

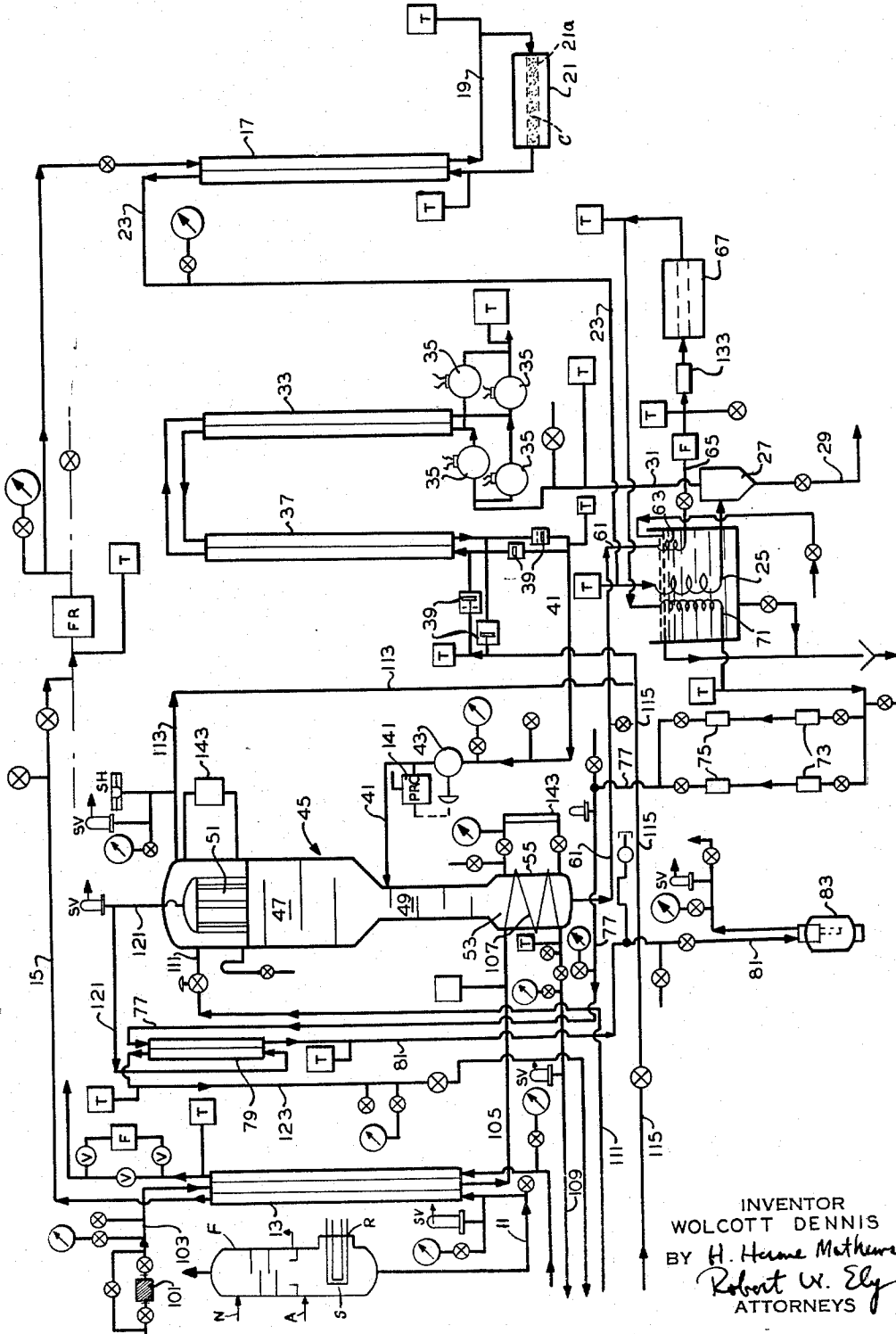
Dec. 6, 1960

W. DENNIS

2,962,868

METHOD OF CONCENTRATING KRYPTON-XENON

Filed Feb. 17, 1956



INVENTOR
WOLCOTT DENNIS
BY *H. Home Mathews*
Robert W. Ely
ATTORNEYS

1

2,962,868

METHOD OF CONCENTRATING KRYPTON-XENON

Wolcott Dennis, Union, N.J., assignor to Air Reduction Company, Incorporated

Filed Feb. 17, 1956, Ser. No. 566,308

13 Claims. (Cl. 62-22)

This invention relates to the separation of air for the purpose of obtaining a krypton-xenon fraction and more particularly concerns the production of a high-purity krypton-xenon fraction comprising 1% K-X in essentially liquid oxygen.

The letter symbols K, X, and O are sometimes used hereinafter for convenience in designating krypton, xenon and oxygen respectively. Similarly, the designation LQO is used for liquid oxygen. Use of these symbols together identifies mixtures thereof, such as K-X-O for a mixture of krypton, xenon and oxygen, K-X for a mixture of krypton and xenon, etc.

Much has been written and proposed recently concerning the excellent prospects for K-X in filling incandescent lamps and in other uses. It has been suggested that oxygen production will become a byproduct of the production of krypton or xenon or a mixture of the two.

The primary object of the present invention is to produce a high-purity K-X fraction in LQO. A further object is a method for the production of high-purity liquid oxygen and a crude K-X fraction which is converted to high-purity krypton and xenon. An additional object is a method for obtaining a 1% K-X in liquid oxygen (LQO) from a crude K-X fraction which contains about 100 parts per million (p.p.m.) of K-X in a purge liquid.

The preferred embodiment of the present invention accomplishes the above objects by warming a crude K-X stream of oxygen, hydrocarbons, krypton, xenon, argon, and nitrogen, passing said warmed stream through a device which converts the hydrocarbons (HC) to CO₂ and H₂O and consequently a desensitized K-X stream. Thereafter this desensitized stream is cooled to about 70° F. whereby the H₂O is separated and a substantially dewatered K-X stream results. This K-X stream is then passed through reversing heat exchangers so that the CO₂ and residual H₂O are deposited on the heat exchange surfaces and then flushed out by a suitable pick-up stream. Next, the so-treated K-X stream is fractionated with the assistance of certain relatively warm and cold fluids so that a liquid product containing about 1% K-X in LQO and an effluent of argon, oxygen, and nitrogen are obtained. Thereafter, it is preferred to vaporize the 1% K-X in essentially LQO and to remove any residual traces of hydrocarbons.

The accomplishment of the above objects and other objects, along with the features and advantages of the present invention, will be better understood by reference to the following description and the accompanying drawing of the preferred embodiment of the present invention.

It is to be understood that in the present application all references to temperature are by degrees (°) Fahrenheit and all references to pressure are by pounds (#) per square inch absolute (p.s.i.a.). The use of the letter "V" means vapor state and the use of the letter "L"

2

means liquid state. The flows (quantities before V or L) are standard cubic feet of gas per minute (s.c.f.m.).

For the sake of clarifying and simplifying the drawing, various well known components which are well understood to those skilled in the art have been shown schematically and identified in certain instances by descriptive symbols. Thus, in the drawings, the following symbols will be understood to designate conventional and commonly understood instrumentation and apparatus as follows: T designates a temperature gauge; SV identifies a safety valve; SH, a safety head; FR, a flow recorder instrument; V, a valve; PRC, a pressure regulator controller and F, a flow meter.

In the drawing, a liquid consisting essentially of 40% oxygen, 30 p.p.m. HC, 100 p.p.m. K-X and the remainder nitrogen and traces of argon with perhaps a 30-40 p.p.m. CO₂ flows in pipe 11 at the left-hand side of the drawing. This liquid is obtained from the air separation process disclosed in U.S. application S. N. 326,985 now U.S. Patent No. 2,762,208 by the inventor of the present invention. This copending patent application teaches the step of scrubbing incoming air with oxygen-enriched liquid air in a liquid oxygen producing plant so that a purge liquid or crude K-X stream constituting about 1% of the incoming air and consisting essentially of oxygen, argon, K-X, hydrocarbons, and nitrogen is obtained. The incoming air flow to the scrubber is, for example, about 5,284V+216L at about 94# and about -277°. This air is scrubbed in the scrubber portion of a nitrogen column containing about 10 plates by 316L of oxygen-enriched air and the liquid in the bottom of the scrubber section is boiled by a composite stream of nitrogen at -270° and 160#. Under the above-described flow conditions, a K-X purge of about 50-54L at -277° and 94# is continually removed. The reflux proportion is 316L (descending liquid) to 5762V (ascending vapors). In the drawing, a purge liquid containing a krypton-xenon fraction which is delivered in the line 11, is shown taken from the scrubber section S of a nitrogen column F of a conventional air rectification system, the incoming air stream to the column being shown at A. Liquid reflux for the nitrogen column may be supplied in any conventional manner such, for example, by introduction of liquid nitrogen at N and the scrubber section may be reboiled by circulating a nitrogen stream as above described through a reboiler shown at R, such operation of a scrubber being well understood to those skilled in the art. It is to be appreciated that, when a plant is built to produce LQO, there are special considerations involved if it is also desired to recover the p.p.m. of K-X.

The above-described K-X purge liquid containing about 100 p.p.m. K-X enters the apparatus shown on the accompanying drawing by means of conduit 11 (as above mentioned) and then passes through heat exchanger 13. In heat exchanger 13, this K-X purge is converted to vapors at 60° and 90#. From heat exchanger 13, the K-X stream moves through pipe 15 to exchanger 17 in which it is preferably further warmed to about 525°. This further warmed K-X stream at about 81# flows through pipe 19 to purge gas combustion furnace 21 wherein the hydrocarbons are converted to CO₂ and H₂O and a desensitized K-X stream is formed. This desensitized stream containing CO₂ and H₂O and perhaps about 1 p.p.m. HC in some cases then returns to heat exchanger 17 wherein it is cooled to 150° and leaves this exchanger at about 65#. The desensitized K-X stream then moves through pipe 23 to the water-cooled coil 25 wherein it is cooled to about 70°. From coil 25 the stream moves to a conventional water separator 27 wherein most of the water is separated and removed from the apparatus through outlet 29. A dewatered K-X

stream leaves the separator 27 through pipe 31 and moves to reversing heat exchangers 33 which are controlled by the four reversing valves 35. These valves 35 automatically provide for directing the dewatered K-X stream from pipe 31 alternately into one of the two passages of heat exchanger 33. From reversing exchanger 33 the dewatered K-X stream passes to a second section of reversing exchangers 37 having exit control valves 39. These valves 39, together with valves 35, complete the controls necessary to alternately pass for about fifteen minutes the K-X stream through one side of exchangers 33 and 37 while a pickup gas (55V at about -310°) is passing through the other sides of these exchangers and then for alternating these flow paths. In reversing exchangers 33 and 37, the dewatered K-X stream is sufficiently refrigerated so that any CO_2 therein and any residual traces of H_2O are deposited as solids in these heat exchangers. This results in a purified K-X stream at about -295° and 59#. This purified K-X stream with the valves positioned as shown in the drawing leaves the reversing heat exchangers through pipe 41 and flows through expansion valve 43 to the fractionating or rectification column 45. The purified K-X stream leaves expansion valve 43 at about 35#.

In the rectification column 45, a pressure of about 35# is maintained. Column 45 basically consists of a stripper section 47, lower rectification section 49, a reflux condenser 51 and a heater coil 53 in heat exchanger relation with the bowl 55 of the column. In column 45 a K-X stream of 50V containing oxygen, K-X, nitrogen and traces of argon is fractionated in such a manner that a liquid fraction is produced, containing about 1% K-X in LQO at about -280° and about 35# and constituting 1L. Essentially complete recovery of all of the K-X entering the apparatus is thus achieved. It will be recalled that the initial concentration of K-X in pipe 11 was about 100 p.p.m. while the final concentration is 10,000 p.p.m. The effluent from column 45 is at about -293° and 34#. The larger sized stripper section of column 45 contains 8 trays and the lower rectification section also contains 8 trays. The respective proportion of descending liquid to ascending vapors are 5 to 54 (upper) and 5 to 4 (lower). The liquid fed to the reflux condenser preferably is a fluid at about -315° . The fluid actually used is liquid nitrogen at about -315° and 20# being 5.8L+2V. The fluid which boils the liquid in the bowl 55 preferably is at -272° . The fluid which is actually used is a recycle nitrogen stream at -270° and 2500# and being 200V.

The K-X liquid withdrawn from the column 45 by means of pipe 61 preferably passes to water-warmed coil 63 because this liquid fraction in some cases may contain residual traces of hydrocarbons. Thus, it is greatly preferred that this liquid stream (1% K-X in LQO 1L, 50, 34#) as it exists after passing through coil 63 is passed through pipe 65 to a second combustion furnace 67. In combustion chamber 67, any residual trace of hydrocarbons is eliminated by conversion to H_2O and CO_2 . The so-treated K-X stream, as a vapor at 150° , is next cooled in water-cooled coil 71 to about 70° . Thereafter it passes alternately through product decarbonizers 73 and product driers 75 wherein the CO_2 and H_2O are removed. The decarbonized and dried, very purified, K-X stream is passed through pipe 77 to heat exchanger 79 wherein it is liquefied by heat exchange with a suitable fluid. The liquefied K-X fraction is made because in some plants there are no means for further separating the K-X and it is necessary to transport the 1% K-X to a separation facility. Since often this separation facility is some distance from the K-X producing plant, it is greatly preferred to ship it as a liquid. Thus, the liquid leaving heat exchanger 79 moves through pipe 81 to a conventional Dewar flask 83 which is then shipped to a distant K-X separation

plant. It is to be understood that the gaseous K-X fraction leaving combustion furnace 67 decarbonizers and driers can be separated into pure K-X locally. It is also to be understood that in many cases the final purification may not be required and that the product leaving column 45 would be the end K-X product like that collected in the flasks.

Referring now to the fluids which condition the crude K-X fraction which entered at pipe 11, it can be seen that heat exchanger 13 has two such fluids flowing in opposite directions therethrough. The fluid passing through the center path enters said exchanger 13 as nitrogen vapor (200V) at 70° and 2500# after passing through filter 101 for foreign matter removal in pipe 103. This nitrogen stream preferably is part of the closed recycle stream disclosed in the above-mentioned copending patent application. It leaves exchanger 13 at about -272° and flows through pipe 105 to coil 107 in heat exchange with the bowl of column 45. This nitrogen stream, after effecting the boiling of liquid in the bottom of column 45 leaves coil 107 through pipe 109 at about -270° and 160#.

The fluid which is supplied by pipe 111 to reflux condenser 51 preferably is liquid nitrogen (5.8L+0.2V) at about -315° and 20#. The vaporized nitrogen leaving reflux condenser 51 by means of pipe 113 is used after a slight pressure reduction by valve 39, along with waste nitrogen (55V) entering via pipe 115 as a pickup gas (at least about 10% more than the incoming K-X flow) for the reversing exchangers 33 and 37. This pickup gas sublimates the CO_2 and H_2O deposited in the reversing heat exchangers by the purified K-X stream. Thereafter this pickup stream is sent to waste or discharged to atmosphere. The waste gas (61V) moving through pipe 115 is at about -310° and 17#.

The other fluid used in the process, other than the water used to cool coils 25, 63, and 71, is the fluid used to liquefy the very purified K-X fraction in heat exchanger 79. This fluid is effluent which leaves the top of reflux condenser 51 through pipe 121 and is comprised of nitrogen, oxygen, and argon. This stream (49V) in pipe 121 is at about -293° and 34#. After liquefying the very purified K-X stream in exchanger 79, this effluent stream moves through pipe 123 as purged gas residue. This purged gas (49V) residue is at about -278° and 28#.

The combustion furnace 21 preferably contains a palladium on alumina catalyst such as disclosed in the Rosenblatt U.S. Patent No. 2,582,885, which may, for example, be in granular form such as is indicated by the reference C designating a bed of the catalyst packed in a reactor tube section 21a of the furnace. It is heated to 600° - 700° and the gas leaves at about 600° . In some instances, it may be desirable to use an Inconel tube (78% Ni, 7% Fe and 14% Cr). This catalyst tube preferably is heated to about 1500° - 1600° . The gas would enter at about 1425° and leave at about 1500° . A silver on alumina catalyst operated at about 1300° has also proved effective. Combustion furnace 67 preferably contains the above-mentioned palladium on alumina catalyst and preferably is preceded by a conventional flash arrester 133 to prevent blow-back to the column. Product decarbonizers 73 preferably contain soda lime (8 x 16 mesh). Product driers 75 preferably contain silica gel.

It is to be understood that the gas which may escape during the filling or storage of Dewar containers 83 can be vented to atmosphere or saved in a gas holder. It is also to be understood that pipe 81 could discharge into a large vacuum powder storage container or the like or into a railway tank car or tractor trailer having a liquefied gas container, all preferably refrigerated. Expansion valve 43 is automatically controlled by conventional air-operated pressure regulating control 141. Two

liquid level controls 143 are shown operatively connected to the reflux condenser 51 and the column bowl 55.

The operation of the apparatus and the steps of the process are believed to be apparent, to those skilled in the art, from the foregoing description. Some of the noteworthy features which can be again mentioned are the arrangement for purifying the K-X stream after it passes through the purged gas combustion furnace by means of the reversing exchangers in which the 10% greater nitrogen flow for picking up deposited CO₂ is provided; another such feature is the purification of the 1% K-X-O stream by means of a combustion furnace and then the second liquefaction of the highly purified stream; of note also is the particular manner in which the rectification column is operated since it is refluxed by a fluid at -315° and is suitably boiled by an external fluid and it has the very efficient reflux proportions between descending liquids and ascending vapors. The agents attributable to the above-mentioned features are believed to be obvious, for example, an efficient primary purification step is provided, a substantially 100% recovery of K-X is provided, and a continual purge of the column and purification of the final 1% K-X in oxygen is also provided.

It is to be understood that the person skilled in the art can make changes in the above-described preferred embodiment without departing from the invention as defined in the following claims.

I claim:

1. A method of concentrating krypton and xenon in oxygen comprising warming a crude krypton-xenon stream of oxygen, hydrocarbons, krypton, xenon, argon, and nitrogen, passing said warmed stream through a combustion furnace containing a palladium catalyst at about 600° F. to 700° F. whereby hydrocarbons are converted to CO₂ and H₂O and a desensitized krypton-xenon stream is formed, removing said CO₂ and H₂O from said desensitized krypton-xenon stream by cooling so that said CO₂ and H₂O are deposited as solids whereby a purified krypton-xenon stream is formed, fractionating said purified krypton-xenon stream by refluxing with the aid of a fluid at about -315° F. and by boiling with the aid of a fluid at about -272° F. whereby said purified krypton-xenon stream is separated into a liquid fraction comprised of krypton-xenon in liquid oxygen.

2. The method according to claim 1 and further including the step of converting said liquid fraction into a gas and passing it through a combustion furnace so residual traces of hydrocarbons are removed and a very purified krypton-xenon-oxygen stream results.

3. A method of concentrating krypton and xenon in liquid oxygen comprising warming a crude liquid krypton-xenon stream of oxygen, hydrocarbons, krypton, xenon, argon, and nitrogen at about 90 p.s.i.a. to about 60° F., further warming said stream to about 525° F., passing said further warmed stream through a combustion furnace containing a palladium alumina catalyst at about 600° F.-700° F. whereby hydrocarbons are converted to CO₂ and H₂O and a desensitized krypton-xenon stream is formed, cooling said desensitized krypton-xenon stream to about +70° F. and separating H₂O therefrom to form a dewatered krypton-xenon stream, removing said CO₂ and H₂O from said dewatered krypton-xenon stream by cooling so that said CO₂ and H₂O are deposited as solids whereby a purified krypton-xenon stream at about -295° F. is formed, fractionating said purified krypton-xenon stream at about 35 p.s.i.a. by refluxing with the aid of a fluid at about -315° F. and about 20 p.s.i.a. and by boiling with the aid of another fluid at about -272° F. whereby said purified krypton-xenon stream is separated into a liquid fraction comprised of 1% krypton-xenon in liquid oxygen at about -281° F.

4. The method according to claim 3 and further including the steps of converting said liquid fraction into a gas and passing it through a combustion furnace so

residual traces of hydrocarbons are removed and a very purified krypton-xenon-oxygen stream results, and then liquefying said krypton-xenon-oxygen stream by refrigeration derived from the effluent from said fractionating step.

5. A method of concentrating krypton and xenon in oxygen comprising warming a crude krypton-xenon stream of oxygen, hydrocarbons, krypton, xenon, argon, and nitrogen, passing said warmed stream through a combustion furnace whereby hydrocarbons are converted to CO₂ and H₂O and a desensitized krypton-xenon stream is formed, cooling said desensitized krypton-xenon stream to about 70° F. and separating H₂O therefrom to form a dewatered krypton-xenon stream, removing said CO₂ and residual H₂O from said dewatered krypton-xenon stream by cooling in such a manner that said CO₂ and H₂O are deposited as solids whereby a purified krypton-xenon stream is formed, fractionating said purified krypton-xenon stream by refluxing with the aid of a fluid at about -315° F. and by boiling with the aid of a fluid at about -272° F. whereby said purified krypton-xenon stream is separated into a liquid fraction comprised of krypton-xenon in liquid oxygen, and converting said liquid fraction into a gas and passing it through a combustion furnace so that residual traces, if any, of hydrocarbons are removed and a very purified krypton-xenon-oxygen stream results.

6. A method of concentrating krypton and xenon in liquid oxygen comprising warming a crude liquid krypton-xenon stream of oxygen, hydrocarbons, krypton, xenon, argon, and nitrogen at about 90 p.s.i.a., passing said warmed stream into contact with a heated catalyst whereby hydrocarbons are converted to CO₂ and H₂O and a desensitized krypton-xenon stream is formed, removing said CO₂ and H₂O from said desensitized krypton-xenon stream by cooling in reversing heat exchangers on about fifteen-minute cycle so that said CO₂ and H₂O are deposited as solids whereby a purified krypton-xenon stream at about -295° F. is formed, fractionating said purified krypton-xenon stream into a liquid fraction containing about 1% krypton-xenon in liquid oxygen and an effluent containing all of the nitrogen of said crude krypton-xenon stream, said fractionation being done in an upper zone in which the proportion of descending liquid to ascending vapors is about 5 to 54 and the reflux fluid temperature is about -315° F. and in a lower zone in which the proportion of descending liquid to ascending vapors is 5 to 4 and the boiling fluid temperature is -272° F.

7. The method of fractionating air to obtain a high purity liquid oxygen and an enriched, substantially pure krypton-xenon-oxygen mixture in the order of about 1% of said krypton-xenon in oxygen, as a by-product, which comprises scrubbing the incoming air to be fractionated with liquefied oxygen-enriched air, to form a gaseous fraction and a crude krypton-xenon liquid fraction amounting to about 1% of the incoming air and containing about 0.01% concentration of krypton-xenon together with oxygen, nitrogen, argon and hydrocarbons, separating said crude liquid fraction from said gaseous fraction, warming and passing said crude liquid fraction through a combustion furnace containing a palladium catalyst at about 600° F. to 700° F. to convert the hydrocarbons to CO₂ and H₂O and form a desensitized krypton-xenon bearing stream, removing said CO₂ and H₂O from said desensitized krypton-xenon stream whereby a purified krypton-xenon bearing stream is formed, fractionating said purified krypton-xenon bearing stream, and removing the desired high purity krypton-xenon enriched product as a liquid from said fractionation.

8. A method of concentrating krypton and xenon in oxygen comprising warming a crude krypton-xenon stream containing oxygen, hydrocarbons, krypton, xenon, argon and nitrogen, passing said warmed, crude krypton-xenon stream through a catalytic furnace reactor where-

in said hydrocarbons are converted to CO_2 and H_2O to form a desensitized, crude, krypton-xenon stream, removing the H_2O and CO_2 formed in said furnace from said desensitized stream, cooling said desensitized stream by heat exchange with a relatively cold nitrogen vapor stream, delivering said cooled, desensitized krypton-xenon stream to a rectification column wherein said desensitized stream is separated into a gaseous fraction substantially free of krypton and xenon which is removed at the top of said column and a substantially pure krypton-xenon-oxygen liquid fraction which is collected at the bottom of said column, condensing vapor to produce liquid reflux at the top of said column by indirect heat exchange with liquid nitrogen and delivering said liquid nitrogen which is vaporized in furnishing said reflux liquid for use in cooling said desensitized krypton-xenon stream.

9. A method of concentrating krypton and xenon in oxygen according to claim 8 wherein said substantially pure krypton-xenon-oxygen liquid fraction is warmed, passed through a catalytic reactor furnace to convert any residual hydrocarbons to water and carbon dioxide, removing the carbon dioxide and water so formed and cooling the thus further purified krypton-xenon stream by heat exchange with the gaseous fraction separated in said rectification column and effecting the liquefaction of said further purified stream.

10. A method of concentrating krypton and xenon in oxygen according to claim 8 wherein said crude krypton-xenon stream is warmed by heat exchange with a relatively warm nitrogen vapor stream and said relatively warm nitrogen vapor stream is then passed through reboiler coil means at the bottom of said rectification column to produce reflux vapor therein.

11. A method of concentrating krypton and xenon in oxygen comprising warming a crude krypton-xenon stream of oxygen, hydrocarbon, krypton, xenon, argon and nitrogen, passing said warmed, crude krypton-xenon stream through a catalytic reactor furnace wherein said hydrocarbons are converted to CO_2 and H_2O , to form a desensitized, crude krypton-xenon stream, passing said desensitized stream through a flow passage of a reversing exchanger, cooled by a relatively cold nitrogen vapor stream in an adjacent flow passage, to cool said desensitized stream and remove the H_2O and CO_2 formed in said furnace as solidified deposits, delivering the resulting cooled, desensitized krypton-xenon stream to a rectification column, wherein said desensitized stream is separated to form a substantially pure krypton-xenon-oxygen liquid fraction and a gaseous fraction substantially free of krypton and xenon, producing liquid reflux for said column by indirectly condensing a portion of the vapors at the top of said column with liquid nitrogen, delivering the nitrogen vapors resulting therefrom to said reversing exchanger for use in cooling said desensitized krypton-xenon stream and periodically reversing the flow passages of said desensitized krypton-xenon stream and said nitrogen vapor stream to remove solids deposited in said exchanger from said desensitized krypton-xenon stream.

12. The method of concentrating krypton and xenon in oxygen comprising warming a crude krypton-xenon stream containing, along with the krypton and xenon constituent, amounts of oxygen and hydrocarbons, passing said warm stream through a combustion furnace containing a palladium catalyst at about 600°F . to 700°F .

whereby the hydrocarbon constituent is converted to CO_2 and H_2O and a desensitized krypton-xenon stream is formed, passing said desensitized krypton-xenon stream through a reversing exchanger in counterflow cooling relation with a stream of cold nitrogen vapor so as to effectively deposit the carbon dioxide and H_2O content of said desensitized stream as solids in said exchanger, said cold nitrogen vapor being derived from a gaseous effluent separated from the liquid fraction of a partial condensation of air, intermittently reversing and alternating the flows of said desensitized krypton-xenon stream and said cold nitrogen vapor in said reversing exchanger and delivering said nitrogen vapor in an amount which is at least about 10% larger than the flow of said desensitized stream so as to effectively periodically purge the deposited carbon dioxide and water solids, at least partially liquefying and fractionating said purified krypton-xenon stream derived from said reversing exchanger and obtaining therefrom an enriched liquid fraction containing krypton and xenon in liquid oxygen.

13. The method of fractionating air to obtain a high purity liquid oxygen and an enriched, substantially pure krypton-xenon-oxygen mixture as a by-product, which comprises providing a separate nitrogen recycle for producing refrigeration for the air rectification, cooling an air stream to be rectified by indirect heat exchange with said recycle nitrogen, scrubbing the cool air stream with liquefied oxygen enriched air to form a gaseous fraction which is subsequently rectified to produce separate oxygen and nitrogen rectification products and a crude krypton-xenon liquid fraction amounting to about 1% of the incoming air and containing about 0.01% concentration of krypton-xenon together with oxygen, nitrogen, argon and hydrocarbons, separating said crude liquid fraction from said gaseous fraction, warming and passing said crude liquid fraction through a combustion furnace to convert the hydrocarbons to CO_2 and H_2O and form a desensitized krypton-xenon stream, removing said CO_2 and H_2O from said desensitized krypton-xenon stream to form a purified krypton-xenon stream, rectifying said purified krypton-xenon stream in a rectification column wherein said stream is at least partially liquefied, vaporizing at least a portion of the liquid collected at the bottom of said column by heat exchange with a relatively warm stream of said recycle nitrogen and removing the desired high purity krypton-xenon enriched product as a liquid from said rectification column.

References Cited in the file of this patent

UNITED STATES PATENTS

2,007,116	Walker	July 2, 1935
2,032,925	Ferguson	Mar. 3, 1936
2,060,940	Kahle	Nov. 17, 1936
2,433,536	Van Nuys	Dec. 30, 1947
2,527,623	Fausek et al.	Oct. 31, 1950
2,582,885	Rosenblatt	Jan. 15, 1952
2,650,482	Lobo	Sept. 1, 1953
2,664,719	Rice	Jan. 5, 1954
2,688,238	Schilling	Sept. 7, 1954
2,793,507	Hnilicka	May 28, 1957
2,793,511	Bonnaud	May 28, 1957

OTHER REFERENCES

"The Separation of Gases," second edition (Ruhemann), published by Oxford University Press (London), 1949, pages 86-88, 183-185, and 228-236 are relied on.