

NEW METAL CATALYSTS FOR ORGANIC REACTIONS

By

L. MÉSZÁROS and ZS. SZIJGYÁRTÓ

Institute of Applied Chemistry, József Attila University, Szeged

(Received May 2, 1969)

Pyrophoric lead, prepared by the vapour-phase heterogeneous reaction of lead oxide with furfural [1], has been successfully applied as an agent for coupling organic halides in Wurtz-like syntheses at temperatures between 20° and 100° [2]. According to a recent process, the metal powder was prepared by pneumatic atomization [3]. Another method consists in the application of a current density of about 10 A/mm² to a point of molten lead in an electromagnetic field, resulting in the formation of a metal powder of very small particle size [4]. If an inert gas atmosphere is used, the product contains no oxides, and it is as pure as the lump of metal used in its preparation. This oxide-free, highly pyrophoric metal powder, especially if the particle size is small (below 2—3 μ), is a useful agent to effect Wurtz-like syntheses, and the production of the metal powder can be counted continuously together with the organic reaction. Suitable selection of the parameters of this electromagnetic atomization process allows the adjustment of a desired particle size, as shown by experiments checked by means of electronmicroscopic measurements [5].

This method has now been extended to prepare pure nickel catalysts for hydrogenation reactions [6]. If aluminium and nickel electrodes are used in combination in the electromagnetic process, a Raney-type catalyst may be obtained with the particular advantage that the aluminium component is reactive enough to become converted into Al(OH)₃ on contact with water, and the use of an alkali hydroxide for its removal is unnecessary. In this way nickel catalysts completely free from any alkali metal contamination can be prepared. These catalysts have been found useful in the hydrogenation of the double and triple bonds of organic compounds.

As examples, vanillin was hydrogenated in ethyl acetate solution in the presence of a sample of the above catalyst (particle size 10 μ) to give vanillin alcohol in 75.6% yield. Cinnamic acid underwent hydrogenation under similar conditions (particle size 20 μ) in 1.5—3 hr to give cinnamic aldehyde in yields between 73 and 85%. The sodium-free Raney-type nickel catalyst effected the hydrogenation of butene-2-diol-1,4 within 2 hours, to give butene-2-diol-1,4 in 75% yield.

Another use for the electromagnetic metal atomization process tried so far has been the preparation of copper of small particle size (below 1 micron) and application of the product as catalyst in the dehydration of methanol and ethanol.

A further improvement consists in the sintering of the metal powders either

by pressure or by an electric resistance melting method. The sintered products, pills or platelets, retain their catalytic activity and ensure convenient handling in vapour or of liquid phase processes; further, they offer the possibility of application in fuel cells.

References

- [1] *Mészáros, L.*: Brit. P. 1. 045. 032 (1966); U.S.P. 3. 278. 296 (1966); Japan P. 37. 929 (1964); French P. 1. 390. 406 (1964); *Mészáros, L., S. A. Gilde*: Heterogen-katalytische Reaktoren zur Herstellung von pyrophoren Metallen und Furan in der Dampfphase, XXXVI^e Congrès International de Chimie Industrielle. Bruxelles, 1966. a) *Ind. Chim. Belge* 31, Special No. 16/25 (1966); b) *Ind. Chim. Belge* 32, Compte-rendu II, Special No. (1967); *Mészáros, L., S. A. Gilde*: *Acta Phys. et Chem. Szeged* 14, 127 (1968).
- [2] *Mészáros, L.*: *Tetrahedron Letters*, 4951; (1967) *Hung. P.* 152. 090 (1964).
- [3] *Mészáros, L.*: *Hung. P.* 153.189 (1963); *Mészáros, L., S. A. Gilde*: Laboratory for pneumatic pulverization, Type LK-004. Specification, 2 p.
- [4] *Mészáros, L.*: French P. 1. 480. 209 (1966).
- [5] *Mészáros, L.*: ME-773. Hungarian patent application (1966); Chemische und physikalische Operationen mit elektrizitätsleitenden, flüssigen Stoffen bei elektromagnetischer Bewegung, XXXVII^e Congrès International de Chimie Industrielle. Madrid, 1967; *Chim. Ind. Génie Chimique* 98, No. 19, 163 (1967).
- [6] *Horányi, G., F. Nagy*: *M. Kémiai Folyóirat* 70, 475 (1964).

НОВЫЙ КАТАЛИЗАТОР-ПЛЁНКА ДЛЯ ОРГАНИЧЕСКОГО АНАЛИЗА

Л. Месарош, Ж. Сийдьярто

Порошок сплава никеля-алюминия микронного размера, приготовлен в воде, переходит в Ni. Al. Al. (OH) , и способен для гидрирования. Этот Рэнэ-никелевый катализатор не содержит и следы гидроксида натрия.