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APPENDICES

APPENDIX A

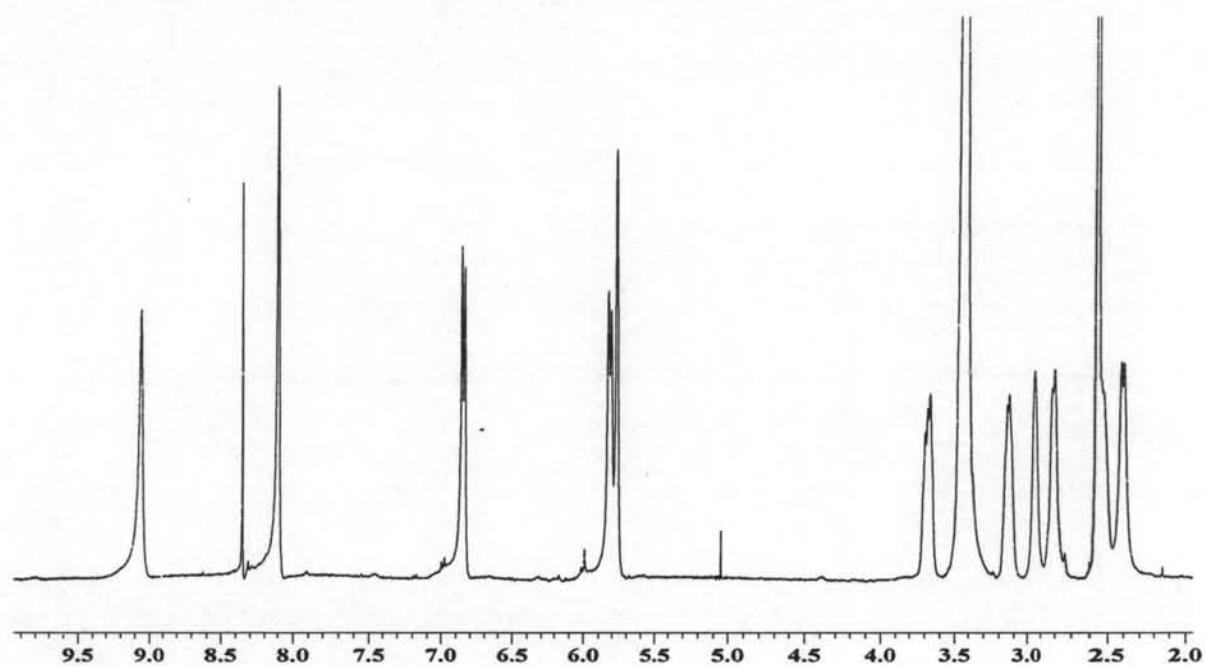


Figure A.1 ^1H NMR spectrum of ZnL in $\text{DMSO-}d_6 + \text{CDCl}_3$

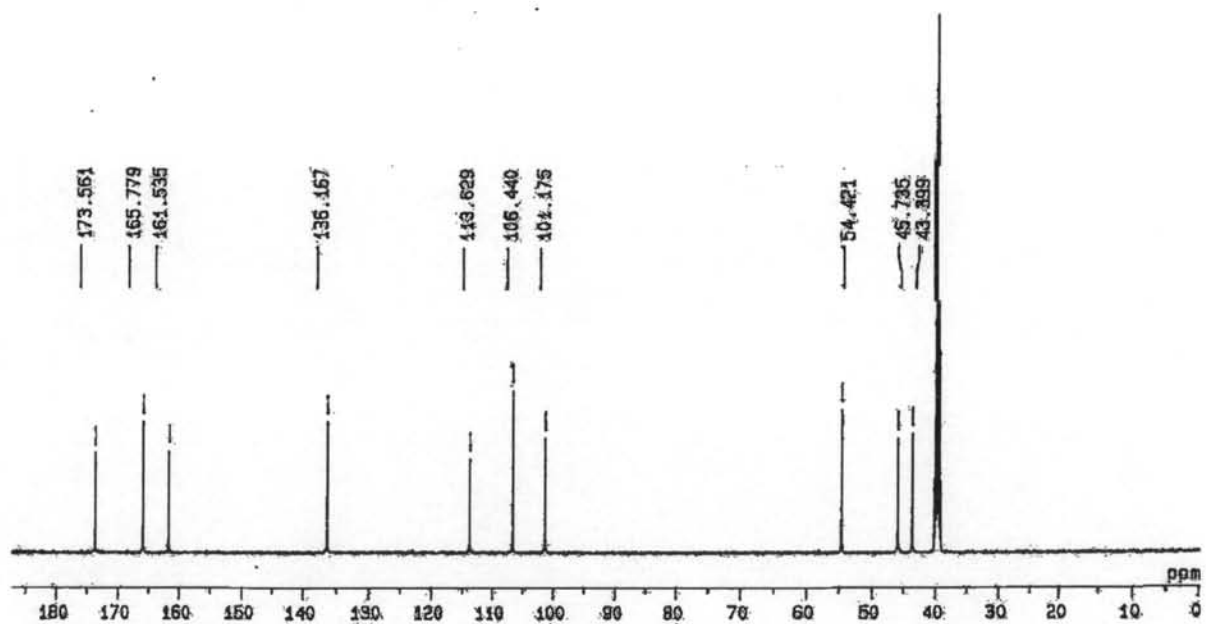


Figure A.2 ^{13}C -NMR spectrum of ZnL in $\text{DMSO-}d_6$

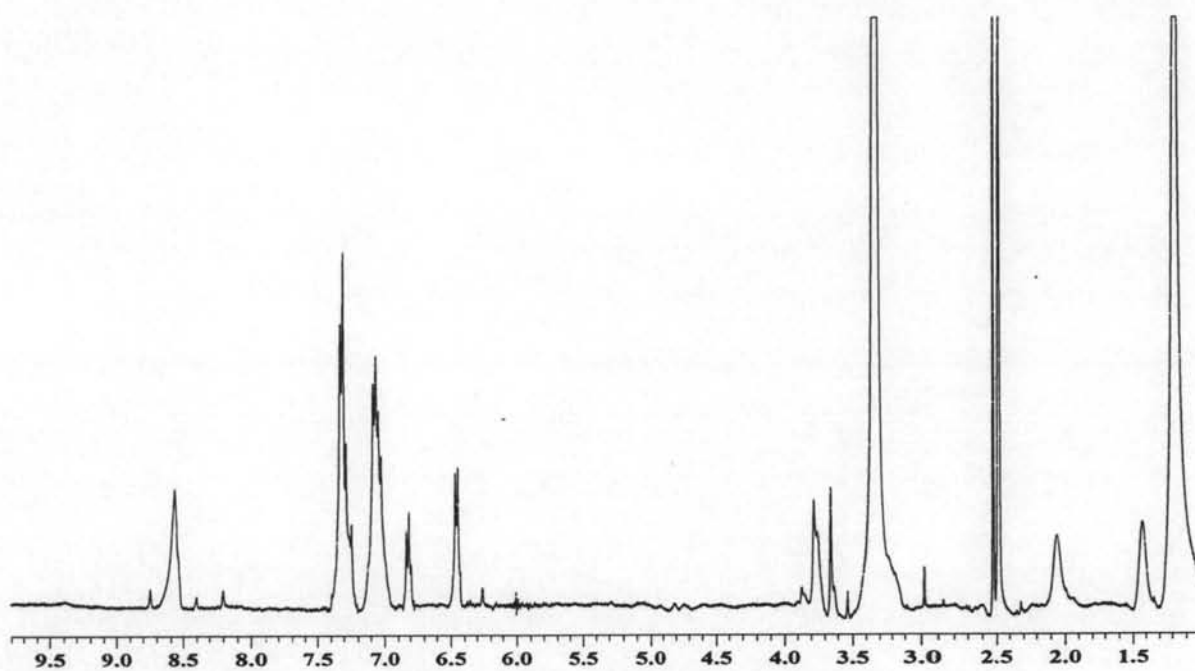


Figure A.3 ^1H NMR spectrum of ZnL-MDI (1:2) in $\text{DMSO-}d_6$

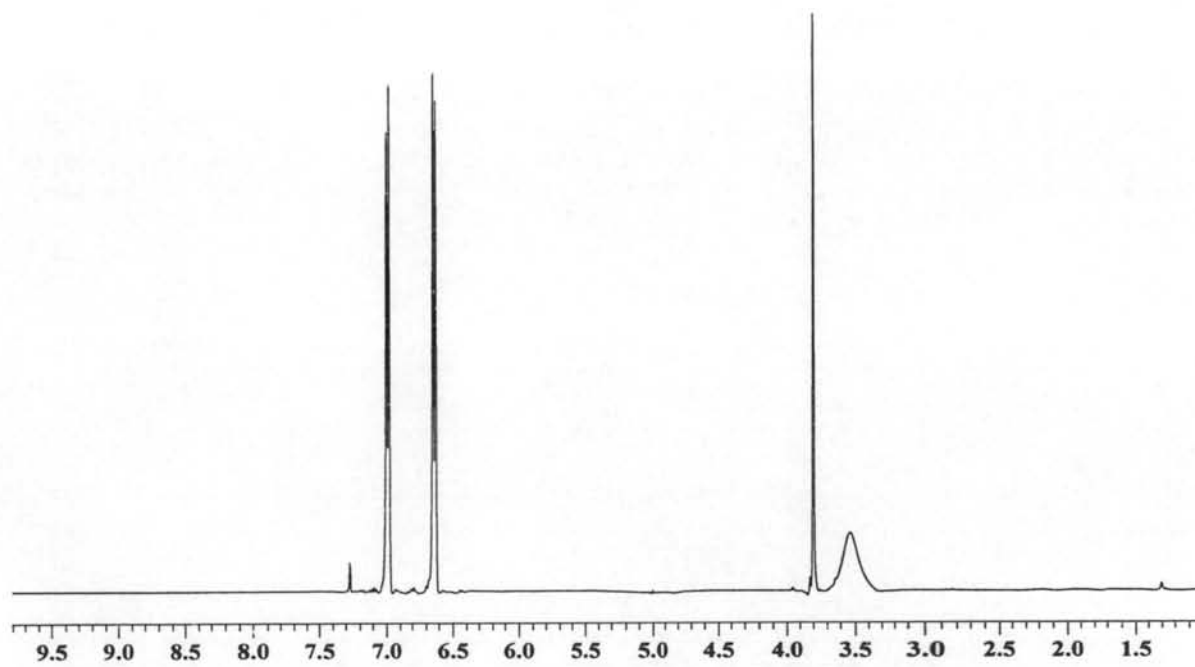


Figure A.4 ^1H NMR spectrum of MTDA in $\text{DMSO-}d_6 + \text{CDCl}_3$

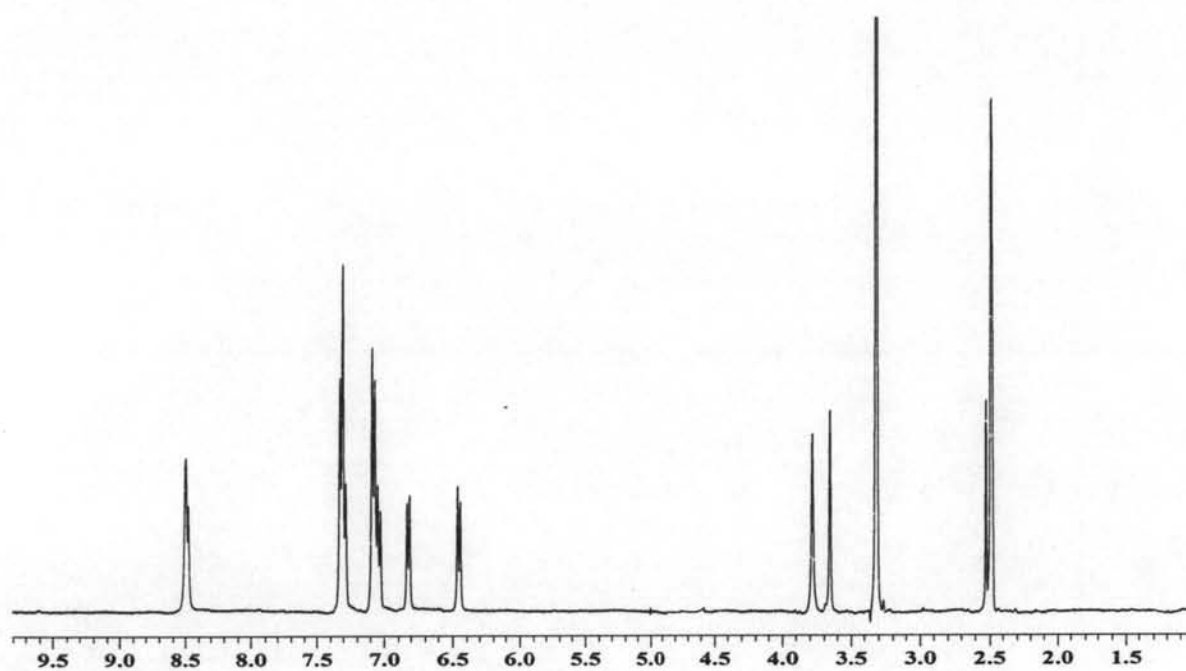


Figure A.5 ^1H NMR spectrum of MDI-MTDA (1:1) in $\text{DMSO-}d_6$

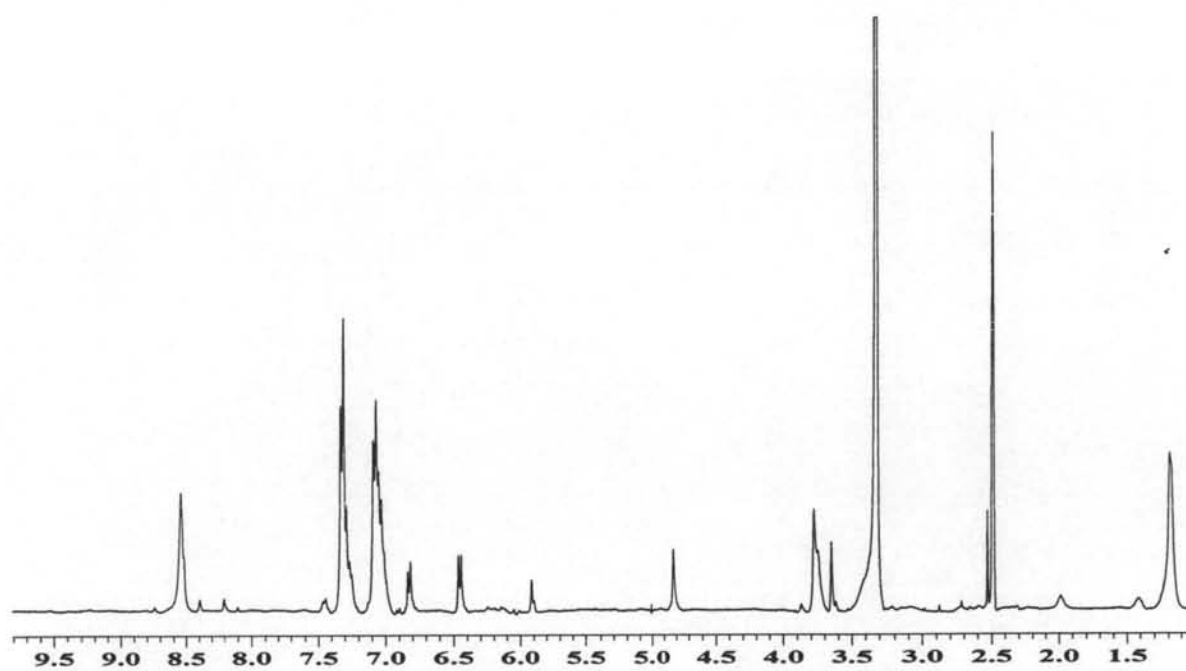


Figure A.6 ^1H NMR spectrum of ZnL-MDI-MTDA (1:3:1) in $\text{DMSO-}d_6$

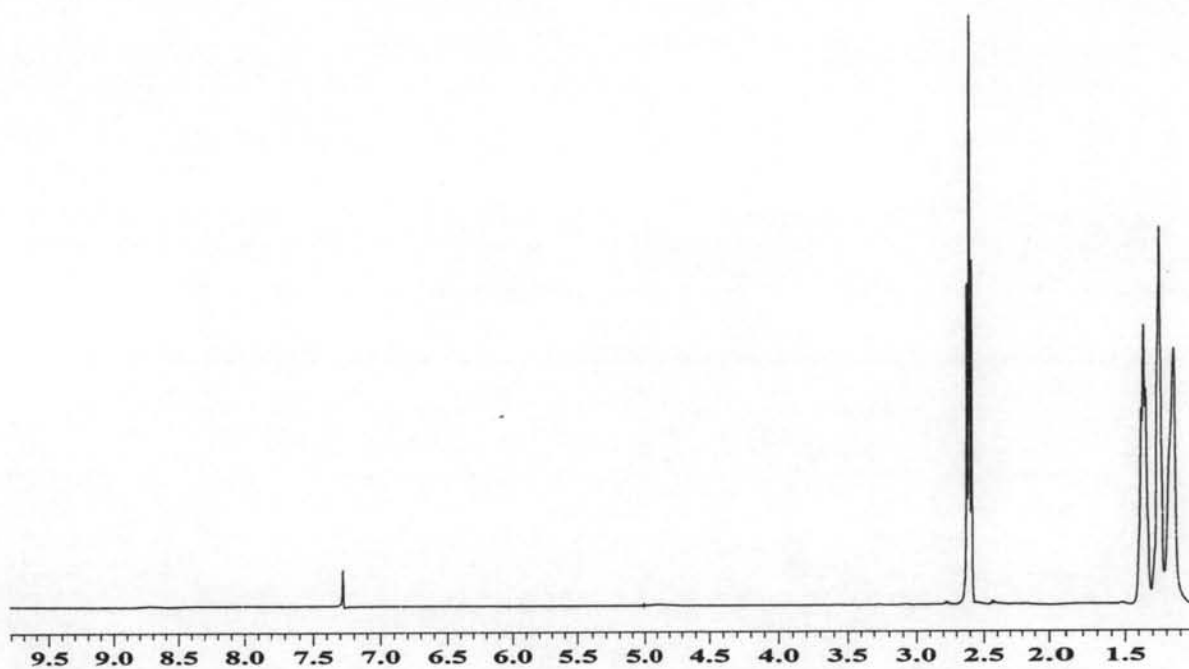


Figure A.7 ¹H NMR spectrum of HMDA in DMSO-*d*₆ + CDCl₃

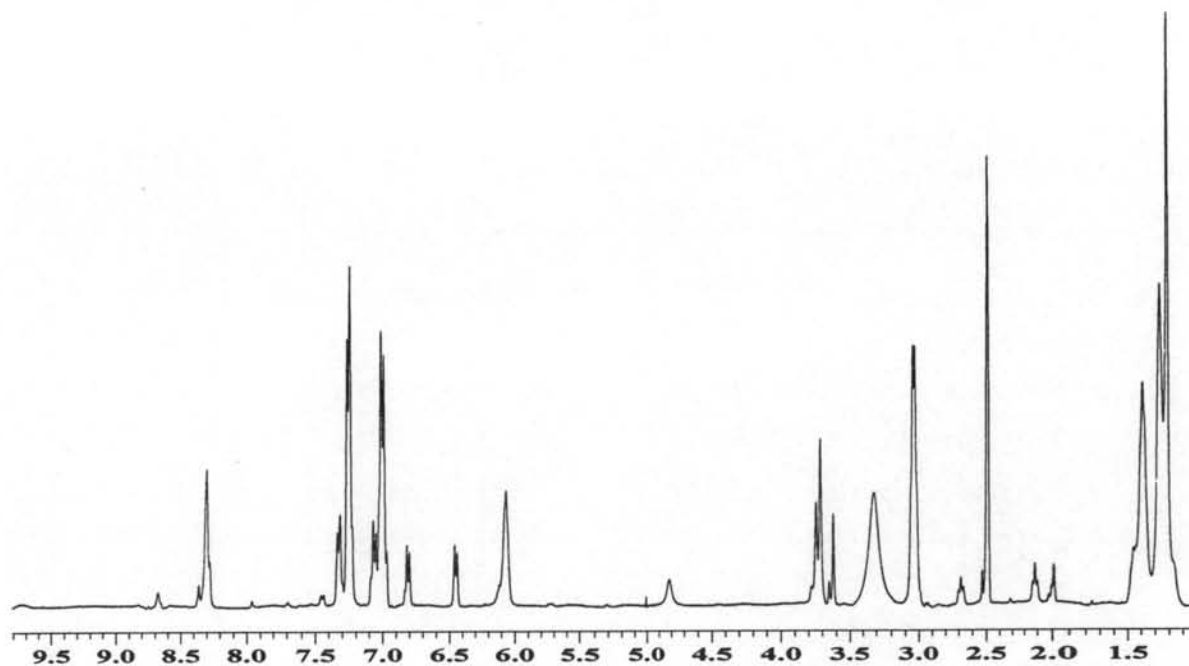


Figure A.8 ¹H NMR spectrum of MDI-HMDA (1:1) in DMSO-*d*₆

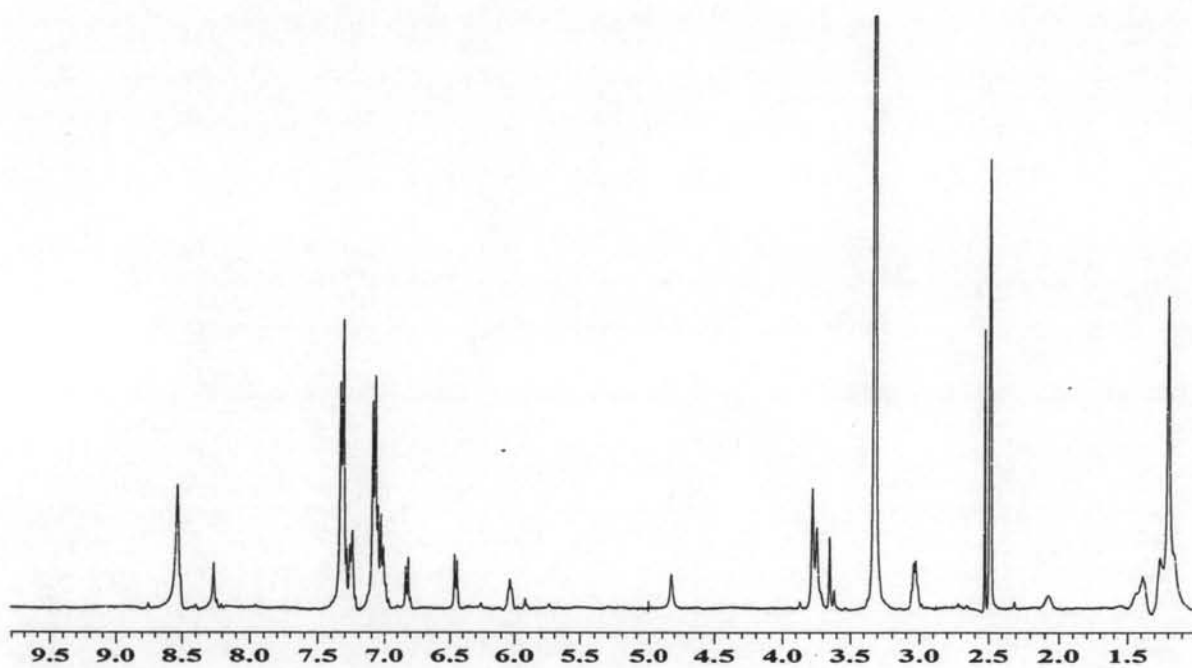


Figure A.9 ^1H NMR spectrum of ZnL-MDI-HMDA (1:3:1) in $\text{DMSO-}d_6$

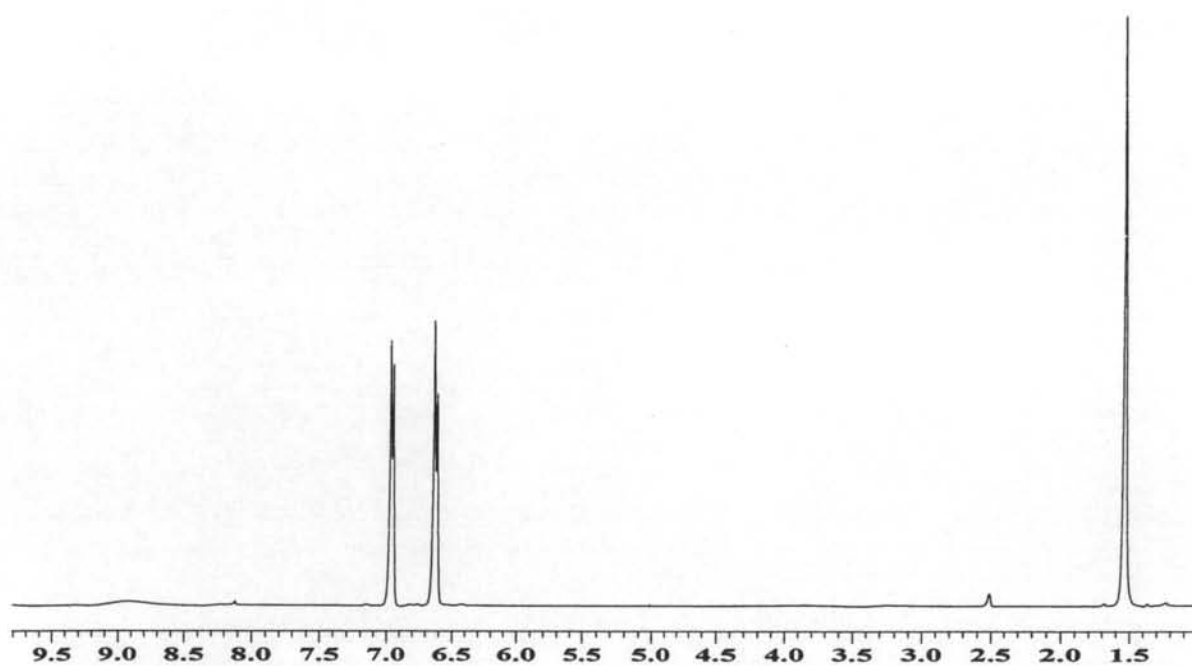


Figure A.10 ^1H NMR spectrum of BA in $\text{DMSO-}d_6 + \text{CDCl}_3$

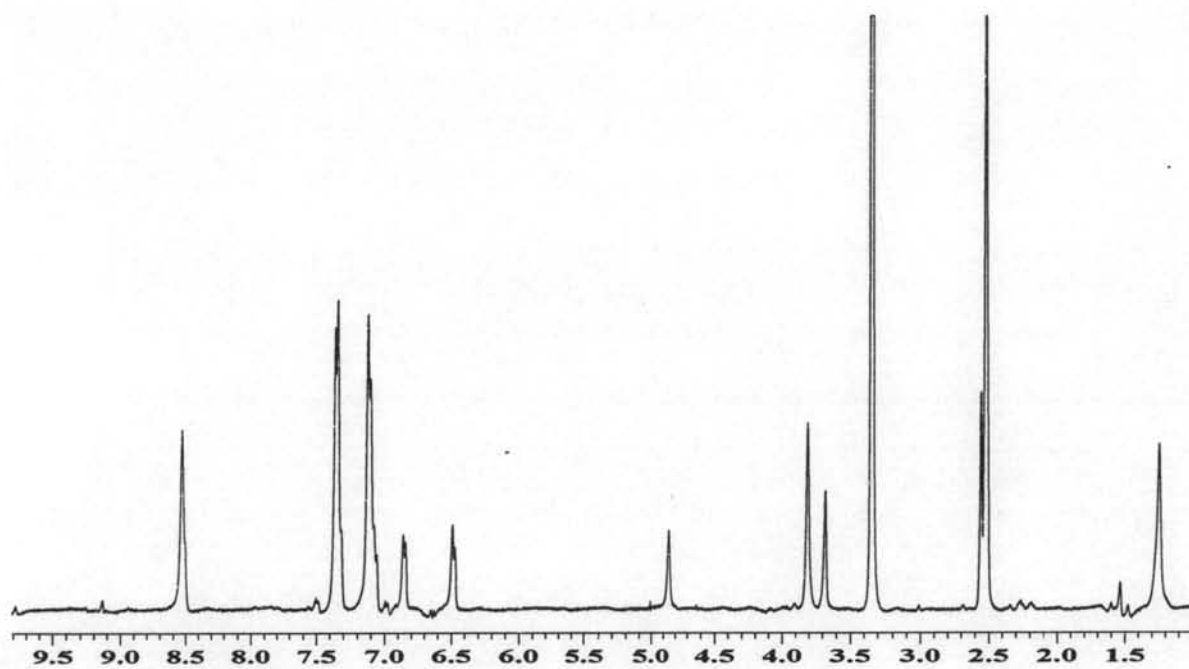


Figure A.11 ^1H NMR spectrum of MDI-BA (1:1) in $\text{DMSO-}d_6$

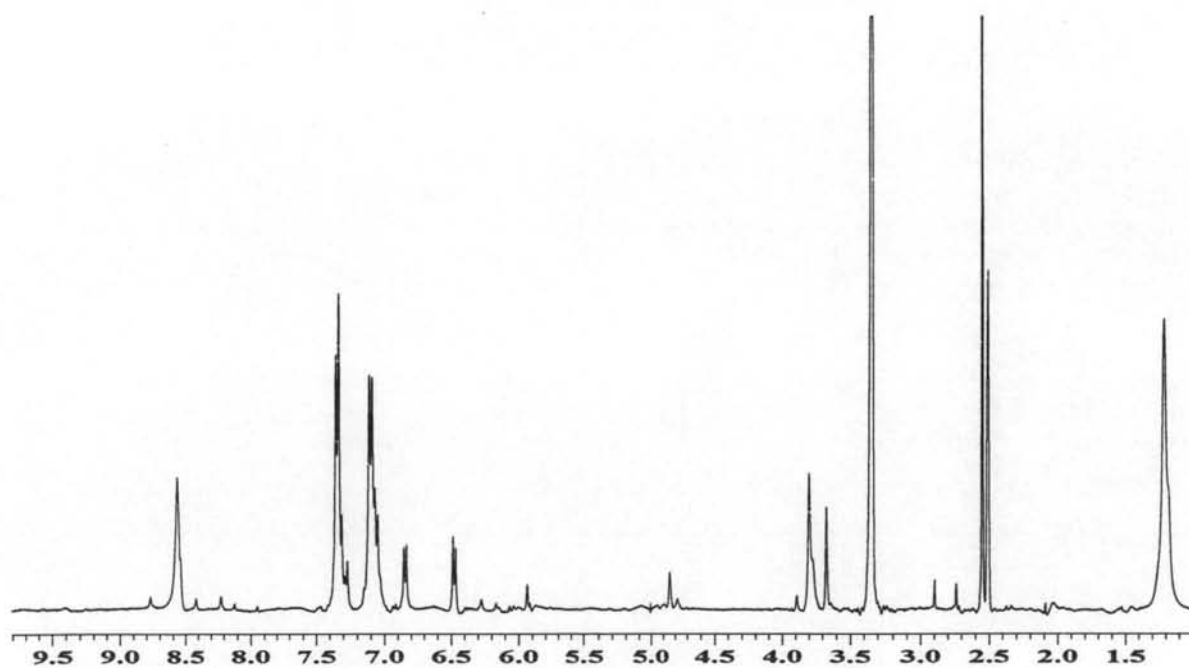


Figure A.12 ^1H NMR spectrum of ZnL-MDI-BA (1:3:1) in $\text{DMSO-}d_6$

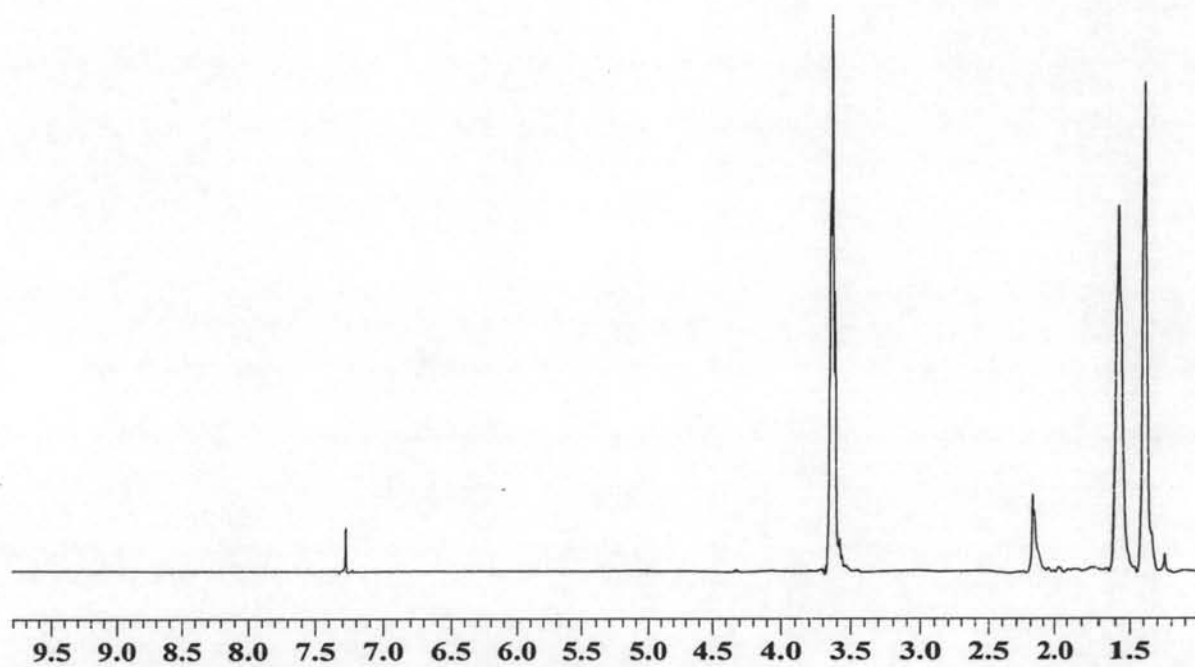


Figure A.13 ^1H NMR spectrum of HD in $\text{DMSO-}d_6 + \text{CDCl}_3$

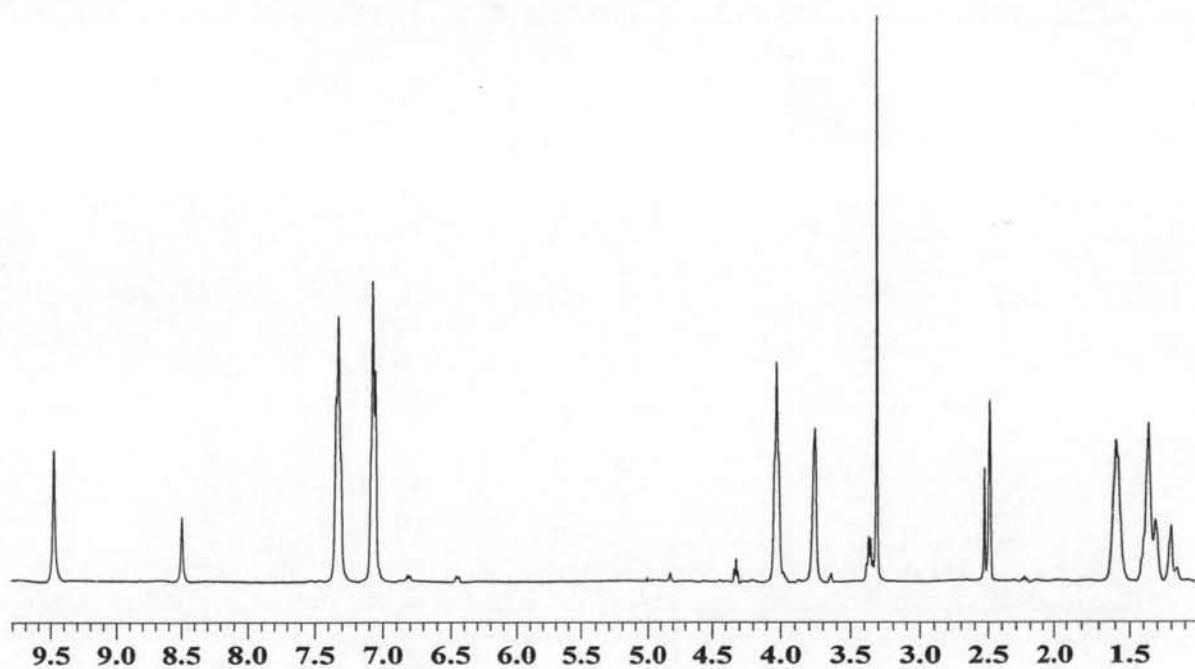


Figure A.14 ^1H NMR spectrum of MDI-HD (1:1) in $\text{DMSO-}d_6$

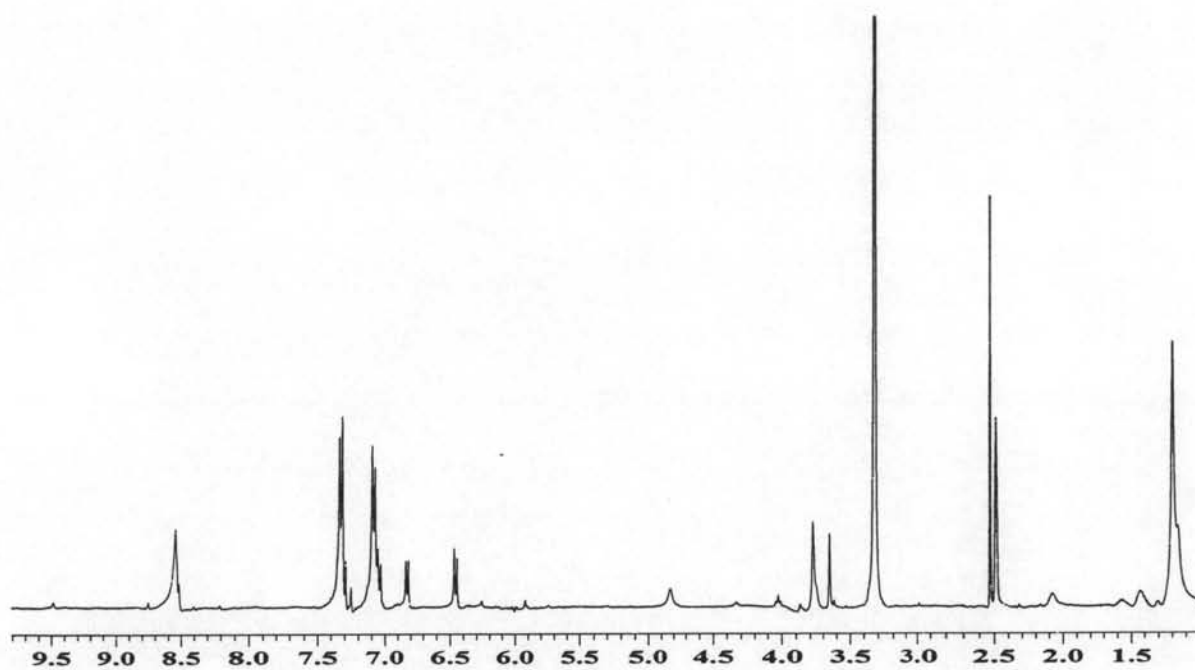


Figure A.15 ^1H NMR spectrum of ZnL-MDI-HD (1:3:1) in $\text{DMSO-}d_6$

APPENDIX B

B-1 DETERMINATION OF INHERENT VISCOSITY

Inherent viscosity [η_{inh}] ASTM D2270: Inherent viscosity is calculated from the dilute solution (1% or less) relative viscosity of the polymer. The inherent viscosity is calculated as:

The relative viscosity is given by:

$$\eta_{rel} = \frac{\text{solution flow time (t), sec}}{\text{solvent flow time (t}_0\text{), sec}}$$

The inherent viscosity is calculated as:

$$\eta_{inh} = \frac{\ln \eta_{rel}}{C}$$

where

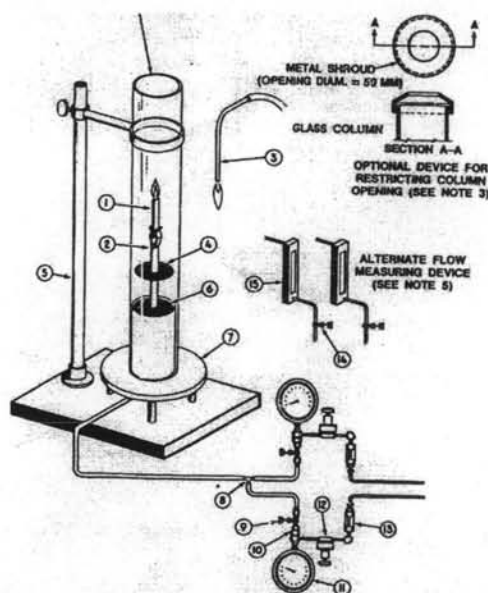
- C = concentration of the polymer in grams per 100 ml of solvent; usually, C = 0.5 g/100 mL
- $\ln \eta_{rel}$ = natural logarithm of the relative viscosity of the dilute polymer solution
- K = 0.01431, $t_0 = 98.97$ sec, $Kt_0 = 1.4163$ sec

Relative viscosity can be taken as the ratio of the flow times of a polymer solution and the pure solvent in the same viscometer and at the same temperature. Relative viscosity values generally are used for calculating the intrinsic or inherent viscosity of a polymer. The solvent to be used will depend on the polymer solubility. In general, the solvent should completely dissolve the sample in less than 30 minutes. It is desirable that the polymer be dissolved at room temperature although, heating is permissible if no degradation occurs. Select the viscometer through which the solvent will flow in not less than 100 seconds and not more than 200 seconds.

B-2 Limiting Oxygen Index (LOI)

ASTM D2863-70: the minimum concentration of oxygen, expressed as volume percent, in a mixture of oxygen and nitrogen that will just support flaming combustion of a material initially at room temperature. The LOI method used for self-supporting samples has been modified as described below to accommodate the viscous of the powdery samples. The measurement was carried out as follows. About 1 g. of the polymer sample was placed in a glass cup (diameter 20 mm, height 10 mm) fitted to the specimen holder. An external flame of 20 mm length was maintained in contact, for 10 s, with the polymer. The LOI value was taken as the minimum percentages of oxygen required in a nitrogen-oxygen atmosphere, surrounding the sample, to maintain its combustion for at least 30 s after ignition. The LOI value was taken as the average of five experiments each.

Apparatus



- | | | |
|----------------------------|-------------------------|---------------------------------|
| 11. Burning Specimen | 6. Glass Beads in a Bed | 1. Pressure Gage |
| 12. Clamp with Rod Support | 7. Brass Base | 2. Precision Pressure Regulator |
| 13. Igniter | 8. Tee | 3. Filter |
| 14. Wire Screen | 9. Cut-off valve | 4. Needle Valve |
| 15. Ring Stand | 10. Orifice in Holder | 5. Rotameter |

Figure A.1 LOI apparatus

Procedure

1. Calibrate the flow-measuring system using a water-sealed rotalin drummeter in accordance with Method D 1071.
2. The test shall be conducted at room temperature condition in accordance with Practice D 618.
3. Clamp the specimen vertically in the approximate center of the column.
4. Select the desired initial concentration of oxygen. If the specimen burns rapidly, start at a concentration of about 18 %.
5. Set the flow valves so that desired initial concentration of oxygen is flowing through the column.
6. Allow the gas to flow for 30 s to purge the system.
7. Ignite the entire top of the specimen with the ignition flame so that the specimen is well lighted. Remove the ignition flame and start the timer.
8. Do not adjust the oxygen concentration after igniting the specimen.
9. The concentration of oxygen must be raised if the flaming of the specimen extinguishes before meeting.
10. Adjust the oxygen concentration, insert a new specimen.

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