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PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Process for the Purification of Alcohols Obtained by the Catalysed Interaction of Hydrogen with the Oxides of Carbon.

I, ARTHUR CARPMAEL, British subject of 24, Southampton Buildings, London, W.C. 2, do hereby declare the nature of this invention (which has been communicated to me by I. G. Farbenindustrie Aktiengesellschaft, a Joint Stock Company organised under the laws of Germany, of Frankfort-on-Main, Germany,) and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the purification of synthetic products, comprising alcohols of the most varied kind, such as methanol, isobutyl alcohol and the like, obtained by the catalysed interaction of hydrogen with the oxides of carbon.

Synthetic products from carbonic oxides and hydrogen, such as methanol, isobutyl alcohol and the like, sometimes possess a peculiar unpleasant odour caused by the presence of small quantities of various undesired substances. These impurities generally interfere with the purification of the synthetic products and of their distillation fractions, since the customary purification methods applied, for example, to methyl alcohol, e.g. fractional distillation in a column apparatus, in most instances do not suffice to produce a high quality product as regards boiling point, stability to permanganate, odour and the like.

In accordance with the present invention this disadvantage is removed.

The process of purification in accordance with this invention is by the treatment of the synthetic product or of the distillation fractions thereof with oxidising agents, with or without the addition of relatively non-volatile alkalies or organic bases and of metallic halides other than alkali halides, if necessary diluting with water before or during distillation and with or without treatment with purifying agents acting by adsorption.

The quantities of the oxidising agents required are very small. It is not necessary to employ an amount of oxidising agent, for example, permanganate, which would correspond to the oxidisable substances, which are destroyed thereby at ordinary temperature, but it is sufficient to add, for example, only 0.1 to 0.8% of permanganate calculated on the weight of the raw product in order to improve its quality very considerably. The treatment is extremely simple. The requisite quantity of potassium permanganate or other oxidising agent, such as hydrogen peroxide, persulphate, perborate, percarbonate, hypohalite and the like is dissolved in water and the synthetic raw product or its distillation fractions are gradually stirred with this solution. The purifying action begins immediately.

The addition of other purifying substances, for example, caustic soda solution, sodium bisulphite or oxalic acid to the distilling liquid during the distillation is very often advantageous. It is further of considerable advantage to subject the raw product or the fractions thereof to a treatment with purifying agents acting by adsorption, such as for example, Fuller's earth, decolourising carbon, active carbon, silica gel and like active purifying substances.

In the case of certain especially impure products it has also been found to be advantageous to add to the synthetic product or the distillation fractions thereof, after the treatment with oxidising agents, small quantities (about 0.3 to 0.5% of the weight of the raw product) of relatively non-volatile organic bases, such as for example, crude phenylene diamine (chiefly *m*-phenylenediamine), crude tolylene diamine (chiefly 1.2.4-tolylene diamine), aminophenols, phenyl hydrazine and the like. The initial purifying effect due to the action of the oxidising agent is supplemented by this after treatment with organic bases.

The following example illustrates the invention:—

20,000 kg. of methanol produced from hydrogen and the oxides of carbon are gradually mixed at ordinary temperature in a stirring apparatus (in the course of about one hour) by stirring, with a solution of 30 kg. of potassium permanganate in water and the mixture is then fractionated in an ordinary column in the

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known manner with or without the addition of water and of relatively non-volatile organic bases.

The raw methanol previously treated with potassium permanganate gives, on distillation, as mentioned above, a higher yield of pure methanol and a considerably purer end product than the raw methanol without previous chemical purification with permanganate.

In such cases when the raw produce also contains, besides other impurities, ammonia or relatively volatile organic bases, it is treated for the purpose of fixing these impurities (in addition to the treatment with oxidising agents) with metallic halides other than alkali halides or with solutions thereof, for example, zinc chloride, and then distilled.

Products are thus obtained, which are completely free from ammonia and organic bases. The quantity of metallic halide used depends on the quantity of impurity present.

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. Process for the purification of alcohols obtained by the catalysed interaction of hydrogen with the oxides of carbon, such as methanol, isobutyl alcohol and the like, in which the synthetic product or a distillation fraction thereof is treated with an oxidising agent.

2. Process as claimed in claim 1 in which a relatively non-volatile alkali or organic base and/or metallic halide other than an alkali halide is added.

3. Process as claimed in claim 1 or 2 in which the products to be purified are diluted with water before or during distillation.

4. Process as claimed in claims 1—3 in which a purifying substance acting by adsorption is employed.

5. Process for the purification of methanol obtained by the catalysed interaction of hydrogen with the oxides of carbon, substantially as described with reference to the example.

Dated the 16th day of February, 1928.
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