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TOXICITY OF FIBER IN CHEMI-THERMOMECHANICAL PULP WASTEWATERS
TO ANAEROBIC BACTERIA: A BATCH ASSAY AND REACTOR STUDY
COMPARISON

by

Debra Ann Richardson

A thesis
submitted under the supervision of
Dr. Ronald L. Droste and Dr. Kevin J. Kennedy

in partial fulfillment
of the requirements for the degree of
Master of Applied Science
in
Civil Engineering

Department of Civil Engineering
University of Ottawa
Ottawa, Canada
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ABSTRACT

Toxicity of chemi-thermomechanical pulping (CTMP) wastewaters was tested in batch assays and in two-stage anaerobic sludge blanket reactors. In batch serum bottle tests particulate constituents were responsible for 80 - 90% of the inhibition of acetoclastic activity and the soluble (fiber-free) fraction accounted for 10 - 20%. Performance of three two-stage anaerobic sludge blanket reactor systems was compared. One reactor received fiber-free, one received clarified, and the other unaltered wastewater for 140 days. All reactors became acclimatized to the CTMP and developed a tolerance to resin acid concentrations as high as 300 to 1500 mg/L in the sludge bed. The performance of the fiber-free system was superior throughout the experiment, but the difference in system performances did not reflect the batch test results. At an organic loading rate of 17 - 22 g soluble COD/g VSS·d the fiber-free system treated 100% CTMP wastewater with 42% and the fiber system treated with 38% removal efficiency. Fiber accumulation in the sludge bed of the systems treating raw and clarified CTMP caused a 70% increase in bed volume, resulting in deterioration of sludge settleability and led to an increased likelihood of biomass loss.

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NOMENCLATURE

ADMT	=	air dried metric tonne
ATA	=	Anaerobic Toxicity Assay
BCTMP	=	bleached chemi-thermomechanical pulp
BM	=	reactor biomass [M]
BOD ₅	=	five day biochemical oxygen demand [M/L ³]
COD	=	chemical oxygen demand [M/L ³]
CTMP	=	chemi-thermomechanical pulp
DTPA	=	Diethylene-triamine-penta-acetic acid
DBS	=	Division of Biological Sciences
DOM	=	Domtar
F	=	conversion factor
FSS	=	fixed suspended solids [M/L ³]
H	=	wastewater containing wood fines in excess of N
HRAT	=	high rate anaerobic treatment
HRT	=	hydraulic retention time [T]
MATA	=	Modified Anaerobic Toxicity Assay
MBAT	=	Modified Batch Activity Test
N	=	wastewater containing normal levels of wood fiber and fines
OLR	=	organic loading rate [M/M·T]
PA	=	preacidification
PGW	=	pressurized groundwood pulp
Q	=	flow rate [L ³ /T]
QRP	=	Quesnel River Pulp Company
RMP	=	refiner mechanical pulp
RCF	=	relative centrifugal force

SVI = sludge volume index
 SLR = specific loading rate [M/M·T]
 SMPR = specific methane production rate [M/M·T]
 SRR = specific reaction rate [M/M·T]
 SGW = stone groundwood pulp
 t = time [T]
 TMP = thermomechanical pulp
 TSS = total suspended solids [M/L³]
 UASB = upflow anaerobic sludge blanket
 V = volume [L³]
 VFA = volatile fatty acid [M/L³]
 VOA = volatile organic acid
 VSS = volatile suspended solids [M/L³]
 V₅₀ = upflow velocity corresponding to washout of 50% of
 the sludge in the sludge settleability test [L/T]

Subscripts

e = effluent
 f = feed
 g = biogas
 H = headspace
 M = manometer
 s = sediment
 S = soluble
 T = total

CHAPTER 1

INTRODUCTION

Canadian regulations controlling the discharge of pulp and paper liquid effluents to watercourses were established by the federal government in 1971. By 1982, the discharge of effluents by several of the older chemical pulping mills was still not in compliance with the federal regulations.

Chemical pulping involves cooking wood chips in a pressure vessel with chemicals and steam to separate the wood chips into fibers. The use of strong chemicals to delignify the wood solubilizes a large portion of the raw wood resulting in low yields and highly contaminated effluents.

A number of these mills were upgraded to mechanical pulping mills. This reduced the effluent emissions and thereby improved the level of compliance with the discharge regulations.

There are several mechanical pulping technologies available which produce paper products of various qualities. Mechanical pulp is produced either by grinding the logs into separate fibers with rotating stones or by forcing chips between grooved rotating steel plates in refiners; stone and pressurized groundwood pulp (SGW and PGW), respectively.

The results are high yields with extremely low pollution levels. Other mechanical pulping technologies include: refiner mechanical pulp (RMP), thermomechanical pulp (TMP), and chemi-thermomechanical pulp (CTMP). CTMP has been a popular chemical-mechanical pulping replacement choice from a chemical process because the pulp has the superior properties similar to chemical pulps and the high yield advantages of mechanical pulps. However, relative to the traditional mechanical pulping technologies, CTMP pulping results in more highly contaminated effluents. The necessity for an appropriate pollution abatement technology for CTMP effluents in order to comply with the federal regulations remains paramount.

Research into pollution abatement technology for CTMP effluents was not a priority consideration when CTMP mills were implemented because the pollution emissions from the CTMP are much lower than those from the old chemical mills. The emission levels of CTMP effluents, although much lower, do not satisfy the current regulations for discharge into watercourses. Pretreatment of CTMP effluents prior to discharge into watercourses is required to satisfy the regulations. The compilation of effluent characteristics, assessment of viable treatment options, and implementation of CTMP mill effluent treatment systems to satisfy the effluent emission criteria has now been made mandatory.

The unique characteristics of CTMP effluents (high temperatures, toxicity, organic and extractive concentra-

tions with low nutrient levels and low five day biochemical oxygen demand (BOD₅)/chemical oxygen demand (COD) ratio and the lack of data describing the characteristics of CTMP wastewaters makes the selection of an appropriate waste treatment technology difficult (Cornacchio and Hall, 1988). Typically, wastewater treatment in the pulp and paper industry includes one or more of the following: primary treatment for suspended solids removal proceeded by aerobic treatment and/or anaerobic treatment.

The application of aerobic and anaerobic technologies to treat CTMP effluents has been reviewed by Turk (1988). These include aerobic processes: aerated lagoons, activated sludge systems, and high rate anaerobic processes; fixed film, fluidized bed, and suspended growth systems.

Aerobic treatment has been the treatment method of choice at most Canadian pulp mills. Anaerobic treatment has received little attention because wastes from pulp mills have been deemed to be too dilute to render it cost effective. The increased use of internal recirculation of process waste and the advent of TMP and CTMP technologies has resulted in the production of more concentrated effluents which are more amenable to anaerobic treatment. Although most Canadian CTMP mills treat their effluents in aerated lagoons, the high rate anaerobic treatment (HRAT) process in combination with aerobic polishing has been identified by government agencies, academia, and private enterprise as an appropriate alternate method of treatment (Turk, 1988).

HRAT is a technology developed within the past decade. In HRAT systems, the biomass is retained within the system. This allows higher hydraulic loading of the system. The retention time required for treatment, relative to conventional anaerobic treatment technologies, is significantly reduced. In other words, a high rate of treatment is possible as the name, HRAT, implies.

Depending on the characteristics of the wastewater to be treated, an acidification tank preceding the bioreactor may be incorporated into the treatment system. In the acidification tank, hydrolysis, liquefaction, and acidification of the wastewater occurs. Stabilization and methane formation take place in the second stage. Systems which include this tank are referred to as two-stage anaerobic digestors.

Several commercial designs of this system have been developed. The Biopaq system, developed in the Netherlands by Paques BV, was the first two-stage HRAT process treating a paper mill effluent to be implemented. The system, which consists of a preacidification (PA) tank followed by an up-flow anaerobic sludge blanket (UASB) bioreactor, became operational in 1983 at the Roermond waste paper processing mill in the Netherlands (Turk, 1988).

The Biopaq system was purchased by the Quesnel River Pulp Company (QRP), a TMP/CTMP pulp and paper mill in Quesnel River, British Columbia, from Paques Lavalin, a company which markets the Biopaq system in North America. A

pilot scale two-stage system followed by aerobic lagoons was made operational in 1987.

The pilot plant was operated from June 1987 to June 1988. The main objective of the pilot plant study was to see whether or not the British Columbia Ministry of the Environment Level A discharge criteria could be achieved with the pilot scale treatment facility. Specifically, the pilot plant study was conducted to investigate system performance, efficiency of sludge production, behavior of wastewater change from TMP to CTMP, treatment system effluent concentrations, and fish toxicity reduction.

During the pilot study, operational difficulties were encountered. One problem was associated with the inclined screen, a primary treatment unit for the removal of suspended matter from the waste stream prior to the biological treatment process. The inclined screen was effective for the removal of wood chips, however, it was not effective for removal of suspended wood fiber. As a result, wood fibers in the wastewater entered the treatment facility and caused blockage of system lines.

Granular sludge loss was also associated with the high fiber concentration in the influent to the UASB reactor. The fibers appeared to attach to the granules. This was believed to have reduced the settling capacity of the granules causing them to float to the top of the UASB and to be discharged from the reactor. A Krofta, an air flotation unit for fiber removal, was installed in week twelve of the

pilot scale operation to eradicate this problem. Seventy percent of the granular sludge had been lost by week fourteen. The reactor was reseeded with 5 m³ of sludge. The addition of a polymer to the wastewater in combination with the Krofta stopped the flow of fibers to the UASB and the granular sludge stayed in the reactor.

After reseeded, the target loading rate of 18.5 kg total COD (COD_T)/m³ reactor·day with a COD_T removed over the UASB of 32% was achieved. This was an indication of the maintenance of active sludge.

The performance of the UASB was poor during the seventh to ninth months of operation. Colloidal material coagulated and formed a layer on the surface of the UASB. Small granules were incorporated into this layer and lost from the system as a result. This was attributed to the discharge of mill effluents with high concentrations (total suspended solids (TSS) = 1500 mg/L) of tiny wood fiber called "fines". Fines were discharged during a trial run of a new grade of CTMP at the beginning of the seventh month, December 1987. These fines were continuously discharged in the mill white-water at high concentrations. These fines were ineffectively removed with polymer additions. In addition, the sludge bed underwent a toxic shock believed to be due to the resin acids which saturate the fines. Regeneration of the sludge bed was not possible and as a result, the sludge bed had to be replaced with active granular sludge in the tenth month.

During the subsequent three months, testing indicated 70 to 80% fines removal was possible with a combination of 5 ppm Berol non-ionic polymer with 20 ppm Berol resin. Reactor performance in the tenth to the thirteenth month of operation was stable. Polymer additions to reduce the concentration of fines in the effluent eliminated the toxicity effects on the sludge and the quality of the sludge was maintained without any significant losses.

To date little technical information has been released regarding treatment process details or the nature and extent of operational problems encountered during TMP/CTMP wastewater treatment. Despite the fact that the two-stage anaerobic concept has already been implemented in full scale operation, there is still much research needed to fully understand and describe system operation. For the optimization of full scale treatment plant operation it is important to explore the advantages of removing or neutralizing inhibitory compounds in the wastewater.

Therefore, the main purpose of this study was to examine how the integrity and activity of granular sludge can be maintained during two-stage anaerobic digestion of CTMP wastewater under practical conditions of operation. Specifically, the objective of this experimental study was to ascertain the source of the problems encountered at the Quesnel River pilot scale treatment plant by:

1. examining the partition of toxicity between soluble

(filtrate from 0.80 um filtration) and particulate constituents (fiber/fines > 0.45 um) in CTMP wastewater,

2. examining the performance, operational stability, and loading limitations of two-stage anaerobic digestion during treatment of CTMP wastewater with different fiber/fines contents in laboratory scale reactors, to investigate whether treating unaltered CTMP had any significant disadvantage over pretreated (fiber/fines removal) CTMP, and
3. determining experimentally the tolerance of biomass when exposed to CTMP wastewaters with different fiber/fines contents on a continuous basis by examining the effects of fiber/fines on reactor biomass activity and physical characteristics.

CHAPTER 2

LITERATURE REVIEW

2.1 QRP Plant History

The QRP is an unincorporated company jointly owned by Daishowa Canada Ltd. and the West Fraser Timber Company Ltd.. Daishowa is a Japanese company which produces mainly TMP for the Japanese market. West Fraser Timber is a Canadian company which produces CTMP for several international markets.

QRP started production of TMP in 1981. In 1983, the mill was converted to a bleached CTMP (BCTMP) operation, the first North American mill of this type. The mill operates as a dual process with bimonthly alternation between TMP and CTMP production. With a production capacity of 900 air dried metric tonne (ADMT)/d, QRP supplies the largest fraction of the world's BCTMP.

2.1.2 The QRP TMP/CTMP Process

The wood chip species used depends on the type and grade of pulp to be produced. TMP is produced from a combination of softwood chips: 50% spruce, 40% pine, and 10% balsam. There are three combinations of wood chips used to produce the various grades of CTMP. These include:

a) the combination used for TMP, b) 50% aspen plus 50% of the above mixture, and c) 75% aspen plus 25% of the aforementioned mixture.

The chips are delivered by truck from nearby sawmills and stored outside awaiting delivery by conveyor to the pulping process. The chips are mechanically screened to remove oversized chips, then washed and drained. The chip washers use water to remove foreign material (sand, grit, and tramp metal) by the difference in specific gravity and size. The removal of foreign matter is essential to the operation of the steel plates in the refiners which operate at high speed and a fraction of a millimeter apart (McCubbin, 1983).

In the TMP process, chips are softened by steam under pressure in a presteaming vessel without chemicals. This thermal treatment results in a high strength pulp by softening the lignin to reduce damage of the long fiber component during mechanical defibration (NovaTec Consultants Inc., 1987). The chips proceed to be defibered under pressure in a disc refiner. The pulp is further refined in a second stage refiner.

The CTMP process is similar to the TMP process except that wood chips in the CTMP process are impregnated with a 1 to 5% sodium sulfite solution under alkaline conditions during the steaming step. The sulfite impregnation further softens the wood chips, thereby improving the quality of the final pulp by increasing the flexibility of the long fiber

fraction over that produced by the TMP process (Cornacchio and Hall, 1988). The improved quality and the increase in overall strength makes the CTMP more suitable for products typically made from chemical pulps such as tissue and disposable diapers (NovaTec Consultants Inc., 1987). The pulp is then refined in two stages followed by screening, washing, and cleaning.

TMP is brightened using a Borol sodium hydrosulfite process which leaves some residual sulfite in the wastewater (de Vegt, 1988).

The bleaching of CTMPs is a brightening process (Cornacchio and Hall, 1988). CTMP is bleached using a MoDo-Chemetics medium consistency hydrogen peroxide system (de Vegt, 1988). Hydrogen peroxide is used when a high brightness is required and, unlike the chlorine-based bleaches used for chemical pulps, the lignin is retained within the pulp (Cornacchio and Hall, 1988). This process leaves 50 - 100 mg/L of residual hydrogen peroxide in the mill white water system (de Vegt, 1988). Diethylene-triamine-pentaacetic acid (DTPA), a chelating agent, is added during the bleaching stage to bind metal ions that accelerate the catalytic decomposition of the brightening agents (NovaTec Consultants Inc., 1987).

Thune presses dewater the bleached pulp which is subsequently dried in a gas fired flash dryer. The dry pulp is pressed and bailed in preparation for shipment. A process schematic is provided in Figure 2.1.

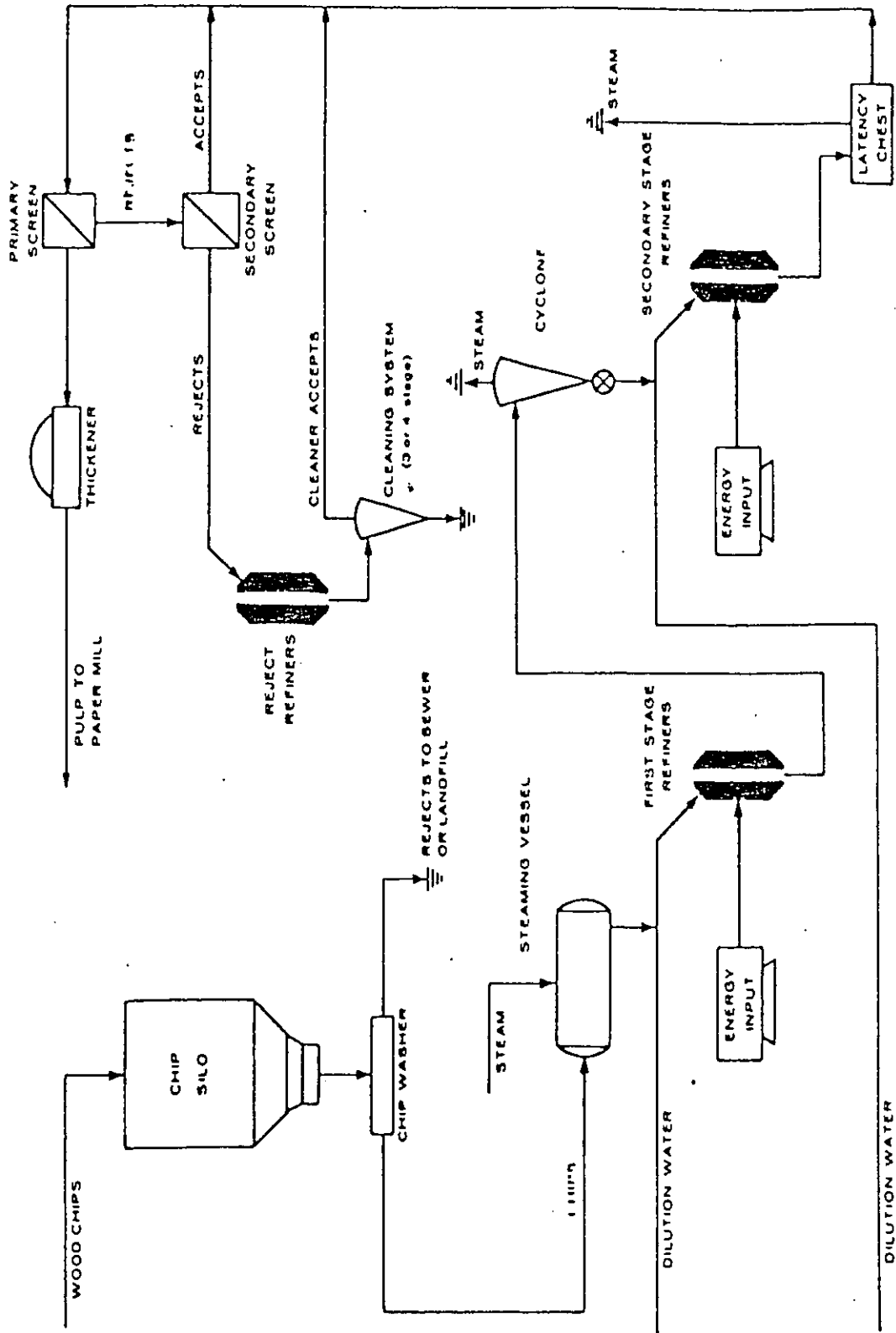


Figure 2.1 TMP/CTMP process (McCubbin, 1983)

2.2 General Wastewater Characteristics

Water is used in the CTMP process for washing and pretreating the wood chips, and for cooking and refining of the pulp. A portion of the water is recirculated and the remainder is discharged from the mill. Although the use of recirculated water results in a decrease in the total mill water demand, the effluent has higher contaminant concentrations and has a higher temperature than without recirculation.

Thermal and chemical pretreatment during the CTMP process causes extensive solubilization of biodegradable wood components and extractives such as resin and fatty acids, lignins, and tannins. The degree of solubilization is a function of temperature, sulfite loading, impregnation time, and bleaching.

The composition of organics in CTMP effluents has not been well documented. The composition has been measured to be: carbohydrates (10 - 15%), organic acids (35 - 45%), and lignins (30 - 40%) (Pichon *et al.*, 1986).

The concentration of BOD₅ and COD in CTMP effluents are considerably higher (up to 45%) than TMP. CTMP effluents contain a larger fraction of high molecular weight non-biodegradable components than TMP effluents (Urbantas and Macewen, 1985). The BOD₅/COD ratios of CTMP effluents range from 0.30 to 0.45. Biological treatment will be capable of removing 55 to 70% of the organic material (COD) in CTMP effluents (Hall and Cornacchio, 1988). Anaerobic bacteria

are unable to degrade the high molecular weight organics. This is the main reason for the moderate COD reduction rates by anaerobic treatment (Eriksson, 1985). Aerobic post-treatment is able to degrade the high molecular weight organics.

The bleaching of CTMP with hydrogen peroxide (H_2O_2) impacts the amount of BOD_5 produced. Hydrogen peroxide may cause the production of more low molecular weight lignins and volatile organic acids resulting in a contribution of 25 to 50% of the total BOD_5 , depending on the size of the charge (Hall and Cornacchio, 1988). Residual H_2O_2 and DTPA are present in the effluent water. The residual concentrations are a function of the process and the size of the charges used.

Sodium sulfite used in the CTMP process results in sulfite contamination of CTMP wastewater. Elevated sulfite concentrations of 100 - 300 mg/L and sodium concentrations of 700 - 1600 mg/L, as compared to less than 100 mg/L of each in TMP effluents, have been reported (NovaTec Consultants Inc., 1987).

CTMP wastewater resin acid concentrations have been found to be twice that of TMP as a result of the chemical pretreatment. TMP resin acids are negligible (2 - 21 mg/L), whereas CTMP resin acids are considerably higher (26 - 65 mg/L) (NovaTec Consultants Inc., 1987). Dehydroabietic acid is typically the dominant resin acid present.

2.2.1 QRP Wastewater Characteristics

The characteristics of the QRP CTMP and TMP discharges are presented in the following table.

Table 2.1 QRP wastewater characteristics

Parameter	Unit	TMP	CTMP
Production	ADMT/d	900	900
Total Flow	m ³ /d	15000	18250
Water Use	m ³ /ALMT	17	20
COD	mg/L	4200	7800
BOD ₅	mg/L	2000	3500
BOD ₅ /COD		0.48	0.45
TSS	mg/L	300	400
VFA	mg/L	4	20
H ₂ O ₂	mg/L	0	50
S	mg/L	200	300
Temperature	°C	35-40	35-40
pH		5-6	7-8
Resin Acids	mg/L	40-125	40-250
DTPA	mg/L	30	54

ADMT - Air Dried Metric Tonne

Source: de Vegt, 1988. 1987 wastewater characteristics and expected characteristics after October 1988 expansion.

2.3 British Columbia Emission Standards

There have been no standards set dealing specifically with CTMP mill wastewaters. CTMP mill wastewaters are subject to regulations set for other mill types (Turk, 1988). In British Columbia, CTMP mill discharges must meet the mechanical mill regulations for new mills (Level A). These emission guidelines are listed in Table 2.2.

Table 2.2 British Columbia emission standards for CTMP mills

Parameter	Unit	
Fish Toxicity*	96 hr LC ₅₀	100%
BOD ₅	kg/ADT	7.5
TSS	kg/ADT	10.0
-After primary		8.5
Dissolved Oxygen	mg/L	2.0
pH		6.5-8.0
Temperature	°C	35

* Fish toxicity based on 80% survival in 65% effluent concentration.

2.4 Effluent Treatment

CTMP and TMP discharges are concentrated effluents due to the process and internal recycling of water. These wastewaters are most amenable to anaerobic treatment. Methods for treatment of CTMP discharges typically combine anaerobic pretreatment for the removal of organics followed by detoxification by aerobic polishing.

Anaerobic treatment is unable to degrade high molecular weight organics. For example, lignins of molecular mass higher than 850 are not degraded anaerobically. Although high BOD₅ reductions of up to 95% are attainable with anaerobic treatment, an associated moderate COD reduction of only 50 to 60% is achievable (Eriksson, 1985). Anaerobic treatment by itself is insufficient to detoxify a waste and meet stringent effluent standards therefore, post-treatment by aerobic processes is required. An anaerobic/aerobic treatment process flow diagram is illustrated in Figure 2.2.

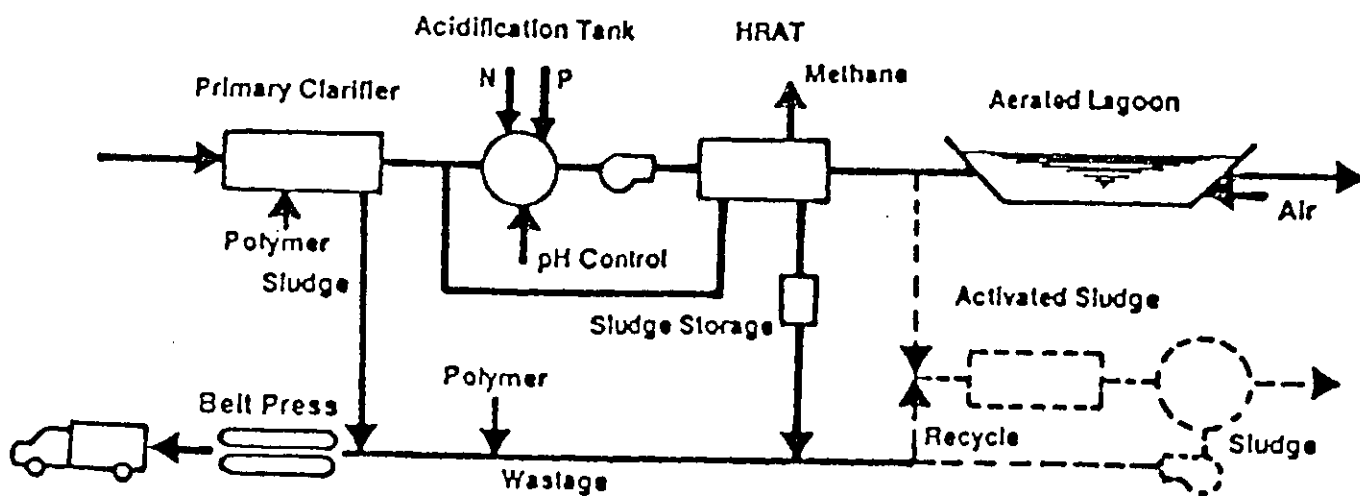


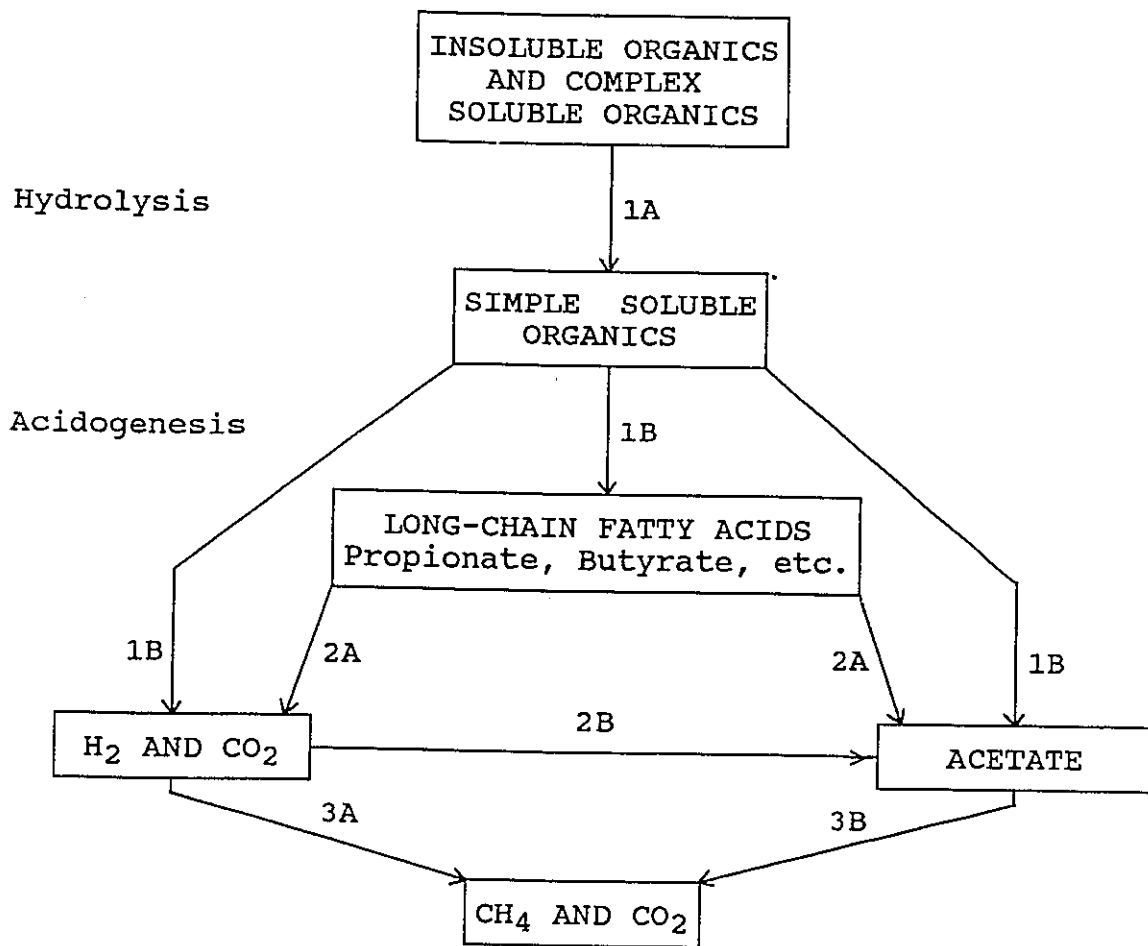
Figure 2.2 High rate anaerobic treatment/aerobic polishing process flow diagram (Turk, 1988)

2.5 Two-Stage Anaerobic Digestion: Concept

The anaerobic digestion of complex organics to carbon dioxide and methane is described as a three-step process involving three major metabolic groups of bacteria: acidogens, acetogens, and methanogens (McInerney and Bryant, 1980; Speece, 1983; Kirsop, 1984; Zinder, 1984). The process scheme is illustrated in Figure 2.3.

The first step, 1A and 1B, involves a community of fermentative bacteria that liquefy insoluble organics and hydrolyze complex organics into simpler molecules. The reactions responsible for solubilization and size reduction are hydrolytic and are catalyzed by enzymes which have been released extracellularly to the medium. The primary soluble substrate polymers: polysaccharides, proteins, and lipids are subsequently fermented by bacteria to amino acids, peptides and sugars. The rate of hydrolysis depends on the nature of the waste. The hydrolysis of anaerobic digestion is rate limiting in wastewaters containing primarily suspended material or other slowly hydrolyzed compounds (O'Rourke, 1968; McInerney and Bryant, 1980; Kennedy and Van den Berg, 1982).

In the second step, 2A and 2B, known as acidogenesis or acid fermentation, the simple soluble organic molecules are converted by strict and facultative anaerobic bacteria to long-chain fatty acids, hydrogen (H_2) and carbon dioxide (CO_2). The formation of the products by acidogenesis is affected by the partial pressure of H_2 (McInerney and



Stage 1

- 1A. Fermentative bacteria, hydrolysis
- 1B. Fermentative acid-producing bacteria, acidogenesis
- 2A. Hydrogen-producing acetogenic bacteria, acetogenesis
- 2B. Hydrogen-consuming acetogenic bacteria, acetogenesis

Stage 2

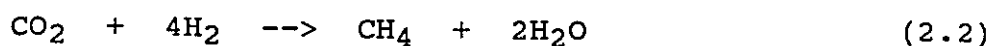
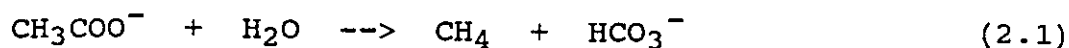
- 3A. CO₂-reducing methanogens; methanogenesis
- 3B. Acetoclastic methanogens; methanogenesis

Figure 2.3 Multistep nature of anaerobic operations (Grady and Lim, 1980; Zinder, 1984).

Bryant, 1980). When the H_2 partial pressure is kept below 10^{-3} atm by the hydrogen consuming methanogens, then acidogenesis results in acetate, H_2 and CO_2 . If, however the H_2 partial pressure exceeds this level, more reduced compounds (propionate, butyrate, ethanol, lactate, etc.) are formed. These products are converted to acetate by a separate group of bacteria, the acetogens.

Finally, in the third step, 3A and 3B, methane (CH_4) fermentation or methanogenesis, methanogenic bacteria convert the intermediate products to CH_4 and CO_2 . A small amount of the organic material is consumed for microbial growth. About 90% of the available chemical energy in the organic material is retained in the methane produced (McInerney and Bryant; 1979). Theoretically, anaerobic biodegradation of carbohydrates produces a gas containing equal quantities of carbon dioxide and methane. Dissolution of carbon dioxide in the liquid results in a methane yield accounting for 60 to 70% of the gas generated (Webb, 1983).

Seventy percent of the CH_4 produced originates from acetate and 30% is formed via the reduction of CO_2 by H_2 (Mah *et al.*, 1977; Verstraete *et al.*, 1981) according to the following equations (Daniels *et al.*, 1984; Kirsop, 1984):



The rate limiting step during anaerobic biodegradation for most soluble substrates is the final step of methane production. Hydrolysis is rate limiting for more complex mixtures such as pulp and paper wastes which contain high concentrations of cellulose and lignin.

Two dominant groups of facultative anaerobic and strict anaerobic microorganisms are responsible for the conversion of complex substrates to methane. Since the anaerobic digestion of complex substrates is mediated by two distinct microbial groups, and since these two groups are believed to differ significantly with respect to physiology, nutritional requirements, growth and metabolic characteristics, environmental optima, and sensitivity to environmental stresses, culturing of these organisms in isolated, optimized environments should result in improved overall process efficiency, reaction rate, stability and operational control (Pohland and Ghosh, 1971; Ghosh and Klass, 1978). A treatment process that employs physical separation of these two groups is referred to as the two-stage concept.

2.6 Two-Stage Anaerobic Digestors

Originally, it was proposed that stage separation might be accomplished by:

1. the dialysis technique (Hammer and Borchardt, 1969; Schaumburg and Kirsch, 1966), or
2. selected inhibition of each group of organisms through the addition of inhibitors such as oxygen, nitrates,

- sulfates, metals, etc. (Borchardt, 1967), or
3. potential poisoning (Borchardt, 1967), or
 4. kinetic control (Pohland and Ghosh, 1971).

Operational difficulties with dialysis membranes and the uncertainties associated with the determination of inhibitor concentrations and potential poisoning made kinetic control the more attractive choice (Pohland and Ghosh, 1971; Ghosh and Klass, 1978).

In conventional high-rate anaerobic digestors, two physiologically different digesting organisms are harbored under the same environment. When the substrate loading rate is increased above an upper limit and the hydraulic retention time is decreased below a critical level, the system will experience retardation and eventually fail. Failure occurs because higher organic loading (*i.e.* higher feed COD concentrations) and shorter hydraulic retention time (HRT) favour the enrichment of acidogenic organisms by encouraging volatile acid production and preclude the establishment of the methane-forming bacteria. Conditions promoting efficient substrate-to-acids conversion are not conducive to stable and efficient acids-to-methane fermentation (Ghosh *et al.*, 1985). The two-stage process configuration employs separate reactors for acidification and methanogenesis, connected in series, allowing for optimization of both stages.

The two dominant cultures can be maintained in separate reactors by exerting kinetic control on each of the stages by operational adjustment of the dilution rates and recycle

ratios (Pohland and Ghosh, 1971). The objective is to set the dilution rate in the first reactor at a rate that is higher than the maximum specific growth rate of the methane formers and to achieve a high degree of conversion of substrate to volatile acids and other intermediates acceptable to the methane formers. If this objective is achieved, then it is possible to preclude the growth of the methane formers in the first reactor while impeding the growth of the acid formers in the second. Therefore, separate enrichment of non-methanogenic and methanogenic organisms is contingent upon proper selection of dilution rate (or detention time) and organism recycle ratios for the two digestors. The optimum dilution rate and organism recycle ratios are a function of the waste being treated and therefore are obtainable through experimentation. Generally, recirculation ratios are in the 2 - 4 : 1 range (Paques Lavalin, 1985).

The HRT in the non-methanogenic digester should be selected to promote conversion of the substrates to volatiles acids at the maximum rate and at the same time be shorter than the critical detention time of the methane formers to promote the selective enrichment of acidogenic metabolism in a separate environment. The optimum detention time for acidogenic fermentation is much shorter than that for methane fermentation in most cases (Ghosh and Klass, 1978). The low retention time in the acid digester prevents the establishment of the methanogens because the methane formers grow at a much slower rate than acid formers (Ghosh *et al.*,

1975). These factors combined allow process operation at higher loading rates and shorter HRT; operating conditions under which the conventional digestion process often fails. The same or higher methane yields and COD stabilization efficiencies can be attained in a two-stage system over a conventional high-rate process treating the same waste (Ghosh *et al.*, 1985).

In summary, in the first stage, a preacidification tank (PA), hydrolysis, liquefaction, and acidification occur. Stabilization of the waste COD and methanation of the effluents from the first stage acid digester take place in the second stage, an UASB reactor (Pohland and Ghosh, 1971; Ghosh and Klass, 1978). The physical/biological division of the treatment process into two stages, where Stage 1 and Stage 2 are the PA tank and the UASB digestion reactor respectively, is illustrated in Figure 2.3.

2.6.1 Preacidification Tank

The PA tank is a complete mix reactor with a low solids retention time. This provides an enhanced environment for the development of a community of acidogenic bacteria. The presence of a PA tank offers several advantages:

1. improved process control in terms of pH, temperature and nutrients;
2. degradation of reactive and unstable compounds that are toxic to the methanogens (such as hydrogen peroxide and oxygen);

3. hydrolysis of some complex organics into volatile fatty acids;
4. faster recovery from toxic effects due to dilution by internal recycle;
5. some buffering ability with respect to hydraulic and organic shocks; and
6. buffering of temperature extremes (Turk, 1988).

2.6.2 Upflow Anaerobic Sludge Blanket Reactor

The UASB reactor was developed by Lettinga and co-workers in the Netherlands during the early seventies (Lettinga et al., 1980a). The reactor consists of a dense biological blanket of granulated sludge at the reactor base with a gas-solids separator at the reactor top. The principal feature of the UASB reactor is the formation of a highly active, dense and pelletized consortia, or granules with superior settling properties which are easily separated from the effluent and retained in the reactor (Guiot et al., 1985). The anaerobic sludge will obtain and maintain superior settling characteristics if chemical and physical conditions favorable to sludge flocculation and to the maintenance of a well flocculated sludge are provided.

The sludge blanket (bed) is considered as a separate fluid phase with its own specific characteristics. A well established sludge blanket forms a stable phase capable of withstanding high mixing forces. The redispersion of the sludge in the liquid phase therefore may require a significant amount of mixing energy. A well granulated sludge can

maintain a stable, although expanded, bed at superficial velocities as high as 4 m/h.

Washout of discrete sludge particles (flocs) released from the sludge blanket is minimized by the presence of the gas-solids separator, a quiescent zone within the reactor, where flocculation and settling of sludge particles is permitted and gas is trapped and vented from the system (Lettinga *et al.*, 1980a). This configuration enables the system to retain biomass independently of the hydraulic retention time. This allows for the treatment of both low and high strength wastes at high loading rates and low hydraulic retention times.

2.6.3 Granulation

The concept of the UASB process lies in the ability of anaerobic sludge to flocculate and form a granular bed. Formation of sludge granules (pelletization) has been observed in many studies using UASB reactors (Lettinga *et al.*, 1980a, 1980b). In their work, Lettinga and co-workers observed the settleability of granular sludge (sludge volume index (SVI) = 10 - 20 mL/g) was better in comparison to flocculated sludge (SVI = 20 - 40 mL/g). The improved sludge settleability resulted in much greater solids retention times of several hundred days with very short hydraulic retention times, ranging from several hours to several days (Lettinga *et al.*, 1980a).

Anaerobic experiments with non-granular sludge treating volatile fatty acid (VFA) feed were conducted by Hulshoff Pol and co-workers (1982) to examine the pelletization process. Three phases of pelletization were distinguished in their research. These were:

Phase 1 (0 to 1 g COD/g VSS·d)

During this phase expansion of the sludge bed occurs due to increasing surface loads and the associated production of gas. This induces the growth of filamentous organisms with poor settleability.

Phase 2 (1 to 5 g COD/g VSS·d)

Washout of biomass occurs as the space load is increased. Excessive bed expansion occurs and flocculant sludge appears present. Towards the end of this phase, the formation of granules commences and a concomitant increase in specific activity and a decrease in the biomass washout rate occurs.

Phase 3 (5 to 9 g COD/g VSS·d)

A point is reached where the washout rate is less than the yield of newly developed sludge pellets. This is the beginning of Phase 3. At this time, space loads can be increased. The maximum space load is dictated by the presence of granular sludge.

Similar phases of the pelletization process are visible when treating alternate feed solutions. The start-up loading

rates during the three phases are a function of the feed characteristics.

Studies concluded biological (type of seed sludge), engineering (operating conditions applied during start-up and continuous operation), and environmental (process conditions) factors combined contribute to the pelletization process (Hulshoff Pol, *et al.*, 1982).

The type of seed sludge accounts for the specific activity and its settleability. The seed sludge must be viable and active. Hulshoff Pol *et al.* (1982) suggested a prerequisite to granulation is an adapted sludge with a specific activity above 0.6 g COD/g VSS·d.

Operating conditions applied during start-up account for the initial amount of seed sludge used as well as the procedure followed in increasing the loading rate. A procedure to follow during start-up to acclimate a granular sludge quickly to the wastewater and enhance the granulation process is outlined below (Lettinga *et al.*, 1980a, 1983).

1. The initial seed sludge concentration should be 10 to 20 g VSS/L of reactor.
2. The initial organic load should be below 0.05 to 0.10 g COD/g VSS·d.
3. The loading rate of the reactor should not be increased, unless all volatiles acids present or formed are effectively decomposed (*i.e.* less than 100 mg/L).
4. Permit the washout of poorly settling voluminous sludge.
5. Retain the heavy part of the sludge.

Separation of the acidification stage from the UASB allows the application of higher COD's without disrupting the granules (Lettinga et al., 1980a).

Environmental conditions such as the availability of nutrients, operating temperature, pH, and the type of wastewater play a dominant role in the pelletization process.

The flocculation of sludge has been shown to be dependant upon the occurrence of mono- and divalent cations (e.g. barium; Ba^{2+} , calcium; Ca^{2+} , magnesium; Mg^{2+} , ammonium; NH_4^+) (Lettinga et al., 1980a; Hulshoff Pol et al., 1982; Mahoney, 1984). Ions were shown to have a positive effect on the flocculation ability of anaerobic sludge, presumably because they improve the mechanical bond between bacterial cells and flocs. Hulshoff Pol et al. (1982) conducted studies to determine the effect of Ca^{2+} and NH_4^+ on the granulation process. At low concentrations, Ca^{2+} enhanced the formation of pellets, however, at high concentrations, sludge retention worsened. Concentrations of NH_4^+ at 400 mg/L resulted in less washout than at 40 mg/L. When the concentration of NH_4^+ was 1000 mg/L, the third phase of the pelletization process did not occur.

The existence of trace metals has also been shown to have an effect on the granulation of sludge. Murray and Van den Berg (1981) observed an appropriate mixture of trace metal elements (e.g. nickel; Ni, cobalt; Co, molybdenum; Mo) improved the growth of methanogens and the specific activity

of biofilm. Guiot et al. (1985) observed trace metal supplementation gives rise to preferential growth of acetoclastic methanogens, which results in the granulation process.

High concentrations of finely dispersed, non- or poorly flocculating matter has an adverse effect on sludge flocculation and therefore on the sludge retention in a reactor. This material can be inorganic matter such as clay, organic matter such as acid-forming organisms either present in or formed from the feed. The absence of this material in the feed or continuous removal (washout) from the system is therefore beneficial to the granulation process (Lettinga et al., 1980a).

To ensure bacterial growth, contact between sludge and the feed solution must be maintained. This is achieved through mechanical agitation. Inefficient mass transfer from the feed solution to the sludge occurs if cracks and canals are formed in the sludge bed. Mechanical agitation prevents the formation of cracks and canals. In some cases, there is no need for a more intensive agitation than that brought about by gas production.

The factors affecting the rate of pelletization and the pellet size distribution include Brownian motion, laminar shear, and turbulent transport (Boyle et al., 1972). The configuration of the UASB reactor promotes pelletization as the result of the combination of these factors. Except through the action of bacterial growth in and on the sludge

flocs, the formation of granular sludge is presumably mainly driven by gravity compression forces. The sludge is exposed to varying forces of gravity compression (thickening) as a result of vertical mixing brought about by gas production. Gravity compression is dependent on the height of the sludge bed, sludge concentration, and place of the sludge in the reactor. Sludge thickening is stimulated by increasing the height of the sludge blanket and, within certain limits, also by increasing the stirring intensity.

Erosion also takes place in the sludge bed under the influence of friction forces to which the sludge particles are exposed, especially during high mixing intensities. Disintegration of sludge particles by erosion is kept at a minimum by allowing only a gentle and homogeneous agitation mode such as that resulting from the escape of produced gas or the upward flow of liquid (Lettinga *et al.*, 1980a).

The erosion and formation of granular sludge will be affected by the gas production rate. As gas moves through the sludge bed, the bubbles erode sludge pellets. Sludge particles are ejected from the bed along with the gas bubbles. The sludge particles are kept in suspension in the liquid bulk above the sludge blanket as a result of the agitation caused by the gas bubbles. When the suspension concentration increases, the reflocculation to larger more easily settling flocs gradually improves (Lettinga *et al.*, 1980a).

The effect of the hydraulic loading rate on the sludge retention is closely related to the agitation intensity in the sludge bed. When insufficient mixing of the bed is provided, the sludge bed will expand due to the hold-up of entrapped gas and sludge bulking can occur. Washout of reactor sludge is often associated with sludge bulking. The quantity of reactor sludge retained will decrease as a result. Also, improved agitation brought about by the vertical flow of liquid will result in a decrease of the hold-up of entrapped gas, prevent sludge bulking, and will improve the thickening of the sludge. Guiot *et al.* (1985) through experimentation determined a minimal liquid upflow velocity of 1 m/d is required to promote pelletization. In addition, at high organic loading rates, agitation caused by gas evolution will usually be sufficient to prevent an excessive buoying of sludge.

Sludge pellet formation and size enlargement proceeds slowly with anaerobic sludge, often requiring months of operation (Lettinga *et al.*, 1980a). The doubling time of methanogens varies from thirty minutes to several weeks (Hall *et al.*, 1986). However, the slow start-up periods can be accelerated by the seeding with a large quantity of biomass from a similar treatment process.

Due to the low biomass yield under anaerobic growth conditions, it is necessary to retain the active biomass in the reactor and prevent its washout. Slow loss of biomass over an extended period of time can be difficult to recog-

nize until process performance is severely affected. Pelletization and granular sludge build-up is a slow process that can be adversely affected by process upsets. Scum and floating mat accumulation at the top of the reactor can cause major loss of biomass leading to failure of the system, therefore, constant removal is required (Hall et al. 1986).

The physical characteristics of the granular sludge in UASB process accommodates fairly well to hydraulic and organic shock loads, and temperature fluctuations provided the pH remains well above 6.0 and that the sludge load applied is below the maximum specific COD removal rate of the sludge at the prevailing temperature in the digester. The loading rate on a reactor with granulated sludge can be four to five times higher than for non-granulated sludge (Habets and Knelissen, 1985).

The mechanisms underlying the pelletization process are not yet sufficiently understood. Also, granular sludge cannot be achieved with all types of waste (Van den Berg and Kennedy, 1983). Special attention should be given in this respect to the significance of optimum start-up and to the development of a granular type of sludge.

2.7 Process Environmental Conditions

2.7.1 Temperature

The predominant bacteriological species in a system is determined by the temperature at which the microbial habitat is kept because rates and limits of growth of a population are temperature dependent. Most microorganisms have an optimal growth rate over a fairly narrow temperature range. Microorganisms are grouped according to these rates and limits and are classified as follows: Psychrophiles (0 - 20°C), Mesophiles (20 - 45°C) and Thermophiles (45 - 65°C) (Zehnder *et al.*, 1981; Henze and Harremoes, 1983).

The majority of methanogenic bacteria are active in the mesophilic range (Zehnder *et al.*, 1981; Henze and Harremoes, 1983). Anaerobic treatment processes can be operated in the temperature range of 20 to 55°C, although, they are most commonly operated in the range of 30 to 35°C (Turk, 1988).

The two-stage anaerobic treatment system operates at 35°C. The temperature of raw CTMP and TMP wastewater exceeds 35°C, therefore cooling of raw wastewater is necessary to facilitate treatment.

2.7.2 pH

The optimal pH range for methanogens is between 6.0 and 8.0. Maintenance of a neutral pH in a digester provides an optimum condition for methanogenesis (*i.e.* methanogenesis

yields maximum methane production in this range) (Henze and Harremoes, 1983).

Acidogens are less sensitive to low pH levels than methanogens. The production of acids by acidogenesis tends to depress the pH of the liquid. The operating pH dictates the type of end product that is formed from acidogenesis. The optimum pH range for acidogens is 5.0 to 6.0 (Henze and Harremoes, 1983).

Two-stage anaerobic systems will establish their own operating pH and will remain stable at that pH during steady state conditions without pH adjustment (Turk, 1988).

Sodium hydroxide and sodium sulfite used in CTMP cooking results in effluents with high levels of sodium and neutral to alkaline pH's. Therefore, minimal caustic addition is required for pH control during anaerobic treatment because of the alkalinity level. The lower sodium levels associated with TMP effluents are usually inadequate for pH control in an anaerobic system (Beak Consultants Ltd., 1988).

2.7.3 Nutrients

Adequate (non-limiting) amounts of nutrients are required for biological processes to function properly. The nutrients are needed for microbial growth (*i.e.* the formation of new bacterial biomass). Different microbe strains require certain nutrients in varying quantities to supple-

ment their metabolism. Certain strains have been shown to require calcium, cobalt, nitrogen, phosphorus, magnesium, sodium, sulfide, trace metals such as cobalt, iron, molybdenum, nickel, selenium, and vitamins (Henze and Harremoes, 1983; Kirsop, 1984). Nitrogen (N) and phosphorus (P) are the major nutrients required for bacterial growth during biological treatment (Cornacchio and Hall, 1988).

Pulp and paper wastes are deficient in nitrogen and phosphorus, and supplemental addition of these two elements is practiced as part of biological treatment.

2.7.4 Inhibitors/Toxicity

Toxic compounds are commonly removed from wastewater before biological treatment to avoid process deterioration due to biological inhibition. This is feasible if the concentration of toxins is constant. If the concentration of toxins in the wastewater is sporadic, the treatment process can be impaired. Toxins can slow down microorganism substrate conversion rates. Prolonged exposure to toxins can reduce microorganisms' growth rates and change the microbial population (Lewandowski, 1987). This can render a biological system incapable of sufficiently metabolizing the wastewater.

The following constituents have been identified as being inhibitory to anaerobic bacteria: hydrogen peroxide (Morris, 1976) which is used as a bleaching agent; DTPA, a

strong chelating agent used for stabilizing hydrogen peroxide (Wilkinson, 1967; Beak Consultants Ltd., 1988); wood extractives (Mueller et al., 1976; Walden and Howard, 1981), especially resin acids; tannins and lignins; sulfate and other oxidized sulphur compounds which are reduced anaerobically to sulfide, a substance very toxic to methanogens (Turk, 1988). The toxicity of these compounds to anaerobic bacteria is described in the following sections.

2.7.4.1 Hydrogen Peroxide

Hydrogen peroxide has long been known to be toxic to bacteria (Morris, 1976) and is even used for sterilization and disinfection (Fraser, 1986). It is also known that obligate anaerobic bacteria are more sensitive to H_2O_2 than are aerobes and facultative anaerobes (Morris, 1976). Aerobic and facultative anaerobic bacteria generally possess the enzyme catalase that catalyzes the decomposition of H_2O_2 to water and oxygen, while obligate anaerobes (*i.e.* methanogens) usually do not (McCord et al., 1971). The reaction is presented in Equation 2.3 (Welander and Andersson, 1988).



As a result, obligate anaerobic methanogens are very sensitive to H_2O_2 (Welander and Hansson, 1983).

To avoid process failure when treating wastewater containing H_2O_2 , the peroxide should be removed before treating the wastewater in a methane producing reactor. The use of the two-stage reactor prevents the contact between the toxic compounds, in this case H_2O_2 , and the methanogenic bacteria. The decomposition of peroxide in an acidogenic reactor is mainly caused by the biocatalytic action of facultative anaerobes and by chemical reactions between peroxide and reduced compounds (Welander and Andersson, 1985). Welander and Hansson (1983) showed that H_2O_2 can be instantly removed from a CTMP wastewater by means of degradation in the acidogenic stage of a two-stage process. Their experiments showed that it is possible to remove H_2O_2 from the wastewater in an acidogenic reactor up to a concentration of 200 mg/L. This was confirmed by H_2O_2 degradation studies conducted at the University of British Columbia Chemical Engineering Department (Turk, 1988).

The redox potential in the reactor is used for control of the hydrogen peroxide destruction. Incomplete removal of hydrogen peroxide results in a rapid increase of the redox potential, which is normally low (about 200 mV) (Welander and Andersson, 1988).

The highest peroxide concentration that can be removed by the acidogenic stage without disturbance of the microflora depends on the operation conditions (e.g. pH and temperature). The reactor should be run at a pH between 6.5

and 8.0 and a temperature about 37°C to achieve the best results (Welander and Andersson, 1988).

2.7.4.2 DTPA

DTPA is added to the pulp during the bleaching stage to bind metal ions that accelerate the catalytic decomposition of the brightening agents. After bleaching there is residual DTPA in the wastewater. Metal cations are required for anaerobic growth and granule formation. The binding of these metal cations by DTPA in the wastewater from the CTMP and TMP process may cause toxicity to anaerobic bacteria (Welander et al., 1988).

Studies conducted by Welander and Wilhelmsson (1985) on methane formation by a methanol enrichment culture showed the inhibitory concentration of a chelating agent depended on the concentration of non-alkali metal ions present. Also, the higher the affinity of a certain chelating agent for metal ions, the lower the concentration level at which it became inhibitory.

Kennedy and co-workers (1987) conducted continuous experiments to examine the acute and chronic exposure of anaerobic biomass to DTPA. Long term exposure to DTPA resulted in anaerobic inhibition. This effect can be controlled or reversed by adding metals to the wastewater, such as in a ferric chloride (FeCl_3) solution.

DTPA has a preference for chelating Fe^{3+} molecules. It is possible that excess amounts of iron will bind the DTPA and release essential micronutrients needed by the anaerobic bacteria (Beak Consultants Ltd., 1988; Kennedy et al., 1987; Welander et al., 1988). Two moles of Fe^{3+} are required to neutralize 1 mole of DTPA (de Vegt, 1989).

2.7.4.3 Resin Acids

Resin acids are important constituents of a number of wood processing wastewaters. The concentration in a wastewater is a function of the wood species used and the pulping process. Resin acids have been measured in CTMP wastewaters in varying concentrations. The survey conducted by Cornacchio and Hall (1988) on Canadian mills reported resin acid concentrations ranged from 2 - 21 mg/L in TMP effluents, and from 26 to 65 mg/L in CTMP effluents. The high levels of extractives in CTMP effluents are suspected of being toxic. The high resin acid levels in CTMP effluent make this wastewater one of the most toxic of all pulp and paper industry liquid discharges.

Little is known about the fate of resin acids and their toxicity to microorganisms in biological treatment processes (McFarlane and Clark, 1988). The long term effects of very high resin acid levels are not known although, studies conducted by Vuoriranta et al. (1985) suggests that resin acids can be anaerobically degraded. McFarlane and Clark (1988)

using UASB technology demonstrated that some resin acids, in particular abietic acid, can be metabolized anaerobically. Dehydroabietic acid, however was not metabolized. Laboratory scale two-stage anaerobic systems have good potential for CTMP treatment but the recalcitrance of dehydroabietic acid in these reactors is a cause for concern (McFarlane and O'Kelly, 1988). The potential impact of resin acids in CTMP wastewaters requires further investigation.

2.7.4.4 Tannins and Lignins

Tannins and lignins account for about half of the aqueous extractable COD of tree bark. The principal methanogenic toxins of bark soluble matter have been identified as the tannins (Maat *et al.*, 1987; Field *et al.*, 1988). Information pertaining to the toxicity of tannins and lignins to anaerobes is limited. Further research is required.

2.7.4.5 Sulfate and Sulfide

Methanogenesis is inhibited by sulphur in two ways. Firstly, sulfide acts as a direct inhibitor of methanogenic activity. Under anaerobic treatment conditions, sulfite and sulfate may undergo biological reduction to hydrogen sulfide. Hydrogen sulfide produced during anaerobic treatment is volatile and will be stripped out of the treated effluent

by the biogas produced. However, if more than 200 mg as S/L of sulfide remains, inhibition of the anaerobic bacteria may result (Webb, 1983; Springer, 1986; Cornacchio and Hall, 1988). The tolerance of anaerobic bacteria to hydrogen sulfide up to this level illustrates that methanogens can acclimate to hydrogen sulfide (Welandar and Andersson, 1985).

Secondly, sulfate reducing bacteria compete directly with the methane forming bacteria for the hydrogen ion. This results in a reduction of methane yield (Turk, 1988). Vuoriranta *et al.* 1985 conducted batch tests using TMP white water spiked with sulfate (100 mg) inhibited biogas production from 65 mL to 15 mL. Lag times to reach these levels were longer at higher doses.

Methods for preventing the inhibition of methanogenesis by high sulphur levels in wastewater have been developed. Hydrogen sulfide toxicity is controllable in anaerobic reactors by precipitation with heavy metals, continuous gas scrubbing, and pH control at values of 8.0 or above to reduce free hydrogen sulfide (the toxic constituent) (Webb, 1983; Ferguson and Benjamin, 1986; Ruffer and Boeck, 1986; Hall and Cornacchio, 1988).

2.8 QRP Pilot Treatment Facility

The wastewater from the Quesnel River Pulp Company mill in central British Columbia (Chapter 1) consists of

approximately 40% clarifier effluent and 60% white water. The pilot facility to treat this wastewater, illustrated in Figure 2.4, consists of the following components:

Inclined Screen Box/Krofta

During the first eleven weeks of pilot plant operation, the combined wastewater was screened on an inclined screen where large solids, wood chips, were removed before entering the PA tank. In week twelve a Krofta air flotation clarifier replaced the inclined screen to remove wood fibers from the wastewater. The Krofta was a pilot scale unit, 1.25 m in diameter with 0.50 m effective water depth.

PA Tank

The PA tank is a fiberglass insulated tank, 2.4 m in diameter and 4.8 m high (22.0 m³). An automated chemical dosing system provides the addition of caustic (50% NaOH) for pH control and nutrients (Aqua Ammonia, 60% NH₄OH; Phosphoric Acid, 57% H₃PO₄) to this tank.

Caustic was added to the TMP wastewater (pH 5.0 - 6.0) to keep the pH in the UASB above 6.0. The CTMP wastewater had a pH of 7.5 - 8.0 therefore, no caustic addition was necessary.

Nitrogen and phosphorus supplementation was achieved by adding NH₄OH (24% N) and H₃PO₄ (57% P) in the ratio of COD:N:P = 350:5:1.

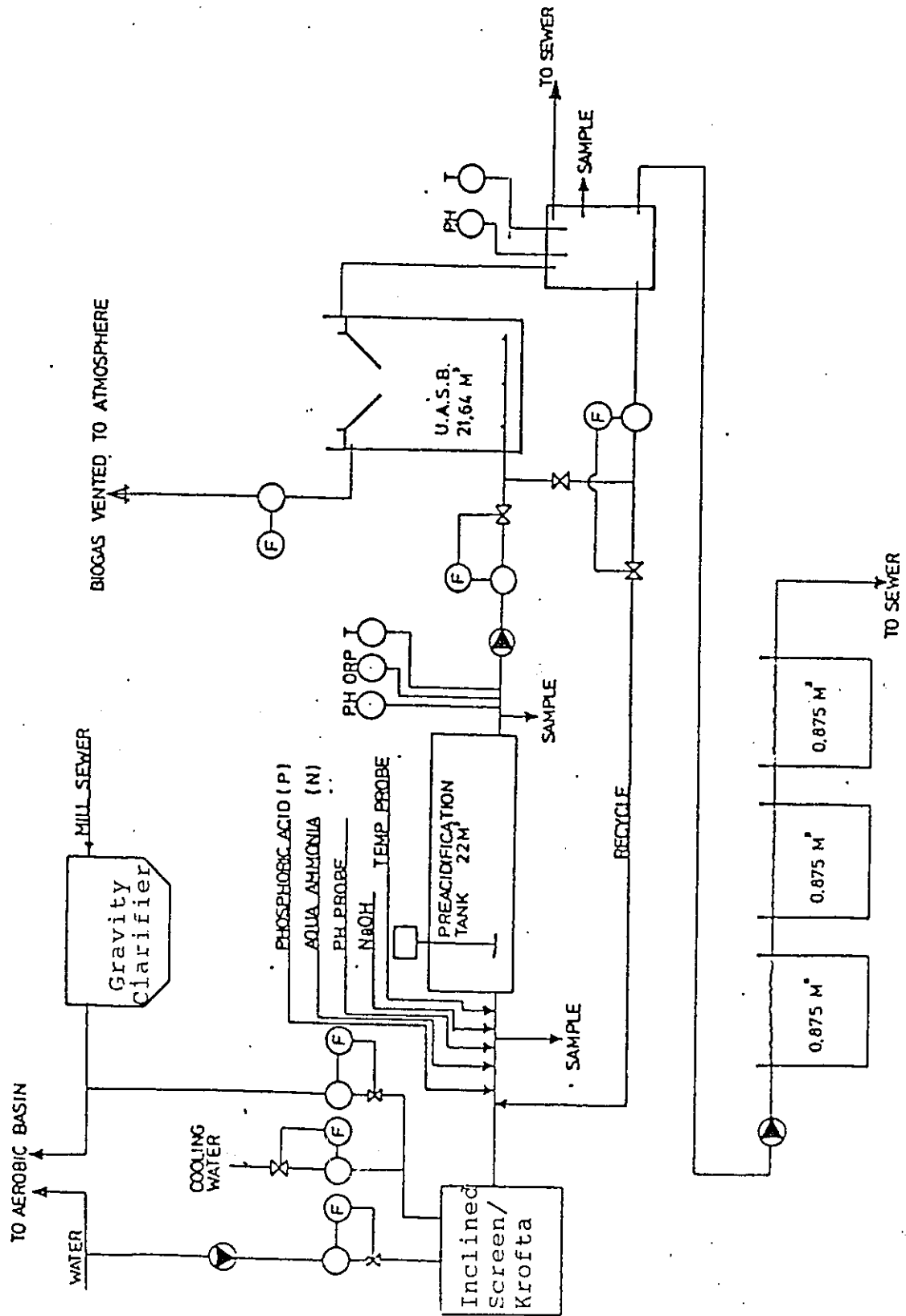


Figure 2.4 QRP pilot treatment facility flow schematic (de Vegt, 1988)

Biopaq UASB Reactor

The UASB reactor is a fiberglass insulated tank, 5.0 m in diameter and 4.5 m high (21.6 m³). A schematic of the Biopaq UASB reactor is illustrated in Figure 2.5. The flow distribution network, consisting of four ports, is located at the base of the reactor. This network was designed to distribute the flow evenly throughout the reactor to prevent short-circuiting. The reactor is equipped with eight sampling ports on its side. The UASB incorporates the patented Biopaq HDPE 3-phase settler at the top of the reactor to separate the sludge, biogas, and effluent. The reactor was seeded with 7 m³ of granular anaerobic sludge from an UASB reactor treating recycled paper mill wastewater.

Aerobic Pilot Plant

A fraction of the UASB effluent was pumped into an aerobic pilot plant which simulates an aerobic lagoon. It consists of three circular cells connected in series. Each cell has an hydraulic volume of 0.875 m³. The cells are completely mixed by bottom mounted aerators. The air flow ranged from 10 to 100 LPM for each cell to maintain an oxygen concentration of 2 - 3 mg O₂/L. The pH was measured but not controlled. The water temperature in the aerobic pilot plant was maintained at 31 ± 2°C. The design hydraulic retention time in the aerobic system was 2.2 days.

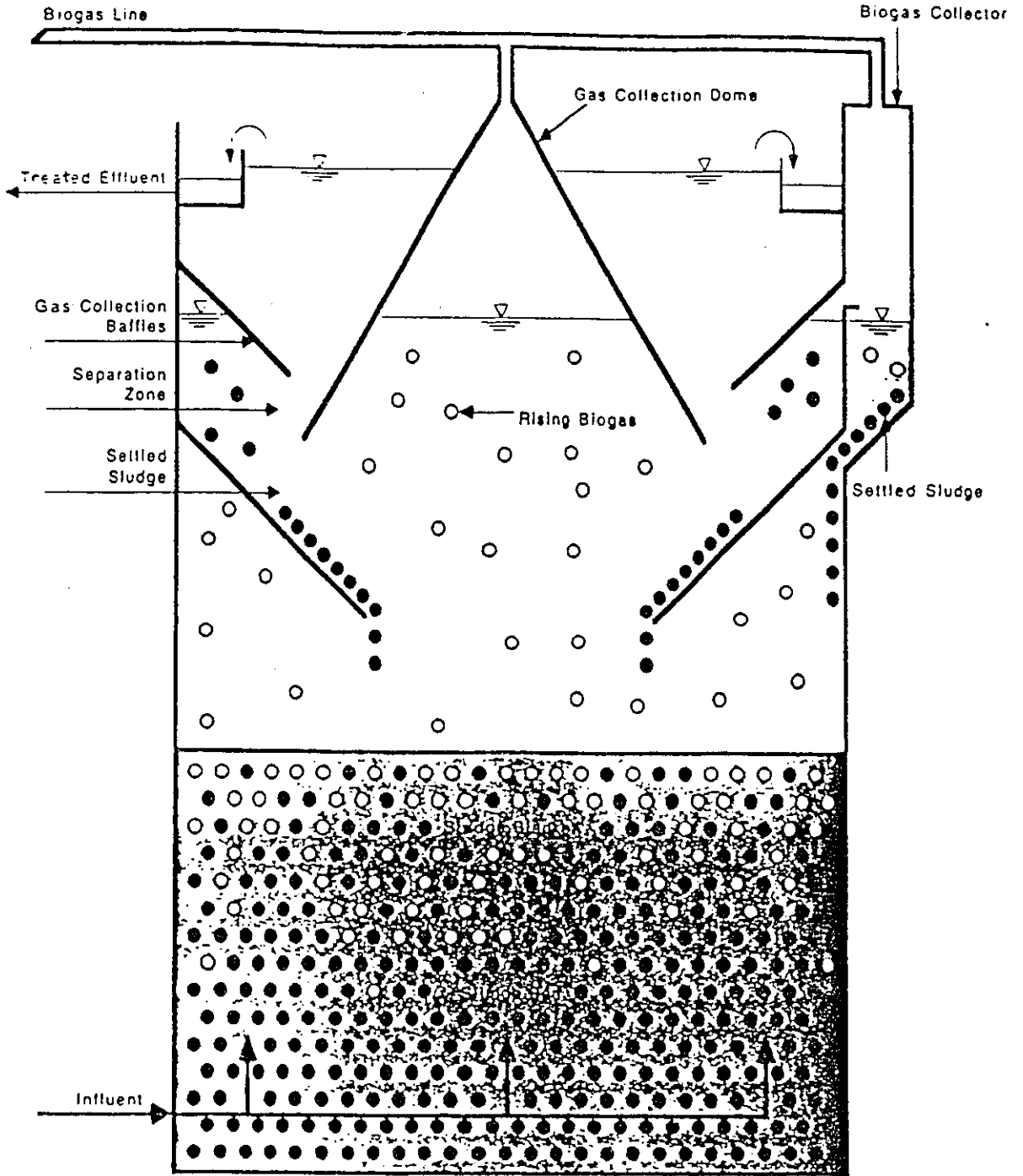


Figure 2.5 Biopaq UASB reactor schematic (Paques Lavalin, 1985)

The content of this thesis pertains to the operation of the two-stage anaerobic component of the pilot treatment facility therefore, details of the aerobic pilot system will not be further addressed. The research was conducted using a small scale system which has the same characteristics as the QRP PA tank and UASB reactor. These characteristics, such as operating parameters, are discussed in subsequent chapters.

CHAPTER 3

MATERIALS AND METHODS

3.1 Experimental Plan

The experimental study was carried out in two phases over a nine month period. Phase One involved a series of batch tests to determine to what extent the fiber/fines are responsible for the toxicity of CTMP-N (normal fiber/fines content) and CTMP-H (high fiber/fines content) wastewaters to granular anaerobic bacteria unacclimated to the wastewater and to investigate the effect of wastewater dilution on toxicity to these bacteria.

Phase Two involved an investigation of toxicity effects and fiber interaction with granular biomass in a continuous treatment system. A 140-day reactor study was undertaken to investigate whether treating unaltered CTMP-N had any significant disadvantage over CTMP-N with reduced fiber content. Phase Two was conducted based on the results of the batch inhibition tests in Phase One and the problems associated with the pilot plant operation previously discussed in Chapter 1.

CTMP-N was mill effluent collected under usual conditions of operation. CTMP-H was collected during a period when the mill was producing paper products of

extremely low fines (very small fibers) content, consequently fines concentration increased in the effluent.

3.2 Phase One

A series of inhibition tests or Modified Anaerobic Toxicity Assays (MATA) were performed on CTMP-N and CTMP-H wastewater. Total and soluble (filtrate from 0.8 μm filtration) phases of CTMP-N and CTMP-H were assayed. The CTMP-N and CTMP-H were obtained from QRP in February 1988 and frozen at -20°C until used. The wastewaters were centrifuged at 12,100 relative centrifugal force (RCF) for thirty minutes and the supernatant was filtered through an 0.8 μm pore size Millipore Type AA filter (cellulose, nitrate, acetate mixture) to remove the fiber/fines.

The tests were performed on unacclimatized granules that were cultivated on a 5.0 g/L substrate (50% sucrose - 50% acetate) in an UASB reactor (Division of Biological Sciences, National Research Council of Canada). Thus the inoculum was unacclimatized to CTMP wastewater.

Wastewater dilutions of 20, 40, and 60% (wastewater volume/total liquid volume) of total assay volume (50.5 mL) were tested. The tests were conducted according to a modified version of the Anaerobic Toxicity Assay (ATA) described by Cornacchio *et al.* (1986). Each bottle was given two consecutive spikes of an acetic acid/propionic acid substrate mixture, eight days apart. Initial substrate

concentrations were 1,630 mg acetic acid/L and 600 mg propionic acid/L in each bottle. Methane production was monitored and specific activity was calculated as g COD converted/g VSS·d.

3.3 Phase Two

Three two-stage anaerobic digestion systems treating CTMP-N wastewater of different fiber content are to be subsequently referred to as PL1, PL2, and PL3. During the CTMP-N run, system PL1 was fed unaltered CTMP-N wastewater. In system PL2, partial fiber removal was achieved in a clarifier prior to entering the PA tank. System PL3 received the soluble fraction (filtrant from 0.45 μ m filtration) of the CTMP-N wastewater.

The goal of the experiment was to reach the same hydraulic and organic loading as the QRP pilot plant (PA tank HRT 3.0 hours, UASB HRT 5.0 hours, and constant load of 18.5 g COD_T/L reactor·d) while monitoring system operation, performance, and granulation effects. Once steady state operation at these loading conditions was achieved, the goal was to maximize the loading rates by decreasing the dilution of the wastewater while maintaining the same hydraulic loading.

3.3.1 Experimental System

Laboratory scale models of the two-stage anaerobic treatment system, PA tank followed by an UASB reactor, used in this study are schematically presented in Figure 3.1. A photograph of the two-stage system is shown in Figure 3.2.

3.3.1.1 PA Tank

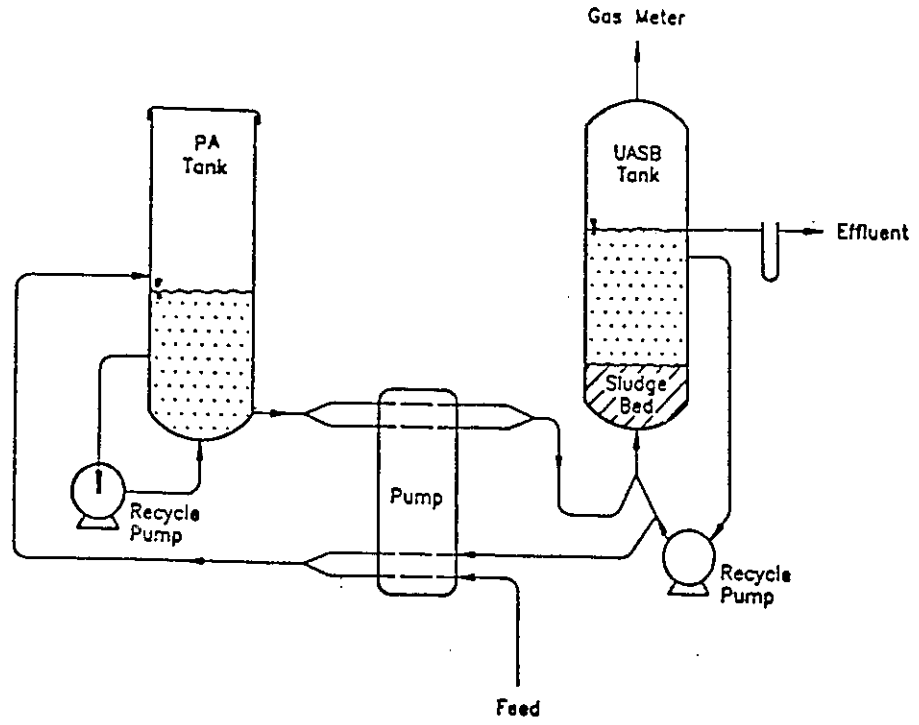
The PA and the UASB tanks were of the same construction. They were modified cylindrical Sovirel Pyrex reaction vessels (model 296-65) with 130 mm i.d.. Modifications included lengthening the vessel to 55 cm and addition of sampling ports along their lengths to allow for sample extraction and points of connection.

The active volume of the PA tanks ranged from 2.00 - 2.10 L. Complete mixed conditions were attained by high rate internal recycle at 22 L/min by centrifugal pumps (Cole-Palmer model N-07004-10). The tank top was covered with aluminum foil. The design HRT was 3.0 hours.

3.3.1.2 UASB Reactor

The UASB reactor was of a similar design as the PA tank. Unlike the PA tank, the UASB reactor had a layer of biorings at the liquid/headspace interface. The biorings which were made of polyethylene plastic (manufactured by Flexiring Koch Inc.), float. They were however partially submerged in the liquid under their own weight. The bior-

PL1 and PL3



PL2

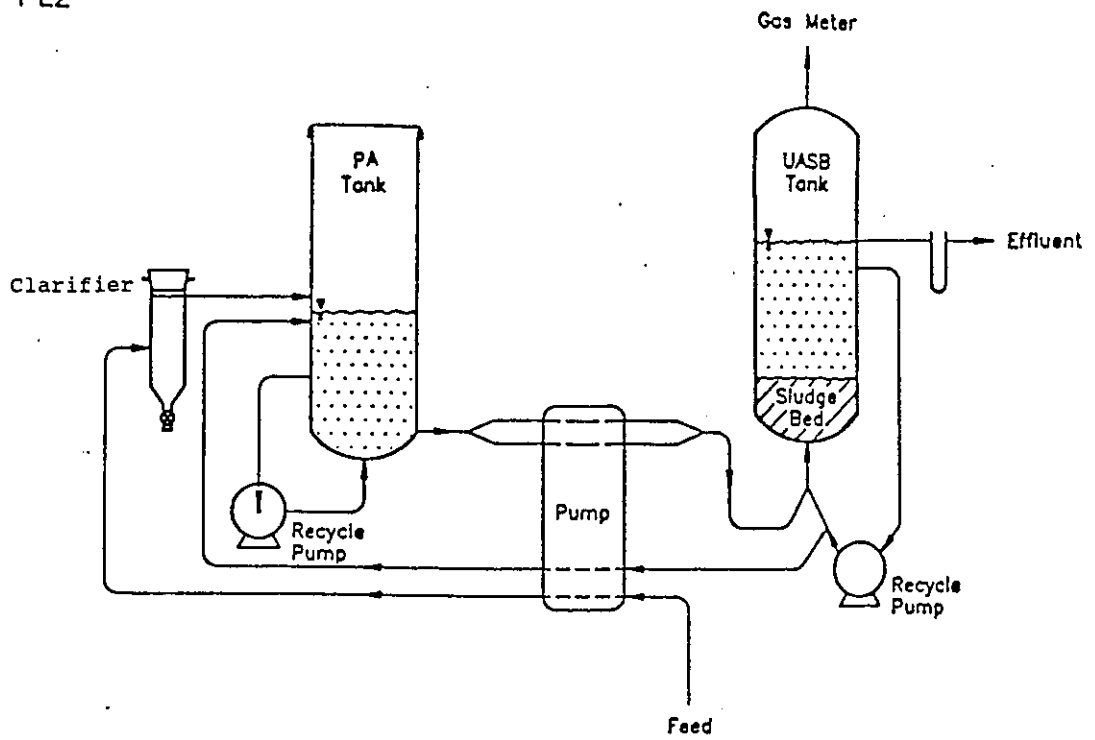


Figure 3.1 Experimental system

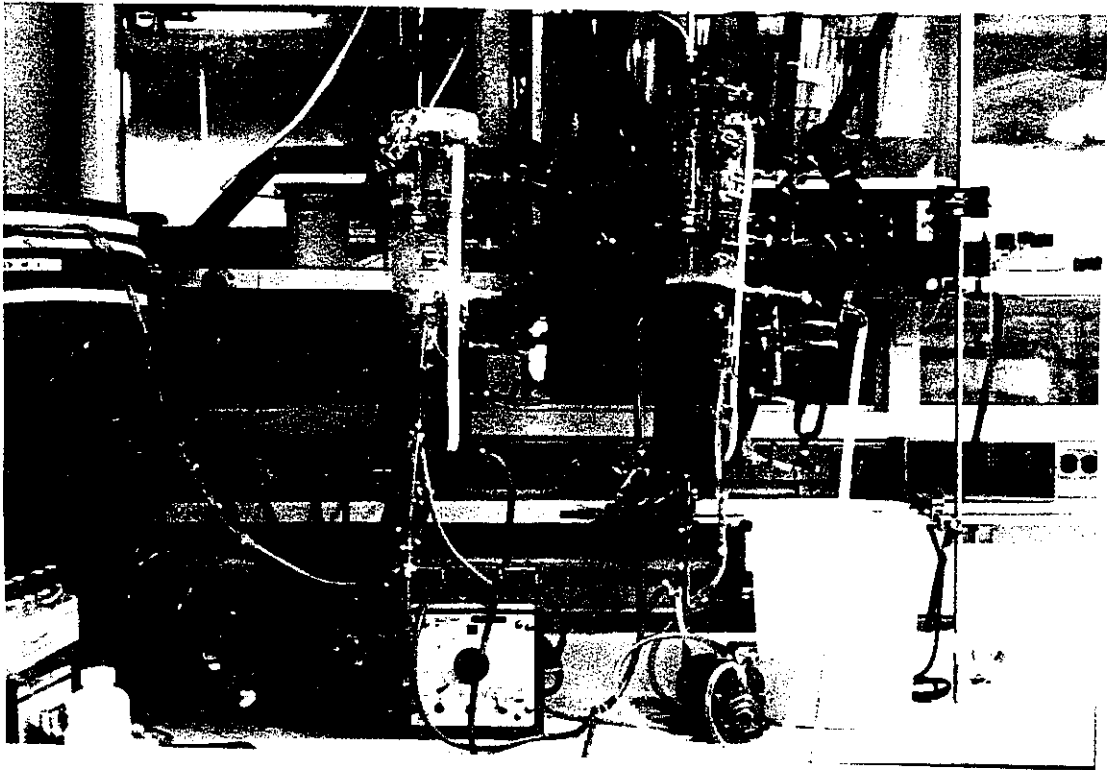


Figure 3.2 Photograph of the experimental system

ings acted as a gas/solids separator and prevented floating biomass from entering the overflow and exiting the reactor. Each bioring measures 1.5 cm in diameter and length (refer to Figure 3.3) with a surface area to volume ratio of $235 \text{ m}^2/\text{m}^3$. The active volume of the UASB reactors ranged from 3.34 - 3.44 L. The liquid volume displaced by the biorings was 0.03 L or 10%.

A variable speed Masterflex pump (Cole-Palmer model 7553-10, 1-600 rpm) provided internal recycle of the liquid at 0.15 L/min to ensure sufficient mixing of the sludge bed. The recirculation line was situated directly below the biorings.

A Tee-shaped liquid distributor at the reactor base inlet was constructed to minimize risks of channelling in the sludge bed. The liquid velocity at the Tee-shaped distributor was approximately 5 cm/s. The upflow velocity in the UASB was 0.73 m/h and the HRT was maintained at 5.0 hours.

The reactor lid was a clamped five port SVL reaction vessel lid. A gas line for the exit of biogas was attached to one of the ports of the lid. The other ports were fitted with screw caps which were easily opened to release pressure in the case of overflow tube blockage.

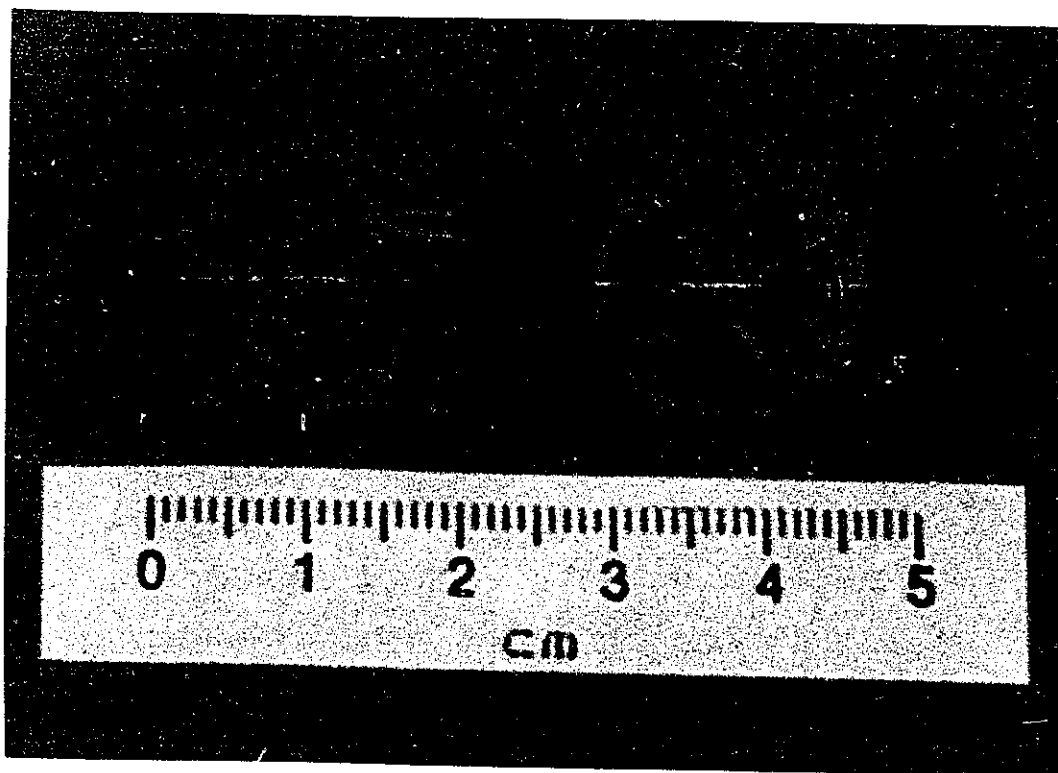


Figure 3.3 Photograph of the biorings

3.3.1.3 Clarifier

System PL2 (refer to Figure 3.1) had an additional component; a clarifier (settler) installed in-line preceding the PA tank for solids removal. The clarifier position in the laboratory scale system directly corresponded to the location of the inclined screen/Krofta in pilot treatment facility configuration at QRP.

The clarifier was constructed from a cylindrical glass column, 4.8 cm i.d., with a liquid volume of approximately 540 mL. Feed entered the clarifier at a liquid height of 17.5 cm in the column and overflowed at the 30.0 cm, the top of the column. The top of the clarifier was sealed with a rubber stopper.

Clarifiers that provide preliminary treatment ahead of biological treatment units are designed to provide detention periods of 30 to 60 minutes (Metcalf and Eddy, 1972). The HRT of the laboratory clarifier was 48 minutes. Settled solids were drawn off daily via a two-way valve at its base.

3.3.2 System Design Parameters and Operation

The system design parameters are presented in Table 3.1. Operation of the system (refer to Figure 3.1) was as follows. Feed was stored in a refrigerated tank at $4 \pm 1^{\circ}\text{C}$. A main feed line recirculated the feed to the reactor and back to the storage tank. A Harvard peristaltic pump (model 5006-033) drew feed from the main line and pumped it into

the PA tank in systems PL1 and PL3. Note that this pump had 4 lines for liquid transfer: feed (1), PA to UASB (2), and UASB to PA recirculation (1) with equal flow in each line. In system PL2, feed was initially pumped into the clarifier and overflowed into the PA tank.

In all systems; PL1, PL2, and PL3, the acidified effluent from the PA tank and introduced into the bottom of the UASB reactor. Waste then flowed up through a dense sludge bed and exited the reactor through an overflow port located at mid-height of the biorings. A port located just below the biorings was connected to a pump which provided internal recycle (232 L/d) to the bottom of the sludge bed.

Table 3.1 System design parameters

SYSTEM	PL1	PL2	PL3
VOLUME (L)			
Clarifier		0.54	
PA	2.00	2.00	2.10
UASB*	3.37	3.43	3.47
FLOW Q (L/d)	16.0	16.3	16.5
HRT (h)			
Clarifier		0.8	
PA	3.0	3.0	3.0
UASB	5.0	5.0	5.0
UASB Upflow Velocity (m/h)	0.73	0.73	0.73
UASB Recirculation to PA	50%	50%	50%

* 50 biorings occupied 0.03 L in each reactor

Approximately, fifty percent of the flow from the UASB was recirculated back to the PA tank via the internal recycle line by the peristaltic pump. Biogas produced by the sludge bed and collected in the UASB reactor headspace was measured with a Wet Tip Gas Meter. As shown in Figures 3.1 and 3.2, anaerobic conditions were maintained by employing water seals at the gas and effluent exits. Glass Tees (3/8") fitted with septa were installed in various lines for the purpose of providing locations to sample system liquids or gases. These locations included: the feed line, PA and UASB recycle lines for liquid phase samples, and the line between the reactor lid and the Wet Tip Gas Meter for gas sampling.

All experiments were performed in a temperature controlled room maintained at $35 \pm 2^{\circ}\text{C}$.

3.3.3 Wastewater Preparation

Frozen CTMP wastewater (6 m^3 CTMP-N and 2 m^3 CTMP-H) was received from the QRP in February 1988. The wastewater was 60% white water (after the inclined screen) and 40% gravity clarifier effluent (before the inclined screen). TMP wastewater (2 m^3) was received from QRP in October 1987. The in-line source of the TMP waste is unknown. The wastewaters had not received any chemical or nutrient additions prior to shipment. The wastewater characteristics are presented in Table 3.2.

Table 3.2 Wastewater characteristics

Wastewater	CTMP-N	CTMP-H	TMP
pH	7.4	7.6	4.5
COD (mg/L)			
COD _T	13,300	8,200	3,035
COD _S	8,200	5,500	2,255
Solids (g/kg)			
TSS	3.76	1.52	0.42
VSS	3.11	1.28	0.38
FSS	0.65	0.24	0.05
Protein (g/L)			
PR _T	1.55	1.98	0.95
PR _S	0.88	1.54	0.74
PR _{SUS}	0.67	0.44	0.21

The wastewater was thawed, mixed, transferred to 200 L barrels, refrozen, and put into cold storage (-20°C). Barrels were individually thawed, nutrients added, mixed, transferred to 20 L buckets, and refrozen to facilitate waste manipulation. Mixing was achieved by a submersible pump. Thaw time was minimized by thawing over a 65°C steam vent.

3.3.3.1 Filtration

A large volume of the soluble fraction (*i.e.* fiber/fines free) wastewater was required for system PL3. A high rate method to remove fiber was required. Filtration was selected as the preferred method of fiber removal instead of centrifugation because of this factor.

Two large (24 cm diameter) Whatman Qualitative filters (No.4 and No.1) were selected for comparison. The basis of filter paper selection were: efficiency of fiber removal, rate of removal, and cost effectiveness. This comparison is presented in Table 3.3. The evaluation was based on three sets of data per filter paper type.

Table 3.3 Filter paper data

Whatman Filter	No.1	No.4
Porosity (um)	11	20-25
Filtration Rate (L/min)	1.2	1.2
(L/filter)	3	12

Wastewater	Raw CTMP-N	Filtered CTMP-N	
		No.1	No.4
COD (mg/L (% Removal))			
COD _T	8,225	5,665 (31)	5,690 (31)
COD _S	5,460	5,185 (5)	5,275 (3)
Solids (g/kg (% Removal))			
TSS	1.53	0.13 (92)	0.24 (84)
VSS	1.28	0.08 (94)	0.14 (89)
FSS	0.25	0.05 (80)	0.10 (60)

The filters had comparable removal efficiencies and time required for filtration however, a No.4 paper could filter four times more wastewater than a No.1. Therefore, the Whatman Qualitative No.4 filters were selected. Fines greater than 20 to 25 um only were removed by filtration.

To prepare the filtered feed for system PL3, wastewater was thawed, gravity settled, and the supernatant was filtered. Nutrients were added to the waste after filtration. The wastewater was transferred to 20 L buckets and frozen at -20°C .

Wastewater stored at room temperature for extended periods would degrade. The time required to filter a barrel of CTMP-N was one day, therefore, the wastewater used for system PL1 was kept at room temperature one day longer than that used for systems PL1 and PL2. The degree of degradation of the wastewater due to extended storage at room temperature is visible in the measured COD_5 of the feeds (Appendix A, B, and C).

3.3.3.2 Nutrient Requirements

CTMP effluent does not contain sufficient concentrations of nitrogen and phosphorus for successful biological treatment. These elements were added to the wastewater in the form of ammonium hydroxide (NH_4OH , 28% w/v) and orthophosphoric acid (H_3PO_4 , 85% w/v) in the ratio $\text{COD}_5:\text{N}:\text{P} = 350:5:1$, the same ratio as was used at the pilot plant. This ratio was based on an ultimate biodegradable soluble COD removal of 60% (de Vegt, 1988) where the COD_5 was 5500, 8200, and 2300 mg/L for CTMP-N, CTMP-H, and TMP respectively. The nutrient quantities added per cubic meter of wastewater are presented in Table 3.4.

Table 3.4 Nutrient requirements per cubic meter wastewater

Wastewater	g NH ₄ OH/m ³ (28% w/v)	g H ₃ PO ₄ /m ³ (85% w/v)
CTMP-N	421	35
CTMP-H	628	52
TMP	194	16

3.3.4 Inoculum

The inoculum was a well granulated anaerobic sludge obtained from the UASB treatment system at the Roermond Paper Company. The Roermond Paper Company is a Dutch company which produces corrugated medium from recycled paper. Characteristics and performance criteria of the waste treatment system at the Roermond Paper Company are presented in Table 3.5. This sludge was also used to seed the pilot UASB reactor at the QRP. The sludge was received via Domtar and is therefore referred to as DOM seed sludge.

3.3.5 Schedule

A two-stage laboratory scale treatment system was tested prior to commencing the three system experiment to establish the process controls and the operational requirements of the system. The optimal system configuration and start-up procedures were determined.

Table 3.5 Roermond waste treatment system criteria

Reactor Volume (m ³)	1,000
Flow (m ³ /d)	3,000
Influent COD (mg/L)	3,500
Temperature (°C)	30-40
Volumetric Loading (g COD/L·d)*	11
HRT (hrs)	8.0
COD Removal Efficiency (%)	75

* Loading is based on total reactor volume.

3.3.5.1 Trial Run

The liquid volumes selected for the PA tank and the UASB reactor were 2.5 and 4.4 L, respectively. The reactors were initially filled with 2.0 L of effluent from an UASB reactor treating a 5.0 g/L substrate (50% sucrose - 50% acetate). Air in the system lines was bled. The UASB was purged with a gas mixture containing 20% CO₂ and 80% N₂ to maintain anaerobic conditions during inoculation. The UASB reactor was inoculated with a volume of DOM sludge equal to approximately one third the reactor volume (1.125 L). The PA tank was inoculated with 500 mL of DOM sludge and covered with foil. The sludge was left to sit in the system for one day.

On the second day, the UASB reactor was opened, filled to the overflow port with effluent from the sucrose/acetate fed reactor, and resealed. This corresponded to sludge bed

and reactor VSS concentrations of 44.0 g VSS/L of bed and 11.2 g VSS/L reactor, respectively.

Effluent from the sucrose/acetate fed reactor was also placed in the feed tank on the second day. The UASB reactor effluent line was placed in the feed tank and this "feed" was recycled through the system until the third day.

Feeding of CTMP-N wastewater, 50% diluted with tap water, was initiated on the third day at a flow rate of 8.0 L/d. The volumetric loading on the system was progressively increased to the design level of 21.1 L/d (PA tank HRT = 2.8 (approximately 3.0 hrs), UASB reactor HRT = 5.0 hrs). The trial run was conducted over a two month period. Start-up and operational parameters required for the three two-stage treatment system experiment were established.

3.3.5.2 Main Experiment Start-Up and Schedule

System start-up and operation was similar to the trial run except for the information presented in this section.

The operating liquid volumes of the PA tank and the UASB reactor in the three system two-stage anaerobic treatment experiment were decreased to approximately 2.0 and 3.4 L respectively due to the limited wastewater supply.

A 1.23 kg quantity of DOM sludge was added to each of the three UASB reactors. This corresponded to a total bed VSS of 55.5 g and a reactor VSS concentration of 16.1 g VSS/L reactor in each system at start-up.

Unlike the trial run, the PA tanks were not inoculated with the seed sludge. The acidogen culture in the PA tanks was permitted to establish itself possibly from bacteria present in the wastewater and/or from biomass recycled from the UASB during operation.

Changes to the start-up of the three two-stage systems included adding fifty biorings to each UASB reactor after the sludge addition to reduce biomass washout during operation.

Reactor biomass was reactivated by initially feeding the reactors the effluent from an UASB system which was fed the 5.0 g/L sucrose/acetate solution for three days. The initial flow rate was set at 4.0 L/d; one quarter the design flow rate (16.0 L/d). This corresponded to an HRT of approximately 12.0 hours in the PA tank and 20.0 hrs in the UASB reactor.

On the fourth day of operation (Day 0), the reactor feeds were switched to CTMP wastewater diluted 50% with tap water. Volumetric feed rates were increased gradually to the design levels (refer to Table 3.1) with the 50% diluted feed. Feed rate step increases were made only when a system's VFA levels were stable.

The reactors were allowed to operate at this level for forty days until Day 60. The reactors were then subjected to successively increasing feed strengths (lower dilution of wastewater). Increases were made only after a period of stable operation.

Reactor performance was monitored at each test condition. This included routine monitoring of the feed rate to the systems, the pH, alkalinity, and VFA concentration of the feed, PA, and UASB contents, COD and total and soluble suspended solids concentrations of the feed, PA tank, and effluent, as well as biogas quantity and composition, and operating temperature. Visible changes in the sludge bed were also recorded. UASB reactor biomass was characterized by VSS, COD, settleability and specific microbial activity by the Modified Batch Activity Test (MBAT) at the end of operation of each test segment to quantify the progressive changes in reactor biomass. The wastewaters, and sludges and effluents were analyzed for resin and long-chain fatty acids concentration at the end of the experiment prior to reactor shut down.

3.4 Analytical Methods

3.4.1 pH

The pH was measured using a Radiometer/Copenhagen pH meter model 26 with a glass electrode. Sensitivity of the unit was 0.05 pH units.

3.4.2 Alkalinity

Alkalinity as CaCO_3 was measured with an automatic titrator equipped with a Radiometer/Copenhagen pH meter. A pH of 3.75 marked the end point of titration. The titrant used was 0.1 N hydrochloric acid.

3.4.3 Gas Measurement

Biogas volumes were measured with a wet tip gas meter (Wet Tip Gas Co.). The sensitivity of the gas meter is approximately 10 mL/tip.

3.4.4 Gas Composition

Biogas composition was determined by gas chromatography using a Hewlett Packard model 5710A gas chromatograph with an integrator model (3386A). The column, held at 70°C , was packed with Porapak T 50/80 mesh. Helium gas flowing at a rate of 40 mL/min was the carrier gas. The injection port temperature was set at 100°C . The percent biogas fractions were corrected to standard temperature and pressure.

3.4.5 Volatile Fatty Acids

Volatile fatty acids (VFA): acetic, propionic, and butyric) were measured by the method of Ackman (1972) with a Hewlett Packard gas chromatograph (model 5730A) equipped with an automatic sampler (model 7671A) and integrator

(model 3380A). The glass column, Chromosorb 101, was kept at 180°C while the injection port temperature was 200°C. Helium passing over formic acid at a flow rate of 15 mL/min was used as the carrier gas. Samples were prepared for analysis by centrifuging a 1.0 mL volume in a microcentrifuge, withdrawing and mixing 0.5 mL of supernatant with 0.5 mL of internal standard containing 1000 mg/L isobutyric acid.

3.4.6 Chemical Oxygen Demand (COD)

COD was measured with the spectrophotometric method described by Knechtel (1978) except that samples were heated to 150°C in an oven for three hours rather than two hours.

3.4.7 Solids

Total (TSS), volatile (VSS), and fixed (FSS) suspended solids were measured by the method outlined in Standard Methods (APHA, 1985).

3.4.8 Specific Activity Test

Potential activity was measured in the following manner. Ten mL of a sludge sample diluted with a reduced phosphate buffer (the dilution factor was selected based on the anticipated activity) was transferred anaerobically into a serum bottle, for each of three bottles. A 0.2 mL volume

of a substrate solution, discussed below, was added to each of two bottles. A 0.2 mL volume of buffer was added to the third bottle, the control. After substrate or buffer addition, the bottles were capped and sealed and placed on a New Brunswick 4556 shaker rotating at 40 RPM in a room maintained at 35°C.

One mL samples were withdrawn anaerobically for substrate concentration determination at the time of substrate addition (time 0 hours) and at prescribed intervals thereafter. Withdrawn samples were centrifuged for five minutes and the supernatant was analysed. Typically, four or five samples were taken. The sampling schedule depended on the anticipated activity and the substrate.

The rate of substrate consumption less the control was computed and related to the biomass (VSS) present, yielding the specific substrate activity. Acetoclastic, propionic, and glycolytic activities were measured.

Acetoclastic activity is the ability of methanogens to convert acetate to methane. The acetate substrate solution was 200 g/L. Samples were withdrawn at time 0 hours, and every three hours thereafter until the ninth hour. If the rate of substrate consumption was low, an additional sample was taken at twenty-four hours. Acetate concentrations were determined immediately using gas chromatography.

Propionic activity is the ability of non-methanogens to break down long chain volatile acids. A 50 g/L substrate solution was used. Samples were taken at time 0, 6, 9, and

24 hours. Propionate levels were immediately measured by gas chromatography.

Glycolytic activity is the ability of non-methanogens to break down glucose. The glucose substrate solution was 200 g/L. Samples were extracted at time 0 hours and every fifteen minutes thereafter for an hour. Glucose concentrations were determined with the Dubois method (Dubois *et al.*, 1956). Extracted samples were prepared for glucose measurement as follows:

1. the supernatant from centrifuged extracted samples was made up to 2.0 mL with glass distilled water,
2. 50 uL of phenol was added, and
3. 5.0 mL of H₂SO₄ was added.

The absorbance of the prepared samples were then determined using a spectrophotometer set at a wavelength of 490 nm. Absorbances were converted to glucose concentrations using predetermined calibration curves.

3.4.9 Modified Anaerobic Toxicity Assay

In the ATA, varying concentrations of wastewater, a nutrient supplement, and an acetic acid/propionic acid substrate were combined and inoculated with an active anaerobic culture in a closed serum bottle. Toxic effects of the wastewater on the methanogenic and/or acetogenic microorganisms may be indicated by differences in methane production patterns (Cornacchio *et al.*, 1986). This

procedure was modified (MATA) for use in this thesis. A detailed description of the modified procedure is provided in Appendix F.

3.4.10 Modified Batch Activity Test

The Modified Batch Activity Test (MBAT) serum bottle technique combines aspects of the specific activity test and the MATA. This assay was performed to determine the activity of sludge on wastewater and specific substrates.

The wastewater received nutrients in the ratio of $\text{COD}_5:\text{N}:\text{P} = 350:5:1$, was diluted with glass distilled water (5:2) and flushed with 20% $\text{CO}_2/80\% \text{N}_2$ gas. Reactor sludge samples were removed anaerobically from the top and bottom of the sludge bed and combined in equal volumes. An 8.0 mL volume of sludge was added to 42.0 mL of wastewater (60% wastewater/total liquid volume) in 150 mL serum bottles which were gas flushed, capped with a butyl rubber stopper, and sealed. Quantity and composition of the biogas produced were monitored. Activity was expressed as the rate of CH_4 produced g COD/g VSS \cdot d.

Activity on a specific substrate was determined by injecting the serum bottle with 1.5 mL of a 75 g/L substrate (acetate, formate, glucose, or methanol) and monitoring biogas production. The injection was made when the residual COD in the serum bottle after wastewater consumption was

non-biodegradable (i.e. no further biogas production from the wastewater).

3.4.11 Settleability Test

The Settleability Test is a simple upflow velocity test to characterize settling properties of granular sludges from anaerobic sludge bed reactors (Andras et al., 1989). A 10.0 mL sludge sample was placed in a glass column (20 cm long, 2.5 cm i.d.) and water at 33 - 35°C is pumped via a variable speed pump through the test column in an upflow mode. Ten flow velocities (2 - 68 m/h) were maintained for five minutes each. Granules exited the glass column via an overflow port. The fraction of sludge exiting at each particular velocity was collected on filter paper (Whatman Qualitative No.1, 11 cm), photographed, and the dry weight of each fraction determined.

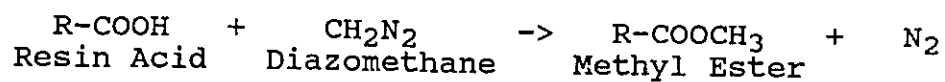
The percentage of suspended solids exiting the glass column plotted against upflow velocity gave the settling velocity profile of a particular sample. The upflow velocity corresponding to washout of 50% of the sludge (V_{50}) and the area under a plot of cumulative percent of TSS washed out vs upflow velocity quantify the results. In general, a high V_{50} and small area indicates good settleability.

Photographs assisted in the comparison of sludges in terms of granule size, regularity of granule shape and homogeneity of the fractions.

3.4.12 Resin Acids

Resin acid analysis was performed by Paracel Laboratories Ltd., Nepean, Ontario, Canada. The original method was developed by PAPRICAN and is summarized below.

A measured sample volume, typically 50 mL, was adjusted to a pH of 9 and funnel extracted with separate portions of methyl t-butyl ether having a volume equal to that of the sample. Sludge samples were initially prepared by diluting a 10 mL aliquot of sample with deionized water to 50 mL. The acids present in the concentrated extract were converted to their methyl esters using diazomethane as the derivatizing agent (as illustrated below).



After concentration of the methylated extract, the methyl ester derivatives of the resin acids were determined by capillary gas chromatography with a flame ionization detector.

CHAPTER 4

PHASE ONE: ANAEROBIC TOXICITY OF FIBER AND FINES (MATA) RESULTS AND DISCUSSION

The Modified Anaerobic Toxicity Assay (MATA) provides the most reliable results when the inoculum is active, healthy and acclimatized to the substrate (Cornacchio *et al.*, 1986). A comparison of the specific activities on three different substrates for two sludges, DBS and DOM, was made to determine which sludge would be more amenable for the MATA.

4.1 Sludge Characterization and Selection by the Specific Activity Test

The DBS sludge was taken from the active UASB reactor cultivated on a 5.0 g/L substrate (50% sucrose - 50% acetate, Division of Biological Sciences (DBS), National Research Council of Canada). The alternate sludge was the DOM sludge previously discussed in Section 3.3.4. This sludge had been in cold storage (+4°C) for a year. Neither sludge was acclimatized to CTMP wastewater. The sludges were similar physically; both were homogeneous and well granulated with medium size granules (2 - 5 mm in diameter). The specific activity of the sludges on acetate, propionate, and glucose were tested in duplicate for each

sludge. The average results of these are presented in Table 4.1. These results are accurate to $\pm 10\%$.

Table 4.1 Specific activity of DBS and DOM sludge

Sludge	DBS		DOM	
	Activity gCOD/gVSS·d	Lag h	Activity gCOD/gVSS·d	Lag h
Acetate	0.406	0	0.027	5.0
Propionate	0.053	0	0.001	0
Glucose	1.065	0	0.278	2.5

In terms of specific activity, the DOM sludge displayed only a fraction of the specific activity of the DBS sludge on all three substrates. In addition, the DOM sludge experienced an initial lag period before metabolizing the acetate and glucose. The DBS sludge displayed the highest specific activity on all three substrates without any lag time. The DBS sludge best satisfied the criteria for the MATA inoculum and was therefore used in the assay.

4.2 Inhibition Test

Batch toxicity tests (MATA) were performed on CTMP-N and CTMP-H wastewater to determine to what extent the fiber/fines were responsible for the inhibition to methanogenesis of acetate and propionate (*i.e.* toxicity) by anaerobic biomass unacclimatized to the CTMP waste.

Inhibitory effects of the total and soluble phases of the CTMP wastewaters at 20, 40, and 60% dilutions (wastewater volume/total liquid volume) were examined in duplicate. Methane production from an acetate/propionate spike was monitored. The specific activity was calculated as g COD converted to $\text{CH}_4/\text{g VSS}\cdot\text{d}$. Inhibition, the decrease in activity compared to the control, was expressed as a percentage. A detailed description of the MATA and the calculation of the percent inhibition are contained in Appendix F.

4.2.1 Inoculum Preparation

DBS sludge was removed anaerobically from the stock UASB reactor. The sludge was settled in a sealed flask. The supernatant was removed after settling and the sludge was resuspended with defined medium (Appendix F) in the ratio of eight parts sludge + ten parts defined medium. Eighteen mL of this mixture was anaerobically dispensed into each test serum bottle. The average TSS and VSS of the sludge mixture were 25.5 and 22.3 g/L, respectively.

4.2.2 CTMP Wastewater Preparation

The total and soluble phases of both CTMP-N and CTMP-H wastewater were tested. The soluble phase was prepared by removing the fiber/fines fraction by centrifugation at 10,000 RPM for thirty minutes followed by filtration with

Millipore Type AA filter paper (cellulose, nitrate, acetate mixture), porosity 0.80 μm . The wastewater characteristics are presented in Table 4.2.

Table 4.2 MATA wastewater characteristics

Wastewater	TSS g/L	VSS g/L	COD _T mg/L	COD _S mg/L
CTMP-H (total)	3.76	3.11	13,500	8,115(CF)
CTMP-H (soluble)	0.11	0.07	6,845(F)	6,845(F)
CTMP-N (total)	1.63	1.36	7,300	5,415(CF)
CTMP-N (soluble)	0.09	0.05	5,200(F)	5,200(F)

CF: centrifuged 20 minutes at 10,000 RPM
 F: filtered 0.80 μm Millipore Type AA.

Three dilutions, 20, 40, and 60% (wastewater volume/total liquid volume) of both the total and soluble fractions of each wastewater were prepared. The pH of the wastewater solutions were adjusted to 7.0 at 35°C with 1 N H₂SO₄.

4.2.3 MATA Test Results

A single control bottle was prepared for each of the four series of wastewaters. The biogas production from the initial volatile organic acid (VOA) spike was monitored each day for seven days. The bottles were removed from the shaker on the seventh day and settled. The weight of the

bottles was recorded and 1.5 mL of supernatant was removed for COD_S analysis. The bottles were reweighed and the masses recorded.

The bottles were flushed with 20% CO₂/80% N₂ for twenty-five minutes and injected with a second VOA spike of 3.0 mL on day eight. Each serum bottle was shaken, settled, and 1.5 mL of the supernatant was removed for COD_S analysis after addition of the spike. The assay was then continued as described in the MATA procedure (Appendix F) and terminated on day sixteen.

The initial substrate concentrations in the serum bottles were: 1,085 mg acetate/L, 400 mg propionate/L after the first spike and 1,630 mg acetate/L, 600 mg propionate/L after the second spike.

The calculated average microbial (*i.e.* neglecting solids contribution from the wastewaters) TSS and VSS in each serum bottle was 9.1 and 7.9 g/L, respectively.

A graphical presentation of the CH₄ production is illustrated in Figures 4.1 to 4.4 and the activity and inhibition results are listed in Table 4.3. Inhibition is expressed as a percent of the control, for example, the test serum bottle specific activity was 8% of the control activity if the inhibition was 92%. Results are accurate to approximately 10%.

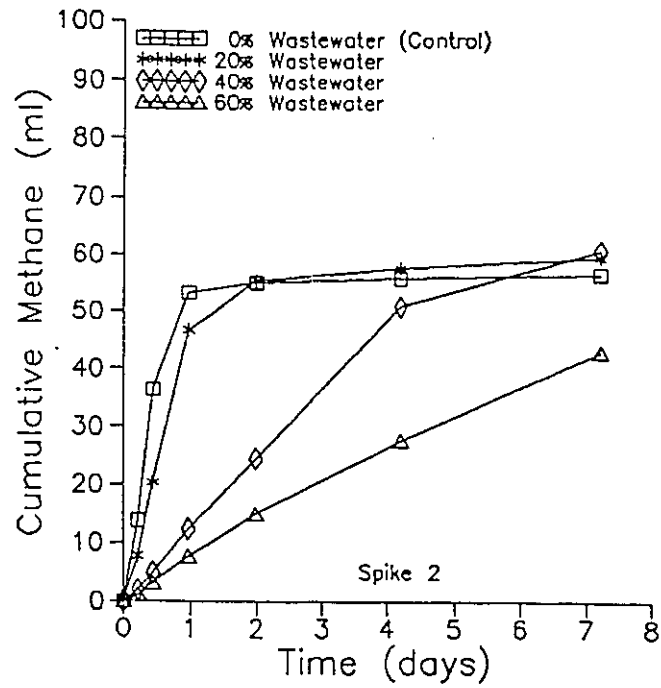
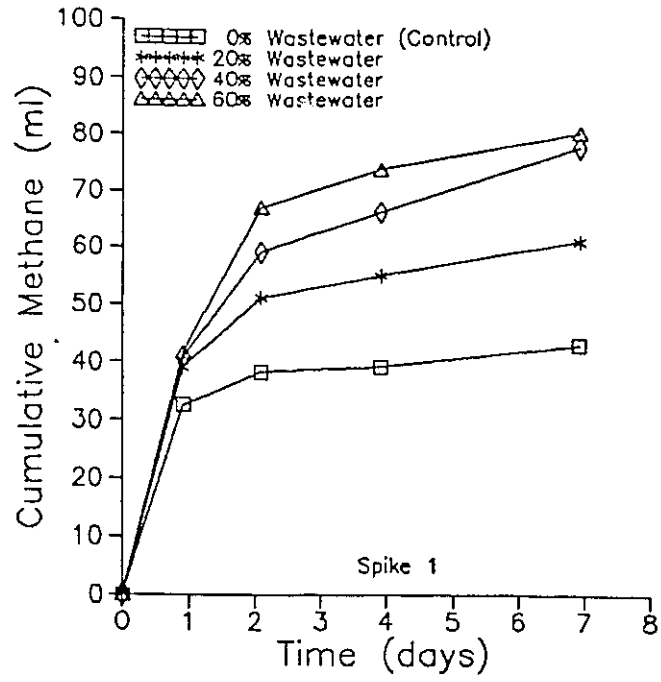


Figure 4.1 Cumulative CH₄ production for CTMP-N (total)

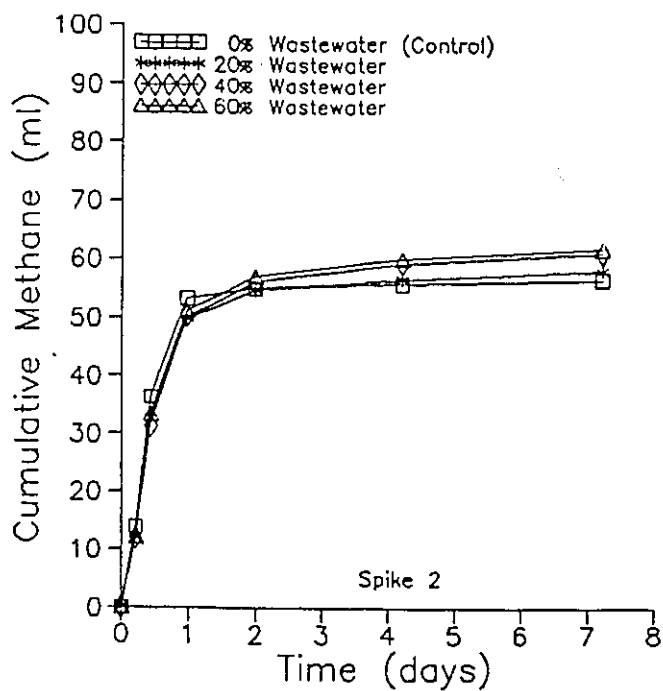
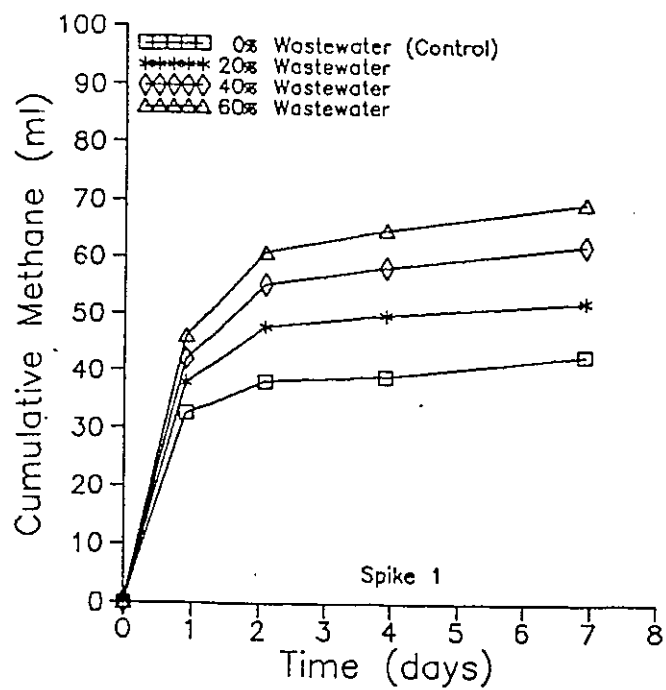


Figure 4.2 Cumulative CH₄ production for CTMP-N (soluble)

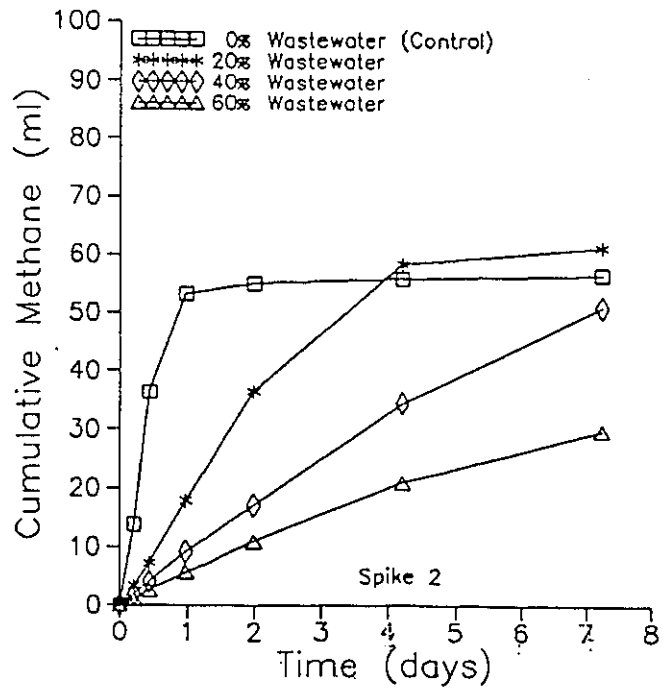
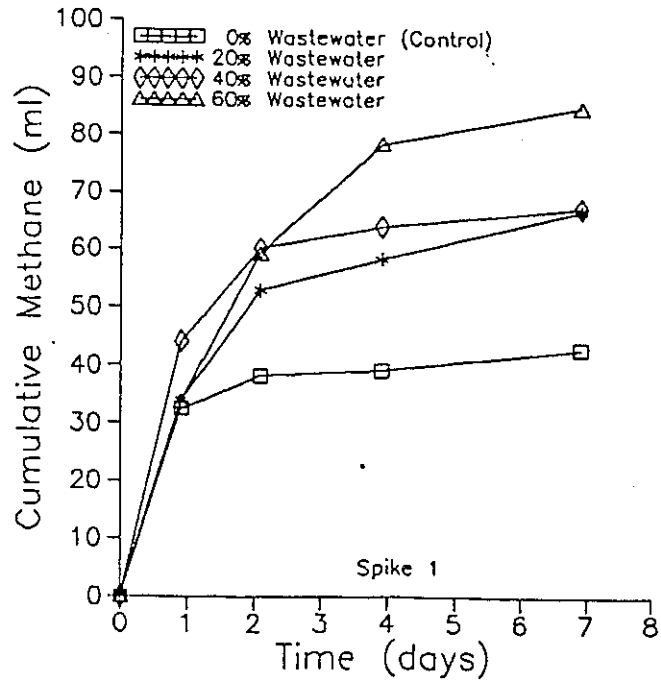


Figure 4.3 Cumulative CH₄ production for CTMP-H (total)

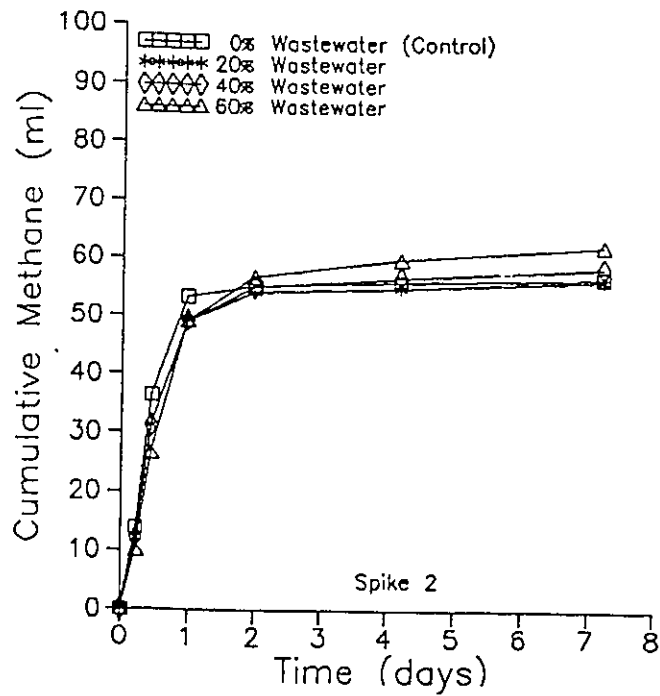
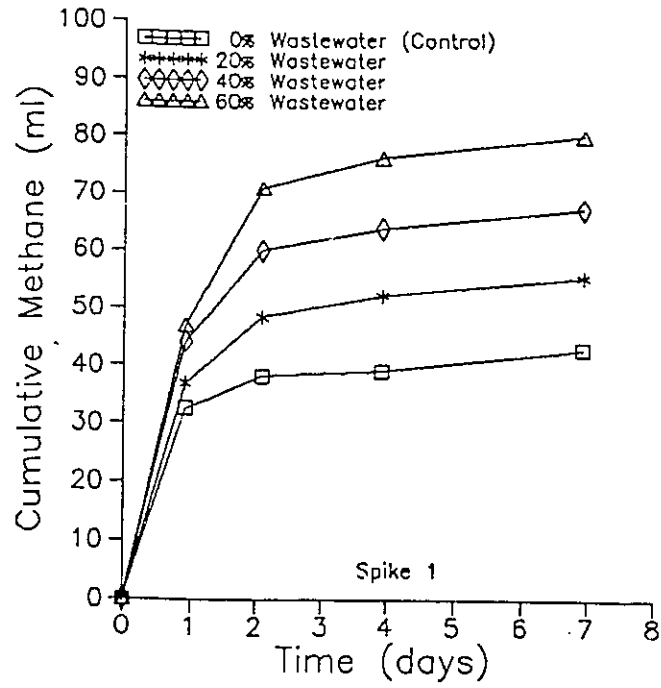


Figure 4.4 Cumulative CH₄ production for CTMP-H (soluble)

Table 4.3 MATA results

Wastewater		Activity gCOD/gVSS·d	Inhibition %
CTMP-H (total)	20%*	0.081	84.4
	40%	0.047	91.0
	60%	0.029	94.4
CTMP-H (soluble)	20%	0.440	14.8
	40%	0.438	15.2
	60%	0.371	28.2
CTMP-N (total)	20%	0.261	49.5
	40%	0.064	87.5
	60%	0.042	91.9
CTMP-N (soluble)	20%	0.459	11.1
	40%	0.441	14.6
	60%	0.466	9.7
CONTROL (average of four samples)		0.517	

* wastewater volume/total liquid volume expressed as a percent

4.2.3.1 Results of the First Spike

A decrease in biological activity after the first VOA spike was not observed. A possible explanation of this observation follows. Methane produced from the biodegradable portion of the CTMP waste in addition to methane production from consumption of the spike exceeded that of the controls (VOA substrate only), for all test serum bottles with the exception of the CTMP-H (total) samples. In these samples, the additional volume of methane produced from CTMP masked the inhibition effect. A second VOA spike was injected when the biogas production from the first spike plus wastewater

ceased. The recalcitrant COD₅ was measured at this point to be approximately 40% of the initial COD₅ in the wastewater

The VOA spike as sole substrate was provided by the second spike methane production profile. Fiber inhibition could be determined since the masking effects of the biodegradable fraction of the wastewaters would be non-existent.

4.2.3.2 Results of the Second Spike

CTMP-H (total) wastewater contained 2.4 times more suspended solids (fiber/fines) than CTMP-N (total). Methane production in the presence of CTMP-H (total) wastewater was reduced compared to CTMP-N (total). Unaltered CTMP-H wastewater caused an 84 - 94 percent reduction of activity, while CTMP-N had inhibition, relative to the control, of 50 - 92%.

The presence of soluble fraction (filtrant from 0.80 um filtration) of the wastewaters also resulted in a lower methane production relative to the control, however to a much lesser degree than the unaltered (unfiltered) waste. Soluble phase exhibited 10 - 15% and 15 - 28% inhibition for CTMP-N and CTMP-H wastewaters, respectively.

Dilution decreased the toxicity of both unaltered wastewaters. CTMP-N inhibited activity by 50% when applied as a 20% solution, while an activity reduction of 92% was measured with the 60% solution. CTMP-H inhibited activity

84% as a 20% solution, while an activity reduction of 94% was measured with the 60% solution. The dilution effect was not pronounced for the soluble phases.

In general, solids removal and dilution of CTMP wastewaters significantly reduces their toxicity to unacclimatized anaerobic granular sludge. A graphical presentation of the inhibition data is provided in Figure 4.5.

Another study conducted at the Division of Biological Sciences, National Research Council of Canada, using the DOM sludge as the inoculum and CTMP-N fines wastewater demonstrated comparable levels of inhibition (McCarthy *et al.*, 1990).

Based on the results of these MATA tests future investigations with CTMP wastewater was split into two sub-projects. One of the projects was defined to expand the knowledge base of CTMP-N fiber toxicity using the MATA (McCarthy *et al.*, 1990). The following items were investigated in the MATA study conducted by McCarthy and co-workers (1990).

1. Wastewater was reconstituted with different concentrations of fiber to examine fiber toxicity.
2. The methanol soluble fraction of the fiber was investigated to answer the question of whether it is the fiber or the soluble compounds associated with the fines which are responsible for the toxicity.
3. Potential contribution of resin acids to overall toxicity was investigated.

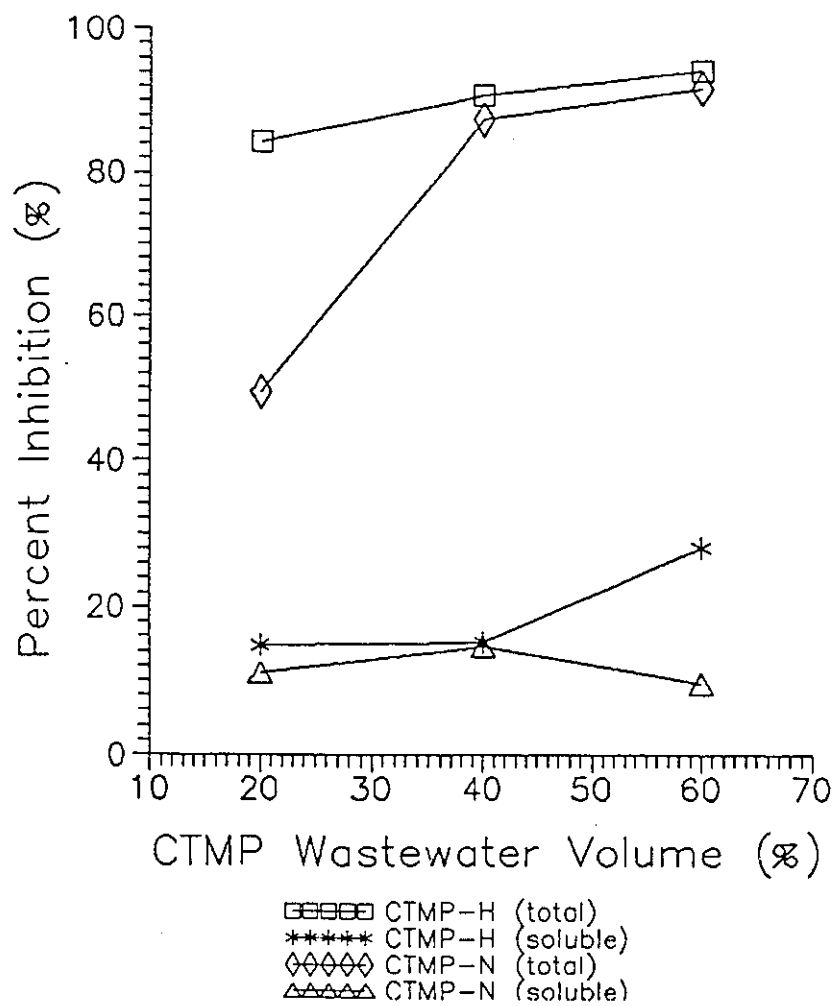


Figure 4.5 Percent inhibition (Spike 2)

The second set of experiments in this thesis was the investigation of the fiber associated toxicity and granulation effects in continuous systems treating CTMP-N wastewater with varying fiber concentrations (Chapter 5).

CHAPTER 5

PHASE TWO: REACTOR STUDY AND COMPARISON RESULTS AND DISCUSSION

5.1 Preliminary Test Operation: PL

Initial start-up of the trial system was unsuccessful because the volumetric loading was increased too rapidly. Following inoculation, the loading rate of wastewater diluted 50% with tap water was doubled in two days from 8 to 16 L/d (UASB volume = 4.4 L, UASB HRT decrease from 13.0 to 6.5 hours). Volatile fatty acid concentrations in the UASB reactor were equivalent to the concentrations in the PA tank (485 mg acetic acid/L and 25 mg propionic acid/L). The hydraulic loading was decreased to 14 L/d with hopes of stabilizing the system, however, the UASB reactor VFA concentrations remained approximately equal to the PA tank. System recovery did not occur within the next ten days. The UASB reactor was reinoculated on Day 17.

The second start-up procedure was more regulated. Incremental changes of the hydraulic loading rate and associated UASB reactor VFA stabilization periods were permitted. Wastewater feeding was commenced on Day 3 at 8 L/d. The hydraulic loading was increased to 10, 15, and 21 L/d on Day 7, 11, and 15 respectively.

A clarifier was installed in front of the PA tank on Day 20 to investigate the clarifier's capacity for fiber/fines removal. A deterioration of the UASB reactor performance followed; the UASB reactor VFA concentrations were equivalent to the PA tank concentrations. This process upset was suspected to be the result of the clarifier installation and/or a delayed response of the biomass to the increased loading rate. In order to restabilize the system, operating conditions were changed to a state under which the system's operation was stable (i.e. the clarifier was removed the second day after installation and the volumetric loading was decreased to 14 L/d). The reactor restabilized in four days.

The volumetric loading was increased daily until the design loading of 21 L/d was reached on Day 28. The system was successfully operated at this loading for twelve days. Organic loading rates were 20.8 g COD_T/L·d and 13.3 g COD_S/L·d. Specific loading rates were 1.85 g COD_T/g VSS·d and 1.18 g COD_S/g VSS·d based on the initial inoculum VSS concentrations. Total and soluble COD average removals were 38 and 44%, respectively.

Progressive loss of biomass from the UASB was observed. Fifty biorings were added to the UASB on Day 39 of operation to restrict biomass washout.

The clarifier was reinstalled before the PA tank on Day 40. This did not have a negative effect on system operation. Fines were successfully removed. It was observed

that the quantity of solids settled would required daily draw off from the clarifier.

Wastewater concentration was increased from 50% (1 part wastewater + 1 part tap water) to 70% (7 parts wastewater + 3 parts tap water) for the final three days of operation. Soluble COD removal was maintained at 40%. In general, no major technical or system performance problems were encountered.

5.2 Treatment of CTMP-N Wastewater with Varying Fiber Content

This section presents results of the operation of three two-stage anaerobic digestion systems (PL1, PL2, and PL3). System start-up and operation are described and discussed. Results during the loading periods are summarized. Operating data and comments are contained in Appendices A (PL1), B (PL2), and C (PL3).

5.2.1 Start-Up and Loading Schedule

Initially, all three systems were fed effluent from sucrose/acetate reactors for the first three days of operation to reactivate the sludge. Subsequent feeding of 50% diluted CTMP-N wastewater at a rate of 4.0 L/d was commenced. The volumetric loading rate was progressively increased to design levels by Day 18 (volumetric feed rate

16 L/d at wastewater dilution of 50%). The systems were operated at this loading until Day 60.

The wastewater concentrations in the feeds were then increased by decreasing the dilution of the wastewater until feeding 100% wastewater (0% dilution) was achieved. At the end of the CTMP-N run, systems PL2 and PL3 were shut down. System PL1 was additionally fed 100% TMP wastewater followed by 100% CTMP-H wastewater. The treatment periods are specified in Table 5.1.

Table 5.1 Loading schedule for PL1, PL2, and PL3

% Wastewater Concentration	Operation Interval d	Delta Time d
Start-up	0-18	18
50	19-60	42
70	61-87	27
80	88-94	7
90	95-108	14
100	109-143	35 (PL1)
	-138	30 (PL2)
	-140	32 (PL3)
PL1 Only:		
100% TMP	144-150	7
100% CTMP-H	151-159	9

5.2.2 Volumetric Loading Rate

Maintenance of a constant liquid volume in each of the three PA tanks was difficult (Figure 5.1). The liquid volume in the tanks tended to increase or decrease from day to day. Maintaining the desired liquid volumes was not feasi

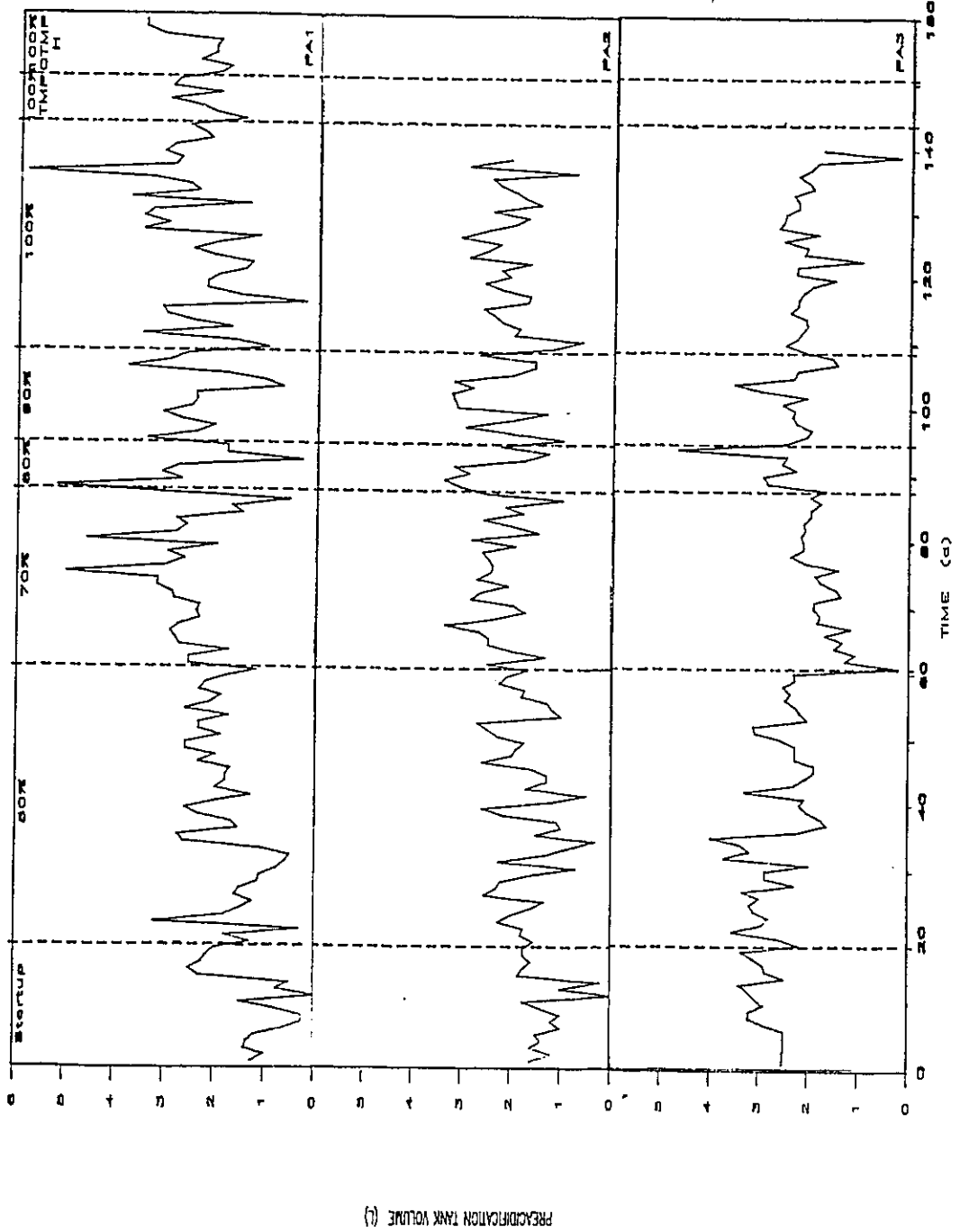


Figure 5.1 PA tank volumes versus operating time

ble through operational adjustments. To compensate, excess liquid was removed and discarded to the effluent tank when liquid volumes were high. Effluent was used to bring the volume up to design levels when liquid heights were low. These adjustments were made daily. An example calculation of the flow rate, Q , is presented in Appendix D. The flow rate for each system is presented schematically in Figure 5.2.

5.2.3 Temperature

System operating temperatures, illustrated in Appendix A, Figure A6, remained stable at $34 \pm 1^{\circ}\text{C}$ throughout the course of the experiment.

5.2.4 Chemical Oxygen Demand

The COD_T and COD_S of the feed, PA tank and effluent are presented in the Appendices as Figures of A1 a-b, B1 a-b, and C1 a-b for system PL1, PL2, and PL3, respectively. Interpretation of the COD data is discussed in subsequent sections.

5.2.5 pH and Alkalinity

As shown in Appendices A, B, and C, the feed, PA tank, and UASB reactor were stable in terms of pH and alkalinity under all loading conditions for each of the three systems.

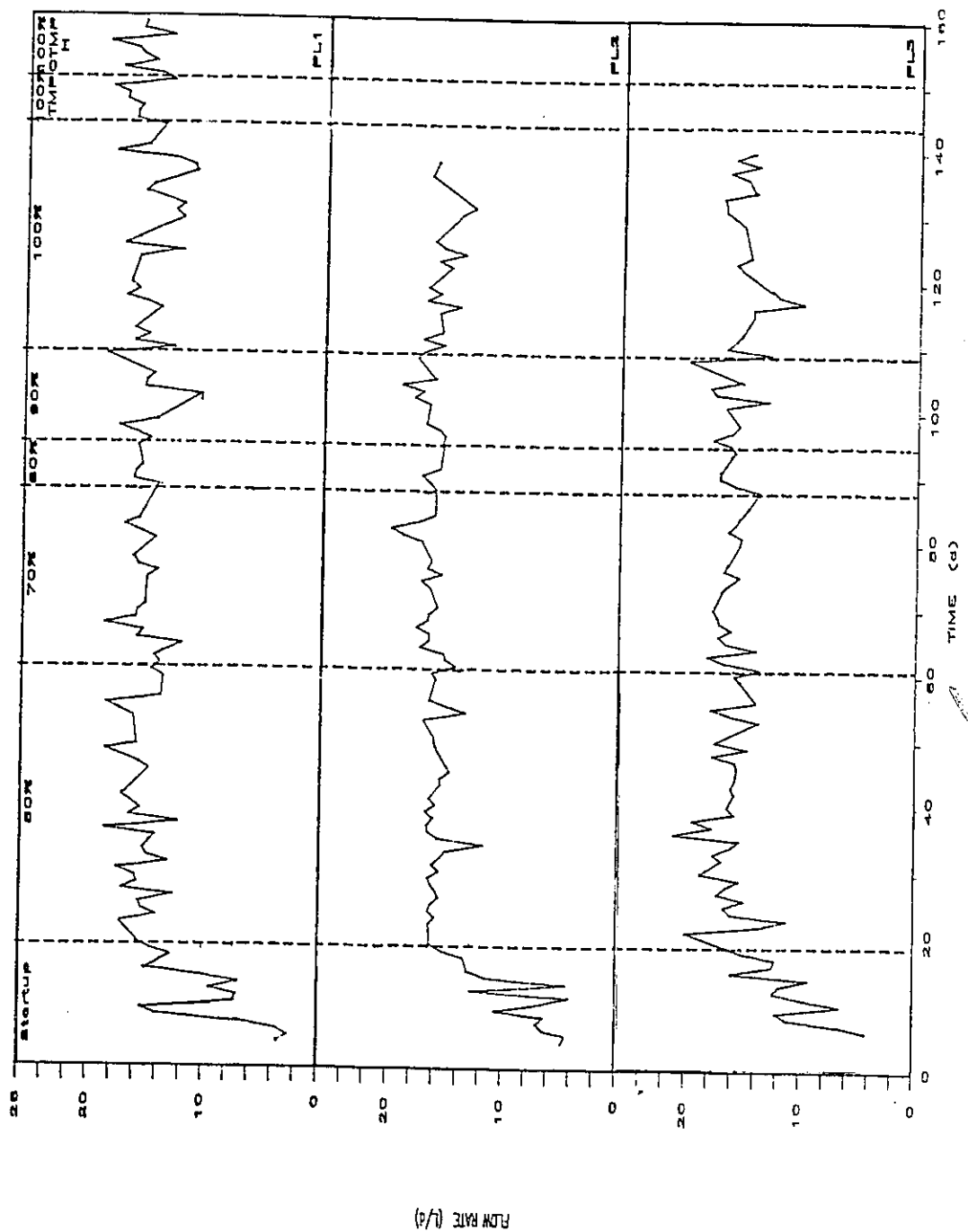


Figure 5.2 Volumetric loading rates (Q) versus operating time

The average influent pH was 6.4 for all three systems over the course of the experiment excluding the start-up period. In each system, the average pH in the UASB reactor was approximately 7.0. The average pHs in the PA tanks were 6.5, 6.9, and 6.8 in system PL1, PL2, and PL3, respectively.

Alkalinity in systems PL1 and PL2 were comparable. The average alkalinity of the feed containing 100% wastewater was 1,364 mg as CaCO₃/L in PL1, 1,340 mg/L in PL2, and 1,186 mg/L in PL3. The corresponding average alkalinity in the PA tanks was 1,772 (PL1), 2,015 (PL2), and 1,532 (PL3) and the UASB reactors was 1,793 (PL1), 2,178 (PL2), and 1,846 (PL3) mg/L.

5.2.6 Volatile Fatty Acids

Volatile fatty acids (acetic, propionic, and butyric) in the feeds, PA tanks, and UASB reactors were monitored. Acetic and propionic acid concentrations versus operating time are illustrated in Figures A4 a-b, B4 a-b, and C4 a-b for systems PL1, PL2, and PL3, respectively.

System VFA concentrations readjusted to higher CTMP-N wastewater concentrations in the feed within five days on average. Feed acetic acid concentrations were relatively consistent (concentration proportional to the soluble organic content) for PL1 and PL2 whereas the levels for the PL3 feed were more variable. A microbial film tended to form on the inside wall of PL3's feed tank. It is likely

the microorganisms in this film decreased the VFA content of the feed entering the preacidification tank. The average acetic acid concentration at 100% wastewater concentration was 1,362 in PL1, 1,266 in PL2, and 1,064 mg/L in PL3.

Propionic acid concentrations were inconsistent for all feeds. A general trend for propionic acid to increase with increased wastewater concentration in the feed was observed. Throughout the course of the experiment, propionic acid ranged from 0 - 250 mg/L in all feeds.

Butyric acid was detected only once in the fines free feed to PL1 and PL2 (40 mg/L) and not at all in the fines free feed to PL3. It was also detected once in PA tank of system PL2 (25 mg/L) during the experiment. The occurrence of butyric acid was not significant and it is therefore not graphically presented.

All VFA concentrations decreased in the UASB reactors. Propionic acid was reduced to below 40 mg/L in the first two systems and 5 mg/L in the third. Acetic acid was removed to less than 400 mg/L in PL1 and PL2 and to approximately 100 mg/L in PL3.

PL3 had unusually high concentrations of acetic acid during feeding of 100% wastewater. This was attributed to elevated VFA concentrations in the feed and a series of mechanical problems associated with PL3. If feed to the system was not maintained while internal recycle in the UASB continued, the VFA concentrations in the UASB diminished. When feeding was restored, acids increased to a concentra-

tion greater than before the pumping problem arose. This was indicative of microbial stress and disruption of sludge bed activity. PL3 was subject to three upsets during the experiment. VFA concentration in the UASB dropped when feed was not supplied, and spiked when restored. The overall effect was an elevated average VFA concentration in UASB3. PL1 and PL2 underwent similar mechanical problems but less frequently. In summary, the sludge beds were able to recover from the process upsets which occurred due to minor mechanical problems.

5.2.7 Total and Volatile Suspended Solids Content

The TSS and VSS content of the feed, PA tank, and effluent over the course of the experiment are graphically presented in Figures A5 a-b, B5 a-b, and C5 a-b for systems PL1, PL2, and PL3, respectively.

The TSS and VSS concentrations of the effluent relative to the feed and the PA tank were lower for systems PL1 and PL2. This is an indication that a portion of the suspended matter in the influent does not pass through the system but is retained in the system. In system PL3 however, the TSS and VSS of the feed, PA tank and effluent are comparable. The net accumulation of TSS and VSS in PL3, on the other hand, was approximately zero.

5.2.8 Hydraulic Retention Time

The constancy of the hydraulic retention time (HRT) is reflected in the volumetric loading rate. Liquid retention time in the PA tanks was variable, as previously mentioned, due to operational constraints. Calculation of the PA tank HRT accounted for the fluctuation in liquid volume with the tank. A sample calculation is presented in Appendix D. The average HRTs over the course of experimentation excluding the start-up period were 3.4 ± 1.0 , 3.0 ± 0.5 , and 3.3 ± 0.6 hours in PA1 of PL1, PA2 of PL2, and PA3 of PL3, respectively. Graphs of the HRT versus operating time for the PA tanks are presented in Figure 5.3.

Maintenance of constant HRTs in the UASB reactors was feasible. The times of retention were 5.3 ± 0.7 , 5.2 ± 0.6 , and 5.1 ± 0.4 in UASB1 of PL1, UASB2 of PL2, and UASB3 of PL3, respectively. Graphs of the HRT versus operating time for the UASB reactors are presented in Figure 5.4 and a sample calculation is contained in Appendix D.

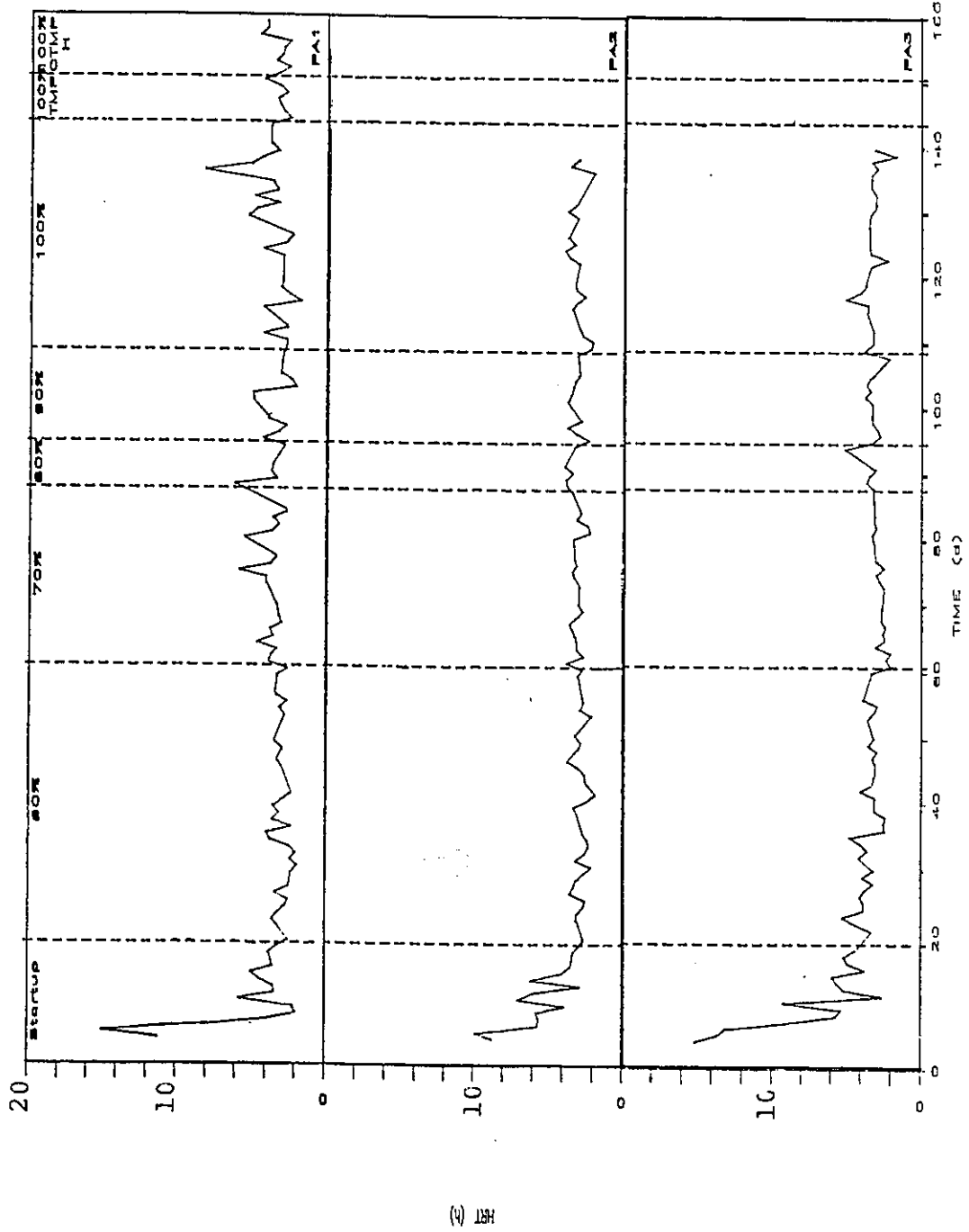


Figure 5.3 PA tank HRT versus operating time

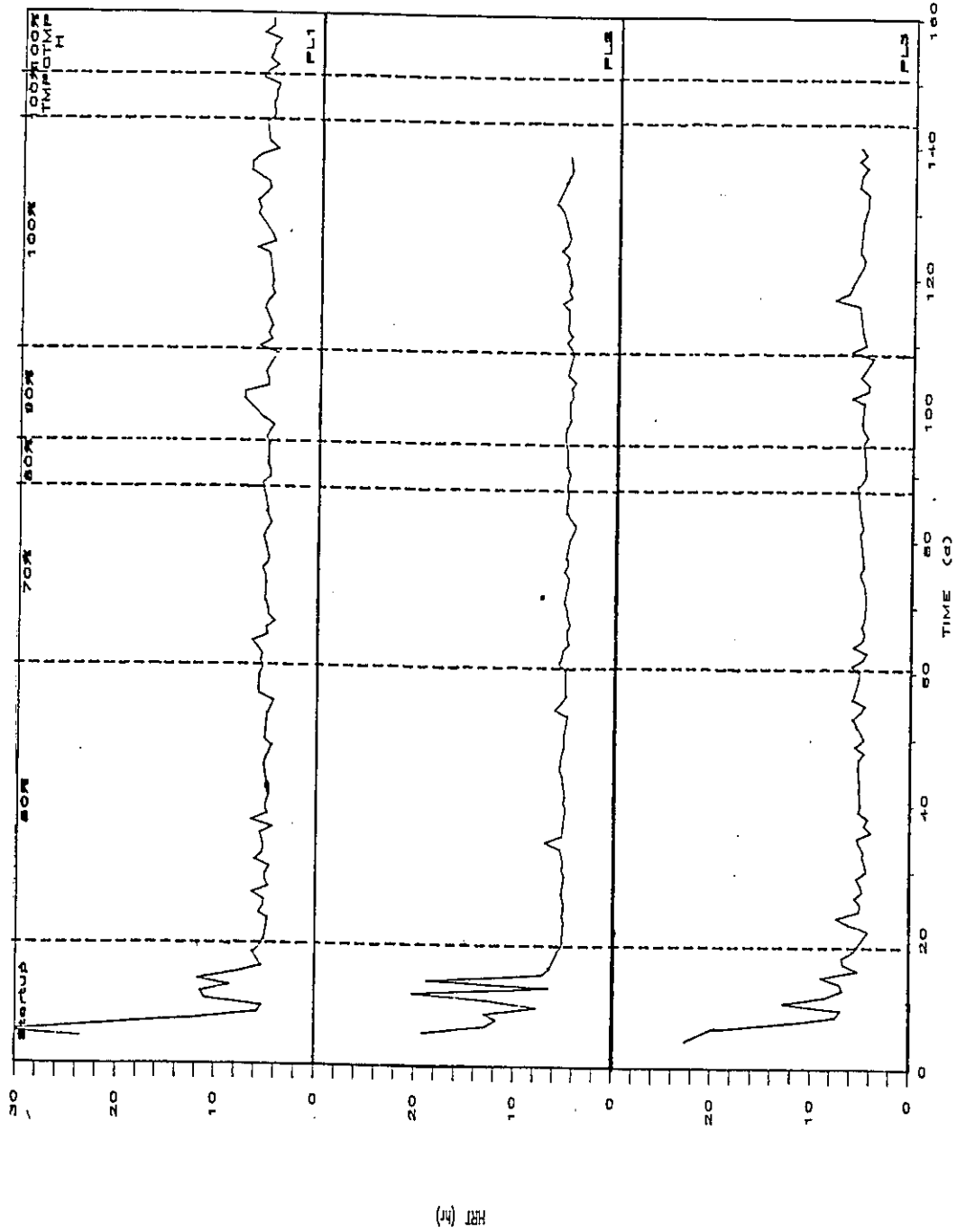


Figure 5.4 UASB reactor HRT versus operating time

5.2.9 Basis of UASB Reactor Performance Comparison

Overall UASB reactor performance must be compared on the same basis. PL1 received raw wastewater, PL3 received filtered wastewater, and PL2 received clarified wastewater. The wastewater data for PL2 after clarification, not the raw wastewater, are therefore appropriate for this comparison. The influent characteristics after the clarifier in PL2 were not measured, therefore these values must be calculated. The performance of the clarifier and its affect on the calculation of the influent characteristics is presented in the proceeding sections. The calculated wastewater characteristics after the clarifier are presented in Section 5.2.9.3.

5.2.9.1 PL2: Clarifier Sediment Removal

The volume of sediment removed daily from the clarifier is presented in Appendix B. A photograph of settled solids at the base of the clarifier in system PL2 is provided in Figure 5.5. The percent reduction of COD, TSS, and VSS by removal of solids from the influent by the clarifier is listed in Table 5.2. Sample calculations are presented in Appendix E.

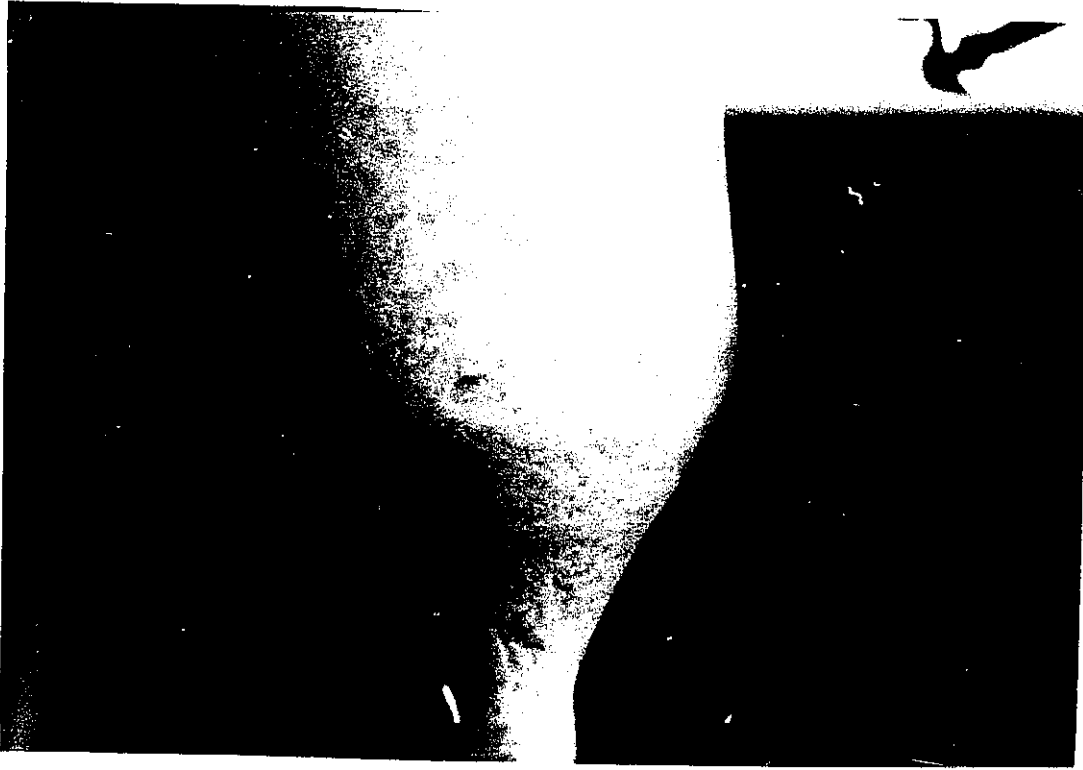


Figure 5.5 Photograph of settled solids at the base of the clarifier, system PL1

Table 5.2 Percent removal of COD and solids by the clarifier

% Wastewater Concentration	Removal by the Clarifier %			
	COD _T	COD _S	TSS	VSS
Start-up	20	2	33	36
50	10	1	14	15
70	12	1	10	11
80	18	3	19	20
90	10	2	20	21
100	10	1	9	9

Removal of suspended matter by the clarifier was highest during the start-up period when the volumetric loading rates were low (*i.e.* longer HRT relative to the design HRT). Throughout the remainder of the experiment, the clarifier removed approximately 10% of the feed COD_T on average except during feeding of 80% wastewater concentration when removal was higher (18%). The COD_S removal was minimal (1 - 3%).

Removal of TSS and VSS from the feed by the clarifier varied from 9 to 36% over the course of the experiment. The VSS reduction was 0 - 1% higher than the TSS removal under all loading conditions, excluding the start-up period.

Solids and COD removal were similar at 70, 80, and 100% wastewater concentration. Solids removal, however, was much greater than the COD_T removal during start-up, at the 50 and 90% wastewater concentration. The higher removals were attributed to longer hydraulic retention in the clarifier

during start-up and the variability of the fiber settleability.

5.2.9.2 Solubilization

The COD_s concentration of the sediment removed from the clarifier was greater than the COD_s concentration of the wastewater fed to the clarifier. This is a possible indication that solubilization of suspended COD occurred in the clarifier. The percent of COD_s generated from solubilization of suspended COD has been calculated for each loading period (Appendix E). The COD_s concentration of the feed and clarifier sediment as well as the calculated solubilized percentages are presented in Table 5.3.

Table 5.3 Solubilization

% Wastewater Concentration	COD _s		
	Feed mg/L	Sediment mg/L	Solubilization %
Start-up	2477	3040	19
50	2519	3614	30
70	3144	4660	33
80	3670	5276	30
90	4397	5610	22
100	5021	6219	19

5.2.9.3 PL2 Influent Wastewater Quality: After the Clarifier

The COD_T of the wastewater after the clarifier was expected to be lower than before the clarifier due to the removal of suspended COD (*i.e.* fiber/fines sediment). Calculation of the COD of the wastewater after the clarifier was based on the following premise. It was possible a larger percentage of the solubilization of suspended COD occurred in the sediment at the base of the clarifier where the suspended COD was concentrated rather than in the supernatant. If mixing between the liquid fraction in the sediment and the supernatant was poor, then the newly solubilized COD would have been removed from the system with the sediment when the sediment was withdrawn from the base of the clarifier. Therefore, even though solubilization did occur in the clarifier, the net result would be a decrease of both COD_T and COD_S across the clarifier.

The average COD and solids concentrations of the wastewater after the clarifier in system PL2 have been calculated (Appendix E) and are presented in the following table. These values will be used in subsequent evaluation of the performance of system PL2.

Table 5.4 Average calculated wastewater characteristics after the clarifier for system PL2

% Wastewater Concentration	COD _T mg/L	COD _S mg/L	TSS g/L	VSS g/L
Start-up	3511	2422	0.723	0.504
50	3881	2496	0.744	0.581
70	5025	3111	1.231	0.993
80	6527	3574	1.520	1.246
90	6581	4325	1.035	0.826
100	8361	4951	2.225	1.789

5.2.10 Organic Loading Rate

The average organic loading rates (OLR) applied to each system are graphically presented in Figure 5.6. The OLR_T of PL1 was higher than PL2 over the course of the experiment. This was attributable to the variation in the solids content of the feed. Similarly PL3, which had fiber removal by filtration, had approximately one half the OLR_T compared to the other systems. In other words, approximately half of the COD_T was present in the fiber in the wastewater.

The OLR_S of PL3 was less than both systems PL1 and PL2. Feed prepared for PL3 was left at room temperature for periods longer than just thaw time to settle the fiber prior to filtration. It is possible the wastewater partially degraded during this interval resulting in a reduced COD_S. Also, it is possible filtration of CTMP-N wastewater with 20 um filters more effectively removes fine suspended matter than centrifugation, as in the COD_S assay.

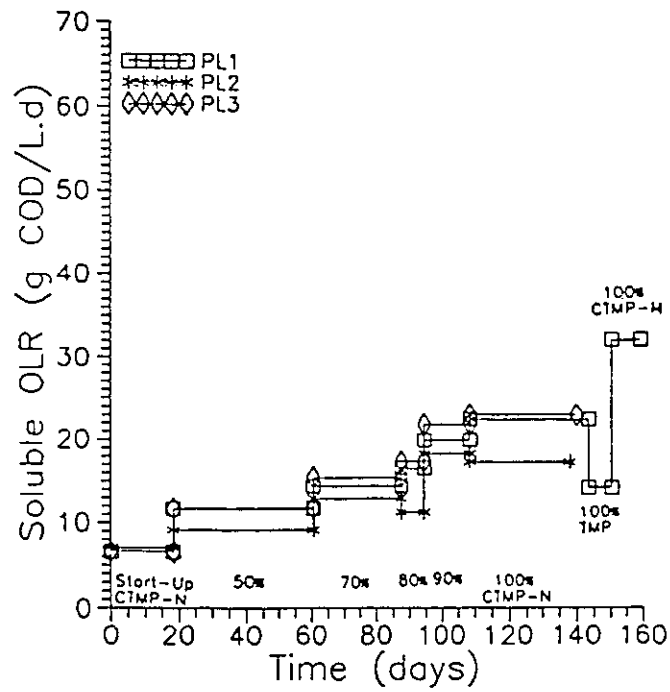
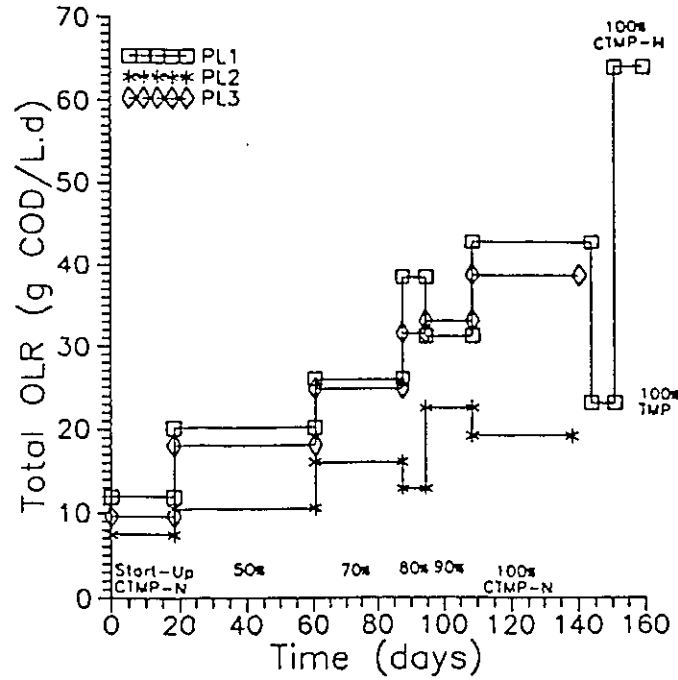


Figure 5.6 Average OLR versus operating time

In summary, removal of the fiber by clarification (PL2) resulted in 0 - 10% higher COD_S and 0 - 19% lower COD_T relative to the raw wastewater (PL1). Removal of fiber by filtration (PL3) resulted in 0 - 32% lower COD_S and 28 - 66% lower COD_T . Since the flow rate was kept constant, systems PL2 and PL3 received lower total organic loadings than system PL1 at identical waste dilutions, however, the COD_S loadings were comparable for all three systems. The COD_S rather than COD_T provided the best basis for a relative performance comparison of the three systems.

The OLR did not always increase linearly with increases in the percent wastewater concentration (Table 5.5, Figure 5.7). This was the result of variations in the composition and quality of the wastewater from barrel to barrel. The OLR will be used as the integer variable (x-axis) instead of the percent wastewater concentration of the feed, in further analysis, due to the non-linearity of the OLR relative to the percent wastewater concentration.

Table 5.5 Average OLR (g COD/L·d)

COD _T	Start-Up	50%	CTMP-N				TMP %100	CTMP-H %100
			70%	80%	90%	100%		
PL1	11.9	20.2	26.0	38.4	31.2	40.5	23.1	64.0
PL2	9.6	18.1	24.9	31.6	33.0	38.7	-	-
PL3	7.5	10.5	16.0	12.9	22.5	19.2	-	-
COD _S								
PL1	6.6	11.7	14.4	16.5	19.8	21.5	14.2	32.1
PL2	6.6	11.6	15.4	17.3	21.7	22.9	-	-
PL3	7.0	9.1	12.9	11.2	18.2	17.2	-	-

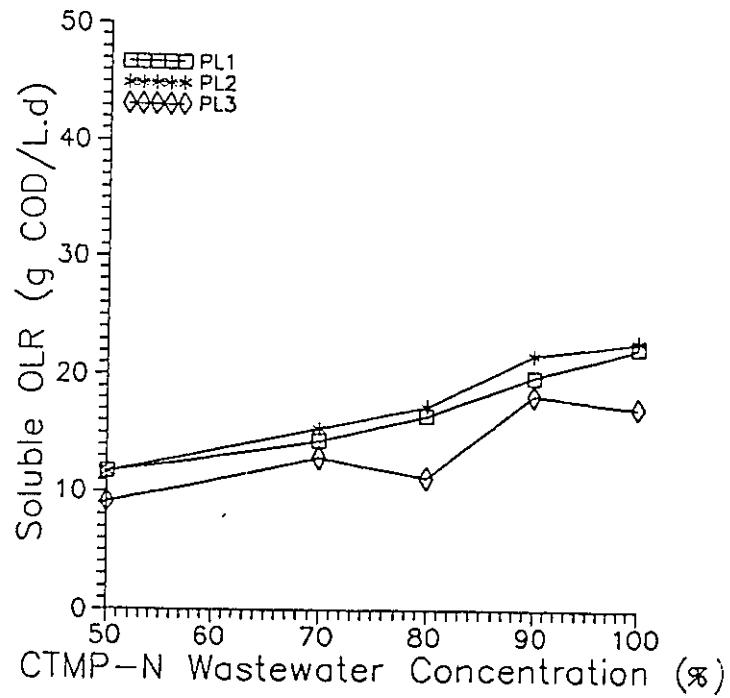
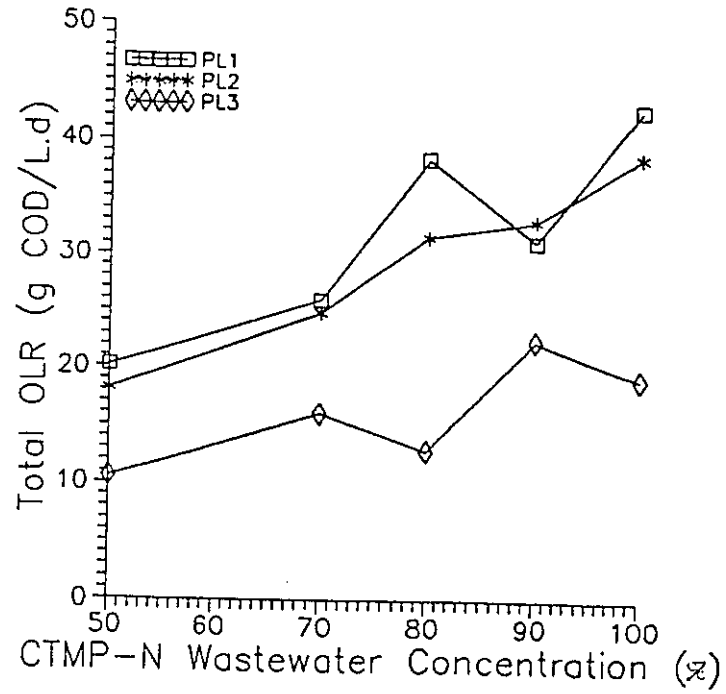


Figure 5.7 Average OLR versus percent wastewater concentration

5.2.11 COD Removal Efficiency

Unaltered (PL1), clarified (PL2), and fiber-free (PL3) CTMP-N wastewaters were treated successfully in the two-stage system with and 33 - 50% COD_T and 33 - 52% COD_S removal efficiencies (Table 5.6, Figure 5.8). The COD_S removal efficiency of system PL3 was superior to that of systems PL1 and PL2 at all wastewater dilutions. All three systems showed gradual improvement in COD_S removal as the wastewater concentration increased to 80%. A subsequent decrease was observed in all systems as the wastewater concentration was increased to 90 and 100%. A similar trend was observed for COD_T removal for systems PL1 and PL2. The COD_T removal for system PL3 varied from 40 to 48%.

Table 5.6 Average COD removal percentage

COD _T	CTMP-N					TMP %100	CTMP-H %100
	50%	70%	80%	90%	100%		
PL1	37	37	50	43	38	29	23
PL2	38	40	50	33	36	-	-
PL3	42	46	40	48	40	-	-
COD _S							
PL1	35	37	48	46	38	41	34
PL2	38	40	49	43	33	-	-
PL3	41	47	52	49	42	-	-

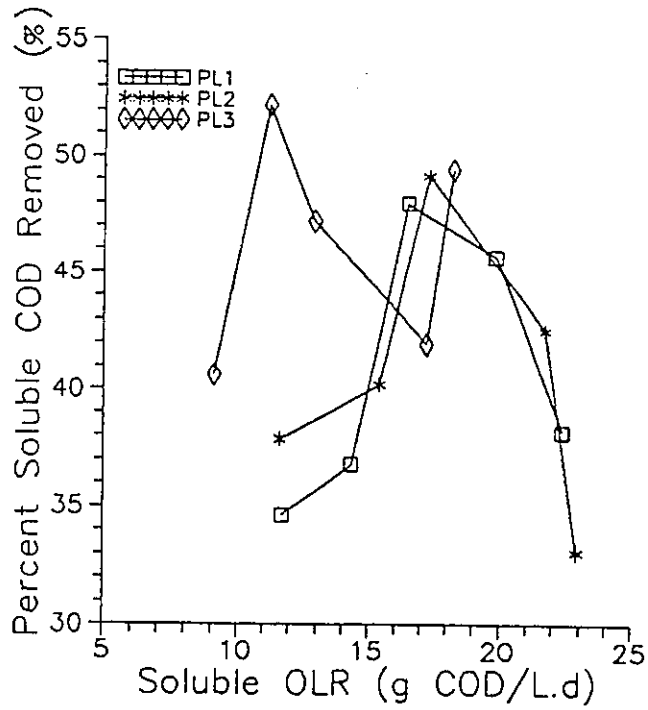
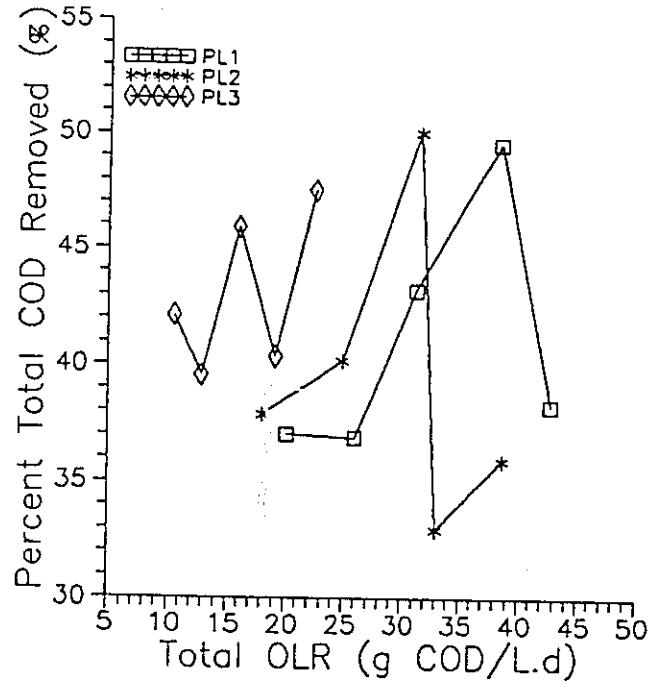


Figure 5.8 Average COD Removal Percentage versus the average OLR

System PL1 was able to treat raw wastewater and had no problem switching from CTMP-N to TMP to CTMP-H wastewater. The percent COD_T removal efficiency on these two alternate wastes was lower than for the CTMP-N. The percent COD_S removal efficiency while treating TMP was comparable to CTMP-N wastewater. The percent COD_S removal efficiency was lowest for the treatment of CTMP-H wastewater.

The COD_T removal efficiency in system PL1 increased from approximately 37 to 50% as the OLR_T was increased from 20 to 38 g $COD_T/L \cdot d$. A subsequent decrease to 38% COD_T removal efficiency was observed as the OLR_T was further increased to 43 g $COD_T/L \cdot d$. The COD_T removal efficiency in system PL2 increased from approximately 38 to 50% as the OLR_T was increased from 18 to 32 g $COD_T/L \cdot d$. A subsequent decrease to approximately 34.5% COD_T removal efficiency was observed as the OLR_T was further increased to 39 g $COD_T/L \cdot d$. The COD_T removal efficiency of system PL3 varied between approximately 40 and 48% over a range of OLR_S from 9 to 18 g $CODS/L \cdot d$.

The COD_S removal efficiency in systems PL1 and PL2 increased from approximately 36 to 42% until the OLR_S reached approximately 17 g $COD_S/L \cdot d$. A subsequent decrease of COD_S removal efficiency was observed as the OLR_S was increased above this value. The COD_S removal efficiency of system PL3 varied between 40 and 50% at all OLR_S .

5.2.12 Biogas Production

Systems PL1 and PL2 produced similar daily volumes of biogas (Table 5.7). Biogas production by system PL3 was comparable to systems PL1 and PL2 during feeding of 50% and 70% wastewater concentration. The low volumes measured for system PL3 at 80% to 100% wastewater concentration was due to a faulty gas meter and operation errors (e.g. feed pump off). The percentage of CH₄ in the biogas was consistent from system to system and constant (75 - 79%). Nitrogen, when detected, was due to leakage from the atmosphere. Trace amounts of hydrogen sulfide were also present but not measured. Typically, in a properly operating digester treating medium to high strength wastewater, the composition of biogas measures about 65% CH₄ and 35% CO₂/N₂ mix (Grady and Lim, 1980).

Table 5.7 Biogas production

System	50%	70%	CTMP-N		100%	TMP	CTMP-H
			80%	90%		%100	%100
Volumetric Biogas Production Rate (L/d)							
PL1	6.8	10.4	13.9	16.6	16.2	9.8	14.1
PL2	8.2	12.0	13.0	16.6	14.6	-	-
PL3	5.9	10.2	>8.8*	>9.8*	>8.4*	-	-
Biogas Methane Content (%CH ₄)							
PL1	77	77	75	76	75	76	70
PL2	78	78	78	75	76	-	-
PL3	79	78	78	77	76	-	-

* measured volumes low due to a faulty gas meter and/or system operational error (e.g. feed pump off)

5.2.13 Fiber Accumulation and Sludge Granularity

5.2.13.1 Fiber Accumulation

In system PL3 the bed appeared to be well mixed and of relatively stable volume. The granules remained black in colour with a shiny surface (Figure 5.9) during the experiment. Systems PL1 and PL2 underwent changes during the course of the study. Fiber accumulated in the UASB reactors of systems PL1 and PL2 in a similar pattern, however, accumulation was more extreme in PL1.

Fibers, pinkish-orange in colour, initially passed through the sludge bed and accumulated on top of the bed or passed through the system (Day 5). Fiber progressively accumulated in the bed in clumps while the layer of fiber on top of the bed grew and became greyish in colour (Day 6 to 22). The maximum fiber accumulation at the top of the sludge beds was 1.0 L and 0.5 L in UASB1 and UASB2 of systems PL1 and PL2, respectively (Figures 5.10).

A brown fibrous scum layer developed around the bio-rings (Day 20) in UASB1 and UASB2 and remained there throughout the experiment (Figure 5.11). The scum layer plugged the effluent overflow ports of the UASB reactors in PL1 and PL2 (Day 22 and 40). This caused the UASB reactors to fill up with wastewater. A reactor filled until the hydrostatic pressure created by the liquid above the overflow port exceeded the strength of the plug. When this occurred, the plug was subsequently forced out and the ex-

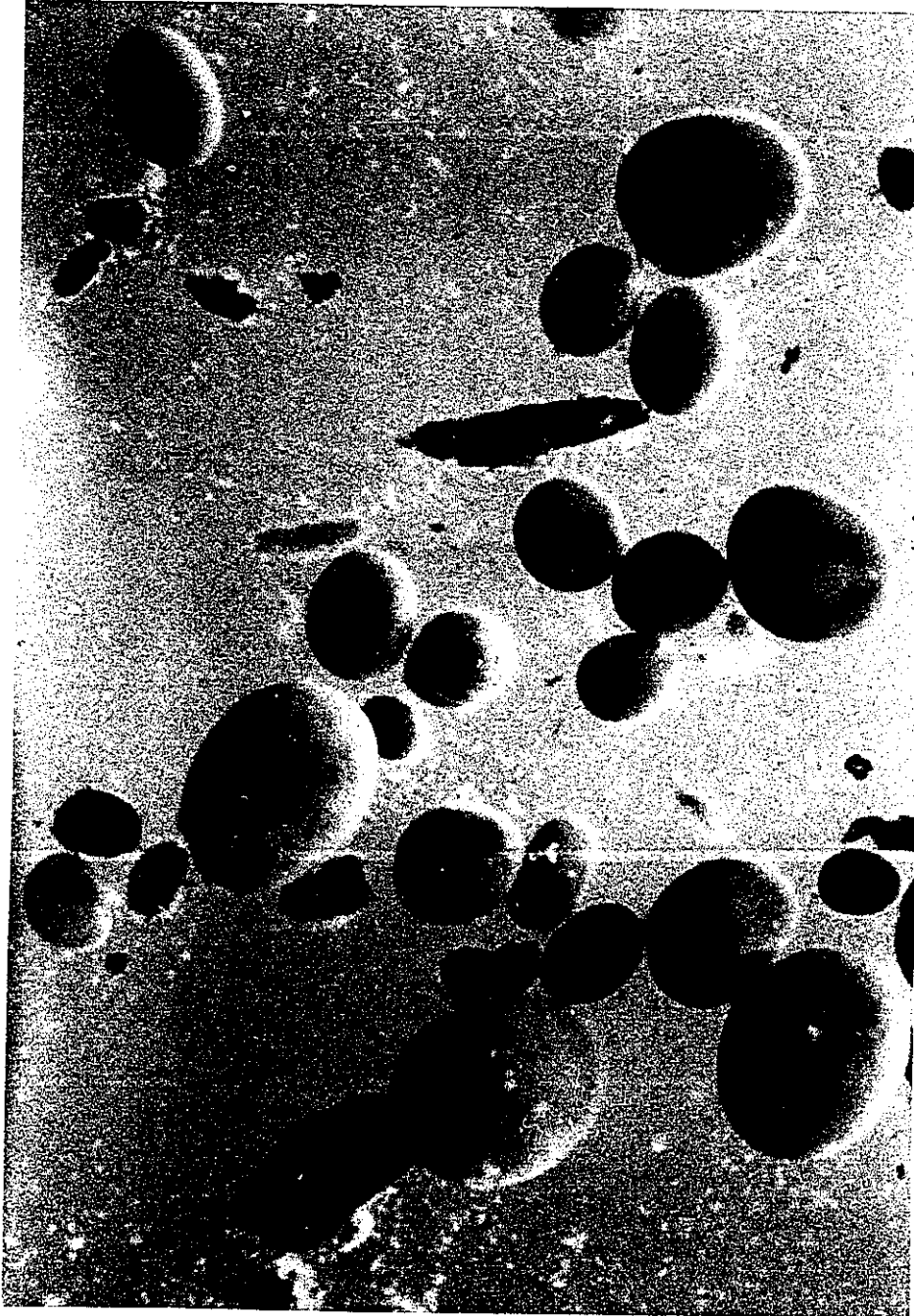


Figure 5.9 Seed granules (magnified x5)



Figure 5.10 Layer of fiber on top of sludge bed (PL1)

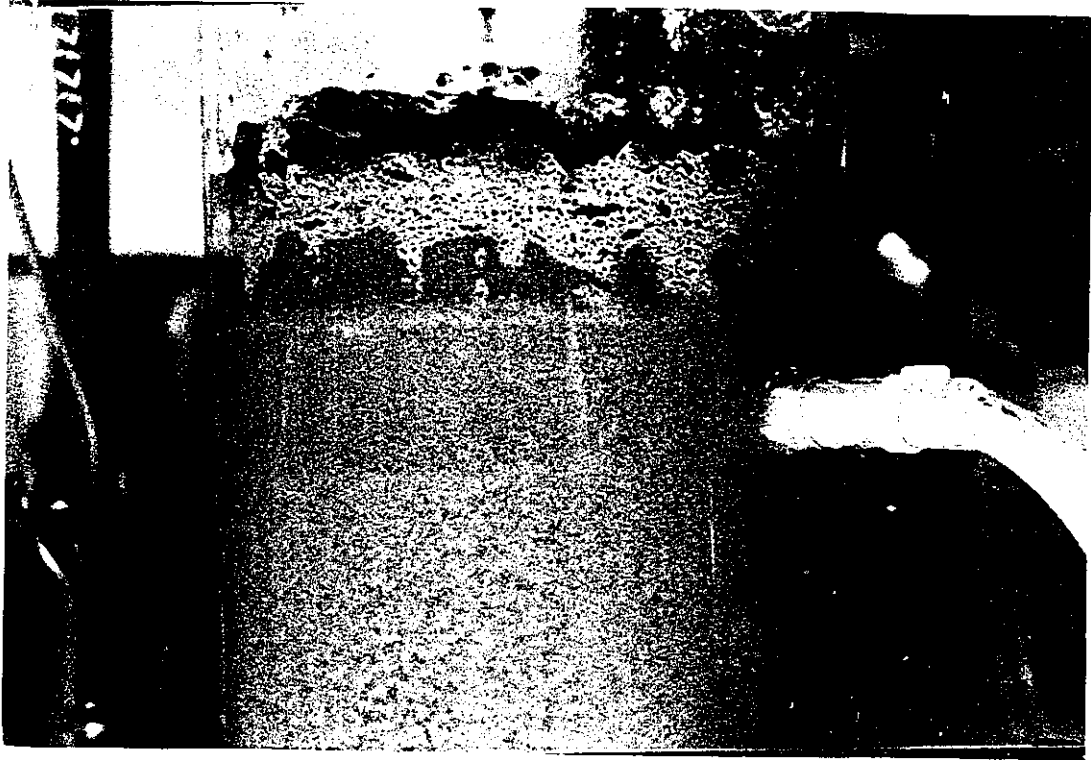


Figure 5.11 Brown scum

cess liquid discharged from the system rapidly. The sludge beds were severely disturbed by the rapid washout. This caused the fiber layer on top of the beds to be washed out. The fiber layer in systems PL1 and PL2 were subject to complete washout twice. A rapid decrease of the bed volume on approximately Days 20 and 40 of operation is illustrated in Figure 5.12.

The tendency for fiber to accumulate on top of the sludge beds was significantly reduced after the initial washout. Fiber then tended to accumulate more uniformly throughout the sludge beds. Visual observation indicated there was channeling and minimal mixing of the sludge beds and fiber before the initial washout occurred, whereas afterwards, the beds were well mixed. The changes in the reactors' VSS concentrations is illustrated in Figure 5.13.

A loose pink coating of fiber formed on the granules in systems PL1 and PL2 (Figure 5.14). The quantity of coated granules increased with time.

Absence of fiber in the feed of system PL3 resulted in a more stable and well mixed bed. The biogas was observed to bubble through the bed and carry some granules upwards (Day 92). Granules periodically entered the recycle line and were crushed by the recycle pump. A fine layer of crushed granules (0.05 L) developed on top of the sludge bed.

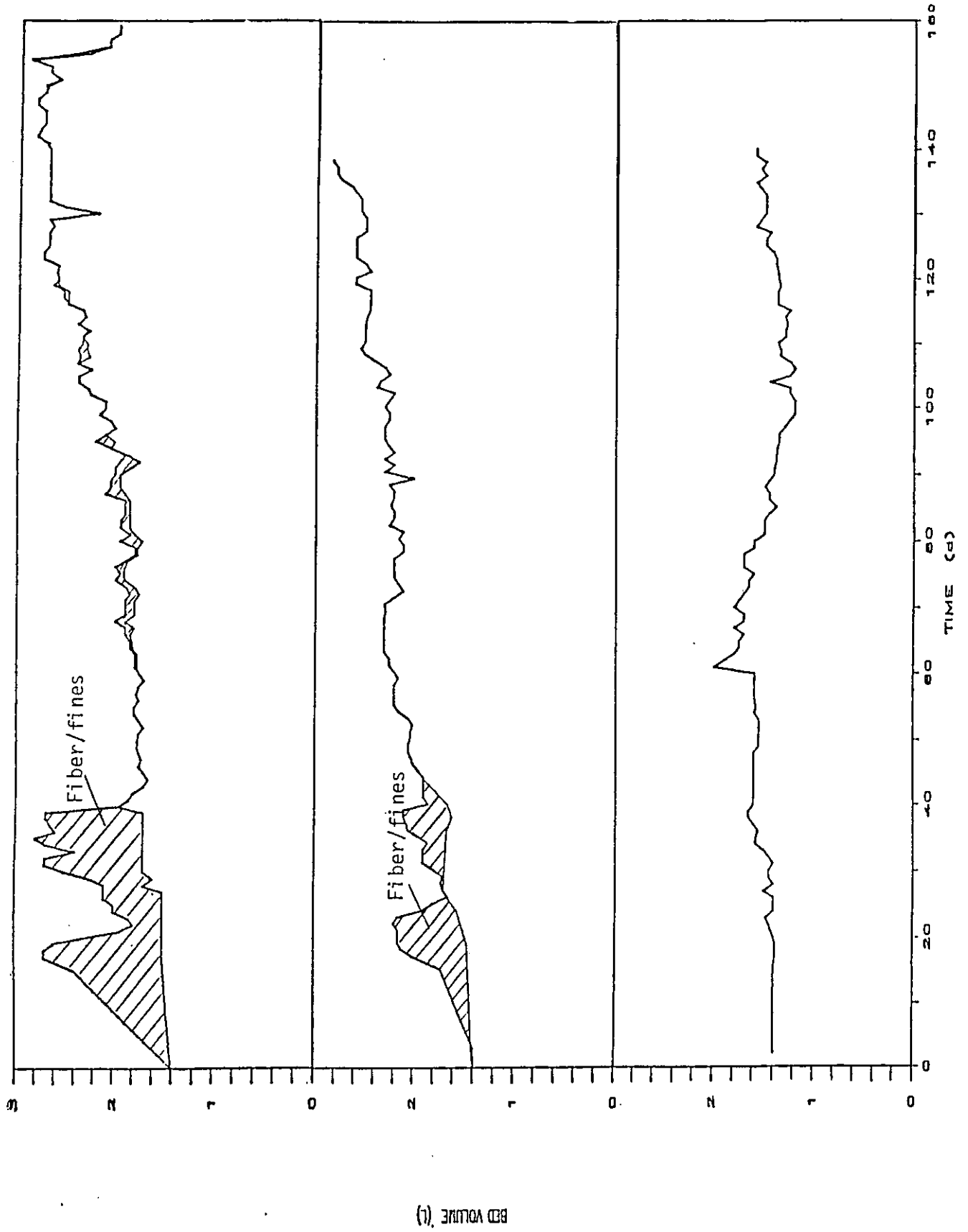


Figure 5.12 Sludge bed volume versus operating time

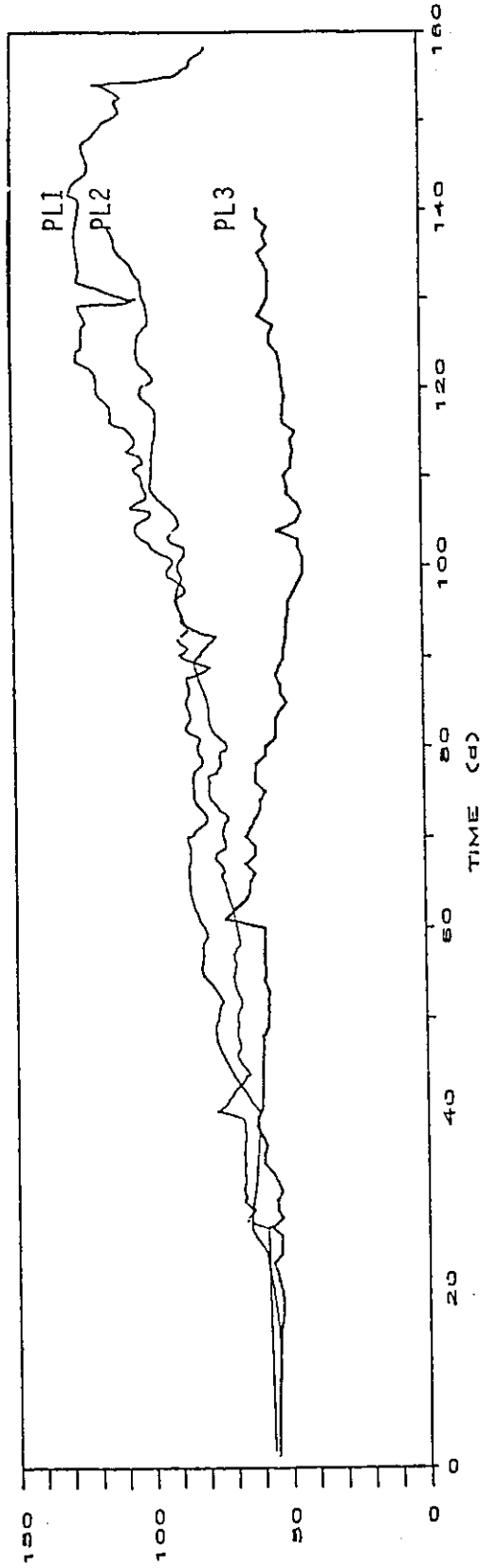


Figure 5.13 Reactor VSS versus operating time

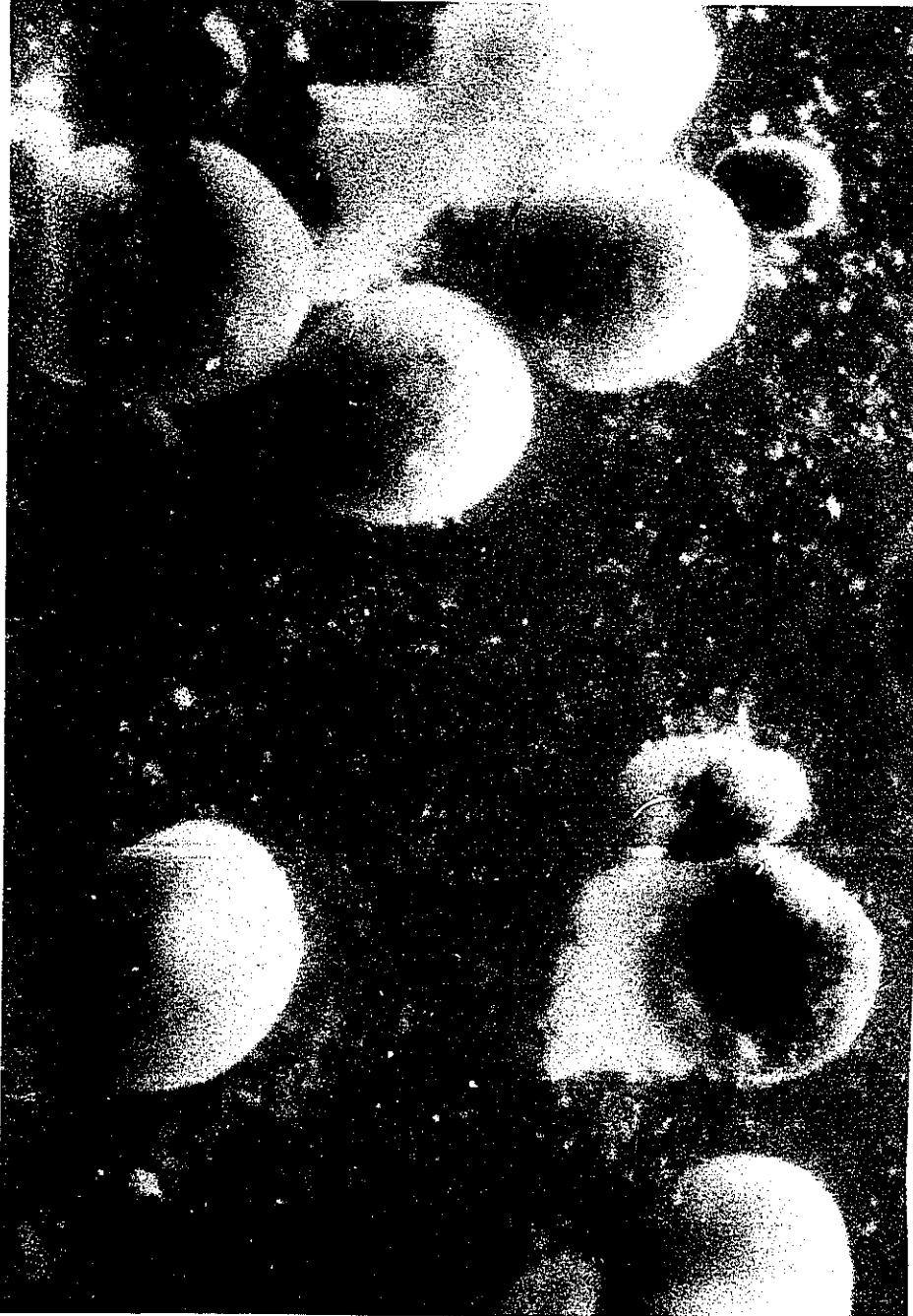


Figure 5.14 Granules coated with fiber (magnified x5)

The sludge bed VSS increase observed in both systems PL1 and PL2 was clearly due partially to fiber incorporation into the bed. Interpreting the measured large increases of the sludge bed VSS as biomass growth would not be valid. Biomass yield will be based on system PL3 because it was not exposed to fiber.

5.2.13.2 Biomass Yield

During the 140 days of operation, the total biomass in system PL3 showed little increase, as measured by VSS of the sludge bed (Table 5.8). Typical anaerobic sludge yields from 0.08 to 0.23 g VSS/g COD_S removed (Henze and Harremoes, 1982), depending on the type of wastewater, have been reported. The net biomass yield in system PL3 was 0.025 g VSS/g COD_S removed. The biomass yield from the on site pilot study was also reportedly low (de Vegt, 1988). If the net biomass yield measured in this study and observed in the pilot study signals a low yield that is typical of anaerobic treatment of CTMP wastewater, and if the low yield is coupled with biomass washout, then maintaining a constant reactor biomass could be a significant problem in full-scale operation. A possible solution to this problem may be the implementation of a system for the collection and storage of washed out biomass to be recirculated into the UASB or for reinoculation could be advocated in full scale plants.

Table 5.8 Total reactor VSS (g)

System	Day 0	Day 60	Day 80	Day 138	Day 159
PL1	55.5	71.5	74.8	131.6	81.7
PL2	55.5	80.4	81.0	110.8	-
PL3	55.5	58.6	57.6	59.4	-

5.2.13.3 Sludge Granularity

Sludge granularity was assessed with the settleability test. A sludge sample was exposed to successively increasing upflow velocities and a washout profile was determined. The basis of the evaluation is the V_{50} , the upflow velocity at which 50% of the sample (as VSS) is washed out. Higher V_{50} indicates better settling sludge.

The test was performed on the seed sludge and at various stages of the experiment on samples taken from both the bottom and top of the sludge bed. The sludge beds were tested at the end of the 50, 70, and 100% runs on Day 47, 80, and 138, respectively. The cumulative settling velocity profiles for the sludge samples taken from the reactor top and bottom are illustrated in Figure 5.15 a-c. The average of the top and bottom cumulative settling velocity profiles are illustrated in Figure 5.16. The V_{50} results are presented in Table 5.9.

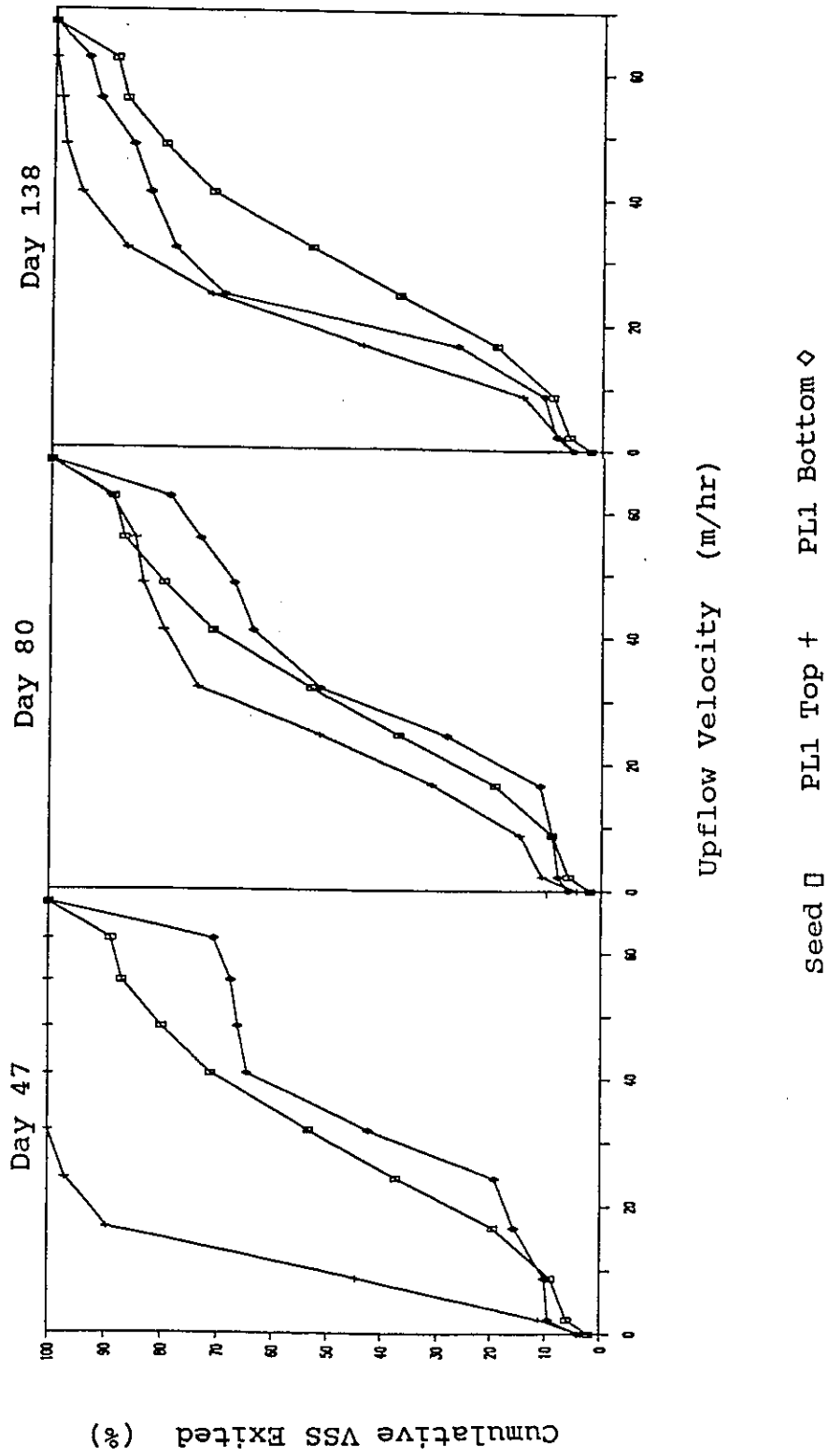


Figure 5.15a PL1: sludge bed settleability profiles, top and bottom

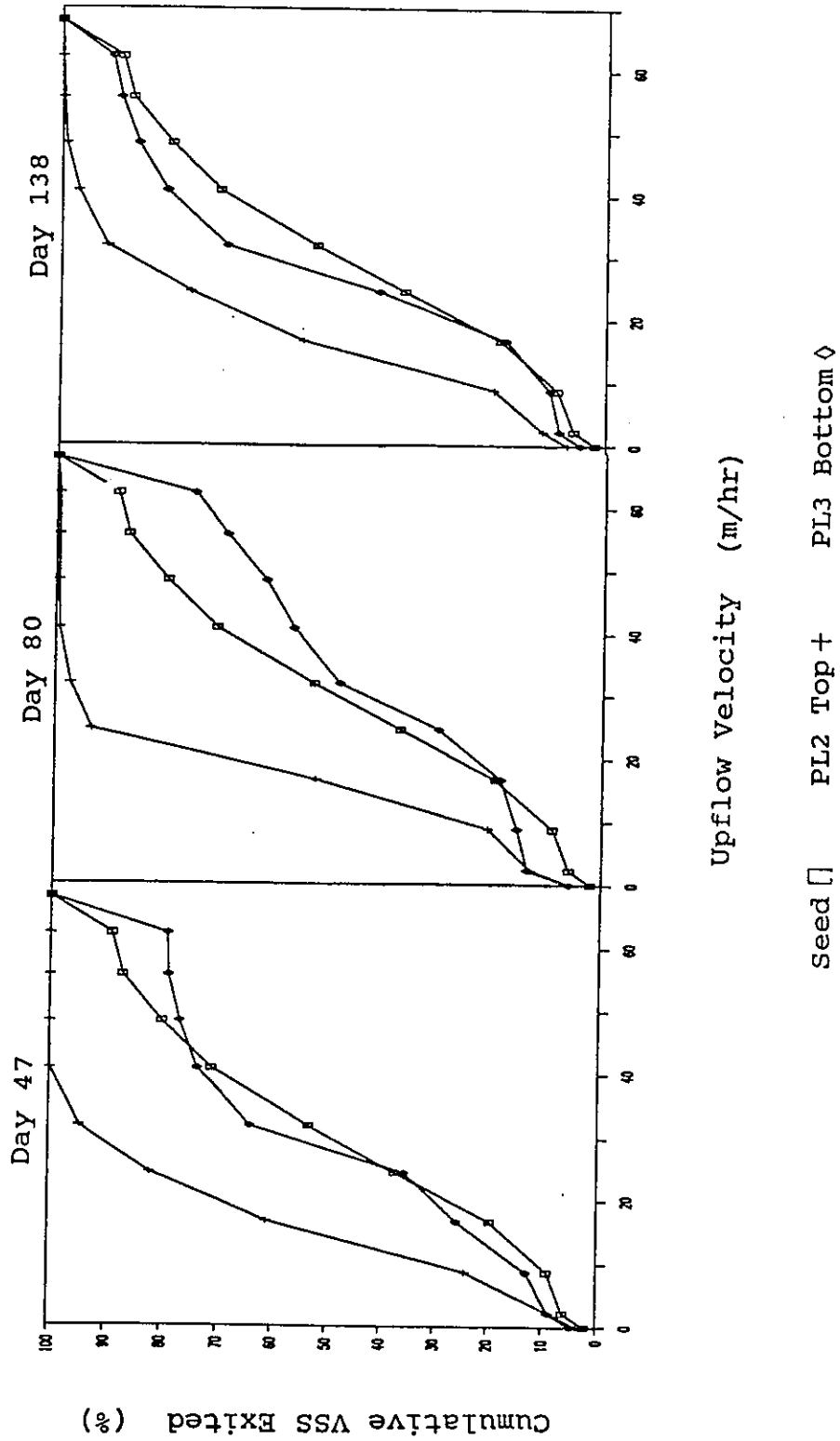


Figure 5.15b PL2: sludge bed settleability profiles, top and bottom

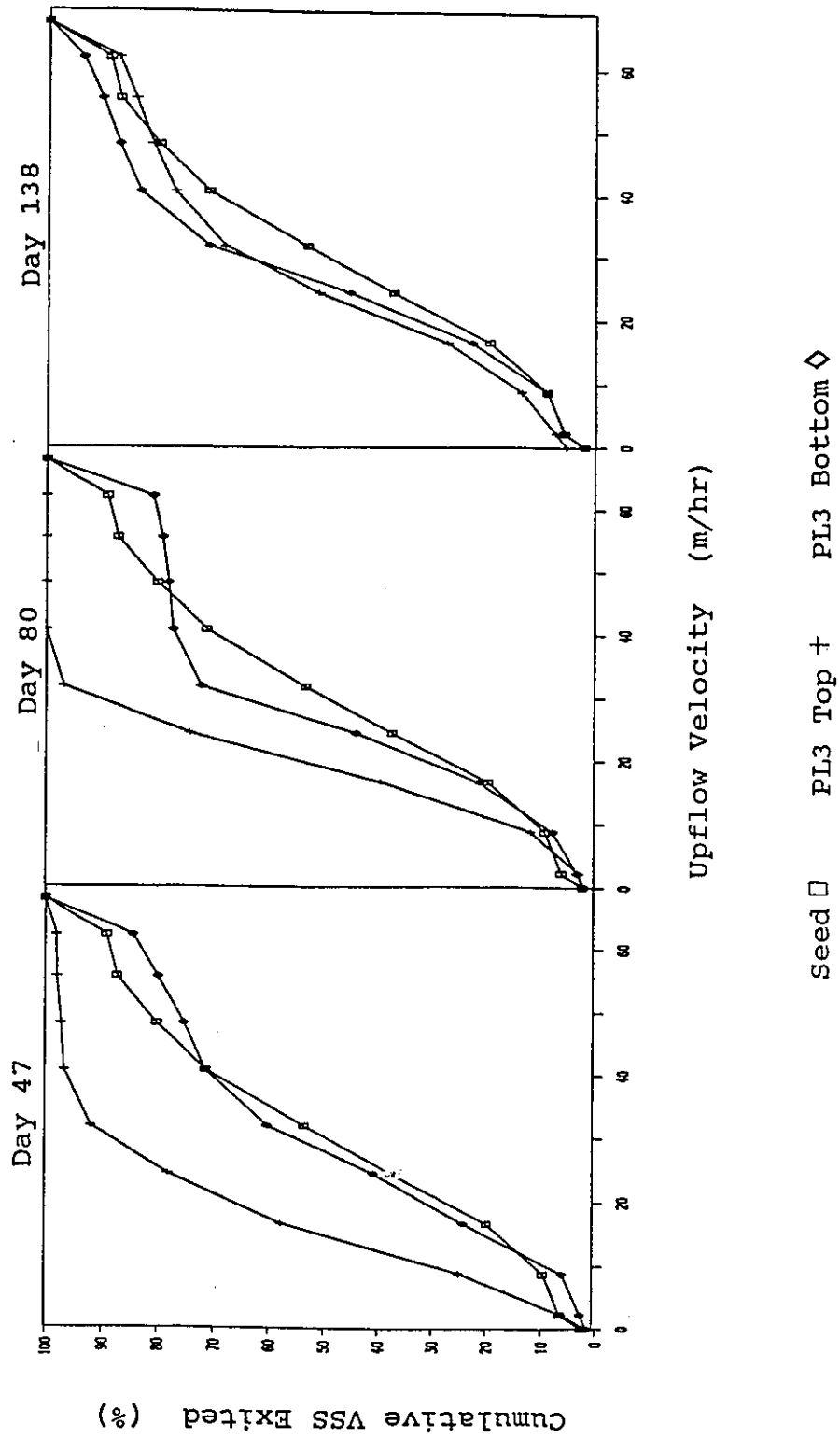


Figure 5.15c PL3: sludge bed settleability profiles, top and bottom

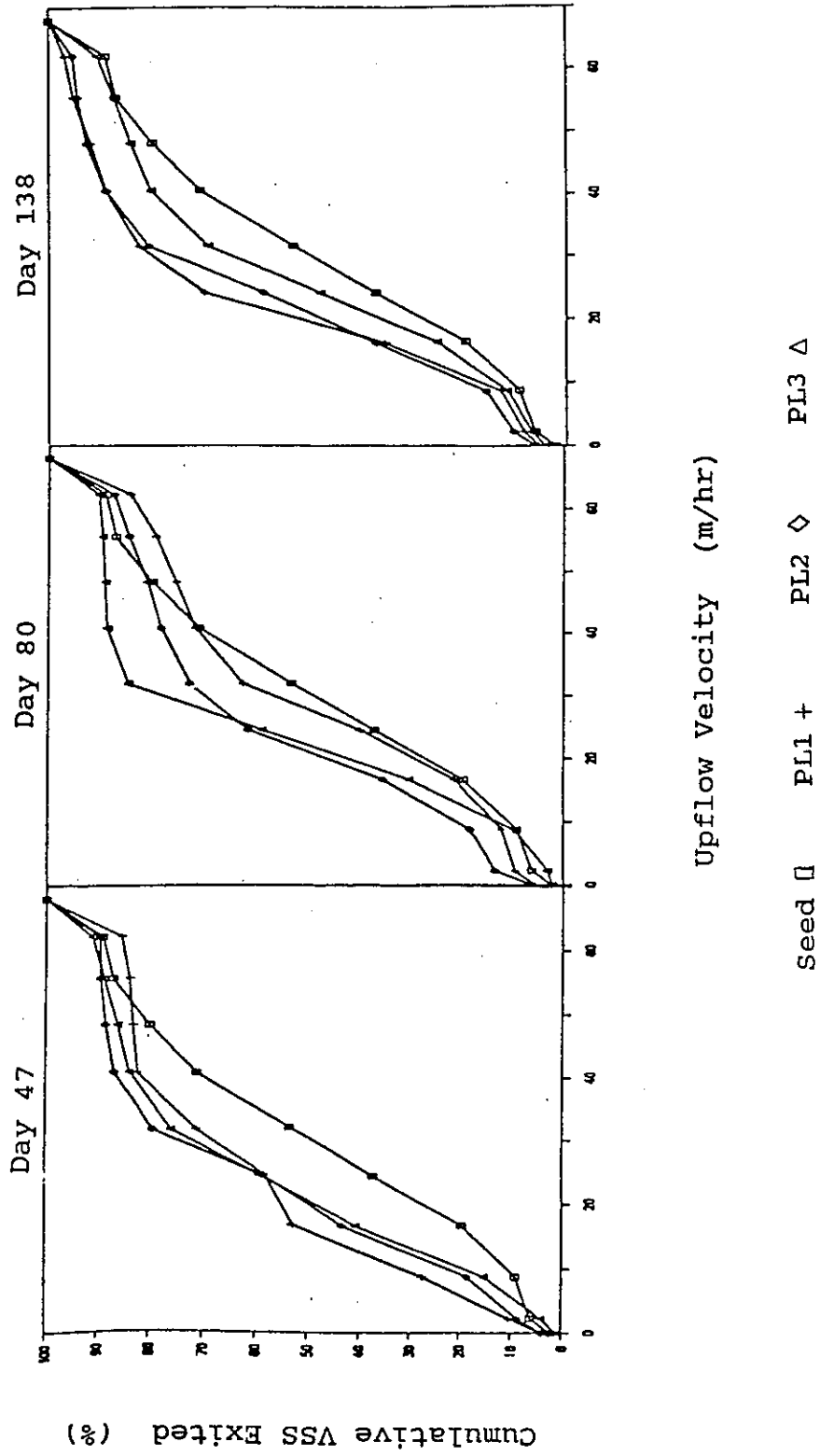


Figure 5.16 Sludge bed average settleability profiles

Changes in settleability are relative to the seed. Fines accumulation in the bed affects the interpretation of the settleability tests results. The V_{50} for sludge from the bottom of PL1, on day 47, was slightly greater than the seed.

Table 5.9 Sludge bed settleability; V_{50}

Sludge	Reactor Bottom			Reactor Top			Average			
	Day 0	Day 47	Day 80	Day 138	Day 47	Day 80	Day 138	Day 47	Day 80	Day 138
	50%	70%	100%	50%	70%	100%	50%	70%	100%	
Seed	30									
PL1	35	31	21	10	24	18	16	28	20	
PL2	28	33	27	14	16	15	20	21	21	
PL3	28	26	27	15	19	24	21	22	25	

Note: The V_{50} averages are not the averages of the calculated V_{50} values but were determined from the average of the top and bottom curves.

The value for the top of the bed was significantly less than the seed. V_{50} values of the top and bottom of a bed that are different implies bed non-homogeneity. This was the case at that time for PL1; the bed was stratified with larger granules and a small quantity of fiber on the bed bottom and small granules with a large quantity of fiber intermixed at the top.

Stratification of the bed diminished as bed mixing improved; the sludge from the top of the bed had slightly improved settling and the bottom slightly less. The sludge

bed in system PL1 by Day 138 was homogeneous. However, as more fiber was retained in the bed and on the granules, the overall settleability diminished. Although the fiber coating increased granule size, the larger coated granules tended to break up thereby reducing the overall settleability of the bed.

The granularity of the sludge in system PL2 was comparable to that in PL1 on Day 47 and the sludge granules and fiber were a homogeneous mixture by Day 80. A distinctly separate phase of fines on top of the sludge bed was not visible, however, the difference in settleability between samples from the top and bottom of the reactor bed was significant. Therefore, the bed was still stratified. A large difference between the settleability of the sludge granules from the top and bottom of the bed was measured on Day 138 as well. A noticeable deterioration in granule settleability of sludge from the bottom of the bed had occurred.

A decrease in the settleability of PL3 sludge was measured on Day 47 of reactor operation. This could be attributed to the growth of biofilm in the PA tank (small flocculant biomass) which was transferred to and retained in the bed. Or possibly, granules which were observed to enter the recycle line were crushed by the recycle pump. The bed, nonetheless, was homogeneous based on settleability, size, and colour of granules. Settleability of granules removed from the bottom of the reactor improved by Day 80 (compared

to the seed) but granules from the top of the sludge bed had deteriorated. Observation indicated the bed was stratified and more granules appeared to have been crushed. The settleability measured on Day 138 was comparable to the seed.

Initially, attachment of fiber to sludge granules appeared beneficial, however, an overall deterioration of the settling profile of systems PL1 and PL2 was observed at 100% wastewater.

In summary, fiber can cause the physical deterioration of granules and can severely disrupt biomass retention in an UASB system. Apparently, presence of fiber in the feed led to incorporation of the fiber into the sludge bed, which in turn resulted in increased bed volume and decreased granule settleability. These factors increase the likelihood of occasional biomass washout from the reactor. The control of fiber entering the two-stage treatment system is a high priority problem which requires an effective solution for operational success.

5.2.14 Biomass Activity

5.2.14.1 Modified Batch Activity Tests

Sludge bed modified batch specific activity tests (MBAT) were performed at the end of the 50, 70, and 100% runs on Day 59, 80, and 138, respectively. Sludge samples were removed from the top and bottom of the bed and combined in equal volumes to provide an average sample representative of the entire bed. Specific activity, based on CTMP-N wastewater consumption and an acetate spike, was investigated for the 50 and 70% waste concentration runs. Specific activity based on CTMP-N wastewater and acetate, methanol, formate and glucose spikes were investigated for the 100% waste concentration run. Rates are CH_4 production, or activity rates, expressed as g COD/g VSS·d. All tests were performed in duplicate.

MBAT: CTMP-N Wastewater Consumption

Methane production from stabilization of the wastewater in the MBAT (Table 5.10, Figure 5.17) increased for the sludge from system PL1 over the course of the experiment. The PL2 sludge activity on wastewater increased relative to the seed and was stable after the 70% wastewater concentration run. The PL3 sludge activity was comparable to the PL2 sludge on Day 59 and 80. However, the measured activity of PL3 sludge on Day 138 had diminished. The PL1

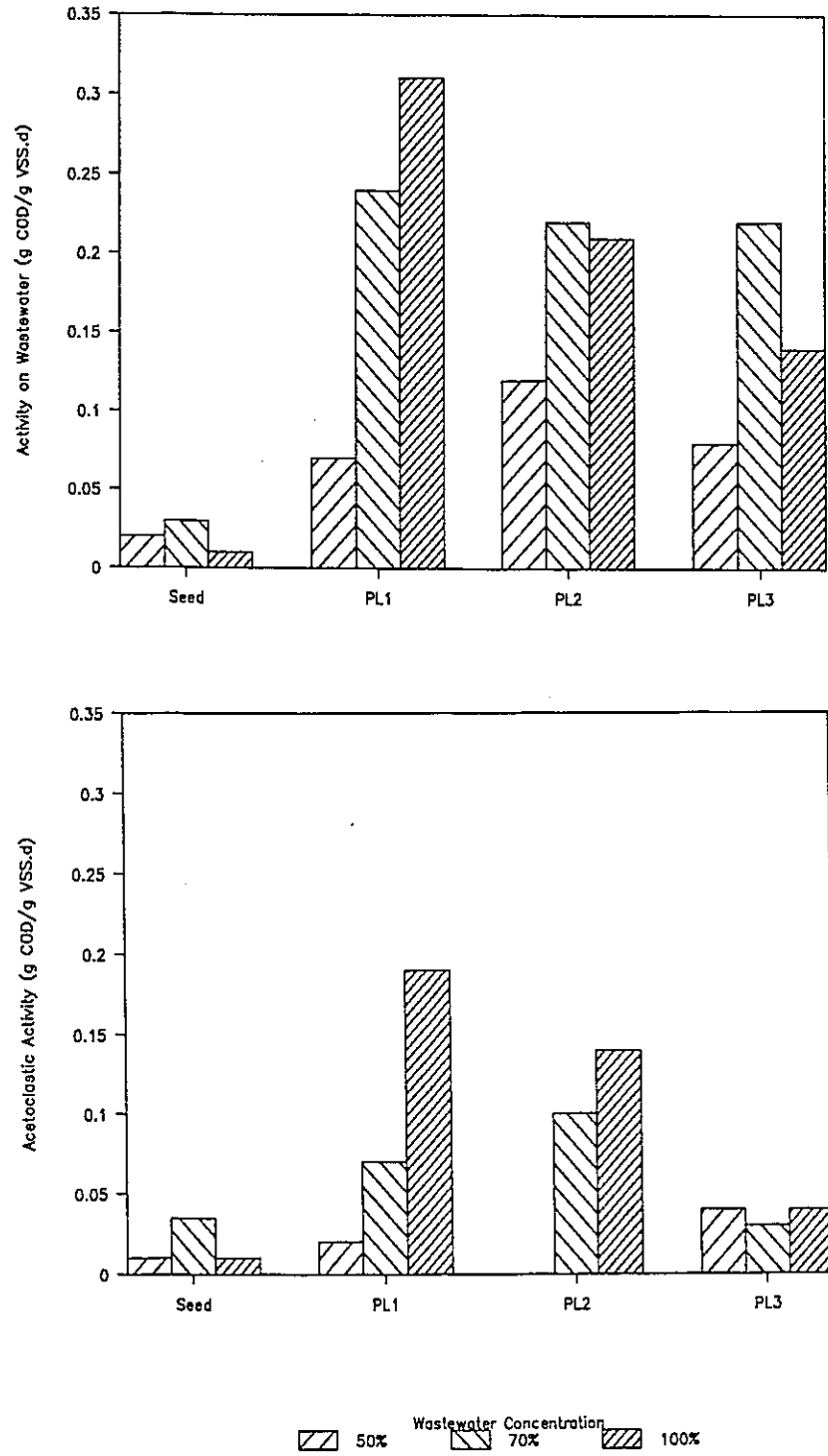


Figure 5.17 Specific activity: CTMP-N wastewater and acetate

sludge displayed the highest activities in batch (maximum 0.31) and the activity of PL2 sludge was greater than PL3.

Table 5.10 MBAT: CTMP-N wastewater consumption

Day	% Wastewater Concentration	Wastewater Activity (g COD/g VSS·d)			
		Seed	PL1	PL2	PL3
59	50	0.02	0.07	0.12	0.08
80	70	0.03	0.24	0.22	0.22
138	100	0.01	0.31	0.21	0.14

Note: The content of the wastewater in the serum bottles in the MBATs was 60% (volume/volume) unaltered CTMP-N. It is possible that the test wastewater was inhibitive to the sludges which had not been exposed to fiber or this concentration of fiber (*i.e.* at 50% wastewater concentration) and organic loading.

Assuming an average seed activity of 0.02 g COD/g VSS·d, the final activities of PL1, PL2, and PL3 on CTMP-N wastewater were 15, 10, and 7 times greater, respectively, than the activity of the seed sludge. In general, the sludges became better acclimatized to the CTMP-N with continued exposure.

The results indicate that sludge not exposed to fiber degrade fiber-containing wastewater at a lower rate than sludge that was acclimatized to fiber. The difference was insignificant after 87 days of operation (70%), but became apparent after 140 days of operation (after 100%). The significance of these results to full-scale plant operation is

that a sudden inhibition (*i.e.* decrease of activity) may be expected when anaerobic sludge which typically treats fiber-free wastewater is exposed to fiber-containing wastewater (*i.e.* a shock-load).

MBAT: Acetate Consumption

The specific acetoclastic activity increased for the PL1 and PL2 sludges during the course of the experiment, however, the PL3 sludge remained constant at approximately four times higher than the seed sludge (Figure 5.17, Table 5.11). The PL1 sludge displayed the highest levels of activity on acetate.

Table 5.11 MBAT: acetate consumption

Day	% Wastewater Concentration	Acetoclastic Activity (g COD/g VSS·d)			
		Seed	PL1	PL2	PL3
59	50	0.01	0.02	na	0.04
80	70	0.35*	0.07	0.10	0.03
138	100	0.01	0.19	0.14	0.04

* unusually high value
na not available

MBAT: Formate, Glucose, and Methanol

The sludge from the three systems were able to metabolize formate in the MBAT (Table 5.12, Figure 5.18). The ranking of the reactors based on formate activity was identical to that of acetate.

The MBAT on methanol and glucose indicated there was an increase in the activities on these substances in systems PL1 and PL2, but not PL3 (Table 5.12, Figure 5.18). In general, the glucoclastic and methanolic activities by all three sludges were comparable but low.

Table 5.12 MBAT Day 138

Sludge	Specific Activity (g COD/g VSS·d)		
	Formate	Glucose	Methanol
Seed	0.11	0.02	0.02
PL1	0.22	0.05	0.05
PL2	0.18	0.06	0.06
PL3	0.09	0.03	0.03

Overall, the MBAT demonstrated that the sludge from each system had progressively acclimatized to the CTMP-N wastewater.

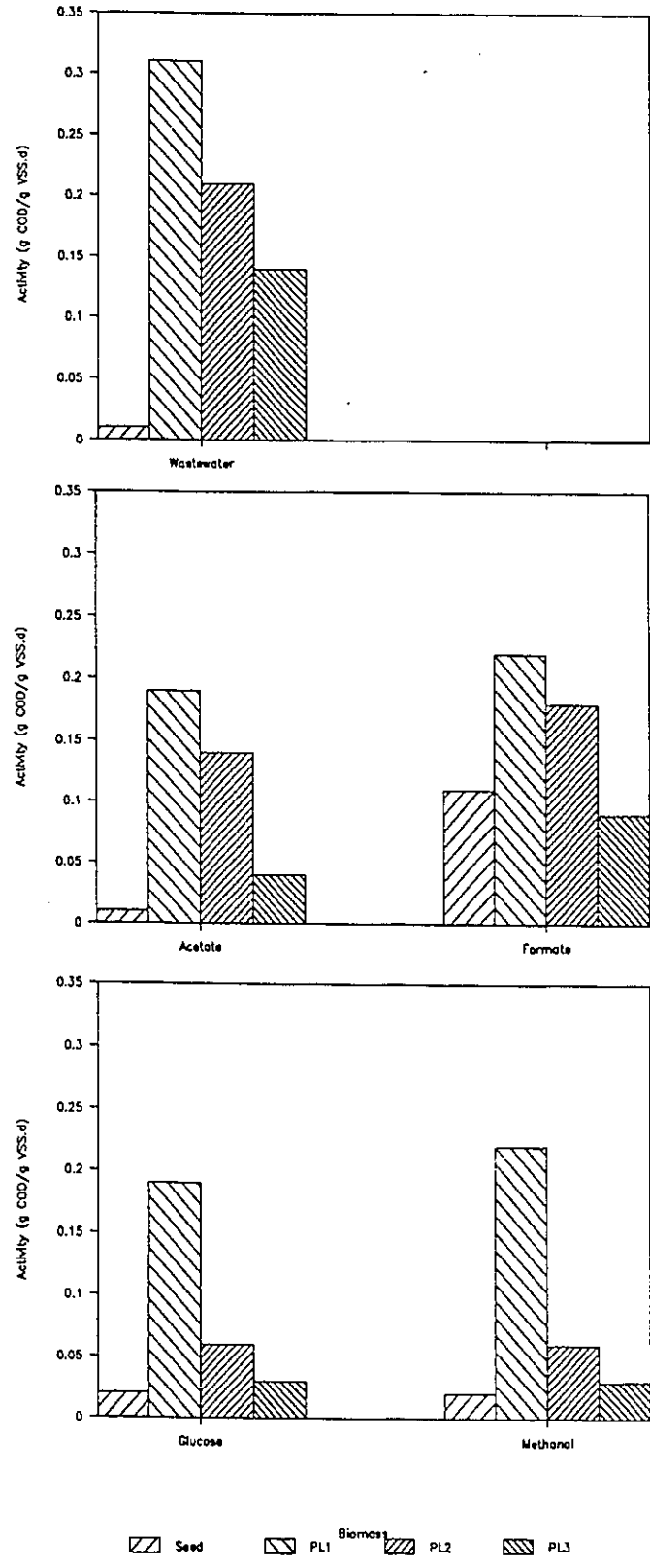


Figure 5.18 Specific activity Day 138

5.2.14.2 Activity - Batch Test and Reactor Performance Comparison

In batch tests specific activity is measured as COD converted to methane per unit time by unit weight of biomass (VSS). The variables that measure similar activity in a continuous operation are specific removal rate (SRR), calculated from COD removed per reactor biomass per unit time, and specific methane production rate (SMPR), calculated from methane produced per reactor biomass per unit time.

The trends from the SRR and SMPR calculations (Appendix D) were similar (Table 5.13). They were, however, significantly higher than activities obtained from batch tests (Figure 5.17, Table 5.10). These results suggest that bottle test results may not always be directly transferrable to continuous systems.

Table 5.13 Comparison of SRR (g COD_g/g VSS·d) and SMPR (CH₄ produced as g COD/g VSS·d)

Wastewater	% Wastewater Concentration	PL1		PL2		PL3	
		SRR	SMPR	SRR	SMPR	SRR	SMPR
CTMP-N	50	0.25	0.24	0.28	0.30	0.24	0.21
	70	0.33	0.37	0.38	0.41	0.38	0.35
	80	0.49	0.48	0.46	0.44	0.36	0.30
	90	0.55	0.57	0.58	0.58	0.57	0.34
	100	0.53	0.55	0.47	0.50	0.45	0.28
TMP	100	0.34	0.34	-	-	-	-
CTMP-H	100	0.64	0.45	-	-	-	-

Low SMPR for system PL1 at 80% to 100% CTMP-N feeding was due to a faulty gas meter. Note that in system PL1 for CTMP-H feeding the activity based on COD_S removal was higher than activity calculated from CH₄ production (*i.e.* COD_S removal could not be fully accounted for by CH₄ formation).

5.2.14.3 Activity - Specific Removal Rate vs Specific Loading Rate

All three systems, especially PL1 and PL2, received specific loadings over a relatively broad range (0.70 - 1.40 g COD_S/g VSS·d). All systems can be considered highly loaded (Henze and Harremoes, 1982). Specific reaction rates plotted against specific loading rates (SLR), g COD_S/g VSS·d, show that reaction rates were a function of loading rates (Figure 5.19).

Initially the SRR of system PL3 was higher than PL1 and PL2. This indicated the sludge could more readily degrade (*i.e.* decreased inhibition) fiber free feed. A period of acclimatization to the fiber was required by PL1 and PL2. When the SLR reached 0.95 g COD_S/g VSS·d the SRR of PL1 and PL2 were equivalent to PL3. The results indicate that at lower loadings systems PL1 and PL2 did not function at their full capacity and/or biomass activity increased during the gradual acclimatization to fiber containing wastewater.

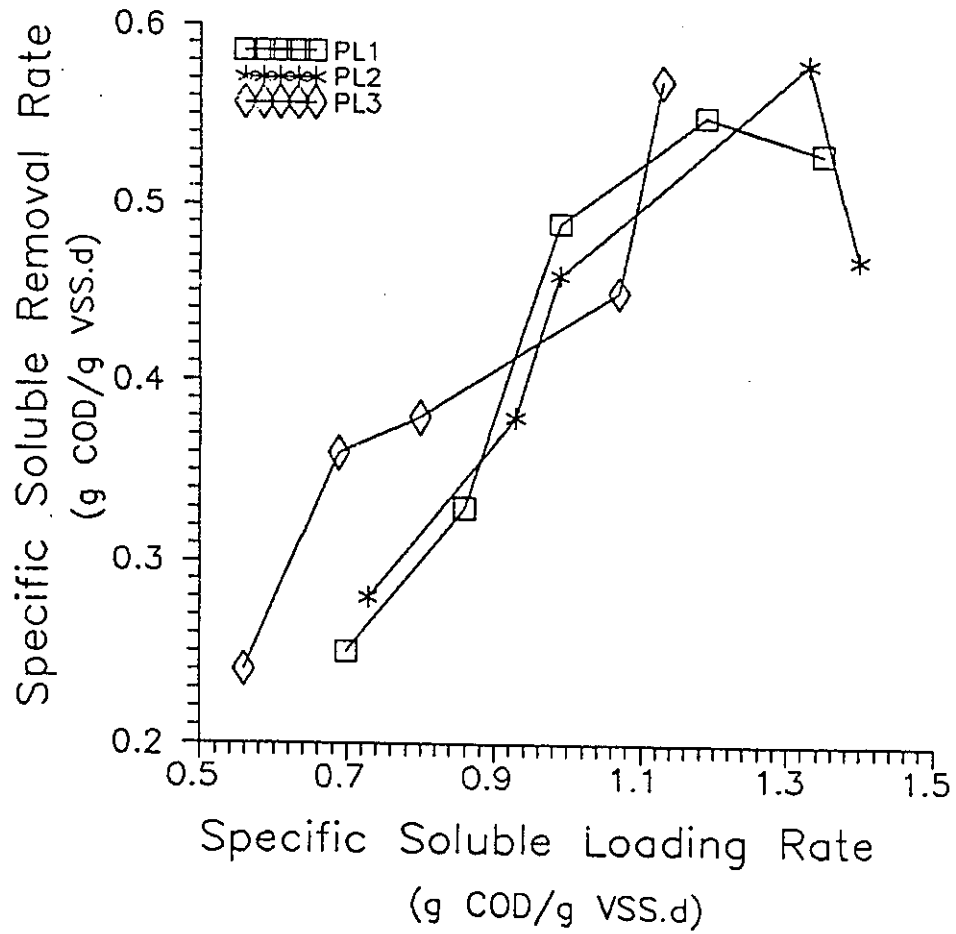


Figure 5.19 Specific substrate removal rate versus specific substrate loading rate

The SRR of systems PL1 and PL2 plateaued at a SLR of approximately 1.2 g COD₅/g VSS·d. This corresponded to a maximum SRR of 0.57 g COD₅/g VSS·d. The SRR decreased beyond this loading. It was possible the increased concentration of wastewater in the feed was toxic at the higher loading.

It was suspected that a reduction in the SRR (*i.e.* inhibition) in systems treating CTMP wastewaters may be the result of: a) a diffusion effect from granules becoming coated with fiber/fines; or b) an inhibition effect; or c) a diffusion/inhibition effect. The diffusion effect is a restriction of the mass transfer between the media and the biomass. If the pore size of the fiber/fines coating is less than the size of the substrate particles that must pass through it, the transfer of the substrate particles will be restricted. The hypothesis of inhibition by the diffusion effect would have been supported if the SRR of system PL1 was less than system PL3 when the system PL1 granules were coated with fiber, however, this was not the case. It is possible toxicity during treatment of CTMP wastewaters may be partially due to this physical phenomenon, however it is mainly due the physio/chemical effect of inhibition.

5.2.15 Resin Acids

Resin acids and long-chain fatty acids are some of the components of CTMP and TMP wastewaters that are known to be toxic to methanogenic bacteria (Welander and Andersson, 1985; McFarlane and Clark, 1988; Field et al., 1988). Unaltered CTMP-N wastewater contained 19 mg/L resin and fatty acids, while filtered CTMP-N contained 12 mg/L (Table 5.14, Appendix F). The distribution of resin acid was similar in both feeds: dehydroabiatic, palustric, abiatic and isoprimeric acids were predominant, in that order. Fatty acid concentration was 13 - 17% of the resin acid concentration. The seed sludge had a resin and fatty acid content of 22 mg/L at inoculation. The average daily resin and fatty acid loading on UASB1 of system PL1 ranged from 44 (at 50% wastewater concentration) to 83 mg/L of the reactor's d (at 100% wastewater concentration). Similarly the loading on UASB3 of system PL3 ranged from 28 to 53 mg/L of the reactor's d.

At the termination of the experiment it was found that both resin and fatty acids had accumulated in the sludge beds. Furthermore, resin and fatty acid concentrations in the effluent of systems PL1 and PL2 exceeded that of the feed. Approximately 50% of the resin and fatty acids in the unaltered CTMP-N was suspended (i.e. fiber). The total and soluble resin and fatty acid concentrations were equal in the effluent of both systems PL1 and PL2. It is possible that resin and fatty acids accumulated in the sludge bed

have undergone solubilization and subsequent leaching into the effluent.

Table 5.14 Resin and long-chain fatty acid concentrations at termination of the experiment (mg/L)

System	Seed	Feed	Sludge Bed	Effluent
Total				
PL1	22	19	1518	60
PL2	22	-	998	56
PL3	22	12	315	11
Soluble				
PL1	-	6	-	56
PL2	-	-	-	50
PL3	-	11	-	15

The concentrations found in the sludge beds have been shown to be inhibitory (McCarthy *et al.*, 1990) to methanogenesis when applied as a shock-dose. It seems that during the course of the study the bacteria were able to gradually acclimatize to these high concentrations and the reactors performed satisfactorily. The somewhat lower COD removal efficiencies in systems PL1 and PL2 may be attributed to the higher concentration of the toxic resin and fatty acids associated with the granular sludge.

CHAPTER 6

CONCLUSIONS

Treatment of chemi-thermomechanical wastewater of varying fiber content was investigated in batch and continuous two-stage anaerobic digestion systems. The following conclusions have been drawn from the study.

6.1 Phase One

1. CTMP wastewaters containing fiber are inhibitive to methanogenesis.
2. High fines content CTMP wastewater (contains 2.4 times more fiber than normal fiber wastewater) is more inhibitive to anaerobic bacteria than normal fiber CTMP wastewater.
3. The inhibition of anaerobic bacteria by CTMP wastewater containing fiber is reduced by dilution.
4. Removal of fiber from CTMP wastewater reduces the inhibition of CTMP wastewater to anaerobiosis.

6.2 Phase Two

1. CTMP wastewater containing fiber and/or fines, and fiber free wastewater can be successfully treated without dilution anaerobically if biomass acclimation is permitted.
2. Modified batch activity tests conducted on CTMP wastewater do not yield values equivalent to the specific removal rate (soluble COD) and the methane production rate in continuous systems under maximum loading conditions.

3. Anaerobic granular biomass exposed to fiber attain the highest specific batch activities on wastewater and synthetic substrates (acetic acid and formic acid).
4. Biomass not exposed to fiber during two-stage treatment degrades fiber-containing wastewater in the MBAT at a lower rate than biomass exposed to fiber during two-stage treatment. In other words, fiber contains compounds which require acclimation by biomass.
5. The presence of fiber affected the sludge bed in two ways. Fiber accumulation in the bed increased the bed volume and led to deterioration of the sludge settleability. As a result, the sludge became more prone to washout.

Long term operation would require periodic removal of fiber, if possible, from the reactor and reinoculation if primary treatment to remove fiber from mill effluent is not improved.

6. Fiber adhesion to granules affects settleability of the sludge. Initially, adhesion increases granule size thereby improving settleability. The settleability of granules coated with fiber eventually deteriorates.
7. The design organic loading rate for the pilot plant is 18.5 g COD_T/L reactor·d (wastewater diluted 50%) with an average COD_S removal efficiency of 55% for the anaerobic reactor. The laboratory scale system is capable of operating at 40.5 g COD_T/L reactor·d (0% dilution) with a COD_S removal efficiency of 38%.
8. The maximum specific loading and removal rates based on COD_S are 1.20 g COD_S/g VSS·d and 0.57 g COD_S/g VSS·d respectively. The SRR plateaus at this loading. Loading above this level results in a decrease of the specific removal rate. It is possible methanogenesis was inhibited by the wastewater at this loading and/or fiber interaction with and physical degradation of the granules diminishes the removal capacity of the sludge.
9. The biomass yield on CTMP-N wastewater was very low. In this study it was 0.025 g VSS/g COD_S removed.
10. Forty percent of the total resin acids found in the CTMP wastewater in this study was associated with the fiber.
11. Resin acids are retained in sludge beds at concentrations higher than known to be toxic to anaerobic sludge. This demonstrates that sludge can be acclimated to high resin acid concentrations. Resin acid

inhibition may be more evident in substrate limited situations.

12. A maximum specific methane (COD equivalent) production rate of 0.55 g COD/g VSS·d was attained in a two-stage system treating 100% CTMP-N wastewater (0% dilution) at an HRT of 5.1 hours in this experiment.
13. Applicability of batch toxicity test results to the continuous reactor operation was limited. The batch tests predicted a high degree of inhibition by unaltered CTMP-N wastewater and little for fiber free wastewater. No such great differences in reactor performances were encountered in continuous systems. The system treating fiber free wastewater was superior at all times, but the performances of the three systems were comparable.

CHAPTER 7

FUTURE STUDIES

The following topics are suggested as possible subjects for further investigation:

1. Examine the effect of effluent wood solids (fines, fiber, and wood chips) on system operation, reactor performance, biomass retention, and toxicity as a function of wood fiber/fines size.
2. Short term reactor shutdown can drastically reduce sludge activity. Investigate the affects of temporary feed shortages, temperature shock, internal reactor recycle failure, and combination to identify which factor(s) contribute to activity reduction.
3. Investigate the benefits of supplementing CTMP wastewater with nutrients, minerals, and vitamins to reactive sludges and increase yield.
4. DTPA (Diethylene-triamine-penta-acetic acid) is a chelating agent used in the CTMP process. Examine acute toxicity and quantify the inhibitory effects of DTPA.
5. Assess toxicity and degradability of resin acids by an active anaerobic culture acclimatized to CTMP wastewater. Quantify, in batch experiments, the toxicity and treatability of individual resin acids commonly found in CTMP wastewater and the effect of shock resin acid loads on a operating reactor.
6. Separate the components of CTMP wastewater into different fractions and identify which component(s) is(are) toxic.
7. Design and develop improvements to the existing primary treatment system to improve the efficiency of fiber/fines removal at the pilot plant in Quesnel River.

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APPENDIX A
PL1 EXPERIMENT RECORDS

Table A1. Daily operating data of system PL1

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N2 (%)	CH4 (%)	CO2 (%)						
START-UP CTMP-N															
16-Jul-88	0														
18-Jul-88	1														
17-Jul-88	2														
18-Jul-88	3	0	0	0	8.22		87	4	3						
19-Jul-88	4	130	0	0	7.72		74	15	6						
20-Jul-88	5	50	0	0	7.44		65	24	6						
21-Jul-88	6	5	5	0	7.05		61	28	7						
22-Jul-88	7	185	0	0	6.92		43	43	8						
23-Jul-88	8	140	0	0			28	58	9	1720					
24-Jul-88	9				6.8		37	47	10						
25-Jul-88	10	165	0	0	6.7		19	68	9	2000	0.613	0.483			
26-Jul-88	11	185	0	0	6.6		15	89	10						
27-Jul-88	12	105	0	0	6.55		13	71	10						
28-Jul-88	13	75	0	0	6.55		10	71	12	1380					
29-Jul-88	14	325	0	0											
30-Jul-88	15	330	0	0											
31-Jul-88	16	350	0	0			8	73	12						
01-Aug-88	17	355	0	0	6.8		9	72	12						
02-Aug-88	18	470	0	0											
50% CTMP-N															
03-Aug-88	19	340	0	0	6.78		8	73	13	3310	1990	0.756	0.573		
04-Aug-88	20														
05-Aug-88	21	180	0	0	6.42	1100	58	27	8	3620	2050				
06-Aug-88	22						25	56	12						
07-Aug-88	23	465	0	0	6.93	1550	14	89	11	3340	1180				
08-Aug-88	24	760	0	0			10	71	12						
09-Aug-88	25	520	0	0			7	78	9	2840	1880	0.502	0.408		
10-Aug-88	26	515	0	0	6.83	1650				2770	1840				
11-Aug-88	27	510	0	0											
12-Aug-88	28	530	0	0											
13-Aug-88	29														
14-Aug-88	30														
15-Aug-88	31	370	0	0	6.8	1280	3	78	13	2920	1680				
16-Aug-88	32	345	0	0											
17-Aug-88	33	285	0	0											
18-Aug-88	34	385	0	0	6.75	1600	6	75	13	2500	1440	0.706	0.67		
19-Aug-88	35	385	0	0											
20-Aug-88	36														
21-Aug-88	37														
22-Aug-88	38	255	0	0	6.84	1150	6	74	11	2220	1520				
23-Aug-88	39	455	25	0											
24-Aug-88	40	315	0	0						4210	1920	1.076	0.920		

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N2 (%)	CH4 (%)	CO2 (%)						
25-Aug-88	41	235	0	0	7.04	1140	3	78	13						
28-Aug-88	42	95	25	0											
27-Aug-88	43														
28-Aug-88	44														
29-Aug-88	45	310	0	0						1410	1080				
30-Aug-88	46	240	15	0	6.95	1230	6	80	8	3350	1710				
31-Aug-88	47	40	0	0											
01-Sep-88	48	190	0	0			5	78	11	3080	1700	0.574	0.46		
02-Sep-88	49	235	0	0											
03-Sep-88	50														
04-Sep-88	51														
05-Sep-88	52														
06-Sep-88	53	170	0	0	7	1180	4	81	9	2660	1490	0.562	0.437		
07-Sep-88	54	100	0	0											
08-Sep-88	55	80	0	0			4	79	11	2270	1910				
09-Sep-88	56	80	0	0											
10-Sep-88	57														
11-Sep-88	58														
12-Sep-88	59	70	0	0	7.06	1200	0	79	15	2150	1440				
13-Sep-88	60	80	0	0											
70% CIMP-N															
14-Sep-88	61	140	0	0						2480	1950	0.756	0.506		
15-Sep-88	62	305	0	0											
16-Sep-88	63	305	0	0	7.09		4	76	15	3740	2130				
17-Sep-88	64														
18-Sep-88	65														
19-Sep-88	66	415	20	0			3	77	14	4740	2170				
20-Sep-88	67	530	35	0	6.92	1580				4680	2100	0.657	0.73		
21-Sep-88	68	150	0	0											
22-Sep-88	69	300	0	0	6.96		3	79	12	3030	1840				
23-Sep-88	70	300	0	0											
24-Sep-88	71														
25-Sep-88	72														
26-Sep-88	73	205	25	0			3	77	13	3640	2230				
27-Sep-88	74	280	0	0	6.92	1400									
28-Sep-88	75	110	0	0											
29-Sep-88	76	160	0	0	7.18		3	78	13	3480	1840	0.831	0.728		
30-Sep-88	77	60	0	0						2840	1670				
01-Oct-88	78														
02-Oct-88	79														
03-Oct-88	80	195	0	0	7.11	1450	3	78	12	2630	1670				
04-Oct-88	81	180	0	0											
05-Oct-88	82	215	0	0						3390	2010	0.836	0.698		

		FEED										PREACIDIFICATION TANK														
Date	Day	Time (hrs)	Time (min)	Elapsed Time (hrs)	Biogas Meter Reading	Effluent Volume (L)	Room T (°C)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)
								Acet (mg/L)	Pro (mg/L)	But (mg/L)								Acet (mg/L)	Pro (mg/L)	But (mg/L)						
13-Nov-88	121	11	45	28.33	8908	7.95	33.4	1130	0	0	6.07	1380	6750	3970	1.47	840	115	0	6.74	1870	5870	3240				
14-Nov-88	122	10	36	22.85	9058	16.25	33.0	1065	130	0	6.07	1380	6750	3970	1.34	880	150	0	6.74	1870	5870	3240				
15-Nov-88	123	14	43	28.12	9233	12.16	33.2	1280	130	0	6.07	1380	6750	3970	2.10	975	155	0	6.74	1870	5870	3240				
16-Nov-88	124	9	31	18.80	9382	17.54	33.8	1375	175	0	6.07	1380	6750	3970	2.60	1015	160	0	6.74	1870	5870	3240				
17-Nov-88	125	12	0	28.48	9540	16.66	33.5	965	105	0	6.07	1380	6750	3970	2.00	960	115	0	6.74	1870	5870	3240				
18-Nov-88	126	11	15	23.25	9698	34.2	34.2	34.2	34.2	0	6.07	1380	6750	3970	3.00	975	165	0	6.07	1380	6750	3970				
19-Nov-88	127	14	38	27.38	9888	11.09	34.4	1390	105	0	6.07	1380	6750	3970	3.30	980	165	0	6.07	1380	6750	3970				
20-Nov-88	128	12	58	22.33	44	12.67	33.0	1410	65	0	6.07	1380	6750	3970	3.30	980	165	0	6.07	1380	6750	3970				
21-Nov-88	129	10	12	21.23	186	12.74	33.8	1620	105	0	6.07	1380	6750	3970	3.75	885	135	0	6.07	1380	6750	3970				
22-Nov-88	130	10	10	23.07	381	13.15	33.8	1320	135	0	6.07	1380	6750	3970	2.40	1120	160	0	6.07	1380	6750	3970				
23-Nov-88	131	10	12	24.03	488	16.6	33.0	1570	185	0	6.07	1380	6750	3970	2.80	1120	160	0	6.07	1380	6750	3970				
24-Nov-88	132	10	40	24.47	625	14.73	32.0	1670	185	0	6.07	1380	6750	3970	3.32	1015	170	0	6.07	1380	6750	3970				
25-Nov-88	133	11	30	24.83	787	9	33.5	1550	135	0	6.07	1380	6750	3970	5.85	985	160	0	6.07	1380	6750	3970				
26-Nov-88	134	10	47	23.28	945	11.11	33.0	1665	95	0	6.07	1380	6750	3970	2.76	1130	175	0	6.07	1380	6750	3970				
27-Nov-88	135	14	20	27.55	1121	12.74	32.8	1630	55	0	6.07	1380	6750	3970	3.10	1210	180	0	6.07	1380	6750	3970				
28-Nov-88	136	9	32	19.20	1243	17.89	33.8	1635	170	0	6.07	1380	6750	3970	2.90	1280	180	0	6.07	1380	6750	3970				
29-Nov-88	137	9	55	24.38	1389	15.08	33.0	1640	80	0	6.07	1380	6750	3970	2.18	1190	180	0	6.07	1380	6750	3970				
30-Nov-88	138	8	45	22.83	1498	13.03	34.5	435	150	0	6.24	400	4100	2740	2.82	365	140	0	6.08	550	4470	2040				
01-Dec-88	139	13	30	28.75	1656	16.95	31.8	925	215	0	6.18	1630	9150	6280	1.50	670	165	0	6.18	1990	11190	4250				
02-Dec-88	140	11	45	22.25	1771	16.02	33.5	665	175	0	6.27	1630	9150	6280	2.10	460	160	0	6.18	1990	11190	4250				
03-Dec-88	141	16	10	28.42	1832	14.85	35.8	620	185	0	6.75	1630	9150	6280	2.40	400	150	0	6.18	1990	11190	4250				
04-Dec-88	142	12	56	20.77	2045	17.07	35.0	605	180	40	6.65	1630	9150	6280	3.00	400	145	0	6.18	1990	11190	4250				
05-Dec-88	143	9	30	20.57	2143	17.42	34.9	445	145	0	6.37	1630	9150	6280	2.00	335	145	0	6.18	1990	11190	4250				
06-Dec-88	144	9	44	24.23	2256	18.3	34.2	480	160	0	6.37	1630	9150	6280	2.96	380	120	0	6.18	1990	11190	4250				
07-Dec-88	145	9	43	24.08	2366	13.03	34.5	435	150	0	6.24	400	4100	2740	2.82	365	140	0	6.18	1990	11190	4250				
08-Dec-88	146	9	35	23.77	2464	14.56	34.8	1125	135	0	6.18	1630	9150	6280	2.00	940	185	0	6.18	1990	11190	4250				
09-Dec-88	147	11	13	25.63	2568	16.01	35.0	1580	160	0	6.27	1630	9150	6280	1.80	1345	170	0	6.18	1990	11190	4250				
10-Dec-88	148	12	38	25.42	2689	16.13	34.8	2040	185	0	6.65	1630	9150	6280	2.41	1475	215	0	6.18	1990	11190	4250				
11-Dec-88	149	14	15	25.82	2774	16.06	34.8	2155	90	0	6.37	1630	9150	6280	2.10	1760	230	0	6.18	1990	11190	4250				
12-Dec-88	150	12	15	22.00	2848	18.09	34.7	1970	120	0	6.37	1630	9150	6280	2.10	1685	115	0	6.18	1990	11190	4250				
13-Dec-88	151	9	18	21.05	2886	19.09	34.7	1870	120	0	6.6	1850	11080	6990	2.00	1730	150	0	6.18	1990	11190	4250				
14-Dec-88	152	11	12	25.90	3171	12.88	34.5	1915	85	0	6.6	1850	11080	6990	3.15	1630	120	0	6.18	1990	11190	4250				
15-Dec-88	153	9	14	22.03	3333	15.51	34.2	1890	65	0	6.8	1850	11080	6990	3.50	1690	130	0	6.18	1990	11190	4250				
16-Dec-88	154	9	6	23.85	3497	15.27	34.0	1800	70	0	6.72	1850	11560	6970	3.50	1690	130	0	6.18	1990	11190	4250				
17-Dec-88	155	9	45	24.67	3648	16.95	34.8	1970	120	0	6.37	1630	9150	6280	2.00	1730	150	0	6.18	1990	11190	4250				
18-Dec-88	156	13	50	28.08	3807	14.85	35.8	2040	185	0	6.65	1630	9150	6280	2.41	1475	215	0	6.18	1990	11190	4250				
19-Dec-88	157	9	30	19.67	3901	16.13	34.8	2155	90	0	6.37	1630	9150	6280	2.10	1685	115	0	6.18	1990	11190	4250				
20-Dec-88	158	9	30	24.00	4016	18.09	34.7	1870	120	0	6.37	1630	9150	6280	2.10	1685	115	0	6.18	1990	11190	4250				
21-Dec-88	159	9	30	24.00	4119	15.27	34.0	1800	70	0	6.72	1850	11560	6970	3.50	1690	130	0	6.18	1990	11190	4250				

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)						
13-Nov-88	121	490	0	0											
14-Nov-88	122	145	15	0			2	76	15	5430	2850				
15-Nov-88	123	170	30	0	7	2130				6160	2360	1.772	1.443		
16-Nov-88	124	300	55	0			2	75	17	5100	2770				
17-Nov-88	125	430	45	0											
18-Nov-88	126	365	60	0											
19-Nov-88	127														
20-Nov-88	128														
21-Nov-88	129	325	80	0	7.34		2	75	17	7350	3330				
22-Nov-88	130	285	50	0											
23-Nov-88	131	330	0	0			2	77	14	6200	3350	1.373	0.897		
24-Nov-88	132	105	15	0	7.14	2100				5880	3080				
25-Nov-88	133	520	35	0											
26-Nov-88	134														
27-Nov-88	135														
28-Nov-88	136	165	45	0			3	77	16	6730	4170				
29-Nov-88	137	120	35	0	7.08	2300									
30-Nov-88	138	485	45	0						5620	3030	1.208	1.045		
01-Dec-88	139	670	50	0			3	76	16	5640	3840				
02-Dec-88	140	795	55	0	6.81										
03-Dec-88	141	590	75	0											
04-Dec-88	142	640	55	0											
05-Dec-88	143	785	55	0			3	78	15	5980	3920				
100% TMP															
06-Dec-88	144	245	85	0						5810	2870	1.276	1.039		
07-Dec-88	145	85	100	0			2	77	15	4000	2050	0.961	0.8		
08-Dec-88	146	60	105	0			3	76	15	3010	1620	0.882	0.791		
09-Dec-88	147	55	105	0						3040	1420	0.831	0.834		
10-Dec-88	148	45	120	0			3	75	16	3279	1380	1.027	0.966		
11-Dec-88	149	55	85	0			4	75	15	2810	1260	0.785	0.699		
12-Dec-88	150	45	110	0	6.5	500	4	75	16	2610	1290	0.749	0.701		
100% CTMP-H															
13-Dec-88	151	380	90	0	6.91		3	73	18	6970	2250				
14-Dec-88	152	830	100	0	7.06	2460	3	73	18	8940	3420	3.409	3.056		
15-Dec-88	153	965	100	0	7.11		2	74	18	10230	4200	3.349	2.978		
16-Dec-88	154	1205	115	0	7.04		3	72	19	10820	4420	3.670	3.241		
17-Dec-88	155	1345	100	0	6.74		2	68	24	10230	4810	3.625	3.184		
18-Dec-88	156	1425	90	0	6.87		2	70	21	10870	5230	3.115	2.739		
19-Dec-88	157	1355	105	0	6.78	2790	3	66	26	10230	5340	3.150	2.787		
20-Dec-88	158	1450	105	0	6.74		3	66	25	9840	5030	2.320	2.011		
21-Dec-88	159	1490	125	0	6.74		3	67	25	10540	4680	2.051	1.884		

Table A2. Daily operating record comments of system PL1

DATE	PUMP SETTING	COMMENTS
15-Jul-88	3:30%	Recycle pump clicking. PA tank height stable.
16-Jul-88		UASB volume to 3.5 L - opened to release pressure - reactor tilted to avoid blocking of the effluent port. PA tank low - topped to 2 L. Sucrose/acetate effluent added to the floor tank.
17-Jul-88		UASB volume to 3.6 L - opened top to release pressure. Recycle pump labouring - line moved->better. PA tank low - topped to 2 L.
18-Jul-88	3:25%	UASB reactor has some gas bubbles in the bed. Recycle pump labouring. UASB volume to 3.5 L - opened top to release pressure and line moved. Effluent collection started (all reactors @ 08:45). Pump decreased @ 09:50 - black particles blocking inlet to the feed tank recycle pump, flow low, squeezed though-> flow high. Feed tank emptied of sucrose/acetate effluent - CTMP-N added.
19-Jul-88		UASB gas bubbles in sludge bed - no channelling. PA recycle line crimped 07:50. ACCIDENT Added 5L CTMP undiluted to tank, tubing jumped out of tank and feed spilled. No more thawed feed found, added 4L tap water + 1.7 L PL1 effluent + 2 L PL2 effluent @ 17:00.
20-Jul-88		Bubbles in the UASB lines - bled. Forming of pink coloured sediment on top of the sludge bed. PA tank error - pH was measured after topping the tank. The PA tank recycle line was crimped - fixed @ 08:00. System connected to main feed line from basement.
21-Jul-88	3:33% 3:66%	Feed tank pump does not keep the feed homogeneous (ie fiber settles out). VFA ok, pump increased @ 14:00. PA tank 0.4L effluent added @ 14:00, 0.6 L added 17:00.
22-Jul-88	3:67%	PA tank empty, recycle pump off @ 07:45, topped feed, pump on @ 08:30 - 11:10 add 0.5 L effluent - moved pump lines to fix - 15:00 add 0.66 L, left hand clamp tightened - 16:30 add 0.5 L. UASB reactor has fiber in the sludge bed. Feed and recycle lines walked. Mixer installed in feed tank to stop fibers settling.
23-Jul-88	3:60%	PA tank recycle pump primed 15:10, restarted recycle 18:20, and added 0.2 L effluent. Gas bled from UASB recycle line @ 15:10. Fiber accumulation in the bed is evident.
24-Jul-88	3:50%	
25-Jul-88		Cold room feed tank does not adequately recirculate the feed -> no fibers in feed. Restart at 12:30.
26-Jul-88	3:55%	7:30 PA empty -> N2 is high in the UASB. 2 L effluent added to PA tank. Feed line choking. Added 325 mL more to PA tank. Changed feed line tubing to 3/8" silicone. Filling up of the PA tank resulted, - changed back to original tubing. Installed Y connector in main feed recycle line to diminish sucking back of feed by the basement pump. PROBLEM: acid in feed is zero.

27-Jul-88		PA tank very low volume. Low VFA problem is caused aeration of the basement feed tank by the stirrer. Acid in freshly thawed barrel is 1,100 mg/L acetate, in freshly thawed bucket it is 905 mg/L acetate.
28-Jul-88		Installed 100 L stainless steel feed tank in fermenter room. Tank has the tendency to vortex. Tank recycle line goes directly to reactors. Shortened reactor feed line to avoid fiber accumulation in the line. Fresh feed added. Sampling ports after main feed recycle line installed, take VFA samples from this location.
29-Jul-88 30-Jul-88	3:65%	PA tank volume O.K. Fiber accumulation in bed.
31-Jul-88		
01-Aug-88	3:78%	Increased feed rate @ 10:30. Walked feed line. Bed has fibers from 1.5 L to 2.7 L.
02-Aug-88	3:82%	@ 10:40 brown foam on top of the UASB. Changed system recycle pump tubing. Conducted Imhoff test on the feed and effluent (1 L sample settled 45 min, gently stirred, settled 15 min more. Feed contains 1.4 mL of sediment/L, effluent: 5 mL sediment/L.
03-Aug-88		ACCIDENT: 5 L feed with no chemicals was added. Rest of feed is from previous barrel -> lower acid conc. ERROR: PA tank COD taken after topping. PA tank cloudy pinkish brown. Fines in the UASB reactor from 1.5 L to 2.6 L grey in color. POWER FAILURE at 14:30, all pumps off.
04-Aug-88		Pumps on at 8:00. 1 cm thick brown foam on the UASB top. VFA acid sample taken at 17:00.
05-Aug-88		PA tank empty and feed line choking. Placed weight to end of feed recycle line to hold it down and prevent vortex. Loosened Harvard pump inlet side clamp -> stopped choking. Walked Harvard lines. 2 cm thick brown foam on top of the sludge bed. Solids deposition on UASB wall - also in the PA tank. PA tank temperature is 38 C.
06-Aug-88		@ 13:00 PA tank 3.2 L. PA tank to UASB line crimped. 4 cm of scum + biorings on UASB top. Black/grey colored fines in the effluent.
07-Aug-88	3:82%	Black wall growth in the PA tank especially around the PA to the UASB port.
08-Aug-88	3:79%	Pump decreased @ 13:30. From 1.5 L mark to 2.0 L mark in the UASB greyish fibers. Top 3 cm contains biorings plus fibrous scum. Biological growth on wall near outlet. Crimp in recycle line. pH and alkalinity low - high CH ₄ content - high room temperature - possible meter affected. Feed COD sample taken from port - solids high - COD T high.
09-Aug-88	3:77%	Pump decreased @ 14:00. UASB reactor acids high. Feed line changed. Effluent to floor, collection restart @ 12:50.
10-Aug-88	3:79%	Pump increased @ 10:45. UASB reactor has greyish fibers from the 1.5 L mark to 2.1 L. Wall deposits. VFA high. PA tank ERROR topped before COD sample taken.
11-Aug-88	3:81%	Pump increased @ 11:35. PA tank growth on the back wall.

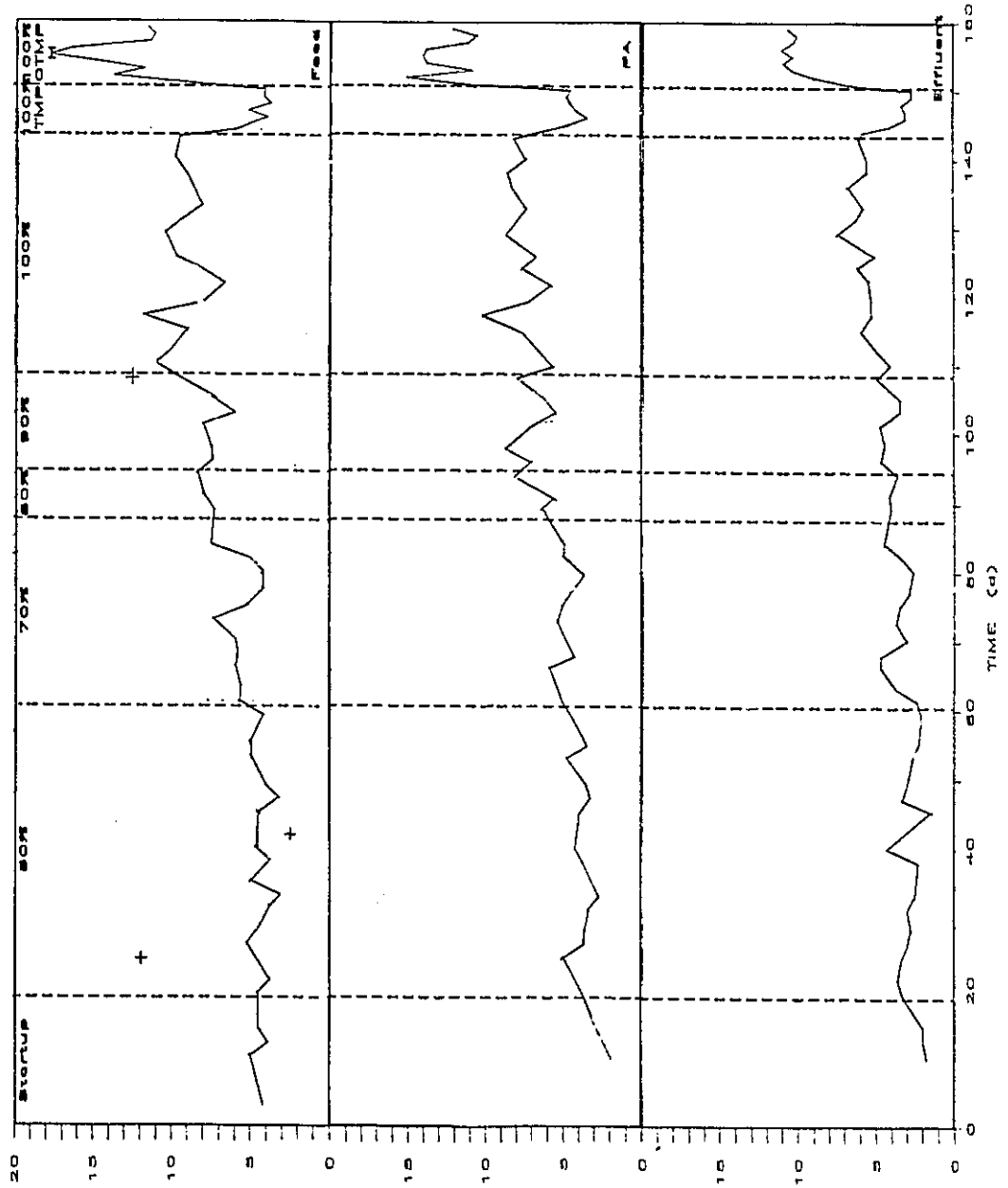
12-Aug-88		UASB reactor greyish fibers from the 1.7 L mark to the 2.1 L. Feed line walked.
13-Aug-88	3:79%	Pump decreased @ 10:40. UASB reactor greyish fibers from the 1.7 L mark to the 2.25 L mark.
14-Aug-88		UASB reactor greyish fibers from the 1.7 L mark to the 2.5 L mark. No recycle to the PA tank. Feed almost empty, cleaned tank.
15-Aug-88		Fibers in the UASB reactor below the 1.7 L line - 2 cm scum in the UASB. Walked the feed line (worn).
16-Aug-88		Liquid level high in the UASB reactor (above the overflow) and has scum on top - higher gas volume. Opened tank to release pressure. The feed recycle line not strong as it used to be. Squeezed tubing.
17-Aug-88		No recycle of feed. Plug (fiber) removed from the recycle line -> O.K. Low solids in the feed is expected since the samples was taken before the problem was fixed. Walk all tubings except the feed line. Observed foam mark at the 4.7 L volume level. Gas accumulated under the scum. The water level in the gas meter is low -> filled.
18-Aug-88		Cleaned the overflow tube.
19-Aug-88		Changed all tubing. Feed line was choking. 50 mL was taken from the fresh effluent for COD test.
20-Aug-88		Granules up to the 1.7 L UASB mark.
21-Aug-88		
22-Aug-88	3:80%	Pump increased @ 10:25. Granules up to the 1.7 L UASB mark. Growth on the PA tank walls.
23-Aug-88		Lines walked.
24-Aug-88	3:78%	Pump decreased @ 09:25. UASB bed volume is 1.75 L. Liquid level was at 4.75 L. Fibers washed out when the liquid level was reestablished by opening the tank to relieve the pressure.
25-Aug-88	3:74%	Pump decreased @ 10:10. Pump off to fix tank recycle. ERROR pump not turned on until 26/08.
26-Aug-88		Pump on 09:30. Harvard pump lines moved. Fibers in the UASB reactor are mixed with granules from the 1.5 L mark to the 1.75 L mark.
27-Aug-88		White colored pellets on top of the feed tank - recycle low. 250 mL of water added to the gas meter.
28-Aug-88	3:96%	Feed tank recycle low.
29-Aug-88		Cleaned the feed tank and flushed the recycle line -> recycle improved.
30-Aug-88		Walked all tubing and changed sample port septa. 250 mL added to the gas meter. Feed recycle poor - fixed @ 11:30. ERROR: pumps were left turned off until 31/08.
31-Aug-88		Pumps discovered off - turned on @ 09:00. Cleaned overflow tube. Effluent is very thick. VFA sample of the PA tank is cloudy.
01-Sep-88		
02-Sep-88		Settling test samples taken - 20 mL from 1.7 L port plus 20 mL from the very bottom port. Walked all tubing.
03-Sep-88		Cleaned feed tank.
04-Sep-88		
05-Sep-88		
06-Sep-88	3:81%	@ 16:30 cleaned the feed tank and lowered the feed rate. Walked all the tubing.

07-Sep-88		
08-Sep-88		
09-Sep-88	3:77%	Walked all the tubing. Acetate in the barrel at 990 mg/L.
10-Sep-88		
11-Sep-88		
12-Sep-88	3:79%	@ 13:25 bed samples taken for TSS/VSS, COD and activity tests (20 mL from the 1.7 L port and the 1.5 L port, 20 mL from the <0.5 L port. For the activity test, combined sludge from the top and bottom ports. Fiber is mixed in the bed of the UASB. Additional fiber on top of bed. INCREASED CTMP CONCENTRATION - 40% DILUTION.
13-Sep-88		Walked all tubing. Water added to the gas meter. The UASB liquid mark is @ 4.5 L - this resulted in biomass and fiber washout.
14-Sep-88		3 mm of solids on top of the sludge bed. INCREASED CTMP CONCENTRATION - 30% DILUTION.
15-Sep-88	2:50%	Pump broken, changed. Walked Harvard lines.
16-Sep-88		Harvard pump lines passed through the pump therefore cable ties to prevent this.
17-Sep-88	2:62	
18-Sep-88	2:65	
19-Sep-88		Cleaned overflow ports. Fibers on top of the sludge bed (0.5 to 1.0 cm). Feed diluted to 40% was topped off with 30% diluted feed @ 17:20. No recycle in the feed tank. Cleaned tubing lines of fiber build up.
20-Sep-88	2:54%	@ 12:15 a bucket from the previous feed barrel is fed. The bottom of the bucket fell out during thaw - some fibers lost. Walked lines.
21-Sep-88		
22-Sep-88		Cleaned the feed tank. Scum on top of the UASB is becoming pinkish.
23-Sep-88		Walked all tubing and changed the sampling port septa.
24-Sep-88		
25-Sep-88		
26-Sep-88		Effluent collection was started in the morning. Walked all tubing.
27-Sep-88	2:57%	Mixing of the bed by gas evolution is vigorous.
28-Sep-88		Fiber in the lines from the PA tank to the UASB - no biomass. Coating of the granules with fibers (less than UASB2). Fibers on top of the sludge bed.
29-Sep-88	2:60%	1 cm of fiber on top of the sludge bed. All lines walked.
30-Sep-88	2:62%	3 mm of fiber on top of the sludge bed. FEED VFA are very low. Crimp in the PA tank recycle line - fixed.
01-Oct-88		ERROR Harvard pump was left turned off.
02-Oct-88		Harvard pump turned on.
03-Oct-88		Walked all lines, changed recycle line, and cleaned overflow tube. Some fine fiber blended with the top of the sludge bed (0.5 cm). 80 mL sludge sample taken for tests.
04-Oct-88		Bed in motion from gas evolution. 0.5 cm of fiber on top of the bed.
05-Oct-88	2:58%	@ 15:25 Pinkish fiber 1 cm on top.
06-Oct-88		1 cm of fiber on top of the sludge bed. Changed all Harvard pump tubing.
07-Oct-88		0.5 cm fiber on top of the sludge bed.
08-Oct-88		
09-Oct-88		0.5 cm fiber on top of the sludge bed. Overflow spill, feed tank empty.
10-Oct-88		2 cm of fiber on top of the sludge bed. The sludge bed is fluid.
11-Oct-88	2:62%	All lines walked. INCREASED CTMP CONCENTRATION - 20% DILUTION
12-Oct-88		
13-Oct-88		Waiked all lines. The recycle line rubber tube stopper was on the wrong side and the line moved through the pump on its own. This was fixed.

14-Oct-88		Overflow port clogged - high liquid level in the UASB (3.5 L) - released by opening the top of the reactor. 0.5 cm fiber on top of sludge bed. Cleaned the feed tank - discarded old feed from the tank. Observed fiber in the bottom of the feed tank.
15-Oct-88		Cleaned the overflow tube. 2 cm of fiber on top. PA recycle line crimped - no recycling - fixed. Granules in the feed line.
16-Oct-88		No feed recycle. Squeezed tubing->O.K. PA empty, filled from the feed. 0.3 cm fiber on top of the sludge bed.
17-Oct-88	2:50%	All lines walked. N2 high.
18-Oct-88		INCREASED CTMP CONCENTRATION - 10% DILUTION
19-Oct-88		
20-Oct-88	2:46%	All lines walked.
21-Oct-88	2:49%	Feed tank recycle not working, unclogged T connector near bottom valve.
22-Oct-88	2:32%	Room temperature low - auxiliary heater turned on. UASB acids high -
23-Oct-88		decreased Q to 10 L/d.
24-Oct-88	2:39%	Q increased 13 L/d.
25-Oct-88	2:49%	Scum on wall of UASB - cannot see liquid height. Q = 16 L/d. VFA O.K.
26-Oct-88		PA tank low - pump tubing came off of the tank outlet when the tubing squeezed - replaced and attached with gear clamp instead of the cable tie.
27-Oct-88	2:50%	
28-Oct-88		Small spill out of the overflow tube - cleaned overflow tube. Harvard
29-Oct-88		pump lines changed.
30-Oct-88		
31-Oct-88		Sludge at the feed tank bottom. All lines walked. 150 mL of water added to the gas meter.
01-Nov-88		Liquid height in the UASB reactor at the 3.5 L mark. Tank opened to release the pressure and lower the liquid height. INCREASED CTMP CONCENTRATION - 0% DILUTION
02-Nov-88		UASB reactor sludge bed is very fluid.
03-Nov-88		Overflow tube cleaned. All lines walked.
04-Nov-88		COD S not clear after centrifugation. Note: this barrel fewer larger fibers than the previous barrels.
05-Nov-88		
06-Nov-88		
07-Nov-88	2:53%	Alkalinity increased. Changed two of the lines - all others walked. PA tank empty. Feed tank recycle not working. Unblocked the Tee connector to the PA tank.
08-Nov-88	2:50%	The UASB sludge bed is fluid. PA tank acetate level is low.
09-Nov-88	2:46%	VFA good.
10-Nov-88	2:47%	All lines walked.
11-Nov-88	2:48%	
12-Nov-88	2:40%	
13-Nov-88		
14-Nov-88		Overflow tube is blocked - squeezed -> O.K. Effluent volume is low due to spill from the overflow tube. Also, liquid level in the UASB high (3.7 L) - opened tank to release pressure and lower the liquid height. Add 125 mL water to the gas meter. All lines walked.
15-Nov-88	2:46%	Changed the recycle pump tubing.
16-Nov-88		Granules in the overflow tube.
17-Nov-88	2:47%	Walked all tubing.
18-Nov-88		75 mL of water added to the gas meter.
19-Nov-88		
20-Nov-88		

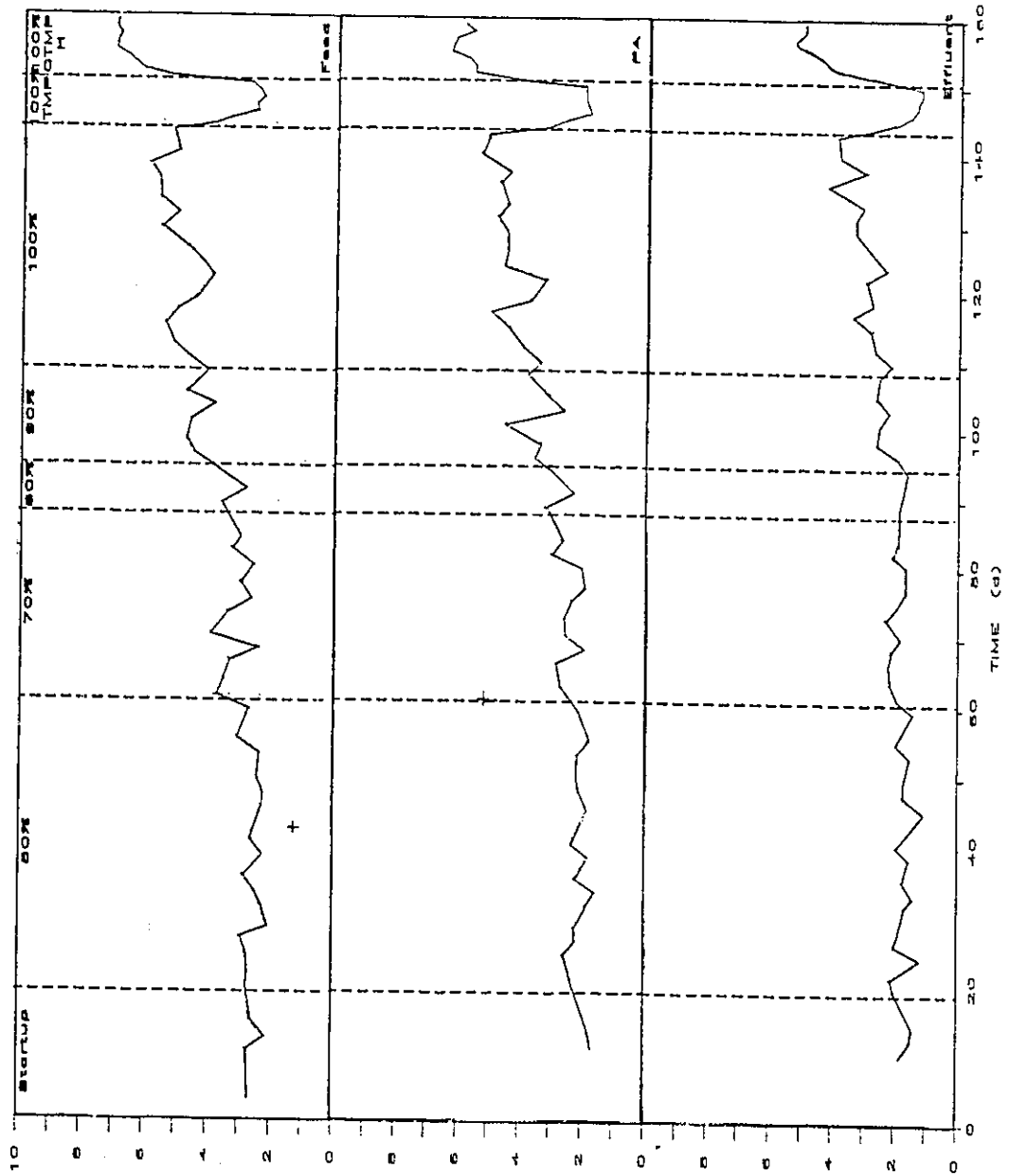
21-Nov-88		
22-Nov-88	2:50%	Moved all lines, changed 1. Pump off at 7, on at 15:15.
23-Nov-88		Acids done at 2 pm.
24-Nov-88	2:55%	Walked all lines. PA tank topped with 1 L.
25-Nov-88		Cleaned the overflow tube. Added 80 mL of water to the gas meter.
26-Nov-88	2:56%	
27-Nov-88		
28-Nov-88	2:27%	<p>Changed 2 lines, moved all others. PA tank line to the UASB connected backwards. By the time this was noticed, 1.15 L water was sucked from the gas meter - all liquids turned black possible due to sulfide precipitation or because of biomass crushed in the pump. The latter is less likely since the top portion of the reactor (not touched by liquid) is black also. The liquid level did not decrease below the recycle port.</p> <p>Biomass and gas in the overflow tube. 4.42 L taken from PL2 effluent. and used to replace PA1. Rinsed PL1 repeatedly by draining down to the recycle port and filling again). Decreased blackness of the water. Some biomass was lost. Finished @ 16:50.</p>
29-Nov-88	2:39%	<p>Effluent charcoal grey in colour - contains some granules and fine black particles.</p> <p>Crushed biomass in the UASB to PA tank recycle line. Recycle pump squeaking.</p>
30-Nov-88	2:45%	Removed 60 mL from < 0.5 L and 1.7 L ports. VFA in the UASB up.
01-Dec-88	2:48%	UASB VFA high.
02-Dec-88		Moved all lines. Large movements of biogas. UASB VFA high.
03-Dec-88		
04-Dec-88		Cleaned the overflow tube. No fiber on top of the bed. Height of the bed at the recycle line height
05-Dec-88		<p>Lines moved, 1 changed. 10 mL sludge removed at the 1.75 L port for photography.</p> <p>After sampling, TMP FEEDING STARTED.</p> <p>0.31 g 28% NaOH and 0.0275 g 85% H3PO4 added per L TMP waste.</p>
06-Dec-88		
07-Dec-88		
08-Dec-88		
09-Dec-88		Moved all tubing. 5 mm fiber on top of the sludge bed. Feed is dark brown in colour in the bucket. It turns pale in the feed tank by the next bed.
10-Dec-88		Sludge bed is fluid.
11-Dec-88		
12-Dec-88		<p>All lines moved.</p> <p>After sampling, CTMP H FEEDING STARTED.</p> <p>0.63 g 28% NaOH and 0.0524 g 85% H3PO4 added per L CTMP H waste.</p>
13-Dec-88		<p>Suspended flocculant mass from: the top of the sludge bed to the overflow port.</p> <p>High solids washout of fine particulate matter. This matter is distributed throughout the bed. Effluent solids are high.</p> <p>14:30 overflow tube just blocked. Opened tank to release pressure.</p> <p>Block in outlet (from inside), difficult to remove.</p> <p>17:10 overflow tube blocked as above. Liquid level 4 L. Released.</p>
14-Dec-88		Fibers visibly accumulate between granules. Top liquid is thick with fibers.
15-Dec-88		Same as yesterday. All lines moved. Large granules in the effluent. Medium granules entered the UASB recycle line.

16-Dec-88		Bed fully expanded just below blorings. A lot of biomass in the recycle being crushed. Fibers observed in 14/12/88 now accumulate in patches in the bed. VFA very high.
17-Dec-88		Overflow spill. Scum mark on the UASB wall at 4.7 L. Gas meter water volume O.K. Biogas CH ₄ content low.
18-Dec-88		Overflow spill. 900 mL of water added to the gas meter. Granules in the effluent. Bed moving in "chunks".
19-Dec-88		Moved all tubing.
20-Dec-88		
21-Dec-88		TERMINATION OF EXPERIMENT 10 mL removed from the < 0.5 L and 1.5 L ports for VSS analysis. Removed biomass into plastic jug while flushing the system with N ₂ /CO ₂ gas. Stored sludge in the airlock at 4 C. The remaining feed was refrozen.



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Figure A1a COD_T versus operating time



(7/8) STAGES COD

Figure 11b COD_g versus operating time

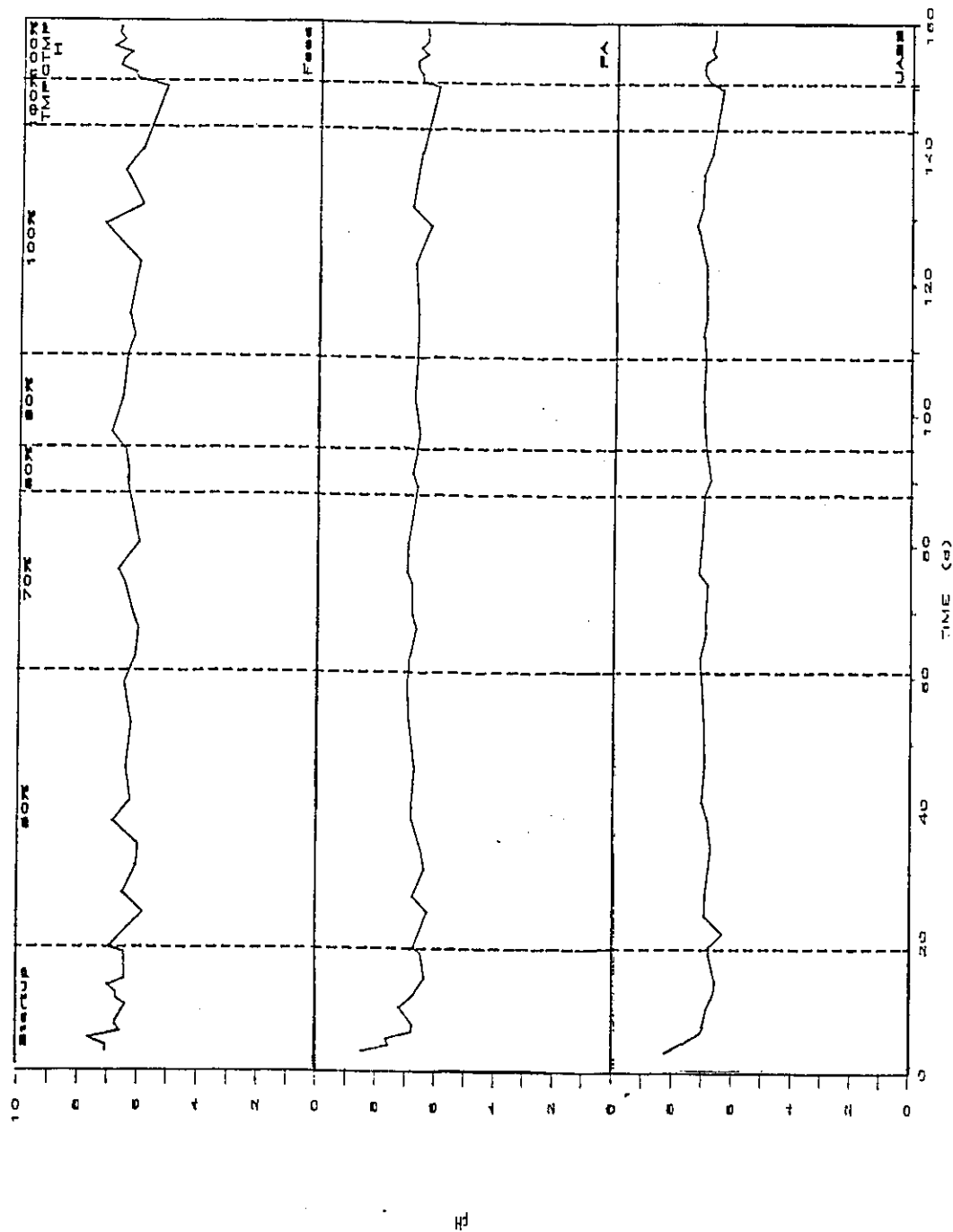


Figure A2 pH versus operating time

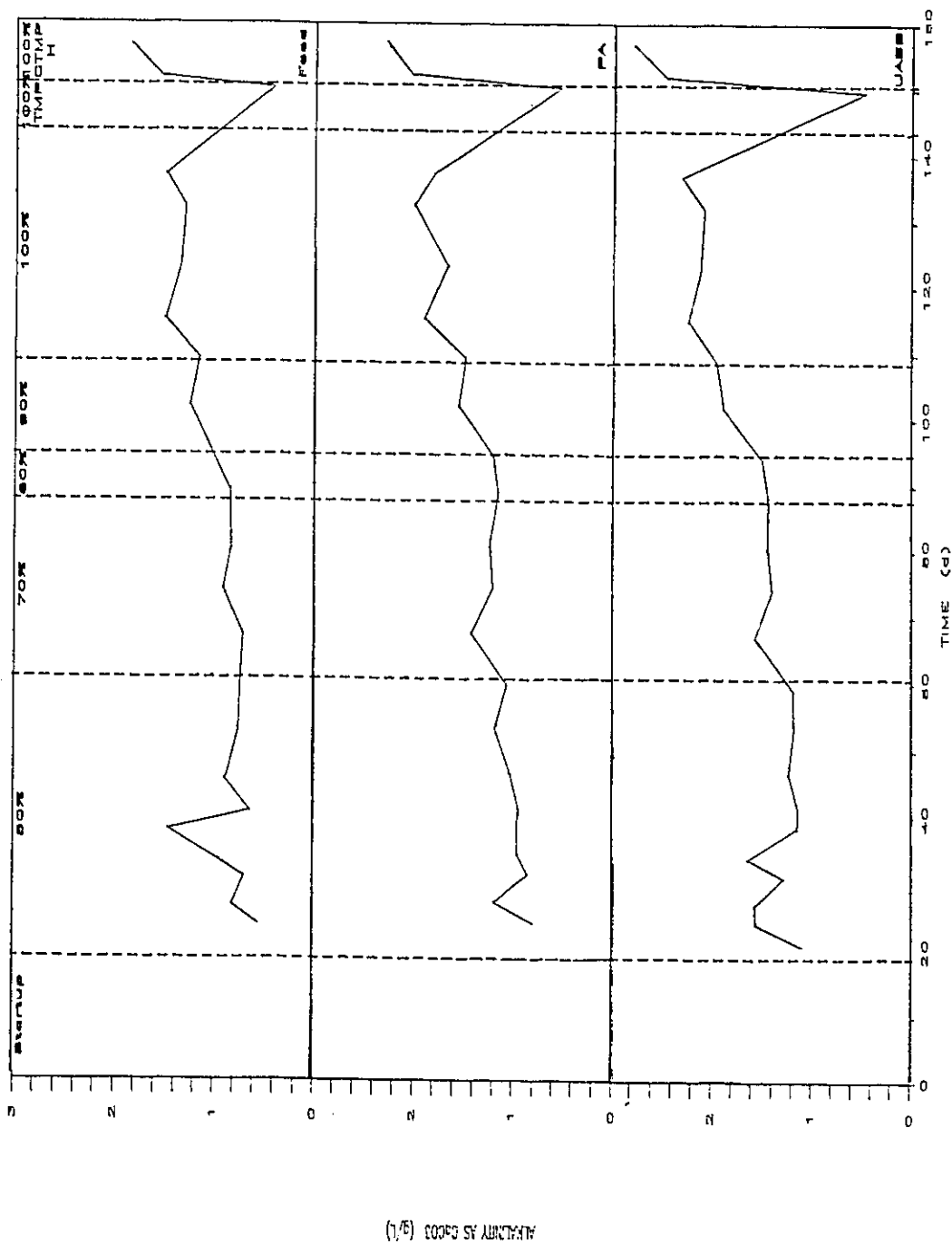


Figure A3 Alkalinity versus operating time

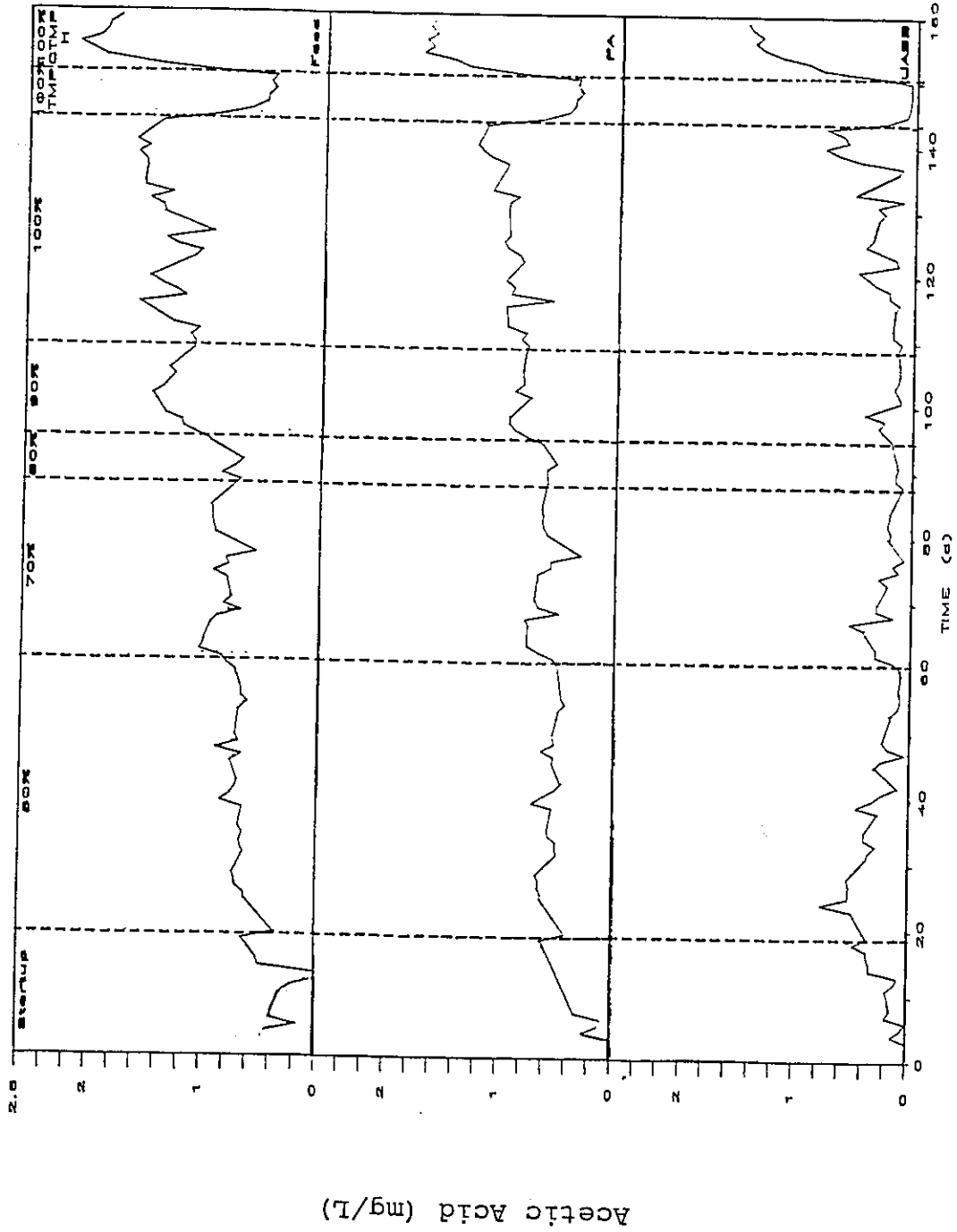


Figure A4a Acetic acid versus operating time

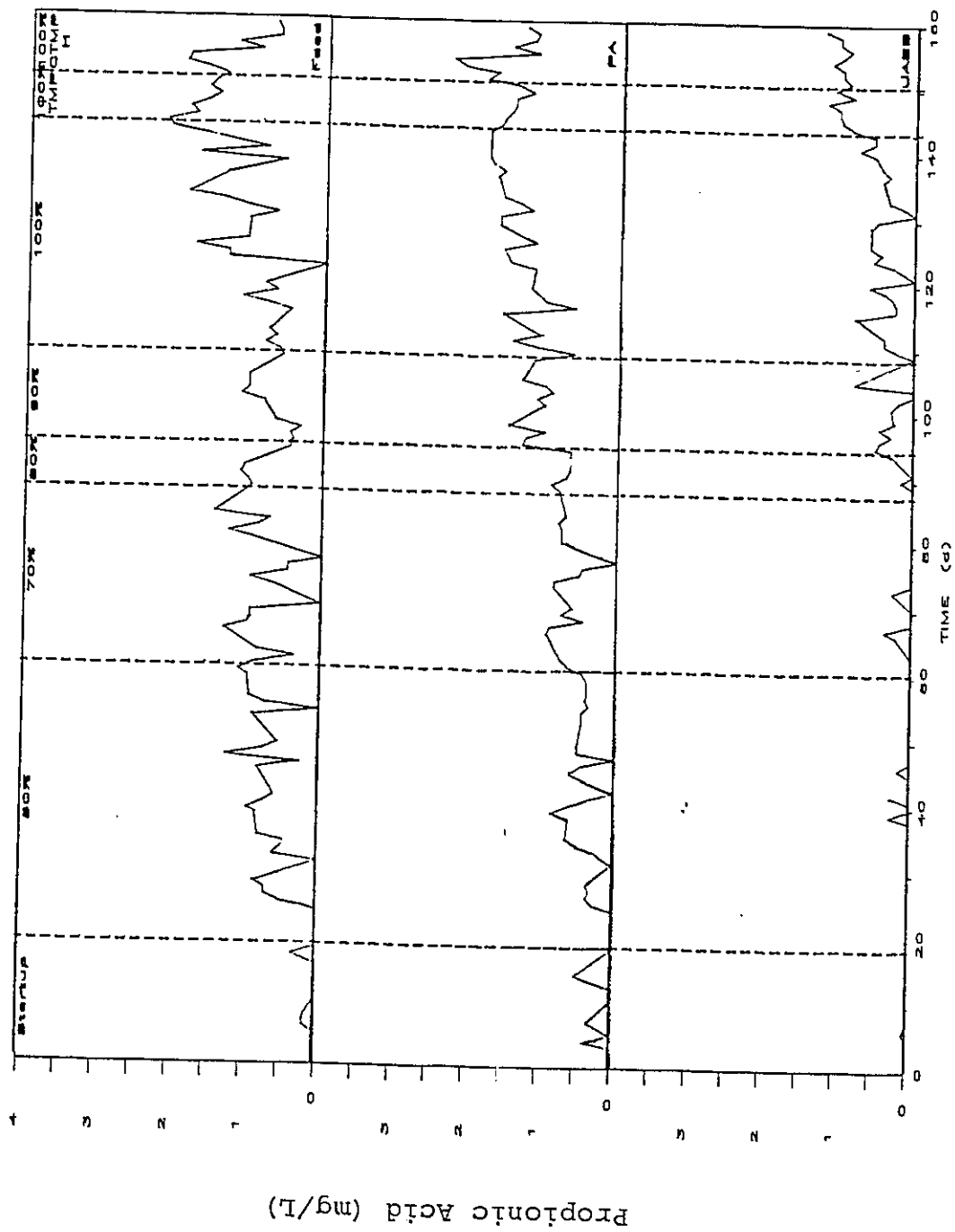
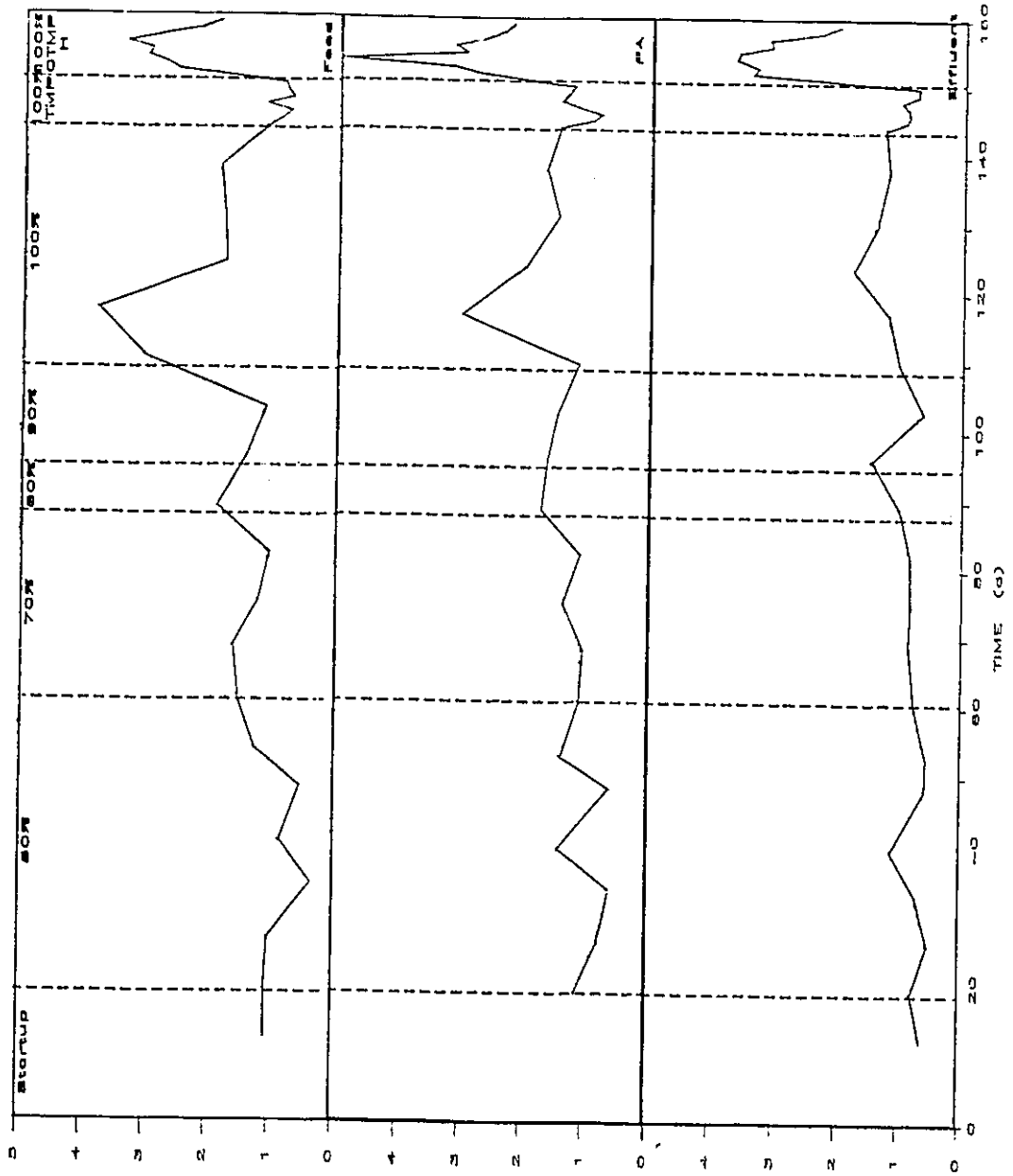
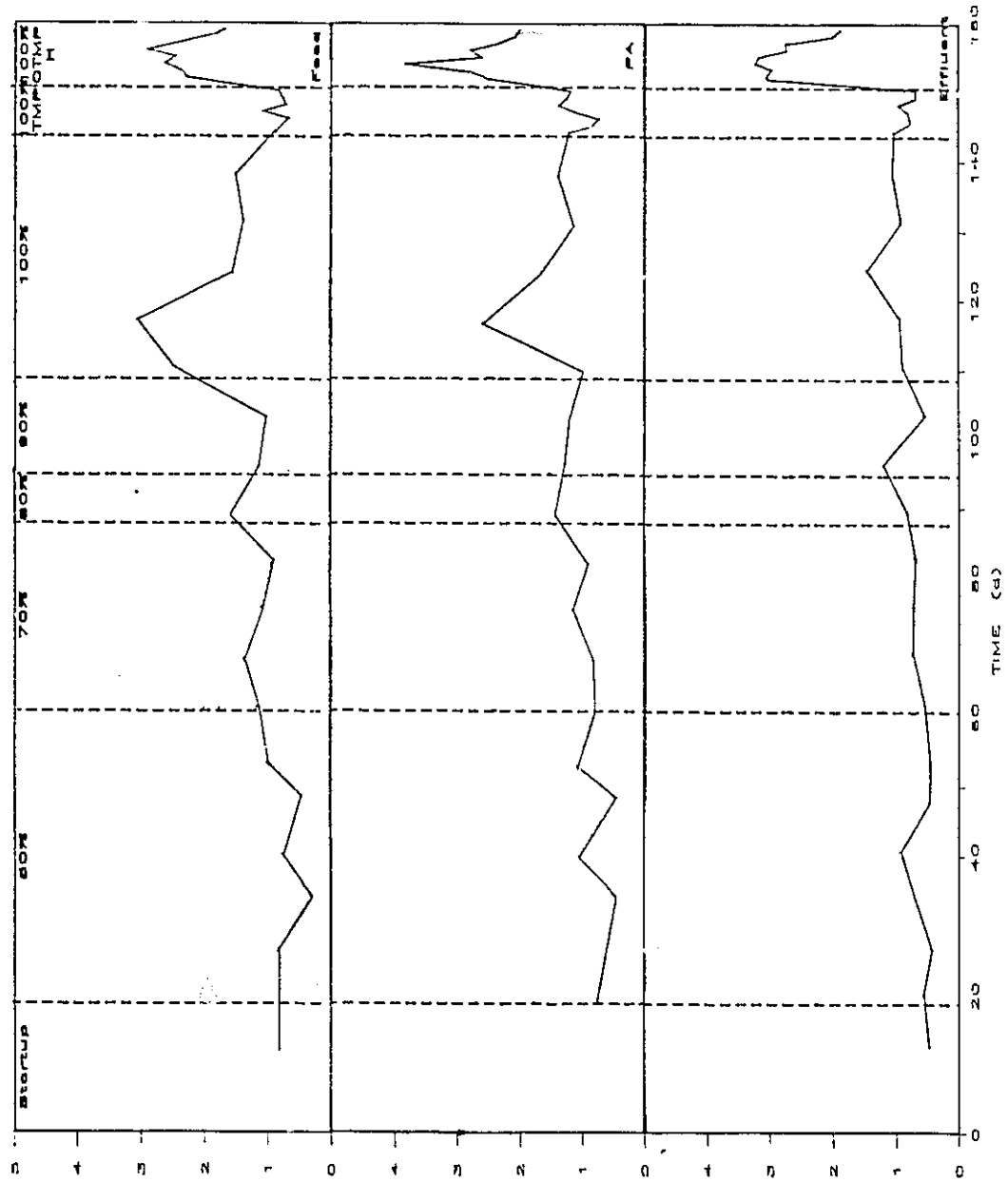


Figure A4b Propionic acid versus operating time



(1/6) SSI

Figure A5a TSS versus operating time



(7/8) SSA

Figure A5b VSS versus operating time

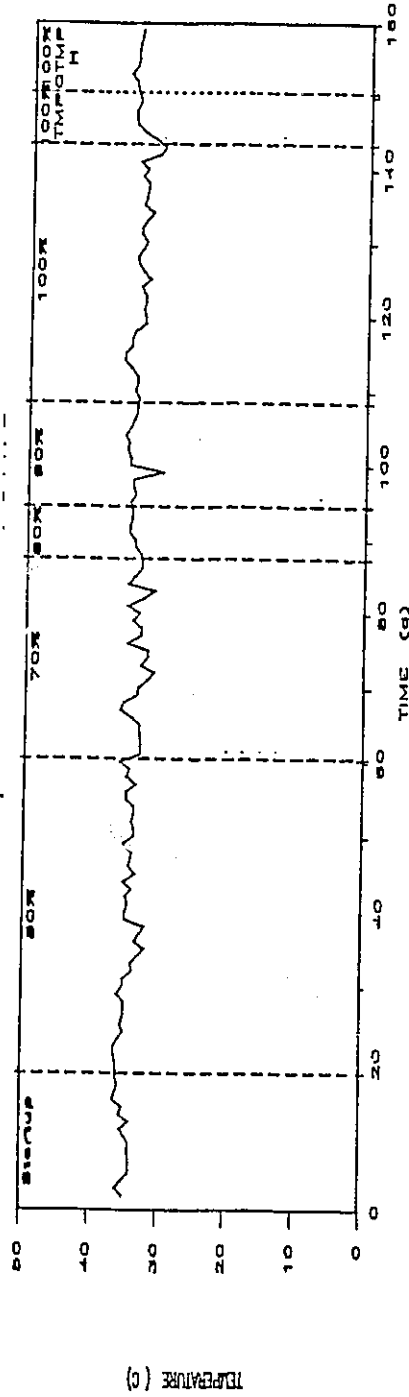


Figure A6 Temperature versus operating time

Table A3. Calculated period averages for system PL1

Wastewater %	FEED										PREACIDIFICATION TANK									
	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	
	Acc (mg/L)	Pro (mg/L)	But (mg/L)								Acc (mg/L)	Pro (mg/L)	But (mg/L)							
CTMP-N 0-50%	353	6	4	6.62	4453	2453	1.077	0.808	1.22	305	15	4	6.69	2535	1770	0.730				
50%	688	62	0	6.37	4275	2538	0.967	0.688	1.80	530	35	0	6.67	3686	2101	0.940				
70%	967	78	0	6.29	5675	3144	1.371	1.091	2.68	634	68	0	6.86	4807	2707	1.128				
80%	796	91	0	6.45	7920	3393	1.878	1.553	2.52	801	74	0	6.76	6683	2807	1.436				
90%	1282	73	0	6.80	7300	4397	1.295	1.050	2.42	852	115	0	6.71	7007	3407	1.251				
100%	1362	106	0	6.38	9318	5033	2.447	1.980	2.48	968	138	0	5.83	7126	4120	1.847				
TMP 100%	568	170	6	5.24	4817	2650	0.882	0.810	2.40	430	146	0	6.06	4824	2248	1.197				
CTMP-H 100%	1822	120	0	6.59	13054	6617	2.661	2.313	2.51	1543	159	0	6.58	12461	6741	3.057				

Wastewater %	UASB REACTOR										EFFLUENT			
	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
	Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)						
CTMP-N 0-50%	224	0	0	6.75		28	57	10	1960	1517	0.613	0.463		
50%	205	2	0	6.86	1296	7	77	12	2650	1667	0.666	0.578		
70%	235	4	0	7.03	1477	3	77	13	3554	1953	0.820	0.663		
80%	131	9	0	6.90	1475	4	75	17	3977	1733	1.001	0.832		
90%	198	31	0	7.04	1800	2	76	16	4290	2378	1.062	0.861		
100%	338	39	0	7.05	1783	2	75	17	5707	3103	1.318	1.040		
TMP 100%	84	101	0	6.50	500	3	76	15	3480	1699	0.643	0.833		
CTMP-H 100%	1159	103	0	6.89	2623	3	70	22	9893	4408	3.069	2.733		

Table A4. Data calculation for system PL1

Date	Day	Flow Rate Measured	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.4 g VSS/L reactor			
			PA (hrs)	UASB (hrs)	Total (g/L-d)	Soluble (g/L-d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	SLR (gCOD/gVSS.d)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	SRV (gCOD/gVSS.d)
START-UP CTMP-N																			
15-Jul-88	0									1.40	1.40	55.79	39.65						
16-Jul-88	1									1.41	1.48	55.72	39.68	17.5	56.6				
17-Jul-88	2									1.41	1.55	55.98	39.87	18.4	61.6				
18-Jul-88	3									1.42	1.83	55.59	39.88	19.4	64.7				
19-Jul-88	4	3.57	11.2	22.5						1.42	1.71	55.53	39.70	20.3	67.7				
20-Jul-88	5	2.57	15.0	31.2						1.43	1.78	55.48	39.71	21.2	70.8				
21-Jul-88	6	3.70	8.4	21.7						1.44	1.88	55.40	39.72	22.1	73.8				
22-Jul-88	7	6.80	4.0	11.8						1.44	1.94	55.33	39.73	23.0	76.9				
23-Jul-88	8	14.08	1.9	5.7						1.45	2.01	55.26	39.74	23.9	80.0				
24-Jul-88	9	15.38	2.2	6.2						1.46	2.09	55.20	39.75	24.9	83.0				
25-Jul-88	10	7.27	5.8	11.0	10.64	6.00	62.4	36.5		1.47	2.18	55.13	39.76	25.8	86.1	0.64	0.35	0.40	62.4
26-Jul-88	11	7.00	3.4	11.5						1.48	2.24	55.07	39.78	26.7	88.1	0.66	0.36	0.33	49.1
27-Jul-88	12	9.39	3.5	8.5	11.05	5.99	48.1	31.9		1.47	2.32	55.00	39.79	27.6	92.2	0.66	0.36	0.12	31.9
28-Jul-88	13	6.86	4.4	11.7						1.48	2.39	54.94	39.80	28.5	95.3	0.66	0.36	0.12	49.1
29-Jul-88	14	10.40	6.0	7.7	14.13	7.84	55.1	45.2		1.48	2.47	54.87	39.81	29.4	98.4	0.65	0.47	0.21	45.2
30-Jul-88	15	15.09	3.6	5.3						1.49	2.55	54.81	39.82	30.4	101.4	0.65	0.47	0.21	55.1
31-Jul-88	16									1.49	2.62	54.74	39.83	31.3	104.5	0.65	0.47	0.21	45.2
01-Aug-88	17	12.97	3.8	6.2						1.50	2.70	54.67	39.85	17.9	107.6	0.65	0.47	0.21	55.1
02-Aug-88	18	14.73	3.3	5.4						1.50	2.70	54.61	39.86	17.9	107.6	0.65	0.47	0.21	55.1
50% CTMP-N																			
03-Aug-88	19	16.75	2.5	5.1	21.23	12.88	26.4	27.1	4.84	1.50	2.60	54.54	39.87	17.9	103.7	1.28	0.77	0.34	26.4
04-Aug-88	20								2.76	1.50		54.48	39.88	17.9					
05-Aug-88	21								2.72	1.50	1.85	54.41	39.89	17.9	77.8				
06-Aug-88	22	17.19	3.6	4.7			23.8		4.10	1.50	1.80	54.35	39.90	17.9	71.8				
07-Aug-88	23	14.15	3.2	5.7					4.42	1.50	1.85	54.28	39.91	17.9	73.8				
08-Aug-88	24	15.41	2.7	5.2					4.72	1.50	2.00	54.21	39.93	17.9	79.9				
09-Aug-88	25	15.70	2.5	5.1					5.14	1.50	2.00	54.15	39.94	17.9	79.9				
10-Aug-88	26	12.66	3.4	6.3	19.33	10.88	42.4	31.0	4.69	1.50	2.10	54.08	39.95	17.9	83.9	1.16	0.85	0.49	31.0
11-Aug-88	27	17.07	2.5	4.7					4.84	1.50	2.10	54.02	39.96	17.9	83.9				
12-Aug-88	28	16.82	2.4	5.1	21.17	9.90	36.0	12.0	5.50	1.70	2.10	53.95	39.97	20.3	83.9	1.27	0.80	0.43	12.0
13-Aug-88	29	18.10	2.3	5.0					5.68	1.60	2.25	53.89	39.98	19.2	88.8				
14-Aug-88	30	17.58	1.9	4.8					6.07	1.70	2.50	53.82	39.99	20.4	100.0				
15-Aug-88	31	13.18	2.4	6.1	15.63	9.12	26.3	27.3	7.91	1.70	2.70	53.75	40.01	20.4	108.0	0.94	0.65	0.25	27.3
16-Aug-88	32	15.01	2.0	5.3					9.91	1.70	2.70	53.69	40.02	20.4	108.0				
17-Aug-88	33	15.35	2.4	5.2	14.52	11.44	20.9	42.2	9.09	1.70	2.40	53.62	40.03	20.4	96.1	0.87	0.69	0.18	20.9
18-Aug-88	34	14.81	3.6	5.4					4.85	1.70	2.70	53.56	40.04	20.4	108.1				
19-Aug-88	35	14.30	4.0	5.6	21.23	12.11		38.9	4.89	1.70	2.80	53.49	40.05	20.4	112.1	1.28	0.73	0.28	38.9
20-Aug-88	36	18.63	2.3	4.3					4.92	1.70	2.60	53.43	40.06	20.4	104.2				
21-Aug-88	37	12.38	3.6	6.5					4.75	1.70	2.65	53.38	40.08	20.4	108.2				
22-Aug-88	38	16.53	3.1	4.8	19.96	10.84	42.0	31.2	5.07	1.70	2.70	53.29	40.09	20.4	108.3	1.14	0.66	0.48	42.0
23-Aug-88	39	15.63	3.5	5.1					6.58	1.70	2.70	53.27	40.10	20.4	108.3				
24-Aug-88	40	16.37	2.9	4.9	22.20	12.84	7.1	26.7	7.33	1.95	1.95	53.16	40.11	23.4	78.2	1.34	0.77	0.09	7.1

Date	Day	Flow Rate		HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Bludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 10.4 g VSS/L reactor					
		Measured (L/d)	O (L/d)	PA (hrs)	UASB (hrs)	Total (g/L.d)	soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (%)	Soluble (%)
25-Aug-88	41	17.16	2.3	4.7						7.01	1.65	1.85	63.10	40.12	22.2	74.2						
26-Aug-88	42									2.56	1.80	1.80	53.03	40.13	21.6	72.2						
27-Aug-88	43									4.97	1.70	1.70	52.97	40.14	20.4	68.2						
28-Aug-88	44									5.99	1.65	1.65	52.90	40.16	19.8	66.3						
29-Aug-88	45	14.61	3.0	6.4		19.82	10.24	68.5	63.2	5.93	1.70	1.70	52.84	40.17	20.4	66.3	1.19	0.62	0.33	0.65	53.2	
30-Aug-88	46	15.77	3.3	5.1						7.46	1.75	1.75	52.77	40.18	21.1	70.3						
31-Aug-88	47									2.60	1.72	1.72	52.70	40.19	20.7	69.1						
01-Sep-88	48	16.63	3.0	4.3						6.21	1.75	1.75	52.64	40.20	21.1	70.4						
02-Sep-88	49	15.88	3.5	5.0		19.40	11.65	24.5	30.6	7.07	1.77	1.77	52.67	40.21	21.3	71.2	1.17	0.70	0.28	0.21	24.5	30.6
03-Sep-88	50									6.43	1.76	1.76	52.61	40.22	21.2	70.8						
04-Sep-88	51									7.28	1.76	1.76	52.44	40.24	21.1	70.4						
05-Sep-88	52									8.01	1.70	1.70	52.38	40.25	20.5	68.4						
06-Sep-88	53	16.22	2.8	4.9		23.85	11.22	45.8	35.5	8.50	1.76	1.76	52.31	40.26	21.1	70.5	1.43	0.68	0.34	0.24	45.8	35.5
07-Sep-88	54	17.47	3.2	4.6						8.85	1.80	1.80	52.24	40.27	21.7	72.5						
08-Sep-88	55	16.63	2.8	4.3		27.66	17.18	54.0	38.0	8.44	1.80	1.80	52.18	40.28	21.7	72.5	1.66	1.03	0.30	0.30	54.0	38.0
09-Sep-88	56	13.97	3.4	6.7						9.46	1.75	1.75	52.11	40.29	21.1	70.5						
10-Sep-88	57									9.29	1.78	1.78	52.05	40.31	21.6	71.7						
11-Sep-88	58									9.81	1.75	1.75	51.98	40.32	21.1	70.8						
12-Sep-88	59	13.60	3.3	5.8		17.19	10.99	48.3	45.9	10.23	1.70	1.70	51.92	40.33	20.6	68.6	1.03	0.66	0.30	0.30	48.3	45.9
13-Sep-88	60	14.78	2.8	6.4						11.96	1.75	1.75	51.85	40.34	21.1	71.5						
70% CTMP-N																						
14-Sep-88	61	14.08	3.9	6.7		23.82	15.51	58.1	47.0	10.52	1.77	1.80	51.96	40.47	21.4	72.6	1.43	0.93	0.50	0.44	58.1	47.0
15-Sep-88	62	14.60	3.7	6.6						10.11	1.77	1.80	52.07	40.60	21.6	72.8						
16-Sep-88	63	13.44	3.3	6.0		22.70	14.13	33.7	38.3	9.84	1.77	1.80	52.18	40.72	21.6	73.1	1.37	0.86	0.46	0.33	33.7	38.3
17-Sep-88	64	12.21	4.7	6.8						10.04	1.82	1.85	52.29	40.85	22.3	76.4						
18-Sep-88	65	16.07	3.6	5.0						10.02	1.82	1.85	52.40	40.98	22.3	76.8						
19-Sep-88	66	16.55	3.8	5.2		27.79	16.31	20.6	34.0	10.20	1.85	1.90	52.51	41.11	22.8	77.1	1.67	0.92	0.34	0.31	20.6	34.0
20-Sep-88	67	16.85	3.0	4.3						11.19	1.80	1.90	52.62	41.24	22.2	76.2						
21-Sep-88	68	16.19	3.2	5.0		28.35	11.49	21.7	11.4	9.06	1.90	2.00	52.73	41.36	23.6	79.7	1.71	0.99	0.37	0.08	21.7	11.4
22-Sep-88	69	16.10	3.3	5.0						8.68	1.80	1.90	52.84	41.49	22.4	76.7						
23-Sep-88	70	15.36	3.4	5.2		27.45	17.85	49.2	52.6	9.08	1.80	1.90	52.96	41.62	22.4	76.9	1.65	1.07	0.81	0.56	49.2	52.6
24-Sep-88	71									9.62	1.80	1.90	53.07	41.75	22.6	78.2						
25-Sep-88	72									9.07	1.75	1.85	53.18	41.88	21.9	74.3						
26-Sep-88	73	15.29	4.1	5.2		33.24	15.58	49.9	34.4	10.84	1.80	1.90	53.29	42.03	22.6	76.8	2.00	0.94	1.00	0.32	49.9	34.4
27-Sep-88	74	15.22	4.1	5.3						10.36	1.90	2.00	53.40	42.13	24.0	81.1						
28-Sep-88	75	14.37	5.8	5.6		22.11	11.53	32.7	31.3	11.13	1.92	1.95	53.51	42.26	24.3	82.2	1.33	0.60	0.43	0.22	32.7	31.3
29-Sep-88	76	16.04	3.7	5.0						11.50	1.90	2.00	53.62	42.39	24.1	81.8						
30-Sep-88	77	16.39	3.4	4.9		20.85	14.62	33.2	44.0	10.43	1.87	1.90	53.73	42.52	23.8	80.6	1.25	0.88	0.42	0.36	33.2	44.0
01-Oct-88	78									10.92	1.77	1.80	53.84	42.64	22.6	78.5						
02-Oct-88	79									2.27	1.77	1.80	53.96	42.77	22.7	78.7						
03-Oct-88	80	14.60	6.4	5.5		16.23	11.50	36.9	38.5	9.43	1.72	1.95	54.06	42.90	22.1	74.8	1.10	0.69	0.41	0.25	36.9	38.5
04-Oct-88	81	15.73	3.7	5.1						10.04	1.80	1.85	54.10	42.99	23.2	78.4						
05-Oct-88	82	17.24	3.2	4.6		26.76	16.42	32.1	36.8	10.77	1.85	1.95	54.13	43.05	23.9	80.8	1.65	0.90	0.50	0.36	32.1	36.8

Date	Day	Flow Rate Measuretec (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.4 g VSS/L reactor					
			PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	SLR (gCOD/gVSS.d)	Total (gCOD/gVSS.d)	SRR (gCOD/gVSS.d)	Total (%)	Soluble (%)	SRR/SLR
06-Oct-88	83	15.94	3.6	5.0					10.34	1.85	1.95	64.17	43.17	23.9	80.9	2.12	0.84	0.84	0.31	36.5	37.2
07-Oct-88	84	16.64	2.7	5.1	36.30	13.96	39.5	37.2	10.51	1.86	1.90	64.21	43.26	24.0	81.1						
08-Oct-88	85								12.20	1.85	1.91	64.26	43.35	24.0	81.3						
09-Oct-88	86								11.90	1.85	1.90	64.28	43.44	24.1	81.4						
10-Oct-88	87								12.58	1.90	2.10	64.32	43.63	24.8	83.6						
80% CTMP-N																					
11-Oct-88	88	14.10	6.1	5.7					12.17	1.96	2.05	64.36	43.82	25.5	86.2	2.20	1.05	0.96	0.50	44.7	47.7
12-Oct-88	89	16.54	3.4	4.8	36.60	17.43	44.7	47.7	13.96	1.96	2.05	64.40	43.71	25.5	86.4						
13-Oct-88	90	16.34	3.7	4.9					15.01	1.96	2.00	64.43	43.80	25.6	86.6						
14-Oct-88	91	15.81	3.6	5.1	37.82	13.44	48.3	38.4	13.38	1.85	2.00	64.47	43.89	24.3	82.3	2.28	0.91	1.10	0.31	48.3	38.4
15-Oct-88	92								13.83	1.76	1.95	64.51	43.96	23.0	78.0						
16-Oct-88	93								13.88	1.87	1.90	64.55	44.07	24.7	83.5						
17-Oct-88	94	16.25	2.8	4.9	40.77	18.59	55.7	57.9	14.96	2.05	2.05	64.58	44.18	27.1	91.7	2.45	1.12	1.37	0.85	55.7	57.9
80% CTMP-N																					
18-Oct-88	95	15.08	4.3	5.3					16.81	2.00	2.20	64.62	44.25	26.5	89.7						
19-Oct-88	96	16.25	3.2	4.9	36.35	22.04	37.1	59.3	17.05	2.10	2.10	64.66	44.34	27.9	94.4	2.19	1.30	0.81	0.77	57.1	68.3
20-Oct-88	97	17.81	2.7	4.5					17.13	2.00	2.00	64.70	44.43	26.6	90.1						
21-Oct-88	98	14.48	3.9	5.5	32.64	20.37	40.4	45.5	17.40	2.05	2.05	64.73	44.52	27.3	92.5	1.90	1.23	0.79	0.56	40.4	45.5
22-Oct-88	99								16.87	2.15	2.18	64.77	44.61	28.7	97.2						
23-Oct-88	100								14.26	2.10	2.10	64.81	44.70	28.1	95.1						
24-Oct-88	101	10.72	4.9	7.5	26.74	14.60	41.3	48.2	14.50	2.10	2.10	64.85	44.79	28.2	95.3	1.55	0.88	0.64	0.41	41.3	48.2
25-Oct-88	102	10.67	5.0	7.5					15.55	2.25	2.25	64.88	44.88	30.2	102.3						
26-Oct-88	103	15.62	2.1	6.1	28.02	17.91	43.6	41.8	16.27	2.29	2.29	64.92	44.97	30.8	104.4	1.72	1.08	0.76	0.45	43.6	41.8
27-Oct-88	104	15.49	2.4	5.2					16.38	2.38	2.38	64.96	45.05	32.1	108.7						
28-Oct-88	105	14.65	3.1	6.4	32.75	20.92	53.7	44.0	17.30	2.38	2.38	65.00	45.15	32.2	108.9	1.97	1.26	1.06	0.55	53.7	44.0
29-Oct-88	106								17.95	2.25	2.25	65.03	45.24	30.5	103.2						
30-Oct-88	107								17.35	2.40	2.40	65.07	45.33	32.6	110.2						
31-Oct-88	108	16.97	2.9	4.2					17.36	2.27	2.37	65.11	45.42	30.9	104.5	1.39	1.39		0.53		
100% CTMP-N																					
01-Nov-88	109	13.16	2.7	6.1					17.82	2.30	2.40	65.14	45.50	31.3	108.1	3.26	1.40	2.01	0.70	61.7	64.3
02-Nov-88	110	16.57	2.7	4.5	54.24	23.32	61.7	54.3	17.34	2.30	2.36	65.18	45.59	31.4	106.3						
03-Nov-88	111	15.37	4.3	6.2					16.22	2.35	2.35	65.22	45.68	32.1	108.8	2.98	1.54	1.50	0.75	50.2	48.8
04-Nov-88	112	16.59	2.7	4.8	49.58	25.54	60.2	48.6	20.37	2.27	2.30	65.26	45.77	31.1	105.3						
05-Nov-88	113								21.82	2.40	2.40	65.29	45.86	33.0	111.6						
06-Nov-88	114								21.83	2.32	2.32	65.33	45.95	31.9	108.1						
07-Nov-88	115	14.31	4.3	5.6	38.25	23.09	35.1	47.7	21.83	2.35	2.35	65.37	46.04	32.4	109.7	2.30	1.39	0.81	0.86	34.1	47.7
08-Nov-88	116	15.44	1.8	5.2					19.91	2.60	2.60	65.41	46.13	34.5	116.9						
09-Nov-88	117	17.34	2.5	4.6	60.85	25.91	55.0	33.1	19.01	2.50	2.55	65.44	46.22	34.5	117.1	3.06	1.55	2.01	0.82	55.0	33.1
10-Nov-88	118	16.25	3.1	4.9					17.87	2.50	2.55	65.48	46.31	34.7	117.3						
11-Nov-88	119	16.94	3.0	4.7	40.89	22.37	34.5	36.5	18.33	2.60	2.85	65.52	46.40	36.1	122.3	2.48	1.35	0.85	0.49	34.5	36.5
12-Nov-88	120								17.28	2.60	2.80	65.55	46.49	36.2	122.5						

Date	Day	Flow Rate Measured Q (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSSG		CONSTANT BIOMASS = 16.4 g VSS/L reactor				
			PA (hrs)	UASB (hrs)	Total (g/L-d)	soluble (g/L-d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	SLR (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (%)
13-Nov-88	121								16.70	2.82	2.82	55.59	48.58	38.5	123.7	0.96	0.43	23.6		
14-Nov-88	122	16.29	3.0	4.9			19.6	25.7	15.39	2.80	2.80	55.63	46.87	36.3	123.0					
15-Nov-88	123	16.29	3.0	4.9					14.96	2.75	2.75	55.67	46.76	38.5	130.3					
16-Nov-88	124	12.45	4.3	6.4	30.11	16.92	23.6	44.6	16.30	2.75	2.75	56.71	46.85	38.6	130.6	1.81	0.96	23.6	44.6	
17-Nov-88	125	17.54	2.7	4.6					16.97	2.70	2.70	55.74	46.94	37.9	128.5					
18-Nov-88	126	16.25	2.4	4.9	48.84	23.06	47.2	41.6	16.23	2.70	2.70	55.78	47.03	37.0	128.7	2.62	1.39	47.2	41.6	
19-Nov-88	127								17.27	2.70	2.70	55.82	47.12	38.1	128.9					
20-Nov-88	128								15.53	2.65	2.65	55.86	47.21	37.5	126.8					
21-Nov-88	129	12.54	5.3	6.4	38.83	20.86	29.1	39.6	17.01	2.70	2.70	55.89	47.30	38.2	129.4	2.34	1.24	29.1	39.6	
22-Nov-88	130	13.22	4.8	6.1					16.36	2.20	2.20	55.93	47.39	31.2	105.7					
23-Nov-88	131	12.44	3.3	6.4	35.01	18.86	34.0	33.1	12.36	2.55	2.55	55.97	47.48	36.3	122.7	2.11	1.12	34.0	33.1	
24-Nov-88	132	14.01	4.9	5.7					13.50	2.70	2.70	56.01	47.57	36.5	130.2					
25-Nov-88	133	15.79	3.3	5.1	38.54	26.57	27.9	45.2	15.50	2.70	2.70	56.04	47.66	38.5	130.4	2.32	1.60	27.9	45.2	
26-Nov-88	134	15.04	3.7	5.3					16.12	2.70	2.70	56.08	47.75	38.5	130.7					
27-Nov-88	135								15.15	2.70	2.70	56.12	47.84	38.7	130.9					
28-Nov-88	136	11.41	8.3	7.0					15.10	2.70	2.70	56.16	47.93	38.7	131.2	1.15	0.30		25.8	
29-Nov-88	137	11.54	5.1	6.9					12.26	2.70	2.70	56.19	48.02	38.8	131.4					
30-Nov-88	138	13.14	4.3	6.1	35.95	23.25	38.5	48.7	13.42	2.70	2.70	56.23	48.11	38.9	131.6	2.16	1.40	38.5	48.7	
01-Dec-88	139	18.35	3.3	4.4					13.06	2.70	2.70	55.77	47.74	38.8	130.6					
02-Dec-88	140	15.57	3.8	5.1	45.72	23.49	42.5	23.8	12.28	2.70	2.70	56.32	47.37	38.3	129.6	2.75	1.41	42.5	23.8	
03-Dec-88	141								13.45	2.72	2.75	54.86	46.99	38.3	129.5					
04-Dec-88	142								12.93	2.64	2.64	54.41	46.82	39.8	134.0					
05-Dec-88	143	14.26	3.9	5.6	40.56	22.16	36.9	24.5	11.32	2.80	2.80	53.95	46.25	38.8	131.2	2.44	1.33	36.9	24.5	
		100% TMP																		
06-Dec-88	144	16.70	2.5	4.8	30.05	19.70		27.2	11.08	2.75	2.75	53.49	45.88	37.8	127.9	1.81	1.19		27.2	
07-Dec-88	145	16.65	3.0	4.8	25.87	16.80	22.9	38.4	10.85	2.75	2.75	53.04	45.50	37.5	126.0	1.56	1.00	38.4	36.4	
08-Dec-88	146	16.22	3.3	4.9	19.14	12.24	23.6	36.7	9.80	2.75	2.75	52.55	45.13	37.2	125.6	1.15	0.74	23.6	36.7	
09-Dec-88	147	17.54	3.4	4.6	26.94	13.90	40.7	45.2	9.64	2.83	2.83	52.13	44.78	37.9	128.4	1.82	0.82	40.7	45.2	
10-Dec-88	148	17.42	2.8	4.6	19.56	12.26	12.6	41.3	9.44	2.83	2.83	51.67	44.39	37.5	127.3	1.16	0.74	12.6	41.3	
11-Dec-88	149	16.75	3.2	4.3	23.58	13.92	37.9	49.2	9.74	2.75	2.75	51.22	44.01	36.2	122.7	1.42	0.84	37.9	49.2	
12-Dec-88	150	13.45	4.3	5.9	16.54	11.00	36.3	52.9	7.99	2.72	2.75	50.75	43.64	35.5	120.3	1.00	0.67	36.3	52.9	
		100% CTMP-H																		
13-Dec-88	151	14.56	3.3	5.5	39.59	23.02	24.9	57.4	15.58	2.80	2.80	50.30	43.27	33.7	114.0	2.40	1.38		57.4	
14-Dec-88	152	17.92	2.5	4.5	73.55	33.10	34.8	44.6	16.97	2.70	2.70	49.85	42.90	34.7	117.4	4.43	1.90	34.8	44.6	
15-Dec-88	153	16.07	3.5	5.3	53.25	28.97	13.3	34.6	17.47	2.70	2.70	49.39	42.53	34.4	116.4	3.20	1.74	13.3	34.6	
16-Dec-88	154	16.18	3.0	5.0	70.87	32.46	26.0	34.0	16.34	2.90	2.90	48.94	42.16	36.6	123.9	4.26	1.95	26.0	34.0	
17-Dec-88	155	16.71	2.9	4.8	89.15	35.32	42.6	31.9	14.54	2.30	2.40	48.43	41.78	28.8	97.4	5.36	2.13	2.88	31.9	
18-Dec-88	156	19.09	2.5	4.2	93.51	40.18	32.9	25.6	13.45	2.10	2.10	48.02	41.41	26.0	86.1	6.83	2.42	1.65	25.6	
19-Dec-88	157	13.55	4.6	5.9	46.28	28.02	10.1	22.6	11.36	2.10	2.10	47.57	41.04	25.8	87.3	2.78	1.69	10.1	22.6	
20-Dec-88	158	16.26	4.1	4.9	53.94	34.22	10.3	28.4	11.39	2.00	2.00	47.11	40.86	24.3	82.4	3.25	2.06	10.3	28.4	
21-Dec-88	159	16.02	4.1	5.0	55.45	33.43	8.8	28.0	10.10	2.00	2.00	46.66	40.29	24.1	81.7	3.34	2.01	8.8	28.0	

Table A5. Calculated period averages of the data
calculations for system PL1

Wastewater %	Flow Rate Measured	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 10.4 g VSS/L reactor				
		PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (g COD/g VSS.d)	Soluble (g COD/g VSS.d)	Total (g COD/g VSS.d)	Soluble (g COD/g VSS.d)	Total (%)
		Q (L/d)																	
CTMP-N	50%	10.31	4.10	9.31	11.94	6.53	37.90	2.86	1.45	2.08	55.2	39.8	23.3	82.9	0.72	0.40	0.15	56.62	37.90
	60%	15.70	2.90	5.16	20.16	11.71	34.63	6.92	1.86	2.05	53.2	40.1	20.2	82.3	1.21	0.70	0.25	37.02	36.40
	70%	16.44	3.78	5.23	25.96	14.35	36.87	10.39	1.82	1.90	53.3	42.1	23.0	77.8	1.56	0.96	0.33	36.87	36.78
	80%	15.81	3.94	5.09	38.40	16.49	49.56	13.89	1.91	2.00	64.5	43.9	25.1	86.0	2.31	0.99	1.15	49.56	47.99
	90%	14.99	3.45	5.52	31.22	19.83	43.20	16.59	2.19	2.22	54.9	44.8	29.5	99.7	1.88	1.19	0.81	43.20	45.99
	100%	14.87	3.74	5.49	42.74	22.37	38.28	18.22	2.59	2.60	55.0	48.8	36.3	122.9	2.57	1.35	1.07	39.70	39.07
TMP	100%	16.68	3.20	4.85	23.10	14.20	29.01	9.79	2.77	2.77	52.1	44.8	37.1	125.6	1.39	0.85	0.39	29.01	41.41
CTMP-H	100%	16.15	3.40	5.01	63.99	32.08	34.17	14.13	2.38	2.39	48.5	41.8	29.6	101.0	3.85	1.93	0.94	22.85	34.17

APPENDIX B
PL2 EXPERIMENT RECORDS

Table B1. Daily operating data of system PL2

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N2 (%)	CH4 (%)	CO2 (%)						
START-UP CTMP-N															
15-Jul-88	0														
16-Jul-88	1				8.40										
17-Jul-88	2														
18-Jul-88	3														
19-Jul-88	4	200	0	0	7.80		83	0	1						
20-Jul-88	5	155	0	0	7.34		93	1	0						
21-Jul-88	6	140	0	0	6.95		70	20	5						
22-Jul-88	7	165	0	0	7.00		51	37	7						
23-Jul-88	8	15	0	0			36	51	8						
24-Jul-88	9														
25-Jul-88	10	0	0	0	7.00		23	65	7	1240					
26-Jul-88	11	0	0	0	7.05		60	28	6						
27-Jul-88	12	50	0	0	6.80		44	41	8	1360	0.408	0.188			
28-Jul-88	13	0	0	0	6.85		56	30	8						
29-Jul-88	14	384	0	0	6.60		33	51	10	1890					
30-Jul-88	15	350	0	0			15	67	12						
31-Jul-88	16	320	0	0			7	75	12						
01-Aug-88	17	300	0	0			7	75	12						
02-Aug-88	18	375	0	0	6.78										
50% CTMP-N															
03-Aug-88	19	285	0	0	6.83		5	77	12	2460	0.692	0.466			
04-Aug-88	20														
05-Aug-88	21	310	0	0						2740	1800				
06-Aug-88	22														
07-Aug-88	23	270	0	0			5	79	10						
08-Aug-88	24	490	0	0	6.49	1450	6	77	11	3710	1720				
09-Aug-88	25	315	0	0											
10-Aug-88	26	300	0	0						2540	1720	0.329			
11-Aug-88	27	310	0	0	6.90	1200	5	78	11	2740	0.422				
12-Aug-88	28	330	0	0											
13-Aug-88	29									1550					
14-Aug-88	30	150	0	0	6.90	1170	4	78	12	2200	1550				
15-Aug-88	31	125	0	0											
16-Aug-88	32	145	0	0						2020	1260				
17-Aug-88	33	110	0	0											
18-Aug-88	34	110	0	0			29	55	9						
19-Aug-88	35	220	0	0			11	71	11	2490	1410	0.512	0.419		
20-Aug-88	36														
21-Aug-88	37														
22-Aug-88	38	95	0	0	7.01	950	8	75	11	1860					
23-Aug-88	39	215	20	0											
24-Aug-88	40	125	0	0						2020	1720	0.604	0.489		

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)						
25-Aug-88	41	125	0	0	7.09	1140	5	78	11	2850	1390				
26-Aug-88	42	75	0	0											
27-Aug-88	43									2000	1330				
28-Aug-88	44	105	0	0	7.05	1230	3	82	9	2330	1470				
30-Aug-88	46	80	20	0						2320	1710		0.279		
31-Aug-88	47	35	0	0											
01-Sep-88	48	60	0	0											
02-Sep-88	49	60	0	0											
03-Sep-88	50														
04-Sep-88	51														
05-Sep-88	52														
06-Sep-88	53	30	0	0	7.02	1230	4	81	9	1980	1430		0.255		
07-Sep-88	54	35	0	0											
08-Sep-88	55	20	0	0						1900	1580				
09-Sep-88	56	35	0	0											
10-Sep-88	57														
11-Sep-88	58														
12-Sep-88	59	55	0	0	7.16	1250	0	80	14	1940	1270				
13-Sep-88	60	60	0	0											
70% CTMP-N															
14-Sep-88	61	95	0	0						2710	1780	0.584	0.352		
15-Sep-88	62	195	0	0											
16-Sep-88	63	285	0	0	7.17		3	81	10	3040	1850				
17-Sep-88	64														
18-Sep-88	65														
19-Sep-88	66	290	0	0						3470	2190				
20-Sep-88	67	280	0	0	7.14	1550	3	79	12	2980	1730	0.667	0.552		
21-Sep-88	68	110	0	0											
22-Sep-88	69	220	0	0	7.01		3	79	12	2790	1670				
23-Sep-88	70	220	0	0											
24-Sep-88	71														
25-Sep-88	72														
26-Sep-88	73	165	0	0											
27-Sep-88	74	230	0	0	6.95	1500	3	77	14	4150	2000				
28-Sep-88	75	125	0	0											
29-Sep-88	76	150	0	0	7.01		4	76	14	2960	1720	0.630	0.556		
30-Sep-88	77	155	0	0						2500	1570				
01-Oct-88	78														
02-Oct-88	79														
03-Oct-88	80	160	0	0	7.16	1380	3	80	11	2260	1650				
04-Oct-88	81	200	0	0											
05-Oct-88	82	255	0	0						3180	2080	0.598	0.490		

		CLARIFIER										FEED										PREACIDIFICATION TANK									
Date	Day	Time (hrs)	Time (min)	Elapsed Time (hrs)	Biogas Meter Reading	Effluent Volume (L)	Room T (C)	Solids Volume (mL)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)				
									Ace (mg/L)	Pro (mg/L)	But (mg/L)								Ace (mg/L)	Pro (mg/L)	But (mg/L)										
13-Nov-88	121	11	60	26.33	7117		33.2	198	1150	140	0	6.11	6750	3970			2.27	850	115	0	8.33	1770	6020	3180							
14-Nov-88	122	10	58	23.13	7259	14.91	33.0	207	1285	200	0	6.11	8050	4260	1.763	1.551	1.70	940	150	0	8.33	1770	6310	3380	1.406	1.074					
15-Nov-88	123	15	2	28.07	7412	18.07	33.2	330	1215	145	0	6.11	8050	4260	1.763	1.551	2.93	915	155	0	8.33	1770	6310	3380	1.406	1.074					
16-Nov-88	124	9	39	19.62	7513	10.29	33.8	132	1265	180	0	6.11	9650	4740			2.55	1000	150	0	6.11	1770	6090	4050							
17-Nov-88	125	12	9	26.50	7668	17.19	32.5	217	1210	150	0	6.11	10370	5510			3.10	975	145	0	6.11	1770	7320	5020							
18-Nov-88	126	11	30	23.35	7791	15.08	32.5	190	1260	105	0	6.12	10370	5510			2.60	1010	150	0	7.01	2580	7320	5020							
19-Nov-88	127	14	44	27.23	7943		34.2	150	1400	210	0	6.12	9400	5010	1.801	1.368	2.00	1025	150	0	7.01	2580	8530	5660	0.967	0.788					
20-Nov-88	128	13	6	22.37	8068		34.4	136	1560	165	0	6.08	8150	5620			1.80	880	105	0	7.77	2580	7570	4840							
21-Nov-88	129	11	11	21.90	8184	13.80	33.8	250	1315	250	0	6.08	8150	5620			2.00	1105	155	0	7.77	2580	7570	4840							
22-Nov-88	130	10	20	23.33	8333	13.45	33.0	100	1455	225	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
23-Nov-88	131	10	25	24.08	8457	13.75	33.8	180	1425	215	0	6.08	9140	5910	1.850	1.493	2.47	1195	170	0	6.08	1450	8040	5180							
24-Nov-88	132	10	50	24.42	8494	0.00	33.8	180	1425	215	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
25-Nov-88	133	11	40	24.83	8604	12.86	33.0	200	1405	225	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
26-Nov-88	134	11	2	23.37	8721	15.78	32.0	145	1385	210	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
27-Nov-88	135	14	25	27.38	8855	14.97	33.2	80	1425	215	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
28-Nov-88	136	9	51	19.43	8935	15.88	33.0	70	1425	215	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
29-Nov-88	137	10	10	24.15	9051	15.88	33.0	70	1425	215	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							
30-Nov-88	138	8	50	22.83	9158	15.49	32.8		1425	215	0	6.08	9140	5910	1.850	1.493	2.30	1205	135	0	6.08	1450	8040	5180							

Table B2. Daily operating record comments of system PL2

DATE	PUMP SETTING	COMMENTS
15-Jul-88	3:30%	Recycle pump clicking. PA tank height stable.
16-Jul-88		UASB volume to 3.5 L - opened to release pressure - reactor tilted to avoid blocking of the effluent port. Recycle pump labouring - oiled the pump and moved the line. Volume of the PA tank low (1.6 L) - topped from the feed tank. Clarifier - no problems detected.
17-Jul-88		Channelling observed in the UASB sludge bed. Volume of the PA tank low (1.2 L) - topped from the feed tank.
18-Jul-88	3:25%	Gas bubbles in the sludge bed. Recycle line moved. Pump decreased @ 09:50. Feed tank emptied and CTMP-N was added.
19-Jul-88	3:20%	Gas bubbles in the sludge bed. No channelling observed. Fibers were observed on top of the sludge bed. Spill - pump off @ 08:55 - 09:10 to clean up. Replaced one of the lines. All other lines walked. Pump decreased @ 11:45. ACCIDENT see PL1. Cleaned the overflow port of the clarifier.
20-Jul-88		Fibers on top of the sludge bed. Gas bubbles in the pump lines. PA tank error - pH sample taken after the tank was topped off. Crushed biomass in the influent line. Line from the clarifier was shortened. Settled solids observed in the clarifier. A rubber stopper was put on top of the clarifier to seal it. CH4 content is low. The gas meter has not changed since the 17th - the top screw on the lid was found open -> closed. Meter reading 4884. Feeding from the basement feed tank was commenced.
21-Jul-88	3:13% 3:27%	PA samples taken after topping. Pump decreased in the morning because the measure flow rate was high. Pump increased @ 14:00 since VFA O.K. PA tank 0.4 L of effluent was added @ 17:00.
22-Jul-88		Supernatant in the UASB reactor is very dark in color. Feed and recycle lines walked. Solids at the clarifier base were stuck - poked with mixing stick to remove the blockage. PA tank 0.4 L of effluent added @ 15:00. Left hand clamp tightened. 0.25 L effluent added @ 16:00.
23-Jul-88		UASB reactor - no fiber collected in the sludge bed. Growth/scum on top of the biorings. PA tank 0.2 L effluent added.
24-Jul-88		
25-Jul-88	3:46%	Cold room feed tank does not adequately recirculate the feed -> no fiber in the feed. Restart @ 12:30.
26-Jul-88	3:55%	@ 07:30 PA tank empty. N2 is high in UASB. 2 L effluent added to PA. Feed line choking. Intake line collapsed - fixed. Changed feed line tubing to 3/8" silicone. PA tank filling up resulted, -> changed back to the original tubing. Installed Y connector in main feed recycle line to diminish sucking back of the feed by basement pump. PROBLEM: acid in feed is zero.

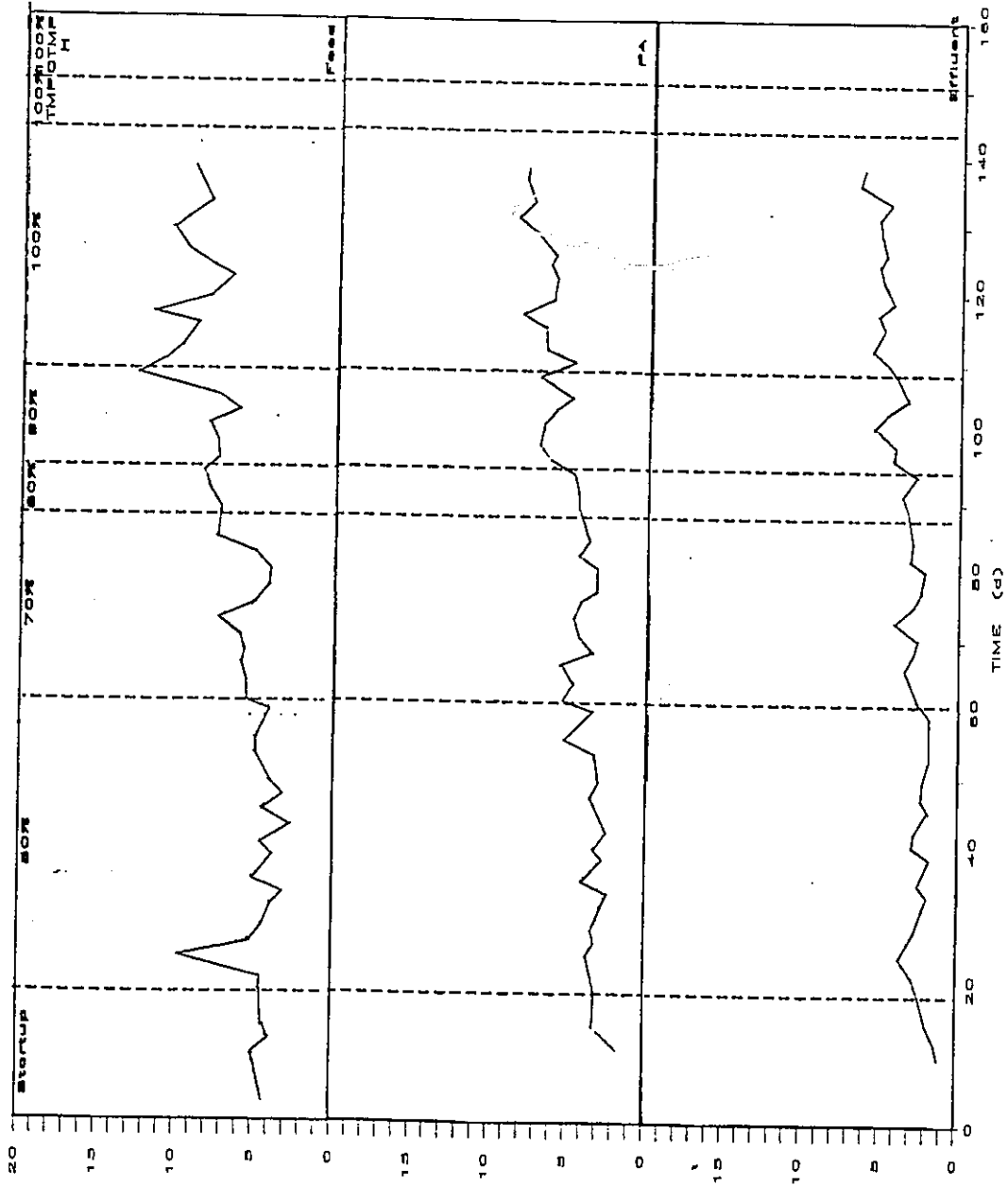
27-Jul-88		PA tank very low. Low acid problem is caused by aeration of the basement feed tank by the stirrer in the tank. VFA in freshly thawed barrel: 1,100 mg/L acetate, in freshly thawed bucket: 935 mg/L. Feed line seems blocked.
28-Jul-88		Installed 100 L stainless steel feed tank in the fermenter room. Tank has the tendency to vortex. The tank recycle line goes directly to the reactors PL1 and PL2. Shortened reactor feed line to avoid fiber accumulation. Fresh feed added. Sampling ports after the main feed recycle line installed, take VFA samples from this location.
29-Jul-88	3:65%	PA tank liquid volume O.K. Sediment in the clarifier is dilute.
30-Jul-88		Sediment thick, some does not exit the clarifier removal port.
31-Jul-88		
01-Aug-88	3:55%	Increased feed rate @ 11:00. Small fibers on sludge bed from the 1.2 to 2.0 L tank mark.
02-Aug-88	3:60%	Brown foam on top of the UASB @ 10:40. Changed the UASB recycle pump tubing. Conducted Imhoff test on feed and effluent (1 L sample settle 45 min, gently stir, settle 15 min more). Feed contains 4.5 mL sediment/L, effluent: 2.4 mL/L. A different clarifier design may be needed.
03-Aug-88		ACCIDENT: 5 L feed with no chemicals was added. The rest of the feed is from the previous barrel -> lower VFA concentration. PA tank is cloudy pinkish brown. Line from the clarifier to the PA tank is blocked - cleaned. Raised the clarifier to increase head. Fiber in from 1.45 to 2.15 L in the UASB - grey in color.
04-Aug-88		POWER FAILURE @ 14:30 all pumps off. Pumps on @ 08:00. 1 cm thick brown foam on top of the UASB biorings. VFA sample taken @ 17:00.
05-Aug-88		Placed a weight at the end of the feed tank recycle line to hold it down and prevent the vortex. Walked pump lines. Brown scum/growth in the PA tank. PA tank temperature is 40 C.
06-Aug-88		Line from the clarifier to the PA tank is blocked. Removed stopper,
07-Aug-88	3:61%	cleaned line. Black scum/growth on PA tank wall.
08-Aug-88	3:60%	Pump decreased @ 13:30. Greyish fiber in the UASB above the sludge from 1.55 L to 1.9 L. VFA high. Growth on PA tank wall near exit to the UASB reactor. COD sample taken from the port - solids high - COD T high.
09-Aug-88	3:59%	Pump decreased @ 14:00. Greyish fiber on sludge bed from 1.6 L to 1.8 L mark in UASB. Growth on walls. All lines walked.
10-Aug-88	3:60%	Pump increased @ 10:45. Deposition on UASB wall. PA tank now filling. Recycle pump changed to same as PL3. The change in the flow is not much less than it was originally. The high recycle rate in PA1 & 2 may prevent wall growth. IDEA: match pump and veins-> same rate. UNSUCCESSFUL
11-Aug-88	3:61%	Pump increased @ 11:35. UASB bed has no fiber on top. There is 2 cm of scum plus biorings. More growth/deposition on PA tank walls.
12-Aug-88		0.5 cm of fiber on top of the sludge bed.

13-Aug-88	3:59%	Pump decreased @ 10:40.
14-Aug-88		UASB pump is recycling air. Feed tank almost empty. Cleaned.
15-Aug-88		Gas accumulation under scum in the UASB reactor.
16-Aug-88		Feed recycle rate is not as high as it was originally. Squeezed the tubing to remove fiber build up in the line -> flow rate improved.
17-Aug-88		No feed is recycling - fiber blockage removed from the recycle line -> flow resumed. Low solids content in the feed expected (sampled before the problem was fixed). Filled the gas meter with water to the operating level.
18-Aug-88		PA tank empty - probably due to blocked tubing. Changed all Harvard pump tubing and walked the recycle pump tubing. Air entered the reactor. Cleaned the overflow tube.
19-Aug-88		
20-Aug-88		Granules up to 1.65 L.
21-Aug-88		
22-Aug-88	3:61	Granules up to 1.6 L in the UASB reactor. Little wall growth in the PA.
23-Aug-88		Walked all lines.
24-Aug-88		Granules in the UASB to 1.65 L. Washout of fiber observed from the 2.1 L mark to 1.65 L. Added 250 mL water to the gas meter. Liquid level mark in the UASB reactor at 4.5L. PA tank topped to 1.4 L before the sample for analysis was taken. Squeezed feed line in pump.
25-Aug-88		Harvard pump off to fix tank recycle. ERROR not turned on again until 26/08.
26-Aug-88		Pump on 09:30. PA not recycling well, unplugged, better.
27-Aug-88		Added 250 mL water to the gas meter.
28-Aug-88		Feed tank recycle low. No sediment to remove.
29-Aug-88		Cleaned feed tank, flushed recycle line. Good recycle.
30-Aug-88		Walked all tubing. Changed sample port septa. Feed recycle poor - fixed @ 11:30. ERROR: left pumps off until 31/08.
31-Aug-88		Pumps were off - turned back on @ 09:00. Cleaned the overflow tube. Effluent is very thick. VFA sample of the PA tank is unusually cloudy.
01-Sep-88		
02-Sep-88		Settling test samples taken. 20 mL from 1.7 L port plus 20 mL from the port at the bottom of the reactor. Walked all lines.
03-Sep-88		Cleaned the feed tank and squeezed tubing to remove fiber build up.
04-Sep-88		
05-Sep-88	3:55% 3:53%	Started effluent collection. @ 16:30 cleaned the feed tank. Lowered the feed rate. Walked all
06-Sep-88		tubing.
07-Sep-88	3:66%	
08-Sep-88	3:70%	
09-Sep-88		Walked all tubing. Acetate in the barrel is 990 mg/L.
10-Sep-88		Added 300 mL of water to the gas meter.
11-Sep-88		Sludge bed samples were taken from the UASB for TSS, VSS, COD and activity tests. 20 mL from the 1.725 L port, 20 mL from the < 0.5 L port. Combined top and bottom sample for the activity test.

12-Sep-88		INCREASED CTMP CONCENTRATION - 40% DILUTION.
13-Sep-88		Walked all tubing. Water added to the gas meter. UASB liquid mark @ 4.5 L -> washout has occurred.
14-Sep-88		INCREASED CTMP CONCENTRATION - 30% DILUTION.
15-Sep-88	3:73%	VFA up @ 11:30.
16-Sep-88	3:80%	Lines passed through the Harvard pump on their own - tightened cable ties to fix.
17-Sep-88	3:76%	
18-Sep-88	3:75%	
19-Sep-88		Cleaned the overflow tube. Feed diluted to 40% topped off with 30% feed @ 17:20. No recycle back to the feed tank - cleaned lines
20-Sep-88		@ 12:15 a bucket from the previous feed barrel is fed. The bottom of the bucket fell out during thaw - some fibers lost. Walked the Harvard pump lines.
21-Sep-88		
22-Sep-88	3:73%	Cleaned the feed tank.
23-Sep-88		Walked all tubing and changed the sample port septa.
24-Sep-88		Sludge bed in the UASB "fluidized" - a lot of mixing and gas movement.
25-Sep-88		
26-Sep-88	3:75	Effluent collection started in the morning. Walked all tubing. PA tank recycle rate high (4.1) compared to other systems. Lowered to 3.5.
27-Sep-88	3:73	Crushed biomass in the recycle line from the UASB to the PA.
28-Sep-88	3:78	Crushed biomass in the recycle line from the UASB to the PA. Granules in the UASB are pinkish in colour - coated with fiber.
29-Sep-88		Walked the Harvard pump lines.
30-Sep-88		
01-Oct-88		ERROR: Harvard pump was off.
02-Oct-88		Harvard pump turned back on.
03-Oct-88		Walked the Harvard pump lines and changed the feed recycle line. Cleaned the overflow tube. 80 mL sludge is removed from the UASB sludge bed for tests.
04-Oct-88		The UASB reactor sludge bed is fluid. When pumps off the settled volume is 1.6 L.
05-Oct-88	3:63%	@ 15:30 the bed was fluid. Added 500 mL water to the gas meter.
06-Oct-88	3:58%	@ 12:15 walked Harvard pump tubing.
07-Oct-88		
08-Oct-88		Settler solids frozen in fridge.
09-Oct-88		Feed tank empty.
10-Oct-88		Added 250 mL of water to the gas meter.
11-Oct-88		Walked the Harvard pump lines and changed the recycle pump line. INCREASED CTMP CONCENTRATION - 20 % DILUTION.
12-Oct-88	3:62%	
13-Oct-88		Unusually large volume of sediment (380 mL) in the clarifier. Clarifier pH is 6.30. Walked Harvard pump lines and changed 1.
14-Oct-88		UASB overflow tube is blocked and the liquid level in the UASB is 3.8 L. Opened the UASB to release the pressure and lower the liquid level. The UASB sludge bed is fluid. Spill from the top of the clarifier - cleaned the overflow tube. The clarifier COD t is 5,400 mg/L and COD S is 2,730.

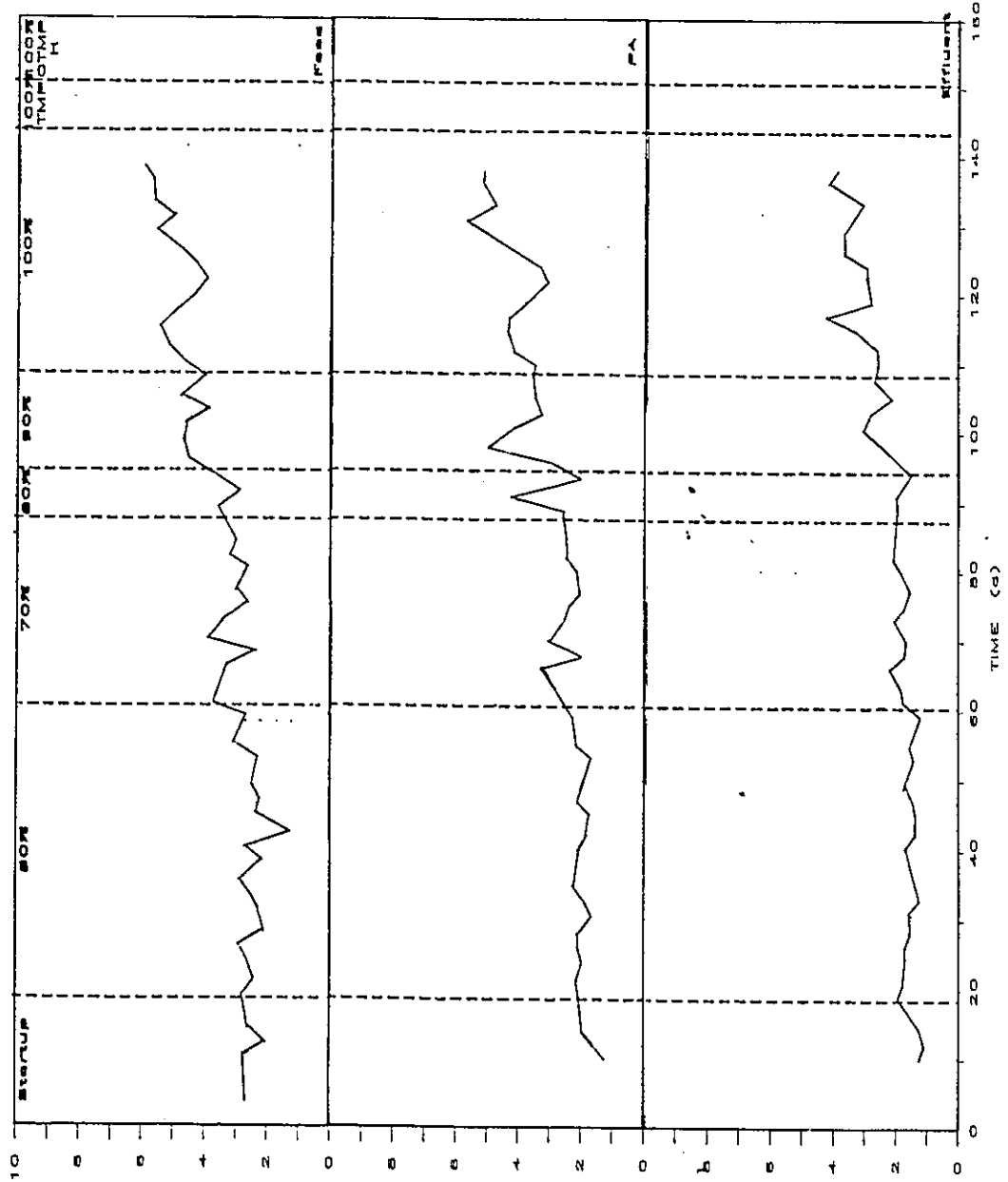
15-Oct-88		Overflow tube is clogged - spill occurred - cleaned overflow.
16-Oct-88		Feed is not recycling - tubing clogged - fixed @ 14:30. Low volume of solids in the clarifier.
17-Oct-88		All Harvard pump lines were walked. Clarifier pH 6.49 and alkalinity is 1,050 mg/L.
18-Oct-88		INCREASED CTMP CONCENTRATION - 10 % DILUTION. Added 200 mL water to the gas meter. Clarifier settled solids TSS is 1.507 mg/L and VSS is 1.198.
19-Oct-88	3:65%	Clarifier COD T 6,100, COD S 5,110.
20-Oct-88	3:70%	Walked the Harvard pump lines. Acetate level in the UASB is high. Clarifier pH is 6.56.
21-Oct-88	3:66%	Feed tank recycle pump not working - Tee connector unclogged. Note: clarifier solids were low the previous days because the recycle was blocked and solids did not enter the system -> low COD T. Clarifier COD T is 7,330 and COD S is 4,880 mg/L.
22-Oct-88		Settler solids back up to normal. Room temperature low -> auxillary heater turned on.
23-Oct-88		Overflow tube blocked - spilled into catch bucket - cleaned overflow.
24-Oct-88	3:72%	Clarifier COD T 7,280 and COD S 4,270.
25-Oct-88	3:70%	All Harvard pump lines walked. Clarifier pH 6.41 and alkalinity is 1,150 mg/L.
26-Oct-88		Clarifier COD T is 6,760 mg/L and COD S is 5,790 mg/L.
27-Oct-88		
28-Oct-88		Sludge bed very expanded. 7 cm of fiber floating on top of the PA tank - stirred to settle but it wouldn't. Fibers blocking the overflow line from the clarifier to the PA tank. Clarifier emptied and refilled with feed to solve the problem. No COD sample was taken. Walked the Harvard pump lines.
29-Oct-88		
30-Oct-88		
31-Oct-88		Overflow tube blocked - cleaned the tube. 650 mL of water added to the gas meter. Sludge bed is expanded. Fiber sludge at the bottom of the feed tank. Walked the Harvard pump lines.
01-Nov-88	3:65%	INCREASED CTMP CONCENTRATION - 0 % DILUTION. Bed expanded. Granules large, even on top. Clarifier pH is 6.32 and alkalinity is 1,240 mg/L.
02-Nov-88		Sludge bed is fluid and expanded. Clarifier COD T is 5,970 mg/L and COD S is 4,390 mg/L. Clarifier VSS is 0.742 mg/L and TSS is 0.882 mg/L.
03-Nov-88	3:65%	Sludge bed is fluid and expanded. Overflow from the clarifier to the PA tank was cleaned. Walked the Harvard pump lines.
04-Nov-88		Sludge bed is expanded. ERROR: COD PA tank sample taken after topping the PA tank. COD S sample not clear after centrifuging. Clarifier pH is 6.1, COD T is 8,572 mg/L and COD S is 5,520 mg/L.
05-Nov-88		
06-Nov-88		

07-Nov-88	3:63%	Sludge bed is fluid. UASB VFA high. Clarifier pH is 6.17 and alkalinity is 1,450 mg/L. Clarifier COD T is 9,350 mg/L and COD S is 5,300 mg/L.
08-Nov-88	3:69%	All Harvard pump lines changed and the recycle pump walked.
09-Nov-88	3:64%	UASB VFA high but the flow was 1.5 L greater than normal. Clarifier COD T 9,850, COD S 5,000, TSS 2.553, VSS 2.080 mg/L.
10-Nov-88	3:66%	Settler solids bulking, 2/3rd removed, topped with feed. Walked Harvard pump lines. UASB VFA high.
11-Nov-88	3:50%	VFA high, Q decreased by 25%.
12-Nov-88	3:55%	
13-Nov-88	3:60%	
14-Nov-88		Overflow tube blocked - effluent on lab bench and in the catch bucket - tube cleaned - poured content of the catch bucket into the effluent. 600 mL water added to the gas meter. Walked the Harvard pump lines.
15-Nov-88		Solids floating in the clarifier - settled when stirred. Changing the recycle pump tubing. Clarifier pH 6.00 and alkalinity 1,330 mg/L.
16-Nov-88	3:70%	Granules in the overflow tube. Clarifier COD T 7,330, COD S 4,550, TSS 1.467, VSS 1,211 mg/L.
17-Nov-88	3:72%	Walked the Harvard pump lines.
18-Nov-88		Clarifier COD T 9,400 and COD S 5,740 mg/L.
19-Nov-88		
20-Nov-88		
21-Nov-88		Clarifier COD T 9,400 and COD S 5,740 mg/L.
22-Nov-88	3:77%	All lines moved.
23-Nov-88		Clarifier COD T 14,560, 14,640, COD S 5,220, 5,330 mg/L. Acids run at 2 pm. Pump must have been off.
24-Nov-88		No effluent - pump on but not working. PA tank liquid dark brown in color. Clarifier half full. Restarted pump @ 09:15 but it ceased. Changed pump @ 10:30 and set to 3:77. Walked the Harvard pump lines.
25-Nov-88	3:100	Clarifier COD T 7,920, COD S 5,180 mg/L.
26-Nov-88		Cleaned the overflow tube. Add 420 mL water to the gas meter. Biomass in the UASB recycle line and the overflow tube.
27-Nov-88		
28-Nov-88	3:96%	Moved all Harvard pump lines. VFA high in the UASB. Clarifier COD T is 8,820 and COD S is 5,300 mg/L. Biomass in the UASB recycle line to the PA tank.
29-Nov-88		Clarifier pH is 6.32 and alkalinity is 1,500 mg/L.
30-Nov-88		TERMINATION OF EXPERIMENT Clarifier COD T 8,910, COD S 5,040, TSS 1.638, VSS 1.327 mg/L. 2x100 mL taken for P,N analysis from the feed tank, UASB effluent and the clarifier. Stored in -40 C room on shelf. Bed very expanded. Biomass sample (60 mL) taken from the 1.725 L port and < 0.5 L port. 14:30 system shut off. Bed allowed to settle 30 min. Settled bed volume 2.25 L. Biogas still produced. A lot of bed movement, layered movement. All except the bottom 0.5 L biomass removed into preflushed 2 L plastic jug. Stored in air lock on shelf. Feed line clamped off.



(1/5) 7101 G03

Figure B1a COD_T versus operating time



(1/6) 578795 000

Figure B1b COD_S versus operating time

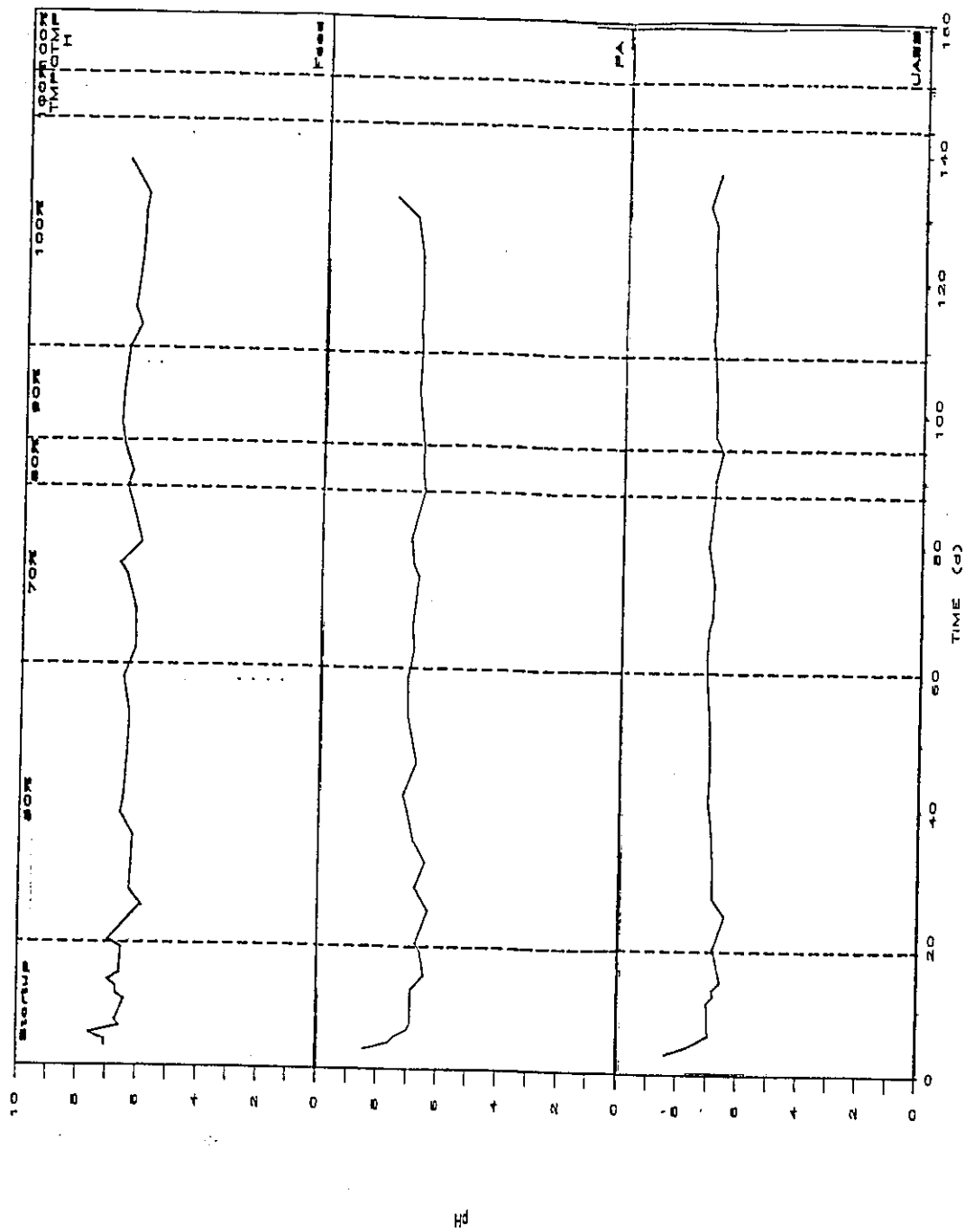


Figure B2 pH versus operating time

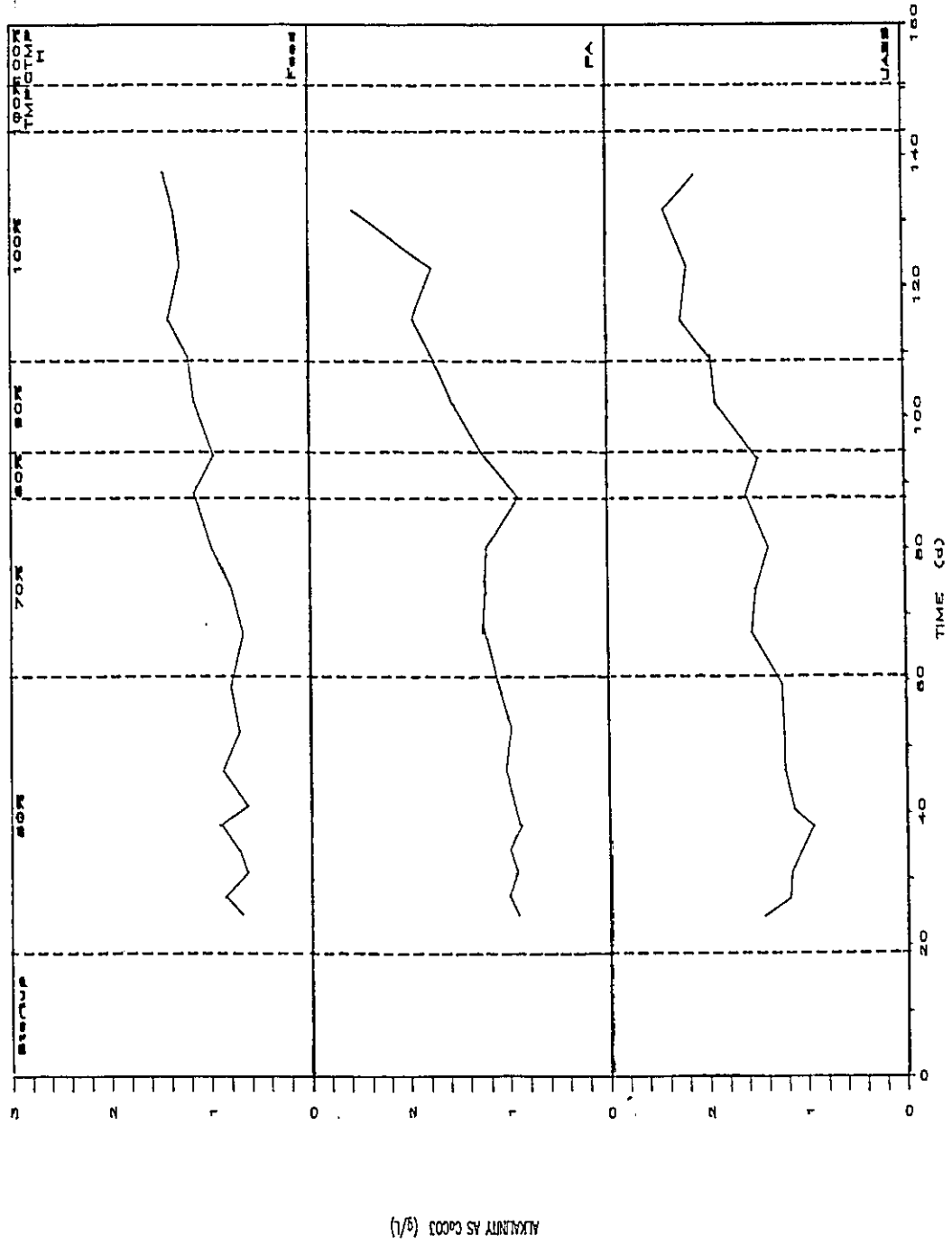


Figure B3 Alkalinity versus operating time

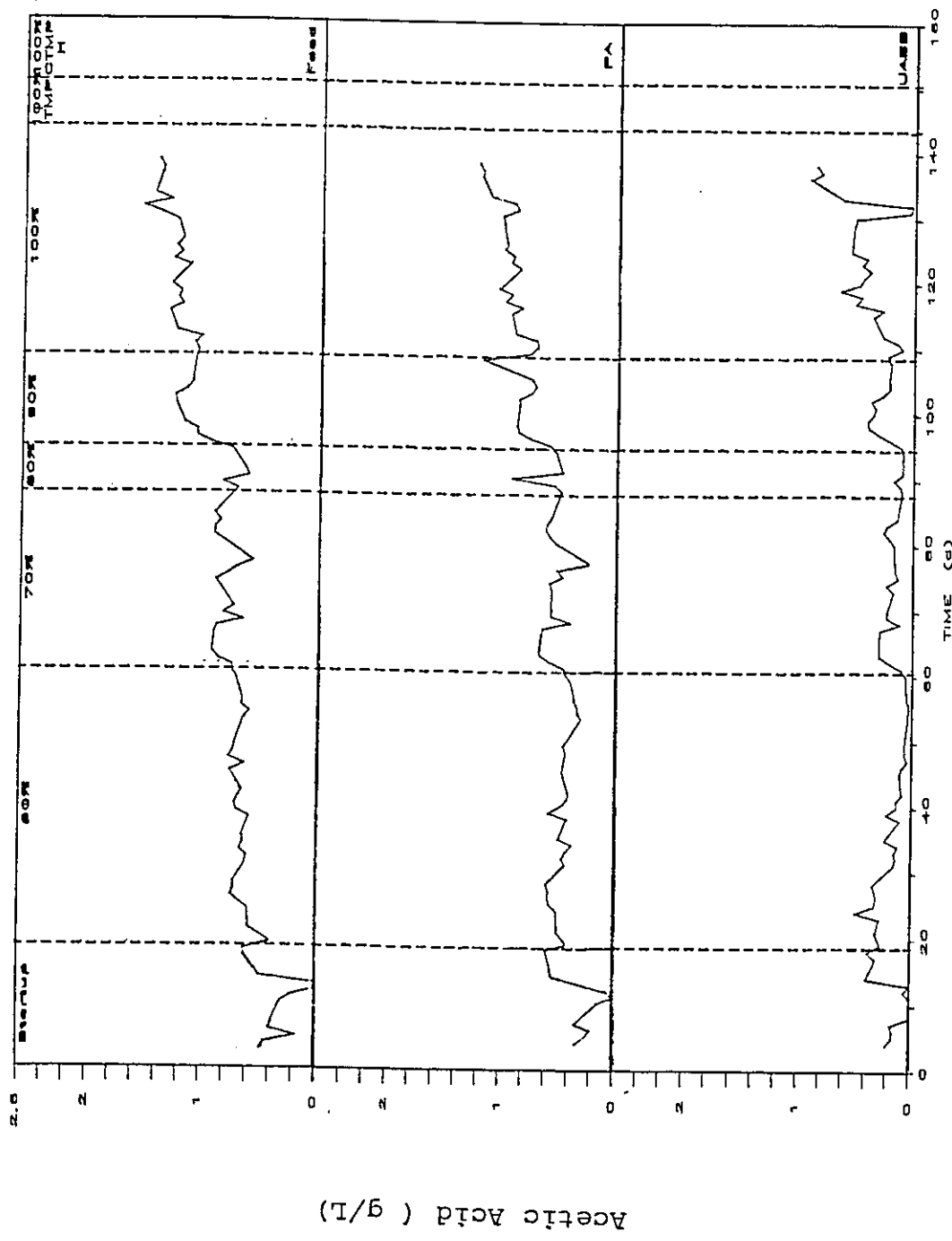


Figure B4a Acetic acid versus operating time

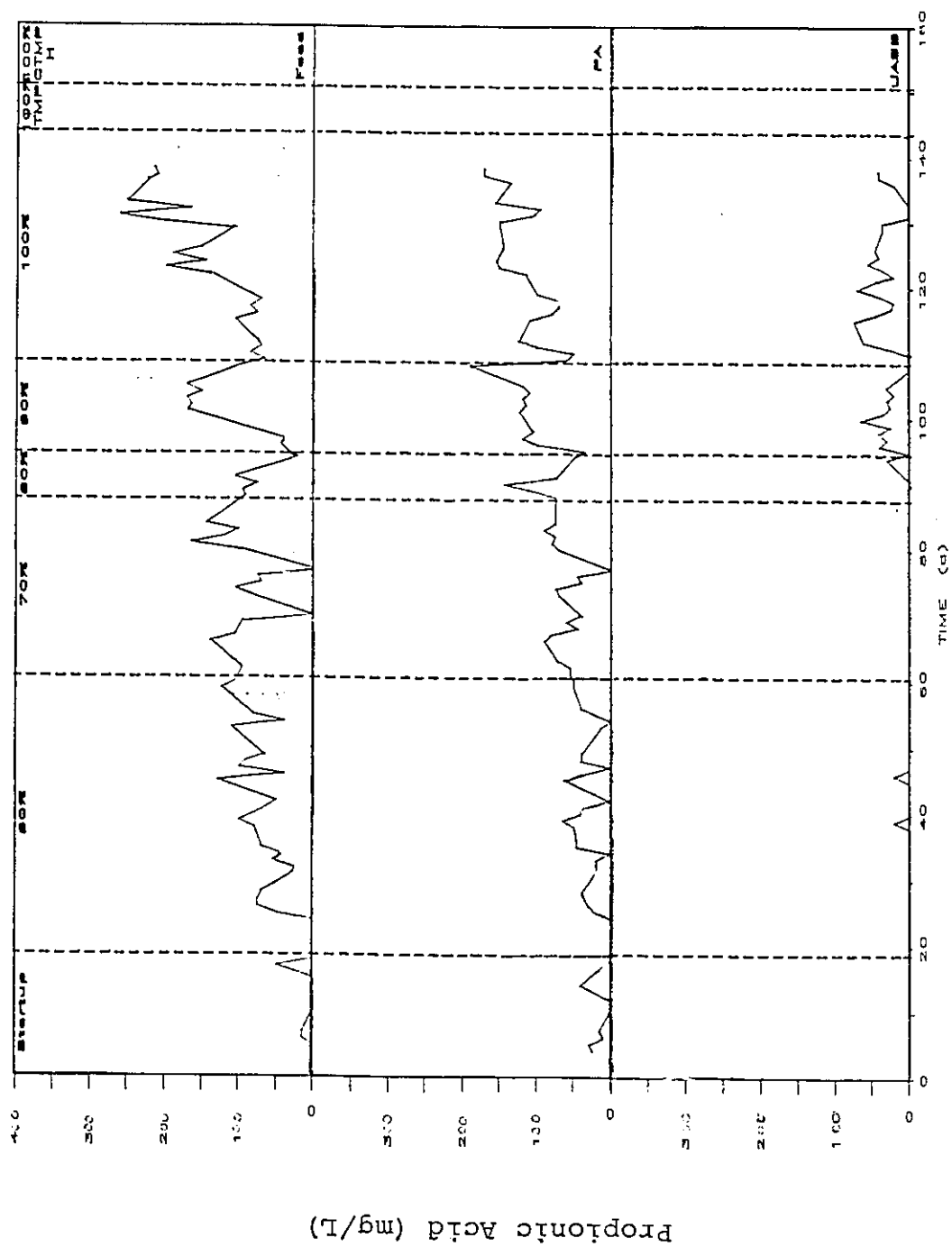
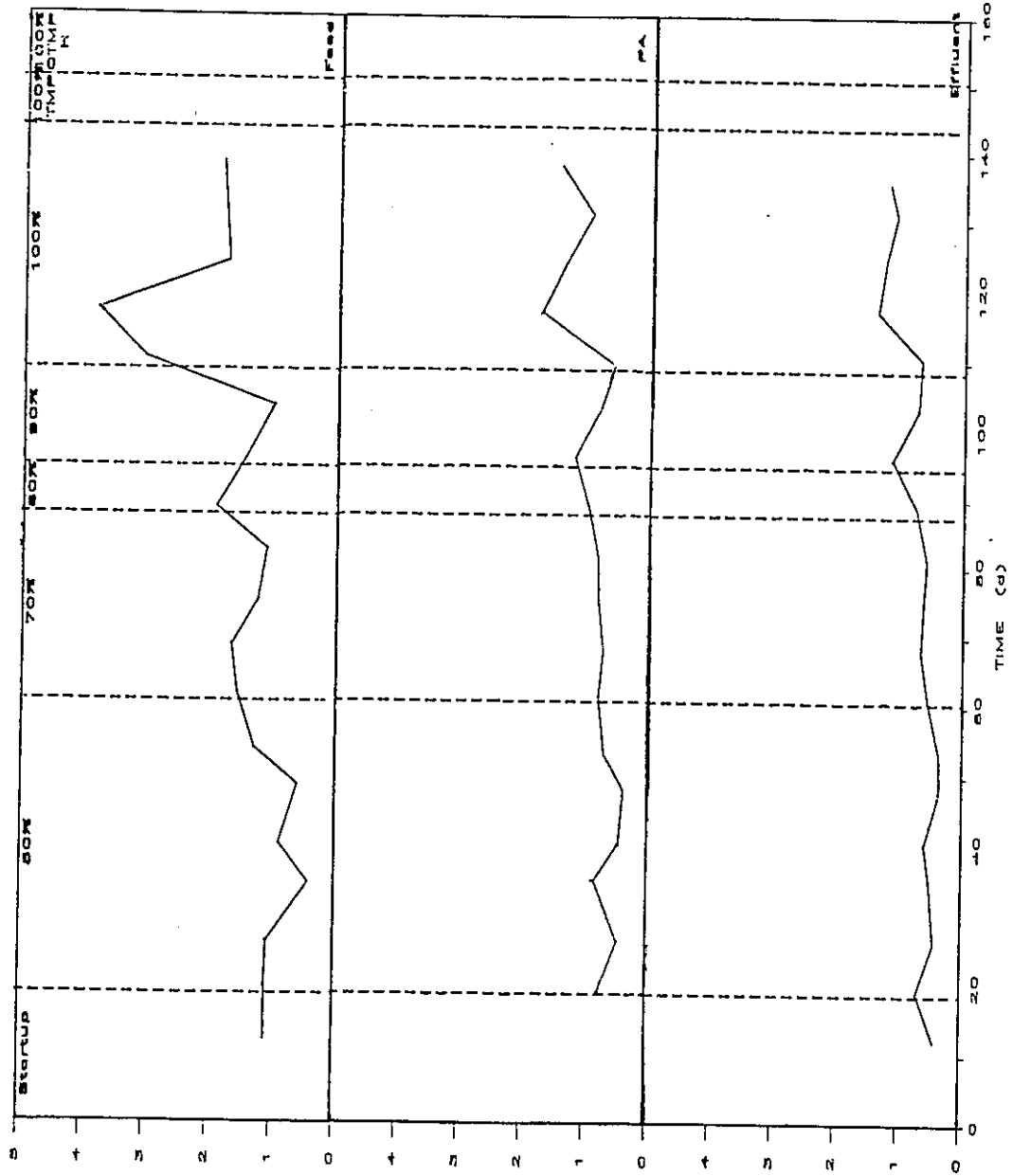
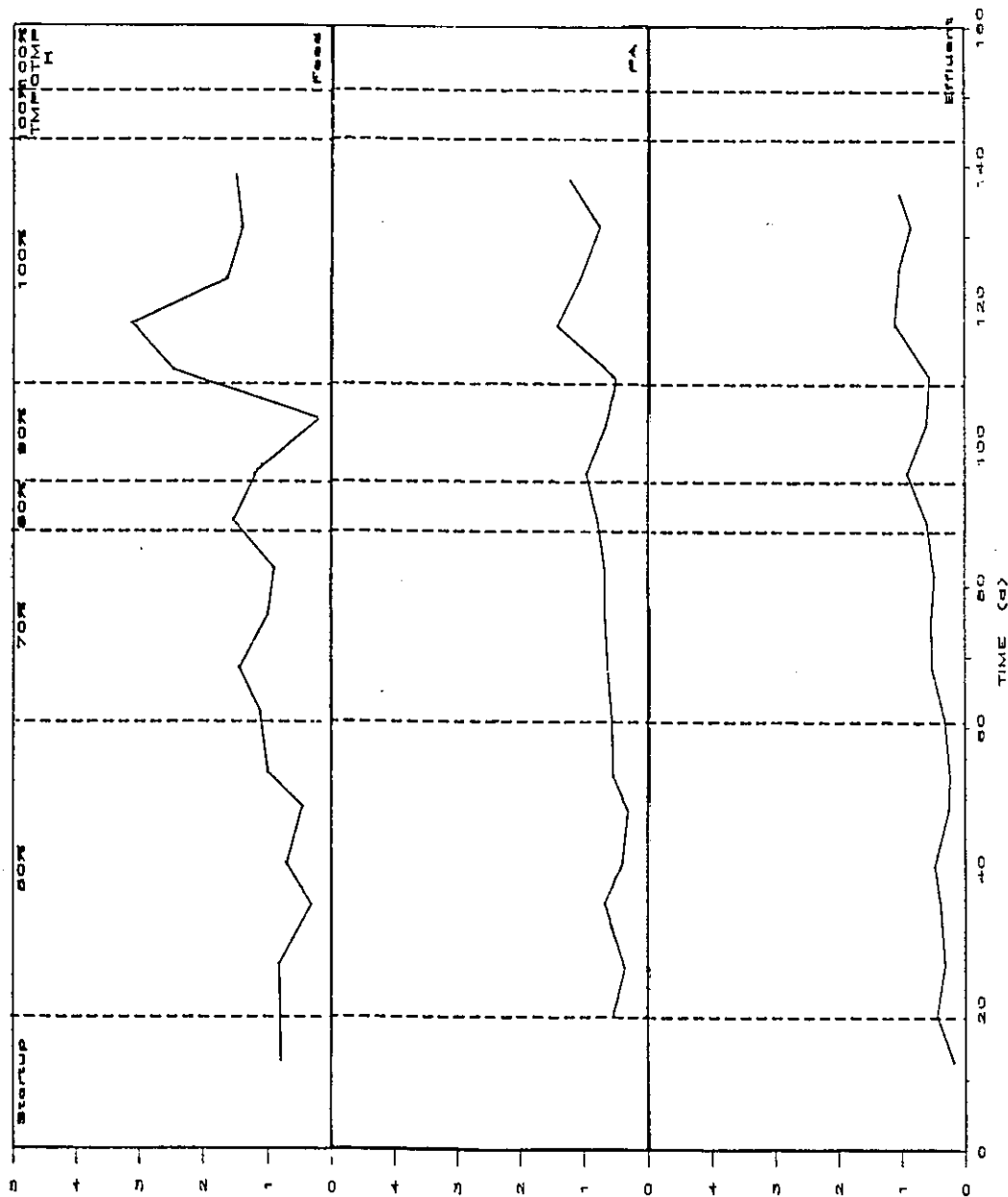


Figure B4b Propionic acid versus operating time



(1/5) SSI

Figure B5a TSS versus operating time



(1/6) SSI

Figure B5b VSS versus operating time

Table B3. Calculated period averages for system PL2

		PREACIDIFICATION TANK																	
Wastewater %	Solids Volume (mL)	FEED																	
		VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA							
Ace (mg/L)	Pro (mg/L)	But (mg/L)	Ace (mg/L)	Pro (mg/L)								But (mg/L)	pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	
CTMP-N 0-50%	103	353	8	4	6.64	761	4403	2477	1.077	0.808	1.31	266	9	0	6.76	1685	2375	0.653	0.481
50%	92	660	66	0	6.36	820	4320	2519	0.867	0.688	1.69	448	28	0	6.87	2068	3430	0.816	0.657
70%	114	820	94	0	6.31	1050	6675	3144	1.371	1.114	2.28	545	62	0	6.97	2559	4193	0.887	0.812
80%	258	715	77	0	6.49	1150	7920	3870	1.878	1.553	2.55	592	90	0	6.70	3027	4557	0.987	0.832
90%	217	1125	112	0	6.71	1340	7900	4397	1.204	0.639	2.35	819	114	0	6.82	3770	6293	1.028	0.832
100%	172	1276	145	0	6.28	1340	9282	5021	2.447	1.980	2.10	957	119	0	7.02	4362	6942	1.249	1.013

Wastewater %	UASB REACTOR										EFFLUENT			
	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
	Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)						
CTMP-N 0-50%	175	0	0	6.88		0	49	8	1497	1210	0.409	0.189		
50%	157	1	0	6.94	1203	0	78	11	2412	1551	0.488	0.375		
70%	184	0	0	7.07	1477	3	78	12	3005	1860	0.812	0.488		
80%	112	6	0	6.87	1550	3	75	18	3260	1817	0.768	0.654		
90%	282	28	0	6.97	1900	2	76	16	4413	2485	0.940	0.775		
100%	449	32	0	7.08	2178	2	75	17	5358	3314	1.128	0.828		

Table B4. Data calculations for system PL2

Date	Flow Rate Measured	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.3 gVSS/L reactor					
		PA (hrs)	UASB (hrs)	Total (g/L/d)	Soluble (g/L/d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	SLR (gCOD/gVSS.d)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	SRR (gCOD/gVSS.d)	SRR/SLR	Total (%)
		Q (L/d)																		
25-Aug-88	41	16.45	1.5	5.0				8.83	1.71	1.90	50.26	37.62	18.9	64.3						
26-Aug-88	42	16.64	2.5	5.2				3.92	1.76	1.90	50.12	37.47	19.6	66.5						
27-Aug-88	43	16.05	2.5	5.2				7.86	1.84	1.90	49.98	37.42	20.2	68.8						
28-Aug-88	44	14.77	2.9	5.5				9.24	1.90	1.96	49.85	37.37	20.9	71.0						
29-Aug-88	45	16.13	3.6	5.4	19.42	10.03	65.3	8.35	1.95	2.00	49.72	37.31	21.4	72.8	1.19	0.61	0.06	0.26	66.3	42.4
30-Aug-88	46	16.13	3.6	5.4				9.69	2.00	2.00	49.56	37.26	21.9	74.5						
31-Aug-88	47	15.79	3.0	5.2				3.71	2.03	2.03	49.45	37.21	22.2	76.4						
01-Sep-88	48	16.07	2.5	5.1	19.28	11.58	43.1	10.10	2.05	2.05	49.18	37.16	22.4	78.2						
02-Sep-88	49	16.16	3.2	5.0				9.55	2.03	2.03	49.04	37.06	22.1	76.0	1.18	0.71	0.51	0.21	43.1	30.2
03-Sep-88	50	16.16	3.2	5.0				9.47	2.03	2.03	48.91	37.00	22.0	74.9						
04-Sep-88	51	17.06	2.1	4.5	24.64	11.66	59.7	10.39	2.00	2.00	48.78	36.96	21.7	73.9						
05-Sep-88	52	13.46	2.9	6.1				8.82	2.05	2.05	48.64	36.90	22.2	76.6	1.61	0.71	0.90	0.27	56.7	36.1
06-Sep-88	53	16.13	2.6	5.4	21.98	13.71	61.5	9.49	2.16	2.16	48.51	36.85	23.3	79.2						
07-Sep-88	54	16.58	2.9	4.9				8.98	2.20	2.20	48.37	36.80	23.8	81.0	1.36	0.84	0.63	0.41	61.5	49.4
08-Sep-88	55	16.58	2.9	4.9				10.06	2.20	2.20	48.24	36.75	23.8	80.8						
09-Sep-88	56	16.00	3.1	5.1	19.56	12.62	53.4	9.69	2.20	2.20	48.10	36.69	23.7	80.7						
10-Sep-88	57	16.43	2.7	5.0				9.55	2.20	2.20	47.97	36.64	23.7	80.6						
11-Sep-88	58	16.43	2.7	5.0				10.28	2.16	2.16	47.83	36.59	23.1	78.7	1.20	0.77	0.64	0.40	53.4	52.3
12-Sep-88	59	16.43	2.7	5.0				13.15	2.20	2.20	47.70	36.54	23.6	80.4						
13-Sep-88	60	16.43	2.7	5.0																
70% CTMP-N																				
14-Sep-88	61	14.35	3.8	5.7	23.84	15.63	62.0	11.02	2.26	2.26	47.62	36.60	24.2	82.3	1.46	0.96	0.76	0.49	62.0	51.6
15-Sep-88	62	15.26	2.6	5.3	25.70	16.00	46.1	11.12	2.26	2.26	47.53	36.65	24.3	82.5						
16-Sep-88	63	17.48	3.1	4.7				11.39	2.30	2.30	47.45	36.71	24.8	84.4	1.67	0.96	0.73	0.49	46.1	47.3
17-Sep-88	64	16.64	3.2	4.9				11.60	2.30	2.30	47.37	36.77	24.9	84.6						
18-Sep-88	65	16.64	3.2	4.9				11.08	2.36	2.30	47.29	36.83	24.9	84.7						
19-Sep-88	66	16.64	3.4	4.9	29.29	16.14	41.9	10.69	2.30	2.30	47.20	36.88	25.0	84.8	1.79	0.99	0.76	0.33	41.9	33.4
20-Sep-88	67	17.83	3.6	4.6				12.63	2.30	2.30	47.12	36.94	25.0	85.0						
21-Sep-88	68	16.83	3.1	4.8	28.96	11.70	49.1	10.14	2.30	2.30	47.04	37.00	25.0	85.1	1.77	0.72	0.87	0.19	49.1	27.0
22-Sep-88	69	16.71	2.7	4.9				9.26	2.28	2.28	46.96	37.06	24.9	84.6						
23-Sep-88	70	15.97	3.0	5.1	29.05	18.23	53.3	11.21	2.30	2.30	46.87	37.11	25.1	86.4	1.72	1.12	0.92	0.64	63.3	57.0
24-Sep-88	71	16.89	2.9	4.9				10.60	2.20	2.20	46.79	37.17	24.1	81.8						
25-Sep-88	72	16.89	2.9	4.9	35.64	16.09	42.8	10.48	2.10	2.10	46.70	37.23	23.0	78.2						
26-Sep-88	73	17.41	3.3	4.7				12.13	2.16	2.16	46.62	37.29	23.6	80.2	2.18	1.02	0.94	0.42	42.8	41.2
27-Sep-88	74	16.06	3.4	5.2	23.67	12.34	42.4	11.28	2.20	2.20	46.54	37.34	24.2	82.2						
28-Sep-88	75	16.88	3.1	4.8				11.76	2.20	2.20	46.46	37.40	24.2	82.3	1.45	0.76	0.62	0.27	42.4	36.8
29-Sep-88	76	16.88	3.1	4.8				13.13	2.20	2.20	46.37	37.46	24.2	82.4						
30-Sep-88	77	16.63	3.3	4.9	20.06	14.48	41.2	11.77	2.20	2.20	46.29	37.52	24.3	82.5	1.27	0.69	0.62	0.42	41.2	47.3
01-Oct-88	78	17.36	3.3	4.7				12.31	2.10	2.10	46.21	37.57	23.2	78.9						
02-Oct-88	79	16.04	2.2	4.3				10.42	2.16	2.16	46.12	37.63	23.2	79.0						
03-Oct-88	80	19.04	2.2	4.3	21.31	13.44	46.6	10.42	2.16	2.16	46.04	37.69	23.6	81.0	1.31	0.82	0.60	0.24	46.6	29.7
04-Oct-88	81	20.15	2.4	4.0	29.57	15.85	36.3	11.39	2.10	2.10	46.00	37.71	23.3	79.2						
05-Oct-88	82	20.15	2.4	4.0				13.54	2.26	2.26	45.96	37.73	25.0	84.9	1.81	1.15	0.96	0.40	36.3	34.6

Date	Day	Flow Rate Measured		HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.3 gVSS/L reactor					
		Q (L/d)	PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)	TSS (g/L)				VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	BLR	SFR	SRR/SLR
06-Oct-88	83	17.71	3.1	4.6						11.19	2.20	2.20	46.92	37.76	24.4	63.1	2.22	0.88	1.33	0.26	59.9	32.2
07-Oct-88	84	16.32	2.8	5.0						10.87	2.20	2.20	45.89	37.77	24.4	63.1	2.22	0.88	1.33	0.26	59.9	32.2
08-Oct-88	85	17.44	3.4	4.7						12.19	2.24	2.24	45.85	37.79	24.9	64.7	2.24	0.88	1.33	0.26	59.9	32.2
09-Oct-88	86	16.02	3.0	5.1						12.36	2.20	2.20	45.81	37.81	24.5	63.2	2.24	0.88	1.33	0.26	59.9	32.2
10-Oct-88	87	16.30	3.4	5.0						12.98	2.20	2.20	45.77	37.83	24.5	63.2	2.24	0.88	1.33	0.26	59.9	32.2
90% CTMP-N																						
11-Oct-88	88	16.27	3.7	5.0						12.63	2.26	2.26	45.73	37.85	25.1	65.2	2.24	1.07	1.20	0.48	63.6	44.9
12-Oct-88	89	16.83	3.6	4.6						13.16	2.00	2.00	45.69	37.87	22.3	75.7	2.24	1.07	1.20	0.48	63.6	44.9
13-Oct-88	90	17.44	3.4	4.7						12.94	2.30	2.30	45.06	37.90	26.6	87.2	2.31	0.82	1.26	0.26	64.4	31.0
14-Oct-88	91	16.02	3.0	5.1						14.05	2.20	2.20	45.62	37.92	24.5	63.4	2.31	0.82	1.26	0.26	64.4	31.0
15-Oct-88	92									12.70	2.30	2.30	45.58	37.94	25.7	87.3	2.37	1.08	1.00	0.04	67.7	59.4
16-Oct-88	93									12.94	2.20	2.20	45.54	37.96	24.6	83.5	2.37	1.08	1.00	0.04	67.7	59.4
17-Oct-88	94	15.70	3.2	5.2						12.79	2.26	2.26	45.50	37.98	25.2	86.6	2.37	1.08	1.00	0.04	67.7	59.4
90% CTMP-N																						
18-Oct-88	95	15.60	2.3	5.2						13.22	2.30	2.30	45.46	38.00	25.7	87.4	2.09	1.27	0.87	0.72	41.0	57.0
19-Oct-88	96	15.51	3.1	5.3						16.21	2.30	2.30	45.42	38.02	25.7	87.4	2.09	1.27	0.87	0.72	41.0	57.0
20-Oct-88	97	18.01	3.7	5.1						16.17	2.30	2.30	45.39	38.04	25.7	87.5	2.33	1.46	1.04	0.71	44.4	48.5
21-Oct-88	98	17.21	2.8	4.7						15.14	2.25	2.25	45.35	38.06	25.2	85.6	2.33	1.46	1.04	0.71	44.4	48.5
22-Oct-88	99									16.07	2.25	2.25	45.31	38.08	25.2	85.7	2.33	1.46	1.04	0.71	44.4	48.5
23-Oct-88	100									16.20	2.30	2.30	45.27	38.10	25.8	87.6	2.43	1.38	0.76	0.46	30.9	33.4
24-Oct-88	101	16.80	3.7	4.9						17.15	2.25	2.25	45.23	38.12	25.2	85.8	2.43	1.38	0.76	0.46	30.9	33.4
25-Oct-88	102	18.23	3.6	4.5						16.19	2.20	2.20	45.19	38.14	24.7	83.9	1.92	1.20	0.43	0.35	22.5	28.7
26-Oct-88	103	17.41	3.3	4.7						17.20	2.38	2.38	45.16	38.16	26.7	90.8	1.92	1.20	0.43	0.35	22.5	28.7
27-Oct-88	104	19.39	3.2	4.2						17.92	2.34	2.34	45.12	38.18	26.3	89.3	2.17	1.38	1.18	0.78	54.5	64.9
28-Oct-88	105	16.34	2.9	5.0						16.45	2.25	2.25	45.09	38.20	25.3	86.0	2.17	1.38	1.18	0.78	54.5	64.9
29-Oct-88	106									17.63	2.30	2.30	45.04	38.22	25.9	87.9	2.17	1.38	1.18	0.78	54.5	64.9
30-Oct-88	107	16.04	3.1	4.5						16.40	2.40	2.40	45.00	38.24	27.0	91.5	2.17	1.38	1.18	0.78	54.5	64.9
31-Oct-88	108									16.05	2.50	2.50	44.96	38.26	28.1	96.7	2.17	1.38	1.18	0.78	54.5	64.9
100% CTMP-N																						
01-Nov-88	109	17.44	2.2	4.7						16.12	2.55	2.55	44.93	38.28	26.7	97.0	3.09	1.33	1.75	0.80	50.7	45.3
02-Nov-88	110	16.70	2.0	5.2						17.03	2.50	2.50	44.89	38.31	26.2	96.6	3.09	1.33	1.75	0.80	50.7	45.3
03-Nov-88	111	17.55	2.7	4.6						16.86	2.50	2.50	44.85	38.33	26.2	96.8	2.86	1.47	1.20	0.73	41.9	49.6
04-Nov-88	112	15.92	2.9	5.1						19.00	2.50	2.50	44.81	38.35	26.2	96.9	2.86	1.47	1.20	0.73	41.9	49.6
05-Nov-88	113									19.55	2.50	2.50	44.77	38.37	26.2	96.9	2.86	1.47	1.20	0.73	41.9	49.6
06-Nov-88	114									20.14	2.48	2.48	44.73	38.39	26.2	96.2	2.86	1.47	1.20	0.73	41.9	49.6
07-Nov-88	115	16.18	3.4	5.0						19.49	2.45	2.45	44.69	38.41	27.7	94.1	2.86	1.47	1.20	0.73	41.9	49.6
08-Nov-88	116	14.34	3.1	5.7						17.91	2.45	2.45	44.66	38.43	27.7	94.2	2.86	1.47	1.20	0.73	41.9	49.6
09-Nov-88	117	17.25	2.6	4.7						16.51	2.45	2.45	44.62	38.45	27.7	94.2	3.04	1.55	1.06	0.25	63.8	16.4
10-Nov-88	118	16.06	3.2	5.1						16.25	2.45	2.45	44.58	38.47	27.7	94.3	3.04	1.55	1.06	0.25	63.8	16.4
11-Nov-88	119	17.14	3.2	4.8						14.81	2.60	2.60	44.54	38.49	29.4	100.1	3.04	1.55	1.06	0.25	63.8	16.4
12-Nov-88	120									14.10	2.60	2.60	44.50	38.51	29.4	100.1	2.49	1.36	1.09	0.50	43.5	36.5
13-Nov-88	121									14.82	2.45	2.45	44.46	38.53	29.4	100.1	2.49	1.36	1.09	0.50	43.5	36.5
14-Nov-88	122	15.16	2.9	5.4						14.58	2.50	2.50	44.43	38.55	27.8	94.4	1.64	1.03	0.44	0.28	23.9	25.7
15-Nov-88	123	16.25	3.5	5.0						12.95	2.60	2.60	44.39	38.57	28.3	96.4	1.64	1.03	0.44	0.28	23.9	25.7
16-Nov-88	124	13.97	3.9	5.8						12.89	2.60	2.60	44.35	38.59	29.5	100.3	2.03	1.07	0.70	0.33	34.5	30.8
17-Nov-88	125	15.84	3.3	5.2						13.72	2.60	2.60	44.31	38.61	29.5	100.3	2.03	1.07	0.70	0.33	34.5	30.8
18-Nov-88	126	16.63	3.7	4.9						12.72	2.60	2.60	44.27	38.63	29.6	100.4	2.60	1.42	1.40	0.33	48.5	23.2

Date	Day	Flow Rate Measured	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Perztor VSS		CONSTANT BIOMASS = 16.3 gVSS/L reactor					
			PA (hrs)	UASB (hrs)	Total (g/L-d)	Soluble (g/L-d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (%)	Soluble (%)	Total (%)	Soluble (%)
13-Nov-88	121								14.62	2.45	2.45	44.45	38.53	27.8	94.4						
14-Nov-88	122	15.16	2.9	5.4	30.09	17.70	23.9	25.7	14.58	2.50	2.50	44.43	38.55	28.3	96.4	1.84	1.08	0.44	0.28	23.9	25.7
15-Nov-88	123	16.25	3.6	5.0					12.95	2.60	2.60	44.39	38.57	29.6	100.3						
16-Nov-88	124	13.97	3.9	5.8	33.13	17.51	34.5	30.8	12.89	2.60	2.60	44.35	38.59	29.5	100.3	2.03	1.07	0.70	0.33	34.5	30.8
17-Nov-88	125	16.84	3.3	5.2					13.72	2.60	2.60	44.31	38.61	29.5	100.4						
18-Nov-88	126	16.63	3.7	4.9	47.20	23.18	48.5	23.2	12.72	2.60	2.60	44.27	38.63	29.5	100.4	2.89	1.42	1.40	0.33	48.5	23.2
19-Nov-88	127								13.28	2.50	2.50	44.23	38.65	28.4	96.6						
20-Nov-88	128								13.28	2.50	2.50	44.19	38.67	28.4	96.7						
21-Nov-88	129	14.65	3.0	5.5	45.29	24.06	45.2	33.9	13.67	2.50	2.50	44.16	38.70	28.5	96.7	2.77	1.47	1.34	0.50	45.2	33.9
22-Nov-88	130	14.30	3.7	5.7					14.15	2.55	2.55	44.12	38.72	29.0	96.7						
23-Nov-88	131	13.20	3.2	6.2	36.51	19.45	42.9	32.9	12.23	2.55	2.55	44.08	38.74	29.1	96.5	2.24	1.19	0.96	0.39	42.9	32.9
24-Nov-88	132								3.60	2.55	2.55	44.04	38.76	29.1	96.6						
25-Nov-88	133	12.43	3.9	6.6	29.79	20.54	42.6	45.0	10.62	2.66	2.66	44.00	38.78	29.7	100.8	1.63	1.26	0.78	0.57	42.6	45.0
26-Nov-88	134								11.90	2.66	2.66	43.96	38.80	30.2	102.8						
27-Nov-88	135								11.63	2.75	2.75	43.93	38.82	31.4	106.5						
28-Nov-88	136	16.97	2.0	4.8		26.05		26.3	9.78	2.80	2.80	43.89	38.84	32.0	106.7	1.72			0.45		26.3
29-Nov-88	137	16.72	3.5	4.9					11.41	2.80	2.80	43.85	38.85	32.0	106.8						
30-Nov-88	138	16.39	3.0	5.0	44.05	28.45	28.3	34.5	11.13	2.85	2.85	43.81	38.88	32.5	110.5	2.70	1.75	0.76	0.60	28.3	34.5

Table B5. Calculated period averages of the data
calculations for system PL2

Wastewater %	Flow Rate Measured Q (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.3 gVSS/L reactor					
		PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (%)	Soluble (%)	Total (%)	
		SRRVSLR		SRR		SRRVSLR														
CTMP-N -50	9.27	4.69	10.7	12.96	7.30	66.66	60.74	2.27	1.42	1.60	54.56	39.17	16.40	66.77	0.79	0.45	0.51	0.22	66.06	60.74
50%	16.84	2.77	5.19	20.56	11.89	47.30	36.30	6.22	1.81	2.00	60.46	37.00	20.00	66.02	1.26	0.73	0.60	0.28	47.30	36.30
70%	16.83	3.09	4.67	27.64	16.26	46.44	39.74	12.00	2.22	2.22	46.58	37.31	24.33	82.71	1.00	0.63	0.70	0.36	46.44	39.74
80%	16.46	3.62	4.97	37.64	16.16	56.66	46.10	13.03	2.22	2.22	46.62	37.92	24.71	84.01	2.31	0.99	1.36	0.46	56.66	46.10
90%	17.06	3.16	4.80	35.71	21.79	38.79	42.85	16.64	2.31	2.31	46.21	36.13	26.89	88.03	2.19	1.33	0.65	0.58	38.79	42.85
100%	16.73	3.10	6.23	42.16	22.92	42.23	33.78	14.63	2.67	2.67	44.37	36.68	29.12	99.02	2.66	1.40	1.12	0.47	42.23	33.78

APPENDIX C
PL3 EXPERIMENT RECORDS

Table C1. Daily operating data of system PL3

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N2 (%)	CH4 (%)	CO2 (%)						
START-UP CTMP-N															
15-Jul-88	0														
16-Jul-88	1				6.34		91	1	2				0.079		
17-Jul-88	2				7.83		80	10	4				0.079		
18-Jul-88	3	0	0	0	7.51		71	19	4				0.079		
19-Jul-88	4	125	20	0	7.20		67	31	6				0.079		
20-Jul-88	5	50	0	0	7.02		35	62	8				0.079		
21-Jul-88	6	35	0	0			20	68	8	1380			0.079		
22-Jul-88	7	45	0	0	6.70		15	70	9				0.079		
23-Jul-88	8	260	0	0	6.70		12	71	10	1810	0.268		0.079		
24-Jul-88	9	125	0	0	6.60		8	75	10				0.081		
25-Jul-88	10	90	0	0	6.85		7	76	11				0.082		
26-Jul-88	11	430	0	0			6	77	11				0.084		
27-Jul-88	12	280	0	0			6	77	11				0.086		
28-Jul-88	13	100	0	0			6	77	11	1060			0.088		
29-Jul-88	14	140	0	0			6	77	11				0.089		
30-Jul-88	15	120	0	0											
31-Jul-88	16	115	0	0											
01-Aug-88	17	0	0	0	6.95										
02-Aug-88	18	0	0	0											
50% CTMP-N															
03-Aug-88	19	0	0	0	7.16		10	76	8	1150	0.237		0.091		
04-Aug-88	20												0.103		
05-Aug-88	21									1230	1080		0.114		
06-Aug-88	22												0.128		
07-Aug-88	23	65	0	0	6.82	1300	7	79	8				0.137		
08-Aug-88	24	0	0	0			6	78	10				0.149		
09-Aug-88	25	90	0	0			6	78	10				0.160		
10-Aug-88	26	110	0	0			6	79	10		0.206		0.172		
11-Aug-88	27	75	0	0	6.93	1060	6	79	10				0.171		
12-Aug-88	28	120	0	0									0.170		
13-Aug-88	29												0.169		
14-Aug-88	30	0	0	0			7	79	8				0.168		
15-Aug-88	31	0	0	0									0.167		
16-Aug-88	32	185	0	0									0.166		
17-Aug-88	33	35	0	0						1000			0.166		
18-Aug-88	34	0	0	0	6.90	1150	6	78	10				0.165		
19-Aug-88	35	55	0	0						1100			0.164		
20-Aug-88	36												0.163		
21-Aug-88	37						3	79	12				0.162		
22-Aug-88	38	110	0	0	7.01	1350							0.161		
23-Aug-88	39	40	0	0									0.160		
24-Aug-88	40	65	0	0						1310	1050	0.100	0.160		

		FEED										PREACIDIFICATION TANK														
Date	Day	Time (hrs)	Elapsed Time (hrs)	Biogas Meter Reading	Effluent Volume (L)	Room T (C)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	
							Ace (mg/L)	Pro (mg/L)	But (mg/L)								Ace (mg/L)	Pro (mg/L)	But (mg/L)							
25-Aug-88	41	9	50	24.87	7911	16.13	34.5	495	50	0	8.48	590			2.10	310	0	0	0	6.78	840					
26-Aug-88	42	10	5	24.25	7989	15.20	35.0	580	80	0					3.30	375	20	0								
27-Aug-88	43	12	10	26.08	8063	16.02	34.0								2.33											
28-Aug-88	44	11	50	23.87	8128	15.78	36.3								2.10											
29-Aug-88	45	10	10	22.33	8198	15.78	33.5	640	80	0	6.65	860	2230	0.211	1.90	420	0	0	0	7.05	1110	1770	1830			
30-Aug-88	46	9	33	23.38	8268	15.67	34.5	630	65	0				0.224	1.90	385	0	0								
31-Aug-88	47	9	12	23.85	8337	15.73	34.2	610	80	0			1990	0.238	2.30	360	0	0				1800	1560	0.273	0.217	
01-Sep-88	48	12	15	27.05	8415	17.65	34.0	610	70	0				0.254	2.30	375	0	0								
02-Sep-88	49	10	11	21.93	8461	14.61	35.5	410	45	0			1420	0.271	2.30	240	0	0				1380	1090			
03-Sep-88	50	12	33	26.37	8549	17.25	33.8							0.289	2.80											
04-Sep-88	51	13	22	24.82	8611									0.308	3.10											
05-Sep-88	52	13	21	23.98	8665									0.324	3.15											
06-Sep-88	53	9	54	20.55	8690	13.80	33.8	235	30	0	7.39	730	1120	0.341	2.05	155	0	0			700	1320	1060	0.237	0.188	
07-Sep-88	54	11	30	25.60	8732	16.13	33.8	170	0	0				0.327	2.20	215	0	0								
08-Sep-88	55	14	40	27.17	8787	17.77	35.0	220	0	0			1690	0.313	2.30	195	0	0				2050	1570			
09-Sep-88	56	10	30	18.83	8837	13.80	35.0	380	0	0				0.299	2.50	260	0	0								
10-Sep-88	57	13	25	26.92	8886									0.285	2.40											
11-Sep-88	58	13	40	24.25	8924	15.14	34.5	135	0	0	6.70	500	560	0.258	2.30	110	0	0			800	1090	930			
12-Sep-88	59	11	25	21.75	8950			175	15	0				0.244	2.33	140	0	0								
13-Sep-88	60	10	5	22.67	8986	16.78	36.0																			
14-Sep-88	61	9	40	23.58	9050	14.61	33.0	190	50	0			1880	0.230	0.30	80	0	0						2.852	2.254	
15-Sep-88	62	9	0	23.33	9134	17.30	33.0	790	86	0				0.277	1.30	560	0	0								
16-Sep-88	63	11	10	26.17	9244	18.73	33.0	930	100	0	6.38		4140	0.325	1.10	655	0	0			6.98					
17-Sep-88	64	9	35	22.42	9337	14.33	33.2							0.372	1.63											
18-Sep-88	65	10	10	24.58	9455	17.07	33.2							0.419	1.36											
19-Sep-88	66	10	47	24.62	9585	17.77	34.4	815	125	0			2680	0.466	1.70	490	0	0				2900	1960			
20-Sep-88	67	10	9	23.37	9760	17.19	36.0	900	140	0	6.46	860		0.514	1.20	520	0	0			1230					
21-Sep-88	68	9	44	23.58	9858	17.64	35.5	445	100	0				0.561	1.86	340	0	0								
22-Sep-88	69	10	5	24.35	9973	17.77	33.5	590	0	0	7.03			0.504	1.80	490	0	0								
23-Sep-88	70	10	30	24.42	85	18.01	33.3	335	0	0			2050	0.446	1.93	295	0	0				2200	1460			
24-Sep-88	71	11	30	25.00	148									0.389	1.92											
25-Sep-88	72	11	5	23.58	223	31.0								0.332	1.40											
26-Sep-88	73	9	25	22.33	305			615	45	0			2900	0.275	1.50	395	0	0				2680	2000			
27-Sep-88	74	8	40	23.25	403	16.66	32.0	740	55	0	6.63	900		0.217	1.80	465	0	0			1200					
28-Sep-88	75	6	50	24.17	509	15.78	32.0	780	60	0			3070	0.172	1.90	485	0	0				3040	2150	0.488	0.418	
29-Sep-88	76	9	5	24.25	610	17.54	35.0	715	95	0	6.75			0.165	1.45	440	0	0								
30-Sep-88	77	10	0	24.92	719	16.80	33.0	645	0	0			2700	0.189	2.10	470	0	0				2430	1740			
01-Oct-88	78	11	35	25.68	835									0.174	2.40											
02-Oct-88	79	12	40	25.08	950									0.178	2.14											
03-Oct-88	80	11	40	23.00	1055	15.55	33.2	870	145	0	6.08	980		0.183	2.15	640	0	0				2500	1880			
04-Oct-88	81	10	40	23.00	1158	15.32	35.0	875	155	0				0.187	2.20	615	0	0								
05-Oct-88	82	11	12	24.63	1265	16.80	33.6	910	110	0			3250	0.192	2.10	530	0	0				2430	1530	0.713	0.601	

70% CTMP-N

Date	Day	UASB REACTOR										EFFLUENT			
		VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)						
26-Aug-88	41	45	0	0	7.00	1040	4	79	11				0.160		
26-Aug-88	42	55	0	0									0.161		
27-Aug-88	43												0.161		
28-Aug-88	44	86	0	0			3	80	11	1230	970		0.162		
29-Aug-88	45	35	0	0	7.22	7310	3	80	11	1260	1010	0.163	0.163		
30-Aug-88	46	55	0	0									0.157		
31-Aug-88	47	50	0	0						1190	1010		0.151		
01-Sep-88	48	20	0	0									0.145		
02-Sep-88	49												0.138		
03-Sep-88	50												0.132		
04-Sep-88	51												0.128		
05-Sep-88	52	0	0	0	7.05	860	5	83	7	940	740	0.162	0.122		
06-Sep-88	53	0	0	0									0.118		
07-Sep-88	54	0	0	0						1580	1490		0.113		
08-Sep-88	55	20	0	0			4	78	12				0.109		
09-Sep-88	56	30	0	0									0.105		
10-Sep-88	57												0.100		
11-Sep-88	58									840	710		0.098		
12-Sep-88	59	0	0	0	7.04	750	6	80	6						
13-Sep-88	60	10	0	0											
70% CTMP-N															
14-Sep-88	61	20	0	0								0.257	0.082		
15-Sep-88	62	185	0	0									0.112		
16-Sep-88	63	190	0	0	7.23		3	76	16	1930	1640		0.133		
17-Sep-88	64												0.153		
18-Sep-88	65	70	0	0									0.173		
19-Sep-88	66												0.193		
20-Sep-88	67	90	0	0	7.22	1460	2	80	10	1870	1610		0.214		
21-Sep-88	68	45	0	0			32	52	10	1750	1350	0.281	0.234		
22-Sep-88	69	95	0	0			6	78	11				0.230		
23-Sep-88	70	40	0	0	7.00		3	76	16	1630	1300		0.227		
24-Sep-88	71												0.223		
25-Sep-88	72												0.220		
26-Sep-88	73	40	0	0	7.02	1400	4	77	13	2000	1310		0.216		
27-Sep-88	74	85	0	0									0.213		
28-Sep-88	75	60	0	0						1720	1580	0.220	0.209		
29-Sep-88	76	40	0	0	7.01		3	79	12	1460	1260		0.204		
30-Sep-88	77	50	0	0									0.199		
01-Oct-88	78												0.183		
02-Oct-88	79												0.188		
03-Oct-88	80	100	0	0	7.08	1630	2	76	14	1460	1290		0.163		
04-Oct-88	81	56	0	0									0.177		
05-Oct-88	82	60	0	0						2290	1630		0.172		

FEED										PREACIDIFICATION TANK																	
Date	Day	Time (hrs)	Time (min)	Elapsed Time (hrs)	Biogas Meter Reading	Effluent Volume (L)	Room T (C)	VFA (mg/L)			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA (mg/L)			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	
								Ace	Pro	But								Ace	Pro	But							
06-Oct-88	83	11	35	24.38	1368	15.90	31.0	560	120	0							2.15	615	45	0							
07-Oct-88	84	10	55	23.33	1463	15.67	35.0	825	130	0			4440	2560			2.00	440	30	0			3720	2780			
08-Oct-88	85	9	10	22.25	1581		34.0										2.00										
09-Oct-88	86	13	25	28.25	1685		33.0										1.80										
10-Oct-88	87	13	35	24.17	1802		33.0										2.00										
90% CTMP-N																											
11-Oct-88	88	10	40	21.08	1903	14.44	33.0	960	165	0	6.43						1.70	565	50	0	6.84	1250				0.242	
12-Oct-88	89	10	40	24.00	1904	15.26	34.0	820	130	0			3080	2580	1.683		2.69	435	40	0			2430	1400			
13-Oct-88	90	10	40	24.00	2092	16.49	34.0	920	145	0	6.15		5110	2280			2.96	0.169	0.172	0.172	0.54		2460	1510			
14-Oct-88	91	10	40	24.00	2168	17.10	35.0	580	95	0							2.32	325	25	0			2460	1510			
15-Oct-88	92	8	36	21.93	2242		34.8										2.60	0.163	0.163	0.163			2170	1550			
16-Oct-88	93	15	0	30.40	2353		34.5	680	100	0	6.51	800	2430	2150			2.50	0.160	0.160	0.160	6.91	1100					
17-Oct-88	94	10	20	19.33	2421	12.86	34.5										4.70	375	30	0							
90% CTMP-N																											
18-Oct-88	95	11	2	24.70	2426	14.13	34.9	705	80	0							2.50	355	30	0							
19-Oct-88	96	10	45	23.72	2377	16.00	34.9	630	35	0					0.191		2.10	350	25	0					0.134	0.117	
20-Oct-88	97	10	50	24.08	2448	16.49	34.5	690	40	0	6.42						2.00	410	30	0	6.72						
21-Oct-88	98	9	35	22.75	2531	15.55	34.5	860	130	0			6150	2840			2.30	455	40	0			2840	2090			
22-Oct-88	99	11	55	26.33	2647		30.0										2.40	0.142	0.142	0.142							
23-Oct-88	100	13	35	25.67	2767	16.49	35.0	780	110	0			3940	3530			2.35	0.139	0.139	0.139			2820	2280			
24-Oct-88	101	13	55	24.33	2879	16.49	35.0	825	85	0	6.11	1200					2.00	0.136	0.136	0.136							
25-Oct-88	102	9	35	19.67	2969	13.21	35.5	1090	180	0			5070	3830	0.997		2.10	427	100	0	6.77	2150					
26-Oct-88	103	10	35	25.00	3087	16.95	35.0	870	130	0							3.04	20	170	0			4880	4770	0.181		
27-Oct-88	104	11	20	24.75	3192	16.84	35.5	1105	150	0							3.56	530	60	0							
28-Oct-88	105	9	55	22.58	3309	15.32	36.0	1105	150	0			4270	3850			2.37	895	75	0			3650	2630			
29-Oct-88	106	13	20	27.42	3443		34.5										2.28	0.122	0.122	0.122			3280	3220			
30-Oct-88	107	10	15	20.92	3539	20.56	34.0	780	95	0			4220	3990			1.50	0.119	0.119	0.119							
31-Oct-88	108	15	15	29.00	3680		34.0										1.90	0.116	0.116	0.116							
100% CTMP-N																											
01-Nov-88	109	9	58	18.72	3765	12.98	34.8	790	100	0	5.98	1340	4700	4440	0.123		2.15	890	90	0	6.70	1710				0.238	
02-Nov-88	110	10	35	24.52	3801	16.49	34.5	1270	170	0							2.55	890	100	0			3710	3140			
03-Nov-88	111	10	50	24.25	4030	16.02	34.0	635	110	0							2.30	920	100	0							
04-Nov-88	112	9	55	23.08	4150	15.55	34.2	1270	140	0	6.96		4910	4940			2.20	940	90	0			5190	3810			
05-Nov-88	113	10	55	25.00	4178		35.2										2.10	0.152	0.152	0.152							
06-Nov-88	114	11	45	24.83	4184	14.32	36.0	1300	225	0			4910	4720			2.15	0.165	0.165	0.165			4030	3810			
07-Nov-88	115	9	40	21.92	4242	14.44	36.0	1345	255	0	5.82	1300					2.45	0.179	0.179	0.179			4030	3810			
08-Nov-88	116	9	20	23.67	4308	14.44	35.0	1155	215	0			5160	4650	0.284		2.30	1465	165	0			4030	3310		0.544	
09-Nov-88	117	8	35	23.25	4366	10.11	35.0	1230	215	0							2.28	865	70	0			4030	3310			
10-Nov-88	118	9	35	25.00	4439	12.39	34.5	1230	215	0							2.18	935	100	0							
11-Nov-88	119	11	10	25.58	4508	13.15	33.0	1120	170	0							2.00	915	85	0			4750	3700			
12-Nov-88	120	9	15	22.08	4565		33.2										1.56	0.230	0.230	0.230							

		UASB REACTOR										EFFLUENT				
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)			
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)							
08-Oct-88	83	70	0	0			2	76	14		1470	1060	0.208	0.167		
07-Oct-88	84	45	0	0										0.170		
08-Oct-88	85													0.173		
09-Oct-88	86													0.177		
10-Oct-88	87													0.180		
80% CTMP-N																
11-Oct-88	88	76	0	0	7.09	1800	3	79	12	2060	1430	0.244	0.183			
12-Oct-88	89	55	0	0									0.188			
13-Oct-88	90	40	0	0	7.05		3	78	13	1360	970		0.331			
14-Oct-88	91	45	0	0									0.475			
15-Oct-88	92												0.620			
16-Oct-88	93												0.764			
17-Oct-88	94	45	0	0	7.14	1200	43	44	6	1320	960		0.909			
90% CTMP-N																
18-Oct-88	95	40	0	0			3	76	13	1270	960	1.507	1.063			
19-Oct-88	96	45	0	0									1.198			
20-Oct-88	97	55	0	0	6.95		2	80	12	1680	1873		1.062			
21-Oct-88	98	65	0	0									0.905			
22-Oct-88	99												0.759			
23-Oct-88	100												0.813			
24-Oct-88	101	80	0	0			2	75	18	1710	1400		0.487			
25-Oct-88	102	95	0	0	6.92	1600	2	76	16	4550	1760	0.222	0.320			
26-Oct-88	103	90	0	0									0.174			
27-Oct-88	104	90	0	0									0.187			
28-Oct-88	105	155	0	0									0.201			
29-Oct-88	106												0.214			
30-Oct-88	107	240	15	0			3	76	16	2350	1850		0.227			
31-Oct-88	108									2490	2120		0.240			
100% CTMP-N																
01-Nov-88	109	365	20	0	7.02	1980				2660	2170	0.308	0.254			
02-Nov-88	110	325	30	0									0.287			
03-Nov-88	111	385	20	0			2	75	16	2500	2680		0.262			
04-Nov-88	112	415	25	0	6.99								0.258			
05-Nov-88	113												0.251			
06-Nov-88	114												0.245			
07-Nov-88	115	655	0	0	6.79	2000	3	73	17	3220	2860		0.240			
08-Nov-88	116	540	0	0									0.234			
09-Nov-88	117	270	0	0									0.229			
10-Nov-88	118	485	0	0									0.228			
11-Nov-88	119	495	0	0									0.223			
12-Nov-88	120	560	0	0						3060	2300		0.220			

FEED										PREACIDIFICATION TANK																
Date	Day	Time (hrs)	Time (min)	Elapsed Time (hrs)	Biogas Meter Reading	Effluent Volume (L)	Room T (C)	VFA Ace (mg/L)	VFA Pro (mg/L)	VFA But (mg/L)	pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA Ace (mg/L)	VFA Pro (mg/L)	VFA But (mg/L)	pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)
13-Nov-88	121	11	40	28.42	4844	16.43	33.8	1065	190	0	6.89	1180	3530	3530	0.241	0.241	2.34	835	85	0	6.76	1500	2950	2950	0.213	0.184
14-Nov-88	122	11	43	24.05	4718	17.13	33.0	1116	205	0	6.89	1180	3530	3530	0.250	0.250	2.30	850	80	0	6.76	1500	2950	2950	0.213	0.184
15-Nov-88	123	15	12	27.48	4803	14.85	33.8	960	105	0	6.89	1180	3530	3240	0.305	0.287	2.18	820	60	0	6.83	1550	3350	2970	0.213	0.184
16-Nov-88	124	9	50	18.83	4858	1.70	33.4	975	95	0	6.89	1180	3530	3240	0.305	0.236	2.10	840	0	0	6.83	1550	3350	2970	0.213	0.184
17-Nov-88	125	12	18	28.47	48822	15.78	33.5	950	135	0	6.89	1180	3530	3070	0.206	0.206	2.60	735	60	0	6.83	1550	2970	2970	0.213	0.184
18-Nov-88	128	11	45	23.45	48891	15.78	33.5	950	135	0	6.89	1180	3530	3070	0.206	0.206	2.60	735	60	0	6.83	1550	2970	2970	0.213	0.184
19-Nov-88	127	14	56	27.18	48781	15.78	33.5	950	135	0	6.89	1180	3530	3070	0.206	0.206	2.60	735	60	0	6.83	1550	2970	2970	0.213	0.184
20-Nov-88	128	13	11	22.25	48864	15.78	33.5	950	135	0	6.89	1180	3530	3070	0.206	0.206	2.60	735	60	0	6.83	1550	2970	2970	0.213	0.184
21-Nov-88	129	11	10	21.98	48949	15.08	33.8	820	25	0	6.3	1110	4600	4110	0.145	0.145	2.70	730	45	0	6.3	1550	3330	2930	0.213	0.184
22-Nov-88	130	10	26	23.25	49041	15.90	33.0	1000	115	0	6.3	1110	4600	4110	0.145	0.145	2.70	730	45	0	6.3	1550	3330	2930	0.213	0.184
23-Nov-88	131	10	30	24.08	49135	16.95	33.8	1050	145	0	6.3	1110	4600	3960	0.086	0.086	2.56	795	60	0	6.3	1550	3300	2780	0.252	0.215
24-Nov-88	132	11	0	24.50	49229	16.95	33.8	1030	160	0	6.24	1110	4600	3960	0.086	0.086	2.30	795	60	0	6.24	1550	3300	2780	0.252	0.215
25-Nov-88	133	11	45	24.75	49328	17.07	33.0	1050	145	0	6.24	1110	4050	3700	0.106	0.106	2.40	775	65	0	6.83	1550	3230	3150	0.252	0.215
26-Nov-88	134	11	22	23.82	49422	14.81	32.0	1050	145	0	6.24	1110	4050	3700	0.106	0.133	2.00	775	65	0	6.83	1550	3230	3150	0.252	0.215
27-Nov-88	135	14	30	27.13	49442	0	33.5	1095	190	0	6.42	1000	10040	3900	0.159	0.159	2.10	810	70	0	6.80	1400	4520	3650	0.507	0.454
28-Nov-88	136	9	56	19.43	49506	15.08	33.2	1070	170	0	6.42	1000	10040	3900	0.188	0.188	2.30	810	70	0	6.80	1400	4520	3650	0.507	0.454
29-Nov-88	137	10	10	24.23	49588	16.84	30.0	1040	165	0	6.42	1000	10040	3900	0.212	0.212	2.05	780	70	0	6.80	1400	4520	3650	0.507	0.454
30-Nov-88	138	9	5	22.92	49685	14.5	32.8	1040	165	0	6.42	1000	10040	3900	0.239	0.239	1.90	740	65	0	6.80	1400	4520	3650	0.507	0.454
01-Dec-88	139	13	35	28.50	49780	17.89	33.8	1075	175	0	6.42	1000	3990	3730	0.239	0.239	0.25	795	75	0	6.80	1400	3840	2770	0.507	0.454
02-Dec-88	140	11	50	22.25	49865	15.08	33.0	1090	160	0	6.42	1000	4840	3280	0.239	0.239	1.80	805	80	0	6.80	1400	4440	2920	0.507	0.454

		UASB REACTOR										EFFLUENT			
Date	Day	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)		
		Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)						
13-Nov-88	121	180	0	0									0.217		
14-Nov-88	122	505	0	0						2510	2220		0.214		
15-Nov-88	123	465	0	0	7.04	1740							0.211		
16-Nov-88	124	525	0	0						2220	2070	0.231	0.208		
17-Nov-88	125	50	0	0									0.211		
18-Nov-88	126	405	0	0						1950	1690		0.215		
19-Nov-88	127												0.218		
20-Nov-88	128												0.222		
21-Nov-88	129	345	0	0	7.08					3000	2100		0.225		
22-Nov-88	130	400	0	0									0.229		
23-Nov-88	131	370	0	0						2200	2010	0.220	0.232		
24-Nov-88	132	396	0	0	7.09	1700							0.226		
25-Nov-88	133	375	0	0						2420	2130		0.220		
26-Nov-88	134												0.214		
27-Nov-88	135												0.208		
28-Nov-88	136	430	0	0	6.88	1800				2880	2650		0.202		
29-Nov-88	137	400	0	0									0.196		
30-Nov-88	138	330	0	0						2390	1920	0.190	0.190		
01-Dec-88	139	430	0	0									0.180		
02-Dec-88	140	490	0	0	6.77					2780	2310		0.190		

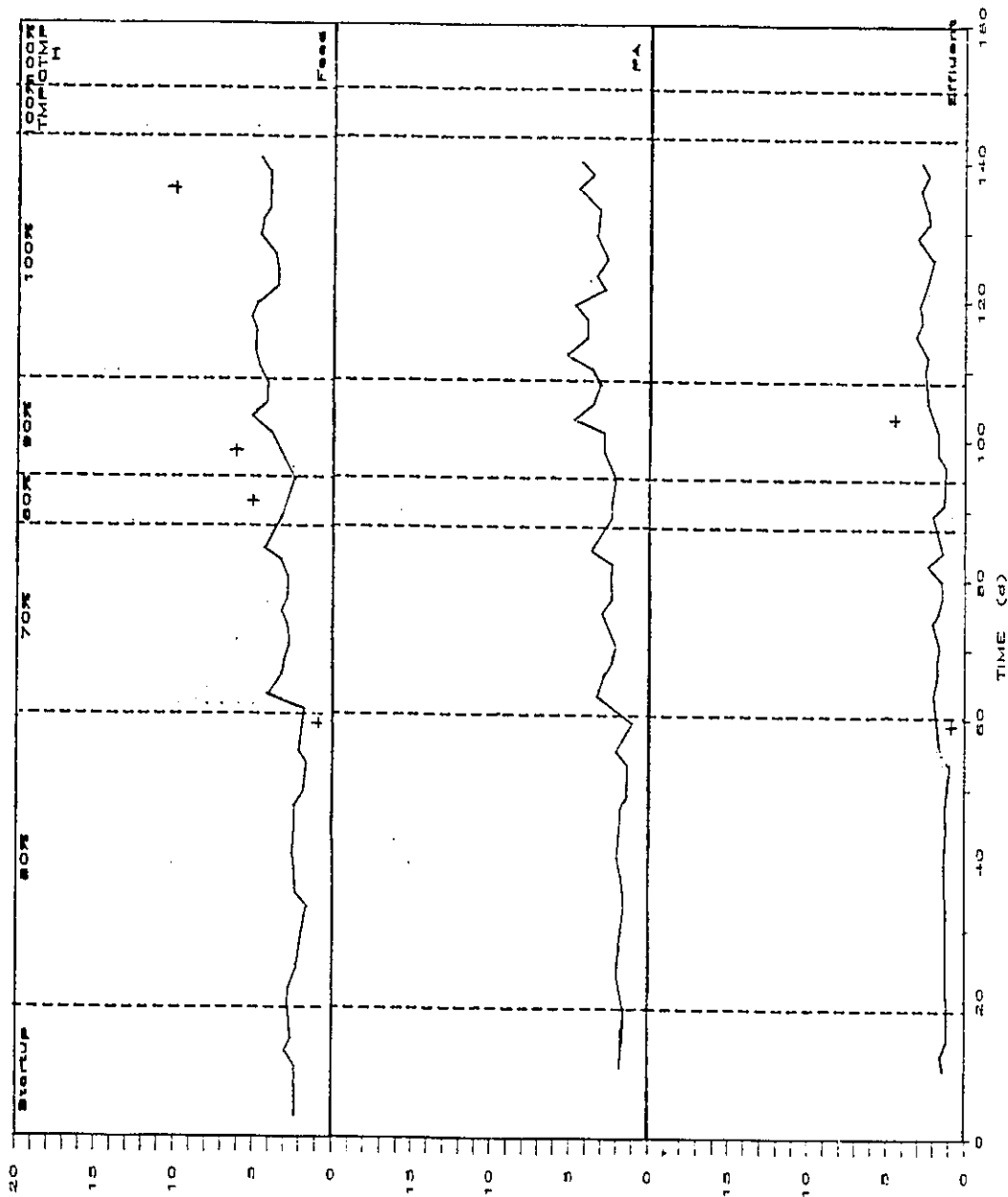
Table C2. Daily operating record comments of system PL3

DATE	PUMP SETTING	COMMENTS
15-Jul-88		PL stable. PA height stable.
16-Jul-88		PL @ 3.5L, opened to release, tilted to prevent blocking. PA @ 2.55L, removed .4L, tightened Lt hd clamp. ATIR eff added to overhead tanks.
17-Jul-88		PL @ 3.6L, opened to release. Channelling. PA @ 2.75L, removed .65L, Lt hd clamp loosened. 6L from floor feed tank transferred to overhead tank.
18-Jul-88	3:25%	PL @ 3.6L, opened to release. Overhead tanks emptied, CTMP F feeding started. Pump decreased @ 09:50.
19-Jul-88		PL @ 3.5L, gas sampled then released. Channeling (gap in bed). Recycle line walked.
20-Jul-88		PL bed well mixed. Feed VFA unusually low (check).
21-Jul-88	3:50%	Feed recycle pump off since timer not working, restarted @ 09:45. Pump increased since VFA ok.
22-Jul-88		Feed tank empty 07:30. Recycle and feed lines walked. Rotated Lt hd tubing. PA increasing, Lt hd clamp loosened @ 15:00.
23-Jul-88		PA removed 1.34L @ 16:30.
24-Jul-88		Feed tank empty @ 19:30.
25-Jul-88	3:68%	Timer not working, replaced.
26-Jul-88		Feed line Y connector installed.
27-Jul-88		Timer on always, reset. PA acids higher than Feed->wall growth acidogens? Timer replaced.
28-Jul-88		50L feed tank installed on bench
29-Jul-88	3:89%	
30-Jul-88	3:80%	
31-Jul-88		
01-Aug-88	3:97%	PA white foam removed from top.
02-Aug-88	3:100	Recycle pump tubing changed.
03-Aug-88		PL white specks in bed. PA dark brown, growth on walls. Pump off, restarted 09:55. Feed acids high.
04-Aug-88		Pump clamp tightened.
05-Aug-88	3:86%	SS generated in PA enters PL seen in E.
06-Aug-88	3:90%	
07-Aug-88	2:58%	
08-Aug-88	2:59%	PA recycle not as high as PA1 & 2. Lower rate->advantage for wall growth. Must change pumps 1 & 2, OR 3. BM in off. Squeezed PA to PL lines (BM in lines). Pump decreased @ 14:00.
09-Aug-88	2:50%	PL some scum on top of biorings. Lines walked. Pump increased @ 10:45.

10-Aug-88	2:54%	VFA good. Pump increased @ 11:35.
11-Aug-88	2:55%	PL some growth on walls.
12-Aug-88		PA didn't increase as much as previous day. Pump increased @ 10:50.
13-Aug-88	2:57%	Feed ran out almost. Tank cleaned.
14-Aug-88		PA to 2.3L because of no feed.
15-Aug-88		Pump off, restarted @ 10:00.
16-Aug-88		PL changed feed loop into it.
17-Aug-88		PL @ 2.3L dripping->loss 100ml.
18-Aug-88		off cleaned.
19-Aug-88		
20-Aug-88	2:76%	E overflow.
21-Aug-88	2:57%	Feed line empty, pump speed higher than recorded, dec.
22-Aug-88	2:50%	PL and PA growth on walls. F foam in tank from mixing.
23-Aug-88		
24-Aug-88		
25-Aug-88		F line and recycle out of liquid, reconnect F line to recycle and submerge.
26-Aug-88		
27-Aug-88		
28-Aug-88	2:51%	Wiped slime from feed tank walls. FILTRATE IS NOT CLEAR FROM NOW UNTIL 19.9.88
29-Aug-88		PL bright reddish brown growth on top (noticed 15.8.88). Feed tank cleaned.
30-Aug-88		
31-Aug-88		off cleaned. Sampled for settling test.
01-Sep-88		
02-Sep-88		Gas low, feed acids low.
03-Sep-88		Bottom of feed not used, contains fibers, dk brown, cleaned.
04-Sep-88		White growth in feed line tubing.
05-Sep-88		
06-Sep-88		Empty, clean feed line and tank to prevent feed decay.
07-Sep-88		Acids low despite cleaning.
08-Sep-88	2:54%	
09-Sep-88		
10-Sep-88		
11-Sep-88		
12-Sep-88		INCREASE WW CONCENTRATION: 40% DILUTION
13-Sep-88		PL brown scum on top. WW poorly filtered, fibers on bucket bottom.
14-Sep-88		PA emptied, acids & cods after topping, solids high. BGAS production up. INCREASE WW CONCENTRATION: 30% DILUTION
15-Sep-88	2:50%	
16-Sep-88	2:48%	
17-Sep-88	2:55%	
18-Sep-88	2:53%	
19-Sep-88		CLEAR FILTRATE (vacuum grease under paper edge). PL to PA line choking, loosened.
20-Sep-88		BGAS unusually high, air must have entered PL but PA vol not below PA to PL port.

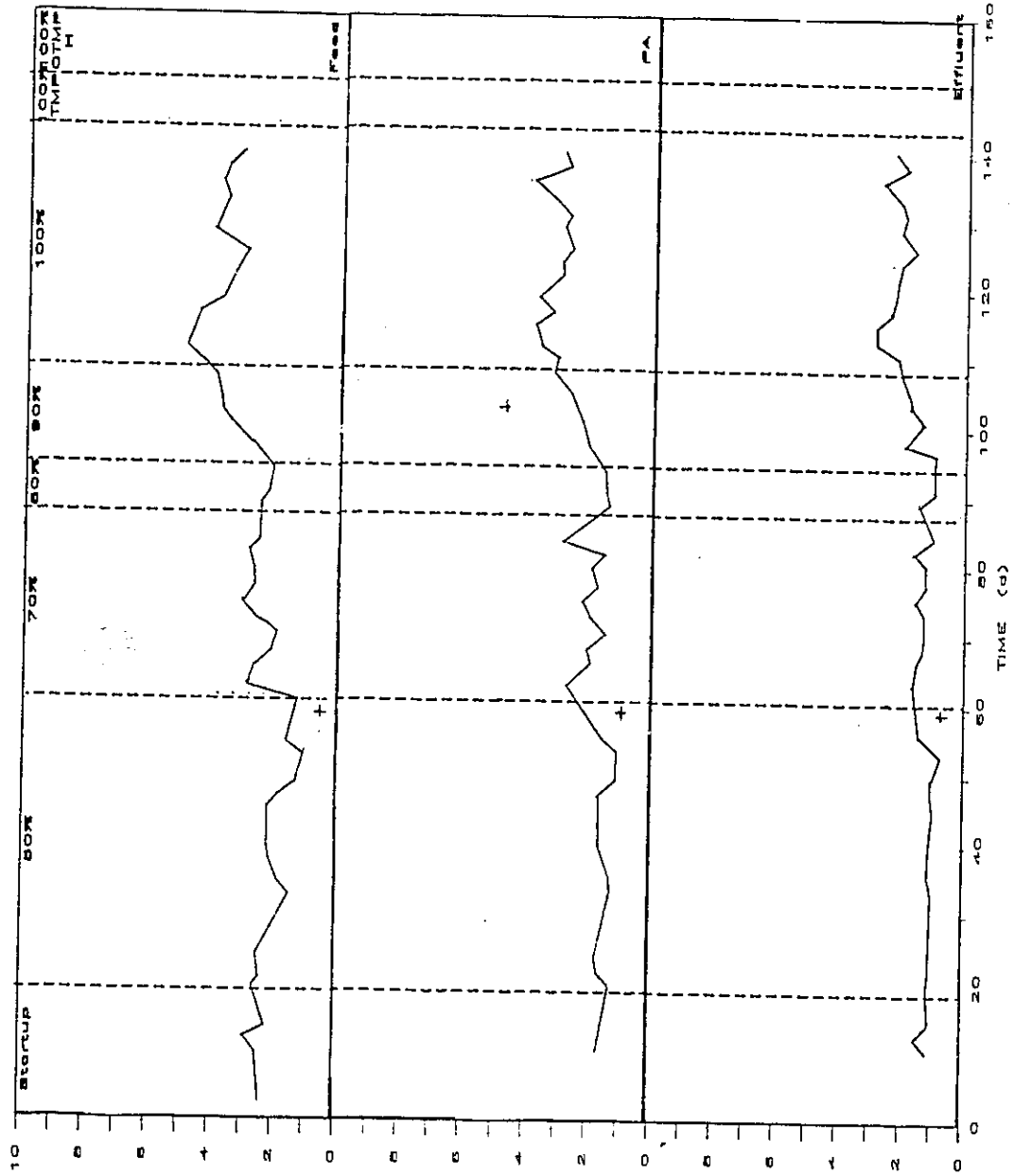
21-Sep-88		Cleaned feed tank and line, lots of growth. Feed ran out, air->PA.
22-Sep-88	2:54%	
23-Sep-88	2:50%	Walked all tubes, changed septa.
24-Sep-88		
25-Sep-88		
26-Sep-88	2:54%	H lines changed, other walked.
27-Sep-88	2:50%	STARTED USING CLEAN FILTRATE HERE.
28-Sep-88	2:53%	Crushed BM PA to PL line. Recycle off 2 hrs ERROR. PA very dark brown, thick scum on walls.
29-Sep-88	2:50%	Tank cleaned. H lines walked. FEED used to top PA since E discarded.
30-Sep-88	2:51%	Tried to add feed before checking tank, 5 L WW added as day progressed since all wouldn't fit in tank.
01-Oct-88		
02-Oct-88		
03-Oct-88		Bed is in motion. Removed 80 mL from bed for tests. Walked all tubing. Cleaned off.
04-Oct-88		Rich black wall growth in PA.
05-Oct-88		Bed top uneven.
06-Oct-88		Walked H tubing.
07-Oct-88		
08-Oct-88		
09-Oct-88		
10-Oct-88		
11-Oct-88		Lots of biomass in eff. H lines changed, rec. walked. INCREASE WW CONCENTRATION: 20% DILUTION
12-Oct-88	3:55%	
13-Oct-88		3 H lines changed, 1 walked. Recycle had been off? Turned on at 14:30.
14-Oct-88		Less gas than earlier.
15-Oct-88		Less gas than earlier. Cleaned off. Bed volume seems to decrease gradually. Bed mixed, granules enter recycle -> leave reactor and system.
16-Oct-88		
17-Oct-88		Liquid level high in PL, released. Lot of bed movement. All lines walked.
18-Oct-88		Gas production low? WTGM broken->replaced(94ml/tip). INCREASE WW CONCENTRATION: 10% DILUTION
19-Oct-88		
20-Oct-88		Bgas production still low. H lines walked.
21-Oct-88		
22-Oct-88		
23-Oct-88		
24-Oct-88		
25-Oct-88		All lines walked. PA3 black, alk high.
26-Oct-88		Unusual PA3 acids. Recirc pump for PA not working, replugged->OK.
27-Oct-88		
28-Oct-88		PL bed has 1.2L granules and .05L crushed BM on top. H lines walked.
29-Oct-88		A lot of air into PL->moved T connector.
30-Oct-88		

31-Oct-88		Walked all lines. INCREASE WW CONCENTRATION: 0% DILUTION PA->PL tubing loose in pump, tightened.
01-Nov-88		200ml added to WTGM.
02-Nov-88		
03-Nov-88		o/f cleaned. H lines moved. F line cleaned.
04-Nov-88		ERROR: FEED PUMP OFF VFA UP.
05-Nov-88		Changed gas sampling septum.
06-Nov-88		FEED PUMP ON @ 11:25
07-Nov-88	2:53%	PL VFA high. BGAS low. ERROR: pump should have been turned down.
08-Nov-88	2:31%	08:50 PL recycle not working, line pulled, pump->10 ->5.1 ok but watch. BGAS low. PL acids high, decrease Q.
09-Nov-88	2:38%	Q increase.
10-Nov-88		PL VFA high. H lines moved.
11-Nov-88	2:45%	PL VFA stable, Q increased.
12-Nov-88		VFA better.
13-Nov-88	2:49%	VFA low, Q increased.
14-Nov-88		VFA high leave as is. H lines walked.
15-Nov-88	2:59%	o/f cleaned. Growth in feed bucket. Wiped scum F tank side. F line cleaned. WTGM changed @ 16:16.
16-Nov-88	2:51%	Gas low, changed spl port septum. Changed F line septum. ERROR: FEED PUMP OFF @ 09:50
17-Nov-88		PUMP ON @ 11:40
18-Nov-88		
19-Nov-88		
20-Nov-88		
21-Nov-88		
22-Nov-88		All lines walked.
23-Nov-88		
24-Nov-88		Walked lines. Water in gasoline. PL liquid mark @ 4.5L.
25-Nov-88		
26-Nov-88	2:57%	o/f cleaned. Small SS on bed top. ERROR: FEED PUMP OFF @ 11:20
27-Nov-88		PUMP ON @ 14:30
28-Nov-88	2:50%	All lines walked, 1 changed.
29-Nov-88	2:49%	
30-Nov-88	2:53%	Removed BM: 60ml @ 0.5L, 60ml @ 1.0L for activity tests.
01-Dec-88		Crushed BM on top of bed. PA tank empty, F line out of liquid, E volume high, therefore couldn't have happened long ago.
02-Dec-88		All lines walked. Crushed BM on top of bed. END OF 100% CTMP NORMAL RUN.



(2/6) 74101 030

Figure C1a COD_T versus operating time



(1/8) FERTOS COG

Figure C1b COD_g versus operating time

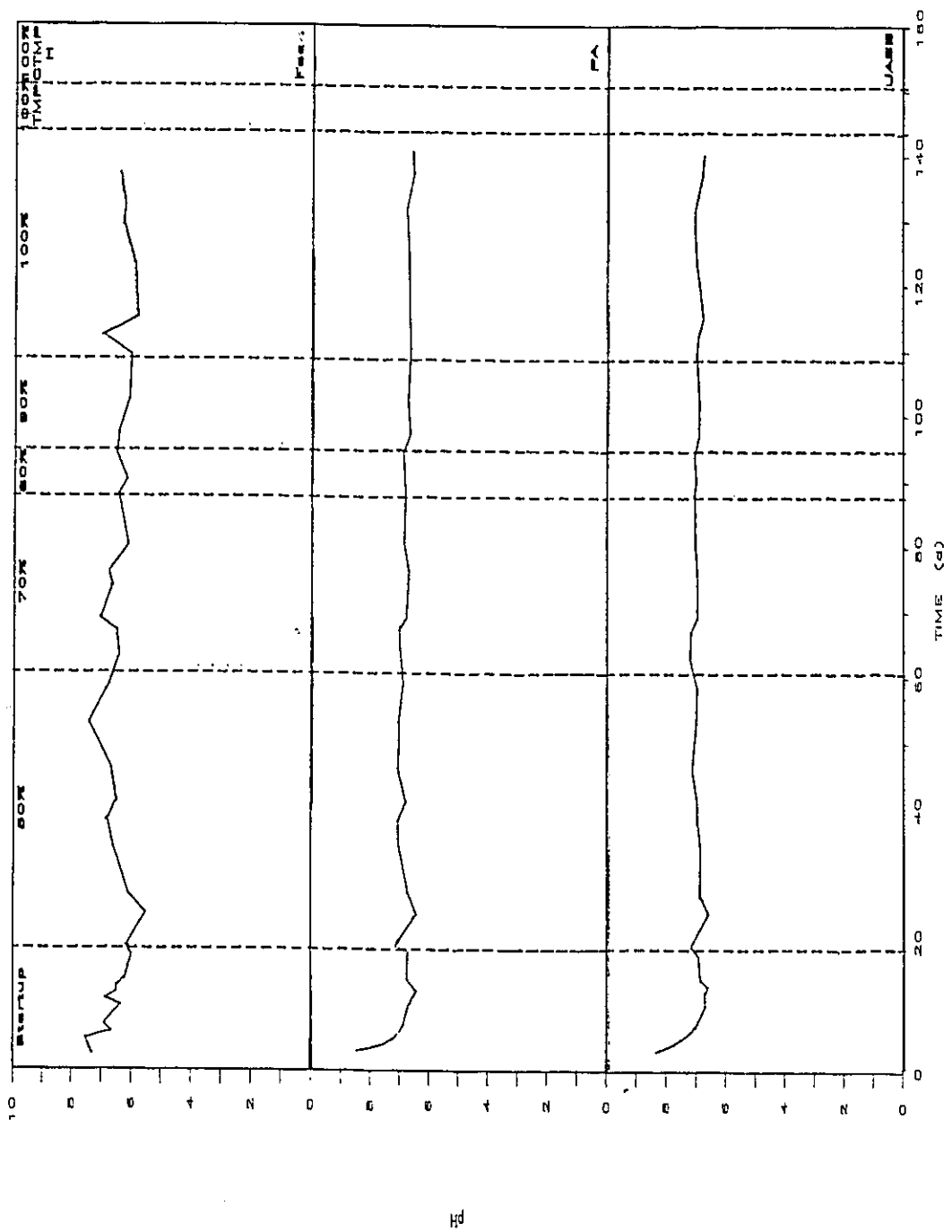


Figure C2 pH versus operating time

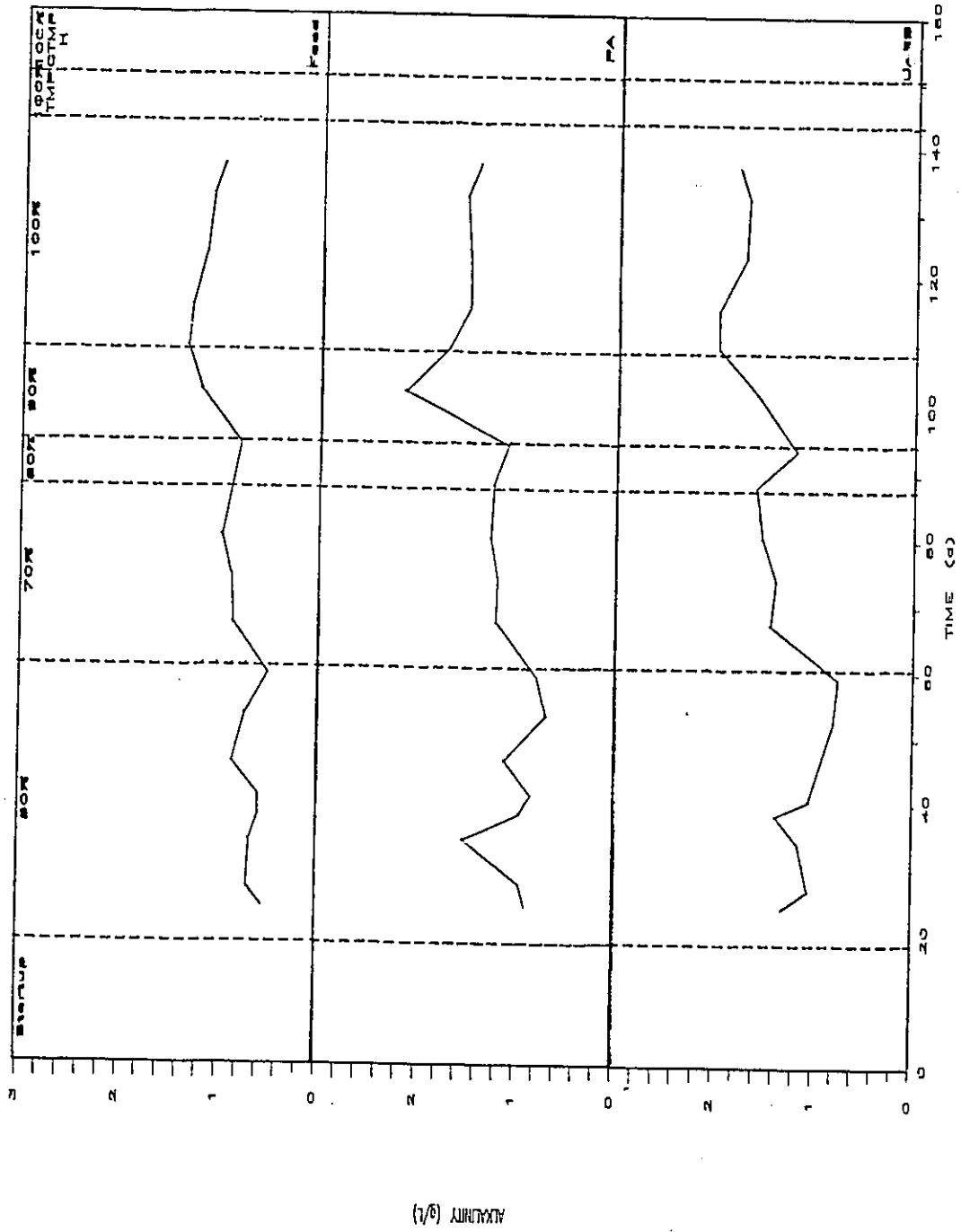


Figure C3 Alkalinity versus operating time

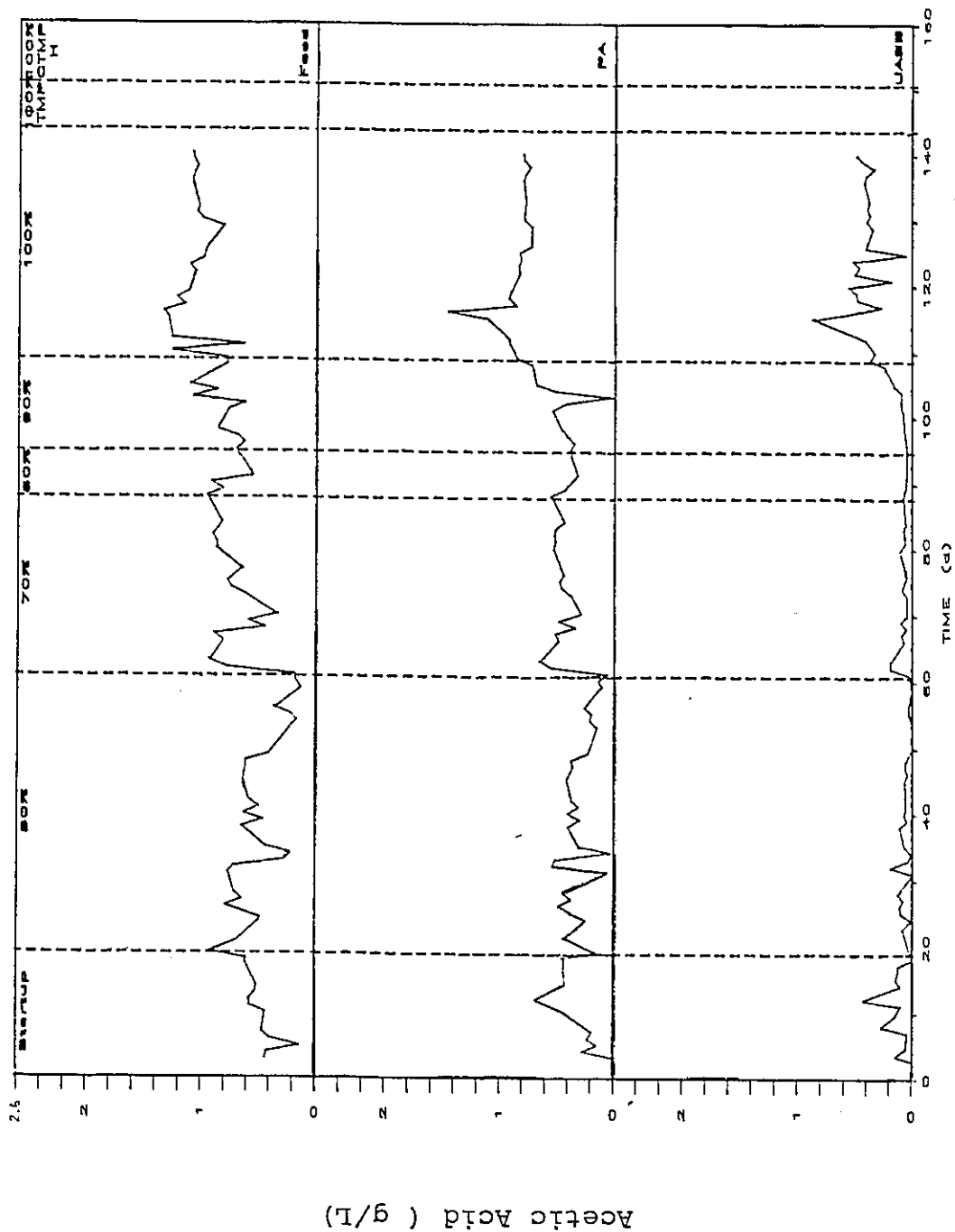


Figure C4a Acetic acid versus operating time

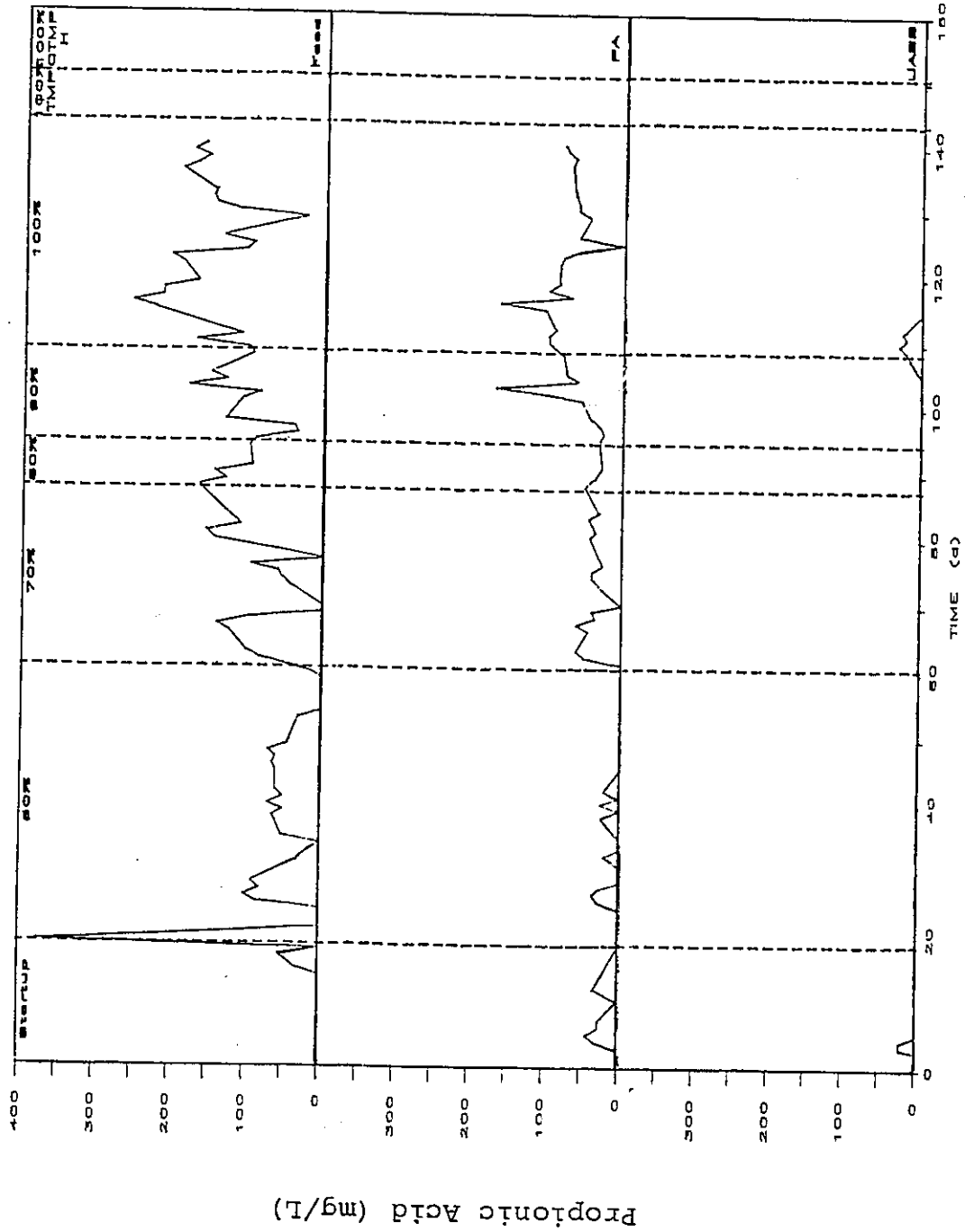
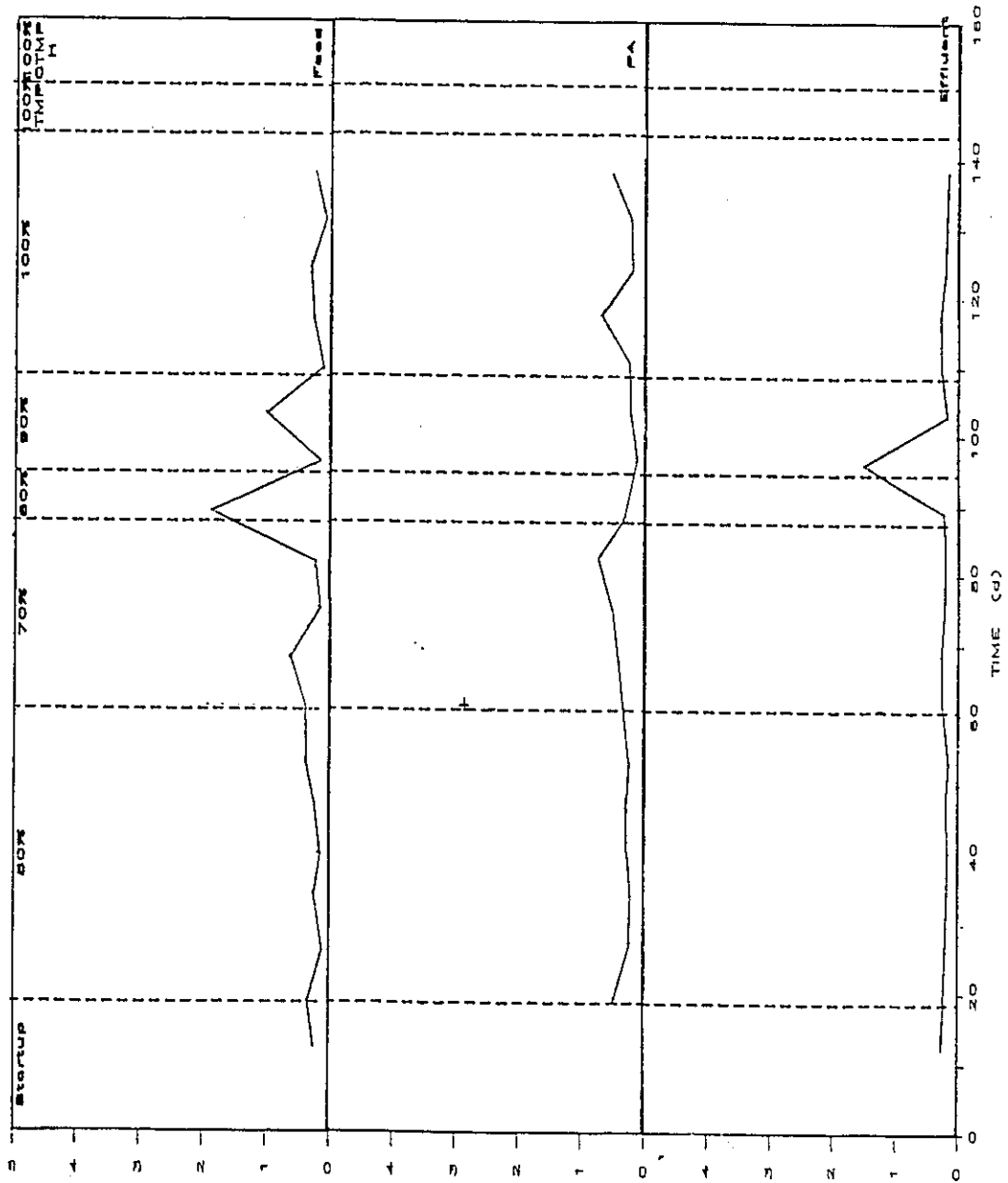
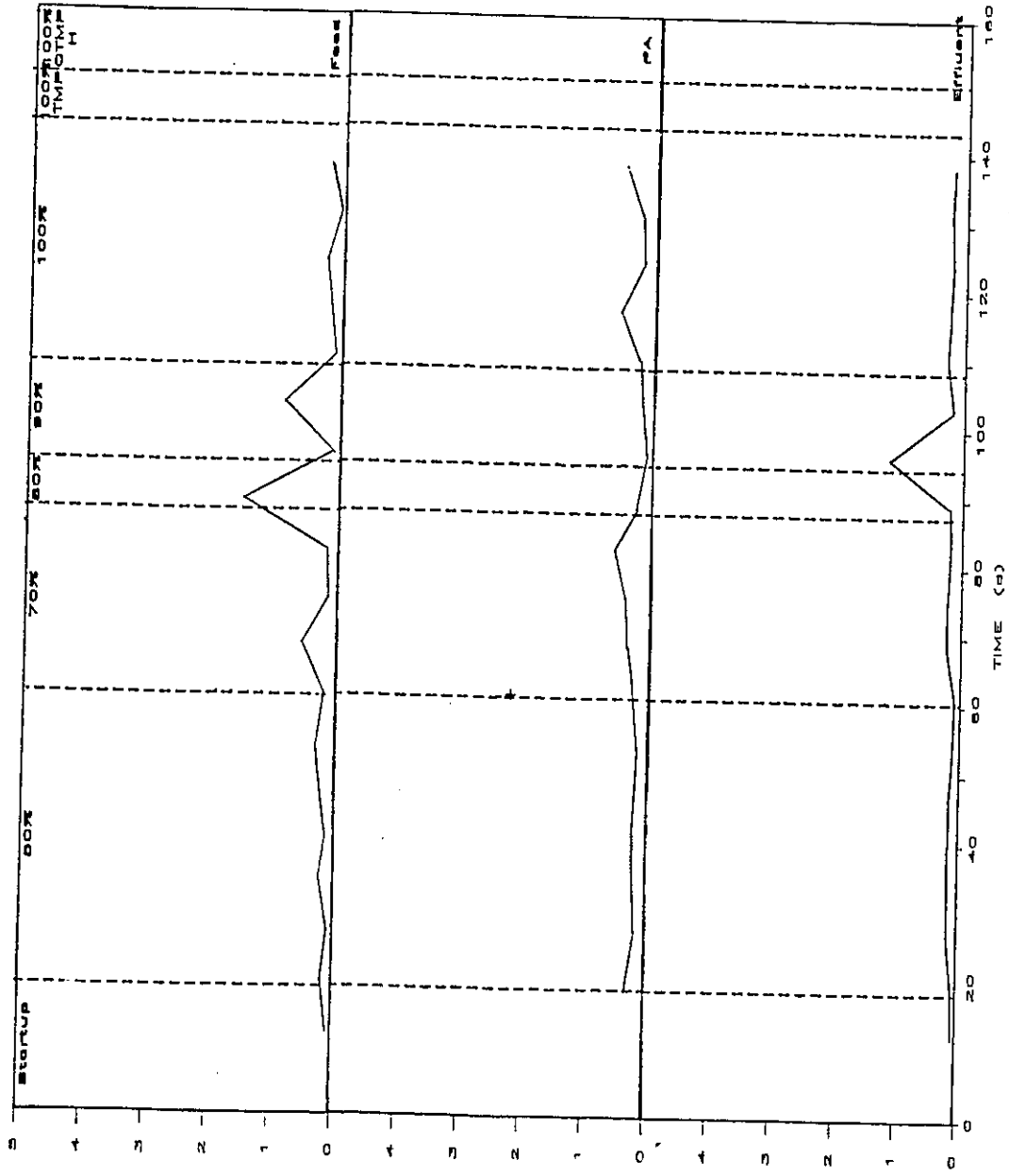


Figure C4b Propionic acid versus operating time



(1/6) SSI

Figure C5a TSS versus operating time



(1/8) SSA

Figure C5b VSS versus operating time

Table C3. Calculated period averages for system PL3

PREACIDIFICATION TANK															
FEED															
Wastewater %	VFA			pH	Alk (mg/L)	COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	Tank Volume (L)	VFA				
	Ace (mg/L)	Pro (mg/L)	But (mg/L)								Ace (mg/L)	Pro (mg/L)	But (mg/L)		
0-50%	524	9	0	6.50	2690	2537	0.231	0.07	2.88	403	17	0	1780	1540	0.227
50%	498	53	0	6.46	2246	1941	0.278	0.209	2.55	299	6	0	1734	1416	0.280
70%	744	86	0	6.56	3282	2651	0.350	0.304	1.80	479	36	0	2757	2019	0.542
80%	796	127	0	6.36	2755	2340	1.063	0.116	2.81	425	36	0	2350	1487	0.323
90%	813	105	0	6.27	4375	3606	0.594	0.135	2.34	455	67	0	3514	2994	0.184
100%	1064	157	0	6.23	4582	3926	0.207	0.177	2.12	867	77	0	3900	3181	0.391

Wastewater %	UASB REACTOR										EFFLUENT		
	VFA			pH	Alk (mg/L)	Biogas Fractions			COD Total (mg/L)	COD Soluble (mg/L)	TSS (g/kg)	VSS (g/kg)	
	Ace (mg/L)	Pro (mg/L)	But (mg/L)			N ₂ (%)	CH ₄ (%)	CO ₂ (%)					
CTMP-N 0-50%	145	0	0	6.84	1644	24	63	9	1420	1213	0.268	0.057	
50%	47	0	0	6.99	1644	5	79	10	1241	1055	0.203	0.114	
70%	78	0	0	7.08	1463	3	78	13	1738	1388	0.230	0.140	
80%	52	0	0	7.09	1400	3	78	13	1577	1127	0.244	0.143	
90%	98	2	0	6.84	1600	2	77	15	1900	1661	0.865	0.544	
100%	414	4	0	6.89	1646	3	76	15	2447	2114	0.252	0.222	

Table C4. Data calculations for system PL3

Date	Day	Flow Rate Measured (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS Concentration (g/L)	SLR		SRR		SLRSPRR		
			PA (hrs)	UASB (hrs)	Total (g/L-d)	Soluble (g/L-d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)		Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (%)	Soluble (%)	Total (gCOD/gVSS.d)	Soluble (gCOD/gVSS.d)	Total (%)
25-Aug-88	41	16.13	3.1	5.1				6.42	1.60	1.60	50.84	37.59		17.6	90.1						
26-Aug-88	42	15.79	4.1	5.2				7.49	1.60	1.60	50.72	37.54		17.5	90.1						
27-Aug-88	43	16.13	3.3	5.1				6.60	1.60	1.60	50.60	37.49		17.4	90.0						
28-Aug-88	44	15.76	3.2	5.2				6.39	1.60	1.60	50.48	37.44		17.4	89.9						
29-Aug-88	45	15.87	3.1	5.3	10.84	10.16	48.3	7.30	1.60	1.60	50.35	37.39		17.4	89.8	0.67	0.63	0.36	0.32	48.32	56.50
30-Aug-88	46	15.67	3.1	5.3	11.09	9.16	47.7	6.97	1.60	1.60	50.23	37.34		17.4	89.7	0.69	0.67	0.33	0.28	47.72	49.26
31-Aug-88	47	15.83	3.3	5.2				6.71	1.60	1.60	50.11	37.29		17.3	89.7	0.69	0.67	0.33	0.28	47.72	49.26
01-Sep-88	48	17.74	3.0	4.7	7.06	6.05	36.0	4.85	1.55	1.55	49.87	37.24		17.3	89.6	0.49	0.38	0.18	0.11	36.02	28.87
02-Sep-88	49	14.72	3.6	5.6				7.77	1.55	1.55	49.76	37.19		16.8	87.0						
03-Sep-88	50	17.45	3.2	4.7				5.82	1.55	1.55	49.63	37.09		16.7	87.5						
04-Sep-88	51	16.24						6.24	1.55	1.55	49.51	37.04		16.7	87.4						
05-Sep-88	52	15.00	3.6	6.0	7.05	4.48	46.6	2.83	1.55	1.55	49.39	36.99		16.7	87.3	0.44	0.28	0.20	0.09	46.58	33.93
06-Sep-88	53	13.77	3.2	5.1				3.82	1.55	1.55	49.27	36.94		16.7	87.3	0.66	0.54	0.17	0.06	24.76	11.83
07-Sep-88	54	16.16	3.0	4.6	10.90	6.77	24.6	4.71	1.58	1.58	49.15	36.89		16.9	88.3						
08-Sep-88	55	17.85	3.0	4.6				5.87	1.60	1.60	49.03	36.84		17.1	88.9						
09-Sep-88	56	14.04	3.9	5.9				4.24	1.60	1.60	48.91	36.79		17.1	88.9						
10-Sep-88	57	14.45						3.65	1.60	1.60	48.79	36.74		17.1	88.8						
11-Sep-88	58	14.85	3.6	5.4				2.76	1.60	1.60	48.67	36.69		17.1	88.7						
12-Sep-88	59	15.25	3.5	5.2				3.70	1.60	1.60	48.55	36.64		17.0	88.6						
13-Sep-88	60	15.90	3.3	5.2				3.70	1.60	1.60	48.55	36.64		17.0	88.6						
70%CTMP-N																					
14-Sep-88	61	13.99	2.1	6.0	7.46	6.21		6.32	2.00	2.00	48.41	36.61		21.3	73.2	0.48	0.32				
15-Sep-88	62	16.89	2.4	4.9				6.36	1.90	1.90	48.27	36.56		20.2	69.5						
16-Sep-88	63	18.27	2.1	4.5	21.99	15.87	53.4	9.79	1.80	1.80	48.13	36.54		19.1	65.8	1.36	0.97	0.73	0.43	53.38	44.41
17-Sep-88	64	14.02	3.1	5.9				9.66	1.76	1.76	47.99	36.51		18.6	63.9						
18-Sep-88	65	16.71	2.5	4.9				11.17	1.75	1.75	47.85	36.46		18.6	63.6						
19-Sep-88	66	17.38	2.6	4.8	18.42	13.54	49.6	10.40	1.70	1.70	47.70	36.45		18.0	62.0	1.02	0.84	0.49	0.37	48.02	43.06
20-Sep-88	67	16.27	2.4	5.1				19.43	1.80	1.80	47.56	36.41		19.1	65.5						
21-Sep-88	68	17.29	2.7	4.8	15.63	10.90	43.7	9.67	1.70	1.70	47.42	36.36		16.0	61.8	0.97	0.66	0.42	0.28	43.73	37.79
22-Sep-88	69	17.47	2.7	4.7				10.99	1.70	1.70	47.28	36.35		16.0	61.6						
23-Sep-88	70	17.85	2.7	4.6	14.42	10.64	41.4	8.77	1.80	1.80	47.14	36.32		19.0	65.4	0.69	0.66	0.37	0.24	41.37	36.59
24-Sep-88	71	17.60						7.73	1.75	1.75	47.00	36.28		16.5	63.5						
25-Sep-88	72	17.35						7.40	1.70	1.70	46.86	36.25		16.5	63.5						
26-Sep-88	73	17.10	2.6	4.8	14.41	13.52	31.0	6.56	1.65	1.65	46.72	36.22		17.4	59.8	0.69	0.64	0.28	0.43	31.03	51.84
27-Sep-88	74	16.35	2.9	5.0				9.81	1.65	1.65	46.58	36.19		17.4	59.7						
28-Sep-88	75	15.58	3.1	5.3	14.58	13.91	46.6	10.21	1.60	1.60	46.44	36.15		16.8	57.8	0.90	0.66	0.42	0.42	46.58	48.63
29-Sep-88	76	16.90	2.5	4.9				10.21	1.60	1.60	46.29	36.12		16.8	57.8						
30-Sep-88	77	16.90	3.0	6.0	13.70	13.03	48.6	10.18	1.70	1.70	46.15	36.09		17.9	61.4	0.85	0.81	0.41	0.43	48.59	53.33
01-Oct-88	78	16.27						10.56	1.70	1.70	46.01	36.06		17.8	61.3						
02-Oct-88	79	15.93						10.87	1.60	1.60	45.87	36.02		17.8	61.3						
03-Oct-88	80	15.90	3.3	6.3	13.11	12.56	49.6	10.63	1.60	1.60	45.73	35.99		16.8	57.6	0.81	0.78	0.40	0.42	49.48	54.51
04-Oct-88	81	15.42	3.3	6.4				10.22	1.50	1.50	45.70	36.01		16.7	57.4						
05-Oct-88	82	16.90	3.0	5.0	15.66	13.70	29.5	10.34	1.60	1.60	45.67	36.03		16.7	54.0	0.97	0.65	0.29	0.36	29.54	42.81
06-Oct-88	83	15.95	3.2	6.2				9.83	1.60	1.60	45.64	36.05		16.7	54.1						

Date	Dry	Flow Rate Measured (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		SLR		SRR		Soluble (%)	
			PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOO/gVSS.d)	Soluble (gCOO/gVSS.d)	Total (%)	Soluble (%)		
			CONSTANT BIOMASS = 16.0 gVSS/L reactor BLR/SRR																		
06-Oct-86	83	15.95	3.2	5.2	20.09	11.68	66.9	69.0	9.83	1.50	1.50	45.84	36.06	16.7	54.1	1.25	0.72	0.83	0.42	66.89	68.96
07-Oct-86	84	16.67	3.2	5.3	20.09	11.68	66.9	69.0	9.46	1.45	1.45	45.61	36.07	16.2	52.3	0.89	0.75	0.30	0.34	33.44	44.79
08-Oct-86	85	16.17	3.2	5.3	20.09	11.68	66.9	69.0	10.25	1.38	1.38	45.57	36.09	14.5	49.8	0.71	0.71	0.41	0.41	57.46	57.46
09-Oct-86	86	14.77	3.1	4.8	11.48	11.48	57.5	57.5	7.37	1.40	1.40	45.39	36.20	14.7	50.7	0.70	0.62	0.32	0.34	45.68	54.42
10-Oct-86	87	14.37	3.1	4.8	11.48	11.48	57.5	57.5	7.85	1.36	1.36	45.36	36.22	14.5	50.0	0.81	0.81	0.27	0.27	34.05	34.05
11-Oct-86	88	16.72	3.1	4.8	11.48	11.48	57.5	57.5	8.50	1.38	1.38	45.33	36.24	14.6	50.0	1.08	1.08	0.66	0.66	60.34	60.34
12-Oct-86	89	16.09	3.1	4.8	11.48	11.48	57.5	57.5	8.19	1.37	1.37	45.30	36.26	14.4	49.7	1.23	1.23	0.67	0.67	54.05	54.05
13-Oct-86	90	16.52	3.3	5.9	14.37	12.08	33.4	44.8	11.16	1.50	1.50	45.46	36.14	15.8	54.2	1.53	1.45	0.63	0.63	41.00	46.87
14-Oct-86	91	17.32	3.5	4.8	11.48	11.48	57.5	57.5	8.83	1.46	1.46	45.45	36.16	16.2	52.4	1.43	1.35	0.62	0.62	43.40	51.13
15-Oct-86	92	17.02	3.1	4.8	11.48	11.48	57.5	57.5	9.61	1.40	1.40	45.42	36.18	14.7	50.7	1.38	1.38	0.58	0.58	48.08	42.11
16-Oct-86	93	16.72	3.1	4.8	11.48	11.48	57.5	57.5	7.85	1.36	1.36	45.36	36.22	14.5	50.0	1.30	1.30	0.54	0.54	36.41	36.41
17-Oct-86	94	16.09	3.1	4.8	11.48	11.48	57.5	57.5	8.50	1.38	1.38	45.33	36.24	14.6	50.0	1.20	1.20	0.44	0.44	45.28	45.28
18-Oct-86	95	16.52	3.3	5.9	14.37	12.08	33.4	44.8	8.19	1.37	1.37	45.30	36.26	14.4	49.7	1.53	1.45	0.63	0.63	36.56	36.56
19-Oct-86	96	16.00	2.8	4.6	13.01	13.01	34.0	34.0	10.32	1.25	1.25	45.05	36.41	13.2	45.5	1.43	1.35	0.62	0.62	43.40	51.13
20-Oct-86	97	16.39	3.0	5.0	13.01	13.01	34.0	34.0	10.65	1.25	1.25	45.02	36.43	13.2	45.5	1.38	1.38	0.58	0.58	48.08	42.11
21-Oct-86	98	16.78	3.4	5.2	13.01	13.01	34.0	34.0	9.94	1.20	1.20	45.14	36.35	12.7	43.6	1.30	1.30	0.54	0.54	36.41	36.41
22-Oct-86	99	16.17	3.4	5.2	13.01	13.01	34.0	34.0	10.65	1.25	1.25	45.02	36.43	13.2	45.5	1.20	1.20	0.44	0.44	45.28	45.28
23-Oct-86	100	16.67	3.3	4.9	19.45	17.43	66.6	60.3	10.32	1.25	1.25	45.05	36.41	13.2	45.5	1.53	1.45	0.63	0.63	36.56	36.56
24-Oct-86	101	16.96	3.3	4.9	19.45	17.43	66.6	60.3	10.65	1.25	1.25	45.02	36.43	13.2	45.5	1.43	1.35	0.62	0.62	43.40	51.13
25-Oct-86	102	13.21	3.8	6.2	20.31	19.88	64.0	64.0	10.65	1.25	1.25	45.02	36.43	13.2	45.5	1.38	1.38	0.58	0.58	48.08	42.11
26-Oct-86	103	17.85	3.5	4.6	20.31	19.88	64.0	64.0	9.67	1.45	1.45	44.99	36.45	15.4	52.9	1.20	1.20	0.44	0.44	45.28	45.28
27-Oct-86	104	18.28	3.7	4.6	19.37	17.47	45.0	61.9	11.03	1.20	1.20	44.92	36.49	12.7	43.8	1.53	1.45	0.63	0.63	36.56	36.56
28-Oct-86	105	15.61	3.4	5.3	19.37	17.47	45.0	61.9	10.35	1.25	1.25	44.95	36.47	13.3	45.6	1.43	1.35	0.62	0.62	43.40	51.13
29-Oct-86	106	17.12	3.4	5.3	19.37	17.47	45.0	61.9	10.35	1.25	1.25	44.95	36.47	13.3	45.6	1.38	1.38	0.58	0.58	48.08	42.11
30-Oct-86	107	18.63	2.2	4.1	24.71	23.37	41.0	40.9	10.97	1.35	1.35	44.86	36.53	14.3	49.3	1.53	1.45	0.63	0.63	36.56	36.56
31-Oct-86	108	20.15	2.2	4.1	24.71	23.37	41.0	40.9	10.97	1.35	1.35	44.86	36.53	14.3	49.3	1.43	1.35	0.62	0.62	43.40	51.13
01-Nov-86	109	13.04	3.0	6.3	23.13	21.86	43.4	61.1	10.25	1.35	1.35	44.83	36.55	14.3	49.3	1.30	1.30	0.54	0.54	36.41	36.41
02-Nov-86	110	16.93	3.3	4.9	23.13	21.86	43.4	61.1	12.45	1.35	1.35	44.80	36.56	14.7	50.5	1.43	1.35	0.62	0.62	43.40	51.13
03-Nov-86	111	16.22	3.3	5.1	22.48	22.48	49.1	42.1	12.00	1.30	1.30	44.77	36.58	13.8	47.6	1.38	1.38	0.58	0.58	48.08	42.11
04-Nov-86	112	15.65	3.3	5.3	22.48	22.48	49.1	42.1	11.73	1.30	1.30	44.74	36.60	13.8	47.6	1.30	1.30	0.54	0.54	36.41	36.41
05-Nov-86	113	16.34	3.3	5.3	22.48	22.48	49.1	42.1	2.63	1.28	1.28	44.71	36.62	13.6	46.9	1.38	1.38	0.58	0.58	48.08	42.11
06-Nov-86	114	15.02	3.7	6.8	20.99	20.17	34.4	39.4	6.65	1.31	1.31	44.67	36.64	14.0	48.0	1.20	1.20	0.44	0.44	45.28	45.28
07-Nov-86	115	14.70	3.6	6.6	20.99	20.17	34.4	39.4	6.97	1.25	1.25	44.64	36.66	13.3	45.8	1.30	1.30	0.54	0.54	36.41	36.41
08-Nov-86	116	14.64	3.6	6.6	20.99	20.17	34.4	39.4	6.10	1.38	1.38	44.61	36.68	14.7	50.6	1.43	1.35	0.62	0.62	43.40	51.13
09-Nov-86	117	10.30	5.1	8.0	16.63	13.62	45.3	48.8	5.82	1.38	1.38	44.58	36.70	14.7	50.6	1.53	1.45	0.63	0.63	36.56	36.56
10-Nov-86	118	12.47	4.1	6.6	16.63	13.62	45.3	48.8	6.59	1.35	1.35	44.55	36.72	14.7	50.7	1.43	1.35	0.62	0.62	43.40	51.13
11-Nov-86	119	13.06	3.6	6.3	16.03	14.50	35.6	39.8	6.08	1.35	1.35	44.52	36.74	14.4	49.6	1.20	1.20	0.44	0.44	45.28	45.28
12-Nov-86	120	13.91	3.6	6.3	16.03	14.50	35.6	39.8	6.82	1.35	1.35	44.49	36.76	14.7	50.7	1.53	1.45	0.63	0.63	36.56	36.56

Date	Day	Flow Rate Measured (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS Concentration (g/L)	SLR		SFR		SLR/SRR	
			PA (hrs)	UASB (hrs)	Total (g/L-d)	Soluble (g/L-d)	Total (%)	Soluble (%)				Total (gCOD/gVSS-d)	Soluble (gCOD/gVSS-d)		Total (%)	Soluble (%)				
13-Nov-88	121	14.77						6.76	1.38	1.38	44.46	36.77	14.5	50.7						
14-Nov-88	122	16.83	3.4	6.3	16.04	16.04	28.9	37.1	7.04	1.40	1.40	44.43	36.79	15.0	51.5	0.90	0.29	0.37	26.90	37.11
15-Nov-88	123	16.17	2.3	6.1	16.34	14.08	37.1	36.1	6.90	1.40	1.40	44.40	36.81	15.0	51.6	0.96	0.36	0.32	37.11	36.11
16-Nov-88	124	14.95	3.4	6.6	17.24	14.64	46.4	45.0	7.31	1.43	1.43	44.38	36.83	16.3	52.7	1.07	0.50	0.41	46.43	44.96
17-Nov-88	125	16.82						7.06	1.50	1.50	44.33	36.85	16.1	55.3						
18-Nov-88	126	16.29	3.6	6.1	20.90	18.07	34.8	48.9	7.96	1.46	1.46	44.30	36.87	16.1	55.3	1.30	0.45	0.57	34.78	46.91
19-Nov-88	127	16.07						9.28	1.55	1.55	44.24	36.91	17.2	59.1						
20-Nov-88	128	16.85	3.6	6.3	22.63	19.74	49.3	49.2	8.95	1.60	1.60	44.21	36.93	16.8	57.2	1.40	0.60	0.80	49.34	49.24
21-Nov-88	129	16.37	3.4	5.0	20.44	18.07	40.2	42.4	9.50	1.50	1.50	44.18	36.95	16.1	56.4	1.27	0.51	0.49	40.25	42.43
22-Nov-88	130	17.16	3.1	4.8	17.38	17.38	32.1	32.1	9.37	1.50	1.50	44.16	36.97	16.1	56.4	1.06	0.36	0.36	32.06	32.06
23-Nov-88	131	17.16	3.1	4.8	16.58	16.58	40.1	48.6	9.21	1.50	1.50	44.12	36.99	16.1	56.5	1.03	0.41	0.47	40.10	48.63
24-Nov-88	132	17.36	3.1	4.8	19.90	13.98	40.1	29.1	9.00	1.50	1.50	44.09	37.00	16.1	56.5	1.23	0.49	0.26	40.09	29.14
25-Nov-88	133	14.61	3.4	6.7				8.09	1.55	1.55	44.05	37.02	16.7	57.4						
26-Nov-88	134	14.92						8.09	1.77	1.77	44.02	37.04	16.7	57.4						
27-Nov-88	135	16.33	3.4	6.4				8.09	1.80	1.80	43.99	37.06	17.2	59.3						
28-Nov-88	136	16.79	3.0	4.9				8.84	1.55	1.55	43.96	37.08	16.2	56.6						
29-Nov-88	137	14.29	3.4	6.8				8.09	1.55	1.55	43.93	37.10	16.2	56.7						
30-Nov-88	138	16.33	1.7	6.1				8.09	1.60	1.60	43.93	37.10	16.2	56.7						
01-Dec-88	139	14.76	3.2	6.0				8.09	1.60	1.60	43.93	37.10	17.3	59.4						
02-Dec-88	140							8.09	1.60	1.60	43.93	37.10	17.3	59.4						

Table C5. Calculated period average of the data
calculations for system PL3

Wastewater %	Flow Rate Measured Q (L/d)	HRT		OLR		COD Remove		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.0 gVSS/L reactor					
		PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOOY/gVSS.d)	Soluble (gCOOY/gVSS.d)	Total (gCOOY/gVSS.d)	Soluble (gCOOY/gVSS.d)	Total (%)	Soluble (%)
		SRR		SLR		SLR		SRR		SLR		SRR		SLR		SRR				
CTMP-N 0-50	10.67	6.16	6.61	7.45	7.01	48.89	62.25	2.99	1.40		54.08	39.19	16.92	54.76	0.46	0.43	0.22	0.23	46.89	52.25
50%	16.21	3.47	6.16	10.64	9.11	42.12	40.62	6.85	1.64		50.96	37.64	16.89	58.10	0.65	0.66	0.28	0.24	42.12	40.62
70%	16.36	2.81	6.02	16.00	12.91	45.92	47.22	10.21	1.65		48.62	38.22	17.33	59.62	0.99	0.80	0.46	0.38	45.92	47.22
80%	16.36	3.73	5.14	12.87	11.21	39.66	62.22	6.77	1.41		46.39	36.20	14.65	51.09	0.80	0.69	0.31	0.36	39.66	62.22
90%	16.95	3.25	4.96	22.46	18.23	47.62	49.45	9.75	1.28		46.06	36.40	13.60	48.44	1.39	1.13	0.62	0.57	47.62	49.45
100%	16.23	3.37	5.50	19.16	17.23	40.37	41.96	6.37	1.44		44.36	36.84	15.45	53.16	1.19	1.07	0.48	0.46	40.37	41.96

APPENDIX D
SAMPLE CALCULATIONS

D1. Flow Rate - Real Q

The flow rate, Q, is calculated as follows.

$$\text{Real Q (L/d)} = \frac{V_e}{t}$$

Where,

V_e = daily effluent volume (L)

t = time (d).

D2. Hydraulic Retention Time - HRT

The hydraulic retention time the PA tank has been adjusted for the average change in volume of the PA tank, relative to the design volume, over a twenty-four hour period.

$$\text{HRT}_{\text{PA}} \text{ (hr)} = \frac{(\text{Design } V_{\text{PA}} + \text{Actual } V_{\text{PA}})/2}{Q}$$

The volume of the UASB reactor is constant therefore, the HRT of the UASB can be calculated as follows:

$$\text{HRT}_{\text{UASB}} \text{ (hr)} = \frac{\text{Design } V_{\text{UASB}}}{Q}$$

where,

V_{UASB} = UASB reactor volume (L).

D3. Organic Loading Rate - OLR

The organic loading rate is calculated from the COD load per volume of reactor per unit time:

$$\text{OLR (g COD/L reactor.d)} = \frac{\text{COD}_f \times Q}{V_{\text{UASB}}}$$

where,

COD_f = the COD of the feed (g/L).

D4. Percent COD Removal Efficiency

The COD removal efficiency expressed as a percentage can be calculated as follows:

$$\text{COD Removed (\%)} = \frac{(\text{COD}_f - \text{COD}_e)}{\text{COD}_f} \times 100.$$

D5. Specific Loading Rate - SLR

The specific loading rate is calculated from the COD loading per reactor biomass per unit time:

$$\text{SLR (g COD/g VSS.d)} = \frac{\text{COD}_f \times Q}{\text{BM}}$$

where,

BM = reactor biomass (g VSS).

D6. Specific Reaction Rate - SRR

The specific reaction rate is calculated from the COD removed per reactor biomass per unit time:

$$\text{SRR (g COD/g VSS.d)} = \frac{(\text{COD}_f - \text{COD}_e)}{\text{BM}} \times Q.$$

D7. Specific Methane Production Rate - SMPR

The specific methane production rate is calculated from the methane produced, expressed as COD, per reactor biomass per unit time:

$$\text{SMPR (g COD/g VSS.d)} = \frac{Q_g \times \%CH_4 \times F}{\text{BM}}$$

where,

Q_g = biogas production rate (L/d)

$\%CH_4$ = methane content of the biogas

$F = 2.532$; a factor to convert L CH_4 at 35°C to g COD.

APPENDIX E
CLARIFIER DATA AND CALCULATIONS

E1. Clarifier Sediment COD and Solids Analysis

Clarifier sediment was collected throughout the experiment and stored frozen in seven plastic bottles. On February 2, 1989 the bottles were thawed and analysed for COD_T, COD_S, TSS, and VSS concentrations. The analytical results are presented in Table D1.

Table E1. Clarifier sediment data

Bottle Number	Collection Period d	COD _T mg/L	COD _S mg/L	TSS g/L	VSS g/L
1	1 - 18	49730	3040	19.713	16.929
2	19 - 36	70170	3420	28.829	24.745
3	38 - 59	70170	3420	28.399	23.888
4	60 - 77	83130	4560	34.207	27.926
5	78 - 90	108780	4830	32.143	26.664
6	91 - 110	56500	5610	26.754	22.587
7	111 - 138	80880	6240	32.036	26.683

The COD and solids concentrations measured for each bottle did not equal the actual COD and solids content of the clarifier sediment for a particular day. However, they represent an average of the values for the time period during which the clarifier sediment was collected daily. For the purpose of analysis, for any given day, the daily COD and solids concentration of the sediment will be assumed equal to the period values during which the day occurred. Using these data, the COD and solids concentrations of clarified wastewater can be determined.

E2. Calculation of the COD and Solids Removed by the Clarifier

The characteristics of the clarified wastewater (COD_T , COD_S , TS, and VSS) were calculated based on the measured influent and settler sediment concentrations as follows:

From Table B1, on Day 11, the volume of the sediment (V_S) removed from the clarifier and the measured effluent volume (V_e) were:

$$V_S = 145 \text{ mL}$$

$$V_e = 4.86 \text{ L}$$

The COD of the sediment for the interval Day 11 to 18 was:

$$COD_T = 49730 \text{ mg/L of sediment}$$

$$COD_S = 3040 \text{ mg/L of sediment}$$

Calculate COD_T removed by the clarifier:

$$= \frac{[V_S \times COD_{TS}]}{V_e} \times \frac{1 \text{ L}}{1000 \text{ mL}}$$

$$= \frac{145 \times 49730}{4.86} \times \frac{1}{1000}$$

$$= 1541 \text{ mg/L of flow}$$

Similarly, the COD_S removed by the clarifier = 94 mg/L of sediment.

The COD, TSS, and VSS removed by the clarifier were calculated for each day of operation for which there was clarifier sediment removal data. These values were then averaged over the operation ranges (i.e. for each wastewater percent concentration period). These data are presented in

Table E2 and E3. These average values were used to calculate the average concentrations of COD, TSS, and VSS of the clarified wastewater as follows:

For the start-up period, 0 to 50% wastewater concentration, from Table D2, the average COD removed was:

$$\text{COD}_T = 893 \text{ mg/L}$$

$$\text{COD}_S = 55 \text{ mg/L}$$

and, the average COD of the feed was:

$$\text{COD}_T = 4403 \text{ mg/L}$$

$$\text{COD}_S = 2477 \text{ mg/L}$$

The average COD of the clarified wastewater was therefore:

$$\begin{aligned} \text{COD}_T &= (4403 \text{ mg/L}) - (893 \text{ mg/L}) \\ &= 3511 \text{ mg/L} \end{aligned}$$

and,

$$\begin{aligned} \text{COD}_S &= (2477 \text{ mg/L}) - (55 \text{ mg/L}) \\ &= 2422 \text{ mg/L} \end{aligned}$$

The percent COD removed can be calculated as follows:

$$\text{COD}_T \text{ removed} = (893/4403) \times 100 = 20.3\%$$

and

$$\text{COD}_S \text{ removed} = (55/2477) \times 100 = 2.2\%$$

Similarly, TSS and VSS removal can be determined.

E3. Calculation of Percent COD Solubilized in the Settler

The increase in the COD_S of the settler sediment relative to the feed COD_S is attributed to solubilization of

the wastewater. Percent solubilization is calculated as follows:

For the start-up period, 0 to 50% wastewater concentration, the average soluble COD of the sediment was:

COD_S = 3040 mg/L of sediment

and, the feed

COD_S = 2477 mg/L

$$\begin{aligned} \% \text{ COD}_S \text{ by Solubilization} &= \frac{(3040 - 2427)}{3040} \times 100 \\ &= 20.3\% \end{aligned}$$

Table E2. Calculated COD data after the clarifier

% Wastewater	Effluent Volume L	Clarifier Sediment			Removed by Clarifier				Feed		Influent to PL2		Removed by Clarifier		Clarifier - Feed		Clarifier % Solubilized
		Volume mL	COD Total mg/Lsediment	COD Soluble mg/Lsediment	COD Total mg	COD Soluble mg	COD Total mg/L flow	COD Soluble mg/L flow	COD Total mg/L flow	COD Soluble mg/L flow	COD Total mg/L flow	COD Soluble mg/L flow	COD Total %	COD Soluble %	COD Soluble mg/L	COD mg/L	
0-50%	9.83	103	49730	3040	8042	492	893	55	4403	2477	3511	2422	20.3	2.2	563	18.5	
50%	14.98	92	70486	3614	6226	350	439	23	4320	2519	3881	2496	10.2	0.9	1095	30.3	
70%	16.25	114	92630	4660	10719	532	650	33	5675	3144	5025	3111	11.5	1.0	1516	32.5	
80%	14.27	258	78906	5276	20261	1363	1393	96	7920	3870	6527	3574	17.6	2.6	1606	30.4	
90%	16.12	217	56500	5610	11342	1128	719	71	7300	4397	6581	4325	9.8	1.6	1213	21.6	
100%	15.3	172	80087	6219	13268	1031	900	70	9262	5021	8361	4851	9.7	1.4	1198	18.3	

Table E3. Calculated TSS and VSS data after the clarifier

% Wastewater	Clarifier Sediment		Removed by Clarifier				Feed		Influent to PL2		Removed by Clarifier			
	Effluent Volume L	Volume mL	TSS g/kg sediment	VSS g/kg sediment	TSS sediment	VSS sediment	TSS g/L flow	VSS g/L flow	TSS mg/L flow	VSS mg/L flow	TSS %	VSS %		
0-50%	9.83	103	19.713	16.929	3.188	2.738	0.354	0.304	1.077	0.808	0.723	0.504	32.9	37.6
50%	14.96	92	28.729	24.363	1.910	1.640	0.123	0.106	0.867	0.688	0.744	0.581	14.2	15.4
70%	16.25	114	33.443	27.459	2.255	1.937	0.140	0.120	1.371	1.114	1.231	0.993	10.2	10.8
80%	14.27	258	29.064	24.334	5.089	4.370	0.357	0.307	1.878	1.553	1.520	1.246	19.0	19.8
90%	16.12	217	26.754	22.587	4.272	3.669	0.260	0.223	1.295	1.050	1.035	0.826	20.1	21.2
100%	15.30	172	31.684	26.410	3.394	2.914	0.222	0.191	2.447	1.980	2.225	1.789	9.1	9.6

Table E4. Calculated period averages of the data calculations for system PL2 corrected for clarifier removal

Wastewater	Flow Rate Measured Q (L/d)	HRT		OLR		COD Removed		Biogas Production Rate (L/d)	Sludge Bed Volume (L)	Total Bed Volume (L)	Sludge Bed Concentration		Reactor VSS		CONSTANT BIOMASS = 16.3 gVSS/L reactor					
		PA (hrs)	UASB (hrs)	Total (g/L.d)	Soluble (g/L.d)	Total (%)	Soluble (%)				TSS (g/L)	VSS (g/L)	Concentration (g/L)	Total (g)	Total (gCOOY/gVSS.d)	Soluble (gCOOY/gVSS.d)	Total (%)	Soluble (%)	Total (%)	Soluble (%)
		SRR/SLR		SRR		SRR/SLR														
CTMP-N	9.27	4.69	10.7	9.57	6.90	57.37	50.04	2.27	1.42	1.60	64.56	39.17	16.40	66.77	0.79	0.46	0.51	0.22	66.06	50.74
-50%	15.84	2.77	6.19	16.08	11.63	37.86	37.85	8.22	1.81	2.00	50.45	37.60	20.00	68.02	1.26	0.73	0.80	0.26	47.30	36.30
70%	18.83	3.09	4.87	24.87	15.40	40.19	40.21	12.00	2.22	2.22	46.58	37.31	24.33	82.71	1.69	0.93	0.79	0.38	46.44	36.74
80%	16.46	3.62	4.97	31.59	17.30	50.05	49.17	13.03	2.22	2.22	45.62	37.92	24.71	84.01	2.31	0.90	1.35	0.46	56.56	45.10
90%	17.06	3.18	4.80	33.03	21.71	32.94	42.54	16.84	2.31	2.31	45.21	38.13	25.89	88.03	2.19	1.33	0.85	0.58	38.79	42.85
100%	15.73	3.10	5.23	36.66	22.90	35.92	33.07	14.83	2.57	2.57	44.37	36.58	29.12	99.02	2.56	1.40	1.12	0.47	42.23	33.76

APPENDIX F
MODIFIED ANAEROBIC TOXICITY ASSAY

F1. Theory

In the Modified Anaerobic Toxicity Assay, varying concentrations of wastewater, a nutrient supplement, and an acetate/propionate substrate are combined and inoculated with an active anaerobic culture in a closed serum bottle. Inhibitory effects of the wastewater are indicated by reduced rates of methane production or by the presence of lag periods. The degree of inhibition is expressed as a % activity loss relative to the control (contains no wastewater).

F2. Procedure

1. Prepare the defined medium, volatile organic stock solution, sulphide solution, and the dilution water.

All solutions should be prepared with glass distilled water (GDW), flushed continuously with 20 % carbon dioxide (CO₂/80% nitrogen (N₂) gas during preparation, transferred to sealed bottles with positive nitrogen gas pressure in the headspace, and stored at +4°C.

2. Maintain all constituents at 35°C in a water bath (or temperature controlled room). Prevent oxygen contamination during dispensing (i.e. anaerobic dispensation) by continuous gassing with 20% CO₂/80% N₂. This includes solutions, sludges, wastewater, and equipment (i.e. bottles, syringes, pipettes, etc.).

3. Adjust the wastewater pH to 7.0 with either 10 N NaOH or H₂SO₄ as required.

4. Tare the 160 mL bottles with caps and seals.

5. Using glass pipettes, anaerobically dispense 10 mL of the Defined Medium and 30 mL of the wastewater solution (wastewater plus dilution water where necessary) into calibrated 160 mL bottles. Depending on the concentration of the wastewater, select final assay volume fractions (volume wastewater/total liquid volume) of 5% to 60%. For high strength wastewater, select the highest volume fractions to produce maximum final assay COD_S of < 12,000 mg/L.

6. Perform tests in triplicate (eg. 5%, 35%, and 60% v/v) with two spiked controls. Treatment of the control bottles is identical to the three test bottles, except dilution water is substituted for the wastewater solution.
7. Add 0.5 mL of the sulphide solution to each serum bottle.
8. Bubble flush the serum bottle contents with 20% CO₂/80% N₂ until the resasurin redox indicator becomes colourless.
9. Add 8.0 mL of sludge, cap and seal the bottles, record the bottle weight, and place on a shaker incubating at 35°C.
10. After thirty minutes of incubation, inject 2.0 mL of the volatile organic acid (VOA) solution with a glass syringe (time $t = 0$ hours) for a total volume 50.5 mL in the bottle. Weigh the bottle and record.
11. Measure the initial soluble COD of each bottle. After injection of the VOA solution, sample 1.0 mL of the mixed liquor, micro-centrifuge, and perform a COD on the supernatant. The total volume is now 49.5 mL. Weigh and record the sample bottle.
12. Adjust the headspace to 1 atm by first putting an overpressure of 20% CO₂/80% N₂ gas in the serum bottle. Then insert a needle to release the over pressure. Check the bottle headspace with a pressure gauge. Repeat if necessary.
13. Measure the gas quantity and quality at $t = 0$ hours, after twenty-four hours, and every forty-eight hours thereafter for the first seven to ten days for each serum bottle. Make subsequent measurements one to three times a week depending on the production rate. This should correspond to measurements at least once for every 20 mL biogas produced. The experiment ends when the production of biogas ceases.

Gas volumes can be measured with a manometer (Figure F1). During measurement, slightly agitate the serum bottle to release gas from the liquid phase. Adjust the pressure in the manometer to 1 atm by raising or lowering the manometer reservoir to equilibrate the liquid levels of the manometer and reservoir.

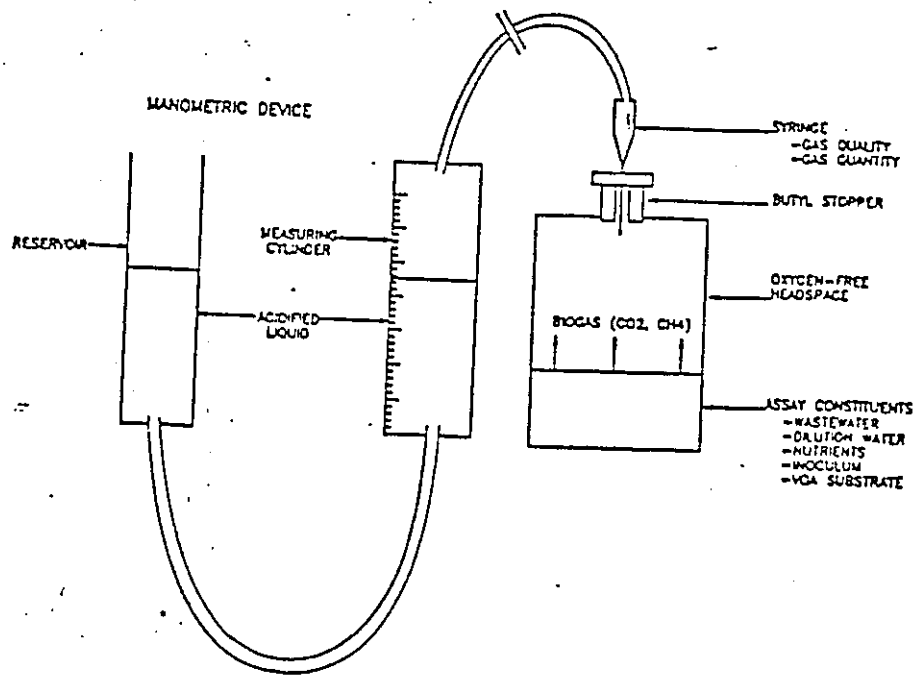


Figure F1. Schematic of serum bottle and manometric biogas measuring device

14. Determine the TSS and VSS concentration of the stock sludge. Calculate the initial TSS and VSS concentration the serum bottles.
15. Measure the final COD_T, COD_S, pH, TSS, and VSS in each serum bottle.

Defined Medium

Prepare the stock solutions listed in Table F1. Combine the stock solutions in the proportions listed in Table F2. Boil for 5 minutes and add 3.4 g sodium bicarbonate (NaHCO_3). Cool the solution to 35°C . Top the solution to 1 L with GDW.

Solution	Components	Concentration in distilled water (g/L)
Mineral I(1)	NaCl	50
	$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	10
	NH_4Cl	189.4
	$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	10
Mineral II	$(\text{NH}_4)_5\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	10
	$\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	0.1
	H_3BO_3	0.3
	$\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$	1.5
	$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	10
	$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$	0.03
	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	0.03
Vitamin B	$\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	0.1
	Nicotinic acid	0.1
	Cyanocobalamine	0.1
	Thiamine	0.05
	p-aminobenzoic acid	0.05
	Pyridoxine	0.25
Phosphate	Panpöthenic acid	0.025
	KH_2PO_4	50
Resazurin		0.1
2-methyl-n-butyric acid(2)		102

(1): Dissolved in 0.01 M HCl rather than distilled water.

(2): Adjusted to pH 6.5.

Table F1. Stock solutions used in defined medium (Cornacchio et al., 1986)

Solution	Volume (ml)
Distilled water	900
Mineral I	10
Mineral II	1
Vitamin B	1
Phosphate	10
Resazurin	15
2-methyl-n-butyric acid	1

Table F2. Defined Medium (Cornnachio *et al.*, 1986)

Volatile Organic Acid (VOA) Stock Solution

Dissolve 37.5 g sodium acetate and 13.25 g sodium propionate in 1 L GDW.

Sulphide Solution

Dissolve 5 g $\text{Na}_2\cdot 9\text{H}_2\text{O}$ in 200 mL freshly boiled GDW. Prepare in small aliquots prior to use.

Dilution Water

Deoxygenate GDW by boiling for five minutes while simultaneously flushing with 20% CO_2 /80% N_2 gas.

F3. Calculations

The inhibitory effects of the wastewater on the anaerobic micro-organisms is identifiable by comparing the rate and extent of methane production in the serum bottles containing wastewater to the spike control bottles. Mathematically, the degree of inhibition, expressed as a percentage relative to a control, can be calculated as follows:

$$\% \text{ CH}_4 = \frac{\text{Fraction CH}_4}{\text{SUM(Fractions N}_2, \text{CO}_2, \text{CH}_4)} \times 94$$

where 94 is a moisture content correction factor at 35°C.

At time $t = 0$ hours,

$$\text{Cumulative CH}_4 = \frac{\% \text{ CH}_4}{100} \times V_H \quad (\text{mL})$$

where V_H = serum bottle head space (mL).

At time $t = n$ hours,

$$\begin{aligned} (\text{Cumulative CH}_4)_n = (V_H + V_M) \times \frac{(\% \text{ CH}_4)_n}{100} - \frac{h \times (\% \text{ CH}_4)_{n-1}}{100} \\ + (\text{Cumulative CH}_4)_{n-1} \end{aligned}$$

where n = integer and

V_M = manometric volume (mL).

$$\begin{aligned} \text{Activity} &= \frac{(\text{initial slope L CH}_4/\text{d}) \times (2.53 \text{ g COD/L CH}_4 \text{ 35}^\circ\text{C})}{\text{g VSS in bottle}} \\ &= \text{g COD converted/g VSS.d} \end{aligned}$$

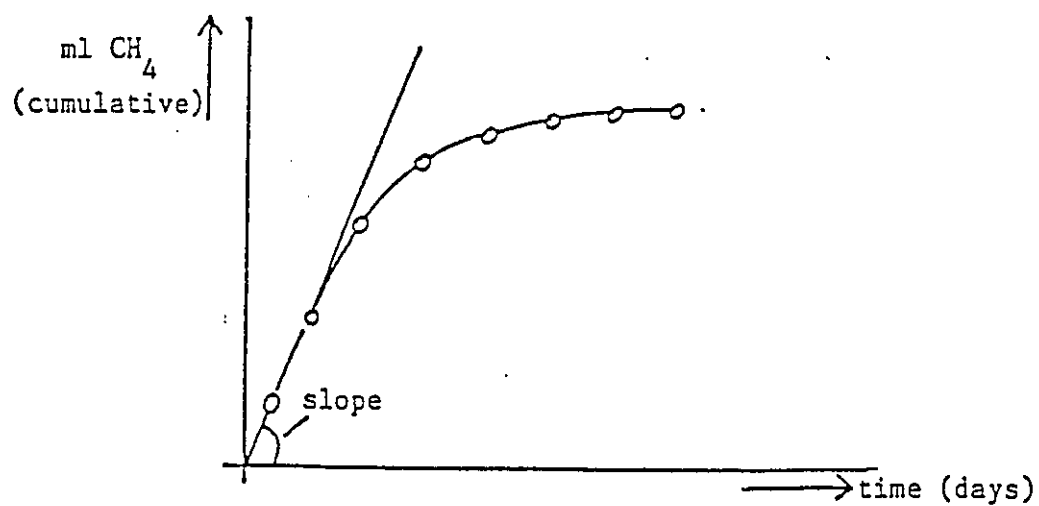


Figure F2. Cumulative methane gas production curve

$$\% \text{ Inhibition} = \left[1 - \frac{\text{Activity}}{\text{Activity of the control}} \right] \times 100$$

APPENDIX G

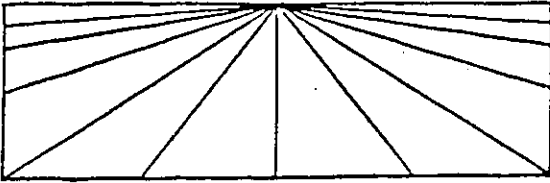
ANALYTICAL REPORT: RESIN AND FATTY ACIDS

G1. Resin and Fatty Acids

Feed, effluent, and sludge samples were collected during the feeding of 100% wastewater and analysed for their resin and fatty acid content. Paracel Laboratories Ltd., Nepean, Ontario performed the analytical work. The analytical report is presented herein. The sample records and analysis codes are presented in Table G1.

Table G1. Sample Records

Sample	I.D.	Date	Phase Total	Soluble	Paracel Code
PL1 feed	F1T F1S	05.12.88	x	x	B212 B208
PL3 feed	F3T F3S	05.01.89	x	x	B213 B207
PL1 effluent	1T 1S	05.12.88	x	x	B204 B209
PL2 effluent	2T 2S	30.11.88	x	x	B210 B211
PL3 effluent	3T 3S	02.12.88	x	x	B205 B206
DOM Sludge		22.02.89			B317
PL1 Sludge		22.02.89			B316
PL2 Sludge		22.02.89			B315
PL3 Sludge		22.02.89			B314



**PARACEL
LABORATORIES LTD.**

8 - 17 Grenfell Crescent
Nepean, Ontario K2G 0G3

260

March 2, 1989

ANALYTICAL REPORT

SUBMITTED BY: Eva Andras,
R.F.L., N.R.C.,
1 John St.
Ottawa.

DATE RECEIVED: January 21st, 1989.

The ten (10) water samples received - B204 to B213 (Inclusive) - were analysed for resin and fatty acids by gas chromatography with flame ionization detection according to PAPRICAN Draft Method, May 1988. Results and detection limits are shown in the attached Tables. Calculations are based on an internal standard and recovery is monitored by the addition of a surrogate compound. Copies of chromatograms are enclosed.

All chromatographic data will be kept on file. Results are not corrected for recovery.

All the samples will be returned and Paracel's laboratory number can be found on each label. Attached is a matched list.

W.G. Craig, Ph.D.,
President.

PARACEL NUMBER/SAMPLE IDENTIFICATION

B204 PL1 5-12-88 CTMP/N 100% Min.
B205 PL3 2-12-88
B206 PL3 SOLUBLE 2-12-88
B207 FILTERED SOLUBLE CTMPN/N 5-1-89
B208 PL TANK SUPPLEMENTED CTMP/N 5-12-88
B209 PL1 SOLUBLE 5-12-88 CTMPN 100% RUN
B210 PL2 30-11-88
B211 PL2 30-11-88 SOLUBLE
B212 SUPPLEMENTED PL TANK CTMPN (T) 5-12-88
B213 FILTERED CTMPN LAST BUCKET 5-1-89

CONCENTRATION OF RESIN AND FATTY ACIDS IN WATER SAMPLES
(µg/L)

COMPOUND	B204	B205	B206	B207	B208	B209	B210	B211	B212	B213
LINOLEIN ACID	2.66	0.1	0.23	0.21	0.49	1.43	2.5	1.66	0.98	0.28
LINOLENIC ACID	<0.1	<0.01	<0.01	<0.01	<0.01	<0.1	<0.1	<0.1	<0.1	<0.01
OLEIC ACID	1.27	0.08	0.12	<0.01	0.23	0.63	1.6	0.71	0.52	0.16
PIMARIC ACID	2.78	0.39	0.48	0.52	0.32	2.84	2.91	2.59	1.13	0.60
SANDARACOPIMARIC ACID	0.63	0.07	0.09	0.15	0.1	0.67	0.67	0.54	0.36	0.18
ISOPIMARIC ACID	9.86	1.87	2.2	0.94	0.62	8.55	9.27	7.58	2.11	1.03
PALUSTRIC ACID]	1.95	0.12	0.18	1.8	0.93	1.88	3.70	2.85	4.30	2.30
LEVOPIMARIC ACID]										
DEHYDROABIETIC ACID	26.3	6.57	8.26	<u>4.42</u>	<u>1.50</u>	26.1	22.0	20.0	4.08	4.60
ABIETIC ACID	0.65	0.16	0.23	1.17	0.63	0.79	0.91	0.76	2.69	1.33
NEOABIETIC ACID	0.39	0.05	0.06	0.55	0.31	0.35	0.52	0.43	1.18	0.63
STEARIC ACID	12.8	1.69	3.44	<u>0.98</u>	<u>0.79</u>	12.4	11.8	12.8	1.26	0.97
CHLOROBIETIC ACID	<0.1	<0.01	<0.01	<0.01	<0.01	<0.1	<0.1	<0.1	<0.1	<0.01
DICHLORODEHYDROABIETIC ACID	<0.1	0.02	0.03	<0.01	0.08	<0.1	<0.1	<0.1	0.13	<0.01

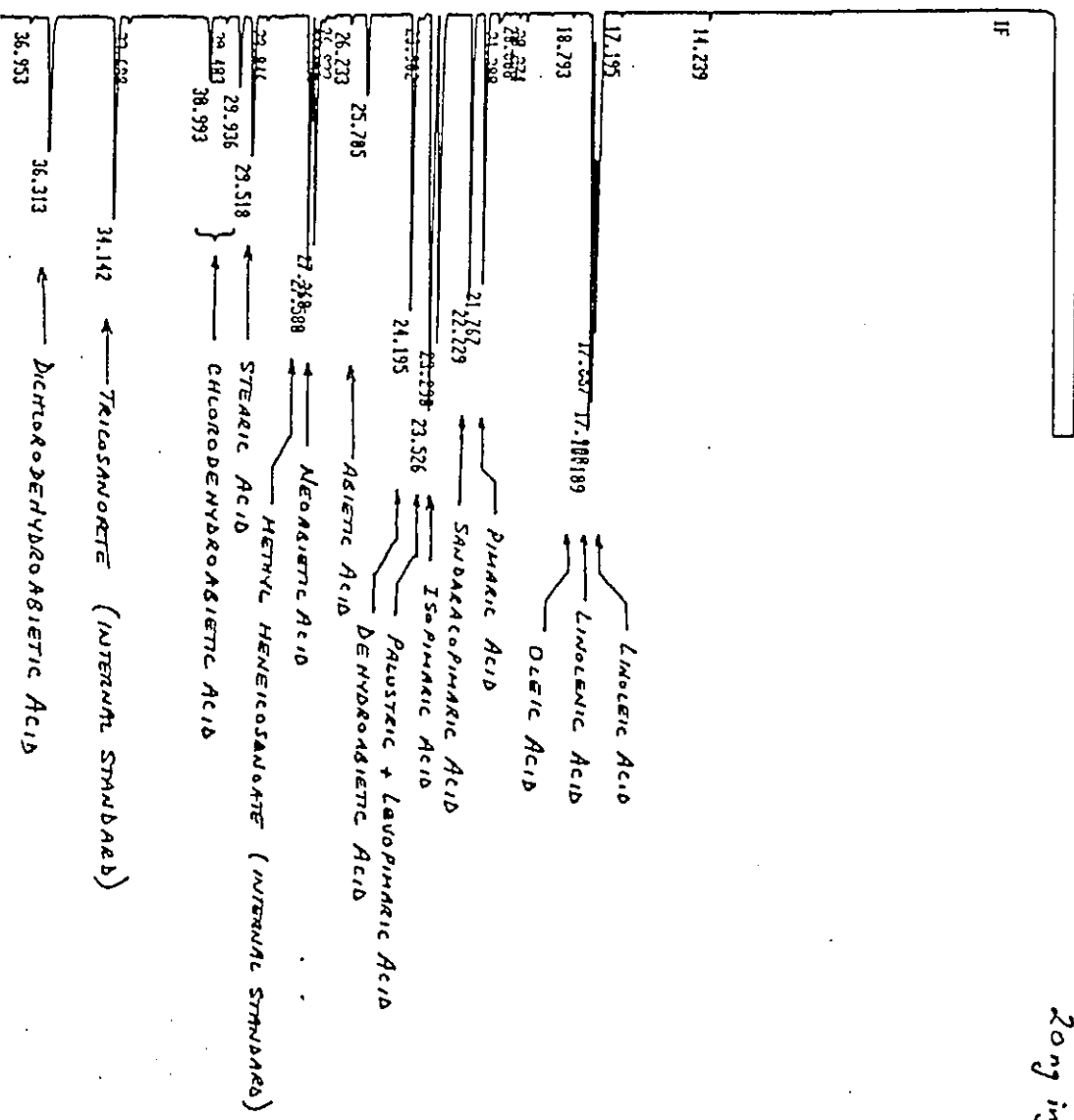
RECOVERY OF SURROGATE STANDARD
(%)

COMPOUND	B204	B205	B206	B207	B208	B209	B210	B211	B212	B213
O-METHYLPODOCARPIC ACID	100	93.1	94.4	63.1	63.1	100	100	100	74.2	70.9

START IF FEB 11, 1989 08:41:54

RESIN AND FATTY ACID STANDARDS

Zong in



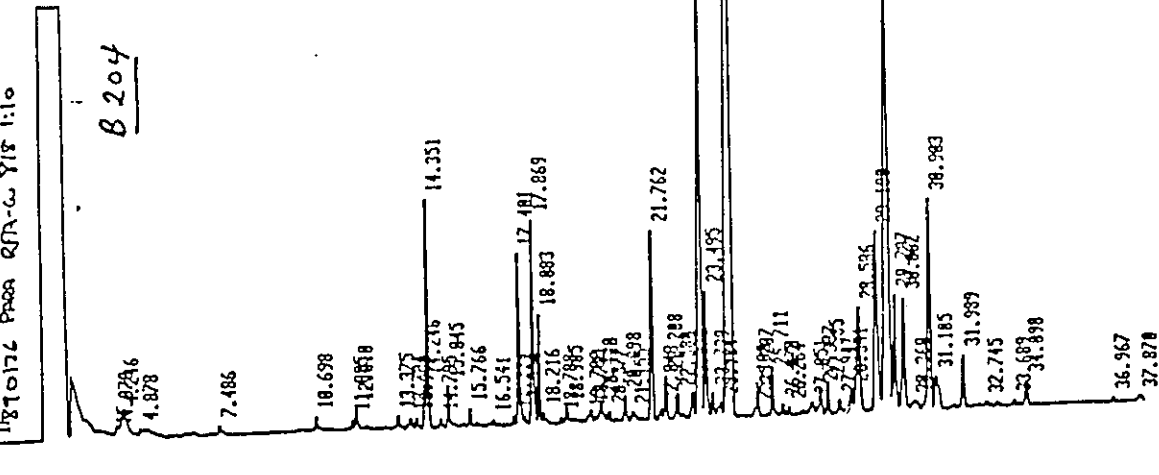
RUN# 521 FEB 11, 1989 08:41:54

SAMPLE# 21

AREA#

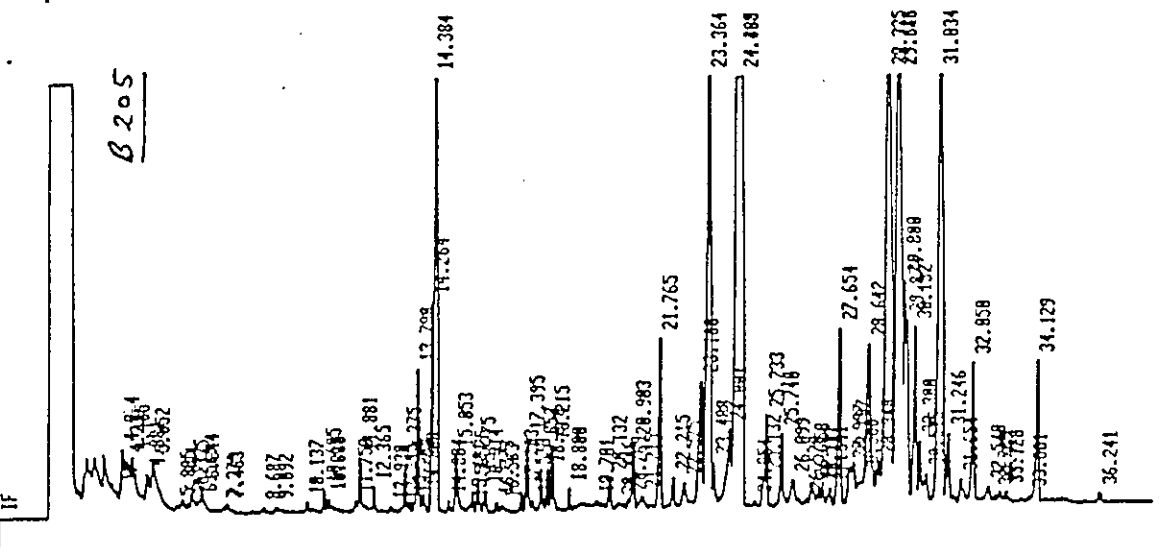
RT	AREA	TYPE	WIDTH	AREA
17.887	112891	8V	.878	7.88133
17.980	183551	VV	.859	7.28887
18.189	112883	VV	.868	7.93782
20.374	2578	BP	.867	.18126
21.380	4892	BP	.117	.34397
21.767	92731	P8	.876	6.52818
22.229	96314	V8	.874	6.77282

RUN # 544 FEB 14, 1989 08:40:34
 START IF
 S40126 PRC-QTR-C YR 1:10



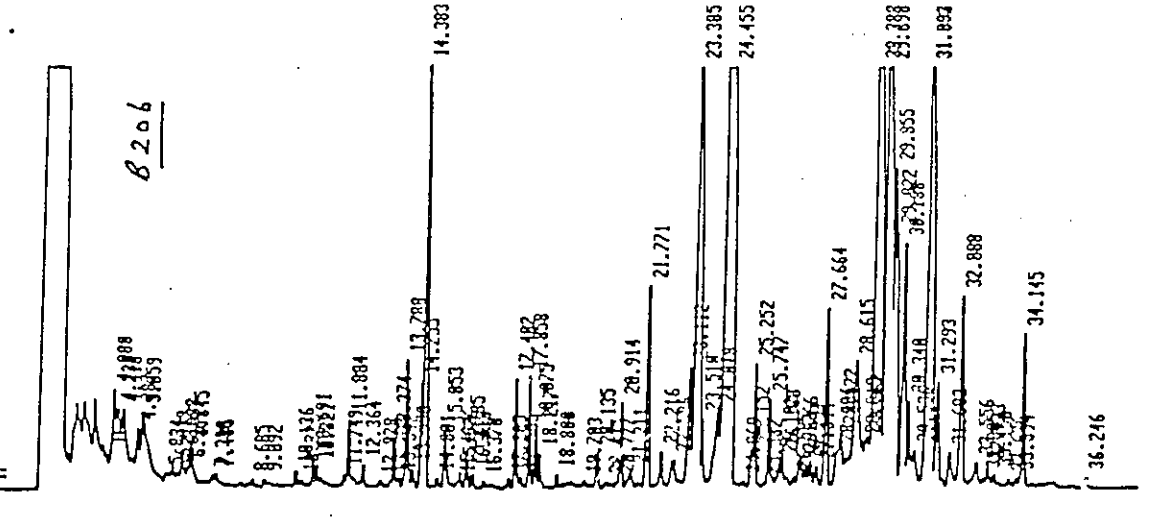
RUN# 544 FEB 14, 1989 08:40:34
 TIME TABLE STOP
 AREA% RT AREA TYPE WIDTH AREA2
 4.246 21338 VP .232 1.00044
 4.246 21338 VP .232 1.00044
 13.214 13214

RUN # 531 FEB 11, 1989 07:40:52
 START IF
 S40126 PRC-QTR-C YR 1:11



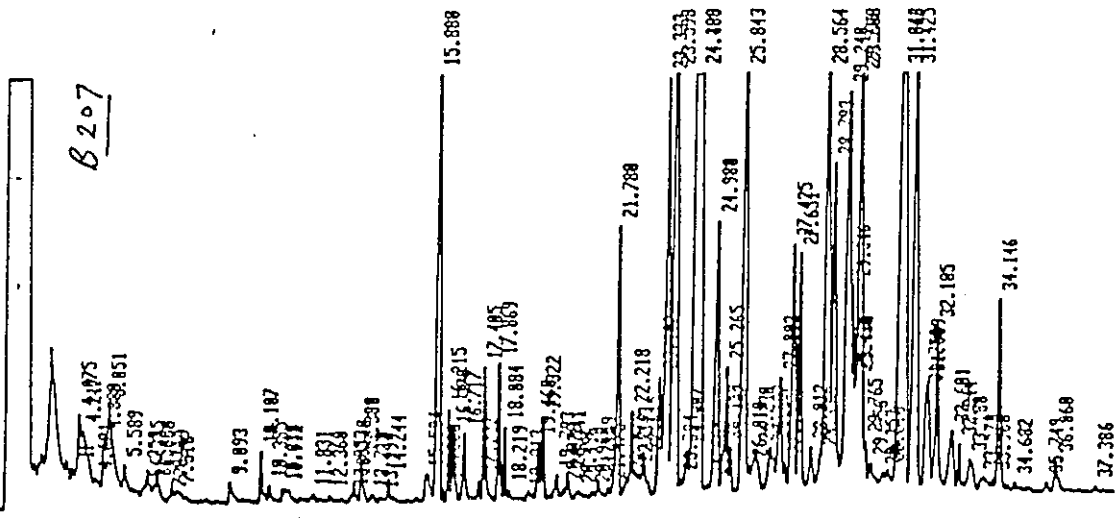
RUN# 531 FEB 11, 1989 07:40:52
 TIME TABLE STOP
 AREA% RT AREA TYPE WIDTH AREA2
 4.880 28451 BY .106 .55422
 4.880 28451 BY .106 .55422

RUN # 532 FEB 11, 1989 08:22:47
 START IF
 S40126 PRC-QTR-C YR 1:11



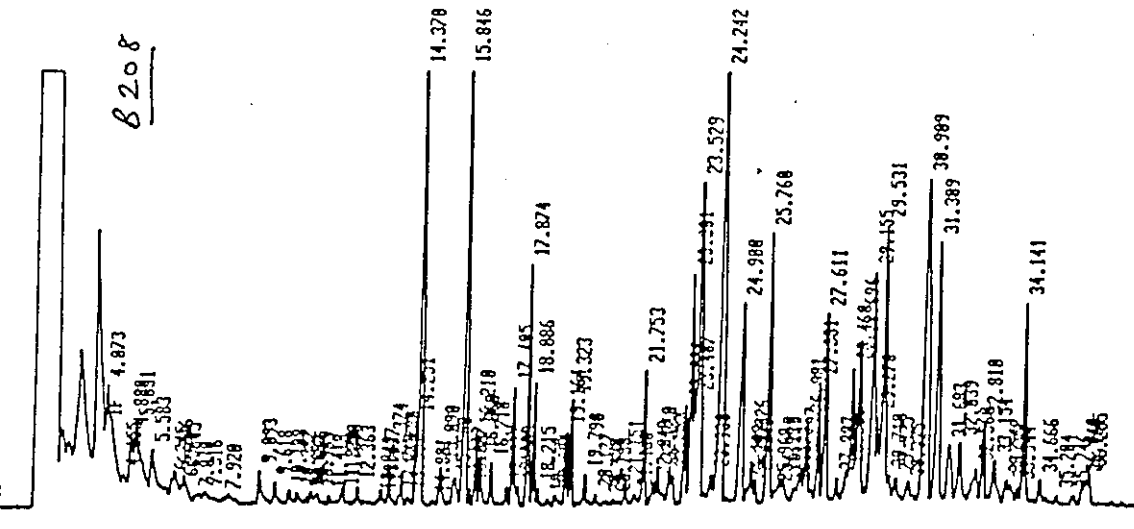
RUN# 532 FEB 11, 1989 08:22:47
 TIME TABLE STOP
 AREA% RT AREA TYPE WIDTH AREA2
 4.880 28451 BY .106 .55422
 4.880 28451 BY .106 .55422

SA0126 PAR-RFA-w-821 1:1
 RUN # 533 FEB 11, 1989 09:04:43
 START IF



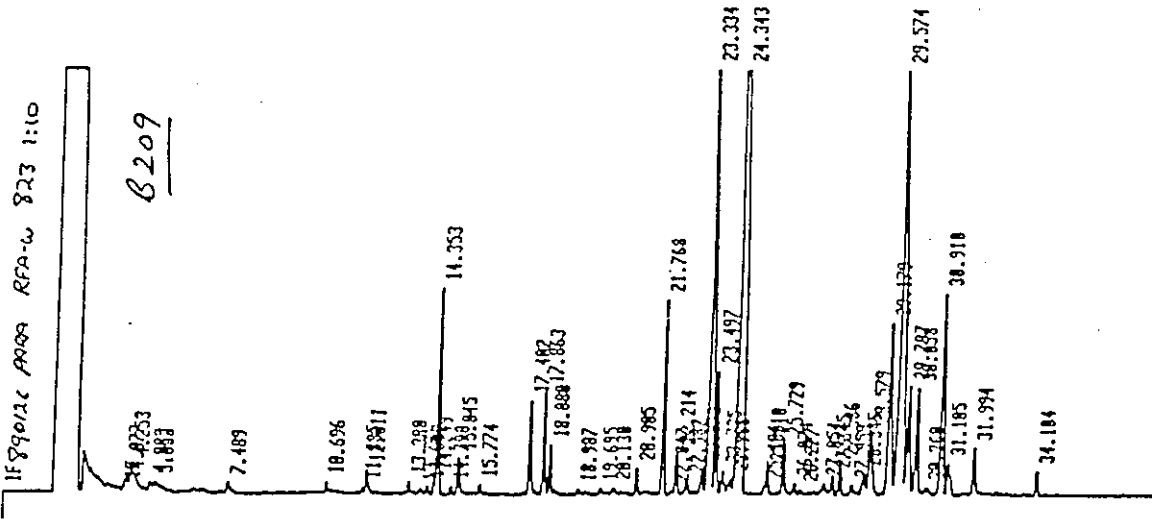
TIMETABLE STOP
 RUN# 533 FEB 11, 1989 09:04:43
 SAMPLE# 33
 AREA# RT AREA TYPE WIDTH AREA%

SA0126 PAR-RFA-w-822 1:1
 RUN # 534 FEB 11, 1989 09:46:37
 START IF



TIMETABLE STOP
 RUN# 534 FEB 11, 1989 09:46:37
 SAMPLE# 34
 AREA# RT AREA TYPE WIDTH AREA%

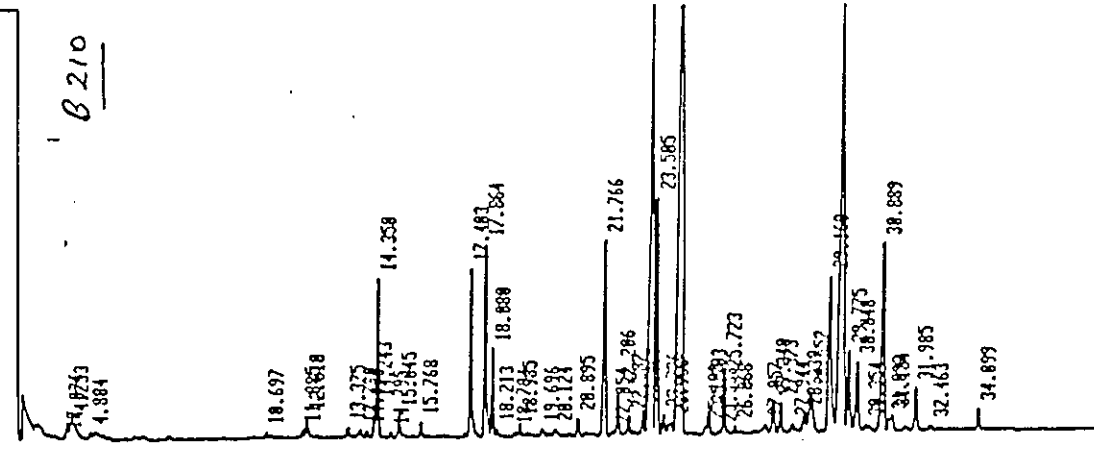
RUN # 545 FEB 14, 1989 09:22:29
 START IF



TIMETABLE STOP
 RUN# 545 FEB 14, 1989 09:22:29
 SAMPLE# 629
 AREA# RT AREA TYPE WIDTH AREA%

RUN # 546 FEB 14, 1989 10:05:27

START IF 890126 PARA 824 1:10



B 210

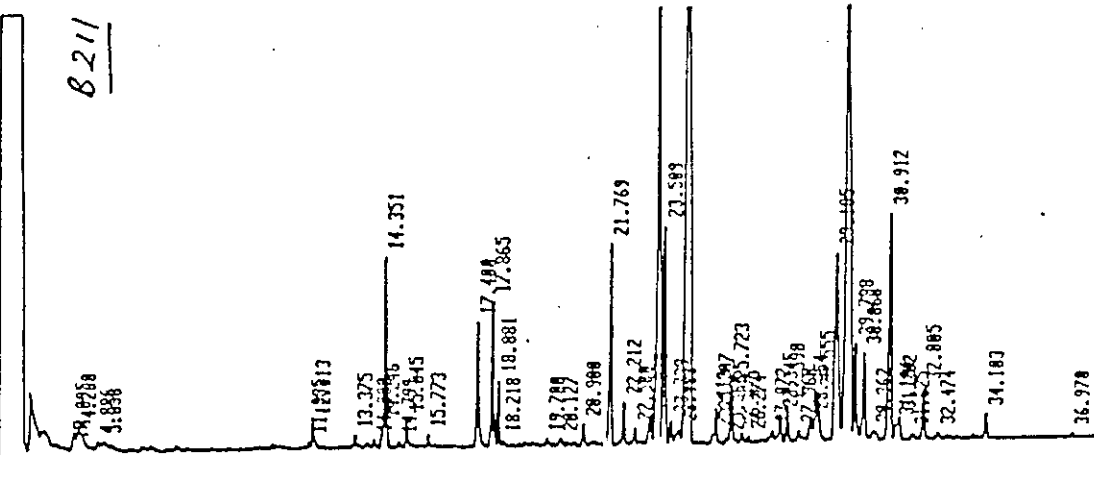
TIMETABLE STOP

RUN# 546 FEB 14, 1989 10:05:27 SAMPLE# 4

AREA#	RT	AREA	TYPE	WIDTH	AREA#
4.076	5185	BY	.119	28663	
4.253	16138	VV	-.285	98565	
4.884	2825	BY	.117	15862	

RUN # 547 FEB 14, 1989 10:47:24

START IF 890126 PARA 825 1:10



B 211

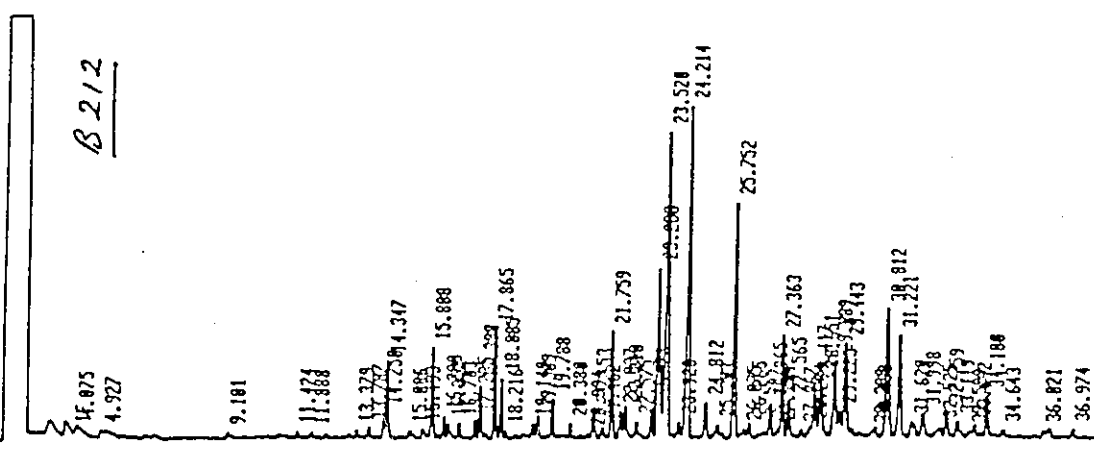
TIMETABLE STOP

RUN# 547 FEB 14, 1989 10:47:24 SAMPLE# 5

AREA#	RT	AREA	TYPE	WIDTH	AREA#
4.095	6427	BY	.123	33557	
4.268	19840	VV	-.215	1.03589	
4.892	2254	BY	.122	14000	

RUN # 548 FEB 14, 1989 11:29:25

START IF 890126 PARA 826 1:10



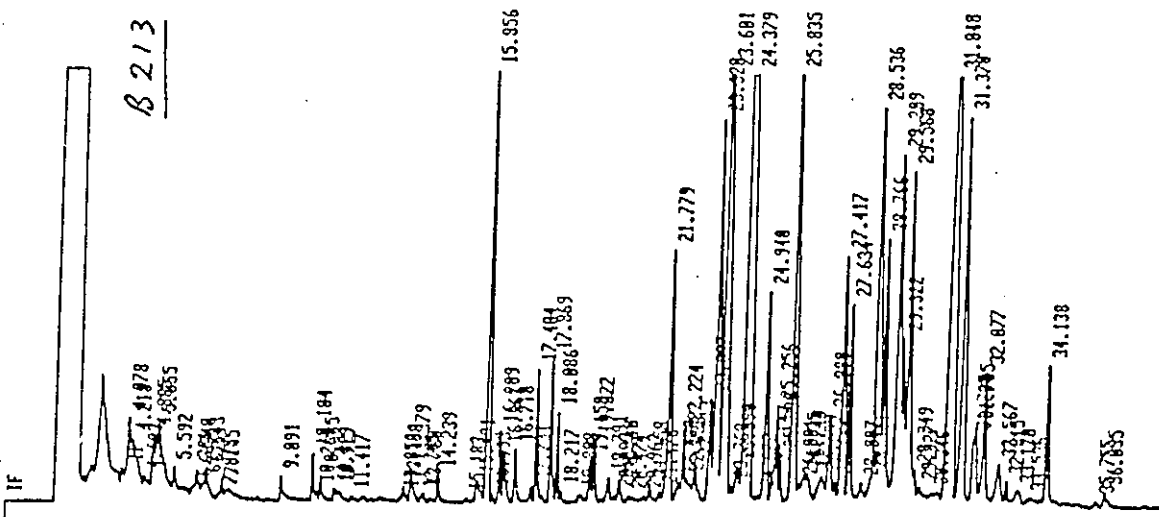
B 212

TIMETABLE STOP

RUN# 548 FEB 14, 1989 11:29:25 SAMPLE# 6

AREA#	RT	AREA	TYPE	WIDTH	AREA#
4.075	7487	EP	.276	65316	
4.927	3583	PV	.134	31258	

SA 0126 Pac- PFAW- 827 1:1
RUN # 541 FEB 11, 1989 14:18:06
START

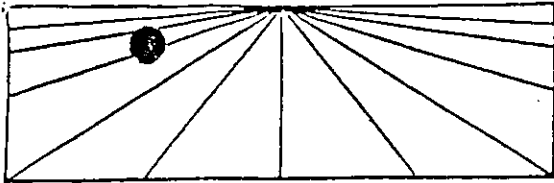


TIMETABLE STOP

RUN# 541 FEB 11, 1989 14:18:06

SAMPLE# 39

AREA#	RT	AREA	TYPE	WIDTH	AREA#
4.878	24356	RV	109	51801	



**PARACEL
LABORATORIES LTD.**

8 - 17 Grenfell Crescent
Nepean, Ontario K2G 0G3

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April 5th, 1989.

Eva Andras, ✓
NRC,
Division of Biological Sciences,
Rideau Falls Laboratory,
Ottawa, K1A 0R6.

RE: Analysis of Sludge Samples for Resin Acids

Four (4) sludge samples, received March 5th, 1989, were analysed for resin and fatty acids by gas chromatography with flame ionization detection. Because these samples appeared concentrated and had solid particulate matter, a 10 ml aliquot of each sample was made up to 50 ml with deionized water and then extracted. Results and detection limits are shown in the attached table.

B314 = PL 3, Sludge, cold room stored, 22.2.89.
B315 = PL 2, Sludge, " " " "
B316 = PL 1, Sludge, " " " "
B317 = Seed Sludge, (Domtar) 22.2.89

Sincerely,

PARACEL LABORATORIES,

W.G. Craig per P.

W.G. Craig, Ph.D.,
President.

CONCENTRATION OF RESIN AND FATTY ACIDS IN SLUDGE
(mg/L)

COMPOUND	B314	B315	B316	KDL	B317	KDL
LINOLEIN ACID	0.943	10.2	315	0.100	105	0.010
LINOLENIC ACID	-	-	-	100	-	10
OLEIC ACID	4.11	22.4	28.8	100	0.181	10
PIMARIC ACID	32.	115	104.	100	0.665	10
SANDARACOPIMARIC ACID	4.15	19.2	18.5	100	0.291	10
ISOPIMARIC ACID	146	543	515	100	52	10
PALUSTRIC ACID]	-	-	171	100	0.187	10
LEVOPIMARIC ACID]						
DEHYDROABIETIC ACID	122,	287	349	100	13.8	10
ABIETIC ACID	0.969	167	639	100	0.067 9	10
NEOABIETIC ACID	-	-	197	100	-	10
STEARIC ACID	-	-	-	100	-	10
14-CHLORODEHYDROABIETIC ACID	403	-	881	100	0.075 7	10
12-CHLORODEHYDROABIETIC ACID	0.77	-	-	100	0.219	10
12,14-DICHLORODEHYDROABIETIC ACID	-	-	-	100	-	10

KDL = DETECTION LIMIT

Total concentration of palustric acid and levopimaric acid is shown in the row for palustric acid.