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APPENDICES

APPENDIX A

(Scanning electron microscopy & Energy dispersive X-ray spectroscopy)

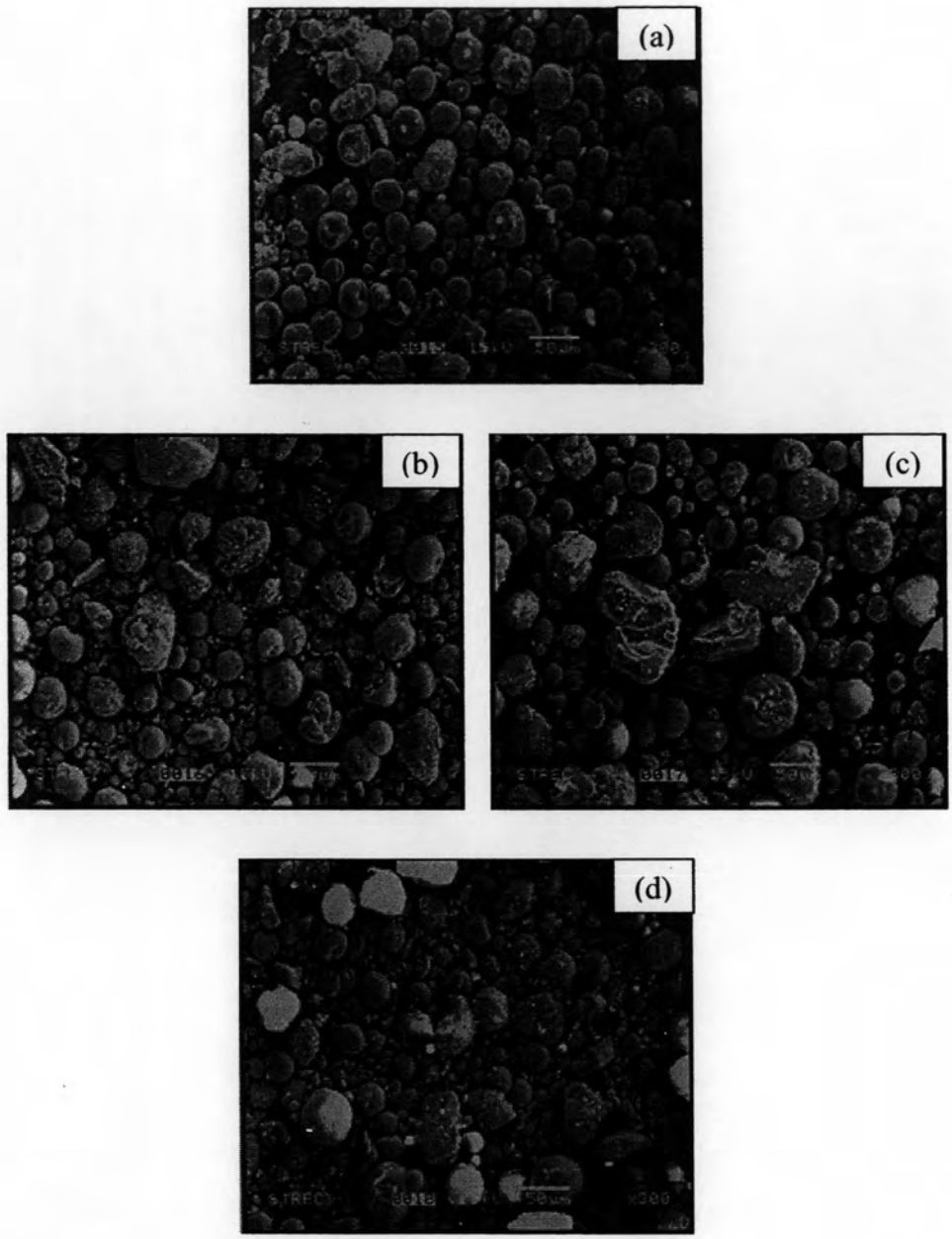

Figure A-1 SEM micrographs of various zirconia-modified supports before MAO impregnation; (a) SiO_2, (b) 1%Zr-SiO_2, (c) 2%Zr-SiO_2, (d) 5%Zr-SiO_2

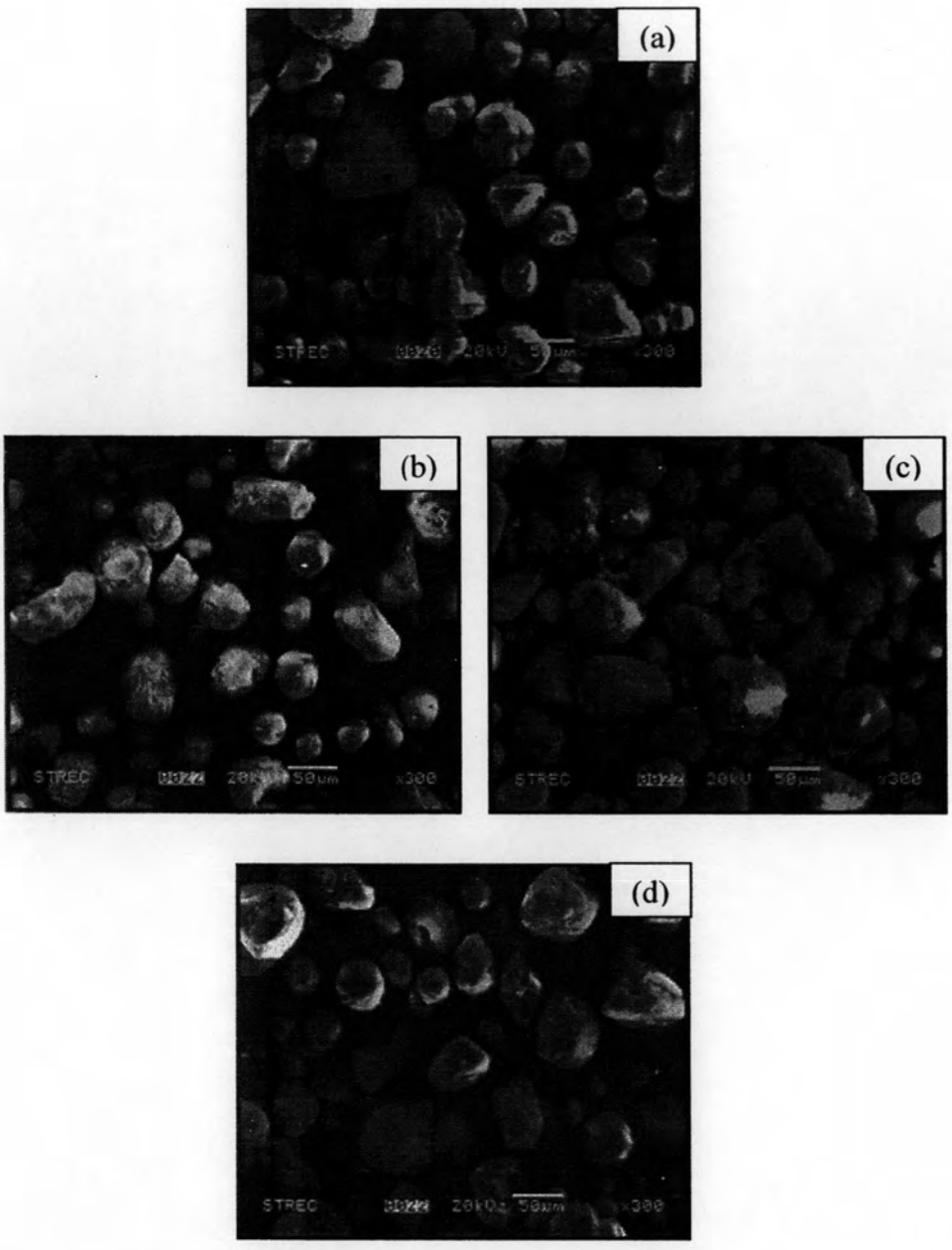

Figure A-2 SEM micrographs of various zirconia-modified supports after MAO impregnation; (a) SiO_2, (b) 1%Zr-SiO_2, (c) 2%Zr-SiO_2, (d) 5%Zr-SiO_2

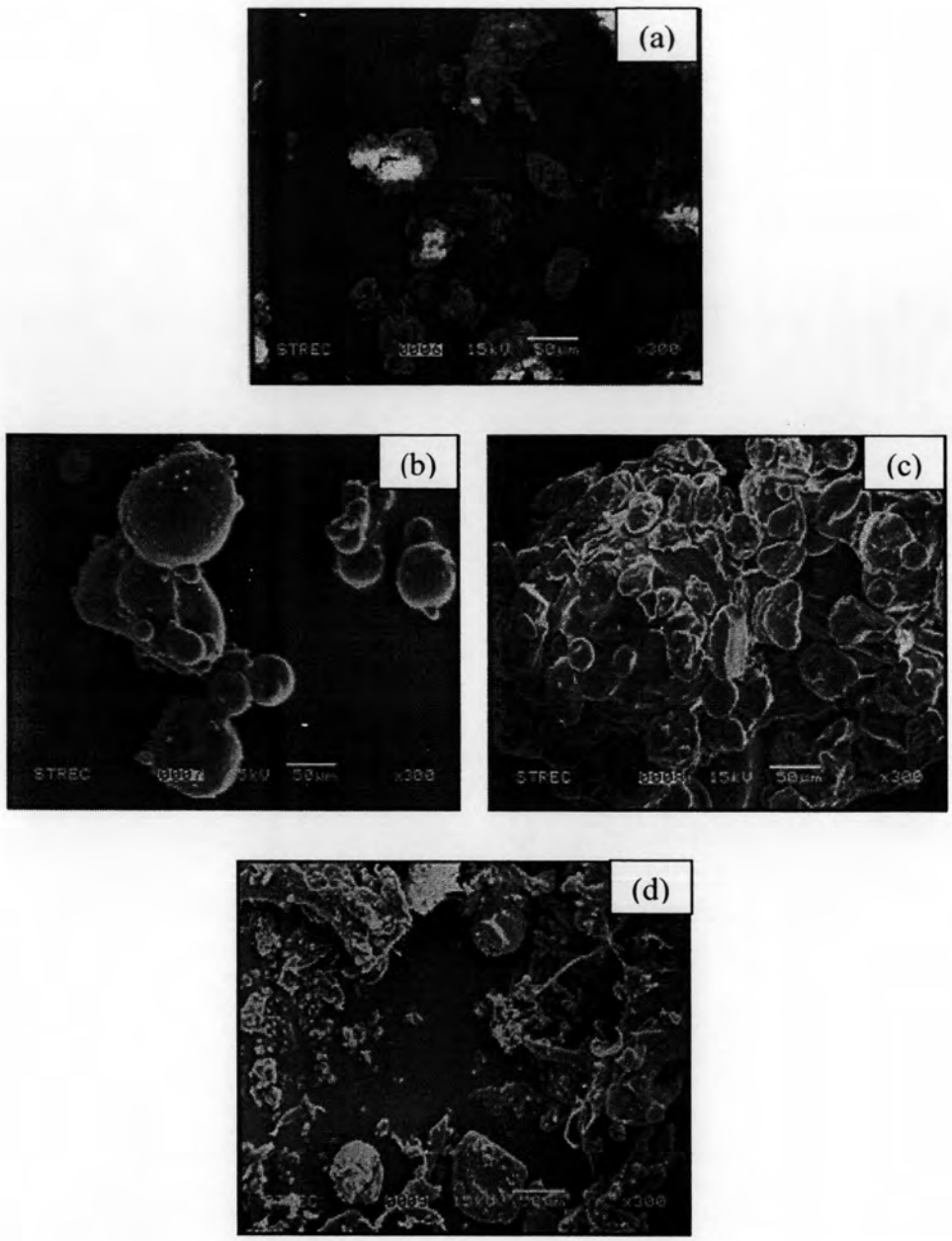

Figure A-3 SEM micrographs of ethylene/1-octene copolymers obtained with various zirconia-modified supports ; (a) SiO_2, (b) 1%Zr-SiO_2, (c) 2%Zr-SiO_2, (d) 5%Zr-SiO_2

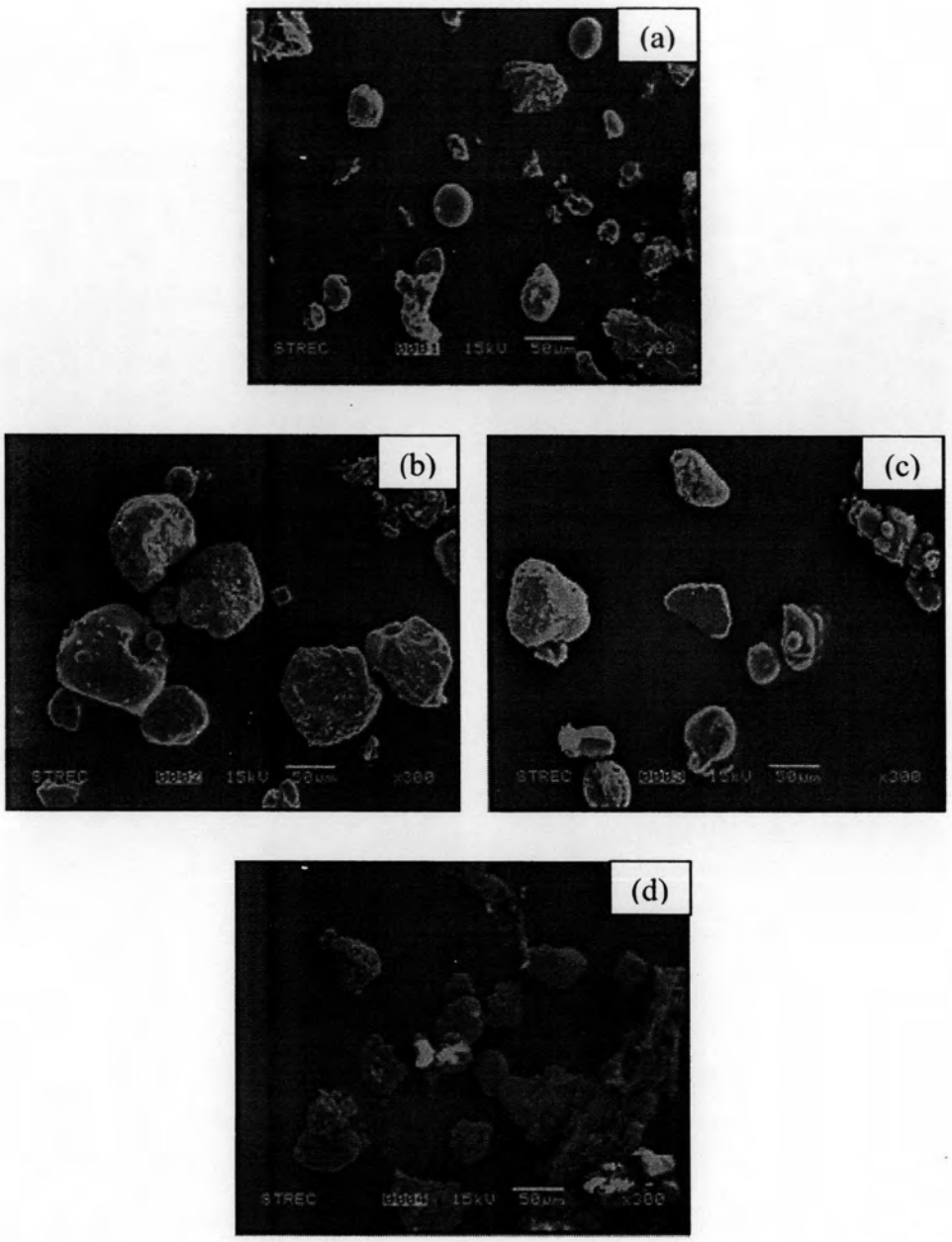

Figure A-4 SEM micrographs of ethylene/1-hexene copolymers obtained with various zirconia-modified supports ; (a) SiO_2, (b) 1%Zr-SiO_2, (c) 2%Zr-SiO_2, (d) 5%Zr-SiO_2

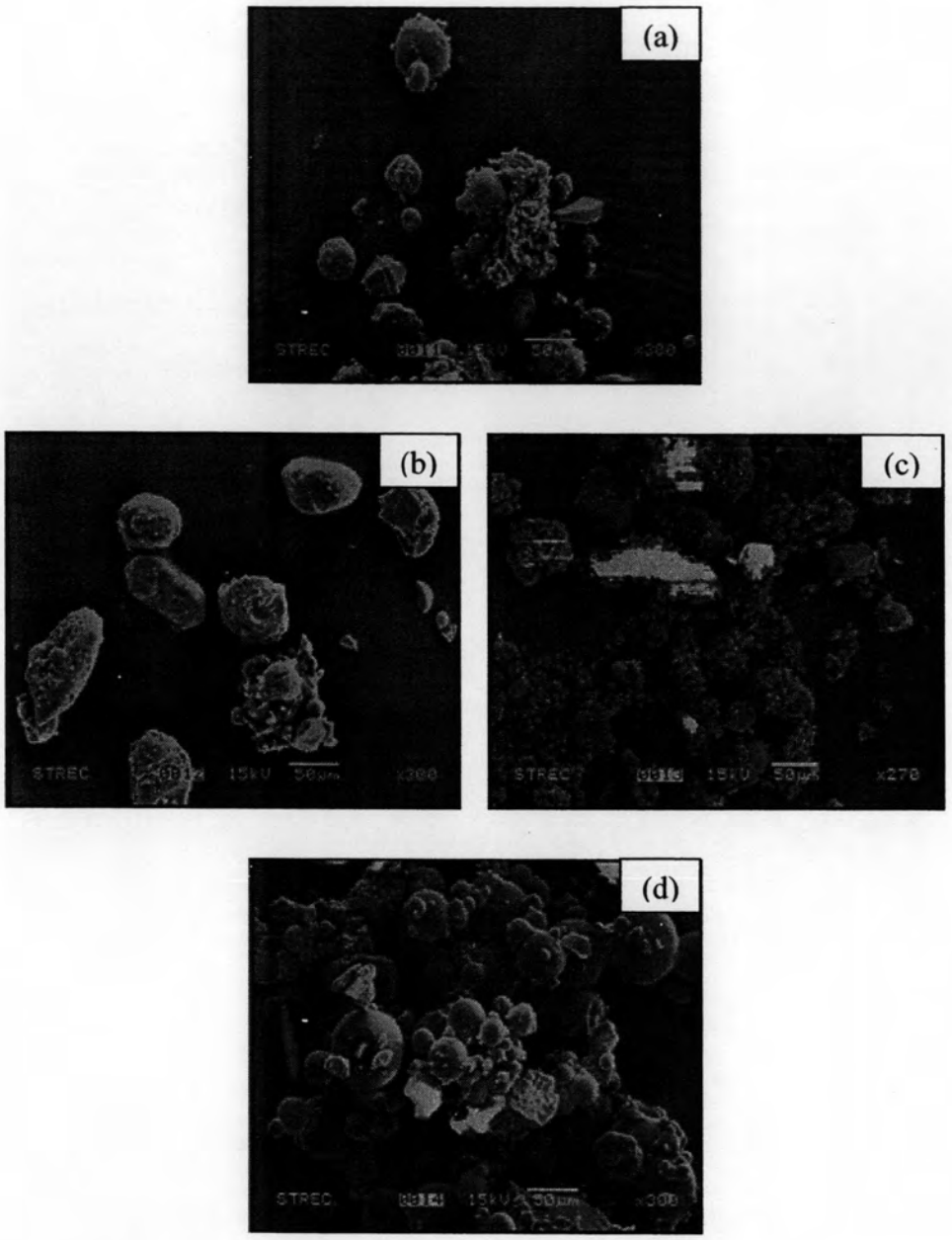

Figure A-5 SEM micrographs of ethylene/1-decene copolymers obtained with various zirconia-modified supports ; (a) SiO_2, (b) 1%Zr-SiO_2, (c) 2%Zr-SiO_2, (d) 5%Zr-SiO_2

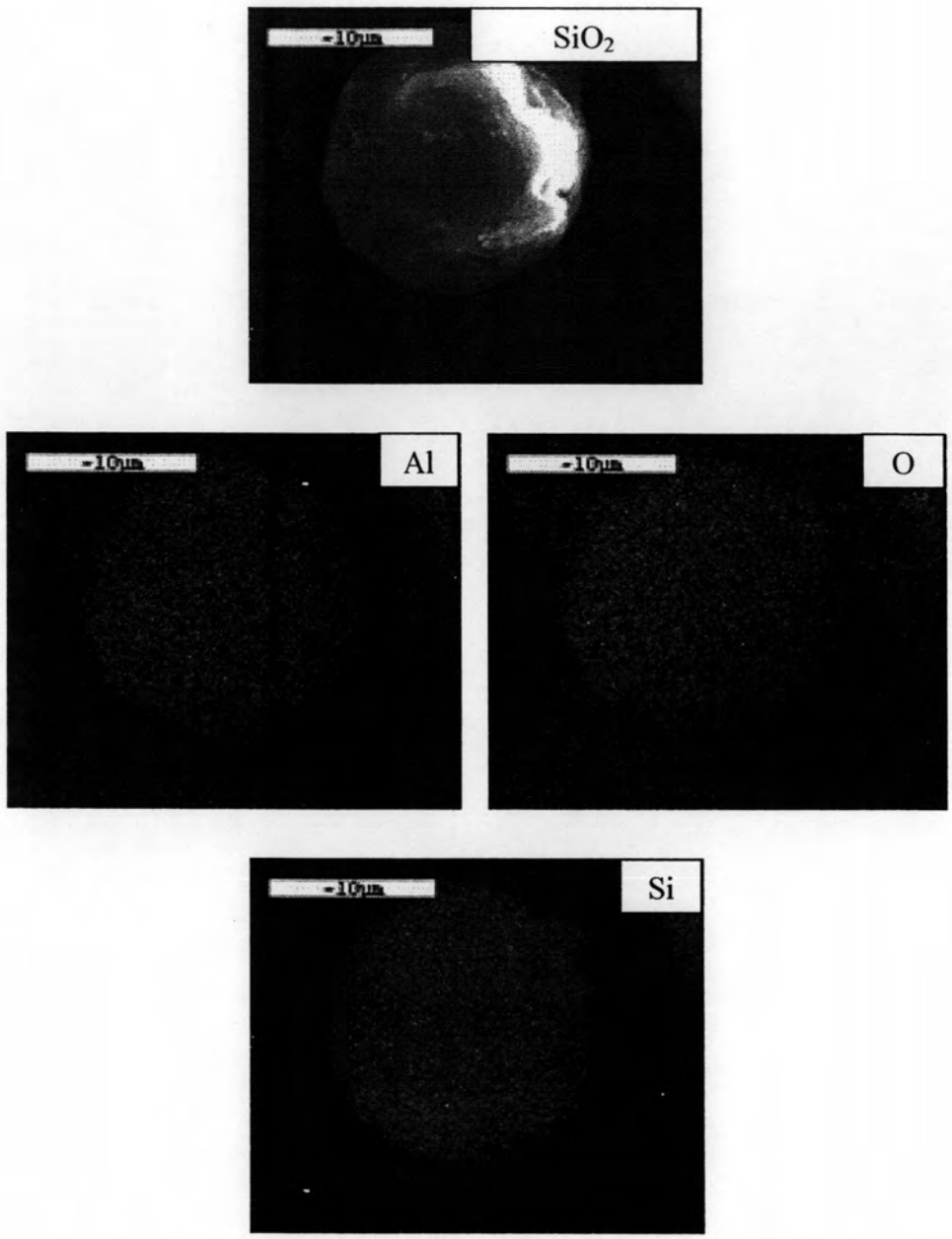

Figure A-6 EDX mapping of various zirconia-modified supports after MAO impregnation with SiO_2

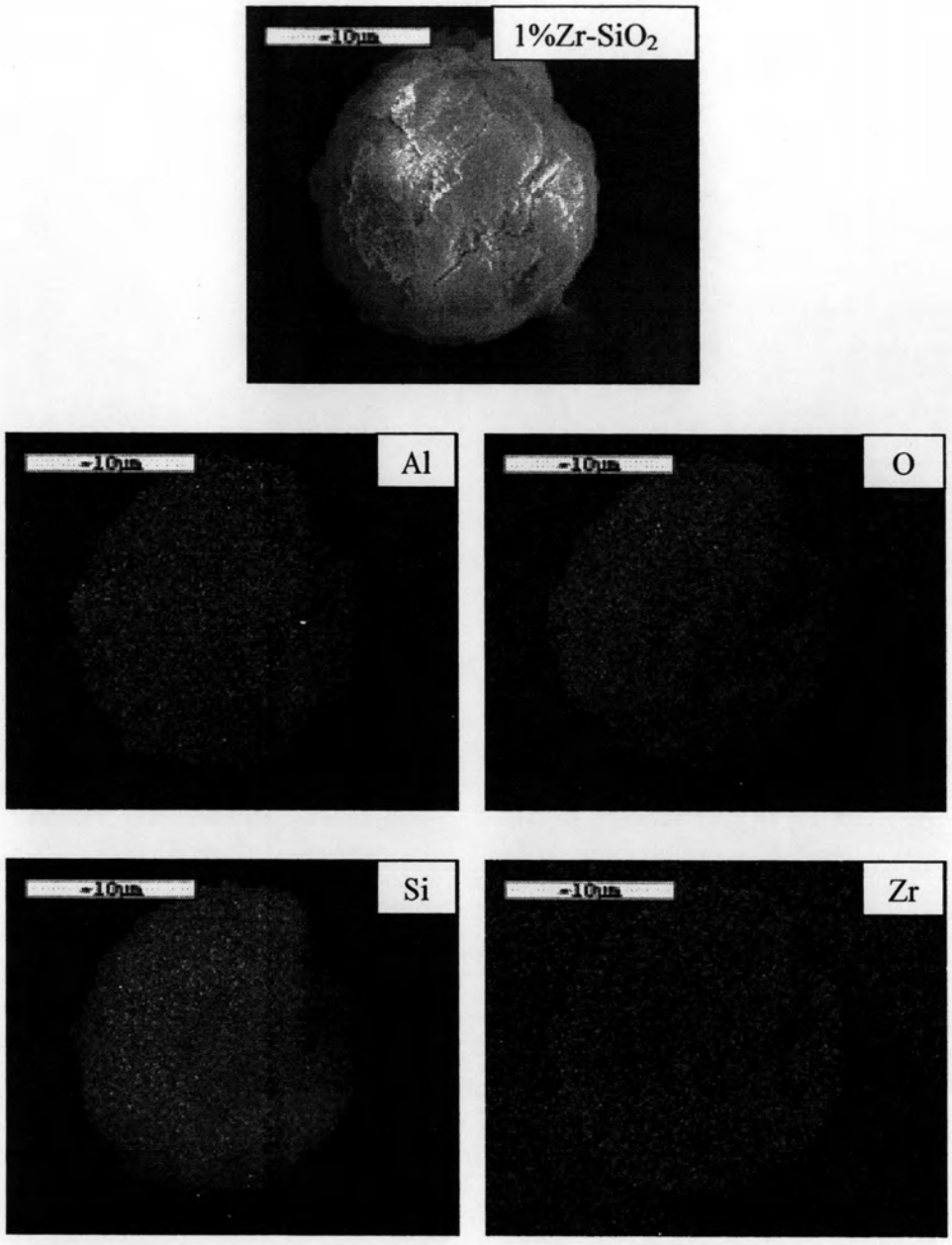

Figure A-7 EDX mapping of various zirconia-modified supports after MAO impregnation with 1%Zr-SiO_2

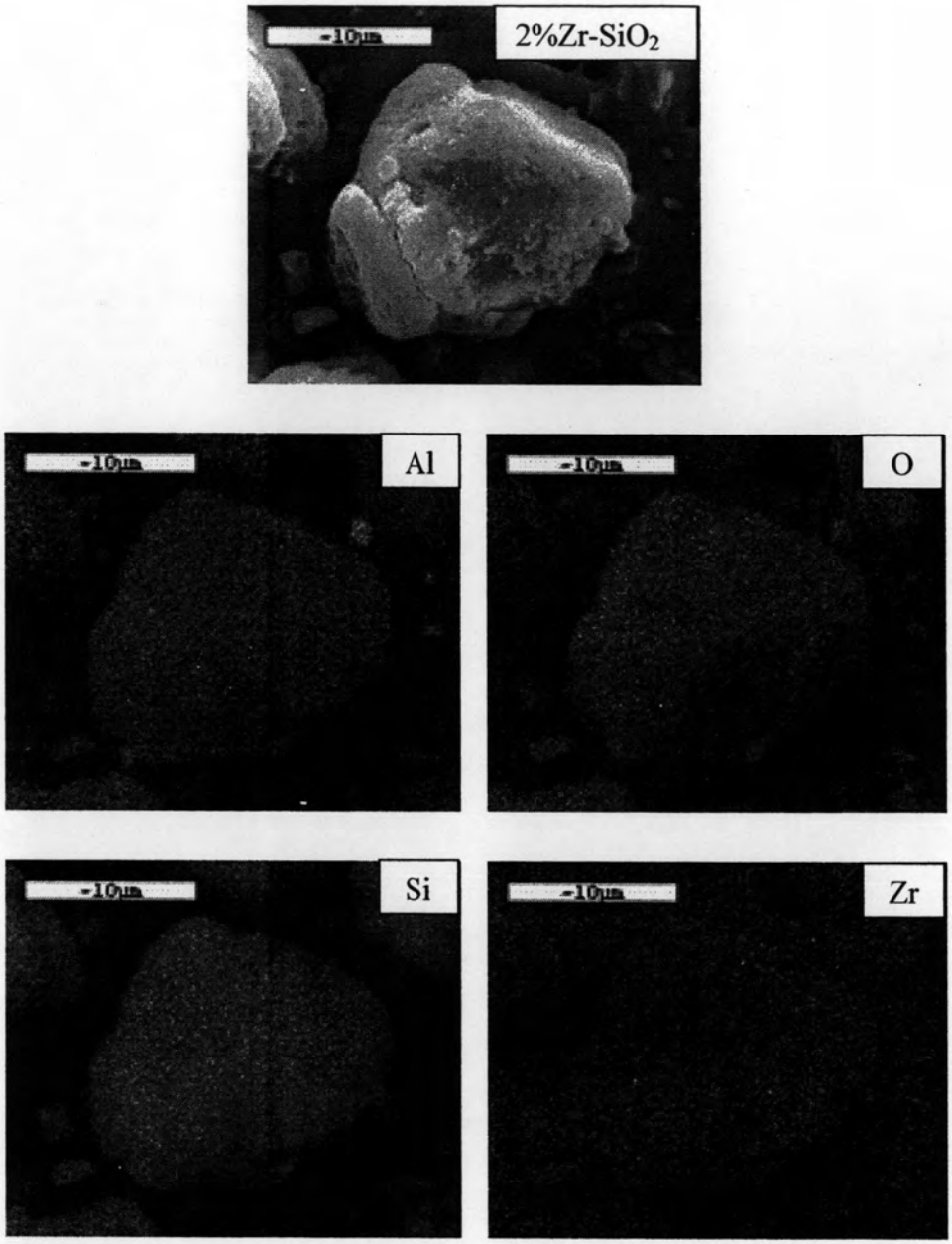

Figure A-8 EDX mapping of various zirconia-modified supports after MAO impregnation with 2%Zr-SiO_2

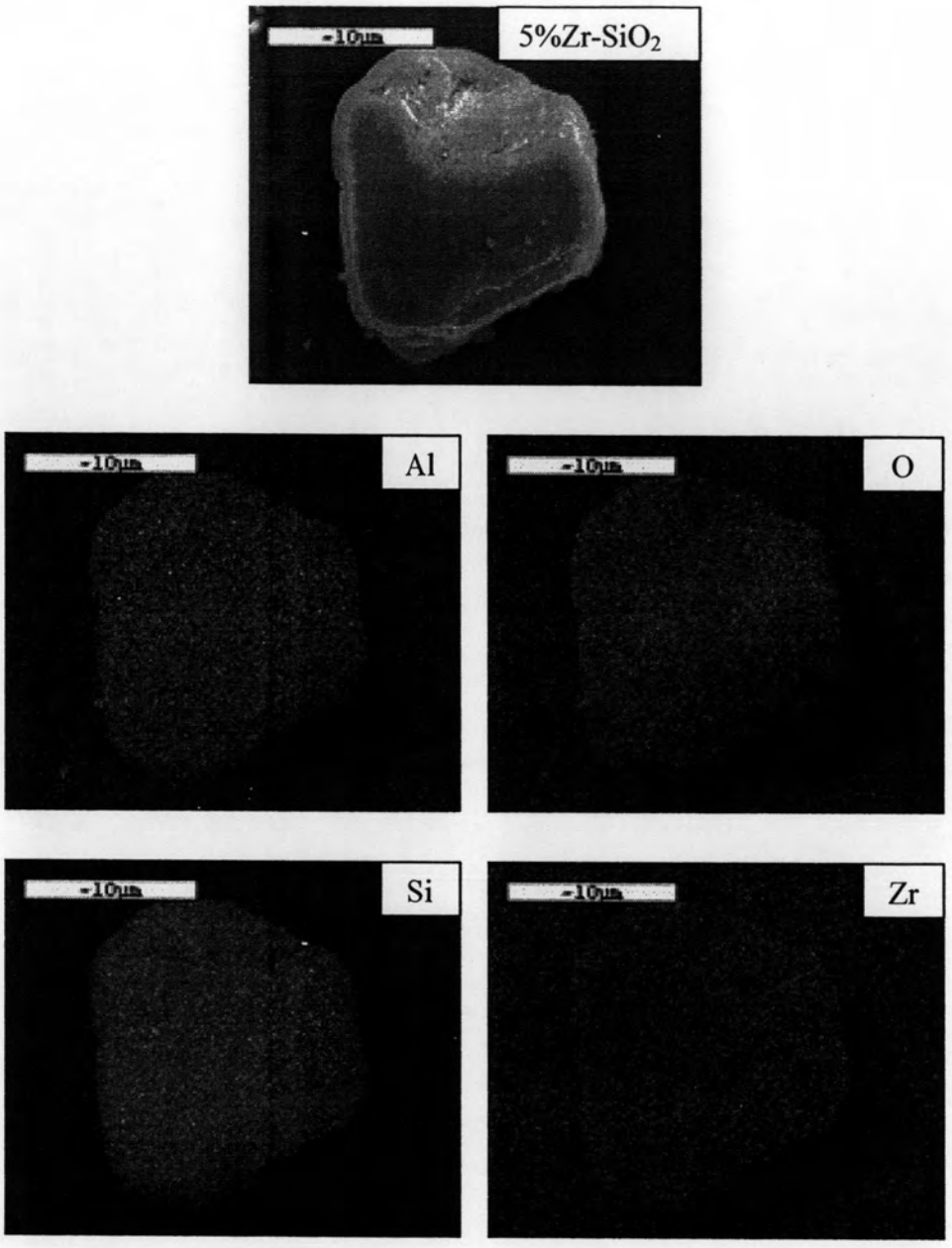

Figure A-9 EDX mapping of various zirconia-modified supports after MAO impregnation with 5%Zr-SiO_2

SEMQuant results. Listed at 9:46:17 AM on 5/14/07
Operator: sirirat
Client: none
Job: EDX
Spectrum label: silica+PMAO_2

System resolution = 69 eV

Quantitative method: ZAF (6 iterations).
Analysed all elements and normalised results.

Standards :

C	K	CaCO3 01/12/93
O	K	Quartz 01/12/93
Al	K	Al2O3 23/11/93
Si	K	Quartz 01/12/93
Cu	K	Cu 01/12/93
Zn	K	Zn 01/12/93
Zr	L	Zr 01/12/93
Au	M	Au 01/12/93

Elmt	Spect. Type	Element %	Atomic %
C K	ED	19.95	30.39
O K	ED	42.43	48.55
Al K	ED	4.93	3.34
Si K	ED	25.68	16.73
Cu K	ED	0.97	0.28
Zn K	ED	0.59	0.16
Zr L	ED	0.33	0.07
Au M	ED	5.14	0.48
Total		100.00	100.00

* = <2 Sigma

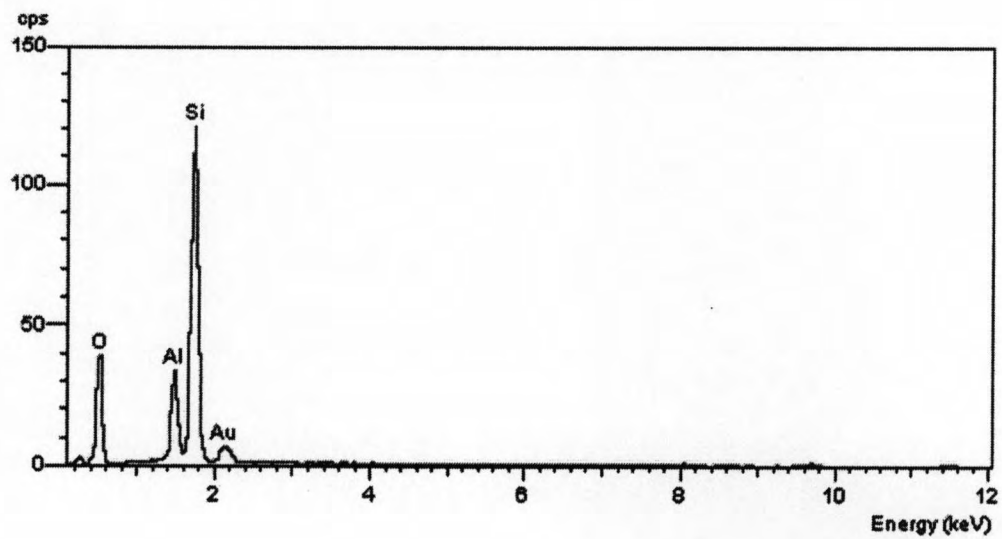

Figure A-10 EDX profile of $[Al]_{MAO}$ in catalyst precursor of SiO_2

SEMQuant results. Listed at 9:54:00 AM on 5/14/07
Operator: sirirat
Client: none
Job: EDX
Spectrum label: 1%Zr-Si+PMAO_1

System resolution = 69 eV

Quantitative method: ZAF (6 iterations).
Analysed all elements and normalised results.

Standards :

C	K	CaCO3 01/12/93
O	K	Quartz 01/12/93
Al	K	Al2O3 23/11/93
Si	K	Quartz 01/12/93
Cu	K	Cu 01/12/93
Zn	K	Zn 01/12/93
Zr	L	Zr 01/12/93
Au	M	Au 01/12/93

Elmt	Spect. Type	Element %	Atomic %
C K	ED	19.31	29.14
O K	ED	45.13	51.13
Al K	ED	6.63	4.46
Si K	ED	22.25	14.36
Cu K	ED	0.75	0.21
Zn K	ED	0.61	0.17
Zr L	ED	0.34	0.07
Au M	ED	4.98	0.46
Total		100.00	100.00

* = <2 Sigma

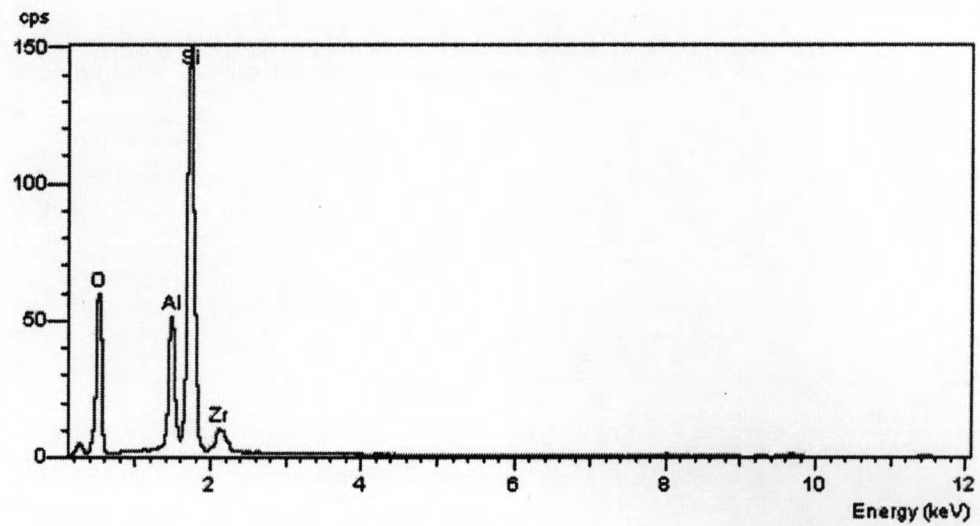

Figure A-11 EDX profile of $[Al]_{MAO}$ in catalyst precursor of 1%Zr-SiO_2

SEMQuant results. Listed at 9:52:54 AM on 5/14/07
Operator: sirirat
Client: none
Job: EDX
Spectrum label: 2%Zr-Si+PMAO_2

System resolution = 69 eV

Quantitative method: ZAF (6 iterations).
Analysed all elements and normalised results.

Standards :

C	K	CaCO3 01/12/93
O	K	Quartz 01/12/93
Al	K	Al2O3 23/11/93
Si	K	Quartz 01/12/93
Cu	K	Cu 01/12/93
Zn	K	Zn 01/12/93
Zr	L	Zr 01/12/93
Au	M	Au 01/12/93

Elmt	Spect. Type	Element %	Atomic %
C K	ED	19.75	30.29
O K	ED	41.74	48.06
Al K	ED	6.78	4.63
Si K	ED	24.51	16.08
Cu K	ED	0.63	0.18
Zn K	ED	0.60	0.17
Zr L	ED	0.28*	0.06*
Au M	ED	5.72	0.53
Total		100.00	100.00

* = <2 Sigma

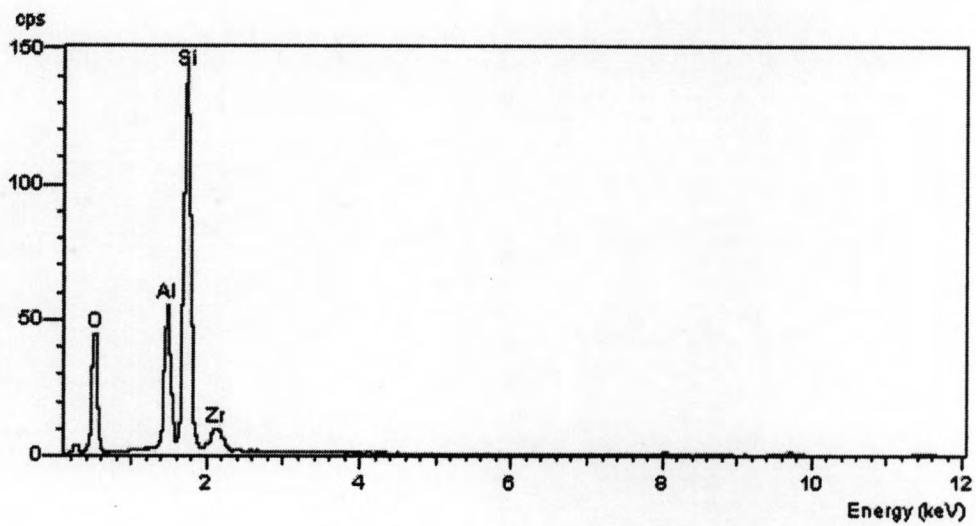

Figure A-12 EDX profile of $[Al]_{MAO}$ in catalyst precursor of 2%Zr-SiO_2

SEMQuant results. Listed at 9:49:31 AM on 5/14/07
Operator: sirirat
Client: none
Job: EDX
Spectrum label: 5%Zr-Si+PMAO_2

System resolution = 69 eV

Quantitative method: ZAF (6 iterations).
Analysed all elements and normalised results.

Standards :

C	K	CaCO3 01/12/93
O	K	Quartz 01/12/93
Al	K	Al2O3 23/11/93
Si	K	Quartz 01/12/93
Cu	K	Cu 01/12/93
Zn	K	Zn 01/12/93
Zr	L	Zr 01/12/93
Au	M	Au 01/12/93

Elmt	Spect. Type	Element %	Atomic %
C K	ED	24.61	36.76
O K	ED	39.07	43.80
Al K	ED	7.36	4.89
Si K	ED	20.82	13.30
Cu K	ED	1.32	0.37
Zn K	ED	1.13	0.31
Zr L	ED	0.47	0.09
Au M	ED	5.21	0.47
Total		100.00	100.00

* = <2 Sigma

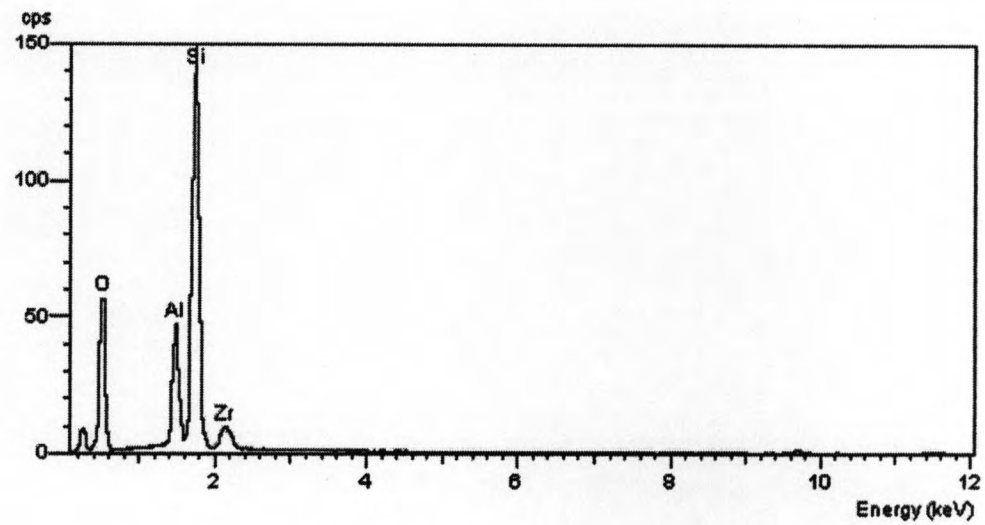

Figure A-13 EDX profile of $[Al]_{MAO}$ in catalyst precursor of 5%Zr-SiO_2

APPENDIX B
(Differential Scanning Calorimeter)

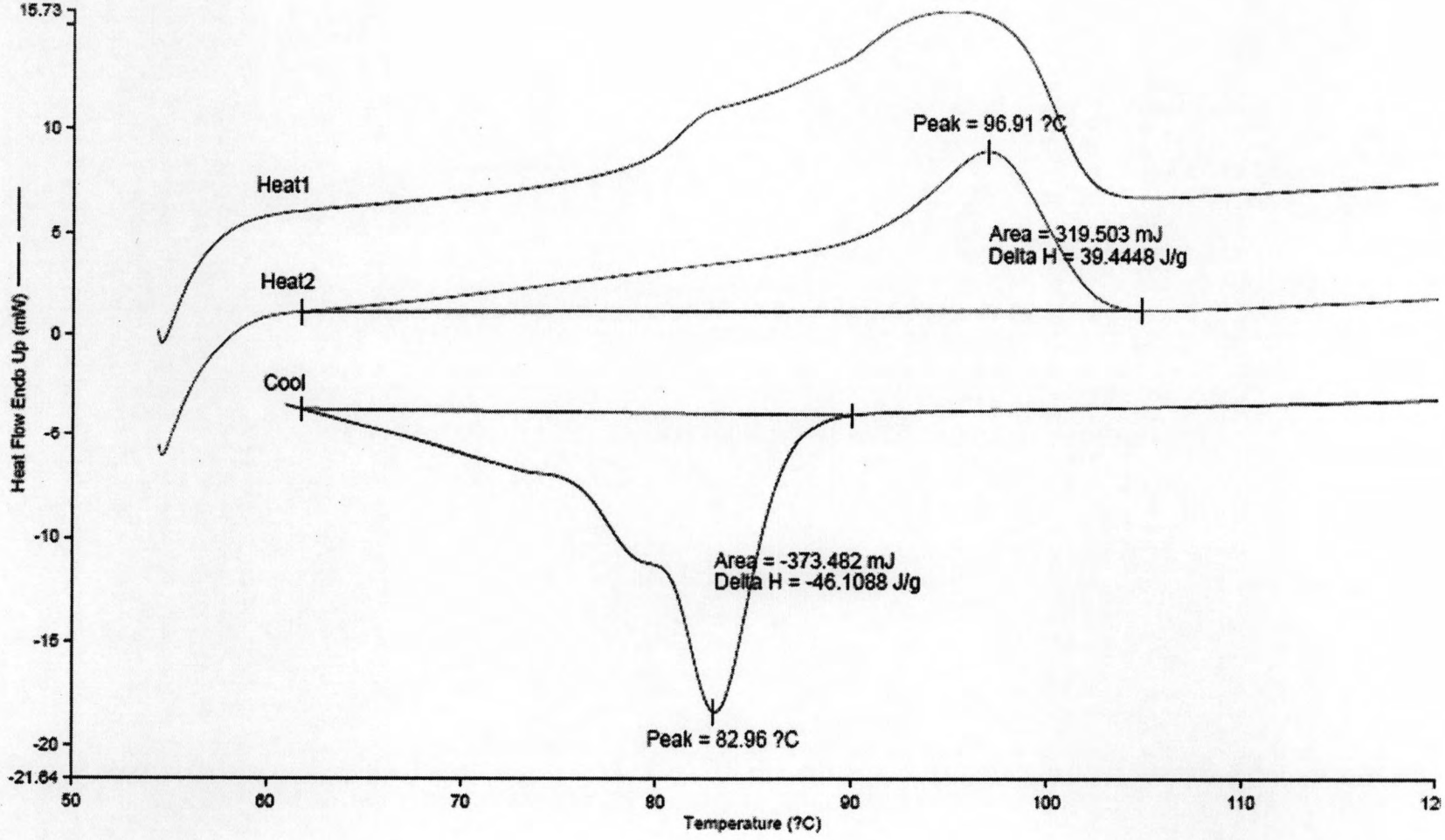

Figure B-1. DSC curve of ethylene/1-octene copolymer produce with SiO_2

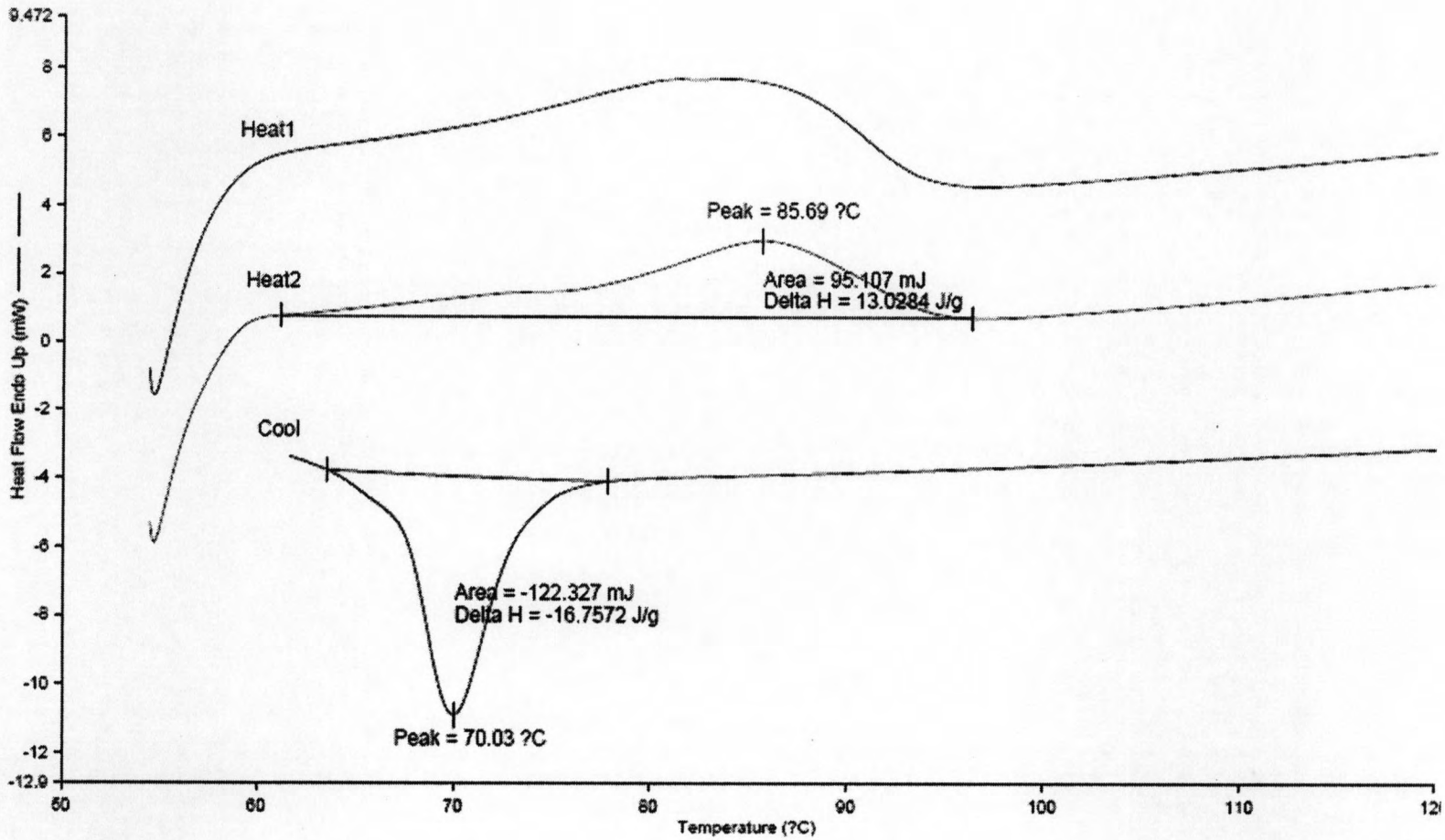

Figure B-2. DSC curve of ethylene/1-octene copolymer produce with 1%Zr-SiO_2

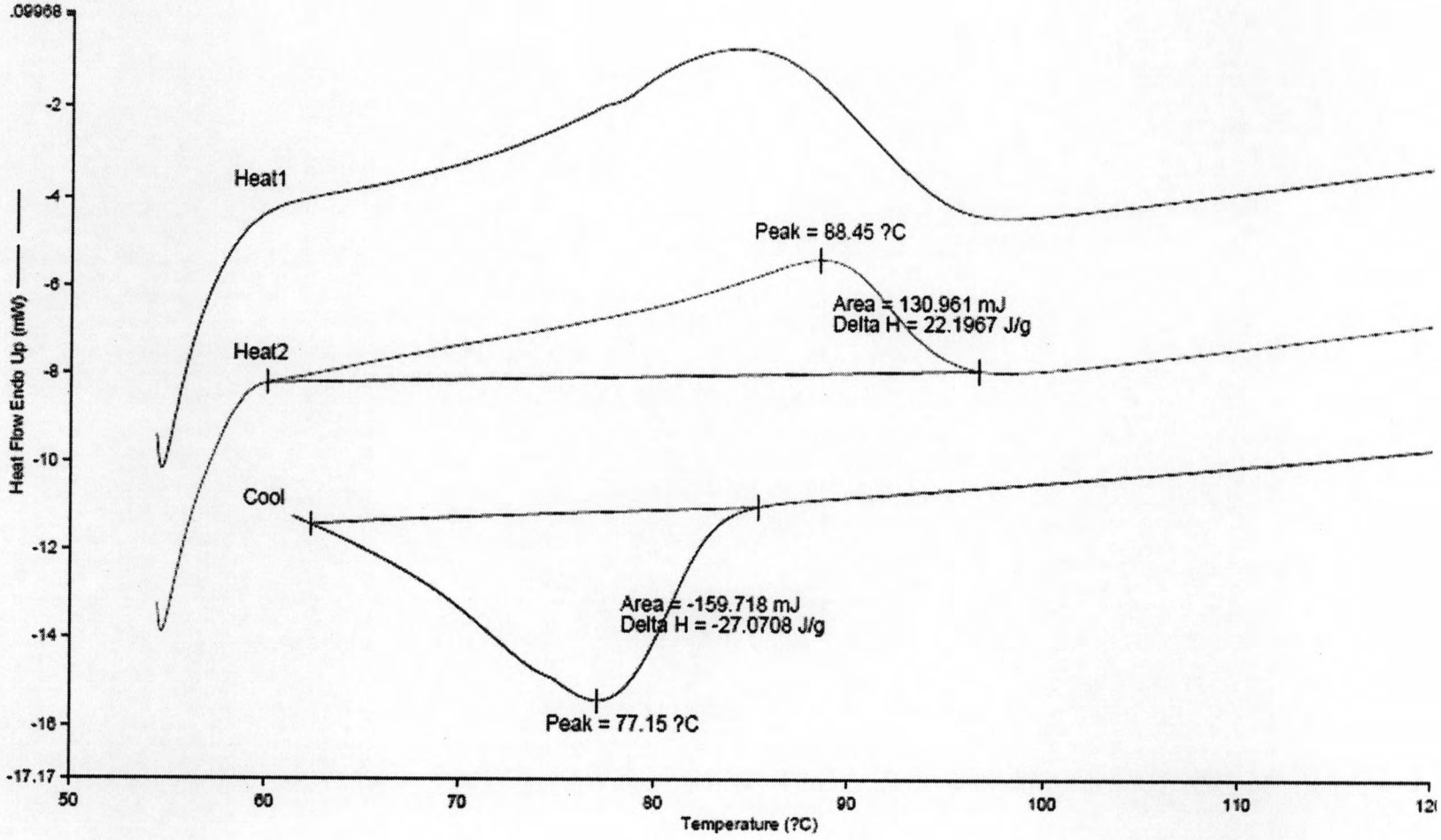

Figure B-3. DSC curve of ethylene/1-octene copolymer produce with 2%Zr-SiO_2

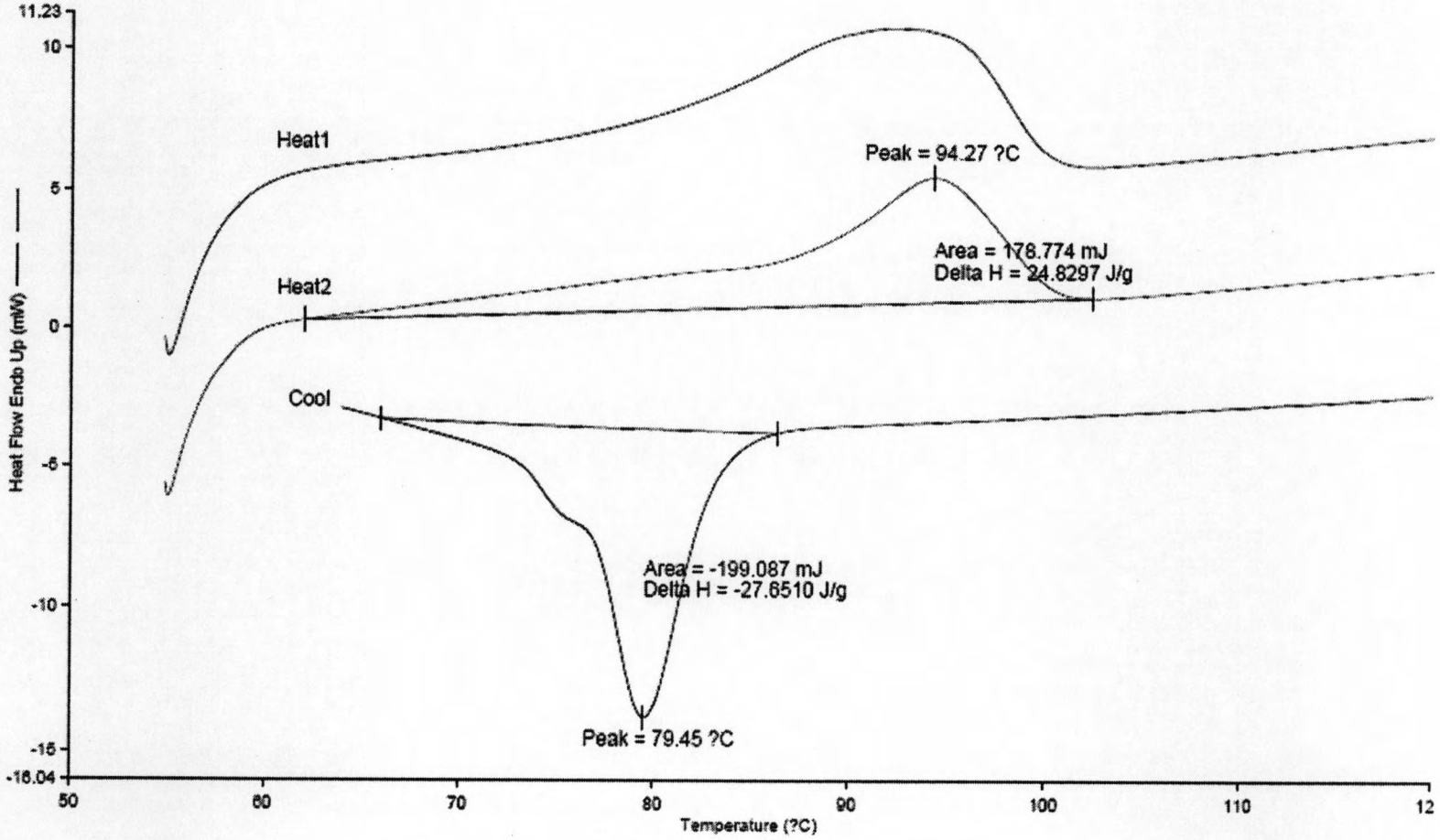

Figure B-4. DSC curve of ethylene/1-octene copolymer produce with 5%Zr-SiO_2

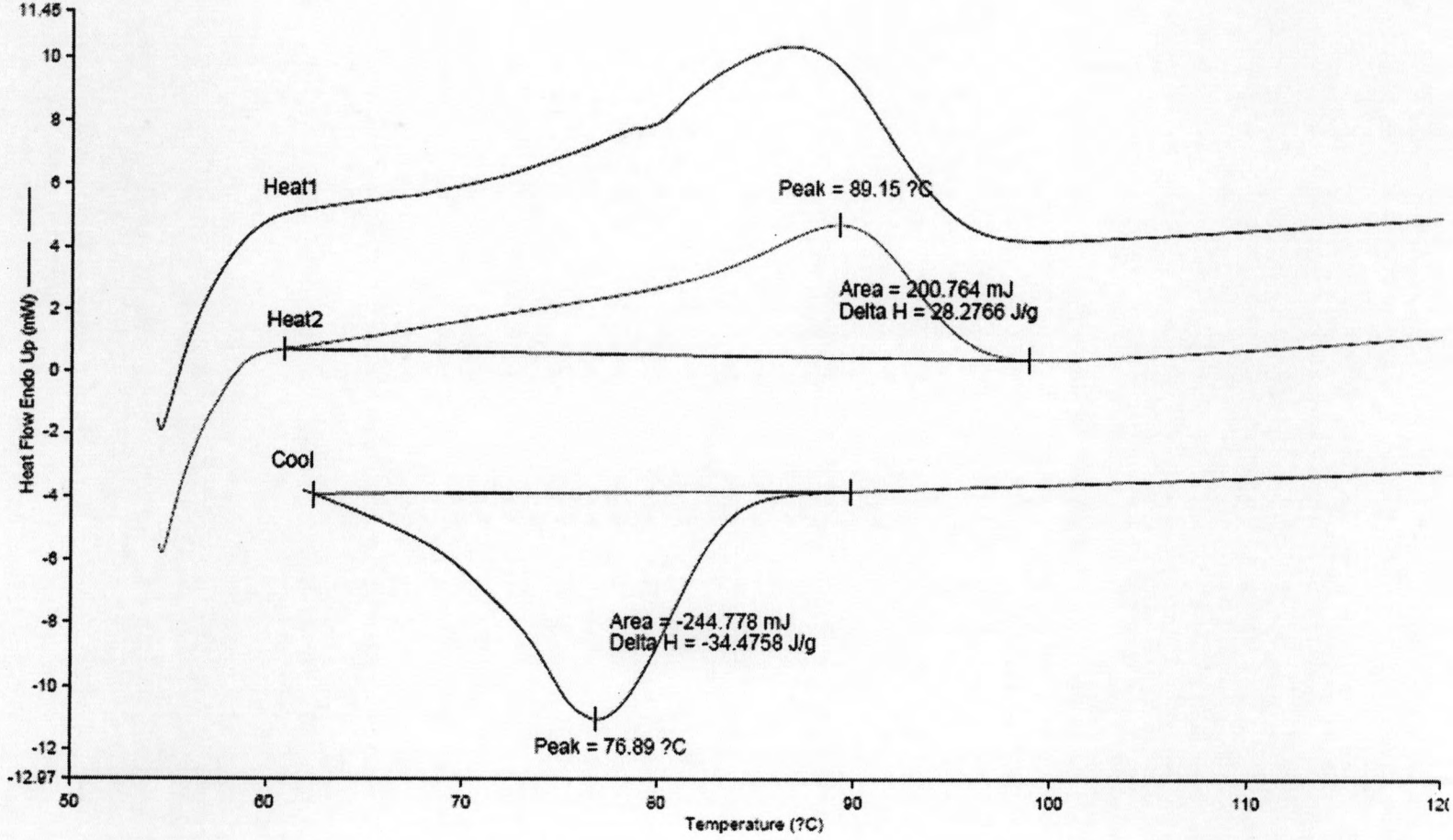

Figure B-5. DSC curve of ethylene/1-hexene copolymer produce with SiO_2

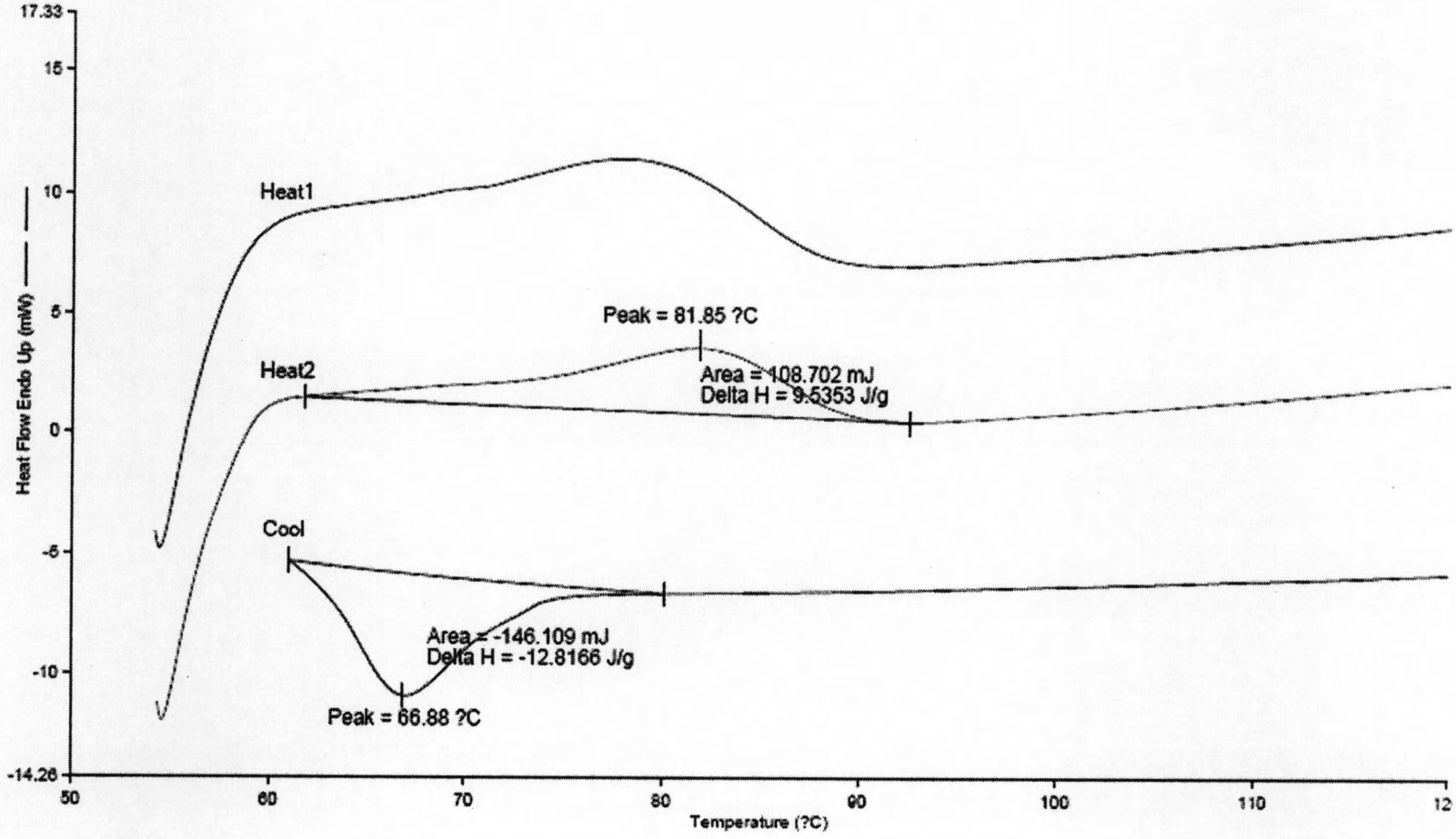

Figure B-6. DSC curve of ethylene/1-hexene copolymer produce with 1%Zr-SiO_2

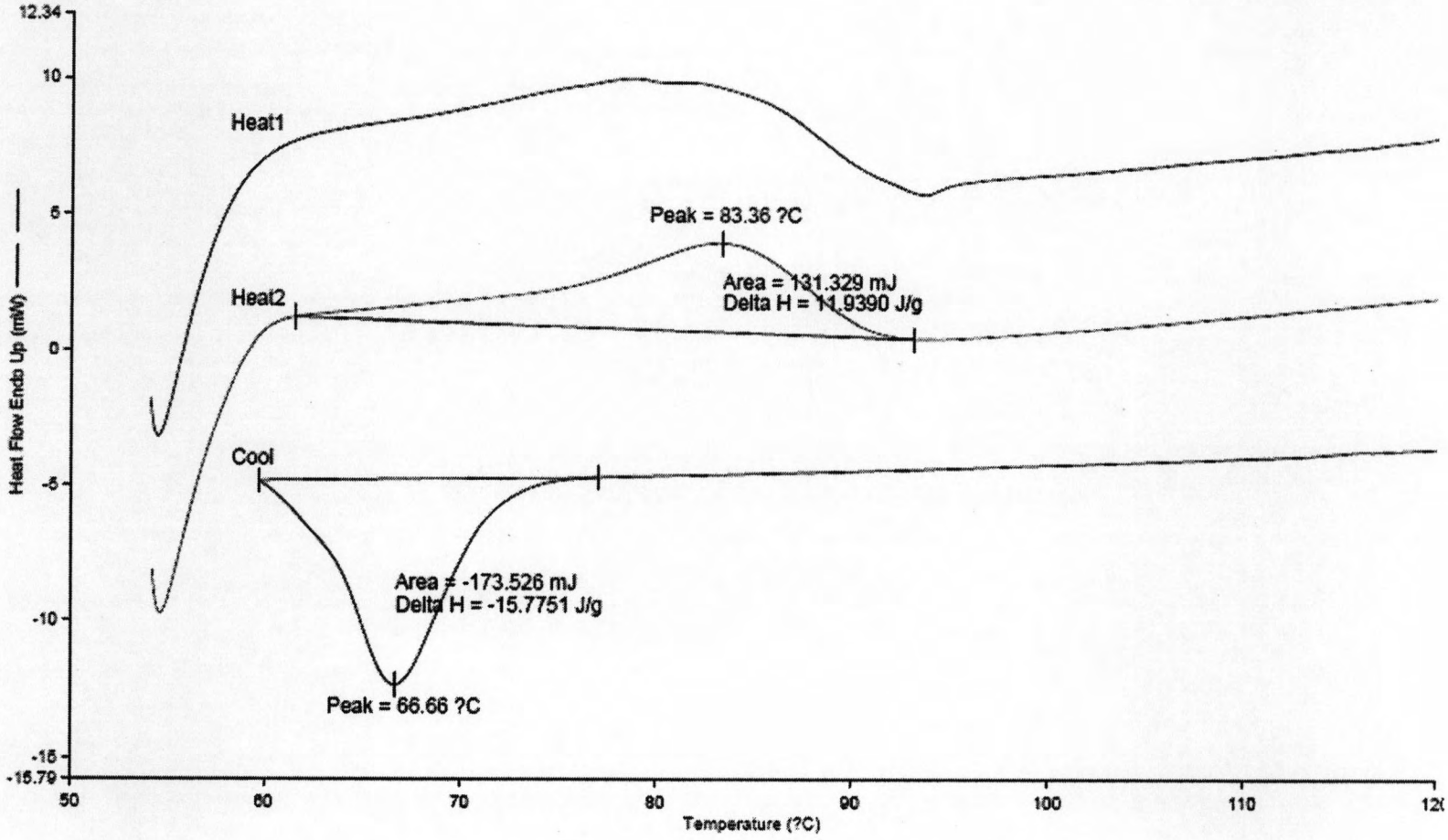

Figure B-7. DSC curve of ethylene/1-hexene copolymer produce with 2%Zr-SiO_2

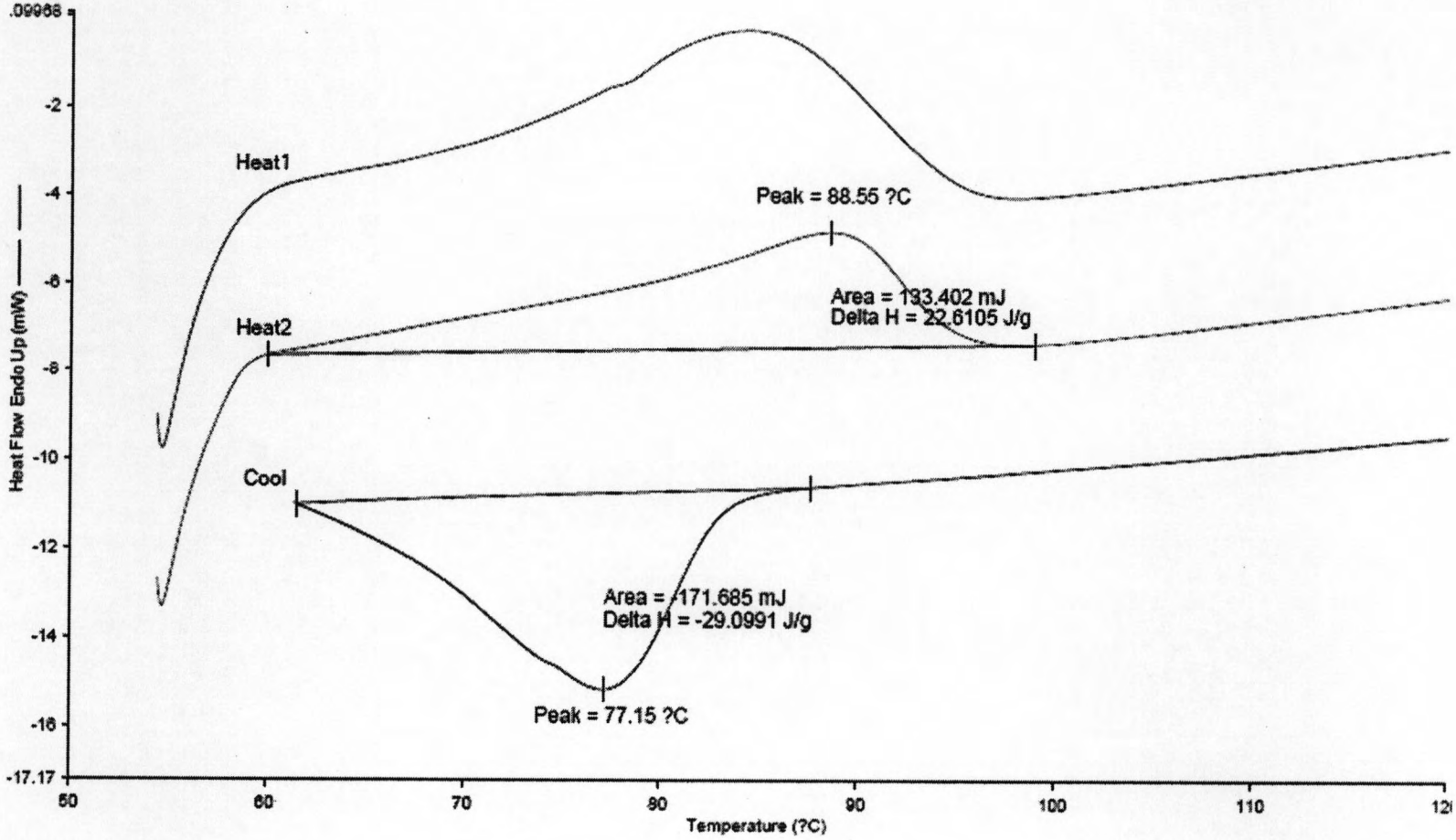

Figure B-8. DSC curve of ethylene/1-hexene copolymer produce with 5%Zr-SiO_2

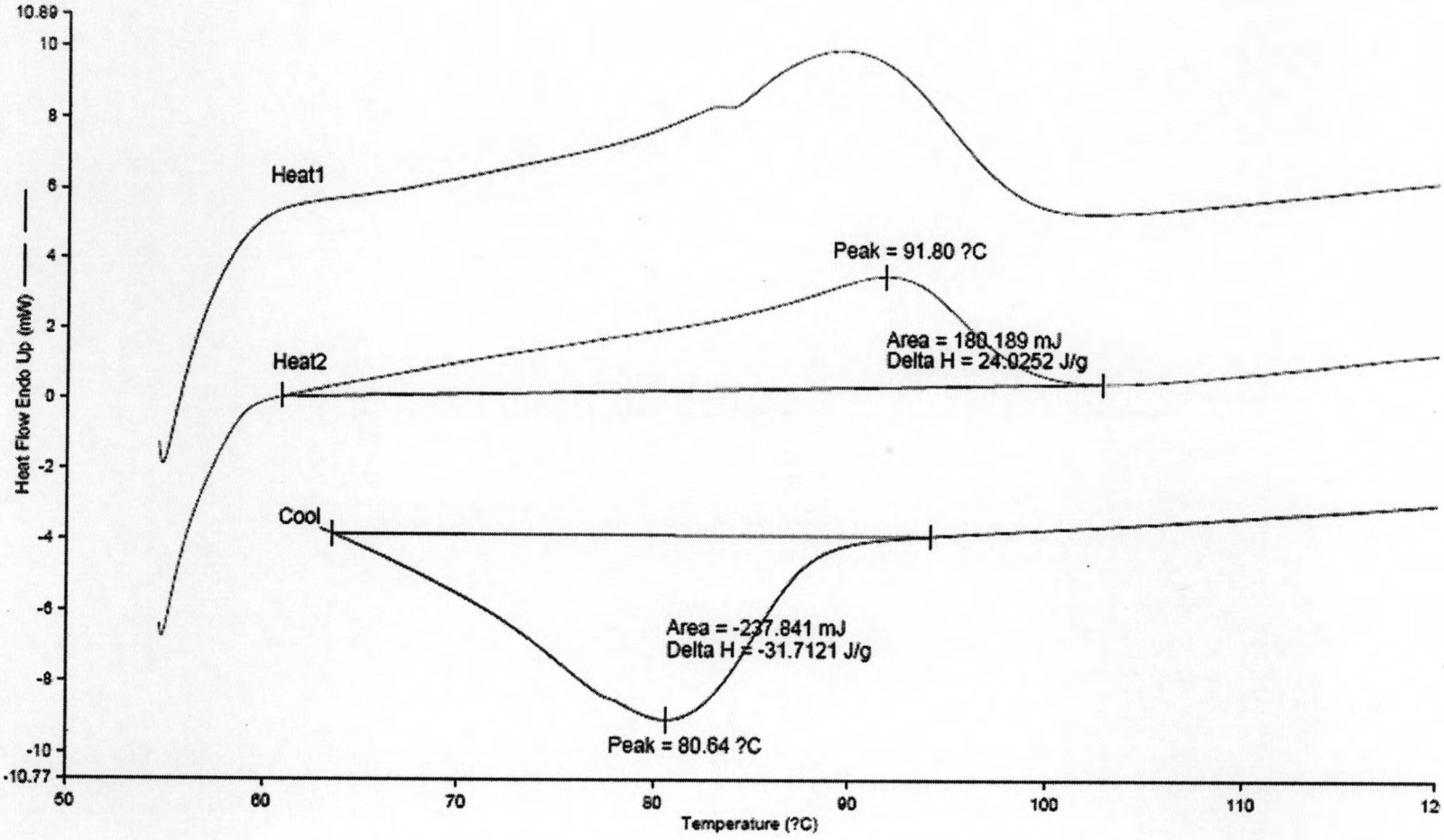

Figure B-9. DSC curve of ethylene/1-decene copolymer produce with SiO_2

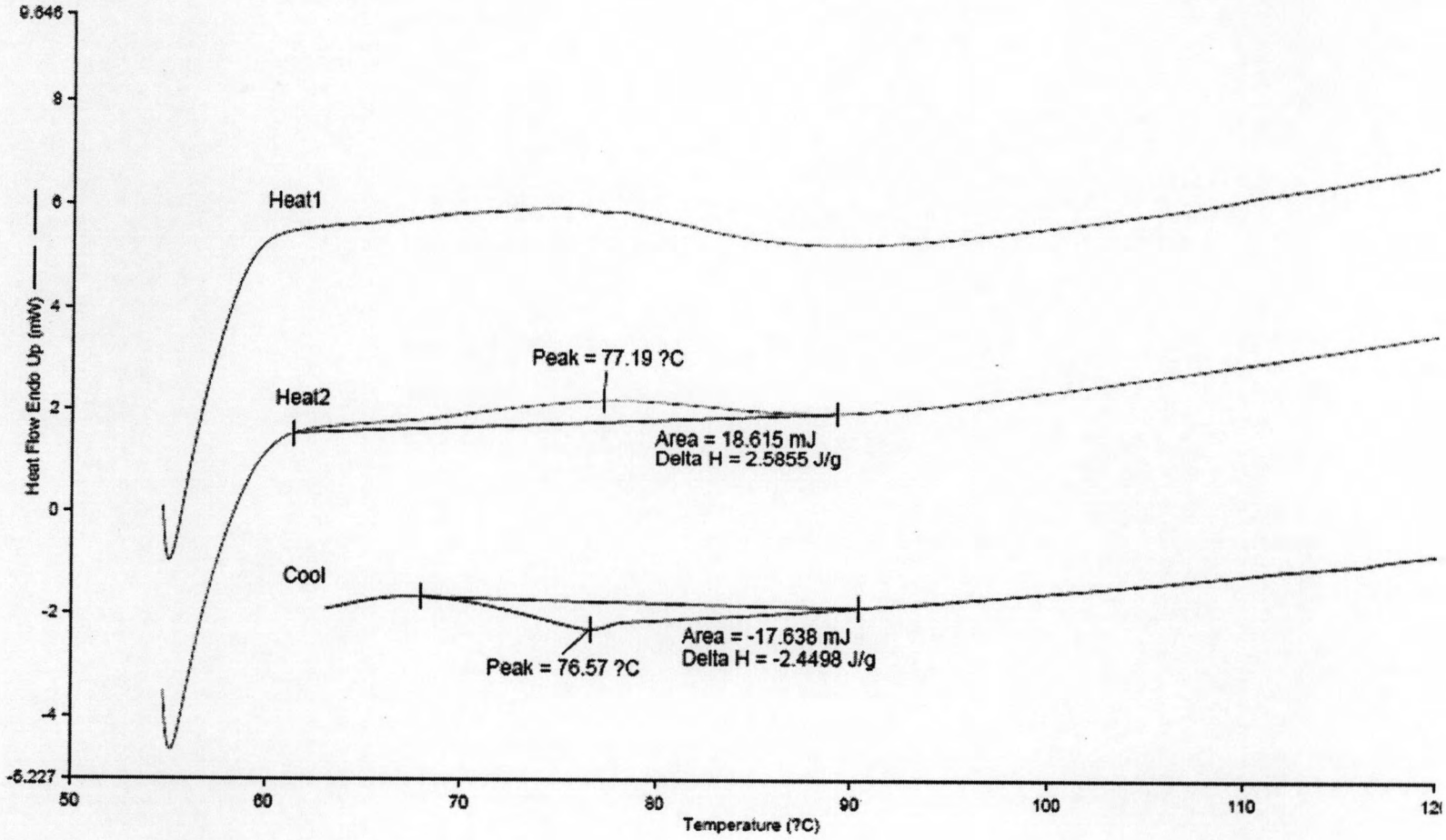

Figure B-10. DSC curve of ethylene/1-decene copolymer produce with 1%Zr-SiO_2

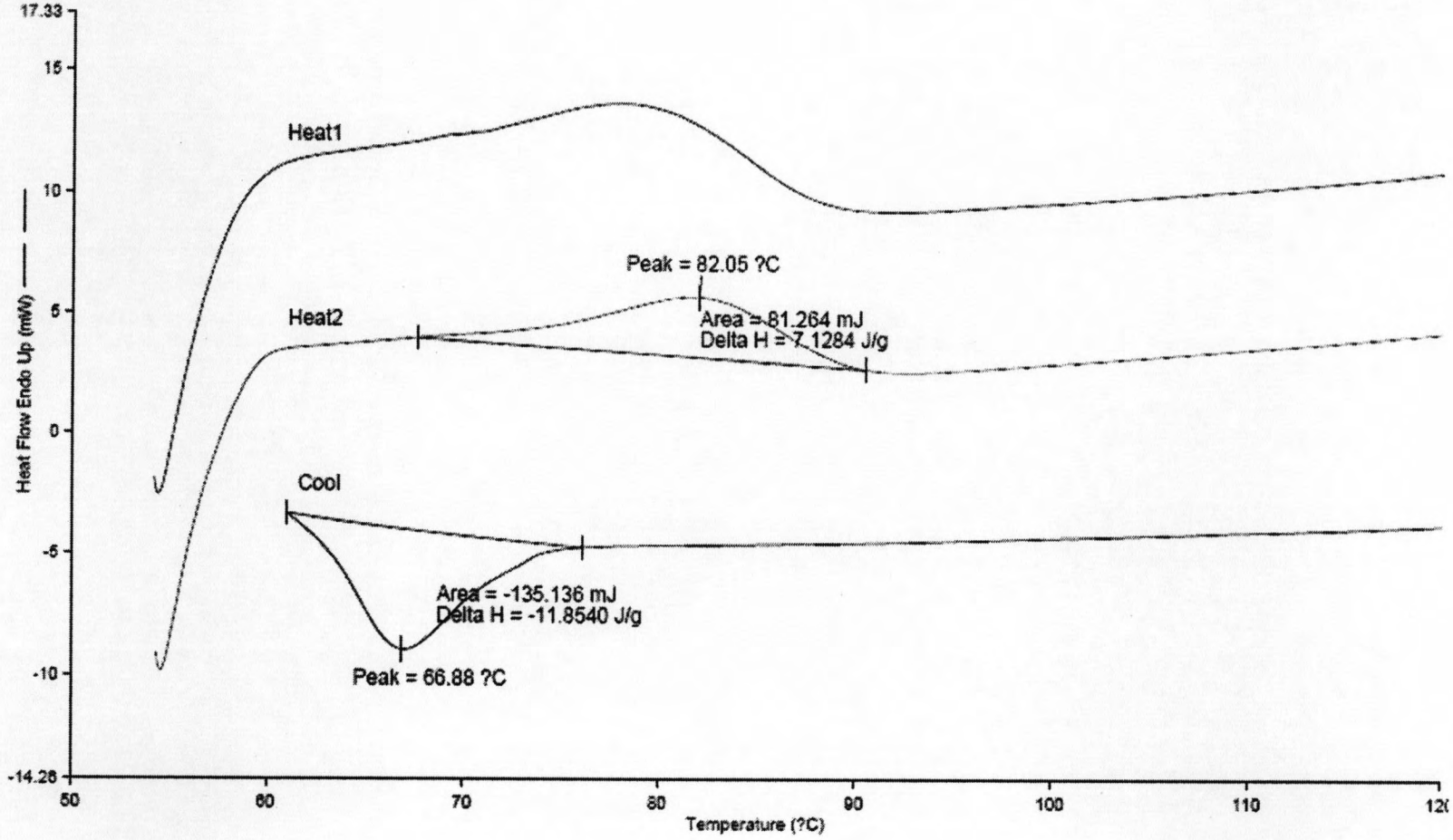

Figure B-11. DSC curve of ethylene/1-decene copolymer produce with 2%Zr-SiO_2

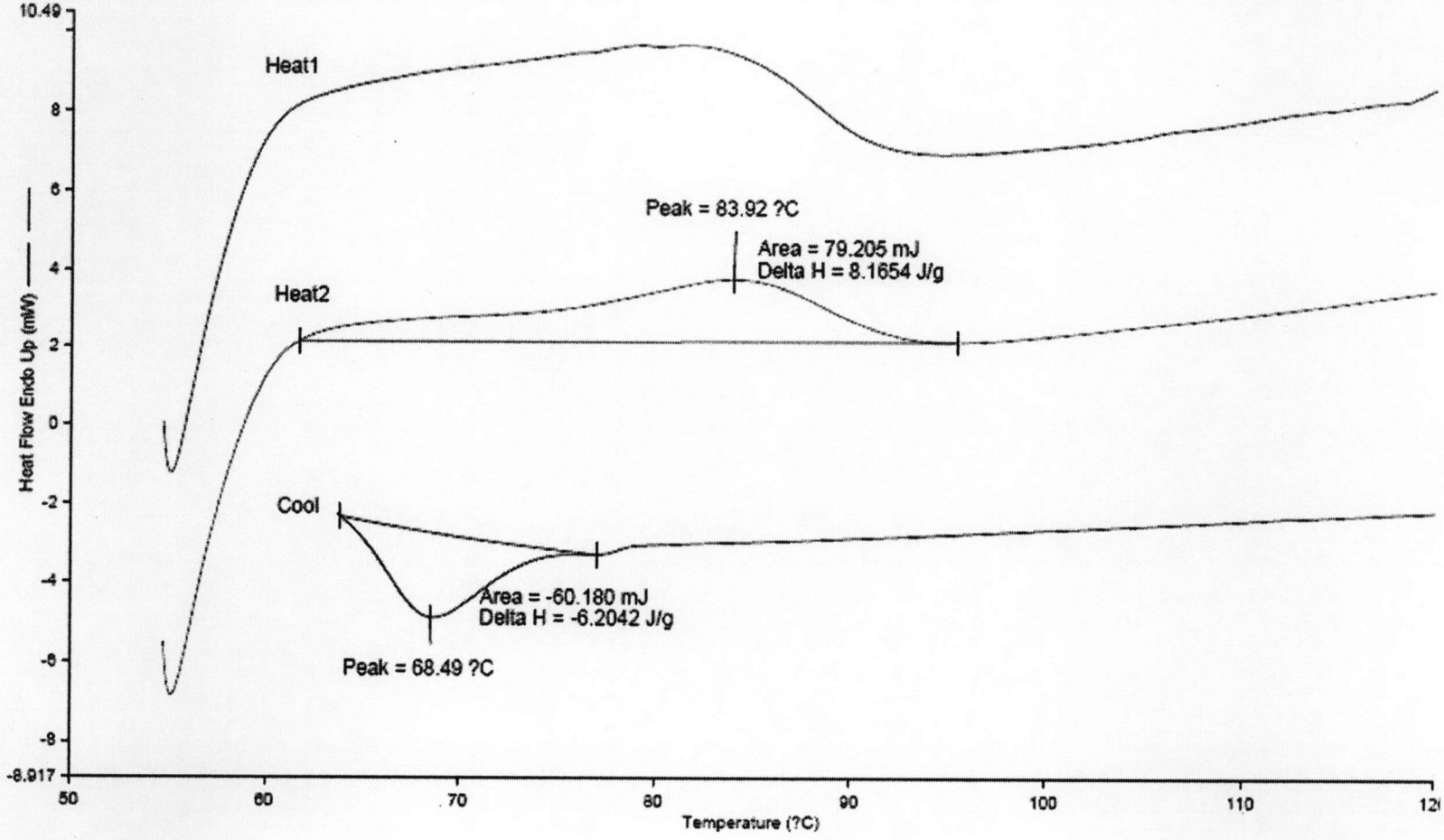

Figure B-12. DSC curve of ethylene/1-decene copolymer produce with 5%Zr-SiO_2

APPENDIX C
(Nuclear Magnetic Resonance)

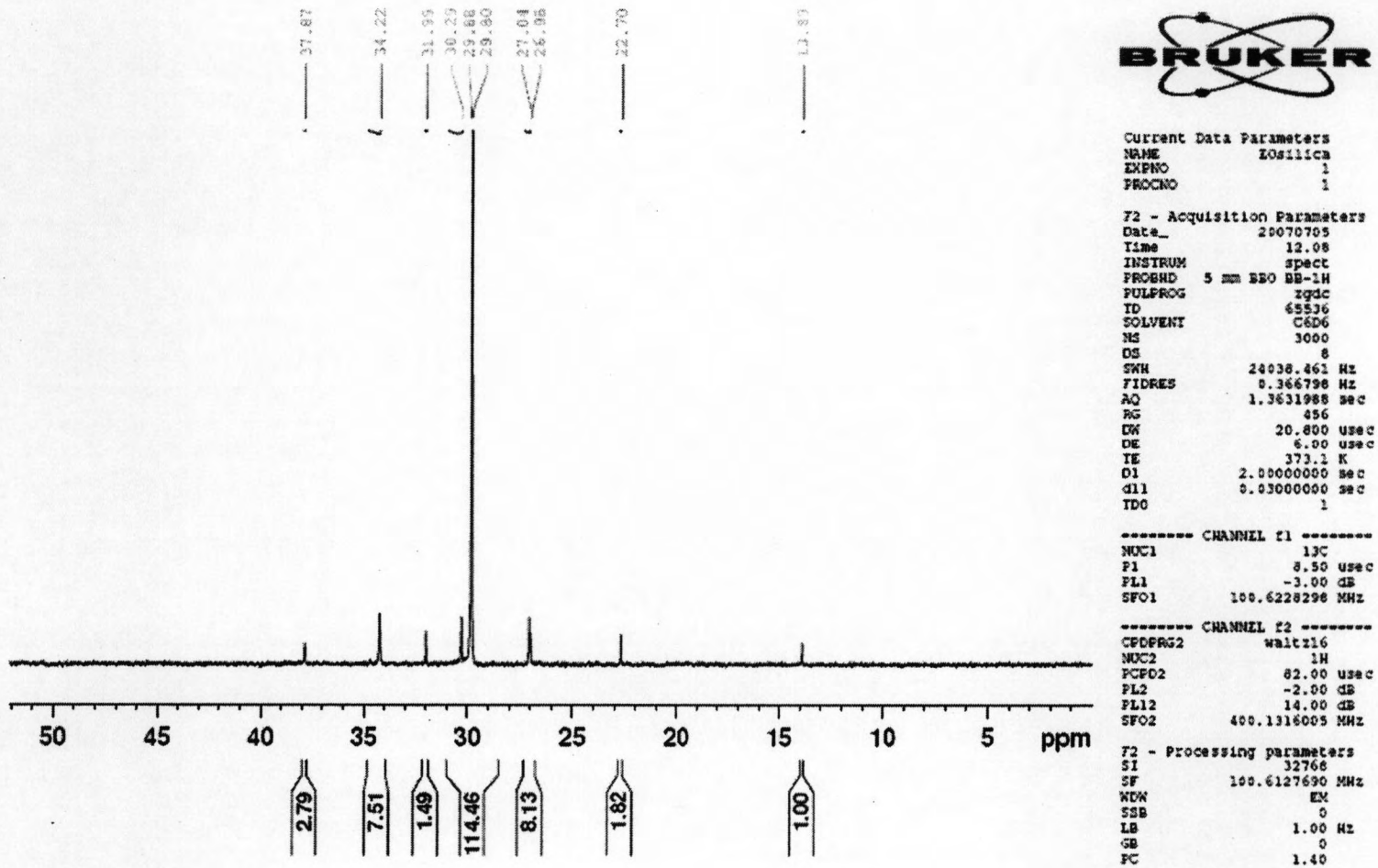

Figure C-1. ^{13}C-NMR spectrum of ethylene/1-octene copolymer produce with SiO_2

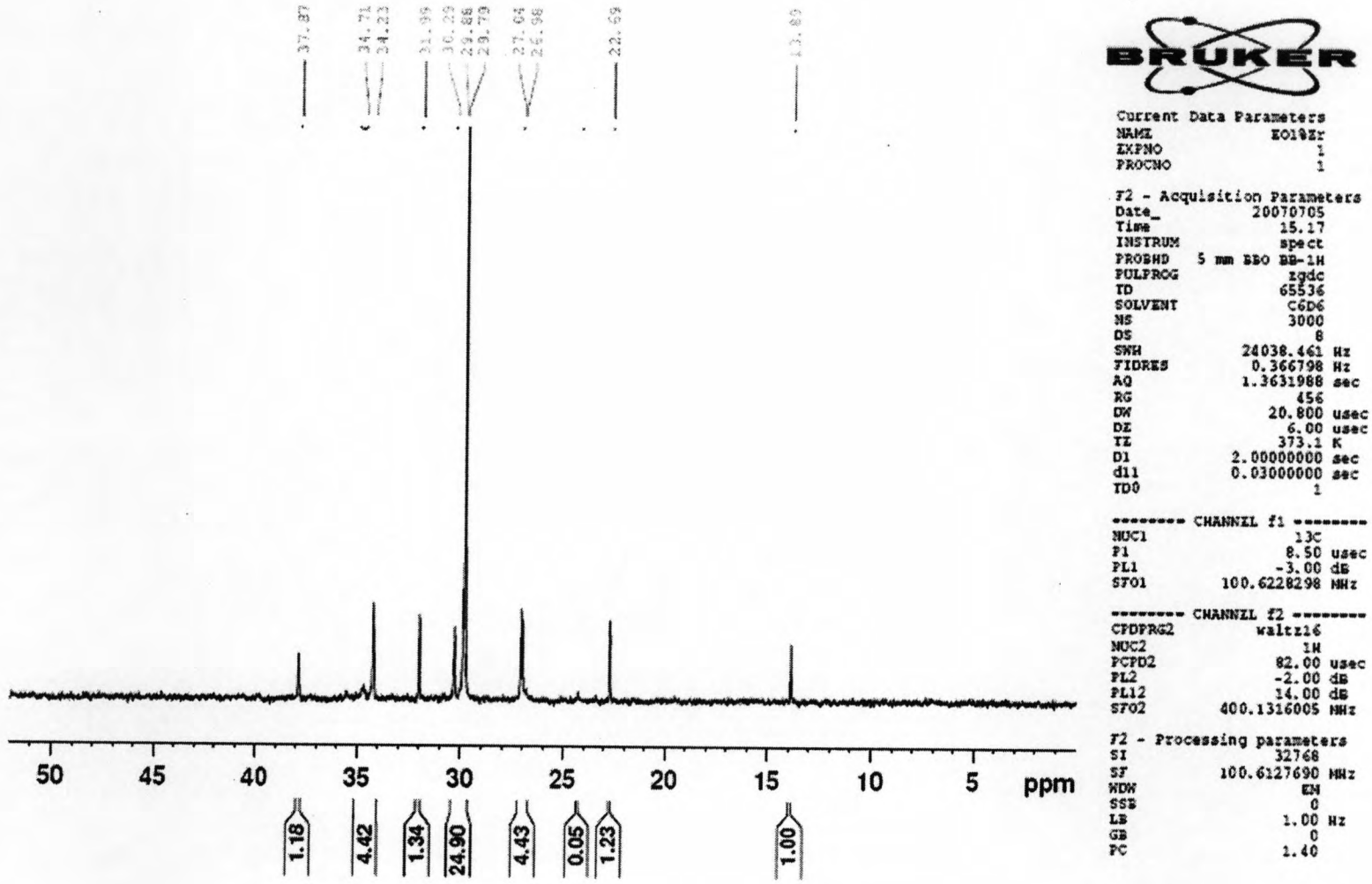

Figure C-2. ^{13}C-NMR spectrum of ethylene/1-octene copolymer produce with 1%Zr-SiO_2

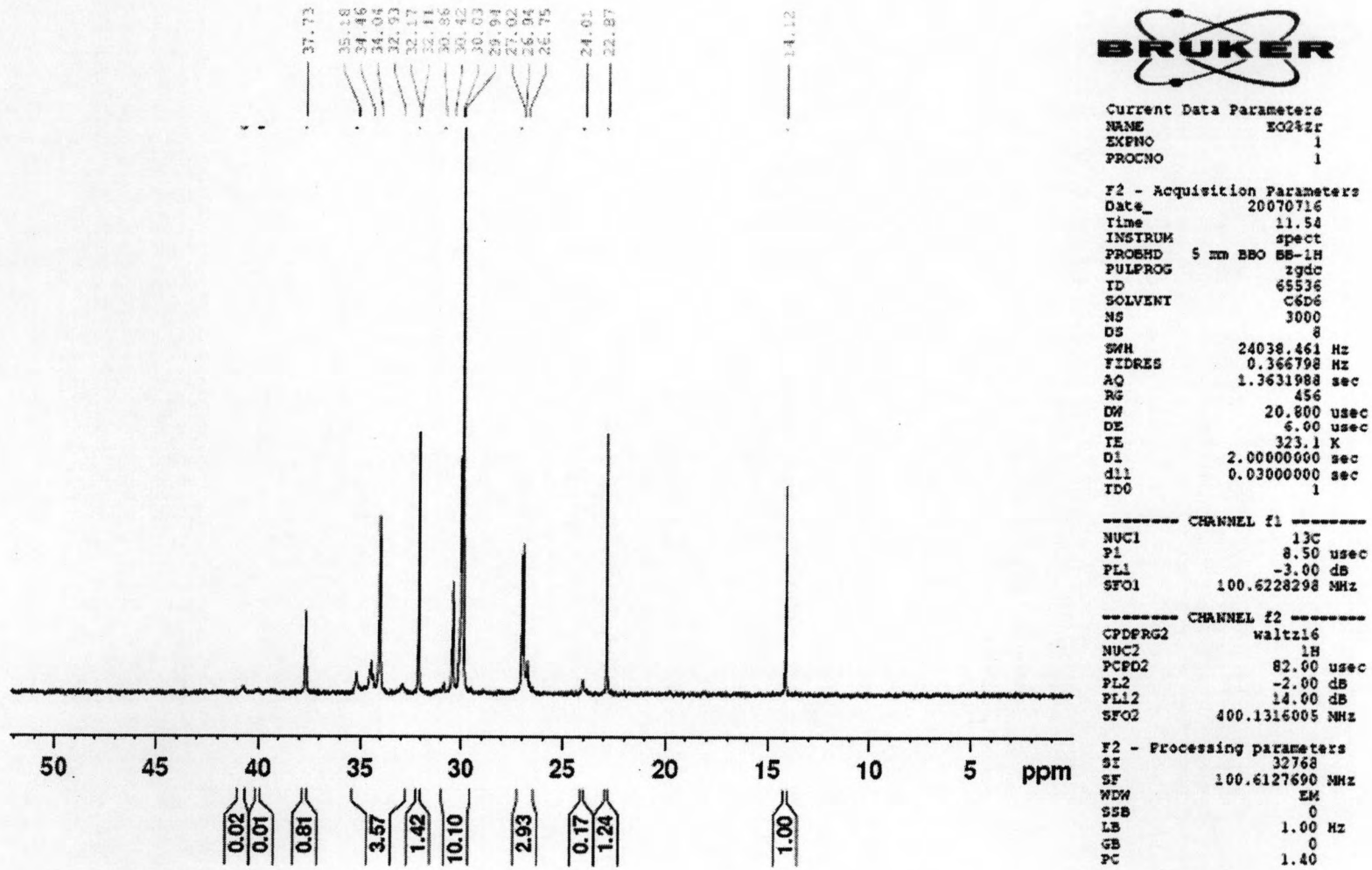

Figure C-3. ^{13}C-NMR spectrum of ethylene/1-octene copolymer produce with 2%Zr-SiO_2

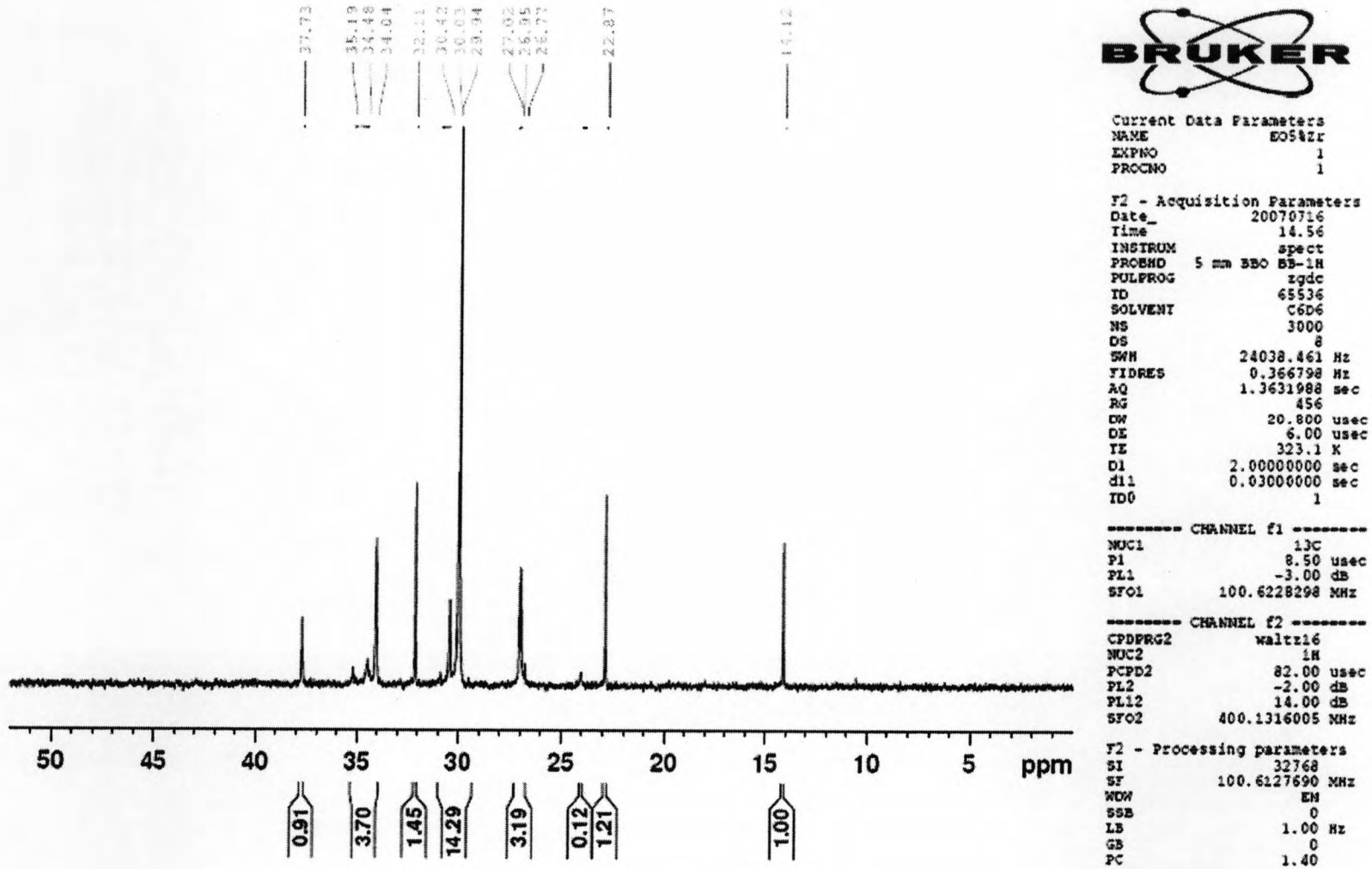

Figure C-4. ^{13}C-NMR spectrum of ethylene/1-octene copolymer produce with 5%Zr-SiO_2

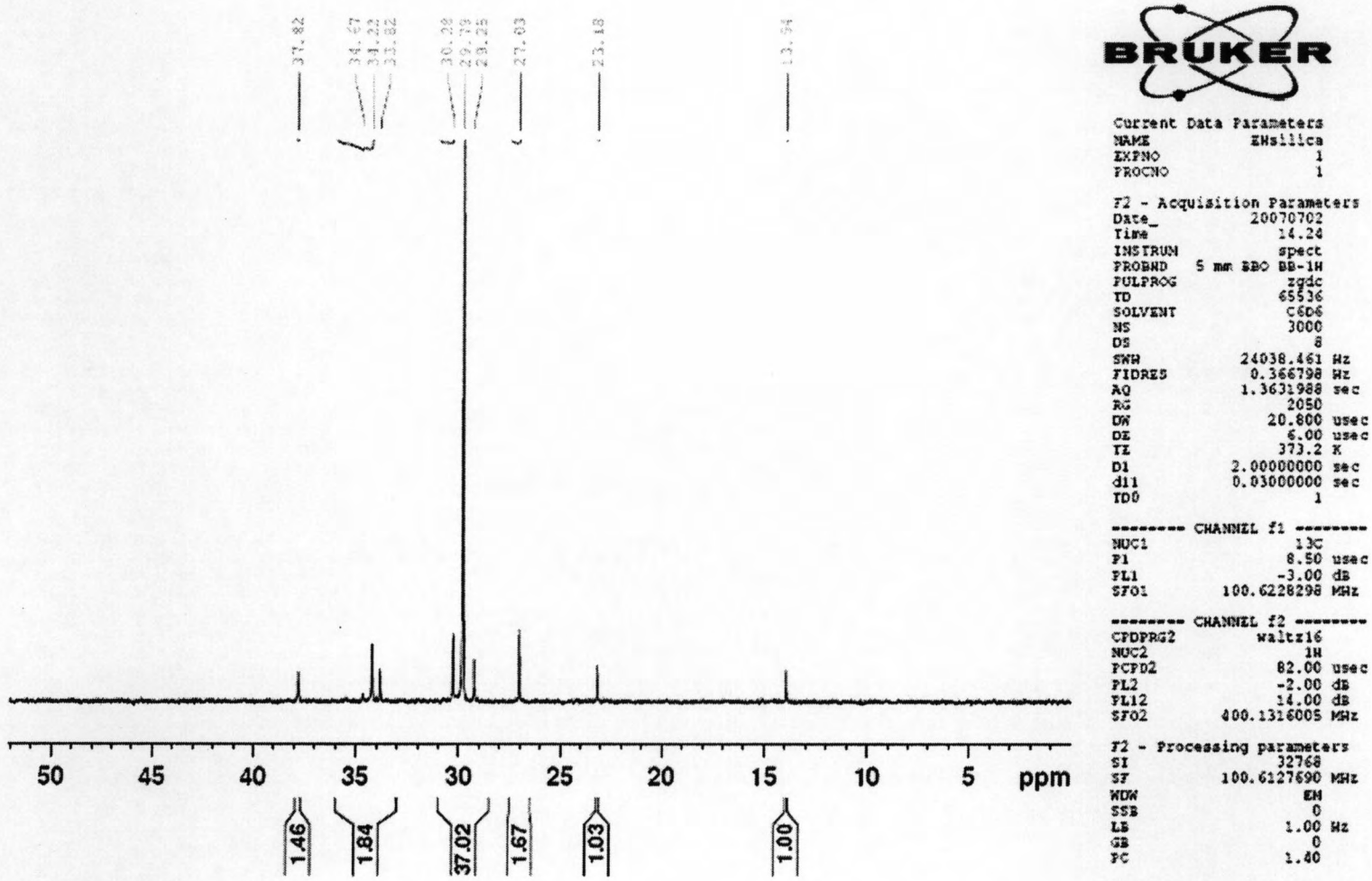

Figure C-5. ^{13}C-NMR spectrum of ethylene/1-hexene copolymer produce with SiO_2

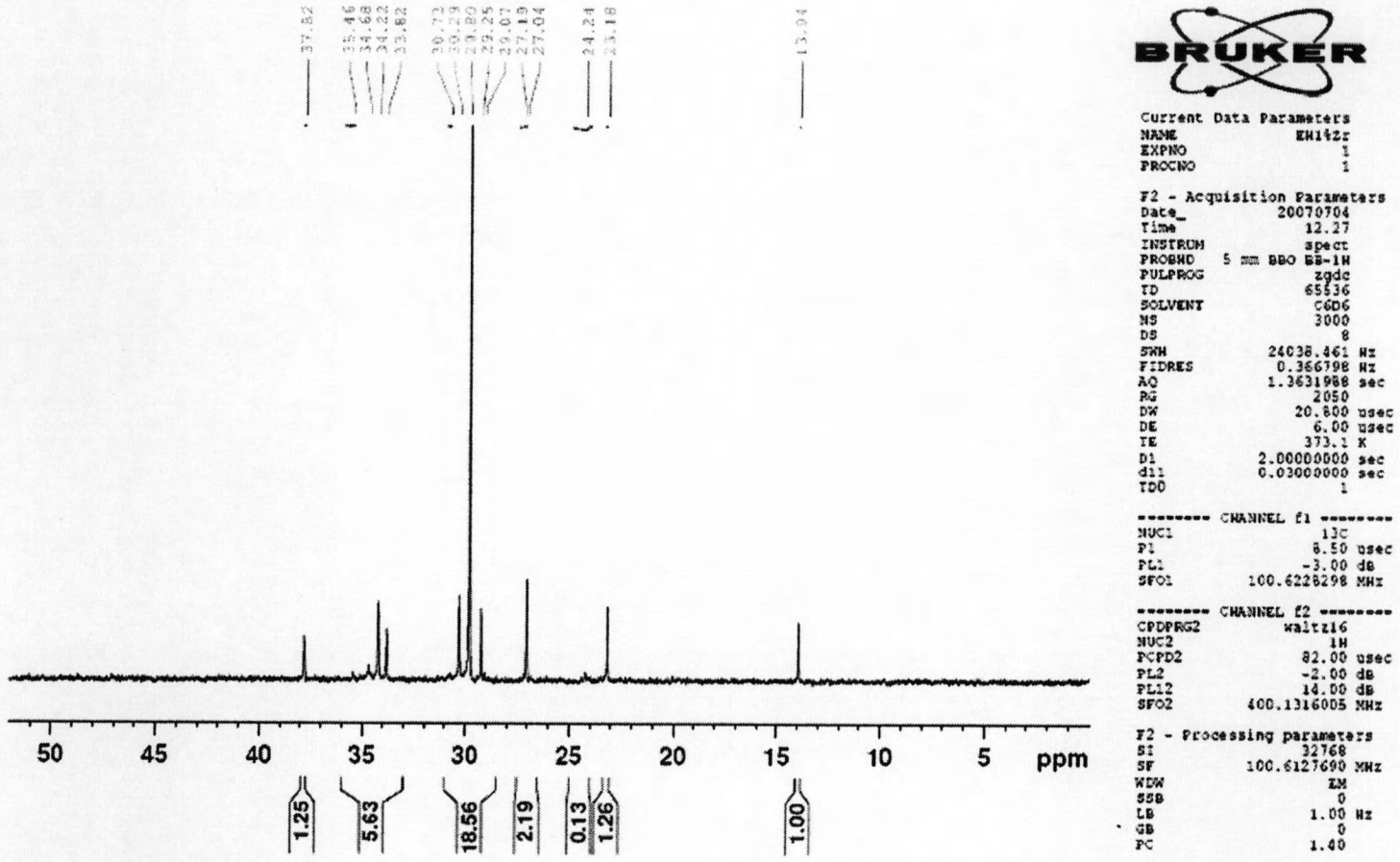

Figure C-6. ^{13}C-NMR spectrum of ethylene/1-hexene copolymer produce with 1%Zr-SiO_2

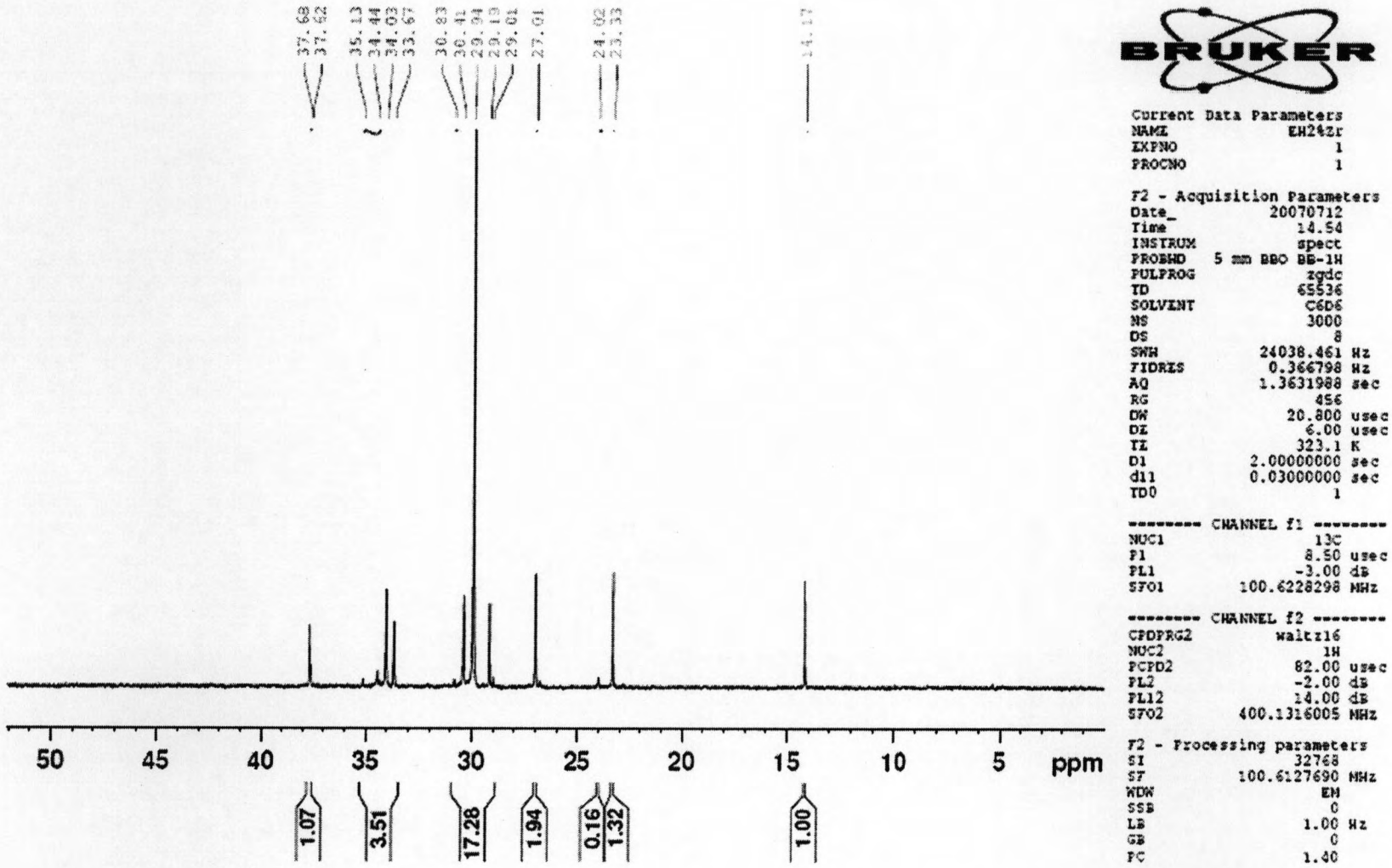

Figure C-7. ^{13}C-NMR spectrum of ethylene/1-hexene copolymer produce with 2%Zr-SiO_2

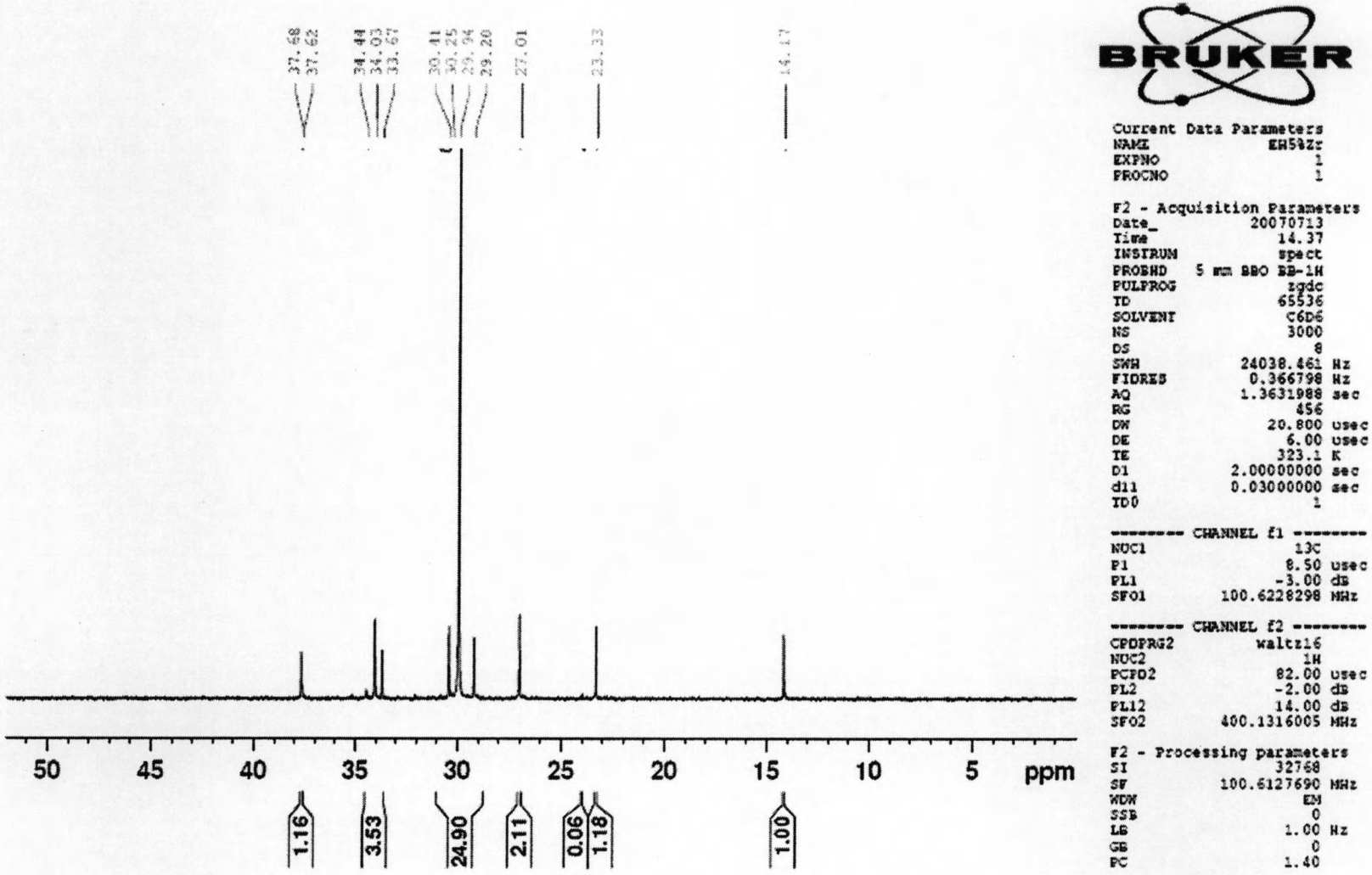

Figure C-8. ^{13}C-NMR spectrum of ethylene/1-hexene copolymer produce with 5%Zr-SiO_2

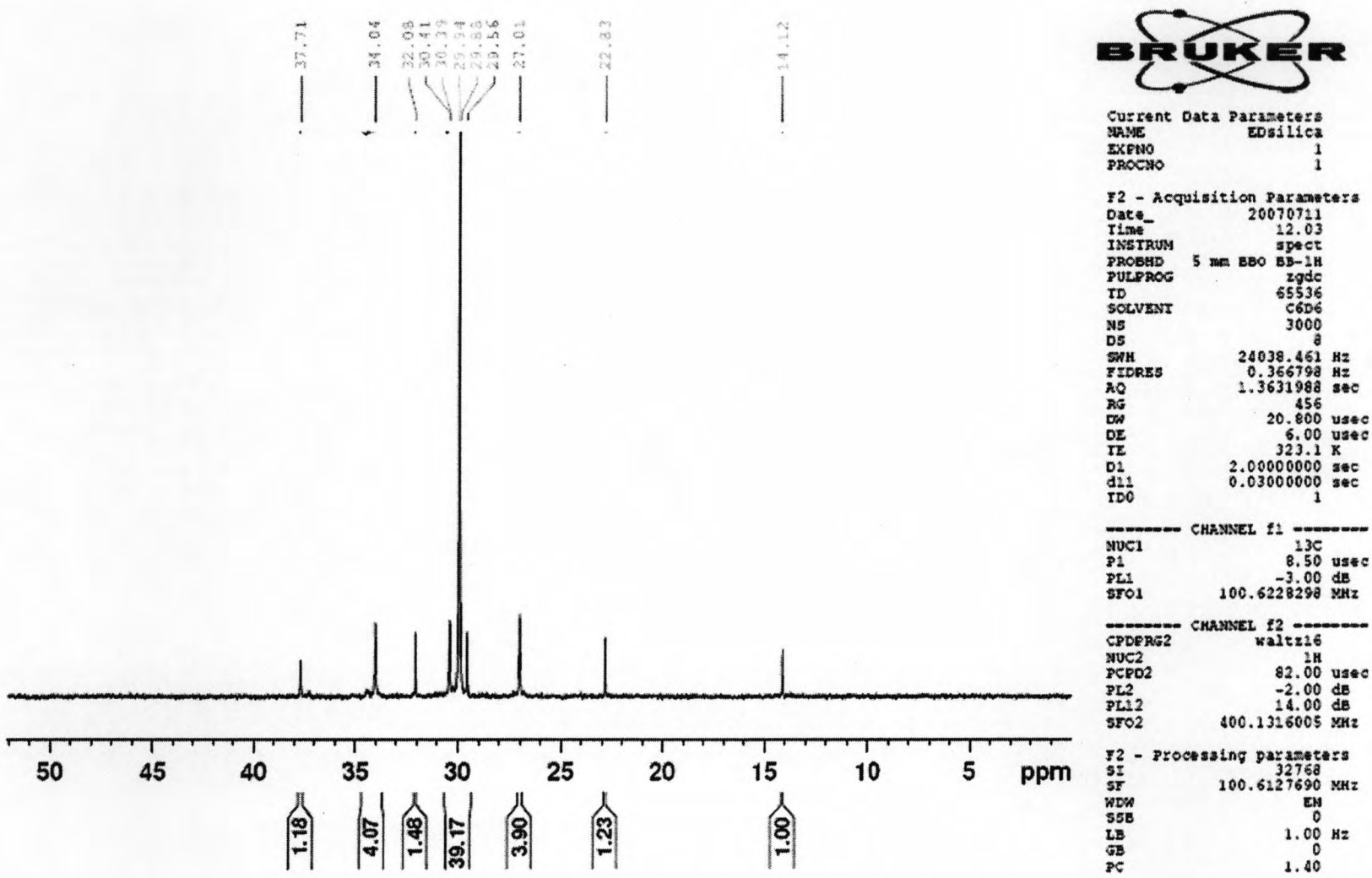

Figure C-9. ^{13}C-NMR spectrum of ethylene/1-decene copolymer produce with SiO_2

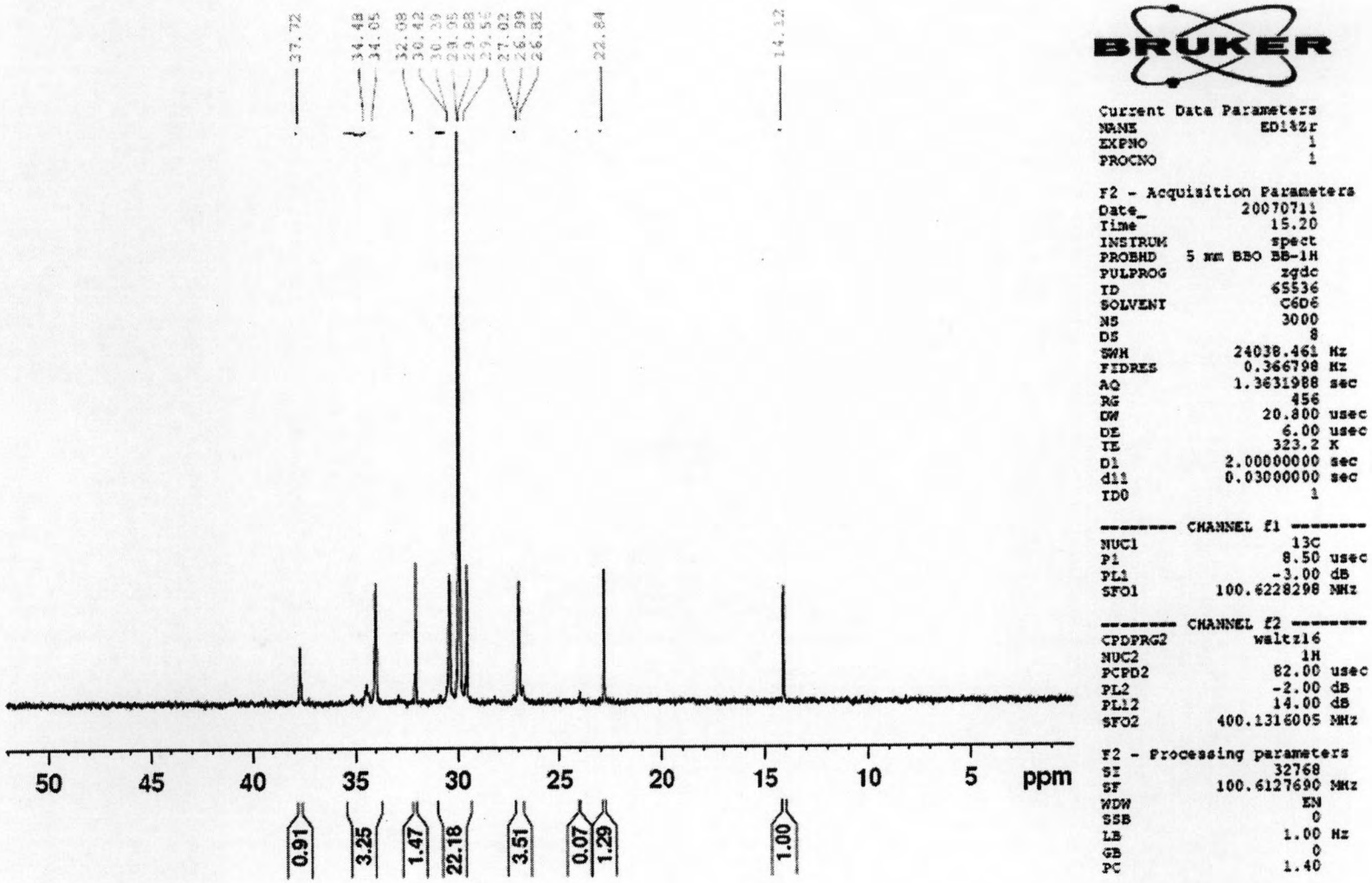

Figure C-10. ^{13}C-NMR spectrum of ethylene/1-decene copolymer produce with 1%Zr-SiO_2

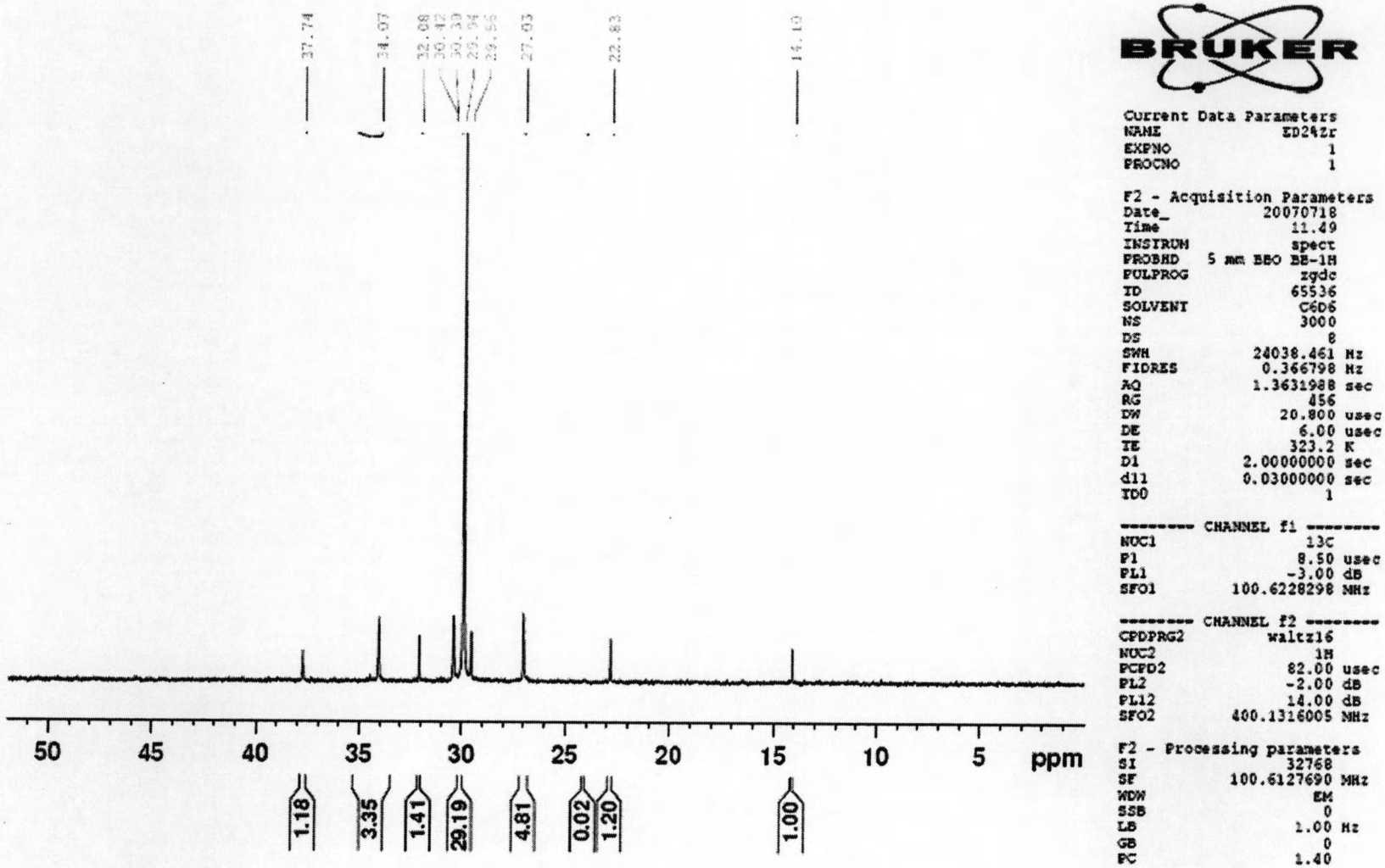

Figure C-11. ^{13}C-NMR spectrum of ethylene/1-decene copolymer produce with 2%Zr-SiO_2

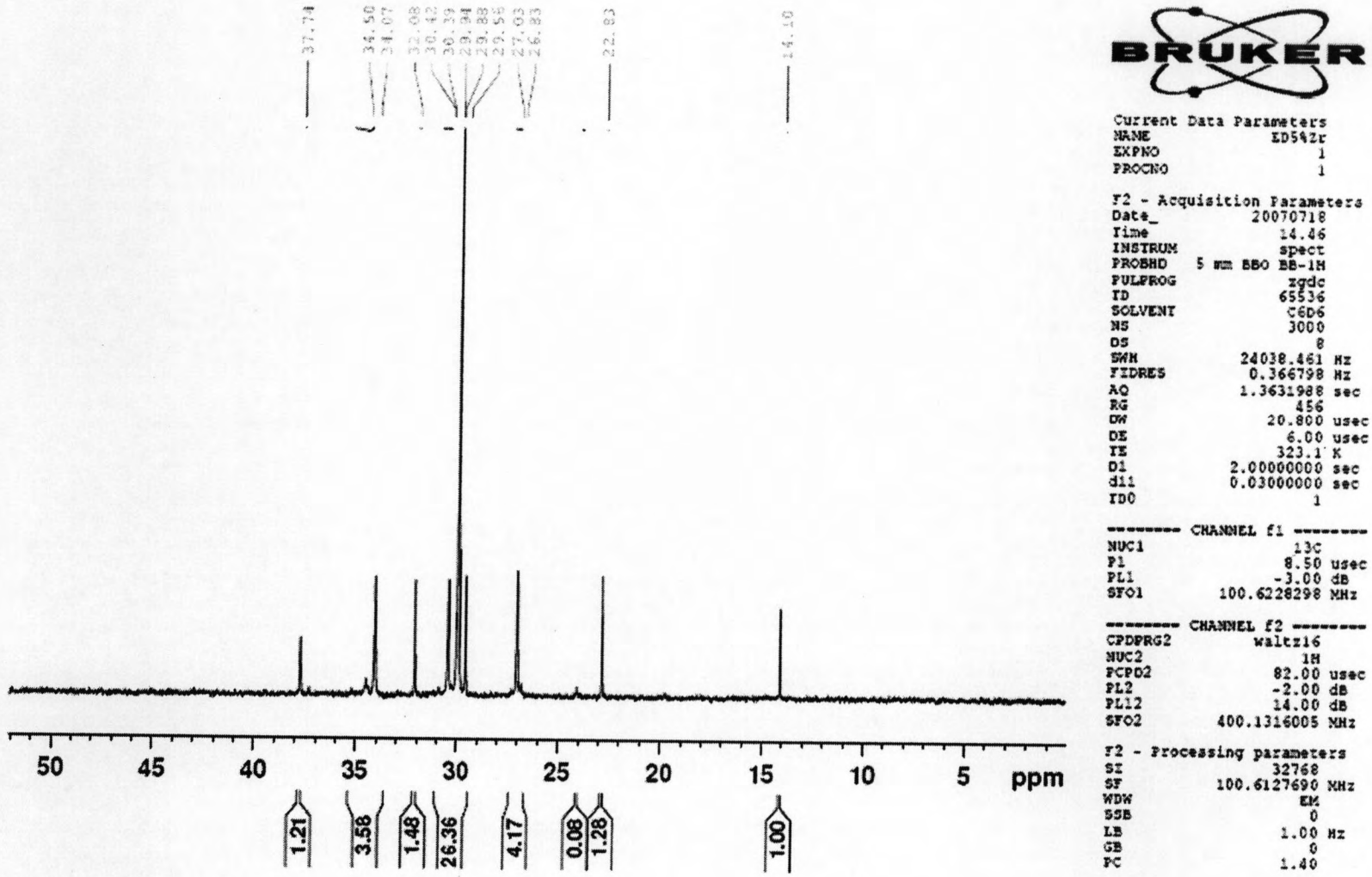

Figure C-12. ^{13}C-NMR spectrum of ethylene/1-decene copolymer produce with 5%Zr-SiO_2

APPENDIX D
(Thermogravimetric analysis)

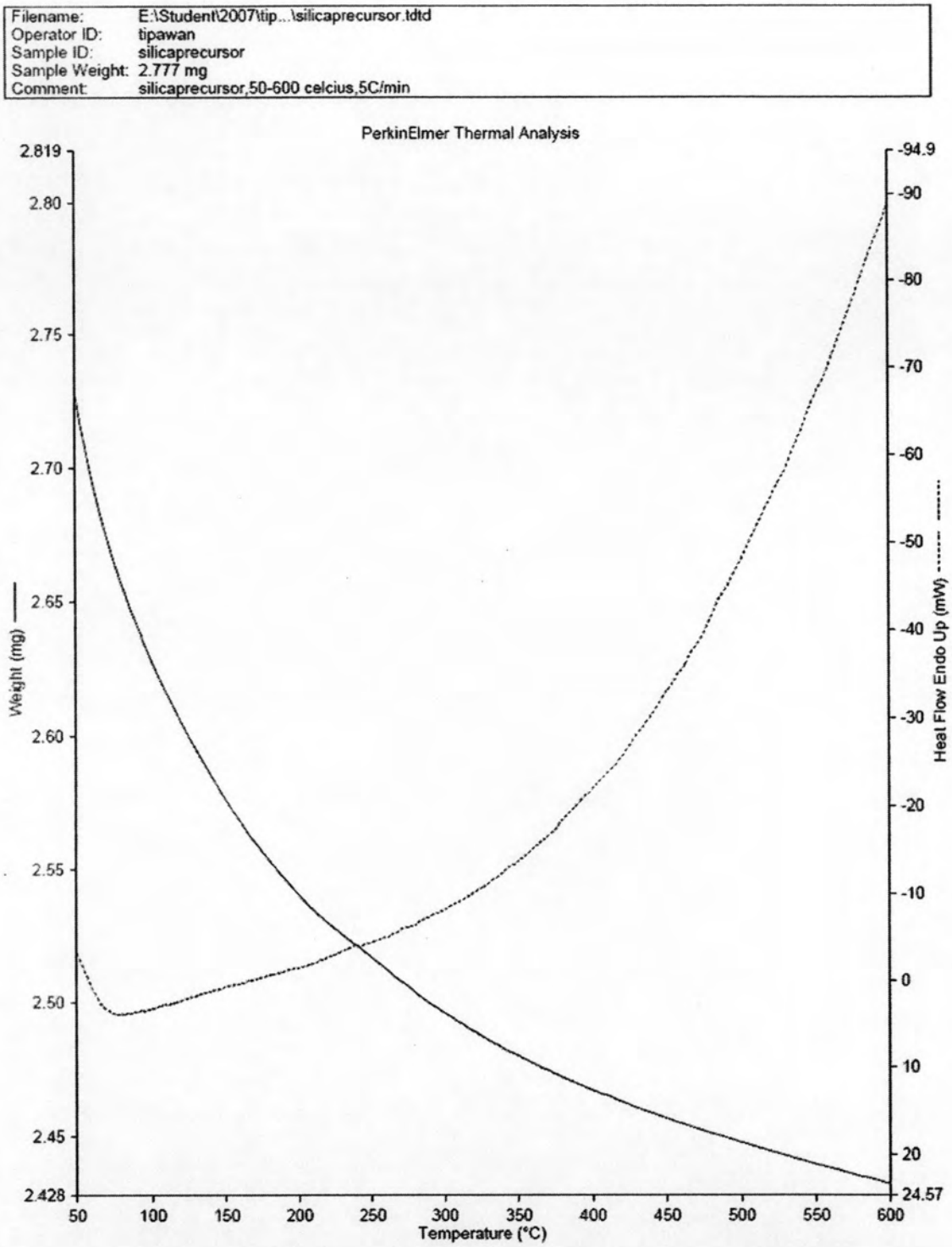

Figure D-1. The TGA profile of various zirconia-modified supports after MAO impregnation at SiO_2

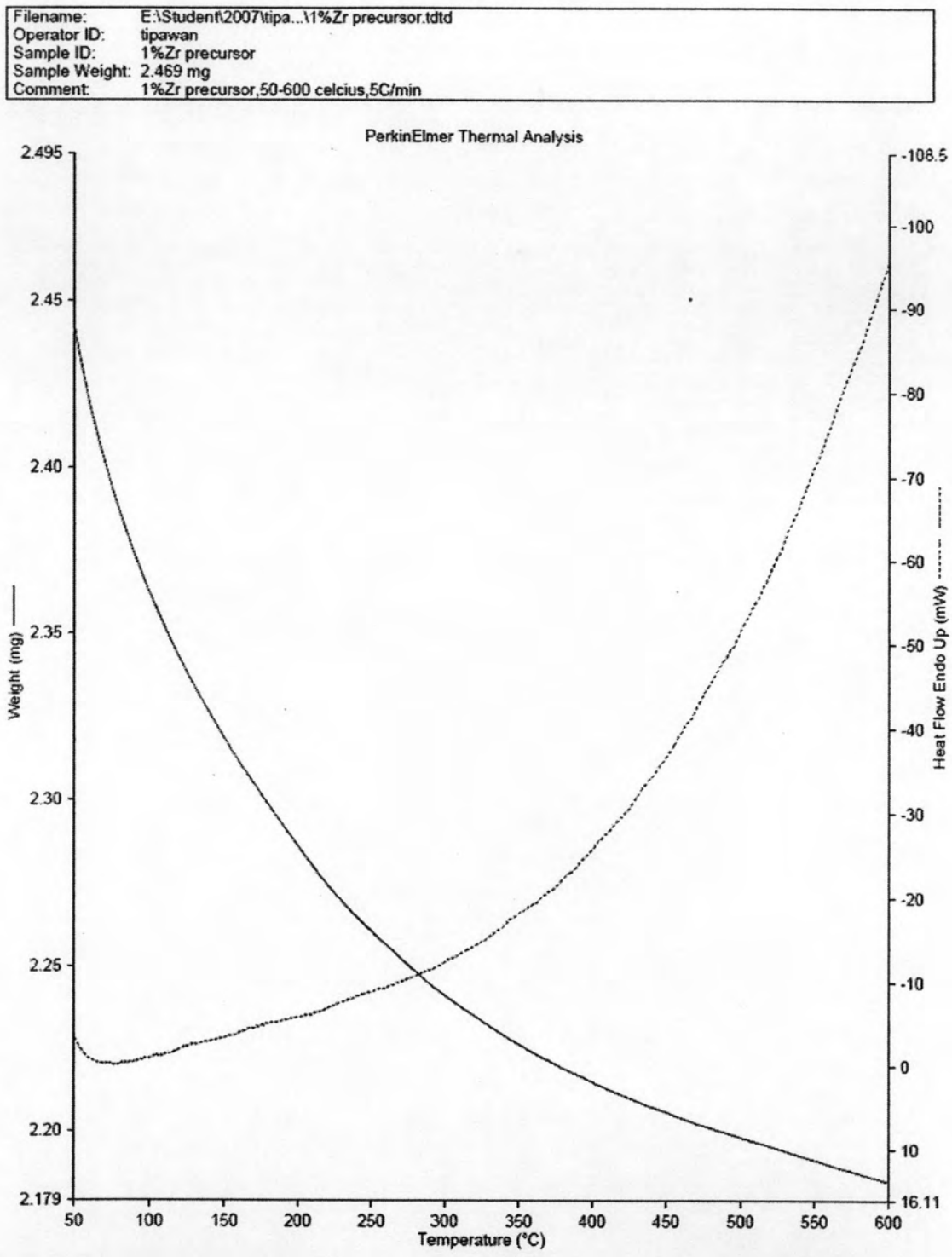

Figure D-2. The TGA profile of various zirconia-modified supports after MAO impregnation at 1%Zr-SiO_2

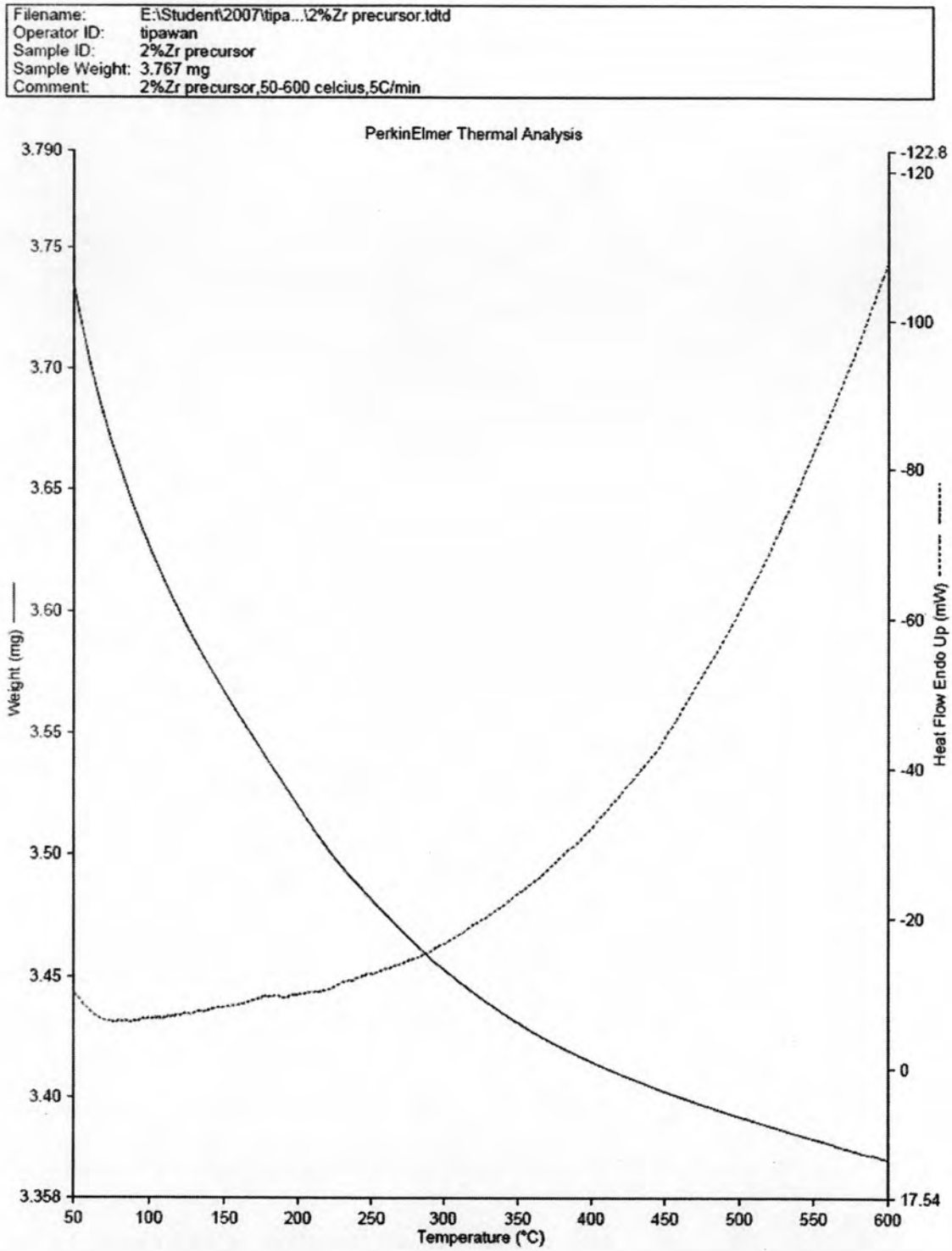

Figure D-3. The TGA profile of various zirconia-modified supports after MAO impregnation at 2%Zr-SiO_2

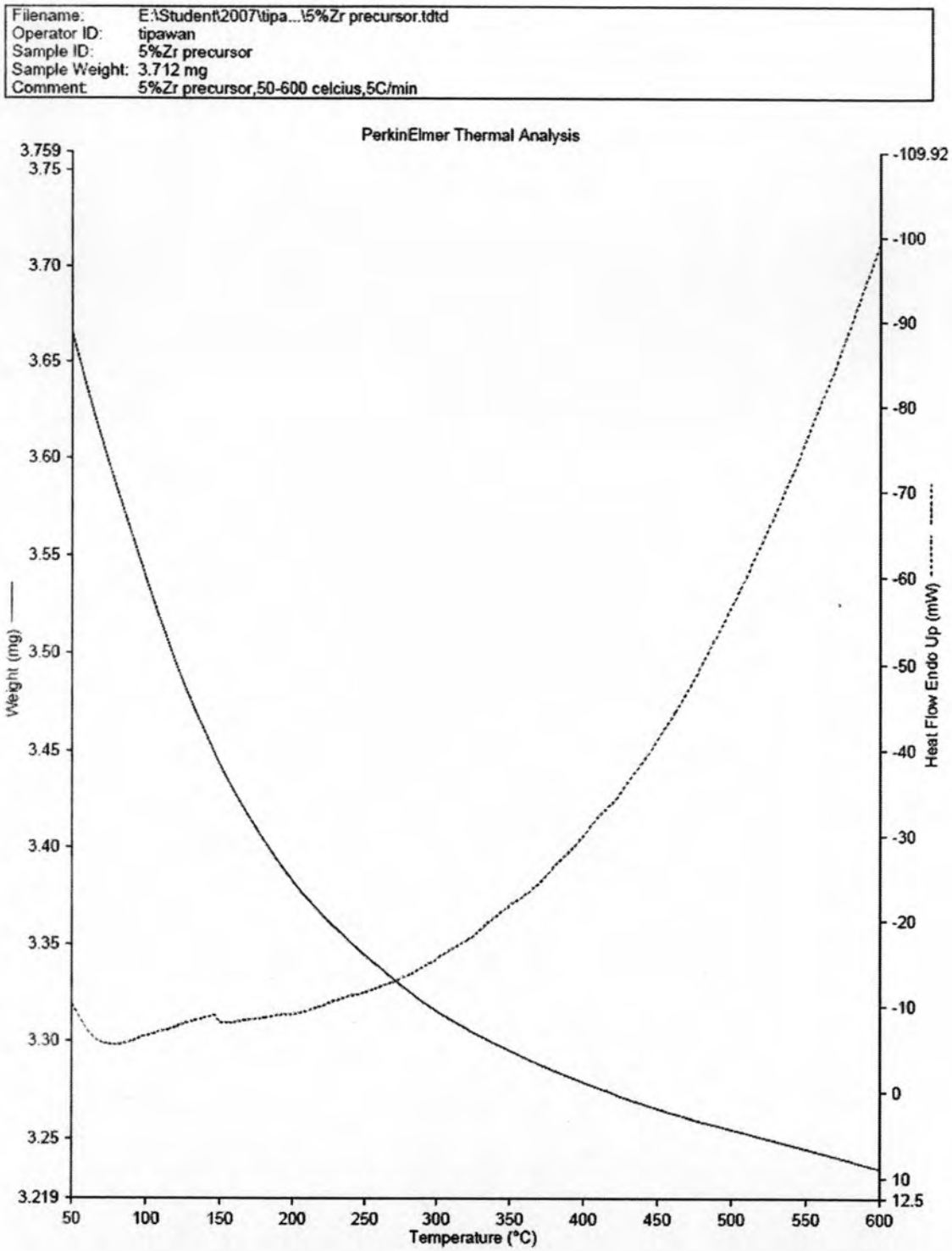

Figure D-4. The TGA profile of various zirconia-modified supports after MAO impregnation at 5%Zr-SiO_2

APPENDIX E
(Calculation of polymer properties)

E-1 Calculation of polymer microstructure

Polymer microstructure and also triad distribution of monomer can be calculated according to the Prof. James C. Randall [70] in the list of reference. The detail of calculation for ethylene/α-olefin copolymer was interpreted as follow

Ethylene/1-hexene copolymer

The integral area of ^{13}C-NMR spectrum in the specify range are listed.

T_A	=	39.5 - 42	ppm
T_B	=	38.1	ppm
T_C	=	33 - 36	ppm
T_D	=	28.5 - 31	ppm
T_E	=	26.5 – 27.5	ppm
T_F	=	24 - 25	ppm
T_G	=	23.4	ppm
T_H	=	14.1	ppm

Triad distribution was calculated as the followed formula.

k[HHH]	=	$2T_A - T_C + T_G + 2T_F + T_E$
k[EHH]	=	$2T_C - 2T_G - 4T_F - 2T_E - 2T_A$
k[EHE]	=	T_B
k[EEE]	=	$0.5T_D - 0.5T_G - 0.25T_E$
k[HEH]	=	T_F
k[HEE]	=	T_E

Ethylene/1-octene copolymer

The integral area of ^{13}C-NMR spectrum in the specify range are listed.

T_A	=	39.5 - 42	ppm
T_B	=	38.1	ppm
T_C	=	36.4	ppm
T_D	=	33 - 36	ppm
T_E	=	32.2	ppm
T_F	=	28.5 - 31	ppm
T_G	=	25.5 - 27.5	ppm
T_H	=	24 - 25	ppm
T_I	=	22 - 23	ppm
T_J	=	14 – 15	ppm

Triad distribution was calculated as the followed formula.

k[OOO]	=	$T_A - 0.5T_C$
k[EOO]	=	T_C
k[EOE]	=	T_B
k[EEE]	=	$0.5T_F - 0.25T_E - 0.25T_G$
k[OEE]	=	$T_G - T_E$
k[OEO]	=	T_H

Ethylene/1-decene copolymer

The integral area of ^{13}C-NMR spectrum in the specify range are listed.

T_A	=	39.5 - 42	ppm
T_B	=	38.1	ppm
T_C	=	36.4	ppm
T_D	=	33 - 36	ppm
T_E	=	32.2	ppm
T_F	=	28.5 - 31	ppm
T_G	=	25.5 - 27.5	ppm
T_H	=	24 - 25	ppm
T_I	=	22 - 23	ppm
T_J	=	14 – 15	ppm

Triad distribution was calculated as the followed formula.

k[DDD]	=	$T_A - 0.5T_C$
k[EDD]	=	T_C
k[EDE]	=	T_B
k[EEE]	=	$0.5T_F - 0.5T_E - 0.5T_G - T_I$
k[DEE]	=	$T_G - T_I$
k[DED]	=	T_H

Table E-1 Reactivity ratios of ethylene and 1-olefin

Comonomer	Systems	$r_E r_H$
1-Hexene	SiO_2	11.94
	1%Zr-SiO_2	3.64
	2%Zr-SiO_2	0.39
	5%Zr-SiO_2	0.94
Comonomer	Systems	$r_E r_O$
1-Octene	SiO_2	0
	1%Zr-SiO_2	0
	2%Zr-SiO_2	0.11
	5%Zr-SiO_2	0
Comonomer	Systems	$r_E r_D$
1-Decene	SiO_2	0
	1%Zr-SiO_2	0
	2%Zr-SiO_2	0
	5%Zr-SiO_2	0

All copolymer was calculated for the relative comonomer reactivity (r_E for ethylene and r_C for the comonomer) and monomer insertion by using the general formula below

$$r_E = 2[EE]/([EC]X) \qquad r_C = 2[CC]X/[EC]$$

where r_E = ethylene reactivity ratio

r_C = comonomer (α -olefin) reactivity ratio

[EE] = [EEE] + 0.5[CEE]

[EC] = [CEC] + 0.5[CEE] + [ECE] + 0.5[ECC]

[CC] = [CCC] + 0.5[ECC]

X = [E]/[C] in the feed = concentration of ethylene (mol/L) / concentration of comonomer (mol/L) in the feed.

%E = [EEE] + [EEC] + [CEC]

%C = [CCC] + [CCE] + [ECE]

E-2 Calculation of crystallinity for ethylene/α-olefin copolymer

The crystallinities of copolymers were determined by differential scanning calorimeter. %crystallinity of copolymers is calculated from equation [68].

$$\chi(\%) = \frac{\Delta H_m}{\Delta H_m^\circ} \times 100$$

Where $\chi(\%)$ = %crystallinity

ΔH_m = the heat of fusion of sample (J/g)

ΔH_m° = the heat of fusion of perfectly crystalline polyethylene (286 J/g) [68]

APPENDIX F
(Research Output)

List of Publications

1. Pothirat, T.; Jongsomjit, B. and Praserthdam, P. "Effect of Zr-modified SiO_2-supported metallocene on ethylene/1-octene copolymerization." Catalysis Letters (In review)

2. Pothirat, T.; Jongsomjit, B. and Praserthdam, P. "Effect of comonomers on Zr-modified SiO_2-supported metallocene on ethylene/α-olefin copolymerization." (Be submitted)

3. Pothirat, T.; Jongsomjit, B. and Praserthdam, P. "A comparative study of SiO_2-supported and ZrO_2-supported zirconocene/MAO catalysts for ethylene/1-olefin copolymerization." (Be submitted)

Proceeding

1. Pothirat, T.; Jongsomjit, B. and Praserthdam, P. "Effect of Zr-modified SiO_2-supported metallocene on ethylene/1-octene copolymerization." RGJ Seminar Series in Chemical Engineering Science and Technology, Chulalongkorn University, Thailand , 28th September, 2007.

VITA

Miss Tipawan Pothirat was born on June 9, 1982 in Prachuapkirikhan, Thailand. She received the Bachelor's Degree of Science from the Department of Chemistry, Faculty of Science, Kasetsart University on March 2004. She continued the Master's Degree in the Department of Chemical Engineering, Faculty of Engineering at Chulalongkorn University on June, 2004.