

## REFERENCE

- Antony, T. Tu. Marine Toxins and Venoms. Vol. 3. New York: Marcel Deller, Inc., 1988.
- Apsimon, F.W., *et al.* Sponge sterols, Marine Natural Products Chemical and Biological Perspectives. Vol. V., p. 57. New York : A Subsidiary of Harcourt Brace Jovanovich Publishers, 1983.
- Barnes, R.D. Invertebrate Zoology. 4<sup>th</sup> ed. Tokyo : Japan Holt - Saunders, 1980.
- Bohlin, L., Gehrken, H.P., Scheuer, P.J., and Djerassi, C. Minor and trace sterols in marine invertebrates XVI. 3 $\xi$ -Hydroxymethyl-A-nor-5 $\alpha$ -gorgostane, a novel sponge sterol. Steroids. Vol. 35 (1980) : p. 295.
- Braekman, J.C., *et al.* The sterols of marine invertebrates : Composition, biosynthesis, and metabolites. Marine Natural Products. Vol. II., pp. 76 - 159. New York : A Subsidiary of Harcourt Brace Jovanovich Publishers, 1987.
- Brusca, R.C., and Brusca, G.J. Invertebrate. Sunderland : Sinauer Associates, 1990.
- Chmitz, F.J. In P.J. Scheuer (ed.), Marine Natural Products. Vol. I., p. 241. New York : Academic Press, 1978, p. 241.
- De Stefano, A., and Sodano, G. Metabolism in porifera. XII. further informations on the biosynthesis of 3 $\beta$ -hydroxymethyl-A-nor-steranes in the sponge *Axinella verrucosa*. Experientia. Vol. 36 (1980) : p. 630.

- Delseth, C., Kashman, Y. and Djerassi, C. Helvetica Chimica Acta Vol. 62, Fasc. 6 (1979) - Nr. 208, P. 2037 - 2045.
- Desqueyroux - Faundez, R. Révision de la collection d'éponges d'Amboine (Moluques, Indonésie) constituée par Bedot and Pictet et conservée au Muséum d'histoire naturelle de Genève. Revue Suisse de Zoologie Vol. 88, No. 3 (1981) : 723 - 764, Figs. 1 - 132.
- Ding D., Huaibin W., and Guangyi L., Chemical research on the metabolites of *Lactarius hygginus*. Tianran Chanwn Yanjiu Yu Kaifa Vol. 4, No. 2 (1992) : p. 44-47 (Ch.).
- Djerassi, C., Theobald, N., Kokke, W.C.M.C., and Pak, C.S. & Carlson R.M.K., Pure Appl. Chemistry, in press.
- Fautin, D.G. Biomedical Importance of Marine Organisms. San Francisco : California Academy of Sciences, 1988.
- Fieser F.L., and Fieser M., in M. Asian ed. Steroid. New York : Reinhold publishing corporation (1959).
- Fusetane, N. Marine metabolites which inhibit development of echinoderm embryos. In P.J. Scheuer, ed., Bioorganic Marine Chemistry, pp. 60 - 92. Berlin : Springer - Berlag, 1987.
- Fusitani, N., Matsunaga, S., and Konosu, S. Bioactive marine metabolites. II. Halistanol sulfate, an antimicrobial novel steroid sulfate from the marine sponge *Halichondria* CF. *moorei* Bergquist. Tetrahedron Lett. Vol. 22 (1981) : p. 1985.

- Giudice, G. Developmental Biology of the Sea Urchin Embryo. London : Academic Press, 1973.
- Goad, L.J. The sterols of marine invertebrates : composition, biosynthesis, and metabolites, In P.J. Scheuer (ed.), Marine Natural Products. Vol II. New York : Academic Press, New York 1978.
- Gunatilaka, A.A.L., Gopichand, Y., Schmitz, F.J., and Djerassi, C., Minor and trace sterols in marine invertebrates. 26. isolation and structure elucidation of nine new  $5\alpha,8\alpha$ -epidioxy sterols from four marine organisms. J. Org. Chem. Vol. 46 (1981) : p. 3860.
- Harrison, D.M., The Biosynthesis of Triterpinoids, Steroids, and Carotenoids. Natural Product Reports. (1990) : pp. 459 - 484.
- Hentschel, E. Kiesel-und Hornschwammme der Aru und Kei-Inseln. Abhandlungen Senckenbergiana naturforschende Gessellschaft (1912) : 295 - 448.
- Hill, R.A., Krebs, H., Verpoorte, R., and Wijusma, R. Progress in the Chemistry of Organic Natural Product. Vol. 49. New York : Springer - Berlag Wien, 1986.
- \_\_\_\_\_, Makin, H.L.J., Kirk, D.N., and Murphy, Dictionary of Steroids, Chemical data, Structures and billiographies. London : Chapman & Hall Ltd., University Press, Cambridge, 1991.
- Hirata, Y., and Uemura, D. Halichondrius - Antitumor polyether macrolides from a marine sponge. Pure & App. Chem. 58 (1986): 701 - 710.

- Ikegani, S., Kawada, K., Kimura, Y., and Suzuke, A. A rapid and convenient procedure for the detection of inhibitors of DNA synthesis using starfish oocytes and sea urchin embryos. Agric. Biol. Chem. Vol.43, No 1 (1979) : pp. 161 - 166.
- Ireland, C.M., *et al.* Uniqueness of the marine chemical environment : Categories of marine natural products from invertebrates. In D.G. Fautin (ed.), Biomedical importance of marine organisms, pp. 41 - 58. San Francisco : Californis Academy of Sciences, 1988.
- Itoh, T., Sica, D., and Djerassi, C. Minor and trace sterols in marine invertebrates. Part 35. isolation and structure elucidation of seventyfour sterols from the sponge *Axinella cannabina*. J. Chem. Soc., Perkin Trans. Vol. I (1983), p. 147.
- Kashman, Y., *et al.* Recent research in marine natural products from the red sea. Pure & Appl. Chem. Vol. 54, No. 10 (1982) : 1995 - 2010.
- Kobayashi, J., Ishibashi, M. The Alkaloids. Vol. 41. San Diego : Academic Press., 1992.
- Li, X., and Djerassi, C. Minor and trace sterols in marine invertebrates. 40. structure and synthesis of axinyssasterol, 25-methylfucosterol and 24-ethyl-24-methyl-cholesterol-novel sponge sterols with highly branched side chains. Tetrahedron Lett. Vol. 24 (1983) : p. 665.
- Luckner, M. Biosynthesis of Isoprenoids, Secondary Metabolism in Microorganisms, Plants, and animals, pp. 182 - 235. New York : The German Democratic Republic, 1990.



Majchacheep, S. Marine Animals of Thailand. Bangkok : Praepittaya Publisher, 1989.

Makarieva, T.N., Shubina, L.K., Kalinovsky, A.I., Stonik, V.A., and Elyakov, G.B. Steroids in porifera. II. steroid derivatives from two sponges of the Family *Halichondriidae*. Sokotrasterol sulfate, a marine steroid with a new pattern of side chain alkylation. Steroids Vol. 42 (1983), p. 267.

Meyer B.N., *et al.* Brine shrimp : A convenient general bioassay for active plant constituents. Plants Medica. 45 (1982) : 31 - 34.

Minale, L. & Sodano, G. In D.J. Faulkner, and W.H. Fenical (ed.), Marine Natural Products Chemistry, p. 87. New York : Plenum Press, 1977.

Morris, R.J. & Culkin F. In H. Barnes (ed.), Oceanographt and Marine Biology, Annual Review, p. 73 Aberdeen : Aberdeen University Press, 1977.

Mosmaun, T. Rapid colorimetic assay for cellulargrowth and survival : Application to proliferation and cytotoxic assay. J. Immulol. Met. 65 (1983) : 55 - 63.

Narahashi, T. Marine organisms as models for the study of neuropharmacology. In D.G. Fautin (ed.), Biomedical importance of marine organisms, pp. 97 - 108. San Francisco : The California Academy of Sciences, 1988.

Nakatsu, T., Walker, R.P., Thompsn, J.E., and Faulkner, D.J. Biologically active sterol sulfates from the marine sponge *Toxadocia zumi*. Experientia. Vol. 39 (1983) : p. 759.

Nes, W.R. J. Amer. Chem. Soc. Vol. 78 (1956) : p. 193.

Nes, W.R., Krevitz, K., and Behzadan, S., Configuration at C-24 of 24-Methyl and 24-Ethylcholesterol in Tracheophytes, Lipids, Vol. 11, No. 2 (1975), pp. 118 - 126.

\_\_\_\_\_, Krevitz, K., and Behzadan, S. Lipid. Vol. 11 (1976) : p.118.

\_\_\_\_\_, and Steele, J.A. J. Org. Chemistry Vol. 22 (1957) : p. 1457.

Piccialli, V. and Sica, D. Four new trihydroxylated sterols from the sponge *Spongionella gracilis*. J. Nat. Prod. Vol. 50, no. 5 (1987) : p 15-20.

Romo, B.J., Rosenkranz, G. & Djerassi, C. Steroid. XXIII.,  $\Delta^7,9(11)$ -Allopregnadiene- $3\beta,20\beta$ -diol and related compounds. J. Amer. chem. Soc. Vol. 73 (1951) : p.5489.

Rubinstein, I. & Goad, L.J. Occurrence of (24S)-24-Methylcholesta-5, 22E-Diene- $3\beta$ -ol in the diatom *Phaeodactylum tricornutum*. Phytochemistry. Vol. 13 (1974) : p. 485.

\_\_\_\_\_, Goad, L.J., Clague, A.D.H., and Mulheirn, L.J., The 220 MHz NMR spectra of phytosterols. Phytochem. Vol. 15 (1976) : pp. 195 - 200.

\_\_\_\_\_, I., Goad, L.J., Clague, A.D.H. and Mulheirn L.J. Phytochemistry Vol. 15 (1976) : p.195. See also Theobald, N. & Djerassi, C. Determination of the absolute configuration of Stelliferasterol and Strongylosterol - Two marine sterols with "Extended" side chains. Tetrahedron Letters (1978) : p. 4369 - 4372.

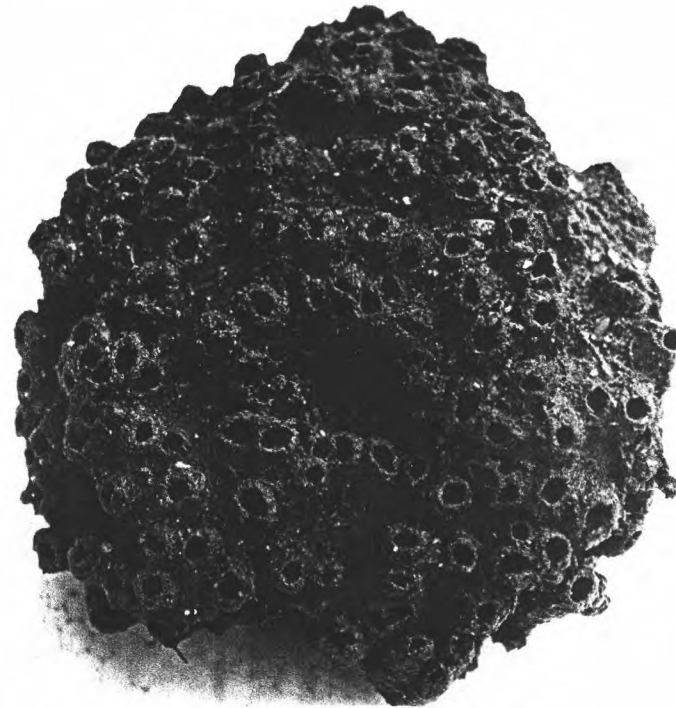
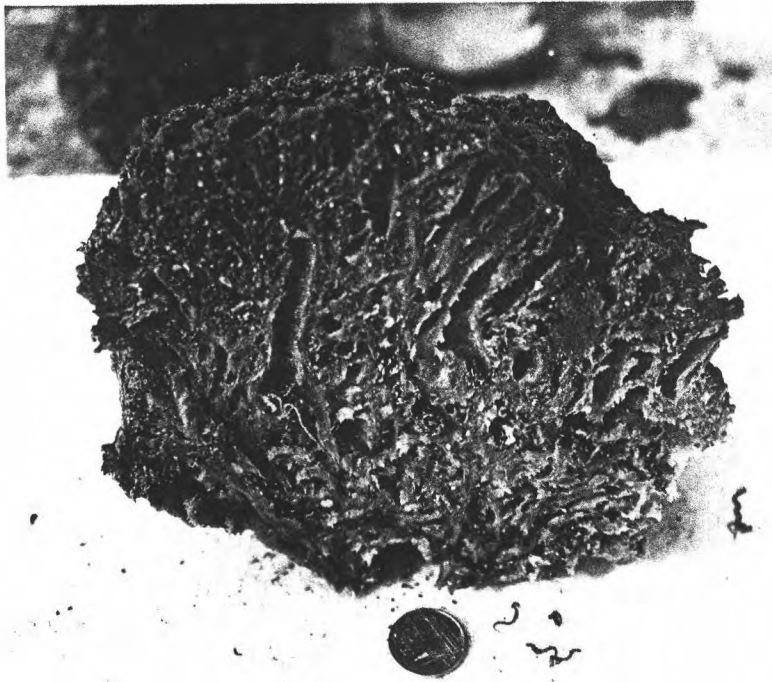
Scheuer, P.J. Chemistry of Marine Natural Products New York : Academic Press, 1973.

- Scott, A.I., *et al.* Interpretation of the Ultraviolet spectra of Natural Products. New York : Pergamon Press, The Macmillan Co., 1960.
- Shoppee C. Structure and geometry in introduction, Chemistry of The Steroids, 2<sup>nd</sup> ed. pp 9 - 29. London: Butterworths, 1964.
- Shubina, L.K., Makarieva, T.N., Boguslavskii, V.M., and Stonik, V.A. Steroid compounds of marine sponges. 1. sterols of *Esperiopsis digitata*. Khim. Prir. Soedin., 1983, p. 740.
- Sollas, I.B.J. On the sponges collected during the "Skeat Expedition" to the Malay Peninsula, 1899 - 1900. Proceedings of the Zoological Society of London Vol. 2, No. 1 (1902) : 210 - 221, pls. 14 - 15.
- Tokes, L., Jones, G., and Djerassi, C. Mass spectrometry in structural and stereochemical problems. CLXI. Elucidation of the course of the characteristic ring D fragmentation of steroids. Journal of the American Chemical Society. Vol. 90, no. 20 (1968) : p.5465 -5477.
- Topsent, E. Spongiaires de la Baie d' Amboine. Voyage de MM. M. Bedot et C. Pictet dans l' Archipel Malais. Revue Suisse de Zoologie Vol. 4 (1897b) : 421 - 487.
- Wardroper, A.M.K., and Maxwell, J.R. & Morris, R.J. Steroids. Vol. 32, (1987) : p. 203.
- Windaus, A. & Linsert, O. Liebigs Ann. Chem. Vol. 465 (1932) : p.148.

- Wolfgang, S. A systematic guide to the identification of marine organisms, Marine Fauna and Flora of Bermuda, p. 120. New York : A Wiley - Interscienc Publication, 1986.
- Wright, J.L.C., McInues, A.G., Shimiau, S., Smith, D.G., and Walter, J.A. Indentification of C-24 alkyl epimers of marine sterols by  $^{13}\text{C}$  nuclear magnetic resonance spectroscopy. Can. J. Chem. Vol. 56 (1978) : pp. 1898 - 1903.
- Yoshiro, H. Marine Toxins and Other Bioactive Marine Metabolites. Japan : Japan Scientific Societies Press, 1979.
- Zeng, C., *et al.* Two new polycyclic aromatic alkaloids from the Okinawan marine sponge *Biemna* sp. Tetrahedron Vol 49, 37 (1993) : 8337 - 8342.
- \_\_\_\_\_, Ishibashi, M., Kobayashi, J. Biemnasterol, A new cytotoxic sterol with the rare 22,25-diene side chain, isolated from the marine sponge *Biemna* sp. J. of Nat. Prod. Vol. 56, No.11 (1993) : pp. 2016 - 2018.

## **APPENDIX**

Long section



Top view

Figure 1. The photographs of Thai marine sponge, *Biemna fortis* (Topsent).

BIEMNA-F169 IN CDCL3 1H

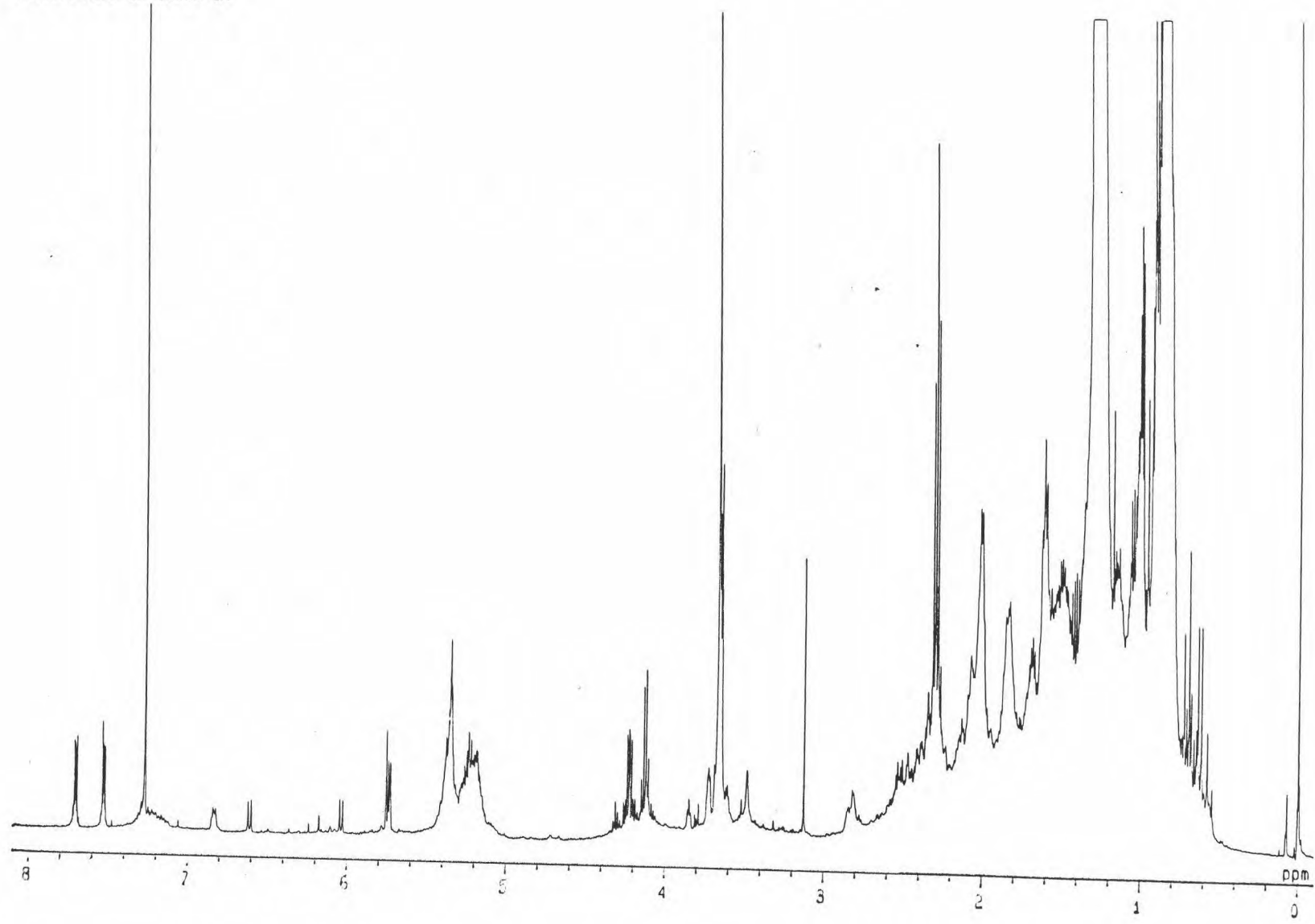


Figure 2. The 500 MHz  $^1\text{H}$  NMR spectrum of F169 in  $\text{CDCl}_3$



BIEMNA F183

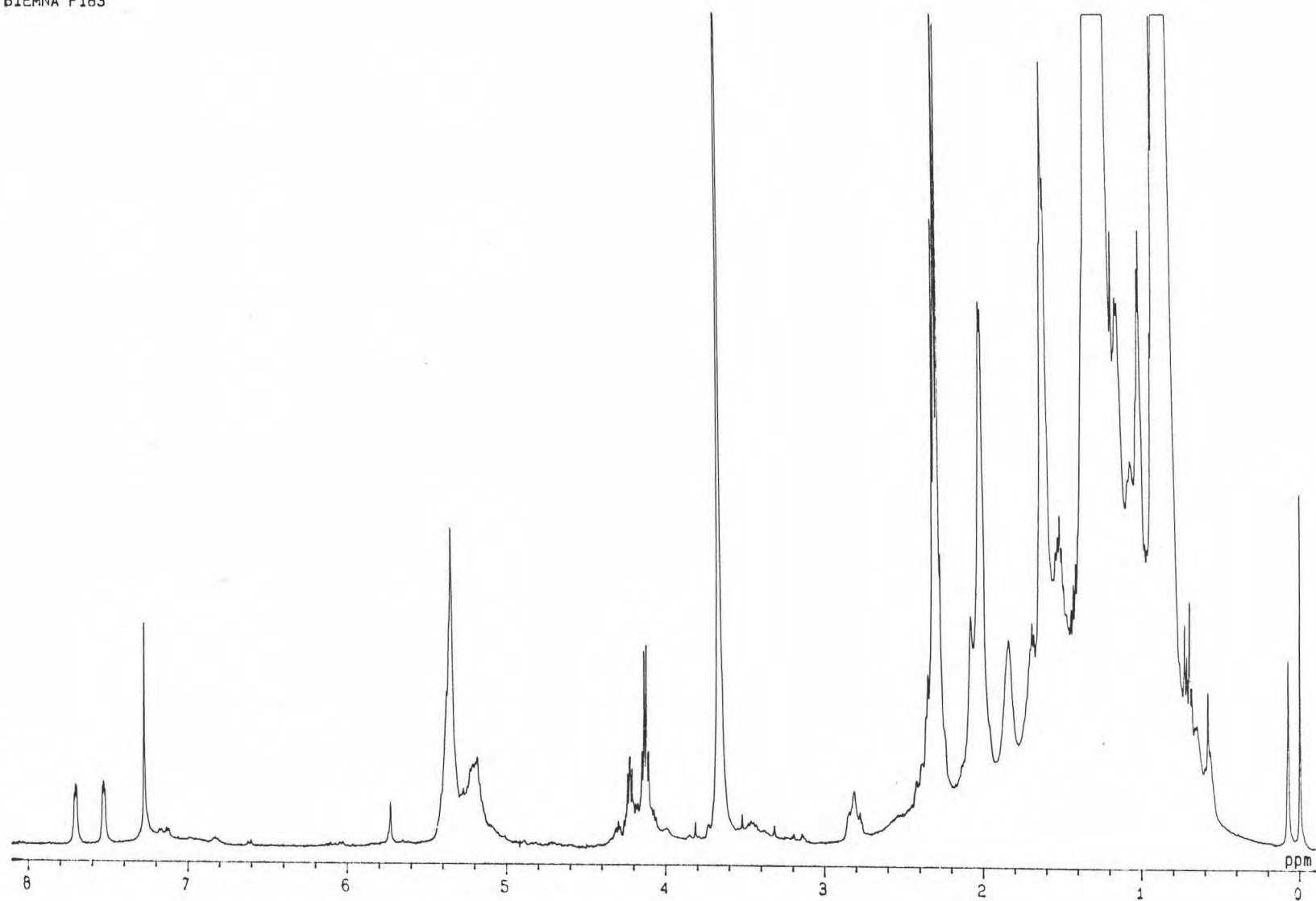


Figure 3. The 500 MHz  $^1\text{H}$  NMR spectrum of F183 in  $\text{CDCl}_3$

BIEMNA F184

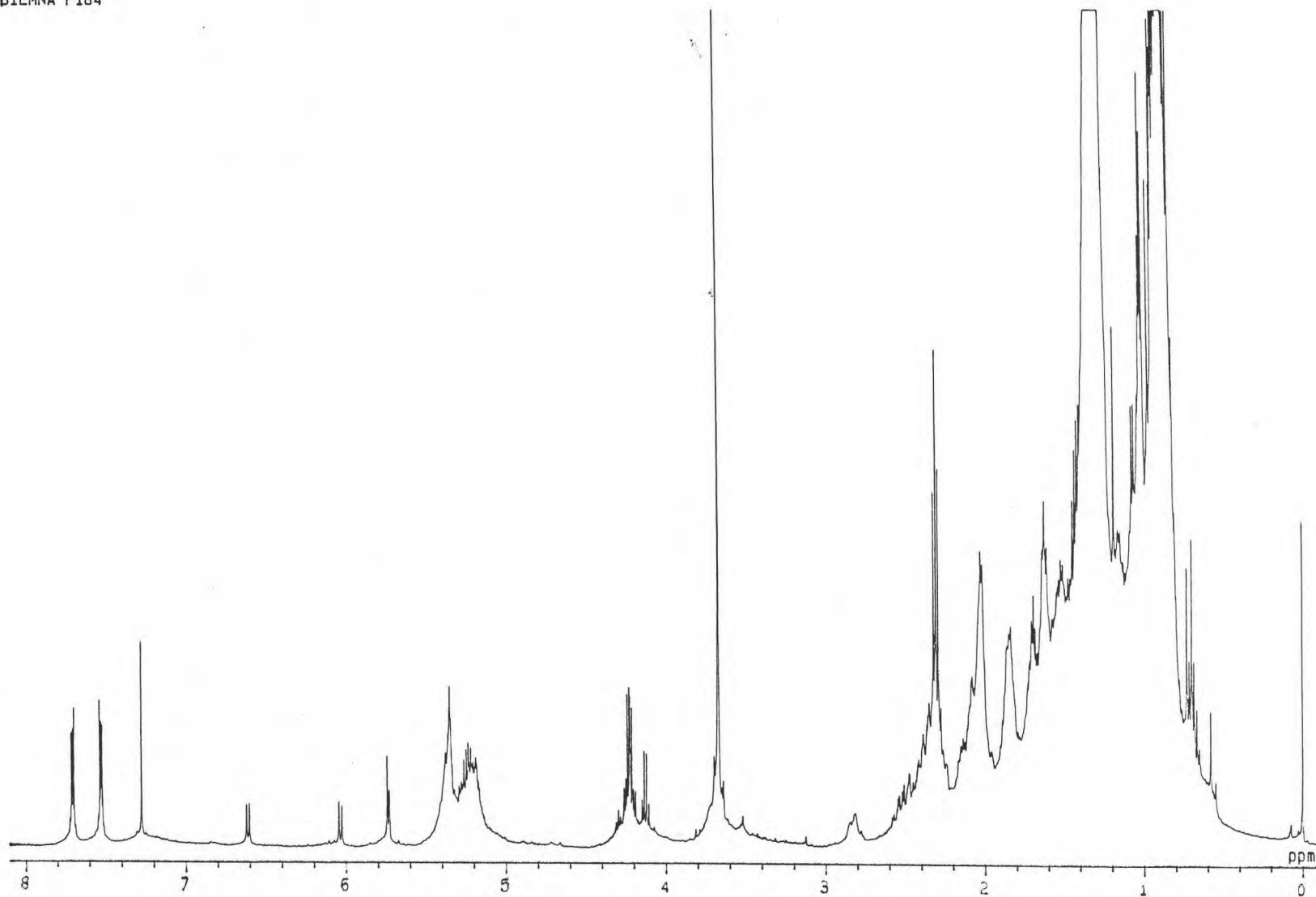


Figure 4. The 500 MHz  $^1\text{H}$  NMR spectrum of F184 in  $\text{CDCl}_3$

BIEMNA F187

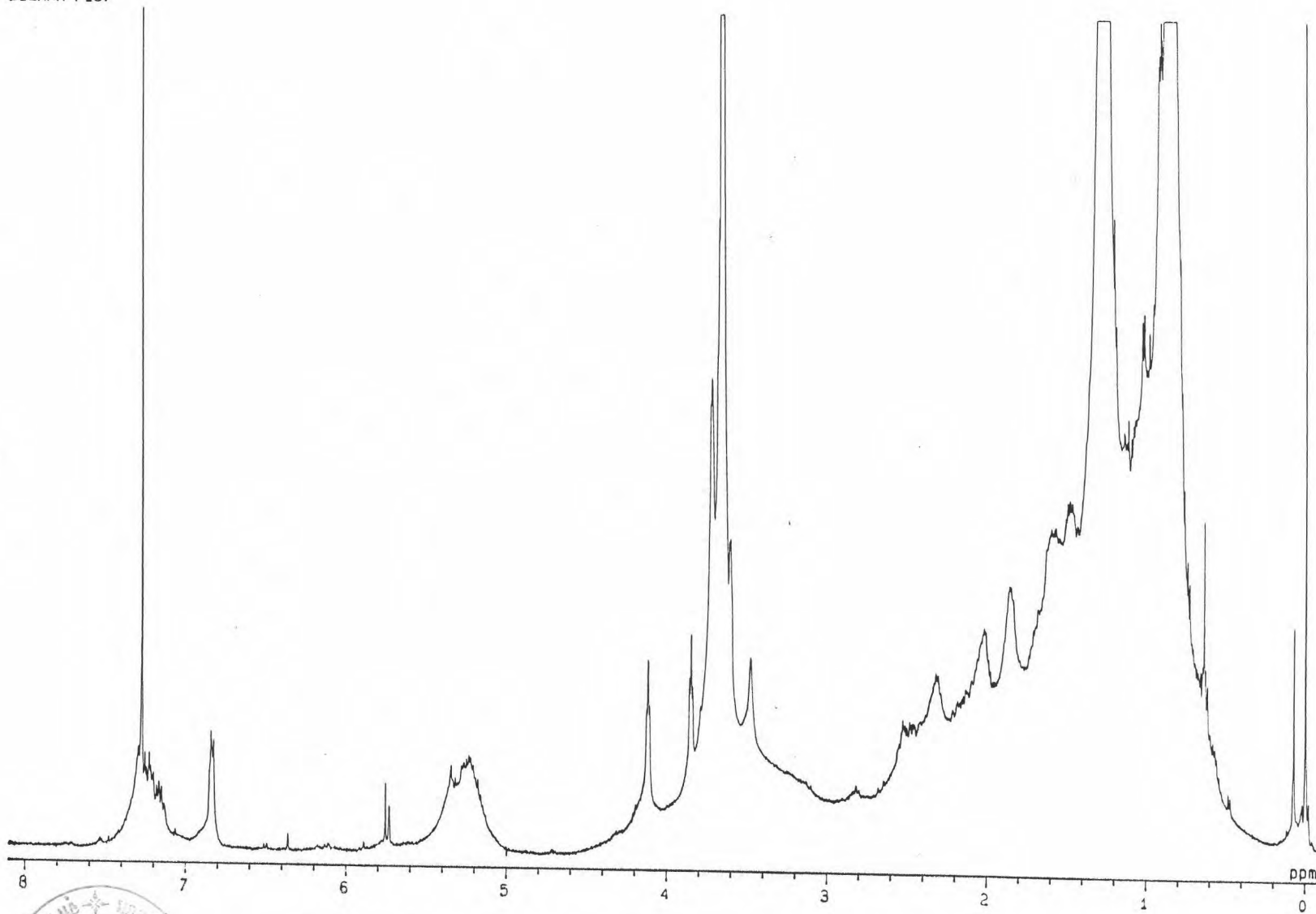


Figure 5. The 500 MHz  $^1\text{H}$  NMR spectrum of F187 in  $\text{CDCl}_3$



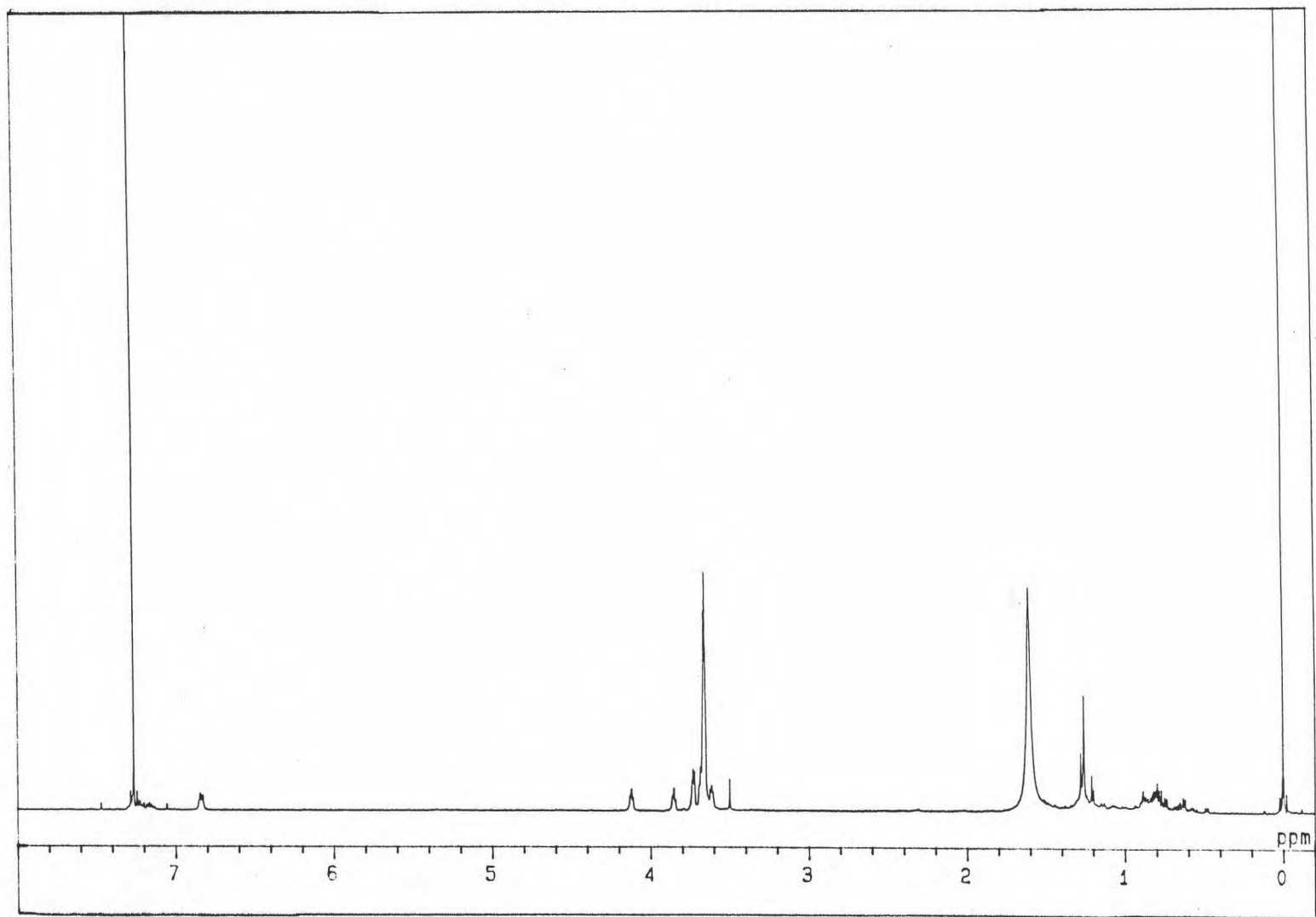


Figure 6. The 500 MHz <sup>1</sup>H NMR spectrum of F209 in CDCl<sub>3</sub>...

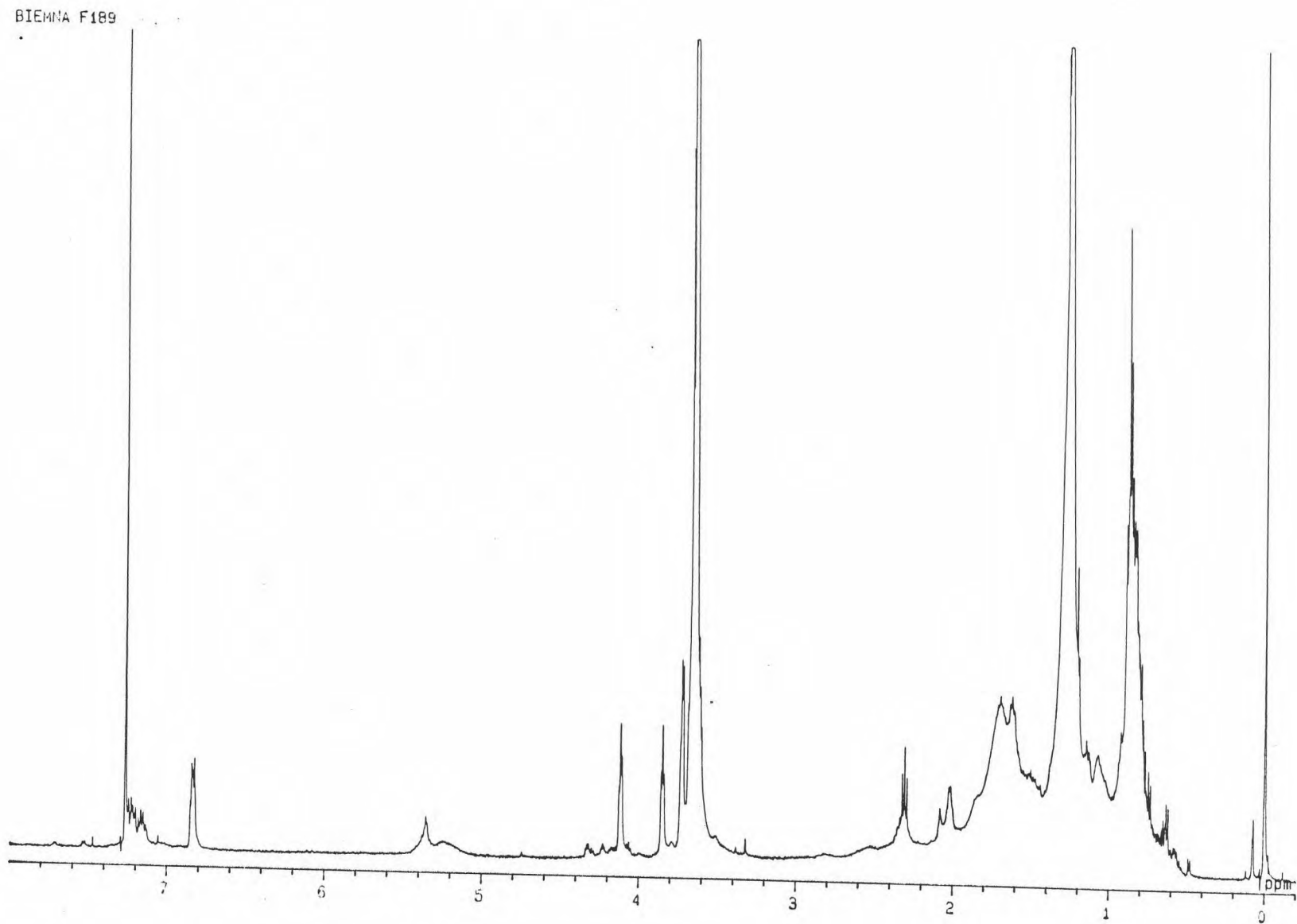


Figure 7. The 500 MHz  $^1\text{H}$  NMR spectrum of F189 in  $\text{CDCl}_3$

BIEMNA F190

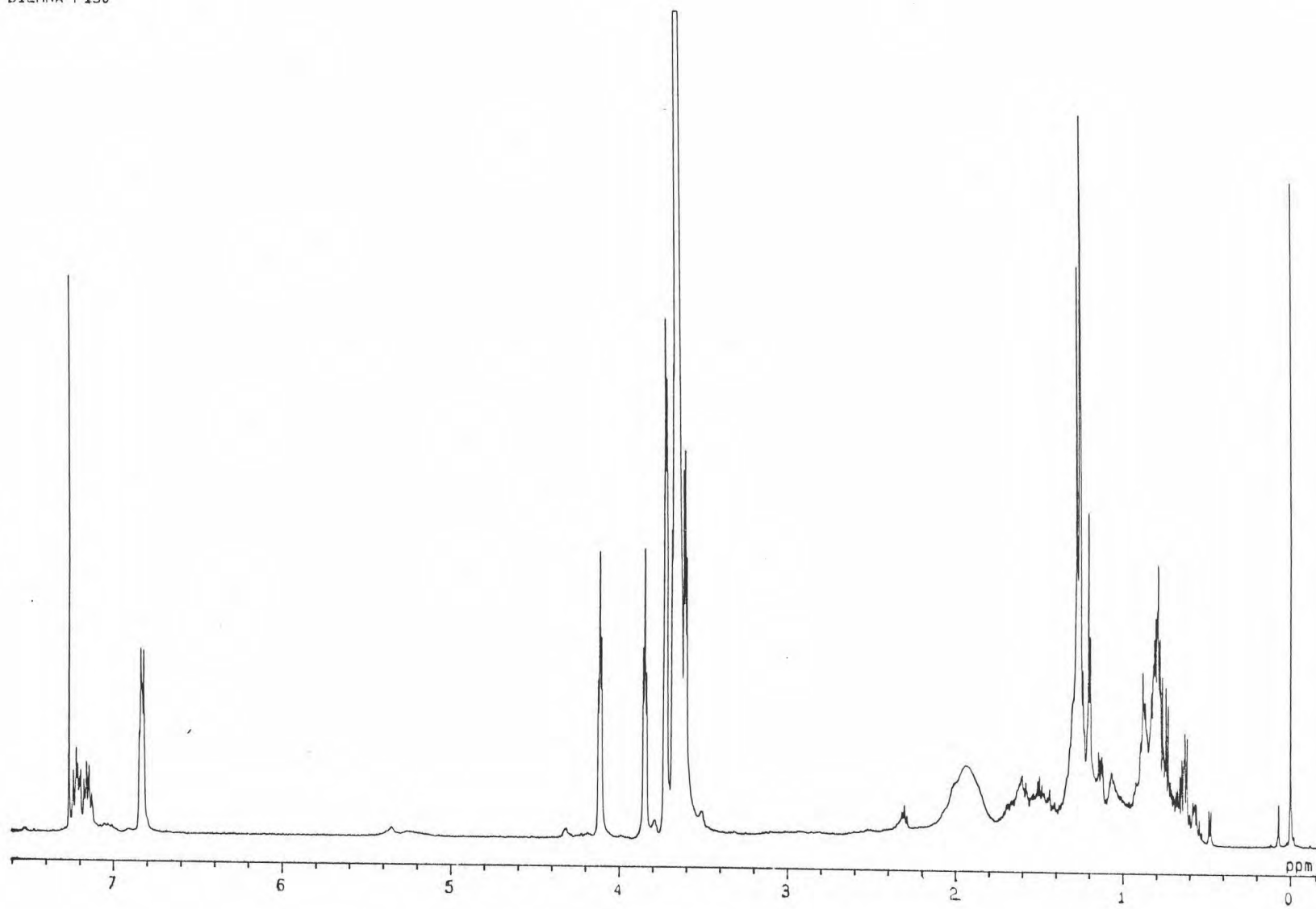


Figure 8. The 500 MHz  $^1\text{H}$  NMR spectrum of F190 in  $\text{CDCl}_3$

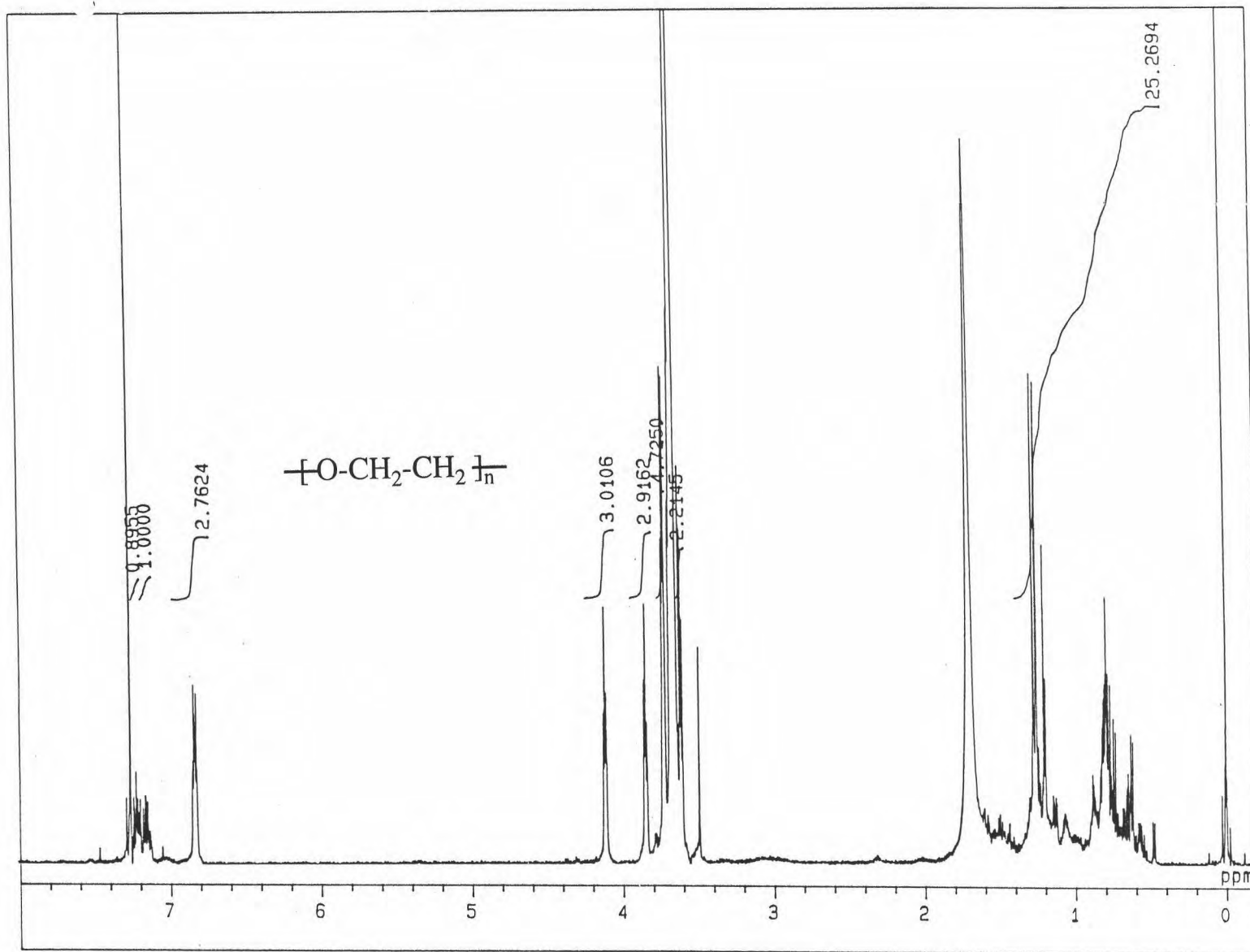


Figure 9. The 500 MHz  $^1\text{H}$  NMR spectrum of F201 in  $\text{CDCl}_3$ .



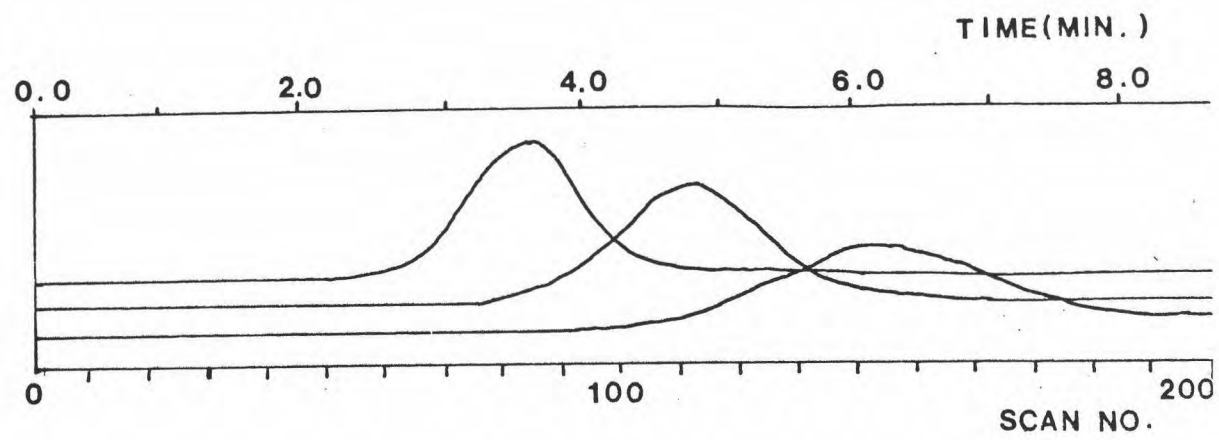


Figure 10. The FAB MS chromatogram of F201

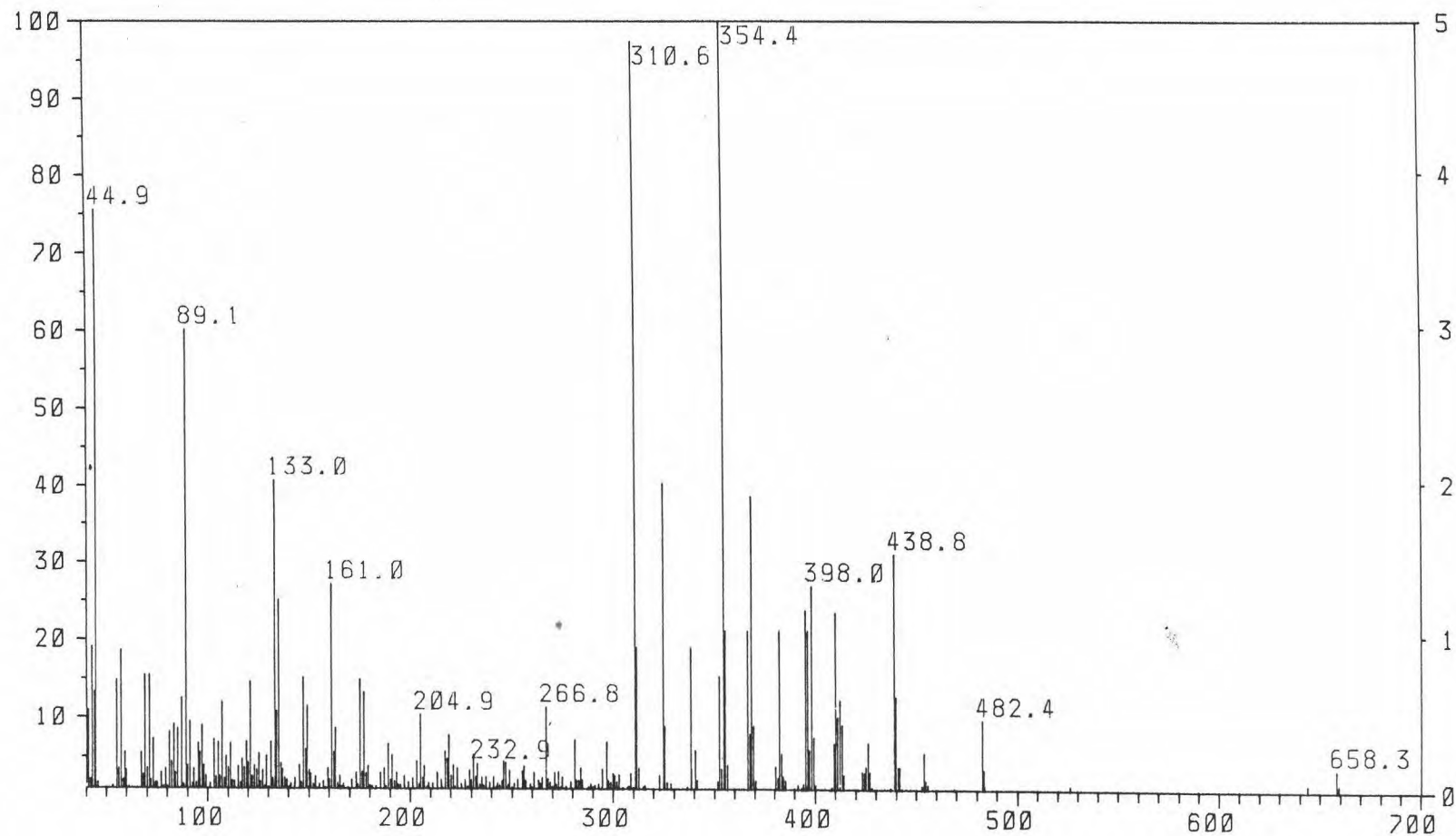


Figure 11. The FAB MS spectrum of the first main peak of F201  
(Scan No.: 70x79, Time 3.0 min)

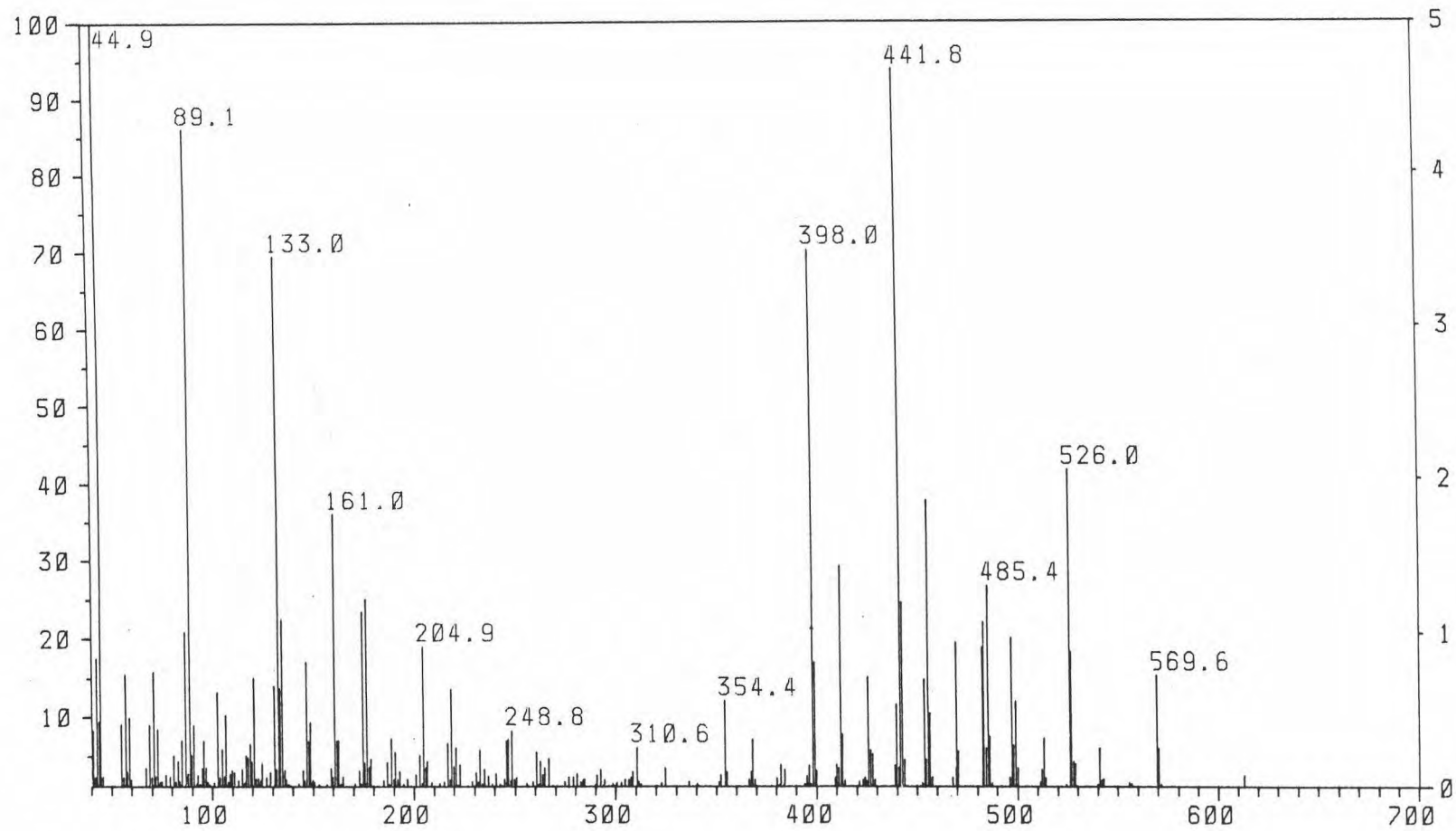


Figure 12. The FAB MS spectrum of the second main peak of F201 (Scan No.: 103x115, Time 4.4 min)

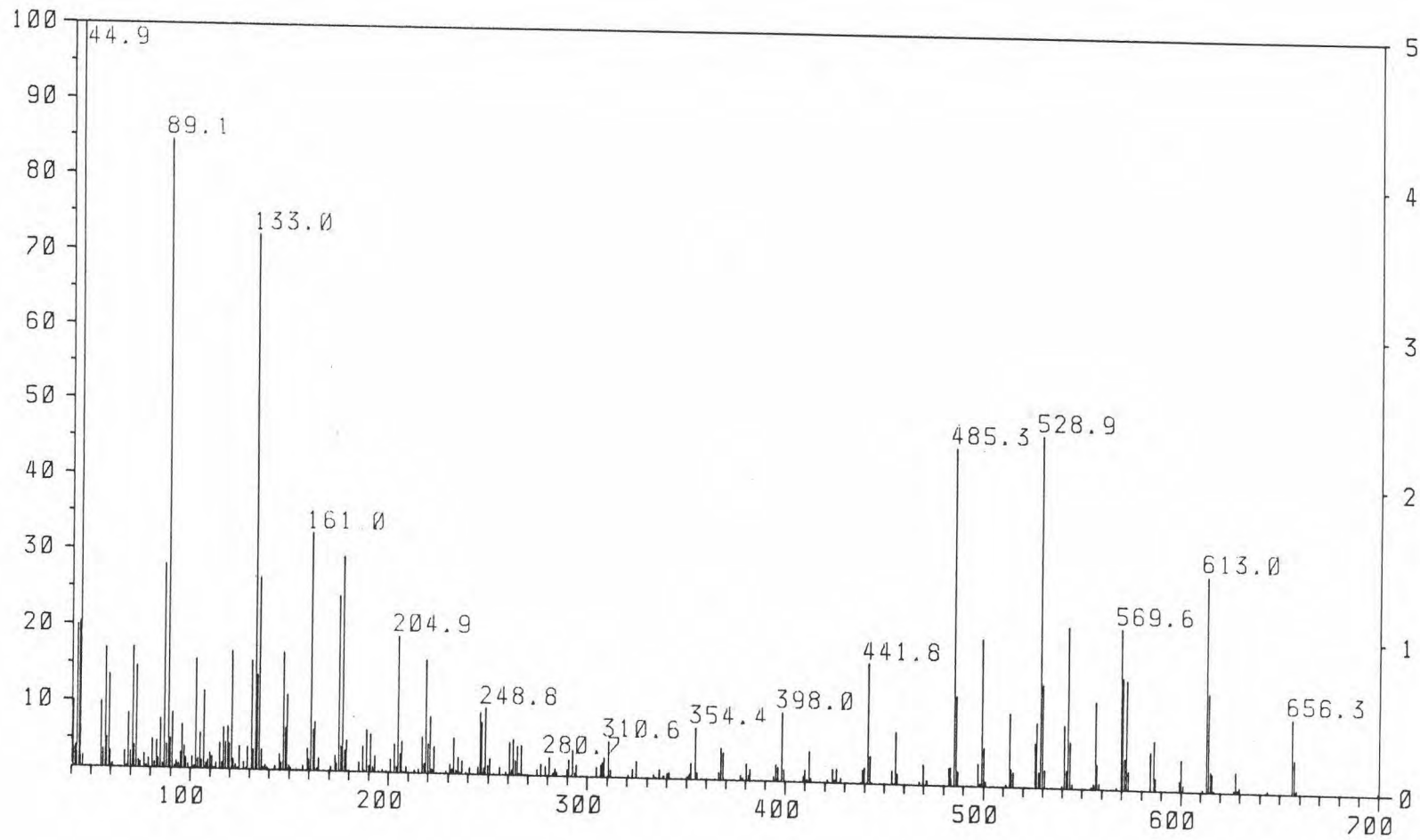
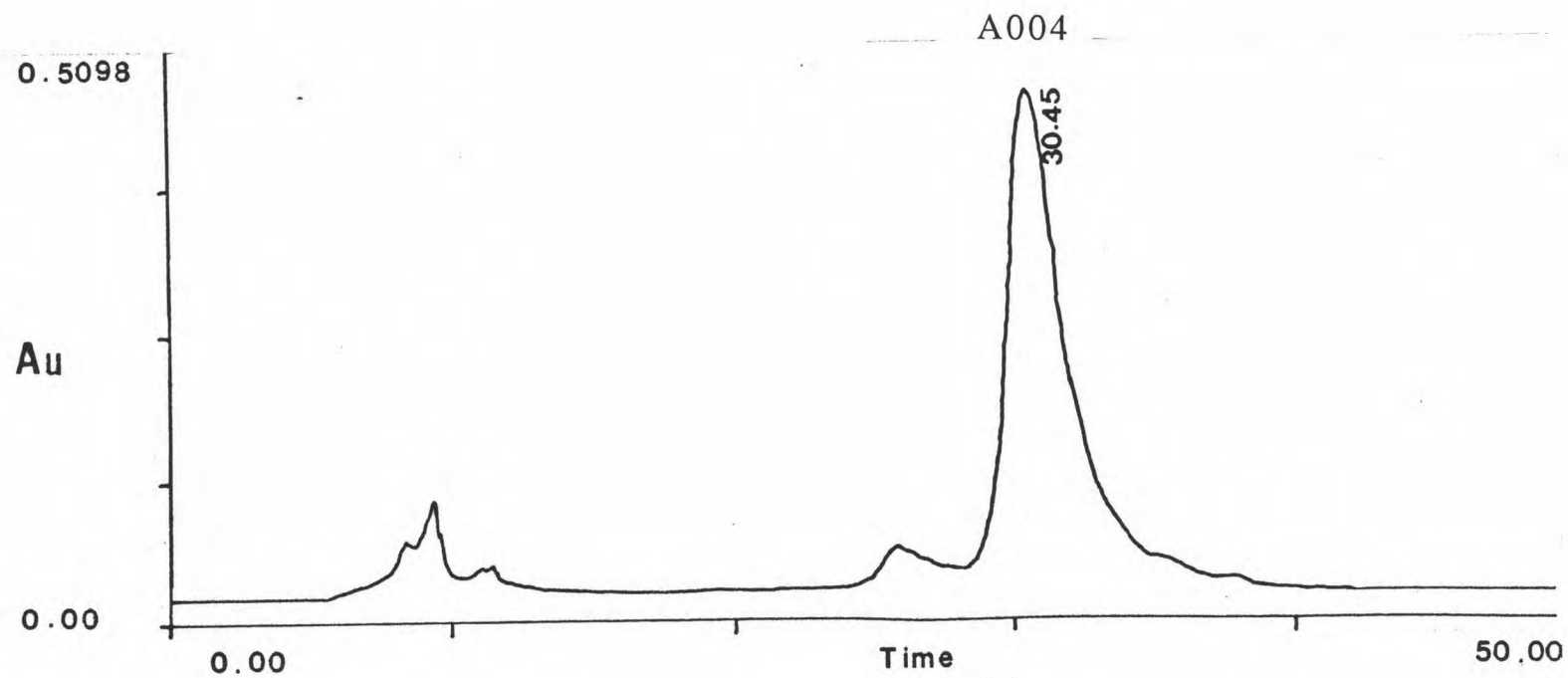


Figure 13. The FAB MS spectrum of the third main peak of F201 (Scan No.: 141x171, Time 6.1 min)



Reversed phase RP-2 column (250 x 4 mm)  
Flow rate 1 ml/min  
detected at  $\lambda_{\max}$  233 nm

Figure 14. The HPLC chromatogram of F201 (15% H<sub>2</sub>O in CH<sub>3</sub>OH)

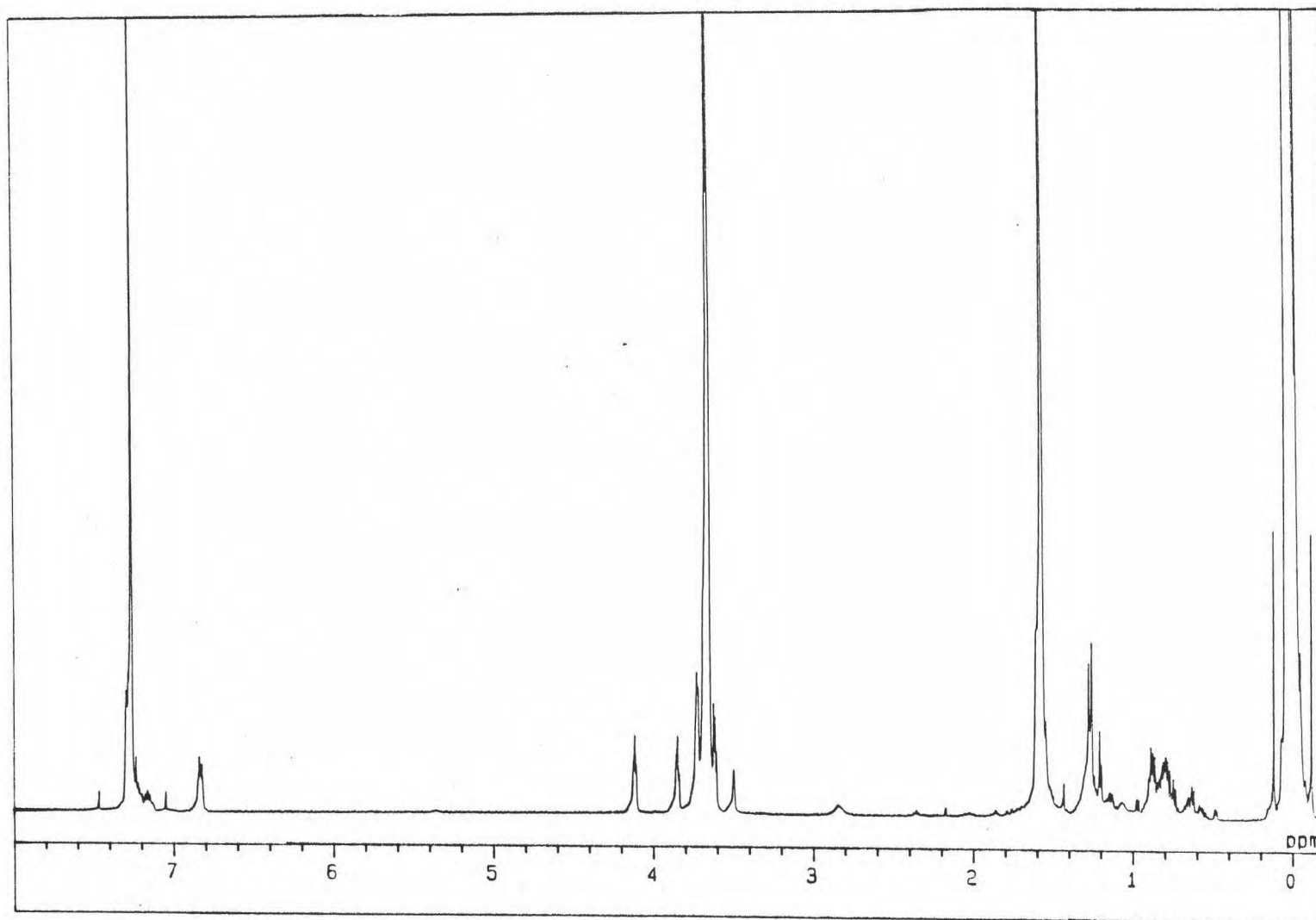


Figure 15. The 500 MHz  $^1\text{H}$  NMR spectrum of A004. in  $\text{CDCl}_3$

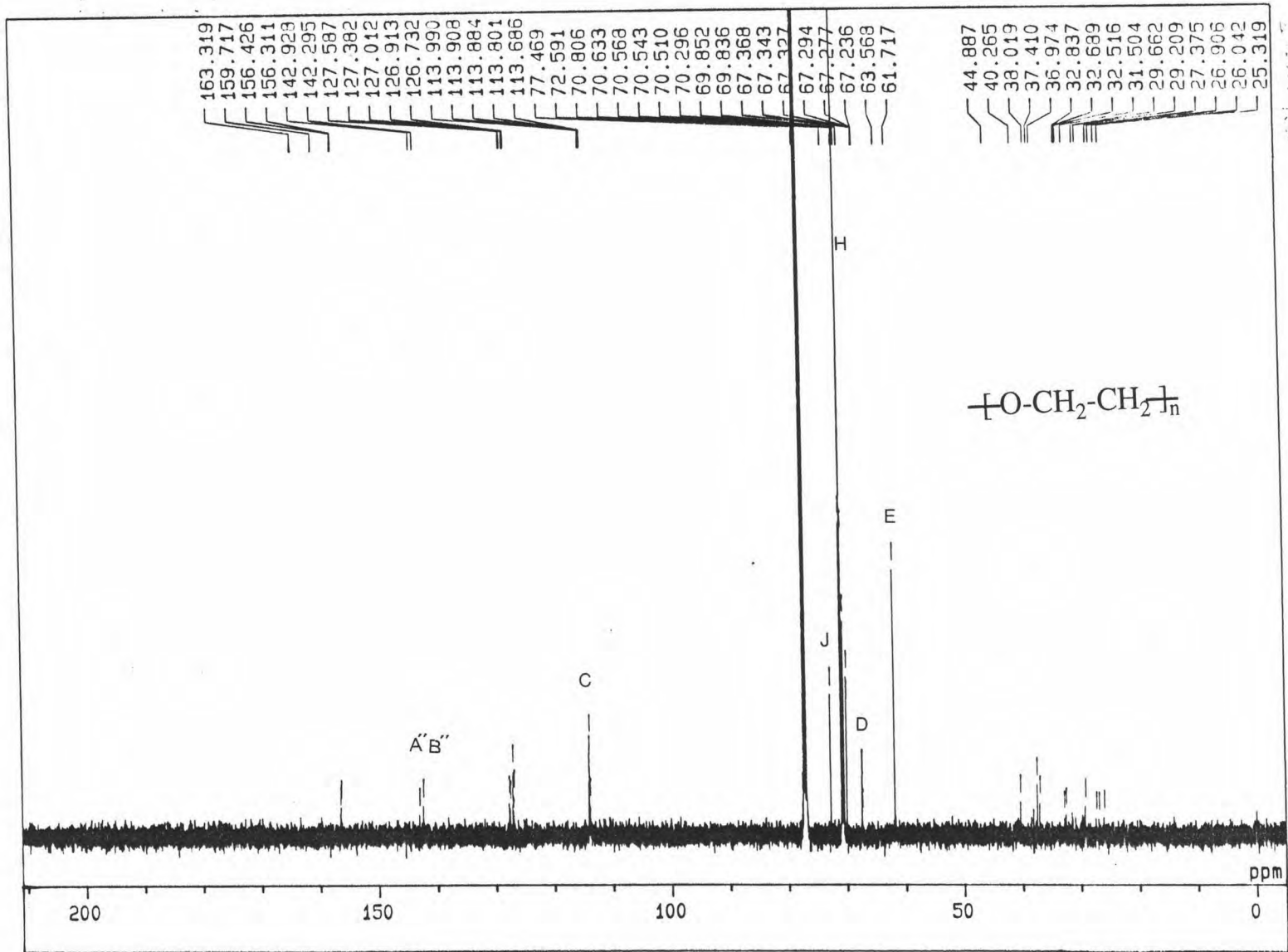


Figure 16. The 125 MHz  $^{13}\text{C}$  NMR spectrum of F201 in  $\text{CDCl}_3$ .



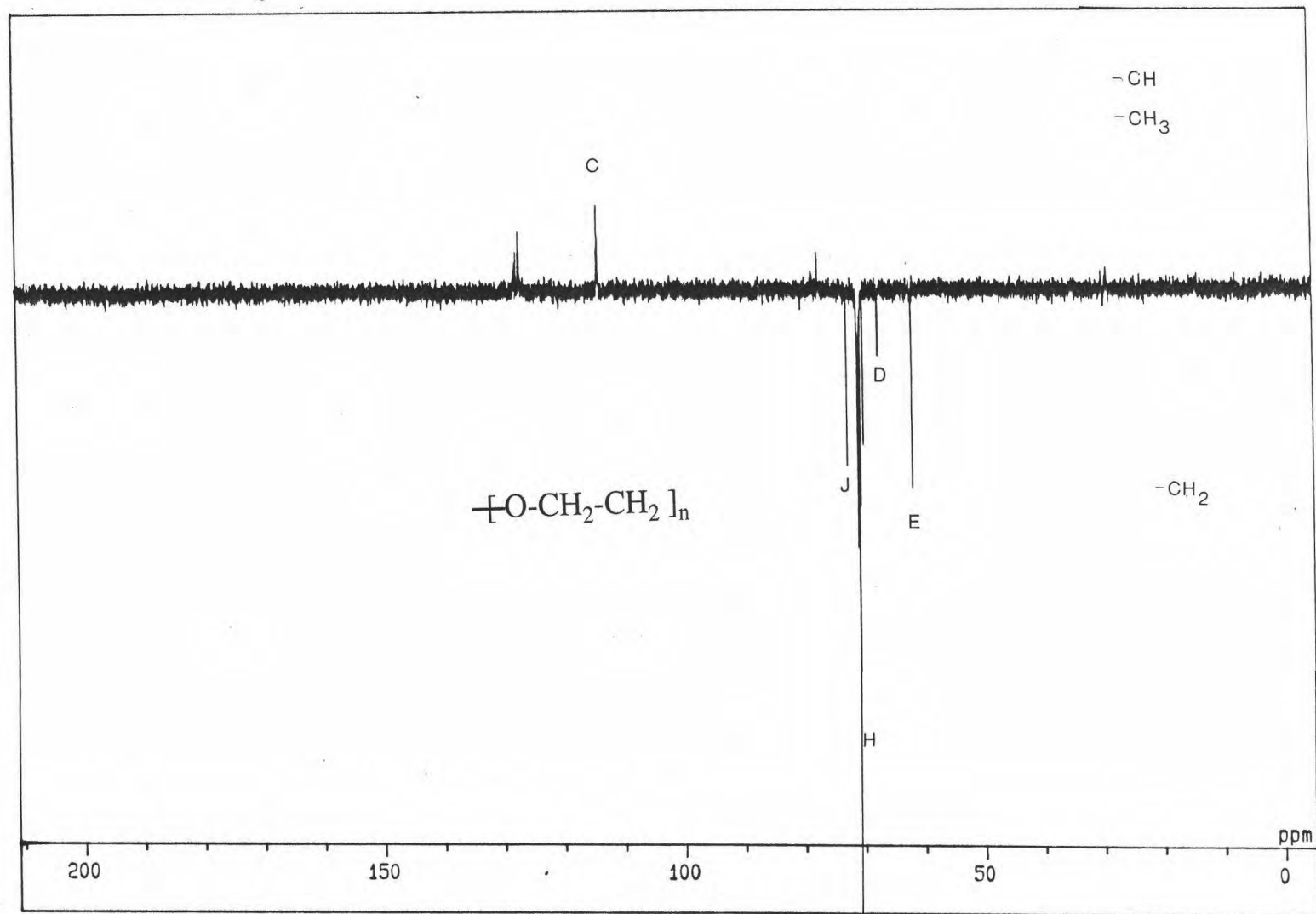


Figure 17. The DEPT 135° spectrum of F201

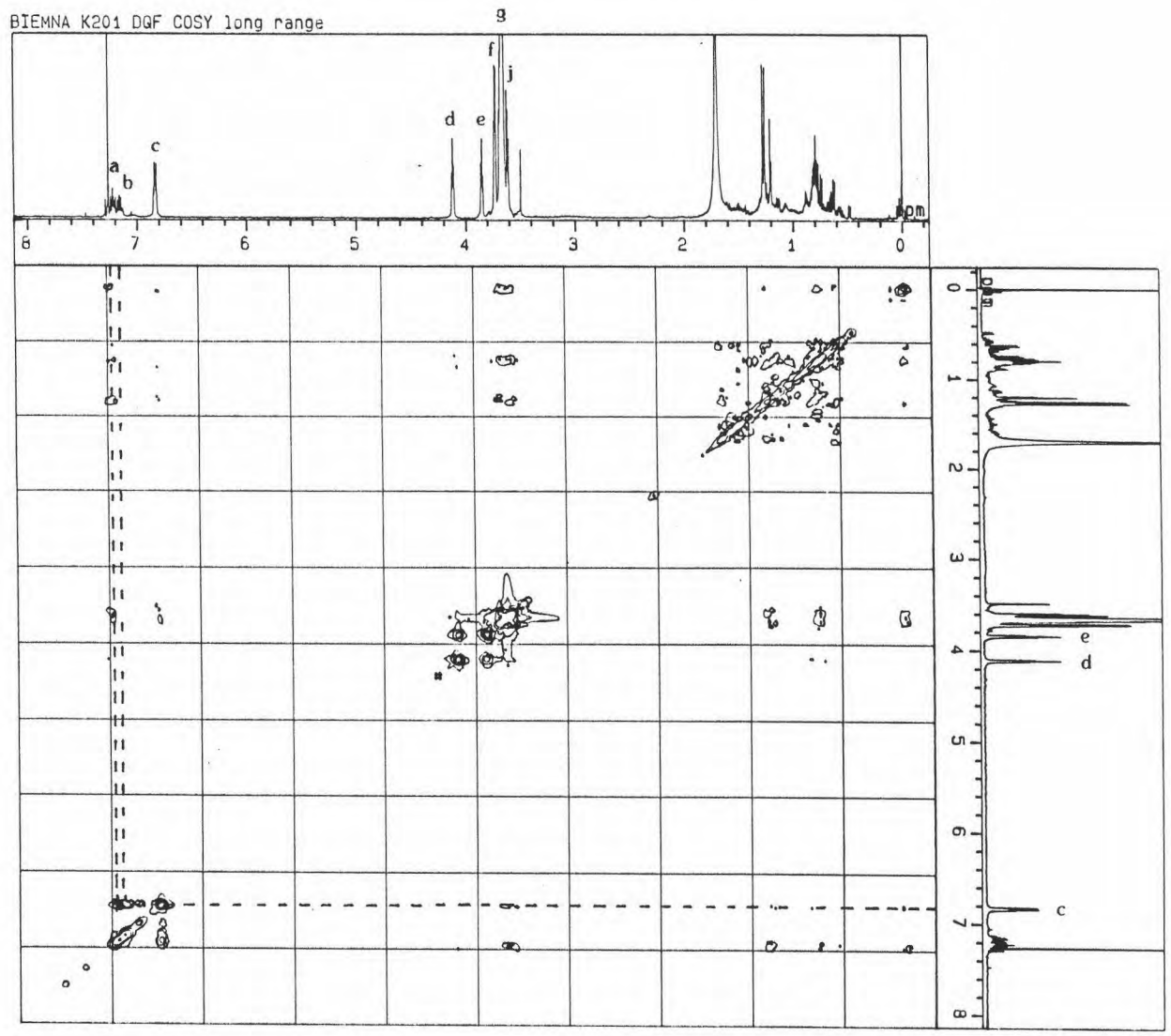


Figure 18. The DQF long range spectrum of F201

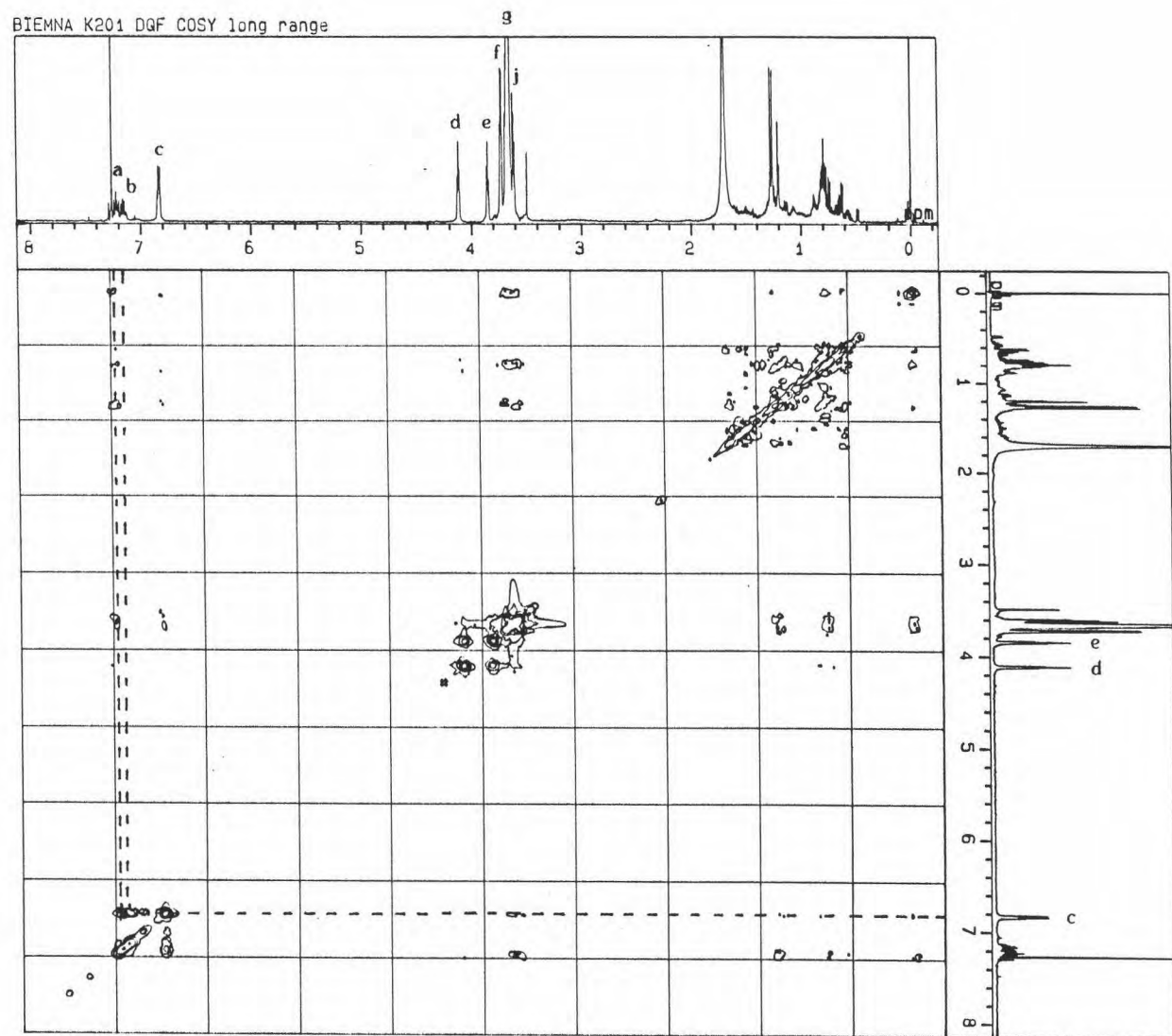


Figure 18. The DQF long range spectrum of F201

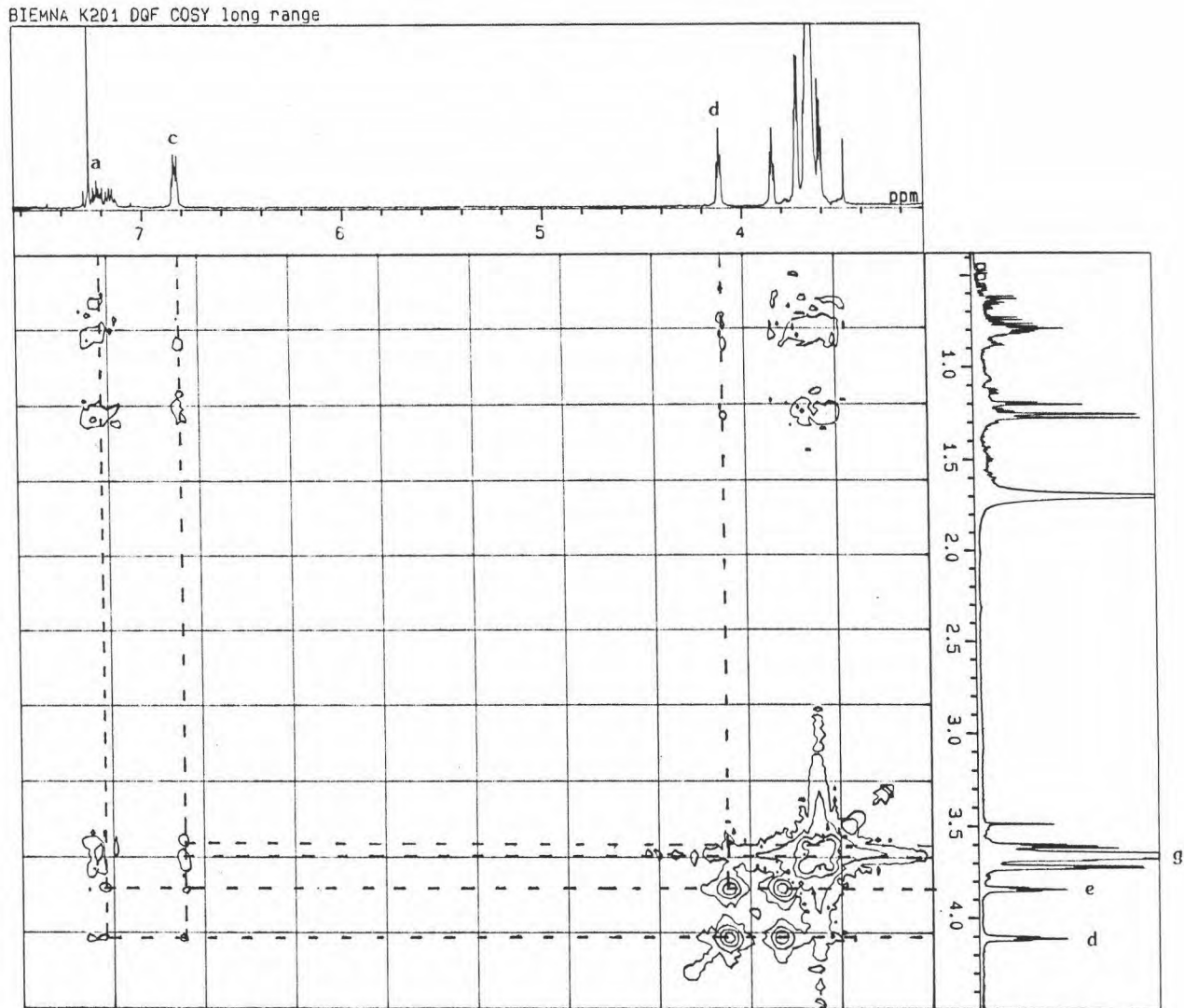


Figure 19. The partial DQF long range spectrum of F201

BIEMNA K201 HOHAHA mixing time 60 msec

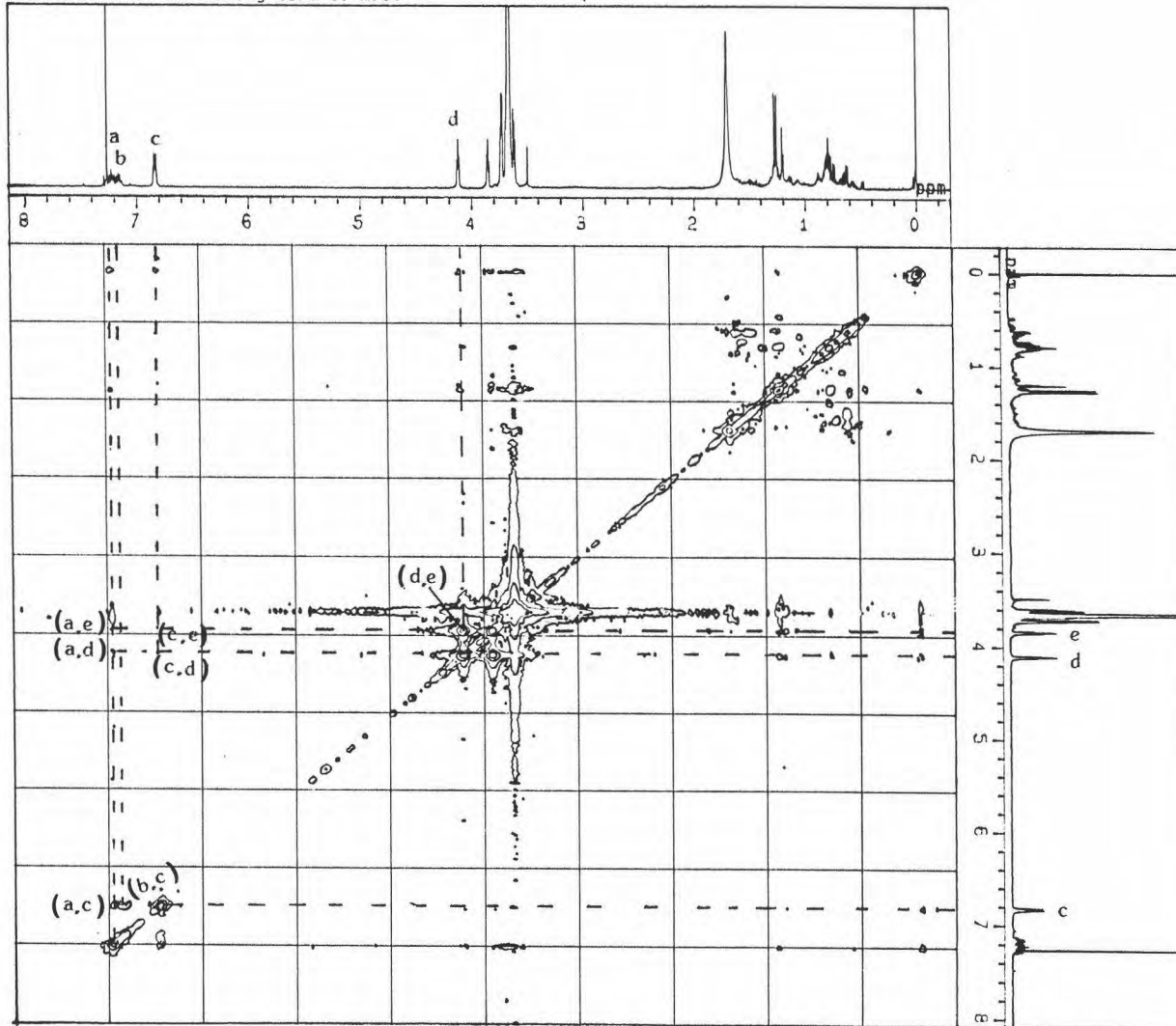


Figure 20. The HOHAHA spectrum of F201

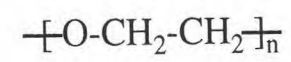
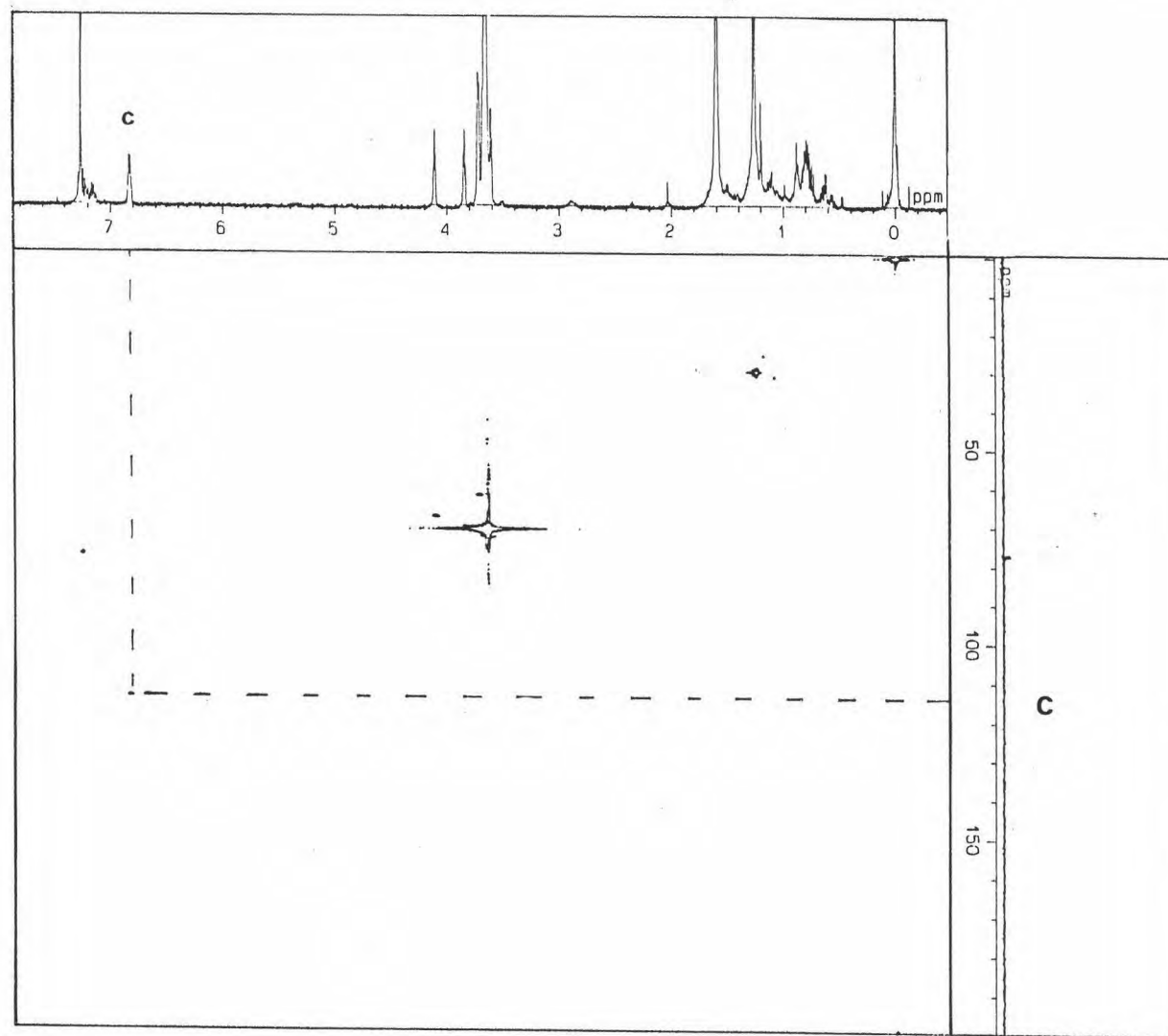


Figure 21. The HMQC spectrum of F201

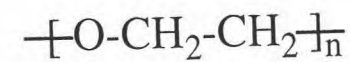
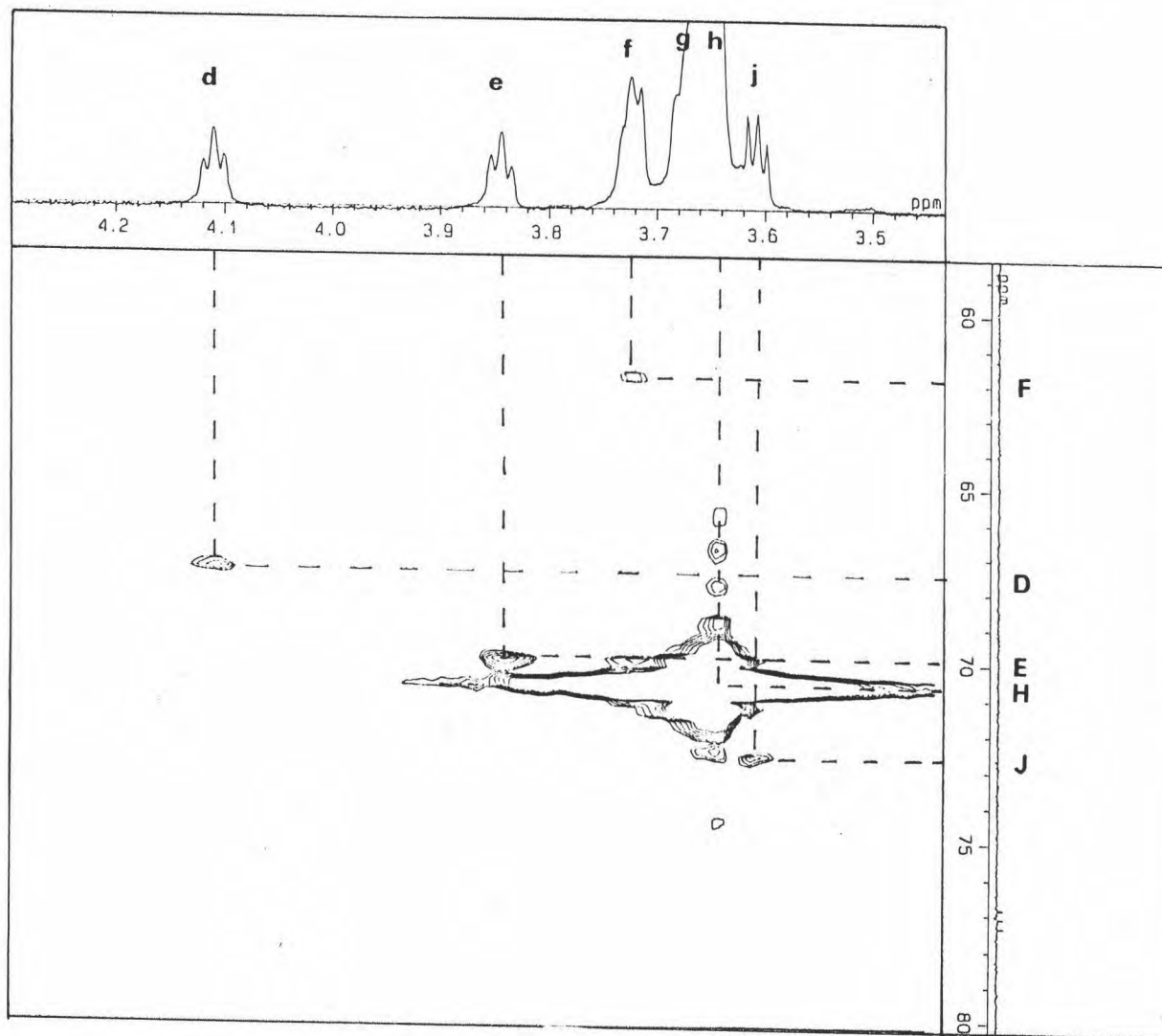


Figure 22. The HMQC spectrum of F201



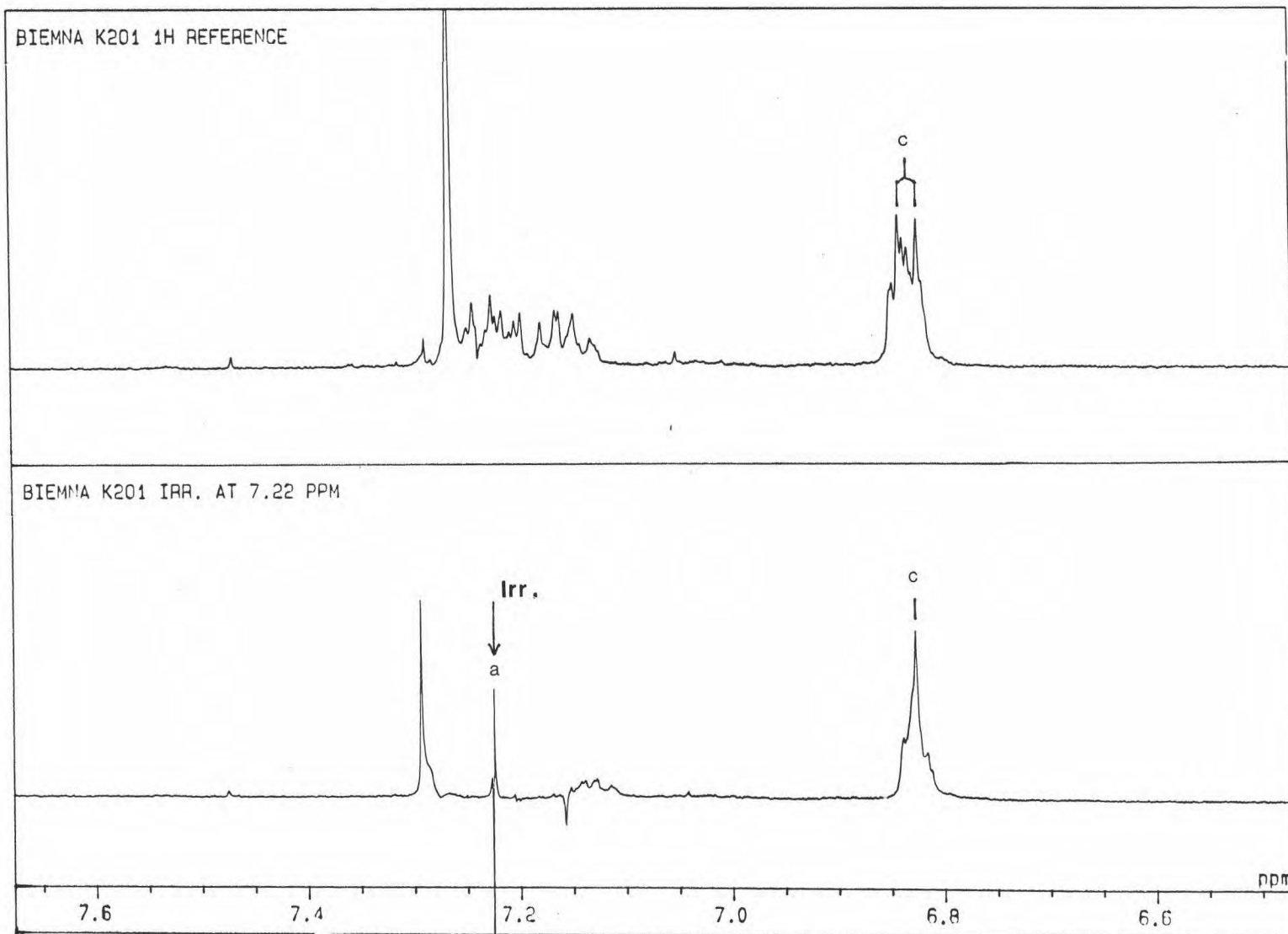


Figure 23. The Decoupling experiment spectrum of F201 (Ha)

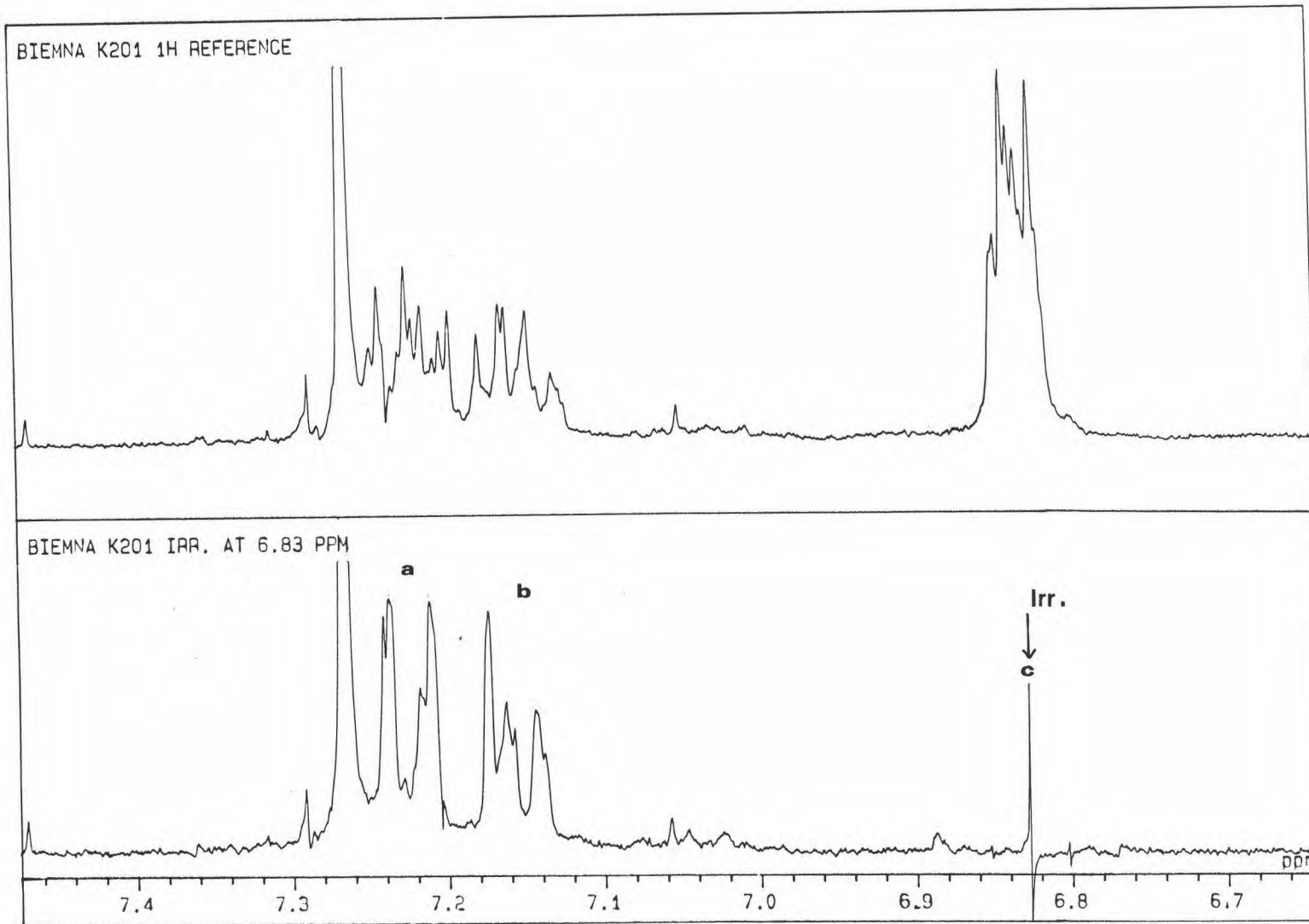


Figure 24. The Decoupling experiment spectrum of F201 (Hc)

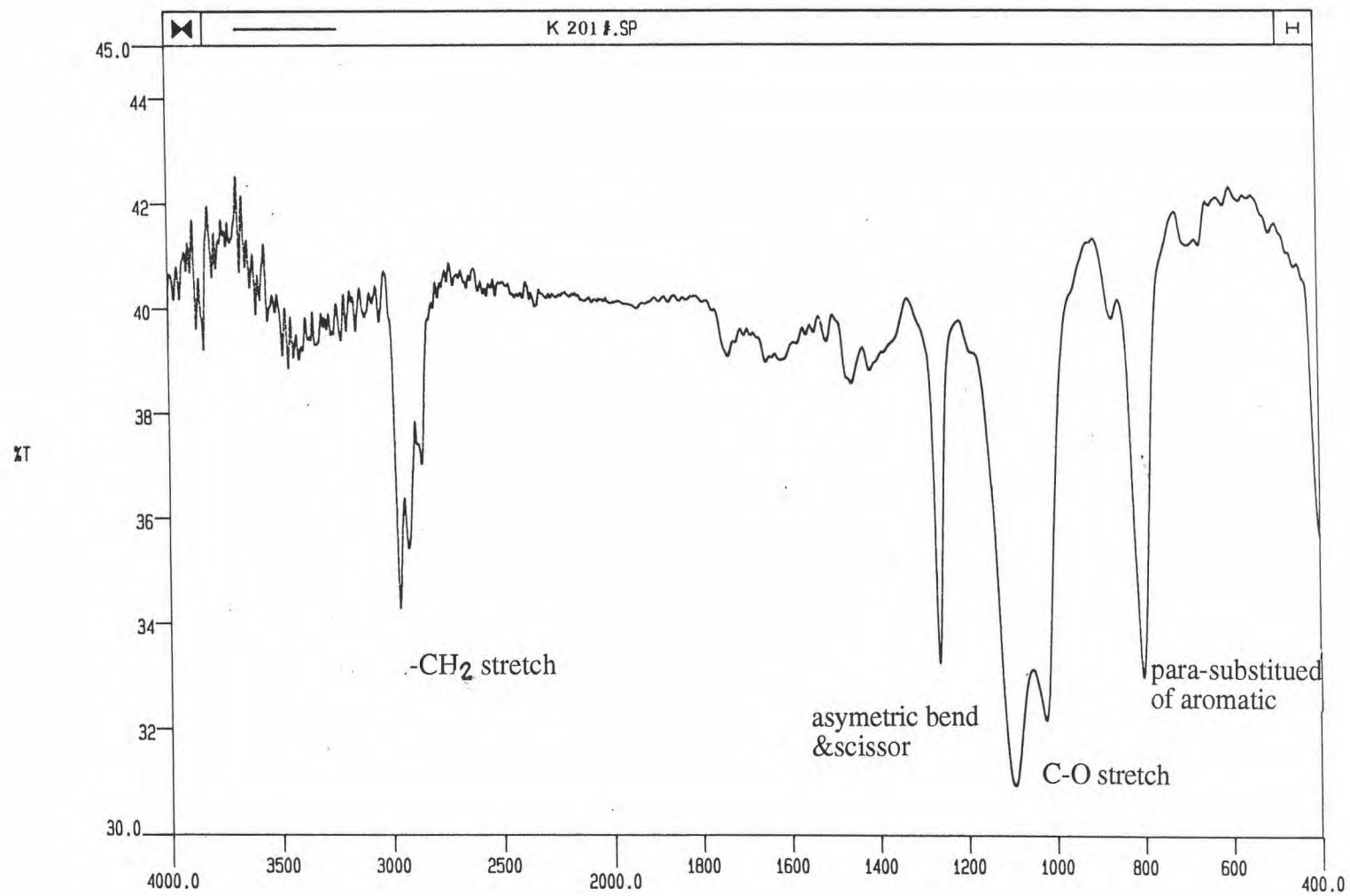


Figure 25. The IR spectrum of F201 (KBr disc)

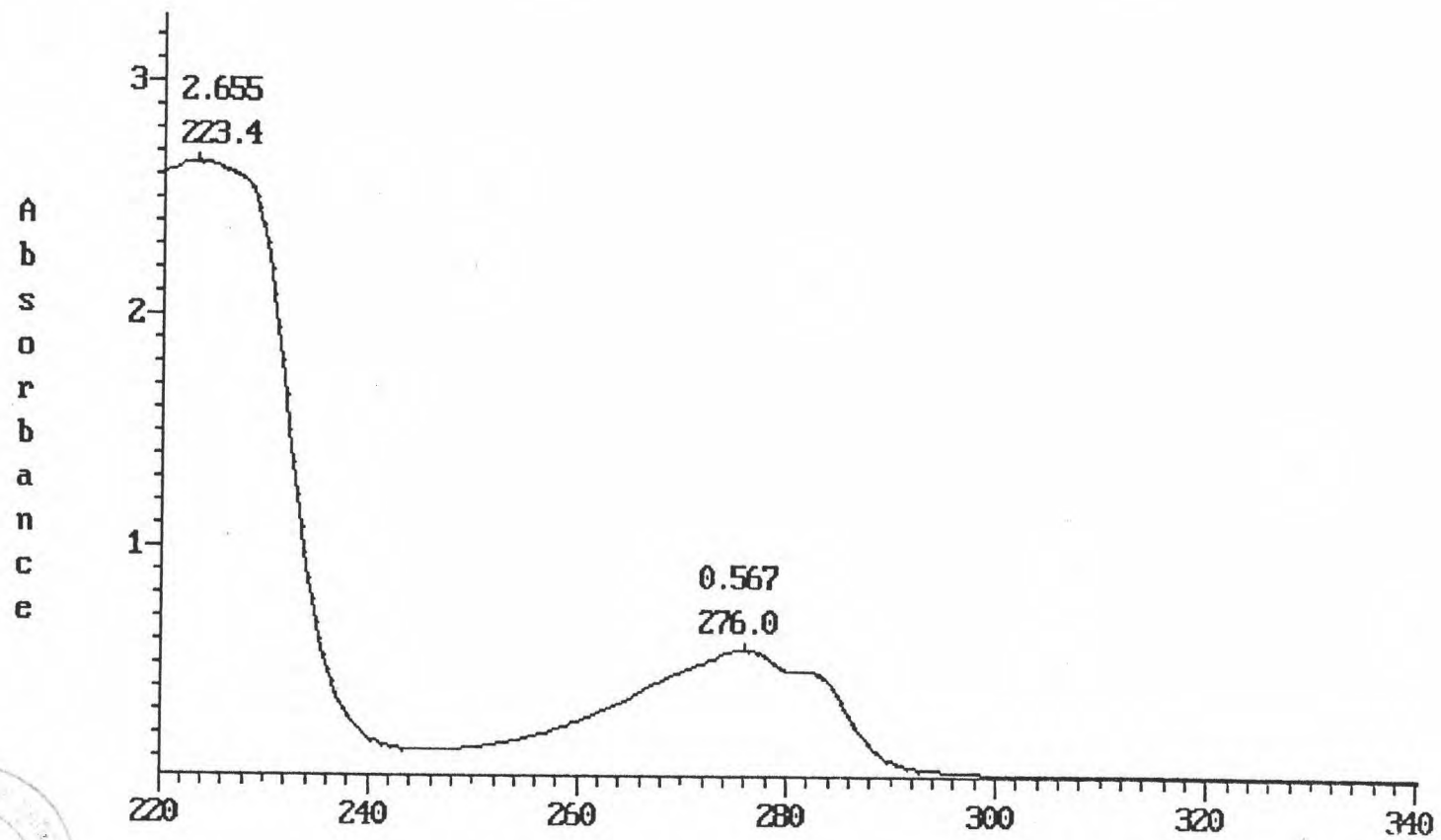


Figure 26. The UV spectrum of F201 in chloroform



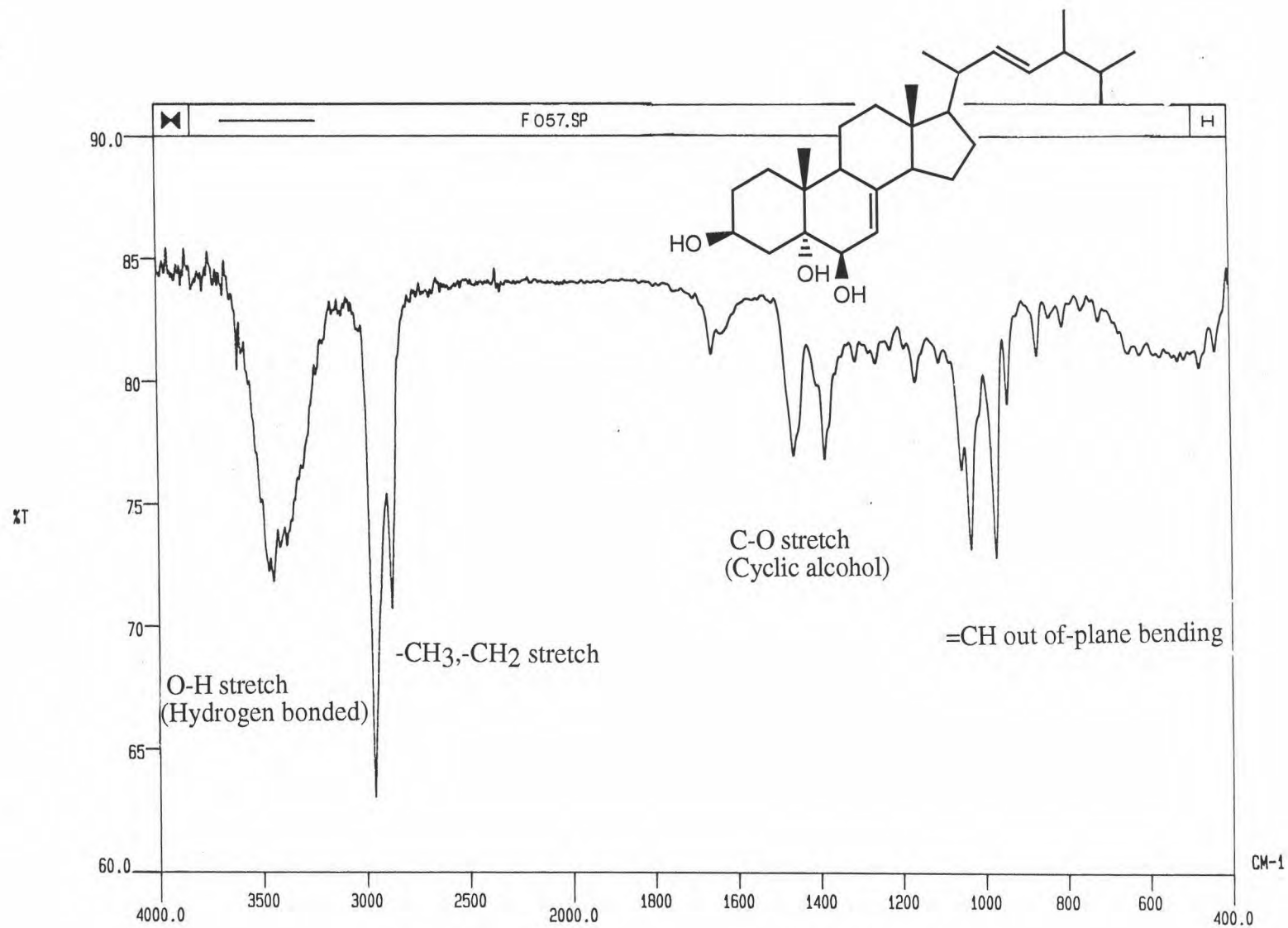


Figure 27. The IR spectrum of K057 (KBr disc)

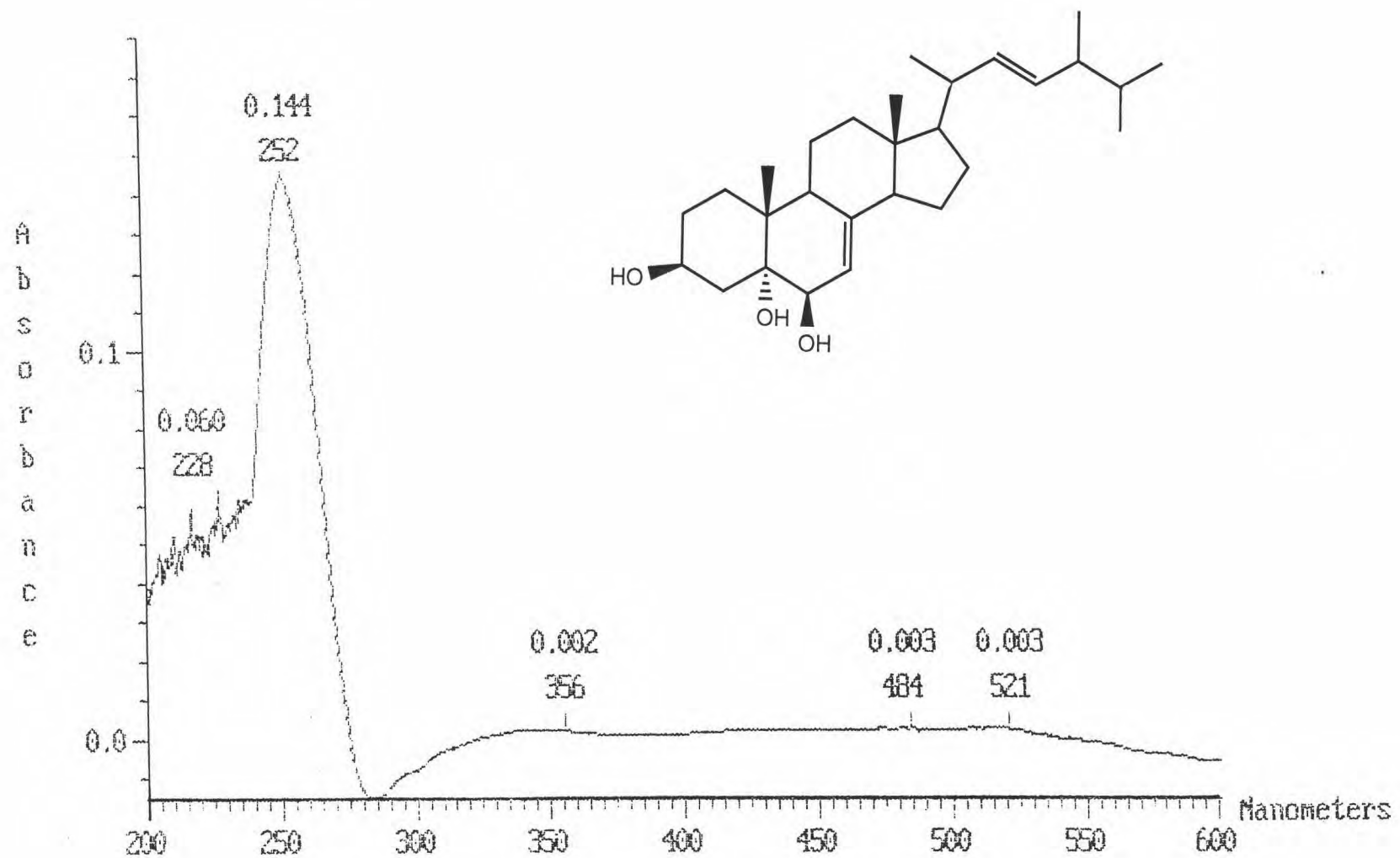


Figure 28. The UV spectrum of K057 in chloroform

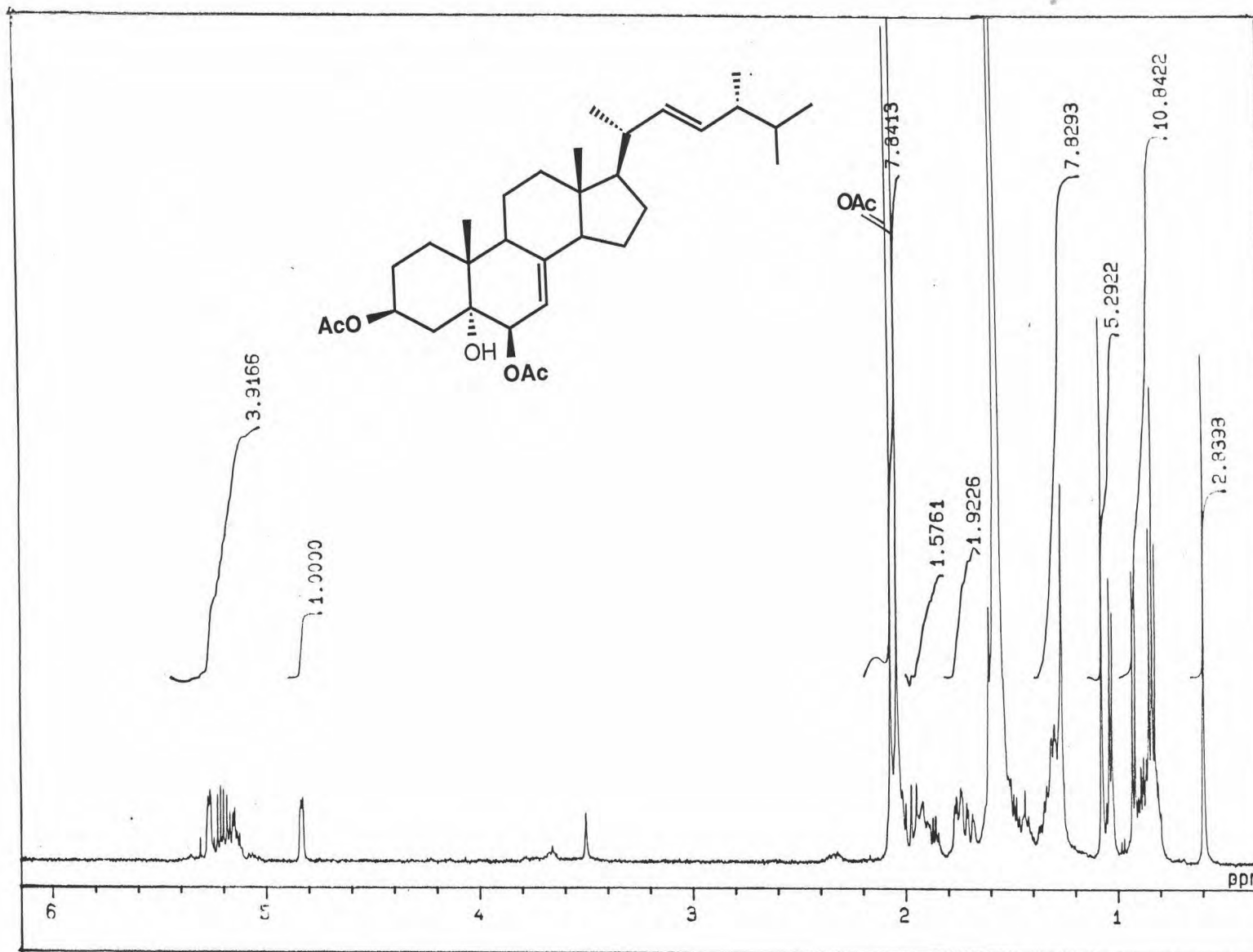


Figure 29. The 500 MHz <sup>1</sup>H NMR spectrum of K057 (Acetate) in 30% CD<sub>3</sub>OD in CDCl<sub>3</sub>

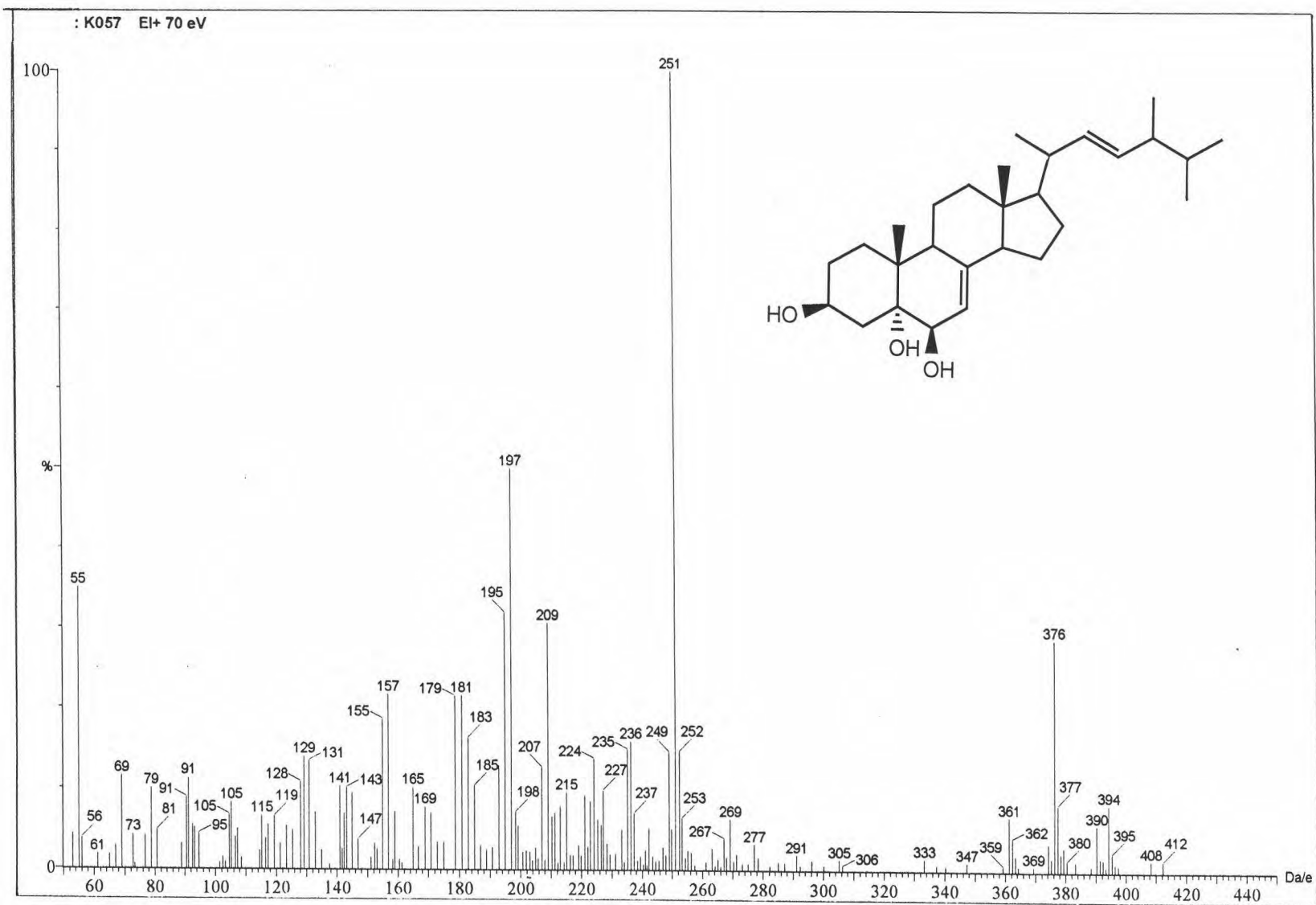


Figure 30. The EIMS spectrum of K057



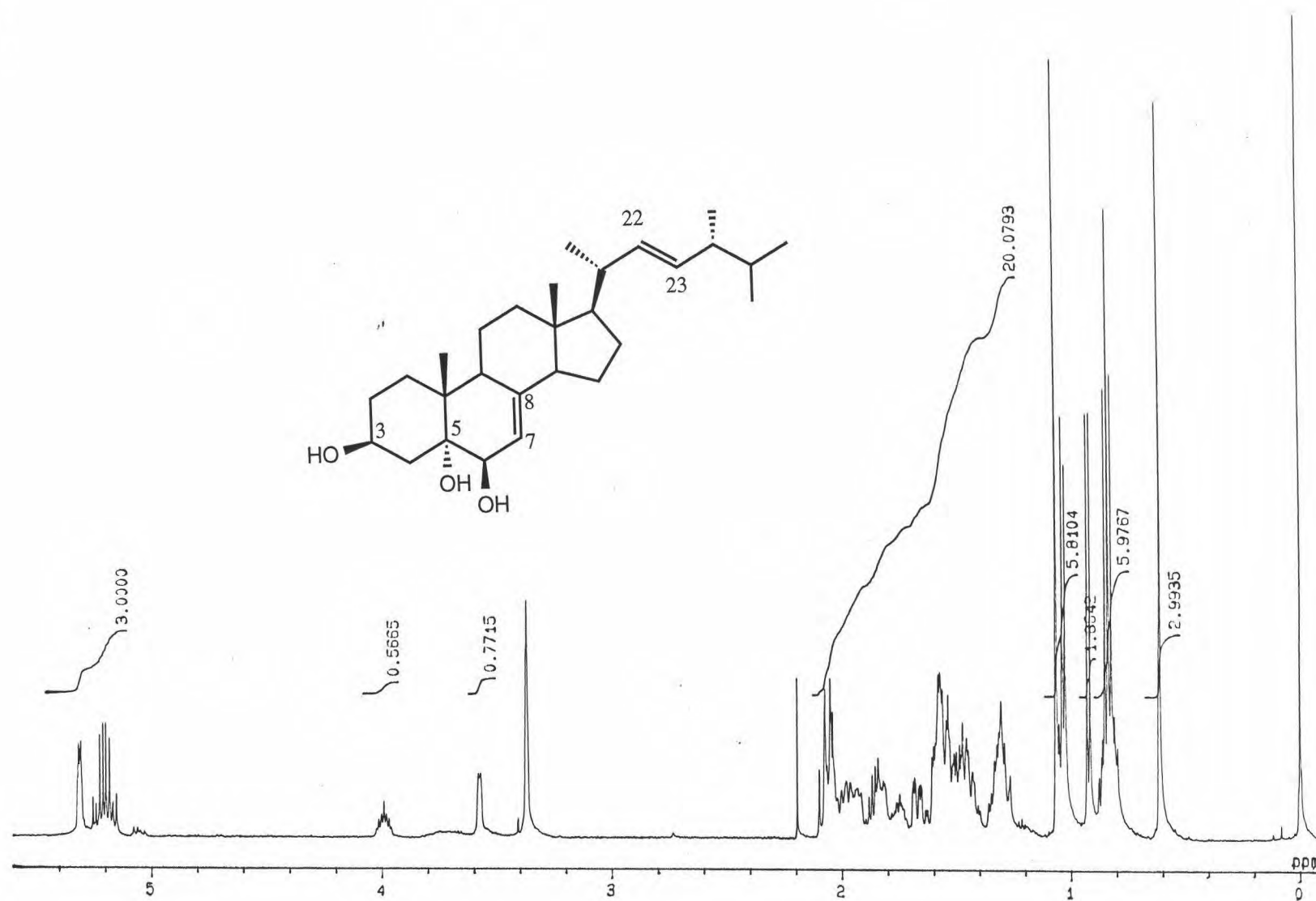


Figure 31. The 500 MHz <sup>1</sup>H NMR spectrum of K057 in 30% CD<sub>3</sub>OD in CDCl<sub>3</sub>

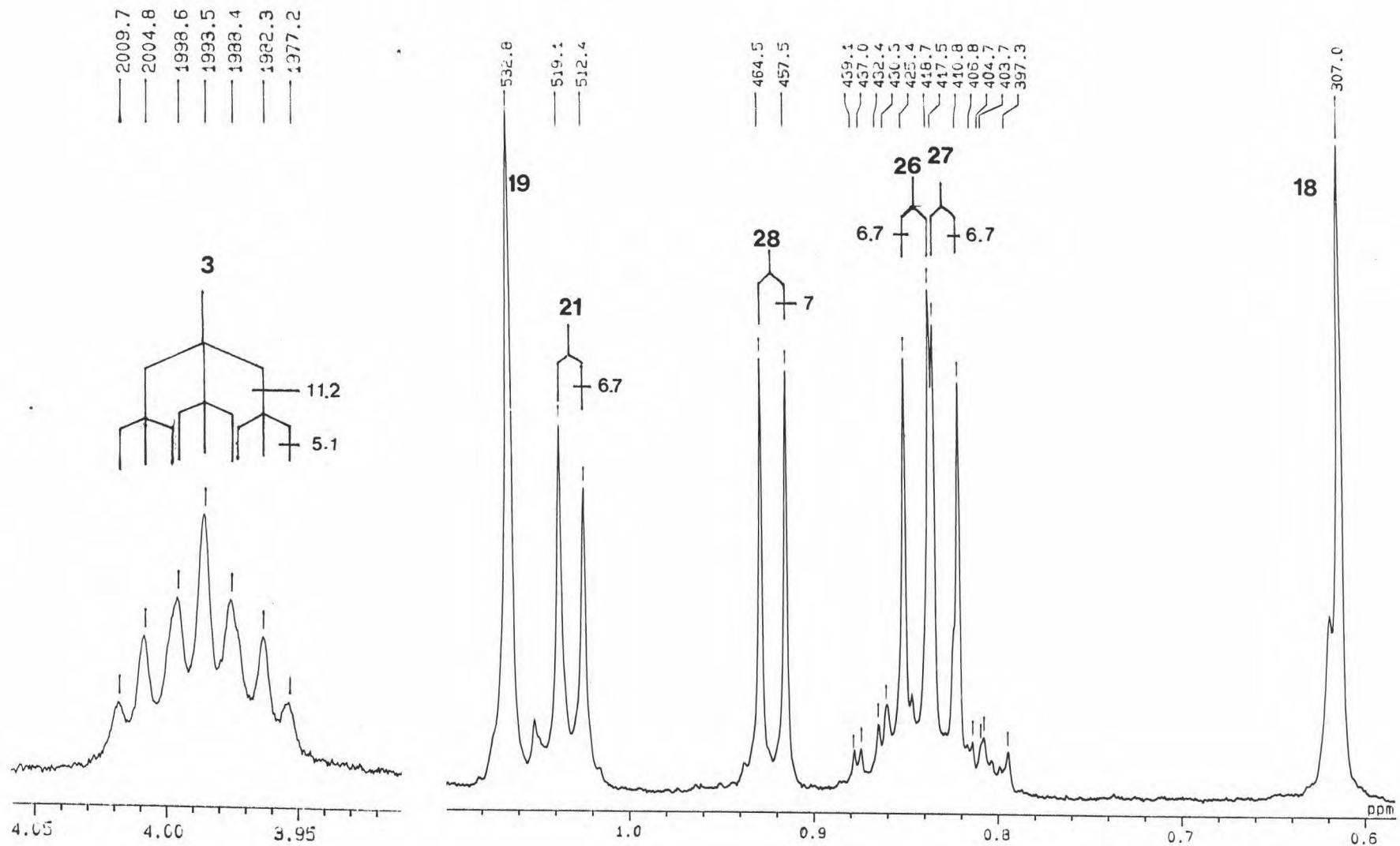


Figure 32. The 500 MHz  $^1\text{H}$  NMR spectrum of K057 in 30%  $\text{CD}_3\text{OD}$  in  $\text{CDCl}_3$  (expanded)

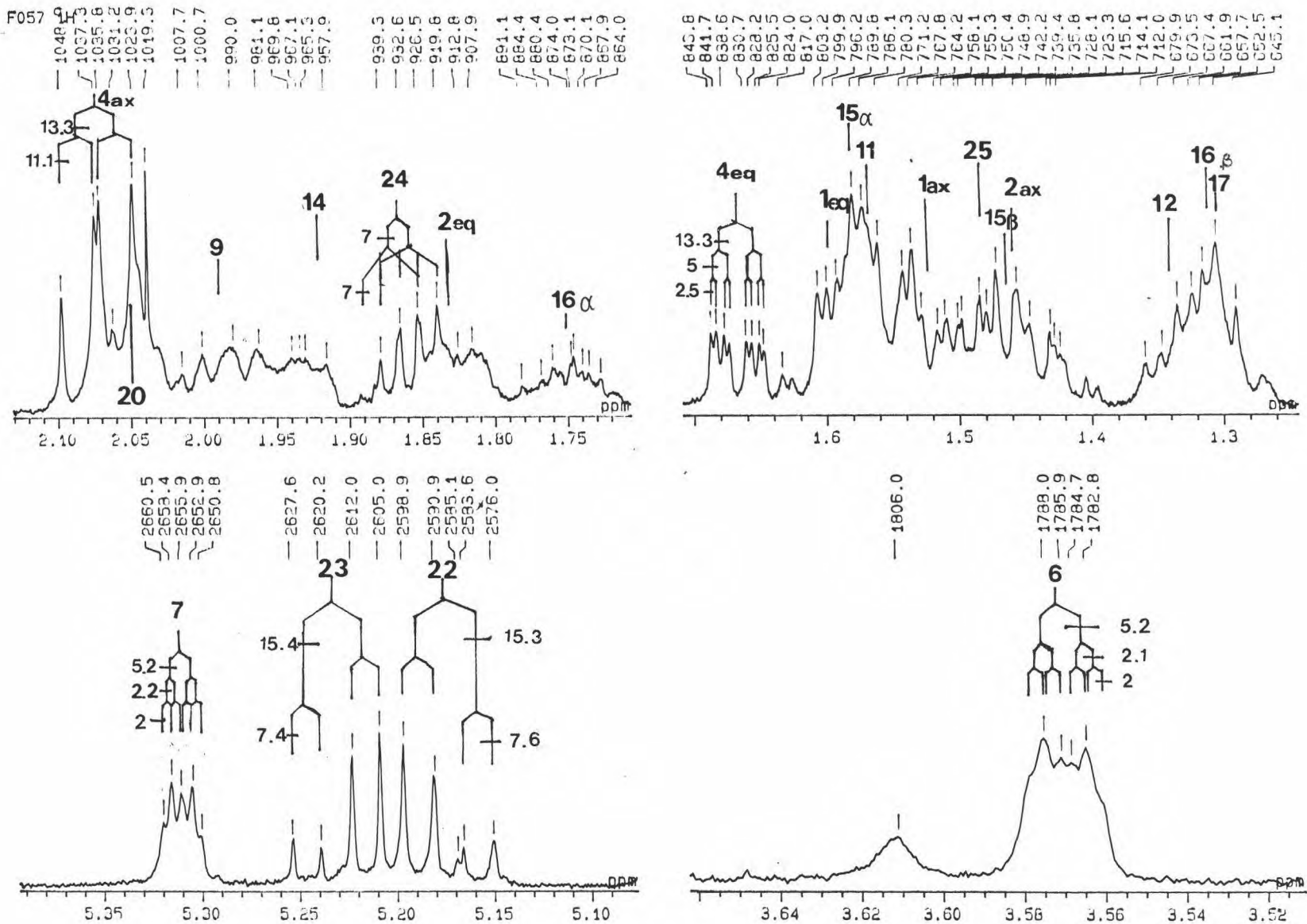


Figure 33. The 500 MHz  $^1\text{H}$  NMR spectrum of K057 in 30%  $\text{CD}_3\text{OD}$  in  $\text{CDCl}_3$  (expanded)

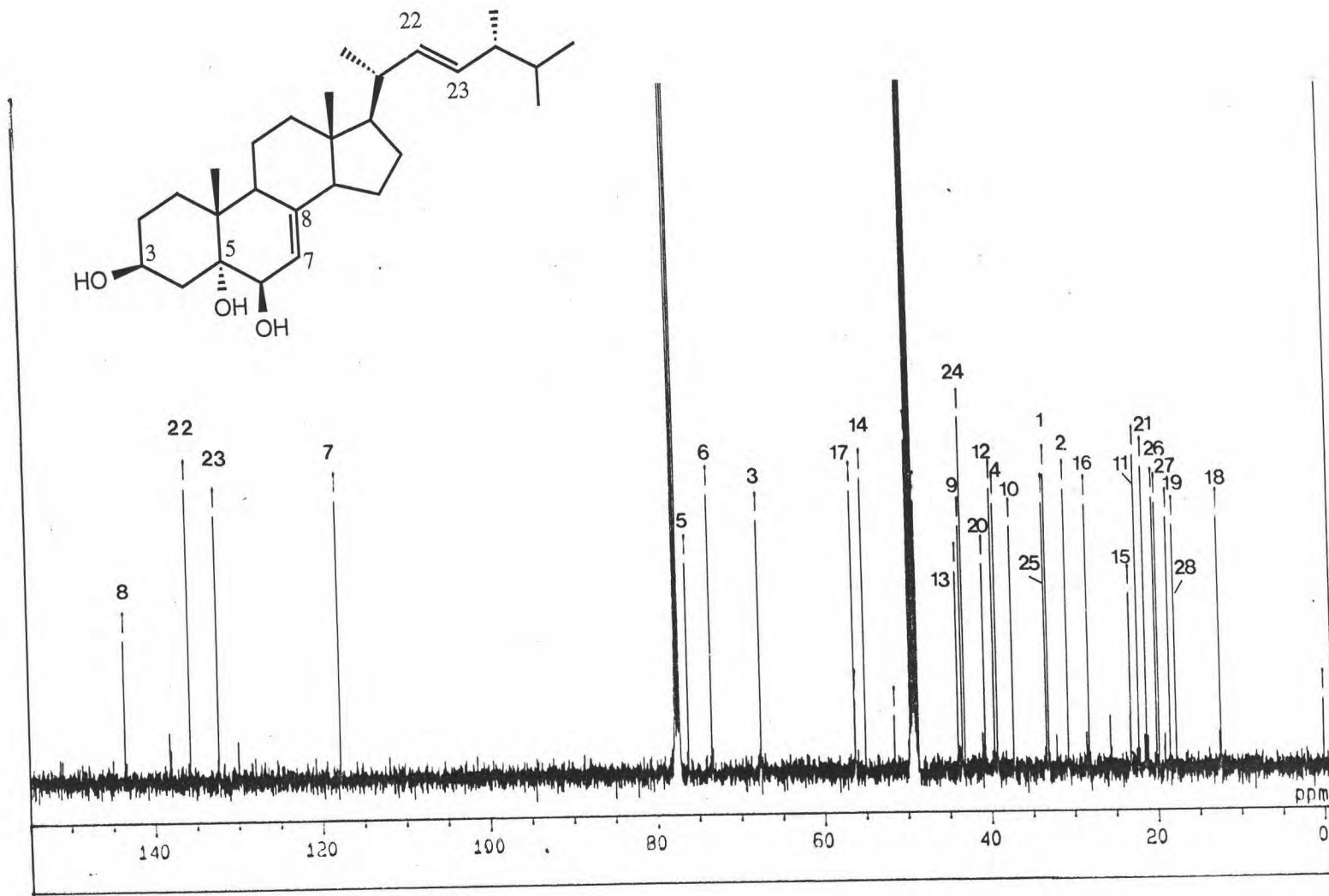


Figure 34. The 125 MHz <sup>13</sup>C NMR spectrum of K057 in 30% CD<sub>3</sub>OD in CDCl<sub>3</sub>

BIENNA K057 PHASE HSQC

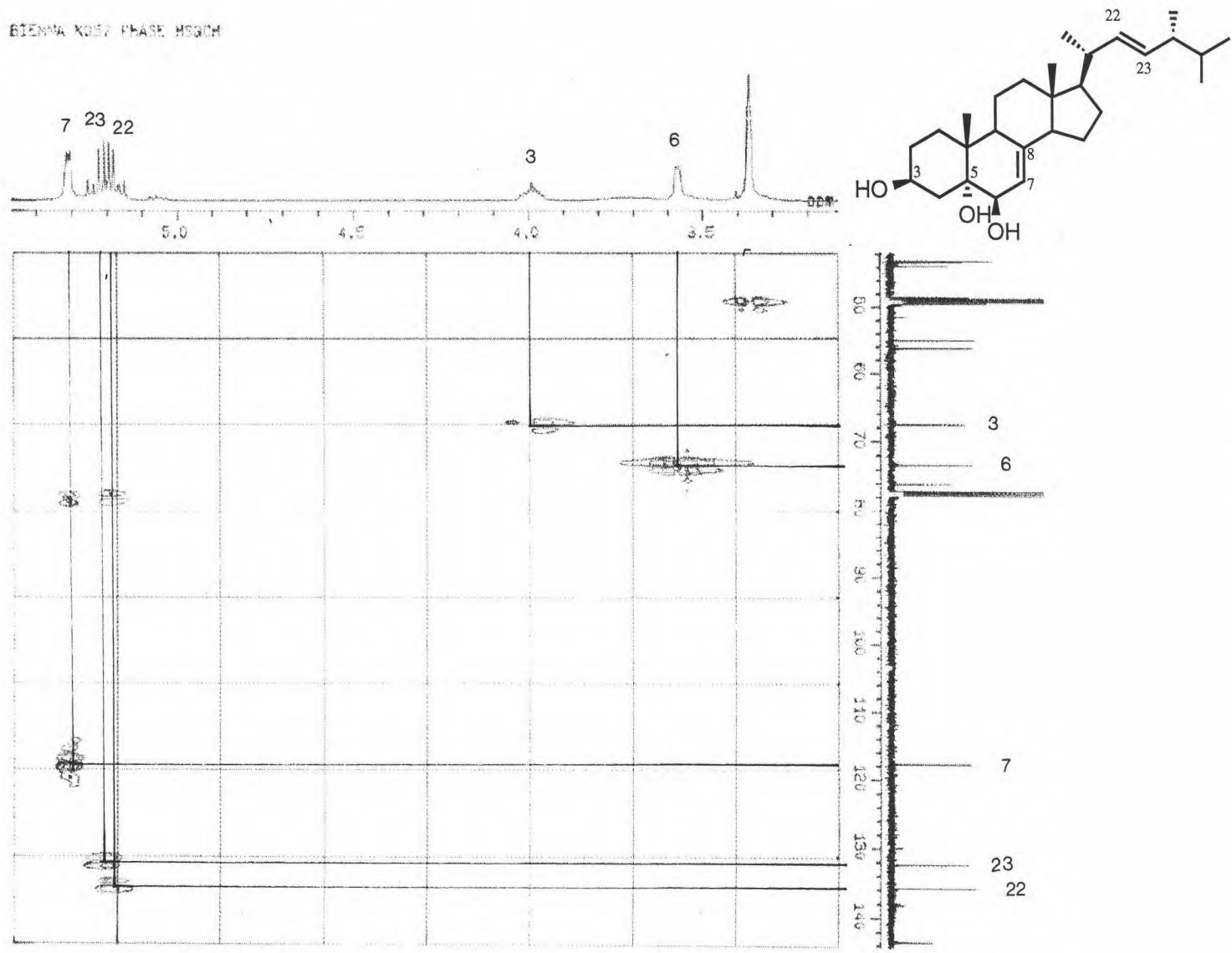


Figure 35. The partial HSQC spectrum ( $\delta_H$  3.0-5.5 ppm) of K057

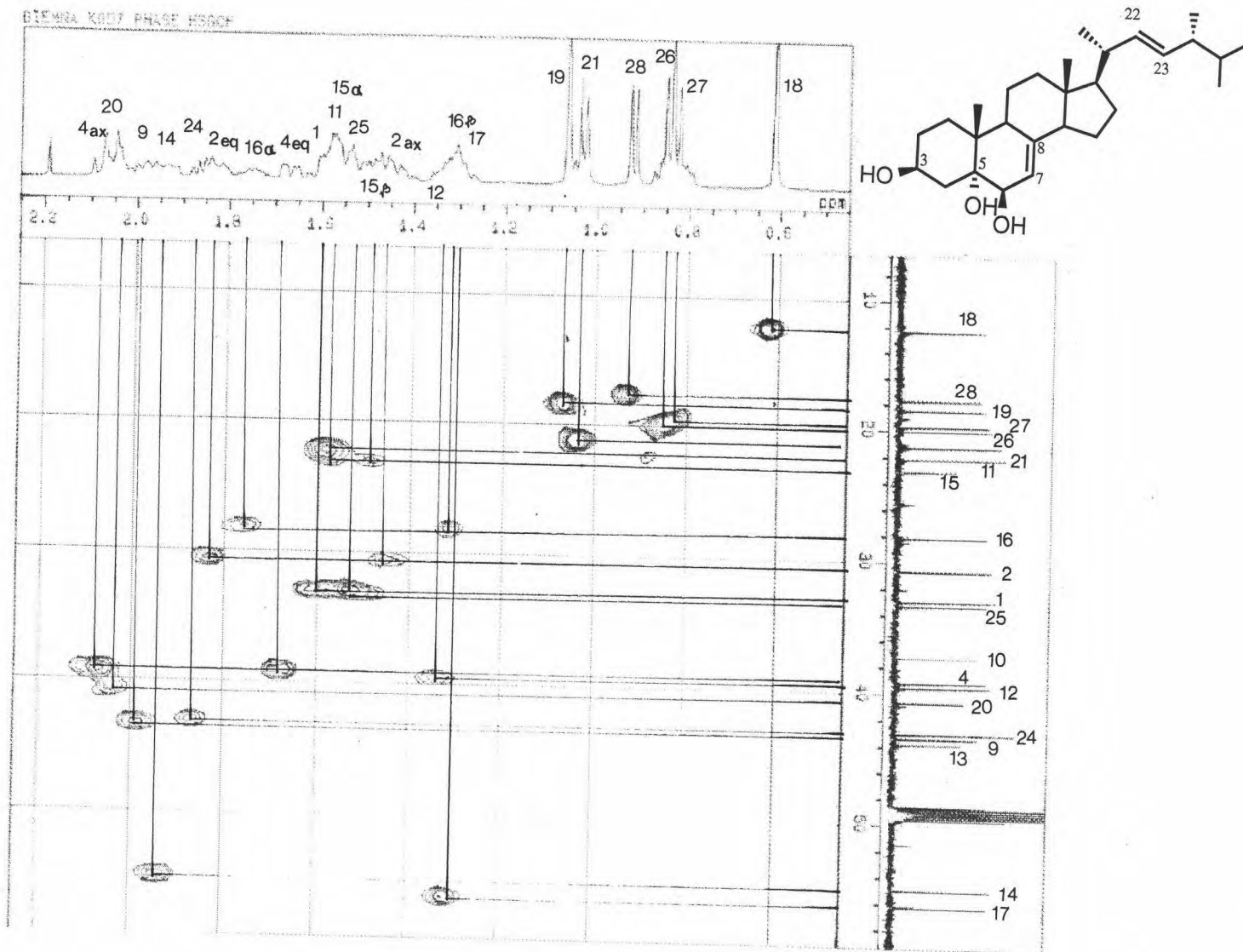


Figure 36. The partial HSQC spectrum ( $\delta_H$  0.45-2.2 ppm) of K057

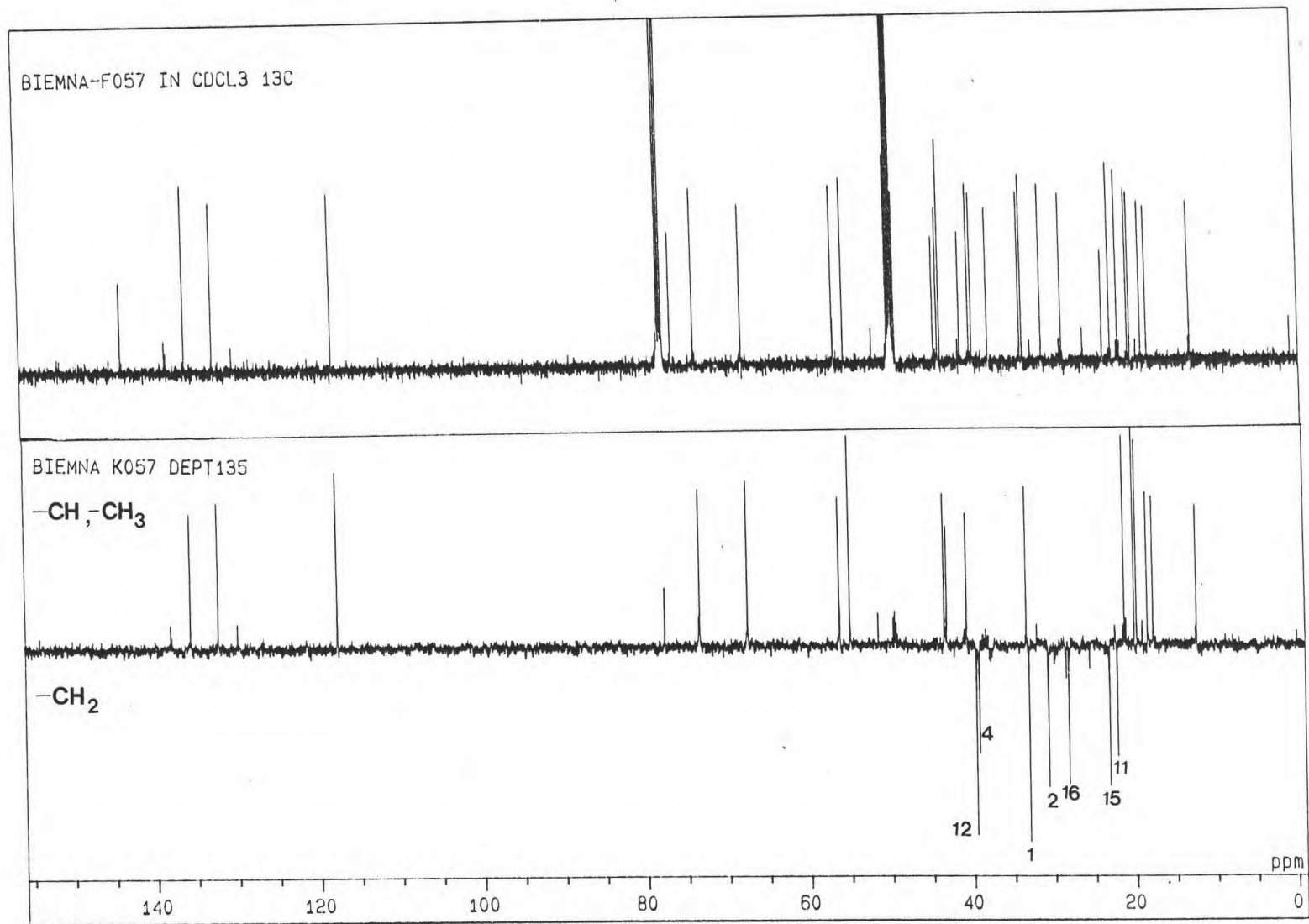


Figure 37. The DEPT 135° spectrum of K057

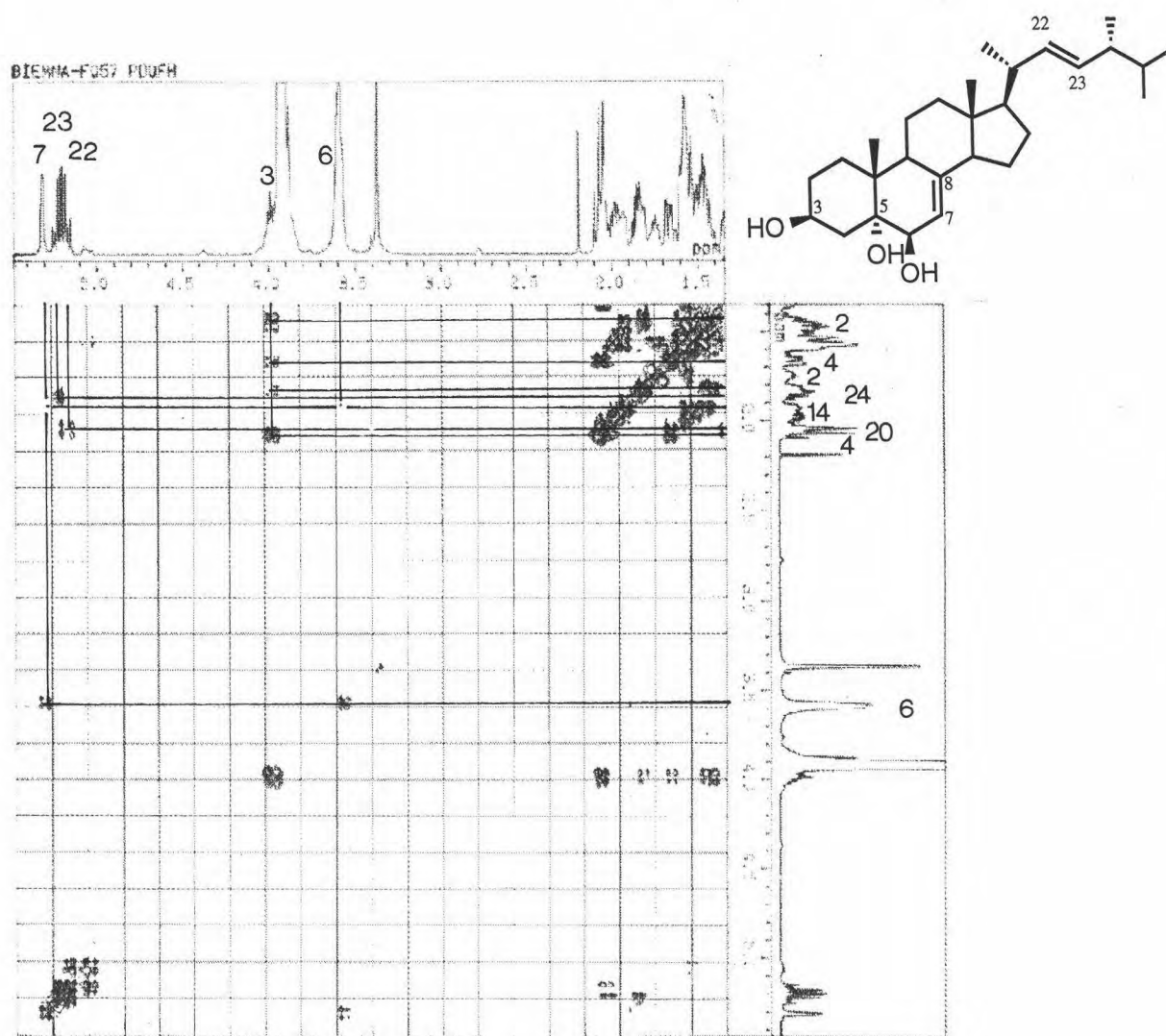


Figure 38. The partial PDQFH spectrum (δ<sub>H</sub> 1.5-5.5 ppm) of K057



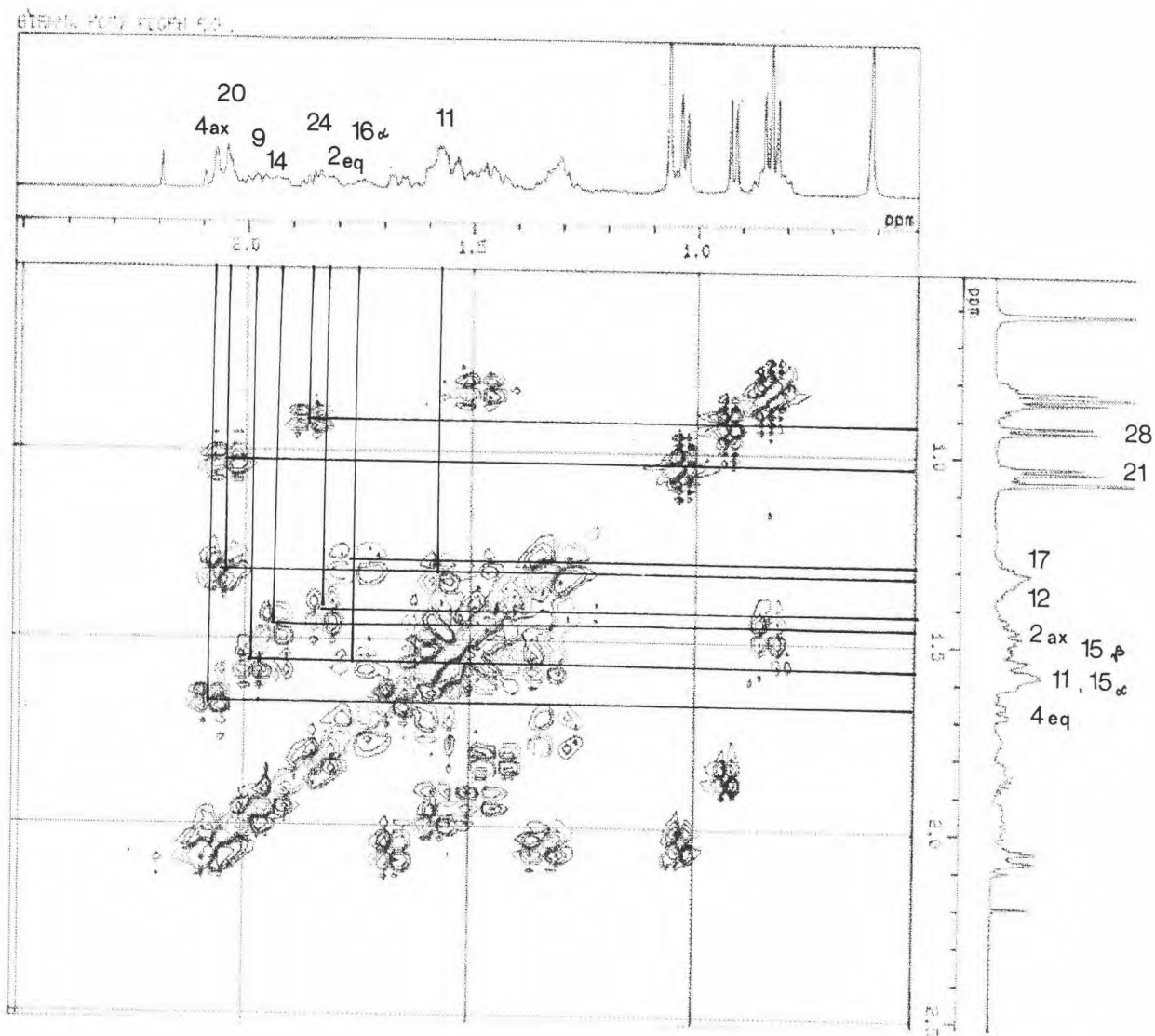


Figure 39. The partial PDQFH spectrum ( $\delta_H$  0-2.5 ppm) of K057

BIENA K057 HMBC 8 HZ

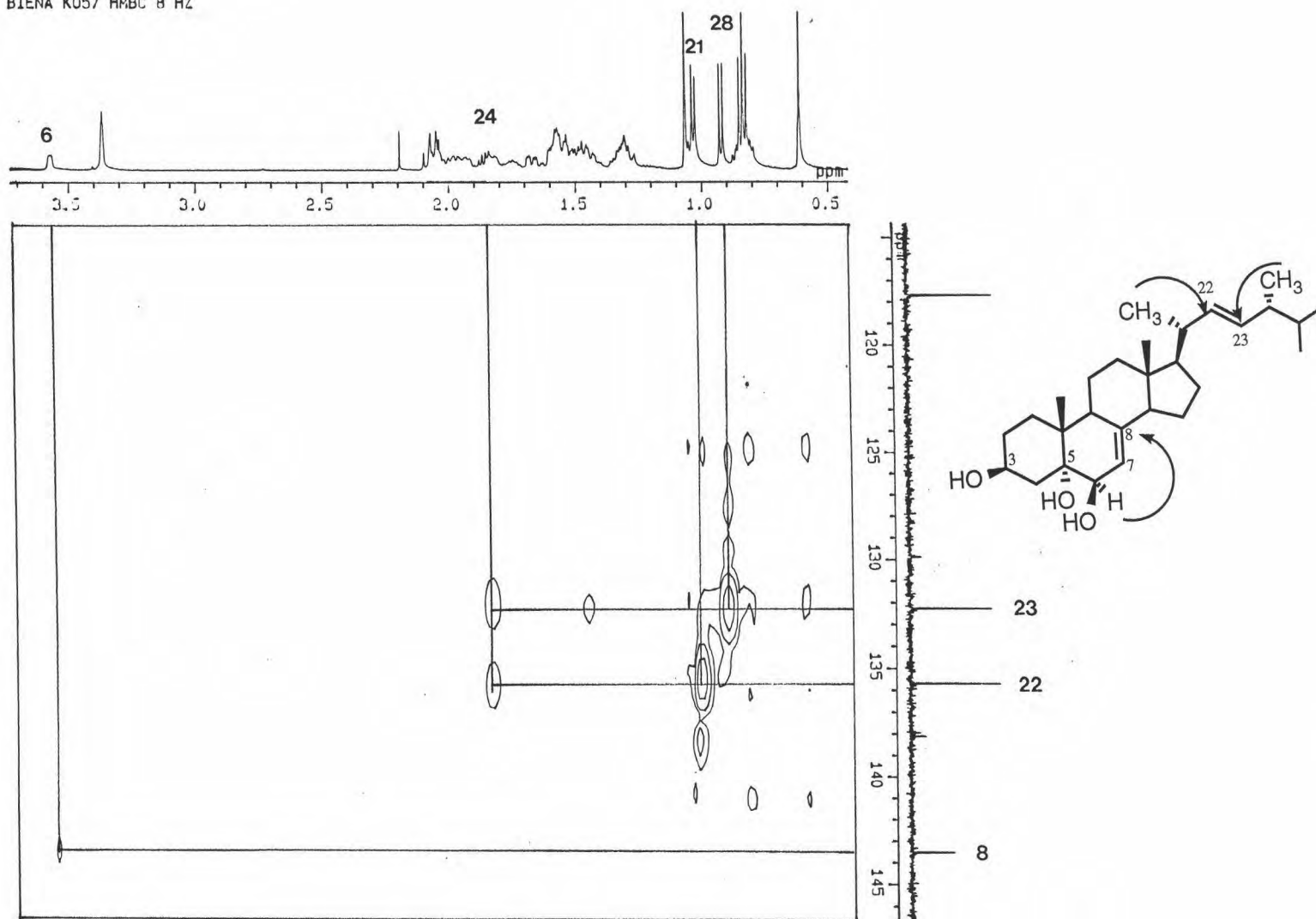


Figure 40. The partial HMBC spectrum of K057 ( $^3J_{\text{CH}} = 8\text{Hz}$ )

BIENA K057 HMBC 8 HZ

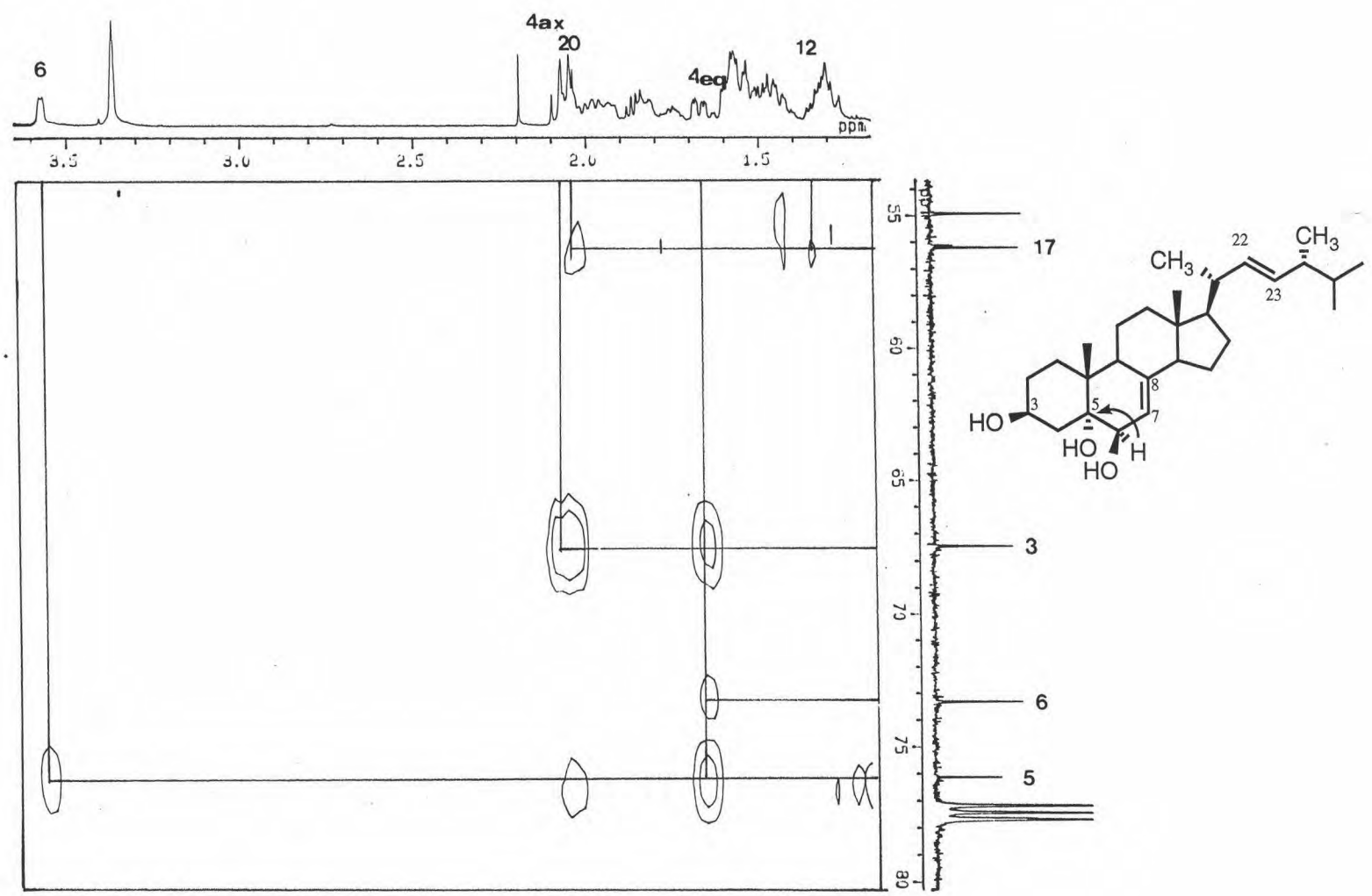


Figure 41. The partial HMBC spectrum of K057 ( $^3J_{CH} = 8\text{Hz}$ )

BIENA K057 HMBC 8 HZ

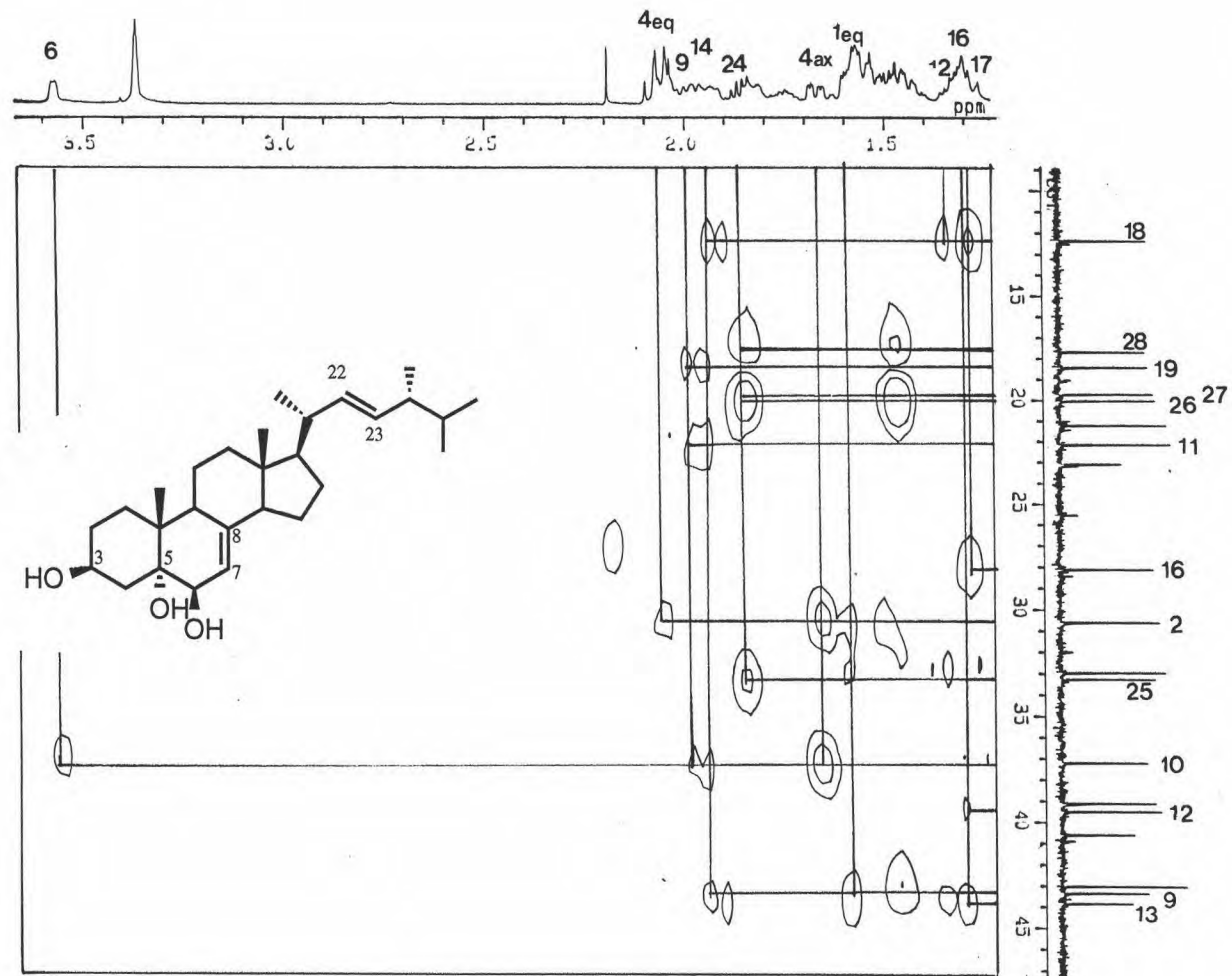


Figure 42. The partial HMBC spectrum of K057 ( $^3J_{CH} = 8\text{Hz}$ )

BIENA K057 HMBC 8 HZ

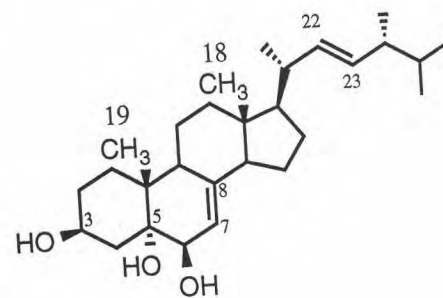
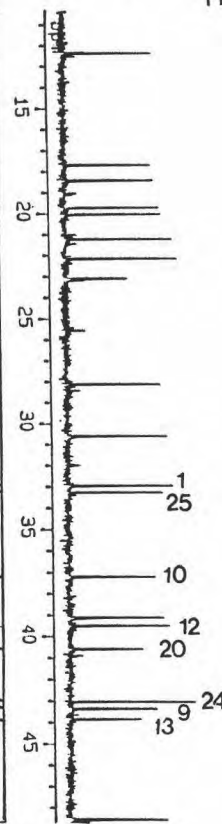
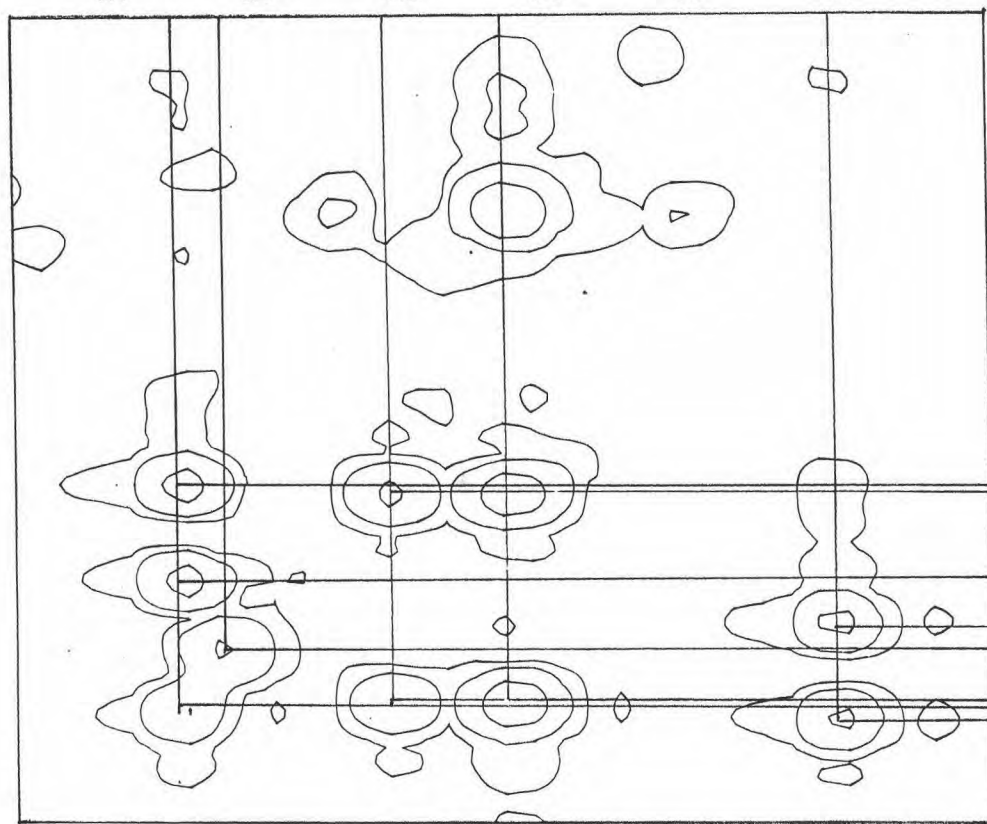
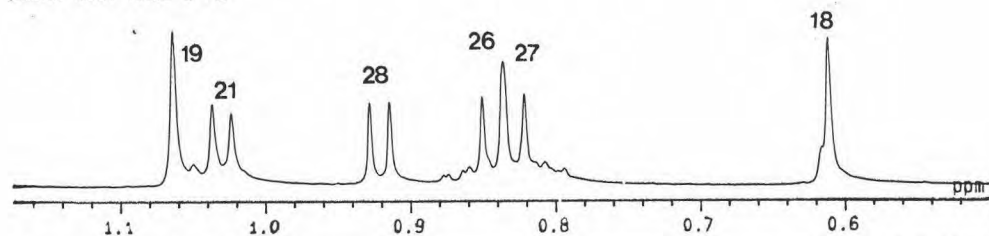


Figure 43. The partial HMBC spectrum of K057 ( $^3J_{CH} = 8\text{Hz}$ )

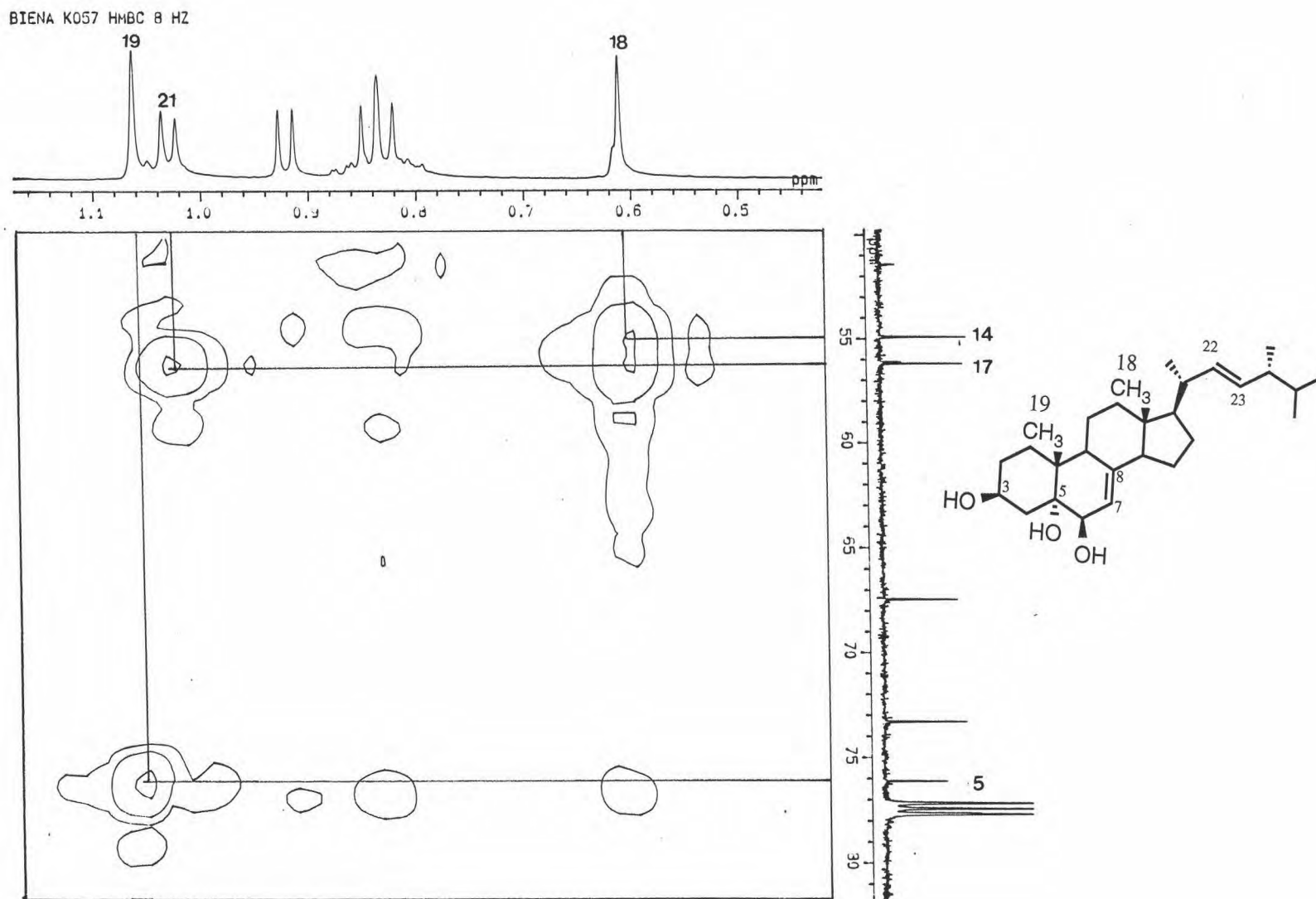


Figure 44. The partial HMBC spectrum of K057 ( $^3J_{CH} = 8\text{Hz}$ )

BIENA K057 HMBC 8 HZ

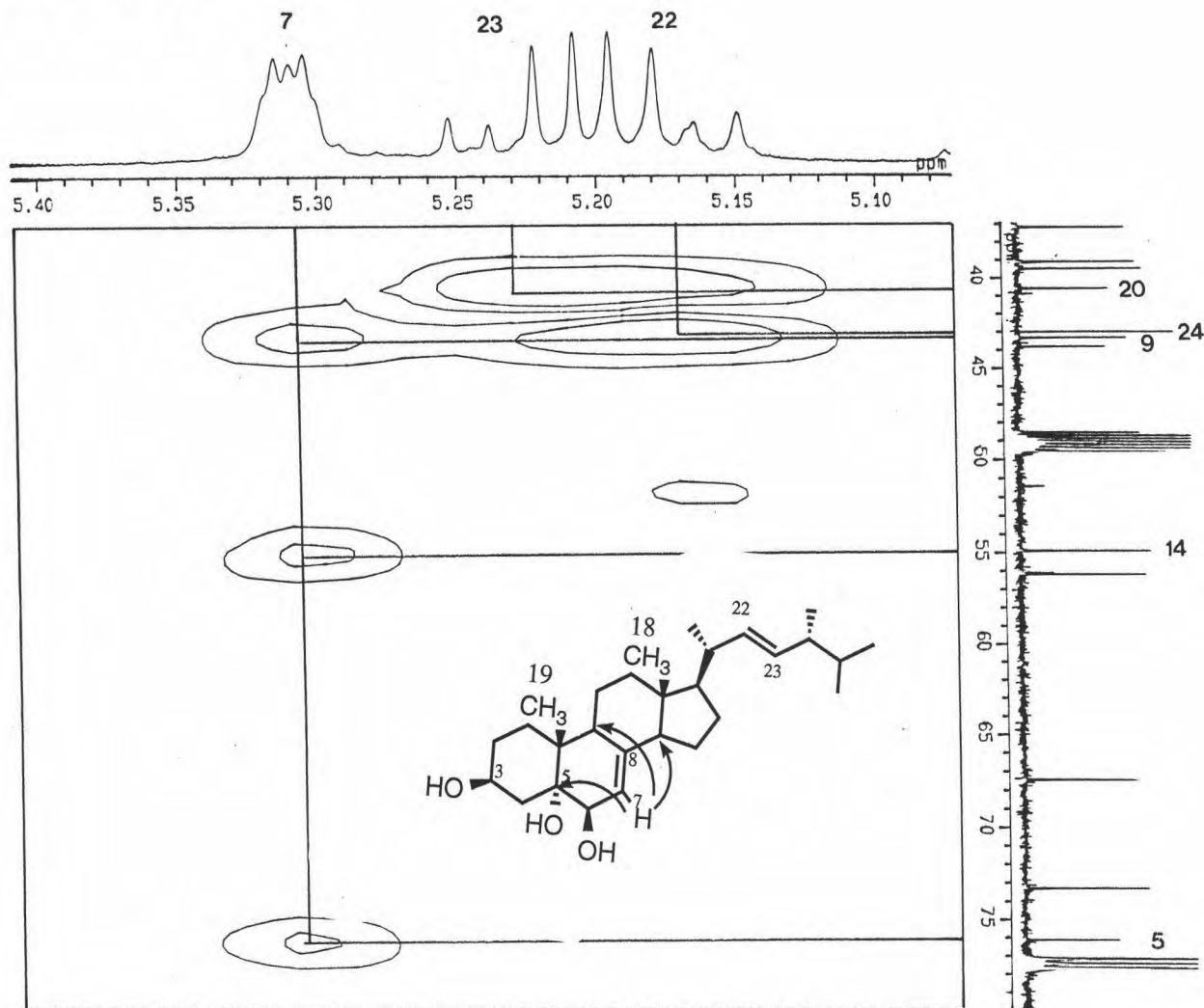


Figure 45. The partial HMBC spectrum of K057 ( $^3J_{\text{CH}} = 8\text{Hz}$ )

BIENA K057 HMBC 8 HZ

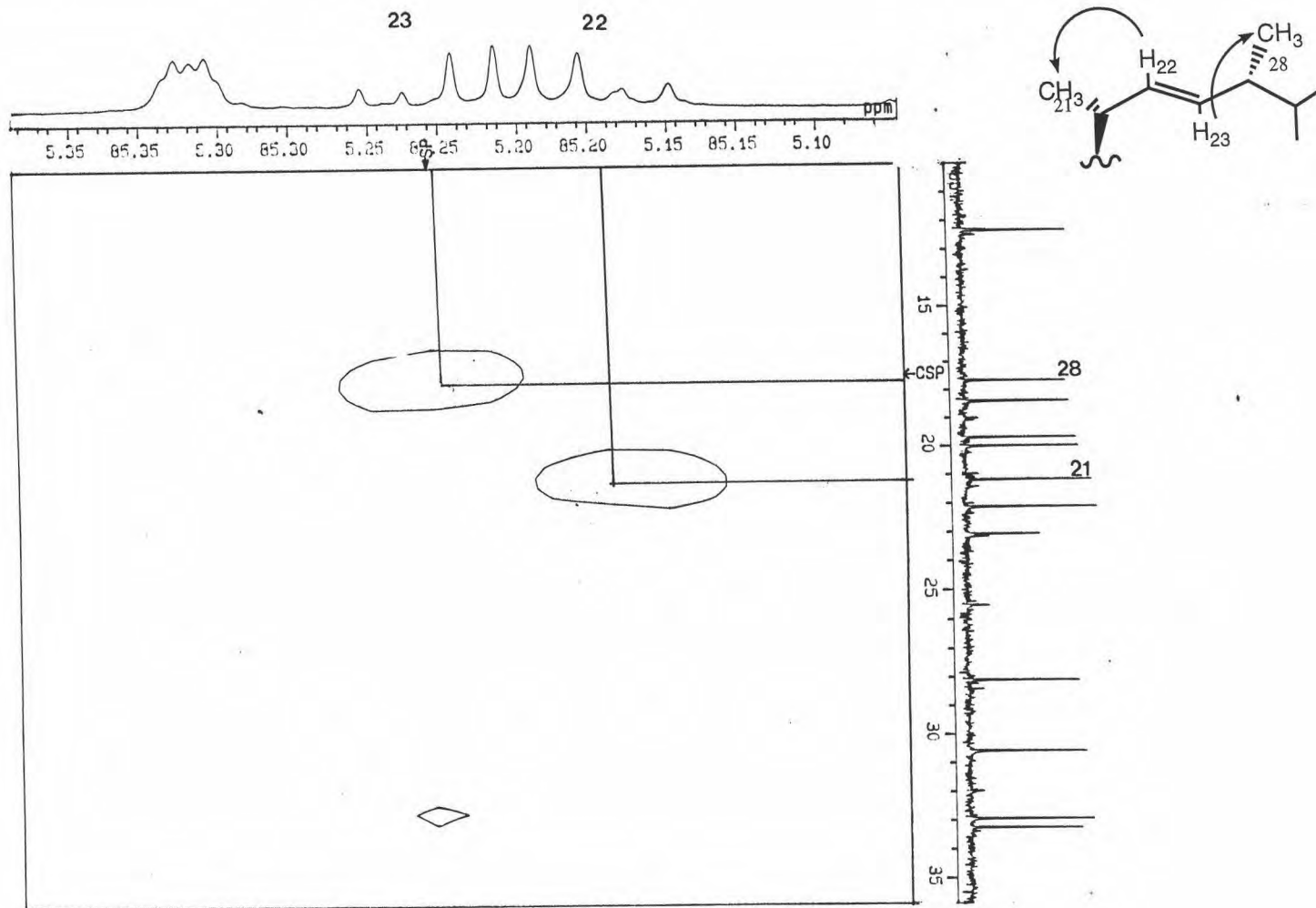


Figure 46. The partial HMBC spectrum of K057 ( $^3J_{CH} = 8\text{Hz}$ )



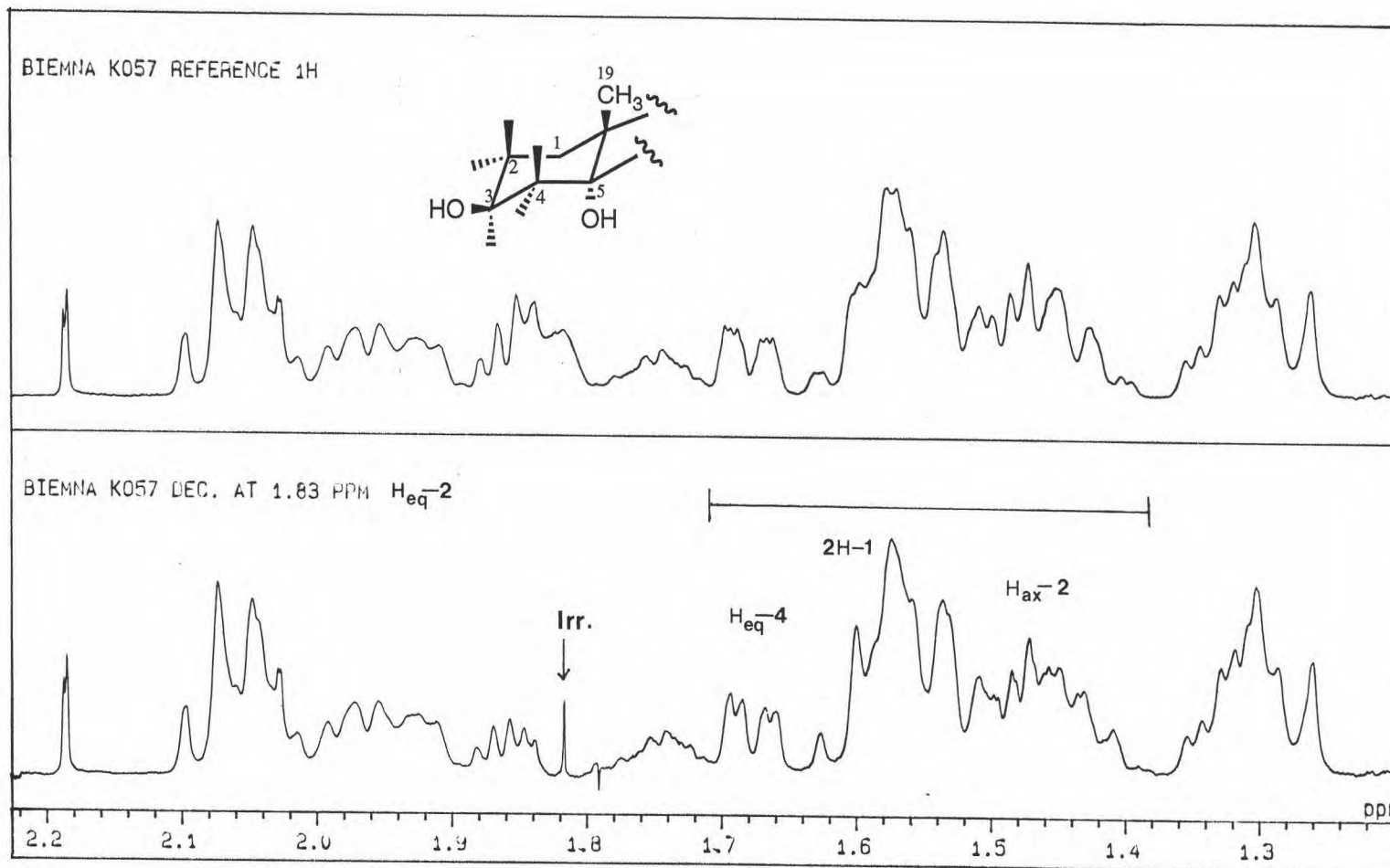


Figure 47. The Decoupling experiment spectrum of K057 ( $H_{eq-2}$ ,  $\delta$  2.3-1.1 ppm )

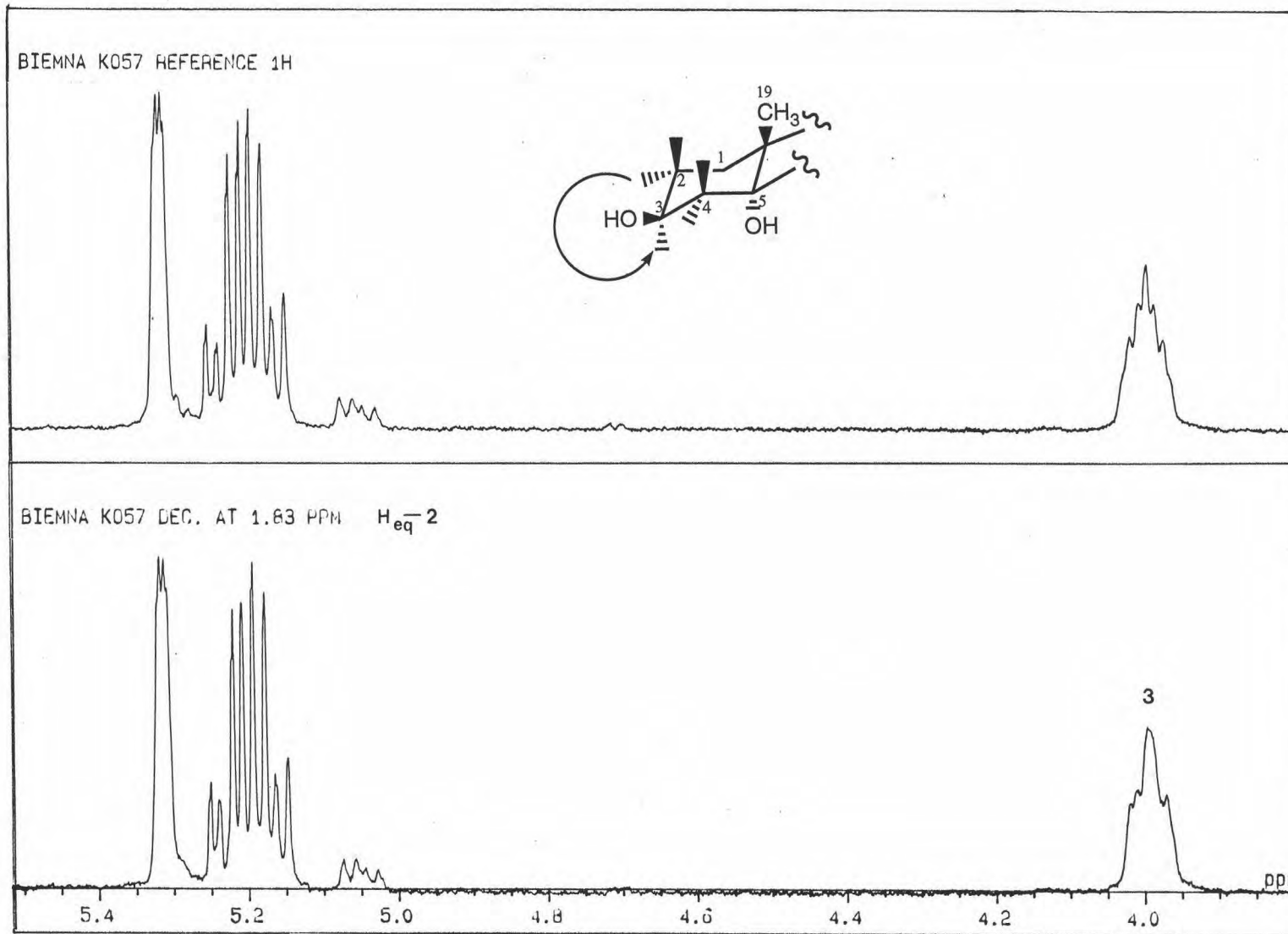


Figure 48. The Decoupling experiment spectrum of K057 ( $H_{eq-2}$ ,  $\delta$  5.5-3.8 ppm ).



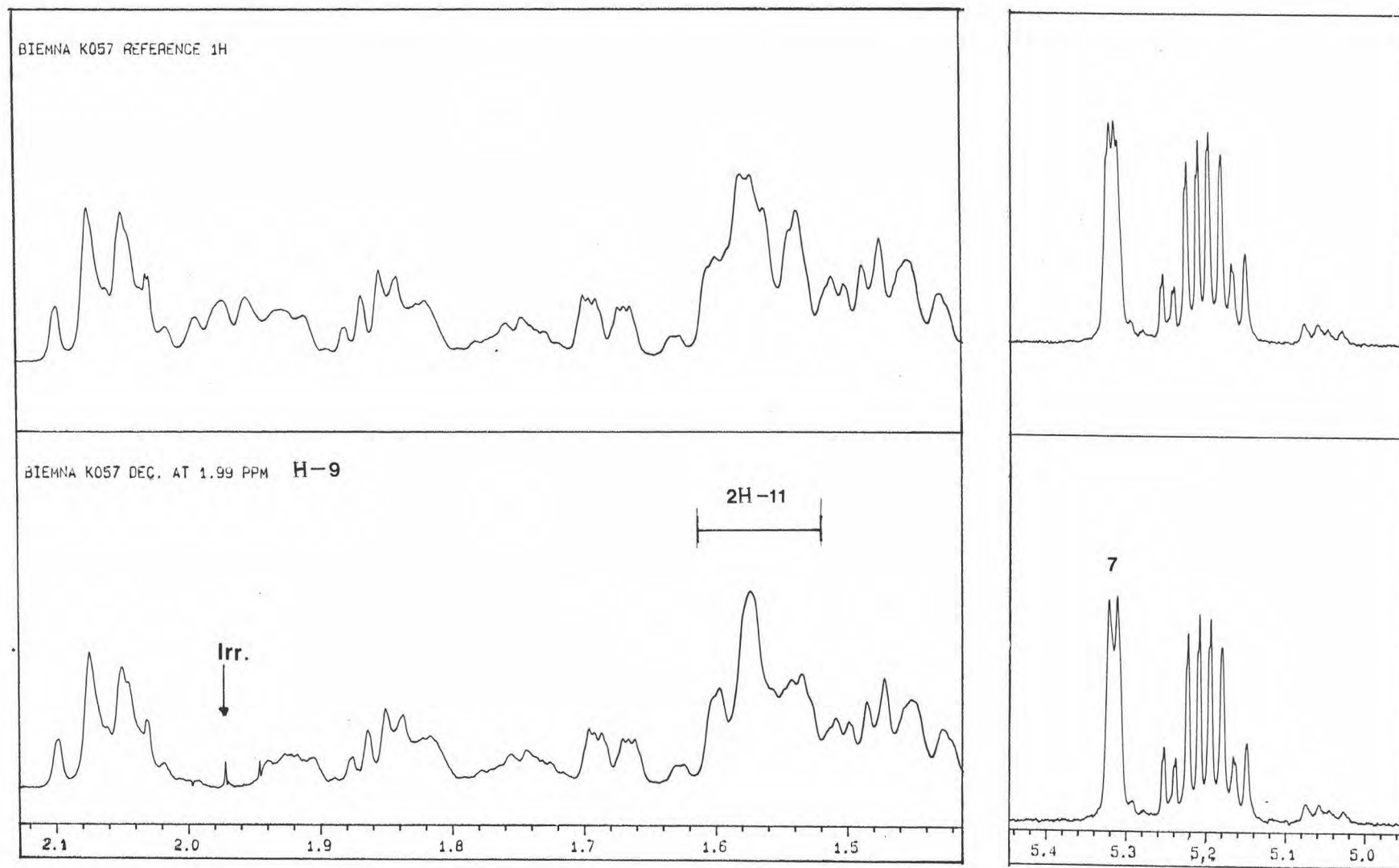


Figure 49. The Decoupling experiment spectrum of K057 (H-9)

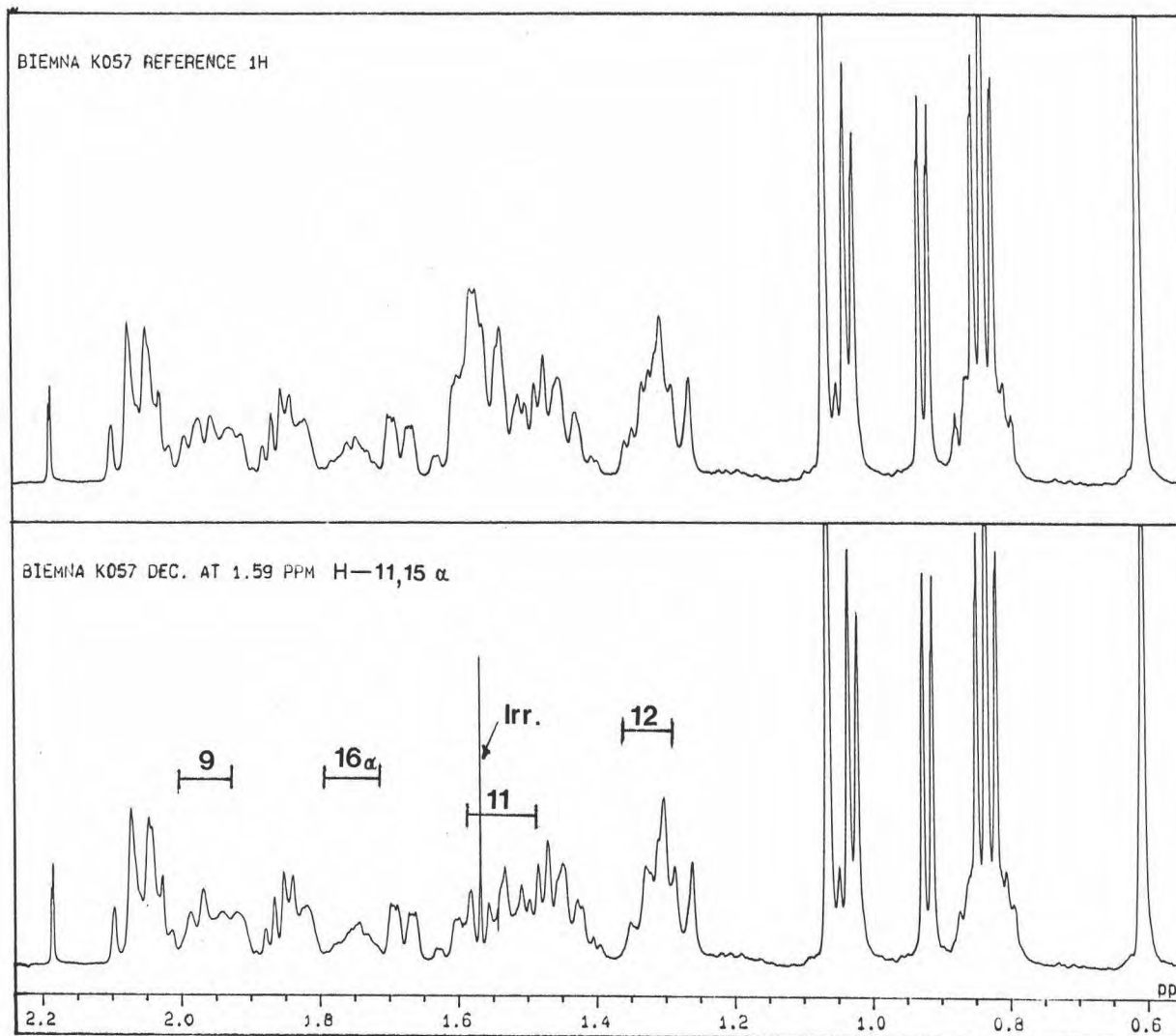


Figure 50. The Decoupling experiment spectrum of K057 (H-11, H $\alpha$ -15)

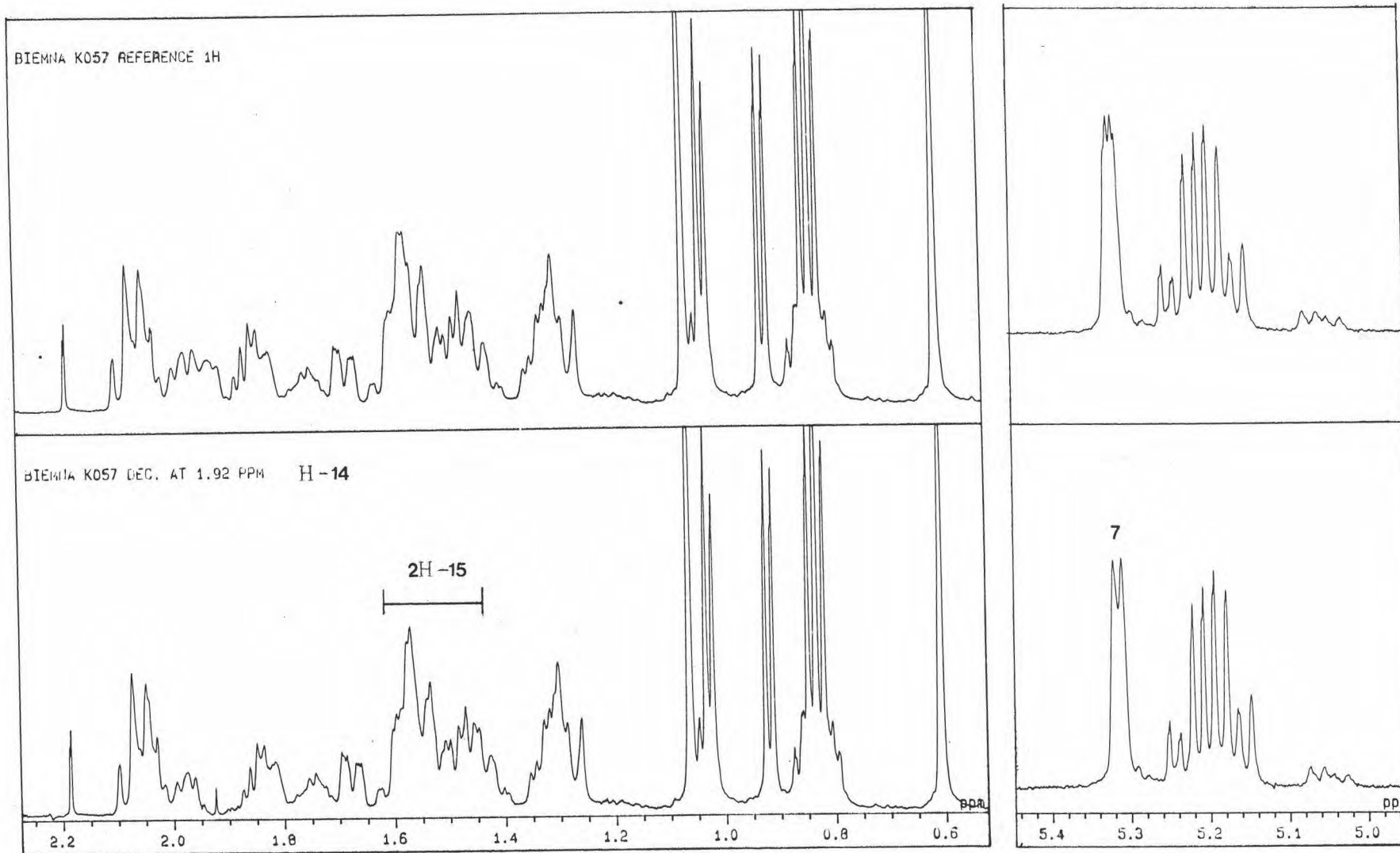


Figure 51. The Decoupling experiment spectrum of K057 (H-14)

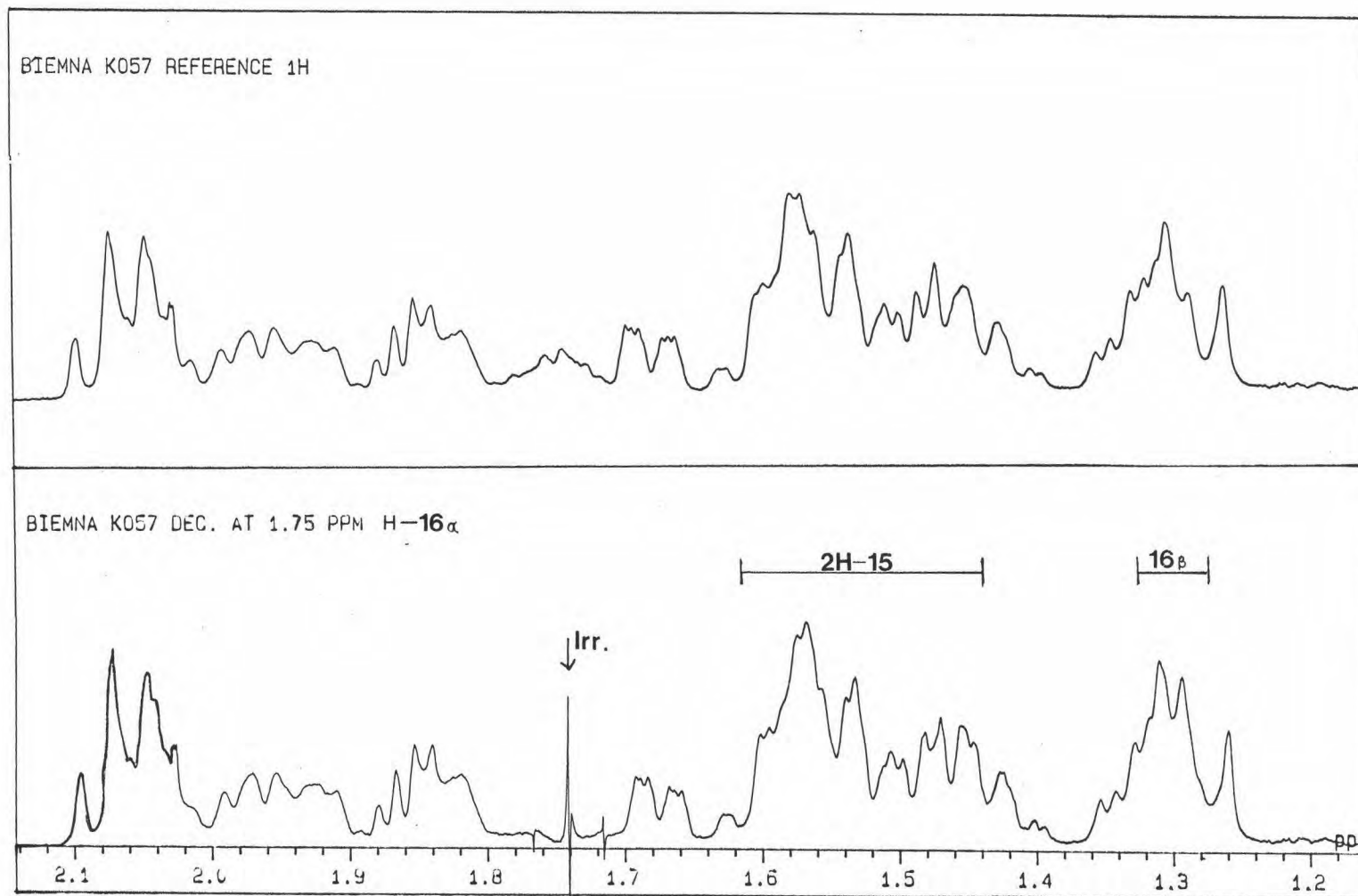


Figure 52. The Decoupling experiment spectrum of K057 (H $\alpha$ -16)

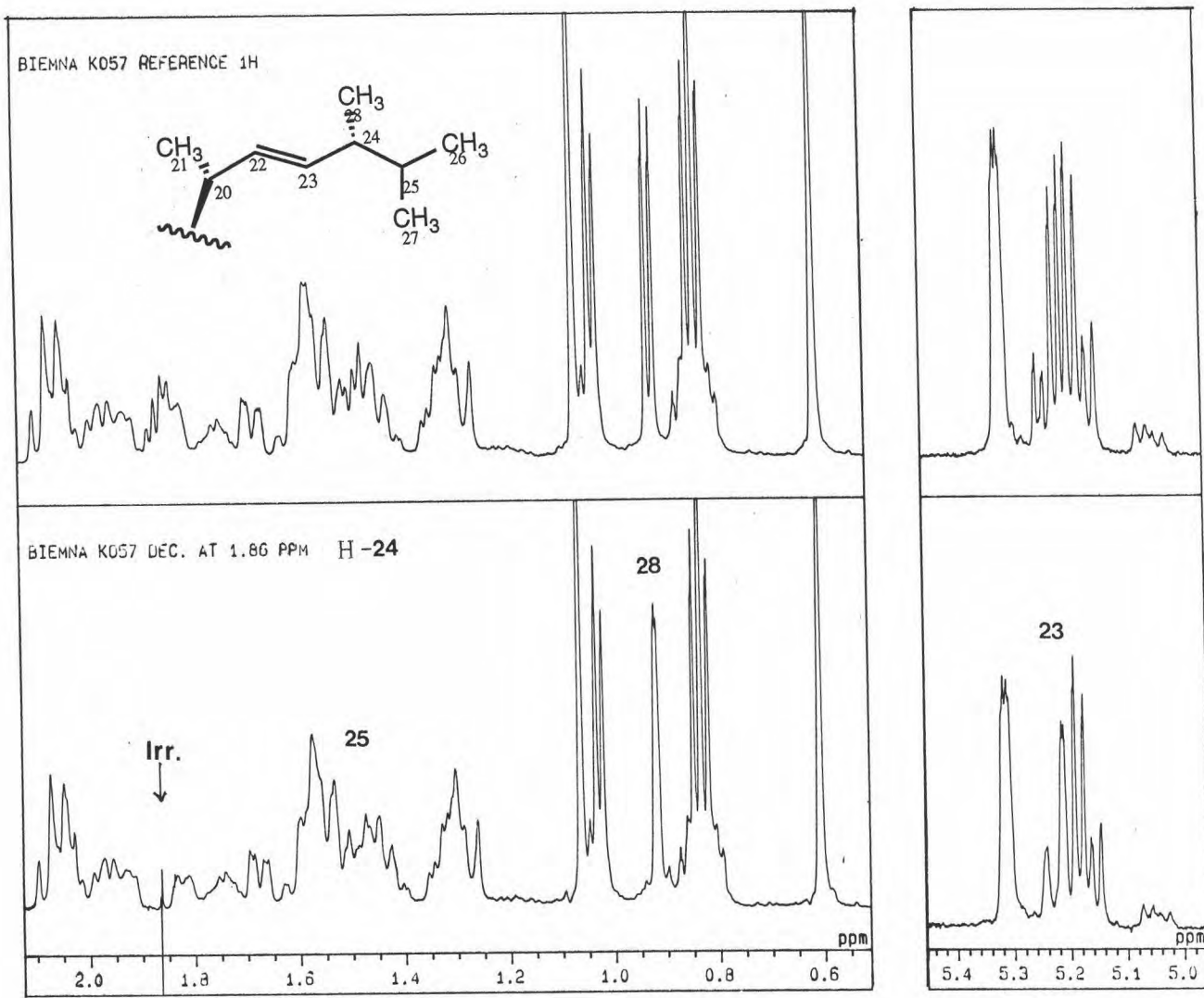


Figure 53. The Decoupling experiment spectrum of K057 (H-24)

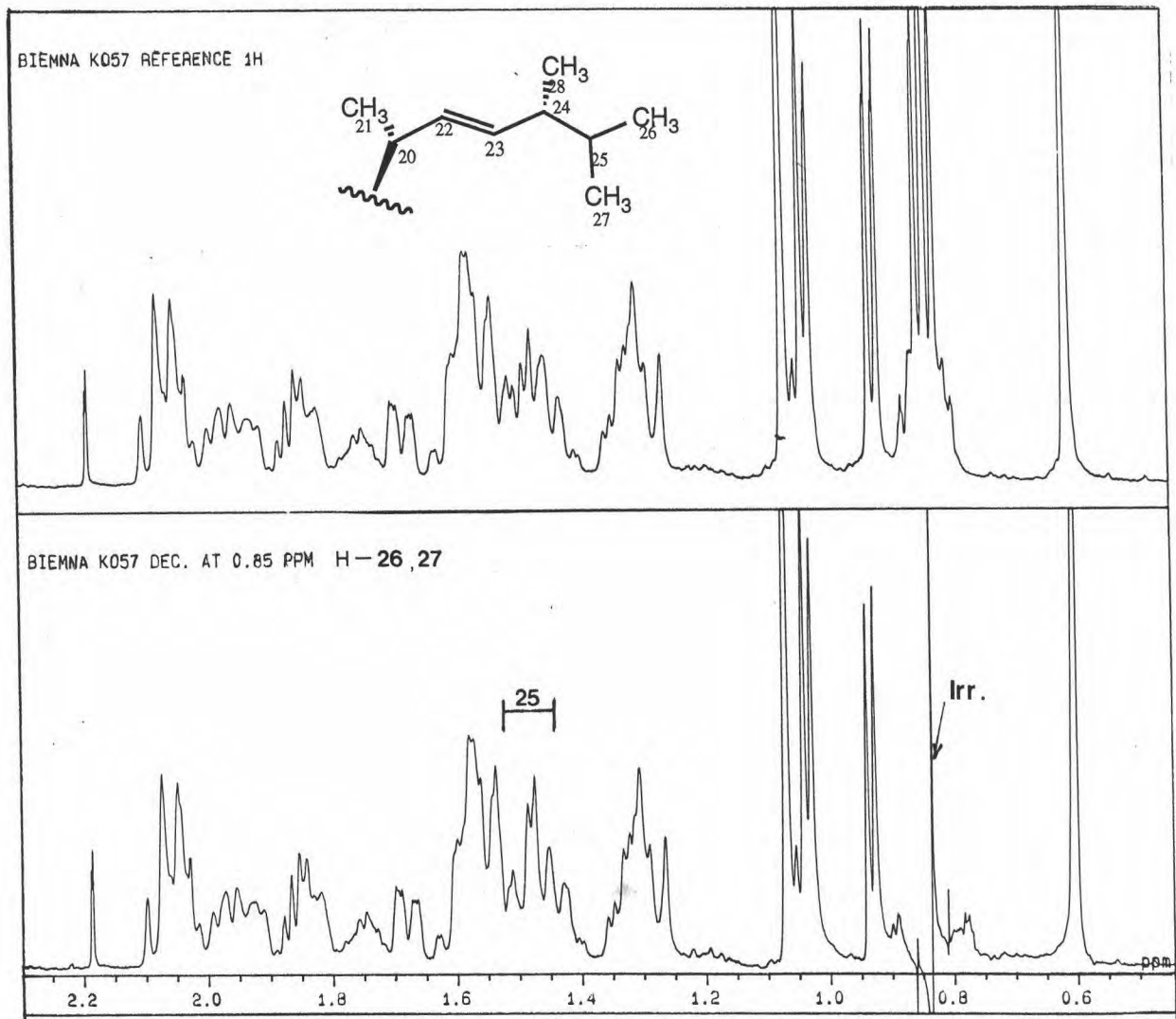


Figure 54. The Decoupling experiment spectrum of K057 (H-26, H-27)



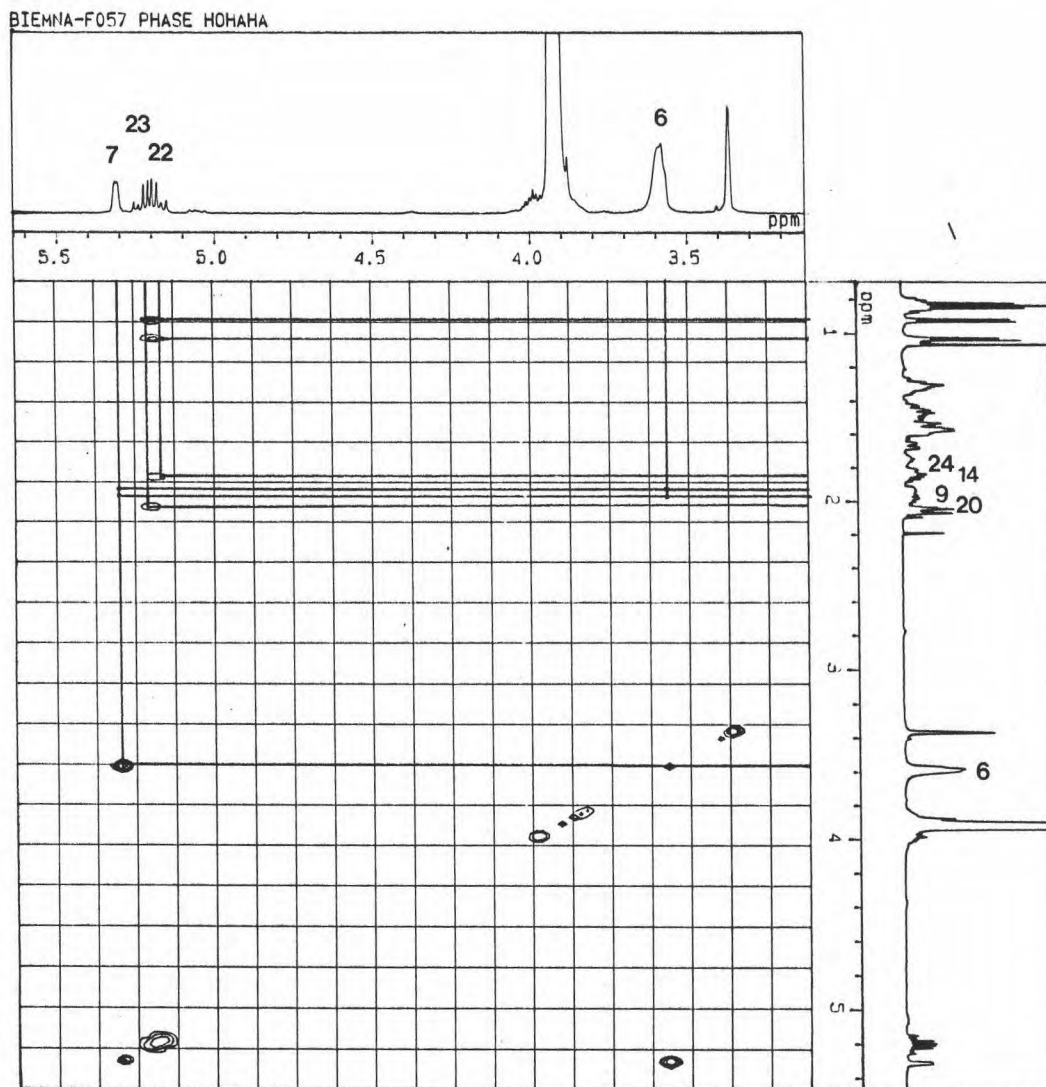


Figure 55. The partial HOHAHA spectrum ( $\delta_H$  3.0-5.5 ppm) of K057

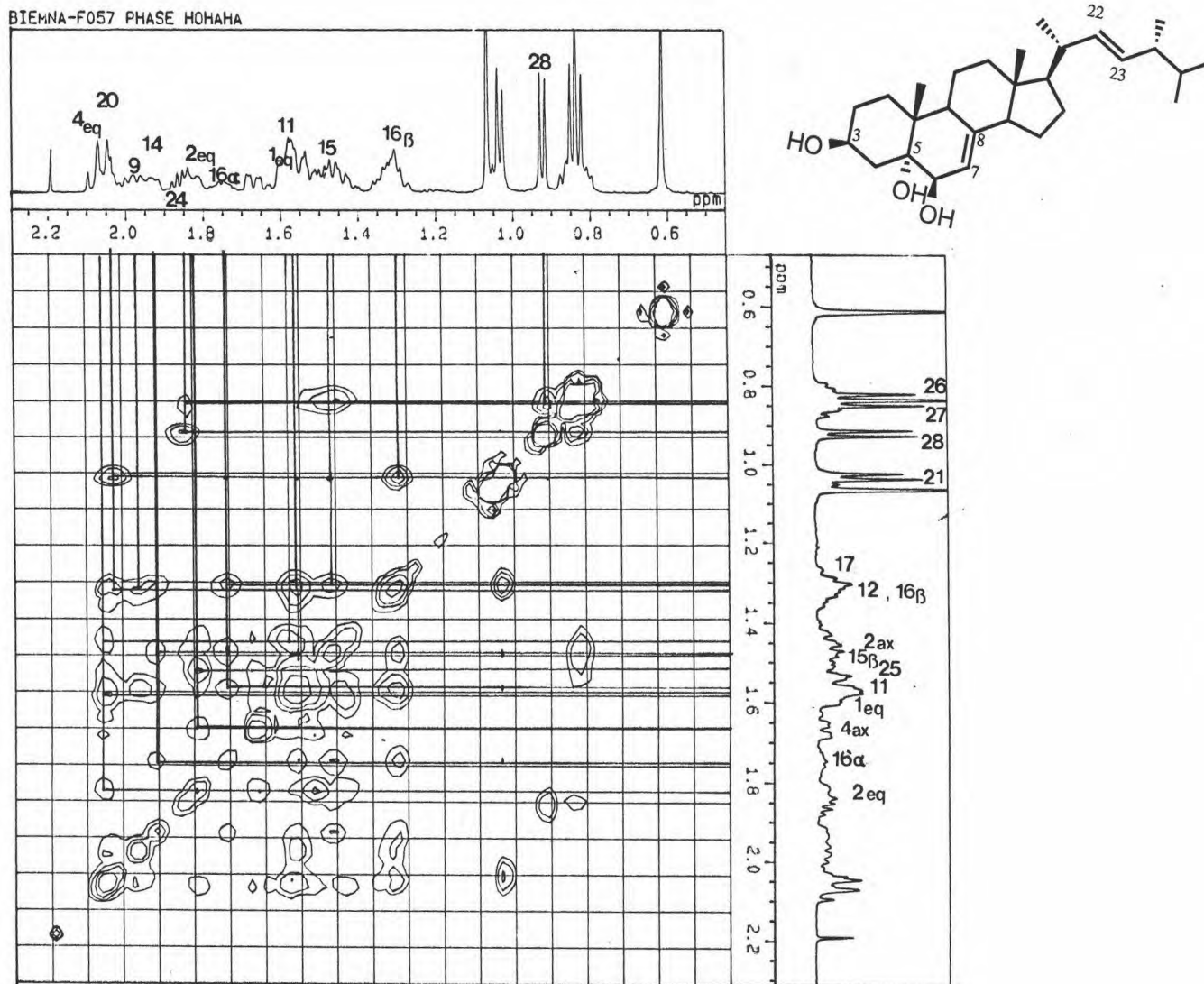


Figure 56. The partial HOHAHA spectrum ( $\delta_H$  0.5-2.2 ppm) of K057

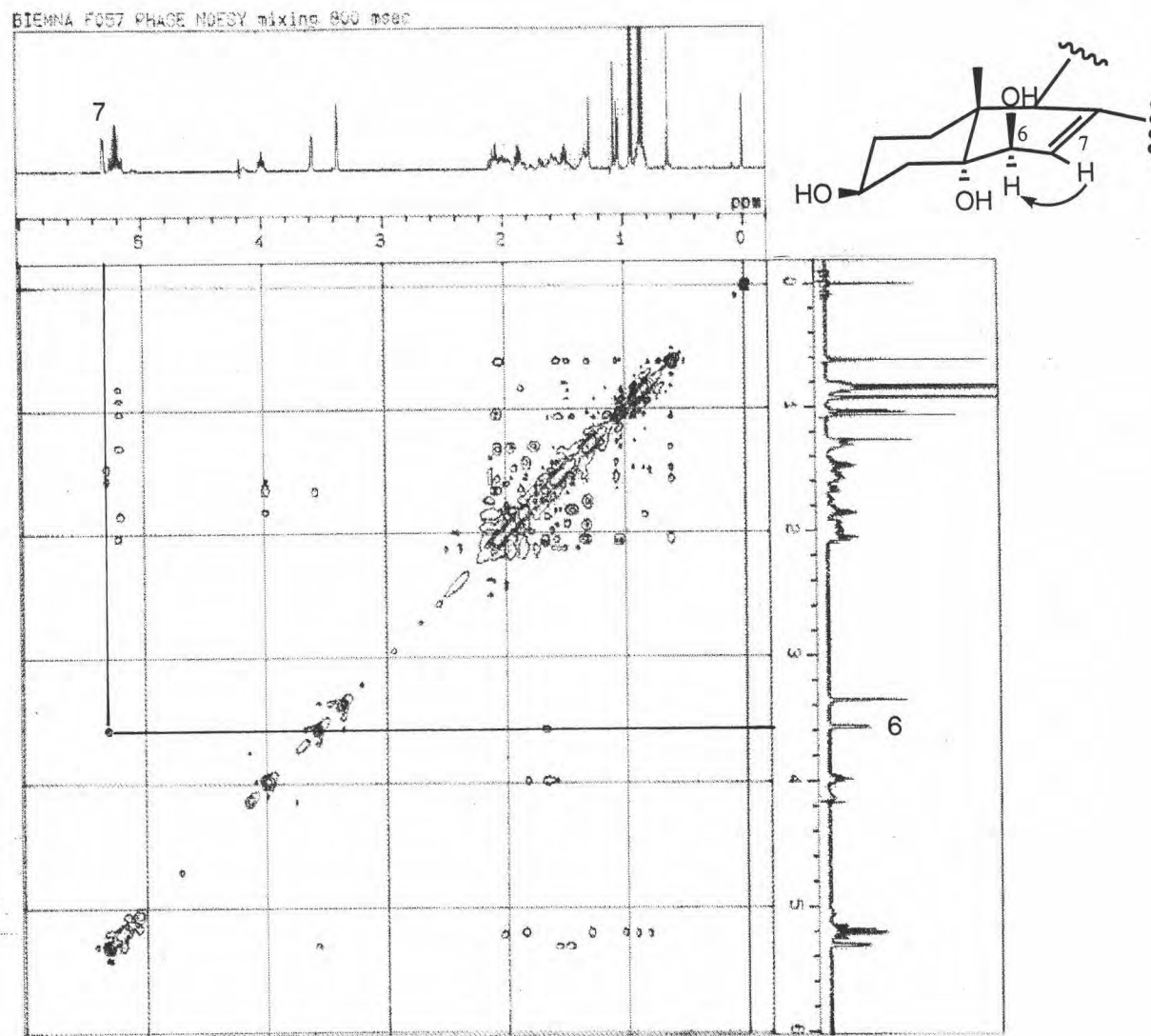


Figure 57. The NOESY spectrum of K057

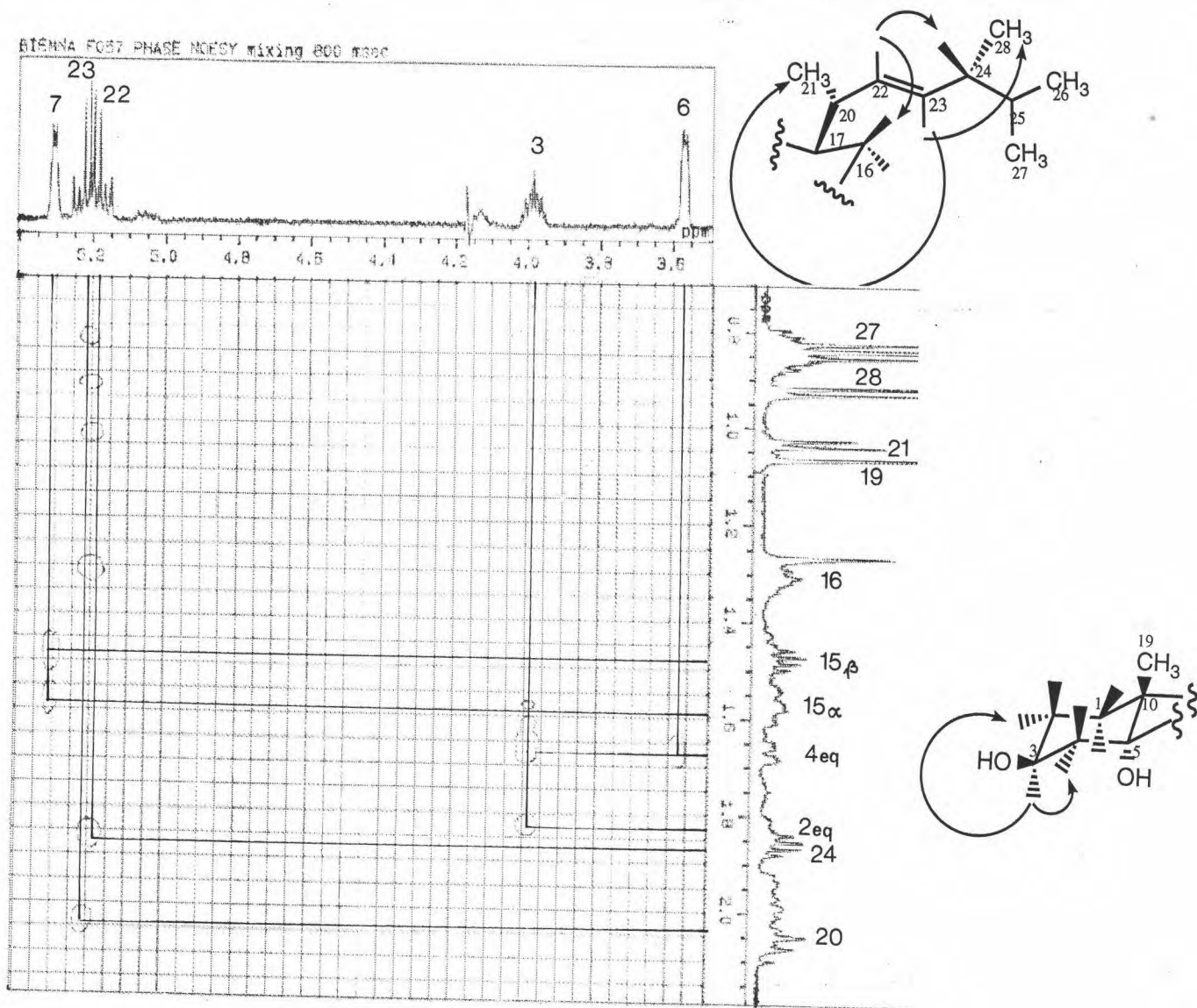


Figure 58. The partial NOESY spectrum ( $\delta_H$  3.5-5.4 ppm) of K057

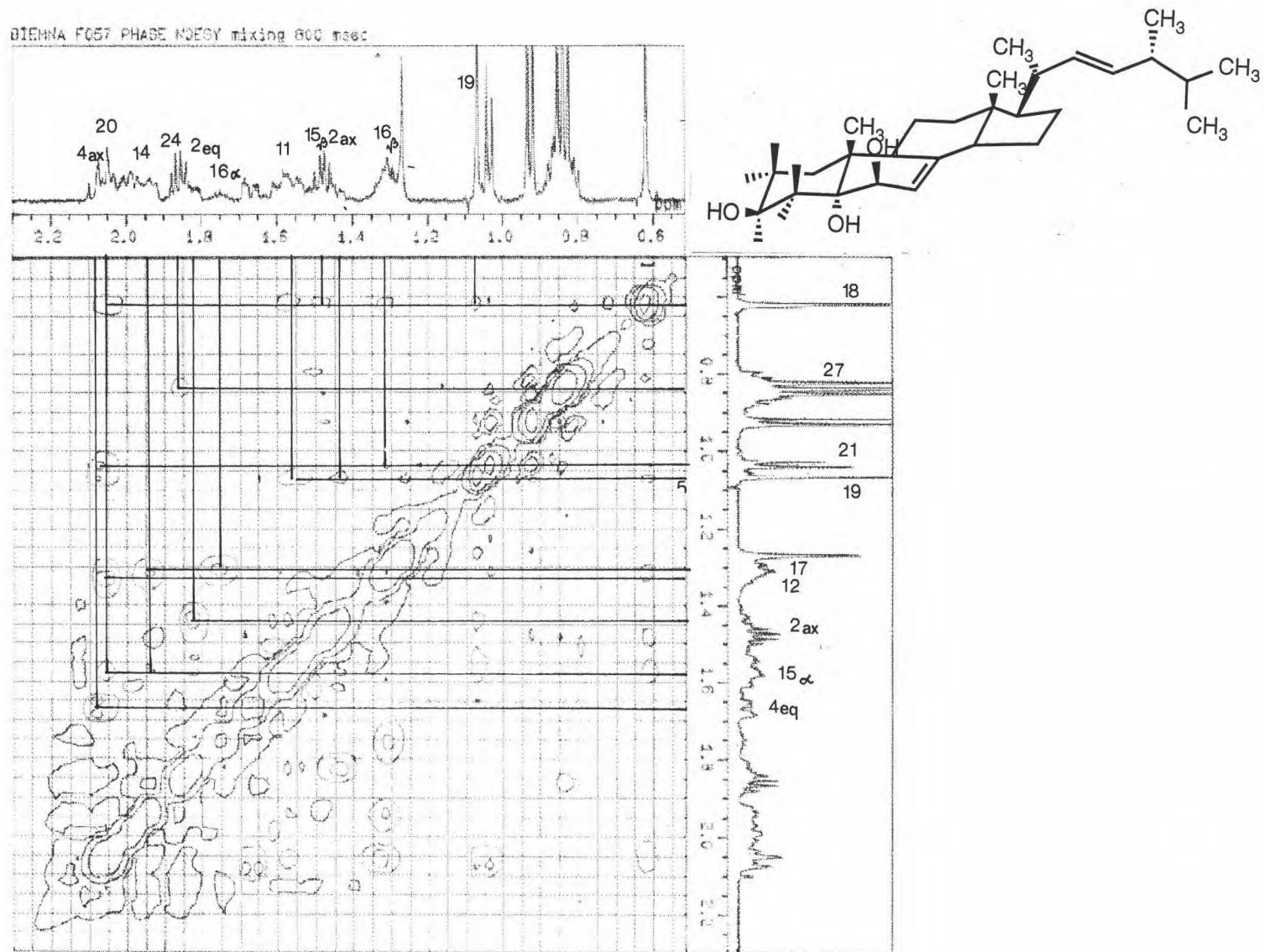


Figure 59. The partial NOESY spectrum ( $\delta_H$  0.6-2.2 ppm) of K057

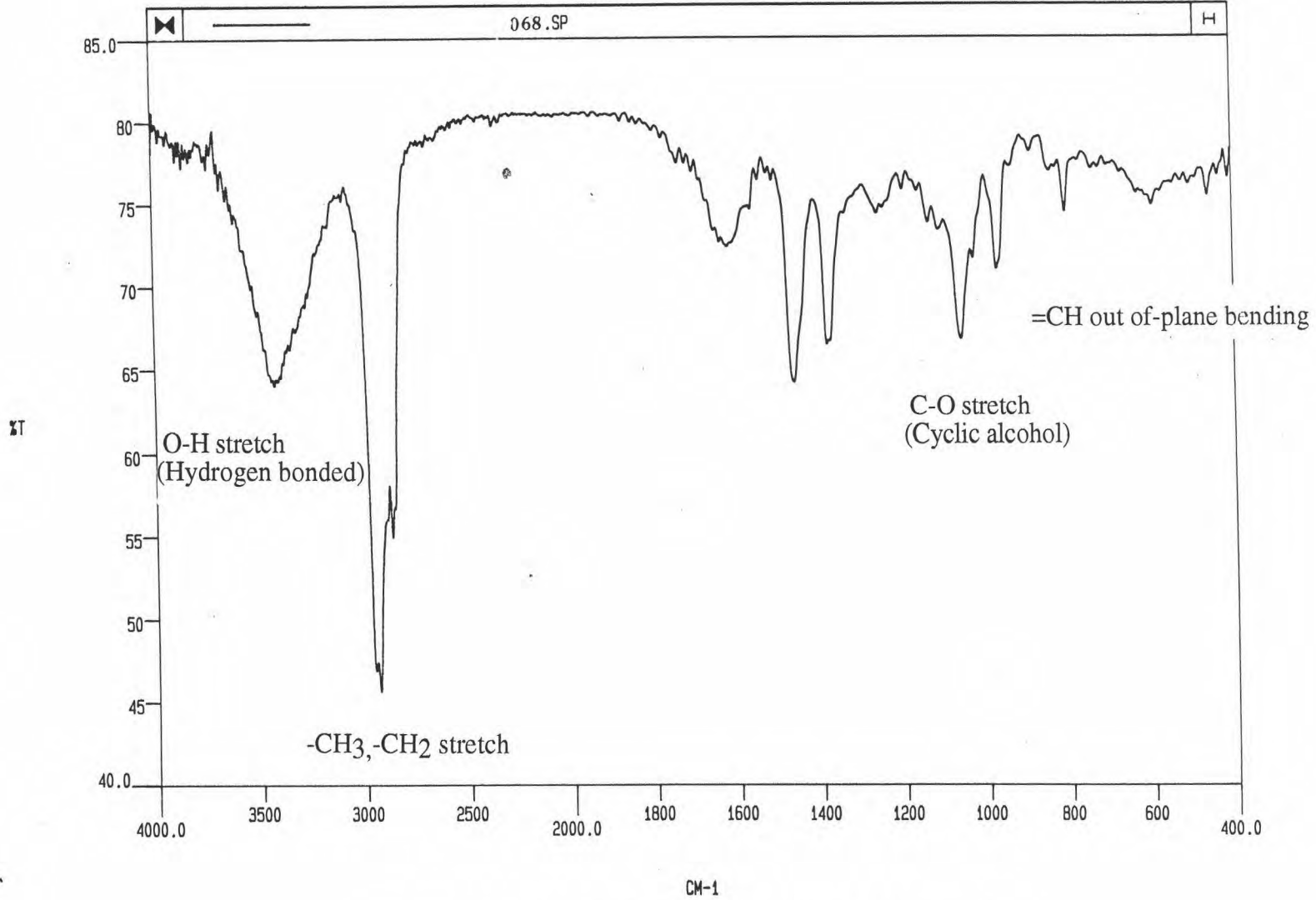


Figure 60. The IR spectrum of K068 (KBr disc)

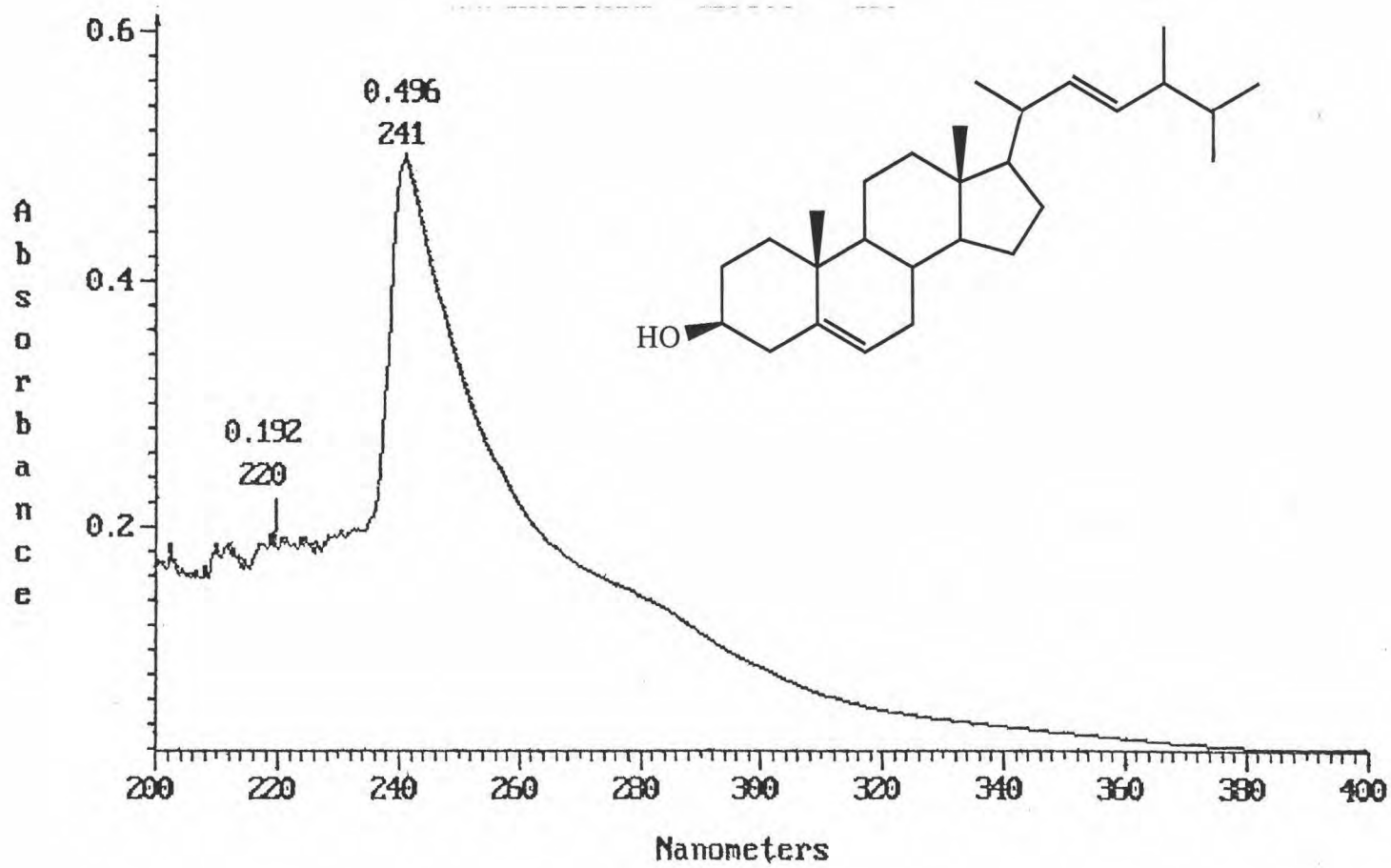


Figure 61. The UV spectrum of K068 in chloroform.

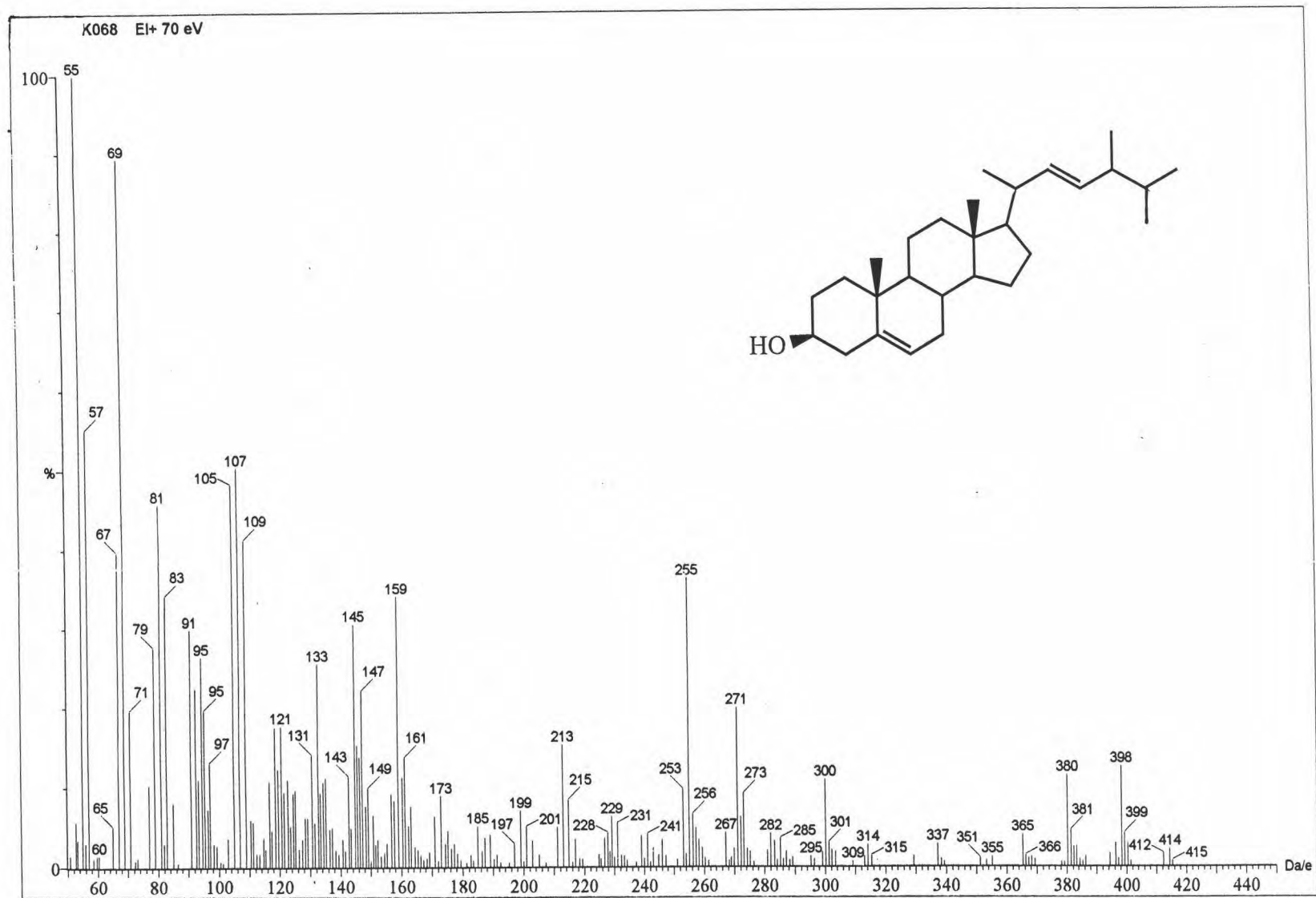


Figure 62. The EIMS spectrum of K068.



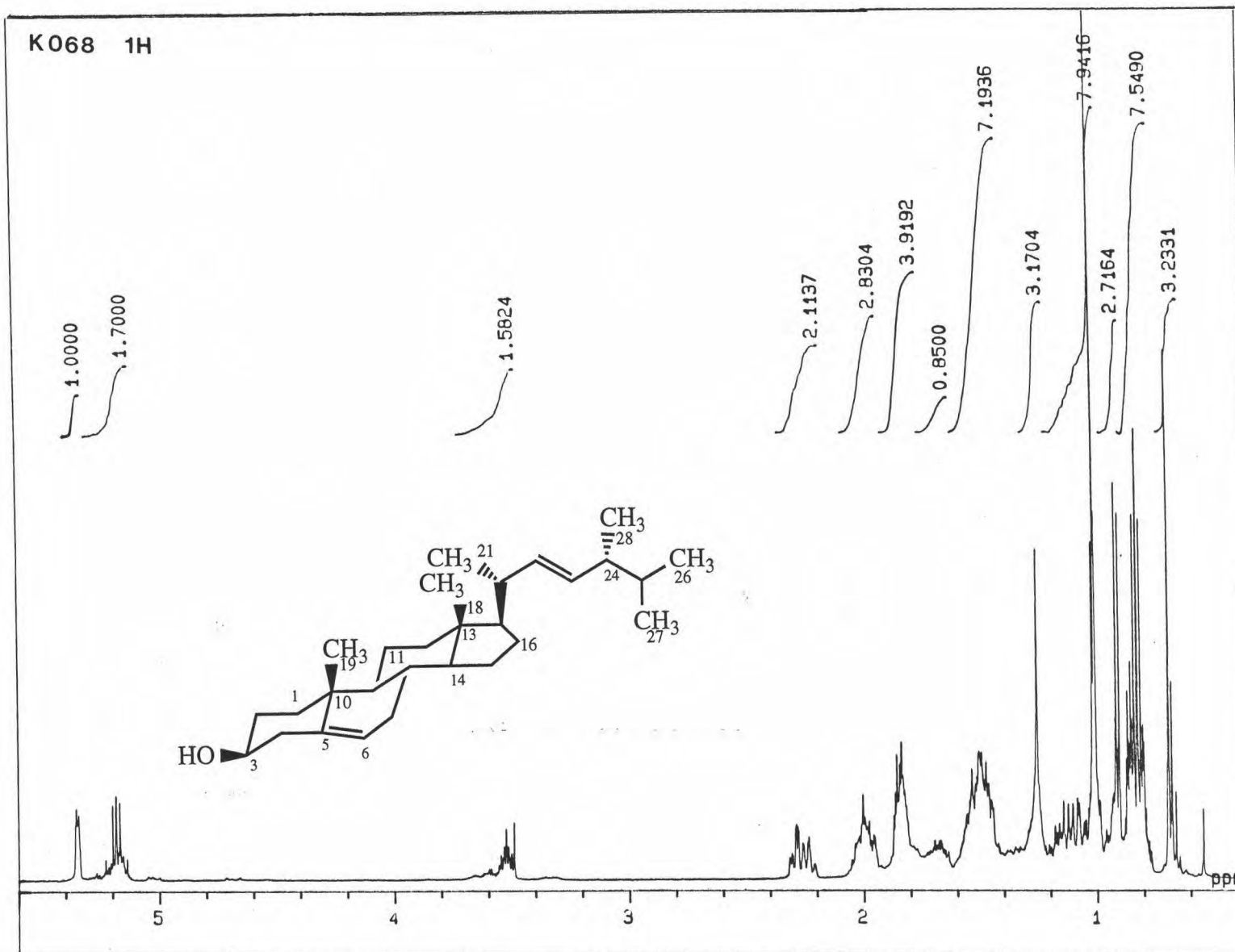


Figure 63. The 500 MHz <sup>1</sup>H NMR spectrum of K068 in CDCl<sub>3</sub>

1H SINGL NOM K068

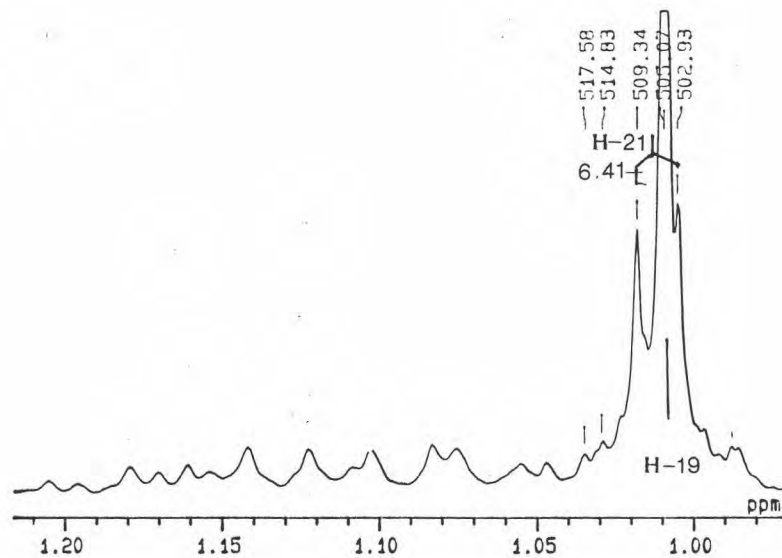
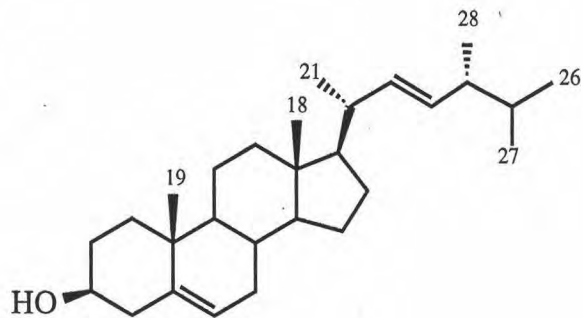
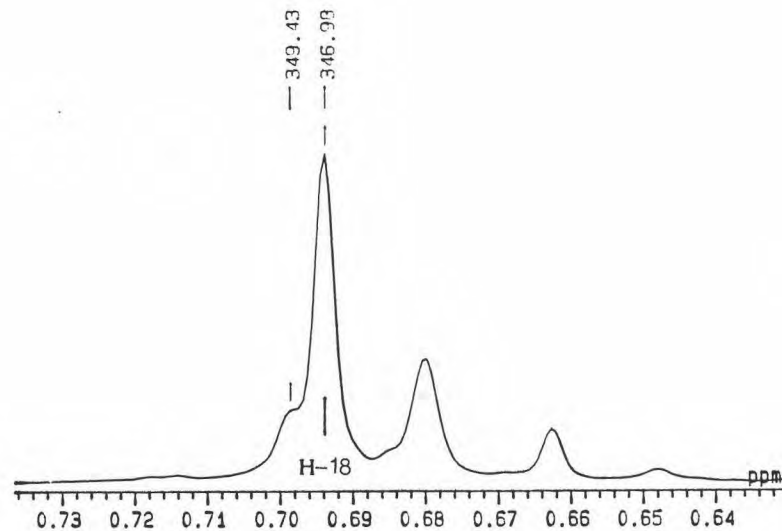
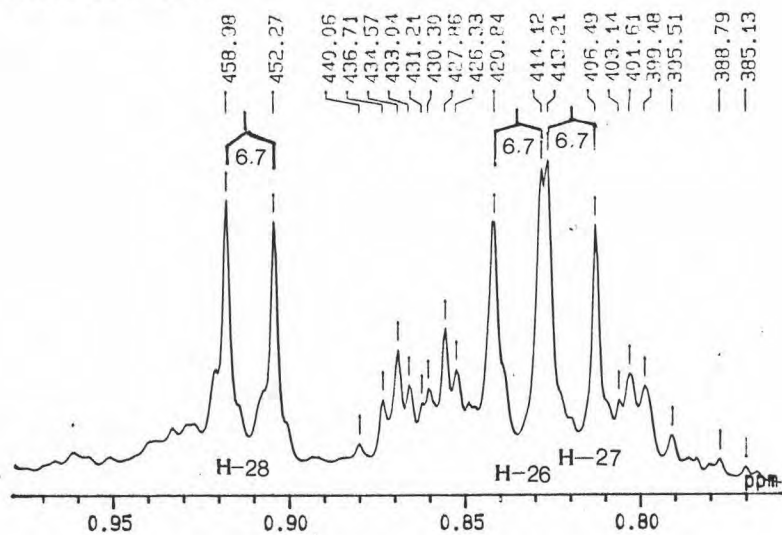


Figure 64. The 500 MHz  $^1\text{H}$  NMR spectrum of K068 in  $\text{CDCl}_3$  (expanded)

<sup>1</sup>H SINGL NON K.068

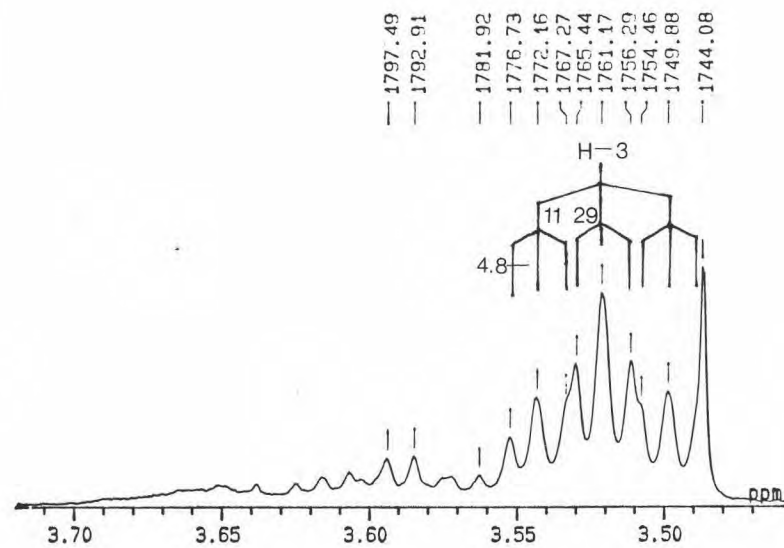
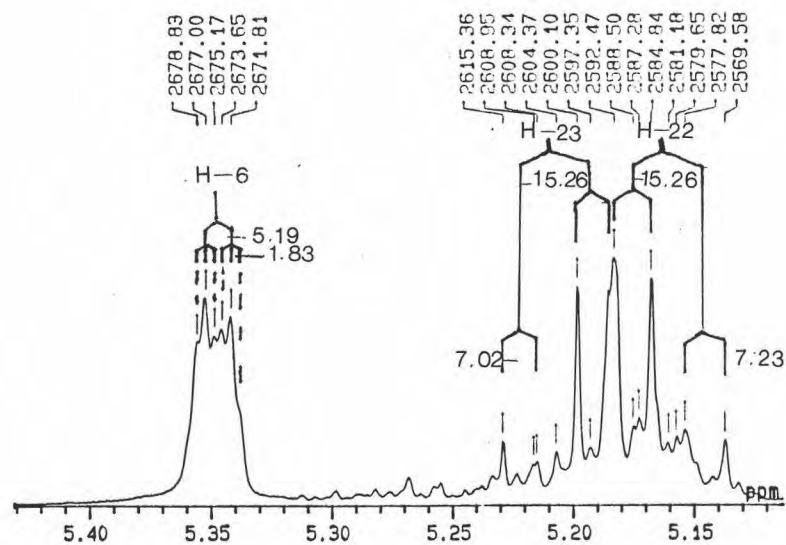
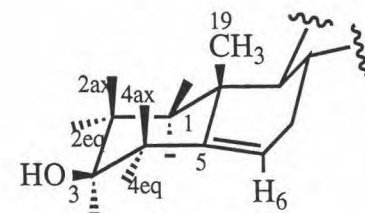
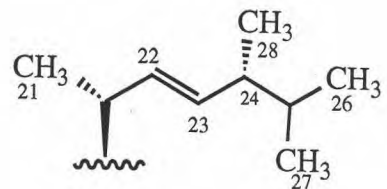
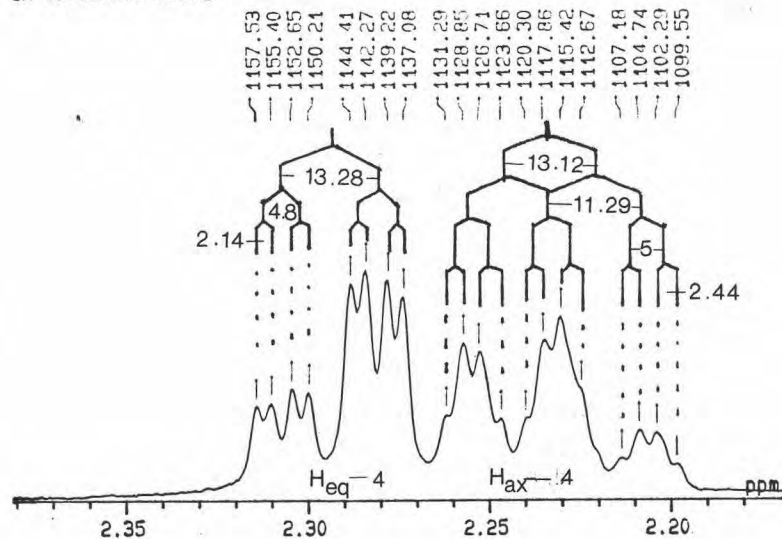


Figure 65. The 500 MHz <sup>1</sup>H NMR spectrum of K068 in CDCl<sub>3</sub> (expanded)

BIEMNA K068 13C

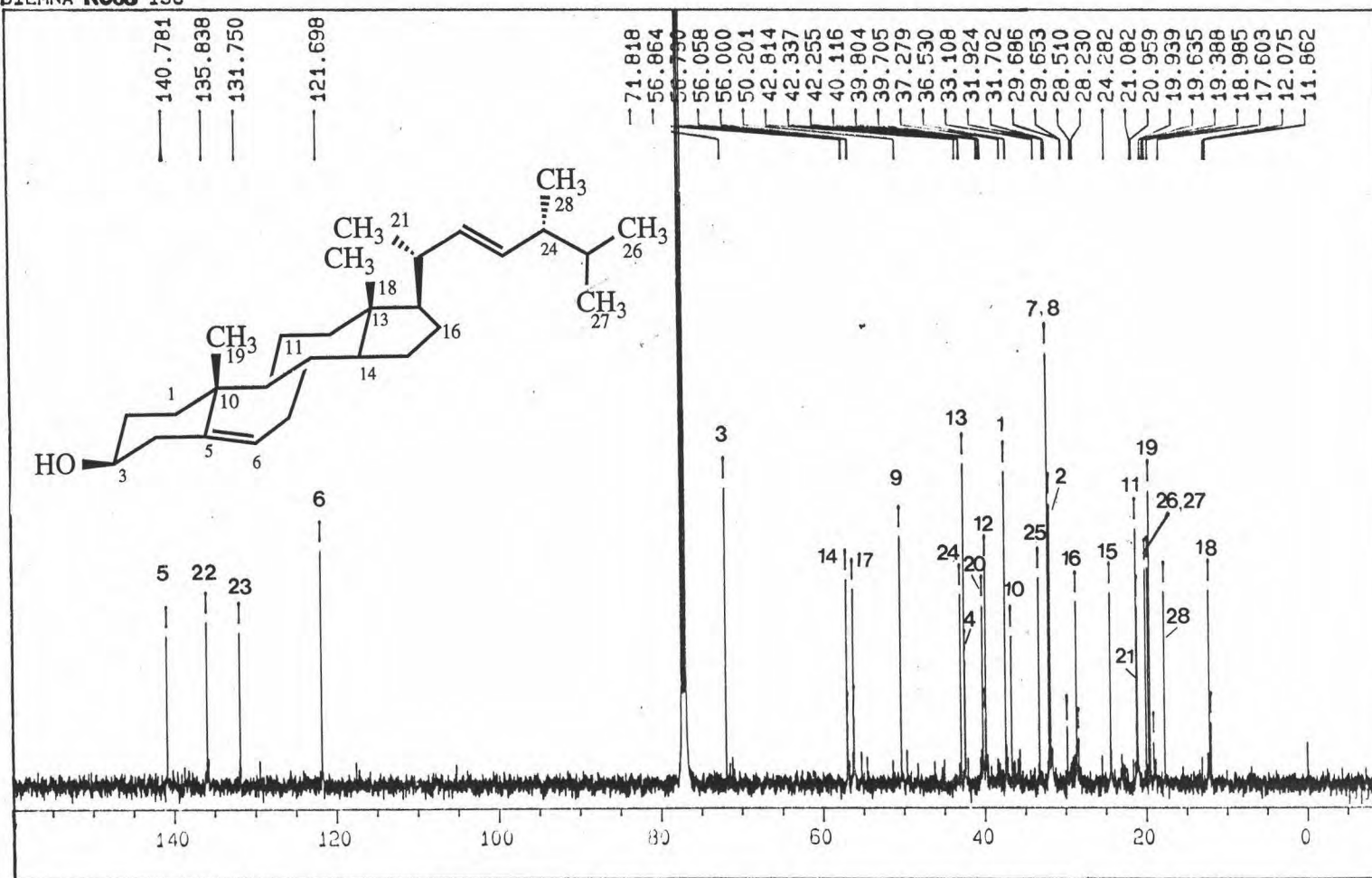


Figure 66. The 125 MHz  $^{13}\text{C}$  NMR spectrum of K068 in  $\text{CDCl}_3$

K068CHSHF

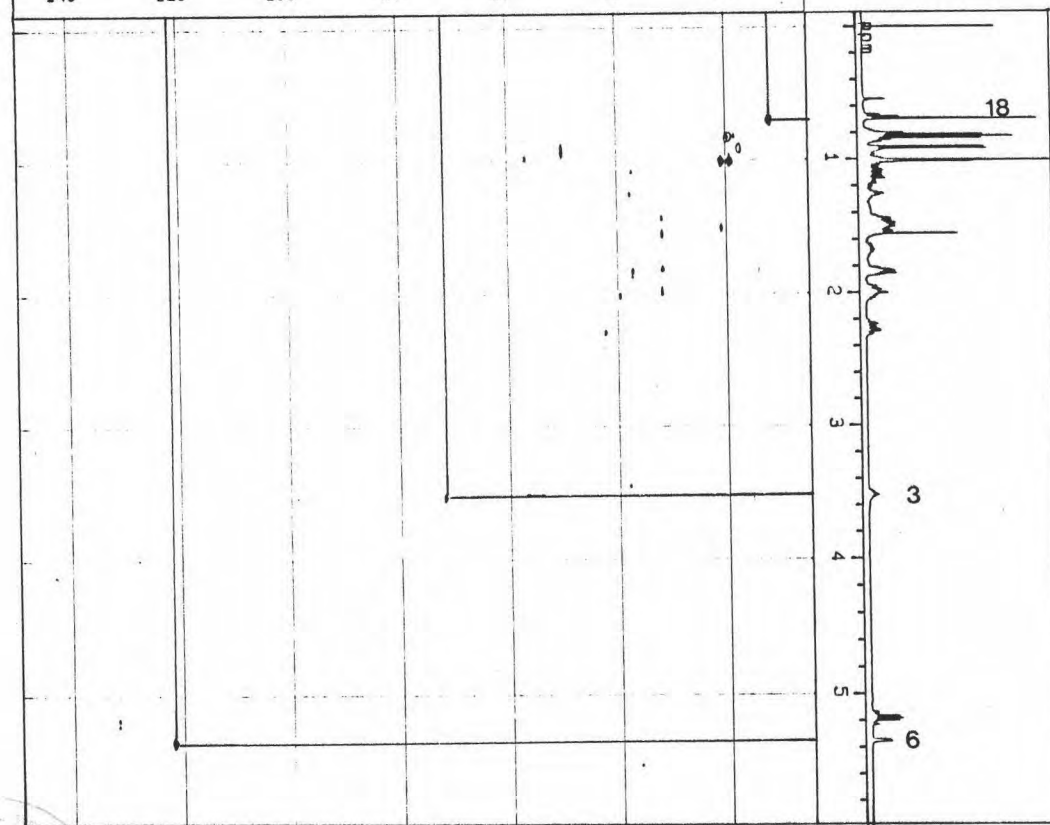
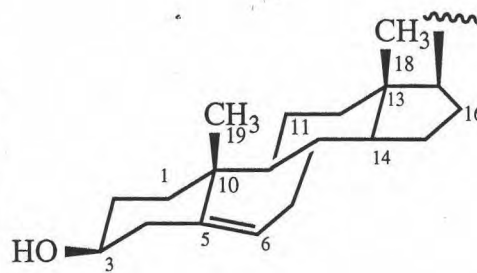
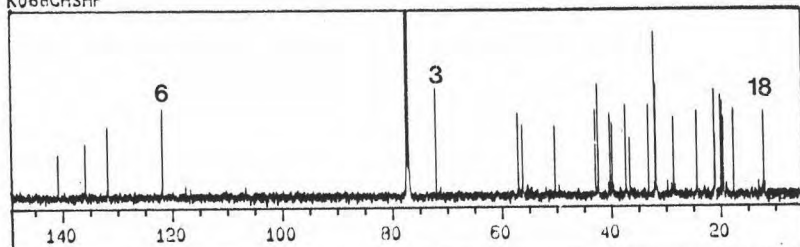


Figure 67. The CHSHF spectrum of K068.



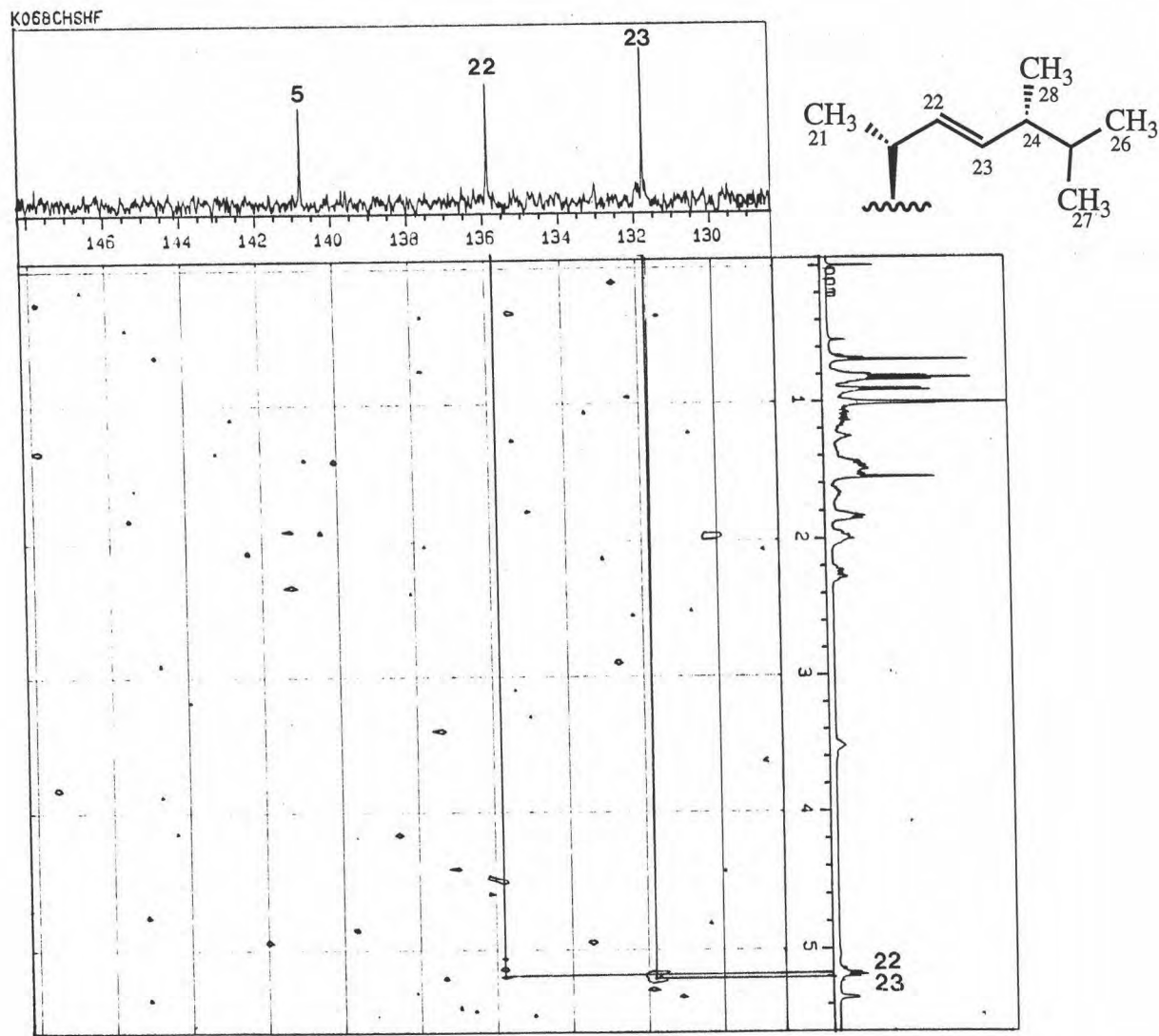


Figure 68. The partial CHSHF spectrum ( $\delta_C$  130-137 ppm) of K068

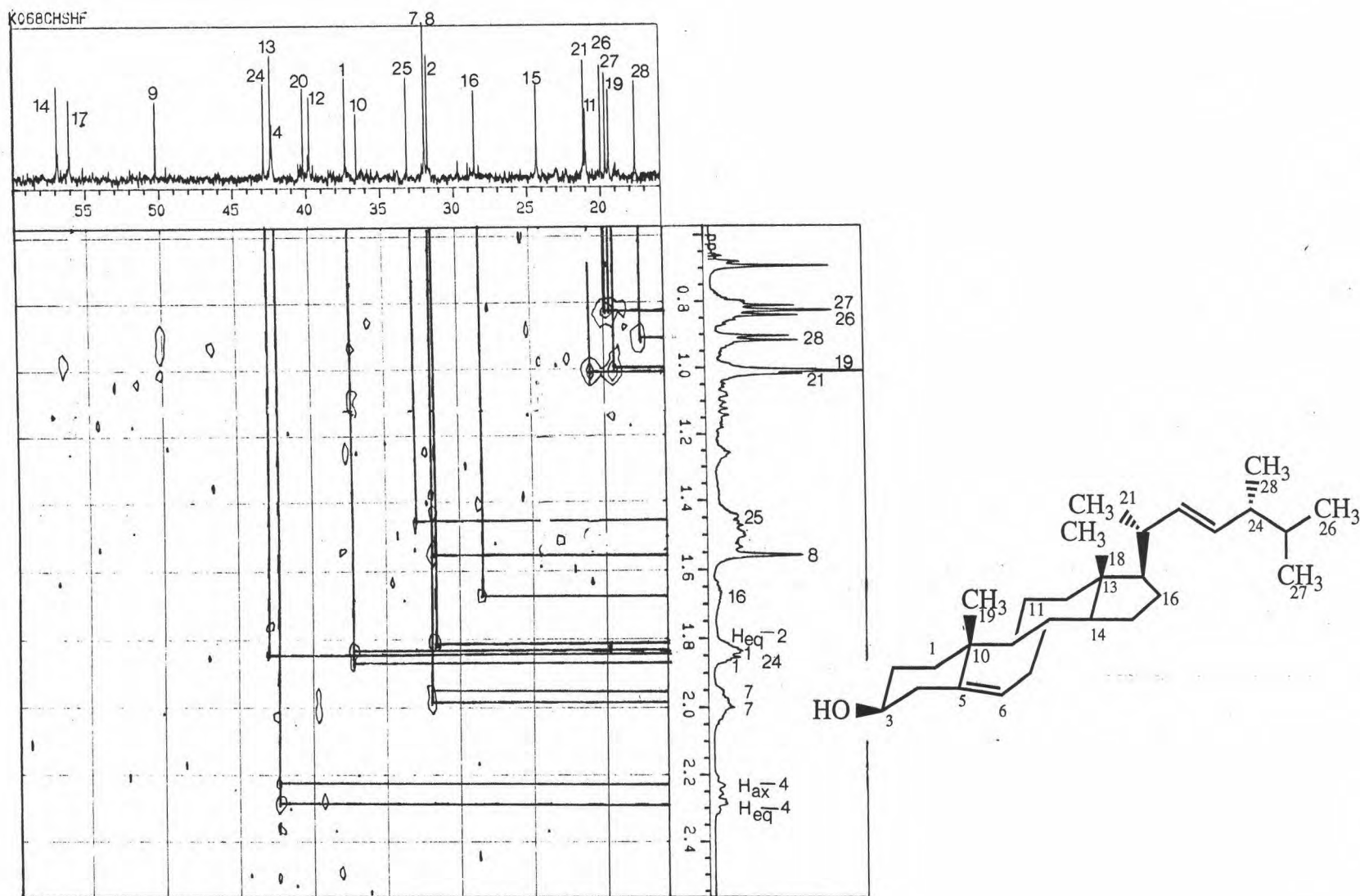


Figure 69. The partial CHSHF spectrum ( $\delta_C$  15-60 ppm) of K068

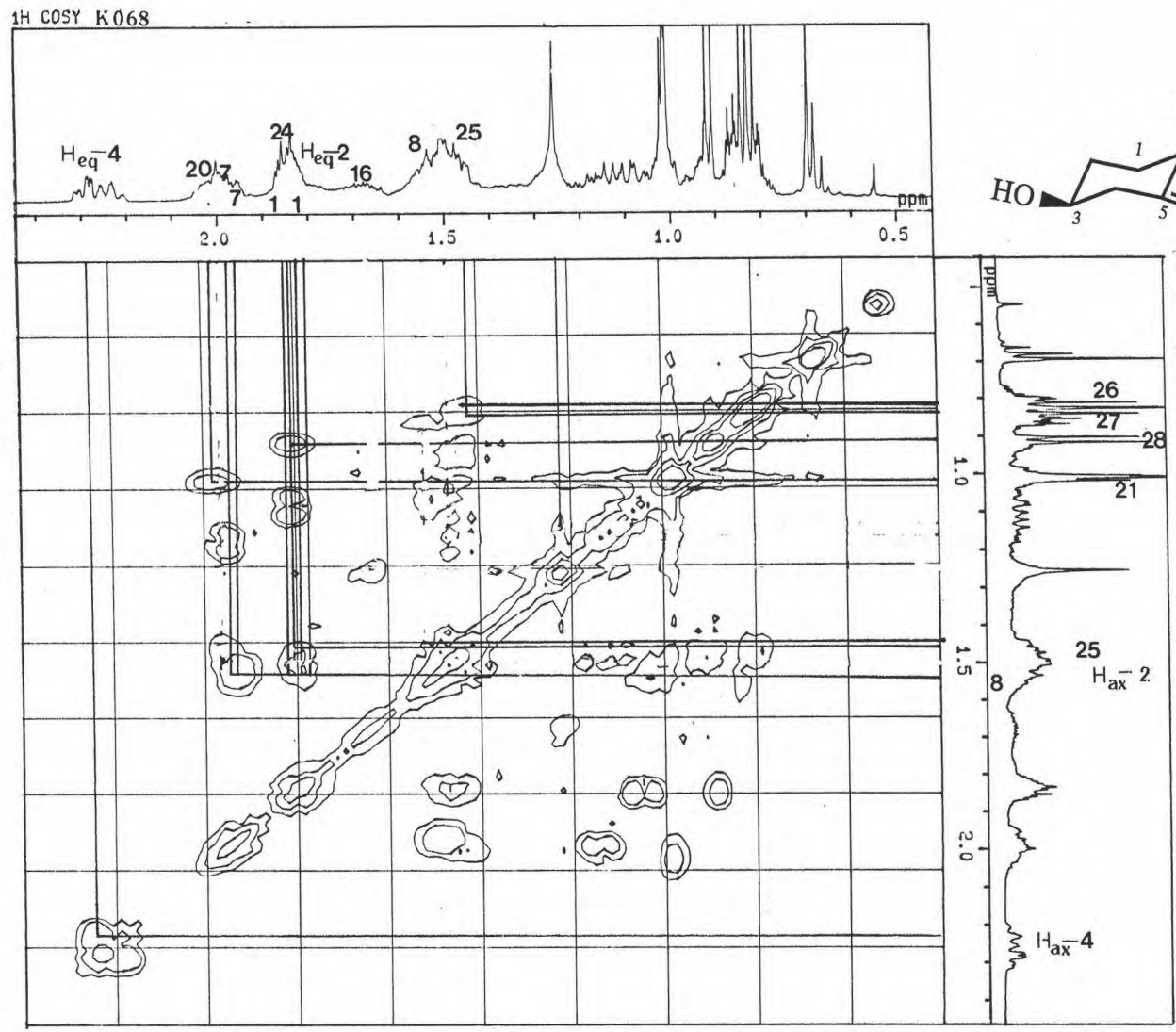


Figure 70. The  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of K068



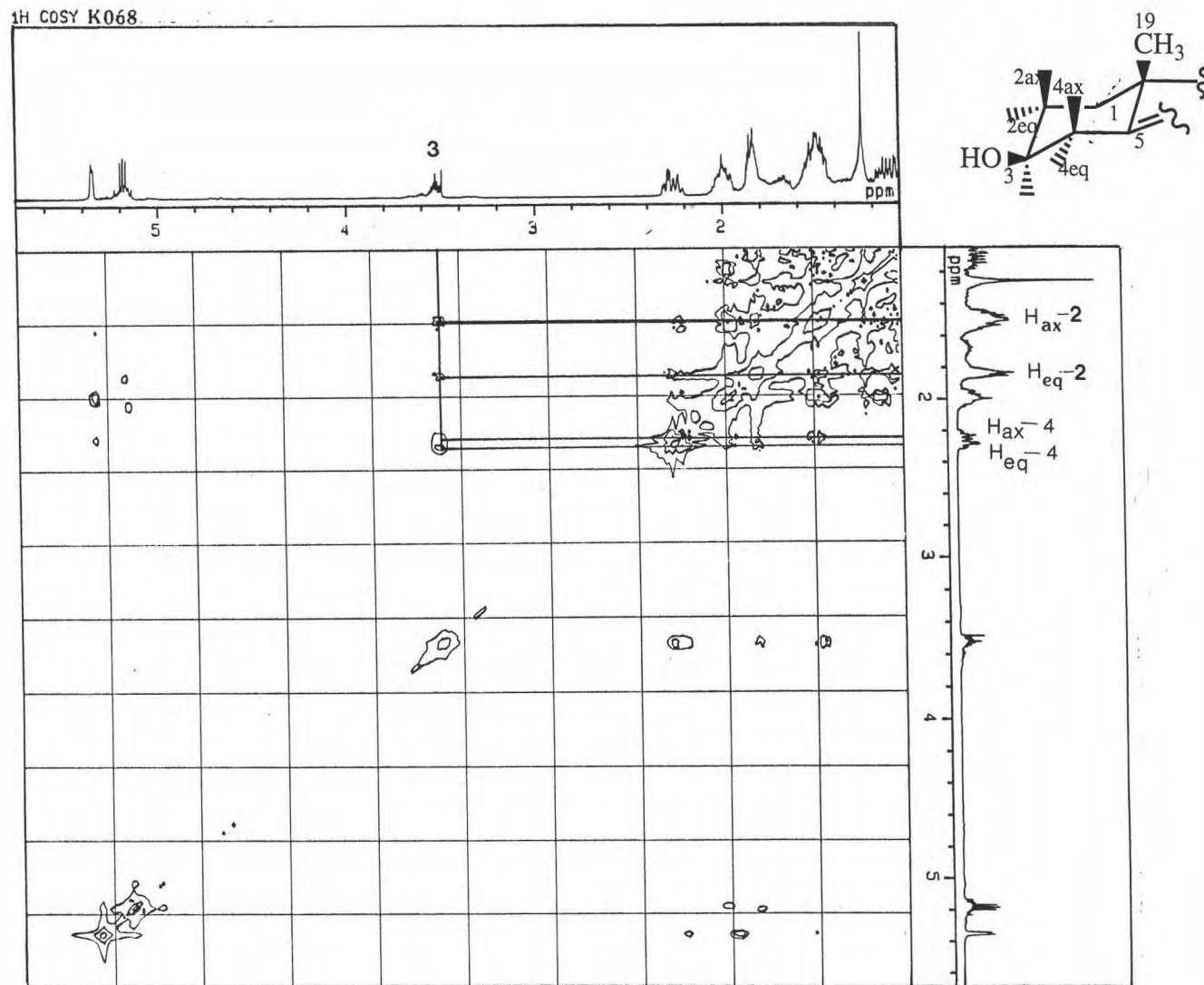


Figure 71. The partial <sup>1</sup>H-<sup>1</sup>H COSY spectrum ( $\delta_{\text{H}}$  0.6-2.2 ppm) of K068.

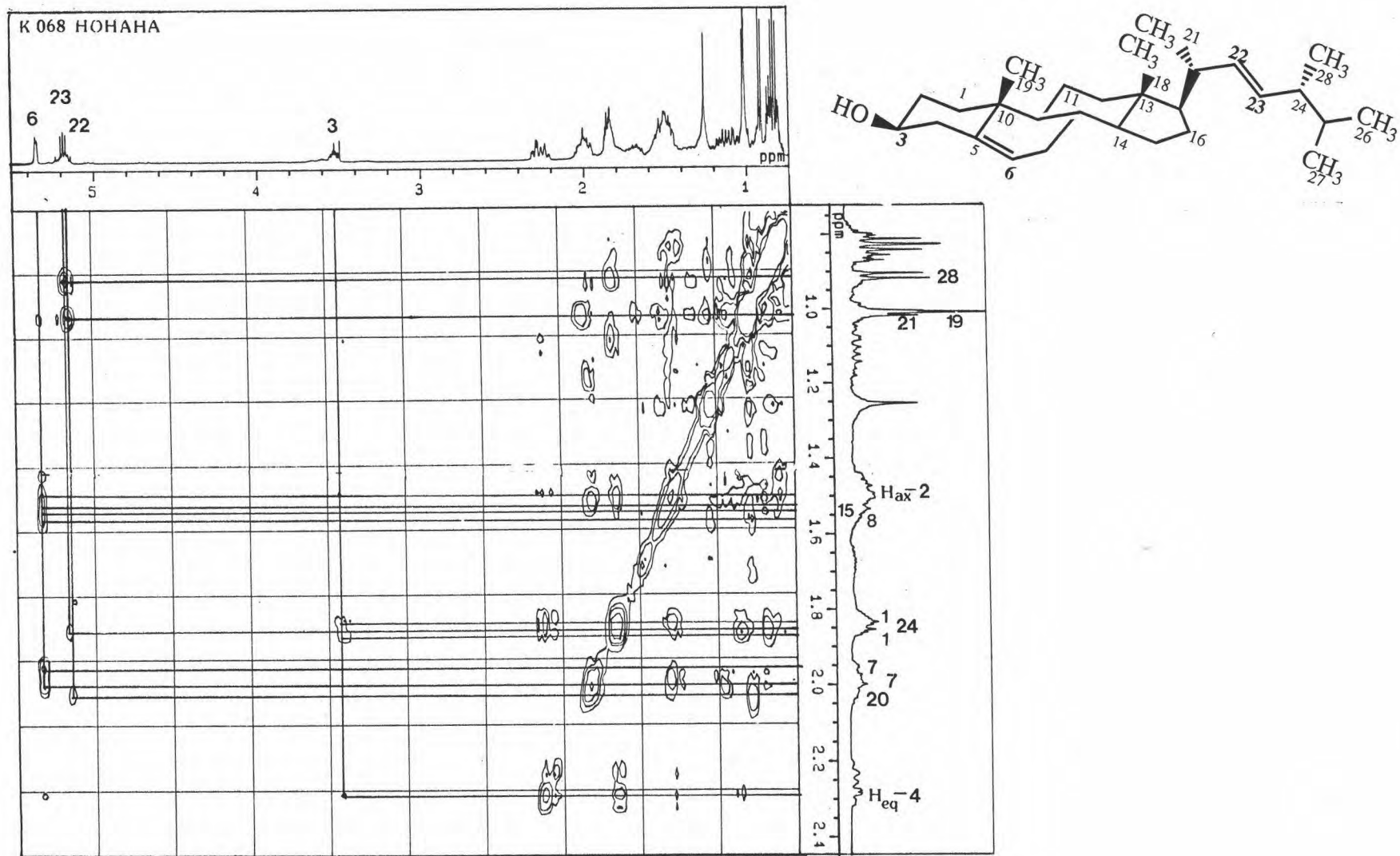


Figure 72. The HOHAHA spectrum of K068

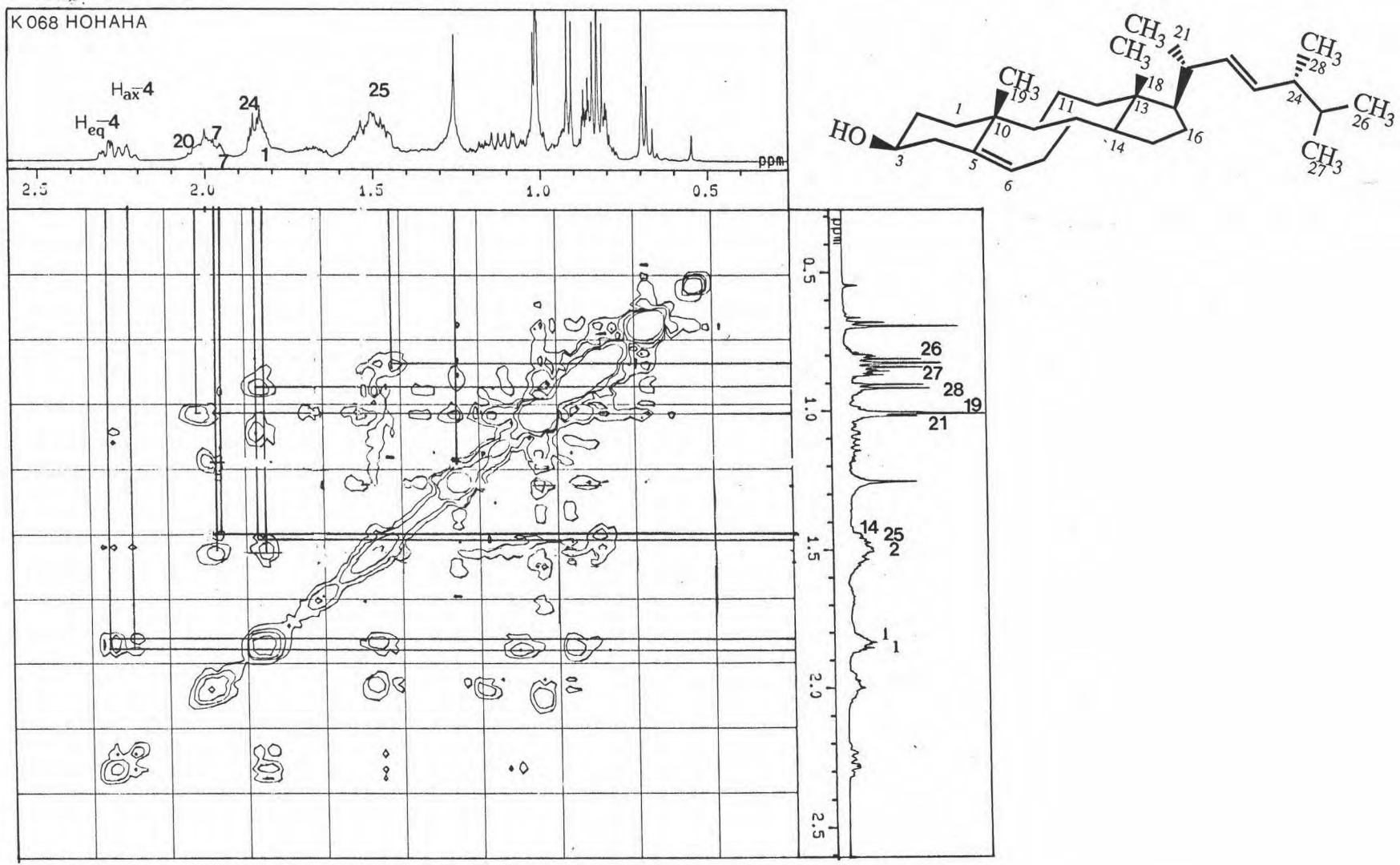


Figure 73. The partial HOHAHA spectrum ( $\delta_H$  0.6-2.2 ppm) of K068

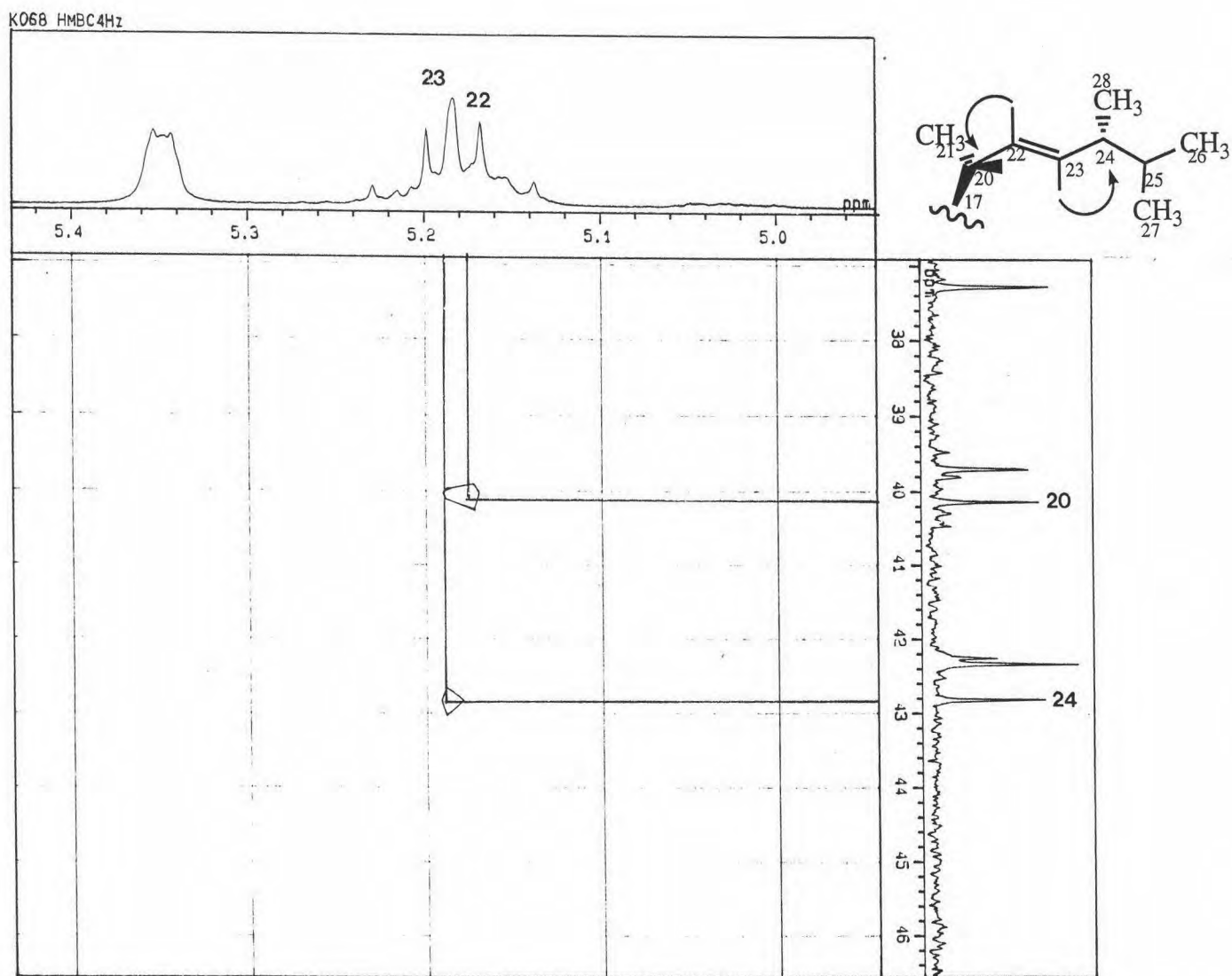


Figure 74. The partial HMBC spectrum ( $\delta_H$  5.0-5.4 ppm) of K068 ( $^3J_{CH} = 4$  Hz)

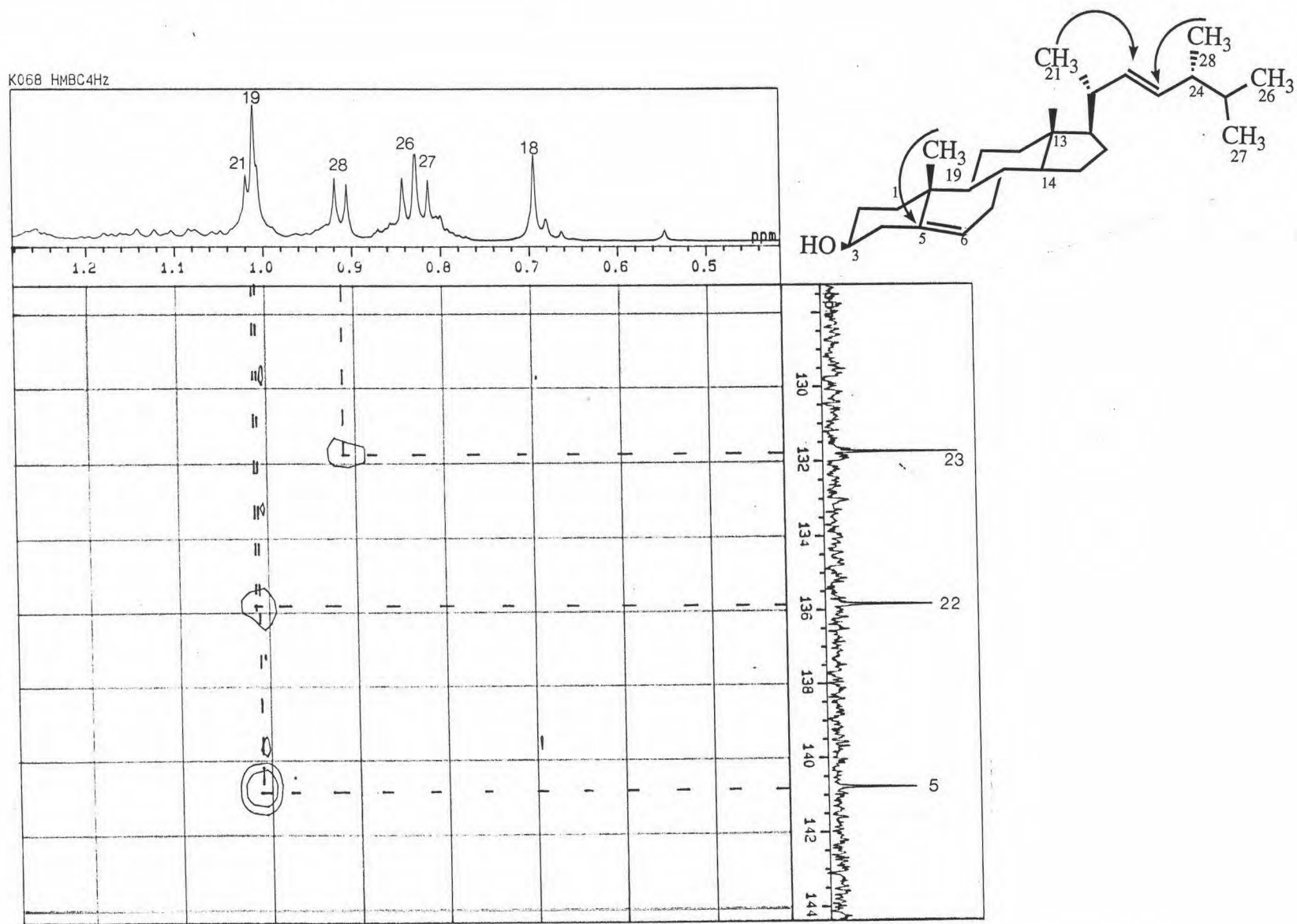


Figure 75. The partial HMBC spectrum ( $\delta_{\text{H}}$  0.5-1.2 ppm) of K068 ( $^3J_{\text{CH}} = 4$  Hz)

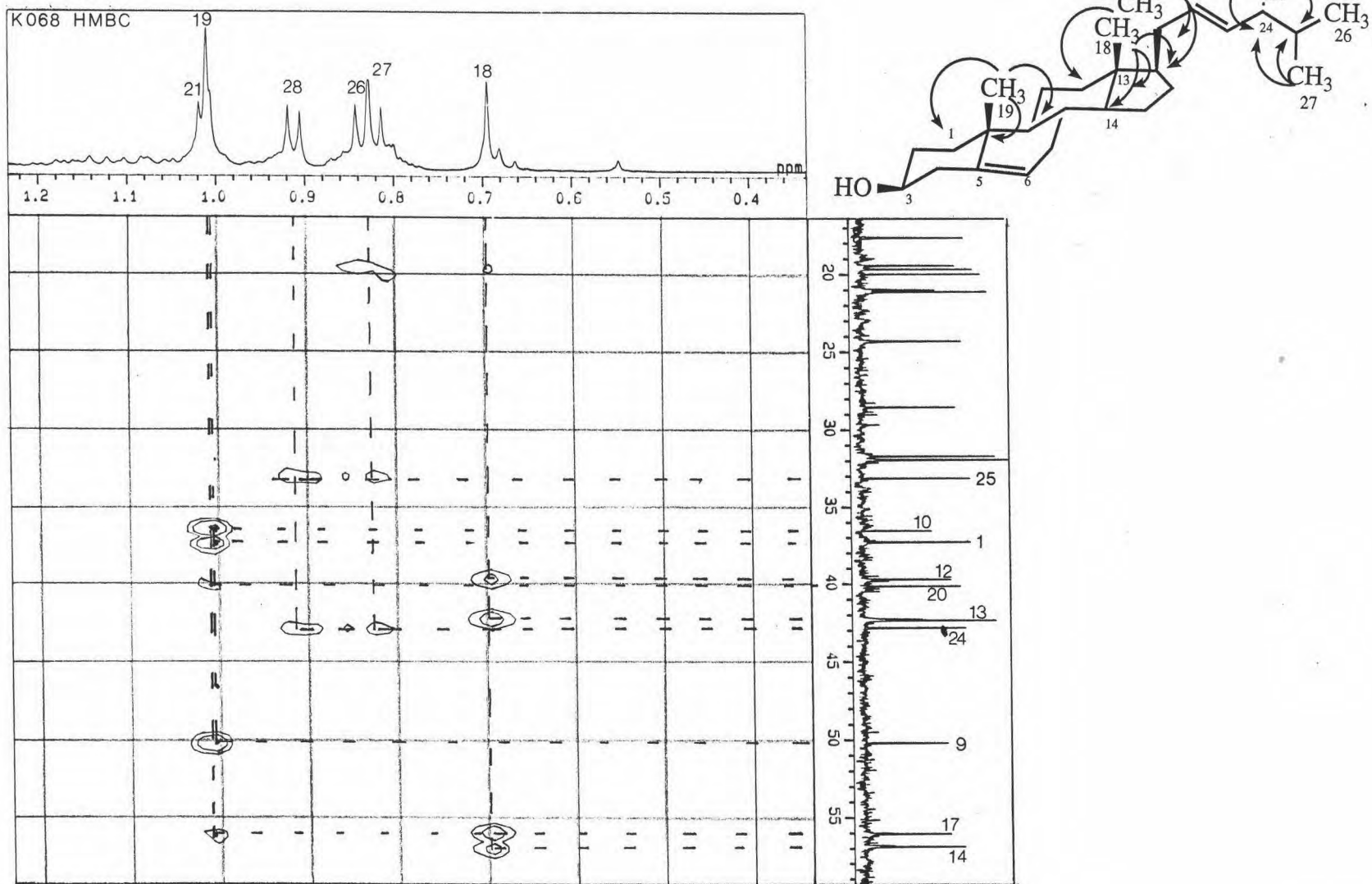
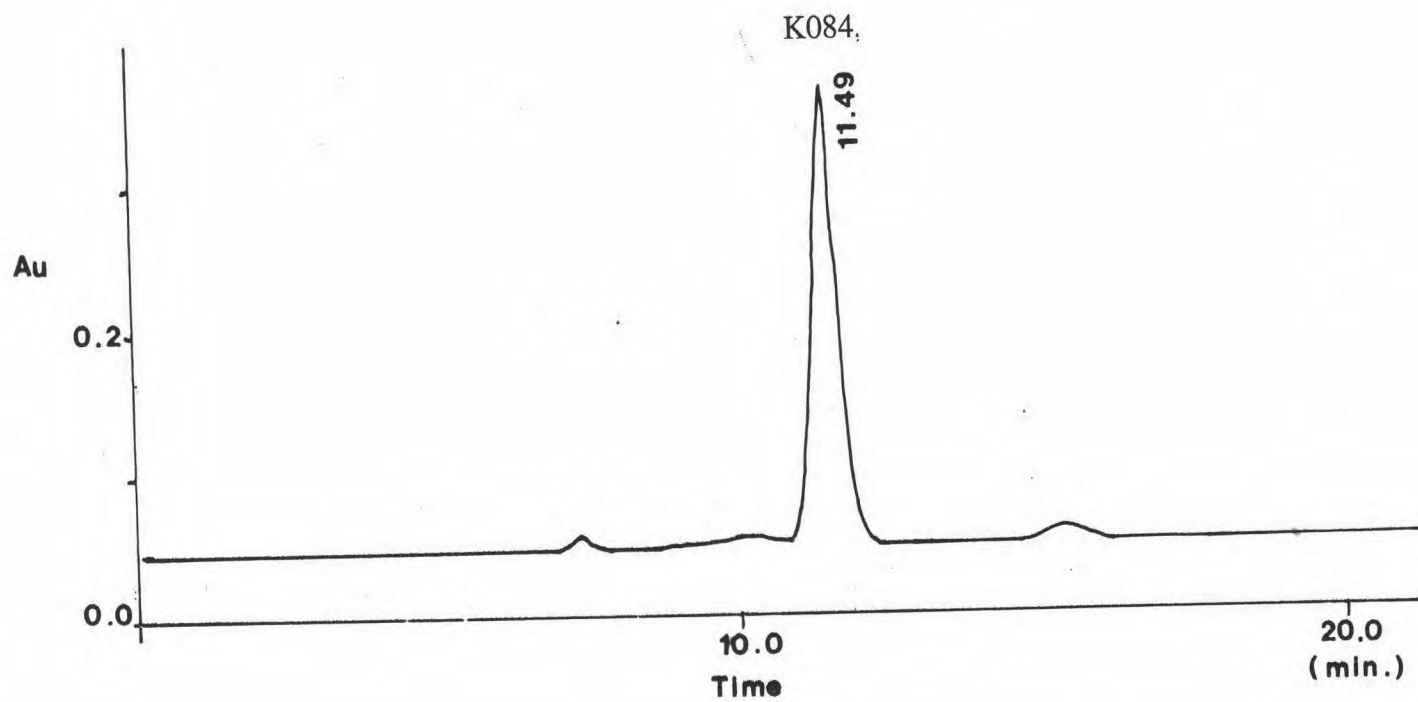
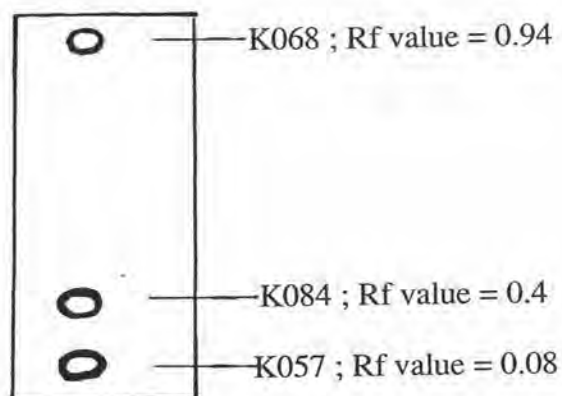


Figure 76. The partial HMBC spectrum ( $\delta_H$  0.4-1.2 ppm) of K068 ( $^3J_{CH} = 4$  Hz)

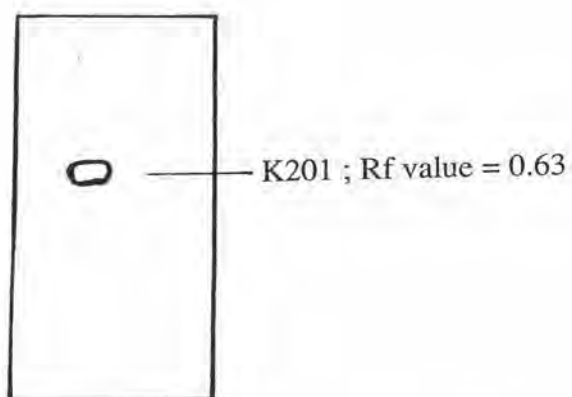


Normal phase column (125 x 4 mm)  
Flow rate 1 ml/min  
detected at  $\lambda_{\max}$  250 nm

Figure 77. The HPLC chromatogram of K084  
(2% CH<sub>3</sub>OH in CHCl<sub>3</sub>)



The TLC pattern of K068, K084, K057 (95% CH<sub>3</sub>OH in CHCl<sub>3</sub>)



The RP2-TLC pattern of F201 (20% H<sub>2</sub>O in CH<sub>3</sub>OH)

Figure 78. The TLC pattern of K068, K084, K057 (5% CH<sub>3</sub>OH in CHCl<sub>3</sub>) and the RP2-TLC pattern of F201 (20% H<sub>2</sub>O in CH<sub>3</sub>OH)



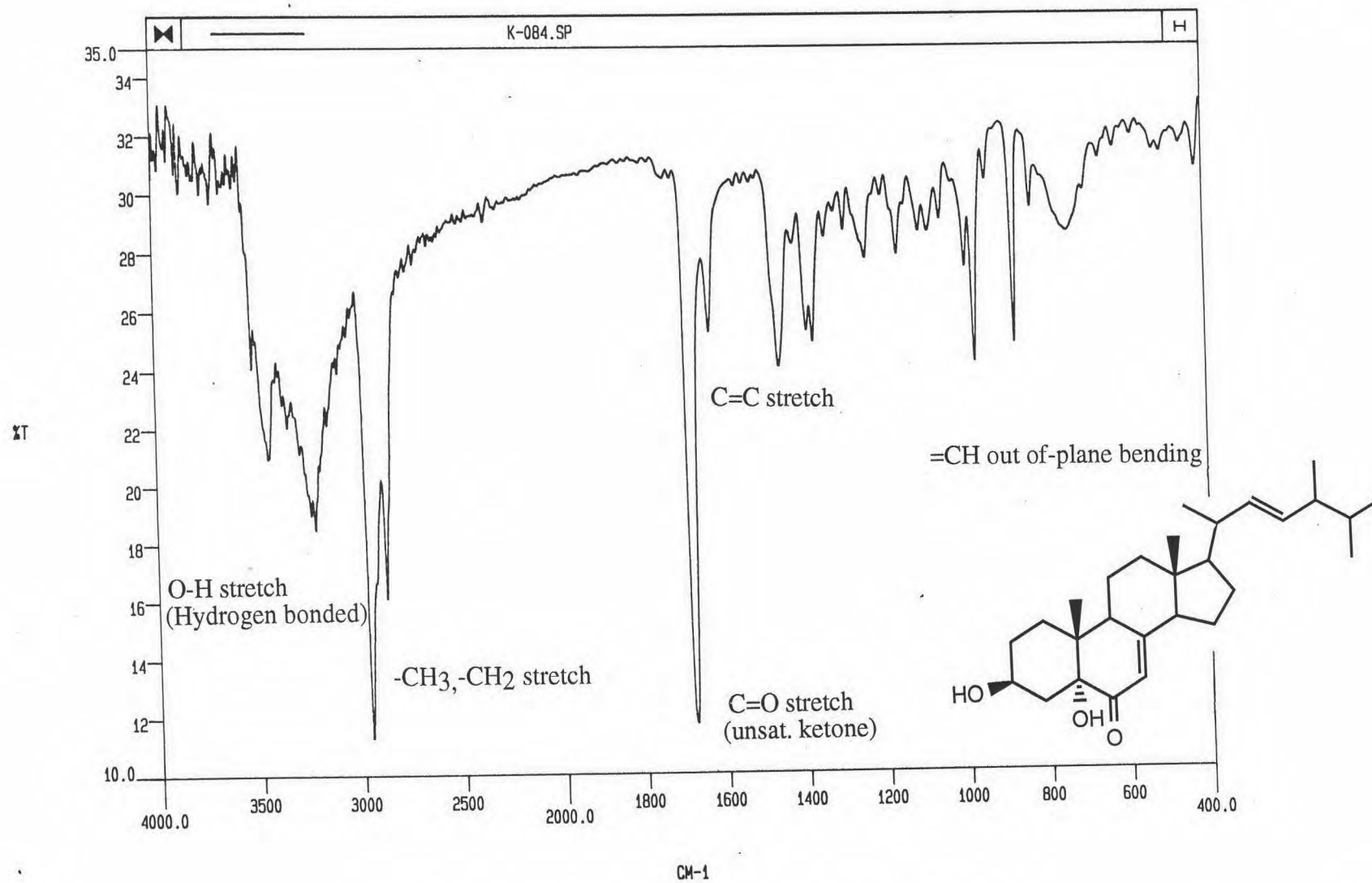


Figure 79. The IR spectrum of K084 (KBr disc)

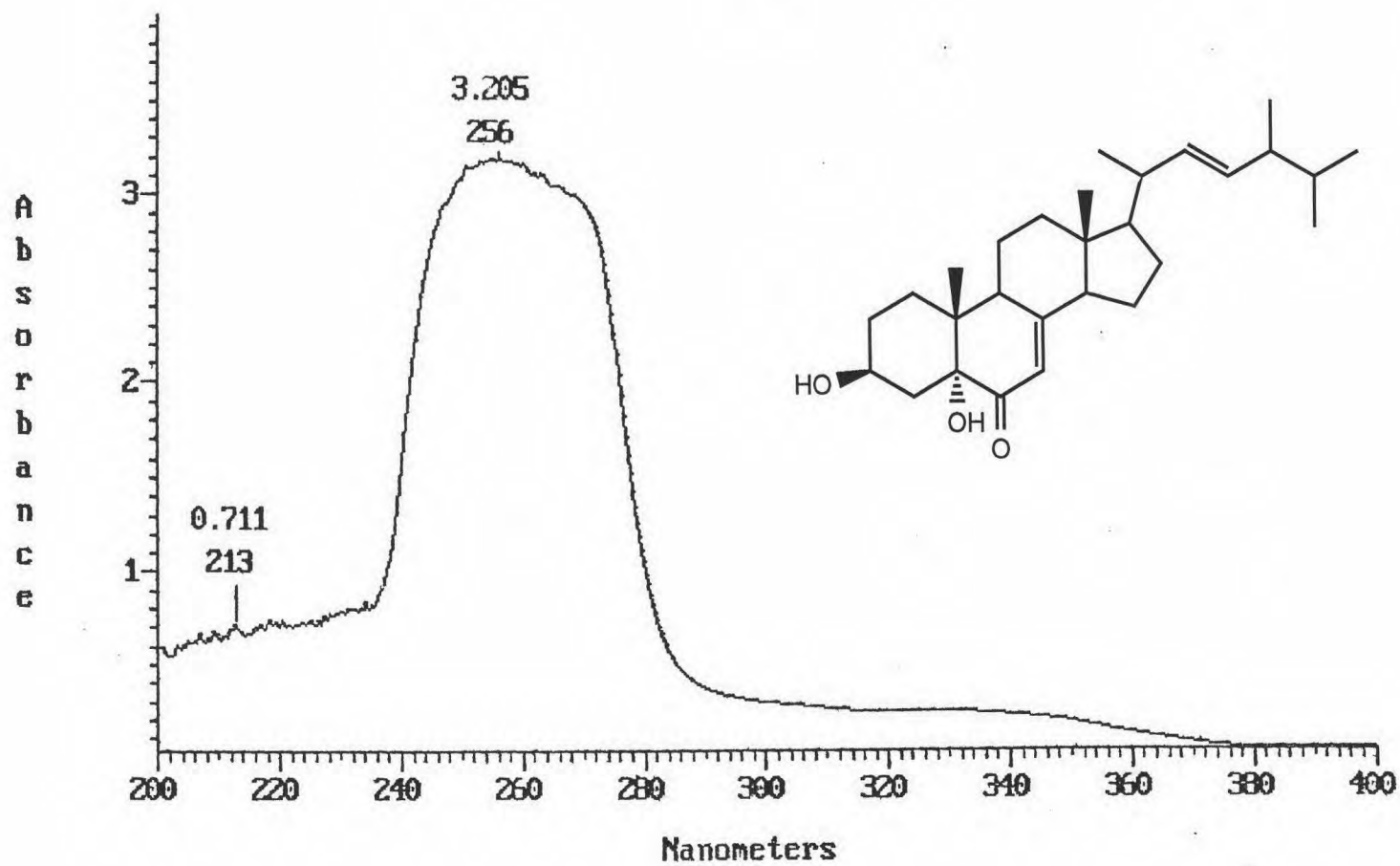


Figure 80. The UV spectrum of K084 in chloroform

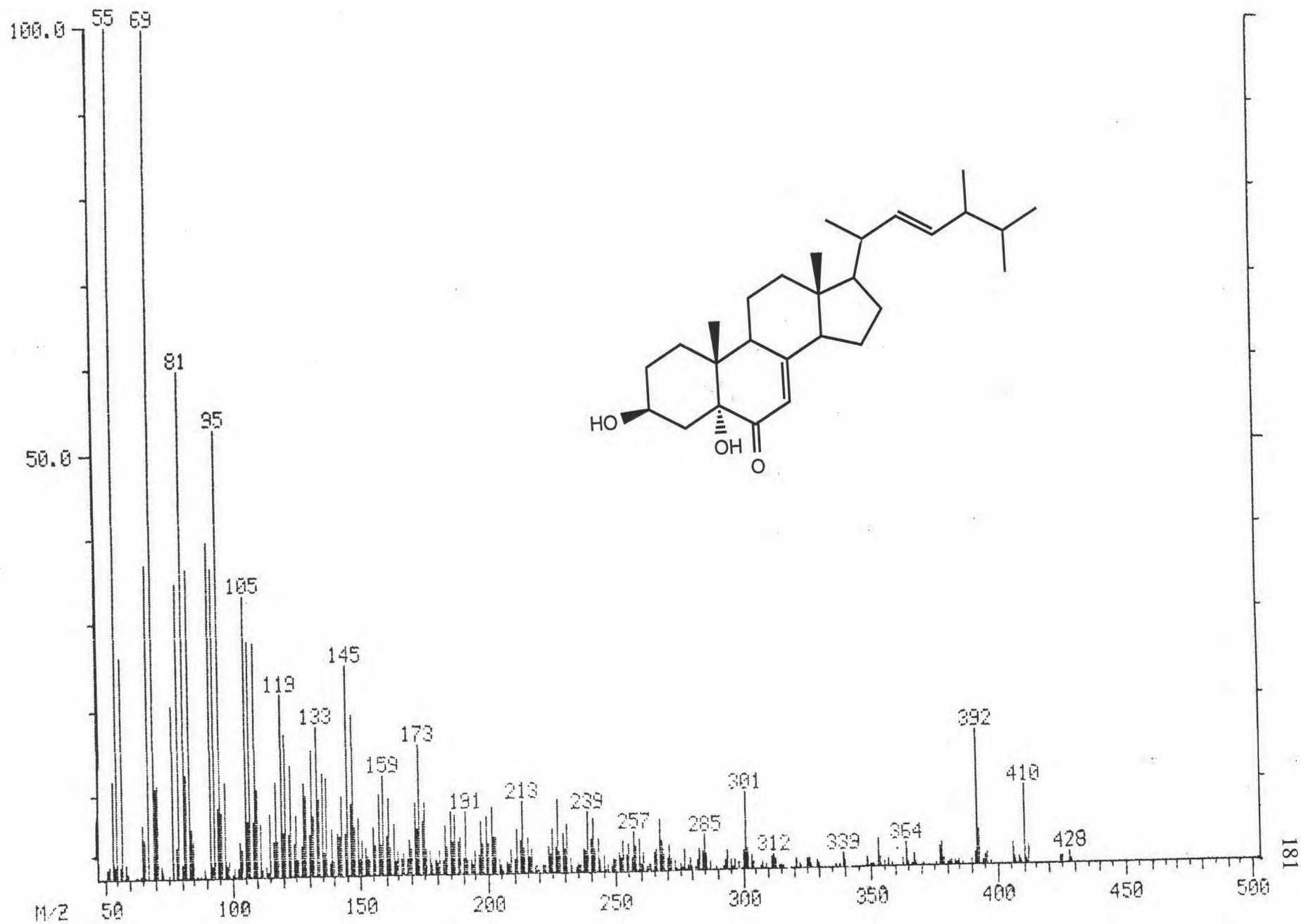


Figure 81. The EIMS spectrum of K084

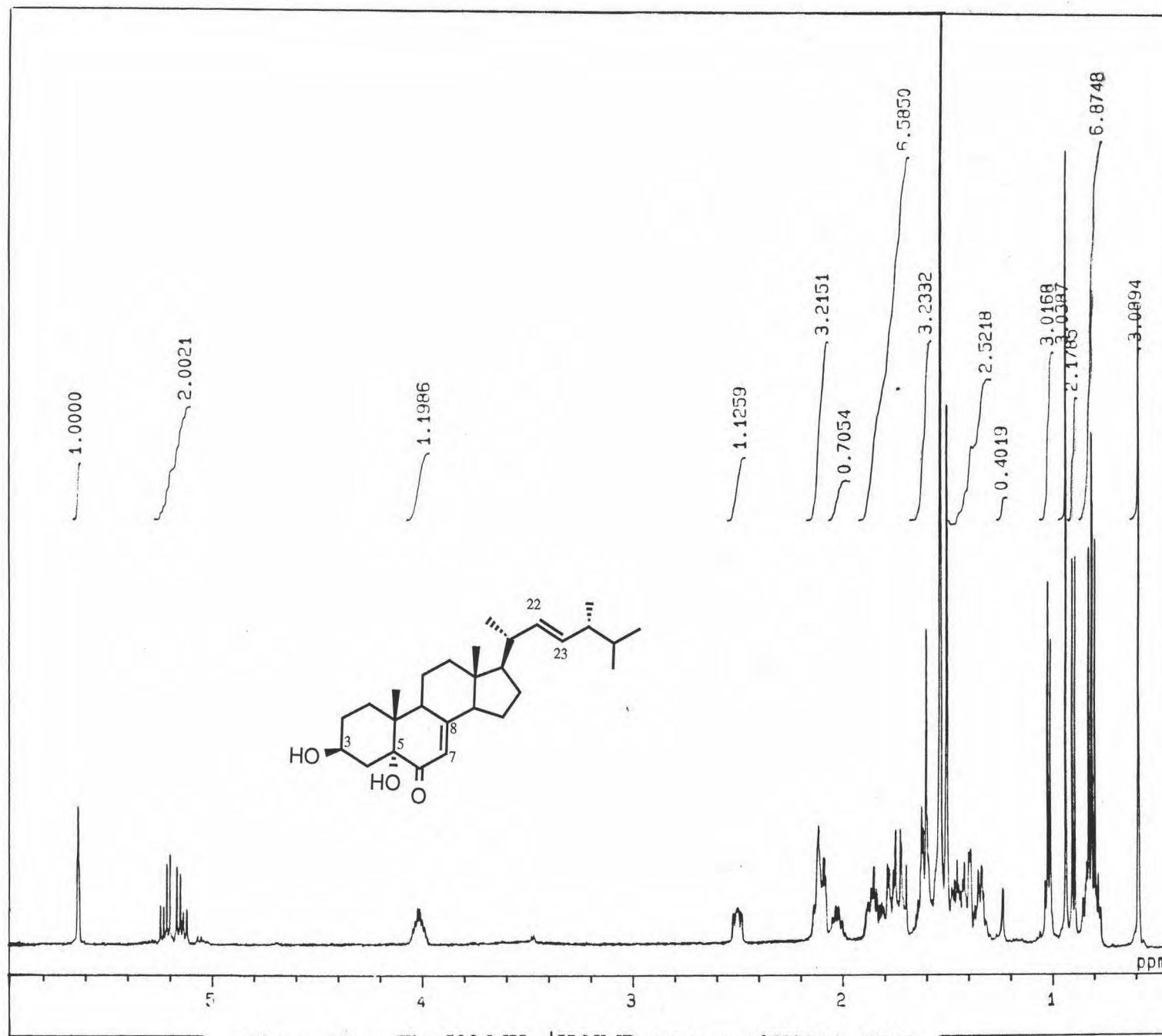


Figure 82. The 500 MHz  $^1\text{H}$  NMR spectrum of K084 in  $\text{CDCl}_3$

K084 IN MICROTUBE

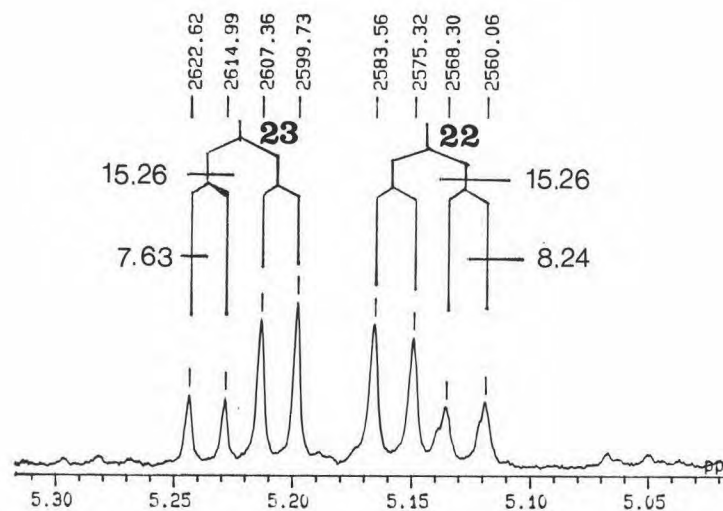
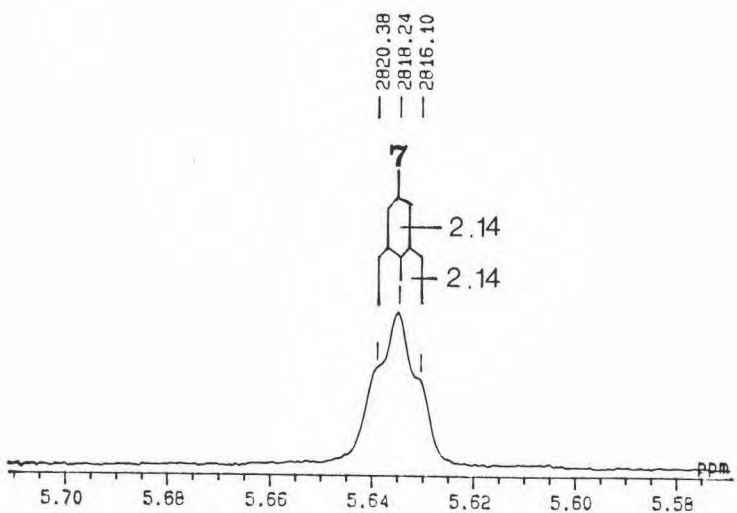
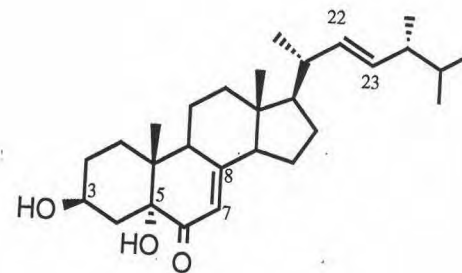
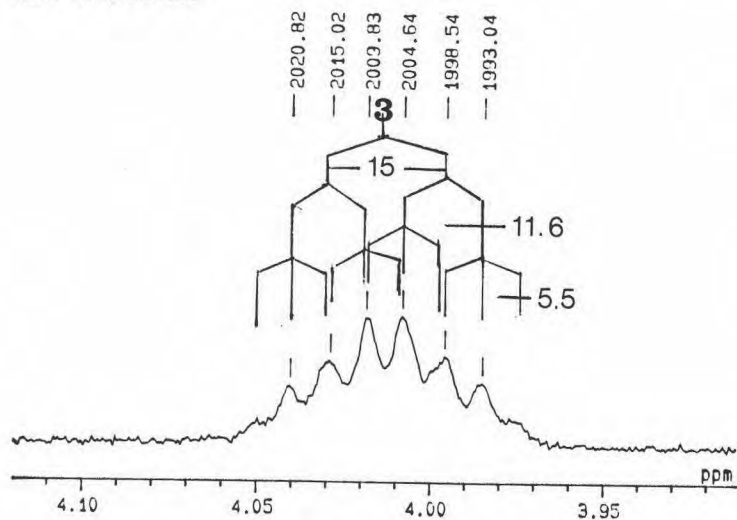


Figure 83. The 500 MHz  $^1\text{H}$  NMR spectrum of K084 in  $\text{CDCl}_3$  (expanded)

K084 IN MICROTUBE

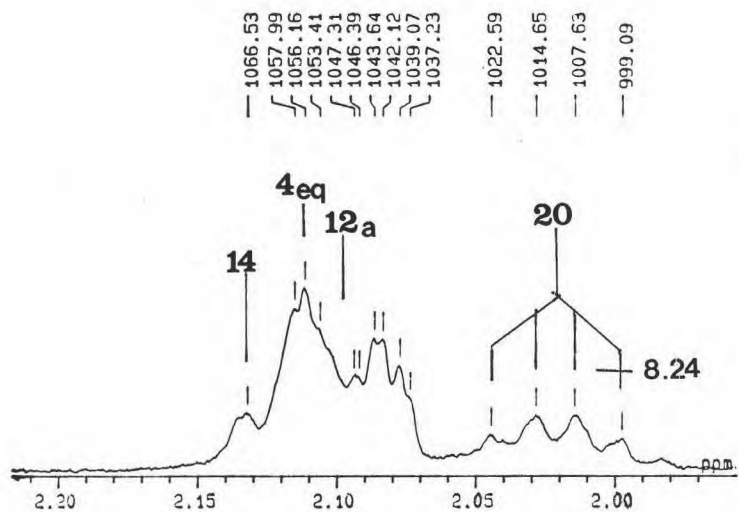
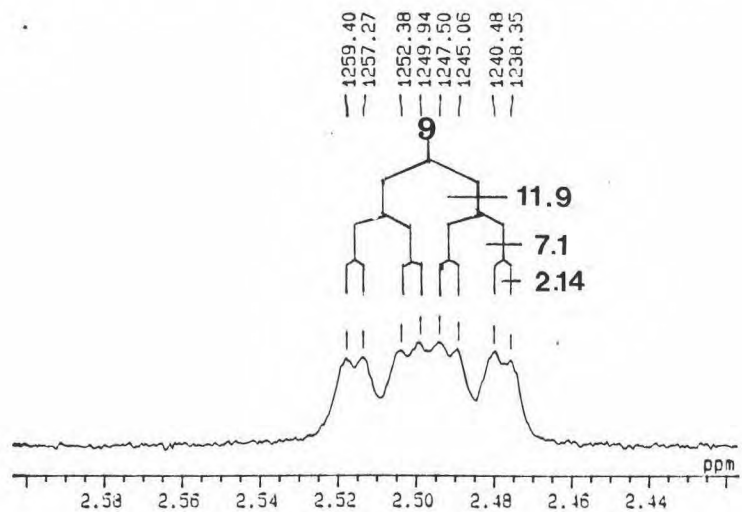
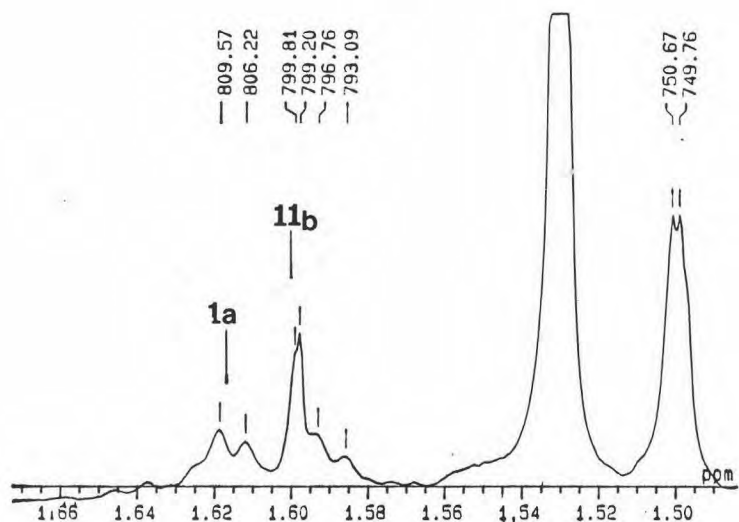
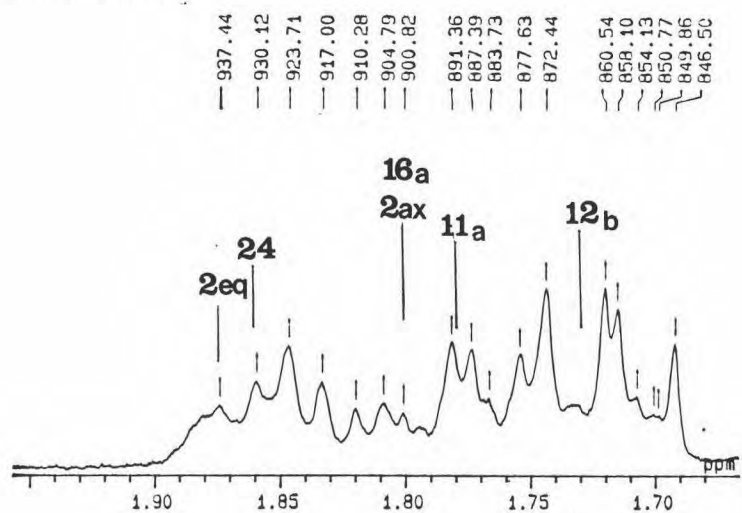


Figure 84. The 500 MHz  $^1\text{H}$  NMR spectrum of K084 in  $\text{CDCl}_3$  (expanded)

K084 IN MICRO TUBE

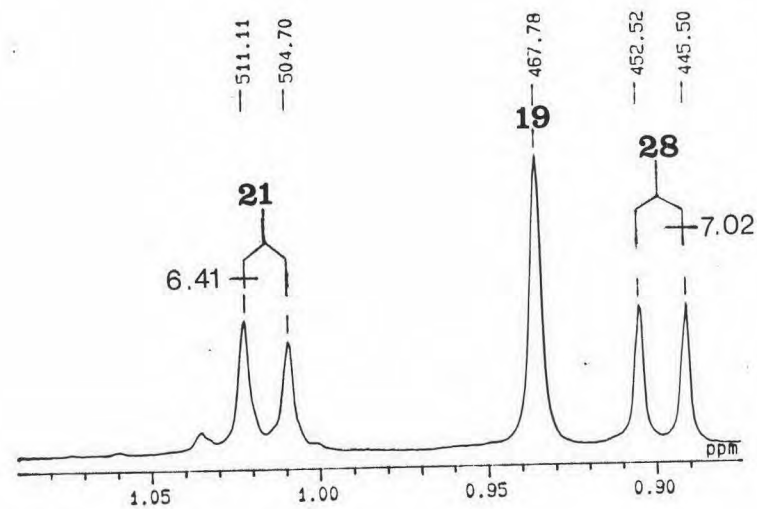
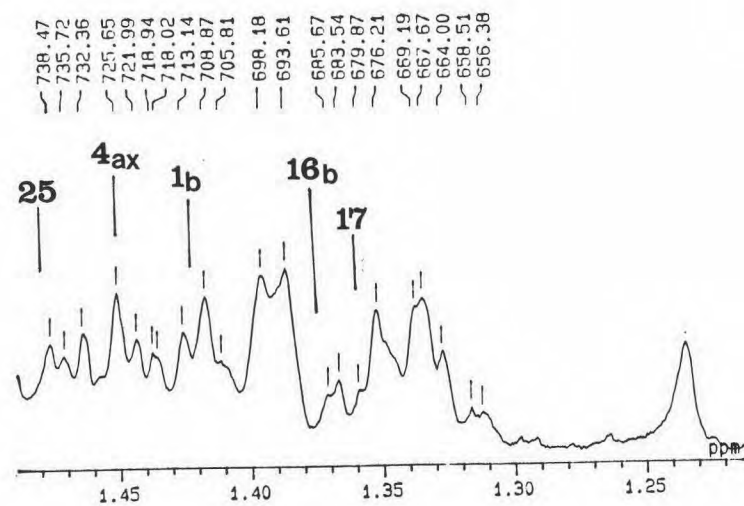
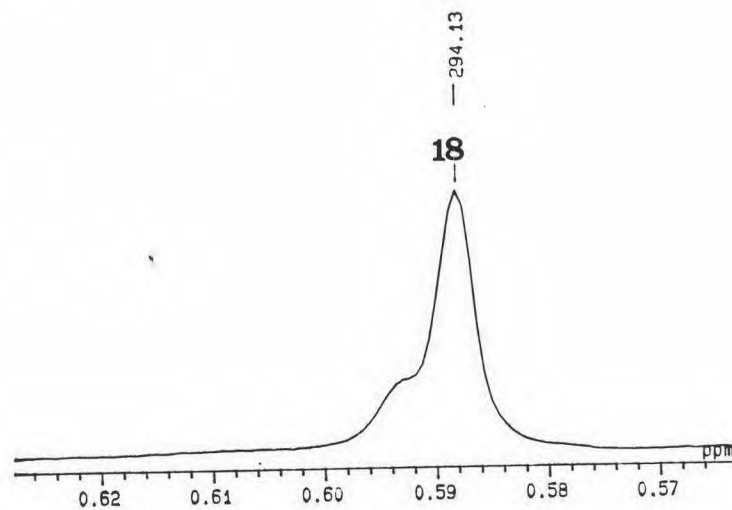
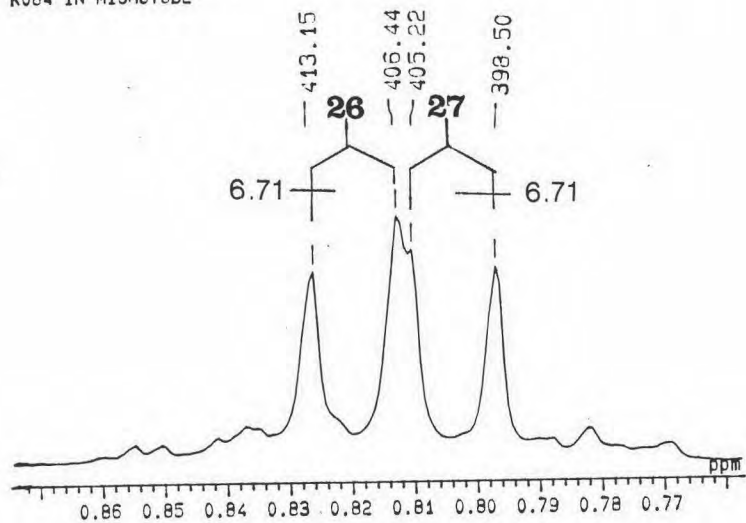


Figure 85. The 500 MHz  $^1\text{H}$  NMR spectrum of K084 in  $\text{CDCl}_3$  (expanded)

K084BCM

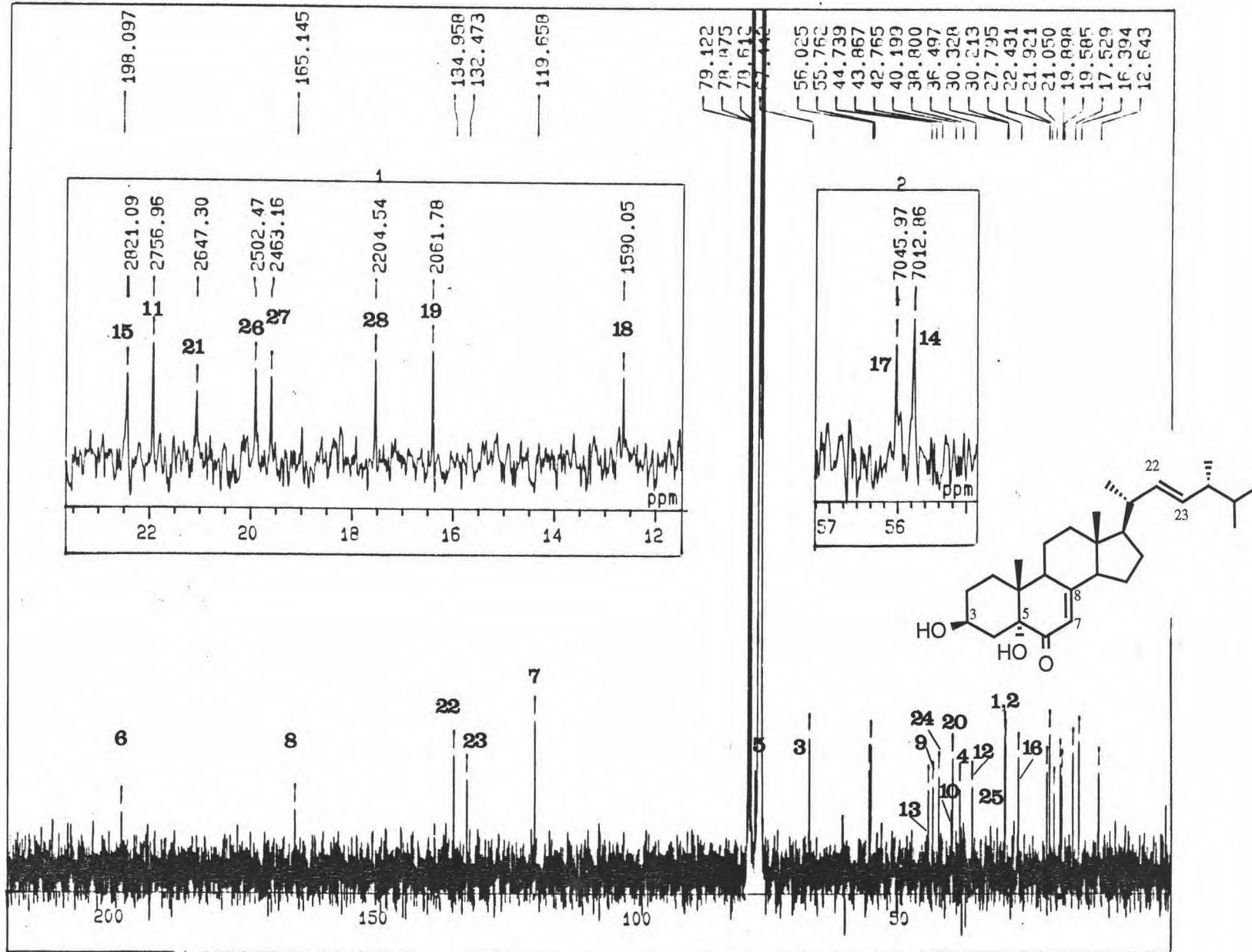


Figure 86. The 125 MHz  $^{13}\text{C}$  NMR spectrum of K084 in  $\text{CDCl}_3$ .



K084 HMQC MICROTU3E

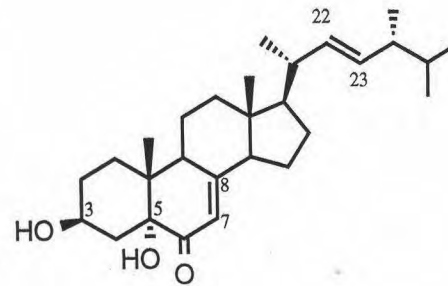
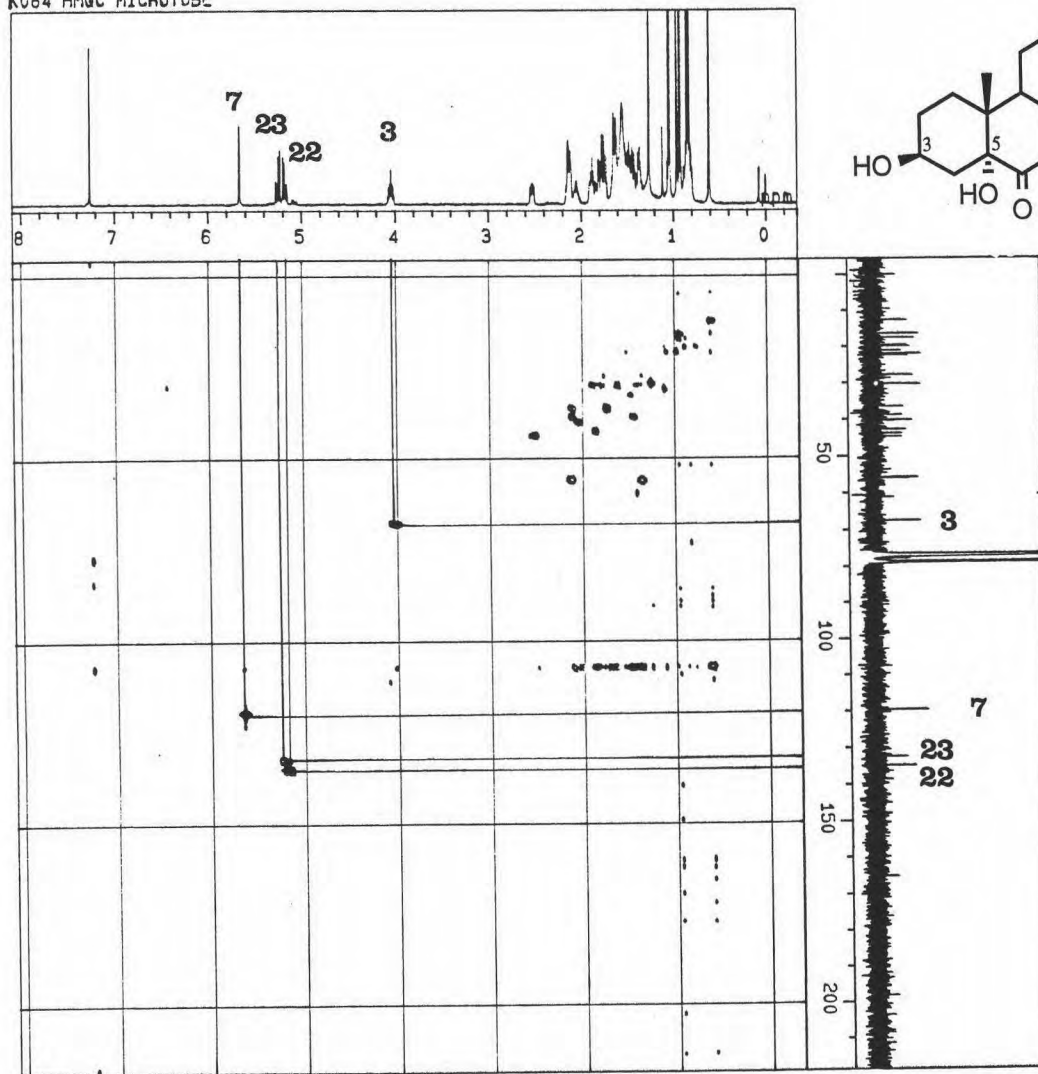


Figure 87. The HMQC spectrum of K084

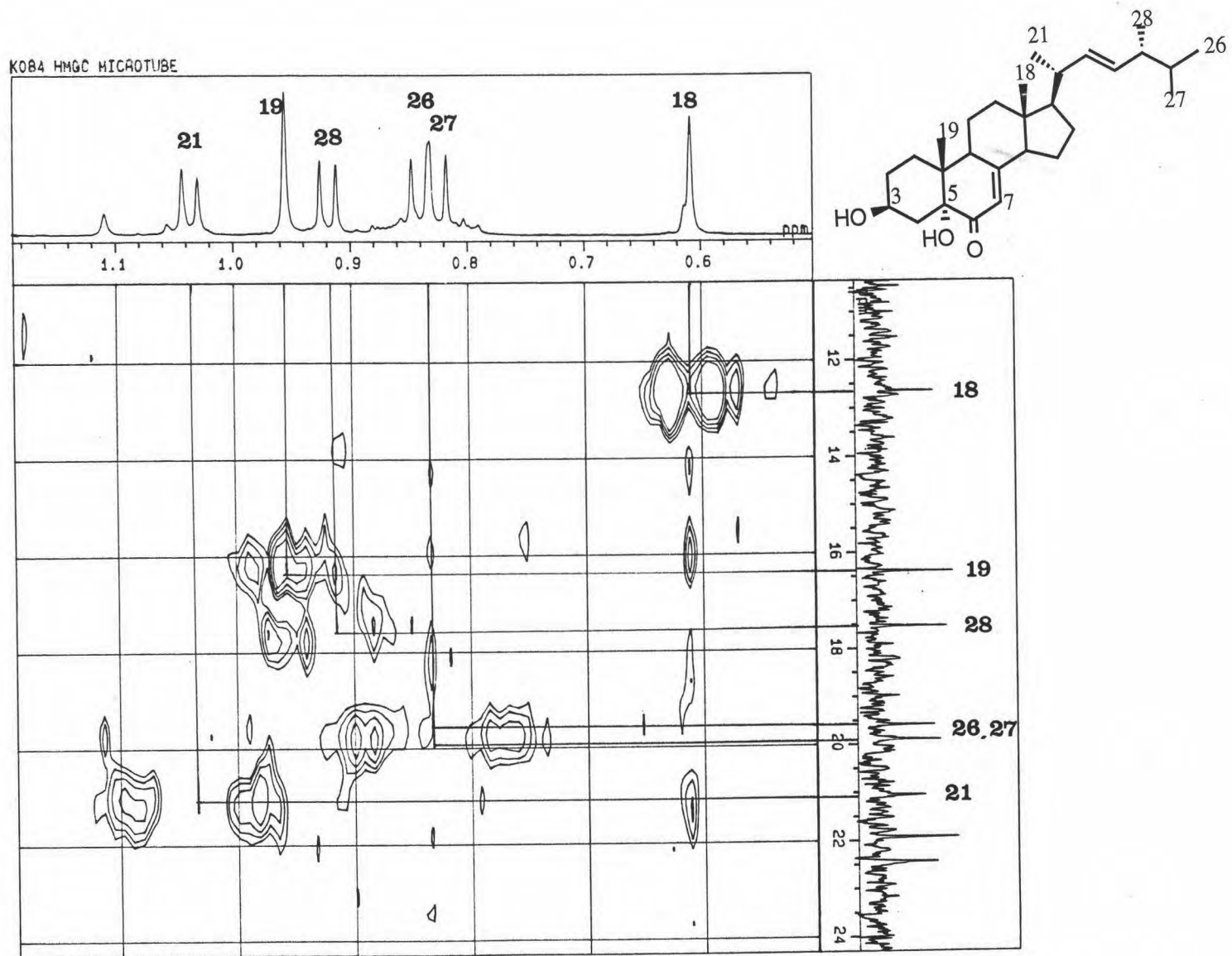


Figure 88. The partial HMQC spectrum ( $\delta_H$  0.5-1.2 ppm) of K084

K084 HMQC MICROTUBE

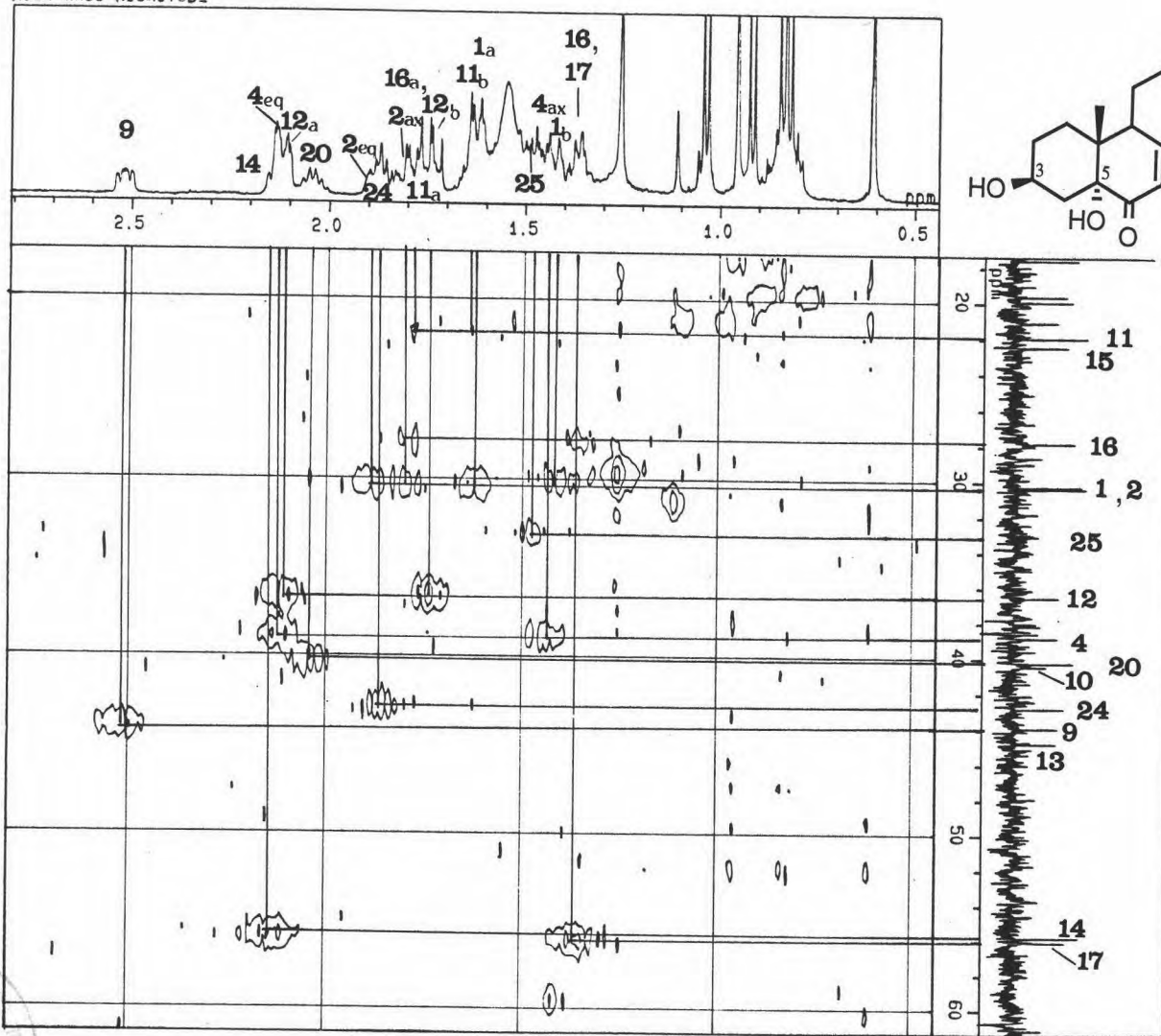


Figure 89. The partial HMQC spectrum ( $\delta_{\text{H}}$  0.5-2.6 ppm) of K084

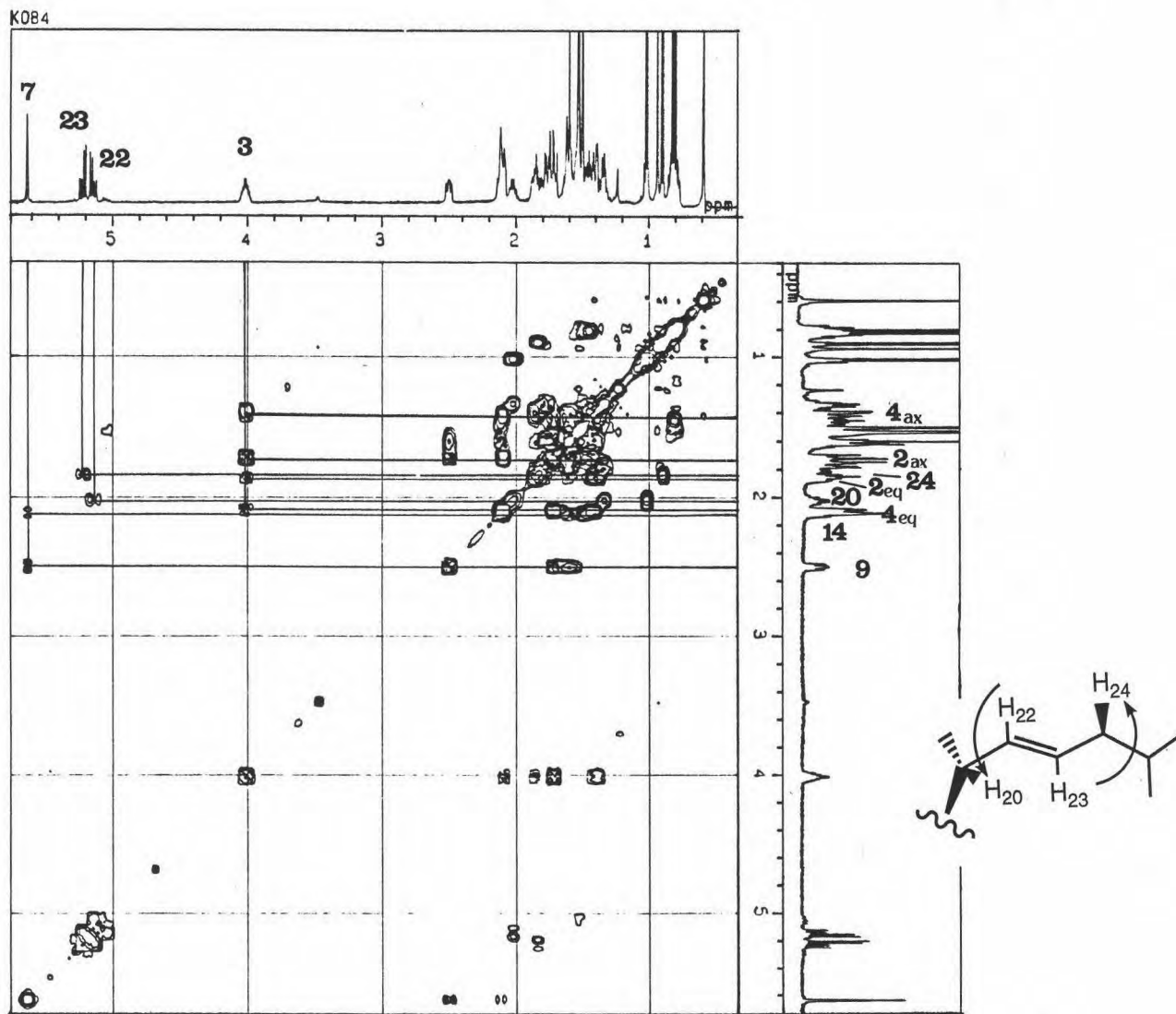


Figure 90. The  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of K084

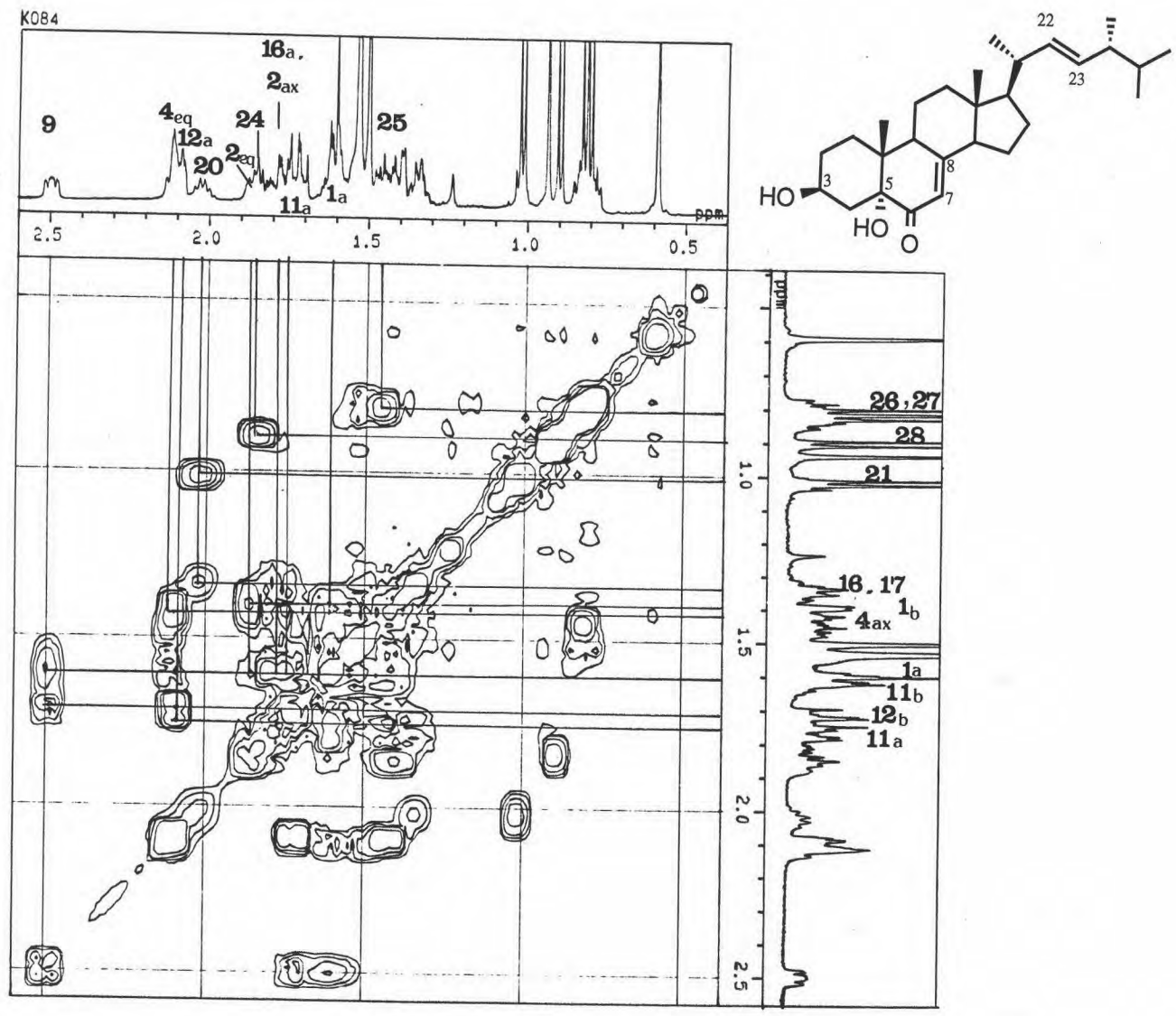


Figure 91. The partial  $^1\text{H}$ - $^1\text{H}$  COSY spectrum ( $\delta_{\text{H}}$  0.5-2.6 ppm) of K084

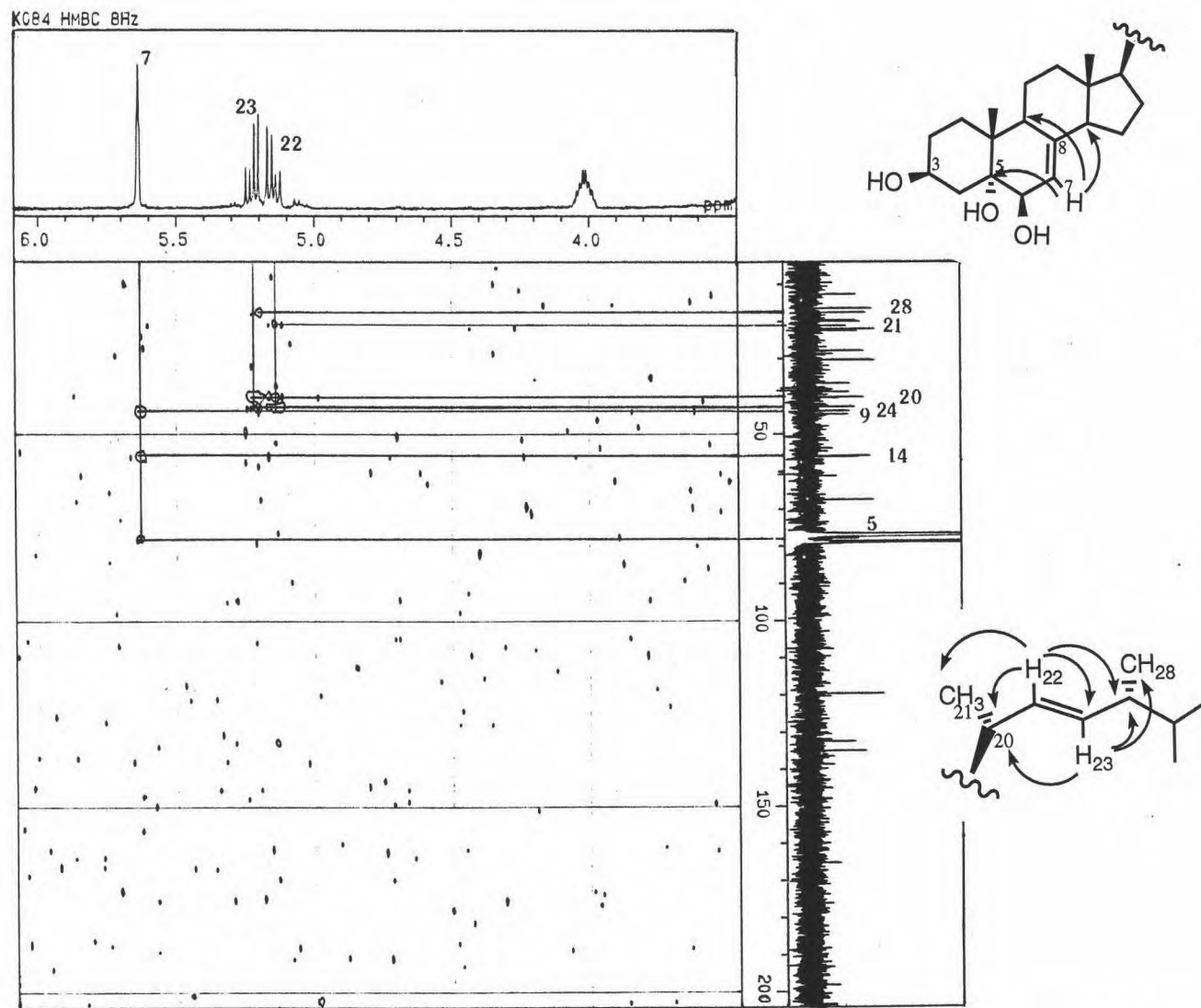


Figure 92. The partial HMBC spectrum ( $\delta_H$  3.6-6.0 ppm) of K084 ( $^3J_{CH} = 4$  Hz)

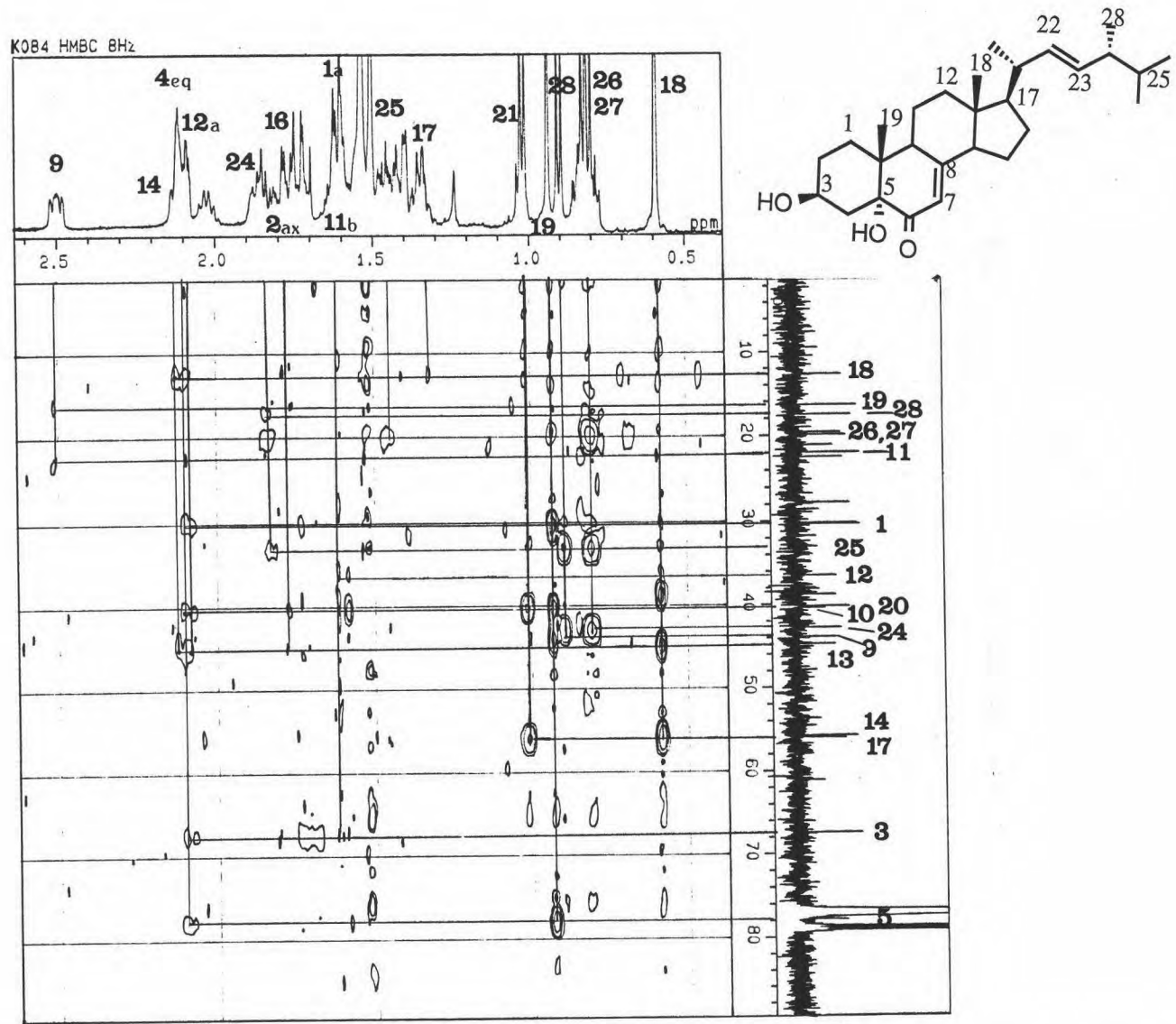


Figure 93. The partial HMBC spectrum ( $\delta_{\text{H}}$  0.5-2.6 ppm) of K084 ( $^3J_{\text{CH}} = 4$  Hz)

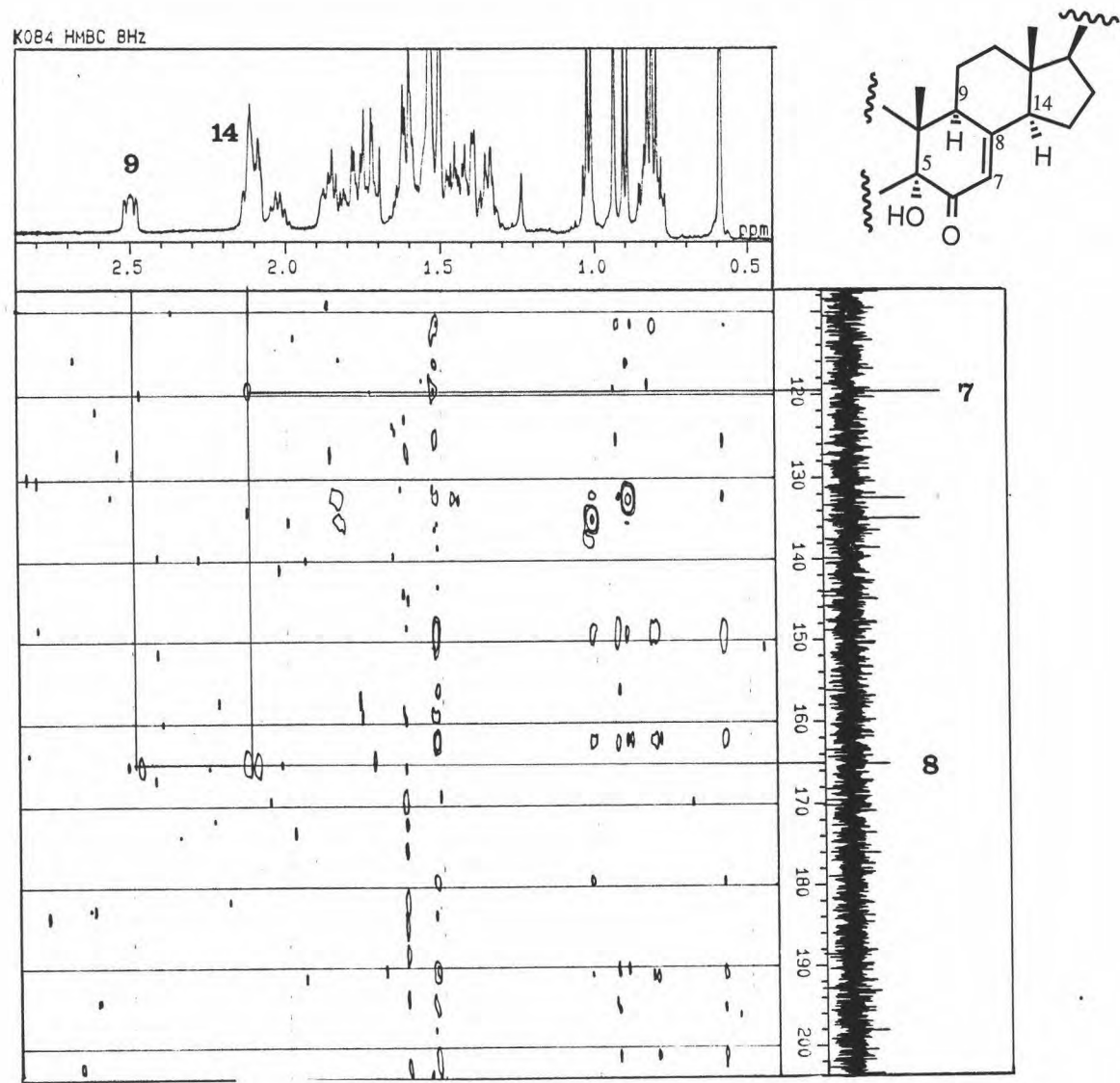


Figure 94. The partial HMBC spectrum ( $\delta_{\text{H}}$  0.4-1.2 ppm) of K084 ( $^3J_{\text{CH}} = 4$  Hz)



## VITA

Miss Ladda Thitithanaphuk was born on February 19, 1969 in Lampang, Thailand. She received her Bachelor degree of Science in Pharmacy in 1992 from the faculty of Pharmaceutical Sciences, Chiang Mai University, Chiang Mai, Thailand. At present, she is a faculty member of the Department of Medicinal Chemistry and Natural Products, Faculty of Pharmacy, Naresuan University, Phitsanulok, Thailand.