UNITED STATES PATENT OFFICE.

OTTO GRAUL, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, ASSIGNOR TO BADISCHE ANILIN & SODA FABRIK, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, A CORPORATION.

PRODUCING ISO-OLEFINS.

1,102,655. Specification of Letters Patent. Patented July 7, 1914.

No Drawing. Application filed March 13, 1913. Serial No. 754,147.

To all whom it may concern:

Be it known that I, OTTO GRAUL, citizen of the German Empire, residing at Ludwigshafen-on-the-Rhine, Germany, have invented new and useful Improvements in Producing Iso-Olefins, of which the following is a specification.

I have found that normal olefins containing at least five carbon atoms, by being heated to a high temperature, can be converted into iso-olefins, that is to say, an intramolecular change takes place and the normal hydrocarbon is converted into one with a branched chain of carbon atoms. For instance, normal pentene can be converted into trimethyl-ethylene. It is often advantageous to carry out the reaction in the presence of a catalytic agent and under reduced pressure. It is not necessary to start directly from the normal olefins when carrying out this reaction, since compounds which are easily convertible into these can be employed. For instance, instead of normal pentenes, halogen normal pentane and pentyl alcohols, can be used, which are easily convertible into normal pentenes. Such a procedure is of course equivalent to starting with the normal olefins.

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

Example 1: Pass 1,065 parts of monochlor normal pentane (prepared by chlorinating the fraction of gasolene boiling at from 36° to 38° C.) over 2,000 parts of powdered quicklime at from 385° to 400° C., and then pass over calcined aluminium oxid the vaporized normal pentene which is formed, the said oxid being maintained at about 450° C., although, if desired, the temperature can be maintained somewhat lower, and this is useful at the commencement of the reaction. Isolate the product by cooling well, whereupon a liquid of boiling point of from 34° to 38° C. is obtained. In order to prepare the isopentene in a pure form, agitate it with concentrated aqueous hydrochloric acid, whereby the trimethyl-ethylene is converted into isoamyl chlorid of boiling point of from 84° to 86° C. Then separate this from unaltered normal pentene and any small quantities of isopropyl-ethylene by distillation. These latter can be separated or can be passed together anew over heated alumina. The hydrochloric acid can be split off again from the isoamyl chlorid, whereby isopentene of boiling point of from 34° to 36° C. is obtained.

Example 2: Mix 86 parts of normal methyl-propyl-ketone (prepared by boiling ethyl-aceto acetate with caustic potash, *Annalen* 138, 216) with 100 parts of 30% caustic soda solution and 250 parts of ether. Then add, slowly, while stirring, 50 parts of sodium and at the same time allow 200 parts of water to flow slowly into the mixture. In this way the corresponding secondary alcohol of boiling point of from 115° to 117° C. is obtained. Then pass this alcohol, in the form of vapor, over aluminium oxid at from 450° to 475° C. Cool the product and, if desired, purify the obtained isopentene as described in the foregoing example.

Example 3: Pass normal hexylene (obtained from monochlor-n-hexane) over calcined aluminium oxid at about 400° C. An almost quantitative yield of a distillate is obtained which, upon treatment with concentrated aqueous hydrochloric acid, gives a monochlorid boiling at about from 110° to 115° C., and consisting principally of 2-chlor-2-ethyl-butane.

Now what I claim is:—

1. The process of producing iso-olefins by heating normal olefins containing at least 5 carbon atoms to a high temperature.

2. The process of producing iso-olefins by heating normal olefins containing at least 5 carbon atoms to a high temperature in the presence of a catalytic agent.

3. The process of producing iso-olefins by heating normal olefins containing at least 5 carbon atoms to a high temperature in the presence of a catalytic agent and under reduced pressure.

4. The process of producing isopentene by passing vaporized normal pentene over aluminium oxid at a high temperature.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses.

OTTO GRAUL.

Witnesses:
JOSEPH PFEIFFER,
J. ALEC. LLOYD.