

CHAPTER II

EXPERIMENTAL SECTION

2.1 Materials

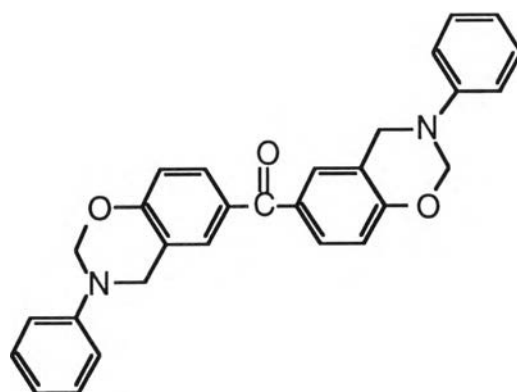
Paraformaldehyde, aniline (99.5%), methylamine (40% in water) and bisphenol-A (polycarbonate grade) were purchased from Aldrich Chemical Company. The 4,4'-dihydroxybenzophenone was kindly supplied by Ken Seika Corporation. All chemicals were used without further purification.

2.2 Instrument

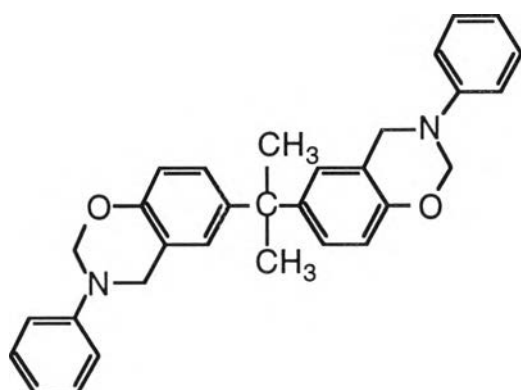
Infrared spectra were taken on Fourier transform infrared spectrometer, Biorad FTS 60A, equipped with high sensitivity, liquid nitrogen cooling system. The detector is mercury-cadmium-telluride (MCT) with specific detectivity $1 \times 10^{10} \text{ cm.Hz}^{1/2} \cdot \text{W}^{-1}$. The resolution was chosen to be 4 cm^{-1} for 200 scans.

2.3 Benzoxazine Synthesis and Sample Preparation

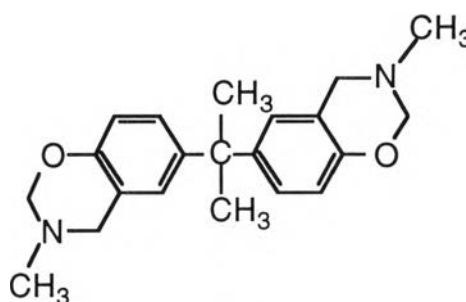
The 6,6'-bis (2,3-dihydro-3-phenyl-4H-1,3-benzoxazinyl) ketone, abbreviated as 44O-a, bisphenol-A and aniline based benzoxazine (B-a) and bisphenol-A and methylamine-based benzoxazine (B-m) were synthesized and the chemical structures of these three benzoxazine resins are depicted below. A detailed description of the synthesis procedures and molecular characterization has been presented elsewhere [Ning and Ishida, 1994].



440-a



B-a



B-m

The as-synthesized 440-a was dissolved in tetrahydrofuran and cast on a KBr plate. The sample was then cured at 290° C in air for 4 h. B-a, B-m, and the traditional phenolic resin were each melted, cast on a KBr plate, and then cured at 180° C for 3, 2, and 4 h, respectively. All cured samples were cooled in the oven and kept in a dessicator until used.

2.4 Hydrogen Bonding as a Function of Temperature

The sample was placed in a variable temperature infrared hot cell (Connecticut Instrument). A temperature controller was used with a thermocouple in intimate contact with the KBr disc containing the sample. The temperature accuracy of the controller was $\pm 0.1^\circ$ C. The infrared spectra

were obtained using Fourier transform infrared spectrophotometer (Biorad FTS 60A) equipped with a high sensitivity, liquid nitrogen cooling system, mercury-cadmium-telluride (MCT) detector with a frequency range from 4000 to 650 cm^{-1} . The specific detectivity, D^* , of the linearized MCT detector was $1 \times 10^{10} \text{ cm.Hz}^{1/2}.\text{W}^{-1}$. The resolution was chosen to be 4 cm^{-1} for 200 scans. The references were obtained by using empty KBr plates at the same temperature as the sample. The spectra were collected at 20° C increments with 15 min. to reach a constant temperature. The spectra were recorded over the temperature range from 50° C up to the temperature at which the absorbance of the C-H band fell below 95% of the original band in integrated intensity. After subsequent cooling, spectra were taken at room temperature in order to study the recovery of hydrogen bonding and observe possible degradation of the samples.