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APPENDICES

APPENDIX A

CALCULATION FOR CATALYST PREPARATION

Calculation of Si/Ti = 50 and Si/Cr = 150 atomic ratio for Cr-TS-1 samples

For example, to prepare Cr(III)-TS-1-A1 and Cr(VI)-TS-1-A1 samples

1. Calculation of mole of Si

The calculation is based on weight of sodium silicate for Si source.

Molecular Weight of Si = 28.0855

Molecular Weight of silicon oxide = 60.0843

Concentration of SiO_2 in sodium silicate = 28.5 %

Using sodium silicate 69 g

mole of Si used $= \frac{28.5}{100} \times \frac{69}{60.0843}$

$= 0.3272$ mole

2. Calculation of amount of titanium butoxide

At Si/Ti atomic ratio of 50

Using titanium butoxide 97 %, $Ti[O(CH_2)_3CH_3]_4$, for Ti source

Molecular Weight of Ti = 47.88

Molecular Weight of titanium butoxide = 340.26

Si/Ti atomic ratio of = 50

mole of titanium required = 0.3272/50

$= 6.546 \times 10^{-3}$ mole

amount of titanium butoxide 97% required $= 6.546 \times 10^{-3} \times 340.26 \times \frac{100}{97}$

$= 2.2962$ g

3. Calculation of amount of chromium nitrate nanohydrate and chromium oxide

To prepare Cr(III)-TS-1-A1

Using chromium nitrate nanohydrate , $CrNO_3 \cdot 9H_2O$, for Cr source.

Molecular Weight of Cr	= 51.99
Molecular Weight of $CrNO_3 \cdot 9H_2O$	= 400.15
Si/Cr atomic ratio	= 150
mole of chromium required	= 0.3272/150
	= 2.181×10^{-3} mole
amount of $CrNO_3 \cdot 9H_2O$ required	= $2.181 \times 10^{-3} \times 400.15$
	= 0.873 g

Therefore amount of $CrNO_3 \cdot 9H_2O$ added in A1 solution = 0.873×2

= 1.746 g

To prepare Cr(VI)-TS-1-A1

Using chromium oxide, CrO_3, for Cr source.

Molecular Weight of Cr	= 51.99
Molecular Weight of CrO_3	= 99.99
Si/Cr atomic ratio	= 150
mole of chromium required	= 0.3272/150
	= 2.181×10^{-3} mole
amount of CrO_3 required	= $2.181 \times 10^{-3} \times 99.99$
	= 0.218 g

Therefore amount of CrO_3 added in A1 solution = 0.218×2

= 0.436 g

APPENDIX B

CALCULATION OF G VALUE

g value is the best index for determination of transition metal state in the catalyst framework. This value is derived from the following equation.

$$g = 71.448\left(\frac{mT}{MHz}\right)\times\frac{\text{frequency (MHz)}}{\text{magnetic field (mT)}}$$

For example, Cr(III)-TS-1-A1A2, peak of spectra by ESR appears at magnetic field of 341.51 mT with 9.44 MHz. Consequently, g value for Cr(III)-TS-1-A1A2 can be calculated from

$$g = 71.448\left(\frac{mT}{MHz}\right)\times\frac{9.44\,(MHz)}{341.51\,(mT)}$$

$$g = 1.974$$

APPENDIX C

DATA OF CATALYTIC EXPERIMENTS

Table C1 Data of Figure 4.6a

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	0	19.84	0.00	0.00	80.16	0.00
150	2	2.19	0.00	0.00	97.81	0.00
200	17	0.75	0.00	0.05	99.20	0.00
250	26	5.41	0.07	0.22	94.29	0.00
300	41	25.31	0.43	0.40	73.07	0.78
350	72	39.51	1.56	0.21	57.28	1.44
400	94	38.11	1.77	0.08	42.82	17.21
450	88	22.39	2.13	0.09	60.51	14.87
500	83	22.32	2.18	0.10	69.11	6.29

Table C2 Data of Figure 4.6b

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	0	17.80	0.00	0.00	82.20	0.00
150	1	3.14	0.00	0.0	96.86	0.00
200	6	3.34	0.00	0.44	96.22	0.00
250	20	17.07	0.79	1.46	79.99	0.69
300	63	27.41	2.32	0.85	65.74	3.68
350	85	21.19	3.18	0.24	54.61	20.78
400	94	19.61	3.12	0.16	56.22	20.90
450	89	19.12	4.55	0.14	54.31	21.87
500	89	21.30	3.90	0.07	53.16	21.57

Table C3 Data of Figure 4.6c

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	0	14.28	0.00	2.03	83.69	0.00
150	0	5.76	0.00	0.90	93.34	0.00
200	1	13.24	0.00	0.82	85.94	0.00
250	8	26.51	0.11	1.25	72.14	0.00
300	46	32.46	0.90	0.93	63.23	2.48
350	84	17.00	2.77	0.24	64.92	15.07
400	91	17.26	3.54	0.16	51.30	27.74
450	91	20.63	2.44	0.16	49.19	27.57
500	93	24.34	2.38	0.12	49.49	23.68

Table C4 Data of Figure 4.6d

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	1	28.73	0.00	1.52	69.75	0.00
150	1	19.70	0.00	1.00	79.30	0.00
200	7	4.58	0.00	0.14	95.28	0.00
250	13	14.16	0.31	1.53	84.01	0.00
300	38	22.21	1.40	1.10	71.45	3.86
350	80	20.13	2.78	0.35	62.65	14.11
400	93	17.10	3.75	0.16	58.05	20.94
450	94	17.39	4.15	0.14	57.63	20.70
500	84	20.18	3.68	0.10	63.09	12.95

Table C5 Data of Figure 4.6e

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	0	12.42	0.00	0.00	87.58	0.00
150	0	2.31	0.00	0.00	97.69	0.00
200	2	2.55	0.00	0.13	97.32	0.00
250	12	11.37	0.54	0.88	87.21	0.00
300	42	24.10	1.67	1.06	69.90	3.28
350	74	25.74	2.75	0.52	59.72	11.28
400	86	26.21	3.12	0.20	52.48	17.99
450	93	28.46	3.54	0.11	50.81	17.08
500	91	27.80	2.56	0.06	54.74	14.83

Table C6 Data of Figure 4.6f

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	0	28.55	0.00	0.00	71.45	0.00
150	1	37.36	0.00	0.81	61.84	0.00
200	6	2.07	0.00	0.16	97.77	0.00
250	13	17.66	0.24	1.35	80.75	0.00
300	39	26.45	0.96	1.01	70.89	0.70
350	79	23.75	2.99	0.39	58.79	14.08
400	91	16.14	4.38	0.16	49.04	30.29
450	95	18.97	3.37	0.10	48.83	28.74
500	98	21.11	2.52	0.08	52.71	23.58

Table C7 Data of Figure 4.6g

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	0	35.10	0.00	4.03	60.86	0.00
150	1	1.47	0.00	0.19	98.34	0.00
200	7	0.74	0.00	0.09	99.17	0.00
250	10	7.30	0.37	0.74	91.96	0.00
300	28	25.86	0.59	1.42	71.54	1.18
350	64	26.37	1.42	0.67	68.26	4.70
400	85	23.41	2.17	0.23	57.77	18.59
450	83	22.94	1.77	0.20	57.84	19.03
500	99	23.83	2.77	0.14	59.52	16.51

Table C8 Data of Figure 4.6h

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	1	28.49	0.00	1.03	70.48	0.00
150	2	14.30	0.00	0.40	85.30	0.00
200	4	8.90	0.00	0.32	90.78	0.00
250	9	10.80	0.07	0.75	88.39	0.00
300	27	24.87	0.54	1.24	71.74	1.61
350	62	24.27	1.61	0.62	69.08	4.42
400	86	20.16	2.52	0.20	59.60	17.52
450	90	19.83	2.31	0.15	59.92	17.78
500	96	22.08	2.34	0.10	58.86	16.62

Table C9 Data of Figure 4.6i

Reaction temperature(°C)	% 2-propanol (C)	% propylene (S)	% acetic (S)	% isopropyl ether (S)	% acetone (S)	% CO_2 (S)
100	1	31.89	0.00	1.31	66.79	0.00
150	1	23.65	0.00	0.97	75.38	0.00
200	3	10.08	0.00	0.55	89.37	0.00
250	11	10.89	0.10	0.77	88.25	0.00
300	33	22.98	0.71	1.10	73.58	1.63
350	70	25.58	2.07	0.55	68.02	3.78
400	89	20.00	2.90	0.21	58.60	18.29
450	87	17.42	1.66	0.15	66.27	14.50
500	91	20.21	1.89	0.12	62.95	14.84

APPENDIX D

CALIBRATION CURVE

Flame ionization detector gas chromatograph, model 8A, was used to analyze the concentrations of oxygenated compounds. 2-propanol, propylene, isopropyl ether acetone and acetic acid were analyzed by GC model 8A with using Carbopack B/3% SP-1500.

Gas chromatograph with the thermal conductivity detector, model 8A, was used to analyze the concentration of CO_2 by using molecular sieve 5A and porapak-Q columns respectively.

The calibration curves of 2-propanol, propylene, isopropyl ether, acetone, acetic acid and CO_2 are illustrated in the following figures.

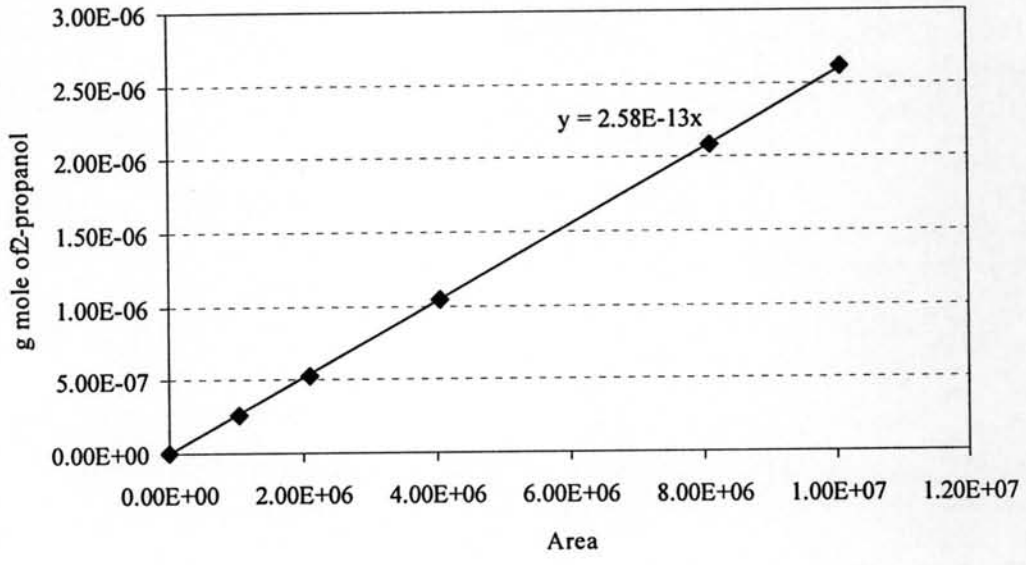

Figure D1 The calibration curve of 2-propanol

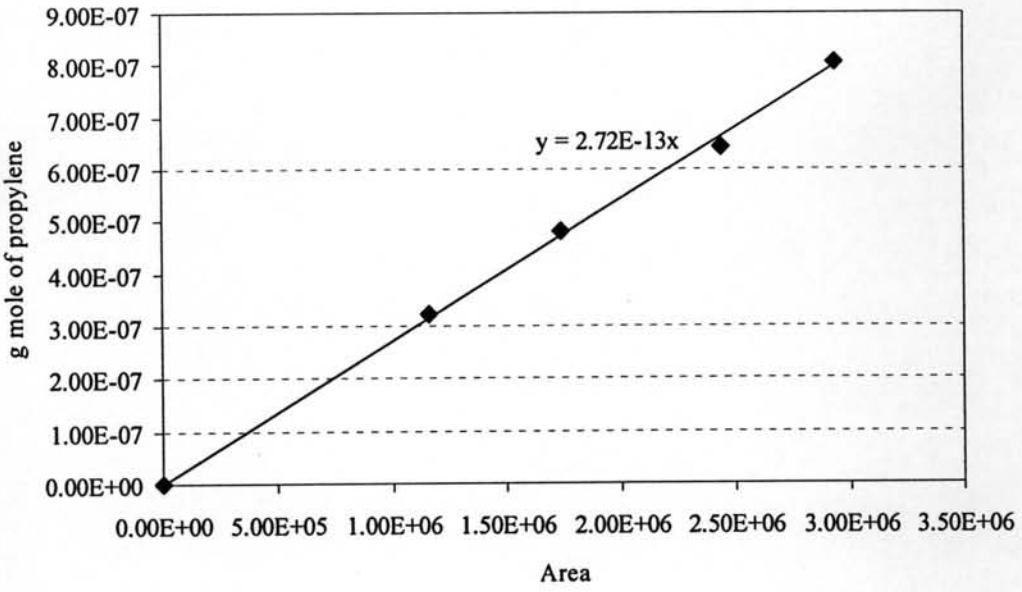

Figure D2 The calibration curve of propylene

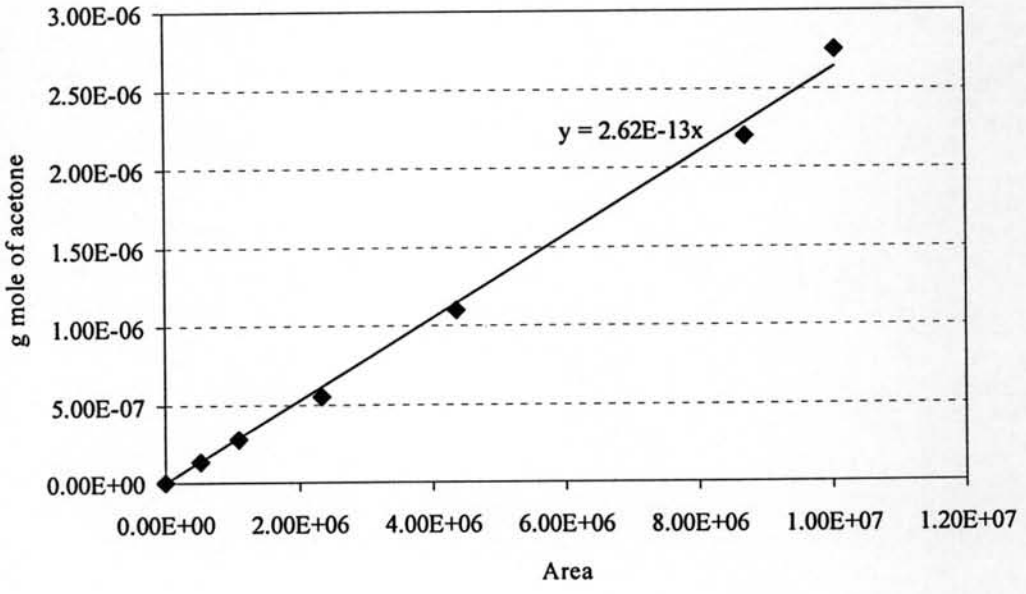

Figure D3 The calibration curve of acetone

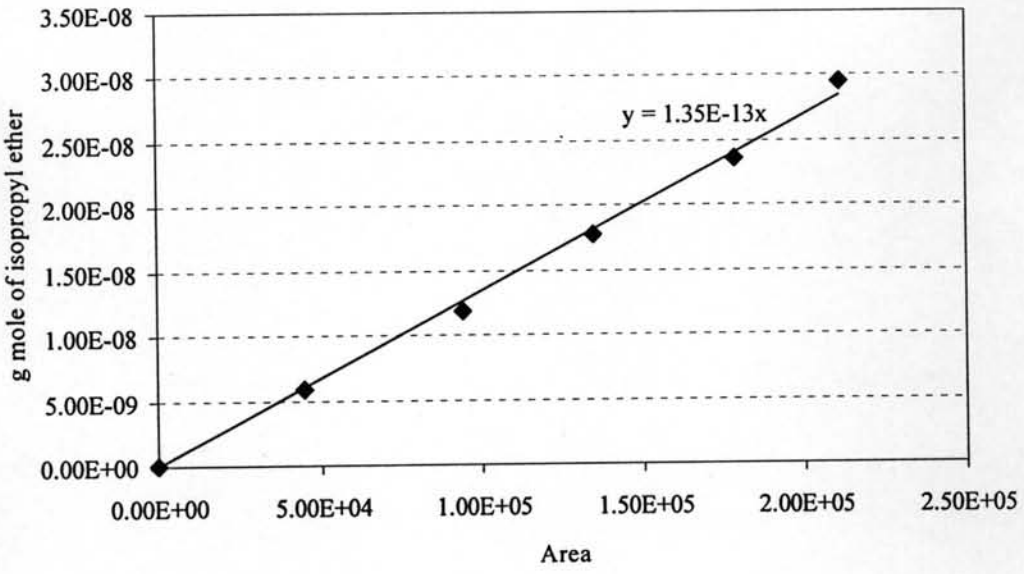

Figure D4 The calibration curve of isopropyl ether

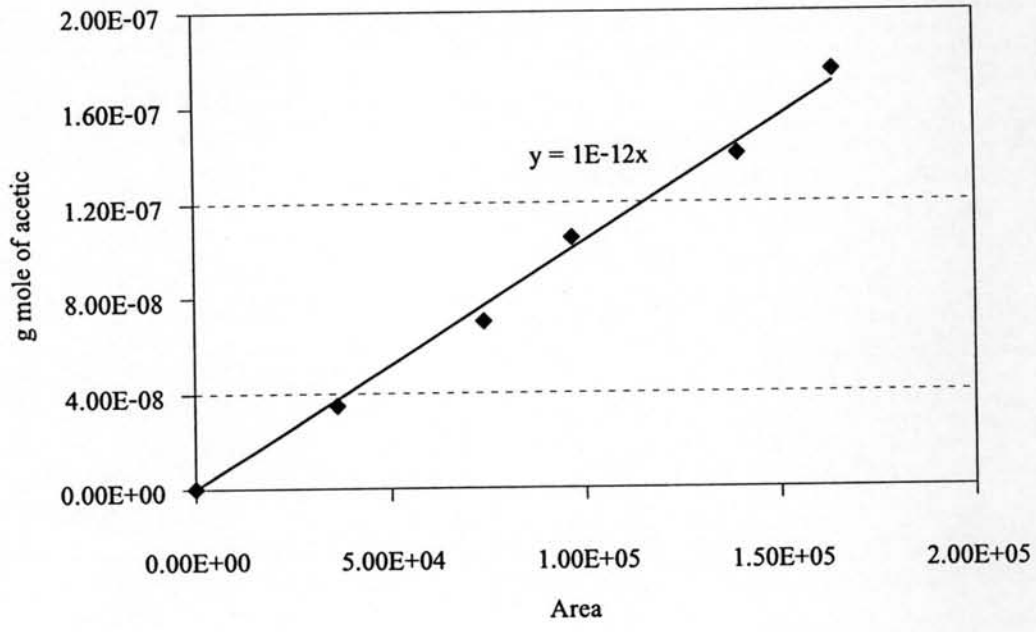

Figure D5 The calibration curve of acetic acid

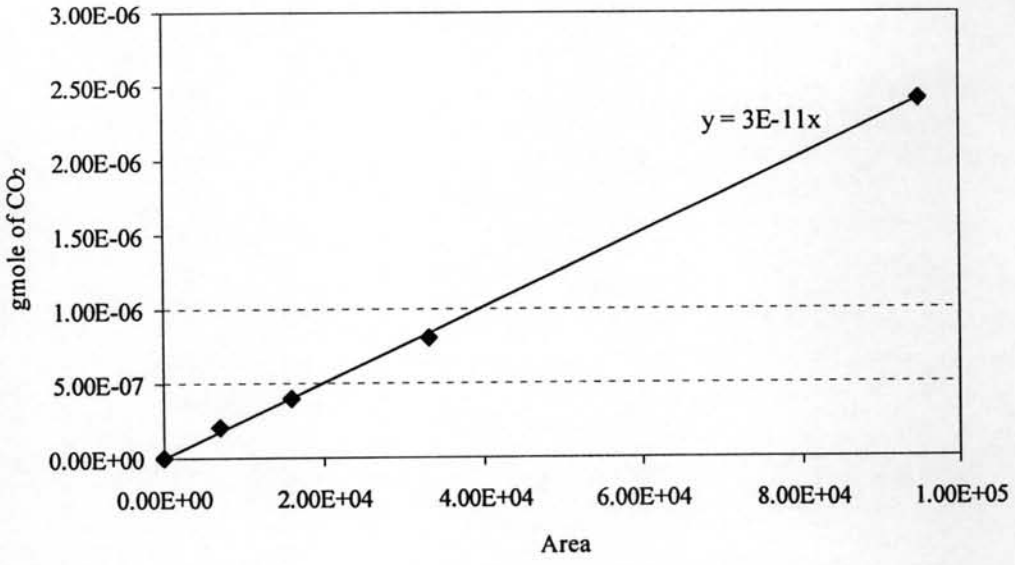

Figure D6 The calibration curve of CO_2

APPENDIX E

DATA AND CALCULATION OF ACID SITE

Table E1 Reported total peak area from Micromeritrics Chemisorb 2750

Sample	Reported total peak area
Cr(III)-TS-1-A1	0.0386025
Cr(III)-TS-1-A1A2	0.0384434
Cr(III)-TS-1-A2	0.0293940
Cr(III)-TS-1-B1	0.0291932
Cr(III)-TS-1-C1	0.0292235
Cr(VI)-TS-1-A1	0.0282431
Cr(III)-TS-1-A1A2	0.0259882
Cr(III)-TS-1-B1	0.0411846
Cr(III)-TS-1-C1	0.0413656

1. Calculation of total acid sites

For example, Cr(III)-TS-1-A1, total acid site is calculated from the following step.

1.1 Conversion of total peak area to peak volume

Conversion factor from Micromeritrics Chemisorb 2750 is equal to 77.57016 ml/area unit. Therefore, total peak volume is derived from

Total peak volume = 77.57016 × total peak area

= 77.57016 × 0.0386025

= 2.99440 ml

1.2 Calculation for adsorbed volume of 15% NH_3

adsorbed volume of 15% NH_3 = 0.15 × total peak volume

= 0.15 × 2.99440 ml

= 0.44916 ml

1.3 Total acid sties are calculated from the following equation

$$\text{Total acid sites} = \frac{(\text{Adsorbed volume, ml}) \times 101.325\ \text{Pa}}{\left(8.314 \times 10^{-3}\ \frac{\text{Pa} \cdot \text{ml}}{\text{K} \cdot \mu\text{mol}}\right) \times 298\ \text{K} \times (\text{weight of catalyst, g})}$$

For Cr(III)-TS-1-A1 sample, 0.1050 g

$$\text{Total acid sites} = \frac{0.44916\ \text{ml} \times 101.325\ \text{Pa}}{\left(8.314 \times 10^{-3}\ \frac{\text{Pa} \cdot \text{ml}}{\text{K} \cdot \mu\text{mol}}\right) \times 298\ \text{K} \times (0.1050\ \text{g})}$$

= 175 μmol /g

2. Calculation of acid site ratio

To calculate acid site ratio, experiment data from Micromeritrics Chemisorb 2750 are transferred to peak fitting program to separate peak. As known, the first peak of desorption process is indicated as weak acid, relative with another peak, and the second one is strong acid. Ratio of each acid site on the catalyst surface is calculated from peak areas reported by peak fitting program as shown above.

For example, Cr(III)-TS-1-A1, the ratio of each acid site on catalyst surface is calculated from the following equation.

$$\text{The ratio of weak acid} = \frac{1^{\text{st}}\ \text{peak area}}{\text{summation of both peak areas}} \times 100\ \%$$

From figure E1b., 1st peak area and 2nd peak area are equal to 0.0197663 and 0.0226847, respectively.

$$\text{The ratio of weak acid} = \frac{0.0197663}{0.0197663 + 0.0226847} \times 100\ \%$$

$$= 46.56\ \%$$

$$\text{therefore, the ratio of strong acid} = 100 - 46.56\ \%$$

$$= 53.44\ \%$$

Note. Reported center values of both peaks from peak fitting program reveal times at the maximum of both peaks occur. Since, we know the relationship between time and temperature during desorption process from Micromeritrics Chemisorb 2750, hence, the temperature at the maximum of both peaks as we state as desorption temperature of both acid sites can be located.

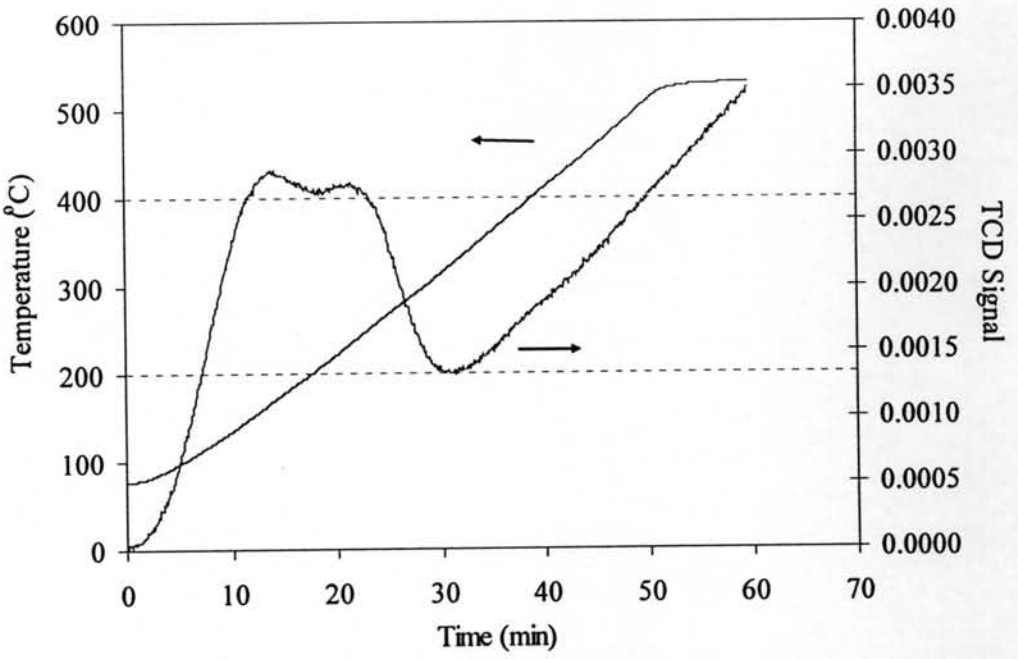

Figure E1a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(III)-TS-1-A1

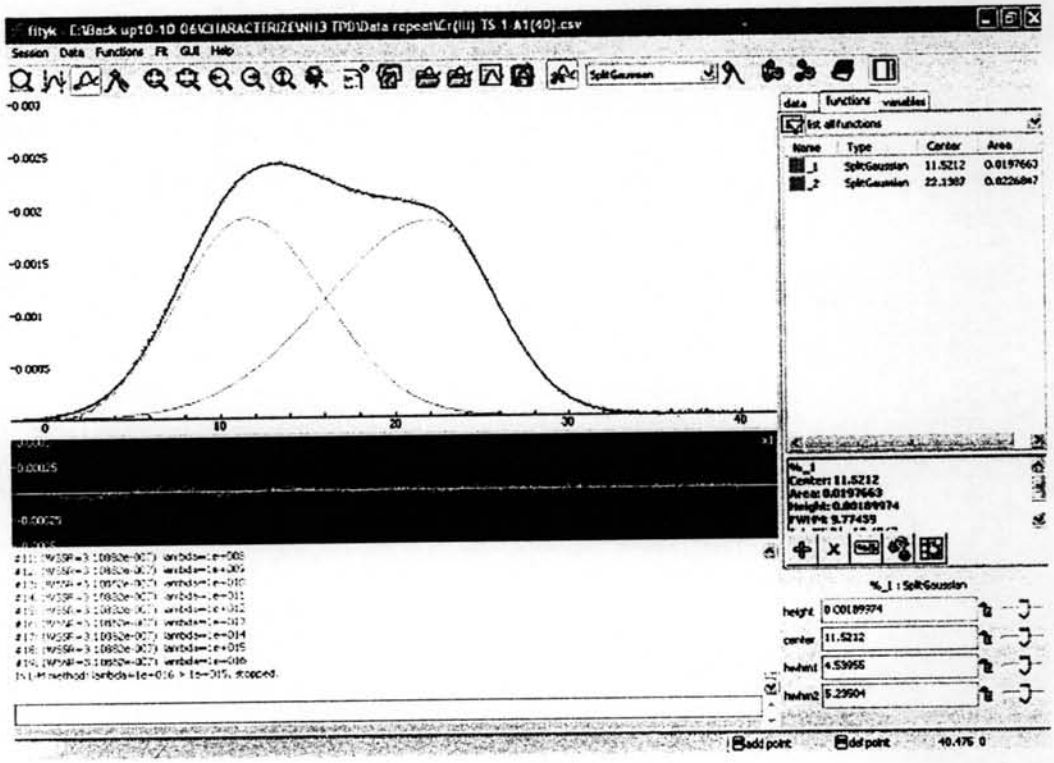

Figure E1b. Data for calculating of acid site ratio of Cr(III)-TS-1-A1 from peak fitting program

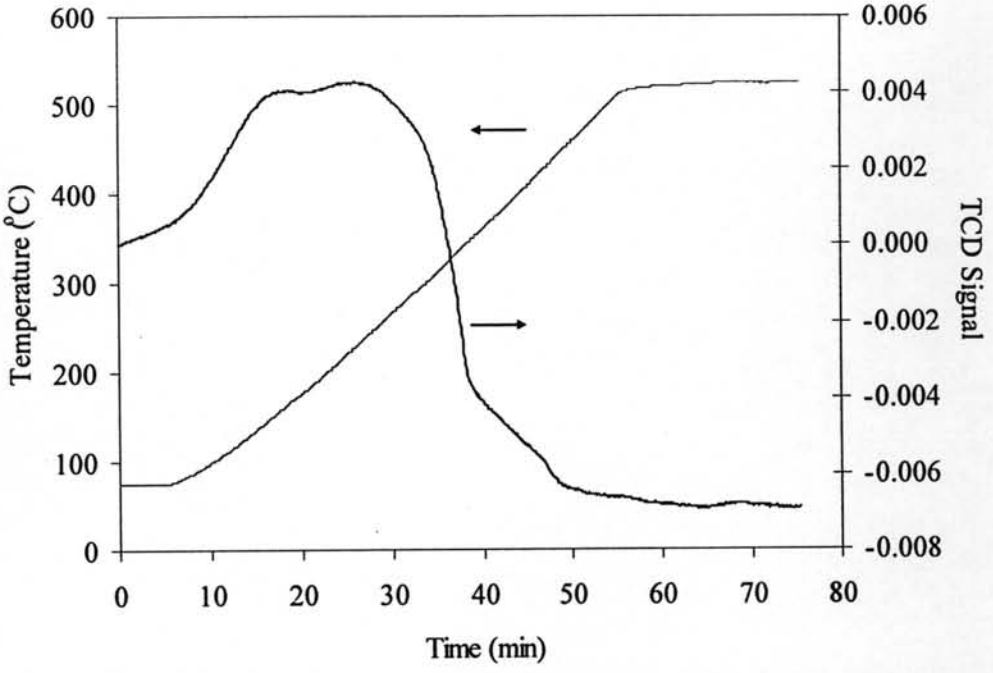

Figure E2a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(III)-TS-1-A1A2

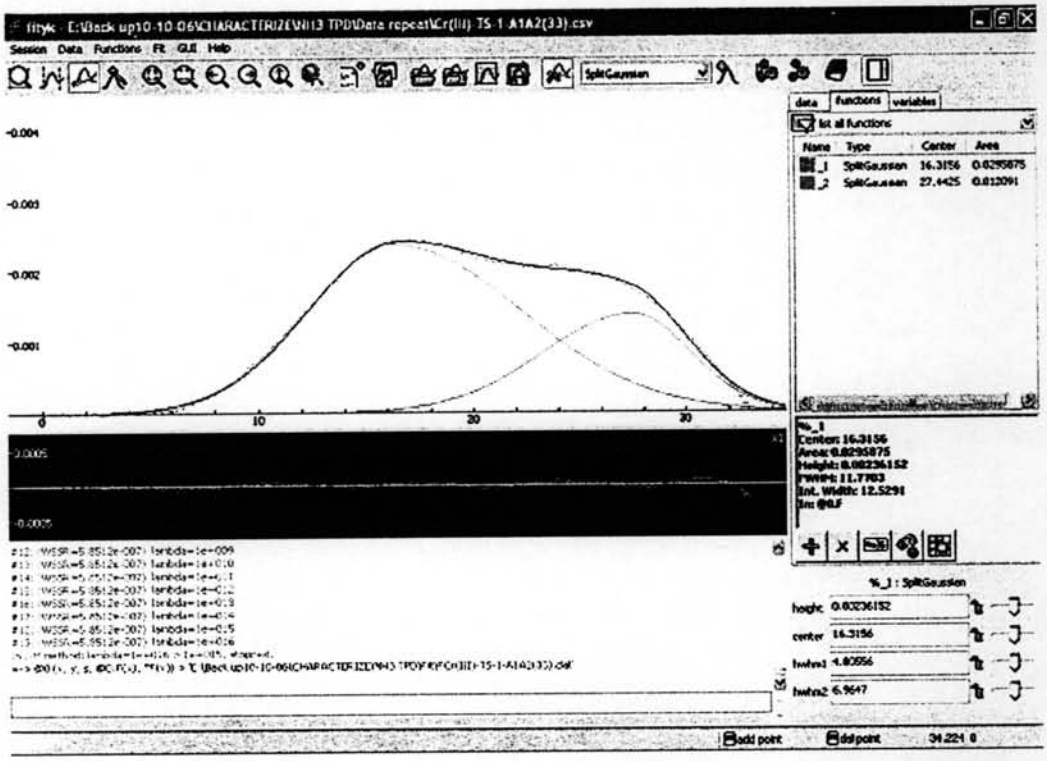

Figure E2b. Data for calculating of acid site ratio of Cr(III)-TS-1-A1A2 from peak fitting program

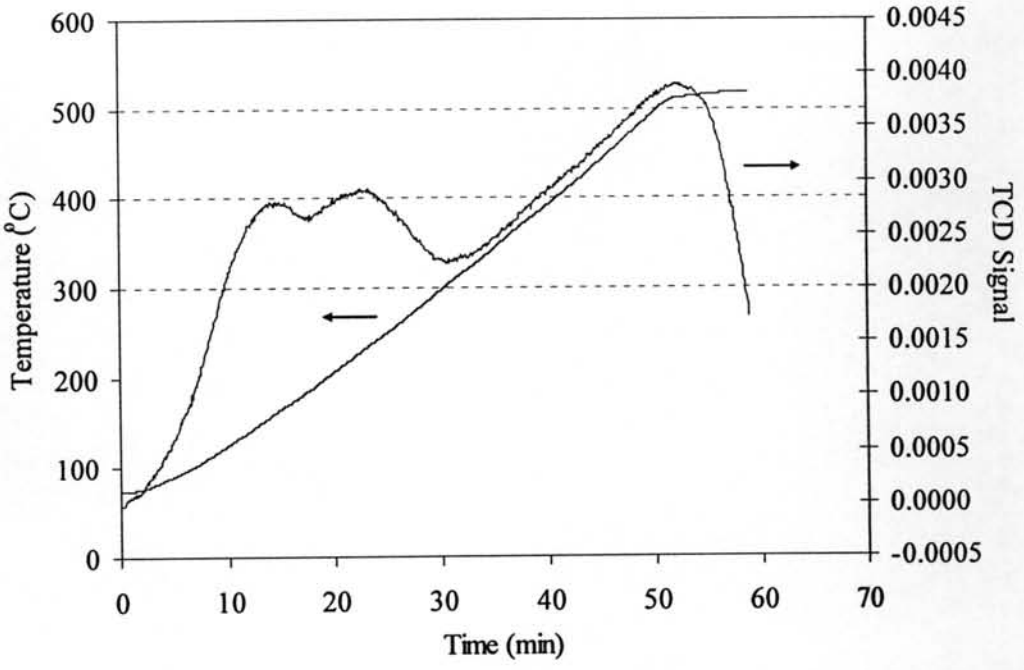

Figure E3a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(III)-TS-1-A2

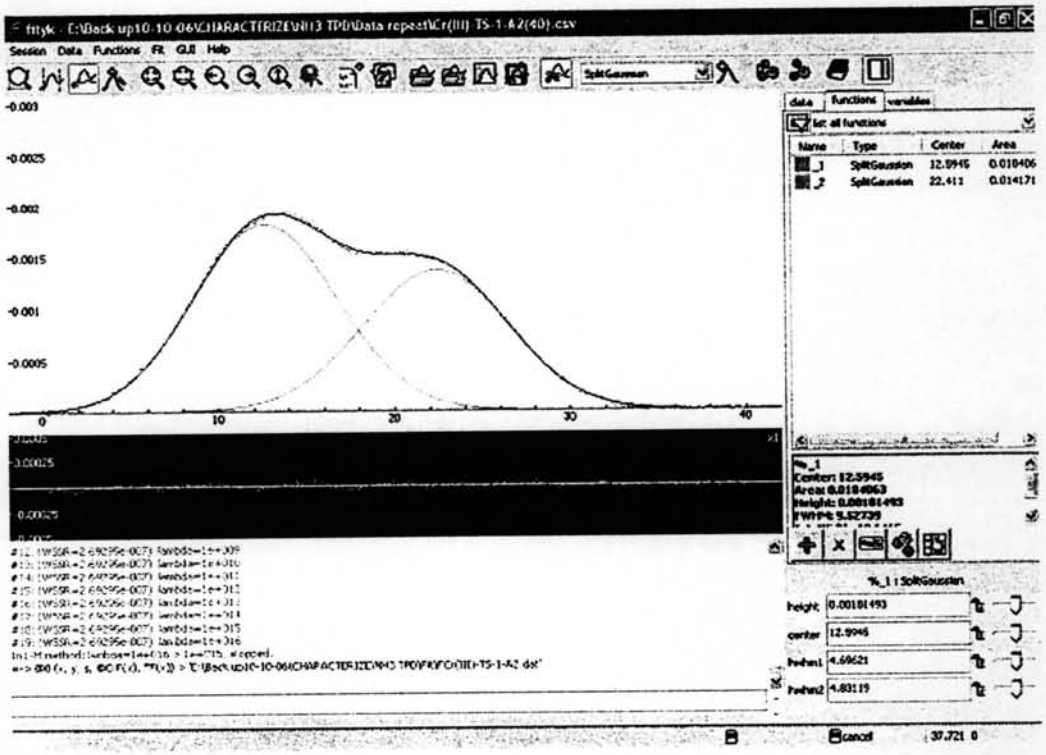

Figure E3b. Data for calculating of acid site ratio of Cr(III)-TS-1-A2 from peak fitting program

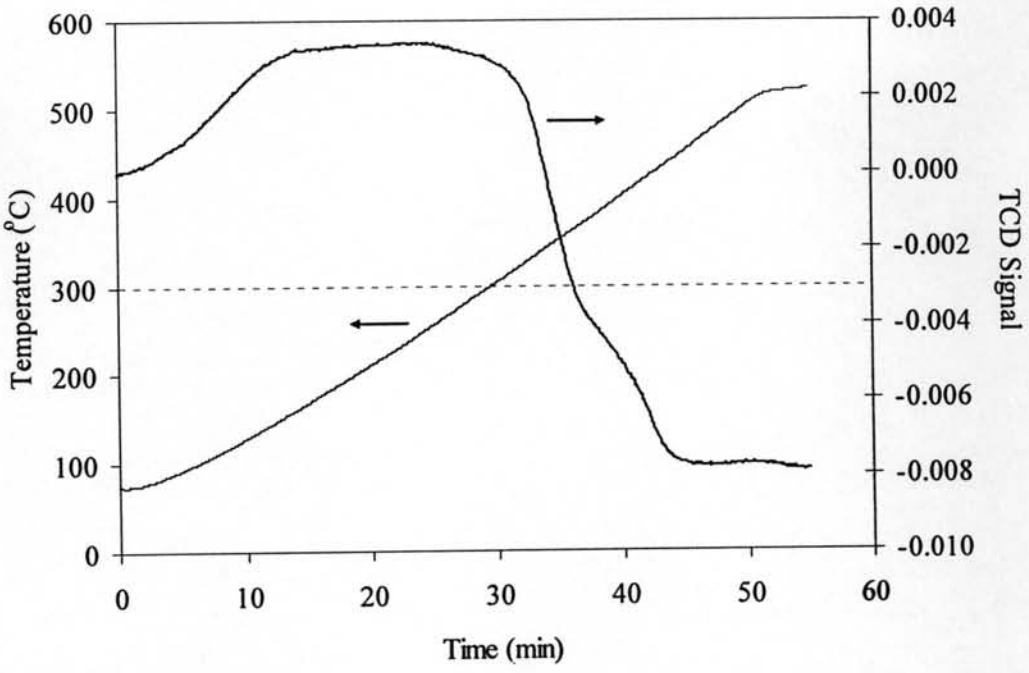

Figure E4a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(III)-TS-1-B1

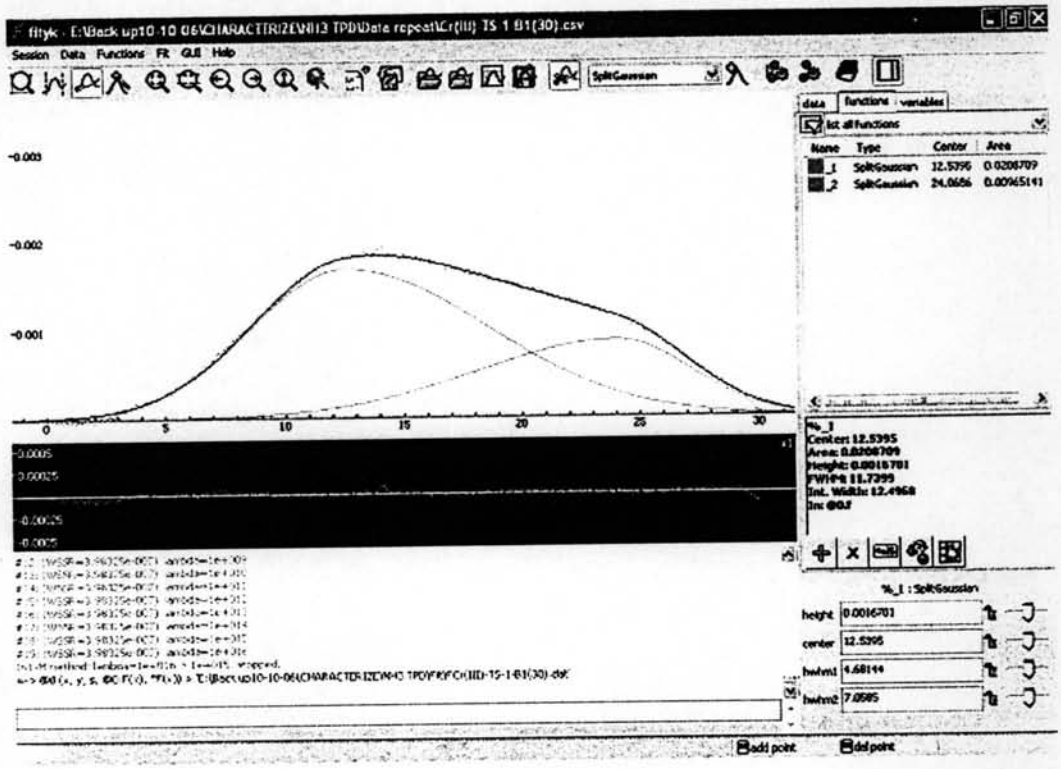

Figure E4b. Data for calculating of acid site ratio of Cr(III)-TS-1-B1 from peak fitting program

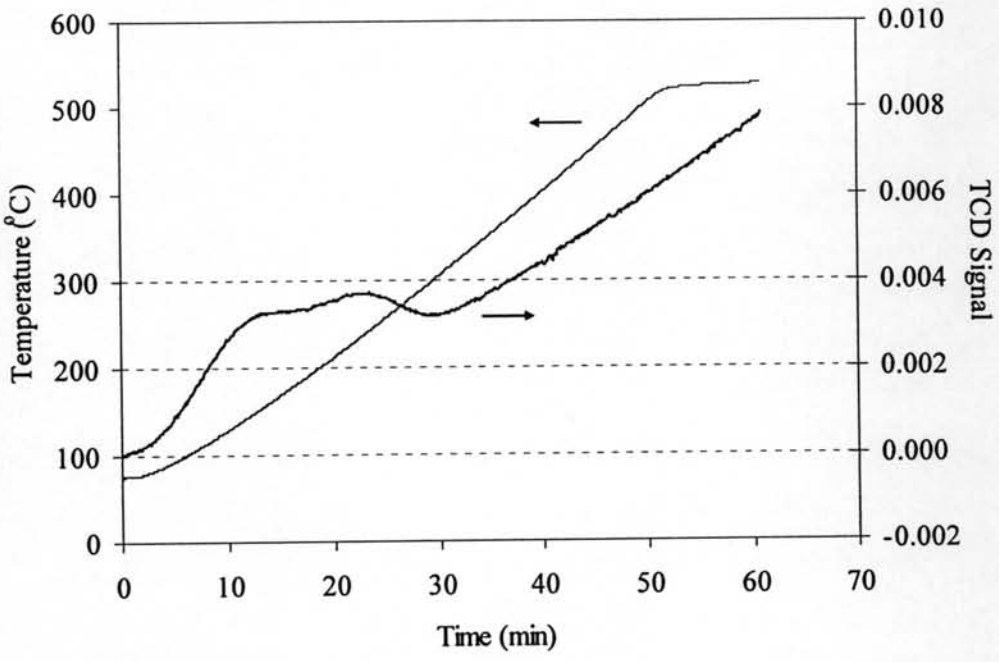

Figure E5a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(III)-TS-1-C1

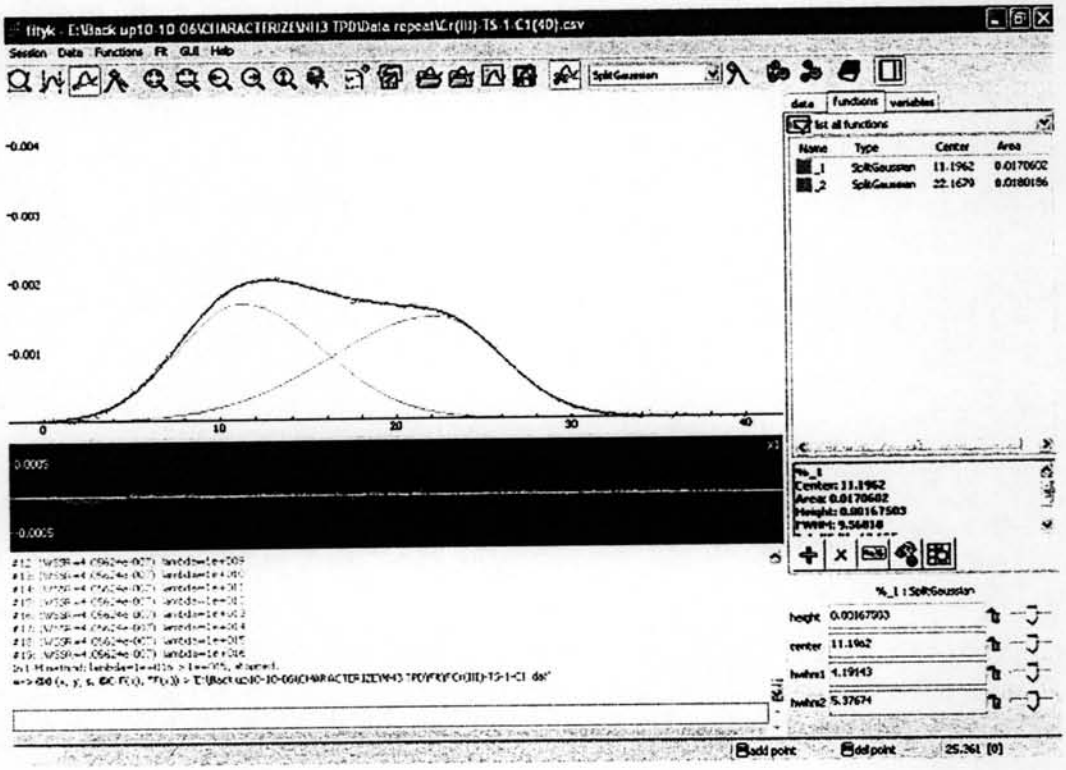

Figure E5b. Data for calculating of acid site ratio of Cr(III)-TS-1-C1 from peak fitting program

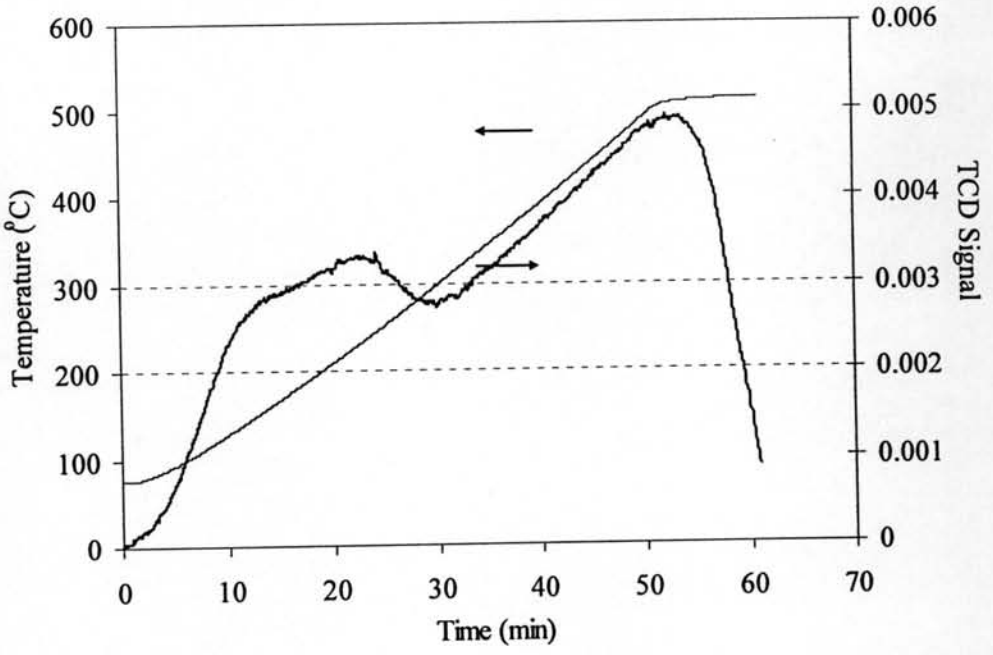

Figure E6a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(VI)-TS-1-A1

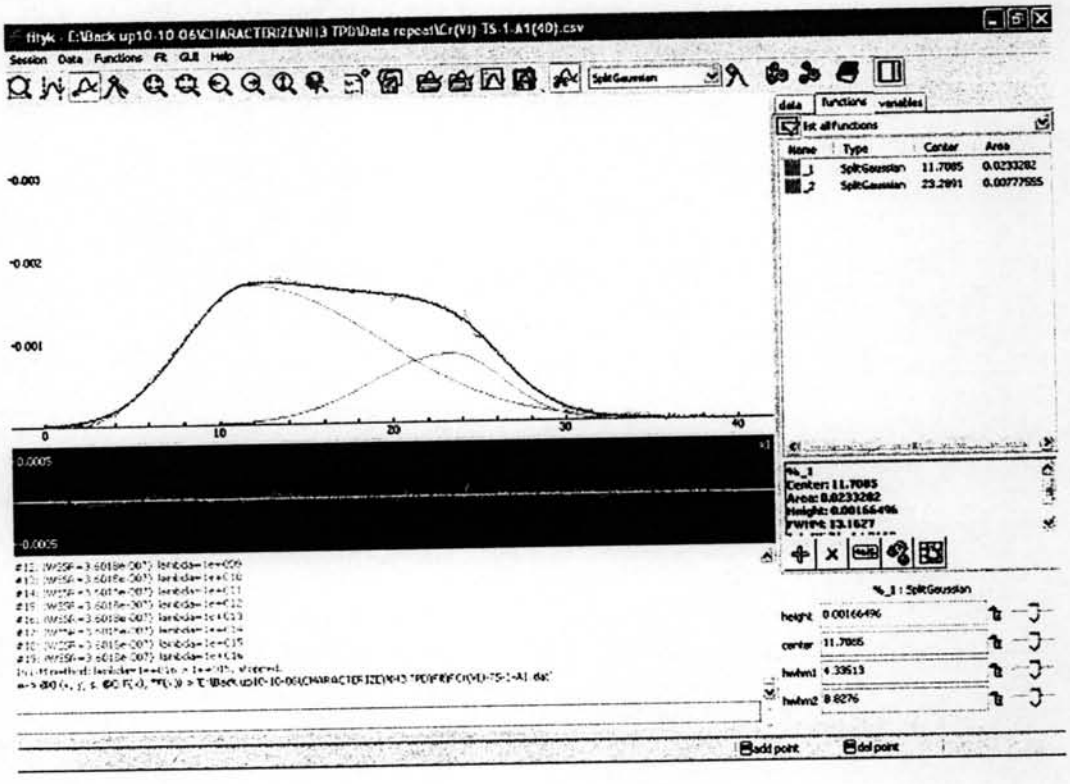

Figure E6b. Data for calculating of acid site ratio of Cr(VI)-TS-1-A1 from peak fitting program

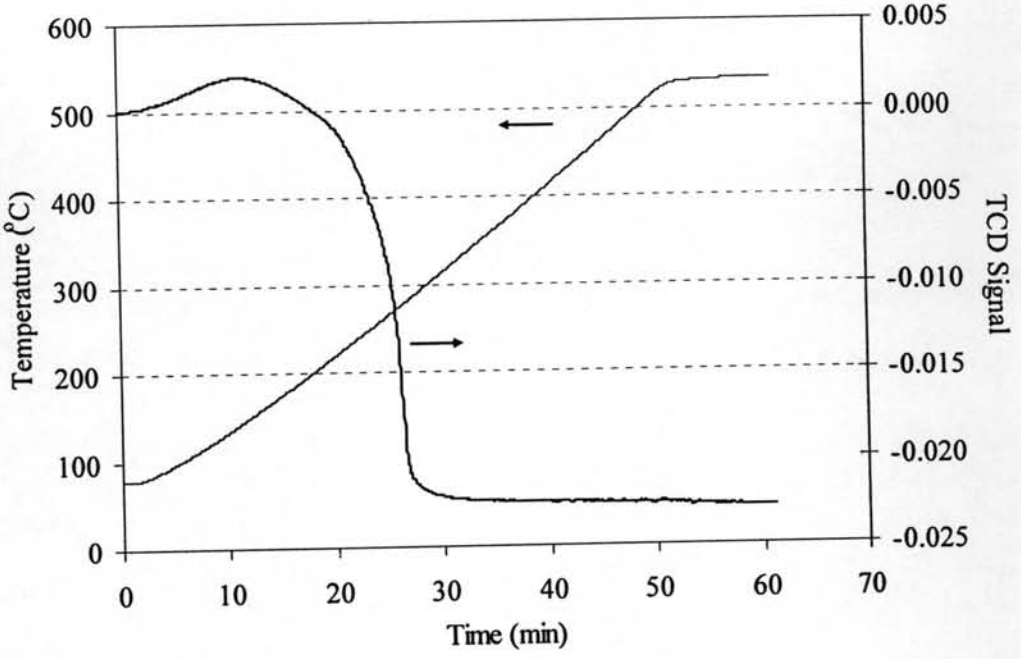

Figure E7a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(VI)-TS-1-A1A2

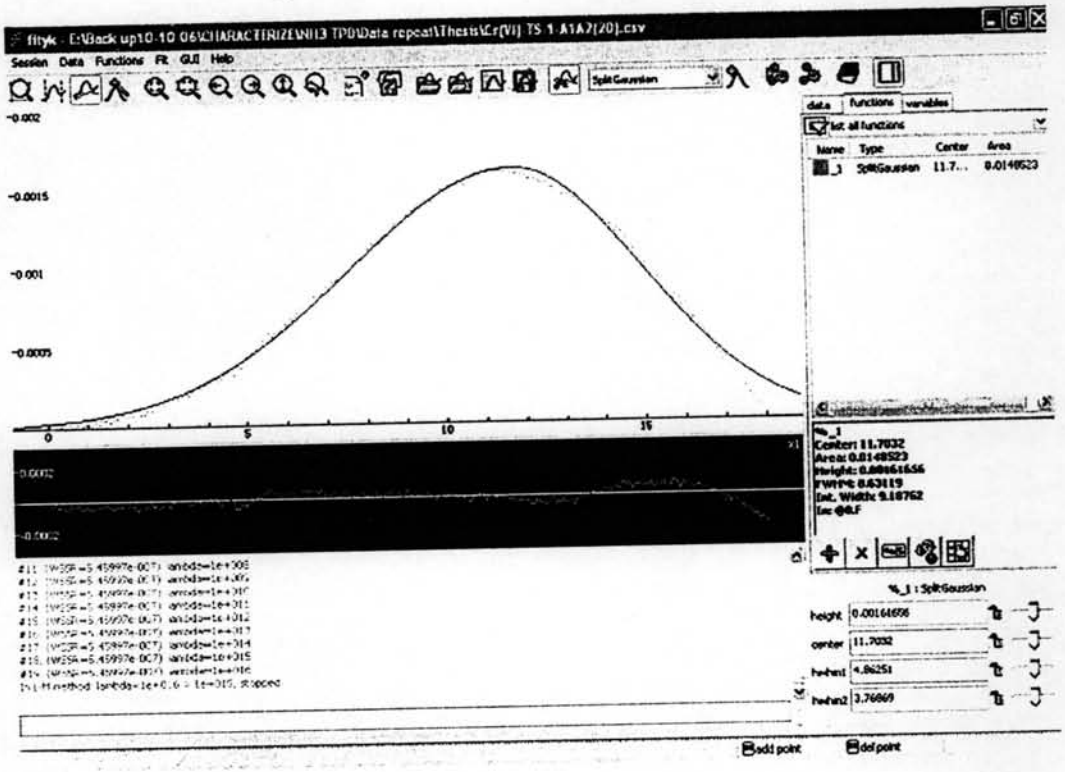

Figure E7b. Data for calculating of acid site ratio of Cr(VI)-TS-1-A1A2 from peak fitting program

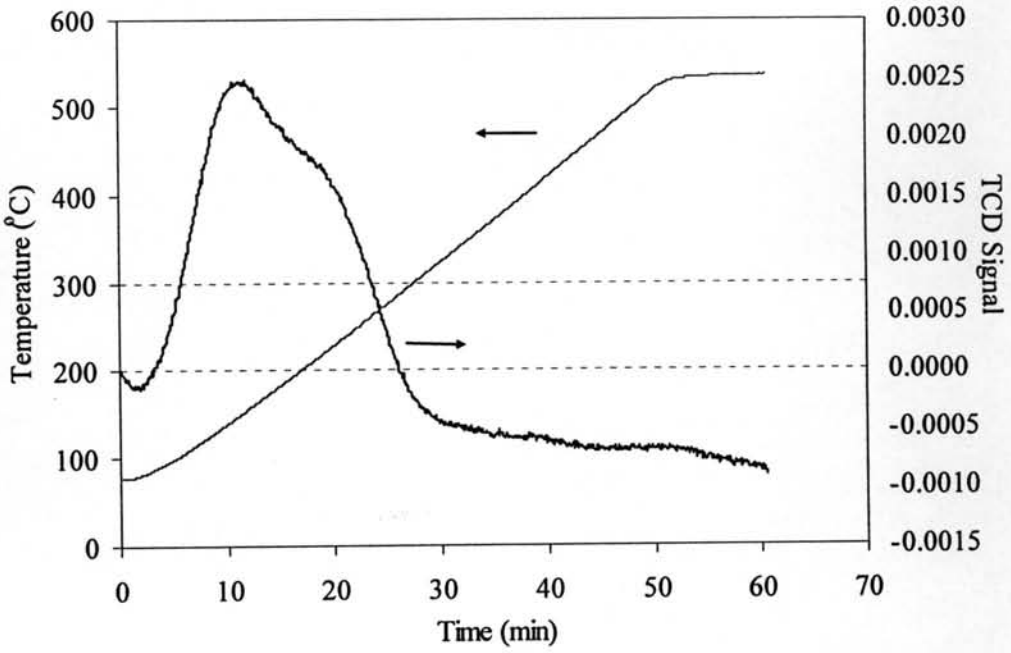

Figure E8a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(VI)-TS-1-B1

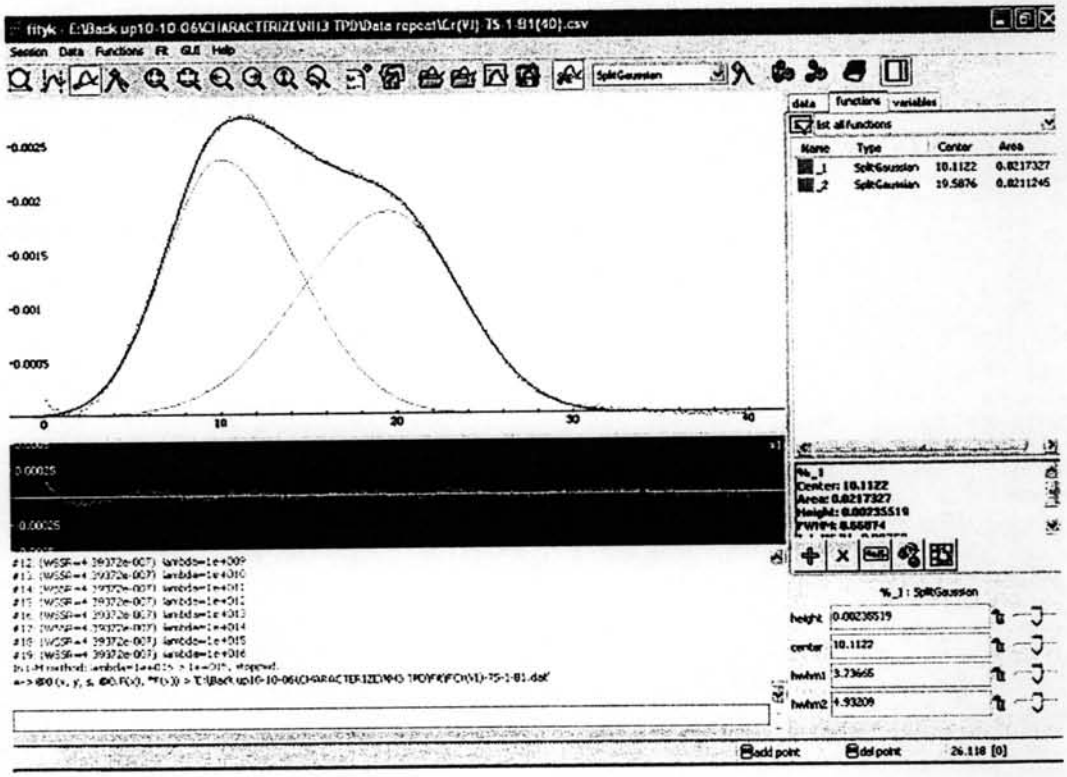

Figure E8b. Data for calculating of acid site ratio of Cr(VI)-TS-1-B1 from peak fitting program

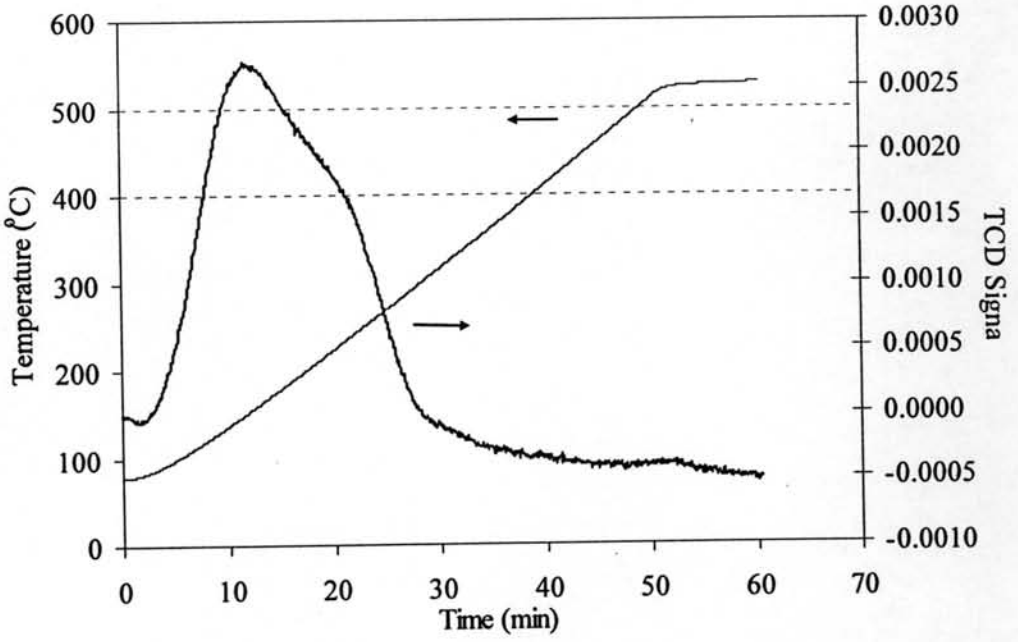

Figure E9a. Raw Data from Micromeritrics Chemisorb 2750 for Cr(VI)-TS-1-C1

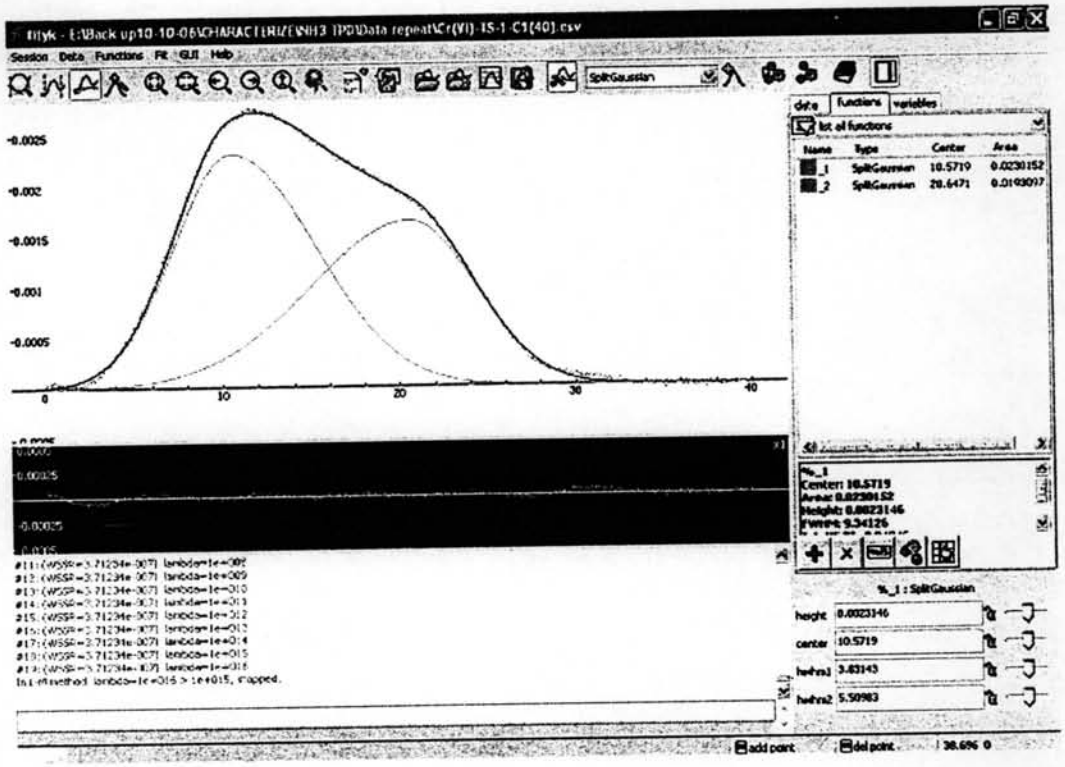

Figure E9b. Data for calculating of acid site ratio of Cr(VI)-TS-1-C1 from peak fitting program

APPENDIX F

DATA AND CALCULATION OF ATOMIC RATIO

1. Calculation of atomic ratio of Si/Ti and Si/Cr by mole

For example, Cr(III)-TS-1-A1, atomic ratio of Si/Ti and Si/Cr is calculated from the following step.

Data from analysis by X-ray fluorescence spectroscopy (XRF) is presented in figureF1.

SiO_2 = 99.30 wt%

TiO_2 = 0.48 wt%

Cr_2O_3 = 1303 ppm

Molecular weight of Si = 28.0855

Molecular weight of Ti = 204.383

Molecular weight of Cr = 51.996

Molecular weight of SiO_2 = 60.0843

Molecular weight of TiO_2 = 236.3818

Molecular weight of Cr_2O_3 = 151.9902

1.1 Calculation of mole of SiO_2

mole of SiO_2 = g/MW

= 99.30/60.0843

= 1.6527 mol

1.2 Calculation of mole of Si

SiO_2 1 mol, Si = 1 mol

SiO_2 1.6527 mol, Si = 1×1.6527/1

= 1.6527 mol

1.3 Calculation of mole of TiO_2

mole of TiO_2 = g/MW

= 0.48/236.3818

= 0.0020306 mol

1.4 Calculation of mole of Ti

TiO_2 1 mol, Ti = 1 mol

TiO_2 0.0020306 mol, Ti = 1×0.0020306 /1

= 0.0020306 mol

1.5 Calculation of mole of Cr_2O_3

Cr_2O_3 1303 ppm meaning Cr_2O_3 1303×10^{-6} part

If Cr_2O_3 100 part = $1303\times10^{-6}\times100$

= 1303×10^{-4}

mole of Cr_2O_3 = g/MW

= $1303\times10^{-4}/151.9902$

= 0.0008573 mol

1.6 Calculation of mole of Cr

Cr_2O_3 1 mol, Cr = 2 mol

Cr_2O_3 0.0008573 mol, Cr = 2×0.0008573 /1

= 0.0017146 mol

1.7 Calculation of atomic ratio of Si/Ti and Si/Cr

Si/Ti = 1.6527/0.0020306

= 814

Si/Cr = 1.6527/0.0017146

= 964

Sample: Sample494067-No.4
Thu 12/07/2006 at 11:13:03 AM
Method Name: Method494067

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.2	89.3	100.01
2	30	-0.9	121.3	100.01
3	30	-1.9	122.5	100.01
4	30	-0.5	89.3	100.01

Total: 67.89% (Normalised to 100%)

El Line	Concentration
K2O K	123 ppm
CaO K	169 ppm
TiO2K	0.48 Wt %
Cr2O3Ka	1303 ppm
Fe2O3Ka	258 ppm
Cu2OKa	132 ppm
ZnO Ka	96 ppm
Br Ka	39 ppm
ZrO2Ka	25 ppm
MoO2Ka	0.00 Wt %
SiO2K	99.30 Wt %

Figure F1. Data of Cr(III)-TS-1-A1 sample from X-ray fluorescence spectroscopy

Sample: Sample494279No.6
Wed 12/20/2006 at 2:23:17 PM
Method Name: Method494279-SetSTD

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.1	100.01
2	30	-1.0	121.2	100.01
3	30	-2.0	122.5	100.01
4	30	-0.5	89.3	100.01

Total: 91.23% Factor: 1.096130

El Line	Concentration
SiO2K	90.59 Wt %
CaO K	87 ppm
TiO2K	0.61 Wt %
Cr2O3Ka	0.01 wt %
Fe2O3Ka	67 ppm
Cu2OKa	27 ppm
ZnO Ka	26 ppm
ZrO2Ka	27 ppm

Figure F2. Data of Cr(III)-TS-1-A1A2 sample from X-ray fluorescence spectroscopy

Sample: Sample494067-No.1
Thu 12/07/2006 at 10:50:47 AM
Method Name: Method494067

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.1	100.01
2	30	-0.9	121.1	100.01
3	30	-1.8	122.3	100.01
4	30	-0.5	89.3	100.01

Total: 67.16% (Normalised to 100%)

El	Line	Concentration	
K2O	K	200	ppm
CaO	K	188	ppm
TiO2	K	1.10	Wt %
Cr2O3	Ka	33	ppm
Fe2O3	Ka	133	ppm
Cu2O	Ka	60	ppm
ZnO	Ka	49	ppm
Br	Ka	4	ppm
ZrO2	Ka	37	ppm
MoO2	Ka	0.00	Wt %
SiO2	K	98.83	Wt %

Figure F3. Data of Cr(III)-TS-1-A2 sample from X-ray fluorescence spectroscopy

Sample: Sample494067-No.6
Thu 12/07/2006 at 11:28:10 AM
Method Name: Method494067

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.1	100.01
2	30	-0.9	121.2	100.01
3	30	-1.9	122.4	100.01
4	30	-0.5	89.3	100.01

Total: 65.66% (Normalised to 100%)

El Line	Concentration
K2O K	100 ppm
CaO K	351 ppm
TiO2K	0.92 Wt %
Cr2O3Ka	0.40 Wt %
Fe2O3Ka	151 ppm
Cu2OKa	81 ppm
ZnO Ka	54 ppm
Br Ka	15 ppm
ZrO2Ka	79 ppm
MoO2Ka	0.00 Wt %
SiO2K	98.61 Wt %

Figure F4. Data of Cr(III)-TS-1-B1 sample from X-ray fluorescence spectroscopy

Sample: Sample494067-No.5
Thu 12/07/2006 at 11:20:42 AM
Method Name: Method494067

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.2	89.1	100.01
2	30	-1.0	121.2	100.01
3	30	-1.9	122.5	100.01
4	30	-0.5	89.4	100.01

Total: 67.48% (Normalised to 100%)

El	Line	Concentration
K2O	K	156 ppm
CaO	K	159 ppm
TiO2	K	1.02 Wt %
Cr2O3	Ka	0.29 Wt %
Fe2O3	Ka	125 ppm
Cu2O	Ka	43 ppm
ZnO	Ka	54 ppm
Br	Ka	8 ppm
ZrO2	Ka	51 ppm
MoO2	Ka	0.00 Wt %
SiO2	K	98.62 Wt %

Figure F5. Data of Cr(III)-TS-1-C1 sample from X-ray fluorescence spectroscopy

Sample: Sample494067-No.3
Thu 12/07/2006 at 11:05:34 AM
Method Name: Method494067

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.2	100.01
2	30	-0.9	121.2	100.01
3	30	-1.9	122.4	100.01
4	30	-0.4	89.3	100.01

Total: 65.58% (Normalised to 100%)

El Line	Concentration
K2O K	89 ppm
CaO K	203 ppm
TiO2K	1.04 Wt %
Cr2O3Ka	362 ppm
Fe2O3Ka	97 ppm
Cu2OKa	50 ppm
ZnO Ka	48 ppm
Br Ka	25 ppm
ZrO2Ka	49 ppm
MoO2Ka	0.00 Wt %
SiO2K	98.87 Wt %

Figure F6. Data of Cr(VI)-TS-1-A1 sample from X-ray fluorescence spectroscopy

Sample: Sample494067-No.2
Thu 12/07/2006 at 10:58:13 AM
Method Name: Method494067

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.2	100.01
2	30	-0.9	121.2	100.01
3	30	-1.6	122.3	100.01
4	30	-0.5	89.3	100.01

Total: 65.85% (Normalised to 100%)

El	Line	Concentration
K2O	K	139 ppm
CaO	K	191 ppm
TiO2	K	1.26 Wt %
Cr2O3	Ka	302 ppm
Fe2O3	Ka	126 ppm
Cu2O	Ka	61 ppm
ZnO	Ka	48 ppm
Br	Ka	33 ppm
ZrO2	Ka	48 ppm
MoO2	Ka	0.00 Wt %
SiO2	K	98.65 Wt %

Figure F7. Data of Cr(VI)-TS-1-A1A2 sample from X-ray fluorescence spectroscopy

Sample: Sample494279No.1
Wed 12/20/2006 at 1:44:35 PM
Method Name: Method494279-SetSTD

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.1	100.01
2	30	-0.9	121.0	100.01
3	30	-1.9	122.3	100.01
4	30	-0.5	89.0	100.01

Total: 88.66% Factor: 1.127904

El Line	Concentration
SiO2K	87.97 Wt %
CaO K	136 ppm
TiO2K	0.66 Wt %
Fe2O3Ka	95 ppm
Cu2OKa	32 ppm
ZnO Ka	26 ppm
ZrO2Ka	39 ppm

Figure F8. Data of Cr(VI)-TS-1-B1 sample from X-ray fluorescence spectroscopy

Sample: Sample494279No.2
Wed 12/20/2006 at 1:53:34 PM
Method Name: Method494279-SetSTD

FCD	Name
1	Liquids A (S,Cl)
2	Medium Elmts - Liquids
3	V. Heavy Elmts - Liquids
4	V. Light Elmts - Liquids

FCD	LT,s	Zero	FWHM	Gain
1	30	-0.1	89.1	100.01
2	30	-0.9	121.0	100.01
3	30	-1.9	122.2	100.01
4	30	-0.5	89.2	100.01

Total: 90.24% Factor: 1.108156

El Line	Concentration
SiO2K	89.49 Wt %
CaO K	131 ppm
TiO2K	0.72 Wt %
Fe2O3Ka	45 ppm
Cu2OKa	30 ppm
ZnO Ka	25 ppm
ZrO2Ka	26 ppm

Figure F9. Data of Cr(VI)-TS-1-C1 sample from X-ray fluorescence spectroscopy

APPENDIX G

MATERIAL SAFETY DATA SHEET

Chromiumnitrate nanohydrate

Safety data for Chromiumnitrate nanohydrate

General

Synonyms: Chromium (III) Nitrate, nonahydrate; Nitric acid,Chromium (3+) salt, nonahydrate

Chemical formula: $Cr(NO_3)_3.9H_2O$

Physical data

Boiling point: 212°F decomposes

Specific Gravity: 1.85 (Water = 1)

Solubility: Soluble in water

Stability and reactivity

Chemical Stability: Stable under normal temperatures and pressures.

Conditions to Avoid: Incompatible materials, ignition sources, dust generation, excess heat, combustible materials, reducing agents.

Incompatibilities with Other Materials: Reducing agents.

Hazardous Decomposition Products: Nitrogen oxides, chromium dioxide.

Potential health effect

Inhalation: Causes respiratory tract irritation.

Eye Contact: May cause irritation.

Skin Contact: May cause severe irritation and possible burns.

Ingestion: Causes gastrointestinal irritation with nausea and vomiting.

Chronic: May cause methemoglobinemia.

First aid measures

Inhalation: Remove from exposure and move to fresh air immediately.

Eye Contact: Flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids.

Skin Contact: Flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes.

Ingestion: Do not induce vomiting. If victim is conscious and alert, give 2-4 cupfuls of milk or water.

Fire fighting measures

As in any fire, wear a self-contained breathing apparatus in pressure-demand. During a fire, irritating and highly toxic gases may be generated by thermal decomposition or combustion.

Chromium (VI) oxide

Safety data for Chromium (VI) oxide

General

Synonyms: Chromium (VI) oxide (1:3); chromic acid, solid; chromic Anhydride

Chemical formula: CrO_3

Physical data

Boiling point: Decomposes on melting

Melting point: 197°C

Specific Gravity: 2.7

Solubility: 63g/100g water @ 20°C

Stability and reactivity

Stability: Stable under ordinary conditions of use and storage.

Hazardous Decomposition Products: Burning may produce chrome oxides.

Hazardous Polymerization: Will not occur.

Potential health effect

Inhalation: Extremely destructive to tissues of the mucous membranes and upper respiratory tract.

Eye Contact: Contact can cause blurred vision, redness, pain and severe tissue burns.

Skin Contact: Symptoms of redness, pain, and severe burn can occur.

Ingestion: Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death.

First aid measures

Inhalation: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

Ingestion: If swallowed, do not induce vomiting. Give large quantities of water. Never give anything by mouth to an unconscious person.

Skin Contact: Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes.

Eye Contact: Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally.

Fire fighting measures

Fire:

Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Will ignite on contact with acetic acid and alcohol. Releases oxygen upon decomposition, increasing the fire hazard.

Explosion:

Contact with oxidizable substances may cause extremely violent combustion. Containers may explode when involved in a fire.

Fire Extinguishing Media:

Use water, however, the decomposing material will form a hot viscous foam and caution should be exercised against the possibility of a steam explosion.

2-Propanol

Safety data for 2-Propanol

General

Synonyms: Isopropanol, Isopropyl alcohol

Molecular formula: C_3H_8O

Chemical formula: $CH_3CH(OH)CH_3$

Physical data

Melting point: -89.5 °C

Boiling point: 82.4 °C

Ignition temperature: 425 °C

Flash point: 12 °C

Explosion limits: 2 % - 12.7 %

Vapor pressure: 31.68 mm (14.7 °C)

Relative vapor density: 2.07

Density: 0.786 g/cm^3 (20 °C)

Solubility in water: soluble (20 °C)

Stability

Conditions to be avoided: Strong heating.

Substances to be avoided: alkali metals, alkaline earth metals, aluminium in powder form, oxidizing agent, organic nitro compounds, aldehydes, amines, fuming sulfuric acid, phosgene.

Hazardous decomposition products: no information available.

Further information: highly inflammable; hygroscopic, explosive with air in a vapor/gas state.

Toxicology

Further toxicological information

After inhalation: Irritation symptoms in the respiratory tract Drowsiness

After skin contact: degreasing effect on the skin possibly followed by secondary inflammation.

After eye contact: Irritations.

After swallowing: after accidental swallowing the substance may pose a risk of aspiration. Passage into the lung can result in a condition resembling pneumonia.

After absorption: headache, dizziness, inebriation

After uptake of large quantities: respiratory paralysis, coma.

Personal protection

Personal protective equipment: Protective clothing should be selected specifically for the working place, depending on concentration and quantity of the hazardous substances handled. The resistance of the protective clothing to chemicals should be ascertained with the respective supplier.

Industrial hygiene: Change contaminated clothing. Application of Skin-protective barrier cream recommended. Should be wash hands after working with substance.

Sodium chloride

Safety data for Sodium chloride

General

Synonyms: extra fine 200 salt, extra fine 325 salt, H.G. blending, salt, sea salt, table salt, common salt, dendritis, rock salt, top flake, white crystal, saline, halite, purex, USP sodium chloride

Molecular formula: NaCl

Physical data

Appearance: colourless crystals or white powder

Melting point: 804 °C

Boiling point: 1413 °C

Vapour pressure: 1 mm Hg at 865 °C

Specific gravity: 2.16 g cm^{-3}

Solubility in water: 35.7 g/100g at 0 °C

Stability

Stable. Incompatible with strong oxidizing agents.

Toxicology

May cause skin, eye or respiratory irritation.

Personal protection

Not believed to present a significant hazard to health.

Sodium hydroxide

Safety data for Sodium hydroxide

General

Synonyms: caustic soda, soda lye, lye, white caustic, aetznatron, ascarite, Collo-Grillrein, Collo-Tapetta, sodium hydrate, fotofoil etchant, NAOH, STCC 4935235, sodium hydroxide pellets, Lewis red devil lye

Molecular formula: NaOH

Physical data

Appearance: odourless white solid (often sold as pellets)

Melting point: 318 °C

Boiling point: 1390 °C

Vapour pressure: 1 mm Hg at 739 °C

Specific gravity: 2.12

Water solubility: High (Note: dissolution in water is highly exothermic)

Stability

Stable. Incompatible with a wide variety of materials including many metals, ammonium compounds, cyanides, acids, nitro compounds, phenols, combustible organics. Hygroscopic. Heat of solution is very high and may lead to a dangerously hot solution if small amounts of water are used. Absorbs carbon dioxide from the air.

Toxicology

Very corrosive. Causes severe burns. May cause serious permanent eye damage. Very harmful by ingestion. Harmful by skin contact or by inhalation of dust. Typical TLV 2 mg m^{-1}.

Sodium silicate

Safety data for Sodium silicate

General

Synonyms: silicic acid sodium salt, water glass, sodium water glass, soluble glass, silicate of soda, silicon sodium oxide, sodium orthosilicate, sodium sesquisilicate, sodium silicate glass, agrosil S, barasil S, britesil, carsil 2000, chemfin 60, chemsilicate, crystal 79, crystal 96, ineos 140, inosil Na 4237, portil A, pyramid 8, vitrosol N40, ZhS 3, very large number of further trade names

Molecular formula: Na_4O_4Si

Physical data

Appearance: colourless liquid as usually supplied (solution)

Boiling point: ca. 102 °C for a 40% aqueous solution

Specific gravity: approximately 1.3 for a ca. 40% solution

Stability

Stable. Incompatible with acids, most metals, many organic materials.

Toxicology

Harmful by ingestion. Corrosive - may cause burns through skin or eye contact. Very destructive of mucous membranes.

Personal protection

Safety glasses, gloves.

Sulfuric acid

Safety data for Sulfuric acid

General

Synonyms: oil of vitriol, mattling acid, vitriol, battery acid, dipping acid, electrolyte acid, vitriol brown oil, sulphuric acid

Molecular formula: H_2SO_4

Physical data

Appearance: Colourless oily liquid

Melting point: -2 °C

Boiling point: 327 °C

Specific gravity: 1.84

Vapour pressure: <0.3 mm Hg at 20 °C (vapour density 3.4)

Water solubility: miscible in all proportions

Stability

Stable, but reacts with moisture very exothermically, which may enhance its ability to act as an oxidizing agent. Substances to be avoided include water, most common metals, organic materials, strong reducing agents, combustible materials, bases, oxidizing agents. Reacts violently with water - when diluting concentrated acid, carefully and slowly add acid to water, not the reverse. Reaction with many metals is rapid or violent, and generates hydrogen (flammable, explosion hazard).

Toxicology

Extremely corrosive, causes serious burns. Highly toxic. Harmful by inhalation, ingestion and through skin contact. Ingestion may be fatal. Skin contact can lead to extensive and severe burns. Chronic exposure may result in lung damage and possibly cancer.

Personal protection

Safety glasses or face mask; acid-resistant gloves. Suitable ventilation. In the UK use of this material must be assessed under the COSHH regulations.

Tetrapropylammonium bromide

Safety data for tetrapropylammonium bromide

General

Synonyms: 1-Propanaminium, N, N, N-tripropyl, bromide or Tetra-n-propylammonium bromide or TPBr or TPABr

Molecular formula: $C_{12}H_{28}N.Br$

Chemical formula: $(C_3H_7)_4NBr$

Physical data

Solubility in Water: 60% (20 °C)

pH: 5 - 10 for solution

Melting Point: 275-278 °C (decomposes)

Stability and reactivity

Stable at ambient temperatures. Do not expose to high temperatures. Oxidizers should be tested for compatibility before use.

Hazardous decomposition

In fire conditions: Carbon monoxide, Hydrobromic acid and Nitrogen oxides. If heated to decomposition, tripropylamine may be released.

Potential health effect

Inhalation: May cause irritation.

Eye Contact: May cause irritation.

Skin Contact: May cause irritation.

Ingestion: No toxicity or other health effects information available.

Chronic: May cause irritation. No additional information available.

First aid measures

Inhalation: Remove to fresh air. If breathing has stopped, give artificial respiration. Consult a physician.

Eye Contact: Immediately flush with water until no evidence of chemical remains (at least 15-20 minutes) and consult a physician.

Skin Contact: Immediately flush with water with sufficient volume until there is no evidence of the chemical on the affected area.

Ingestion: If person is conscious and able to swallow, have them drink a large volume of water and milk and induce vomiting. Contact a physician.

Fire fighting measures

Wear S.C.B.A. May use water spray, carbon dioxide, dry chemical or chemical foam to fight fire.

Hazardous product combustion

May emit Nitrogen oxides, Hydrobromic acid and Carbon monoxide.

Titanium (IV) n-butoxide

Safety data for titanium (IV) n-butoxide

General

Synonyms: Tetra-n-butyl titanate, TNBT, Titanium(IV) n-butoxide (TYZOR TNBT), Tetra-n-butyl orthotitanate for synthesis, titanium tetrabutanolate, Titanium(IV)n-butoxide (TYZOR TBT), Butyl Titanate, Titaniumbutoxidecolorlessliq, Titanium n-butoxide, Titanium (IV) n-butoxide, 99+%, Tetra-n-butoxytitanium(IV), Tetra-n-butyl orthotitanate, Titanium tetrabutoxide, Triethoxy methane, Titanium tetrabutylate, Orthotitanic acid tetrabutyl ester

Molecular formula: $C_{16}H_{36}O_4Ti$

Chemical formula: $Ti[O(CH_2)_3CH_3]_4$

Physical data

Boiling point: 310-314 °C

Flash point: 78 °C

Density: 1.486 g/cm^3

Toxicology

Irritating to eyes, respiratory system and skin.

Personal protection

Avoid contact with skin and eyes.

APPENDIX H

LIST OF PUBLICATION

Eakawut Poompichate, Em-orn Phromphet and Tharathon Mongkhonsi, "Preparation of chromium-titanium silicalite-1 catalyst", Proceedings of Thai Institute of Chemical Engineering and Applied Chemical Conference 16th, Bangkok, Thailand, October, 2006, Ref. No.CRE-009.

Em-orn Phromphet and Tharathon Mongkhonsi, "Synthesis methodology affecting chromium-titanium silicalite-1 catalyst", 7th National Graduate Research Conference GRAD-RESEARCH 2007, Surat Thani, Thailand, April,2007, Ref. No.O-30-Phys.

VITA

Miss Em-orn Phromphet was born on July 23th, 1981 in Songkhla, Thailand. She finished high school from Hatyaiwittayalai School, Songkhla in 2000, and received the bachelor's degree of Chemical Engineering from Faculty of Engineering, Prince of Songkla University in 2004. She continued her master's study at Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University in June, 2005.