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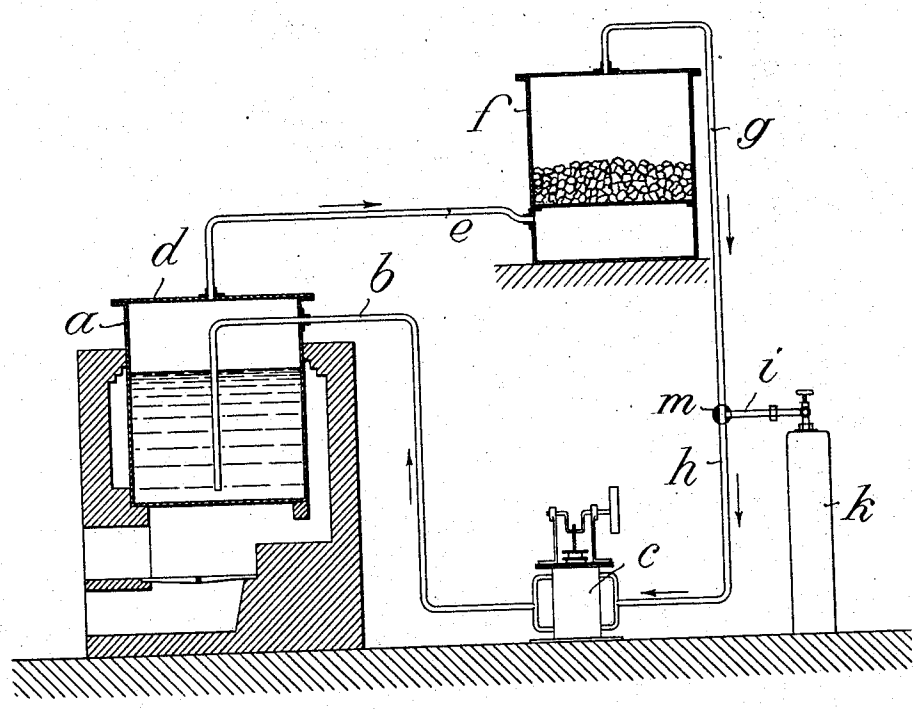
PROCESS FOR THE MANUFACTURE OF AMIDS OF HIGHER FATTY ACIDS.

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SPECIMENS.



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UNITED STATES PATENT OFFICE.

OSCAR AUGUST HUBERT HUGO KÖSTERS, OF HEMELINGEN, AND LOUIS OTTEMANN, OF BREMEN, GERMANY.

PROCESS FOR THE MANUFACTURE OF AMIDS OF HIGHER FATTY ACIDS.

No. 915,680. Specification of Letters Patent. Patented March 16, 1909.

Original application filed March 29, 1907, Serial No. 365,247. Divided and this application filed February 8, 1908. Serial No. 414,905. (Specimens.)

To all whom it may concern:

Be it known that we, OSCAR AUGUST HUBERT HUGO KÖSTERS, a resident of Hemelingen, Germany, and LOUIS OTTEMANN, a resident of Bremen, Germany, both subjects of the Emperor of Germany, have jointly invented certain new and useful Improvements in Processes for the Manufacture of Amids of the Higher Fatty Acids, of which the following is a specification.

It is well known that amids of the higher fatty acids are formed upon heating the ammonium salts of said acids in a closed vessel (digester). This process for the production of said amids is not available commercially, for the reason that the production of ammonium salts of the higher fatty acids is very difficult, and that the water formed during the reaction in the closed vessel has a tendency to decompose the amids produced. Moreover this water, which remains in the vessel during the entire process, is detrimental particularly as it exerts a pressure of from 25 to 30 atmospheres (at the temperature employed), and this pressure is further increased, when the ammonium salts of the fatty acids are heated, by the liberated ammonia; the consequence is that very strong apparatus is required for carrying out the process above referred to. The term "higher fatty acids" as used herein is applied to fatty acids containing at least six carbon atoms.

Our present invention avoids the defects just mentioned, and consists, briefly described, in heating the higher fatty acids in a closed vessel under regulatable pressure in the presence of gaseous ammonia.

The accompanying drawing illustrates in a diagrammatic manner, an apparatus suitable for carrying out our process hereafter explained.

The apparatus comprises a suitable closed vessel *a* (for instance, a digester), connected with the delivery port of a pump *c* by a pipe *b* extending almost to the bottom of the vessel. From the cover *d* a pipe *e* leads to a drying chamber or tower *f* of any suitable construction, which by pipes *g*, *h* is connected with the suction port of the pump *c*. A branch pipe *i* connects the pipes *g*, *h* with a cylinder *k* containing liquid ammonia; a three-way cock *m* is preferably located at the junction of the pipes *g*, *h*, *i*. A circulating path is thus provided from the pump *c* through pipe *b* to vessel *a*, thence through pipe *e* to drying chamber *f*, and back to the pump through pipes *g*, *h*. Any suitable drying agent, such as caustic lime, may be used in the chamber *f*. The vessel *a* is heated in any suitable manner, for instance, as shown, by means of a furnace in which it is set.

The operation is as follows: The vessel *a* of a capacity (of say 300 liters) is filled with a suitable amount (say 100 kilograms) of a higher fatty acid, such as stearic acid, and heated to the proper temperature for instance 190° centigrade. Thereupon, the pump *c* is set in motion and the cock *m* as well as the discharge valve of the receptacle *k* are opened, so as to allow the liquid ammonia to escape in gaseous form into the pipes *i*, *h* and to pass into the heated mass contained in the vessel *a*. This gas, which is dry, is supplied in greater amount than is necessary for the reaction. An amid and water are formed by the action of the ammonia gas on the heated fatty acid, said water being carried away as vapor with the excess of ammonia gas passing to the drying chamber *g* through the pipe *e*. The drying agent in the chamber *f* absorbs such water, so that dry ammonia gas returns to the pump *c* through the pipes *g*, *h*. The deficiency caused by the combination of the ammonia with the fatty acid, is supplied by the admission of new gas from the receptacle *k*. The pressure maintained in the apparatus during the reaction may be about two atmospheres or less, and the process is completed in about three hours. The ammonia first forms an ammonium salt with the fatty acid, and from this ammonium salt the amid of the fatty acid is formed; both reactions, however, are practically simultaneous.

The first reaction may be expressed by the equation:

$$\underset{\text{Stearic acid.}}{C_{17}H_{35}COOH} + \underset{\text{Ammonia.}}{NH_3} = \underset{\text{Stearate of ammonium.}}{C_{17}H_{35}COONH_4}$$

and the second reaction by the equation:

$$\underset{\text{Stearate of ammonium.}}{C_{17}H_{35}COONH_4} = \underset{\text{Stearic amid.}}{C_{17}H_{35}CONH_2} + \underset{\text{Water.}}{H_2O}$$

One of the great advantages of our method consists in the immediate removal of the reaction water from the heated vessel; another important advantage is the fact that the pressure within the digester is independ-

ent of the temperature employed. Since the ammonia gas travels in a continuous path or cycle, and is dried before it returns to the vessel *a*, a perfect utilization of the gas is secured, and therefore a very economical working.

This application is a division of one filed by us in the United States Patent Office on March 29, 1907, Serial No. 365, 247.

We claim as our invention:

1. The process of producing amids of the higher fatty acids, which consists in heating the said acids in a closed vessel in the presence of ammonia gas, and withdrawing the excess of ammonia gas together with any water formed during the reaction.

2. The process of producing amids of the higher fatty acids, which consists in heating the said acids in a closed vessel in the presence of ammonia gas, withdrawing the excess of ammonia gas together with any water formed during the reaction, drying such withdrawn ammonia gas, and returning it into contact with the fatty acid.

In testimony whereof, we have hereunto signed our names in the presence of two subscribing witnesses.

OSCAR AUGUST HUBERT HUGO KÖSTERS.
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Witnesses:
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