Nitrogen in Sewage.
Abstract.

C. R. Allen.

May 19th, 1885.

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Sewage is a body of very variable and complex composition, containing, as it does, much of the waste matter of a community. It consists of a large amount of water, carrying other material in solution or suspension. Much of the material passing into sewage is good plant food, but it is still an open question as to whether the sewage contains it in sufficiently concentrated form to render its use as a fertilizer available, and avoid the present somewhat dangerous practice of allowing it to run into the nearest body of water. Efforts in this line have been made in England, and, under these circumstances, a determination of the nitrogen present by a technical process might be of value; and a series of analyses showing the present composition of Boston sewage might be interesting compared with those made before the new system went into effect.

The greater part of the nitrogen in sewage comes from the waste material of animal wear and tear, and among these, an important one is urea. This body. on entering the sewage undergoes a fermentation which changes it into ammonium carbonate. which is always found in sewage in greater or less amount.

On treating with alkaline permanganate, a portion of the organic matter yields its nitrogen as ammonia. which is known as "albuminoid" ammonia, the amount of this giving some indication of the nature of the organic matter.

A very minute quantity may also exist in some nitrite or nitrate, and its determination was of interest, owing to the fact that English authorities say that it seldom occurs in that form, while in analyses of Boston sewage, in 1873, it seemed a normal constituent.

Boston sewage is a liquid resembeing dirty water, somewhat opaque, of no disagreeable smell when fresh, but corroding metals when allowed to act for some time, and said to cause poisoning on entering the blood. In addition to the organic matter, chlorine in considerable quantity and traces of phosphorus, sulphuric acid and the alkalis were found. It aslo contains the uric ferment and other microscopic organisms.

The organic nitrogen was first determined by a modification of Kjeldahls process. This process consists in treating the body with fuming sulphuric acid, at a temperature near its boiling-point, for several hours and then adding permanganate to the hot liquid. This converts all nitrogen [except that present as nitric, nitrous &c. acids] into ammonia, which remains in the acid as ammonium sulphate. On diluting, rendering alkaline and distilling in a suitable apparatus, the ammonia is obtained in the distillate and may be determined by titration, precipitation or the Nessler color test, as circumstances dictate.

This process was shown to give reliable results as applied, and was then used in a series of determinations.

By acidifying a portion of sewage to hold the free ammonia, and evaporating to dryness, then treating as above, the total nitrogen was obtained as ammonia.

The presence of small quantities of nitrogen as nitrates or nitrites was determined by freeing the sewage from organic matter and placing a portion in a distilling flask provided with electrodes through which, after all ammonia was expelled, an electric current was passed. It was proved by experiments on solutions of known strength that the nascent hydrogen so formed reduced any nitrogen present in the form of the compounds named, to ammonia, which was distilled and determined as usual.

It being an unsettled question regarding the presence of urea, as urea, in the sewage, or whether the change to ammonium carbonate was a rapid one, a few experiments were made, showing its presence in the samples.

This was effected as follows.

Similar portions in two flasks were boiled to sterilize the liquid, the necks filled with cotton-wool to protect from spores &c. in the air, and, when cool, one was infected with the ferment. The free ammonia in each was determined after a few days, the infected one always giving the greater amount. As this ammonia could only come from the action of the ferment, which is supposed to act on urea alone, the excess of ammonia so found in the infected sample, indicated the presence of urea in the sewage.

Finally a series of determinations were made on samples from the pumping station, showing the constitution of the sewage at that point, which showed the material to be of variable composition, but not differing greatly from that analyzed in 1873.

An average of six analyses gave the following results, expressed in parts in one hundred thousand.

Residue on Evaporation = 44.80
Free Ammonia = 2.26
Album. " = 0.55
Combined " = 2.67

The normal ratio of the albuminoid to the free ammonia appeared to be about 1/3. After a severe storm this dropped down to 1/20 in one sample. The analyses were made in unfiltered samples, hence exibited some differences due to the liquid not being homo-geneous. As the abnormal samples mentioned are included in the averages given, the ratio of the albuminoid and free ammonias does not appear as that which was given for the normal one; one third.

Respectfully submitted
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Thesis.

"Nitrogen in Sewage"

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May. 18th, 1885.

Nitrogen in Sewage.

Sewage is a very complex substance and, consisting as it does of nearly all the waste products of a community, may contain a great variety of substances, both organic and inorganic.

It is very dilute, [Massachusetts Board of Health Report. 1873. Tables of sewage composition &c.] the solid matter remaining on evaporation being only a few grammes to the litre, and hence requires methods of considerable delicacy and accuracy for its analysis.

One of the elements in sewage valuable as plant food is nitrogen. This is, of course, in the combined form and its determination is at times desirable from a technical point, especially now that efforts are being made to utilize sewage as a fertilizer. [Report cited above]

The nitrogen in sewage comes from many sources but that most generally taken into account is the nitrogen contained in the by-products of the animal economy. Of course, [as in Boston] in a large manufacturing city some nitrogen may come from other than animal sources.

One of the important nitrogenous bodies entering the sewage is urea, one of the products of animal "wear and tear", containing a large percentage of nitrogen. This substance, either on entering the sewer, or soon after, is converted into ammonium carbonate by the action of a living organism, a ferment, [Journ. Chem. Soc. April. 1885. Frankland.] or by the possible action of any dilute acid or alkali that may be present. This ammonium carbonate decomposes on boiling, furnishing all, or a part, of the ammonia so determined, called the "free ammonia". The nitrogenous compounds undecomposed by boiling give the "combined" or "organic" nitrogen.

A very minute quantity may also exist as some salt of nitric acid, and also possibly as some nitrite. This quantity is very small. [Bd. of Health. Rep. 1873. Table II.

page 70] the results on some samples of Boston sewage giving from .081 to .005 part in a hundred thousand; and English authorities say that sewage contains no nitrogen in this form. [Ibid. p. 71].

The following experiments bear on the composition of Boston sewage and include the determination of the free ammonia, the "combined ammonia" [or, more accurately, the ammonia given by the combined nitrogen,] by a modification of a well known process, the total solids and also some work on the presence or absence of urea and of nitrates and nitrites.

The sewage samples used in making the application of the process were all taken from the main sewer at the side entrance on E. Chester Park, just below the junction of the Roxbury Sewer, and probably gave a very fair average sample of Boston sewage, as only one sewer [So. Boston] enters below that point.

This sewage is a whitish liquid resembling dirty water - Had no apparant action when litmus paper was simply dipped in. but exercises a very corrosive action on iron &c; it is in long contact with, and will cause a bad sore if it gets onto an abraded place on the skin. [This last statement is the general warning given me by the workmen, and I was shown chains &c. badly eaten by being under the surface.] When fresh, has a slight, peculiar but not offensive odor and on standing deposits a black precipitate and becomes transparent, then turns black and becomes horribly offensive, evolving sulphuretted hydrogen. It contains considerable chlorine, some sulphates & phosphates, and the residue on the filter by filtering a twenty-four-hour-old sample, infected a pure aqueous urea solution with several forms of bacteria &c. as determined by a microscopical examination. [See work on Urea.]

The first work was on the determination of the combined nitrogen: the free ammonia being determined as in water analysis, i.e. by boiling off, required no modification.

I could find but two methods stated for the determination of all the combined nitrogen in water and sewage: the combustion process of Dr. Frankland and the fusion with an alkali, of Dittmar and Robinson [Frankland. page 54].

The combustion process [Frankland's Water Analysis. pp. 67-96.] consists in evaporating a considerable quantity of water with proper precautions, transferring the residue to a small combustion tube and burning it with copper oxide. The gaseous product of this combustion, consisting of nitrogen and carbonic anhydride, is analyzed in a suitable apparatus. [See also. Mass. Board of Health Rep. 1879]

This process is not in extensive use in this country owing to the complex and frangile apparatus and great care and dexterity required: demanding also time and a special training in the use of the apparatus on the part of the analyst. It has, however, the advantage of enabling the measurement of the elements in the gaseous form. hence to a very great degree of accuracy.

It is the standard process however. it was used by the Rivers Pollution Commission in their work. and has been used here to some extent. [Examination of Mystic Water. Board of Health Report. 1879. Supplement.]

This process has been attacked by some chemists. notably Wanklyn, who declares that the errors of manipulation and of reading. exceed the results found, and that the results come in accordance with the expectations of the analyst. rather than in accordance with the amount of the elements existing in the sample. [Wanklyn's. Water Analysis. Supplement]

The fusion process is a modification of Varrentrapp and Will's method. and consists in fusing with caustic potash; this treatment changing the nitrogen, except that in the form of nitric acid. hyponitric acid &c. [by the action of the O from the KOH] all into ammonia. [Fres. Quant. Anal. p. 442.]

For the determination of the nitrogen in water by this process, the residue from evaporation is transferred to a boat, [silver] this placed in a combustion tube and the contents fused with a mixture of lime and caustic potash, or with potash alone, in an atmosphere of hydrogen. The resulting ammonia passes directly into a Varrentrapp and Will's bulb containing very dilute standard HCl. and excess of acid is titrated back by standard alkali as usual. [Frankland's Water Analysis. p. 55]. This process is not generally used here.

The "albuminoid ammonia" process in general use for Sanitary work is valueless as a means of determining the absolute amount of organic nitrogen existing in the sample, as it only gives ammonia corresponding to a portion of the total combined nitrogen. [Wanklyns Water Analysis, p. 31 and Appendix]

A sewage analysis for technical purposes would hardly be performed by either of these methods and the first experiments were attempts to modify some of the processes now used for determining nitrogen "in the wet way", so as to enable their application to the technical determination of the nitrogen in sewage.

The processes now in use for determining the nitrogen in nitrogeneous bodies [other than nitrates, nitric acid &c.] are not very numerous and depend generally on the conversion of the nitrogen into ammonia, and its subsequent determination in that form. Of these the most promising seemed to be some modification of Varrentrapp and Wills process [Fres. Quant. Anal. p. 442] or of Kjeldahl's process, [Fres. Zeit. 1883. p. 366], both yielding the nitrogen as ammonia, and distilling or passing ~~the~~ it into standard acid and determining as usual. As a check the ammonia may be precipitated by platinic chloride and determined gravametrically, as usual.

The first attempts were to modify Kjeldahl's process to sewage analysis. This process seems to be in considerable use for some classes of determinations as in manures, [Chem. News. Volume 50- p.180][J. Chem. News. Vol. 50. p.68] and in urea [Zeit. Phys. Chem. 8.200-213], which are of somewhat similar nature to sewage matter. It is inapplicable to those substances in which the nitrogen is present in the form of a volatile acid, to all the cyanogen compounds, and to oxides of nitrogen. [Thesis. N. Ward. M. I. T. page 48]

Application of Kjeldahls Process to Sewage Analysis.

This process oxydizes in an acid solution and this is a great advantage as an excess of concentrated acid will hold ammonium sulphate through boiling and other hard treatment, thus enabling the retention of all the ammonia until it is ready to be distilled off. There is also small danger of loss by manipulation.

The process has been well tested since it came out and has generally given good results[+]. It was well tested by Mr. Ward. M.I.T. (N. Ward. Thesis. "Nitrogen in the Wet Way". M.I.T. Chemical Library] and gave satisfactory results. Other references on this process have been quoted.

The process as conducted for ordinary analysis will be found in the thesis quoted above and in Fres. Zeit. 1883. p. 366. and is as follows.

The body, which should be in a fine powder is heated as given by the references above, essentially as follows. It is weighed into a flask of one or two hundred cc. capacity and preferably round bottomed. From 0.2 grm. to 1.0 grm.

[+] F. W. Dafert in a paper on this process [Setzungs-ber d. niederrh. Ges. f. Nat. u. Heilk. Bonn. '84 203-206] finds that some nitrogen compounds fail to give satisfactory results, and explains action of acid.

is used according to the amount of nitrogen in the body. if this is approximately known, the object being to neutralize about twenty cm. of standard acid, [1 c.c. = about 10 mg. nitrogen] or one to two hundred milligrammes of ammonia. So large a quantity is however not necessary for the successful use of the process.

The oxydizing mixture is now added. This may be, Fuming sulphuric acid, commercial "C.P." sulphuric acid rendered fuming by addition of phosporic anhydride [P_2O_5], or a mixture of strong H_2SO_4, and a metallic oxide. [The process with this latter mixture is given on page 17-].

The reagents must be as free as possible from ammonia or substances liable to yield ammonia during the process, [i.e. any nitrogeneous bodies]. My experience has been that the reagents never are absolutely pure, and for careful work on small quantities a blank had best be made.

The addition of phosphoric anhydride greatly increases the rapidity of the action and shortens the time required for the process.

The quantity of acid usually added is twenty c.c. but if under 500 mmg. of substance is taken for analysis. Ten c.c. will usually suffice unless the body contains a great deal of carbon. Too great an excess of acid causes the formation of a large amount of acid sulphate when the solution is made alkaline for distillation: this may cause heavy bumping which may result in smashing the flask and spoiling the determination. To prevent this zinc is sometimes used. [Ward. p.51]

The flask is now placed in an inclined position over a low flame [preferably on wire gauge as you have better control of the heating] and gently heated. A violent reaction ensues whose nature is unknown.[+] SO_2 is evolved and the liquid turns dark brown or becomes tarry. Some

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[+] See foot-note on page 10.

ing a good deal and sometimes spurting violently. The flask is inclined to prevent loss and had best be two to three inches above the burner, which is turned on just enough to prevent backing down.

When the violence of the reaction has ceased, [which may be in ten or fifteen minutes] the flame is increased and the contents of the flask brought to a point just under the boiling-point of the acid. This point is indicated by a slight bumping and can be hit and retained without any special care or attention.

As the heating continues, the color of the liquid becomes lighter and finally a light yellow or "rhine-wine color". This indicates the end of the reaction for the acid. The time required to obtain this color is generally called two hours, with H_2SO_4 and P_2O_5; but varies greatly with the substance undergoing analysis, the amount used &c. Uric acid gave a clear yellow in two hours, but excrement was not the same color after eight hours heating.

During the heating the heat should be high enough to cause the slight bumping mentioned, as otherwise low results may be obtained.[†] It is not advisable to allow the acid to come to a violent boil either, as a slight loss of ammonium sulphate may result. A momentary boiling however does not appear to affect results.

As a rule it does not appear absolutely necessary to heat until the light color is obtained, though it is certainly more sure, and should be done if time allows. Generally however two to three hours heating gives a light brown solution, perfectly clear, and containing no suspended matter.

In many compounds the acid alone forms ammonia from the nitrogen present. [Fres. Zeit. 1883. p. 366.] In the gluten-proteins, uric-

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[†] Ward's analysis of uric acid.

acid. asparagine &c. the action is complete at this point: [i.e. all the nitrogen is converted into ammonia] most members of the fat series yield 90%-95% of their nitrogen by this action. [of H_2SO_4]. while aromatic bodies are but little affected.

The final oxydation is effected by means of permanganate, and no other oxydizing agent has been found to take its place. It must be finely powdered[†] and is added a little at a time from a glass tube having one end covered by a netting, which, by gentle tapping, delivers a fine continuous stream. [as recommended by Kjeldahl] or, in default of this arrangement, from the flat end of a stirring-rod, spatula, or any similar article.

The process is as follows- The lamp is removed and the permanganate added direct-

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[†] Czeczetka adds the permanganate dissolved in strong sulphuric acid. [Monatsh. fur. Chem. VI- pp. 63-64]

ly to the hot acid. A very violent action ensues, brown fumes are given off, accompanied by black flakes and a choking gas. Flames are often seen in the flask. Many analyses have shown beyond a doubt that during this action there is no loss of ammonia. [Kjeldahl.]

The addition of sufficient permanganate is indicated by the turning green of the solution. [or bluish-green if P_2O_5 be used], giving a solution of ammonium sulphate in an excess of concentrated acid.

The flask is now carefully warmed for ten minutes. any boiling causes a loss of ammonia.

When the contents of the flask are perfectly cold it is washed into the distilling flask. [See figure of apparatus] with about 500 c.c. of pure water, giving a brown solution.

An excess of alkali is now added and the ammonia distilled off as usual. Too great an excess of alkali causes foaming and so may spoil the determination. It also may cause heavy bumping and break the flask. [The addition of a few pieces of Zn will partially prevent this.]

The ammoniacal distillate is distilled into standard acid and excess titrated for as usual.

Among the modifications of this process is one consisting in using a metallic oxide as an oxydizing agent.

Dr H. Wilfarth [Chem. Cent. Blatt. January 14th 1885] greatly diminishes the time required by using a metallic oxide as an oxygen carrier between the acid and the body undergoing oxydation, it playing somewhat the same part as the nitrous fumes do in the manufacture of sulphuric acid.

He made a series of determinations showing the time required when various oxides were used. He says:—

" I proceeded as usual --- by treating a gram of the body under analysis with twenty cm. of a mixture of phosphoric anhydride [P_2O_5] and sulphuric acid. and adding a gram of the oxide to be tested. Heating was as usual over a wire gauze and a free flame.

The flask is warmed one quarter hour over a low flame and then one quarter hour nearly to boiling. The time given is from the time the heating began to the time when the liquid turned yellow."

The oxydizer was a mixture of:—

800 cm. concentrated H_2SO_4.
200 " fuming H_2SO_4
100 gm. P_2O_5.
Use 20 c.c.

Oxide	Diffusion. Resid.	Clover.	Peas.
Fe	2.00 hours.	1	1
Hg	1/2 "	1/2	—
Mn	2 1/2-3 "	3 1/2	3 1/2
Cu	1 1/4 "	1 1/2	1 1/4
None	4 "	3 1/2 - 4	3 - 3 1/2

The table given is part of the one given in the paper. It will be seen that a very material saving of time is effected. He tried a large number of bodies among which were:- Leather Scraps. Peas. Rape-seed cake. clover. meat scraps. fish guano. dried blood. egg albumen &c. and claims to save 50% to 60% of time.

In a later communication he recommends as best of all, HgO. [Chem. Cent. Blatt. Feb. 18. 1885].

This modification was not tried as fuming sulphuric acid and phosphoric anhydride alone, take up sewage residue in a comparatively short time, but, speaking from my experience with Kjeldahl's process proper, it would, if it saves the time stated, be exceedingly valuable. as the oxydizing is the part of the process requiring time, the other portions (distillation &c.) being short operations.

I made a few analyses by this process to render myself familiar with the process its.

self, apparatus &c.

The apparatus used was similar, in a general way, to that used by Mr. Ward, and is shown by the figure.

(a) is the distilling flask, holding about a litre and connected as shown with

(b) a second flask to hold any alkali passing from (a). Later a "boiling flask was substituted for the ordinary one shown.

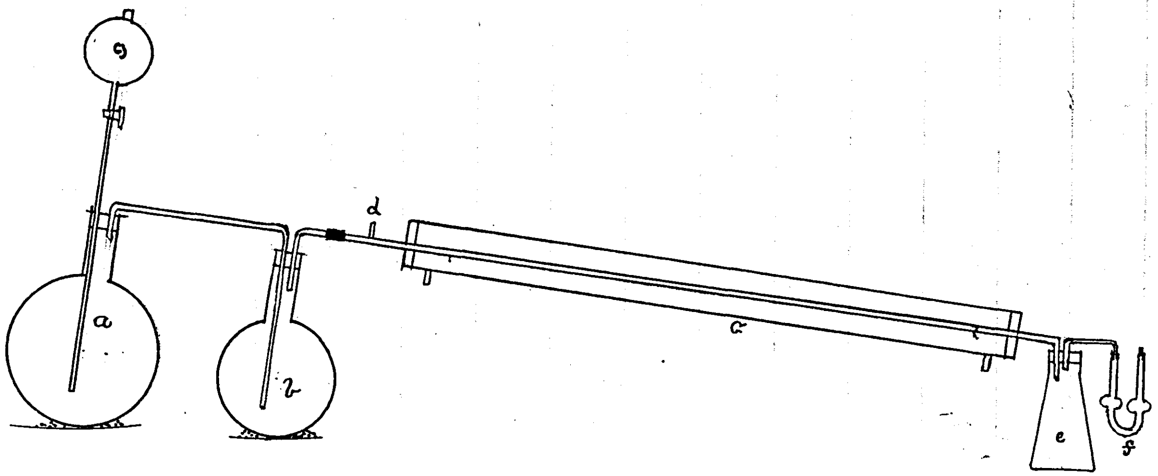
(c) is a common condenser provided with a tube (d) for washing out.

(e) is a receiver.

(f) is a Peligot tube, small size.

(g) is a separation funnel for adding the alkali, holding 250 c.c.

(a) and (b) were heated on two sand-baths as usual, and (b) and (c) were connected by a rubber connector to prevent rigidity and chance of breakage. Common corks were used. The end of the condenser was bent as shown, and the condenser was formed of two pieces of glass tubeing and rubber corks, being rather longer


g
d
a
b
c
e
f

than usual.

The acid solution from oxydation is put into (a) with about half a litre of pure water, a little water is put into (b), generally enough to cover the end of the delivery tube from (a), a measured quantity of standard acid is run into (e) from a burette and a few c.c. of water, containing 0.1 to 0.3 c.c. standard acid and a drop of indicator, is put into (f).

Acid is usually put into (f) and my reasons for departing from the regular usage were these:-

When the distillation is nearly finished, heavy bumping may occur and a part of the liquid in the Peligot tube may be driven out by some sudden "bump", if great care is not exercised. [The addition of Pt or Zn tends to keep this however, as I found in sewage work.] In my analyses by this process, I never knew enough ammonia to get to the Peligot tube to neutralize the little acid added, passing over, as it does, with water and steam.

Hence the Peligot tube serves its purpose as well when filled with acidulated water; that is, to give assurance that no ammonia has passed out of the apparatus, this being the case as long as the indicator remains red. But if a little be thrown out, if the tube is filled with standard acid, the determination is ruined: while if acidulated water be used, the loss of a centimetre or so is imperceptible in the titration, representing, as it does, only a fraction of 0.1 or 0.2 c.c. of acid.

For rendering solution alkaline I used a saturated solution of 98% lye. A sufficient quantity of this to render the contents of (a) alkaline [as determined by previous experiment] was put into (g) and, when all was ready, allowed to run into (a). If, during this, bubbles of air pass through (e), the apparatus is known to be tight. The reason for adding the alkali thus, by means of a separating funnel, is to prevent the possible loss of ammonia during the heating consequent on the addition of the strong alkali to the acid.

The flasks (a) and (b) are now brought to a boil. The steam and ammonia pass into (b), [the water in which retains any alkali &c. that may distill over] and thence into (e). When all ammonia has passed over, which will require distillation of at least one half of the contents in (a), the water in (b) is violently boiled for a few moments to expel any traces of ammonia, the wash-bottle connected with (d) by a short rubber tube, and the condenser washed out into e. The contents of (f) are poured into (e), washings added, more indicator added if necessary, and the distillate titrated as usual. It is a good plan to distill over another portion and see if any more ammonia comes over as the last traces come off with some difficulty. This can be effected by again adding an excess of acid to (e) and, after distilling, titrating again, this enabling any further amount of ammonia to be determined.

The acid used was the usual "standard" sulphuric and was standardized against pure sodium carbonate as usual. The alkali corresponding was ammonia. The values were.

1 c.c. H_2SO_4 = 1.006 c.c. NH_4OH

1 c.c. NH_4OH = 0.993 c.c. H_2SO_4.

1 c.c. H_2SO_4 = .00961 grm N.

Indicator used was "Orange III."

Blanks were made showing reagents to be practically free from ammonia.

A solution of ammonium sulphate was made up and aliquot parts rendered alkaline and distilled in the apparatus, the distillate being titrated.

Theory was 21.21% N.

Found. I. 21.44% N

II. 21.24% N.

This showed standardization &c. to be correct, and I now tried uric acid, taking from 0.6 to 0.8 grm. With 20 c.c. H_2SO_4 and 5 grm. P_2O_5 this dissolved easily in two hours, giving a light yellow solution. Later I used

10 c.c. H_2SO_4. The results came high, running from 34.7% up to 36.6% in three analyses. I then left out the P_2O_5 and found the uric acid went up perfectly well in sulphuric acid alone. This determination gave 33.24% Nitrogen against 33.20% theory. These high results were probably owing to the lack of sufficient water in the wash flask, this allowing some alkali to pass over. At least, this is the only explanation I can give. The last determination was distilled more slowly and more water was in the wash flask. Uric acid has often been determined by this process and has given good results. [Ward. cc.]

The next substance tried was sulphurea, as a somewhat similar body to that just tried. It was a crystallized sample, and, by the formula $[(NH_2)_2C{=}S]$ contained 36.84% N. It dissolved as usual, giving a deposit of sulphur around the neck of the flask. 0.471 grm. was taken and in two hours a clear solution was obtained. This gave 36.86% N.

The question having been raised as to whether it was necessary for the application of this process to have one or more hydrogen atoms attached to the nitrogen atom, a sample of azo-benzol, $C_6H_5\cdot N=N\cdot C_6H_5$ was tried, as a body having no hydrogen connected with the nitrogen. 0.538 grm. was dissolved as usual, giving a tarry liquid which required long heating to bring it to the proper color. During the distillation the apparatus broke down near the end, and the determination was lost. By titrating what had come over I found 12.82% N against theory 15.38% N. Though not absolutely correct, this would indicate that the nitrogen was changed to ammonia as usual.

I now tried material of varying composition, more nearly allied to the material in sewage.

Horse-dung was dried at 100°-105° [during which much ammonia &c. came off.], powdered and sifted, and two samples of about half a gram each were dissolved. They required long heating and much SO_2 was evolved. H_2SO_4 and P_2O_5 were used, and heating for eight or ten hours was required. On oxydation and distillation gave.

I. 3.12% N.

II. 3.16% N.

As a check a combustion was made of a portion of the same sifted sample. giving 3.28% N.

Human excrement dried at 100°-105°. powdered and sifted gave by this process - 6.20% while a combustion gave 6.08% N.

Finding that the process gave good results on the materials that form a portion of the nitrogenous material in sewage, I now determined to attempt the application of the process to that article. While waiting for a sample of the real article I worked on an artificial sample which had a somewhat similar composition.

Application of the process to sewage.

A mixture having a resemblance to sewage was made up. It consisted of excrement, ammonium carbonate, sulphate of potash, phosphate of sodium, sodium chloride and water and was treated by boiling off the free ammonia and evaporating the residue to dryness. This residue was analyzed by Kjeldahl's process as usual. The mixture contained approximately 240 mmg. to the litre. [of nitrogen]. I attempted to titrate as usual. but found that the acid was too strong, giving varying results. By using more dilute acid and alkali, titration might be employed. but I preferred to use Nesslerization. as that method admits of the use of much less material. and also dispenses with the second distillation flask, as the presence of a trace of alkali in the distillate for Nesslerization does no harm. Finding however that the solution was too strong to represent sewage, and that the sediment present prevented getting two samples alike. I made up 50 c.c. to 250 c.c. and filtered.

The points to be determined were:-

(1). If a sample would give similar results on a series of determinations.

(2) If the residue from direct evaporation on the water-bath, and that from the evaporation of the residue left in the flask after boiling off the free ammonia, would give practically identical results.

Portions of 50 c.c. were put into 250 c.c. distilled water, the free ammonia boiled off, the residue evaporated and treated as usual with fuming sulphuric acid, for Kjeldahl's process. Similar portions were evaporated on the water bath and treated the same way. I found results as follows:

From direct evaporation - 0.96, 0.90, 0.98, 0.94.

From estimation of free ammonia - 1.00, 1.00.

[Parts in 100,000.]

The distilled water gave a little ammonia and that may account for the difference. [See a paper handed into the San. Lab. in December 1884.] between the two latter cases and the four former ones.

Finding the samples of sewage could now be obtained, I tried the process on that.

The following points were to be proved.

(1.) That the residues from direct evaporation and boiling gave practically the same results.

(2) That the results found in any case were the true amount of nitrogen present in the sample.

(3) That the process gave results sufficiently accurate for technical work, and this being so-

(4) That the process did, or did not give results accurate and delicate enough for Sanitary work on water and sewage.

(1). This was proved as follows- portions were oxydized from direct evaporation and after boiling off the free ammonia. The following results were obtained-

From free ammonia det.	3.75 3.50 3.75
From direct evaporation.	3.70 3.75 3.75 3.80

[Parts in 100.000.]
[Ammonia.]

This shows that the two methods give practically the same results.

(2) A portion of the same sample [250 c.c.] was evaporated on the water-bath, the residue rubbed up with copper oxide and a combustion made. The average nitrogen found had been 9.20 mmg. to 250 c.c. The combustion gave 9.14 mmg. [of N]. It will therefore be seen that this process [Kjeldahls] gives all the organic nitrogen in the sample as ammonia. The traces of ammonia in the reagents used [notably in the distilled water],

would naturally run a little over the true amount.

(3) I made a series of determinations on the same sample. which was kept in a glass stoppered battle, so that the total nitrogen could change but little from any decomposition taking place on standing.

F.	C.	T.	
2.80	5.00	7.80	Had stood two hours.
2.80	5.10	7.90	
4.20	3.75	7.95	" " 24 "
4.50	3.50	8.00	
4.50	3.75	8.25	" " 48 "
4.25	3.70	7.95	Had stood three days
4.20	3.75	7.95	" " " "
——	3.75	——	On direct evaporation
——	3.80	——	" " "

[Pts in 100,000.]

F. is free ammonia. C. is combined ammonia. T. is total ammonia. Results expressed in parts per 100.000.

The change on standing apparently indicates a further production of ammonia and, while the two factors F. and C. change, it will be seen that T. [F+C.] changes but little.

The free ammonia was determined as usual by distillation, using 25 c.c. of the sample.

The "combined ammonia" was determined by the evaporation and treatment of a similar quantity

The "total ammonia" was determined by acidifying, to hold the free ammonia, evaporating and proceeding as usual.

On trying a series of analyses, using the common distilled water the results did not check, on using varying quantities of that substance. I therefore prepared a lot of distilled water free from ammonia and, on an analysis of a sewage sample using this water found-

Free ammonia	4.50
"Combined" "	3.20
	= 7.70
"Total" "	7.76

[Parts in 100.000]

This work showed that the process as applied by the methods used, gave fairly accurate results for the nitrogen in the residue on evaporation, and for the "total nitrogen."

As a result of this work the following process was decided on as giving the best results in the least time.

For "combined ammonia"

A measured quantity of the sewage is put into a small, flat-bottomed flask holding about 100 c.c., a little carbonate of soda added and the liquid boiled to expel the bulk of the free ammonia, then evaporated on the water-bath in a current of dry air. This did not require over two hours to evaporate 25 c.c.

When the sewage has evaporated to dryness the fuming sulphuric acid was run in from a glass stoppered burette having a chloride of calcium tube inserted in the top to prevent any change in the acid by hydration. From two to five centimeters were used. The subsequent treatment is as usual, generally giving the yellow color in an hour.

A few grains of permanganate are enough, and are added as usual, to the hot acid.

The distilling apparatus was merely a flask and a condenser, as the wash-flask and Peligot tube of the regular Kjeldahl's apparatus were not required.

The contents of the little flask were washed into the large one as usual, but, as the 98% lye appeared to contain traces of ammonia, stick potash was substituted. Generally five to six gram was used, giving a slightly alkaline solution.

The alkaline solution appears to act on the corks, giving ammonia, and, if it gets onto the cork, a new one should be substituted. An excess of alkali is also to be avoided for reasons already given.

The contents of the flask were boiled, [a 250 c.c. flask being used for a reciever] till all ammonia had passed over, distillate made up to a definite volume and aliquot parts nesslerized.

For "total ammonia"

A measured quantity is acidified with a few drops of sulphuric acid, and treated as above. From 25 to 100 c.c. should be taken according to the "strength" of the sample. If over 25 c.c. be used, concentration on the water-bath and subsequent transference to the evaporating flask is the quickest method.

For "free" and "albuminoid" ammonia

This process was conducted as is usual

for a water analysis. The only point of divergence was that, owing to the large amounts present, it was, of course necessary to greatly dilute the sample. My usual method was to put half a litre of pure water in the flask, add ten c.c. of the sample and treat as usual for the free and albuminoid ammonias. If sodium carbonate is added here, it must be added if the "combined" ammonia is determined, as directed, in the account of that process, in order to obtain concordant results.

Of course, if albuminoid ammonia is not required, the residue in the flask can be evaporated and the "combined" ammonia determined there, but this process is less satisfactory and requires more time.

The reagents used were what would be commonly called "free from ammonia", and were so, for any ordinary work.

But for such small quantities of material as were used for these determinations, the traces of ammonia given by blanks were a sufficiently large factor to have a slight, but perceptible influence on the results, and, after the process was found to work, these reagents were tested in order to obtain results as accurate as possible, and also with a view of applying the process to a rough determination of the organic nitrogen in water, where this correction would form an important one.

The reagents used were distilled water, sulphuric acid, phosphoric anhydride, permanganate and alkali [KOH or NaOH.]

Distilled water was freed by double distillation from battery fluid. [$H_2SO_4 + K_2Cr_2O_7$]

Sulphuric acid:— Samples of fuming H_2SO_4, "C.P." acid &c. all gave ammonia on treatment for a Kjeldahl blank. Of course, any nitrogenous matter in the acid was as bad as ammonia. The acid that had been used contained very little, but no more of that lot could be obtained. Finally a bottle was taken, marked, and two blanks made.

I found 1 c.c. = .00007 grm. ammonia.

The distilled water was freed by redistillation as mentioned.

The soda lye contained traces only and the other reagents were free.

(4) Having now means for an approximate correction for reagents, I made a few determinations to show the accuracy of the process as applied to the determination of the nitrogen in potable water.

Cochituate water was drawn into a bottle and portions of this sample were concentrated and treated as usual.

By the Board of Health Report - 1873. the organic nitrogen in this water is given as 0.054 part in 100,000. Since then the organic matter in the water has unquestionably grown worse, but I could find no figures. [I have given in the Report were by Franklands' process.]

The following results were obtained.

Sample	of	1000 c.c.	gave	0.107 part.
"	"	500 "	"	0.067 "
"	"	250 "	"	0.121 "
"	"	250 "	"	0.125 "

The sample giving 0.67 part had stood four days in the bottle. This would naturally come somewhat low. On the whole, however, fairly concordant results were obtained, as may be seen, indicating a doubling of the organic nitrogen since 1873.

Of course these results are not absolutely correct, owing to the large correction required, but, if pure reagents can be obtained, it might be made useful.

A comparison of the albuminoid ammonia and these results may be of interest.

	Album.	Organ.	Ratio.
Sample I	0.23	0.152	0.15
" II	0.23	0.150	0.15
" III.	0.23	0.147	0.17

[Parts in 100.000.]

In this water the ratio seems to be very low. As will be seen later, in sewage, the ratio tends to run higher, as might be expected.

In relation to Kjeldahls process I would mention the fact that an attempt to substitute phosphoric acid for sulphuric acid was a failure. The substance tried was uric acid. It gave a carbonaceous mass but did not appear to have any oxydizing action.

Thus, of the propositions on page 31, 1, 2 and 3 were shown to be true, while (4) was shown probably possible with pure reagents, which were, at that time, not to be obtained.

As mentioned in the first part of this paper; beside the Kjeldahl process, I hoped to be able to apply some modification of the processes depending on the action of fused potassic hydrate, of these, the most suitable appeared to be the process of H. Bungener and L. Fries. [Wagners Jahr. 1883. p. 465 and Zeit. fur d. ges. Brauwesen. 1883. s. 40.]

An attempt to apply Bungener & Fries's process to Sewage Analysis.

This process is used by the originators for determining the nitrogen in such liquids as beer, and is also used for grain &c. The principle of the process has already been spoken of. [Page 6]

The apparatus consists of a flask (a) made out of copper and having the neck surrounded by a water cooler. [See figure 2] This is connected with a condenser and a U tube containing standard acid. The flask is connected with a

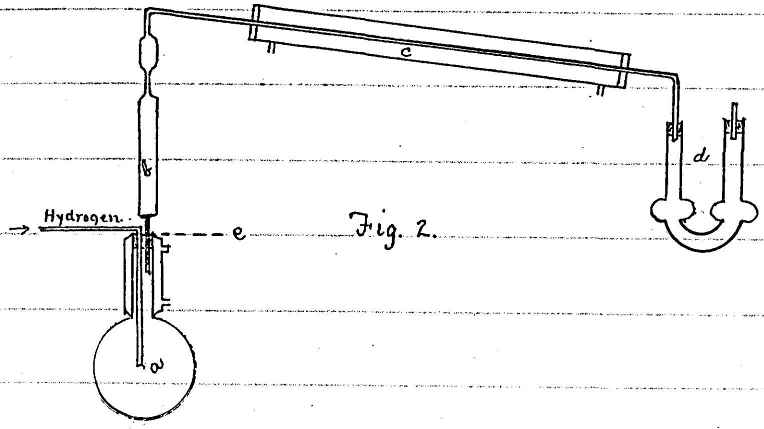


Fig. 2.

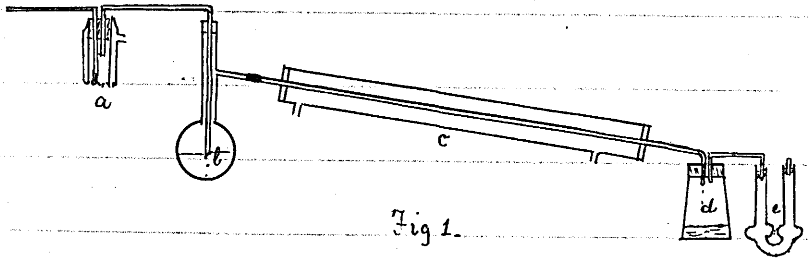


Fig 1.

drogen generator as shown.

The process is as follows.

For beer, 50 cm. is put into the flask, 20 gm. caustic soda added, and the flask connected with the rest of the apparatus.

On heating, a part of the steam condenses in (b) [the stem (e) being filled with glass wool] and forms a layer of boiling water, through which the steam, and later, the products of the fusion, pass. During the distillation of the water a stream of hydrogen is run in. When the contents of the flask are dry, the heat is increased and the flask heated to dull redness, care being taken to keep the cooler full of water. After heating half an hour the lamp is removed, and the stream of hydrogen increased so as to prevent any sucking back. After the flask has cooled to 100° C. the Hydrogen is shut off and the water in (b) allowed to run into (a). The operation is then repeated, fusing ten or fifteen minutes.

The acid is titrated as usual.

After many ineffectual attempts to get a bulbed tube to act as described, trying a bead-tower &c., a "boiling flask" was substituted as in Fig. 1., which served the same purpose. [i.e. washing the ammonia].

I now tried some bodies of known percentages - as follows.

The substance was put into the flask, "soda lye" added, 50 c.c. pure water put in and the process conducted as described above, allowing the water from the wash-flask to suck back for the second part of the operation.

The first analyses were made [using the apparatus as in Wagner. Fig. 2.] on uric acid. Later, I used the modification Fig. 2., as the distillate of 50 c.c., on getting into the Peligot tube in addition to the liquid already there, cansed to great a pressure. Beads were also put into the Peligot tube on some occasions, with no apparant advantage. The gases were allowed to bubble very slowly [one bubble per second] through the acid, some hydrogen being required to prevent any sucking back. The usual acid was used, [1 c.c. = 9.60 mmg. N], and the amounts of alkali varied from ten to twenty grammes.

I. Uric acid gave. Theory [33.20%]

a) 31.90% b.) 30.80% c.) 30.50%

II. Horse dung. [3.2%] gave 1.40%. This was fused till no more ammonia came over.

III. Excrement - [6%] gave -

a) 4.0% b.) 4.90.

This gave a peculiar, fatty looking substance which distilled over, and whose composition is unknown. It had a bad smell and appeared to be insoluble in alcohol, ether, water, and dilute hydrochloric acid. A qualatative test was tried and it apparantly contained no nitrogen.

IV. Using 1/10 acid. [1 c.c. = 0.96 mmg. N] and 100 mmg uric acid I found 18% N.

V. Sewage containing about four parts. [Kjeldahl] being the artificial sample already described, gave with same acid

a.) 3.6. b.) 3.6 c.) 3.2 d.) 3.1

VI. Sewage containing 7.5 parts gave.

a.) 5.5 b.) 6.0.

These determinations were carefully made, and, as a result, I considered the process as unsuited for the small quantaties of nitrogen handled in sewage analysis.

The objection seems to be in the difficulty of retaining all the ammonia formed. This comes through the long apparatus mixed with considerable hydrogen, and requires strong acid to hold it. It is noticeable that uric acid when comparatively large quantaties were used, gave results nearly up to the theory, while on using acid 1/10 strong and 100 mng., only about half the ammonia was retained.

If titration be employed for sewage work, dilute acid must be used, and this dilute acid seems unable to hold all the ammonia, as the mixed gases pass through it. Some hydrogen must be always run in, to prevent any sucking back into the copper flask, which will certainly result in an explosion.

The process is founded on a correct action and would doubtless give good results for the purpose it was intended for, but seems incapable of the delicacy required for this sort of work, and the attempt to apply it was, therefore, abandoned.

Determination of Nitrogen
as nitrates and nitrites.

As before stated, minute quantities of nitrogen may exist in sewage in combination with oxygen, as nitrous or nitric salts. The very small proportion of these present require. for their detection and determination, the same processes in use in water analysis.

The existance of these nitrous compounds is not always apparent. English authorities consider fresh sewage as seldom containing them: some analyses of Boston sewage a few years ago, give from 0.009 to .081 part [in 100.000] of nitrogen as nitrates and nitrites. [Board of Health Report. 1873. p. p. 70. 71.] the amount increasing rapidly on standing. From the analyses of English sewage given in the same report, [p. 43] it will be seen that little nitrogen appears in that form.

The methods in use for detecting nitric and nitrous acids in water. generally depend on the reduction of the oxy-group by nascent hydrogen, the nitrogen forming ammonia which is determined as usual.

This hydrogen is provided by acting on aluminium with strong alkali, or by galvanic action between two dissimilar metals.

There is also a method founded on the action of nitrates on indigo, which serves to determine both acids. [Franklins Water Analysis. pp. 38 and 39]

The best method founded on galvanic action that was in use in the laboratory, was the "zinc-copper couple". [This process is given in the notes of the Sanitary Laboratory, in Franklands Water Analysis. p. 110, and in Cairns Quantitative Analysis, p. 181.]

Griesses process [Frankland. p. 48 and 111] for the detection of nitrites, is strongly recommended by Dr. Frankland, and is stated to be capable of detecting one part of nitrous acid in thirty million parts of water. It depends on the color formed by metaphenylenediamine with nitrous acid.

It was hoped that, if the presence of nitrogen as nitrates and nitrites could be determined, that the nitrogen as nitrites could be also obtained.

As the processes given do not give entirely satisfactory results in inexperienced hands, I determined to try the well known reducing action of the galvanic current.

The apparatus consisted of a small flask, [150 c.c.] connected with a small condenser, and having two zinc electrodes running down to the bottom.

With this apparatus tests were made using a dilute nitric solution. [1/32 normal] and a current from an eight cell trough battery. [Grove]

The results found in testing this process are given in the following table: the first column giving the ammonia corresponding to the KNO_3 put in, the second, the ammonia found, and the third, the difference.

I	0.000265	0.000260	0.000005
II	0.000265	0.000255	0.000010
III	0.000132	0.000130	0.000002
IV	0.000528	0.000520	0.000008

[Figures are decimals of a g'm.]

The process used was to place 100 c.c. water free from ammonia in the flask, add a piece of freshly ignited lime, and distill, until a distillate free from ammonia was obtained. The battery circuit was then closed, the water kept at just a "simmer" during half an hour, during which time not over twenty cm. should distill over. The water is then brought to a boil, and evaporated to dryness, or nearly so, and the distillate nesslerized as usual. When the flask has cooled 50 c.c. pure water is added: and, after repeating this operation, the distillate is again tested. If any amount of ammonia is found in the second distillate, a third repitition may be necessary. In my experiments however, two boilings were sufficient.

This process has the disadvantage of needing a battery, but has the advantage of needing no especial care during the analysis, and of requirement of no reagents, and of no introduction of anything into the flask during this process.

The electrodes were about 1 cm. apart, of zinc, and later of platinum, which I should prefer.

This process was now applied to sewage as follows-

A sample of sewage, twenty-four hours old, was taken. 50 c.c. was put into 100 c.c. pure water, all ammonia boiled off, and this process conducted as described. Ammonia, corresponding to 0.0098 part N as nitrates and nitrites, was found. This is about what was found in Boston sewage in 1873. [Board of Health Rep 73]

Having found great difficulty in getting a distillate free from ammonia, owing to that given by the decomposition of urea, after several trials, the following modification was adopted.

250 c.c. sewage was evaporated by boiling, in an evaporating dish with an excess of lime, the residue ignited for half an hour, the process repeated, with addition of more water, the residue taken up, and filtered into the boiling flask. This gave a distillate which was soon free from ammonia, and the process was then conducted as usual. From a fresh sample, I found ammonia corresponding to 0.0003 part of N in 100.000.

I regret, that, owing to the miscarriage of samples, I was unable to make a series of determinations by this process, and from the same cause, was unable to do any work on the nitrites, to which it was intended to apply the iodo-zinc method or Griess color test.

Urea in Sewage.

It is a well known fact that urea in large quantities passes into the sewage, composing, as it does, a large percentage of some waste products of animal economy.

It is also well known, that by the action of a "ferment," and also by action of dilute acids and alkalis, if any such be present, it is converted into ammonium carbonate.

$$\begin{matrix} NH_2 \diagdown \\ NH_2 \diagup \end{matrix} CO + 2\,H_2O = \begin{matrix} NH_4 \diagdown \\ NH_4 \diagup \end{matrix} CO_3$$

The ammonia so formed, appears in the "free ammonia" determinations. Some points in regard to this are not settled and it was hoped to throw some light on them. The object was, if possible, to show whether any urea existed, as such, in the sewage at the point where the samples were taken, or whether, by the time it had reached this point it was entirely hydrated. In other words, whether the change required a short time, or a long one, and whether or not it was finished at that point.

There were some circumstances pointing to the presence of urea, but not proving it: these were.

(1.) The decomposition on standing: from the analyses made I select a sample.

	F	C.	T
1" day	2.80	5.00	7.80
2" "	4.20	3.75	7.95
3" "	4.40	3.50	7.90

[Pts in 100.000.]

Thus, during the time that elapsed after the sample was taken, [three days] during which analyses were made, a decomposition, resulting in a change of "ammonia" from the form of "combined" to "free", took place. This change would indicate the presence of some easily decomposed body, and urea is the most probable body that would be present.

(2) It was also noticed that the "albuminoid ammonia" changed little or none on analyzing a sample during a few days after standing. As urea is unaffected by alkaline permanganate, the "ammonia evolved by this reagent would be independent of any decomposition of urea, if such be present, and this state of things. [i.e. change on standing having no effect on albuminoid ammonia] would indicate the presence of some body acting as urea does. [i.e. decomposing on standing, and giving no "albuminoid ammonia."]

(3) In Frankland. [p. 26] the statement is made, that a long continued evolution of free ammonia, indicates the decomposition of urea, by the boiling. Sewage apparantly gives ammonia as long as

it is boiled, [of course in very minute quantity after the regular "free ammonia" is driven off.] and this may be considered as another indication.

Of course, the urea, if present, is so in very small amount, and would require a process of considerable delicacy for its determination. The decomposition referred to, if at all due to urea, would only indicate some 25 mmg. urea per litre, and considering how easily it is decomposed it was doubtful if any quantatative process would show it.

An attempt was made to use the process for urea in blood [Phys. Chem. of the Animal Body. p.66], being Picards method as modified by Messiner and Schneidler. By this process, a quantity of blood is boiled with sulphuric acid to destroy proteins, filtered, filtrate concentrated, and treated with Barium hydrate, and excess of Ba removed by cautious addition of sulphuric acid. Filtrate is evaporated to a syrup, and treated with absolute alcohol. filtered. filtrate evaporated and residue dissolved in water, giving a yellow solution and an acid reaction. Solution of mercuric nitrate is added and a white ppc. falls: this is filtered off and filtrate rendered alkaline by Na_2CO_3 or BaO_2H_2- and more mercuric nitrate added, till a drop of the mixture gives a yellow color with Na_2CO_3.

The white ppc. is diffused in water, mercury ppc. by H_2S, HgS filtered off, and filtrate evaporated with a drop of pure HNO_3. Under the microscope, characteristic crystals of nitrate of urea separate out.

This process was tried on sewage with no satisfactory results owing to the readiness of the impure urea to decompose on heating, thus preventing any concentration. On trying 100 c.c. a ppc. was obtained by mercuric nitrate, but too little to filter. On decomposing without filtering and evapcoating as given obtained. among crystals of NaCl, NH_4NO_3 &c. were a few bearing a great resemblence to those sought, but as they were too few to identify and there was no way of separating them, I was forced to abandon this process.

The next attempt was to utilize the action of the ferment.

This ferment is an organism belonging to the class of aerobes. is 0.006 mm. long, 0.0008 mm. broad, and bears a heat of 90°C. Changes to elliptical spores bearing a heat of 96°C. [Frankland. "Chemical changes in their relations to micro organisms" J. Chem. Soc. April-1885. p.178] The same authority states that they are lively as long as any urea remains and then become dormant, and probably, dead.

To show the presence of this organism in sewage, a sample that had stood twenty four hours, was filtered, the filter paper being dried and preserved. A solution [of C.P. distilled water] of urea, which gave no test for ammonia, was made up. In a portion of this, a piece of the filter paper was placed, the solution being in a glass stoppered bottle. After a few days, the liquid became turbid, and, on opening the bottle the smell of ammonia was apparent. The solution also gave a decided test for ammonia.

A drop of this solution was put under the microscope, and [1/8 in. objective] showed living bodies in plenty. Several species of Bacteria and some bodies corresponding to the organism sought.

To detect the presence of urea the following process was tried.

A similar portion of sewage was placed in two flasks, boiled to sterilized and drive off free ammonia. While boiling, the necks were stopped by cotton-wool, and the flasks were allowed to cool. By means of a glass rod, which was dipped in the infected solution of urea, and then in the flask] one flask was infected with the ferment, the other remaining sterile. After a few days, the flasks were opened and 10 c.c. from each tested for free ammonia by distillation. Two experiments by this process, gave in each case much more ammonia in the infected, than in the sterilized portion, thus showing the presence of urea in the sewage.

Experiment I. The sewage was boiled with BaO_2H_2, filtered off, and equal parts tested as above. The sterilized portion gave no ammonia, while the infected gave ammonia corresponding to .003 gm per litre, equalling approximately .005 gm urea per litre.

Experiment II. Sewage was boiled for fifteen minutes, and treated as above. The "free" flask gave some ammonia, but the infected flask gave about .0001 gm more, distilling from 10 c.c.

Lack of time prevented carrying these experiments any further, but they appeared to show the presence of the urea in the sewage at the points taken-viz. at E. Chester-park, [Ex. I] and at the pumping station (II), as was indicated by the "circumstantial evidence" stated, thus showing the decomposition to be of a gradual nature, and, judging from the change in composition referred to, requiring from twenty four to forty-eight hours for its completion.

Summary.

The following points have been considered in this thesis, and the results are as follows:

(1) Kjeldahl's process seemed to give good technical results for the "combined" and "total" ammonias. For water analysis, too little work was done to allow giving any opinion of its value.

(2.) Bungener and Fries's process failed to give good results on sewage, the results coming low.

(3) The presence of nitrogen as nitrates, nitrites, or both, was shown by the modification of the "galvanic" process, and the action of the galvanic current was shown to be applicable to the determination of such small amounts as exist in water and sewage.

4. The presence of urea in the sewage, was shown, and the qualatative test by means of the ferment applied.

As a result of the analysis made during the investigation, a table is given, which may give some idea of the composition of Boston Sewage. It includes free ammonia, total nitrogen, and in some cases nitrates or nitrites, total solids, urea, and albuminoid ammonia. Where no determinations were made a line is drawn.

Date.	Hour.	Where.	T.	C.	F	a	a:c	Slds.	Sp. Gr.
Mar. 16"	10 A.M.	E. Chester P'k.	7.93[1]	4.04[1]	3.85[1]	—	—	2.7	—
April 13"	10 "	" " "	7.48	4.60	2.88	1.53	1/3	—	—
" 14"	10 "	" " "			2.90	1.32	1/3	—	—
" 15"		" " "	7.9[2]	4.40	3.20	1.30	1/3	—	—
May 12"	7.30 P.M.	Pumping Station.	3.32	0.52	2.80	0.27	1/2	5.92	1.004
May 13"	8.20 A.M.	" "	4.20	1.02	3.18	0.44	5/12	5.48	1.0037
May 13"	7.20 P.M.	" "	7.00	3.58	3.42	1.15	1/3	2.54	1.0045
[4] May 14"	7.20 A.M.	" "	2.20	0.88	1.32	0.35	1/2	5.70	1.0040
May 15"[3]	1.00 A.M.	" "	5.62	4.33	1.29	0.87	1/5	4.82	1.0023
[5] May 15"[3]	8.15 A.M.	" "	7.30	5.71	1.59	0.25	1/23	4.16	1.0032
May 15"	4.00 P.M.	" "							

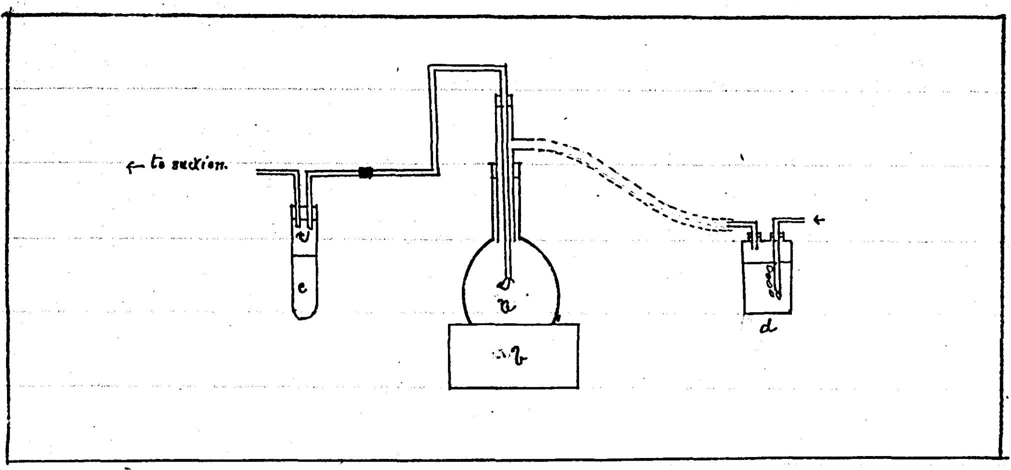
[1] Av. of eight determinations during a week.

[2] " " two "

[3] After a severe rain storm contained much dirt &c.

[4] Nitrates & nitrites 0.003 as NH_3 [5] Nitrites & nitrates 0.00008 as NH_3.

[Parts per 100.000.]



The arrangement used for evaporating, is as in the figure.

(a) is the small flask in which the residue is afterwards oxydized.

(b) is the water-bath.

(c) is a reciever, introduced as a means of testing the "distillate" for ammonia, to detect any loss. [Of many tests of the liquid in (c), none ever gave any traces of ammonia.]

(d) is a drying-bottle of sulphuric acid. The tube connecting c and the air pump was provided with a screw-clamp to hold the suction under control.

I would draw attention to the ratio of the "combined" and "albuminoid" ammonias, and the large change in this ratio when the sample contained storm-water.

The processes were conducted as usual, the free and albuminoid ammonias being determined from portions of 10 c.c. [except in the first four] and the total being determined in portions of 25 or 50 c.c. [The apparatus used for this is figured, and described.] Solids were determined by evaporation of portions of 50 c.c. Sp. Grav. was determined in portions of 100 c.c. In the last determinations assistance was rendered by the Sanitary Laboratory, as the analysis of two samples a day could not be done alone.

I regret, that, owing to lack of time, a longer series could not have been determined, and that a more complete table could not have been presented.

also, that the work on the urea and on the nitrates and nitrites could not have been made more complete. The figures given in the table, having been determined from small quantities of material, and the material being of such varying constitution, give, of course, only approximate values, which is all the table is intended to express. One other noticeable fact is the difference between the samples from E. Chester Park and from the pumping station, this rather contradicting the inference stated on page 3.

Respectfully submitted.
Charles Ricketson Allen.

May. 19th, 1885.

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