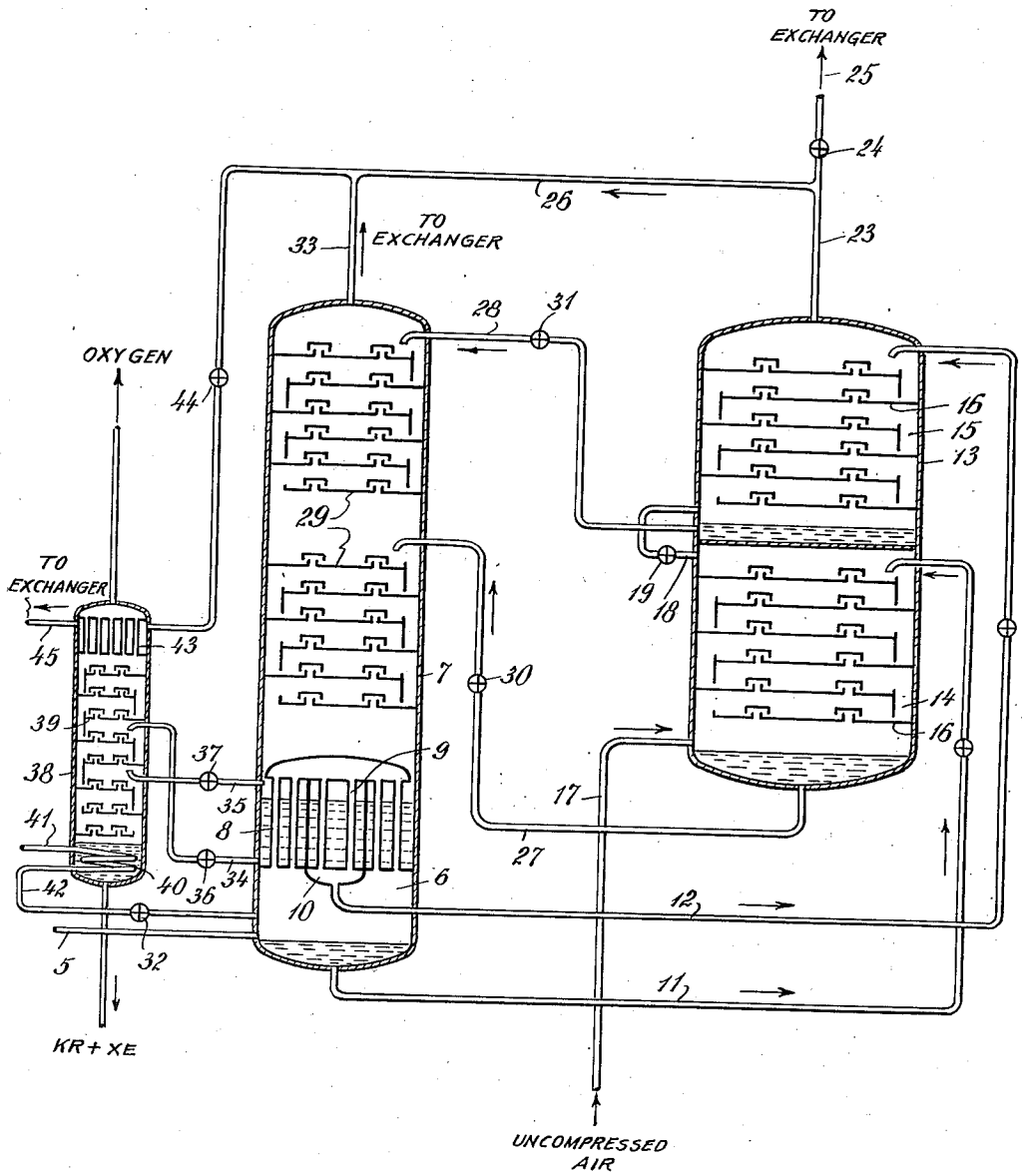
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RECOVERY OF KRYPTON AND XENON

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RECOVERY OF KRYPTON AND XENON

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This invention relates to the separation of the rare gases krypton and xenon from the atmosphere and the recovery of these gases in quantities such as to permit commercial applications thereof.

Air contains only minute proportions of krypton and xenon, and the ordinary methods of separating air for the recovery of nitrogen, oxygen and argon take no account of the rarer gases. No practicable method of recovering these gases has been available heretofore, and consequently the gases have been used only for experimental or scientific purposes.

It is the object of the present invention to provide a simple, practical and effective method of separating and recovering krypton and xenon from the atmosphere.

A further object of the invention is the provision of a method of treating relatively tremendous volumes of air at a cost which permits the recovery and use of krypton and xenon for various commercial and industrial purposes.

In carrying out the present invention, no attempt is made to recover all of the nitrogen, oxygen and argon as separate gases of substantial purity, since the primary function of the method is the recovery of the rarer gases krypton and xenon. Advantage is taken of the known methods of separating the nitrogen and oxygen constituents of the atmosphere, but following the primary separation of these constituents they are utilized for the purpose of concentrating krypton and xenon from additional quantities of air. The subsequent rectification of the liquids which ordinarily are rectified directly after initial separation produces a liquid containing considerably greater proportions of krypton and xenon than are usually present in the liquid product of the rectification. This liquid is subjected then to a further rectification to effect separation of oxygen, leaving a liquid concentrate rich in krypton and xenon which can be withdrawn and utilized. A portion of the oxygen and also of the nitrogen is also recoverable, but the major portion of all of the air treated is returned to the atmosphere as a gaseous mixture from which the krypton and xenon has been removed.

The method as thus generally outlined will be better understood by reference to the following specification and the accompanying diagrammatic drawing, illustrating an apparatus which is adapted for use in the practice of the method. It will be understood that details of the apparatus well known in the art are not illustrated or described, the purpose being to present simply and clearly the essentials of the invention as hereinafter claimed.

Referring to the drawing, a portion of the air to be treated, after initial compression to a suitable pressure and cooling in the usual exchangers, is introduced through a pipe 5 to the bottom compartment 6 of a column 7. The air passes upwardly through tubes 8 immersed in a liquid product accumulated as hereinafter described, and thence downwardly through tubes 9 into a collector 10. In passing through the tubes 8, the air is subjected to selective liquefaction with the resulting formation of a liquid in phase equilibrium with the composition of the air, that is to say, a liquid containing approximately from 30 to 40% of oxygen, the balance being nitrogen with some of the argon and a considerable proportion of the krypton and xenon of the air treated. The liquid flows backwardly and accumulates in the compartment 6.

The unliquefied residue, consisting largely of nitrogen, in passing downwardly through the tubes 9, is also liquefied and accumulates in the collector 10. This liquid also may contain some krypton and xenon as well as a portion of the argon.

The two liquids which, in an ordinary air liquefaction system, are introduced at separate levels in a rectification column to immediately effect a further separation of the constituents, are conducted instead through pipes 11 and 12 to a column 13 having separate compartments 14 and 15, each provided with the usual trays 16, over which the liquids flow in direct contact with relatively large volumes of air introduced through a pipe 17 under pressure sufficient only to ensure movement of the air through the apparatus. For the purpose of moving the air, an ordinary blower may be employed.

The air is introduced through the pipe 17 to the lower compartment 14 and thence through a pipe 18 and valves 19 and 20 to the upper compartment 15. The effect will be to wash from the large volume of entering air, by means of the liquids introduced through the pipes 11 and 12, substantially all of the krypton and xenon, together with some of the oxygen, while the effluent from the upper compartment 15, escaping through the pipe 23, will consist of a gaseous mixture, principally nitrogen, but containing oxygen and argon. The effluent may be sent to an exchanger through a valve 24 and pipe 25, or delivered through a pipe 26 for use as hereinafter described.

The two liquids, enriched in krypton and

xenon, which accumulate in the bottom of each of the compartments 14 and 15, are withdrawn through pipes 27 and 28, controlled by valves 30 and 31, and are delivered to separate levels of the column 7 in which the usual rectification trays 29 are disposed.

In flowing downwardly over the trays 29, the two liquids introduced to the column 7 are subjected to rectification with vapors arising from the body of liquid surrounding the tubes 8 and 9. This liquid is vaporized in the initial condensation of the entering air and the resulting vapors, together with those formed during the rectification, pass upwardly through the column 7 and escape as an effluent, consisting principally of nitrogen with some or all of the argon present in the liquid, through a pipe 33. The liquid flowing downwardly through the column finally accumulates about the tubes 8 and 9 and consists principally of oxygen with substantially all of the krypton and xenon which has been separated from the air entering through the pipes 5 and 17.

A portion of the liquid, together with vapors from the lower part of the column 7, is delivered through pipes 34 and 35 controlled by valves 36 and 37 to intermediate levels of an auxiliary column 38 containing the usual trays 39 and having at the bottom a coil 40 which is supplied with air previously compressed and cooled through a pipe 41. In traveling through the coil, the air causes vaporization of the liquid surrounding the coil and is itself liquefied and, it may be, somewhat subcooled. The liquid travels through the pipe 42 and valve 32 to the chamber 6, thus joining the liquid accumulating in that chamber. The vapor passes upwardly through the trays in contact with the downwardly flowing liquid and passes finally through tubes 43 of a condenser which is cooled by the effluent escaping from the column 7 through the pipe 33. A valve 44 controls the flow of the effluent which escapes finally through a pipe 45 to an exchanger. By utilizing the pipe 26 a portion of the effluent from the rectifier 13 may be similarly utilized. The condenser 43 separates from the vapors a liquid which becomes much concentrated in krypton and xenon when it reaches the bottom of rectifier 38. The effluent from this rectifier escapes through a pipe 46 and consists of substantially pure oxygen which may be delivered through an exchanger to a suitable collector in which the oxygen may be accumulated for use. The liquid at the bottom of the rectifier may be withdrawn through a pipe 47. It consists of a concentrate of krypton and xenon and is the principal product of the method as described. It may be utilized after further purification for any desired purpose.

From the foregoing description it is very evident that the process forming the subject matter of this application is an efficient and practicable method of extracting the krypton and xenon from large bodies of atmospheric air while at the same time an air separation apparatus for the production of high purity oxygen is operated in connection therewith. The relation between the air separation apparatus, which is of the well known Claude type, and the apparatus by means of which krypton and xenon are extracted from a large body of air not treated in the air separation apparatus proper, is such that an important advantage and economy in the air separation process itself is secured. This arises from the fact that the two liquids into which the air entering the Claude column is converted are enriched in oxygen by reason of their passage through the krypton-xenon extractors 9 and 15. That is to say, the two liquids entering the krypton-xenon extractors through pipes 7 and 13 respectively and re-entering the Claude rectifier through pipes 27 and 28 respectively both carry a higher percentage of oxygen after leaving the krypton-xenon extractors than when they enter. Of course, a consequence of this fact is that the effluent vapor leaving the top of the krypton-xenon extractors contains a higher percentage of nitrogen than atmospheric air.

The method, as will be evident, affords the possibility of treating large quantities of air and the recovery therefrom in a simple and satisfactory manner of the krypton and xenon content of the air, together with a separate portion consisting of oxygen in substantial purity. Thus, krypton and xenon in quantities enabling the use of these products for various commercial purposes are made available.

Various changes may be made in the details of the procedure and apparatus employed without departing from the invention or sacrificing any of the advantages thereof.

I claim:

1. The method of separating and recovering krypton and xenon from the atmosphere which comprises, separating the air initially into liquid fractions each enriched in one of the major constituents of air, washing volumes of air relatively large with respect to said liquid fractions with said liquid fractions to produce liquids enriched in krypton and xenon, rectifying the liquids with vapors produced therefrom and thereby accumulating a liquid containing substantially all of the krypton and xenon from the air treated.

2. The method of separating and recovering krypton and xenon from the atmosphere which comprises, separating the air initially into liquid fractions each enriched in one of the major constituents of air, washing volumes of air relatively large with respect to said liquid fractions with said liquid fractions to produce liquids enriched in krypton and xenon, rectifying the liquids with vapors produced therefrom and thereby accumulating a liquid containing substantially all of the krypton and xenon from the air treated, and subjecting the last mentioned liquid to further rectification with vapors produced therefrom to further concentrate the krypton and xenon by separating oxygen therefrom.

3. The method of separating and recovering oxygen, krypton and xenon from the atmosphere which comprises compressing a portion of the air to be treated and separating this portion into two liquid fractions, one of which is enriched in oxygen, krypton and xenon, and the other of which is enriched in nitrogen, washing volumes of uncondensed, substantially uncompressed cold air which are relatively large with respect to said liquid fractions in order to enrich both liquids in krypton, xenon and oxygen, and thereafter rectifying the liquid resulting from the washing of said air to produce a liquid containing substantially all the krypton and xenon contained in both the compressed and uncompressed air and substantially more oxygen than was contained in the primary compressed portion of the air.

4. The method of separating and recovering oxygen, krypton and xenon from the atmosphere which comprises compressing a portion of the air to be treated and separating this portion into two liquid fractions, one of which is enriched in oxy-

gen, krypton and xenon, and the other of which is enriched in nitrogen, washing volumes of uncondensed, substantially uncompressed cold air which are relatively large with respect to said liquid fractions in order to enrich both liquids in krypton, xenon and oxygen, thereafter rectifying the liquid resulting from the washing of said air to produce a liquid containing substantially all the krypton and xenon contained in both the compressed and uncompressed air and substantially more oxygen than was contained in the primary compressed portion of the air, and subjecting the last-mentioned liquid to further rectification with vapors produced therefrom to further concentrate the krypton and xenon and separate oxygen therefrom.

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