

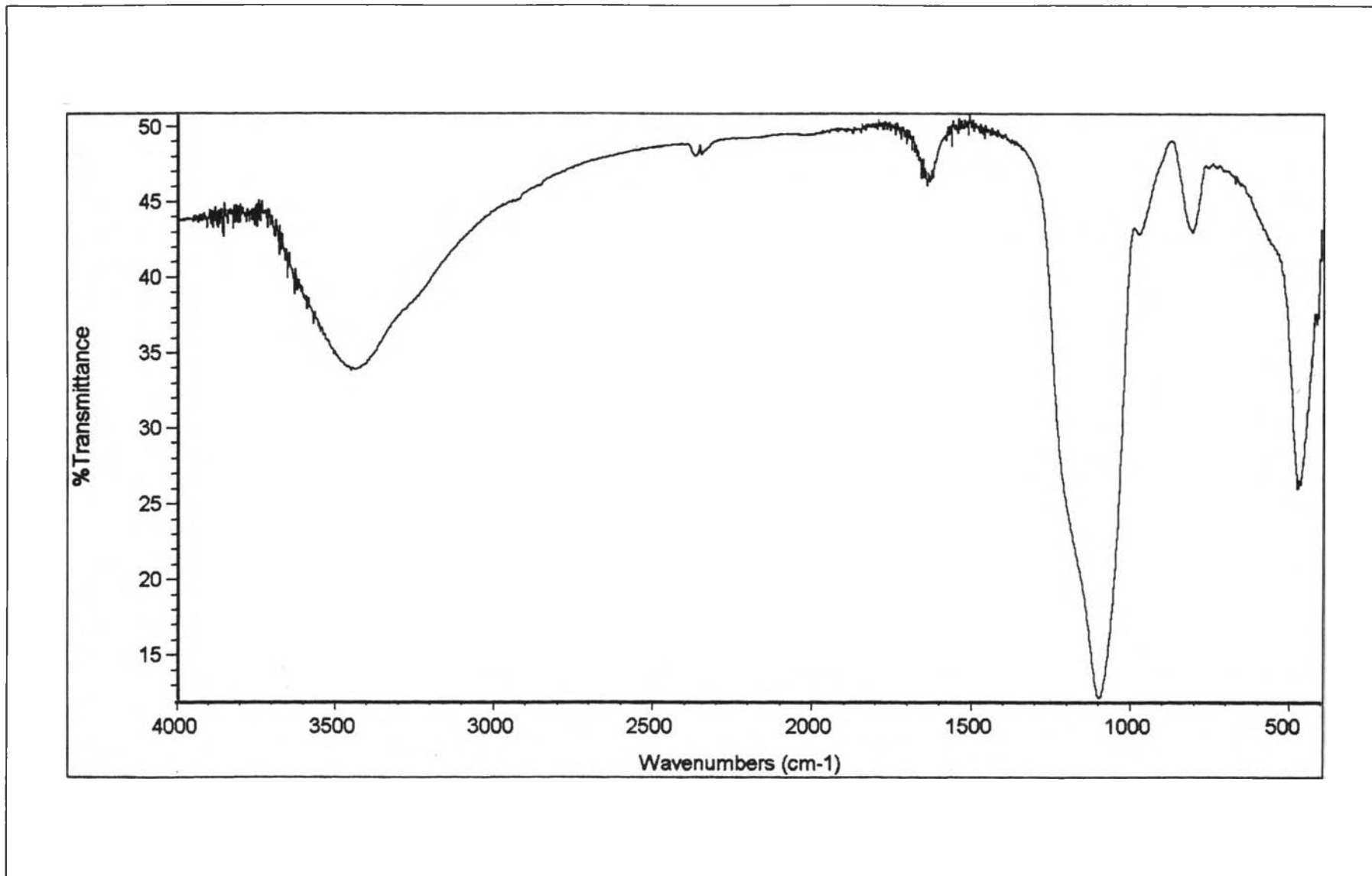
## REFERENCES

1. McGram, H. **Encyclopedia of Chemistry Second Edition vol 5**, John Wiley & Sons, Inc., New York 1992: 764-766.
2. Laine, R.M.; Blohowiak, Y.K.; Robinson, T.R.; Hoppe, M.L.; Nardi, P.; Kampf, J.; Uhm, J. *Synthesis of pentacoordinate silicon complexes from SiO<sub>2</sub>* **Nature** 353 (1991): 642-644.
3. Iler, R.K.; **The Chemistry of Silica** John Wiley & Sons, Inc., New, York 1979: 1-76.
4. Kirk-Othemer **Encyclopedia of Chemical Technology Second Edition vol. 18**, John Wilon & Sons, Inc. New York 1969: 46-72.
5. Leela-Adisorn, U. *Preparation and Characterization of high-grade Silica from rice husk*, **Master's Thesis**, Department of Materials Science, Graduate School, Chulalongkorn University, 1992.
6. Tungboriboon, N. *Preparation and sintering of silica glass from rice-husk ash by hydrogel method*, **Master's Thesis**, Department of Materials Science, Graduate School, Chulalongkorn University, 1993.
7. Luk, B.S. **Rice Production and Utilization** The AVI publishing company Inc. New York 1980: 739-742.
8. Basu, P.K.; King, C.J.; Lynn, S. *Manufacture of Silicon Tetrachloride from rice hulks* **Journal AIChE** 19 (1973): 439-445.
9. Rochow, E.G. **Silicon and Silicones** Springer-Verlag Berlin Heidelberg Germany 1987: 155-165.
10. Laine, R.M.; Youngdahl, K.A.; Nardi, P. *Silicon and Aluminum Complexes* **US Patent 5,099,052** (March 2, 1992).

11. Laine, R.M.; Mich, A.A. *Ion Conducting Polymer* **US Patent 5,440,011** (August 8, 1995).
12. Bickmore, C.; Hoppe, M.L.; Laine, R.M. *Processable Oligomeric and Polymeric Precursors to Silicate Prepared Directly from SiO<sub>2</sub>* **Mat.res.Soc.Symp.Proc.** 249 (1992): 81-86.
13. Flynn, J.J.; Boer, F.P. *Structural Studies of Hexacoordinate Silicon Tris (o-phenylenedioxy) silicate* **J.Am.Chem.Soc.** 91 (1969): 5756-5761.
14. Barnum, D.W.; *Catechol Complexes with Silicon* **Inorganic Chemistry** 9 (1970): 1942-1943.
15. Barnum, D.W. *Reaction of Catechol with Colloidal Silica and Silicic acid in Aqueous Ammonia* **Inorganic Chemistry** 11 (1972): 1424-1429.
16. Frye, C.L. *Pentacoordinate Silicon Derivatives IV. Alkylammonium Siliconate Salts Derived from Aliphatic 1,2-diol* **J.Am.Chem.Soc.** 92 (1970): 1205-1210.
17. Frye, C.L.; Vogel, G.E.; Hall, J.A. *Triptych-Siloxazolidines: pentacoordinate bridgehead silanes resulting from transannular interaction of nitrogen and silicon* **J Am.Chem.Soc.** 83 (1961): 996-667.
18. Voronkov, M.G.; Dyakov, V.M.; and Kirpichenko **Silatranes** Elsevier Sequoia S.A. 1982: 1-83.
19. Garant, R.J.; Daniels, L.M.; Das, S.K.; etc. *Lewis Basicity of Silatranes and the Molecular Structures of EtOSi(OCH<sub>2</sub>CH<sub>2</sub>)<sub>3</sub>N, Me<sub>2</sub>OSi(OCH<sub>2</sub>CH<sub>2</sub>)<sub>3</sub>N, and CF<sub>3</sub>CO<sub>2</sub>HEtOSi(OCH<sub>2</sub>CH<sub>2</sub>)<sub>3</sub>N* **J.Am.Chem.Soc.** 91 (1991): 5728-5735.

20. Frye, C.L.; Vincent, G.A.; and Finzel, W.A. *Pentacoordinate Silicon Compound V. Novel Silatrane Chemistry* **J.Am.Chem.Soc.** 93 (1971) : 6805-6811.
21. Laine, R.M.; Mueller, B.L.; Hinklin, T. *Neutral and mixed neutral/anionic polymetallooxanes* **US Patent 5,418,298** (May 23, 1995)
22. Iwamiya, J.H.; Maciel, G.E. *Chemical Shifts in Silatrane and Its Derivatives: A Study of the Transannular Interaction* **J.Am.Chem.Soc.** 115 (1993): 6835-6842.
23. Kumara Swamy, K.C.; Chandrasekhar, V.; Harland, J.J.; Holmes, J.M.; Day, R.O.; Holmes, R.R. *Pentacoordinate Acyclic and Cyclic Anionic Oxysilicates A <sup>29</sup>Si-NMR and X-ray Structural Study* **J.Am.Chem.Soc.** 112 (1990): 2341-2348.

## **APPENDIX**



**Figure A1** FT-IR Spectrum of silica, 0.007  $\mu\text{m}$ , from Aldrich Chemical Co., Inc.,

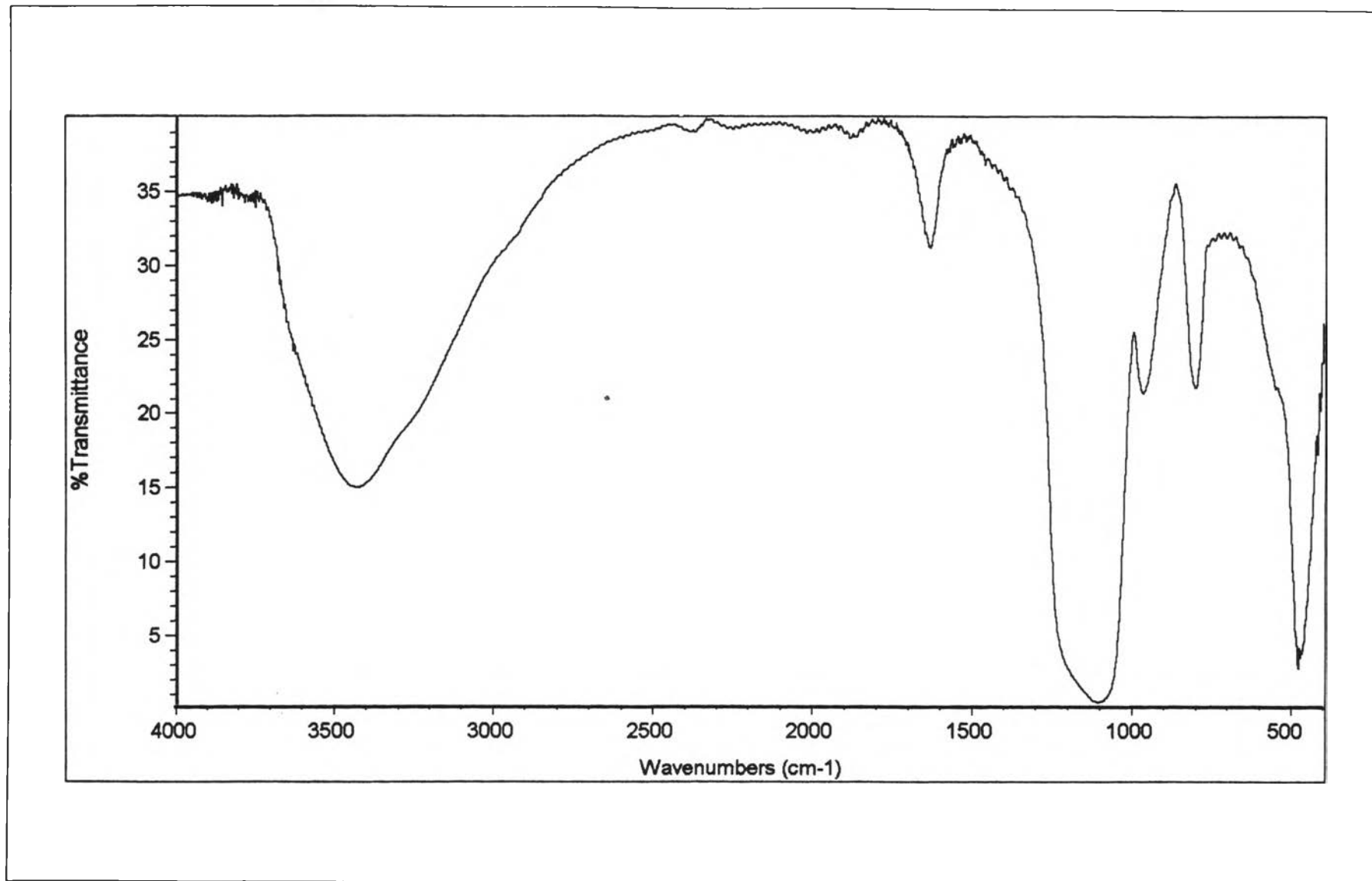


Figure A2 FT-IR Spectrum of silica, 10.97  $\mu\text{m}$ , from PPG Siam Silica Co., Ltd.

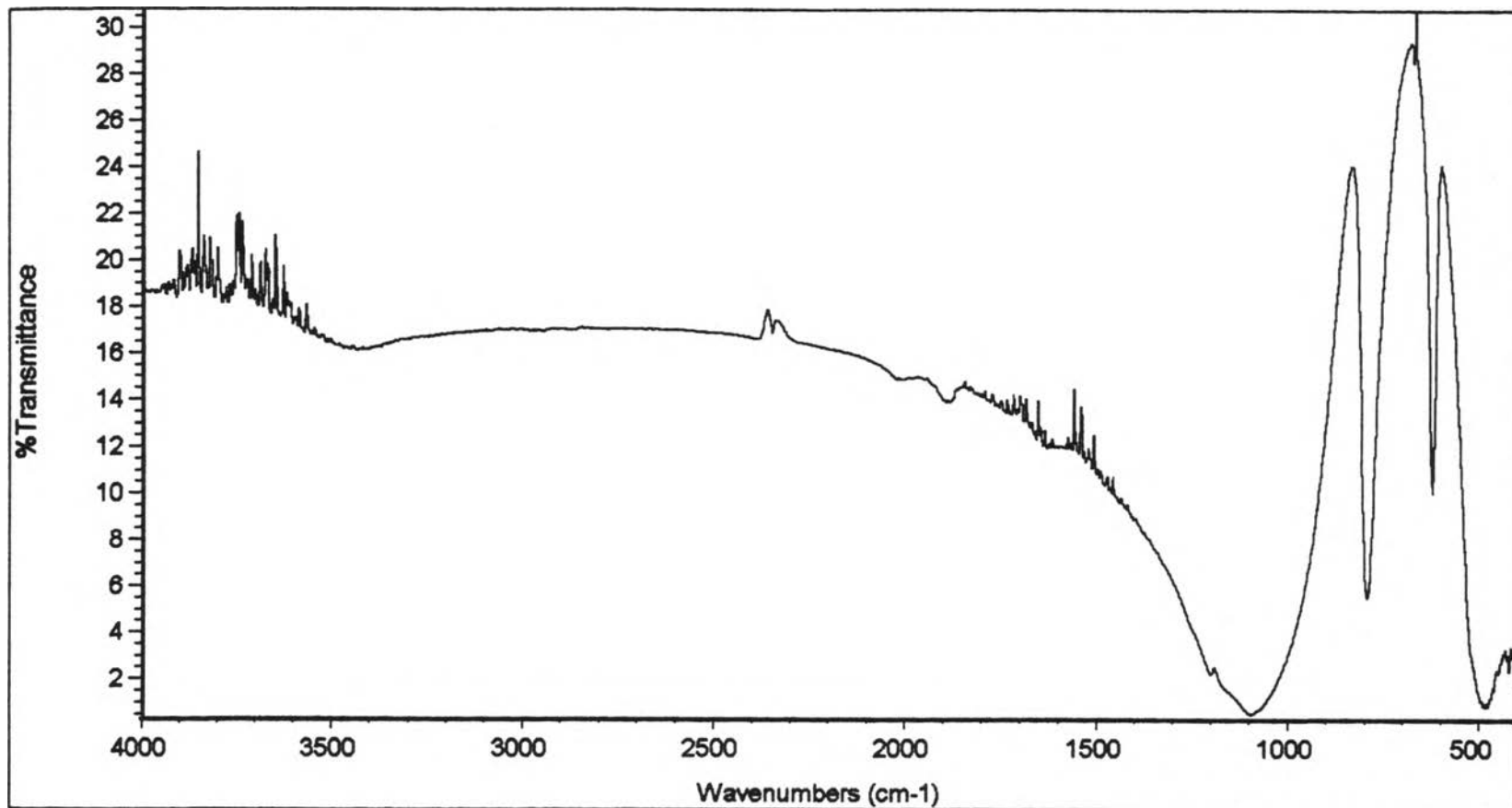


Figure A3 FT-IR Spectrum of rice husk ash, 13.47  $\mu\text{m}$ .

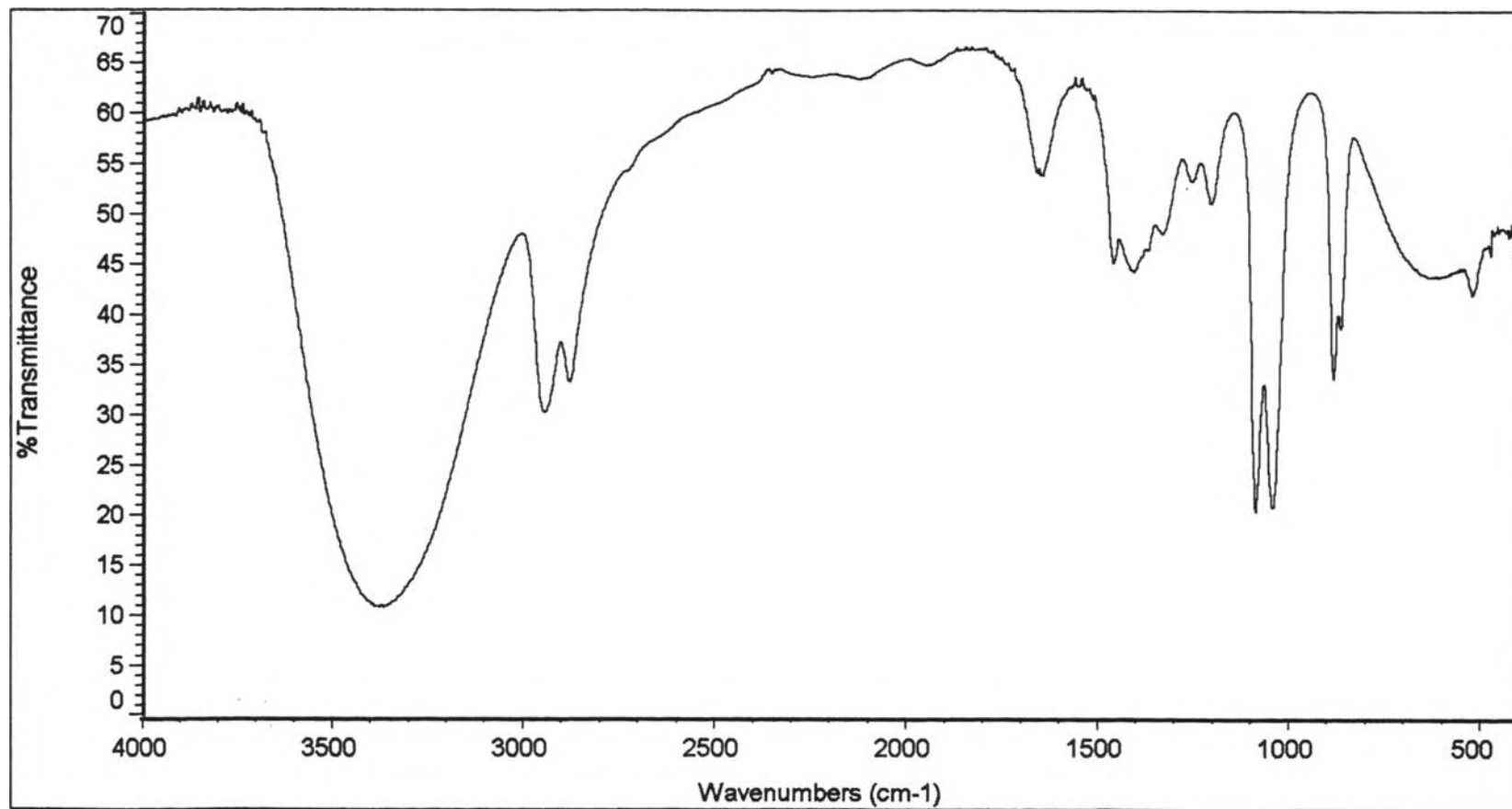


Figure A4 FT-IR Spectrum of ethylene glycol (EG)



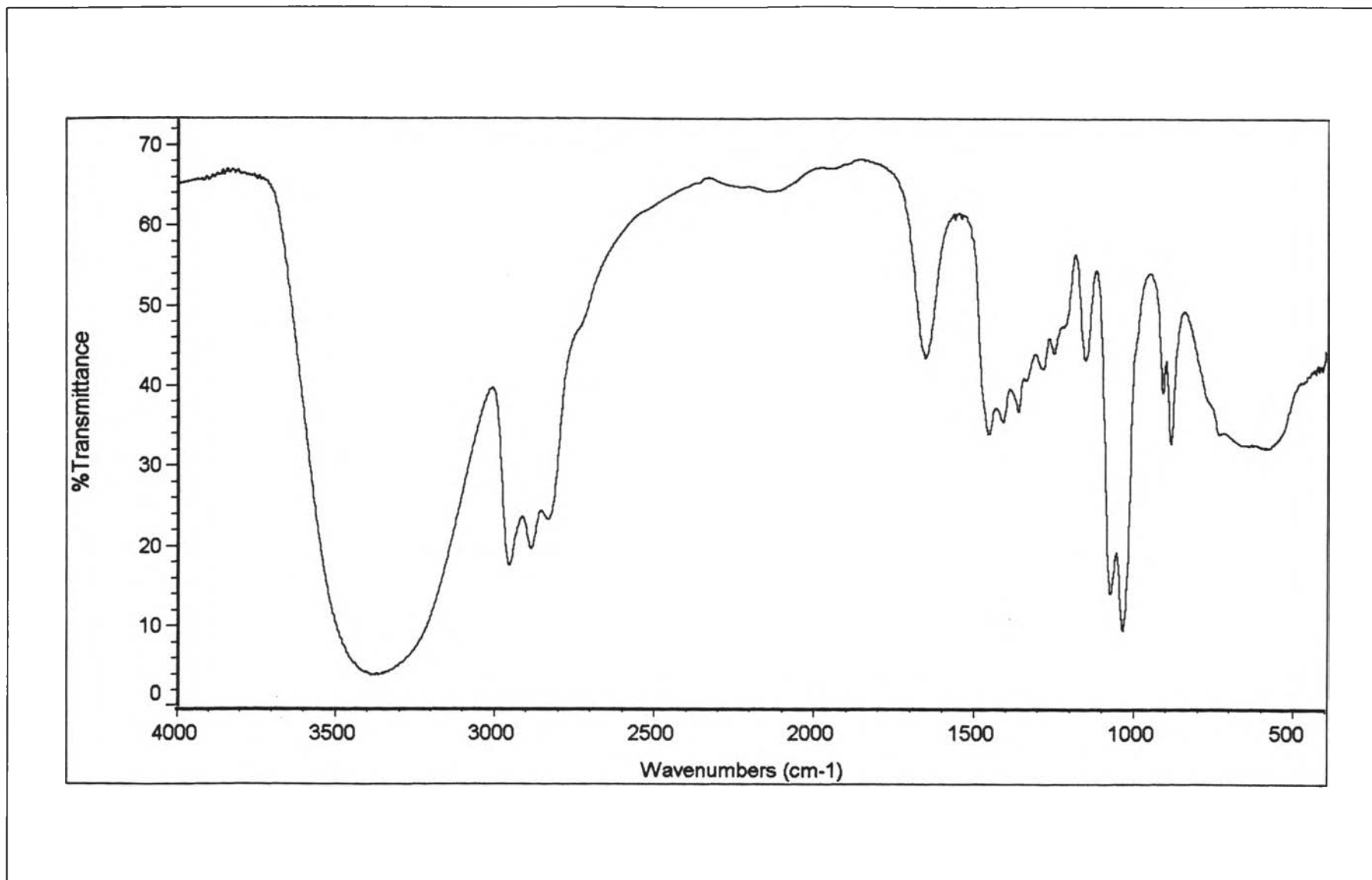


Figure A5 FT-IR Spectrum of triethanolamine (TEA)

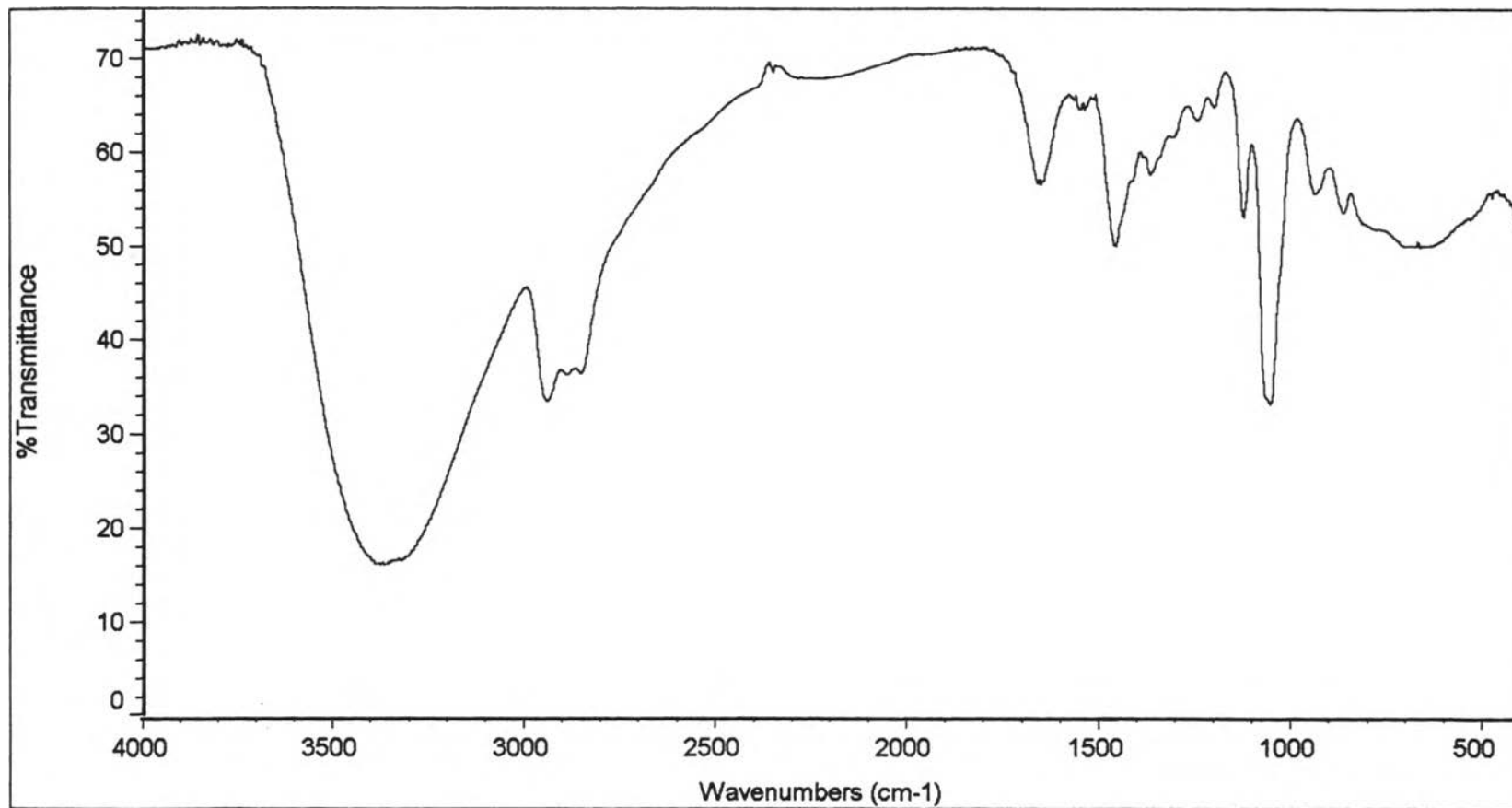


Figure A6 FT-IR Spectrum of diethanolamine (DEA)

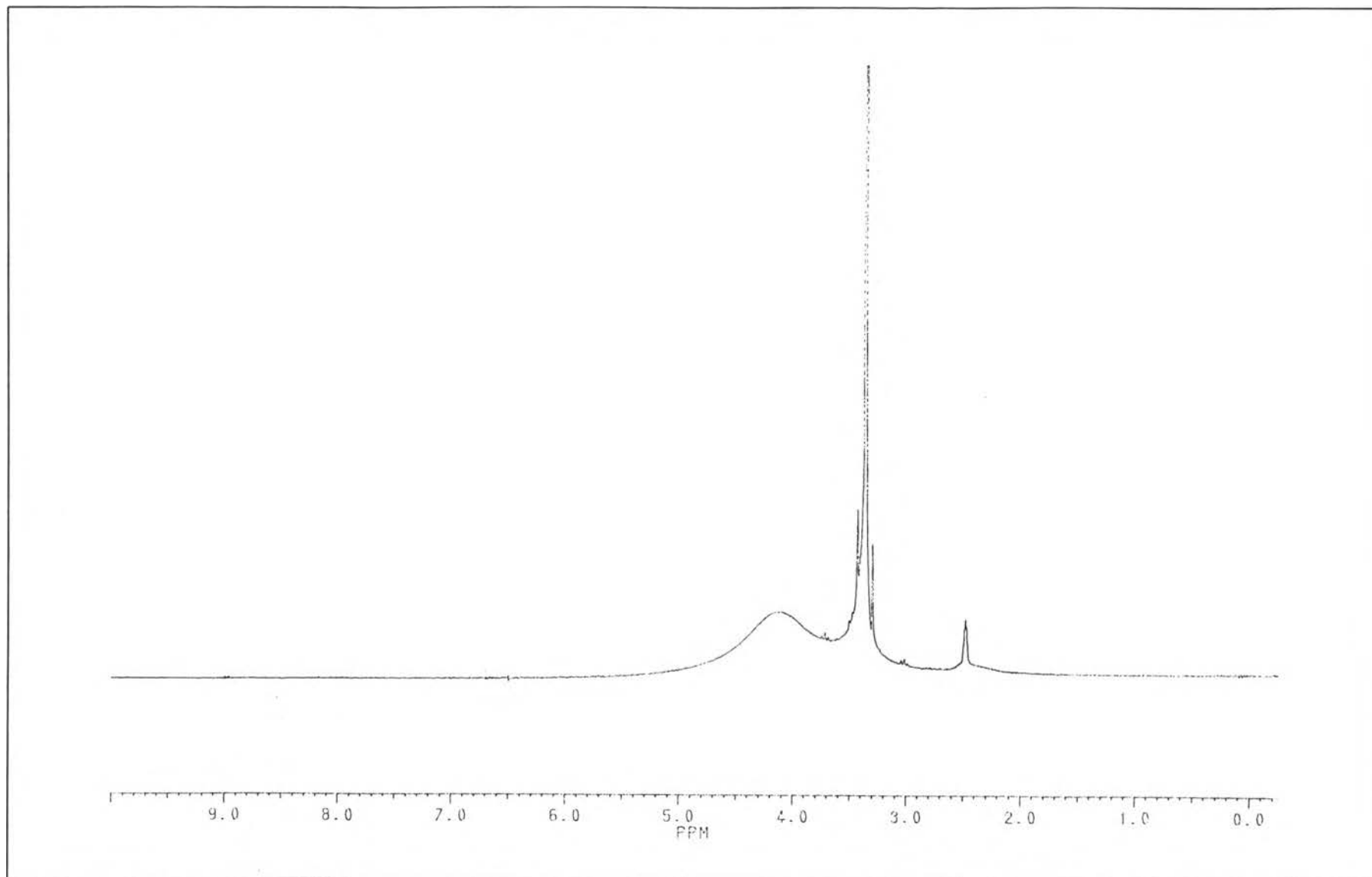
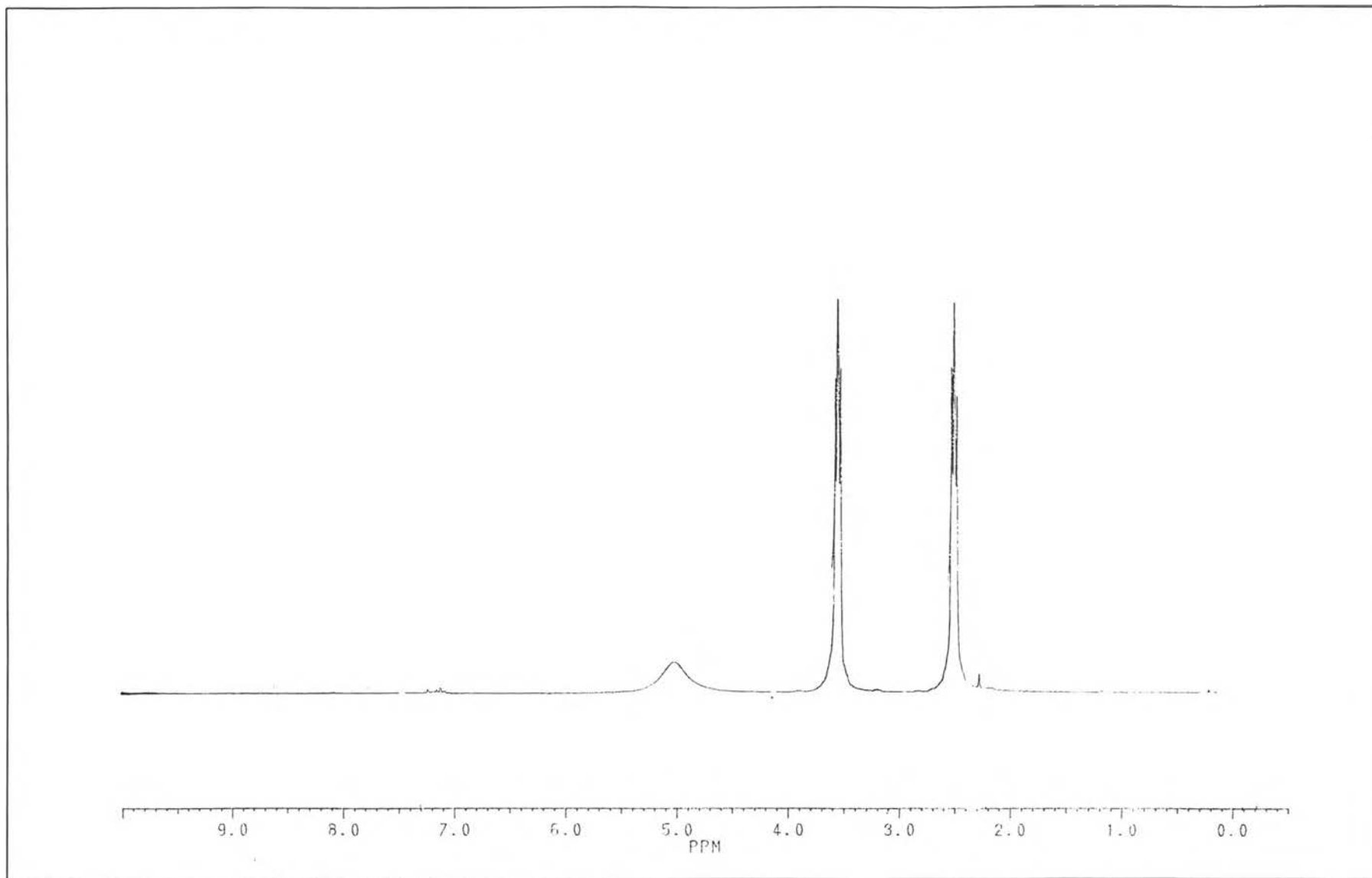
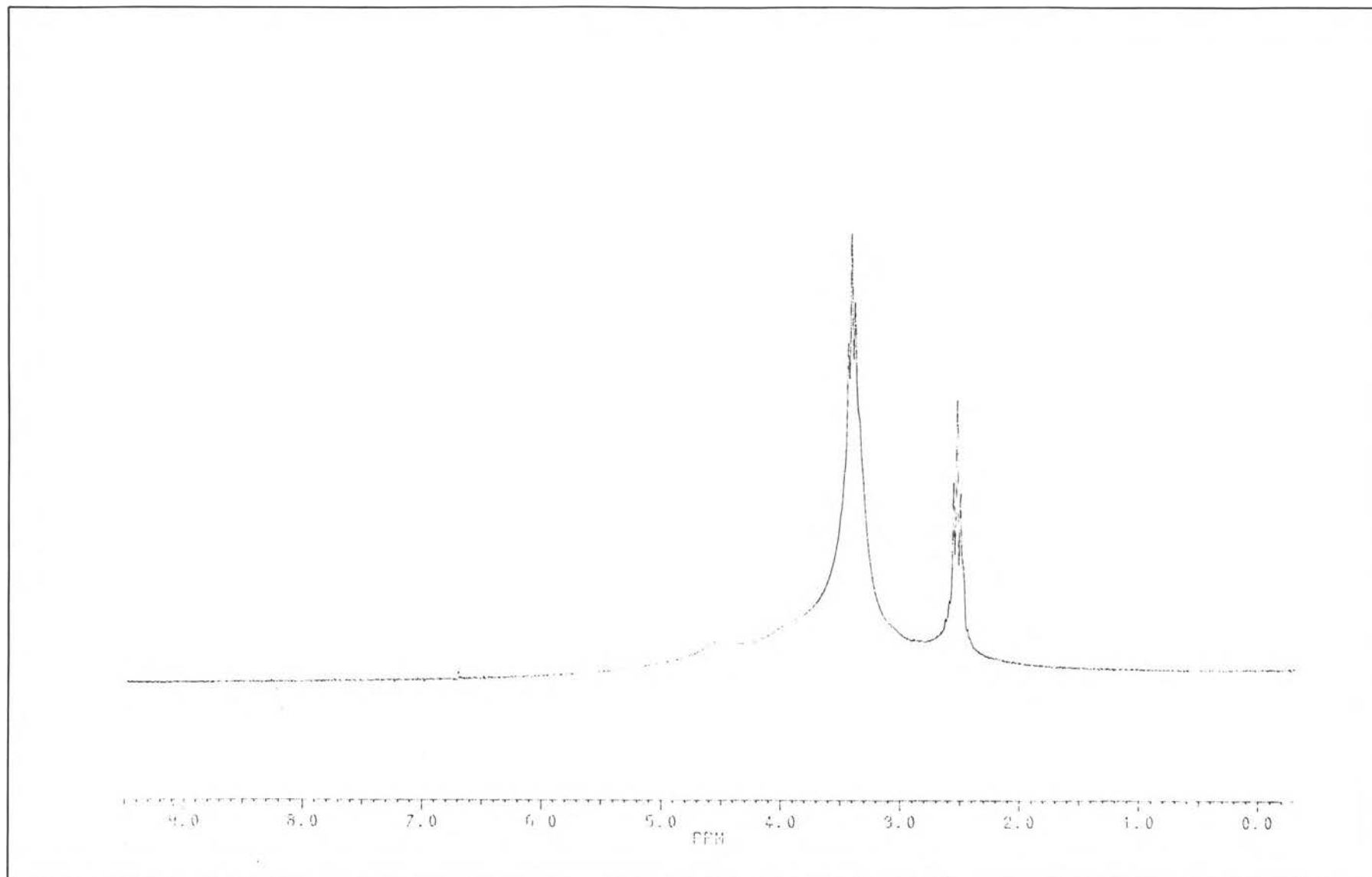


Figure A7  $^1\text{H-NMR}$  ( $d_6$ -DMSO) Spectrum of EG



**Figure A8**  $^1\text{H-NMR}$  ( $d_6\text{-DMSO}$ ) Spectrum of TEA



**Figure A9**  $^1\text{H-NMR}$  ( $\text{d}_6\text{-DMSO}$ ) Spectrum of DEA

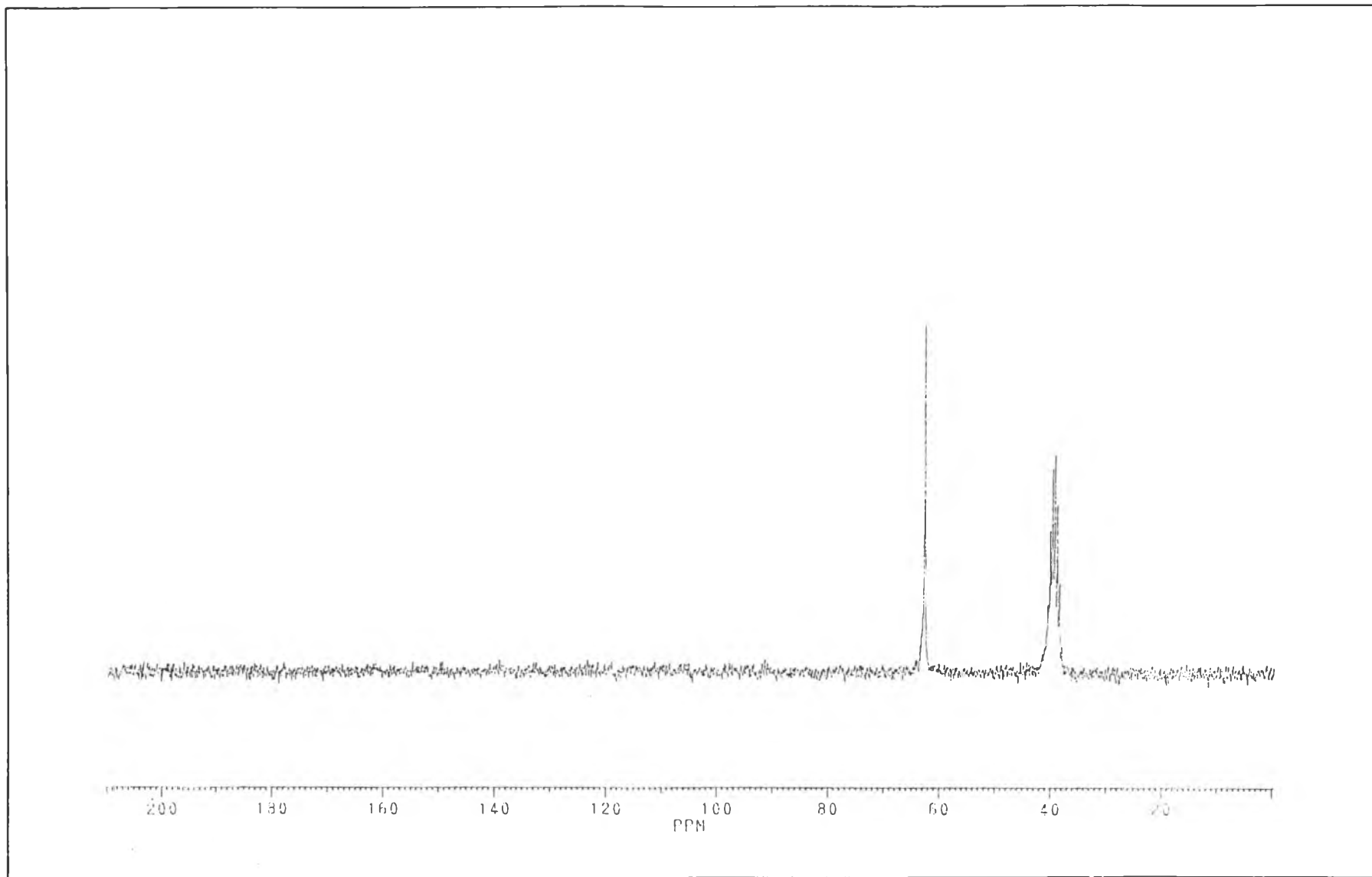
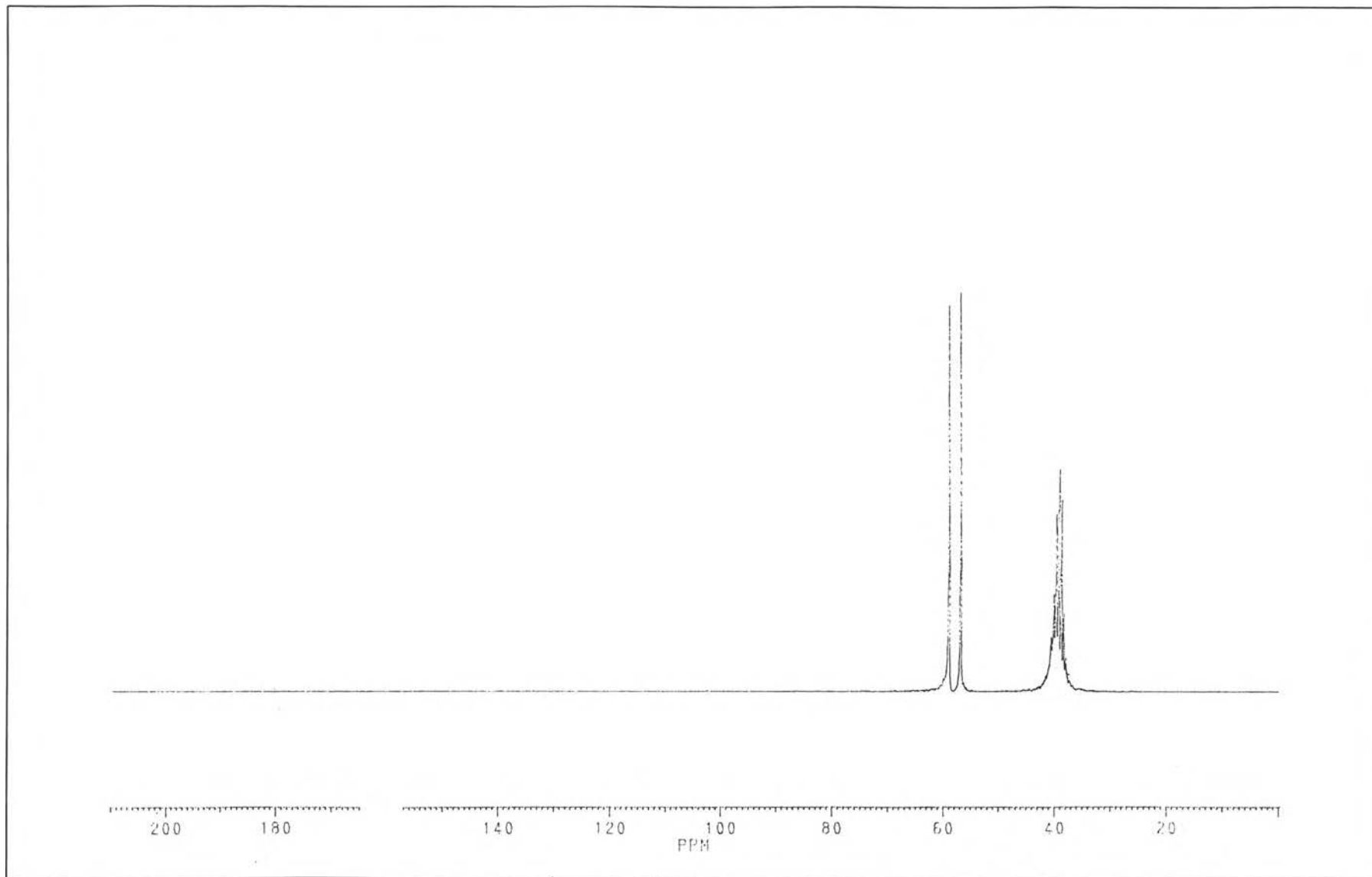
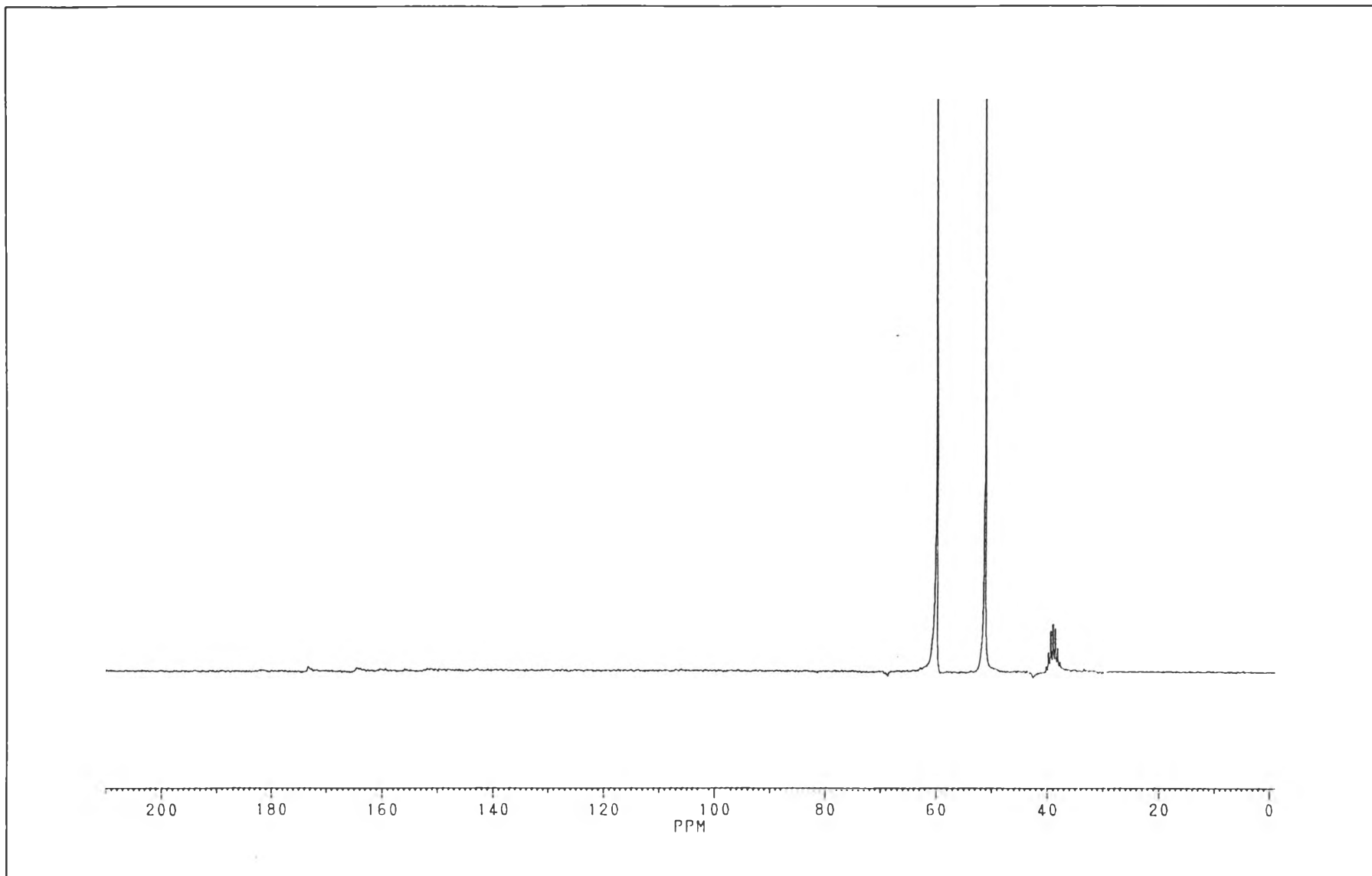


Figure A10  $^{13}\text{C}$ -NMR ( $\text{d}_6$ -DMSO) Spectrum of EG



**Figure A11**  $^{13}\text{C}$ -NMR ( $\text{d}_6$ -DMSO) Spectrum of TEA



**Figure A12**  $^{13}\text{C}$ -NMR ( $\text{d}_6$ -DMSO) Spectrum of DEA



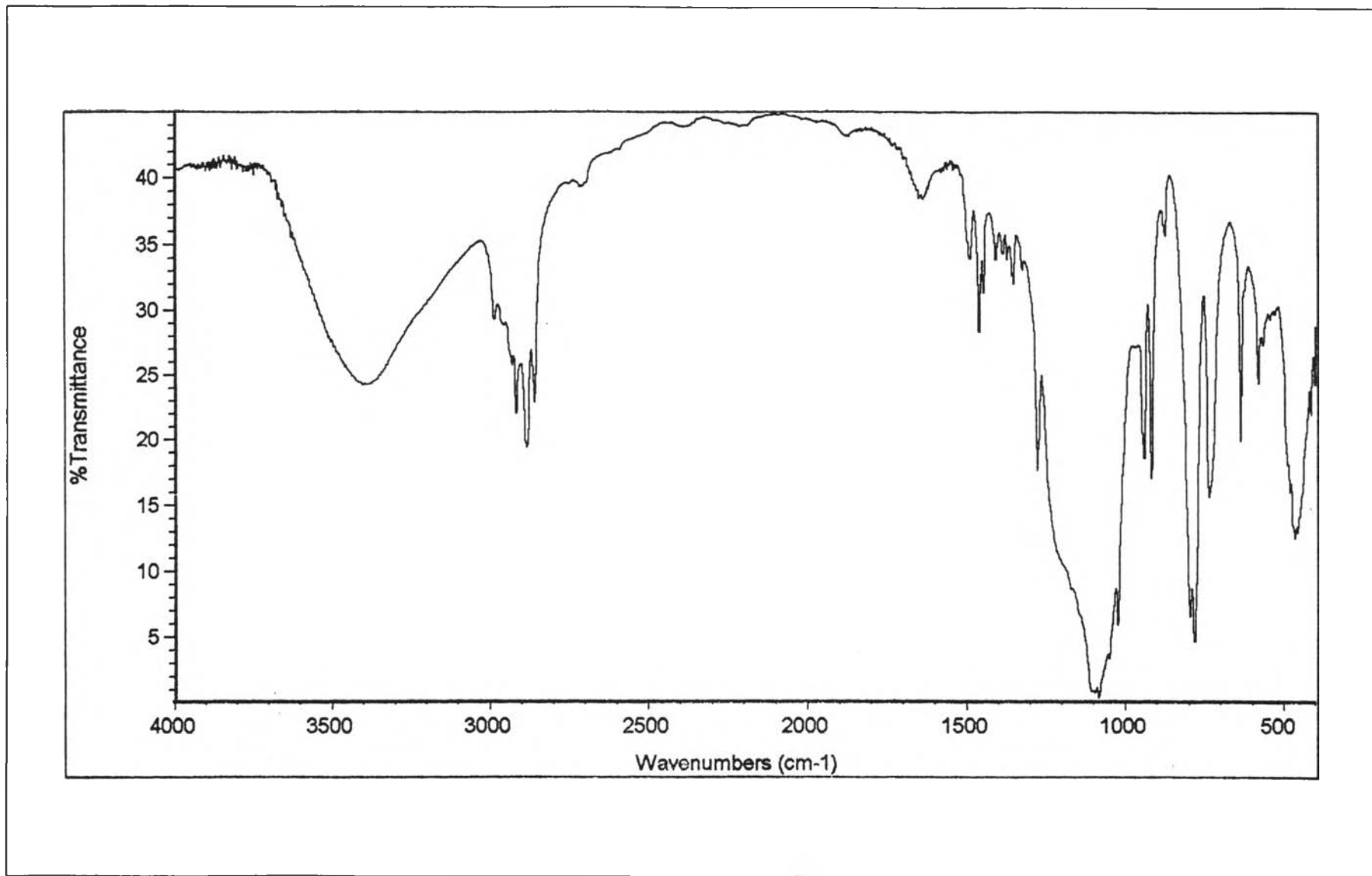


Figure A13 FT-IR Spectrum of sample 1 synthesized from silica 0.007  $\mu\text{m}$ , TEA and EG.

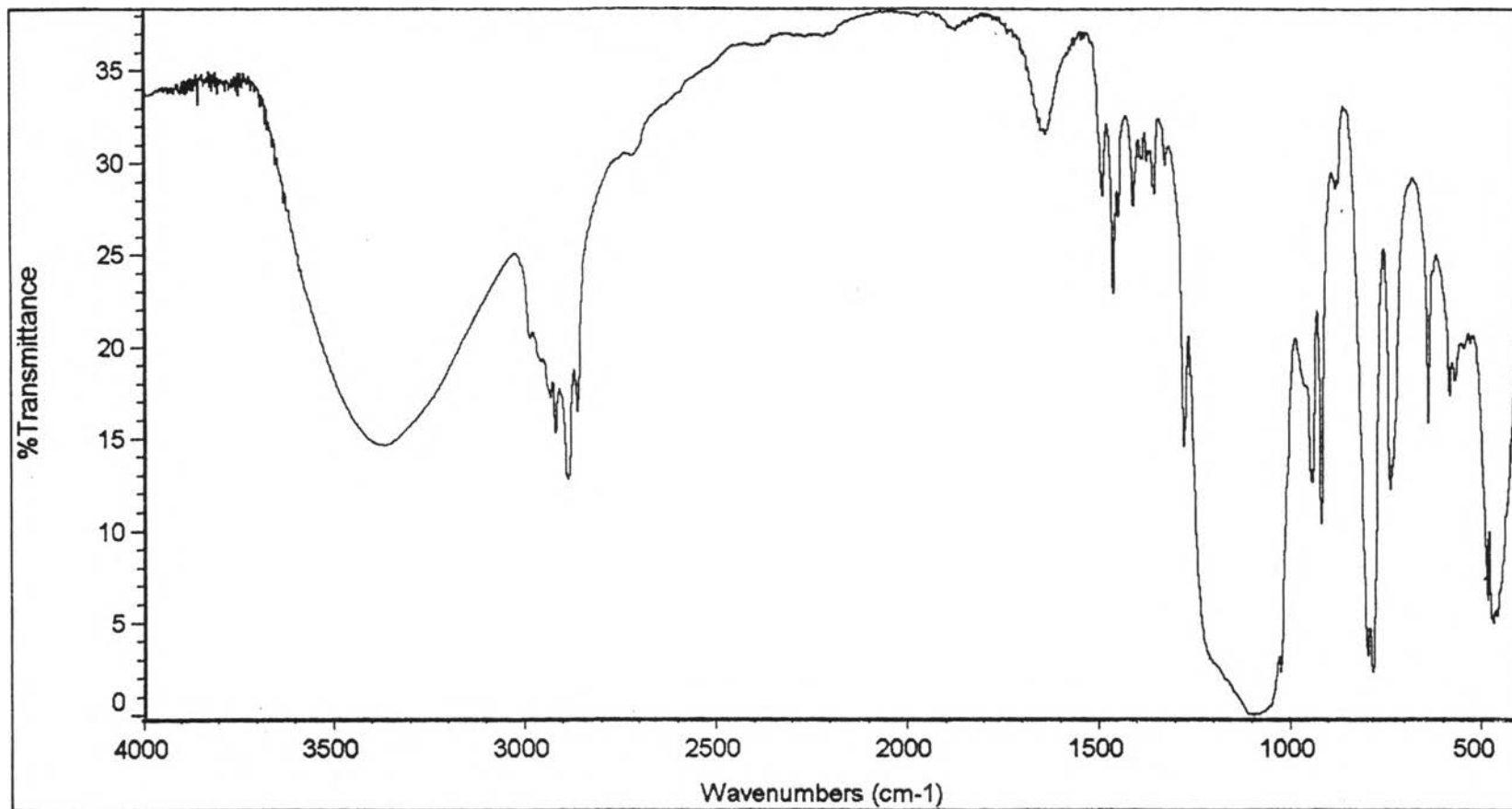


Figure A14 FT-IR Spectrum of sample 2 synthesized from silica 10.97  $\mu\text{m}$ , TEA and EG.

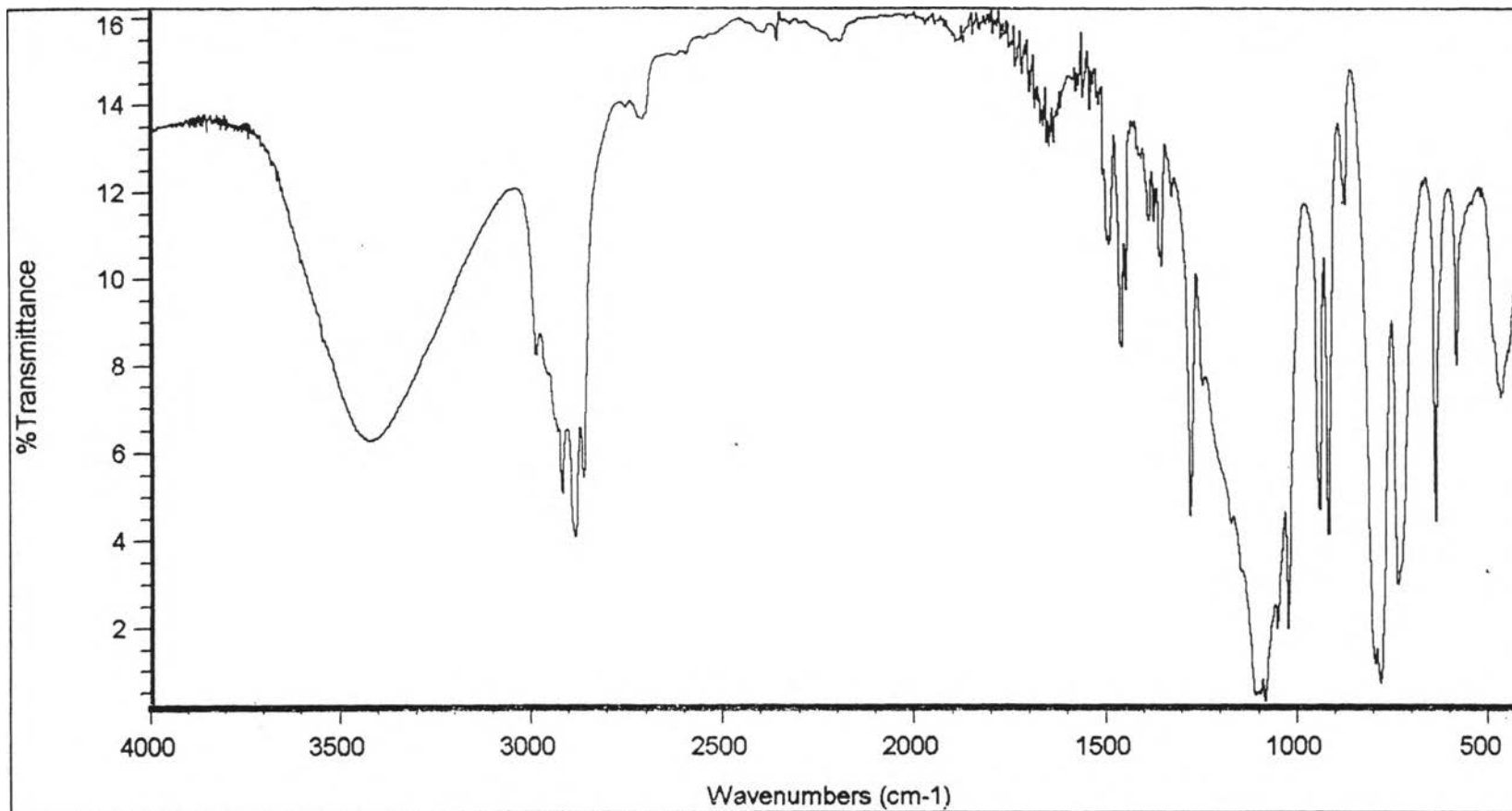
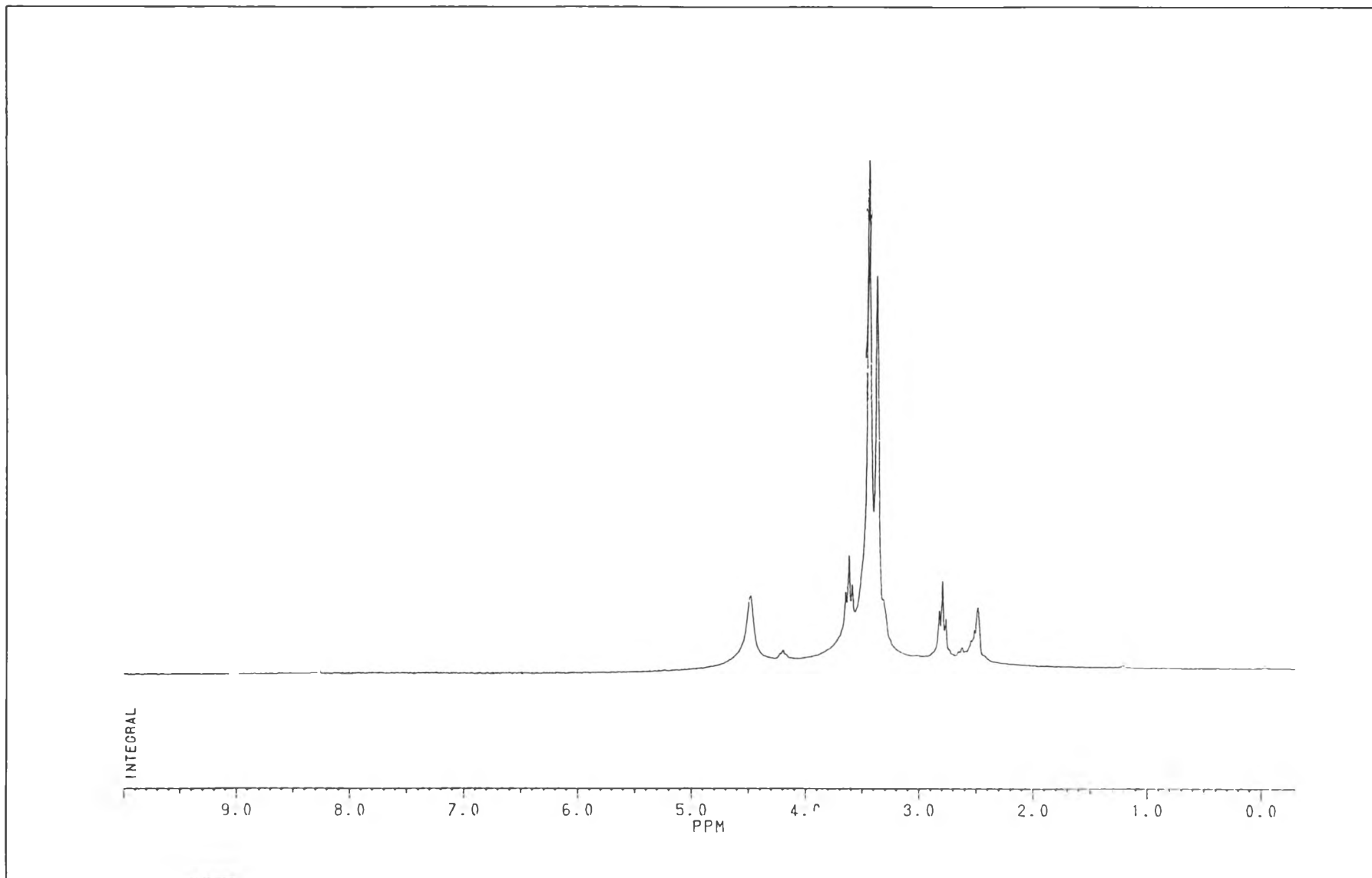
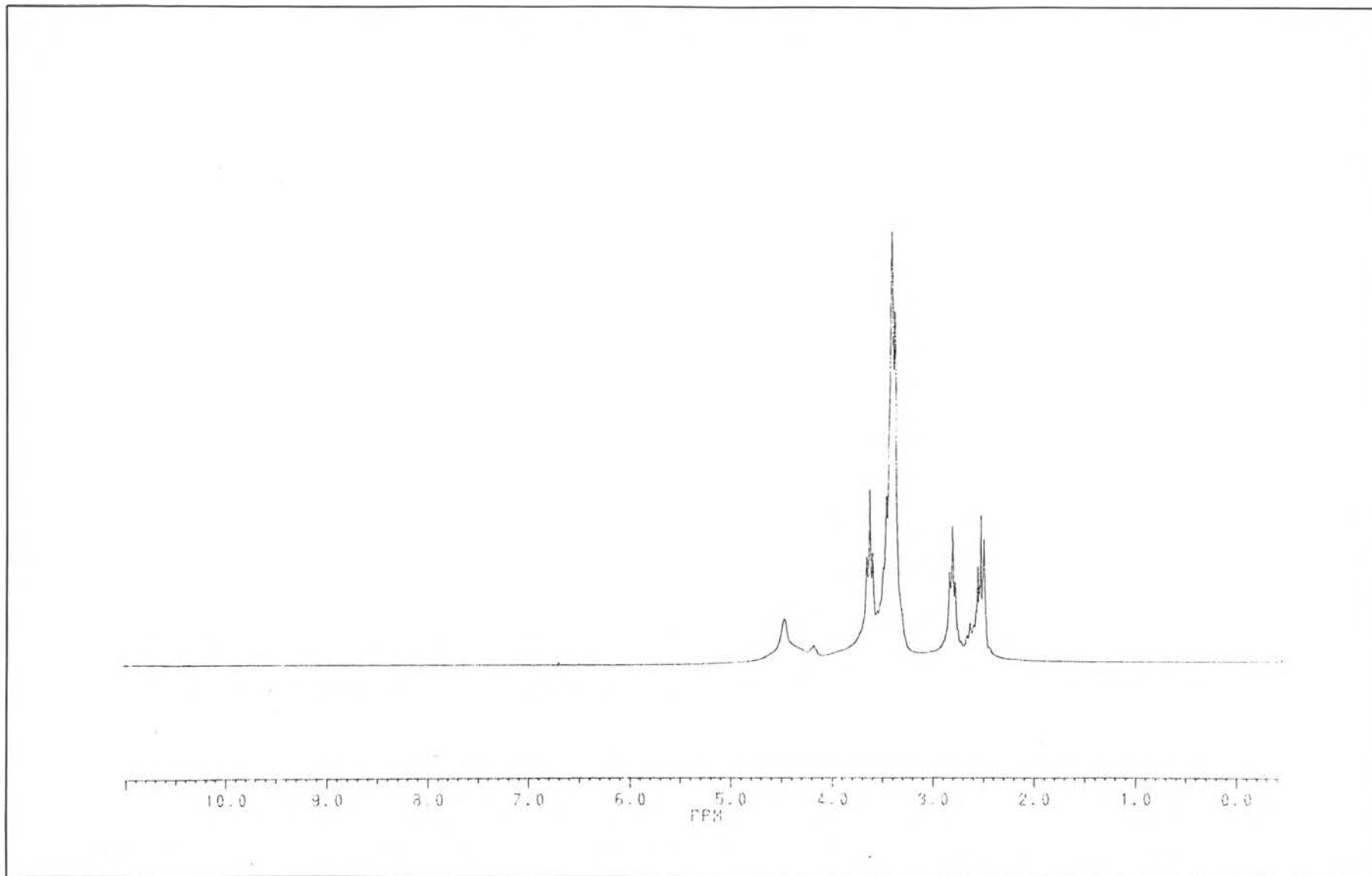


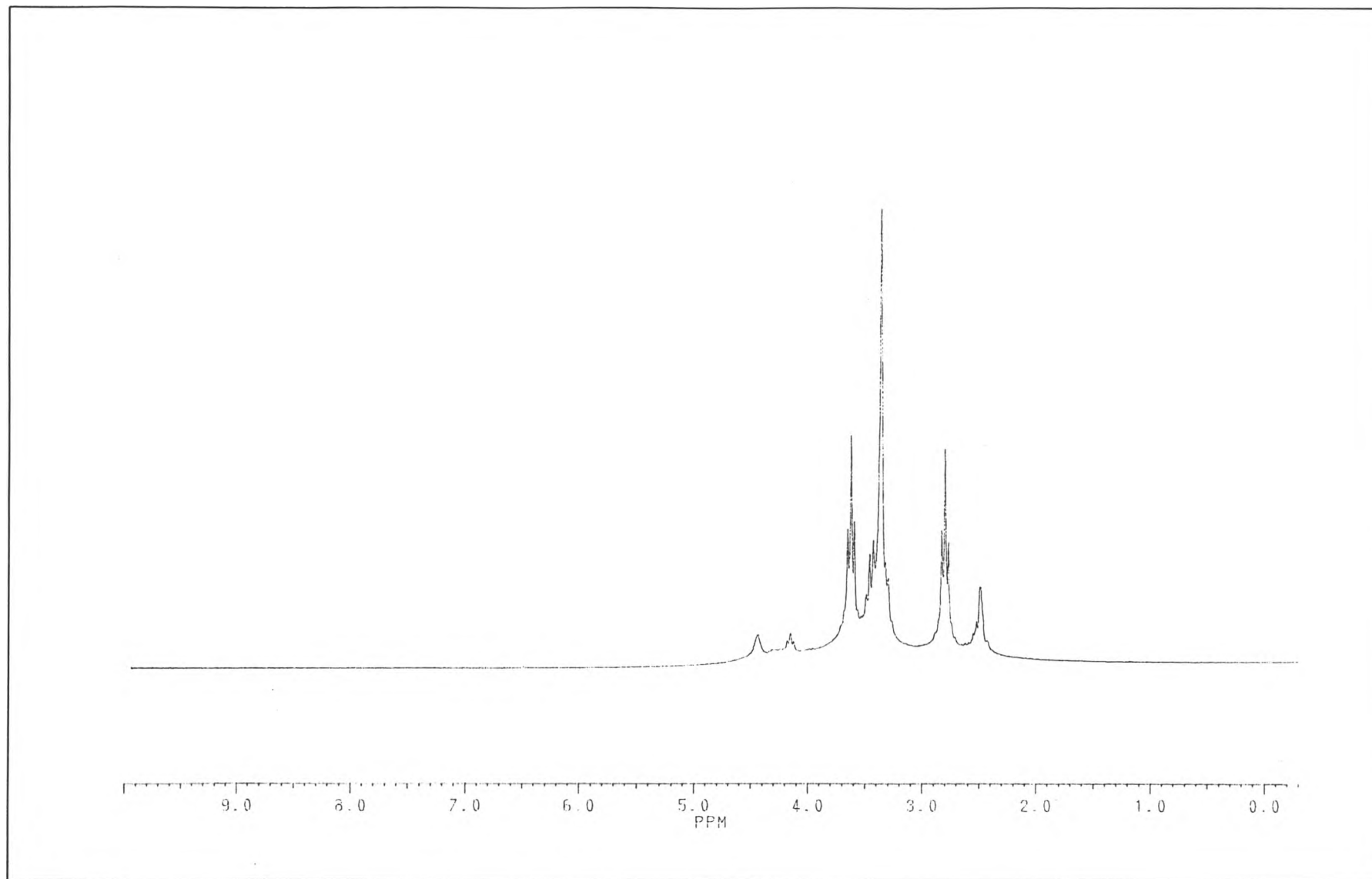
Figure A15 FT-IR Spectrum of sample 3 synthesized from rice husk ash 10.97  $\mu\text{m}$ , TEA and EG



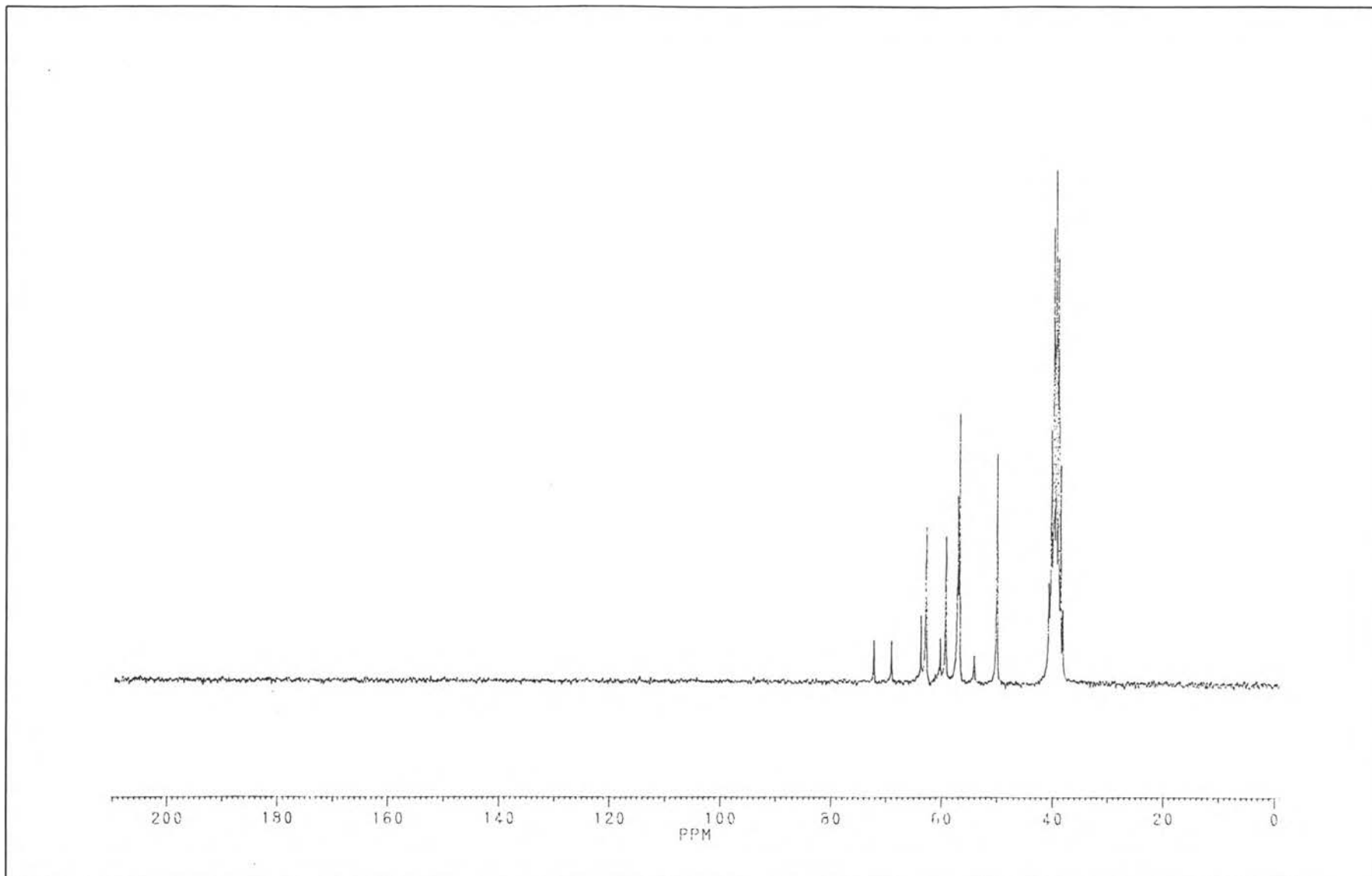
**Figure A16** <sup>1</sup>H-NMR (d<sub>6</sub>-DMSO) Spectrum of sample 1 synthesized from silica 0.007 μm, TEA and EG.



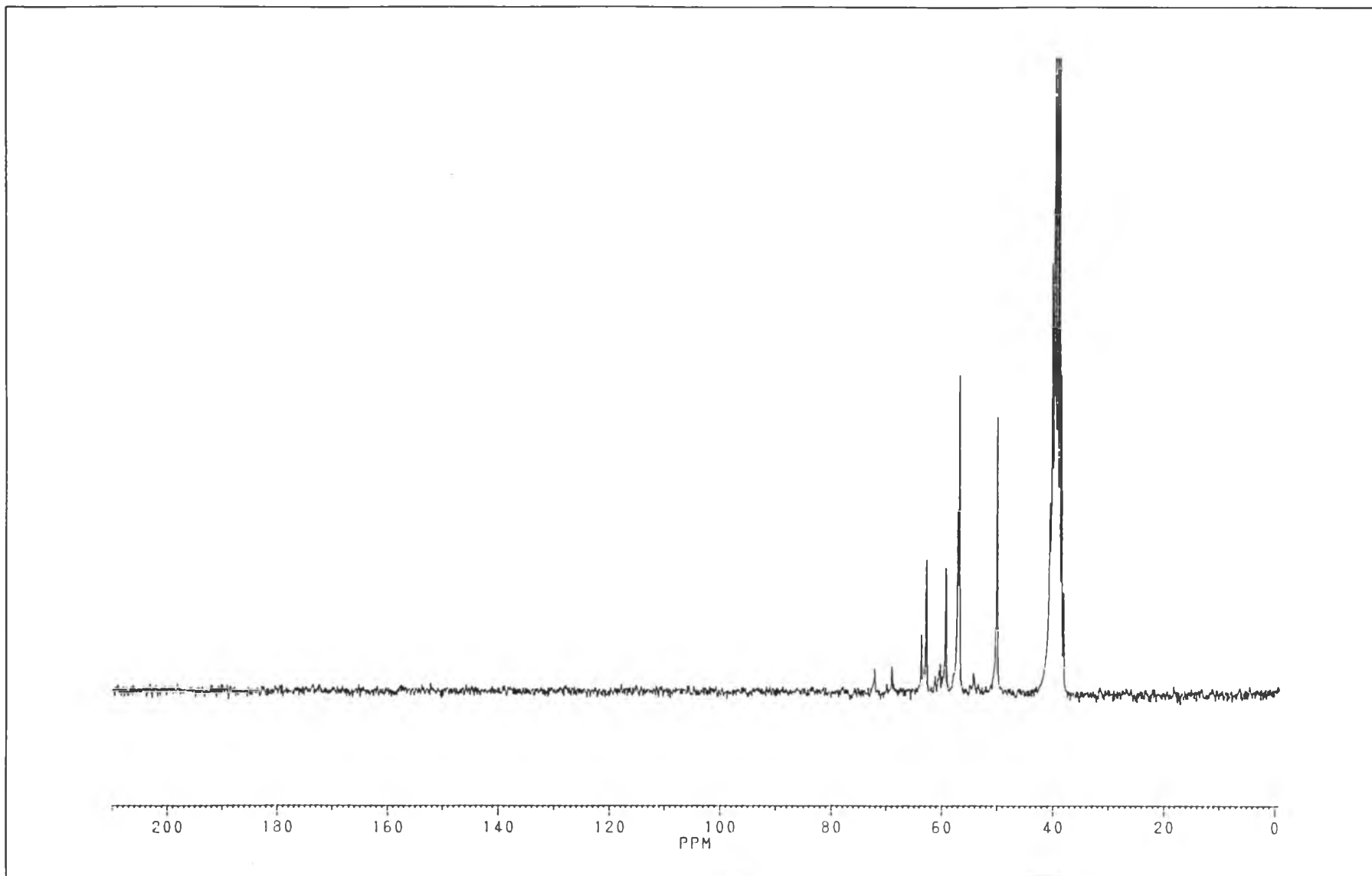
**Figure A17**  $^1\text{H-NMR}$  ( $d_6\text{-DMSO}$ ) Spectrum of sample 2 synthesized from silica 10.97  $\mu\text{m}$ , TEA and EG.



**Figure A18** <sup>1</sup>H-NMR (d<sub>6</sub>-DMSO) Spectrum of sample 3 synthesized from rice husk ash 13.47 μm, TEA and EG.

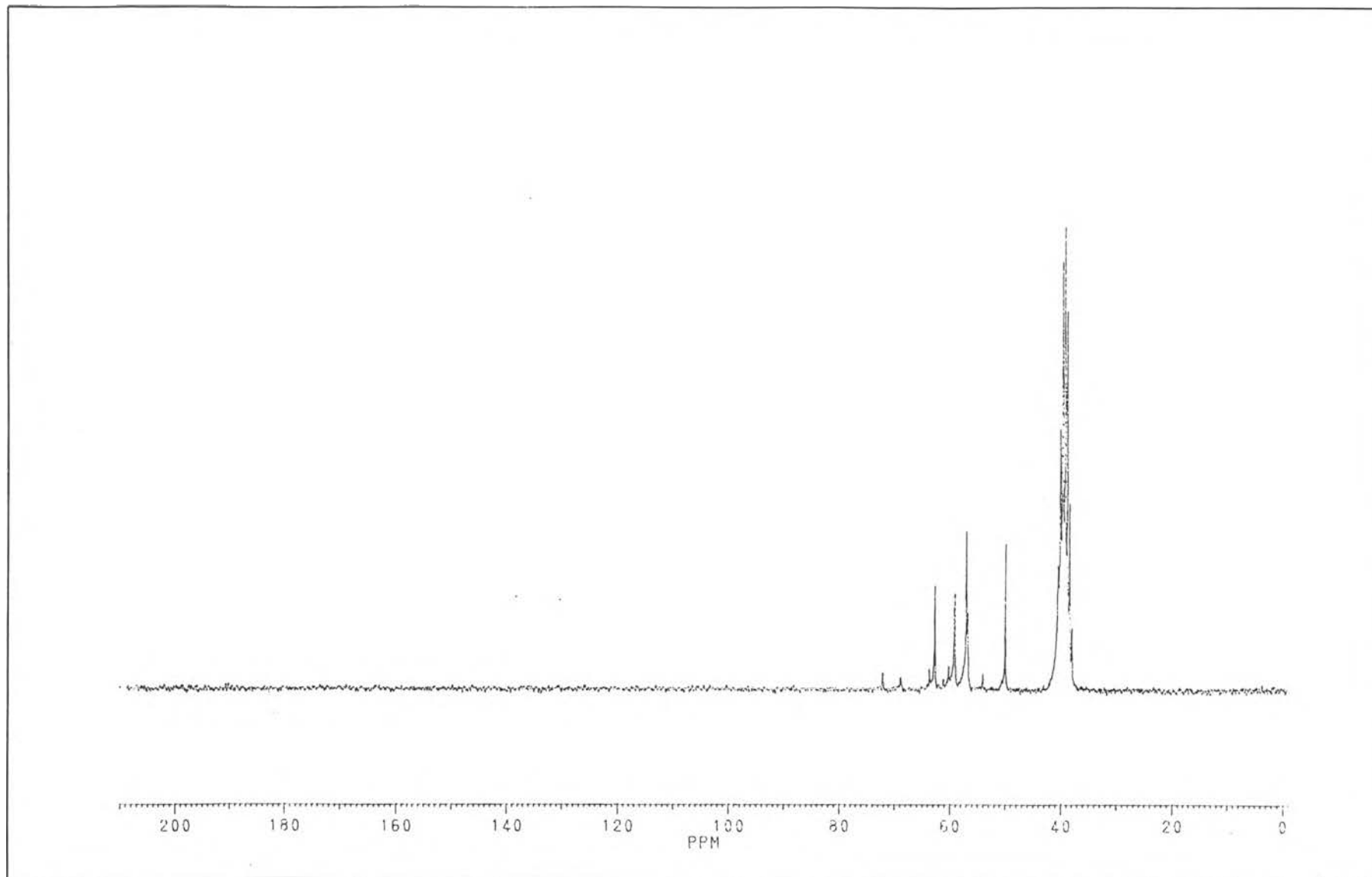


**Figure A19**  $^{13}\text{C}$ -NMR ( $\text{d}_6$ -DMSO) Spectrum of sample 1 synthesized from silica 0.007  $\mu\text{m}$ , TEA and EG.

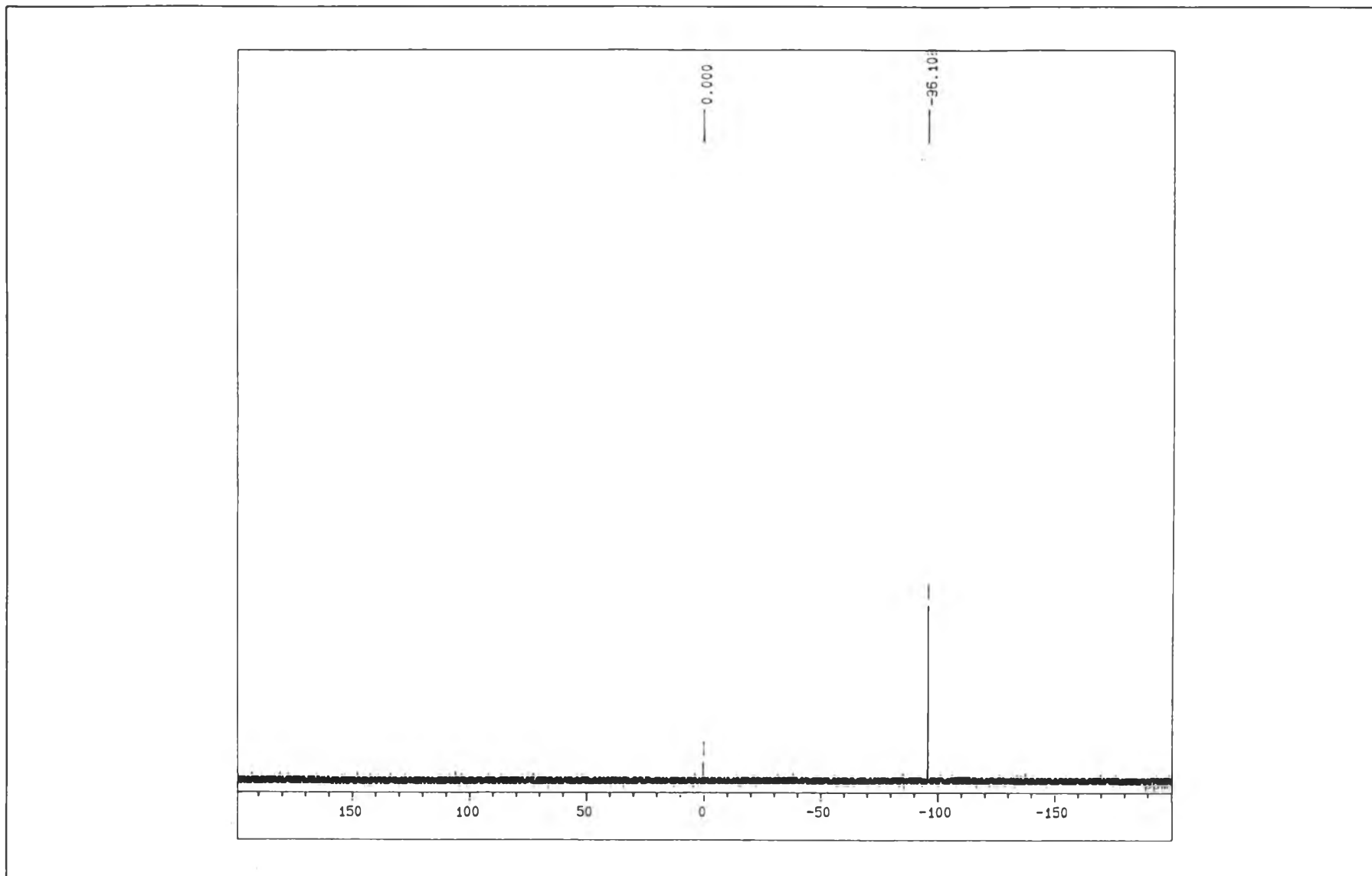


**Figure A20**  $^{13}\text{C}$ -NMR ( $d_6$ -DMSO) Spectrum of sample 2 synthesized from silica 10.97  $\mu\text{m}$ , TEA and EG





**Figure A21**  $^{13}\text{C}$ -NMR ( $\text{d}_6$ -DMSO) Spectrum of sample 3 synthesized from rice husk ash 13.47  $\mu\text{m}$ , TEA and EG.



**Figure A22**  $^{29}\text{Si}$ -NMR ( $d_6$ -DMSO) Spectrum of sample 1 synthesized from silica  $0.007\ \mu\text{m}$ , TEA and EG.

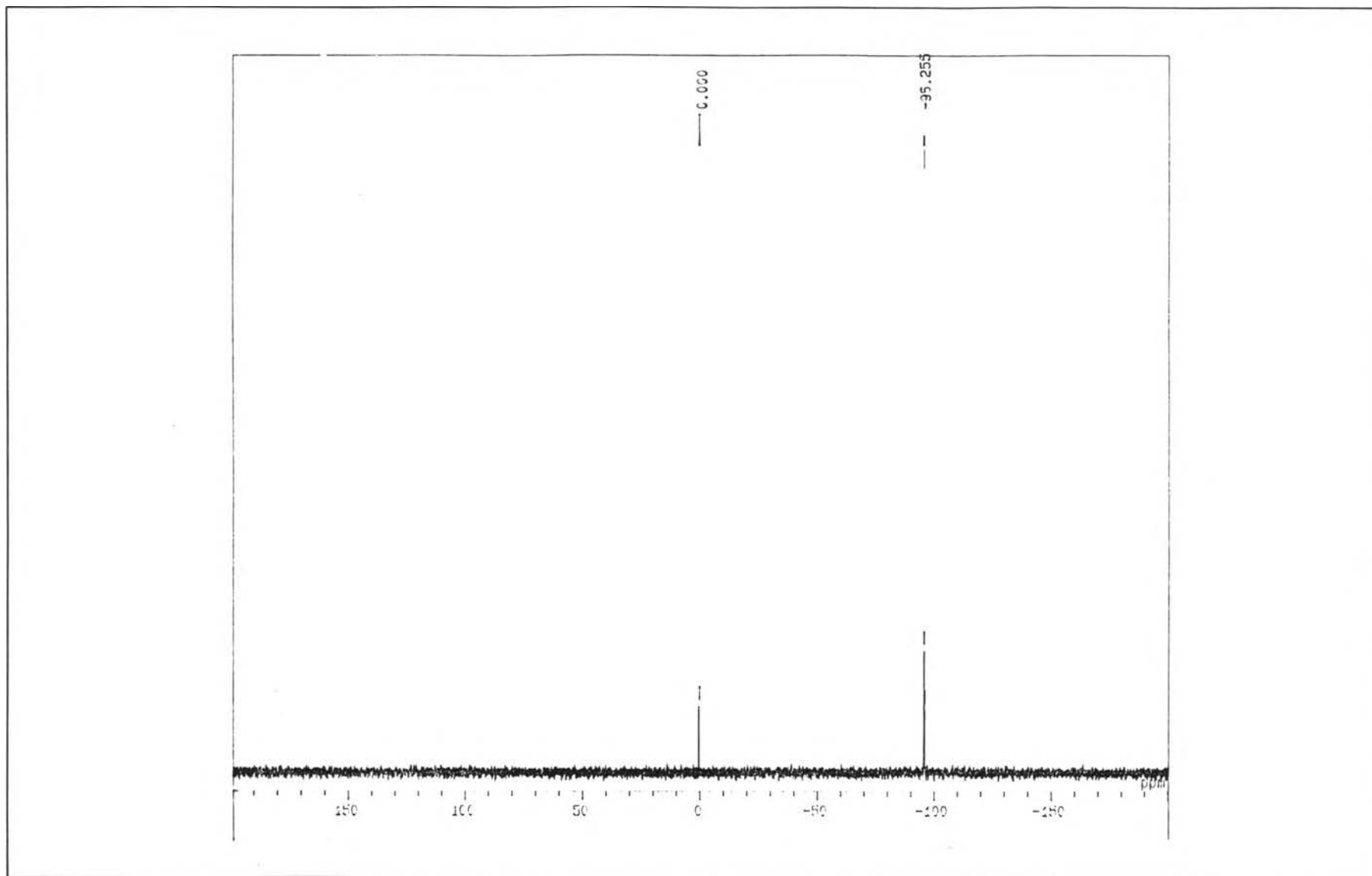
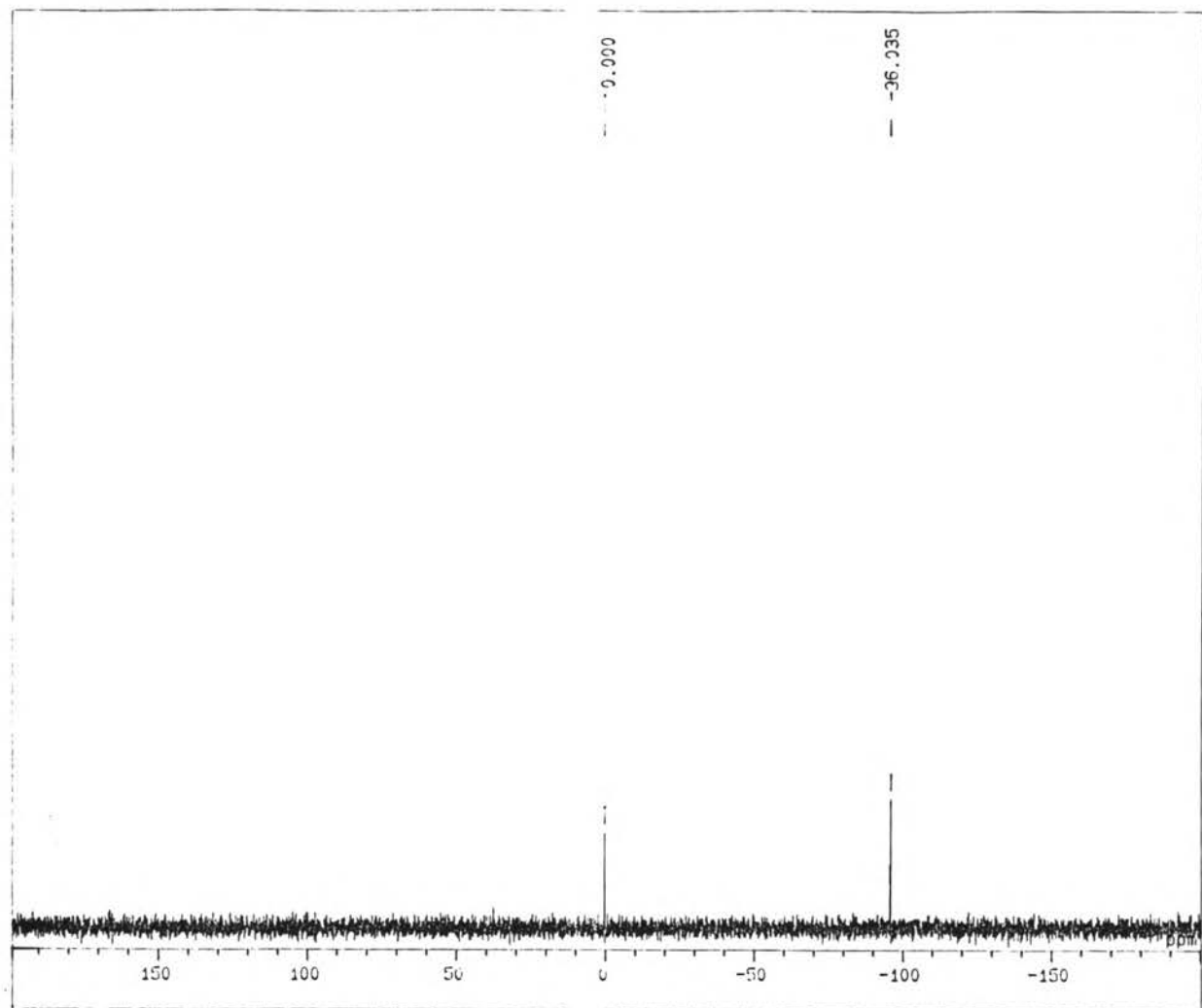
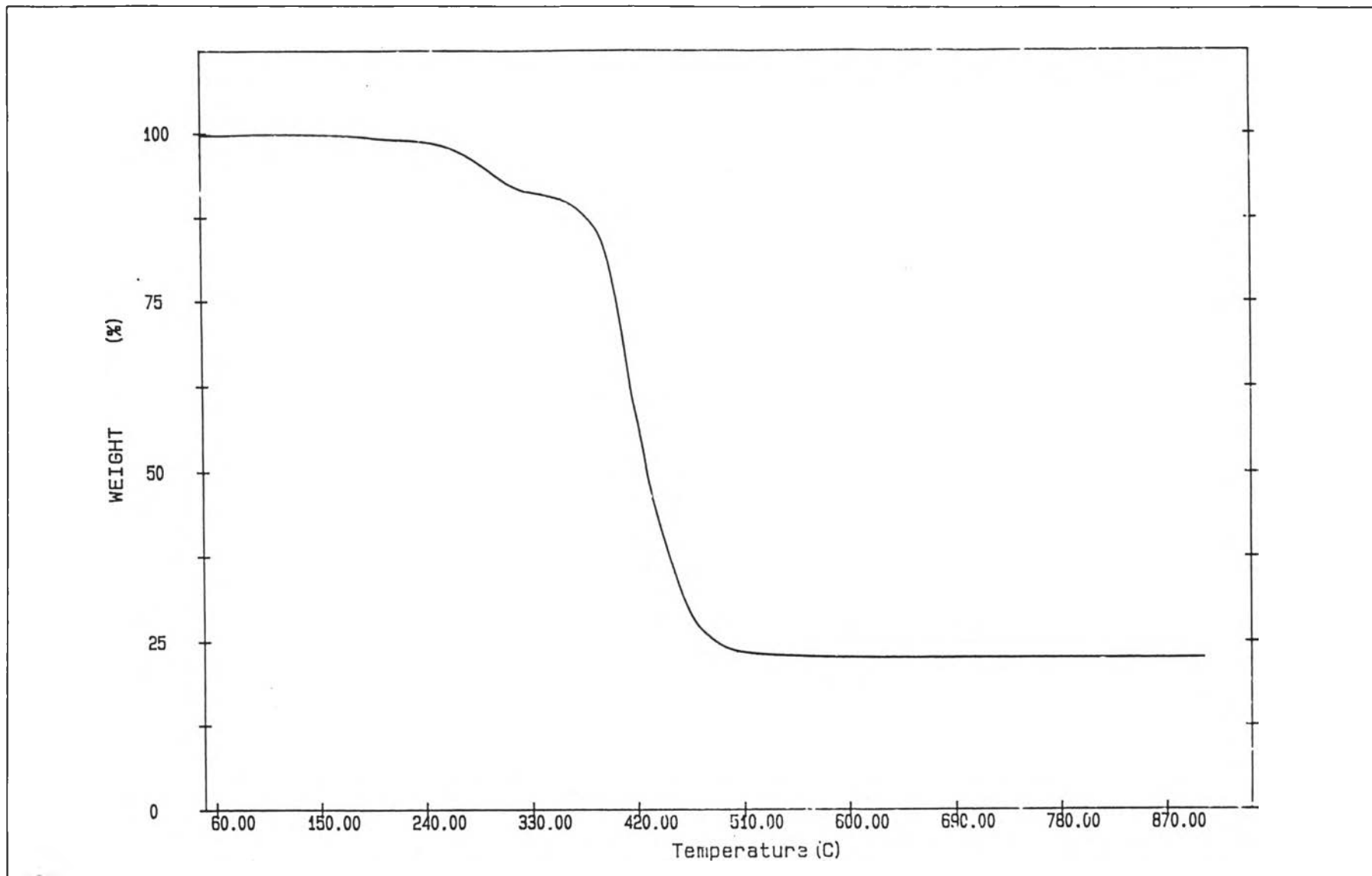


Figure A23  $^{29}\text{Si}$ -NMR ( $d_6$ -DMSO) Spectrum of sample 2 synthesized from silica  $10.97\mu\text{m}$ , TEA and EG.



**Figure A24**  $^{29}\text{Si}$ -NMR ( $d_6$ -DMSO) Spectrum of sample 3 synthesized from rice husk ash 13.47  $\mu\text{m}$ , TEA and EG.



**Figure A25** TGA Thermogram of sample 1 synthesized from silica 0.007  $\mu\text{m}$ , TEA and EG.

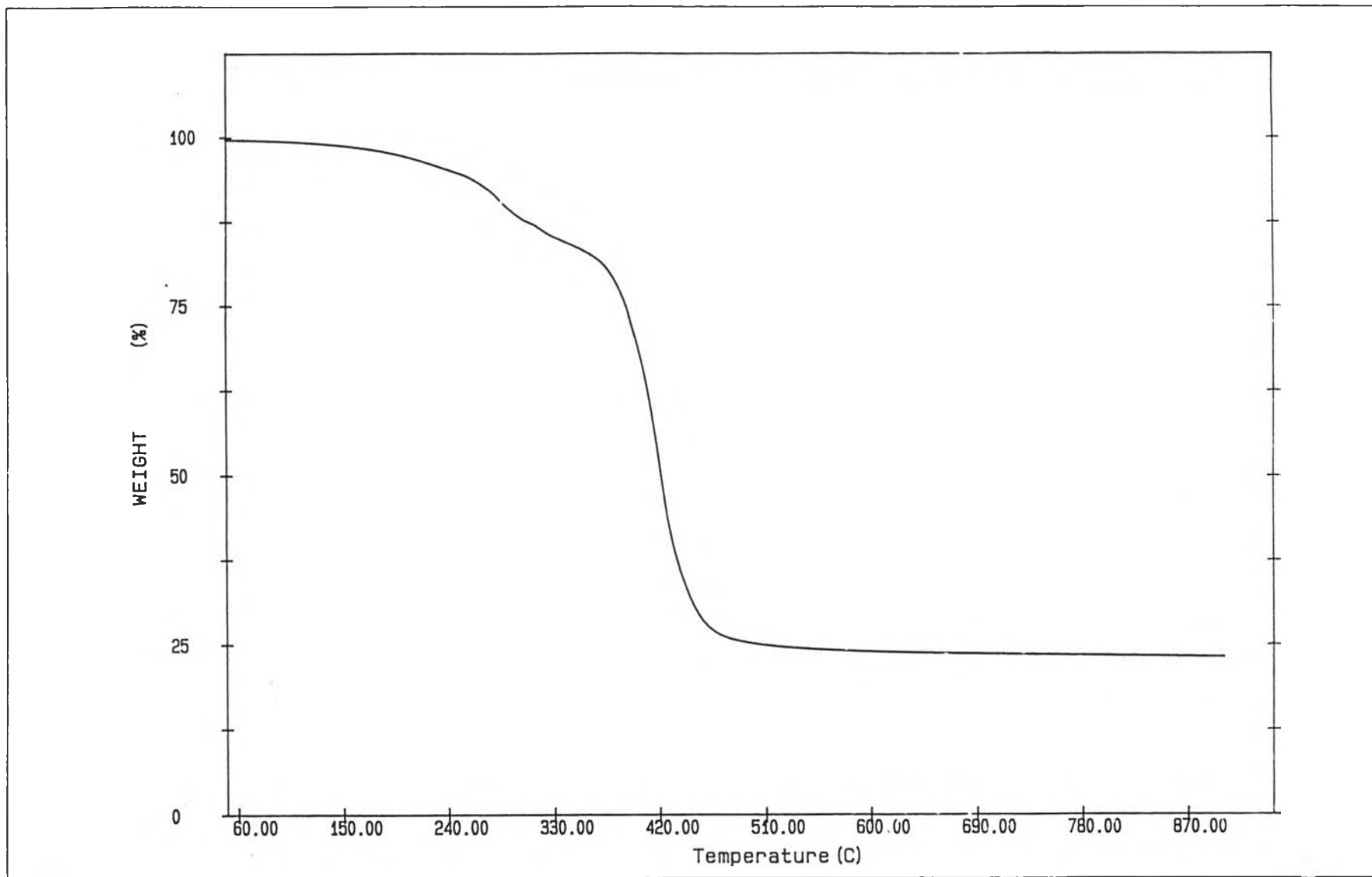


Figure A26 TGA Thermogram of sample 2 synthesized from silica 10.97  $\mu\text{m}$ , TEA and EG.

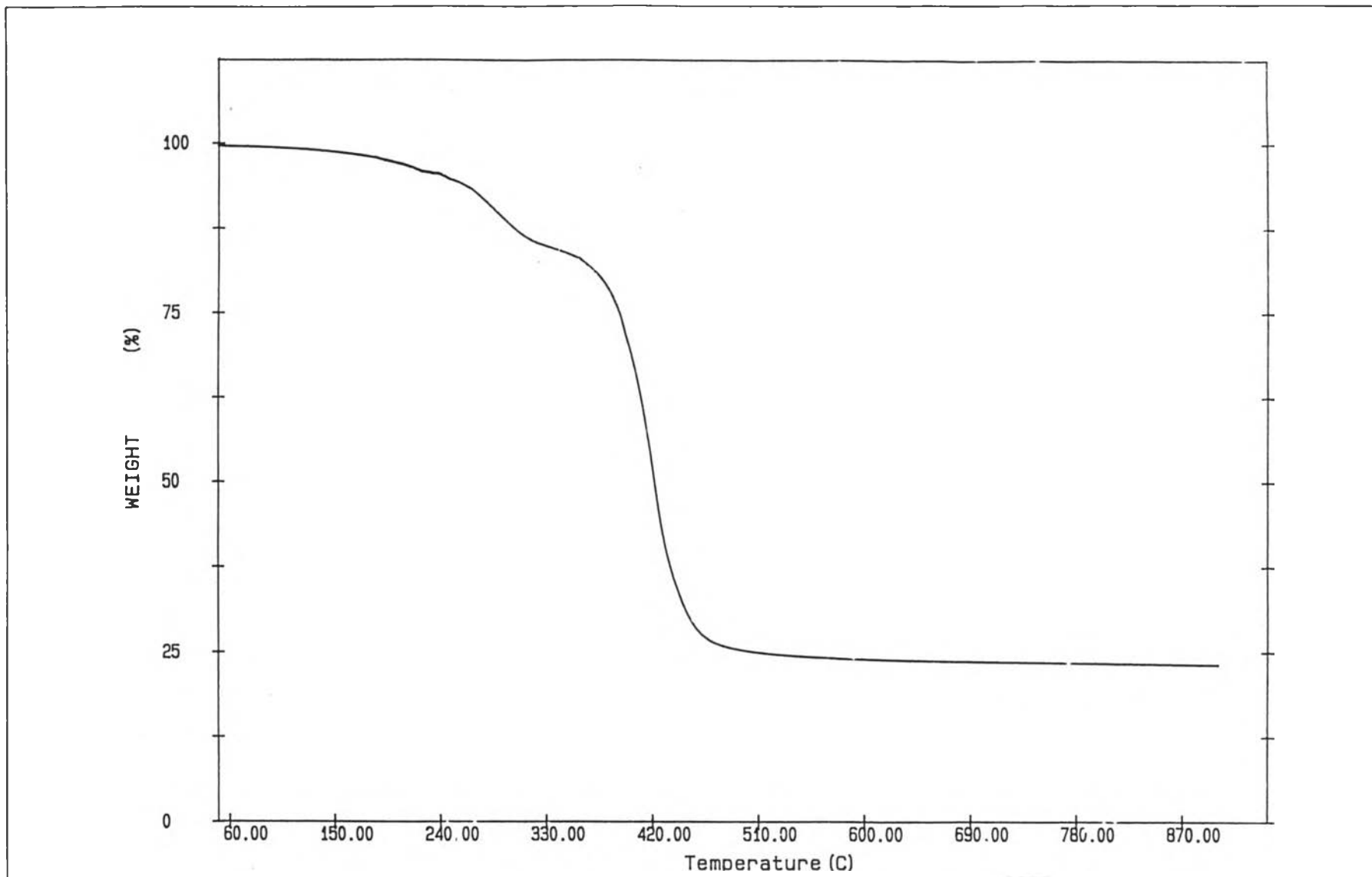


Figure A27 TGA Thermogram of sample 3 synthesized from rice husk ash 13.47  $\mu\text{m}$ , TEA and EG.

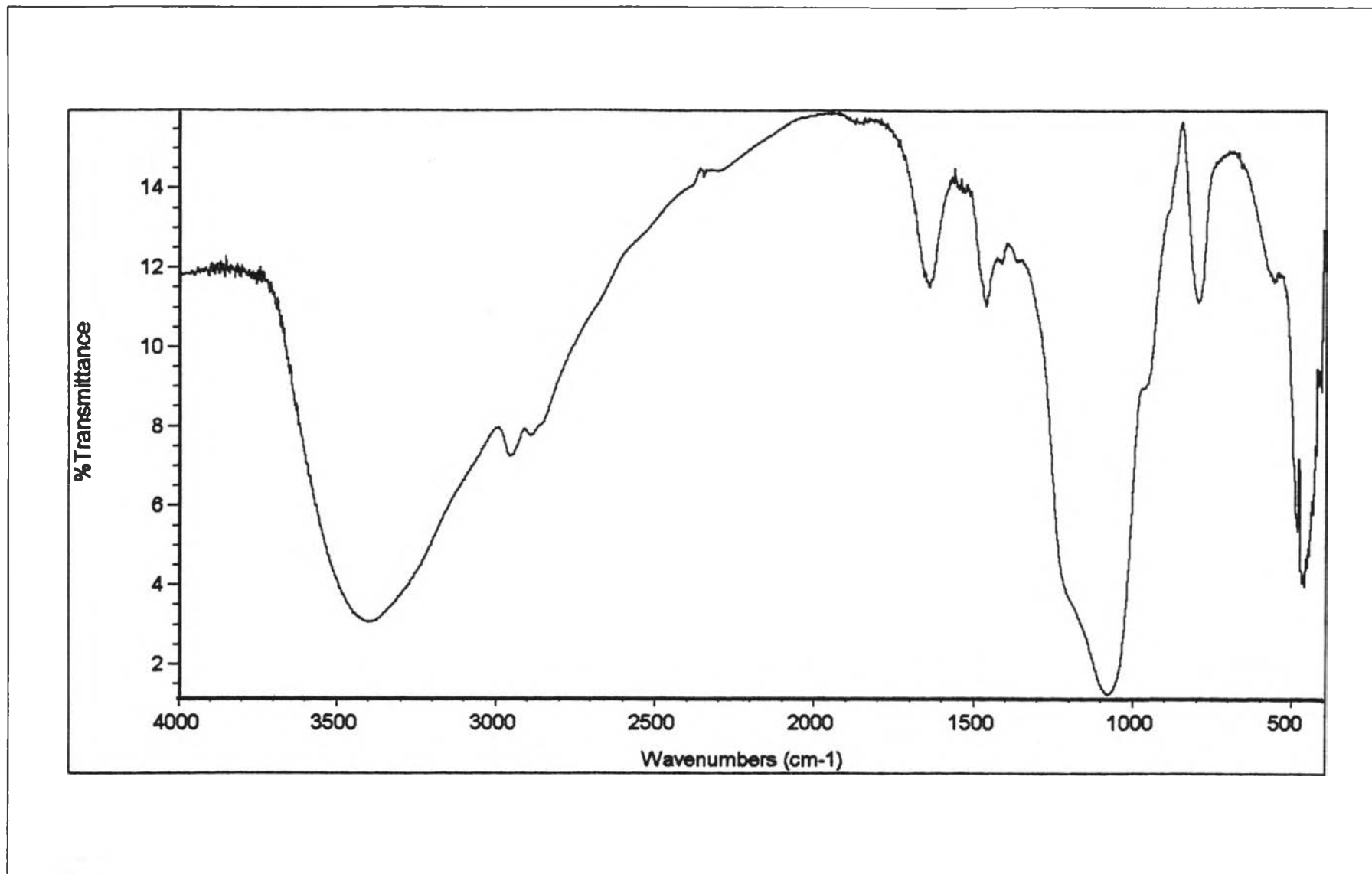


Figure A28 FT-IR Spectrum of sample 4 synthesized from silica 0.007  $\mu\text{m}$ , DEA and EG.



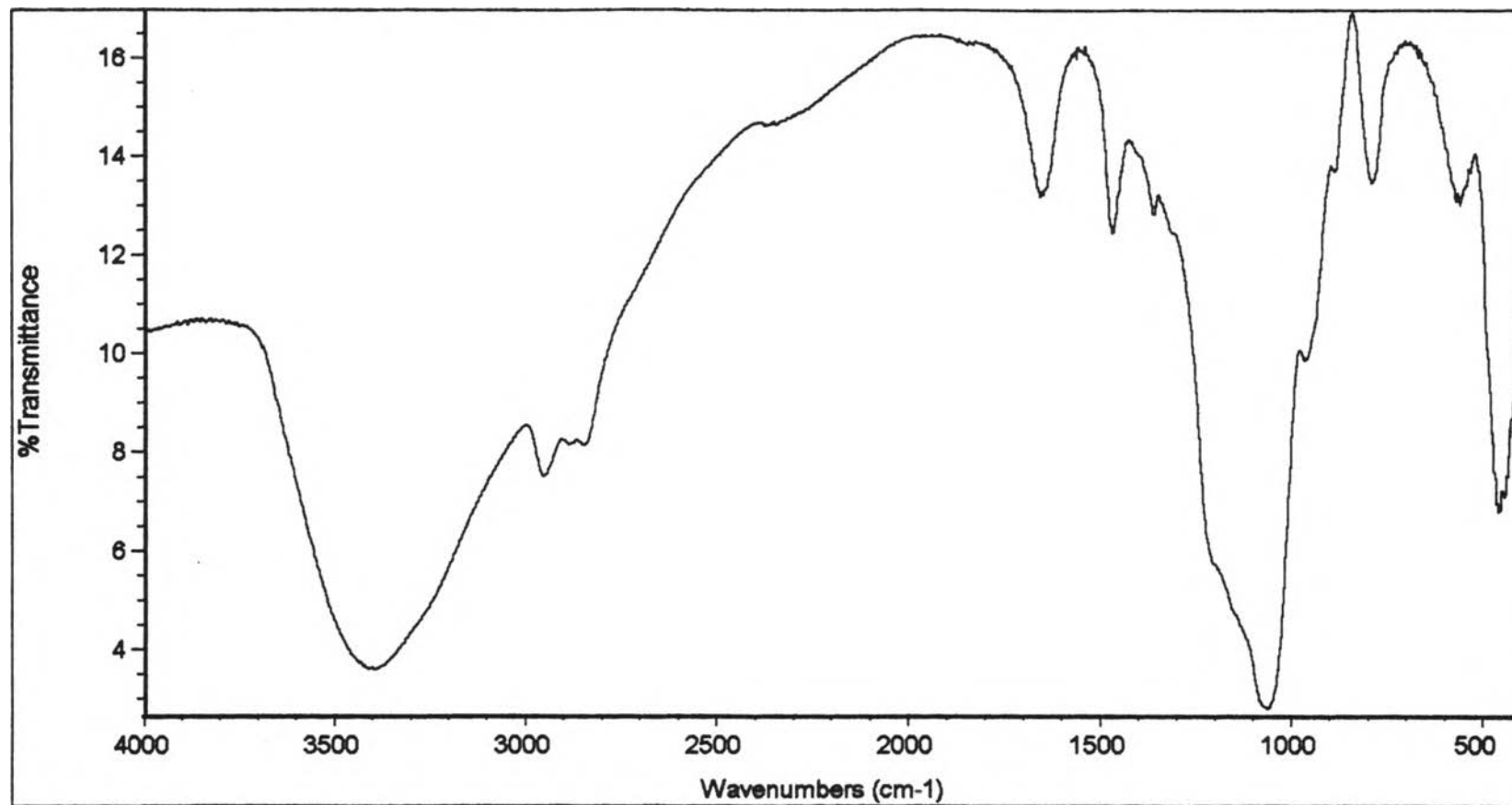


Figure A29 FT-IR Spectrum of sample 5 synthesized from silica 10.47  $\mu\text{m}$ , DEA and EG.

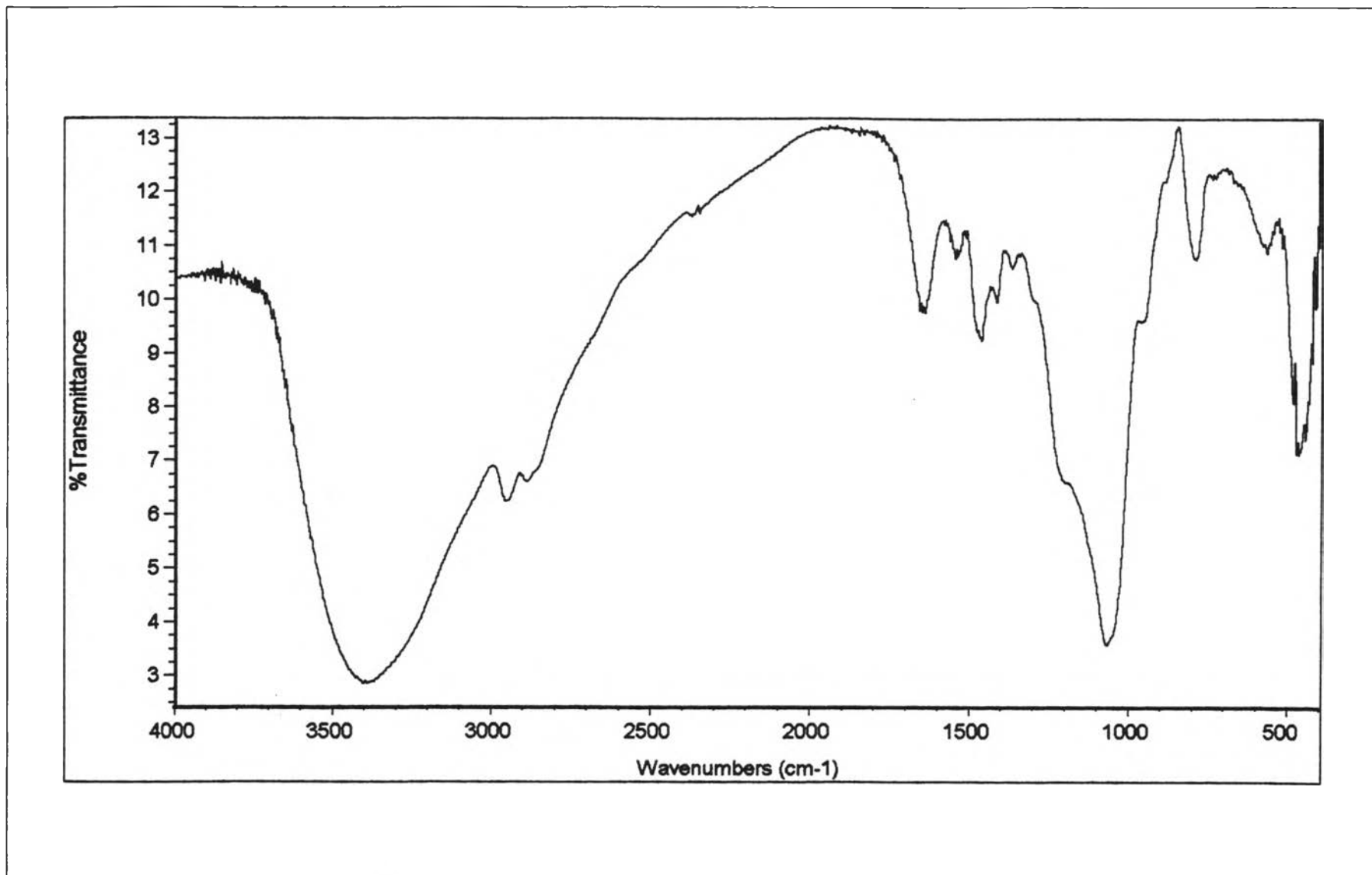
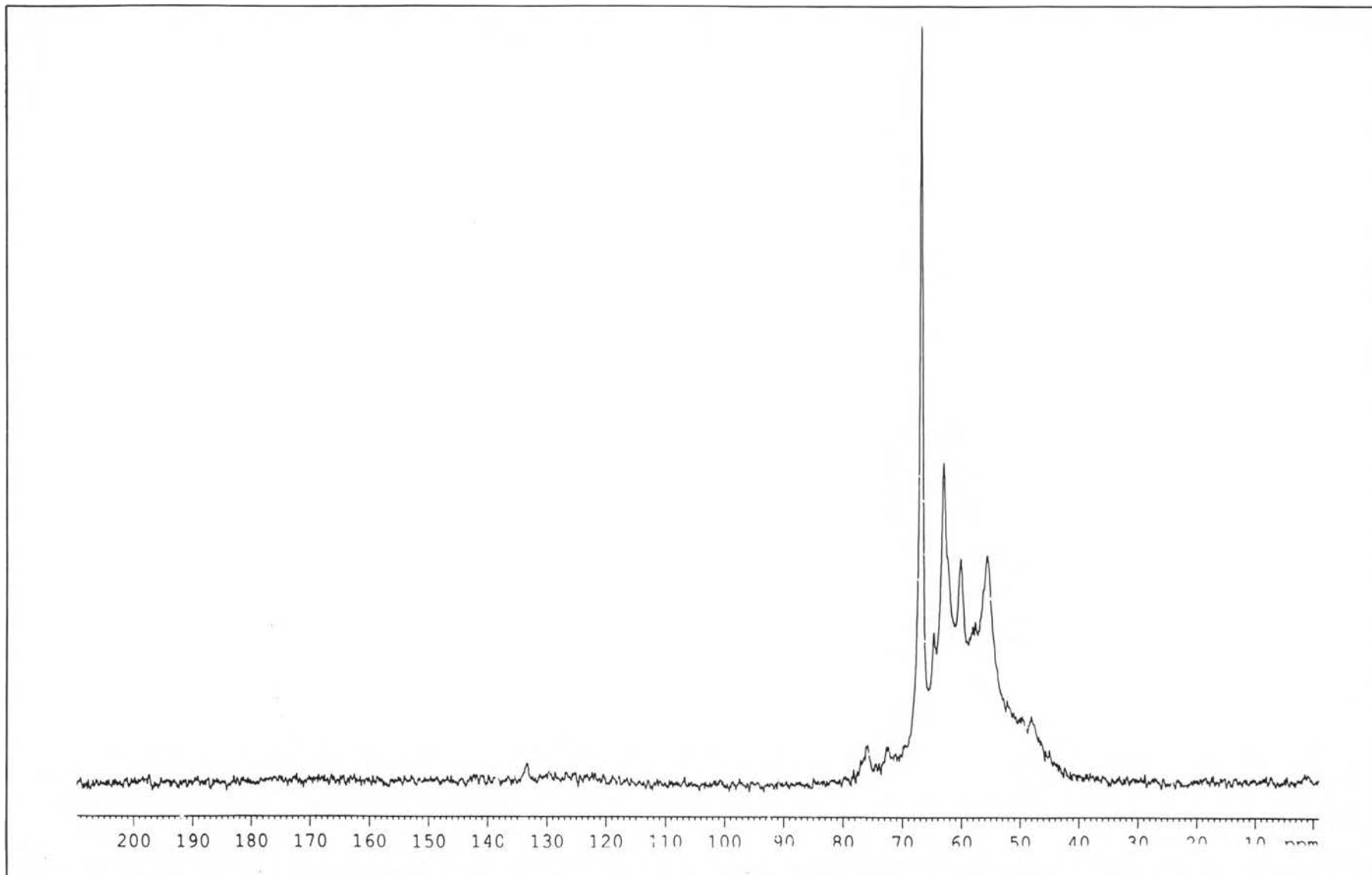
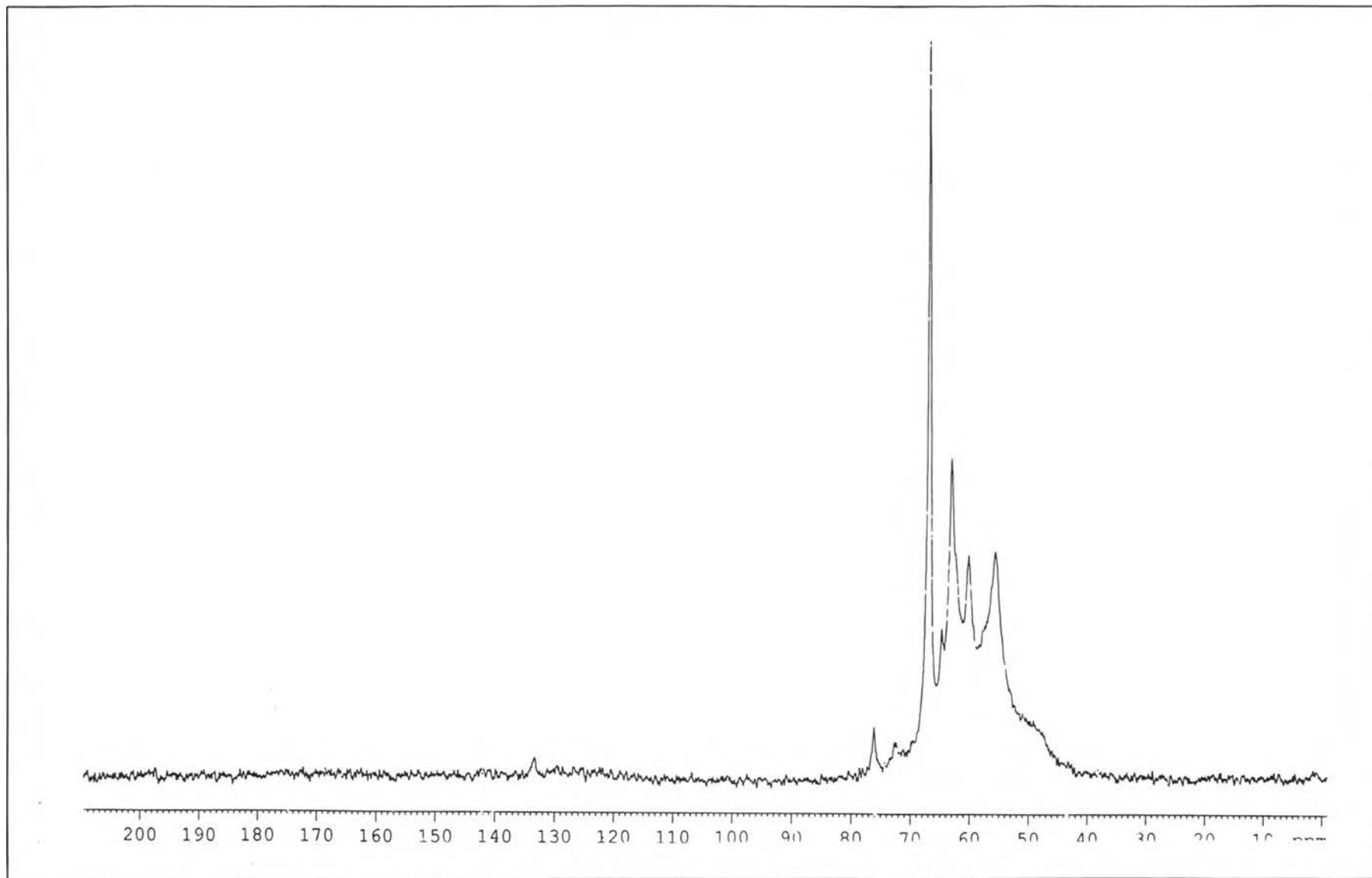


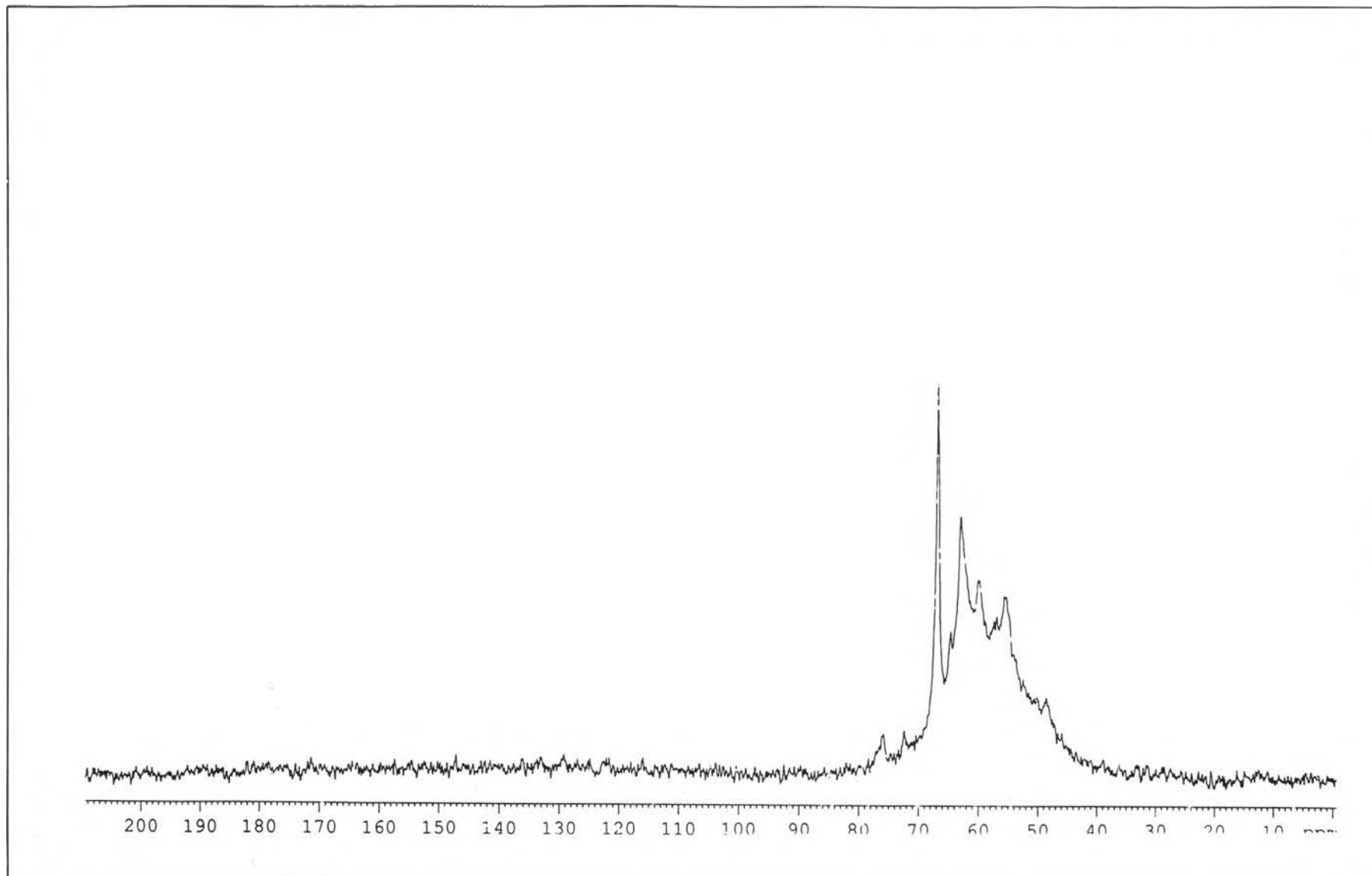
Figure A30 FT-IR Spectrum of sample 6 synthesized from rice husk ash 13.47  $\mu\text{m}$ , DEA and EG.



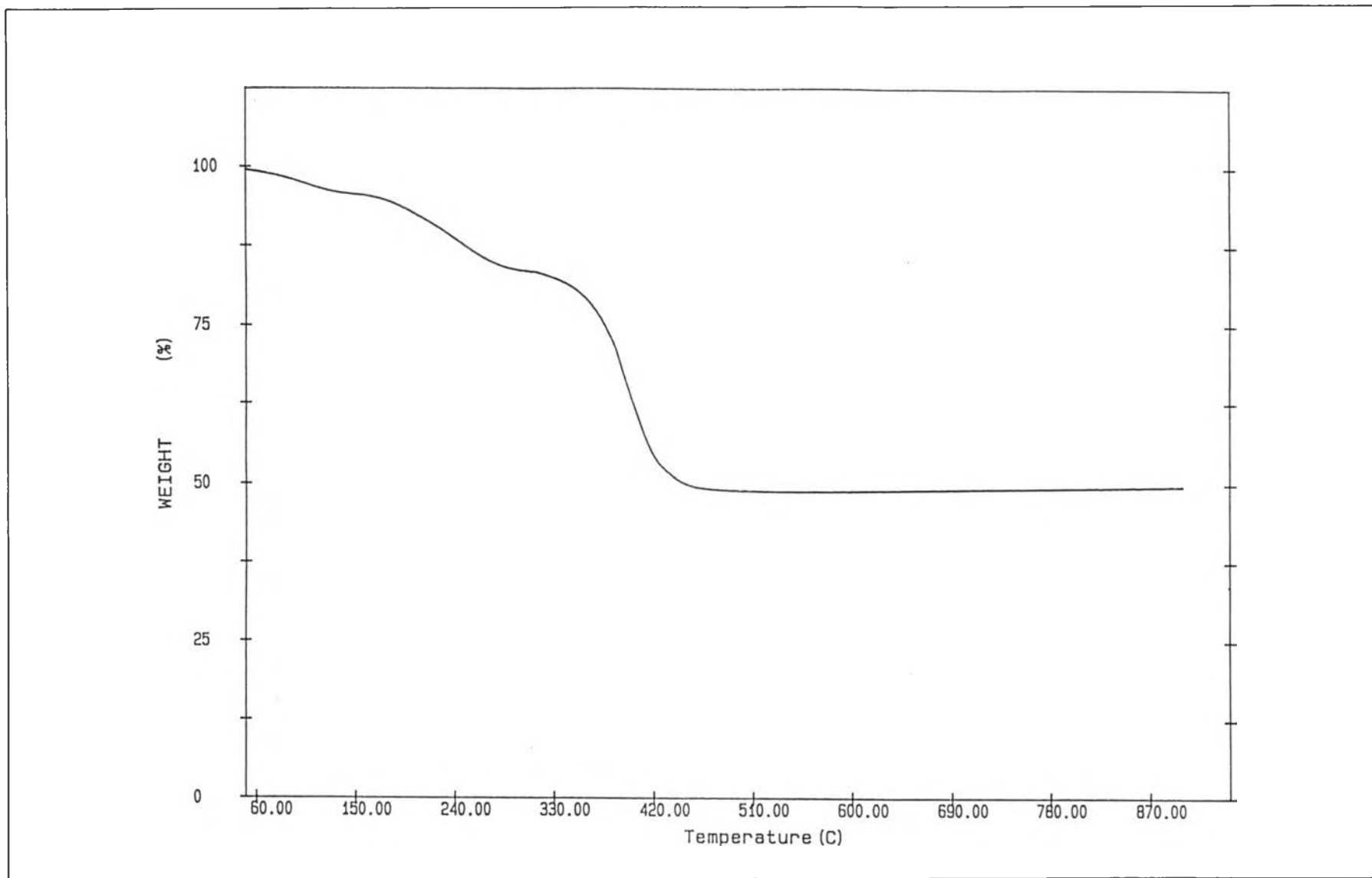
**Figure A31**  $^{13}\text{C}$ -NMR Solid State Spectrum of sample 4 synthesized from silica 0.007 $\mu\text{m}$ , DEA and EG.



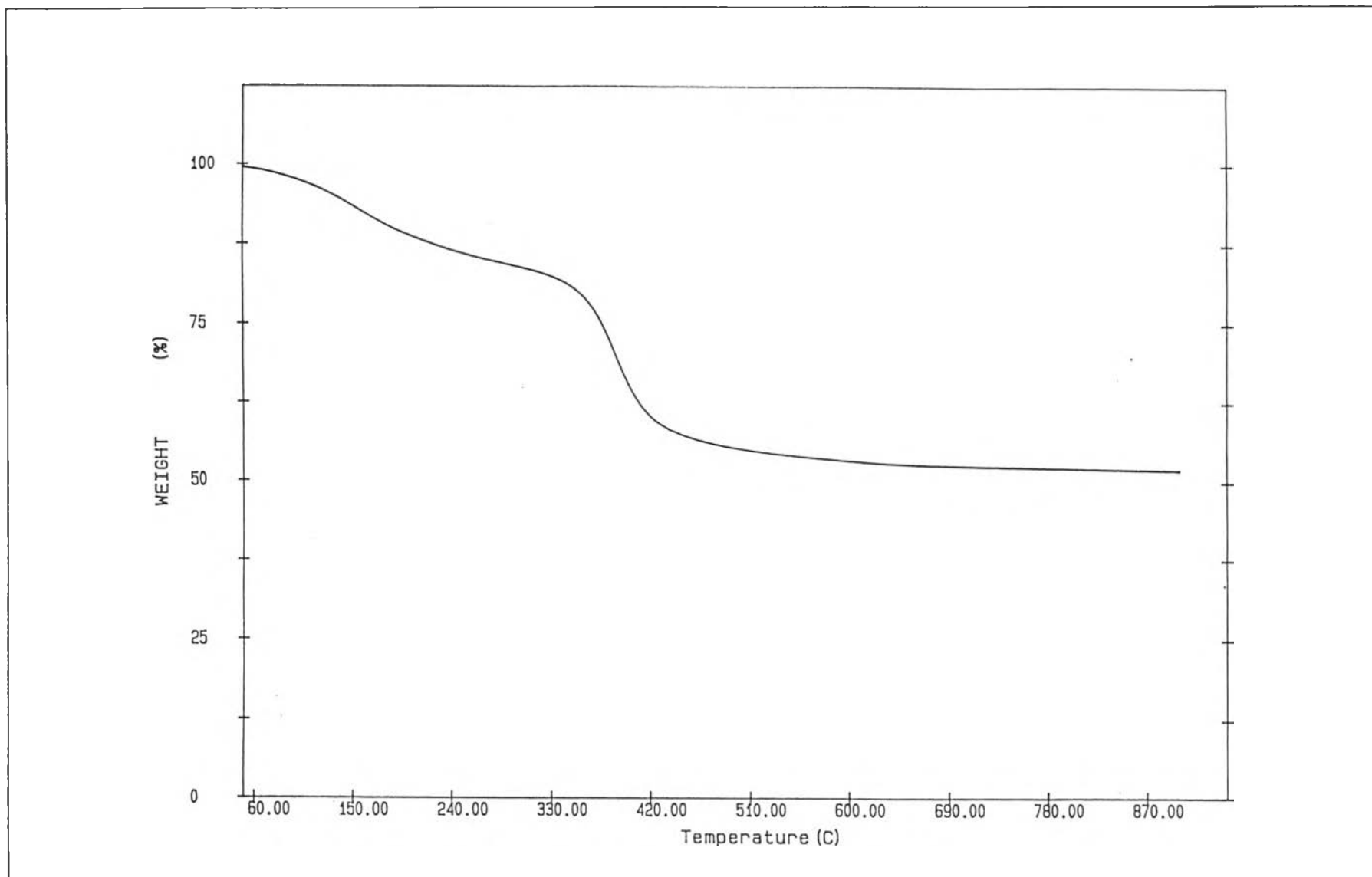
**Figure A32**  $^{13}\text{C}$ -NMR Solid State Spectrum of sample 5 synthesized from silica 10.97  $\mu\text{m}$ , DEA and EG.



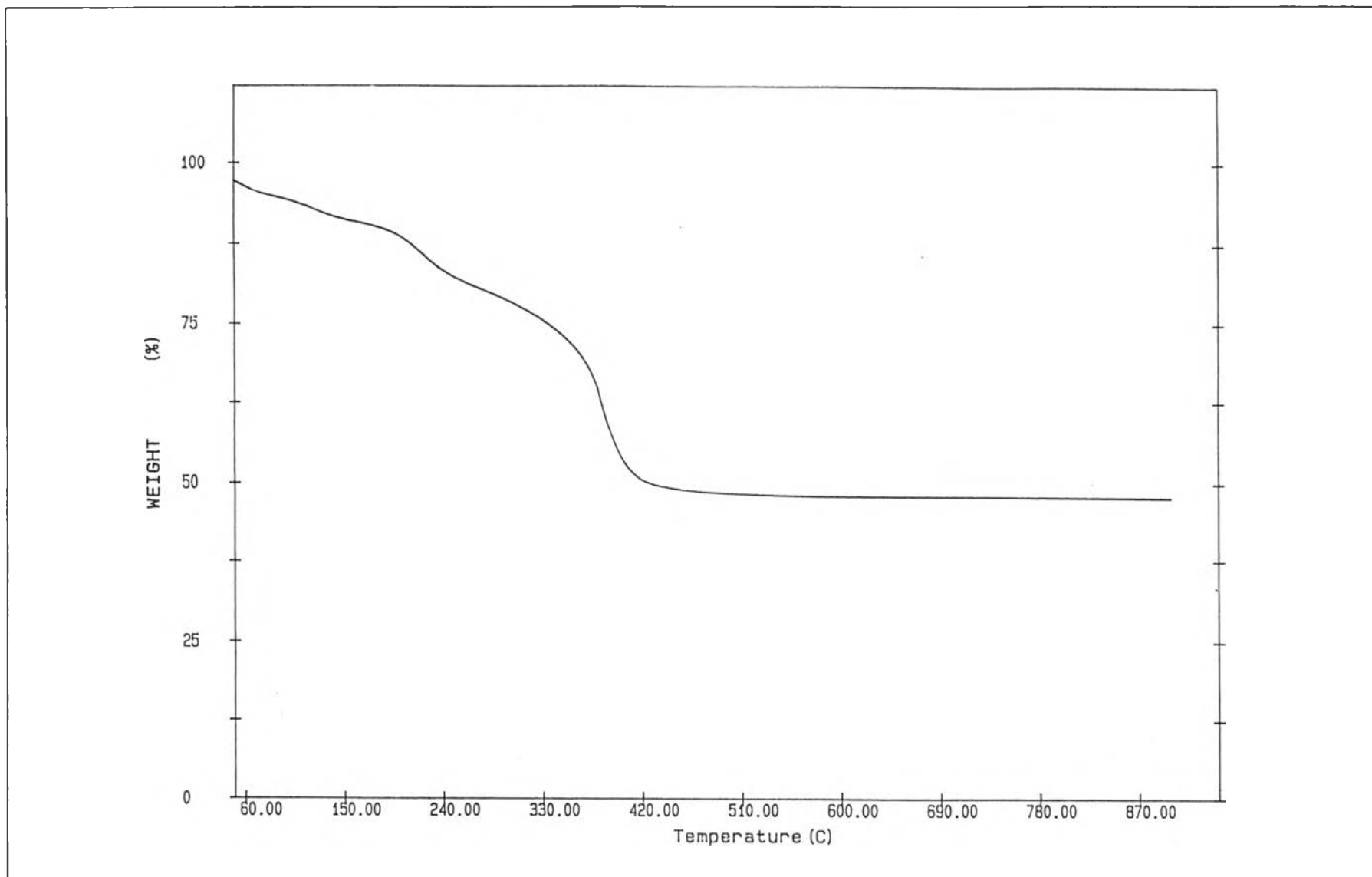
**Figure A33**  $^{13}\text{C}$ -NMR Solid State Spectrum of sample 6 synthesized from rice husk ash 13.47  $\mu\text{m}$ , DEA and EG.



**Figure A34** TGA Thermogram of sample 4 synthesized from silica 0.007  $\mu\text{m}$ , DEA and EG.



**Figure A35** TGA Thermogram of sample 5 synthesized from silica 10.97  $\mu\text{m}$ , DEA and EG.



**Figure A36** TGA Thermogram of sample 6 synthesized from rice husk ash 13.47  $\mu\text{m}$ , DEA and EG.





## VITA

Miss Pensiri Navasetthakul was born on August 6, 1971 in Narathiwat, Thailand. She received the Degree of Bachelor of Science in Polymer Science from Department of Polymer Science, Faculty of Science, Prince of Songkla University in 1993. She became a student in graduate school at Multidisciplinary of Petrochemistry and Polymer, major Polymer Science, Chulalongkorn University in 1994 and she graduated with a Master's Degree of Polymer Science in 1997.