.8, 27.2, 26.5

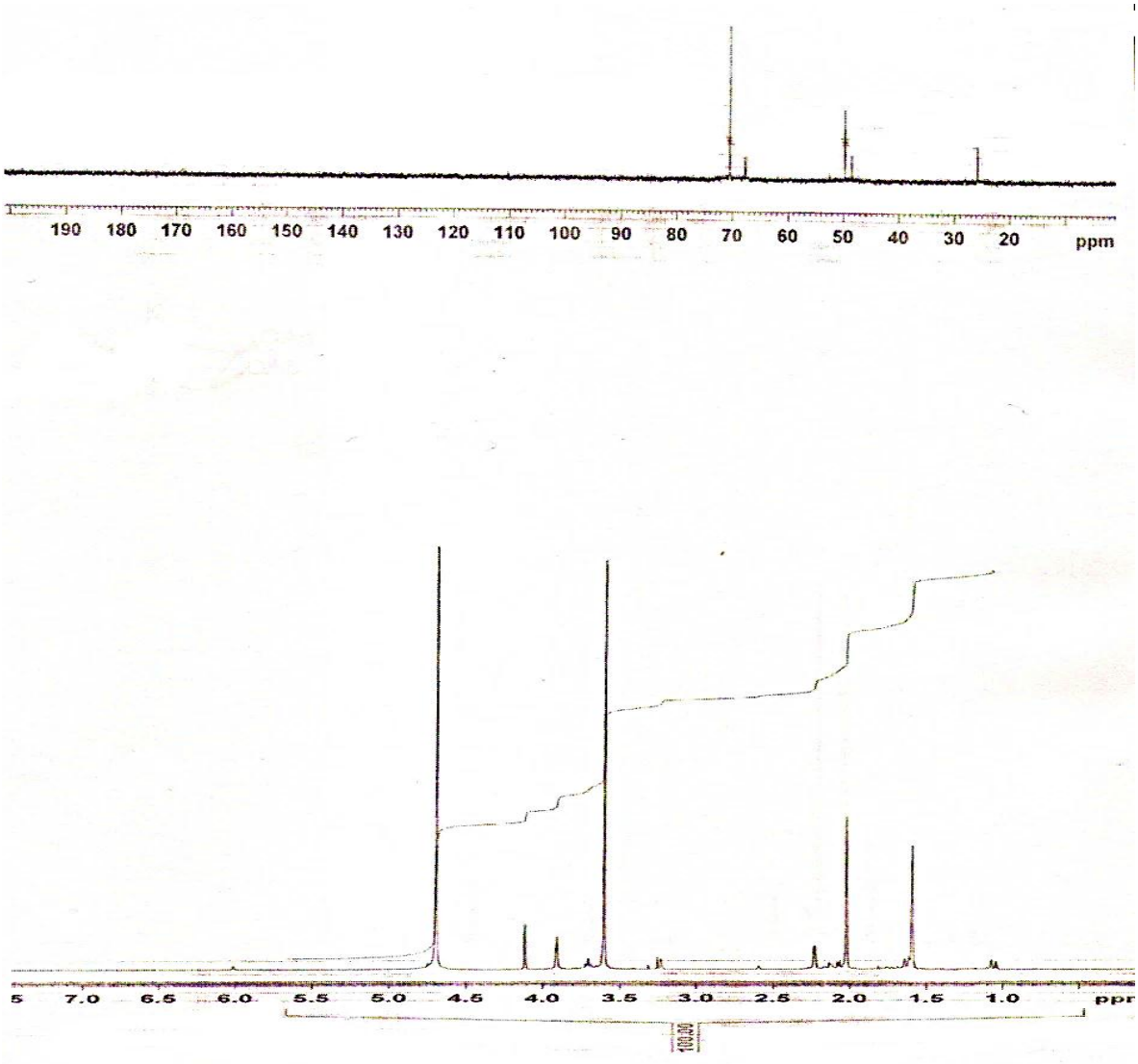


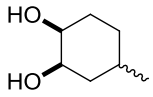




^{1}H-NMR (400MHZ, $CDCl_3$, ppm): δ= 3.7(2H, brs), 3.1(2H, (OH) brs), 2.1(2H, brs), 1.8(1H, dt *J*= 1.4, 10.4 Hz), 1.4(1H, m), 1.1(2H, m), 1.0(2H, m)

^{13}C-NMR (100 MHz, $CDCI_3$, ppm): δ=75.0, 43.2, 31.8, 24.7



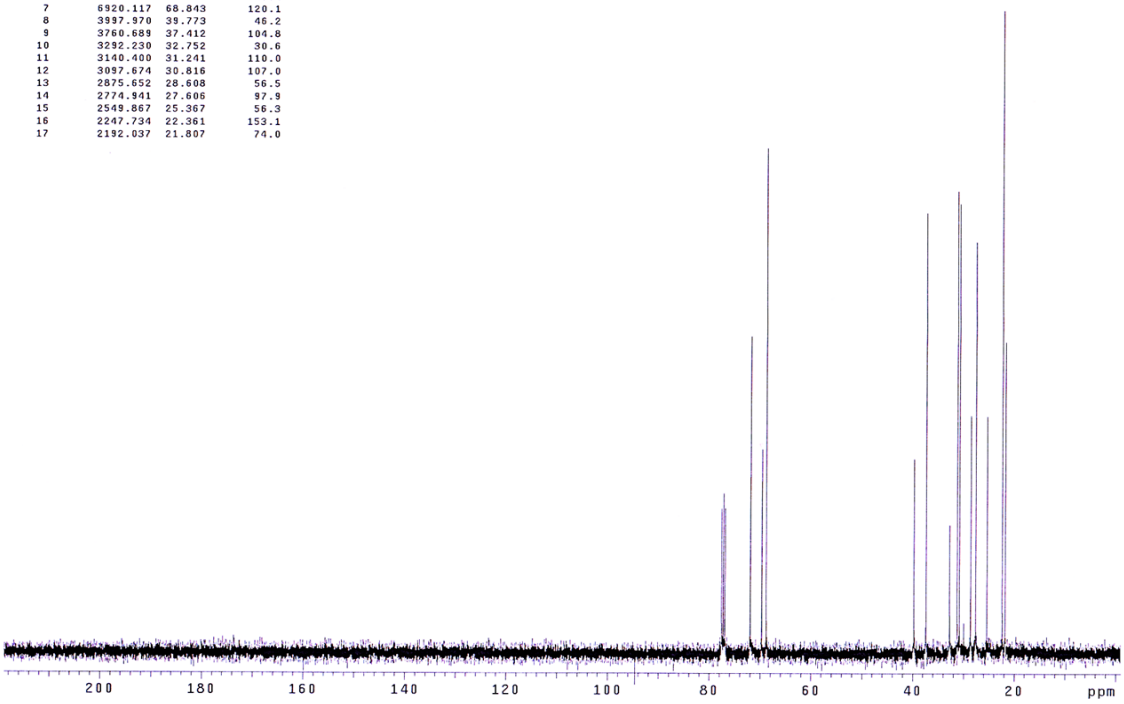


Anti/syn isomers

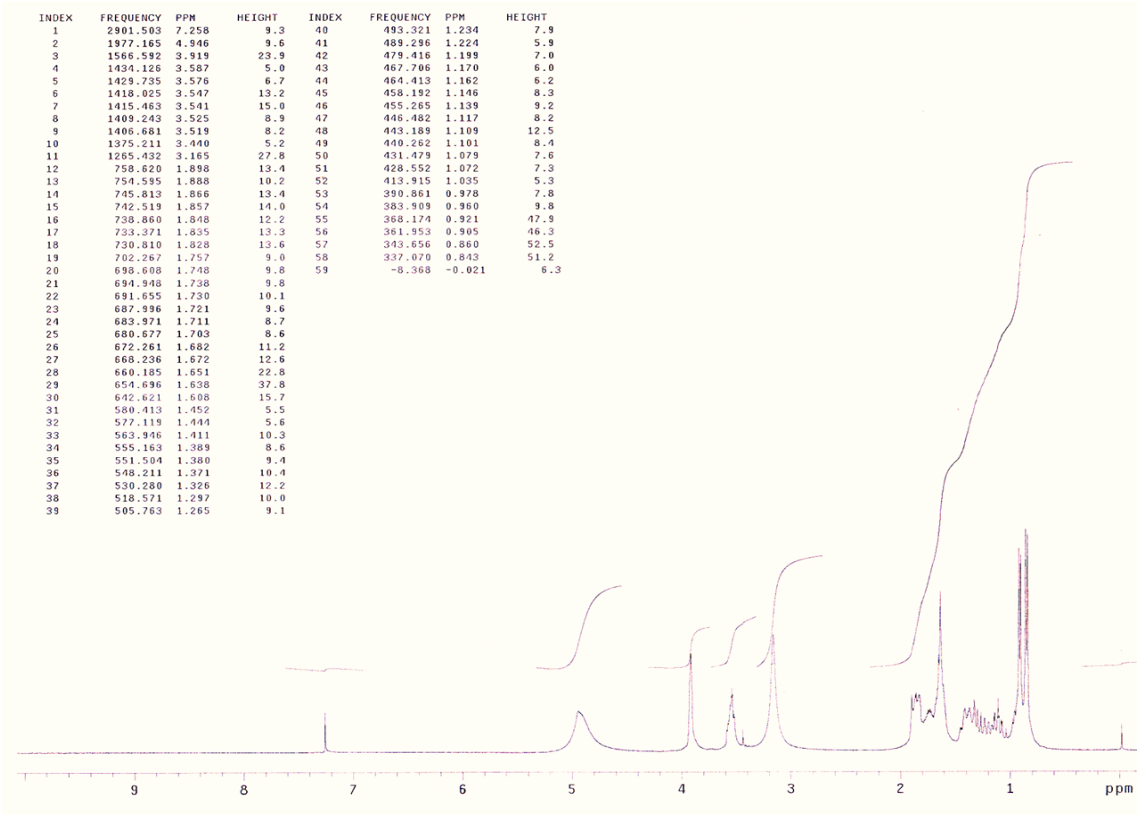
^{1}H-NMR (400MHz, $CDCl_3$, ppm): δ= 4.94(bs, 2H), 3.9(m, 1H), 3.5(m, 1H), 3,1(s, 2H), 1.5, (m,14H), 0,9(3H, dt, *J*=6.25), 0.8(3H, dt, *J*=6.58)

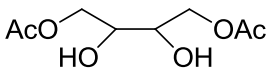
^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ= 76.9, 71.9, 31.2, 30.7, 28.5, 27.6, 20.2

INDEX	FREQUENCY	PPM	HEIGHT
1	7798.288	77.579	34.4
2	7766.243	77.261	38.0
3	7734.199	76.942	34.5
4	7233.695	71.963	75.3
5	7232.169	71.947	72.0
6	7008.621	69.724	48.4
7	6920.117	68.843	120.1
8	3997.970	39.773	46.2
9	3760.689	37.412	104.8
10	3292.230	32.752	30.6
11	3140.400	31.241	110.0
12	3097.674	30.816	107.0
13	2875.652	28.608	56.5
14	2774.941	27.606	97.9
15	2549.867	25.367	56.3
16	2247.734	22.361	153.1
17	2192.037	21.807	74.0



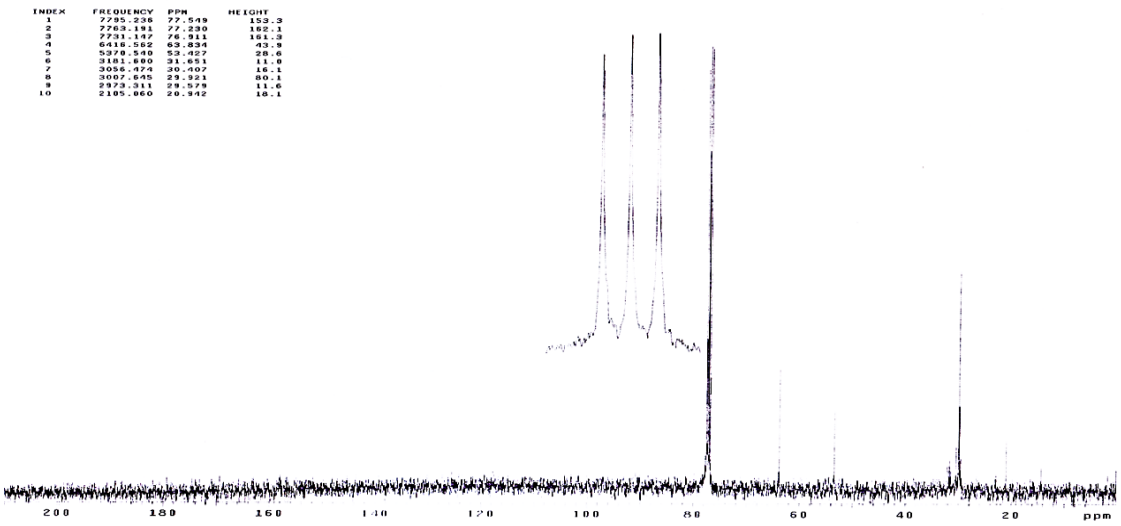
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2901.503	7.258	9.3	40	493.321	1.234	7.9
2	1977.165	4.946	9.6	41	489.296	1.224	5.9
3	1566.592	3.919	23.9	42	479.416	1.199	7.0
4	1434.126	3.587	5.0	43	467.706	1.170	6.0
5	1429.735	3.576	6.7	44	464.413	1.162	6.2
6	1418.025	3.547	13.2	45	458.192	1.146	8.3
7	1415.463	3.541	15.0	46	455.265	1.139	9.2
8	1409.243	3.525	8.9	47	446.482	1.117	8.2
9	1406.681	3.519	8.2	48	443.189	1.109	12.5
10	1375.211	3.440	5.2	49	440.262	1.101	8.4
11	1265.432	3.165	27.8	50	431.479	1.079	7.6
12	758.620	1.898	13.4	51	428.552	1.072	7.3
13	754.595	1.888	10.2	52	413.915	1.035	5.3
14	745.813	1.866	13.4	53	390.861	0.978	7.8
15	742.519	1.857	14.0	54	383.909	0.960	9.8
16	738.860	1.848	12.2	55	368.174	0.921	47.9
17	733.371	1.835	13.3	56	361.953	0.905	46.3
18	730.810	1.828	13.6	57	343.656	0.860	52.5
19	702.267	1.757	9.0	58	337.070	0.843	51.2
20	698.608	1.748	9.8	59	-8.368	-0.021	8.3
21	694.948	1.738	9.8				
22	691.655	1.730	10.1				
23	687.996	1.721	9.6				
24	683.971	1.711	8.7				
25	680.677	1.703	8.6				
26	672.261	1.682	11.2				
27	668.236	1.672	12.6				
28	660.185	1.651	22.8				
29	654.696	1.638	37.8				
30	642.621	1.608	15.7				
31	580.413	1.452	5.5				
32	577.119	1.444	5.6				
33	563.996	1.411	10.3				
34	555.163	1.389	8.6				
35	551.504	1.380	9.4				
36	548.211	1.371	10.4				
37	530.280	1.326	12.2				
38	518.571	1.297	10.0				
39	505.763	1.265	9.1				





^{1}H-NMR(400 MHz $CDCI_3$,ppm) : δ=4.4(2H dd, *j*= 12, 3Hz), 3.9(2H, dd, *J=5.5, 12Hz),* 3.1(2H, dd, *J*=3, 4.8Hz) 2.1(6H s)
^{13}C-NMR(100MHz $CDCl_3$,ppm) : δ= 170.0, 76.9, 63.83, 20.9

INDEX	FREQUENCY	PPM	HEIGHT
1	7795.236	77.549	153.3
2	7763.191	77.230	162.1
3	7731.147	76.911	161.3
4	6416.582	63.834	43.9
5	5378.548	53.497	28.6
6	3181.600	31.651	11.0
7	3056.474	30.407	16.1
8	3007.645	29.921	80.1
9	2973.311	29.579	11.6
10	2105.060	20.942	18.1



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2901.503	7.258	90.9	40	26.396	0.066	5.8
2	1768.585	4.424	9.0	41	-1.415	-0.004	95.9
3	1765.658	4.417	10.4				
4	1756.144	4.393	11.0				
5	1753.216	4.386	11.2				
6	1602.453	4.009	9.0				
7	1596.964	3.995	9.6				
8	1590.912	3.977	8.7				
9	1584.523	3.964	8.2				
10	1269.824	3.176	8.0				
11	1266.530	3.168	7.7				
12	1265.066	3.165	7.7				
13	1262.139	3.157	8.8				
14	849.737	2.126	12.6				
15	847.541	2.120	6.3				
16	844.614	2.113	14.3				
17	842.784	2.108	9.9				
18	839.125	2.099	135.8				
19	631.277	1.579	9.5				
20	577.851	1.445	15.6				
21	569.069	1.424	7.9				
22	564.312	1.412	21.3				
23	555.529	1.390	6.4				
24	531.378	1.329	36.1				
25	525.889	1.316	26.1				
26	523.328	1.309	17.5				
27	516.009	1.291	34.4				
28	511.984	1.281	56.4				
29	499.908	1.251	162.1				
30	480.118	1.201	9.6				
31	475.391	1.189	9.9				
32	468.438	1.172	6.6				
33	464.047	1.161	5.8				
34	456.728	1.143	5.6				
35	452.703	1.132	5.9				
36	441.725	1.105	12.1				
37	356.464	0.892	10.4				
38	350.243	0.876	22.0				
39	343.290	0.859	12.8