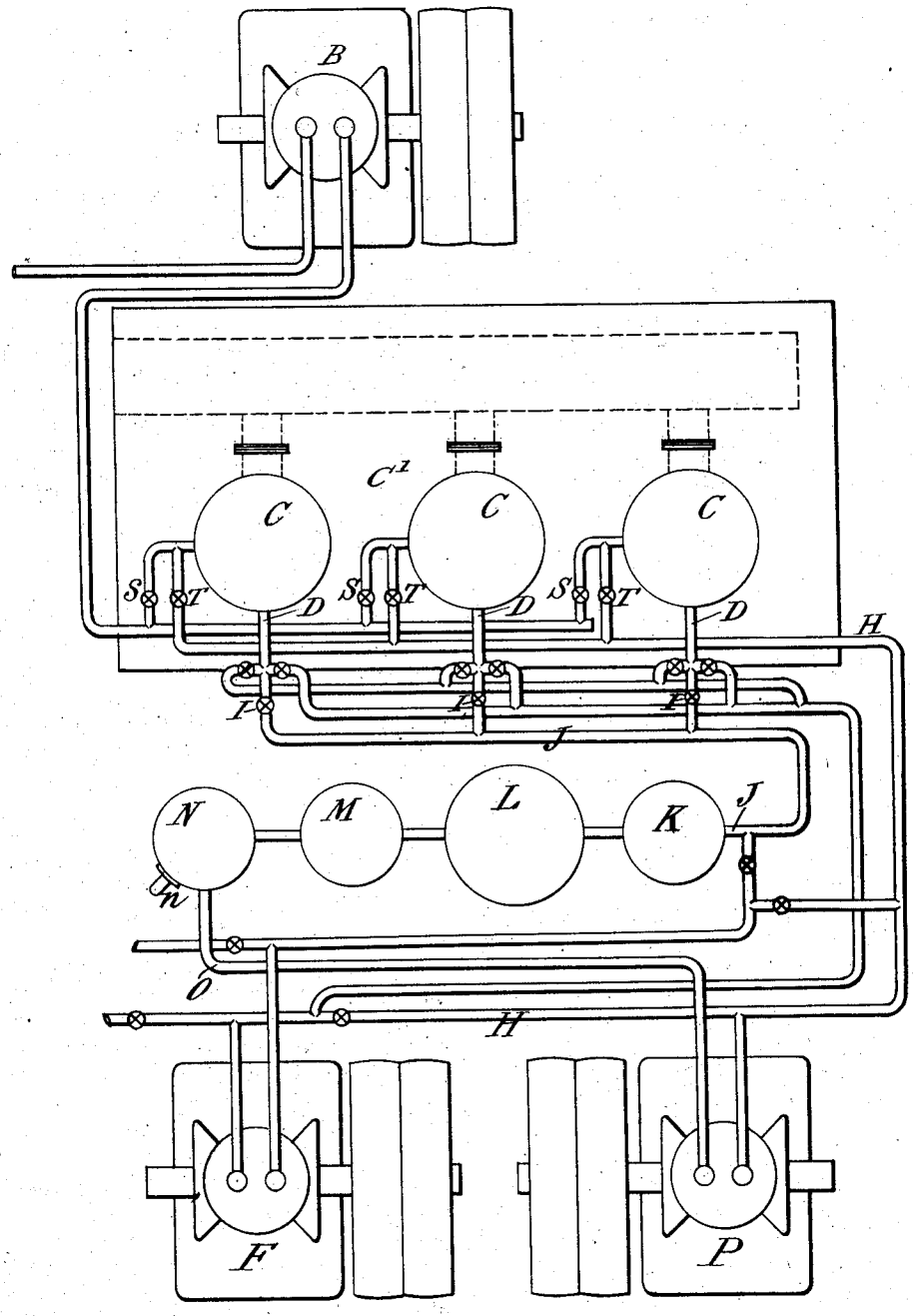
No. 760,852. PATENTED MAY 24, 1904.

J. DEWAR.

MANUFACTURE OF NICKEL CARBONYL.

APPLICATION FILED NOV. 21, 1902.

NO MODEL.




Witnesses:
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THE NORRIS PETERS CO., PHOTO-LITHO., WASHINGTON, D. C.

No. 760,852. Patented May 24, 1904.

# UNITED STATES PATENT OFFICE.

JAMES DEWAR, OF CAMBRIDGE, ENGLAND.

## MANUFACTURE OF NICKEL CARBONYL.

SPECIFICATION forming part of Letters Patent No. 760,852, dated May 24, 1904.

Application filed November 21, 1902. Serial No. 132,320. (No specimens.)

*To all whom it may concern:*

Be it known that I, JAMES DEWAR, a citizen of England, residing at No. 1 Scroop Terrace, Cambridge, in the county of Cambridge, England, have invented a certain new and useful Improvement in the Manufacture of Nickel Carbonyl, (for which I have applied for a patent in Great Britain, dated April 22, 1902, No. 9,300,) of which the following is a specification.

In Mond's British specification, No. 12,626 of 1890, is described a process for recovering nickel from its ores by calcining the ore, so as to get into the condition of an oxid, reducing the oxid by subjecting it to the action of reducing-gases, treating with carbonic oxid the metal obtained by the reduction, and thereby producing a compound vapor mixed with the excess of carbonic oxid called in the specification "nickel carbon oxid," but now generally known as "nickel carbonyl." This substance can be separated as a liquid, which on being heated vaporizes and becomes decomposed, the carbonic oxid being liberated and pure nickel being deposited as coherent metal.

The nickel carbonyl vapor at ordinary pressures is very unstable, its components becoming readily dissociated with explosion on moderate elevation of temperature, and therefore its production has hitherto been carried on at a comparatively low temperature, such as 50° centigrade.

Now I have discovered that under considerable pressure ranging from two to one hundred atmospheres the compound, either as vapor or liquid, is much more stable, and therefore higher temperatures can be employed in its production, while the rapidity of the process of manufacture is greatly increased. I apply this discovery to the manufacture of nickel carbonyl, from which metallic nickel may be produced in the following manner.

The nickel obtained by reduction of the oxid I subject in a strong vessel to the action of carbonic oxid compressed to from two to one hundred atmospheres according to the temperature, applying heat so as to raise the contents of the vessel to temperatures from 50° to 250° centigrade. The nickel thus treated rapidly combines with the carbonic oxid, producing vapor of nickel carbonyl. This vapor, with the excess of carbonic oxid in which it is diffused while still under pressure, on being passed through tubes at a higher temperature becomes dissociated, depositing metallic nickel, while the separated and diluent carbonic oxid can be returned to act on fresh charges of spongy nickel.

If the vapor along with the excess of carbonic oxid in which it is diffused while still under pressure be allowed to pass through a cooled condenser, the nickel carbonyl is deposited as a liquid which can be used for production of metallic nickel in the manner described in the specification above referred to or for other purposes, while the carbonic oxid still under pressure can be returned to act on fresh charges of spongy nickel.

The accompanying drawing shows diagrammatically a plant for the manufacture of nickel carbonyl according to my invention.

By means of a compressor B water-gas or any other suitable gas containing hydrogen is passed at any desired pressure into the bottom of a retort C, set in a furnace C′ and previously charged with nickel oxid which is kept at a temperature of about 300° centigrade. The hydrogen of the water-gas or other gas used reduces the nickel oxid, and the vapor of water formed and the excess of gas used pass out of the top of the retort by a pipe. (Not shown in the drawing.) The reduced nickel oxid in the retort C is now cooled to a temperature of about 100° centigrade. Carbonic oxid prepared in any convenient way and compressed to about fifteen atmospheres by the compressor F is circulated through the retort, passing in from the carbonic-oxid main H and out at the pipe D, through the regulating-valve I into the nickel-carbonyl main J, which conveys it into the dust-chamber K. From here it enters the cooler L, passing through a coil immersed in water into the condenser M, which may be cooled in any convenient manner. The condensed nickel carbonyl flows into the receiver N, from which it may be drawn off by means of a cock *n*. The excess of carbonic oxid passes on by pipe O to the circulating-pump P, which passes it into the carbonic-oxid

main H, where it again circulates as in the first operation. As the carbonic oxid combines with the nickel and the pressure is thus reduced, a fresh supply of carbonic oxid is introduced by the compressor F. The circulation of the carbonic oxid is kept up as long as nickel carbonyl is produced. When this is the case, the inlet and outlet circulating-valves I and S T on the retort are closed, and the gas in the retort is pumped out by means of the compressor F, after which the retort is cleared by removing the cover and is recharged for a second operation.

The three retorts shown are for continuous working. While the nickel oxid in No. 1 is being reduced, No. 2 is in use for gasifying the nickel and No. 3 is being recharged. These retorts are heated by gas-jets regulated by index-cocks. Regulating-valves are distributed throughout the circuit to control the various operations.

For the purpose of description the temperature of gasification of the nickel has been taken as 100° centigrade and the pressure of fifteen atmospheres is found amply sufficient; but it it is in no way to be inferred that this is the only temperature or pressure available. Thus at 180° centigrade a pressure of eighty atmospheres has been found equally ample. These examples are illustrative and indicate the fact that the temperature and pressure within the scope of my invention are correlated. As the pressure is increased the temperature is correspondingly increased, the two illustrative examples being representations of the relative increase of pressure and temperature.

It is obvious that instead of condensing the nickel carbonyl the gas containing it may be passed into a chamber heated to a temperature high enough to decompose the nickel carbonyl for obtaining metallic nickel, the temperature necessary depending on whether the pressure of the gas is maintained or allowed to fall.

According to my invention an acceleration of the reaction and a more rapid output is obtained, and this useful result is one that is increased gradually as the pressure is raised from two atmospheres to one hundred atmospheres, because as the pressure is raised the temperature can also be raised.

Having thus described the nature of this invention and the best means I know of carrying the same into practical effect, I claim—

1. A process for the manufacture of nickel carbonyl consisting in passing carbonic oxid over heated metallic nickel under a pressure between two atmospheres and one hundred atmospheres, substantially as described.

2. A process for the manufacture of nickel carbonyl, consisting in passing carbonic oxid over heated metallic nickel under a pressure between two atmospheres and one hundred atmospheres, condensing the nickel carbonyl to separate it from the excess of carbonic oxid while the gases are still under pressure and passing the carbonic oxid again over metallic nickel, while still under pressure, substantially as described.

3. A process for the manufacture of nickel carbonyl, consisting in passing carbonic oxid over metallic nickel heated to substantially 100° centigrade under a pressure of approximately fifteen atmospheres, substantially as described.

4. A process for the manufacture of nickel carbonyl, consisting in passing carbonic oxid over a metallic nickel heated to substantially 100° centigrade under a pressure of approximately fifteen atmospheres, condensing the nickel carbonyl to separate it from the excess of carbonic oxid while the gases are still under pressure and passing the carbonic oxid again over metallic nickel, while still under pressure, substantially as described.

In testimony whereof I have hereunto set my hand in presence of two subscribing witnesses.

JAMES DEWAR.

Witnesses:
OLIVER INNACE,
GERALD L. SMITH.