Among the boron-nitrogen compounds tested as solid hydrogen storage materials, ammonia-borane has received increasing attention owing to its impressive gravimetric hydrogen content of 19.6% wt (6.5 and 13.1% wt for the first and second equivalents of H₂, respectively), stability in the solid state under ambient conditions, and nontoxicity [, ]. In this study, oleylamine stabilized Ni(0) nanoparticles were in situ prepared from the reduction of nickel(II) acetylacetonate by ammonia-borane in THF under inert gas atmosphere at room. Oleylamine stabilized Ni(0) nanoparticles were reproducibly isolated from the reaction solution by filtration and characterized by using various spectroscopic techniques. These nanoparticles were found to be highly active catalyst in the dehydrogenation of ammonia-borane providing a release of 2.0 equivalent H₂ per mole of ammonia-borane and an initial turnover frequency of 251 (mol H₂)(mol Ni)⁻¹(h)⁻¹ at °0.1 ± 25.0C. They preserve %79 of their initial catalytic activity even after the fifth run of dehydrogenation of ammonia-borane with the complete conversion of cyclopolyborazane to polyborazylene derivatives and plus 2 equivalent of H₂ at room temperature [ ]. Moreover, the work reported here includes a wealthy collection of kinetic data to determine the rate law and apparent activation energy for the catalytic dehydrogenation of ammonia-borane (Eaapp = 32 + 2 kJ mol⁻¹).

**Keywords:** Ammonia-borane, dehydrogenation, nickel, oleylamine.