

## A New Method of Catalytic Hydrogenation<sup>(1)</sup>

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The author with co-workers has found that precipitated nickel and copper are active catalysts for hydrogenation in the presence of caustic alkali or when treated with aqueous caustic alkali before use in hydrogenation. In the hydrogenation in aqueous caustic alkali, metallic aluminum or zinc can be used as well as hydrogen gas. In this preliminary note some examples are presented with no detailed

accounts,

Metallic nickel is precipitated by adding zinc dust to an aqueous solution of nickel chloride heated on a water bath until the supernatant liquid becomes colorless. It is washed with water by decantation, digested with 10 percent aqueous sodium hydroxide or potassium hydroxide on a water bath for one hour or two, and again well washed with water. The nickel catalyst thus prepared is shaken with hydrogen for two hours in the hydrogenating medium, aqueous caustic alkali, alcohol, or alcoholic caustic alkali, and then is ready for use as the catalyst in the hydrogenation of organic compounds by shaking with hydrogen at the ordinary temperature and pressure. Thus, estrone is reduced to estradiol either in aqueous caustic potash or in neutral alcoholic solution. Cholestenone is reduced to cholestanol in alcoholic solution, neutral or alkaline. Cinnamic acid is reduced to hydrocinnamic acid in alkaline aqueous solution. Nitrobenzene is reduced to aniline in alkaline alcoholic solution.

The time needed for hydrogenation naturally depends on the amount of nickel, and the use of a large amount seems desirable especially in the hydrogenation in the neutral medium. The metallic nickel precipitated from the chloride solution contains adsorbed chloride ions and the preparatory treatment with caustic alkali seems advantageous even in the hydrogenation in the alkaline medium, because the digestion with caustic alkali desorbs much of the adsorbed chloride. The nickel treated with caustic alkali retains a considerable quantity of adsorbed alkali even though washed several times with hot water, the washings keeping persistently alkaline to phenolphthalein, while it loses the retained alkali promptly if digested with a sodium chloride solution. The adsorbed alkali probably plays an important rôle in the hydrogenation, because the nickel not treated with caustic alkali, and the nickel treated with caustic alkali and then washed with acetic acid, are totally inactive. In conclusion, it may be added that the author has come to understand why Raney nickel is an active catalyst.

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