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STATE OF ILLINOIS
DEPARTMENT OF REGISTRATION AND EDUCATION

DIVISION OF THE
STATE WATER SURVEY
A. M. BUSWELL, *Chief*

BULLETIN NO. 29

STUDIES ON TWO-STAGE SLUDGE DIGESTION

1928–29

AUG. 26th 1818

[Printed by authority of the State of Illinois.]

URBANA, ILLINOIS

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W. D. HATFIELD, *Editor*

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ORGANIZATION

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STATE OF ILLINOIS

DEPARTMENT OF REGISTRATION AND EDUCATION
STATE WATER SURVEY DIVISION

Urbana, Illinois, May, 1930.

M. F. Walsh, Chairman, and Members of the Board of Natural Resources and Conservation Advisers:.

GENTLEMEN: Herewith is submitted a report of studies on two-stage sludge digestion carried out during the years of 1928-29. I recommend that they be published as Bulletin No. 29 of the Illinois State Water Survey Division.

We have been greatly aided in the preparation of this manuscript by Dr. W. D. Hatfield, Superintendent of the Sanitary District of Decatur, who has reviewed and edited these studies, and made several valuable suggestions. We wish to take this opportunity to express our appreciation of his assistance.

Respectfully submitted,
A. M. BUSWELL, *Chief.*

INTRODUCTION AND SUMMARY

By A. M. BUSWELL

In January, 1928, the State Water Survey started a series of experiments on separate sludge digestion to verify certain results which had been obtained in the laboratory. It has long been held that the digestion of sewage solids occurs in two stages, a so-called acid or foul stage, followed by the inoffensive stage. In well-operating tanks the two stages of digestion go on simultaneously and at such a rate that the alkalies produced in the second stage apparently neutralize the acids produced in the first*. In some cases, however, the acid stage predominates and it is difficult to obtain satisfactory digestion without special procedure. For regulating such conditions the addition of lime has long been employed and Imhoff early recommended the maintenance of a certain minimum amount of old sludge to keep the acid stage from predominating. In the laboratory experiments we have found that the acid stage of digestion resulted largely in the degradation of so-called grease, which includes animal and vegetable fats, and the soaps.

A study of the possible chemical reactions which must occur during the decomposition of fats and greases suggested that, if the acids and alkaline stages of digestion were separated, that is, carried on in different tanks, there was a probability of obtaining some interesting results. Whether these results would prove of practical value or not was not the question. We expected to find out something further about the chemistry of sludge digestion.

Another reason for carrying out a fairly large scale experiment on sludge digestion was to determine the degree of decomposition of the solids. So far as is known practically all of the data in the literature[4,14] are based on plant operation. In calculations from plant operating data the figure for the original amount of fresh sewage solids is usually obtained from suspended solids determination on the raw sewage. Wagenhals, Theriault and Hommon[50] have shown that this figure may easily be in error by 25 per cent. This experiment was set up in such a way that the fresh solids could be accurately measured and sampled before being transferred to the digestion tank. Arrangements

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* The present investigation indicates a second explanation of the absence of acidity in well-operating tanks; namely, that under favorable conditions the fatty acids are decomposed into methane and carbon dioxide as rapidly as they are formed.

were also made to permit accurate sampling of all solids removed from the tanks during the runs. Recently various investigators[51] have reported on laboratory experiments on sludge digestion in which accurate control was possible, but conditions of the experiments varied more or less widely from those in plant operation.

Information was also sought regarding the production and composition of gas from each stage, as was data on the digestion of fatty substances and cellulose, and the change in the relation of volatile matter to ash before and after digestion of the solids.

Description of Experimental Plant

The tanks and equipment used for the work are located in the Sewage Experiment Station of the State Water Survey and the Department of Civil Engineering of the University of Illinois. An external view of the plant is shown in Figure 1.

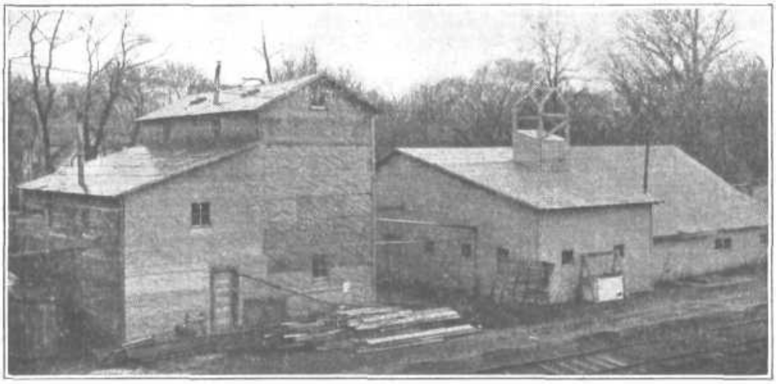

FIG. 1.—GENERAL VIEW OF EXPERIMENTAL SEWAGE TREATMENT PLANT.

The equipment actually used in this experiment had been designed for other purposes and was converted for the present use by appropriate reconstruction. The experimental equipment consisted of a Nidus sedimentation tank, which allowed for three hours detention, a primary and secondary digestion tank of 2,000 and 1,560 gallons capacity, respectively. Prom an estimated per capita load of 200, the primary and secondary digestion tanks had a per capita capacity of 1.3 and 1.0 cubic feet, respectively. General layout and details of the equipment are shown in Part I, Figures 4, 5, 6, and 9.

Scum Control

After about 5 or 6 weeks of operation a difficulty was encountered which has frequently been met with in connection with sludge digestion, namely, the accumulation of scum. The scum reached a thickness of 26 inches in about a month. It had a moisture content of 80 per cent and was so stiff that a shovel was forced into it with difficulty. The. liquor expressed from the scum had a pH of 5. The installation of a grid beneath the gas dome to hold the scum submerged as suggested by Imhoff[28] did not solve the difficulty. The grid soon became clogged and the escape of the gases was prevented.. Wooden arms turned by a crank from the outside were installed to break up the scum. When the arms were rotated slowly several times a day for 3 to 5 minutes at a time no improvement was observed. . When they were rotated rapidly they whipped up a froth that filled the whole collecting dome.

Besides mechanical means there are three obvious ways by which a gummy scum or colloidal gel of this sort can be softened and disintegrated. All three depend on decreasing the viscosity of the material.

1. It is known that many organic substances change their viscosity with change in pH, especially is this true of their water emulsions. The use of lime to aid sludge digestion has been known for many years and its beneficial action when observed is no doubt due in part to this effect.
2. Heating lowers the viscosity of gummy material such i as that under discussion.
3. Dilution of a colloid with the continuous phase will lower viscosity.

The use of lime was not suitable since it would change the chemical conditions of the experiment. Heat has various effects on the processes of digestion as well as on the viscosity of the medium. Its use was not attempted. Dilution was found effective but the volume of water required was too great to be practical. In many plants scum is reduced by hosing but in some cases the cost for water is an appreciable item.

Since the scum is practically a gel it seemed likely that the liquor from which it had separated might serve to dilute and soften it. A pump and pipe connections were installed (Figure 2) so that liquor from beneath the scum could be pumped up and allowed to flow onto the scum in a gentle stream-. One-inch pipe was used, the rate of pumping was 10 gallons per minute and the discharge pipe was placed two to three inches above the scum. The operation was carried out so as to avoid all violent jet or spray action since experience had shown that such action resulted in extensive foam formation. Ten days circulation

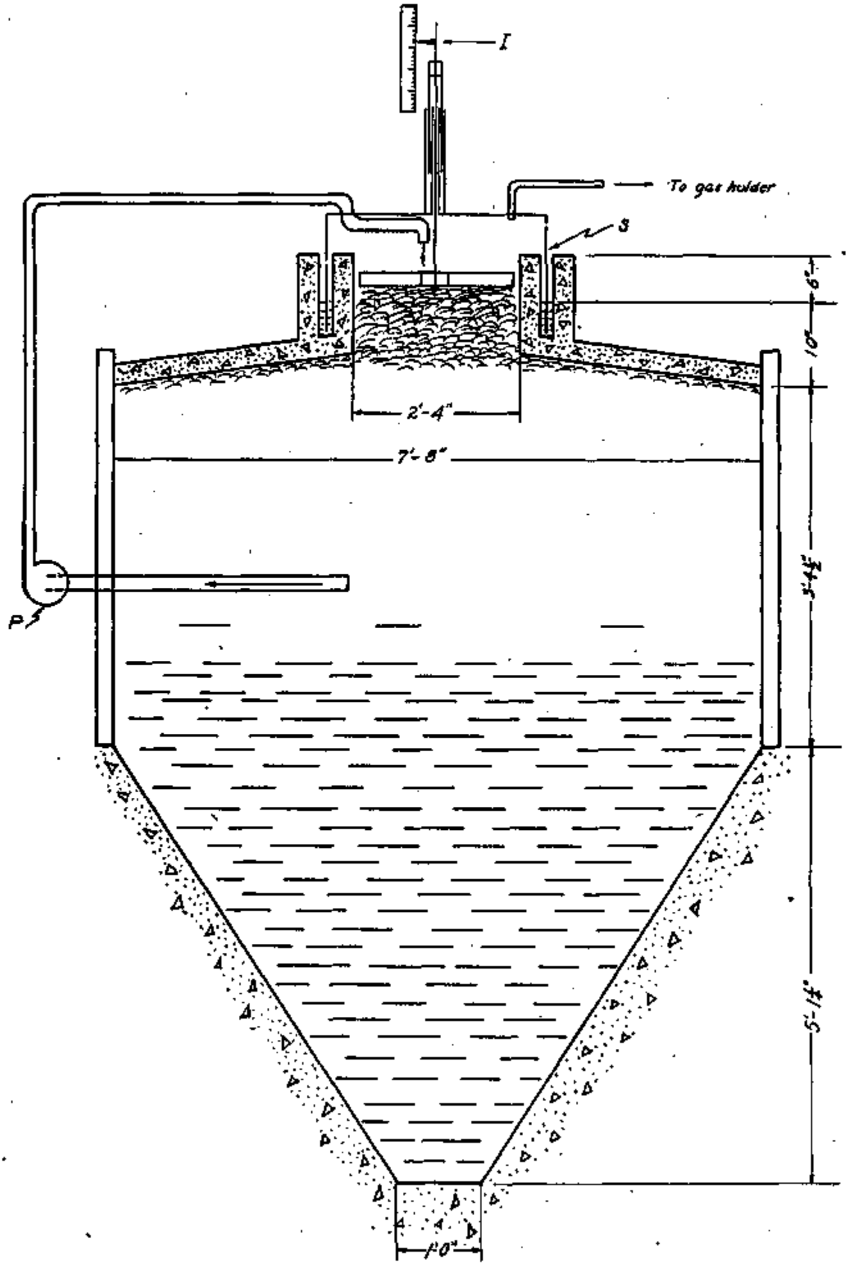

FIG. 2.—SKETCH OF CIRCULATOR FOR SLUDGE DIGESTION TANK.

under these conditions completely disintegrated a 26-inch layer of stiff scum and the gases evolved during digestion were allowed to escape smoothly into the gas collector. After the scum had once been softened and disintegrated it was found that circulation for from 5 to 10 minutes a day prevented any further scum formation.

The Foam Problem

It sometimes happens that the fermentation of sewage sludge results in the formation of froth or foam rather than scum. Foaming appears to be rather spasmodic and of variable intensity. When it occurs in tanks with restricted gas vent areas (e. g. Imhoff tanks, or tanks provided with gas collectors) it may completely upset the operation of the plant. Foam has been seen coming out of the vents of an Imhoff tank like the froth out of a bottle of warm soda pop. In tanks equipped with gas collectors the foam will sometimes fill the gas dome, clog the delivery pipe and force its way out through the water seal running "all over everything."

The factors which bring about this condition during sludge digestion do not appear to be the same in all cases; but the formation and stability of any foam depends upon the viscosity rather than the surface tension of the film. The rate or violence of foaming will depend on the rate of gas formation.

Since it is known that the liquid in the froth film in such cases is more concentrated than the liquor from which the foam is formed, it seemed possible that circulation of the liquor, as was done in scum control, would dilute and break the foam. As an experiment a vigorous foaming was produced in the tank under observation by raising the temperature to 37°C. thus greatly increasing the rate of gas production. After a few hours the foam broke the water seal *8* and flowed over the top of the tank. The circulating pump *P* was then started and within three minutes the foam level had subsided seven inches as shown by the indicator I. Thereafter it was. possible to control the foaming by starting the pump whenever the indicator showed that the level was rising. Three to five minutes circulation at a time was sufficient to break the foam, and routine circulation for 5 to 10 minutes per day was usually sufficient to prevent foam formation.

Dr. W. D. Hatfield reports a test on the control of foam by circulation in an Imhoff tank as follows; "A somewhat similar arrangement (to that here described) was installed in four of the eighteen gas collectors of a badly foaming Imhoff tank. During six weeks operation the foaming seemed to be so well controled that connections have been placed in all the 108 gas collectors of the six-tank plant. Circulation for scum and foam control is now in use on one tank (18 collectors) and permanent connections to all gas holders are contemplated."

Lime treatment is usually effective in controlling foaming but in aggravated cases the quantity required is high, amounting in one case to one hundred dollars worth per day. Circulation cost for the same plant is estimated at $5.00 per day.

We believe that the installation of some such circulating device would result in an appreciable economy in the cost of Imhoff tanks. An average of the area allowed for gas vents in six Imhoff tanks in Illinois was 29 per cent. We feel confident that it would be possible to reduce this figure to a nominal one or two per cent.

The cost of Imhoff tanks is in the neighborhood of five dollars per capita. Since in the average Imhoff tank half of the total volume is devoted to sludge digestion and storage, the cost of the portion for sedimentation may be placed at $2.50 per capita. The saving resulting from the increased capacity due to the practically complete elimination of gas vents has been estimated to be fifty cents per capita.

This method of scum control has the following features in its favor.

1. The power costs will be about one-half cent per million gallons per day treated in Imhoff tanks, if the cost of power is three cents per kilowatt hour and the liquor is circulated for ten minutes a day at a rate of 10 gallons per minute per gas collector, against a head of three inches plus friction in the pipe. The 10-minute period is probably the minimum for ordinary operation but may not be enough under aggravated conditions.
2. The installation cost should not be excessive since with proper connections one small motor and pump could be made to serve a large number of gas vents by pumping to one at a time.
3. The method is effective. It remedied the most aggravated case which has come under our observation.

Summary

Having solved these operating difficulties, we were next able to proceed with the main purpose of the investigation, namely, to study the chemical changes during two-stage sludge digestion. The data obtained during this first period was so interrupted by difficulties requiring plant shut-downs that they are considered valueless. The plant, as already described, was operated as a two-stage digestion unit at a temperature of 23°-24°C. from June 15, 1928, to January 18, 1929. On June 15, the primary digestion tank was filled with raw sewage and the first sludge added. Thereafter sludge was pumped from the sedimentation tanks into the primary tank three times a day, about 20 gallons at a time, except for three weeks in September when no sludge was added.

Six weeks elapsed before the secondary tank was put in operation. To start this secondary tank 520 gallons of digesting sludge were transferred from the primary tank to the bottom of the secondary tank, the remainder of the tank being filled with tap water. Sludge was next transferred three weeks later.

It was soon observed that relatively little gas was produced in the second digestion tank. Therefore, the frequency with which sludge was transferred from the first to the second tank was increased until transfers were made every 5 to 8 days, or about once a week as compared to longer periods between transfers at the beginning of the experiment. This schedule of frequent transfers was followed from about October 1 until the end of the experiment in January. The amount transferred averaged about 115 gallons at each transfer.

The summary of the chemical data are given in Table I. It will be noted that this table gives the results of digestion in terms of total overall digestion only. Analytical and sampling difficulties, which could not be detected until the end of the experiment, made it impossible to present the data in any other form. The figures for total- solids, "grease", cellulose, and gas produced are direct determinations. The cellulose refers to so-called alpha-cellulose (Schweitzer's reagent followed by acid precipitation), and does not include crude fiber. The figure for protein is obtained by multiplying the organic nitrogen (total nitrogen minus ammonia nitrogen) by the factor 6.25. The data for sulfate is calculated from figures obtained by Elder[11]. Since Elder's data were not collected for this particular investigation, the figures must be regarded as approximations. The figure for crude fiber digested is obtained by subtracting the amount of "grease", cellulose, and protein digested from the total weight of gas. We justify this calculation on the basis of bottle experiments which have shown a very close relation between organic matter digested and gas produced. In subsequent experiments we plan to make a direct determination of this factor.

TABLE I

EFFECT OF TWO-STAGE DIGESTION ON SEWAGE SOLIDS

Material	Added lbs.	Residue lbs.	Digested lbs.	Produced lbs.	Liquefied lbs.	Digestion per cent	Liquefaction per cent
Total Solids (1)	2,305.0	858.8	1,446.2			62.6	
Grease (2)	747.96	72.9	675.06			90.3	
Cellulose (3)	40.3	3.0	37.3			92.5	
Protein (4)	308.0	112.3	195.7			63.5	
Sulfate (SO_4) (5)	90.0	15.0	75.0			83.0	
Crude Fiber (6)			294.0				
Dehydration (7)			169.2				
Ammonia Comp.	24.5	130.9		106.4			
Settling Solids	2,250.0	567.6			1,682.4		74.8

Gas Produced (CH_4, CO_2, H_2)[8]

1st tank = 15,687 cubic feet = 1063.4 pounds
2nd tank = 2,036 cubic feet = 138.6 pounds
Total = 17,723 cubic feet = 1202.0 pounds

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[1] Includes ammonium compounds (acetate and carbonate). [2] Petroleum ether extract.
[3] Alpha Cellulose. [4] Protein = 6.25. + (Total Nit.-Amm. Nit.)
[5] Estimated from Data of A. L. Elder (11). [6] Crude fiber = Gas — (Grease + Cellulose + protein digested).
[7] Solids digested — (Gas + Sulfate) = Dehydration. [8] Includes dissolved and bicarbonate CO_2.

The difference between the total solids digested and the sum of the "grease", cellulose, protein, sulfate, and crude fiber digested has been attributed to dehydration or loss of hydrophylic properties. We have had several indications that this occurs. Experiments now in progress will furnish a check on this assumption.

The most interesting data in this table are those on gas production. These data were collected under favorable conditions and we feel satisfied that they are accurate to within one per cent. The biggest error involved is the loss of gas during sampling and sludge transfer operations. It will be observed that practically 90 per cent of the gas was produced in the first tank, although the sludge during the major part of the experiment remained in that tank for only seven or eight days. It is also interesting to note that 90 per cent of the "grease" is digested and that the weight of "grease" digested corresponded to more than 58 per cent of the gas. The gas produced amounts to .39 cubic feet per capita per day from both tanks, or .34

cubic feet per capita per day in the primary stage. The gas produced in both tanks is 12.3 cubic feet per pound of solids digested, not including the dissolved and bicarbonate CO_2, and corrected to a nitrogen free basis.

Though the major part of the digestion takes place in the primary stage, there is a further ripening in the secondary tank necessary to complete the biochemical reaction and produce an inoffensive and rapidly draining sludge.

An average of 150 analyses of the gas show it to consist of 64 per cent methane, 28 per cent carbon dioxide, 3.4 per cent hydrogen, and 4.3 per cent nitrogen, with a calculated heat value of 640 B. T. U. per cubic foot.

The data suggest a somewhat clearer picture of sludge digestion than we have had heretofore. It might be described as follows:

There is at first a relatively rapid fermentation which results in the decomposition of the simpler compounds and the production of a large quantity of gas, including most of the hydrogen sulfide. This fermentation is apparently complete in a very few days. This observation is in accord with that of Hatfield[17] and others, who have observed that 50 per cent of the gas is evolved in the first 24 hours. After this fermentation has reached completion, it is still necessary to allow the sludge to undergo some sort of a ripening process. The exact nature of this is not understood but the net result is that the sludge loses its water-binding properties and can then be drained on sand beds. Such a process could best be carried out in a separate sludge digestion plant composed of a relatively small primary tank designed to allow 6 or 8 days detention and equipped with the necessary circulating devices to prevent scum and foam formation, and then a secondary tank or even a lagoon* of sufficient size to allow for the necessary ripening of the sludge to a state where it will drain on sand beds (Figure 3). This would result in the following economies: (1) cost of cover would be reduced to approximately one-tenth; (2) since no scum is observed in the secondary stage of digestion, no special measures would have to be taken to prevent scum formation; (3) it is possible that only the primary tank would need to be heated, and (4) the secondary tank might be replaced by lagoons.

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* Suggested by Dr. W. D. Hatfield.

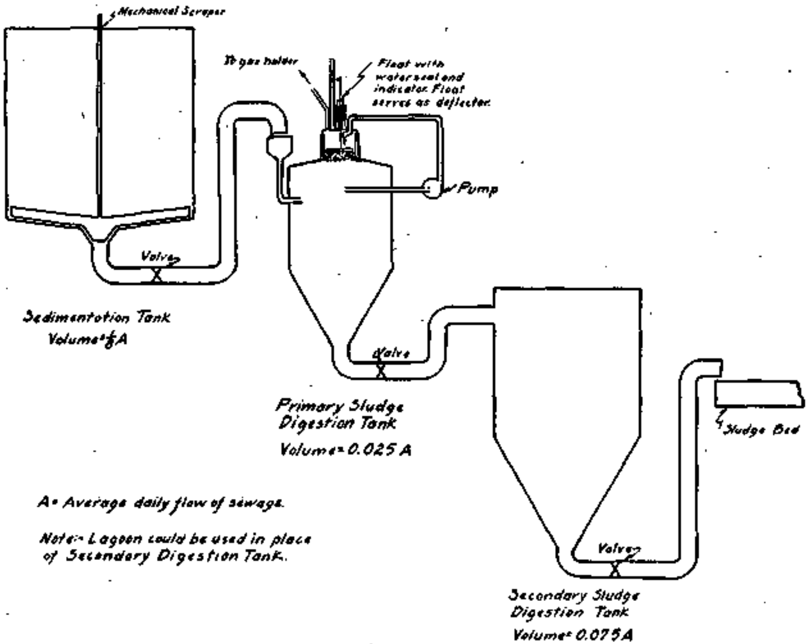


FIG. 3.—SCHEMATIC DIAGBASM OF TWO-STAGE SLUDGE DIGESTION SYSTEM.

PART I

PLANT DESIGN AND OPERATION

By A. M. BUSWELL, H. L. WHITE AND H. E. SOHLENZ

A general layout of the equipment used in the experimental plant is shown in Figure 4. The equipment used in this particular experiment is indicated by Roman numerals.

Design of Equipment

The' digestion tanks (Fig. 4, Nos. I & II) were of wood stave construction with hoppered concrete bottoms. The primary tank was 7 feet 7 inches in diameter and 9 feet 10 inches deep to the bottom of the cone. The secondary tank was 7 feet 6 inches in diameter and 5 feet deep. Each tank was provided with an overflow valve just above center for the removal of liquor when additions to the tank were made; opposite this was an inlet valve just below center for the additions of sludge. This inlet was horizontal to prevent any undue agitation of the tank contents when additions were being made. Discharge connections in the bottom of the tanks provided for the removal of accumulated sludge. Sludge was removed from the primary tank to the secondary tank by means of a pump. In the secondary tank, however, the sludge was removed either by gravity flow or by means of a pitcher pump with a movable suction, introduced into the sludge through the gas vent; removal of the gas collector being necessary for this latter operation. Details of the two tanks are shown in Figures 5 and 6. The capacity of the primary tank was determined to be 2,000 gallons, and the capacity of the secondary tank was determined to be 1,560 gallons, from the computed capacity curves (Figures 7 and 8). Both tanks were provided with a steam heating coil to maintain a constant temperature of about 25°C. and with pumps for circulating liquor from the center of the tank up on to any scum or foam which might collect on the surface.

Each tank was covered With a metal hood for collection of the gases formed. These gas collectors were connected to separate gas holders (Fig. 4, Nos. III and IV) so that the gas from each tank could be measured and analyzed. The gas was collected under a slight positive pressure and each time a holder had filled, temperature, pressure, and volume readings were taken. The design of the gas collectors is shown in Figures 5 and 6, and that of the gas holders in Figure 9.

Fresh sewage sludge for feeding the primary tank was obtained from a nidus sedimentation tank (Fig. 4, No. V) treating 20,000 gallons a day of Champaign-Urbana domestic sewage, equivalent to a contributing population of about 200 persons. This experimental nidus tank was being used for the experiments reported by Buswell and Pearson[6]. The sludge was a "by-product". One advantage in using this sludge was that it consisted of 85 per cent of the suspended solids in fresh sewage as compared to 70 per cent removed by an Imhoff tank[24]. This gave a greater amount of sludge to treat from the same volume of sewage though the composition of the sludge from the two sources was about the same.

Methods of Operation

Bate of Flow in Nidus Tank. Since the flow of the sewage carrying only domestic wastes varied from hour to hour, **and** since **the** concentration also varied over a wide range during the twenty-four hours,

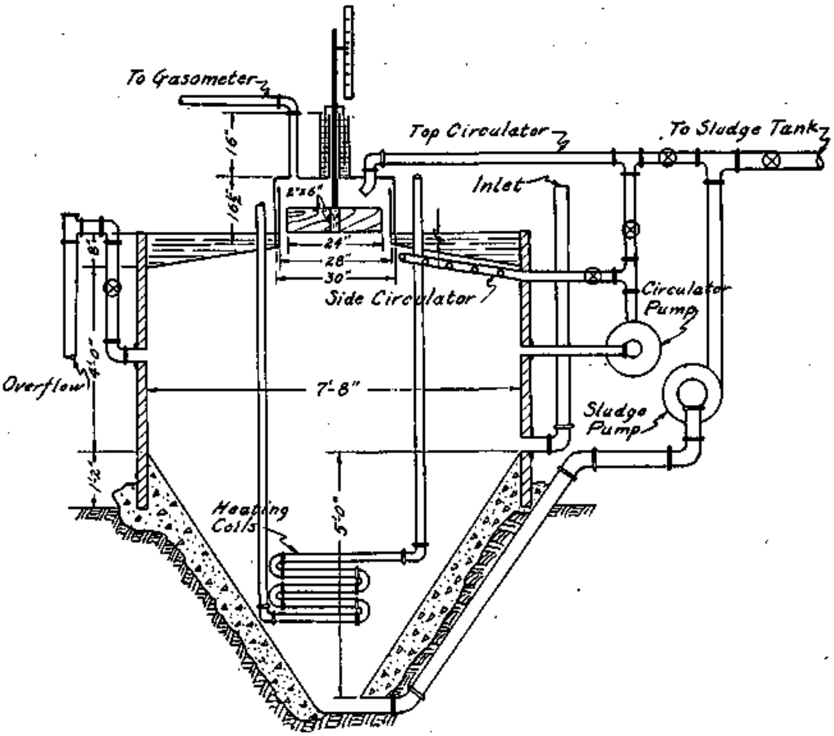


FIG. 5.—DETAIL OF PRIMARY DIGESTION TANK (TANK I).

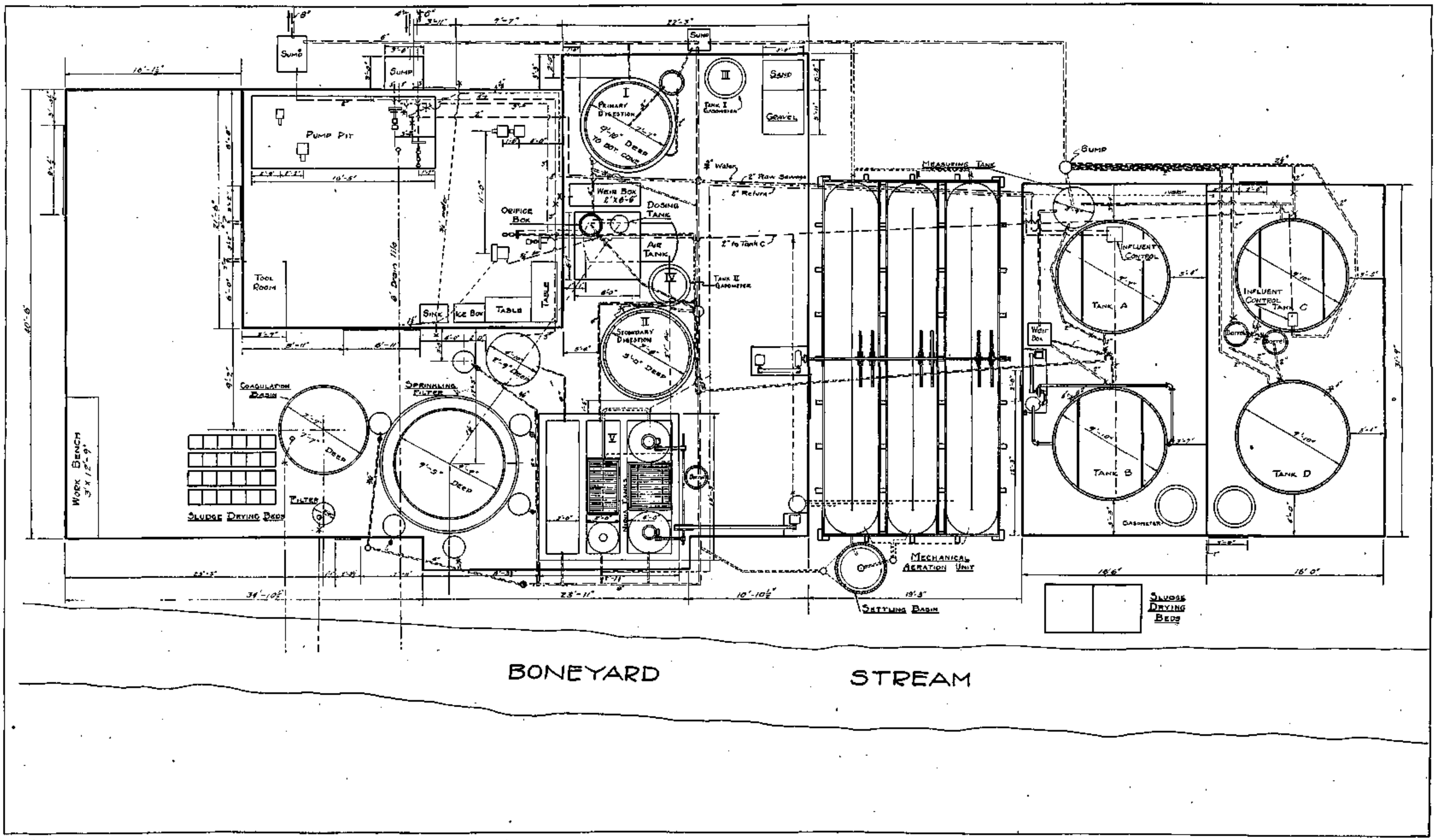


FIG. 4.—GENERAL LAYOUT OF SEWAGE EXPERIMENT STATION.

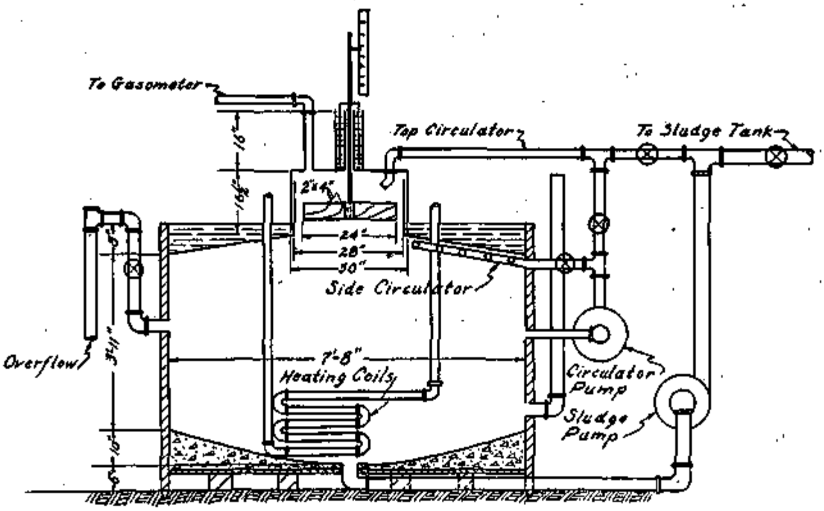


FIG. 6.—DETAIL OF SECONDARY DIGESTION TANK (TANK II).

it was apparent that a treatment device fed at a constant rate would give erroneous results. This was due to the fact that at a constant rate the device would treat less organic matter per gallon per day than was actually discharged into the sewer. The ideal method of meeting this difficulty would have been to feed the treatment device at a rate proportional to the flow in the sewer from which it was fed. Such a method would have required an automatic control which has several disadvantages when used with raw sewage. Hourly or semi-hourly control was out of the question because it required too much attention. The nidus tank was fed with sewage closely approximating a proportional relation to the volume and concentration of the wastes in the sewer, by adjusting the feed every eight hours. Figure 10, curve No. 1, shows the hourly variation of flow in the Champaign-Urbana sewer. Curve No. *2* shows the rate of feeding the Nidus tank for each of the eight-hour periods. These are similar to the curves shown in Water Survey Bulletin No. 25, Figure 12, page 56.

Operating Records. Hourly and daily records of all plant data and measurements were kept. Individual record sheets of each transfer of sludge were made at the time of the transfers. These records were used in tabulating and compiling the data and results of the experiment.

Sampling Schedule. A routine schedule for pumping sludge and sampling was carried out daily. This schedule was for the hours of 6 :00 a. m., 2:00 p. m., and 10:00 p. m. Since the study was experi-

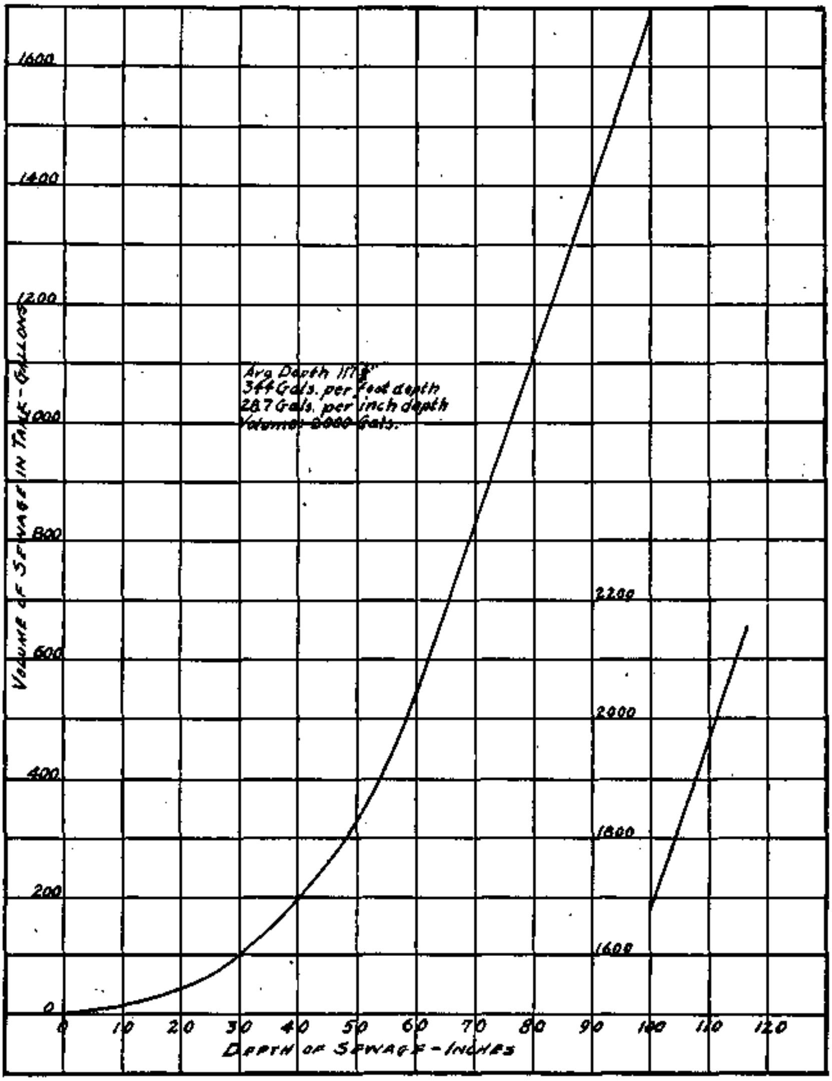


FIG. 7.—CAPACITY CURVE OF PRIMARY SLUDGE DIGESTION TANK.

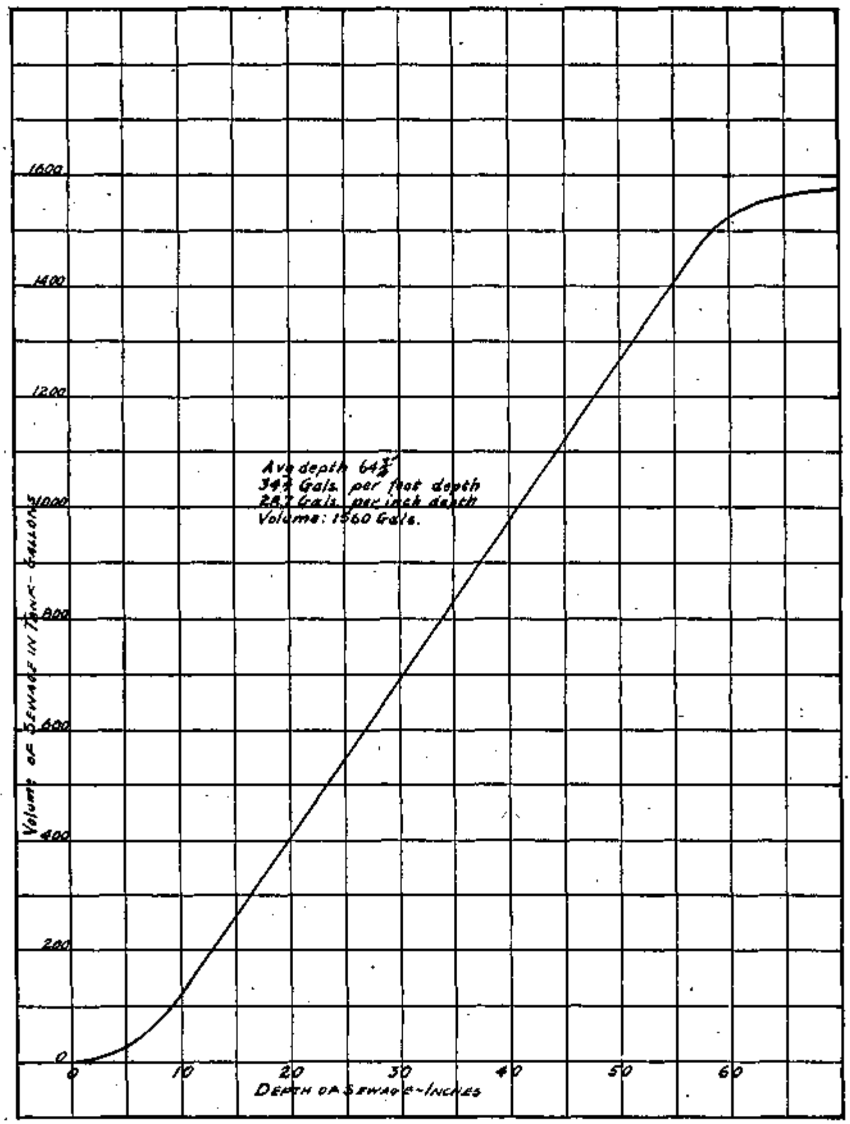


FIG. 8.—CAPACITY CURVE OF SECONDARY SLUDGE DIGESTION TANK.

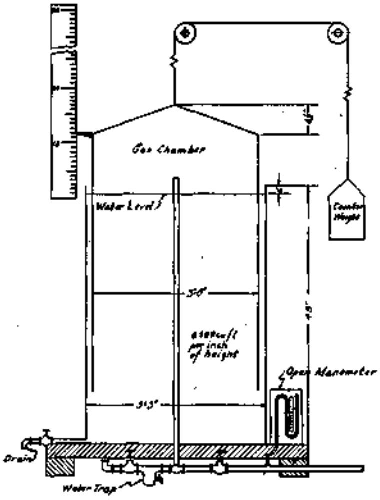


FIG. 9.—DETAIL OF GASOMETER.

mental in character a strict routine in regard to transferring of sludge from the first to the second tank was not adhered to, but changes were made to fit the apparent needs of the experiment. The data on transferring sludge are shown in Figure 20, Part II.

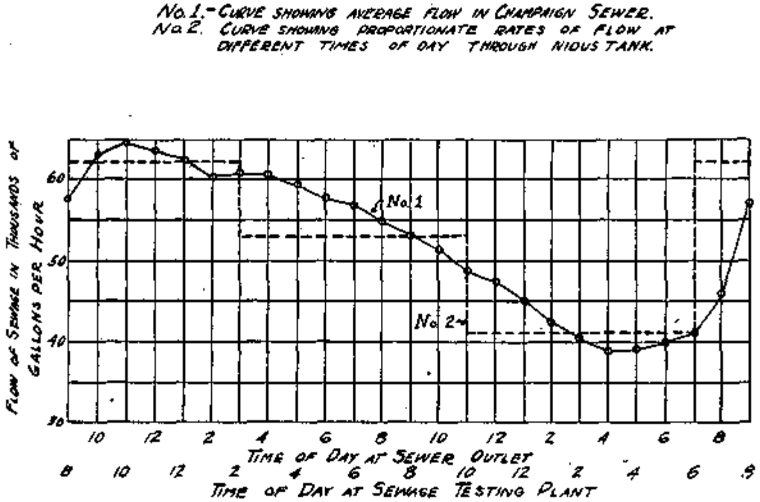


FIG. 10.—CURVES SHOWING HOURLY VARIATION OF FLOW OF SEWAGE. (DATA FROM ILL. STATE WATER SURVEY BULL. NO. 18.)

Pumping Sludge. Sludge from the three sedimentation sumps of the nidus tank was pumped according to schedule into a calibrated tank where its volume was measured. It was then sampled and allowed to flow into the primary digestion tank. Each of the sumps had a capacity of 6 to 7 gallons so that approximately 20 gallons of sludge were added to the primary tank every eight hours or about 60 gallons a day. However, during the first 5 weeks only two pumpings a day were made or about 40 gallons a day.

Overflow 'Measurement. When the sludge entered the primary tank the displaced liquor was measured, sampled, and discharged into the sewer. When circulation was not necessarily continuous, it was done at a time sufficiently in advance of the sampling schedule so that the overflow liquors would contain a minimum amount of suspended matter.

Temperature Regulation. At each sampling period the temperature of the tanks was recorded, and if it had fallen below the desired point, steam was passed into the coil until the temperature was raised the necessary amount. In extremely cold weather, since the buildings were not heated, it was necessary to run the steam almost continuously and keep closer check on the temperature. An average temperature of 24°C. was maintained during the entire period. The attempt was made to keep the temperature at 25°C. or slightly below this figure rather than above. This is near the optimum for sludge digestion according to the views of Sierp[47], Rudolfs[45], Baity[8], and Imhoff[27], though Hatfield[17] reports 32°-33°C. as the optimum.

Circulating Schedules. Circulation of the liquor in the primary tank was done as a routine twice daily for a period of from 10 to 15 minutes. Circulation of the secondary tank liquor was not routine but was done at frequent intervals during the experiment. Both tanks were circulated following each transfer of sludge.

Miscellaneous. Gas measurements as described were made by reading the calibrated gas holder. Pressure was determined from manometer reading plus barometric pressure. Amounts of scum or foam on top of the tanks was indicated by a float arrangement (Figures 5 and 6) which allowed the level of the scum or foam above the liquor level to be read in inches. Thickness of scum on the tank could be measured only when the gas collectors were removed. The device used (Figure 11) consisted of a metal plate (D) mounted on the end of a 3/8-inch pipe so that the plate could be moved from a vertical to a horizontal position (or vice versa) by means of the wire control (E) and was designed by Kelleher. The method is described on page 15 of Water Survey Bulletin No. 27 and is as follows:

"The plate (in vertical position) is forced down through the scum and then turned to the horizontal position. The plate is then raised until it comes in contact with the under surface of the scum, and the measuring rod is placed in a vertical position with one end in contact with the upper surface of the scum. A reading of the index (F) on the measuring rod (G) then gives directly the thickness of the scum. The measuring rod is graduated in feet and hundredths of a foot, reading downward from the top."

The volume of the sludge in the bottom of the tanks was determined by a hose attached to the suction of a pitcher pump. When sludge began to be discharged from the pump, the depth to which the suction hose had been lowered was measured. Prom the capacity curves the amount of sludge in the tank could be calculated.

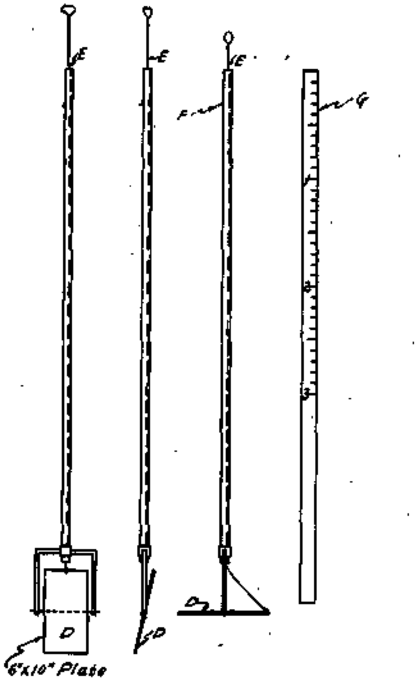


FIG. 11.—DEVICE FOR MEASURING THE THICKNESS OF SCUM (AFTER KELLEHER).

Methods of Transferring Sludge. After measuring the level of the sludge in the primary tank and calculating the sludge volume, this volume or an excess was pumped into the secondary tank, thus insuring practically complete removal of sludge from the first to the second tank. At the beginning of the experiment, the volume of sludge transferred was determined from the lowering of the liquor level in the primary tank. Later the practice was to feed sewage into the first tank as fast as sludge was removed, thus keeping the tank full of liquor to the exclusion of air. When this practice was followed the amount of sludge transferred was determined from the volume of liquor displaced from the secondary tank. Samples were taken of each transfer and of the displaced liquor from the second tank. These samples were analyzed separately and the data recorded in the proper place, as removals from the respective tanks. Analysis of the sewage used to fill up the primary tank was not made at each transfer, but an average analysis of raw sewage was used in computing the amount of solids, etc., added to the primary tank in this manner.

One of the difficulties encountered in sampling was that of obtaining a representative sample of the transferred sludge. The sludge was transferred through a closed system to prevent aeration and it was impossible to procure a sample of the transfer as a whole. Small samples were composited from the pump at intervals during the transfer. The error involved in this procedure could not be determined until after the experiment was completed, and though as stated in the introduction this error prevents the separation of the summary of the digestion data into two stages, it does not minimize the value of a discussion of transfers.

Description of each transfer was recorded. Four typical record sheets follow.

Record of Sludge Transfer No. 1

July 30,- 1928

Sludge and Scum Measurements.

The scum on the primary tank was 12 inches thick and was quite light. There were no large masses.

The sludge level was 58 inches below the top of the tank = 420 gallons of sludge.

Amount of Sludge Transferred.

Sludge was displaced from the primary tank until the liquor level was 18½ inches below the normal level = 517 gallons.

The transferred sludge reached a level in the secondary tank of 24 inches at the center of the cone of that tank = 520 gallons.

Record of Sludge Transfer No. 2

August 21, 1928

Sludge and Scum Measurements.

Primary tank:—There were 6 inches of light scum on this tank and no large masses.

The sludge level was 7 feet from the water level = 160 gallons.

Secondary tank:—There was no scum on this tank. The sludge level was 53 inches from the water level or 11¾ inches from the bottom = 175 gallons.

Amount of Sludge Transferred.

About 14 gallons of sludge were removed from the secondary tank to the drying bed, after which, sludge from the primary tank was pumped into the bottom of the secondary tank. The overflow from the second tank was 100 gallons indicating that 114 gallons of sludge were transferred from Tank I to Tank II.

The primary tank was drawn down about 3½ inches = 100 gallons.

Record of Sludge Transfer No. 9

November 16,1928

Sludge and Scum Measurements.

The hoods on the two tanks were not removed so that these measurements could not be made.

Amount of Sludge Transferred.

Sludge was pumped from Tank I to Tank II until a total of 110 gallons of liquor had been displaced from the latter to the measuring tank. A composite sample of the transferred sludge was taken. It consisted of three parts collected at the start of the pumping, at the time half of the sludge had been pumped and at the end of the pumping. A sample of the secondary tank overflow liquor was also taken.

Record of Sludge Transfer No. 19

January 18, 1929

Scum Measurements.

There were 5 inches of thick scum on the primary tank and ½ inch of very thin scum on the secondary tank.

Sludge Levels.

In the secondary tank the average sludge level as determined by three trials was 41½ inches from the water level, or 23½ inches from the bottom of the tank = 510 gallons. Adding 50 gallons removed previous to the measurement the total volume of sludge in the secondary tank at the end of the experiment was 560 gallons.

Sampling Procedure.

The liquor and sludge in the primary tank were circulated for about 4 hours and two samples of the stirred contents of the tank were taken, one at the pump and the other at the top of the tank where the circulating liquor was discharged.

Eight gallons of very thick sludge were removed from the bottom of Tank II by means of gravity flow. Forty-two more gallons were removed by a hand pump, mixed with the first eight gallons and placed on sludge drying beds. A sample of the 50 gallons was taken. The secondary tank was then allowed to settle for a sludge level measurement.

After this level was determined the 1510 gallons in the secondary tank were completely stirred up and a sample taken.

Samples were also taken of the scum on each tank.

Some idea of the routine of sludge transferring can be obtained from the above records taken at the beginning, during, and at the end of the experiment.

Scum collecting on the Nidus tank was skimmed off, saved until a considerable amount had been collected, weighed, sampled, and added to the primary tank.

PART II

SOLIDS BALANCE

By A. M. BUSWELL AND G. E. SYMONS

1. *Samples.* Samples of the sludge and overflow liquors were received at the laboratory each morning about 8:30 a. m. These samples had been collected the previous day according to the regular sampling schedule and had been kept in a refrigerator until the following morning. There were three samples each of the sludge, primary and secondary tank overflows. The sludge samples were collected in 250 cc. wide mouth, cork stoppered bottles and the overflow samples were in 1-liter small mouth, cork stoppered bottles.

Each sample was labeled with a tag bearing a key number indicating its source (4= sludge, 5= primary overflow, 6= secondary overflow, etc.) and a sample number indicating the position of the sample in plant records. These tags also contained the hour and the date of sampling. On the reverse side of the tag was the number of gallons of material that the sample represented. In the case of the overflow liquors the back of the tag also contained the temperature of the overflow liquor at the sampling time.

The three samples from each source were composited in proportion to the number of gallons that each represented, into one sample. This was then called the sample for the day and the analyses were made on it. Of this daily composite sample of the sludge, and the two overflows, respectively, an amount proportional to the total gallons for that day was added to a larger composite bottle and acidified with H_2SO_4 (1 cc./100). This composite was for the weekly or monthly determination of total nitrogen. A similar composite for volatile acids was made basic with 2 ml. of 33 per cent NaOH/100 ml. These latter composites were analyzed weekly, during the early part of the experiment and monthly during the latter part. Weekly or monthly composites of the dried solids (to be used for grease, cellulose, volatile matter, and ash determinations) were made by evaporating volumes of the daily composite samples proportional to the number of gallons of sludge pumped or liquor overflowing and adding these together.

For example, if 63 gallons of sludge were pumped during the day, then 63 cc. of the daily sludge composite sample would be evaporated, dried, and the residue added to a tightly stoppered composite bottle. If the liquor overflow were 60 gallons, then 300 cc. of the daily composite sample would be evaporated, dried, and the residue added to the proper composite bottle. This larger volume was necessary in order to obtain an amount of residue which would be sufficient for the samples necessary in the determination of grease, cellulose, volatile matter, and ash.

Samples of sludge in the two tanks were taken several times during the experiment, for the purpose of determining the drainability of the sludge. These were obtained by using a pitcher pump, or from samples of transferred sludge.

2. *Analytical Determinations.* Most of the determinations on the sludge, overflow liquors, etc. were made in accordance with Standard Methods for the Examination of Water and Sewage[49], but since some slight variations were introduced, a discussion of each determination performed follows.

(a) **Total Solids.** Total solids were determined on the overflow liquors by evaporating a 100 ml. sample (measured in a volumetric flask) on a steam bath, drying the residue for one hour at 103°C. and weighing and reporting as parts per million. On samples of sludge (both fresh and digested) the solids were determined by weighing out a wet sample, evaporating, drying, weighing and reporting as per cent solids.

Soon it was observed that the alkalinity of the secondary overflow samples was higher than the total solids as determined above. This fact showed that there is volatile material lost in drying. Since the sludge enters the tank with the positive and negative ions in equilibrium the only change in dissolved material is in ammonia, volatile acids and bicarbonate ion. Ammonium in combination with these negative radicals is volatile under the conditions of the above determination. This fact was taken into consideration in the calculations of the data for the summaries and balances. How this is done is discussed under "calculations". The experimental data tables and curves show, however, the data as actually determined.

(b) **Alkalinity.** Alkalinity to methyl orange was determined on a 10 ml. portion of a settled sample according to Standard Methods[49] and reported as parts per million of $CaCO_3$.

(c) **Ammonia Nitrogen.** Ammonia nitrogen was determined by adding 25 ml. of the sample to 225 ml. of ammonia free water containing 5 ml. of sodium carbonate solution (1 g./100 ml.) and distilling 200 ml. into N/20 H_2S0_4. Back titration was done with N/20 NaOH using sodium alizarine sulfonate as the indicator. Ammonia nitrogen was reported in parts per million. Another indicator used in the absence of the above was a mixture of 1 part methylene blue (.4 g./lOO ml.) plus 3 parts methyl red (2 gms./1OO ml. 70 per cent alcohol) which has a color change of lavender to green at a pH about 4. to 5.

(d) **Total Nitrogen.** A 25 ml. sample was digested with 25 ml. of concentrated H_2S0_4, 3 gms. of Na_2S0_4, and 1 ml. of $CuS0_4$ solution (1 gm./100 ml.), for a period of 2 hours after it had been decolorized. This was then neutralized with 33 per cent NaOH solution and distilled as above. Determinations were made on composite samples and reported as parts per million.

(e) pH. Using indicators made in accordance with the directions of Clark[8], the pH was determined on a 5 ml. portion of a settled sample by comparison with a color chart (after Clark) published by Williams and Wilkins.

(f) **Volatile Acids.** Volatile acids were determined only on basic composite samples. Two hundred ml. of sample were measured into a distilling flask; 5 ml. of concentrated H_2SO_4 added and 150 ml. distilled into a receiver. The distillate was titrated with N/10 NaOH and results calculated as acetic acid and reported in parts per million.

(g) **Settling Solids.** This determination as distinguished from the Imhoff Cone determination for settleable solids determines the weight of the solids that will settle under quiescent conditions in two hours. The determination as devised in this laboratory, is described and discussed in detail in Part V, page 86.

(h) **Volatile Matter and Ash.** Samples were ashed in a muffle at 800-900°C. for one hour. Owing to difficulties with sampling, the muffle used, and the determination itself, these data were not sufficiently accurate for use in the summary and balance sheets.

(i) **Mineral Oil.** Mineral oil was determined on the final contents of each tank by steam distilling a 200 ml. sample; extracting the distillate with ether, evaporating the ether at 25°C, drying and weighing the oil.

(j) Sludge Drainability. The drainability of the various samples of sludge was determined by pouring 1 liter of sludge on ¾ inch of wet sand in a 28-mesh sieve. The amount remaining in per cent of the total drained was plotted against time intervals. The curve of per cent remaining of total liquor drained, against time, together with the per cent solids before and after draining, gave a good idea of how well the sludge was digested. The weight of the sludge cake was also determined, which with the amount of liquor drained gave a check on the amount of evaporation. The apparatus used is shown in Figure 12. All drainability tests were made by Mr. H. E. Schlenz.

(3) *Experimental Data.* Typical daily data sheets for the records of the analysis of fresh sludge, primary and secondary tank overflow liquors, and transferred sludge are shown in Figures 13, 14, 15, and 16. Analysis of secondary tank overflow liquor at times of transfers were recorded similar to the daily analyses.

From the daily data sheets the average daily analyses of the sludge and overflow liquors for the different compositing periods were determined. These averages were used in calculating the composition of the material added and removed during the compositing periods. (See Part II, division 4, for calculation sheets.)

Analyses of scum from the tanks, and skimmings from the nidus tank were made and recorded similar to analyses of sludge except that not all determinations were made on these samples.

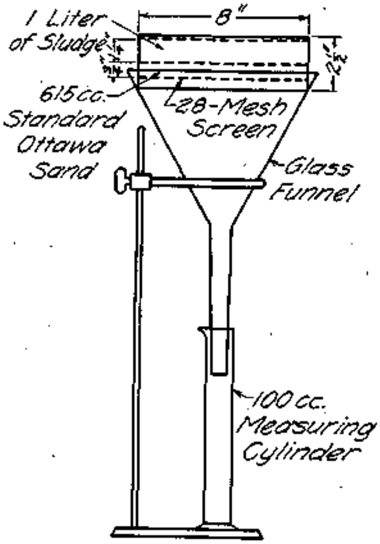
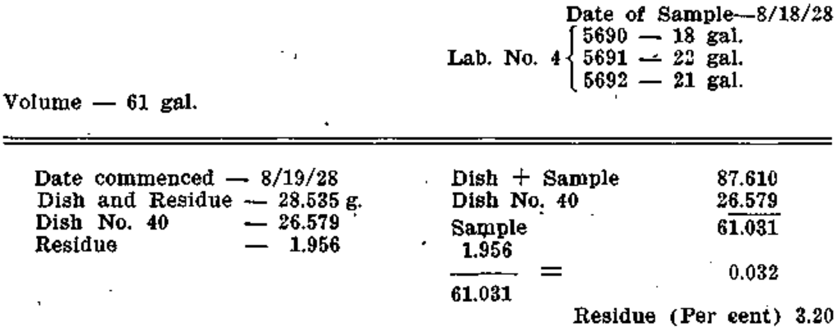


FIG. 12.—APPARATUS USED IN SLUDGE DRAINABILITY TEST.

NIDUS TANK SLUDGE DATA



Date of Sample—8/18/28

Lab. No. 4
5690 — 18 gal.
5691 — 22 gal.
5692 — 21 gal.

Volume — 61 gal.

Date commenced — 8/19/28
Dish and Residue — 28.535 g.
Dish No. 40 — 26.579
Residue — 1.956

Dish + Sample 87.610
Dish No. 40 26.579
Sample 61.031

$$\frac{1.956}{61.031} = 0.032$$

Residue (Per cent) 3.20

FIG. 13.—DAILY DATA SHEET—NIDUS TANK SLUDGE.

PRIMARY TANK OVERFLOW DATA

Date of Sample—8/18/28

$$\text{Lab. No. 5}\begin{cases}\text{5696 — 16 gal.}\\ \text{5697 — 15 gal.}\\ \text{5698 — 10 gal.}\end{cases}$$

Temp.—23.5°—23.5°—21°C.
Volume—41 gallons.

Date commenced — 8/19/28
Dish and Residue — 28.5621 g.
Dish No. 35 — 28.3813
Residue — 0.1808 Residue ppm. — 1808
Sample cc. — 10 $\frac{N}{50}$ H_2SO_4 cc. — 15.1 Alk. MO. ppm. — 1510

Titration	cc. NaOH =	H_2SO_4 cc.	
Sample cc.	1.00	7.00	
25	0.00	1.00	
	1.00	6.00	6 × .753 × 40 = Ammonia N ppm. — 181

pH — 7.1

Settling Solids

Vol. of liquor evaporated = 50 cc.
Dish + Residue — 35.930 g.
Dish No. 30 — 35.598

Residue — 0.332

$$\left.\begin{array}{l}\text{Dissolved +}\\ \text{non-settling solids +}\\ \text{settling solids in 50 cc.}\end{array}\right\}\ 0.090$$

Settling solids in 950 cc. — 0.242

$$\text{Settling Solids} = \frac{.242 \times 1000}{.95} = 255 \text{ ppm.}$$

FIG. 14.—DAILY DATA SHEET—PRIMARY TANK OVERFLOW LIQUOR.

SECONDARY TANK OVERFLOW DATA

Date of Sample—8/18/28

Lab. No. 6 $\left\{\begin{array}{l}\text{5702 — 16 gal.}\\ \text{5703 — 15 gal.}\\ \text{5704 — 10 gal.}\end{array}\right.$

Temp.—23.5°—23.5°—22°C.
Volume—41 gallons.

Data commenced — 8/19/28
Dish and Residue — 35.1607 g.
Dish No. 25 — 35.0074
Residue — 0.1533 Residue ppm. 1533

Sample cc. — 10 $\frac{N}{50}$ H_2SO_4 cc. — 14.8 Alk. M. O. ppm. 1480

Titration — cc. NaOH = cc. H_2SO_4

Sample cc.	0.75	6.00
25	0.00	0.75
	0.75	5.25

5.25 × .753 × 40 = Ammonia N. ppm. 158

pH — 7.2
Volume of Liquor Evaporated = 47 cc.
Dish + Residue — 23.611 g.
Dish No. 4 — 23.421

Residue 0.190
Dissolved + non-settling solids + settling solids in 47 cc. — .047 × .1533 } = 0.072

Settling solids in 953 cc. = 0.118

$$\text{Settling solids} = \frac{0.118 \times 1000}{.953} = 124 \text{ ppm.}$$

FIG. 15.—DAILY DATA SHEET—SECONDARY TANK OVERFLOW LIQUOR.

SLUDGE TRANSFER—NO. 2

Date of Sample—8/21/28
Lab. No. . . . .

Volume—114 gallons.

Date commenced	— 8/21/28		Dish + Sample	— 86.281 g.
Dish and Residue	— 29.901 g.		Dish No. 1	26.681
Dish No. 1	— 26.681			
			Sample	59.600
Residue	— 3.220	$\frac{3.220}{59.600} = 0.054$	Residue (%)	= 5.40

Sample cc. — 10 $\frac{N}{50}$ H_2SO_4 cc. — 18.0 Alk. M. O. ppm. — 1800

Titration —	cc. NaOH =	cc. H_2SO_4	
Sample cc.	3.6	10.0	
25	0.0	3.6	
	3.6	6.4	6.4 × .753 × 40 = Ammonia N. ppm. — 193
20	2.0	50.0	
	0.0	2.0	
	2.0	48.0	48.0 × .753 × 50 = Total N. ppm. — 1810

pH — 7.0

Settling Solids (Calc.)

Residue	5.40%
Average non-settling solids in several random samples	0.20
Settling Solids	5.20

Volatile Acids (as acetic)

Titration —	N/10 NaOH
200 cc.	4.30
Sample	0.00
	4.30

4.30 × 6 × 5 = 129 ppm.

FIG. 16.—DATA SHEET—TRANSFERRED SLUDGE.

The daily data obtained are not listed, but Figures 17, 18, and 19 show the average daily analyses of the sludge, primary and secondary overflow liquors, respectively, for the compositing periods. Also, the pounds per day of solids entering and leaving the system are shown. The most significant facts obtained from these curves are (1) the *practically constant* solids and total nitrogen in the sludge, and (2) the *upward trend* of the solids, ammonia, total nitrogen, alkalinity, and ammonium compounds in the overflow liquors. They also show the constancy of the pH of the overflow liquors.

In Tables II, III, and IV are shown data on the average daily analyses of the sludge and overflow liquors, for monthly periods instead of compositing periods (weekly and monthly). The tables present the analyses of material handled more clearly than the curves in Figures 17, 18, and 19, but do not show so clearly the trend of the analysis.

In Table V are shown the analyses of the various transfers of sludge from the first tank. The per cent solids as has been explained is not sufficiently accurate for use in the summaries, but the table as a whole gives valuable data on the composition of sludge after having passed through the first digestion stage. Figure 20 shows the volume of sludge transferred plotted against the time (in days) of the experiment.

TABLE II
AVERAGE DAILY ANALYSIS ON FRESH SLUDGE
(Monthly Periods)

Date	Volume gallons per day	Per cent solids	pH
6/16/28 7/30/28	38.0	3.58	6.5
7/31/28 8/31/28	55.8	2.42	6.5
9/21/28 10/21/28	59.0	2.98	6.4
10/22/28 11/15/28	55.0	2.96	6.3
11/16/28 12/15/28	57.6	2.78	6.4
12/16/28 1/11/29	48.5	2.33	6.5
Average 6/16/28 1/11/29	50.0	2.89	6.44

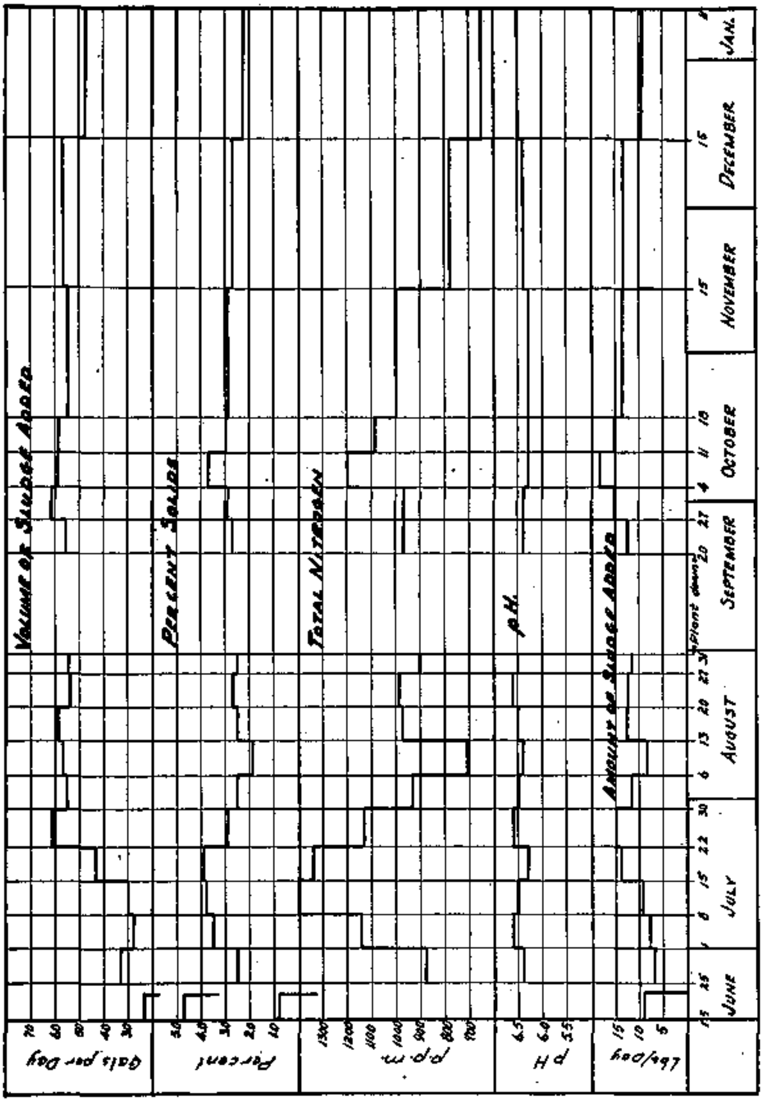


FIG. 17.—AVERAGE DAILY ANALYSIS OF FRESH SLUDGE (COMPOSITE PERIODS).

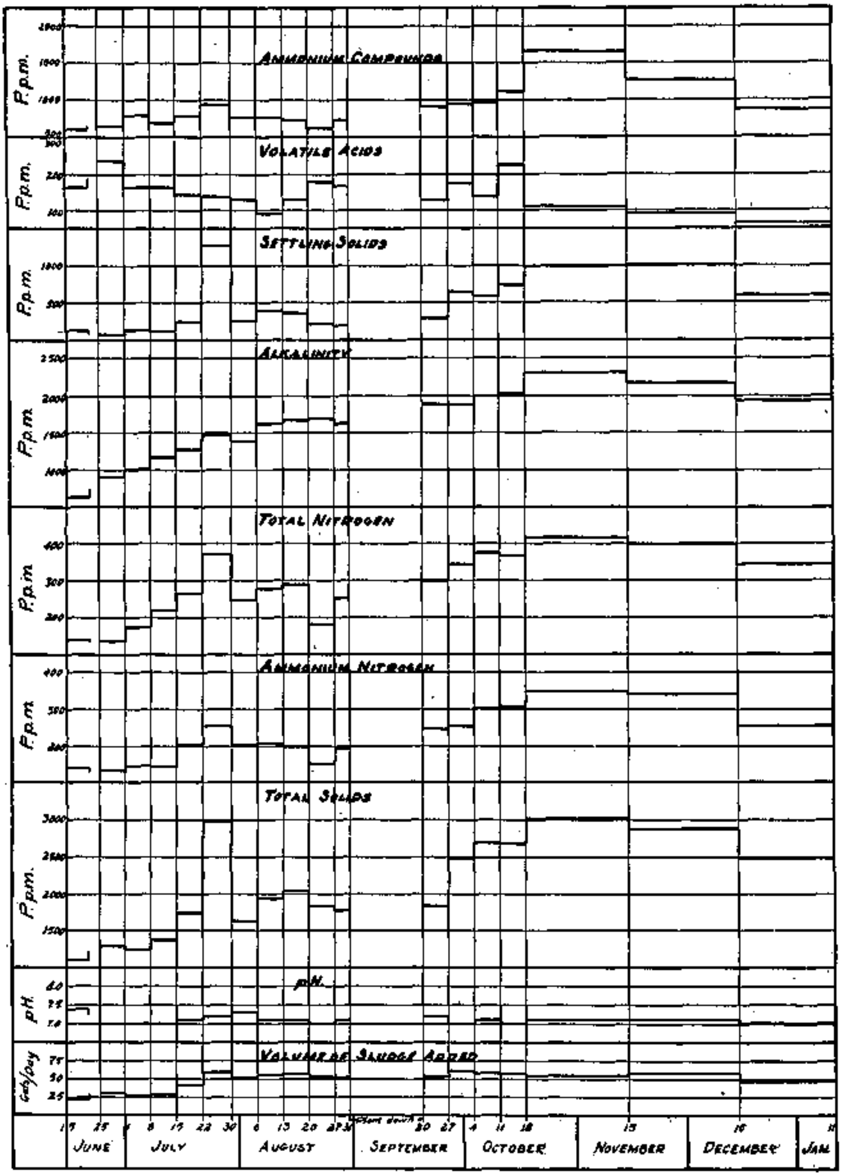


FIG. 18.—AVERAGE DAILY ANALYSIS OF PRIMARY TANK OVERFLOW LIQUOR (COMPOSITE PERIODS).

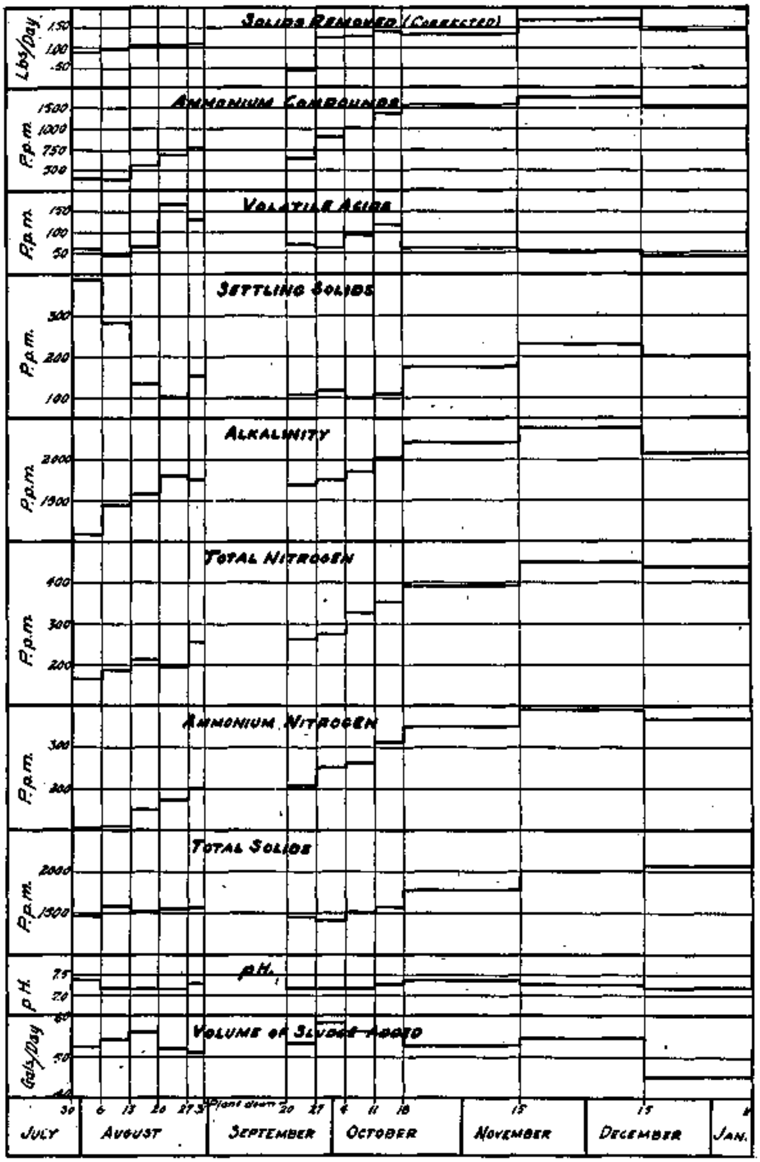


FIG. 19.—AVERAGE DAILY ANALYSIS OF SECONDARY TANK OVERFLOW LIQUOR (COMPOSITE PERIODS).

TABLE III

AVERAGE DAILY ANALYSIS OF PRIMARY TANK OVERFLOW

(Monthly Periods)

Date	Volume gallons per day	Temperature Deg. C.	Total solids p.p.m.	Settling solids p.p.m.	Alkalinity p.p.m.	Ammonia nitrogen p.p.m.	pH
6/16/28 7/30/28	37.0	23	1,633	373	1,108	170	7.1
7/31/28 8/20/28	54.3	23	1,882	338	1,573	203	7.1
8/21/28 8/31/28	54.3	23	1,808	208	1,681	177	7.0
9/21/28 10/21/28	57.5	24	2,434	579	1,974	251	7.1
10/22/28 11/15/28	53.6	24	3,005	994	2,311	349	7.1
11/16/28 12/15/28	56.1	25	2,861	1,010	2,164	340	7.1
12/16/28 1/11/29	47.3	26	2,492	600	1,938	252	7.0
Average 6/16/28 1/11/29	48.7	23.9	2,300	605	1,775	250	7.08

TABLE IV

AVERAGE DAILY ANALYSIS OF SECONDARY TANK OVERFLOW

(Monthly Periods)

Date	Volume gallons per day	Temperature Deg. C.	Total solids p.p.m.	Settling solids p.p.m.	Alkalinity p.p.m.	Ammonia nitrogen p.p.m.	pH
7/31/28 8/20/28	36.0	23	1,521	276	1,378	123	7.2
8/21/28 8/31/28	53.0	23	1,563	117	1,794	188	7.2
9/21/28 10/21/28	56.1	23	1,550	111	1,856	269	7.3
10/22/28 11/15/28	52.3	24	1,799	176	2,205	352	7.4
11/16/28 12/15/28	57.6	24	2,007	230	2,390	391	7.3
12/16/28 1/11/29	46.0	24	2,088	200	2,347	369	7.2
Average 7/31/28 1/11/29	47.5	23.5	1,760	185	1,979	282	7.28

TABLE V
ANALYSES OF TRANSFERRED SLUDGE

Date	Volume gallons	Per cent solids	Alka-linity p.p.m.	Ammonia nitrogen p.p.m.	Total nitrogen p.p.m.	pH	Volatile acids p.p.m.
7/31/28	520	3.39	1,400	226	1,242	7.0	138
8/21/28	114	5.40	1,800	193	1,810	7.0	129
8/30/28	61	3.02	1,680	196	1,340	7.0	169
9/21/28	175	9.78	2,020	271	3,580	7.2	169
10/9/28	152	3.95	2,000	295	1,650	7.1	138
10/15/28	47	6.58	2,040	306	1,875	7.0	225
11/5/28 (1) 11/8/28	178	7.43	2,440	472	2,690	7.2	120
11/16/28 (1) 11/21/28 11/26/28 12/1/28 12/7/28 12/13/28	651	4.22	2,145	408	1,780	7.1	328
12/19/28 (1) 12/27/28 1/4/29 1/12/29	504	4.71	1,935	312	1,720	7.0	257
Average (2)	134	5.06	2,030	340	1,850	7.08	232

[1] Analysis available only on composites of the several samples after 10/15/28.

[2] Average time between transfers, 10 days; total time of experiment, 218 days; 32 days without additions = 186 days of addition of sludge.

Transfers = 18. $\frac{186}{18} = 10$ days.

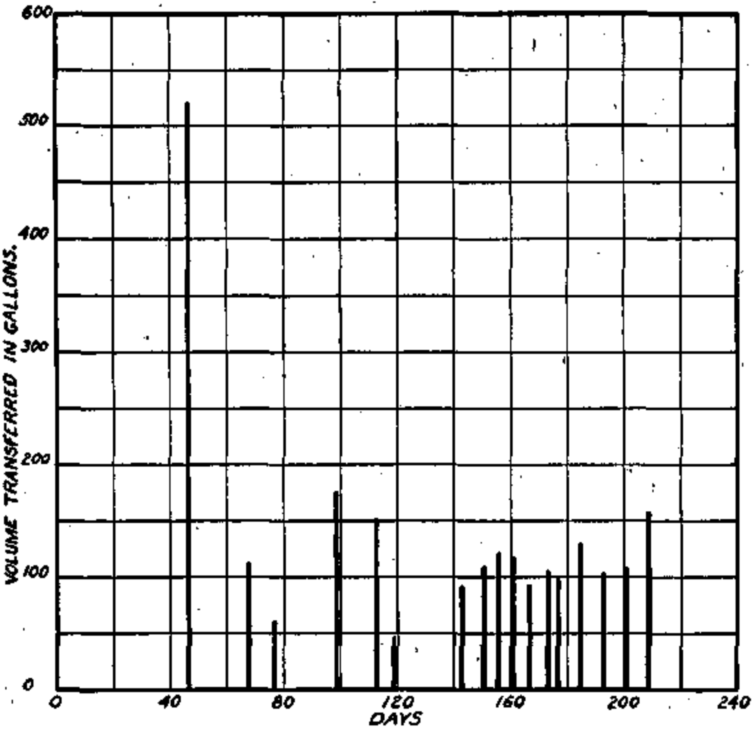


FIG. 20.—VOLUME OF SLUDGE TRANSFERRED PLOTTED AGAINST DAYS OF EXPERIMENT.

The analyses made of the secondary tank overflow liquor at the time of sludge transfers are shown in Table VI. An inspection of this table shows that the secondary overflow liquor at the time of transfers had practically the same analysis as the average analysis of the overflow liquor during the same periods except that it carried more settling solids (cf. Table IV). This is due not so much to agitation caused by transfer, but to circulation.

The data in Table VII show the analyses of several samples of sludge from the secondary stage of digestion. These data when compared with the data in Table V show the difference between sludge from the two stages.

In Table VIII are shown the analyses of the skimmings from the nidus settling tank that were added to the digestion system at three different times during the experiment. This material runs high in solids, grease and volatile matter.

When the two' digestion tanks were opened samples were taken of the scum collected on the surface. Analyses of these scums are shown in Table IX.

Analysis of the final contents of each tank showed that there was .04 per cent by weight (wet) of mineral oil in the primary tank contents and .073 per cent in the secondary tank.

Experimental data obtained on sludge drainability were recorded as in the following example. The data on all drainabilities are plotted in Figures 21 to 23 inclusive, and are discussed under "summary."

SLUDGE DRAINABILITY DETERMINATION

Sample from—Primary Digestion Tank Color—Black
Date —1/12/29 Odor—Strong, tarry

Time	Time hours	ml.	Remaining per cent of total drained	Remarks.
				1 liter sample
2:50 p. m.	0.0	0	100.0	
2:51 p. m.	0.016	5	99.1	Per cent moisture.
2:52 p. m.	0.032	8	98.6	Before draining — 95.25.
2:53 p. m.	0.05	18	96.8	After draining — 80.55.
2:54 p. m.	0.067	26	95.5	
2:55 p. m.	0.083	38	93.5	Wt. of screen + sludge cake — 1702 gms.
2:56 p. m.	0.1	49	91.6	Wt. of screen — 1362 gms.
2:58 p. m.	0.13	68	88.4	
3:00 p. m.	0.16	83	85.7	Wt. of sludge cake 342 gms.
3:05 p. m.	0.25	114	80.5	
3:13 p. m.	0.38	150	74.3	584 gms. liquor.
3:20 p. m.	0.5	178	69.4	342 gms. sludge cake.
3:38 p. m.	0.8	233	60.1	
3:55 p. m.	1.08	272	53.3	926 gms.
4:22 p. m.	1.53	322	44.8	1020 gms.= Weight of 1 liter of sludge.
4:52 p. m.	2.03	365	37.4	926 gms. = Weight of liquor + sludge cake.
5:25 p. m.	2.58	406	30.4	
6:15 p. m.	3.41	457	21.7	94 gms. of liquor evaporated.
7:20 p. m.	4.5	500	14.9	
10:30 a. m.	16.66	584	0.0	

TABLE VI

ANALYSES OF SECONDARY TANK OVERFLOW WHEN TRANSFERS WERE MADE

Date	Volume gallons	Total solids p.p.m.	Alkalinity p.p.m.	Ammonia nitrogen p.p.m.	Total nitrogen p.p.m.	Settling solids p.p.m.	Volatile acids p.p.m.	pH
8/21/28	100	1,575	1,700	136	248	120	87	7.2
8/30/28	61	1,715	1,760	191	234	89	166	7.2
9/21/28	137	1,935	1,780	225	271	700	70	7.2
10/ 9/28	152	1,950	1,860	275	347	64	93	7.3
10/15/28	45	2,080	2,000	331	370	125	63	7.3
10/ 5/28 10/ 8/28 11/16/28	174	1,785	2,230	343	407	330	60	7.4
11/21/28 11/26/28 12/ 1/28 12/ 7/28	651	2,421	2,420	410	436	661	78	7.4
12/13/28 12/19/28 12/27/28 1/ 4/29 1/12/29	504	3,104	2,340	382	484	1,300	44	7.25
Average.........	107	2,338	2,202	342	390	644	73	7.3

TABLE VII.

ANALYSES OF SLUDGE FROM SECONDARY TANK

Date	Volume gallons	Per cent total solids	Alkalinity p.p.m.	Ammonia nitrogen p.p.m.	Total nitrogen p.p.m.	Volatile acids p.p.m.	pH
8/21/28	14	.685 (3)	1,700	136	452	87	7.1
8/30/28	160 (1)	9.85	1,760	191	3,130	130	7.1
9/21/28	38	8.40	1,780	225	2,590	70	7.2
1/18/29	50	10.12	2,400	574	2,595	256	7.4
Average	....	9.45	1,910	281	2,180	136	7.2
Tank contents 1/18/29	1,510 (2)	2.73	2,400	448	1,045	56	7.0

[1] This sludge was not removed from the tank.

[2] Sludge volume was about 500 gallons and contained about 9.5% solids, but the figures in Table VII represent the analysis of the mixed sludge and tank liquor (1510 gals.).

[3] Not included in avg.

TABLE VIII

ANALYSES OF SKIMMINGS FROM NIDUS TANK

Date	Weight pounds	Per cent solids	Total nitrogen p.p.m.	Volatile matter per cent	Ash per cent	Grease per cent
9/26/28	40	12.09	1,900	81.4	18.6	33.8
11/5/28	25	12.15	1,859	86.1	13.9	47.6
12/27/28	55	17.28	2,295	89.7	10.3	72.3
Average	40	13.60	1,689	85.7	14.3	51.6

TABLE IX

ANALYSES OF SCUM FROM DIGESTION TANKS

Date	Thickness inches	Total solids per cent	Total nitrogen per cent	Volatile matter per cent	Ash per cent
Primary tank					
9/21/28	1	15.00	.5625	62.1	37.9
12/27/28	12½	17.6	....	75.6	24.4
1/18/29	5	28.65	....	79.0	21.0
Secondary tank					
10/24/28	2	3.45	....	78.8	21.2
1/18/29	1	21.19	....	64.9	35.1

4. *Calculations.* For the calculation of the data to the form in which the results are presented, (all results expressed in pounds) the factor 8.34 was used as the weight of a gallon of liquid. Specific gravity was not taken into account since it is so near 1.00 on the liquors and no definite information was available on the specific gravity of the sludge. At most it would probably not make a difference of more than 1 to *2* per cent, which is well within the limit of sampling error. Calculations from the original experimental data were made up in the following manner.

Nidus Tank Sludge Composite

11/16/28 } incl.
12/15/28 }

Gals. 1730
Solids 2.78% = 401 pounds (uncorr.)
Total nitrogen 784 p.p.m. = 11.3 pounds
pH 6.0

Primary Digestion Tank Overflow

11/16/28
12/15/28 } incl.

Gals. 1686
Solids .286% = 40.3 pounds (uncorr.)
Ammonia nitrogen 340 p.p.m. = 24.3 equivalents
Total nitrogen 398 p.p.m. = 5.59 pounds
Protein 6.25 × (398–340) = 362 p.p.m. = 5.09 pounds
Settling solids 1010 p.p.m. = 14.2 pounds
Volatile acids 93 p.p.m. = 1.55 equivalents

1.55 equiv. $NH_4C_2H_3O_2$ = 119 p.p.m.
22.75 equiv. $(NH_4)_2CO_3$ = 1140 p.p.m.

Ammonium Compounds = 1259 p.p.m. = 17.69 pounds

Total Solids (uncorr.) 40.3 pounds
Ammonium Compounds 17.69 pounds

Total Solids (Corr.) 57.99 pounds

Secondary Digestion Tank Overflow

11/16/28
12/15/28 } incl.

Gals. 1645
Solids 2002 p.p.m. = 27.53 pounds (uncorr.)
Ammonia nitrogen 391 p.p.m. = 28 equivalents
Total nitrogen 453 p.p.m. = 6.23 pounds
Settling solids 230 p.p.m. = 3.16 pounds
Volatile acids 53 p.p.m. = .88 equivalents
Protein = 6.25 × (453–391) = 388 p.p.m. = 5.32 pounds

.88 equiv. $NH_4C_2H_3O_2$ = 68 p.p.m.
27.12 equiv. $(NH_4)_2CO_3$ = 1304 p.p.m.

Ammonium Compounds = 1372 p.p.m. = 18.86 pounds

Total Solids (uncorr.) 27.55 pounds
Ammonium Compounds. 18.86 pounds

Total Solids (corr.) 46.41 pounds

Similarly calculations on transferred sludge and sludge drawn were made, though the former has not been used, as has been stated. From the calculation sheets were obtained the data for the summaries (Tables X, XI, XII).

Data for the final summaries on grease and cellulose were calculated on the total solids uncorrected. Dissolved and bicarbonate CO_2 were calculated from the pH and alkalinity, and were added to the gas volume. These data and calculations are all discussed in Part III.

(a) **Total Solids.** Before discussing the correction of total solids it is necessary to discuss the determinations of the volatile constituents. According to the findings of Neave and Buswell[37] the ammonia nitrogen as determined by distillation is high in some cases by an appreciable amount. This, however, is offset by the fact that the volatile acid determined as acetic by the method used determines only 58 per cent of the acid[10]. In the present experiment, the volatile acid was not calculated to 100 per cent. This would not make a considerable error in the calculation of ammonium compounds since they are based on the amount of ammonia nitrogen (which is high). The result would be to increase the equivalents of ammonium acetate and lower the equivalents of ammonium carbonate. This amounts to about 6 per cent. Since the ammonium nitrogen is at least 6 per cent high, then the total correction as made for volatile ammonium acetate and carbonate is nearly correct by weight.

The predominant volatile acid in digestion tank liquors is generally considered to be acetic. This is probably not true of fresh sludge, though we have ventured to make the correction on the fresh sludge on the basis of the average of several random determinations (routine data were not available). This amounts, however, to about 1 per cent of the total solids and is probably low, since we do not have any definite knowledge of the volatile compounds in sludge.

For making up the hypothetical combinations for the volatile ammonium salts, the ammonia is shown to exist, first, as the acetate, and then, as the carbonate. The former is volatile at the temperature of drying (103°C.) and the latter decomposes at the temperature of evaporation, namely, 80°-85°C.

In further proof of the above corrections a sample of overflow liquor was titrated to neutrality with standard sulfuric acid, evaporated, dried, and the residue weighed. This residue when corrected for ammonium acetate (which is lost under these conditions) differed only by about 2 per, cent from what the neutralized residue was calculated to be from the hypothetical combinations. Further, the validity of the assumption was proved by analysis of the residue. The following equation expresses the relation.

$$[\text{Tot. Solids (Det.)}] - [(Na^+ + Ca^{++} + Mg^{++} + Cl^- + SO_4^{--}) + \text{Protein}] + (NH_4)_2CO_3 = \text{Alkalinity.}$$

This relation proved to be true within less than five-tenths of one per cent. Based on these facts the correction of total solids was made according to the formula:

$$\text{Tot. Solids (corr.)} = \text{Tot. Solids (det.)} + NH_4C_2H_3O_2 + (NH_4)_2CO_3.$$

Though this correction is probably from 5 per cent to 10 per cent low, it is a step in the right direction, especially on digestion tank *liquors* where the total solids are not high and the error due to these volatile compounds is as great as 50 to 60 per cent. On digested sludges containing about 4 per cent solids the error is not so great. It amounts to only about 2 to 3 per cent. Had not these volatile compounds (130.9 pounds in the undigested material) been included in calculating the results, the error in the amount of material remaining undigested during the experiment (858 pounds) would have been 15.2 per cent. An error of 5 to 10 per cent in the correction for volatile material would therefore not amount to more than from 0.7 to 1.5 per cent of the total solids undigested.

Heukelekian[21] reports a possible loss of 25 per cent on digesting material containing 4 per cent solids. His figures are based on the loss of total carbon and total nitrogen, assuming that the carbon is 30 per cent of the material with which it is associated when lost. It may be that some of the loss he reports is the dissolved, free, and bicarbonate CO_2. In the experiment reported here the CO_2 from these two sources is calculated and added to the gas. Were this not done and the loss of CO_2 (free and bicarbonate) considered as a loss of total solids, the error in the amount of undigested material would amount to approximately 32 per cent. This figure is slightly higher than that reported by Heukelekian, but it must be remembered that, if the undigested material (858 pounds) were evenly distributed throughout the liquor passing out of the system and left at end of experiment, the average solids would only be .7 per cent, which is much less than the material containing 4 per cent solids with which he worked. Though, as he points out, other volatile substances (H_2S, amines, etc.) might be lost on drying, these substances are probably negligible, as compared to the loss of ammonium salts.

A comparison of his 25 per cent loss with the 15 per cent possible error noted in this experiment must not be taken as conflicting data since the results were not arrived at in the same manner or on samples of the same percentage of dry digested solids. The important fact shown by both experiments is that the total solids determination cannot safely be used in analyzing digesting solids and liquor *unless* some account is taken of the volatile compounds present in the original sample and lost on evaporation and drying.

(b) **Total Nitrogen.** Total nitrogen as determined by the Kjeldahl method determines ammonia nitrogen, most of the protein nitrogen, urea, and a portion of the cyclic compounds but not nitrite and nitrate nitrogen. Amounts of nitrogen from these latter two sources are, however, negligible in sludge and digestion tank liquors. According to unpublished data of C. S. Boruff of this laboratory, the usual method of digesting the sample for 30 minutes after it has become clear does not determine all of the nitrogen. Based cm the salicylic acid determination of total nitrogen, his data show that at least two hours digestion after the sample has become clear is necessary to even closely approximate a complete determination of the nitrogen.

(c) **Protein Nitrogen.** Based on the figures given in various texts on Physiological Chemistry[18,33] that the average nitrogen content in proteins is 16 per cent, the protein nitrogen was determined by the following equation.

6.25 X (Tot. Nit. — Ammonia Nit.) = Protein.

(d) **Settling Solids.** Since the amount of settling solids in the sample, determined as described in Part. V contain an amount of dissolved and non-settling solids in the 50 ml. portion used for evaporation, it is necessary to correct the results for this.

This correction necessitates the use of the residue on evaporation determination which is also made on a 100 cc. portion of the original sample as stated above.

In order to find the amount of true settling solids in the original sample, a correction equation is used. This equation is derived in Part V, page 87. On the data from this determination is based the amount of liquefaction during the process of digestion. The determination is, however, difficultly applicable to fresh sludge and digested sludge, or on samples where the solids run as high as 1 per cent. The amount of settling solids in the fresh and digested sludge were calculated by subtracting the average non-settling solids (determined on several random samples) from the total solids and using this figure in the calculations.

(e) **Sulfate.** No arrangements were made during the experiment to determine the amount of sulfate present in the samples. From the balances which showed an apparent ash digestion it appears that the determination of sulfate is important. From data obtained by A. L. Elder[11] the amounts of sulfate entering the system and accounted for as leaving or left at the end of the experiment were calculated. From these calculated data were obtained the figure for sulfate digestion.

(f) **Dehydration.** Since the sum of the known material digesting was more than the gas produced, it appeared that dehydration had taken place. The amount of this dehydration was calculated by the following equation.

Dehydration = Total solids digested. — [gas + sulfate digested].

The sulfate digested is added to the amount of gas because the gas does not include the products of sulfate digestion. The above equation then is valid, assuming the following relation, to be true.

Gas = [Protein + Grease + Cellulose + Crude fiber] digested.

(g) **Drainabilities.** The determination as designed does not give any one figure which can be compared with others. However, if the original and final moisture content is noted and the per cent remaining of the total liquor drained is plotted against time of draining a good picture of the condition of the sludge may be obtained.

(h) **Overflow Liquor Volume.** One of the difficulties encountered in the experiment was the leaking of the tanks near the top, between the staves. This appeared to be worse after the period of shutdown, during which the level of the tanks dropped several inches and allowed the staves to shrink. By carrying the liquor level lower than had been the custom, the trouble was partly eliminated. In view of the fact that the overflow measured less than the amount of sludge entering the tanks,. though there was known to be a loss, the arbitrary figure of 2.5 per cent was set as the shrinkage in each tank. This figure approximated conditions as they existed during the first few weeks of the experiment, and all overflow volumes were calculated on this basis from the volume of the sludge added. Eecent measurements indicate that the apparent shrinkage may be less than this. Most of this is probably due to the seepage through the staves. The error involved, however, is small, because the amount of solids carried in the tanks liquors is small compared to the total solids treated.

(5) *Summary.*

(a) **Drainability.** The curves in Figures 21 to 23 inclusive present the information obtained on sludge drainability. In Figure 21 the curve for secondary sludge was obtained on a sample of sludge that had passed through the primary stage in a previous experiment and had been in the secondary stage for 6 weeks. The sludge is not properly a part of this experiment but may be treated so in view of the fact that it had undergone the same course of digestion to which sludge in this experiment was subjected.

In Figure 22 is shown the drainability curve on primary sludge that had been digesting 9 days and secondary sludge that had been digesting 11 weeks. The data on the moisture after draining was not obtainable but the curves show the better draining properties of a secondary sludge even with a lower moisture content.

On August 30, 1928, the sludge in the primary tank was all transferred to the secondary tank, leaving only the scum. During the next three weeks no additions were made to the primary tank and it remained quiescent. At the end of this period it was found that the scum had settled to the bottom and appeared as sludge. This sludge (settled scum) was transferred to the secondary tank and a drainability was run on a sample of the same. Figure 23 shows this drainability curve in comparison with secondary tank sludge of the same date. This latter

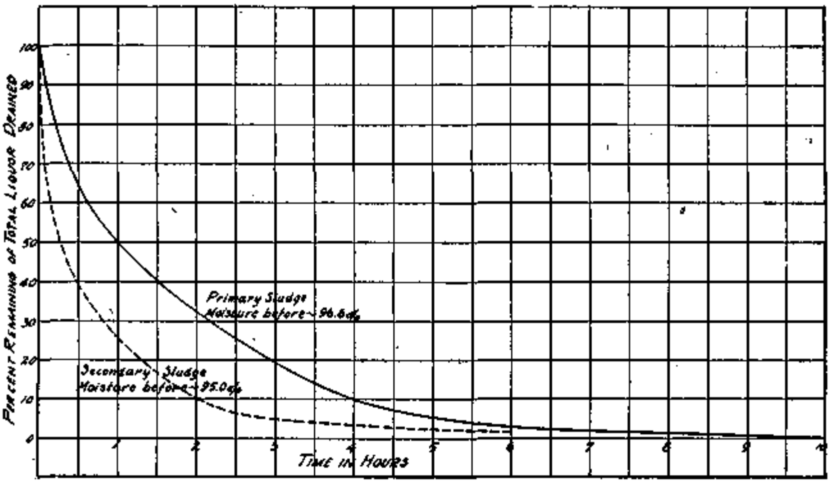


FIG. 21.—SLUDGE DRAINABILITY CURVES—7/30/28.

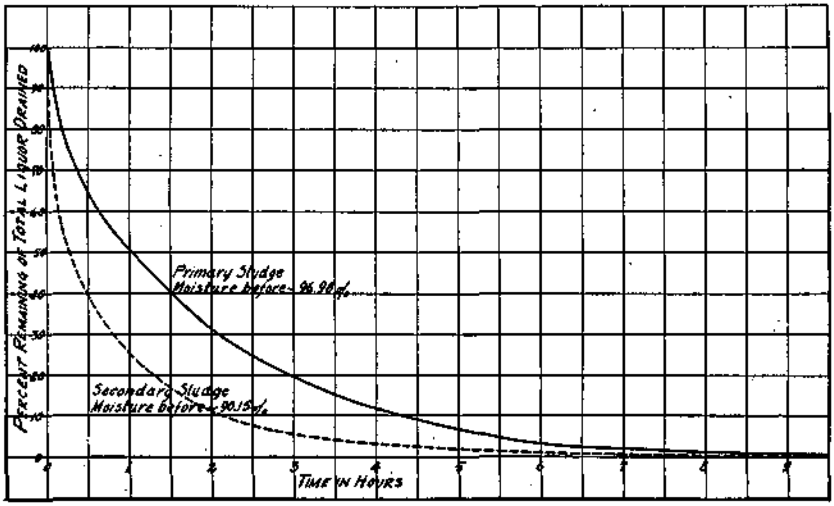


FIG. 22.—SLUDGE DRAINABILITY CURVES—8/30/28.

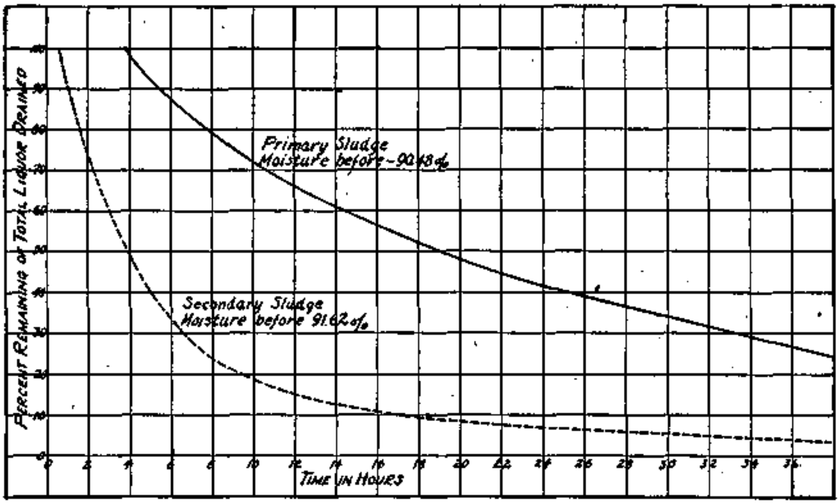


FIG. 23.—SLUDGE DRAINABILITY CURVES—9/21/28.

sludge was then 14 weeks old. From these curves it is apparent that the type of sludge plays an important role in the drainability. The test, as described, no doubt needs some refinement and standardization, for there were some determinations made which were not in accord with those shown nor with observations on sludge draining beds.

(b) **Digestion.** The data (in pounds of material) were transferred from the calculation sheets to the summary sheets shown in Tables X, XI, and XII, which show the additions and removals from the system. These tables are self-explanatory. From the summations of the different items in these tables, the balance sheets which are the final results of the experiment were made up. On these results are based the conclusions derived from the experiment.

Under the conditions of this experiment a pH of 7.0-7.3 was maintained without adjustment, and an increasing alkalinity and ammonia content was apparent in the liquors from tank I. The average per cent solids in the material added was 2.89 per cent, and the pH of the fresh sludge 6.4. The solids in the primary sludge transferred was 5.06 per cent and the pH 7.1. The secondary tank sludge averaged 9.45 per cent solids and had a pH of 7.2. The average time between transfers for the whole period was 10 days, though during the latter 3 months of the experiment the period was nearer 5-7 days.

Scum was usually less than 6 inches thick, containing 15-20 per cent solids which were about 75 per cent volatile matter. Physically it appeared to contain a large proportion of sticks, matches, hair, leaves, heavy papers, and particles of rags.

TABLE X

ADDITIONS TO PRIMARY TANK[1]

Date	Character	Volume gallons	Dry solids	Ammonium compounds	Solids including ammonium compounds	Settling solids	Total nitrogen	Protein
6/15/28	Sewage	2,000[2]	14.6	--------	--------	--------	.20	------
6/15/28- 6/21/28	Sludge	164	64.8	--------	--------	--------	2.02	------
6/22/28- 6/24/28	No additions	--------	--------	--------	--------	--------	--------	------
6/25/28- 7/ 1/28	Sludge	230	49.3	--------	--------	--------	1.71	------
7/ 2/28- 7/ 8/28	Sludge	196	57.1	--------	--------	--------	1.86	------
7/ 9/28- 7/15/28	Sludge	208	65.8	--------	--------	--------	2.41	------
7/16/28- 7/22/28	Sludge	302	97.6	--------	--------	--------	3.38	------
7/23/28- 7/30/28	Sludge	493	120.0	--------	--------	--------	4.63	------
7/31/28- 8/ 6/28	Sludge	389	81.6	--------	--------	--------	3.14	------
8/ 7/28- 8/13/28	Sludge	402	61.3	--------	--------	--------	2.38	------
8/14/28- 8/20/28	Sludge	415	88.4	--------	--------	--------	3.38	------
8/21/28- 8/27/28	Sludge	384	89.3	--------	--------	--------	3.18	------
8/28/28- 8/31/28	Sludge	218	46.1	--------	--------	--------	1.65	------
9/ 1/28- 9/20/28	No additions	--------	--------	--------	--------	--------	--------	------
9/21/28- 9/27/28	Sludge	392	86.0	--------	--------	--------	3.18	------
9/28/28-10/ 4/28	Sludge	434	107.2	--------	--------	--------	3.53	------
9/29/28	40 lbs. nidus tank skimmings	--------	4.8	--------	--------	--------	0.08	------
10/ 5/28-10/11/28	Sludge	417	114.9	--------	--------	--------	4.18	------
10/12/28-10/18/28	Sludge	415	107.6	--------	--------	--------	3.76	------
10/19/28-11/15/28	Sludge	1,392	342.8	--------	--------	--------	11.70	------
11/ 5/28	25 lbs. nidus tank skimmings	--------	3.0	--------	--------	--------	.04	------
11/16/28-12/15/28	Sludge	1,730	401.0	--------	--------	--------	11.30	------
12/16/28- 1/11/29	Sludge	1,295	250.2	--------	--------	--------	7.06	------
12/27/28	55 lbs. nidus tank skimmings	--------	9.5	--------	--------	--------	.12	------
Transfer dates	Make up sewage	2,402	17.6[3]	--------	--------	--------	.24	------
1/18/29	Total	11,878	2,280.5	24.5[4]	2,305	2,250	75.1	308.0

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[1] Data expressed in pounds.
[2] Not included in total.
[3] Calculated from average sewage analysis.
[4] Estimated from later analysis.

TABLE XI

REMOVALS FROM PRIMARY TANK AND ADDITIONS TO SECONDARY TANK

(Pounds)

Removals									Additions
Date	Character	Volume gallons	Dry solids	Ammonium compounds	Solids including ammonium compounds	Settling solids	Total nitrogen	Protein	Volume gallons
6/15/28– 6/21/28	Overflow	160	1.47	0.74	2.21	0.15	0.19	0.18	none
6/22/28– 6/24/28	No removals								none
6/25/28– 7/ 1/28	Overflow	224	2.42	1.05	3.47	0.16	0.26	0.09	none
7/ 2/28– 7/ 8/28	Overflow	191	2.02	0.92	2.94	0.22	0.28	0.31	none
7/ 9/28– 7/15/28	Overflow	203	2.22	0.96	3.18	0.20	0.38	0.87	none
7/16/28– 7/22/28	Overflow	294	4.23	1.92	6.15	0.60	0.65	0.94	none
7/23/28– 7/30/28	Overflow	481	11.98	3.80	15.78	5.15	1.49	2.87	none
7/30/28	Transfer sludge	520							520
7/31/28– 8/ 6/28	Overflow	379							379
8/ 7/28– 8/13/28	Overflow	392							392
8/12/28	Foaming		4.90	0.67	5.57	3.12	0.24	0.37	
8/14/28– 8/20/28	Overflow	404							104
8/19/28	Foaming		5.90	0.68	6.58	4.32	0.25	0.43	
8/21/28– 8/27/28	Overflow	374							374
8/21/28	Transfer sludge	114							114
8/28/28– 8/31/28	Overflow	213							213
8/30/28	Transfer sludge	61							61
9/ 1/28– 9/20/28	No removals								none
9/21/28– 9/27/28	Overflow	382							382
9/21/28	Transfer sludge	175							175
9/28/28–10/ 4/28	Overflow	423							423
10/ 5/28–10/11/28	Overflow	406							406
10/ 9/28	Transfer sludge	152							152
10/12/28–10/18/28	Overflow	405							405
10/15/28	Transfer sludge	47							47
10/19/28–11/15/28	Overflow	1,357							1,357
11/ 5/28 and 11/ 8/28	Transfer sludge	178							178
11/16/28–12/15/28	Overflow	1,686							1,686
11/16, 21, 26/28 and 12/1, 7, 13/28	Transfer sludge	651							651
12/16/28– 1/11/29	Overflow	1,262							1,262
12/19, 27/28 and 1/4, 12/29	Transfer sludge	504							504
1/18/29	Total[1]	11,683	35.14	10.74	45.88	13.92	3.74	6.07	9,565
1/18/29	Left in tank	2,000	132.00	15.60	147.60	138.50	7.65	20.20	
	Sum		167.14	26.34	193.48	152.42	10.39	26.27	

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[1] Total material out of Tank I, not entering Tank II.

TABLE XII

REMOVALS FROM SECONDARY TANK

(Pounds)

Date	Character	Volume gallons	Dry solids	Ammonium compounds	Solids including ammonium compounds	Settling solids[1]	Total nitrogen	Protein
6/15/28- 7/30/28	No removals	--------	--------	--------	--------	--------	--------	--------
7/31/28- 8/ 6/28	Overflow	369	4.53	1.21	5.74	1.19	0.51	1.13
8/ 7/28- 8/13/28	Overflow	382	5.09	1.24	6.33	1.24	0.91	1.57
8/14/28- 8/20/28	Overflow	394	4.97	1.81	6.28	0.44	0.69	1.19
8/21/28- 8/27/28	Overflow	365	4.74	2.08	6.82	0.31	0.59	0.31
8/21/28	Sludge	14	0.80	0.06	0.86	0.70	0.05	0.23
8/31/28	Overflow	100	1.31	0.42	1.73	0.10	0.20	0.58
8/28/28- 8/31/28	Overflow	208	2.94	1.31	4.05	0.27	0.44	0.56
8/30/28	Overflow	61	0.84	0.37	1.24	0.45	0.12	0.13
9/ 1/28- 9/20/28	No removals	--------	--------	--------	--------	--------	--------	--------
9/21/28- 9/27/28	Overflow	372	4.54	2.00	6.54	0.34	0.82	1.06
9/21/28	Sludge	38	26.60	0.23	26.83	26.20	0.82	4.68
9/21/28	Overflow	137	2.22	0.85	3.07	0.80	0.31	0.32
9/28/28-10/ 4/28	Overflow	412	4.91	3.08	7.99	0.41	0.96	0.55
10/ 5/28-10/11/28	Overflow	396	5.03	3.00	8.03	0.33	1.09	1.40
10/ 9/28	Overflow	152	2.44	1.21	3.65	0.08	0.43	0.57
10/12/28-10/18/28	Overflow	395	5.22	3.90	9.12	0.36	1.17	0.81
10/15/28	Overflow	45	0.78	0.43	1.21	0.47	0.14	0.06
10/19/28-11/15/28	Overflow	1,323	19.80	13.52	32.32	1.94	4.38	4.27
11/ 5/28 and 11/ 8/28	Overflow	174	2.67	1.73	4.40	0.48	0.59	0.58
11/16/28-12/15/28	Overflow	1,046	27.55	18.86	46.41	3.16	6.23	5.32
11/16, 21, 26/28 and 12/1, 7, 13/28	Overflow	651	13.15	7.85	21.00	6.40	4.22	0.88
12/16/28- 1/11/29	Overflow	1,230	21.25	13.21	34.46	2.06	4.55	4.73
12/19, 27/28 and 1/4, 12/29	Overflow	504	13.10	5.60	18.75	5.47	1.99	2.68
1/18/29	Sludge	50	42.25	0.86	43.11	41.00	1.08	5.28
1/18/29	Total	9,417	216.76	84.83	301.6	94.20	32.29	38.90
1/18/29	Left in tank	1,510	344.00	19.70	363.7	321.00	12.60	47.10
	Sum	--------	560.76	104.53	665.3	415.20	44.89	86.00

[1] Settling solids on sludge is calculated.

Tables XIII to XIX inclusive present the balances of the various materials considered in Part II under Solids Balance. Balances on grease, cellulose, crude fiber, and gas appear in Part III.

1. **Total Solids.** On a total solids basis the total overall digestion in the system was 62.6 per cent. From the standpoint of settling solids, Table XIV, the amount of liquefaction (gasification plus solution) is shown to have been 74.8 per cent. While this figure may be high due to the fact that the amounts of settling solids in sludges were calculated, it is to be expected that the liquefaction is greater than, the gasification.

2. **Protein.** The amount of protein digested, 63.5 per cent (see Table XV), is of the same order of magnitude as the digestion of total solids.

3. **Sulfate.** Table XVIII shows the sulfate digestion to be 83 per cent, but since the data is calculated, and not much is known about sulfate digestion, little can be said as to the accuracy of this figure.

4. **Ammonium Compounds.** Table XVI shows the total amount of ammonium compounds produced during the experiment.

5. **Total Nitrogen.** Table XVII shows that there is an apparent liberation of nitrogen (19.8 pounds) during digestion, but this amount of nitrogen was not considered in the gas for the reasons discussed in section 6 of Part II.

(6) *Conclusions.*

(a) Table XX presents a general summary of the chemical data treated in Part II. This table shows that under the conditions of this experiment (two-stage digestion maintained at 24°C. with sludge transferred about once a week) there can be obtained 63 per cent digestion of total solids, about 75 per cent liquefaction, 63 per cent digestion of protein, 83 per cent digestion of sulfate, and a dehydration of about 5 per cent of the total solids received.

TABLE XIII

TOTAL SOLIDS BALANCE[1]

	Pounds
Solids added to system	2305.0
Residue[2]	858.8
Solids digested in system	1446.2
Per cent digestion = 62.6.	

[1] Includes ammonium compounds.

[2] Sum of removals from system and residue In system at end of experiment.

TABLE XIV

SETTLING SOLIDS BALANCE

	Pounds
Settling solids added to system[1]	2250.0
Residue[1,2]	567.6
Settling solids liquefied	1682.4
Per cent liquefaction = 74.8.	

[1] Settling solids on fresh and digested sludge were calculated.

[2] See note 2, Table XIII.

TABLE XV

PROTEIN BALANCE[1]

	Pounds
Protein added to system	308.0
Residue[2]	112.3
Protein digestion	195.7
Per cent digestion = 63.5.	

[1] Protein = 6.25x (Total nitrogen — ammonia nitrogen).
[2] See note 2, Table XIII.

TABLE XVI

AMMONIUM COMPOUNDS BALANCE[1]

	Pounds
Ammonium compounds added to system	24.5
Residue[2]	130.9
Ammonium compounds produced	106.4

[1] Ammonium acetate and carbonate.
[2] See note 2, Table XIII.

TABLE XVII

TOTAL NITROGEN BALANCE[1]

	Pounds
Total nitrogen added to system	75.1
Residue[2]	55.3
Total nitrogen liberated[3]	19.8

[1] Kjeldahl nitrogen.
[2] See note 2, Table XIII.
[3] This is not considered in the gas.

TABLE XVIII

SULFATE BALANCE

	Pounds
Sulfate added to system[1]	90.0
Residue[1,2]	15.0
Sulfate digested	75.0
Per cent digestion = 83.0.	

[1] Based on data of A. L. Elder (11).
[2] See note 2, Table XIII.

TABLE XIX

DEHYDRATION

	Pounds
Solids digested	**1446.2**
Gas + sulfate	**1277.0**
Dehydration	**169.2**

TABLE XX

SUMMARY OF TABLES XIV TO XIX, INCLUSIVE

Material	Added, pounds	Residue, pounds	Digested, pounds	Produced, pounds	Liberated, pounds	Liquefied, pounds	Digestion, per cent	Liquefaction, per cent
Total solids	2,305.0	858.8	1,446.2				62.6	
Settling solids	2,250.0	567.6				1,682.4		74.8
Protein	308.0	112.3	195.7				63.5	
Sulfate	90.0	15.0	75.0				83.0	
Dehydration			169.2					
Ammonium compounds	24.5	130.9		106.4				
Total nitrogen	75.1	55.3			19.8			

(b) Scum collecting on the tanks can be controlled by the circulator.

(c) In the experiment it was shown that the production of ammonium compounds (acetate and carbonate) would, if not calculated in the balances, cause an error (due to loss on evaporation and drying of the solids) in the residue (see note *2*, Table XIII) of 15 per cent, and that carbon dioxide both dissolved and in bicarbonate form in the tank liquors must be calculated and added to the gas volume or an additional error will be introduced.

(d) Table XX shows an apparent liberation of nitrogen. While this is the strongest evidence so far obtained in this laboratory that there is liberation of gaseous nitrogen, nitrogen was not considered in the gas because, (1) of the known unreliability of the total nitrogen determination, and (2) because the nitrogen found in the gas could be accounted for as coming from solution, as shown by Buswell and Strickhouser[7].

(e) The apparent dehydration may have been a cumulative error. If so, it amounts to but 7.2 per cent which is probably within the limit of experimental error. However, other experiments and observations in this laboratory indicate that it may be an actual loss of hydrophyllic properties.

(f) Drainability curves in general bear out experience with sludge draining on small sand beds, that the secondary digestion tank sludge has better draining properties than primary tank sludge.

(g) Mineral oil (kerosene, etc.) found in the tanks at the end of the experiments was probably collected in the skimmings from the nidus tank or dispersed in the fresh sludge. Some of it was found to have vaporized from the digestion and collected in the pipes leading to the gas holders. Circulation probably dispersed some of the oil in the primary tank so that some of it was pumped to the secondary tank with each transfer of sludge. From there it had little chance to get out except in the gas. This accounts for the larger amount present in the secondary tank.

PART III

GAS, GREASE AND CELLULOSE BALANCE*

By A. M. BUSWELL AND E. L. PEARSON

Gas Production

Source of Gases. The earlier investigations of the processes in sewage sludge digestion tanks were mainly concerned with the bacteriology of the phenomena.

The chemistry of the anaerobic bacterial action was first studied by Popoff[41], who showed that methane, carbon dioxide and sometimes hydrogen arise from sewage slime diluted with water. Later Hoppe-Seyler[23] discovered that methane arose in the bacterial decomposition of calcium acetate; and Sohngen[48] developed a decantation method for obtaining a sludge rich in methane fermenters and a very vigorous digestion of this salt. He also observed that when he poured off the greater part of the liquor and treated the sludge with a mixture of carbon dioxide and hydrogen these gases disappeared quantitatively according to the equation:

$$4H_2 + CO_2 = CH_4 + 2H_2O.$$

The fact that less hydrogen is evolved than expected in experiments on digestion has been attributed by some to this reaction, but, although thermodynamically possible, the occurrence of the reaction has not been conclusively established by experiment.

Groenewege[16] reports "that methane can arise not only from calcium acetate but also from calcium formate, calcium butyrate, salts of higher fatty acids with an even number of carbon atoms, acetone, methyl, ethyl and n-butyl alcohols, glucose, lactose, peptone, egg albumin, pectin and gelatin."

Bach and Sierp[2] have studied the course of decomposition of a variety of typical food substances such as are likely to be present in domestic wastes. From each food they obtained carbon dioxide, methane and hydrogen, but in widely varied proportions and amounts. In general, foods rich in carbohydrates gave a higher proportion of carbon

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* Abstract of a thesis submitted in partial fulfillment of the requirements for the degree of Master of Arts in Chemistry, in the Graduate School of the University of Illinois, 1929.

dioxide, and those rich in proteins more methane. They also find that cooking the food changes the proportion of its decomposition products.

The occurrence of small amounts of hydrogen in the evolved gases is usually observed. Groenewege[16], Lymn and Langwell[31], and others have suggested various mechanisms for its formation.

Since carbon dioxide is produced in all the processes which give methane and hydrogen, there is left to consider only nitrogen, but the literature on nitrogen losses and denitrification is both voluminous and conflicting and is not of sufficient interest here to attempt a review. Buswell and Neave[5] have presented a comprehensive critical review of the question and conclude that strictly anaerobic experiments normally show no nitrogen liberation, thus confirming the early findings of Popoff.

Gases from Sewage Sludge. Among the older available data on the composition of sludge gases are those of Kinnicutt and Eddy at Worcester[30], Clark and Gage[9], Jesse[29], Fales[13], and Hommon[22]. Their results emphasize the striking difference in composition of gases under different conditions of production. However, under the present more or less standardized conditions of plant operation, most investigators agree that the composition of the gas liberated from sewage sludge may normally vary within somewhat narrower limits; methane 65 per cent to 90 per cent, carbon dioxide 5 per cent to 35 per cent, hydrogen 0 to 10 per cent, nitrogen 0 to 8 per cent.

Practically all the investigators have found the same factors to have an important effect on the rate of gas production, these factors being composition of sludge, time of digestion, temperature, and pH. Thus, it is generally agreed that gasification proceeds most favorably when the temperature is near 25°C. and the pH from 6.5 to 7.6. According to Sierp[47], Rudolfs[45], Baity[3], and Imhoff[27] maximum gas production occurs at about 25° to 28°C. Hatfield[17] at Decatur, where corn-product wastes comprise a large proportion of the sewage, reports an optimum temperature of 33° to 33°C. Baity[3], Fair and Carlson[12], and Eudolfs[44] recommend a pH range of 6.8 to 7.6.

Method of Gas Analysis. Gas analyses were made with an Illinois gas apparatus, as developed by Parr and Vandaveer[40]. With this apparatus absorptions (CO_2 and O_2) are made in a Morehead burette, the carbon dioxide being removed with 10 per cent NaOH, and the oxygen with alkaline pyrogallol. Hydrogen is burned in an electrically heated copper oxide furnace at 300°C., and methane is burned in oxygen over mercury in a slow combustion pipette. The accuracy of the apparatus has been verified by its inventors, Parr and Vandaveer[40], by comparison with standard methods of gas analysis. Since the absorptions are carried out over water and the reagents are washed down with water, the results are subject to error proportional to the solubility of the various constituents. However, since the most soluble constituents were removed first and minimum amounts of water were used for washing, these errors are considered negligible.

Results. As stated before records were kept of the amount of gas produced and analyses were made at frequent intervals. Since the gas holder was small and frequently filled three times in 24 hours, it was impossible to analyze a sample from each full holder. However, in order to secure as representative an average as possible, the number of samples taken was roughly proportional to the rate of gas production.

The data in Table XXI show that the CO_2 content of the gas from the primary tank varied from 17.6 to 34.8 per cent with an average of 29.5 per cent. This average is somewhat higher than that reported by most investigators. No certain explanation for this is evident from the data collected, but it may possibly be due to the predominance of cellulose and grease digestion in the primary tank. Even though the average CO_2 content of the gas from the secondary tank, where relatively little grease and cellulose are digested, is almost as high (28.3 per cent), the percentage of CO_2 in the latter half of the experiment, when transfers were made more often and considerably more grease entered the secondary tank, was higher than in the first half when less grease entered. Eudolfs[46] reports a similar correlation between CO_2 content and rate of grease digestion. At three different times during this experiment nidus tank skimmings, rich in grease, were added to the primary tank and no definite effect on CO_2 content could be noted; however, the actual weight of grease added in the form of skimmings was not an appreciable percentage of the digesting organic solids.

The greatest fluctuations in CO_2 content occurred at the beginning of the experiment and following a rest period (from August 31 to September 21 when no sludge was added to the tank). The two weeks following the rest period also showed the highest CO_2 content (31 per cent). Eudolfs[43], stated that an increase in CO_2 content could be used as an index of approaching trouble, and Buswell and Strickhouser[7] found some correlation between foaming and CO_2; however, no trouble of any kind was experienced in the present experiments, perhaps because of the liquor circulation already described in the introduction.

Of the 140 gas samples from both tanks, only three (from the primary tank) failed to show the presence of hydrogen; in the rest the percentage of hydrogen varied from 0.1 to 7.5 per cent, with an average of 3.4 per cent.

At various times during the experiment the gas covers were removed for inspection of the tank contents; at these times air entered the system and oxygen was found in the gas. Since oxygen was found at no other time the amount present was multiplied by five and reported as air; the measured volume, and the analyses, corrected to an air-free basis.

The nitrogen content of the gas varied from 0 to 35 per cent with an average of 4.3 per cent. The amount was highest following the admission of air to the system. In view of the findings of Buswell and Strickhouser[7] it is assumed here that the nitrogen can be accounted for by the air admitted, and as dissolved nitrogen in the added sludge. Therefore, the percentages of the other constituents are corrected to a nitrogen free basis.

The dissolved free CO_2 in solution in the overflow liquor shown in Table XXIII was determined as follows:

From the alkalinity and pH on all samples of material entering and leaving the tanks, the CO_2 was calculated according to the formula developed by Greenfield and Baker[15].

$$(H^+) = \frac{4CO_2 \times 10^{-7}}{(HCO_3^-)} + 1 \times 10^{-8}.$$

To this was added the bicarbonate CO_2 calculated from the alkalinity[49]. The 67 pounds of CO_2 (free + bicarbonate) shown in Table XXV represent the difference between the CO_2 leaving the tank in sludge and overflow liquor and that entering in the fresh sludge. It is admittedly only a rough approximation, because the above formula is not valid in the presence of buffers other than bicarbonates and the overflow liquors contain considerable amounts of volatile acid salts. Prom the solubility constants of methane was calculated the amount of dissolved methane that was lost. This amounted to so small a fraction of the total (less than .2 per cent) that it was not considered.

The entire gas data for the experiment are given in Tables XXI, XXII, XXIII, XXIV, and XXV.

TABLE XXI

ANALYSES OF GAS

Results Expressed in Per Cent[1]

Date	Primary tank					Secondary tank				
	CO_2	H_2	CH_4	N_2[2]	Air[3]	CO_2	H_2	CH_4	N_2[2]	Air[3]
6/20/28	19.8	4.4	65.3	10.5	0.0	--------	--------	--------	--------	--------
6/26/28	24.1	5.6	51.8	18.5	13.0	--------	--------	--------	--------	--------
6/28/28	25.2	0.6	61.5	12.7	0.0	--------	--------	--------	--------	--------
7/ 3/28	27.8	2.8	60.7	8.7	0.0	--------	--------	--------	--------	--------
7/ 9/28	28.4	2.8	60.6	8.2	0.0	--------	--------	--------	--------	--------
7/16/28	27.0	3.6	60.5	8.9	0.0	--------	--------	--------	--------	--------
7/23/28	28.0	2.0	62.0	8.0	0.0	--------	--------	--------	--------	--------
7/30/28	25.6	3.4	68.3	2.7	0.0	--------	--------	--------	--------	--------
7/31/28	20.4	3.6	61.8	13.8	0.0	--------	--------	--------	--------	--------
8/ 6/28	25.0	3.4	64.4	7.2	0.0	21.0	0.0	75.0	4.0	0.0
8/21/28	26.4	4.8	65.6	3.2	7.5	27.3	2.5	69.4	0.8	13.0
9/20/28	29.4	4.7	58.4	7.5	5.0	20.4	4.7	71.3	3.6	3.0
9/22/28	17.6	6.5	41.1	35.8	16.0	17.8	6.1	53.1	23.0	11.0
9/24/28	30.0	4.2	65.9	0.0	6.5	26.3	5.9	61.1	6.7	2.0
9/25/28	31.7	4.2	68.7	0.0	4.5	28.5	5.3	63.2	5.0	7.0
9/26/28	34.8	6.0	61.0	0.0	9.0	27.6	7.5	63.6	0.0	8.5
9/27/28	30.6	5.1	--------	--------	2.0	25.8	2.3	66.1	5.8	3.0
10/ 1/28	30.3	5.9	57.0	6.8	1.0	22.9	7.7	64.7	7.7	1.3
10/ 2/28	30.4	3.9	63.1	2.6	2.0	--------	--------	--------	--------	--------
10/ 3/28	32.5	3.8	62.6	1.1	7.0	--------	--------	--------	--------	--------
10/ 4/28	31.9	3.7	64.6	0.0	6.0	24.4	3.9	64.4	8.3	3.5
10/ 5/28	32.6	0.0	68.8	0.0	9.5	--------	--------	--------	--------	--------
10/ 8/28	30.1	2.6	63.0	4.3	1.0	--------	--------	--------	--------	--------
10/10/28	28.9	2.6	63.9	4.6	1.0	--------	--------	--------	--------	--------
10/11/28	29.4	5.7	63.1	1.8	1.0	--------	--------	--------	--------	--------
10/12/28	28.6	3.8	63.8	3.8	0.0	23.5	4.2	64.9	7.5	3.0
10/15/28	27.8	4.2	64.3	3.7	0.0	--------	--------	--------	--------	--------
10/16/28	24.6	2.6	66.5	6.3	0.0	--------	--------	--------	--------	--------
10/17/28	30.1	2.1	69.9	0.0	7.0	24.7	1.8	66.2	7.3	3.0
10/18/28	28.6	2.6	67.1	1.7	0.0	22.8	1.8	71.0	4.4	0.0
10/19/28	28.6	4.3	67.1	0.0	2.0	--------	--------	--------	--------	--------
10/22/28	27.5	5.6	63.8	3.2	1.0	--------	--------	--------	--------	--------
10/23/28	28.5	4.8	64.7	2.0	4.0	--------	--------	--------	--------	--------
10/24/28	27.7	2.2	67.7	2.4	6.0	--------	--------	--------	--------	--------
10/25/28	27.8	4.8	68.3	0.0	3.5	24.5	5.3	61.8	8.4	24.0
10/26/28	30.2	4.7	64.9	0.2	2.0	26.8	6.8	63.8	2.6	23.0
10/29/28	29.6	3.1	61.4	5.9	2.0	--------	--------	--------	--------	--------
10/30/28	30.5	2.3	65.4	1.8	3.0	30.6	3.7	60.6	5.1	2.0
10/31/28	29.1	0.1	62.8	8.0	2.0	--------	--------	--------	--------	--------
11/ 1/28	28.6	1.8	64.4	5.2	2.0	28.8	1.6	63.1	6.5	2.0
11/ 3/28	28.7	--------	--------	--------	0.0	28.5	--------	--------	--------	--------
11/ 5/28	26.9	2.4	69.3	1.4	2.0	25.4	3.7	60.6	5.1	2.0
11/ 6/28	26.9	2.3	63.0	7.8	2.0	--------	--------	--------	--------	--------
11/ 7/28	29.7	2.0	68.6	0.0	2.0	28.4	3.3	61.4	6.9	1.0
11/ 8/28	28.9	6.7	65.0	0.0	2.0	28.3	4.1	63.4	4.2	4.0
11/12/28	29.4	1.8	67.6	1.2	0.0	27.7	4.4	64.3	3.6	1.0
11/13/28	30.9	2.0	65.2	1.9	1.0	30.1	4.6	60.8	4.5	1.0
11/14/28	29.2	5.8	62.9	2.1	0.0	30.0	5.0	60.3	4.7	0.0
11/15/28	28.4	1.7	66.4	3.5	0.0	28.0	5.2	59.0	7.8	0.0
11/19/28	29.0	3.2	64.6	3.2	0.0	29.6	4.4	60.9	5.1	0.0
11/20/28	29.4	5.6	60.3	4.7	0.0	32.4	6.8	57.4	3.4	0.0
11/21/28	28.2	7.0	61.2	3.6	0.0	--------	--------	--------	--------	--------
11/22/28	29.2	6.0	64.8	0.0	0.0	28.4	6.4	62.8	2.4	0.0
11/26/28	29.4	3.8	59.4	7.4	0.0	--------	--------	--------	--------	--------
11/27/28	29.6	3.8	62.0	4.6	0.0	26.8	5.4	62.2	5.6	0.0
11/28/28	29.2	6.0	59.8	11.0	0.0	--------	--------	--------	--------	--------
11/30/28	27.6	5.4	64.6	2.4	0.0	25.0	7.4	66.4	1.2	0.0
12/ 4/28	29.0	7.0	61.2	2.8	0.0	27.5	6.7	65.1	0.7	0.0
12/ 7/28	30.4	6.8	58.3	4.5	0.0	27.7	7.0	64.5	0.8	0.0
12/10/28	26.1	2.7	68.8	2.4	0.0	24.5	1.0	64.2	10.3	1.0
12/11/28	30.4	3.8	58.5	7.3	0.0	30.0	3.4	60.8	5.8	0.0
12/12/28	28.2	4.6	61.8	6.4	0.0	27.7	2.5	66.6	3.2	0.0
12/13/28	29.2	1.0	62.4	7.4	0.0	26.8	2.8	66.0	4.4	0.0
12/14/28	27.2	2.4	65.4	5.0	0.0	25.2	1.0	68.8	5.0	1.0
12/17/28	28.0	1.4	63.4	7.2	0.0	29.7	2.1	61.1	7.1	0.0
12/18/28	28.2	2.9	66.6	2.3	0.0	28.4	2.8	60.7	8.1	0.0
12/19/28	26.2	1.8	69.1	2.9	0.0	29.0	2.4	61.5	7.1	0.0
12/20/28	26.4	2.8	63.5	7.3	15.0	28.4	1.0	67.8	2.8	0.0

TABLE XXI—Concluded.

Date	Primary tank					Secondary tank				
	CO_2	H_2	CH_4	N_2[2]	Air[3]	CO_2	H_2	CH_4	N_2[2]	Air[3]
12/21/28	28.1	2.2	64.4	5.3	11.0	28.0	2.0	66.1	3.9	0.0
12/24/28	28.5	2.1	65.3	4.1	0.0	28.0	1.8	66.5	3.7	0.0
12/26/28	32.0	2.4	62.5	3.1	0.0	29.4	1.5	62.7	6.4	0.0
12/27/28	29.0	1.7	65.1	4.2	6.0	27.0	1.6	69.0	2.4	0.0
12/28/28	30.4	1.8	67.4	0.4	2.0	28.6	1.6	66.7	3.1	0.0
12/31/28	31.0	0.0	64.2	4.8	0.0	28.6	1.4	67.4	2.6	0.0
1/ 2/29	26.4	2.2	70.0	1.4	0.0	25.4	3.0	71.2	0.4	0.0
1/ 3/29	31.0	1.6	67.0	0.4	0.0	27.2	0.4	68.8	3.6	0.0
1/ 4/29	29.7	2.1	64.6	3.6	0.0	31.0	1.6	61.1	6.3	0.0
1/ 7/29	26.8	2.2	69.6	1.4	0.0	28.0	2.4	67.8	1.8	0.0
1/ 8/29	28.8	2.5	61.9	6.8	0.0	29.8	2.0	63.6	4.6	0.0
1/ 9/29	32.7	---------	---------	---------	0.0	33.1	---------	---------	---------	---------
1/10/29	29.2	---------	---------	---------	0.0	29.4	---------	---------	---------	---------
1/11/29	29.8	2.3	65.4	2.5	0.0	31.6	4.0	58.8	5.6	0.0
1/14/29	22.2	2.8	71.0	4.0	0.0	25.5	2.2	71.7	0.6	2.0
1/15/29	23.3	5.0	69.9	1.8	8.0	---------	---------	---------	---------	---------
1/16/29	26.6	0.0	71.9	1.5	1.0	---------	---------	---------	---------	---------
1/17/29	27.5	4.7	62.0	5.8	3.0	23.9	3.1	70.3	2.7	0.0
Average	28.3	3.4	64.2	4.3	(2.2)	27.0	3.5	64.8	4.9	(2.3)
Corrected to N_2 Free basis	29.5	3.6	66.9	---------	---------	28.3	3.7	68.0	---------	---------

[1] Calculated to an air-free basis ; [2] By difference ; [3] The oxygen determined was multiplied by five and reported as air.

TABLE XXII

GAS VOLUME SUMMARY

Month	Primary tank					Secondary tank			
	Mean monthly barometric pressure	Manometer pressure	Total pressure	Cubic feet of gas at 0°C.	Cubic feet of gas at 0°C. 760 m.m.	Manometer pressure	Total pressure	Cubic feet of gas at 0°C.	Cubic feet of gas at 0°C. 760 m.m.
June (10 days)	755.14	.44	755.58	107.5	107	---------	---------	---------	---------
July	758.95	.99	759.94	1,739.5	1,739	---------	---------	---------	---------
August	759.72	1.06	760.78	2,349.0	2,350	1.03	760.75	691.0	692
September	760.99	1.17	762.16	1,090.0	1,095	1.47	762.46	97.5	98
October	761.75	1.10	762.85	3,559.0	3,565	1.17	762.92	129.7	130
November	762.26	1.03	763.29	3,657.0	3,670	1.17	763.43	356.0	358
December	762.26	1.10	763.36	2,820.0	2,830	1.17	763.43	432.0	434
January (18 days)	762.51	1.40	763.91	921.0	924	1.03	763.54	373.0	375
Total					16,280	Total			2,087
Air (2.2%)					358	Air (2.3%)			46
Air free volume					15,922	Air free volume			2,041
N_2 (4.3%)					684	N_2 (4.4%)			100
Gas produced (CH_4, H_2, CO_2)					15,238	Gas produced (CH_4, CO_2, H_2)			1,941

TABLE XXIII

COMPOSITION OF GAS

Primary tank				Secondary tank			
Component	Per cent[1]	Cubic feet	Pounds	Component	Per cent[1]	Cubic feet	Pounds
CH_4	66.9	10,200	455.0	CH_4	68.0	1,320	59.0
CO_2	29.5	4,490	550.0	CO_2	28.3	550	67.5
H_2	3.6	548	3.1	H_2	3.7	71	0.4
Sum of CH_4, CO_2 and H_2		15,238	1,008.1	Sum of CH_4, CO_2 and H_2		1,941	126.9
CO_2 in overflow liquor		449	55.3	CO_2 in overflow liquor		95	11.7
Total produced in primary tank		15,687	1,063.4	Total produced in secondary tank		2,036	142.6
Total produced in both tanks, 1,202 pounds.							

[1] Corr. to nitrogen free basis.

TABLE XXIV

CARBON DIOXIDE IN SOLUTION*

Primary Stage

	Pounds
Out	111.5
In	77.0
Difference	34.5
Left in tank	20.8
CO_2 produced	55.3

Secondary Stage

In	100.2
Out	91.0
Difference	9.2
Left in tank	20.9
CO_2 produced	11.7

Overall Production

Primary stage	55.3
Secondary stage	11.7
Total	67.0

Distribution of Production

		Per cent
Primary stage	$\frac{55.3}{67.0} =$	82.5
Secondary stage	$\frac{11.7}{67.0} =$	17.5

---

* From data in Tables XXVI, XXVII, and XXVIII.

TABLE **XXV**

GAS PRODUCTION*

Primary Stage

	Pounds
Methane, Carbon dioxide, hydrogen, in gas	1008.1
Carbon dioxide (dissolved and bicarbonate)	55.3
Total gas produced	1063.4

Secondary Stage

Methane, carbon dioxide, hydrogen, in gas	126.9
Carbon dioxide (dissolved and bicarbonate)	11.7
Total gas produced	138.6

Overall Production

Primary stage	1063.4
Secondary stage	138.6
Total	1202.0

Distribution of Production

		Per cent
Primary stage	$\frac{1063.4}{1202.0} =$	88.4
Secondary stage	$\frac{138.6}{1202.0} =$	11.6

* Based on data in Table XXVII.

The relation between gas production and total solids added to the system is shown in Figures 24 and 25, and by the following summary:

	Cubic Feet
Gas produced per pound of solids added[1]	7.7
Gas produced per pound of solids digested[1]	12.1
Gas produced per capita per day[2]	0.39
Gas produced per capita per day—	
Primary	0.34
Secondary	0.05

---

[1] These figures include dissolved and bicarbonate CO_2 and are corrected to an air and nitrogen free basis.

[2] This figure is corrected to an air and nitrogen free basis and does not include dissolved and bicarbonate CO_2.

Any discrepancy between the above summary and the data in Figures 24 and 25 is due to the fact that the latter data are not corrected to an air free and nitrogen free basis, and do not account for dissolved and bicarbonate carbon dioxide. Since most of the gas was produced in the primary tank without a concurrent production of a well digested sludge, it seems that an essential difference in the functions of the two tanks is that digestion in the primary stage is a gasifying process, while that in the secondary stage is a liquefying process.

Of more practical significance is the fact that nearly 89 per cent, of the total gas was produced in the primary tank. This quantity, representing 0.39 cubic feet per capita per day, compares favorably with the production at other installations utilizing complete digestion. Thus, Imhoff[26] reports 0.28 cubic feet per capita per day from an unheated tank. Priiss[42] reports 4.55 cubic feet per pound volatile matter from a heated tank without circulation; 5.32 cubic feet per pound from a heated tank with circulation, but incomplete digestion; and 9.1 cubic feet per pound from a heated tank with sludge circulation and complete digestion.

Figure 24 gives a graphic representation of the difference it the gasification from the two stages of digestion. The general slope of the curve (except when there were no sludge additions) shows that the rate of gas production was fairly constant, and following the general constancy of the rate of solids added (Figure 25). The curve showing the cubic feet of gas per pound of sludge added (Figure 25) shows that the system was not working at capacity until after the first of November. From that time on transfers were being made at intervals of 5 to 7 days. This may point to the information that frequent transfer is necessary to produce the greatest efficiency and gas production. Data based on pounds of sludge added may be misleading because the gas production is in reality dependent on volatile matter. For comparison with data from other sources it might be noted here that since the average volatile matter in the sludge is about 75 per cent, the gas per pound of volatile matter added was approximately 10.3 cubic feet. On this basis also gas per pound of volatile matter digested was 16.4 cubic feet.

Grease and Cellulose Digestion

Digestion of Fatty Substances. O'Shaughnessy[39] pointed out that a little over one-third of the fatty matter of sewage sludge is lost during digestion. Groenewege[16] has shown that calcium formate, calcium butyrate, and salts of higher fatty acids having an even number of carbon atoms can produce methane, and Neave and Buswell[34] have found that the "grease" (petroleum ether extractable matter) in Urbana sewage sludge consists of nearly equal amounts of lime soaps and unsaponified fats, the bulk of the fatty acids being of animal origin, mainly palmitic and stearic.

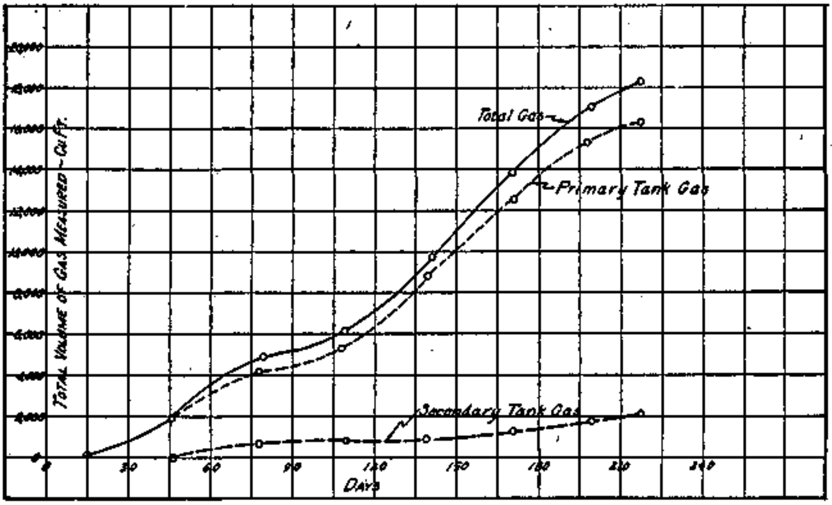


FIG. 24.—CURVE SHOWING TOTAL GAS MEASURED DURING EXPERIMENT.

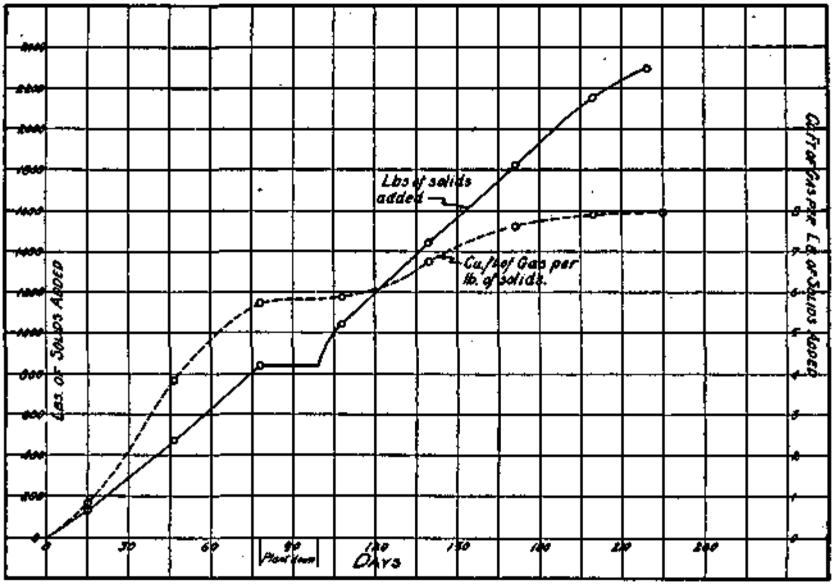


FIG. 25.—RELATION BETWEEN GAS EVOLUTION AND SOLIDS ADDED.

In 1926 Neave and Buswell started an extensive investigation of the anaerobic decomposition of fatty substances which is still being carried on. Two progress reports have so far been published. In the first[35] they reported that in the acid type of digestion a rapid destruction of grease and calcium soaps occurred with the production of lower fatty acids some of which ferment further to give methane; also that the rate of digestion measured by gas production was roughly proportional to the grease content of the solids.

In a later paper[36] the same authors showed that "(1) sewage grease and soaps are decomposed during the normal alkaline digestion of sludge as well as during the acid phase; (2) the lower fatty acids produced can, under these conditions, decompose further to give methane; and (3) high temperatures (37°C.) favor the digestion of sludge components other than grease more than the digestion of grease itself."

Heukelekian[20] has reported that the total yield of gas is not correlated with the fat content of the New Jersey sludge.

Digestion of Cellulose. The earliest work on the decomposition of cellulose was done by Popoff[41], who inoculated cellulose with sewer slime and found that the evolved gases contained carbon dioxide, methane, hydrogen, and nitrogen in various percentages depending on the time of incubation. He concluded that cellulose gave carbon dioxide and methane only, the hydrogen and nitrogen coming from other sources.

Hoppe-Seyler[23] reported a long series of experiments begun in 1881 in which about 25 grams of cellulose were incubated with sewage slime. At the end of the 5 years' incubation 15 grams of cellulose had disappeared, and 3,281 cc. of carbon dioxide and 2,571 cc. of methane had been produced. He concluded that cellulose fermented to carbon dioxide and methane in equal proportions.

Omeliansky[38] demonstrated the anaerobic decomposition of cellulose by two organisms, the one forming methane and carbon dioxide, and the other hydrogen and carbon dioxide. In both digestions acetic and butyric acids were produced as end-products; however, more recent work (McBeth and Scales[32]) has shown that Omeliansky's alleged organisms were mixed cultures, and the bacteriology of anaerobic cellulose decomposition still requires investigation.

Likewise there appears to be little known about the chemistry of cellulose degradation in sewage sludge. The only study of this nature in the literature is that of Heukelekian[10], who found that cellulose decomposed rapidly (79 per cent in three weeks in unlimed and 96 per cent in three weeks-in limed bottles); the products were not reported except that they were acid in character.

Analytical Methods.

1. **Fatty Substances.** Patty substance was determined by complete extraction with petroleum ether (B. P. 60-70°C.) in a Soxhlet apparatus. Since the extracted material consists of about equal parts of calcium soaps and unsaponified fats, the results are high by the amount of calcium present.

2. **Cellulose.** The sample left after the grease determination was transferred to a test tube, 20 cc. of Schweitzer's reagent added and allowed to stand, with intermittent shaking, for 48 hours. The extract was filtered through a Gooch crucible and an aliquot portion placed in a 50 cc. Nessler tube. This was diluted with water and 7 cc. of concentrated HC1 added to precipitate the cellulose. When the precipitate had settled out it was filtered onto a Gooch crucible, washed with water, dried at 105°C. and weighed as cellulose.

Time did not permit investigation of the accuracy of this procedure, but some inconsistencies in the determinations indicate that the method may give low results. It determines only the alpha-cellulose and not ligno-cellulose nor cellulose degradation products; however, the work of Heukelekian[10] indicated that only the alpha-cellulose is attacked by the anaerobic flora in the digestion periods under consideration.

Summary. Tables XXVI, XXVII, and XXVIII show the summaries of the grease, cellulose, and carbon dioxide additions and removals to the system. These tables were made in the same manner that the summaries in Part II were made. Tables XXIX and XXX show the grease and cellulose balances, made from the data in Tables XXVI, XXVII, and XXVIII. From these balances it is apparent that 90.3 per cent of the grease received in the system is digested and gasified, and that 92.5 per cent of the alpha-cellulose treated is digested. The amount of crude fiber digested is shown in Table XXXI, and the method of calculating the figure of 294 pounds is apparent from the table.

Conclusions. (a) The experiment gives strong indirect evidence that grease digests to give gas, for the amount of grease that was digested must be accounted for in the gas, there being no other products that would account for this weight of material digested.

b. This statement holds also for alpha-cellulose.

c. The fact that the majority of the gas is produced in the primary stage and analysis of transferred sludge showed very little grease and cellulose present, leads to the conclusion that both grease and cellulose digest rapidly (at least within a period of 6 to 8 days) to give gas.

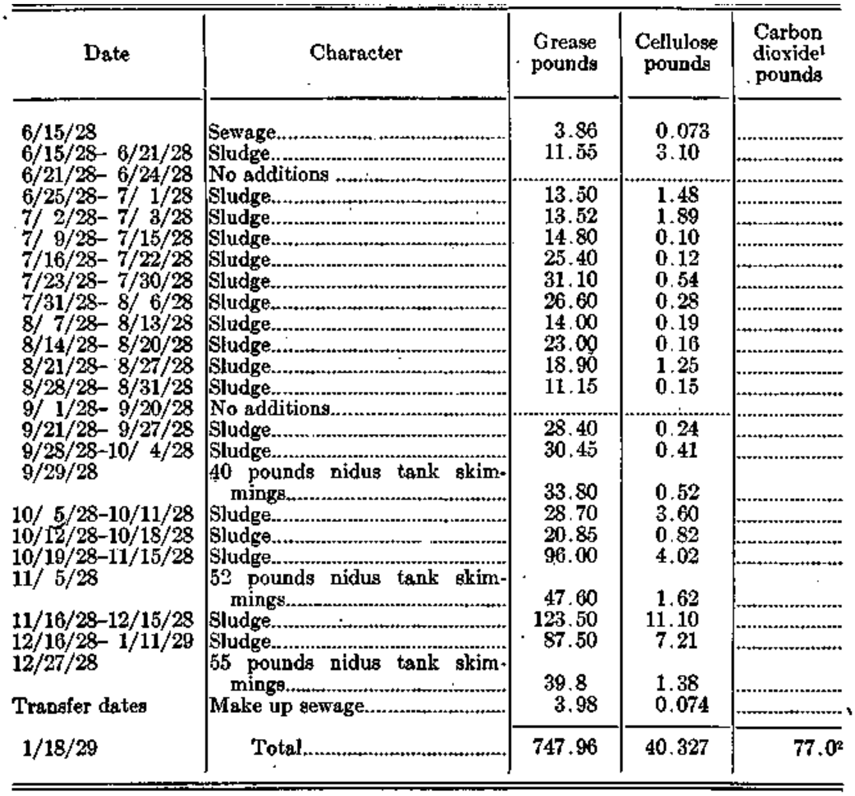
d. Protein as has been shown in Part II, also digested to give gas.

e. Since the amounts of "grease", cellulose, and protein digested do not equal the amount of gas produced, it is apparent that crude fiber also digested to give gas.

f. It can also be pointed out that two-stage digestion favors gas production when the pH of the system remains above 7.0 and the system is kept at a temperature of 24° - 25°C.

TABLE XXVI

ADDITIONS TO PRIMARY TANK



Date	Character	Grease pounds	Cellulose pounds	Carbon dioxide[1] pounds
6/15/28	Sewage	3.86	0.073	
6/15/28- 6/21/28	Sludge	11.55	3.10	
6/21/28- 6/24/28	No additions			
6/25/28- 7/ 1/28	Sludge	13.50	1.48	
7/ 2/28- 7/ 3/28	Sludge	13.52	1.89	
7/ 9/28- 7/15/28	Sludge	14.80	0.10	
7/16/28- 7/22/28	Sludge	25.40	0.12	
7/23/28- 7/30/28	Sludge	31.10	0.54	
7/31/28- 8/ 6/28	Sludge	26.60	0.28	
8/ 7/28- 8/13/28	Sludge	14.00	0.19	
8/14/28- 8/20/28	Sludge	23.00	0.16	
8/21/28- 8/27/28	Sludge	18.90	1.25	
8/28/28- 8/31/28	Sludge	11.15	0.15	
9/ 1/28- 9/20/28	No additions			
9/21/28- 9/27/28	Sludge	28.40	0.24	
9/28/28-10/ 4/28	Sludge	30.45	0.41	
9/29/28	40 pounds nidus tank skimmings	33.80	0.52	
10/ 5/28-10/11/28	Sludge	28.70	3.60	
10/12/28-10/18/28	Sludge	20.85	0.82	
10/19/28-11/15/28	Sludge	96.00	4.02	
11/ 5/28	52 pounds nidus tank skimmings	47.60	1.62	
11/16/28-12/15/28	Sludge	123.50	11.10	
12/16/28- 1/11/29	Sludge	87.50	7.21	
12/27/28	55 pounds nidus tank skimmings	39.8	1.38	
Transfer dates	Make up sewage	3.98	0.074	
1/18/29	Total	747.96	40.327	77.0[2]

[1] Dissolved CO_2 and bicarbonate CO_2.

[2] Estimated.

TABLE XXVII

REMOVAL FROM PRIMARY TANK AND ADDITIONS TO SECONDARY TANK

Removals from primary tank					Additions to secondary tanks
Date	Character	Grease	Cellulose	Carbon dioxide[1]	Carbon dioxide[1]
6/16/28– 6/21/28	Overflow	0.24	.050	0.46	
6/22/28– 6/24/28	No removals				
6/25/28– 7/ 1/28	Overflow	0.48	.025	1.17	
7/ 2/28– 7/ 8/28	Overflow	0.28	.036	1.12	
7/ 9/28– 7/15/28	Overflow	0.69	.022	1.36	
7/16/28– 7/22/28	Overflow	0.99	.059	2.02	
7/23/28– 7/30/28	Overflow	2.89	.017	3.60	
7/30/28	Transfer sludge			4.18	4.18
7/31/28– 8/ 6/28	Overflow			2.48	2.48
8/ 7/28– 8/13/28	Overflow			3.42	3.42
8/12/28	Foaming	0.99	.088	0.68	0.68
8/14/28– 8/20/28	Overflow			3.62	3.63
8/19/28	Foaming	1.08	.035	0.89	0.89
8/21/28– 8/27/28	Overflow			3.66	3.66
8/21/28	Transfer sludge			1.18	1.18
8/28/28– 8/31/28	Overflow			1.97	1.97
8/30/28	Transfer sludge			0.60	0.60
9/ 1/28– 9/20/28	No removals				No additions
9/21/28– 9/27/28	Overflow			3.58	3.58
9/21/28	Transfer sludge			1.77	1.77
9/28/28–10/ 4/28	Overflow			3.40	3.40
10/ 5/28–10/11/28	Overflow			3.30	3.30
10/ 9/28	Transfer sludge			1.60	1.60
10/12/28–10/18/28	Overflow			3.81	3.81
10/15/28	Transfer sludge			0.54	0.54
10/19/28–11/15/28	Overflow			18.86	18.86
11/ 5/28 and 11/ 8/28	Transfer sludge			2.24	2.24
11/16/28–12/15/28	Overflow			17.69	17.69
11/16, 21, 26 and 12/1, 7, 13/28	Transfer sludge			7.45	7.45
12/16/28 and 1/11/29	Overflow			9.31	9.31
12/19, 27/28 and 1/4, 12/29	Transfer sludge			5.57	5.57
1/18/29	Total	7.84	.332	111.53	100.2
1/18/29	Left in tank	17.9	.70	20.8	
	Sum	25 74	1.03	132.3	

[1] Dissolved CO_2 and bicarbonate CO_2.

TABLE XXVIII

REMOVALS FROM SECONDARY TANK

Date	Character	Grease	Cellulose	Carbon dioxide
6/15/28– 7/29/28	No removals			
7/13/28– 8/ 6/28	Overflow	0.73	.064	1.83
8/ 7/28– 8/13/28	Overflow	0.90	.122	2.76
8/14/28– 8/20/28	Overflow	0.59	.125	3.34
8/21/28– 8/27/28	Overflow	0.79	.123	3.30
8/21/28	Sludge	0.05	.001	0.12
8/21/28	Overflow	0.75	.002	0.85
8/28/28– 8/31/28	Overflow	0.13	.018	1.73
8/30/28	Overflow	0.05	.004	.55
9/ 1/28– 9/20/28	No removals			
9/21/28– 9/27/28	Overflow	0.18	.027	3.20
9/21/28	Sludge	2.24	.019	0.34
9/21/28	Overflow	0.11	.008	1.22
9/28/28–10/ 4/28	Overflow	0.24	.116	3.68
10/ 5/28–10/11/28	Overflow	0.21	.137	3.35
10/ 9/28	Overflow	0.13	.050	1.31
10/12/28–10/18/28	Overflow	0.24	.117	3.74
10/15/28	Overflow	0.04	.020	0.42
10/19/28–11/15/28	Overflow	0.83	.242	13.10
11/ 5/28 and 11/8/28	Overflow	0.18	.050	1.75
11/16/28–12/15/28	Overflow	1.02	.182	18.52
11/16,21,26/28 and 12/1, 7, 13/28	Overflow	0.63	.140	6.94
12/16/28– 1/11/29	Overflow	0.96	.128	12.72
12/19, 27/28 and 1/4, 12/29	Overflow	1.01	.150	5.70
1/18/29	Sludge	4.27	.040	0.54
1/18/29	Total	16.28	1.885	91.01
1/18/29	Left in tank	30.9	.100	20.9
	Sum	47.18	1.99	111.9

TABLE XXIX

GREASE[1] BALANCE

	Pounds
Grease added to system	747.96
Residue[2]	72.9
Grease digested	675.06
Per cent digestion = 90.3.	

[1] Petroleum ether extractable material.

[2] Sum of removals from system and residue in system at end of experiment.

TABLE XXX

CELLULOSE[1] BALANCE

	Pounds
Cellulose added to system	40.33
Residue[2]	3.02
Cellulose digested	37.3
Per cent digestion = 92.5.	

[1] Alpha cellulose.

[2] See note 2, Table XXIX.

TABLE XXXI

CRUDE FIBRE DIGESTION[1]

	Pounds
Gas	1202.0
Grease + cellulose + protein	908.0
Crude fibre digestion	294.0

[1] Does not include alpha-cellulose.

PART IV

THE USE OF DIGESTION TANK LIQUOR INSTEAD OF SLUDGE FOR SEEDING

By A. M. BUSWELL, G. E. SYMONS, AND E. L. PEARSON

Following the end of the experiment reported in Parts II and III, no further data were obtained on sludge digestion until March. "From March 4, 1929, until September 24, 1929, three separate experiments were carried out. The value of seeding tanks with sludge has long been known, but the possibility of seeding with liquor has never been determined, and so was investigated by comparing the digestion in a seeded tank with two separate runs on an unseeded tank. Data were also collected on lagooning as suggested in the introduction.

The first run on an unseeded tank was started in tank No. I (Figure 4) on March 4, 1929. Additions were made to this tank until May 21, 1929, at which time it was apparent that normal digestion, as evidenced by gas production, had been taking place for two weeks. On this latter date 1,330 gallons of liquor (.173 per cent solids) were transferred for seeding purposes from this tank to tank No. II (Figure 4). Both tanks were filled with sewage. On June 19 the tank (No. I) was drained-down, to make ready for another unseeded experiment.

From May 21, 1929, to June 21, 1929, additions were made to the seeded tank (No. II). Additions to this tank were stopped on June 21, and it was allowed to continue digestion for 7 weeks. On August 9 it was drained down to be rebuilt for further experimentation.

On June 22 the primary tank (No. I) was filled with sewage (no seeding) and regular additions of sludge were made to this tank until September 24. During this latter experiment with an unseeded tank, sludge was transferred once a week to an open tank of about 600 gallons capacity. This latter tank was thus used in the capacity of a lagoon. In regard to operating conditions, sampling procedures, analyses, etc. in these three experiments, there were few departures from the methods used in the previous experiment on two-stage digestion. The following instances cover the exceptions:

(1) The average temperature in the three experiments was 25°C.

(2) During the first few weeks of the first experiment on an unseeded tank, the fresh sludge was circulated without aeration from the bottom to the top of a barrel, for a period of one-half to three hours, before it was added to the tank. This circulation was done by a small centrifugal pump. Circulation in this manner produced a dispersing or emulsifying effect. Though a beneficial result in the way of stimulating digestion was hoped for, none was evidenced and the procedure was stopped. Apparently normal digestion, as evidenced by gas production, requires the development of a proper flora, and this development is established either naturally or by seeding and is not aided by increasing the degree of dispersion of the sludge to be digested. The process of emulsification served to increase the ammonia nitrogen, alkalinity, and per cent of non-settling solids.

(3) In all three experiments alkalinity and pH were determined on centrifuged samples.

(4) Cellulose and crude fiber were not determined.

(5) Per cent solids by volume was determined on digested sludges (both from tank *No.* I and lagoon tank) by centrifuging a sample for 30 minutes at 1,200 r. p. m. and measuring the volume occupied by the sludge.

(6) In the third experiment (unseeded tank), as has been stated, the sludge from the tank was transferred once a week to a lagoon tank. As often as possible when liquor and sludge in the lagoon had separated, the liquor was siphoned off, care being taken not to disturb the sludge that had settled or that had floated to form a scum. This procedure served to concentrate the sludge in the lagoon tank.

Daily data and summaries show that the character of the sludge added in the three experiments did not vary. It carried about 2.5 per cent solids, so that data collected on gas production are comparable.

Figure 26 shows the gas production and the methane content of the gas in the three experiments. The dotted portion of curve No. 1 from the thirty-seventh to the sixty-second day covers a period of days when there were gas leaks. The data were estimated from measurements of the leaks. It is evident from the curves that gasification in unseeded tanks does not start appreciably for 25 days and that maximum gasification or normal digestion is not reached for about 15 days after gasification starts. It is also apparent that the digestion followed the same trend in both unseeded tanks. In the tank seeded with liquor, however, gasification starts immediately and the rate of maximum gasification is reached in 18 to 20 days, or in half the length of time required to reach the same state of digestion in unseeded tanks.

The daily data show that during the first month of feeding the methane content in the unseeded tank gas (first experiment) was higher than that in the gas from the seeded experiment by about 10 per cent, and that the reverse was true in the third experiment.

The carbon dioxide content follows an inverse course to the change in methane content of the gas.

Data obtained on the lagooning of sludge from a primary digestion stage at weekly intervals is shown in Table XXXII. It will be noted that the sludge was concentrated from an average of 4.44 per cent by weight and 21 per cent by volume to 9.75 per cent by weight and 40 per cent by volume or practically doubled. The loss in volume may be due partly to evaporation and partly to error in measuring of volumes. The apparent loss in weight is negligible and is probably due to errors in sampling.

Figure 27 shows sludge drainability on primary tank sludge and lagooned sludge. There was such a slight difference in the drainabilities of the three samples of lagooned sludge that only one is shown. The drainability of the primary sludge improved as digestion improved. A curve (No. 1) for a sludge before digestion was normal (third experiment) and one (curve No. 2) for a sludge drawn near the end Of the third experiment illustrate this point.

The fact that the drainability of lagooned sludge (curve No. 3) lay between the drainabilities of the primary sludge samples is explained by the fact that the lagooned sludge contained about twice as much solids as the primary sludge.

Conclusions. Owing to difficulties encountered in short experiments no attempt was made to summarize and balance the data on solids, grease, etc. The following conclusions concerning the experiments may be drawn:

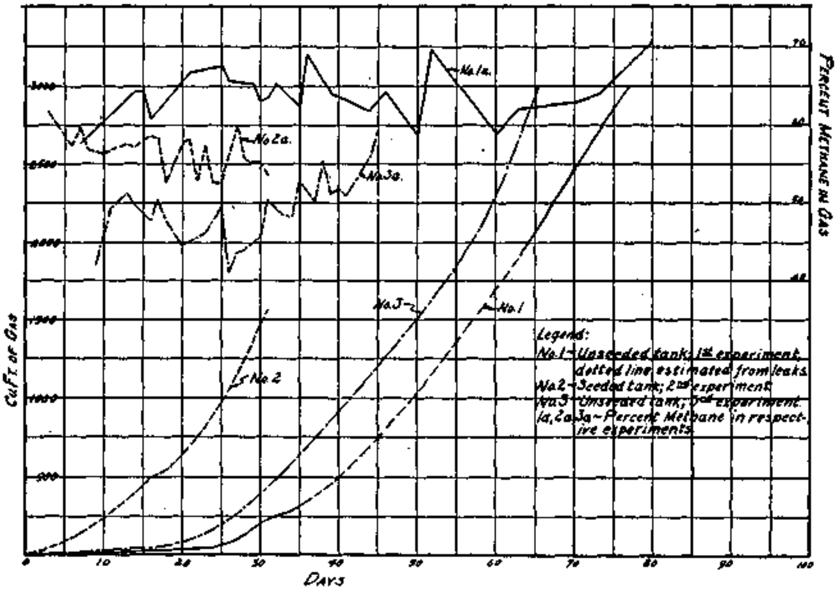


FIG. 26.—GAS PRODUCED FROM UNSEEDED AND SEEDED TANKS.

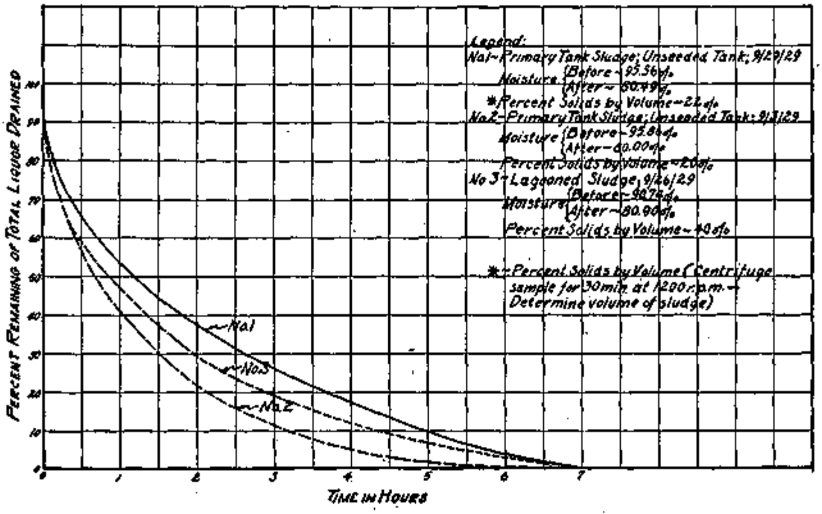


FIG. 27.—SLUDGE DRAINABILITY—LAGOON AND PRIMARY TANK SLUDGE.

Digestion proceeded more rapidly in a tank seeded with liquor than in an unseeded tank. Gasification did not start in unseeded tanks for 25 days, and normal digestion (maximum gas rate) was not reached for 15 days after gasification started (temperature of digestion 25°C).

Gasification in a liquor seeded tank started immediately and normal digestion was reached in 18 days. Primary tank sludge of 4.4 per cent solids was removed weekly to a lagoon where it was concentrated to 9.7 per cent solids. Sludge from the lagoon containing more than twice as much solids as primary tank sludge, had the same draining characteristics as the latter. Though the sludge from the primary tank had been digesting for a period of only a week, when drawn it was a fairly well digested sludge. It did not have an exceptionally obnoxious odor, and this odor apparently disappeared within a day or two when the sludge was placed in the lagoon tank. Sludge from the lagoon tank when drawn had all the appearances of a well digested sludge. Its odor, texture, draining qualities and appearance were comparable to secondary digestion tank sludge.

TABLE XXXII

DATA ON SLUDGE LAGOONING

	Sludge from primary tank to lagoon		Liquor siphoned from lagoon		Sludge from lagoon	
	Gallons	Per cent solids	Gallons	Per cent solids	Gallons	Per cent Solids
7/22/29	372	4.71				
7/29/29	145	4.44	50	1.02		
7/31/29–8/ 8/29			286	.340		
8/ 5/29	97	4.95				
8/ 8/29					270	9.47
8/12/29	482	5.73				
8/14/29 and 8/15/29			299	.286		
8/19/29	149	3.75				
8/22/29					272	10.40
8/26/29	183	3.32				
9/ 3/29	145	4.14				
9/ 9/29	142	3.49				
9/16/29	155	3.66				
8/26/29–9/21/29			391	.264		
9/26/29					208	9.26
Total gallons	1,870		1,036		750	
Average solids		4.44		.32		9.75
Per cent solids by volume		21.0				40.0
Pounds solids		705		28		610

Loss in volume, 84 gallons.
Loss in weight, 67 pounds.

PART V

NEW METHOD FOR DETERMINATION OF SETTLING SOLIDS

By A. M. BUSWELL, A. L. ELDER, C. V. ERICKSON, AND G. E. SYMONS

Our attention was called to this question by some apparently inconsistent results on the effect of different sedimentation periods on the removal of solids in Imhoff tanks, the analytical control for which we are responsible. The amount of solids removed as shown by the Gooch solids determination did not appear to check the solids removed, as indicated by the residue on evaporation determination, nor did they accord with any consistency with the results which might be expected from the changes in the detention periods.

Wagenhals, Theriault and Hommon[50] had previously called attention to difficulties in the determination of the suspended solids with the use of the Gooch crucible. They discussed the question as follows:

"The main source of error is due to the difficulty of securing a representative sample since, without a disproportionate expenditure of time, it is seldom possible to filter more than 50 to 100 cc. of sewage. With a raw sewage the tendency is for the results to be too low, due to the necessary exclusion of the larger particles such as fruit skins, and other substances of similar nature which are best described as settleable solids. If these coarser particles are first removed and the balance of the sewage is well disintegrated, as by vigorous shaking, the accuracy of the test is considerably increased. This is well illustrated by the following results obtained at Eochester in a study of Eiensch-Wurl screens:

TABLE NO. 13

VARIATIONS IN SUSPENDED MATTER DETERMINATIONS.

(Results in parts per million)

Date	Raw sewage				Riensch-Wurl screen effluent			
	Duplicates		Average	Per cent deviation	Duplicates		Average	Per cent deviation
	A	B			C	D		
1920								
July 24	460	296	378	21.8	312	308	310	0.6
26	250	264	257	2.7	286	280	283	1.1
27	204	190	197	3.5	202	206	204	1.0
28	138	128	133	3.8	132	136	134	1.5
29	164	122	143	14.7	144	148	146	1.4
Averages	243	200	222	±9.3	215	216	215	±1.1

"All tests recorded in the above table were made on samples composited over 24-hour periods. Before removing a portion for the test, the bottles were shaken vigorously and every precaution was taken to make the analyses comparable in every way. The results tabulated under columns A and B are duplicate determinations made on the raw sewage, and the results under columns G and D are duplicate determinations made on the Riensch-Wurl screen effluent. This effluent was in every way comparable with the raw sewage excepting that the coarser particles had been removed and the rest of the suspended matter had been well disintegrated by the action of the brushes. The result was a sample with a very homogeneous suspended matter content. This is indicated by the excellent agreement between the duplicate determinations and the low average deviation, 1.1 per cent, which in this case is a measure of the probable error involved. The corresponding raw sewage samples collected and tested under exactly the same conditions show an average deviation of 9.3 per cent from the average value obtained when duplicate tests are made.

"The series of tests presented is not sufficiently extensive to be made the basis of very definite conclusions. It nevertheless confirms a general opinion and shows an unmistakable tendency. A more extensive series of tests presented in Table No. 41 indicates that the suspended matter content increased by about 5 per cent on passing through the Riensch-Wurl screen. This obviously questionable result is readily explained, if it is considered that the results for the suspended matter content of the raw sewage may be in error by over 9 per cent and that the tendency is for the raw sewage results to be too low. The amount of suspended matter removed by the Kiensch-Wurl screen would have to be in the neighborhood of 10 or 15 per cent before any removal could be indicated with certainty by suspended matter determinations. A direct weighing of the screenings removed is, of course, the indicated method of procedure. The limitations of the suspended matter determination do not lead to serious errors of interpretation, however, when the test is applied to the effluents of the ordinary devices. Since in any complete system of sewage treatment the removal of the bulk of the suspended matter in the form of sludge is one of the main objectives, this determination is of great value and in most cases an error of 10 per cent, or even more, does not affect the validity of the conclusions drawn. The test is especially useful for the estimation of the per cent removal effected by the various types of tanks and is the best index available for judging the efficiency of tank treatment."

In making the determination of solids in sewage there are really two sampling errors. In the first place the individual portions which are taken, at say hourly intervals, are usually from 100 to 300 cc. in volume and the vessel which is used in collecting the sample is seldom over 5 inches in diameter and frequently not over an inch to an inch and a half in diameter. A sampling vessel of this sort will obviously fail to pick up the proper share of the larger objects. If a large sampling vessel is used, it is necessary to pour off the proper proportion into the smaller container and from the writers' observation, it is impossible to avoid a certain amount of sedimentation or flotation of large particles no matter how vigorously the sample in the large vessel is stirred. The tendency is, therefore, as Theriault states, to obtain too low a proportion of large particles in the raw sewage sample.

A second sampling error occurs when the portion for analysis is taken out from the composite sample. The composite 24-hour sample of sewage commonly amounts to two or three liters. From this, 100 cc. is withdrawn for the determination of total solids and 50 to 100 cc. for the determination of the suspended solids (Gooch). In withdrawing these relatively small portions the tendency will almost always be to obtain too low a proportion of larger particles. These errors will effect the sample of raw sewage almost exclusively.

The obvious way to avoid these sampling errors is to increase the size of the sample. If it were possible to determine the total amount of fresh solids which settle out in a sedimentation tank, that would appear to be the most accurate method of determining the effect of the sedimentation process. Data are available on the amount of solids removed by plain sedimentation, but we have not been able to find any extensive tests where these have been correlated with laboratory determinations of the solids in the influent and effluent.

Table XXXIII shows some results on sludge removal calculated from data in U. S. Public Health Bulletin No. 132. While the data do not show the actual pounds of sludge removed, they do show that comparable results between different determinations, or more correctly stated in these cases, between the same determination made on different samples. Assuming that the results obtained from the difference in suspended matter in influent and effluent are correct, then the results obtained from the difference between the suspended matter in the influent to the tank and the supernatant liquor in the same after settling, are about 60 per cent high.

TABLE XXXIII
DATA ON SUSPENDED MATTER AND SLUDGE REMOVAL
Calculations from U.S.P.H. Bulletin 132
(Gooch Methods)

Table No.	Suspended matter in parts per million					Pounds sludge per million gallons calculated from*	
	I Raw	II Tank effluent	III Raw supernatant	I—II	I—III	I—II	I—III
19	152	69	37	83	115	690	954
24	159	107	45	52	114	433	947
27	261	93	62	168	199	1,400	1,660
28	206	79	60	127	146	1,055	1,215
31	297	63	56	234	241	1,950	2,000
38	166	87	14	99	152	824	1,265
39	145	85	57	60	88	500	734
40	101	40	18	61	83	508	692
41	188	119	50	69	138	575	1,150
42	110	42	10	68	100	565	834
43	264	76	85	188	179	1,565	1,490

* There is a 59.8 per cent variation between the two sets of results, assuming that I-II is correct.

It was to more nearly correlate the amount of sludge removed by sedimentation with laboratory data that the new method described in the following pages was devised. Table XXXIV presents comparative data obtained in this laboratory. These data are all determined on a nidus tank. Daily analysis of the influent and effluent were made and at the same time sludge settled from the sewage treated was removed, measured, and per cent solids determined. This offered an excellent means of checking the reliability of the determinations. Most of the data were collected during the experiments on sludge digestion reported in Parts I, II, and III, so that the operating conditions of the nidus tank described in Part I apply here. Only one result is given on the Gooch method, but it confirmed previous data, experience, and data from other sources (cf. data on Baltimore and Eochester, Table XXXIV), so the method was abandoned in this laboratory, in favor of the new determination, "settling solids." This method and the calculations involved, and a discussion of the accuracy obtained follow.

Determination of Settling Solids

Procedure. A liter of the sample is placed in a glass tube 1¾ inches in diameter and about 3 feet long, set in a vertical position and fitted with a rubber stopper in the lower end. After one hour of quiescent settling, the sample is stirred to loosen any particles attached to the sides of the tube. Following another hour of settling, the supernatant liquor is siphoned down to a residual volume of about 50 cc. of liquor and settled solids. Eemoval of the stopper allows the residual liquor and solids to be transferred to an evaporating dish. After measuring and recording the volume of this liquor by means of a cylinder graduated in cc, it is placed in an evaporating dish. The measuring cylinder is then rinsed with not more than 50 cc. of distilled water and this wash water is used to rinse out the settling tube and then added to the liquor in the evaporating dish, in order to avoid losing solids which adhere to the measuring cylinder. The liquor is evaporated on a steam bath and the residue is weighed. The weight multiplied by 1,000 gives this residue in parts per million. A correction must be applied to this result, because of the dissolved and non-settling solids contained in the liquid portion of sample. This correction is worked out as follows.

Let x = weight of settling solids in 1 liter of original sample.
Let y = weight of non-settling solids + dissolved solids in 1 liter of original sample.
Let R = weight of total residue in 1 liter of original sample. (Note: This is equal to 10 times the weight of the residue on evaporation as determined on a 100 cc. sample, as described in correction for dissolved solids.)
Let. w = weight of residue in residual volume of solids and liquor determined as described in the procedure, on a 1 liter sample.
Let v = volume in cc. of residual volume of solids and liquor, remaining after siphoning.

Then: $$w = x + \frac{v}{1000}(y)$$

But: $$y = R - x$$

Hence: $$w = x + \frac{v}{1000}(R - x)$$

$$w = x + \frac{v}{1000}(R) - \frac{v}{1000}(x)$$

$$x\left(1 - \frac{v}{1000}\right) = w - \frac{v}{1000}(R)$$

$$x = \frac{w - \dfrac{vR}{1000}}{1 - \dfrac{v}{1000}}$$

If $v = 50$ cc. formula reduces to $$x = \frac{w - 0.06R}{0.95}$$

Instead of substituting for "w" and "R" the actual weights, the results in parts per million may be used.

Accuracy of Results.

(1) The vertical sides on the settling tube and stirring gently once or twice during the 2-hour period eliminate adhesion of settling solids to the wall.

(2) Siphoning down to a residual volume of about 50 cc. can be done with sufficient care to avoid disturbing the settled solids.

(3) At times, a small amount of solids float on the surface, but on stirring these solids gently, a large portion of them settle to the bottom.

(4) Duplicate determinations on various samples have shown that checks to one per cent can be obtained no matter what residual volume of liquor containing the settling solids is used so long as this volume

is known and care is taken not to disturb the solids in the bottom of the tube during siphoning.

(5) The test as described eliminates any error due to volatile compounds and gives a true estimate of the amount of settling solids, when used in sludge digestion experiments.

Over the entire period studied, the sludge removed, as determined by residue on evaporation, is 23.7 per cent high, while sludge removed as calculated from the settling solids determination is 6.5 per cent low.

The fact that the residue on evaporation gives high results is not in accord with expectations nor previous data. An examination of the daily data and operating conditions gives an explanation of this apparent anomaly. Table XXXV shows the daily results of one of the periods studied. It will be noted that there are a great number of days when according to the total residue determination, there was an exceptional removal of solids and other days there was a negative removal. These negative removals usually followed the days of high removal. This lead to an examination of the daily analyses. It was found that on the days of high removal there was also an apparent high removal of chlorides, and vice versa.

Since the city water in Champaign-Urbana is quite hard, there are many zeolite water softeners in the twin cities, and when these are charged there would be an introduction of a great amount of brine into the sewage. The fact that our operating condition did not permit 24-hour sampling made it necessary to proportion the 10:00 p. m. and 6:00 a. m. samples to approximate the amount of sample that would have been collected during the interval. The practice was based on the assumption that the concentration changed little during that time. The practice no doubt led to the anomalous results obtained on chloride removal.

TABLE XXXIV
ESTIMATION OF SLUDGE SOLIDS
(Average Daily Pounds per Million Gallons)

Date	Gooch solids[1]	Total residue[2]	Settling solids[3]	Actual[4]
7/ 9/26–10/ 5/26	250	380*		734*
9/21/27–11/ 6/27		526	586	809
1/13/28– 2/18/28		272	207	356
2/19/28– 3/17/28		624	213	275
3/18/28– 4/25/28		624	518	422
4/28/28– 5/19/28		647	308	337
5/20/28– 6/15/28		343	395	499
6/15/28– 7/30/28		1,314	722	571
7/31/28– 8/31/28		1,083	956	602
Total		5,811	3,905	4,705
Error (per cent)[5]	66 low	23.7 high	6.5 low	
Hydrolytic tanks, Baltimore[6]	432			847
Error (per cent)[5]	49 low			
Secondary tanks, Rochester (Brighton)[6]	158			208
Error (per cent)[5]	24 low			

[1] Gooch solids in influent — gooch solids in effluent.
[2] Total residue in influent — total residue in effluent.
[3] Settling solids in influent — settling solids in effluent.
[4] Gallons sludge from settling tank × 8.34 × per cent solids.
[5] Error based on sludge removed.
[6] U. S. P. H. Bulletin 132.
*Not included in total.

A check of the data, calculating the chlorides to sodium chloride and correcting for the same gives results which are much more comparable and do not vary widely.

It will be noted in Table XXXV that there are no negative results obtained from the settling solids determination. This was with 3 exceptions true throughout the entire study. On the other hand there were 35 days in 13 months when this determination showed excessive removal. These, and the negative results occurred on days when similar results were obtained from the total residue determination. This would indicate that though the settling solids determination is corrected for dissolved solids, there are times when the correction is not quantitative. It might be expected that the determination would be subject to such an error, since the correction is based on the total residue determination. While this determination gave anomalous results only 38 days out of 13 months, the total residue determination gave erroneous results more than half of the time.

An inspection of Table XXXV shows that the pounds of sludge per million gallons as calculated from the settling solids determinations on influent and effluent from settling tanks are more consistent and more nearly approach the actual sludge removed than when calculated from the determinations of the residue on evaporation. That this statement is true is borne out by an inspection of the remainder of the data collected.

TABLE XXXV
ESTIMATION OF SLUDGE SOLIDS
(Pounds per Million Gallons)

Date	Actual[1]	Total residue[2]	Settling solids[3]
May 20, 1928	232		
May 21, 1928	472	1,319	475
May 22, 1928	644	—366	367
May 23, 1928	464	41	125
May 24, 1928	433	—175	409
May 25, 1928	456	1,270	67
May 26, 1928	342	1,190	200
May 27, 1928	175		
May 28, 1928	533	—710	308
May 29, 1928	597	368	566
May 30, 1928	557		
May 31, 1928	582	1,420	509
June 1, 1928	624	0	609
June 2, 1928	588	—434	634
June 3, 1928	507		
June 4, 1928	592	—326	234
June 5, 1928	600	25	409
June 6, 1928	565	117	592
June 7, 1928	502	617	542
June 8, 1928	527	717	509
June 9, 1928	453	900	334
June 10, 1928	348		
June 11, 1928	464	1,070	284
June 12, 1928	483	269	342
June 13, 1928	654	300	443
June 14, 1928	586	660	418
June 15, 1928	496	—17	284
Average 5/20/28 to 6/15/28	499	343	395

[1] Gallons sludge from settling tank × 8.34 × percent solids.,
[2] Total residue in influent — total residue in effluent.
[3] Settling solids in influent — settling solids in effluent.

The above statement presents a strong argument in favor of the method. It is apparent from Table XXXIV that this method has a great advantage over the Gooch method (which may be from 50 to 75 per cent in error) for determining suspended matter. The method also offers a convenient means of determining the amount of liquefaction during sludge digestion.

Additional data collected when the sampling procedure has been improved here, or at other plants where facilities for actually measuring the sludge removed are available, will do much toward furthering information on the applicability of the determination. Babbit and Schlenz[1] have used this determination in studying detention periods and settling efficiency in sedimentation tanks.

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