[Second Edition.]

PATENT SPECIFICATION

Application Date: Dec. 10, 1929. No. 37,858 / 29.

344,796

Complete Left: June 23, 1930.

Complete Accepted: March 10, 1931.

2581

PROVISIONAL SPECIFICATION.

Improved Method for the Purification of Synthetic Alcohols.

We, JAMES RITCHIE PARK, a British subject, of Norton Hall, The Green, Norton-on-Tees, County Durham, and IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W. 1, do hereby declare the nature of this invention to be as follows:—

Synthetic alcohols prepared by catalytic hydrogenation of oxides of carbon under pressure are often contaminated with iron compounds, e.g. iron carbonyl. The present invention relates to an improved method of purifying such products. Methods for purifying synthetic alcohols are known in which a mild oxidising agent in alkaline solution is employed.

We have now found that if an alkali, e.g. caustic soda, is dissolved in crude methanol or other synthetic alcohol and the mixture allowed to stand for some time preferably at the ordinary temperature, the iron compounds, especially iron carbonyl, are decomposed and on distillation of the alcohol a product free from iron is obtained. The mixture consisting of crude alcohol and alkali may be warmed if desired, the reaction being accelerated in this way, but it is essential that the mixture be allowed to stand for some time, preferably one day or more, before distillation. The invention does not extend to processes in which alkali is added during distillation.

In an example 50—100 g. of solid caustic soda were dissolved without heating in 10 litres of methanol containing 10 mg. of iron carbonyl. The mixture was allowed to stand for 100 hours at room temperature, after which time it was distilled. The distillate was found to be free from iron.

In a second experiment, the crude methanol was heated to its boiling point after the addition of caustic soda and maintained at that temperature for 24 hours, when on distillation, no iron was found in the product.

Dated the 10th day of December, 1929.

E. C. J. CLARKE,
Imperial Chemical House, Millbank,
London, S.W. 1,
Solicitor for the Applicants.

COMPLETE SPECIFICATION.

Improved Method for the Purification of Synthetic Alcohols.

We, JAMES RITCHIE PARK, a British subject, of Norton Hall, The Green, Norton-on-Tees, County Durham, and IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W. 1, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

Synthetic alcohols prepared by catalytic hydrogenation of oxides of carbon under pressure are often contaminated with iron compounds, e.g. iron carbonyl, which cannot be completely removed by simple distillation. With a view to the removal of small quantities of various undesired substances causing a peculiar unpleasant odour, it has already been proposed to treat such synthetic alcohols, or a distillation fraction thereof, with an oxidising agent, if desired with the addition of other purifying substances such as caustic soda solution, sodium bisulphite, or oxalic acid to the distilling liquid during the distillation.

According to the present invention crude synthetic alcohols of the kind described, or a distillation fraction thereof, are subjected to a prolonged treatment with alkali for the purpose of eliminating iron compounds. Thus crude synthetic methanol, i.e. the condensate separating on cooling a gas mixture containing oxides of carbon and hydrogen which has been passed under pressure and at a suitable temperature over a methanol-

[P

forming catalyst, may be mixed with solid caustic soda and the mixture allowed to stand for several days at the ordinary temperature. If desired, the mixture may be maintained at a raised temperature, employing a reflux condenser if required, the necessary period of treatment being thereby diminished. The mixture whether at the ordinary or a raised temperature, may be stirred during the treatment, and a slow current of air may be employed for this purpose. It may also be advantageous, especially when the mixture is allowed to stand at the ordinary temperature without subsequent stirring to aerate the mixture at the commencement of the treatment, e.g. by passing a current of air through the mixture, to ensure the presence of dissolved oxygen in the liquid, which is believed to play a part in the purification. After the prolonged treatment with alkali it will generally be convenient to distil the mixture as such to recover a product substantially free from iron, but this distillation may be preceded by separation of any solid matter from the liquid, e.g. by filtration.

Instead of caustic soda, other alkaline substances, e.g. potash or potassium or sodium carbonate, may be employed, if desired in aqueous solution.

The vessels employed for handling the crude and pure methanol may be made of mild steel as there is no appreciable contamination of the methanol through contact with iron or steel.

EXAMPLE.

One cubic metre of crude synthetic methanol, estimated to contain 130 parts of iron per thousand million, was allowed to flow slowly at the ordinary temperature over 6 kilograms of solid caustic soda supported on a perforated steel tray, whereby most of the caustic soda was dissolved. The solution was collected in a steel tank and a slow stream of air was passed through the liquid while filling, which occupied about 2 hours. The mixture was then allowed to stand at the ordinary temperature, without further supply of air, for 5 days. It was then distilled, the methanol obtained being estimated to contain not more than 2 parts of iron per thousand million.

The method of estimating the very small quantities of iron was to vaporise a known quantity of methanol and to pass the vapours together with air over a roll of silver gauze at a red heat, as in formaldehyde manufacture. The iron was retained in the gauze and the quantity in the methanol used could be estimated by weighing the gauze before and after the passage of the vapours.

The purified methanol obtained according to the invention is especially suitable for the manufacture of formaldehyde by catalytic oxidation, using a silver gauze catalyst, since it causes no appreciable loss of activity of the catalyst during an extended run, whereas the use of crude methanol results in a relatively rapid deterioration of the catalyst.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed we declare that what we claim is:—

1. Process for the removal of iron compounds from crude synthetic alcohols, or distillation fractions thereof, prepared by catalytic hydrogenation of oxides of carbon, which includes the step of subjecting the impure material to a prolonged treatment with alkali.

2. Process as claimed in Claim 1 in which the impure alcohol is allowed to stand for several days in the presence of caustic soda.

3. Process as claimed in Claim 1 or 2 in which the mixture is stirred during the treatment.

4. Process as claimed in Claim 3 in which a current of air is employed for stirring.

5. Process as claimed in Claim 1 or 2 in which the liquid is aerated at the commencement of the treatment and is then allowed to stand.

6. Synthetic alcohols, particularly methanol, substantially freed from iron by the process claimed in any of the preceding claims or by the obvious chemical equivalent of such process.

Dated the 17th day of June, 1930.

E. C. J. CLARKE,
Imperial Chemical House, Millbank,
London, S.W. 1,
Solicitor for the Applicants.

Abingdon: Printed for His Majesty's Stationery Office, by Burgess & Son.
[Wt. 95a.—125/9/1932.]