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# UNITED STATES PATENT OFFICE.

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## METHOD OF APPLYING NICKEL CARBONYL IN MANUFACTURING PROCESS.

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*To all whom it may concern:*

Be it known that I, RUDOLF LESSING, Ph. D., a subject of the King of Great Britain, residing at High Holborn, in the county of London, England, have invented certain new and useful Improvements in Methods of Applying Nickel Carbonyl in Manufacturing Processes; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

In certain manufacturing processes there is used a gas-containing vapor of nickel carbonyl,—for instance, in the well-known Mond process of obtaining nickel, and in my process for hydrogenating unsaturated organic compounds, as described in my United States Patent No. 1,162,623, dated November 30, 1915.

Hitherto, the gas containing the vapor has been that which was passed over finely-divided nickel for the purpose of making nickel carbonyl. In order to obtain a sufficiently high concentration of nickel carbonyl in the gas, it has always been considered necessary heretofore to use a gas containing carbon monoxid in considerable excess over that which actually combines with the nickel. This excess is undesirable either as reducing the partial pressure of some other gas which is essential to the process or as giving rise to secondary reactions. On the other hand, it is expedient for the ready formation of nickel carbonyl that a gas containing a large percentage of carbon monoxid be used.

I have found that both conditions (1) of carbon monoxid concentration in the nickel carbonyl formation, and (2) low carbon monoxid concentration in the use of the gas-carrying nickel carbonyl, can be complied with by using different gases in separate cycles for the two phases of such processes.

By my invention, the nickel carbonyl is absorbed from the gas or gas carrying it, by passing said gas or gases through or over an inert absorbent, and the elastic fluid which is to convey the carbonyl to the manufacturing process is afterward passed over or through the absorbent.

In this manner, it is possible to store nickel carbonyl, practically free from an excess of uncombined carbon monoxid, and to incorporate it subsequently with a suitable elastic fluid, in any desired ratio, for the manufacturing process intended. Thus, for instance, if the nickel carbonyl is to be used for the production of commercial nickel, I may pass hydrogen, nitrogen, carbon dioxid, or any mixture of these or other suitable gases over or through the absorbent carrying nickel carbonyl, and heat the mixture of gas and vapor for the decomposition of the nickel carbonyl in the known way. The advantage of this mode of working is that the nickel carbonyl concentration of the gas can be adjusted at will so as to deposit the metal in the desired physical condition, such as size or density. A further advantage is that the bulk of the iron carbonyl which is mostly present as an impurity and which is less volatile than nickel carbonyl may be left behind in the absorbent and that the decomposition takes place in the absence of an excess of carbon monoxid, which excess usually gives rise to the deposition of carbon in the nickel product.

If the nickel carbonyl is to be employed as a catalyst for the purpose of hydrogenating unsaturated organic compounds (as, for instance, for saturating fatty acids and their glycerides) the advantage is that the diluent effect of free or uncombined carbon monoxid, and any possible side reactions which might be brought about by such uncombined carbon monoxid, are excluded, and that the amount of nickel carbonyl in the reacting mixture can be carefully adjusted independently of the formation of nickel carbonyl.

In carrying out my invention, I pass a gas containing carbon monoxid over finely-divided nickel or a mixture of nickel with other metal, such as reduced nickel (copper matte) in the known way; the time of contact being dependent on the percentage of carbon monoxid in the gas and the concentration of nickel carbonyl required.

The mixture produced is then passed through a scrubber which may be charged with inert porous material in pieces of shape and size suitable for affording a large surface and exerting little back pressure, said material being impregnated with an absorbent for nickel carbonyl, such as an oil. The inert material may consist of broken brick of high porosity, coke, charcoal or the like. If charcoal and particularly "activated" charcoal is used the im-

pregnation may be dispensed with, as I have found this charcoal itself to be an excellent absorbing agent for nickel carbonyl.

Other porous materials, such as alumina, fuller's earth, kieselguhr, which are known to absorb vapors readily, may be used. I prefer to arrange two or more absorbers in series, so that the first can be completely saturated, whilst those that follow act as check vessels for retaining the last traces of nickel carbonyl, which, however, may be left in the gas to be returned to the volatilizer. The gas stripped of nickel carbonyl is returned to the nickel volatilizer, with or without a further addition of some gas rich in carbon monoxid. If no gas is added to take the place of the carbon monoxid removed in form of nickel carbonyl, the carbon monoxid of the circulating gas is gradually diminished, and, in this manner, the carbon monoxid may be substantially or almost completely removed from gas mixtures such as water gas, leaving hydrogen of high concentration behind.

When one of the absorbers is saturated, it is cut off from the carbon monoxid cycle by the operation of suitable valves, and the carrying gas or the gas for reaction is passed through the absorber to pick up nickel carbonyl in form of vapor. The amount of nickel carbonyl in the reaction gas can be regulated by passing either the whole or only a portion of this gas through the absorber or by the application of heat or cooling to the absorber by suitable means.

When making commercial nickel, the carrying gas preferably consists of hydrogen or a gas rich in hydrogen, nitrogen, or any mixture of inert gases not giving rise to a deposition of impurities in the nickel.

When using the nickel carbonyl for hydrogenation purposes, hydrogen or a gas rich in hydrogen is used.

Having thus described my invention, what I claim is:

1. The method of applying nickel carbonyl in manufacturing processes, which comprises first storing the nickel carbonyl by passing gas or gases carrying it in contact with an absorbent for nickel carbonyl, and then taking up and conveying the stored nickel carbonyl to the manufacturing process by passing a suitable elastic fluid in contact with the absorbent; substantially as described.

2. The method of storing nickel carbonyl for subsequent use in manufacturing processes, which comprises passing a gas containing carbon monoxid into contact with nickel, and then passing the mixture of gas and vapor through an absorbent for nickel carbonyl, said absorbent being practically inert to carbon monoxid, substantially as described.

3. The method of storing nickel carbonyl for subsequent use in manufacturing processes, which comprises passing a gas containing carbon monoxid and hydrogen into contact with nickel, and then passing the mixture of gas and vapor through an absorbent for nickel carbonyl, said absorbent being practically inert to carbon monoxid and hydrogen.

4. The method of applying nickel carbonyl in manufacturing processes, which comprises first storing the nickel carbonyl in an absorbent and then taking up and conveying the stored nickel carbonyl to the manufacturing process by passing a suitable elastic fluid into contact therewith; substantially as described.

5. The method of applying nickel carbonyl in hydrogenation processes, which comprises first storing the nickel carbonyl in an absorbent, and then taking up and conveying the stored nickel carbonyl to the hydrogenation process by passing a gas rich in hydrogen into contact therewith; substantially as described.

6. The method of applying nickel carbonyl in manufacturing processes, which comprises passing a suitable elastic fluid into contact with a body of nickel carbonyl stored in an absorbent and conveying the resulting mixture to the manufacturing process; substantially as described.

7. In a process of hydrogenating organic compounds with aid of nickel carbonyl, the method of avoiding excess of carbon monoxide in the hydrogenator, which consists in separating the nickel carbonyl from the volatilizer gases by means of a suitable absorbent before introducing the nickel carbonyl into the hydrogenator.

8. In a process of hydrogenating organic compounds with nickel carbonyl, absorbing the nickel carbonyl in a suitable absorber and subsequently removing the nickel carbonyl from the absorbent and conveying it to the hydrogenator by means of the hydrogenating gas.

9. The method of hydrogenating organic compounds with the aid of nickel carbonyl and with avoidance of excess of carbon monoxide in the hydrogenator, which comprises separating the nickel carbonyl from the carbon monoxide by means of an absorbent and supplying the separated nickel carbonyl, in admixture with hydrogen and substantially free from carbon monoxide, to the hydrogenator.

In testimony whereof I affix my signature.

RUDOLF LESSING.