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APPENDIX

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## APPENDIX

The isocyanate compounds react readily with primary and secondary amines to yield the corresponding substituted ureas. The equation for the reaction with primary amines is as follows :



**Scheme A-1** The reaction between isocyanate compound and primary amine

In the prepolymer step of PU<sub>2</sub>NiL<sub>1</sub>, the extent of the reaction was determined by titration of unreacted NCO groups by the butylamine method . The reagent was *n*-butylamine with purified dioxane, standard hydrochloric acid and methyl red indicator. To purify the dioxane allow to stand over hydroxide pellets change the pellets each day until they no longer become brown.

### A-1 Titration procedure

Hexamethylene diisocyanate (0.267 ml, 1.65 mmol) was added dropwise to the solution of 1,6-hexane diol (0.177 g, 1.5 mmol). The mixture was refluxed under N<sub>2</sub> atmosphere and stirred for 8 hours. The reagent (*n*-butylamine reagent) 20 ml was added to the mixture and allow it to stand at room temperature for 15 minutes. Rinse the condenser with water. Titrate the contents of the flask with 0.1 N acid, using methyl red as an indicator.

### A-2 Calculation

Milliliters of acid for blank minus milliliters acid for sample = A

$$\frac{A \times N \text{H}_2\text{SO}_4 \times \text{mol. wt. of compound} \times 100}{\text{Grams of sample} \times 1000} = \% \text{compound}$$

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