Vapor Phase Catalytic Hydrogenation of Organic Compounds Using Stabilized Nickel Catalyst[1]

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It has been known for a long time that reduced nickel catalyst prepared by thermal decomposition of nickel formate is effective for hydrogenation of many organic substances. "Stabilized Nickel Catalyst" (a kind of catalyst obtained from nickel formate) has been developed and can be prepared more conveniently for practical use, and it has been reported to show excellent quality with regard to activities and stability[1].

However, investigations in catalytic hydrogenation of organic substances using the nickel catalyst mentioned above were carried out exclusively in liquid phase, and none in vapor phase has been reported. In this paper, a new study applying this catalyst to vapor-phase hydrogenation is introduced.

As a result of experiments in which some typical organic compounds were hydrogenated, it was found that hydrogenation which this catalyst was able to be carried out in vapor phase as well as in liquid phase, and the preparation of this catalyst is simpler than that of any other nickel catalyst.

Experimental Method

The hydrogenation was carried out according to the Sabatier's method[2]. The catalyst was put into a hard-glass tube and was heated up to reaction temperature with an electric furnace, while dry hydrogen was passed through the tube. After a small amount of water from the catalyst was expelled by heating, the vaporized sample together with a large excess of hydrogen was introduced into the reaction tube. The reaction products flowing out of the tube were collected into the receiver through a condenser, and were isolated by suitable methods especially by means of fractional distillation. Then each product was identified by preparing an appropriate derivative respectively.

Results

Representative results of the experiments are summarized in Table I. Styrene and acetophenone were reduced to ethylbenzene each in high yield. Benzene was completely reduced to cyclohexane, phenol to cyclohexanol with some cyclohexanone, and benzaldehyde to benzene and toluene. Nitrobenzene was mostly reduced to aniline, however, a small amount of diphenylamine and white crystals (m. p. 193~197°C) were obtained as secondary products. Benzonitrile was reduced to toluene in good yield at about 250°C, and to benzene and toluene at about 350°C[3].

From these experiments, it is proved that the effectiveness of this nickel catalyst is as good as Sabatier's reduced nickel catalyst, and that the reaction procedure is quite simplified because of the stability of the catalyst.

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[1] T. Yamanaka and Y. Takagi, *J. Sci. Res. Inst. Tokyo*, **51**, 168 (1957).

[2] P. Sabatier, "La Catalyse en Chimie Organique", Ch. Béranger (1913).

[3] M. Tanaka, K. Watanabe and K. Hata, *J. Chem. Soc. Japan, Pure Chem. Sec.*, (*Nippon Kagaku Zassi*), **76**, 1392 (1955).

TABLE I

VAPOR PHASE CATALYTIC HYDROGENATION OF ORGANIC COMPOUNDS USING STABILIZED NICKEL CATALYST

Catalyst: a) contains 28% Ni; b) contains 40% Ni, the remainder is kieselguhr

Compound	Wt. of sample (g.)	Wt. of catalyst (g.)	Temp. (°C)	Time (hr.)	Flow velocity of sample (g./min.)	Vol. of H_2 passed (l.)	Wt. of products (g.)	Products and yields (%)
Styrene	5	20[a)]	203	$1\frac{3}{4}$	0.077	12.7	5	Ethylbenzene (98)
Benzene	5	20[b)]	182	$1\frac{1}{2}$	0.073	12.1	4.1	Cyclohexane (77)
Phenol	5	20[a)]	180	2	0.063	16.0	5	Cyclohexanol (77) Cyclohexanone (7)
Benzaldehyde	5	20[b)]	230	$1\frac{1}{6}$	0.122	9.0	3.8	Benzene (25) Toluene (40)
Acetophenone	5	20[a)]	203	2	0.079	14.5	4.95	Ethylbenzene (93)
Nitrobenzene	5	20[a)]	230	$1\frac{1}{2}$	0.091	10.2	4.85	Aniline (66)
Benzonitrile	5	20[a)]	250	2	0.089	11.3	3.3	Toluene (72)
Benzonitrile	6	20[a)]	354	2	0.094	12.6	3.1	Benzene (43) Toluene (18)

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