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APPENDICES

DSC kinetic program

Theory

For any reaction process being followed by DSC the conversion process can be represented by :

$$A \xrightarrow{k} B + \Delta H \qquad (1)$$

Where :   A is the material before conversion

B is the material after conversion

ΔH is the heat absorbed or given off

k is the Arrhenius rate constant

With a power-compensated DSC, the rate of reaction (dx / dt) is measured directly and can be expressed as :

$$dx / dt = k (1-x)^n \qquad (2)$$

Where :   dx / dt is the rate of reaction

x is the fraction reacted

t is time

n is the reaction order

k is the Arrhenius rate constant

The Arrhenius relationship is given by :

$$k = Z \times e^{-E_a / RT} \qquad (3)$$

Where :   Z is the pre-exponential constant

Ea is the activation energy

R is the universal gas constant

T is the absolute temperature

The relationship to which the DSC data are fit is based on the derivations of A.P. Gray[33], the earlier work of Freeman and Carroll[34], and others. This treatment combines equations 1, 2, and 3, above, and assumes nth order reaction kinetics and constant program rate, activation energy, and preexponential constant, yielding :

$$\ln (dx / dT) = \ln(Z) - E_a / RT + n \ln (1-x)$$
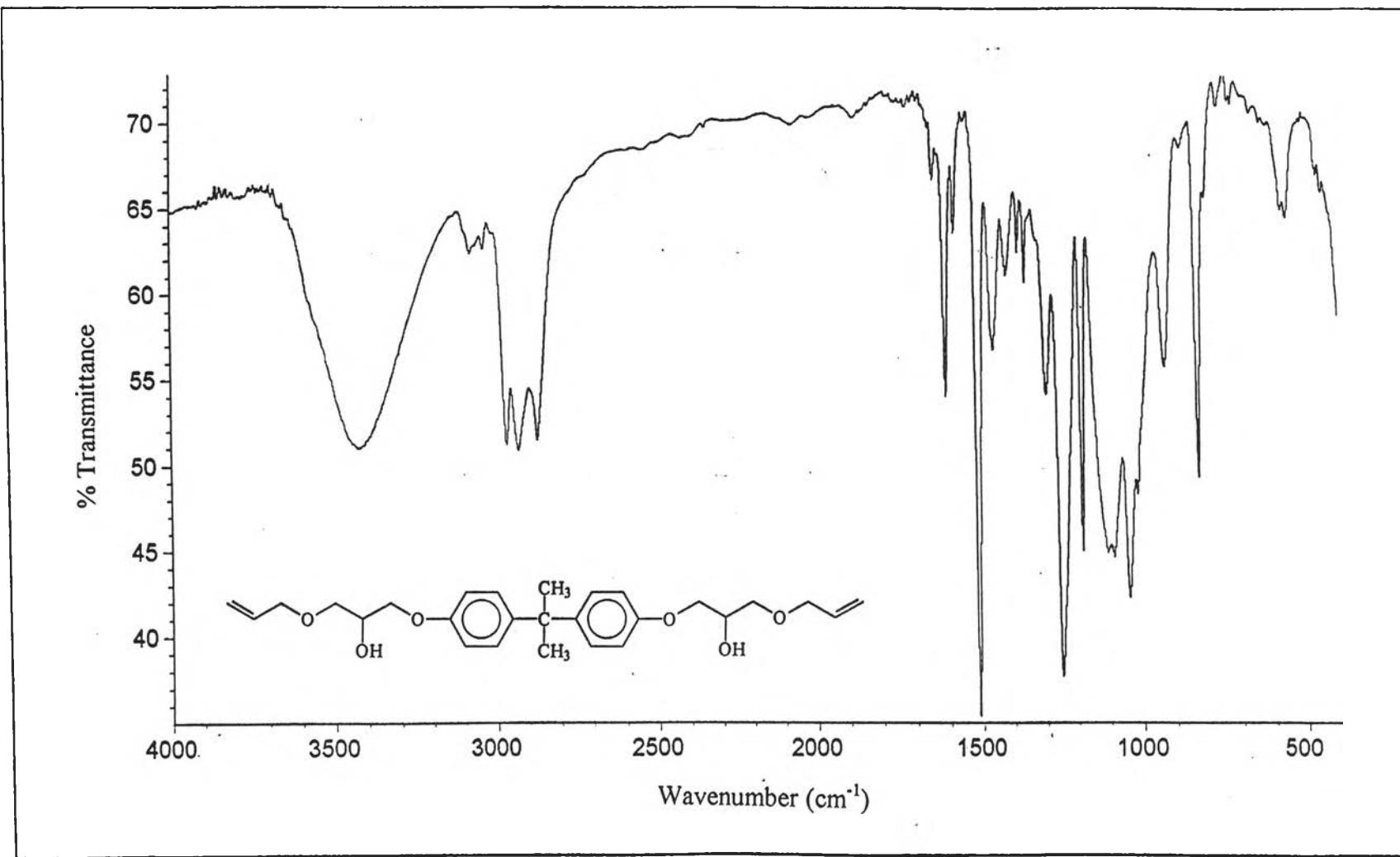
Figure 4   IR spectrum of Bis-(3-allyloxy-2-hydroxy-1-propoxy)diphenoxypropane (3)
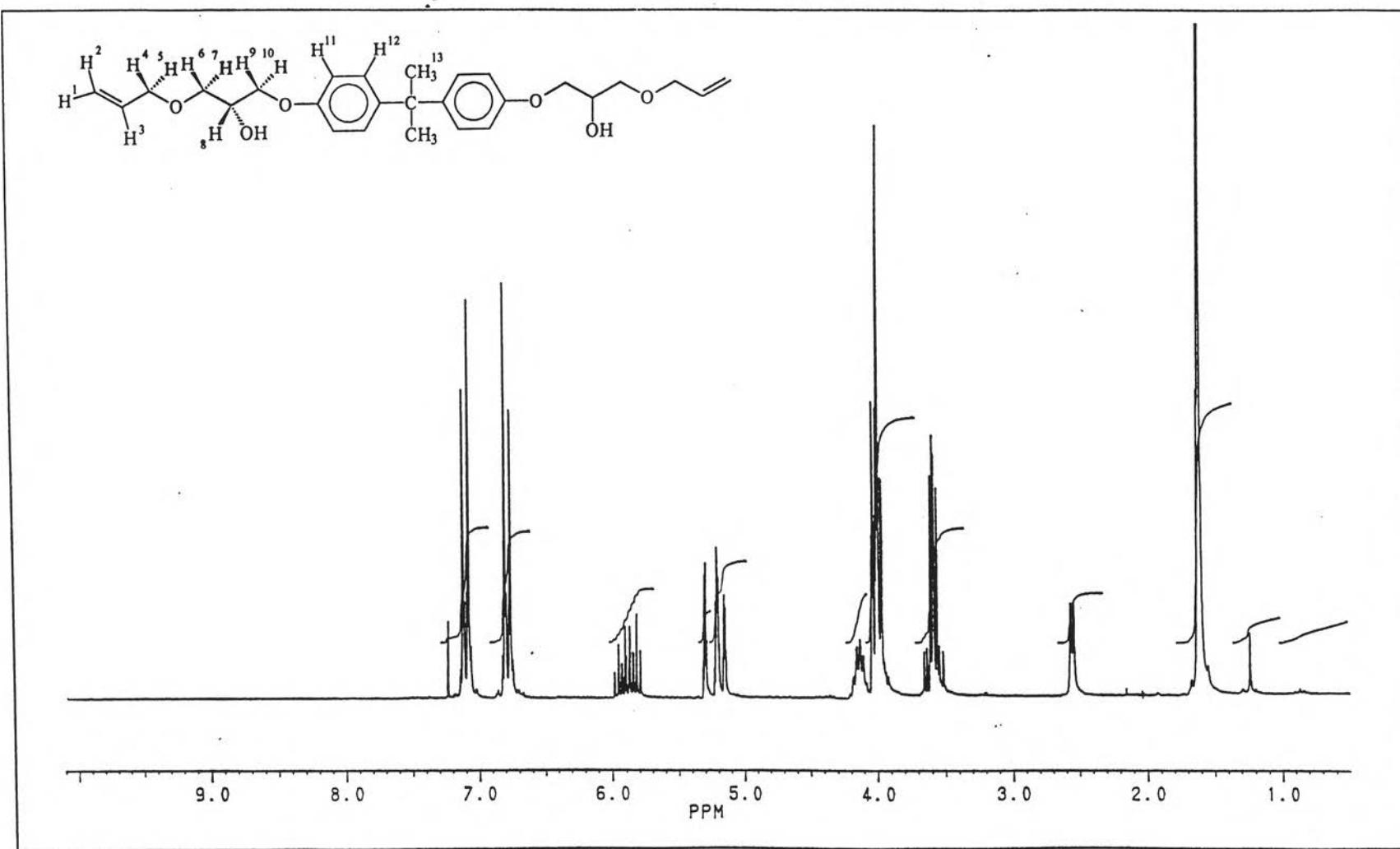
Figure 5 ^{1}H NMR (CDCl$_3$) spectrum of Bis-(3-allyloxy-2-hydroxy-1-propoxy)diphenoxypropane (3)
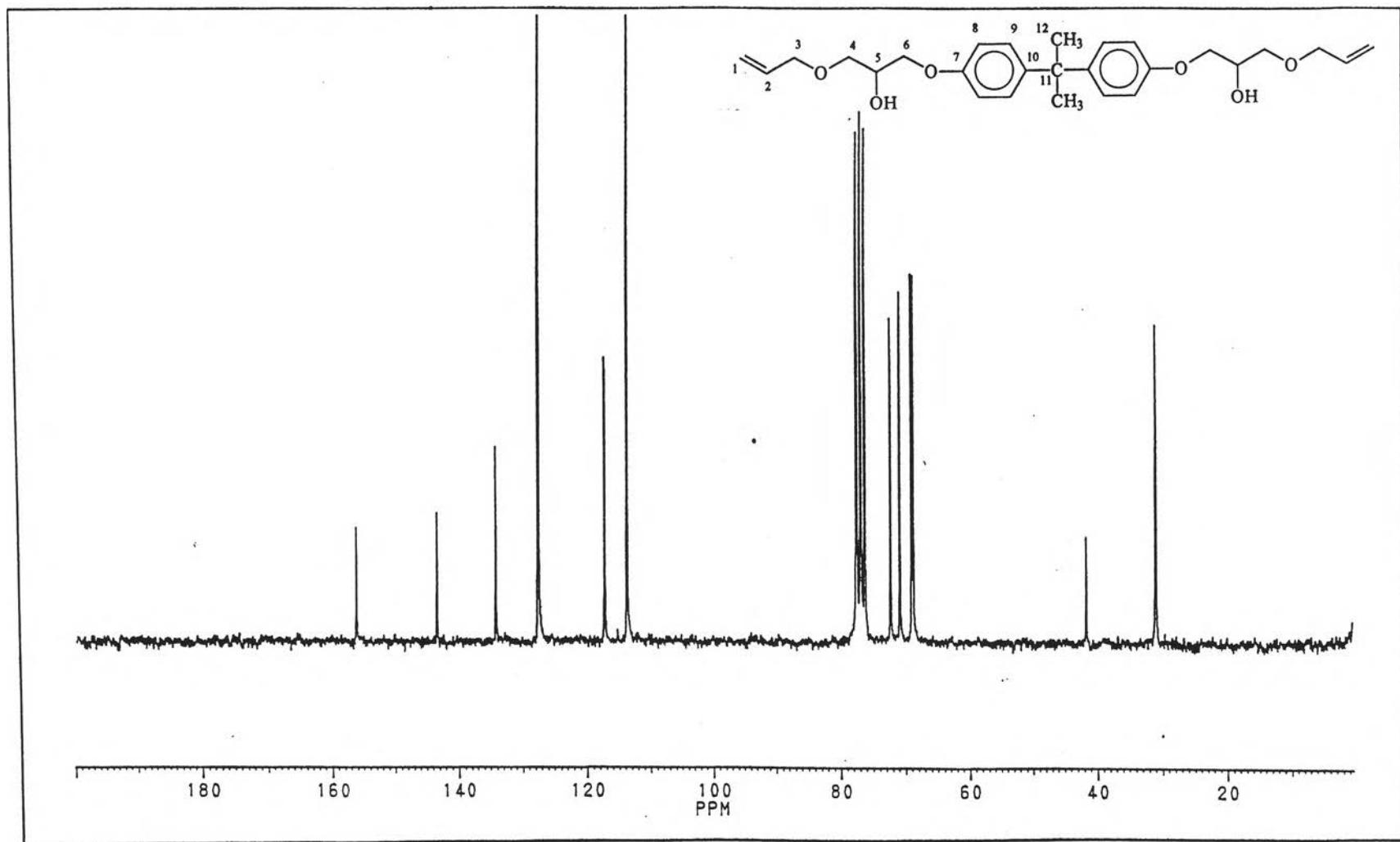
Figure 6 ^{13}C NMR (CDCl$_3$) spectrum of Bis-(3-allyloxy-2-hydroxy-1-propoxy)diphenoxypropane (3)

28034001 87 (2.345) Cm (79:113-(145:164+51))

Scan EI+
4.99e5
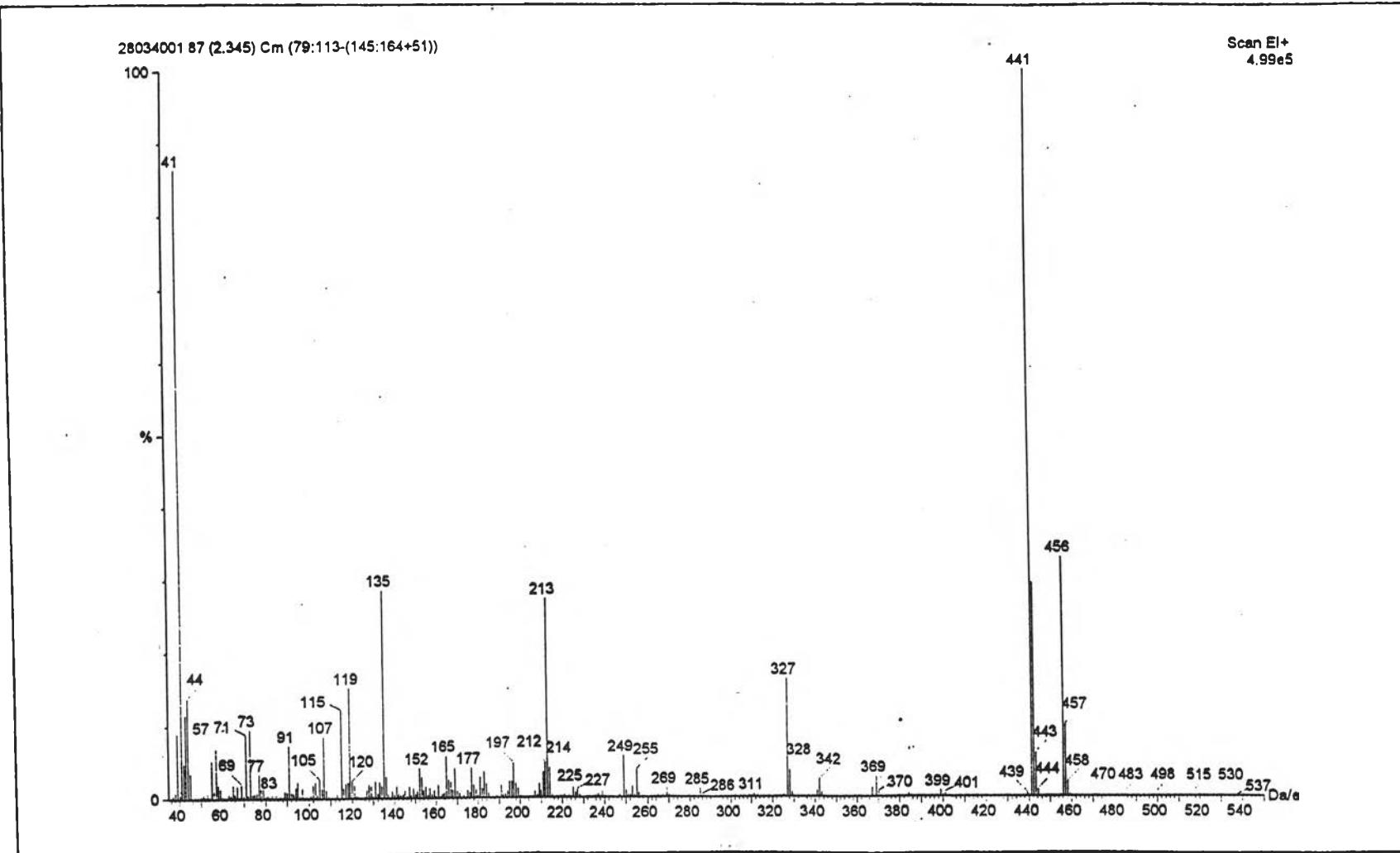
Figure 7   Mass spectrum of Bis-(3-allyloxy-2-hydroxy-1-propoxy)diphenoxypropane (3)
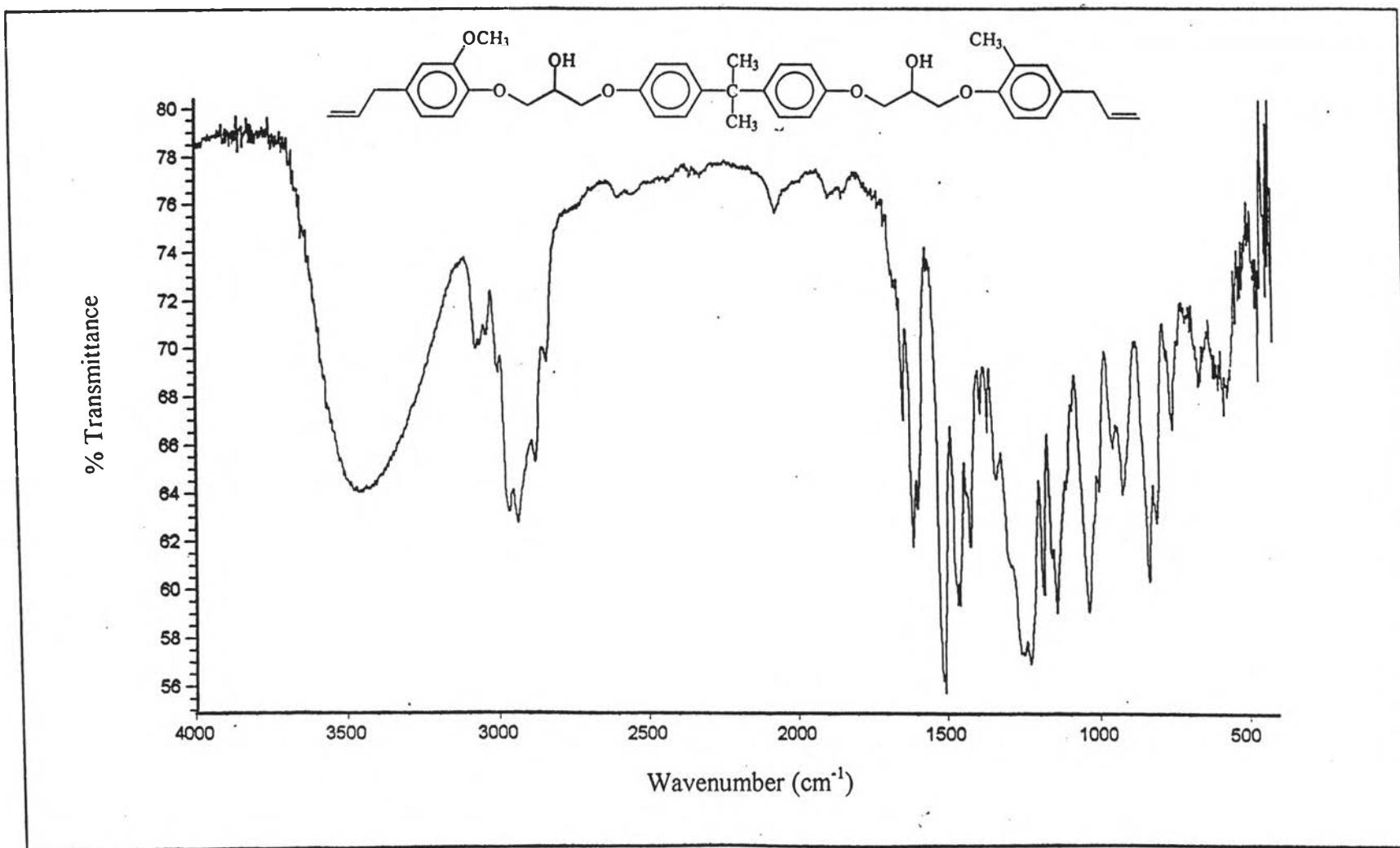
Figure 8   IR spectrum of Bis-[3-(4-allyloxy-2-methoxy)phenoxy-2-hydroxy-1-propoxy)diphenoxypropane (7)
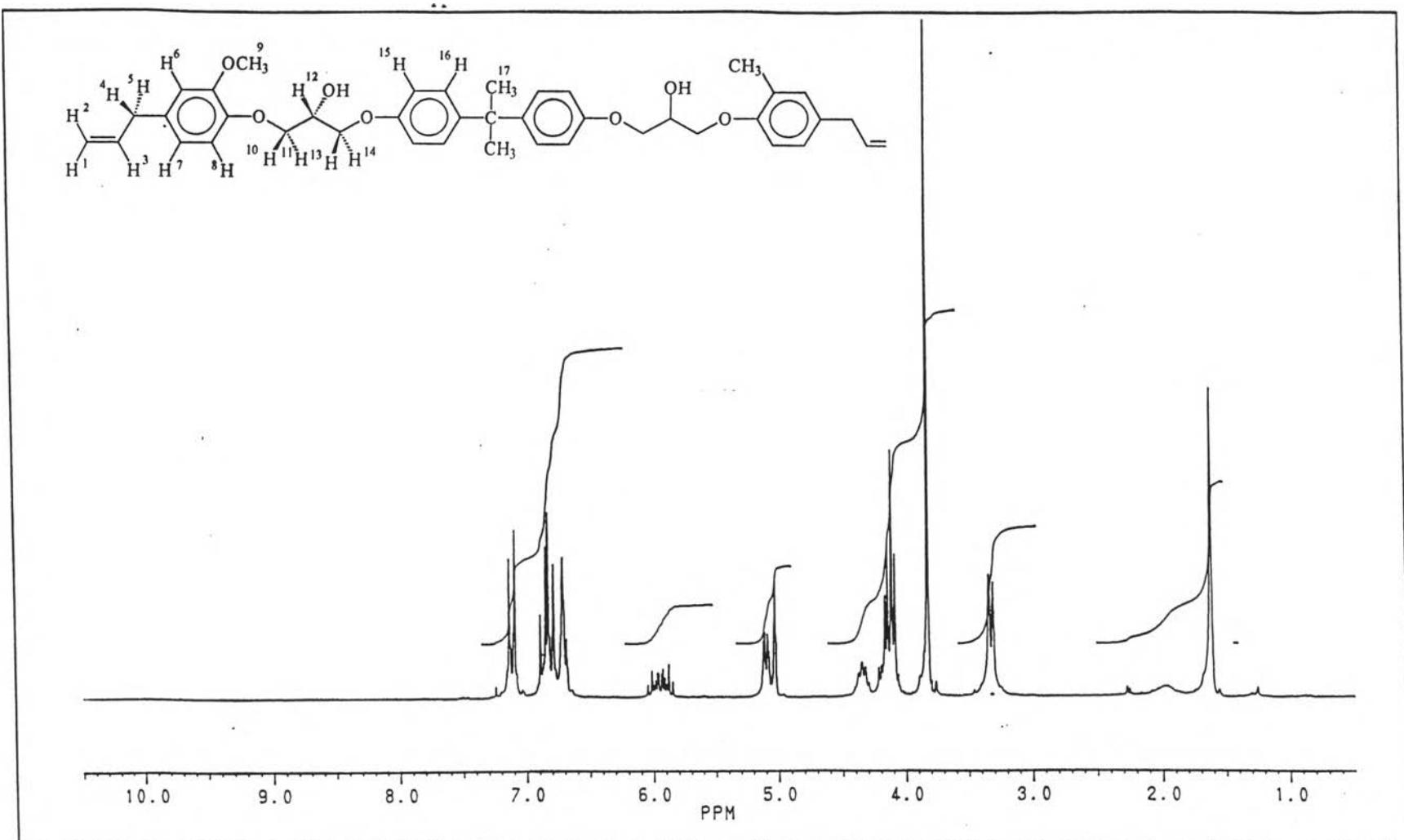
Figure 9   ¹H NMR (CDCl₃) spectrum of Bis-[3-(4-allyloxy-2-methoxy)phenoxy-2-hydroxy-1-propoxy)diphenoxypropane (7)
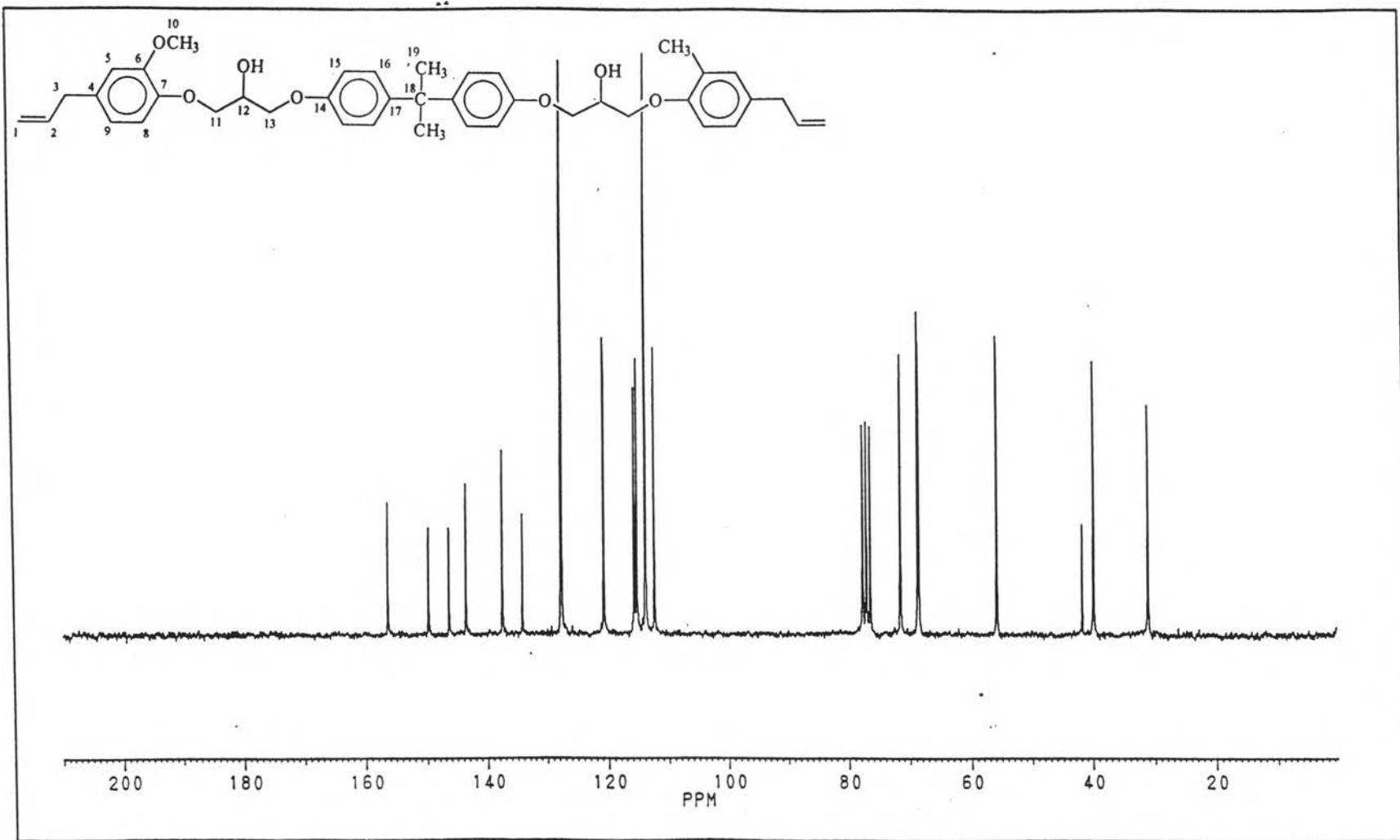
Figure 10 ^{13}C NMR (CDCl$_3$) spectrum of Bis-[3-(4-allyloxy-2-methoxy)phenoxy-2-hydroxy-1-propoxy)diphenoxypropane (7)
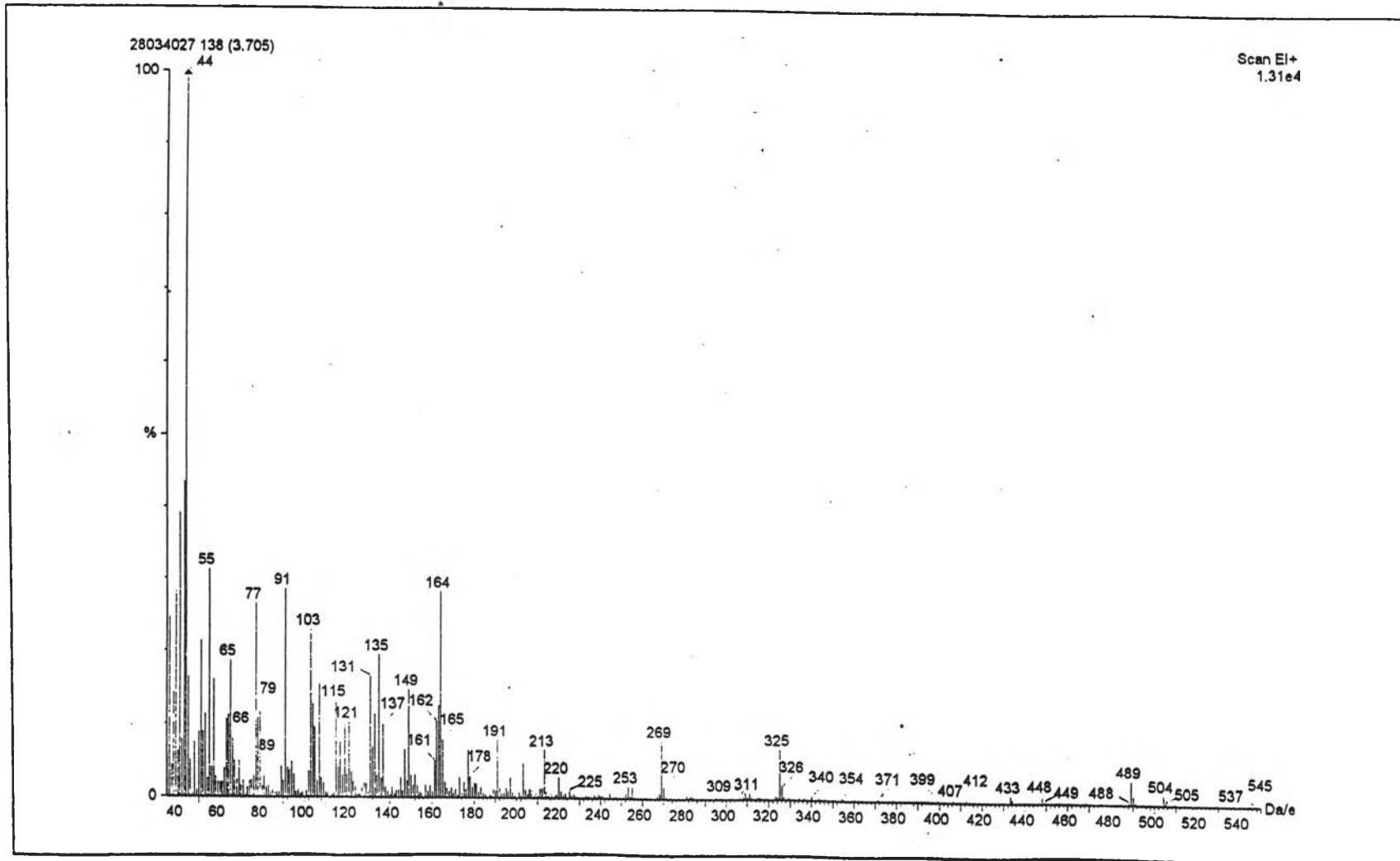
Figure 11   Mass spectrum of Bis-[3-(4-allyloxy-2-methoxy)phenoxy-2-hydroxy-1-propoxy)diphenoxypropane (7)
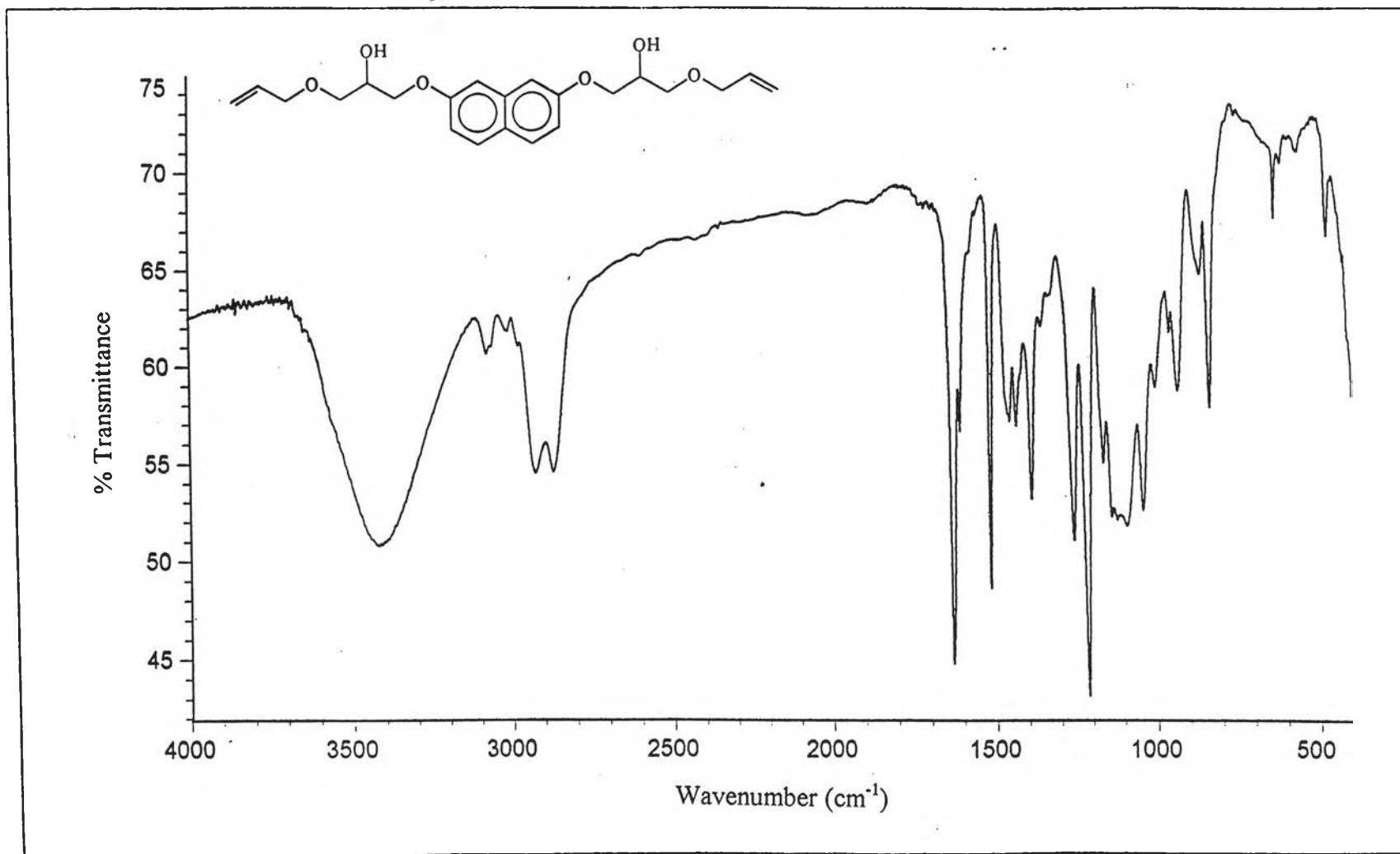
Figure 12 IR spectrum of 2,7-Bis-(3-allyloxy-2-hydroxy-1-propoxy)naphthalene (9)
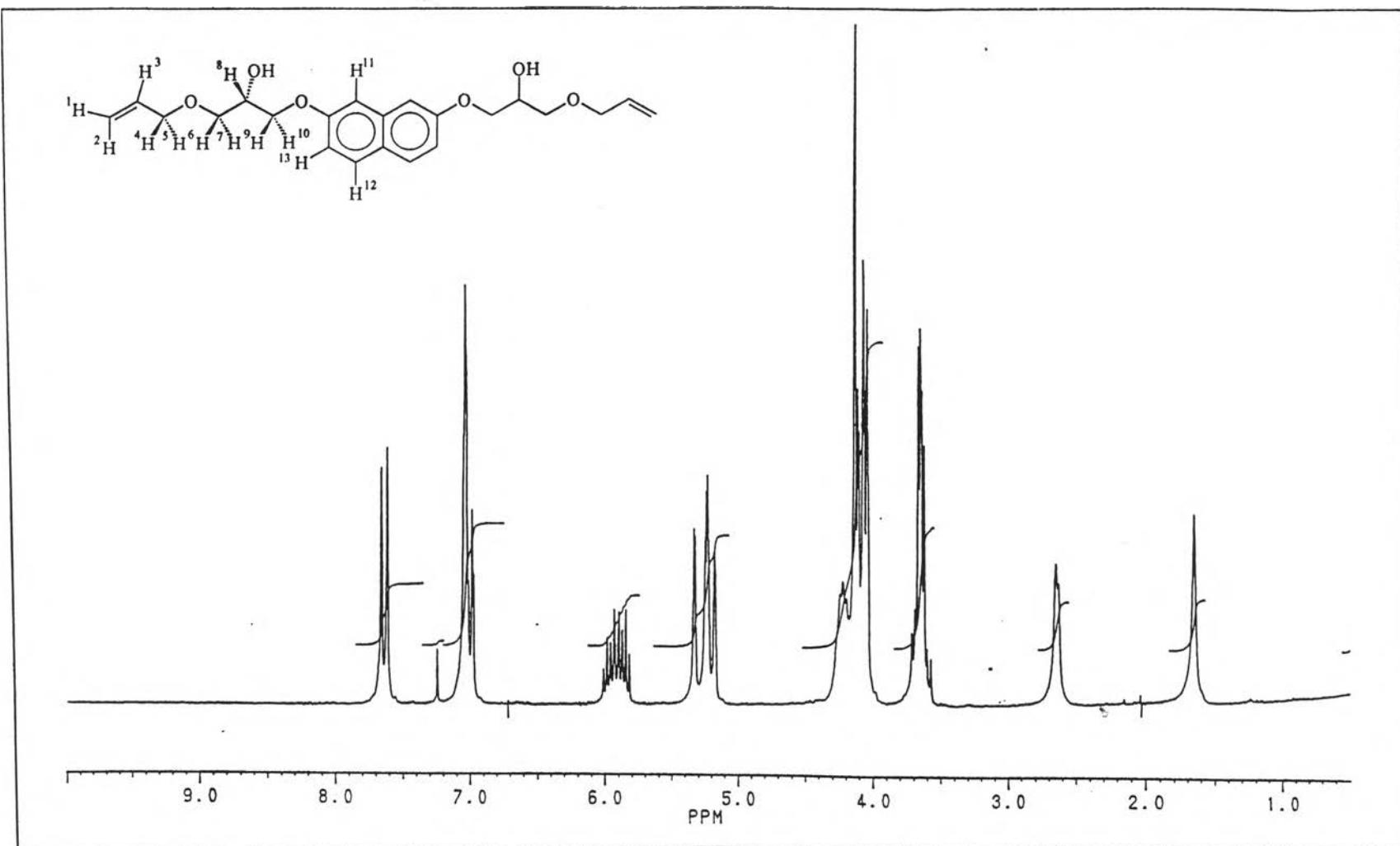
Figure 13 ^{1}H NMR (CDCl$_3$) spectrum of 2,7-Bis-(3-allyloxy-2-hydroxy-1-propoxy)naphthalene (9)
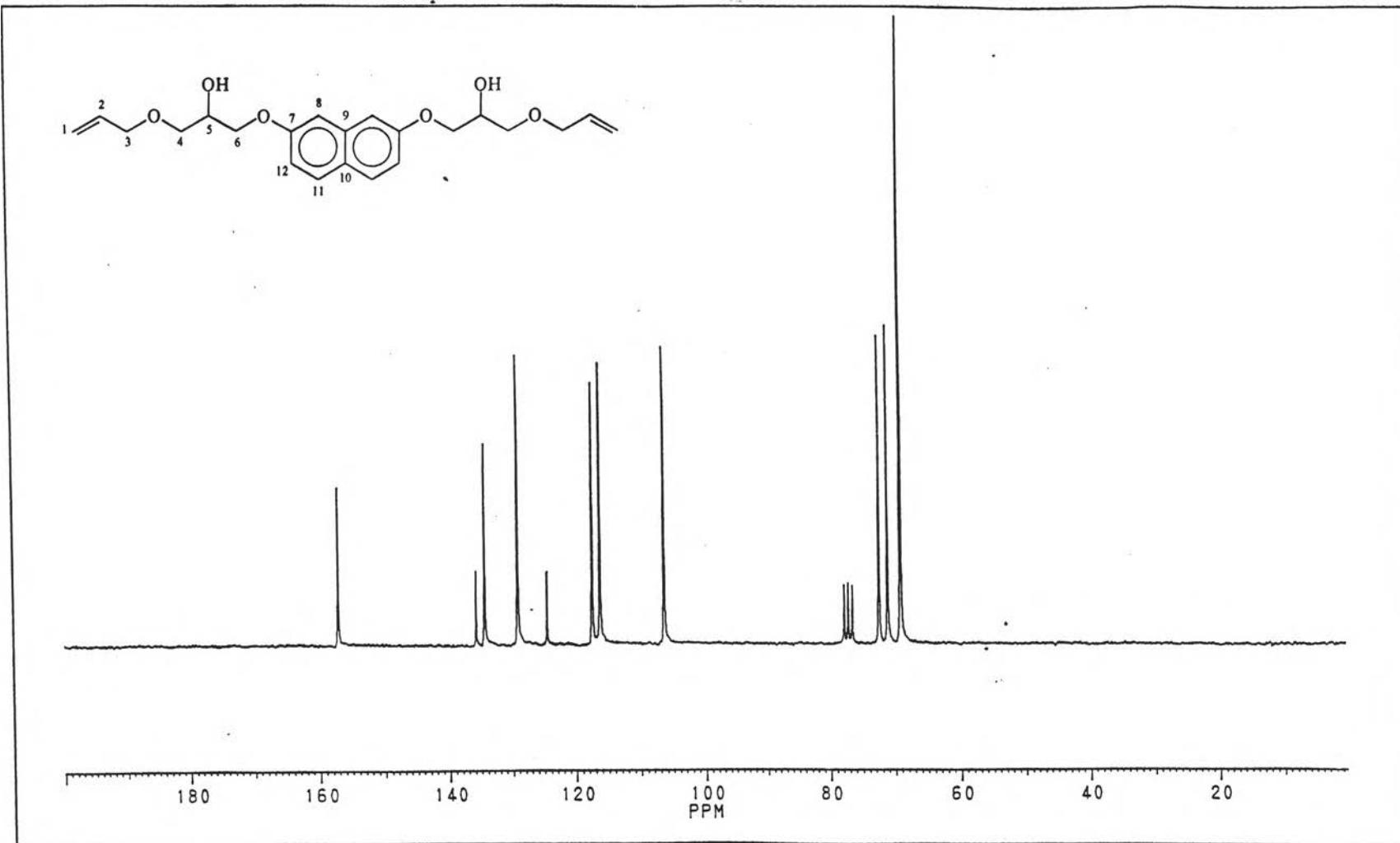
Figure 14 ^{13}C NMR (CDCl$_3$) spectrum of 2,7-Bis-(3-allyloxy-2-hydroxy-1-propoxy)naphthalene (9)
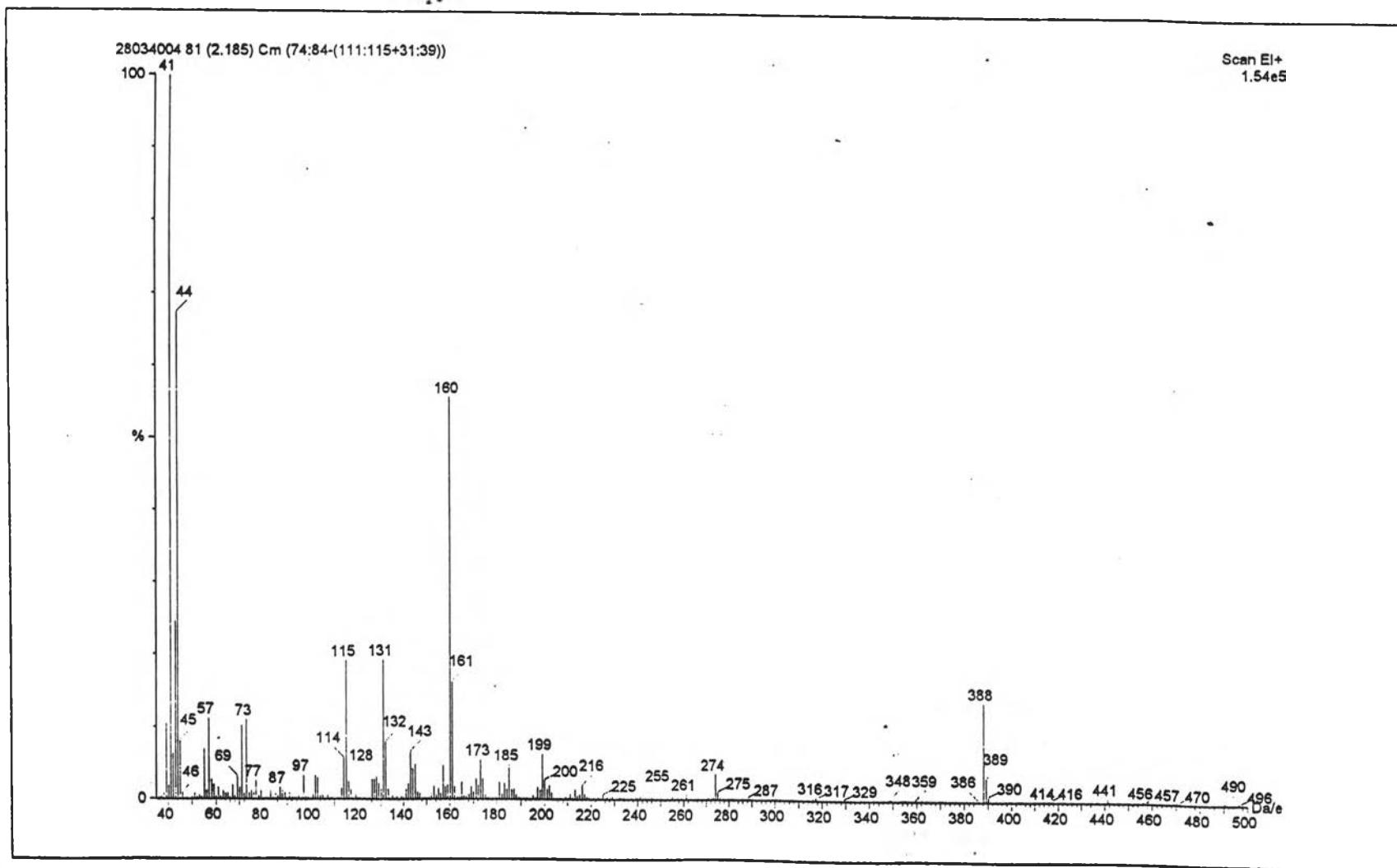
Figure 15   Mass spectrum of 2,7-Bis-(3-allyloxy-2-hydroxy-1-propoxy)naphthalene (9)
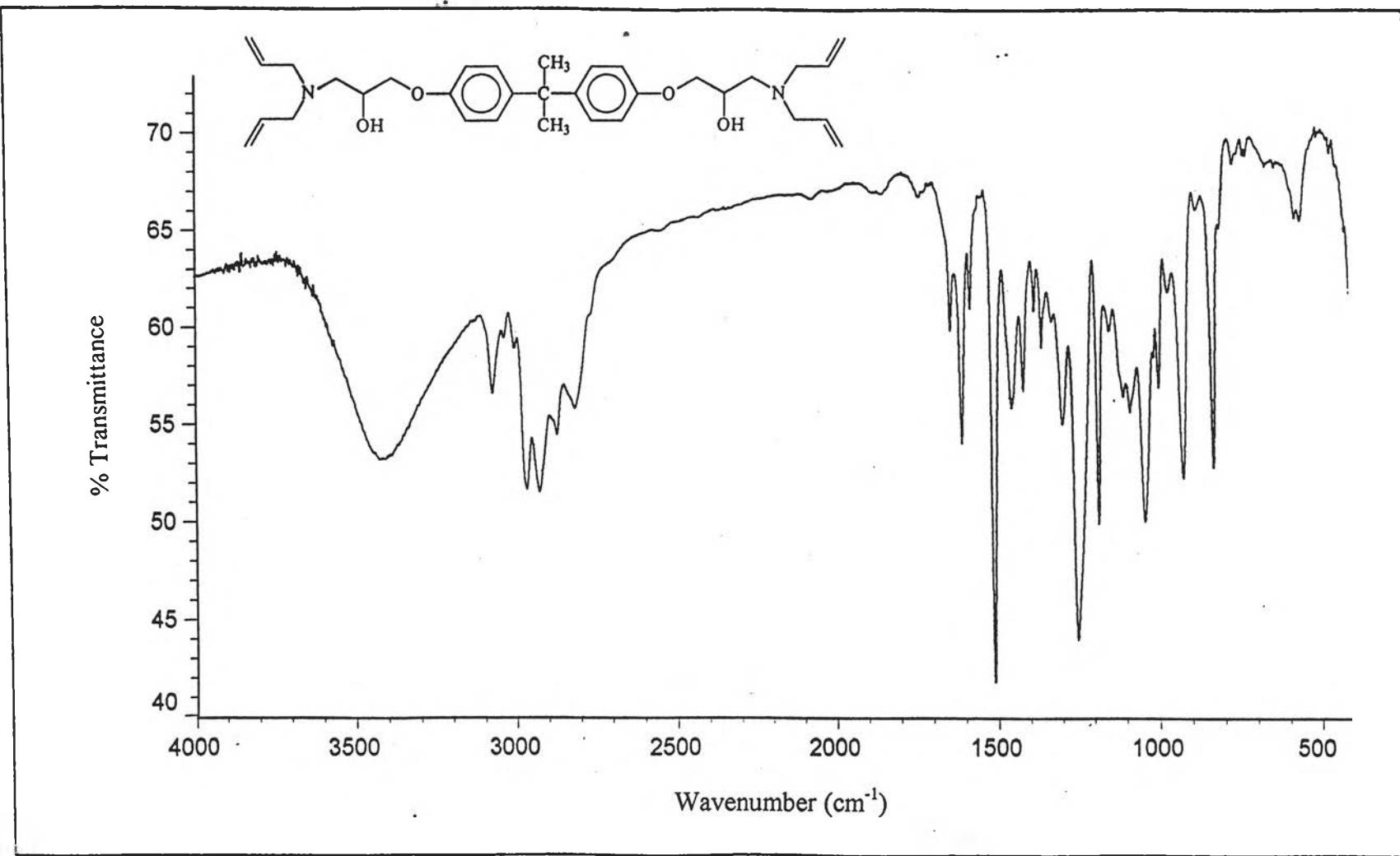
Figure 16   IR spectrum of Bis-(3-N,N-diallylamino-2-hydroxy-1-propoxy)diphenoxypropane (11)
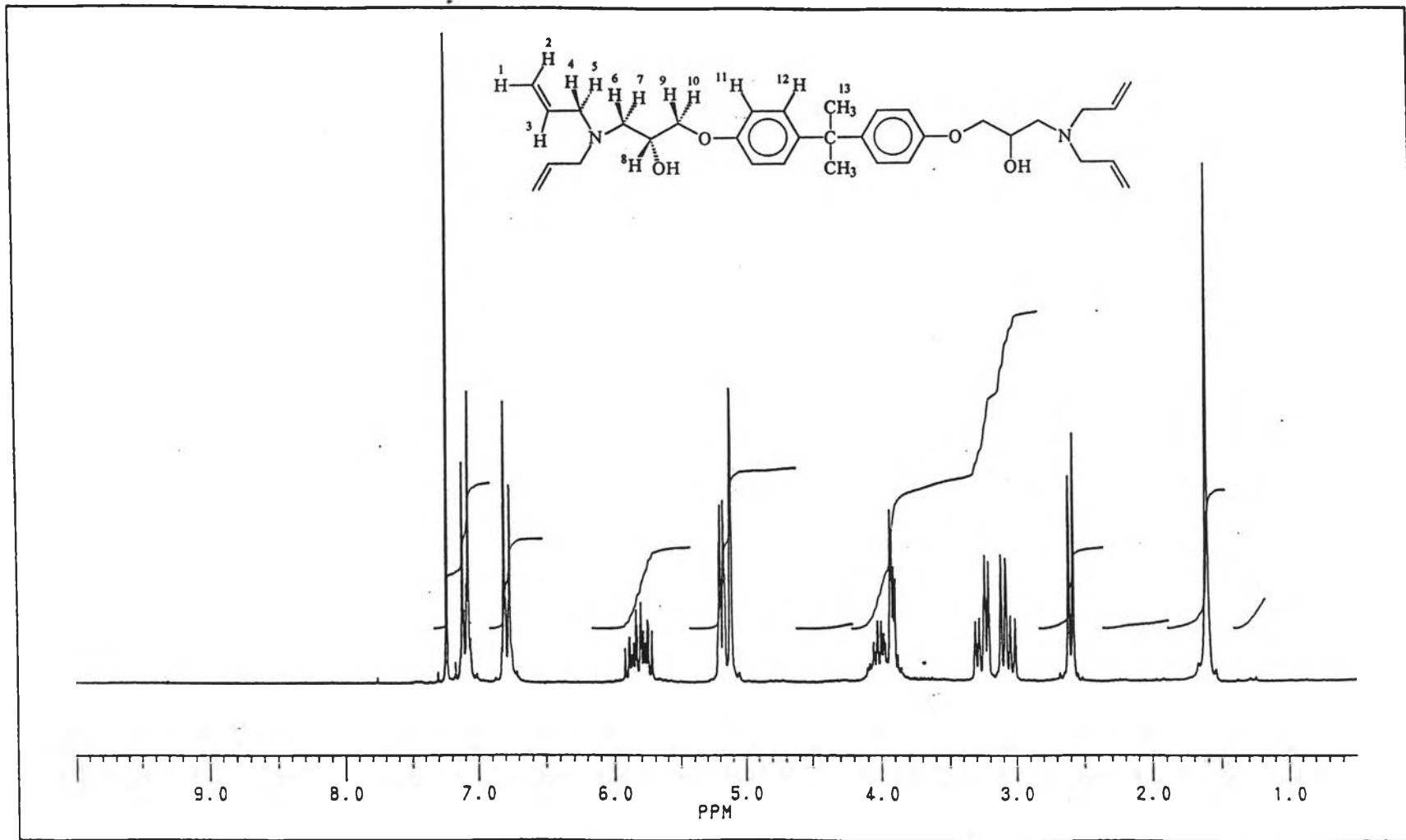
Figure 17   ¹H NMR (CDCl₃) spectrum of Bis-(3-N,N-diallylamino-2-hydroxy-1-propoxy)diphenoxypropane (11)
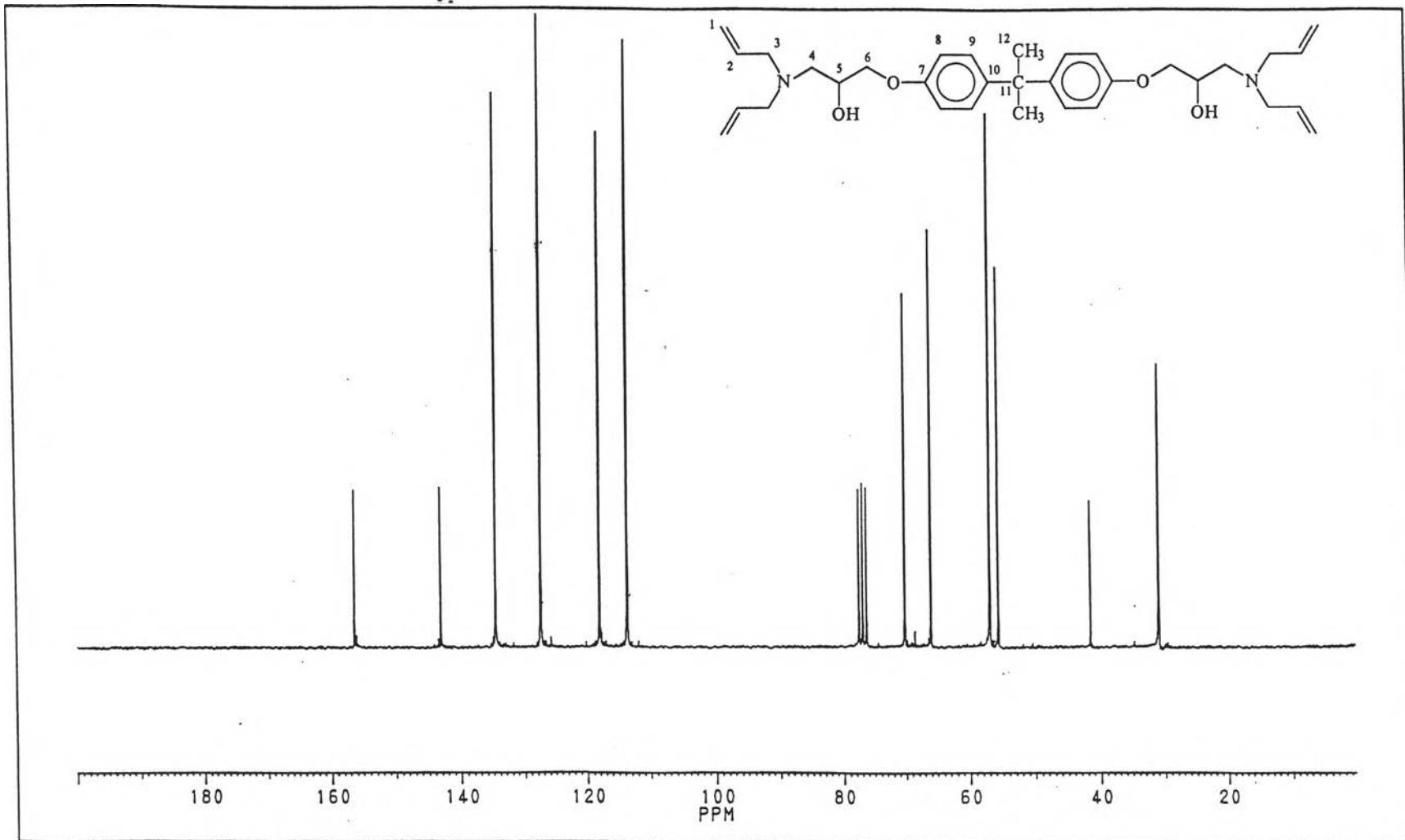
Figure 18 ^{13}C NMR (CDCl$_3$) spectrum of Bis-(3-N,N-diallylamino-2-hydroxy-1-propoxy)diphenoxypropane (11)
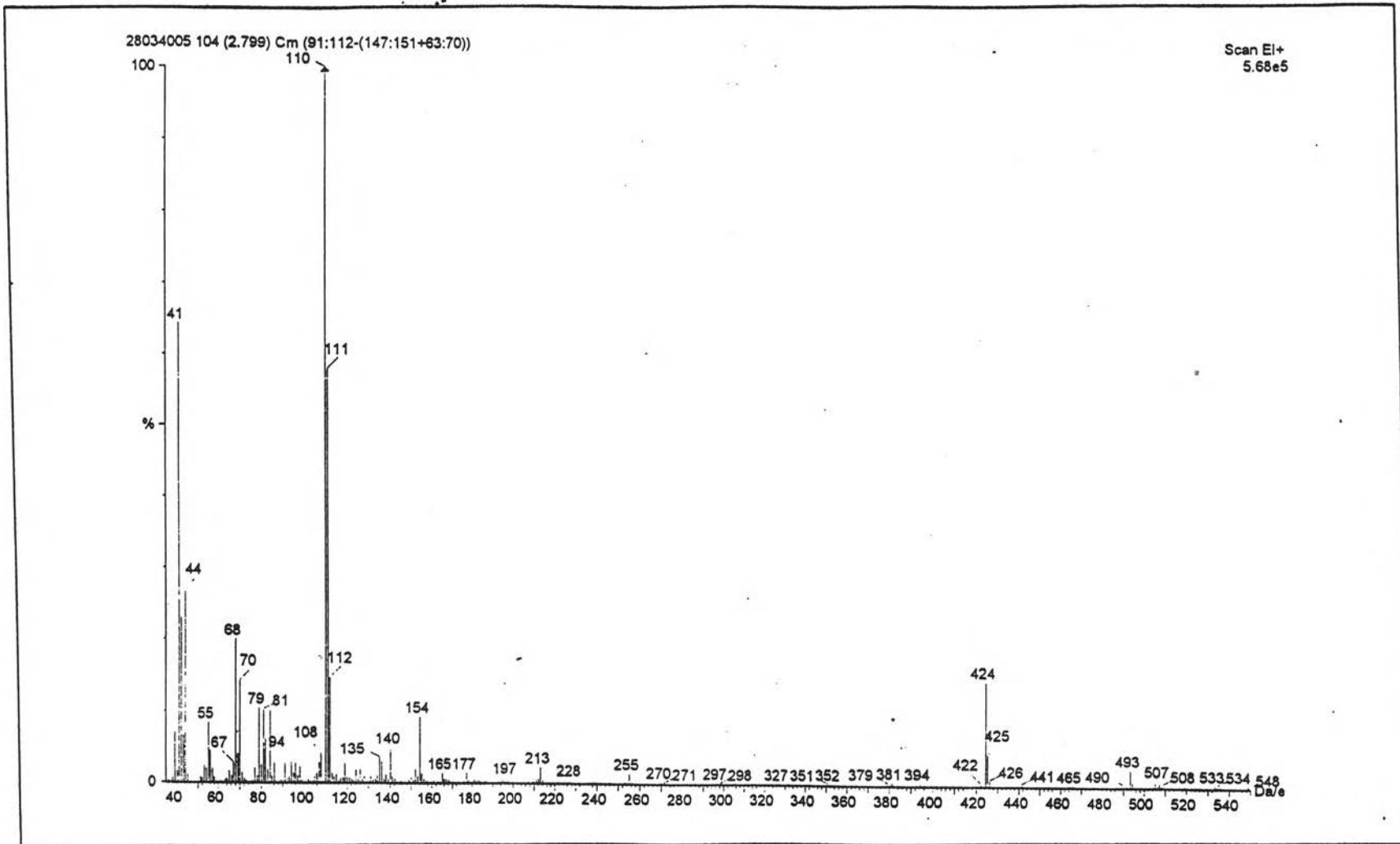
Figure 19   Mass spectrum of Bis-(3-N,N-diallylamino-2-hydroxy-1-propoxy)diphenoxypropane (11)
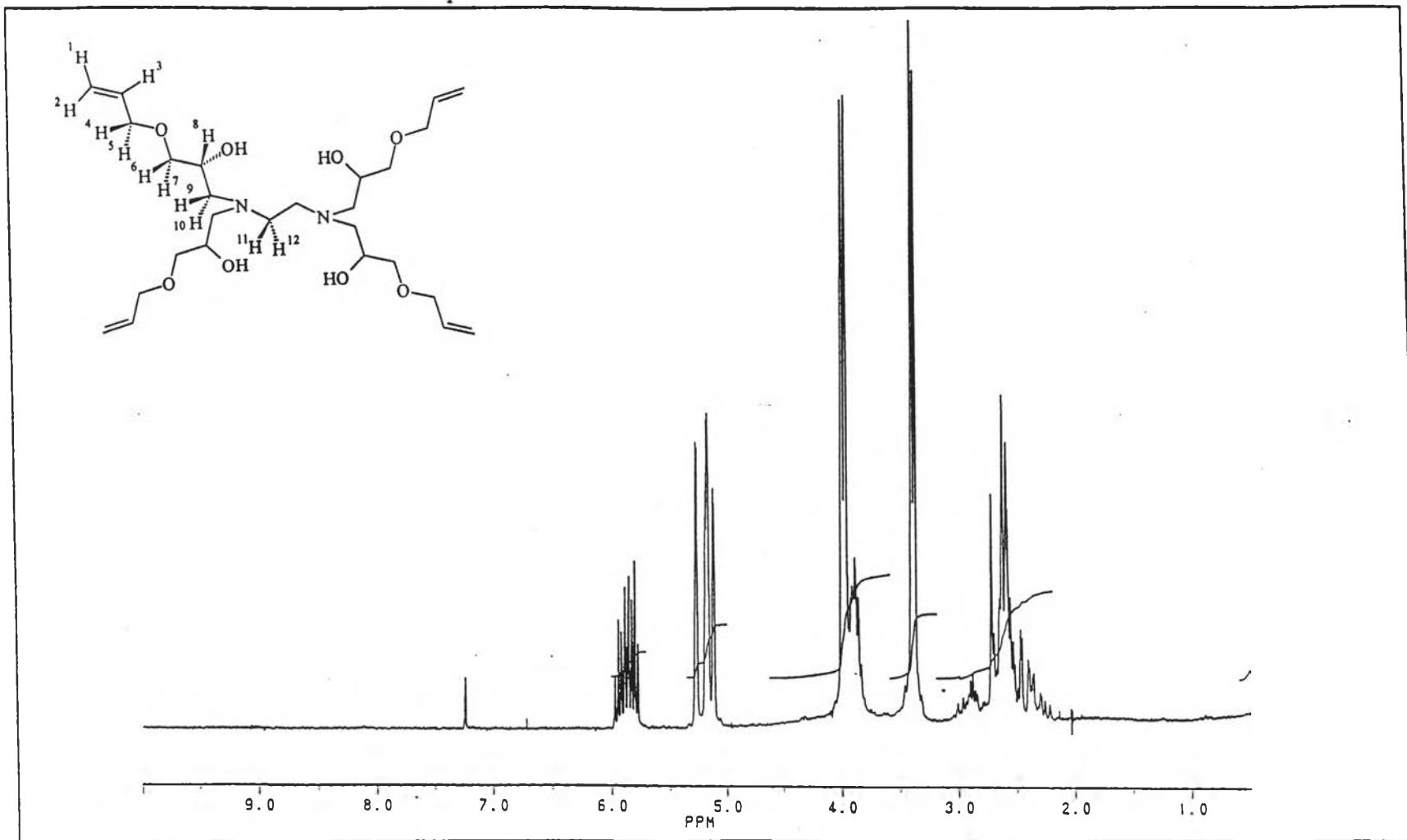
Figure 20 ^{1}H NMR (CDCl$_3$) spectrum of N,N,N′,N′-tetrakis-(3-allyloxy-2-hydroxy-1-propoxy)ethylenediamine (13)
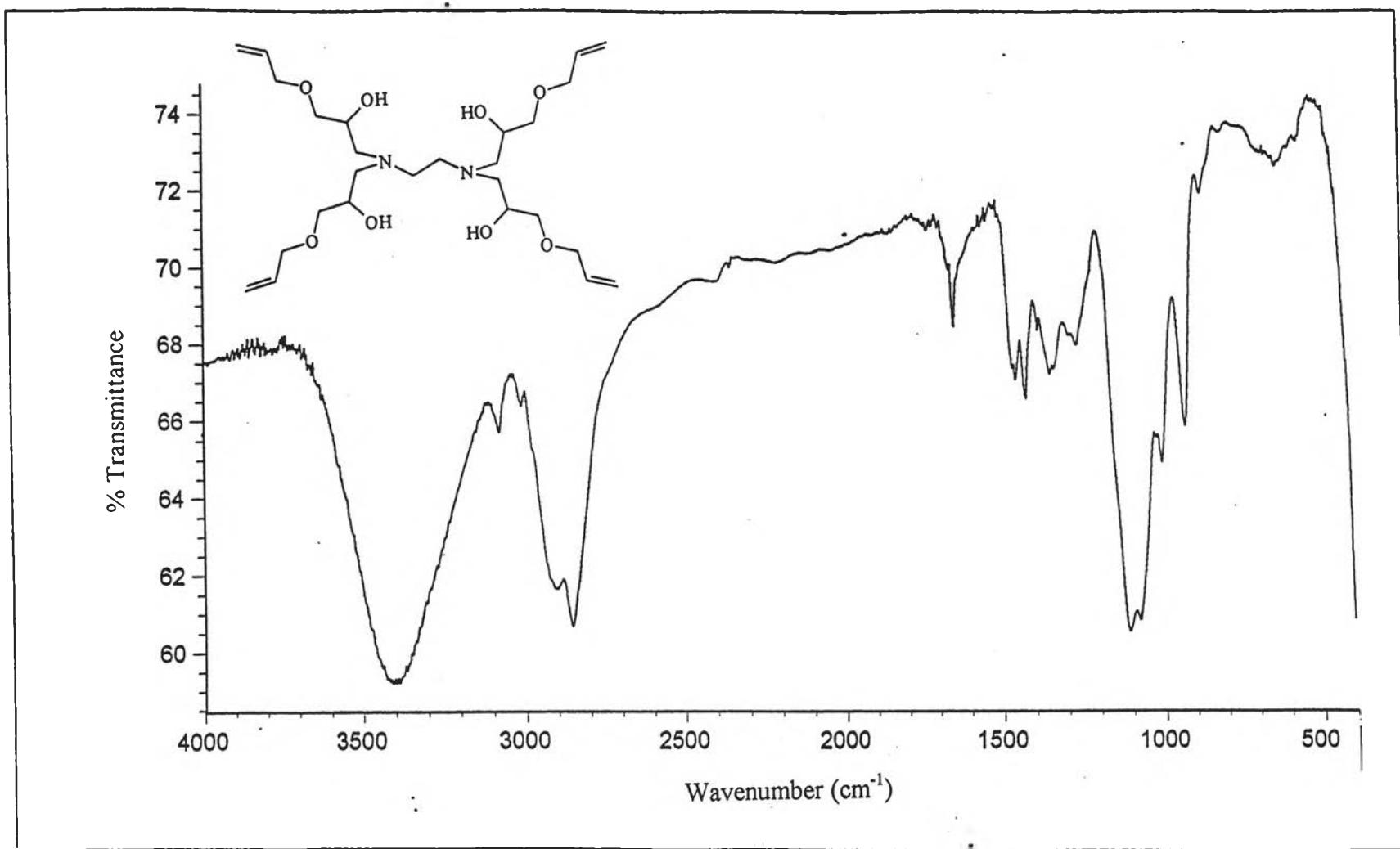
Figure 21   IR spectrum of N,N,N′,N′-tetrakis-(3-allyloxy-2-hydroxy-1-propoxy)ethylenediamine (13)
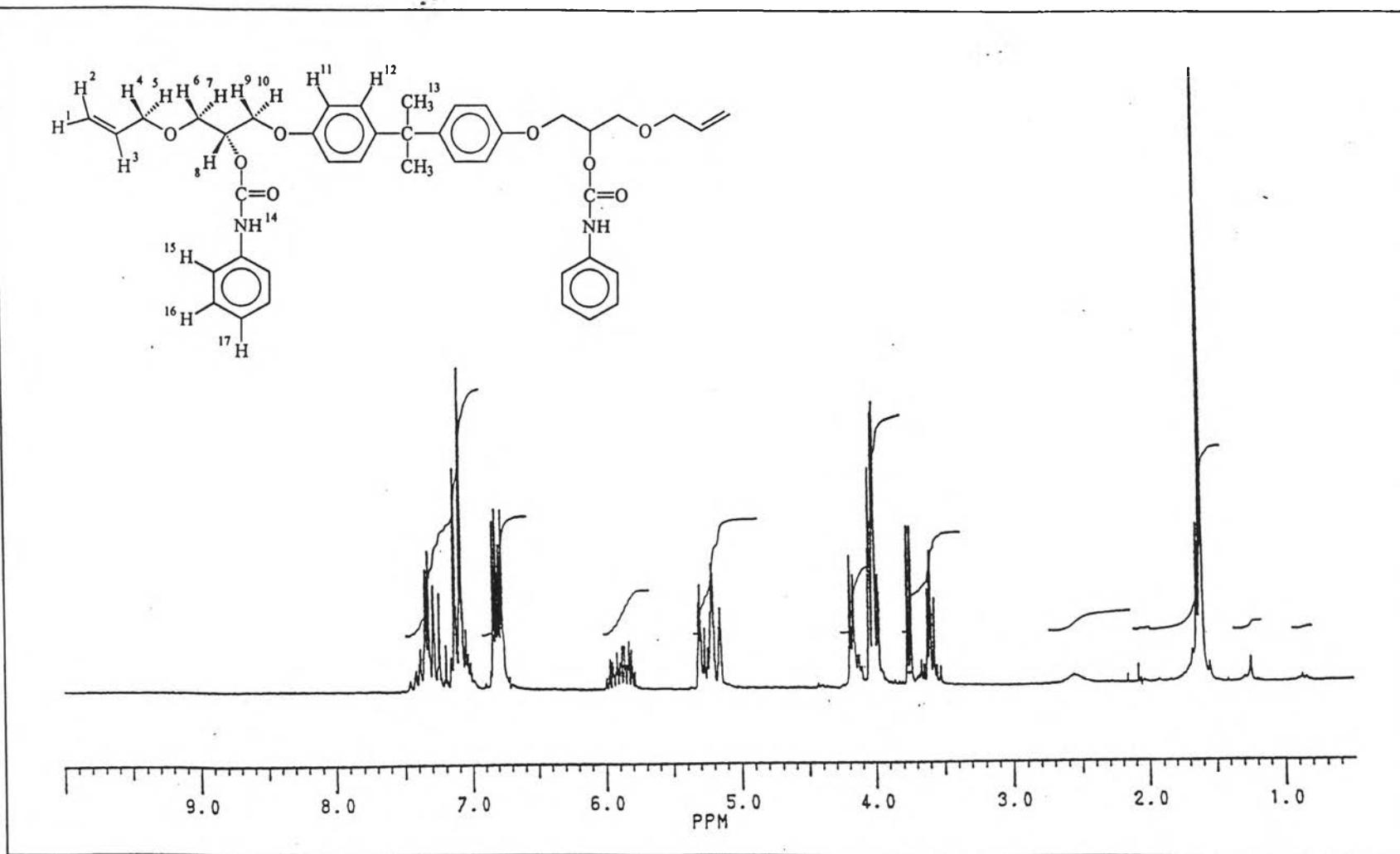
Figure 22   ¹H NMR (CDCl₃) spectrum of Carbamate Derivative (15)
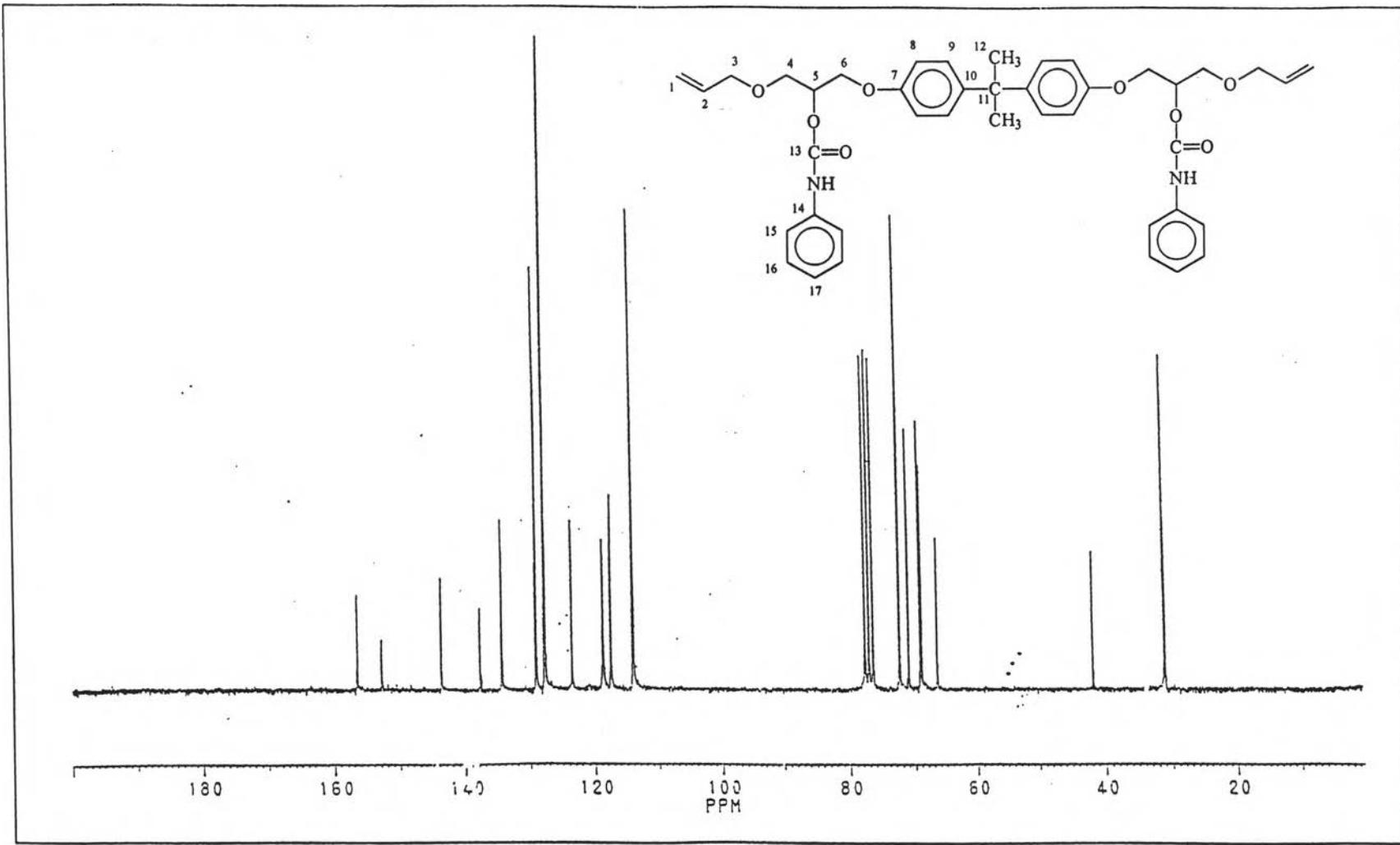
Figure 23 ^{13}C NMR (CDCl$_3$) spectrum of Carbamate Derivative (**15**)
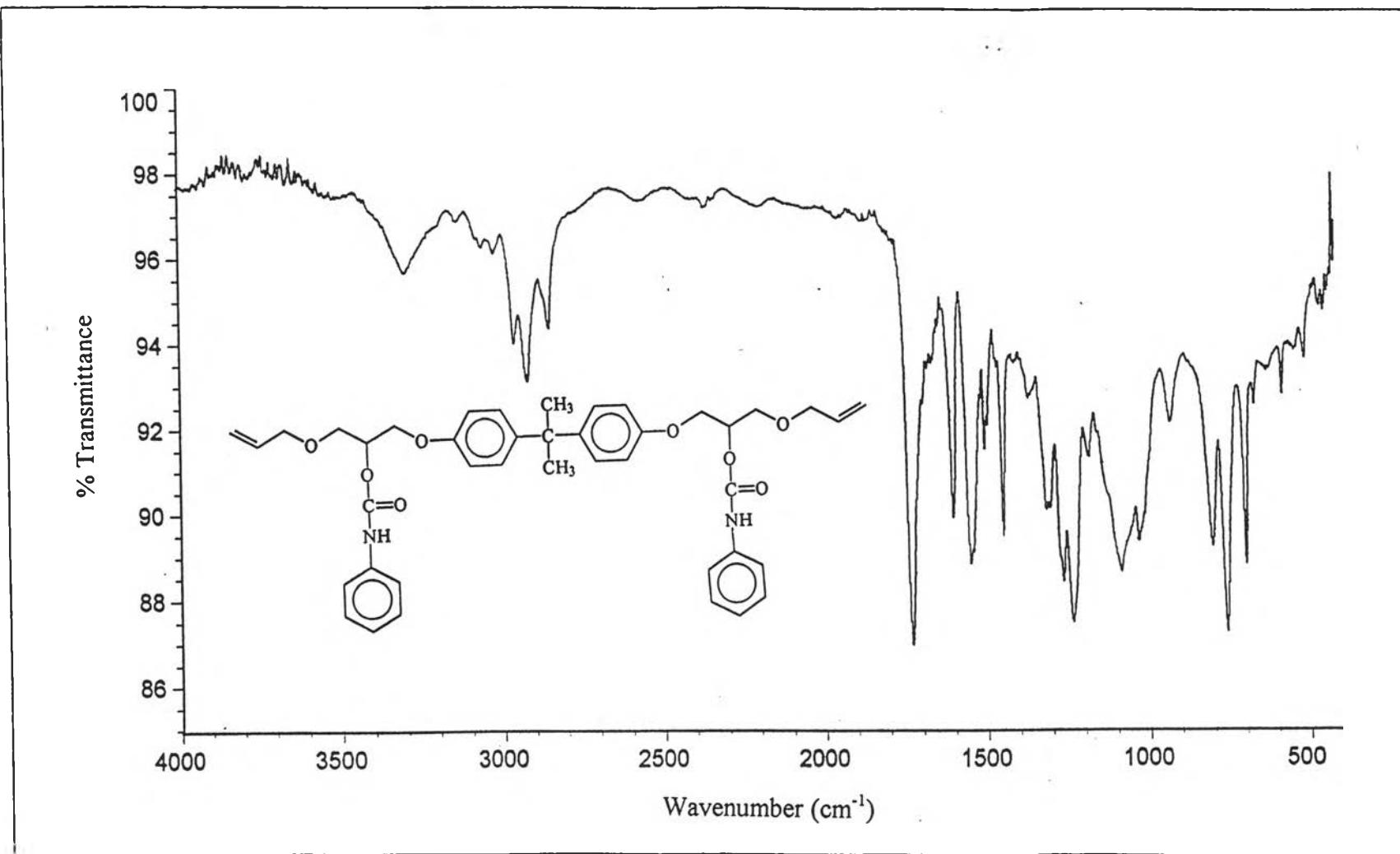
Figure 24   IR spectrum of Carbamate Derivative (15)
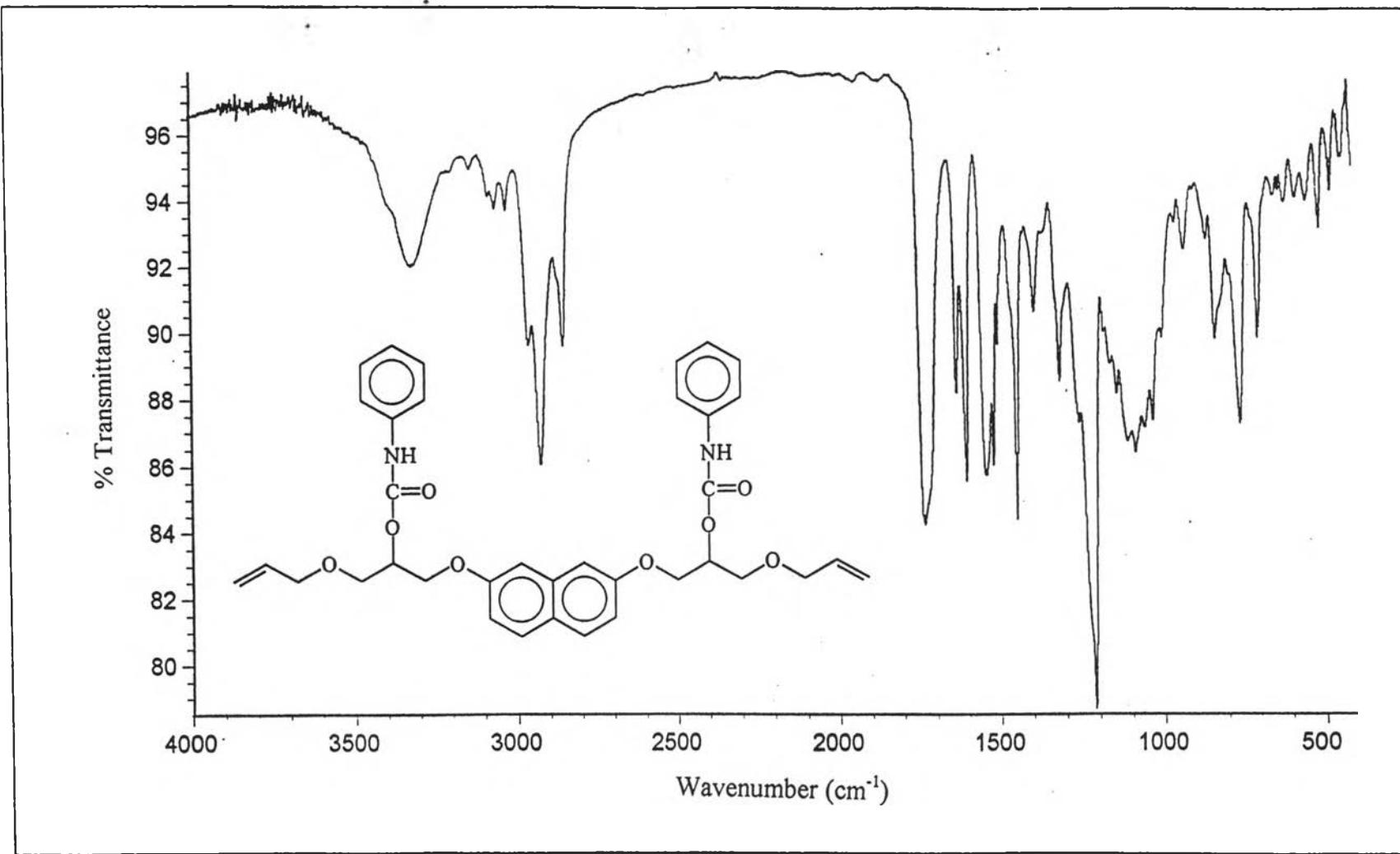
Figure 25   IR spectrum of Carbamate Derivative (16)
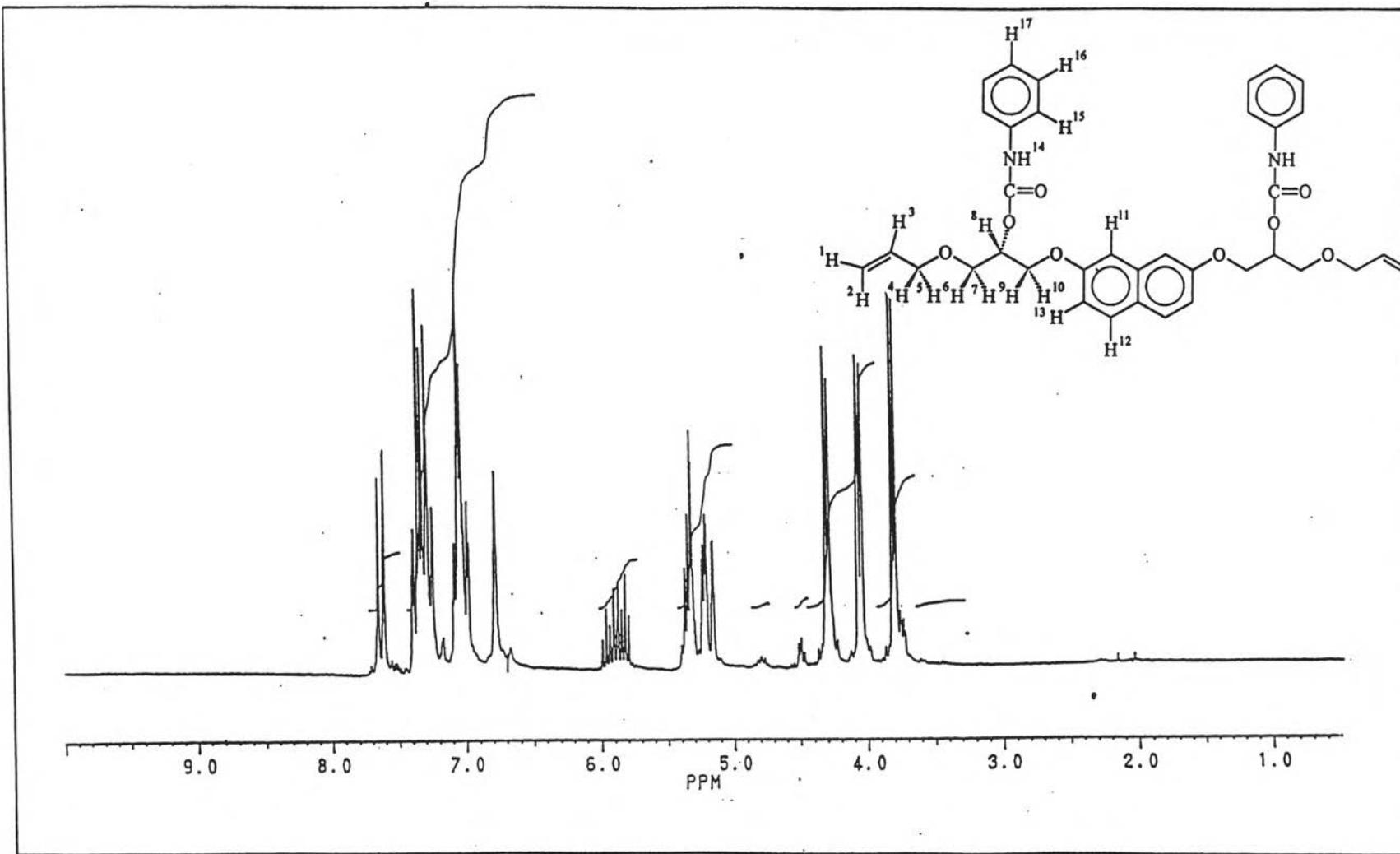
Figure 26 ^{1}H NMR (CDCl$_3$) spectrum of Carbamate Derivative (16)
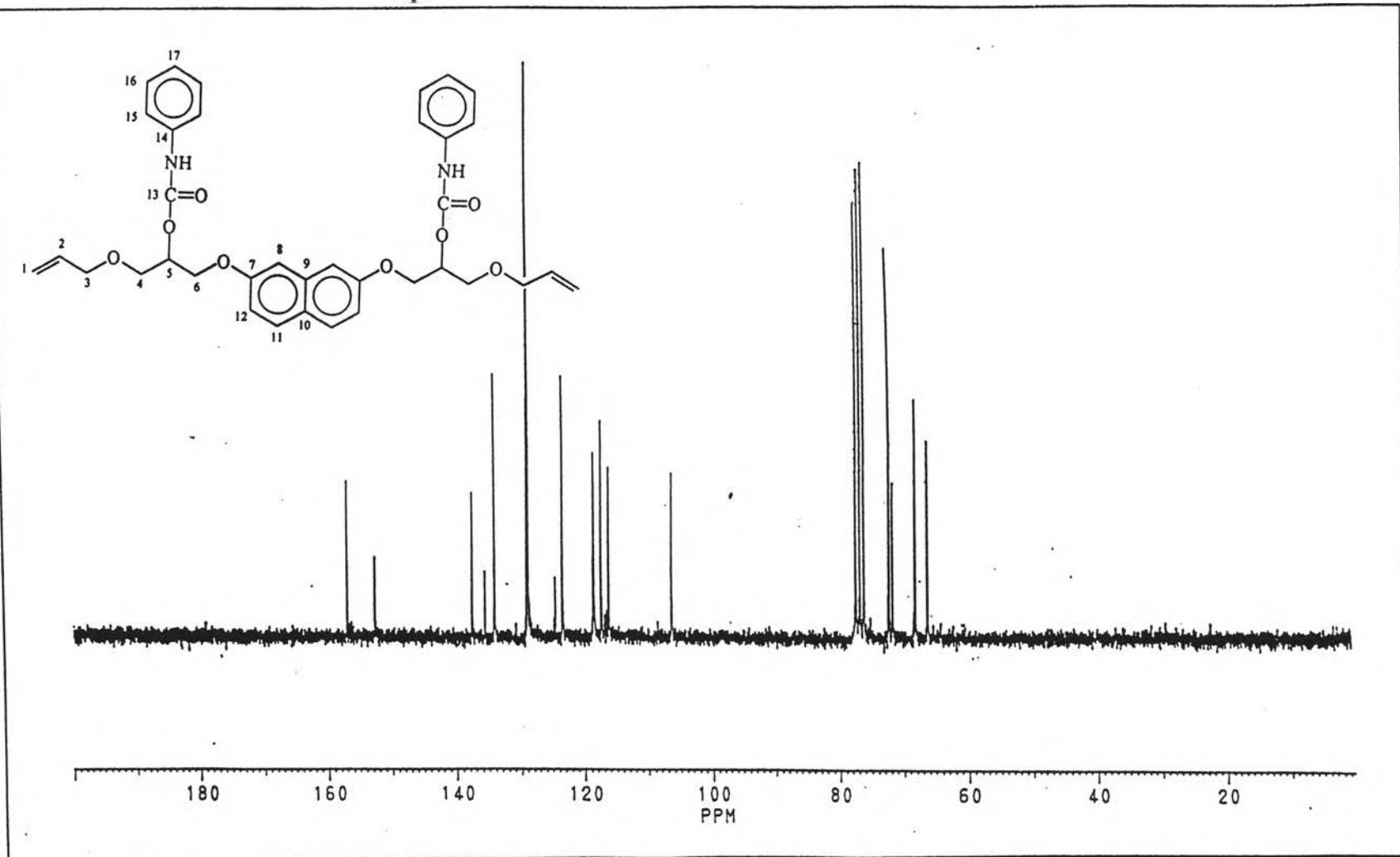
Figure 27 ^{13}C NMR (CDCl$_3$) spectrum of Carbamate Derivative (16)
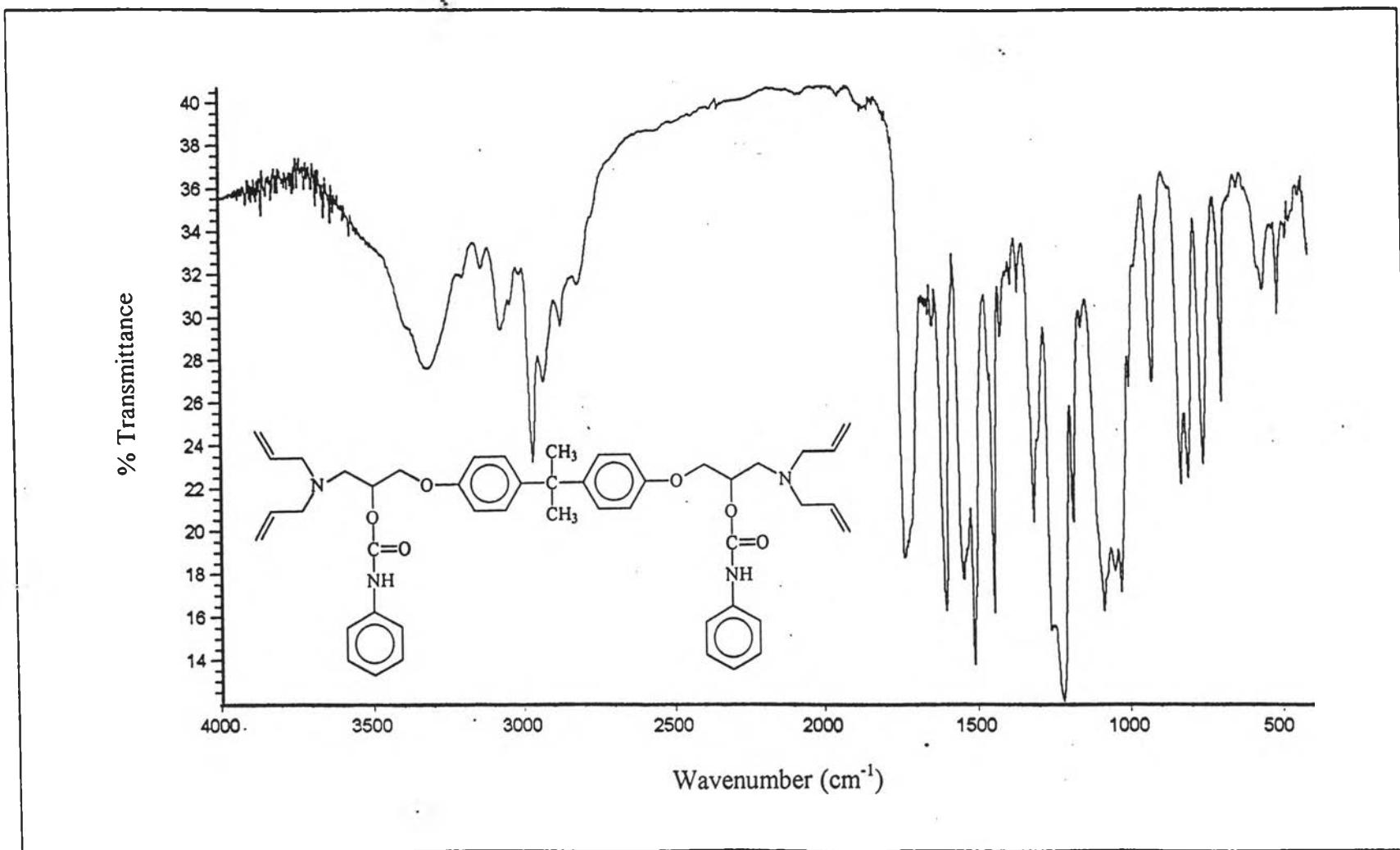
Figure 28   IR spectrum of Carbamate Derivative (17)
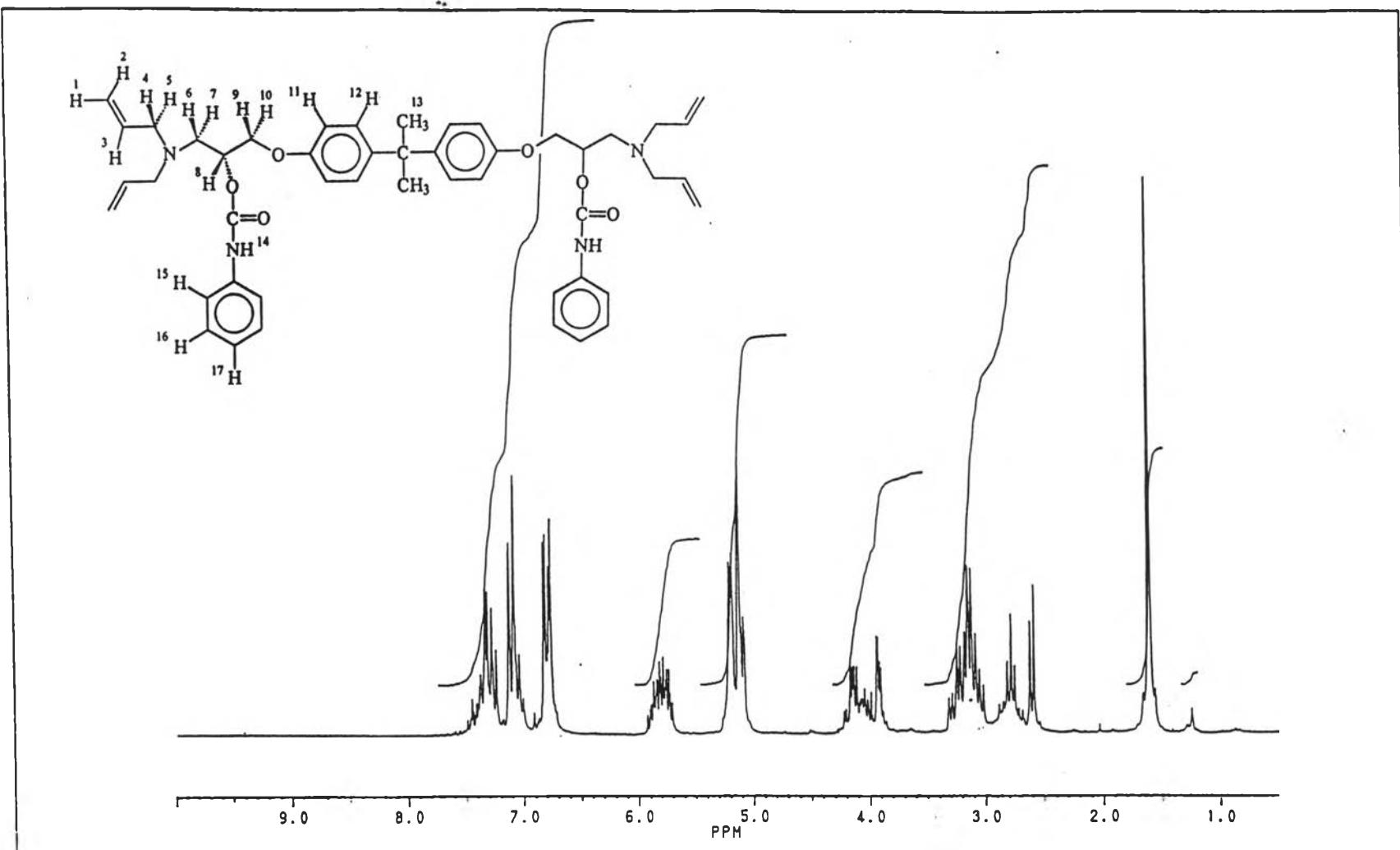
Figure 29 ^{1}H NMR (CDCl$_3$) spectrum of Carbamate Derivative (17)
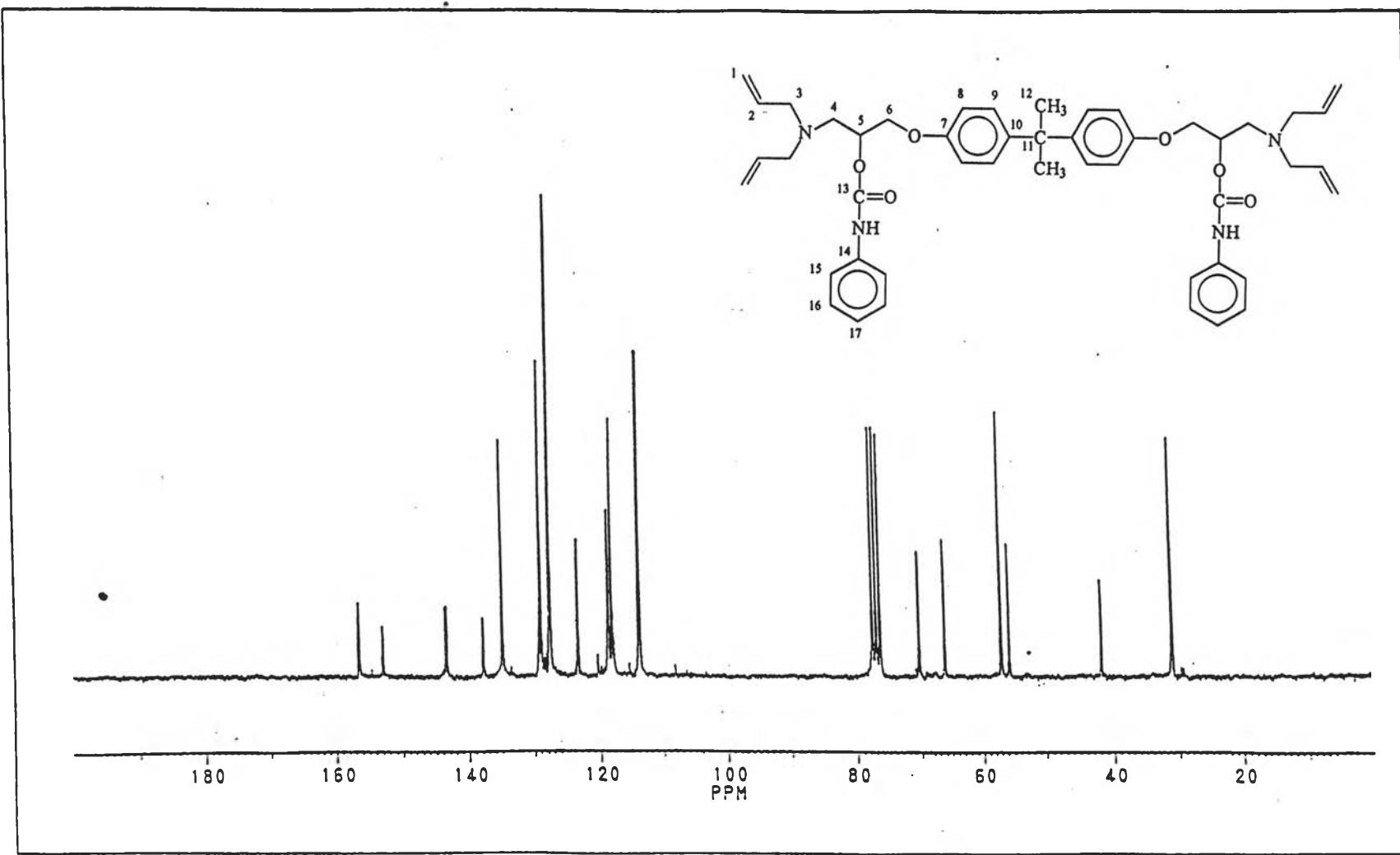
Figure 30 ^{13}C NMR (CDCl$_3$) spectrum of Carbamate Derivative (**17**)
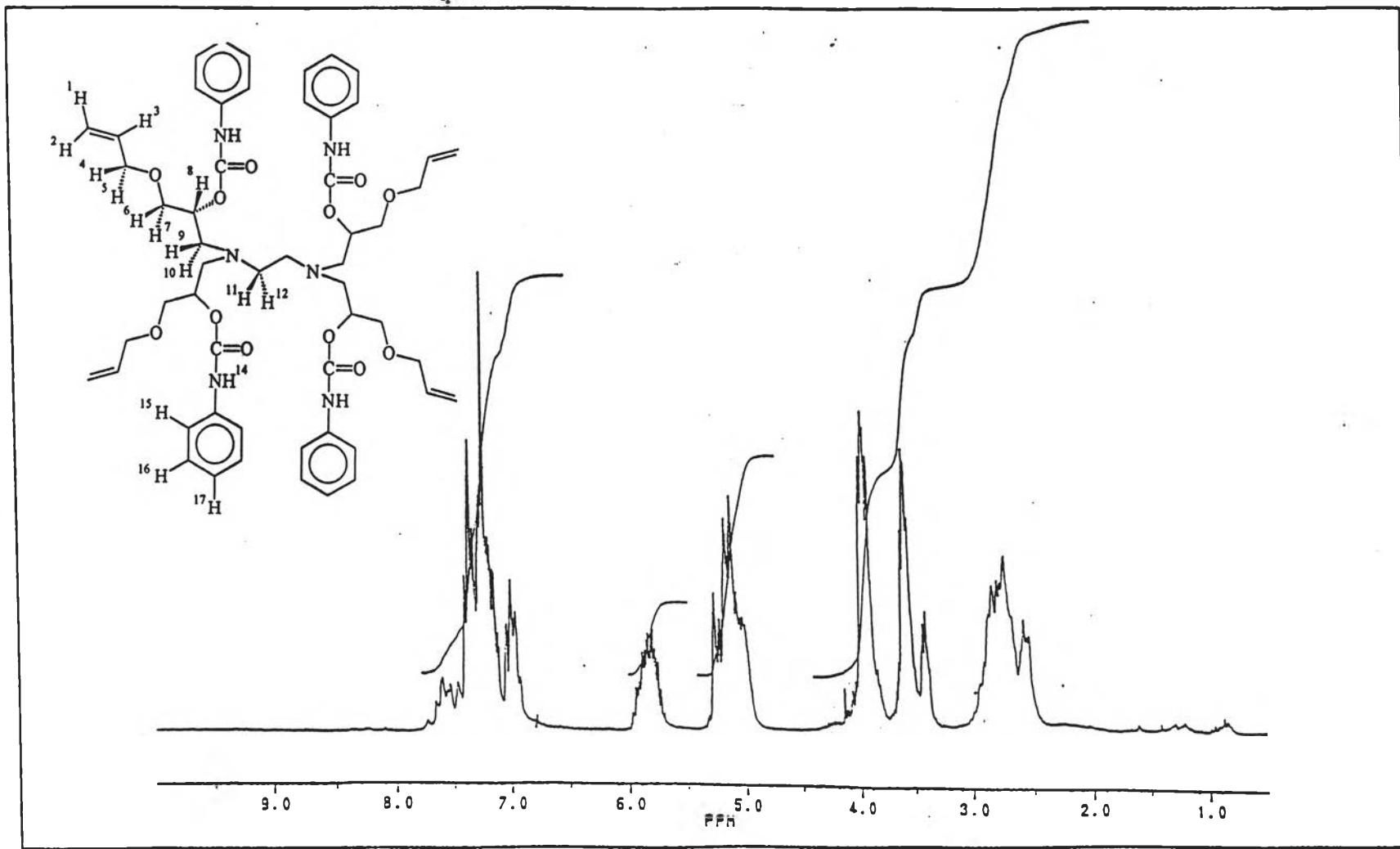
Figure 31 ^{1}H NMR (CDCl$_3$) spectrum of Carbamate Derivative (18)
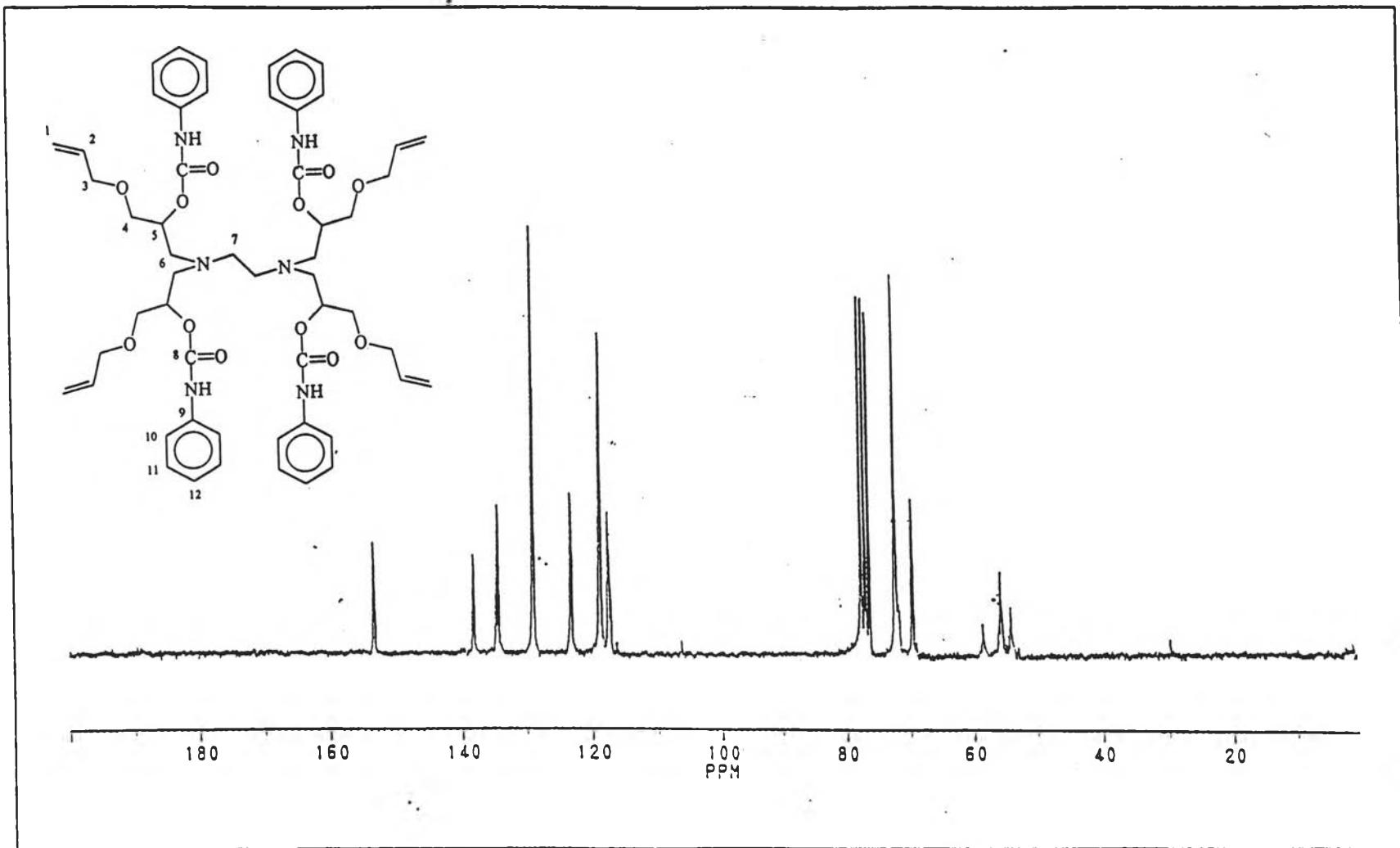
Figure 32 ^{13}C NMR (CDCl$_3$) spectrum of Carbamate Derivative (18)
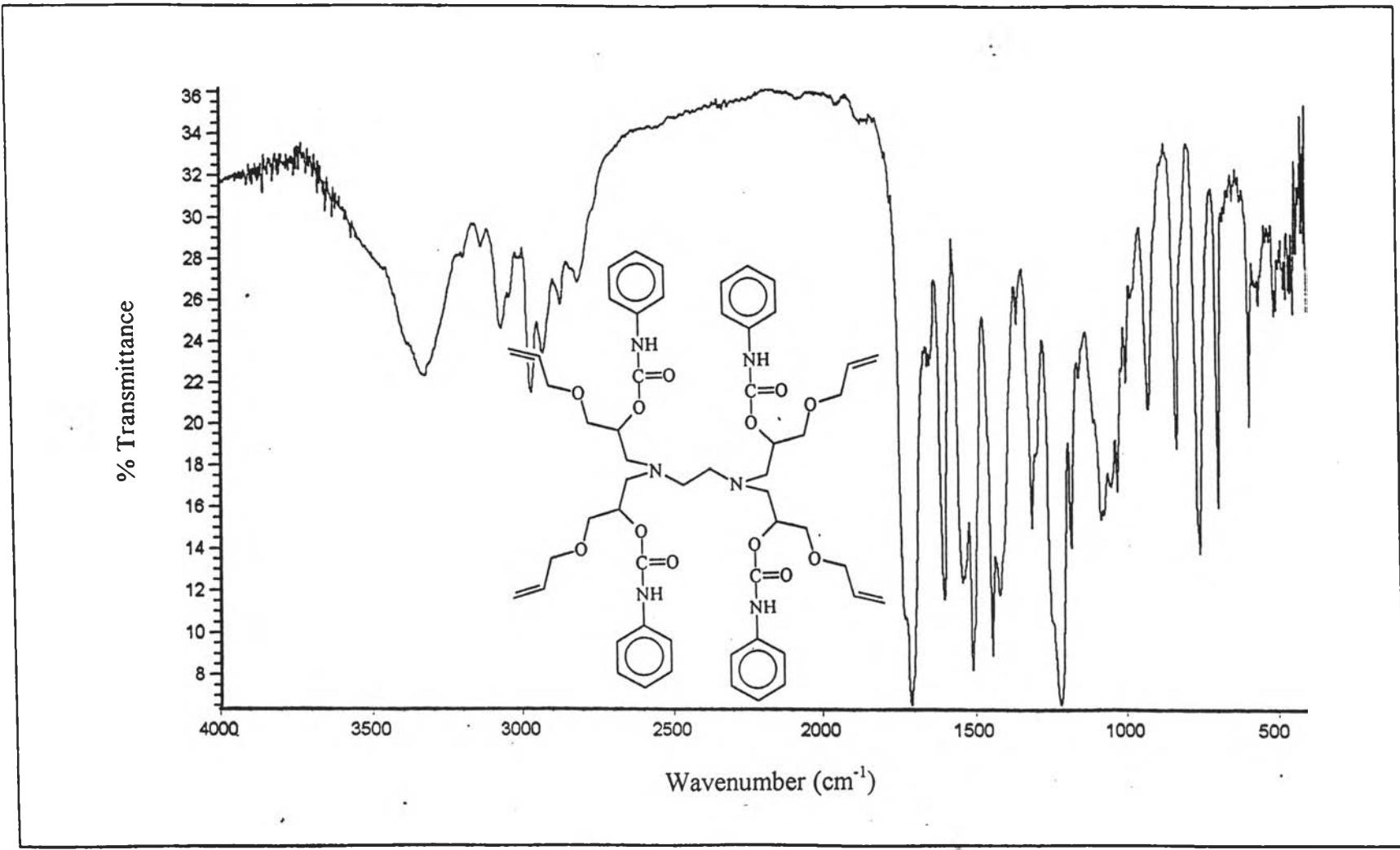
Figure 33   IR spectrum of Carbamate Derivative (18)
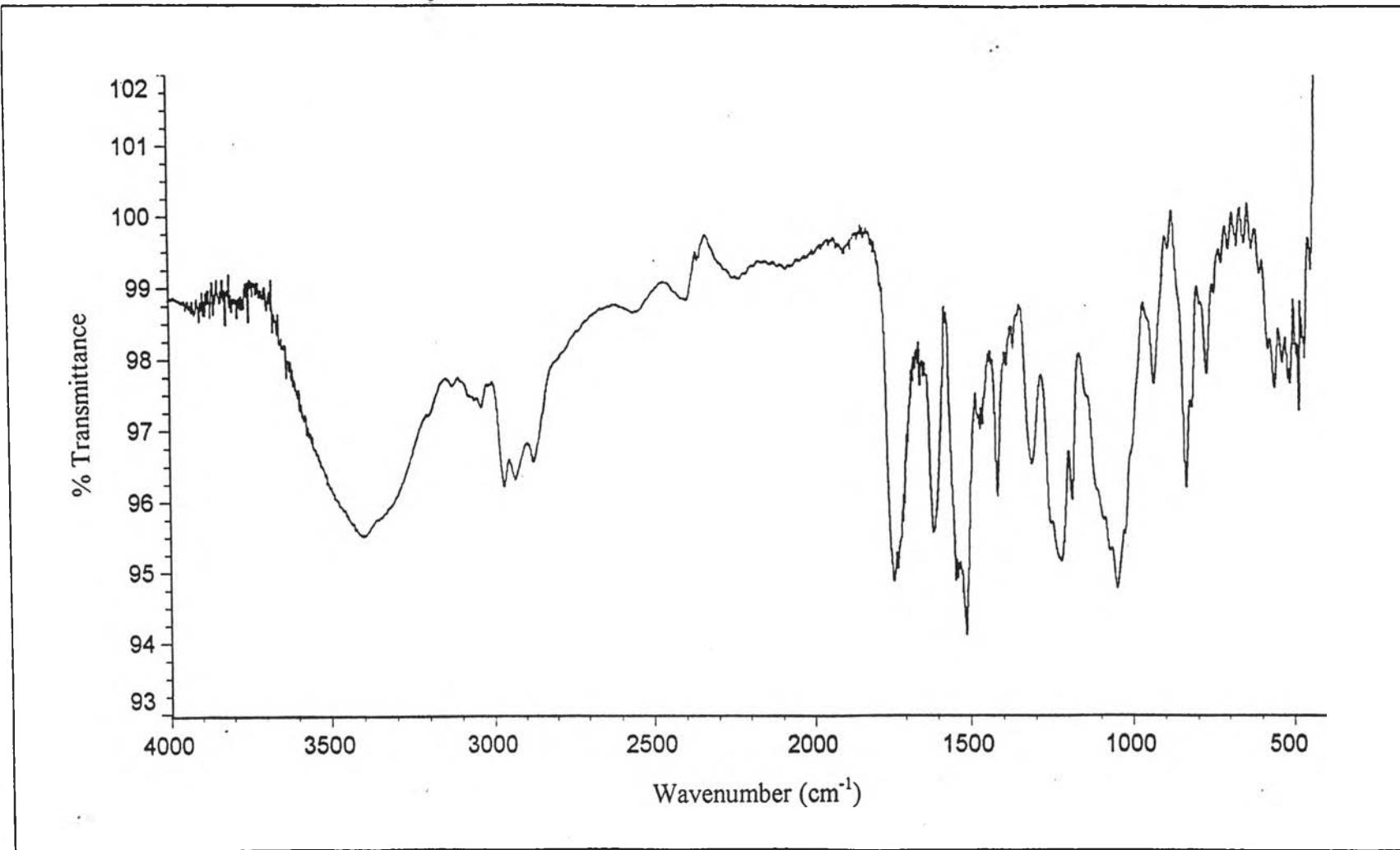
Figure 34   IR spectrum (KBr) of Polyurethane (20)
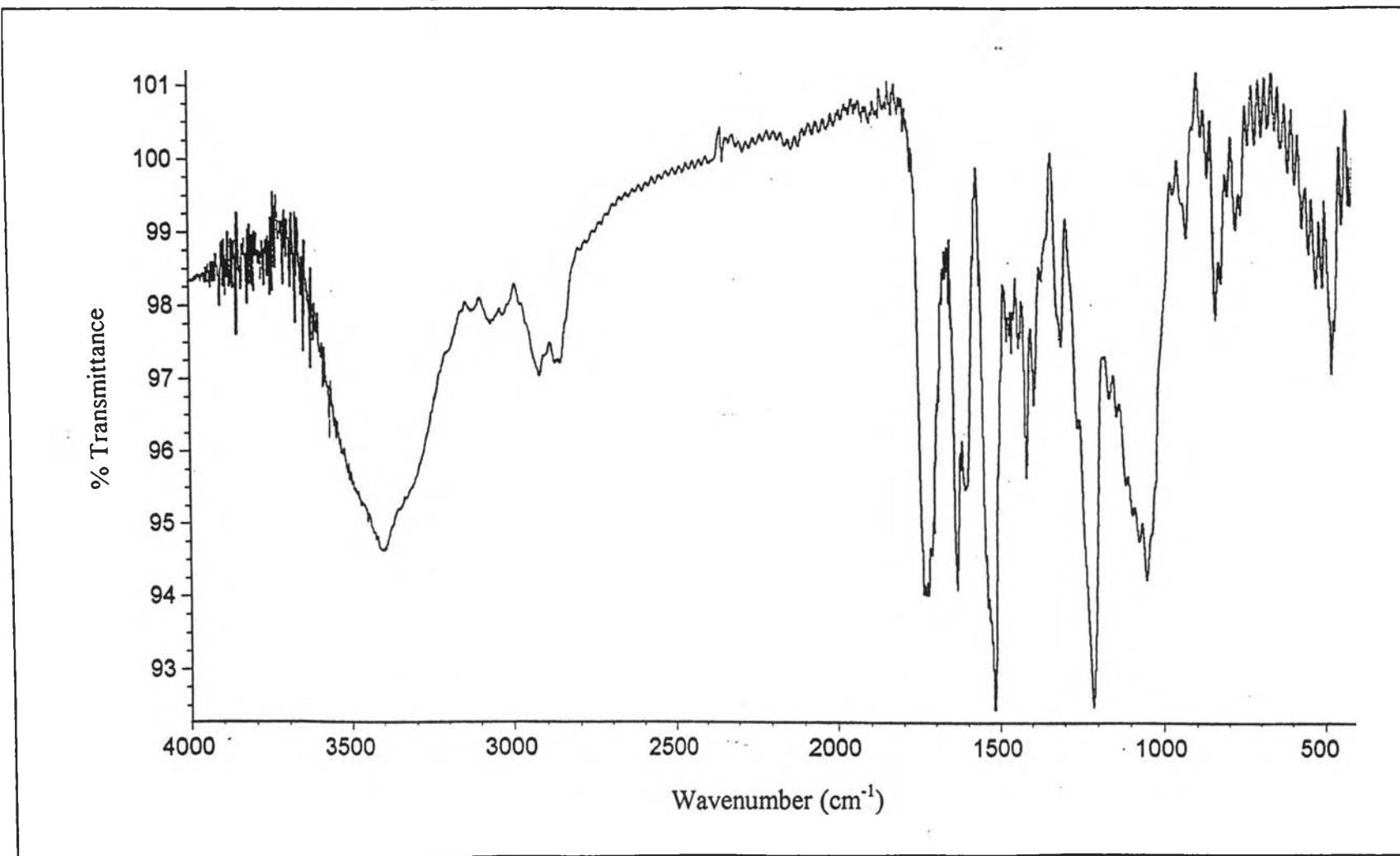
Figure 35   IR spectrum (KBr) of Polyurethane (21)
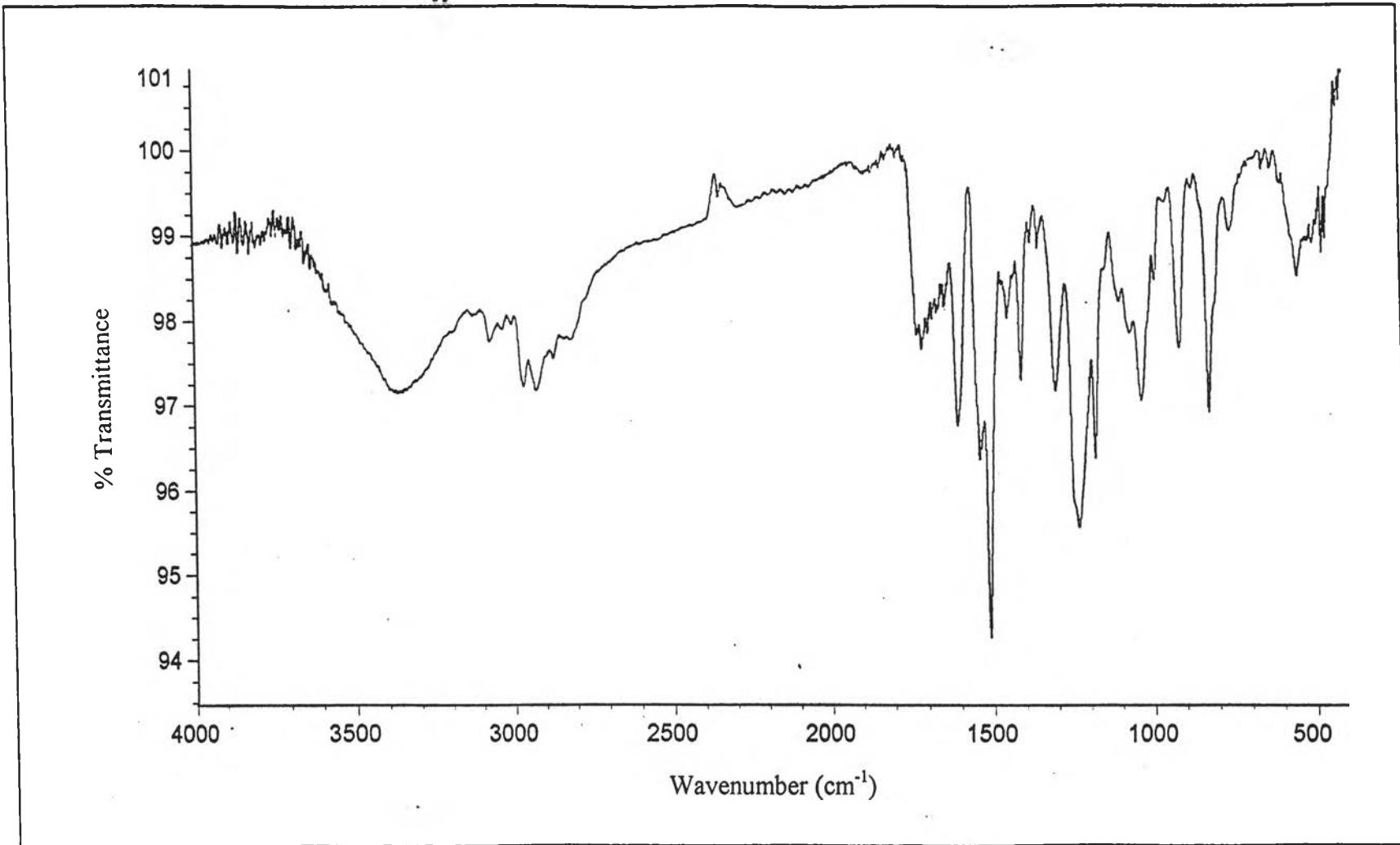
Figure 37   IR spectrum (KBr) of Polyurethane (22)
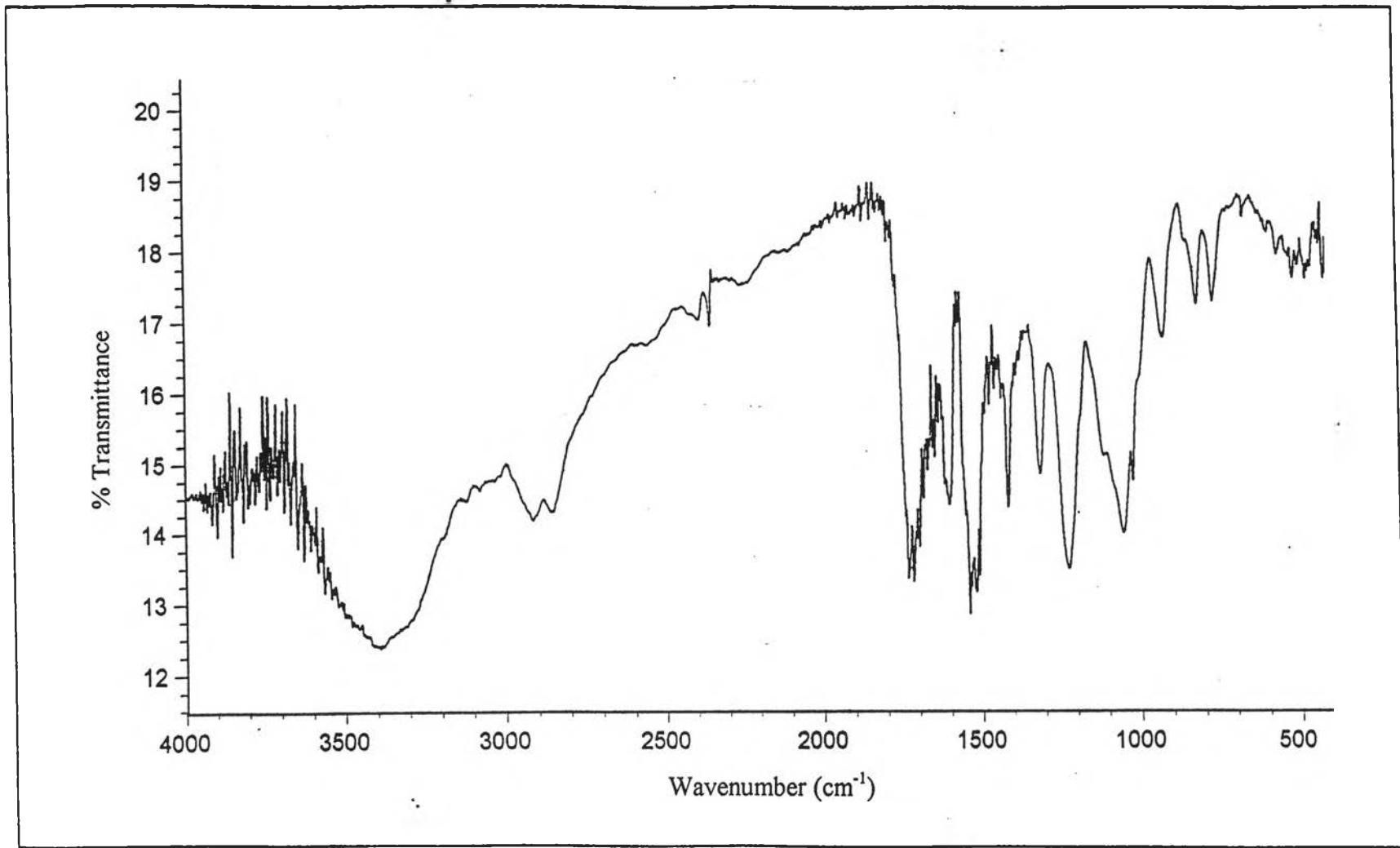
Figure 38   IR spectrum (KBr) of Polyurethane (23)
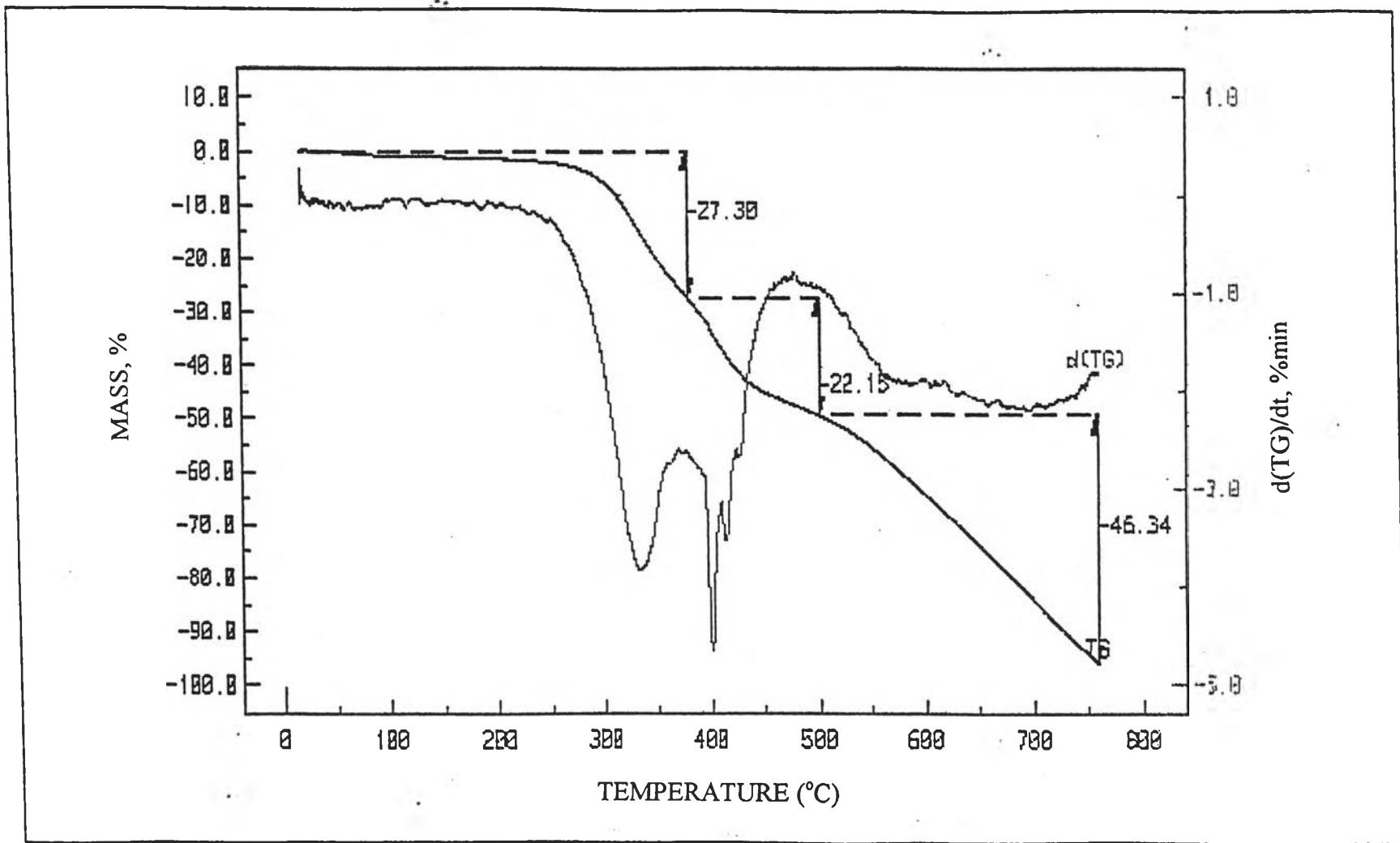
Figure 39   TGA thermogram of Polyurethane (20)
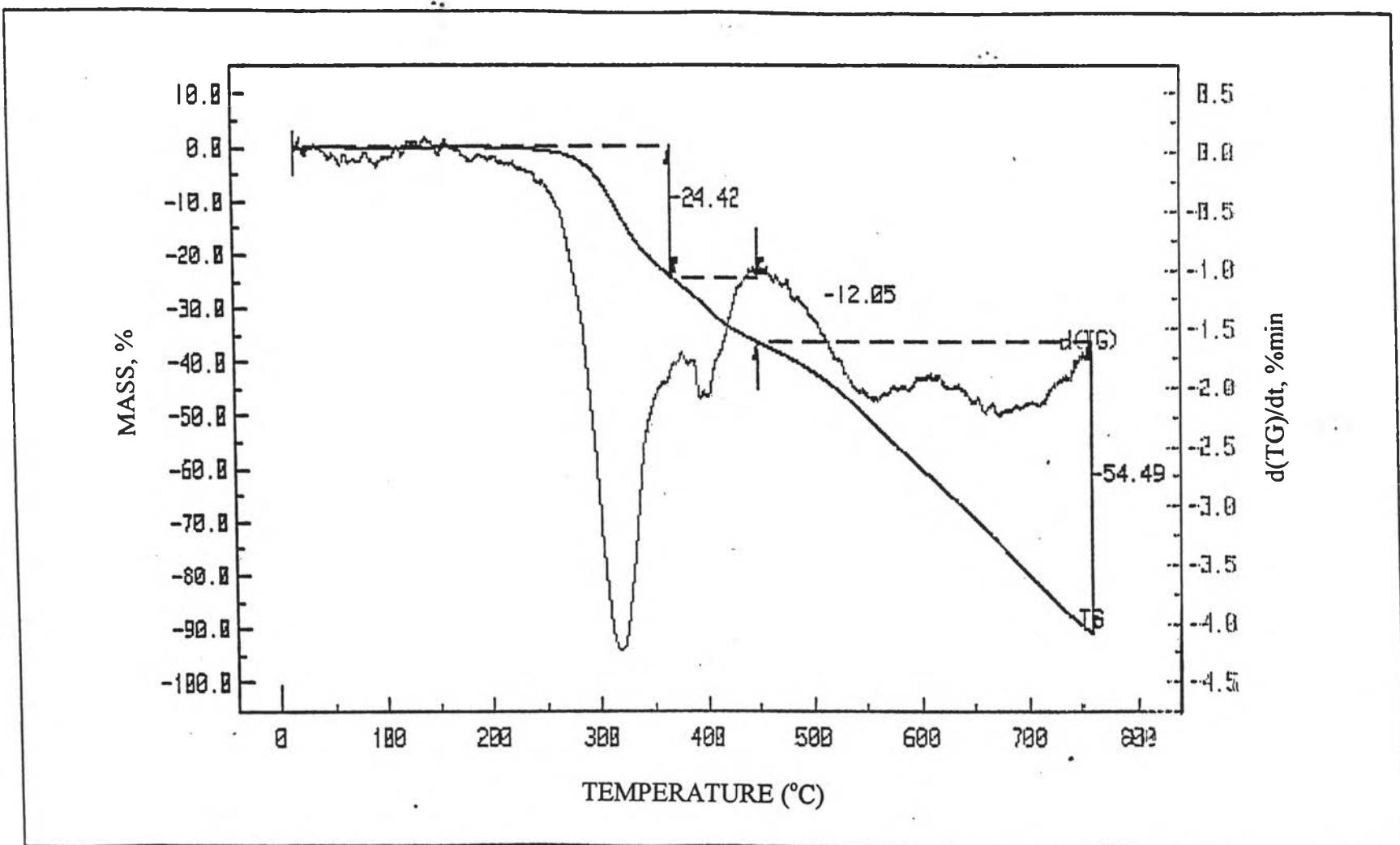
Figure 40   TGA thermogram of Polyurethane (21)
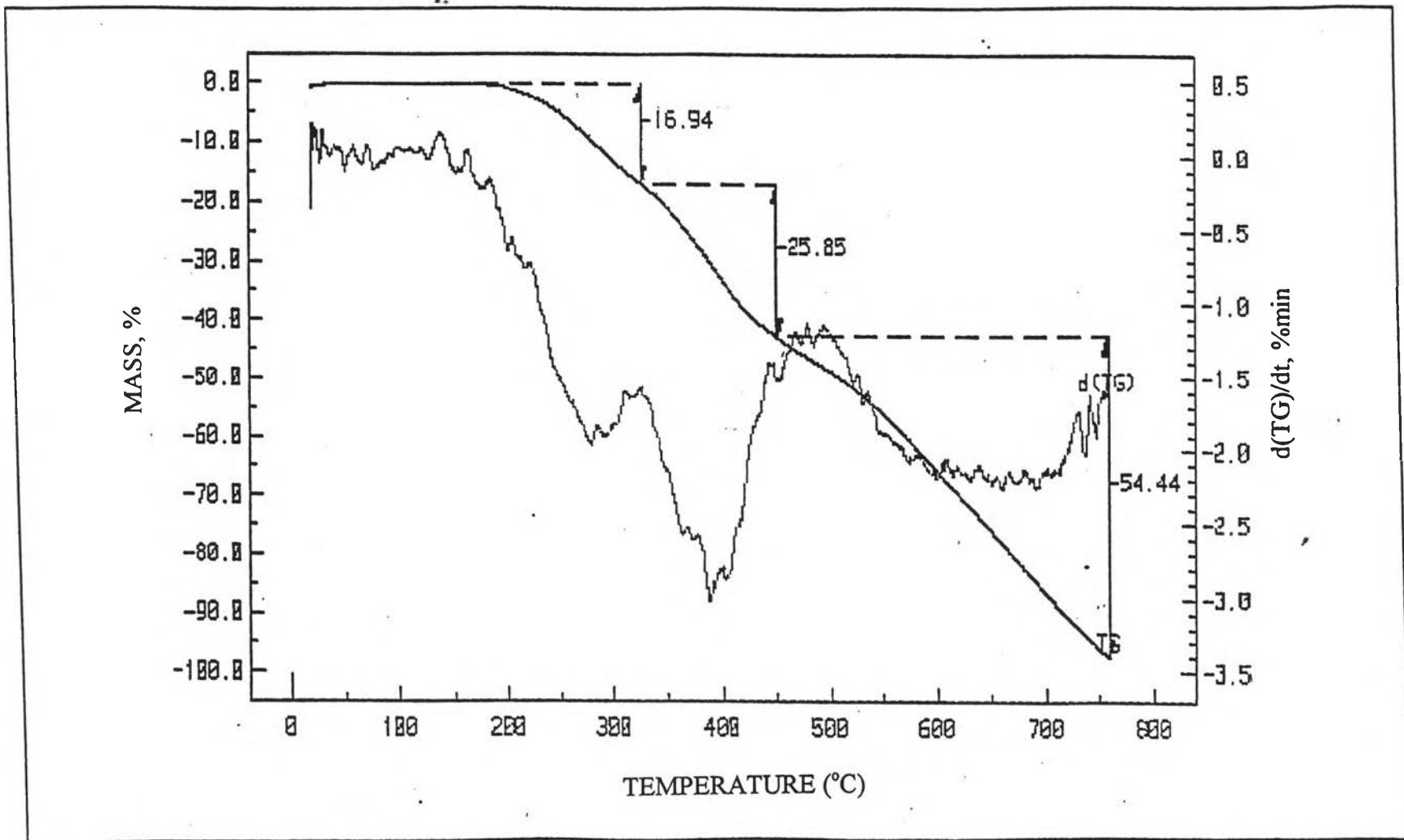
Figure 41   TGA thermogram of Polyurethane (22)
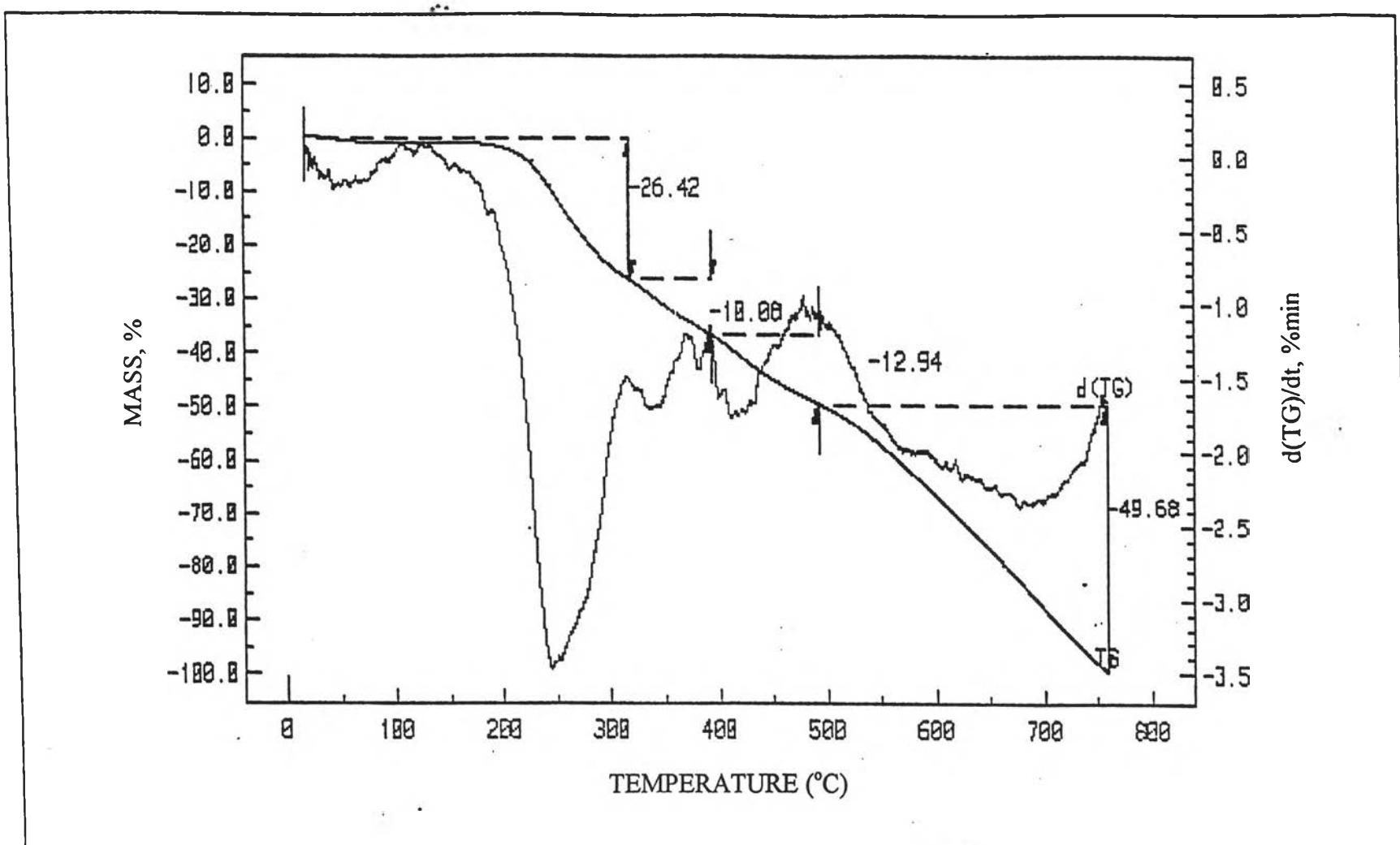
Figure 42   TGA thermogram of Polyurethane (23)
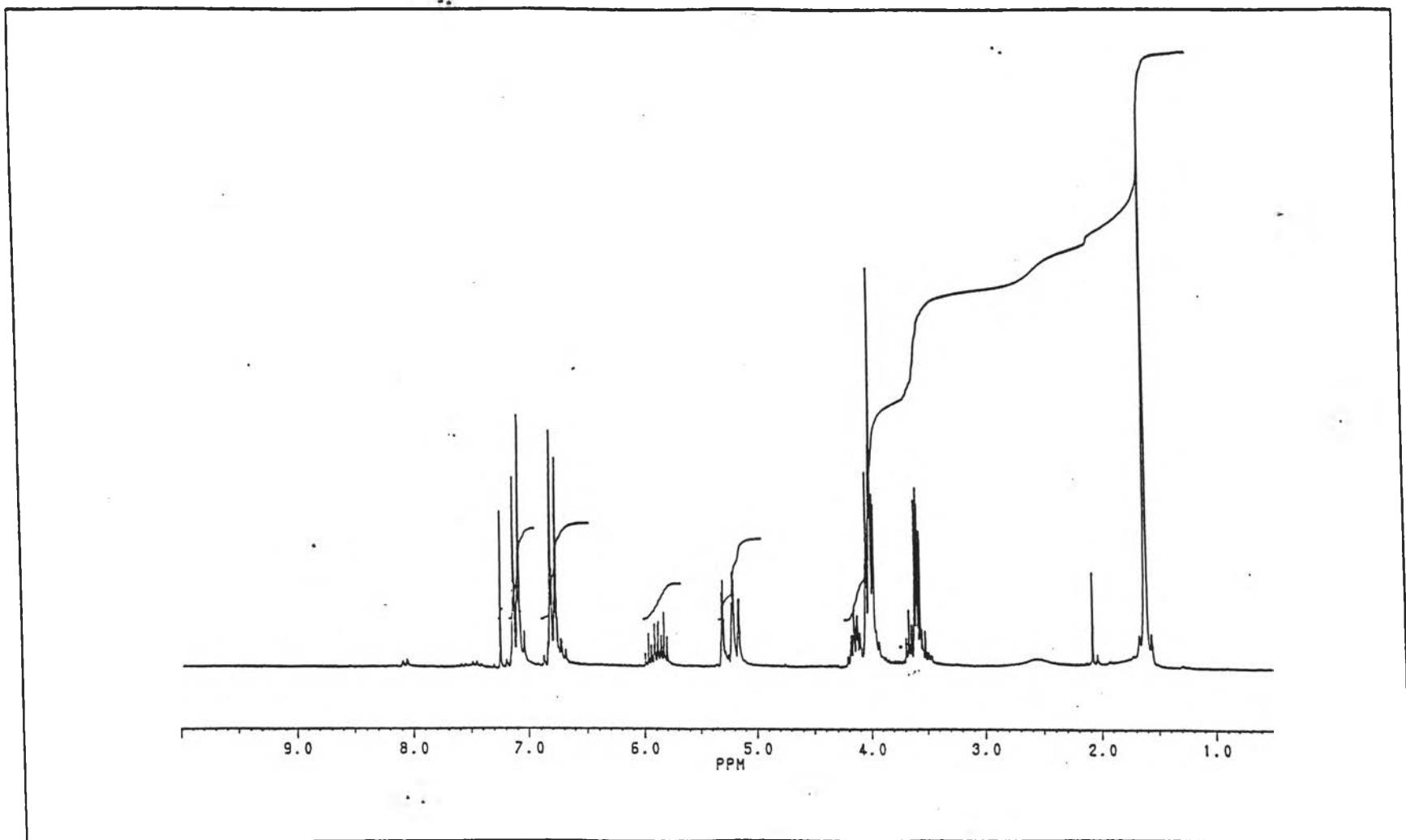
Figure 43 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (3) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 3 hours
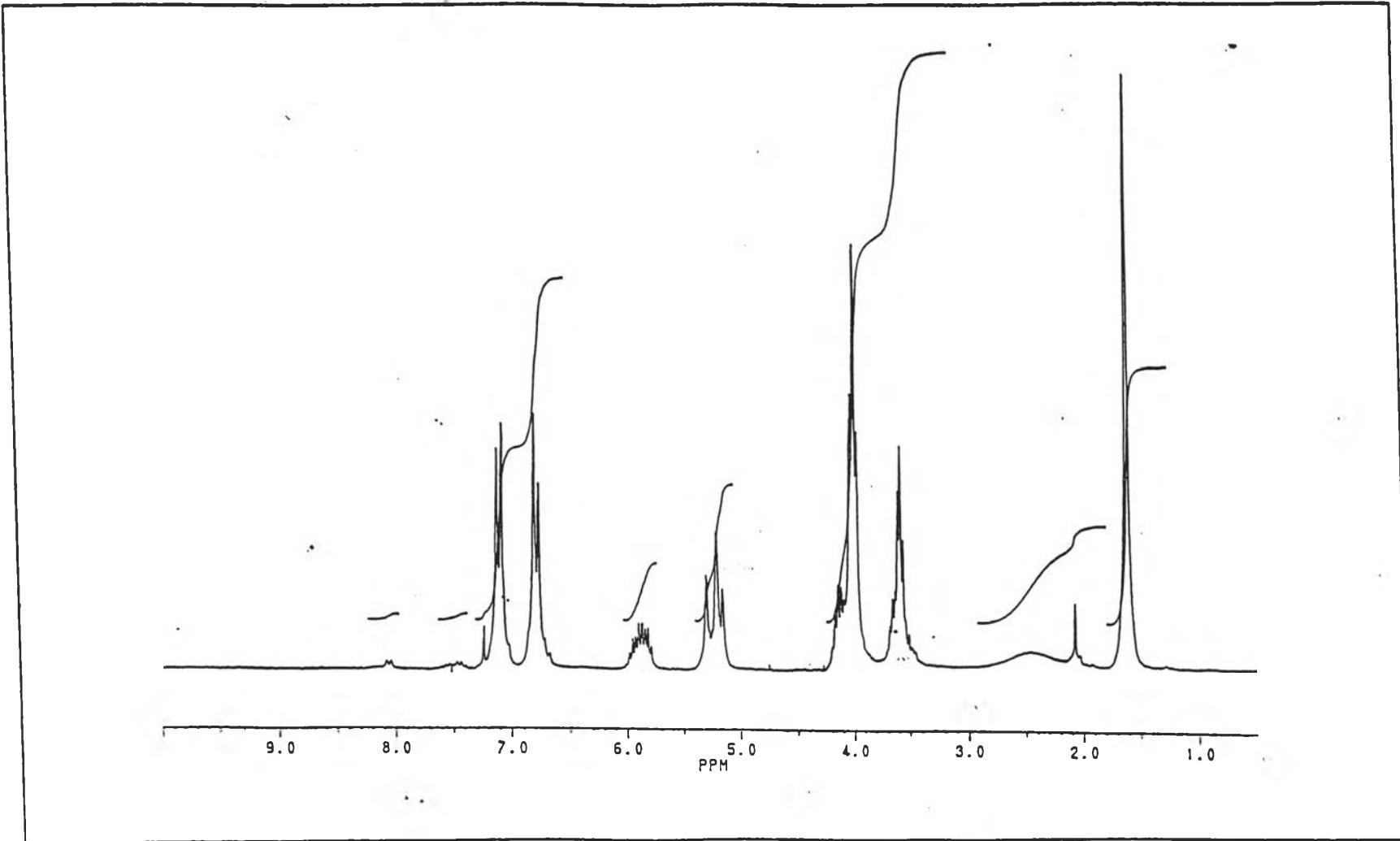
Figure 44 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (3) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 6 hours
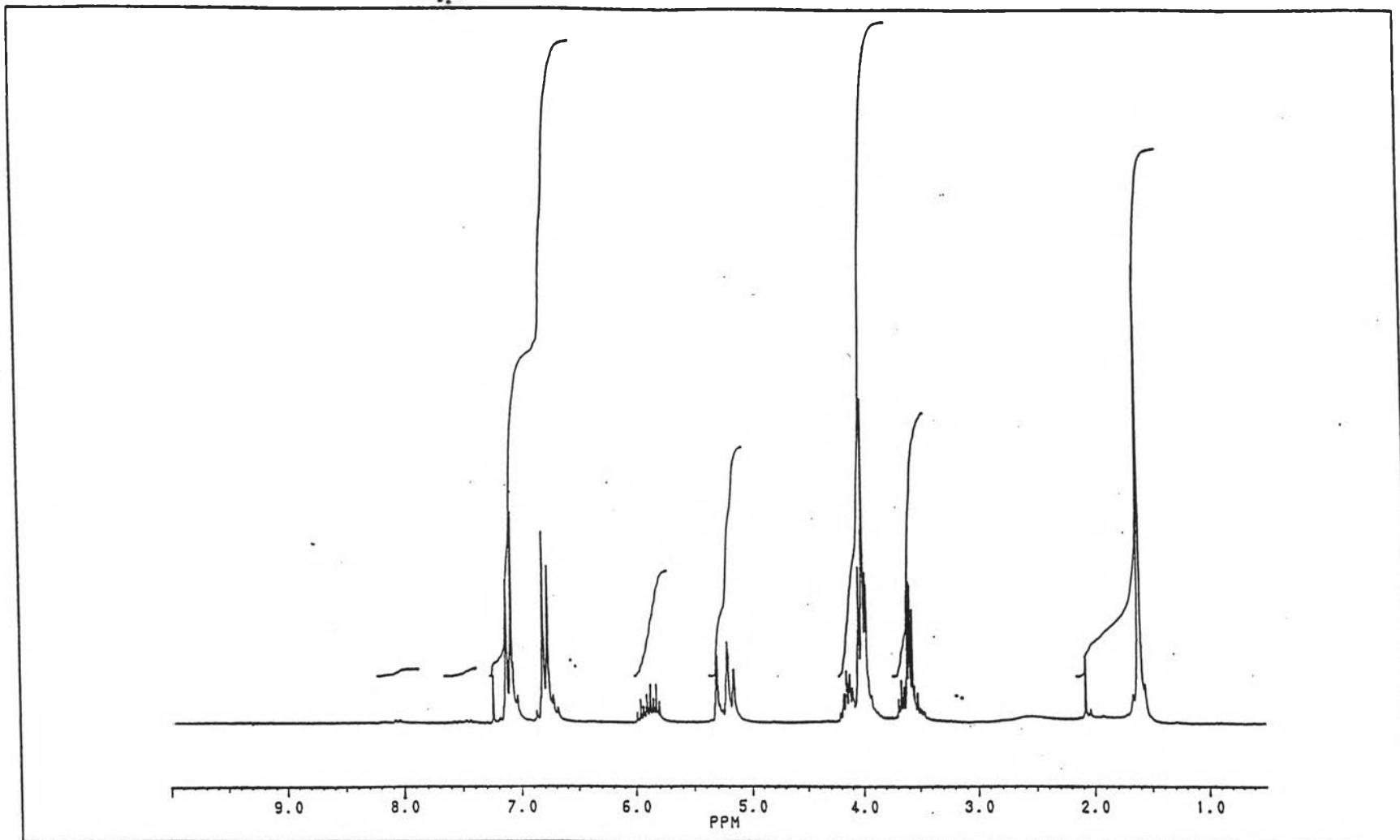
Figure 45 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (3) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 12 hours
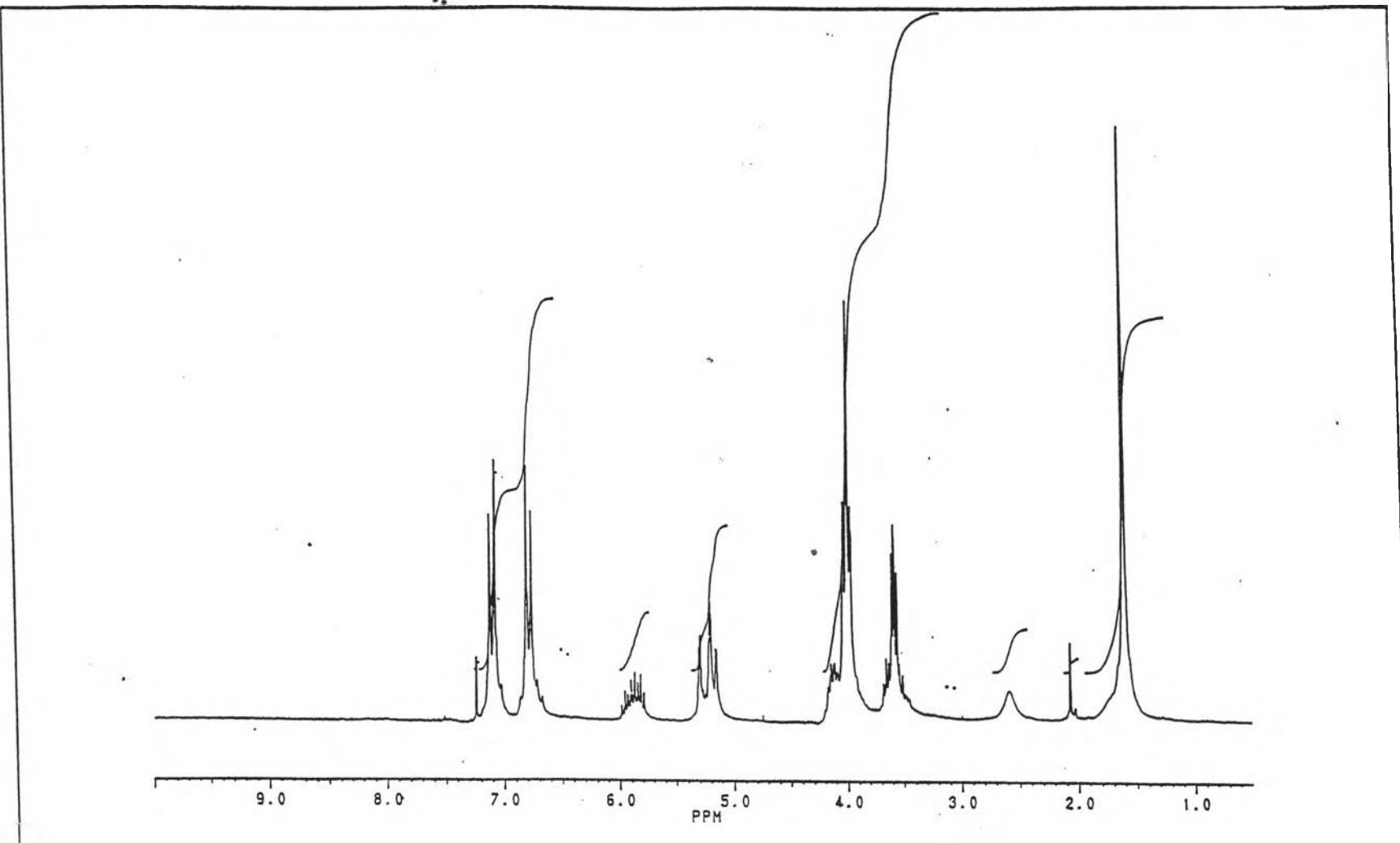
Figure 46 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (3) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 24 hours

Table 3 Integration ratio of H3 (olefinic protons) : H11 (aromatic protons) of

(25) at various reaction time

| Reaction time (hours)	Integration ratio of H3 (olefinic protons) : H11 (aromatic protons) of (25)
0	0.5
3	0.42
6	0.35
12	0.34
24	0.32
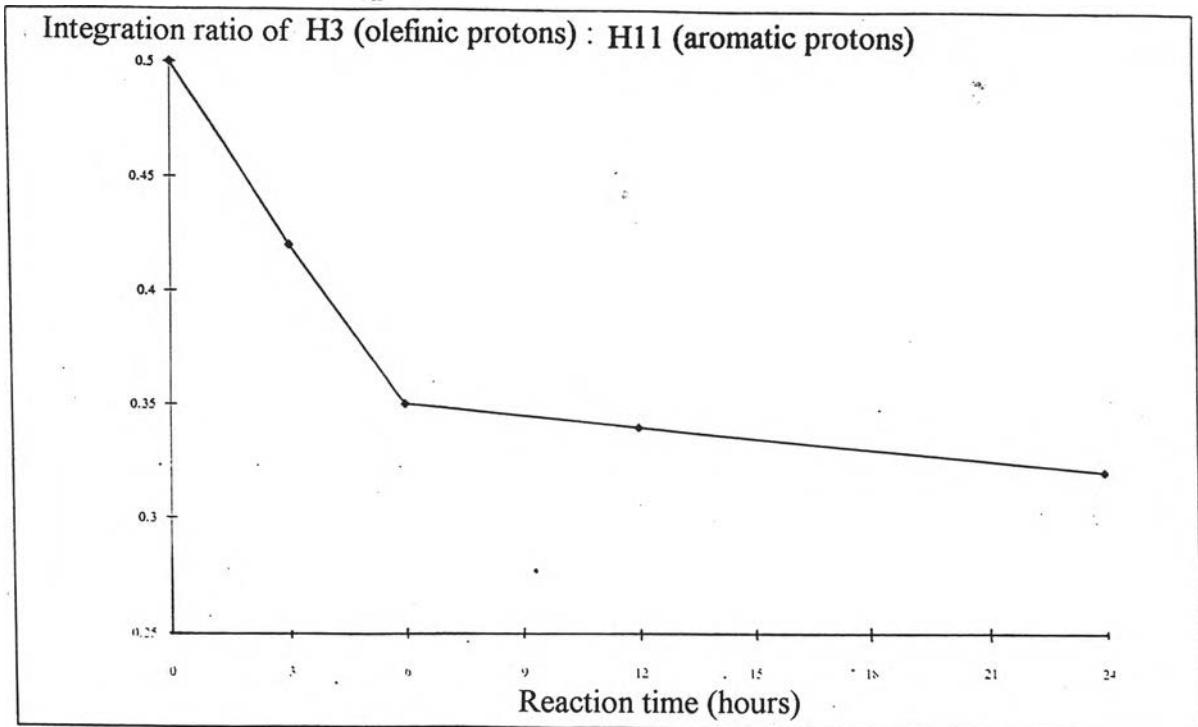
Figure 47 Relationship between the integration ratio of H3 (olefinic protons) :

H11 (aromatic protons) of (25) and reaction time

Table 4   Integration ratio of H4 + H5 + H8 + H9 + H10 (allylic protons + H8
          + H9 + H10) : H11 (aromatic protons) of **(25)**

| Reaction time (hours)	Integration ratio of H4 + H5 + H8 + H9 + H10 (allylic protons + H8 + H9 + H10) : H11 (aromatic protons) of **(25)**
0	2.53
3	2.32
6	2.18
12	2.19
24	1.73

Figure 48   Relationship between the integration ratio of H4 + H5 + H8 + H9 + H10
             (allylic protons + H8 + H9 + H10) : H11 (aromatic protons) of **(25)**
   and reaction time
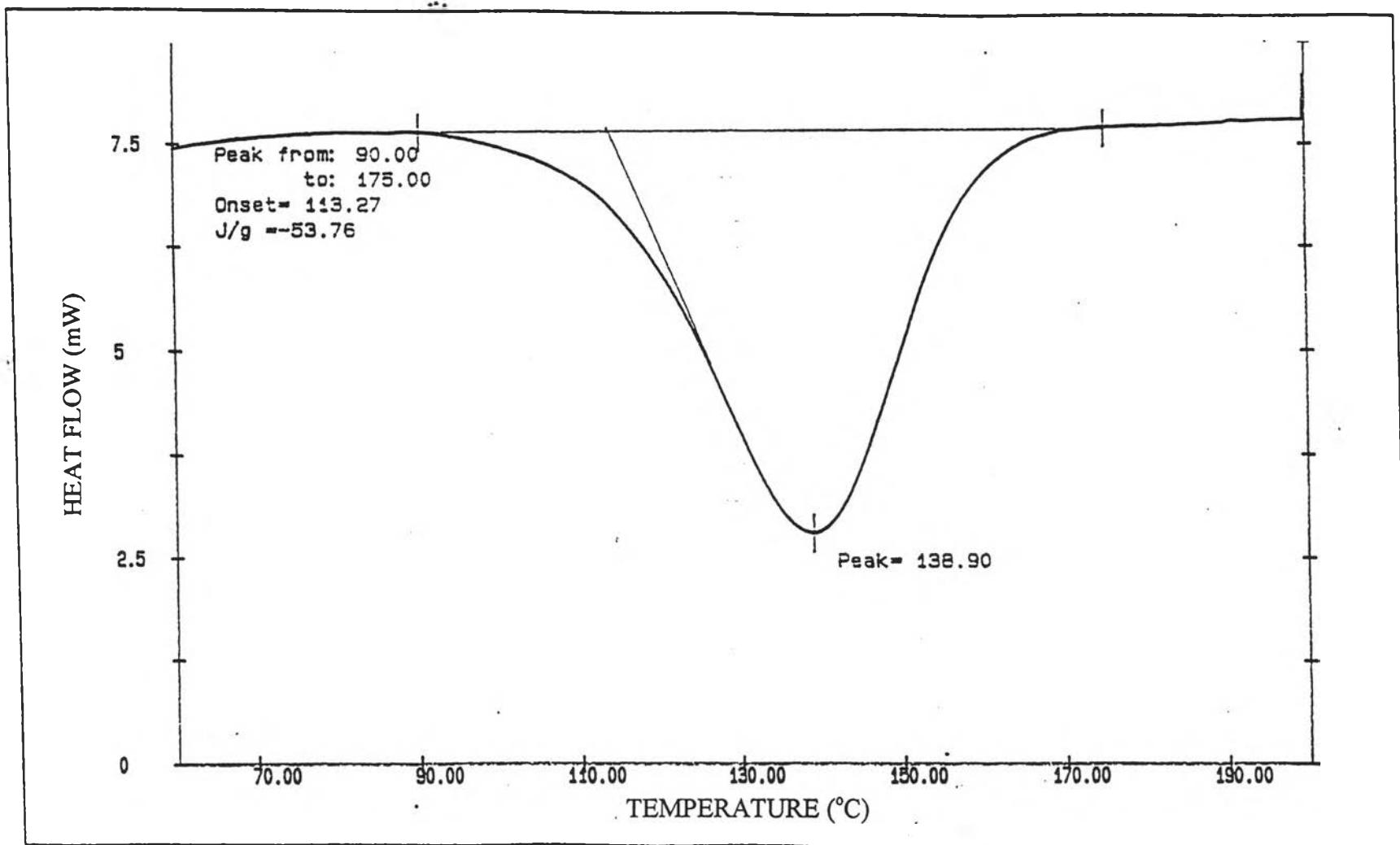
Figure 49   DSC thermogram of the mixture of (3) and 5 mole % of Benzoyl Peroxide (24)
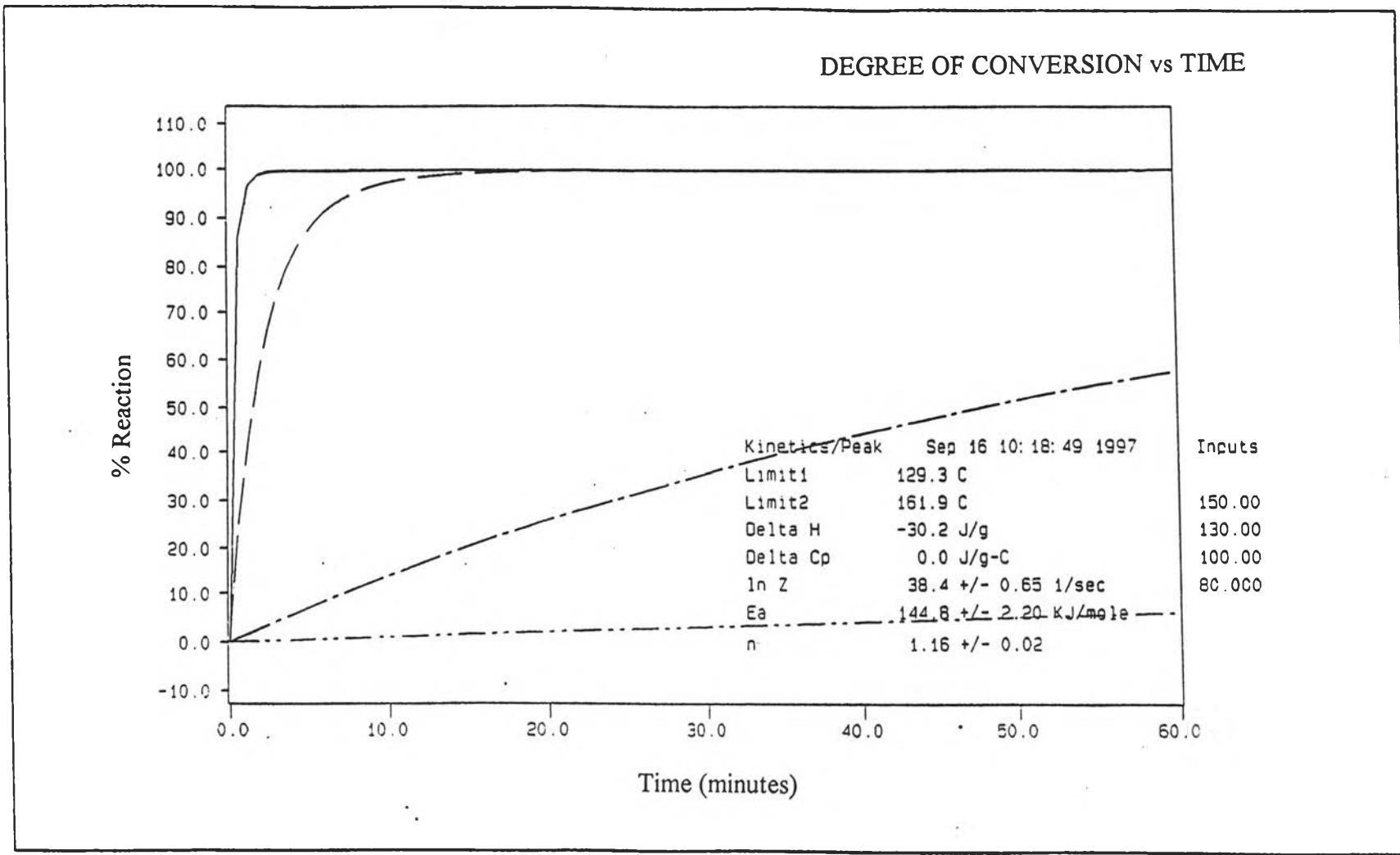
Figure 50   Kinetic DSC thermogram of the mixture of (3) and 5 mole % of Benzoyl Peroxide (24)
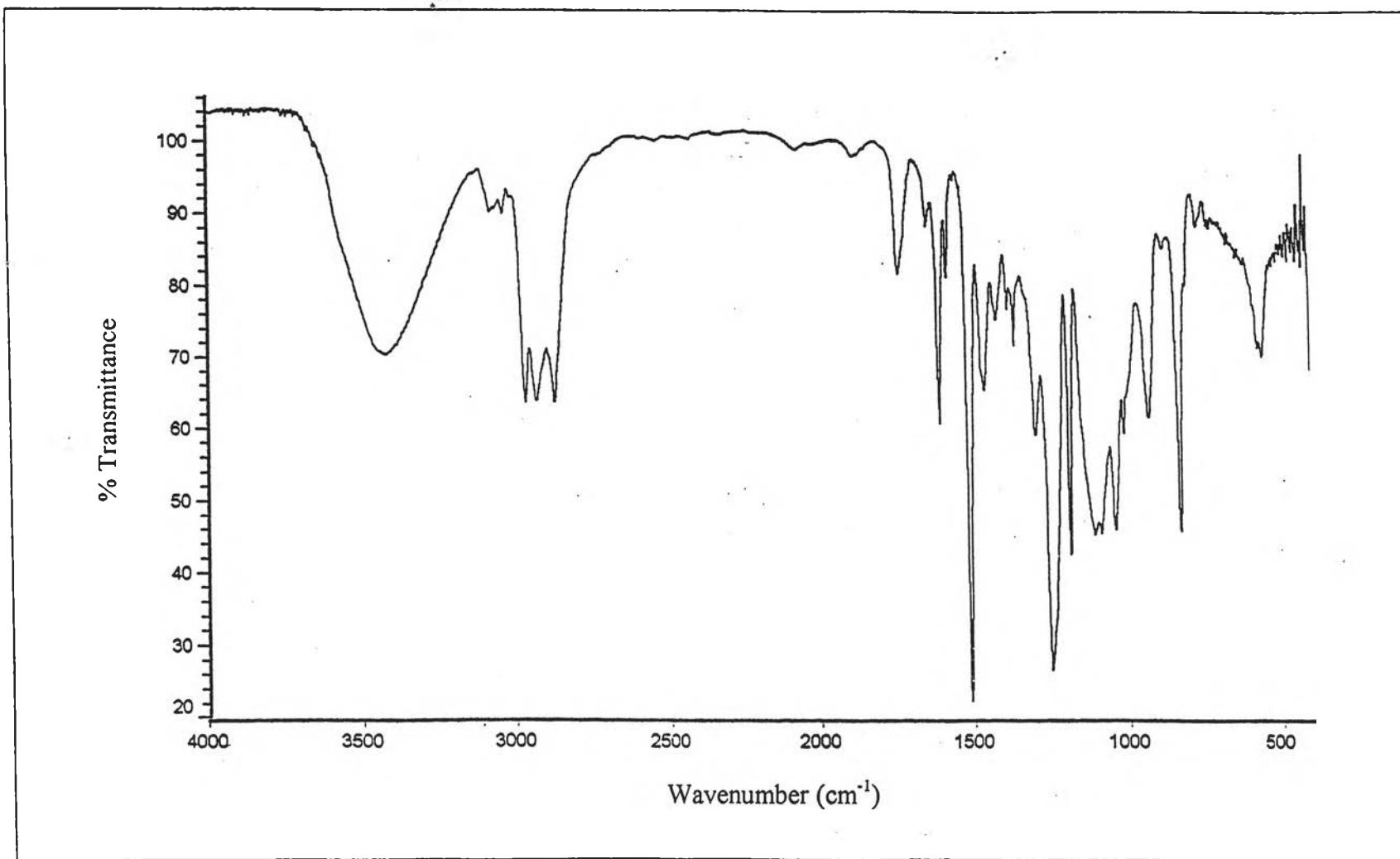
Figure 51   IR spectrum (KBr) of (25) obtained from the reaction between (3) and 5 mole % Benzoyl Peroxide (24) at 80°C for 24 hours
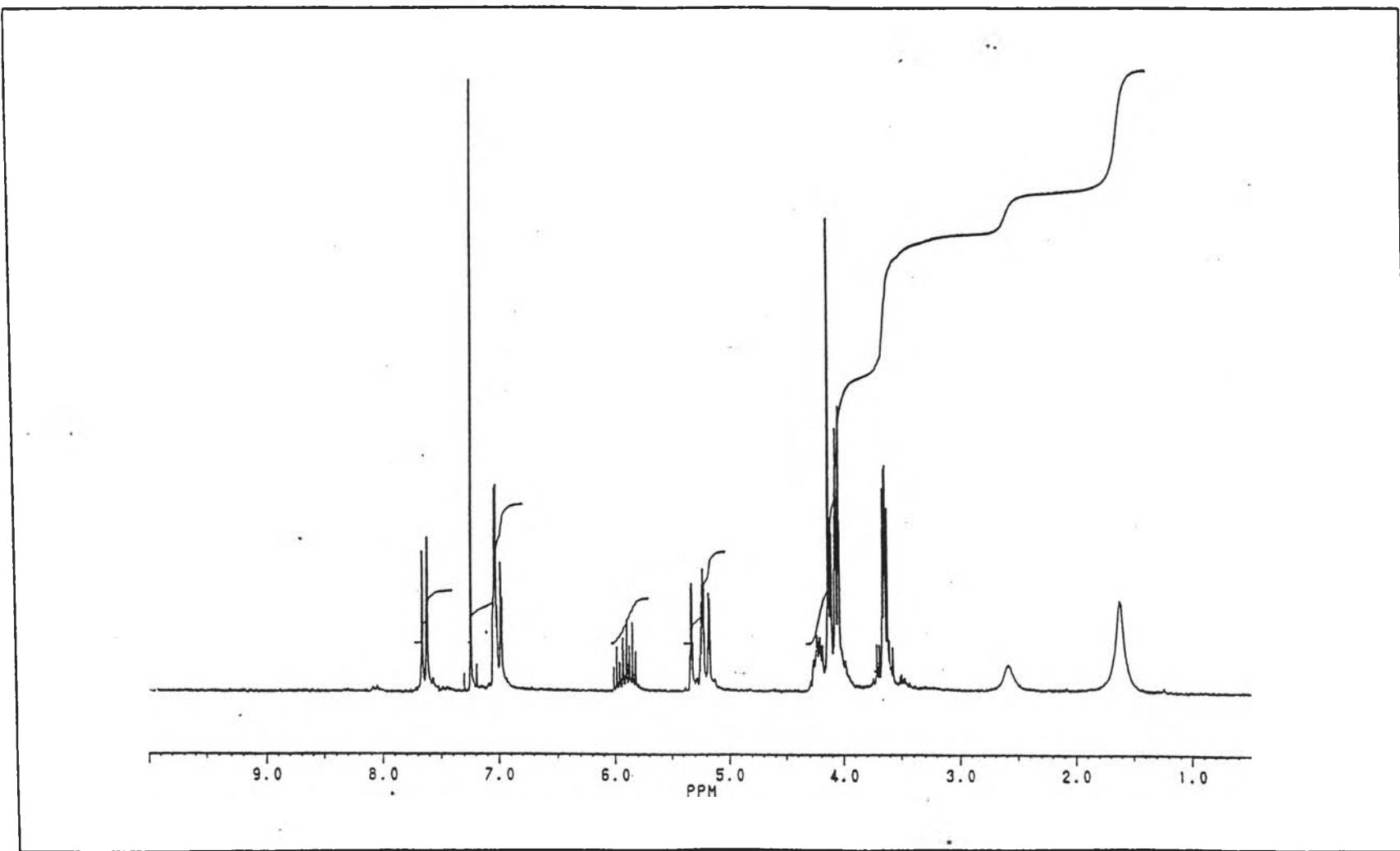
Figure 52 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (9) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 3 hours
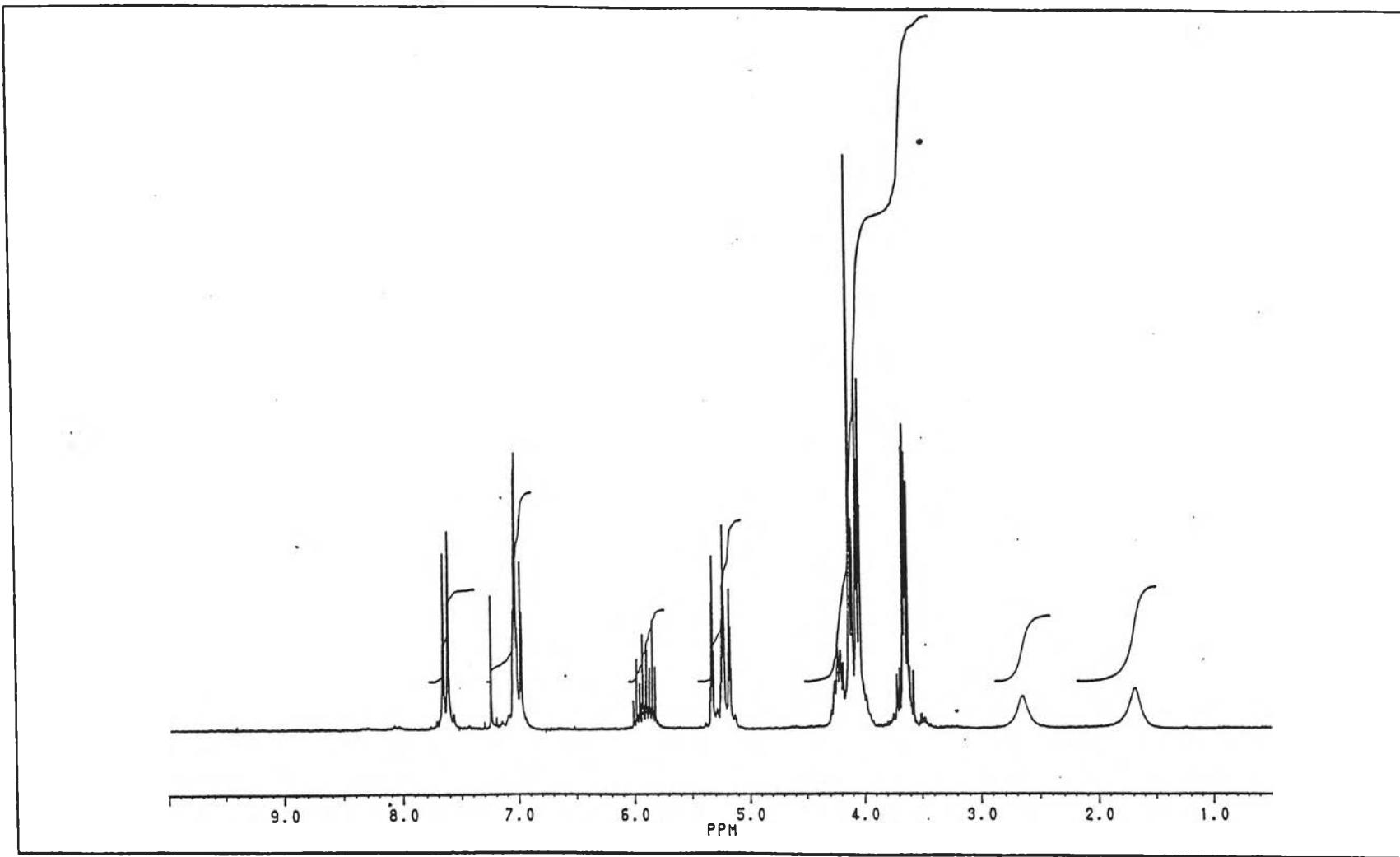
Figure 53　^{1}H NMR (CDCl$_3$) spectrum of the reaction between (9) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 6 hours
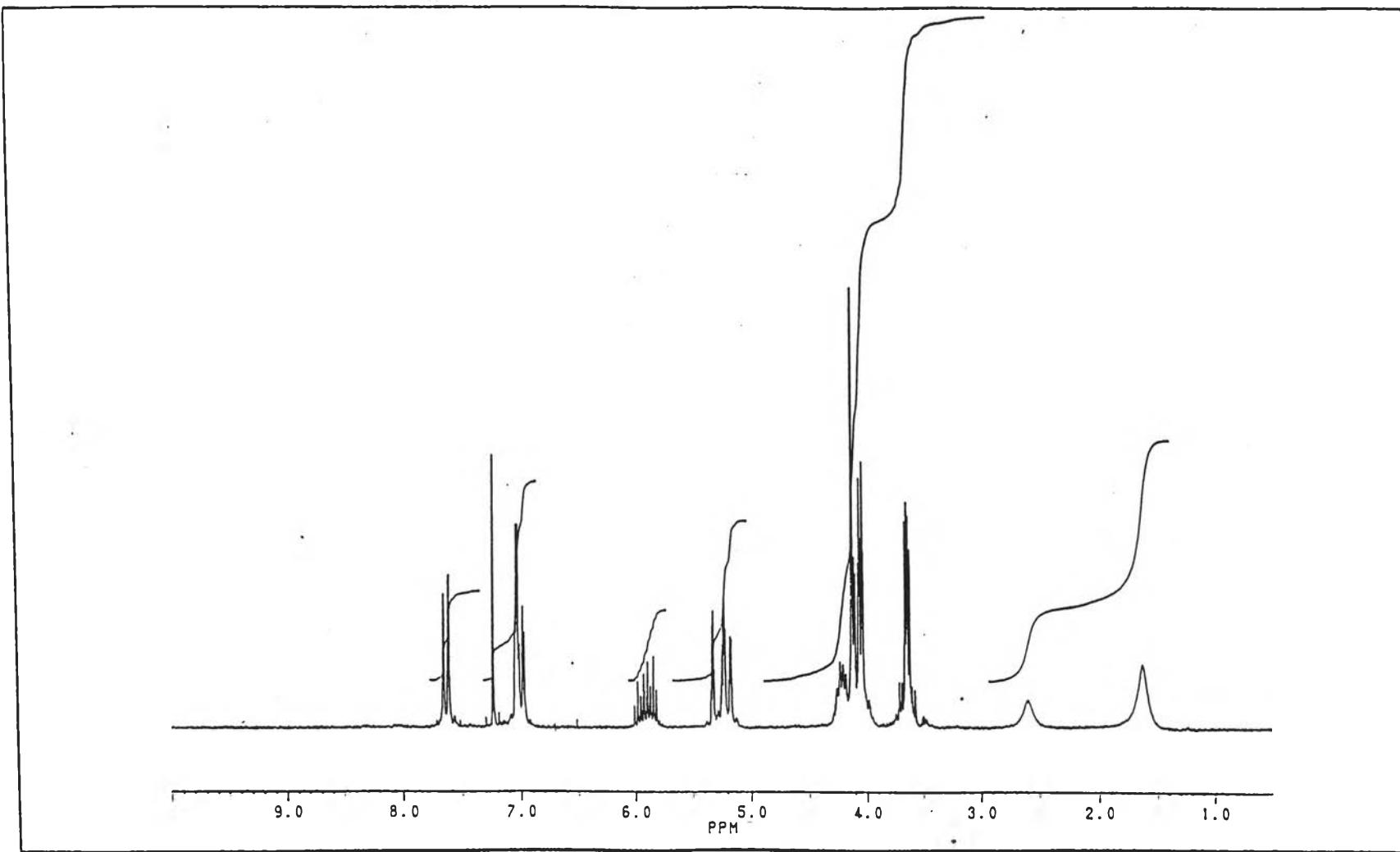
Figure 54 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (9) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 12 hours
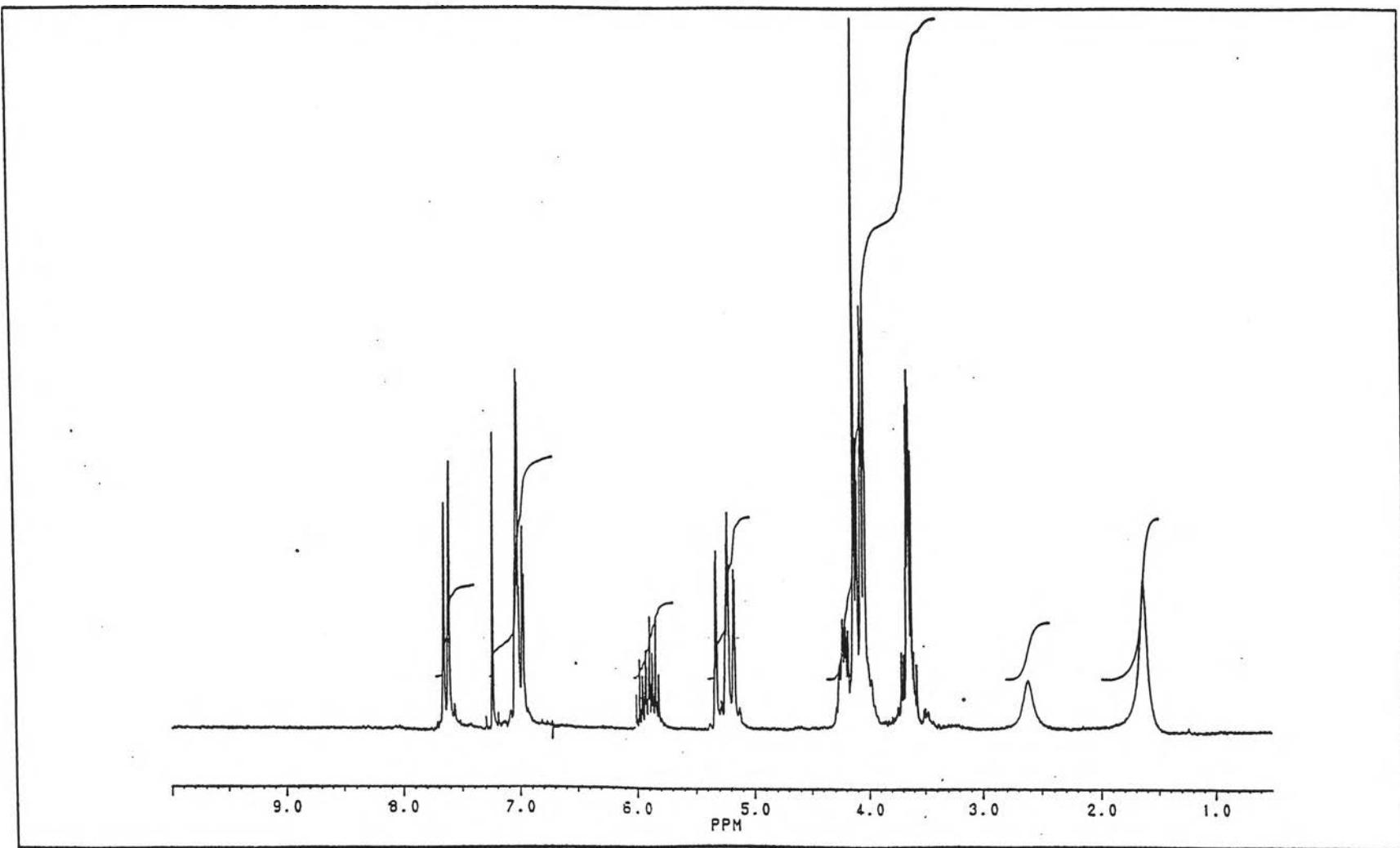
Figure 55 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (9) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 24 hours

Table 5   Integration ratio of H3 (olefinic protons) : H11 + H12

(aromatic protons) of (26) at various reaction time

| Reaction time (hours)	Integration ratio of H3 (olefinic protons) : H11 + H12 (aromatic protons) of (26)
0	0.48
3	0.44
6	0.41
12	0.40
24	0.39
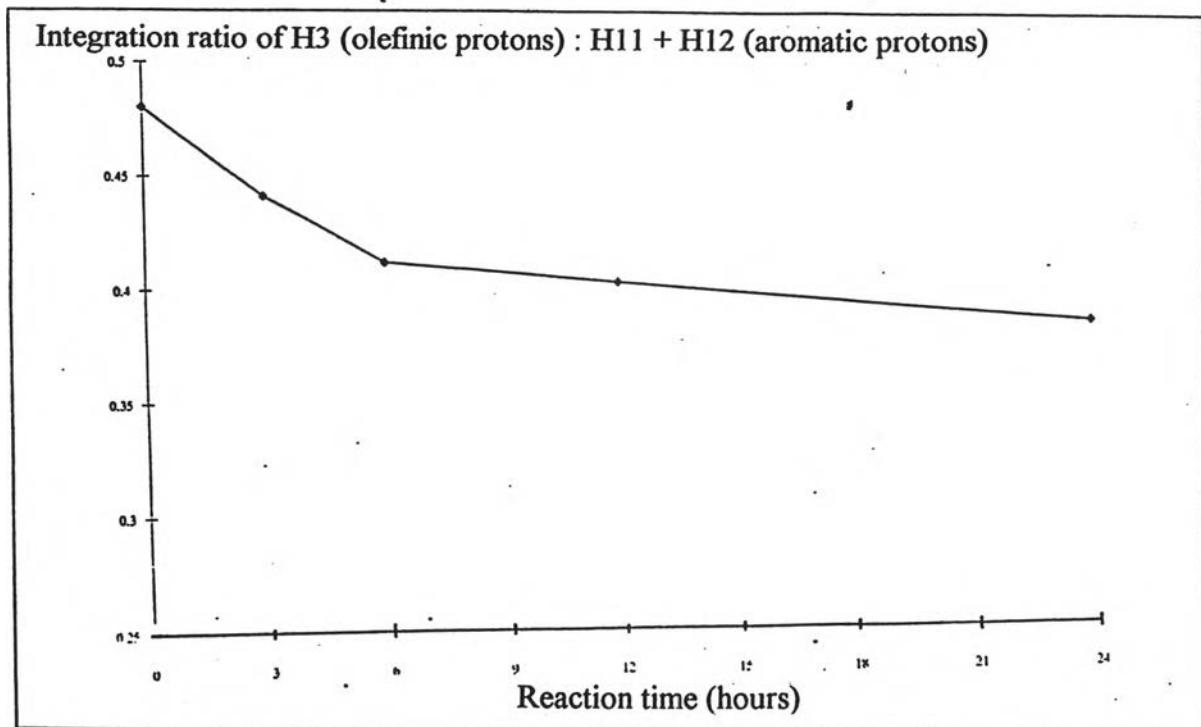
Figure 56   Relationship between the integration ratio of H3 (olefinic protons)

: H11 + H12 (aromatic protons) of (26) and reaction time
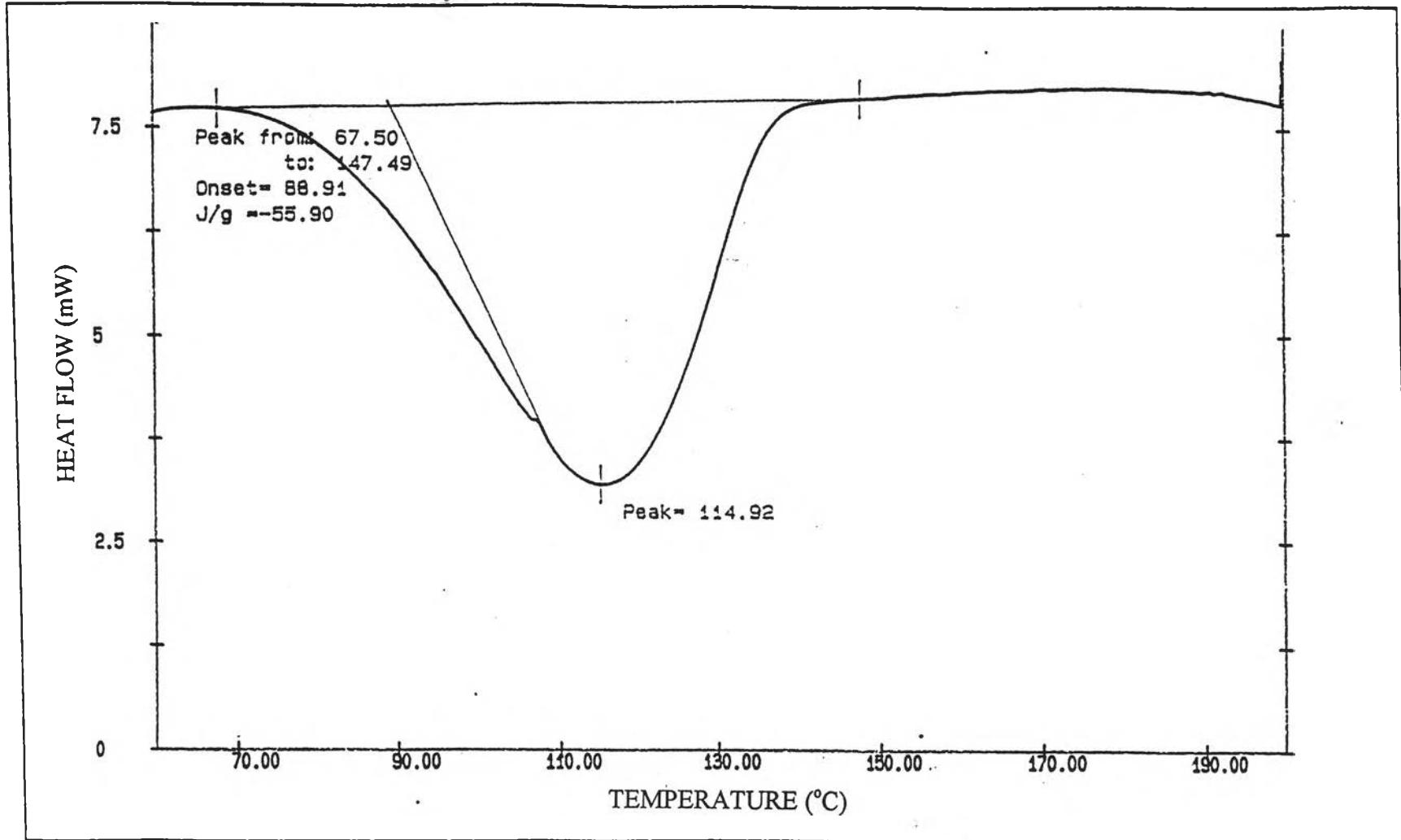
Figure 57   DSC thermogram of the mixture of (9) and 5 mole % of Benzoyl Peroxide (24)

DEGREE OF CONVERSION vs TIME

| Kinetics/Peak	Sep 16 10:32:52 1997	Inputs
Limit1	97.6 C	
Limit2	137.9 C	150.00
Delta H	-30.5 J/g	130.00
Delta Cp	0.0 J/g-C	100.00
ln Z	22.1 +/- 0.41 1/sec	80.000
Ea	83.9 +/- 1.28 KJ/mole	
n	0.84 +/- 0.02	
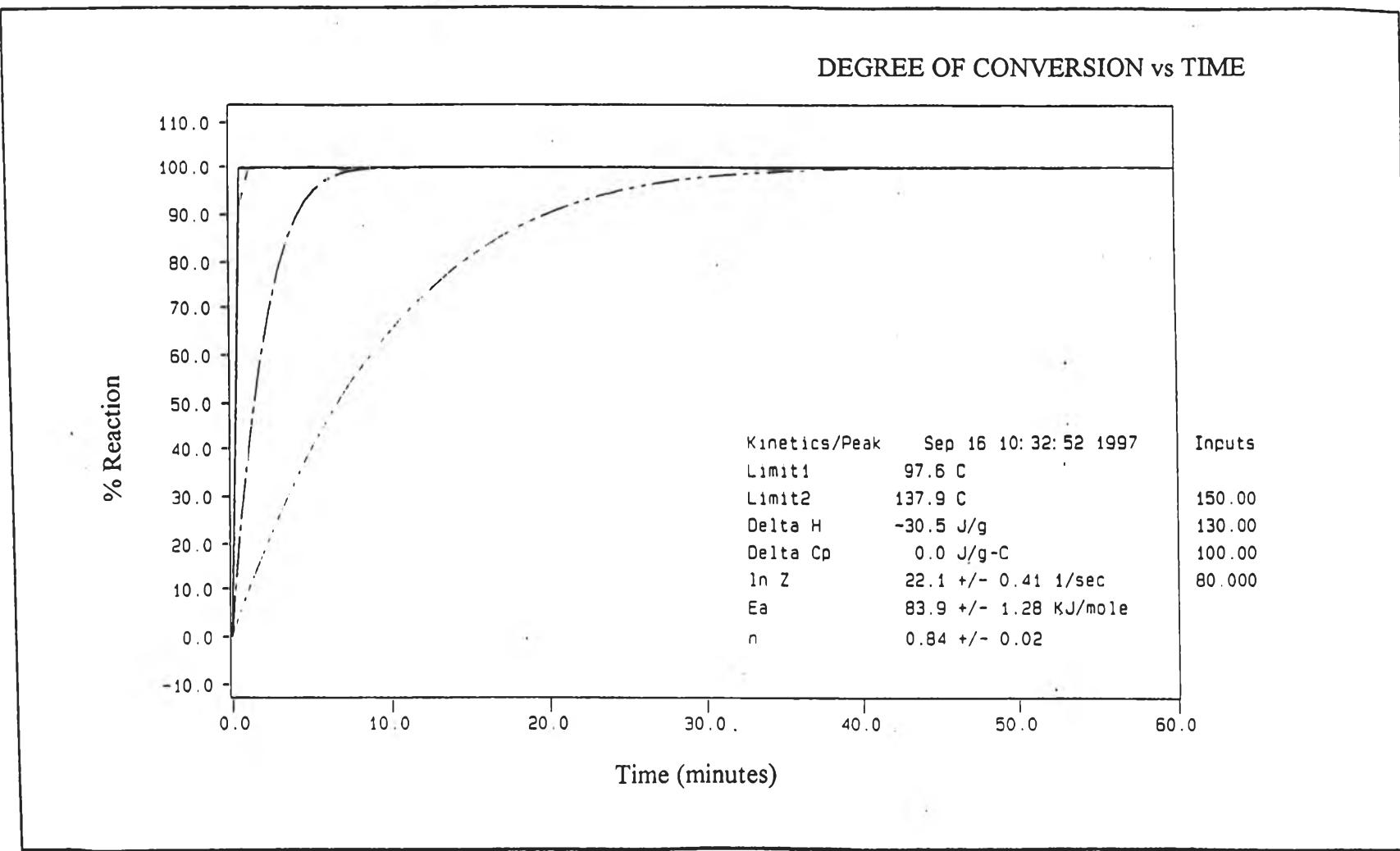
Figure 58   Kinetic DSC thermogram of the mixture of (9) and 5 mole % of Benzoyl Peroxide (24)
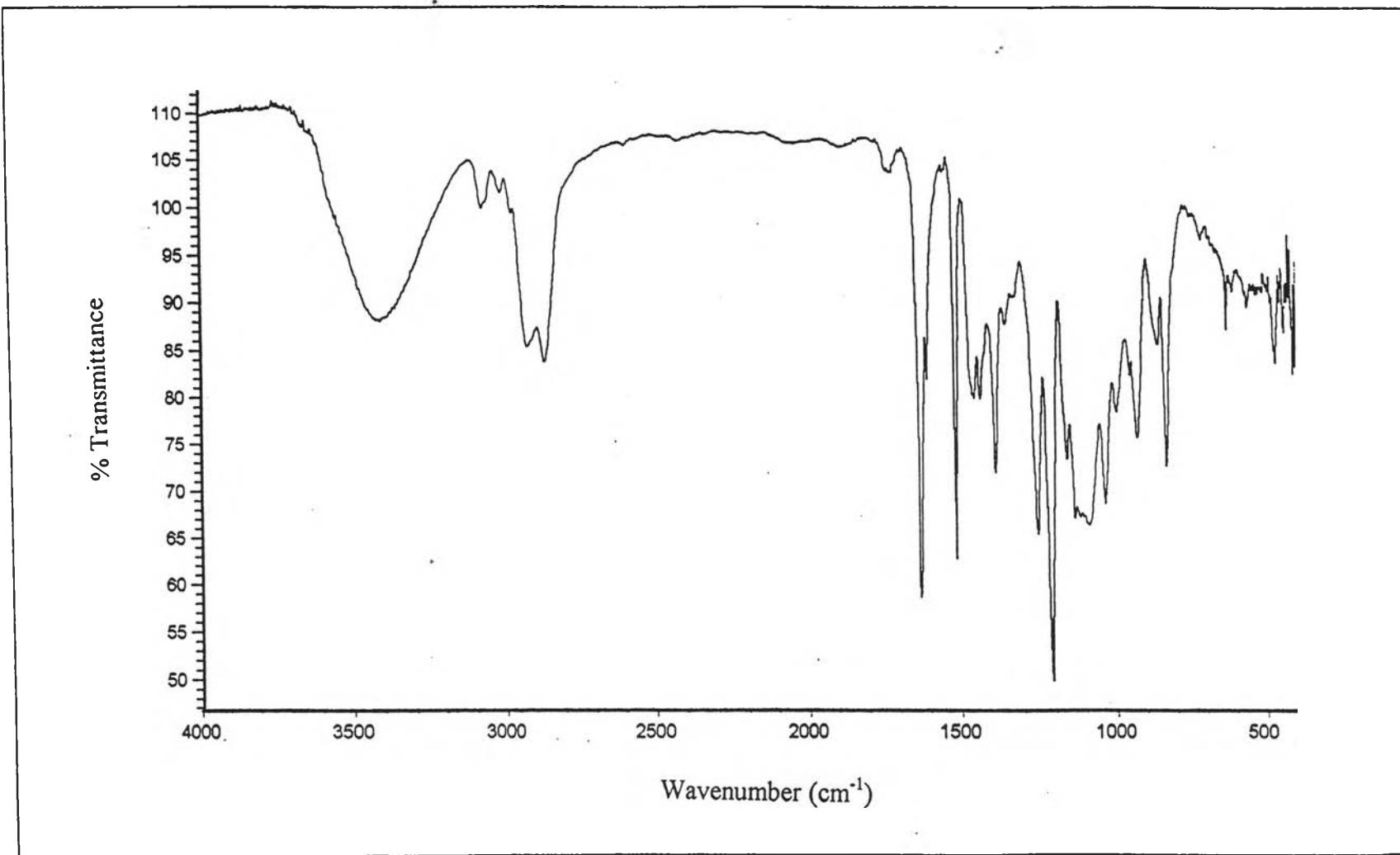
Figure 59   IR spectrum (KBr) of (26) obtained from the reaction between (9) and 5 mole % Benzoyl Peroxide (24) at 80°C for 24 hours
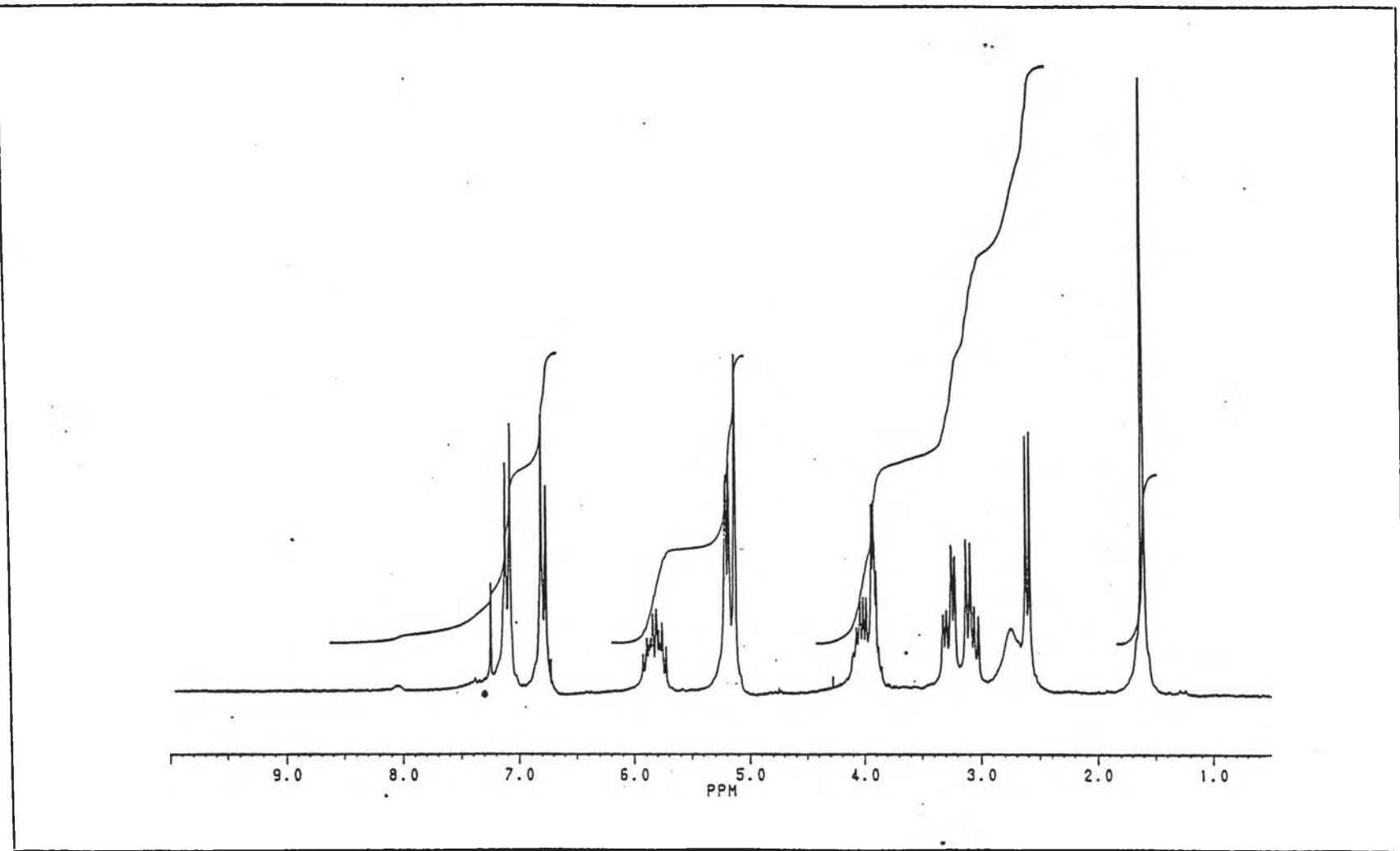
Figure 60 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (11) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 3 hours
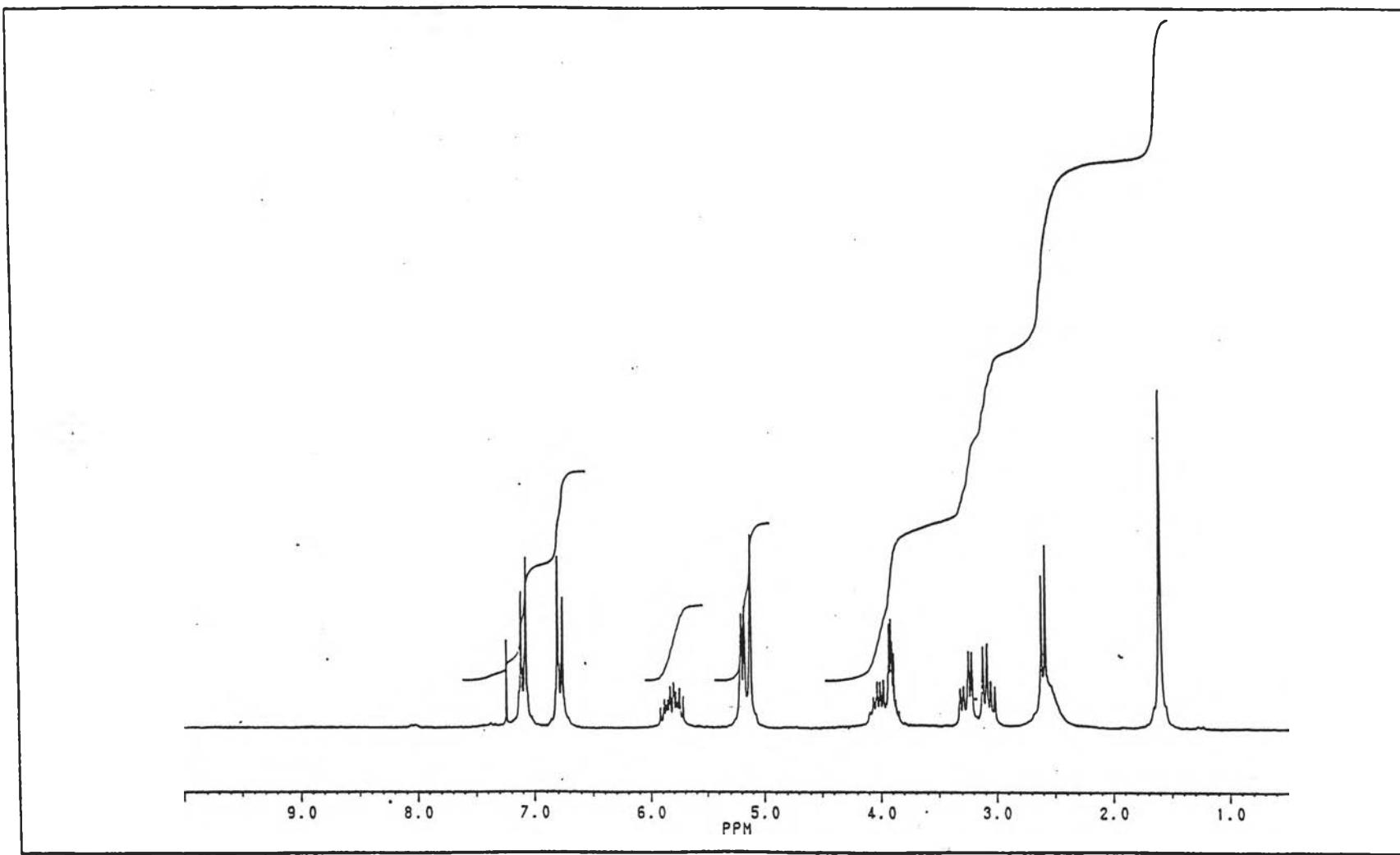
Figure 61 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (11) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 6 hours
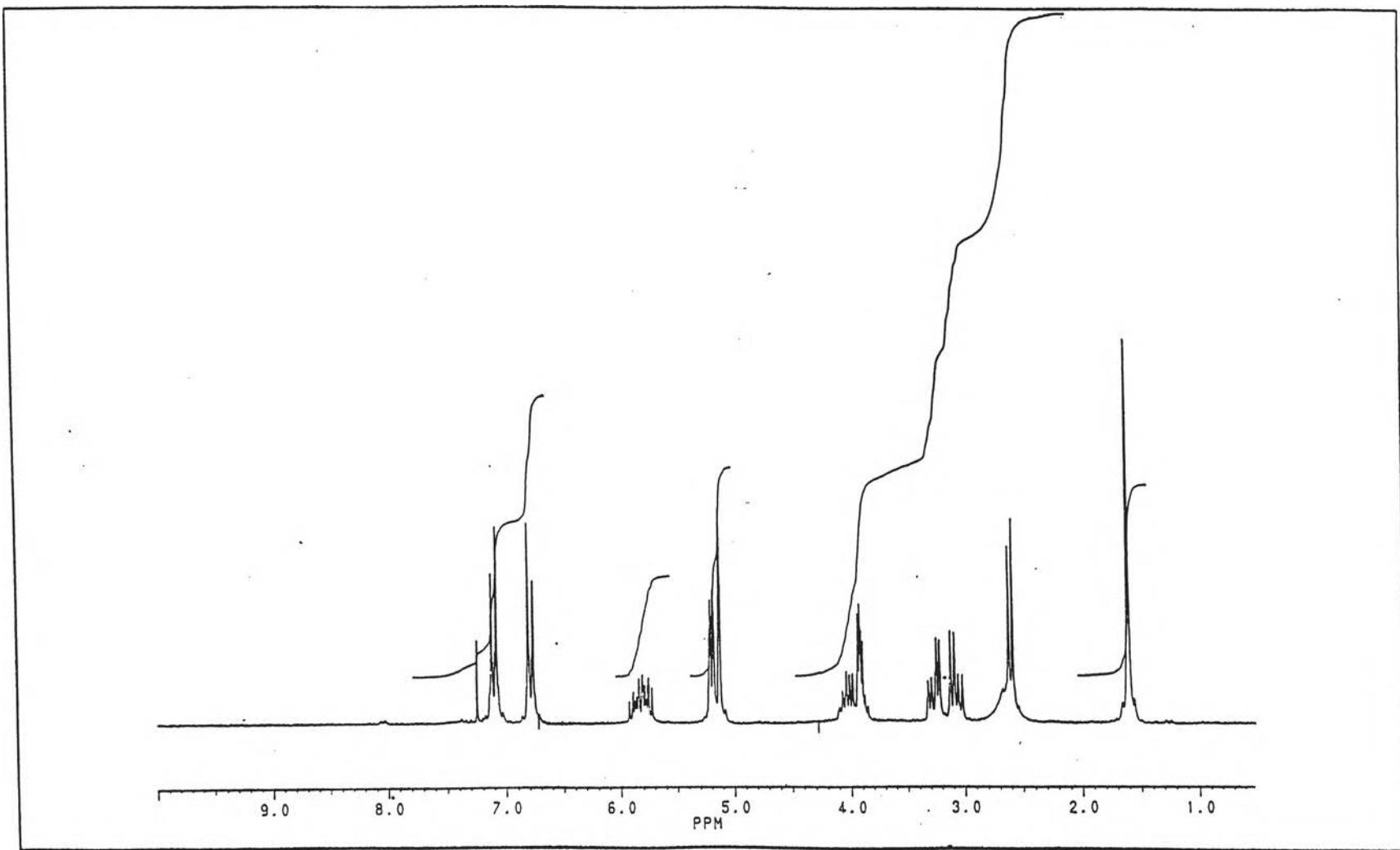
Figure 62 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (11) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 12 hours
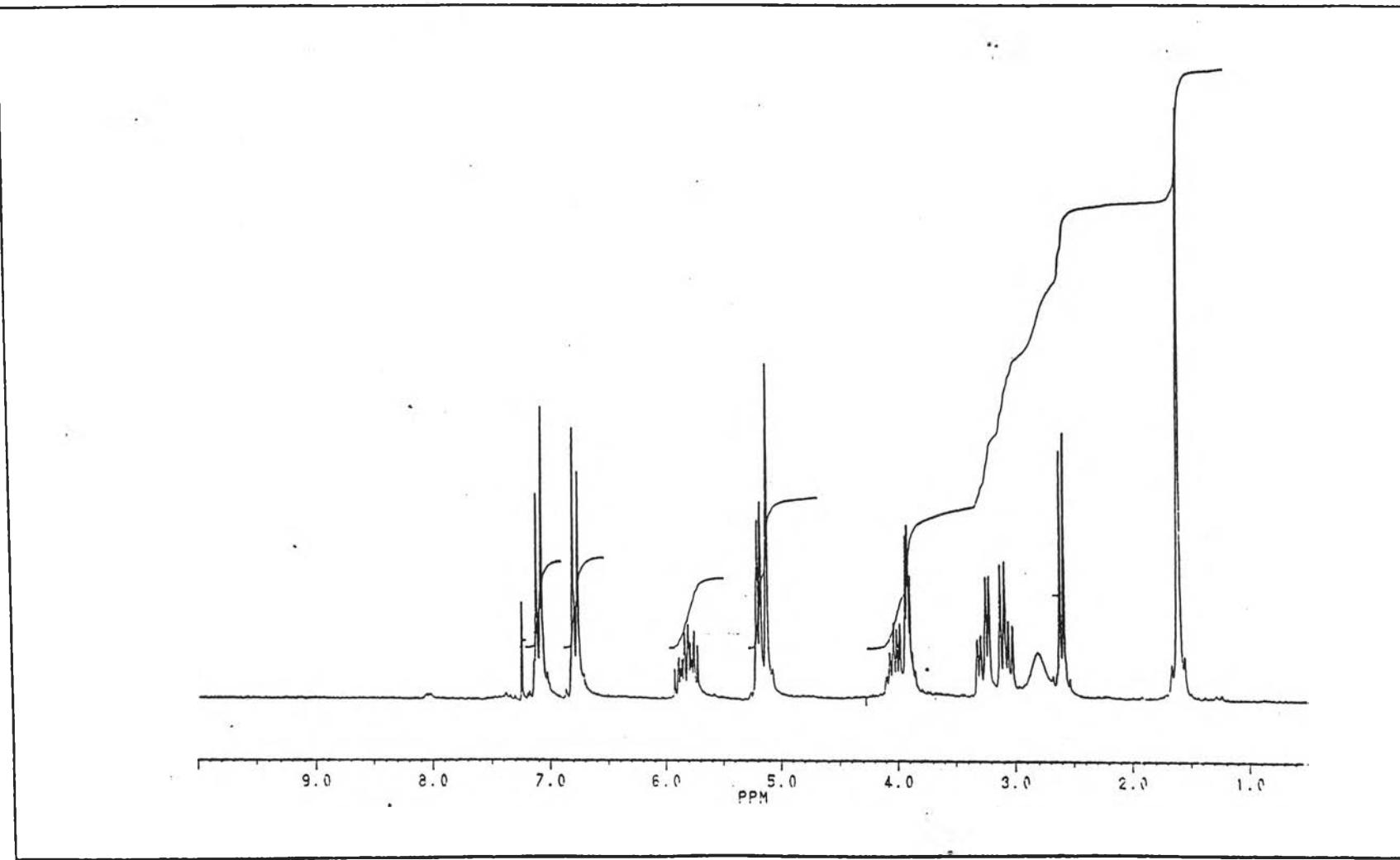
Figure 63 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (11) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 24 hours

Table 6   Integration ratio of H3 (olefinic protons) : H11 (aromatic protons) of
(27) at various reaction time

| Reaction time (hours)	Integration ratio of H3 (olefinic protons) : H11 (aromatic protons) of (27)
0	1.0
3	0.8
6	0.8
12	0.8
24	0.8
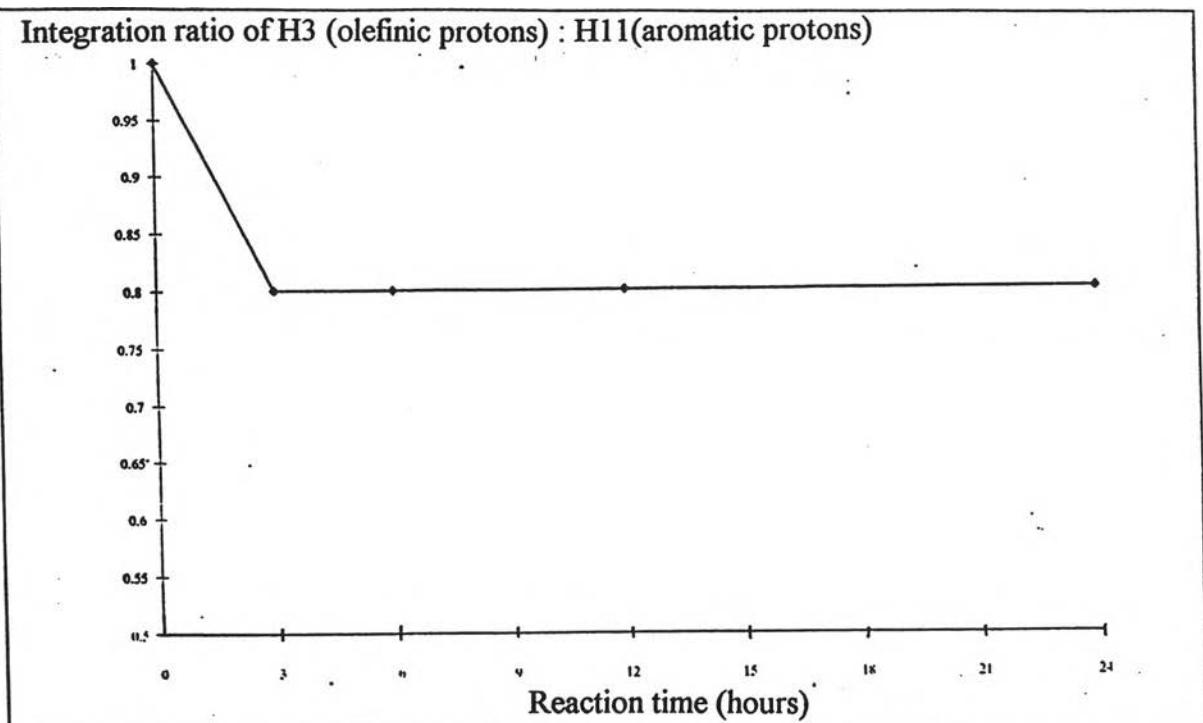
Figure 64   Relationship between the integration ratio of H3 (olefinic protons) : H11
(aromatic protons) of (27) and reaction time

Table 7   Integration ratio of H4 + H5 (allylic protons) : H11 (aromatic protons)
of (27) at various reaction time

Reaction time (hours)	Integration ratio of H4 + H5 (allylic protons) : H11 (aromatic protons) of (27)
0	2.00
3	1.69
6	1.66
12	1.59
24	1.55

Integration ratio of H4 + H5 (allylic protons) : H11(aromatic protons)


Reaction time (hours)
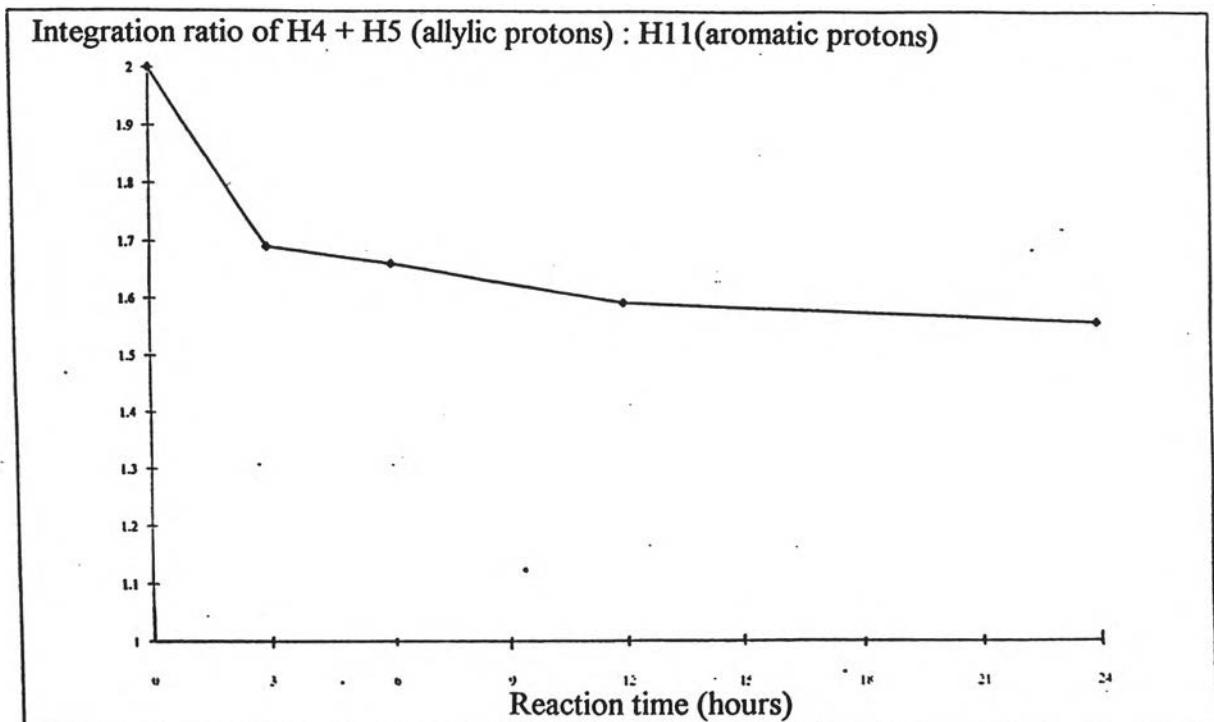
Figure 65   Relationship between the integration ratio of H4 + H5 (allylic protons)
: H11 (aromatic protons) of (27) and reaction time
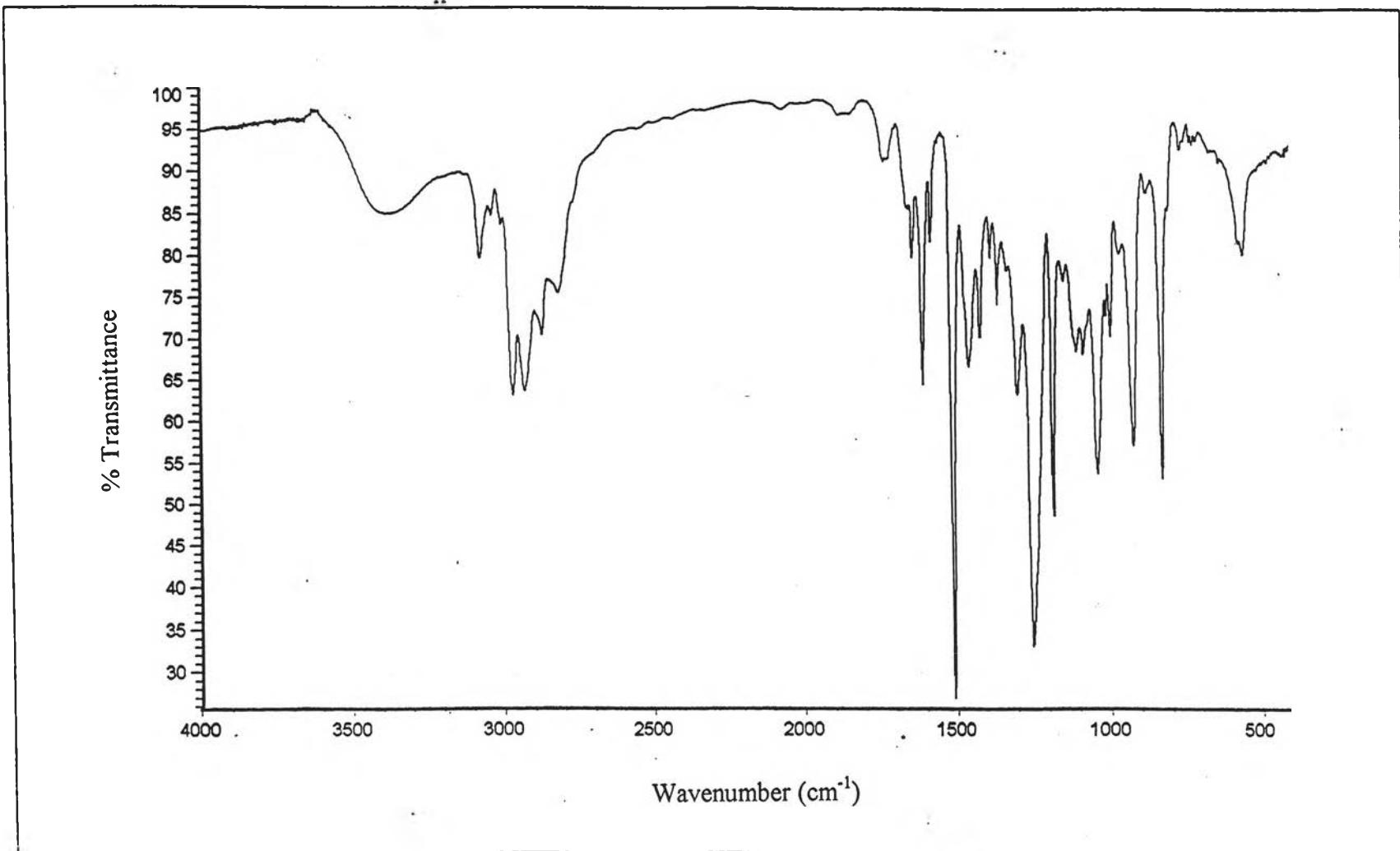
Figure 66   IR spectrum (KBr) of (27) obtained from the reaction between (11) and 5 mole % Benzoyl Peroxide (24) at 80°C for 24 hours
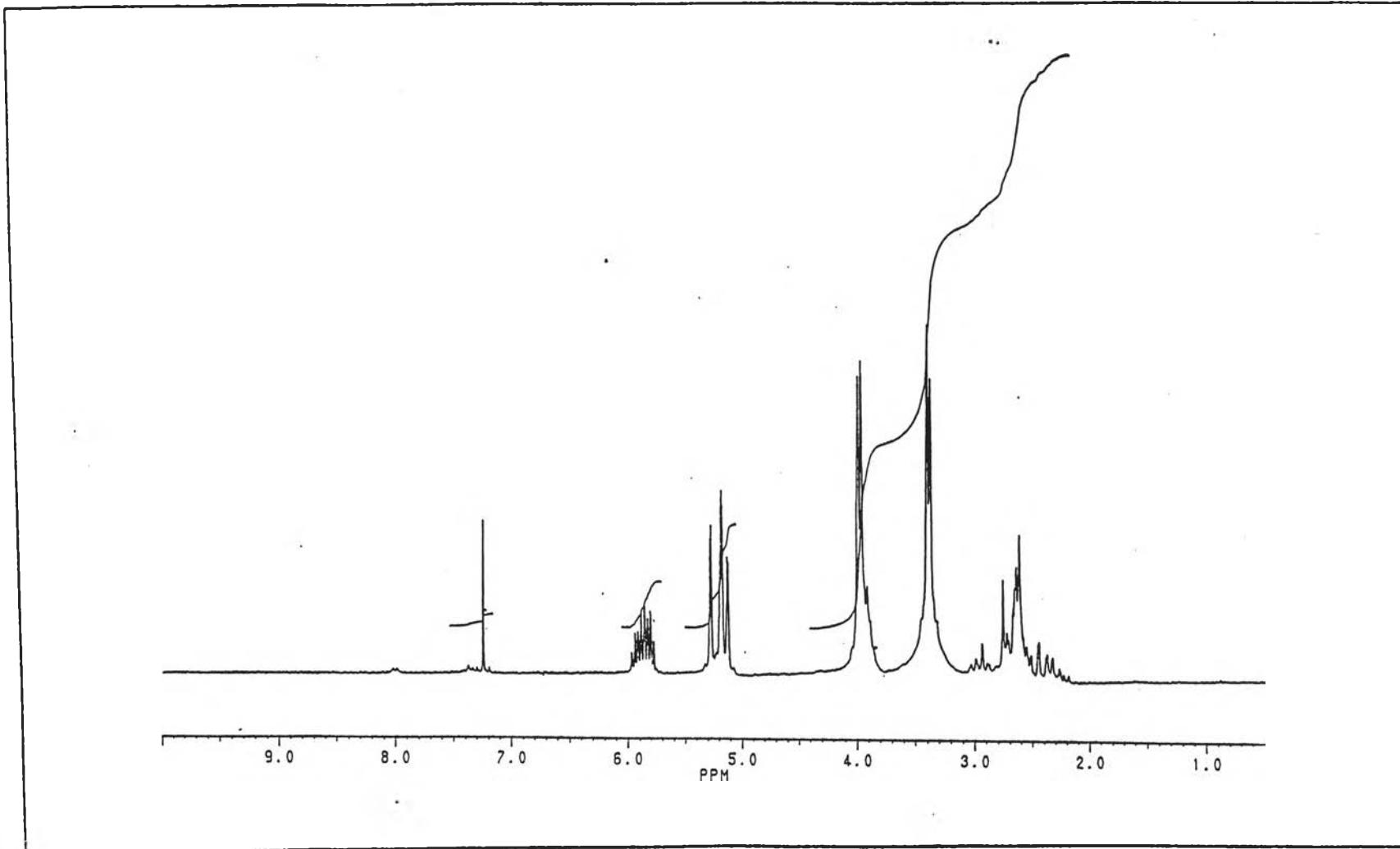
Figure 67 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (13) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 3 hours
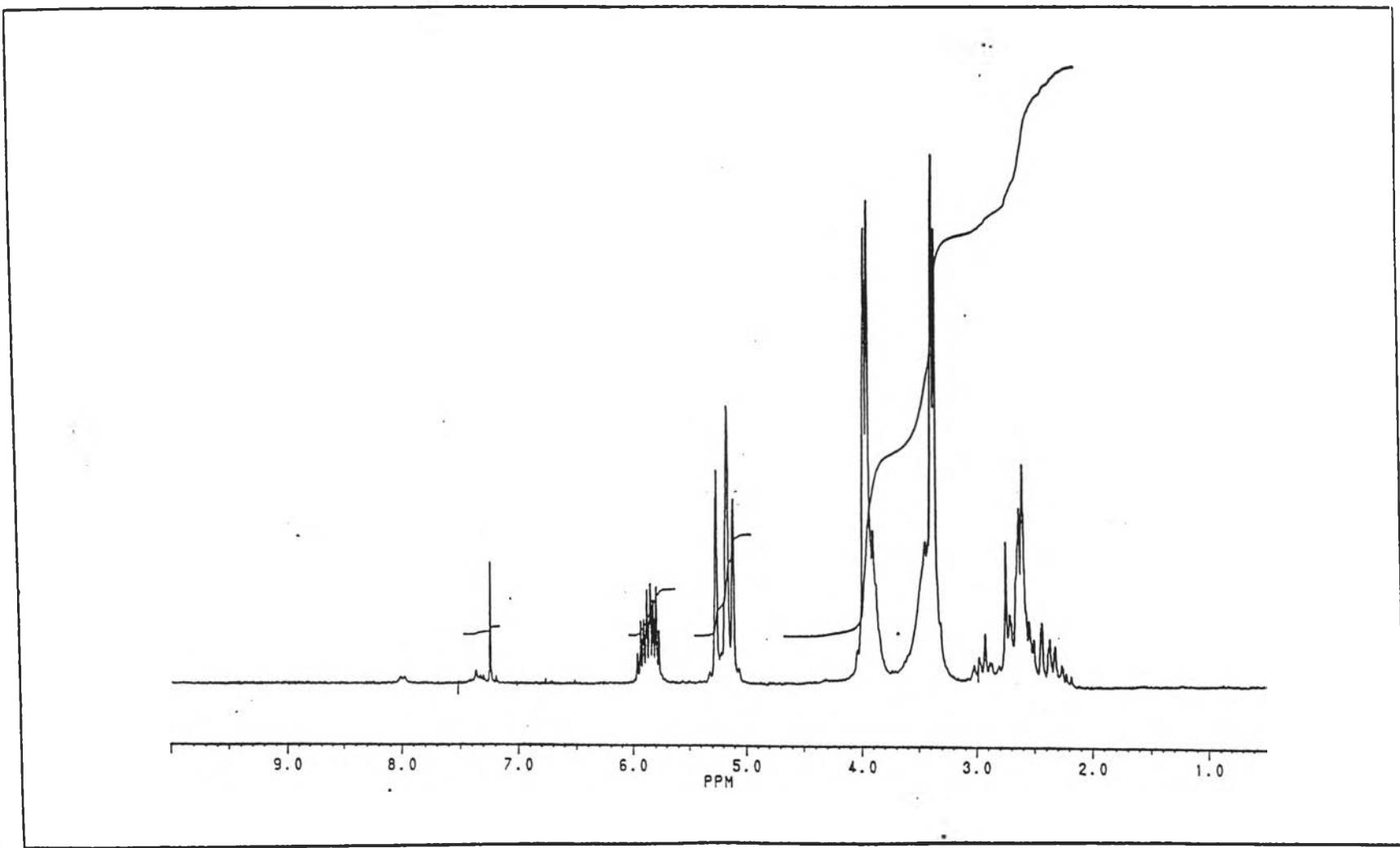
Figure 68 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (13) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 6 hours
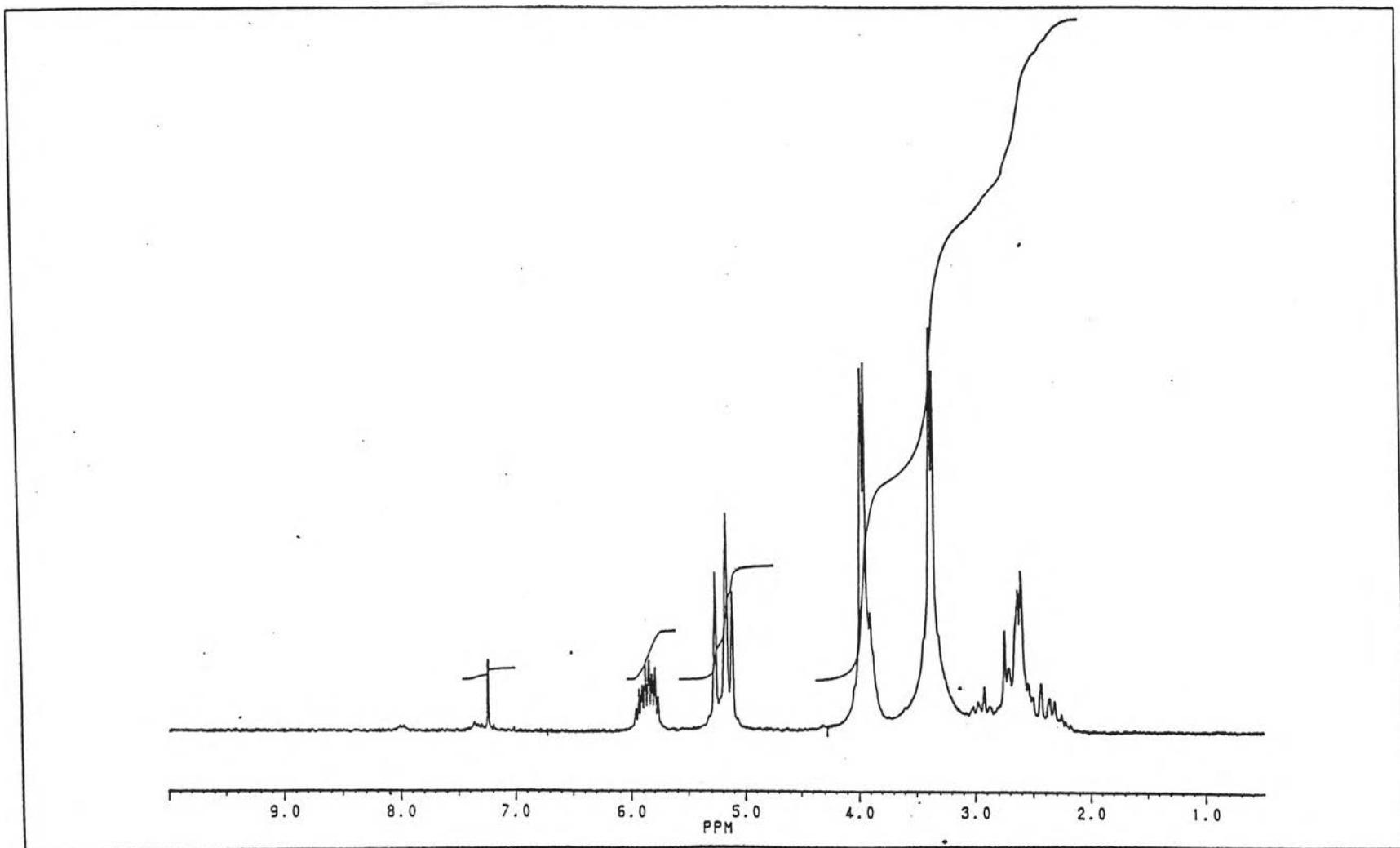
Figure 69 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (13) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 12 hours
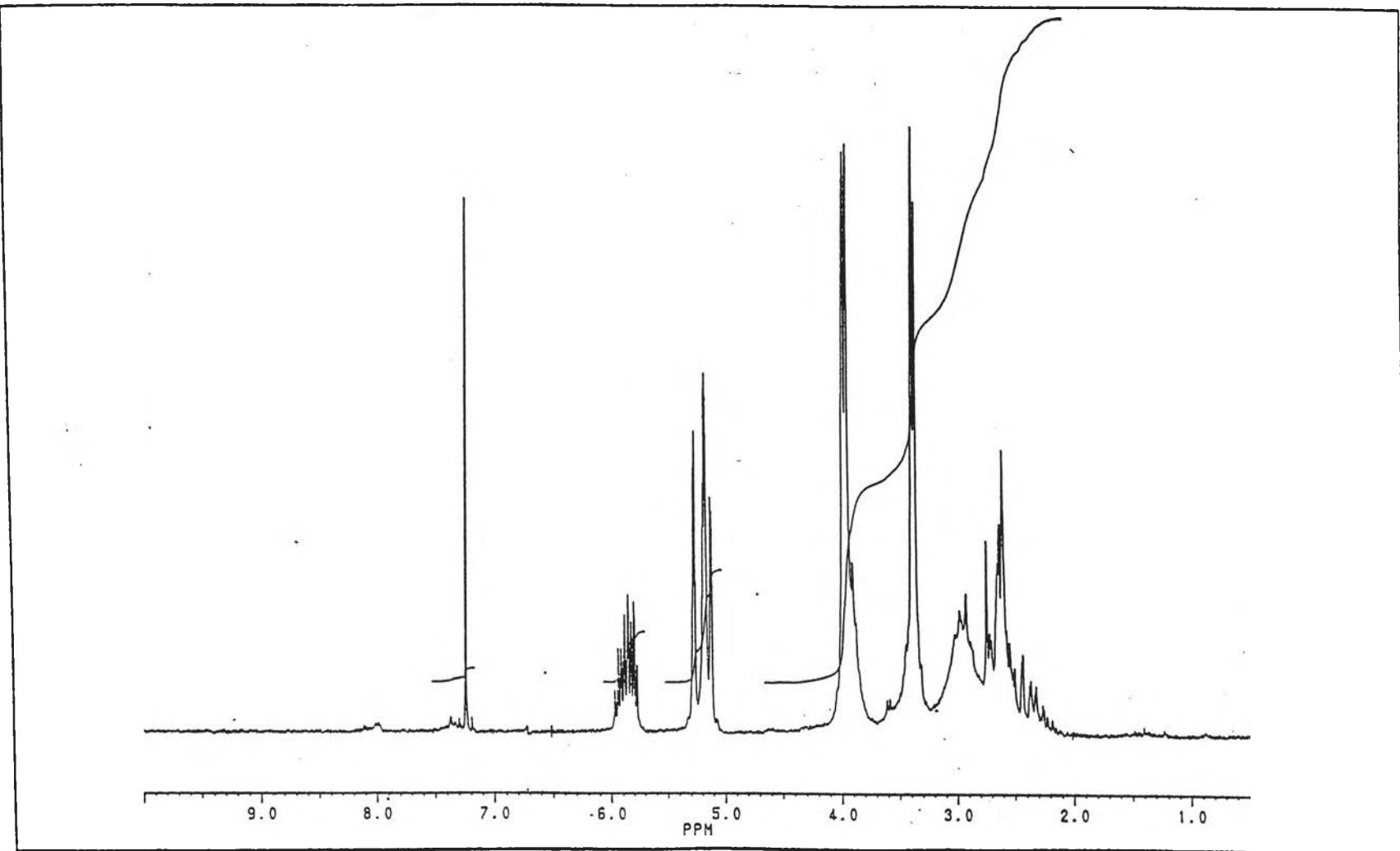
Figure 70 ^{1}H NMR (CDCl$_3$) spectrum of the reaction between (13) and 5 mole % of Benzoyl Peroxide (24) at 80°C for 24 hours

Table 8   Integration ratio of H3 (olefinic protons) : H9 - H12 (aliphatic protons) of (28) at various reaction time

| Reaction time (hours)	Integration ratio of H3 (olefinic protons) : H9 - H12 (aliphatic protons) of (28)
0	0.30
3	0.29
6	0.29
12	0.26
24	0.18
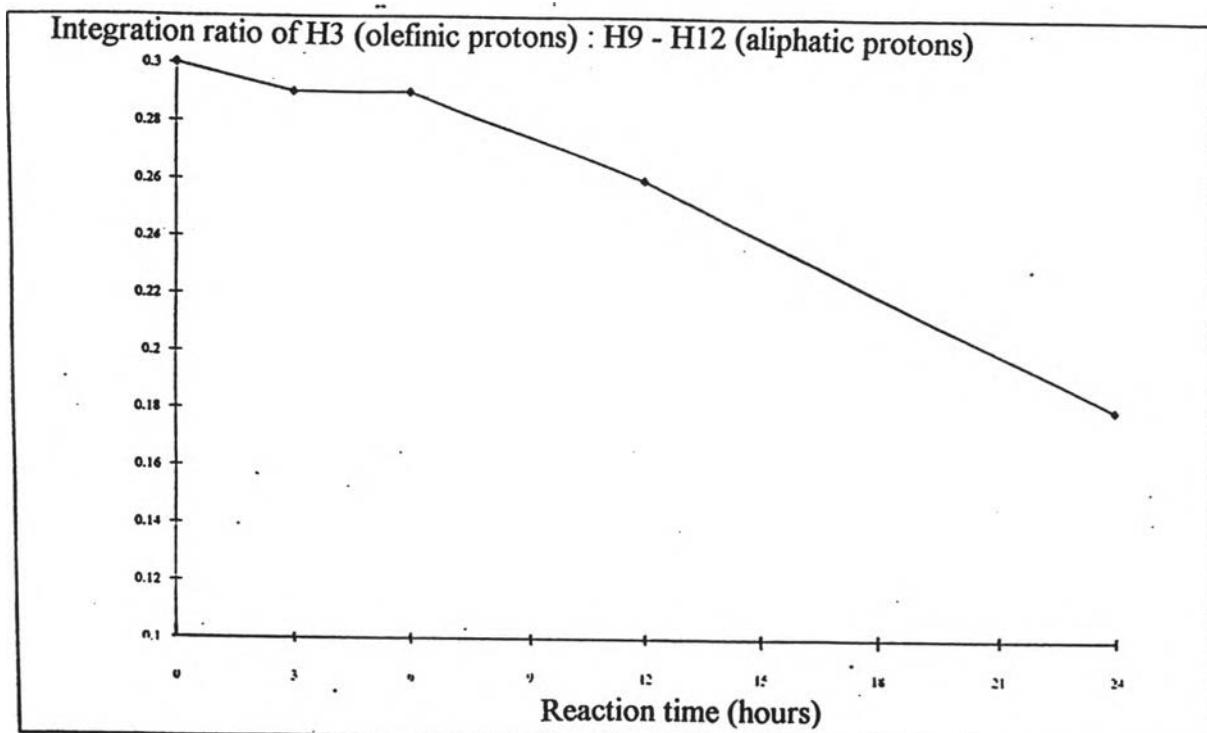
Figure 71   Relationship between the integration ratio of H3 (olefinic protons) : H9 - H12 (aliphatic protons) of (28) and reaction time

Table 9   Integration ratio of H4 + H5 + H8 (olefinic protons + H8) :

H9 - H12 (aliphatic protons) of (28) at various reaction time

Reaction time (hours)	Integration ratio of H4 + H5 + H8 (allylic protons + H8) : H9 - H12 (aliphatic protons) of (28)
0	1.05
3	1.00
6	1.00
12	1.00
24	0.66
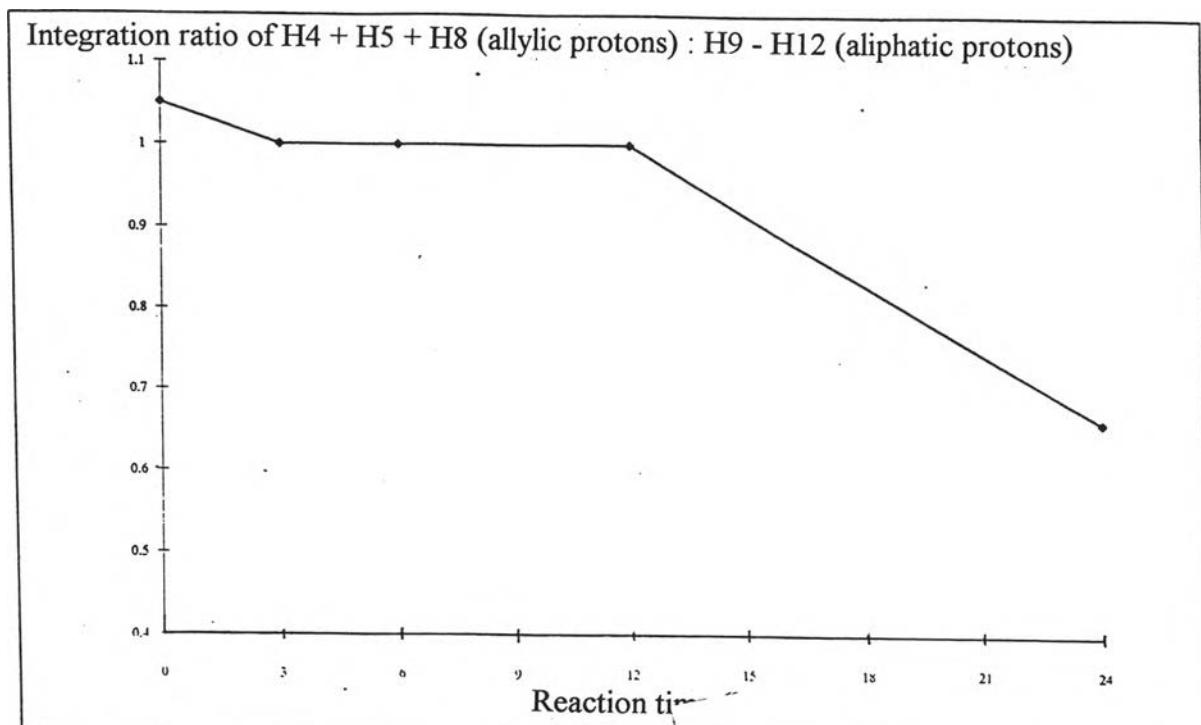
Figure 72   Relationship between the integration ratio of H4 + H5 + H8 (allylic protons + H8) : H9 - H12 (aliphatic protons) of (28) and reaction time
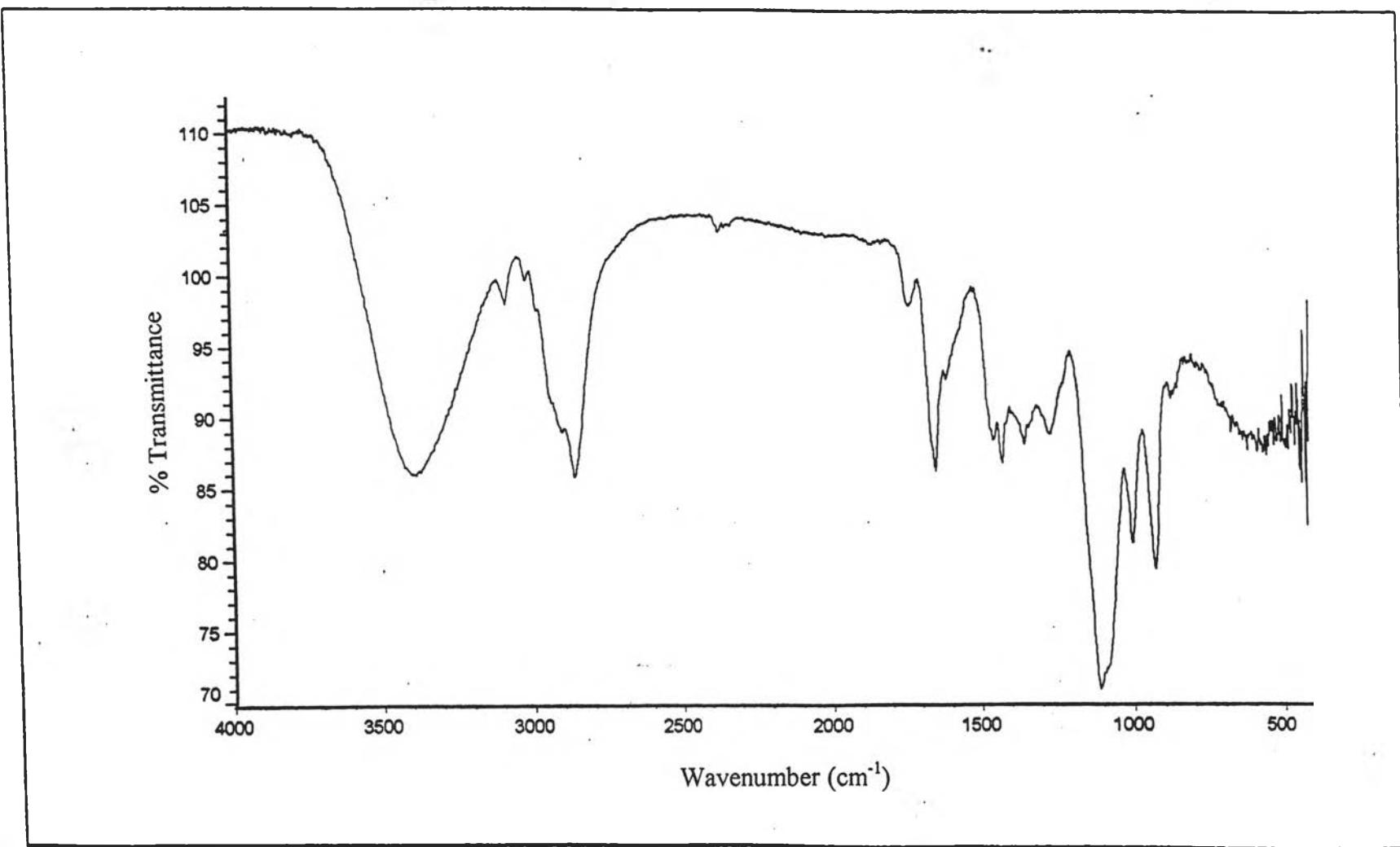
Figure 73   IR spectrum (KBr) of (28) obtained from the reaction between (13) and 5 mole % Benzoyl Peroxide (24) at 80°C for 24 hours
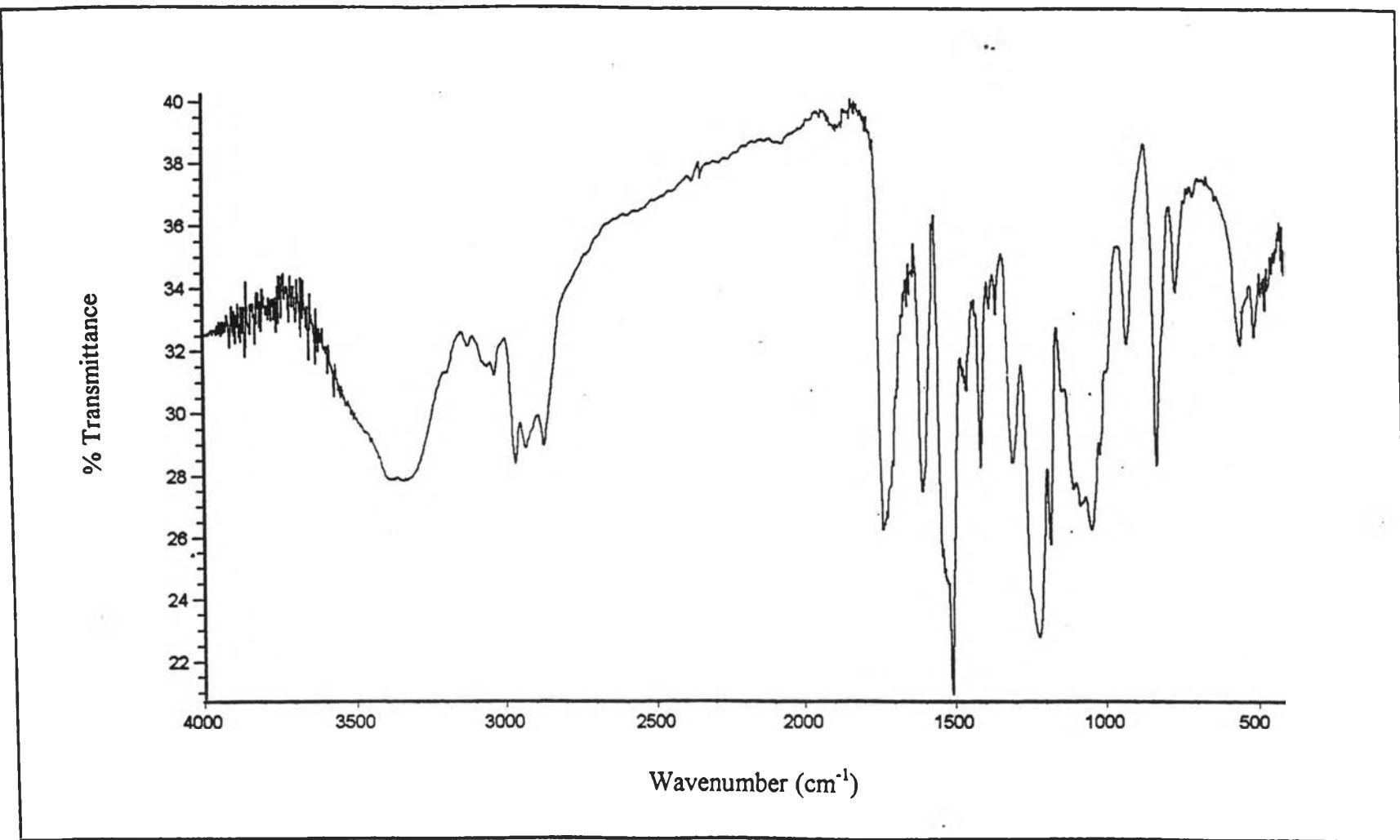
Figure 74   IR spectrum (KBr) of Polyurethane(29)
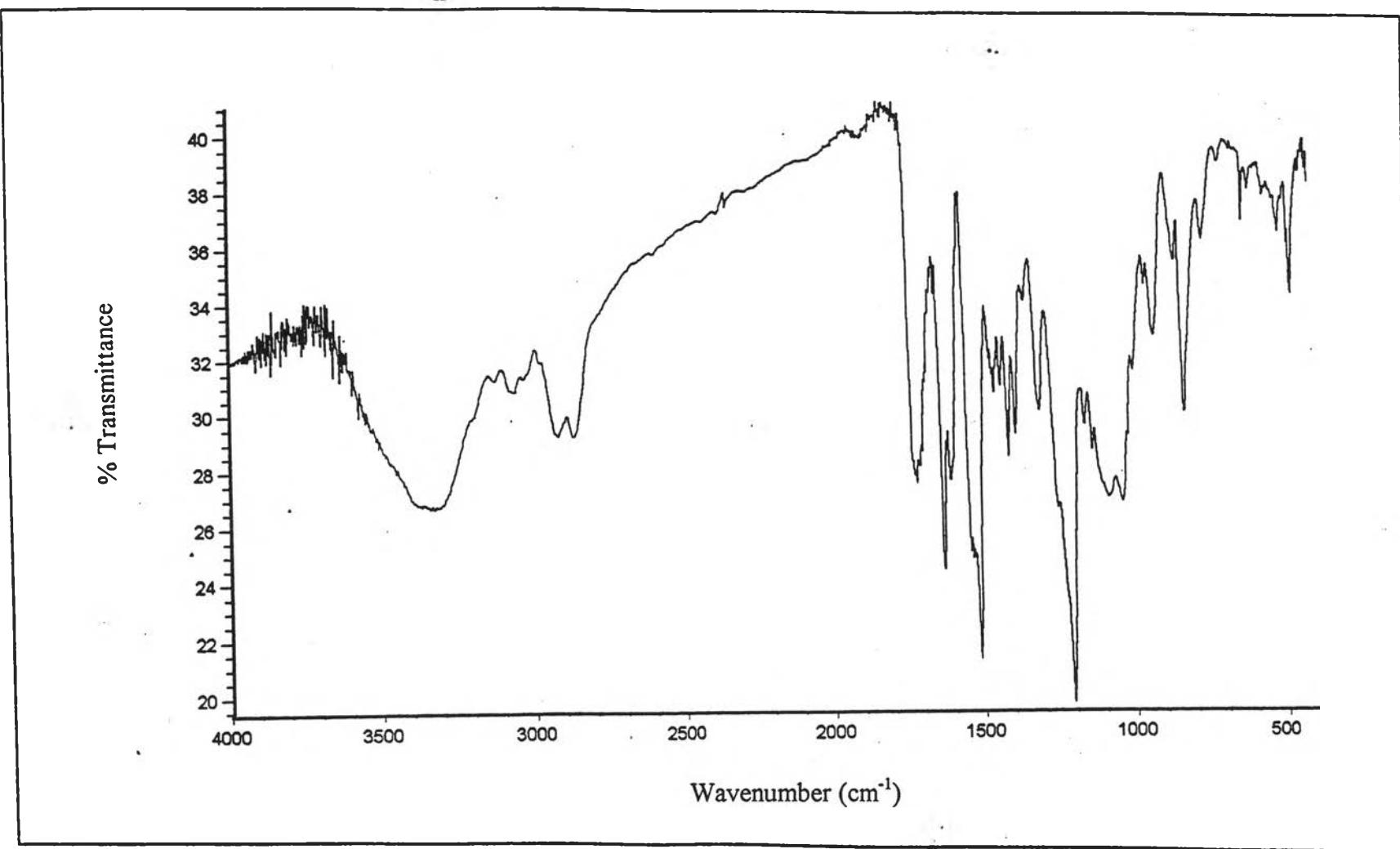
Figure 75   IR spectrum (KBr) of Polyurethane(**30**)
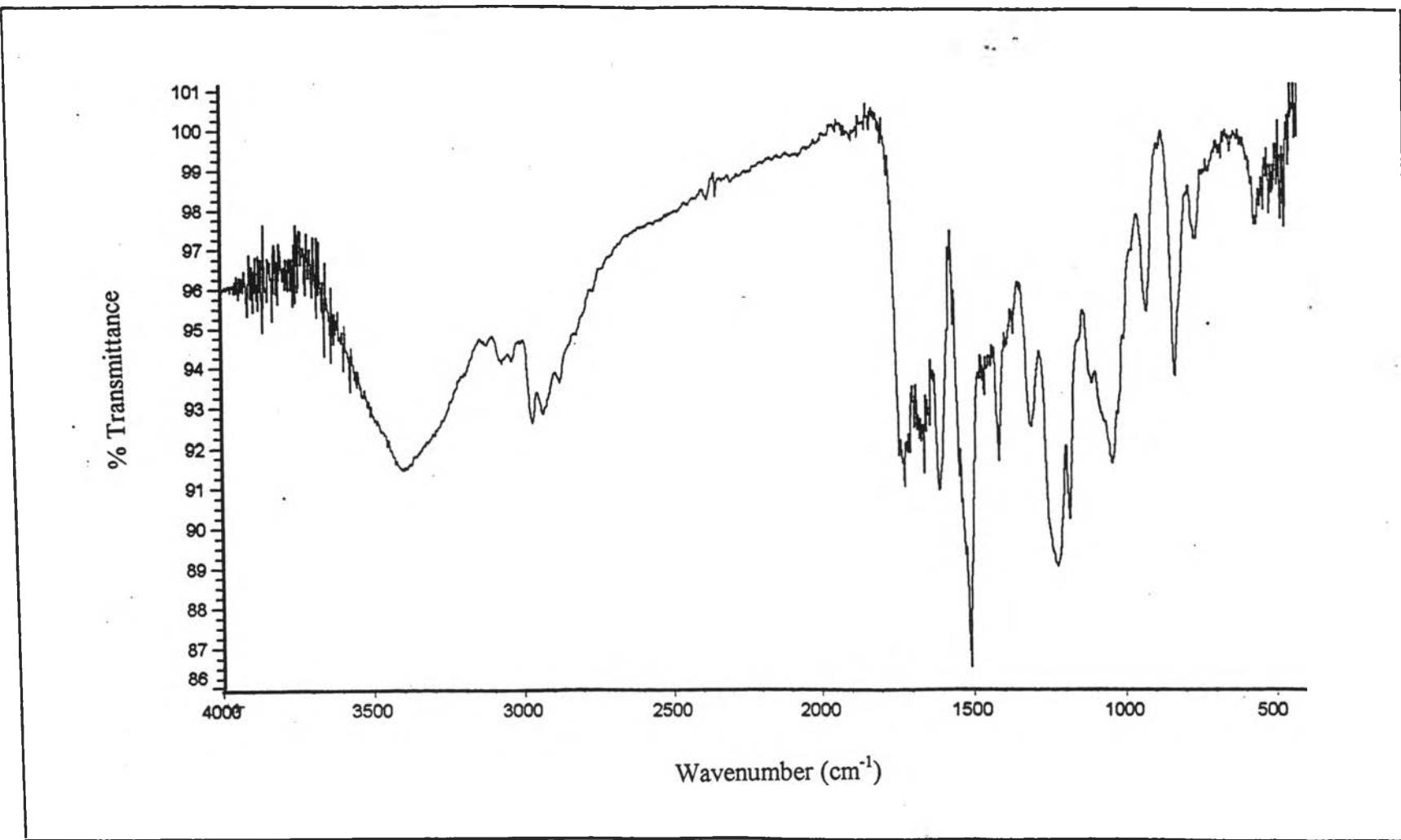
Figure 76   IR spectrum (KBr) of Polyurethane(**31**)
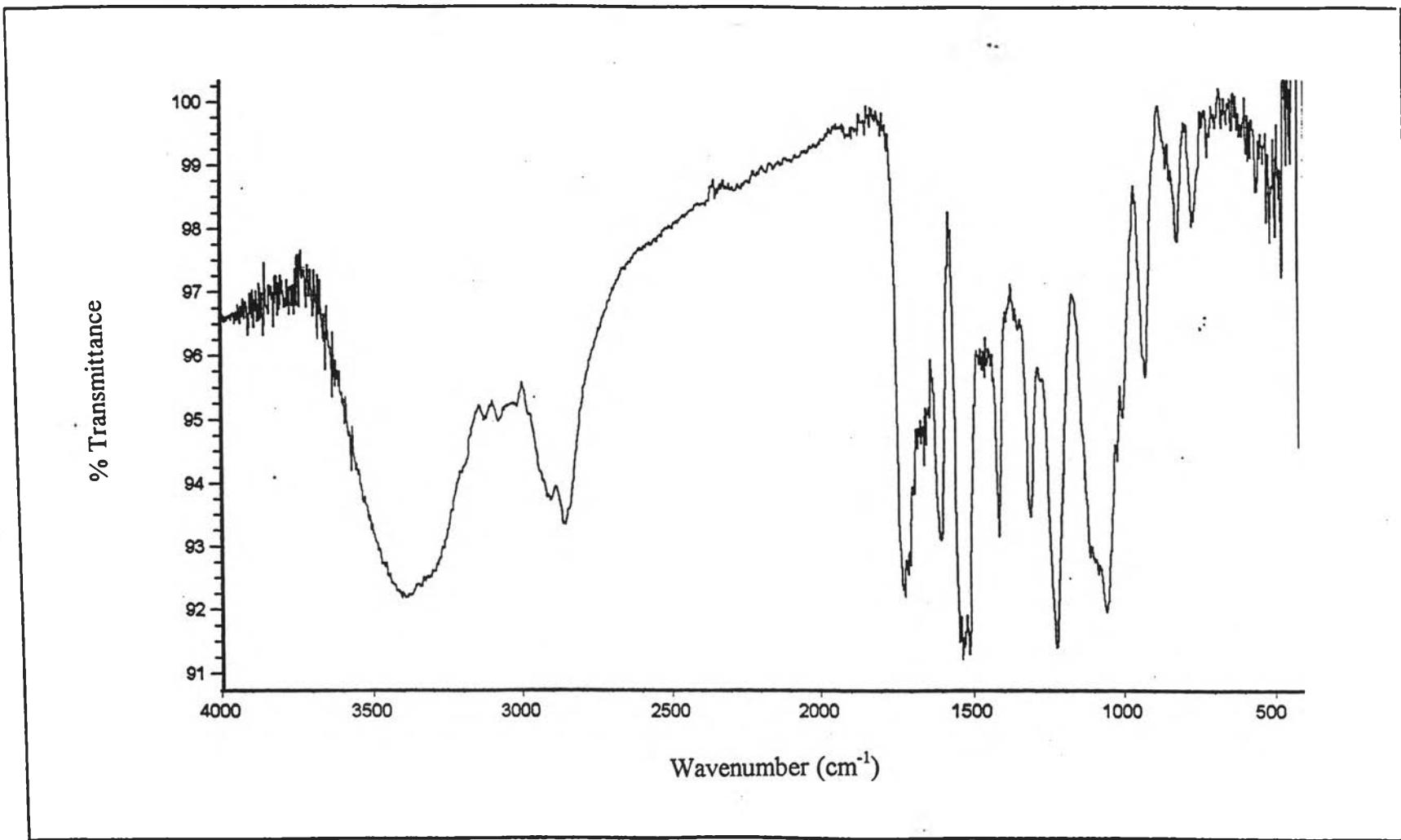
Figure 77   IR spectrum (KBr) of Polyurethane(**32**)
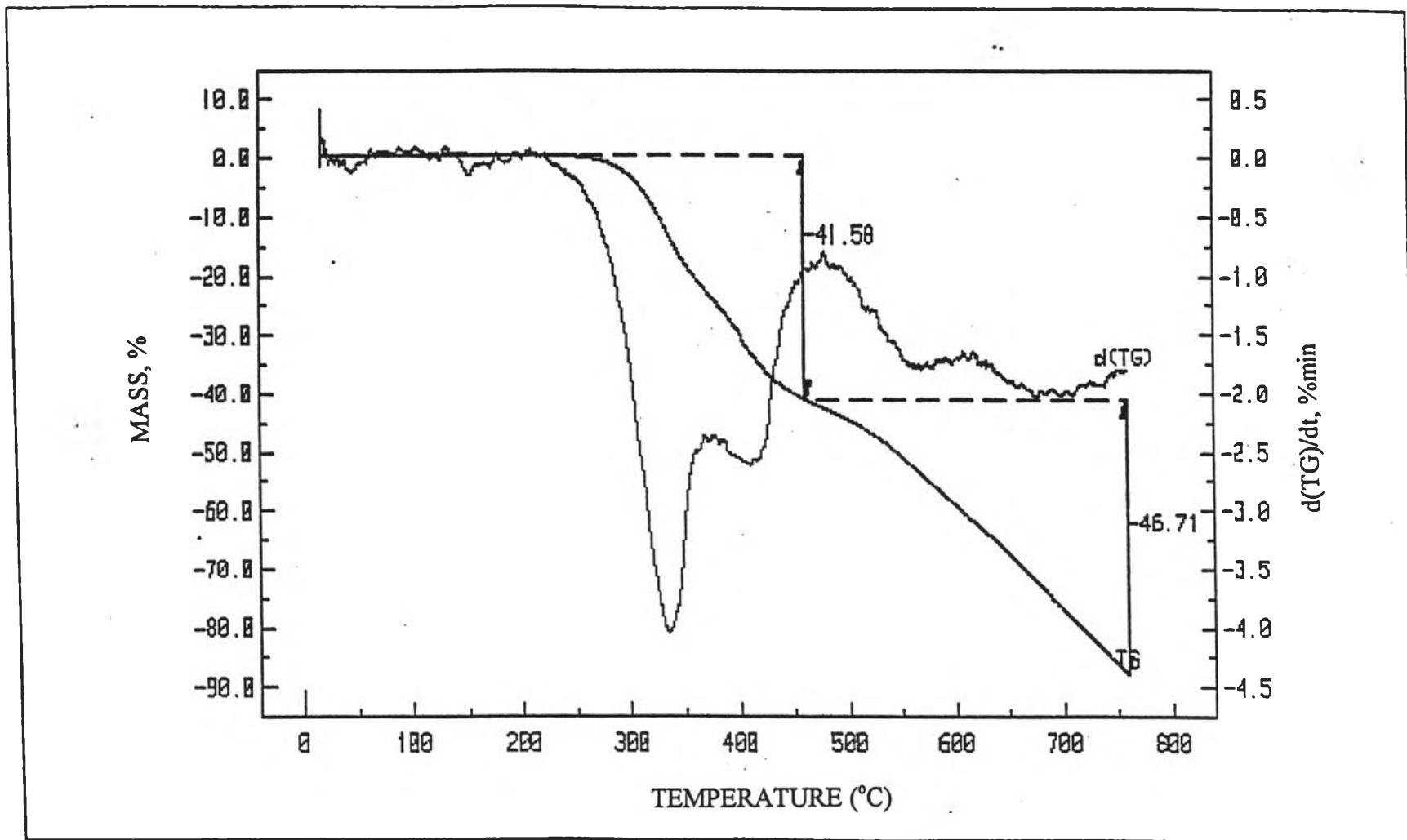
Figure 78   TGA thermogram of Polyurethane(29)
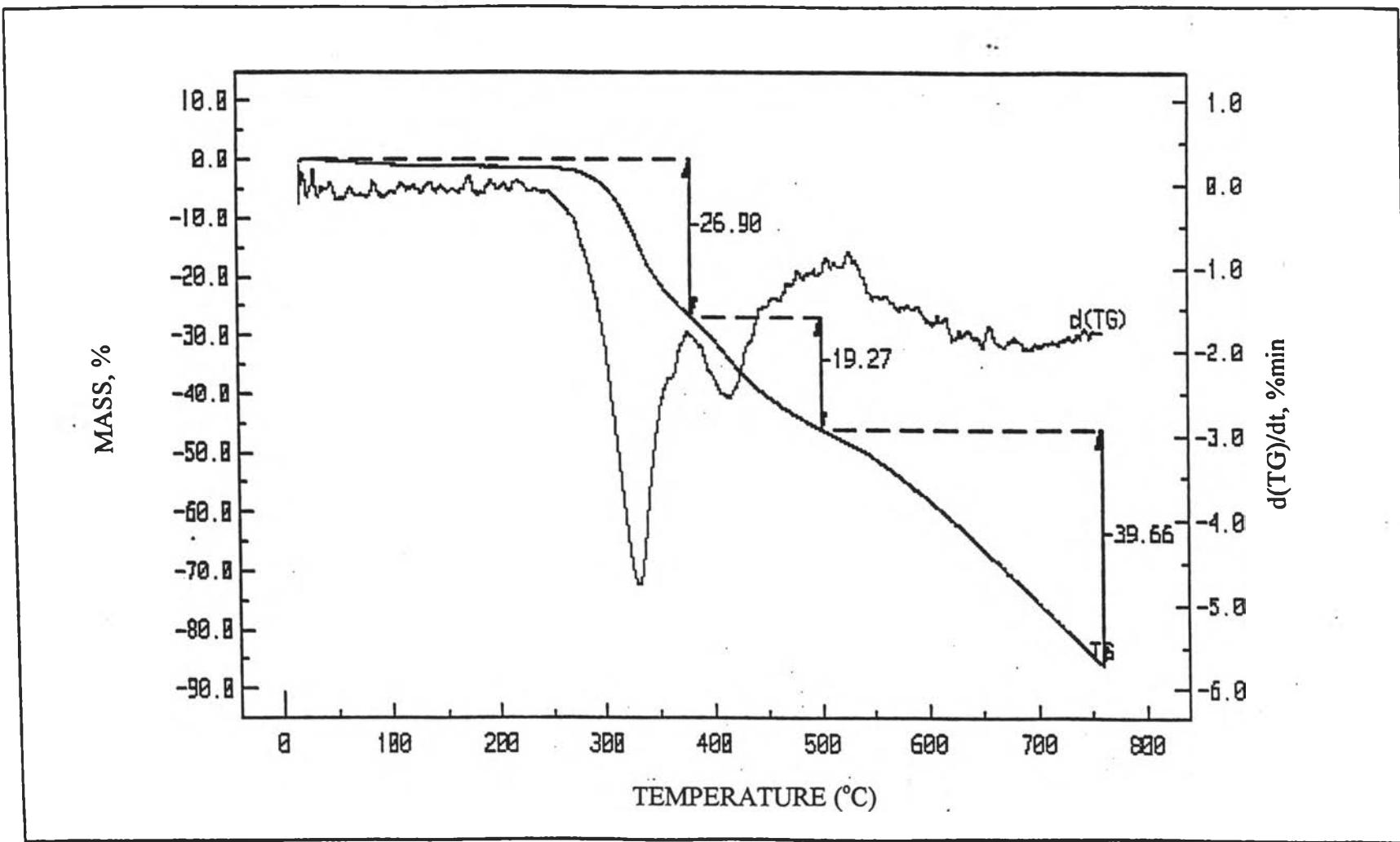
Figure 79   TGA thermogram of Polyurethane(**30**)
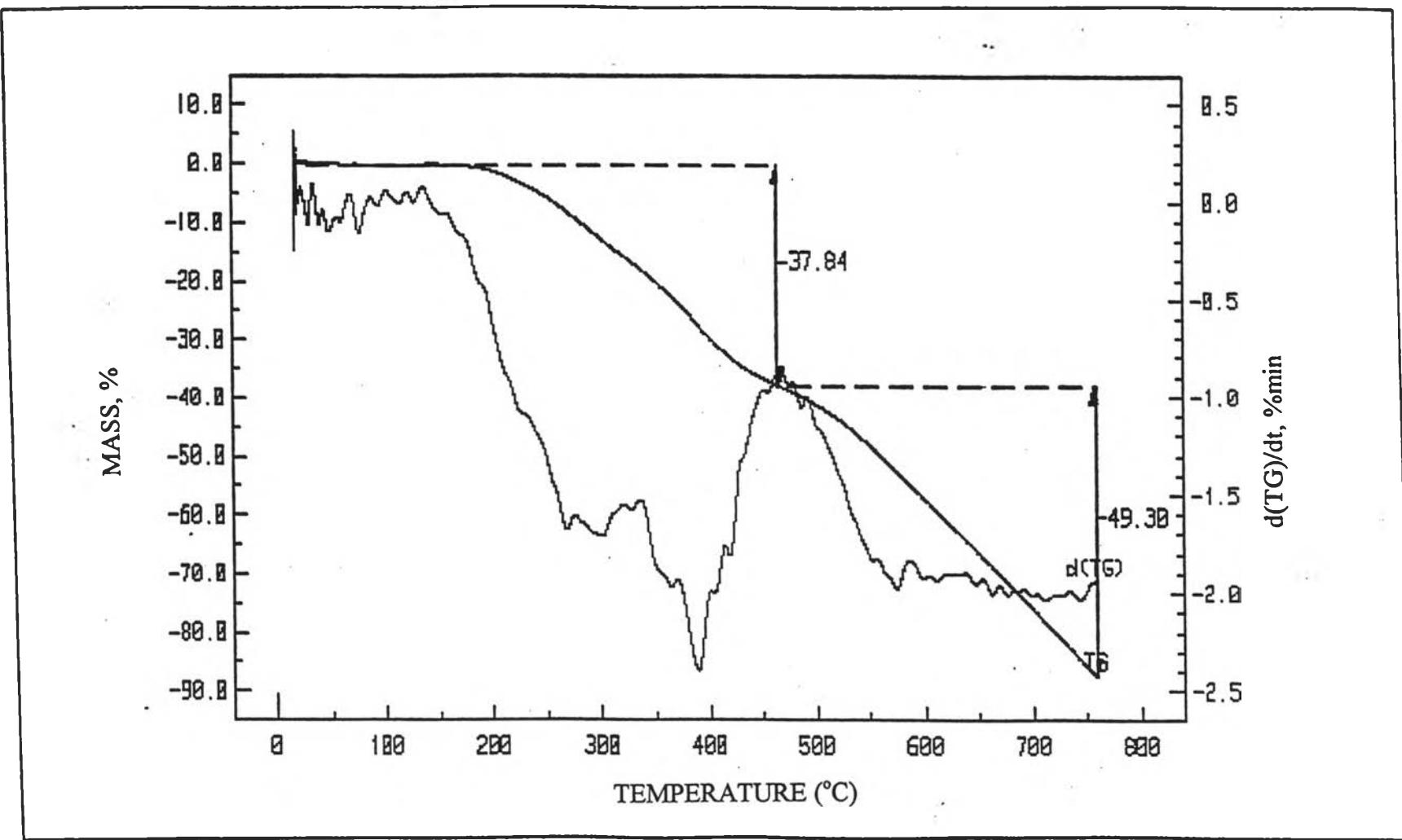
Figure 80   TGA thermogram of Polyurethane(**31**)   .
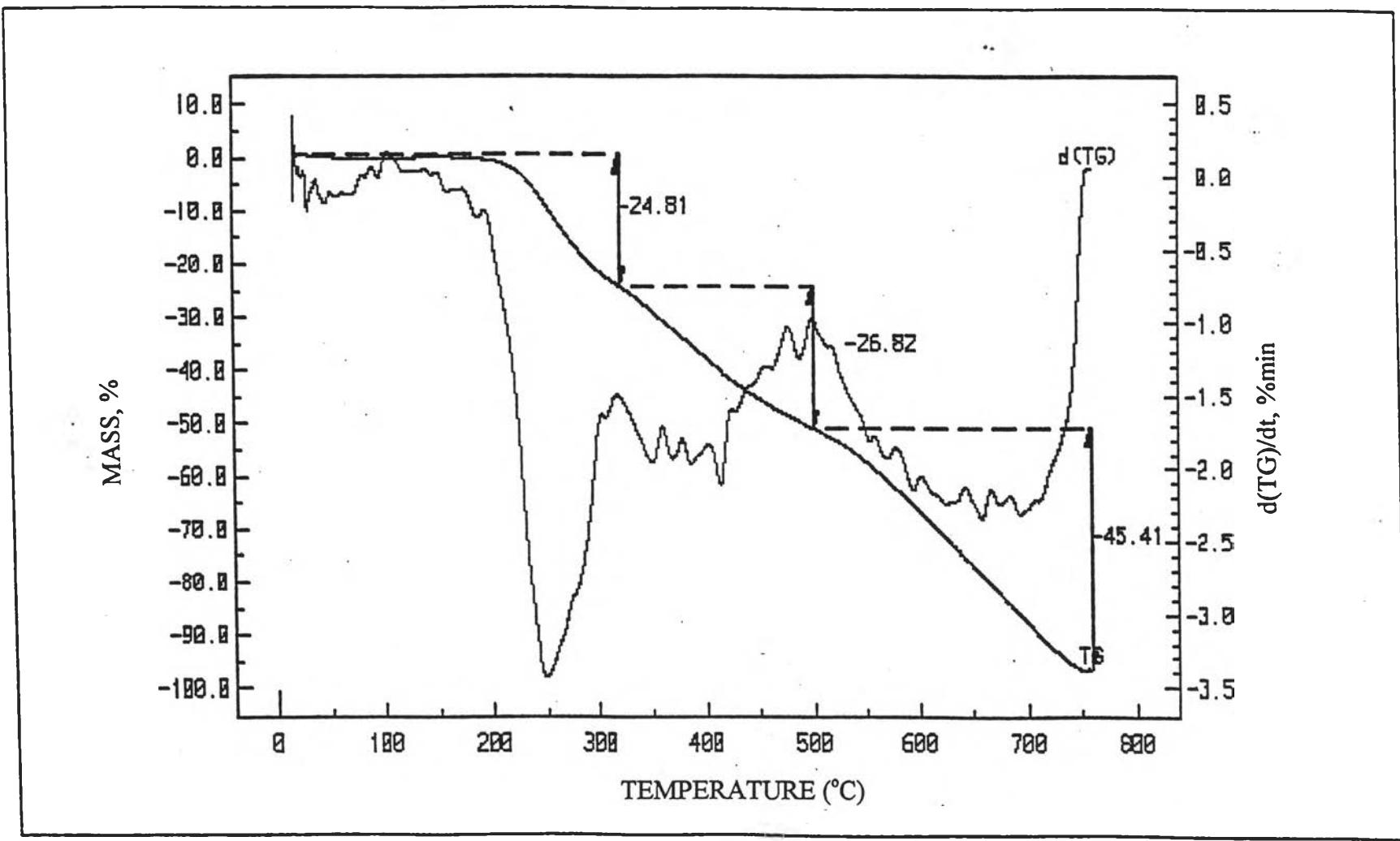
Figure 81   TGA thermogram of Polyurethane(**32**)

VITA

Nawee Farkrachang was born on December 23, 1973 in Lopburi, Thailand. He received the Bachelor Degree of Science in Chemistry from Chulalongkorn University in 1994. In the same year, he became a student in graduate school at Chulalongkorn University studying in Chemistry and has been studying since then. He graduated with the Master Degree of Science in 1997.