

UNITED STATES PATENT OFFICE.

CARL BOSCH AND ALWIN MITTASCH, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY,
ASSIGNORS TO BADISCHE ANILIN AND SODA FABRIK, OF LUDWIGSHAFEN-ON-
THE-RHINE, GERMANY, A CORPORATION.

PROCESS OF PRODUCING MOLYBDENUM NITRID.

1,061,725.

Specification of Letters Patent.

Patented May 13, 1913.

No Drawing.

Application filed September 19, 1911. Serial No. 650,246.

To all whom it may concern:

Be it known that we, CARL BOSCH and ALWIN MITTASCH, subjects, respectively, of the King of Prussia and the King of Saxony, residing at Ludwigshafen-on-the-Rhine, Germany, have invented new and useful Improvements in Producing Molybdenum Nitrid, of which the following is a specification.

The usual process of preparing metallic nitrids from the corresponding oxids and nitrogen has hitherto been carried out in the presence of solid reducing agents. We have now found that we can obtain nitrogen compounds of molybdenum with great facility by treating an oxygen compound of molybdenum with nitrogen in the presence of a reducing gas, the process being carried out at a temperature under eight hundred degrees centigrade. We have also found that it is not necessary to react on the oxygen compound of molybdenum simultaneously with nitrogen and with the reducing gas, but that, if desired, the oxygen compound can first be treated with the reducing gas, so that more or less complete reduction of the molybdenum oxygen compound is brought about, and the product be afterward treated with nitrogen. Since the reaction, and particularly the fixation of the nitrogen, proceeds more rapidly under pressure, we prefer to employ pressure above that of the atmosphere when carrying out our process. As instances of oxygen compounds of molybdenum which can be used according to our invention, we mention the oxids and hydroxids of molybdenum and also mixtures of oxids of molybdenum with metallic molybdenum, such for instance as are obtainable by the incomplete reduction of oxids of molybdenum. Other compounds containing oxygen, for instance, molybdates, can be employed. If desired the process of this reaction can be carried out in the presence of a suitable catalytic agent, such for instance as iron, cobalt, or nickel, or their compounds, without departing from the nature of the invention.

The molybdenum-nitrogen compounds obtainable according to our invention can be employed in various ways, for instance, on

being heated to a higher temperature, preferably *in vacuo*, they can be converted into metallic molybdenum, which is thus obtainable with facility in a pure state. Or the nitrids of molybdenum can be employed for producing valuable nitrogen compounds, for instance on treating the nitrid with hydrogen, or with steam, ammonia is formed, and in the latter case the oxid produced can be re-converted into the nitrid, so that the same quantity of molybdenum compound can be employed repeatedly in the manufacture of ammonia.

The following examples will serve to illustrate further the nature of our invention and how it can be carried into practical effect, but the invention is not confined to these examples.

Example 1: Heat molybdic acid (H_2MoO_4) at a temperature of from five hundred to six hundred degrees centigrade, in a mixture containing equal parts of hydrogen and nitrogen, while maintaining a pressure of sixty atmospheres.

Example 2: Reduce sublimed molybdenum trioxid by means of hydrogen at from five hundred to six hundred degrees centigrade, either at ordinary, or at increased, pressure, and then, at five hundred degrees centigrade and at a pressure of fifty atmospheres, treat, with nitrogen, the product thus obtained.

Example 3: Reduce precipitated molybdic acid at $700^\circ C.$ with hydrogen and then, at the same temperature, pass over pure nitrogen at atmospheric pressure.

Instead of hydrogen, other reducing gases, such for instance as carbon monoxid and hydrocarbons, or mixtures of reducing gases, such for instance as water gas, can be employed. If desired, hydrogen or other indifferent gas can be mixed with the nitrogen.

Example 4: Pass a current of hydrogen and nitrogen, either simultaneously or one after the other, over a mixture containing 90 parts of molybdic acid and 10 parts of nickel oxid at from 500° to $600^\circ C.$ and at ordinary atmospheric pressure. In this example, the nickel oxid can be replaced by other oxids; such for instance as iron oxid

and cobalt oxid, or by the metals themselves or other suitable compounds thereof.

Example 5: Pass first methane and then nitrogen over molybdic acid at from 500° to 600° C. at a pressure of 50 atmospheres.

Now what we claim is:—

1. The process of producing molybdenum nitrid by heating a compound of molybdenum containing oxygen with a reducing gas and with nitrogen at a temperature between 400° and 800° C.

2. The process of producing molybdenum nitrid by heating a compound of molybdenum containing oxygen with hydrogen and with nitrogen at a temperature between 400° and 800° C.

3. The process of producing molybdenum nitrid by heating a compound of molybdenum containing oxygen with a reducing gas and with nitrogen under pressure, at a temperature between 400° and 800° C.

4. The process of producing molybdenum nitrid by heating a compound of molybdenum containing oxygen first with a reducing gas and then with nitrogen under pres-

sure at a temperature between 400° and 800° C.

5. The process of producing molybdenum nitrid by heating an oxid of molybdenum with hydrogen and with nitrogen under 30 pressure at a temperature between 400° and 800° C.

6. The process of producing molybdenum nitrid by heating molybdenum trioxid with hydrogen and with nitrogen under pressure 35 at a temperature between 400° and 800° C.

7. The process of producing molybdenum nitrid by heating molybdenum trioxid first with hydrogen and then with nitrogen under pressure at a temperature between 40 400° and 800° C.

In testimony whereof we have hereunto set our hands in the presence of two subscribing witnesses.

CARL BOSCH.
ALWIN MITTASCH.

Witnesses:

J. ALEC. LLOYD,
JOSEPH PFEIFFER.