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(54) METHOD AND APPARATUS FOR CONTROLLED CONDENSATION ISOTOPE SEPARATION

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Abstract of the Disclosure

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Supersonic flow of a molecular gas under the appropriate conditions through an expansion nozzle, produces homogenous condensation of the gas. If the gas is vibrationally excited, the condensation is inhibited. Thus, selective vibrational excitation of an isotopic species in the gas provides a means of controlling the condensation of the gas. Such selective vibrational excitation may be readily accomplished by means of laser irradiation of the supersonically flowing gas. The controlled condensation in turn may be used as the basis of isotope separation and enrichment by subjecting the stream of gas and condensate particles to aerodynamic processes whereby the condensate particles are separated from the main flow of the gas.

METHOD AND APPARATUS FOR CONTROLLED CONDENSATION ISOTOPE SEPARATION

The invention described herein relates to methods and apparatus for achieving controlled homogenous condensation and to the use of such controlled homogenous condensation in aerodynamic isotope separation or enrichment.

Aerodynamic isotope separation processes may conveniently be characterized as those involving diffusion of disparate masses, normally in a binary or ternary gas mixture, driven principally by a pressure gradient generated by geometry effects. Such processes may be either equilibrium or nonequilibrium, with flow rarefaction levels varying from near free molecular, i.e., almost collisionless, to near continuum. Techniques for aerodynamic isotope separation include: (1) the curved separation nozzle process, (2) the opposing jet process which is a variant of the curved nozzle, (3) the supersonic vortex tube process, (4) the separation probe process, (5) the jet membrane process, (6) the crossed beam process, and (7) the velocity slip process. For a discussion of these processes, see R. Compargue et al., "On Aerodynamic Separation Methods," in Nuclear Energy Maturity, edited by Pierre Zaleski (Pergamon Press, 1975).

It will be apparent that in any aerodynamic process it is desirable to maximize the mass difference between the fraction carrying the lighter isotopic species and the fraction carrying the heavier isotopic species, since this serves to increase the efficiency of separation in any single state. In all aerodynamic processes taught in the prior art the feed gas remains gaseous throughout the separation so that the only mass difference involved is that of the isotopic species themselves. If, however, the mole fraction of the feed gas containing a particular isotopic species can be made to condense while the other isotopic species remains a gas, then the diffusion of the two species away from each other may be greatly enhanced.

Because of the inability in prior art aerodynamic processes to control condensation so that it is at least preferential for a particular isotopic species, condensation has been considered to be a problem to be avoided. It is known, however, that the condensation probability of a molecule is a function of its vibrational state. In "Condensation of Vibrationally Excited Gas," 22 JETP Letters 302 (1975), Basov et al. demonstrated that the isotopes of carbon can be separated through nonselective excitation of CO_2 molecules followed by heterogeneous condensation on the walls of the containing tube. The explanation for the separation effect set forth by the Russian authors is that the $^{13}CO_2$ molecule acquires a larger total vibrational energy than the $^{12}CO_2$ molecule. This seems reasonable since the vibrational energy states of $^{13}CO_2$ are more closely spaced than those of the $^{12}CO_2$. Thus, in collisions at low temperature where the molecular kinetic energy is less than the difference in vibrational energy spacing between the molecules, the most probable result is an exchange of vibrational energy from the light to the heavy molecules. The authors suggest that the excess of vibrational energy in the heavier molecule appears as a local point source of heat when the molecule strikes the wall, thereby reducing its sticking probability and delaying its condensation.

A more recent Russian article extends the work of Basov et al. and shows boron isotope separation through selective excitation with a CO_2 laser. See Gochelashvili et al., "Methods for Selective Heterogeneous Separation of Vibrationally Excited Molecules," 43 Sov. Phys.-JEPT 274 (1976). The separation was achieved by adsorbing $BC\ell_3$ on the wall of a stainless steel tube that was cooled to 160 K. The inside of the tube was irradiated by the CO_2 laser.

Both of these demonstrated isotope separations depend on the heterogeneous reaction of the subject molecule with a cooled wall. Collisions with the wall result in either preferential condensation or adsorption of the molecular species with the lowest vibrational energy.

Summary of the Invention

We have found that homogenous condensation of molecular gases can be controlled such that in a molecular feed gas containing a mixture of isotopes, condensation can be inhibited with respect to those molecules containing only a particular isotopic species under conditions at which all other molecules of the feed gas tend to condense. The inhibiting effect is produced by selectively vibrationally exciting those molecules containing the particular isotopic species. This controlled condensation may be made the basis of an isotope separation technique by applying any of a variety of aerodynamic processes to a flowing stream of condensate particles and feed gas to separate the condensate particles from the gas.

In its broad sense, our invention encompasses a method for producing controlled homogenous condensation of a molecular feed gas containing a plurality of isotopes which comprises (a) supersonically flowing the feed gas through an expansion nozzle under flow conditions at which the expansion of the supersonically flowing feed gas produces condensation of the gas when the gas is not vibrationally excited, and (b) irradiating the supersonically flowing gas with laser radiation of a wavelength which selectively excites those molecules in the feed gas containing a particular isotope. This method for producing controlled homogenous condensation may readily be used as the basis for an isotope separation technique by merely incorporating the additional step of aerodynamically separating condensate particles from the flowing gas stream.

The invention also comprehends an apparatus for producing controlled homogenous condensation of a molecular feed gas containing a plurality of isotopes. The apparatus includes (a) a supersonic nozzle, the nozzle producing condensation in the feed gas when the gas is flowed supersonically through the nozzle and the gas is not vibrationally excited, (b) means for producing supersonic flow conditions in the nozzle whereby condensation of the molecular feed gas occurs when the gas is not vibrationally excited, and (c) means for irradiating the supersonically flowing gas stream with laser radiation of a wavelength which selectively excites those molecules in the feed gas containing a particular isotope.

The invention is generic in nature and is applicable to the separation of a wide variety of elemental isotopes. It is particularly useful for the separation of uranium isotopes using UF_6 as the molecular feed gas.

Brief Description of the Drawings

Figure 1 is a schematic flow chart of controlled condensation aerodynamic isotope separation or enrichment in accordance with the present invention.

Figure 2 is a schematic cross section of a curved supersonic nozzle useful in controlled condensation aerodynamic isotope separation.

Figure 3 is a schematic cross section of apparatus for controlled condensation aerodynamic isotope seaparation using a free jet expansion.

Figure 4 is a schematic cross section of apparatus for controlled condensation aerodynamic isotope separation using a probe.

Figure 5 shows schematically an experimental setup used to demonstrate the effect of CO_2 laser radiation on SF_6 condensation.

Figure 6 shows a plot of absorbance versus starting pressure for two separate SF_6 mixtures.

Figure 7 shows a plot of absorbance as a function of distance from the nozzle throat for a feed gas consisting of 2% SF_6 in He.

Description of the Preferred Embodiments

The initial stage of formation of a condensation nucleus includes the formation of dimers, trimers, and higher order polymers. The presence of vibrational excitation serves to inhibit the formation of the dimers and trimers of a selected isotopic species in the early stage of condensation and reduces the sticking probability of excited molecules throughout the condensation zone. In this way the vibrational excitation profoundly influences the subsequent condensation process which is initially dependent on the presence of such dimers and trimers. Accordingly, the presence of vibrational excitation serves to control the condensation process. If the vibrational excitation occurs only in a particular isotopic species, then condensation of that particular species can be selectively inhibited or controlled. The present invention is directed (a) to the use of such selective vibrational excitation to produce controlled homogenous condensation, and (b) to the use of such controlled condensation in an aerodynamic isotope separation process.

A schematic flow chart of controlled condensation aerodynamic isotope separation or enrichment in accordance with the present invention is shown in Fig. 1. In the feed gas makeup 1, a molecular gas containing the isotopes to be separated is mixed with a carrier gas in the desired ratio. Typically, there is a substantial excess of carrier gas, with the ratio of carrier gas to molecular gas being routinely on the order of at least 10:1 and frequently as high as 20:1 or more, depending on the desired operating conditions. The molecular gas is usually a polyatomic molecule, but in some instances may be a diatomic, depending on which elemental isotopes are to be separated. The molecular gas must have an adequate vapor pressure at the temperature and pressure found in plenum chamber 2 so that the ratio of carrier gas to molecular gas in chamber 2 does not change from the desired ratio. In addition, the molecular gas must be one capable of having those molecules containing a particular isotopic species undergo selective vibrational excitation. The carrier gas should have a γ such that the feed gas mixture on undergoing expansion through supersonic expansion nozzle 3 cools sufficiently to cause the molecular gas to begin to condense. The noble, i.e., monatomic, gases as well as nitrogen are quite suitable for this purpose. The carrier gas should not react with the molecular gas and should not absorb the radiation which selectively excites certain molecules of the molecular gas.

The feed gas mixture from plenum chamber 2 is flowed supersonically through expansion nozzle 3. The flow conditions are such that there is an onset of condensation in a region 4 downstream for those molecules that are not vibrationally excited. The condensation in region 4 is homogenous rather than heterogeneous and does not depend on the presence of walls. Condensation region 4 is irradiated by laser 5 to selectively excite those molecules in the feed gas mixture containing a particular isotopic species. While it is desirable for the flux of the irradiating beam to be sufficiently intense to produce multiple photon aborption, it should not be so intense as to cause photodissociation or photoionization. This irradiation serves to prevent or inhibit condensation of those molecules which are selectively excited. As a result, flowing from the irradiation zone 9 is a mixture of gas and condensate particles, with the gas containing more of one isotopic species and the condensate particles containing more of the other isotopic species. It is desirable that the irradiation zone 9 and the condensation region 4 be substantially coextensive and inclusive. The reason for this is that in most molecules there is rather rapid relaxation of the vibrational excitation imparted by the radiation from laser 5. Thus, if the condensation region 4 extends substantially downstream of irradiation zone 5 those molecules which had been vibrationally excited will begin to condense. Such condensation is not controlled and serves to dilute any selective effect which has been produced in condensation within the irradiation zone 5. Thus it is preferable that the irradiation zone 5 extend somewhat upstream of the onset of condensation and extend downstream to the point of significant physical separation of molecules and particles. In this circumstance, the vibrational excitation precludes or inhibits condensation when the excited molecules reach the condensation onset and the excited molecules are maintained uncondensed to the point of physical separation. In region 6, the condensate particles are separated from the gas to a significant degree, resulting in a stream 7 enriched in the particular isotope of interest and a stream 8 depleted in this isotope. Separation may be accomplished in region 6 in a variety of ways, all of which depend on aerodynamic effects.

An embodiment of the invention which makes use of a curved nozzle is shown in schematic cross section in Fig. 2. Feed gas 10 is introduced in plenum 11 and flows sonically through throat 12 of supersonic turning nozzle 13. In region 14 of nozzle 13, the feed gas expands and undergoes rapid cooling until condensation onset 15. The position at which condensation onset 15 occurs depends on the configuration of nozzle 13 and the flow conditions. At condensation onset 15, nucleation of the molecular gas occurs and condensate particles 16 form. In the absence of isotope selective vibrational excitation of the molecular gas, all of it will tend to condense. This is prevented, however, by irradiating the flowing gas in nozzle 13 in region 17 called the irradiation zone by laser radiation of a wavelength which vibrationally excites only those molecules containing a particular isotopic species. As a result of this irradiation, condensation of the selectively excited molecules is prevented or inhibited so that condensation particles 16 contains a greater amount of the unexcited molecules. The curvature of nozzle 13 produces a centrifugal effect which is much greater on the condensate particles 16 than on the gas. As a result, region 18 becomes depleted in condensate particles whereas such particles grow and accumulate in region 19. At the point 24 where skimmer 21 splits the feed flow, flow containment duct 20 incorporates supersonic diffusers 30 and 30'. Diffuser 30 is located in enriched stream 22 while diffuser 30' is located in depleted stream 23. The purpose of the diffusers 30, 30', is to optimize pressure recovery before passing the processed streams 22, 23 on to the next stage. As shown in Fig. 2, the stream 22 is enriched in the lighter isotope and the stream 23 is depleted in the lighter isotope.

Figure 3 shows in schematic cross section another embodiment of the invention, in which aerodynamic isotope separation is achieved through a free jet expansion. In this embodiment, controlled condensation occurs in the same manner as in the embodiment of Fig. 2, but a straight supersonic nozzle 25 replaces the curved nozzle 13 of Fig. 2, so that there is no centrifugal force acting on the gas and condensate particles. Instead, both are confined by walls 26 of nozzle 25 until they exit into low pressure tank 27. This produces a free jet expansion in which the gas expands, radially outward into tank 27 as shown by gas streamlines 28, 28'. However, due to their inertia, the condensate particles 16 are confined to a region near the centerline of the jet as shown by the particle streamlines 29. The core of the jet which contains a high proportion of the condensed particles is collected by skimmer 32 and goes to form depleted stream 33. The gas, however, goes to form enriched stream 34.

Figure 4 is a schematic cross section of still another embodiment of the invention. In this embodiment, controlled condensation occurs in the same fashion as in the embodiments of Figs. 2 and 3 and a straight supersonic nozzle 25 is again employed, but low pressure tank 27 of Fig. 3 is replaced by probe 35. Here, a highly curved flow field of the gas is created by bow shock 36 and the flow around probe 35. By maintaining the proper back pressure within probe 35, most of the gas stream can be forced 28, 28' to flow around rather than through probe 35. The heavy condensate particles 16, however, are unable to follow this rapid turning of the gas and instead flow 29 into probe 35. This separation results in an enriched stream 38 and a depleted stream 37.

The interaction of 10.6 μm CO_2 laser radiation with supersonically flowing SF_6 molecules provides an example of the manner in which vibrational excitation serves to control condensation. Since the binding energy of the SF_6 dimer is substantially smaller than one infrared quantum at 10.6 μm, the lifetime of the excited dimer is very short. Thus, SF_6 molecules in a vibrationally excited state will tend to remain as monomers. Figure 5 schematically shows the experimental setup used to demonstrate this.

Nozzle 40 has an area ratio 10:1 and the throat height is 0.325 mm. The optical path length in test section 44 is 20 cm. The CO_2 laser (not shown in Fig. 5) was a CRL Model 42 operated in the power output range of 10 to 15 W. A liquid-helium-cooled 16-μm diode laser 41 was used to monitor the SF_6 monomer fraction at a position 1 to 3 cm downstream from the CO_2 laser beam. Absorption of rotational lines R(18)-R(24) of the SF_6 ν_4 vibrational mode was monitored during supersonic flow by infrared spectrometer 43. The absorbance $[A = \ell n(I_0/I)]$ of a particular rotational line was measured with CO_2 laser irradiation and without CO_2 laser irradiation and the two results were compared. To establish the condensation characteristics in terms of SF_6 mole fraction and starting pressures, a wide range of starting pressures in supply tank 42 from 1624 to 150 torr, with 5% and 2% SF_6 concentrations in He carrier gas, were used. In addition, in some runs N_2 was used as the carrier gas.

Figure 6 shows the variation of absorbance with starting pressures of the SF_6 mixtures. When 2% SF_6 in He was monitored 2 cm downstream from the CO_2 beam, condensation set in around 700 torr starting pressure, as evidenced by a sharp downward curvature of the absorbance values after an initial, nearly linear, increase of absorbance with increasing pressures. Figure 7 is a plot of absorbance as a function of distance from the nozzle throat. The rapid decay of absorbance indicates that condensation progresses as the gas expansion continues in the contoured nozzle. Although not shown in Fig. 7, a nearly constant monomer concentration was observed over 5 cm distance when the starting pressure was reduced to about 250 torr. Also, the onset of condensation occurred at a lower pressure for the 5% SF_6 in He mixture, and the decrease in the monomer fraction was more rapid in that mixture than for the 2% SF_6 in He mixture used to obtain the data of Fig. 7. Conversion of the absorbance to absolute monomer densities can be done by integrated absorption measurements, but this is not necessary for the present discussion.

The Table summarizes experimental results showing the effect of 10.6 μm CO_2 laser irradiation on SF_6 monomer concentration in the supersonically flowing stream. The changes in monomer fraction are given as ratios of absorbance values of a rotational line with and without CO_2 irradiation. For each value of the absorbance ratio, several experimental data were averaged, and the uncertainty quoted indicates the spread of the data points. For the 5% mixture, the increase in the monomer fraction under CO_2 laser irradiation was about 14% in the intermediate pressure range. For the 2% mixture, the increases in monomer fraction were 60% and 24% for the 1624 and 765 torr starting pressures, respectively. Essentially no change in monomer fraction was observed at 150 torr starting pressure.

TABLE

SF_6 Conc in He	Expansion[a] Press (torr)	Ratio $\frac{A(CO_2\ on)}{A(CO_2\ off)}$[b]
5%	1624	1.03 ± 0.02
	817	1.14 ± 0.04
	150	0.94 ± 0.05
2%	1624	1.6 ± 0.2
	765	1.24 ± 0.06
	150	0.98 ± 0.04

(a) These are starting pressures in supply tank 42. The pressures inside test section 44 during expansion cooling vary nearly linearly with the supply tank pressures in the absence of condensation.

(b) The changes in the monomer fractions are given as ratios of absorbance values of the absorbance $A - [\ell n(I_0/I)]$ of a SF_6 rotational line with and without CO_2 laser radiation.

The foregoing experimental data can be explained in terms of the condensation characteristics described in relation to Figs. 6 and 7. At 150 torr for both the 5% and 2% mixtures there is essentially no condensation regardless of whether there is CO_2 laser irradiation. This follows from the fact that the monomer densities over the distance range of 1 to 5 cm from the nozzle throat are essentially constant. The reason for the nearly unitary value of the absorbance ratio at 1624 torr for the 5% mixture is not clear; however, it may well be that the vibrational relaxation is so rapid under these flow conditions that in the time duration between CO_2 laser irradiation and probing with the diode beam substantial condensation occurs. Based on the CO_2 laser power consumed (∿ 7 W), the photon flux is on the order of 4×10^{20} photons per second. This is a relatively low power level which vibrationally excited only a relatively small fraction (> 15%) of the SF_6 molecules under the supersonic flow condition (5% SF_6 in He at ∿ 100 K). Nonetheless, these experimental results provide strong evidence that vibrational excitation of SF_6 hinders condensation.

Although the foregoing experimental data were obtained without isotopic selective vibrational excitation of the SF_6 molecules, it is well known that laser radiation can be made to selectively excite only those molecules of a polyatomic gas which contain a particular isotopic species. Thus, for example, U. S. patent 4,049,515 teaches that multiple photon absorption from an intense beam of infrared laser radiation may be used to induce selective chemical reactions in molecular species which result in isotope separation. Specific examples given therein teach that isotope selective intense CO_2 laser irradiation may be used to highly enrich ^{34}S in natural SF_6 and ^{11}B in natural $BC\ell_3$.

It is thus apparent that isotope selective CO_2 laser irradiation of supersonically flowing SF_6 or $BC\ell_3$ in accordance with the present invention results in controlled condensation which, in turn, can be used to produce isotope separation or enrichment by aerodynamic means. Less intense CO_2 laser fluxes are required than are necessary to produce chemical reaction, although some multiple photon absorption is desirable. A laser suitable for producing isotope selective vibrational excitation in UF_6 leading to controlled condensation thereof from a supersonic flow is an

HF pumped CdSe optical parametric oscillator tunable in the region of 628 cm^{-1}.

The scope of the invention is as set forth in the Summary of the Invention and the appended claims and is not limited to the specific embodiments described herein. Such embodiments are merely illustrative of the best mode contemplated for the performance of the invention.

Claims

WHAT WE CLAIM IS:

1. A method of producing controlled homogenous condensation of a molecular feed gas containing a plurality of isotopes which comprises (a) supersonically flowing said feed gas through an expansion nozzle under flow conditions at which the expansion of said supersonically flowing feed gas produces condensation of said gas when said gas is not vibrationally excited, and (b) irradiating said supersonically flowing gas with laser radiation of a wavelength which selectively excites those molecules in said feed gas containing a particular isotope.

2. The method of claim 1 wherein said molecular feed gas is mixed with a carrier gas.

3. Apparatus for producing controlled homogenous condensation of a molecular feed gas containing a plurality of isotopes which comprises (a) a supersonic nozzle, said nozzle producing condensation in said feed gas when said gas is flowed supersonically through said nozzle and said gas is not vibrationally excited, (b) means for producing supersonic flow conditions in said nozzle whereby condensation of said molecular feed gas occurs when said gas is not vibrationally excited, and (c) means for irradiating the supersonically flowing gas stream with laser radiation of a wavelength which selectively excites those molecules in said feed gas containing a particular isotope.

4. Apparatus for obtaining elemental values enriched in a particular isotope which comprises in combination (a) a supersonic nozzle, said nozzle producing condensation in a molecular feed gas containing a plurality of isotopes

to be separated when said gas is flowed supersonically through said nozzle and said gas is not vibrationally excited, (b) means for producing supersonic flow conditions in said nozzle whereby condensation of said molecular feed gas occurs when said gas is not vibrationally excited, (c) means for irradiating the supersonically flowing gas stream with laser radiation of a wavelength which selectively excites those molecules in said feed gas containing a particular isotope, and (d) aeroydynamic means for separating condensate particles from the flowing gas stream.

5. The apparatus of claim 4 wherein the irradiation zone in said nozzle is substantially coextensive with the condensation region in said nozzle.

6. The apparatus of claim 5 wherein said irradiation zone extends upstream of the region of condensation onset.

7. The apparatus of claim 4 wherein said nozzle is a curved nozzle having a turning zone either downstream or encompassing the expansion region with a radius of curvature sufficient to exert a substantial centrifugal force on the stream flowing through said turning zone, and said apparatus has skimmer means at the exit of said turning zone for separating condensate particles from the flowing gas stream.

8. The apparatus of claim 7 wherein said molecular feed gas is UF_6 said UF_6 has mixed therewith a carrier gas selected from the class consisting of the monatomic gases and N_2, the supersonically flowing gas mixture is cooled sufficiently before the onset of condensation that substantially all the UF_6 molecules are reduced to the ground state, and said means for irradiating said

supersonically flowing gas mixture is an infrared laser tunable in the spectral region of 628 cm^{-1} sufficiently to selectively vibrationally excite those UF_6 molecules containing ^{235}U.

9. The apparatus of claim 4 wherein said nozzle is a straight nozzle, and said nozzle has disposed at the exit end thereof a low pressure expansion tank adapted to allow a free jet expansion therein of the flowing stream exiting from said nozzle, said tank having disposed on the axis of the free jet expansion skimmer means for separating condensate particles from the gas in the free jet expansion.

10. The apparatus of claim 4 wherein said nozzle is a straight nozzle, and said nozzle has axially disposed downstream of the condensation region a hollow probe, said probe being so disposed that a substantial portion of the condensate particles flow through said probe while a majority of the gas flows around it.

11. A method for obtaining elemental values enriched in a particular isotope which comprises (a) supersonically flowing a molecular feed gas containing a plurality of isotopes to be separated through an expansion nozzle under flow conditions at which the expansion cooling of said supersonically flowing molecular feed gas produces condensation of said gas when said gas is not vibrationally excited, (b) irradiating said supersonically flowing gas with laser radiation of a wavelength which selectively excites those molecules in said feed gas containing a particular isotope, and (c) aerodynamically separating condensate particles from the flowing gas stream.

12. The method of claim 11 wherein the irradiation region is substantially coextensive with and includes the condensation region.

13. The method of claim 12 wherein the irradiation region extends upstream of the region of condensation onset.

14. The method of claim 11 wherein said molecular feed gas has mixed therewith a carrier gas.

15. The method of claim 14 wherein said supersonically flowing gas has a substantial centrifugal force exerted on it in the condensation region, said centrifugal force acting to separate the flowing gas from the condensate particles.

16. The method of claim 15 wherein said molecular feed gas is UF_6, said carrier gas is N_2, the supersonically flowing gas mixture is cooled sufficiently before the onset of condensation that substantially all of the UF_6 molecules are reduced to the ground state, and the supersonically flowing stream is irradiated with infrared laser radiation near 628 cm^{-1} which selectively vibrationally excites those UF_6 molecules containing ^{235}U.

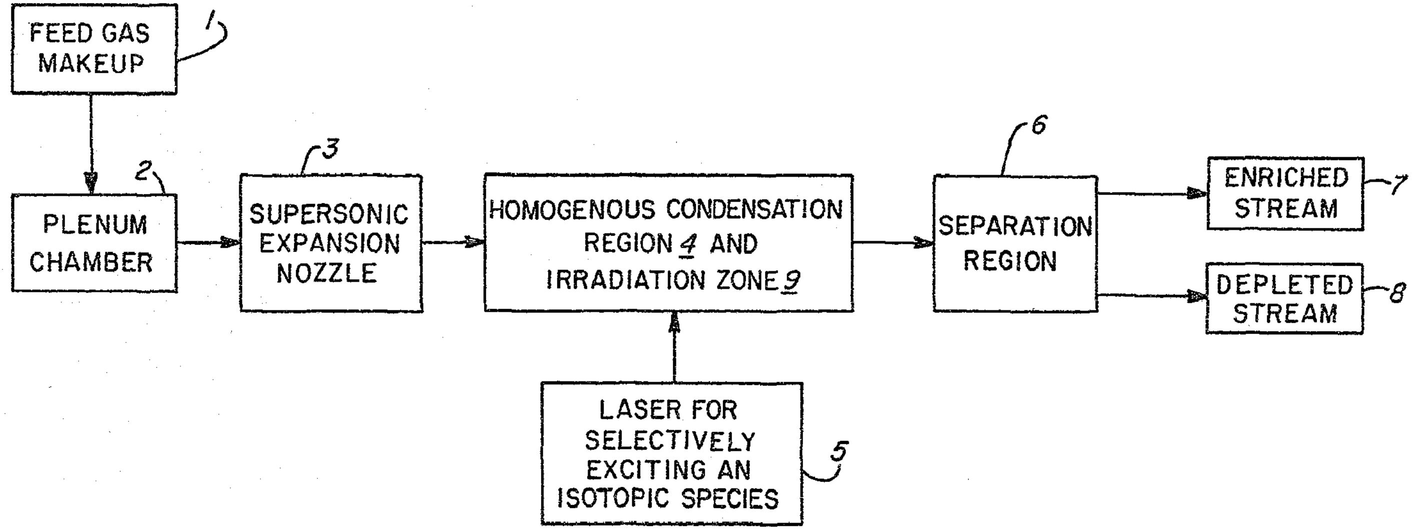

FIG. 1

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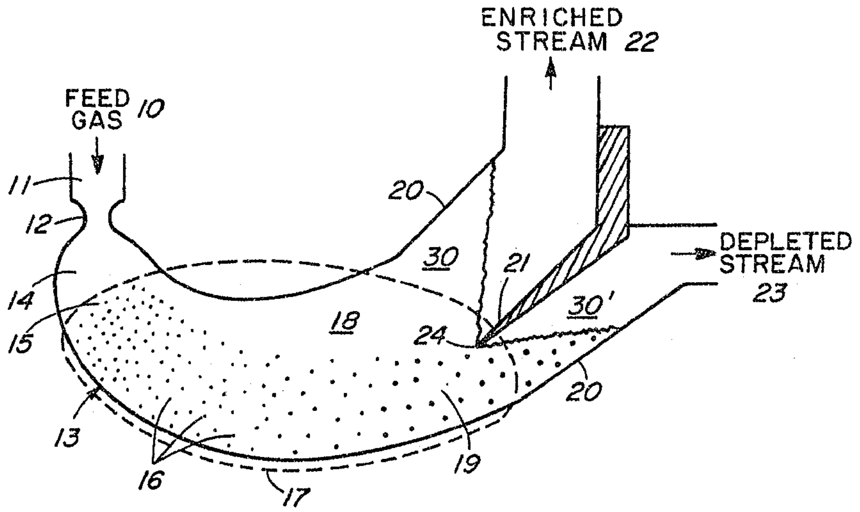

FIG. 2

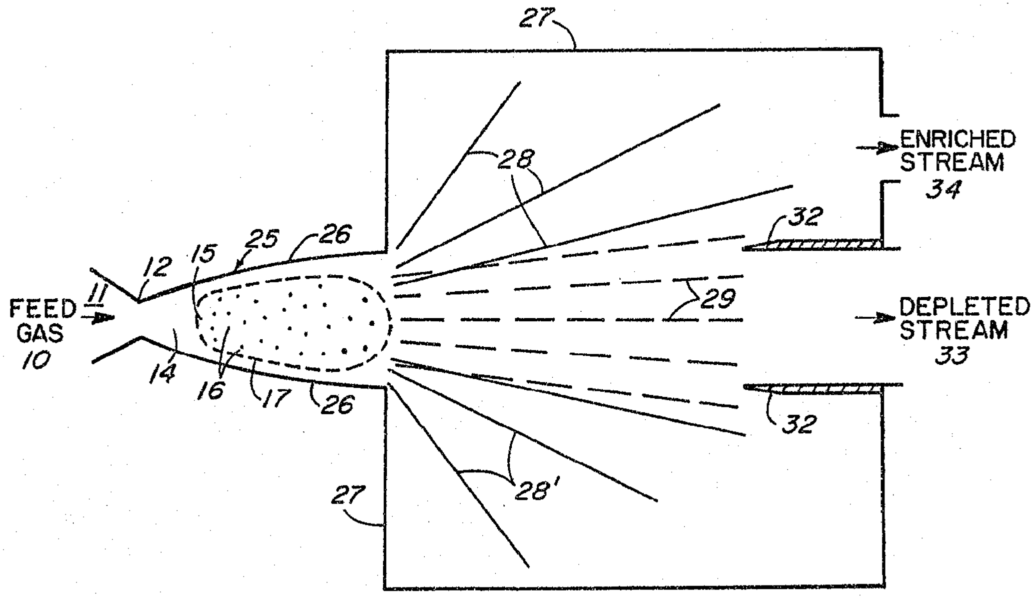

FIG. 3

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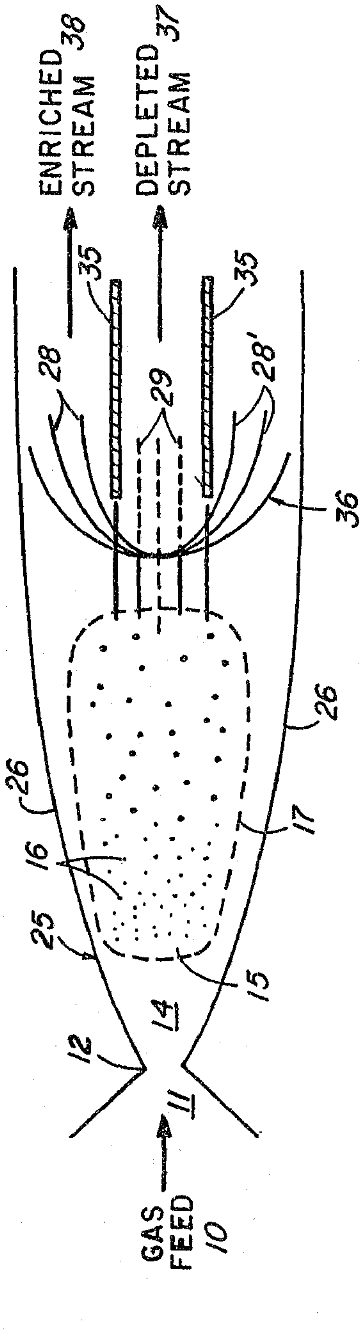

FIG. 4

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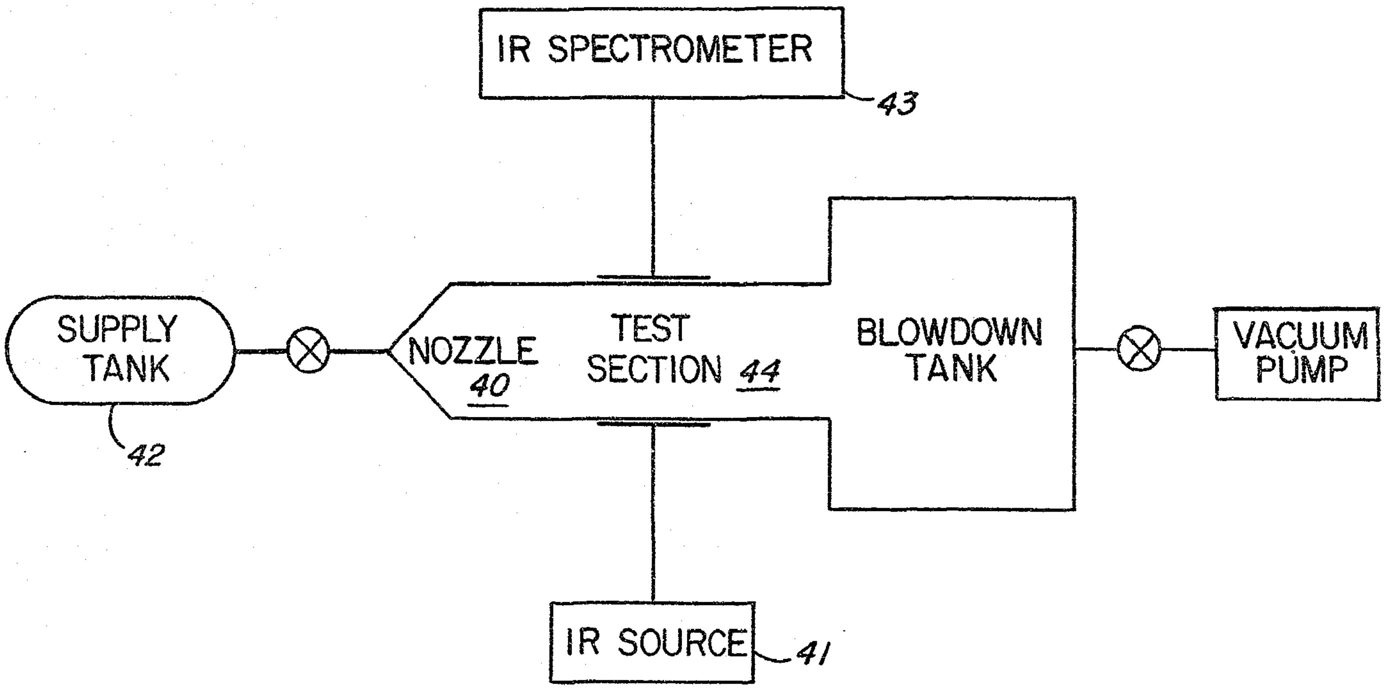

FIG. 5

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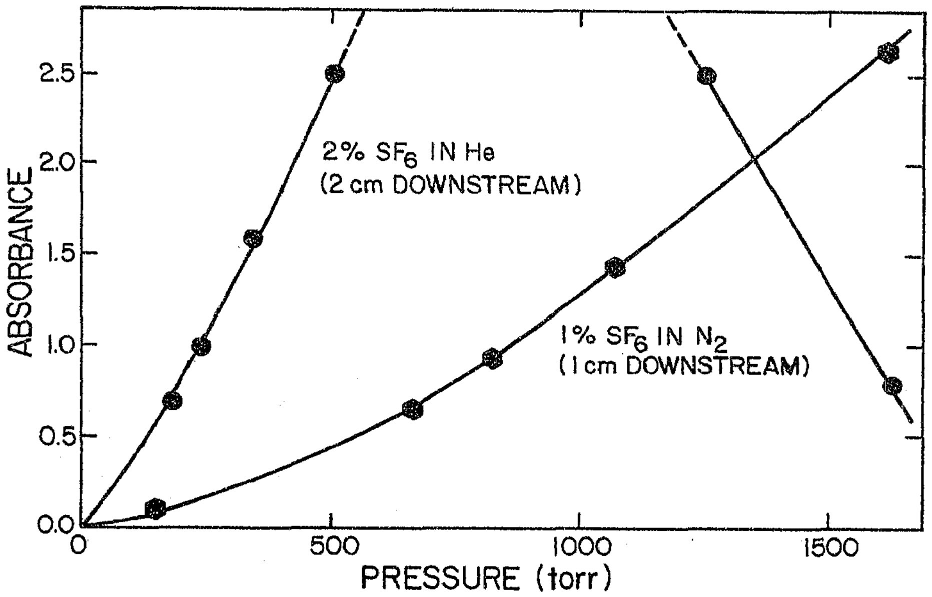

FIG. 6

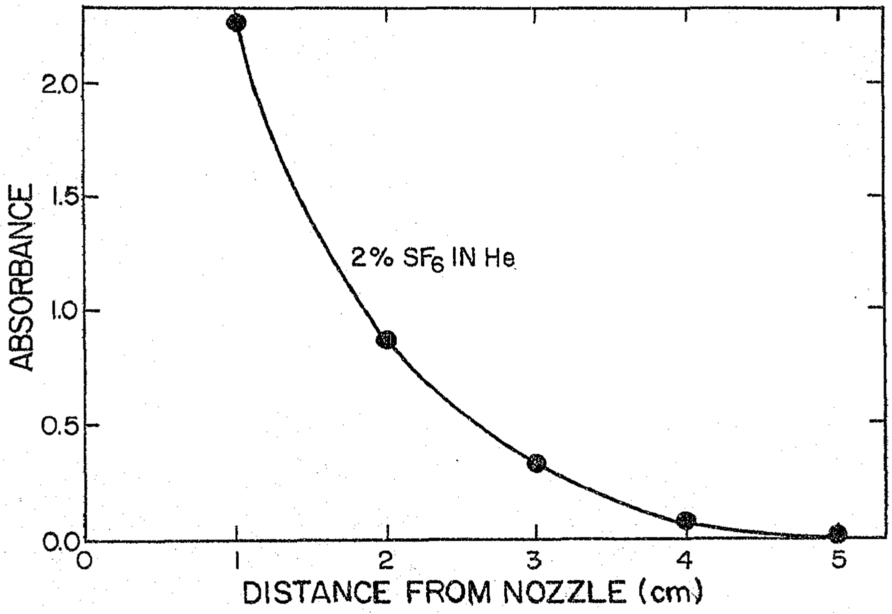

FIG. 7

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