

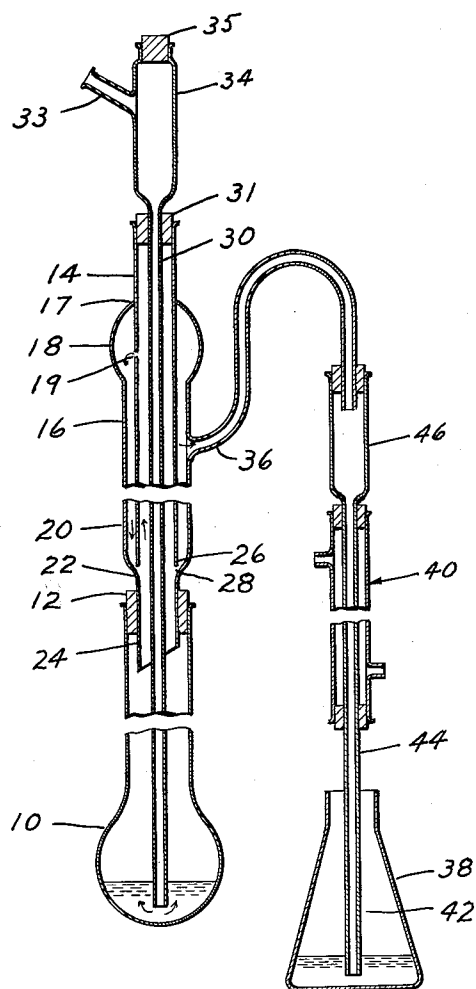
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FRACTIONATING COLUMN FOR KJELDAHL APPARATUS AND THE LIKE

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FRACTIONATING COLUMN FOR KJELDAHL
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13 Claims. (Cl. 202-190)

This invention relates to an improved apparatus for nitrogen analysis or the determination of the total nitrogen in a specimen. Apparatus of this type is generally known as "Kjeldahl" apparatus and the present invention particularly provides an improved fractionating device especially useful with apparatus of this type.

In Kjeldahl apparatus, the nitrogen analysis is accomplished essentially by a process of fractional distillation. The specimen to be analyzed is placed in a flask or still which is sealed except for openings to permit steam to be introduced and gaseous mixture to escape. The gaseous mixture is fractionated and passed on to a receiving vessel through a condenser.

The fractionating device of the present invention consists essentially of an inner jacket which receives vapor from the still, and an outer jacket surrounding the inner jacket, with the upper portion of the outer jacket forming an expansion chamber and the lower portion forming a condensation chamber. An aperture is provided in the inner jacket to permit the passage of vapor to the expansion chamber and a return passage is provided at the lower portion of the condensation chamber to permit the return of the fractionated substance to the still. A discharge passage leads from the upper portion of the condensation chamber. Preferably a steam supply line for the still passes through the inner jacket.

The resulting construction is a relatively simple one and I have found that it affords superior control of the fractionating process and accuracy of the analysis.

A representative embodiment of the invention is shown in the accompanying drawing in a laboratory form designed for micro analysis. This drawing consists of FIGURE 1 which shows the apparatus somewhat schematically in elevation, certain parts being illustrated in section.

In the construction shown, the specimen to be analyzed is placed in the flask or still 10 and the fractionating device is attached to the flask 10 in sealed relation therewith through a stopper 12.

The fractionating device includes an inner tubular jacket 14 and an outer jacket 16, the upper end of which is joined to the inner jacket at 17. The outer jacket is formed with an enlarged upper portion 18 to provide an expansion chamber which communicates with the inner jacket through an opening 19, preferably spherical in shape. The lower portion 20 of the outer jacket forms a condensation chamber and its lower end extends inwardly at 22 and onwardly at 24 to form an extension of the inner jacket. The lower end 26 of the inner jacket 14 terminates adjacent the inwardly extending wall portion 22 of the outer jacket so as to provide an annular distillate return passage 28 to the inner jacket extension 24 and thence to the flask 10. If desired, the inner jacket and its extension can naturally be made in one piece and a suitable return passage cut in the wall thereof but the construction shown is somewhat simpler. The passage 28 is relatively small so that distillate coming from the condensation chamber 20 will slowly pass through it while backing up slightly above the passage and thereby forming a liquid seal so that vapor coming from the flask 10 will pass through the inner jacket 14. In other words, the passage 28 is essentially one-way.

A steam delivery tube 30 extends through a stopper 31 at the upper end of the inner jacket, thereby sealing

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this end, and into the flask 10 through the inner jacket 14-24. The steam delivery tube is preferably formed with a Y-shaped upper end 32. Connection to a steam generator (not shown) is made at the branch passage 33 and when the fractionating device is connected to the flask 10 as shown, an alkaline solution may be introduced into the flask through the branch passage 34 which is otherwise closed by a stopper 35.

A discharge passage 36 is provided in the wall of the outer jacket toward the upper portion of the condensation chamber thereof and fractionated vapor is conducted through this passage to a receiving vessel 38 through a suitable condenser 40.

The flask 10 containing a specimen such as standard ammonium sulfate solution is attached to the lower portion 24 of the fractionating device and then alkaline solution is introduced into the flask 10 as by removing the stopper 35 and pouring it through the steam delivery tube 30. Steam is then supplied through the tube 30 and steam distillation is carried on in the flask 10. The vapor, continuously heated by incoming steam, rises through the inner jacket 14 and its extension 24 and passes through the small opening 19 into the expansion chamber portion 18 of the outer jacket. A sudden change in the pressure and temperature of the vapor results from the expansion and from contact with the relatively cool wall of the outer jacket. Fractionating of the vapor takes place and volatile NH_3 gas is easily separated from the steam phase, conducted through the discharge passage 36, the condenser 40 and collected in the receiving flask 38. An alkaline liquid condensate is formed in the condensing chamber portion 20 of the outer jacket and drips back into the still 10 through the return passage 28.

The rate of distillation should be adjusted so that bubbles do not pass through the surface of the solution in the receiving flask 38. Under ideal conditions any bubbles will move back and forth near the lower end 42 of the conduit 44. The upper portion of this conduit 44 is enlarged at 46 to prevent any serious backing up of the solution.

In the use of traditional types of Kjeldahl apparatus, there is a tendency to distill over alkali, contaminating the solution being collected in the receiving flask. With the present apparatus, alkali contamination has never been observed unless foam is seen passing through the opening 19. If foam is produced during distillation, it tends to be dispersed before passing through this opening due to the heating obtained from the incoming steam.

Versatility is another feature of this fractionating device. A micro-Kjeldahl flask containing a digested specimen may be directly attached to the stopper 12 at the lower end 24 of the outer jacket. A macro-Kjeldahl set-up can be obtained by merely changing the flask 10, condenser 36 and receiving vessel 34. Two sets of apparatus, to carry out the distillation of two specimens simultaneously, may be used with one steam generator placed on a stand, because the apparatus is constructed rather simply and occupies mainly vertical space. This feature also facilitates the easy moving of the apparatus.

Other features and advantages will be readily apparent to those skilled in the art.

While preferred embodiments have been described above in detail, it will be understood that numerous modifications might be resorted to without departing from the scope of my invention as defined in the following claims.

I claim:

1. Fractionating apparatus for nitrogen analysis comprising a steam delivery tube having a delivery opening at one end thereof, means for connecting a still to said delivery opening, an inner jacket surrounding said tube and having a vapor inlet opening spaced along said tube

from said one end thereof, an outer jacket surrounding at least a portion of the length of said inner jacket and forming a chamber between said inner and outer jackets, an aperture for the passage of vapor from said inner jacket to said chamber, a discharge opening in said outer jacket, means for connecting a condenser to said discharge opening, and a substantially one-way return passage from said chamber to said inner jacket at the lower end of said chamber.

2. Fractionating apparatus according to claim 1 wherein said inner jacket has a substantially cylindrical wall, said aperture for the passage of vapor from said inner jacket to said chamber comprising a spherical opening formed in said cylindrical jacket wall, the size of said opening being such as to cause a sudden expansion and fractionating of vapor passing therethrough.

3. Fractionating apparatus according to claim 1 wherein said discharge opening is located vertically intermediate said aperture and said one-way passage.

4. Fractionating apparatus for use with a still and a condenser comprising an inner jacket, an outer jacket surrounding said inner jacket, the upper portion of said outer jacket forming an expansion chamber, a condensing chamber formed by the lower portion of said outer jacket, the wall of said outer jacket at the lower end of said lower portion extending inwardly and onwardly beyond the end of said inner jacket to form an extension thereof and an opening for receiving vapor from said still, the lower end of said inner jacket terminating adjacent to the inwardly extending wall portion of said outer jacket to form an opening of capillary size between said inner and outer jackets, an aperture between said inner jacket and said expansion chamber, the size of said aperture being such as to cause a sudden expansion and fractionating of vapor passing therethrough, and a discharge passage in the wall of said outer jacket for connecting said fractionating apparatus to said condenser.

5. Fractionating apparatus according to claim 4 further characterized by a steam delivery tube extending through said inner jacket, through the said extending portion of said outer jacket and beyond the end of said extending portion.

6. Fractionating apparatus according to claim 4 wherein the cross-sectional area of said inner jacket is substantially equal to the cross-sectional area of the extending portion of said outer jacket.

7. Fractionating apparatus comprising a vertically extending inner jacket having an upper end and a lower end, a vapor inlet opening at the lower end thereof, means for sealing the upper end thereof, means for detachably

connecting a still to said lower end, an outer jacket surrounding at least a portion of the length of said inner jacket and forming a chamber between said inner and outer jackets, an aperture for the passage of vapor from said inner jacket to said chamber, the size of said aperture being such as to cause a sudden expansion and fractionating of vapor passing therethrough, a discharge opening in said outer jacket for connecting a condenser thereto, and a substantially one-way return passage from the lower portion of said chamber to said inner jacket.

8. Fractionating apparatus according to claim 7 further characterized by means for heating said inner jacket along the length thereof between said outlet opening and said aperture.

9. Fractionating apparatus according to claim 8 wherein said means for heating said inner jacket comprises a tubular member extending through said inner jacket, and means for passing a heated fluid through said tubular member.

10. Fractionating apparatus according to claim 9 wherein said tubular member has a discharge end extending into said still.

11. Fractionating apparatus according to claim 10 wherein said tubular member is provided with a branch passage through which a specimen to be fractionated can be introduced into said still.

12. Fractionating apparatus according to claim 7 wherein said discharge opening is vertically intermediate said aperture and said return passage, said aperture being located to discharge vapor into the upper portion of said chamber.

13. Fractionating apparatus according to claim 7 wherein said passage comprises a small capillary opening having a width of 0.2 to 0.5 mm.

References Cited in the file of this patent

UNITED STATES PATENTS

1,585,217	Uebelmesser	May 18, 1926
1,765,960	Wrenn	June 24, 1930
2,410,045	Burk et al.	Oct. 29, 1946
2,470,230	Andreasen et al.	May 17, 1949
2,667,451	Larson	Jan. 26, 1954
2,675,346	Maclean	Apr. 13, 1954
2,859,155	Cichelli et al.	Nov. 4, 1958
2,907,638	Dryer et al.	Oct. 6, 1959
2,907,641	Nottebrock	Oct. 6, 1959

OTHER REFERENCES

Industrial and Engineering Chemistry, 1939, volume 11, No. 11, page 635, C. E. Redemann.