EPA-600/2-77-089
May 1977

Environmental Protection Technology Series

A COMPLETE DISPOSAL—RECYCLE SCHEME FOR AGRICULTURAL SOLID WASTES

Robert S. Kerr Environmental Research Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Ada, Oklahoma 74820

RESEARCH REPORTING SERIES

Research reports of the Office of Research and Development, U.S. Environmental Protection Agency, have been grouped into nine series. These nine broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The nine series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies
6. Scientific and Technical Assessment Reports (STAR)
7. Interagency Energy-Environment Research and Development
8. "Special" Reports
9. Miscellaneous Reports

This report has been assigned to the ENVIRONMENTAL PROTECTION TECHNOLOGY series. This series describes research performed to develop and demonstrate instrumentation, equipment, and methodology to repair or prevent environmental degradation from point and non-point sources of pollution. This work provides the new or improved technology required for the control and treatment of pollution sources to meet environmental quality standards.

This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.

EPA-600/2-77-089
May 1977

A COMPLETE DISPOSAL-RECYCLE SCHEME FOR AGRICULTURAL SOLID WASTES

by

Michael R. Busby
Greg Tragitt
Roland Norman
Kenneth Hillsman
Tennessee State University
Nashville, Tennessee 37203

Grant No. R802739

Project Officer

S. C. Yin
Source Management Branch
Robert S. Kerr Environmental Research Laboratory
Ada, Oklahoma 74820

ROBERT S. KERR ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
ADA, OKLAHOMA 74820

DISCLAIMER

This report has been reviewed by the Robert S. Kerr Environmental Research Laboratory, U.S. Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the U.S. Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

ABSTRACT

With the advent of the 70's, there has been an increasing national concern for the growing energy shortage as well as the problem of organic waste disposal. These two problems, while at first glance appear unrelated, are dealt with simultaneously by an anaerobic digestion process. Such a process produces not only a useful fuel, methane, but also is a potential source of a stabilized fertilizer and a nutritive supplement to animal diets. This biological process has been used for decades, but the economic feasibility of incorporating such a process on a typical small farm has not been clearly established.

This investigation applied the anaerobic process to the production of methane gas and a stabilized sludge from cow manure and farm clippings in laboratory pilot plants as well as a full-scale (2,000 gal.) digester system. The quantity and quality of gas produced, the biochemical and chemical oxygen demands, and the nutritional value of the digested sludge for both the laboratory and full-scale plants were evaluated.

CONTENTS

FIGURES

TABLES

SECTION 1

CONCLUSIONS

An anaerobic waste treatment process was successfully operated in three laboratory scale plants. A full-scale (two 2,000 gal. digester units) bio-gas plant was designed and constructed to treat the agricultural wastes typical of a small farm. Utilizing the experience gained in the laboratory, the full-scale plant was operated over a six-week period.

Both the full-scale and laboratory systems functioned well, producing methane and a stabilized sludge from feedlot cow manure by biological degradation. The methane content of the dry gas produced was typically 62 percent and the full-scale plant gas volume generation rate varied from 3.5 to 5 ft. ^{3}/hr. (sfc) after a fourteen-day retention time. In all samples, the COD and BOD were reduced in the plant effluent as compared to the influent.

Both the pilot and full-scale plants were fed on a daily basis by wasting digested material and adding an equal volume of fresh mixture. In the full-scale plant the pH was controlled by the addition of lime, and mixture temperature (104-112°F) was maintained by an internal cooper coil heat exchanger.

A nutritive study of the digested sludge was completed utilizing weanling rats. It was found that feed conversion and digestibility of diets decreased as the level of sludge increased. However, the experiment revealed that anaerobically processed organic wastes are consumed readily and when incorporated into a balanced diet will support animal growth.

SECTION 2

RECOMMENDATIONS

A full-scale bio-gas plant has been operated; however, several operational difficulties were encountered. Specifically, mixing and pumping of the feeding mixture and foaming of the supernatant in the reactor caused the gas outlets to become clogged, presenting obstacles to plant operation. Only a small sample of data has been taken from the large scale system. It became apparent that environmental changes significantly affect the plant outputs. Therefore, before any realistic evaluation of the economic feasibility of small farm operation can be made, data must be collected over a long time period. This period should include seasonal changes as well as day to day fluctuations in weather. The Cooperative States Research Service (CSRS) has funded such an investigation for a two-year period beginning in June, 1976. Data collected from this study should establish a comprehensive basis for the economic evaluation of the anaerobic process.

SECTION 3

INTRODUCTION

There are many challenges which confront the United States in the mid 70's. Among those are two problems which at first appear unrelated but upon closer inspection are found to be complementary. The growing concern for a feasible means of organic waste disposal and an increasing worldwide energy shortage may be dealt with simultaneously by the anaerobic digestion process. The development of an economical process to convert organic wastes into useable methane gas would provide at least a partial solution to both these problems. In this report only organic agricultural wastes will be considered.

AGRICULTURAL WASTE DISPOSAL

Farm animals were previously maintained at low areal densities, however, now up to 20,000 beef cattle, 6,000 dairy cows, and 2,000,000 chickens are confined in single locations (1). A high percentage of beef cattle spend one-third of their life in feedlots for fattening, and nearly all dairy cattle, swine, and poultry are restricted to a concentrated area during their entire life. With at least one-half or more of the 1.2 billion tons of excreta being defecated in feedlots and animal enclosures, an environmental problem has arisen. An acceptable disposal scheme has become a necessity in order to avoid surface and groundwater pollution, oxygen depletion, eutrophication of surface waters, undesirable odors, and fly breeding. The problems of disposal are aggravated by the marginal nature of the economics of the agricultural industry in general and its consequent inability to treat its wastes satisfactorily.

Ecologically, land spreading is the best method of disposal to recycle nutrients. Unfortunately, sufficient land is unavailable for this method of disposal. The quantity of waste that can safely be applied to the land is dependent upon soil vegetative cover, slope, climate, and distance to receiving waters. While manure is being produced continuously, it can only be spread at certain times due to crop covering, weather, etc.

Biological processes provide a means for treating large volumes of waste materials (2). Their suitability is evidenced by the fact that their energy requirements are minimal, the organic mass produced is negligible, methane gas is a useful by-product, and a humus-like slurry for land reclamation is produced after sludge stabilization (3).

ENERGY SHORTAGE

In 1960 the total energy demand of the United States was 4.5 X 10^{16} BTU, 6 X 10^{16} BTU in 1970, and is expected to exceed 9 X 10^{16} BTU by 1980. The share of natural gas (methane) in this energy market was approximately 31 percent in 1968 (4). This total energy demand, and in particular that on natural gas, cannot continue without exhausting the available fuels. For example, gains in production of natural gas from 1947 to 1968 averaged more than six percent per year with production growing from 5.6 to 19.3 trillion cubic feet. During the same period reserves increased on an average of 2.5 percent annually, i.e., 165 to 282 trillion cubic feet. However, the reserves-to-product ratio has declined from 29.5 to 14.6 years. Even with optimistic views on reserves and future production, demand for gas will remain greater than additions to reserves. In order to at least ease the inevitable gas shortage, Bahn has estimated that anaerobic gasification of urban and agricultural wastes would yield 30 trillion cubic feet of methane annually (5). This quantity of gas would meet all of the present U. S. needs while significantly alleviating the problem of animal waste disposal.

PURPOSE OF THE STUDY

Since the anaerobic process could be utilized effectively in the solution of two of this country's problems, the present study was undertaken in order to investigate the design and operating parameters for an agricultural waste disposal scheme. Anaerobic digesters have been used for years to treat domestic wastes but have not been used widely in the disposal of agricultural wastes. Therefore the primary objectives of this investigation were to operate pilot digester reactors in a controlled environment, to design and operate a large scale plant, and monitor gas production and investigate the feasibility of using the stabilized sludge as a food supplement for animals. The overall objective of the project was to obtain data which could be utilized by a small farmer for the construction and operation of a plant capable of alleviating his waste disposal problems as well as providing significant energy for farm operation.

SECTION 4

THEORECTICAL CONSIDERATIONS

FUNDAMENTALS OF ANAEROBIC DIGESTION

The fundamentals of the anaerobic process have been excellently presented by Pfeffer (6), and his discussion will be given in this report for completeness.

Anaerobic treatment of complex organic materials is considered to be basically a two-stage process, as indicated in Figure 1. In the first stage, a group of facultative and anaerobic bacteria, known as the "acid-formers," act upon the complex organics and change the form of complex fats, proteins and carbohydrates to simple soluble organic materials. The end products of this first stage conversion are primarily short chain organic acids, also known as volatile acids, and small amounts of bacterial cells. This stage accomplishes no stabilization of the waste material but it is an essential prerequisite for the second stage in which the actual stabilization of the waste matter occurs. It places the organic matter in a form suitable for the second stage of treatment.

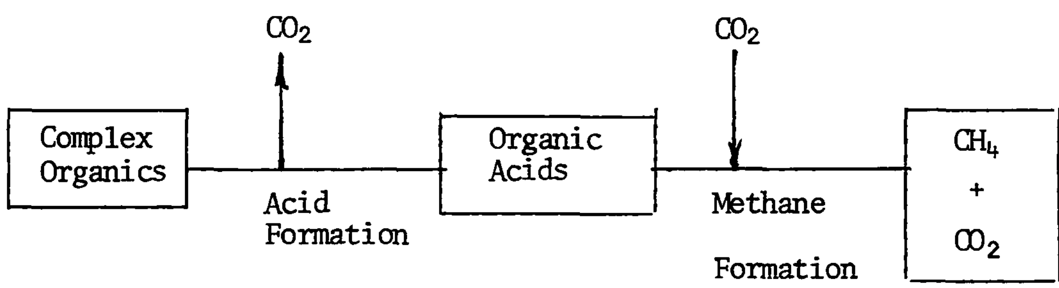

Figure 1. Anaerobic stabilization of complex organics.

In the second stage, the short chain organic acids are acted upon by a strictly anaerobic group of bacteria, termed the "methane formers," and are coverted to gaseous end products, methane and carbon dioxide. The methane formed in this second stage, being insoluble in water, escapes from the system. It can be collected for use as a fuel. The carbon dioxide involved partially escapes in the gaseous form and partially goes into solution. It is in this second stage that stabilization occurs through the removal of oxygen-demanding material in the form of methane gas. Cell production is also minimal compared to aerobic processes. This is a direct result of the high energy content of the products, in particular methane (7). This is an advantage as the amount of solids requiring ultimate disposal is minimized by eliminating any significant microbial protoplasm production in the process of stabilizing the organic material.

There are several different groups of methane formers with each group characterized by its ability to ferment only a specific number of compounds. Therefore, several different bacteria are required for stabilization of the organic material (8). The most important of these methane formers, those utilizing acetic and propionic acids for substrates, grow quite slowly and for most organic compounds are rate limiting at the lower retention times. For sewage sludge digestion, Pfeffer (9) found that in systems operating with solids retention times of approximately ten to fifteen days, the rate limiting step then becomes the hydrolysis of organic solids.

Tracer studies have indicated the major sources of methane (8). One source is the direct cleavage of acetic acid into methane and carbon dioxide. Most of the remaining methane is formed from the reduction of carbon dioxide. Carbon dioxide, functioning as an electron accepter, is reduced by hydrogen atoms enzymatically removed from organic compounds. The availability of carbon dioxide in such a step is never a rate limiting factor as there is always a large excess of it from the bicarbonate buffer system that is present in anaerobic systems (8).

Organic destruction in anaerobic treatment is directly related to methane production and vice versa. Buswell and Mueller (10) developed Equation 1 to predict the quantity of methane from a knowledge of the chemical composition of the waste:

$$C_n H_a O_b + \left(n - \frac{a}{4} - \frac{b}{2}\right) H_2O \longrightarrow \left(\frac{n}{2} - \frac{a}{8} + \frac{b}{4}\right) CO_2 + \left(\frac{n}{2} + \frac{a}{8} - \frac{b}{4}\right) CH_4 \qquad (1)$$

McCarty (8) showed that the theoretical methane production from the complete stabilization of one pound of BOD_L or COD was 5.62 cubic feet at standard temperature and pressure. From this he forwarded Equation 2 for estimating methane production from waste strength:

$$C = 5.62 \ (eF - 1.42A) \qquad (2)$$

where: C = cubic feet of CH_4 produced per day (STP)
e = efficiency of waste utilization
F = pounds of BOD_L added per day
A = pounds volatile biological solids produced per day.

The efficiency of waste utilization, e, should not be confused with the stabilization efficiency. It is a factor designating the efficiency of the conversion of the organic waste to both the gaseous form and to biological solids. Whereas the former form represents waste stabilization, the latter conversion does not. The pounds of volatile biological solids produced per day, A, can be estimated from Equation 3 (11):

$$A = \frac{aF}{1 + b(SRT)} \qquad (3)$$

where: a = growth constant
b = endogenous respiration rate
SRT = solids retention time in days.

Values for a and b are shown in Table 1 for various organic compounds (12).

TABLE 1. GROWTH CONSTANTS AND ENDOGENOUS RESPIRATION RATES

Substrate	Growth Constant, a	Endogenous Respiration Rate, b
Fatty Acid	0.054	0.038
Carbohydrate	0.240	0.033
Protein	0.076	0.014

The percentage of added BOD which is stabilized, S, is given by Equation 4 (11):

$$S = \frac{100C}{5.62\ F} = \frac{100(eF - 142\ A)}{F} \qquad (4)$$

The efficiency of waste stabilization is related to the solids retention time. As the solids retention time decreases, the relative proportion of active cells washed out of the system increases. If the solids retention time falls below a certain limit, the micro-organisms responsible for anaerobic digestion will be washed out faster than they can reproduce themselves and the result is failure of the process. The minimum retention time is dependent on the temperature and the types of substrate being utilized. Though it is possible to operate near the minimum retention times, efficiencies are low and process dependability is poor. Solids retention times of at least two and a half times the minimum are recommended (11).

While there are many different methane forming bacteria, there are also many different acid-forming bacteria. For attaining good efficiencies of waste stabilization, a proper balance among all these different organisms is required. The establishment and maintenance of this balance dictates operation under optimum environmental conditions. These are discussed in brief in the next paragraphs.

Temperature is a very important operational parameter in anaerobic fermentation process. As temperatures increase, rates of reaction proceed much faster and this results in more efficient operation, and lower retention time requirements (13). Two optimum temperature levels have been established. In the mesophilic level the temperatures range from 30°C to 37.5°C and in the thermophilic level, they range from 49°C to 51°C. Although the rates of reaction in the thermophilic level are much faster than those in the mesophilic level, the economics of most sewage sludge digestion systems have dictated operation in the mesophilic level or lower (13). This stems from the fact that the methane requirements to maintain thermophilic temperatures in most digesters are excessive and uneconomical. This, in turn, is a result of the inability to thicken the feed sludges sufficiently such that the organic loading and the resulting methane production per unit digester volume are sufficiently high to make such an operation economically attractive.

Another environmental requirement for anaerobic treatment is the maintenance of anaerobic conditions in the digester. The methane formers are strict anaerobes and even small amounts of oxygen can be quite detrimental to them. This necessitates, in most cases, a closed digestion tank which is also convenient because collection of the methane produced is facilitated.

The third environmental requirement for optimum operation is that for a proper pH. McCarty (13) reports that anaerobic treatment can proceed quite well with a pH varying from about 6.6 to 7.6 with an optimum range of about 7.0 to 7.2. Beyond these limits anaerobic digestion proceeds with decreasing efficiency until at a pH of 6.2 and lower, the acid conditions become quite toxic to the methane formers and waste stabilization comes to a virtual halt.

Control of pH should be exercised when the pH appears likely to drop below 6.6. This is done by the addition of an alkali. In sewage sludge digestion the use of lime for such control has been the most widespread, but because of its many advantages over lime, sodium bicarbonate has lately been receiving increasing attention as a substitute for lime for pH control.

The bacteria responsible for waste conversion and stabilization in the anaerobic process require nitrogen, phosphorus and other materials in trace quantities for optimum growth. Therefore, another important environmental condition is the presence of the required nutrients in adequate quantitites. Municipal waste sludge usually contains all the required nutrients in adequate quantities but other substrates, industrial and solid wastes in particular, might not. If the nutrients are not present in the required quantities, they must be either added or supplemented.

McCarty (8) calculated the nitrogen and phosphorus requirements based on an average chemical formulation of biological cells of $C_5H_9O_3N$. This yielded a nitrogen requirement of about 11 percent of the cell volatile solids weight and a requirement for phosphorus equal to about one-fifth of this figure. Although these requirements should theoretically be based on the fraction of waste removed during treatment rather than on the waste added, it is good practice to base them on waste additions (8). Other elements having stimulatory effects at low concentration include, but are not limited to sodium, potassium, calcium, magnesium, and iron (14). All of these preceding elements exhibit inhibitory effects at higher concentrations.

The fifth and final environmental requirement for successful anaerobic treatment is that the waste be free from toxic material. This is particularly true for concentrated organic wastes which, though normally are more susceptible to anaerobic treatment, are also more likely to have high or inhibitory concentrations of various materials ranging from inorganic salts to toxic organic compounds.

Some alkali and alkaline earth-metal salts above certain concentrations exhibit degrees of inhibition and toxicity. The threshold levels vary, depending on whether these metals act singly or in combination. Certain combinations have synergistic effects, whereas others display antagonistic effects.

Ammonia, particularly when in the ammonium form, is inhibitory when present in high enough concentrations. At concentrations between 1,500 and 3,000 mg/l and a pH greater than 7.4, the ammonia concentration can become inhibitory. At concentrations above 3,000 mg/l, the ammonium ion itself becomes quite toxic regardless of pH (14).

Other common forms of toxicity include those of sulfides, heavy metals and toxic organic materials. Concentrations of soluble sulfide varying from 50 to 100 mg/l, can be tolerated in anaerobic treatment with little or no acclimation, whereas concentrations up to 200 mg/l can be tolerated with some acclimation (14). Low soluble concentrations of copper, zinc, and nickel salts are associated with most of the problems of heavy metal toxicity in anaerobic treatment. Also, there are many organic materials that exhibit inhibitory effects. These range from organic solvents to many common materials such as alcohols and long chain fatty acids in high concentrations (14).

It is well to know that microorganisms usually have the ability to acclimate to some extent to inhibitory concentrations of most materials if the process is acclimated to the inhibitory substance. Also, it should be recognized that only materials in solution can be toxic to biological life (14). Control of toxicity or inhibition can, in general, be achieved by one or more of three ways: (1) the removal from the waste stream or inactivation of toxic materials by such means as chemical precipitation, (2) the dilution of the waste stream below the "toxic threshold" of the toxicity causing material, and (3) the addition of an antagonistic material.

SECTION 5

EXPERIMENTAL PROCEDURE

LABORATORY PILOT DIGESTER REACTORS

In the laboratory three anaerobic digestion pilot plants were fabricated from plexiglass, as shown in Figure 2. Each unit had a volume of approximately 17 liters (15.24 cm dia X 91.44 cm). The top and bottom of each unit were threaded, and plexiglass plugs were fitted and sealed with rubber "O"-rings. A funnel - stopcock inlet arrangement was used for feeding the unit and a drain for wasting was located at the bottom of the pilot digester. At the unit top, a glass tube was located to provide a passage via a Tygon tube to the gasometer. The entire unit assembly was mounted on a stand to facilitate feeding and wasting.

The gas generated from the pilot unit was collected in a gasometer shown in Figure 3. The apparatus consisted of an outer glass cylinder filled with a sulfuric acid solution. An inverted inner plexiglass cylinder constituted the gas collection system as shown. The cylinder would rise as the gas was collected. The side of the gasometer was calibrated in cubic feet.

All units were housed in a room where temperature could be controlled. The units were in a continuous, steady-state reaction, with a fourteen-day detention time. Each plant contained ten liters of digestible material. The units were fed every twenty-four hours with a 100 ml of fresh material being added to each plant after 100 ml of digested material was removed. The digestible material was made by diluting varying ratios of grass clippings and fresh cow manure to 6 percent total solids. In Unit I the solids were 100 percent cow manure; Unit II had 70 percent manure and 30 percent grass clippings; and Unit III contained 50 percent - 50 percent mixture. The pH of the units was maintained between 6.7 and 7.0 for all experiments with NaOH being added to raise the pH to the desirable level.

FULL-SCALE DIGESTER PLANT

A full-scale anaerobic digester experimental plant was designed and constructed on the Swine Area of Tennessee State University (Figures 4a, 4b, and 4c). A schematic of the plant lay-out is shown in Figure 5. Two 7,570 liter (1.62 m dia X 3.66 m) digester reactors were constructed from cold-rolled steel and mounted on concrete piers (Figure 6). The tanks were coated inside and out with an epoxy paint which is resistant to the corrosive properties of the waste slurry. The influent and effluent pipes are shown in Figure 6. The tank's contents were circulated by pumping the

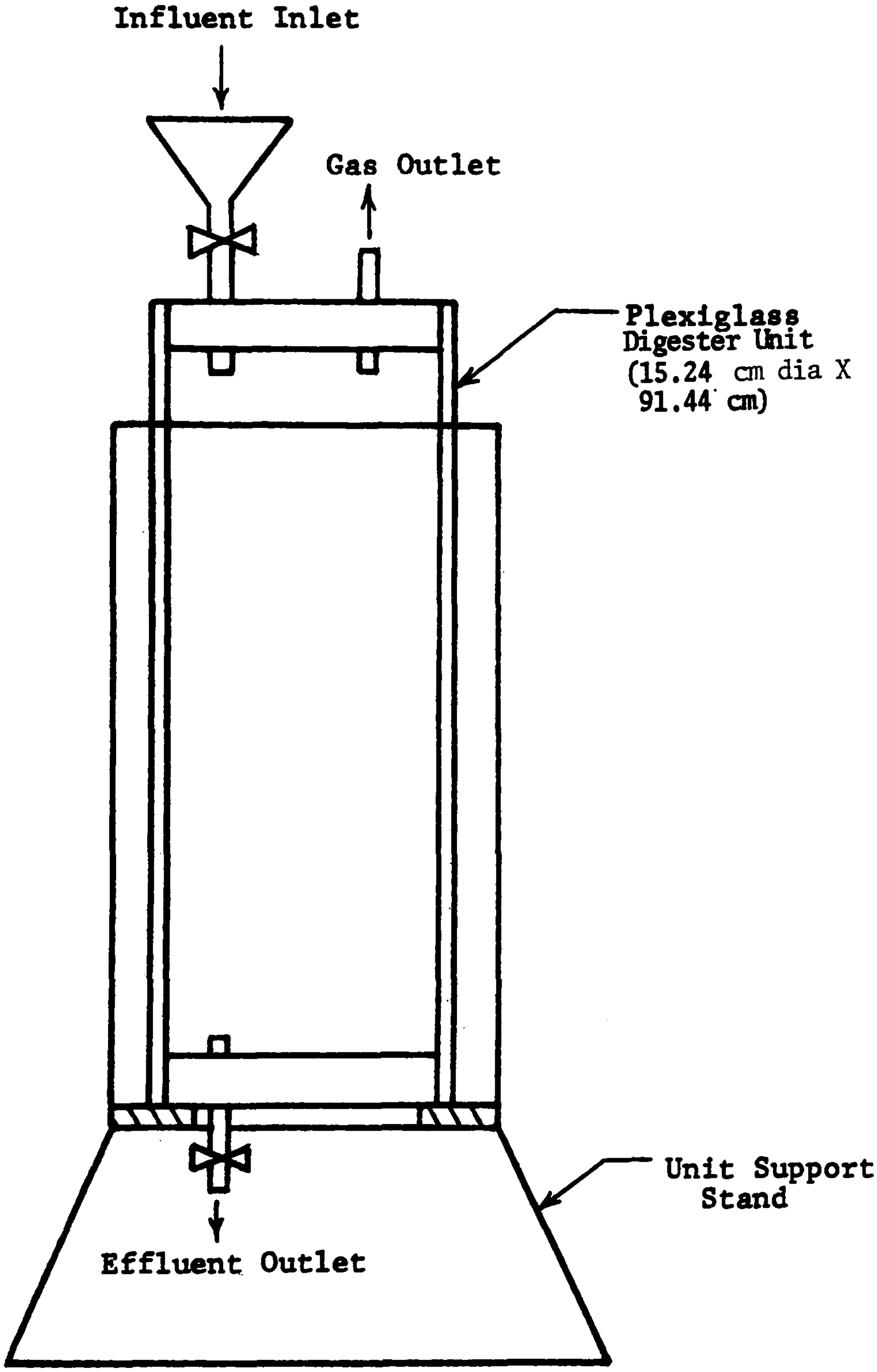

Figure 2. Schematic of pilot plant digester.

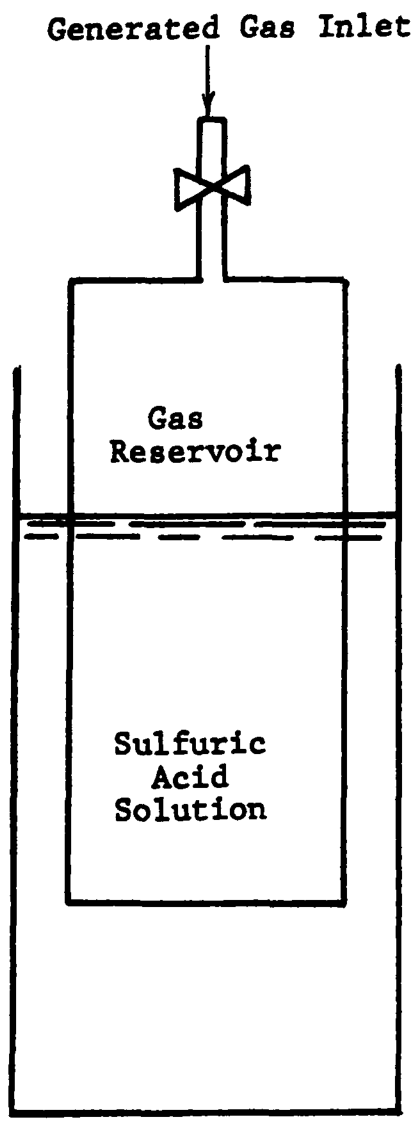

Figure 3. Pilot plant gasometer.

mixture through a manifold in the lower influent pipe, and extracting it from the center influent pipe (Figure 7). The digestible material (feed-lot cow manure and water) was mixed in a modified 189 liter stainless-steel coffee urn and pumped into the digester through a series of valves as shown in Figure 8. Two gas ports and a manhole fitted with a rubber gasket are located on the top of each tank (Figure 6). A flexible rubber hose was connected to a gate valve at the effluent pipe to facilitate in digested sludge removal.

Platinum resistance thermometers were located in the sides of each (Figure 6) to monitor the sludge temperature. In order to control the mixture temperature, a heat exchanger, consisting of a copper tube (1.3 cm diam) coil, an external water heater and circulation pump, was fabricated and positioned as shown in Figure 9.

Monitoring equipment was located in the utility shed. Two 63125 Precision Scientific wet test gas meters, one for each tank, were connected to the digesters by 0.64 cm O. D. copper tubing (Figure 10). The tank pressure was monitored continuously by a strip-chart pressure recorder located in the shed. A digital voltmeter was used to measure the output of the platinum resistance thermometers. A schematic of the gas handling connections is also shown in Figure 10.

The entire plant site was located on a 15.2 m by 12.2 m reinforced concrete slab. A cyclone fence and gate arrangement insured plant security.

The typical procedure for charging the plant was as follows:

(1). Thirty gallons of water were mixed with 80 pounds of hammer-milled dried cow manure in the coffee urn. The liquid was throughly mixed by means of a Lightin Mixturer (1/3 HP; ND-1 Model).

(2). The urn contents were then pumped into the digester. The procedure was continued until the tank was approximately 70 percent full, i.e., 5300 liters.

(3). The proper valves were opened and closed and the tank contents were continuously circulated.

(4). After a minimum of twenty-four hours of circulation, a sludge sample was taken and the percent solids and pH of the mixture were determined.

(5). Calculations were made to determine the required dilution or solids addition to provide a 8 to 12 percent solids solution. Also, the required amount of lime was calculated to assure a solution pH of 6.5 to 7.6. The necessary additions were then made.

(6). A fourteen-day retention time was required before ideal operating conditions could be achieved. After this period of time, equal amounts of digested sludge and new digestible material were withdrawn and added (usually 303 liters) on a daily basis.

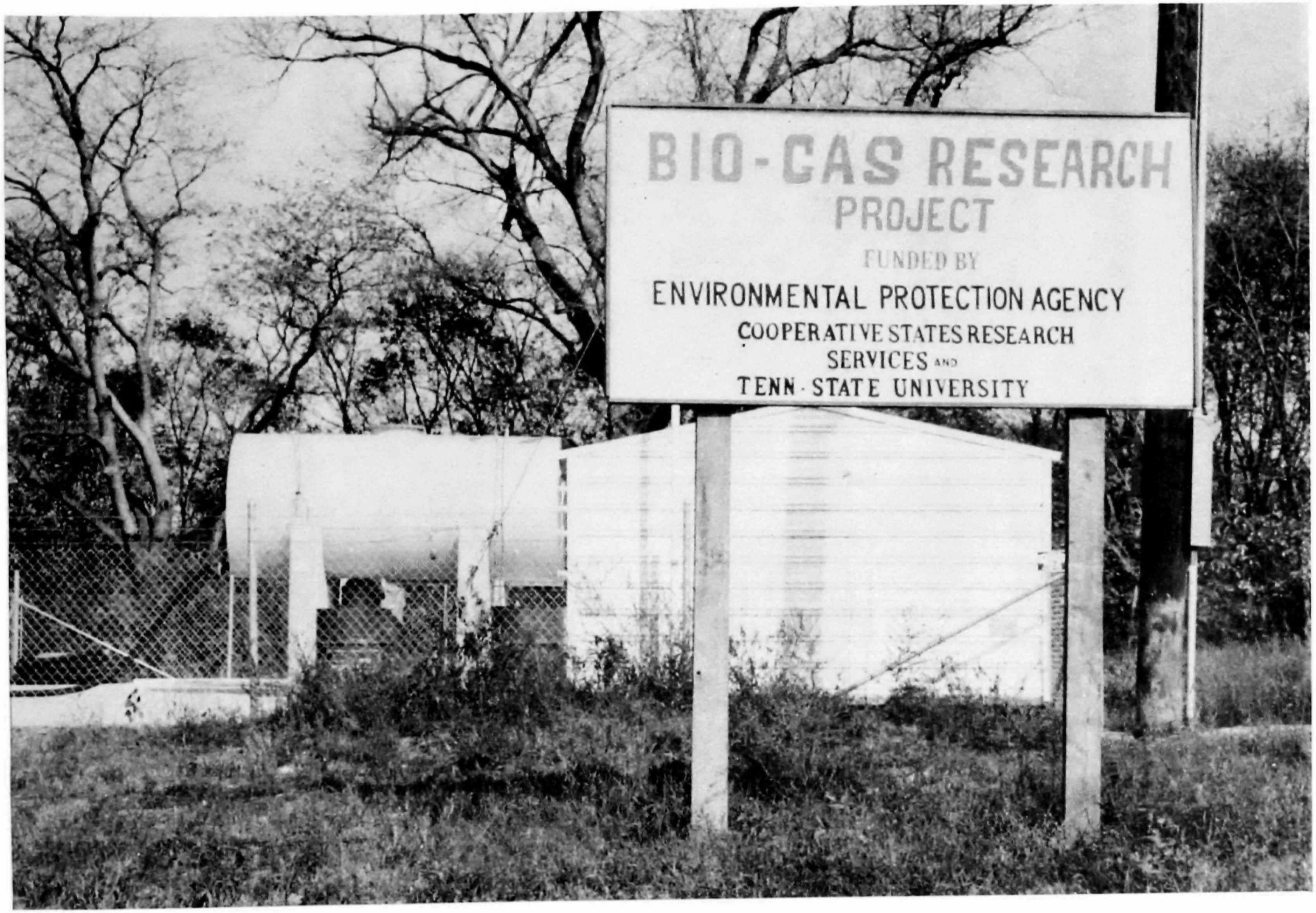

Figure 4a. EPA-CSRS-TSU anaerobic digester plant.

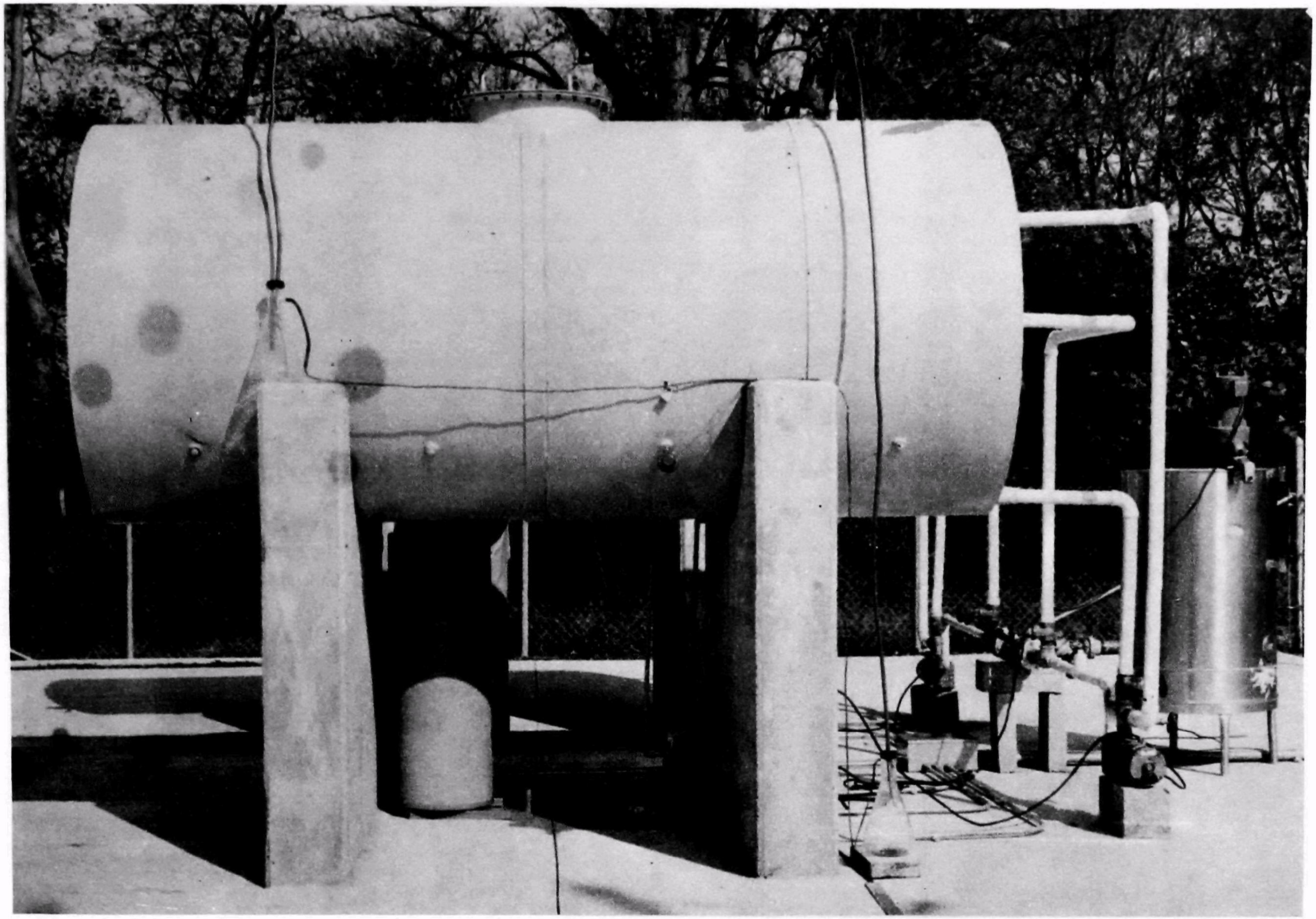

Figure 4b. EPA-CSRS-TSU anaerobic digester plant.

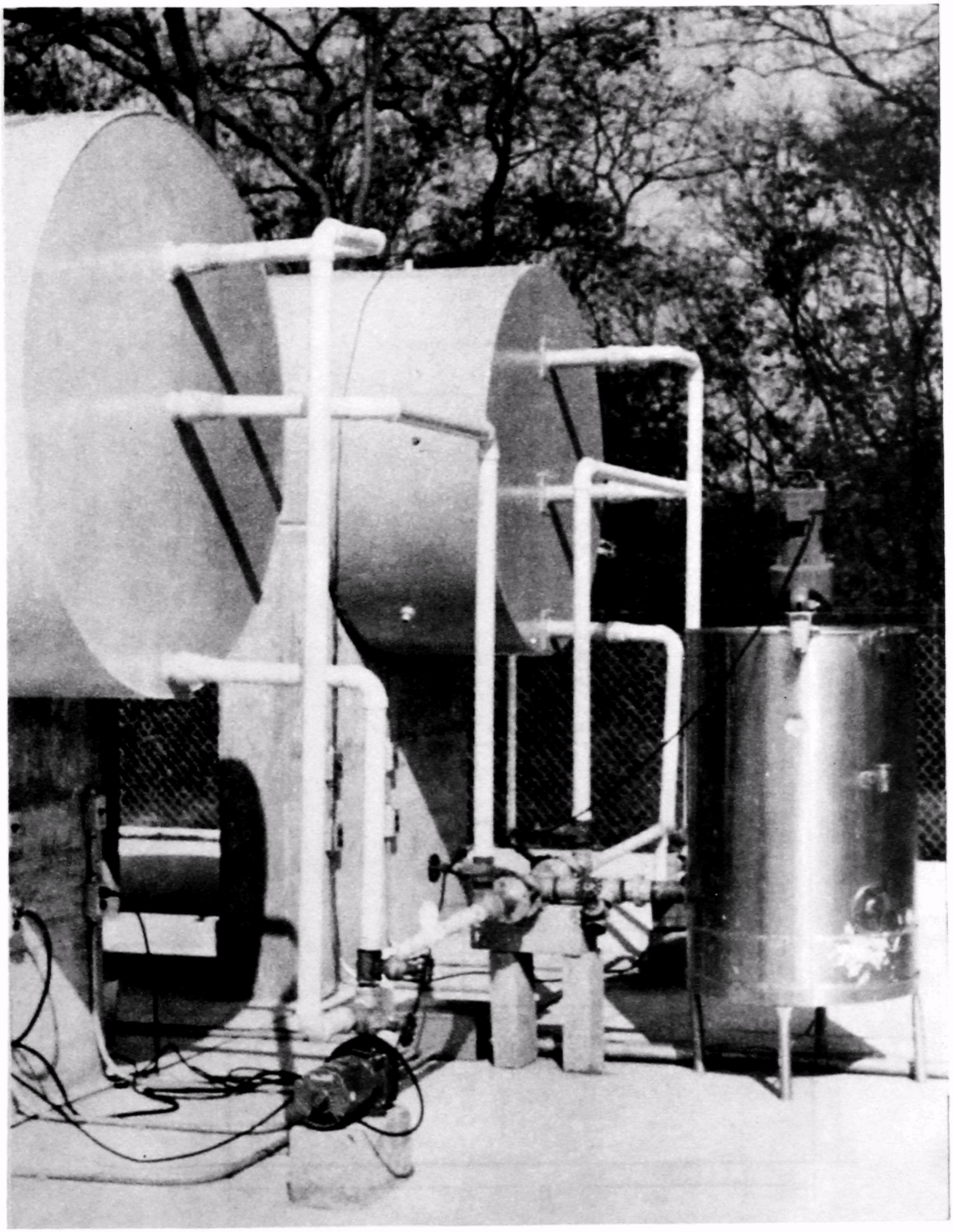

Figure 4c. EPA-CSRS-TSU anaerobic digester plant.

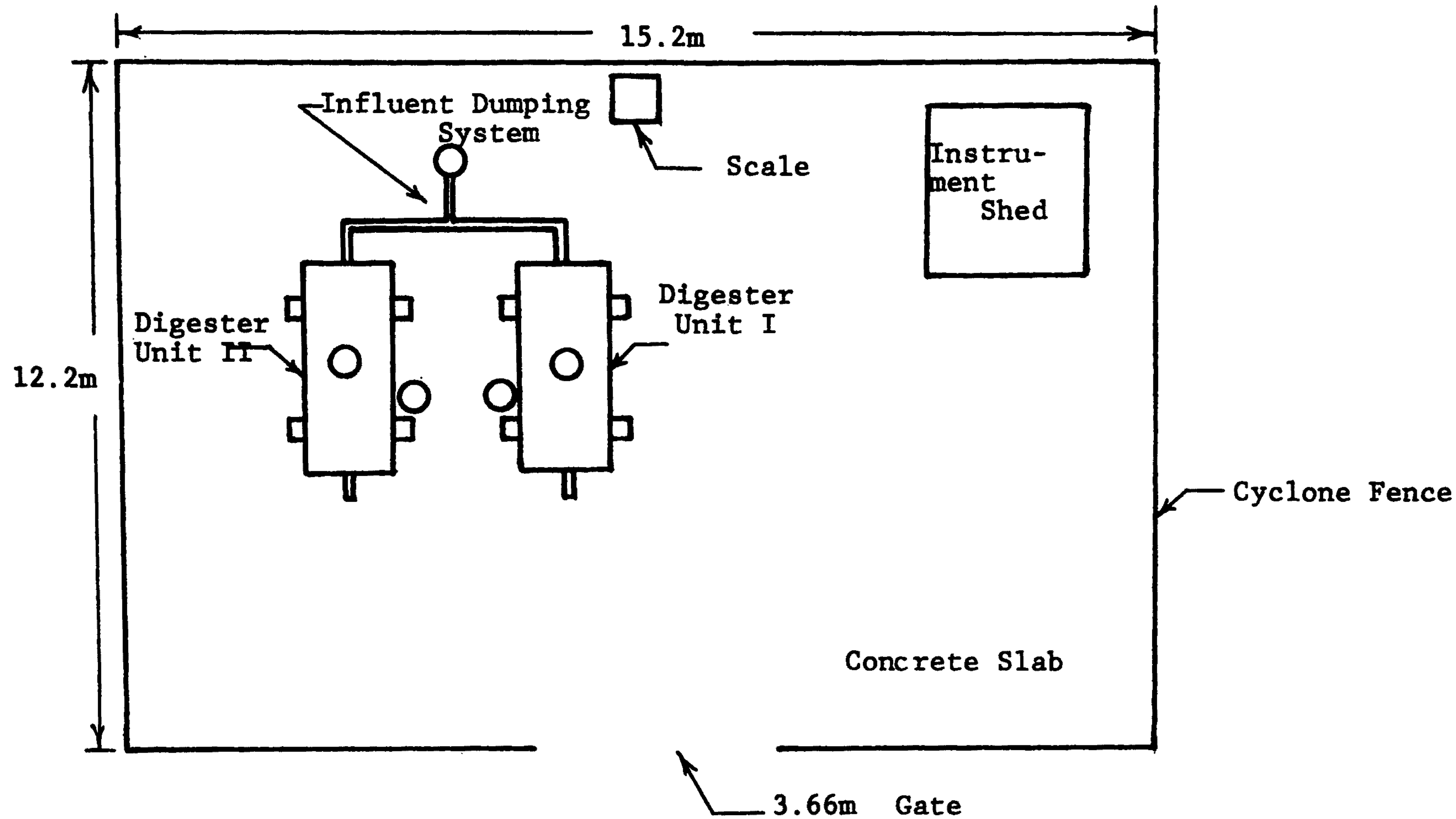

Figure 5. Schematic of full-scale digester plant.

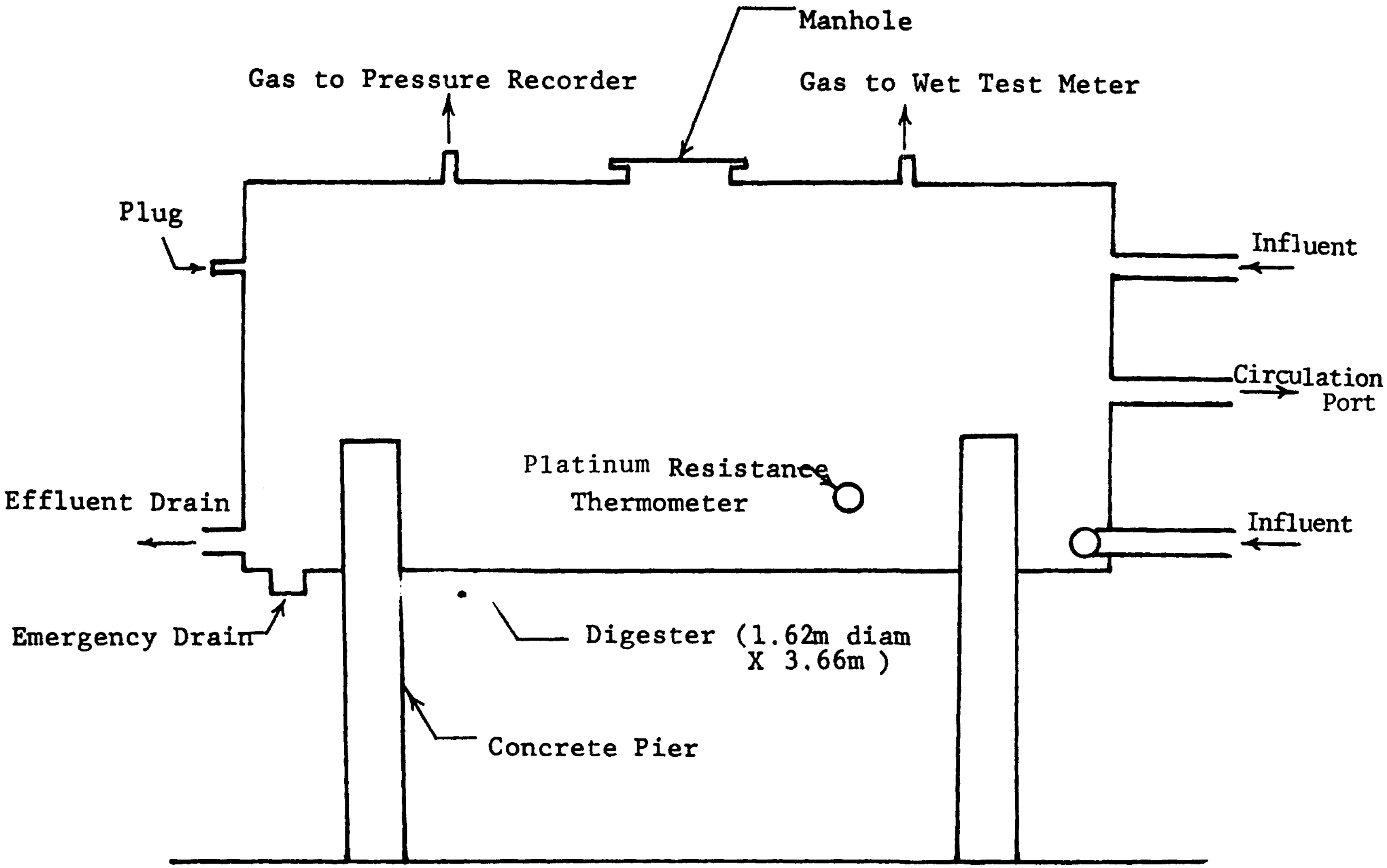

Figure 6. Schematic of digester tanks.

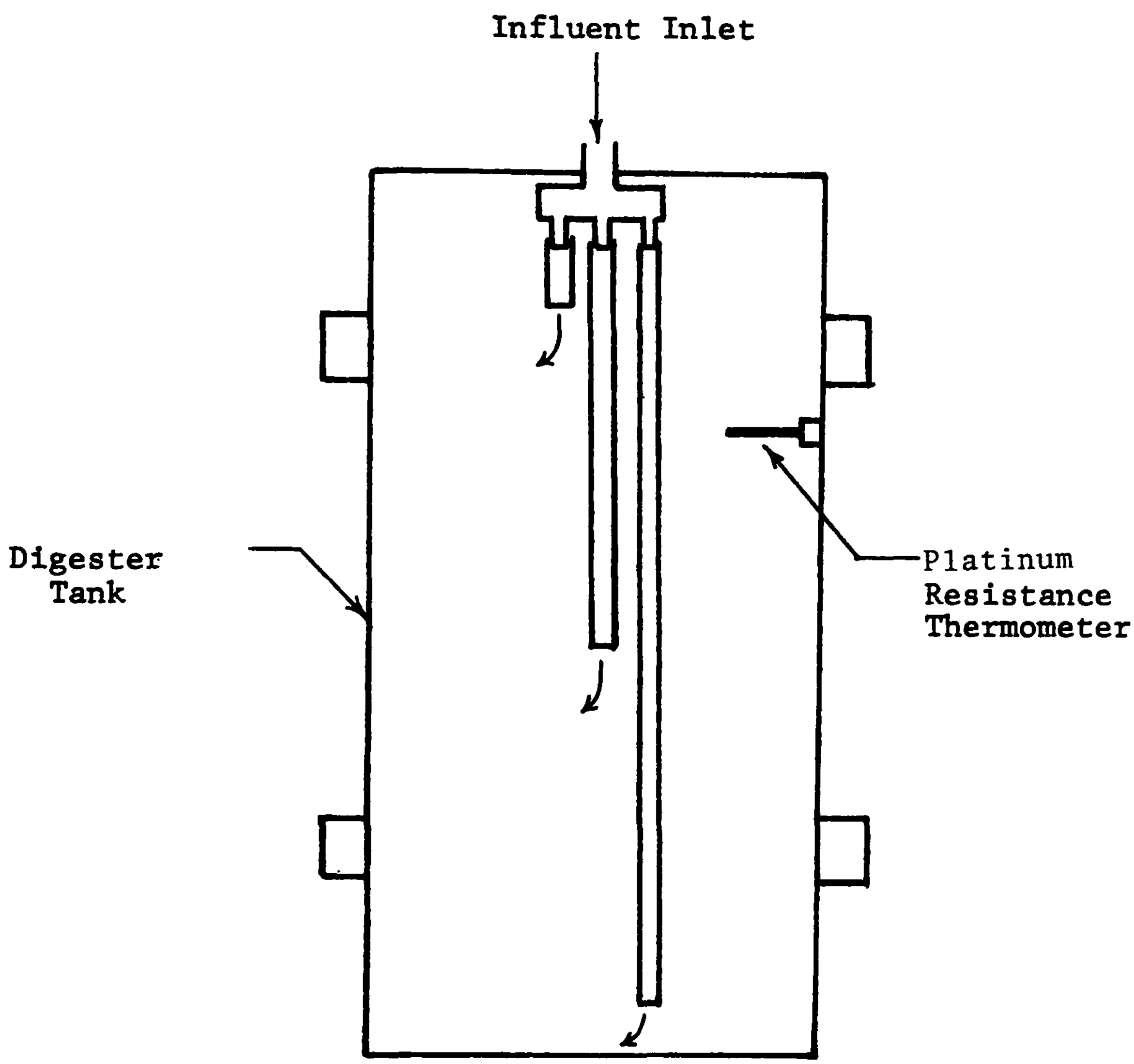

Figure 7. Schematic of Internal Tank Circulation System.

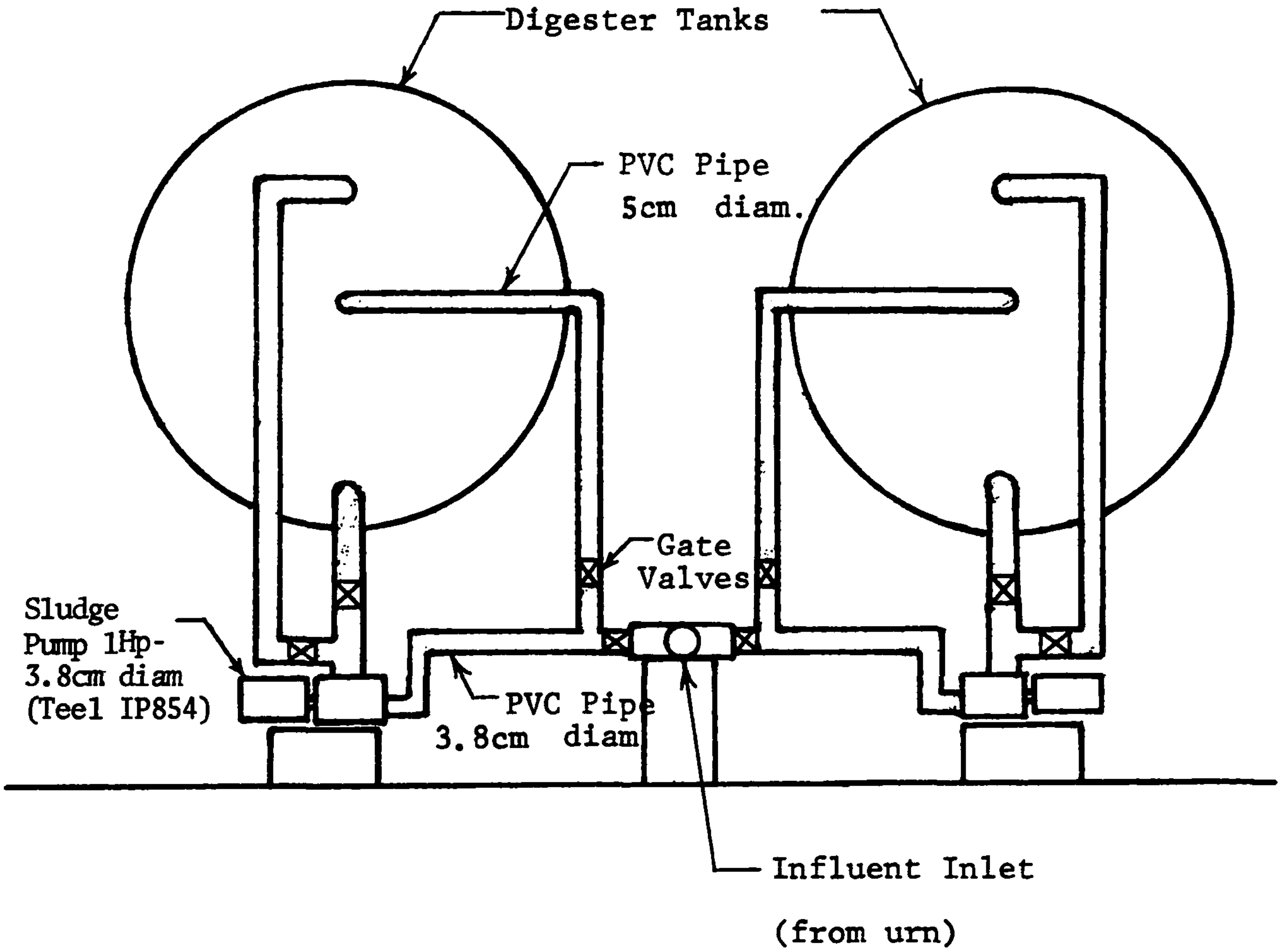

Figure 8. Schematic of influent pumping arrangement.

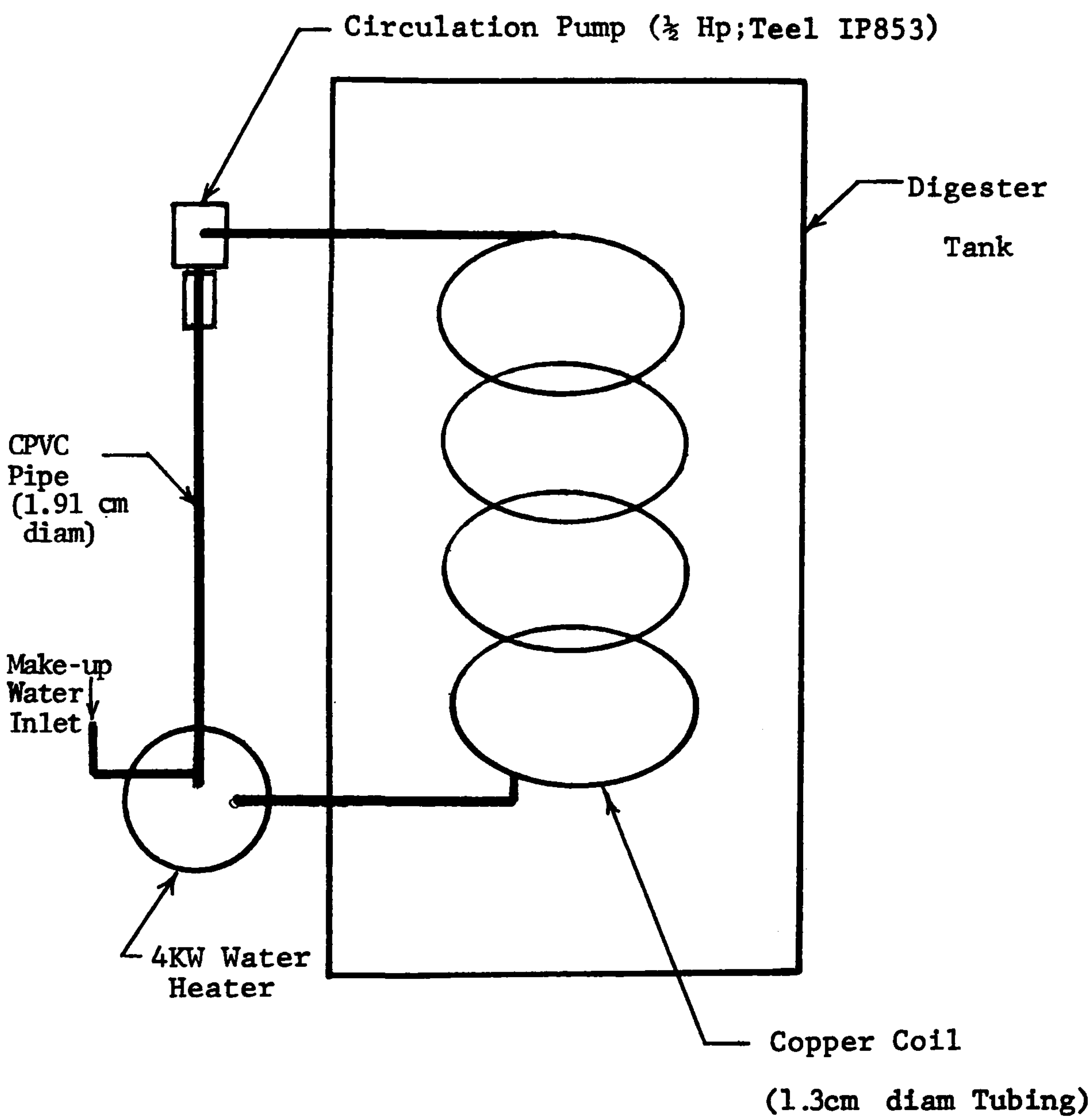

Figure 9. Digester heat exchanger system.

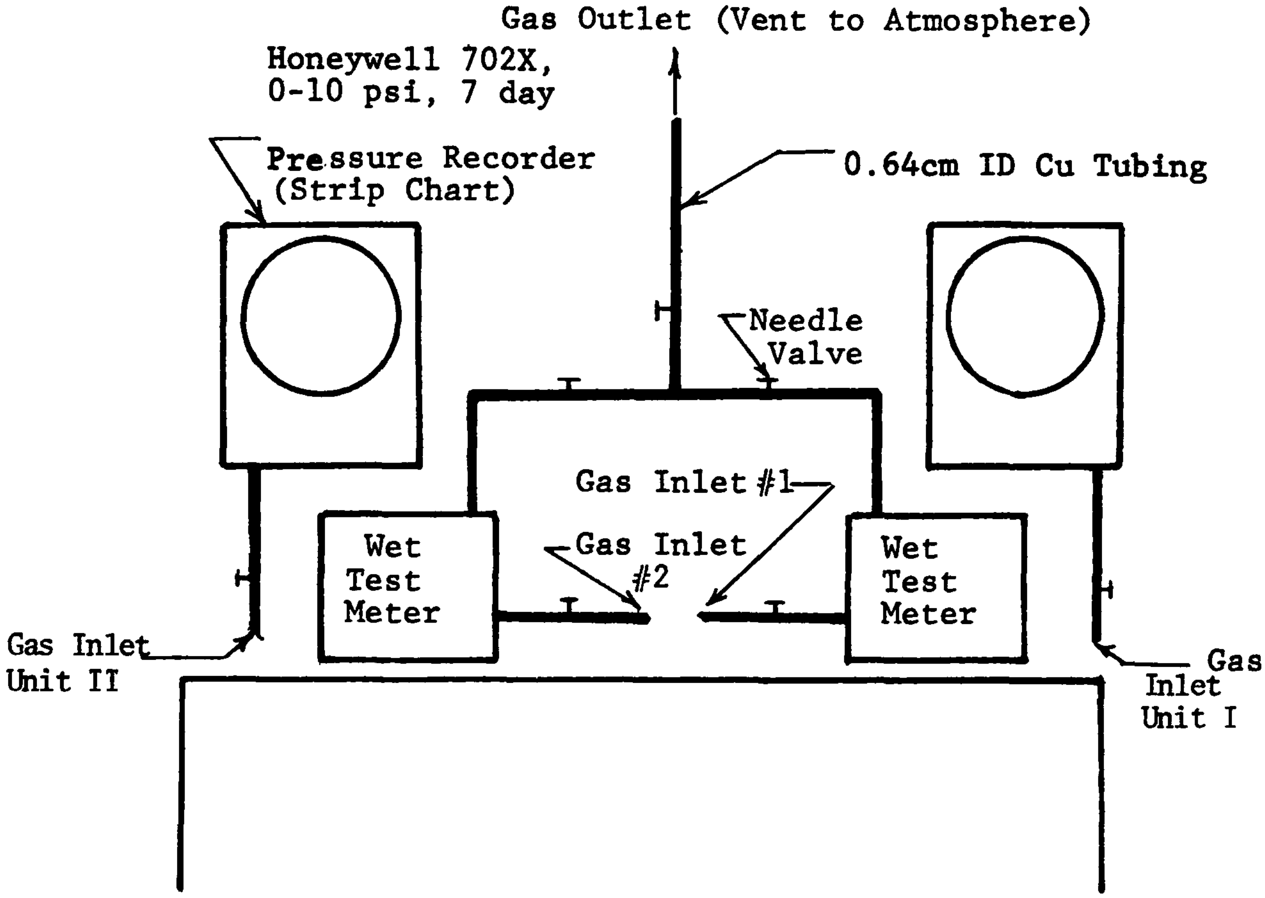

Figure 10. Schematic of digester monitoring equipment arrangement.

(7). Sludge temperature was monitored and adjustments were accomplished by the heat exchanger. Tank pressures were continuously monitored by the pressure recorder. Total volume of gas generated and the gas generation rate were monitored twice during a twenty-four-hour period by utilizing the wet test meters.

ANALYTICAL PROCEDURES

pH

The pH of the pilot plants and full-scale digester mixtures were made on a daily basis using a Beckman Electromate pH Meter.

Alkalinity

A sample of the digester effluent was centrifuged and the supernatant was then used for alkalinity determination according to the procedures described in (15) p. 49. The solution was titrated with 0.02 N H_2SO_4 to a pH of 4.5. All alkalinity reported are in mg/l as $CaCO_3$

Biochemical Oxygen Demand (BOD)

The BOD's for the influent, centrifuged effluent, and mixed liquor were determined by the procedure presented in Standard Methods (15), p. 415. The influent was the prepared manure or manure-grass mixture to be digested. The mixed liquor was the digested material removed from the digester, and the centrifuged effluent is the liquid portion of the mixed liquor. A secondary influent from the Metropolitan Nashville Waste Treatment Plant was used for seed in the BOD measurements. A DO meter was used for the dissolved oxygen determination. The sample size in each case was 20 ml.

Chemical Oxygen Demand (COD)

COD's for the influent, mixed liquor, and centrifuged effluent were determined according to procedures outlined in (15), p. 510. The sample size for these cases was also 20 ml.

Ammonia Nitrogen

Ammonia nitrogen measurements in the mixed liquor for all three units and the plant were by use of the distillation method described in (15), p. 391.

Gas Composition Analysis

The generated gases were analyzed using a Varian GLC gas chromatograph with a thermal conductivity detector. An eight-foot Poropak Q column at 40°C was utilized to determine the carbon dioxide and methane content of the sample. The instrument was calibrated using a standard gas mixture containing 40 percent carbon dioxide and 60 percent methane by volume.

The peak heights recorded on a strip chart recorder were used to determine the concentrations of CO_2 and CH_4.

Volatile Solids

The volatile solid measurements were performed by the procedure outlined in (15), p. 425. The results are given in mg/l.

Heavy Metal Analysis

Sludge samples from the three laboratory units and a digested manure sample were analyzed for Pb, Ca, Fe, Zn, Mn, Cd, and Al using an atomic absorption spectrophotometer. Only a very small number of samples were analyzed since the availability of the instrument was quite limited.

SECTION 6

RESULTS

PILOT PLANTS

The pilot plants were operated over a two-month period and data were taken on a daily basis. Typical experimental results are presented. The three units were loaded and after a fourteen-day detention time, analyses of the influent and effluent were begun. Gas production varied greatly from day to day as shown in Figure 11. Units II and III produced considerably more gas than Unit I. Gas production ranged from 8.5 to 17.0 liters per day (0.3 to 0.6 $ft.^3$/day).

The biochemical oxygen demand (BOD) for the influent, mixed liquor, and centrifuged effluent was measured during a fifteen-day test period. The results for each unit are presented in Figures 12, 13, and 14. The BOD's were reduced within all units, indicating that the dissolved oxygen was consumed by the microbial life while assimilating and oxidizing the organic matter present. In Unit I the BOD was reduced from around 9,000 mg/1 in the influent to nearly 3,000 mg/1 in the mixed liquor and 1,400 mg/1 in the centrifuged effluent (Figure 13). For the 50 percent-50 percent mixture in Unit III, similar results were obtained, as shown in Figure 14.

The chemical oxygen demand (COD) was determined on various sampling days for all three units. The experimental results are presented in Figures 15, 16, and 17. In all units there was a reduction in COD, indicating that organic matter was being oxidized.

Volatile solids were also measured in the influent, mixed liquor, and centrifuged effluent of each unit. As shown in Figures, 18, 19, and 20, there was a reduction in volatile solids upon digestion.

A 400 ml sample from each unit was oven-dried to a constant weight at 100°C and ground in Wiley mill. Total nitrogen was determined by the Kjeldahl Method and the crude protein content was calculated by multiplying the total nitrogen by the factor 6.25. Carbon and phosphorus contents were determined by the Walkley Black and Vanadomolybdophosphoric Yellow Color Methods, respectively (16). The organic matter (O.M.) was calculated by multiplying carbon by the factor of 1.72. The results of the analyses are presented in Table 2. There was very little difference in the analyses of the dried sludge taken from the three digestion units. The undigested manure, however, had a higher organic matter content and a lower crude protein percent than the digested samples. The ammonia nitrogen, pH and alkalinity of the mixed liquor varied only slightly among the units. These quantities were maintained within the range for optimal operating conditions as discussed previously.

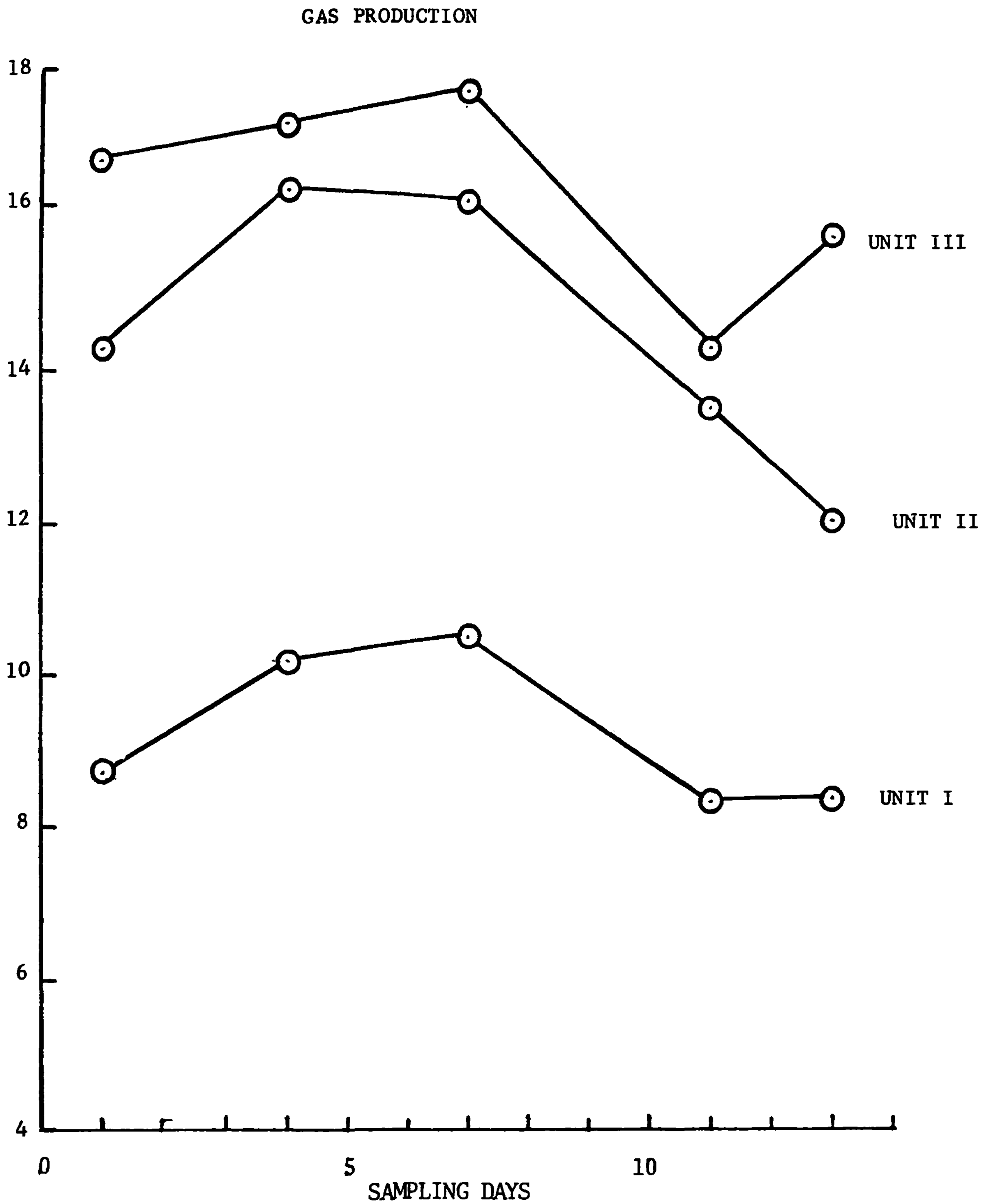

Figure 11. Laboratory pilot plant gas production.

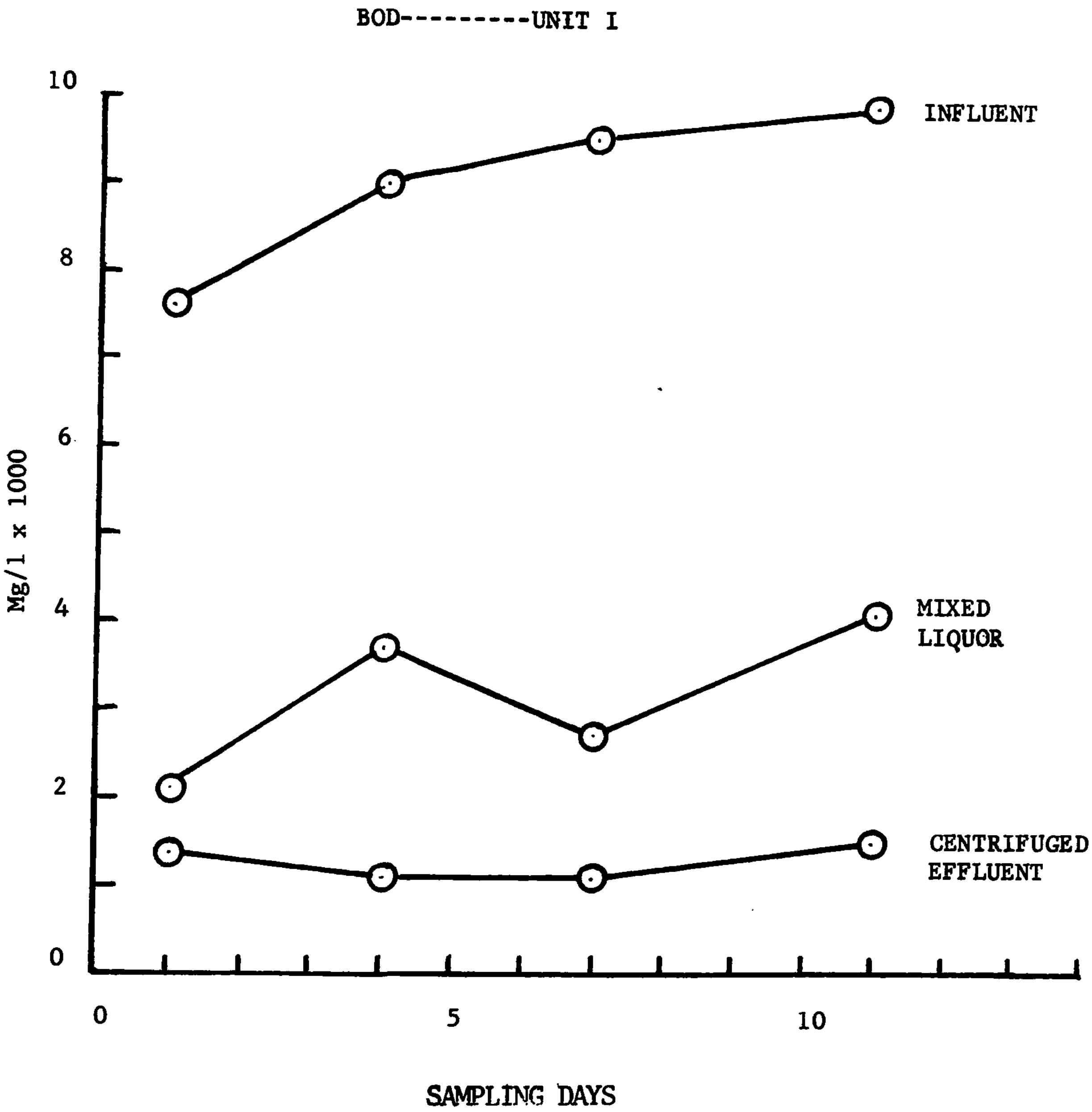

Figure 12. Biochemical oxygen demand for Unit I.

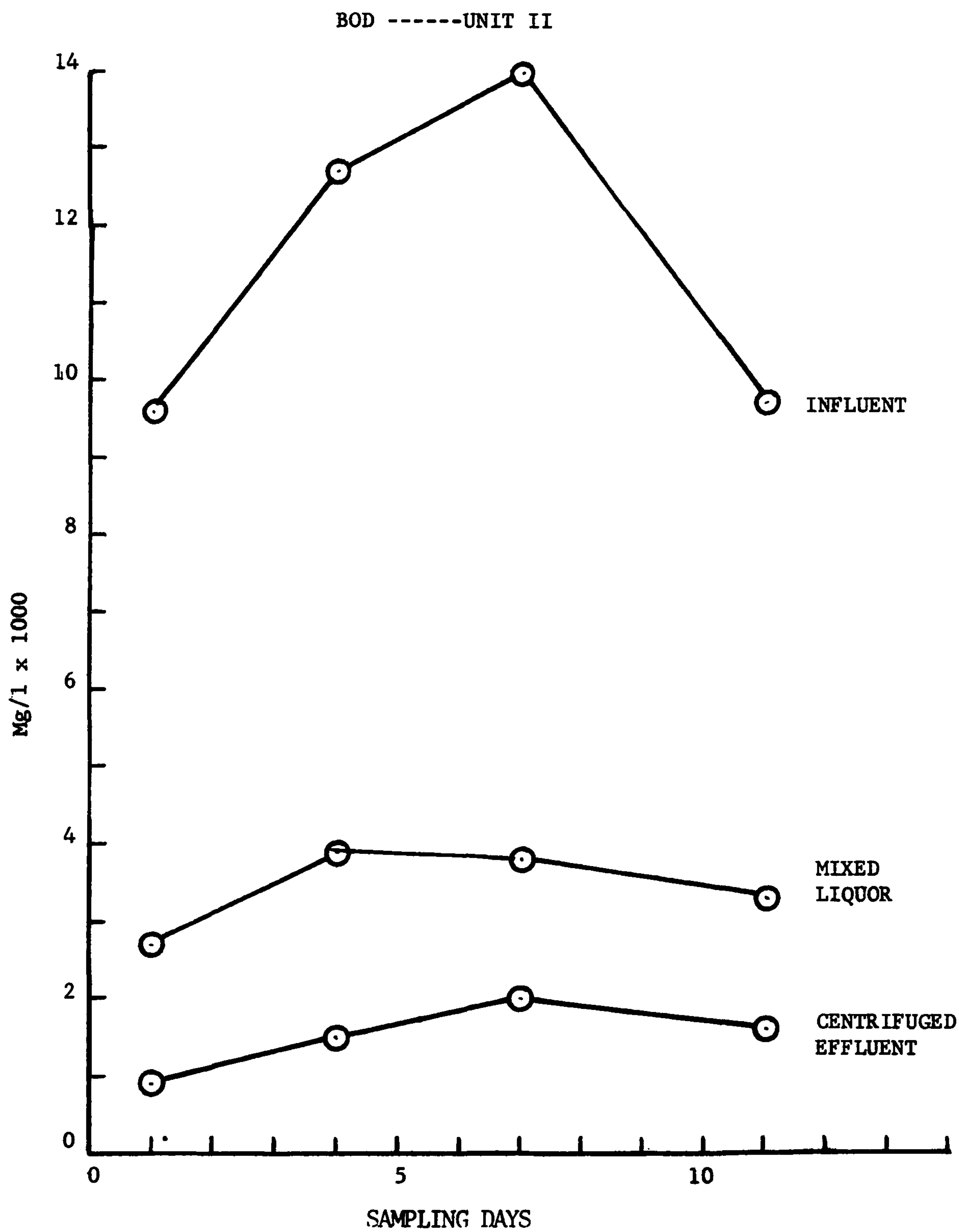

Figure 13. Biochemical oxygen demand for Unit II.

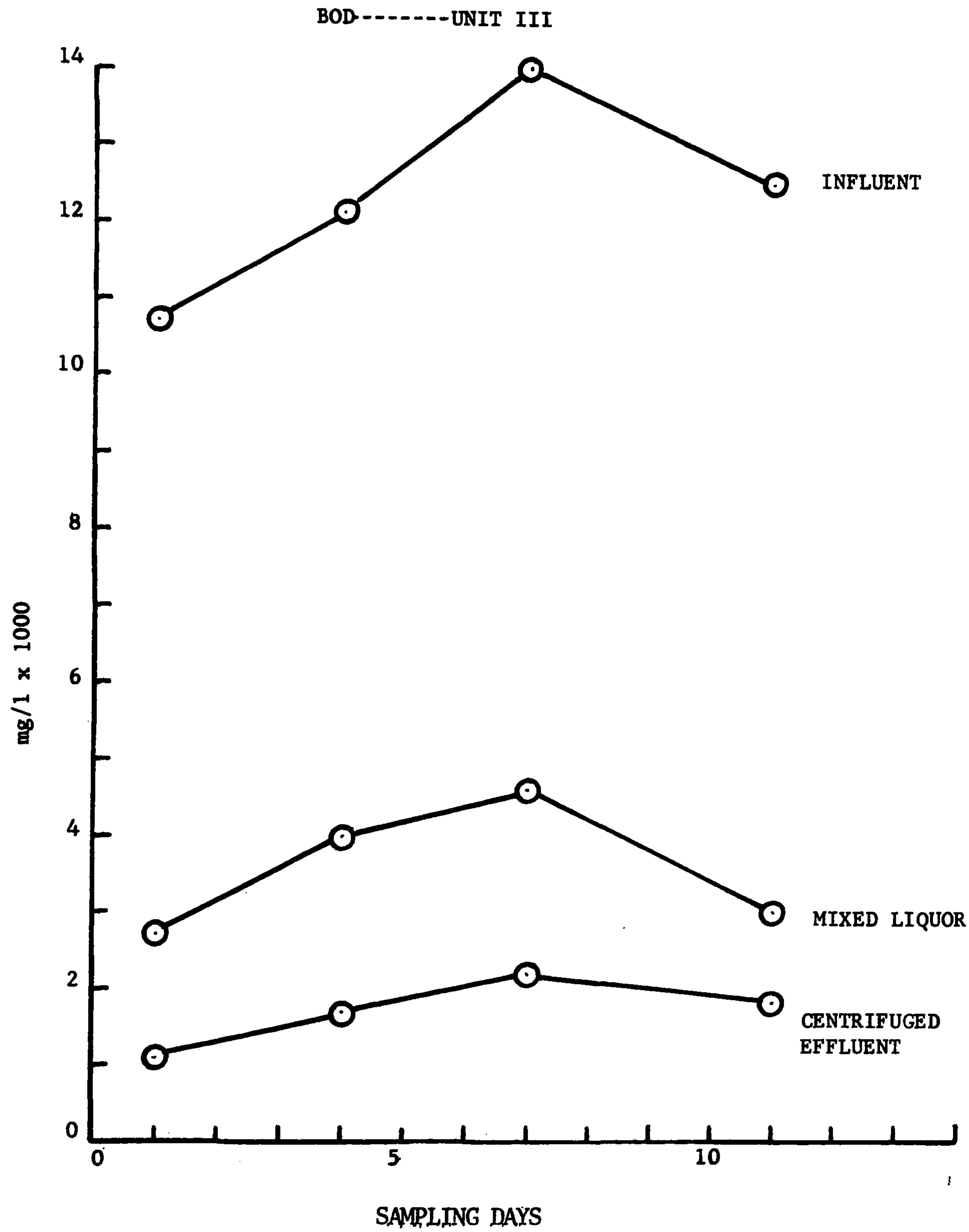

Figure 14. Biochemical oxygen demand for Unit III.

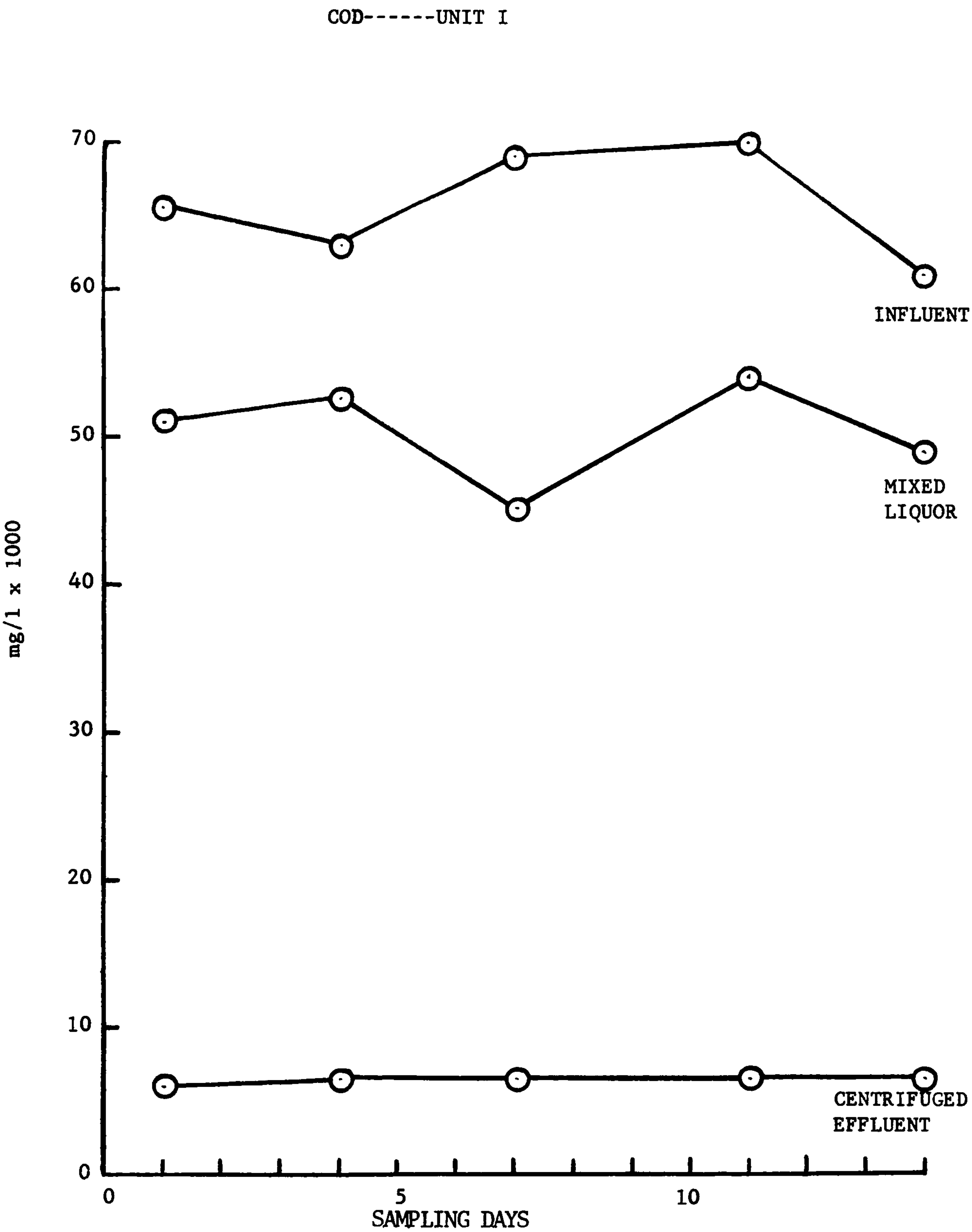

Figure 15. Chemical oxygen demand for Unit I.

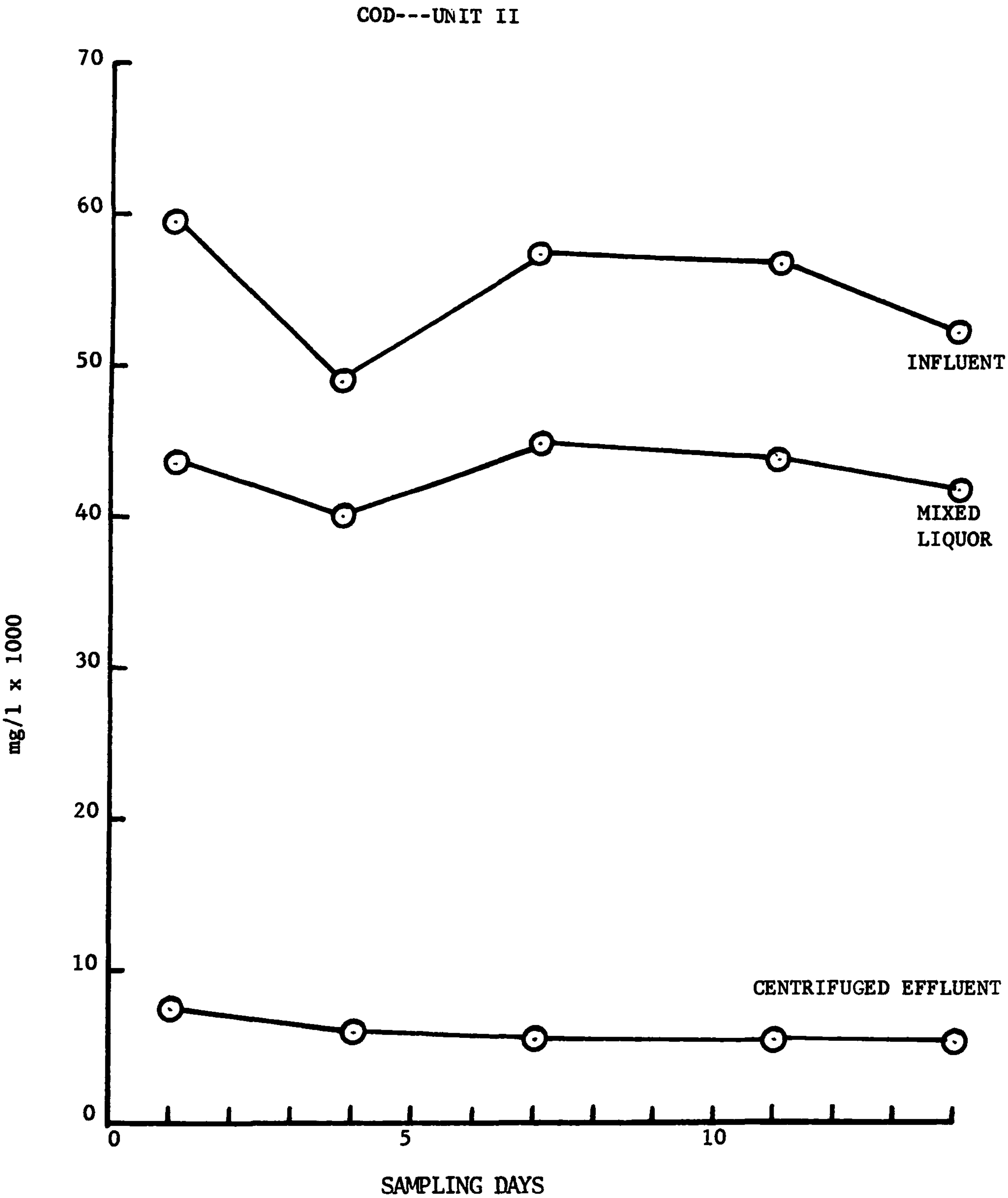

Figure 16. Chemical oxygen demand for Unit II.

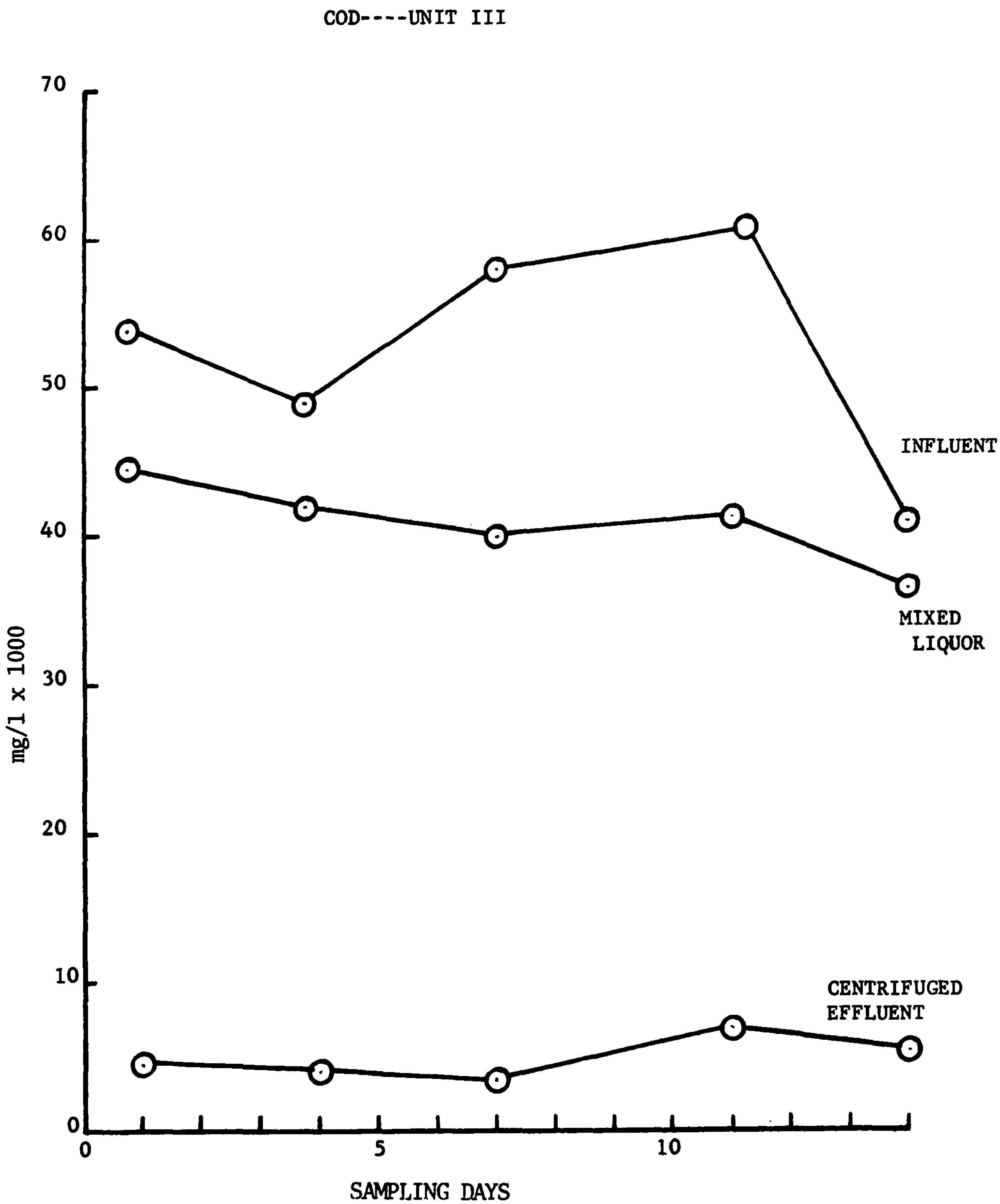

Figure 17. Chemical oxygen demand for Unit III.

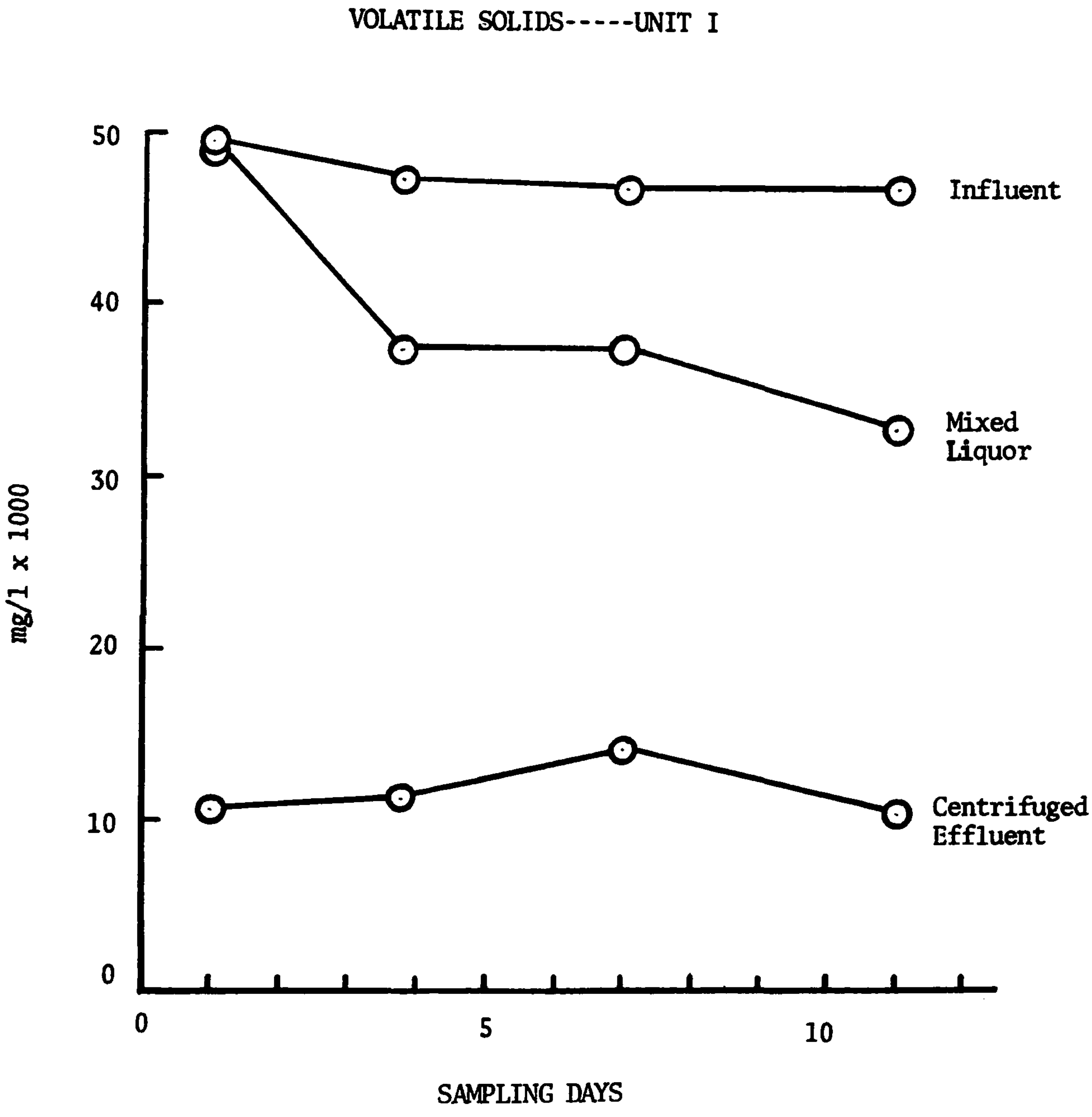

Figure 18. Volatile solids for Unit I.

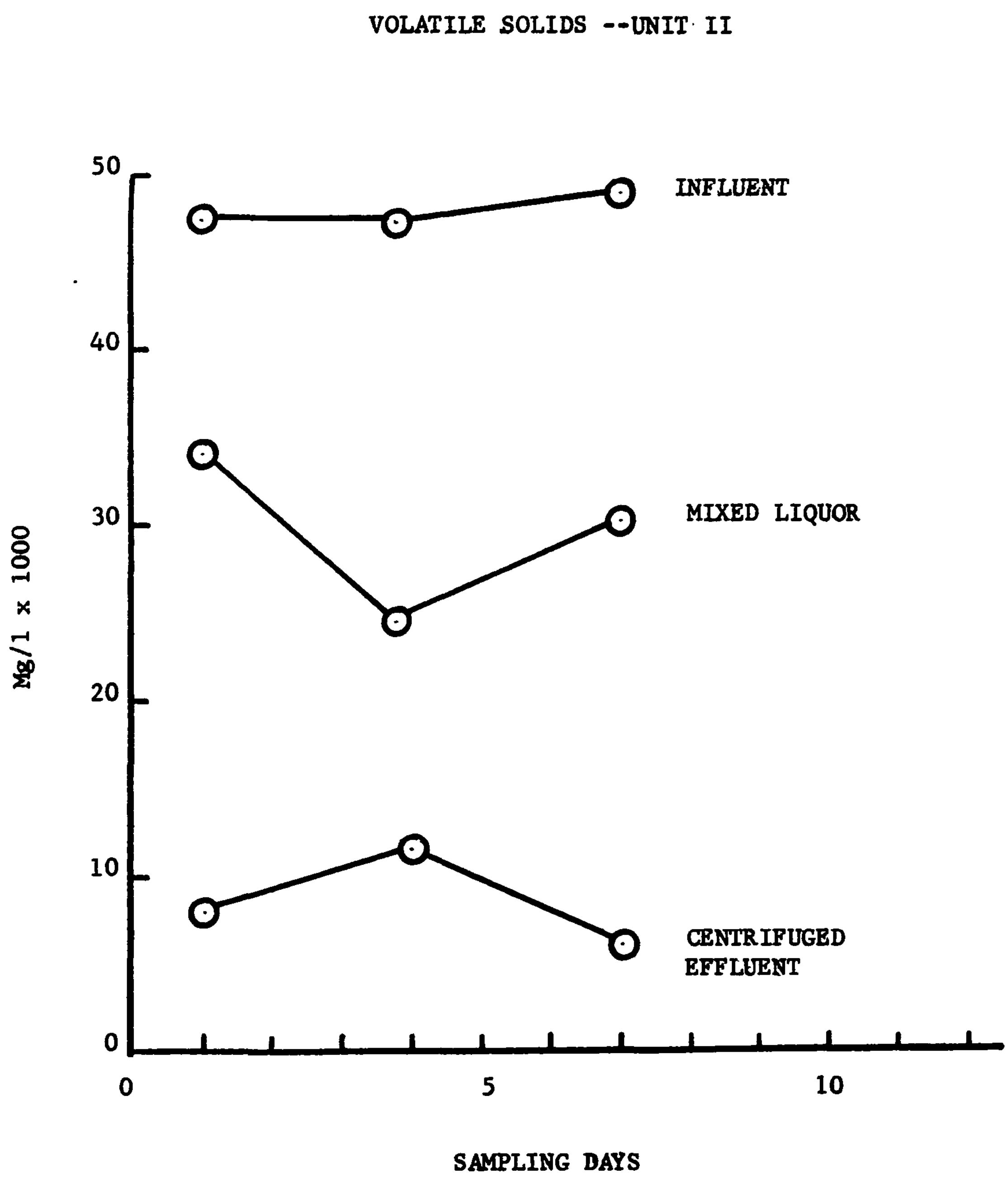

Figure 19. Volatile solids for Unit II.

VOLATILE SOLIDS ----UNIT III

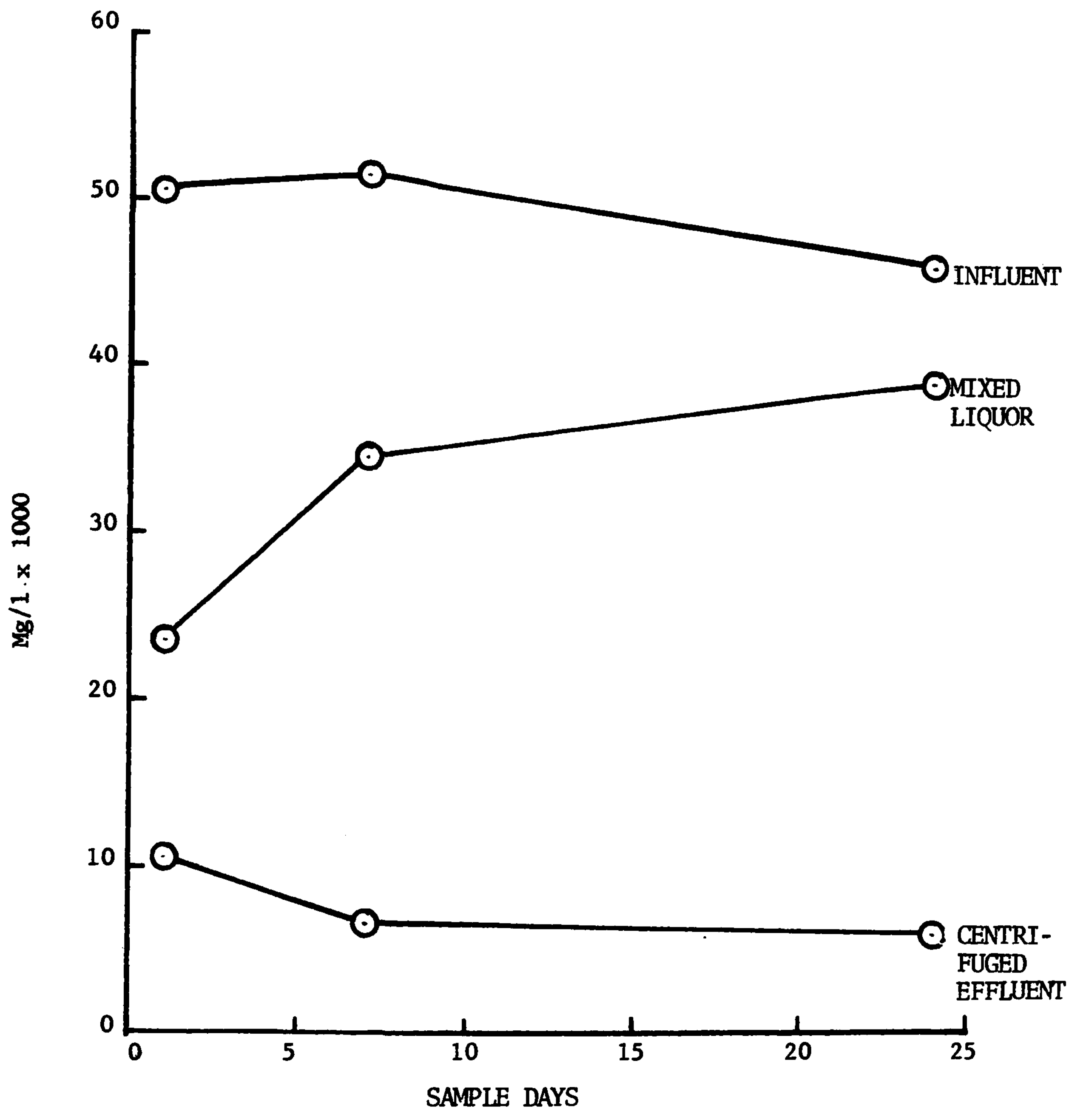

Figure 20. Volatile solids for Unit III.

TABLE 2. COMPOSITION OF DRIED SLUDGE AND MIXED LIQUOR

Unit	Dried Sludge (Percent)				Mixed Liquor		
	O.M.	Fat	Crude Protein	Phosphorus	NH_3-N (mg/l)	pH	Alkalinity (mg/l)
I	56.43	2.61	13.44	0.0247	212.5	7.74	8400
II	57.41	2.00	13.86	0.0285	168.9	7.84	7404
III	59.51	1.99	13.63	0.0266	169.8	7.83	7042
Undigested Manure	71.19	2.02	10.56	0.0252	-	-	-

TABLE 3. ANALYSIS OF HEAVY METALS (mg/kg dry weight)

	Pb	Cu	Fe	Zn	Mn	Cd	Al
Unit I	42	33	3300	320	380	1.5	3100
Unit II	50	45	3600	240	380	2.7	3000
Unit III	52	80	3000	300	340	7.5	2700
Undigested Manure	48	22	1800	480	280	1.4	1900

Analyses for heavy metals were made with an atomic absorption spectrophotometer but there were no great differences among the units, with the exception of higher Cu and Cd levels in Unit III (Table 3). All metal concentrations were higher in the digested samples than in the undigested manure, except for lead and zinc.

FULL-SCALE PLANT

Unit I of the full-scale plant was charged with 4765 liters of a mixture composed of water and manure (2.8 percent solids). After a fourteen-day detention time, seventy-five gallons of mixture were wasted and seventy-five gallons of fresh mix were added to the unit. Data from this operation for a six-week period are presented in Figures 21 and 22. The pH of the mixture was maintained above 6.5 by the addition of lime. The sludge temperature varied from 40°C to 44.4°C, being controlled by the internal copper coil heat exchanger. After a two-week detention time, the gas volume generation rate was 3.5 $ft.^3$/hr. and eventually increased to 5 $ft.^3$/hr. near the end of the testing period. Gas samples were analyzed in a gas chromatagraph. Typically, the generated gas was 62 percent methane and 38 percent carbon dioxide. The influent and effluent BOD and COD were measured. As in the pilot plants, both these quantities were reduced upon digestion.

Unit II was charged with 6056 liters of a water and 30 percent farm clippings- 70 percent manure mixture. However. operational difficulties prevented continuous data acquisition for this unit.

OPERATIONAL DIFFICULTIES

Several operational difficulties were encountered when large scale plant start-up was attempted. The coffee urn mixing arrangement was not satisfactory. When the measured quantity of feedlot manure was placed in the urn and mixed with water, the solution contained large lumps of manure and animal hair which soon caused the pumps to become clogged. Several methods of straining the mixture were attempted, but no feasible technique was found which could handle a 5 cm layer over the concrete pad of the plant and was allowed to dry. The dried manure was then ground into a fine powder in a hammer mill and stored in 190 liter barrels. When feeding the unit, the proper amount of manure was weighed, placed in the urn with water, and the contents were thoroughly mixed. The pumping and circulation problems were then solved.

Several days after the plant had been charged with a manure-water mixture, foaming of the supernatant was discovered. This phenomenon was possibly due to the lime which was added to insure the proper pH of the solution. The foam clogged all gas lines leading to the pressure recorder and wet test meter. On one occasion the tank pressure was well above 10 psig before the clogging was discovered. The situation was remedied by decreasing the volume of the mixture to approximately 4765 liters and by replacing the original 0.64 cm ID copper gas line with 1.3 cm ID tubing. The 1.3 cm ID line was connected to a flask by a rubber stopper and a second 0.68 cm ID tube allowed passage of the gas to the recording instruments (Figure 23).

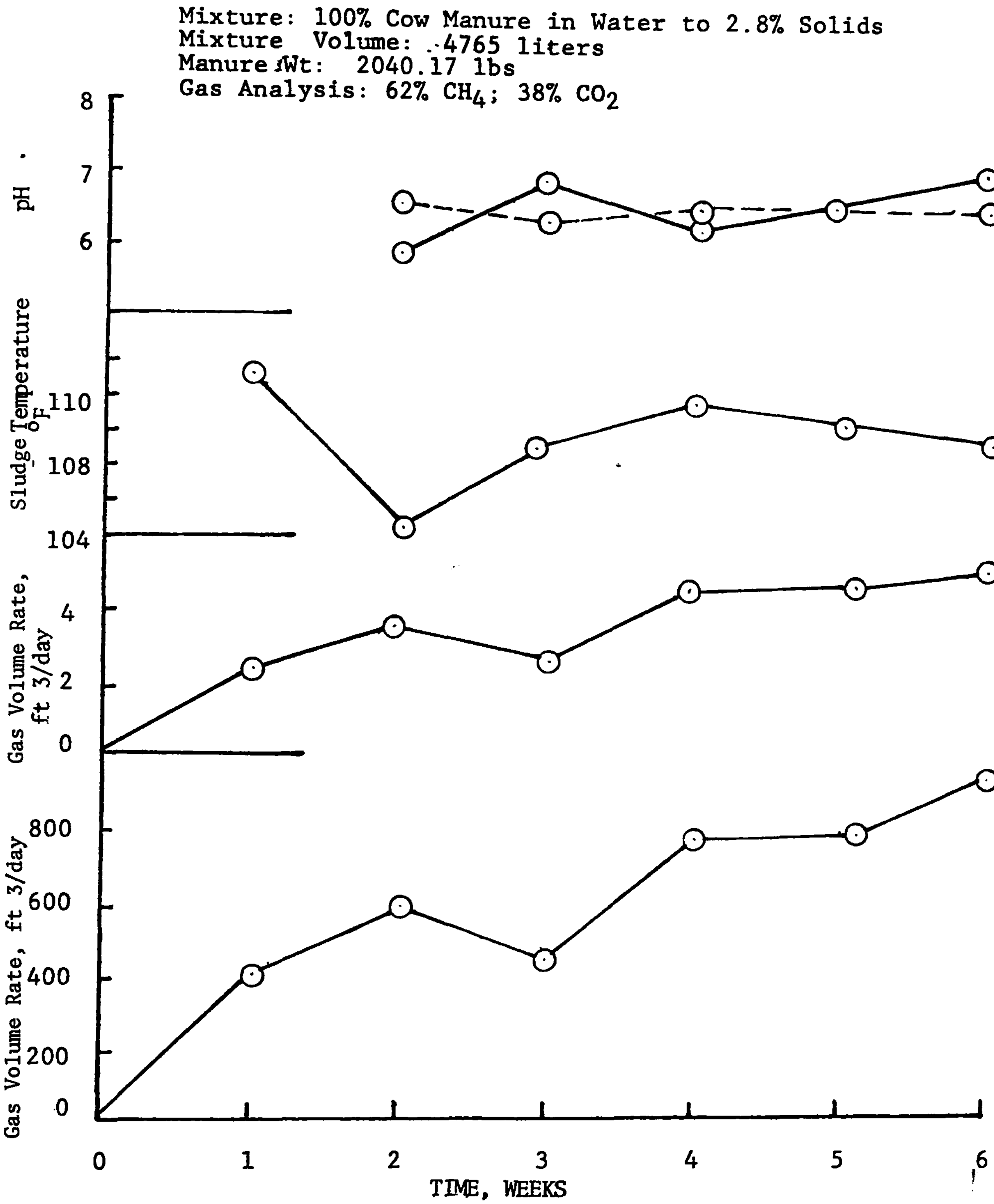

Figure 21. Full-scale plant pH, temperature, and gas volume generation rate.

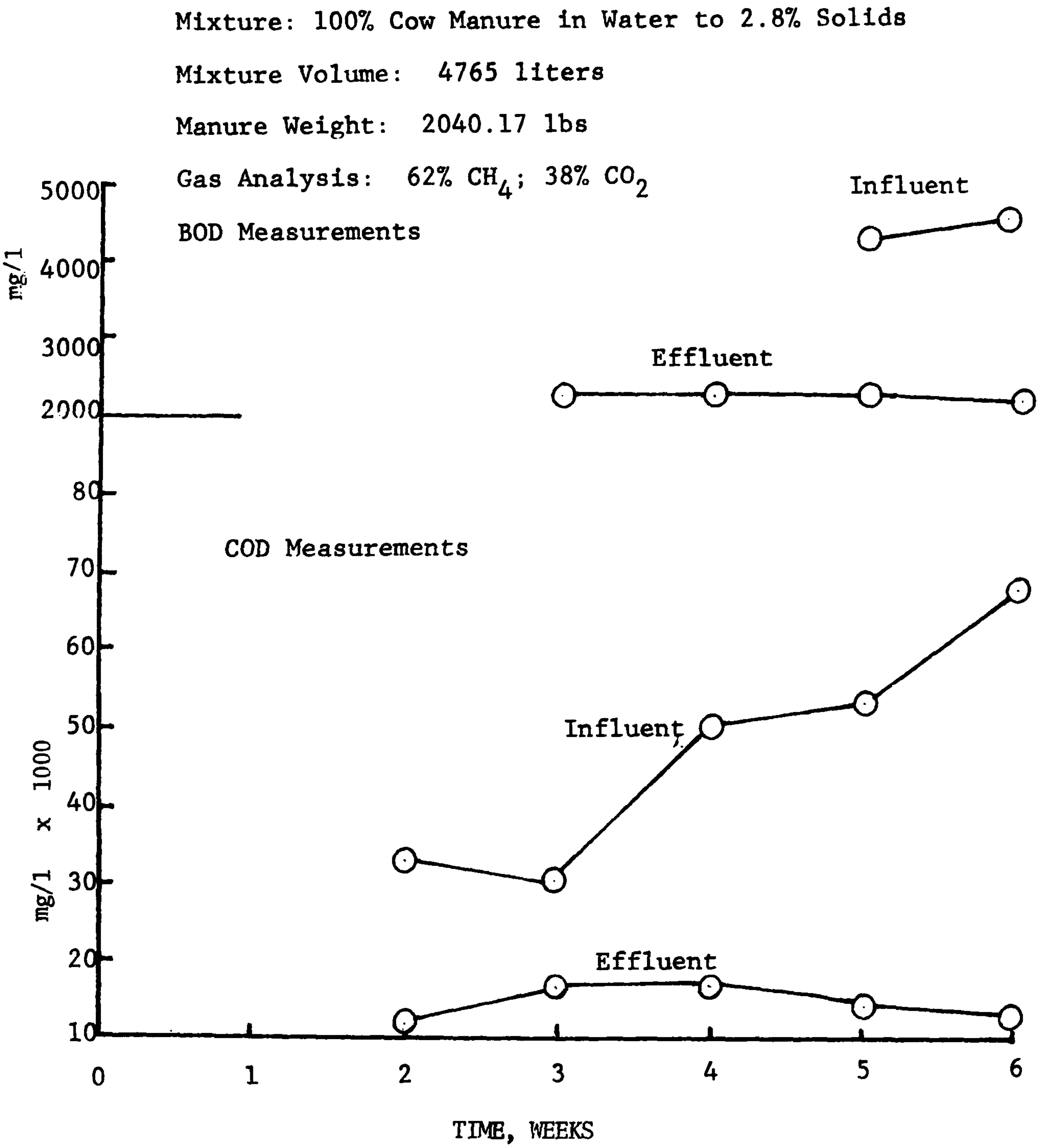

Figure 22. BOD and COD for the full-scale plant.

As the season began to change, temperature control became quite difficult. Eventually the daily temperature was such that heat transfer losses were greater than the heat exchanger could supply. In the new investigation, both tanks will be insulated with styrofoam sheets, and thus, temperature control should be easily maintained even in the colder months.

Measurement difficulties were also encountered. The gas chromatograph and atomic absorption spectrophotometer at Vanderbilt University were found in to inoperable. A division of the American Tobacco Company granted permission to use their gas chromatograph. However, the number of samples analyzed was limited. In the new investigation a GC will be purchased for the University. Only one heavy metal analysis of dried sludge sample could be made. The Varian Company was gracious enough to analyze the sample as an equipment performance demonstration.

Equipment delivery and construction delays greatly hampered the completion of the full-scale plant. It was impossible to gather a sufficient set of data. Fortunately, a two-year investigation to collect a comprehensive set of data has been funded.

NUTRITIVE VALUE OF THE ANAEROBICALLY DIGESTED SLUDGE

In addition to methane gas another useful byproduct of the anaerobic digestion process is the stabilized sludge. Specifically, the possibility of supplementing animal diets with the digested material has been investigated.

Ruminants possess a uniquely high capacity for digesting cellulose and hemicellulose of plant cell walls. However, 40 percent to 60 percent of this potential energy source escapes digestion and appears in the feces. As a result, millions of tons of undigested cell wall residues are excreted by ruminants annually. Such materials represent a vast potential source of energy for microbial fermentation. Anaerobic digestion of this excrement makes it a potential source of nutrients for livestock.

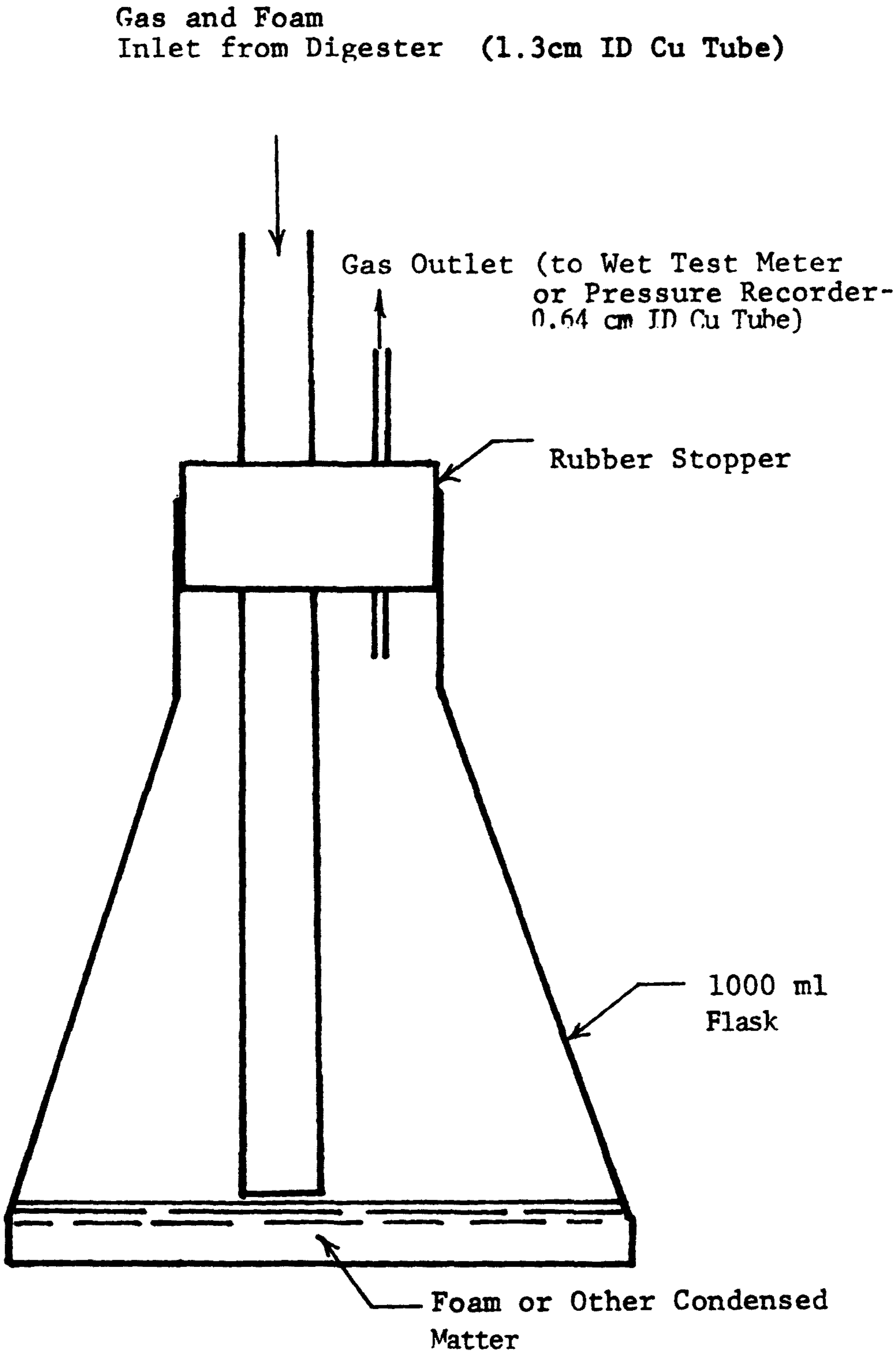

Figure 23. Output gas handling arrangement.

The sludge used in this study was taken from three laboratory pilot digestion units, (Unit I, 100 percent manure; Unit II, 70 percent manure and 30 percent grass; and Unit III, 50 percent manure and 50 percent grass) after a detention time of fourteen days at approximately 35°C. The sludge was dried to a constant weight at 105°C and ground in a Wiley mill, using a 2 mm screen. Composition of the sludge is shown in Table 4.

TABLE 4. COMPOSITION OF SLUDGE PERCENT

Unit	Moisture	Protein	Fat	Phosphorus	Ash	Cu	Fe
I	6.25	13.44	2.61	.0247	34.54	.0033	.33
II	6.16	13.86	2.00	.0285	34.52	.0045	.36
III	6.69	13.63	1.99	.0266	34.47	.0080	.30

A fortified corn - soybean meal diet (Table 5) served as the basal diet. Either 10% or 25% sludge from each unit was added to the diets at the expense of corn and soybean meal. All diets were calculated to be isonitrogenous (16% crude protein). Sixty-three female weanling rats weighing 40-70g were randomly distributed into 7 groups (three rats per treatment). The trial was replicated three times. All rats were individually caged. Feed and water were offered <u>ad libitum</u> for 21 days.

Individual rat weights and feed consumption data were recorded at weekly intervals. Average daily gain, average daily feed intake and feed per gain were computed.

The rats were also subjected to a three-day digestion trial in which the feces were collected and feed consumption monitored. The feed and feces were dried and the percent dry matter digestibility was determined. From this trial, the digestibility of protein was also determined. The feces of three rats from each treatment (one from each replica) along with each feed were analyzed for protein (Kjeldahl N x 6.25). The percent protein was multiplied by the dry feces collected and feed consumed, and the protein digestibility determined from the results.

The data from all trials were subjected to statistical treatment by the analysis of variance as outlined by Fisher (17).

TABLE 5. COMPOSITION OF DIETS PERCENT

Ingredient	Ration Number						
	1	2	3	4	5	6	7
Soybean Meal	22.0	20.7	18.9	20.7	18.9	20.7	18.9
Corn	70.0	61.3	48.1	61.3	48.1	61.3	48.1
Sludge Unit I	-	10.0	25	-		-	-
Sludge Unit II	-	-	-	10.0	25.0		
Sludge Unit III	-	-				10.0	25.0
Lard	5.0	5.0	5	5.0	5.0	5.0	5.0
Calcium Phosphate	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Limestone	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Trace Mineral Salt	0.5	0.5	.5	0.5	0.5	0.5	0.5
Vitamin Pre Mix	0.5	0.5	.5	0.5	0.5	0.5	0.5

The results of this experiment are presented in Tables 6 and 7. Rats fed ration 4 and 6 had similar growth rates to rats fed the basal ration; whereas, rats fed ration 2 showed a growth depression ($P < .05$). Growth depression of rats receiving 25 percent sludge (3,5, and 7) was highly significant ($P < .01$).

Average daily feed intake for all sludge treatments was higher than the basal. Rats fed treatments 2, 5, and 6 showed an average daily feed intake significantly higher ($P < .05$), and for rats fed treatments 3, 4, and 7 it was significantly higher ($P < .01$) than for rats fed the basal ration.

Feed per gain ratios of all rats fed sludge treatments were significantly higher than for rats fed the basal ration ($P < .05$) with the exception of rats fed treatments 3 and 7 which had feed per gain ratios that were significantly higher ($P < .01$) than the rats fed the basal ration.

Dry matter digestibility of diets decreased as the level of sludge was increased in the diets. The digestibility of treatments 2, 4, and 6 were significantly lower ($P < .05$) than the basal, and it was significantly lower ($P < .01$) than the basal for treatments 3, 5, and 7.

Protein digestibility of all diets with sludge treatments was significantly lower ($P < .01$) than the basal. The protein digestibility of all sludge treatments was similar with the exception of treatment 3, which was significantly lower ($P < .01$) than the other sludge treatments.

Growth performance was not greatly reduced by the addition of 10 percent sludge when compared to the control diet; however, 10 percent sludge did considerably lower feed conversion and digestibility of the diets. Performance of rats receiving 25 percent sludge was lowest of all treatment groups. This included average daily gain, feed conversion and digestibility of dry matter and protein. This reduction in performance agrees with the findings of Harmon (18).

The lower protein digestibility of the sludge-containing diets may be a consequence of a high proportion of non-proetin nitrogen, since microorganisms are rich sources of non-protein nitrogen. The feeding of this sludge to ruminant animals would be desirable, since the non-protein nitrogen fraction would be utilized more efficiently than in non-ruminants.

The overall performance of the rats on the different dietary treatments was in the following order:

1. Treatment 1 Basal
2. Treatment 4 10 Percent of 70-30 manure-grass sludge
3. Treatment 6 10 Percent of 50-50 manure-grass sludge
4. Treatment 2 10 Percent of 100 Percent manure-sludge
5. Treatment 5 25 Percent of 70-30 manure-grass sludge
6. Treatment 7 25 Percent of 50-50 manure-grass sludge
7. Treatment 3 25 Percent of 100 Percent manure-sludge

The results of the experiment indicate that:

(1) Ration with up to 10 percent anaerobically processed cow manure and grass mixtures can be fed rats with results similar to those on non-manure diets. This was probably due to increased feed intake by rats fed the 10 percent sludge diets.

(2) The use of anaerobically processed wastes resulted in less efficient feed conversion.

(3) The addition of sludge did not reduce acceptability of the diets.

(4) The incorporation of sludge in the diets decreased dry matter and protein digestibility.

(5) The poor overall performance of rats fed the 25 percent sludge diets probably resulted from the inability of rats to efficiently digest the dry matter and protein of these diets.

(6) The different sludges had very little effect on the performance of rats. The slight difference favored Unit II followed by Unit III.

TABLE 6. SUMMARY OF GROWTH AND FEED DATA

	Treatments No.	Treatments Percent Sludge	Average daily gain (g)	Average daily feed intake (g)	g Feed/ g gain
Basal	(1)		4.31a	11.18a	2.59a
100 Percent manure	(2)	10 Percent	4.04c	12.63b	3.31b
Sludge	(3)	25 Percent	3.86c	13.62c	3.52c
70 - 30 manure Grass mixture	(4)	10 Percent	4.26a	12.90c	3.03b
Sludge	(5)	25 Percent	3.91c	12.68b	3.24b
50 - 50 manure Grass mixture	(6)	10 Percent	4.19a	12.91b	3.05b
Sludge	(7)	25 Percent	3.80c	13.14c	3.46c

b Significantly different ($P < .05$) from a

c Significantly different ($P < .01$) from a

TABLE 7. SUMMARY OF DRY MATTER AND PROTEIN DIGESTIBILITY DATA

	Treatments No.	Treatments Percent Sludge	Dry Matter Digestibility Percent	Protein Digestibility Percent
Basal	(1)		86.5a	86.3a
100 Percent manure	(2)	10 Percent	81.4b	81.9c
Sludge	(3)	25 Percent	73.7c	75.6c
70-30 manure	(4)	10 Percent	76.0b	80.1c
Grass mixture Sludge	(5)	25 Percent	73.6c	80.3c
50-50 manure	(6)	10 Percent	79.4b	80.4c
Grass mixture Sludge	(7)	25 Percent	73.2c	79.8c

b Significantly different ($P < .05$) from a

c Significantly different ($P < .01$) from a

(7) There were no noticeable signs of sickness or abnormalities as a result of consuming any of the diets.

Although feed conversion and digestibility of diets decreased as the level of sludge was increased, this experiment reveals that anaerobically processed organic wastes are consumed readily, and when incorporated into a balanced diet will support animal growth.

REFERENCES

1. Dugan, G. L., Young, R., and Takamiya, G. "Animal Waste Management in Hawaii." Journal of Water Pollution Control Federation, 45, No. 4, April, 1973, p. 742.

2. Ghosh, S. and Pohland, F. G. "Kinetics of Substrate Assimilation and Product Formation in Anaerobic Digestion." Journal of Water Pollution Control Federation, 46, No. 4, April, 1974, pp. 748-759.

3. Graef, S. P., and Andrews, J. F. "Stability and Control of Anaerobic Digestion." Journal of Water Pollution Control Federation, 46, No. 4, April, 1974, pp. 66-683.

4. Anon. "Natural Gas: Worry About Supply." Chemical and Engineering News, 48, No. 14, March 30, 1970, p. 8.

5. Ghosh, S. "Anaerobic Processes." Annual Literature Review - Journal of Water Pollution Control Federation, 45, No. 6, June, 1973, pp. 1063-1074.

6. Pfeffer, J. T. "Reclamation of Energy from Organic Waste." EPA-670/ 2-74-016, March, 1974.

7. McKinney, R. E., and Conway, R. A. "Chemical Oxygen in Biological Waste Treatment." Sewage and Industrial Wastes, 29, No. 10, October, 1957, pp. 1097-1106.

8. McCarty, P. O. "Anaerobic Waste Treatment Fundamentals--Part One, Chemistry and Microbiology." Public Works, 95, September, 1964, pp. 107-112.

9. Pfeffer, J. T. "Increased Loadings on Digesters with Recycle of Digested Solids." Journal of Water Pollution Control Federation, 40, No. 11, November, 1968, pp. 1920-1933.

10. Buswell, A. M., and Mueller, H. F. "Mechanisms of Methane Fermentation." Industrial and Engineering Chemistry, 44, 1952, pp. 550-552.

11. McCarty, P. L. "Anaerobic Waste Treatment Fundamentals--Part Four, Process Design." Public Works, 95, December, 1964, pp. 95-99.

12. Speece, R. E., and McCarty, P. L. "Nutrient Requirements and Biological Solids Accumulation in Anaerobic Digestion." Advances in Water Pollution Research, 2, pp. 305-322. Eckenfelder, W. W., Editor. Pergamon Press, New York, 1964.

13. McCarty, P. L. "Anaerobic Waste Treatment Fundamentals--Part Two, Environmental Requirements and Control." Public Works, 95, October, 1964, pp. 123-126.

14. McCarty, P. L. "Anaerobic Waste Treatment Fundamentals--Part Three, Toxic Materials and Their Control." Public Works, 95, November, 1964, pp. 91-94.

15. Standard Methods for the Examination of Water and Wastewater. Orland, H. P., Editor. 12th Ed., American Public Health Assocation, Inc., New York, 1965.

16. Jackson, M. L. Soil Chemical Analysis. Prentice Hall, Inc. Englewood Cliff, N. J. pp. 219-221; 153-154, 1958.

17. Fisher, R. A. Statistical Methods for Research Workers. 14th Ed. Oliver and Boyd, Edinburough, 1970.

18. Harmon, B. G., Day, D. L., Baker, D. H., and Jensen, A. H. "Nutritive Value of Aerobically and Anaerobically Processed Swine Waste." Journal of Animal Science, 1973, 37, p. 510.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/2-77-089		

4. TITLE AND SUBTITLE	5. REPORT DATE / 6. PERFORMING ORGANIZATION CODE
A COMPLETE DISPOSAL-RECYCLE SCHEME FOR AGRICULTURAL SOLID WASTES	5. May 1977 issuing date 6.
7. AUTHOR(S) Michael R. Busby, Greg Tragitt, Roland Norman, and Kenneth Hillsman	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Tennessee State University Nashville, Tennessee 37203	10. PROGRAM ELEMENT NO. 1HB617 11. CONTRACT/GRANT NO. R-802739
12. SPONSORING AGENCY NAME AND ADDRESS Robert S. Kerr Environmental Research Lab. - Ada, OK Office of Research and Development U.S. Environmental Protection Agency Ada, Oklahoma 74820	13. TYPE OF REPORT AND PERIOD COVERED Final (1/74-6/76) 14. SPONSORING AGENCY CODE EPA/600/15

15. SUPPLEMENTARY NOTES

16. ABSTRACT

With the advent of the 70's, there has been an increasing national concern for the growing energy shortage as well as the problem of organic waste disposal. These two problems, while at first glance appear unrelated, are dealt with simultaneously by an anaerobic digestion process. Such a process produces not only a useful fuel, methane, but also is a potential source of a stabilized fertilizer and a nutritive supplement to animal diets. This biological process has been used for decades, but the economic feasibility of incorporating such a process on a typical small farm has not been clearly established.

This investigation applied the anaerobic process to the production of methane gas and a stabilized sludge from cow manure and farm clippings in laboratory pilot plants as well as a full-scale (2,000 gal.) digester system. The quantity and quality of gas produced, the biochemical and chemical oxygen demands, and the nutritional value of the digested sludge for both the laboratory and full-scale plants were evaluated.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Wastes, Anaerobic processes, Fertilizers, Methane, Nutritive value, Animal nutrition	Farm wastes, Anaerobic digestion, Energy,	02/C

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	60
	20. SECURITY CLASS *(This page)* UNCLASSIFIED	22. PRICE

EPA Form 2220-1 (9-73)

☆ U. S. GOVERNMENT PRINTING OFFICE: 1977-757-056/6449 Region No. 5-11