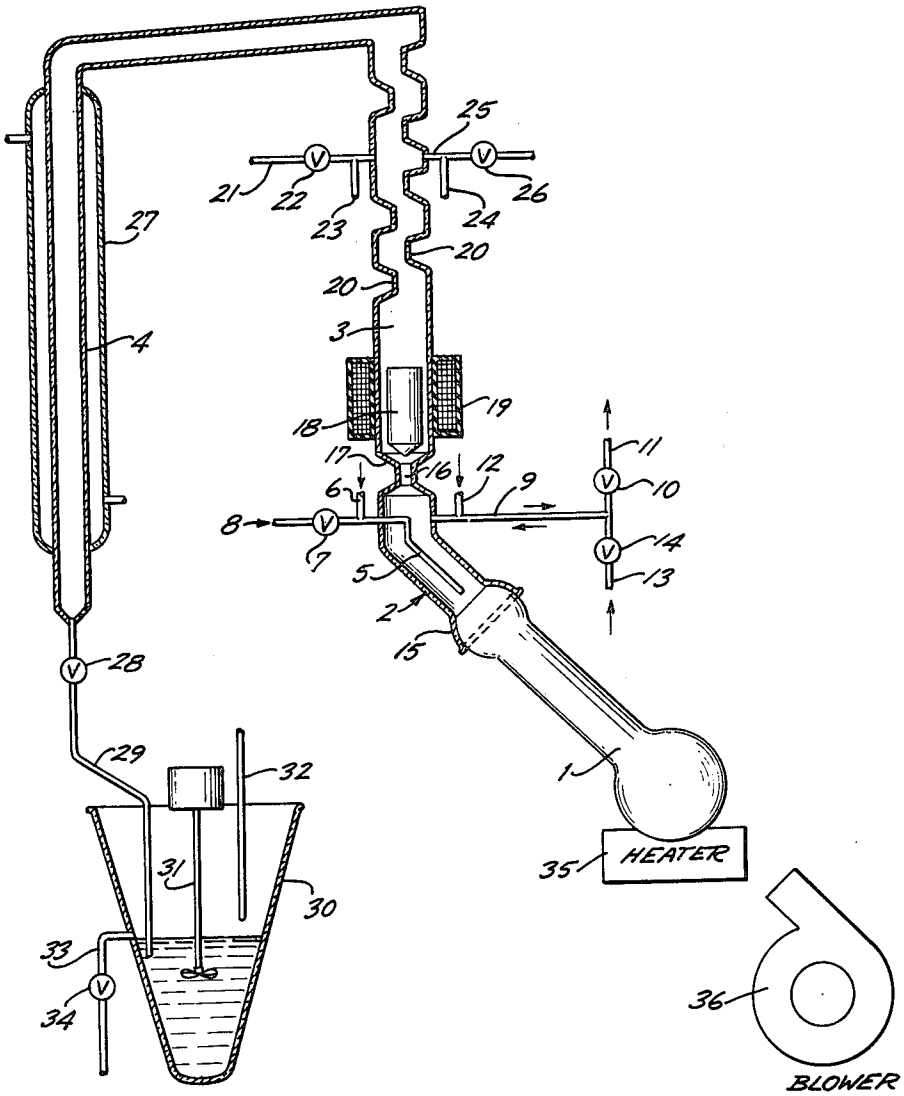
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APPARATUS FOR KJELDAHL NITROGEN DETERMINATION

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INVENTOR.
EVAN L. R. STOKSTAD
BY Harvey W. Eschbach
ATTORNEY

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APPARATUS FOR KJELDAHL NITROGEN DETERMINATION

Evan L. R. Stokstad, Pearl River, N.Y.
(769 Arroyo Court, Lafayette, Calif.)

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This is a continuation-in-part of my application Serial No. 837,810, filed September 3, 1959, and now abandoned.

This invention relates to an improvement in methods and apparatus for the determination of nitrogen by the Kjeldahl method.

The Kjeldahl method for the determination of organically bound nitrogen involves three distinct operations. The first of these is digestion of the organic material with a strong sulphuric acid mixture to convert the nitrogen into ammonium sulfate. The second step involves decomposition of the ammonium sulfate with a strong alkali and distillation of the resulting ammonia. The third step is measurement of the amount of ammonia that is released, distilled and collected.

The Kjeldahl method of determining nitrogen is very cumbersome and time-consuming. In many cases it is desirable to know the analytical results as soon as possible so that the manufacturing operation may continue without interruption or the loaded materials may be classified and shipped to market. Although many efforts have been make to speed up and simplify the process, very little progress has been made.

Difficulties in improving the manner in which the Kjeldahl analysis is conducted have been due in large measure to the nature of the operation. The digestion requires a moderately long heating period at high temperatures and with very strong acid. During the digestion period, acid fumes are given off. Since the final result depends upon measurement of a weakly alkaline ammonium hydroxide solution, it is essential that none of the acid or acid fumes be permitted to enter into the flask which receives the subsequent ammonia distillate.

On the other hand, decomposition of the ammonium sulfate that is formed during the digestion is carried out with the aid of strong alkalis and traces of these materials must not be permitted to enter the distillate. For these reasons the acid digestion and alkali decomposition must be carried out with great care and are usually done in apparatus particularly suited to the individual operation being undertaken. The operator must, therefore, be in almost constant attendance to rearrange the apparatus and see that the proper cycles of operation are carried out.

The new apparatus of the present invention eliminates many of the disadvantages of the Kjeldahl process as it is generally carried out in that the entire operation can be conducted in a single piece of apparatus without requiring the attendance of an operator during the process. These advantages are made possible because of the unique construction of the apparatus of the invention and a system of programming the several operations on an automatic or semiautomatic basis. In using the new apparatus, the operator need only to start the analysis and record the final result. Transfer of flasks from digestion rack to distillation column and other manual operations are unnecessary.

In order that the nature of the invention may be more clearly understood, reference is made to the accompanying drawing which is an elevational view partly in section and partly diagrammatic showing the principal structural elements of the new apparatus.

The apparatus consists essentially of a Kjeldahl flask

1, a digestion chamber head 2, a distillation chamber 3 and a condenser 4. The digestion chamber head is preferably fitted with a purge tube 5 disposed as shown to permit introduction of a stream of air. The air entering this tube may come from a source (not shown) at a pressure of one to ten cm. of mercury. A capillary tube 6 admits a small stream of air (0.1 to 1.0 ml. of air per minute) during the entire operation of the apparatus. A larger volume of air controlled by valve 7 may be admitted from time to time as will be described later through tube 8. Tube 9 permits air and vapors from the digestion to leave the digestion chamber head and distillation chamber through valve 10 and exhaust line 11. A capillary tube 12 admits a small quantity of air into tube 9 during the entire operation in the same manner as does capillary tube 6.

A tube 13 to admit water to the digestion flask at a selected period of time through valve 14 is also provided as shown.

The digestion flask 1 is connected to digestion chamber head 2 by means of a ground glass ball joint 15 as shown. These parts may be held together with the aid of a spring clamp (not shown) or by other suitable means.

A relatively small passage 16 connects the digestion chamber head 2 to the distillation chamber 3. In a preferred embodiment of the apparatus, the lower portion 17 of the distillation chamber may be provided with a valve plunger 18 as shown to make a tight seal. This plunger may be made of corrosion resistant steel or may be of a magnetic material covered with an acid and alkali resistant substance such as glass or an organic polymer.

Positioned outside the distillation chamber is a wound coil 19 which actuates the solenoid valve member 18 when electricity is passed through when it is desired to open the passage. This may be in response to a time cycle control mechanism, for instance.

The distillation chamber which is of glass may be constructed somewhat as shown with protruding sections 20 which act as baffles to minimize entrainment of acid or alkali particles through the chamber into the condenser as the operation continues. These also provide a large surface over which alkali may flow and insure that no volatile acid such as sulfur dioxide reaches the top of the chamber.

The distillation chamber is provided with an air line 21 preferably located near the top of the chamber, with control valve 22. This air line may also be located elsewhere between the distillation chamber and valve 28. This purpose is to provide a pressure of air within the distillation chamber greater than that in the digestion chamber to prevent acid fumes from entering the distillation chamber as the acid digestion cycle takes place, and particularly at the end of the digestion period when the digestion flask is cooled. Capillary tubes 23 and 24 are provided to permit a small volume of air to flow into the distillation chamber during the entire operation. The capillary tubes are of such size as to permit 0.1 to 1.0 ml./min. of air to enter and are similar to capillary tubes 6 and 9. They perform the important function of sweeping ammonia out of those sections of the apparatus where the ammonia might otherwise tend to collect. All of the various air inlet tubes may be connected to a common source of air which is at a pressure of one to ten centimeters of mercury.

Also entering the distillation chamber near the upper section is a tube 25 through which sodium hydroxide solution may be admitted through valve 26 at a predetermined part of the analytical cycle.

The condenser 4 is of conventional design and is shown with a water jacket 27 through which water flows to condense the ammoniacal vapors which are distilled from the

Kjeldahl flask. At the lower section of the condenser is a valve 28 which may be closed in accordance with a programming cycle to be explained later.

The distillate containing the ammonia may be collected and titrated in any conventional manner. As shown in the drawing, line 29 carries the dilute ammonium hydroxide condensate to titration vessel 30 which is fitted with a motorized stirrer 31 and means of introducing titration acid 32. An outlet line 33 which may be controlled with valve 34 is provided to allow discharge of the titrated liquor after the titration has been completed.

The Kjeldahl flask 1 may be heated in any conventional manner preferably with an electrically controlled heater 35 of conventional design. This heating element may be pivoted in manner not shown whereby it can be withdrawn from the vicinity of the flask after the digestion in order to permit rapid cooling of the flask by blower 36.

A preferred method of operating the apparatus of the present invention will now be described. A weighed sample of the material to be analyzed for nitrogen, usually one gram, is placed in the digestion flask 1 along with 12 grams of potassium sulfate, 1½ grams mercuric oxide, 15 ml. of concentrated sulfuric acid and a few porcelain chips. The chemicals used throughout the operation are those conventionally used. The Kjeldahl flask is then fitted to the digestion chamber head as is shown in the drawing and fastened with suitable clamps. The heating element 35 is then placed in operative position.

At the beginning of the digestion period, the solenoid plunger valve 18 is in a lowered position effecting a seal between the digestion chamber head and the distillation chamber, to prevent fumes from the subsequent digestion from entering the distillation chamber.

Valve 28 at the base of the condenser is in the closed position. Valve 26 is in a closed position. Air is allowed to flow through valve 22 and through each of the capillary inlets 6, 12, 23 and 24 maintaining a pressure of about three centimeters of mercury in the distillation chamber. Valve 7 is open permitting air to flow through the purge tube 5 at the rate of 1–5 liters per minute. Valve 10 is open permitting air and sulphur dioxide fumes which are formed during the digestion period to be swept out of the apparatus. Valve 14 is closed. The heater is brought into operation, and the digestion of the material in the flask is allowed to take place for a suitable period of time, from about fifteen to thirty minutes, as may be necessary.

After a predetermined interval of time, for instance twenty minutes, the heating element is removed and the blower is turned on and remains on for approximately three minutes or until the flask is cool enough to receive water. The operation of the several valves, heater and blower may be controlled by a synchronous motor driving suitable cams which control the various operations. After a period of approximately three minutes, the blower is turned off by the automatic mechanism, and the heater is returned to operating position. Valve 22 is closed and valve 28 is opened to permit the ammoniacal condensate to run into the titration vessel. Electrical current is turned into the solenoid coil 19 and the valve plunger 18 is raised. The air purge valve 7 is closed as is the sulphur dioxide exhaust valve 10. Valve 14 is open for a period of one minute during which time 150 ml. of water is allowed to enter the digestion flask. Valve 26 is then opened for one minute during which time 50 ml. of twelve normal sodium hydroxide solution containing 5 grams of $Na_2S_2O_3$ is allowed to enter the distillation chamber and flow down the walls thereof into the digestion flask. The large surface presented by the baffled surface of the distillation chamber permits the sodium hydroxide solution to absorb any sulphur dioxide that may have leaked past the solenoid valve into the distillation chamber.

While the alkali and water are being admitted, valve 34 is opened, and the contents of the titration vessel down into the valve of line 33 are allowed to drain away leaving approximately 100 ml. of water or previously titrated solution in the vessel.

As the contents of the digestion flask are brought up to a boil, the decomposed ammonia which is formed upon alkalizing the solution in the flask is distilled off, condensed and collected.

The distillate may be titrated continuously and automatically, or it may be titrated by the operator at the end of the distillation cycle in accordance with usual practice.

The apparatus may also be operated in an alternative manner differing from that first described in that passageway 16 is kept open during the entire operation. If desired, the valve assembly may be omitted from the apparatus entirely. In this alternative method of carrying out the process, valve 7 is closed throughout the entire operation. In fact, the entire assembly of tubes 5, 6 and 8 may be omitted from the apparatus thus simplifying the construction of the apparatus.

In the alternative method, the addition of sample and digestion mixture is carried out in the same manner as previously described. At the beginning of the digestion period, valve 28 at the base of the condenser is in the closed position. Valves 26 and 14 are in the closed positions. Valves 10 and 22 are open. Air is admitted through valve 22 which is open during the digestion stage. The air which goes in through valve 22 moves down through the distillation column, down through passage 16 and out through tube 9, sweeps out the SO_2 formed during digestion. The downward movement of air through the restricted passage 16 prevents any SO_2 from entering into the distillation chamber during the digestion stage. The flow of air is maintained either by pressure in line 21 or by suction in line 11. The digestion is effected as in the previous method by bringing a heater into position under the flask for a period of fifteen to thirty minutes as may be necessary.

After the digestion stage, the heating is discontinued and the flask cooled for approximately three minutes. Valves 22 and 10 are closed when the distillation cycle begins. Valve 28 is opened. Valves 14 and 26 are opened for about one minute. During this time 150 ml. of water are added through valve 14 and approximately 50 ml. of twelve normal NaOH containing 5 grams of $Na_2S_2O_3$ are added through valve 26 into the distillation chamber and flows down into the digestion flask. The distillation of the ammoniacal solution into titration flask 30 is the same as in the previous procedure.

The various operations of turning on and off valves, the heater, blower, titration and so forth can be conducted by hand as described above. It is one of the advantages of the apparatus, however, that these several inter-related operations can be programmed and done automatically. One or more synchronous motors can be fitted with a cam shaft with cams designed to operate switches which turn the several valves on and off at predetermined intervals of time from the time the Kjeldahl digestion flask is fitted to the digestion chamber head until the final titration of the analytical distillate.

I claim:

1. Apparatus for conducting Kjeldahl nitrogen determinations which comprises a digestion chamber, a distillation chamber and a condenser, said elements being joined with a passageway for vapors, said distillation chamber having at its lower end means to open and close the passageway from the digestion chamber to the distillation chamber at desired intervals of time, a purge tube to introduce an air stream into the digestion chamber and an exit tube to remove vapors from said digestion chamber, a capillary tube in said purge tube to permit flow of a small volume of air through said purge tube during operation of the apparatus, a tube permitting in-

troduction of water into said digestion chamber, means in the upper section of said distillation chamber to introduce air into said chamber, a tube for the introduction of liquid into the upper portion of said distillation chamber, and a valve in said passage leading from the condenser controlling the flow of fluids through said condenser.

2. Apparatus in accordance with claim 1 in which the exit tube and the tube for introducing water into the digestion chamber and the tubes for introducing air and liquid into the distillation chamber are fitted with capillary tubes capable of introducing 0.2 to 1.0 ml. of air per minute.

3. Apparatus in accordance with claim 1 in which the means to open and close the passageway from the digestion chamber to the distillation chamber is a solenoid valve.

4. Apparatus in accordance with claim 1 in which each of the valves is controlled by a programming device whereby said valves are opened and closed at predetermined intervals of time.

5. Apparatus for conducting Kjeldahl nitrogen determinations comprising a digestion chamber and a distillation chamber, a passageway from said digestion chamber to said distillation chamber, a solenoid operated valve in said passageway whereby the passageway may be opened and closed, a passageway from said distillation chamber having a valve therein whereby said passageway may be opened and closed and means for maintaining a positive pressure in said distillation chamber whereby digestion fumes from said digestion chamber are prevented from entering the distillation chamber.

6. Apparatus for conducting Kjeldahl nitrogen determinations which comprises a digestion chamber and a distillation chamber, a passageway connecting said digestion chamber and the lower section of said distillation chamber, an automatically controlled solenoid valve in said passageway whereby the passageway from the digestion chamber to the distillation chamber may be opened and closed, a passageway leading from the upper section of said distillation chamber and a valve therein whereby the passageway from the distillation chamber may be closed and means for maintaining positive pressure in said distillation chamber whereby vapors are prevented from entering said distillation chamber through said solenoid valve controlled passageway.

7. Apparatus for conducting Kjeldahl nitrogen determinations which comprises a digestion chamber, a distillation chamber and a condenser, a passageway connecting said distillation chamber with said condenser, a restricted passageway connecting said digestion chamber with said distillation chamber, air inlet means for introducing a stream of air into the upper section of said distillation chamber, air outlet means for removing air and gases from the digestion chamber said means being between the restricted passageway and the digestion chamber whereby air entering the distillation chamber may pass downwardly through the restricted passage and prevent entry of gases from said digestion chamber into the distillation chamber, means whereby the said air inlet may be closed and means whereby the said air outlet may be closed.

8. Apparatus for conducting Kjeldahl nitrogen determinations which comprises a digestion chamber, a distillation chamber and a condenser, a restricted passageway connecting the upper section of said digestion chamber and the lower section of said distillation chamber, a passageway from the upper section of said distillation chamber to said condenser, means for closing the outlet from said condenser, means for introducing a stream of air into the upper section of said distillation chamber and maintaining an air pressure therein greater than in said digestion chamber, means for stopping flow of air through said means into said distillation chamber, means for removing air and gases from the digestion chamber said means being between the restricted passageway and the digestion chamber whereby air entering the distillation chamber may pass downwardly through the restricted passage and prevent entry of gases from said digestion chamber into said distillation chamber, means whereby the said air removal passage may be closed and means for introducing a liquid into the upper section of said distillation chamber whereby said liquid flows down over the inner surface of said distillation chamber through the restricted passageway and into the digestion chamber and means for closing said liquid introducing means.

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MORRIS O. WOLK, *Primary Examiner.*

ANTHONY SCIAMANNA, *Examiner.*