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APPENDICES

Appendix A The glycerol conversion, diglycerol selectivity and diglycerol yield of the studied catalysts

In this study, the catalytic activities of some heterogeneous catalysts were compared with homogeneous catalyst in terms of total glycerol conversion, diglycerol selectivity, and diglycerol yield. The representative of homogeneous catalysts was Na_2CO_3, while the heterogeneous catalysts were MgO, CaO, BaO SrO and ZrO_2. The reaction temperature was fixed at 240°C and 2.0 wt% of catalyst.

Table A1 The glycerol conversion, diglycerol selectivity and diglycerol yield when studied the effect of catalyst types

Time (hrs)	No Catalyst		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
1.0	0.08	0.00	0.00
2.0	0.17	0.00	0.00
3.0	1.62	0.00	0.00
4.0	2.41	0.00	0.00
5.0	3.72	0.00	0.00

Time (hrs)	Na_2CO_3		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
1.0	42.14	36.20	15.26
2.0	55.59	35.16	19.55
3.0	84.77	21.88	18.55
4.0	85.08	20.13	17.13
5.0	89.83	20.29	18.22

Time (hrs)	MgO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
1.0	22.77	2.88	0.66
2.0	31.49	13.36	4.21
3.0	35.73	18.39	6.57
4.0	47.24	22.04	10.41
5.0	61.12	21.91	13.39

Time (hrs)	CaO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
0.3	10.72	12.72	1.36
0.5	36.73	36.64	13.46
1.0	54.90	69.34	38.07
2.0	67.97	70.34	47.81
3.0	64.47	67.27	43.37
4.0	66.13	63.37	41.91
5.0	71.97	53.31	38.37

Time (hrs)	SrO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
1.0	45.45	70.57	32.07
2.0	57.53	59.02	33.96
3.0	67.63	47.23	31.94
4.0	80.81	25.97	20.98
5.0	82.93	25.04	20.76

Time (hrs)	BaO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
0.3	7.44	64.47	4.80
0.5	13.55	88.96	12.06
1.0	30.59	94.27	28.84
2.0	45.95	81.18	37.30
3.0	52.81	73.06	38.58
4.0	54.94	56.63	31.11
5.0	81.87	21.95	17.97

Time (hrs)	ZrO2		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
6.0	9.72	48.32	4.70
8.0	28.24	58.93	16.64
10.0	44.83	55.43	24.85
12.0	62.25	41.30	25.71

Table A2 The glycerol conversion, diglycerol selectivity and diglycerol yield when studied the effect of catalyst concentration

Amount of catalyst (wt %)	CaO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
0.5	32.59	52.73	17.18
1.0	55.13	64.57	35.60
2.0	61.53	55.62	34.22
4.0	64.10	43.27	27.74
6.0	64.79	41.05	26.60

Amount of catalyst (wt %)	BaO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
0.5	23.35	77.03	17.99
1.0	30.59	94.27	28.84
2.0	35.32	78.93	27.88
4.0	40.45	61.09	24.71
6.0	44.54	52.32	23.31

Table A3 The glycerol conversion, diglycerol selectivity and diglycerol yield when studied the effect of reaction temperature

Reaction Temperature (°C)	CaO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
220.0	25.67	31.74	8.15
230.0	34.34	45.23	15.53
240.0	55.06	65.25	35.92
250.0	62.03	51.11	31.70
260.0	67.79	31.53	21.37

Reaction Temperature (°C)	BaO		
	% Glycerol Conversion	% Diglycerol Selectivity	% Diglycerol Yield
220.0	16.19	35.85	5.81
230.0	23.11	57.71	13.33
240.0	32.98	81.31	26.81
250.0	43.46	58.17	25.28
260.0	61.42	29.71	18.25

Appendix B The BET surface area of the studied catalysts

Table B1 The BET surface area of MgO

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.19

Sample ID	MgO				
Description					
Comments					
Sample Weight	0.1606 g				
Adsorbate	NITROGEN	Outgas Temp	250.0 °C	Operator	Dee
Cross-Sec Area	16.2 Å²/molecule	Outgas Time	14.0 hrs	Analysis Time	53.6 min
NonIdeality	6.580E-05	P/Po Toler	2	End of Run	04/02/2008 09
Molecular Wt	28.0134 g/mol	Equil Time	3	File Name	AS840201.RAW
Station #	1	Bath Temp.	77.35		

MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
5.6534e-02	10.7694	4.452E+00
7.6158e-02	11.3757	5.798E+00
1.0155e-01	11.9962	7.539E+00
1.4848e-01	12.9890	1.074E+01
1.9956e-01	14.0133	1.424E+01
2.4981e-01	15.0276	1.773E+01
3.0027e-01	16.0941	2.133E+01

Area = 5.005E+01 m²/g

Slope = 6.906E+01

Y - Intercept = 5.181E-01

Correlation Coefficient = 0.999970

C = 1.343E+02

Table B2 The BET surface area of CaO

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.19

Sample ID	CaO-1						
Description							
Comments							
Sample Weight	0.5413 g						
Adsorbate	NITROGEN	Outgas Temp	250.0 °C	Operator	Dee
Cross-Sec Area	16.2 Å²/molecule	Outgas Time	14.5 hrs	Analysis Time	45.5 min
NonIdeality	6.580E-05	P/Po Toler	2	End of Run	04/03/2008 10
Molecular Wt	28.0134 g/mol	Equil Time	3	File Name	AS840302.RAW
Station #	1	Bath Temp.	77.35		

MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
5.5154e-02	0.4796	9.739E+01
8.0405e-02	0.5422	1.290E+02
1.0619e-01	0.5927	1.604E+02
1.5528e-01	0.6763	2.175E+02
2.0591e-01	0.7610	2.726E+02
2.5648e-01	0.8442	3.269E+02
3.0655e-01	0.9311	3.799E+02

Area = 3.002E+00 m²/g

Slope = 1.120E+03

Y - Intercept = 3.965E+01

Correlation Coefficient = 0.999618

C = 2.926E+01

Table B3 The BET surface area of SrO

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.19

Sample ID	SrO				
Description					
Comments					
Sample Weight	0.4777 g				
Adsorbate	NITROGEN	Outgas Temp	250.0 °C	Operator	Dee
Cross-Sec Area	16.2 Å²/molecule	Outgas Time	7.0 hrs	Analysis Time	62.3 min
NonIdeality	6.580E-05	P/Po Toler	2	End of Run	04/02/2008 18
Molecular Wt	28.0134 g/mol	Equil Time	3	File Name	AS840204.RAW
Station #	1	Bath Temp.	77.35		

MULTIPOINT BET

P/Po	Volume (cc/g) STP	1/(W((Po/P)-1))
5.1488e-02	0.9210	4.716E+01
7.9008e-02	1.0244	6.701E+01
1.0574e-01	1.0962	8.631E+01
1.5476e-01	1.2050	1.216E+02
2.0519e-01	1.3106	1.576E+02
2.5604e-01	1.4178	1.942E+02
3.0598e-01	1.5352	2.298E+02

Area = 4.783E+00 m²/g

Slope = 7.177E+02

Y - Intercept = 1.034E+01

Correlation Coefficient = 0.999998

C = 7.040E+01

Table B4 The BET surface area of BaO

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.19

Sample ID	BaO				
Description					
Comments					
Sample Weight	1.5767 g				
Adsorbate	NITROGEN	Outgas Temp	250.0 °C	Operator	Dee
Cross-Sec Area	16.2 Å²/molecule	Outgas Time	17.0 hrs	Analysis Time	45.4 min
NonIdeality	6.580E-05	P/Po Toler	2	End of Run	04/03/2008 09
Molecular Wt	28.0134 g/mol	Equil Time	3	File Name	AS840301.RAW
Station #	1	Bath Temp.	77.35		

MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
5.1493e-02	0.0403	1.078E+03
8.1291e-02	0.0491	1.441E+03
1.0707e-01	0.0560	1.714E+03
1.5648e-01	0.0686	2.163E+03
2.0734e-01	0.0814	2.571E+03
2.5712e-01	0.0935	2.961E+03
3.0778e-01	0.1078	3.301E+03

Area = 3.738E-01 m²/g

Slope = 8.573E+03

Y - Intercept = 7.443E+02

Correlation Coefficient = 0.996306

C = 1.252E+01

Table B5 The BET surface area of ZrO_2

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.19

Sample ID	ZrO2				
Description					
Comments					
Sample Weight	1.1072 g				
Adsorbate	NITROGEN	Outgas Temp	250.0 °C	Operator	Dee
Cross-Sec Area	16.2 Å²/molecule	Outgas Time	12.5 hrs	Analysis Time	51.8 min
NonIdeality	6.580E-05	P/Po Toler	2	End of Run	04/02/2008 11
Molecular Wt	28.0134 g/mol	Equil Time	3	File Name	AS840202.RAW
Station #	1	Bath Temp.	77.35		

MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
5.3652e-02	1.9066	2.379E+01
7.5502e-02	2.0083	3.254E+01
1.0126e-01	2.1085	4.276E+01
1.4880e-01	2.2592	6.191E+01
2.0078e-01	2.4002	8.374E+01
2.5180e-01	2.5266	1.066E+02
3.0274e-01	2.6477	1.312E+02

Area = 8.149E+00 m²/g

Slope = 4.278E+02

Y - Intercept = -3.998E-01

Correlation Coefficient = 0.999387

C = -1.069E+03

Appendix C The XRD of the studied catalysts

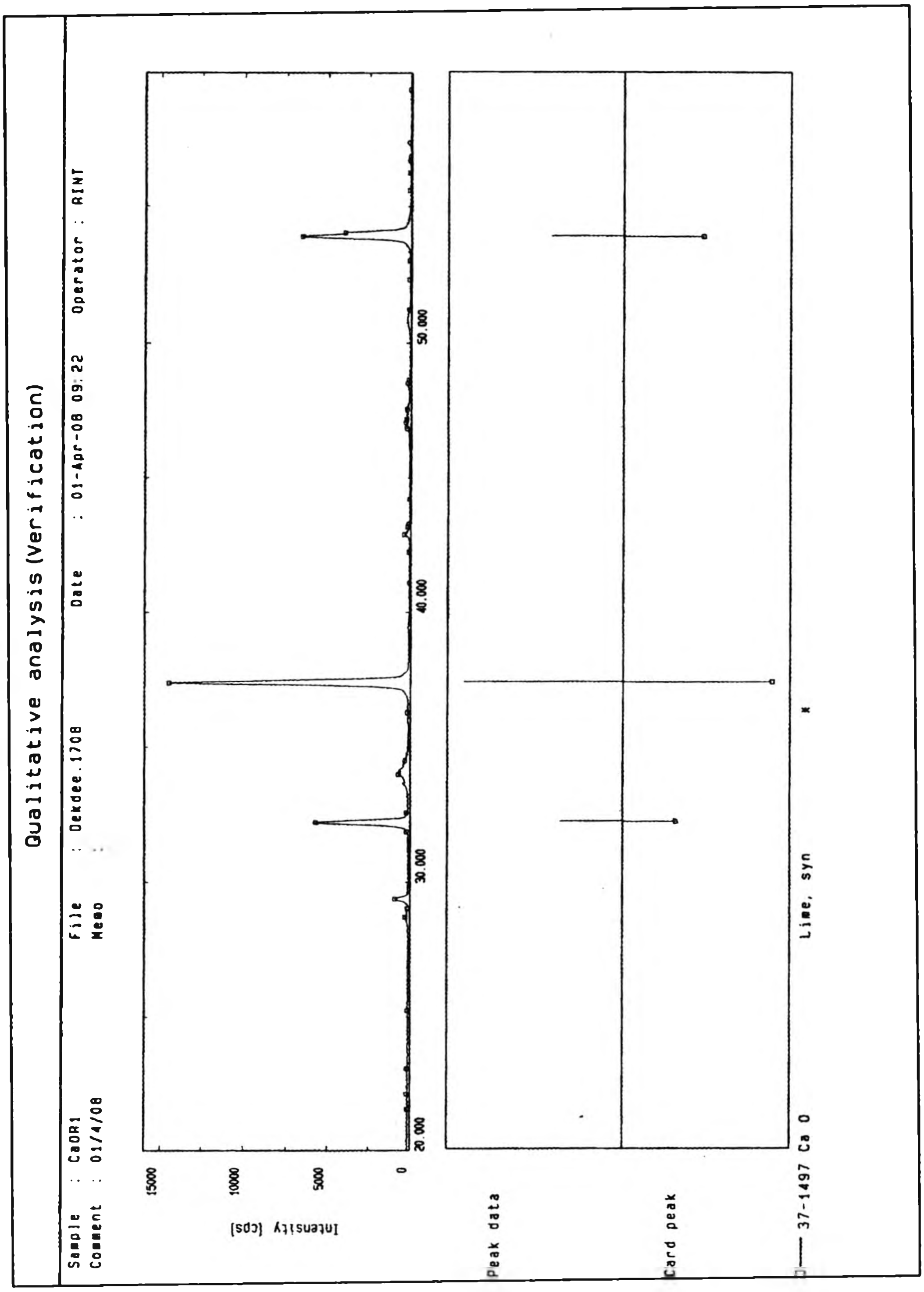

Figure C1 The X-Ray Diffraction spectrum of CaO

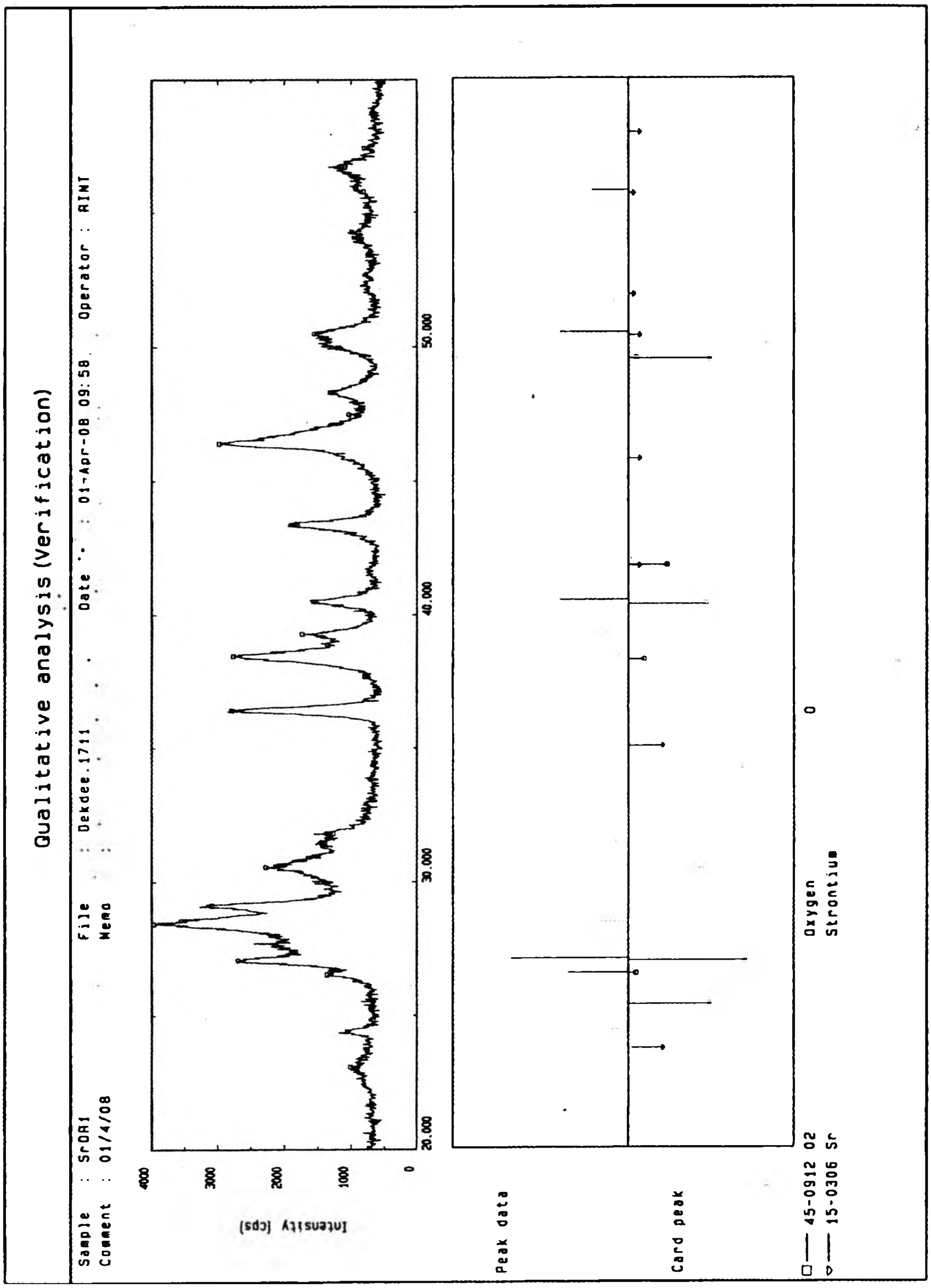

Figure C2 The X-Ray Diffraction spectrum of SrO

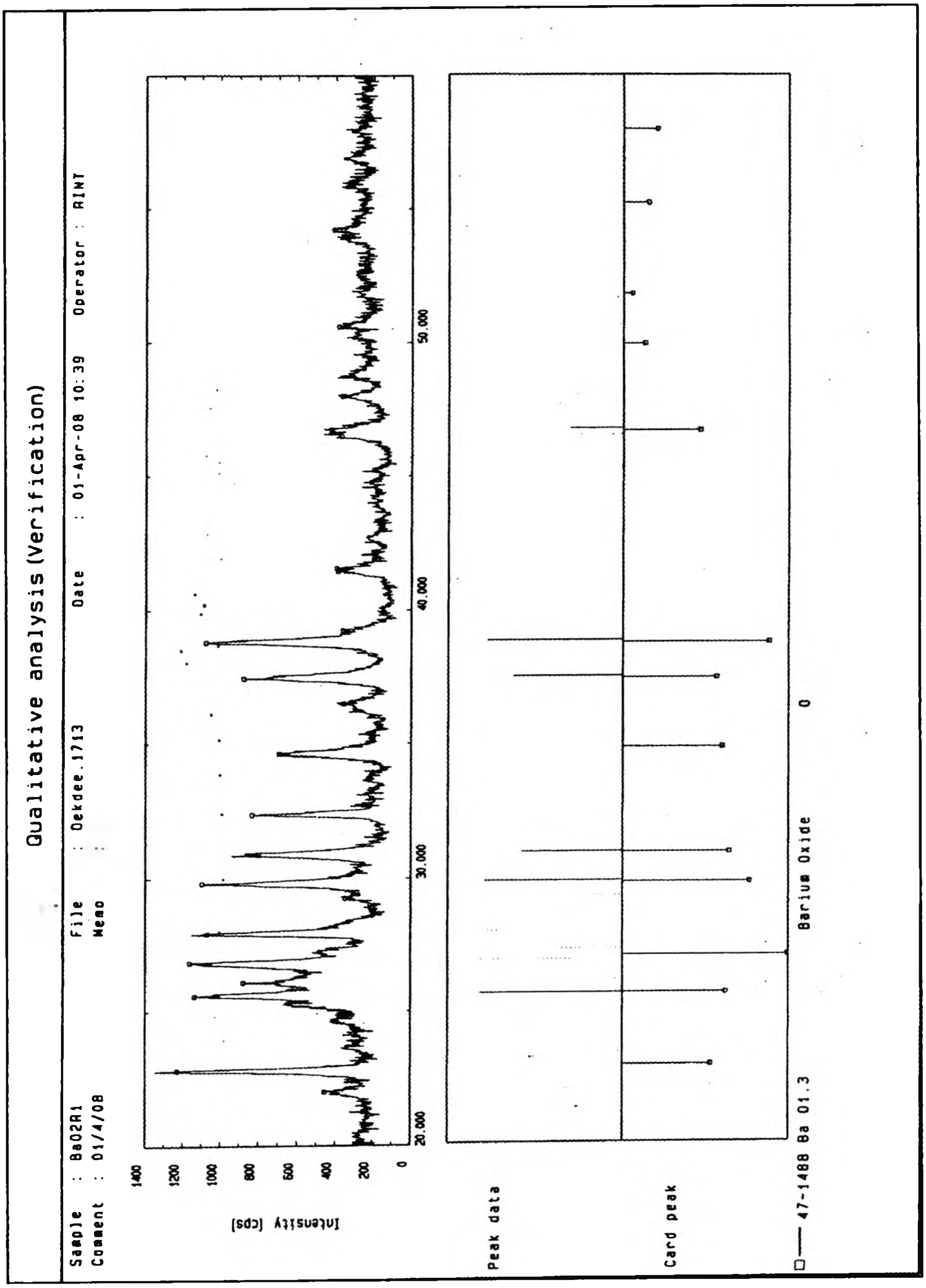

Figure C3 The X-Ray Diffraction spectrum of BaO

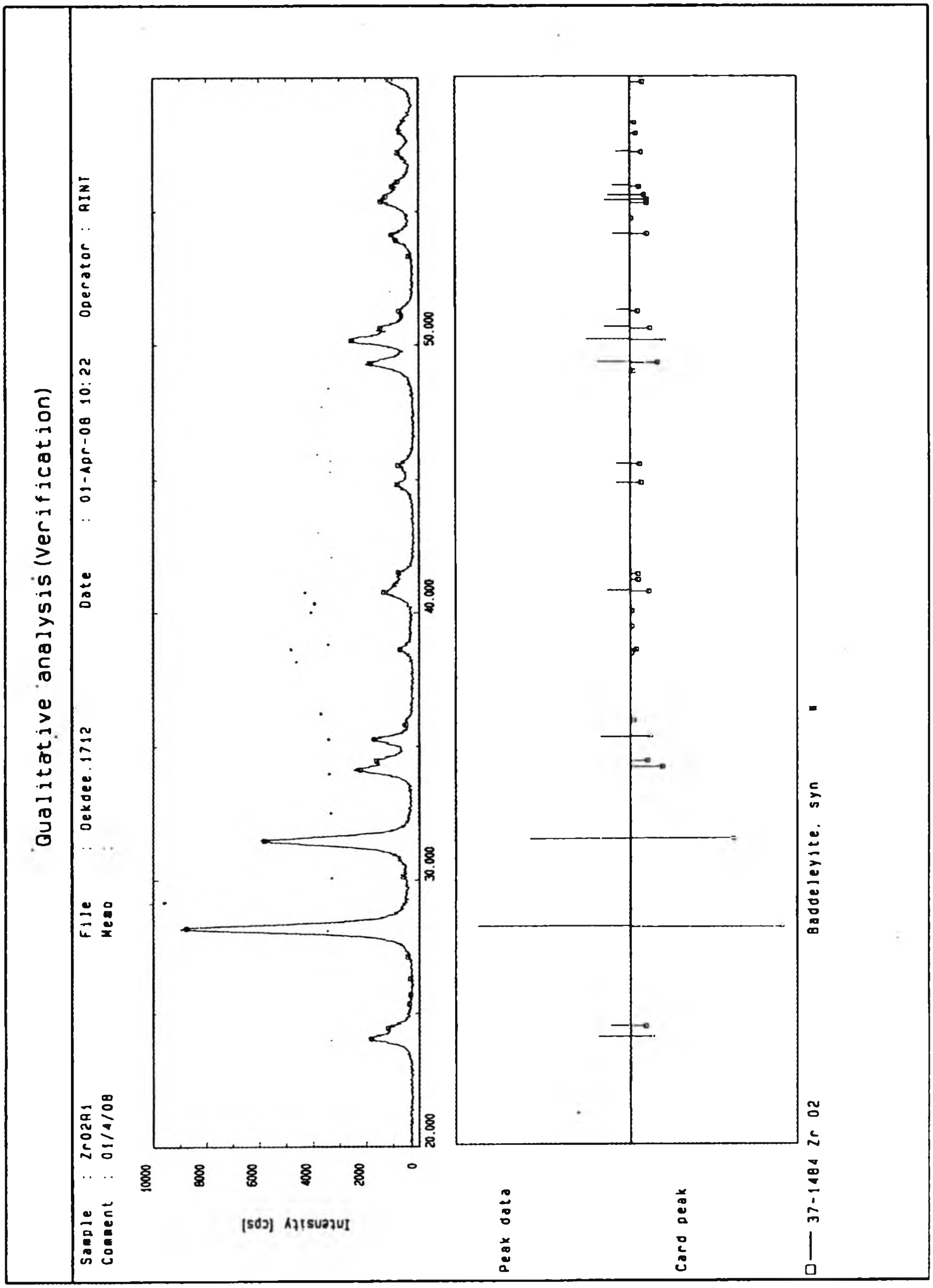

Figure C4 The X-Ray Diffraction spectrum of ZrO_2

Appendix D The number of basic sites of the studied catalysts obtained from TPD

Catalysts	number of basic sites (μmol/g)
MgO	26.70
CaO	18.70
SrO	11.00
BaO	5.39
ZrO_2	6.20

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Proceedings:

Thanasanvisut, D., Kitiyanan, B., and Abe, M. (2008, April 23) Synthesis of Diglycerol from Glycerol by Heterogeneous Base Catalysts. Proceedings of 14th PPC Symposium on Petroleum, Petrochems, and Polymers, Bangkok, Thailand.

Presentations:

1. Thanasanvisut, D., Kitiyanan, B., and Abe, M. (2008, April 23) Synthesis of Diglycerol from Glycerol by Heterogeneous Base Catalysts. Poster presented at the 14th PPC Symposium on Petroleum, Petrochems, and Polymers, Bangkok, Thailand.
2. Thanasanvisut, D., Boonpokkrong, V., and Kitiyanan, B. (2007, October 29-30) Homogeneous and Heterogeneous Catalytic Production of Diglycerol. Poster presented at 17th Thailand Chemical Engineering and AppliedChemistry Conference, Chiangmai, Thailand.