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EVALUATION OF THE EFFECTS OF LOW TEMPERATURE ON THE ANAEROBIC DIGESTION OF ANIMAL WASTES

BY

ANTHONY THOMAS VOELL

A thesis submitted
in partial fulfillment of the requirements for the
degree Master of Science, Major in Civil
Engineering, South Dakota State
University

1966

## EVALUATION OF THE EFFECTS OF LOW TEMPERATURE ON THE ANAEROBIC DIGESTION OF ANIMAL WASTES

This thesis is approved as a creditable and independent investigation by a candidate for the degree, Master of Science, and is acceptable as meeting the thesis requirements for this degree, but without implying that the conclusions reached by the candidate are necessarily the conclusions of the major department.

Thesis Adviser Date

Head, Civil Engineering Date

Department

2661 8

#### ACKNOWLEDGMENTS

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The patience and understanding of my wife, Paula, were an unending source of encouragement.

Above all, thanks be to God, who is the source of all knowledge.

ATV

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#### INTRODUCTION

The history of anaerobic sludge digestion has shown an ever increasing body of knowledge concerning the causes and operation of anaerobic treatment of organic wastes. Two of the earliest methods used in the anaerobic treatment of organic wastes were cesspools and septic tanks. The theory of the design of these units was based upon retaining the waste material and allowing it to undergo bacterial decomposition. An improvement in the anaerobic treatment process was made with the advent of the Imhoff tank. In this unit the sedimentation and digestion portions were separated so that the sludge digestion unit was not in contact with the incoming sewage. Imhoff tanks followed the design criteria of providing enough volume to hold the sludge until it was decomposed or until it was discharged.

The above processes were operated at the temperature established by the incoming waste material and the environmental conditions. It was observed that the waste was broken down faster in the summer than in the winter. Based on this knowledge many studies were undertaken to determine the optimum temperatures for the degradation of organic material under anaerobic conditions. The vast majority of these investigations were in the mesophilic and thermophilic temperature ranges. This is understandable because it is at these higher temperatures that the faster reduction of organic waste is accomplished. Some study is needed, however, to determine the rate of solids reduction in the cryophilic (below 10°C) and temperate (10°-28°C) zones.

The animal waste lagoons in northern climates utilize anaerobic processes in the stabilization of their organic waste material. The design criteria for these units gives little consideration to the solids reduction occurring in the bottom sludge layer of the lagoon. The seasonal and daily variation in air temperature made the problem of evaluating solids reduction even more acute. The variation in temperature of the lower sludge layers of an animal waste lagoon does not have the extreme variation of the air temperature.

#### OBJECTIVE OF THE RESEARCH . Activated adades and decide and decide

The objective of this laboratory investigation was to study the anaerobic reduction of organic fecal material at temperatures approximating those found in the sludge layers of animal waste lagoons in northern climates. These ambient temperatures are generally less than 25°C and in most cases are those temperatures that prevail for the ground water at the lagoon location.

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#### REVIEW OF LITERATURE

#### INTRODUCTION

The treatment of organic waste material is based on the principle that microorganisms will continuously stabilize organic matter from liquid wastes. These microorganisms are classified as anaerobic or aerobic according to the type of oxygen they utilize for metabolism.

The most common methods of treating organic wastes aerobically are with trickling filters, activated sludge units and oxidation ponds. The aerobic microorganisms in these processes require free, uncombined oxygen to obtain their energy supply. The supplying of this oxygen is a major economic factor in the use of aerobic processes.

Anaerobic digestion of sewage sludge does not require free oxygen. The organisms responsible for this metabolism of organic material are obligate anaerobes and facultative anaerobes. Some of the reasons for anaerobic treatment of sludge are as follows:(1)

- 1. Organic matter is stabilized so that it will not exert an oxygen demand.
- 2. Sludge is conditioned so that it will give up water more readily than undigested sludge.
- 3. There is a reduction in the volume of sludge for ultimate disposal.

#### THEORY OF ANAEROBIC DIGESTION

All organic matter, living and dead is composed of carbon (C), hydrogen (H) and relatively small amounts of a few other elements

which can be represented by X. The symbol for organic matter then becomes C-H-X. Buswell (2) summarized the anaerobic process of decomposition of organic matter by the following relationship:

This equation represents the ideal or complete removal of carbon, partly as the completely oxidized carbon dioxide  $(CO_2)$  and partly as methane  $(CH_k)$  (2).

Sewage sludge contains the necessary constituents for its own decomposition (3). First, organic matter or food is present; this includes carbohydrates, proteins, lipids, fatty or organic acids and many others. Second, other necessary nutrients such as nitrogen, phosphorus, minerals, vitamins, etc. are present. Third, a variety of organisms is present. While many of these organisms have not been identified and their properties are not entirely known, they include many different species of bacteria, protozoa, yeasts and molds, as well as some of the higher plant and animal forms (3).

The largest part of the digestion of sewage sludge is attributed to the bacteria. The two groups of bacteria generally agreed to be responsible for the liquefaction and gasification processes are the saprophytic organisms and the methane formers, respectively (4). The first group, normally present in sewage in great numbers and capable of rapid rates of reproduction, attacks the complex organic substances, such as fats, carbohydrates, and proteins and converts them to simple organic compounds. These saprophytes consist of many acid forming

bacteria which produce low molecular-weight fatty acids during degradation of the organic material. The second group, the methane formers, are capable of utilizing the acids and other end products formed by the first group. These methane formers do not occur in as great a number in raw sewage nor does their slower reproduction rate compare with that of many saprophytic organisms. Consequently, organic acids may be formed faster than the limited population of methane formers can assimilate them and, as a result, the accumulated acids reduce the pH to an unfavorable level for the methane formers (4).

The early stages of the decomposition of organic matter brought about by the acid forming organisms is quite similar to those which occur in the early stages of human metabolism (3). For these earlier stages the name digestion seems appropriate. However, more than just digestion occurs in a sludge digester. Human digestion ends with the breakdown of complex foods, such as starches, proteins and fats, into simpler compounds which are either soluble or small enough to pass through the walls of the stomach and the intestines into the blood stream for further usage. The subsequent breakdown and conversion of these absorbed substances to yield even simpler compounds and energy is commonly called the "breaking-down process" or technically, catabolism. Some of the simple substances so produced recombine to form the complex chemicals of cell tissue. This process is called the "building-up process" or anabolism. Actually these processes occur simultaneously and the overall process is called metabolism. In the decomposition of

sewage sludge under anaerobic conditions both digestion and metabolism are occurring simultaneously (3).

The anaerobic metabolism processes as applied to the anaerobic digestion of sewage sludge is depicted in Figure 1. The acid forming bacteria attack the carbohydrates, fats and proteins and hydrolyze and liquefy them. The carbohydrates, which are usually polysaccharides, are split into simple sugars, then to alcohols and finally into volatile organic acids to be utilized by the methane bacteria. The fats are converted to alcohols and on to volatile acids. The proteins are reduced to sub proteins and further to amino acids. The amino acids follow one of two paths. They are either further metabolized to volatile acids or mercaptans, indoles, skatoles, H<sub>2</sub>S and NH<sub>3</sub>. These latter compounds are primarily responsible for the obnoxious odors present in anaerobic digestion.

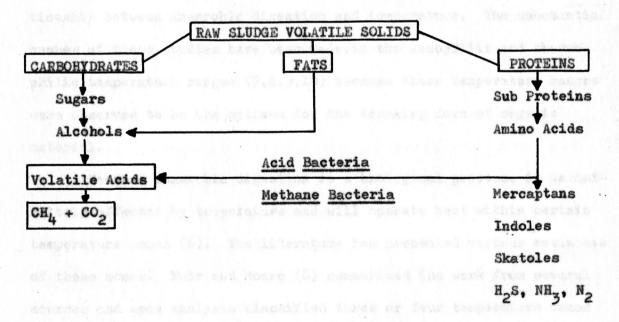


Figure 1. Simplified Anaerobic Metabolism System (5).

#### FACTORS INFLUENCING ANAEROBIC SLUDGE DIGESTION

No single species of organisms can liquefy and gasify organic matter; and for the successful maintenance of a highly mixed microbial population in digestion operation, certain conditions must be at an optimum. These conditions are determined by observations and laboratory tests such as temperature, pH, alkalinity, volatile acids, gas production, and solids determination (6).

Study and application over a number of years have demonstrated that certain factors may greatly accelerate or retard digestion and are, therefore, indicative of digestion progress. The following paragraphs review these factors as a basis for laboratory tests utilized to judge the progress of sludge digestion.

#### Temperature

Numerous studies have been undertaken to investigate the relationship between anaerobic digestion and temperature. The substantial number of these studies have been made in the mesophilic and thermophilic temperature ranges (7,8,9,10) because these temperature ranges were observed to be the optimum for the breaking down of organic material.

Because anaerobic digestion is a biological process, it is definitely affected by temperature and will operate best within certain temperature zones (6). The literature has presented various estimates of these zones. Fair and Moore (8) summarized the work from several sources and upon analysis classified three or four temperature zones of activity for sludge digestion. These zones were identified as the thermophilic (above 42°C), the intermediate or mesophilic (28-42°C), the temperate (below 28°C and possibly above 10°C) and the cryophilic (below 10°C).

Very little information was uncovered that pertained to the cryophilic temperature zone and its relationship to the microbial activity in anaerobic digestion. Bloodgood (11) states that it has been found by practical experience that digestion does not take place at temperatures of around 39°C. Babbitt and Baumann (12-584) place the temperature at which bacterial action practically ceases at 50°F. Whitehead and 0'Shaughnessy (13) report that digestion is very sluggish below 8°C, fairly vigorous at 15°C and that an optimum is reached between 25-28°C. On the other hand, it has been reported (6) that Buswell did not observe a specific temperature limitation for anaerobic treatment and that he found that biological formation of methane proceeds at temperatures as low as 0°C.

Most of the experience gained in low temperature anaerobic digestion has been with Imhoff tanks. Daniels (14) reported that the average temperature in the Imhoff tanks at Trenton, New Jersey was around 60°F and ranged from 55°F to 75°F. Hatfield (15) stated that the variations in gas production, in the Imhoff tanks at Decatur, Illinois, above and below the average followed the temperature variations more closely than any other factor. The temperature of the

sewage at the Decatur plant ranged from 90°F to 105°F in the summer and from 60°F to 70°F in the winter.

Imhoff and Fair (16-187) present a chart as shown in Figure 2 relating time required for 90% digestion of sewage sludge to temperature. They show a geometric decrease in time required for digestion as the temperature increases. The mesophilic optimum is reached at about 100°F. Another optimum is reached in the thermophilic range about 128°F. The chart shows that some activity would be present at 50°F (10°C) and that it should take approximately 80 days for 90% digestion of sewage sludge at this temperature.

#### Volatile Acids and Alkalinity

During the liquefaction and hydrolyses processes volatile organic acids are produced. In a digester these acids vary greatly in concentration and individual acid composition (17). Sawyer (18-338) states that the kinds and amounts of volatile acids present are a function of the nature of the substrate undergoing decomposition. These volatile acids are the end products of hydrolyzing the fats, proteins and carbohydrates which make up the organic matter in sludges (17). Pohland and Bloodgood (19) represented these biochemical processes schematically as follows:

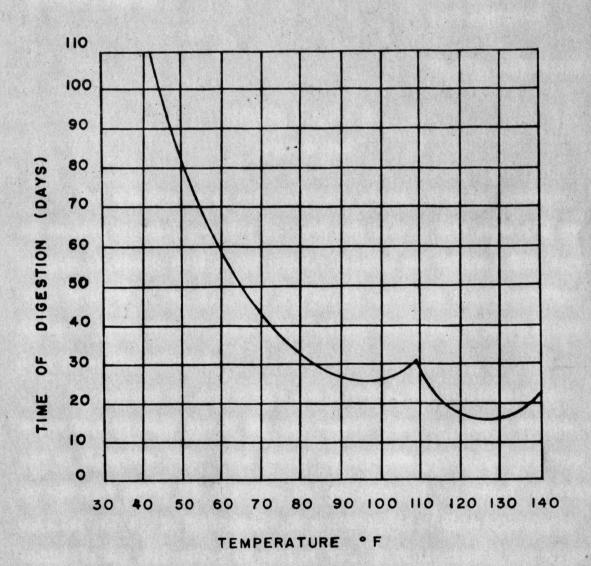
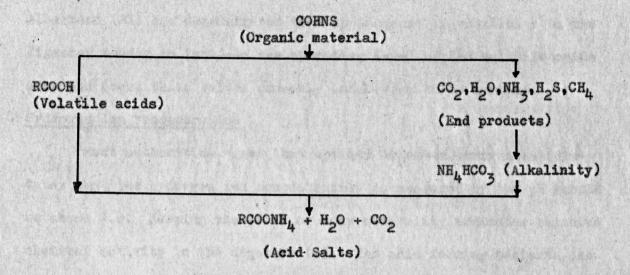


FIGURE 2. TIME REQUIRED FOR 90% DIGESTION OF SEWAGE SLUDGE IN CONVENTIONAL TANKS (16).



The volatile acids and alkalinity derived from the breakdown of organic material are free to react and form an acid salt with the release of carbon dioxide and water. McCarty and McKinney (20) report that toxicity to the digestion process is caused by high concentrations of these volatile acid salts and not the volatile acids as has been generally believed.

Regardless of what causes the toxicity, the volatile organic acids test is still one of the most useful tools in gauging the process of digestion (17). Once stable equilibrium conditions have been established within the digester, any variation of the normal volatile acid content can be interpreted as a disruption of this equilibrium necessitating some method of control (6). Both alkalinity and volatile acids have been proposed as indices of digestion operation at one time or another. The value of alkalinity to the digestion process is generally believed to be embodied in its buffering ability (6).

Albertson (21) had demonstrated that an increase in alkalinity in the digester tended to increase the operating level of the volatile acids over and above those values commonly considered to be maximum.

Hydrogen Ion Concentration

Most authorities agree that optimum digester environment dictates that the hydrogen ion concentration as measured by the pH should be about 7.0. Keeping the pH at or near neutrality indicates balanced chemical activity in the digester (6). The acid forming bacteria can tolerate a lower pH than the methane formers. Acid producing bacteria, however, can thrive at pH levels favorable to methane formers. The pH requirements of the environment are controlled by the needs of the methane bacteria. The methane formers are most active in the pH range from 6.4-7.2; at a pH value below 6.0 and above 8.0 the growth rate falls off rapidly. Observations of the effect of pH variations in sludge digestion on the rate of gas production agrees with the conclusions of laboratory culture studies (22). Laboratory investigations have indicated that as temperature of digestion increases, the pH of the medium will increase (9,10).

Smith (5) depicts the progress of anaerobic digestion of a batch of sludge showing three main phases. (See Figure 3).

23). A CO2 content above this value is indicative of forthocoming and

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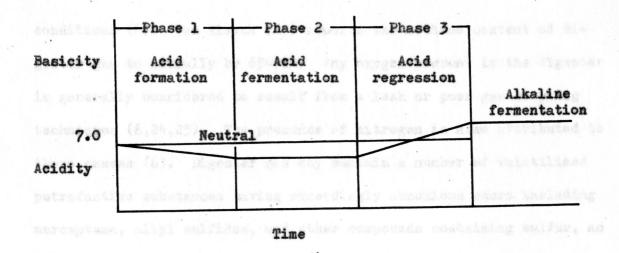


Figure 3. Batch Progress of Anaerobic Digestion (5).

#### Smith (5) explains these phases as follows:

Phase 1--Intensive volatile acid formation, little or no acid destruction by methane bacteria; pH decreases; no methane production; gas, if any, high in CO<sub>2</sub>.

Phase 2-Methane bacteria become established and volatile acids production is balanced by acid metabolism by methane bacteria; stable gas production composed of about 70% methane and 30% CO<sub>2</sub> is established; pH is constant.

Phase 3--Nutrient becomes depleted and volatile acids are formed at a slower rate than they are metabolized by methane bacteria; volatile acid concentration decreases; pH increases; gas production falls off as volatile acid level decreases.

#### Gas Production and Quality

The quality and quantity of gas produced during digestion is generally considered indicative of the progress of digestion and variations from the normal foretell digestion difficulties (6).

The consensus of opinion among researchers is that a CO<sub>2</sub> content of 35-40% or less is necessary for a properly operating digester (6, 23). A CO<sub>2</sub> content above this value is indicative of forthcoming acid

conditions (6). Van Kleeck (23) reports the methane content of digester gas to normally be 65-70%. Any oxygen present in the digester
is generally considered to result from a leak or poor gas sampling
techniques (6,24,25). The presence of nitrogen is also attributed to
these causes (6). Digester gas may contain a number of volatilized
putrefactive substances having exceedingly obnoxious odors including
mercaptans, allyl sulfides, and other compounds containing sulfur, as
well as organic and inorganic products such as indole, skatole, and
phosphine (6,25).

Bloodgood (11) averaged the gas characteristics from five separate plants and reported 12.1 cu. ft. of gas produced per pound of volatile matter destroyed. Van Kleeck (23) reports a value of 18 cu. ft. of gas produced per pound of volatile solids destroyed.

#### SUMMARY

This literature search has shown that the parameters used for measuring progress of anaerobic digestion are well established. Therefore, the parameters of volatile acids, alkalinity, hydrogen ion concentration and gas production and quality were used in this investigation to evaluate the effects of temperature on the anaerobic digestion of animal waste.

Studies relating the rate of solids destruction to temperature in the mesophilic and thermophilic ranges were abundant. These investigations were helpful in developing the digestion equipment and

experimental procedures. However, a lack of information pertaining to anaerobic digestion in low temperature environments was noted.

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#### EQUIPMENT AND PROCEDURES

#### DIGESTION EQUIPMENT

The anaerobic digestion of animal wastes was carried out in the laboratory in six 20 liter pyrex carboys. Hog lagoon sludge was selected as the waste material for this study. The digesters were equipped as shown in Figure 4.

The gas produced in the digestion process was collected over an aqueous-salt solution. This was necessary because serious errors would otherwise be introduced into the analysis by the loss of certain constituents through solubility, if the confining liquid were pure water (27-11). The confining fluid was of the following composition:

20% Na SOL by weight

5% HoSOh by volume

Methyl orange was added to the confining fluid to impart a color and thus facilitate taking gasometer readings.

Three groups of duplicate digesters were incubated in separate temperature controlled environments of 5°C, 15°C and 25°C. The 5°C and 15°C digesters were placed in incubators which controlled the temperature variations to within ± 1°C of the desired temperature. The digesters at 25°C were placed in an insulated, constant temperature, curing room, in which, an electric hot plate, with a thermostat connected into its power line, was installed to maintain the desired temperature. No cooling system was utilized in the curing room because

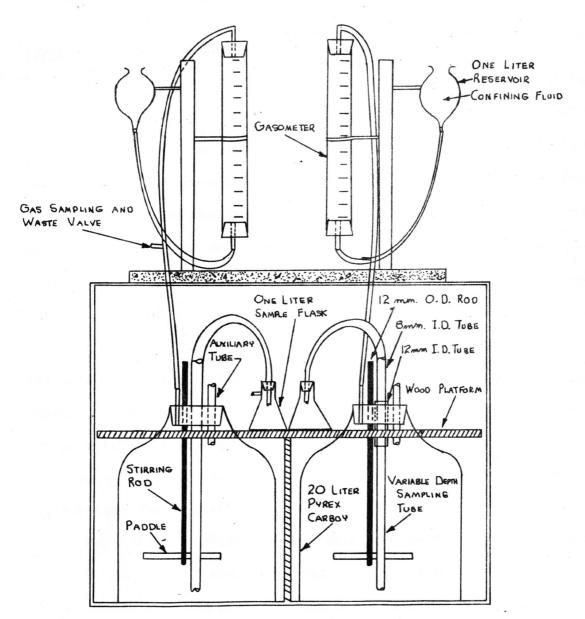


FIGURE 4. EQUIPMENT USED FOR ANAEROBIC DIGESTION STUDIES.

the ambient temperatures were almost exclusively below 25°C. The digestion installations, along with their gas collecting mechanisms, are shown in Figures 5, 6 and 7.

#### EXPERIMENTAL PROCEDURES

Animal wastes for the anaerobic digestion studies were obtained from the anaerobic hog lagoon of Mr. Donald Thaden located at Bancroft, South Dakota. An Ekman dredge sampler was used to secure the sludge sample. Thirty gallons of sludge were obtained on October 31, 1964, and after being transported to South Dakota State University remained in a frozen condition until March of 1965. At this time a model digester was built to study testing procedures, gas collection apparatus, and solids sampling techniques.

While experimenting with the model digester, it was found that the solid particles in the digestion media were too large to fit through the sampling tube and were clogging this tube. The problem was alleviated by passing all the remaining 25 gallons of waste material through a number four mesh Tyler sieve. This size of sieve approximated the opening of the sampling tube.

Preliminary studies were conducted during the months of March,
April and May. Experimentation with digesters showed that the accuracy
of the sludge sampling procedure was not sufficient to obtain reproducible solids results without mixing. The original sampling technique
was to take sludge sample from the different sludge layers in the
unit. Mixing was introduced to insure withdrawing a representative

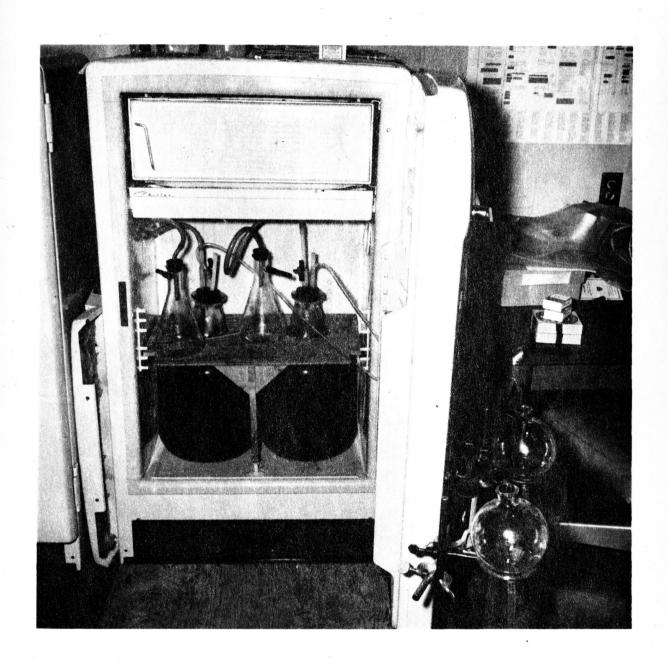


FIGURE 5. DIGESTION EQUIPMENT AT 5°C. NOTICE SMALL GASOMETERS USED.



FIGURE 6. DIGESTION EQUIPMENT AT 15°C.



FIGURE 7. DIGESTION EQUIPMENT AT 25°C.
NOTICE LARGE GASOMETERS NEEDED FOR GAS COLLECTION.

solids sample. The digester contents were mixed with a 1" x 6" x 1/4" plexiglass paddle placed on the end of a nalgene rod. The paddle was connected to the rod so that it could swivel vertically and be removed easily from the digester. Power to turn the mixing paddle was supplied by a 1/2" electric drill. This large size drill was used to obtain a slow speed for mixing. Each digester was mixed for 30 to 45 seconds before sampling. The method of withdrawing the sample remained the same.

The digesters were fed twice during the period of study (June 11 to August 8). This was done to replace the volume of material taken out for sampling and also to supply additional nutrients to the microorganisms. Each battery of tests necessitated that approximately 300 ml. of sludge be removed from every digester. When the total amount of material removed from a digester reached 900 to 1200 ml., additional sludge was added. The sludge additions were allowed to attain the temperature of the environment by storing them in the temperature controlled incubators for a two day period prior to their addition to the digesters. The material which was fed to the digesters was stored at ambient temperature in a 25 gallon galvanized container prior to the temperature conditioning it underwent before addition to the digesters. These additions were usually made every three to four weeks. Sludge was fed to the digesters by opening the top of the digesters to the atmosphere and pouring in the sludge addition. The digesters were

immediately closed to the atmosphere, mixed, and a sample was withdrawn for analysis.

The sludge addition to the 5°C units was diluted 50% to prevent any shock loadings in these digesters. There was the possibility that the low metabolic activity at this temperature could be easily upset by a large addition of nutrients. The second sludge addition to the 25°C digesters was for the purpose of increasing the concentration of organic matter in these units. This was accomplished by removing one fourth of the contents of the units and replacing it with stored feed sludge.

every week to ten days. Sludge samples for analysis were withdrawn through a pyrex sampling tube which was inserted into the digester through another, larger size, tube which acted as a casing. Rubber O-rings were inserted between the two tubes to create a seal and prevent any escape of gas from the digester. The sampling tube was adjustable and capable of being set at any depth in the digester. This did not prove necessary because mixing was introduced to insure a uniform sample. A one liter vacuum flask was used to collect the sludge sample, and vacuum was furnished by a two way rubber laboratory bulb.

Atmospheric air was admitted to the units when sludge additions were made to the digesters or when sludge samples were withdrawn. Even though air entered the digesters in these procedures, the measurement

of gas production was not hampered because this measurement was based on quantity of gas and not quality. The frequency of gasometer observations was governed by the capacity of the individual gas collecting units. The usual interval between readings was twelve hours.

A true evaluation of the quality of the gas could only be determined after all the air had been flushed from the digester. This was accomplished by the repeated wasting of gas following each gasometer observation and the subsequent production of more gas by the microbial population. The wasting and production processes quickly diluted the air to a negligible concentration, except in the 5°C units. The rate of gas production in these units was too slow during the period of study to replace the volume of air taken in during the sampling procedure. In order to eliminate any errors in the gas analysis in the two 5°C units caused by the presence of atmospheric air, the digester volume above the sludge was purged with nitrogen gas. A method was then used to compensate for the presence of the added nitrogen in the gas analysis. An explanation of the procedure used is given in Appendix C.

On May 28, 1965 each digester was charged with 20 liters of sludge. The digestion systems remained open to the atmosphere during which time the operating temperatures of 5°C, 15°C and 25°C were reached, approximately two weeks. At this time the digesters were closed off from the atmosphere and data collection began.

#### ANALYTICAL METHODS

The laboratory procedures employed for the collection of data were dependent upon the laboratory equipment employed. The procedures devised and utilized were as follows.

#### Gas Volume

The volume of gas produced was read directly from the gasometers shown in Figures 5, 6 and 7. The air temperature at the time of observation was also recorded. The uncorrected barometric pressure for the time of observation was obtained from the office of the State Climatologist at South Dakota State University. This information was secured to facilitate conversion of the gas volume observed to standard conditions, viz. 0°C and 760 mm of mercury. The procedure used is shown in Appendix A.

#### Gas Quality

Because of the lengthy procedure for the determination of the constituents of the gas evolved, only one test for gas quality was made for each of the digesters. The test verified the presence of carbon dioxide (CO<sub>2</sub>) and methane (CH<sub>4</sub>) and also gave some indication of their variation between digesters. The composition of the gas evolved was determined with a Burrell Junior Cabinet Model 611, gas analysis apparatus. In the use of this device, a sample of gas, usually 100 ml, was drawn into the apparatus. Carbon dioxide, illuminating gases and oxygen were found by absorption while the hydrocarbons were analyzed by catalytic oxidation.

#### Alkalinity and Solids Determinations

Alkalinity (28-423), total solids and volatile solids (28-428), determinations were made in accordance with the procedure outlined in "Standard Methods for the Examination of Water and Wastewater, Eleventh Edition."

#### Volatile Acids Determination

The method of determining concentrations of volatile acids in the sludge was based on the procedures developed by Westerhold (29). A detailed description of the process is given in Appendix B.

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#### RESULTS OF LABORATORY STUDIES

#### INTRODUCTION

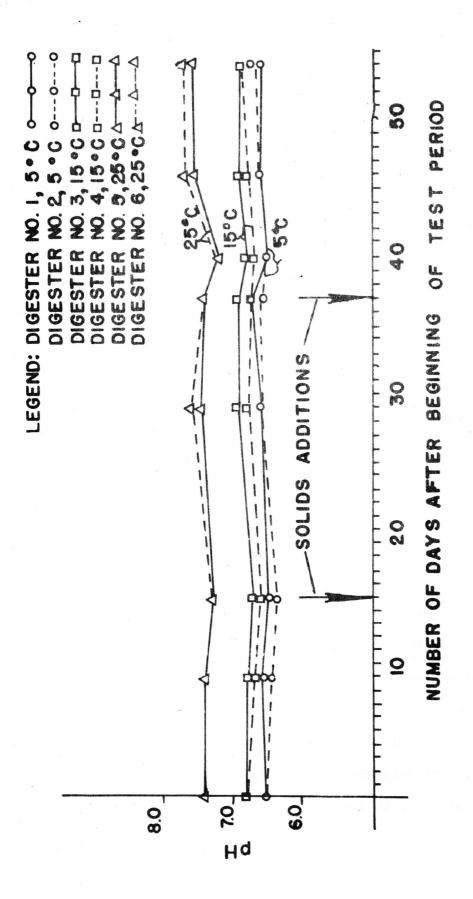
The performance of the digesters was evaluated by the observation of gas production and the established tests for digester performance. These tests included pH, volatile acids, volatile solids, alkalinity and gas quality. The following paragraphs present the information collected during the period of study.

#### HYDROGEN ION CONCENTRATION

The pH as used in this investigation, was a measure of the hydrogen ion concentration in the digesting sludge. Figure 8 shows the variation of pH with temperature and time. The pH of the original waste material at the time it was obtained from the manure lagoon was 6.5. As the digestion medium was acclimated at the three temperature levels of 5°C, 15°C and 25°C, it also exhibited three initial pH levels. Higher pH levels were associated with higher temperatures from 5°C to 25°C. The pH in the 5°C digesters had a range from 6.4 to 6.7 during the testing period. The units at 15°C had a pH variation from 6.6 to 6.9 and the range in pH in the 25°C digesters was from 7.3 to 7.6.

#### GAS PRODUCTION

The gas produced in anaerobic digestion is the result of the fermentation of the products of liquefaction (30). Temperature has also shown itself to be an important factor in establishing the rate



PH VARIATION WITH TIME AND TEMPERATURE ANAEROBIC DIGESTION STUDIES œ DURING FIGURE

of gas production. Figure 9 represents the observed effect of temperature on the rate of gas production. Gas production in this figure is expressed as liters per day at standard conditions, viz; 0°C and 760 mm of mercury. The curves indicate that the lowest rate of gas production occurs at 5°C and the rate increases with an increase in temperature to 25°C. The two sets of digesters at 15°C and 25°C exhibited a relatively constant spread in their rates of gas production within each duplicate digester. However, this may be attributed to the variation in the concentration of total solids and volatile matter in these digesters. A cumulative curve of gas produced for each digester is shown in Figure 10. These curves show the same variation in rate of gas production as do the curves in Figure 9.

The rate of gas production in the 25°C digesters seen in Figure 9, showed a slight decrease following the first sludge addition. The rate then increased sharply. Following this increase the rate of gas production in these units showed a gradual decrease until the second sludge addition. This second addition to the 25°C units was intended to build up the depleted supply of nutrients. This was accomplished by removal of one quarter of the contents of digesters #5 and #6 and replacement by stored waste material. The rate of gas production in these units increased markedly following this addition, as is seen in Figure 9 and as the increase in the slope of Figure 10 illustrates.

The 15°C digesters showed little variation in gas production because of the sludge additions. The level of activity in the 5°C

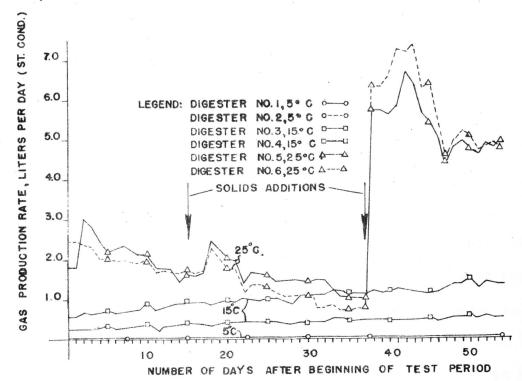


FIGURE 9. VARIATIONS IN RATE OF GAS PRODUCTION DURING ANAEROBIC DIGESTION STUDIES

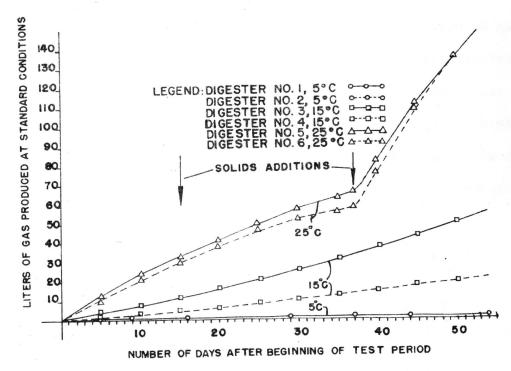


FIGURE 10. CUMULATIVE CURVES OF GAS PRODUCTION

units made it impossible to determine any variation in rate of gas production in these digesters caused by the sludge addition.

# GAS QUALITY

The gaseous end products in anaerobic digestion are the result of the final breakdown of soluble organic compounds by the methane bacteria (6). The quality of the gas produced can be used as a measure of the progress of digestion. The gas produced in anaerobic digestion of sewage sludge is reported to contain from 33 to 38 per cent carbon dioxide, from 55 to 65 per cent methane, small amounts of hydrogen, some nitrogen and traces of hydrogen sulfide (18-342).

The experimental data given in Table 1 show an increase in the per cent of methane  $(CH_{\downarrow})$  with increasing temperatures while the concentration of carbon dioxide  $(CO_{2})$  decreases. The presence of oxygen  $(O_{2})$  in the gas analysis may have been caused either by errors in gas analysis, or leaks in the sampling tubes. Hydrogen  $(H_{2})$  was observed to decrease with an increase in temperature and carbon monoxide gas (CO) was not detected in any of the digesters. Nitrogen  $(N_{2})$ , which included all the error from the gas analysis because it was obtained by subtraction, was observed to decrease with an increase in temperature.

TABLE 1.

Results of Gas Analyses of Samples Collected From Laboratory Anaerobic Digestion Systems.

Amount of gas present given as a per cent of the total.

| Digester Number                   | #1   | #2   | #3   | #4   | #5   | #6   |
|-----------------------------------|------|------|------|------|------|------|
| Temperature °C<br>Gas Constituent | 5°C  | 5°C  | 15°C | 15°C | 25°C | 25°C |
| CO2 (Carbon Dioxide)              | 43.7 | 39.7 | 29.7 | 35.8 | 25.1 | 25.1 |
| CH4 (Methane)                     | 36.0 | 36.8 | 64.0 | 54.8 | 69.6 | 71.1 |
| N <sub>2</sub> (Nitrogen)         | 17.1 | 20.7 | 5.5  | 8.1  | 4.6  | 3.3  |
| Oxygen (O2)                       | 0.3  | 0.3  | 0.5  | 0.9  | 0.5  | 0.3  |
| Illuminating Gases                | 0.6  | 0.5  | 0.1  | 0.1  | 0.2  | 0.2  |
| Hydrogen (H <sub>2</sub> )        | 2.3  | 2.0  | 0.2  | 0.3  | 0.0  | 0.0  |
| Carbon Monoxide (CO)              | 0.0  | 0.0  | 0.0  | 0.0  | 0.0  | 0.0  |

## **ALKALINITY**

Alkalinity in the form of carbonates and bicarbonates results from the recombination of the products of liquefaction and gasification. The alkalinity has a buffering effect on the sludge digestion system (30). Figure 11 shows the variation of alkalinity with time at the three temperatures of digestion studied.

The lowest concentration of alkalinity was found in the 5°C digesters. The alkalinities were observed to be higher at the 15°C and 25°C temperatures. The concentration of alkalinity in the 15°C and 25°C units decreased after the second sludge addition and then increased until the end of the period of study. This was not observed following

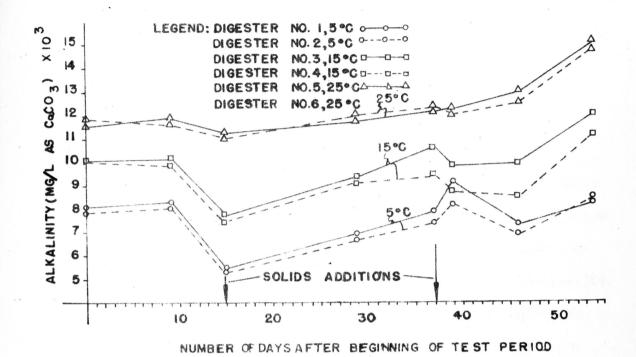


FIGURE II. ALKALINITY VARIATION WITH TIME AND
TEMPERATURE DURING ANAEROBIC DIGESTION STUDIES

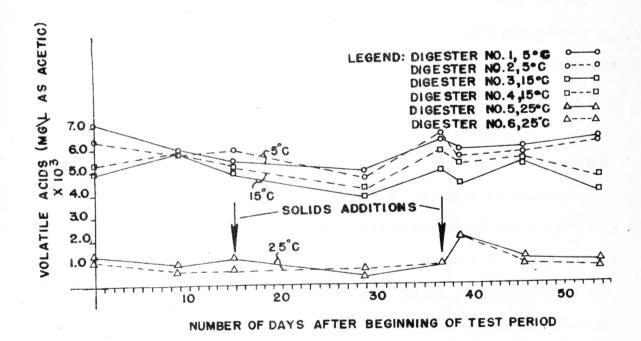


FIGURE 12. VOLATILE ACIDS VARIATION WITH TIME AND TEMPERATURE DURING ANAEROBIC DIGESTION STUDIES

the first sludge addition because insufficient samples were collected during this interval. The two digesters at 5°C did not show this decrease until two days after the second addition.

#### VOLATILE ACIDS

Short-chain fatty acids are a major component of the products of liquefaction in anaerobic digestion. These volatile organic acids result from the action of saprophytic organisms on the organic substrate and are subsequently used by the methane forming bacteria (30).

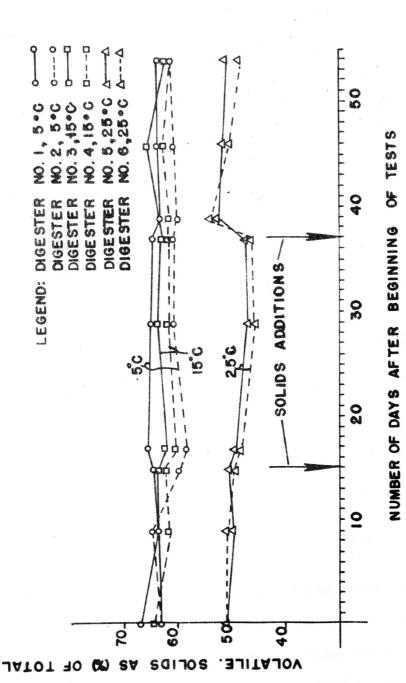
The various levels of volatile acids observed throughout the period of study are depicted in Figure 12. The variation of these concentrations of acids with temperature is also shown. The two 25°C digesters had the lowest concentration of volatile acids. In these high temperature units the acids ranged from a low value of 370 mg/l to a high of 2000 mg/l. The volatile acids in the 15°C digesters ranged from 3960 mg/l to 5850 mg/l. The highest strengths of volatile acids were found in the 5°C units. These concentrations ranged from 4700 mg/l to 7000 mg/l during the period of study.

Because of the amount of substrate added to the 25°C digesters at the second sludge addition, a substantial increase in volatile acids was observed. An increase in volatile acids was not observed following the first sludge addition primarily because of an inadequate sampling program during this period. Only slight variations were encountered in the 5°C and 15°C digesters following the sludge additions.

The general trend between sludge additions was a decrease in the concentrations of volatile acids. An increase in the strength of organic acids following the sampling made on the 29th day of the test period is shown in Figure 12.

#### VOLATILE SOLIDS

Volatile solids determinations are a measure of the amount of substrate available to the microbial population. The volatile matter present in the digestion medium when it was obtained from the animal waste lagoon was approximately 66% of the total solids. At the beginning of the test period the organic matter in the 25°C digesters was down to 51.5%. The 15°C and 5°C units still had volatile matter ranging from 65% to 66% respectively. The variation in organic material throughout the period of study is shown in Figure 13. This figure shows a gradual reduction in organic matter in the 25°C units. Very little reduction is seen in the 5°C and 15°C units throughout the period of study. A relatively large increase in organic matter was seen in the 25°C digesters after the second sludge addition.



TEMPERATURE DURING ANAEROBIC DIGESTION STUDIES FIGURE 13. VOLATILE SOLIDS VARIATION WITH TIME AND

# DISCUSSION OF RESULTS to file and the second of the

# ACCLIMATION PERIOD

Preliminary studies were undertaken during the months of March,
April and May during which time the organisms for the anaerobic digestion were acclimated to their specific temperature environment.

At the beginning of this period the six digesters at the three temperatures were filled with waste material which had characteristics as
shown in Table 2.

TABLE 2.

Comparison of Average Chemical Data Collected from Digesters
Before and After Acclimation Period.

|   | Start of Acclimation<br>Period | End o | f Acclim | ation |
|---|--------------------------------|-------|----------|-------|
| Magapletel boot there                   |                                | 5°C   | 15°C     | 25°C  |
| pH                                      | 6.7                            | 6.5   | 6.8      | 7.4   |
| Volatile Acids<br>(mg/l as Acetic)      | 4640                           | 6680  | 5190     | 1280  |
| Alkalinity (mg/l as CaCO <sub>2</sub> ) | 3250                           | 7990  | 10000    | 11670 |
| Volatile Solids                         | 66%                            | 65%   | 64%      | 51%   |

During the period of preliminary studies certain adjustments took place within the anaerobic environments. The microbial populations were establishing themselves and becoming acclimated to their respective temperature environments. This was observed by the increased rates of gas production at the end of this study period compared to the start.

The 5°C digesters had not produced gas until the final month of the preliminary studies when gas production could be detected.

From Table 2 it can be observed that the chemical environment of the organisms underwent changes during the acclimation period. A variation in pH from the original value was noted, especially in the 25°C digestion unit. This most likely resulted from the activity of the bacterial populations, which changed the environment. These pH changes can be seen to be related to the changes in volatile acids and alkalinity in all digesters during this period. The alkalinity by the end of the preliminary study period had risen from 3250 mg/l to 11670 mg/l in the 25°C digesters while the volatile acids had decreased from 4640 mg/l to 1280 mg/l. The pH increase from 6.7 to 7.4 was associated with these changes. The chemical changes in the 5°C and 15°C units were not as pronounced, however, some change was noted. These changes in chemical environment were associated with only slight decreases in volatile solids concentration compared to the 25°C digesters.

The chemical changes which took place during the acclimation period resulted in different environments at the beginning of the period of study. This is illustrated by the different levels of the chemical data for the three temperature environments as shown in Figures 8, 11 and 12.

### INVESTIGATION PERIOD

During the period of study the three temperature environments were observed utilizing the parameters already mentioned. These environments were evaluated as to the success or failure of maintaining a favorable digestion equilibrium.

For optimum operation, the pH of an anaerobic digestion system should be about 7.0 (6). The 5°C digestion units never attained this pH. The low temperature environment in the 5°C units probably retarded the activity of the methane bacteria, thereby allowing an accumulation of volatile acids which kept the pH at the levels shown in Table 3.

TABLE 3.

Ranges of Chemical Data Collected for Two Digesters at Each
Temperature of 5°C, 15°C and 25°C During
the Period of Investigation.

|   | 5°C          | 15°C         | 25°C          |
|---|--------------|--------------|---------------|
| pH Range                                      | 6.4 - 6.7    | 6.6 - 6.9    | 7.2 - 7.7     |
| Alkalinity Range (mg/l as CaCO <sub>3</sub> ) | 5290 - 9050  | 7560 - 11950 | 11090 - 15050 |
| Volatile Acid Range<br>(mg/l as Acetic Acid)  | 4700 - 7010  | 3960 - 5850  | 370 - 2000    |
| Range of Volatile Acid<br>to Alkalinity Ratio | 0.694 - 1.13 | 0.33 - 0.69  | 0.031 - 0.16  |

These levels were unfavorable for the establishment of a balance of the activities of acid forming organisms and methane forming organisms during the acclimation period and the period of study. The methane bacteria will utilize the products formed by the acid formers as fast as they are formed only if a sufficient population of organisms has

been developed and the conditions of the environment are favorable to the methane formers (18-337).

A larger population of methane organisms was apparently established in the 15°C and 25°C digesters during the acclimation period as evidenced by the signs of proper biological balance in these digesters. The increased activity of the methane bacteria in the higher temperature environments, especially at 25°C, enabled them to convert the organic acid products of liquefaction as fast as they were produced and thereby maintain a proper biological equilibrium in these digestion systems. The resulting pH values for the 15°C and 25°C temperature conditions shown in Table 3 are generally indicative of a favorable digestion environment.

The production of bicarbonate buffer from organic matter in a digester was shown by Simpson (1) in the following relationships:

Ammonia combines with carbon dioxide and water to form the ammonium bicarbonate buffer. The ammonium bicarbonate produced then reacts with the volatile acids to form volatile acid salts, water and carbon dioxide as shown below (30).

$$NH_4HCO_3 + RCOOH \longrightarrow RCOONH_4 + H_2O + CO_2$$

This relationship demonstrates that if the concentration of volatile acids exceeds that of alkalinity then free volatile acids result. In

the action of saprophytic bacteria on organic matter, the excess acids must be buffered by the available alkalinity or the pH may fall to a level inhibitory to the growth of the methane bacteria (18-337).

The ranges of volatile acids and alkalinity concentrations in the laboratory digesters are also given in Table 3. The relatively high volatile acids concentrations in the 5°C digesters most likely indicated a poor environment for the activity of the methane forming organisms. The comparatively low alkalinity did not appear to be sufficient to buffer the 5°C environment and as a result conditions in the digester tended to remain unfavorable for the complete stabilization of the animal waste.

Sawyer (18-336) stated that for optimum digester operation, the concentration of volatile acids should be 2000 mg/l or less. The concentrations of these acids in the 15°C digesters were greater than 2000 mg/l and therefore the environment in these digesters probably was not at an optimum for digester operation. However, the increased concentration of alkalinity in these units would tend to increase the buffering capacity of the environment and therefore allow higher concentrations of volatile acids without creating unfavorable pH conditions. The 25°C digestion systems appeared to be operating under the most favorable conditions. The alkalinity was quite high and the volatile acids concentrations was generally at a level of 1000 mg/l or lower. The 2000 mg/l upper limit given in Table 3 was recorded shortly after the second sludge addition, therefore it is not indicative

of the usual chemical environment in these units. The increased production of alkalinity in the 15°C and 25°C digesters would, according to Simpson's equations, indicate more reduction in organic matter than in the 5°C units.

Curtis (31), in his study on the operation of anaerobic hog
lagoons, used the ratio of volatile acids to alkalinity as an indicator
of proper conditions in the digestion environment. A high value of
his ratio, usually above unity, was generally indicative of a biological imbalance in the system and the presence of unfavorable conditions.

If the value of this ratio were relatively low, there would probably
be sufficient alkalinity available to buffer the excess acids and
biological balance would tend to be maintained.

Table 3 reveals that in the 5°C units the volatile acid to alkalinity ratio was relatively high. This probably meant that there was insufficient alkalinity available to serve as a buffer for the excess volatile acids being formed and as a result the alkalinity would be combined with the volatile acids. This ratio was observed to decrease for the other two temperatures studied. The ratio in the 15°C and 25°C digesters had an average value of 0.51 and 0.09 respectively. These relatively low values point out that sufficient alkalinity was available to buffer the excess volatile acids and balance between the acid formers and methane formers could be expected.

Sawyer (18-342) reports that the organisms primarily responsible for the production of gas in anaerobic digestion are the methane formers. The total gas production in the 5°C units as seen in Table 4 was less than that at 15°C and only a small fraction of that at 25°C. This extremely low rate of gas production is indicative of low metabolic activity of methane forming organisms in the 5°C digesters.

These organisms are not completely stagnant as is seen by the presence of methane in the gas quality data shown in Table 4.

TABLE 4.

Summary of Gas Production, Gas Quality and Volatile Solids Data

Collected at 5°C, 15°C and 25°C During the Period

of Investigation.

| <u> </u>   | 5°C         | 15°C        | 25°C        |
|--|-------------|-------------|-------------|
| Total Gas Produced in Liters*                                  |             |             |             |
| Per Cent of Methane (CH <sub>4</sub> )<br>in Digester Gas*     | 36.4        | 59.4        | 70.4        |
| Per Cent of Carbon Dioxide (CO <sub>2</sub> ) in Digester Gas* |             |             |             |
| Range of Volatile Solids<br>as a Per Cent of Total Solids      | 63.1 - 66.7 | 62.0 - 65.1 | 46.1 - 52.8 |

<sup>\*</sup>Average of two digesters at each temperature

Because of the low gas production in the 5°C units, nitrogen purging was necessary before a satisfactory gas sample could be secured for analysis in the 5°C units. This low gas production and sampling technique would not alter the fact that methane gas was detected in

these digesters, which is an indication of the activity of methane bacteria.

The increase in gas production in the 15°C and 25°C digesters substantiated the presence of a more favorable biological balance in these units. In order to produce methane, carbon dioxide and water as their end products, the methane organisms must first utilize the volatile organic acids produced in the liquefaction process. Therefore, a greater rate of gas production would indicate an increased rate of volatile acids utilization.

The literature search has shown that in a properly operating digestion system the per cent of methane in digester gas would be 65 to 70% and that of carbon dioxide would be 35 to 40% or less. The 41.7% carbon dioxide and 36.4% methane in the 5°C digesters are not indicative of favorable conditions in the digesters according to these criterion. The increased percentage of methane in the 15°C and 25°C digesters along with the decrease in carbon dioxide were probably associated with more favorable environmental conditions in these digesters than were the levels found at 5°C.

The solids destruction in anaerobic digestion is probably a function of the microbial activity in the system. Table 2 points out that the reduction in volatile matter during the acclimation period was practically negligible for the 5°C digesters. The experimental data, Tables D-1 through D-6, show that the rate of solids reduction also remained minimal in these digesters throughout the period of

evidenced by the chemical parameters observed and the low rate of gas production reaffirm this fact of low solids destruction. The solids destruction in the digestion units at the elevated temperatures was more noticeable. The increase in the amount of volatile matter destroyed was greatest in the 25°C units. This might be expected because these units indicated the highest rate of microbial activity and also had the most favorable environment for maintenance of a bacterial population for anaerobic digestion.

There were certain similarities between this laboratory study on the anaerobic digestion of animal waste and the operation of an animal waste lagoon in Northern climates. The temperatures of digestion studied were similar to those prevalent in these lagoons. The 5°C temperature would be representative of autumn, winter and early spring and the 25°C temperature would be prevalent at times during the summer.

The digestion media used for the laboratory studies was obtained from an anaerobic hog lagoon. The periodic sludge additions to the laboratory digestion units were similar to the intermittent discharges to a hog lagoon.

However, certain dissimilarities did exist. The mixing of the sludge in the digesters, which was done prior to each sampling, was not common to an animal waste lagoon. The sustained environmental temperature would not compare to the seasonal fluctuations found in nature.

The minimal reduction of animal wastes by anaerobic digestion during the winter months could possibly be attributed to two factors. First, the microbial population might not have sufficient time to establish the numbers needed for digestion; or second, the environment at around 5°C is unfavorable for this activity. Most of the solids destruction in animal waste lagoons apparently takes place at temperatures of 15°C or above as indicated by the activity of these respective laboratory systems.

#### CONCLUSIONS

The foregoing laboratory data and discussions have led to the following conclusions:

- 1. Low temperatures (15°C or less) have a retarding effect on the rate of anaerobic digestion of animal waste.
- 2. The rate of destruction of solids in animal wastes can be related to temperature through observation of the rate of gas production.
- 3. The rate of solids reduction in the sludge layers of animal waste lagoons during periods of temperature of 5°C or less may be considered negligible.

#### SUGGESTIONS FOR FUTURE STUDY

It is suggested that an investigation be made to determine:

- 1. The effects of various levels of periodic loading on the anaerobic digestion of animal wastes at temperatures approximating those found in animal waste lagoons.
- 2. The effect of mixing on the anaerobic digestion of animal wastes at temperatures approximating those found in animal waste lagoons.
- 3. The effect of varying the temperature on a single batch of animal waste to more nearly duplicate the field conditions prevalent in northern climates.

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#### APPENDIX A

# CALCULATION FOR CONVERSION OF OBSERVED GAS VOLUMES TO STANDARD TEMPERATURE AND PRESSURE, O°C AND 29.92 INCHES OF MERCURY.

A combination of Boyle's Law and Charle's Law gives the following relationship:

$$V_2 = V_1 \times \frac{P_1}{P_2} \times \frac{T_2}{T_1}$$

Where: V2 = Volume at Standard Conditions

V<sub>1</sub> = Volume of gas observed

P1 = Barometric pressure at time of reading

P<sub>2</sub> = 29.92 in. Hg. = 760 mm Hg.

T<sub>2</sub> = 0°C = 273°R

T<sub>1</sub> = Temperature of the air at time of reading + 273°

This then gives a factor of

$$V_2 = V_1 \times \frac{P_1}{29.92} \times \frac{273}{T_1}$$

$$V_2 = \frac{V_1 P_1}{T_1} \times 9.124$$

#### APPENDIX B

# DETERMINATION OF VOLATILE ACIDS IN DIGESTER LIQUOR BY CHROMOTOGRAPHY

### Principle

Organic acids are separated from inorganic acids and alkalies by placing an acidified sample on a short silicic acid column and eluting with a n-butanol-chloroform solvent. The organic acids are measured in the eluate by titration with standard NaOH.

#### Materials

Vacuum source.

Gooch crucibles, Coors No. 3 tall form.

Crucible holder or #6 one-hole rubber stopper.

Filtering flask--250 ml.

Buret, 10 ml. and buret holder. wasses it lear thank hand them

Pipets, 1 and 5 ml.

Graduates, 50 ml., Table vaccing fallow thank and put 10 grants of

Glass fiber filter paper 2.1 cm. #x-934-AH.

Funnel, 65 mm, short stem.

Filter paper, Whatman #2, 11.0 cm.

# Reagents sair, resolutes on through fill best file to the seemply

Silicic acid: Mallinckrodt's Silicic Acid No. 2847 "specially prepared for chromatography." The silicic acid is dried at 103°C and stored in a desiccator.

CB<sub>25</sub> solvent: Mix 250 ml of n-butyl alcohol with 750 ml of chloroform.

NaOH: Normality of NaOH is determined on the day of test.

H2SO4: Approximately 10 N H2SO4.

Thymol Blue indicator: O.1 per cent by weight in absolute methyl alcohol or ethyl alcohol.

Phenolphthalein indicator: O.l per cent by weight in absolute methyl alcohol or ethyl alcohol.

Methyl alcohol: Absolute. As all all anthyl alcohol and 15 deeps

# Procedure a to the cluster in the flashe

- (1) Filter through 11.0 cm #2 filter paper, sufficient sludge to obtain approximately 10 to 15 ml filtrate.
- (2) Place a glass fiber filter pad on the bottom of the Gooch crucible and draw down by suction on a vacuum filter flask and then release vacuum.
- (3) Remove column from vacuum filter flask and put 10 grams of dry silicic acid in the column tapping lightly to aid in packing.
- (4) Replace column on vacuum filter flask and apply vacuum to pack the silicic acid. It may be necessary to remove the column from the flask, tap again, replace on vacuum filter flask and re-apply vacuum in order to firmly pack the silicic acid with no drawing away from the column wall.
- (5) Add three drops of thymol blue indicator to filtrate from step #1.

- (6) Add 10 N  $H_2SO_4$  drop-wise with mixing until sample is red to thymol blue, a pH of about 2.0.
- (7) Place 5.0 ml of acidified sample on the silicic acid in the column. Release vacuum as soon as sample is drawn into silicic acid.
- (8) With suction, draw CB<sub>25</sub> solvent through the silicic acid into the filtering flask, keeping the column full until 50 ml have been added and drawn into the flask.
- (9) Remove filter flask, add 40 ml methyl alcohol and 15 drops of phenolphthalein to the eluate in the flask.
  - (10) Titrate with NaOH of known normality.
- (11) Run a flank titration on the solvents (approximately 45 ml CB<sub>25</sub> and 40 ml methyl alcohol) and subtract from the above titration.

### Reporting

N x (ml of NaCH used on sample - ml used on blank) x 1.2x1000

Volume of Sample

#### APPENDIX C

# PROCEDURE FOR THE DETERMINATION OF GAS CONSTITUENTS IN THE 5°C DIGESTER GAS.

#### Principle:

Because of the low rate of gas production in the 5°C units, a method was set up to compensate for the inability of the digesters to flush the atmospheric air from the digester. The procedure is outlined as follows.

- The volume of gas above the digesting sludge (about 1000 ml) was replaced with nitrogen gas by purging.
- The digester was permitted to produce sludge gas, thereby creating a mixture of nitrogen plus sludge gas in the digester.
- 3. A standard gas analysis was run on the mixture present in the digester.
- 4. The values from the gas analysis were corrected to a 100% sludge gas value by multiplying them by a factor of

100

amount of gas in mixture

amount of sludge gas and initial volume of  $N_2$ 

APPENDIX D

8 8 9

TABLE D-1. Summary of Data Collected During the Period of Investigation for the Anaerobic Digestion Studies.

Period from June 11th to August 8th, 1965

| 200      |
|----------|
| -        |
| NO.      |
| DIGESTER |

| Date                                   | 17-9 | 6-20 | 97-9 | 6-28 | 7-10 | 7-18 | 7-20 | 7-27 | 4-8  |  |
|--|------|------|------|------|------|------|------|------|------|--|
| Temperature (°C)                       | 0.9  | 5.0  | 5.0  |      | 5.5  | 0.9  | 5.5  | 5.5  | 5.5  |  |
| нф                                     | 6.5  | 9.9  | 6.5  |      | 9.9  | 2.9  | 6.5  | 9.9  | 9.9  |  |
| Volatile Acids (mg/l as Acetic Acid)   | 2010 | 2980 | 5450 |      | 0464 | 6270 | 0485 | 5910 | 9300 |  |
| Alkalinity (mac/1 ac CaCO)             | 8100 | 8300 | 5380 |      | 6830 | 7780 | 9050 | 7220 | 8130 |  |
| Total Solids (%)                       | 5.3  | 4.88 | 66.4 | 4.76 | 4.91 | 4.95 | 4-38 | 4.36 | 4.37 |  |
| Volatile Solids<br>(% of Total Solids) | 2.99 | 9.69 | 4.49 | 65.2 | 4.49 | 64.3 | 63.1 | 63.1 | 63.1 |  |
|  |      |      |      |      |      |      |      |      |      |  |

Summary of Data Collected During the Period of Investigation for the Anaerobic Digestion Studies. TABLE D-2.

Period from June 11th to August 8th, 1965

|  |      | IG   | DIGESTER NO. 2 5°C | 0. 2 5°C |      |      |      |       |      |
|--|------|------|--------------------|----------|------|------|------|-------|------|
| Date                                       | 6-11 | 6-20 | 6-26               | 6-28     | 7-11 | 7-18 | 7-20 | 7-27  | 4-8  |
| Temperature (°C)                           | 0.9  | 5.0  | 5.0                |          | 5.5  | 0.9  | 5.5  | 5.5   | 5.5  |
| на   | 6.5  | 6.5  | 4.9                |          | 9.9  | 9.9  | 6.5  | 9.9   | 6.7  |
| Volatile Acids (mg/l as Acetic Acid)       | 0429 | 5720 | 5980               |          | 4700 | 0459 | 5650 | 5730  | 6120 |
| Alkalinity<br>(mg/l as CaCO <sub>2</sub> ) | 7890 | 8190 | 5290               |          | 6580 | 7280 | 8140 | 90099 | 8230 |
| Total Solids (%)                           | 4.61 | 5.18 | 4.32               | 3.80     | 4.19 | 4.15 | 3.79 | 3.93  | 3.99 |
| Volatile Solids<br>(% of Total Solids)     | 62.8 | 9.49 | 59.6               | 58.1     | 60.9 | 4.09 | 59.9 | 60.3  | 60.7 |

TABLE D-3. Summary of Data Collected During the Period of Investigation for the Anaerobic Digestion Studies

Period from June 11th to August 8th, 1965

|   |       | d     | GESTER N | DIGESTER NO. 3 15°C | OI.  |       |       |      |       |
|---|-------|-------|----------|---------------------|------|-------|-------|------|-------|
| Date                                    | 6-11  | 6-20  | 97-9     | 6-28                | 7-10 | 7-18  | 7-20  | 7-27 | 8-4   |
| Temperature (°C)                        | 15.0  | 15.0  | 15.0     | 15.0                | 15.0 | 15.0  | 15.0  | 15.0 | 15.0  |
| Hď                                      | 8.9   | 6.8   | 6.7      |                     | 6.9  | 6.9   | 6.8   | 6.9  | 6.9   |
| Volatile Acids (mg/l as Acetic Acid)    | 4980  | 5850  | 14830    |                     | 3950 | 0064  | 4380  | 5360 | 3960  |
| Alkalinity (mg/l as CaCO <sub>5</sub> ) | 10040 | 10120 | 7770     |                     | 9220 | 10530 | 04/26 | 9820 | 11950 |
| Total Solids (%)                        | 3.80  | 4.11  | 4.24     | 3.59                | 3.95 | 4.17  | 3.74  | 3.92 | 3.71  |
| Volatile Solids<br>(% of Total Solids)  | 63.1  | 63.8  | 63.3     | 62.0                | 63.9 | 62.4  | 63.2  | 65.1 | 61.5  |

TABLE D-4. Summary of Data Collected During the Period of Investigation for the Anaerobic Digestion Studies

Period from June 11th to August 8th, 1965

DIGESTER NO. 4 15°C

| Date                                    | 6-11  | 6-20 | 92-9 | 6-28 | 2-10 | 7-18 | 7-20 | 7-27 | 4-8   |  |
|---|-------|------|------|------|------|------|------|------|-------|--|
| Temperature (°C)                        | 15.0  | 15.0 | 15.0 | 15.0 | 15.0 | 15.0 | 15.0 | 15.0 | 15.0  |  |
| Hď                                      | 8.9   | 2.9  | 9.9  |      | 8.9  | 2.9  | 2.9  | 8.9  | 6.9   |  |
| Volatile Acids (mg/l as Acetic Acid)    | 2400  | 5850 | 5220 |      | 4500 | 5720 | 5290 | 5540 | 4680  |  |
| Alkalinity (mg/l as CaCO <sub>2</sub> ) | 10000 | 9830 | 7560 |      | 9130 | 9370 | 8790 | 8480 | 10040 |  |
| Total Solids (%)                        | 3.71  | 3.59 | 3.64 | 3.19 | 3.40 | 3.43 | 3.28 | 3.45 | 3.27  |  |
| Volatile Solids<br>(% of Total Solids)  | 63.9  | 61.7 | 62.5 | 4.09 | 61.5 | 2.19 | 61.2 | 62.7 | 60.7  |  |

TABLE D-5. Summary of Data Collected During the Period of Investigation for the Anaerobic Digestion Studies

Period from June 11th to August 8th, 1965

|   |       | 티     | DIGESTER NO. 5 25°C | 0. 5 25 | OI    |       |       |       |                   |  |
|---|-------|-------|---------------------|---------|-------|-------|-------|-------|-------------------|--|
| Date                                    | 611   | 6-20  | 97-9                | 6-28    | 7-10  | 7-18  | 7-20  | 7-27  | \$ <del>-</del> 8 |  |
| Temperature (°C)                        | 25.0  | 25.0  | 24.5                |         | 25.5  | 26.5  | 26.5  | 26.0  | 25.5              |  |
| Hď                                      | 7.4   | 7.4   | 7.3                 |         | 7.5   | 7.4   | 7.2   | 2.6   | 2.6               |  |
| Volatile Acids (mg/l as Acetic Acid)    | 1350  | 890   | 1150                |         | 370   | 820   | 2000  | 0111  | 8                 |  |
| Alkalinity (mg/l as CaCO <sub>2</sub> ) | 11520 | 11830 | 02111               |         | 11770 | 12160 | 12140 | 12990 | 15050             |  |
| Total Solids (%)                        | 3.15  | 3.04  | 3.17                | 2.93    | 2.89  | 3.02  | 3.8   | 3.80  | 3.76              |  |
| Volatile Solids                         | 50.5  | 49.5  | 50.1                | 49.2    | 46.5  | 47.1  | 52.3  | 51.3  | 50.5              |  |
| (% of Total Solids)                     |       |       |                     |         |       |       |       |       |                   |  |

TABLE D-6. Summary of Data Collected During the Period of Investigation for the Anaerobic Digestion Studies.

Period from June 11th to August 8th, 1965

|   |       | I     | GESTER N | DIGESTER NO. 6 25°C | ol    |       |       |       |       |
|---|-------|-------|----------|---------------------|-------|-------|-------|-------|-------|
| Date                                    | 6-11  | 6-20  | 92-9     | 6-28                | 7-10  | 7-18  | 7-20  | 7-27  | 4-8   |
| Temperature (°C)                        | 25.0  | 25.0  | 24.5     |                     | 25.5  | 26.5  | 26.5  | 56.0  | 25.5  |
| на                                      | 7.4   | 7.4   | 7.3      |                     | 2.6   | 7.4   | 7.2   | 7.7   | 7.7   |
| Volatile Acids (mg/l as Acetic Acid)    | 1210  | 049   | 049      |                     | 620   | 820   | 2000  | 920   | 720   |
| Alkalinity (mg/l as GaCO <sub>2</sub> ) | 11820 | 11610 | 11090    |                     | 11980 | 12280 | 12050 | 12450 | 14710 |
| Total Solids (%)                        | 3.13  | 3.39  | 3.16     | 2.77                | 2.66  | 2.98  | 3.88  | 3.79  | 3.63  |
| Volatile Solids<br>(% of Total Solids)  | 50.7  | 50.1  | 49.7     | 4.84                | 45.9  | 46.1  | 22.8  | 50.5  | 48.1  |