

PATENT SPECIFICATION



Application Date : June 10, 1926. No. 14,657 / 26.

278,777

Complete Accepted : Oct. 10, 1927.

COMPLETE SPECIFICATION.

Improvements in the Manufacture and Production of Valuable Organic Compounds.

I, JAMES YATE JOHNSON, a British subject, of 47, Lincoln's Inn Fields, in the County of London, Gentleman, do hereby declare the nature of this invention (which has been communicated to me from abroad by I. G. Farbenindustrie Aktiengesellschaft, of Frankfurt-on-Main, Germany, a corporation organized according to German laws) and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :—

In the catalytic hydrogenation of oxides of carbon, mixtures of oxygen-containing organic compounds of a higher order than methanol, and of an oily nature, consisting chiefly of saturated alcohols with four or more carbon atoms besides unsaturated alcohols, aldehydes, ketones, esters and other compounds, are obtained under certain conditions, for instance, in accordance with the processes described in the Specifications Nos 227,147, 229,714, 237,030 and 238,319. These oily products are often coloured and have an unpleasant smell.

My foreign correspondents have now found that these oily products can be rapidly and easily converted into colourless oils which do not grow dark and with only a very slight smell, by subjecting them while in the vapour state to catalytic hydrogenation. The reaction product so obtained consists substantially of alcohols which can be separated for example by fractional distillation much more simply than is possible with the crude mixture containing aldehydes and ketones, part of which compounds forms very difficultly separable fractions. Hydrogenation may be applied not only to the crude product as it is directly obtained, but also to fractions of such product, among which those boiling above 150° Centigrade often contain

compounds causing darkening and unpleasant smell.

Preferably the hydrogenation is carried out at moderately elevated temperatures between about 100° and 180° Centigrade. It is advantageous, especially when treating oils boiling above 200° Centigrade, to work at temperatures below the boiling point of the oil, as under these conditions conversion into valuable compounds is effected in a particularly simple manner.

The catalysts to be used in the hydrogenation are those usually employed for this purpose, as for example nickel, cobalt, copper, platinum alone or in mixtures, with or without a support or activating admixture or both.

The oils to be hydrogenized may also be subjected to a preliminary purification for example by treating them for about an hour at 90° Centigrade with about 10 per cent. their weight of granular adsorbing silica and filtering or by treating them with caustic soda lye.

The following examples will further illustrate how the present invention may be carried out in practice, but the invention is not limited to these examples. The parts are by weight, unless otherwise stated.

EXAMPLE I.

A contact mass is prepared by making 100 parts of granulated pumice stone, 9 parts of nickel carbonate and 1 part of copper carbonate into a paste by means of 9 parts of sodium silicate and water and reducing the mixture at from 300° to 320° Centigrade. A mixture of the vapours of an oily product obtained by the catalytic hydrogenation of oxides of carbon in accordance with any of the examples of Specification No. 238,319 and boiling between about 180° and 250°

Centigrade, with hydrogen, which mixture consists of about 2 parts, by volume, of organic vapour and 98 parts, by volume, of hydrogen, is passed at about 150° to 170° Centigrade over the said contact mass. On cooling the gases leaving the reaction vessel, a colourless oil similar to amyl alcohol and smelling only slightly is obtained.

EXAMPLE 2.

A mixture of hydrogen with vapours of an oily product obtained by the catalytic hydrogenation of oxides of carbon in accordance with any of the examples of Specification No. 238,319 and boiling between 80° and 250° Centigrade is passed at about 130° and 140° Centigrade over a contact mass consisting of 100 parts of granulated pumice stone, 9 parts of nickel carbonate and 3.6 parts of aluminium hydroxide, which are made into a paste by means of 9 parts of sodium silicate and water and reduced at from about 300° to 320° Centigrade. The speed of the gas and the degree of dilution of the vapours is controlled so as to avoid condensation of components of higher boiling point in the reaction vessel. A colourless oil with a pleasant smell of alcohols is obtained.

Instead of aluminium hydroxide the contact mass may also contain aluminium salts, for instance aluminium phosphate, Thoria, tungsten oxide and other like activating additions are also suitable.

Having now particularly described and

ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process for the conversion of oxygen-containing organic compounds of a higher order than methanol obtainable by the catalytic hydrogenation of oxides of carbon, for instance in accordance with the process described in the Specifications Nos. 227,147, 229,714, 237,030 and 238,319, into colourless products consisting substantially of higher alcohols which consists in treating the said products while in the vapour state with hydrogen free from oxides of carbon at an elevated temperature, but preferably below their boiling point, in the presence of a hydrogenating catalyst, and if desired after a preliminary purification.

2. The process for the conversion of oxygen-containing organic compounds of a higher order than methanol into colourless products consisting substantially of higher alcohols, substantially as described in each of the foregoing examples.

3. Higher alcohols when prepared in accordance with the preceding claiming clauses.

Dated this 10th day of June, 1928.

JOHNSONS & WILLCOX,
47, Lincoln's Inn Fields, London,
W.C. 2,
Agents.