



11) Publication number:

0 242 028 A2

(12)

EUROPEAN PATENT APPLICATION

21 Application number: 87301339.5

(51) Int. Cl.4: C01B 23/00

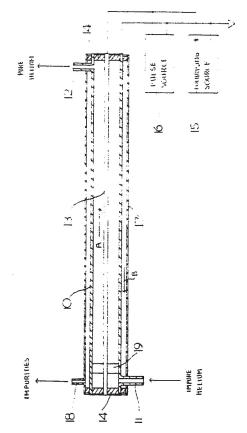
2 Date of filing: 17.02.87

3 Priority: 16.04.86 GB 8609325

Date of publication of application: 21.10.87 Bulletin 87/43

Designated Contracting States:
DE FR GB

- Applicant: Hewlett-Packard Company Mail Stop 20 B-O 3000 Hanover Street Palo Alto California 94304(US)
- ② Inventor: Lovelock James Ephraim Combe Mill St Giles on the Heath Launceston Cornwall(GB)
- (2) Representative: Squibbs, Robert Francis Hewlett-Packard Limited Nine Mile Ride Wokingham Berkshire RG11 3LL(GB)
- Method and apparatus for purifying a continuous flow of helium and/or neon gas.
- 5 Continuous production of ultra-pure helium and neon gases is achieved by utilising the fact that these gases have the highest ionisation and excitation potentials of all chemical substances. The helium/neon gas stream to be purified is excited by, for example, a corona discharge not only to directly ionise some of the impurities but also to produce rare-gas ions and metastable atoms. These ions and metastables subsequently collide with and ionise impurities. A polarising field is used to draw the impurity ions out of the main gas flow and into a subsidiary flow bled from the main flow. In a preferred embodiment, the polarising field is established between a central rod-shaped anode(I3) and a surrounding tubular cathode(I0) through which the main gas flow passes. The cathode is made of a porous material so that positive impurity ions can be re-Moved by the radial outwards flow of gas thereth-▼rough.



INTEL 1215





METHOD AND APPARATUS FOR PURIFYING A CONTINUOUS FLOW OF HELIUM AND/OR NEON GAS

10

15

20

25

35

40

The present invention relates to a method and apparatus for purifying a continuous flow of helium and/or neon gas; in particular, but not exclusively, the invention is concerned with the continuous production of ultra pure helium or neon (or mixtures thereof) where 'ultra pure' means that contamination with any impurity gas or gases is less than one part in ten million by volume. Helium and neon gases of this purity have many uses including as sample carrier gases in modern analytical techniques, filling gases for discharge tubes and lasers, and purge gases in the semiconductor industry.

1

It is an object of the present invention to provide for the production of ultra pure helium and neon gases on a continuous basis and in a manner relatively easy to implement.

According to one aspect of the present invention, there is provided a method of purifying a continuous flow of helium or neon gas or a mixture thereof, said method involving the operations of:

-passing a stream of the gas to be purified along a predetermined flow path;

-ionising impurities in the gas both directly and by exciting the gas to generate rare-gas ions and metastable atoms which thereafter ionise said impurities; and

-removing the positive impurity ions from the main gas flow by setting up a polarising field across said stream to cause migration of said impurity ions out of the main gas flow.

The effectiveness of this purification method rests on the fact that helium and neon have the highest ionization and metastable excitation potentials of all chemical substances. As a result, if impure helium or neon is subject to an ionising energy input, the more easily ionized impurity atoms and molecules are preferentially ionised. Furthermore, the helium and neon metastables and ions produced both readily exchange their stored energy and charge respectively to impurity molecules they encounter by collision which greatly enhances the efficiency of the ionisation process and leaves the helium or neon as uncharged, unexcited atoms. The ionising effect of rare-gas 'metastable' atoms is, of course, a known effect that has previously been exploited in sensitive detectors of the form described in UK Patent Specification No. 882,977.

According to the another aspect of the present invention, there is provided apparatus for purifying a continuous flow of helium or neon gas or a mixture thereof, said apparatus comprising:

-an inlet for the gas to be purified;

-an outlet for purified gas;

-means defining an elongate gas flow path

between said inlet and outlet;

-exhaust means for bleeding off a subsidiary flow of gas from the main gas flow passing along said gas flow path;

-ionisation means operative to cause ionisation of impurities in the gas passing along said flow path, both by direct ionisation of the impurities and by exciting the rare gas atoms such as to generate rare-gas ions and metastable atoms which subquently ionise the impurities in the gas; and

-polarising-field means operative to generate a polarising field across the gas flow path for drawing positive impurity ions out of the main gas flow and into said subsidiary flow for removal through said exhaust means.

Preferably, the polarising-field means includes a central elongate anode coaxially surrounded by a tubular cathode that also serves as said means defining the gas flow path between said inlet and outlet.

Advantageously, the tubular cathode is porous and additionally serves as said exhaust means, the gas passing outwards through the cathode constituting said subsidiary gas flow and including the impurities drawn to the cathode by the polarising field.

Conveniently the ionisation means may take the form of a corona discharge device constituted by the anode and cathode of said polarising-field means and by a source of high potential, short duration pulses connected between the anode and cathode. Other forms of ionisation means can also be used such as radioactive sources, ultraviolet sources, or external X-ray or gamma-ray sources.

A helium/neon gas purifier embodying the invention will now be described by way of non-limiting example with reference to the accompanying diagrammatic drawing, the sole Figure of which is a longitudinal cross-section through the purifier.

Although the purifier to be described is suitable for purifying helium or neon gases individually or in mixture, for ease of explanation the following description will be given in terms of the purification of a helium gas flow only.

As shown in the drawing, the purifier comprises a tubular member 10 defining a main gas flow path between an inlet II for impure helium and an outlet I2 for purified helium. The tubular member I0 is formed of a porous conducting material such as sintered metal or sintered metallized glass or quartz. The tubular member I0 constitutes a cathode of the purifier, the complementary anode I3 being formed by a metal rod positioned coaxially in the tubular cathode I0 by insulating end seals I4.



10

20



A polarising field is set up between the anode 13 and cathode 10 by a DC polarising source 15. This source 15 applies a potential between the anode and cathode which is substantially less than would cause an arc or discharge in helium at the pressure existing within the purifier during operation (generally slightly above atmospheric pressure); the applied potential is, however, sufficient to cause a rapid migration of any positive ions present to the cathode 10.

In order to ionise impurities in the gas flowing through the cathode IO, a pulse source I6 is provided for supplying high frequency (100 kHz to 50MHz), high potential, short duration (less than one microsecond) pulses between the anode and cathode whereby to set up an ionising corona discharge between the electrodes. Such an arrangement is disclosed in UK Patent Specification No. 1,597,623; and will not, therefore, be further described herein except to note that since to initiate corona discharge at least one ion or electron must be present, it is prudent to provide some means for generating the required ion or electron (although background radiation could be relied upon). Consequently, the polarising source could be made adjustable to cause an initial discharge before being set back to its normal operating potential.

Surrounding the tubular cathode I0 is a cylindrical jacket I7 for collecting gas which, in operation, passes radially outwards through the porous cathode I0. This jacket I7 has an exhaust port I8 adjacent the inlet end of the main gas flow path defined by the cathode I0.

A diffuser 19 is provided in the main gas flow path adjacent to inlet II to establish streamline flow down the inside of the cathode I0.

In operation, impure helium typically, one part per million impurity) is supplied to the inlet II at a slight over-pressure relative to the surrounding atmosphere to establish a streamlined main gas flow coaxially down the inside of the cathode I0 in the direction of arrow A to the outlet I2. Due to the porous nature of the cathode I0, a small subsidiary gas flow is set up that passes radially outwards through the cathode and then in the direction of arrow B to the exhaust port I8.

The corona discharge established between the anode I3 and cathode I0 by the pulse source I6 causes ionisation of any impurity atoms or molecules within the main gas stream either directly or by first generating helium metastable atoms and helium ions which subsequently collide with and ionise any impurities. Ultra-violet raditation emitted from the transitions of the helium ions and metastables will also produce ionisation of impurities by photo-ionisation. The predominant impurity-ionising

process will generally be that due to the collision of helium metastables with impurity atoms and molecules; however at high field intensities ionisation by electron impact may predominate.

The polarising field set up between the anode I3 and cathode II by the polarising source I5 causes positive impurity ions to migrate to the cathode where they pass out of the main gas stream through the porous cathode II as part of the subsidiary gas flow.

The purified helium typically with an impurity level of less than one part in ten million exits the purifier through the outlet I2 while the impurities are exhausted through exhaust port I8.

The actual dimensions of the purifier will depend upon the desired flow rate of helium and the degree of purity sought. However, typical dimensions would be a tube I0 mm in internal diameter with a length of 0.3 metres.

An ion current of 4.3 microamperes would be needed to remove all of a one part per million impurity in helium flowing at one millilitere per second at N.T.P. This assumes that all of the ion current is carried by impurity ions and that the impurities are completely removed from the main helium flow after the impurity ions encounter the cathode. In practice, ionising efficienes of I to 10% are more usual.

The purifier can be thought of as analogous to a distillation column with gaseous diffision processes tending to hinder the smooth operation of the separation. For a cathode I0 mm in internal diameter and an anode 2 mm diameter and with the flow rate and impurity level quoted in the preceding paragraph, each 5 to I0 mm section of the purifier corresponds to a plate of a distillation column and 67% of impurity will be removed in each of the sections. Thus, five sections should theoretically remove about 99% of the impurity. A typical purifier is in fact 0.3m or more in length to extend the range of purification and the maximum flow rate.

The structural and electrical characteristics of the purifier are preferably such as to avoid the development of ion densities in the purifier in the plasma region. In plasma, ions and electrons are closely associated in a gaseous cloud and the ions cannot be so readily moved to the electrodes by the applied polarising field. For a 10 mm section of purifier, with a cathode having an internal diameter of 10 mm, the polarising field current should not exceed one microampere if plasma conditions are to be avoided.

It will be appreciated that various modifications can be made to the described form of purifier. Thus, instead of providing an ionising source in the form of a corona discharge device, other forms of ionising source could be employed such as, for



15

40



example, a radioactive source located within the cathode I0, an ultraviolet source generated by a subsidiary discharge, an external X-ray or gammaray source.

Furthermore, whilst the use of a porous cathode is a particularly convenient and efficient way of removing the impurity atoms and molecules, it would be possible to use a non-porous cathode and then to separate off from the main gas flow, the gas stream that pases directly over the cathode and within which the neutralised impurities will be concentrated.

Although the heavier rare gases such as argon could be subject to the same purification process as described and illustrated above for helium and neon, the effectiveness of the process would be greatly reduced since many of the common impurities of the heavier rare gases would not be preferentially ionised.

Claims

I. A method of purifying a continuous flow of helium or neon gas or a mixture thereof, characterised in that said method involves the operations of:

-passing a stream of the gas to be purified along a predetermined flow path;

-ionising impurities in the gas both directly and by exciting the gas to generate rare-gas ions and metastable atoms which thereafter ionise said impurities; and

-removing the positive impurity ions from the main gas flow by setting up a polarising field across said stream to cause migration of said impurity ions out of the main gas flow.

Apparatus for implementing the method of claim I, characterised in that said apparatus comprises:

-an inlet for the gas to be purified;

-an outlet (I2) for purified gas;

-means (I0) defining an elongate gas flow path between said inlet and outlet;

-exhaust means (I0) for bleeding off a subsidiary flow of gas from the main gas flow passing along said gas flow path;

-ionisation means (I0,I3,I6) operative to cause ionisation of impurities in the gas passing along said flow path, both by direct ionisation of the impurities and by exciting the rare gas atoms such as to generate rare-gas ions and metastable atoms which subsequently ionise the impurities in the gas; and

-polarising-field means (I0,I3,I5) operative to generate a polarising field across the gas flow path

for drawing positive impurity ions out of the main gas flow and into said subsidiary flow for removal through said exhaust means (I0,I7,I8).

6

- 3. Apparatus according to claim 2, wherein said polarising-field means includes a central elongate anode(I3) coaxially surrounded by a tubular cathode (I0) that also serves as said means defining the gas flow path between said inlet and outlet.
- 4. Apparatus according to claim 3, wherein the tubular cathode (I0) is porous and additionally serves as said exhaust means, the gas passing outwards through the cathode constituting said subsidiary gas flow and including the impurities drawn to the cathode (I0) by the polarising field.
- 5. Apparatus according to claim 4, wherein the cathode (I0) is surrounded by a jacket (I7) for collecting the subsidiary gas flow passing through the cathode, the jacket (I7) being provided with an exhaust port (I8) positioned such that the subsidiary gas flow passes along the outside of the cathode (I0) in the opposite direction to the main gas flow within the cathode (I0).
- 6. Apparatus according to claim 3 or claim 4, wherein said ionisation means comprises a corona discharge device constituted by the anode (I3) and cathode (I0) of said polarising field means and by a source (I6) of high potential, short duration pulses connected between the anode and cathode.
- 7. Apparatus according to any one of the claims 2 to 6 further comprising a diffuser (I9) positioned in the gas flow path adjacent the inlet to streamline the flow of gas along said flow path.



