

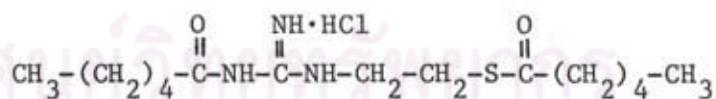
CHAPTER IV

CONCLUSIONS

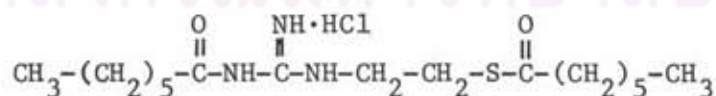
In the course of this research 19 compounds were synthesised including 6 starting materials of N,S-diacyl-2-mercaptoethylguanidine hydrochlorides (S-series compounds) and their thermal reaction products. The thermal reactions of the starting materials resulted in the formation of 2-amino-2-thiazoline hydrochloride (A-series compound) in all cases and 6 of each series of aliphatic primary amides (B-series compounds) and N,N'-diacyl-cystamines (C-series compounds). The structures of all the reaction products were inclusively characterized by spectroscopic and chemical methods.

The following list shows the structures of all the compounds made in this course:

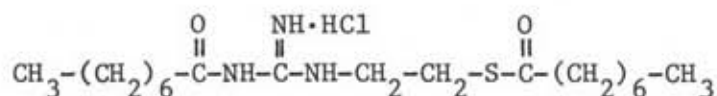
S-series compounds



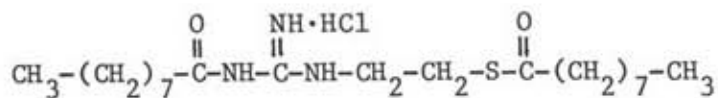
(N,S-dihexanoyl-2-mercaptoethylguanidine hydrochloride)



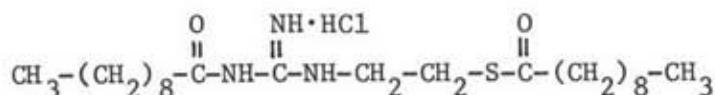
(N,S-diheptanoyl-2-mercaptoethylguanidine hydrochloride)



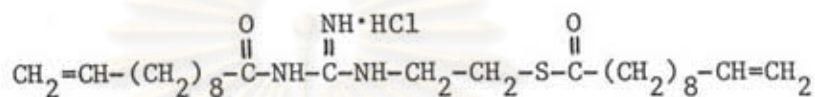
(N,S-dioctanoyl-2-mercaptoethylguanidine hydrochloride)



(N,S-dinonanoyl-2-mercaptoethylguanidine hydrochloride)

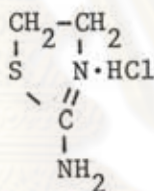


(N,S-didecanoyl-2-mercaptoethylguanidine hydrochloride)



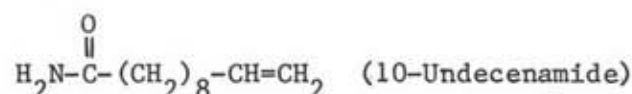
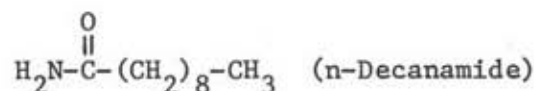
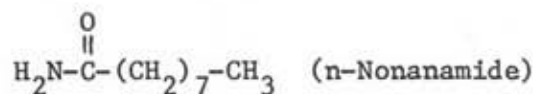
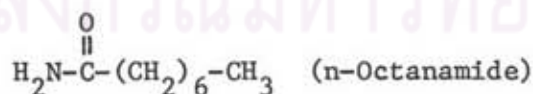
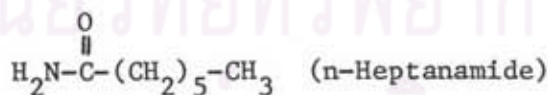
(N,S-di-10-undecenoyl-2-mercaptoethylguanidine hydrochloride)

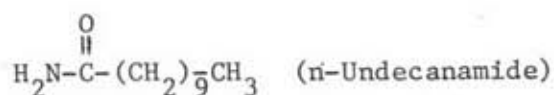
A-series compound



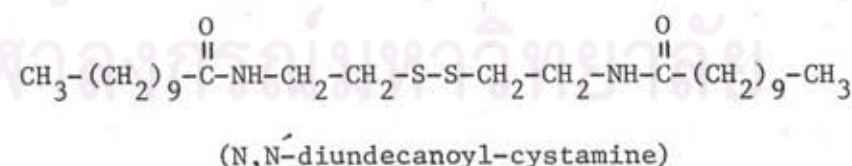
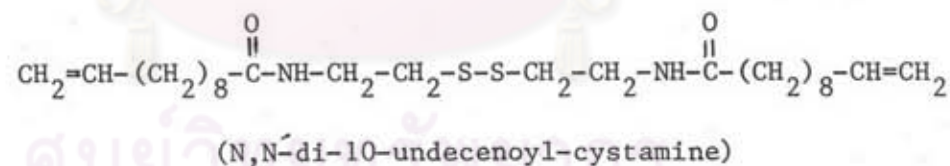
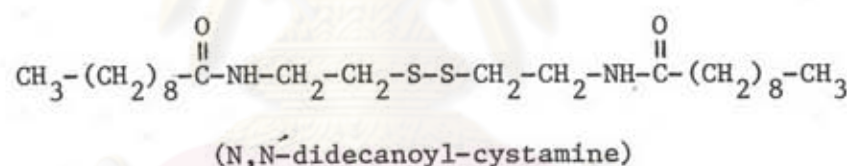
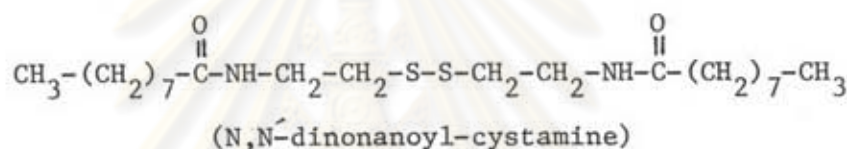
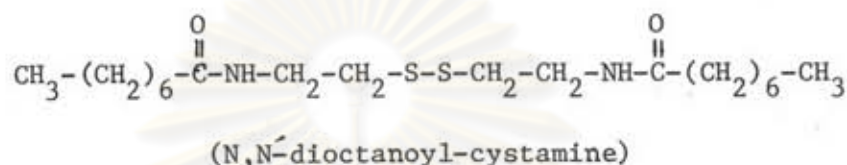
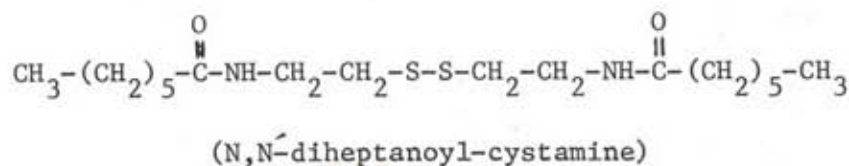
(2-Amino-2-thiazoline hydrochloride)

B-series compounds





C-series compounds



This is a novel reaction of such compounds as N,S-diacyl-2-mercaptoethylguanidine hydrochlorides. It is also a new method for the synthesis of 2-amino-2-thiazoline hydrochloride, aliphatic primary amides and N,N'-diacyl-cystamines. In addition, all of the N,N'-diacyl-cystamines (C-series compounds) obtained from this research work are new compounds.

Although no attempts were made to study the mechanisms of all the reactions in details, but some reasonable suggestions were made concerning the mechanisms of both the thermal reaction and also the mass spectral fragmentations.



ศูนย์วิจัยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

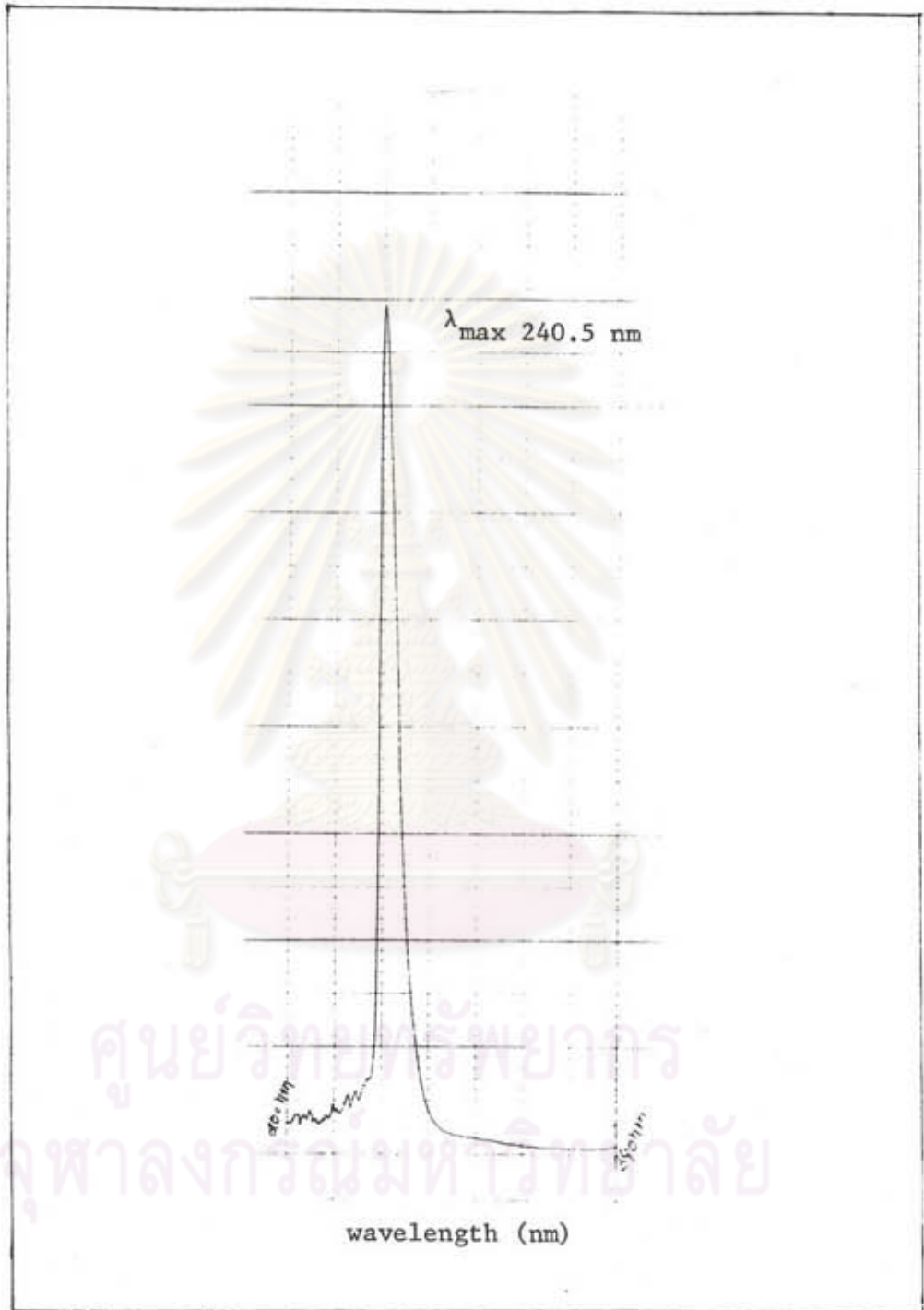


Figure 1 Ultraviolet spectrum of Compound A in CHCl_3

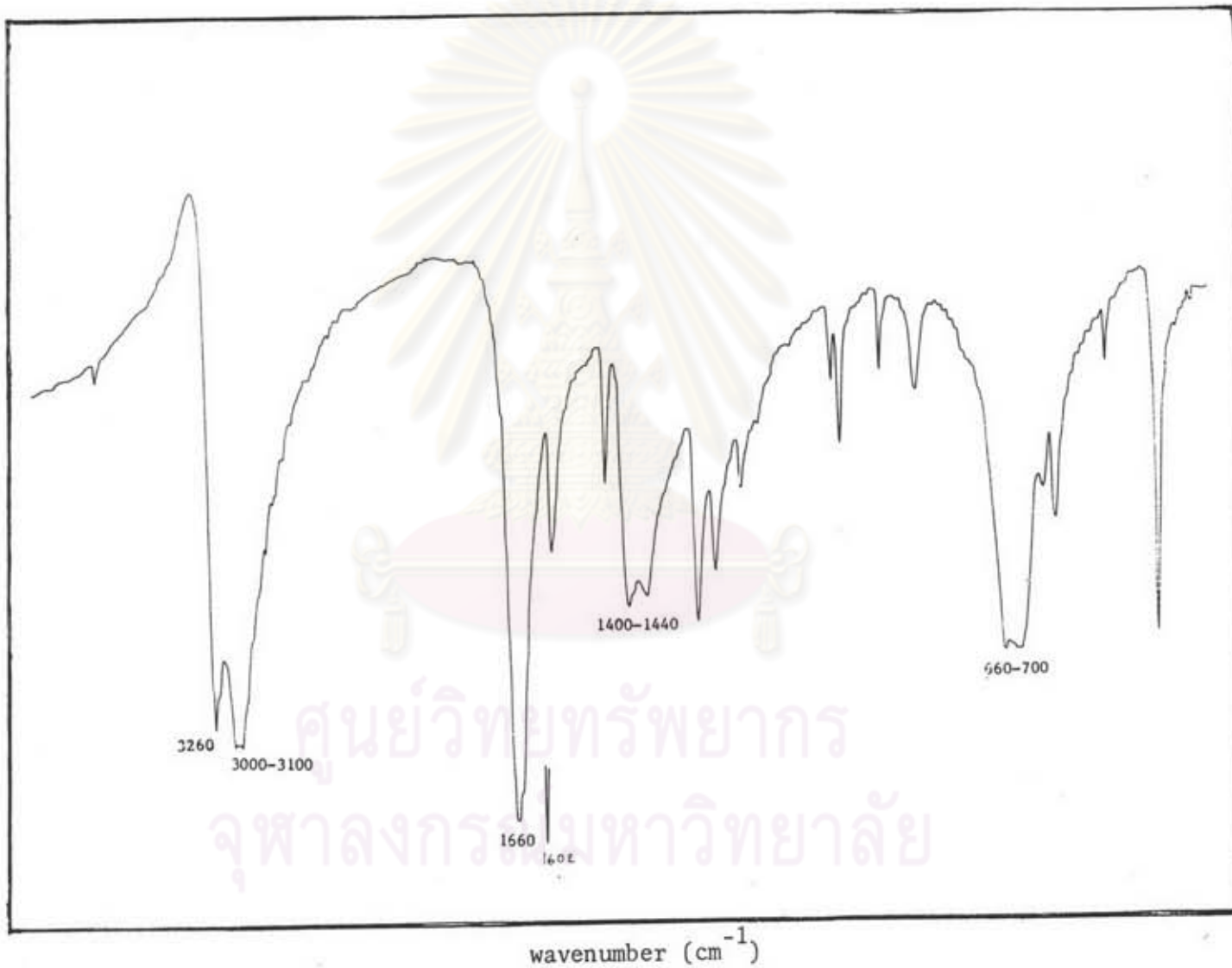


Figure 2 Infrared spectrum of compound A in KBr disc.

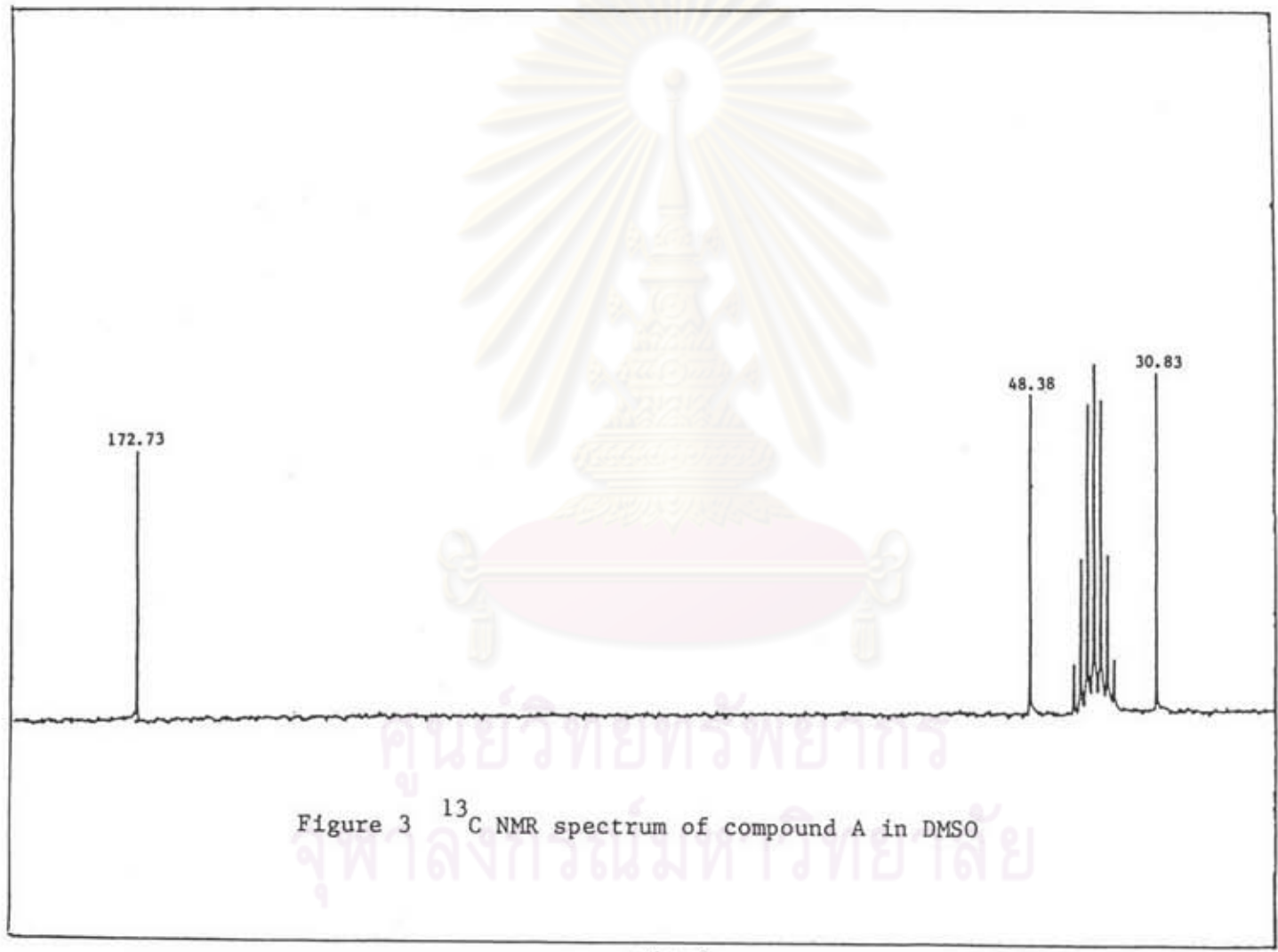
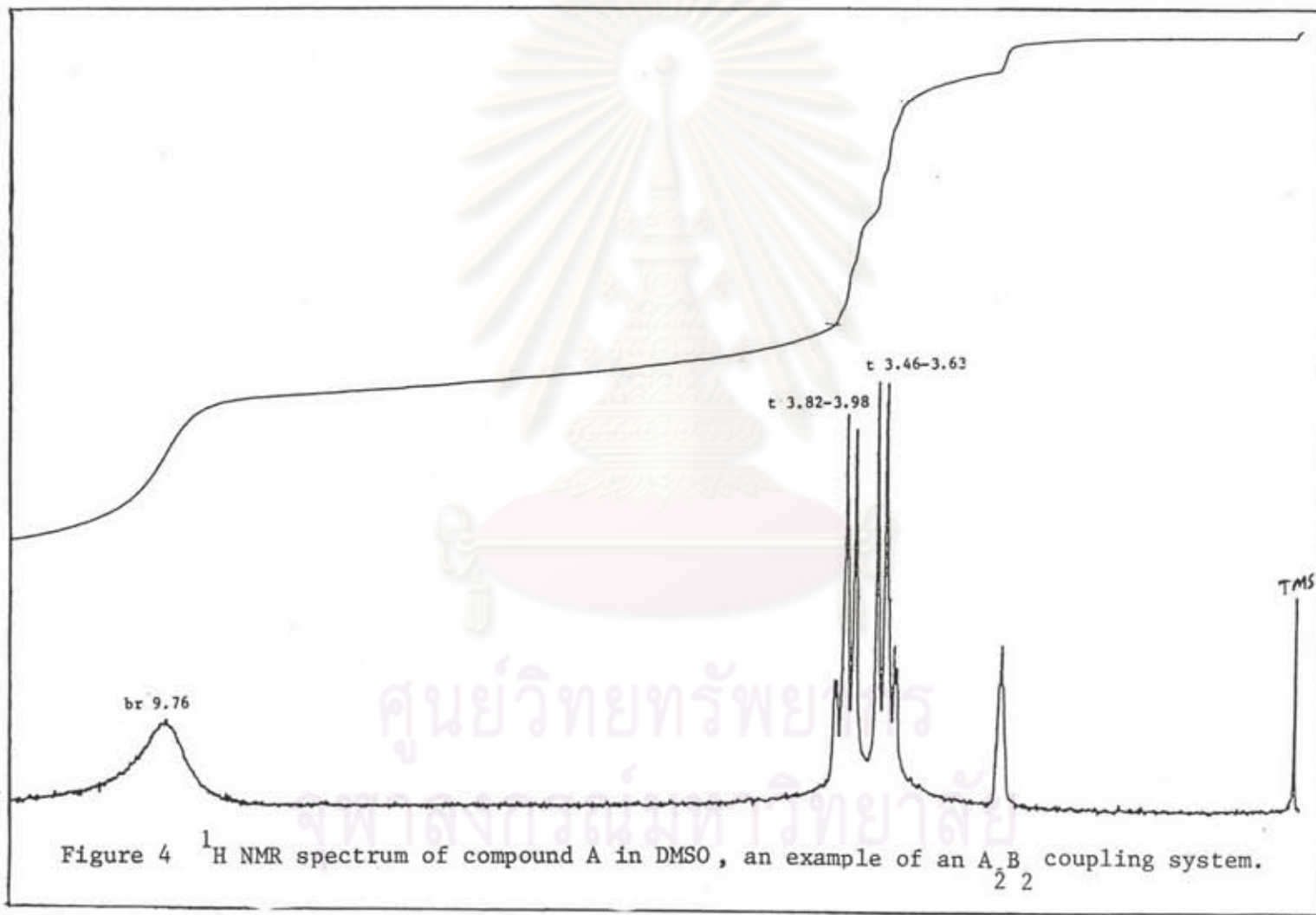


Figure 3 ^{13}C NMR spectrum of compound A in DMSO

δ (ppm)



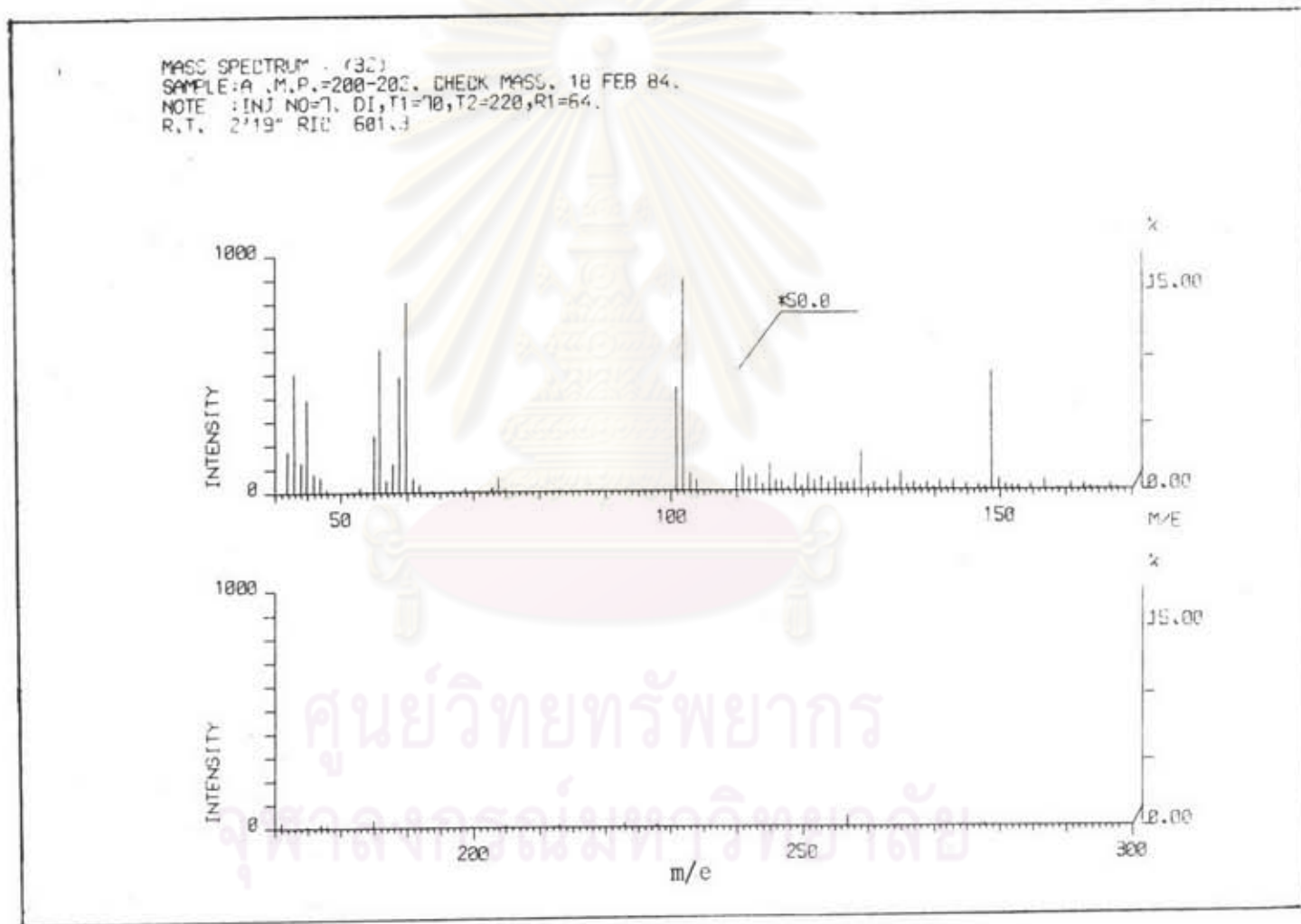


Figure 5 Mass spectrum of compound A.

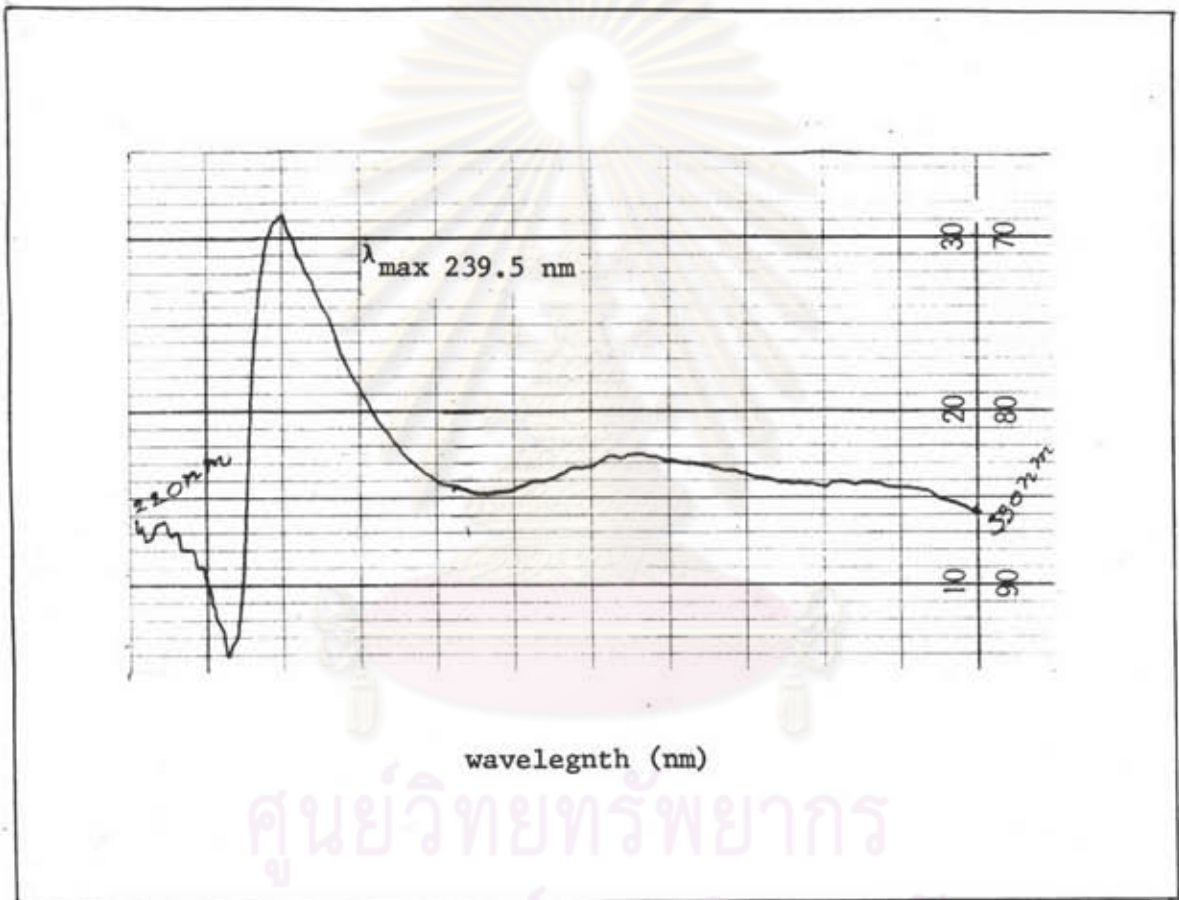


Figure 6 Ultraviolet spectrum of compound B-7 in CHCl_3

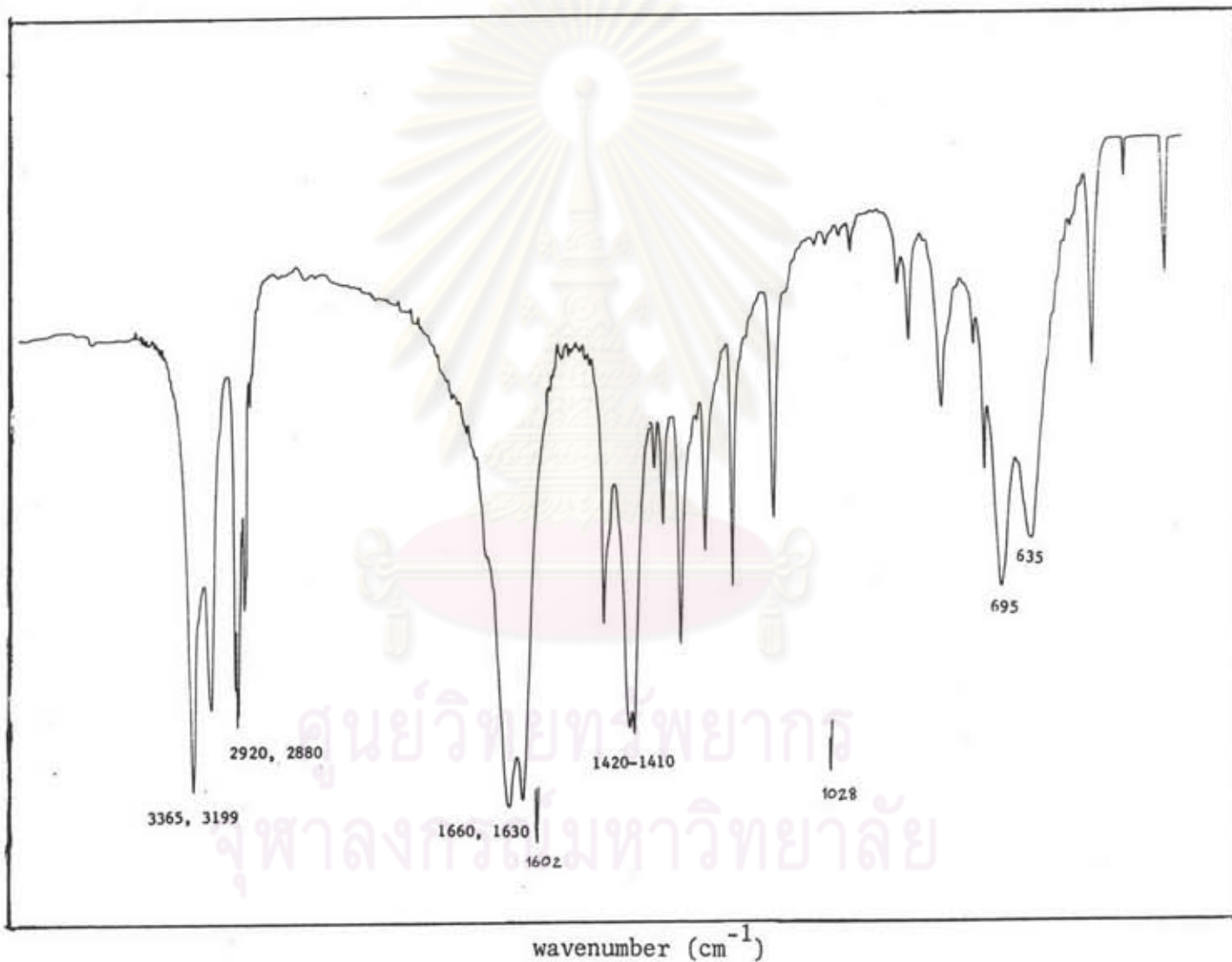


Figure 7 Infrared spectrum of compound B-7 in KBr disc.

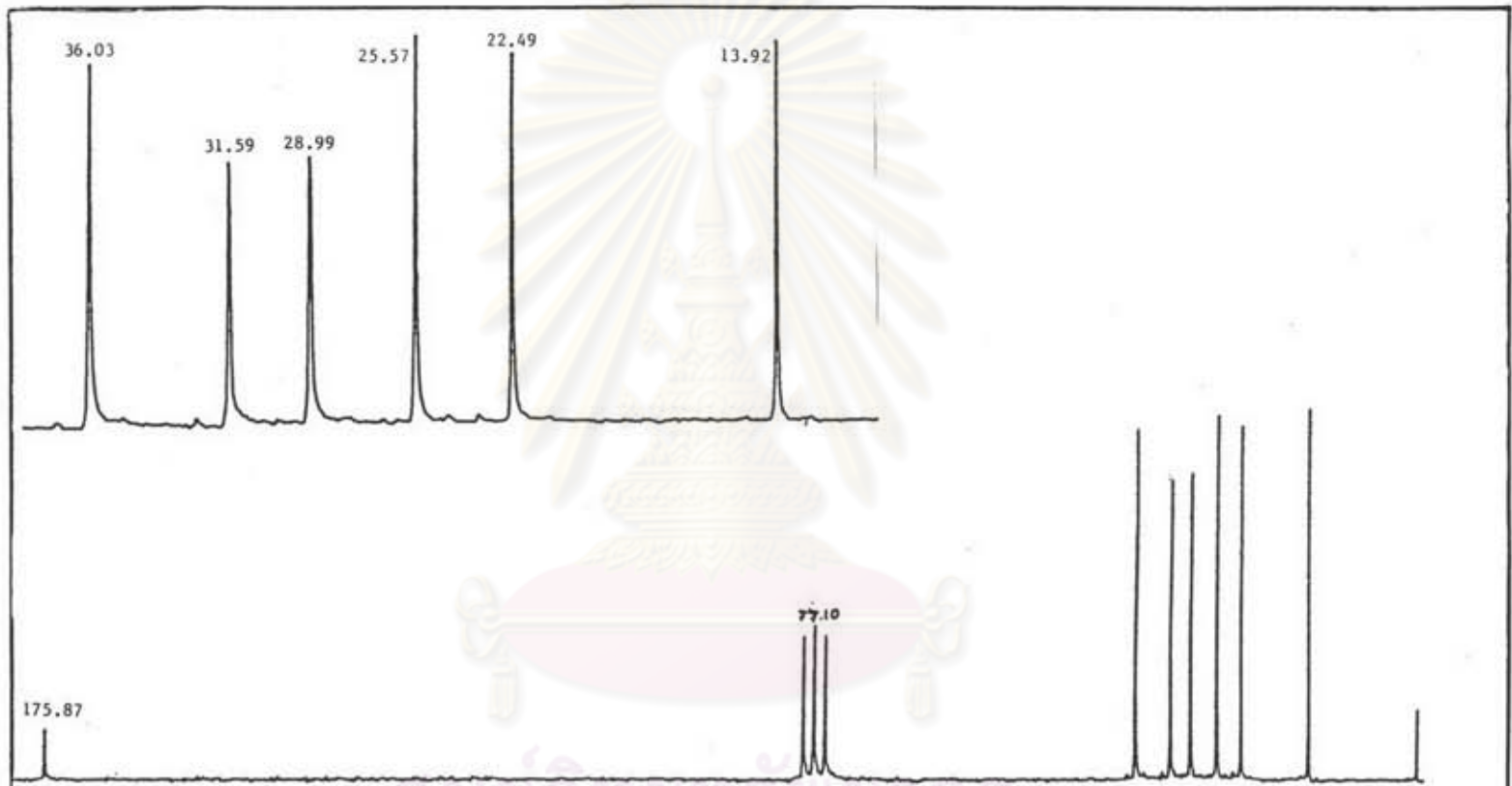
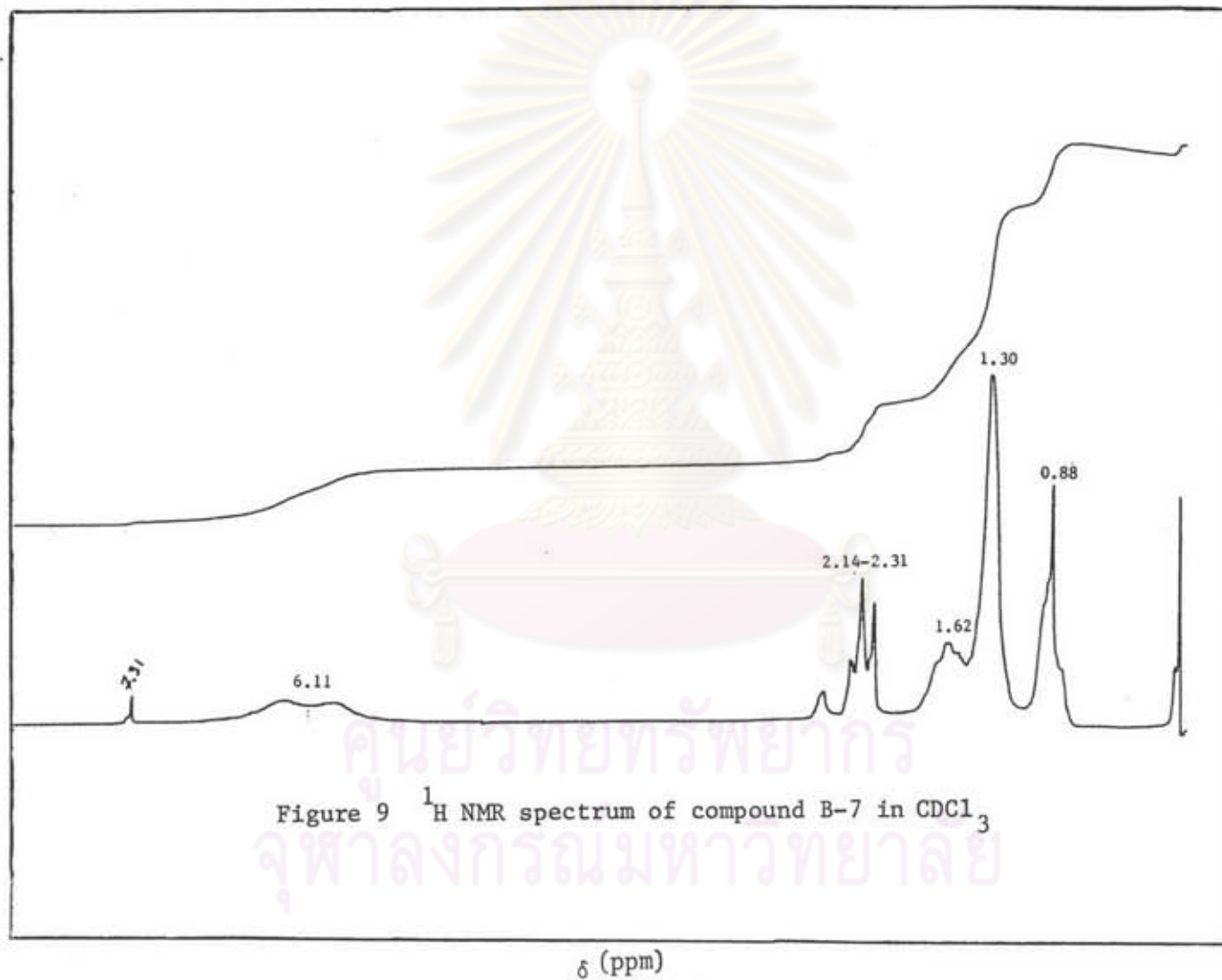


Figure 8 ^{13}C NMR spectrum of compound B-7 in CDCl_3

δ (ppm)



MASS SPECTRUM : (2)
SAMPLE: B7, M.P. = 91-93, CHECK MASS, 18 FEB 84 (SAT)
NOTE : INJ NO=6, DI, T1=RT, T2=105, R1=16,
R.T. 0'04' RIL 552.9

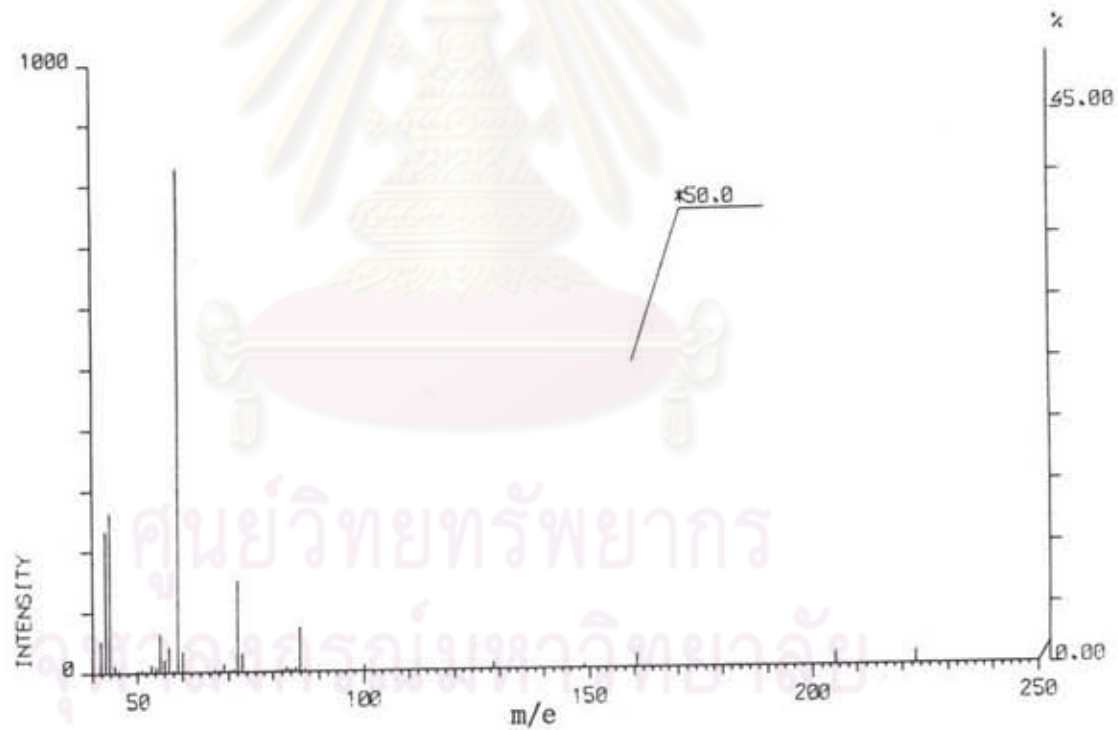


Figure 10 Mass spectrum of compound B-7

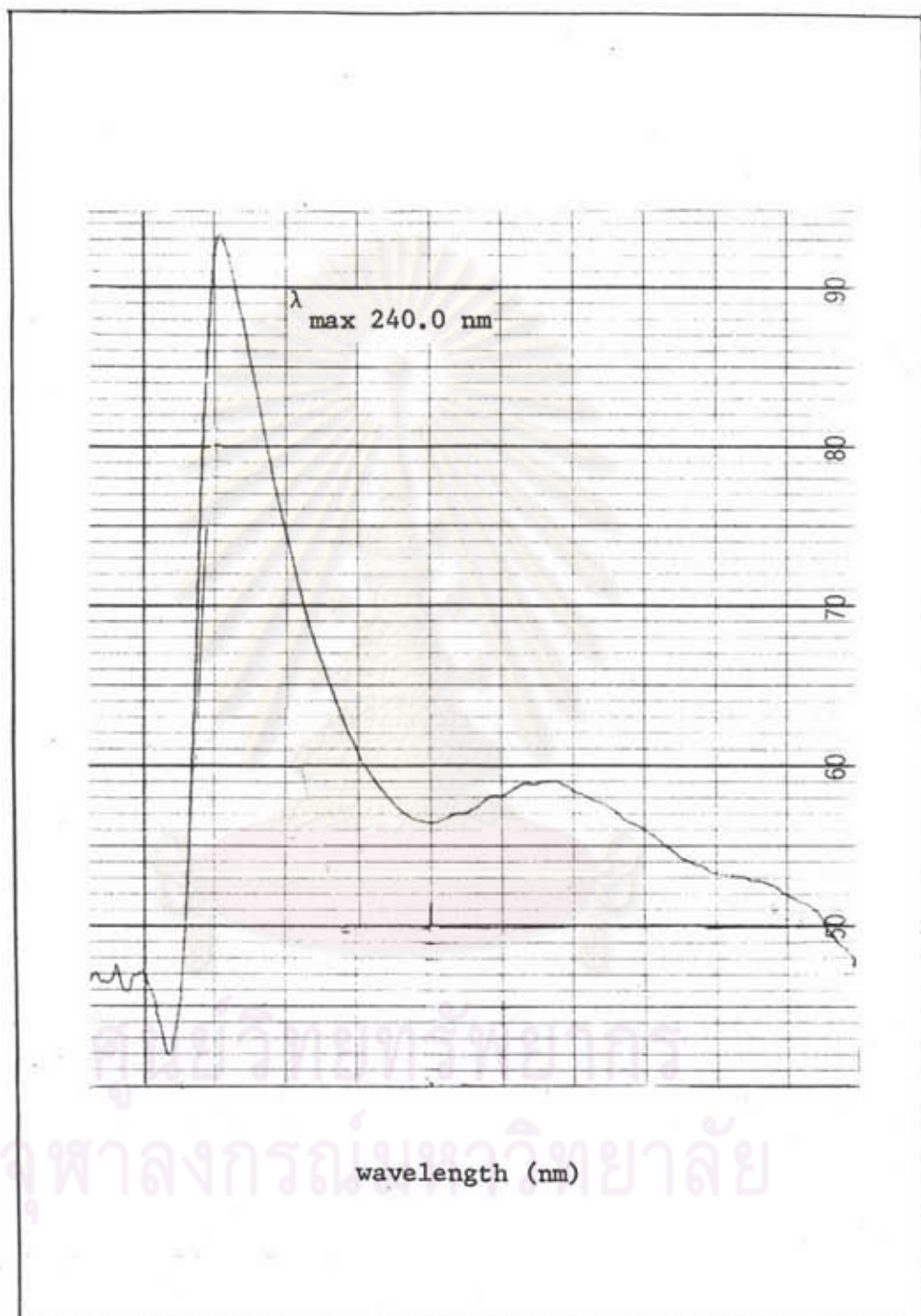


Figure 11 Ultraviolet spectrum of compound B-8 in CHCl_3

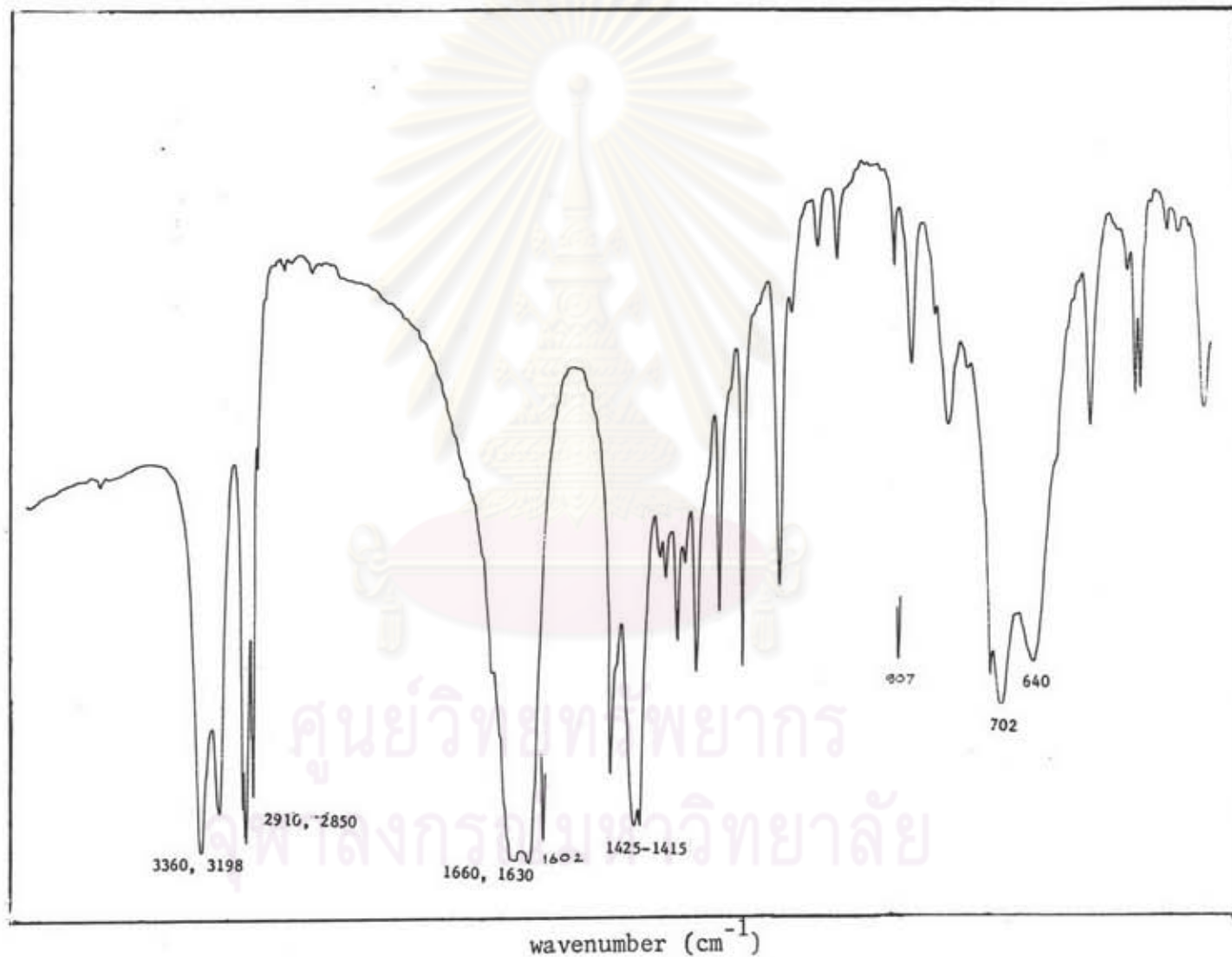


Figure 12 Infrared spectrum of compound B-8 in KBr disc.

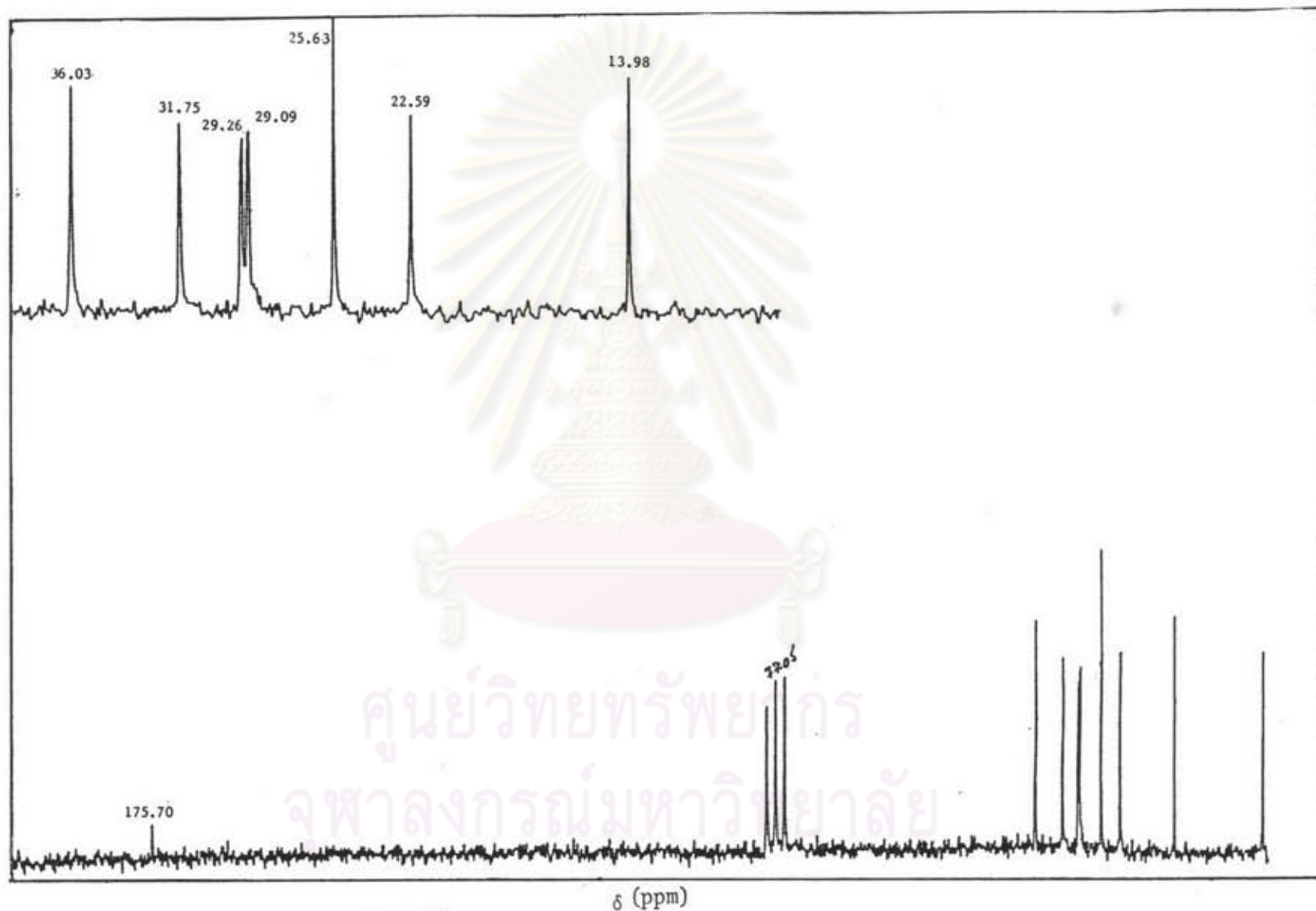
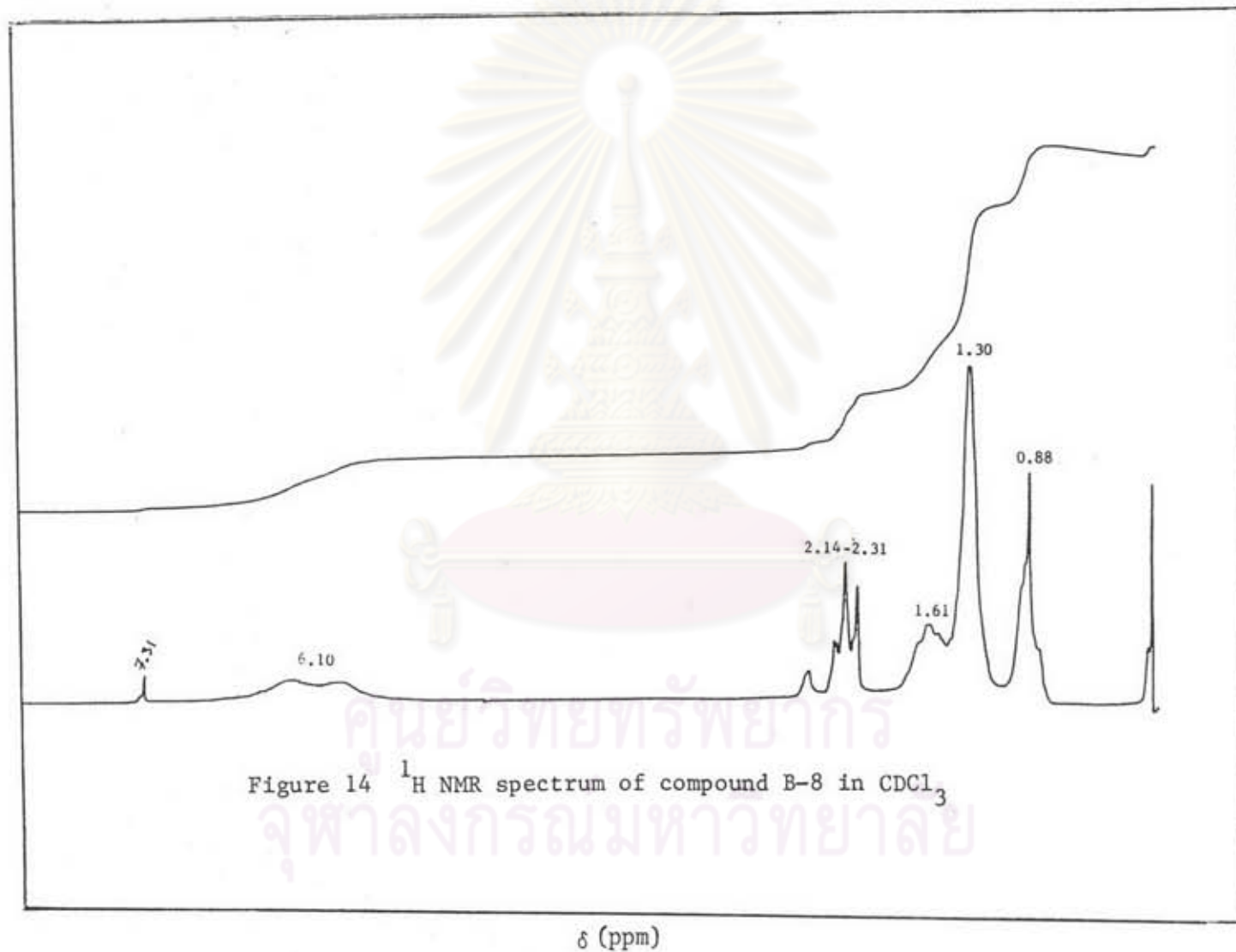


Figure 13 ^{13}C NMR spectrum of compound B-8 in CDCl_3



MASS SPECTRUM : (9 TO 10)
SAMPLE: B B.M.P.=100-102, CHECK MASS, 2 FEB 84,
NOTE : INJ NO=112, DI, T1=RT, T2=120, R1=32.

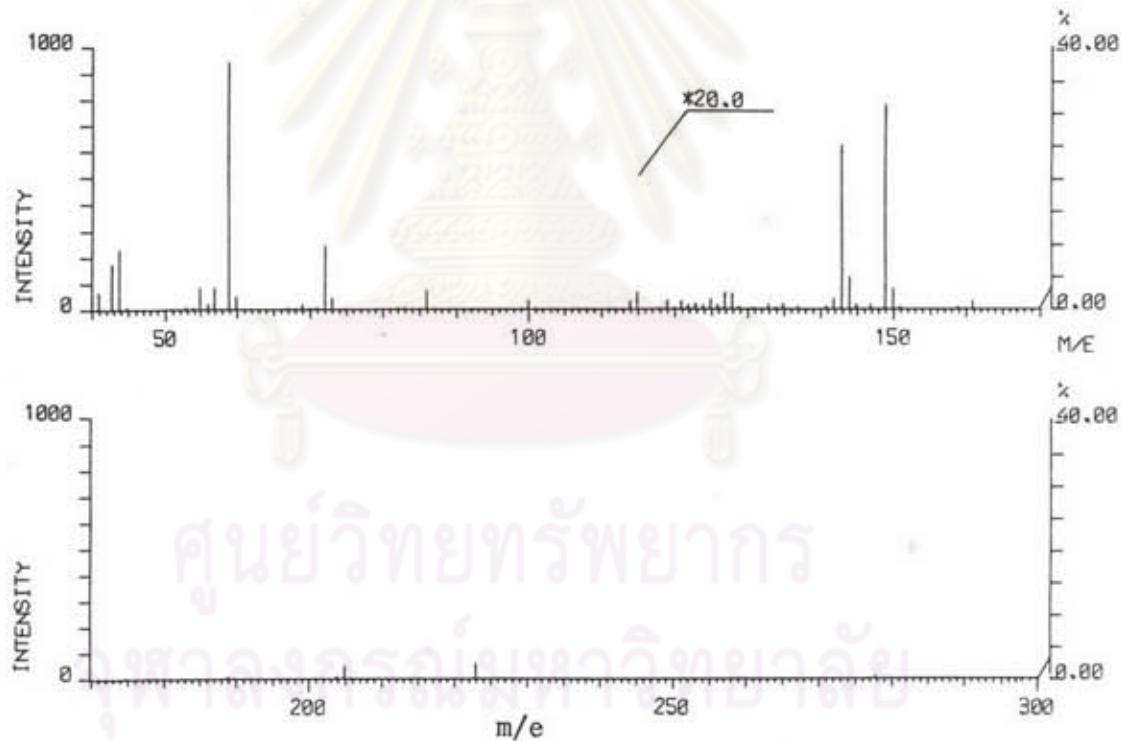


Figure 15 Mass spectrum of compound B-8



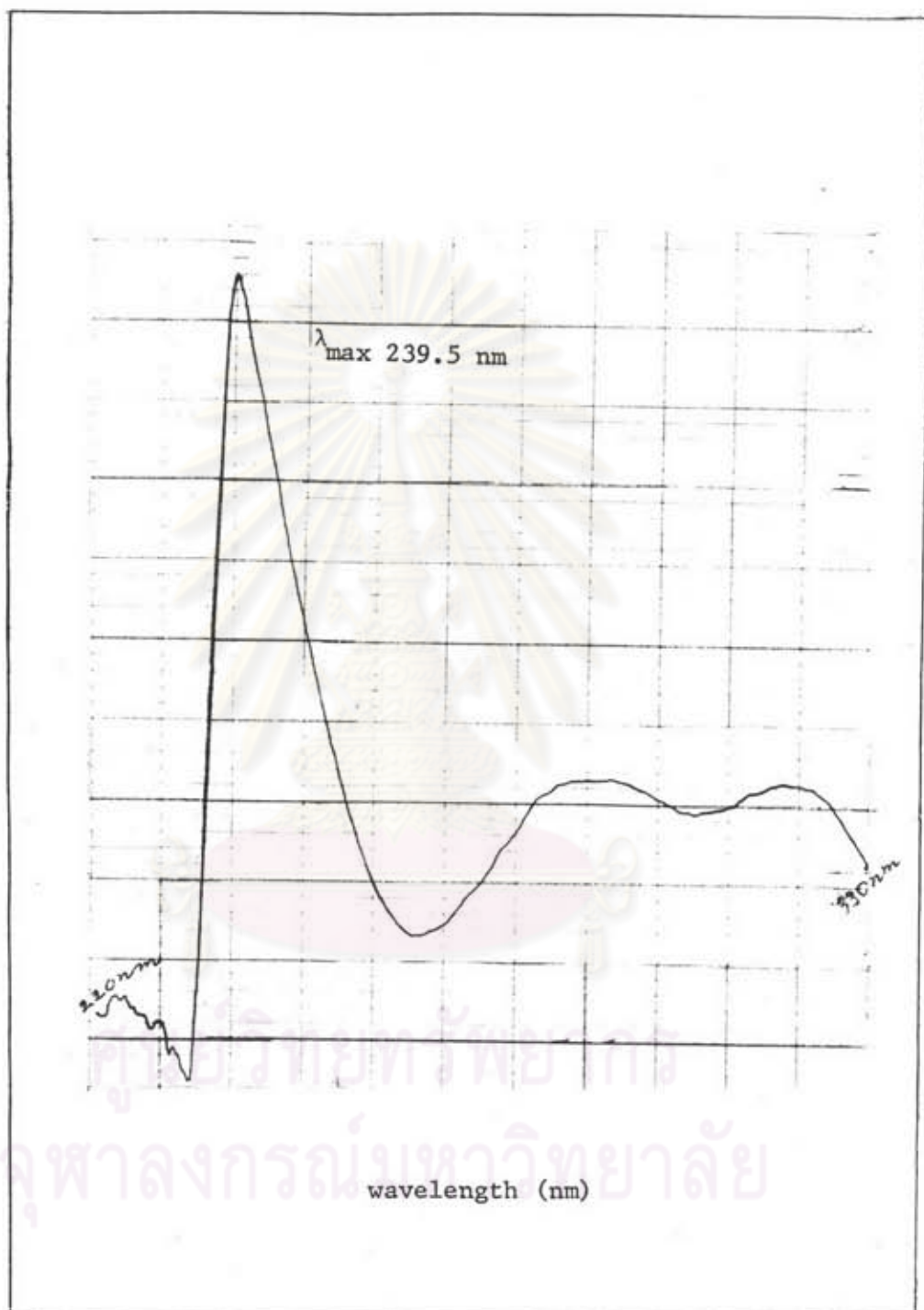


Figure 16 Ultraviolet spectrum of compound B-9 in CHCl_3

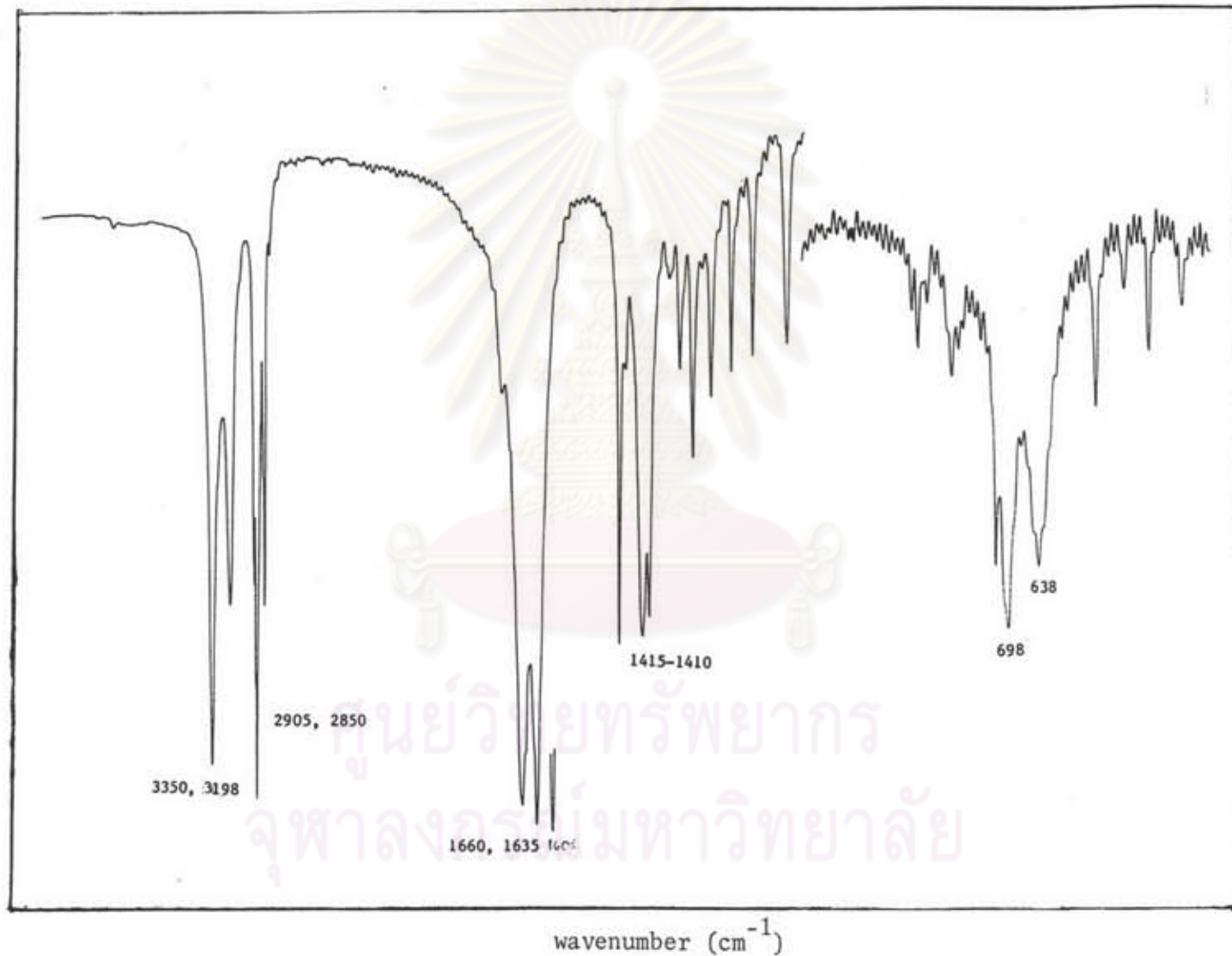
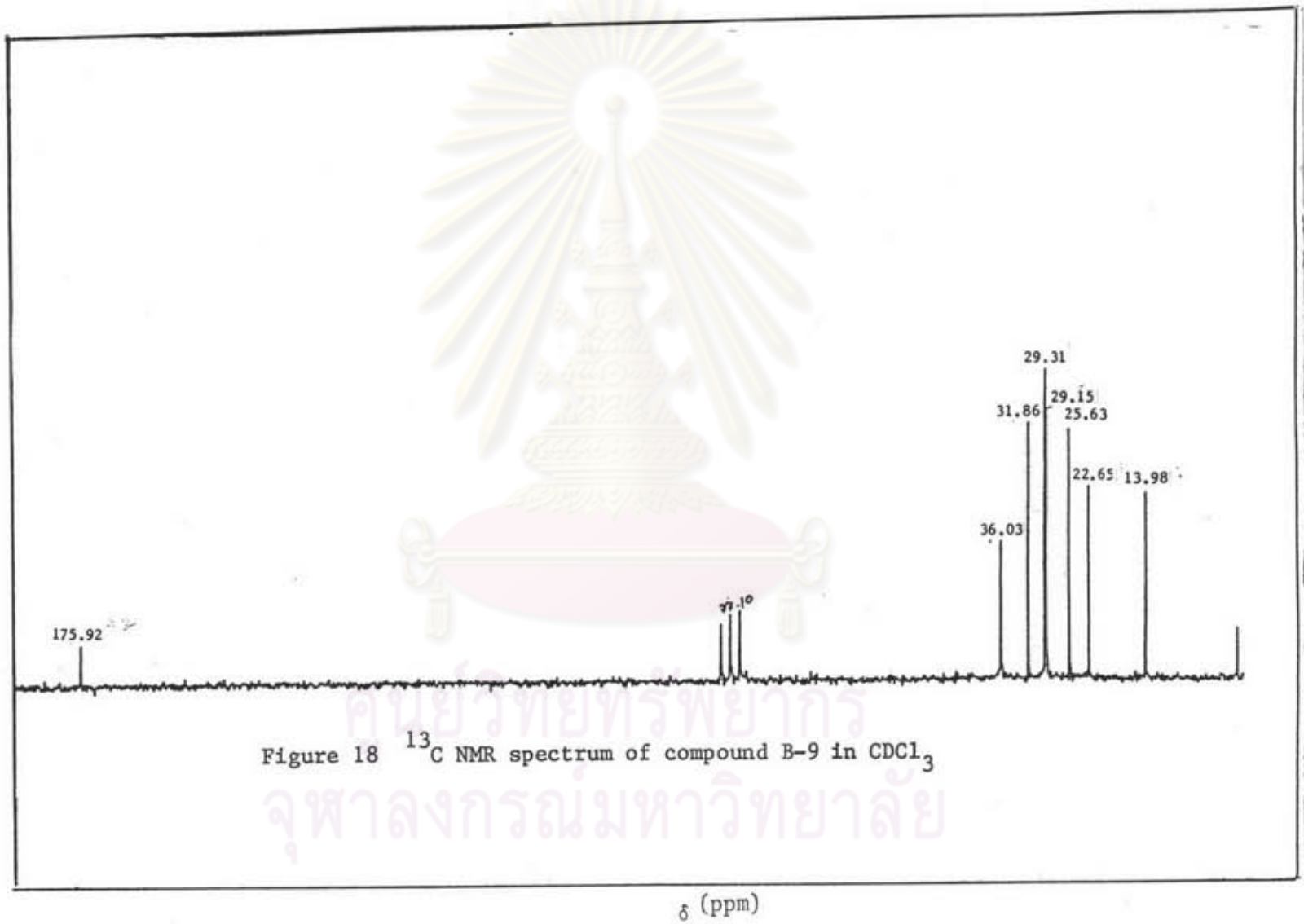


Figure 17 Infrared spectrum of compound B-9 in KBr disc.



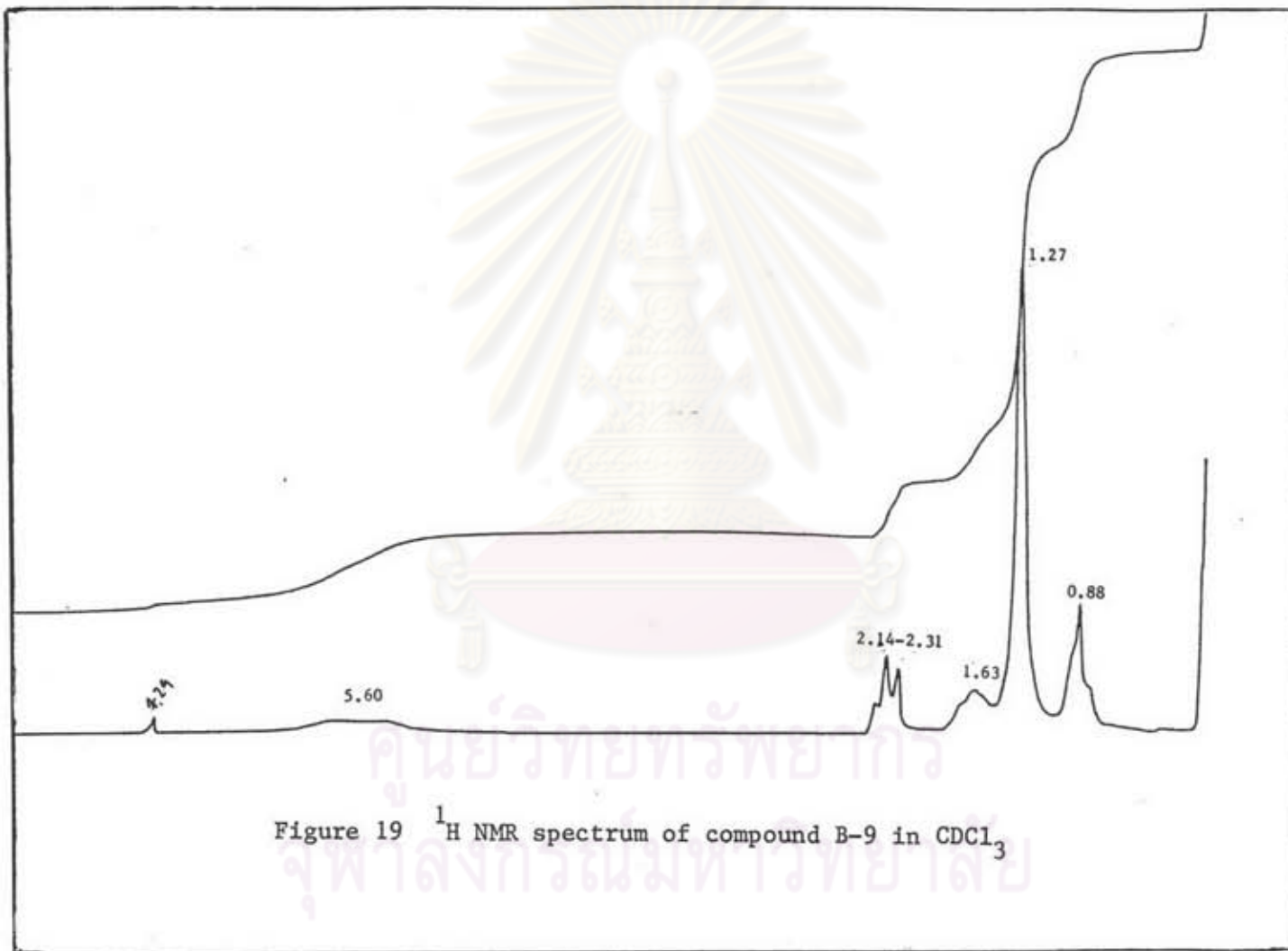


Figure 19 ^1H NMR spectrum of compound B-9 in CDCl_3

δ (ppm)

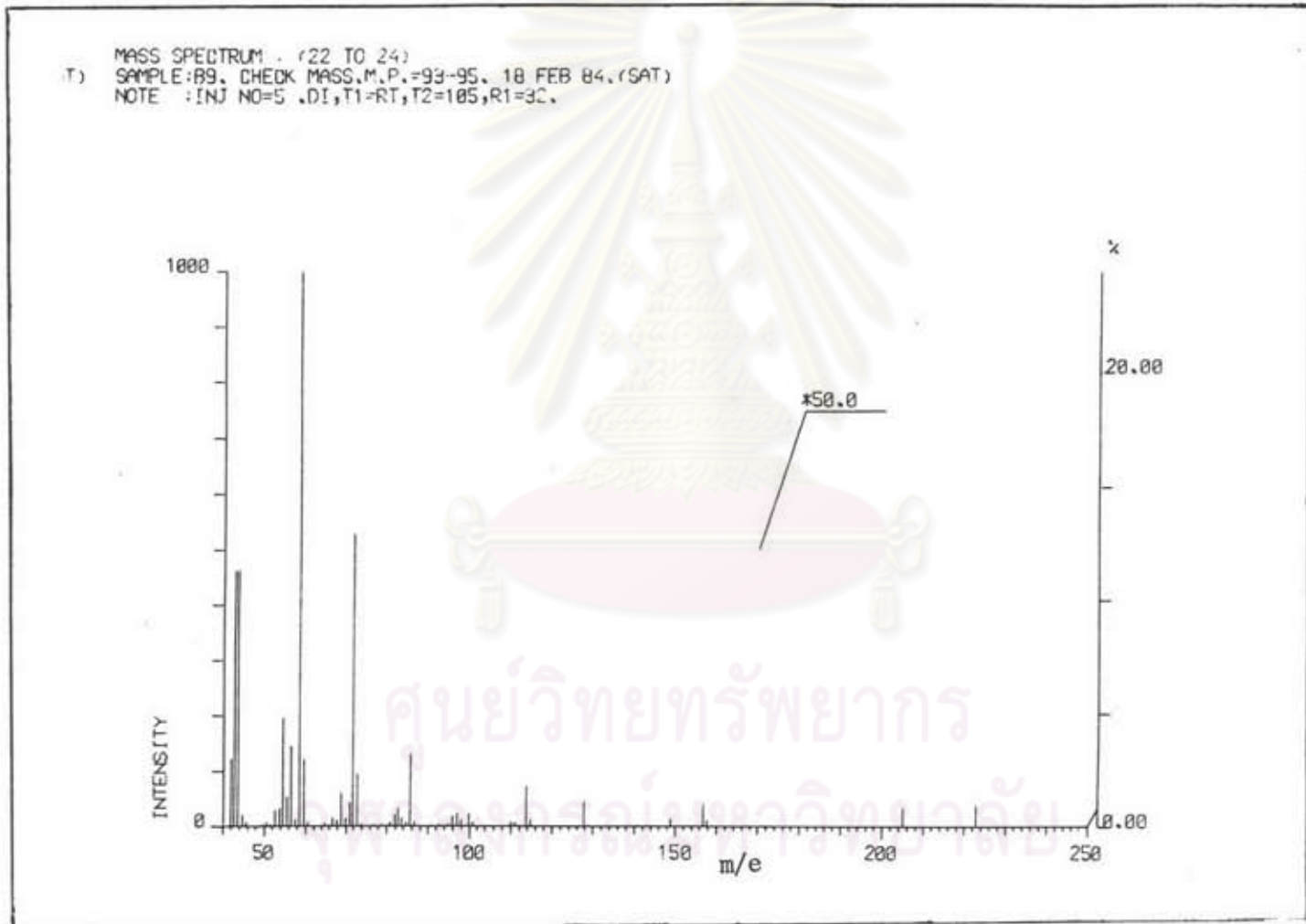
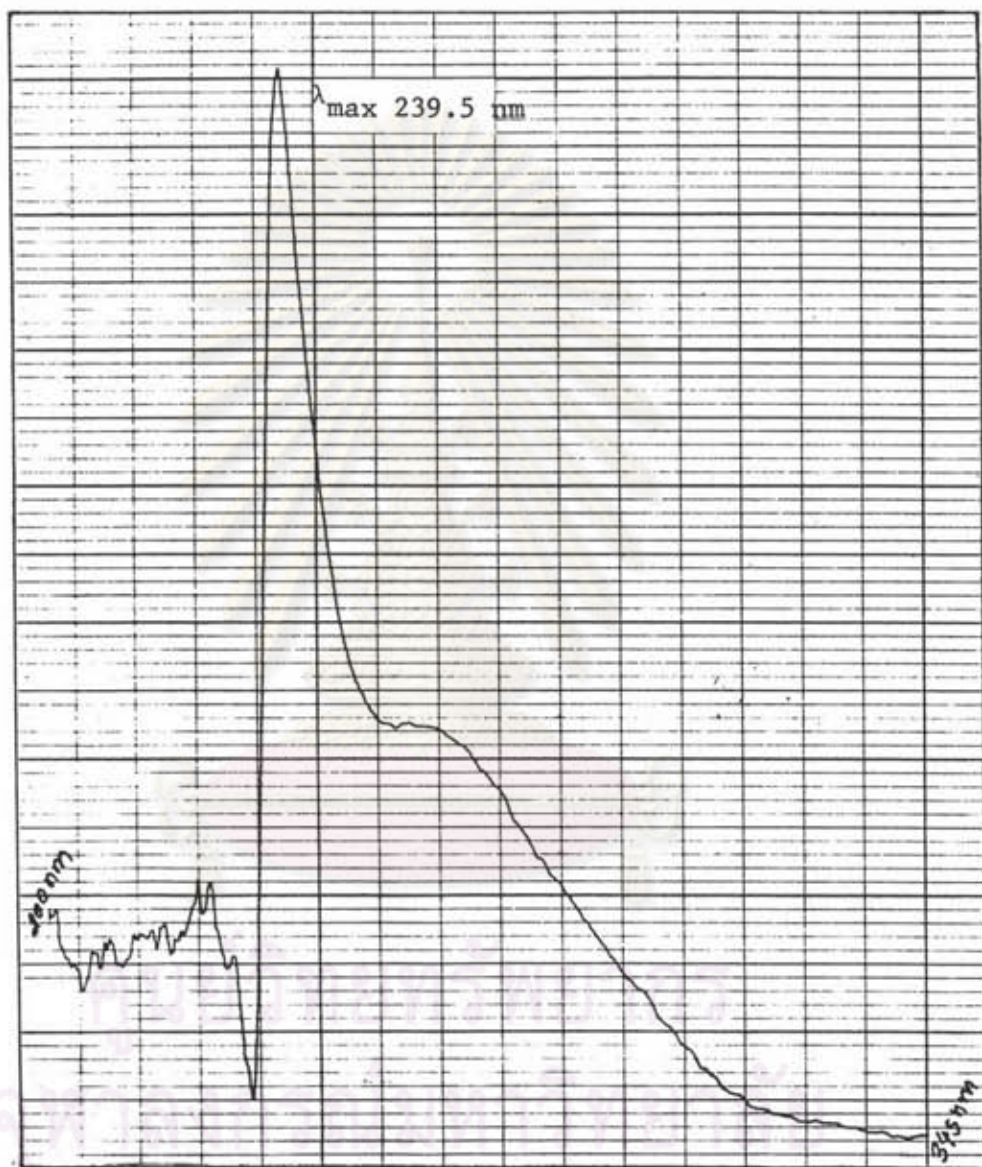


Figure 20 Mass spectrum of compound B-9



wavelength (nm)

Figure 21 Ultraviolet spectrum of compound B-10 in CHCl_3 .

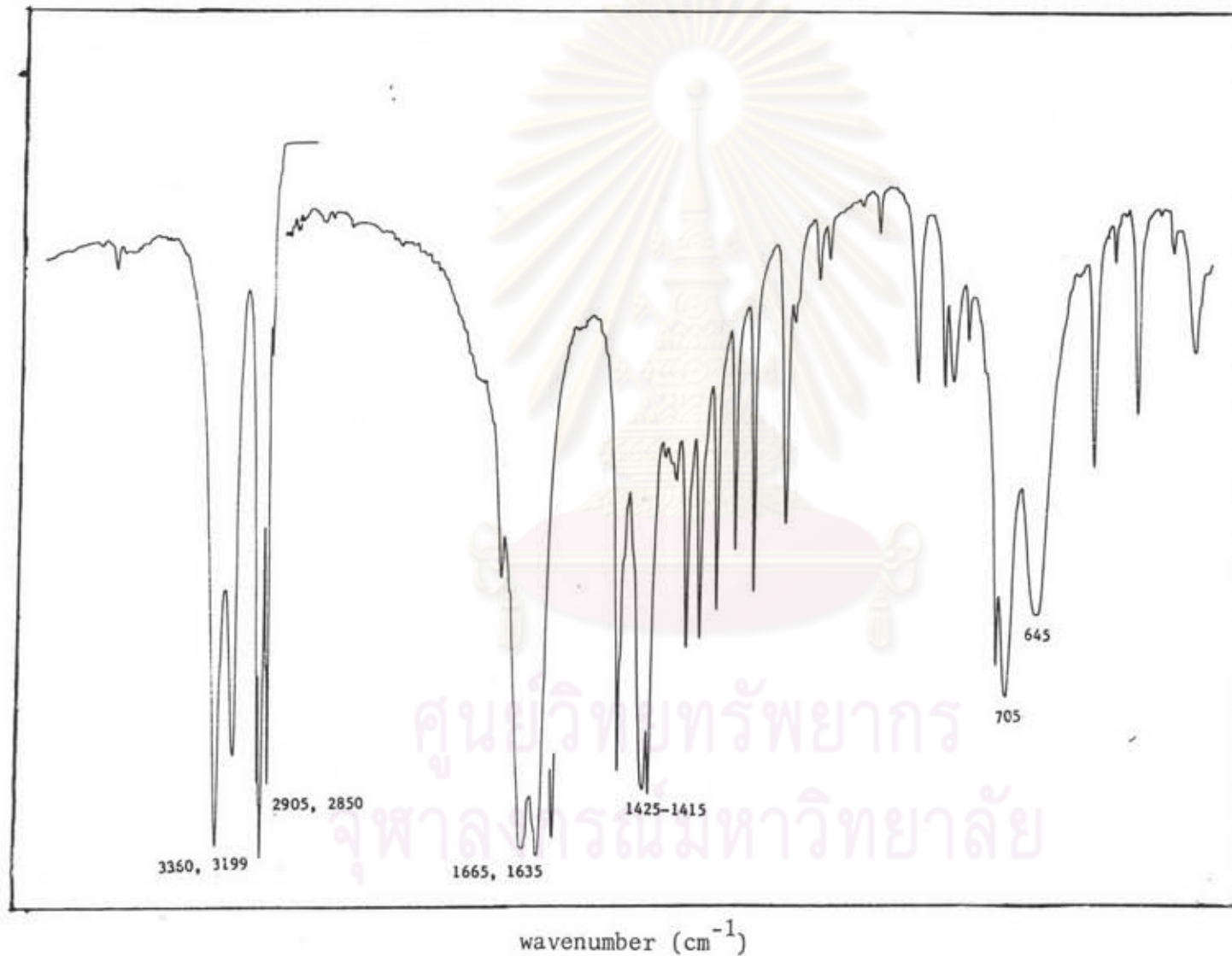
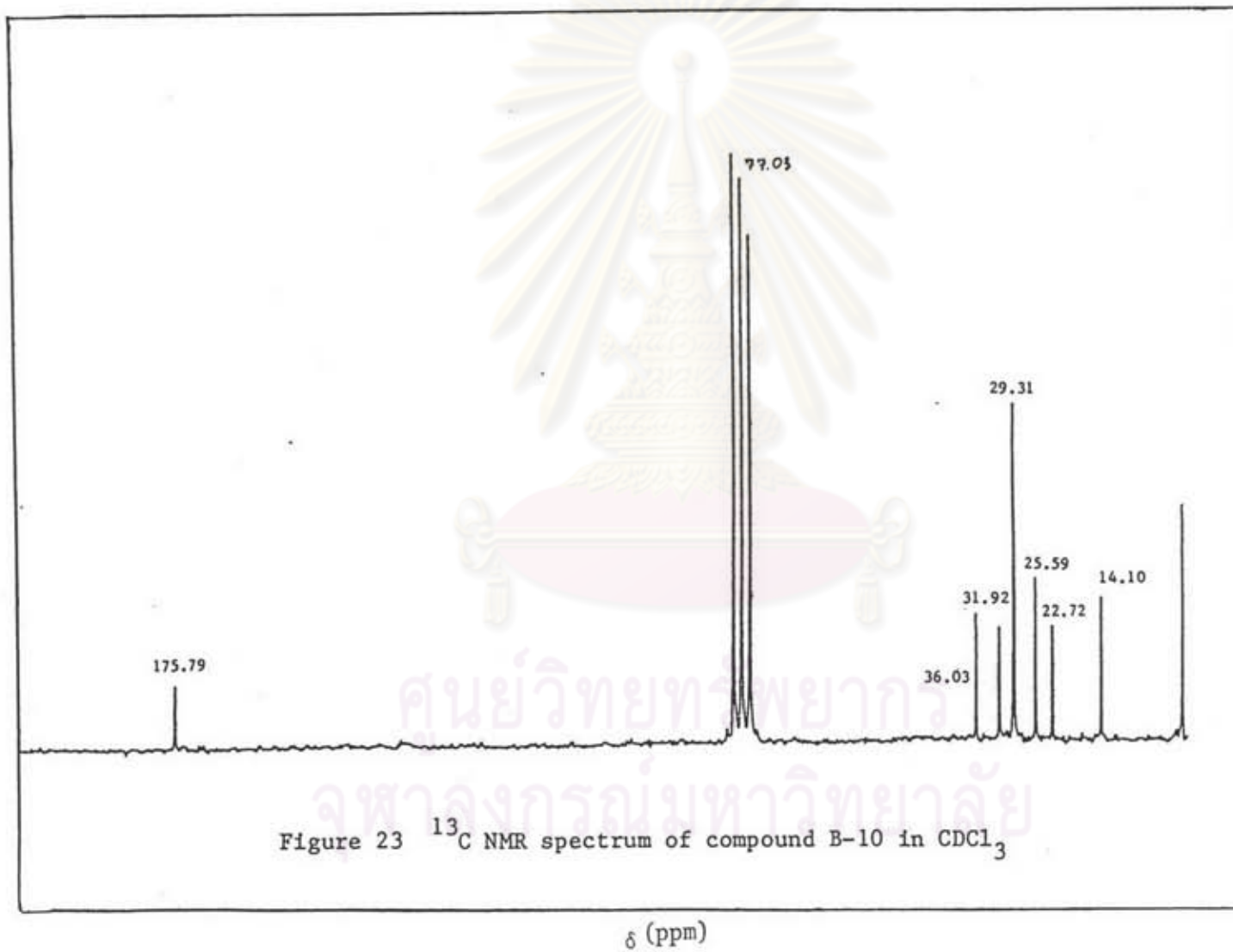


Figure 22 Infrared spectrum of compound B-10 in KBr disc.



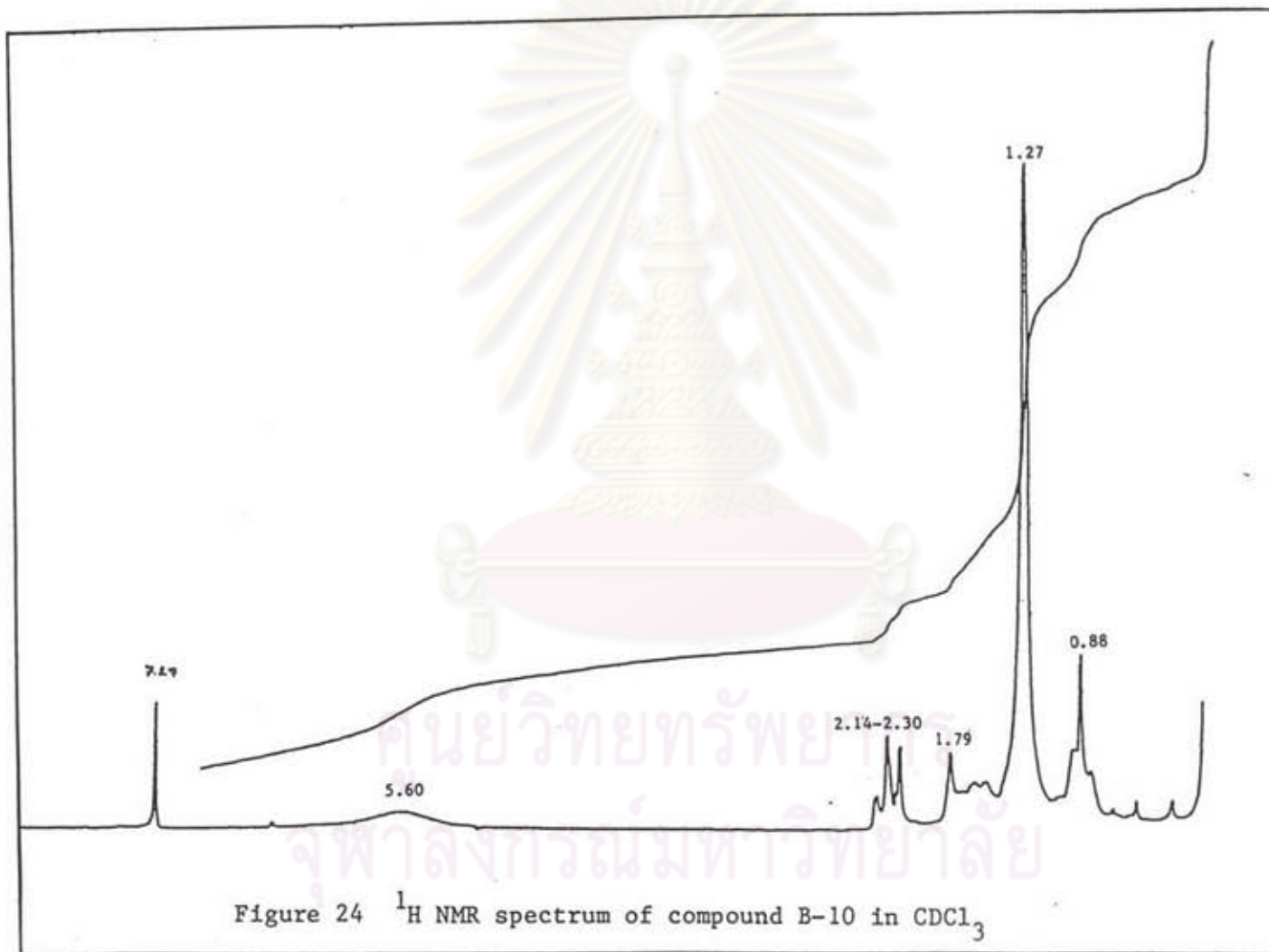


Figure 24 ^1H NMR spectrum of compound B-10 in CDCl_3

δ (ppm)

MASS SPECTRUM : (21)
SAMPLE: B 10. M.P.=93-95. CHECK MASS. 2 FEB 84.
NOTE : INJ NO=110 .DI,T1=RT,T2=120,R1=32.
R.T. 1'30" RIC 201.2

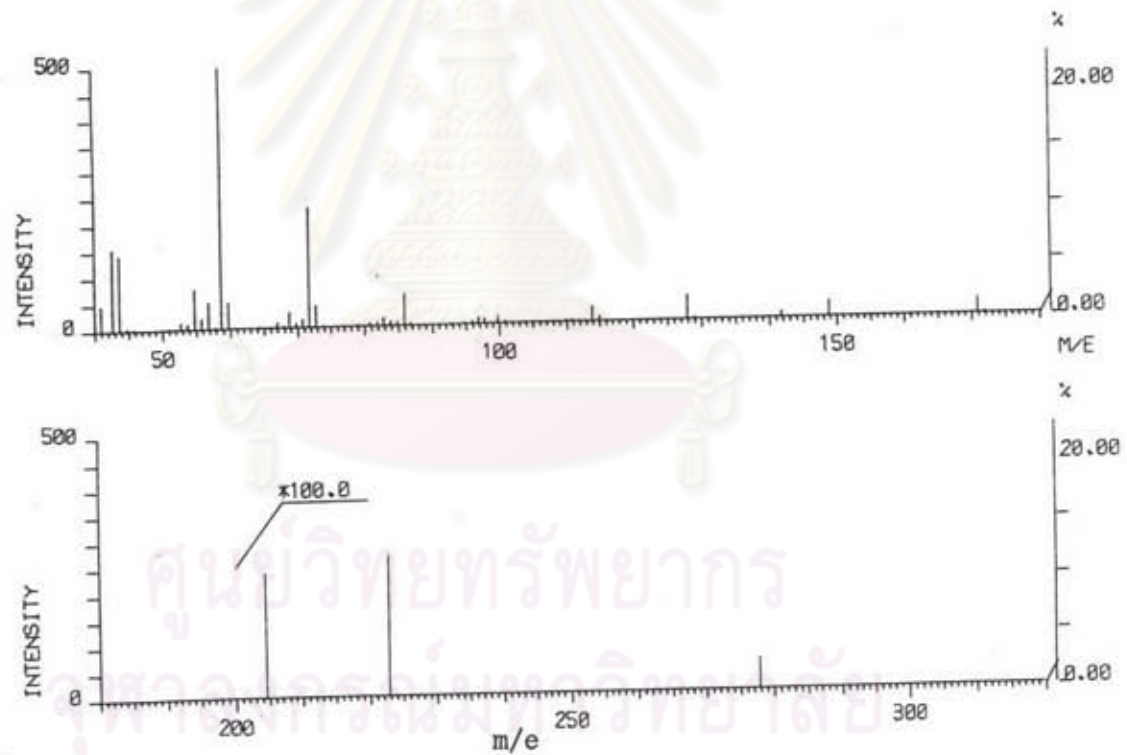


Figure 25 Mass spectrum of compound B-10

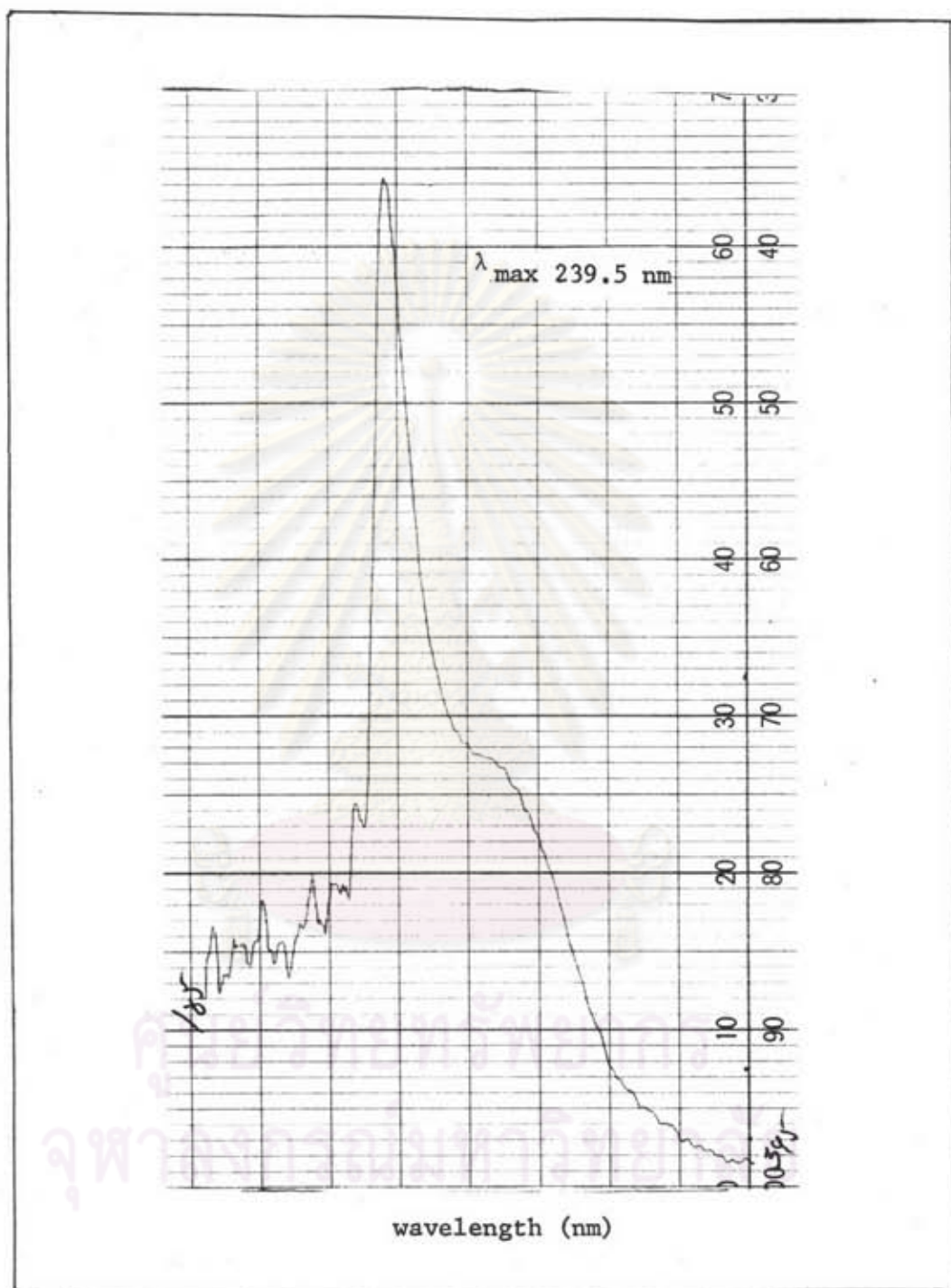


Figure 26 Ultraviolet spectrum of compound B-11 in CHCl_3

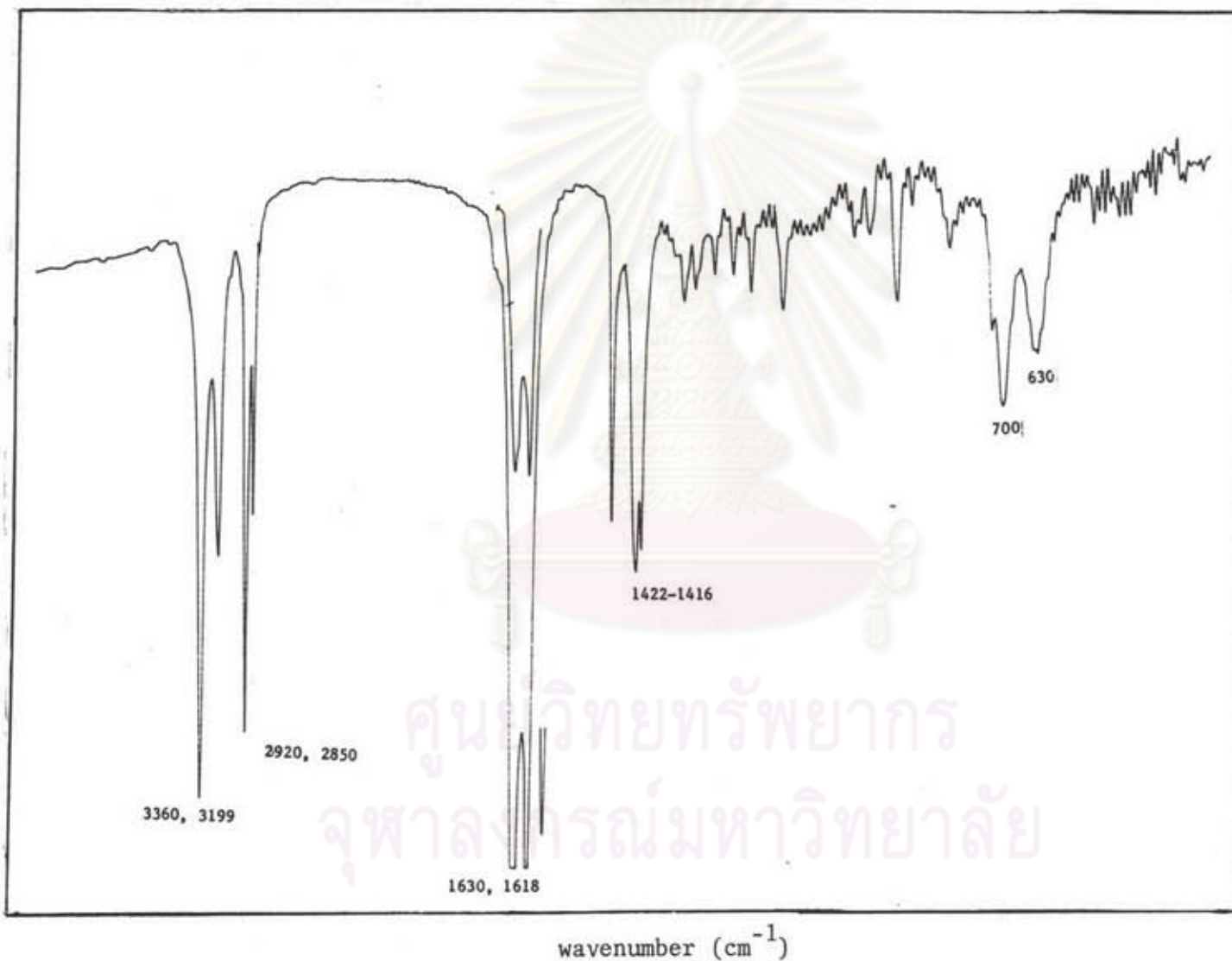


Figure 27 Infrared spectrum of compound B-11 in KBr disc.

MASS SPECTRUM : (13 TO 14)
SAMPLE: B1C, M.P. = 81-83, 30 JAN 84.
NOTE : [N] NO=91, [I], T1=RT, T2=150, R1=30.

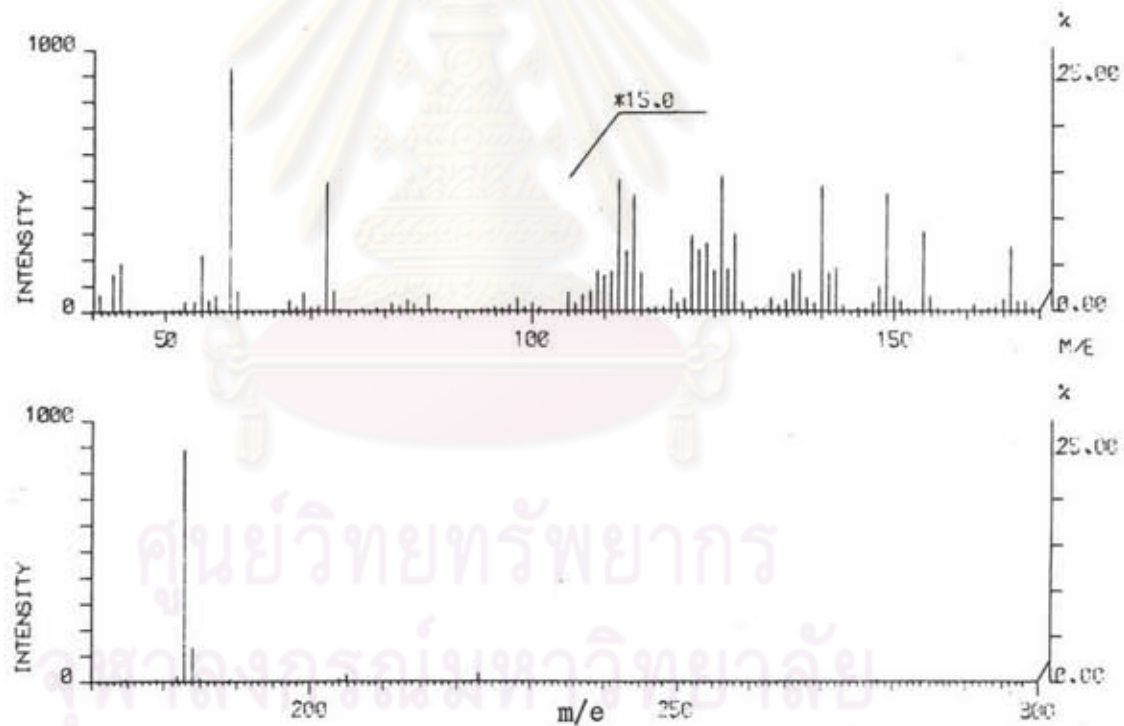


Figure 28 Mass spectrum of compound B-11

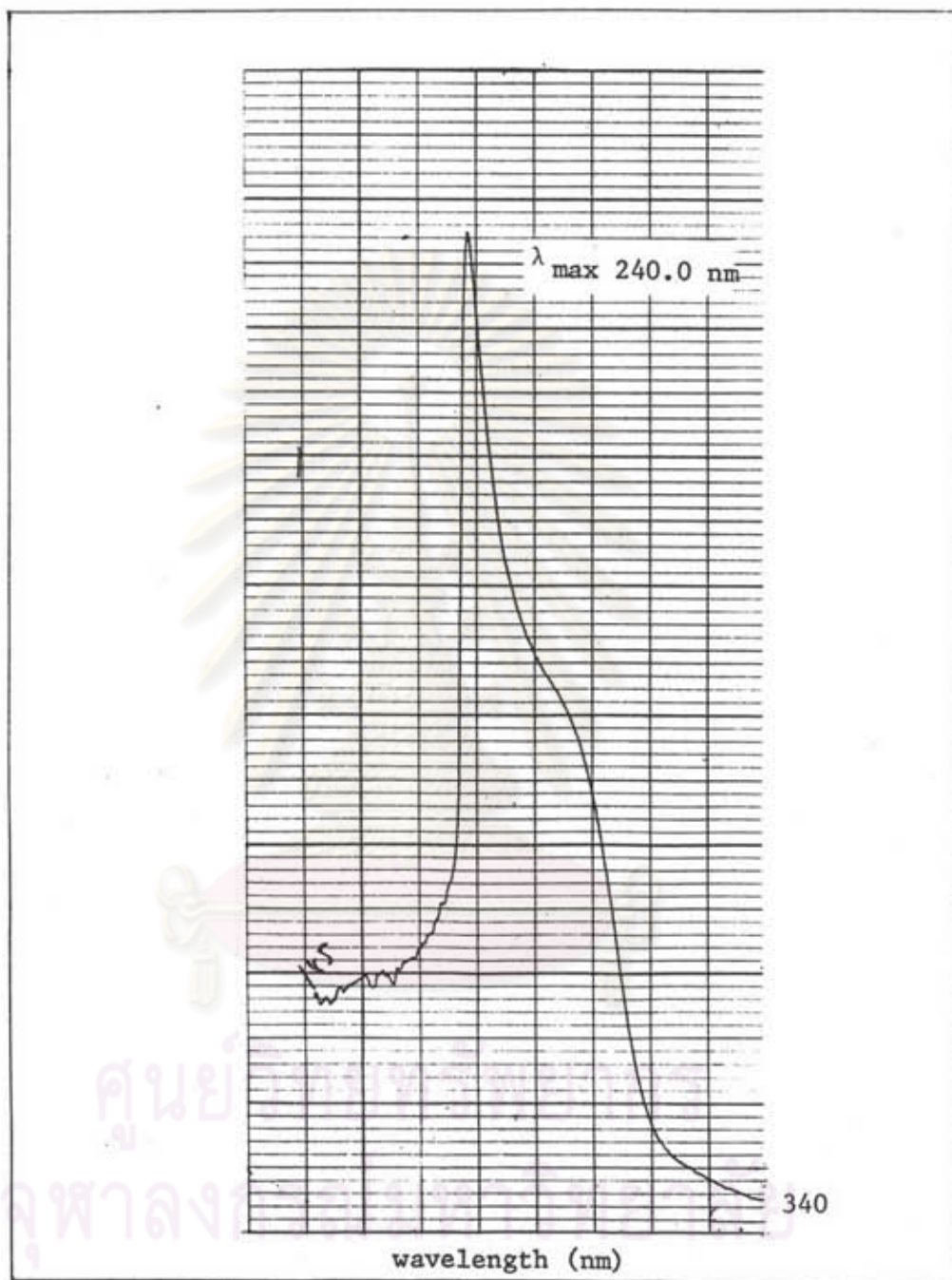


Figure 29 Ultraviolet spectrum of compound B-12 in CHCl_3

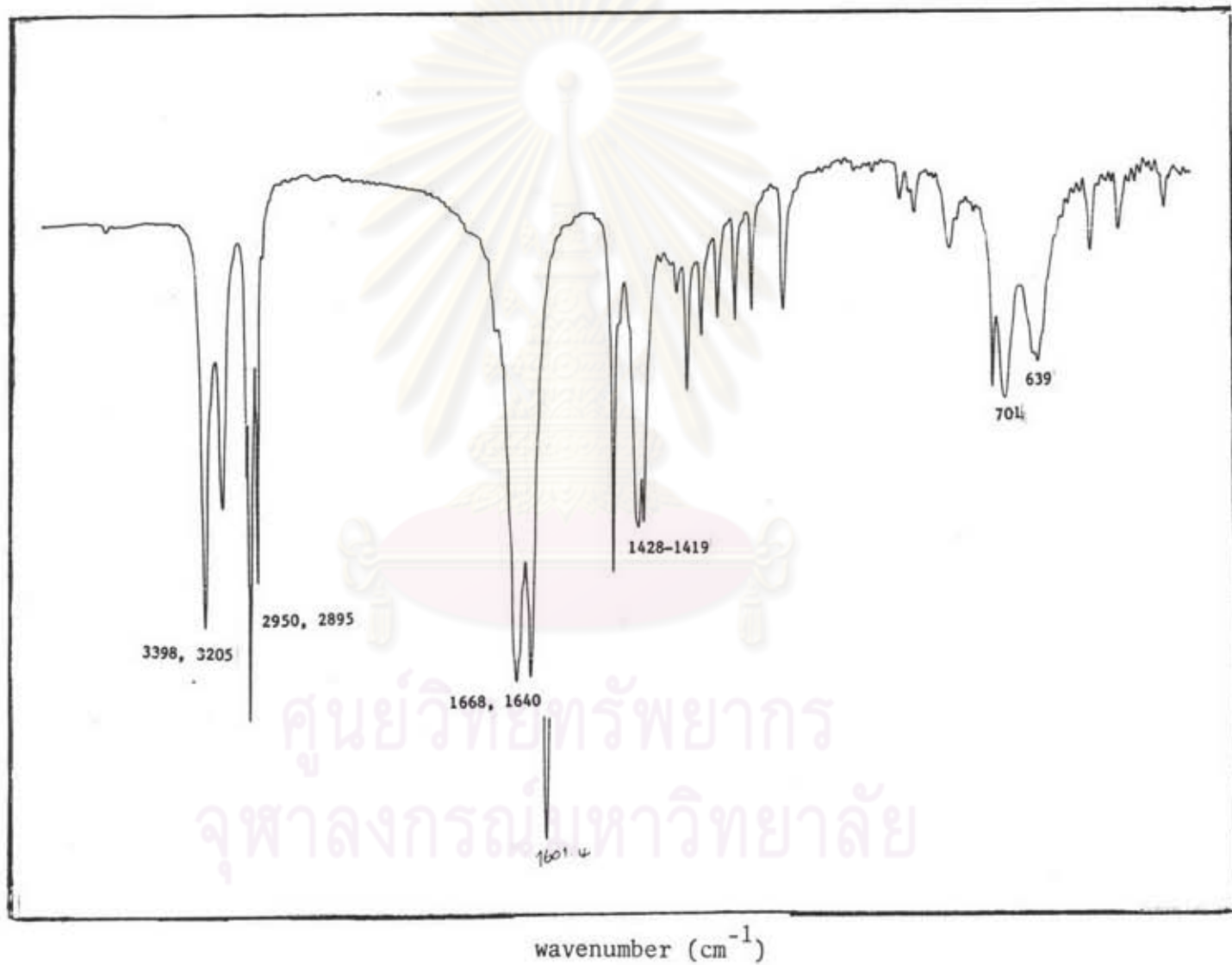


Figure 30 Infrared spectrum of compound B-12 in KBr disc

MASS SPECTRUM : (10)
SAMPLE: B11, M.P. = 91-93, 30 JAN 84.
NOTE : INJ NO=93.D1, T1=ERT, T2=150, R1=32.
R.T. 0'40" RIC 100.0'

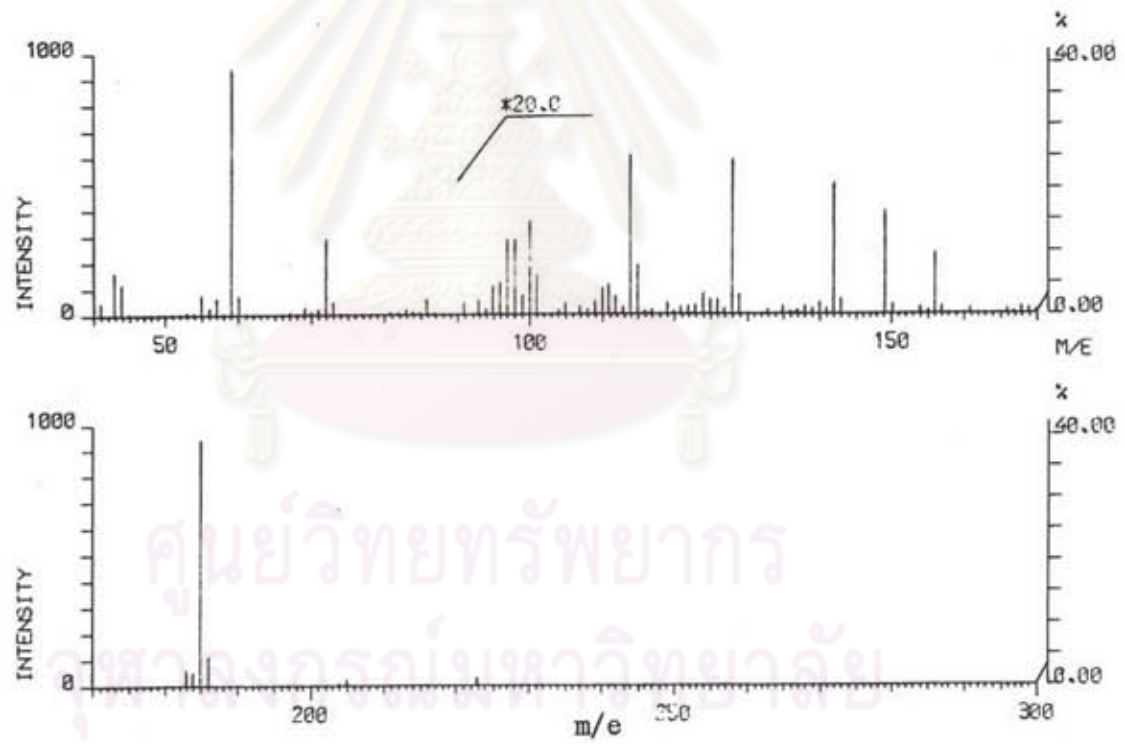


Figure 31 Mass spectrum of compound B-12

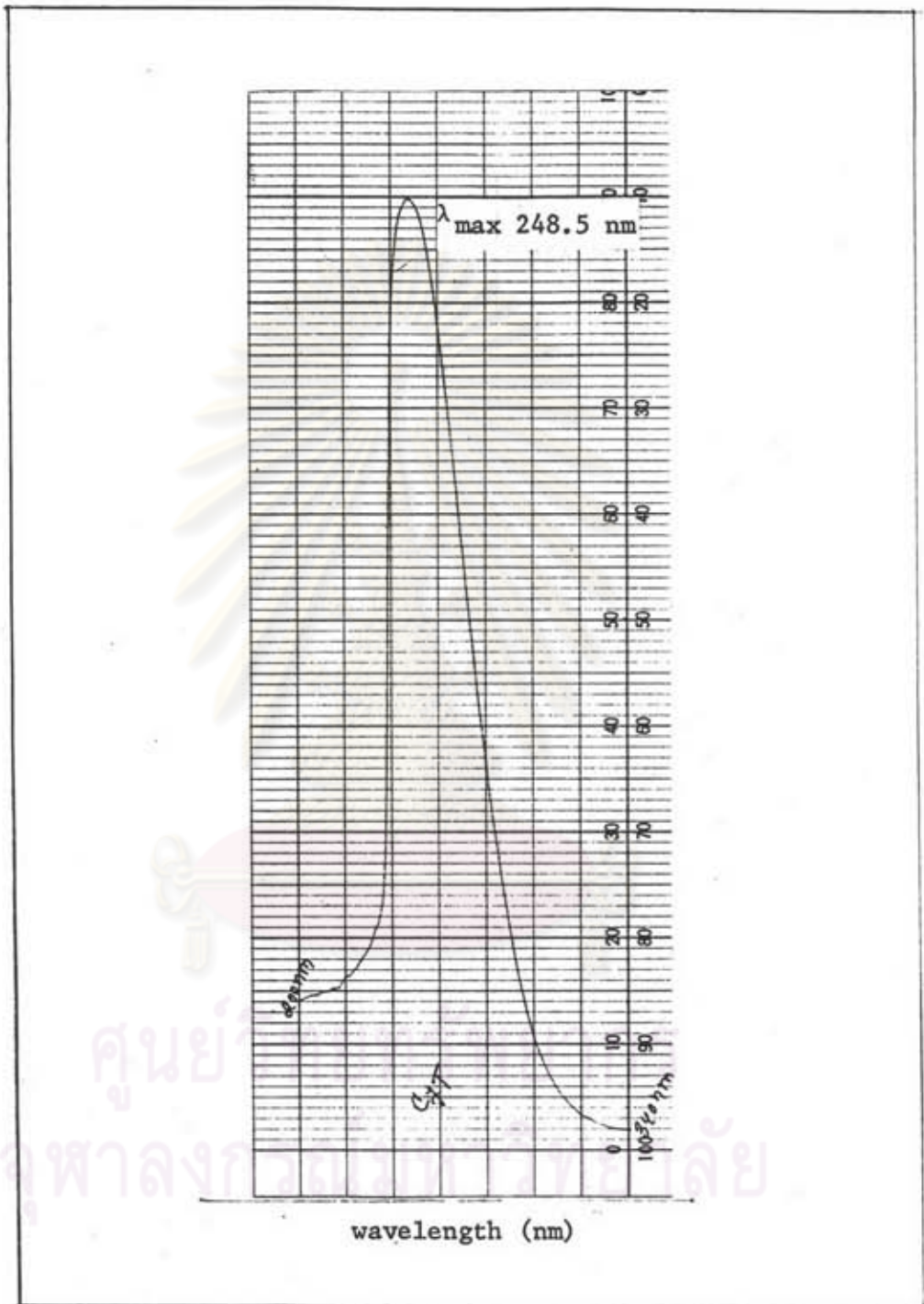


Figure 32 Ultraviolet spectrum of compound C-7 in CHCl_3

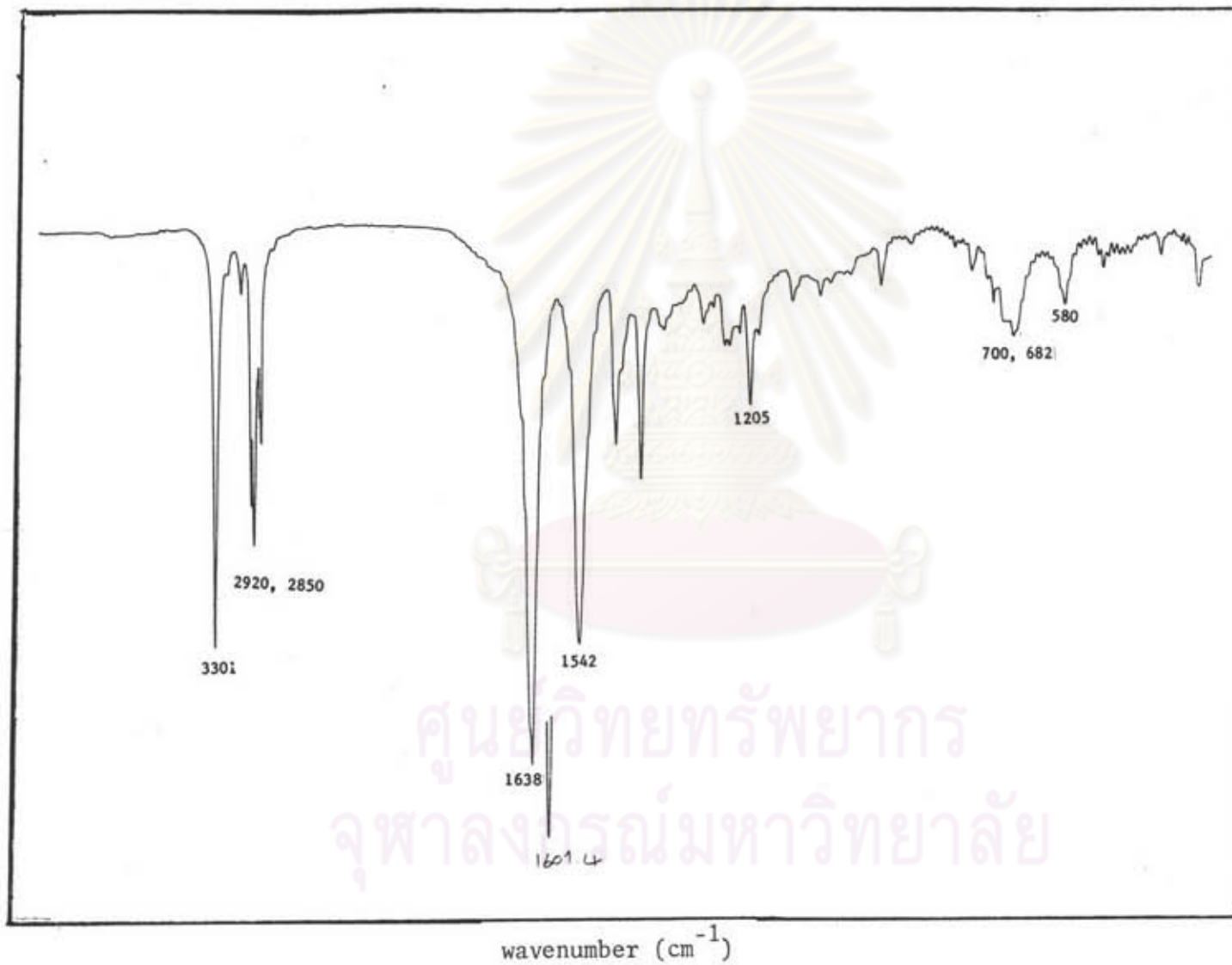


Figure 33 Infrared spectrum of compound C-7 in KBr disc

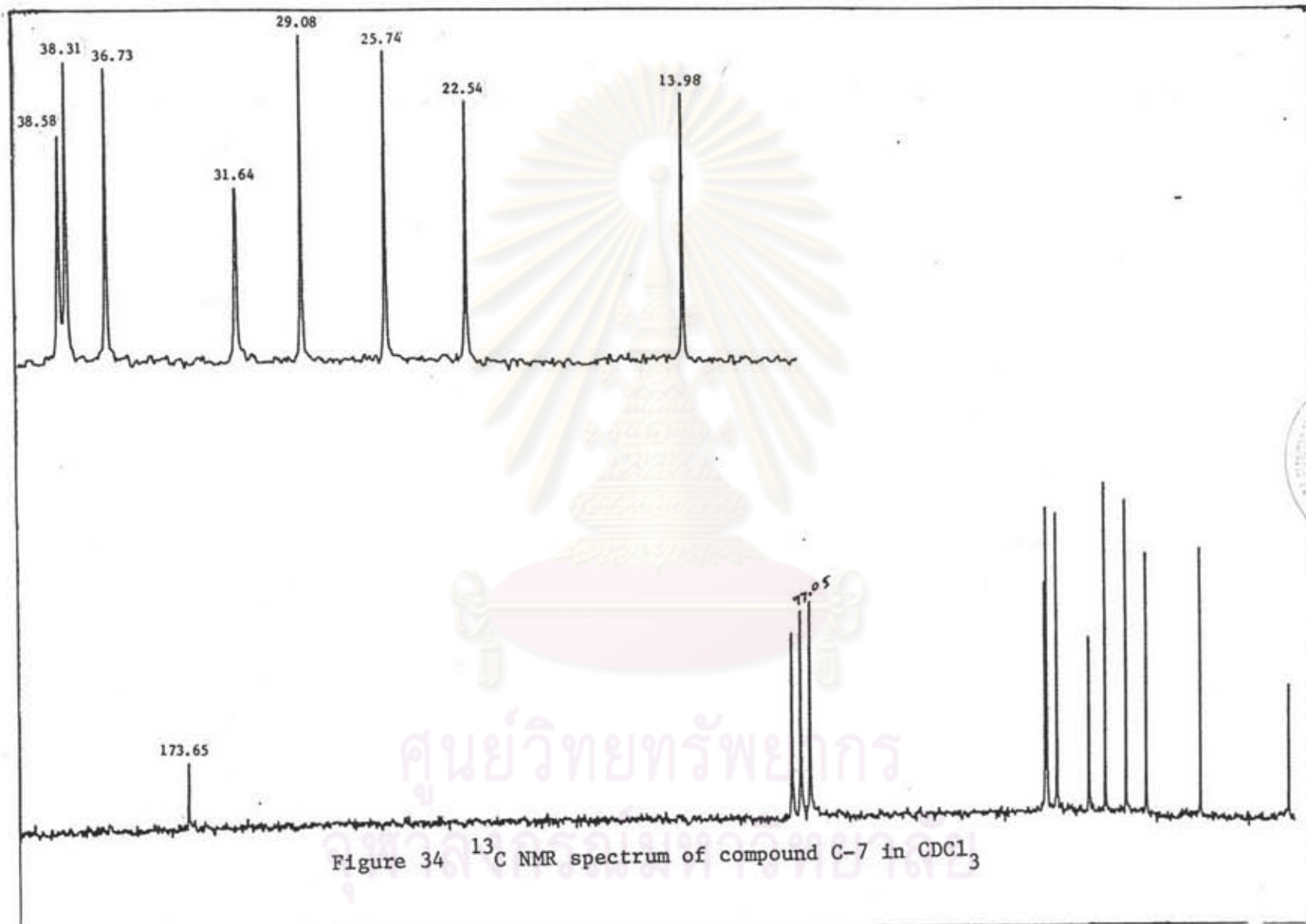


Figure 34 ^{13}C NMR spectrum of compound C-7 in CDCl_3

δ (ppm)

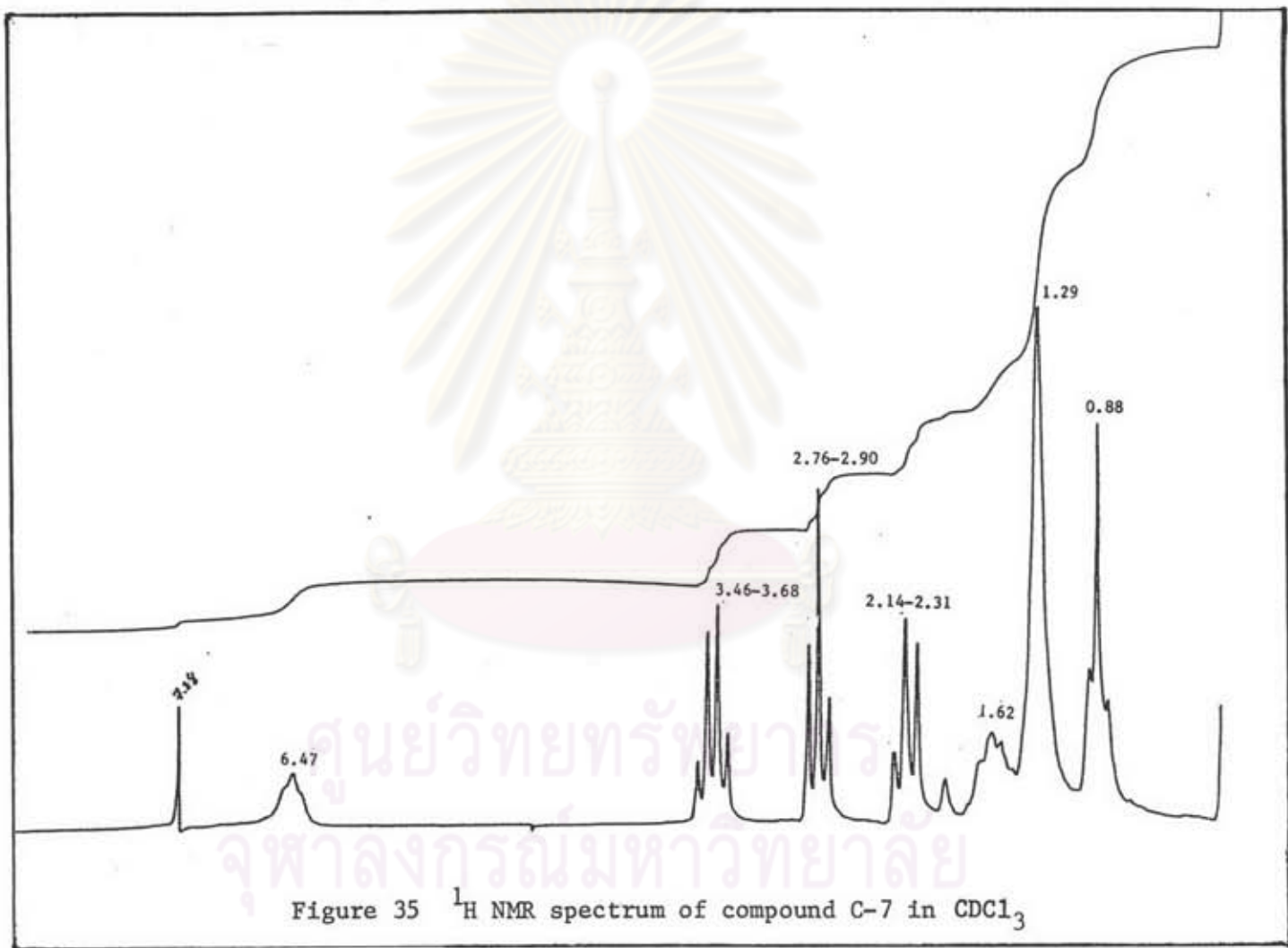


Figure 35 ^1H NMR spectrum of compound C-7 in CDCl_3

δ (ppm)

MASS SPECTRUM : (64)
SAMPLE: 07, M.P. = 108-110, CHECK MASS, 2 FEB 84.
NOTE : INJ NO=113, DI, T1=RT, T2=170, R1=32.
R.T. 4'43" RID 299.2

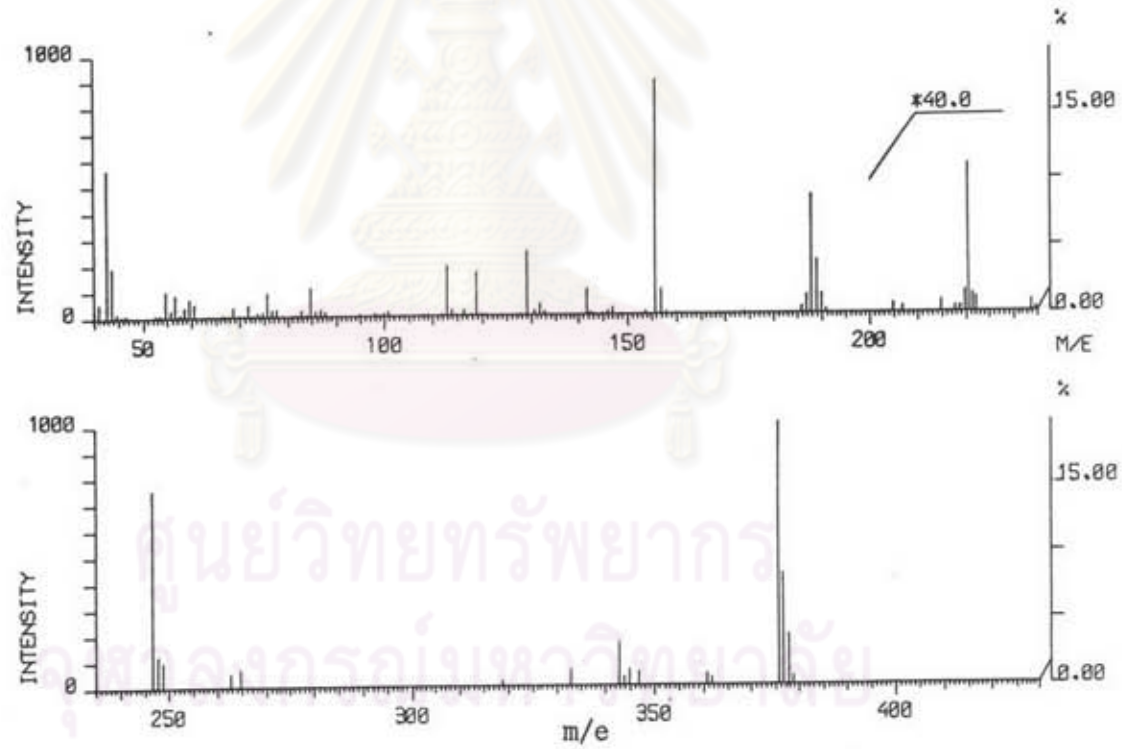


Figure 36 Mass spectrum of compound C-7

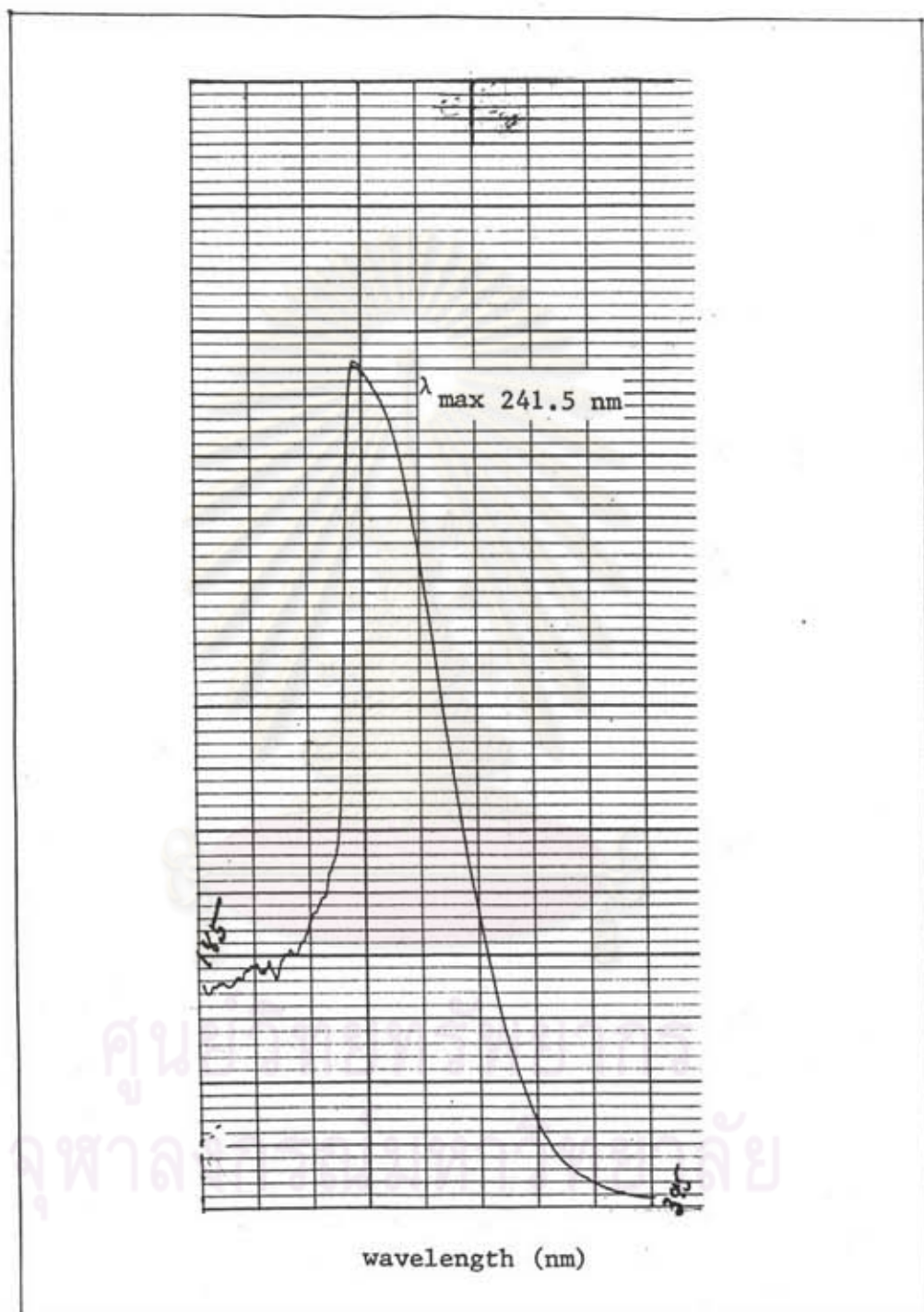


Figure 37 Ultraviolet spectrum of compound C-8 in CHCl_3

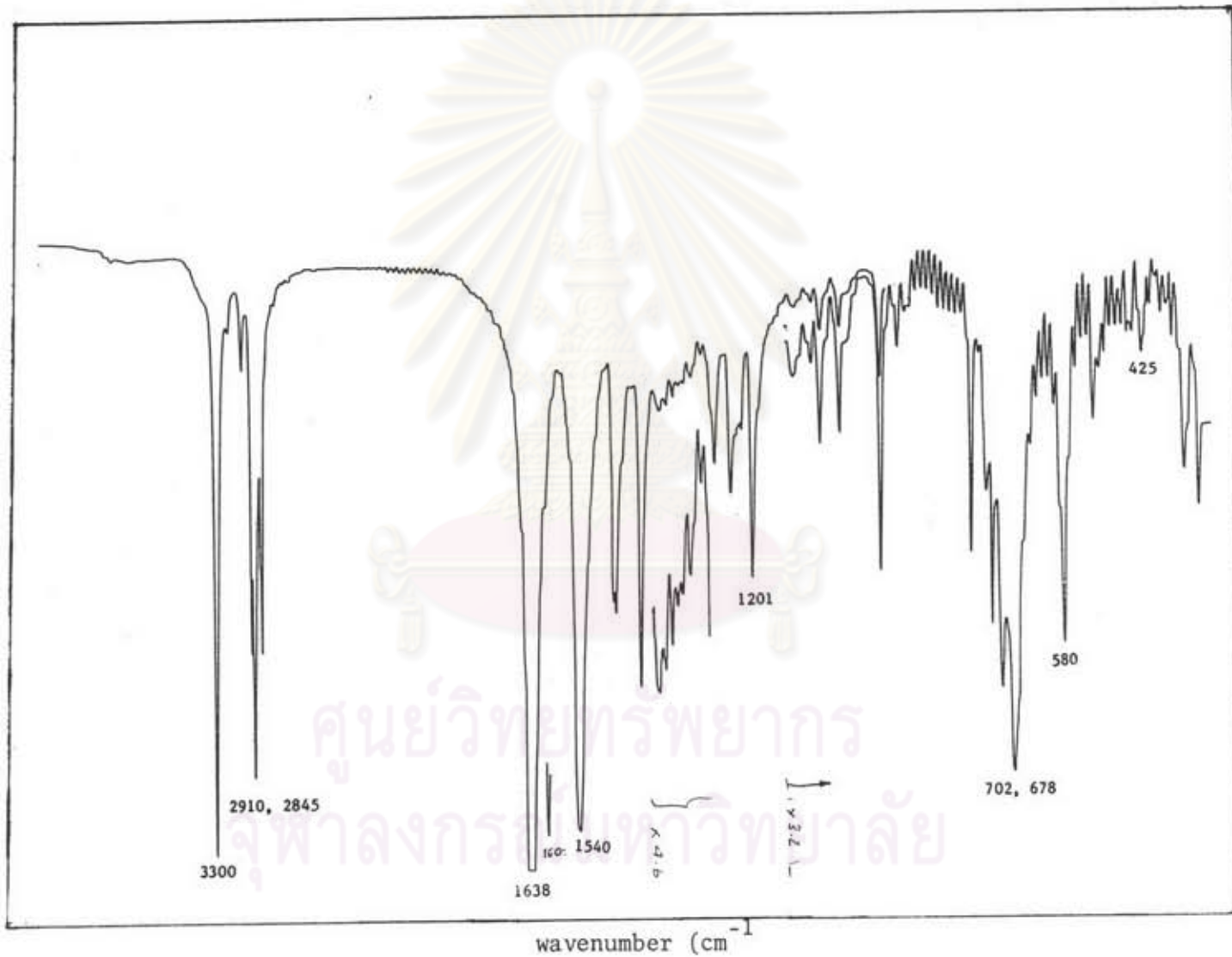


Figure 38 Infrared spectrum of compound C-8 in KBr disc

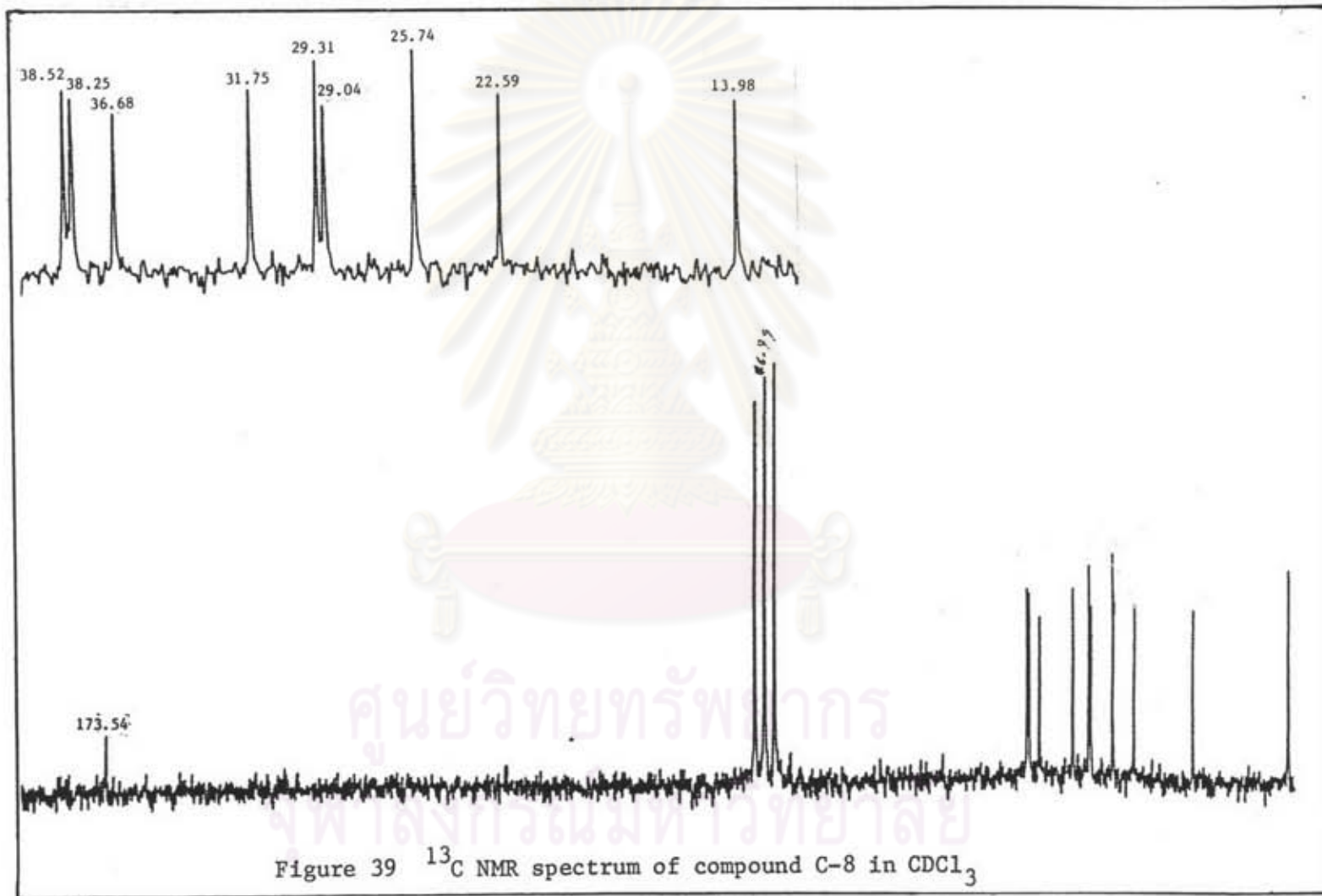
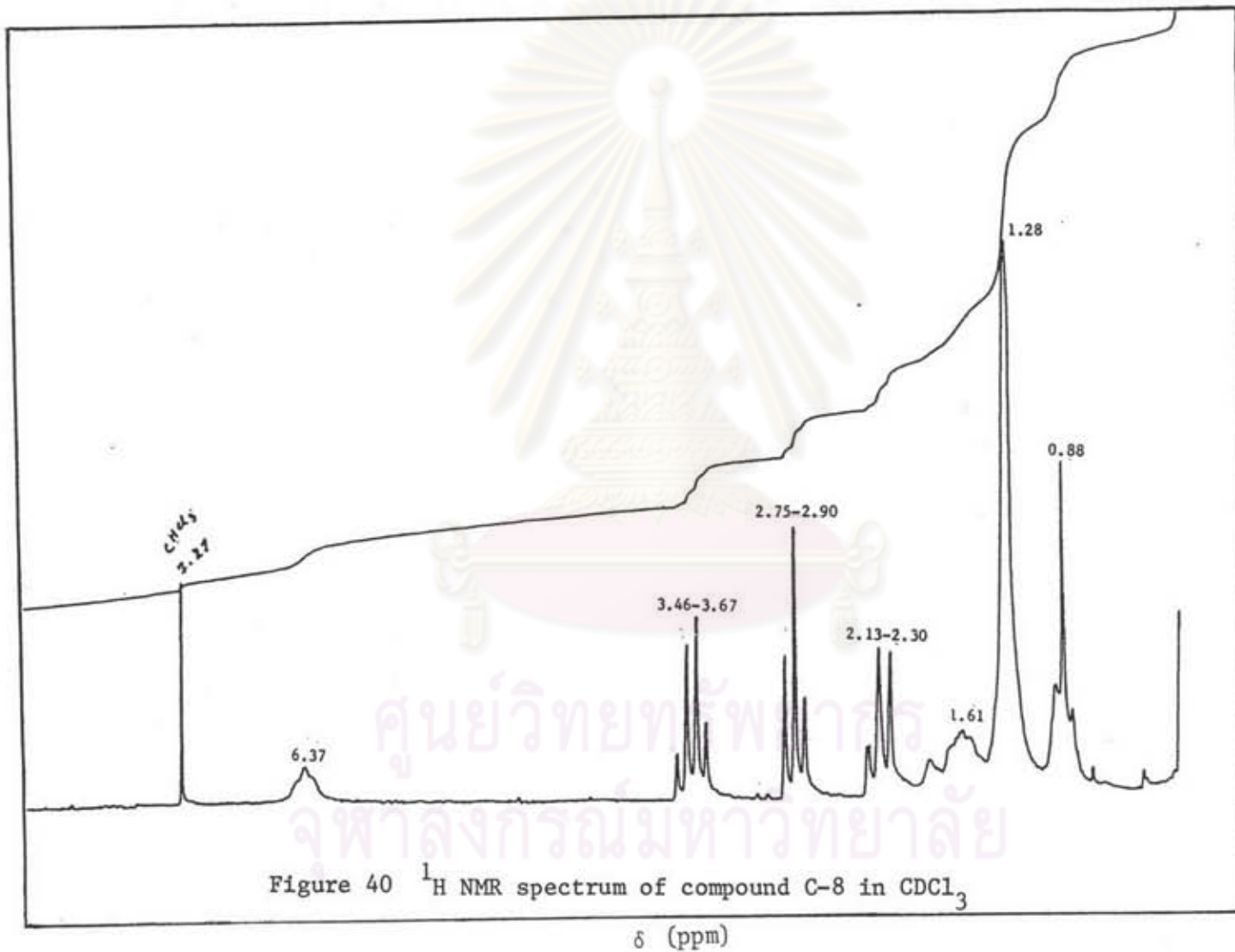


Figure 39 ^{13}C NMR spectrum of compound C-8 in CDCl_3

δ (ppm)



MASS SPECTRUM : (65)
SAMPLE:08, M.P.=114-116, 30 JAN 84.
NOTE : INJ NO=95 DI, T1=80, T2=220 R1=3.
R.T. 4'48" RI' 241.1

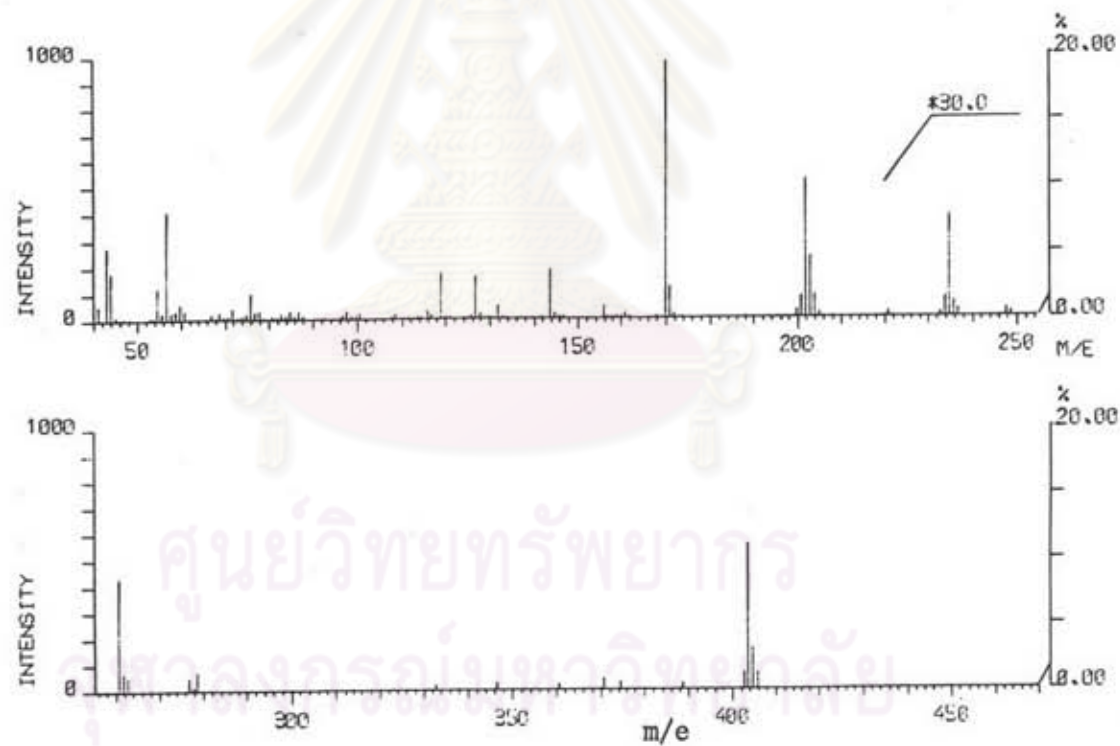


Figure 41 Mass spectrum of compound C-8

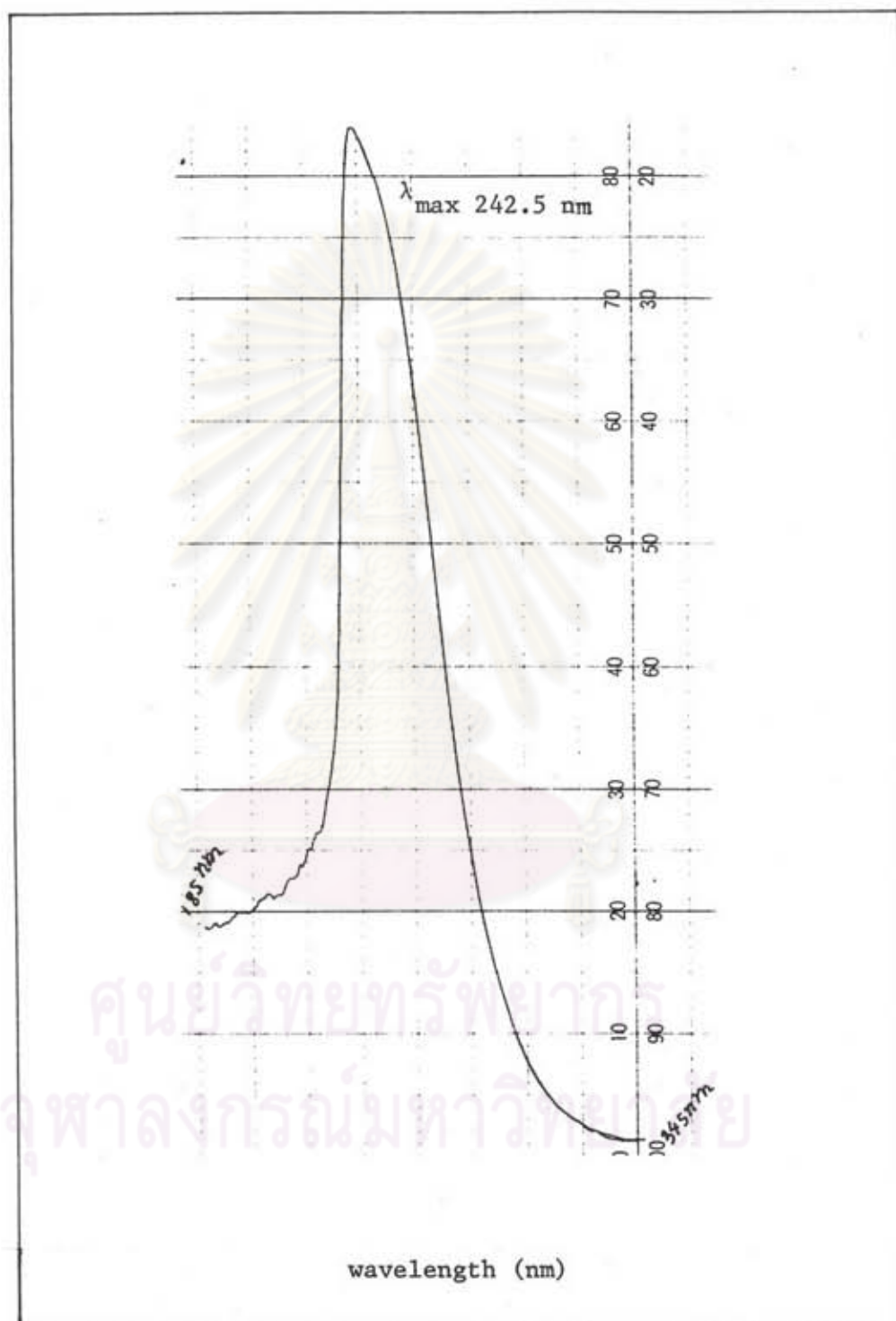


Figure 42 Ultraviolet spectrum of compound C-9 in CHCl_3

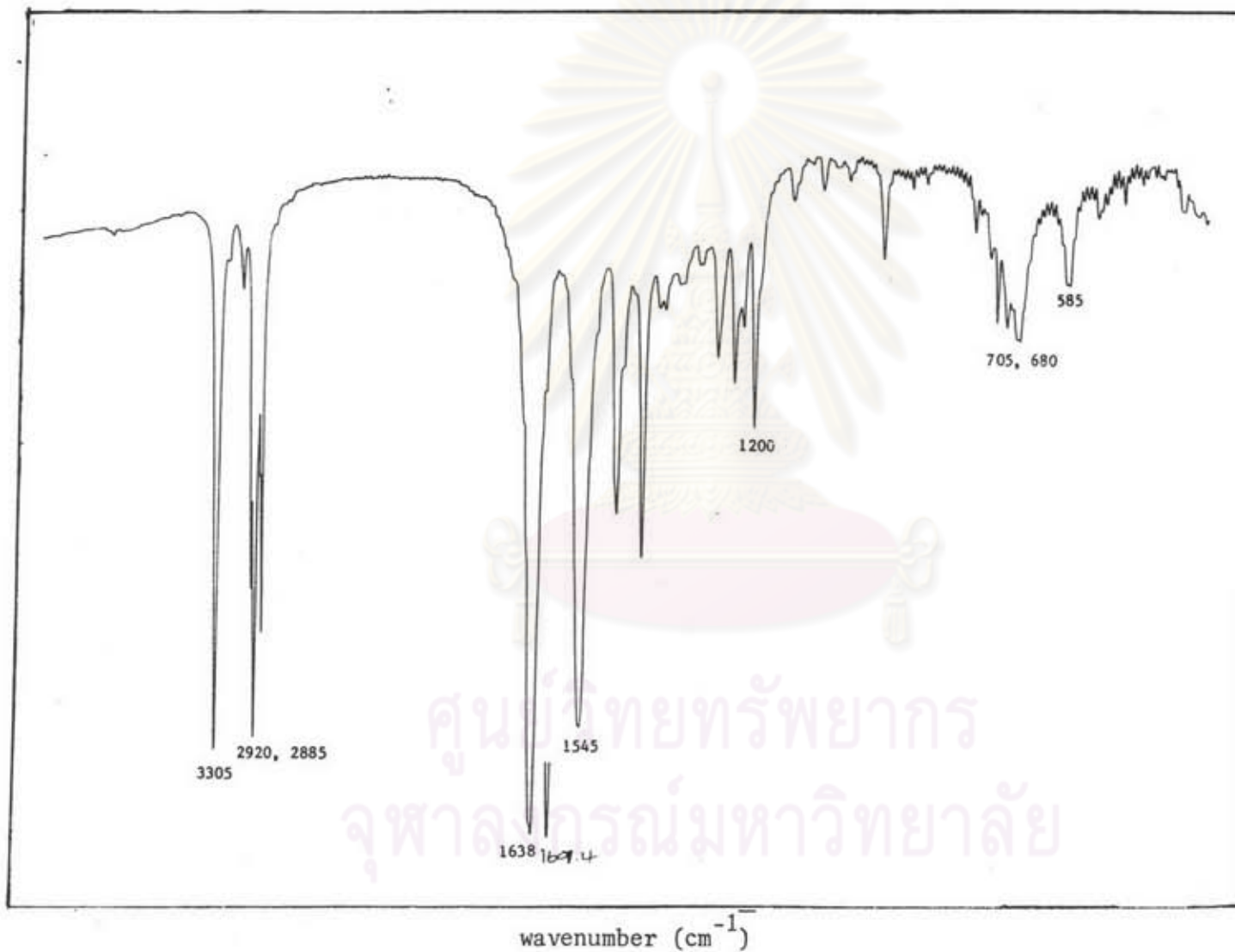


Figure 43 Infrared spectrum of compound C-9 in KBr disc

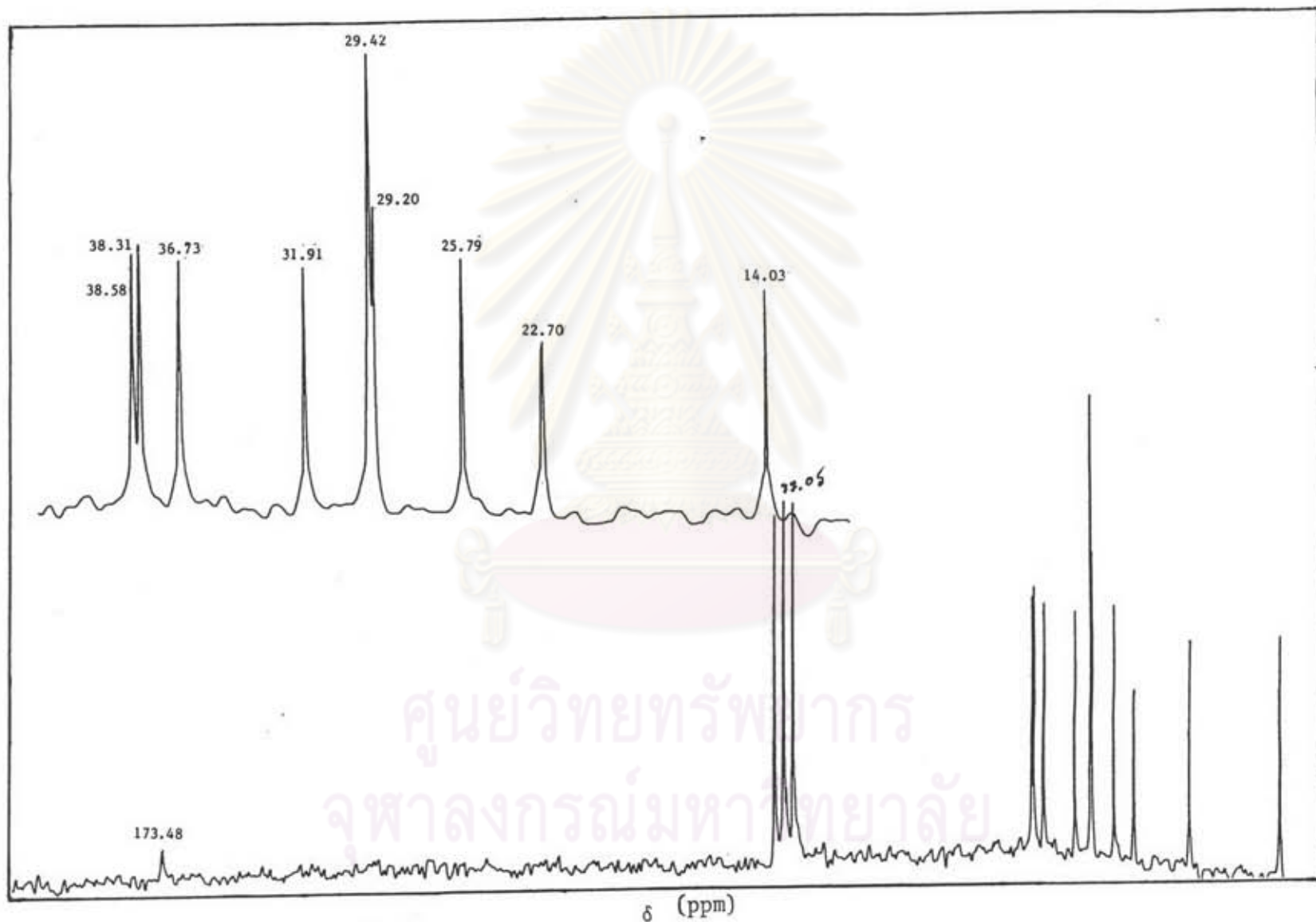


Figure 44 ^{13}C NMR spectrum of compound C-9 in CDCl_3

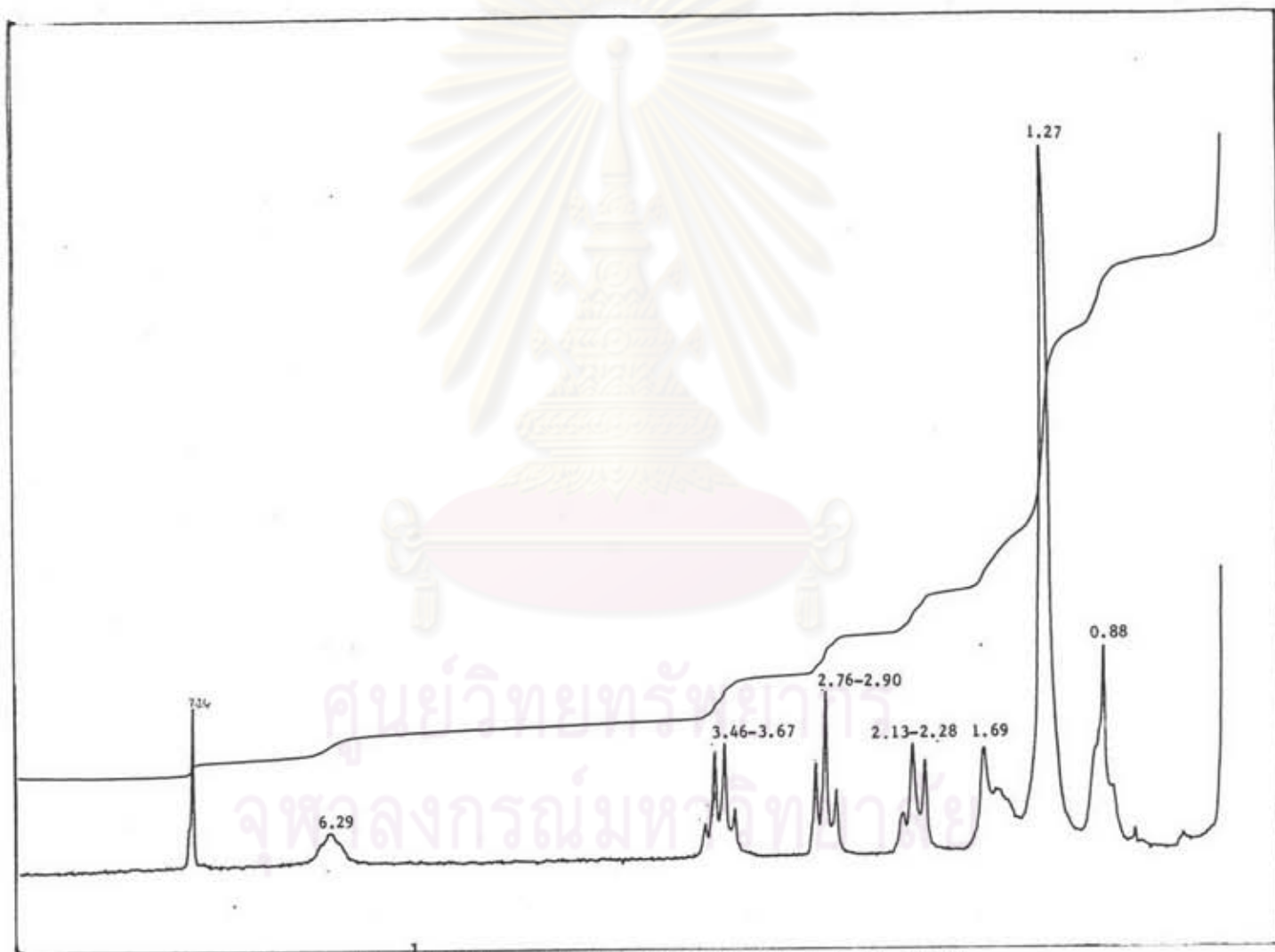


Figure 45 ^1H NMR spectrum of compound C-9 in CDCl_3

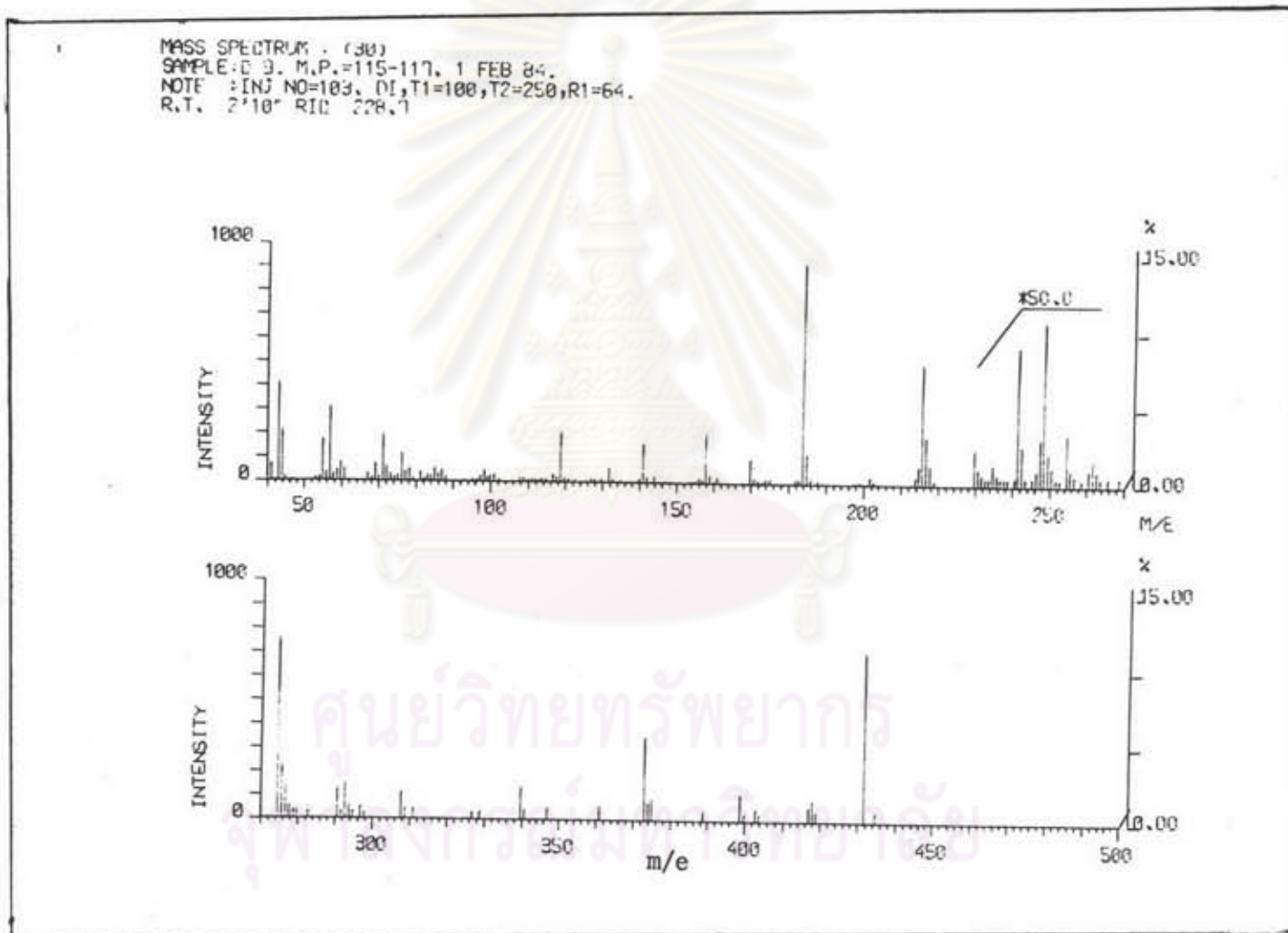
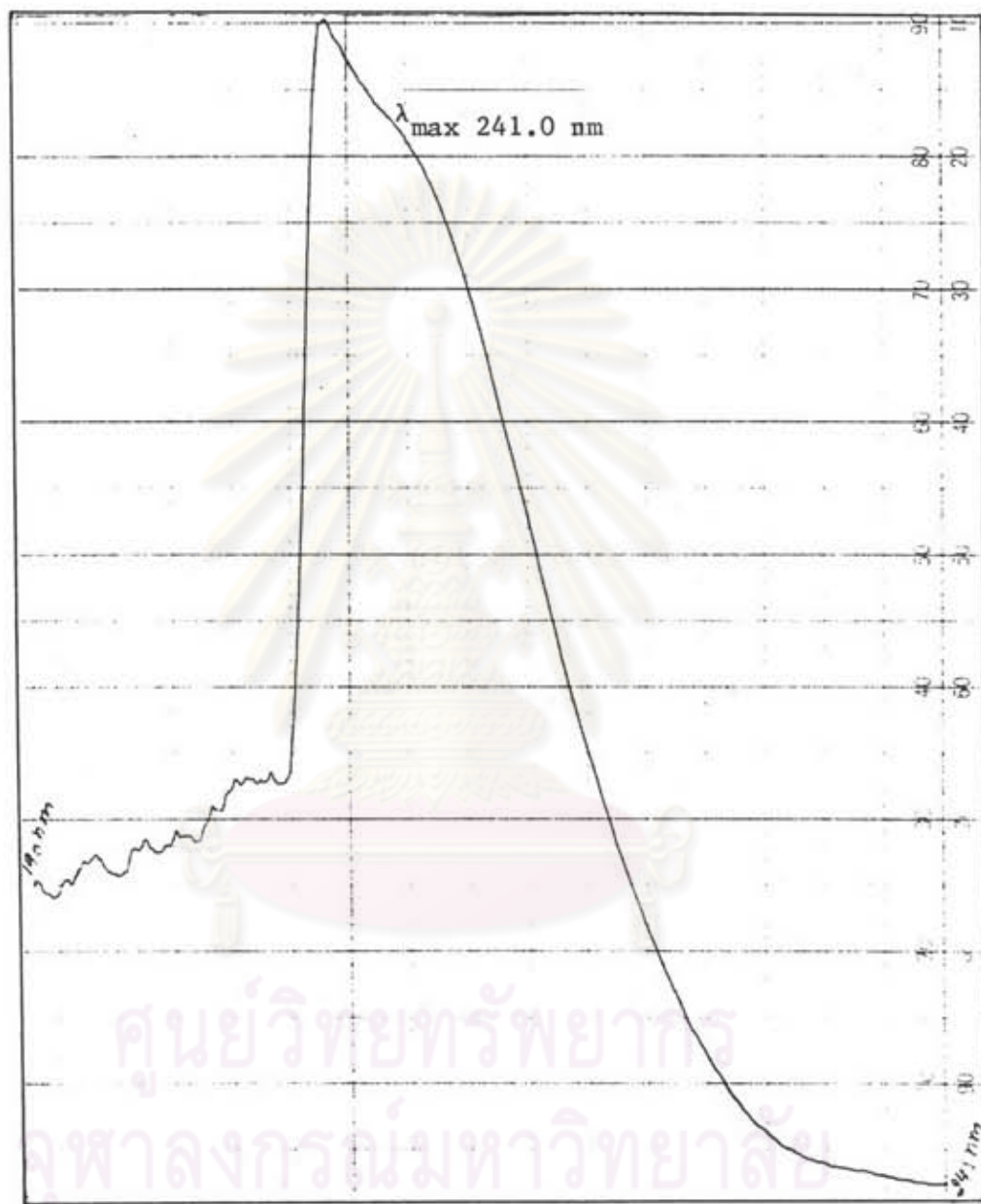
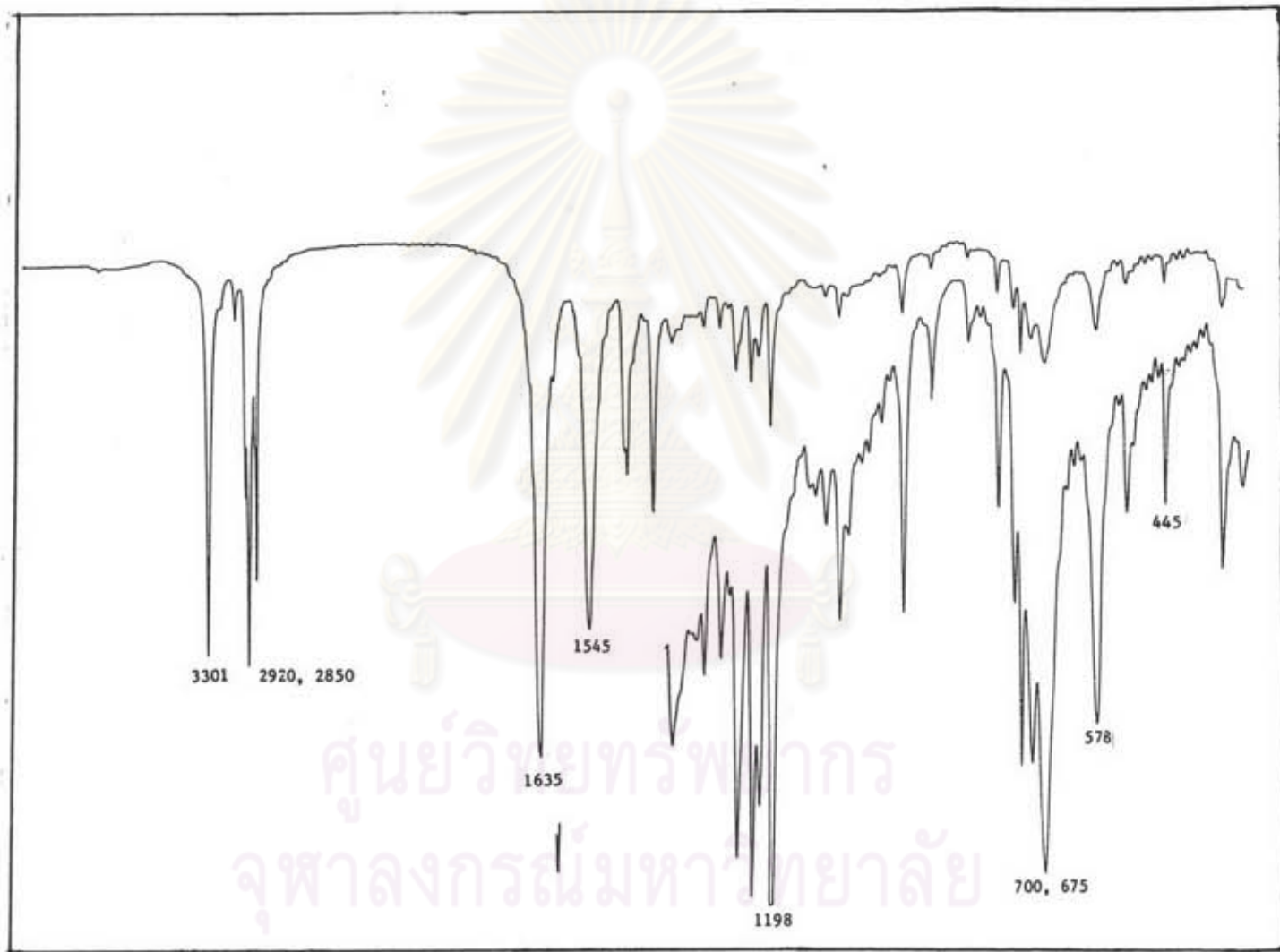


Figure 46 Mass spectrum of compound C-9



wavelegnth (nm)

Figure 47 Ultraviolet spectrum of compound C-10 in CHCl_3



wavenumber (cm⁻¹)
Figure 48 Infrared spectrum of compound C-10 in KBr disc

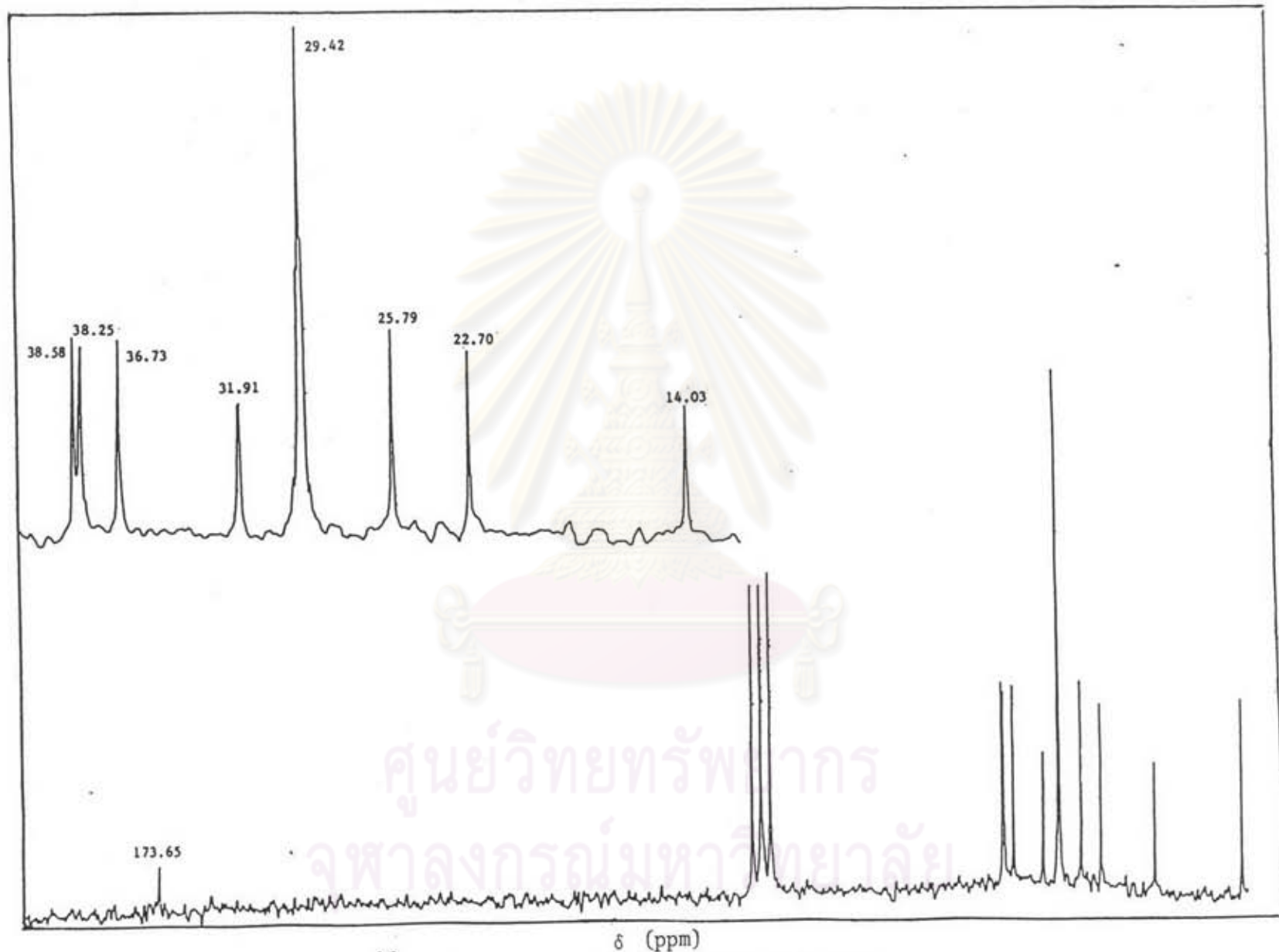
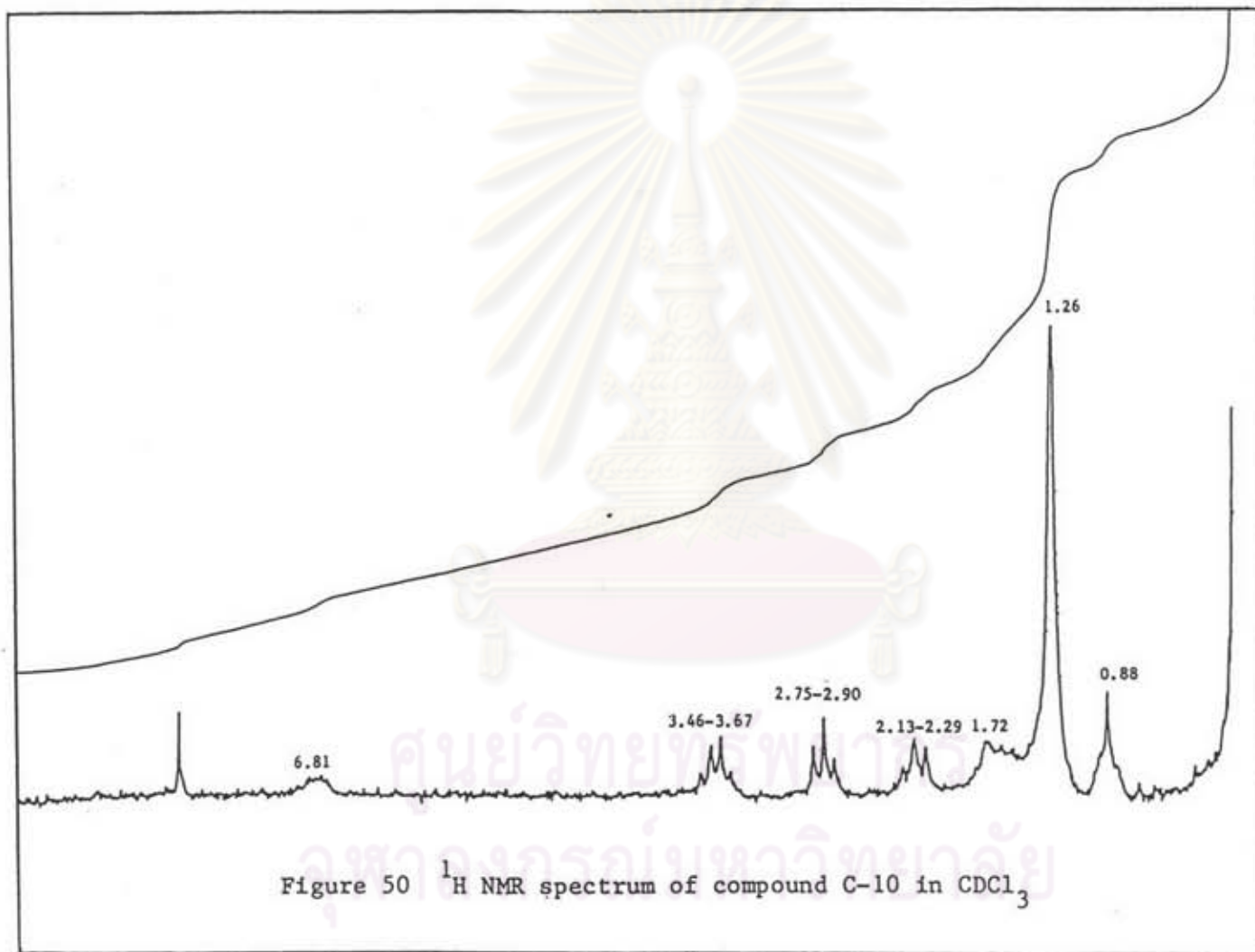


Figure 49 ^{13}C NMR spectrum of compound C-10 in CDCl_3 .



δ (ppm)

MASS SPECTRUM : (30)
SAMPLE: C10. M.P.=119-121. 2 FEB 84.
NOTE . INJ NO=111. DI, T1=100, T2=300, R1=54.
R.T. 2'10" RIC 308.1

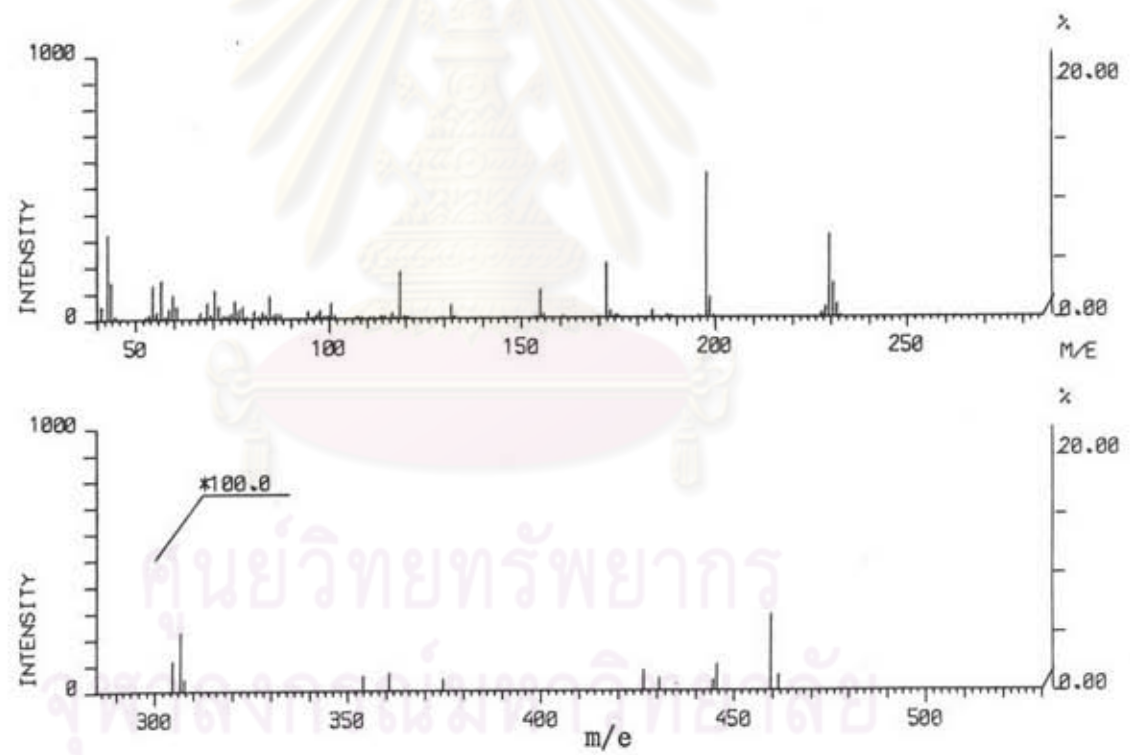


Figure 51 Mass spectrum of compound C-10

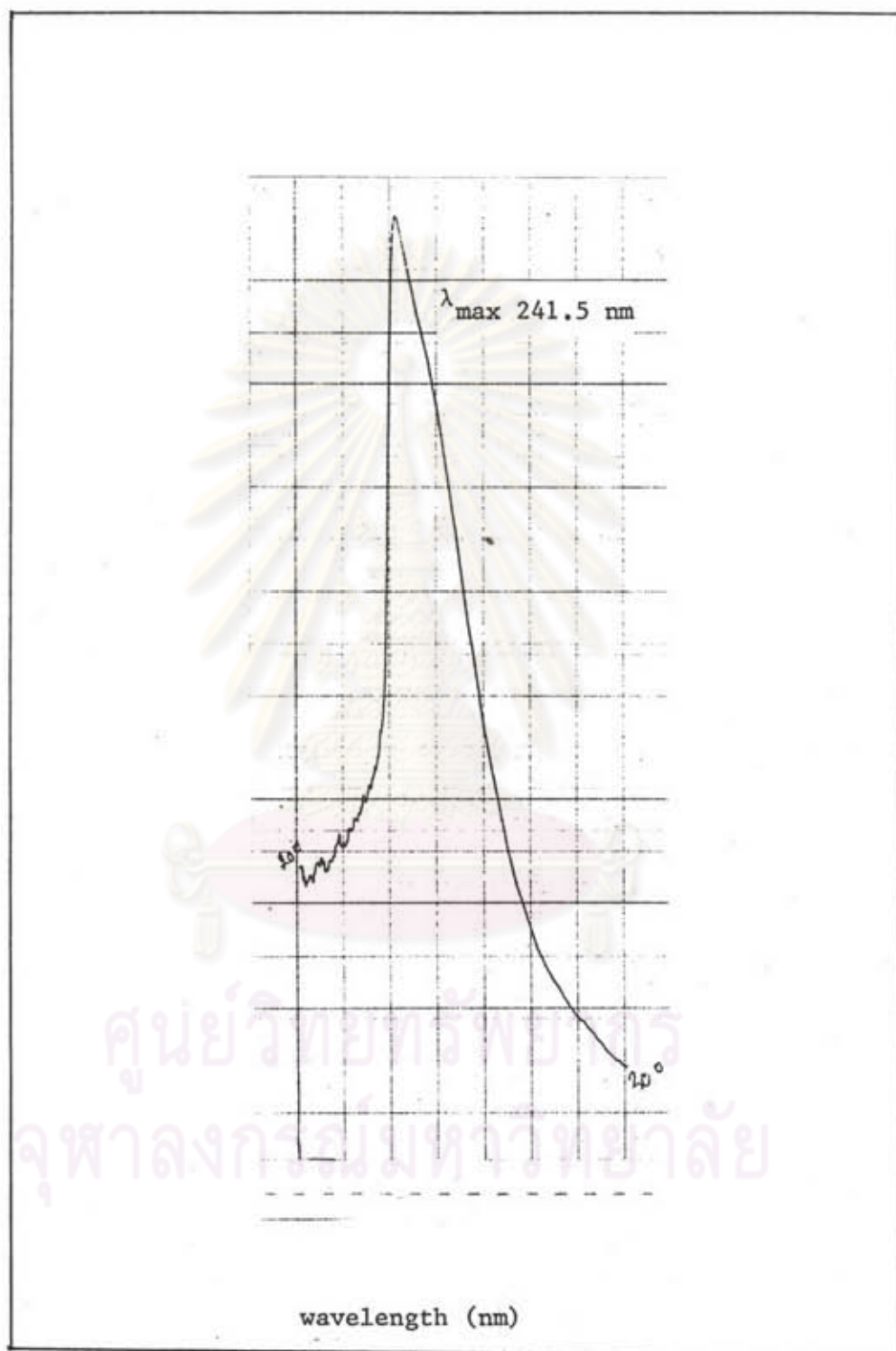
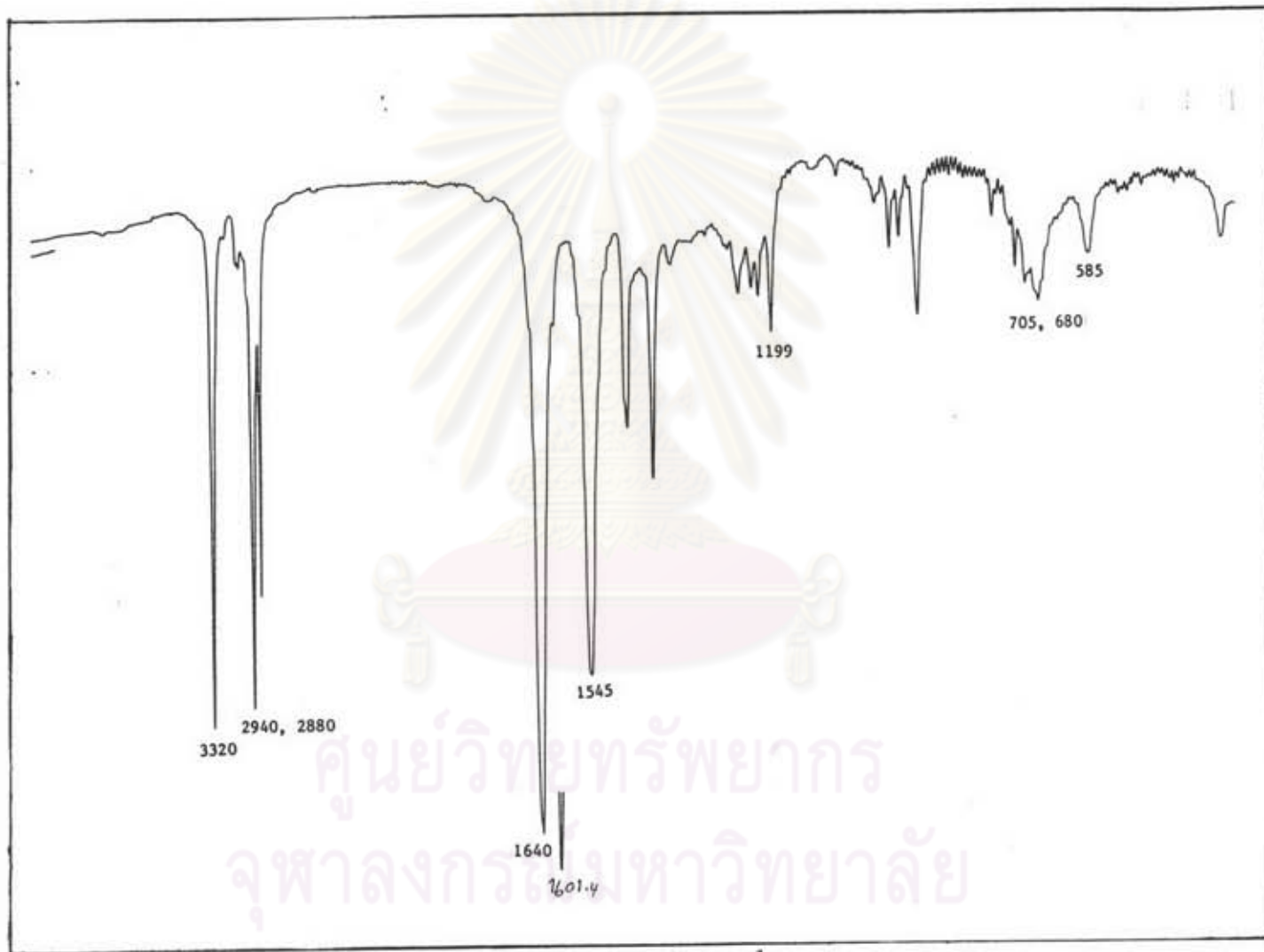


Figure 52 Ultraviolet spectrum of compound C-11 in CHCl_3



wavenumber (cm⁻¹)

Figure 53 Infrared spectrum of compound C-11 in KBr disc



MASS SPECTRUM : (31)
SAMPLE: D 12, M.P.=101-103,
NOTE : [IN] NO=109, DI, T1=100, T2=300, R1=64,
R.T. 2.151, RID 366.9

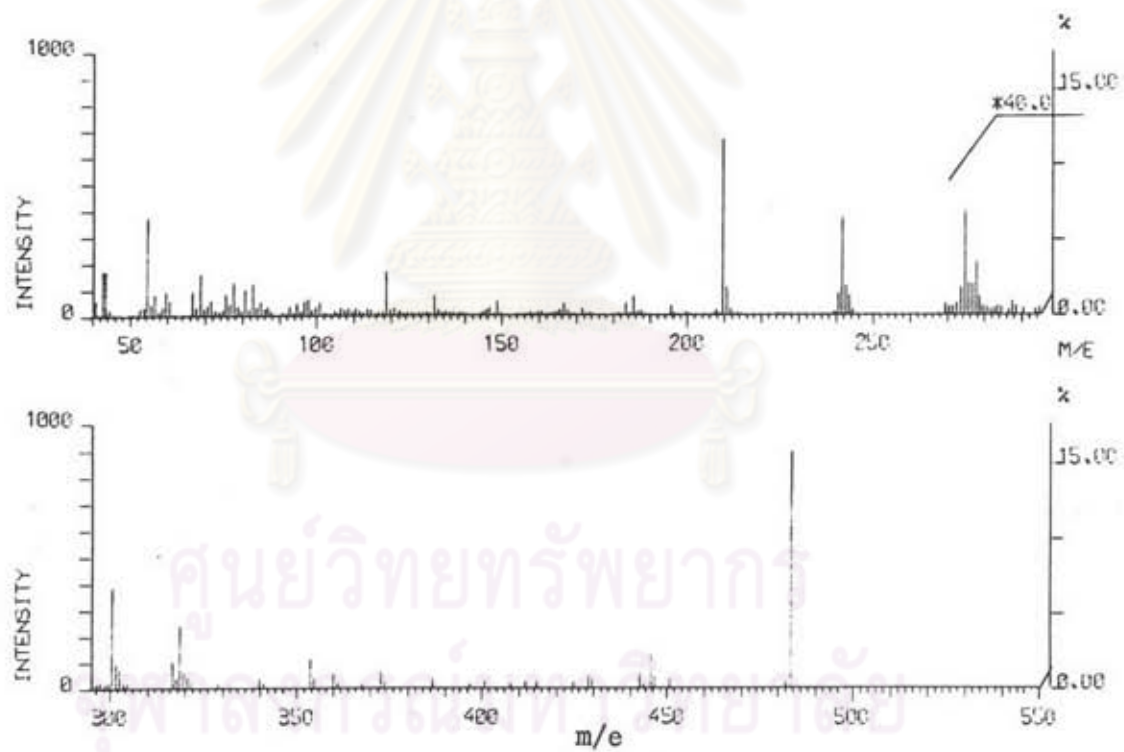


Figure 54 Mass spectrum of compound C-11

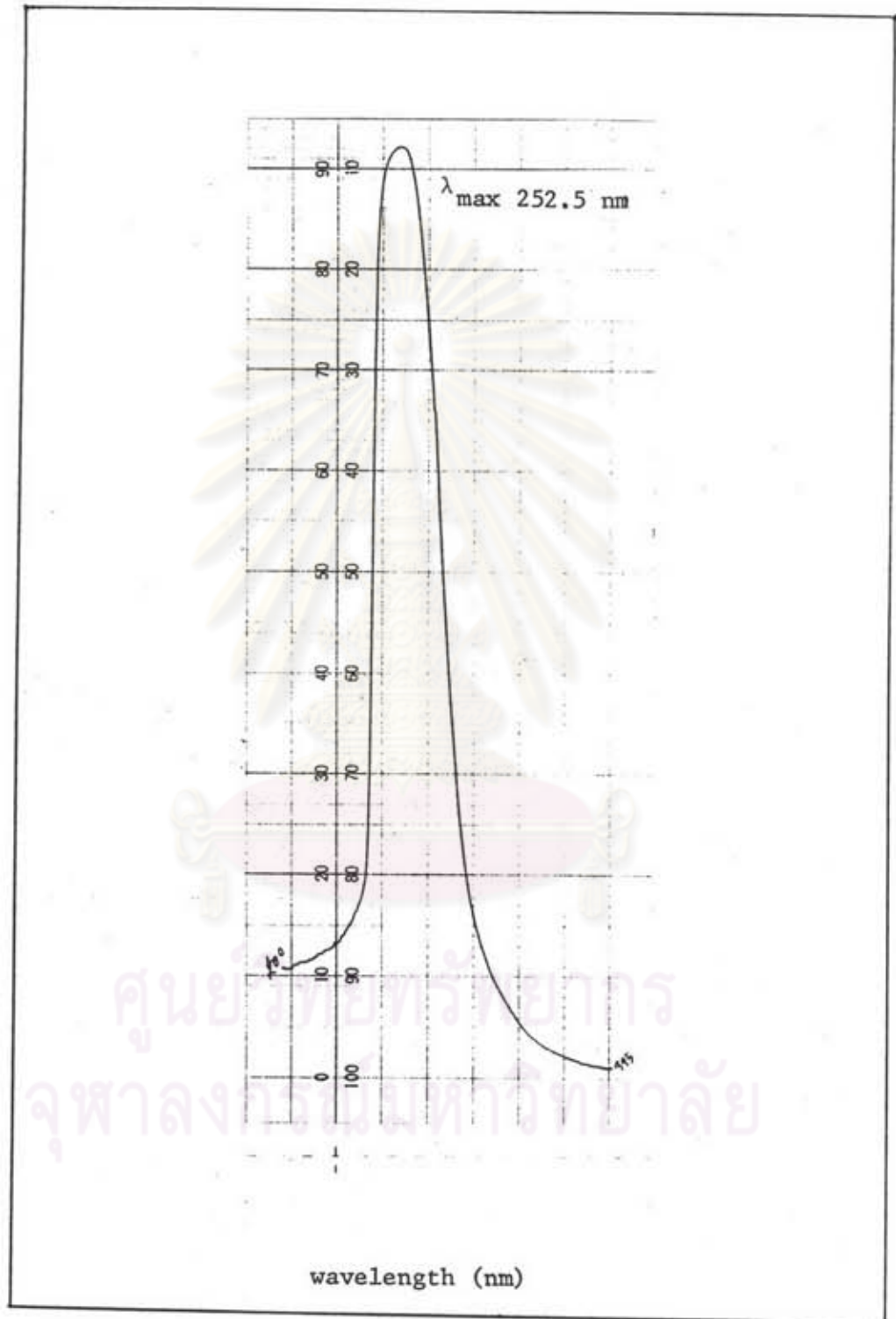
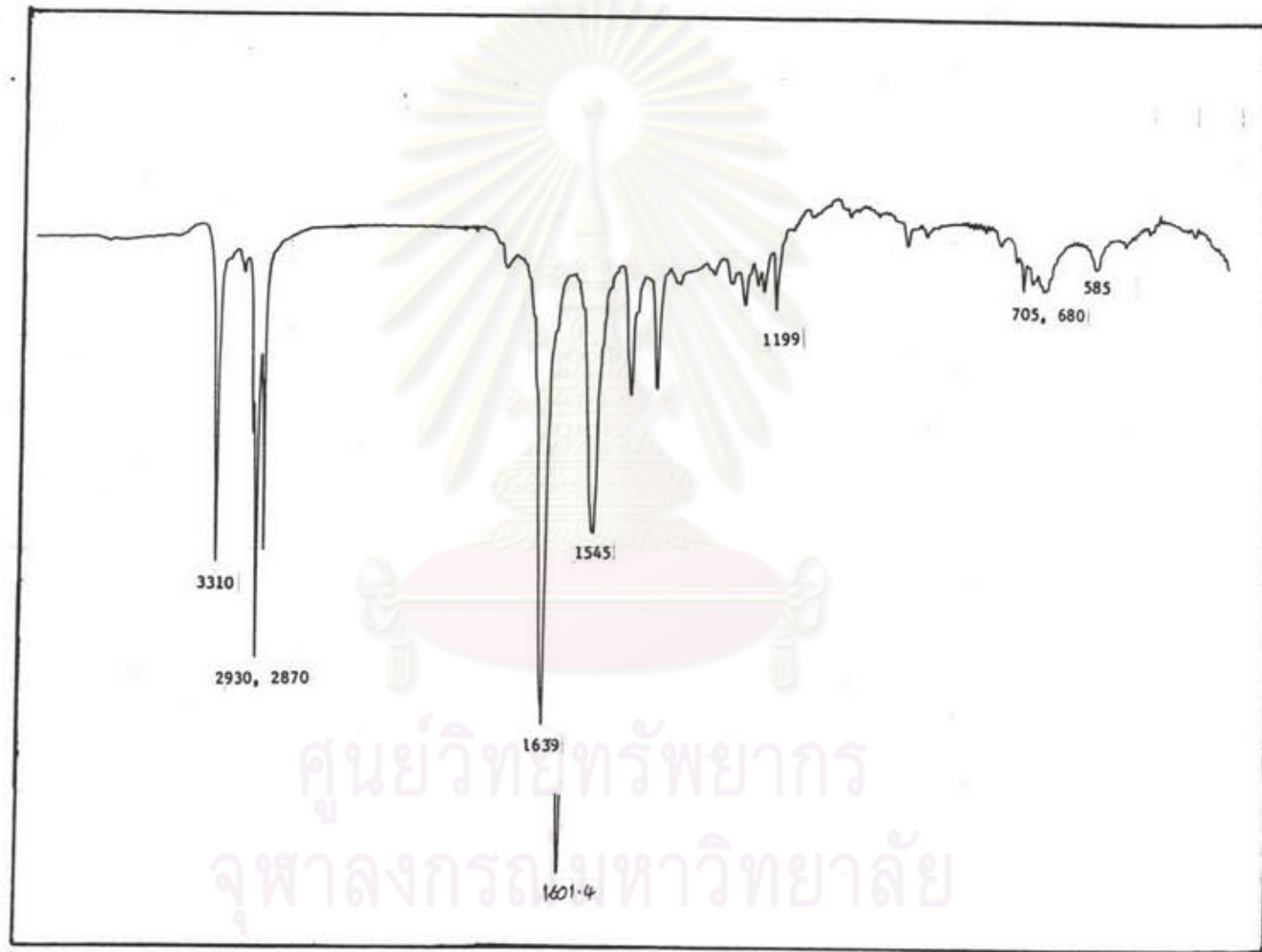


Figure 55 Ultraviolet spectrum of compound C-12 in CHCl_3



wavenumber (cm⁻¹)
 Figure 56 Infrared spectrum of compound C-12 in KBr disc

MASS SPECTRUM : (30)
SAMPLE: 0 1g, M.P.=98-100, 1 FEB 84.
NOTE : INJ NO=105, DI, T1=100, T2=300, R1=64.
R.T. 2'10" RI: 19'1

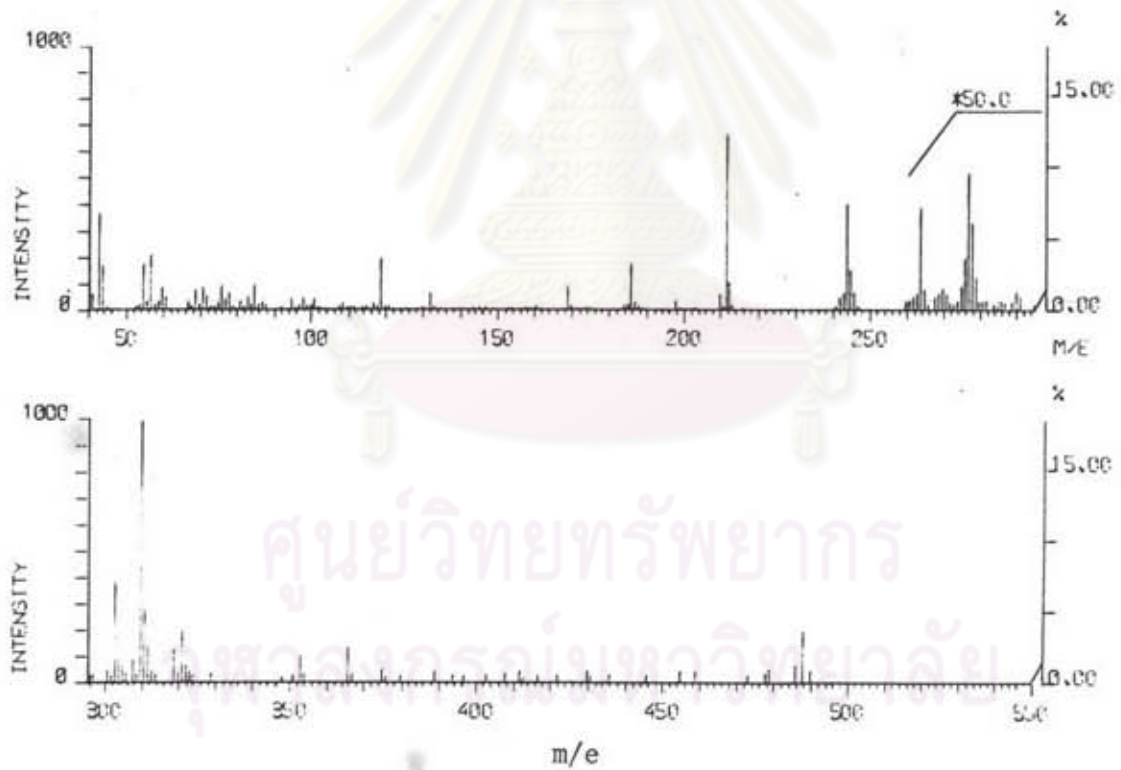


Figure 57 Mass spectrum of compound C-12