1965

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Recommended Citation

Edmonds, Clinton James, "Effect of solids concentration on the anaerobic digestion of domestic sewage sludge " (1965). *Retrospective Theses and Dissertations*. 3913.
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EDMONDS, Clinton James, 1939–
EFFECT OF SOLIDS CONCENTRATION ON
THE ANAEROBIC DIGESTION OF DOMESTIC
SEWAGE SLUDGE.

Iowa State University of Science and Technology
Ph.D., 1965
Engineering, sanitary and municipal

University Microfilms, Inc., Ann Arbor, Michigan

EFFECT OF SOLIDS CONCENTRATION ON THE ANAEROBIC DIGESTION OF DOMESTIC SEWAGE SLUDGE

by

Clinton James Edmonds

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Sanitary Engineering

Approved:

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

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Signature was redacted for privacy.

Dean of Graduate College

Iowa State University
Of Science and Technology
Ames, Iowa

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I. INTRODUCTION

Sewage contains mineral and organic matter in suspension, in the colloidal state and in solution. Removal or stabilization of these matters may be accomplished at a sewage treatment works by physical, chemical and biological methods. The methods used will depend upon the type of sewage and the degree of treatment required. Settleable organic and inorganic materials in the raw, untreated sewage and settleable material from the biological portions of treatment processes are frequently removed by gravitational sedimentation or filtration. Several methods of disposing of the solids which are removed are available. Solids disposal practice includes barging out to sea, burial, landfill, placing the solids on the land to improve fertility, incineration and wet combustion. The particular method used is determined by economic considerations and the characteristics of the sludge. The first stage in the preparation of the sludge for disposal is usually concentration which reduces the quantity of material to be handled. Concentration increases the solids concentration in sludge by reducing the liquid content. The liquid is removed by drying raw and biologically treated sludges on sand beds or using concentration tanks or vacuum filtering the sludge. Combinations of the above methods are frequently used. A factor common to all the methods of preparing sludge for disposal is that they are expensive. One of the less expensive and more successful of the methods is to treat the sludge biologically and to dry the treated sludge on sand beds. Two forms of biological treatment are available, the one using micro-organisms which require molecular oxygen for their metabolic processes and the other using micro-organisms which require oxygen in a combined form. It has been found uneconomical in practice to supply molecular oxygen to sewage sludge at normal concentrations at a rate sufficient to keep the system aerobic. For this reason the biological treatment of sewage sludge usually takes place under anaerobic conditions. The advantages achieved by biologically treating sewage sludge may be illustrated by the differences in characteristics of raw sewage sludge and digested or biologically treated sewage sludge. Raw sewage sludge is difficult to dewater, it is a potential odor nuisance, the organic content of the sludge is unstable, it contains pathogenic organisms and when it is spread on soil it tends to clog the soil pores. Digested sewage sludge dewaters more readily, the odor problem is relieved to a large extent, the coliform count of sewage sludge digested for 30 days at 35°C to 38°C is reduced by 99.8 percent and the sludge is useful as a soil builder. The fertilizing value of digested sewage sludge is poor compared to commercial fertilizers.

Recent research in the anaerobic digestion of sewage sludge has been directed towards two principal objectives:

to increase knowledge of the basic mechanism of the digestion process and to develop the process so that it is more efficient. The first objective has stimulated research in the biochemical and biological events taking place in the digestion process. To the engineer, the second objective is of primary importance because a more efficient process usually means a more economical process. One of the avenues of research is concerned with increasing the loading rates to anaerobic digesters. Several methods have been tried and these are discussed in the Literature Review. One method would be to increase the solids concentration of the feed sludge to a digester by reducing the water content. This allows a longer time period for the solids to remain in the digester if the rate of solids loading remains unchanged. Alternatively, the detention time may be held constant and the loading rate to the digester increased. Although sludge concentration prior to digestion is used in many plants, little research has been directed towards evaluating the effect of varying the solids concentration in a digester on the treatment process. The purpose of this dissertation is to determine the effect of the solids concentration inside a digester on the anaerobic digestion process.

II. LITERATURE REVIEW

A. Historical Development of Sludge Digestion

Until the latter part of the nineteenth century almost all sewage was disposed of in one of two ways; it was discharged untreated to either the nearest body of water or to the nearest plot of available land. Problems were encountered with both methods. When the untreated sewage was discharged to a body of water, the water became polluted and a danger to the public health, unsightly sludge banks and scum bodies would form and in many cases foul odors would be emitted. The contaminating effects of sewage disposed in the area of shellfish farms were well known by the latter part of the nineteenth century. Discharge of the untreated sewage on land created a public health hazard especially if there was some connection between the disposal location and the source of a domestic water supply. A great disadvantage of land disposal of sewage was the clogging of the soil pores with the solids in the sewage rendering the soil impervious to percolation of more sewage. The land frequently was unsightly and odiferous (48).

Many attempts were made to correct the poor state of sewage disposal and in the eighteen eighties the first major progress was made. In 1858 the pollution of rivers in England was legally prohibited but the laws were loosely administered. In 1892 the Mersey and Irwell act was passed. This act was the first of several acts setting up Joint Committees composed of representatives from all the sanitary authorities bordering on a specific river. The Joint Committees succeeded to some degree in enforcing river pollution laws. Similar legislation against river pollution was being enacted on the Continent (39). In 1886 the Lawrence Experiment Station was established by the Massachusetts State Board of Health to advance the study of the purification of water and sewage (123). This was the first institution of its kind in the world. In the United States legislation to prevent river pollution came later. Probably the first serious effort in this direction was made by the U. S. Public Health Service in 1913 when it established a stream pollution investigation station in Cincinnati, Ohio. Committees for the prevention of river pollution, similar to those organized in England, were set up on many of the rivers in the United States. In the 1940's and 1950's legislation gave the U. S. Public Health Service the power to order communities and industries to stop polluting interstate waters.

The unfavorable effects of solids on sewage disposal methods gave rise to the practice of settling sewage to remove the solids. A sludge was produced in the settling process and this had to be disposed of. The cesspool was probably the first sewage settling tank to be used by man. Wastes were allowed to flow into it without control. Some solids would settle to the bottom where they would begin to decompose due to the action of anaerobic organisms and the supernatant liquid would either overflow or percolate into the surrounding earth. The cesspool was merely used as a place to dispose of waste and any treatment action due to sedimentation and anaerobic action was not considered in its design. A report (114) in 1857 describes a tank designed by Henry Austin to retain solids by sedimentation. This tank had a capacity adequate to remove solids from the wastes from sizable towns. Austin did not appreciate the solids reduction which took place in the settled sludge by septic action.

In France in 1860, Louis H. Mouras developed the Mouras Automatic Scavenger which was virtually an overflow cesspool. Although not very successful, the Automatic Scavenger was designed to remove solids from the sewage from individual households by sedimentation and to reduce the quantity of settled solids by anaerobic action. Many variations of the Automatic Scavenger were constructed; all with limited success (39). Plain settling was introduced in 1891 as a preliminary sewage treatment process in Massachusetts, but few installations were made thereafter because of the difficulty of sludge removal and disposal. In 1900, there were some 62 sewage treatment plants in the United States and of these, two employed plain sedimentation, 14 employed some form of chemical treatment and 46 plants made no attempt to remove suspended solids (123). The problem of removing sludge from settling tanks was not solved until 1916 when the first mechanical sludge collecting device was invented by Dorr, who subsequently founded an equipment company to service the waste treatment field. Previous to this time it was necessary to drain the sedimentation tank and to remove the sludge by hand. In 1900 the preparation of sewage sludge for ultimate disposal was accomplished mainly in two ways: the sludge was dewatered by drying on sand beds, or by filtration with expensive and messy plate presses. Drying on sand beds was a lengthy process with unfavorable weather conditions. As early as 1904 George Warren Fuller, a person with much foresight, indicated that sludge disposal would be an important and expensive item of waste treatment.

In 1895 Cameron developed the septic tank to treat a portion of the sewage from Exeter, England (94). The septic tank was regarded by many as the answer to all sewage treatment problems but Cameron regarded it only as a preliminary treatment. The septic tank is defined as, "a horizontal, continuous flow, one story sedimentation tank through which sewage is allowed to flow slowly to permit suspended matter to settle to the bottom where it is retained until anaerobic decomposition is established, resulting in the changing of some of the suspended organic matter into liquid and gaseous substances, and a consequent reduction in the quantity of sludge to be disposed of" (5). One of the first septic tanks installed in the United States was at Champaign, Illinois in 1897. A problem with the action of the septic tank was the buoying up of sludge by the gas produced in the anaerobic action and the appearance of this sludge in the effluent. About 1906 Travis invented the Travis Hydrolytic Tank. The tank was divided into three compartments, two in which solids could settle and a third into which the settled solids moved to undergo anaerobic action. Gas bubbles from the sludge compartment rose and interfered with the settling action in the other two compartments. Dr. Karl Imhoff of Germany designed the first Imhoff tank in 1906. This tank was also separated into three compartments: a sedimentation chamber, a digestion chamber situated below the sedimentation chamber and a scum chamber. A baffle was placed below the sedimentation chamber to prevent gas bubbles from the digestion chamber rising and interfering with the process of settling. Gas produced by anaerobic action left the tank through the scum chamber. The scum chamber was connected directly to the digestion chamber (5).

The Imhoff tank was introduced in the United States (5) in 1909 and the first unit was constructed in New Jersey in 1911. The tank was used extensively almost immediately because of the patents on the Cameron Septic Tank which restricted their use. A disadvantage of the Imhoff tank was the depth of the tank, 25 to 35 ft, and hence the high construction cost. The digestion chamber of the Imhoff tank was usually designed to store sludge for 6-12 months.

About 1915 the Kremer cell was invented in Germany. This consisted of a sedimentation tank to which two chambers were attached. Sludge was removed from the sedimentation tank to the first chamber where digestion of the mixed sludge and sewage took place. Partially digested sludge from this chamber was transferred to the second chamber where it was allowed to remain under a layer of sewage until completely digested.

H. W. Clark of the Lawrence Experiment Station suggested in 1899 that separate sludge digestion, i.e. the removal of the sludge from the sedimentation tanks to another tank where it could undergo anaerobic decomposition, might solve many problems of dealing with the sludge. A separate sludge digestion tank was tested in 1899 but the difficulties involved with the process were such that the Travis and Imhoff tanks were preferred. The advantages of separate digestion are many because the optimum conditions for digestion are not the optimum conditions for settling. Kniebuhler in 1887 recommended removing sludge from septic tanks frequently and in a fresh condition to obtain a higher degree of treatment of the liquid. The first separate sludge digestion tanks were placed in service between 1910 and 1912 at Birmingham in England, Baltimore in the U.S.A. and at the Essen-Rellinghausen plant in Germany. Prior to this time, sludge lagoons were used in England and America. The sludge remained in the lagoons for years and eventually digested and dried out. At Birmingham, the design capacity of the separate sludge digestion tank was 9.2 cu ft per capita and at Baltimore the design capacity was 3.95 cu ft per capita. The sludge from the Imhoff tanks at the Essen-Rellinghausen plant was pumped to separate digesters daily. In the separate digesters the rate of anaerobic action did not equal that in the Imhoff tank. The reason for this unexpected finding was assumed to be the low temperature of the sludge since no warming effect from inflowing sewage was available as in the Imhoff tank.

By 1925 many separate digestion plants were in operation. Records indicate that the gas produced in the anaerobic process was utilized as early as 1907. In this year James at the Matunga Leper Asylum at Bombay, India (50) utilized the gas to drive a small engine. When Cameron covered his septic tanks in 1912, he used the gas produced to light the grounds of the treatment plant and to run some small engines. In 1921 Birmingham, England utilized the gas from their separate digesters to run internal combustion engines. This was the first use of gas on a commercial scale, a practice which did not become common in the United States until 1933. Gas was collected on a large scale at the Essen-Rellinghausen plant and first delivered to a municipal system in 1923.

Until 1926, if it was necessary to cover separate digesters, Imhoff tanks or septic tanks to either collect gas or control odors, fixed covers were universally used (123). The dangers of fixed covers were amply illustrated by the digester explosions which occurred at Saratoga Springs in 1906 and at Florenceville, N. C., in 1915 (99). In 1926 the first floating covers were placed on sludge digesters at Birmingham, England and Plainfield, New Jersey. At Birmingham the covers, which were a series of concrete rafts with a small cone in the center, were installed to collect gas to run a 150 hp engine (50). The advantages of the floating cover over the fixed cover were numerous. They submerged solids at the top of the sludge thus helping to prevent scum formation; it was not found necessary to balance additions of sludge to the digester with removals of sludge; the quality of supernatant was better because it could then be withdrawn at will; and the floating cover was safer. Digester gas forms an explosive mixture with air and, when withdrawing sludge from a fixed cover digester, it was not unusual to draw in some air.

The disappointing results of the separate sludge digesters at the Essen-Rellinghausen plant due to low temperatures of the sludge promoted the first heating system installed in a separate sludge digester in 1927. Almost immediately the rate of digestion surpassed that of the Imhoff tank (123). Although heat had been applied with success to the contents of an earthen digester at Plainfield, N. J. prior to 1927, the first sludge digestion tank built in the U. S. with the heating coils installed as an integral part of the tank was constructed at Antigo, Wisconsin in 1928. In 1927 Imhoff used the gas produced in an Imhoff tank to heat water which was injected into the Imhoff tank to supply heat to the digestion compartment. Goudy as cited by Mau (101) in 1929 reported that there were 59 separate sludge digestion plants in the United States, 41 of which were heated. The average design capacity of the heated digesters was 1.5 cu ft per capita. Digesters have been heated in many ways with varying degrees of success. From 1927 to 1947 the majority of digesters in the United States were heated by circulating hot water through heating coils placed inside the digester mounted on the walls of the digester (47). Walraven (174) suggested that the water in the coils should be less than 150° F to reduce sludge incrustation on the pipes which would lead to a decrease in heat transfer efficiencies. He also mentioned the improved circulation obtained by leaving a space between the walls of the digester and the heating coils. About 1947 there was a movement towards the use of external heat exchangers. Raw sludge or supernatent liquor was pumped from the digester through a heat exchanger and back into the digester. Advantages in heat transfer efficiencies, temperature control, circulation and maintenance were obtained. Other methods of heating have been attempted and some are still used: injecting live steam into a digester; mixing hot water with raw sludge; direct heating by submerged combustion; and diffusion of heated combustion products through raw sludge. At the present time, the most popular method is the use of external heat exchangers.

The benefits obtained from mixing digesting sludge were realized by Imhoff when he designed the Imhoff tank. Imhoff tanks are deep to keep the temperature of the sludge in the digestion compartment reasonably uniform and to allow the gas bubbles evolved in the digestion process to give more stirring action to the sludge. Prüss (129) advocated digester mixing to remove the organisms in the digestion process from the local concentrations of their own end products. He mentioned that mixing also helps to maintain uniform temperature conditions throughout the digester and to control scum layers.

A portion of the material entering a digester will float and form a scum layer. Scum layers over 8 ft thick have been reported. According to Simpson (160), scum formation in a digester has been described as the greatest single problem in sludge digestion. It will decrease the effective volume of the digester and can damage floating covers and other equipment. Adequate mixing will control scum layer formation. Bacon (8) in 1944 discussed mixing practices and mentioned that there were advantages and disadvantages. The advantages of mixing included a greater and more uniform gas supply, scum reduction and improved seeding of raw incoming sludge. The disadvantages were a poor supernatent and increased power costs. The methods of mixing include: mechanical mixing by means of revolving arms, propellors and sweeps; gas mixing by recirculating digester gas; recirculation of sludge through a heat exchanger; and pumping the raw sludge into the digester at various locations. Morgan (116) studied gas mixing as a means of increasing the rate of sludge digestion. He found that adequate mixing could be obtained with gas recirculation and that the rate of sewage sludge digestion could be increased by a factor of approximately four times. A very real advantage of the process was that a sludge having good drying characteristics was produced. With the advent of complete mixing of the digestion tank contents, the stage process of digestion and a sludge concentration process became necessary. In 1931 the first stage digestion process was placed in operation at the Harbor City Plant, Los Angeles. This process consisted of four stages in which the solids of higher specific gravity in the digesting sludge, i.e., lower volatile solid content, were moved ahead to the following stage until the solids were completely digested. A modified form of this system, the two-stage process, is common practice in sludge digestion today. In a two-stage process, the present practice is to keep the sludge in the first stage digester for six to eight days (6). Most of the gas is given off in this stage. To separate the solids from the supernatant and to complete the process of digestion, the sludge is then aged in a second stage digester for 22 to 24 days.

When domestic sewage sludge is held in a tank the solids in the sludge will tend to settle to the bottom of the tank leaving a relatively clear liquid at the top of the tank. The clear liquid is known as supernatant. In operating separate sludge digesters, raw sludge is pumped into the digester several times a day. Just prior to the addition of raw sludge an equivalent amount of supernatant is withdrawn to keep the volume of waste in the tank nearly constant. Thus, the solids are allowed to remain in the digester but the liquid is removed daily. With the increased use of heat exchangers and artificial mixing sludge does not separate into a supernatant portion and a solids portion. Then, to keep the volume of waste in the tank constant sludge solids are removed daily as raw sludge is added. It is obvious that the average length of time a solid particle will remain in a digester is less in mixed digesters than in unmixed digesters. Thus, if a raw sludge volume equal to one tenth of the volume of the digester is added daily to a mixed digester the average length of time a solid particle will remain in the digester is ten days. The length of time a solid particle will remain in an unmixed digester, from which supernatant is withdrawn, will be more than ten days. If a certain detention time, or the average length of time a solid particle remains in a digester, is required by a particular sludge digestion system then a greater digester volume will have to be provided for a mixed digester than for an unmixed digester. Other disadvantages of the mixed digester are: maintaining a larger volume of sludge at a certain temperature and dispersion of the food available to the organisms in the digester. Torpey (168) has alleviated many of the problems with mixed digesters by concentrating the sludge prior to digestion. If sludge is concentrated from three percent solids to six percent solids the volume of the sludge is halved. The reduction in volume of the sludge will allow a longer detention time for the solids in a digester or alternatively if the detention time is held constant a smaller digester can be used.

B. The Anaerobic Digestion Process

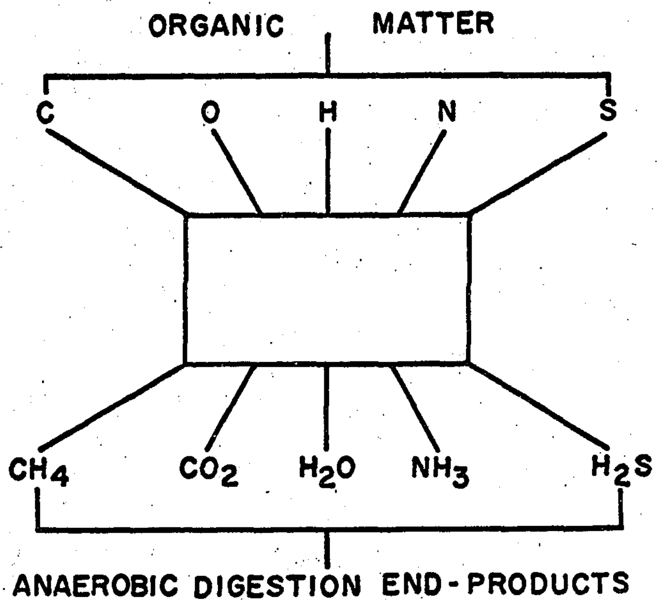
The total solids content of domestic sewage sludge depends to a large extent upon the composition of the sludge and the process by which it is produced. Fair and Geyer (41) give values of total solids in sludge ranging from 2.5 percent from a plain sedimentation process up to 10 percent for the fresh humus sludge from a trickling filter. Many analyses have been made of the solids portion of sludge (23, 71, 75, 115). In general, the raw solids consist of 60 to 80 percent volatile matter and 20 to 40 percent fixed matter determined by solids analyses according to Standard Methods (3). Heukelekian (71) analyzed the organic portion of domestic sewage sludge and found the basic composition to be approximately 34 percent ether soluble material, 27 percent crude protein and 24 percent ash. Other compounds were also present including starches, sugars, celluloses and organic acids. Buswell and Neave (23) obtained similar results: protein content of 19.4 percent;

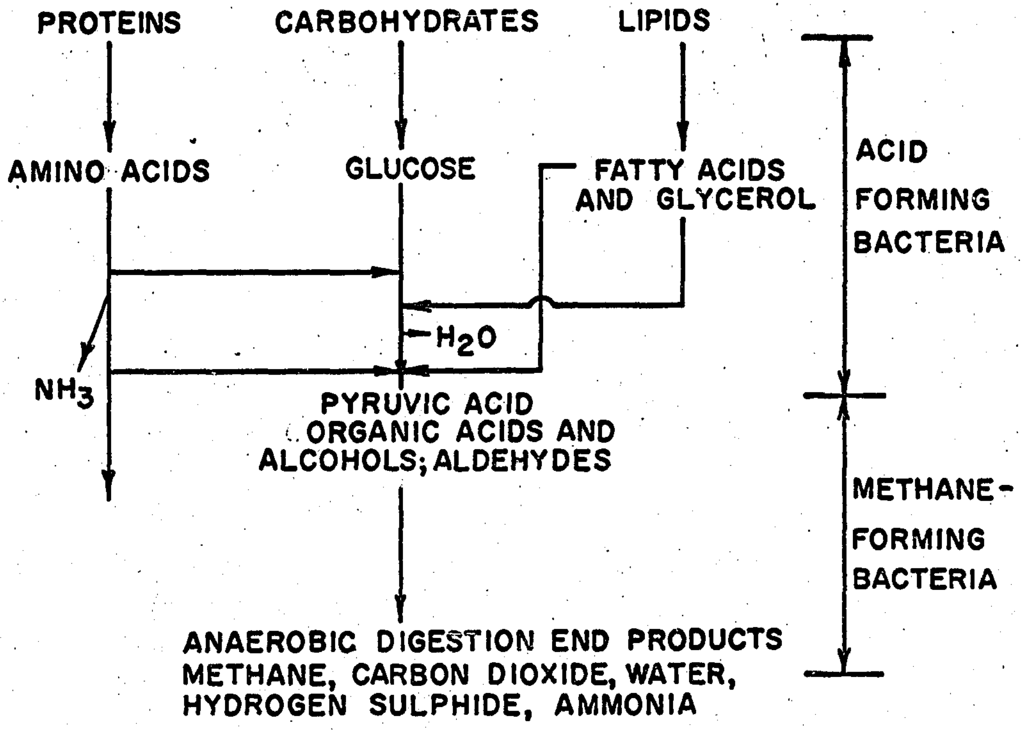
grease content of 25.2 percent; and crude fiber content of 10.8 percent. The total volatile matter by analysis was 60.9 percent. Heukelekian and Balmat (75) obtained 19.1 percent grease, 25.1 percent nitrogenous matter and 23.6 percent carbohydrate. The total volatile matter was 78 percent of the total solids. In summary, the composition of fresh domestic sewage sludge consists of various proportions of fats, carbohydrates and proteins. Variations may be due to variations in habits, type of analysis and season.

The heterogeneous mixture of sludge described above provides an ample food source for biological life. In anaerobic digestion the complex materials such as proteins, fats and carbohydrates are broken down by micro-organisms into simpler substances (Figure 1). Energy is thus provided for the growth of the micro-organisms. According to Heukelekian (71), the breakdown of the organic material consists of two major steps, the liquefaction of solids and the conversion of some of the liquids to gases. Simpson (160) states that at least two distinct groups of organisms are involved in the anaerobic decomposition of sewage sludge. The organisms in the group responsible for the liquefaction of solids are called "acid-producers" and the organisms in the group responsible for gasification are called the "methane producers." Successful digestion is based upon the synchronization of the two groups of organisms in the liquefaction and gasification processes. An unbalanced state

Figure 1. Formation of anaerobic digestion end products

Figure 2. Simplified decomposition pathways for organic matter


ORGANIC MATTER
C
O
H
N
S
CH_4
CO_2
H_2O
NH_3
H_2S
ANAEROBIC DIGESTION END-PRODUCTS


PROTEINS
CARBOHYDRATES
LIPIDS
AMINO ACIDS
GLUCOSE
FATTY ACIDS AND GLYCEROL
ACID FORMING BACTERIA
H_2O
NH_3
PYRUVIC ACID
ORGANIC ACIDS AND ALCOHOLS; ALDEHYDES
METHANE-FORMING BACTERIA
ANAEROBIC DIGESTION END PRODUCTS
METHANE, CARBON DIOXIDE, WATER, HYDROGEN SULPHIDE, AMMONIA

often occurs in the sludge digestion process where the products of the acid producing organisms accummulate and inhibit the methane producing organisms. The methane producing organisms can be inhibited to the extent that no gas is formed. The condition of the digestion process is then called "stuck" digestion. In this condition the materials in the digester may be more obnoxious than the original sewage sludge.

1. Liquefaction

The modes of action of the two groups of organisms are described by Heukelekian (71). Liquefaction as applied to this discussion implies the transformation of large solid particles of sludge into a soluble or finely dispersed state. The liquefaction process is brought about by hydrolytic reactions catalyzed by extra-cellular enzymes. The acid producing organisms make contact (13, 78) with, or come into the close proximity of, the sludge particles and excrete extra-cellular, hydrolytic enzymes to liquefy the solid material. The enzymes do not diffuse into the surrounding medium and become diluted. Also the products of the hydrolytic reaction diffuse directly into the cell for utilization by the cell rather than diffusing into the surrounding medium. The end products of the metabolic activity of the acid producing organisms do diffuse into the surrounding liquid medium and are utilized by the methane producing organisms. Formation of end products is the result of many biochemical reactions. In many instances the reactions are linked to form pathways which represent in logical order the decomposition of organic compounds. The pathways may not be always correct but they best fit the known facts at the present time.

a. Carbohydrate decomposition Carbohydrate decomposition has been studied extensively because of its importance to the fermentation industries. Lackey and Hendrickson (96) indicate that carbohydrates must be in a form no larger than dissacharides or monosaccharides before they can pass through the cell wall and be metabolized. Accordingly, the first step in carbohydrate decomposition must be the hydrolysis of the large polysaccharide molecules by extra-cellular enzymes. Starch is hydrolyzed by the enzyme amylase which can be produced by a variety of anaerobic or facultative organisms including *Bacillus subtilis*, *Clostridium perfringens*, *Bacillus macerans*, *Escherichia coli*, *Staphylococcus aureus*, and *Clostridium acetobutylicum*. Maltase is the product and this can pass through the cell wall into the cell where it may be further hydrolyzed to glucose by the intra-cellular enzyme maltase. Cellulose is hydrolyzed to cellobiose by the extra-cellular enzyme cellulase and the cellobiose is broken down to glucose units by the intra-cellular enzyme cellobiase. The organisms which have been reported as performing these actions are *Clostridium dissolvens*, *Clostridium thermocellum* (167) and *Clostridium omelianskii* (96). Visser (172) suggests that actinomycetes also play a part in the decomposition of the hemicelluloses under anaerobic conditions, out of which, according to the composition, various hexoses and pentoses are formed.

Once the carbohydrates are in a form capable of passing into the cell, further metabolic reactions take place to produce energy for the cell and to synthesize new cellular material. According to Oginsky and Umbreit (121) there are many pathways which can be used to decompose the mono- and disaccharides. The relative proportion that a particular pathway is used depends upon the organism and the enviromental conditions in the digester. Two compounds glucose and pyruvic acid hold key positions in carbohydrate metabolism (160, 175). Only a few bacteria are known which do not utilize glucose. The degradation of glucose in the absence of free oxygen is often known as anaerobic decomposition, glycolysis or fermentation. Pyruvic acid is an end product of glucose fermentation and acts as a link between carboydrate and protein metabolism. (Figure 2). The organisms operative in fermentation may be divided into six types. The types and names of typical organisms involved and their end products are shown in Table 1. Nickerson (120) also mentions the organisms Clostridium thermoaceticum, Lactobacillus casei and Streptococcus fecalis as being operative in glucose fermentation.

The formation of pyruvic acid from glucose and other carbohydrates is discussed in most biochemical textbooks (51,

Table 1. Organisms operative in glucose fermentation

Type of Organism	Products	Typical Organisms
Alcoholic fermentation	Alcohols	Saccharomyces cerevisiae
Simple lactic	Lactic acid	Streptococcus lactis
Mixed lactic	Lactic acid Carbon dioxide Two-carbon compounds	Lactobacillus brevis
Propionic	Lactic acid Propionic acid Acetic acid Succinic acid	Propionibacterium arabinosum
Colon-dysentery typhoid type	Lactic acid Hydrogen or formic acid, ethanol Sometimes acetoin Butylene glycol	Escherichia coli Aerobacter aerogenes Salmonella typhosa
Butyric, butyl	Carbon dioxide Hydrogen Acetic acid Butyric acid Acetate, butylene glycol Butyl alcohol Isopropyl alcohol	Clostridium saccharobutyricum Clostridium acetobutylicum Clostridium butylicum

121, 175). Simpson (160), Lackey and Hendrickson (96) and Nickerson (120) have elaborated on the scheme as it may be applied to anaerobic sludge digestion. The glucose molecule is converted to a phosphate ester, glucose-6-phosphate, which can then follow the Embden-Meyerhof pathway or the pentose phosphate pathway (121). Other pathways of carbohydrate metabolism have been demonstrated in micro-organisms such as the Entner-Doudoroff pathway and the glyoxylate pathway, but their significance is not fully understood (175). The Embden-Meyerhof scheme has been found to be operative in a variety of micro-organisms (121). Basically the scheme consists of an alteration in the ring structure of glucose-6-phosphate that yields fructose-6-phosphate. The fructose-6-phosphate enters a series of reactions in which it is broken down to two three-carbon compounds both of which can be converted to pyruvic acid. The pentose phosphate pathway has been shown to be the major pathway of carbohydrate metabolism in some micro-organisms, e.g. Leuconostoc sporogenes. Glucose-6-phosphate is oxidized to 6-phosphogluconic acid which in turn is oxidized and decarboxylated to form a pentose phosphate. The five-carbon compound is degraded to a two-carbon compound and a three-carbon compound. The three-carbon compound enters the glycolytic pathway and is converted to pyruvic acid.

Pyruvic acid can be broken down to a number of organic acids, alcohols and ketones under anaerobic conditions.

Simpson (160) cites Baldwin (9) in describing the four main pathways by which pyruvic acid is decomposed anaerobically: reduction to lactic acid; decarboxylation to a two-carbon intermediate which can eventually lead to the formation of acetic, aceto-acetic and butyric acids, ethyl, butyl and propyl alcohols and/or acetone; decarboxylation to acetaldehyde which is eventually transformed into ethyl alcohol; and a reaction which produces acetic and formic acids.

b. Lipid decomposition Lipid metabolism has not been studied to the same extent as carbohydrate and protein metabolism and much work remains to be done (175). The hydrolysis of fats by lipase enzymes yields glycerol and fatty acids. Glycerol may be converted to glyceraldehyde phosphate which is able to enter the Embden-Meyerhof pathway for further degradation to pyruvic acid. The fatty acids are believed to be decomposed aerobically by a series of reactions known collectively as Knoop's β-oxidation theory (160). The straight chain fatty acids are broken down to one-, two-, or three-carbon compounds which may be used by organisms to produce carbon dioxide and methane. Neave and Buswell (118) proposed an adaption of Knoop's theory, as a mechanism for the degradation of the higher fatty acids, to fit anaerobic conditions. Experiments with higher fatty acids than acetic acid showed a carbon dioxide production greater than that available from the carboxyl group of the fatty acid. They suggested that the extra oxygen came from water and the resulting hydrogen combined with carbon atoms of the same molecule. Lackey and Hendrickson (96) did not agree with the explanation and concluded that although fatty acids are believed to be the principle precursors of methane in sludge digestion little is known of the mechanism of their metabolism.

c. Organic nitrogen decomposition The forms of organic nitrogen in raw sewage sludge are mainly urea, whole proteins and degradation products of proteins (96). Urea is the predominant form but this is probably decomposed aerobically before the onset of anaerobic conditions. The degradation of proteinaceous material is initiated by hydrolytic reactions which reduce the size of the protein particles. The proteins are hydrolyzed by specific enzymic action yielding in the following order, in decreasing complexity; proteoses, peptones, peptides and eventually amino acids. Lackey and Hendrickson (96) state that the largest protein degradation products which can enter the cell are the peptones, which may be further degraded by intra-cellular enzymes to the amino acids.

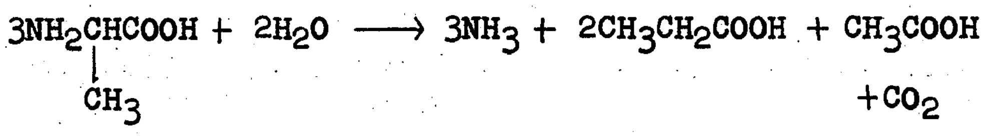
After hydrolysis of the proteins to amino acids, several types of amino acid breakdown can occur depending upon the organisms present and the environment. For example, at low pH values amino acids tend to be degraded by decarboxylation reactions yielding alkaline amines, and at high pH values deamination of amino acids yielding ammonia is more prevalent.

Gale (52) reported a pH of 4 to 5 for optimum decarboxylation and Stephenson and Gale (166) reported a pH of 7 to 8 for optimum deamination. Not all of the reactions can be performed by all bacteria and not all of the amino acids are degraded in the same fashion. For a complete study of amino acid metabolism, each amino acid must be considered separately (113). Some of the more general reactions are deamination, transamination and decarboxylation (51). Under anaerobic conditions certain members of the genus *Clostridium* are known to deaminate glycine to acetic acid.

$$CH_2NH_2COOH + 2H + H_2O \longrightarrow CH_3COOH + NH_3 + H_2O$$

The hydrogen is supplied by a concurrent oxidation of alanine.

Cardon and Barker (27) described the overall equation for the deamination and decarboxylation of alanine.



$$3NH_2\underset{\substack{|\\ CH_3}}{C}HCOOH + 2H_2O \longrightarrow 3NH_3 + 2CH_3CH_2COOH + CH_3COOH + CO_2$$

According to West and Todd (175) a very important reaction in the formation and deamination of amino acids is the transamination reaction. The amino group of one amino acid may be reversibly transferred to the keto acid of another amino acid thus effecting amino acid-keto acid interconversion. Many micro-organisms can perform the following reactions:

L-Glutamic acid + oxaloacetic acid $\rightleftharpoons$ α-Ketoglutaric acid + L-aspartic acid

L-Glutamic acid + pyruvic acid $\rightleftharpoons$ α-Ketoglutaric acid + L-alanine.

The example illustrates the importance of this reaction in enabling an organism to interrelate carbohydrate metabolism, protein metabolism and lipid metabolism. From these and other amino acid reactions end products are obtained which are similar to the breakdown products of lipids and carbohydrates.

2. Gasification

The end products of the acid producers are utilized by the methane formers to produce methane and carbon dioxide. Grune *et al.* (67) cited Omelianski, who in 1902 claimed to have isolated two organisms from the anaerobic fermentation of cellulose, one of which was capable of producing hydrogen and the other methane. In 1936 Barker (15) presented a survey of the known methane producing organisms. Until this date a pure culture of a methane producing organism had not been obtained. He developed methods for obtaining highly purified cultures of four of these organisms. Since that time four additional species of methane producing bacteria have been isolated but not in pure culture. The nine known species of methane bacteria at the present time and the substrates they utilize as listed by Barker (12) are shown in Table 2.

Table 2. The known species of methane bacteria

Methane bacteria	Substrate
Methanobacterium formicium	Formate, carbon monoxide, hydrogen
Methanobacterium omelianskii	Primary and secondary alcohols
Methanobacterium propionicum	Propionate
Methanobacterium sohngenii	Acetate and butyrate
Methanobacterium suboxydans	Butyrate, valerate and caproate
Methanococcus mazei	Acetate and butyrate
Methanococcus vanielii	Formate and hydrogen
Methanosarcina methanica	Acetate and butyrate
Methanosarcina barkerii	Methanol, acetate, aldehyde

Heukelekian and Heinemann (77, 78), following Barker's early work, developed a method of enumerating the methane organisms in sewage sludge. They also established a correlation between the number of methane organisms and the rate of gas production in the digestion of sewage sludge under a number of different conditions.

The characteristics of the methane producing organisms are discussed by Heukelekian (71). They are strictly anaerobic to the extent that, even in the absence of molecular oxygen, methane fermentation will not take place in the presence of nitrates. Sulfates retard methane fermentation at 100 mg/l sulfide concentration (4, 139). Methane producing organisms require carbon dioxide as a hydrogen acceptor, except in the case of the fermentation of acetic acid. The organisms are extremely sesitive to pH, the optimum range being 6.4 to 7.2. Levels of pH below 6.0 and above 8.0 cause the rate of growth to fall off rapidly. Heukelekian and Heinemann (78) found more methane producing organisms in the sludge than in the supernatant. The methane producing organisms are believed to develop slowly and they have a long generation time.

McCarty (103) in 1963 discussed the chemistry of methane fermentation summarizing the existing knowledge on the subject. He states that methane results from two major sources, as shown in the equations:

Carbon dioxide reduction $C^{14}O_2 + 8H \longrightarrow C^{14}H_4 + 2H_2O$

Acetic acid fermentation $C^{14}H_3COOH \longrightarrow C^{14}H_4 + CO_2$

A third source which appears to be of relatively minor importance is the reduction of methanol as reported by Stadtman and Barker (164) and confirmed by Pine and Vishniac (126). Pine and Vishniac suggested that a common intermediate was involved in fermentation of methanol and acetate to methane. Omelianski as cited by Grune _et al._ (67) suggested a preliminary decomposition of acetic acid to hydrogen and carbon dioxide to methane by the hydrogen. Buswell (18) doubted the validity of this mechanism because hydrogen is only formed in a digester in barely detectable quantities.

The reduction of carbon dioxide to methane was proposed by Barker (13) in 1936 based on the results of experiments with ethyl and butyl alcohol. Carbon dioxide acts as a hydrogen acceptor and is reduced to methane by hydrogen enzymatically removed from the organic molecules. He formulated a general equation for the process:

$$4H_2A + CO_2 \longrightarrow 4A + CH_4 + 2H_2O$$

where H_2A represents any organic compound which can be activated by the methane producing bacteria so that it can act as a hydrogen donor for the reduction of carbon dioxide.

The fermentation of acetic acid results in the production of methane and carbon dioxide by a split in the acetic acid molecule. Methane comes from the methyl group and the carbon dioxide comes from the carboxyl group. Stadtman and Barker (164) used radioactive tracers to show this occurrence. Buswell and Sollo (24) reasoned that methane originates primarily by a simple decarboxylation of acetic acid. The above theory was supported by the preliminary investigations of Grune <u>et</u> <u>al</u>. (67) on the mechanism of methane fermentation. They labeled sodium acetate with C^{14} in the one and two carbon positions separately and fed the labeled compounds to a digester. The products showed that most of the carbon dioxide is derived from the carboxyl group directly without going to methane and that some of the carboxyl group is decomposed by a slower, longer pathway to form methane directly.

The importance of each mechanism in the fermentation of complex materials has been demonstrated by Stadtman and Barker (161) and Barker (14) with reference to the overall fermentation of ethyl alcohol.

$$\begin{array}{lll} 2CH_3CH_2OH + 2H_2O & \longrightarrow & 2CH_3COOH + 8H \\ 8H + CO_2 & \longrightarrow & CH_4 + 2H_2O \\ 2CH_3COOH & \longrightarrow & 2CH_4 + 2CO_2 \\ \hline \end{array}$$

$$\text{TOTAL} \quad 2CH_3CH_2OH \longrightarrow 3CH_4 + CO_2$$

The equations show that 67 percent of the methane results from acetic acid fermentation and 33 percent results from the reduction of carbon dioxide.

Even carbon fatty acids such as acetic acid are believed to be fermented in the above manner. The formation of methane from propionic acid, an odd carbon fatty acid, has been shown by Stadtman and Barker (162) to proceed in the following way:

$$\begin{array}{lll} 4CH_3CH_2COOH + 8H_2O & \longrightarrow & 4CH_3COOH + 4CO_2 + 24H \\ 3CO_2 + 24H & \longrightarrow & 3CH_4 + 6H_2O \\ \hline \end{array}$$

$$\text{TOTAL} \quad 4CH_3CH_2COOH + 2H_2O \longrightarrow 4CH_3COOH + CO_2 + 3CH_4$$

The tracer experiments of Stadtman and Barker indicated that the number two and three carbons of propionic acid ended up as the carboxyl and methyl carbons respectively, of acetic acid. Buswell et al. (21) did not agree with these results, but their differing results may have been due to biochemical side reactions. McCarty et al. (105) gave equations to represent the fermentation and gas production from four volatile acid salts.

	Moles gas per mole acid
Formate	
$HCOONa + 1/2H_2O + 1/4CO_2 \longrightarrow NaHCO_3 + 1/4CH_4$	0
Acetate	
$CH_3COONa + H_2O \longrightarrow NaHCO_3 + CH_4$	1
Propionate	
$CH_3CH_2COONa + 1/2H_2O \longrightarrow CH_3COONa + 3/4CH_4 + 1/4CO_2$	
$CH_3COONa + H_2O \longrightarrow NaHCO_3 + CH_4$	2
Butyrate	
$CH_3CH_2CH_2COONa + NaHCO_3 \longrightarrow 2CH_3COONa + 1/2CH_4 + 1/2CO_2$	
$2CH_3COONa + 2H_2O \longrightarrow NaHCO_3 + 2CH_4$	3

Formate and acetate are fermented directly while propionate and butyrate are both first converted to acetic acid which is then converted to methane and carbon dioxide giving two stage reactions. The relative gas production per mole of each acid salt is also shown.

Gases other than carbon dioxide are formed in anaerobic digestion. Rudolfs and Heisig (140) and Buswell and Hatfield

(22) reported the production of small amounts of hydrogen. Buswell and Hatfield concluded that it was the nature of the compound fermented and not the culture which determines the production of hydrogen. In 1930 Buswell and Neave (123) showed that <u>Escherichia</u> <u>coli</u> ferments glucose to produce hydrogen and carbon dioxide. Rudolfs and Heisig (140) have also reported the presence of small quantities of carbon monoxide in the digestion of screenings.

According to Heukelekian (71) a small amount of nitrogen is produced in a digester. There is no known fermentation process that can produce nitrogen gas directly from proteins or amino acids and thus Buswell and Strickhouser (25) put forward the explanation of the dissolution of nitrogen gas due to changes in partial pressures present in the fresh solids.

Hydrogen sulfide is present as the result of the reduction of sulfates and sulfur in organic combination.

C. Digester Operation

Fair and Moore in 1934 (44) summarized the work of many authors regarding the effect of temperature on the time and rate of sludge digestion. They discussed the results of work by Heukelekian (72a, 73), Keefer and Kratz (90), Rudolfs (141), Zack and Edwards (178) and others, and supplemented these results with data of their own. All results were obtained from experiments with batch digestion and the digestion time was the time required to produce 90 percent of the total gas produced during the run[1]. Optimum temperatures for the maximum rate of digestion (shortest detention time) were observed to be 28°C in the mesophilic zone and 42°C in the thermophilic zone. Temperature changes of a few degrees from the optimum adversely affected the rate of digestion in the thermophilic zone to a much larger extent than in the mesophilic zone. The authors believed that two different groups of organisms were operative in digestion and each group had an optimum temperature requirement. Between the mesophilic and thermophilic zones, both groups of organisms were working at a disadvantage. Fair and Moore (42) concluded that activated sludge may not digest in a manner similar to primary tank sludge since the digestion rate with activated sludge increased with temperature from 25°C to 50°C. The authors mentioned that data were not available for determining the effect of temperature on the digestion of a single type of sludge. At a later date (43), they obtained these data which also showed that digestion rate seemed to pass through two optimums which were related to temperature. The optimum temperatures for mesophilic and thermophilic digestion were 37°C and 53°C, respectively. The times of digestion at these temperatures

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[1] Gas productions are measured usually in relationship to the total or volatile solids added to the digester, and are expressed as cu ft of gas produced per day per lb of total or volatile solids added to the digester (cu ft per lb volatile or total solids added per day).

were 22 days and 8 days, respectively. Heukelekian partially disagreed with the findings of Fair and Moore. He observed (72) that the time of digestion did not materially alter between 28°C and 42°C. Malina (100) studied the effect of temperature on the digestion process at 32.5°C, 42.5°C and 52.5°C. He observed the effect of temperature to be independent of loading rate and detention time. A reduction in volatile material and an increase in alkalinity and volatile acids occurred as the temperature increased. The digestion process was most inefficient, as measured by gas production, gas quality and pH, at 42.5°C thus supporting Fair and Moore's (44) observations. At 52.5°C most of the digestion parameters indicated that the digestion process was inferior to digestion at 32.5°C although more volatile matter was destroyed at 52.5°C. Many experimenters (46, 73, 138, 142, 143) have compared the thermophilic and mesophilic digestion processes. Thermophilic digestion can operate at higher organic loading rates, it produces a more concentrated sludge, a larger gas yield is obtained and a greater reduction in volatile solids is observed. The disadvantages of the thermophilic process are: in a single stage system a poorer supernatant is produced the digested sludge is more difficult to dewater; disagreeable odors are a more frequent occurrence; micro-organisms in the process are more susceptible to the effect of toxic materials; and the economics of the process may not be favorable in many instances. Rudolfs and Heukelekian in 1930 (141) indicated that the heating of a digester to a temperature of approximately 50°C would be uneconomical. This would depend upon climatic features.

In 1948, Heukelekian and Kaplovsky (79), obtained results from studies of the effect of temperature changes on thermophilic digestion at 50°C. The results showed that with active thermophilic digestion a decrease in digestion rate would occur if the temperature were decreased. The rate was found to return to normal values if the temperature was again increased. Garber in 1954 (53) compared the digestion of sludge at 30-38°C, with the digestion of sludge at 50°C. He found no significant difference between the processes at 30°C and 38°C but at 50°C the digestion process was much more rapid. The thermophilic process was stable and it produced a more readily dewaterable sludge than did the mesophilic process. The latter finding contradicts previous ideas of thermophilic digestion. Golueke (56) confirmed Garbers results. He attempted to show that the rate of digestion did not remain constant or decrease between the mesophilic and thermophilic zones. Sludge was digested at temperatures ranging from 30°C to 65°C at 5°C intervals. Within the temperature range 35°C to 60°C, no significant difference in gas production, volatile solids reduction and dewatering characteristics could be noticed. The digested sludges from the 50, 55 and 60°C digesters were of a superior quality to those from the remaining digesters. Volatile acids and pH both increased with temperature.

The pH of digesting sludge has been shown to affect the digestion process. Cassell and Sawyer (31), Schlenz (156), Coulter (35) and others recommend a pH of 6.8 to 7.2 or close to these values for optimum digestion. Stadtman and Barker (164) have shown that certain methane forming bacteria have optimum pH values where they grow most rapidly between 8.0 and 9.0. The use of pH as an indicator of the condition of digestion has been superseded in many instances in recent years, by the level of volatile acids in the digester because by the time a change in pH is noticeable the buffering capacity of the sludge has been virtually destroyed. The volatile acids test is more sensitive, but pH is still used as a control, especially in starting digesters.

The major intermediate compounds formed by the acid producing organisms in the digestion of sewage sludge are the short chain volatile organic acids[1], more often called the "volatile acids." They are formed from the decomposition of complex organic compounds and they serve as the major substrate for the methane producing bacteria (104). The obvious importance of the volatile acids in sludge digestion suggested to many workers (11, 22, 148) that a knowledge of the volatile

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[1] Formic, acetic, propionic, butyric, etc. up to a 6 carbon chain.

acids present in digesting sludge would provide better control for the process than relying on pH values. It was noticed that an increase in the volatile acid concentration in digesting sludge heralded the onset of poorer digestion. This occurred before a drop in pH, and if the volatile acid concentration eventually rose above 2000 to 3000 mg/l, the methane producing organisms were seriously retarded. Two theories have developed regarding the effect of high concentrations of volatile acids on the digestion process:

1. The volatile acids are indirectly toxic to the methane producing organisms by lowering the pH of the digesting sludge.
2. Volatile acid concentrations above a certain level, usually 2000 to 3000 mg/l, inhibit the methane producers regardless of the pH.

In support of the first theory, Golueke et al. (57) concluded from experiments on the anaerobic digestion of algae that high volatile acid concentrations exhibit an inhibiting effect due to a lowering of the pH. Cassell and Sawyer (31) showed that maintenance of an optimum pH is the factor of primary practical importance in initiating digestion. Kaplovsky (87) and Sawyer et al. (149) also support the theory that any detrimental effects due to high volatile acid concentrations can be overcome by maintaining optimum pH levels with lime or some other chemical. The experimenters who supported the second theory believed the acids were directly toxic to the methane producing organisms and that the inhibiting action could only be relieved by reducing the volatile acid concentration. This view was suggested by Buswell and Hatfield (22) in 1936 and has been supported by Schulze and Raju (158) and Mueller *et al.* (117) in recent years.

McCarty and McKinney (108) in 1961 undertook a study to determine the fundamental effect of volatile acids on methane formation in anaerobic digestion and to resolve the apparent difference between the two theories presented above. The results of the study showed that the inhibition of methane bacteria associated with an increase in volatile acid concentrations was not due to volatile acid toxicity but was due in a more general way to "salt" toxicity. The difference is that salt toxicity depends upon the types and concentrations of the cations of the volatile acid salts rather than upon the anions as in volatile acid toxicity. Using this concept as a basis, the authors proceeded to explain that relatively high concentrations of volatile acids can be tolerated provided they are associated with cations of low toxicity. The hydrogen ion is known to be one of the most toxic cations to biological systems. A drop in pH would inhibit the methane producing organisms because of the increased hydrogen ion concentration. If an alkaline material is added to adjust a low pH in a system, no benefits will be obtained unless the cation employed is less toxic than the hydrogen ion. The work of Keefer and

Urtes (92, 93) supports the salt toxicity theory. Volatile acid concentrations as high as 13,000 mg/l were neutralized with lime and active digestion continued. The conclusions which can be drawn from the work of McCarty and McKinney (108) and Keefer and Urtes (92, 93) are:

1. The low pH values associated with high concentrations of volatile acids do inhibit the digestion process. Control of pH is essential in the operation of a digester.
2. The anionic portions of the volatile acids are not directly toxic to the methane producing organisms in the concentrations suggested in the second theory.
3. Consideration must be given to the cationic portions of the volatile acids when studies are made of volatile acid toxicity.

McCarty and McKinney (107) followed up the salt toxicity theory with a study to determine the effect of neutralizing volatile acids with alkalies containing different cations. The cations in order of increasing toxicity were calcium, magnesium, sodium, potassium and ammonium. Certain ion antagonism effects were noticed and these will be discussed later. Sodium, potassium and ammonium compounds were found to be suitable for neutralization of volatile acids concentrations up to 2000 mg/l but from 2000 to 10,000 mg/l, calcium and magnesium compounds were preferable. McKinney suggested that if the volatile acids in a digesting sludge are neutralized with magnesium compounds, the magnesium will remain in the sludge as soluble magnesium bicarbonate when balanced conditions are restored. The increased bicarbonate concentration will result in an increase in pH beyond acceptable values which might slow down the rate of digestion. If calcium compounds are used, the calcium will precipitate from solution.

The effect of high concentrations of individual volatile acids on anaerobic treatment has been discussed by McCarty and Brosseau (104). It was concluded that high concentrations of acetic, propionic or butyric acids do not affect the methane bacteria. Sudden increases of acetic acid and butyric acid up to 6000 mg/l were stimulatory to the process. Propionic acid concentrations up to 8000 mg/l caused an initial inhibition of the digestion process which could be overcome by a short period of acclimatization. It was shown that the propionic acid affected the acid producing organisms. The authors introduced concepts of "temporary" and "permanent" causes of unbalance in a digester. The temporary causes include an insufficient population of methane producing organisms as may occur in digester start-up, increased organic loadings, ineffective mixing and sudden temperature changes. Temporary causes can be removed by maintaining a neutral pH and allowing sufficient time for the methane forming organisms to re-establish themselves. The permanent causes of digestion inhibition may be the presence of toxic materials or materials which may not be toxic in the concentrations normally experienced in domestic sewage sludge but which may be toxic in high concentrations. Permanent causes may only be eliminated by removing the toxic material or reducing its concentration on to a non-toxic level.

The volatile acids normally present in digesting sewage sludge under different operating conditions have been reported in the literature. Liubimov and Kagan (98) state that during the acid fermentation stage in mesophilic digestion caproic, propionic, butyric, isovaleric, acetic and formic acids were present. When normal alkaline digestion was established, butyric, isovaleric and propionic acids disappeared. Hindin and Dunstan (81) and Mueller *et al*. (117) found that acetic and propionic acids were the major acids present at times of high volatile acid concentrations. Butyric acid was found in lower concentrations and formic and lactic acid were found only in low concentrations. Kaplovsky (86) observed the presence of acetic, propionic, butyric and valeric acids in the digestion of yeast and slaughterhouse wastes. He found only acetic and butyric acids in the digestion of white water. Pohland and Bloodgood (127) studied the mesophilic and thermophilic digestion processes and concluded that acetic and propionic acids were the major acids present in both processes. They concluded that acetic acid was the most important volatile acid intermediate in sludge digestion and the primary source of gas production. McCarty _et_ _al_. (105) found during start up of a digester that the principal volatile acids present were butyric and acetic acids. If the process deteriorates after active fermentation has started, the major acids produced are acetic acid and propionic acid. They studied the formation of volatile acids during digestion by adding individual acids to laboratory digesters and also by developing sludges using synthetic substrates. The synthetic substrates were composed of long chain fatty acids, carbohydrates of different degrees of complexity, proteinaceous materials and mixtures of complex organic compounds. The results indicated that acetic acid is the most prevalent intermediate volatile acid produced in the decomposition of all the above substrate types. The major volatile acids obtained from the digestion of carbohydrates were propionic and acetic acid; from proteins, they were propionic, butyric and acetic acid; and from fats, they were acetic and butyric acids. Again, the major acids found during the occurrence of unbalanced conditions were acetic and propionic acids. Formic and butyric acids were found to be metabolized easily during the digestion process but the authors thought that butyric acid does not occur as a true intermediate volatile acid. It may be synthesized by _Clostridia_ or similar organisms prior to methane fermentation.

Schulze and Raju (158) showed with experimental digesters that maximum feed rates of 0.2 g of propionic acid or 1.0 g of acetic acid or 1.6 g of butyric acid per liter of digester volume per day could be quantitatively converted to gas. Keefer and Urtes (92, 93) disagreed with these maximum feed rates, suggesting maximum rates of 0.13, 0.31 and 0.18 g per day per liter for propionic, acetic and butyric acids, respectively. They did agree that propionic acid was the most difficult to convert to gas. These authors also showed that the digestibility of the three acids varied directly with their ionization constants.

Many studies have been made of the toxic effects of heavy metals on the anaerobic digestion process. The results show a high degree of variance depending upon the particular study. Copper has been shown (135, 136, 138) to be inhibitive to the digestion process at concentrations above 0.07 percent of the dry solids. However, some digestion units have been able to withstand copper concentrations of 1.5 percent of the dry solids content without noticeable effects (32, 110).

Nickel has been reported by Wischmeyer and Chapman (176) to stimulate digestion in concentrations up to 500 mg/l and to retard digestion above this concentration.

McDermott _et_ _al_. (109) evaluated the effect of zinc on sludge digestion and found that concentrations above 340 mg/l in the digested sludge produced toxic effects.

The effect of iron compounds on the sludge digestion process has been reported by Rudolfs et al. (144) and Setter (159). Concentrations ranging from 100 mg/l to 1000 mg/l have caused retardation of the digestion process.

Pagano (122) discussed the ability of the digestion process to handle successfully 50 to 200 mg/l of trivalent chromium on a slug-feed basis. On a continuous feed basis, 3 to 6 mg/l of trivalent chromium and 1.25 to 1.5 mg/l of hexavalent chromium could be tolerated. Ross (32) supported these results showing that hexavalent chromium was more toxic than trivalent chromium.

McCarty et al. (106) studied the effects of copper, zinc nickel, and iron singly, and in combination, on anaerobic digestion. Copper, zinc and nickel were found to be toxic to anaerobic digestion but high concentrations of iron could be tolerated on a daily feed basis. The sum total concentration of all the heavy metals was determined to be the significant factor in evaluating heavy metal toxicity. Masselli et al. as cited by McCarty et al. (106) showed the toxicity of copper to depend upon the sulfide concentration in the digester because of precipitation of copper sulfide. McCarty et al. (106) found that toxicity due to heavy metals will only result when the sum total concentration of heavy metals exceeds the equivalent concentration of sulfides for precipitation. The authors suggested the addition of ferrous sulfate to a digester as a control or precautionary method against the effect of toxic heavy metals. Iron is not toxic to the organisms in digesting sludge except in very high concentrations. Inside the digester ferrous sulfate is reduced to ferrous sulfide which is more soluble than the sulfides of copper, lead, cobalt, nickel and zinc. Thus, if a toxic heavy metal is added to a digesting sludge which contains ferrous sulfide, the sulfide of the toxic heavy metal will be precipitated. As a precipitate the toxic heavy metal cannot exert a toxic effect on the organisms in the sludge. Care should be exercised not to achieve excessive concentrations of sulfides because they are toxic themselves to the methane bacteria in concentrations greater than 200 mg/l as sulfur and they are corrosive. Experiments have shown that the addition of sulfide precursors has allowed the presence of more than 10 percent of heavy metals, on a dried volatile solids basis, without inhibiting the digestion process.

Between 1960 and 1964 McCarty _et al_. (106) determined the effects of different ions on the methane fermentation stage in the anaerobic waste treatment process. A synthetic medium was used to maintain the bacterial cultures, and only acetic acid, its salts, and the various cations in the form of their chloride salts were fed to the cultures. The conclusion reached by the experimenters was that the cation effects are a function of the types and concentrations of all the cations present in the environment. Optimum ionic concentrations of sodium, potassium, ammonium, calcium and magnesium were found and concentrations either lower or higher than the optimum resulted in less than maximum efficiency of the methane fermentation process. Synergistic and antagonistic effects were noticed. Inhibition by one cation could be increased, synergism, by adding certain other cations, even though the concentration of the synergistic cation was well below its own inhibitory level. However, inhibition due to an excessive concentration of any one of the ions studied could be antagonized (minimized) by the addition of the optimum concentration of at least one of the other four cations. Maximum antagonism was attained with addition of the optimum concentrations of several other cations.

The increasing use of radioactive isotopes in science and industry has stimulated the study of their effects on waste treatment processes. Dietz and Harmeson (37) used batch and continuous sludge digestion units to determine the effects of three radioactive isotopes on the two processes and to determine the concentration of the isotopes in the solid and liquid portions of the sludge. Control experiments were run using the non radioactive forms of the isotopes. The results of the batch tests are shown in Table 3.

Table 3. Effect of radioactive materials on anaerobic digestion.

Radioactive material added to provide 200 millicuries per liter	Effect on digestion as measured by reduction in gas production
P^{32}	15 percent
I^{131}	4 percent
S^{35}	50 percent
Mixed fission products	20 to 40 percent

Results obtained with the continuous process showed no significant effect of P^{32}, I^{131} and S^{35} at the 110 microcurie per liter level of activity. The fission products caused a 17 percent reduction in gas production over a 30 day period. P^{32}, S^{35} and the fission products tended to concentrate in the sludge in the continuous process. Radioactive iodine did not concentrate in the sludge. Grune _et al_. studied the effect of radiophosphorus (62) and radioiodine (63) on batch sludge digestion. They found no significant effect on the digestion process due to the radioactivity of the P^{32} up to concentrations of 100 millicuries per liter. The uptake of P^{32} by the solid component of the sludge increased exponentially from 12 to 18 percent with an increase of the initial radioactivity concentration from 50 to 800 millicuries per liter. The percentage removed in the liquid phase also increased with an increase in the initial radioactivity concentration. No significant changes were noticed in the uptake of I^{131} with increases in the initial radioactivity up to 100 millicuries per liter. At least 60 percent of the activity appeared to be concentrated in the solid component. In 1963, Grune et al. (67) extended their work on radioactivity and digestion. The effects on anaerobic digestion of C^{14} and Sr^{90} in concentrations up to one millicurie per liter and the distribution of these isotopes between the solid and liquid phases were determined. Batch digestion results indicated no significant effects of C^{14} or Sr^{90} on gas production and quality, rate of digestion, volatile acids, lag phase period and reduction of volatile matter. Both isotopes were found to concentrate in the solid component of the sludge.

In the past decade, the term high rate sludge digestion has been increasingly used to indicate a digestion system which is heated, which is uniformly mixed, i.e., no supernatant liquor, and in which feeding is on an intermittent or continuous basis with a corresponding displacement of "mixed liquor" rather than supernatant or sludge. Secondary units may or may not be used in the system. According to Sawyer (148) four major factors separate high rate digestion units from conventional digestion units. The factors are all dependent upon complete mixing of the contents of the high rate digester: thermal homegeneity; biological balance, or the elimination of the predominance of the acid forming organisms in a scum layer and a predominance of methane producing bacteria in the sludge layer below; the ability to bring fresh food into contact with the active organisms; the elimination of local high concentration of solids, e.g., in the scum layer, which have been shown to be detrimental to the activities of the functional organisms in digestion.

The loading rates of so-called conventional digestion units and high rate digestion units are not separated by any well defined line of demarcation. Pearse (123) in 1938 cited Greeley as giving the volatile solids loading rate[1] for sludge digestion tanks as 0.047 to 0.07 lb added per cu ft per day for complete digestion and 0.105 lb added per cu ft per day for partial digestion. He reported the average loading of the digesters at a number of plants as 0.041 lb volatile solids added per cu ft per day. Digestion units operated in this range of loading rates are often said to be operated at conventional rates. Imhoff _et al_. (84) in 1956 suggested a conventional volatile solids loading rate of 0.08 lb added per cu ft per day and Steel (165) suggested 0.02 to 0.06 lb added

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[1] The term volatile solids loading rate means the amount of volatile solids fed to a digester. It is expressed in terms of the pounds of volatile solids added to a digester per day divided by the volume of digesting sludge in cu ft. Often synonyms of volatile solids loading rate will be used, such as organic loading rate or just loading rate, expressed as lb volatile solids added per cu ft per day or lb per cu ft per day.

per cu ft per day. The Ten States Standards (59) recommends loading rates of 0.02 to 0.04 lb volatile solids added per cu ft per day, or greater than 50 days detention time for conventional digestion. Babbitt and Baumann (6) indicate that conservative designs use values of 0.04 lb volatile solids added per cu ft per day and less, or more than 30 days detention time. The trend in large plants is towards loading digesters with 0.08 to 0.21 lb volatile solids added per cu ft per day or 10 to 15 days detention time.

The method of referring to digester loading by quoting the additions of solids on a dry or volatile solids basis was questioned by Rankin (130a). He noticed that detention time[1] appeared to bear a closer relation to performance as measured by volatile matter reduction. Sawyer (148) and Sawyer and Schmidt (152) agreed with this observation.

In 1954 Morgan (116) attempted to increase the rate of sludge digestion above conventional rates by recirculating digester gas to mix the digesting sludge. He found it possible to digest sludge at a loading rate of 0.345 lb volatile solids added per cu ft per day with a sludge detention time of 7.2 days. Also in 1954 Torpey (168) succeeded in feeding concentrated primary and activated sludge to plant scale sludge

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[1] Detention time is the theoretical time a sludge particle remains in a digester. It is based upon raw sludge additions to the digester and the removal of digested sludge from the digester.

digesters at a rate of 0.19 lb volatile solids added per cu ft per day with a detention time of 11 days. The contents of the primary digesters were mixed by recirculating digesting sludge by pumping it from the bottom of the digester and putting it back in at the top. At a later date (169) he loaded a pilot scale digester almost continuously, without failure, at a loading rate of 0.87 lb volatile solids added per cu ft per day and 3.2 days detention time. Sawyer and Roy (151) reported operating laboratory digestion units at detention periods of 6 to 20 days with volatile solids loadings of 0.38 lb to 0.26 lb volatile solids added per cu ft per day respectively. Sawyer and Schmidt (152) successfully operated digestion units with 11 days detention time and a solids loading rate of 0.34 lb volatile solids added per cu ft per day. Garrison et al. (54) state that a minimum detention time of eight days is required in practice to produce a well digested sludge. These authors succeeded in loading plant digesters at a rate of 0.43 lb volatile solids added per cu ft per day.

The effects of the increased loading rates on the end products of digestion have been reported. Buswell and Boruff (20) state that the maximum volume of gas which can be generated in digestion is 8 to 9 cu ft per lb of volatile solids added to a digester and slightly more than double these values per lb of volatile solids destroyed in a digester. Higher gas productions ranging up to 11.0 cu ft per lb of volatile solids added and 24 cu ft per lb of volatile solids destroyed have been reported (160, 151, 168). The quality of the gas produced is an indicator of the state of digestion; good digestion being characterized by a gas made up of approximately 60 to 70 percent methane and 30 to 40 percent carbon dioxide (171). Other gases are present in small amounts. As the digestion process deteriorates, the carbon dioxide content of the gas increases (7). Several authorities have agreed that the optimum pH for good digestion is close to 7.0. Volatile acids concentrations which are increasing, or are above about 2000 mg/l, usually indicate the onset of inferior digestion. If the volatile acids concentration in a digester is steady and below an inhibitory level, then the process is probably functioning satisfactorily. The generally accepted value of sludge alkalinity for good digestion is between 2000 and 3000 mg/l. Volatile solids reductions in the neighborhood of 35 to 65 percent are expected in a healthy digester.

Morgan (116) obtained normal gas productions of 9.0 cu ft per lb of volatile solids added at higher loading rates. Reductions in volatile matter destruction, gas production, grease destruction, alkalinity and pH were noticed by Sawyer and Roy (151) and Sawyer and Schmidt (152) as the detention time was decreased from 20 days to six days. Mueller _et al_. (117) maintained a constant detention time of 33 days and varied the loading rates to a digester from 0.05 to 0.135 lb volatile solids added per cu ft per day. At the increased loading rates, they noticed an increase in volatile acids, alkalinity, suspended solids in the supernatant, percentage of carbon dioxide in the gas produced and the volatile solids content of the digested sludge. Gas production and volatile solids reduction both decreased.

Garber (53) noticed in experiments on thermophilic digestion that the particle size of sludge which had been digested at 85°F to 100°F differed from the particle size of sludge digested at 120°F. About 80 percent of the sludge which was digested at 85°F to 100°F passed a 200 mesh screen but only 65 percent of the 120°F sludge passed the same screen. Balmat (10) reported that the smaller the particle size of sewage sludge the faster the rate of decomposition. These experiments suggested to Pierce (125) the possibility of increasing digestion rates by reducing the particle size of raw sewage sludge prior to digestion. Pierce observed a higher volatile acids concentration and a higher rate of sludge digestion with comminuted sludge as compared to the digestion of uncomminuted sludge. The gas quality and pH remained unchanged. Edmonds (40) agreed with the results of Pierce and in addition obtained higher gas production rates with comminuted sludge.

The concentration of raw sludge prior to digestion is practiced to some extent in practically all sewage treatment plants whether it is intentional or not. Many benefits are obtained by concentration. Less digester capacity is required, there is less sludge to heat when using heated digesters, and for a fixed loading rate the detention time of the solids in the system is increased.

Keefer (88) in 1947 recognized these advantages and attempted to digest sewage sludge on a batch basis at total solids concentrations of 5.3, 15.0, 25.0 and 32.5 percent. The sludge was thickened by adding ferric chloride in concentrations up to 0.24 percent to a mixture of three parts raw sludge to two parts digested sludge. A control digester was set up containing the sludge mixture at 5.2 percent total solids, to which no ferric chloride had been added. The results obtained indicated that the digestion rate of sludge containing ferric chloride was retarded. Increasing the solids to 32.5 percent also retarded digestion. In 1951 Torpey (168) at the New York Bowery Bay sewage treatment plant was faced with the problem of having insufficient digester capacity to digest the sludge produced in the plant. To solve the problem, he thickened the sludge from the primary tanks and a modified aeration process to 11.2 percent (170), reducing the volume to such an extent that only a small fraction of the digester plant capacity was then required for digestion Loadings as high as 0.123 to 0.19 lb volatile solids added per cu ft per day at a detention time of 31 days were achieved. After standing without feeding for 70 days in a secondary digester, the supernatant contained 1.4 percent solids while the concentration of settling sludge increased from 5.4 to 8.5

percent. In 1953 the secondary treatment process in the plant was changed from modified aeration to step aeration activated sludge. It was found possible to thicken the combined primary and activated sludge to 6 percent total solids. A single digester was loaded with thickened sludge at rates of 0.157 to 0.193 lb volatile solids added per cu ft per day at a detention time of 15 days. Torpey mentioned that certain precautions should be taken when digesting sludge at high concentrations and high loading rates. The system should be well mixed, the sludge should be fed almost continuously and, when starting the digesters, the load should be increased gradually over slightly longer periods of time than are usually required.

A comment on the foregoing work regarding the volatile matter remaining in the digested sludge is necessary. When the raw sludge volatile solids content was approximately 79 percent, the volatile solids content of the digested sludge was approximately 60 percent giving a reduction of only 24 percent. According to a graph of percent volatile solids in raw sludge versus percent reduction of original volatile matter when sludge is considered to be digested, published by Schlenz (153b), a reduction of 83 percent would be expected. However, at the Bowery Bay Plant digested sludge is barged to sea for ultimate disposal. A plant which dries digested sludge on sand beds may find it necessary to reduce the volatile matter to a greater degree than that obtained by Torpey.

Rankin (130b) discussed the foregoing article and suggested that a disadvantage of continuous feeding is the need for the continuous disposal of sewage sludge. To overcome this problem, sludge prethickening and a sludge holding tank could be used. A distinct advantage of the prethickening step is the elimination of the digester supernatant return to the plant, a practice that frequently has detrimental effects on treatment results. However, Kraus (95) in 1945 used digested sludge and supernatant liquor to successfully control the bulking of sludge in the activated sludge process at Peoria, Illinois. As a continuation and extension of his studies of feeding thickened sludge at high loading rates to plant scale digesters, Torpey (169) in 1953 put into operation a six cubic foot capacity pilot high rate digester. Thickened sludge at approximately 5.3 percent total solids and 75 percent volatile solids was fed to the pilot digester at increasing loading rates until the digestion process failed. Failure occurred at detention times less than 3.2 days and at loading rates greater than 0.87 lb volatile solids added per cu ft per day. At this critical condition, the gas production was 6.8 cu ft per day per lb of volatile solids added, the volatile acid concentration was 1700 mg/l and the volatile solids content of the digested sludge was 63.1 percent. As the loading rate increased the volatile solids content of the digested sludge and the volatile acids concentration of the digester liquor increased slowly, until a detention time of five days was reached. Below that detention time, there was a rapid increase in the volatile solids and volatile acids content of the sludge. The plant digester was fed similar sludge at a rate of 0.18 lb volatile solids added per cu ft per day with a detention time of 13.3 days. The digested sludge had a volatile solids content of 62.7 percent and the gas production was 8.5 cu ft per day per lb of volatile solids added. At equivalent loading rates, the data appeared to show that the pilot plant was slightly more efficient in gas production and volatile solids reduction.

Based upon a comparison of the efficiency and rate of digestion of the pilot plant and the plant digester Torpey concluded that the plant loading rate could be tripled if the total volume of the plant digester could be effectively utilized. He recommended digestion capacity of 0.4 cu ft per capita in the design of digesters as opposed to 2.0 to 6.0 cu ft per capita as was common practice at that time (86).

Schlenz (153b) discussed Torpey's work and was critical of the possible interpretation of the results. He showed the plant digester to be only 90 percent as efficient as the pilot digester after taking into account the variability of the feed to the plant digester and the uniform feed to the pilot digester, the comparison of data for the same time periods and the reduction in volatile solids. Schlenz showed an unfavorable comparison between volatile solids reductions obtained by Torpey and expected reductions as based on the experiences at many sewage treatment plants. He disagreed with the low values for detention time recommended by Torpey for design purposes and presented data which showed a possible raw sludge production for a five day period up to 240 percent of the average raw sludge production.

Heukelekian (72b) also discussed Torpey's work and stated that the success obtained in operating the pilot digester at such a high loading rate could be attributed to sludge thickening, effective utilization of tank volume and uniform feeding of raw sludge. The reason for failure may have been related to the generation time of the organisms in the digesting sludge.

In 1955 (152) Sawyer and Schmidt studied the digestion of sewage sludge at total solids concentrations ranging from 1.35 to 5.20 percent. They found that the most important consideration in operating high rate digestion units was detention time. The volatile solids destruction, gas production, pH and alkalinity declined with decreasing detention times. Satisfactory operation was achieved with detention times as short as 11 days and loading rates of 0.48 lb volatile solids added per cu ft per day. The reduction in volatile matter at this loading rate was approximately 54 percent and the gas production was 9.0 cu ft per day per lb of volatile solids added.

An attempt to digest sludge on a batch basis at solids concentrations of 10, 20, 30 and 50 percent was made by Schulze (157) in 1958. The digestion process was retarded with 10 percent solids and stuck digestion occurred after two days with the higher sludge concentrations. The cause of retardation and stuck digestion was thought to be the extremely high values of volatile acids concentrations obtained in the experiment i.e. 25,000 mg/l with the 30 percent solids and 55,000 mg/l with 50 percent solids. The pH values remained above 6.0 except in the case of the digester containing the 50 percent solids. This experiment shows that the acid forming organisms can function in the presence of high concentrations of volatile acids. Successful digestion was eventually obtained with a sludge solids concentration of 37 percent. A normal rate of gas production was obtained, as measured by a control digester of actively digesting sludge. Schulze concluded from the studies that feed rate is the most important parameter in maintaining good digestion. In 1963, Buzzell and Sawyer (26) while attempting to determine the cause of digester failure, digested sewage sludge at 4, 6, 8 and 10 percent total solids in the feed sludge. The digesters were operated successfully with a feed sludge having a volatile solids content of 62 to 86 percent and with a detention time of 10 days. The decision of the author to study the effect of solids concentration on the digestion process was based on the results of the foregoing works. It was felt that a more thorough study covering a wider range of solids concentrations was needed.

Sawyer and Grumbling (150) reported an association between digester upset and mixing difficulties. Buzzell and Sawyer (26) noticed in experiments where the solids concentrations fed to digesters ranged from 4 to 10 percent that gas bubbles tended to become trapped in the units receiving 6, 8 and 10 percent solids feed. Mixing under this condition was inefficient. These observations suggested to Buzzell and Sawyer the necessity of a study to determine the relationship between the sludge viscosity and the solids in digesting sludge. Previously, Hatfield (70) had shown that the apparent viscosity of sewage sludge increased exponentially as the total solids content increased. Behn (16-17) discussed the results of several studies and concluded that digested sludge is probably a Bingham[1] plastic with the possibility of pseudoplastic[2] and thixotropic[3] behavior. Buzzell and Sawyer (26)

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[1] Bingham plastics possess a rigidity enabling them to withstand a certain amount of stress, termed the yield value. When the shearing force exceeds this, the internal structure seems to collapse and the shear stress increases proporionately with the shear rate.

[2] Pseudoplastic liquids behave as though their particles become more aligned at higher shearing rates thereby offering relatively less resistance to flow.

[3] Thixotropic liquids tend to become less viscous as the period of shear at a given rate continues.

obtained results which indicated that digesting sewage sludge is a pseudoplastic material with only slight thixotropic behavior. The viscosity of digesting sewage sludge is dependent upon the total and volatile solids concentrations of the sludge. They concluded that the viscous nature of digesting sludge may be a limiting factor in the design and operation of high rate digesters because of inefficient mixing and inflation of the sludge mass by entrapped gas bubbles.

Study of effect of activated carbon on the rate of sludge digestion has produced conflicting results (91, 145, 173). Rudolfs and Trubnick (145) and Keefer and Kratz (91) observed an increased rate of digestion and gas production with additions of activated carbon in concentrations up to 20 gm/l. Rudolfs and Trubnick noticed that 7.5 gm/l of carbon reduced digestion time from 127 days to 69 days and 15 gm/l reduced the digestion time to 42 days. They did conclude that carbon was not so effective in a digester operating correctly as when unbalanced conditions prevail. When activated carbon was added to the digesting sludge, the drainability of the sludge was improved, the pH value was maintained at a higher level, volatile matter reduction was greater and the carbon dioxide content of the gas was lower. Walker (173) found that additions of 5 to 15 mg/l of carbon did not affect the digestion process at 28° C and 45°C. Concentrations of carbon above this amount decreased the gas production. Digestion at 20° C was improved by the addition of 5 to 15 mg/l of activated carbon. At all temperatures carbon increased the drainability of the sludge, the optimum dose being 30 mg/l, and increased the methane content of the gas produced. Keefer and Kratz (91) obtained optimum benefits with regard to total gas production, rate of gas production and the quality of the gas produced with activated carbon additions of approximately 45 mg/l. The temperature of digestion was 28° C. The authors quoted Rogers (134) as having been able to increase the rate of sludge digestion in a full size plant with activated carbon.

However, at a later date, Keefer and Kratz (89) studied the comparative effects of lime and activated carbon on sludge digestion and found no benefits using activated carbon at concentrations of 116 mg/l. Flower <u>et al</u>. (49) discussed operating experiences at various plants using activated carbon in the sludge digestion process. Reports of odor reduction, scum nuisance elimination, improved drainability of sludge, accelerated and increased gas production were mentioned. They found from experiments that 200 mg/l activated carbon increased gas production but higher dosages did not help. The carbon had little effect on the pH of the digesting sludge. Morgan (116) in 1954 studied the effect of gas mixing and the addition of coke on the sludge digestion process. The results showed that coke additions up to 1.05 lb per 100 gal of raw sludge (a concentration of approximately 1260 mg/l) had little effect upon the digestion process.

Rudolfs (137) in 1932 attempted to improve the digestion process with regard to time for digestion, gas production and drainability of the digested sludge by adding protein and fat hydrolizing enzymes to digesting sludge under optimum conditions for the action of the enzymes. No beneficial effects were obtained. In 1953, McKinney (111) discussed the biochemistry of waste treatment processes and concluded that a biological treatment system which is designed correctly and operated according to the design principles will not benefit from the addition of organic catalysts. This conclusion has been supported by McKinney and Poliakoff (112) Heukelekian and Berger (76) and Grune and Sload (68). Heukelekian and Berger (76) also attempted to increase the rate of sludge digestion by culture additions. They found that the addition of bacterial cultures and yeast to non-sterile, fresh solids had no significant effect and such addition to sterile, fresh solids initiated the liquefaction portion of the digestion process.

D. Digester Control

An important factor in digestion control is the buffering capacity, or resistance to change in pH, of the digesting sludge. The alkalinity of a digesting sludge is directly proportional to its buffering capacity. Simpson (160) states that "the buffering capacity of raw sludge is due, almost entirely, to the alkalinity of the carriage water, and if this alkalinity is low it will be destroyed by the organic acids produced in the first stage of digestion resulting in a fall in pH and inhibition of the methane producing bacteria." However, in normal digestion several reactions occur which produce ammonia which can combine with carbon dioxide and water to produce the buffer ammonium bicarbonate. The ammonium bicarbonate also contributes to the alkalinity of the digesting sludge. Desirable values of alkalinity in digesting sludge range from 2000 to 4000 mg/l (7, 34, 35, 58, 177). Higher alkalinities have been reported by Haseltine (69) and Garber (53) with thermophilic digestion, presumably due to a greater degradation of proteinaceous material. Albertson (2) studied the effect of ammonia-nitrogen on digester operation and observed that concentrations above 1250 mg/l at a pH close to 7.0 were associated with digester failure. He stated that an increase in alkalinity in a digester tends to increase the permissible operating level of the volatile acids. An empirical equation relating pH, ammonia-nitrogen, carbon dioxide and volatile acids was presented. The toxicity of ammonia to the organisms in the sludge digestion process appears to be due to the free ammonium ion (107). At any given ammonia-nitrogen concentration, the existence of free ammonia will depend upon the pH of the system. As the pH is decreased, a greater ammonia-nitrogen concentration can be tolerated without free ammonia occurring. Prethickening of sludge tends to increase the ammonia-nitrogen concentration but Albertson (2) does not believe that this will have a significant effect on the digestion process if the solids concentration of the sludge is less than 12 percent. He does suggest that, if problems do arise due to excessive ammonia-nitrogen concentrations, they can be solved by neutralizing with hydrochloric acid or by reducing the pH of the digesting sludge with carbon dioxide.

The practice of liming to start up digesters and control their performance has received much discussion (45, 55, 119, 133, 149, 155, 156). The outcome of a sewage treatment plant operators' forum in 1949 (45) was to recommend the use of lime. Giles (55), Rockercharlie (133) and others have found the addition of lime to digesters to benefit the process and to assist in scum control. Cassell and Sawyer (31) found the use of lime to be beneficial in the starting of high rate digesters. A loading rate of 0.162 volatile solids added per cu ft per day and a detention time of 20 days could be tolerated in the start up of high rate digesters if the pH were maintained at 6.8 to 7.2 by liming. Without liming, digestion did not develop in a high rate digester operated at a 30 day detention time and volatile solids loading of 0.045 lb added per cu ft per day. Schlenz (155, 156) did not advocate the liming of digesters. Simpson (160) believed that in many instances the disappointing results obtained in the past with liming digesters may have been caused by liming in an unscientific fashion. He stated that some of the problems which can be associated with irresponsible liming are: incomplete mixing of the lime such that it gravitates to the bottom of the digester and solidifies; the creation of areas of intense alkalinity; over-adjustment of the pH; absorption of carbon dioxide by the lime, which may cause a partial vacuum in a fixed cover digester; the precipitation of calcium carbonate scale on the equipment inside the digester; and the removal of carbon dioxide which is necessary to the metabolic activity of some organisms. Sawyer et al. and Neuspiel and Morgan (149, 119) used the volatile acids concentration present in a digester as a basis for liming. The conclusion reached was that the addition of lime in a quantity equivalent to 200 percent of the volatile acids present was the optimum amount for the relief of stuck digesters. Values of pH up to 10.0 can be tolerated. The addition of lime on a controlled basis of 14 lb lime per 1000 cu ft of digester capacity started the digestion process more rapidly than without lime. Lime was not essential to start the digesters. McCarty (102) has discussed the use of lime for controlling the pH in digesters and concluded that no beneficial effects result from the addition of lime to raise the pH above 6.7 to 6.8. After this point, the lime combines with the carbon dioxide in the digester to form insoluble calcium carbonate which is ineffective in neutralizing excessive volatile acids or for raising the pH.

Sodium bicarbonate is recommended as one of the most effective materials for pH control because of the greater ease of addition, control and handling.

Foaming is a problem experienced in many anaerobic digestion systems. Schlenz (154) defines foaming in digesters as the situation in which froth, gas and scum rise in the gas vents to overflowing. Some of the possible causes of foaming as given by Schlenz are: excessive loadings of raw solids with respect to the digesting solids in a digester, causing a rapid production of acids which, when they react with carbonates and bicarbonates, produce a large amount of carbon dioxide; changes in pH; the viscosity of the scum or liquid in the digester; and a restricted gas vent area. Another possible cause of foaming in an unheated digester may be the onset of warmer weather after a cold period thus stimulating the gas-producing organisms. The remedies suggested by Schlenz (154) include the balancing of the quantities of raw and digesting sludge solids in the digester, the addition of lime, chlorination with 3 to 4 mg/l chlorine, and reducing the loading to the digester.

Oxidation-reduction potential has been used as a method of process control in sewage treatment. Little attention has been paid to its use in anaerobic digestion (65). The biochemical decomposition of organic compounds involves several oxidation-reduction reactions. According to Hewitt (80), the oxidation-reduction systems in cells are so essential that life may be defined as a continuous oxidation-reduction system. Electrons are transferred between compounds in oxidative and reductive reactions resulting in potential changes. The basic equation (38) relating the oxidative state of a thermodynamically reversible system to the resulting potential is:

$$E_h = E_o + \frac{RT}{nF} \ln \frac{(OXID)}{(RED)}$$

E_h is the potential of the system referred to the normal hydrogen electrode and E_o is a specific constant for the system, both being measured in volts. R is the universal gas constant, T is the absolute temperature, n is the number of electrons transferred in the reaction and F is the Faraday Constant. The molar concentrations of oxidant and reductant are (OXID) and (RED) respectively. During the metabolic processes of microorganisms, a definite oxidation-reduction potential is maintained in a particular organism-substrate system. The factors which influence the potential and the rate of change of the potential are: the pH; the tendency of the system to take up or give off electrons; the temperature of the solution; and the ratio of the concentration of the oxidant to that of the reductant (65). Many who have discussed the measurement of oxidation-reduction potentials emphasize that the measurement is difficult and a profound understanding of the underlying principles is necessary for meaningful interpretation of the results. Grune et al. (65) determined the range of potentials for raw sludge to be 85 to -20 mv with a rapid rate of change under proper seeding and temperature conditions. For well digested sludge the potential was found to be -250 mv with a slow rate of change, and for actively digesting sludge the potential range was -100 mv to -250 mv. Dirasian et al. (38) found that optimum digestion appeared to be a function of a healthy methane producing flora which seemed to thrive best at potential levels between -520 and -530 mv. The organisms continued to function, however, over a range from -490 to -550 mv.

Agardy et al. (1) experimented with the use of enzyme activity as a parameter of digester performance. They employed a synthetic substrate seeded with screened, digested sludge. The results showed a rapid increase in proteolytic enzyme activity during the onset of digestion failure, and a rapid decrease in proteolytic activity when complete fermentation failure occurred.

III. GENERAL

The purpose of this dissertation is to determine the effect of solids concentration in the digester on the anaerobic digestion of domestic sewage sludge. This aspect of sludge digestion was studied as a thesis topic because of the increasing number of sewage treatment plants which practice sludge thickening before the raw sludge is added to sludge digesters. As a result of the increased use of sludge thickening prior to digestion, the solids concentrations in many treatment plant digesters have increased to levels not usually obtained previously. The increased solids concentrations in the feed sludge and in the digesting sludge have allowed the use of higher loading rates to digesters. Keefer (88), Torpey (168, 169), Sawyer and Schmidt (152) and Schulze (157) studied the effect of sludge solids concentration on the anaerobic digestion process. However, the sludge solids concentrations used in their studies did not cover the range of concentrations expected in practice using sludge thickened prior to digestion. Sawyer and Schmidt (152) studied the digestion of sludge at solids concentrations of 1.35 to 5.2 percent. Torpey (168) obtained solids concentrations in digesting sludge as high as 6.8 percent and Keefer (88) digested sludge at a solids concentration of 15 percent. The solids concentration of sludge thickened prior to digestion is usually between 4 and 12 percent. The upper limit is set by the equipment available for conveying the sludge from the thickening unit to the digestion unit.

In this study six temperature controlled, completely mixed, experimental digesters were used to study the effect of solids concentration on digestion. The digesters were numbered 1, 2, 3, 4, 5 and 6 and the solids concentration of the sludge in each digester was maintained approximately constant. An attempt was made to keep the solids concentrations close to 2, 4, 6, 8, 10 and 12 percent in digesters 1 through 6, respectively. Raw sewage sludge was fed to the digesters once daily just after withdrawing an equivalent amount of digesting sludge. The solids concentration of the raw feed sludge was controlled so that the desired solids concentrations in the digesting sludges could be maintained. The sludge withdrawn from the digesters was analyzed to determine the characteristics of the digestion process. Analyses were made on the digesting sludge to determine the total and volatile solids concentrations, the volatile acids concentration and the individual volatile acids present, the dewatering characteristics of the sludge and the other parameters normally used to follow the course of sludge digestion, The gas produced in the digesters was also analyzed.

Three test runs were made. The first run was made using Pyrex bottles for digesters. In this run the digesters were not started at the ultimate desired total solids concentrations. Actively digesting sludge from the Ames, Iowa, sewage treatment plant was used to start all of the digesters at the same solids concentration. The total solids concentrations in the digesters were changed over a period of time to the desired values by adjusting the solids concentrations in the feed sludge. After 43 days and before the desired concentrations were reached, the digester bottles failed.

The second test run was made using aluminum digesters. They were started at 2, 4, 6, 8, 10 and 12 percent solids concentrations as discussed in the procedure. However, although the run lasted for 51 days, the digestion reached a stable condition for short periods of time only in digesters 1, 2 and 3. In digesters 4, 5 and 6, stable conditions did not exist at any time. The author believes that this was due to an attempt to start the digesters at too high a loading rate.

The third test run was started and proceeded satisfactorily throughout the run, which lasted 72 days. For this reason, most of the data and discussion refer to the results of this run. If other data are referred to, they will be specifically mentioned. Initial loading rates to the digesters were low. When the gas production appeared to reach a uniform value, the loading rate was increased. At each loading rate, the various analyses were made to determine the digestion characteristics under those conditions. This procedure was repeated until digestion failed.

The results expected from the study were:

1. Detention time will be the dominant parameter affecting the digestion process. The rate and degree of decomposition of the sludge solids in the process are determined by the organisms involved and the detention time of the sludge particles in the digester. If the detention time is so short that the methane producing organisms cannot become firmly established in the digesting sludge, digestion failure will occur. The minimum time a sludge particle must remain in a digester for good digestion is correlated to the generation time of the methane producing organisms.
2. As the solids concentration of the digesting sludge increases, the maximum organic loading rate which can be achieved without digestion failure will increase. Assuming a constant detention time, the organic loading rate will increase as the solids concentration is increased. If detention time is the most important parameter affecting the digestion process, then the expected result regarding the maximum loading rate will be correct.
3. A factor which will limit the continuous increase of loading rate without digestion failure by increasing the solids concentration is the viscosity of the

sludge. As the solids concentration increases, the viscosity of the sludge increases. The increased viscosity will adversely affect the mixing efficiency and eventually digestion failure will occur.

4. As the detention time decreases, the degree of decomposition of the sludge solids will decrease because of the shorter time the organisms will have to metabolize the organic portion of the solids. This should result in lower gas production per weight of sludge handled.
5. The volatile acids concentration in the digesting sludge will increase as the solids concentration increases, since there will be less liquid available to dilute the volatile acids produced by the acid producing organisms.
6. Propionic acid will appear in digesting sludge when digestion begins to fail.
7. The specific resistance of the digesting sludge will decrease as digestion continues. Digested sludge should be easier to dewater than raw sludge.
8. The pH of digesting sludge will drop as the detention time decreases due to the accumulation of volatile acids. The acid producing organisms are apparently less affected by detention time than are the gas producers.
9. The alkalinity of digesting sludge will decrease as the detention time decreases, but will increase as the solids concentration increases.

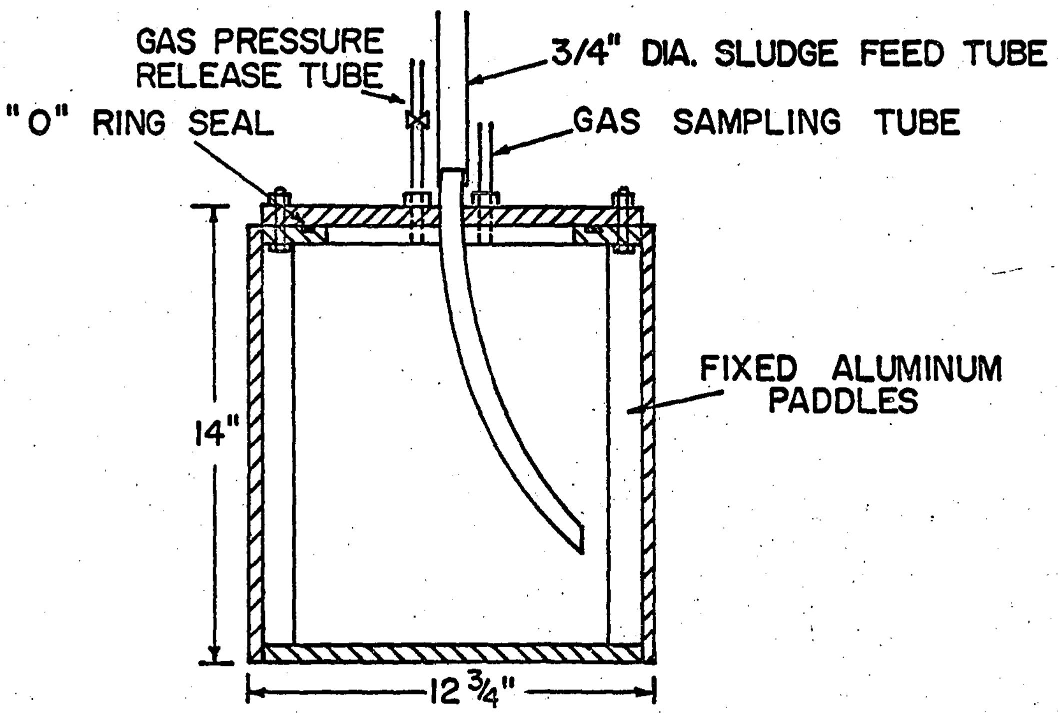
IV. EXPERIMENTAL INVESTIGATION

A. Apparatus

The fundamental digestion apparatus consisted of six cylindrical digesters containing sewage sludge placed inside a temperature controlled cabinet. A diagram of a typical digester is shown in Figure 3. The digesters were constructed from 12-inch I.D., 3/8-inch wall thickness, Schedule 40, 6063-T6 aluminum pipe. At the end of the study, no visible signs of corrosion of the aluminum were evident. A 1/2-inch thick, circular aluminum plate was welded to one end of the cylinder. A flange ring was welded in the other end, and a 1/2-inch thick cover plate was bolted to the flange ring. An "O" ring was used for a seal between the flange ring and the cover plate. Sludge was added to and withdrawn from the digesters through a 3/4-inch diameter aluminum tube which extended into the digester. A short length of rubber hose which could be sealed with a clamp was attached to the portion of the aluminum tube protruding from the digester. Two other means of access to the digester contents were made with 1/4-inch copper tubing connected to holes in the cover plate of the digester. A quarter-turn gas tight valve was placed in one of the pieces of tubing to seal off the contents of the digester at all times, except when reading or relieving gas pressures. The other piece of tubing was sealed with a self-sealing rubber serum stopper. This tube was used for obtaining gas samples for analysis. Mixing of the contents of the digester was achieved by rotating the six digesters about a horizontal axis on rubber covered rollers. Figure 4 shows the position of a digester on the rollers within the constant temperature cabinet. Inside the digester, four strips of aluminum, 1-1/2 inches wide and 1/4 inch thick, were welded to the interior surface parallel to the longitudinal axis. The strips, which remained stationary with respect to the digesters, effectively mixed the sewage sludge. A one-horsepower, 1700 rpm electric motor, connected through a gear reduction box and a chain, was used to drive one of the rollers. The rollers were made of 2-inch diameter steel tubing covered with reinforced rubber hose. Self-aligning, ball-bearing, pillow blocks were used to support the axles of the rollers at each end. The drive roller was connected to all but two of the remaining rollers by a drive chain. The six digesters were placed between alternate pairs of rollers and as the rollers revolved, the overall effect was to rotate the digesters at a rate of nine revolutions per minute.

The temperature controlled cabinet enclosed both the mixing apparatus and the digesters, Figure 4. Two sets of doors, one set glass and the other wood, were built into the cabinet during construction. The inner set of doors, i.e. the glass set, were used to view the operation of the mixing apparatus and digesters without disturbing the temperature

Figure 3. Schematic diagram of a typical aluminum digester


GAS PRESSURE RELEASE TUBE
"O" RING SEAL
3/4" DIA. SLUDGE FEED TUBE
GAS SAMPLING TUBE
FIXED ALUMINUM PADDLES
14"
12 3/4"

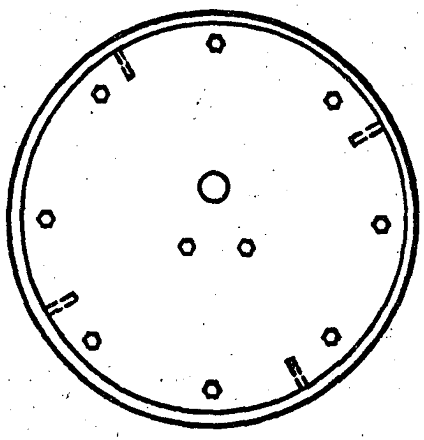
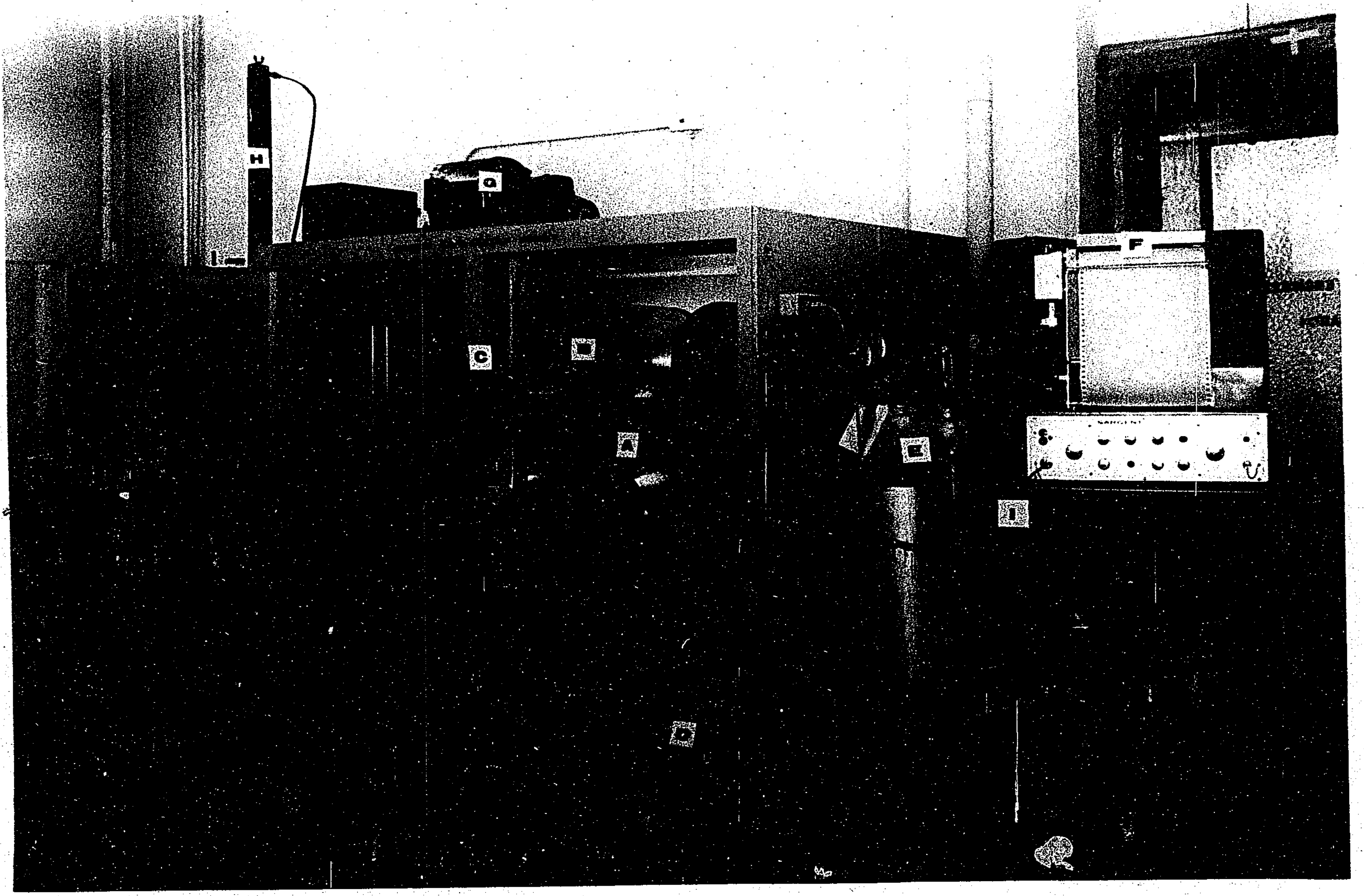


Figure 4. The digesters in position on the mixing apparatus inside the constant-temperature cabinet.

A. Mixing apparatus
B. Aluminum digester
C. Temperature controlled cabinet
D. Gas chromatography apparatus
E. Helium cylinder
F. Recording potientiometer
G. Drive motor for mixing apparatus
H. Manometer
I. Connection between conductivity cell and recording potentiometer


H
G
C
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I

inside the cabinet. The temperature in the cabinet was maintained at 35° C ±1° C using a heater and a thermostat. A fan circulated the heated air throughout the cabinet to maintain uniform temperature conditions.

Several difficulties with the operation of the apparatus occurred. Initially, five-gallon capacity pyrex bottles were used as digesters. Pyrex bottles had been used satisfactorily in a previous study (40), but during an early run in this work several bottle failures occurred. The loss in data and time resulting from such a failure emphasized the need for a more durable material with which to construct digesters. To provide the strength required glass would be too expensive, steel was too heavy, plexiglass was not economically feasible and aluminum was subject to corrosion. A rigid digester was necessary because gas volumes were calculated using the volume of the digester as a basis. This excluded easily deformable materials. A further investigation into the possibility of using aluminum was made and the conclusion was drawn that the corrosion of the aluminum due to the action of sewage sludge would be small. This was realized in actual operation.

Gas leakage through the aluminum welds in the digesters presented problems in the early portion of this study. The welds were cut out and the digesters rewelded but the problem still existed although it was not quite so severe in nature. A solution could not be found to this problem even though gasket forming compounds were used and the insides of the digesters were coated with fiberglass. The leakages were eventually stopped by placing sewage sludge inside the digesters and allowing the gas pressure to build up. Solids were forced into the porous welds and an effective gas seal was obtained.

During rotation on the mixing device, the digesters tended to move either forwards or backwards on the rollers. This problem was solved by placing guard rails in front of and behind the digesters. To the guard rails were attached rubbing brushes to prevent the rotating digesters from rubbing against the rails.

B. Experimental Procedure

Before the start of a test run, the volumes of the digesters were determined and recorded. The digesters were weighed empty and then filled with water and reweighed. The temperature of the water was noted and the volume occupied by the water was calculated. The digesters were emptied. Actively digesting sludge from the primary digester at Ames sewage treatment plant was thickened and added in combination with non-thickened actively digesting sludge to each experimental digester. This sludge was chosen to provide a strong population of methane producing bacteria in the pilot digesters. A total weight of 8000 grams 50 grams of sludge was added to each digester. The proportion of ordinary sludge to thickened sludge was governed by the total solids content desired in a particular digester. Where the solids concentration desired was less than that of the non-thickened sludge, distilled water was added until the desired concentration was obtained. An attempt was made to maintain the total solids concentration at 2, 4, 6, 8, 10 and 12 percent in digesters 1, 2, 3, 4, 5 and 6 respectively. It was found more convenient to maintain the concentrations at values slightly different than those listed. The actual concentrations obtained can be seen in Figure 22. The remaining air in the digesters was flushed out with helium gas. Oxygen is harmful to many anaerobes and is undesirable in an anaerobic digester. All the inlets to the digesters were sealed. The digesters were placed on the mixing apparatus in the temperature controlled cabinet and the mixing apparatus was set in motion. The digesters remained in the cabinet for four days during which time interruptions in the mixing process were made once a day to relieve the gas pressure inside the digesters. No sludge was added or removed during this period to enable the organisms in the sludge to become acclimatized to the new environment. After the fourth day, a raw sludge feeding program was initiated. A typical feeding program is indicated in Figure 17.

Raw sludge for feeding the digesters was collected from the sludge well adjacent to the primary sedimentation tank at the Nevada, Iowa sewage treatment plant. The sludge was mixed thoroughly in a 55-gallon drum and a portion of it was stored immediately while another portion was thickened before storage. The sludge was stored in quart size ice cream containers in a deep freeze at -20°C until required (Figure 5). Prior to feeding the digesters, the raw sludge was removed from the deep freeze and allowed to thaw in a covered bucket placed in hot water. The sludge was then heated to approximately 35°C prior to feeding to a digester.

Thickened sludge was prepared by filling four, one gallon pails with ordinary sludge. The pails previously had small holes drilled through their sides. Masking tape was stuck over the holes to prevent the sludge from running out when they were filled. The filled pails were placed in a deep freeze until the sludge was frozen. The masking tape was removed from the holes in the pails and the sludge was thawed. As the sludge thawed a large proportion of the water separated from the solids and drained from the pails leaving the thickened sludge. Using this method sludge could be thickened to a concentration greater than 20 percent total solids. The thickened sludge was placed in quart size ice cream containers and placed in a deep freeze until required.

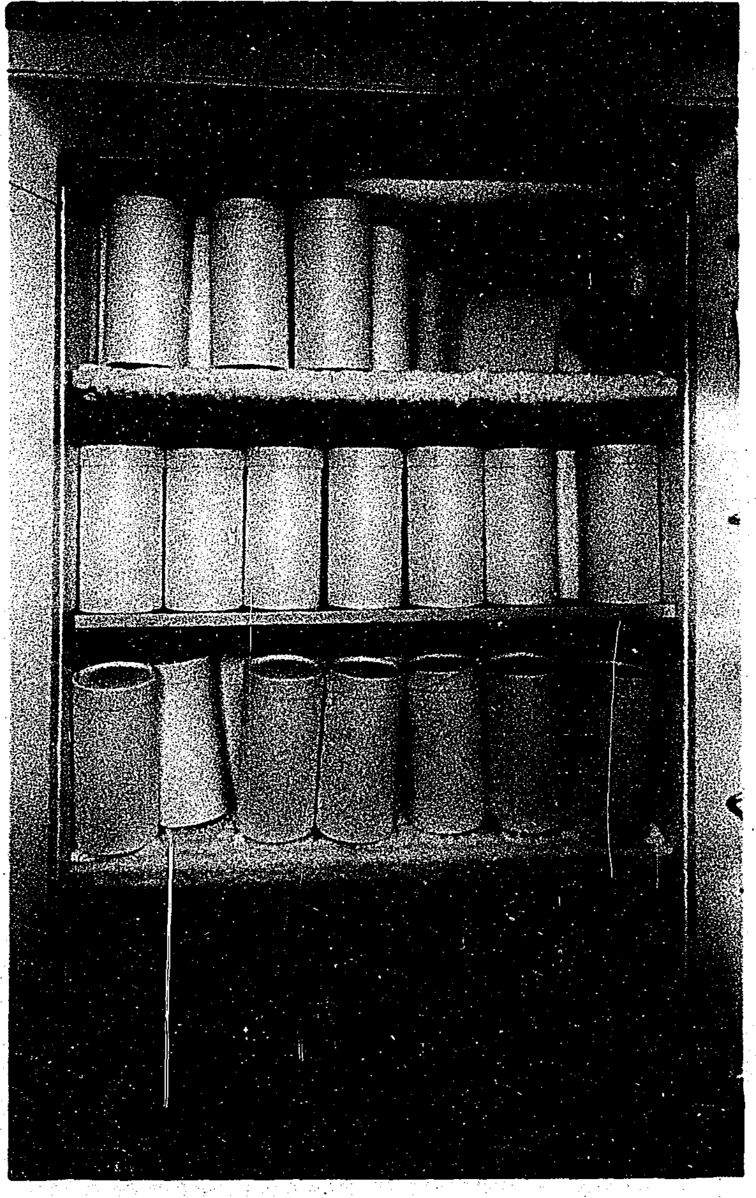
Each day, the digesters were removed from the cabinet one at a time for feeding and withdrawal of sludge. The digester was weighed to the nearest 50 grams on a single pan

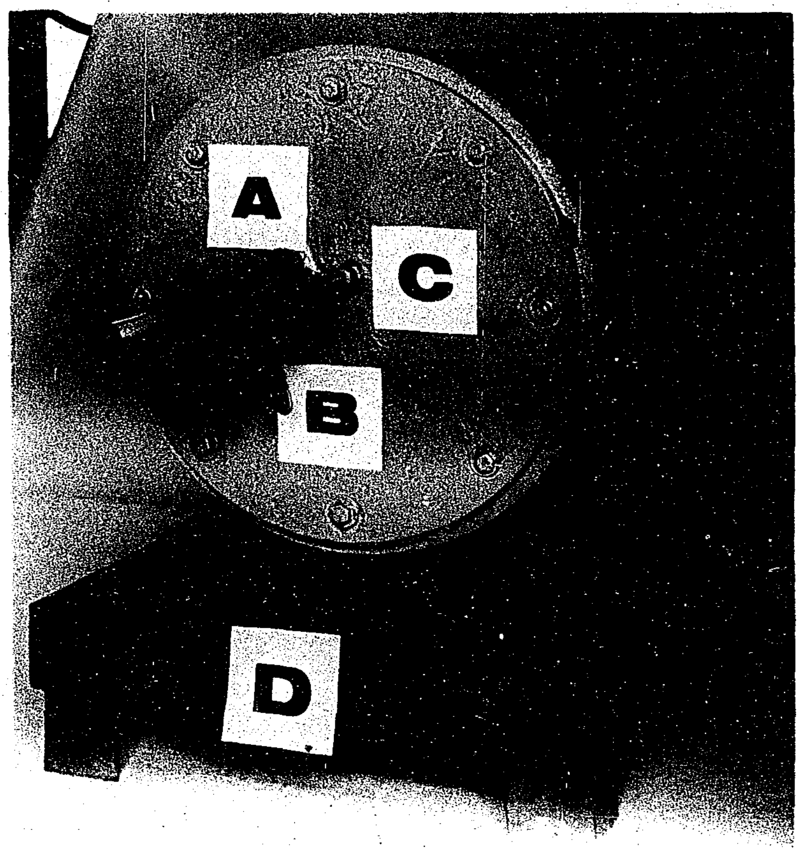
Figure 5. Sludge stored in the deep freeze.
(left)

Figure 6. Digester on hand mixing roller.
(upper right)

A. Sludge feed and withdrawal tube.
B. Gas pressure relief valve.
C. Gas sampling tube.

Figure 7. Sludge gun used for feeding digesters.
(lower right)

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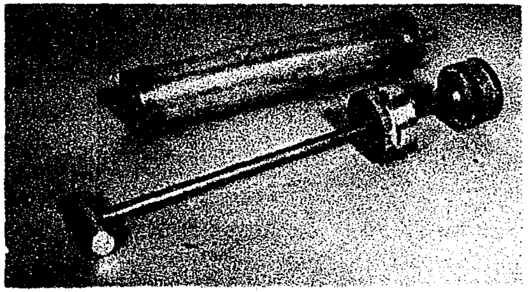


balance to determine the weight of sludge in the digester. The error caused by this approximation was less than one percent. The digester was placed on a hand mixing roller (Figure 6) and rotated until the end of the 3/4-inch aluminum feed tube inside the digester was below the surface of the sludge. Sludge was withdrawn from the digester feed tube utilizing the gas pressure inside the digester to force it out of the digester and into a one liter "Nalgene" graduated cylinder. As the clamp on the rubber hose attached to the feed tube was slowly opened, sludge was forced out of the digester. In previous work (40) difficulties had been experienced with obtaining representative samples of the contents of the digesters. To eliminate this difficulty, a pair of rollers were constructed so that a digester could be rolled at a relatively high speed by hand immediately before withdrawing sludge (Figure 6). After an amount of sludge in excess of that required had been withdrawn, the remaining gas pressure was released to the atmosphere. In cases where high gas pressures were obtained, it was found to be safer to release some of the gas before withdrawing sludge. The excess sludge withdrawn was used as seed and was returned to the digester along with the raw sludge being fed that day. The amount of sludge withdrawn from a digester was determined by the weight of sludge in the digester and the amount of sludge to be fed to the digester that day. The weight of sludge in each digester was kept approximately constant at 8000 gm. Usually it was necessary to withdraw and

and waste less sludge than was to be added on a particular day because of the amount of sludge lost due to digestion.

The daily production of gas was measured by observing the gas pressure build up in the digester from one daily feeding to the next. The gas outlet tube of a digester was connected to a seven-foot Meriam mercury manometer and the pressure was measured in centimeters of mercury. This reading was converted to ft^3 of gas produced at standard temperature and pressure. A correction was made to allow for the solubility of carbon dioxide in the sludge. The analyses made on the gas and the digested sludge are discussed in the section on analyses.

Sludge was fed to the digester using the sludge gun shown in Figure 7. The gun was made of a 14-inch length of 2-3/4-inch diameter brass tubing to the end of which had been brazed a nozzle. A piston made watertight with two "O" rings was used to force sludge from the gun. The amount of sludge to be fed to a digester was poured into the gun and the piston replaced. While holding the gun in a vertical position with the nozzle upmost, the piston was depressed until all the air had been expelled. The nozzle was then connected to the feed tube of a digester and the sludge was forced into that digester.

The feed sludge for a particular digester was made up from non-thickened sludge and thickened sludge or distilled water, depending upon the total solids concentration of the feed sludge required to maintain the desired total solids concentration inside the digesters. A 1000 gm capacity spring balance was used to weigh out the quantities of the materials needed to make up the feed sludge. The sludge was heated to 35° C and thoroughly mixed with excess digesting sludge previously drawn from the digester.

The operating procedure for feeding the digesters and making the analyses to determine the course of digestion may be summarized in the typical daily schedule shown below:

1. Cartons of thickened and non-thickened sludge were taken from the deep freeze and placed in buckets in a hot water bath. The approximate amount of each type of sludge required had previously been determined according to the feeding rate schedule for the digesters.
2. The amounts of sludge required by each digester were made up in the specified concentrations and heated to 35° C.
3. The temperature in the cabinet was checked.
4. The driving motor for the cabinet mixing apparatus was stopped.
5. Each digester was connected to the manometer in turn and the gas pressure measured.
6. If desired, a sample of gas was taken for analysis.

7. A digester was removed from the cabinet, weighed, then placed on the two hand-operated rollers and the digester contents were mixed.
8. Sludge was withdrawn from the digester as described previously.
9. Excess gas pressure was released to the atmosphere.
10. Sludge withdrawn in excess of that which was necessary was mixed with the feed sludge to the digester.
11. The remaining sludge withdrawn from the digester was either kept for analyses or wasted.
12. The digester was fed, sealed and replaced in the cabinet and the procedure was repeated with the other digesters.
13. After all the digesters had been fed, the cabinet was closed and the mixer drive motor was restarted.

The time required to complete the procedure from making up the feed sludge to restarting the apparatus was between one and two hours. The times taken to complete the various analyses and operations in this study are shown in Table 35 in the Appendix.

The only serious problem encountered in the overall procedure was the collection of supplies of raw sewage sludge. On three or four occasions, the raw sludge from the primary sedimentation tanks at Nevada showed the characteristics of a partially digested sludge. It was black, the total solids concentration was high and the volatile solids concentration was low. Fresh sludge is usually brown in color, has a relatively low total solids concentration, and has a high volatile solids concentration. When this occurred, sufficient sludge was collected to last only until fresh sludge could be obtained. The results of using such partially digested sludge are indicated in Figure 17 by the gas production values between days 40 to 45.

C. Analyses

The digestion within the six digesters was controlled and its progress evaluated by making several analyses of the sludge and the gas produced. In most cases, the tests are recognized by Standard Methods (3) as being the standard tests for use in sludge digestion studies. It was necessary throughout the digestion studies to obtain certain information about the digestion process for which no standard test is recommended. When this situation occurred, a test was either obtained from another source or developed to suit the particular purpose. The exceptions to the standard tests are fully described in this section. A discussion of the value of most of the tests will be found in the Literature Review.

1. pH

The pH of a liquid is defined as the logarithm to the base 10 of the reciprocal of the hydrogen ion concentration. All pH values of the raw and digested sludges were measured in accordance with Standard Methods using a Beckman Zeromatic pH meter, Model 9600 equipped with a manually operated temperature compensating circuit.

2. Volatile acids

The sludge withdrawn from each digester was tested for volatile acids using the "Distillation Method (Tentative)" as described in Standard Methods.

3. Alkalinity

Alkalinity of the supernatant obtained after centrifuging the sludge obtained from the digesters was determined in accordance with the potentiometric method described in Standard Methods. An exception to Standard Methods was made in the cases of high alkalinity values. Instead of using 0.02 N sulfuric acid as recommended, 0.1 N sulfuric acid was used. This was necessary because of the size of titration beaker used.

4. Total and volatile solids test

The total solids and volatile solids in the raw and digested sewage sludges were determined by the standard tests commonly used in sludge digestion studies and fully described in Standard Methods. A resume of the tests will be given to assist in understanding the results of the digestion studies.

The total solids test determines by evaporation on a 100 C^o water bath the quantity of solid material, including settleable, suspended, colloidal and soluble solids, in a given weight of sludge. The results are usually reported in percentage form. The volatile solids test attempts to determine the quantity of organic matter present in a given sludge. The dried solids obtained in the total solids test are burned in a muffle furnace at 600°C for one hour. The organic matter is volatilized leaving the "fixed" solids. The volatile solids content, or the loss in weight of the total solids, is expressed as a percentage of the total solids weight.

In the operation of the digesters, the total solids test by itself was necessary to determine the make-up of the feed sludge and the solids content maintained in the digesters. The total solids test and the volatile solids test were used together to determine the loading rates to the digesters and to determine the destruction of volatile matter in the digesters.

5. Gas production

The volume of gas produced in digesters 1 through 6 was measured once a day just before feeding the digesters. After feeding a typical digester, the gas pressure relief valve (Figure 3) was left open until the pressure inside the digester was the same as the pressure outside the digester. When the pressure inside the digester was the same as atmospheric pressure the digester was sealed by closing this valve. Suppose the volume of gas inside the digester at this time was V_D at a standard temperature (T_S) and a standard pressure (P_S). Let the atmospheric pressure be represented by P_{AB}.

The digester was then placed on the mixing apparatus inside the temperature controlled cabinet until it was fed again, approximately 24 hours later. Gas which was produced inside the digester during the 24 hours could not escape from the digester, resulting in a pressure build up. At the end of 24 hours the pressure inside the digester was measured using a mercury manometer. Let the pressure indicated by the manometer reading be represented by P_M. Suppose the volume of gas inside the digester at the end of 24 hours was V_E at a standard temperature (T_S) and a standard pressure (P_S). Let the atmospheric pressure be represented by P_{AE}. Thus the volume of gas produced (V_P) in the 24 hours of digestion is the volume of gas at the end of the 24 hours (V_E) minus the volume of gas at the beginning of the 24 hours (V_B).

$$V_P = V_E - V_B$$

The temperature of digestion is represented by T_D and the volume of the digester occupied by gas is represented by V_D (liters). V_D is determined by subtracting the volume of digesting sludge from the volume of the empty digester. V_B and V_E may be determined using ideal gas laws. The volume of gas at the beginning of the 24 hours is determined from:

$$\frac{V_D \cdot P_{AB}}{T_D} = \frac{V_B \cdot P_S}{T_S} \quad \text{OR} \quad V_B = \frac{V_D \cdot P_{AB} \cdot T_S}{P_S \cdot T_D}$$

The total pressure inside the digester at the end of the 24 hours is:

$$P_{total} = P_{AE} + P_M$$

The volume of gas inside the digester at the end of the 24 hours is determined from:

$$\frac{V_D \cdot (P_{AE} + P_M)}{T_D} = \frac{V_E \cdot P_S}{T_S}$$

$$\text{or} \qquad V_E = \frac{V_D \cdot (P_{AE} + P_M) T_S}{P_S \cdot T_D}$$

$$\text{Hence } V_P = V_E - V_B = \frac{V_D T_S}{P_S T_D} \left[(P_{AE} + P_M) - P_{AB} \right]$$

A study of previous records indicated that the difference in barometric pressure in 24 hours was negligible (i.e. $P_{AB} = P_{AE}$). The maximum variation was approximately one centimeter of mercury in a 24-hour period. The average variation was only three millimeters of mercury.

$$\begin{aligned} \text{Thus, } V_P &= C.P_M \\ &= \frac{V_D T_S}{P_S T_D} \cdot P_M \end{aligned}$$

The following standard conditions were assumed:

$T_S = 0^oC = 273^oK$ (degrees Kelvin)

$P_S = 76$ centimeters of mercury

$T_D = 95^oF = 35^oC = 308^oK$

$P_M =$ manometer reading (centimeters of mercury)

$$\text{Therefore } V_P = \frac{(V_D)(P_M)(273^oK)}{(76)\ (308^oK)} \quad \text{liters}$$

$$= \frac{(V_D)(P_M)(273^oK)}{(76)(308^oK)(28.32)} \quad \text{cu ft}$$

$$V_P = (4.14 \times 10^{-4})(P_M)(V_D) \text{ cu ft}$$

All gas productions were calculated from this expression. The exact quantitative volumes of gas may vary from those determined by this method by about 1.3 percent, but comparisons between digesters are always valid.

A correction was made to the gas production data to account for the loss of carbon dioxide due to the solubility of carbon dioxide in the sewage sludge. Henry's law was used to calculate the correction. Henry's constant (K) for sludge was assumed equal to Henry's constant (K) for water (36) because a literature search did not reveal any work which had been done on the solubility of carbon dioxide in sewage sludge. Henry's law states that:

$$K = \frac{P_A}{N_A} = \frac{\text{Pressure of gas A in millimeters}}{\text{Mole fraction gas A in solution}}$$

$$n_{CO_2} = \frac{(P)(R_{CO_2})(W)}{(K_{CO_2})(18.02)}$$

where n_{CO_2} is the number of moles of carbon dioxide in solution.

P is the pressure in the digester in millimeters of mercury.

R_{CO_2} is the percentage of carbon dioxide in the gas in the digester.

W is the weight of sludge in the digester in grams.

K_{CO_2} is Henry's Constant for the solubility of carbon dioxide in water, 1.25×10^6 at 25° C (36).

The volume of carbon dioxide dissolved in sludge at a standard temperature and pressure of 0° C and 76 centimeters of mercury is:

$$V_{CO_2} = \frac{(P)(R_{CO_2})(W)}{K_{CO_2}(18.02)} \times \frac{(22.4 \text{ liters per mole})}{} \quad \text{liters}$$

$$V_{CO_2} = (3.34 \times 10^{-8})(P)(R_{CO_2})(W) \qquad \text{cu ft}$$

where V_{CO_2} is the volume of carbon dioxide dissolved in the sludge at standard temperature and pressure. To obtain the total amount of gas produced each day, the volume of carbon dioxide dissolved in the sludge was added to the volume of gas calculated at standard temperature and pressure from the gas pressure alone. The gas production based on the quantity of volatile matter added and destroyed was calculated by dividing the gas production for a particular day by the pounds of volatile solids added and destroyed in that day. An IBM 7074 computer was used to calculate the gas corrections. The program was written in Fortran II language and is shown in Figure 34. All gas production and gas quality data reported in this dissertation represent the total production of both methane and carbon dioxide during the run, including the carbon dioxide in solution in the sludge itself. The data shown in the Appendix in Tables 27, 28, 29, 30 and 31 are the corrected data.

For example, Table 28 indicates that on days 6 and 52 during Run 3, the gas productions in digester 3 were 6.5 and 7.7 cu ft per lb volatile solids added respectively. The following data are the raw data for digester 3 on days 6 and 52 (Table 4a). The quantity of carbon dioxide dissolved in the sludge was 12.2 percent of the total gas volume when the pressure was approximately 9 centimeters of mercury and almost 13 percent when the pressure was approximately 81 centimeters of mercury. At both pressures the quantity of carbon dioxide dissolved in the sludge was a significant proportion of the total gas volume.

Table 4a. Raw data for digester 3 on day 6 and 52

Day	Manometer reading[a]	Gas volume[b]	Volume correction[c]	Total gas volume[d]
6	8.9	0.058	0.008	0.066
52	81.5	0.526	0.078	0.604

[a] Centimeters of mercury.

[b] Calculated gas production: V_P $(4.14 \times 10^{-4})(P_M)(V_D)$ cu ft.

[c] Calculated volume of carbon dioxide dissolved in sludge: V_{CO_2} $(3.34 \times 10^{-8})(P)(R_{CO_2})(W)$ cu ft.

[d] Sum of calculated gas volume and calculated carbon dioxide volume correction.

The calculation of gas production based on the volatile solids added to the digester is indicated below.

Table 4b. Example of gas production calculations

Day	Volatile solids added (lb)	Gas production[a]
6	0.0101	6.5
52	0.0785	7.7

[a] Cu ft per lb volatile solids added. Calculated by dividing total gas volume by lb volatile solids added.

6. Gas analysis

a. General background Numerous authors (61, 64, 66, 85, 97, 124) have contributed to the present state of knowledge regarding gas-liquid partition chromatography. In the last decade, gas-liquid partition chromatography (GLPC) has developed into an extremely valuable analytical tool. Grune *et al.* (66) showed that gas analysis is able to detect digester changes with greater sensitivity than many other parameters such as: pH; electrolytic conductivity; oxidation-reduction potential; and volatile acids concentration.

Three principle methods can be employed in gas chromatography, namely, frontal analysis, displacement analysis and elution. In this study only elution was considered for digester gas analysis as it is the only method which will, under favorable conditions give separate peaks with negligible overlap for each component of a sample mixture, (Figures 8, 9, 10). Negligible overlap of the components leaving the partition column was required in this study so that true peak heights could be determined. This is not necessary if one has available the apparatus for analyzing compounded peaks.

The process of elution may be clarified by considering a column packed with an adsorbent over which a stream carrier gas C is passed. A volatile or gaseous sample which consists of components A and B is injected into the gas stream.

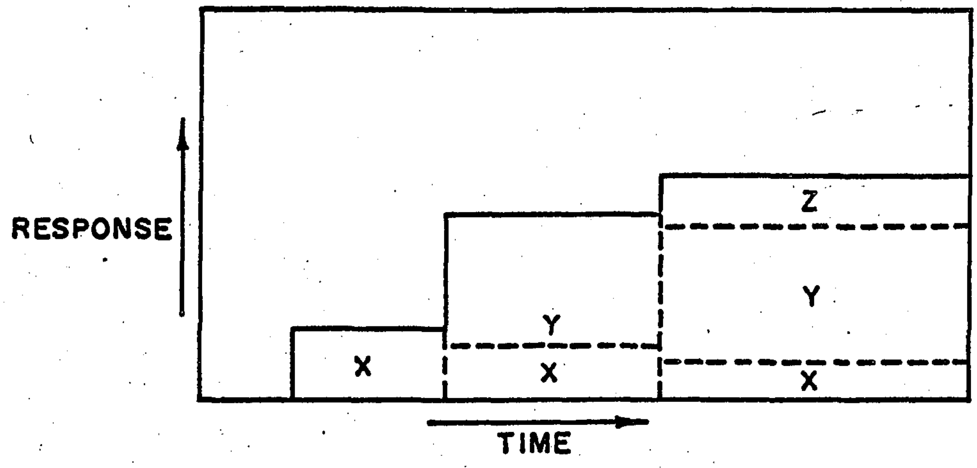
Considering an element of the column, some molecules of component A will be retained by the adsorbitivity of the column packing. Component A molecules will pass from the gaseous phase to the column packing only during the period that the concentration of component A in the gaseous phase is greater than that on the column packing. As the carrier gas moves unadsorbed gas A along the column, the concentration of A in the carrier gas will become less than the concentration of A in the element of packing material. At this time molecules of component A will pass from the column packing back into the carrier gas C. Eventually, since this is a continuous process, a state of equilibrium will be reached in the column where the rate of adsorption of component A on the packing material will be equal to the rate at which component A reverts to the gaseous phase. At equilibrium, a fraction of component A, X_a, is adsorbed in the stationary phase. The result is a probability equal to $(1-X_a)$ for each molecule of A to stay in the gas phase and keep moving. In a finite time interval, component A will move along with carrier gas C only $(1-X_a)$ of the total time.

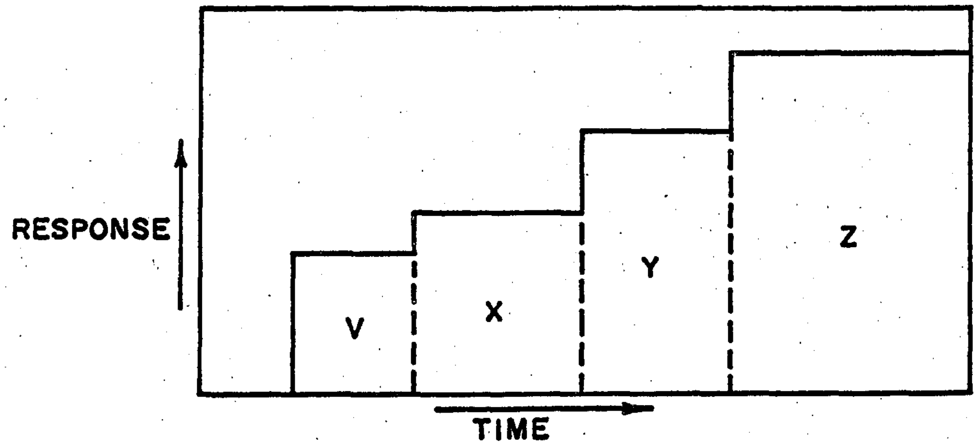
Similar reasoning can be applied to component B which will only move along with the carrier gas C a fraction $(1-X_b)$ of the total time. If X_a is larger than X_b then component B will emerge from the column sooner than component A. The difference between X_a and X_b will indicate the degree of separation to be

Figure 8. Chromatogram for ideal frontal analysis

Figure 9. Chromatogram for ideal displacement analysis

Figure 10. Chromatogram for ideal elution analysis


RESPONSE
TIME
X
Y
X
Z
Y
X


RESPONSE
TIME
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X
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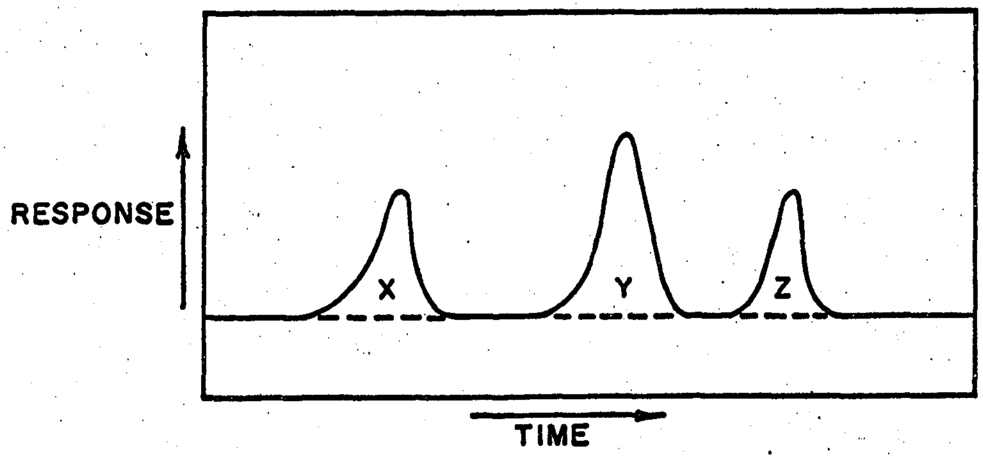
Z


RESPONSE
TIME
X
Y
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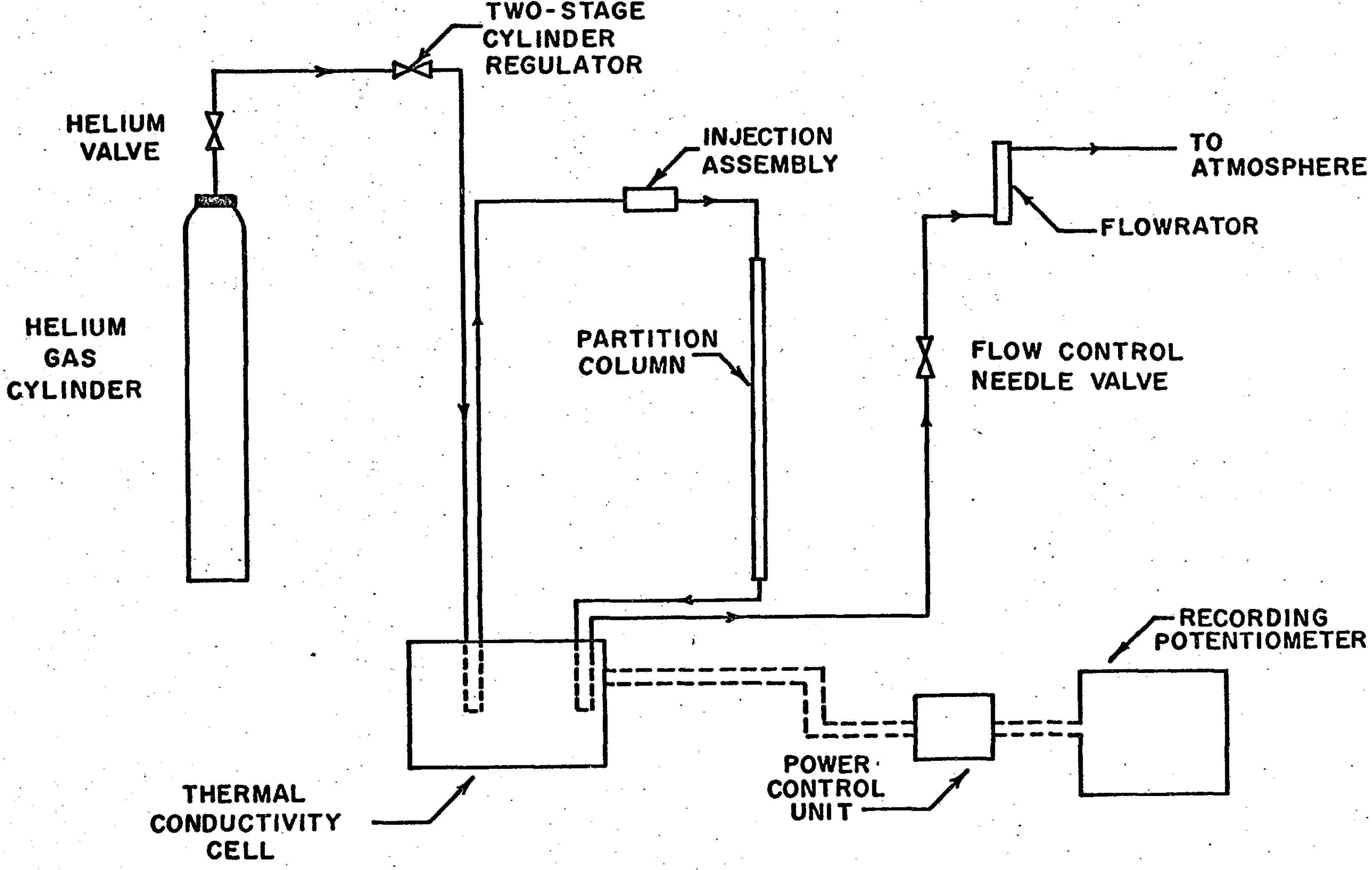
expected. The gaseous components are identified qualitatively by their retention times in the column. Quantitative identification of the components can be made using several methods. Thermal conductivity measurements are predominant at the present time. Several factors influence the degree and type of separation of the components of a gas mixture in GLPC. Among these are the column dimensions, the nature of the carrier gas, the gas flow rate, the adsorbent and support materials in the column, the temperature of the column and the method in which the sample is introduced into the column.

b. Apparatus The system used for analysis is shown schematically in Figure 11. Figure 12 shows the actual system used. The flow pattern of the helium carrier gas will be used to describe the system. Immediately upon leaving the storage bottle, where a two stage cylinder regulator controlled the pressure in the system, the carrier gas passed through a Gow-Mac, TR-11-B temperature regulated thermal conductivity cell. Power was supplied to the thermal conductivity cell by a Gow-Mac, Model 9999-D power supply unit. A temperature of 260° F was maintained in the thermal conductivity cell and a current of 230 ma was applied to the detectors. The helium leaves the cell, passes a sample injection assembly and flows into the partition column. A tee-joint sealed with a self-sealing rubber disc formed the injection assembly. The carrier gas and sample components upon leaving the column pass

Figure 11. Schematic diagram of gas analysis apparatus


TWO-STAGE CYLINDER REGULATOR
HELIUM VALVE
INJECTION ASSEMBLY
TO ATMOSPHERE
FLOWRATOR
HELIUM GAS CYLINDER
PARTITION COLUMN
FLOW CONTROL NEEDLE VALVE
RECORDING POTENTIOMETER
POWER CONTROL UNIT
THERMAL CONDUCTIVITY CELL

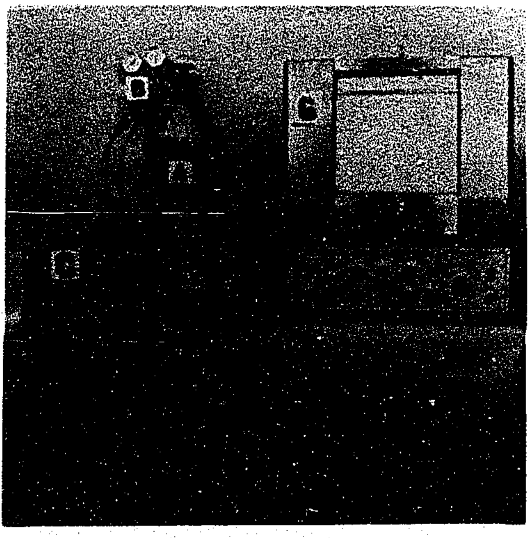
through the thermal conductivity cell. In its simplest form, the thermal conductivity cell consists of two heated sources arranged in a Wheatstone bridge circuit with two reference resistors and trim resistors provided for final balancing of the bridge (124). One source, as previously mentioned, is kept in the pure carrier gas. The other source is placed in the effluent gas from the chromatographic column. In this way, the effects due to the carrier gas are nullified. As the gas flows past the heated sources the heat conducted away is dependent on the thermal conductivity of the gas. The electrical resistance of the source is in turn a strong function of its temperature. Hence, if one source is in the carrier gas and the other is in the carrier gas plus sample components, then a state of unbalance will exist in the bridge. This will be noticed as a signal sent to the recorder. The recorder used in this study was a Sargent recording potentiometer, Model MR. A 1/4-inch flow control needle valve is situated in the gas line following the thermal conductivity cell. To measure the carrier gas flow rate, a flowrator or rotameter was placed after the needle control valve. The carrier gas is released to the atmosphere after leaving the flowrator. All the gas lines were 1/4-inch O.D. copper refrigeration tube and all connections were made with Swagelock gas-tight fittings.

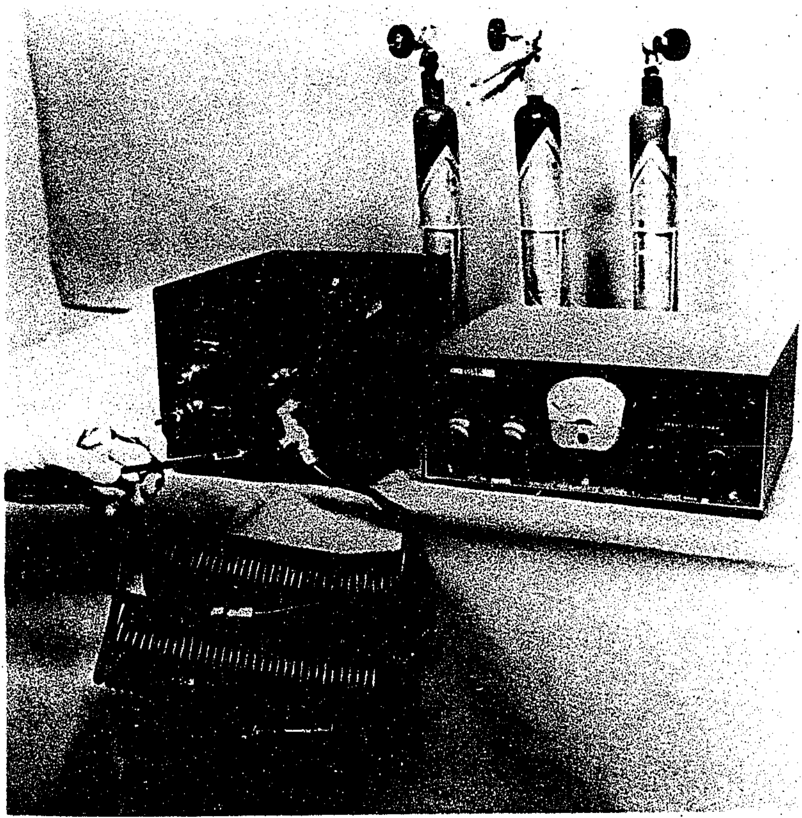
c. Preparation of the chromatographic column Grune et al. (61, 66) constructed a chromatographic column using

Figure 12. Gas analysis apparatus

A. Helium gas cylinder
B. Two stage cylinder regulator
C. Temperature regulated thermal conductivity cell
D. Sample injection assembly
E. Partition column. (This diagram shows a column made from copper refrigeration tubing. A stainless steel column which is physically similar was used in this study.)
F. Power control unit
G. Recording potentiometer

Figure 13. Injecting a gas sample from a digester into the chromatographic apparatus.





silicone-grease coated firebrick which gave exceptionally good separations of carbon dioxide, air, methane and hydrogen sulfide. Based upon their experience, the chromatographic column used in this study was prepared in the following manner.

An amount of Dow-Corning silicone stop-cock grease, sufficient to give a ratio of 40 parts grease to 100 parts of support material by weight, was dissolved in carbon tetrachloride. The support material, which consisted of C 22, 28-40 mesh crushed firebrick, was poured into the grease solution. The grease solvent was evaporated in a hood under heat lamps, using a fan to minimize the risk of toxic effects. When most of the carbon tetrachloride had evaporated, it was necessary to stir the mixture continuously to ensure a uniform coating of the grease over the surface of the brick. After complete evaporation of the carbon tetrachloride, the grease covered brick was dried at 100°C for 24 hours in a temperature controlled oven. When dry, the brick felt as though it were uncoated when held lightly between the fingers. If pressure was applied by the fingers, the grease was noticeable, although even under this condition the brick particles did not adhere to each other. The coated brick was packed in stainless steel tubing, 1/4-inch O.D. and 44 ft long. To make handling of the column more convenient it was coiled.

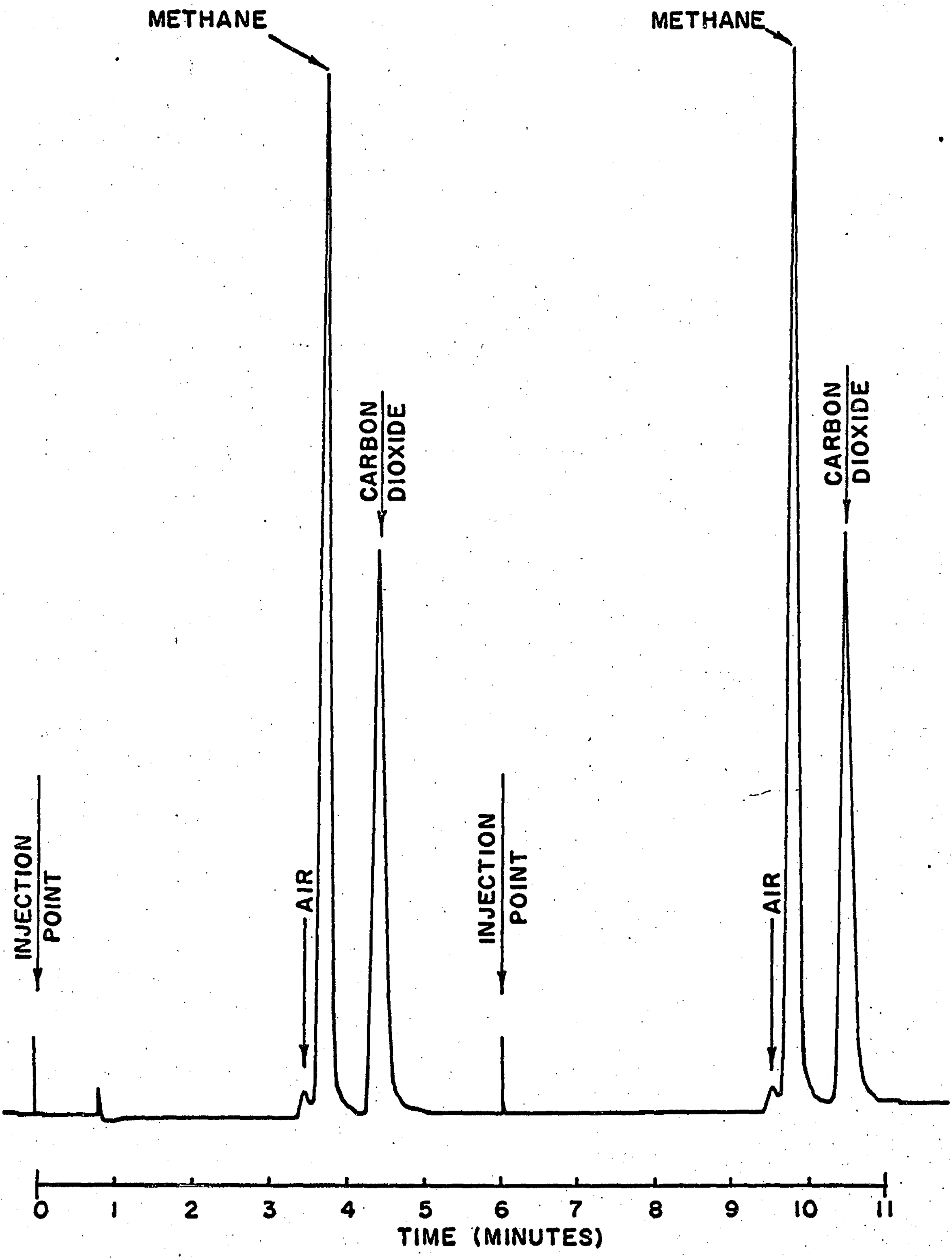
Difficulty was experienced when dissolving the silicone grease in carbon tetrachloride. Methylene chloride, although not used in this study, proved to be a much better solvent for the silicone grease. The original intention of this work was to use a 75-foot column for gas analysis. Tests showed that the desired carrier gas flow rate of 100 ml/minute could not be maintained unless high gas pressures were used in the system. This gave rise to sample injection problems and for this reason the length of the column was reduced to 44 ft. The separation peaks were not quite so pronounced but they were satisfactory.

d. <u>Procedure</u> Calibration curves were plotted for methane, air and carbon dioxide. Analytical grade methane and carbon dioxide, and atmospheric air were used in the calibration procedure. Samples of these gases were injected into the chromatography apparatus with a Hamilton Model 1001 "Microliter" hypodermic syringe. Plots were made of sample size versus peak heights obtained on the recorder. Samples of nitrogen and oxygen were placed in the column for analysis and their elution times were found to be almost identical. The air samples which had been analyzed previously showed a single peak, the elution time of which was the same as that for nitrogen and oxygen. The lack of time difference for the elution times of nitrogen and oxygen explains the single peak obtained for air. To analyze the digester gas, the hypodermic needle was pushed through the serum stopper on a digester. The syringe was flushed several times with the digester gas and then a 300 microliter sample was extracted. Care was taken in transferring the sample to the injection assembly on the chromatography apparatus to avoid contamination of the sample with air. The sample was injected into the carrier gas stream (Figure 13). The resulting increase of gas pressure in the system caused a blip on the recorder chart. As the component gases of the sample mixture passed out of the partition column into the thermal conductivity cell, traces were produced on the recorder chart. These traces were related to the injection blip and in this way the sample components could be identified. A typical digester gas analysis is shown in Figure 14. Peak heights produced by the sample components were taken from the recorder chart and, using the calibration curves, the actual volume of each component in the original sample was determined. The values were converted to a percentage basis.

7. Qualitative volatile acid analysis

The importance of the type of volatile acid present in a digester has been discussed in the Literature Review. In this study an attempt was made to determine qualitatively and quantitatively the volatile acids in the digesting sludge. Chromatography columns were made up according to the specifications of two authors (82, 132) who had succeeded in determining volatile acids both quantitatively and qualitatively. Although these columns were first tested on a GLPC thermal conductivity rig and then on a GLPC flame ionization rig, no identification

Figure 14. Recording of a typical daily gas analysis


METHANE
METHANE
CARBON DIOXIDE
CARBON DIOXIDE
INJECTION POINT
AIR
INJECTION POINT
AIR
0
1
2
3
4
5
6
7
8
9
10
11
TIME (MINUTES)

qualitatively or quantitatively, could be obtained because of the masking effects of water. According to a private comunication the results obtained from GLPC experiments reported in the literature can not always be reproduced due to the omission in the written report of some factor or factors which affect the experiments. For this reason, ascending technique paper chromatography was used to examine the volatile acids in the digesting sludge. Only qualitative determinations were made because of the difficulty in obtaining accurate quantitative results with paper chromatography.

The method used was developed by Buswell et al. (19) following experiments by Hiscox and Berridge (83). A butanol-ethylamine eluent was prepared by shaking one part by volume of 2.0 N ethylamine solution with four parts by volume of n-butanol and allowing it to stand until it separated into two layers. The upper layer was used as the carrier phase. This author obtained better results using a 1.5 N ethylamine solution. Test solutions of sodium formate, acetic acid, propionic acid, butyric acid, iso-butyric acid and valeric acid were made up at concentrations of 2000 mg/l. Whatman No. 1 chromatographic filter paper in strips approximately 18 inches long and 1-1/2 inches wide were spotted with 20 microliters of the test solutions and allowed to dry. When dry, they were placed in glass cylinders containing the butanol-ethylamine eluent and were sealed at one end and stoppered at the other as shown in Figure 15. The strips were hung from hooks fixed in the rubber stoppers such that approximately one inch of the strip was submerged in the eluent. The spots had been positioned on the papers so that they were approximately one inch above the eluent surface. After four hours the strips were removed, dried, and sprayed with a 0.4 percent solution of brom cresol green indicator in 95 percent alcohol. The spots on the paper and the height of the carrier liquid were marked. The Rf values of the acids were determined by measuring the distances the centers of gravity of the spots had moved compared to the distance the eluent front had moved. The Rf values of the acids are given in Table 9. Unknown samples from the supernatant of centrifuged digesting sludge were treated in a similar manner and the acids present were identified by their Rf values. Care was taken in all of this work to avoid touching the chromatography paper with the hands. There are volatile acids present on the skin and these will produce spots on the paper. Forceps were used to handle the paper at all times.

8. Dewatering test

The ability of a sludge to be dewatered is an important consideration in the digestion of sewage sludge. Frequently, the only method for dispersing of digested sludge available is to dry the sludge and either sell or give away the dried product, burn the sludge or bury the sludge. All of these processes are benefited by having the final sludge as dry as possible.

A test which has been used frequently to test the dewatering characteristics of sludges treated with chemicals is the filterability test. Originally a sample of sludge was placed in a Buchner funnel and a vacuum was applied. The time required for a crack to develop in the filter cake in the funnel was noted and taken as a measure of the dewatering ability of the sludge. Coackley and Jones (33) introduced a concept of specific resistance which did not depend upon the initial solids content of the sludge, the volume of sludge being filtered, the area of the filtering surface or the pressure at which the filtration was carried out. They developed further work by Ruth (146) and Carmen (28, 29, 30) and found that the concept of specific resistance was a useful measure of the dewatering ability of sludge. The equation given by Coackley <u>et al</u>. for specific resistance is shown.

$$r = \frac{2PA^2}{\mu C} . b$$

where r is specific resistance in cm per gram

P is the pressure of filtration in gram per sq cm

A is the filter area in sq cm

μ is the filtrate viscosity in poise

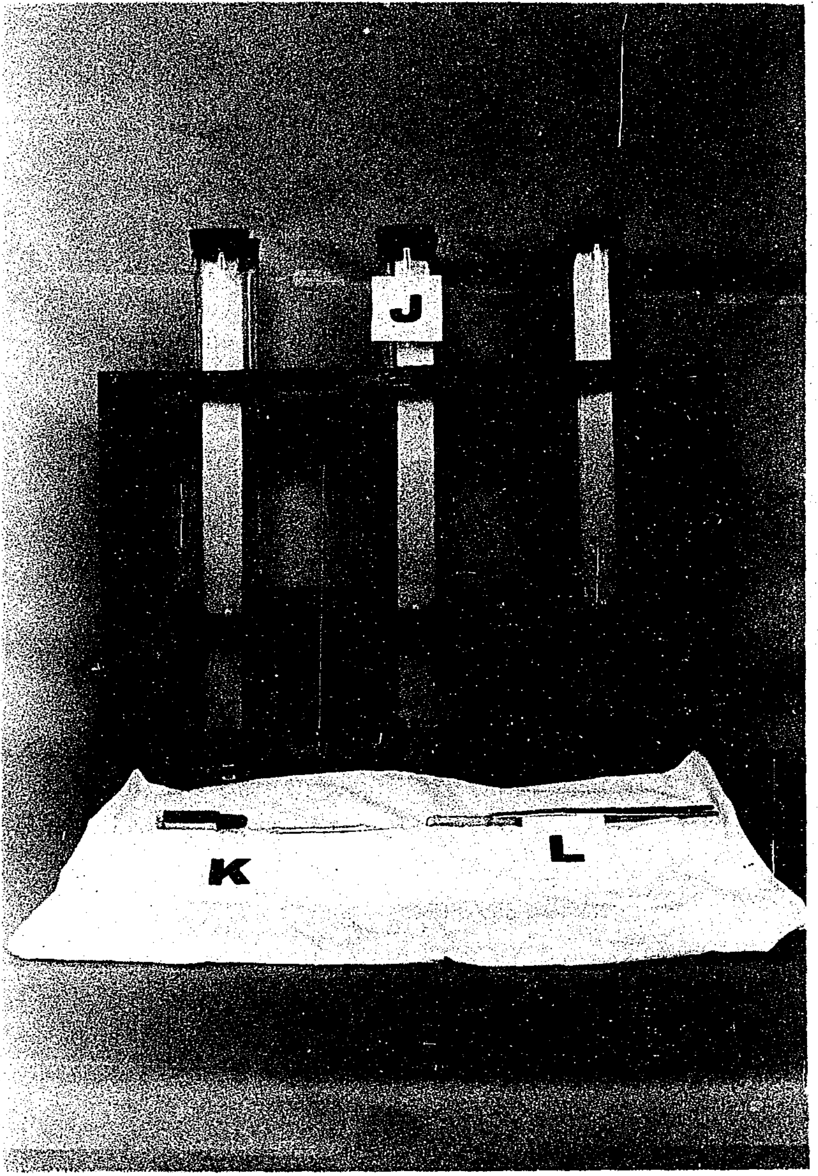
C is the solids content of the sludge in grams of solid per ml of filtrate

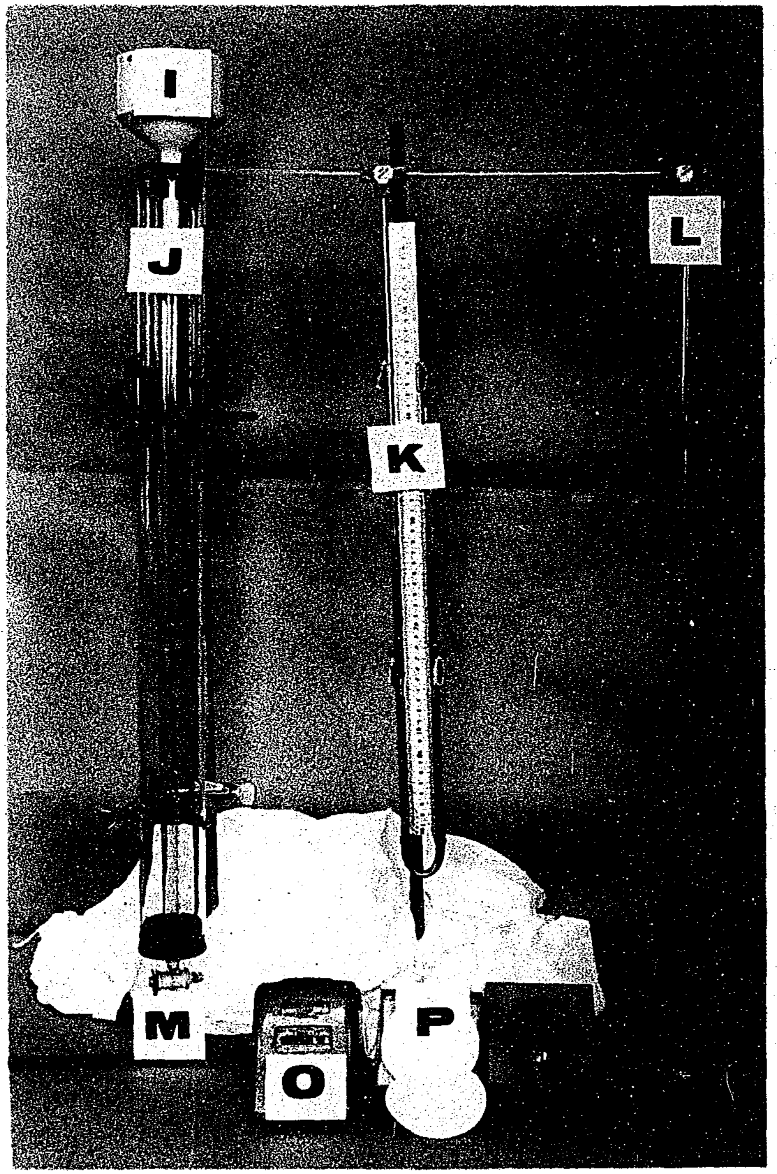
Figure 15. Apparatus for determining individual volatile acids.
(left)

J. Tubes containing chromatography paper and eluent.
K. Micro-pipette.
L. Forceps for handling chromatography paper.

Figure 16. Sludge dewatering apparatus.
(right)

I. Buchner funnel.
J. Vacuum cylinder containing burette.
K. Manometer.
L. Tubing leading to a source of vacuum.
M. Drain for burette.
O. Electric timer.
P. Whatman No. 1 filter paper.


J
K
L


I
J
L
K
M
O
P

b is the slope of the line obtained if the time divided by the filtrate volume at that time is plotted against the filtrate volume itself.

The units are sec per ml^2.

Rich (131) presented the same equation in terms of the English system of units.

The specific resistance test was made using a system in which a known vacuum could be applied to a Buchner funnel containing Whatman No. 1 filter paper and a sludge sample. The filtrate volume was measured and recorded with respect to time. Figure 16 shows the apparatus used.

The complete procedure for the test is outlined below:

1. A disc of Whatman No. 1 filter paper was placed in the funnel and wetted down with distilled water.
2. The funnel was placed on the apparatus and the excess water was drawn out of the filter paper by applying a small vacuum for a short time.
3. The pressure at all parts in the system was made atmospheric and 100 ml of sludge were added to the Buchner funnel.
4. The temperature of the sludge was recorded.
5. A valve, not shown in Figure 16, which was used to control the vacuum to the apparatus was closed. A vacuum was applied such that a vacuum of 52 centi-

meters of mercury was built up over a period of approximately four minutes and 15 seconds. Different results were obtained if the time of building up the vacuum was changed.

6. When the vacuum reached 52 centimeters of mercury, a stopwatch was started and the volume of filtrate in the burette was noted.
7. Readings of filtrate volume were made every minute for 15 minutes.
8. A graph of time divided by filtrate volume versus filtrate volume was plotted. The slope of the resulting line on this graph was determined.
9. The specific resistance of the sludge sample was determined by substituting the slope of the graph in the equation and determining the other values by measurement or with the use of tables.

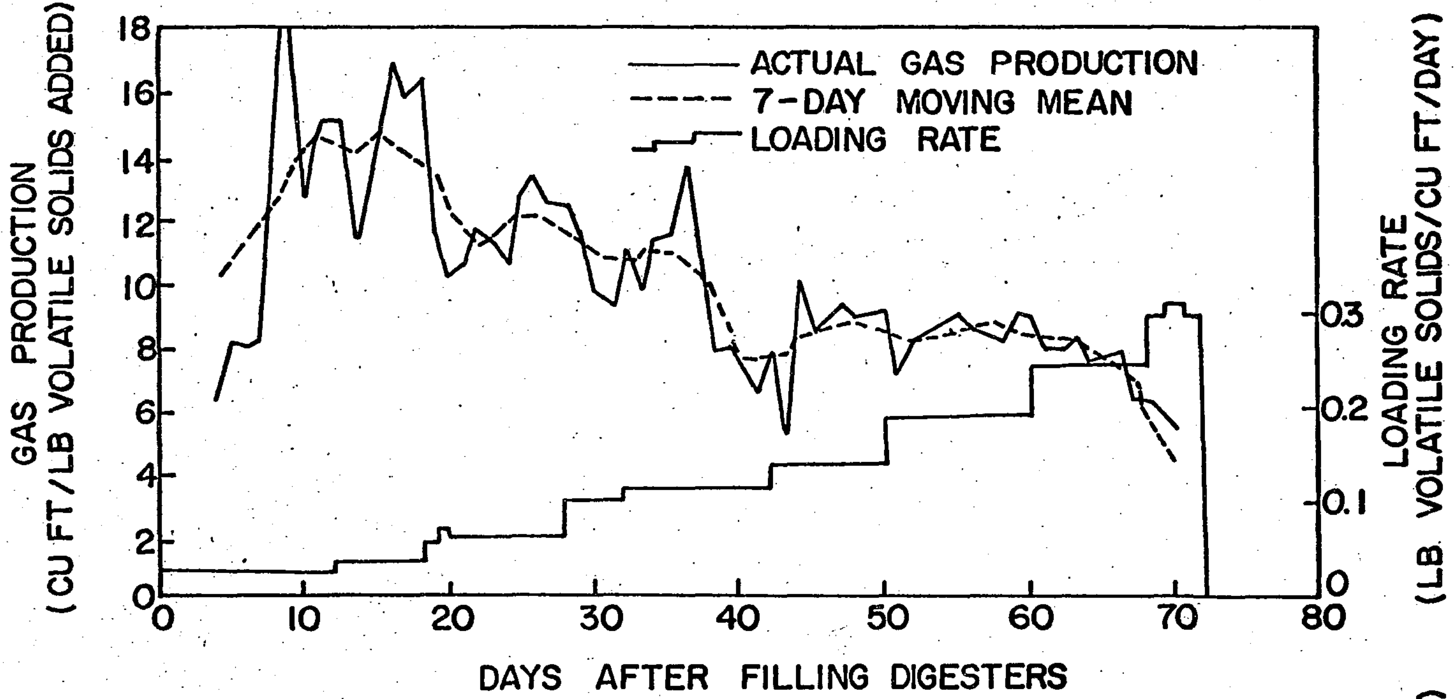
V. ANALYSIS AND DISCUSSION OF TEST RESULTS

Data were collected in this study to determine the effect of the concentration of solids in a digester on the anaerobic digestion process. As previously mentioned, the most reliable indication of the state digestion was believed to be the gas production, expressed in terms of cu ft of gas produced per lb volatile solids added to the digester per day. The gas production based on the volatile solids added and destroyed each day were calculated and plotted. Figure 17 is a typical plot of gas production in digester 2 based on the volatile solids added. The loading rate to the digester each day is also shown in Figure 17. The variations in volatile acids and volatile solids reduction during a run were also plotted (Figure 18). Complete data for Run 2 and Run 3 are included in the Appendix.

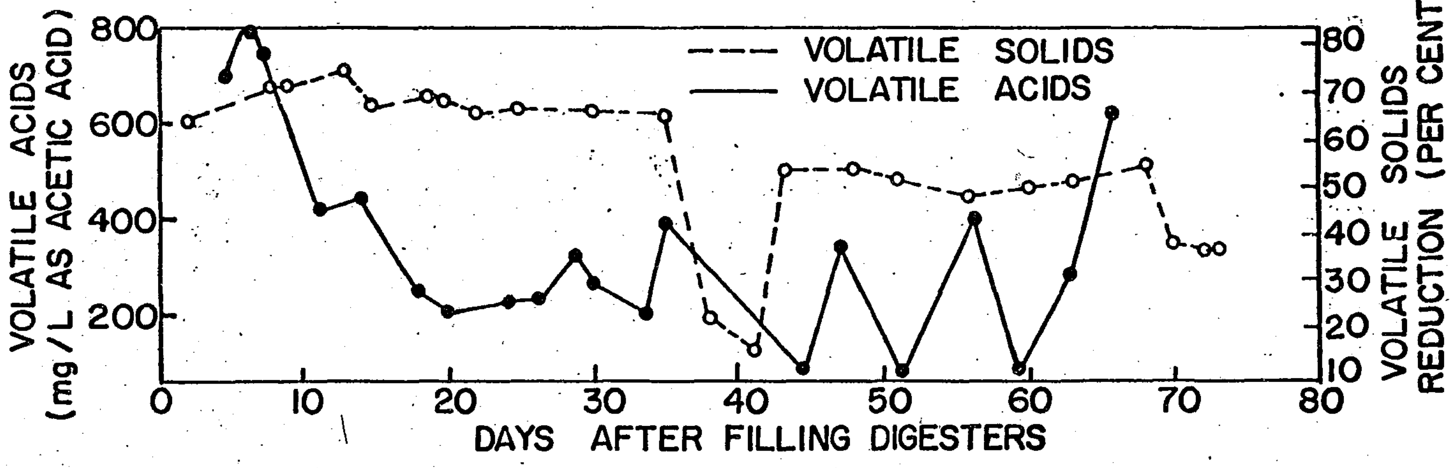
All conclusions are based on data included in the tables in the Appendix. In most cases, the data were extracted when stable conditions, as evidenced by a period of uniform, gas production, existed in the digesters. The data for each day within a particular period were averaged over the length of the period. Typical graphs such as are shown in Figure 17 and 18 were used to choose the periods when stable conditions existed. The gas production based on the volatile solids destroyed in the digesters was calculated for each day of operation during Run 3. However, because of the small change in

Figure 17. Gas production and loading rate for digester 2, Run 3

Figure 18. Volatile solids reduction and volatile acids concentrations in digester 2, Run 3



ACTUAL GAS PRODUCTION
7-DAY MOVING MEAN
LOADING RATE
GAS PRODUCTION (CU FT/LB VOLATILE SOLIDS ADDED)
0
2
4
6
8
10
12
14
16
18
LOADING RATE (LB VOLATILE SOLIDS/CU FT/DAY)
0
0.1
0.2
0.3
0
10
20
30
40
50
60
70
80
DAYS AFTER FILLING DIGESTERS



VOLATILE SOLIDS
VOLATILE ACIDS
VOLATILE ACIDS (mg/L AS ACETIC ACID)
200
400
600
800
VOLATILE SOLIDS REDUCTION (PER CENT)
10
20
30
40
50
60
70
80
0
10
20
30
40
50
60
70
80
DAYS AFTER FILLING DIGESTERS

weight of the digesters due to volatile solids reduction and the relatively inaccurate method of weighing the digesters, the calculated gas productions based on volatile solids destruction were not accurate. For example, suppose 500 gm of sludge containing 8 percent total solids and 75 percent volatile solids were added to a digester and suppose the volatile solids reduction (see section V, B) was 80 percent.

Weight of volatile solids added to the digester

$= (500)(0.08)(0.75)$

$= 30$ grams.

If 80 percent of the volatile solids are destroyed the change in weight of the digester will be 24 grams. The digesters, including the digesting sludge, weighed approximately 23,000 grams and during operation they were weighed to 50 grams. For this reason, gas production data based on volatile solids destruction are not presented in this dissertation.

A summary of the data averaged over periods of reasonable uniform digestion periods (from Tables 18, 20, 22, 24, 26, and 28) is presented in Table 5. The usable data from Run 2, because of unstable conditions inside the digesters, were not extensive and are not presented in Table 5. The author believes this was due to an initial overloading of the digesters such that a high volatile acids concentration accumulated. The organisms in some of the digesters were inhibited. For this reason the following discussion will apply to results from Run 3 unless otherwise stated.

Where loading rate studies have been reported in the literature, it is usual to find the total solids concentration in the feed to a digester given as the parameter describing solids concentration. In this study, the total solids concentration of the digesting sludge was thought to be a more informative parameter. To correlate the data presented in this dissertation with that of other experimenters, a graph was drawn to relate the concentration of total solids in the feed sludge to the concentration of total solids in the digesting sludge (Figure 19). The data were obtained from Tables 14 and 18. It was noticed that groups of points were obtained on the graph at particular raw sludge and digesting sludge solids concentrations. The groups of points were replaced by representative points. This accounts for the relatively few plotted points in Figure 19 as opposed to the more numerous data in Tables 14 and 18. A straight line relationship was obtained. If T_F is the feed solids concentration and T_D is the digesting sludge solids concentration; the relationship between them was found to be:

$$T_F = 1.23\ T_D.$$

Figure 20 was plotted to show the relationship between total solids times volatile solids in the raw and digesting sludges in Run 3. The figure is used later in the discussion of the results in the section concerned with loading rates.

Table 5. Summary of results, Run 3

Digester	Days Averaged	Loading Rate[a]	Detention Time (days)	Solids[b]		Volatile Solids Reduction (percent)	Gas Production[c]
				Total	Volatile		
1	22-28	3.4	34.6	2.00	43.4	76.7	11.7
	29-49	6.1	17.7	1.90	47.5	62.1	8.9
	50-59	9.4	11.4	1.80	52.4	61.2	8.3
2	20-28	6.8	34.4	3.60	52.0	67.2	11.6
	30-37	11.3	20.5	3.45	52.8	66.2	10.8
	44-49	13.7	15.9	3.55	55.1	53.8	9.1
	50-59	19.0	11.4	3.55	56.5	50.9	8.3
	60-66	24.5	8.9	3.55	57.5	50.4	7.9
3	25-29	10.6	34.2	6.15	47.4	71.1	11.3
	30-38	17.7	20.2	5.70	48.3	70.2	9.1
	44-49	19.5	15.9	6.00	50.2	56.0	8.3
	51-59	28.1	11.4	5.85	51.4	60.5	7.7
	60-66	36.6	8.8	5.50	52.7	59.8	7.1
4	22-28	12.4	34.2	7.50	46.6	67.0	10.5
	31-38	21.1	19.1	7.30	45.4	71.6	8.3

---

[a] Loading rate (lb volatile solids added per cu ft per day x 10^{-2}).

[b] Digesting solids concentration (percent), total and volatile.

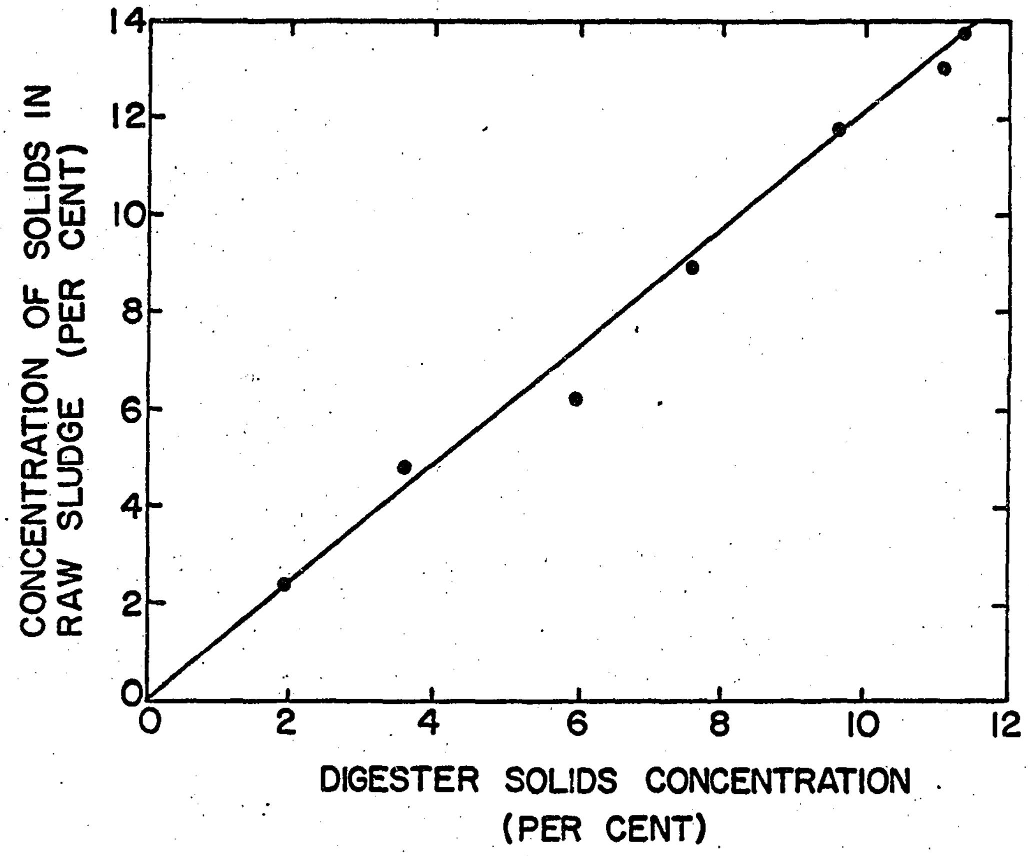
[c] Gas production in cu ft per lb volatile solids added.

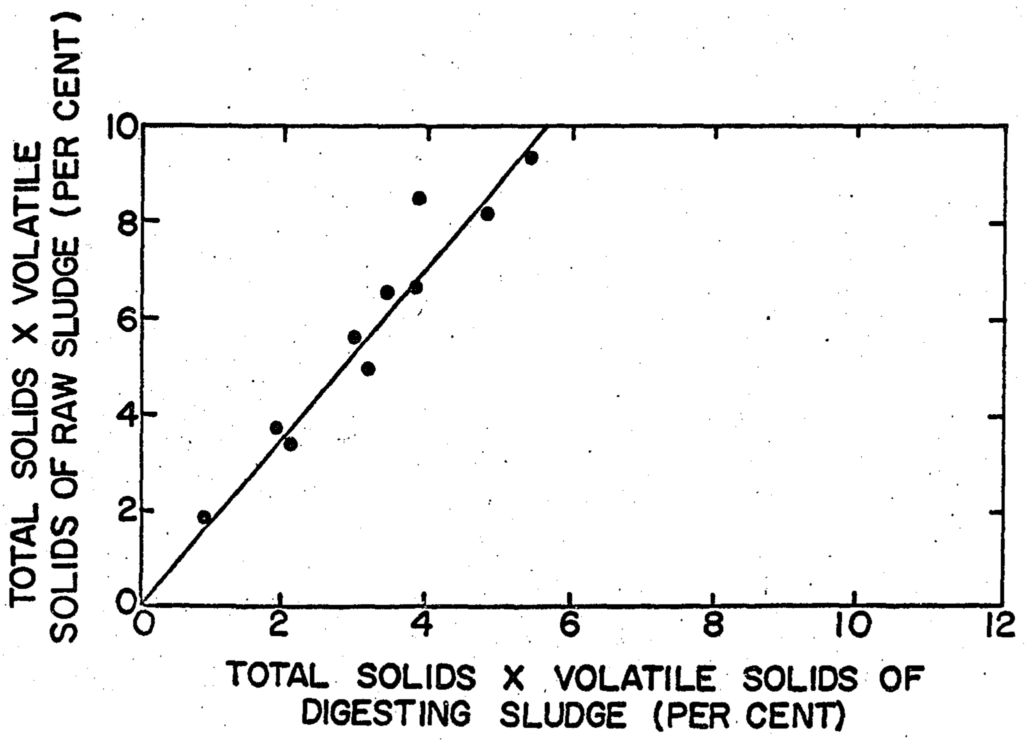
Table 5. (Continued)

Digester	Days Averaged	Loading Rate[a]	Detention Time (days)	Solids[b]		Volatile Solids Reduction (percent)	Gas Production[c]
				Total	Volatile		
4	44-50	24.3	15.9	7.69	49.6	52.8	7.8
	51-66	37.4	11.4	7.22	51.2	59.6	7.2
5	16-20	9.0	54.1	8.50	44.6	70.5	10.5
	20-24	10.1	54.5	8.50	45.6	68.0	11.4
	25-28	13.2	41.6	8.50	45.1	68.5	10.8
	32-38	20.9	22.0	9.25	44.8	69.2	8.2
	44-49	30.8	15.9	9.65	46.6	48.3	6.8
	51-62	49.2	11.3	9.85	49.4	56.6	5.8
6	16-19	10.8	51.1	11.10	48.8	63.3	7.8
	21-24	11.9	51.3	11.10	48.6	61.6	8.0
	26-29	15.4	39.7	10.90	48.6	61.5	7.8
	33-39	24.2	23.7	10.95	48.6	61.7	6.8
	44-49	34.9	15.9	9.65	49.4	36.0	5.7
	51-66	55.8	11.3	9.85	52.6	50.9	5.0

Figure 19. Relationship between solids in raw and digesting sludges, Run 3

Figure 20. Relationship between total solids times volatile solids in raw and digesting sludges, Run 3


CONCENTRATION OF SOLIDS IN RAW SLUDGE (PER CENT)
0
2
4
6
8
10
12
14
0
2
4
6
8
10
12
DIGESTER SOLIDS CONCENTRATION (PER CENT)


TOTAL SOLIDS X VOLATILE SOLIDS OF RAW SLUDGE (PER CENT)
0
2
4
6
8
10
0
2
4
6
8
10
12
TOTAL SOLIDS X VOLATILE SOLIDS OF DIGESTING SLUDGE (PER CENT)

A. Gas Production

Theory indicates that an optimum solids concentration should exist in digesting sludge where gas productions per lb of volatile solids added to a digester are a maximum. For example, at zero percent solids concentration, there can be no gas production. If the solids concentration is low, the solids are widely dispersed and not readily available as food for the microorganisms in digestion. The resulting gas production will be low. Increasing the solids concentration will increase the availability of the solids to the microorganisms causing an increase in gas production. Eventually, some limiting factor such as inefficient mixing or insufficient dilution of metabolic end products will reduce the efficiency of the gas producing microorganisms. The rate of gas production will then decrease. If the solids concentration continues to increase the rate of gas production will be further decreased. Hence at some solids concentration a maximum rate of gas production should exist. In this section data have been analyzed to determine the relationship between gas production and solids concentration.

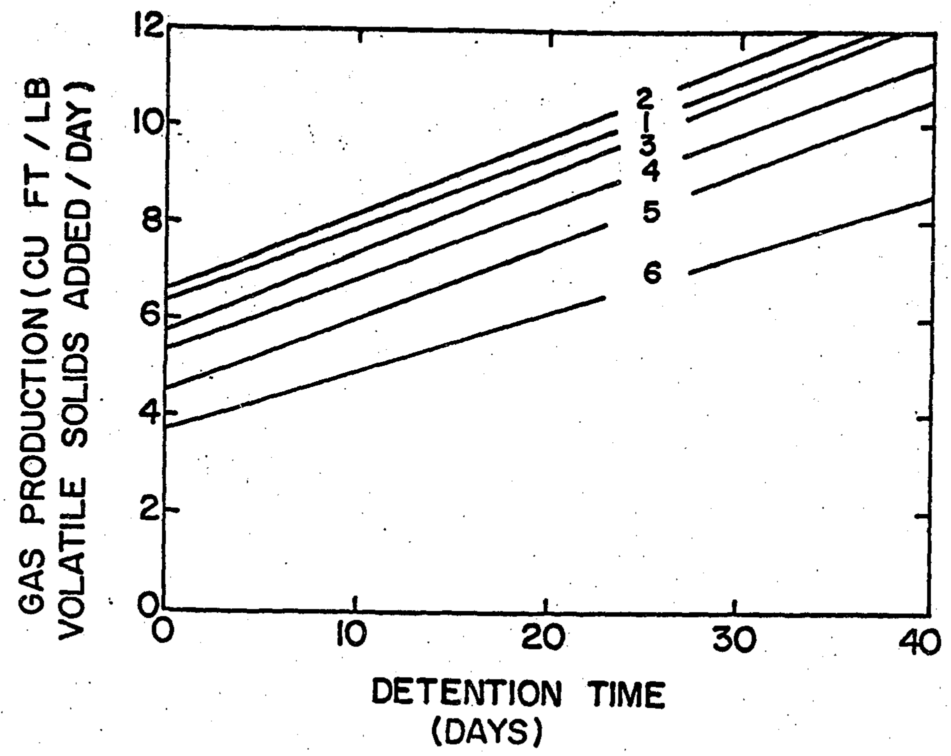
Gas productions based on volatile solids added to a digester were plotted against detention time for each digester using data from Table 5. The trends indicated by the data are shown in Figure 21. The graph indicates that the gas production increased as the detention time increased. The lines are approximately parallel, indicating that the increase in gas production produced by an increase in detention time is almost independent of the solids concentration of the digesting sludge. For example, the increase in gas production from the sludge in digester 5 when the detention time is increased from 10 days to 20 days is 1.5 cu ft. per lb of volatile solids added per day. The increase in gas production from the sludge in digester 3 for the same increase in detention time is 1.6 cu ft per lb of volatile solids added per day. The average solids concentration of the sludges in digesters 5 and 3 was 9.0 percent and 5.8 percent respectively (obtained by averaging the solids concentrations listed in Table 18).

The variations in the solids concentration in the digesting sludges with change in detention time were plotted in Figure 22. The data in Tables 18 and 24 were used to plot this graph. For example, Table 24 indicates that the sludge detention in digester 3 was 19.7 days on the 34th day of Run 3. Table 18 indicates that on the 34th day of Run 3 the solids concentration in digester 3 was 5.6 percent. The solids concentration (5.6 percent) was plotted against the corresponding detention time (19.7 days) in Figure 22 for digester 3. Figure 22 indicates that, with the exception of digester 5, the solids concentrations in the digesters were maintained relatively constant.

Figure 23 was constructed using data interpolated from Figures 21 and 22 and not data from Table 5 directly. The gas production in a particular digester for a specified detention time was taken from Figure 21 and plotted in Figure 23 against

Figure 21. Gas production for digesters 1 through 6, Run 3

Figure 22. Solids concentration in digester for digesters 1 through 6, Run 3


GAS PRODUCTION (CU FT / LB VOLATILE SOLIDS ADDED / DAY)
0
2
4
6
8
10
12
0
10
20
30
40
DETENTION TIME (DAYS)
2
1
3
4
5
6

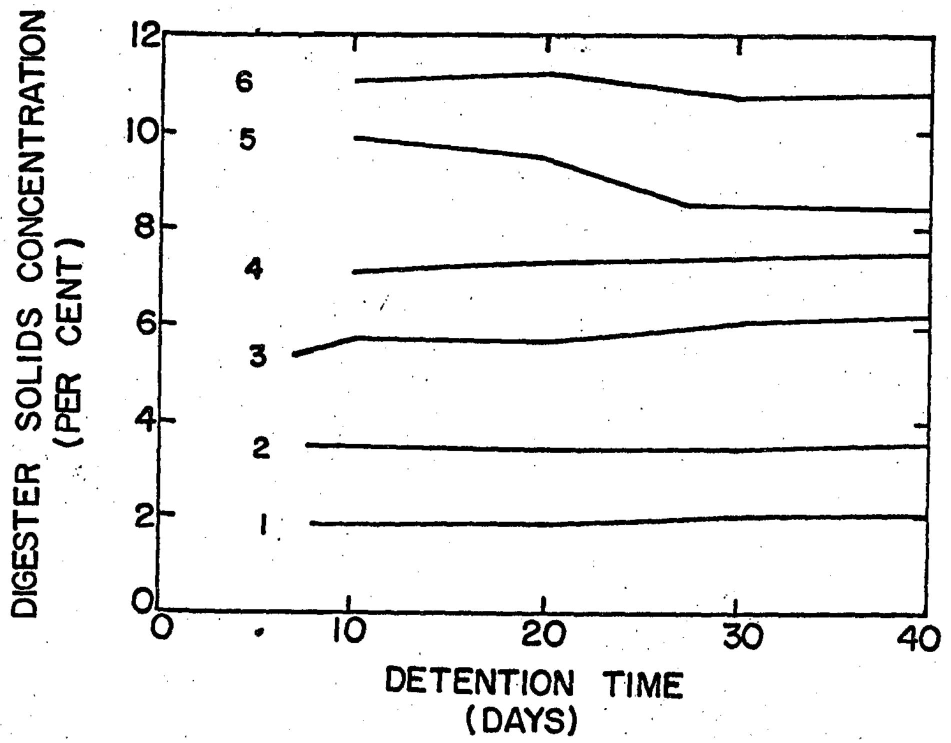
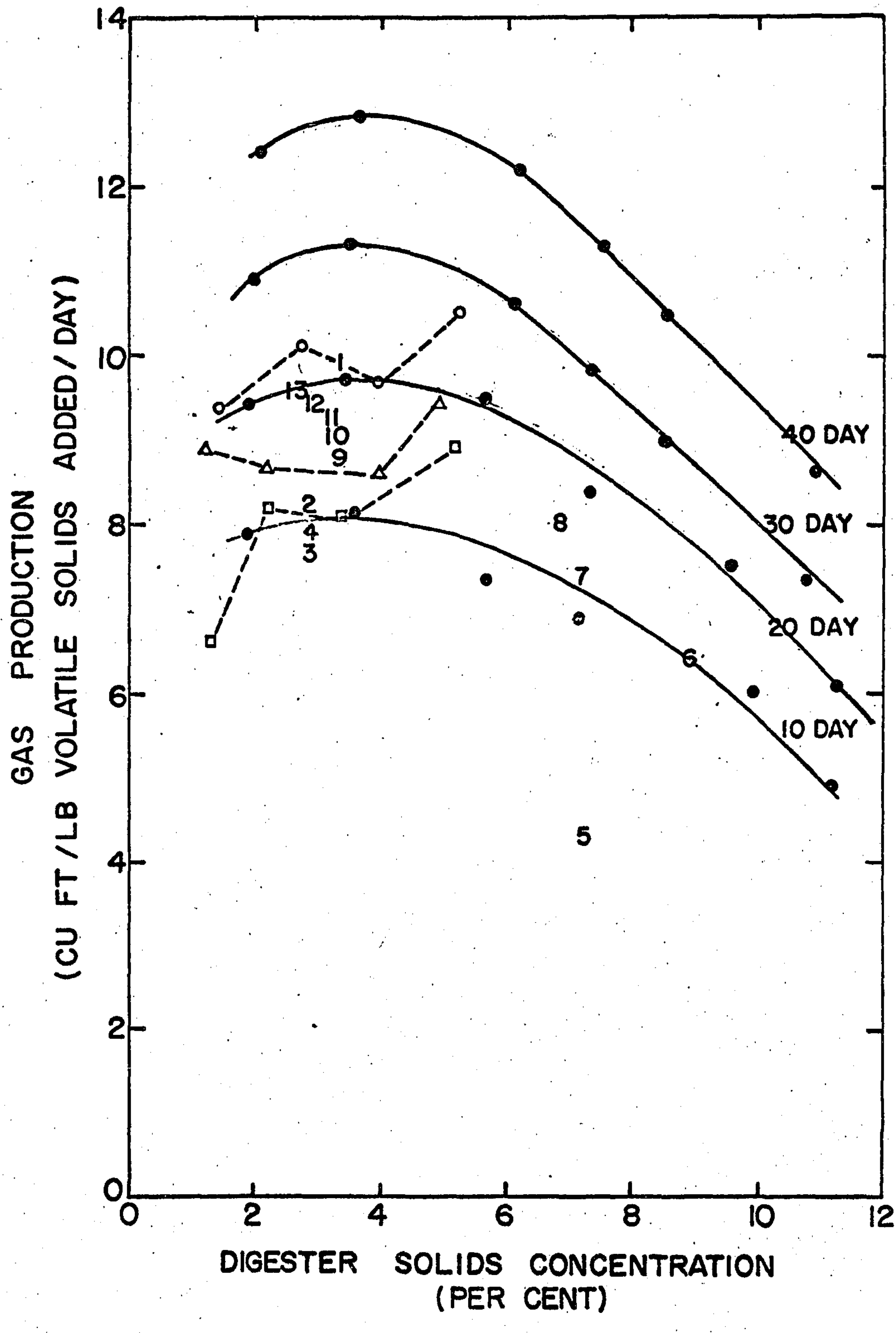

DIGESTER SOLIDS CONCENTRATION (PER CENT)
0
2
4
6
8
10
12
0
10
20
30
40
DETENTION TIME (DAYS)
6
5
4
3
2
1

Figure 23. Gas production versus digester solids concentration at various detention times, Run 3

Symbol[1]	Detention time (days)	Reference
●——●		Run 3
□---□	8	Sawyer and Schmidt (152)
△---△	11	(152)
○---○	14	(152)
1	14	Torpey (168, 169)
2	10	(168, 169)
3	8.3	(168, 169)
4	6.4	(168, 169)
5	3.2	(168, 169)
6	7	Morgan (116)
7	10	(116)
8	10	(116)
9	6	Sawyer and Roy (151)
10	8	(151)
11	10	(151)
12	15	(151)
13	20	(151)

---

[1] Each number is centered over the position of the actual data point.


GAS PRODUCTION
(CU FT / LB VOLATILE SOLIDS ADDED / DAY)
0
2
4
6
8
10
12
14
DIGESTER SOLIDS CONCENTRATION
(PER CENT)
0
2
4
6
8
10
12
40 DAY
30 DAY
20 DAY
10 DAY
1
2
3
4
5
6
7
8
9
10
11
12
13

the solids concentration in that digester for that detention time (Figure 22). Figure 23 shows the gas production versus the solids concentration in the digesters at detention times of 10, 20, 30 and 40 days. Maximum gas production was observed to occur with a digester solids concentration between 3 and 5 percent for detention times of 10, 20, 30 and 40 days. When the digester solids concentration was greater than about 5 percent, the gas production tended to decrease as the solids concentration increased. The relationship between decrease in gas production and increase in solids concentration in this portion of the graph was more of a straight line relationship than in the portion where the solids concentration was less than 5 percent. The decrease in gas production as the digester solids concentration increased from 6 to 10 percent was:

	Decrease in gas production	
Detention time (days)	Cu ft per lb volatile solids added	Percent of maximum gas production (Table 6)
10	2.2	22.2
20	2.3	23.7
30	2.6	23.5
40	2.7	21.1

The decrease in gas production for a change in the digester solids concentration from 6 to 10 percent was larger as the detention time of the sludge in the digester increased.

Three interrelated physical parameters control sludge digestion: loading rate, detention time, and solids concentration in the raw sludge.[1] This is discussed more extensively in section F dealing with loading rates. Any two of these parameters automatically determines the third parameter. For example, if the solids concentration is specified, as in this study, and the detention time is varied, then the loading rate varies inversely as the detention time. Alternatively, if the detention time is constant and the solids concentration varies, the loading rate varies directly as the solids concentration.

The results discussed above indicate that as the solids concentration is increased beyond the optimum, there is a decrease in gas production for a particular detention time. Loading rate may be substituted for solids concentration and the previous sentence would read: the results discussed above indicate that as the loading rate is increased above that associated with the optimum solids concentration, there is a decrease in gas production for a particular detention time. Whenever digestion results are being discussed and one of the parameters of loading rate, detention time or solids concentration is constant, the remaining two parameters are either directly or inversely proportional (see Equation 1 in Section F).

---

[1] There are actually four parameters involved, the fourth one being volatile solids concentration in the raw sludge. For the purposes of this discussion, the volatile solids concentration is assumed to be constant.

The conclusions drawn from the results of this study are:

1. Detention time has a definite effect on gas production. As the detention time increased, or the loading rate decreased, the gas production per lb of volatile solids added increased.
2. There is an optimum solids concentration in digesting sludge at which maximum gas production might be expected. In this study, the optimum solids concentration appeared to be between 3 and 5 percent.
3. As the digester solids concentration increases beyond the optimum concentration, the gas production decreases approximately linearly as the solids concentration increases. The longer the detention time, the greater is the decrease in gas production for the same increase in solids concentration.

The maximum gas productions for various detention times obtained in this and other studies are listed in Table 6.

Levels of gas production per lb of volatile solids added are about the same as those reported in the literature. For example, the gas production at detention times of 10 and 20 days may be compared as follows (Table 6):

Table 6. Maximum gas production, Run 3

Detention time (days)	Total solids (percent)	Gas[a] production	Reference
10	3.1	8.1	Run 3
20	3.2	9.7	
30	3.7	11.3	
40	3.75	12.8	
6	3.15	8.9	Sawyer and Roy (151)
8	3.05	9.15	
10	2.99	9.22	
15	2.92	9.56	
20	2.77	9.72	
7	8.6	6.6	Morgan (116)
10	7.2	7.5	
10	6.7	8.15	
14	3.3	9.9	Torpey (169)
10.3	3.0	8.3	
8.3	3.0	8.0	
6.4	3.0	8.2	
3.2	4.1	6.8	

[a] Cu ft per lb volatile solids added.

Detention time (days)	Gas production cu ft per lb volatile solids added	Reference
10	8.10	Run 3
10	9.22	(152)
10	7.5	(116)
10	8.15	(116)
10.3	8.3	(161) (169)
20	9.65	Run 3
20	9.72	(142) (152)

A wider difference in results of different studies might have been expected because of differences in the percentage of solids in the digester, in the composition of the sludge feed, different feeding procedures, and differences in the degree of mixing provided. The digesters used by Sawyer and Roy (151) and Morgan (116) were fed twice daily and the digester used by Torpey (169) was fed every two hours. In this study, the digesters were fed only once a day. Based on feeding schedule, the results in this study should perhaps have produced less gas. However, the degree of mixing was undoubtedly better than in the operation of any of the other digesters.

Data from other sources, including the data in Table 6, were also plotted in Figure 23. Each number represents a data point. The data of Sawyer and Schmidt had been interpreted by others as showing an increase in gas production as the solids concentration increased. This disagrees with the results obtained in this study. However, their data only covered a solids concentration up to 5.2 percent. The results of this study show that their work was undoubtedly in the region of an optimum solids concentration. The scatter in the data reported by Sawyer and Schmidt tends to invalidate any conclusions concerning a trend to an optimum but they could be interpreted to confirm the trends observed in this study.

Torpey's data (points 1, 2, 3, 4 and 5 in Figure 23) appear to confirm the data showing a decrease in gas production as the detention time decreases. Two of his data, points 3 and 4, appear to contradict this statement. Point 2 agrees with data obtained in this study. Point 5 cannot be correlated because no data were obtained in this study with such low detention times. The data of Morgan, points 6, 7 and 8, agree with the results obtained in this study and confirm that there is a drop in gas production with solids concentration. Sawyer and Roy (points 9, 10, 11, 12 and 13) observed an increase in gas production with detention time, although most of their values were higher than those obtained in this study. Point 13 agrees well with the data obtained here. In the summary, the results reported by other workers are not consistent. However, their research may be interpreted as confirming the conclusions reached in this study that:

1. An optimum solids concentration exists in digesting sludge at which maximum gas production might be expected.

2. As detention time is increased, or loading rate is decreased, the gas production per lb of volatile solids added increases.

B. Volatile Solids Reductions

In this study, gas production was considered to be the most important parameter in evaluating the effect of solids concentration on digestion. Digestion was considered to be in equilibrium if uniform gas productions over a period of approximately 8 to 10 days were obtained. The volatile solids reductions in the digesting sludge were expected to confirm the conclusions reached from the analysis of the gas production data. However, after completion of the experimental work when the data were being analysed, it was realized that in addition to using uniform gas production as a criterion of stable digestion the equilibrium of the volatile solids concentration in the digesting sludge also should have been considered. A much greater length of time is required to achieve equilibrium volatile solids concentrations in a digester than is required to obtain uniform gas production. For example, the time required to obtain uniform gas production after a change of detention time in a digester was approximately 8 to 10 days. The time required to obtain equilibrium volatile solids concentrations in a digester after a change of detention time is directly related to the time required to replace the digesting sludge present at the time of the change with digesting sludge at the new equilibrium volatile solids concentration. This time can be calculated (see Recommendations) for a particular detention time and a particular frequency of feeding the digester. For a digester fed once a day to prove a theoretical 20 day detention time, 59 days are required to replace 95 percent of the sludge. The approximate minimum length of time it would have taken to complete Run 3 if detention times had been held constant until equilibrium volatile solids concentrations (95 percent replacement) in the digesting sludge had been reached is 282 days. If 10 days are required at each equilibrium concentration (in Run 3, six detention times were studied) to collect data for analysis and 20 days were required to start the digesters initially, the total run time would have been 362 days. The time can be decreased if a lower degree of replacement will provide sufficient precision in the data. In making a run lasting 362 days problems of human and machine endurance must be considered. An experiment under conditions of equilibrium volatile solids concentrations in the Recommendations is proposed. Data from the proposed study should confirm the conclusions reached from the analysis of the gas production data in this study.

The inadequacy of the volatile solids reduction data to confirm completely the conclusions based on the gas production data is realized. However, the volatile solids reduction data were analyzed taking into consideration the non-equilibrium conditions under which they were collected.

The percent reduction in volatile solids was calculated for each day on which volatile solids were determined either in the raw or digested sludge. The volatile solids content of the raw sludge fed to each digester on any day was obtained from Table 16. The volatile solids content of the digested sludge on any day was interpolated from Table 20. The formula which was used to calculate volume solids reductions was developed as follows.

Let; VS_R = percent volatile solids in raw sludge

VS_D = percent volatile solids in digested sludge

VS_{Red} = percent of volatile solids destroyed during digestion

T = total weight of digested sludge solids

P = constant weight of raw sludge solids added to a completely mixed digester each day

Volatile solids in raw sludge $= P(VS_R/100)$

Fixed solids in raw sludge $= P - P(VS_R/100)$

$= P(1 - VS_R/100)$

During the course of digestion, the quantity of fixed (non-volatile) solids in the digesting sludge will not change. Hence, the weight of fixed solids withdrawn in the digested sludge each day must be equal to the weight of fixed solids added in the raw sludge each day. If the weight of sludge in the digester is held constant the weight of digesting sludge withdrawn will be less than the weight of raw sludge added because of the loss of volatile solids during digestion.

Let T be the weight of digested sludge solids withdrawn from the digester.

Then, volatile solids in digested sludge $= T \cdot VS_D/100$

and, fixed solids in digested sludge $= T - T \cdot VS_D/100$

$$= T(1 - VS_D/100).$$

However, fixed solids in raw sludge = fixed solids in digested sludge so

$$P(1 - VS_R/100) = T(1 - VS_D/100)$$

$$T = \frac{P(1 - VS_R/100)}{(1 - VS_D/100)}$$

The weight of volatile solids in the digested sludge is the total weight of digested sludge solids (T) minus the weight of fixed solids. Or

Volatile solids in digested sludge $= T - P(1 - VS_R/100)$

$$= \frac{P(1-VS_R/100)}{(1-VS_D/100)} - P(1-VS_R/100)$$

$$= P(1-VS_R/100)\left[\frac{1}{1-VS_D/100} - 1\right]$$

$$= \frac{P(1-VS_R/100)}{(1-VS_D/100)} \cdot VS_D/100$$

The percent of volatile solids reduction due to digestion is the loss in weight of volatile solids during digestion divided by the original weight of volatile solids added. Therefore,

Volatile solids reduction (percent) =

$$\left[\frac{P \cdot VS_R/100 - \dfrac{P(1 - VS_R/100)}{(1 - VS_D/100)} \cdot VS_D/100}{P \cdot VS_R/100}\right] \times 100$$

$$= \frac{VS_R(1 - VS_D/100) \quad - \quad VS_D(1 - VS_R/100)}{VS_R(1 - VS_D/100)} \quad x \quad 100$$

$$= \frac{VS_R \; - \; VS_D}{VS_R(1-VS_D/100)} \quad x \quad 100 \; .$$

It should again be emphasized that this formula is only valid when a digester is operating at equilibrium conditions with regard to volatile solids reduction. For example, suppose the volatile solids concentration in the digesting sludge in a digester fed once a day, as in this study, remains constant at 40 percent and that the volatile solids concentration in the feed sludge is 80 percent. This means that the calculated volatile solids reduction is 83.4 percent. Under these conditions, the formula is valid. Now let the loading rate to the digester be increased such that the organisms functioning in the digesting sludge are comparatively overloaded with food. If the organisms effect a volatile solids reduction of 50 percent at the new loading rate, the calculated volatile solids concentration remaining in the digested sludge will be 66.7 percent. This will mix with the previously digested sludge (40 percent volatile solids concentration) and the level of volatile solids in the digesting mixture will increase to a level greater than 40 percent. Unless digestion is completely inhibited and no volatile solids reduction takes place, the volatile solids concentration in the digested sludge will increase at a rate which is proportional to the quantity of sludge fed each day until the volatile solids concentration in the digested sludge is approximately 66.7 percent.

During the period when the volatile solids concentration in the digested sludge is changing, the formula for volatile solids reduction is not valid. For example, consider the above digester in which the volatile solids concentration in the digested sludge has increased to 45 percent but is not yet at equilibrium. According to the formula:

$$VS_{REDUCTION} = \left[\frac{80-45}{80(1-0.45)}\right] \cdot 100 = \left[\frac{35}{44}\right] \cdot 100 = 79.6 \text{ percent}$$

Thus, the calculated, volatile solids reduction exceeds the actual reduction (79.6 percent as compared to 50 percent respectively).

If a digester is started with a digested sewage sludge which has a lower volatile solids concentration than the equilibrium volatile solids concentration, the volatile solids reductions calculated from the above formula will be greater than the actual volatile solids reductions. For example, suppose a digester is started with digested sewage sludge having a volatile solids concentration of 30 percent. Assume a volatile solids concentration in the raw sludge of 80 percent and a volatile solids reduction of 83.4 percent. At this level of digestion, the volatile solids concentration in the digesting sludge will increase until it is approximately 40 percent. Some intermediate time when the volatile solids concentration in the digesting sludge is 35 percent the calculated volatile solids reduction would be:

$$VS_{reduction} = \left[\frac{80-35}{80(1-.35)}\right] 100 = \left[\frac{45}{52}\right] 100 = 86.6 \text{ percent.}$$

The volatile solids reduction calculated from the formula exceeds the actual reduction (86.6 percent as compared to 83.4 percent) demonstrating again that the formula is invalid if the volatile solids concentration of the digesting sludge is not at equilibrium.

If a digester is started at a volatile solids concentration above the equilibrium level or alternatively, if the loading rate to a digester is decreased such that the organisms in the digesting sludge are able to destroy a greater percentage of the volatile solids in the digesting sludge, then the volatile solids reductions calculated from the above formula will be less than the actual reductions. For example, suppose the initial volatile solids concentration in the digesting sludge is 50 percent, the volatile solids concentration in the feed sludge is 80 percent and the actual volatile solids reduction during digestion is 83.4 percent. Eventually, the volatile solids concentration in the digesting sludge will approach 40 percent. When the volatile solids concentration in the digesting sludge is 45 percent, the calculated volatile solids reduction is:

$$VS_{reduction} = \left[\frac{80-45}{80(1-.45)}\right] 100 = \left[\frac{35}{44}\right] 100 = 79.6 \text{ percent}$$

The calculated volatile solids reduction (79.6 percent) is lower than the actual volatile solids reduction of 83.4 percent.

In summary, if the above formula is used for calculating volatile solids reductions when unstable conditions exist in a digester with regard to volatile solids concentration in the digesting sludge, the following conclusions may be stated:

1. The calculated volatile solids reduction will be greater than the actual volatile solids reduction if the volatile solids concentration in the digesting sludge is increasing.
2. The calculated volatile solids reduction will be less than the actual volatile solids reduction if the volatile solids concentration in the digesting sludge is decreasing.

Volatile solids reductions calculated when the digesters were not in equilibrium[1] were plotted in Figure 24 against the corresponding detention time in each digester using average data from Table 5. Considering all of the plotted data, no definite trends could be observed. The plotted data showed definite inconsistencies in the region of the 16 day detention

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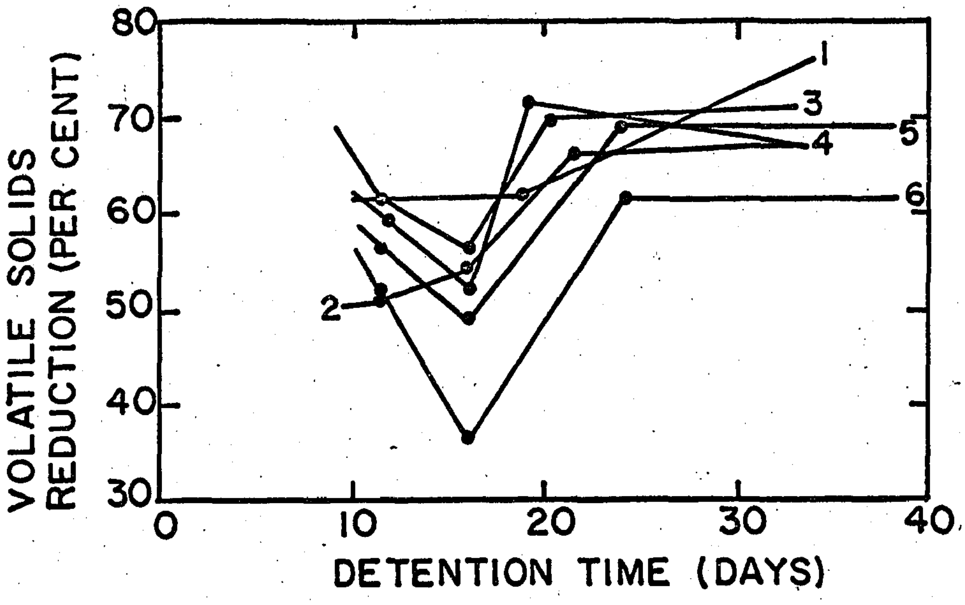
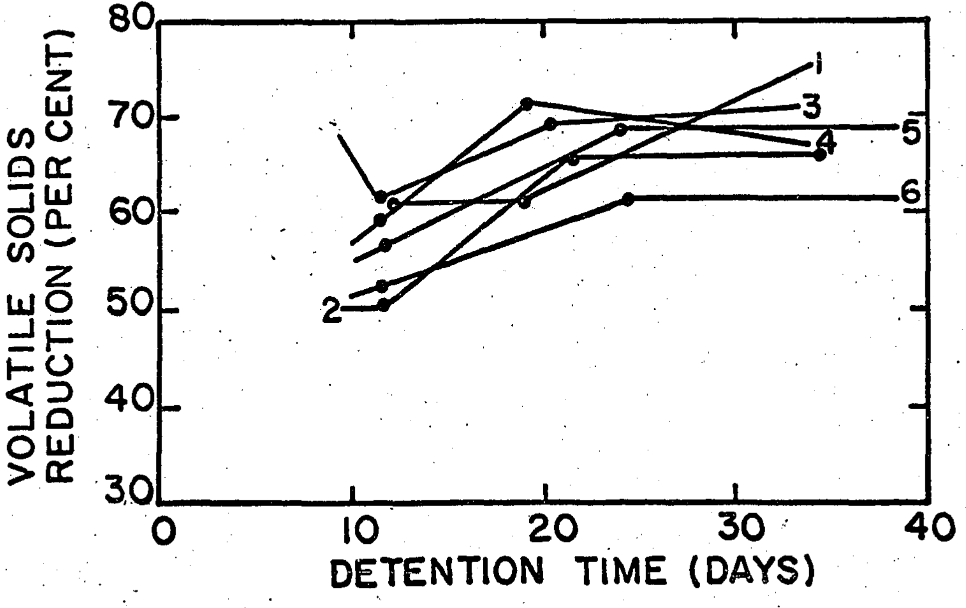
[1] The digesters were not in equilibrium with regard to maintenance of a constant volatile solids concentration in the digesting sludge. For this reason, no attempt has been made in the following discussion to present specific values of volatile solids reduction as conclusions to this portion of the study. Specific values of volatile solids reduction are quoted only to illustrate certain trends.

time for digesters 2, 3, 4, 5 and 6. Volatile solids reductions in digester 1 do not show a similar inconsistency. The data obtained during Run 3 when a 16 day detention time existed in digester 1 was not included in the compilation of Table 5 since the gas production did not appear stable. The inconsistency in the gas production and in the volatile reductions shown in Figure 24 at the 16-day detention period appear to be due to a change in the characteristics of sludge fed to the digesters over a certain time period. Referring to Tables 24 and 26 it was observed that a 16-20 day detention time occurred in all digesters around the 38th to the 43rd day of operation in Run 3 (Table 24), and during this period the volatile solids reductions in all digesters were abnormally low (Table 26). Earlier in this dissertation (see experimental procedure) a raw sludge collection problem was discussed. The sludge from the Nevada, Iowa, sewage treatment plant was found at times to be partially digested. This occurred between the 38th and 43rd days as shown by the low volatile solids contents listed for the raw sludge during this time period (Table 16). Since this sludge was partially digested before it was used as feed sludge to the digesters, low volatile solids reductions could be expected in the digesters because the more easily digestible organic materials have been digested previously. In view of this, data during this period were ignored and new trends of volatile solids reduction versus detention time were plotted in Figure 25. It was observed that in general, as the

Figure 24. Effect of detention time on volatile solids reduction for digesters 1 through 6, Run 3

Figure 25. Modified trends of the effect of detention time on volatile solids reduction for digesters 1 through 6, Run 3


VOLATILE SOLIDS REDUCTION (PER CENT)
80
70
60
50
40
30
0
10
20
30
40
DETENTION TIME (DAYS)
1
2
3
4
5
6


VOLATILE SOLIDS REDUCTION (PER CENT)
80
70
60
50
40
30
0
10
20
30
40
DETENTION TIME (DAYS)
1
2
3
4
5
6

detention time increased, the volatile solids reduction also increased.

Inspection of the data showing the volatile solids concentrations in the digesters (Table 20) indicates that the volatile solids in digesters 1 through 6 generally decreased during the first 8 days of the run. As the run continued beyond 8 days, an increasing trend of volatile solids concentration can be observed. Referring to Table 24 showing the detention times in the digesters during Run 3, the detention time in all digesters up to about 11 days is over 77 days. Detention times of this length were not considered in plotting Figures 24 and 25. Thus, the only values of volatile solids reduction (Table 5) which were used to obtain the trends in Figure 25 were calculated from those volatile solids concentrations in the digesters which were generally increasing. According to the previous discussion regarding the error in calculated volatile solids reductions, this means that the values of volatile solids reduction recorded in Table 26 all exceed actual reductions.

Consider a digester in which the volatile solids concentration of the digesting sludge is increasing due to an increase in loading rate. Suppose the equilibrium volatile solids concentration were 40 percent before a loading rate increase and 60 percent with the new loading rate; i.e., if the volatile solids concentration of the feed sludge is 80 percent and the actual volatile solids reduction in the feed sludge falls from 83.4 to 62.5 percent. The calculated volatile solids reductions obtained when various levels of volatile solids exist in the digesting sludge would be:

Volatile solids concentration in digesting sludge (percent)	Calculated volatile solids reduction (percent)
40	83.4
45	79.5
50	75.0
55	69.4
60 (Equilibrium level)	62.5 (Actual)

The above calculations show that the closer the volatile solids concentration in the digesting sludge approaches the equilibrium volatile solids concentration, the less in error is the calculated volatile solids reduction. Throughout Run 3, the detention times of the sludge in the digesters were increased at certain times (Table 22). The actual increase was sufficient to cause a change in the rate of volatile solids reduction such that there would be an increase in the equilibrium level of volatile solids in the digesting sludge. However, the digesters were not always operated long enough for the equilibrium level to be reached before the detention time was again increased. Changes in detention time or loading rate were made when gas production became relatively constant, and not when the volatile solids levels in the digesters were at equilibrium. During the early phases of the run the volatile solids concentration was at the equilibrium level at some time in all the digesters. This is indicated in Table 20. For example, the volatile solids concentration in digester 2 decreased from 53.9 percent on day 2 to 48.3 percent on day 8. As the run continued beyond the 8th day, the volatile solids content in digester 2 increased indicating that at some time between the 2nd and 9th days the volatile solids concentration was at the equilibrium level. During the later phases of Run 3 the detention time of the sludge in a digester was changed approximately every 10 days. The percent "turnover" of the digesting sludge in a digester which took place between changes in detention time can be calculated. For example, the following data for digester 3 were extracted from Table 24.

Days averaged	Number of days[a]	Average detention time (days)
12-18	7	54.0
19-28	10	33.5
29-36	8	20.3
37-49	13	15.9
50-59	10	11.4
60-66	7	8.8
67-74	8	7.3

An equation is presented in the Recommendations which states that:

$$S = 100\ (1-r^{n})$$

Where S is the percent turnover of the digester contents in the n days of operation between loading rate changes.

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[a] Number of days the digester was maintained at the specified average detention time.

D = is the theoretical detention time for the n days of operation

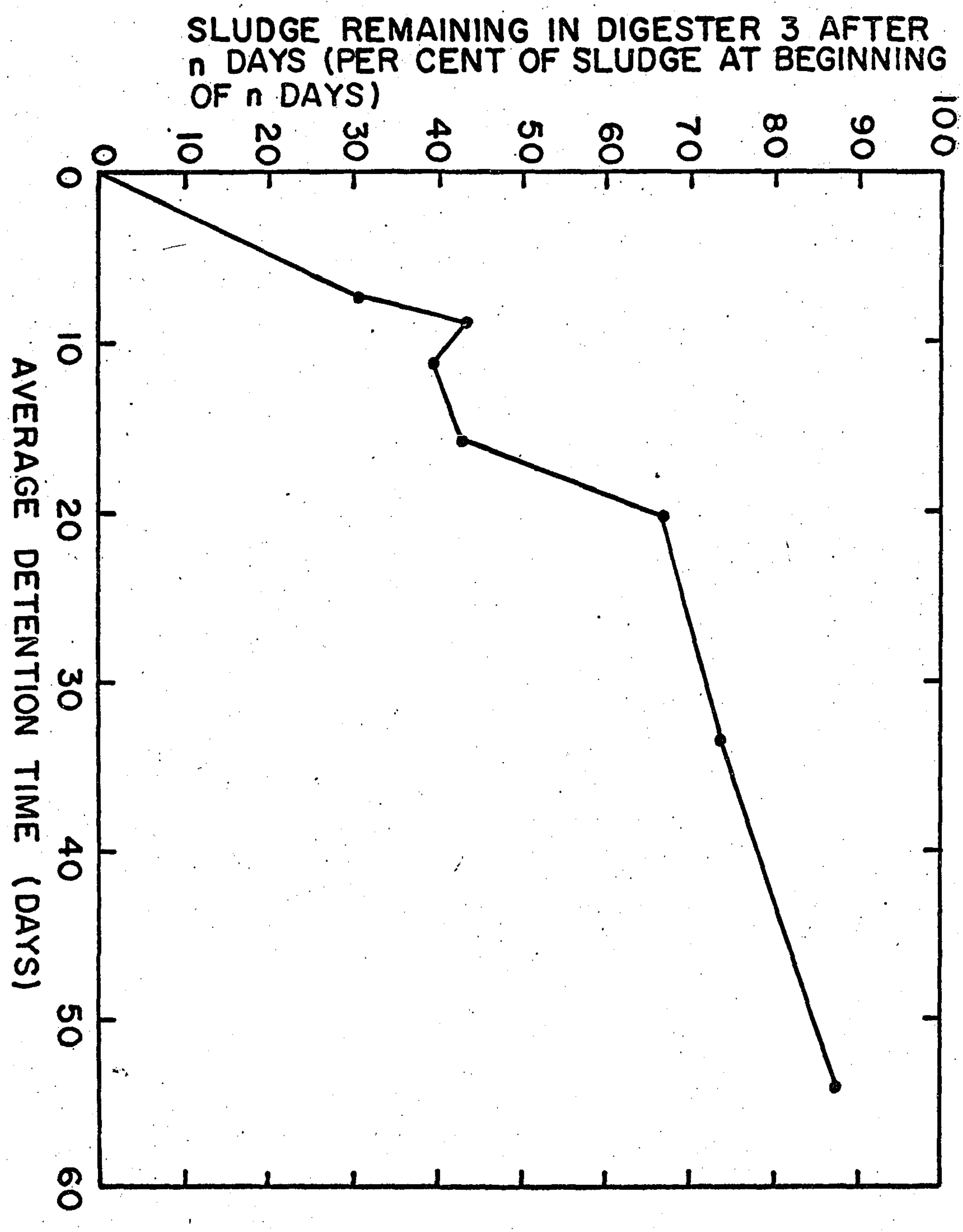
$$r = \frac{D-1}{D}$$

This equation and the above data were used to calculate S at the different detention times.

Average Detention Time (days)	r	r^n	$1-r^n$	S (percent)
54.0	0.981	0.874	0.126	12.6
33.5	0.970	0.738	0.262	26.2
20.3	0.951	0.669	0.331	33.1
15.9	0.937	0.430	0.570	57.0
11.4	0.912	0.398	0.602	60.2
8.8	0.887	0.432	0.568	56.8
7.3	0.863	0.308	0.692	69.2

The percentage of the sludge remaining in digester 3 at the end of n days which was there when the detention time was changed initially was obtained by subtracting S from 100 percent. The percentages of sludge remaining in digester 3 were plotted in Figure 26 against the associated detention times for which they were determined. Figure 26 indicates that as the average detention time decreased the percentage of the sludge which was present at the start of a particular detention time and which remained in digester 3 at the end of the period for which that detention time was operative decreased. Therefore, as the detention times in digester 3 decreased, the volatile solids content of the digesting sludge approached the equilibrium volatile solids concentration for a particular detention time. With reference to a previous discussion of the degree of

Figure 26. Sludge remaining in digester after n days as percent of sludge at beginning of n days versus average detention time

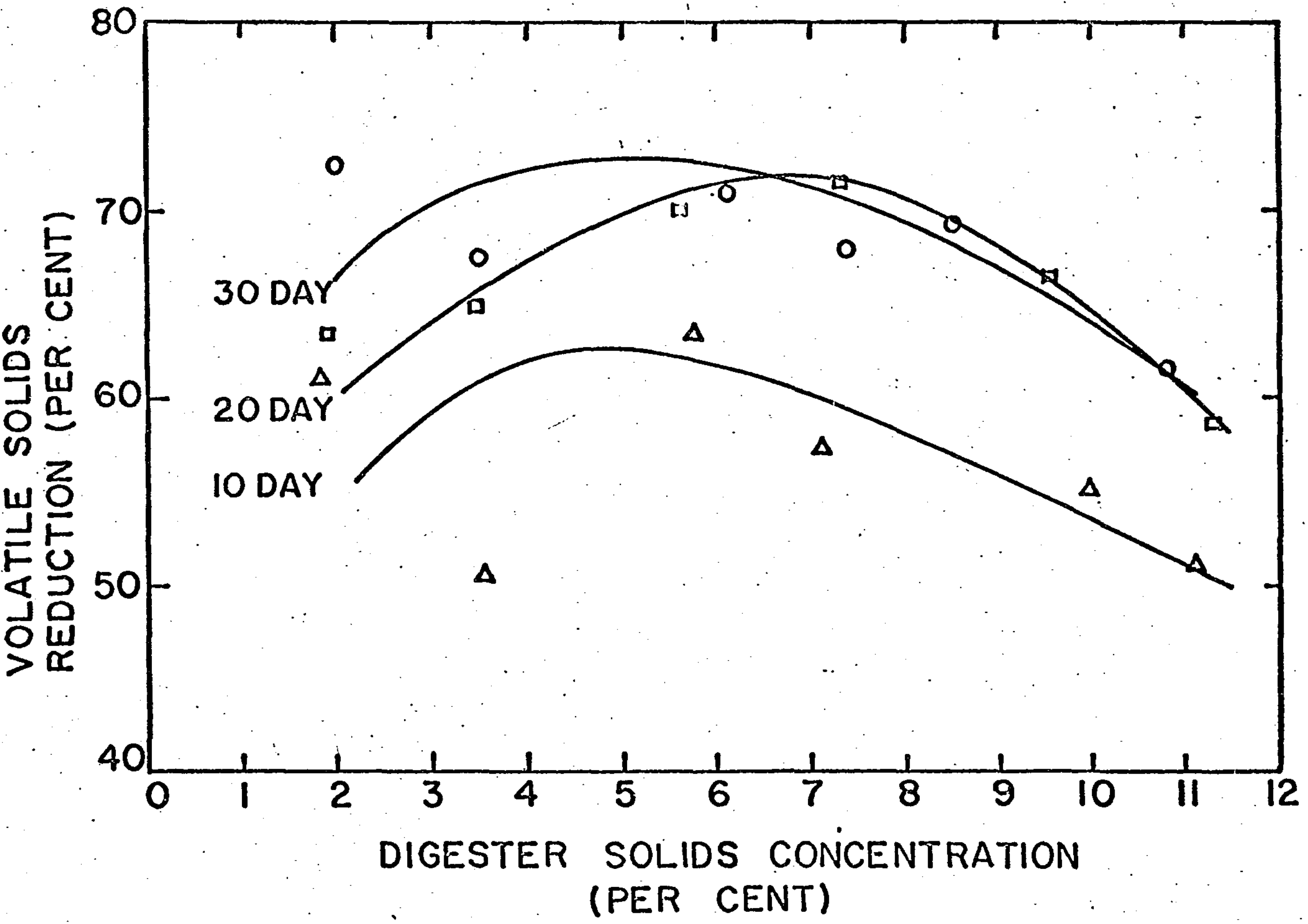

SLUDGE REMAINING IN DIGESTER 3 AFTER n DAYS (PER CENT OF SLUDGE AT BEGINNING OF n DAYS)
0
10
20
30
40
50
60
70
80
90
100
0
10
20
30
40
50
60
AVERAGE DETENTION TIME (DAYS)

error of calculated volatile solids reductions it may be concluded that the error in calculated volatile solids reductions in this study decreased as the detention time increased. Thus, the use of calculated volatile solids reductions under non-equilibrium conditions tends to nullify the trends shown in Figure 25.

Gas productions calculated using volatile solids added to the digesters will be affected by changing volatile solids concentration in the digesting sludge if the volatile solids concentration is decreasing. In this case volatile solids already in the digester are being digested and converted to gas thus increasing calculated gas productions per lb of volatile solids added. If the volatile solids concentration is increasing, as during the major portion of this study, the gas production per lb of volatile solids added is not affected. The volatile solids which are not converted to gas accumulate in the digester and result in a gradual increase in the volatile solids level in the digesting sludge.

Figure 27 was constructed using data interpolated from Figures 22 and 25 and not from average data taken directly from Table 5. The volatile solids reductions in the sludge of a particular digester at specified detention times of 10, 20, 30 and 40 days were obtained from the modified trend curves in Figure 25, and the values were plotted in Figure 27 against the solids concentration at the same detention times (Figure 22). Figure 27 indicates the qualitative effect of digester

Figure 27. Effect of digester solids concentration on volatile solids reduction at detention times of 10, 20 and 30 days, Run 3


80
70
60
50
40
VOLATILE SOLIDS
REDUCTION (PER CENT)
30 DAY
20 DAY
10 DAY
0
1
2
3
4
5
6
7
8
9
10
11
12
DIGESTER SOLIDS CONCENTRATION
(PER CENT)

solids concentration on volatile solids reduction at sludge detention times of 10, 20, 30 and 40 days. Maximum values of volatile solids reduction were expected at digester solids concentrations of 3 to 5 percent, where the gas productions were a maximum.

The trends of the curves in Figure 27 indicate that optimum digesting solids concentrations may exist for maximum volatile solids reductions. Maximum volatile solids reductions and the associated optimum solids concentrations and detention times are shown below.

Optimum digester solids concentration (percent)	Detention time (days)	Maximum volatile[1] solids reduction (percent)
4-6	10	62.6
6-7	20	72.2
4-6	30	72.9

These results also illustrate the effect of detention time on volatile solids reduction, i.e., the volatile solids reduction increases with increase in detention time. According to a previous discussion of the parameters controlling digestion it may be said that the volatile solids reduction decreases as

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[1] Although specific values of volatile solids reductions are presented here they may not be correct. The method of calculating the volatile solids reductions was invalid under the non equilibrium conditions which existed in Run 3. However, the trends shown by this table and Figure 27 may be correct (see later in this section).

the loading rate increases.

Figure 25 was affected by the changing volatile solids concentrations in the digesters as discussed previously. Thus, Figure 27 which was plotted from data obtained from Figure 25 was also affected by non-equilibrium conditions. It was shown previously in this section that the trends shown for each digester in Figure 25 are exaggerated. The effect this would have in Figure 27 would be to decrease the difference between the trends shown for each detention time. For example, the trend shown for the 10 day detention time would be at lower volatile solids reductions and the trend shown for the 30 day detention time would be at still lower volatile solids reductions. The shape of the graphs should not change. Theoretically, the maximum volatile solids reduction should be observed at the optimum solids concentration for maximum gas production. The results of this study do not agree with theory. Since the gas production data were not affected by non-equilibrium conditions of the volatile solids concentration in the digesting sludge, the optimum solids concentration observed for maximum volatile solids reduction is probably in error. The calculated volatile solids reductions were affected by changing volatile solids concentrations in the digesting sludge probably causing the optimum solids concentration for maximum volatile solids reduction to be approximately 2 percent higher than was expected.

Sawyer and Schmidt (152) reported data which agreed in general with trends shown in this study. Their data and that obtained by other experimenters are summarized below.

Solids concentrations (percent)	Volatile solids reduction (percent)	Detention time (day)	Reference
3.4[a]	54.8[b]	8	(152)
2.2[a]	56.0[b]	11	(152)
3.9[a]	58.3[b]	14	(152)
4.9	66	32	(168)
3.7	61	22	(168)
8.66	62.1	7	(116)
7.38	48.9	10	(116)

The data of Sawyer and Schmidt (152) showed trends towards optimum digester solids concentrations for maximum volatile solids reductions. The optimum solids concentrations determined in their work (2-4%) were lower than the values obtained in this study. However, the maximum solids concentration used by Sawyer and Schmidt was in the region of the optimum solids concentration for maximum gas production determined in this study. In general, the results shown by other workers for maximum volatile solids destruction are lower than the values shown here. A previous discussion explained that the values of volatile solids reduction reported in this dissertation would

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[a] Apparent optimum for maximum volatile solids reduction.

[b] Maximum volatile solids reduction.

exceed the actual volatile solids reduction. Sawyer and Schmidt's data confirm the trend of Figure 25, i.e., as the detention time increases the volatile solids reduction also increases.

Summarizing the foregoing discussion on volatile solids reduction in this dissertation, it may be concluded that:

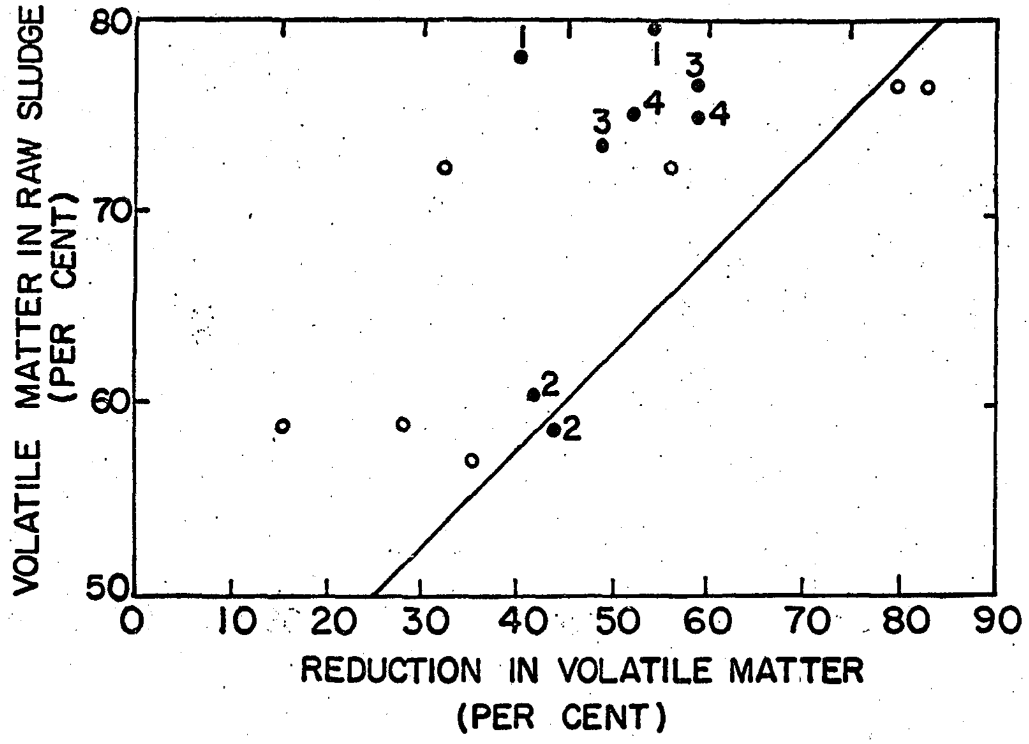
1. To obtain an accurate measure of the volatile solids reductions in a digester the volatile solids in the digester must be at an equilibrium level.
2. If the volatile solids concentration in the digesters is increasing, the calculated volatile solids reductions will exceed the actual volatile solids reductions.
3. If the volatile solids concentrations in the digesters is decreasing the calculated volatile solids reductions will be less than the actual volatile solids reductions.
4. Volatile solids reductions increase as the detention time increases from 10 to 30 days.
5. Optimum solids concentrations should exist between 3 and 5 percent for maximum volatile solids reductions at detention times of 10, 20 and 30 days. Actual optimum solids concentrations for maximum volatile solids reductions observed in this study were between 5 and 7 percent.

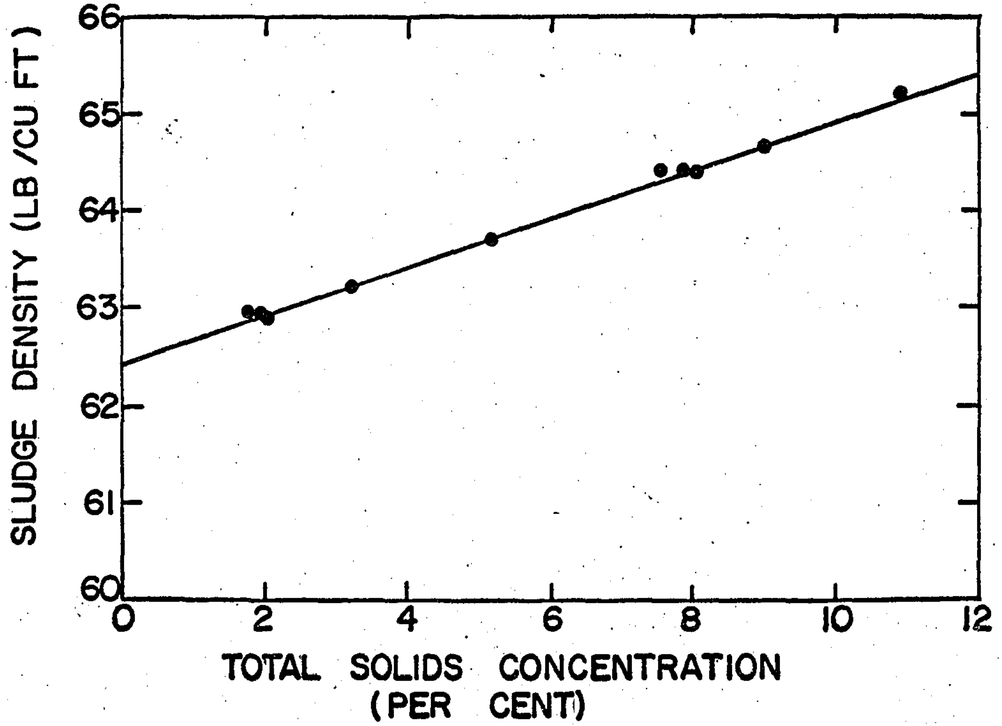
Another aspect of volatile solids reduction which must be considered in the digestion of sewage sludge is the disposal of the sludge after digestion. In general, the higher the volatile solids reduction during digestion the better are the dewatering qualities of the digested sludge. Schlenz (153b) found that the volatile solids reduction necessary during digestion to produce a digested sludge which would show satisfactory dewatering characteristics could be related to the volatile solids content of the raw sludge (Figure 27a). Extreme values of volatile solids concentrations in the raw sludge (Table 16) and the associated volatile solids reductions (Table 26) obtained in this study, are also plotted in Figure 28a. According to the line drawn to represent the data presented by Schlenz, the digested sludges obtained in this study were not considered to be digested in most cases. These results were expected considering the short detention times, the high volatile solids loading rates and the single stage system used in this study. The data which the line represents were taken from the operation data of digesters operating at a conventional rate. Much of the data came from multi-stage digestion systems. The data from other workers are also plotted in Figure 28a. Again only extreme values were plotted. In general, although most of the reported data were obtained with higher volatile solids concentrations in the raw sludge, they compare with the data obtained in this study. The effect of non-equilibrium conditions in this study was to increase the calculated volatile solids reductions. Allowing for

Figure 28a. Volatile matter in raw sludge versus reduction in volatile matter

Symbol	Reference
0	Run 3
1	Torpey (168, 169)
2	Morgan (116)
3	Sawyer and Schmidt (152)
4	Sawyer and Roy (151)

Figure 28b. Effect of solids concentration on sludge density


VOLATILE MATTER IN RAW SLUDGE (PER CENT)
80
70
60
50
1
1
3
3
4
4
2
2
0
10
20
30
40
50
60
70
80
90
REDUCTION IN VOLATILE MATTER (PER CENT)


SLUDGE DENSITY (LB /CU FT)
66
65
64
63
62
61
60
0
2
4
6
8
10
12
TOTAL SOLIDS CONCENTRATION (PER CENT)

reductions above the actual volatile solids this error will bring the data obtained in this study, at the higher raw sludge volatile solids concentrations, closer to the reported data. The data obtained in this study at lower raw sludge volatile solids concentrations indicates lower volatile solids reductions than the data plotted for Morgan (116). Morgan (points 2) observed acceptable dewatering characteristics of the digested sludges obtained from his experiments.

C. Gas Quality

The quality of the gas produced during digestion in Run 3 is summarized in Table 30. The average composition of the gas produced in each digester throughout the run was obtained by taking the average of the data listed in Table 30 for each digester. The average gas compositions in digesters 1 through 6 are reported in Table 7. Average solids concentrations were determined from the data in Table 18 in a similar manner and they are also reported in Table 7. Table 7 indicates that the highest quality gas, 67.9 percent methane, was obtained from digesting sludge having an average solids concentration of 5.8 percent. The gas quality appeared to be highest in the range of solids concentrations of 5.8 to 7.4 percent at which concentrations the methane content was 67.9 to 67.7 percent respectively. Outside of this solids concentration range the gas quality decreased. Included in Table 7 is the average composition of the gas produced in the digestion studies conducted by

Torpey (168). The quality of the gases in this study and in Torpey's study compare favorably. The methane contents are slightly higher in this study but these may be due to different methods of analysis or the assumption made in this study that only carbon dioxide and methane were present.

Table 7. Average composition of gas produced, Run 3

Digester or Reference	Average solids[a] concentration (percent)	Gas composition[b]	
		Percent CH_4	Percent CO_2
1	1.9	65.4	34.6
2	3.5	64.8	35.2
3	5.8	67.9	32.1
4	7.4	67.7	32.3
5	9.0	65.3	34.7
6	10.6	63.3	36.7
Torpey (168)		62-64	38-36

[a] From Table 18.

[b] Assuming the only gases present in the digesters are methane and carbon dioxide. There will be other gases present but the total quantity will be small, i.e., less than 5 percent.

Data from Tables 22, 24 and 30 were analyzed to determine the effect of detention time and volatile solids loading rate on gas quality. For example, the values shown below are the average values for the days indicated. No definite trends could be observed with regard to the effect of detention time on gas quality when the loading rate was approximately 10 x 10^{-2} lb volatile solids added per cu ft per day. An increase in detention time from 33.7 days to 57.1 days in digesters 3 through 6 caused a decrease in gas quality from 70.2 percent methane to 60.7 percent methane. Similar results were obtained at other loading rates.

Digester	Days Averaged	Loading rate[1]	Detention time (days)	Gas quality (percent CH_4)
1	61,63	9.3	8.8	67.4
2	30,32,35	11.2	20.7	66.6
3	19,24,27	10.5	33.7	70.2
4	19,24,27	12.2	33.9	68.7
5	13,14,15 16,19,24	9.2	53.9	65.1
6	13,14,15 16,19,24	11.3	57.1	60.7

The effect of the volatile solids loading rate was determined with data of which the following are typical. The data were again extracted from Tables 22, 24 and 30 and averaged for the days mentioned. These particular data refer to digester 5. No definite trend could be observed to depict the effect of loading rate on gas quality. An optimum gas quality may exist at about a detention time of 40 days and a loading rate of 13.2×10^{-2} lb volatile solids added per cu ft per day but the data are not conclusive. Similar results

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[1] Lb volatile solids added per cu ft per day x 10^{-2}.

were obtained with digesters 1, 2, 3, 4 and 6.

Days Averaged	Detention time (days)	Loading rate[1]	Gas quality percent CH_4
13,14,15 16,19,24	53.9	9.2	65.1
27	41.5	13.2	71.8
30,32,35	26.4	20.8	64.6
49	16.0	30.4	66.1
56,61,63	11.3	47.8	62.9
66,68	13.3	45.3	63.1

The conclusions regarding gas quality reached in this study are:

1. A maximum average gas quality of 67.9 percent methane was obtained at an average solids concentration of 5.8 percent.
2. Loading rate and detention time had no significant effect on gas quality.

Conclusion 2 was not an expected result of this study and it contradicts the effects of loading rate and detention time on gas quality reported in the literature. The literature indicates that gas quality decreases as the detention time decreases and the loading rate increases. The reason for the decrease in gas quality is indicated by the appearance of

---

[1] Lb volatile solids added per cu ft per day x 10^{-2}.

propionic acid, and possibly longer carbon chain volatile acids, in digesting sludge when digestion approaches failure due to high loading rates or low detention times. The composition of the gas produced from the metabolism of propionic acid has been reported (162) as containing seven moles of methane to one mole of carbon dioxide. Longer chain volatile acids also produce a gas richer in methane than carbon dioxide when they are metabolised. Acetic acid which is the most common volatile acid intermediate in digestion is biologically decomposed to a gas containing 50 percent methane and 50 percent carbon dioxide. Acetic acid can normally be identified in digesting sludge at any stage of digestion. It appears that the microorganisms which metabolize propionic acid are affected by adverse environmental conditions to a greater extent than the organisms which metabolize acetic acid. If this is the case, as digestion approaches failure there will be a greater reduction in the rate of decomposition of propionic acid compared to acetic acid and the methane content of the digester gas will decrease. Another possible reason for the increase in the carbon dioxide content and the decrease in the methane content of the digester gas is the inhibition of the gasification stage of digestion but not the liquefaction stage. In the liquefaction stage some carbon dioxide is produced in decarboxylation reactions. Methane is only formed in the gasification stage. Thus, inhibition of the gasification stage would tend to increase the percentage of carbon dioxide in the digester gas.

The explanation of conclusion 2 may depend upon the solubility of carbon dioxide in the digesting sludge. As the detention time decreased and the loading rate increased higher alkalinities were observed in the digesting sludge. Thus more carbon dioxide would be combined in chemical compounds such as carbonates and bicarbonates in the digesting sludge at high alkalinities. This may have prevented the carbon dioxide content of the digester gas increasing significantly.

D. Volatile Acids

The level of the volatile acids in the digesting sludges during Run 3 are shown in Table 32. A typical plot of this data is shown in Figure 18 for digester 2. At first, a high level of volatile acids, approximately 800 mg/l, was observed in the digesting sludge, probably due to the unstable conditions experienced when starting up the digesters. The level decreased as the run progressed until about 20 days after filling the digesters a mean concentration of about 200-250 mg/l of volatile acids appeared to be attained. The volatile acids concentration varied widely after the 20th day, but the variations about the mean concentration appeared to be equal. Towards the end of the run the volatile acids concentration increased to 650 mg/l in a manner which suggested that digestion was failing.

A similar pattern of variation of volatile acids concentration was noticed in digesters 1, 3, 4 and 5. Digester 6 differed from this pattern. The initial high level of volatile acids did not decrease appreciably as the run progressed. The volatile acids concentration at the beginning of Run 3, during the period when it appeard that equilibrium values were reached, and at the end of Run 3 are shown in Table 8. Table 8 shows that as the solids concentration increased in the digesters the equilibrium level of volatile acids also increased. Except for digester 4, the volatile acids concentration during periods when the digestion approached failure at the end of the run also increased with solids concentration. An interesting feature regarding the production of volatile acids was noticed in the results from digester 6. Volatile acids concentrations as high as 5,000 mg/l did not cause the complete inhibition of the acid forming organisms. Schulze (157) obtained volatile acid concentrations as high as 55,000 mg/l when digesting sludge with a 50 percent solids concentration. The ability of the acid forming organisms to function at such high acid concentrations is discussed under the section on recommendations.

Table 8. Volatile acids concentration in digesting sludge,[a] Run 3

	Digester					
Day	1	2	3	4	5	6
Average solids concentration (percent)	1.9	3.5	5.8	7.4	9.0	10.6
Day 6	590	830	2020	3450	2800	2690
Equilibrium value	120	250	420	450	500	2500
Day 66	280	630	1080	450	4870	5680

[a] mg/l as acetic acid.

The volatile acids concentration in the digesting sludge in digester 4 did not show that failure of digestion was about to occur. Although the gas production data did indicate imminent failure of digestion it might have been possible to increase the loading rate to a higher level without failure.

From these results it can be concluded that as the solids concentration increases the equilibrium volatile acids level also increases.

E. Individual Volatile Acids

The individual volatile acids observed in the digesters at various times throughout the study are listed in Table 9. Shortly after starting Run 3, all of the digesters except

Table 9. Volatile acids in digesting sludge, Run 2

Day	Digester					
	1	2	3	4	5	6
5	Acetic Propionic	Acetic Propionic Butyric Valeric	Acetic Propionic Butyric Valeric	Formic Propionic Butyric Valeric	Acetic Propionic Butyric Valeric	Acetic Propionic Butyric Valeric
6	Acetic Propionic or Butyric	Acetic Propionic or Butyric	Acetic Propionic or Butyric	Acetic Propionic or Butyric	Propionic Butyric	Acetic or Propionic Butyric or Valeric
19	- -[a]	- -	Acetic Butyric	2 unidentified Valeric Butyric Acetic	1 unidentified Valeric Butyric Acetic	Valeric Butyric Acetic
29	- -	- -	- -	- -	- -	Acetic Propionic Butyric Valeric
60	- -	- -	- -	Acetic Propionic	Acetic Propionic	Valeric Butyric Propionic Acetic
66	Acetic Propionic	Acetic Propionic	Acetic Propionic	Acetic Propionic	Valeric Butyric Propionic Acetic	Valeric Butyric Propionic Acetic

[a] No acids identified.

Acid	Rf.
Formic	0.13-0.16
Acetic	0.18-0.25
Propionic	0.27
Butyric	0.41-0.42
Iso-butyric	0.37
Valeric	0.51-0.54

digester 1 contained acetic, propionic, butyric and valeric acids in measurable quantities. Digester 1 contained only acetic acid and propionic acid. According to Buswell et al. (19), the ability to identify a volatile acid depends on the concentration of the acid present. A low solids content was maintained in digester 1 and the resulting dilution of volatile acids produced during digestion may explain the absence of butyric and valeric acids. The results agree with similar data reported in the literature which indicates that during unstable digestion, i.e., when starting the digestion process, the acids mentioned above are present. The results of other studies also indicate that as alkaline digestion becomes established, propionic acid tends to disappear. This occurred with all digesters by the 19th day. As the loading rates were increased, propionic acid began to reappear in the digesters until on the 66th day propionic acid was identified in all the digesters. Acetic acid was identified in the digesters whenever volatile acids could be identified, thus confirming the general belief that acid is the most important volatile acid intermediate in sludge digestion. Although digestion in digesters 4, 5 and 6 appeared to be operating in a stable condition with regard to gas production, butyric and valeric acids were frequently identified in the sludge from these digesters throughout the run. The data of Liubimov and Kagan (98) indicate that butyric and valeric acids are not present in digesting sludge in a stable digestion process. The presence of butyric and valeric acids in the digesting sludge will be governed by the efficiency of the gas producing organisms. However, normal uniform gas productions were obtained from digesting sludge when these acids were present indicating that the gas producing organisms were not inhibited in their presence. The results obtained here disagree with the results of Liubimov and Kagan. A definite statement cannot be made with regard to which results are correct because the apparatus used in this study to identify volatile acids was not very sensitive.

F. Loading Rate

All digesters were loaded until a breakdown in the anaerobic digestion process was imminent. The point of breakdown was not rigidly defined. It was taken as the loading rate at which the gas production decreased and continued to decrease even though the loading rate was held constant. Other indicators were used to indicate that a breakdown had occurred. With the loading rate held constant, a continuous increase in volatile acids, a large increase in the specific resistance of the digesting sludge, a decrease in the volatile solids reduction and a decrease in gas quality were among the indicators used. Every indicator did not describe the onset of digester failure in each digester.

An equation can be derived relating the rate of loading a digester with the detention time in the digester and the solids concentration in the feed sludge to the digester.

Suppose: $L =$ loading rate (lb volatile solids added per cu ft per day)

$D =$ detention time (days)

$T =$ total solids concentration in the feed sludge (percent)

$V =$ volatile solids concentration in the feed sludge (percent)

$A =$ volume of digester (cu ft)

Then, the weight of total solids added per day to a digester is given by:

$$100 \frac{(L)(A)}{(V)} \text{ lb.}$$

Hence, the weight of sludge added is:

$$\frac{(L)(A)}{(V)(T)} \times 10^4 \text{ lb.}$$

Assuming a sludge density equal or close to that of water[1], the volume of sludge added is:

$$\frac{(L)(A)\ 10^4}{(V)(T)(62.4)} \text{ cu ft}$$

---

[1] The variation in sludge density with solids concentration is small. Figure 28b is a plot of sludge density versus solids concentration for the sludges used in Run 2. It can be seen that as the solids concentration increases from 0 to 12 percent the sludge density increased from 62.4 lb per cu ft to 65.5 lb per cu ft. The change in density will vary with the characteristics of the solids studied but for domestic sewage sludge the author believes that the variation will be small.

The detention time of the solids in the digester, assuming complete mixing, is given by:

$$D = \frac{(A)}{10^4 (L)(A) \Big/ (V)(T)(62.4)} = \frac{(62.4)(V)(T)}{10^4 (L)} \text{ days} \qquad (1)$$

If three of the four values involved are substituted in the foregoing equation and the fourth value calculated, a straight line plot of the results will be obtained on log-log graph paper. Probably the simplest way to use the above equation would be to consider the volatile solids content as a constant.

$$\log D = \log 62.4 + \log V - 4.0 + \log T - \log L \qquad (2)$$

$$\text{or } \log D = K + \log T - \log L \qquad (3)$$

where $K = \log 62.4 + \log V - 4.0$.

Equation 3 is valid only when using the concentration of total and volatile solids in the feed sludge. A more informative form of Equation 3 could be to have the solids expressed in terms of the solids concentration in the digesting sludge. This would more nearly represent the actual environment inside the digester. Accordingly, a graph of total solids concentration times volatile solids concentration in the feed sludge was plotted versus the total solids concentration times volatile solids concentration in the digesting sludge (Figure 20). The data was obtained from Tables 14, 16, 18 and 20.

A straight line relationship between the two was obtained. If the total and volatile solids concentration of the digesting sludge can be represented by T_1 and V_1 respectively, Figure 20 shows that:

$$(T)(V) = (T_1)(V_1)(1.775).$$

Substituting this relationship in Equation 2

$$\log D = \log 62.4 + \log 1.775 + \log V_1 + 4.0$$
$$+ \log T_1 - \log L$$

$$\text{or} \quad \log D = K_1 + \log T_1 - \log L \qquad (4)$$

$$\text{where} \quad K_1 = \log 62.4 + \log 1.775 + \log V_1 - 4.0.$$

The values for detention time, total solids concentration in the digester and loading rates used in Run 3 were plotted in Figure 29. The conditions prevailing when the digesters became sour are indicated. It will be noticed in Figure 29 that the minimum detention time achieved with good digestion in any digester was approximately 8.8 days.

The value obtained from Figure 19 relating the total solids in the feed sludge to the total solids in the digesting sludge was used in conjunction with the similar value obtained from Figure 20 to determine the relationship between the volatile solids in the feed sludge to the volatile solids in the digesting sludge, i.e.

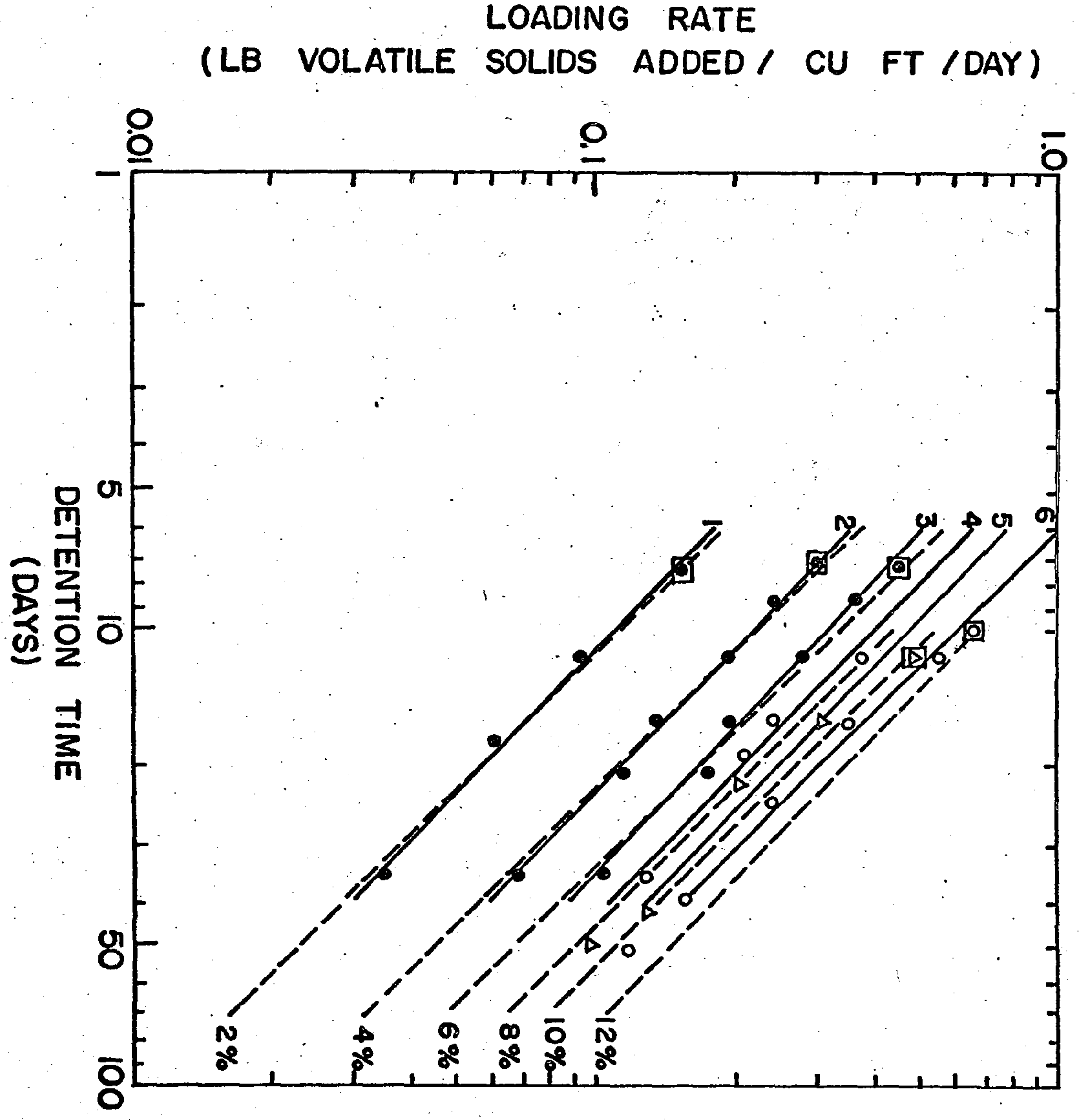
$$(T)(V) = (T_1)(V_1)(1.775)$$

$$\text{and} \quad T = T_1 (1.23).$$

$$\text{Hence,} \quad V = V_1 (1.44). \qquad (5)$$

Figure 29. Loading rate versus detention time for digesters 1 through 6, Run 3

Symbol	
-----	Theoretical lines
⊡	Sour digester

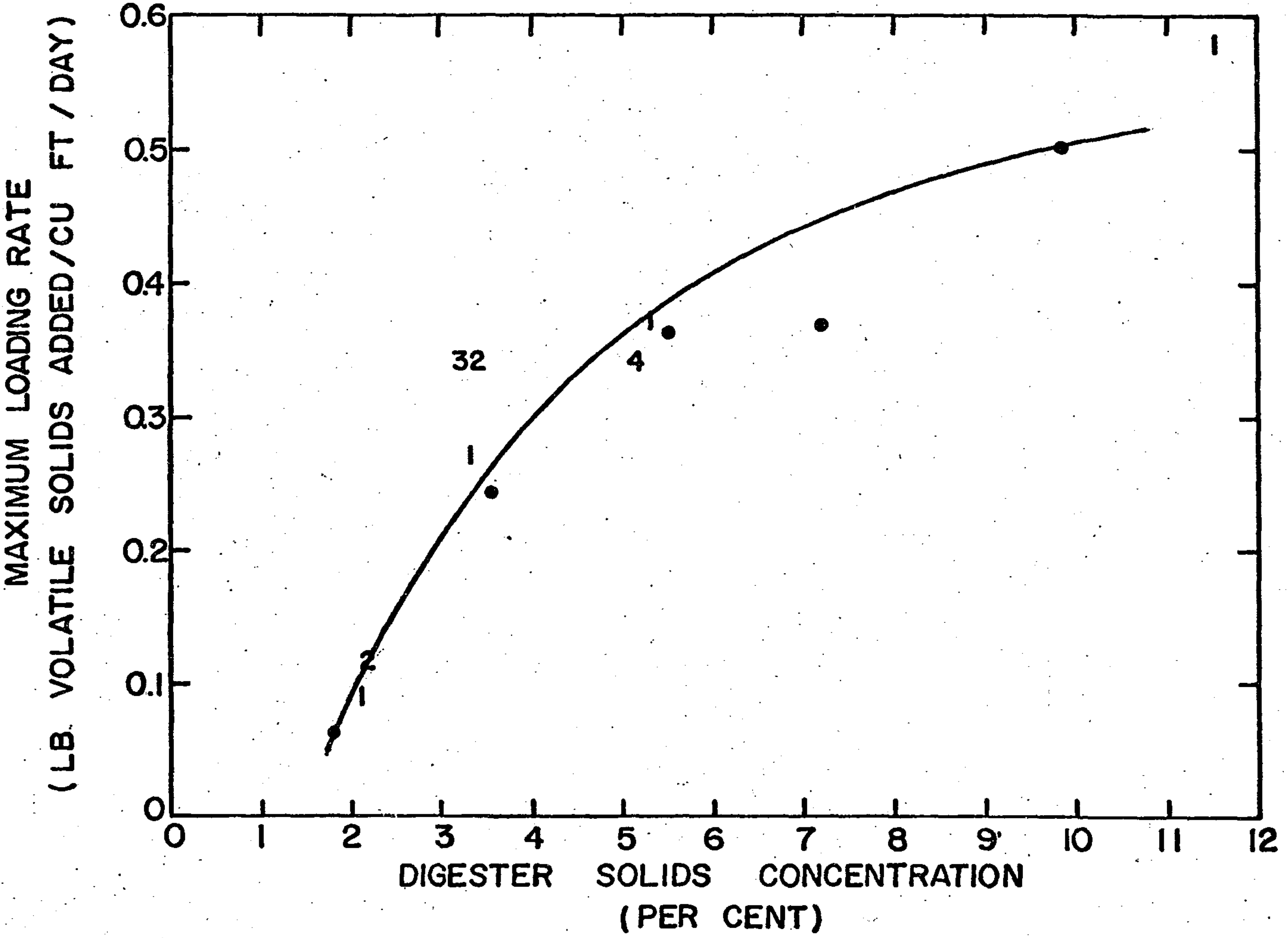

LOADING RATE
(LB VOLATILE SOLIDS ADDED / CU FT / DAY)
0.01
0.1
1.0
DETENTION TIME
(DAYS)
5
10
50
100
1
2
3
4
5
6
2%
4%
6%
8%
10%
12%

Assuming a volatile solids concentration of 72.0 percent in the raw sludge, the volatile solids concentration in the digesting sludge as calculated from Equation 5 would be 50.0 percent. This represents approximately a 61.1 percent reduction in volatile solids. Using a value of 50.0 percent volatile solids in the digesting sludge, values for detention time and loading rate were substituted in Equation 4 and lines were drawn for various solids concentrations in Figure 29. The calculated points closely approximated the points obtained in the experiments thus tending to prove the validity of the derived equations. The slopes of the calculated and experimentally determined lines differ only slightly. The error was probably due to the volatile solids content of the digesting sludge not being 50.0 percent as assumed.

The maximum loading rate achieved in a digester without failure was plotted against the solids concentration maintained in the digester (Figure 30). The curve obtained rose steeply at the lower solids concentrations. As the solids concentration increased, the slope of the curve decreased. The maximum loading rates achieved in Run 2 were also plotted in Figure 30. These values agreed with the values obtained in Run 3. The maximum loading rates achieved by other experimenters compared favorably with results obtained in this study. Figure 30 indicates that loading rates of a digester cannot be increased indefinitely by increasing the

Figure 30. Maximum loading rates

Sumbol	Reference
	Run 3
1	Run 2
2	Sawyer and Schmidt (152)
3	Sawyer and Roy (151)
4	Morgan (116)


MAXIMUM LOADING RATE
(LB. VOLATILE SOLIDS ADDED/CU FT / DAY)
0
0.1
0.2
0.3
0.4
0.5
0.6
0
1
2
3
4
5
6
7
8
9
10
11
12
DIGESTER SOLIDS CONCENTRATION
(PER CENT)
32
1
4
1
2
1
1

solids concentration. As the solids concentration is increased, the corresponding ability to increase loading is decreased. There are several reasons for supposing a maximum loading rate will be achieved. Some of them are: inadequate mixing will result as the solids content is increased due to the increase in viscosity of the sludge; ammonia-nitrogen toxicity in thickened sludges as discussed by Albertson (2); inability of the organisms to circulate freely in the sludge which would prevent the organisms coming into contact with new food and also keep them in the localized concentration of their own end products; physical problems in maintaining and feeding plant scale digesters with very high solids concentrations.

In a situation where the solids concentration is not the limiting factor which determines the extent to which a digester can be loaded, the detention time may become the limiting factor. The minimum detention time at which a completely mixed anaerobic digester can operate is directly related to the average generation time of the gas forming bacteria in the digesting sludge. If the detention time of the digesting sludge in a digester is low, a large proportion of the digesting sludge is replaced at each feeding. Thus, a large proportion of the gas forming bacteria in the digesting sludge are also removed from the digester at each feeding. The minimum detention time at which a digester can operate is reached when the rate of removal of the gas producing bacteria is equal to their rate of reproduction. For example, suppose a minimum detention time D, of the digesting sludge in a digester is achieved without causing digestion failure. Under this condition, the gas forming bacteria must be reproducing at the same rate at which they are being removed in the digesting sludge. Consider a digester of unit volume which is fed once a day and from which sludge is withdrawn immediately before feeding. If the quantity of sludge in the digester is to remain constant from day to day, the volume of raw sludge fed to the digester each day must be equal to the volume of sludge withdrawn from the digester each day, i.e., ignoring the amount of volatile solids which is converted to gas. This amount, relative to the quantity of sludge withdrawn at low detention times, is negligible. If the total number of gas forming bacteria uniformly distributed in the digesting sludge is equal to N then the number of bacteria withdrawn in the digesting sludge at each feeding is $N_B(1/D)$. The number of bacteria remaining in the digester is N_B - N_B (1/D). For digestion to continue without failure, the bacteria must replace those lost in the withdrawn sludge before the digester is fed again. Thus, the number of bacteria which have to be replaced in one day is equal to $N_B(1/D)$.

The number of bacteria, which reproduce by binary fission, resulting from reproduction may be formulated as:

$$b = c \cdot 2^n$$

where b is the number of bacteria resulting from an inoculum c after n generations. In the foregoing example,

$$b = N_B$$

$$c = N_B - N\ (1/D)$$

and n = the number of generations in one day which are necessary for the bacteria to replace the bacteria lost in the withdrawn sludge.

Hence,

$$N_B = \left[N_B - N_B(1/D)\right] \quad 2^n$$

or $\frac{D}{D-1} = 2^n$.

In this study, a minimum detention time of 8.8 days was achieved without digestion failure.

Thus,

$$\frac{8.8}{7.8} = 2^n$$

and

$$n = 0.174.$$

If in one day there were 0.174 generations the generation time of the organisms must be 5.75 days. This means that the average generation time of the gas producing bacteria in the above case must be less than 5.75 days or the digestion would fail simply by removal of the gas producing bacteria. If the average generation time was longer the minimum detention time of 8.8 days could not have been achieved. In this study, digestion failed when the detention time was 7.3 days. In this case,

$$\frac{7.3}{6.3} = 2^n$$

$$n = 0.213$$

and the generation time would be 4.69 days. Since digestion failed, it appears that the average generation time was not sufficient to keep up the level of gas producing organisms and consequently was greater than 4.69 days. These results may indicate an actual generation time greater than 4.69 days but less than 5.75 days.

Generation time depends on many factors such as temperature and the characteristics of the sludge. Thus, the average generation time of the gas forming bacteria applies only to this study and the particular conditions under which it was conducted. However, many reports in the literature indicate that the minimum detention time achieved with good digestion is approximately 8 days. It, therefore, appears likely that the average generation time of the gas forming organisms in high rate sludge digestion is in the region of 5 days.

In conclusion the following results were observed:

1. A minimum detention time of 8.8 days was obtained in this study without digestion failure. The calculated average generation time of the gas forming organisms at this detention time was in the region of 5 days.

2. It appears that as the digester solids concentration increases the allowable loading rate to the digester also increases without digestion failure. The results showed that a maximum loading rate is likely to be obtained because of limiting factors such as detention time and efficiency of mixing.
3. The maximum loading rate achieved in this study, without digestion failure, was approximately 0.56 lb volatile solids added per day per cu ft of digestion capacity.

G. Alkalinity and pH

Data showing the pH and alkalinity levels in the digesting sludges are presented in Tables 33 and 34. The data in Tables 22, 24, and 33 indicate that there was a general decrease in the pH of the digesting sludges as the loading rates to the digesters were increased and the detention times were decreased. In Table 22, the loading rate to digester 2 is shown to increase from 2.5×10^{-2} to 29.5×10^{-2} lb volatile solids added per cu ft per day from day 11 to day 70. Over the same period of time the pH of the digesting sludge in digester 2 (Table 33) decreases from 7.80 to 6.65. Table 24 indicates that the detention time decreased from 81 days on the 11th day to 7.2 days on the 70th day. The pH of the sludges in digesters 5 and 6 (Table 33) appeared to be less in most cases than the pH of the sludges in the other digesters on the same day. On the 70th day, the pH of the sludges indicated the onset of sour conditions in all of the digesters except digester 4. The results obtained by Sawyer and Schmidt (152) which showed that pH increased with solids concentration did not agree with the findings in this study. However, the maximum solids concentration (5.2 percent) they used in their study was not as high as the solids concentrations used in this study. Sawyer and Schmidt noticed a decrease in pH as the detention time decreased.

The alkalinity of the digesting sludge increased as the solids concentration increased and as the detention time decreased. Table 10 which was obtained by selecting the values of solids concentration and alkalinity for a typical day from Tables 18 and 35 shows the increase in alkalinity with solids concentrations. Table 11 was obtained by extracting values of detention time and alkalinity for a typical digester from Tables 24 and 35. This table shows the increase of alkalinity with decrease in detention time. Sawyer and Schmidt (152) observed an increase in alkalinity with solids concentration but when the detention time was decreased they observed a decrease in alkalinity. Albertson (2) reported that digesters could be operated at higher volatile acids levels when high alkalinities were maintained in the sludge. This ability was also observed in this study with digester 6.

Table 10. Alkalinity and solids concentration on day 64, Run 3

Digester	Solids concentration[a]	Alkalinity[b]
1	1.83	1500
2	3.56	2900
3	5.50	3800
4	7.19	5100
5	9.78	6900
6	11.16	8000

[a] Percent.

[b] Mg/l as $CaCO_3$.

Table 11. Alkalinity and detention time of digester 3, Run 3

Day	Detention time[a]	Alkalinity[b]
19	32.4	2930
26	34.8	3300
53	11.4	3400
64	8.9	3800

[a] Days.

[b] Mg/l as $CaCO_3$.

H. Sludge Dewatering

Three determinations of specific resistance were made of the sludge from each digester during Run 3. The results of the determinations are shown in Table 12. Also included in the table are specific resistances observed for raw sludge from the Ames and Nevada sewage treatment plants, and Ames digested sludge.

Table 12. Specific resistance[a] of digesting sludge and raw sludge, Run 3

	Digester					
Day	1	2	3	4	5	6
26	2.7	15.2	13.8	12.4	12.9	15.8
57	15.4	14.7	14.3	14.9	31.6	57.1
74	26.9	19.6	15.1	20.6	19.9	29.4
	Ames raw sludge		10.2			
	Nevada raw sludge		6.9			
	Ames digested sludge		6.2			

[a] Sec^2 per lb mass x 10^{12}.

Practical experience has shown that digested sludge is easier to dewater than raw sludge. Thus, the specific resistance of sludge should decrease as digestion continues. The results of specific resistance determinations of Ames and Nevada raw sludges and Ames digested sludge agree with this supposition (Table 12). A low specific resistance for the

Nevada raw sludge was obtained but this can be explained if the operation of the Nevada sewage treatment plant is considered. The removal of sludge from the primary tank to the digester at the Nevada plant is accomplished for one minute every thirty minutes by the operation of a positive displacement pump, activated by a time clock. All of the sludge is not removed from the tank and it is possible for sludge to remain in the tank for several days. In warm weather, partial digestion of the sludge will occur and the author believes that the sample collected for the dewatering test was collected under these conditions.

Specific resistances of the digesting sludges shown in Table 12 were plotted in Figure 31 against corresponding loading rates (Table 22). The trend of the data plotted in Figure 31 indicated that the specific resistance of digesting sludge increased as the loading rate increased. For example, as the loading rate increased from 0.1 to 0.5 lb volatile solids added per cu ft per day the specific resistance of the digesting sludge increased from 15 to 19 sec^2 per lb mass x 10^{12}. The increase was small indicating that in the range mentioned a change in loading rate has little effect on specific resistance. There was some scatter to the data which will be discussed later in this section.

The effect of detention time on the specific resistance of digesting sludge was determined from Figure 32. Figure 32 was plotted using specific resistance data from Table 12

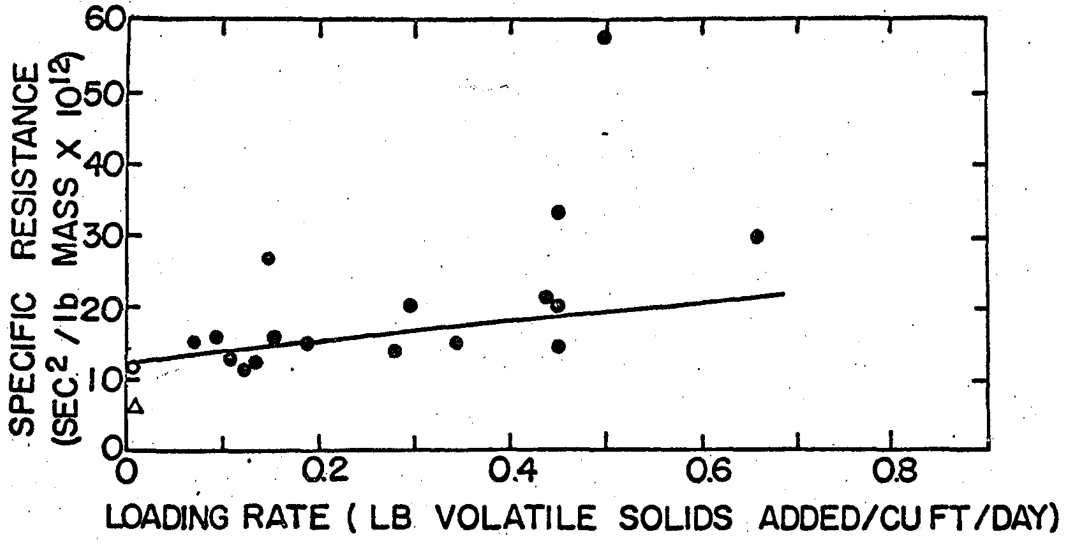
Figure 31. Effect of loading rate on specific resistance, Run 3

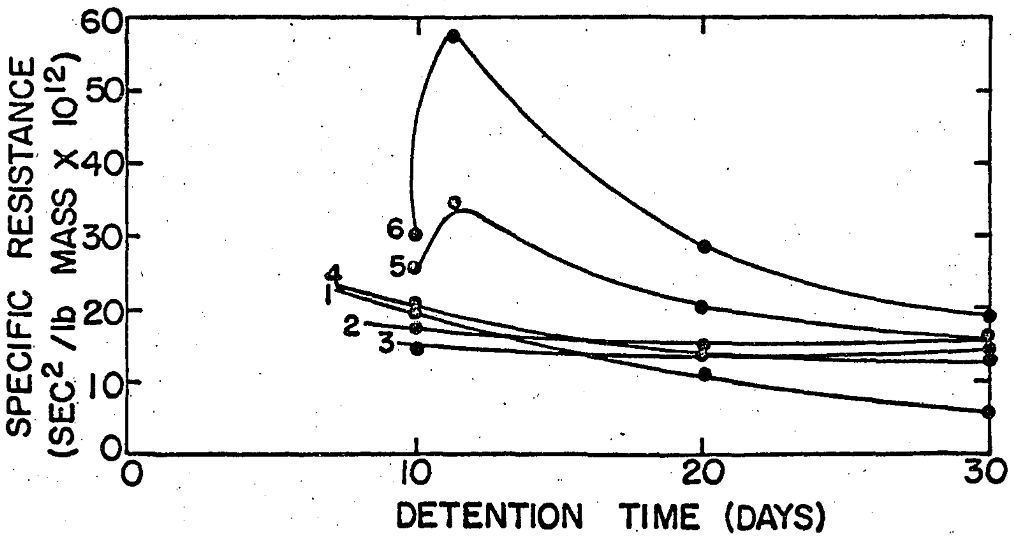
Symbol	
○	Ames raw sludge
△	Nevada raw sludge
●	Run 3

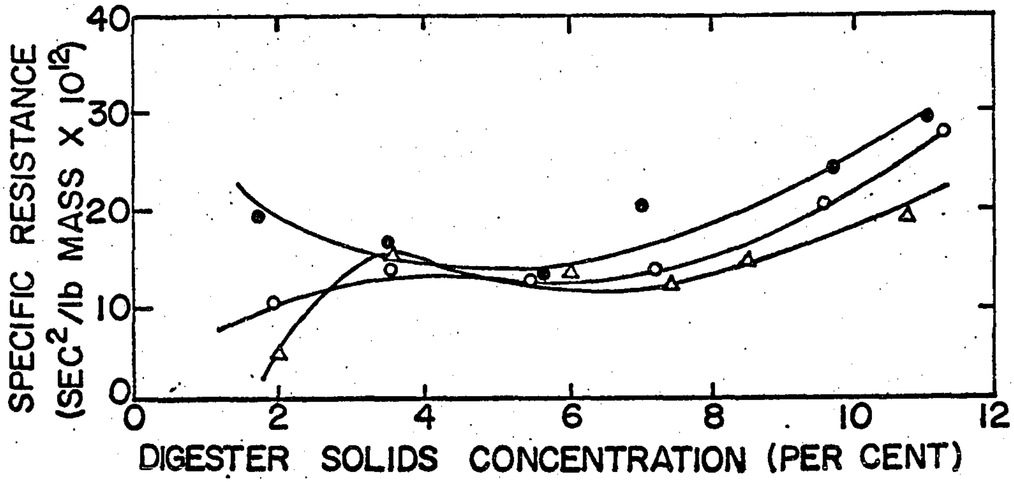
Figure 32. Effect of detention time on specific resistance for digesters 1 through 6, Run 3

Figure 33. Effect of solids concentration on specific resistance at detention times of 10, 20 and 30 days, Run 3

Symbol	
●	10 day detention time
○	20 day detention time
△	30 day detention time


SPECIFIC RESISTANCE
(SEC² /lb MASS X 10¹²)
0
10
20
30
40
50
60
0
0.2
0.4
0.6
0.8
LOADING RATE (LB VOLATILE SOLIDS ADDED/CU FT/DAY)


SPECIFIC RESISTANCE
(SEC² /lb MASS X 10¹²)
0
10
20
30
40
50
60
6
5
4
1
2
3
0
10
20
30
DETENTION TIME (DAYS)


SPECIFIC RESISTANCE
(SEC² /lb MASS X 10¹²)
0
10
20
30
40
0
2
4
6
8
10
12
DIGESTER SOLIDS CONCENTRATION (PER CENT)

and the associated detention times which were obtained from Table 24. An increase in specific resistance was observed with the sludges in all the digesters as the detention time decreased. The specific resistances of the sludges in digesters 5 and 6 deviated from the behaviour of the sludges in the other digesters. As the detention time decreased the specific resistance of these sludges increased rapidly until maximum values of approximately 32.0 and 57.0 sec^2 per lb mass x 10^{12} were reached for digesters 5 and 6 respectively at a detention time of 11.3 days. As the detention time was decreased below 11.3 days a rapid decrease in specific resistance of the sludges in both digesters was noticed. The scatter of data in Figure 31 was mentioned previously. Three isolated points showing high specific resistance were observed between loading rates of 0.4 to 0.7 lb volatile solids added per cu ft per day. If a line were drawn through the three isolated points a trend would be indicated which would agree with the trend shown in Figure 32. In Figure 31 the trend of the line indicates that as the loading rate increases the specific resistance reaches a maximum and then decreases rapidly. At the high specific resistance levels, which appear to be immediately prior to digestion failure, the sludge would be relatively difficult to dewater. Above a loading rate of 0.5 lb volatile solids added per cu ft per day in Figure 31 and below a detention time of 11.3 days in Figure 32, the ability to dewater the sludge increased rapidly as indicated by the decrease in specific resistance. Figure 29 shows that digesters 5 and 6 were sour at these conditions. The conclusion which may be drawn from the foregoing discussion is that the sour sludge in this study was easier to dewater than sludge undergoing alkaline digestion but close to failure.

Pöpel (128) reported data which appeared to confirm the results of this study. Although Pöpel's studies were not concerned with sludge filtration, as we were in the use of the specific resistance test, his data indicated that digested sludge after only a few days of digestion (3 to 5.7 days) was able to concentrate better than was sludge which had digested for periods above or below the times mentioned. He showed that optimum sedimentation of digested sludge solids could be obtained after digestion times of 4 to 5 days. The results indicate that the dewatering characteristics of digested sludge solids, as measured by solids-liquid separation, are better in sour digestion than in alkaline digestion close to failure.

Data from Figures 32 and 22 were used to plot Figure 33. Specific resistances for each digester were extracted from Figure 32 at 10, 20 and 30 day detention times and plotted against the corresponding solids concentrations at the same detention times (Figure 22). The trend for the 10 day detention time showed a minimum specific resistance at a solids concentration about 4 to 6 percent. With 20 and 30 day detention times, the specific resistance increased with the digester solids concentration. However, the specific resistance for these detention times remained relatively constant for solids concentrations between 3 and 8 percent. Above 6 percent solids concentration the trends for the 10, 20, and 30 day detention times do show that specific resistance decreases as the detention time increases.

In summary, the results obtained in this study on the dewatering ability of digested sludge indicate that:

1. Specific resistance increases slightly as loading rate increases.
2. Specific resistance increases as detention time decreases.
3. Sour digesting sludge may have better dewatering characteristics than alkaline digesting sludge close to digestion failure.

The cause of the increase in specific resistance for sludges at a solids concentration of 8 percent or more was not clear. A possible explanation was the entrapment of gas bubbles in the sludge as it became more viscous. At times of high gas production, the sludges from digesters 5 and 6 resembled a thick foam in consistency. This would tend to retard the release of liquid from the sludge. The sudden decrease in specific resistance of the sludge from digesters 5 and 6 at 10.6 days detention time may have been due to the presence of acid conditions inside the digester. The gasification process would be reduced under these conditions thus eliminating any retarding effects on the release of liquid from the sludge due to foam. The pH change was not large when the digesters turned sour so it is doubtful if this was the cause.

The rapid decrease in the specific resistances of the sludges in digesters 5 and 6 when acid conditions prevailed may be of importance in the design of continuous flow anaerobic digesters. This is discussed in the section on recommendations.

VI. CONCLUSIONS

The effect of solids concentration in a digester on the progress of anaerobic digestion was evaluated in this study by periodically increasing the loading rate to six completely mixed digesters maintained at 95°F. The digesting sludge in the digesters was maintained at about 1.9, 3.5, 5.8, 7.4, 9.0, and 10.6 percent total solids concentration. The following conclusions were reached:

1. There is an optimum solids concentration in digesting sludge at which maximum gas production can be expected. In this study, the optimum solids concentration appeared to be between 3 and 5 percent. With higher solids concentrations the gas production decreased approximately linearly.
2. As the detention time increases, or the loading rate decreases, the gas production increases.
3. Optimum solids concentrations appear to exist for maximum calculated volatile solids reductions. In this study, the optimum solids concentrations were between 4 and 7 percent. Theoretically, the optimum solids concentrations should have been the same for both maximum gas production and maximum volatile solids

reduction.

4. To obtain an accurate measure of the volatile solids reductions in digesting sludge, the volatile solids in the digesting sludge must be at an equilibrium level.
5. A maximum gas quality of 67.9 percent methane and 32.1 percent carbon dioxide was obtained from digesting sludge with an average solids concentration of 5.8 percent.
6. As the solids concentration in digesting sludge increases the normal operating concentration of volatile acids in the digesting sludge also increases. A volatile acids concentration of greater than 5000 mg/l was attained with only partial digestion failure.
7. The presence of propionic acid in digesting sludge indicates the onset of unstable digestion conditions. Butyric and valeric acids do not necessarily indicate unstable digestion conditions, since they were observed in digesting sludge during periods of stable digestion.
8. The allowable loading rates to digesters without causing digestion failure increase as the digester solids concentration increases. A maximum loading rate will probably be reached

beyond which digestion failure will occur because mixing will not be as effective in giving uniform conditions of digestion, or possibly because the detention time in the digester will be critical with regard to the generation time of the gas forming bacteria.

9. A minimum detention time of 8.8 days was obtained without digestion failure at a solids concentration of 5.30 percent and a loading rate of 0.37 lb volatile solids added per cu ft per day. The generation time of the gas producing bacteria was calculated, using the minimum time detention time achieved to be approximately 5 days.
10. The maximum loading rate achieved in this study, without digestion failure was 0.56 lb volatile solids added per cu ft per day at 11.3 days detention time and a digesting sludge solids concentration of 11.3 percent.
11. The pH of the digesting sludge decreased as the loading rate to a digester increased and the detention time decreased.
12. As the solids concentration in digesting sludge increases the alkalinity of the sludge increases. At 11.2 percent solids an alkalinity of about 8000 mg/l was observed.

13. The alkalinity of digesting sludge increased as detention time decreased.

14. The digesters in this study which maintained high alkalinities in the digesting sludge could be operated with high volatile acids concentrations in the digesting sludge.

15. The specific resistance, a measure of the dewatering characteristics of sludge, of the digesting sludge increases as the loading rate increases and the detention time decreases.

16. Digesting sludge which has turned sour appears to have better dewatering characteristics than alkaline digesting sludge close to digestion failure.

VII. RECOMMENDATIONS

The results in this study indicated that the domestic sewage sludge used digested best at a digester solids concentration of 3 to 5 percent based on gas production per lb of volatile solids added and 4 to 7 percent based on the maximum calculated volatile solids reductions. Theoretically, the optimum solids concentration should be the same in both cases. The maximum quantity of gas produced should result from a maximum reduction in volatile solids. The reason for the apparent inconsistency is probably the non-equilibrium conditions which existed in the volatile solids content of the digesting sludge. A proposed area of study is the determination of the effect of solids concentration on volatile solids reductions under equilibrium conditions. The general trends of volatile solids reductions have been indicated in this dissertation. Data, taken under equilibrium conditions, are required to locate more precisely the optimum solids concentration at which actual maximum volatile solids reductions exist. The study could be used to either confirm or repudiate the trends shown here.

The experimental portion of the study could consist of a run made at a single detention time, say 15 days which is common practice, and at digesting sludge concentrations of 1, 2, 3, 4, 5 and 7 percent. Solids concentrations of 1, 2 and 3 percent are necessary to confirm the rising limb in the gas production versus solids concentration graph (Figure 23) and the rising limb in the volatile solids reduction versus solids concentration graph (Fiugre 26). The data obtained from the 3, 4 and 5 percent sludges will give a more precise determination of the optimum sludge concentration for digestion based on the maximum volatile solids reduction. A digester containing 7 percent total solids sludge should be included in the experiment to confirm observable trends of the data from the digesters containing a sludge with lower solids concentrations. At a 15 day detention time, the total length of the run would probably be; 5 to 6 days for digester start up; 10 to 15 days to achieve a detention time of 15 days without creating abnormal conditions in the digesting sludge; 43 days after reaching the 15 day detention time to obtain a 95 percent turnover of the digesting sludge; and 10 days to collect data under equilibrium conditions. The total run time would be approximately 74 days. During the study other data can also be collected such as gas production per lb of volatile solids destroyed, volatile acids concentrations, alkalinity concentrations, gas qualities, and the specific resistance of the various sludges.

The time required to obtain a certain degree of turnover of the contents of a completely mixed digester which is fed at periodic intervals depends on:

1. The ratio of the volume of raw sludge added to the digester at each feeding, to the volume of

of digesting sludge.

2. The frequency of feeding.

The time required to obtain a percentage turnover of the digester contents equal to S can be formulated as,

$$S = 100(1-r^n)$$

where S, r and n have been defined previously. In preceding sections n has been defined as the number of days required to obtain the specified degree of turnover. Actually, n is the number of feedings which, in this study where the digesters were fed once a day, is equal to the number of days.

Another proposed area of study was suggested by the results obtained from volatile acids and sludge dewatering determinations. The main cause of upset in anaerobic digestion is the sensitivity of the gas forming bacteria to their environment. The need to preserve the activity of gas forming bacteria establishes an effective control upon the rate at which digesters can be operated. If it were not necessary to maintain these bacteria, the possibility exists that very high loading rates and correspondingly low detention times could be used in digestion. The end product of such a process would be a sour sludge containing organic acids, alcohols, ketones and other compounds normally present in sour sludge. All of these compounds are capable of being metabolised readily by aerobic organisms. If the organic acids etc., are placed, at a reasonable rate, into the main flow of sewage in a sewage treatment plant during periods of normal low flow then could be stabilised in the aerobic biological treatment process. Envisioned in this process is continuous or near continuous feed of raw sludge to a small, heated, completely mixed digester. The detention will possibly be two days or less. Inside the digester, liquefaction of the volatile solids takes place. The results of this study indicated that the acid forming bacteria can work in volatile acid concentrations greater than 5000 mg/l. Experiments have been reported in the literature in which volatile acid concentrations of 55,000 mg/l have been tolerated by the acid forming organisms. A constant displacement of sludge from the digester, equivalent in volume to the quantity of raw sludge entering the digester, will be transferred to a settling tank. Before reaching the settling tank settled sewage could be mixed with the partially digested sludge. The solid material in the sludge would settle in the settling tank and the organic liquids in the sludge would leave the settling tank by means of the overflow weir. The settled solids would be pumped to drying beds. Specific resistance values of sour sludge indicate that the solids will dewater rather readily. The organic liquids washed from the solids will be directed into the main sewage flow to enter the aerobic biological unit employed by the plant.

The advantages of this system over conventional digestion would be:

1. Smaller digesters would be required.

2. The heat requirements will be less.
3. The process will be more stable.
4. More efficient use may be made of the aerobic biological oxidation unit.
5. No gas collection device will be required.

The disadvantages are:

1. There will be no gas production.
2. Extra equipment for separating the digester solids from the digester liquid will be required.

The factors which would need further evaluation are: the quantity of solid organic matter which is converted to liquid organic matter in the digester; the ease of separating digester solids from the organic acids etc., in the settling tank; the ability to treat the liquid anaerobic end products aerobically; and the characteristics of the digested solids.

The characteristics of the methane forming organisms were discussed in the Literature Review. Some of the organisms required carbon dioxide in their metabolic processes. Morgan (116) studied the effect of gas mixing with digester gas and found that the rate of digestion of sewage sludge could be increased. The reason advanced for the improved rate of digestion was the efficient mixing obtained in the process. A study which may be worthy of further investigation is to determine the effect of varying the carbon dioxide content of the gas used for mixing the sludge.

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IX. ACKNOWLEDGEMENTS

The author is appreciative of the assistance provided by numerous people in this study. He would like to express appreciation to Dr. E. R. Baumann for his help and guidance in all phases of the work. He would also like to thank the staffs at the Ames, Iowa, and Nevada, Iowa, sewage treatment plants for their assistance in collecting sewage sludge; R. M. Bethea for the advice given with regard to the work on gas chromatography; J. O. Shearman for writing a computer program to account for solubility of carbon dioxide in sewage sludge when calculating gas volumes; the staff of sanitary engineering section of Iowa State University who have all assisted at some time during this study.

This study was supported by funds from the Engineering Experiment Station, Iowa State University of Science and Technology, Ames, Iowa.

X. APPENDIX

Table 13. Total solids in raw sludge[a], Run 2

	Digester					
Days	1	2	3	4	5	6
1-7	1.71	4.20	5.82	8.56	10.28	12.37
8	1.94	3.84	6.69	8.75	10.93	12.37
9	1.94	3.84	6.69	8.75	9.15	12.37
10	1.94	3.84	6.69	8.75	9.15	12.37
11	1.94	3.84	6.69	8.75	10.86	12.37
12-13	2.30	4.60	6.81	8.94	10.57	12.37
14	2.30	4.60	6.81	8.94	10.57	12.16
15-16	2.30	4.79	7.10	8.97	10.41	12.16
17	2.30	4.60	7.14	8.92	10.56	11.10
18-21	2.36	4.72	7.12	8.92	10.57	11.10
19		NO	FEED			
22-23	2.36	4.72	7.12	8.88	10.57	11.10
24		NO	FEED			
25	2.24	4.60	7.14	8.92	13.23	13.65
26	2.01	4.07	6.06	8.04	10.07	12.23
27-31	2.01	4.07	6.06	----[b]	10.07	12.23
32	2.01	4.07	6.06	----	10.12	12.37
33-35	1.99	4.10	6.07	----	-----	12.37
34		NO	FEED			
36	1.99	4.10	6.07	----	-----	12.37
37	1.99	4.10	6.07	5.09	11.48	12.37
38	2.08	4.10	6.07	8.20	11.26	12.32
39	2.08	4.10	6.07	8.20	10.01	12.32
40-43	1.90	4.10	6.07	8.20	10.01	12.32
44-45	----	4.10	6.07	8.20	10.01	12.32
46	5.09	3.93	5.97	7.95	10.01	12.19
47	----	3.93	5.97	----	-----	12.19
48	2.13	4.06	6.15	8.00	10.11	12.03

[a] Percent.

[b] No feed.

Table 14. Total solids in raw sludge, Run 3

Days	Digester					
	1	2	3	4	5	6
2-12	2.10	4.20	6.00	8.87	11.75	14.62
13-18	2.00	4.20	6.00	7.91	10.31	11.75
19	2.04	4.20	6.00	7.91	10.31	11.75
20-37	2.40	4.80	7.44	8.87	11.75	13.18
38-40	2.56	4.79	7.39	8.89	11.39	13.39
41	2.42	4.84	7.10	11.00	11.30	15.90
42	2.48	4.96	8.96	9.71	13.20	15.60
43-50	2.36	4.72	6.93	8.92	11.96	14.05
51-55	2.36	4.72	7.02	9.31	12.37	13.90
56-59	2.36	4.72	6.68	8.63	11.24	12.53
60-62	2.36	4.72	7.02	9.33	12.40	13.93
63-66	2.36	4.72	7.06	9.44	13.10	14.22
67	2.36	4.72	7.06	9.44	12.97	14.22
68-71	2.36	4.72	7.02	9.43	12.83	13.80
72	2.45	4.91	7.19	9.38	12.85	13.84
73	2.45	4.91	7.19	9.38	12.85	13.84

Table 15. Volatile solids in raw sludge[a], Run 2

Days	Digester					
	1	2	3	4	5	6
1-7	69.0	71.1	70.7	67.8	70.8	70.4
8	72.0	72.0	72.0	72.0	71.6	70.4
9-10	72.0	72.0	72.0	72.0	69.2	70.4
12-13	72.0	72.0	72.0	72.0	71.5	70.4
14	72.0	72.0	72.0	72.0	71.5	71.2
15-16	72.0	72.0	72.0	72.0	71.7	71.2
17	72.0	72.0	72.0	72.0	71.5	69.2
18-23	72.0	72.0	72.0	72.0	71.6	69.2
19			NO FEED			
24			NO FEED			
25	72.0	72.0	72.0	72.0	71.1	71.1
26	71.8	71.8	71.8	71.8	71.8	71.7
27-31	71.8	71.8	71.8	----[b]	71.8	71.7
32	71.8	71.8	71.8	----	71.7	71.7
33-35	71.8	71.8	71.8	----	----	71.7
34			NO FEED			
36	71.8	71.8	71.8	----	----	71.7
37	71.8	71.8	71.8	61.6	66.5	71.7
38	71.8	71.8	71.8	71.8	67.0	71.7
39-43	71.8	71.8	71.8	71.8	71.8	71.7
44-45	----	71.8	71.8	71.8	71.8	71.7
46	61.6	71.8	71.8	71.8	71.8	71.7
47	----	71.8	71.8	----	----	71.7
48-49	72.0	72.0	72.0	72.0	71.9	71.8
50	----	72.0	72.0	72.0	71.9	71.8

[a] Percent

[b] No feed

Table 16. Volatile solids in raw sludge[a], Run 3

Days	Digester					
	1	2	3	4	5	6
2-12	76.7	76.7	76.7	74.5	71.2	70.0
13-19	76.7	76.7	76.7	75.2	73.3	72.2
20-37	76.7	76.7	75.7	74.4	72.3	71.1
38-40	59.3	59.3	59.1	58.6	58.2	57.7
41	59.0	59.0	59.0	58.2	58.0	57.2
42	59.0	59.0	59.0	58.4	57.8	57.2
43-50	72.7	72.7	69.7	67.3	63.0	60.4
51-55	72.4	72.4	72.7	72.6	72.5	72.5
56-59	72.4	72.4	71.8	71.0	70.0	69.6
60-62	72.4	72.4	71.9	71.6	70.7	69.6
63-67	72.4	72.4	71.9	71.3	70.5	70.3
68-71	72.4	72.4	72.2	72.0	71.7	71.7
72	72.2	72.2	72.0	71.9	71.8	71.7
73	72.2	72.2	72.0	71.9	71.8	71.7

[a] Percent

Table 17. Total solids in digester[a], Run 2

Day Sampled	Digester 1	2	3	4	5	6
1	1.42	3.60	4.97	7.60	8.37	11.15
17	1.76	3.28	5.09	7.60	9.11	11.44
26	2.11	3.25	5.09	7.68	9.00	11.48
50	2.11	3.25	5.09	7.68	9.00	11.48

[a] Percent

Table 18. Total solids in digester[a], Run 3

Day Sampled	Digester 1	2	3	4	5	6
2	1.70	2.73	5.71	8.58	10.46	8.84
7	2.24	3.44	6.40	9.26	11.08	9.34
8	2.11	2.84	5.22	7.70	9.94	8.50
14	2.10	4.08	6.72	8.29	9.06	12.00
17	2.05	3.88	6.48	7.95	8.84	11.70
18	2.00	3.68	6.34	7.61	8.62	11.40
21	2.02	3.45	6.24	7.47	8.62	11.20
24	2.07	3.58	6.30	7.43	8.42	11.10
29	1.95	3.53	5.92	7.56	8.50	10.90
34	1.91	3.43	5.60	7.25	8.57	10.70
40	1.87	3.58	6.01	7.70	9.47	11.25
42	1.87	3.58	6.01	7.70	9.47	11.25
47	1.84	3.49	6.52	7.39	9.64	11.20
55	1.77	3.58	5.85	7.33	9.82	11.31
69	1.86	3.55	5.30	7.12	9.75	11.11
72	1.86	3.55	5.30	7.12	9.75	11.11

Table 19. Volatile solids in the digester[a], Run 2

Day Sampled	Digester 1	2	3	4	5	6
1	52.2	61.0	54.3	57.8	52.7	56.2
17	63.0	63.0	61.3	57.8	69.7	66.4
26	62.6	62.9	61.6	59.1	64.1	66.5
50	62.6	62.9	61.6	59.1	64.1	66.5

Table 20. Volatile solids in the digester[a], Run 3

Day Sampled	Digester 1	2	3	4	5	6
2	44.4	53.9	49.9	45.8	45.4	48.8
8	41.3	48.3	46.3	41.8	42.4	46.8
9	38.7	48.5	46.7	42.9	42.0	46.5
15	41.5	51.7	47.1	44.3	44.8	49.2
18	41.5	51.7	47.1	44.3	44.8	49.2
19	41.6	50.9	47.9	45.0	44.4	48.4
22	43.4	52.8	51.3	45.7	46.2	48.6
25	43.4	51.8	47.4	47.0	45.1	48.6
30	44.4	52.3	47.8	44.5	44.2	48.3
35	46.9	53.3	48.7	45.9	45.0	48.6
41	50.9	55.0	50.3	48.4	47.1	49.4
43	50.9	55.0	50.3	48.4	47.1	49.4
48	48.7	55.3	50.1	50.5	46.2	49.4
56	55.3	57.5	52.7	51.5	51.0	53.7
70	63.8	61.8	59.1	55.6	56.1	57.1
73	63.8	61.8	59.1	55.6	56.1	57.1

[a] Percent

Table 21. Volatile solids loading rates[a]

Day	Digester 1	2	3	4	5	6
1	6.0	7.5	6.9	8.4	10.0	10.7
2	5.9	7.6	7.0	8.3	10.0	10.7
3	6.1	7.6	7.0	8.3	7.2	10.7
4	7.5	10.1	10.3	11.6	12.0	10.7
5	7.4	12.2	11.6	14.0	14.9	10.7
6	7.5	12.0	11.4	13.8	14.9	10.7
7	7.3	11.8	11.4	14.2	14.7	----[b]
8	11.1	16.4	11.5	20.6	25.3	----
9	11.0	17.5	23.4	30.5	25.5	----
10	11.1	17.5	23.6	30.8	25.4	----
11	11.0	21.8	30.8	40.7	43.9	----
12	13.2	26.2	31.2	41.4	48.9	----
13	13.0	26.6	32.1	42.5	52.7	----
14	13.0	26.2	31.3	42.1	49.4	21.2
15	13.0	22.1	36.4	46.8	54.4	21.8
16	13.0	22.4	37.1	47.5	54.9	14.3
17	13.2	26.2	41.6	51.6	60.8	31.4
18	12.0	29.5	46.1	52.4	55.9	31.7
19	----	----	----	----	----	----
20	12.0	30.0	44.9	51.6	61.9	31.4
21	12.2	29.5	46.1	52.4	61.2	31.4
22	12.0	29.9	44.9	58.1	61.2	----
23	12.0	29.5	46.1	56.6	62.8	----
24	----	----	----	----	----	----
25	10.3	26.6	41.6	51.6	68.6	32.1
26	9.2	22.8	35.1	47.2	53.3	29.4
27	9.1	23.1	36.5	----	52.0	28.7
28	9.1	23.4	35.1	----	52.8	28.2
29	9.1	23.4	35.1	----	53.4	29.0
30	9.0	22.9	35.5	----	52.7	27.9
31	9.7	23.1	35.5	----	41.4	29.4
32	9.1	28.4	41.7	----	29.0	43.6
33	11.2	28.0	42.4	----	----	43.6
34	----	----	----	-----	----	----
35	11.4	28.0	43.4	----	----	58.4

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[a] lb per day per cu ft of digesting sludge x 10^{-2}

[b] No feed

Table 21 (Continued)

Day	Digester 1	2	3	4	5	6
36	11.2	28.0	41.7	----	----	58.4
37	11.2	28.8	42.3	24.4	48.1	57.8
38	5.9	28.0	42.3	14.4	55.4	57.8
39	5.9	28.0	42.3	14.2	11.5	57.8
40	7.5	28.4	42.3	14.2	11.7	60.0
41	7.5	28.4	41.7	14.4	11.5	57.8
42	7.5	28.0	42.3	14.2	11.8	58.4
43	7.6	28.4	41.7	14.2	12.0	59.3
44	----	28.4	42.8	14.4	12.5	58.4
45	----	27.7	42.3	11.0	12.5	57.8
46	10.5	29.4	44.4	23.3	22.4	57.9
47	----	14.4	22.5	----	----	28.2
48	2.5	29.7	45.9	23.2	25.1	57.1
49	2.5	30.5	45.9	23.2	24.9	56.5
50	----	30.1	45.9	22.8	23.6	56.4
51	----	----	----	----	----	----

Table 22. Volatile solids loading rates[a], Run 3

Day	Digester					
	1	2	3	4	5	6
2	1.3	2.5	3.7	5.4	6.8	8.3
4	1.3	2.5	3.6	5.4	6.8	8.3
5	1.3	2.5	3.7	5.3	7.0	8.3
6	1.3	2.5	3.8	5.4	7.1	8.5
7	1.3	2.6	3.8	5.3	7.1	8.5
8	1.3	2.5	3.7	5.3	6.8	8.3
9	1.3	2.6	3.7	5.3	6.8	8.4
10	1.2	2.6	3.7	5.3	6.8	8.3
11	1.2	2.5	3.6	5.3	6.7	8.2
12	1.8	3.9	5.6	7.4	9.5	11.0
13	1.8	3.9	5.5	7.3	9.2	11.1
14	1.8	3.6	5.2	7.0	9.0	10.8
15	1.8	3.8	5.6	7.4	9.3	11.0
16	1.8	3.8	5.5	7.2	9.2	11.0
17	1.8	3.8	5.5	7.1	9.0	10.8
18	2.0	6.1	5.3	7.0	8.9	10.7
19	3.6	7.2	6.7	7.9	10.2	12.1
20	3.5	7.1	11.3	13.3	10.2	12.3
21	3.5	7.2	11.3	13.1	10.3	12.3
22	3.5	6.9	11.0	12.8	10.1	12.1
23	3.4	6.6	10.6	12.5	10.1	11.8
24	3.3	6.5	10.4	12.1	9.9	11.6
25	3.4	7.0	10.7	12.5	13.1	15.8
26	3.3	6.6	10.5	12.2	12.9	15.3
27	3.3	6.6	10.5	12.2	13.2	15.6
28	3.4	6.8	10.6	12.7	13.6	15.3
29	5.3	10.5	16.8	19.3	19.8	22.7
30	5.4	10.7	17.2	20.0	20.6	23.3
31	5.4	10.7	17.4	20.2	20.6	23.3
32	5.7	11.2	18.0	21.0	20.7	23.3
33	5.8	11.6	17.8	21.2	20.6	22.7
34	5.8	11.6	18.2	21.2	20.5	23.6
35	5.8	11.8	18.0	21.5	20.9	23.6
36	5.8	11.6	18.2	21.2	20.5	23.3
37	6.0	11.3	17.4	21.0	21.7	25.5

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[a] lb per day per cu ft of digesting sludge x 10^{-2}

Table 22 (Continued)

Day	Digester					
	1	2	3	4	5	6
38	6.1	11.4	17.4	21.4	21.3	25.3
39	6.0	11.1	11.0	21.0	21.7	25.5
40	5.6	11.5	16.6	25.8	21.2	29.5
41	5.8	11.8	21.4	23.2	24.6	29.5
42	6.7	13.8	19.3	24.1	30.9	34.8
43	6.7	13.4	19.3	24.1	30.0	34.8
44	6.7	13.8	19.7	24.1	30.9	34.8
45	6.8	13.6	19.3	24.6	30.6	35.0
46	6.7	13.6	19.3	23.8	31.1	35.0
47	6.8	13.8	19.7	24.6	30.6	34.8
48	6.8	13.6	19.3	24.8	30.9	35.0
49	6.7	13.8	19.7	24.1	30.5	34.8
50	9.5	19.2	28.7	38.6	50.7	59.0
51	9.4	18.9	28.9	37.6	51.3	59.0
52	9.4	18.9	28.9	38.6	51.3	58.3
53	9.4	19.0	28.6	38.6	50.7	59.0
54	9.5	19.2	28.7	37.9	50.7	57.6
55	9.4	18.9	27.1	34.4	45.2	49.7
56	9.3	18.9	26.8	34.8	45.1	50.4
57	9.5	18.9	26.8	34.4	43.8	49.2
58	9.4	18.9	28.1	37.7	49.0	56.6
59	9.4	19.1	28.6	38.0	50.4	55.8
60	12.1	24.4	36.4	38.4	49.0	55.0
61	12.2	24.6	36.9	37.6	50.4	55.7
62	12.1	24.4	36.7	37.7	52.9	57.7
63	12.1	24.4	37.4	38.2	26.2	57.7
64	12.1	24.4	36.5	38.2	26.2	57.7
65	12.4	24.4	37.0	38.2	25.9	56.2
66	14.6	29.8	36.5	37.8	45.7	57.7
67	14.6	30.3	45.4	44.2	45.0	64.5
68	15.0	29.5	44.6	44.2	45.0	65.3
69	15.0	29.5	44.6	44.2	45.0	65.3
70	15.0	29.5	44.6	44.2	45.0	65.3
71	15.3	30.9	46.1	43.9	44.5	65.6
72	15.3	30.9	46.1	43.9	44.5	65.6

Table 23. Digester detention times[a], Run 2

Day	Digester					
	1	2	3	4	5	6
1	12.3	25.0	38.1	44.5	47.0	53.3
2	12.5	24.7	37.6	45.0	47.0	53.3
3	12.2	24.7	37.6	45.0	65.0	53.3
4	9.9	18.8	25.5	32.0	39.0	53.3
5	10.0	15.6	22.6	26.7	31.6	53.3
6	9.9	15.8	22.9	27.0	31.6	53.3
7	10.0	16.0	22.9	26.3	32.0	----[b]
8	7.9	10.7	15.8	16.6	20.0	----
9	8.0	10.0	13.2	13.3	16.0	----
10	7.9	10.0	13.0	13.2	15.8	----
11	8.0	8.0	10.0	10.0	11.4	----
12	8.0	8.0	10.0	9.9	10.1	----
13	7.9	8.0	10.0	10.0	10.0	----
14	8.0	7.9	9.8	9.7	10.0	----
15	8.0	9.9	9.2	8.9	8.9	25.0
16	8.0	9.7	8.8	8.8	8.8	26.0
17	7.9	8.0	7.9	8.0	8.1	16.0
18	8.9	7.3	7.1	7.9	8.8	15.8
19			NO FEED			
20	8.9	7.7	7.3	8.0	7.9	16.0
21	8.8	7.3	7.1	7.9	8.0	----
22	8.9	7.2	7.3	7.1	8.0	----
23	8.9	7.3	7.1	7.3	7.8	----
24			NO FEED			
25	9.9	7.9	7.9	8.0	8.9	19.8
26	9.9	8.1	7.9	7.9	8.9	19.5
27	10.0	8.0	7.6	---	8.8	20.0
28	10.0	7.9	7.9	---	8.9	20.3
29	10.0	7.9	7.9	---	8.8	19.8
30	10.1	8.1	7.9	---	8.9	20.5
31	9.8	8.0	7.8	---	11.3	19.5
32	10.0	6.6	6.7	---	16.2	13.3
33	8.0	6.7	6.6	---	----	13.3
34			NO FEED			
35	7.9	6.7	6.4	---	----	9.9

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[a] Days

[b] No feed

Table 23 (Continued)

Day	Digester					
	1	2	3	4	5	6
36	8.0	6.7	6.7	----	----	9.9
37	8.0	6.5	6.6	8.3	10.3	10.0
38	15.8	6.7	6.6	26.3	8.8	10.0
39	15.8	6.7	6.6	26.6	40.5	10.0
40	11.4	6.6	6.6	26.6	40.0	9.6
41	11.4	6.6	6.7	26.3	40.5	10.0
42	11.4	6.7	6.6	26.6	39.5	9.9
43	11.3	6.6	6.7	26.6	39.0	9.7
44	----	6.6	6.5	26.3	37.5	9.9
45	----	6.8	6.6	26.3	37.5	10.0
46	18.7	6.1	6.2	15.8	20.7	9.9
47	----	12.4	12.2	----	----	20.2
48	38.0	6.2	6.2	16.0	18.7	9.9
49	38.0	6.1	6.2	16.0	19.0	10.0
50	----	6.2	6.2	16.2	20.0	10.0
51	----	----	6.2	15.8	20.0	10.0

Table 24. Digester detention times[a], Run 3

Day	Digester 1	2	3	4	5	6
2	83.20	79.0	80.0	79.0	80.0	80.0
3	NO FEED					
4	79.0	79.0	81.0	79.0	80.0	80.0
5	80.0	80.0	79.0	80.0	78.0	80.0
6	80.0	80.0	78.0	79.0	77.0	78.0
7	79.0	80.0	78.0	80.0	77.0	78.0
8	----[b]	----	----	----	----	----
9	79.0	79.0	80.0	80.0	80.0	79.0
10	81.0	79.0	79.0	80.0	80.0	80.0
11	82.0	81.0	81.0	81.0	82.0	81.0
12	52.7	52.0	52.7	52.7	52.0	50.0
13	54.0	52.7	53.4	53.4	53.4	49.3
14	54.6	56.0	56.7	55.4	54.6	51.3
15	53.4	54.0	52.7	52.7	52.7	50.6
16	53.4	53.4	53.4	54.0	53.4	50.6
17	54.0	54.0	54.0	54.6	54.6	51.3
18	33.2	33.2	55.4	56.0	54.6	52.0
19	32.4	32.4	32.4	32.4	54.0	50.6
20	32.8	32.8	32.0	32.0	54.0	50.0
21	32.8	32.4	32.0	32.4	53.4	50.0
22	33.2	34.0	32.8	33.2	54.6	50.6
23	34.4	35.2	34.0	34.0	54.6	52.0
24	35.6	36.0	34.8	35.2	55.4	52.7
25	34.4	34.4	33.6	34.0	42.0	39.0
26	35.2	35.6	34.8	34.8	42.5	40.0
27	35.2	35.2	34.4	34.8	41.5	39.5
28	34.0	34.4	34.0	33.6	40.5	40.0
29	21.7	22.2	21.5	22.0	27.7	27.3
30	21.2	21.7	21.0	21.2	26.6	26.6
31	21.5	21.7	20.7	21.0	26.6	26.6
32	20.2	20.7	20.0	20.2	26.3	26.6
33	19.7	20.0	20.2	20.0	26.6	27.3
34	20.0	20.0	19.7	20.0	26.6	26.3
35	20.0	19.7	20.0	19.7	26.3	26.3
36	20.0	20.0	19.7	20.0	26.6	26.6

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[a] Days

[b] Digester not weighed hence can not calculate detention time.

Table 24 (Continued)

Day	Digester					
	1	2	3	4	5	6
37	15.8	16.0	16.0	16.0	19.7	19.7
38	16.0	15.8	16.0	15.8	20.0	20.0
39	16.2	16.2	16.0	16.0	19.7	19.7
40	15.8	15.8	16.0	16.0	20.0	20.0
41	15.8	15.8	15.8	15.8	19.7	20.0
42	16.0	15.8	16.0	16.0	15.8	16.0
43	16.0	16.2	16.0	16.0	16.2	16.0
44	16.2	15.8	15.8	16.0	15.8	16.0
45	15.8	16.0	16.0	15.8	16.0	15.8
46	16.2	16.0	16.0	16.2	15.6	15.8
47	15.8	15.8	15.8	15.8	16.0	16.0
48	15.8	16.0	16.0	15.6	15.8	15.8
49	16.2	15.8	15.8	16.0	16.0	16.2
50	11.3	11.3	11.4	11.3	11.4	11.6
51	11.4	11.4	11.3	11.6	11.3	11.1
52	11.4	11.4	11.3	11.3	11.3	11.1
53	11.4	11.4	11.4	11.3	11.4	11.3
54	11.3	11.3	11.4	11.4	11.4	11.1
55	11.4	11.4	11.3	11.3	11.3	11.4
56	11.6	11.4	11.4	11.3	11.3	11.3
57	11.3	11.4	11.4	11.4	11.4	11.6
58	11.4	11.4	11.4	11.4	11.4	11.1
59	11.4	11.3	11.3	11.3	11.3	11.3
60	8.9	8.9	8.9	11.3	11.4	11.4
61	8.8	8.8	8.8	11.4	11.3	11.3
62	8.9	8.9	8.9	11.4	11.3	11.3
63	8.9	8.9	8.7	11.3	22.6	11.3
64	8.9	8.9	8.9	11.3	22.6	11.3
65	8.9	8.9	8.8	11.3	23.2	11.4
66	7.4	7.3	8.9	11.4	13.3	11.3
67	7.4	7.2	7.2	9.9	13.2	10.0
68	7.2	7.4	7.3	9.9	13.3	9.9
69	7.3	7.3	7.3	10.0	13.2	9.9
70	7.3	7.3	7.2	9.9	13.5	9.9
71	7.2	7.2	7.6	9.9	13.2	9.9
72	7.2	7.2	7.6	9.9	13.2	9.9

Table 25. Reduction in volatile solids[a], Run 2

Day	Digester					
	1	2	3	4	5	6
1	50.8	36.7	50.8	34.8	54.1	46.1
8	63.2	39.8	58.3	46.0	65.5	----[b]
14	70.1	40.6	61.2	46.0	79.3	59.1
17	33.5	33.2	38.2	46.7	86.8	11.9
18	33.5	33.2	38.2	46.9	85.4	----
25	33.2	33.2	38.4	48.4	25.4	19.7
26	34.5	33.4	37.0	43.3	29.8	21.5
32	34.5	33.4	37.0	43.3	29.5	21.5
37	34.5	33.4	37.0	----	----	----
38	34.5	33.4	37.0	43.3	----	----
39	34.5	33.4	37.0	43.3	29.8	21.5
48	35.3	34.0	37.8	44.0	27.9	22.1
50	35.3	34.0	37.8	44.0	27.9	22.1

[a] Percent. See section VB for method of calculation.

[b] No data.

Table 26. Reduction in volatile solids[a], Run 3

Day	Digester					
	1	2	3	4	5	6
2	75.7	64.4	69.9	71.0	66.5	59.2
8	78.5	71.6	73.7	75.4	70.3	62.2
9	80.9	71.4	73.4	74.2	70.7	62.8
13	82.3	74.3	73.6	76.4	75.7	68.7
15	78.5	67.5	73.0	73.8	70.5	62.8
19	78.3	69.8	71.1	72.2	71.6	65.0
20	78.4	68.6	70.4	71.8	69.4	61.8
22	76.7	66.1	66.1	71.1	67.1	61.5
25	76.7	67.4	71.1	64.0	68.5	61.5
30	75.7	66.7	70.7	72.4	69.7	62.1
35	73.3	65.3	69.6	70.8	68.7	61.5
38	41.2	22.1	34.8	41.1	42.1	31.0
41	27.9	15.1	29.6	32.6	35.5	26.9
43	61.1	54.0	55.9	54.5	47.6	36.0
48	64.3	53.5	56.3	50.5	49.6	36.0
51	67.3	51.7	63.9	62.0	70.6	65.9
56	52.8	48.5	56.2	56.7	55.3	49.2
60	55.8	49.5	58.3	59.0	58.4	50.2
63	60.3	51.0	61.0	59.9	57.0	53.0
68	63.1	54.8	64.4	63.3	64.5	57.7
70	32.9	38.2	44.3	51.4	49.7	47.5
72	32.0	37.7	44.0	51.1	49.9	47.5
73	32.0	37.7	44.0	51.1	49.9	47.5

[a] Percent. See section VB for method of calculation.

Table 27. Daily gas production[a], Run 2

Day	Digester					
	1	2	3	4	5	6
1	7.6	9.4	10.4	12.3	12.1	6.6
2	4.6	5.6	6.6	6.4	5.8	2.9
3	8.6	10.8	12.0	11.2	10.4	4.9
4	7.3	9.5	12.0	12.0	15.4	4.9
5	8.9	9.5	10.6	10.3	11.1	5.1
6	8.7	8.6	9.4	9.1	8.5	4.4
7	9.5	10.8	11.7	10.8	10.5	4.9
8	6.4	9.2	11.5	10.6	10.4	7.6
9	6.4	7.4	7.0	6.8	6.6	4.4
10	7.1	8.0	6.9	6.2	6.2	4.4
11	7.7	8.2	7.8	6.7	7.1	3.9
12	6.3	5.4	6.3	4.9	3.9	5.6
13	5.2	3.6	5.8	4.6	3.9	6.9
14	7.2	4.7	7.7	6.0	4.8	5.4
15	5.1	3.9	5.6	5.0	3.9	1.8
16	5.5	5.8	6.6	5.3	4.0	2.3
17	4.5	5.9	5.9	4.9	3.8	4.7
18	4.6	6.2	6.3	4.8	3.8	2.2
19	3.3	5.1	2.2	4.8	3.7	1.9
20	3.1	4.3	4.2	4.1	2.8	1.6
21	3.7	5.3	5.4	4.9	3.0	1.9
22	4.0	6.1	6.1	5.3	3.2	2.0
23	3.5	5.4	5.6	4.5	2.8	1.9
24	---[b]	---	---	---	---	---
25	3.5	4.2	4.0	3.8	2.0	2.6
26	3.6	5.1	5.1	4.1	1.8	1.8
27	4.5	6.3	6.4	4.6	2.3	2.6
28	4.4	6.2	6.4	4.6	2.0	2.8
29	4.8	7.6	8.1	4.6	1.9	3.0
30	4.2	6.7	7.0	4.6	1.4	2.7
31	4.3	6.3	6.7	4.6	1.5	2.8
32	4.6	6.6	6.8	4.6	1.1	3.0
33	3.1	5.1	5.6	4.6	0.6	1.8
34	3.0	5.1	5.4	4.6	0.5	1.9
35	3.2	4.7	4.6	4.6	0.8	2.3

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[a] Cu ft per lb volatile solids added.

[b] No gas measurement.

Table 27 (Continued)

Day	Digester 1	2	3	4	5	6
36	3.1	4.7	5.3	4.6	0.5	1.3
37	2.9	5.2	5.5	0.8	0.6	1.2
38	4.8	5.3	5.8	2.0	0.5	1.2
39	4.5	5.3	6.0	4.3	0.4	1.1
40	3.1	5.0	5.8	4.3	1.9	1.0
41	2.9	5.0	6.0	4.3	2.2	1.1
42	2.7	4.8	5.6	4.1	2.7	1.1
43	2.3	4.8	5.9	4.2	3.3	1.1
44	1.2	4.3	4.1	3.8	3.1	1.0
45	1.2	4.3	5.3	2.7	4.0	1.5
46	0.8	4.2	5.2	3.3	4.1	1.2
47	0.9	4.2	5.1	2.0	1.8	1.3
48	8.8	8.0	9.7	5.4	4.9	2.5
49	4.7	3.6	4.5	2.6	2.3	1.2
50	3.5	3.5	4.5	2.7	2.4	1.2
51	3.0	3.4	4.4	1.5	1.4	1.0

Table 28. Daily gas production[a], Run 3

Day	Digester					
	1	2	3	4	5	6
1	---[b]	---	---	---	---	---
2	3.2	3.9	3.3	2.3	2.0	2.1
3	---	---	---	---	---	---
4	6.5	6.5	5.3	3.1	3.0	3.4
5	8.7	8.2	7.6	4.1	3.7	4.0
6	7.9	8.1	6.5	3.9	3.3	3.8
7	10.0	8.2	6.5	3.7	3.3	4.0
8	26.7	18.5	15.1	9.6	8.4	4.6
9	27.3	18.0	14.3	9.1	7.8	5.2
10	21.3	12.6	10.4	6.8	5.8	5.0
11	19.5	15.2	12.7	8.8	7.3	6.2
12	17.2	15.0	13.5	7.1	7.7	5.3
13	18.3	11.4	10.6	8.6	6.2	5.1
14	11.6	13.0	11.9	9.2	6.7	3.5
15	12.5	14.4	13.0	10.6	8.0	5.8
16	14.2	17.1	14.5	12.5	10.2	7.6
17	12.1	15.7	16.0	12.9	10.7	7.8
18	13.7	16.3	16.9	14.0	10.6	7.7
19	8.9	11.4	17.7	15.1	11.2	8.1
20	8.9	10.1	13.7	13.9	10.0	7.2
21	10.2	10.5	8.5	8.9	10.3	7.3
22	11.6	11.6	9.7	10.2	11.7	8.2
23	10.7	11.3	9.7	10.4	11.6	7.9
24	10.7	10.6	9.5	10.3	11.9	8.5
25	11.5	12.6	11.3	10.3	12.9	9.2
26	13.6	13.3	11.9	10.9	10.1	7.2
27	12.2	12.5	11.6	10.6	10.1	7.6
28	11.9	12.3	10.9	10.6	10.2	8.2
29	10.5	11.5	10.8	9.7	9.6	8.1
30	9.7	9.4	8.5	7.2	7.0	5.9
31	9.3	9.2	8.3	7.4	6.9	5.7
32	10.7	10.9	10.0	8.6	8.3	5.6
33	9.9	9.7	7.6	7.8	8.3	6.4
34	10.9	11.3	10.3	8.6	8.6	7.1
35	11.5	11.5	10.5	9.0	7.7	7.3

---

[a] Cu ft per lb volatile solids added.

[b] No gas measurement.

Table 28 (Continued)

Day	Digester					
	1	2	3	4	5	6
36	10.5	13.6	9.3	9.0	8.2	6.9
37	10.5	10.7	9.5	8.8	9.3	7.1
38	7.0	7.8	7.8	7.1	7.3	6.2
39	7.3	7.9	8.1	7.3	7.0	6.3
40	6.7	7.3	7.1	6.6	6.5	6.1
41	6.5	6.5	6.6	5.4	6.7	5.3
42	7.8	7.7	6.2	6.7	6.4	5.9
43	5.2	5.2	4.8	4.3	3.6	3.0
44	9.9	10.1	9.5	8.3	6.9	5.7
45	8.5	8.6	7.9	7.6	6.3	5.2
46	9.1	9.0	8.3	8.0	6.9	5.8
47	9.1	9.2	8.5	7.9	7.1	6.1
48	8.2	8.9	7.6	6.9	6.8	5.6
49	8.9	9.0	8.3	7.8	7.0	5.6
50	8.9	9.1	8.4	7.8	7.0	5.8
51	6.7	7.0	6.3	5.3	4.6	3.6
52	7.9	8.0	7.7	6.6	5.5	4.5
53	8.0	8.3	8.0	7.0	5.7	4.7
54	8.3	8.6	8.0	7.1	6.0	5.0
55	9.5	8.9	7.9	7.1	6.0	4.9
56	8.3	8.4	7.8	7.3	6.4	5.2
57	8.3	8.2	7.7	6.8	6.2	4.9
58	8.2	8.1	7.6	6.7	5.9	4.5
59	8.9	8.9	8.2	7.3	5.8	4.8
60	8.9	8.8	8.3	7.6	5.7	5.2
61	7.7	7.8	7.7	7.9	5.8	5.6
62	7.9	7.8	6.5	7.9	5.5	5.7
63	8.0	8.2	6.8	7.7	4.5	5.3
64	7.6	7.3	6.7	7.4	7.6	5.0
65	7.8	7.5	6.9	7.4	7.9	5.1
66	8.0	7.6	6.9	7.9	7.8	5.0
67	6.6	6.2	7.0	7.6	4.2	5.3
68	5.6	6.0	5.8	7.0	4.4	3.8
69	4.1	5.7	5.4	6.2	4.2	4.0
70	2.9	5.3	4.2	6.5	3.9	4.1
71	1.6	2.7	3.4	5.8	3.0	4.0
72	1.1	1.8	2.4	5.0	2.0	2.4

Table 29. Gas quality[a], Run 2

Day Sampled	Digester					
	1	2	3	4	5	6
8	72.1	66.6	65.4	73.8	69.1	54.9
10	73.9	65.8	65.3	70.4	-----[b]	60.4
14	65.0	67.2	65.0	69.9	62.9	63.6
15	57.5	67.4	64.8	65.7	60.9	63.5
16	57.5	67.4	67.3	69.8	68.3	64.8
17	57.9	64.1	72.1	67.3	63.1	65.7
18	58.7	62.7	65.7	70.1	55.5	57.5
19	56.2	64.1	61.6	60.6	54.9	53.6
20	58.4	67.5	67.5	70.5	60.8	51.7
21	64.6	69.8	73.8	70.7	63.1	53.0
41	62.4	67.1	70.6	68.3	58.3	48.7
42	61.9	67.4	68.1	64.6	57.4	49.2
43	59.3	66.9	62.4	70.4	49.8	46.8
44	59.8	69.0	63.3	61.9	46.0	44.7
49	63.5	63.5	57.9	46.6	44.7	44.1

[a] Percent methane.

[b] No data.

Table 30. Gas quality[a], Run 3

Day Sampled	Digester					
	1	2	3	4	5	6
11	69.0	66.7	63.0	57.6	59.7	55.8
13	68.3	66.2	67.5	66.2	63.5	64.0
14	68.9	66.7	65.3	63.1	62.2	59.2
15	68.0	65.5	64.4	62.2	65.9	63.0
16	66.3	65.5	64.8	70.7	63.1	55.1
19	68.7	64.7	67.4	68.1	66.4	60.5
24	66.2	71.5	71.7	65.8	69.9	62.5
27	67.5	70.1	71.5	72.1	71.8	62.5
30	64.9	69.3	69.9	71.4	65.3	62.1
32	63.9	69.1	72.3	-----[b]	63.6	59.8
35	66.0	61.4	66.9	64.7	65.1	67.8
49	66.9	63.7	71.1	67.4	66.1	64.4
52	67.5	63.7	65.9	73.3	68.5	61.7
56	65.3	66.7	64.9	69.3	66.3	67.5
61	67.9	67.4	66.2	----	64.2	61.7
63	67.0	60.8	65.7	----	58.3	59.4
66	65.9	61.4	66.9	64.7	65.1	67.8
68	61.6	63.5	59.8	71.7	61.1	62.4

[a] Percent methane.

[b] No data

Table 31. Volatile acids in digesting sludge[a], Run 2

Day Sampled	Digester					
	1	2	3	4	5	6
3	30	-----[b]	130	250	100	4700
43	1220	730	570	3660	6660	6080
44	1330	810	770	3200	5800	5800
51	950	3970	4730	7880	11740	13880

Table 32. Volatile acids in digesting sludge[a], Run 3

Day Sampled	Digester					
	1	2	3	4	5	6
5	540	695	1460	1760	2020	1840
6	590	830	2020	3450	2800	2690
7	480	750	1730	2540	2540	2220
11	100	425	1480	2220	2410	2340
14	190	460	1230	1680	1830	2280
18	140	250	620	1070	1290	2080
20	130	190	530	1170	1790	2950
24	140	220	460	330	810	2870
26	40	210	410	380	270	----[b]
29	120	330	480	490	500	3060
30	170	270	600	650	500	3150
34	140	190	430	460	680	2960

---

[a] Mg/l as acetic acid

[b] No data

Table 32 (Continued)

Day Sampled	Digester					
	1	2	3	4	5	6
35	130	390	550	570	580	2990
44	90	110	240	350	450	1400
48	60	360	490	410	480	1230
51	120	120	350	180	640	1720
53	---	---	---	540	1610	3330
55	180	330	640	430	1590	3500
56	270	390	560	560	1580	3430
59	130	110	390	320	1560	4260
63	70	390	790	570	4110	5160
66	280	630	1080	450	4970	5680

Table 33. Digesting sludge pH, Run 3

Day Sampled	Digester					
	1	2	3	4	5	6
11	7.85	7.8	7.7	7.6	7.55	6.9
19	7.8	7.7	7.55	7.55	7.5	7.2
26	8.0	7.8	7.8	7.7	7.8	---[a]
53	6.85	7.0	7.0	7.0	6.95	6.8
64	7.1	7.15	7.05	7.2	6.9	6.9
70	6.5	6.65	6.6	6.95	6.45	6.6

[a] No data

Table 34. Alkalinity of digesting sludge[a], Run 3

Day Sampled	Digester					
	1	2	3	4	5	6
19	1435	2250	2930	3170	3550	4010
26	1600	2500	3300	3900	4300	----[b]
53	1600	2600	3400	4500	----	4800
64	1500	2900	3800	5100	6900	8000

[a] Mg/l as calcium carbonate

[b] No data

Table 35. Time requirements for the various operations and analyses in this study[a]

Operation or analysis	Time required (hours)	Remarks
Raw sludge collection and storage	9	Sufficient for 20 days
Raw sludge thickening and storage	2[b] 72[c]	Sufficient for 14 days
Feed sludge preparation	1[b] 6[c]	Thawing and make-up
Digester feeding and clean up	3	
Gas pressure	0.4	
Gas analysis	0.25	
pH	0.25	
Volatile acids	2[b] 3[c]	
Alkalinity	1.25	
Total and volatile solids	3[b] 26[c]	
Qualitative volatile acids analysis	1.5[b] 5[c]	
Dewatering test	4-6	
Equipment maintenance	1	per day

[a] For six digesters.

[b] Actual working time.

[c] Time from start to end of the analysis.

Figure 34. Computer program for calculating carbon dioxide dissolved in sewage sludge. (Fortran II)

```
      DIMENSION PRESS (100),RATIO(100),GAS V(100),V S A(100),V S D(100),
     1V PER A(100),V Per D(100),DATA(15),ID(9)
      COMMON NO PTS, KWIT,I,ANSWR,ID
    1 READ INPUT TAPE 1,199,NO PTS,(ID(J),J=1,9)
      IF(ID(1)-8283767700) 2,999,2
    2 K FULL = NO PTS/15
      KARDS = K FULL
      K REM = NO PTS - 15*K FULL
      IF(K REM) 4,4,3
    3 KARDS = KARDS + 1
    4 NO CDS = 5*KARDS
      DO 11 K = 1,NO CDS
      READ INPUT TAPE 1,198, KIND, INDIC, (DATA(J),J = 1,151)
      J2 = 15*INDIC
      J1 = J2 - 14
      I = 1
      DO 10 J = J1,J2
      GO TO (5,6,7,8,9),KIND
    5 PRESS(J) = DATA(I)
      GO TO 10
    6 RATIO(J) = DATA(I)
      GO TO 10
    7 GAS V(J) = DATA(I)/100.0
      GO TO 10
    8 V S A(J) = DATA(I)/100.0
      GO TO 10
    9 V S D(J) = DATA(I)/100.0
   10 I = I + 1
   11 CONTINUE
      WRITE OUTPUT TAPE 2,299,(ID(J),J = 1,9)
      DO 12 K = 1,NO PTS
      STP V = 0.00281*PRESS(K)*RATIO(K)
      TOT V = GAS V(K) + STP V
      V PER A (K) = TOT V/V S A(K)
      V PER D(K) = TOT V/V S D(K)
   12 WRITE OUTPUT TAPE 2,298, PRESS(K),RATIO(K),GAS V(K),V S A(K),
     1V S D(K),STP V,TOT V,V PER A(K),V PER D(K)
      CALL MEAN  (1,V PER A(1))
      CALL MEAN  (2,V PER D(1))
      GO TO 1
  999 STOP
  299 FORMAT (1H1/1H0/15X,22HVOLUME CORRECTION FOR ,9A5/1H0/15X,56HCO2
     1   MEASURED   V. S.     V. S.     CO2     CORRECTED,2(15H   GAS VO
     2L./LB.)/2X,100H PRESSURE    RATIO    GAS VOL.    ADDED    DESTROYED A
     3BSORBED  GAS VOL.    V. S. ADDED  V. S. DESTROYED /1H0)
  298 FORMAT (1H ,F10.1,6F10.5,2F15.1)
  199 FORMAT (I5,30X,9A5)
  198 FORMAT (I1,I4,15F5.0)
      END
```

```
      SUBROUTINE MEAN  (L,ARRAY)
      COMMON NO PTS,KWIT,I,ANSWR,ID
      DIMENSION ARRAY(100),ANSWR (100),ID(9)
      L = L
      DO 3 I = 4,7
      PTS = I
      J1 = 0
      J2 = 0
      KWIT = NO PTS - I + 1
      DO 2 II = 1,KWIT
      SUM = 0
      J2 = J1 + I
      J1 = J1 + 1
      DO 1 J = J1,J2
    1 SUM = SUM + ARRAY(J)
    2 ANSWR(II) = SUM/PTS
      CALL PRINT (L)
    3 CONTINUE
      RETURN
      END


      SUBROUTINE PRINT (L)
      DIMENSION ANSWR(100),ID(9)
      COMMON NO PTS,KWIT,I,ANSWR,ID
      GO TO (1,2),L
    1 WRITE OUTPUT TAPE 2,299,I,(ID(JJ),JJ=1,9)
      GO TO 3
    2 WRITE OUTPUT TAPE 2,298,I,(ID(JJ),JJ=1,9)
    3 K = KWIT/4
      IF(KWIT - 4*K) 5,5,4
    4 K = K + 1
    5 DO 7 N = 1,K
      KK = N + 3 K
      IF(KWIT - KK) 6,7,7
    6 KK = KWIT
    7 WRITE OUTPUT TAPE 2,297,(ANSWR(J),J = N,KK,K)
      RETURN
  299 FORMAT (1HO/1HO/1HO,5X,I5,21H DAY MOVING MEAN FOR ,9A5,31H Gas VOL
      LUME PER LB. V. S. ADDED/1HO/)
  298 FORMAT (1HO/1HO/1HO,5X,I5,21H DAY MOVING MEAN FOR ,9A5,35H GAS VOL
      LUME PER LB. V. S. DESTROYED/1HO/)
  297 FORMAT (1H ,4(15X,F5.1))
      END
```