

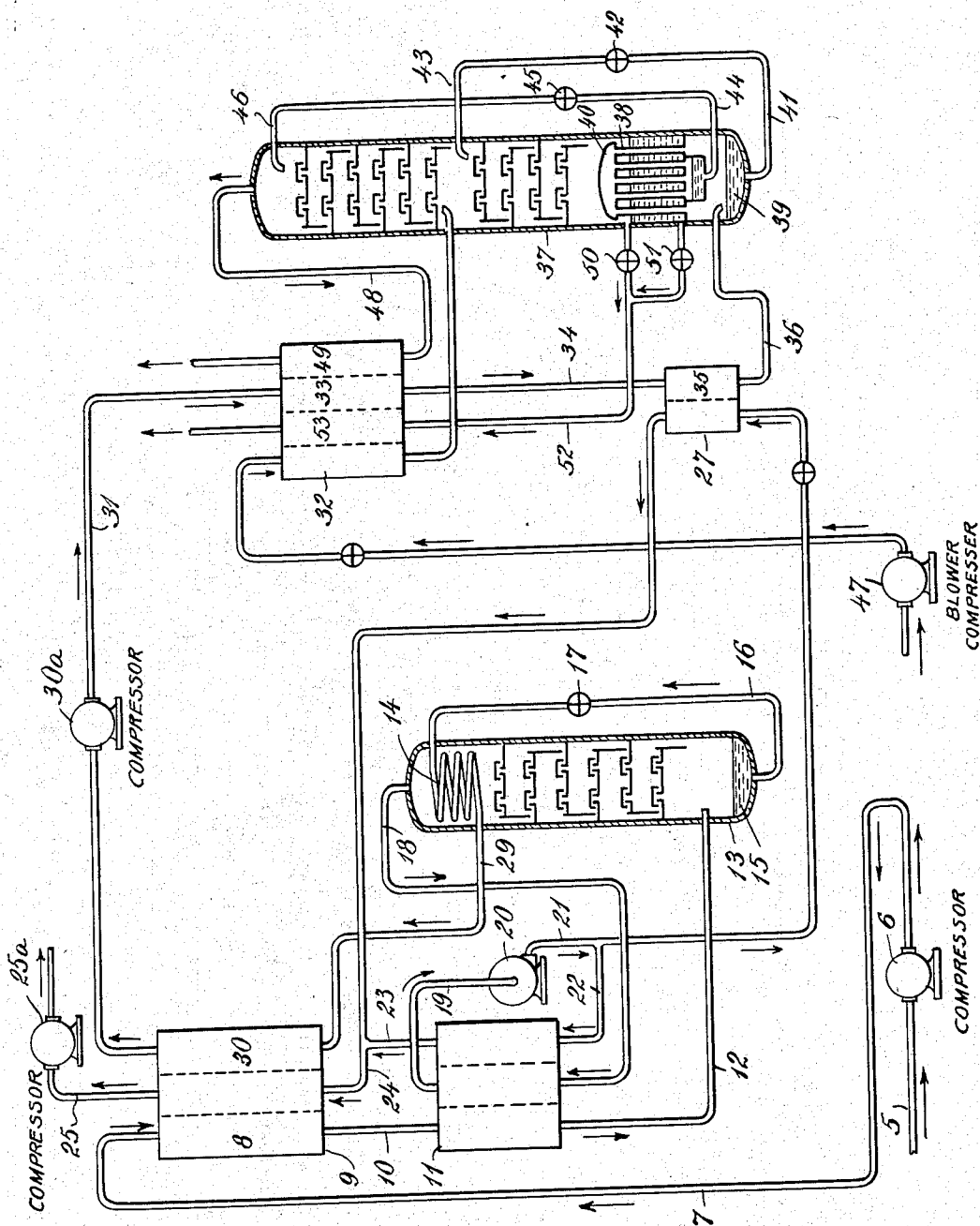
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J. L. SCHLITT

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38. J. E. SCHULTZ
METHOD FOR THE SEPARATION AND RECOVERY OF KRYPTON
AND XENON FROM GASEOUS MIXTURES CONTAINING THEM
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INVENTOR

Joseph L Schlitt

BY

BY
Pennie Davis Maurin & Edwards
ATTORNEYS

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METHOD FOR THE SEPARATION AND RECOVERY OF KRYPTON AND XENON FROM GASEOUS MIXTURES CONTAINING THEM

Joseph L. Schlitt, Darien, Conn., assignor to Air Reduction Company, Incorporated, New York, N. Y., a corporation of New York

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2 Claims. (Cl. 62—175.5)

The invention relates to the economical separation of the gases krypton and xenon from mixtures containing them such as atmospheric air or atmospheric residual gases from which the components oxygen and nitrogen have been more or less completely removed previously.

The present method is not intended to recover either of the two atmospheric gases oxygen or nitrogen in commercial purity from the atmospheric air or other mixture treated, but is directed to an economical method of recovering the elements krypton and xenon in a state of purity suitable for further purification by chemical or adsorption methods.

The method is characterized by the important feature that only a relatively small fraction of the total air treated is liquefied. The major portion of the gaseous mixture passes through the cycle unliquefied and this feature makes possible the operation of the method at relatively low cost. The krypton and xenon are caused to concentrate in that small portion of the original mixture which is condensed and the loss of these gases in the major uncondensed portion is practically nil.

The unliquefied gaseous portion is composed of both oxygen and nitrogen, the relative percentage of nitrogen and oxygen being somewhat increased as the result of the liquefaction of the condensed portion. Nowhere in the method, however, is oxygen produced in a purity suitable for any of the industrial applications existing today.

The liquefied portion, that is the krypton-xenon concentrate, is further treated in order to obtain the krypton and xenon with a purity suitable for further purification treatment by well known methods. This is accomplished by means of re-evaporation of the krypton-xenon liquid concentrate followed by a moderate compression and then by means of selective liquefaction and rectification, most of the impurities oxygen and nitrogen are separated from the krypton-xenon as hereinafter described.

The details of the method may be more readily understood by reference to the accompanying diagrammatic drawing, which illustrates an apparatus suitable for the practice of the invention. Details of apparatus well known in the art are omitted for the purpose of clarity.

The air or other gaseous mixture containing krypton and xenon is delivered by a pipe 5 to a compressor or blower 6 and after traversing pipe 7 enters compartment 8 of an exchanger 9 where in it is cooled by thermal contact with outgoing

products. The gaseous mixture then passes through a pipe 10 to an exchanger 11 wherein it is still further cooled to a temperature in the neighborhood of its dew point. A pipe 12 delivers the gaseous mixture to the selective condenser 13 in which it passes upwardly and is partially liquefied by thermal contact with the cold fluid traversing coil 14 at the top level of condenser 13. This coil condenses a portion of the rising vapors which collect in a pool 15 at the bottom of condenser 13.

The liquid leaves pool 15 by means of pipe 16 and passes through a pressure reducing valve 17 wherein its pressure (and temperature) is lowered sufficiently for use as condensing liquid in coil 14.

The uncondensed vapors leaving condenser 13 through pipe 18 traverse exchanger 11 and by means of pipe 19 enter the turbo-expander 20 in which the temperature is again reduced substantially by reason of its expansion with external work in turbo-expander 20. From the latter the working fluid leaves through pipe 21 and the major portion passes outward through exchangers 11 and 9 by means of pipes 22, 23, 24 and 25.

A portion of the exhaust from turbo-expander 20 is diverted through pipe 26 to the exchanger 27 for reasons hereinafter described. Leaving exchanger 27 by means of pipe 28 it joins that portion of the exhaust passing through exchanger 11 in pipe 24.

The pressure necessary to be produced in compressor 6 is determined primarily by the necessity of the vapors rising in condenser 13 to be partially condensed by means of the lower pressure liquid circulating through coil 14. Since the quantity of fluid liquefied by coil 14 is only a small portion of the total, the composition of the vapor rising to the top of condenser 13 and leaving by means of pipe 18 is not greatly different from that of atmospheric air as far as oxygen and nitrogen are concerned and thus the pressure necessary to accomplish the condensation in condenser 13 is relatively small, for example, from 1 to 2 atmospheres gauge, assuming that the pressure in condenser-rectifier 13 is 1 atmosphere.

The turbo-expander 20 affords the refrigeration necessary in the system in order to overcome external heat leakage into the apparatus. The refrigerative effect produced by expander 20 may be increased either by an increase of pressure in the fluid leaving compressor 6 or, as an alternative, by reducing the pressure in the ex-

haust of expander 20 by means of a vacuum pump 25a attached to pipe 25.

The krypton-xenon concentrate evaporated in coil 14 passes through pipe 29 to compartment 30 of exchanger 9 wherein its temperature is brought up to that of atmospheric by thermal contact with the incoming mixture. It is then compressed in turbo-compressor 30a to a pressure suitable for its subsequent liquefaction as hereinafter described.

After compression, the krypton-xenon concentrate enters, by pipe 31, an exchanger 32 traversing compartment 33 and thence by means of pipe 34 it enters the exchanger 27 traversing the compartment 35 wherein its temperature is reduced to the vicinity of the dew point. Thence by means of a pipe 36, it enters the bottom of the rectification-condensation column 37 and passes upward through the tubular system 32 wherein it is selectively liquefied. The liquefied portion collects in pool 39 while the unliquefied portion enters the inner tubular system 40 and is therein completely liquefied, to provide a second liquefied portion.

The first liquefied portion leaves the pool 39 by a pipe 41 and passes through the pressure reducing valve 42 wherein its pressure is lowered to that prevailing in the interior of rectifier column 37 which it enters at an intermediate level by means of a pipe 43. The second liquefied portion condensed in the tubes 40 passes through pipe 44 and pressure reducing valve 45 and thence by means of a pipe 46 as top reflux liquid into the top level of the rectifier column 37.

Since the vapor entering tubular system 38 is an uncondensed residue of the mixture entering through the pipe 36, it will be practically free from krypton and xenon and after its condensation in tubes 38, it will be able to liquefy out the krypton and xenon from a large volume of vapor containing these elements. This volume of vapor may be many times that of the vapor ascending in rectifier 37 from the bottom. For this reason, it is very advantageous to admit to an intermediate level of rectifier 37 a substantial quantity of unseparated air which is delivered to the system by the turbo-blower 47. The pressure necessary to be delivered by blower 47, is only that required to cover pipe friction to and from exchanger 32 and rectifier 37.

The feature just described is responsible for an important economy in the system; and together with the advantages and economies already pointed out, renders this method by far the most efficient for the recovery of krypton and xenon from mixtures containing them.

The rectifier column 37 separates the incoming mixtures entering it through pipe 36 into two products, (1) the top gaseous product consisting only of oxygen and nitrogen plus small percentages of neon, helium and argon and (2) the bottom product which constitutes the final krypton-xenon concentrate. Product (1) leaves the top of rectifier column 37 by means of pipe 48 and passes outward through compartment 49 of exchanger 32. Product (2) constituting the krypton-xenon concentrate leaves rectifier col-

umn 37 either in the vapor or liquid state by means of pipe and valves 50 and 51, and traverses exchanger 32 by means of pipe 52 and compartment 53.

In the operation of the method, the pressure in coil 14 may be lowered, if desirable, to a sub-atmospheric pressure by means of turbo-compressor 30a as already described. In this case, the pressure necessary in condenser-rectifier 13, i. e., the pressure delivered by compressor 6, may be lowered correspondingly. In either case, however, the pressure differential between condenser-rectifier 13 and coil 14 is much smaller than for any air separation process because the vapor to be condensed by coil 14 contains substantial quantities of oxygen and thus will condense more easily than pure nitrogen, while the liquid passing through coil 14 which produces this condensation contains substantial quantities of nitrogen (around 50%) and thus is much colder at any given pressure than would be pure oxygen at the same pressure. An analogous statement may be made in regard to the liquefaction and evaporation in the tubular systems 38 and 40.

The present method, it is now evident, has as its most important feature the employment of very low pressures relative to the pressures commonly used in the separation of air into its constituent gases. In fact, every pressure produced in the method may be easily attained by means of turbo-compressors. The economy of the method is also increased by reason of the fact that only a very small fraction of the mixture treated is necessary to be liquefied.

Various changes may be made in the procedure and in the details of the 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 air which comprises liquefying selectively a portion of the air to be treated to secure a liquid enriched in krypton and xenon, evaporating the liquid to effect by heat exchange the selective liquefaction of the incoming air, recompressing and cooling the vapor from the enriched liquid, subjecting the vapor to further selective liquefaction, rectifying the liquid products with the addition of a relatively large volume of unseparated air, and withdrawing the liquid product of the rectification consisting essentially of krypton and xenon.

2. The method of separating and recovering krypton and xenon from air which comprises liquefying selectively a portion of the air to be treated to secure a liquid enriched in krypton and xenon, evaporating the liquid at subatmospheric pressure to effect by heat exchange the selective liquefaction of the incoming air, recompressing and cooling the vapor from the enriched liquid, subjecting the vapor to further selective liquefaction, rectifying the liquid products with the addition of a relatively large volume of unseparated air, and withdrawing the liquid product of the rectification consisting essentially of krypton and xenon.

JOSEPH L. SCHLITT.