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APPENDICES

Appendix A

Effect of Pt and Pd metal

Table A-1 Effect of Pd and Pt metal for hexane conversion

Catalyst	1%Pd1%Ga/HM	1%Pt1%Ga/HM
Conversion	90.5	93.0
Methane	0.2	0.1
Ethane	0.8	0.2
Propane	2.5	0.1
Olefins	20.0	8.8
iso-Butane	19.4	11.3
Butane	11.0	8.7
Pentane	0.2	0.2
branched-C_6	15.5	68.1
Benzene	0.2	0.9
Toluene	0.1	1.4
mixed-Xylene	0	0.2

Effect of Zn metal

Table A-2 Effect of Zn for hexane conversion

Catalyst	0.5%Pt1%Zn/HM	0.5%Pt2%ZnHM
Conversion	74.0	75.0
Methane	0.4	0.2
Ethane	17.4	6.8
Propane	3.5	0.1
Olefins	40.4	53.4
iso-Butane	0.6	1.4
Butane	0.2	0.2
Pentane	0.2	0.1
branched-C_6	35.6	35.0
Benzene	1.1	1.3
Toluene	0.4	1.4
mixed-Xylene	0.1	0.1

Effect of Bimetallic

Table A-3 Effect of bimetallic for hexane conversion

Catalyst	0.5%Pt1%Ga/HM	0.5%Pt2%Ga/HM	1%Pt1%Ga/HM
Conversion	85.0	89.0	93.0
Methane	0.3	0.3	0.1
Ethane	28.0	35.1	0.2
Propane	1.0	1.0	0.1
Olefins	17.0	41.8	8.8
iso-Butane	5.4	1.3	11.3
Butane	11.0	0.2	8.7
Pentane	0.6	0.2	0.2
branched-C_6	34.4	16.6	68.1
Benzene	0.8	1.9	0.9
Toluene	1.0	1.3	1.4
mixed-Xylene	0.3	0.3	0.2

Effect of loading methods

Table A-4 Effect of loading method for hexane conversion.

Catalysts	**1%Pd/M**	**1.12%Pd-M**	**1%Pt/M**	**0.95%Pt-M**
Conversion	80.0	84.0	87.0	82.0
Methane	0.4	1.0	2.0	0.2
Ethane	18.4	33.0	5.0	7.8
Propane	14.6	23.1	10.0	1.8
Olefins	20.2	7.0	5.0	2.1
iso-Butane	7.8	0.2	16.1	5.5
Butane	1.3	0	12.0	3.3
Pentane	1.3	0	1.0	1.4
branched-C_6	0.8	35.0	47.0	22.0
Benzene	35.8	0.4	0.2	0.2
Toluene	0.2	0.4	0.6	0.4
mixed-Xylene	0.3	0.1	0.1	0.1

Effect of potassium

Table A-5 Effect of potassium for hexane conversion

Catalyst	0.5Pt%1Ga%/KCl-M	0.5Pt%1%Ga/K_2CO_3-M
Conversion	89.0	78.5
Methane	0.8	1.3
Ethane	16.3	20.5
Propane	3.5	3.0
Olefins	20.2	12.6
iso-Butane	8.4	12.4
Butane	12.5	18.6
Pentane	0.3	0.2
branched-C_6	28.5	29.7
Benzene	5.0	0.8
Toluene	3.0	0.9
mixed-Xylene	1.5	0.0

Effect of time on stream

Table A-6 Effect of time on stream for hexane conversion

0.5Pt1GaM	TOS (h^{-1})		
	3	5	8
Conversion	93.0	90.0	89.0
Methane	0.1	0.1	0.1
Ethane	0.2	1.4	1.4
Propane	0.1	0.2	0.1
Olefins	8.8	10.0	10.0
iso-Butane	11.3	8.2	8.0
Butane	8.7	8.7	9.0
Pentane	0.2	2.2	2.2
branched-C_6	68.1	66.6	67.0
Benzene	0.9	1.0	1.0
Toluene	1.4	1.4	1.0
mixed-Xylene	0.2	0.2	0.2

Effect of mixture

Table A-7 Effect of metal oxide mixture for hexane conversion

Mixture	Ga/Al_2O_3	ZrO_2	MgO
Conversion	85.0	88.2	55.0
Methane	8.2	5.5	0.2
Ethane	7.3	6.8	0.3
Propane	8.6	4.2	0.2
Olefins	7.5	12.4	11.2
iso-Butane	22.5	26.5	30.8
Butane	10.1	9.3	1.1
Pentane	6.0	3.4	0.7
branched-C_6	28.6	30.6	40.5
Benzene	0.3	0.4	5.8
Toluene	0.7	0.6	8.2
mixed-Xylene	0.2	0.3	1.0

Table A-8 Effect of MCM-41 mixture for hexane conversion

Mixture	Mixed-MCM-41
Conversion	63.5
Methane	5.5
Ethane	2.0
Propane	2.1
Olefins	6.2
iso-Butane	4.8
Butane	8.4
Pentane	1.7
branched-C_6	65.4
Bezene	2.5
Toluene	1.2
mixed-Xylene	0.2

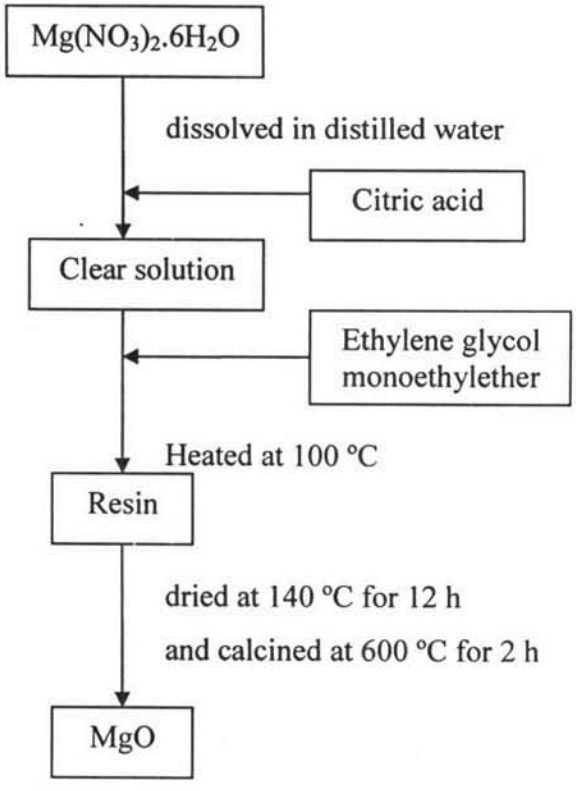
Table A-9 Effect of clay mixture for hexane conversion

Mixture	Bent	K-Bent	HT
Conversion	75.0	50.7	74.9
Methane	2.0	0	2.5
Ethane	2.3	0.6	4.6
Propane	3.4	0.8	1.1
Olefins	11.4	13.2	35.2
iso-Butane	12	13	22.3
Butane	4.8	0.5	1.6
Pentane	2.5	0.6	0.6
branched-C_6	55.6	63.3	28.1
Bezene	2.0	2.5	1.2
Toluene	3.2	3.6	2.2
mixed-Xylene	0.8	1.9	0.6

Appendix B

Preparation of MgO

This oxide was synthesized using modified citrate precursor method. Magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$ (2.04 g, 7.96 mmol), was dissolved in 50 ml distilled water. Then citric acid (5.17 g, 26.91 mmol) was added. After 10 minutes stirring, the solution became clear. Then ethylene glycol monoethyl ether (7.76 g, 86.10 mmol) was added. Then the contents were heat at 100°C to evaporate water. The gel was dried at 200°C to get a brown fluffy. The brown fluffy was grinded and calcined at 600°C 2 h.



MgAl Hydrotalcite

An aqueous mixture (60 ml) of 18.46 g (72 mmol) $Mg(NO_3)_2 . 6H_2O$ and 6.75 g (18 mmol) $Al(NO_3)_3 . 9H_2O$ (molar ratio of Mg/Al = 4) was added slowly to 90 ml of an aqueous solution of 11.52 g (0.11 mol) $(NH_4)_2CO_3$. The pH of the mixture was held at pH

8 by the dropwise addition of NH_4OH. The resulting mixture was heated to 65°C while stirred vigorously for 3 h. Then it was filtered and washed with distilled water until the filtrate was neutral. The precipitate was dried in an oven at 100°C for 18 h and calcined at 450°C for 35 h (in the literature [20], calcination time is 3 h).

The rehydrated hydrotalcite was prepared from the above calcined hydrotalcite by placing it in a desiccator which was saturated with water for 48 h.

Preparation of K-bentonite

Bentonite (5.0g) and 80 ml of 8.0 M of KCl solution were added to the round bottom and heated by using an oil bath at 80°C for 24 h. The solids were separated from solution by filtration and washed several times with deionized water until no chloride observed. The resulting solid was dried at 100°C and then calcined in a muffle furnace at 550°C for 5 h.

Preparation of Ga/Al_2O_3

Gallium nitrate hydrate in aqueous solution was first impregnated onto Al_2O_3 then docomposted into gallium oxide at 550°C in air overnight (gallium oxide content = 18.8%wt).

Appendix C

Calculation of weight hour space velocity

$$\text{WHSV } (ml g^{-1} h^{-1}) = \frac{(\text{volume of hexane})}{(\text{mass of catalyst}) \times (\text{time})}$$

$$= \frac{1 ml}{1 g\ 1 h.}$$

Calculation of chromatogram

Gas chromatography determined liquid products of benzene, toluene and mixed xylene. Liquid products were identified using standard addition method.

The mixed-solution of C_6-C_8 hydrocarbon were prepared and determined by gas chromatography by used *n*-decane as internal standard as following in Table 1-A.

Table 1-C. Standard solution for determined correction factor.

	Volume (ml)	Density ($g.ml^{-1}$)	Mw	Mol
n-Hexane	0.08	0.659	86.18	$6.12X10^{-4}$
Benzene	0.10	0.879	78.12	$1.12X10^{-3}$
Toluene	0.10	0.861	92.14	$9.40X10^{-9}$
Mixed-Xylene	0.10	0.865	106.17	$8.15X10^{-9}$
n-Decane	$0.5X10^{-2}$	0.730	142.29	$2.56X10^{-5}$

H: peak area of *n*-hexane prepared = 233442

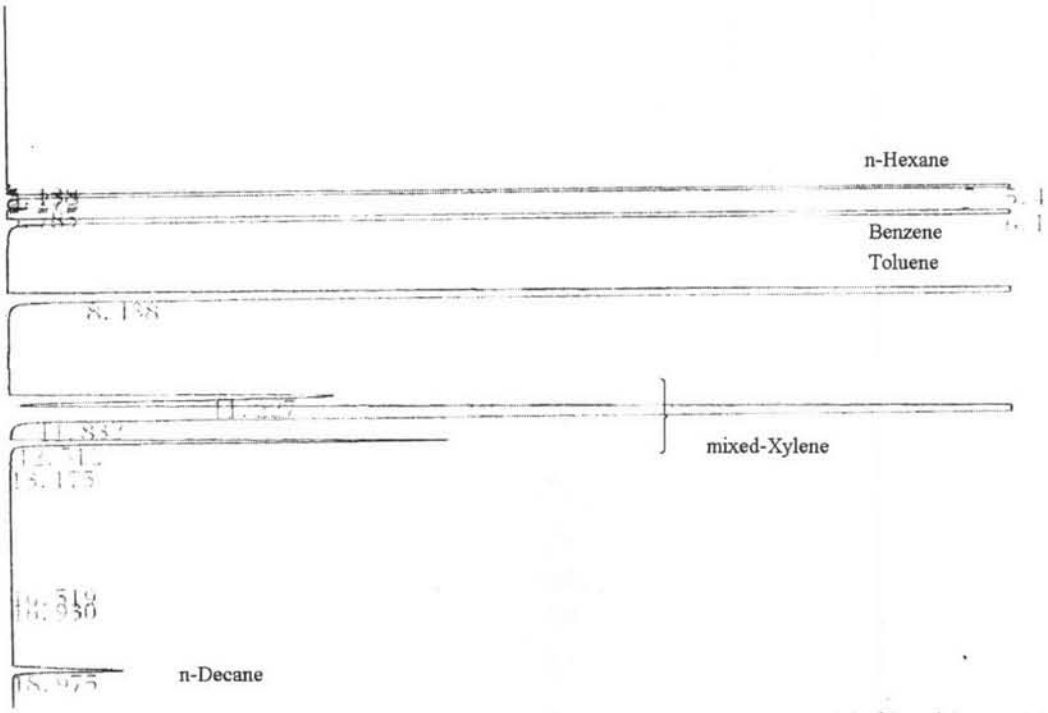
B: peak area of benzene prepared = 352893

T: peak area of toluene prepared = 347184

X: peak area of mixed-xylene prepared = 342815

D: peak area of internal standard prepared = 24369

The chromatogram was shown in Figure 1-C.



CALCULATION REPORT **

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
3	5.44	316824	119289			18.6958	
5	6.188	526519	117414			31.0699	
6	8.438	369393	44352			21.7979	
7	11.227	54010	5138			3.1872	
8	11.832	374596	25867			22.1049	
9	12.512	40204	6964			2.3724	
13	18.975	13079	1719			0.7718	

Figure 1-C Chromatogram of liquid mixture for correction factor calculation.

The calculation of the correction factor can be described as follows:

Correction factor of *n*-hexane:

The amount of *n*-hexane from chromatogram

$$= \frac{(\text{mol of internal standard}) \times H}{D}$$

$$= \frac{2.56 \text{ X } 10^{-5} \times 316824}{12373}$$

$$= 65.68 \times 10^{-5} \text{ mol}$$

Thus, the correction factor of *n*-hexane can be calculated as:

$$= \frac{\text{amount of n - hexane prepared}}{\text{amount of n - hexane from chromatogram}}$$

$$= \frac{6.12 \times 10^{-4}}{65.68 \times 10^{-5}}$$

$$= 0.93$$

Correction factor of benzene:

The amount of benzene from chromatogram

$$= \frac{(\text{mol of internal standard}) \times B}{D}$$

$$= \frac{2.56 \text{ X } 10^{-5} \times 526519}{12373}$$

$$= 109.2 \times 10^{-5} \text{ mol}$$

Thus, the correction factor of benzene can be calculated as:

$$= \frac{\text{amount of benzene prepared}}{\text{amount of benzene from chromatogram}}$$

$$= \frac{1.12 \times 10^{-3}}{109.2 \times 10^{-5}}$$

$$= 1.03$$

Correction factor of toluene:

The amount of toluene from chromatogram

$$= \frac{(\text{mol of internal standard}) \times T}{D}$$

$$= \frac{2.56 \text{ X } 10^{-5} \times 369393}{12373}$$

$$= 76.58 \times 10^{-5} \text{ mol}$$

Thus, the correction factor of toluene can be calculated as:

$$= \frac{\text{amount of toluene prepared}}{\text{amount of toluene from chromatogram}}$$

$$= \frac{9.40 \times 10^{-4}}{76.6 \times 10^{-5}} = 1.23$$

Correction factor of mixed-xylene:

The amount of toluene from chromatogram

$$= \frac{(\text{mol of internal standard}) \times X}{D}$$

$$= \frac{2.56 \text{ X } 10^{-5} \times 468810}{12373}$$

$$= 97.19 \times 10^{-5} \text{ mol}$$

Thus, the correction factor of toluene can be calculated as:

$$= \frac{\text{amount of mixed - xylene prepared}}{\text{amount of mixed - xylene from chromatogram}}$$

$$= \frac{8.15 \times 10^{-4}}{97.2 \times 10^{-5}}$$

$$= 0.83$$

The correction factors of chemicals are listed as follows:

n-hexane = 0.93

benzene = 1.03

toluene = 1.22

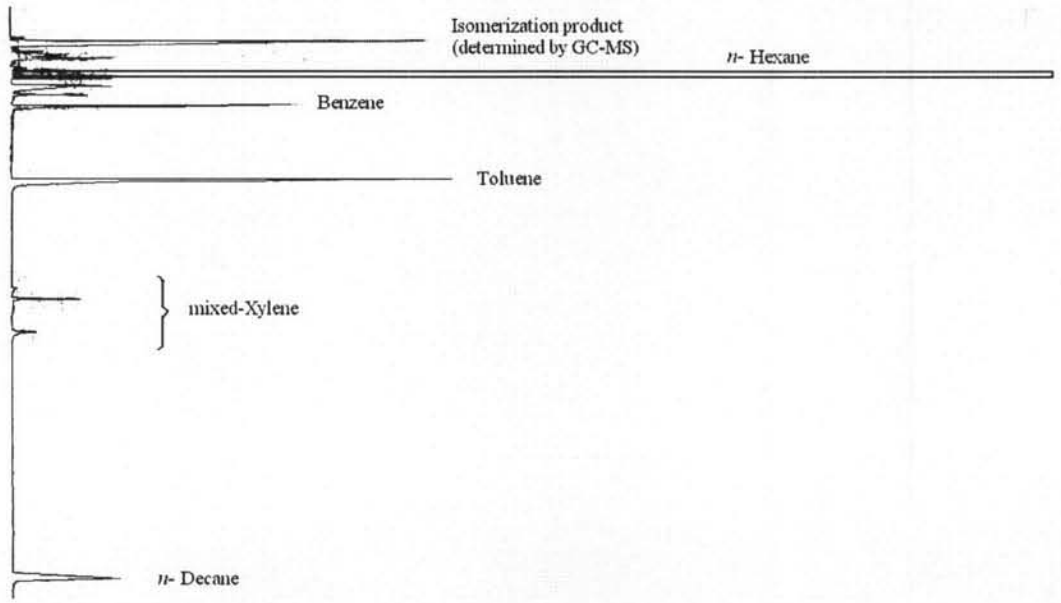
mixed-xylene = 0.83

Sample calculation of products distribution for 1%Pt1%Ga/HM catalyst

Mass of liquid product from reaction = 0.7877 g

$Hexane_{in}$ = 3 ml × (0.659×10^{-3} g.ml^{-1}) = 1.977 g

Mol of *n*-decane (internal standard) = 5.65×10^{-5} mol



CALCULATION REPOORT **

PKNO	TIME	AREA	HEIGH	MK	IDNO	CONC	NAME
1	4.322	30420	6398			1.4721	
2	5.440	1546790	375297			94.8776	
3	6.188	30909	4655			0.5587	
4	8.409	40335	6678			1.0088	
5	11.223	50	112			0.0362	
6	11.418	200	1059			0.2578	
7	12.496	120	391			0.0778	
8	18.971	12373	1714			0.5988	

Figure 2-C Chromatogram of liquid products mixture by 1%Pt/HM catalyst.

Product distribution

$$\text{Hexane} = \frac{2.565\times10^{-5}\times1546790}{12373}\times0.93 = 2.98\times10^{-3}\ \text{mol}$$

$$= 2.98\times10^{-3}\ \text{mol}\times86.18\text{g.mol}^{-1} = 0.2570\ \text{g}$$

$$\text{Benzene} = \frac{2.565\times10^{-5}\times30909}{12373}\times1.03 = 0.66\times10^{-4}\ \text{mol}$$

$$= 0.66\times10^{-4}\ \text{mol}\times78.12\text{g.mol}^{-1} = 5.16\times10^{-3}\ \text{g}$$

$$\text{Toluene} = \frac{2.565\times10^{-5}\times40335}{12373}\times1.22 = 1.03\times10^{-4}\ \text{mol}$$

$$= 1.03\times10^{-4}\ \text{mol}\times92.14\text{g.mol}^{-1} = 9.46\times10^{-3}\ \text{g}$$

$$\text{mixed-Xylene} = \frac{2.565\times10^{-5}\times470}{12373}\times0.83 = 0.81\times10^{-6}\ \text{mol}$$

$$= 0.81\times10^{-6}\ \text{mol}\times106.17\text{g.mol}^{-1} = 8.60\times10^{-4}\ \text{g}$$

$$\%\text{Conversion} = \frac{Hexane_{in} - Hexane_{out}}{Hexane_{in}}\times100$$

$$= \frac{1.977-0.2570}{1.977}\times100$$

$$= 87.0\%$$

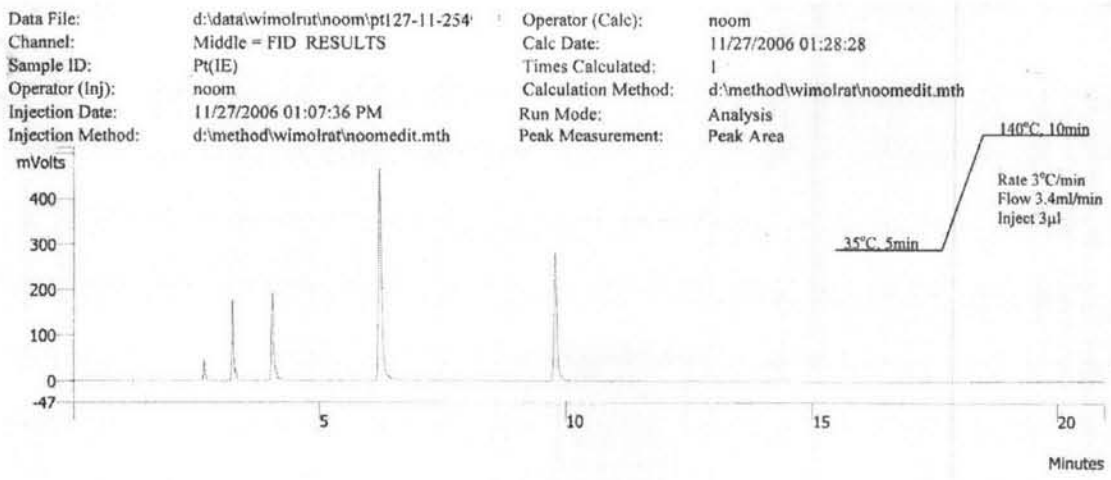
$$\text{So, products were converted} = \frac{1.977\times87.0}{100} = 1.720\ \text{g}$$

$$\%\text{Benzene} = \frac{5.16\times10^{-3}}{1.720}\times100 = 0.30\%$$

$$\%\text{Toluene} = \frac{9.46\times10^{-3}}{1.720}\times100 = 0.55\%$$

$$\%\text{Xylene} = \frac{8.6\times10^{-4}}{1.720}\times100 = 0.05\%$$

All data above were used to calculation gas products by gas chromatography in figure 3-C.



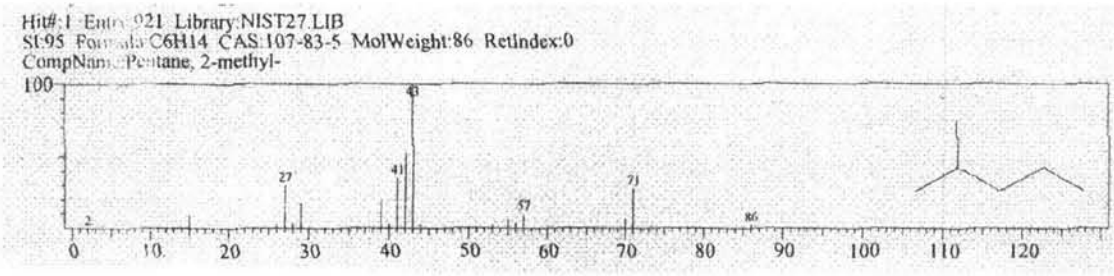
Peak No	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)	Status Codes	Conc %
1	Methane	2.6914	2.671	0.000	139442	0.00	BB	2.3		8.0
2	Ethene	9.9471	3.247	0.000	515361	0.00	BB	2.2		9.0
3	Ethane	13.0087	4.050	0.000	673984	0.00	BB	2.7		5.0
4	iso-Butane	40.7670	6.200	0.000	2112151	0.00	BB	3.7		18.1
5	Butane	24.2446	9.774	0.000	1256119	0.00	BB	3.8		12.0

Figure 3-C Chromatogram of gas products mixture by 1%Pt/HM catalyst.

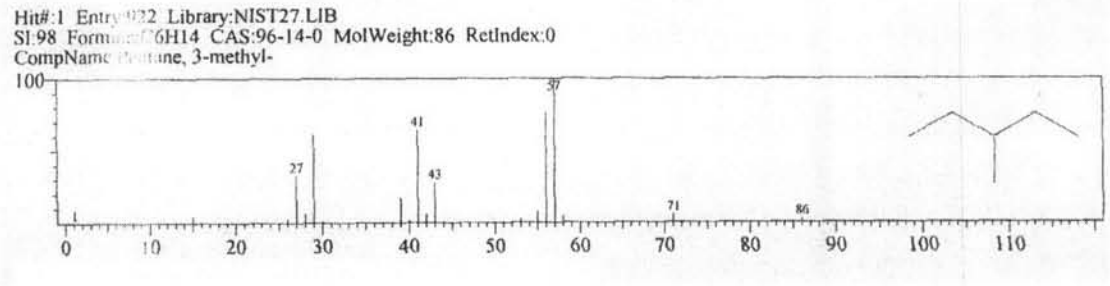
Isomerization products = 100-8.0-9.0-5.0-18.1-12.0-0.30-0.55-0.05

= 47.0%

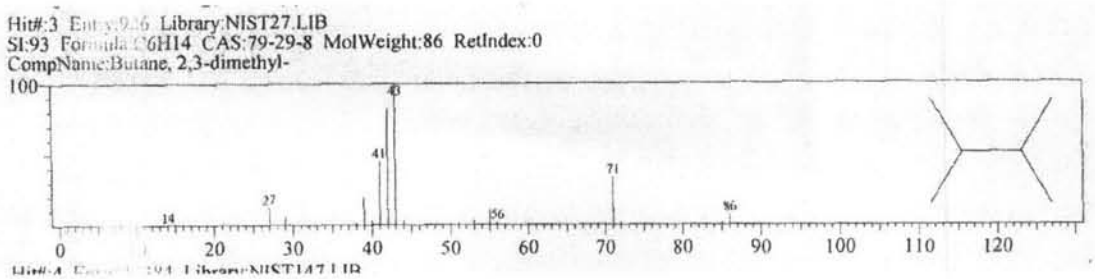
Results from GC-MS of isomerization products by 1%Pt/HM catalyst



Ficgure 4-C. 2-methylpentane



Ficgure 5-C. 3-methylpentane



Ficgure 6-C. 2,3-dimethylbutane