PATENT SPECIFICATION 668,592

Date of Application and filing Complete Specification: March 10, 1950.

No. 6047/50.

Application made in Netherlands on March 31, 1949.

Complete Specification Published: March 19, 1952.

2466

Index at acceptance:—Class 1(i), F3b1.

COMPLETE SPECIFICATION

Process for Preparing Catalysts and the Catalysts so Prepared

We, NAAMLOOZE VENNOOTSCHAP DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, of 30 Carel van Bylandtlaan, The Hague, The Netherlands, a Company organised under the laws of The Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a process for preparing catalysts by impregnating a carrier material with two metal salts in solution and subsequently decomposing these salts.

In some cases a process of this type has the drawback that the two metal salts referred to above react in the solution with each other and form an insoluble compound. This not only involves the loss of valuable catalytic material but, apart from the possibility that the catalyst obtained may be less active, it also renders effective impregnation of the carrier difficult or impossible. Although it is possible to obviate the losses by carrying out the impregnation by using the various metal salt solutions one after the other, the impregnation itself will only be partially improved thereby.

From Fiat Final Report No. 968 it is known to prepare zinc-tungsten-silica gel impregnation catalysts with methylamine as a solubilising agent.

When studying the influence of methylamine it was found that the precipitate which is formed when a 10% aqueous zinc nitrate solution is added to a 4% aqueous ammonium tungstate solution, does not occur when methylamine is added to the tungstate solution. A further examination revealed that with other salt solutions, such as cobalt or nickel nitrate, the use of methylamine cannot avoid the formation of a precipitate.

It has been found that these difficulties can be overcome in preparing certain catalysts from ammonium tungstate or molybdate by using an alkanolamine as a solubilising agent to prevent the undesirable formation of an insoluble compound.

Thus, according to the present invention, a process for preparing catalysts by impregnating a carrier material with an aqueous solution containing a salt from each of two distinct groups, namely a first group consisting of ammonium tungstate and ammonium molybdate and a second group consisting of cobalt and nickel salts is characterised by forming the impregnating solution by mixing or contacting a salt of the first group with a salt of the second group in the presence of water and an alkanolamine.

Particularly suitable alkanolamines for the purposes of the present invention are monoethanolamine, di-ethanolamine and triethanolamine.

In carrying into effect the process of the present invention it is preferred to prepare first an aqueous solution of ammonium tungstate or ammonium molybdate in the presence of the alkanolamine and then to add the cobalt or nickel salt to this aqueous solution in order to mix or contact the salts with one another. This preference is based upon the observation that the alkanolamine increases the solubility of ammonium tungstate and ammonium molybdate in aqueous media. Thus it is possible to dissolve in a 50% aqueous alkanolamine solution at room temperature, for example 15% ammonium tungstate, whereas in water only a solution of approximately 4% concentration can be obtained at the same temperature. Hence, by operating in the preferred manner it is possible to obtain a higher concentration of catalyst on the carrier material.

An alternative way of carrying into effect the process of the present invention is to mix the appropriate salts with each other in the presence of an aqueous alkanolamine solution.

Suitable carrier materials for the catalyst are: silica gel, the German bleaching earth known as terrana, kaolin, magnesium silicate, magnesium carbonate, magnesium oxide, aluminium oxide prepared by precipitation and particularly so-called activated alumina, as well as other active aluminium oxides, such as those prepared by activating

bauxite (for example that known under the Trade Mark Porocel).

After the carrier has been impregnated with the salt solution it is freed from solvent (alkanolamine plus water) by heating, either in vacuo or not, and compressed into pieces, such as pills, tablets or strands of the desired hardness, whereupon the impregnated metal salts are decomposed. The catalyst can subsequently be reduced with gas, particularly hydrogen, at elevated temperature or be subjected to a sulphidising treatment, for example, with hydrogen sulphide.

The catalyst obtained can be used in various reactions such as hydrogenation, dehydrogenation, desulphurisation, hydroforming and reforming.

The following Example will further illustrate how the invention may be carried out in practice, but the invention is not restricted to this Example. The percentages are by weight.

Example

A 10% aqueous solution of nickel nitrate was added to a 15% solution of ammonium tungstate in a 50% aqueous monoethanolamine solution. No precipitate was formed, so that the solution could be used for impregnating an aluminium oxide carrier to prepare a catalyst.

Corresponding results were obtained by using cobalt nitrate instead of nickel nitrate, as well as when the tungstate solution was replaced by an ammonium molybdate solution.

What we claim is:—

1. A process for preparing catalysts by impregnating a carrier material with an aqueous solution containing a salt from each of two distinct groups, namely a first group consisting of ammonium tungstate and ammonium molybdate and a second group consisting of cobalt and nickel salts, characterised by forming the impregnating solution by mixing or contacting a salt of the first group with a salt of the second group in the presence of water and an alkanolamine.

2. A process as claimed in claim 1 in which a solution of a salt of the first group and an alkanolamine is first prepared and a salt of the second group is subsequently added either alone or in solution.

3. A process according to claim 1 or 2, in which mono-, di- or tri-ethanolamine is used as the alkanolamine.

4. A process for preparing catalysts substantially as described with reference to the foregoing Example.

5. Catalysts whenever prepared by the process claimed in any one of the preceding claims.

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Guildford: Printed for Her Majesty's Stationery Office, by Billing and Sons, Ltd.—1952.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2,
from which copies may be obtained.