

UNITED STATES PATENT OFFICE

1,987,660

PREPARATION OF CATALYST FOR THE
HYDROGENATION OF OILS

Owen G. Bennett and Joseph C. W. Frazer, Baltimore, Md.; said Bennett assignor to Catalyst Research Corporation, Baltimore, Md., a corporation of Maryland

No Drawing. Application March 16, 1931,
Serial No. 523,176

4 Claims. (Cl. 23—236)

This invention relates to the hydrogenation of oils, and it is among its objects to provide catalysts for the hydrogenation of oils which are readily prepared in a state of high activity, are free, or substantially free, from oxide and other activity-reducing impurities, possess great hydrogen adsorptive power, are efficient and of long life.

The invention is predicated upon our discovery that particularly satisfactory hydrogenation catalysts may be obtained by alloying or amalgamating nickel with mercury, removing the mercury from the alloy by vacuum distillation, and protecting the residual finely divided and highly active nickel from oxygen-containing gases and similar activity-reducing agents.

In the practice of the invention alloy containing nickel is first prepared as by amalgamating metallic nickel with mercury. Mercury does not readily wet and amalgamate with nickel, but this may be effected quickly and easily if hydrogen is first deposited electrolytically upon the nickel. For instance, an electric current is passed through a solution of sulfuric acid, sodium carbonate or the like, the nickel to be amalgamated acting as cathode. As thus treated the nickel may be amalgamated readily with mercury or solutions of mercury salts.

In the preferred practice of the invention the nickel amalgams are prepared directly by electro-deposition. In this embodiment a nickel salt solution is electrolyzed in a cell having a mercury cathode, the current and voltage being regulated to effect deposition of nickel. The more concentrated the amalgams, the higher are the yields. However, for ease of handling it is preferable to use relatively liquid amalgams.

When an alloy containing the desired amount of nickel has been prepared it is transferred without undue exposure to air, to any suitable distillation apparatus and the mercury is distilled from it under vacuum. The most satisfactory results are obtained by the use of very high vacuum for this results in lowering of the distillation temperature, which is favorable to the production of the most active material. The distillation container should have ample vapor space, because the amalgam usually swells considerably during the first stages of the distillation process. Normally a container having twice the cubic capacity of the amalgam being distilled will suffice.

When the mercury has been entirely removed, the nickel remains in the form of finely-divided particles of metallic nickel which are highly ac-

tive and whose surfaces are free metal, i. e., free from oxide. These characteristics of the nickel prepared by our process appear in various ways. For example, nickel ordinarily can not be wet directly by mercury. But the nickel produced by our process is immediately wet when contacted with mercury. This is indicative of the freedom of our product from oxide, and it shows that the surfaces of the particles consist chiefly, if not wholly, of free metal. The activity of our catalytic nickel is further manifested by the fact that it adsorbs five to six times more hydrogen than nickel prepared by the customary oxide reduction methods.

Furthermore, nickel prepared in this manner oxidizes rapidly when exposed to oxygen, and our tests have shown that it should be protected from oxygen-containing gases and other activity-reducing agents in order to preserve it in a state of maximum activity. This may be done by sealing the nickel in the container when distillation is complete. The container may then be opened under the oil to be hydrogenated. Preferably, however, our catalysts are protected by transferring them to an activity-protective fluid. For instance, the nickel may be transferred, without exposure to the atmosphere, to the oil which is to be hydrogenated. For most purposes, and for ease of adding definite concentrations of catalyst, a known weight of oil, sufficient to cover and protect the nickel, is used. After the nickel has been covered by the oil, the mixture is again weighed. Definite weights of the mixture may then be taken to add to the body of oil to be hydrogenated. Or, if preferred, it may be placed in a container filled with carbon dioxide gas.

Our catalysts may be substituted for those now used in any of the commercial hydrogenations procedures. Their efficacy is shown by tests which we have made. In one test 2.5 grams of nickel, prepared as described above, were suspended in 500 grams of cotton seed oil having an iodine number of 108.5 by the Wijs method. This mixture was then treated during three hours at a temperature of 180° C with hydrogen under 125 pounds pressure, while stirring continuously with a propeller tank stirrer rotated at 860 R. P. M. The hydrogenation efficiency of our catalyst is shown by the fact that after this treatment the iodine number was reduced to 44.

Other things being equal, the rate and degree of hydrogenation are dependent upon the concentration of nickel used. In a further test the amount of catalyst was doubled, the other conditions remaining the same. The hydrogenated

product of this test showed an iodine number of 19.7.

The hydrogenated products obtained in all of our tests were of satisfactory quality, being pure white, of pleasant odor and good taste. The hardness may be varied from a grease-like consistency to very hard products resembling waxes, depending upon the extent of hydrogenation. The nickel prepared by the process provided by this invention is exceedingly finely divided, which is desirable, as it aids the maintenance in suspension in the oil. However, it settles readily when agitation ceases. The settled sludge may be used to hydrogenate further batches.

A further advantage of our nickel catalysts resides in the ease with which they may be revived when their activity finally decreases in use, or if they become inadvertently oxidized. After being freed from oil the catalyst is reamalgamated and processed as described above. Both this procedure, and that of initial preparation, are simpler and less tedious than those previously available. Furthermore, by observing the simple precautions noted hereinabove catalysts of high activity are consistently obtained. That is not always so with the processes known previously.

The claims presented herein are drawn to nickel hydrogenation catalysts and method of preparing them. Claims directed to the preparation of supported metallic catalysts through the amalgamation method disclosed herein are embodied in our copending application Serial No. 523,175, filed concurrently herewith.

According to the provisions of the patent statutes, we have explained the principle of our invention and have described what we now consider to represent its best embodiment. However, we desire to have it understood that, within the scope of the appended claims, the invention may

be practiced otherwise than as specifically described.

We claim:

1. In a process of preparing a metallic catalyst for the hydrogenation of oils, the steps comprising vacuum distilling a nickel-mercury amalgam to remove the mercury therefrom and thereby forming metallic nickel in a finely divided form which is readily suspendible in oil and highly catalytically active in the hydrogenation of oil.

2. In a process of preparing a metallic catalyst for the hydrogenation of oils, the step comprising vacuum distilling a nickel-mercury amalgam to remove the mercury therefrom and thereby forming metallic nickel in a finely divided form which is readily suspendible in oil and highly catalytically active in the hydrogenation of oil.

3. A process of preparing a metallic catalyst for the hydrogenation of oils, comprising removing the mercury from a nickel-mercury amalgam by vacuum distillation and thereby forming metallic nickel in a highly catalytically active finely divided form which is readily suspendible in oil, and without unduly exposing the highly active finely divided metallic nickel residue thus obtained to oxygen-containing gases transferring it to an activity-protecting fluid.

4. A process of preparing a metallic catalyst for the hydrogenation of oils, comprising removing the mercury from a nickel-mercury amalgam by vacuum distillation and thereby forming metallic nickel in a highly catalytically active and finely divided form readily suspendible in oil, and mixing the highly active finely divided metallic nickel residue thus obtained with an oil without exposing it to oxygen-containing gases.

OWEN G. BENNETT.
JOSEPH C. W. FRAZER.