

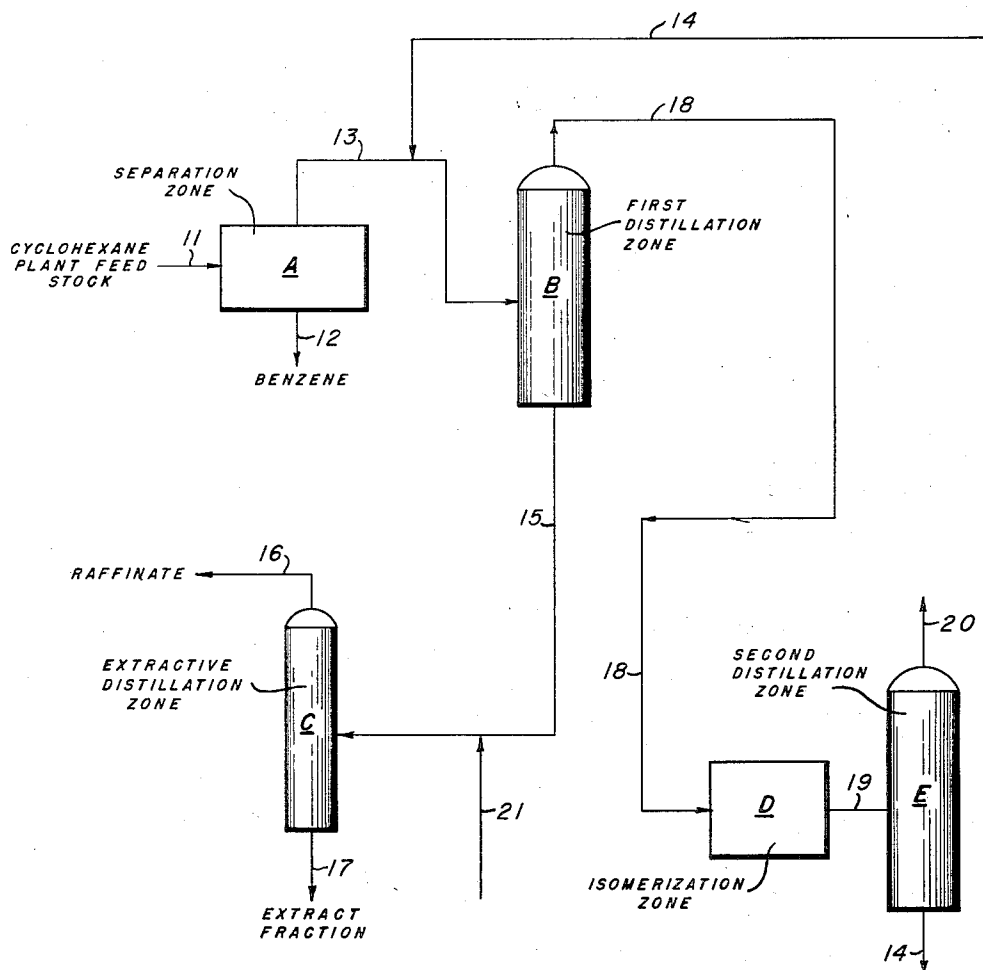
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PRODUCTION OF HIGH PURITY CYCLOHEXANE CONCENTRATE

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PRODUCTION OF HIGH PURITY CYCLOHEXANE CONCENTRATE

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2 Claims. (Cl. 260-666)

The present invention is directed to a procedure for preparing a pure hydrocarbon. More particularly the present invention is directed to a process for preparing a high purity cyclohexane concentrate from a feed stock consisting of a close boiling mixture of saturated and aromatic hydrocarbons having 6 and 7 carbon atoms in the molecule and isomers of cyclohexane including n-hexane, benzene, methylcyclopentane, cyclohexane, and dimethylpentanes. The process of the present invention is applicable to a hydrocarbon feed stock which contains at least 5% by volume of benzene and in which the ratio of cyclohexane to benzene is no greater than 3 to 1 by volume. The process of the present invention is particularly useful when the benzene content of the cyclohexane product must be reduced to less than 0.5%. In accordance with the present invention, a high purity cyclohexane concentrate containing at least 95% by volume cyclohexane and up to greater than 99.5% purity cyclohexane may be secured.

Further objects and advantages of the present invention may be seen from the following description taken in conjunction with the drawing in which the sole figure is in the form of a diagrammatic flow sheet.

In certain commercial operations, as in a petroleum refinery, a mixture of close boiling hydrocarbons is available. For convenience, this fraction may be referred to as cyclohexane plant feed stock. This feed stock may contain n-hexane, benzene, methylcyclopentane, cyclohexane and dimethylpentanes and other hydrocarbon. The boiling points of these compounds are set out in Table I hereafter:

Table I

Hydrocarbon:	Boiling point at 760 mm., ° C.
Normal hexane	68.740
Methylcyclopentane	71.812
2,2-dimethylpentane	79.197
Benzene	80.100
2,4-dimethylpentane	80.500
Cyclohexane	80.738
2,2,3-trimethylpentane	80.882
3,3-dimethylpentane	86.064
1,1-dimethylcyclopentane	87.846
2,3-dimethylpentane	89.784
2-methylhexane	90.052
1,trans-3-dimethylcyclopentane	90.773
1,cis-3-dimethylcyclopentane	91.725
3-methylhexane	91.850
1,trans-2-dimethylcyclopentane	91.869

By way of example in Table II hereafter is given the composition of a typical plant cyclohexane feed stock:

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Table II

Hydrocarbon:	Composition, vol. percent
Lower boiling components	Trace
Normal hexane	37.0
Methylcyclopentane	30.1
2,2-dimethylpentane	2.2
Benzene	9.9
2,4-dimethylpentane	1.3
Cyclohexane	19.5
Higher boiling components	Trace
	100.0

High purity fractions, particularly cyclohexane in the range given, may be secured from such a mixture of hydrocarbons.

It is to be understood that this typical composition is given by way of example only and that actually cyclohexane feeds containing varying amounts of benzene may be satisfactorily treated in accordance with the present invention provided the benzene content is above 5%. For example, feed stocks containing from about 5% to about 30% benzene may be employed. The benzene and cyclohexane contents may vary as shown in Table III:

Table III

Benzene, percent vol.:	Cyclohexane, percent vol.
20	Up to 60
10	Up to 30
5	Up to 15

Turning now specifically to the drawing, a feed stock which may suitably consist of n-hexane, benzene, methylcyclopentane, cyclohexane and dimethylpentanes, designated in the drawing as cyclohexane plant feed stock, is passed through inlet line 11 to separation zone A where it is separated into a first fraction which is a benzene concentrate and removed through outlet line 12 and a second fraction containing the remaining portions of the feed stock and removed through outlet line 13. The separation carried out in separation zone A may be secured by the use of any one of several well known procedures. By way of specific example, the separation may be carried out by extractive distillation or solvent extraction or absorption. The use of these procedures is well known to the art. It will often be found preferable to employ extractive distillation for carrying out the separation in zone A and a process of such character for the separation of aromatic hydrocarbons is described in U. S. Patent 2,288,126. If extractive distillation is used to conduct the separation in zone A the benzene fraction removed through line 12 may be considered the extract fraction and the fraction removed through line 13 may be considered the raffinate fraction.

The fraction passing through line 13 has admixed therewith from line 14 another fraction consisting of a mixture of methylcyclopentane, cyclohexane, and traces of heavier material. The origin of this fraction will be described hereafter. The resulting admixture passes into the first fractional distillation zone B where it is separated into a bottoms fraction removed through line 15 and an overhead fraction removed through line 18. The bottoms fraction in line 15 may contain from about 80% to about 95% by volume of cyclohexane. The bottom fraction in line 15 then passes to an extractive distillation zone C where it is separated into a raffinate fraction removed

from the system through outlet line 16 and an extract fraction consisting of a high purity cyclohexane concentrate (at least 95% and no higher than 99.5% cyclohexane) which is withdrawn from the system through outlet line 17. As heretofore explained, the use of extractive distillation for carrying out separations is well known to the art being described, for example, in U. S. Patent 2,288,126 and in order to simplify the present description, the details of such a process will not be set out herein.

The overhead fraction from first fractional distillation zone B removed through line 18 consists of a mixture of n-hexane, dimethylpentanes and methylcyclopentane, and a trace of lower boiling components and is charged to isomerization zone D where at least a substantial portion of the methylcyclopentane content is converted to cyclohexane. The isomerization of methylcyclopentane to cyclohexane is known to the art. This isomerization step may conveniently be carried out as a catalytic process using for example a Friedel-Crafts type of catalyst such as promoted aluminum chloride, such as with HCl and the like. Operating temperatures are preferably within the range of 120° F. to 250° F. with a space velocity of .2 to 2 v./v./hr. The effluent from isomerization zone D passes through line 19 to second fractional distillation zone E where it is separated into an overhead fraction consisting principally of n-hexane and lighter materials which may be formed in the isomerization zone and which is withdrawn through line 20 and discarded from the system and a bottoms fraction consisting of methylcyclopentane, cyclohexane and traces of heavier material which may be formed in the isomerization zone and which is withdrawn through line 14 and admixed with the fraction in line 13 as previously described.

As an alternative procedure, in addition to the bottoms passed from distillation zone B to extractive distillation zone C for separation therefrom of high purity cyclohexane, a fraction from an extraneous source containing within the range of 65% to 95% cyclohexane and excluding as far as possible methylcyclopentanes and compounds boiling lower than methylcyclopentane and no more than 5% of fractions higher boiling than cyclohexane may be introduced through line 21 and admixed with the fraction in line 15, the mixture passing to extractive distillation unit C for separation of a raffinate fraction withdrawn through line 16 and an extract fraction withdrawn through line 17 and containing cyclohexane of at least 95% purity.

From the foregoing description, it will be evident that the process of the present invention allows the processing of a mixture of closely boiling hydrocarbons in such a manner as to recover efficiently maximum amounts of high purity cyclohexane concentrates therefrom. In addition, other desirable materials such as benzene and n-hexane fractions are also recovered in the process of the present invention.

The invention claimed is:

1. A process for the preparation of high purity cyclohexane and high purity benzene and for the maximum recovery of both cyclohexane and benzene from a feed stock comprising n-hexane, benzene, methylcyclopentane, cyclohexane and dimethylpentanes which comprises the steps of passing the feed stock to a first extractive distillation zone and there separating the feed stock into an extract fraction containing substantially the whole of the benzene in the feed stock and into a raffinate fraction substantially free of benzene and containing the remainder of the feed components and removing said extract fraction as a first fraction from the system, admixing said raffinate fraction as a second fraction with a third fraction containing methylcyclopentane, cyclohexane, and heavier material, distilling the admixed second and third fractions in a first fractional distillation zone to separate it into a bottoms fraction and an overhead fraction, removing the bottoms fraction as a fourth fraction containing cyclohexane, removing the overhead as a fifth fraction containing methylcyclopentane, passing the fifth fraction to an isomerization zone and there converting at least a substantial portion of its methylcyclopentane content into cyclohexane, passing the effluent from said isomerization zone into a second fractional distillation zone and there separating it into an overhead fraction and a bottoms fraction, removing said overhead fraction as a sixth fraction containing n-hexane and lighter materials from the system, taking the bottoms fraction as said third fraction containing methylcyclopentane, cyclohexane and heavier material and admixing it with said second fraction as previously described and charging the fourth fraction as feed to a second extractive distillation zone and therein separating a raffinate fraction containing dimethylpentane and other paraffinic materials and an extract fraction containing high purity cyclohexane and separately removing said last raffinate and said last extract fraction from said second extractive distillation zone.

2. A process as described in claim 1 wherein the feed stock contains at least 5% by volume of benzene; the feed stock has a ratio of cyclohexane to benzene no greater than 3 to 1 by volume; the fourth fraction contains 80 to 95% cyclohexane; and the extract fraction from the second extractive distillation zone contains at least 95% cyclohexane.

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