REPORT No. DOE/CE/
41178

FINAL TECHNICAL REPORT

COMMERCIALIZATION OF THE BIOFINE TECHNOLOGY
FOR
LEVULINIC ACID PRODUCTION FROM PAPER SLUDGE

DOE Patent Clearance Granted
Mark P. Dvorscak June 3, 2002
Mark P Dvorscak Date
(630) 252-2393
E-mail: mark.dvorscak@ch.doe.gov
Office of Intellectual Property Law
DOE Chicago Operations Office

DOE Cooperative Agreement No. DE-FC02-95CE41178

April 23, 2002

BioMetics, Inc.
Waltham, Massachusetts

DOE/CE/41178

NOTICE

TABLE OF CONTENTS

PREFACE

PREFACE

This project involved a three-year program managed by BioMetics Inc. (Waltham, MA) to demonstrate the commercial feasibility of Biofine thermochemical process technology for conversion of cellulose-containing waste or renewable materials into levulinic acid, a versatile platform chemical.

The program, commencing in October 1995, involved the design, procurement, construction and operation of a plant utilizing the Biofine process to convert 1 dry ton per day of paper sludge waste. The plant was successfully designed, constructed and commissioned in 1997. It was operated for a period of one year on paper sludge from a variety of source paper mills to collect data to verify the design for a commercial scale plant. Operational results were obtained for four different feedstock varieties. Stable, continuous operation was achieved for two of the feedstocks. Continuous operation of the plant at demonstration scale provided the opportunity for process optimization, development of operational protocols, operator training and identification of suitable materials of construction for scale up to commercial operation. Separated fiber from municipal waste was also successfully processed.

The project team consisted of BioMetics Inc., Great Lakes Chemical Corporation (West Lafayette, IN), and New York State Energy Research and Development Authority (Albany, NY).

INTRODUCTION

INTRODUCTION

This is the final report of a technology development program carried out by BioMetics Inc. (BMI) Waltham, MA on the Biofine lignocellulose fractionation technology. The program was jointly funded by the U.S. Department of Energy, New York State Energy Research and Development Authority, Great Lakes Chemical and Biofine.

This program involved design, construction and operation of a one ton per day demonstration plant based on technology developed and patented by Biofine, Inc. to fractionate waste paper sludge into the primary product levulinic acid, a valuable chemical intermediate and byproducts formic acid and a bone dry carbonaceous char.

BMI operated the plant to collect data to verify the design for a commercial scale plant. Operating instructions and plant optimization parameters were developed. The operations results and process assessment data are provided in the OPERATIONS RESULTS section.

The project team consisted of BMI (Waltham, MA), Biofine, Inc. (Waltham, MA), Great Lakes Chemical Corporation (West Lafayette, Indiana), and New York State Energy Research and Development Authority (Albany, New York)

BMI provided overall management of the program. This involved all project management, engineering, design, cost estimating, procurement and construction services for the demonstration plant. BMI also provided all plant operations management.

Biofine provided the initial process engineering package containing know-how and technical data for the design. A outline of the Biofine process is presented in the Base Technology section.

Great Lakes Chemical Corporation (GLCC) is a specialty chemical company with worldwide operations. Levulinic acid and its derivatives represents a substantial potential market for GLCC. GLCC conducted laboratory pilot plant testing and confirmation of the proposed purification process for levulinic acid prior to implementation on plant.

New York State Energy Research and Development Authority (NYSERDA) has supported the development of the Biofine technology and continues to support this demonstration development as a method to derive value from biomass resources and as a means to develop new N.Y. business.

The Program Manager and Principle Investigator for BMI and Biofine was Dr. Stephen Fitzpatrick. Dr. Fitzpatrick coordinated all technical aspects of the program including all contractual matters with DOE. The project management company was BioMetics. Project manager at BioMetics was Mr. T. Y. Lam. Mr. Lam was responsible for the overall project execution and coordination. Dr. Colin South of BioMetics headed the process design team.

The demonstration plant was constructed at Epic Ventures Industrial Park, South Glens Falls, New York. In this final report the plant will be referred to as the SGF (South Glens Falls) plant. The final plant design documents and photographs are presented in the BIOFINE DEMONSTRATION PLANT section.

BMI also developed the preliminary design and an order of magnitude cost estimate for a commercial-size Biofine unit that could be installed at a typical paper mill or sludge processing plant. This estimate was used together with the demonstration plant operating results to reach a decision about implementation of a full-scale commercial unit. The commercial scale design report is included in the COMMERCIAL SCALE DESIGN section.

Biofine and GLCC collaborated on the development of the technical requirements for converting levulinic acid into valuable products and assessment of the projected markets for these products. An independent market assessment report commissioned by Biofine "A Market Plan for Commercializing Levulinic Acid Derivatives" is included in the MARKET ASSESSMENT section.

The program was successful in executing all required tasks. One aspect of the work, which was, however, not achieved was the attainment of commercial grade levulinic acid. The quality of product achieved, although useful for conversion on to derivative products, was below the commercial specification of interest to Great lakes Chemical Company. (Subsequent to completion of this program Biofine has modified the levulinic acid recovery and purification steps to allow production of commercial grade material).

The project milestone schedule is shown below. Project milestones on the project were met.

	Milestones	Date
1.	Kickoff Meeting at DOE	Oct. 23, 1995
2.	Issue Last Major Bid Package	Feb. 16, 1996
3.	Complete Cost Estimate Update	Apr. 19, 1996
4.	Start Site Work (ground breaking)	Jun. 03, 1996
5.	Close-In Building	Aug. 02, 1996
6.	Mechanical Completion	Feb. 14, 1997
7.	Complete Test-Run & Start Production	Apr. 11, 1997
8.	Interim Report (Commercial Scale Design Basis)	Sep. 12, 1997
9.	Finish Last Production Run	Mar. 31,1999

BASE
TECHNOLOGY

BASE TECHNOLOGY

Biofine, Incorporated of Waltham, Massachusetts, has developed a thermochemical process to fractionate cellulosic biomass into levulinic acid (LA), formic acid, furfural and a high energy bone dry char using high-temperature, dilute-acid hydrolysis. The primary products from the process can be converted into a wide range of high value chemicals and fuel products. The technology holds U.S. Patents 4,897,497 and 5,608,105, and has been awarded patents in several countries around the world. Typical yields in this two-stage process range from 50-70% of theoretical, resulting in about 0.5 lb LA/lb cellulose. The cellulose in the biomass is first converted into soluble sugars, which are then converted into LA ($CH_3COCH_2CH_2CO_2H$). Furfural, formic acid, and a high BTU solid fuel suitable for burning or gasification are co-products.

The process is economical even without accounting for fees for disposal of waste feedstock. Using the Biofine process the product, promising chemical building blocks, can be made with low-cost and abundant waste feedstocks. Wet feedstocks can be used without drying, thereby saving energy. Paper mill wastes appear to be ideal feedstocks for Biofine's process because they are finely divided and relatively easy to handle.

Prior to this project Biofine's process had been demonstrated at a laboratory scale with a variety of cellulosic feedstocks, including waste paper, waste wood, and agricultural residues. .This work showed that the process could utilize a wide range of feedstocks. Biofine hopes to serve the growing need for options to dispose of these biomass waste streams.

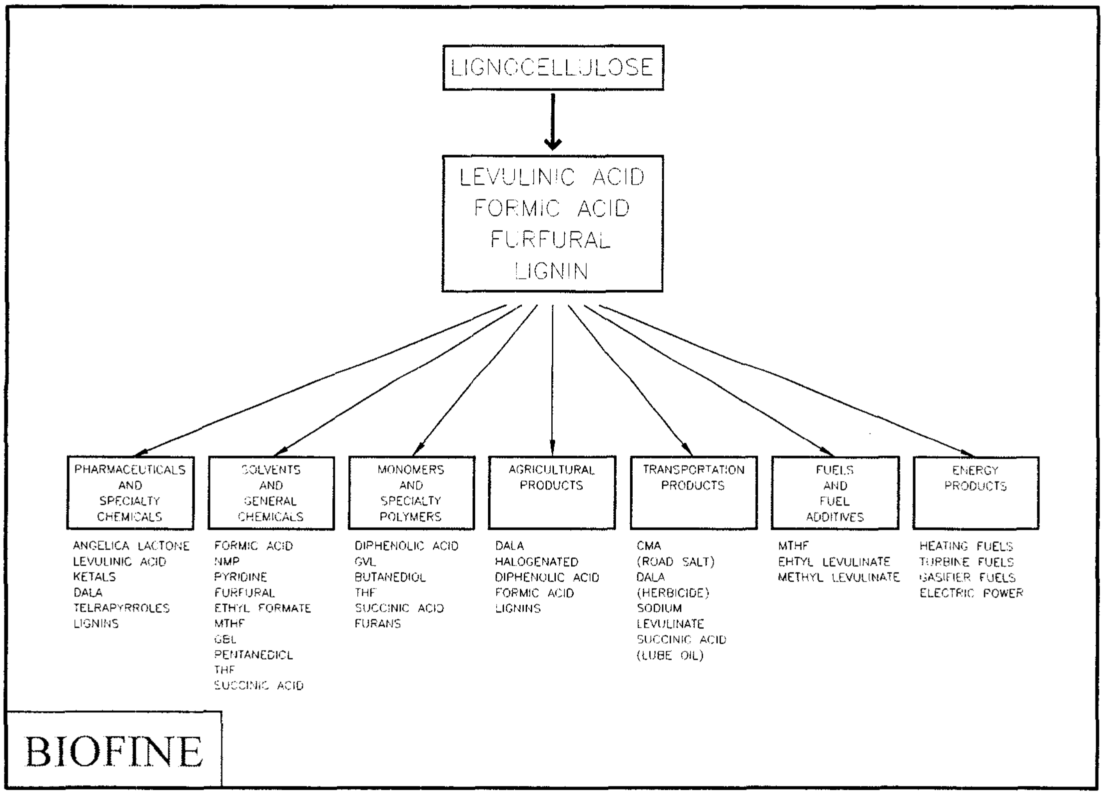
Biofine's research indicated that if the process could be successfully demonstrated it could transform major sectors of the fuels and chemicals industry and play a major role in eliminating dependence on crude oil for chemicals and fuels. The process has the potential to become the key technology in a "bio-refinery" – taking lignocellulose and fractionating it into platform or building block chemicals which would be converted on to high value end products.

LA's niche markets provide excellent small-scale opportunities; large-scale opportunities will open up as Biofine lowers the price of this highly versatile chemical intermediate. LA's present worldwide market is about two million pounds per year at a price of $4-6/lb. Full-scale commercial plants are feasible at 50 to 2000 dry ton/day of feedstock. At large scale, LA could be produced at a price which would allow it to be converted economically into such commodity chemicals as diphenolic acid, a replacement chemical for bisphenol A in polycarbonates, or acrylic acid, which sells for $0.50/lb. Eventually, Biofine hopes to build larger plants converting 1,000 to 2,000 dry ton/day of feedstock into $0.04-0.05/lb LA for conversion into economical fuel additives. The worldwide commercial market for LA and its derivatives could someday reach one trillion lbs/yr (equivalent to around 10 Quads of energy). Full-scale plant opportunities are being assessed for several locations in the U.S. and worldwide. One full-scale commercial plant using 1,000 dry ton/day of waste cellulosic feedstock could produce over 200 million lbs/yr of levulinic acid methyl ester additive for diesel fuel or a similar volume of MTHF for inclusion in fuel formulations such as the "P" Series fuel approved by DOE as an alternative gasoline.

LA derivatives are the key to marketability, and markets for such LA derivative chemicals as tetrahydrofuran, butanediol, succinic acid, and diphenolic acid exist. Established chemical conversion routes for LA are available. Biofine, in cooperation with Texaco Inc., National Renewable Energy Laboratory (NREL), Pacific Northwest National Laboratory (PNNL), and Rensselaer Polytechnic Institute (RPI) are developing market applications and production methods for other derivatives, including:

- Methyl and ethyl esters of levulinic acid as "drop-in" diesel fuel oxygenates
- Methyltetrahydofuran (MTHF) – a gasoline fuel oxygenate
- Delta aminolevulinic acid (DALA - a biodegradable herbicide)
- Diphenolic acid – A replacement for bisphenol A in polycarbonate

The diagram below indicates some of the markets into which levulinic acid derivatives could be sold:



Biomass as a Raw Material

Use of biomass rather than petroleum to manufacture chemicals has numerous advantages. Briefly, renewable biomass contributes no net CO_2 to the atmosphere, conserves fossil fuel, and leads to a secure domestic supply of feedstock capable of making a huge array of chemical products.

The Biofine process allows the possibility of using biomass as a feed to a bio-refinery. Using the process, the biomass feed can be fractionated into levulinic acid, formic acid, furfural and a hydrophobic (bone dry) char which is suitable for gasification to synthesis gas.

Benefits of the overall process

Biofine's process uses a variety of biomass resources, including wastes and, potentially, crops. Management costs for many biomass waste streams are generally rising. One promising feedstock is municipal solid waste (MSW), which is abundant and consists of 50% to 60% cellulose in the form of wood, cardboard, and paper. Sorting facilities currently separate MSW into components for recycling. Much of the cellulose fraction, however, is not recyclable, and disposal of this fraction has always been accomplished by landfilling, landspreading, composting, or incineration, all of which add little value, can be costly, and have poor public acceptance. In contrast, Biofine's process can convert a problem fraction of the waste stream into valuable products, offering a welcome alternative to landfilling. Conversion of MSW into LA at the SGF plant was demonstrated in January 1998. The Biofine process saves energy by enabling the use of wet feedstocks such as paper mill sludge, and could help paper mills achieve "zero discharge" for their wastes. All by-products from the process, including the formic acid, combustible tar, and furfural, can be sold as is. Production of derivatives could be highly integrated. For example, some by-products of LA production could be consumed as reactants for LA derivatives. One potential by-product of DALA production has been identified as a material of great interest to a major domestic chemical producer.

Benefits of the chemicals derived from LA

The esters methyl and ethyl levulinate have significant advantages as diesel fuel additives.

- They have a high flash point (above 150 Deg. F.). This allows them to be safely transported and blended. In addition, the high flash point of these esters provides a means to allow the alcohols methanol and ethanol to be safely blended into diesel formulations.

- They contain a high percentage of oxygen. The oxygen content of these esters is around 33%. Regular diesel blends incorporating methyl or ethyl levulinates have been shown to burn 35% cleaner than low sulfur "city" diesels.

- They have very high lubricity. They can be used to increase the notoriously low lubricity of low sulfur diesel formulations.

MTHF has advantages as a fuel additive. Because it is miscible with gasoline at all proportions, it can be blended at the refinery and transported by pipeline. In contrast, ethanol must be added later in the distribution process because contamination with water can cause a phase separation. MTHF can be blended in gasoline up to 70% by volume without adverse engine performance. Because the octane of MTHF is 87, equivalent to base gasoline, it could fulfill new U.S. EPA requirements for greater oxygenate levels in gasoline and at the same time lower the Reid vapor pressure of gasoline blends. A lower Reid vapor pressure means less fuel evaporates in hot weather, resulting in cleaner air. MTHF has a higher specific gravity than gasoline; mileage from MTHF-blended fuel would not decrease. Lastly, MTHF is, in effect, a high-density (liquid) storage system for hydrogen fuel. MTHF could help to establish hydrogen as a practical source of transportation fuel, a goal sought by long-range planners at U.S. DOE.

Deltaaminolevulinic acid (DALA), a promising new pesticide with enormous environmental benefits, is nontoxic and completely biodegradable. Its broad spectrum of activity rivals and possibly exceeds that of Monsanto's Roundup®. DALA is an entirely new product with unique properties; its pesticidal action is triggered by light, leaving most major crops unaffected. DALA is also used as a crop growth enhancer for agriculture in arid conditions such as desert or tundra.

Diphenolic acid (DPA) is used as a component in protective and decorative finishes. It also can be used as a substitute for bisphenol-A, the primary raw material for polycarbonates and epoxy resin. Indeed, before bisphenol-A was developed, DPA was used more widely in coating applications. Bisphenol-A has been implicated as an endocrine disrupter, which could be a serious human health issue.

Potential Strategic and Economic Impact of the overall process

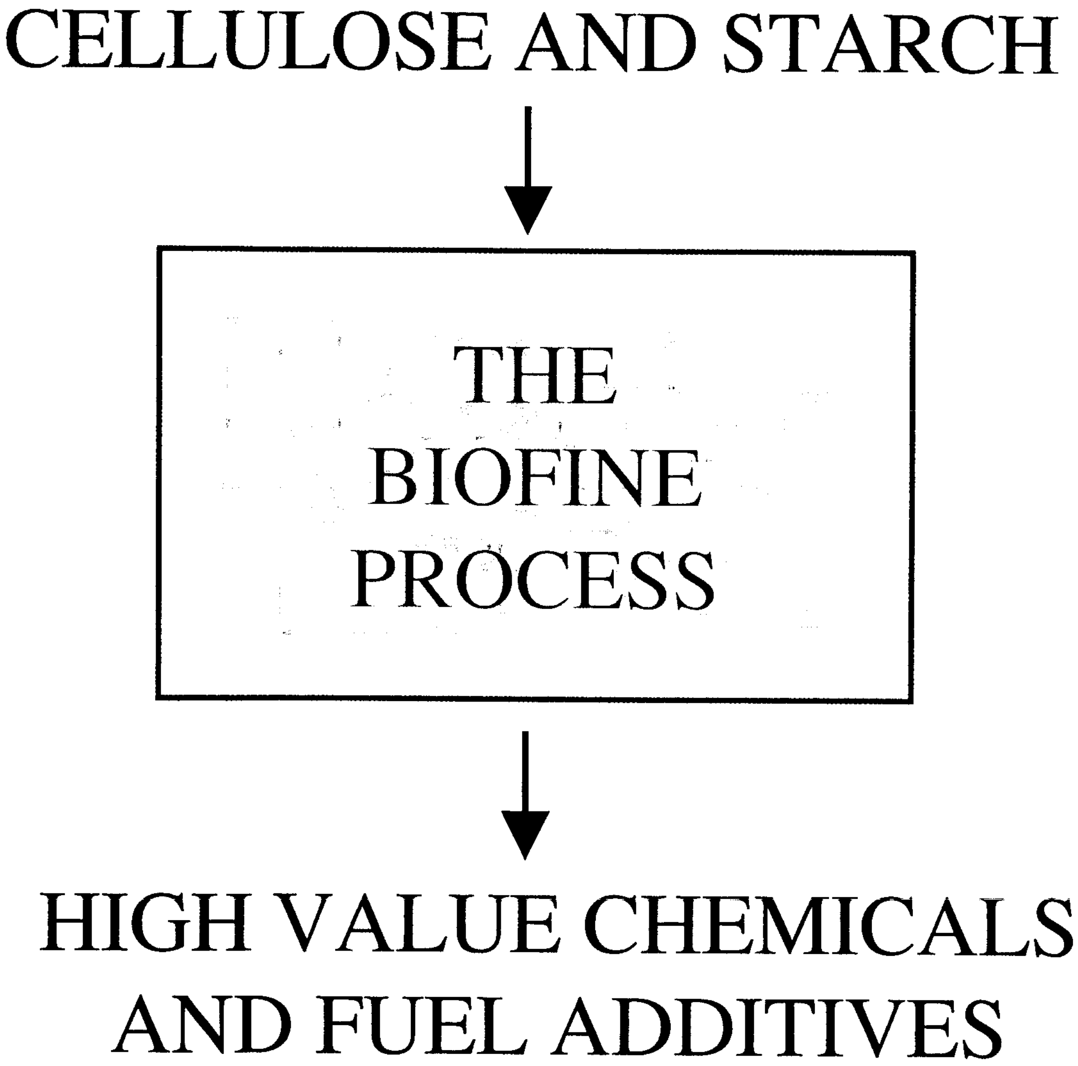
The Biofine process could have profound strategic and economic impact in that it has the potential to alleviate our national dependence on crude oil imports and create jobs in both the biomass industry itself and supporting forest, agricultural, and advanced technologies industries. Jobs could also be created in urban areas, where the most garbage is generated. A vital biomass industry also would support local economies by retaining income within the biomass generation region. In contrast, fossil fuel refining generally creates value-added products elsewhere that are then imported. Producing proven alternative-fuel extenders such as methyl and ethyl ester or MTHF would increase economic activity and energy diversity in the transportation sector, which depends on oil for 99% of its fuel needs.

Helping generators such as paper mills dispose of their waste more cheaply may give them a competitive advantage. Interestingly, growth of this technology appears to be limited by the availability of feedstocks rather than by potential product markets. Larger plants will use a variety of feedstocks, including paper-mill sludge, scrap paper and wood, and agricultural or brewery wastes. Someday, the process may use crops grown specifically as feedstocks. The process has a high degree of replicability; manufacturing facilities could be readily built anywhere there are biomass wastes or crops available, and the products are used worldwide.

Potential impacts of the chemicals and fuel products derived from LA

At large scale, esterification or hydrogenation of LA into MTHF would cost $0.04/lb, allowing it to compete economically in the fuels market. As determined by a U.S. DOE quality metrics evaluation, if MTHF replaces as little as 1% of the U.S. gasoline pool (about 100 billion gal/yr, an amount equivalent to the use of ethanol in gasoline), it could support at least 50 large manufacturing plants in the U.S. alone making one billion gallons of MTHF from more than 10 billion lbs LA. Similarly, the esters as diesel products could support at least 25 large-scale manufacturing plants.

If the Biofine technology is adopted for large scale conversion of fuels and chemicals it could represent an energy saving of at least1 quadrillion BTU per year within the foreseeable future.



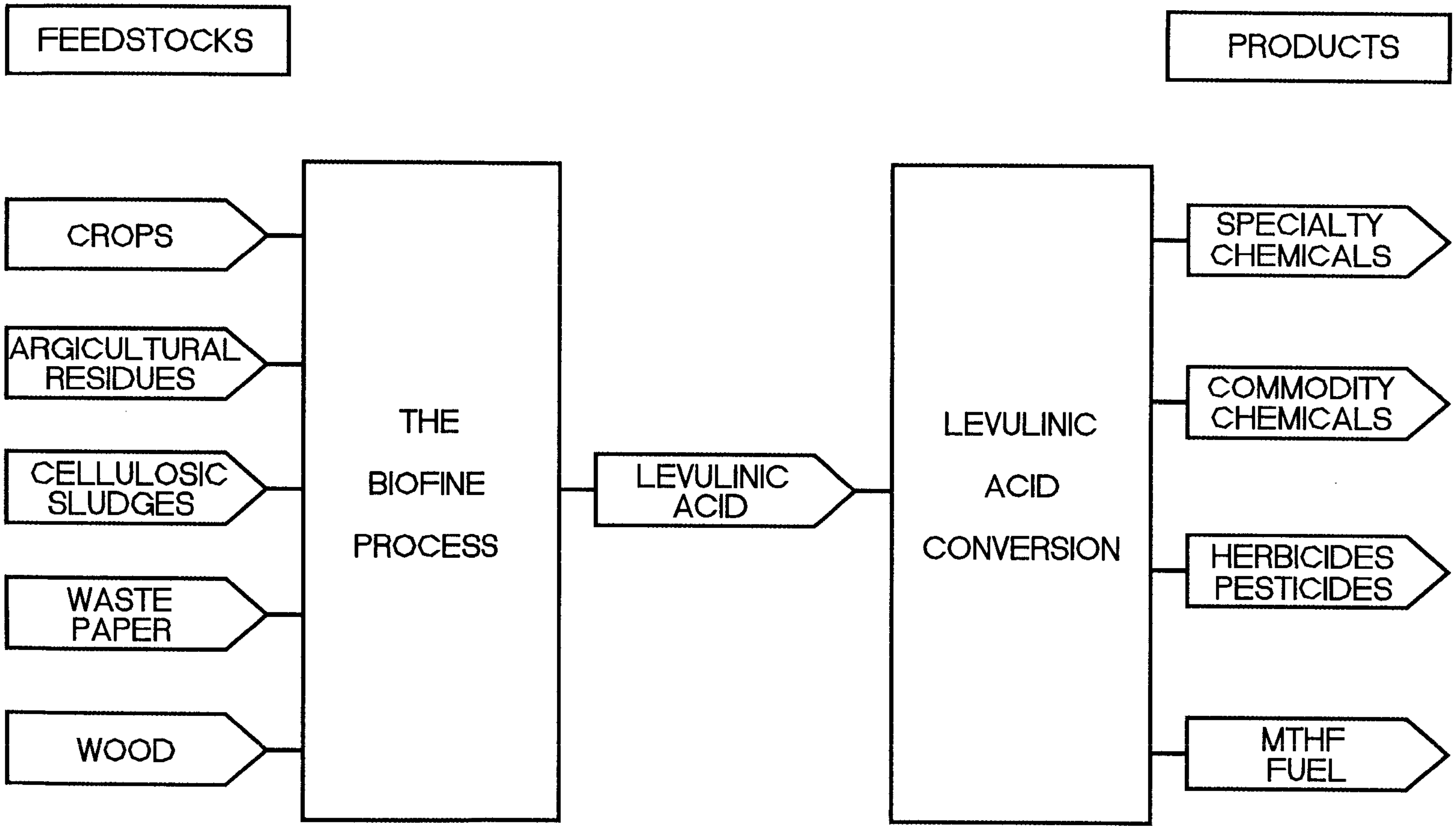
CELLULOSE AND STARCH

THE
BIOFINE
PROCESS

HIGH VALUE CHEMICALS
AND FUEL ADDITIVES

BIOFINE

THE BIOFINE PROCESS



FEEDSTOCKS

CROPS

ARGICULTURAL RESIDUES

CELLULOSIC SLUDGES

WASTE PAPER

WOOD

THE BIOFINE PROCESS

LEVULINIC ACID

LEVULINIC ACID CONVERSION

PRODUCTS

SPECIALTY CHEMICALS

COMMODITY CHEMICALS

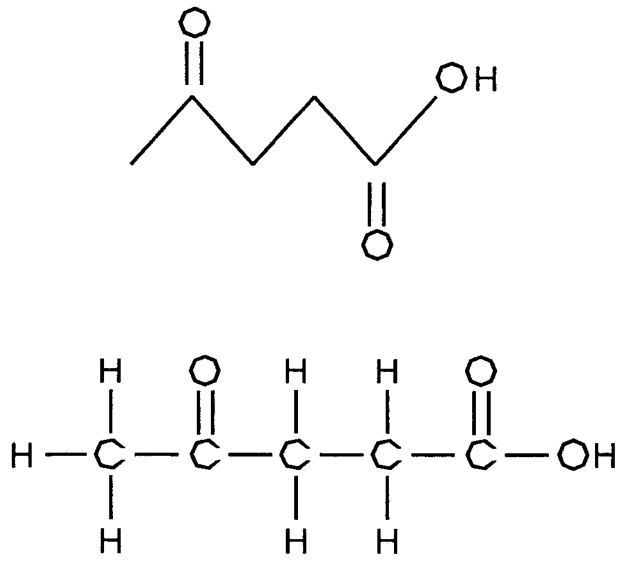
HERBICIDES PESTICIDES

MTHF FUEL

BIOFINE

LEVULINIC ACID (LVAC)

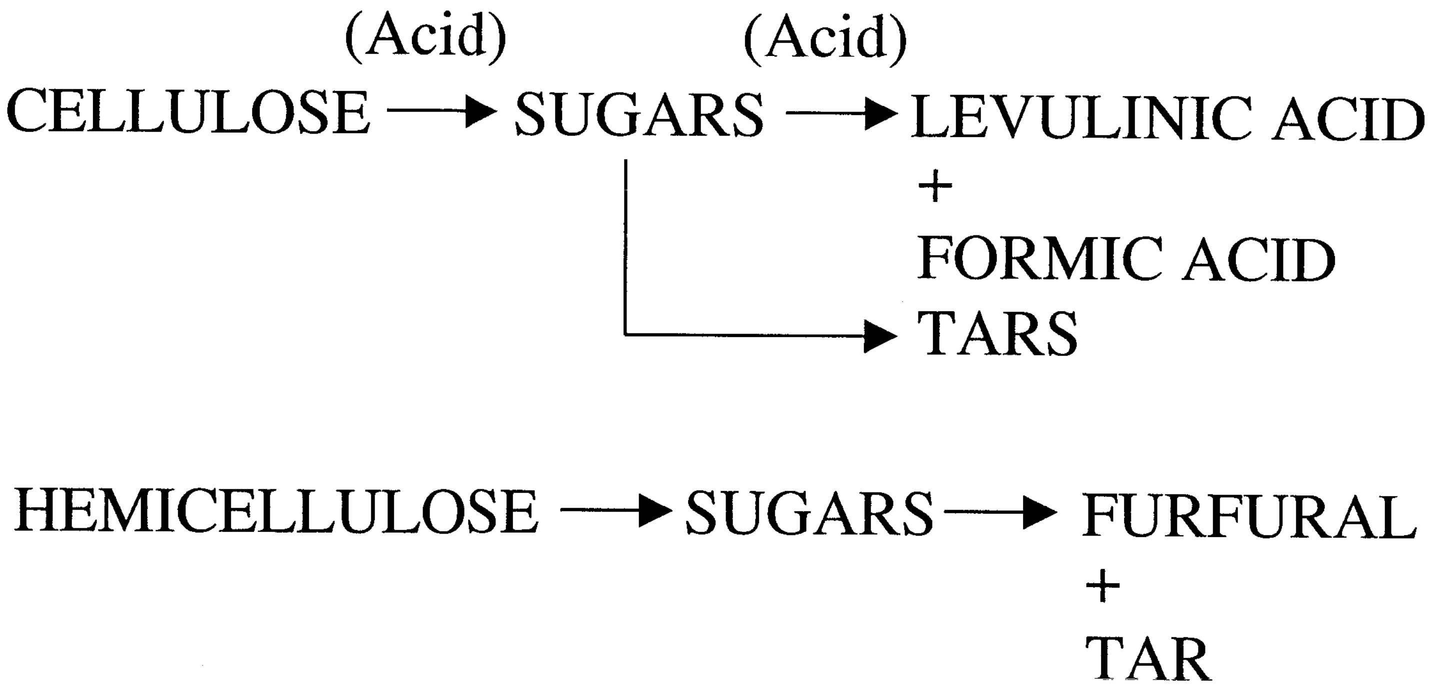
A VERSATILE PLATFORM CHEMICAL



$$\mathrm{H-\underset{H}{\overset{H}{C}}-\overset{O}{\overset{\|}{C}}-\underset{H}{\overset{H}{C}}-\underset{H}{\overset{H}{C}}-\overset{O}{\overset{\|}{C}}-OH}$$

BIOFINE

The Process Technology



$$\text{CELLULOSE} \xrightarrow{\text{(Acid)}} \text{SUGARS} \xrightarrow{\text{(Acid)}} \text{LEVULINIC ACID} + \text{FORMIC ACID}$$

$$\text{SUGARS} \longrightarrow \text{TARS}$$

$$\text{HEMICELLULOSE} \longrightarrow \text{SUGARS} \longrightarrow \text{FURFURAL} + \text{TAR}$$

BIOFINE

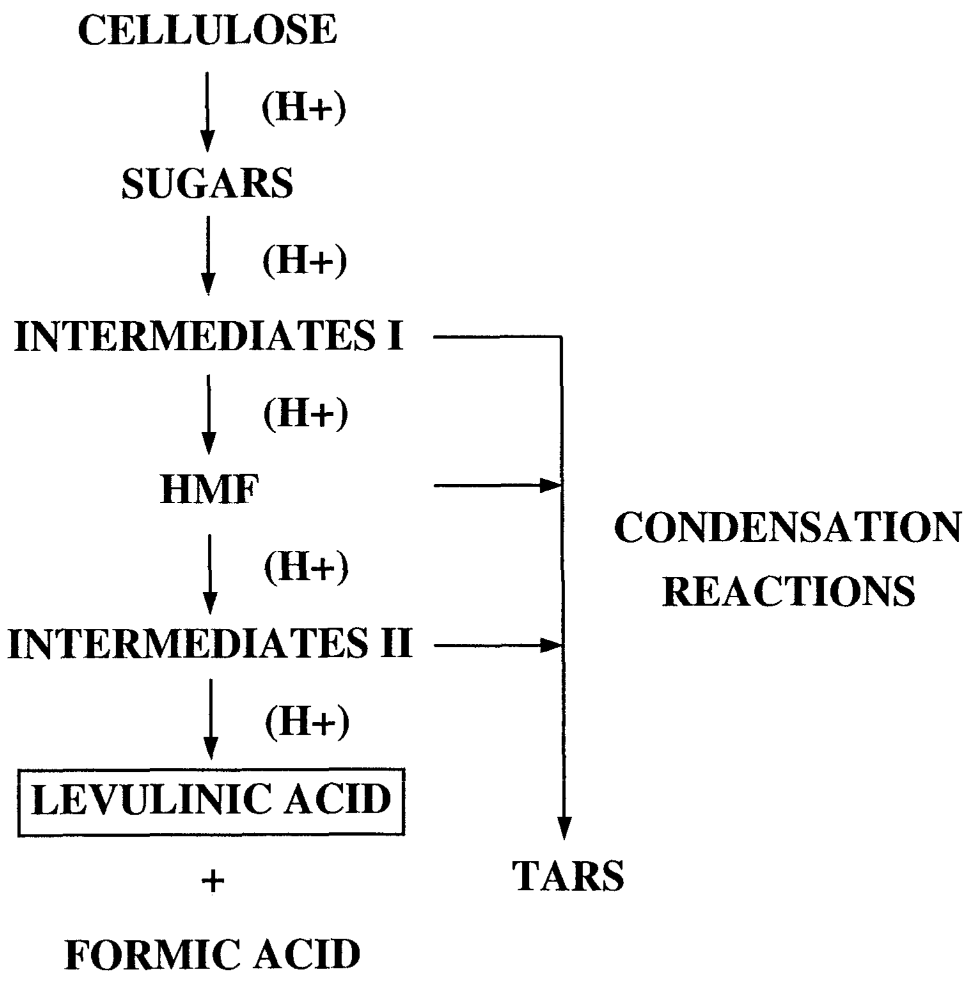
The Process Technology

MOLAR:	**LEVULINIC ACID**	**70%**
YIELDS	**FORMIC ACID**	**50%**
	FURFURAL	**80%**

MASS: ***0.5 Kg Levulinic acid***
from 1.0 Kg cellulose

BIOFINE

PROCESS CHEMISTRY



CELLULOSE

(H+)

SUGARS

(H+)

INTERMEDIATES I

(H+)

HMF

(H+)

INTERMEDIATES II

(H+)

LEVULINIC ACID

+

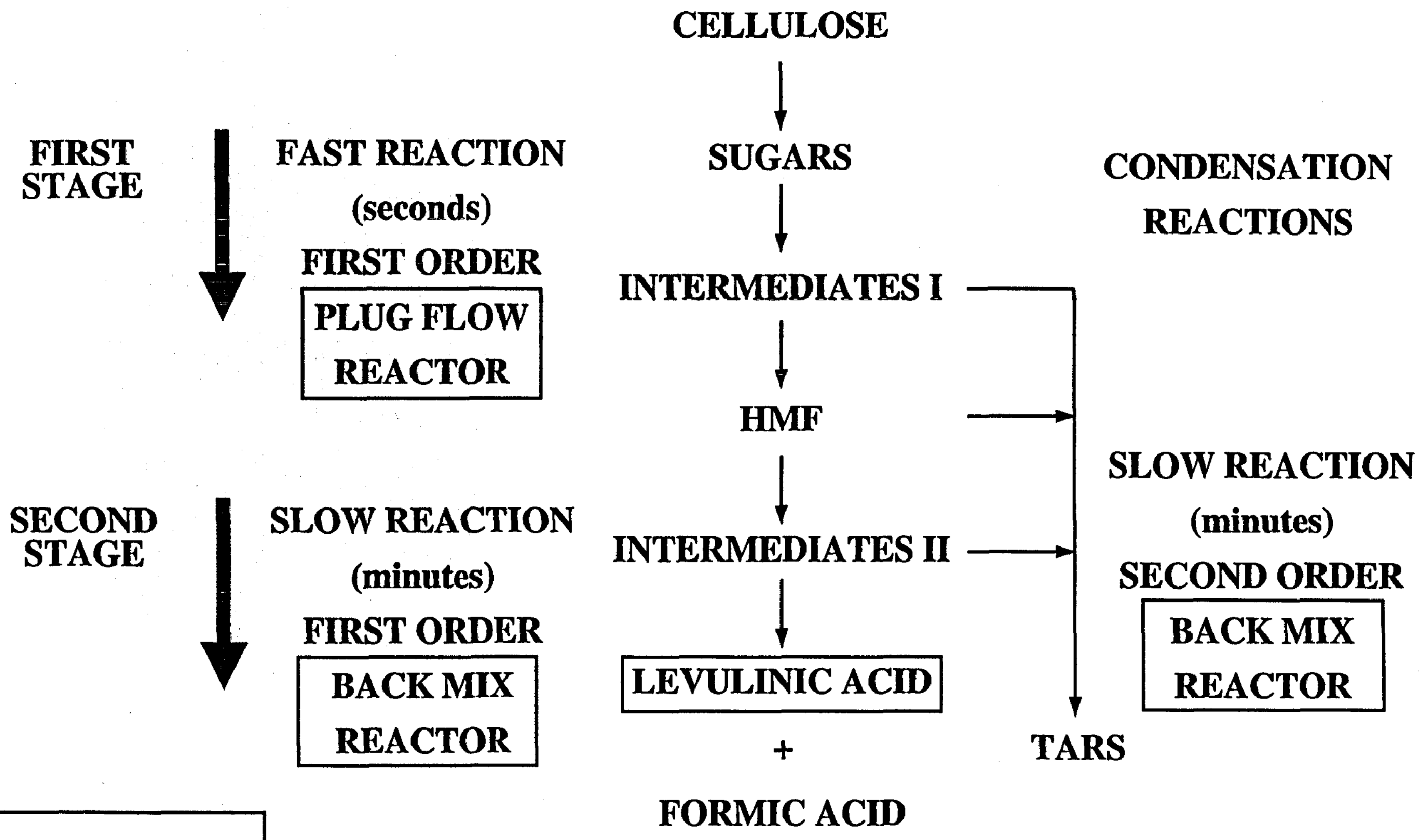
FORMIC ACID

CONDENSATION REACTIONS

TARS

BIOFINE

PROCESS CHEMISTRY & REACTOR DESIGN



FIRST STAGE

FAST REACTION
(seconds)
FIRST ORDER
PLUG FLOW REACTOR

SECOND STAGE

SLOW REACTION
(minutes)
FIRST ORDER
BACK MIX REACTOR

CELLULOSE

SUGARS

INTERMEDIATES I

HMF

INTERMEDIATES II

LEVULINIC ACID

\+

FORMIC ACID

CONDENSATION REACTIONS

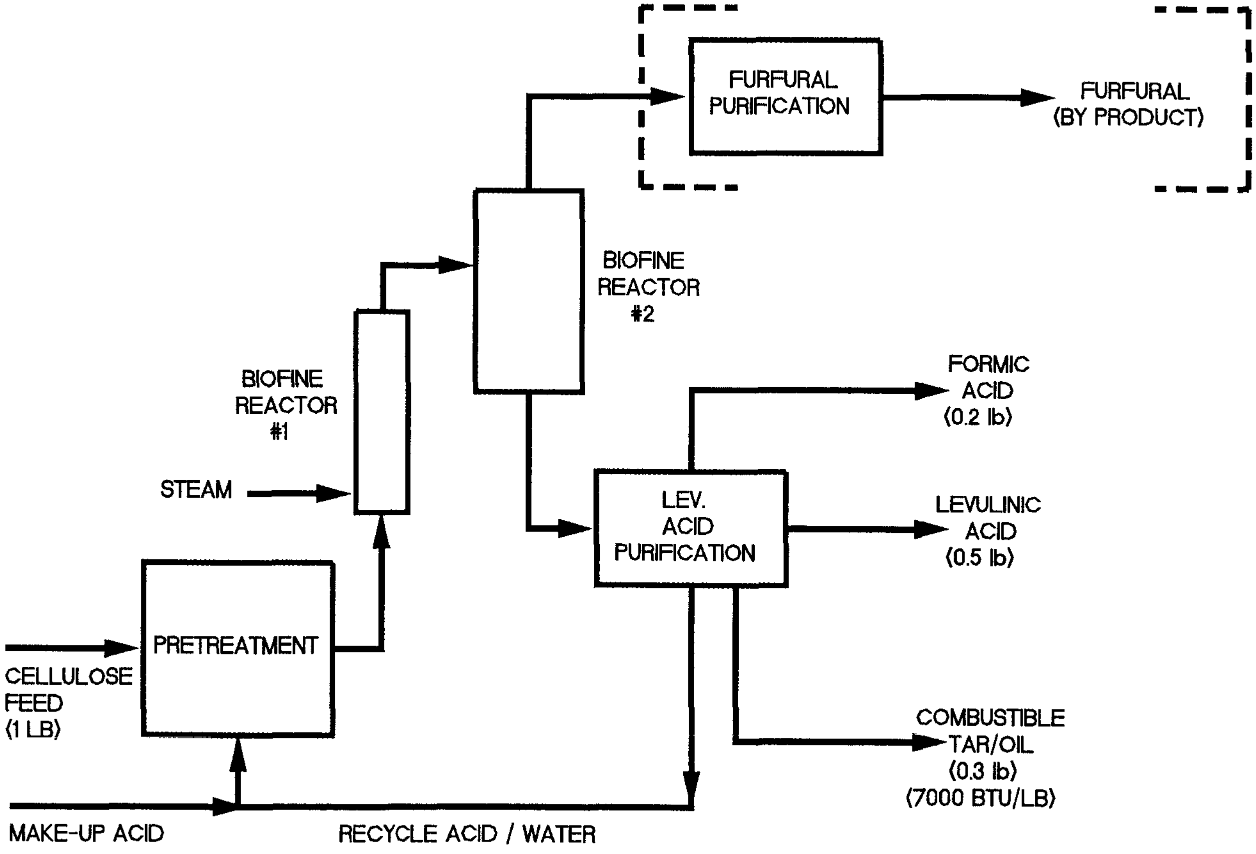
SLOW REACTION
(minutes)
SECOND ORDER
BACK MIX REACTOR

TARS

BIOFINE

THE BIOFINE PROCESS

(CELLULOSE)



FURFURAL PURIFICATION

FURFURAL (BY PRODUCT)

BIOFINE REACTOR #2

BIOFINE REACTOR #1

STEAM

FORMIC ACID (0.2 lb)

LEV. ACID PURIFICATION

LEVULINIC ACID (0.5 lb)

PRETREATMENT

CELLULOSE FEED (1 LB)

COMBUSTIBLE TAR/OIL (0.3 lb) (7000 BTU/LB)

MAKE-UP ACID

RECYCLE ACID / WATER

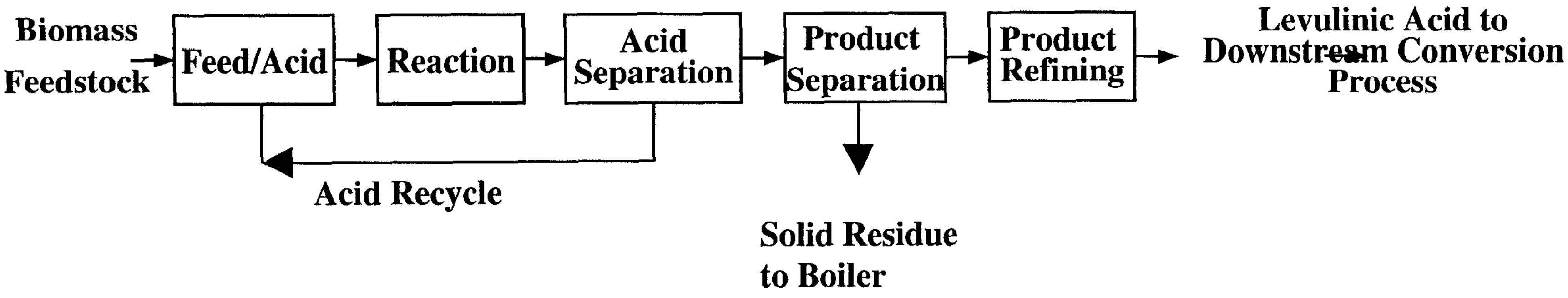
BIOFINE

The Process Technology

REACTOR SYSTEM	-	**TWO STAGE (US Pat. 4897497)**
CONFIGURATION	-	**STAGE 1 PLUG FLOW** **STAGE 2 CSTR**
TEMPERATURE	-	**STAGE 1 420F** **STAGE 2 380F**
ACIDITY (H_2SO_4)	-	**2-5% (Wgt)**
RESIDENCE TIME	-	**STAGE 1 15 SEC** **STAGE 2 12 MIN**
PRESSURE	-	**STAGE 1 444 PSIG** **STAGE 2 200 PSIG**
YIELDS	-	**LEVULINIC ACID: 70% (MOLAR)** **FURFURAL: 80% (MOLAR)** **FORMIC ACID: 50% (MOLAR)**

BIOFINE

BIOFINE LEVULINIC ACID PRODUCTION
OVERALL PROCESS SCHEME



Biomass Feedstock

Feed/Acid

Reaction

Acid Separation

Product Separation

Product Refining

Levulinic Acid to Downstream Conversion Process

Acid Recycle

Solid Residue to Boiler

BIOFINE

PROCESS BY PRODUCTS

- TAR, FORMIC ACID, FURFURAL, LIGNIN
  - TAR
    - PHYSICAL PROPERTIES (M.P. ≈ 200C)
    - CRYSTALLINE, HYDROPHOBIC
    - SPECIFIC GRAVITY ~ 1.5
    - APPEARANCE: BLACK, CRYSALLINE
    - ODOR: NO APPRECIABLE ODOR
    - SLOW TO CRYSTALIZE & PRECIPITATE TOTALLY
    - 12,000 BTU/LB
  - FORMIC ACID
    - VALUABLE BY-PRODUCT
    - GENERATED IN DILUTE SOLUTION
    - CAN BE PURIFIED OR WASTE-TREATED
  - FURFURAL
    - GENERATED FROM HEMICELLULOSE
    - VALUABLE BY-PRODUCT
    - FREE SEPARATION IN PROCESS
  - LIGNIN
    - PRODUCED AS BROWN POWDER
    - SOURCE OF OTHER CHEMICALS
    - ENERGY SOURCE (12,000 BTU/LB)

CELLULOSIC FEEDSTOCKS

RENEWABLE FEEDSTOCKS:

WOODY CROPS (FORREST MANAGEMENT)

SHORT-ROTATION SILVICULTURE (WILLOW, POPLAR, ETC.)

OTHER ENERGY CROPS (SWITCHGRASS, ETC)

POTENTIAL TO REPLACE CRUDE OIL ALMOST COMPLETELY

POTENTIAL AVAILABILITY IN U.S.	2 – 5 BILLION DRY TONS/YR
ADVANTAGES	CONTROLLED COLLECTION CLEAN, CONSISTENT HIGH LIGNIN/HEMICELL. CONTENT UNLIMITED AVAILABILITY
DISADVANTAGES	POSITIVE COST ($20/DRY TON) MODERATE CELLULOSE CONTENT SUPPLY NOT YET ESTABLISHED

BIOFINE

RECURRING FEEDSTOCKS

PAPER SLUDGE:

TOTAL AVAILABILITY (US):	3 TO 4 MILLION DRY TONS/YEAR
COST:	$5 PER TON TO NEGATIVE (TIP FEE)
CELLULOSE FRACTION:	45 TO 65 %
ADVANTAGES/DISADVANTAGES:	FINELY DIVIDED, CONSISTENT, CLEAN, MAY HAVE POSITIVE COST

BIOFINE

RECURRING FEEDSTOCKS

MUNICIPAL WASTE FIBER:

TOTAL AVAILABILITY (US):	100 MILLION DRY TONS/YEAR
COST:	NEGATIVE (TIP FEE)
CELLULOSE FRACTION:	45 TO 65 %
ADVANTAGES:	NEGATIVE COST, HIGH CELLULOSE CONTENT, STARCH,
DISADVANTAGES	VARIABLE QUALITY, NEEDS SORTING

BIOFINE

RECURRING FEEDSTOCKS

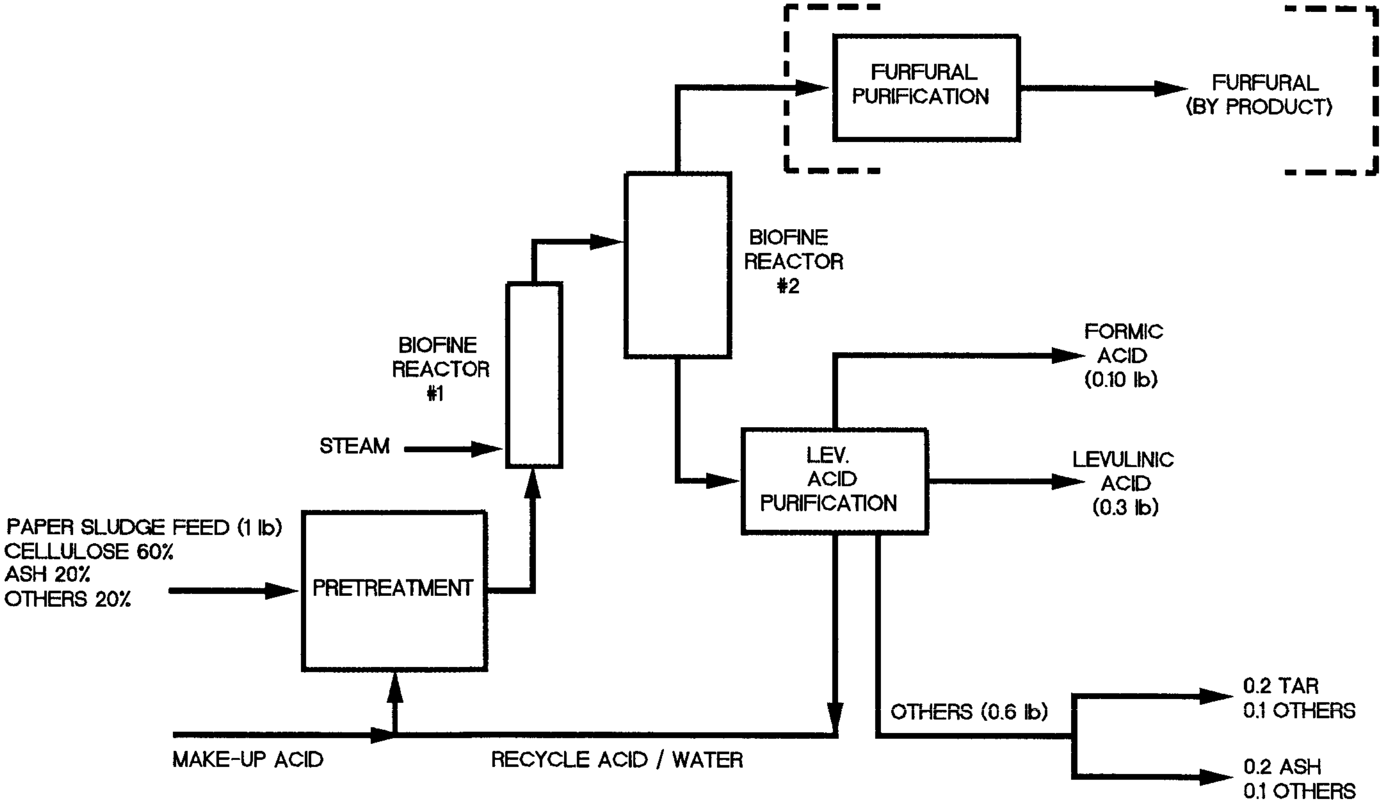
AGRICULTURAL AND LOGGING RESIDUES:

TOTAL AVAILABILITY (US):	200 MILLION DRY TONS/YEAR
COST:	$20 /TON TO NEGATIVE (TIP FEE)
CELLULOSE FRACTION:	45 TO 50 %
ADVANTAGES:	GOOD CELLULOSE CONTENT, HIGH LOCALIZED QUANTITIES, CONSISTENT, CLEAN, HEMICELLULOSE, LIGNIN
DISADVANTAGES	MAY HAVE POSITIVE COST

BIOFINE

THE BIOFINE PROCESS

(PAPER SLUDGE)



FURFURAL PURIFICATION

FURFURAL (BY PRODUCT)

BIOFINE REACTOR #2

BIOFINE REACTOR #1

STEAM

FORMIC ACID (0.10 lb)

LEV. ACID PURIFICATION

LEVULINIC ACID (0.3 lb)

PAPER SLUDGE FEED (1 lb)
CELLULOSE 60%
ASH 20%
OTHERS 20%

PRETREATMENT

OTHERS (0.6 lb)

0.2 TAR
0.1 OTHERS

0.2 ASH
0.1 OTHERS

MAKE-UP ACID

RECYCLE ACID / WATER

BIOFINE

BIOFINE INC. - PAPER SLUDGE PROCESSING CAPITAL COST VS. SCALE OF OPERATION

Installation assumed adjacent to pulp/paper mill(s)
Operating Cost includes 10% depreciation
Tipping Fees not included

Scale of Operation dry ton per day	Capital cost $mm	Production Cost $ per lb.	LVAC Scale mm lbs. per yr.
1.5	3	3.00	0.25
25	10	0.68	5
50	16	0.44	10
100	25	0.33	20
125	27	0.30	25
200	37	0.25	40
400	53	0.18	80
500	73	0.16	100
1000	90	0.13	200

**

INCL. DEPRECIATION

BIOFINE

PROCESS OPERATING COSTS

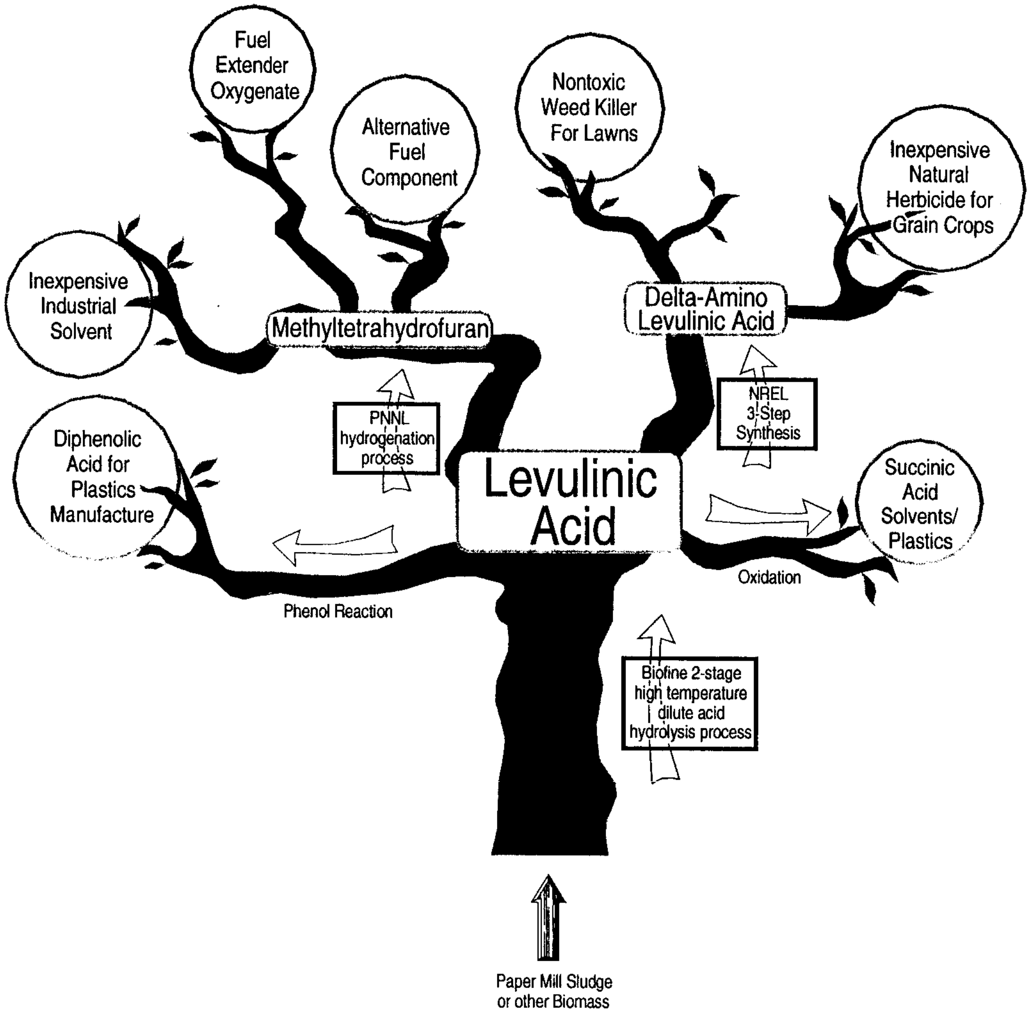
(PROCESSING 300 TO 500 TONS/DAY PAPER SLUDGE)

CENTS PER POUND LVAC

RAW MATERIALS	-	0.7
UTILITIES	-	2.5
LABOR AND MAINT'NCE	-	3.5
OVERHEADS	-	3.5
WASTE DISPOSAL	-	0.5
CREDIT: (TIP FEE)	-	(0.0)
CREDIT: (BYPRODUCTS)	-	(0.0)
DEPRECIATION	-	6.3
TOTAL	**-**	**17.0**

BIOFINE

LA Chemicals “Family Tree”



Fuel Extender Oxygenate

Alternative Fuel Component

Inexpensive Industrial Solvent

Methyltetrahydrofuran

PNNL hydrogenation process

Diphenolic Acid for Plastics Manufacture

Phenol Reaction

Nontoxic Weed Killer For Lawns

Inexpensive Natural Herbicide for Grain Crops

Delta-Amino Levulinic Acid

NREL 3-Step Synthesis

Levulinic Acid

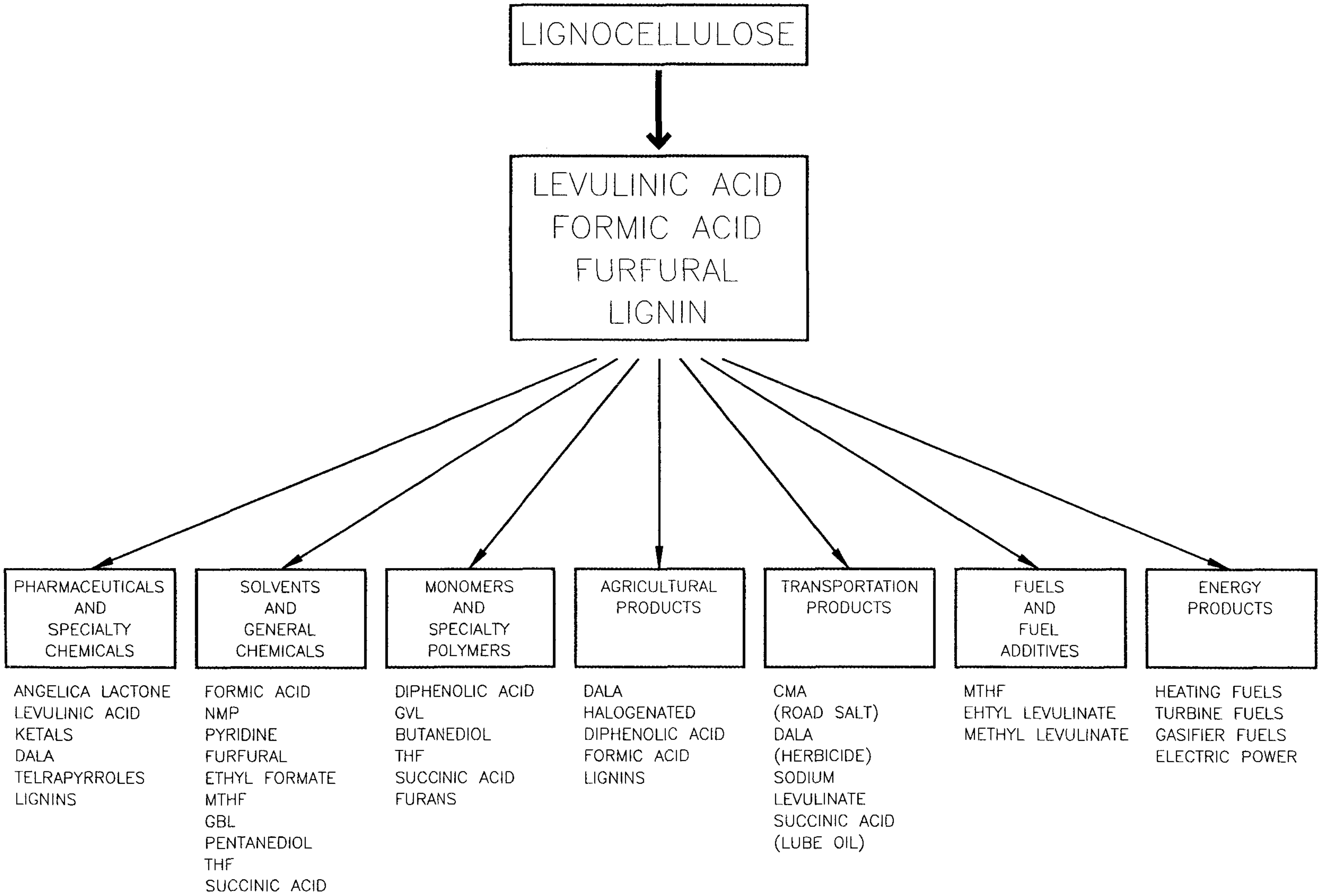
Succinic Acid Solvents/ Plastics

Oxidation

Biofine 2-stage high temperature dilute acid hydrolysis process

Paper Mill Sludge or other Biomass

BIOFINE



LIGNOCELLULOSE

LEVULINIC ACID
FORMIC ACID
FURFURAL
LIGNIN

PHARMACEUTICALS AND SPECIALTY CHEMICALS
- ANGELICA LACTONE
- LEVULINIC ACID
- KETALS
- DALA
- TELRAPYRROLES
- LIGNINS

SOLVENTS AND GENERAL CHEMICALS
- FORMIC ACID
- NMP
- PYRIDINE
- FURFURAL
- ETHYL FORMATE
- MTHF
- GBL
- PENTANEDIOL
- THF
- SUCCINIC ACID

MONOMERS AND SPECIALTY POLYMERS
- DIPHENOLIC ACID
- GVL
- BUTANEDIOL
- THF
- SUCCINIC ACID
- FURANS

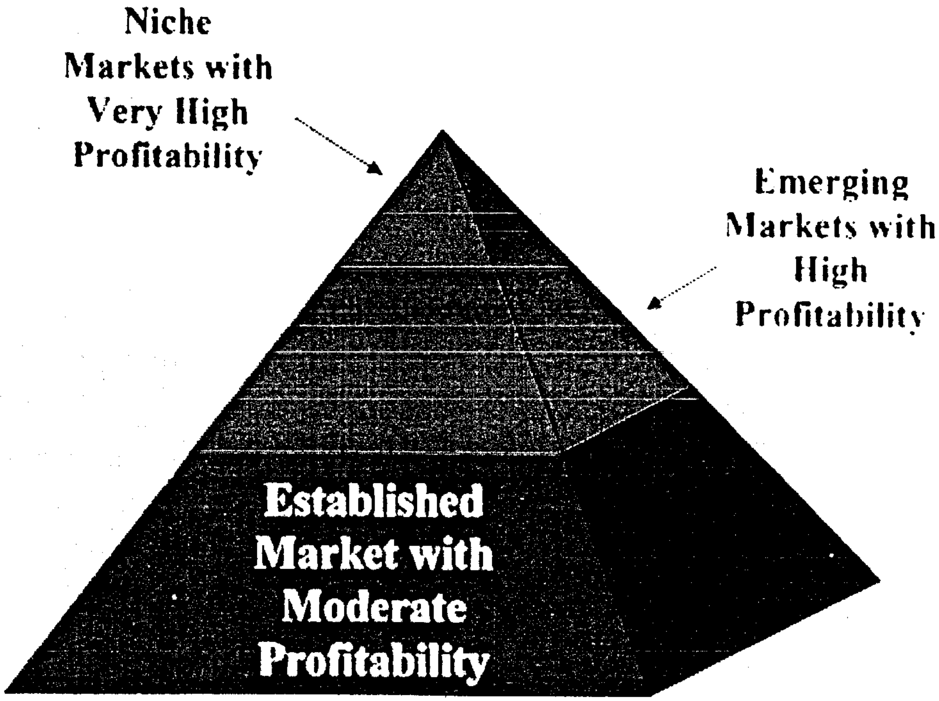
AGRICULTURAL PRODUCTS
- DALA
- HALOGENATED
- DIPHENOLIC ACID
- FORMIC ACID
- LIGNINS

TRANSPORTATION PRODUCTS
- CMA
- (ROAD SALT)
- DALA
- (HERBICIDE)
- SODIUM
- LEVULINATE
- SUCCINIC ACID
- (LUBE OIL)

FUELS AND FUEL ADDITIVES
- MTHF
- EHTYL LEVULINATE
- METHYL LEVULINATE

ENERGY PRODUCTS
- HEATING FUELS
- TURBINE FUELS
- GASIFIER FUELS
- ELECTRIC POWER

BIOFINE



Niche
Markets with
Very High
Profitability

Emerging
Markets with
High
Profitability

Established
Market with
Moderate
Profitability

EXHIBIT 6: Value Added Pyramid

MTHF
A NEW OXYGENATE FOR RFG, ALTERNATIVE GASOLINE AND GAS TURBINES

- **OCTANE VALUE -85 (LIKE GASOLINE)**
- **LOW R.V.P. (LIKE GASOLINE)**
- **HIGH M.P.G. (LIKE GASOLINE)**
- **FULLY COMPATIBLE WITH ETHANOL AND GAS**
- **REDUCES R.V.P. OF ETHANOL/GAS BLENDS**
- **20% OXYGEN CONTENT (LIKE MTBE)**
- **HYDROPHOBIC MOLECULE (LOW AFFINITY FOR WATER)**
- **PROVEN IN ROAD TESTS (UP TO 70% BY VOLUME IN GAS)**
- **APPROVED BY U.S.D.O.E. ("P SERIES")**
- **SUITABLE FOR GAS TURBINE FUEL**

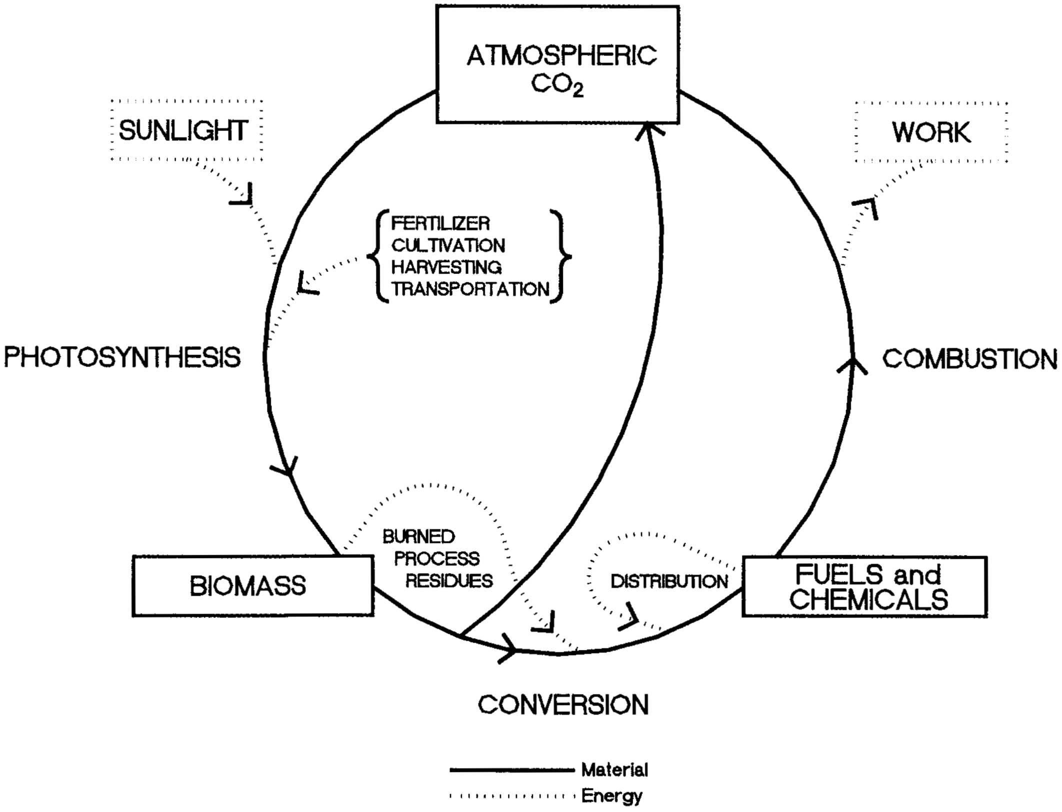
BIOFINE

ETHYL LEVULINATE
A NEW OXYGENATE FOR DIESEL FUEL, AND GAS TURBINES

- **MEETS OR EXCEEDS ASTM D-975 STANDARDS**
- **VERY HIGH LUBRICITY (REDUCED SULFUR FUEL)**
- **LOW VOLATILITY (HIGH FLASH POINT)**
- **HIGH M.P.G. (LIKE GASOLINE)**
- **FULLY COMPATIBLE WITH DIESEL**
- **REDUCES SMOKE (SOOT) SIGNIFICANTLY**
- **OXYGEN CONTENT 33%**
- **NON-TOXIC “GRAS” (PERFUME ADDITIVE)**
- **SUITABLE AS A BLENDSTOCK FOR “FAME” (esters)**
- **SUITABLE AS GAS TURBINE FUEL COMPONENT**
- **PRODUCTION COST (LARGE SCALE) <$0.50/L**

BIOFINE

Material and Energy Flows for Production and Utilization of Chemicals & Fuels from Biomass



BIOFINE

CELLULOSIC FEEDSTOCKS

RECURRING FEEDSTOCKS:

PULP AND PAPER SLUDGE,

MUNICIPAL WASTE,

CONSTRUCTION WASTES,

AGRICULTURAL/FORRESTRY RESIDUES

MANURE

AVAILABILITY IN U.S.	300 TO 400 MILLION DRY TONS/YR
ADVANTAGES	EXISTING COLLECTION SYSTEMS NEGATIVE COST (TIP FEE) HIGH CELLULOSE CONTENT
DISADVANTAGES	VARIABLE QUALITY POLITICAL INFLUENCES LOW LIGNIN AND HEMICELLSE.

DESIGN BASIS

The Biofine demonstration plant at South Glens Falls, New York is to be based on the following design basis assumptions:

Plant location

- EPIC ventures
  22 Hudson Falls Road
  South Glens Falls
  NY 12803

Feedstock composition

Dried paper mill waste with an assumed composition of:

- Moisture Content 10%

Solids consisting of (dry basis):

  - 80% cellulose
  - 5% xylan
  - 15%ash

Plant production time

40 weeks/yr
96 hours per week manufacturing product (4 x 24 hours)

Solids delivery:

Drying, delumping, and sifting
Performed off-site
Slurry feed pump
Progressive cavity pump

- Feed concentration > 5% cellulose concentration
- Flow 2.3 gpm
- Pressure 450 psig

Biofine reactor system:

Process conditions:
First stage plug flow reactor:

- 12 seconds, 230°C, 450psig

Second stage continuously stirred tank reactor:

- Residence time - 30 minutes
- Temperature - 200°C

Extraction:

Extraction using 5 stage centrifugal extractor

- Extractant - 2-methy tetrahydrafuran.

Crude levulinic acid recovery:

- Continuous packed column

Recycle acid stripping column:

- Continuous packed column

Removal of residual water:

- Spray drier

Levulinic recovery:

- Wiped film evaporator

Utilities:
High pressure steam
Electric boiler 800 #/hr at 600 psig

Medium pressure steam from EPIC ventures site
500 #/hr at 250 psig

Cooling water
Once through; 250 gpm at 80 psi

Chilled water
Package chiller for WFE*t*

Electrical supply

▪ EPIC supply to plant transformer:	575 VAC 3 phase
▪ Plant supply:	400 VAC 500 Amps

PROCESS DESCRIPTION

Solids handling

The vendor package VP-101 provides solids to the pre-hydrolysis tank T-101. VP-101 is a slave to the rate of aqueous acid addition to T-101. Once VP-101 is powered by the control start-up sequence, or enabled manually, the rate of addition of solids from the weigh hopper to T-101 is maintained in an operator set ratio against the flow of aqueous acid measured by FE-0103. The flow controller FIC-0103 totalizes the amount of recycle acid until it reached the amount calculated to give the correct concentration with a batch of solids from VP-101. VP-101 runs on demand with the weigh hopper being filled as required from the raw materials storage bin, and the raw materials storage bin being filled with sifted and screened material when this bin reaches low level.

Recycle acid from the acid stripper is returned to the pre-hydrolysis tank, T-101 via the recycle acid pump, P-410. Level in T-101 can be adjusted manually by the operator by adding water into the recycle acid stream using plant water through the rotameter, FI-0636, or flash condensate through rotameter, FI-0303. The acid concentration of this recycle stream is monitored using the pH meter AE-0102 to adjust the addition of sulphuric acid by the dosing pump, P-111. The recycle acid, process water, and make-up acid is mixed using the static mixer SM-100.

The recycle acid and the solids delivered from the solids feeder, VP-101, are mixed in T-101. The slurry is completely wetted by agitation in T-101 using the agitator, AG-101.

The slurry in T-101 is discharged to the Biofine reaction system by the positive displacement pump, P-101. The flow from P-101 is controlled by speed control on the motor of P-101. The speed control is manually adjusted to maintain the desired residence time in the plug flow Biofine reactor system, R-101.

P-101

The speed of P-101 is adjusted through SIC-0115 to give the required process flow rate. This process flow rate is provided in the plant manager run sheet for each run and will set the PFR residence time.

P-101 can only be started automatically due to the danger of equipment damage and personnel safety when starting this pump. P-101 will start once the solids handling and the high pressure reactor units are in run state. Each time P-101 is started a sequenced start up is activated by the control system to ensure the safe start of P-101 and the high pressure reactor system. To ensure that P-101 does not pump against a deadhead the temperature control valve PCV-0202 is opened prior to starting P-101.

High pressure reactor

Acidic pulp slurry from P-101 is pumped to the Biofine plug flow reactor, R-101. In the base of R-101 the slurry is mixed with 550 psig steam to heat the reaction slurry to 220□C. R-101 is maintained at 400 psig by a back pressure valve to ensure that no vapor is present in the reactor.

R-101 discharges into the second Biofine reactor, R-102. R-102 is maintained at 200□C by regulating the flashed stream's condensing rate on HE-101. HE-101 is maintained at the same pressure as R-102 and the condensing rate is changed by TCV-0212 changing the bleed of non-condensible gases from HE-101 to the vent scrubber. The condensate from HE-101 is flashed into T-201, with this flash being condensed on HE-103.

The level in R-102 is maintained by LCV-0205 to regulate the residence time in R-102. Hydrolysate from R-102 is discharged into the atmospheric flash tank, T-102. The steam flashed from the hydrolysate joins the flash from T-201 then are both condensed in the atmospheric flash condenser, HE-103. The flashed condensates from HE-101 and HE-103 are pooled in the condensate drum, T-201. The pooled condensate in T-201 is discharged to aqueous waste by the waste pump, P-203. During a run which is water deficit in the feed, the flashed condensate can be utilized for water balancing by returning the condensate to the recycle acid stream through rotameter, FE-0303.

If excess water is in the system this is removed by addition of steam to the jacket of T-102. The hydrolysate from T-102 is discharged to solids clarification by the hydrolysate pump, P-102.

Solids removal

Hydrolysate from P-102 is cooled to below 70□C by the hydrolysate cooler, HE-201 before being fed to the centrifuge, CF-201. The hydrolysate is clarified in CF-201 and the clarified liquor is discharged to the hydrolysate collection tank, T-205.

The solids removed from the hydrolysate are discharged to the solids collection screw, AG-210. The solids are continuously neutralized using caustic from P-210 which doses 50% caustic solution to neutralize the acidic waste from CF-201 (P-210 is manually set). P-210 runs anytime that P-102 and LIC-0301 are active. AG-210 discharges the neutralized solids to the solids collection tote for disposal.

The clarified hydrolysate in T-205 is pumped using the clarified hydrolysate pump, P-205, to the extraction system for recovery of the Levulinic acid.

Extraction

Clarified hydrolysate is pumped from T-205 by P-205 to the centrifugal extractor, C-301 where it is counter currently contacted with MTHF. The flow rate of hydrolysate to the column is controlled by FIC-0344 whose set point is cascaded from LIC-0326, the level control on T-215. The raffinate from the C-301 is pumped using the centrifugal force of the extractor to T-415, the reflux drum of the acid stripper, C-410. T-415 acts as a final contacting stage for the raffinate before it is sent to the acid stripper for MTHF removal.

The recycled MTHF to C-301 is cooled to optimum extraction temperature through HE-304 prior to entering C-301. The rate of MTHF flow to C-301 is controlled by FIC-0630 which operates as a ratio controller of the hydrolysate feed rate to C-301 via a cascaded set point from by FIC-0344. After extracting the Levulinic acid from the raffinate, the extract is pumped from C-301 using centrifugal force and is collected in T-301. T-301 feeds the MTHF stripper, C-510 and cascades level to flow control to maintain a constant flow to C-510. Make-up MTHF is added to T-301 as required using the drum pump, P-305.

Acid stripping

Raffinate from the extraction column, C-310, is received in the MTHF separator, T-415, which acts as a final extraction stage of the recycle acid stream. Water from VP-513 seal water which is potentially contaminated with MTHF overflows to T-415. The water rich phase for these streams is separated from the MTHF rich phase in T-415.

The aqueous phase is sent to the acid stripper, C-410, for the removal of dissolved MTHF. The interface level in T-415 is maintained by the level controller LIC-0624 which cascaded a set point to the column feed controller FIC-0641. AIC-0628 monitors the conductivity of the aqueous phase to ensure that the MTHF layer does not feed the column. A low interface signal from AAL-0628 will stop the feed to C-410 by stopping P-416, and disable the discharge of bottoms product from C-410 by disabling the output of LCV-0601.

On start-up water is brought into T-415 by opening XV-0639 to reach the minimum interface level in T-415. The stripped MTHF is removed as an azeotrope from the column and condensed in the column overhead condenser, HE-415. The azeotropic condensate from HE-415 is drained back to T-415. The MTHF rich phase from T-415 is separated by decantation and overflows a weir. The MTHF phase is removed from T-415 by the recycle MTHF pump, P-415. The rate of MTHF discharge from T-415 is set by the demand of C-301 for MTHF as controlled from C-301 by FIC-0630. LALL-0625 will stop P-415 and inform the operator that additional MTHF may be required.

The bottoms of the column are reboiled through the reboiler, HE-410. The boil-up rate is maintained by controlling the flow of steam to HE-410. The flow of steam to HE-410 is regulated by FIC-0633 which takes its set point as a ratio of the column feed rate from FIC-0641. The flow of low pressure steam (20 psig) fed to HE-410 is controlled by FCV-0633. The MTHF stripped aqueous bottoms are pumped to T-101 by P-410 with the rate of discharge controlled by LIC-0601. LIC-0601 controls the discharge rate from C-410 by a forward acting, fail close discharge valve, LCV-0601A and a reverse acting, fail open recirculation valve, LCV-0601B. If either TALL-0603, AAL-0628, or LALL-0601 is tripped the controller output is disabled and LCV-0601A is closed. The recycle acid stream is discharged and cooled to 40□C by the recycle acid cooler, HE-104, prior to return to T-101. The temperature of the stream exiting HE-104 is controlled by TIC-0612. TIC-0612 controls the flow of cooling water to HE-104 using TCV-0104.

High high level in T-101 as indicated by LAHH-0104 or LSHH-0129 will cause the recycle acid flow stopped via XV-0629. Routine blowdown of acid recycle to waste is regulated by the operator set rotameter, FI-0626, which blows down accumulated minerals in the recycle acid stream.

MTHF stripping

The MTHF extract from the liquid extraction column, C-301, is fed from T-301 to the MTHF stripper. The MTHF stripping unit consisting of T-511, T-510, HE-510, HE-511, HE-512, and C-510 is maintained at an absolute pressure of 150 mmHg by the vacuum system VP-513. Pressure is controlled by the addition of nitrogen as necessary using PIC-0710 to control PCV-0710. Extract is pumped with P-303 into the MTHF flash heater, HE-510, with the rate of feed controlled by LIC-0405, the level control on T-303. LIC-0405 is an interconnect between extraction and MTHF stripping and is only started once the MTHF stripper unit is in a available to receive status. The feed rate to HE-510 is measured using FT-0702B. The rate of steam addition to HE-510 is regulated as a ratio to FE-0702B as cascaded by FIC-0702 to the steam flow controller FIC-0746. The two phase fluid exiting HE-510 enters the flash separator, T-511. The liquid flows by gravity from T-511 to C-510 through a liquid distributor onto the distribution plate for C-510, while the flashed vapors join C-510's distillate line prior to condensing in HE-511.

The distillate from C-510 is an azeotropic mixture of MTHF/Water. The distillate is condensed in HE-511 and received into the reflux separator drum, T-510. The condensate forms a heterogeneous azeotrope which is separated into a MTHF rich phase and a water rich phase in T-510. The water rich phase is refluxed to C-510 using the reflux pump, P-512. The reflux of the aqueous phase is controlled such that the average feed composition is always on the water rich side of the azeotrope to ensure residual water in the product. To achieve this the speed of P-512 is controlled by FIC-0702 as a ratio of the feed flow, FE-0702B. The MTHF layer is returned to extraction in T-415 by P-511 with level control by LIC-0722.

There is a water deficit in T-510 as more water leaves in the MTHF separated from the distillate and in the crude LA residue that leaves the column. Process water is added to T-510 by control using LIC-0708 which intermittently opens XV-0729 to meet this water deficit and maintain the interface level in T-510.

The residue at the base of C-510 is reboiled using HE-512 which controls the temperature in the base of the column to regulate the water content of the residue. The column reboil temperature is controlled by cascading the temperature controller as a reverse acting controller to the set point of LIC-0720, the condensate level in HE-512. As the temperature measured by TIC-0724 rises the set point for the condensate blanketing in HE-512, LIC-0720, is raised to reduce heat input. The bottoms are metered to LA finishing, by the WFE feed pump, P-513 whose speed is controlled by the column level controller, LIC-0703. The column level controller also controls the LA finishing isolating valve XV-0728 which is not opened unless LIC-0703 and P-513 are running. If the reboiler temperature drops below TALL-0724, or the column pressure rises above PAH-0710 then LIC-0703 is disabled until the temperature/pressure is back within operating bounds.

Finishing

Crude Levulinic extract stripped of MTHF by the MTHF stripper, C-510, is delivered to the air stripper by P-513. If during start up the air stripper by pass option is chosen then P-513 will be feeding the WFE directly. The air heater blower, B-550 is started and the exit air temperature brought up to set point using the steam heated coils in HE-550. The air stripper temperature control is by TIC-0862 which regulates the flow of steam to HE-550 by the steam control valve TCV-0862. On start of B-550, the compressed air isolating valve XV-0869 is opened to allow compressed air to the air stripper atomizer.

The Wiped Film Evaporator, C-515, is evacuated by the plant vacuum vendor package, VP-513 and the WFE vacuum package, VP-520. The WFE vacuum is held by the non return valves in each of the discharge and inlet lines. The temperature in the WFE is regulated by the stand alone temperature controllers TIC-0861, TIC-0862, and TIC-0863 which control the temperature of the electrical tracing on three zones of C-515. The feed from the air stripper (or directly from C-510 if the air stripper is being by passed) is delivered to T-515 where residual water is flashed from the solution. The pressure in T-515 is maintained at 50 mmHg abs. by the process vacuum package, VP-513. The devolitizer feed flashes the residual moisture to VP-513. The vacuum in the WFE sucks the stripped feed from T-515 into the WFE, C-515. The devolitilized crude hydrolysate is uniformly distributed onto the heating surface by the wiped film evaporator stirrer. Pressure in C-515 is maintained at 10-25 mm Hg abs. by the vendor vacuum package, VP-520. Pressure in the WFE is maintained manually by the addition of N_2 through a bleed valve to VP-520. Levulinic acid is evaporated from the crude Levulinic acid slurry by heating on the surface of C-515. The LA distillate from C-515 is condensed on HE-515 using chilled water.

The condensed and subcooled LA product from HE-515 is pumped by P-520 to the Levulinic acid product drums. The Levulinic acid product is received into one of two drums. The WFE controller regulates the speed of P-520 to ensure that the product level does not exceed the height of the product sight glass. Product flow from the WFE is measured and totalized using FIRQ-0834.

Product is received into drums which are switched as they fill. As a drum is filled its level is displayed on the operator interface. When the drum reaches high level an alarm is generated to tell the operator to change drums. The drum change over procedure is for the operator to position a new drum and make process connections which are sensed by the proximity sensor ZS-0845 (or ZS-0846 depending on the drum being added), once this drum is in position the operator actuates the hand switch HS-0847 which opens the new discharge valve and closes the discharge valve for the current drum (XV-0841 and XV-0842). The operator then switches the manual divert valve on the WFE to allow the new product drum to fill. The product drum level sensor, LE-0835, is then swapped to the drum being filled.

Residue from C-515 is pumped to the organic waste drums by P-515. The two waste receiving drums are managed in the same manner as the product drums. The operator allows one drum to fill, and then switches product to the other receiving drum using the hand switched product valve (switching XV-0839 and XV-0840) and the diverter valve. The level sensor which indicates the level in the waste drums is LE-0812.

Effluent treatment, T-701

Effluent is treated by neutralization in T-701. Effluent from process wastes is received in T-701 and pH adjusted to 7.0 by the dosing pump, P-710 while being agitated using AG-701. Effluent is well mixed in the first compartment of T-701 before continuously overflowing to the second partition of T-701. In the second compartment of T-701 the effluent is further trimmed to ensure the pH is 7.0. The second compartment in T-701 is agitated using a continuous recycle from the discharge pump, P-704. The treated effluent is discharged using P-704 which discontinuously discharges effluent by level control. The effluent treatment process starts as a unit with the utilities start from the process overview screen and runs continuously.

Organic hold tank, T-703

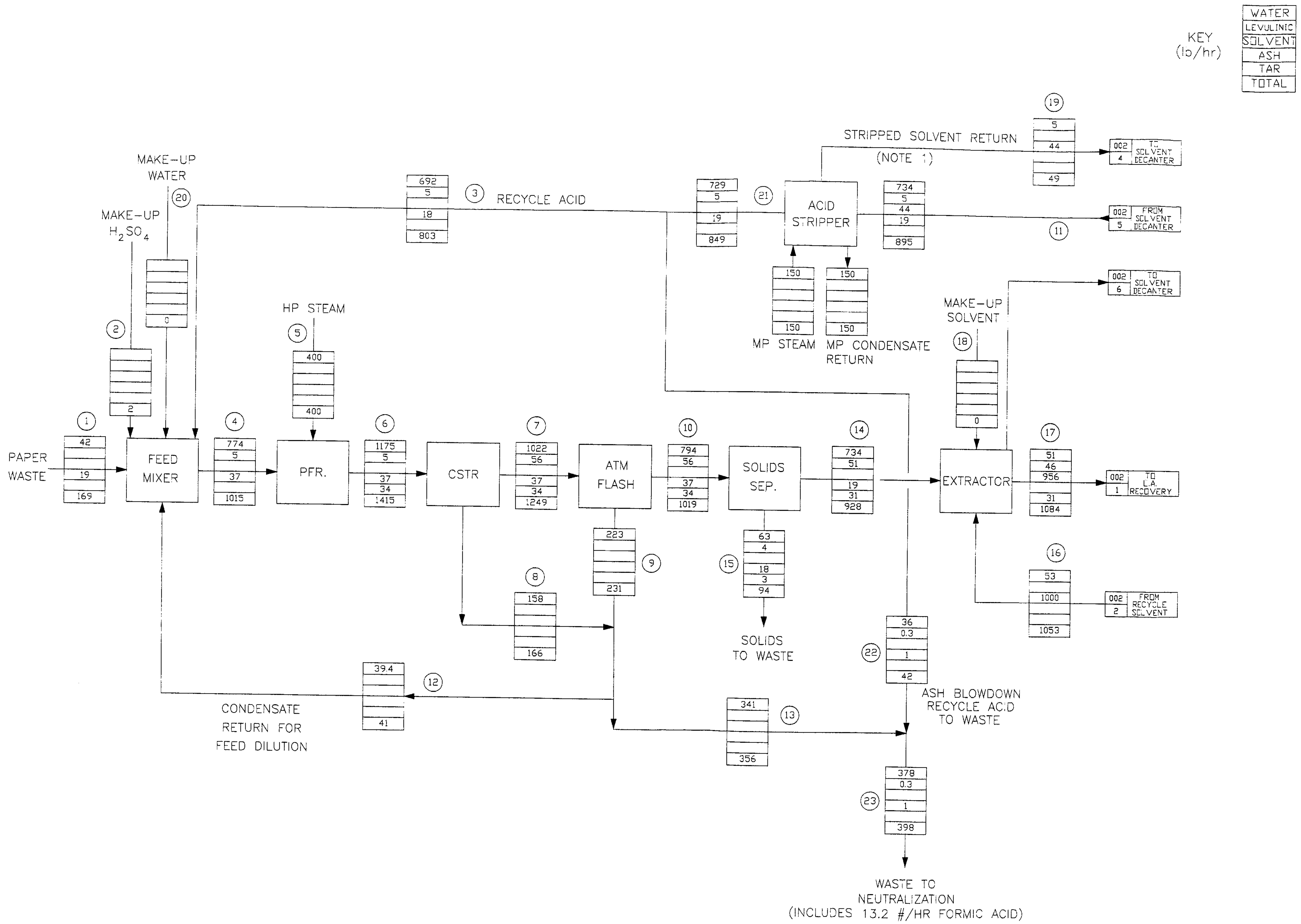
The organic hold tank, T-703, and the organic hold pump are used to hold organic solutions from process prior to reprocessing or discharge to organic waste. Organic fluids can be sent to T-703 from organic process areas. P-703 discharges from T-703 to either organic waste for drumming or to T-301 for reprocessing. The operation of T-703 and P-703 is totally manual.

Inorganic dump tank, T-702

The inorganic dump tank, T-702, and its associated discharge pump, P-702 are used to handle dumped aqueous process liquors. The contents of T-702 can be discharged to either T-102 for preprocessing or to effluent for neutralization and discharge. The operation of T-702 and P-702 is totally manual.

Relief tank, T-704

T-704 is a containment tank used to retain the contents of aqueous pressure relief lines in the process. The contents of T-704 are removed by the use of a mobile transfer pump.



BioMetics

PROCESS ENGINEERING · VALIDATION · ARCHITECTURE · GMP CONSULTING

300 BEAR HILL ROAD, WALTHAM MA 02154
781 · 684-8331 FAX 781 · 684-8335

NOTES:

1. SOLVENT FLOWS SHOWN FROM STRIPPERS ARE NETT FLOWS, REFLUX STREAM SEPARATION & RETURN ARE NOT SHOWN.

COMPOSITION:
25% MOISTURE
SOLIDS COMPOSITION
-DRY BASIS
80% CELLULOSE
15% ASH
5% HEMICELLULOSE

TOTAL CELLULOSE INPUT
= 101.5 #/HR

REV.	DATE	DESCRIPTION	CHECKED BY	APPV'D BY
4	12/12/97	MPS CONSUMPTION EDIT	C.S.	
B	5/20/96	GENERAL	C.S.	
A	4/02/96	GENERAL	C.S.	

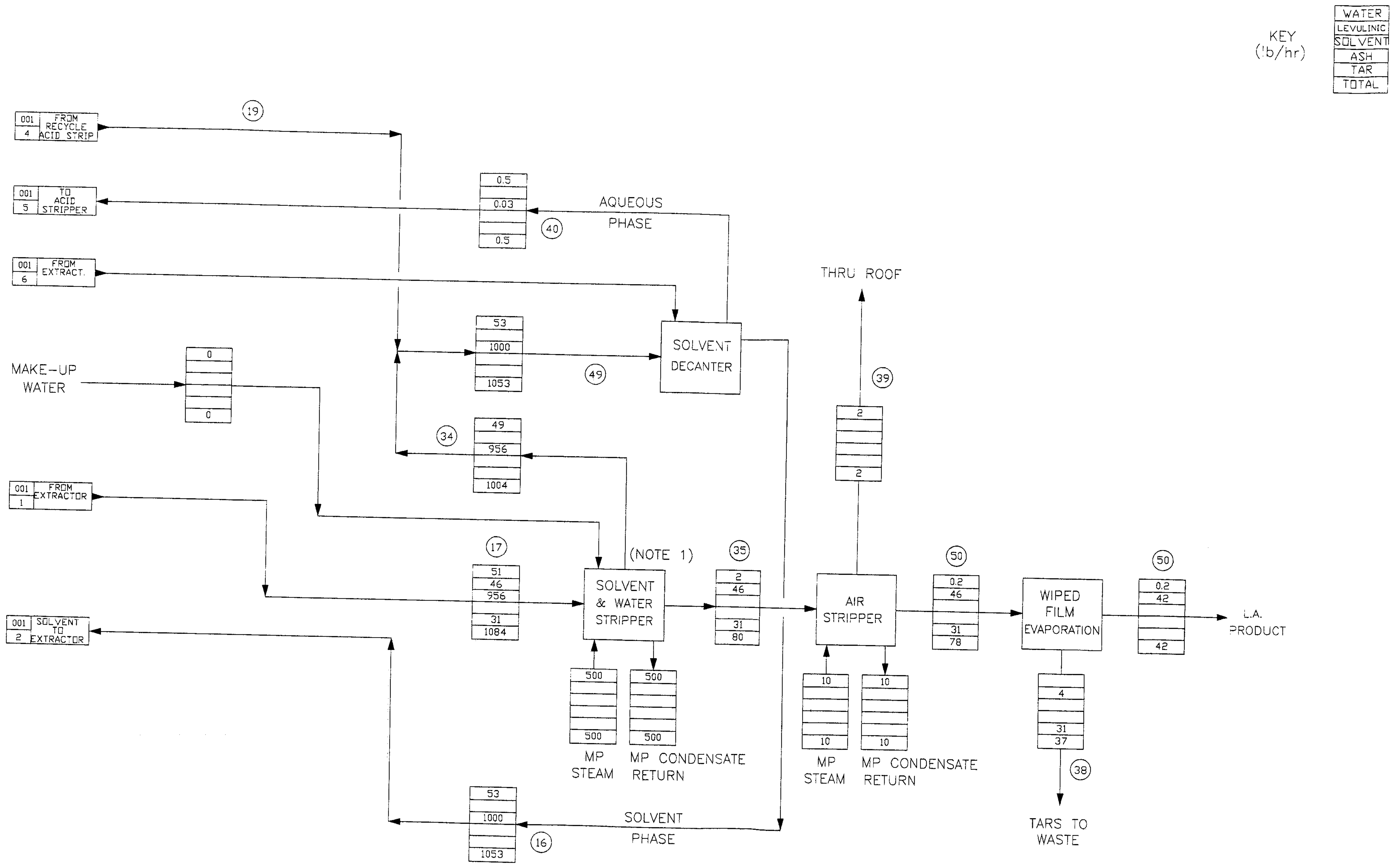
BIOFINE

PROJECT NO: 096 DATE: 1/09/96
DRAWN BY: E.B. CHECKED BY: C.S.
SCALE: N.T.S.

TITLE:
PROCESS MATERIAL BALANCE

NUMBER:
B001

B001.DWG



BioMetics
PROCESS ENGINEERING • VALIDATION • ARCHITECTURE • GMP CONSULTING
300 BEAR HILL ROAD, WALTHAM MA 02154
781 · 684-8331 FAX 781 · 684-8335

NOTES:

1. SOLVENT FLOWS SHOWN FROM STRIPPERS ARE NETT FLOWS, REFLUX STREAM SEPARATION & RETURN ARE NOT SHOWN.

REV.	DATE	DESCRIPTION	CHECKED BY	APPV'D BY
4	12/12/97	MPS CONSUMPTION EDIT	C.S.	
B	5/20/96	GENERAL	C.S.	
A	4/02/96	GENERAL	C.S.	

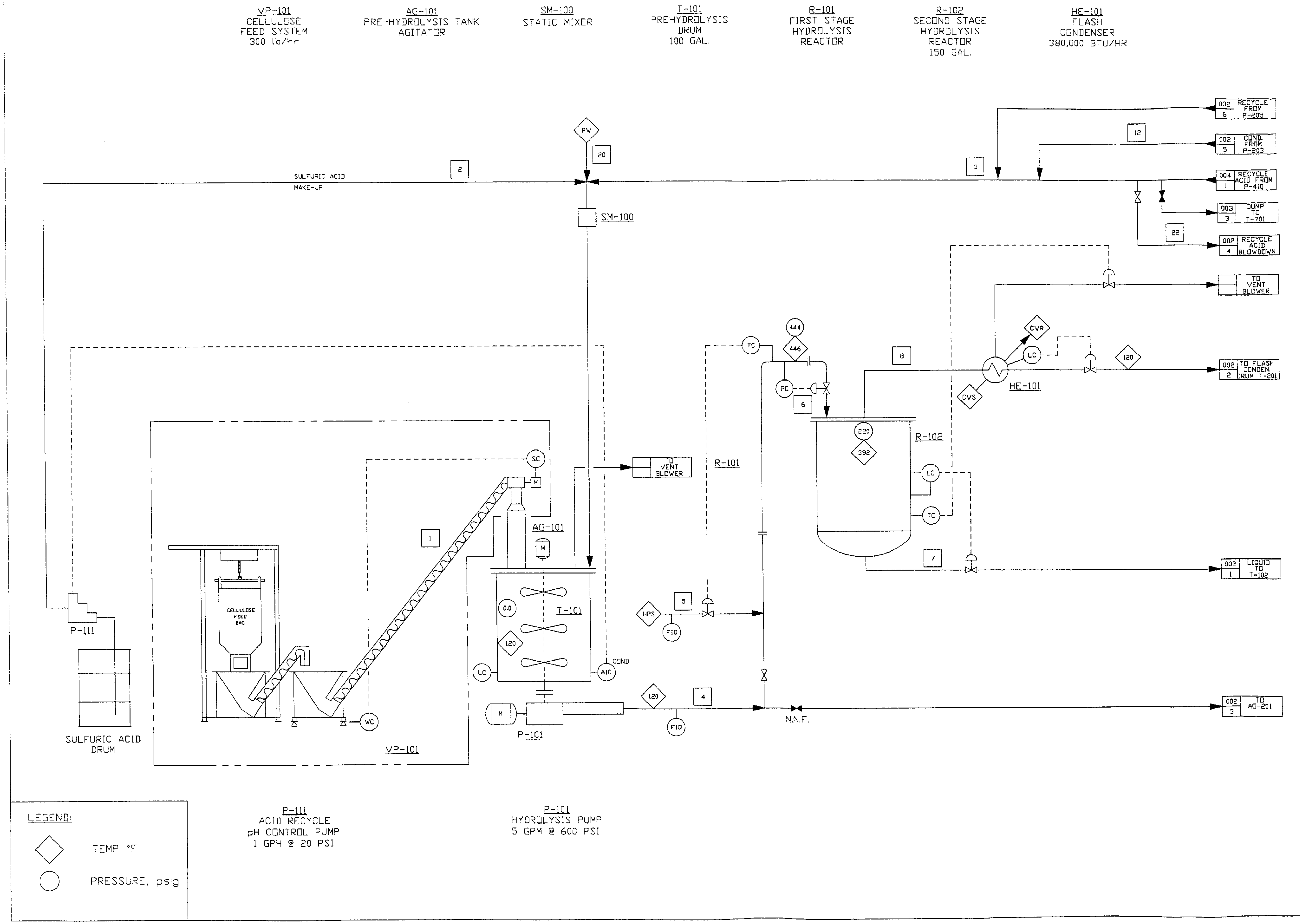
BIOFINE

PROJECT NO: 096 DATE: 1/15/96
DRAWN BY: E.B. CHECKED BY: C.S.
SCALE: N.T.S.

TITLE:
PROCESS MASS BALANCE LEVULINIC ACID RECOVERY

NUMBER:
B002

B002.DWG



BioMetics

PROCESS ENGINEERING · VALIDATION · ARCHITECTURE · GMP CONSULTING

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. STREAM NUMBERS IN ☐ REFER TO THE PROCESS MASS BALANCE. THE BASIS IS GIVEN IN DWG. PROCESS MATERIAL BALANCE - 001. SENSITIVITIES AROUND THIS ARE GIVEN IN: G:\DOCS\096-DOC\PFDBASIS.CRS.

BIOFINE

JOB NUMBER: 96
DRAWN BY: E.B.
CHECKED BY: B.J.P.
DATE: 12/15/95
REVISED: 2/29/96
4/17/96
7/16/96
1/20/97
6/26/97
SCALE: N.T.S.

SOLIDS DELIVERY AND ACID HYDROLYSIS

PFD1

PFD/PFD1.DWG

T-102
ATMOSPHERIC
FLASH DRUM
25 GAL.

HE-103
ATMOS.FLASH
CONDENSER
695,000 BTU/HR

T-201
FLASH
CONDENSATE
DRUM
25 GAL.

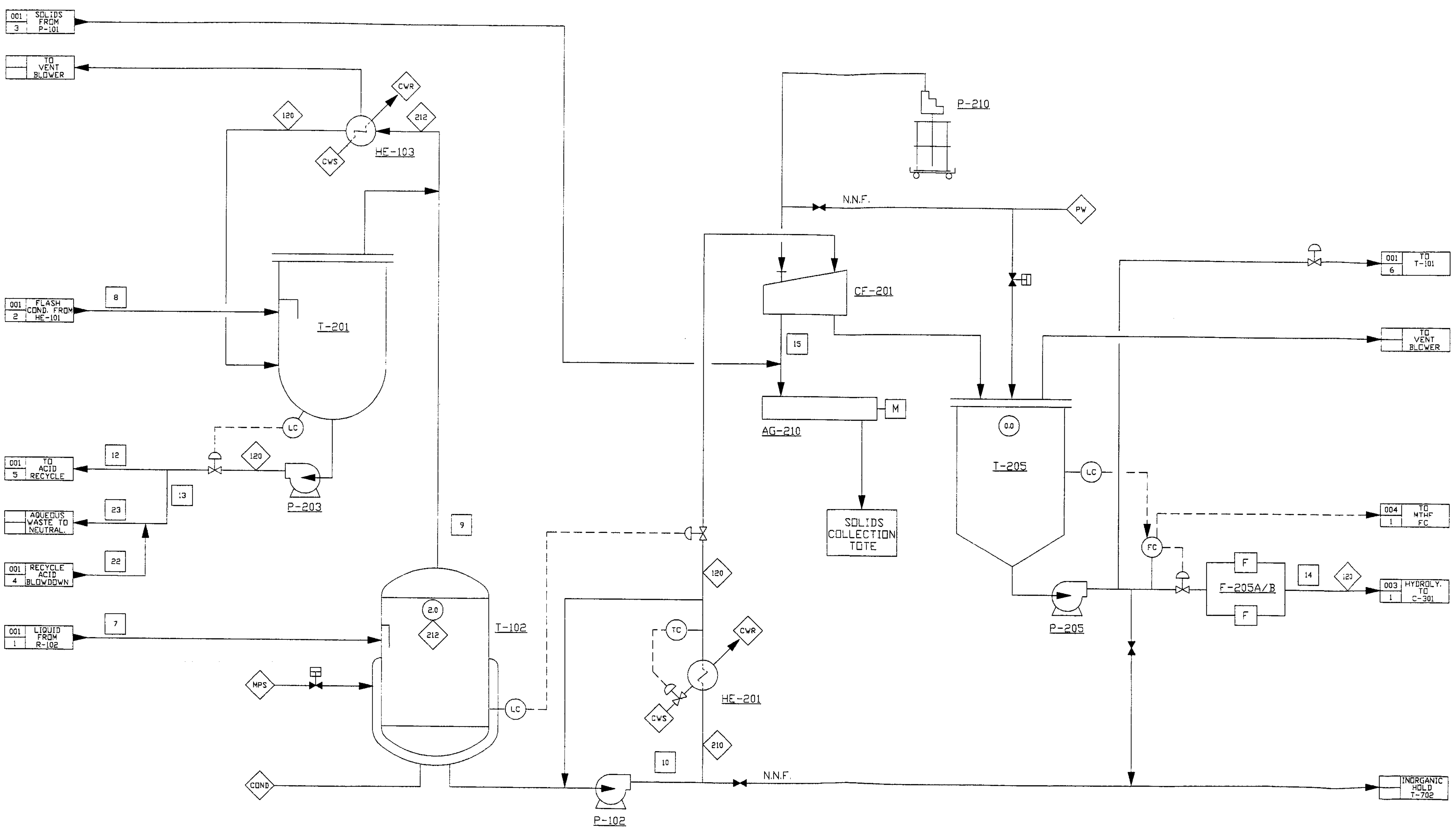
HE-201
HYDROLYSATE
COOLER
175,000 BTU/HR

CF-201
DECANTER
CENTRIFUGE
PACKAGE
1800 LB/HR

AG-210
WASTE SOLIDS
MIXER

T-205
HYDROLYSATE
COLLECTION
TANK
230 GAL.

F-205A/B
POLISHING
FILTERS
5 MICRON



LEGEND:

◇ TEMP °F
○ PRESSURE, psig

P-203
FLASH DRUM
PUMP
2 GPM @ 25 PSI

P-102
HYDROLYSATE PUMP
5 GPM @ 23 PSI

P-210
SOLID WASTE pH
ADJUSTMENT PUMP
1 GPM @ 10 PSI

P-205
CLARIFIED
HYDROLYSATE
PUMP
4 GPM @ 23 PSI

BioMetics
PROCESS ENGINEERING · VALIDATION · ARCHITECTURE · GMP CONSULTING
300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:
1. WASTE SOLIDS TO BE COLLECTED AND TRANSPORTED IN REUSABLE TOTES.

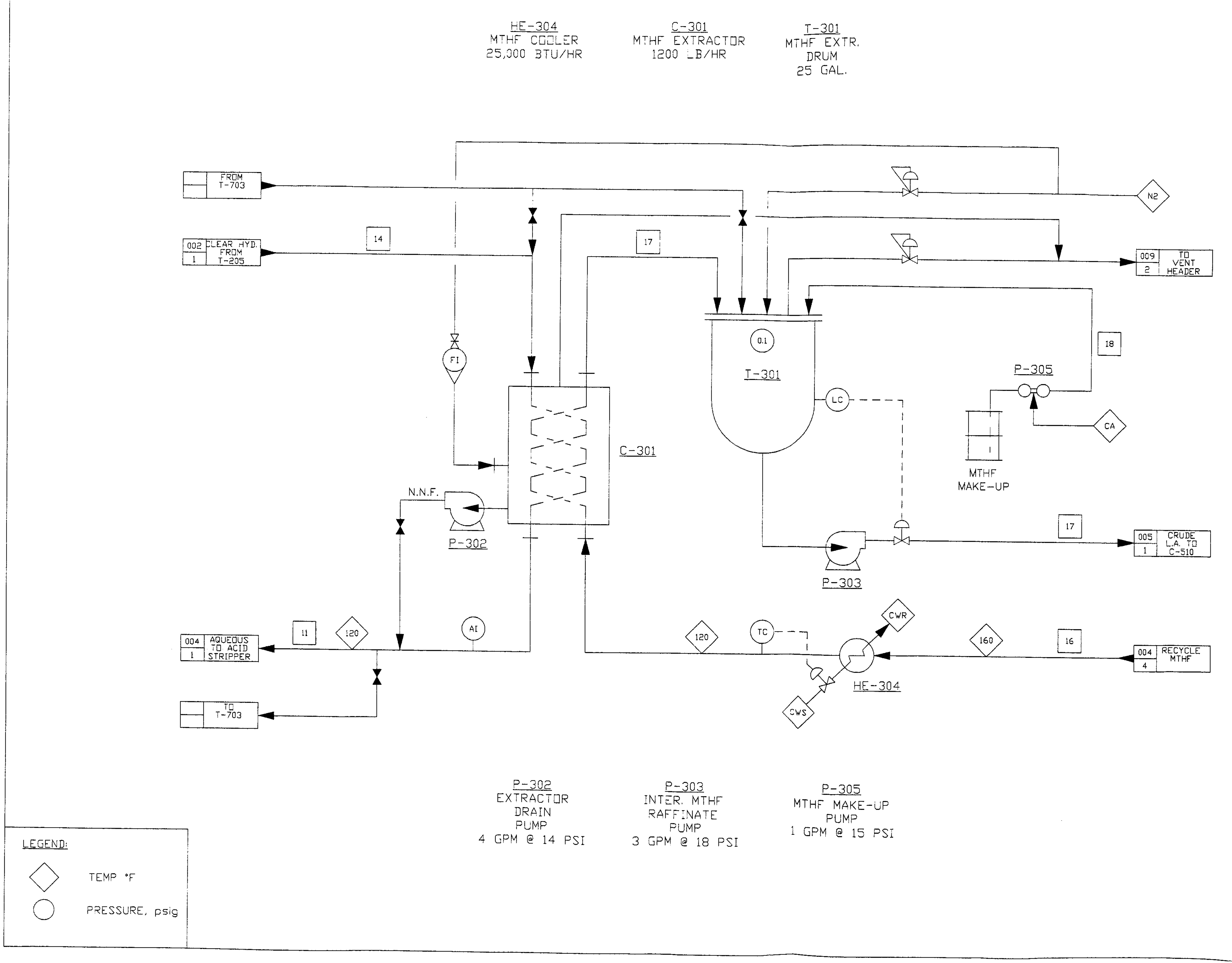
BIOFINE

JOB NUMBER: 96
DRAWN BY: E.B.
CHECKED BY: C.S.
DATE: 1/15/96
REVISED: 2/29/96
4/17/96
7/16/96
1/20/97
6/26/97
SCALE: N.T.S.

FILTRATION
AND
CONCENTRATION

PFD2

REVC/PFD-2.DWG



BioMetics

PROCESS ENGINEERING • VALIDATION • ARCHITECTURE • GMP CONSULTING

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

BIOFINE

JOB NUMBER: 96
DRAWN BY: E.B.
CHECKED BY: C.S.
DATE: 1/16/96
REVISED: 4/18/96
7/16/96
1/20/97
6/26/97

SCALE: N.T.S.

EXTRACTION AND SOLVENT STRIPPING

PFD3

REVC/PFD-3.DWG

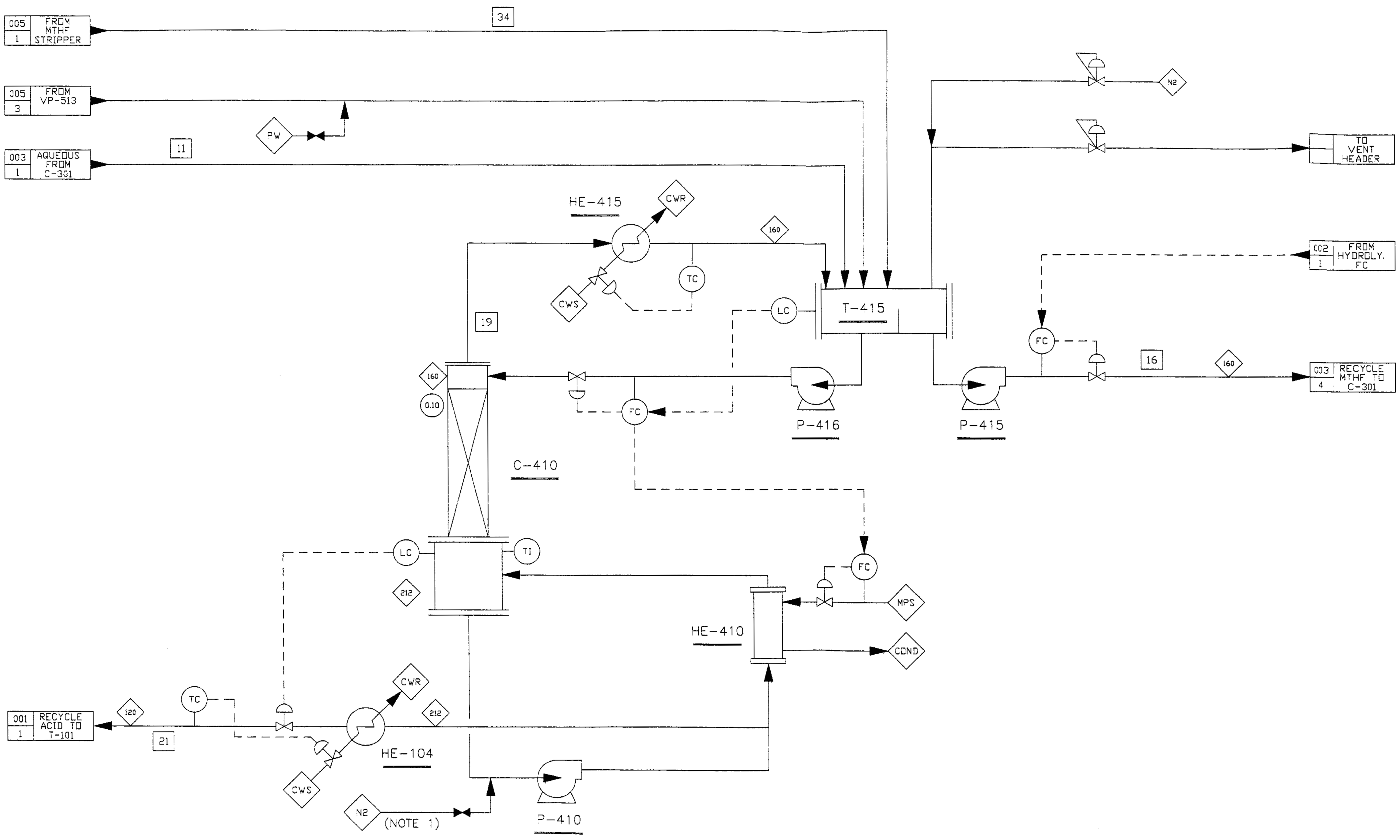
C-410
RECYCLE ACID
STRIPPER
1100 LB/HR

HE-410
RECYCLE ACID
REBOILER
250,000 BTU/HR

HE-415
AZEOTROPE
CONDENSER
94,000 BTU/HR

T-415
MTHF PHASE
SEPARATOR
275 GAL.

HE-104
RECYCLE ACID
COOLER
150,000 BTU/HR



P-410
RECYCLE ACID
PUMP
5 GPM @ 33 PSI

P-415
RECYCLE MTHF
PUMP
3 GPM @ 21 PSI

P-416
RECYCLE AQUEOUS
PUMP
5 GPM @ 33 PSI

LEGEND:

◇ TEMP °F
○ PRESSURE, psig

BioMetics
PROCESS ENGINEERING · VALIDATION · ARCHITECTURE · GMP CONSULTING
300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. NITROGEN FOR START-UP PURGING ONLY.

BIOFINE

JOB NUMBER: 96
DRAWN BY: E.B.
CHECKED BY: C.S.
DATE: 4/02/96
REVISED: 7/16/96
1/20/97
6/26/97
SCALE: N.T.S.

MTHF STRIPPING AND BLOWDOWN

PFD4

REVC/PFD-4.DWG

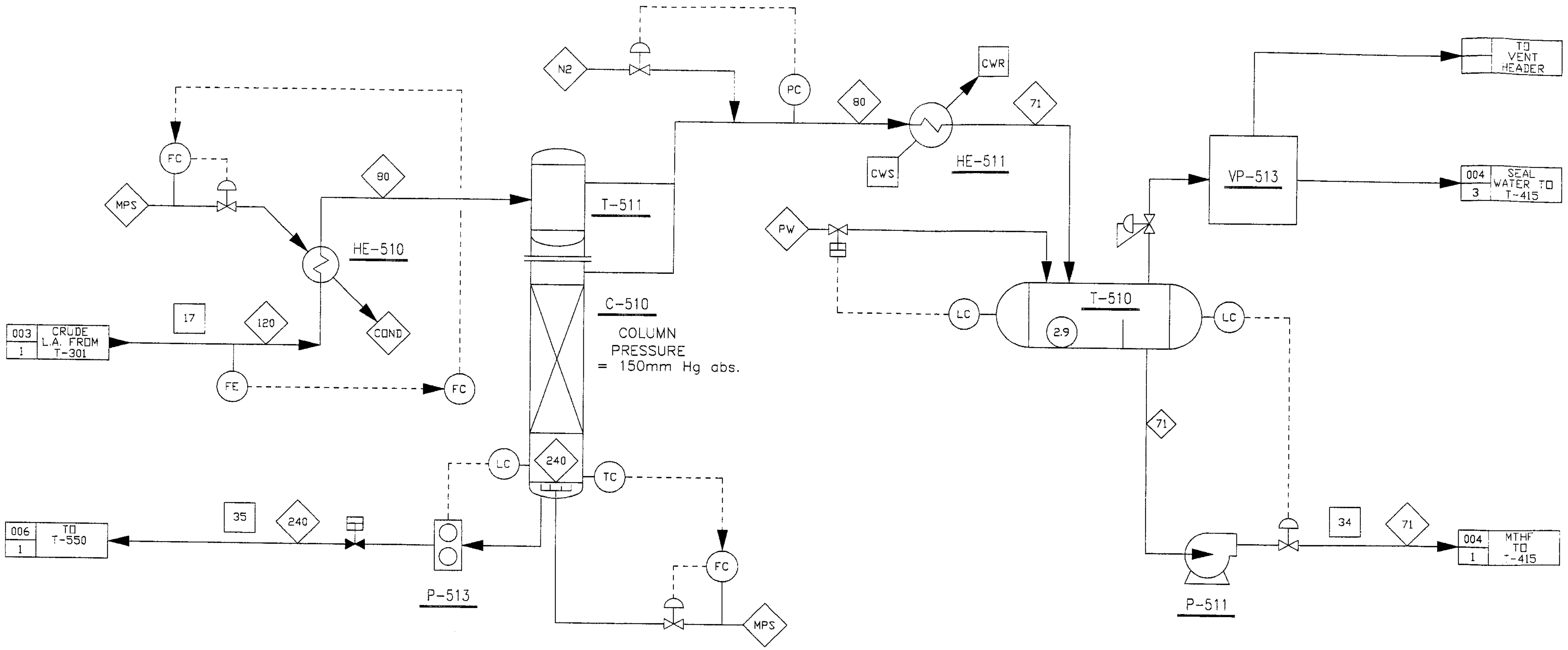
HE-510
MTHF FLASH
HEATER
270,000 BTU/HR

T-511
FLASH
SEPARATOR

C-510
MTHF
STRIPPER
1100 LB/HR

HE-511
MTHF STRIPPER
COOLER
360,000 BTU/HR

T-510
DECANTER
40 GAL.



P-511
MTHF OVERHEADS
PUMP
3 GPM @ 29 PSI

VP-513
MTHF STRIPPER
VACUUM SYSTEM
27 mm Hg abs.

LEGEND:

◇	TEMP °F
○	PRESSURE, psig

BioMetics
PROCESS ENGINEERING • VALIDATION • ARCHITECTURE • GMP CONSULTING
300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

BIOFINE

JOB NUMBER: 96
DRAWN BY: E.B.
CHECKED BY: C.S.
DATE: 1/16/96
REVISED: 4/18/96
7/16/96
1/20/97
6/26/97
SCALE: N.T.S.

LEVULINIC ACID
RECOVERY

PFD5

REVC/PFD-5.DWG

B-550
AIR COMPRESSOR
500 cfm

HE-550
AIR HEATER
140,000 BTU/HR

T-550
AIR STRIPPER

VP-520
W.F.E.
VAC. SYSTEM
25 mm Hg

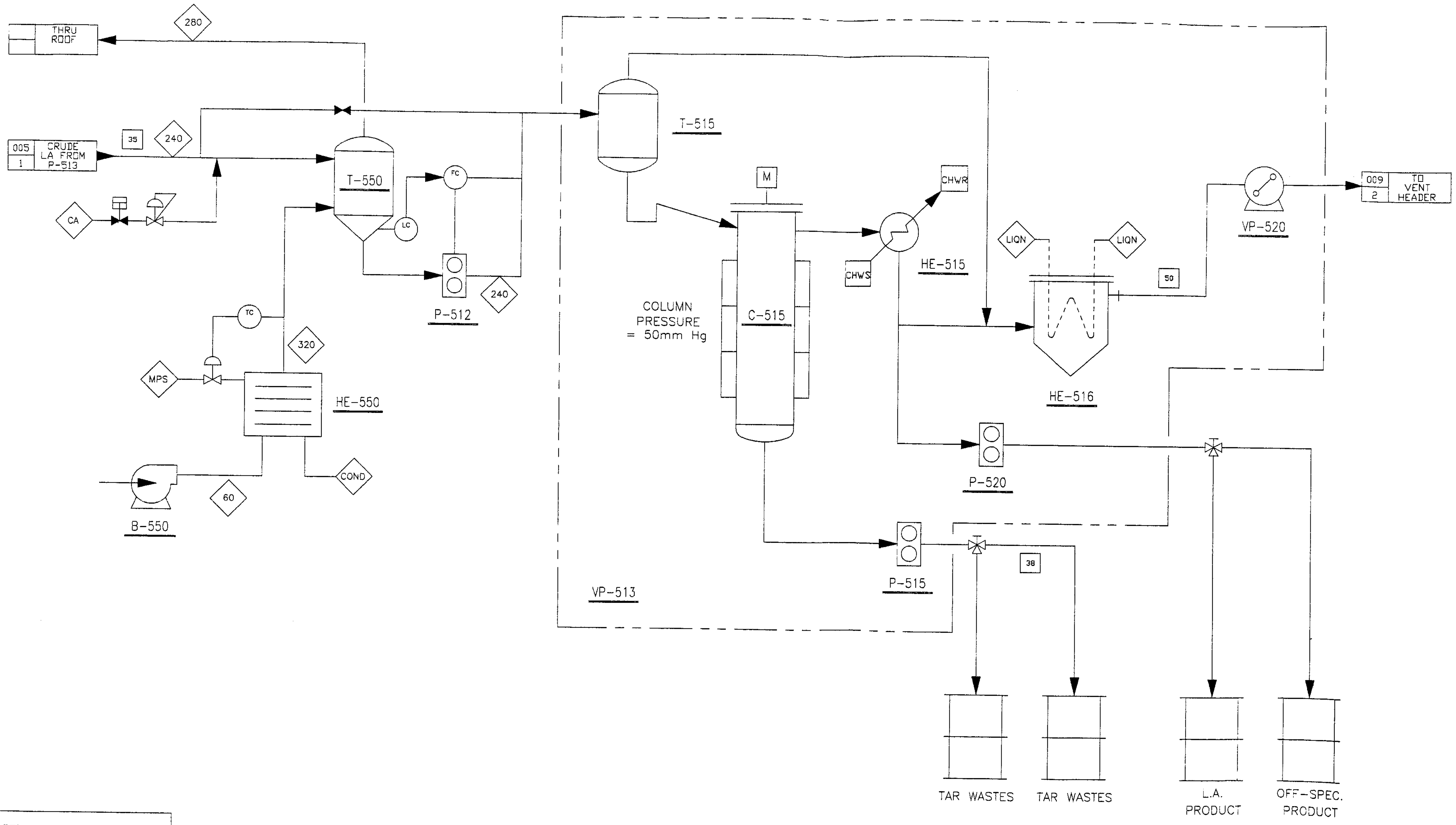
T-515
WFE
DEVOLATIZER

C-515
WIPED FILM
EVAPORATOR
100 LB/HR

HE-515
W.F.E.
PARTIAL COND.
20,000 LB/HR

HE-516
VACUUM
COOLER

P-512
WFE FEED
PUMP
1 GPM @ 20 PSI



P-515
ORGANIC WASTE
PUMP
1 GPM @ 15 PSI

P-520
L.A. PRODUCT
PUMP
1 GPM @ 15 PSI

LEGEND:

◇	TEMP °F
○	PRESSURE, psig

BioMetics
PROCESS ENGINEERING • VALIDATION • ARCHITECTURE • GMP CONSULTING
300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

BIOFINE

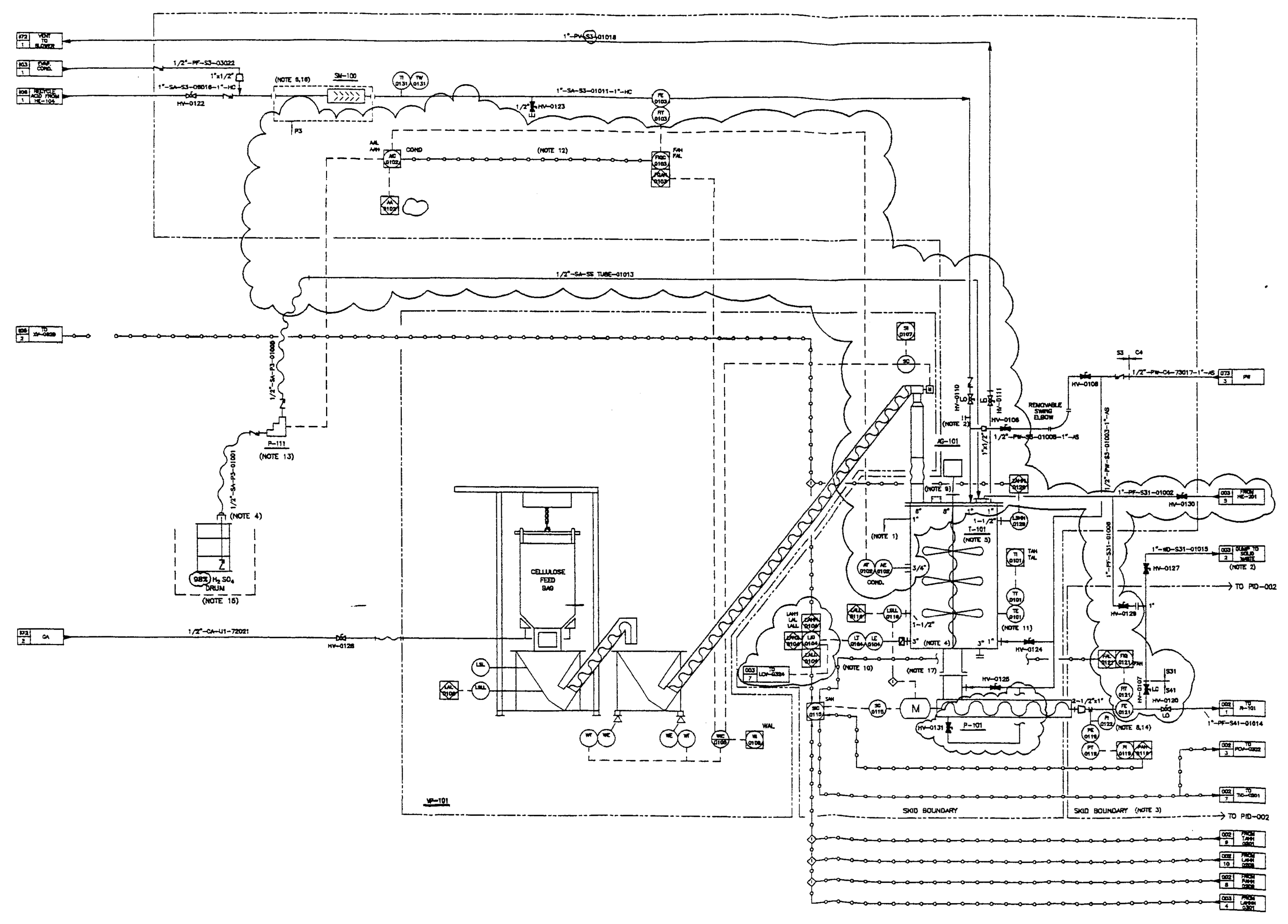
JOB NUMBER: 96
DRAWN BY: E.B.
CHECKED BY: C.S.
DATE: 1/20/97
REVISED: 6/26/97
SCALE: N.T.S.

LEVULINIC ACID RECOVERY

PFD6

REVC/PFD-6.DWG

EQUIPMENT TAG	VP-101	T-101	P-101	AG-101	P-111	SM-100
EQUIPMENT NAME	SOLID FEEDER	PRE-HYD.DRUM	HYDROL. PUMP	AGITATOR	pH CONTROL	STATIC MIXER
CAPACITY	8-10 CU FT/HR	2'6"ø x 4'-0"	5 GPM @600PSIG		1 GPH @ 25PSI	5 GPM
DESIGN RATING	300 LB/HR	ATM.				
MATERIAL OF CONSTRUCTION	CARBON STEEL	316L SS	316L SS	316L SS	PP	TEFLON
HP OR KW	3 HP		8.6 HP	3 HP	0.5 HP	
REMARK	VENDOR PKG.	FLANGE BOTTOM	MOYNO	WITH MIX/AUGER		



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
817 · 884-8331 FAX 817 · 884-8336

NOTES:

1. OVERFLOW DISCHARGE TO SAFE AREA.
2. FOR PROCESS DUMPING OF T-101 SLURRY TO DRUMS AFTER ADDING CAUSTIC TO NEUTRALIZE ACIDIC SLURRY FOR DISPOSAL.
3. REACTOR SKID TO BE COMPLETELY ENCLOSED FOR PERSONNEL PROTECTION.
4. DRUM USES 2" PLUG WITH 1/2" DRILLED OUT SWAGELOCK FITTING FOR SECURING THE TUBE.
5. TANK TOP HAS TWO 4" SIGHTGLASS. ONE WITH A LIGHT.
6. TEE AND MIXER TO BE TEFLON.
7. P-101 SPEED CONTROL SET FROM CONTROL CONSOLE.
8. PSH-0119 & PI-0122. PART OF P-101 PACKAGE.
9. 2' WIDE OPENING WITH SCREEN FOR MANUAL ADDITION OF SOLIDS TO T-101
10. DURING OPERATION LOW FLOW ALARM FAL-0121 STOPS P-101.
11. TE-0101 IS A SURFACE MOUNTED THERMOCOUPLE.
12. NO FLOW PERMISSIVE DISABLES AIC-0102.
13. P-111 SUCTION DISCHARGE TUBING CONNECTED WITH SWAGELOCK FITTINGS.
14. PUMP DISCHARGE PIPING TO BE 2-1/2"ø FOR AT LEAST 6". PIPING TO BE REDUCED TO 1" USING CONCENTRIC REDUCER AND THE SENSOR TO BE LOCATED BETWEEN 2 x 1" 300# FLANGES.
15. WARNING TAPE AROUND SULFURIC ACID DRUM FOR PERSONNEL PROTECTION.
16. SM-100 AND AE-102 TO BE ACCESSIBLE FROM GRADE.
17. P-101 & T-101 ARE TO BE CLOSE COUPLED. T-101 BOTTOM OUTLET PAD TO BE BOLTED DIRECTLY TO P-101.
18. DELETED

USED #s

HAND VALVES:
HV-0101 THRU HV-0131

LINE
01001 THRU 01018

INSTRUMENT #s
0101 THRU 0132

EV.	DATE	DESCRIPTION	CHECKED BY	APPVD BY
4	4/28/98	FINAL AS-BUILT		
3	5/26/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

DOE/BIOFINE

ROJECT NO: 096 DATE: 12/08/95
RAWN BY: E.B. CHECKED BY: S.T.
CALE: N.T.S.
TLE:

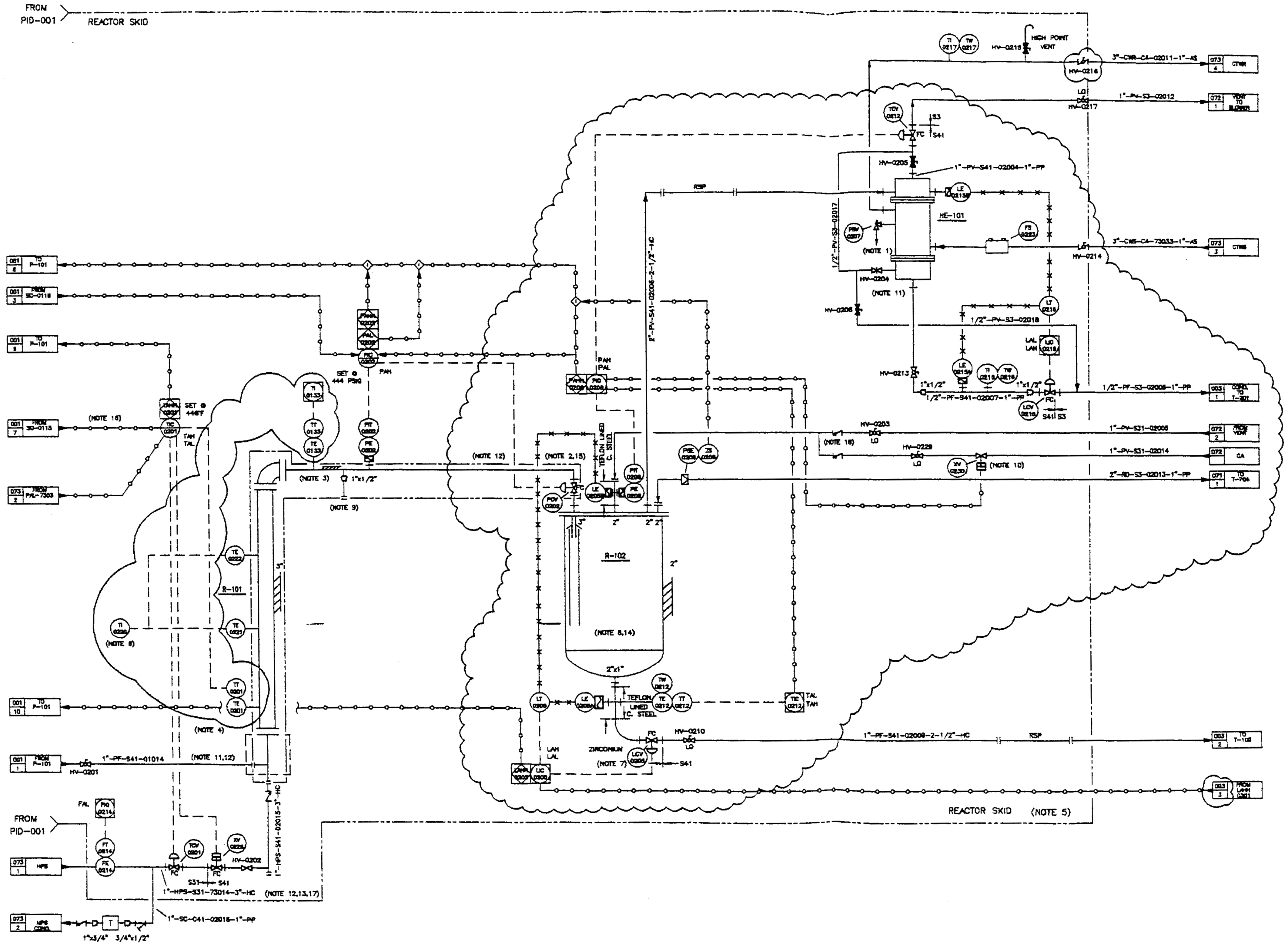
LEVULINIC ACID PILOT PLANT PROJECT

REACTANT BLENDING AND WOOD FEED

JNBER:

PID-001

EQUIPMENT TAG	R-101	R-102	HE-101
EQUIPMENT NAME	1st HYD. REACT.	2nd HYD. REACT.	FLASH COND.
CAPACITY	1"ID x 14'L	2'-6"D x 8'-3"	24 SQ. FT.
DESIGN RATING	475PSIG @550°F	250PSIG @450°F	
MATERIAL OF CONSTRUCTION	ZIRCONIUM	PFA-LINED 316 SS	TUBES-ALLOY 20 SHELL-CS
HP OR KW			
REMARK	PLUG FLOW	CSTR	6"ø x 4' OAL



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. DISCHARGE TO SAFE AREA.
2. "V" TYPE CONTROL VALVE MIN. DISTANCE FROM TANK & SHROUDED.
3. TE-0201 IS SURFACE MOUNTED ELEMENT.
4. SHIELD BASE OF REACTOR WITH MOVABLE LINED CINDERBLOCK HOUSING.
5. A TOTALLY ENCLOSED ISOLATION SHIELD FOR ENTIRE REACTOR SKID TO BE UTILIZED.
6. TI-0220 SWITCHABLE TO VIEW TE-0220, TE-0221, OR TE-0222.
7. 2"x1" REDUCING FLANGE PRIOR TO LCV-0205. DISCHARGE SIDE OF LCV-0205 IS TEFLON LINED FOR 24".
8. ALLOY 20 SPARGER DISCHARGE INSTALLED BELOW LIQUID LEVEL.
9. BLIND FLANGE FOR FUTURE SAMPLING.
10. XV-0230 OPENS WHEN PT-0208 IS LESS THAN 30 PSI.
11. LINE 01014 TO HAVE A 600# FLANGE FOR MATING WITH THE ZIRCONIUM TEE.
12. FLANGE CONNECTIONS TO HAVE SHROUD PROTECTION FROM LEAKS FOR LINES 01014, 02015, & PCV-0201.
13. BLIND FLANGE FOR HYDRAULIC TESTING OF REACTOR, R-101.
14. ALL CONNECTIONS TO HAVE ISOLATION BETWEEN METALLIC AND TEFLON SURFACES. USE PLASTIC BOLT SLEEVE INSERTS AND WASHERS. (VENDOR PROVIDED)
15. PCV-0202 COUPLED TO R-102 NOZZLE. INSERT FLANGED ALLOY 20 SPARGE LINE THROUGH NOZZLE TO AVOID NOZZLE EROSION.
16. TCV-0201 & XV-0228 CLOSED BY P-101 STOPPING (SC-0115), LOW SLURRY FLOW (FAL-0121), LOW HPS PRESSURE (PAL-2311), OR HIGH FEED TEMP (TAHH-0133).
17. LINES 73014 AND 02015 ARE TO BE 600# ANSI RATED PIPING.
18. VACUUM BREAKER

USED #s

HAND VALVES:
HV-0201 THRU HV-0229
LINE #s
02001 THRU 02016
INSTRUMENT #s
0201 THRU 0230

REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY
4	4/28/98	FINAL AS-BUILT		
3	5/28/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/16/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

DOE/BIOFINE

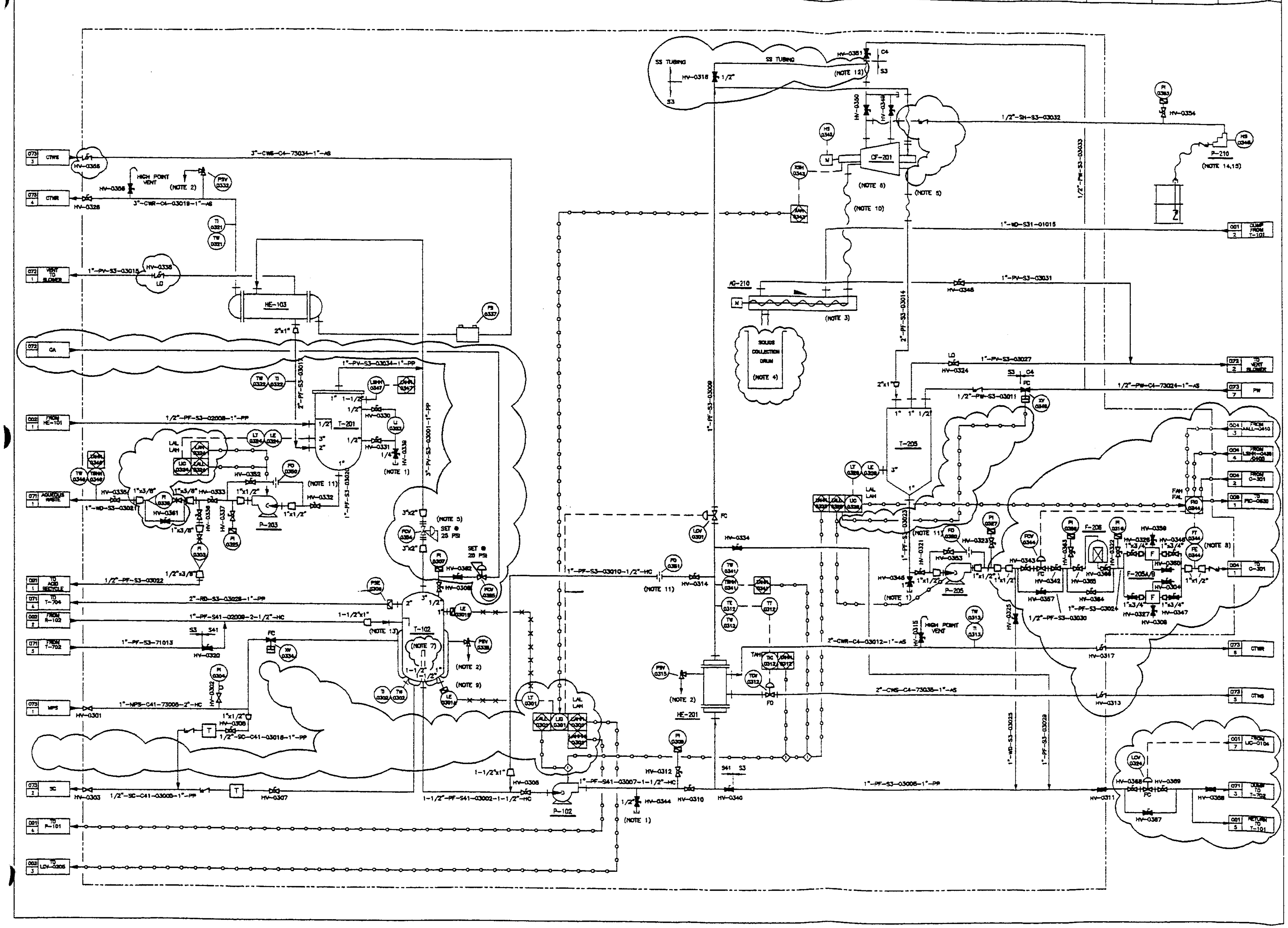
PROJECT NO: 096 DATE: 12/08/95
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.
TITLE:
LEVULINIC ACID
PILOT PLANT PROJECT

REACTION

NUMBER:

PID-002

EQUIPMENT TAG	T-102	HE-201	P-102	CF-201	P-210	T-201	HE-103	P-203	T-205	P-205	F-205A/B	AG-210	F-206						
EQUIPMENT NAME	ATM.FLASH DRUM	HYDROLY.COOLER	HYDROLY.PUMP	CENTRIFUGE	SOLIDS pH PUMP	COND. DRUM	ATM.FLASH COND.	FLASH DRUM PP	HYDR.COLL.DRUM	HYDRO. PUMP	POLISH. FILTER	SOLIDS AUGER	PRE FILTER						
CAPACITY	1"ø x 8'-10"	32 SQ. FT.	5 GPM @ 23PSI	1800 #/HR	5 GPH @ 10PSI	1'øx4' BL/BL	42 SQ. FT.	2 GPM @ 25PSI	3'ø x 4'6"BL/BL	4 GPM @ 23PSI	5 GPM		20 GPM						
DESIGN RATING	50/275; F.V.					10 PSIG @175°F			2 PSIG @175°F		5 MICRON								
MATERIAL OF CONSTRUCTION	ALLOY 20	TUBES-ALLOY 20 SHELL-CS	PFE	316 SS	316L SS/PP	316L SS	TUBES-316L SS SHELL-316L SS	316 SS	316L SS	316 SS	316 SS	304 SS	POLYPRO						
HP OR KW			1.5 HP	7.5 HP	0.5 HP			1.5 HP		1.5 HP		1 HP							
REMARK		8"ø x 8' OAL	CENTRIFUGAL		DOSING PUMP		8"ø x 8' OAL	CENTRIFUGAL	CONE BOTTOM	CENTRIFUGAL									



USED #s

HAND VALVES:
HV-0301 THRU HV-0369

LINE #s
03001 THRU 03034

INSTRUMENT #s
0301 THRU 0356

BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. SAMPLE POINTS ACCESIBLE FROM GROUND LEVEL.
2. DISCHARGE TO SAFE AREA.
3. AG-210 AT 10° ANGLE UP TO DISCHARGE.
4. TO SOLID WASTE DISPOSAL.
5. PCV-0354 USED IN EVENT OF P-102 FAILURE. OTHERWISE SPOOL PIECE INSTALLED.
6. CENTRIFUGE MOUNTED ABOVE AG-210 AND T-205 FOR GRAVITY COLLECTION OF CLARIFIED HYDROLYSATE AND SOLIDS DISCHARGE.
7. 1" TEFLON STRAINER WITH 1/8" HOLES INSTALLED IN BASE OF T-102.
8. FIC-0344 SETS FLOW SETPOINT ON FIC-0830.
9. T-102 BOTTOM NOZZLE AT MINIMUM 6' ELEVATION TO P-102 SUCTION.
10. FLANGE CENTRIFUGE WITH FLEXIBLE CONNECTION TO AG-210.
11. FLOW ORIFICE TO BE PROVIDED WITH 1/8" HOLE.
12. FLEXIBLE CONNECTION FOR WATER FLUSHING OF CENTRIFUGE DISCHARGE SHUTE BOWL.
13. PROVIDE 1/4" GASKET BETWEEN T-102 AND LINE 02009's FLANGES.
14. P-210 SUCTION TUBING IS CONNECTED WITH SWAGELOCK FITTINGS.
15. DRUM USES 2" PLUG WITH 1/2" DRILLED OUT SWAGELOCK FITTING FOR SECURING THE TUBE.

REV.	DATE	DESCRIPTION	CHECKED BY	APPVD BY
4	4/28/98	FINAL ~~AS-BUILT~~		
3	5/27/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/20/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/16/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

DOE/BIOFINE

PROJECT NO: 096 DATE: 12/08/95
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.

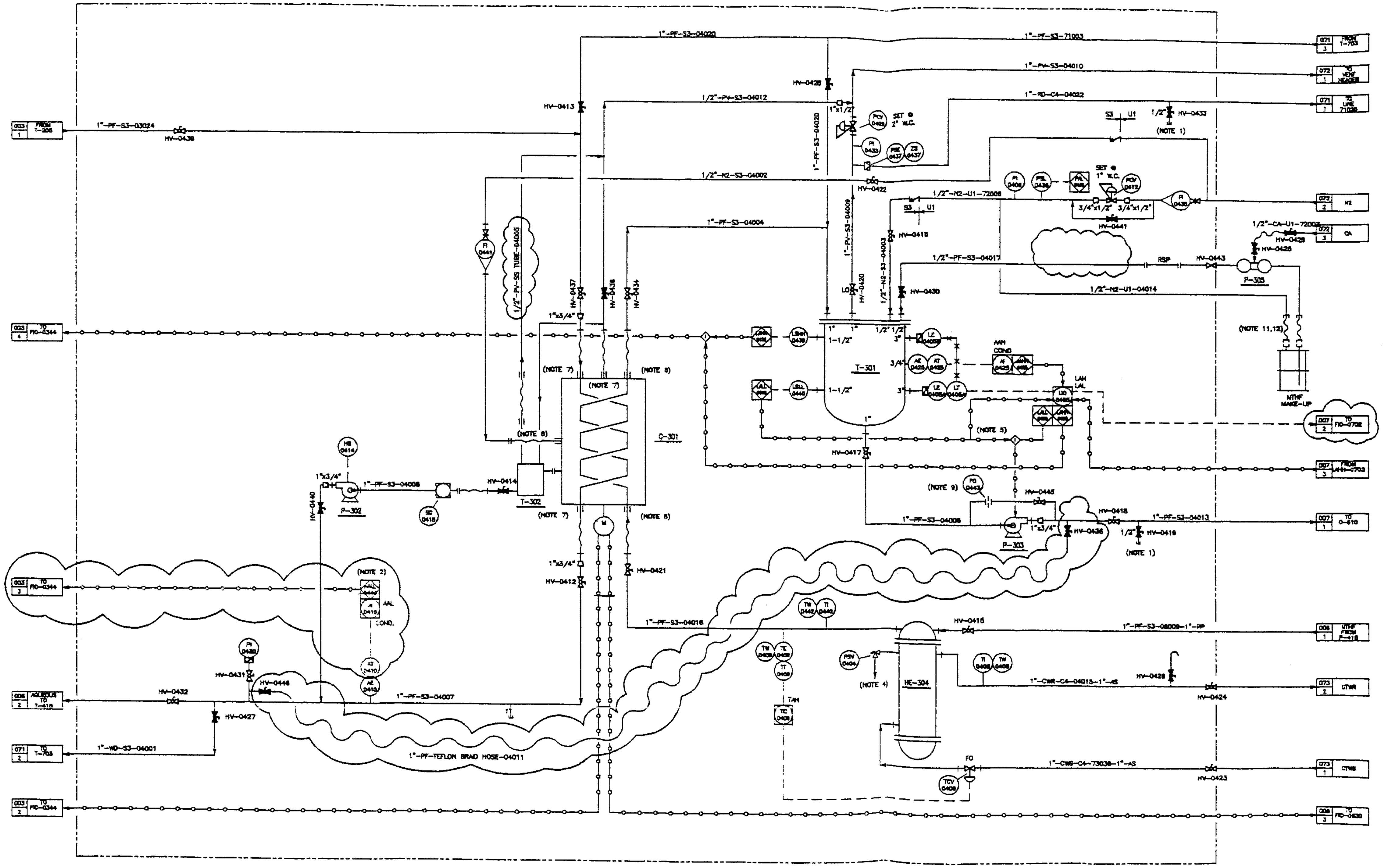
TITLE:
LEVULINIC ACID
PILOT PLANT PROJECT

SOLID SEPARATION

NUMBER:

PID-003

EQUIPMENT TAG	C-301	T-301	P-305	P-302	HE-304	P-303	T-302
EQUIPMENT NAME	CENTR. EXTRACT.	EXTRACT TANK	MTHF MAKE-UP	EXTR. DRAIN PP	MTHF COOLER	MTHF BOOST. PP	DRAIN POT
CAPACITY	8000 #/HR	18"øx30" BL/BL	2 GPM @ 15PSI	4 GPM @ 14PSI	18 SQ. FT.	3 GPM @ 10PSI	12" x 18"
DESIGN RATING	2000 #/HR	15 PSIG/FV 150°F					
MATERIAL OF CONSTRUCTION	SS 316	SS 316L	TEFLON	SS 316	SS 316L TUBES CS SHELL	SS 316	SS 316
HP OR KW	20 HP			1.5 HP		1.5 HP	
REMARK	42"x28"x36"H		AIR DRIVEN	CENTRIFUGAL	6"ø x 6' OAL	CENTRIFUGAL	



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. SAMPLE POINTS ACCESSIBLE FROM GROUND LEVEL.
2. AE-0410 INSTALLED, AT-0410 NOT INSTALLED PENDING INVESTIGATION OF C-301 PERFORMANCE.
3. DELETED
4. DISCHARGE TO SAFE AREA.
5. CLOSE LCV-0406 ON LOW LOW LEVEL IN T-301 BY EITHER LIC-0405 OR LALL-0440.
6. SOLVENT CONNECTIONS ARE 1" NPT.
7. AQUEOUS PHASE CONNECTIONS AND VENTS ARE 3/4" NPT.
8. ADAPT NITROGEN INLET FROM ø6mm TO ø1/2".
9. FLOW ORIFICE TO BE PROVIDE WITH 1/8" HOLE.
10. ALL HAND VALVES IN THE SKID ARE TO BE FIRE RATED. (SEE PIPE SPEC.)
11. POSITIVE CLOSURE QUICK DISCONNECTS.
12. TUBING CONNECTED WITH SWAGELOCK FITTINGS.

USED #s

HAND VALVES:
HV-0411 THRU HV-0446

LINE #s
04001 THRU 04023

INSTRUMENT #s
0401 THRU 0443

4	4/28/98	FINAL AS-BUILT		
3	6/30/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	C.S.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.
REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY

DOE/BIOFINE

[illegible]

PROJECT NO: 096 DATE: 11/09/95
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.

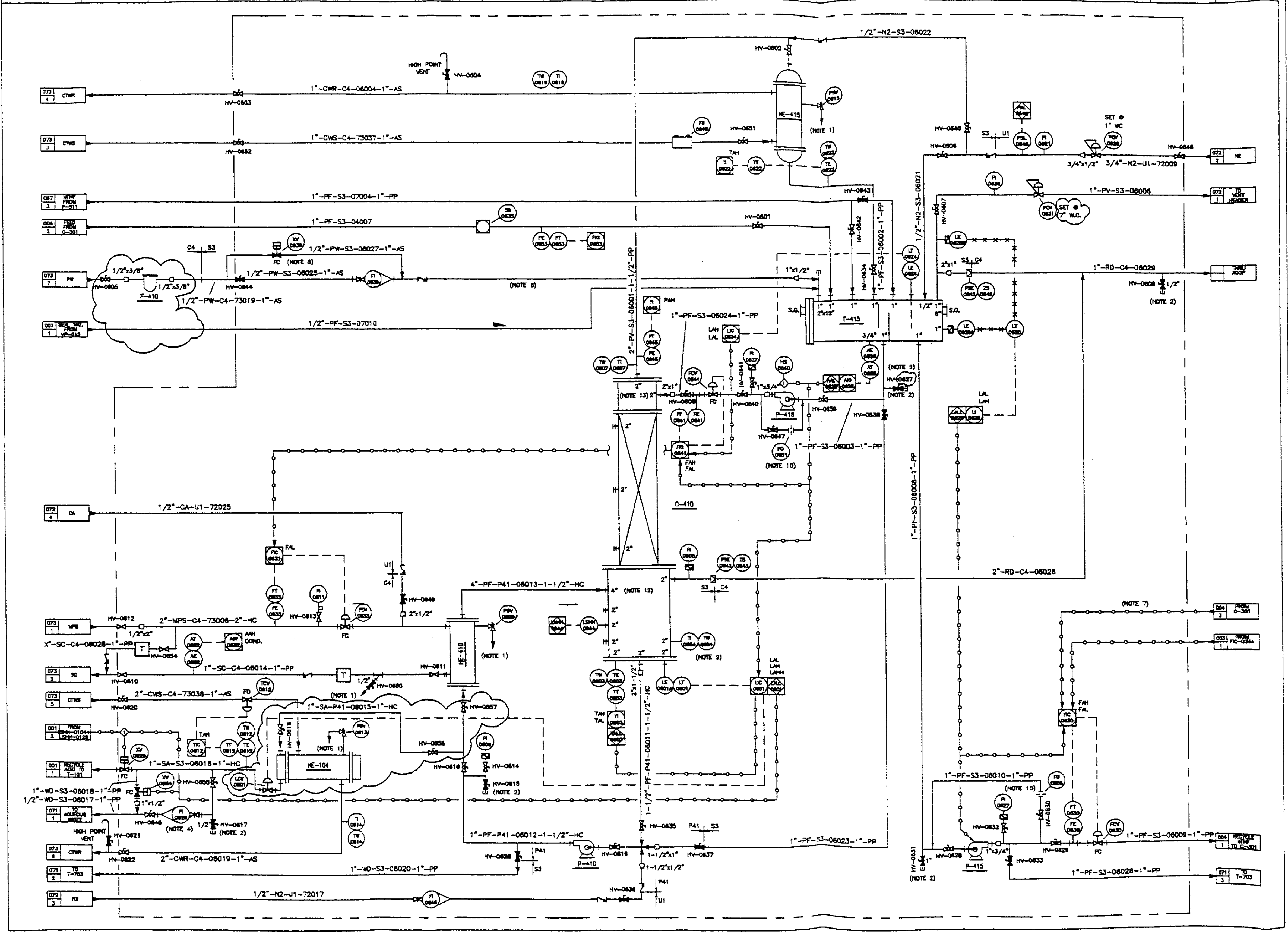
TITLE:
LEVULINIC ACID
PILOT PLANT PROJECT

SOLVENT EXTRACTION

NUMBER:

PID-004

EQUIPMENT TAG	C-410	HE-410	HE-415	T-415	P-410	P-415	HE-104	P-416	F-410
EQUIPMENT NAME	ACID STRIPPER	ACID REBOILER	ACID CONDENSER	MTHF SEPARATOR	RECYC.ACID PUMP	RECYC.MTHF PUMP	RECYC.ACID COOL	RECYC. AQ. PUMP	WATER FILTER
CAPACITY	8"x17'; 18"x4'	63 SQ. FT.	24 SQ. FT.	3'ø x 8'9"	9 GPM @33 PSI	3 GPM @21 PSI	25 SQ. FT.	4 GPM @23 PSI	5 GPM
DESIGN RATING	10 PSIG@250°F			5 PSIG@200°F					5 MICRON
MATERIAL OF CONSTRUCTION	TEFLON LINED C.S.	TUBES-GRAPHITE SHELL-CS	TUBES-316L SS SHELL-CS	316L SS	PFE	316 SS	TUBES-ALLOY 20 SHELL-CS	316 SS	POLYPRO/STYRENE
HP OR KW					2 HP	1.5 HP		1.5 HP	
REMARK	21' OAL	10"ø x 7.5' OAL	6"ø x 4' OAL		CENTRIFUGAL	CENTRIFUGAL	6"ø x 8' OAL	CENTRIFUGAL	



BioMetics

300 BEAR HILL RD. WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. DISCHARGE TO SAFE AREA.
2. SAMPLE VALVE TO BE ACCESSIBLE FROM GROUND LEVEL.
3. DELETED
4. SLIPSTREAM TO PURGE RECYCLE ACID.
5. DELETED
6. PROCESS WATER BYPASS FOR DRY START-UP.
7. DELETED
8. ADD P.W. INTO VERTICAL SECTION OF LINE 1"-PF-S3-07010.
9. MINIMUM ELEVATIONS FOR THE FOLLOWING ARE:

   C-410 – 5' TO BOTTOM NOZZLE TO P-410 SUCTION.

   T-415 – 2-1/2' BOTTOM NOZZLE TO P-416 SUCTION.
10. FLOW ORIFICE TO BE PROVIDED WITH 1/8" HOLE.
11. ALL HAND VALVES IN THE SKID ARE TO BE FIRE RATED. (SEE PIPE SPEC.)
12. REBOILER OUTLET TO C-410 NOZZLE HAS A 3" HALF PIPE INSERT PROVIDED BY COLUMN VENDOR.
13. FEED/REFLUX LINE TO C-410 HAS 1" HALF PIPE INSERT. PROVIDED BY COLUMN VENDOR.

USED #s

HAND VALVES:
HV-0601 THRU HV-0657

LINE #s
06001 THRU 06028

INSTRUMENT #s
0601 THRU 0654

REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY
4	1/22/98	FINAL AS-BUILT		
3	5/27/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

DOE/BIOFINE

PROJECT NO: 096 DATE: 4/18/96
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.

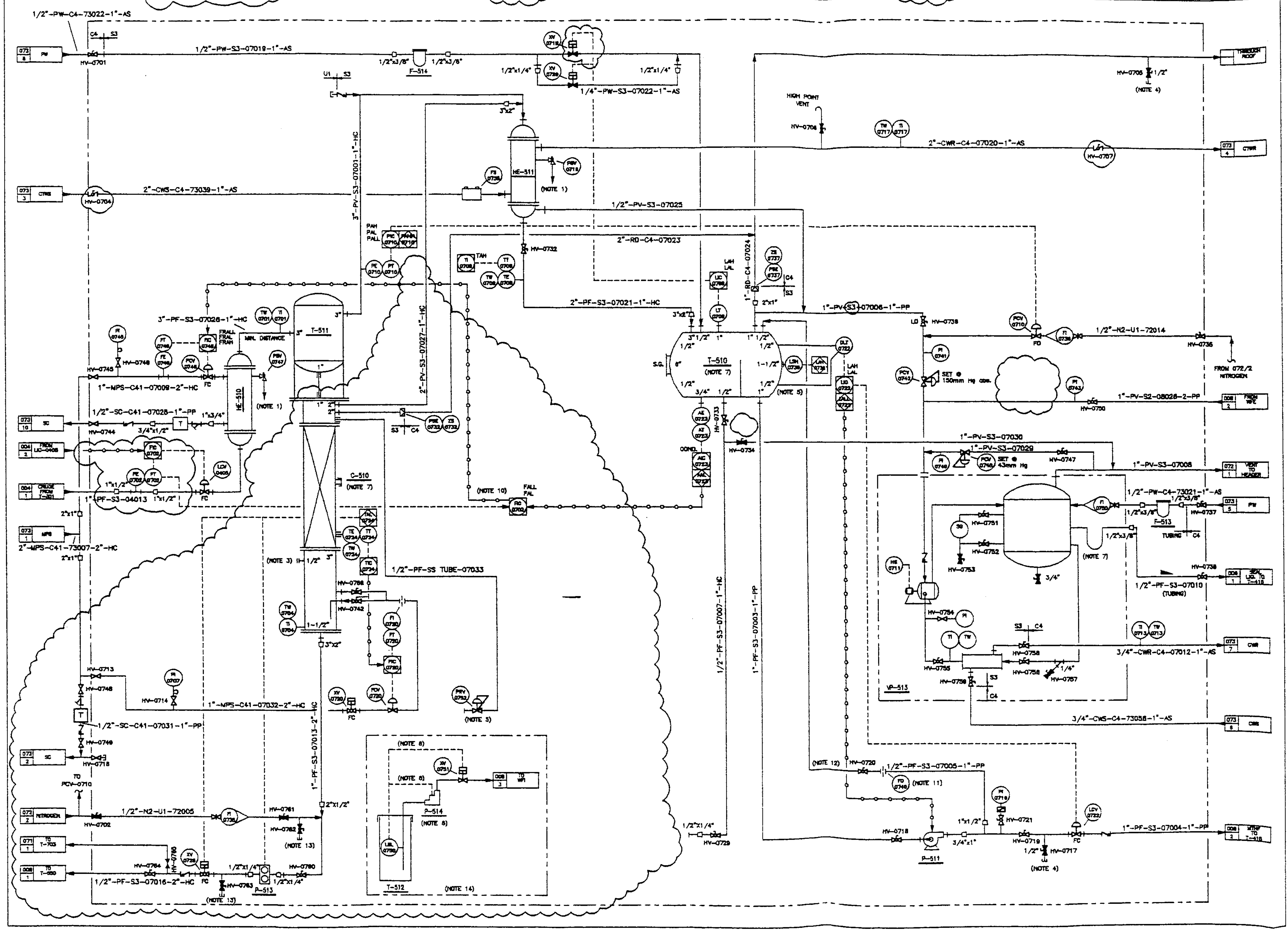
TITLE: **LEVULINIC ACID PILOT PLANT PROJECT**

ACID STRIPPER

NUMBER:

PID-006

EQUIPMENT TAG	C-510	T-512	P-514	HE-511	P-511	VP-513	F-514	T-510	P-513	HE-510	T-511	F-513							
EQUIPMENT NAME	MTHF STRIPPER	THERM.FLUID TK.	THERM.FLUID PP	C-510 COND.	MTHF PUMP	VACUUM PKG.	WATER FILTER	DECANTER	WFE FEED PUMP	MTHF FLASH HTR	FLASH SEPAR.	WATER FILTER							
CAPACITY	8"ø x 10'	50 GAL.	8 GPH	85 SQ. FT.	3 GPM @ 29PSI	150 mm Hg	5 GPM	24"ø x 30"	0.25 GPM	20 SQ.FT.	12"ø x 60"	5 GPM							
DESIGN RATING	10 PSIG/FV 450°F						5 MICRON	50/FV @ 200°F			50/FV @ 200°F	5 MICRON							
MATERIAL OF CONSTRUCTION	316 SS	C.S.	TEFLON	TUBES-316L SS SHELL-CS	316 SS	316 SS	POLYPRO/STYRENE	316 SS	316 SS	TUBES-316L SS SHELL-CS	316 SS	POLYPRO/STYRENE							
HP OR KW					2 HP	7.5 HP			0.5 HP										
REMARK	14'6" OAL			8"ø x 8' OAL	CENTRIFUGAL				GEAR PUMP	8"ø x 4' OAL									



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. DISCHARGE TO A SAFE AREA.
2. NITROGEN SUPPLY FOR PURGE ONLY.
3. BLIND FLANGE FOR FUTURE PROCESS WATER CONNECTION.
4. SAMPLE POINTS ARE TO BE ACCESSIBLE FROM GROUND LEVEL.
5. FOR POSSIBLE THERMAL FLUID ADDITION.
6. P-514 STARTED MANUALLY.
7. MINIMUM ELEVATIONS FOR THE FOLLOWING ARE:

   C-510 – 5' TO BOTTOM NOZZLE TO P-510 SUCTION.

   T-510 – 10' TO BOTTOM NOZZLE TO P-512 SUCTION.

   VP-513 – 10' TO OVERFLOW
8. LOW LEVEL IN T-512 STOPS P-514 AND CLOSES XV-0751.
9. ALL HAND VALVES IN THE SKID ARE TO BE FIRE RATED. (SEE PIPE SPEC.)
10. SOFTWARE SETPOINT TO FIC-0746 IS BASED ON FE-0702 FLOW RATE.
11. FLOW ORIFICE TO BE PROVIDED WITH 1/8" HOLE.
12. NO VAPOR LOCKING.
13. USE HV-0762, HV-0763 FOR NITROGEN FLUSH IN CASE OF PUMP BLOCKAGE.
14. T-512 & P-514 LOCATED IN PROCESS ROOM 2.

USED #s

HAND VALVES:
HV-0701 THRU HV-0766

LINE #s
07001 THRU 07033

INSTRUMENT #s
0701 THRU 0752

4	4/28/98	FINAL AS-BUILT		
3	5/27/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.
REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY

DOE/BIOFINE

PROJECT NO: 096 DATE: 4/18/96
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.

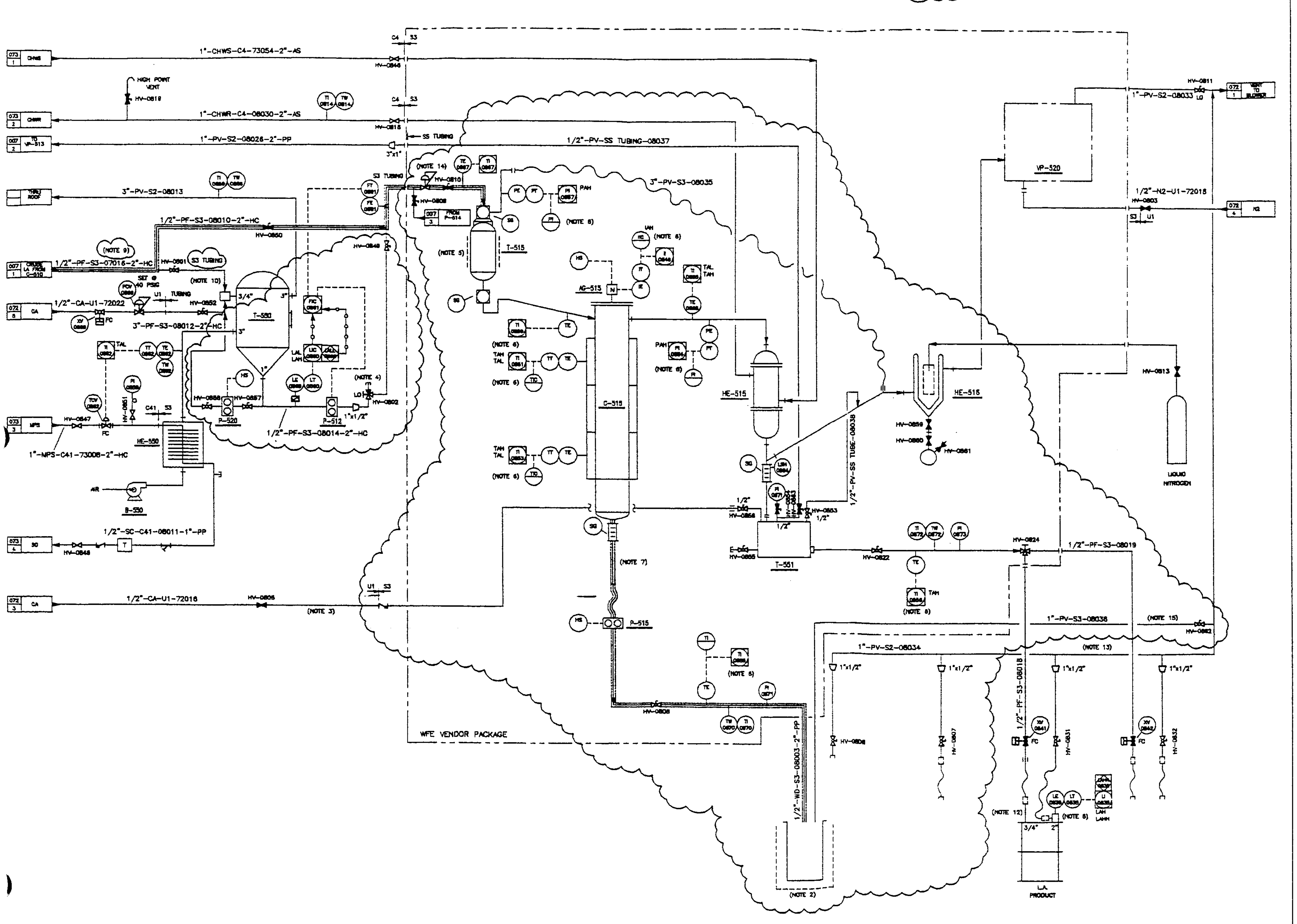
TITLE:
LEVULINIC ACID PILOT PLANT PROJECT

CRUDE L.A. MTHF STRIPPING

NUMBER:

PID-007

EQUIPMENT TAG	C-515	P-515	HE-515	P-520	VP-520	T-515	HE-516	T-550	B-550	HE-550	AG-515	T-551	P-512
EQUIPMENT NAME	FILM EVAP.	ORG.WASTE PUMP	WFE PEUT.COND.	LA PROD. PUMP	WFE VAC. SYS.	WFE DEVOLATIZ.	VACUUN COOLER	AIR STRIPPER	AIR BLOWER	AIR HEATER	AGITATOR	PRODUCT POT	WFE FEED
CAPACITY	100 LB/HR	1 GPM @ 20 PSI	5 SQ. FT.	1 GPM @ 20PSI	25 mm Hg			12"ø x 60"	750 cfm	200,000 BTU/HR		5 GAL.	0.25GPM@20PSI
DESIGN RATING	FV							50/FV@ 400°F				50 PSI/FV	
MATERIAL OF CONSTRUCTION	316 SS	316 SS	TUBES-316L SS SHELL - CS	316 SS	316 SS			316 SS	316 SS	316 SS		SS 316	316 SS
HP OR KW													0.5 HP
REMARK	INCL'D W/W.F.E.	INCL'D W/W.F.E.	INCL'D W/W.F.E.	INCL'D W/W.F.E.	INCL'D W/W.F.E.	INCL'D W/W.F.E.	INCL'D W/W.F.E.		VENDOR PKG.	VENDOR PKG.			GEAR PUMP



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. DISCHARGE TO A SAFE AREA.
2. ORGANIC WASTE COLLECTED IN DISPOSABLE PAPER DRUMS. ISOLATE WITH BARRIER FOR PERSONNEL PROTECTION.
3. N_2 FOR CLEARING OF TAR.
4. DIVERTER VALVE UTILIZED FOR CLEANING OR REPROCESSING.
5. HEAT TRACE DEVOLITIZER.
6. PANEL MOUNTED LOCAL CONTROL BY VENDOR PACKAGE. INSTRUMENT SENDS 4-20mA SIGNALS TO THE PLANT CONTROLLER FOR SUPERVISION AND DATA STORAGE ONLY.
7. ORGANIC WASTE LINES TO BE HEAT TRACED BY WFE VENDOR.
8. MOVE LEVEL ELEMENT TO NEW DRUM WHEN SWITCHING.
9. HEAT TRACE LINE TO 180°F.
10. CRUDE LA ENTERS T-550 THROUGH AN AIR ATOMIZING NOZZLE ASSEMBLY.
11. DELETED
12. POSITIVE CLOSURE QUICK DISCONNECT CONNECTIONS.
13. VENT LINES FROM DRUMS UTILIZE SWAGELOCK COMPRESSION FITTINGS WITH TEFLON TUBING FOR FLEXIBLE CONNECTION.
14. BACK PRESSURE VALVE SET AT 20 PSI.
15. VENT FOR RESIDUE AREA.

USED #s

HAND VALVES:
HV-0801 THRU HV-0863

LINE #s
08001 THRU 08038

INSTRUMENT #s
0801 THRU 0871

4	4/28/98	FINAL AS-BUILT		
3	5/27/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.
REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY

DOE/BIOFINE

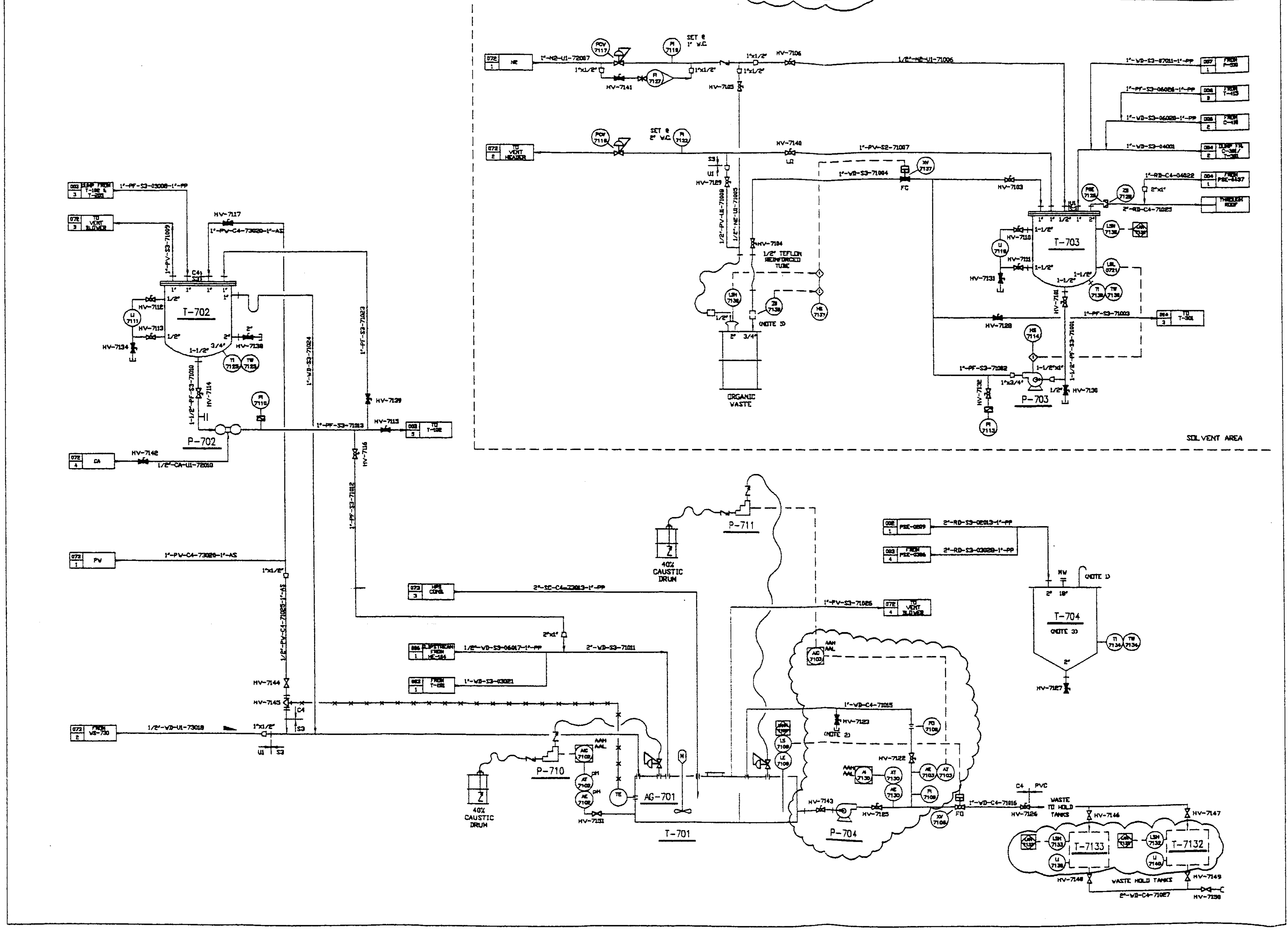
PROJECT NO: 096 DATE: 4/18/96
DRAWN BY: E.B. CHECKED BY: B.J.P.
SCALE: N.T.S.

TITLE:
LEVULINIC ACID PILOT PLANT PROJECT

LEVULINIC ACID FINISHING

NUMBER:
PID-008

EQUIPMENT TAG	T-701	P-704	AG-701	T-702	T-703	P-703	P-702	P-710	T-704	P-711	T-7133	T-7132
EQUIPMENT NAME	NEUTRAL.TANK	NEUTRAL. PUMP	WASTE TK.AGIT.	INORG.DUMP TK.	ORG. HOLD TK.	ORG. HOLD PUMP	INORG.RET.PUMP	CAUST.DOS.PUMP	CONTAIN. TANK	DOSING PUMP	EFFL.STOR.2	EFFL.STOR. 1
CAPACITY	275 GAL.	5 GPM @ 20PSI		150 GAL.	150 GAL.	10 GPM @ 25PSI	10 GPM @ 25PSI	50 GAL. DRUM	175 GAL.	50 GAL. DRUM	3000 GAL.	3000 GAL.
DESIGN RATING	ATM.										ATM	ATM
MATERIAL OF CONSTRUCTION	P.P.	C.S.	C.S.	316L SS	316L SS	316 SS	316 SS	POLYPROP.	304		C.S.	C.S.
HP OR KW		0.5 HP	0.5 HP			2 HP	2 HP	0.5 HP		0.5 HP		
REMARK	4'L x 3'W x 4'H	CENTRIFUGAL		2'9"Ø x 4'-0"	2'9"Ø x 4'-0"	CENTRIFUGAL	CENTRIFUGAL	PACKAGED UNIT	3'Ø x 4'6"		SITE SUPPLY	SITE SUPPLY



NOTES:

1. VENT TO SAFE AREA.
2. SAMPLE PORT
3. CONTENTS TO BE NEUTRALIZED BY CAUSTIC BEFORE DISPOSAL.
4. DRUM USES 2" PLUG WITH 1/2" DRILLED OUT SWAGELOCK FITTING FOR SECURING THE TUBE.
5. POSITIVE CLOSURE QUICK DISCONNECT CONNECTIONS.

USED #s

HAND VALVES:
HV-7101 THRU HV-7151

LINE #s
71001 THRU 71027

INSTRUMENT #s
7101 THRU 7140

REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY
4	4/28/98	FINAL AS-BUILT		
3	5/27/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

DOE/BIOFINE

BioMetics
300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 884-8336

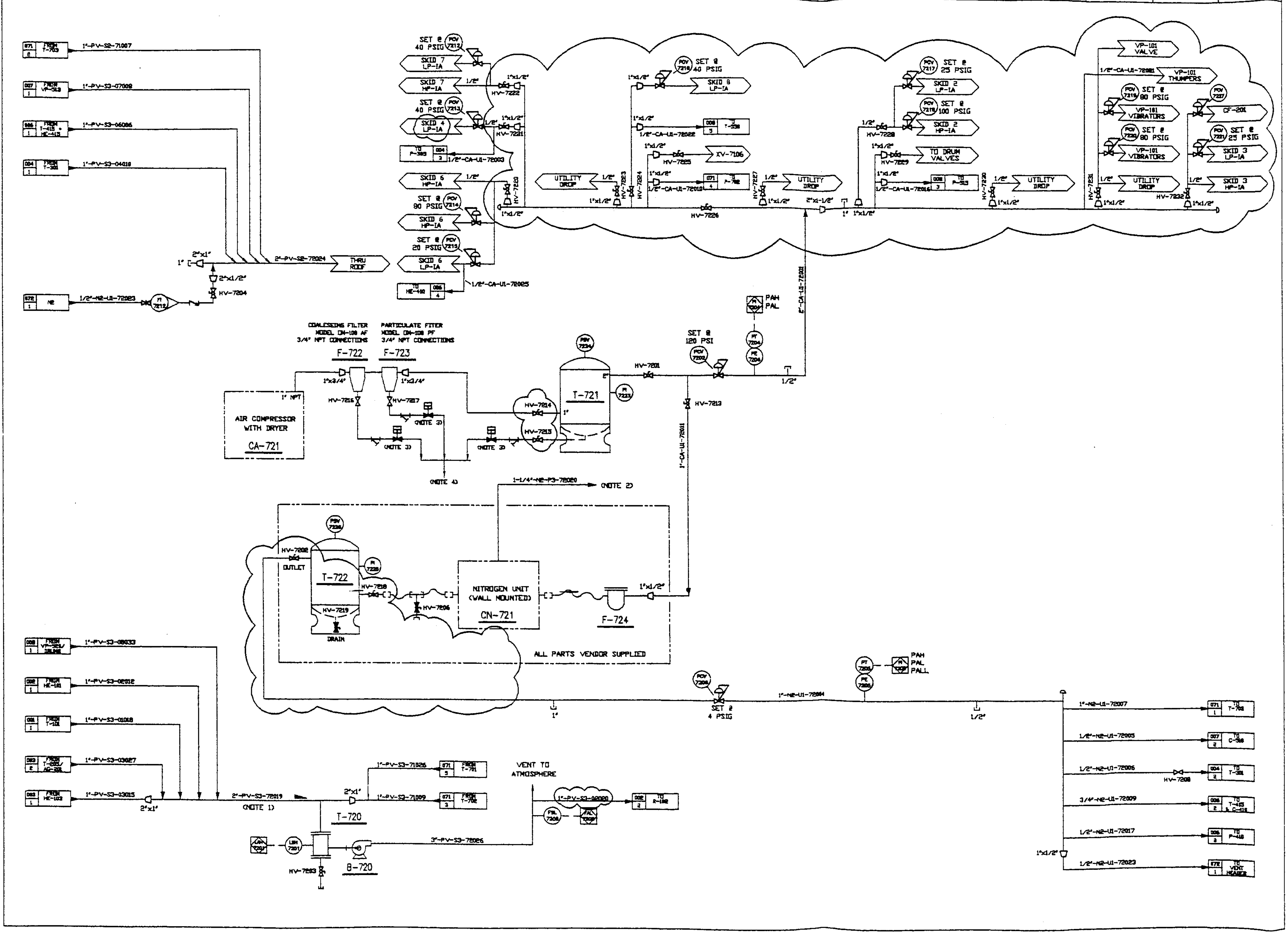
PROJECT NO: 096 DATE: 4/16/96
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.

TITLE:
LEVULINIC ACID
PILOT PLANT PROJECT

COLLECTION & WASTE
NEUTRALIZATION SYSTEMS

NUMBER:
UFD-071

EQUIPMENT TAG	B-720	CA-721	CN-721	T-720	F-722	F-723	F-724	T-721	T-722
EQUIPMENT NAME	BLOWER	COMPRESSED AIR	NITROGEN GEN.	CATCH POT	COAL. FILTER	PART. FILTER	PART. FILTER	COMP. AIR TANK	NITROGEN TANK
CAPACITY	500cfm@4"WC	50cfm@100PSIG	7 cfm	12"Ø x 18"	100 scfm	100 scfm		120 GAL.	400 GAL.
DESIGN RATING		150PSIG @ AMB.		ATM		0.9 MICRON		200 PSI	150 PSI
MATERIAL OF CONSTRUCTION	316 SS			316 SS				PAINTED CS	PAINTED CS
HP OR KW	2.5 HP	20 HP							
REMARK		PACKAGED SYST.	PACKAGE SYST.		PART OF CA-721	PART OF CA-721	PART OF CN-721		



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 · 684-8331 FAX 617 · 684-8335

NOTES:

1. ENTER VENT HEADER LINE 72019 FROM ABOVE. 72019 TO SCOPE TO CATCH POT.
2. EXHAUST POINT FOR CN-721 TO BE 12" ABOVE GRADE AND DIRECTED TO BUILDING VALL.
3. VALVES ARE THE SURE DRAIN VALVES.
4. PIPE TO PROCESS ROOM 2 SUMP.

USED #s

HAND VALVES:
HV-7201 THRU HV-7232

LINE #s
72001 THRU 72026

INSTRUMENT #s
7201 THRU 7227

REV.	DATE	DESCRIPTION	CHECKED BY	APP'VD BY
4	4/28/98	FINAL AS-BUILT		
3	5/27/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

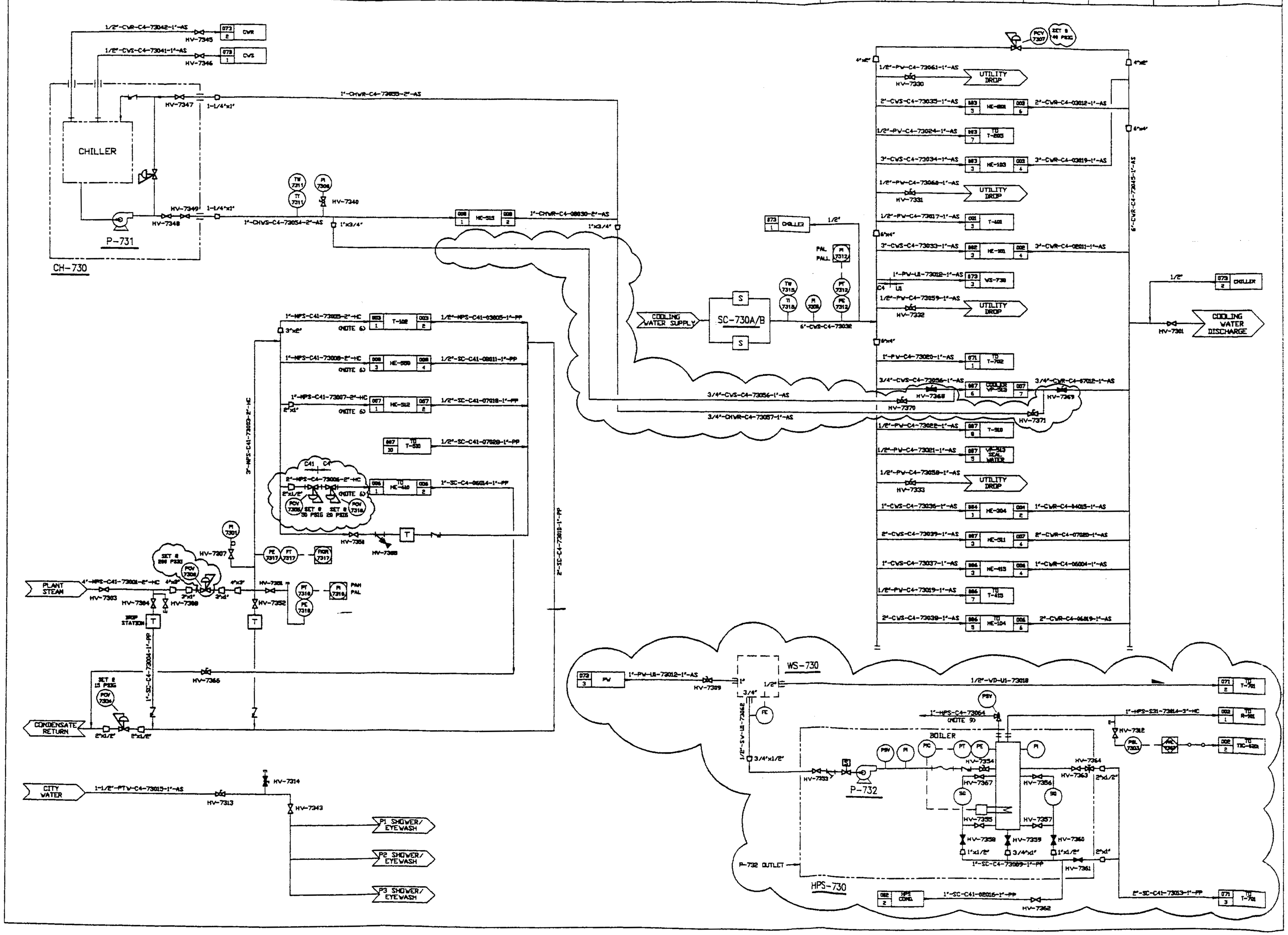
DOE/BIOFINE

PROJECT NO: 096 DATE: 11/09/95
DRAWN BY: E.B. CHECKED BY: S.T.
SCALE: N.T.S.

TITLE:
LEVULINIC ACID
PILOT PLANT PROJECT
COMPRESSED AIR, NITROGEN,
AND VENT SCRUBBING
SYSTEMS

NUMBER:
UFD-072

EQUIPMENT TAG	WS-730	HPS-730	P-732	P-731	CH-730	SC-730A/B
EQUIPMENT NAME	WATER SOFTENER	STEAM GENERAT.	BOILER FEED	CHW SUPP.PUMP	CHILLER	CWS SCREENS
CAPACITY	2.5 GPM	750 LB/HR	790#/LURE800PSI	20GPM@35PSIG	35,000 BTU/HR	500 GPM
DESIGN RATING		500 PSIG	800 PSI			
MATERIAL OF CONSTRUCTION				CAST IRON W/ BRONZE INPELL.		
HP OR KW		240 KW	1.5 HP	2 HP	3 HP	
REMARK	W/BRINE TANK	ELECTRIC FIRED	W/HPS-730	W/CHILL. CH-730		



BioMetics

300 BEAR HILL RD., WALTHAM MA 02154
617 - 884-8331 FAX 617 - 884-8335

NOTES:

1. VENT TO SAFE AREA.
2. OUTDOOR INSTALLATION
3. DIGITAL RUN SIGNAL TO MAIN PLC.
4. COOLING WATER PUMP & STRAINER PROVIDED BY OTHERS.
5. PLANT WATER USED FOR HPS-730 BLOWDOWN QUENCH SYSTEM.
6. TRAP AT LOW POINT.
7. LINE 73014 TO BE 600# ANSI RATED PIPE.
8. CITY WATER USED AS BACK-UP FOR BOILER.
9. TERMINATE HP BOILER PSV IN SAFE AREA BEHIND P-102.

USED #s

HAND VALVES:
HV-7301 THRU HV-7371

LINE #s
73001 THRU 73064

INSTRUMENT #s
7301 THRU 7318

REV.	DATE	DESCRIPTION	CHECKED BY	APPV'D BY
4	4/28/98	FINAL ~~AS-BUILT~~		
3	5/30/97	AS-BUILT	B.J.P.	S.W.F.
2	11/14/96	CONSTRUCTION REVISION	C.S.	S.W.F.
1	9/25/96	CONSTRUCTION REVISION	B.J.P.	C.S.
0	8/07/96	ISSUE FOR CONSTRUCTION	B.J.P.	C.S.
C	7/17/96	ISSUE FOR HAZOP	B.J.P.	C.S.
B	5/28/96	ISSUE FOR CONTROL BID	B.J.P.	C.S.
A	4/19/96	ISSUE FOR BID	B.J.P.	C.S.

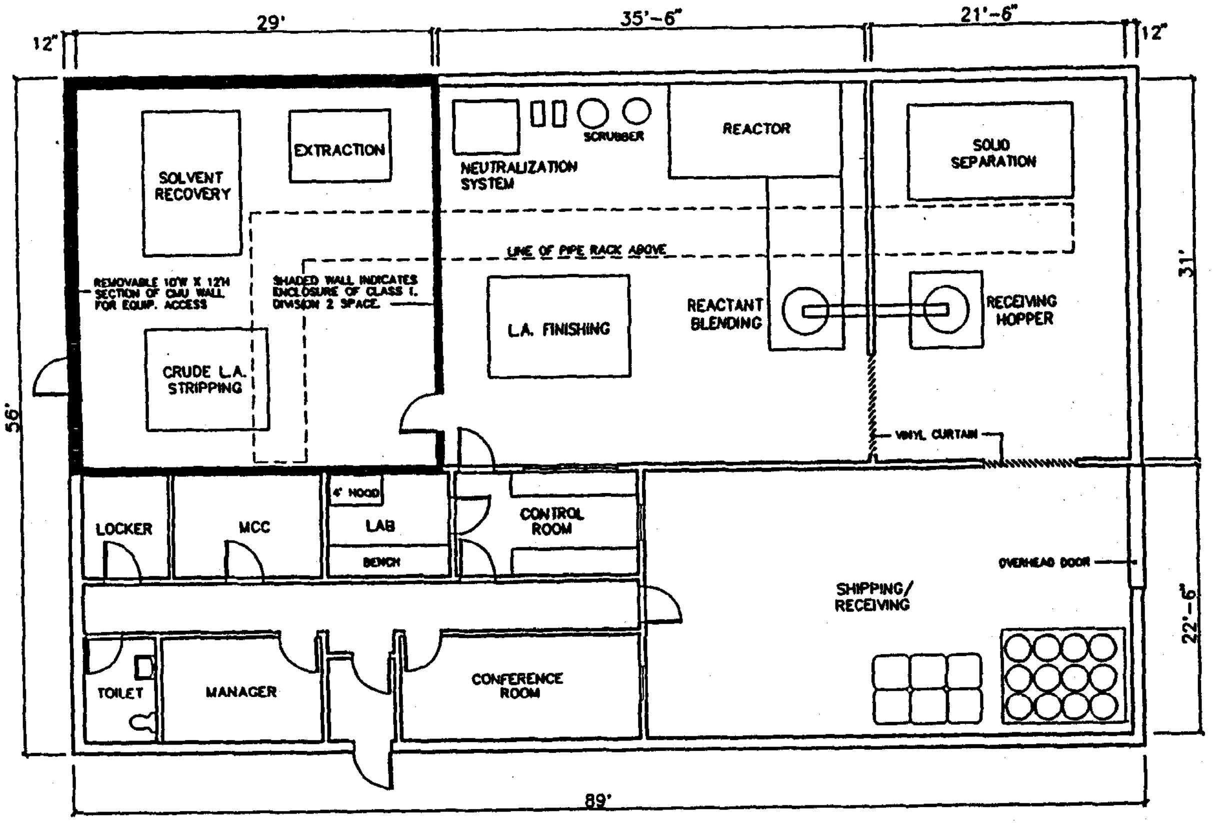
DOE/BIOFINE

[illegible]

PROJECT NO: 096 DATE: 4/18/96
DRAWN BY: E.B. CHECKED BY: B.J.P.
SCALE: N.T.S.

TITLE:
LEVULINIC ACID
PILOT PLANT PROJECT
PLANT STEAM, H.P. STEAM,
COOLING TOWER WATER,
CHILLED WATER & PLANT
WATER SYSTEMS

NUMBER:
UFD-073



SCHEME X 7/3/96
GROSS AREA: 5,000 SF

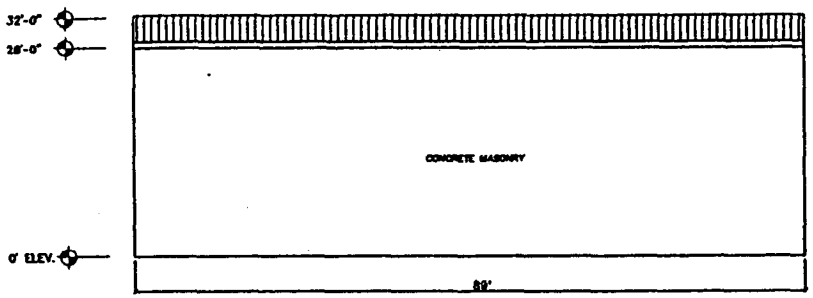
SHEET: 1 OF 1
SCALE: 1" = 10'
DRAWN BY: KG
DATE: 7/3/96
CHECKED BY:
NOTE:

FLOOR PLAN:
BIOFINE DEMONSTRATION PLANT

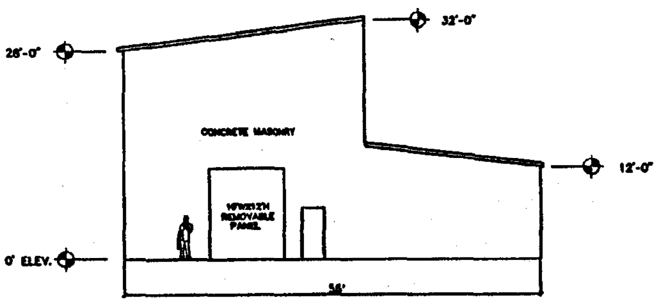
SCHEME X

BioMetics
PROCESS ENGINEERING • VALIDATION • ARCHITECTURE • cGMP CONSULTING
245 WINTER STREET, WALTHAM MA 02154
617 • 684-8331 FAX 617 • 684-8335

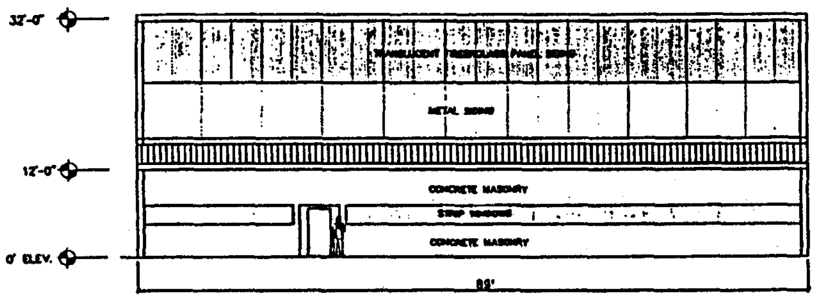
DRAWING:
A-1
BIOFINR/SURVEY1.DWG



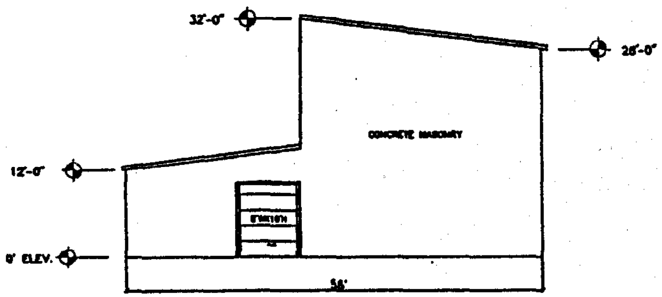
North Elevation



West Elevation



South Elevation



East Elevation

ELEVATION DRAWINGS ILLUSTRATE BUILDING SHAPE AND MATERIALS ONLY. FINAL ELEVATION DESIGN, DETAILS AND CONSTRUCTION DOCUMENTS ARE TO BE DEVELOPED AND PRODUCED BY THE DESIGN/BUILD TEAM.

SHEET: 1 OF 1
SCALE: 1/16" = 1'-0"
DRAWN BY: KG
DATE: 7/3/98
CHECKED BY:
NOTE:

ELEVATIONS:
BIOFINE DEMONSTRATION PLANT

SCHEME X

BioMetics
PROCESS ENGINEERING • VALIDATION • ARCHITECTURE • cGMP CONSULTING
245 WINTER STREET, WALTHAM MA 02154
617 • 684-6331 FAX 617 • 684-6335

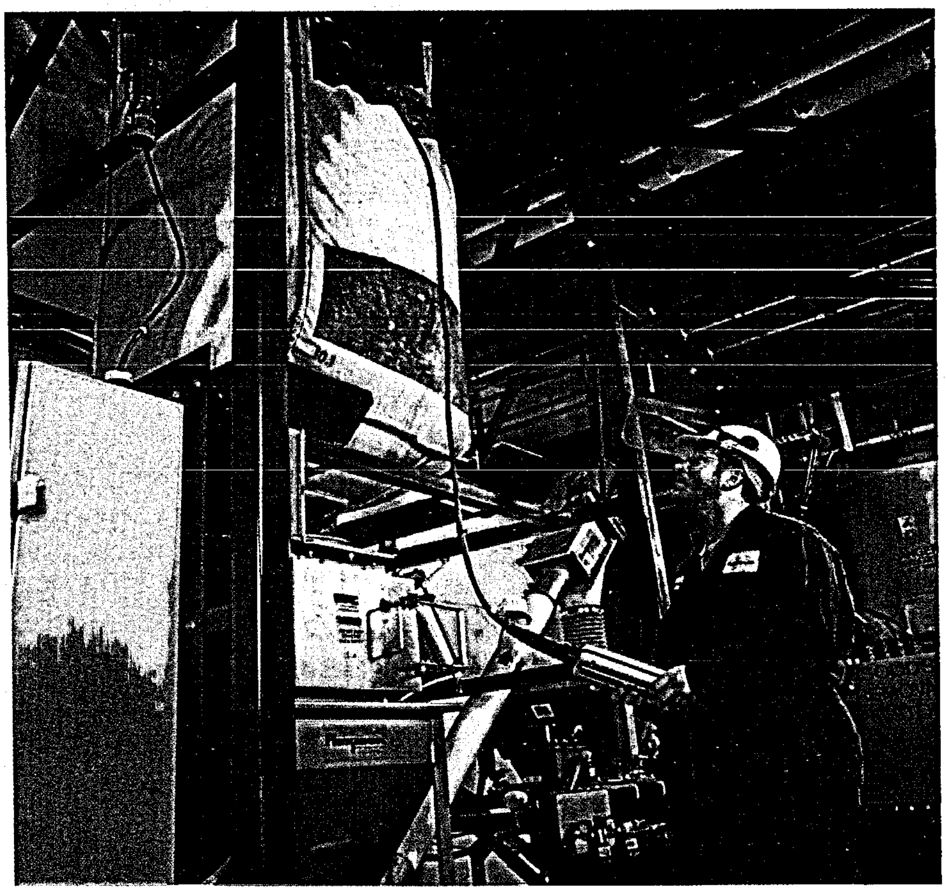
DRAWING:
A-2
BIOFINR/SURVEY1.DWG

BIOFINE
DEMONSTRATION
PLANT

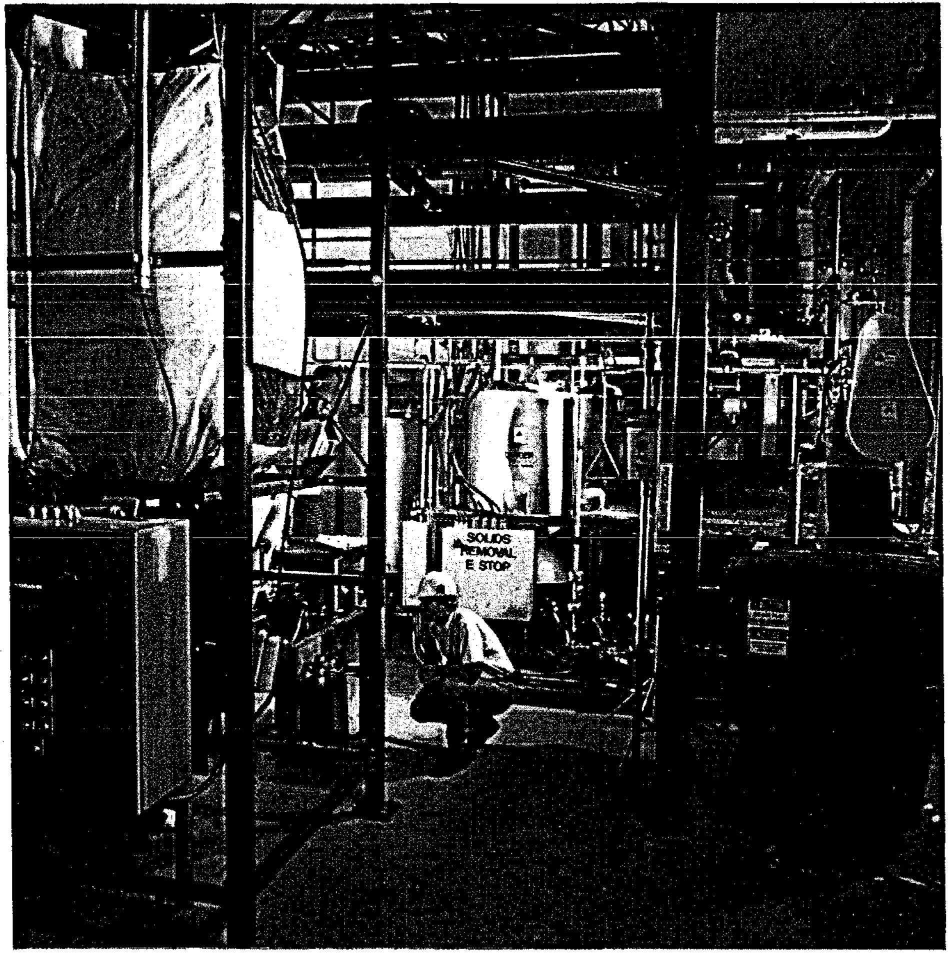
Photos of Biofine's Commercial Demonstration Plant in South Glens Falls, NY

BIOFINE DEMONSTRATION PLANT
FEEDSTOCK STORAGE

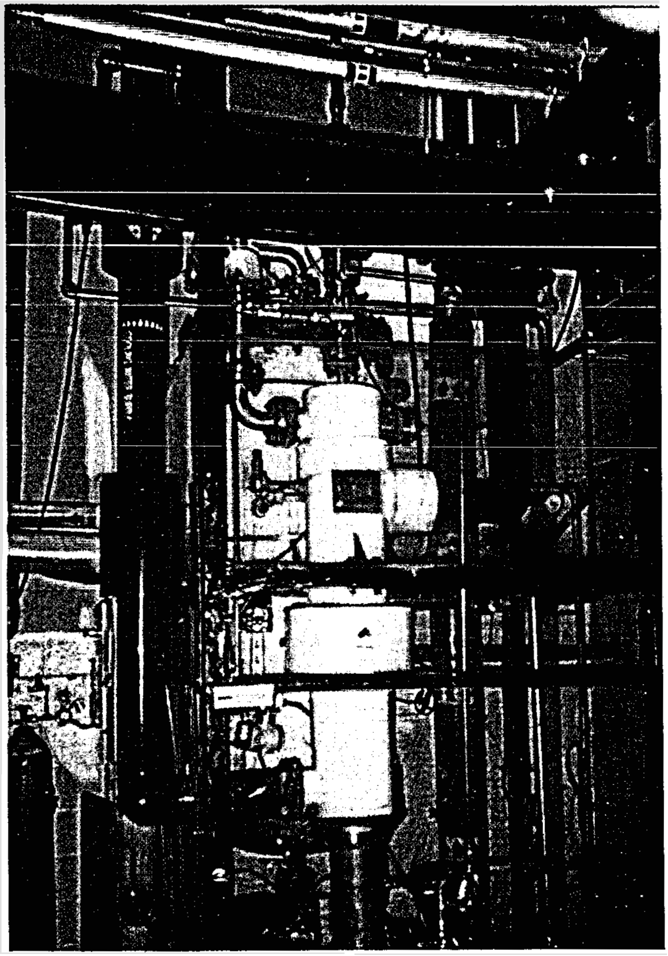
BIOFINE DEMONSTRATION PLANT – FEEDSTOCK HANDLING



BIOFINE DEMONSTRATION PLANT – OPERATING AREA II

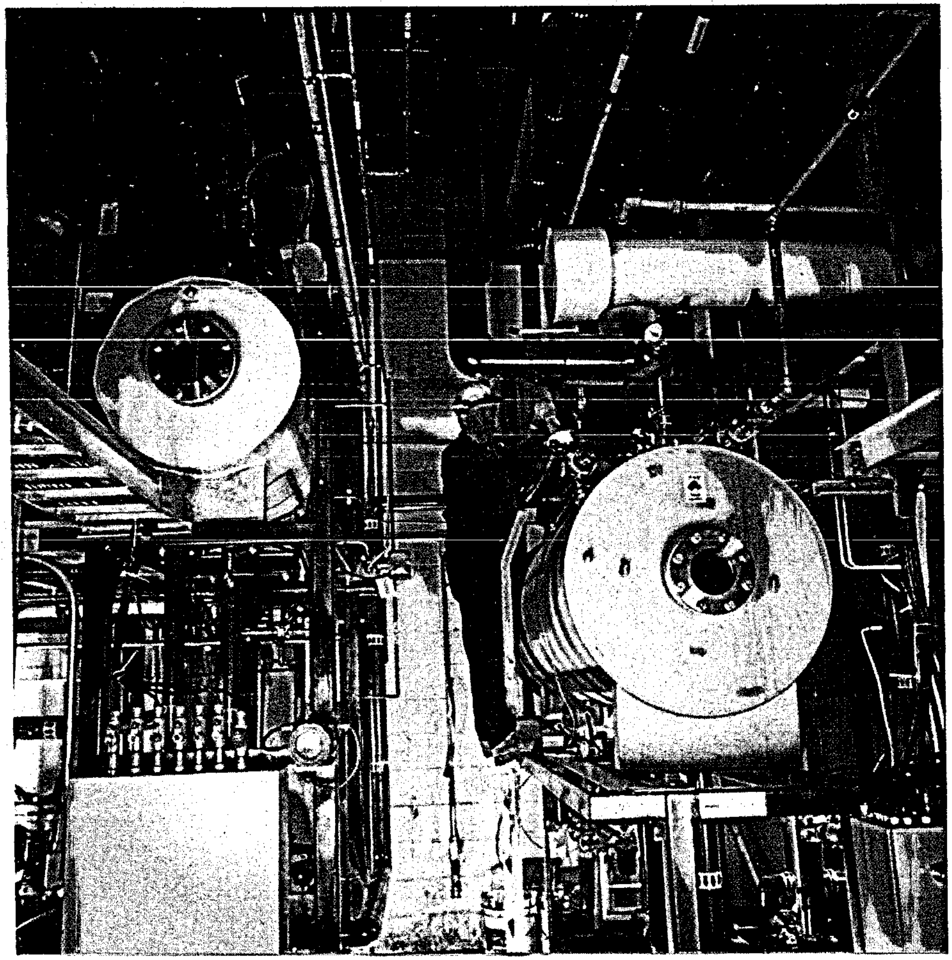


SECOND STAGE REACTOR
(R-102)



WIRED FILM EVAPORATOR AND HIGH PRESSURE BOILER

SOLVENT STRIPPING (C-500) + ACID STRIPPING (C-400) COLUMNS



BIOFINE DEMONSTRATION PLANT – PRODUCT COLLECTION

BIOMETICS
MEMORANDUM

DATE: August 8, 1996

TO: S. Fitzpatrick/B. Pichette/C. South/S. Tsai

FROM: T. Y. Lam

SUBJECT: Biofine Demonstration Plant
HAZOP Review Meeting Report

Attached is the draft HAZOP Review Meeting Report for the Biofine Demonstration Plant. The HAZOP was conducted in accordance with a classical guide word technique for normal plant operation only . HAZOP for startup, shutdown and emergency shutdown has been deferred to a later date. The specific methodology and scope are as outline in the July 16, 1996 BJP Memorandum (also attached).

Please review and address all items with a Risk Rating of A or B or with specific recommendations.

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
1	Flow- NO	1) Empty Bag 2) Bridging of Bag 3) Bridging of feed to T-101 4) Flex conveyor speed control fails 5) Feed system weigh element fails. 6) Foreign object (larger than 2") 7) Solids freezing	No solids fed to the process	1,2) Weigh element and LSL on feed hopper, bridge breakers on bag sides, & software monitoring of time for bag to empty. 3) None 4) Hopper weigh element 5) LCL on feed hopper and the weigh elements have inherent self diagnostics 6) Solids delivered presifted 7) Solids stored in heated area	3 3 3 2 2 3	2 3 3 3 3 3	C C C C C C	
	Flow - MORE	1) Weigh element out of calibration 2) Weigh element failure 3) Flex conveyor speed control fails	More solids fed to T-101 which will inhibit flow to P-101.	1) T-101 level control 2) Weigh elements have inherent self diagnostics 3) Hopper weigh element	2 2 3	3 3 3	C C C	Software to have trending and alarms based on rate deviations >5% for control variables
	Flow - LESS	Same as MORE flow.						
	Composition AS WELL AS	Foreign object larger than 2"	P-101 failure	Solids delivered presifted	2	3	C	SOP and quality requirements on feed materials.
	Composition PART OF	Excess or lack of water and/or cellulose	Improper feed composition which will prohibit LA production	Cellulose feedstock delivered in batches which are quality tested and characterized prior to use.	2	3	C	SOP for cellulose feedstock sampling to include % moisture, % cellulose & titrations.
2	Flow- NO	1) XV-0629 in wrong position 2) LE-0104 on T-101 fails high	T-101 level drops	1) FAL-0103 & valve position indication 2) FAL-0103 & LSHH-0129	3 3	3 3	C C	If position switch differs from required operating position, then alarm.
2	Flow - MORE	1) FE-0103 fails high 2) FI-0132 left open 3) FI-0303 left open	Water balance not maintained	1) LE-0104 2, 3) LE-0104 & FAH-0103	3 3	2 2	C C	SOP for normal operation require operator to monitor T-101 level to maintain water balance.
	Flow - LESS	Same as MORE flow.						
	Composition AS WELL AS	Solids in recycle acid line	Possible plugging	None	3	3	C	Very unlikely to occur

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Composition PART OF	Excess or lack sulfuric acid due to pH meter failure	No acid or high acid in the system	Routine operator sampling at HV-0123.	3	3	C	1) Software to have trending and alarms based on rate deviations >5% for control variables 2) Consider the addition of a spare pH meter. 3) Verify stainless steel compatibilitywith higher concentrations of H_2SO_4
2.1	Flow - REVERSE	P-111 stopped	Water enters 98% H_2SO_4 drum, possible explosive reaction	None	1	2	A	1) Remove PSV 2) Reduce H_2SO_4 concentration from 98% to ≈50% or less. 3) Surround H_2SO_4 drum with plastic shroud.
	Flow - MORE/LESS	Same as 2.0 Composition						
	Pressure - LESS	Same as REVERSE flow						
	Composition - OTHER THAN	MTHF drum used in place of H_2SO_4 drum	Solvent vapors in an unclassified area, electrical hazard.	None	1	3	B	1) SOP for MTHF & H_2SO_4 storage in different areas. 2) Drums to be purchased in different colors.
2.2	Flow - REVERSE	Low PW header pressure	Sulfuric acid contaminating the PW header & MOC of PW header not designed for H_2SO_4	None	1	3	B	1) Add swing elbow to isolate the PW during operation. 2) Move PW point of entry to after the blind flange near T-101 for gravity drainage.
3.0	Level - MORE	WE/LE-0104 fails low	P-101 runs dry	LSLL-0116	3	3	C	
	Level - LESS	WE/LE-0104 fails high	T-101 overflows	LAHH- 0129	3	3	C	
4.0	Flow - NO	1) P-101 failure due to stator 2) PCV-0202 fails closed or is blocked 3) Specticle blind closed 4) Line blockage @ feed to P-101 5) Line blockage after the pump	1) Reactor shutdown 2,3) High pressurization of R-101 system. 4) Pump runs dry and ruins stator	1) FAL-0102, PAL-0202 2) PAHH-0202, PSH-0119 & FAL-0121 3) PAHH-0202, PSH-0119 & FAL-0121 4) FAL-0121, TAHH-0201 5) FAI-0121	3 3 3 3 3	3 3 3 3 3	C C C C C	3) Operating SOP for system start-up will require operators to ensure hand valve and specticle blind positions

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - HIGH	1) P-101 speed controller fails 2) TCV-0201 fails open 3) PSV-0202 remains stuck open 4) Line failure 5) HV-0118	1,2) System shutdown 3) System shutdown and everse flow from R-102 4) Possible personnel injury 5) Wasted feedstock	1,2) None 3) PSE-0209 4) PAL-0202 interlock 5) None	3 2 3 1 3	3 2 3 3 3	C A C B C	1) Add FAH to FIQ-0121 2) Add ON/OFF valve on steam line w/ same operatin permissives as TCV-0201 3) Depressurize R-102 by overriding TCV-0212 control into an open position 4) Reactor area to be fully enclosed & restircted area. 5) Operating SOP for system start-up will require operators to ensure hand valve and specticle blind positions
	Flow - REVERSE	1) HP steam @ low pressure 2) Pump bridging or stator failure	1) Acidic liquid enters boiler & corrodes 2) Steam flow thru P-101 to T-101	1) None 2) FE-0121	2 2	2 3	A C	1) Add PIT to boiler supplyline 2) Set PAHH setpoint @ 550 psi; Add skin temperature indication after FE-0121; Add plexiglass enclosure around the viton joint; Verify if mag meter can differentiate reverse flow.
4.0	Flow - LESS	1) Line blockage	1) Overpressuriation of reactor system	PAHH-0202 if blockage @ line or PAL-0202 if blockage prior to PE-0202	3	3	C	
	Temperature - HIGH	TCV-0201 stuck open	Reduced residence time in reactor	TAHH-0201	3	3	C	Add ON/OFF valve on steam line w/ same operating permissive as TCV-0201
	Temperature - LOW	1) Low steam pressure 2) High reactor pressure 3) TCV-0201 fails closed	Incomplete reaction	1) Added PIT on boiler line 2)TAL-0201 & PAHH-0202 3) TAL-0201	3 3 3	3 3 3	C C C	
	Pressure - MORE	1) Line blockage 2) Excess steam due to TCV-0201 open 3) Specticle blind in wrong position 4) PCV-0202 closed	1) See LESS flow 2) See MORE flow 3) See NO flow 4) See NO flow					
	Pressure - LESS	1) PCV-0202 stuck/failed open 2) TCV-0201 failed/stuck closed 3) P-101 failure	1) See HIGH flow 2) See LOW temperature 3) See NO flow					
	Phase - MORE	1) Steam not condensing due to low pressure 2) Too much steam due to TCV-0201 open	1) See LESS pressure 2) See MORE flow					

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Corrosion/ Erosion - MORE	1) More than anticipated 2) More acid 3) Gritty feed stock	Line blowout	1,2 & 3) Routine hydrotest of R-101 system; Cinder block enclosure around R-101 base; Reactor area to be fully enclosed & restircted area	1	3	B	
4.1	Corrosion/ Erosion - MORE	Low pressure in HPS line therefore reverse flow	Line failure	None	2	3	C	Change material of construction from carbon steel to stainless steel
5, 6	Flow - MORE	1) TCV-0212 failed open 2) LCV-0215 failed open	1,2) R-102 depressurizat'n, vapor dump to vent system & possible shutdown	1) PAL-0208 2) PAL-0208 & LAL-0215	3	3	C	1) Increase the size of the vent header to approx 4" 2) If position switch on valve differs from required operating position, then alarm.
	Flow - LESS	1) TCV-0212 failed closed 2) LCV-0215 failed closed 3) Cooling Water fails	1) HE-101 will blanket w/ vapor so R-102 pressure will rise. 2,3) No condensation - vapor entrained w/ liquid enters the vent system	1) PAHH-0208 2& 3) PAHH-0208	3 3 3	3 3 3	C C C	
5, 6	Flow - OTHER THAN	Low cooling water flow	No condensation - vapor entrained w/ liquid enters the vent system	PSE-0207	3	3	C	
	Temperature - HIGH	1) Malfunction of TE-0212	Weakness of R-102 lining	PAHH-0208	3	3	C	
	Temperature - LOW	TCV-0212 or LCV-0215 fails open	See MORE flow		3	3	C	
	Pressure - MORE	TCV-0212 or LCV-0215 fails closed	See LESS flow		3	3	C	
	Pressure - LESS	TCV-0212 or LCV-0215 fails open	See MORE flow		3	3	C	
	Level - NO / LESS	R-102 1) LE-0205 - fails high 2) LCV-0205 - fails open HE-101 3) LCV-0215 - Fails open 4) No cooling water supply	1) Level rises in R-102 2) Level rises in R-102 See MORE flow See LESS flow	1) LAH-0301 2) LAL-0205 & LAH-0301	3 3	3 3	C C	Add LAHHH to shut P-101 down

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Level - HIGH	R-102 1) LCV-0205 - stuck closed 2) LE-0205 fails low HE-101 3) LCV-0215 - fails closed 4) LE-0215 - element failure	1) Reactor system shutdown 2) Reactor system shutdown 3 & 4) No condensation - vapor entrained w/ liquid enters the vent system	1) LAHH-0219 2) LAHH-0205 & LAHH-0219 3) LAH-0215 & PAHH-0208 4) PAHH-0208	3 3 3 3	3 3 3 3	C C C C	
	Corrosion / Erosion	1) R-102 has a faulty liner 2) Solids impinge on R-102's bottom 3) Dip tube erosion	R-102 failure	Enclosed area to personnel	1 1 2	3 3 3	B B C	Routine leakage monitoring
7.	Flow - NO	1) Line blockage 2) LCV-0205 fails closed 3) LE-0205 fails low	System shutdown	1 & 2) LAHH-0219 & LAHH-0205 3) LAHH-0219	3 3 3	3 3 3	C C C	Add LAHHH to ensure shut-down of P-101
	Flow - MORE	1) LCV-0205 fails open 2) LE-0205 fails high	T-102 level jumps up T-102 level jumps up	1) LAL-0205 & LAHH-0301 2) LAHH-0301	3 3	3 3	C C	
	Erosion - MORE	Erosion in turbulent region post LCV-0205	Line rupture	None	1	3	B	Mount LCV-0205 flush to R-102 and follow it with a zirconimum elbow.
8.	Level - MORE	LE-0301 fails low	Liquid overflows to vent	None	3	3	C	1) T-102 will be banging due to vapor hitting liquid level in T-102 and T-201 level increases at a greater rate than normal.
	Level - LESS	LE-0301 fails high	P-101 cavitates	None	3	3	C	
	Pressure - MORE	Hand valve, HV-0338 closed	T-102 overpressurizes	PSE-0308 ruptures	3	3	C	Operating SOP for system start-up will require operators to verify hand valve positions
9,10, 11	Flow - NO	1) LCV-0324 failed close 2) LE-0324 fails low	1 & 2) Liquid backs up into vent system	1) LAH-0324 2) None	3 2	3 3	C C	Add independent LSHH
	Flow - MORE	Excess feed rate to T-102	HE-103 overloaded and excess vapro escapes to vent system	None	3	3	C	
	Flow - LESS	1) Condenser fouling 2) Less CW	1 & 2) Less condensate to T-201 and potential vapor lock on HE-103	None	3 3	3 3	C C	Verify HE-103 orientation(put process on shell side for better venting & draining)
	Temperature - MORE	1) No cooling water supply 2) LCV-0205 fails open	1) Vapor to vent system 2) Vapor to vent system	1) None 2) Failure alarms @ R-102	3 3	3 3	C C	

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Pressure - MORE	HE-103 vapor locked	Overpressure of T-201	None	2	3	C	Rupture disc on T-102 services both T-102 & T-201 since they share a common vent.
	Phase	1) Condenser fouling 2) Less CW	Same as LESS flow					
12	Flow - MORE	1) High level in T-102 2) LCV-0301 fails open 3) LE-0301 fails high	1) Centrifuge shuts down due to high torque	1) LAHH-0301 & XAH-0343 2) LAL-0301 & XAH-0343 3) XAH-0343	3 3 3	3 3 3	C C C	
	Flow - LESS	1) LE-0301 fails low 2) LCV-0301 fails closed	1) Liquid overflows to vent 2) Liquid overflows to vent	1) None 2) LAH-0301	3 3	3 3	C C	1) T-102 will be banging due to vapor hitting liquid level in T-102 and T-201 level increases at a greater rate than normal.
	Temperature - MORE	1) TCV-0312 fails closed 2) TE-0312 fails 3) CW none existent	1, 2 & 3) High feed temp. to extractor, C-301	1) TAHH-0312 & TAHH-0341 2) TAHH-0341 3) TAHH-0312 & TAHH 0341	3 3 3	3 3 3	C C C	
	Pressure - MORE	LCV-0301 fails closed	Level rises in T-102	LAHH-0301	3	3	C	
	Pressure - LOW	Lack NPSH in pump, P-102	Level rises in T-102	LAHH-0301	3	3	C	T-102 min. 5' elevation
	Erosion / Corrosion	Acid + M.O.C. compatability	Line failure	None	2	2	A	Investigate 316 SS versus Alloy20
13, 14	Flow - NO	Bridging in T-210	Can't remove wastes	Agitator designed to handle bridging	3	3	C	
	Flow - MORE	1) Excess caustic 2) Excess H_2O carryover 3) Excess $CaCO_3$ 4) Overfill drum /or Leave HV-0441 open	1) T-210 @ high pH 2) T-210 fills quicker 3) Higher pH @ T-210 4) Spill	1) T-210 designed to hold hazardous materials 2) LSH-0340 3) Minimum pH for $CaCO_3$ is 6. 4) Sump in area and cleanup process	2 3 3 3	2 3 3 3	A C C C	1) Consider changing to $CaCO_3$ Fed via auger
	Flow - LESS	Neutralizing chemical	pH not adjusted	None	3	3	C	Operating SOP for neutralized material to be neutralize - sample - adjust - sample
	Composition	Solids in liquid line	Solids in extractor	None	2	2	A	Add duplex filters downsteam of P-205
	Temperature - MORE	Heat of Neutralization	M.O.C. in T-210	None	3	3	C	Verify heat of mixing
15.	Flow - NO	1) FCV-0344 fails closed 2) P-205 fails	1) T-205 fills 2) T-205 fills	1) FAL-0344 / LAHH-0326 2) FAL-0344 / LAHH-0326	3 3	3 3	C C	

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - HIGH	FCV-0344 fails open	Effective extraction coefficient reduced.	1) LAL-0326 2) C-301's capacity is 4 times that of design.	3	3	C	
	Flow - LESS	HV-0325 open	Product to waste tank	None	3	3	C	Operating SOP for system start-up will require operators to verify hand valve positions
	Level - LOW	LE-0326 fails high	T-205 empties + P-205 runs dry	FAL-0344	3	3	C	
	Level - HIGH	LE-0326 fails low	LCV-0326 closes and T-205 fills	FAL-0344	3	3	C	
16.	Flow - NO	1) Nitrogen due to low pressure or PCV-0412 fails 2) No Aqueous flow due to LCV-0344 closed 3) No organic phase due to FCV-0630 closed	1) Loss of nitrogen blanketing 2) None 3) LA to acid stripper therefore loss of yield	1) None 2) HE-304 cools MTHF which makes it H_2O rich due to inverse solubility 3) LAH-0625	2 3 3	2 3 3	A C C	1) Add nitrogen rotameter and low pressure switch on feed line. 2) If either feed to the extractor fails, then stop all feeds.
	Flow - HIGH	1) Aqueous FCV-0344 open 2) MTHF FCV-0630 open	1)Effective extraction coefficient reduced. 2) None	1)LAL-0326 and C-301's capacity is 4 times that of design 2) LAL-0625 / FAH-0630	3 3	3 3	C C	
	Pressure - HIGH	1) PCV-0429 fails 2) HV-0436 closed	1) Possibly overpressure C-301 2) Possibly overpressure C-301	1) None 2) Verification of hand valve positions in startup/operating SOP	3 3	3 3	C C	1) Add rupture disc to N_2 lines 2) Hand valve to be lock open valve.
	Presssure - LOW	N_2 loss	See NO FLOW					
16.1	Flow - NO	HV-0432, HV-0412, HV-0601 closed	Possible flooding of C-301	None	3	3	C	Operating SOP for system start-up will require operators to verify hand valve positions
	Flow - LESS	HV-0427 left open	T-703 fills	None	3	3	C	Add level switch high to T-703
	Phase	Phase inversion	MTHF to acid stripper	AALL-0410	3	3	C	
17.	Flow - NO	1) No nitrogen due to low pressure 2) PCV-0412 fails closed	Loss on nitrogen blanketing	None	2	2	A	1) Add nitrogen rotameter and low pressure switch on feed line.
	Flow - HIGH	1) LE-0405 fails high 2) LCV-0405 fails open	1, 2) T-301 empties	1) None 2) LAL-0405	2 2	2 3	A C	1) Add independent level switch low on T-301 2) Nitrogen will prevent full vacuum

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - LESS	1) LE-0405 failslow 2) LCV-0405 fails closed	1) T-301 overflows 2) T-301 overflows	1) None 2) LAH-0405	2 2	2 3	A C	1) Add independent high level switch on T-301
	Pressure - HIGH	1) PCV-0429 fails fully open 2) HV-0420 closed	1) Overpressure T-301 2) Overpressure T-301	1) None 2) Verification of hand valve positions in startup/operating SOP	3 3	3 3	C C	1) Add rupture disc to T-301 2) Hand valve to be lock open valve.
	Pressure - LOW	LCV-0405 fails open	T-301 empties	LAL-0405	3	3	C	N_2 will prevent vacuum + operator will shut HV-0417
	Phase	More Aqueous in T-301	Overload MTHF stripper	AAH-0425	3	3	C	
18. 18.1	Flow - NO	1) No steam flow to HE-510 2) LCV-0405 fails closed	1) Flood C-510 2) Run column down to low level, then shut down	1) LAH-0703 / TALL-0724 2) LAH-0405 / LALL-0703	3 3	3 3	C C	1) Add LAHH-0703 to close LCV-0405
	Flow - MORE	1) LCV-0405 stuck open 2) LE-0405 fails high 3) HV-0702 left open	1) Loss vacuum in C-510 & MTHF in column bottoms 2) LCV-0405 wide open 3) Overload vacuum system	1) TAH-0724 / LAH-0703 / LAL-0405 2) TAH-0724 / PAH-0704 3) PAH-0710	2 3 3	2 3 3	A C C	1) Add PAHH to shut XV-0728 2) If position switch on valve differs from required operating position, then alarm. 3) Verification of hand valve positions in startup/operating SOP
	Temperature - MORE	Loss/lack of CW	Incomplete condensation in HE-511 and overload vacuum sys.	TAH-0709 / PAH-0710	3	3	C	
	Pressure - MORE	1) LCV-0405 fails open 2) VP-513 fails 3) Fire	1, 2 & 3) Loss vacuum in C-510 & overpressurization of C-510 due to MTHF in column bottoms	1) PAH-0710	3 3 2	3 3 2	C C A	Add rupture disc, PSE-0732, on vapor line of C-510
	Pressure - LESS	PCV-0710 fails closed	Additional vacuum pulled & C-510 may flood	None	3	3	C	Add PALL to PIC-0710
	Phase	Loss/lack of CW	MTHF vapor in vacuum system	PAH-0710 / TAH-0709	3	3	C	
18.2 18.3	Flow - NO	1) FCV-0702 fails closed 2) P-512 failure	1 & 2) System shutdown	1 & 2) LAH-0708	3	3	C	1) Add FAL on FIC-0702
	Flow - MORE	1) FCV-0702 stuck open 2) FE-0702 fails low	1 & 2) Excess liquid down column	1) FAL-0702 / LAL-0708 TAL-0724 / AAL-0723 2) LAL-0708 / TAL-0724 AAL-0723	3 3	3 3	C C	

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - LESS	1) FCV-0702 fails closed 2) P-512 failure	Same as NO flow					
	Pressure - MORE	1) LCV-0405 fails open 2) VP-513 fails 3) Fire	Same as MORE Pressure for Node 18.1					
	Pressure - LESS	No N_2 supply	Excessive vacuum on T-510 therfore tank collapses	None	2	2	A	Add rupture disc, PSE-0737, on T-510
	Composition	MTHF in bottoms	Explosive mixture in C-510 bottoms due to peroxide formation	None	2	2	A	SOP on sampling for peroxide formation to be incorporated
	Level - MORE T-510	**Left side** 1) XV-0718 or XV-0729 left open 2) FCV-0702 fails closed **Right side** 1) LE-0722 fails low 2) LCV-0722 fails closed	**Left side** 1) H_2O overflows to MTHF side 2) Same as NO Flow **Right side** 1 & 2) MTHF level rises and T-510 floods	**Left side** 1 & 2) LAH-0722 and the stream has another separation stage in T-415 **Right side** 1) LAH-0708 2) LAH-0708 / LAH-0722	3 3 3 3	3 3 3 3	C C C C	
	Level - LESS T-510	**Left side** 1) FCV-0702 fails open 2) FE-0702 fails low 3) XV-0729 fails closed 4) LE-0708 fails high **Right side** 1) LE-0722 fails high 2) LCV-0722 fails open	**Left side** 1 & 2) Same as MORE flow 3) Aq. layer disappears 4) T-510 empties **Right side** 1 & 2) P-511 cavitates	**Left side** 1& 2) Same as MORE flow 3) LAL-0708 / AAL-0723 4) AAL-0723 / FAL-0702 **Right side** 1) None 2) LAL-0722	3 3 3 3 2 3	3 3 3 3 3 3	C C C C C C	Add level switch high, LSH-0739, to T-510 right side
19.	Flow - NO	**Steam failure:** 1) XV-0720 closed 2) LCV-00720 closed 3) LE-0720 fails low 4) MPS boiler problem **Other sources:** 5) P-510 fails 6) HE-510 blockage	**Steam failure:** 1,2,3 & 4) T-510 bottoms temp low **Other sources:** 5) No circ. leading to possible tar blockage 6) Complete shutdown	**Steam failure:** 1,2 & 3) TAL-0724 4) TAL -0724 and PAL-7311 **Other sources:** 5) Pump run status and LAH-0703 6) TAL-0724 / LAH-0703	3 3 3 3 3 3	3 3 3 3 3 3	C C C C C C	

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - MORE	**Steam failure:** 1) LE-0720 fails high 2) LCV-0720 fails open **Other sources:** 3) FCV-0728 fails open 4) FE-0728 fails low	**Steam failure:** 1 & 2) Excess boil-up **Other sources:** 3 & 4) Excess feed to WFE, C-515 which will reduce LA quality	**Steam failure:** 1 & 2) TAH-0724 **Other sources:** 3) FAH-0728 / LAL-0703 4) LAL-0703	3 3 2 3	3 3 2 3	C C A C	Add an on/off valve, XV-0728, to isolate the LA takeoff during alarm conditions
	Flow - LESS	Same as NO flow						
	Flow - MISDIRECTED	HV-0731 left open	T-703 will overflow	1) LSH on T-703 as added in Node 16.1 2) LAL-0703	3	3	C	Verification of hand valve positions in startup/operating SOP
	Temperature - MORE	Excess steam	Same as MORE Flow		3	3	C	
	Temperature - LESS	Less steam	Same as LESS Flow		3	3	C	
	Level - MORE	1) FCV-0728 fails closed 2) FE-0728 fails high 3) Low boil-up	1 & 2) Same as LESS Flow (Other sources) 3) Same as LESS Flow (Steam failure)		3 3	3 3	C C	
	Level - LESS	1) FCV-0728 fails open or FE-0728 fails low 2) LE-0720 fails high or LCV-0720 fails open	1) Same as MORE Flow (Other sources) 2) Same as MORE Flow (Steam failure)		3 3	3 3	C C	
20.	Flow - NO	1) No chilled water supply 2) No process water supply	1) Loss of vacuum 2) Build-up of MTHF	1) Temperature and pressure alarms in column, C-510 - TAH-0709, TAH-0724 and PAH-0710. 2) Local flow indication	3 3	3 3	C C	2) Operator to frequently check rotameter for water flow and level glass for MTHF interface
	Flow - MORE	Excess process water supply	Additional H_2O in system	Process is designed to handle some excess water via T-101 level	3	3	C	
20	Temperature - MORE	High seal water temperature due to loss of chilled water or higher process inlet temperature	Possible loss of vacuum	PAH-0710	3	3	C	

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Composition	Excess MTHF vapor	Potential sxplosion	Continuous process water bleed into VP-513 and overflow of MTHF to T-415	3	3	C	
20.1	Flow - NO	Process water rotameter not open	MTHF build-up in VP-513	None	3	3	C	SOP to PW into VP-513 on all the line sample VP-513 separator
	Flow - MORE	1) FI-0638 open too much 2) PW rotameter on seal tank open too much	Overall excess PW in system	Process is designed to handle some excess water via T-101 level	3	3	C	
21.	Flow - NO	1) LCV-0722 fails closed 2) LE-0722 fails low 3) No subcoding of MTHF in HE-511	1) T-510 level rises 3) Pump cavitation due to low NPSH	1) LAH-0722 and LSH-0739 on T-510 as added in Node 18.2/18.3 2) LSH-0739 on T-510 as in Node18.2/3 3) None	3 3 2	3 3 3	C C C	Review NPSH calculations for P-511 Consider making P-511 a gear pump
	Flow - MORE	1) LCV-0722 open 2) LE-0722 fails high	1 & 2) T-510 empties and pump, P-511, cavitates	1) LAH-0722 and LSH-0739 on T-510 as added in Node 18.2/18.3 2) LSH-0739 on T-510 as in Node18.2/3	3 3	3 3	C C	
	Flow - REVERSE	1) P-511, pump trips 2) LCV-0722 fails open	N_2 flows to T-510	None	3	3	C	Show interlock for LCV-0722 to be open only if P-511 is running
	Flow - OTHER THAN	No subcooling of MTHF	Same as NO Flow					
	Temperature - MORE	Pump, P-511, in recirculating mode	Pump can not dissipate heat and trips	LCV-0722 closes	3	3	C	
	Pressure - LESS	PCV-0710 fails closed	Pump, P-511, cavitates	PAL-0710	3	3	C	Review NPSH calculations for P-511 Consider making P-511 a gear pump
22.	Addressed in Node 16.1							
23	Phase	Emulsion feedstock variations	Column disruption and shutdown	TAL-0603	2	2	A	Add PI with AAH

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Level - MORE T-415	**Right side** 1) LE-0625 fails low 2) FCV-0630 fails closed 3) P-415 failure **Left side** 1) LE-0624 fails low 2) LCV-0624 fails closed	**Right side** 1,2 & 3) MTHF level rises and T-415 floods **Left side** 1 & 2) Aqueous layer overflows and MTHF w/ excess water flows to the extractor.	**Right side** 1) FAL-0630 2) FAL-0630 / LAH-0625 3) FAL-0630 / LAH-0625 **Left side** 1) FAL-0641 2) LAH-0624 / FAH-0630 / FAL-0641	3 3 3 3 3	3 3 3 3 3	C C C C C	
	Level - LESS T-415	**Right side** 1) LE-0625 fails high 2) FCV-0630 fails open **Left side** 1) LE-0624 fails high 2) LCV-0624 fails open	**Right side** 1 & 2) MTHF may overload C-301 then will starve it. **Left side** 1 & 2) Aq. layer disappears and MTHF may flood the column	**Right side** 1) None 2) LAL-0625 **Left side** 1) FAH-0641 / AAL-0628 2) FAH-0641 / AAL-0628 / LAL-0624	3 3 3 3	3 3 3 3	C C C C	**Right side:** Add FAH to FIC-0630 **Left side:** None
24.	Flow - NO	1) P-416 failure 2) LCV-0624 fails closed 3) LE-0624 fails low 4) AAL-0628 fails	1-4) No feed to column	1) LAH-0624 / FAL-0641 2) LAH-0624 / FAL-0641 3) FAL-0641 4) LAH-0624 / FAL-0641	3 3 3 3	3 3 3 3	C C C C	
	Flow - MORE	1) LE-0624 fails high 2) LCV-0624 fails open	1 & 2) Level in column starts To build	1) FAH-0641 2) FAH-0641 / LAL-0624	3 3	3 3	C C	
	Flow - LESS	Same as NO Flow						
	Flow - REVERSE	While P-416 is shutdown, LCV-0624 is open	Static head of leg drains into T-415	None	3	3	C	
25	Flow - MORE	1) High feed rate 2) High boil-up rate	1 & 2) Potentially overload HE-415	None	3	3	C	
	Temperature - MORE	CWS lost	Vapor out to vent system	TAH-0622	3	3	C	
26.	Temperature - MORE	1) High pressure 2) High boil-up 3) P-410 stops	1) High pressure 2 & 3) High concentration of H_2SO_4	1) None 2 & 3) None	2 2	2 2	A A	1) Add a rupture disc with fusible element to C-410. (PSE-0642) 2) Change material of construction to CS lined with teflon, PVDF, glass lined for temperature and corrosion (HE-410 M.O.C - graphite or teflon??)

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Temperature - LESS	1) Low MPS flow	1) Possible MTHF in bottoms	1) TAL-0603	3	3	C	
	Corrosion	Same as MORE Temperature						
	Level - MORE	1) LE-0601 fails low 2) LCV-0601 fails closed 3) P-410 fails	1,2 & 3) Flood C-410	1) None 2) LAH-0601 3) LAH-0601	2 3 3	2 3 3	A C C	1) Add point level switch for high level to C-410
	Level - LESS	1) LE-0601 fails high 2) LCV-0601 fails open	1 & 2) C-410 pumped to low level	1) TAL-0603 2) TAL-0603 / LAL-0601 / LALL-0601	1 3	3 3	B C	1) Add FAL to FIC-0633
27.	Flow - NO	Same as MORE level for Node 26						
	Flow - MORE	Same as LESS level for Node 26						
	Flow - LESS	FE-0633 fails high	Not enough boil-up	TAL-0603	3	3	C	
	Temperature - MORE	Same as MORE temperature in Node 26						
	Temperature - LESS	Same as LESS temperature in Node 26						
	Composition	MTHF down column	MTHF enters the non-explosion proof area	TAL-0603	3	3	C	
	Corrosion / Erosion	H_2SO_4 aggressiveness	Line failure	None	1	2	A	PVDF, alloy 20 or teflon lined pipe
27.1	Flow - NO	1) LCV-0601 fails closed 2) P-410 stops 3) Plugging due to solids 4) LE-0601 fails low 5) Pressure drop to T-101 too high 6) Line blockage due to minerals salting out	1,2 & 3) No acid recycle to T-101 4) LCV-0601 closed 5) No flow 6) Plugging of FI-0626	1) LAH-0601 / FAL-0103 2) LAH-0601 / FAL-0103 3) FAL-0103 4) LSH added to C-410 in node 26 5) None 6) None	3 3 3 3 2 2	3 3 3 3 2 3	C C C C A C	3) Change pipe size to 1" 5) Change to a 2 valve arrangement to ensure pressure drop 6) Add an isolation valve for periodic cleaning of FI-0626
27.1	Flow - MORE	LCV-0601 fully open	Acid recycle temperature may increases and T-101 level will rise	TAH-0612 / LAH-0104	3	3	C	

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - MISDIRECTED	XV-0629 in diverted position	All acid recycle to neutralization	FAL-0103	3	3	C	
	Temperature - MORE	1) CWS TCV-0612 fails closed 2) TE-0612 fails low	1) Temp. Rises to T-101 2) Temp. Rises to T-101	1) TAH-0612 2) None	3 2	3 3	C C	2) Add TI to the existing themowell to T-101 for an operator to routinely log
	Corrosion / Erosion	H_2SO_4 aggressiveness	Same as Corrosion / Erosion in Node 27					
28.	Flow - NO	1) No solvent in T-415 2) P-415 stops 3) FCV-0630 fails 4) FE-0630 fails high	1-4) MTHF loop stops	In general, T-415 is designed to hold all MTHF inventory 1) FAL-0630 / LAL-0625 / FAL-0721 2) FAL-0630 / LAH-0625 / FAL-0721 3) FAH-0625 / LAH-0625 / FAL-0721 4) LAL-0625 / FAL-0721	3 3 3 3	3 3 3 3	C C C C	
	Flow - MORE	1) FE-0630 fails low 2) FCV-0630 fails open 3) FE-0630 fails in range	MTHF circulated through the system faster than normal. May overload some processes.	1) LAL-0625 2) LAL-0625 / FAH-0630 3) LAL-0625 / FAH-0630	3 3 3	3 3 3	C C C	
	Flow - LESS	FE-0630 fails in range	MTHF flow reduced therefore less extraction of LA product.	FAL-0630	3	3	C	
	Flow - MISDIRECTED	HV-0633 left open	MTHF emptys to T-703	T-703 designed to MTHF inventory and LAL's will activate	3	3	C	
	Temperature - MORE	1) TCV-0408 fails closed 2) TE-0408 fails low	1 & 2) Temp. to C-301 changes efficiency	1) TAH-0408 2) None	3 3	3 3	C C	2) Add a local temperature indicator on line 04016 so TCV-0408 can be manuallyset
29.	Flow - NO	1) XV-0731 fails closed 2) FE-0728 fails high 3) LE-0703 fials low	1) WFE runs dry 2) P-513 stops & WFE runs dry 3) WFE runs dry	1)FAL-0728 & LAH-0703 2) LAH-0703 3) FAL-0728	3 3 3	3 3 3	C C C	1) If position switch on valve differs from required operating position, then alarm. 2) Add an internal relief to P-513
	Flow - MORE	FE-0728 fails low	Overfeed WFE & decreases LA purity	LAL-0703 and verification of P-513 rpm	3	3	C	
	Pressure - HIGH	1) XV-0731 fails closed 2) Line blockage	1) P-513 deadheads 2) Possible rupture of T-515 (MOC is glass) on WFE	1) P-513 has an internal recycle 2) WFE inlet PIT to alarm and chut P-513 via software interlock	3 3	3 3	C C	Verify the wiped film evaporator high pressure rating
30	Flow - NO	1) HV-0809 & HV-0810 closed 2) HV-0827 & HV-0828 closed	1) Overpressurization of the tar line 2) Overpressurization of the LA product line	1, 2) None	2	3	C	1) Ensure that P-515 & P-520 have internal reliefs 2) Change the hand valves to solenoids which are operated by a hand switch to always ensure that one valve is open.

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Flow - MORE	Increased output to the tar & LA product drums	Overfilling of drums	For tar - LAH-0812 For LA product - LAH-0835 & FQAH-0834	3	3	C	Consider making the drum level switches to level transmitters so drum level can be monitored as well as warnings given.
	Temperature - MORE	1) Tar always enters drums hot 2) LA product cooler losses chilled water	LA product and tar drums are very hot and potentially an operator hazard.	1) Tar drums have an isolation barrier to keep personnel away. 2) LA product - NONE	3 3	3 3	C C	
	Temperature - LESS	WFE heating element failure	Line blockage of tar lines	Heat traced tar lines	3	3	C	
	Level - MORE	See MORE flow						
31 T-702	Flow - NO	Bottom outlet plugged due to solids carryover.	Cannot empty T-702	None	2	3	C	Add a tee with blind flange on outlet line and ensure that the tank has a 2"low level nozzle with isolation valve.
	Pressure - MORE	HV-7130 closed	Overpressurization of T-702	None	2	3	C	1) Make HV-7130 a lock open valve 2) Add an overflow line w/ loop seal going to line 71012
31 T-703	Level - MORE	XV-7137 left open	Overfill organic waste drum	None	2	2	C	1) Add level switch to drum to provide the operator with a warning
	Temperature - MORE	C-410 emptied while hot	High temperature in T-703 and potentially an operator hazard.	None	2	3	C	1) Add local temperature indicator to T-703
	Pressure - MORE	1) Overfill T-703 2) HV-7140 closed 3) PCV-7118 failed closed	1) Liquid overflows into the vent system 2, 3) Overpressure of T-703	None	2	3	C	1) Add rupture disc to T-703 2) Ensure HV-7140 is a locked open valve
31 T-704	Temperature - MORE	One of the reactors relieved into T-704	High temperature in T-704 and potentially an operator hazard.	None	2	3	C	1) Add local temperature indicator to T-704
31 T-701	Flow - NO	1) P-710 stops running 2) P-711 stops running 3) P-704 stops running	1) T-701 pH is low in first compartment 2) T-701 pH is low in second compartment 3) Level increases in T-701	1) AAL-7102 2) AAL-7103 3) LAHH-7106	3 3 3	3 3 3	C C C	
	Flow - MORE	1) AIC-7102 fails low 2) AIC-7103 fails low	1) T-701 pH is low in first compartment 2) T-701 pH is low in second compartment	1) AAH-7103 2) None	3 2	3 3	C C	Add a redundant pH meter in the neutralized waste exit line.

Node	Deviation	Causes	Consequences	Safeguards	S	F	R	Recommendations
	Temperature - MORE	1) HE-103 not subcooling enough 2) T-102 emptied without going through HE-201	Temperature limits of T-701 exceeded	None	2	2	A	1) Add temperature indicating transmitter to line 03021 2) Add temperature indicator to T-702 to ensure that the liquid is cooled prior to transfer to T-701 3) Investigate material of construction alternates for T-701 such as rubber lined or fiberglass lined CS.
	Level - LESS	XV-7106 fials wide open	T-701 pumped down	None	3	3	C	
	Level - MORE	1) P-704 stops running 2) XV-7106 fails closed	Overfill T-701	LAHH-7106	3	3	C	

PLANT
OPERATIONS

BIOFINE DEMONSTRATION PLANT

OPERATIONAL PROTOCOLS

1. The plant was operated 24 hours per day, four days per week. This work schedule allowed two operating teams to work four twelve- **Feedstock Average Values** hour shifts with a long weekend. The staffing complement (and qualifications) were as follows:

   - Four operators total organized into two shifts of two working twelve hours with a thirty minute overlap for shift hand-over (chemical plant operating certification and experience)

   - A plant manager and an assistant plant manager (mechanical/chemical engineers)

   - A day-shift laboratory analyst (professional chemist)

   BioMetics Inc. provided engineering technical support, as required.

2. Plant operational campaigns were targeted to run for a total of 96 hours including start-up, steady state and shut-down. Maintenance or equipment inspection was scheduled for the fifth day. Each week, prior to start-up, plant operating conditions for that week were set in the "Plant Manager Run Sheet". Operations that reached steady state for at least 30 hours were reported as operational data.

3. Steady state was adjudged to have occurred when plant conditions were stable and stream analyses changed less than 10% for three consecutive samplings.

4. During operations the computer monitoring system logged all plant data. A summary of the data logging points is included in this section. The plant operation was also monitored via a routine stream sampling and off-line chemical analysis. HPLC and GC analytical methods were used for routine analysis. All chemical methods were validated using an independent analytical laboratory at Dartmouth College, Hanover, NH. A summary of the routine sampling schedule is included in this section.

5. The plant processed a range of feedstocks. Prior to processing, the feedstock was characterized via a series of tests. These were:

   - Cellulose content (via the Quansac method)
   - Moisture (loss on oven drying)
   - Ash (residue)

- Titratable alkalinity (titration against acid)

All chemical/analytical techniques are described in this section.

6. Prior to and regularly during operations, the plant was inspected by insurance underwriters and by OSHA representatives. Also, local representatives of NY State Department of Environmental Protection inspected the operation. All required operating permits were granted.

7. Operation procedures were written for the operation and maintenance of the South Glens Falls demonstration plant. In addition to the a 155 page operating manual, additional 80 procedures were written, for use as follows:

Personnel procedures	9
Laboratory procedures	23
Operating procedures	14
General procedures	20
Report forms	14

The following table gives the title of each of these documents.

Item No.	Title		Document Number
Personnel Procedures			
0	Initial Training Protocols		PP-00
1	Operations Manual		PP-01
2	Job Descriptions -	Assistant Plant Manager	PP-02A
		Senior Operator	PP-02B
		Junior Operator	PP-02C
3	Shift change Protocol		PP-03
4	Disciplinary Procedures & Personnel Records		PP-04
5	Personnel Protective Equipment (PPE) Protocol		PP-05
6	Continuing education, training, and assessment		PP-06
7	Visitors and Site Access		PP-07
8	Safety Manual		PP-08
Laboratory Procedures			
1	Not assigned		LP-01

2	Levulinic Acid Assay via Titration	LP-02
3	Not assigned	LP-03
4	Determination of Ash Concentration in Feedstock	LP-04
5	Quantitative Saccharification for Feedstock Analysis	LP-05
6	Feedstock Titratible Alkalinity Procedure	LP-06
7	Determination of MoistureContent in Feedstock/Residue	LP-07
8	Karl Fisher Moisture for Product	LP-08
9	Determination of Peroxides in MTHF	LP-09
10	Not assigned	LP-10
11	Hazard Communication & Labeling for the Laboratory	LP-11
12	Disposal of Laboratory Wastes Protocol	LP-12
13	Process Sampling Procedure	LP-13
14	Standard Laboratory Operating Procedures	LP-14
15	HPLC: Sulfuric Acid Determination	LP-15
16	HPLC: Levulinic Acid Determination	LP-16
17	HPLC: General Procedures	LP-17
18	Not assigned	LP-18
19	Chemical Storage in Laboratory	LP-19
20	Certificate of Analysis Preparation	LP-20
21	External Validation of Analytical Procedures	LP-21
22	GC: General Procedures	LP-22
23	GC: Capillary Column	LP-23
24	GC: Stainless Steel Packed Column	LP-24
	Operating Procedures	
1	Sulfuric Acid - Storage & Handling	OP-01
2	Sodium Hydroxide - Storage & Handling	OP-02
3	Levulinic Acid - Storage & Handling	OP-03
4	Solvent (MTHF) - Storage & Handling	OP-04
5	Helium Cylinders - Storage & Handling	OP-05
6	Hydrogen Cylinders - Storage & Handling	OP-06

7	Air Cylinders - Storage & Handling	OP-07
8	Tar By-product - Storage & Handling	OP-08
9	Cellulose Feed Stock - Storage & Handling	OP-09
10	Routine Operations & Daily Log Protocol	OP-10
11	Facility Storage Protocol - Layout & Locations	OP-11
12	Data Handling and Reporting	OP-12
13	Computer Back-up	OP-13
14	Record Keeping and Fire files	OP-14
15	Not assigned	
16	Nitrogen Purging Protocol	OP-16
	General Procedures	
1	Change control protocol	GP-01
2	Lockout/Tagout Procedures	GP-02
3	Ladder, Elevated Platforms & Scaffolding Procedures	GP-03
4	Preventative Maintenance Procedures and Calibration	GP-04
5	Maintenance & Sanitization of Building & Facilities	GP-05
6	Contingency Plan for Emergencies	GP-06
7	Contingency Plan for Chemical Spills, Undefined Spills and/or Residual Clean-up Procedures	GP-07
8	Receipt, Inspection & Quarantine of GMP Materials	GP-08
9	Ordering of Raw Materials & Supplies	GP-09
10	Assignment of Tracking Numbers	GP-10
11	Fire Extinguisher Usage Protocol	GP-11
12	Back Injury Prevention Protocol	GP-12
13	Confined Space Entry Protocol	GP-13
14	Hazard Identification Protocol (Wall Chart)	GP-14
15	Evacuation Plan	GP-15
16	MSDS and Chemical Hazard Document Handling	GP-16
17	Plant Access	GP-17
18	On Site Security	GP-18
19	Emergency Management Plan	GP-19

20	Not assigned	
21	Material Specification Protocol	GP-21
Report Forms		
1	Employee Safety Suggestion Form	RF-001
2	New Employee Safety Checklist Form	RF-002
3	Employee Safety Record	RF-003
4	Receipt/Acknowledgement of Training regarding PPE (XXX stand for operator's initials)	RF-004-XXX
5	Notice of Safety Infraction	RF-005
6	Minutes of Safety Meetings	RF-006
7	Employee's Accident Report Form	RF-007
8	Accident Investigation Report Form	RF-008
9	Accident Prevention program Evaluation	RF-009
10	Pressure Inspection Record Form	RF-010
11	Tracking Number Log	RF-011
12	GMP Receiving Log	RF-012
13	Pressure Test Form	RF-013
14	Performance Planning & Evaluation	RF-014

OPERATIONS RESULTS

The demonstration plant was started up in April 1997 and operated under this program until March 1999. Feedstock materials fed to the plant during this time included paper sludge from various paper mills and a sample of sorted municipal solid waste from New York City.

Results

The results are reported as follows:

- Feedstock Characterization – Showing analytical data characterizing a feedstock
- Production Run Summary Sheets – Showing overall weekly inventory changes, and overall levulinic acid yields
- Instantaneous material balances showing instantaneous material flows and levulinic acid yields and recoveries.

A summary of the plant yields for levulinic acid is provided below. The target yield was set at 50%, by weight (0.5 lbs levulinic acid per pound of cellulose). This is the equivalent of 70% of the theoretical maximum yield which is 71.6%. Raw operations data is provided in the report appendix.

In all, paper sludge from twelve different sources was tested and three were successfully processed over extended periods of time. In addition, approximately four tons of municipal solid waste from New York City was successfully processed in one operation.

A summary table of the successful operations is provided below:

South Glens Falls Operations and Summary of Results

Plant operations were carried out on various feedstocks from 8-97 until 8-98.

The following table summarizes the yield of levulinic acid obtained during operations running on several different feedstocks. The most stable operation was experienced on Kimberly Clark sludge and Softwood Kraft fiber. Other feedstocks were processed but steady state was not achieved for the target 30 hours. Steady state was also achieved processing separated municipal waste fiber.

Instantaneous and overall yields were measured. Overall yields were calculated by actual inventory changes of feedstock and crude product. Instantaneous yields were calculated based on sample analysis and stream flowrate measurement.

Run date	Steady State Duration (h)	Instantaneous Yield (lb/lb)	Overall Yield (lb/lb)	Feedstock Processed	Comments
8/97 – 9/97	N/A	0.4 to 0.48	0.42	Benetech Peach	Sustained steady state not achieved
9/11/97	80	0.532	0.53	Kraft	Instantaneous yield numbers only
9/16/97 – 9/18/97	80	0.424 to 0.525	0.50	Kraft	Instantaneous yield numbers only
9/23/97	80	0.39 to 0.513	0.42	Kraft	Instantaneous yield numbers only
2/9/98 – 2/12/98	--	- --	0.446	Kimberly	Sludge analysis 58% cellulose
2/23/98 – 2/26/98	41	0.496	0.583	Kimberly	Sludge analysis 58% cellulose
3/2/98 – 3/6/98	32	0.426 to 0.503	0.595	Kimberly	Sludge analysis 58% cellulose
3/98 – 4/98	N/A	N/A	N/A	Irving	Reactor plugging stopped run
5/98 – 8/98	48	0.34	0.31	Municipal Waste	See report included in with this report

The following tables provide information on the feedstocks provided by various collaborating companies and tested during the program:

Feedstock Average Values

Feedstock	Tag	Moisture gm moisture/ gm wet sample	Ash gm ash/ gm dry sample	Titrateable Alkalinity gm H_2SO_4/ gm dry sample	Cellulose gm cellulose/ gm dry sample
Soft wood Kraft	SWK	0.06	0.00		0.93
American Tissue Mechanicsville	ATM	0.16	0.16	0.035	
Benetech Peach	BT	0.29	0.25		
Benetech Vichey	BTV	0.38	0.13	0.026	
IP - Augusta	IP-Augusta	0.36	0.23		0.34
IP - Androscogin	IP-Adro	0.62	0.33		0.21
IP - Mansfield	IP-ManCF	0.72	0.07		0.42
IP - Mansfield	IP-ManPF	0.84	0.12		0.35
IP - Natchez	IP-Nat	0.62	0.55		0.19
IP - Pine Bluff	IP-PB	0.74	0.25		0.21
IP - Pineville	IP-Pine	0.67	0.28		0.28
IP - Riegelwood	IP-Rie	0.58	0.13		0.72
IP - Thilmany	IP-Thilmany	0.65	0.15		0.40
Ticonderoga	TIC	0.77	0.12		0.40
Irving paper	IRV	0.62	0.11		
Kimberly Clark	KC	0.44	0.07		0.58
Kimberly Clark - hydrofuser	KCF	0.32	0.06	0.085	0.58
Marcel	Marcel	0.65	0.39		
Refuse derived feedstock	RDF	0.12	0.12	0.069	0.49

Feedstock Run Dates & Comments

Feedstock	Tag	Approx. run dates	Comments
Soft wood kraft	SWK	9/97, 10/97, 11/97, 12/97, 2/98	Baseline substrate; consistently processable.
American Tissue Mechanicsville	ATM	5/98	Processing was discontinued after material was found to contain gravel.
Benetech Peach	BT	8/97	Processing discontinue due to insufficient supply of material.
Benetech Vichey	BTV	9/97	Foreign material (latex) caused difficulty with centrifuge operation.
IP - Augusta	IP-Augusta	not processed	Sample sent for analysis only.
IP - Androscogin	IP-Adro	not processed	Sample sent for analysis only.
IP - Mansfield	IP-ManCF	not processed	Sample sent for analysis only.
IP - Mansfield	IP-ManPF	not processed	Sample sent for analysis only.
IP - Natchez	IP-Nat	not processed	Sample sent for analysis only.
IP - Pine Bluff	IP-PB	not processed	Sample sent for analysis only.
IP - Pineville	IP-Pine	not processed	Sample sent for analysis only.
IP - Riegelwood	IP-Rie	not processed	Sample sent for analysis only.
IP - Thilmany	IP-Thilmany	not processed	Sample sent for analysis only.
Ticonderoga	TIC	not processed	Sample sent for analysis only.
Irving paper	IRV	3/98, 4/98	High ash content prompted precipitation reaction in reactor. Processing discontinued due to repeated blockages.
Kimberly Clark	KC	2/98, 3/98	Consistently processable.
Kimberly Clark - hydrofuser	KCF	3/98, 4/98	Consistently processable; fluffy consistency caused sporadic solids feeding problems.
Marcel	Marcel	not processed	Sample sent for analysis only.
Refuse derived feedstock	RDF	6/98, 7/98	Required sorting to remove large solid objects. Ran at high solids concentrations with the addition of a flow aid/antifoaming agent.

PROCESS PERFORMANCE ANALYSIS

1. **Process stability:** The overall integrated process operated consistently and stably on both fresh paper stock (softwood Kraft wet lap) and paper sludge (ex Kimberley Clark) waste for periods in excess of 30 hours per campaign. Both the hydrolysis and the recovery/purification system operated separately at steady state over periods in excess of 50 hours. However, maintaining simultaneous stable operation of the reactor system and the solvent extraction recovery system proved difficult for a shift operating staff complement of only two operators per shift. Two auxiliary staff engineers from BioMetics were required to ensure adequate operational cover during operations.

   To improve process stability and operability it was decided to alter the process by taking the major acid recycle flow back to the feed mixing tank from the centrifuge rather than from the acid stripper recovery column. This led to a more stable reactor system since it had the effect of decoupling its operation from that of the recovery section. In addition, it improved the operation of the solvent recovery section since it now received a much higher levulinic acid concentration due to the build-up of levulinic acid in the recycle.

   Recycle of the reactor output had been tried previously during laboratory operation at Dartmouth College, but results were inconclusive. Operation of the SGF plant in this mode showed good benefits. The following memos from BioMetics' engineering staff provide further analysis of this significant improvement.

   Typical Operating parameters for the process are contained in the plant manager run sheet issued by the plant manager to the operating staff at the start of the run.

Typical major operating parameters are provided in the process description section and are as follows:

Operating Parameter	Value	Comments
Sludge feed rate	85 pounds/hour (actual)	9% moisture
Acid recycle flow	3.0 gallons/min	
PFR Temp	428 Deg F.	Controls HP steam flow
PFR residence time	12 seconds	Pump federate 3.1 gpm
PFR pressure	390 psig	Over 385 psig
CSTR Temp.	392 Deg. F.	
CSTR residence time	25 minutes	40 inches in reactor
CSTR pressure	200 psig	
Acidity (sulfuric acid)	3%	Make up controlled by pH
Extraction solvent ratio	2.5 lb/lb	
Acid stripper steam rate	1000 lb per hour	
Solvent stripper steam rate	1200 lb per hour	

In addition, feedstocks from other sources were also used including municipal waste from New York City. A full list of the feedstocks used is included in the Testing section of this report. Most of these feedstocks ran successfully for a limited period of time. In these instances, the duration of the runs were limited by mechanical failures of equipment rather than for any process or yield problems. Data from these operations are not reported since the plant was not observed to be at steady state. A report on the New York City municipal waste processing is included later in the report.

The modified recycle process was modeled using a Microsoft Excel spread sheet. The results of a typical simulation are shown on the following pages:

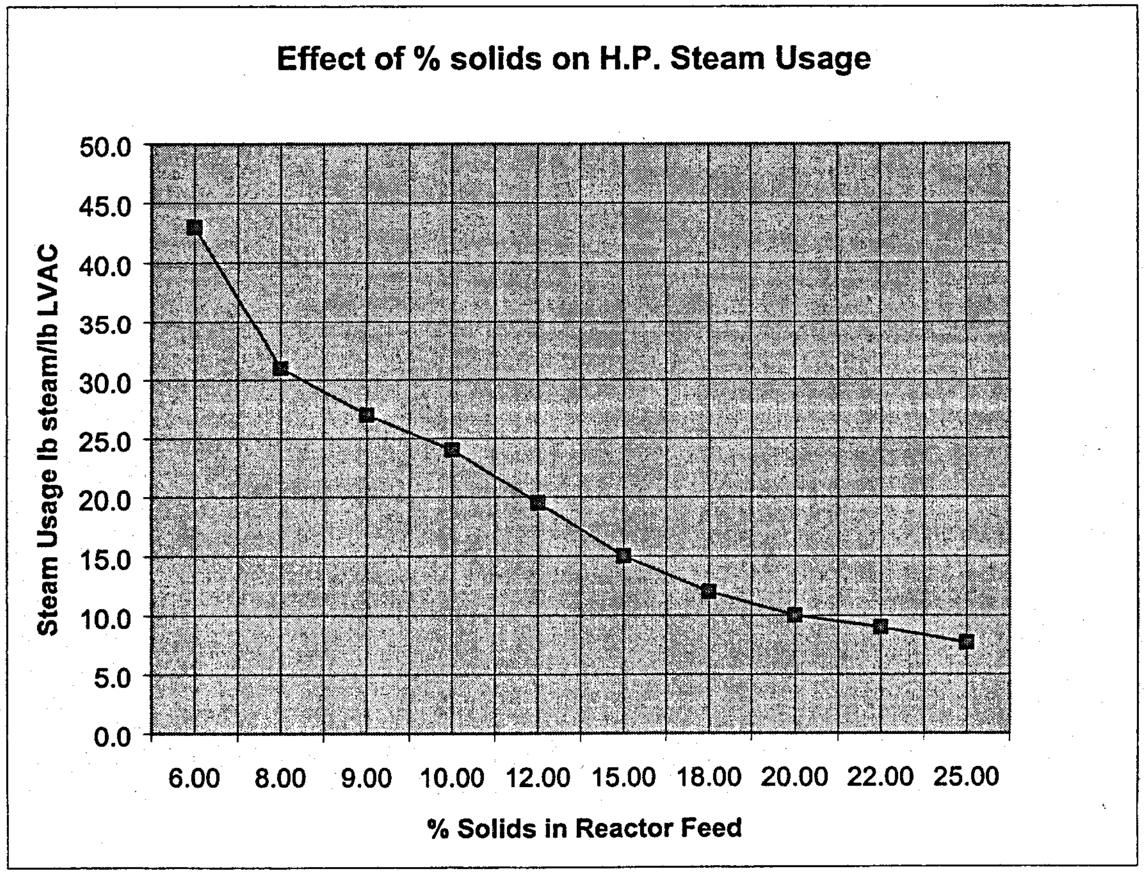
2. **Process Energy Usage:** The main points of energy usage in the process were as follows:

   - The high pressure steam to the plug flow reactor
   - Low pressure steam to the recovery section
   - Electrical energy for pumps and other rotating equipment

   At steady state typical values experienced for the above energy usages are as follows (Target numbers for a commercial process unit are shown above in parentheses.):

Usage type	Lb/hour	lb/lb levulinic acid	KW	KW hrs/lb levulinic acid
H.P. Steam	550	31 (target = 9)		
L.P. Steam	570	35 (target = 10)		
Electrical			300	20 (target = 1.5)

It is anticipated that these numbers would be reduced in a larger scale plant due to appropriate heat integration and higher consistency feeds. Calculations on the effect of feedstock concentration on high-pressure steam usage are summarized graphically below:



Average Process Mass and Heat balances: The average values for energy inputs and outputs were calculated over a series of steady state operations. The numbers are presented below:

MAJOR INPUTS:

Feedstock	470,080	@8,000 BTU per pound
H.P. Steam	550,000	@31 lbs/lb LVAC
L.P. Steam	550,000	@35 lbs/lb LVAC
Electricity[1]	515,000	@ 3550 BTU per KWhr
TOTAL (BTU per hour)	**2.1 million BTU per hr**	

MAJOR OUTPUTS:

Levulinic acid	152,000	@9,700 BTU per pound
Formic acid	30,000	In waste water @4500 BTU per pound
Tar (ash-free)	301,000	@10,000 BTU per pound (dry, ash-free basis) 40% moisture
Cooling water discharge	1,500,000	Estimated - 300 gpm and 10 Deg. F. temp. rise
TOTAL (BTU per hour)	**2.0 million BTU per hr**	

It is interesting to note in the table above that 64% of the BTU value of the feedstock is retained in the Tar component of the process output. This tar component is hydrophobic and similar to powdered coal in physical appearance. It has been shown to be a superior boiler fuel to hydrophilic paper sludge exhibiting an improved efficiency of combustion due to elimination of slagging and the need to de-water. When improved efficiency of combustion is accounted for it is likely that more energy output can be obtained from a boiler burning the waste tar than from the original sludge. This implies that in applications in paper mills burning paper sludge for energy the Biofine process can extract valuable chemicals (in the form of levulinic acid and formic acid) while, at the same time *increasing* the output from the same boiler.

---

[1] Electricity consumption is net of electrical energy required for HP steam production

3. **Raw Material Feeding**: The original SGF design encompasses a Flexicon-type feed solids metering system. This depends upon two screw augurs: One feeding from the bag hopper into a weigh hopper and one feeding from the weigh hopper into the feed tank T-101. This system worked well with feeds of high quality. However, there was no way of separating rocks and tramp metal that often occurred with poor quality feeds. These hard inert materials reduced the life of the Moyno feed pump and, in extreme cases, blocked the plug flow reactor pressure letdown valve. During the program the second screw augur was replaced with an air conveying system. This consisted of an "Ex-air" high pressure air injection system. This caused air entrainment of the feedstock particulates. Harder, denser material was left behind in the feed hopper. A manual removal system was installed at the feed hopper to remove collected tramp material. The scaled up plant will include this air conveying system.

   It was also found beneficial to grind fresh feedstock material to remove tramp material before feeding. The grinder provided a second route for removing tramp material. This will also be installed in the larger scale plant.

4. **Moyno feed pump modifications:** The Moyno pump with a ceramic rotor and Viton elastomer stator gave reliable service with consistent flowrates and pressures. The maximum consistency of feed reached was 6% by weight (dry basis). (This is equivalent to around 3% cellulose). Above this consistency the pump showed increasing indications of cavitation evidenced by variations in output flowrate, feedstock de-watering and audible "groaning". Cavitation is undesirable since it accelerates wear on the stator and causes variations in feed-rate and feed consistency. The primary cause of the cavitation was found to be due to the knuckle connection between the drive and the rotor. This was located at the fluid entrance to the stator and acted as a partial blockage with high consistency feeds.

   Due to this pumping limitation a maximum of 80 lbs per hour of solids at the maximum pump flow of 2.5 U.S. gallons per minute. This gave a daily output of 1920 lbs, or 94% of the target process rate of 2000 pounds per day.

   As a result of discussions with the pump manufacturers (Robbins and Myers) the design of the pump was modified to reposition the drive knuckle further towards the rear of the entrance hopper and away from the entrance to the stator. A modified larger pump was purchased and installed. This pump appears to be capable of feeding up to 15% consistency feedstock.

   Higher consistency feeds to the reactor have the benefit of significant reduction in high pressure steam usage. In the scaled up design a twin screw feeder will be used to feed the Moyno pump. This is expected to allow consistencies of over 20% to be fed.

5. **CSTR (R-102) design:** During commissioning the pressure stability of R-102 proved highly sensitive to the condensing rate in its condenser T-101. This was found to be due to the large over-design factor used in the condenser design. This situation was corrected by plugging of 50% of the condenser tubes to reduce condensing rate. With this modification, R-102 and its condenser system showed good stability.

   The materials of construction of R-102 were a continual source of problems. The initial design was 316L stainless steel with a Teflon interior lining for process fluids contact surfaces. Although the lining showed good resistance to the process conditions, "flow" or movement of the lining proved to be a serious problem leading to frequent tearing of the lining. Due to this problem R-102 was removed for re-lining on two occasions.

   As a result of this experience the material of construction of R-102 was upgraded to zirconium in order to eliminate the Teflon lining and

6. **Centrifuge design:** During operations the main centrifuge bearings were found to fail frequently due to corrosion. The cause was identified to be hot formic acid vapors resulting from reduced cooling capacity of T-103. Although the maximum temperature limitation for feed to the centrifuge was set at 110 Deg. F. the temperature frequently exceeded this specification.

   Two changes were made to remedy this situation: Firstly, the design of the centrifuge bearings was modified to include a pressurized oiling system and a fan to force air flow away from the bearings into the process; secondly, the process side fluid velocity in HE-201 was increased by reducing the number of tubes. This had the effect of increasing the tube-side heat transfer in HE-201 due to establishment of turbulent flow through the tubes.

7. **PC-202 modifications:** The initial pressure control valve design for PC-202 was a ball valve with a characterized "birds head" aperture. This design allowed a small Cv during normal flow conditions but could open rapidly to allow passage of large inert materials such as rocks or metal objects. During operation it was found that the lifetime of these valves were limited to approximately one month of useful operation due to both erosion of the valve aperture and "flogging" or excessive wear of the actuator spindle in the plug. This limited lifetime was manageable in the demonstration plant but would not be economic in a commercial scale plant. The design of the valve was changed to a plug type configuration with a hardened ceramic impingement surface. This configuration appears to be giving acceptable service life.

8. **Solvent-free purification process:** A modified process for levulinic acid recovery was developed after this program had been concluded. This process eliminated the MTHF solvent extraction step. In place of solvent extraction the process now includes a two-stage evaporation. Stage One involves dehydration of the tar stream from the density separation step. Stage Two involves vacuum evaporation of the levulinic acid in the dehydrated tar output from Stage One. The advantages of this process are that there is no solvent usage and the tar is produced as a bone dry byproduct from Stage Two. The recycle loop process can be balanced to increase the concentration of levulinic acid in the centrifuge tar slurry stream to the point where the levulinic acid in this stream is equal to the levulinic acid input to the loop.

9. **Wiped Film Evaporator Operation:** Operation of the wiped film evaporator as a finishing unit operation was inconsistent. The intent of this operation was to evaporate levulinic acid from the residue from the solvent recovery column under vacuum (5 mm Hg). The products from the column were levulinic acid (overhead) and a tarry residue (bottoms). During operation this step had three major drawbacks:

   - It was found that the viscosity of the tarry residue was too high for the bottoms discharge pump to pump out against the vacuum. This prevented consistent operation of the unit. The bottoms pump was replaced with a batch "lock hopper" arrangement. This worked reasonably well, improving reliability of operation.
   - Residual water in the crude levulinic acid stream from the solvent recovery stream tended to overload the WFE vacuum system causing loss of vacuum. This was alleviated by dehydrating the product with hot air in the air stripper column.
   - Residual formic acid in the levulinic acid from the solvent recovery column corroded the vacuum pump. This was alleviated by installation of a liquid nitrogen "ice trap" in the vacuum line. Losses of liquid nitrogen would preclude this measure from being used in a commercial plant.

   As noted above, the solvent extraction/recovery system has been replaced with an evaporation system. This has alleviated the above-reported problems with the WFE unit.

10. **Corrosion Issues – Materials of Construction:** The process operates with elevated temperatures (200 to 420 Deg. F.) and dilute sulfuric acid (2% to 3% by weight). In addition, moderately erosive slurries of paper sludge were being handled (paper sludge consists of cellulosic fibers and inorganic particulate) at some points at high velocity. Materials of construction were a major concern. Alternative materials of construction were tested for the unit operations handling combinations of high temperature, acidity, and erosive slurries. The main points of concern for corrosion/erosion in the process were as follows:

    - The feed mix tank (T-101)
    - The plug flow reactor (R-101)
    - The plug flow reactor pressure control valve (PCV- 202)
    - The second stage CSTR reactor (R-102)
    - The second stage reactor level control valve (LCV- 205)
    - The line from the second stage reactor to the flash vessel (T-102)
    - The acid stripper recovery column (C- 410)

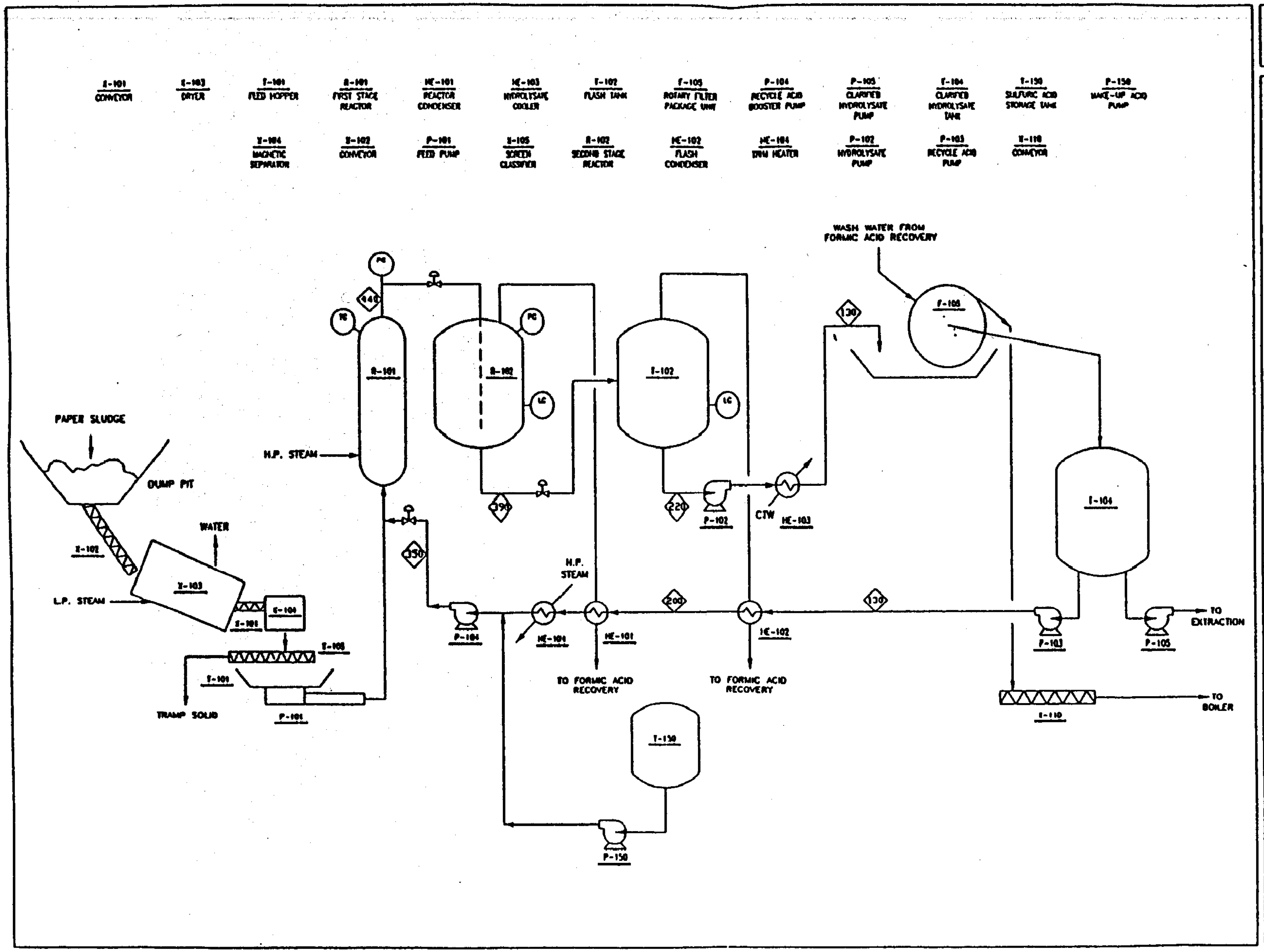
    The following table describes the basis for selection for materials of construction for the above-mentions plant sections:

Unit	Initial M.O.C.	Basis for initial selection of M.O.C.
T-101	316L stainless steel	Acidity – up to 5% sulfuric acid Temperature – up to 135 Deg. F. Fiber/inorganic slurries – up to 15 wt.% Moderate agitation (100 rpm)
R-101	Zirconium	Acidity – up to 5% sulfuric acid Temperature – up to 450 Deg. F. Fiber/inorganic slurries – up to 15 wt.%
PCV-202	Zirconium ball with characterized trim	Acidity – up to 5% sulfuric acid Temperature – up to 480 Deg. F. Fiber/inorganic/char slurries – up to 10% High discharge velocities – up to sonic
R-102	Teflon-lined 316L stainless steel	Acidity – up to 5% sulfuric acid Temperature – up to 350 Deg. F. Fiber/inorganic/char slurries – up to 10% Vibration from dip-pipe vapor discharge
LCV-202	Zirconium ball with characterized trim	Acidity – up to 5% sulfuric acid Temperature – up to 350 Deg. F. Fiber/inorganic/char slurries – up to 10% High discharge velocities – up to sonic
T-102 feed line	Alloy 20 stainless steel	Acidity – up to 5% sulfuric acid Temperature – up to 350 Deg. F. Fiber/inorganic/char slurries – up to 10% High discharge velocities – up to sonic
C-410	Teflon-lined carbon steel	Acidity – up to 5% sulfuric acid Temperature – up to 220 Deg. F. Direct steam injection Presence of MTHF solvent.

The experience gained concerning the performance of the selected M.O.C. and substitute materials is provided in the following table:

Unit	**In-service experience**	**Alternative M.O.C. used/recommendations**
T-101	316 L stainless steel performed well with no apparent pitting or excessive corrosion	None
R-101	Zirconium performed well with no apparent pitting or excessive corrosion	
PCV-202	Initial valve selection performed poorly with limited life of CV due to erosion and "flogging" out of the actuator stem socket in the ball. Typical life of valve due to erosion was three weeks. Failure indicated by loss of acceptable pressure control.	Various valve configurations were tried. The most successful found is a custom-built valve with a characterized zirconium body plug valve with hardened ceramic seat and plug and zirconium stem with sacrificial zirconium inserts at the wear points. This valve has provided satisfactory service, to date.
R-102	Teflon lining exhibited frequent tearing due to "cold-flow" of Teflon.	New R-102 installed which is 100% zirconium. This has provided acceptable service, to date.
LCV-202	Initial valve selection performed poorly with limited life of CV due to erosion and "flogging" out of the actuator stem socket in the ball. Typical life of valve due to erosion was three weeks. Failure indicated by loss of level control.	Various valve configurations were tried. The most successful found is a custom-built valve with a characterized zirconium body plug valve with hardened ceramic seat and plug and zirconium stem with sacrificial zirconium inserts at the wear points. This valve has provided satisfactory service, to date.
T-102 feed line	Pinhole leaks experienced due to erosion/corrosion at bends.	Eliminated sharp bends in line. Points where line changes direction are specified to have heavier gauge alloy 20. This has eliminated problem, to date.
C-410	Teflon lining exhibited frequent tearing due to "cold-flow" of Teflon.	Requirement for stripper column eliminated from process.

COMMERCIAL
SCALE DESIGN



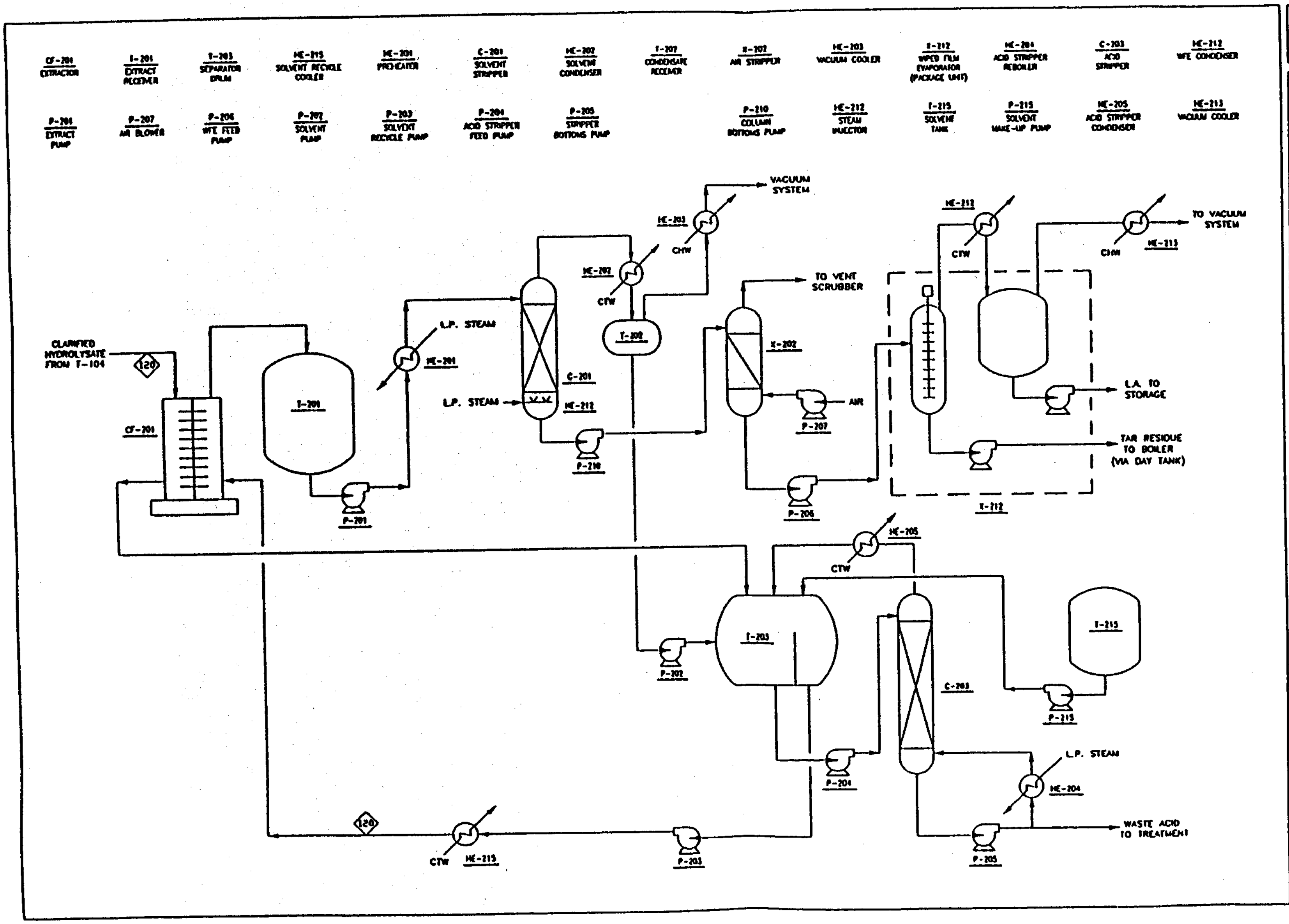
BioMetics

BIOFINE/PENCOR

TITLE
L.A. PLANT
1000 WET TONS/DAY
PAPER SLUDGE FEED

FEED HANDLING,
REACTORS + SEPARATION

PFD-001



BioMetics

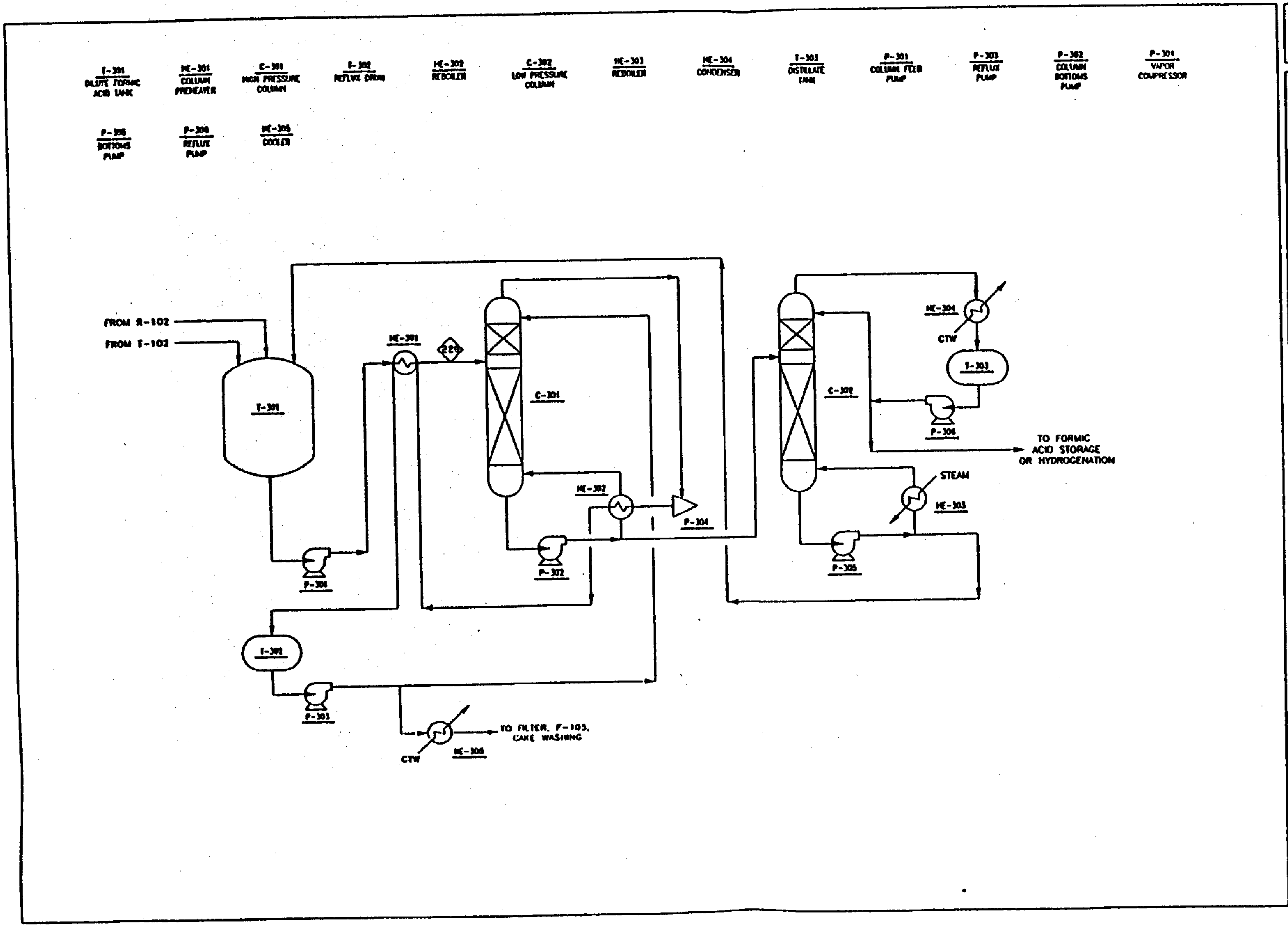
BIOFINE/PENCOR

L.A. PLANT

1000 WET TONS/DAY
PAPER SLUDGE FEED

EXTRACTION + PURIFICATION

PFD-002



BioMetics

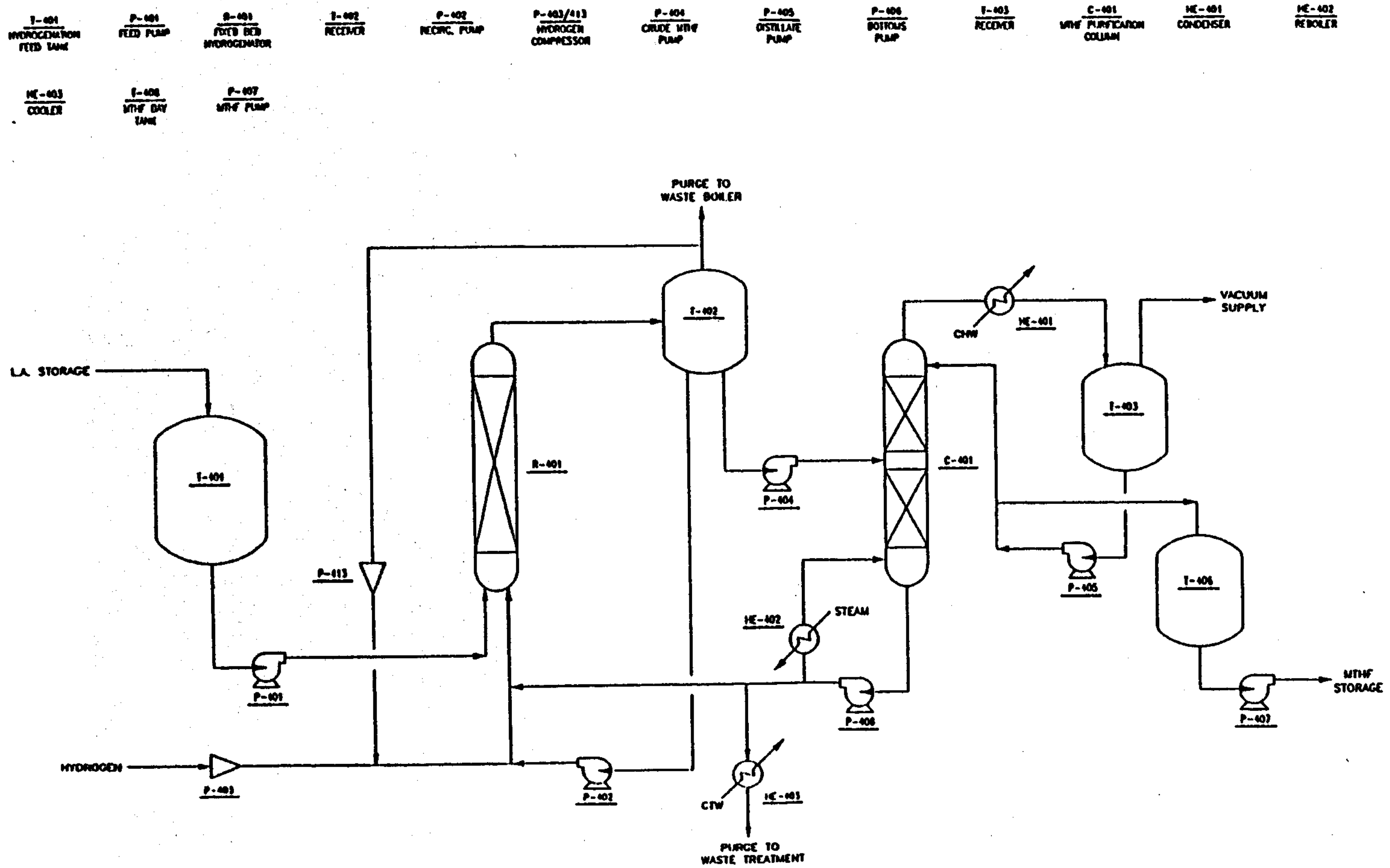
BIOFINE/PENCOR

L.A. PLANT

1000 WET TONS/DAY
PAPER SLUDGE FEED

FORMIC ACID RECOVERY

PFD-003



BioMetics

BIOFINE/PENCOR

LA PLANT

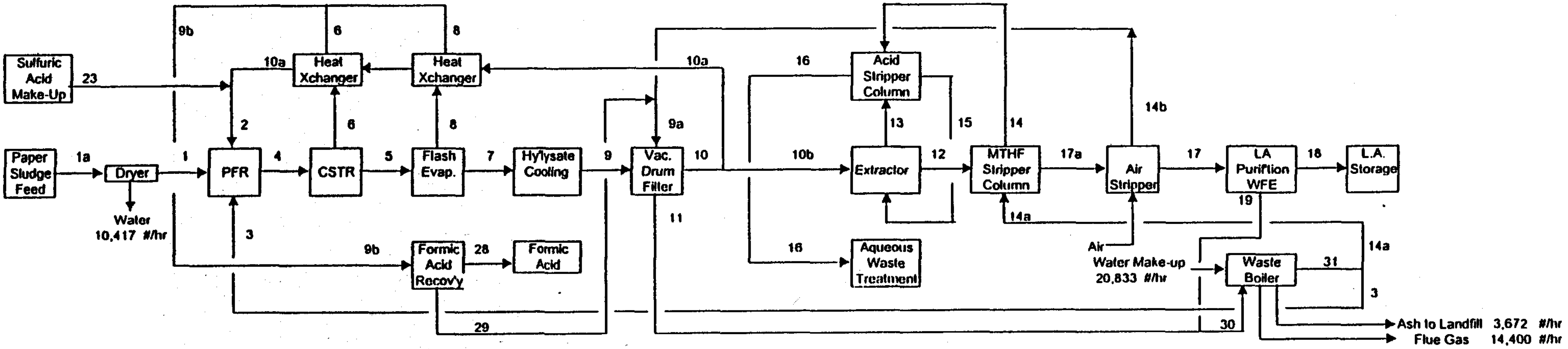
1000 WET TONS/DAY
PAPER SLUDGE FEED

HYDROGENATION

PFD-004

L. A. Plant

500 Wet Tons per Day Paper Sludge Feed



HPS/# Cellulose 0.75

Stream No.	1	1a	2	3	4	5	6	7	8	9a	9b	9	10	11	10a	10b
Pressure, psig	600		600	500	450	250	250	50	Atmos.	30	30	30	30	30	30	30
Temp., 0F	Ambient	Ambient	350	470	460	406	406	300	212	110	110	110	110	110	110	110
Stream Name	Paper Sludge (Dry)	Paper Sludge (Wet)	Recycle Acid (R.A.)	H. P. Steam	PFR Effluent	CSTR Effluent	CSTR Flash	Hydrolysate Slurry	Atm. Flash Evaporator	Water for Filter Wash	Formic Acid Recovery	Cooled Hydrolysate	Filtered Hydrolysate	Washed Filter Cakes	Recycled Acid	Hydrolysate to Extractor
Components (lbs/hr)																
Cellulose	14,583	14,583														
Hemicellulose (Furfural)	833	833	174		591		591			591	591		585	6	174	411
Ash & Inorganics	3,958	3,958	122		4,080	4,080		4,080				4,080	408	3,672	122	286
Lignin (Tar)	1,458	1,458	925		7,175	7,175		7,175				7,175	3,104	4,072	925	2,178
Water	10,417	20,833	8,397	10,938	29,752	25,319	4,434	21,135	4,184	18,507	8,617	21,135	32,056	7,586	8,397	23,658
Sulfuric Acid			1,646		1,646	1,646		1,646				1,646	1,630	16	474	1,155
Formic Acid			936		3,853	3,502	350	3,172	331		681	3,172	3,140	32	936	2,204
Glucose					7,292											
L.A.			3,054		3,054	10,345		10,345				10,345	10,242	103	3,054	7,188
MTHF																
Total, lbs/hr	31,250	41,667	15,255	10,938	57,443	52,068	5,375	47,554	4,514	19,098	9,889	47,554	51,164	15,487	14,083	37,081

Stream Composition:

Cellulose	0.47	0.35														
Hemicellulose (to Furfural)	0.03	0.02	0.01		0.01		0.11			0.03	0.06				0.01	0.01
Ash & Inorganics	0.13	0.10	0.01		0.07	0.08		0.09				0.09	0.01	0.24	0.01	0.01
Lignin (Tar)	0.05	0.04	0.06		0.12	0.14		0.15				0.15	0.06	0.26	0.07	0.06
Water	0.33	0.50	0.55	1.00	0.52	0.49	0.82	0.44	0.93	0.97	0.87	0.44	0.63	0.49	0.60	0.64
Sulfuric Acid			0.11		0.03	0.03		0.03				0.03	0.03	0.00	0.03	0.03
L.A.			0.20		0.05	0.20		0.22				0.22	0.20	0.01	0.22	0.19
Formic Acid			0.06		0.07	0.07	0.07	0.07	0.07		0.07	0.07	0.07	0.00	0.07	0.06
Total	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
tons/day	375	500														
Glucose	0.00	0.00			0.13											

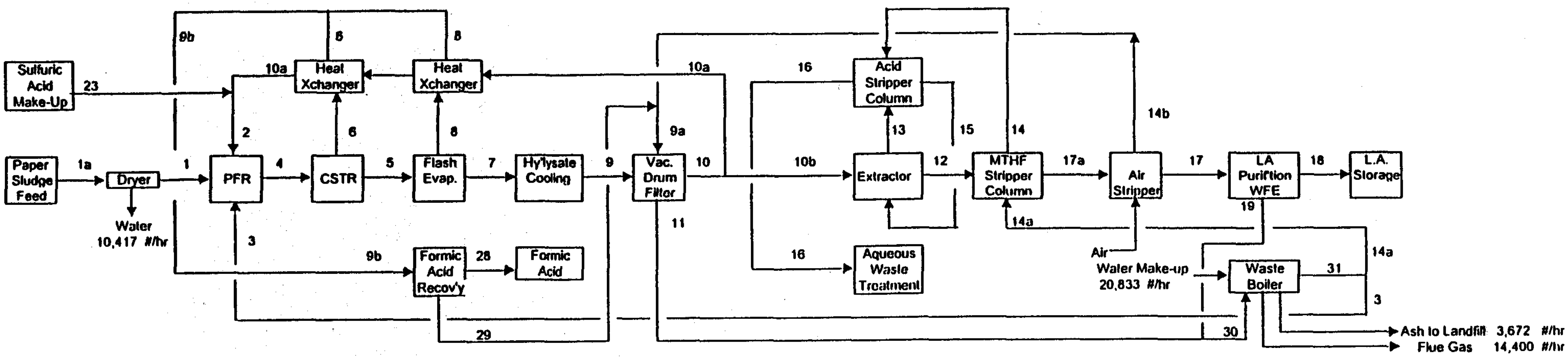
Hemi to Furfural	50%
Hemi to Tar	50%
Cellulose to Tar	30%
Cellulose to Formic	20%
Cellulose to L.A.	50%

Soluble Tar (% of L.A. Produced)	0.30
Soluble Organics (Ash)	0.10
Solid in Washed Cakes	0.50
Entrainment is Washed Cakes	0.01

Date: 12/3/98
G:files/docs/127-penc/st/500tpd5/MatBal

L. A. Plant

500 Wet Tons per Day Paper Sludge Feed



Stream No.	12	13	14	15	16	17	18	19	23	28	29	14a	17a	14b	30	31
Pressure, psig	30	30	150 mm Hg	Atmos	Atmos	Atmospheric	25 mm Hg	25 mm Hg	600	30	30	5	150 mm Hg	30		500 psig
Temp., 0F	110	110	160	110	212	225	310	310	220	110	110	225	285	110		470
Stream Name	Extract	Reffinate	MTHFStrip Overhead	Acid Strip Overhead	Acid Strip Bottoms	Feed to WFI	WFE Overhead	WFE Bottom (for fuel)	Acid Make-Up	Formic Acid	Bottoms F.A. Rec'y	L.P. Steam Inject	MTHF Strip Bottoms	Water Condensate	Fuel for Waste Boiler	H.P. Steam
Components (lbs/hr)																
Cellulose																
Hemicellulose(Furfural)		411			411						591				6	
Ash & Inorganics		286			286										3,672	
Lignin (Tar)	2,178					2,178		2,178					2,178		6,250	
Water	1,178	23,734	911	1,254	23,391	267	184	83	24	5	8,612	9,895	10,163	9,895	7,669	20,833
Sulfuric Acid		1,155			1,155				1,172						16	
Formic Acid		2,204			2,204					681					32	
Glucose																
L.A.	6,469	719			719	6,469	6,146	323					6,469		427	
MTHF	22,234	1,424	22,234	23,658												
Total, lbs/hr	32,060	29,933	23,145	24,912	28,166	8,915	6,330	2,585	1,196	687	9,203	9,895	18,810	9,895	18,072	20,833

Stream Composition:																
Cellulose																
Hemicellulose (to Furfural)		0.01			0.01						0.06					
Ash & Inorganics		0.01			0.01										0.20	
Lignin (to Tar)	0.07					0.24		0.84							0.35	
Water	0.04	0.79	0.04	0.05	0.83	0.03	0.03	0.03	0.02	0.01	0.94	1.00		1.00	0.42	1.00
Sulfuric Acid		0.04			0.04				0.98							
L.A.	0.20	0.02			0.03	0.73	0.97	0.13							0.02	
Formic Acid		0.07			0.08					0.99						
Total	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00				1.00	1.00
MTHF	0.69	0.05	0.96	0.95												

Water Solubility in MTHF	5.30%
MTHF Solubility in Water	6.00%
Extraction Efficiency	90%

LA Recovery in WFE	95 %
LA Conc. in Product	97 %
# H.P. Steam per # L.A.	1.78
# L.A. Product per # Cellulose	0.42

Acid Conc. 98%

Est. Caloric Value of Tar, btu/lb	5,000
Boiler Efficiency	0.50
H.P. Steam Produced, #/hr	20,833

Date: 12/3/98
G:files/docs/127-penc/sl/500tpd5/MatBal

Pencor, Inc
Glen Cove, NY

MTHF

Design Basis: (1) 1,000 wet tons per day of paper sludge containing 50% moisture
(2) Ref. PFD - G/files/docs/127-penc/s/(Mpd5b1/MatBal

Equipment Tag	Equipment Description	Manufaturer	Unit Capacity
	Paper Sludge Feed Handling		
P-101A/B	Reactor Feed Pump, packaged unit, include screw feeder, hydraulic unit, control panel	Schwing	200 gpm
T-101	Feed Hopper, "moving floor" to discharge MSW to feed pump; packaged unit; steel construction	Schwing	5,000 cu ft
X-101	Conveyor, for dry sludge		65,000 lbs/hr
X-102	Conveyor; for wet sludge		85,000 lbs/hr
X-103	Dryer; for drying paper sludge feed		85,000 lbs/hr
X-104	Magnetic Separator, for tramp metal in feed		65,000 lbs/hr
X-105	Screen Classifier, for dry sludge		65,000 lbs/hr
	Reactors		
HE-101	Reactor Condenser, to heat recycled acid with steam from R-102; 316 ss s & t		9.8E+06 btu/hr
HE-102	Flash Condenser; to heat recycled acid with steam from T-102; 316 ss s & t		9.5E+06 btu/hr
HE-103	Hydrolysate Cooler; s & t design; zirconium tubes; c.s. shell	AstroCosmo	1E+07 btu/hr
HE-104	Trim Heater; 316 ss s&t		
P-102A/B	Hydrolysate Pump; TFE-lined; centrifugal		200 gpm
P-103A/B	Recycle Acid Pump; Teflon-lined; centrifugal		75 gpm
P-104A/B	Recycle Acid Booster Pump; zirconium; centrifugal, to 600 psig TDH	Lawrence	75 gpm
R-101A/B	1st Stage Reactor, tubular design for 1000 psig; zirconium construction	AstroCosmo	6 in sch 40 pipe
R-102A/B	2nd Stage Reactor; CSTR design for 500 psig; zirconium-lined 316L ss	AstroCosmo	6.5 ft dia.
T-102	Flash Tank, zirconium lined 316 ss; 15 psig	AstroCosmo	130 cu ft
	Solid Separation		
F-105	Hydrolysate Filter; packaged unit; vacuum rotary drum design; plastic filter	Dorr-Oliver	200 gpm
P-105A/B	Clarified Hydrolysate Pump; Teflon lined, centrifugal		200 gpm
P-150A/B	*Make-up Acid Pump, Teflon-lined centrifugal*		10 gpm
T-104	Clarified Hydrolysate Tank; 316 ss	AstroCosmo	2,000 cu ft
T-150	Sulfuric Acid Storage Tank; for conc. sulfuric acid; c.s.; 1 week inventroy		5,000 cu ft
X-110	Conveyor, for filter cakes; to waste boiler		40,000 lbs/hr
	Extraction		
CF-201A to F	Extractor; BXP 620P centrifugal type; PVDF wetted parts; XP motor	Robatel	25 gpm
HE-215	Solvent Recycle Cooler; s&t design; 316 ss tubes; c.s. shell		7E+05 btu/hr
P-201A/B	Extract Pump; 316 ss, centrifugal, seal-less; XP motor		200 gpm
P-203A/B	Solvent Recycle Pump; 316 ss, centrifugal, seal-less, XP motor		200 gpm
T-201	Extract Receiver, 316L ss		2,000 cu ft
	MTHF Stripper		
C-201A/B	Solvent Stripper Column; tray column; 316 ss; 50 psig/FV		6 ft dia
HE-201A/B	*Preheater, s&t design; 316 ss tubes*		2E+06 btu/hr
HE-202A/B	Solvent Condenser; s&t design; 316 ss tubes		8.2E+06 btu/hr
HE-203A/B	Vacuum Cooler; s&t design; 316 ss tubes		
HE-212A/B	Steam Injector; for C-201A/B, L.P. steam, 316 ss		9.9E+03 lbs/hr
P-202A/B/C	Solvent Pump; 316 ss, centrifugal, seal-less; XP motor		200 gpm
P-210A/B/C	*Column Bottoms Pump; 316 ss, centrifugal*, seal-less; XP motor		200 gpm
P-215A/B	Solvent Make-up Pump; 316 ss, centrifugal, seal-less; XP motor		10 gpm
T-202A/B	Condenser Receiver; for C-201A/B; 316L ss, 50 psig/FV		600 cu ft
T-203	Separator Drum; 316 ss; 50 psig/FV		2,000 cu ft
T-215	Solvent Tank; 316 ss		1,000 cu ft
	Acid Stripper		
C-203	Acid Stripper Column, Teflon-lined c.s.; packed		5 ft dia
HE-204	Acid Stripper Reboiler, graphite tubes, 15 psig		2.4E+07 btu/hr

[illegible]
(2) Ref PFD - G/files/docs/127-penc/sl/1Mpd5b1/MatBal

Equipment Tag	Equipment Description	Manufaturer	Unit Capacity
HE-205	Acid Stripper Condenser; s&t design; 316 ss tubes		1.8E+07 btu/hr
P-204A/B	Acid Stripper Feed Pump; 316 ss, centrifugal; seal-less; XP motor		200 gpm
P-205A/B	Acid Bottoms Pump, Teflon-lined centrifugal		200 gpm
	L.A. Finishing		
HE-212A/B	Condenser for WFE overhead; s&t design, 316 ss tubes		5.0E+06 btu/hr
HE-213A/B	Vacuum Cooler; s&t design; 316 ss		
P-206A/B/C	WFE Feed Pump; positive displacement, 316 ss; XP motor		50 gpm
P-208A/B	Tar Pump, for WFE Bottoms; positive displacement, jacketed, 316 ss		
P-212A/B	Air Blower, 316 ss		80,000 scfm
P-222A/B	LA Pump, seal less, 316 ss		
T-225A/B	Distillate Level Tank, 316 ss		
T-226A/B	Bottoms Level Tank, jacketed, 316 ss		
T-235	LA Storage Tank; 316 ss; 1 week inventory		60,000 cu ft
X-202	Air Stripper; 316 ss		80,000 scfm
X-212A/B	Wiped-Film Evaporator; packaged unit; 25 torr; incl. vacuum unit, level tanks & pumps; 316 ss	Pfaudler	230 sq ft
X-222A/B	Vent Scrubber; packaged unit; FRP		80,000 scfm
	Formic Acid Recovery System		
C-301	High Pressure Column, 3 bar; 20 plates, 316 ss		3 ft dia
C-302	Low Presssure Column; 1 bar		3 ft dia.
HE-301	Column Preheater, 316 ss; s&t		1.8E+06 btu/hr
HE-302	Reboiler; for C-301, 316 ss; s&t		1.7E+06 btu/hr
HE-303	Reboiler; for C-302; 316 ss; s&t		2.1E+06 btu/hr
HE-304	Condenser; for C-302, 316 ss s&t		1.7E+06 btu/hr
HE-305	Cooler, 316 ss s&t		1.8E+06 btu/hr
P-301A/B	Column Feed Pump; 316 ss; seal-less		50 gpm
P-302A/B	Column Bottoms Pump, for C-301; 316 ss; seal-less		50 gpm
P-303A/B	Reflux Pump; for C-301; 316 ss; seal-less		50 gpm
P-304	Vapor Compressor; to compress atm. vapor to 3 bar; 316 ss		2,000 scfm
P-305A/B	Bottoms Pump; for C-302; 316 ss; seal-less		50 gpm
P-306A/B	Reflux Pump; for C-302; 316 ss; seal-less		50 gpm
T-301	Dilute F.A. Tank; for R-102 vapor; to 3 bar; 316 ss		40 cu ft
T-302	Reflux Drum; for C-301; 316 ss		50 cu ft
T-303	Distillate Tank; for C-655; 316 ss; 1 bar		40 cu ft
T-305	Formic Acid Storage; for 1 week; 316 ss		6,000. cu ft
	Hydrogenation - L.A. to MTHF		
C-401	MTHF Purification Column, 316 ss		5 ft dia
HE-401	Condenser, for C-401, 316 ss, s&t design		1.8E+07 btu/hr
HE-402	Reboiler; for C-401, 316 ss, s& t design		2.4E+07 btu/hr
HE-403	Cooler; for C-401 bottoms; 316 ss, s&t design		1.8E+06 btu/hr
P-401A/B	Feed Pump; for R-401; positive displacement; 316 ss, seal-less		50 gpm
P-402A/B	Recirculation Pump; for R-401; 316 ss, seal-less		200 gpm
P-403A/B	Hydrogen Gas Compressor, for hydrogen feed to R-401		
P-404A/B	Crude MTHF Pump; for C-401; 316 ss, seal-less		50 gpm
P-405A/B	Distillate Pump; for purified MTHF; 316 ss, seal-less		50 gpm
P-406A/B	Bottoms Pump; for C-401; 316 ss, seal-less		50 gpm
P-407A/B	MTHF Pump; 316 ss, seal-less		50 gpm
P-413A/B	Hydrogen Gas Compressor; for recirculated hydrogen to R-401; 316 ss		200 scfm
R-401A/B	Hydrogenation Reactor; fixed bed design, 316 ss; (one in service, one in reserve)		2 ft dia
T-401	Hydrogenation Feed Tank; 316 ss		50 cu ft
T-402	Receiver, for R-401, 316 ss		50 cu ft
T-403	Receiver, for C-401; 316 ss		50 cu ft
T-406	MTHF Day Tank, 316 ss		50 cu ft

[illegible]
for
MTHF [illegible]

Pencor, Inc
Glen Cove, NY

Design Basis: (1) 1,000 wet tons per day of paper sludge containing 50% moisture
(2) Ref PFD - G/files/docs/127-penc/st/1Mpd5b1/MatBal

Equipment Tag	Equipment Description	Manufacturer	Unit Capacity
T-416	MTHF Storage Tank; for 1 week; 316 ss		6,000 cu ft
X-401	Hydrogen Generation System (Note 2)		
	Utilities (Note 3)		
CH-803	Chilled Water System; packaged		100 tons
CT-801	Cooling Tower, w/auto. blowdown		5.2E+03 tons
P-801A/B/C	Cooling Water Pump; c s centrifugal		7,000 gpm
P-803A/B	Vacuum Pump, packaged liquid ring design unit, 150 mm Hg		2.50E+03 acfm
T-702	In Process Aqueous Storage Tank, 316 ss		10,000 cu ft
T-703	In Process Organic Storage Tank; 316 ss		10,000 cu ft
X-703	Waste Boiler, packaged design, installed	Dorr-Oliver	40,000 lbs/hr
X-705	Nitrogen Generation System; packaged design		1,000 scfm
X-715	Compressed Air System; packaged design		2,000 acfm
X-810A/B	Auxiliary Boiler; gas-fired; packaged		
X-900	Fire Protection System		

Notes:
1. Cost Estimates Exclusions:
* Operating costs, permitting, taxes and insurances.
2. Rental or leased equipment from gas supplier (e g. Air Product).
3. Liquid waste treatment by other.

Excludes:	Formic Acid Recovery Waste Boiler Hydrogenation of L.A.	Formic Acid Recovery Waste Boiler	Formic Acid Recovery	None (per above List)
Total Equipment	$13,371,000	$16,103,000	$21,103,000	$22,812,000
Equipment Rigging & Shipment (5% of Total Equipment)	$670,000	$810,000	$1,060,000	$1,150,000
Piping (40% of Total Equipment)	$5,350,000	$6,450,000	$8,450,000	$9,130,000
Instrumentation (40% of Total Equipment)	$5,350,000	$6,450,000	$8,450,000	$9,130,000
Installation Total	$24,750,000	$29,820,000	$39,070,000	$42,230,000
Design, Engineering, Construction Management & Start-Up (20% of Installed Total)	$4,950,000	$5,970,000	$7,820,000	$8,450,000
Spare Parts (5% of Total Equipment)	$670,000	$810,000	$1,060,000	$1,150,000
Contingency (15%)	$4,460,000	$5,370,000	$7,040,000	$7,610,000
Total for Process Plant - Note 1	$34,830,000	$41,970,000	$54,990,000	$59,440,000
Building (30,000 sq ft @ $35/sq ft control room, warehouse/machine shop, welfare quarter, etc)	1,050,000	1,050,000	1,050,000	$1,050,000
Grand Total	$35,880,000	$43,020,000	56,040,000	$60,490,000

LVAC PRODUCTION - 500 DRY TPD PAPER SLUDGE

PROCESSING SCALE -	500	DRY TONS PER DAY	1,000	"AS IS" TONS PER DAY PAPER SLUDGE
OPERATING DAYS/YR	334			
PLANT CAPITAL COST - $MM			50	APPROX.
PRODUCTION RATE -	108,550,000	POUNDS LVAC PER YEAR		
	20,000,000	POUNDS FORMIC ACID PER YEAR		

OPERATING COSTS

COST ITEM	QUANTITY BASIS	UNITS	BASIS COST/UNIT ($)		ANNUAL COST
RAW MATERIALS					
FEEDSTOCK	167,000	T/Y	-	$ PER TON	-
LIME	1,000	T/Y	100	$PER TON	$ 100,000.00
SOLVENT	300	T/Y	300	$ PER TON	$ 90,000.00
					$ -
TOTAL ANNUAL RAW MATERIALS COST					$ 1,192,000.00
UTILITIES					
STEAM	78,000	PPH	ON-SITE BOILER		
ELECTRIC POWER	4.0	MW	30	$ PER MWHR	$ 961,920.00
WATER	30	GPM	0.005	$ PER GAL	$ 72,144.00
NAT. GAS (FOR BOILER)	3	MM BTU/HR	5	$ PER MM BTU	$ 103,540.00
COMBUSTIBLE TARS	65	MM BTU/HR		TO BOILER	
COMBUST'BL "OTHERS	10	MM BTU/HR		TO BOILER	
TOTAL ANNUAL UTILITY COSTS					$ 1,137,604.00
LABOR AND MAINTENANCE					
OPERATING STAFF	7	PER SHIFT	18	($ PER HR	$ 1,048,320.00
SUPERVISION	2	PER SHIFT	23	($ PER HR	$ 382,720.00
MAINTENANCE	0.04	CAP. COST		(4% CAP.)	$ 2,000,000.00
TOTAL ANNUAL LABOR AND MAINTENANCE					$ 3,431,040.00
OVERHEADS					
DIRECT	0.40	LAB. COST			$ 572,416.00
GENERAL	0.30	LAB+MAINT			$ 1,029,312.00
INSURANCE	0.02	CAP. COST			$ 1,000,000.00
TAXES	0.02	CAP. COST			$ 1,000,000.00
LAND AND TAX					$ 200,000.00
TOTAL ANNUAL OVERHEADS					$ 2,772,416.00
TIPPING FEES FOR ASH DISPOSAL	33,400	T/Y	20		$ 668,000.00
PLANT OPERATING COSTS PER YEAR					$ 9,201,060.00
DEPRECIATION		% PER YR	Not Incl.		$ -
PRODUCTION COST PER YEAR					$ 9,201,060.00
BYPROD. REV. FOR FORMIC ACID	20,000,000	at	0.02	$ PER LB	$ 400,000.00
TIP FEE REVENUE FOR SLUDGE	334,000	WET T/Y @	25	$/WET TON	$ 8,350,000.00
NET PRODUCTN. COST FOR LVAC				$/Y	$ 451,060.00
NET PRODUCTN. COST FOR LVAC				$/LB	$ 0.0042

ASSUMPTIONS:

1. Tar burned in boiler for process energy - Natural gas burned as auxilliary fuel
2. Cellulose content of paper sludge - 65%
3. Tip fee charged - $25 per wet ton - Moisture Content 50%
4. LVAC yield - 0.5 lbs per pound cellulose
5. Alkalinity in paper sludge 3% (i.e. 3 lbs acid per 100 lbs sludge)
6. Formic acid valued at energy value only @$0.02 per pound

MARKET ASSESSMENT

A Market Plan for Commercializing Levulinic Acid Derivatives

A confidential client study prepared for:
BIOFINE Limited Partnership

January 31, 2001

prepared by:
ANTARES Group Incorporated
Landover, Maryland
www.antaresgroupinc.com

ANTARES
ENGINEERS & ECONOMISTS

TABLE OF CONTENTS

EXECUTIVE SUMMARY

REFERENCES

Appendix A: **Material Safety Data Sheets (1,4-Butanediol and 2-Methyltetrahydrofuran)**

Appendix B: **Chemical Industry Services, Inc. (Levulinic Acid Derivatives Report)**

EXECUTIVE SUMMARY

Biofine Limited Partnership's (Biofine) proprietary and patented technologies offer a pathway for biobased products to penetrate mainstream fuels, chemicals, and electricity markets in an economical and environmentally beneficial manner. Target markets for Biofine's "biorefinery" approach $2 trillion annually and the variety of products derived from the company's process offer many exciting opportunities.

The Biofine biorefinery uses heat and a dilute inorganic acid to convert cellulose into levulinic acid (LA). Levulinic acid is a versatile chemical that can be converted into a variety of intermediate and finished products.

Biofine has dedicated considerable time and money to developing the technologies required to process cellulose into LA and to produce the derivative products discussed in this report. ANTARES believes that the results Biofine has obtained to date and the experience gained at the company's two-ton-per-day facility in South Glens Falls, New York demonstrate the technical feasibility of these technologies.

The purpose of this study was to assess some of the remaining issues which will impact the larger-scale deployment of Biofine's technologies and suggest action items for addressing them. ANTARES examined general market trends, technical/regulatory issues, and specific market opportunities.

Summarizing this effort, the data collected by ANTARES indicates the following:

- Several chemicals appear to have market volume and price points that make exploring these markets and strategic partnerships with industry leaders a high priority.
- Profitable entry into the energy markets for LA derivatives will require green market support and, in some cases, additional legislative action. Biofine should begin immediately to contact green energy marketing groups and discuss the potential for including heat/power generated using Biofine products in their offerings. However, these efforts should be subordinated to an increased effort to target the chemical industry as a customer of LA derivatives.
- Biofine must place a high priority on additional fuel testing to meet government regulations for key markets and to determine ways to resolve key technical issues.
- Biofine should query the IRS on the requirements to obtain a private ruling for receiving the ethanol tax credit on a prorata basis for the manufacture of ethyl levulinate
- Biofine must begin the process of identifying regulatory requirements for marketing a new pesticide/herbicide as soon as possible. Lead times and testing requirements may push deployment of such a product into the latter part of this decade.
- Cost curves detailing the relationship of biorefinery size to LA production costs and derivative products must be developed to determine optimal plant size for given markets.
- A detailed heat and energy balance for a combined cycle plant operating on EL/Fuel Oil (FO) blends should be performed using data collected from additional fuel testing.
- By 2010, revenue from LA derivative products could reach over $300M/year

The results of this effort suggest that several key markets for LA derivative products exist within the chemical industry. Additionally, properly structured incentives within the renewable energy markets may also provide scale benefits to the biorefinery concept. In the case of both of these markets, Biofine needs to seek out partners with the market expertise and access to the infrastructure needed to ensure success.

1.0 BACKGROUND

The markets for biobased fuels, power and chemicals hold great economic (refer to Exhibit 1) and environmental potential. New technologies are being developed around the world seeking to capitalize on this opportunity. Most industry experts agree that, in the long term, the technologies most likely to succeed will process multiple input streams into multiple products to several sectors. The concept of ***biorefineries*** is similar to traditional petroleum refineries. However, inputs will be based on agriculture, chemistry and biology rather than mining and geology.

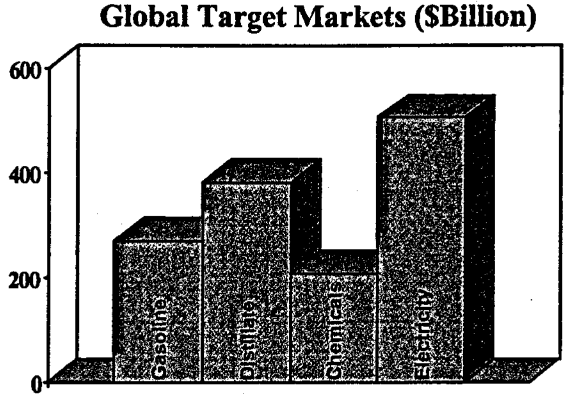


EXHIBIT 1: Annual Global Market

Biofine Limited Partnership has developed a suite of technologies for this purpose. This report focuses on describing the market opportunities and issues Biofine can expect as they deploy their technologies. Green fuels, green energy and biobased chemicals are discussed in this context. The report concludes with recommendations and an action list ANTARES believes will help Biofine penetrate its target markets.

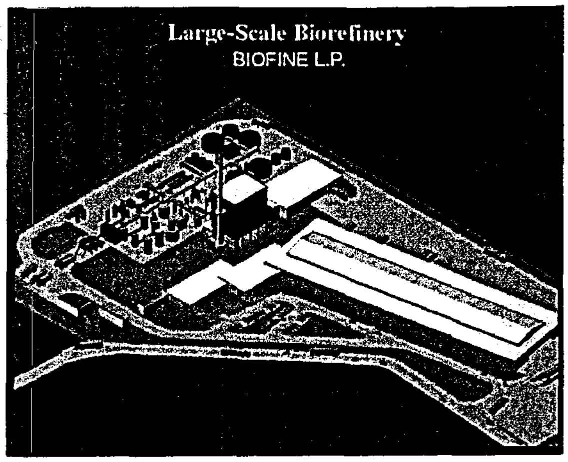
1.1 Biofine Technology

The heart of Biofine's processes is an acid hydrolysis process that uses heat, water and a dilute mineral acid to convert cellulose from various sources into levulinic acid (LA). LA is a platform chemical with many end uses and it can be converted into several different green fuels and a variety of biobased chemicals. It is important to distinguish this chemical process from biological conversion processes which rely on the action of microorganisms to convert organic materials into other products. As a chemical process, the reactions required to convert cellulose into LA are easily controlled and product yields are assured for a given material input. In other words, this process is more akin to manufacturing cars than it is to making wine.

Initial reaction time for this process is about 12 minutes. In contrast, fermentation of corn to ethanol may take several hundred times longer. This rapid reaction has the benefit of reducing processing and storage vessel sizes, thus drastically lowering capital costs. After initial processing, LA maybe refined further to meet the specifications required for the final application. Exhibit 2 shows an example of a full-scale LA manufacturing facility.

Biofine estimates that 60-65% of the ***cellulose*** received into a plant is converted to LA. The remainder of the cellulose is discharged in a stream that has a high lignin content and contains other non-cellulose materials. Biofine's current expectation is to use this stream as fuel for on-site heat/power needs, but they are investigating higher-value uses.[1] Effective utilization of this stream will add value and close the loop on the system. Almost all incoming cellulosic material will be consumed on site and converted into useful, biobased products.

EXHIBIT 2



Processing cellulose to LA also leads to the production of formic acid. Formic acid is an important chemical used heavily in the catalyst industry.

1.2 Market Summary

Biofine is pursuing construction of large- scale ***biorefineries*** which will provide the economies of scale necessary to reduce the costs of producing LA and its derivatives. These biorefineries will rely on low-cost cellulosic feedstocks such as clean urban wood wastes, agricultural residues, wood mill residues, municipal solid waste (MSW) and paper sludge to produce chemicals, fuels or green energy.

These markets are diverse and overarching generalizations are difficult. However, it is apparent that most industrialized countries are encouraging the use and production of biobased fuels, chemicals and energy. Examples include European fossil fuel taxation, America's use of selective tax incentives, and state renewable portfolio standards and system benefits charges. Encouragement manifests itself in varying forms and degrees, but the market environment for these products is promising.

1.3 Fuels Terminology

The jargon used in the fuels sections of this report can be confusing, so this section introduces some commonly used terms for the materials that are sold as or blended with familiar heating and mobility fuels. The potential for confusion has been compounded in recent years by the introduction of new terms (and unconventional definitions for old ones) in Federal laws and regulations. Part of the intent of this section is to establish a consistent usage for this report.

In the petroleum industry, the term *additive* is typically used to mean a functional additive, something blended to a commercial fuel to influence a specific property of the fuel. In this particular usage, an additive is *not* something put in the fuel to function as fuel, that is, to liberate energy when burned. Additives, in many cases are materials not derived directly from crude petroleum, as the balance of the fuel is, but instead synthesized chemically. Their usage in the finished fuel is normally measured/expressed in pounds (of additive) per thousand

---

[1] *The National Renewable Energy Laboratory (NREL) is working to develop a chemical process that converts lignin into high-value chemicals and fuels. An obvious synergy between Biofine's process and this type of research exists and is currently being pursued.*

barrels of fuel, or ptb. This usually corresponds to fractions of a percent. Examples of functional additives include the deposit control additives used in gasolines and certain compounds sometimes added to diesel fuels to improve their lubricity. In this report, we use the term *additive* in this way.

Some materials might need to be used at higher concentrations (1-5%) to function well as additives. This might be true, for example, of ethyl levulinate (EL) or an EL derivative that was used to improve diesel fuel lubricity. In this situation, the term *additive* still applies since the intent of blending such a material would be to modify a specific fuel property, not to add significant volume or energy value to the fuel.

Unfortunately, the term additive is sometimes applied to materials that function primarily as fuel or which modify the fuel's combustion. The *oxygenates* commonly added to gasolines are examples. These compounds, mainly ethanol and methyl tertiary butyl ether, are blended to gasoline to provide extra oxygen to the combustion, particularly when the engine is running fuel-rich. This modifies the combustion, effectively making it leaner (less rich). When used at legal limits, these compounds also add significant energy value to the whole fuel, and significant volume as well (10 volume percent for ethanol and as much as 15 volume percent for MTBE at their respective legal limits). Thus they are not additives as that term is used in this report. Generically, ethanol and MTBE are fuel *blending stocks*, added to the fuel (gasoline, in this case) either to modify its combustion or to add volume to the fuel pool. In that respect, they aren't fundamentally different from *refinery streams* like alkylate (high octane value) or naphtha (basic bulk building block of gasoline). In broad chemical terms, they are *oxygenates*, since they incorporate oxygen in their molecular structures and thus add chemically bound oxygen to gasoline, as called for by the Clean Air Act Amendments of 1990.

Alternative fuel is a term formally defined in Federal legislation, specifically in the Energy Policy Act of 1992. In addition to the alternative fuels named in EPAct, there are criteria by which others may be designated (by the Department of Energy) as alternative fuels. By "alternative," EPAct means "alternative to petroleum." Thus the first of the criteria it specifies is the most intuitively reasonable, namely that a fuel, to be an alternative fuel, must be "substantially not petroleum." DOE has settled on a cut-off of 50 percent as its working definition of "substantially." That is, if at least 50 percent of a fuel's energy value comes from source(s) other than petroleum, it meets the first and most basic criterion for designation as an alternative fuel. *Alternative fuel*, as used in this report, will refer to fuels that are either explicitly named in the Energy Policy Act or meet this definition, since it is widely used and intuitively appealing.

More recently, DOE has begun using the term *replacement fuel*. Again for the purposes of EPAct compliance, replacement fuel is that part of a fuel which is derived from resources other than petroleum, regardless of whether it is the fuel's dominant constituent. An example, and the one which gave rise to the term, is the biodiesel component (20 volume percent) of B20. A blend of 80 percent petroleum diesel fuel with 20 percent biodiesel, B20 obviously derives much less than 50 percent of its energy from the non-petroleum portion of its makeup, hence it can't be an alternative fuel by the EPAct definition, any more than gasohol (10 volume percent ethanol, 90 percent gasoline) could. Under legislative direction, DOE set up a program whereby vehicle fleet owners could get partial credit toward EPAct compliance for the non-petroleum portion of B20 (or other biodiesel blends). That portion, the biodiesel in B20, DOE refers to as replacement fuel, since it replaces part of the petroleum fuel that would otherwise be consumed. In this report, *replacement fuel* is used in the same way, and generally only when EPAct compliance options are being discussed.

As a point of general information, EPAct concerns itself only with automotive (trucks and cars) transportation fuels, not fuels for heating, power generation or non-road use.

Finally, *blend* is a term used in several senses, in reference both to conventional fuels and non-petroleum fuels. Strictly speaking, conventional gasoline and diesel fuel are themselves blends. To produce finished fuels whose properties comply with applicable specifications and meet commercial standards, refiners carefully blend various refinery streams. In this context, blending implies continuous control of the input streams, real-time knowledge of their properties and monitoring of the finished fuel's characteristics. Gasohol is also sometimes referred to as a blend, as in fact it is. However, it is typically *splash-blended* near the end of the gasoline retail chain, rather than being blended continuously and under strict quality control, as in a refinery. For this reason gasohol may or may not meet gasoline specifications, depending on the characteristics of the gasoline to which it is blended. Most informally, *blend* can be used to refer to any mixture of fuels, from any source or sources. In this looser usage, the finished fuel's properties may not necessarily meet any particular standard or specification. How the term *blend* is being used will usually be indicated by context.

2.0 MARKET OVERVIEW

2.1 Review of Derivative Products

The primary products produced directly from Biofine's process are LA, formic acid, and a residue stream that may be used for fuel or further refined to other products. Although formic acid does have a substantial market, it is LA derivatives that will drive plant economics. Conceptually, these products will be either manufactured on-site as an integral part of the plant or LA will be moved to other facilities for conversion. The process is similar in concept to traditional petroleum refining operations and the whole system can be referred to as a ***biorefinery***.

Potential biorefinery products include the items listed below and illustrated structurally in Exhibit 3:

- Ethyl levulinate (EL)
- Delta Amino LA (DALA)
- Succinic acid
- Diphenolic acid (DPA)
- 1,4 Butanediol
- Formic acid
- Green electricity/steam
- Methyl tetrahydrafuran (MTHF)

LEVULINIC ACID & DERIVATIVES

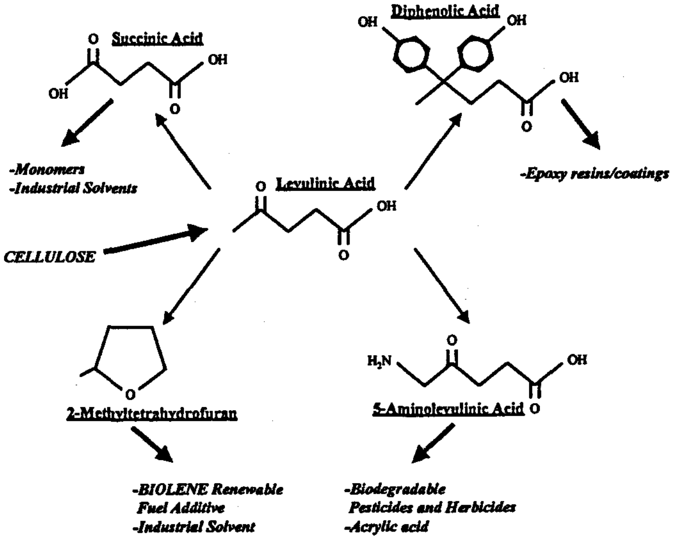


EXHIBIT 3: Derivative Diagram

It is important to recognize that most of these products have many end uses that cross sectors. For example, EL is used in the flavors and fragrances industry. It is also possible to use EL as a diesel replacement fuel. Although both these applications are explored in detail later in this report, general market trends are useful in characterizing the potential for LA derivative products.

2.2 General Market Trends

This section is dedicated to examining some of the key market trends which will influence the penetration of LA derivative products. A more specific, anecdotal look at each of the key derivatives is presented later, but this section provides context to the recommendations and action lists presented later in the report.

2.2.1 Green Fuels

Diesel Replacement Fuels

Between now and 2007, profound changes will occur in the domestic highway diesel fuel market. The principal driver of this most significant trend will be the Environmental Protection Agency's Tier 2 emissions regulations and concomitant rulemaking on road diesel fuel itself. Within the next few months, EPA will promulgate its final rule on road diesel sulfur content. The agency is expected to issue the rule substantially unchanged from the version it proposed, i.e., road diesel fuel will be capped at 15 parts per million sulfur. (Current road diesel fuel is capped at 500 ppm and averages about 350.) Other changes to the fuel will accompany the new limitation on sulfur content, resulting from the severe hydroprocessing diesel blending stocks will undergo. These are discussed elsewhere.

Other forms of distillate fuel will come under keener regulatory scrutiny in coming years. Diesel fuel for non-road uses and Number 2 fuel oil (FO) currently are limited to sulfur contents of 5,000 ppm. The Engine Manufacturers Association (EMA) has recommended that EPA begin rulemaking to lower the sulfur content of non-road diesel fuel. EMA member companies also build the engines used in these applications, which are under increasingly stringent emissions regulations. The EMA has said that EPA's particulate matter standards for non-road diesels may not be achievable with the current fuel and perhaps not even with current highway fuel (500 ppm). They urge EPA to consider mandating ultra-low-sulfur diesel (ULSD - 15 ppm) fuel for non-road engines, as the agency is expected to do for highway diesels. While there may be no comparable pressure to lower the sulfur content of No.2 FO, it's likely that as more and more of the ULSD product is produced, there will be ever-greater "leakage" of the deeply desulfurized product into the FO market. This leakage is already a factor in the non-road market, as 500 ppm product from the highway market finds its way there.

Beyond the potential for LA derivatives, there are two fuels on or entering the market which may become replacement diesel fuels, diesel fuel additives or blending stocks. One is biodiesel, the other is Fischer-Tropsch (synthetic) diesel fuel. The latter is also known as gas-to-liquids (GTL) product. Both can be technically superior fuels to today's conventional product. However, both are more expensive and neither is apt to be available in the next seven years in quantities sufficient to serve a significant portion of the road diesel fuel market.

Biodiesel has become fairly well established in the past several years, benefitting from considerable legislative support, mainly at the state level and particularly in the farm belt. Federal legislation passed in 1998 obliged DOE to promulgate regulations granting credit for the biodiesel component of low-level (ca. 20 volume percent) blends against fleets' EPAct purchases of alternative fuel vehicles. Biodiesel may find wider markets as a lubricity additive to petroleum diesel fuels as the latter are desulfurized. Minnesota will consider legislation during 2001, mandating such usage of biodiesel. It is also possible that pending Federal legislation mandating a "renewable content" in motor fuels will further promote biodiesel's market penetration. Biodiesel remains more expensive to produce than petroleum diesel, although the difference has shrunk recently as diesel fuel prices rose. Nonetheless, it is likely that the principal drivers of biodiesel use will continue to be use mandates and non-technical considerations such as political support of farming and farmers. The emissions benefits of biodiesel, by itself and in blends, are real, but are greatest in older-technology engines. Moreover, they will be dwarfed by the improvements effected by the next generation of diesel emissions controls and improved conventional diesel fuel.

Fischer-Tropsch (F-T) processes can yield diesel-range liquids from virtually any carbonaceous feedstock. F-T coproducts can be converted to diesel-range liquids by conventional refining steps. The initial interest in F-T processes in the U.S. was based on the perception of F-T as a means to monetize natural gas reserves that couldn't be produced economically due to lack of infrastructure (pipelines, LNG plants, tankers, etc.). Natural gas is the most favorable feedstock for F-T processes, but other materials, including coal (the original F-T feedstock) and petroleum coke can also be used.

F-T liquids can be ideal diesel fuels, as demonstrated by recent tests of an all-paraffin synthetic diesel fuel produced by Syntroleum, one of the smaller firms active in the area and a technology licensor. Vehicle and engine emissions reductions were dramatic. However, such a carefully tailored fuel is quite expensive to produce. F-T fuels are essentially sulfur-free, since sulfur must be removed from the feedstock to protect the F-T catalysts. This, too, raises costs. Small amounts of F-T diesel have been imported to the U.S. West Coast from South Africa and marketed as premium diesel fuel. The cost and quality of F-T diesel fuel may make it more economical to employ it as a diesel blending stock than as a separate fuel, even a premium fuel.

Conoco is one of several major oil and gas companies that have recently announced plans to venture into F-T liquids production. It has dissented from the views of smaller companies (Syntroleum and Rentech, for example) regarding the optimum size of GTL plants. Conoco believes those companies have proposed plants too small to be economically feasible. GTL plants need to be at least 60,000 bbl/day, according to Paul Grimmer, manager of the company's gas refining group. This implies daily natural gas consumption of about 500 million cubic feet. If the plant is to operate for the 20 years Conoco says are necessary for amortization, the gas field would need to contain 4 trillion cubic feet. There are very few such fields in the world. The smaller companies, on the other hand, have made a virtue of the potential of their proprietary processes to be scaled down. Rentech has commissioned two studies exploring the feasibility of "piggybacking" its F-T technology on other specialty chemical manufacturing processes to use off-gas or syngas produced in other operations as feedstock for GTL liquids. Rentech's pilot plant was originally tested on landfill gas. Syntroleum likewise has designed its F-T process to be feasible at small plant sizes and to require no air separation plant. Air separation is usually needed to produce the pure oxygen to convert feedstock to synthesis gas (syngas - $CO + H_2$).

For the next five to ten years, it's unlikely that the volume of F-T liquids from all sources will be sufficient to perturb diesel fuel markets. Major refiners' attention will be on conventional processing of distillate fuels to meet the stringent sulfur regulations that will come into force during the period. Outside the California market, available F-T distillate will probably be blended to ULSD, rather than being sold as a distinct, zero-sulfur fuel.

Gasoline Replacement Fuel

Recent developments may herald the closing of the "oxygen era" in gasoline blending and marketing. MTHF, like other oxygenates, may suffer as a result. Major refiners and the American Petroleum Institute have called for elimination of the reformulated gasoline (RFG) oxygen content requirement imposed by the 1990 Clean Air Act Amendments. California refiners have said they can produce all-hydrocarbon gasoline that will meet that state's stringent requirements. Elimination of an oxygen content specification, refiners say, will give them increased flexibility to produce clean gasoline at optimum cost and in the quantities demanded by the marketplace. The position of these fuel refiners is significant, since many have made large investments in oxygenate (ethers such as MTBE) production over the past two decades. Methyl tertiary butyl ether (MTBE), which is and has been the most widely used gasoline oxygenate by far, seems clearly to be on the way out. By itself, California's decision to phase out use of MTBE as a gasoline oxygenate will shrink the world market by 25 percent. Other states, including New York, intend to follow California's lead. Ethanol, the second-most widely used oxygenate, is poised to supplant MTBE in the California market. The response of refiners to the MTBE phase-out however, has not been to embrace ethanol, but to call for an end to the blending of all oxygenates to gasoline. The state of California also has indicated its preference for a waiver from the Federal RFG oxygen content requirement. This would make it possible for the state to accommodate refiners wishing to produce hydrocarbon-only gasoline, and without handing the huge California gasoline market to ethanol.

EXHIBIT 4: Ethanol Producers & Volumes

U.S. Ethanol Production Capacity

Company	Location	Feedstock	Production Mgals/yr
Adkins Energy	Lena, IL	corn	30
A.E. Staley	Louden, TN	corn	45
AGP	Hastings, NE	corn	45
Agri-Energy, LLC	Luverne, MN	corn	12
Alchem	Grafton, ND	corn	11
Al-Corn	Claremont, MN	corn	15
Archer Daniels Midland*	Decatur, IL	corn	750
	Peoria, IL		
	Cedar Rapids, IA		
	Clinton, IA		
	Walhalla, ND		
BC International	Jennings, LA	bagasse/rice hulls	20
Broin Enterprises	Scotland, SD	corn	7
Cargill*	Blair, NE	corn	100
	Eddyville, IA		
Central Minnesota	Little Falls, MN	corn	15
Chief Ethanol	Hastings, NE	corn	40
Corn Plus	Winnebago, MN	corn	18
CVEC*	Benson, MN	corn	17
Eco Products of Plover, Inc.	Plover, WI	cheese whey/potato waste	4
ESE Alcohol	Leati, KS	corn	1
Ethanol2000	Bingham Lake, MN	corn	15
Exol, Inc.	Albert Leo, MN	corn	15
Georgia-Pacific Corp.	Bellingham, WA	paper waste	4
Golden Cheese Co. of California	Corona, CA	cheese whey	3
Grain Processing Corp.	Muscatine, IA	corn	10
Heartland Corn Products	Winthrop, MN	corn	16
Heartland Grain Fuel	Aberdeen, SD	corn	8
	Huron, SD		12
High Plains Corporation*	York, NE	milo/corn	68
	Colwich, KS		
	Portales, NM		
J.R. Simplot	Caldwell, ID	potato waste	6
	Burley, ID		
Janton Alcohol	Edinburg, TX	corn	1
Kraft, Inc.	Melrose, MN	cheese whey	3
MMI/ETOH	Golden, CO	brewery waste	2
Manildra Ethanol	Homburg, IA	corn/milo/wheat starch	7
Midwest Grain Products*	Pekin, IL	corn/wheat starch	108
	Athison, KS		
Minnesota Clean Fuels	Dundos, MN	waste sucrose	2
Minnesota Corn Processors*	Columbus, NE	corn	110
	Marshall, MN		
Minnesota Energy	Buffalo Lake, MN	corn	12
Morris Ag Energy	Morris, MN	corn	8
New Energy Corp.*	South Bend, IN	corn	85
Pabst Brewing	Olympia, WA	brewery waste	7
Parallel Products	Rancho Cucamonga, CA Lou	food and beverage waste	10
Pro-Corn	Preston, MN	corn	15
Reeve Agri-Energy	Garden City, KS	corn/milo	10
Sunrise Energy	Blairstown, IA	corn	5
Vienna Correctional	Vienna, IL	corn	1
Williams Bio-Energy*	Pekin, IL	corn	130
	Aurora, NE		
Wyoming Ethanol	Torrington, WY	corn	5
Total Capacity			*1,805*

** may include beverage and industrial alcohol production*

In this atmosphere, a new oxygenate, renewable or otherwise, will face an uphill marketing struggle, regardless of its technical performance. A biorefinery product, MTHF may also face resistance from environmentalists, fuel distributors and retailers, as well as the general public in the wake of publicity surrounding incidents of groundwater contamination by MTBE. Apart from the sheer similarity of the two chemical acronyms, there is probably a significant portion of the American public that would express strong disapproval of any attempt to add more "chemicals" to familiar motor fuels.

Some major refiners produce and market ethanol-blended gasoline in response to regional market demands, but no refiner, it's safe to say, does so because ethanol offers compelling technical or economic advantages that are unobtainable from MTBE or hydrocarbons. Instead, the market relies on government-sponsored tax incentives for market share.

However, recent trends in ethanol production reflect growing demand, spurred in part by the anticipated phase-out of MTBE and in part by near-record petroleum prices. The Department of Energy reports ethanol production for the years 1998, 1997 and 1996 of 1.4 billion, 1.3 billion and 1.1 billion gallons, respectively. Production for 2000 is on track to reach a record 1.6 billion gallons, according to the Renewable Fuels Association. AUS Consultants completed a study of the industry and its probable markets for the Governors' Ethanol Coalition in March of this year. The report projects annual production to more than double to 3.5 billion gallons by 2004. This growth is predicated on ethanol's being used to replace the MTBE that will be withdrawn from the gasoline pool. That regulatory move is expected to create additional demand for 3.2 billion gallons of ethanol. Projects currently under construction could add a billion gallons of annual capacity to the U.S. ethanol industry. These projects and others in planning are expected to constitute 31 percent of the 1.95 billion gallons of capacity that will be added to the U.S. industry by 2004, according to AUS. The balance will come from plants now on line. Among the latter is ADM's Walhalla, North Dakota plant, which the company restarted in September 2000. It had been closed since June 1999, when oil prices were at near-record lows.

Exhibit 4, from the Renewable Fuels Association website (compiled by Bryan and Bryan, Inc.) summarizes the make-up of the domestic ethanol industry.

2.2.2 Green Electricity

Consumer demand for green power has the potential to modify the way the power industry views its markets. As utilities transition into a deregulated era where it is presumed that emphasis should be placed on generating low cost energy, evidence is emerging that some consumers are willing to pay more to "go green." The allure of higher margins and new marketing opportunities has spurred the electricity market to consider this opportunity carefully. Environmental groups are developing certification processes, holding meetings with stakeholders, and issuing guidelines to consumers about what is green and what is not.

The Center for Resource Solutions (CRS) is one organization seeking to establish standards for green electricity. The group has conducted regional meetings that include representatives from the renewable energy industry, environmental groups, and government. The group offers a certification "Green-e" that power suppliers can use to identify their supplies as green. Power suppliers are granted Green-e certification based on guidelines established at these regional meetings.

CRS, arguably the largest and perhaps best-organized of these groups, has been successful in persuading consumers and suppliers that a standard is beneficial to both parties. Although some aspects of the Green-e certification process draw criticism from renewable energy advocates, the process has proved effective in offering some guarantee to customers that they are getting what they're paying for. In addition to helping set regional standards for green energy, CRS releases market reports on green-e certified power.[2]

---

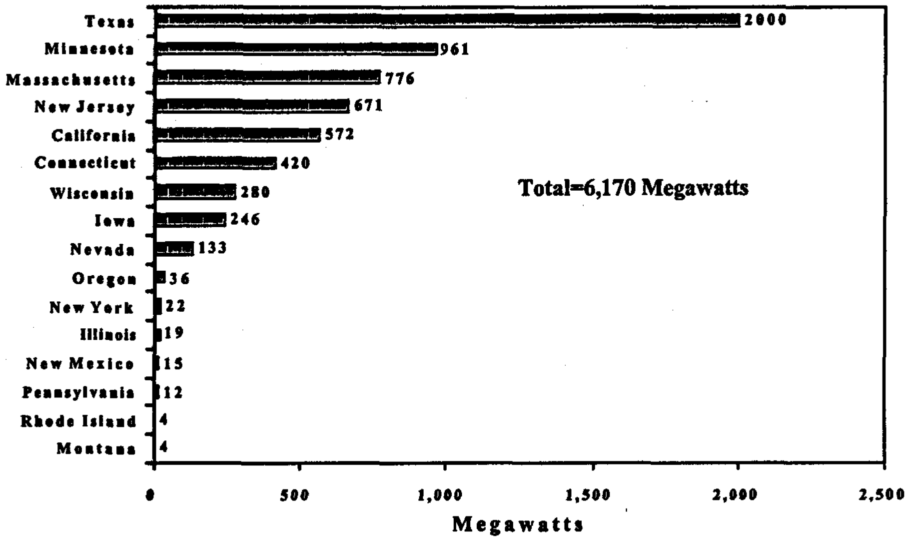
[2] CRS, *Green-e Verification Results 1999*, September, 1999

Based on these reports, approximately 400,000 people are being served by Green-e certified electricity in California and Pennsylvania. In addition, nearly 28,000 non-residential accounts were being served by Green-e certified power. Some of the larger commercial accounts include Kinko's, the Gap, and Fetzer Vineyards. These organizations are accompanied by a few state and Federal facilities including EPA's laboratory in Richmond, California.

The total energy supplied by Green-e electricity products in 1999 was approximately 1,227,000 MWh. Assuming most of this power was generated from average baseload supplies, that amounts to just over 200 MW of capacity. Of the generation sold in California, 98% was either biomass (31%) or geothermal (67%). In Pennsylvania, over 52% of the Green-e certified generation was biomass. Since the Pennsylvania Green-e product mix also allows some non-renewable sources to be marketed under the Green-e label, the bulk of the remaining power (26%) was derived from non-renewable resources. This suggests that although considerable public relations attention is being paid to solar and wind, biomass and geothermal sources will be the most significant contributors to this nascent market.

EXHIBIT 5

New Renewables Capacity by 2012 from State Renewable Electricity Standards and Funds



Several state governments have also encouraged renewable electricity production. Recently the Union of Concerned Scientists (UCS) released a report that quantifies the impact that state renewable electricity standards may have on new renewable capacity. This information is presented in Exhibit 5.[3] UCS suggests that renewable electricity standards may spur approximately 6,170 MW of new renewable capacity by 2012. More than 80% of this will be added in five states: Texas, Minnesota, Massachusetts, New Jersey, and California.

---

[3] UCS, *Clean Power Surge: Ranking the States*, April 2000.

It should also be noted that according to information collected by Oak Ridge National Laboratories, California, Texas, and Minnesota also ranked in the top six of states in the U.S. with biomass resources available for less than $20 per dry ton (their lowest cost category).[4] Texas also has the advantage of being first in the nation both in the number of refineries operating, and operating refinery capacity. Although more work is needed, this may provide an interesting market opportunity for Biofine's biorefinery products.

2.2.3 Biobased Chemical Intermediates & Products

At the turn of the twentieth century, most non-fuel industrial products including dyes, inks, paints, medicines, chemicals, clothing, and synthetic fibers were made from biomass-derived materials. By the 1970s, organic chemicals derived from fossil feedstocks had captured more than 95 percent of the market previously held by biological resources.[5] In some cases, biobased products have made a comeback, but petrochemical feedstocks still dominate.

Biofine is considering deploying a variety of biobased chemical products that span the breadth of these markets. For this overview, these markets are broken into the following categories because of their relevance to LA derivative markets: herbicides/pesticides, solvents, monomers, polyurethane, catalysts, dyes, epoxy resins, pharmaceutical intermediates. Fuels are treated separately.

Pesticides/Herbicides[6]

In 1996, worldwide use of pesticides (including herbicides) increased to an estimated $31.3 billion at the user level. This is an increase of 17% since 1991 and the market expanded primarily in the United States, Western Europe, Asia, and Latin America. Herbicides and insecticides remain the first- and second-largest pesticide sectors, respectively, in terms of value.

Six producers account for 62% of annual U.S. pesticide sales: Monsanto, Novartis, American Cyanamid, DuPont, DowElanco, and Zeneca.

Solvents

In 1999, solvent buyers saw the prices for most solvent materials rise, primarily due to the increased price of feedstocks.

The marketplace for solvents is also changing as consolidation occurs at the production and consumer levels. Large mergers on both ends of the supply chain are being initiated to take advantage of economies of scale. These include a merger between Dow Chemical Co. and Union Carbide Corp.

---

[4] Oak Ridge National Laboratories, *Resource Spreadsheet*, 1999

[5] National Research Council, *Biobased Industrial Products*, 2000

[6] SRI/CEH, Pesticides/Herbicides, Abstract, 1997

Chevron Chemical and Phillips Petroleum Co. have closed a 50/50 joint venture and created Chevron Phillips Chemical Company.

Solvent formulations are being driven by regulatory and environmental issues including those related to volatile organic compounds (VOCs) and hazardous air pollutants (HAPs). In response, most solvent formulations are now emphasizing "high solids" contents or waterborne makeup. Biodiesel (methyl esters of long-chain fatty acids) is also being marketed as a biodegradable industrial solvent.

Demand in the solvents industry is expected to remain fairly flat. Outside analysts suggest that the "booming" nature of the economy is helping to buoy the market, but caution that climbing oil prices or a slowing economy will moderate industry growth rates.

Pharmaceutical Intermediates

Demand for these products is expected to remain strong, with prices holding steady. Analysts suggest that supply seems to be in line with demand and that U.S. market is growing between 6% and 8% annually and could reach $15.8 billion by 2002.

Historically, the quality of internationally produced intermediates has been a major concern. However, foreign producers, especially those in China and India, are emerging as very competitive producers of basic intermediates. Upgraded facilities, low labor costs, and a desire to compete in large international markets are playing an important role in the production from these countries. A significant challenge for pharmaceutical intermediate manufacturers is to develop new processes that are less expensive, more efficient, and environmentally friendly.

Polyurethane Elastomers

The U.S., Europe, and Japan are currently the major producers and consumers of polyurethane elastomers. In 1997 consumption of these materials was 837 million pounds with an estimated value of $1.5 billion. United States consumption accounts for about half the worldwide total.

The polyurethane elastomer business is dominated by major suppliers of raw materials and prepolymers; Bayer is recognized as the global industry leader. Consumption of these materials is expected to grow on the order of 3.6% a year through 2002, with the highest growth expected in the United States.

Monomers

Acrylic acid and esters are versatile monomers. Their performance characteristics, which include tackiness, durability and hardness, make them ideal for many end-use applications. Most of the acrylic acid produced in the world is converted to esters. The remainder is polymerized to make either polyacrylic acid or copolymeric materials.

Approximately 6 billion pounds of acrylic acid are consumed each year. The largest global producers are BASF, Rohm and Haas, Elf Atochem, Hoechst Celanese, and Nippon Shokubai. These companies accounted for 69% of the world's capacity in 1997. A recent report from Hochberg and Company, Inc. suggests that the U.S. market for acrylic monomers and esters will be $1.5 billion by 2005.

Epoxy Resins/Coatings

Epoxy resins are high-performance thermosetting resins and high-performance coatings are their primary application worldwide. In 1996, the estimated epoxy resin production value for the U.S., Western Europe, and Japan was approximately $2 billion and consumption was approximately 1.4 billion pounds.

The major producers of epoxy resins are Shell, Dow, and Ciba. Combined, these companies represent 70% of the world's capacity.

Catalysts[7]

Worldwide, catalysts are a $9 billion per year business and products that rely on them are estimated to command $500 billion per year. About 90% of chemical manufacturing processes and more than 20% of all industrial products in the U.S. use catalysts. Changing automotive emissions regulations and polymers based on single-site catalysts are expected to increase demand for catalysts over the next few years. As outlined later in this report, the former may represent a significant market opportunity for formic acid which is co-produced with LA in Biofine's biorefinery process.

Dyes

In contrast to the vibrant organic pigments sector, dyes are expected to offer below-average gains, with global demand forecasted to reach $8.7 billion by 2004. Heavy reliance on a sluggish textile industry (which accounts for 70% of the demands for dyes), the Asian financial crisis, and rising exports of low-cost dyes from China and India have hurt prices. Pricing concerns have led to merger talks including one between BASF and DyStar that positions the merged company as a giant in the textile dyes and organic pigment market.

The majority of private companies in the $5.8 billion U.S. dye and pigment market are small firms with annual sales under $10 million. Five private firms hold a combined 17 percent of this market. The dye and pigment market is heavily concentrated in New Jersey, California, Ohio, Pennsylvania, and Texas. New Jersey alone has over 60 such facilities.

---

[7] SRI, *SCUP Report Abstract-Catalysts*, 2000

2.3 Market Survey - Products with Short-Term Penetration Potential

This section provides additional details about the scope and market for LA derivative products that either have existing markets or can be readily substituted for existing products. To the extent possible, updated market volumes, prices, and information relevant to each product's potential to capture significant market share is provided. Recommended courses of action for each market are reserved for Section 3.0.

2.3.1 Ethyl Levulinate

Ethyl levulinate has several immediate market applications. It is already used in the flavoring and fragrance industry. Biofine has also conducted tests to assess EL as a diesel replacement fuel. Automotive, gas turbine, and home heating oil market applications are being sought.

Distillate Replacement Fuel Markets

All the markets discussed in this section rely on EL's effectiveness in providing a distillate replacement that is technically feasible and, as important, economical to use. Later sections outline technical issues that need to be addressed, but economic use includes apparent cost ($/gal) and energy cost ($/Btu). The latter is a more realistic cost comparison, since it includes differences in heat contents and the efficiency of processes using different fuels. For example, if substituting EL for fuel oil reduces fuel economy, the real net cost of its use, beyond its apparent cost ($/gal), should be considered. These figures are used as the authors felt appropriate in the forthcoming discussion.

Home Heating Oil Market

Current prices and the short-term forecast for home heating oil reinforce the market opportunity for EL. Evidence of this includes:[8]

- National **Wholesale** heating oil prices in October 1999 averaged about $0.65/gallon. This October they are ranging from $0.97-$1.08/gallon (based on Petroleum Administration for Defense District-PADD averages). October is not peak season and prices are expected to increase.
- **Residential** heating oil prices in October 1999 averaged $1.00/gallon. In October 2000, they ranged from $1.45 - $1.53/gallon.
- In 1999, residential fuel oil sales were 6.3 billion gallons. The Northeast and New England accounted for about 3/4 of that demand.

---

[8] *Most recent data available from EIA/DOE*

- Total distillate stocks are expected to be down from normal levels in December and January 2000/01.
- Base case winter distillate fuel requirements are estimated to be 3.3 percent above last year. (Note that this includes non-home heating oil markets, but indicates that demand will be higher than last year and prices will be exacerbated by low stocks).

During October 2000, the highest prices were paid by residents of the Central Atlantic region which includes Delaware, Washington D.C., Maryland, New Jersey, New York, and Pennsylvania. Of these, New York residents were paying the highest rates, approximately 10 cents/gallon more than the Central Atlantic average. Volumes for this group are also the highest in the nation, with 2.8 billion gallons sold in 1999. Approximately 45% of this was sold to New York customers. In fact, New York residents bought almost 20% of the home heating oil sold in the nation during 1999.

On a strict energy basis (i.e., $/Btu), the cost to produce EL is likely to remain moderately more expensive than the cost of fuel oil. In times when crude oil prices drop dramatically, the disparity between the two fuels can become substantial. However, an EL/Fuel Oil blend has been suggested by Biofine as a means to extend heating oil supplies, reduce the impact of the price disparity between the fuels, and provide customers with a green energy product.
Exhibit 6 uses estimates of wholesale fuel oil costs and ethyl levulinate to present a marketing approach that illustrates this point.

EXHIBIT 6: Home Heating Oil Price Comparison

EL costs based on $0.05/lb LA from a 500 tpd biorefinery and wholesale cost of ethanol of $1.35

VOLUMETRIC GREEN MARKETING STRATEGY

Heating oil price wholesale ($/gal)	**$**	**1.00**
Heating oil (Btu/gal)		136,380
Heating oil price ($/MMBtu)	$	7.33
EL price wholesale ($/gal)	**$**	**0.90**
EL Blend (%)		20%
Blend price wholesale	**$**	**0.98**
Blend Estimated (Btu/gal)		128,189
Est Blend Price ($/MMBtu)	$	7.64
Blend Vol Price Savings ($/gal)	**$**	**0.02**
Blend Energy Cost Incr. ($/MMBtu)	$	0.31
Green Pricing Premium ($/gal)	$	0.04

Given high-volume production, EL could be produced for approximately the same price on a $/gal basis as the current wholesale price of heating oil. A green marketer might demonstrate how ***green energy*** consumers could fill their tanks with an EL/FO blend at the same cost as other customers buying only FO. The marketing campaign could be structured to emphasize the points highlighted in Exhibit 6 to show that consumers could go green conveniently, and with only moderate fuel cost increases.

The actual green pricing premium would also be listed to maintain credibility, since on an energy basis consumers will be paying more to heat their homes. However, based on average Northeast household heating oil requirements of 925 gal/year, the increase in seasonal heating oil costs may only be on the order of an extra $37/ year. For a segment of the consumer market, this would appear to be a small price to pay to switch a measurable portion of ones energy needs away from a depletable fossil fuel and towards a renewable green one.

Combustion Turbine Fuel

Using EL to produce green electricity could be another important outlet for this fuel. Although several technical issues must be addressed, large volumes of EL could be consumed and transformed into a product (green electricity) which could command a premium market price. However, the primary question concerning EL's use in this application is cost.

For comparison purposes, Exhibit 7 presents three cases outlining estimated production costs attributed to fuel price for a combined cycle plant that uses natural gas, fuel oil, and an EL/FO blend. Plant heat rates are assumed to be 6,936 Btu/kWh and 6,800 Btu/kWh for the liquid-fueled and gas-fueled plants, respectively.

EXHIBIT 7: Production Cost Comparison (cents/kWh)

Based on Combined Cycle Plant with Net Plant Heat Rate of 7,000 Btu/kWh

	Fuel Price			
Fuel Type	**FO $/gal**	**EL $/gal**	**$/MMBtu**	**Fuel Cost (¢/kWh)**
Natural Gas[a]	NA	NA	$3.50-5.50	2.43-3.81
Fuel Oil (electric utilities)[b]	$0.86	NA	$6.31	4.40
Fuel Oil/EL Blend (10%)[c,d]	$0.86	$0.90	$6.62	4.69
Fuel Oil/EL Blend (40%)[e]	$0.86	$0.90	$7.62	5.30
EL Only[e]	NA	$0.90	$9.39	6.60

a) *the average price for natural gas to utilities through August 2000 was approximately $3.16/mcf with an obvious upward trend. However, recent price spikes of double this amount are expected to abate as new drilling and gas imports increase. Antares estimates that longer term prices to utilities will remain high until new production is brought on-line, but that current price trends will move back toward historical price levels.*

b) *based on data through June 2000*

c) *based on LA costs of $0.05/lb, Ethanol price at $1.35/gal, and no derate relative to FO only because of lower Btu content of blend*

d) *Blends on a volume basis*

e) *It is unclear what effect if any the lower heat content of EL will have on plant output - testing is required*

On a production cost basis, a low-percentage EL/FO blend seems competitive with a plant operating on fuel oil alone provided LA can be produced at very low cost. However, it should be pointed out that combined cycle plants are intended for baseload operation. In the absence of significant incentives or extenuating circumstances, a plant of this type would not be built based on such high price fuels. In fact, a similar barrier prevents other biomass-fueled power plants from becoming a commercial reality. Biofines challenge is to identify and pursue market opportunities where reliability and environmental profile can provide value to a customer's bottom line.

Of course, a significant green pricing premium or renewable incentive could help close the apparent gap between the EL cases and the natural gas case. Deciding how much of a premium one can expect in this market is difficult. In a recent report, UCS uses an average green pricing premium of 2 cents/kWh for its analysis. Small renewable installations using PV have been known to command electricity prices of $0.56/kWh, but most industry experts suggest much smaller premiums will be required before larger projects, capable of meeting customers' entire demand, will become attractive. Wind projects, which have drawn considerable public and marketing interest, are being built promising that only marginal premiums (if any) will be required.

Based on the admittedly simple calculations above, a premium of only a few cents per kWh would be sufficient to make EL fuels competitive.

However, the more imposing hurdle is one of perception. Green energy premiums are likely to be commanded by products that deliver a high percentage of green content. Low-percentage blends of EL/FO are not as likely to attract much attention or secure substantial premiums. Higher-percentage blends may be able overcome this problem, but the high cost of EL relative to fuel oil may overwhelm any incremental benefit. In addition, the need to address technical issues is also likely to increase disproportionately as higher-percentage EL blends are used.

Both of these issues may be effectively addressed by decoupling the green portion of the electricity generated from the fossil portion. For example, a 100 MW plant that uses a volumetric blend of 20%EL and 80%FO could be seen as a combination of a 15MW green power plant plus an 85MW fossil plant (split based on heat input). Marketing the 15MW separately may afford net gains in green power premiums and public acceptance.

On the other hand, an 80%EL/20%FO blend may be sufficiently "green"that a green premium may be had on the whole volume. A precedent has been set for this type of marketing by green electricity products already on the market. For example, Green-e's two most relevant rules on receiving their logo only state: 1) at least 50% of the electricity supply for the product comes from Renewable Electricity Resources; and 2) any non-renewable part of the product has lower air emissions than your traditional mix of electricity would have if you did not switch.

It is likely that a high percentage blend of EL would be able to meet both of these criteria. Provided that Biofine was willing to abide by Green-e's other rules, a high percentage EL blend could be certified with the Green-e logo.

EL for Transportation Market

The size of the diesel transportation market makes it a very attractive opportunity for Biofine. On-highway distillate fuel use was 32.1 billion gallons in 1999, up 6% from 1998 levels. Displacing a small fraction of this market could mean billions of dollars in sales for Biofine.

Penetrating this market will require Biofine to clear a few technical and regulatory hurdles, but the net cost to use EL is likely to be the major issue. Average retail prices for diesel the week of November 6, 2000 were $1.61/gallon. However, as noted earlier, EL will probably rely on the same distribution network established for diesel, so for comparisons, a wholesale-cost-to-production- cost analysis must be undertaken. The price for biodiesel, EL's primary biobased competitor in this market, is also presented.

EXHIBIT 8: Diesel Price Comparison

Fuel Name	Heating Value (Btu/gallon)	($/gallon)	($/MMBTU)
Diesel Fuel No. 2	131,067	0.80-1.00	6.10-7.63
Ethyl Levulinate[a]	95,425	0.90-1.92	9.39-20.10
Biodiesel	120,910[b]	1.50-2.25[b]	12.41-18.61

a) Assumes LA produced at $0.05-$0.20/lb, Ethanol at $1.35/gal

b) NREL Biodiesel fact sheet

Even at its current high prices, biodiesel has had some success in capturing a minute fraction of the diesel market. Current industry production, according to the National Biodiesel Board, is 2-4 million gallons per year. This figure is expected to rise as biodiesel prices fall. However, improved production processes will only take biodiesel so far, since its cost is highly dependent on the feedstock costs, and in large-scale production, this will be oilseed crops grown for profit. This may offer Biofine a competitive advantage, since waste resources are its preferred feedstocks. In some cases, Biofine may receive a tipping fee for disposing of these wastes, substantially lowering LA production costs.

The issue for both EL and Biodiesel is that under current petroleum pricing scenarios they are likely to remain more expensive than diesel fuel. This is a problem that faces many biobased energy products and a combination of creative marketing and incentives will be required to overcome it. The California market may offer such an opportunity. During the week of November 6, 2000, retail prices in California were approximately $0.30/gallon higher than in the rest of the country. The primary reason for this differential is the restrictive diesel fuel specification promulgated by the California Air Resources Board (CARB). Aromatics content is limited to 10 volume percent, versus the more typical 15 to 25 volume percent of other diesel fuels. CARB permits more aromatic diesel fuels to be sold in California, provided they can be shown not to increase exhaust emissions. The typical approach used in such alternative formulations is to add cetane improvers, which also increase fuel cost.

However, EL may provide an alternative strategy. More testing will be required, but and EL/Diesel blend may also provide emission benefits. The higher price differential in this market would also make EL more economically attractive.

Flavoring and Fragrance Markets

EL is an FDA-approved food additive used in frozen dairy goods, beverages, candy, and baked goods. It is also used in the fragrance markets and, along with 590+ other chemicals, it is used in cigarettes. The scope of the EL market in this application is unclear; no domestic or international production volumes could be obtained at the time of this report. However, current prices for EL range from $17.50/lb. in bulk to $66/lb. in small quantities. Because such small quantities were offered at a variety of suppliers' sites suggests a smaller, but potentially lucrative market.

At $1.00 per gallon, Biofine production cost of EL would be $0.12 per pound. Even at 100 times this cost, it appears Biofine could easily compete in this market.

Of some concern is that some of the higher-priced product appears to be marketed as *natural*. No definition of this term in this context was obtained, but it is unclear how Biofine's process will be viewed even though many of Biofine's process inputs would most likely be considered "natural". The potential is such that additional market research is warranted.

2.3.2 Methyl Tetrahydrofuran (MTHF)

Gasoline Oxygenate Market

Methyl tetrahydrofuran (MTHF) is the sole LA derivative with potential applicability as a gasoline oxygenate. Approximately a third of the motor gasoline (mogas) consumed in the U.S. is reformulated gasoline (RFG), which must contain 2 percent (by weight) oxygen. In 1998, the country consumed 110.7 billion gallons of mogas, of which 32 percent, or about 35.4 billion gallons, was reformulated. Gasohol accounted for 11 percent of gasolines consumption in that year. Numerous areas of the country also have seasonal oxygenated gasoline programs (usually in winter months), but these gasolines were only one percent of 1998 consumption. Thus, some 44 percent of the nation's gasoline, or about 48.7 billion gallons (1998), contained at least 2 weight percent oxygen.[9]

The ability of MTHF to compete in the gasoline oxygenate market will depend, obviously, on the continued existence of that market. As discussed elsewhere, there is an accelerating regulatory trend toward banning MTBE as a gasoline oxygenate for its perceived environmental effects. Faced with this trend, U.S. refiners are unlikely to voluntarily adopt ethanol in lieu of MTBE, preferring instead to produce all-hydrocarbon gasoline. If the Clean Air Act's gasoline oxygen specification is straightforwardly repealed, the way will be cleared for them to do that. However, pending legislation before Congress[10] may mandate use of some "renewable content" in the nation's motor gasoline. Ethanol is the obvious intended beneficiary of this legislation; if it is passed, the repeal of the oxygen

---

[9] U.S. Department of Energy, *Transportation Energy Data Book*, 20th edition, prepared by Oak Ridge National Laboratory, October 2000.

[10] Oil and Gas Journal's *Washington Week*, September 20, 2000.

content requirement will have little practical effect, although it will not support the rapid expansion of the ethanol market that would result from ethanol's substitution for MTBE. In any case, ethanol-blended gasoline will continue to be offered at least in significant regional markets. Ethanol creates some technical problems for refiners and distributors, but it shares one very important characteristic with MTBE - it is a high-octane blending stock, which gives it value to refiners in addition to the generous blender tax credit available from the Federal Government. Information available at this writing indicates MTHF cannot compete with either of the current gasoline oxygenates on the basis of octane.

MTHF suffers in this application for another reason. Unlike ethanol, which is manufactured wholly from renewable resources, MTHF is made from levulinic acid via hydrogenation. There are few, if any, renewable sources of the necessary hydrogen that are also economic compared to hydrogen from natural gas. MTHF will therefore probably not fully meet the standard of renewability, should that standard be legislated.

Exhibit 9 shows the relative volume and energy costs of regular conventional (non-reformulated) gasoline and the oxygenates of interest. Prices are for products on the Gulf Coast market, without taxes or retail distribution costs.

EXHIBIT 9: Motor Fuel Cost of Energy Comparison

Product	$/gallon[a]	$/MMBTU
Regular-grade gasoline	$0.913	7.91
MTBE	$1.12	11.89
Ethanol (w/o credit)	$1.02	13.48
Ethanol (w/credit)	$0.48[b]	6.34
MTHF	$1.30	16.25

a) October 2000
b) reflects 54 cent/gal credit

Solvent Market

The largest solvent markets foreseen for MTHF are predicated on its ability to substitute for tetrahydrofuran (THF). Of the THF consumed in the U.S. (1996), only 23 percent went to the solvent market. This proportion was 41 million pounds in 1996, projected to grow only about 12 percent (to 46 million lb.) by 2001. This compares to the 77 percent of the THF (138 million lb. in 1996) used as a precursor to polytetramethylene ether glycol (PTMEG). PTMEG is used in the manufacture of synthetic fibers (spandex) and elastomers. This usage has been projected to grow almost 41 percent to

194 million pounds in 2001. THF list prices in mid-'96 were reported to be about $1.36 per pound.[11] In those applications for which MTHF can substitute for THF, Pencor/Biofine would be in excellent competitive position if it can produce MTHF for ca. $0.10 a pound, as it believes it can.

Penn Specialty Chemicals, which acquired the furfural chemicals business of Great Lakes Chemicals, markets MTHF (among many furfural-based compounds) as a pharmaceutical solvent. This is likely to be a higher-value solvent market for MTHF for producers able to meet the applicable quality specifications.

2.3.3 1,4 Butanediol[12]

1,4-butanediol (BDO) is used primarily as an intermediate to manufacture other chemicals and polymers through either of two principal reactions: dehydration and dehydrogenation. Total demand for BDO in the United States, Western Europe, and Japan amounted to about 1.1 billion pounds in 1996, approximately half of which was consumed in the United States. In the United States, approximately 45% is used to manufacture THF, 24% for polybutylene terepthalate resins (PBT), 22 percent gamma-butyrolactone, 5 percent polyurethanes, and 4% of other uses including industrial solvents.

The majority of current U.S. BDO production is based on the traditional manufacturing process which entails reacting acetylene with formaldehyde. Other production methods are now being developed and several U.S. companies have plans to add capacity employing these technologies. BP Amoco, for example, is commissioning a 140 million pound per year plant based on butane oxidation.

About 65% of the BDO produced in the U.S. is captively consumed because of its need in downstream products. Approximately 15-20% is covered by long term contracts, and the remaining 15-20% is supplied to the merchant market.

During the past five years the addition of new capacity has led to an oversupply of BDO and prices have fallen in the last three years by 50 percent. Maturity in the PBT resin, spandex, and engineered plastics markets has also slowed the rapid growth experienced early in the past decade. However, internationally, growth in these markets remains ahead of growth in GDP.

---

[11] SRI/CEH, *CEH Product Review-Tetrahydrofuran*, 1997.

[12] SRI/CEH, *CEH Product Review-1,4 Butanediol*, 1997

Key U.S. producers and production capacity of BDO are shown in Exhibit 10. Almost half of U.S. BDO production occurs in Texas.

EXHIBIT 10: Key U.S. BDO Production

Producer	**Capacity (Mlb)**
BASF, Geismar, LA	280
BP Amoco, Lima, OH	140
DuPont, LaPorte, TX	225
ISP, Texas City, TX	65
Lyondell, Channelview, TX	120
Total	***830***

BASF is one of several producers of 1,4 butanediol (BDO). The company quotes list prices (as of October 1, 2000) for the product as follows:

Bulk deliveries (tank trucks) from Geismar, Louisiana -	$1.03/lb.
Bulk deliveries from Bayonne, New Jersey -	1.05/lb.

There is a 24,000 lb. minimum order for the above prices. Lead time distinguishes the Louisiana versus New Jersey prices. With sufficient lead time, the shipment can be made directly from BASF's Geismar plant and the cost of stocking at the Bayonne terminal can be avoided. A full tank truckload is 45,000 lb.

Additional prices are as follows:

Truckloads (of drums) with a 24,000 lb. minimum -	$1.10/lb. (from LA)
One to two drums -	2.23/lb.

All prices are freight collect (purchaser pays shipping costs).

The BASF sales department indicated that long-term contract prices can be discounted from the bulk prices above, but declined to specify the magnitude of such discounts without a firm order.

It is unclear how prices for BDO will be impacted by the new technologies being deployed. However, one would expect them to be driven downward somewhat. This is important when considering Biofine's potential to become a serious player in this market.

Biofine has estimated that its technologies will allow BDO to be produced at prices of $0.25/lb. or approximately a 75% discount over the current selling price of BDO. Provided that industrial standards can be met, one would expect rapid and extensive market penetration even if Biofine charged a 100% mark-up for the product. Recognizing the size of this market, a 25% market share at $0.50/lb translates to annual sales of $138 million. In reality, if these production cost levels are obtainable, market penetration might easily be higher.

2.3.4 Formic Acid[13]

Formic acid is a versatile, colorless liquid that is used in the manufacturing of fumigants, animal feed additives, commercial paint strippers, catalysts, textile dyeing and finishing, leather tanning, nickel plating, electroplating, and coagulating rubber latex. It is also used in the production of aspartame. The largest single use of formic acid is as a silage additive in Europe.

In 1994, U.S. consumption of formic acid was 57-58 million pounds. End-use in the U.S. has remain distributed approximately equally in the manufacture of rubber, catalysts, pharmaceuticals, leather and tanning, textile dyeing and finishing, and plasticizers. However, a substantial portion (22-46%) goes to other uses.

Formic acid prices remained flat through the early '90s, averaging $0.44/lb. The value of the U.S. market using this price was approximately $25 million in 1994. Biofine has suggested that it can produce formic acid at a third of the sale price. At these levels, Biofine could almost certainly capture a significant share of the existing market.

However, the most interesting market for formic acid may be the domestic catalyst market. Formic acid is used in the manufacture of nickel, aluminum and other catalysts. It is also used to regenerate catalysts that are poisoned with sulfur and lead. If new low-sulfur fuel requirements for the transportation sector are promulgated, as they almost certainly will be, the sulfur content of diesel fuels and gasoline will need to be lowered by an order of magnitude. Refiners will depend on nickel-molybdenum and cobalt-molybdenum catalysts to assist them in accomplishing this feat as economically as possible. Formic acid may play a role in this market both in the manufacture and regeneration of such catalysts.

---

[13] SRI/CEH, *CEH Data Summary-Formic Acid*, 1995

2.3.5 Diphenolic Acid (DPA)

Biofine expects to produce diphenolic acid as a replacement for bisphenol A (BPA), a white solid used primarily in the production of polycarbonate resins and epoxy resins. DPA was used for some of these applications until BPA, which is derived from petroleum feedstocks, replaced DPA as the chemical of choice and continued to expand its markets.

The uses of BPA include polycarbonate resins (63%), epoxy resins (27%); and miscellaneous others including flame retardants. In 1996, 13 major producers were located in the United States, Western Europe, and Japan. Total production of BPA in these regions was 3.5 billion pounds, approximately 50% of which was produced in the U.S. Texas-based producers are responsible for about half of U.S. production capacity. Polycarbonate resins are the largest and fastest-growing end use for BPA, but epoxy resins have also experienced substantial growth. At a price of $0.80/lb., this translates to a $2.8 billion market. Major U.S. producers of BPA are shown in Exhibit 11.

EXHIBIT 11: U.S. Producers of BPA and Capacities

Producer	**Capacity (Mlbs)**
Aristech, Haverhill, OH	230
Dow, Freeport, TX	365
GE Plastics, Burkeville, AL	165
GE Plastics, Mount Vernon, IN	585
Shell, Deer Park, TX	550
Total	**1895**

All of these producers have captive phenol and acetone supplies for manufacturing BPA and all but Aristech also have captive downstream requirements for the manufacturing of resins.

For DPA to recapture its market share and compete with BPA in the larger market will require at least two key things: 1) DPA must demonstrate that its cost of utilization (via Biofine's process) is less than the cost of using BPA, and 2) Users (derivative product manufacturers) must be convinced that the products they are making will experience no ill effects or will in fact be improved by the substitution. Ultimately, they must offer their customers the same commitment to quality and they will need to be convinced that this substitution has merit.

For the users, cost of utilization will include any process changes that must be undertaken to switch from BPA to DPA. Since a majority of U.S. producers have both a captive feedstock supply and end-use, the net business cost of converting from BPA to DPA for these companies is uncertain. However, Biofine suggests that DPA could be produced at approximately a third the current price of BPA. If lower margins for this product could be tolerated, sufficient gains on downstream production processes may be made by these companies, 1) to delay the addition of future BPA production capacity, 2) and/or substitute DPA at the expense of their BPA manufacturing operations.

2.3.6 Levulinic Acid[14]

LA is a versatile chemical in that its C5 linear configuration, and activity at the acid and ketone sites allow for a wide range of chemical reactions. LA has been frequently discussed by researchers as a desirable building block (platform chemical) for the production of other chemicals. However, it's availability and high cost ($4.00 - $6.00 / lb) simply did not make its use viable for most applications.

The current market for LA is one million pounds annually with a selling price of $4.00 - $6.00 per pound. With prices of under $0.25 per pound, which is lower than many of the current petro- chemical building blocks, LA should stimulate significant technical and commercial interest.

2.4 Market Survey - Products with Long-Term Penetration Potential

This section discusses the potential of LA derivative products that will require longer time horizons to be introduced into the market either because of long regulatory lead times or the need for extensive research and development.

2.4.1 Delta Amino Levulinic Acid (DALA)

DALA already enjoys a small, but very high-priced market in the pharmaceutical industry. However, Biofine sees the product's real potential in the mass pesticide/herbicide market. Although there are a number of regulatory issues to address, this market provides the volumes necessary to justify large-scale manufacturing of LA and its other derivatives.

---

[14] Excerpts used with permission from Ray Biliski. Complete text of this report can be found in the appendix.

Pharmaceutical Market

DALA has uses in limited quantities within the pharmaceutical industry and prices for the material are reported as high as $50/gram. However, it is DALA's potential in photodynamic cancer treatments that offers the most lucrative applications in this sector. Researchers in several countries, including the United States, are investigating a variety of applications for this treatment of different conditions. Most notably, non-melanoma (basal cell carcinomas) in cancer. Trials using DALA in photodynamic therapy (PDT) suggest that superficial tumors can be very effectively treated.

In this context, skin cancer is at epidemic proportions worldwide. In the U.S. it has been estimated that in 1995, 1.2 million cases of basal cell carcinoma were diagnosed and treated[15]. In 1985, worldwide (excluding the Soviet Union) the figure was 2.7 million. In the U.K. the incidence of this disease has risen by 238% in the past 14 years. Provided that a very effective, low-cost treatment based on PDT is developed using DALA, the market outlook for this product is likely to ramp up quickly and could command premium prices relative to other applications.

PDT using DALA is also being investigated for its effectiveness in treating small lesions in the wall of the gastrointestinal tract and recalcitrant warts. More research will undoubtedly reveal other uses as well.

Pesticide/Herbicide Market

DALA occurs naturally in plant and animal cells and is key to processes such as photosynthesis and oxygen transport. Applied externally, it is an effective pesticide/herbicide that is non-toxic to animals. Further, it is completely metabolized and any overspray decomposes rapidly, leaving no residues. Using the appropriate modulators to control its specificity, Biofine expects DALA to become a significant player in the residential and commercial pesticide/herbicide market.

World use of pesticides (including herbicides) in 1996 increased to $31.3 billion at the use level. Market expansion in the U.S., Western Europe, parts of Asia and Latin America fueled the 17% increase over 1991 levels.

Outside the U.S., herbicide production has decreased steadily in Western Europe. The largest herbicide markets are France, Germany, the United Kingdom, Italy, and Spain. Supply and consumption of pesticides in Japan also declined prior to 1996, due primarily to reductions in active rice acreage and the use of more effective herbicides.

---

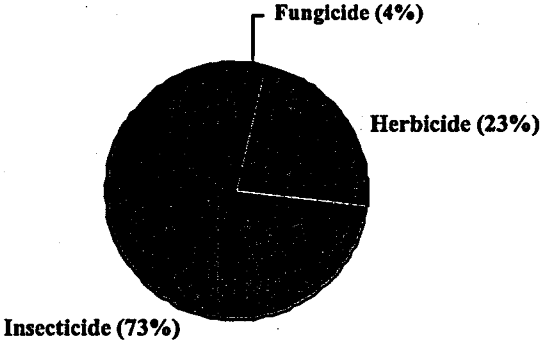
[15] Leonard Goldberg, *Basal Cell Carcinoma*, The Lancet Interactive, March 9, 1996

With the greatest sales volume and total sales value, the herbicides sector is the leader in U.S. pesticide sales. The U.S. herbicide market makes up 39% of the value and 25% of the volume of the world market. The total size of the U.S. pesticides market is estimated to be $9 billion at the producer level. The expected demand in all uses is projected to be 536 million pounds of active ingredient in 2002.

The three chemical types that make up over 70% of U.S. herbicide consumption are heterocyclic nitrogen compounds, carboxylic acids and derivatives, and amides. Market pressures at all levels of the supply chain are forcing companies to develop novel market strategies to maintain profit margins and market share.

The boom in home starts over the last decade is moving producers and distributors to consider the home lawn and garden market more carefully. A breakout of 1999 U.S. pesticide sales is shown in Exhibit 12. Pesticides sales to golf courses, nurseries and other professional landscaping applications are estimated to be about half the total, $1.2 billion.

EXHIBIT 12



1999 U.S. Sales = $2.2 billion
Source: SRI International

Although the sales value of the U.S. pesticide market is estimated at $2.2 billion, at the producer level it is more like $600 million. Formulation, packaging, distribution and a 10-15% profit make up the difference between these figures.

2.4.2 Acetoacrylic Acid

Acetoacrylic acid (AAA) is a by-product of DALA production. In the production of delta amino levulinic acid (DALA), LBLP would produce one mole of AAA per mole of DALA. This compound looks very similar to LA, and may be an attractive platform chemical for AAA derivatives.

No work has been done on identifying high-value-added uses for AAA. However, it should not present a disposal problem since it is likely that AAA can be hydrogenated, a very common chemical procedure, into crude LA and returned to a LA production plant for purification into virgin LA. This would appear to provide a strong motivation to manufacture LA and DALA at the same site, since recycling AAA acid would mitigate any disposal expense that might otherwise be associated with its production.

2.5 Technical Issues

Initial commercial acceptance of the products afforded by Biofine's biorefinery will be based on their ability to meet or exceed industry standards. There is little question that substituting renewable, green biobased feedstocks for fossil feedstocks provides environmental benefits. However, Biofine must be able demonstrate that these products will not adversely affect the end-users' operations. Therefore, Biofine's first line of marketing must be to demonstrate that all the technical issues that may be raised by derivative product users have been adequately answered. The ANTARES Group believes the technical issues discussed in this section must be completely addressed before LA derivative products will be commercially embraced.

2.5.1 Fuels

Replacement Diesel Fuels

Biofine has supplied Texaco with several LA derivative fuels for testing. Biofine supplied ANTARES with the results of the analyses which include results on various blends of a base diesel fuel with:

- ethyl levulinate (EL)
- methyl levulinate (ML)
- levulinic acid (LA)
- 2-methyltetrahydrofuran (MTHF)
- various higher-order alcohols intended to function as cosolvents.

The most complete information available is for a blend of 79 vol. percent base diesel fuel (45.2 cetane index), 20 vol. percent EL and 1 vol. percent iso-amyl alcohol. Texaco expresses reservations regarding four properties of the blend: its cetane number, its energy content, its physical stability and its front-end volatility. [16] All of these are important. Two of these properties, the cetane number effect of the EL and the blend stability, must be addressed before these fuels can become commercial commodities.

Blend Stability

None of the blends containing any of Biofine potentially diesel-applicable products was stable. That is, all mixtures of levulinic acid (LA), ethyl levulinate (EL) or methyl levulinate (ML) with diesel fuel would separate more or less rapidly into two phases. This behavior would obviously be unacceptable to any

---

[16] Copy of Texaco report summary and memo forwarded to ANTARES by Biofine, ca. October 2000.

diesel fuel user. Texaco points out that additives are available that "may" prevent this separation, but has not tested any to date. None of the alcohols tested were effective. Four mixtures containing from 6.67 to 20 percent methyltetrahydrofuran (MTHF) and either or both EL and ML apparently remained stable and clear. However, it is unlikely that MTHF would make an acceptable diesel fuel additive or blendstock due to its volatility and its octane number.

The latter, while too low to improve most commercial gasolines, is nonetheless high enough to imply a poor cetane number. (For a given fuel, octane and cetane numbers, which are relevant to spark-ignition and diesel engines respectively, tend to be inversely related.)

Cetane Number

Ethyl levulinate apparently has a poor cetane number or at least a poor blending cetane number. Texaco notes that while the cetane number of the 79/20/1 blend met the current ASTM D975 specification (40.1 cetane number versus 40), EL lowered the cetane number of the base diesel fuel from 3 to 5 numbers.[17] The current average cetane number of U.S. diesel fuels is about 45. It's likely that in the next five years and beyond, this number will rise. The ASTM specification may not be changed, but commercial diesel fuels will probably improve due to refining changes necessitated by new EPA rules and to competitive commercial considerations. The Engine Manufacturers Association has published a suggested specification for a "premium diesel fuel." It calls for a minimum cetane number of 50. Volkswagen AG has called for even higher values. Higher cetane numbers have long been known to improve diesel engine performance.

Recent work has also shown that cetane numbers are strongly related to emissions, where higher is better. Texaco notes that additives are available that could help recover the cetane number decrement imposed by blending with EL, but additives of any kind increase the cost of a fuel and may create other, unanticipated performance problems.

Texaco also observes that EL fuel blends have significantly lower energy content than diesel fuel. This would directly and proportionally reduce the fuel economy of diesel vehicles using the blended fuel. In cold climates, kerosene is blended to diesel fuel. With an energy content of about 18,200 Btu/lb, kerosene typically reduces over-the-road fuel economy by 2 to 3 percent when it makes up 30 to 50 volume percent of the blend. With an energy content of about 16,300 Btu/lb, the 20 percent EL fuel was 16 per cent less energetic than the base fuel, which implies an in use fuel economy decrease of

---

[17] *Caution: the base fuel's cetane index is given as 45.2. Cetane index is calculated from the fuel's distillation temperatures, a procedure appropriate only for all-hydrocarbon fuels. Since the base fuel is an all-petroleum fuel, its cetane number is probably close to the value predicted by its cetane index. The blend, on the other hand, was apparently tested to determine its cetane number. Cetane number is developed from an actual engine test.*

about the same magnitude. Diesel fuel buyers, especially highway diesel fuel users like truckers, would notice such a loss and see it as a direct loss of productivity and profitability.

The same refining and commercial pressures that will drive cetane numbers up over the next several years will tend to produce lower-density diesel fuels. These will also be less energy-dense and will deliver poorer over-the-road fuel economy. Any non-petroleum diesel blendstock that reinforces this effect will be less welcome in the diesel fuel pool.

Gasoline Replacement Fuels

At the time of this report, ANTARES had very limited information on the technical performance of methyl tetrahydrofuran (MTHF). The SAE technical paper database, for example, contains only a single reference to the chemical. This paper, and a communications with one of its authors, Professor John J. Thomas at the Florida Institute of Technology, forms the basis of ANTARES review of this product.[18]

In the main, Professor Thomas uses estimated values of key MTHF properties in his paper (SAE 932675). These are not adequate to assess MTHF's suitability and attractiveness as a gasoline oxygenate. Thomas said[19] he has data developed in the course of several years' work with MTHF as a gasoline blending agent. However, he declined to provide it to Antares. Thomas said his research had been supported in part by a private company, which he didn't name.

Octane Number

Professor Thomas states in his paper that MTHF has a Research Octane Number (RON) of 87, but gives no source for this information. If correct, this is a significant technical issue for MTHF's use in gasoline. RON is determined by a less severe test that Motor Octane Number (MON) and hence is typically from 3 to as much as 14 numbers higher. [This difference is called the "sensitivity" of the fuel.] The average of the two is the Antiknock Index or AKI. This is the number posted on gasoline pumps and used to distinguish among gasoline grades. If MTHF is a minimally sensitive fuel, its MON would be approximately 84, and its AKI less than 86. In this case, MTHF could not be blended even to regular-grade gasoline (87 AKI, minimum) without degrading its AKI. Its deleterious effect on mid-grade (89 AKI) and premium (93 AKI) gasolines would be even more pronounced. Ethanol's RON has been variously reported as 106 to 120 and MTBE's to be in the range of 115 to 120. The MONs

---

[18] Thomas, J.J., et al., *Exhaust Emissions and Field Trial Results of a New, Oxygenated, Non-Petroleum-Based, Waste-Derived Gasoline Blending Component: 2-Methyltetrahydrofuran*, SAE Technical Paper 932675, October 1993.

[19] Personal communication with Prof. J.J. Thomas, Florida Institute of Technology, September 2000.

of the two oxygenates have been reported to be 90 to 97 for ethanol and 97 to102 for MTBE. Obviously, a more thorough testing for MTHF is required to certify the value estimated by Professor Thomas.

Equally important, potentially, are the Blending Octane Values (BOV) of MTHF. Hydrocarbons and oxygenates do not necessarily behave as predicted by their own octane numbers when they are blended to gasoline. For example, methyl cyclopentane (C_6H_{12}) has an RON of 91 and an MON of 80, for an AKI of 85.5. However, when blended to gasoline, it behaves as though its Research and Motor octane numbers were 83 and 77, a blending AKI of 80. In other words, this compound, tested as a neat fuel, would almost meet the AKI specification for a regular-grade gasoline. Blended to gasoline, however, it behaves as though it were a low-octane material that would significantly degrade the octane value of even a regular-grade base gasoline.[20] Methyl cyclopentane was not selected at random to illustrate this point. MTHF is essentially a mono-substituted methyl cyclopentane and *may* exhibit similar behavior. [Or it may not; the fact that it's a furan, an oxygenate, may trump its structural similarity to methyl cyclopentane in predicting its blending octane behavior.] MTBE and ethanol also have high blending octane values. Sun Tech (the research arm of Sun Oil Co.) reported ethanol to blend at 121 (RON) and 100 (MON) for a blending AKI of 111. MTBE had blending octane values of 116 and 103, for a blending AKI of 110. Again, more thorough testing of MTHF is required.

2.5.2 Chemicals

Technical issues as they relate to chemicals primarily refer to customer specification requirements. In this sense, these markets are probably least problematic since supply, specifications and price are the primary drivers. For each of the chemical markets Biofine intends to penetrate, dealing with product and sales specifications will be crucial. Each chemical must be tested and demonstrated to meet industry and government standards. Sample material and data handling and specification sheets that will be useful in satisfying customers that the products they are receiving will meet their needs. To the extent possible, ANTARES has collected such information and included it in the appendix.

2.5.3 Power

Using EL for Large-Scale Power Production

The production of electricity from products derived from Biofine's technology is not fraught with technical hurdles. Biofine management plans to construct large-scale, green power plants that

---

[20] Mueller Associates, Inc., *Gasoline Octane Enhancement: Technology, Economics and Environmental, Health and Safety Considerations,* prepared for the U.S. Department of Energy/Office of Environmental Analysis, July 1985.

manufacture and consume a diesel replacement fuel, ethyl levulinate. Provided ethyl levulinate and diesel can be effectively blended into an acceptable and economic fuel, market issues are expected to dictate project construction and timing.

Although some technical issues are to be expected, past experience with alternative fuels provides some degree of optimism that they can be effectively addressed.

The preferred and most economic choice for producing large quantities of electricity from this fuel is a combined cycle plant that employs a combustion turbine and a heat recovery steam generator (HRSG). These plants have the advantages of being very efficient and using well- established technology that can be readily guaranteed, even when using alternative fuels. However, using a combustion turbine with an alternative liquid fuel will have an impact on the performance of the plant, compared to operation on natural gas. This is important, since many vendors quote rough turnkey numbers based on natural gas performance specifications and specific ambient conditions.

For example, one might expect that a combustion turbine burning natural gas would have an output 2-3% higher than it would on No. 2 distillate. Therefore the plant heat rate might be 1-2% better for the natural gas plant. In the size ranges being considered by Biofine (approximately 120 MW) a natural-gas-fired, combined cycle plant might have a net plant heat rate of 6,800 Btu/kWh. The same plant configured for a No. 2 distillate might have a plant heat rate of 6,936 (2% decrease in efficiency). For a baseload plant operating 80% of the year, using fuel priced at $2.00/MMBtu, this efficiency loss represents an increased annual fuel cost of $228,000. Further, this impact is directly proportional to the fuel cost. Recognizing that ethyl levulinate at $1.00/gal is equivalent to over $10/MMBtu, such losses are very important to consider in the overall project economics.

Similar losses in efficiency will be experienced as ambient temperatures above those used in rating the gas turbine are experienced. Most gas turbines are rated at ISO conditions (59F). Compressor inlet temperatures at 80F might decrease turbine efficiency by a few percent and a similar economic impact will be experienced.

On the other hand, some work on other alternative liquid fuels such as methanol and ethanol may point to net power increases. Research conducted by GE in the mid-'70s suggested that methanol (low-btu liquid fuel) actually performed quite well as a combustion turbine fuel and a net thermal efficiency gain of

2% was predicted.[21] The net increase resulted from the complex relationship between methanol's required air/fuel ratio and the increased mass flow of fuel through the system. Westinghouse conducted similar research using ethanol and found that the emissions performance of the fuel was quite good.

From these tests, some specific issues were identified that EL/FO blends must address:

- How readily do the proposed blends ignite and do they provide sufficient temperature increases to accelerate the turbine properly?
- Will the blends provide a stable flame over a range of air/fuel ratios?
- Are temperature distributions sufficiently uniform within the system for proper operation?
- Do the blends give rise to any NO_x emission problems?
- How will the blends affect net thermal efficiency?
- Will the blends meet the lubricity requirements of the turbine's fuel system?
- What impacts on long-term operation will these blends have?

A well-designed test protocol and more information about the specific blends will be needed to answer these questions.

Using LA Process Char for Steam

Many cellulose-based industries have found that producing process steam or even power from biomass residues from their manufacturing processes is very economical. The pulp and paper industry, for example, uses black liquor and bark residues for this purpose. Producing process steam (steam has a higher value than electricity, it is not as easily transmitted, and in this application generating it would also dispose of a potential waste problem) is an ideal application for Biofine's processing char and a variety of technologies suitable for conversion are available. Many of these systems are packaged, turnkey operations and many reputable boiler manufacturers offer engineering and design support for specialty applications. These include ESI Inc. and G&S Mills.

The primary technical/regulatory concerns related to using the char for energy include the aeration of any contaminants left behind from the original feedstock that may be contained in the char. A policy of accepting only clean materials will help reduce this possibility. However, an ultimate analysis for the char produced from the processing of specific feedstocks will be required to assess this threat.

---

[21] Mueller Associates, *Status of Alcohol Fuels Utilization Technology for Stationary Gas Turbines*, April 1979

Biofine should also remain open to the possibility of putting this waste stream to an even higher- value use. Research is ongoing at Sherbrooke University, Montreal and at the National Renewable Energy Laboratory to investigate converting lignose into chemicals and fuels. Biofine is pursuing one such opportunity and this area of research continues to attract attention worldwide.

2.6 Description of Regulatory Drivers

There are a number of regulatory issues which will directly impact the markets for biobased fuels, chemicals, and power. They range from specifying fuel performance and attributes to legislative incentives to producers and consumers of these products. This section outlines several of the more important domestic drivers that will influence Biofine's development strategy.

2.6.1 Fuels

<u>The Energy Policy Act of 1992 (EPAct)</u>

EPAct mandates that certain fleets operated by Federal and state governments, alternative fuel providers, local governments and private companies purchase light-duty alternative fuel vehicles as an annually increasing percentage of their new vehicle acquisitions. MTHF is produced by hydrogenating levulinic acid. Hydrogen for this process would probably be derived from natural gas by way of steam methane reforming (SMR). SMR is the most economical route to hydrogen, and most merchant hydrogen is produced that way. In this case, the MTHF will be wholly non-petroleum and will thus meet the first EPAct criterion for designation as an alternative fuel. However, hydrogen from natural gas is not renewable, and thus MTHF from LA will not be a wholly renewable fuel. While this doesn't preclude its being designated a replacement fuel for the purposes of EPAct, it may reduce its appeal to some potential users.

To date, the Department of Energy (DOE), which is charged with writing the implementing regulations for EPAct, has elected not to impose the Act's mandates on local government and private fleets. DOE has promulgated regulations applicable to Federal fleets, state government fleets and fleets operated by alternative fuel providers (electric and gas utilities, oil companies, methanol producers, etc.).

Significantly, EPAct does not mandate *the use of alternative fuels*. It simply requires fleets falling under its jurisdiction to purchase vehicles *capable of operating on alternative fuels*. Not surprisingly, many fleets have opted to comply by purchasing flexible fuel vehicles (FFVs). FFVs, in addition to being capable of operating on an alternative fuel, can operate on gasoline. Most of them will never see a drop of the alternative fuel they're capable of using (E85 in most cases, 85 per cent ethanol and 15 per cent gasoline). Nevertheless, by purchasing them, fleet operators meet the requirements of EPAct.

(An exception to this general observation is bi-fuel natural gas/gasoline vehicles. Unlike the liquid alternative fuels, natural gas is widely available and infrastructure for its compression and dispensing into vehicles is somewhat more available).

EPAct, moreover, applies only to light-duty vehicles (LDVs). The LDV sector, consisting of passenger cars and light trucks (up to a gross vehicle weight rating of 10,000 pounds) is negligibly "dieselized" at present, although there is expected to be growing penetration by diesel engines in coming years, especially at the heavier end of the LDV range. For this reason, alternative diesel engine fuels and diesel fuel replacements have only a very small potential market in fleets covered by EPAct.

In 1998, Congress passed legislation effectively granting alternative fuel status (for EPAct compliance purposes) to B20, a blend of 20 volume percent biodiesel with 80 percent conventional diesel fuel. DOE had been reluctant to do this, despite the importuning of the biodiesel lobby, because B20 is self-evidently not "predominantly non-petroleum" as required by EPAct's definition of "alternative fuel." DOE responded to the 1998 legislation by promulgating regulations that make it possible for B20 users to get alternative fuel vehicle (AFV) credits for their biodiesel use. They may apply these credits toward AFV purchases, but only in the same year in which the biodiesel is purchased (no carry-forward). They may also sell or trade them to other fleets. Fleets using B20 in their diesel vehicles receive credit only for the biodiesel portion of the B20. Each 450 gallons per year of biodiesel entitles them to one credit, allowing the fleet to defer the purchase of one AFV for one year. DOE was determined not to implement a regulation having the effect of turning any diesel-engine vehicle into an AFV, since this would be inconsistent with the purpose of EPAct, namely to replace the maximum feasible amount of petroleum-based motor fuels.

The implications of the above for ethyl levulinate or any other non-petroleum replacement diesel fuel are similar to those for biodiesel. A non-petroleum liquid such as ethyl levulinate, blended to diesel fuel, would probably qualify as an EPAct replacement fuel. DOE would establish an energy-equivalent volume of EL, the annual consumption of which would qualify a fleet to be awarded one AFV credit. Fleets would have to document their purchase and use of the replacement fuel (EL in this case). Each such credit would entitle the fleet to defer the acquisition of one light-duty AFV for that year. Whether fleets would elect to use the EL-blended fuel would depend entirely on their specific technical and/or economic incentives to do so. EPAct per se places no obligation on any fleet to buy or use alternative or replacement fuels.

A bill currently before the Senate Environment and Public Works Committee would phase out use of MTBE as a gasoline blendstock within four years. It would also permit states to waive the Federal gasoline oxygen content requirement. The bill would require a minimum renewable content in all motor fuels (0.6 per cent in 2002, rising by 0.1 percent annually until 2011, when it would be capped at 1.5

per cent). While clearly intended to benefit ethanol interests, the bill is unlikely to be written in such a way as to exclude other renewable fuels. However, no existing law compels the use of renewable components in any motor fuel.

> MTBE Phaseout - *Oil and Gas Journal's Washington Week, October 11, 2000*
>
> In the final days of the current session, the Senate may consider an Environment and Public Works Committee bill to eliminate methyl tertiary butyl ether (MTBE) in gasoline within 4 years. The Environmental Protection Agency had urged Congress to phase out MTBE, an additive that has contaminated water supplies following underground tank leaks. The legislation would allocate $200 million from the Leaking Underground Storage Tank fund for MTBE cleanup activities. And it would allow states to waive the current federal requirement that RFG contain 2%/wt oxygen. The bill would require more use of alternative fuels, including ethanol, by setting a minimum content for "renewable" fuels in all motor fuels sold. The level would start at 0.6% in 2002, rising a tenth of a percentage point yearly until reaching 1.5% in 2011. Refiners could satisfy the requirement by blending renewable fuel additives, or buying credits generated by the sale of alternatively fueled vehicles. EPA would be authorized to exempt small refiners from the program.

EPA Fuel Component Registration and Clean Air Act Waivers

Section 211(f) of the Clean Air Act provides that no fuel or fuel additive may be introduced into commerce if it will cause or contribute to the failure of any motor vehicle to meet the emissions standards to which it was certified. The key provision of section 211(f) may be waived if the fuel or additive is "substantially similar" to fuels or additives used in certifying model year 1975 cars and light trucks. Certain classes of fuel components are deemed to be substantially similar and hence need no waiver. These include aliphatic alcohols and aliphatic ethers such as ethanol and MTBE (methanol is specifically excluded and may not be used by itself as a gasoline blending agent). Other blending stocks, if they are to be distributed commercially, must be granted a waiver of the 211(f) prohibition. Several such waivers were granted for various oxygenates intended for gasoline blending. Interest in such blending components had largely ended by about 1990.

For an oxygenate like MTHF, obtaining a waiver would entail developing emissions data from one or more vehicles operated on base gasoline and gasoline blended with MTHF. Such data characterize the "instant effect" of the oxygenated fuel on tailpipe emissions. Data on evaporative emissions are also required. To address the long-term effects of using the blended fuel, materials compatibility data must also be submitted to EPA. This generally involves 60-day immersion tests of common automotive fuel system materials and components in heated, agitated samples of the blended fuel. Finally, vehicle driveability must be evaluated on base gasoline and the blended fuel to show that use of the fuel does not degrade this important parameter. From the time the waiver application is submitted to EPA, the agency has 180 days to render a decision. If a decision has not been reached in that time, the waiver is granted "by operation of the statute." (Gasohol received its 211(f) waiver in this way; EPA did not explicitly grant a waiver for the 10-percent-ethanol gasoline blend.) The applicable section of the Code of Federal Regulations is Part 40, Chapter 79.[22]

The Clean Air Act section 211(f) waiver described above is a distinct and separate process from the *registration* of a fuel or fuel additive. In the past, registration involved little more than informing EPA by letter that the fuel additive or blend component was in use, and describing its general nature. At present, however, extensive health effects testing is required. The testing protocols are described in 40 CFR Part 80. The American Petroleum Institute has undertaken a lengthy testing program that will develop baseline health data for eight classes of fuels: gasoline, gasohol, gasoline blended with MTBE, gasoline blended with ETBE, with diisopropyl ether (DIPE), tertiary butanol (TBA), or tertiary amyl methyl ether (TAME). In the case of oxygenated blend components, the threshold that triggers mandatory health effects testing is 1.5 percent oxygen in the finished fuel. Below that level, testing is not required.

At the oxygen content limit of 2 weight percent specified for reformulated gasoline (RFG) in the Clean Air Act, health effects testing for MTHF-blended gasoline would be required.[23]

Availability of the Ethanol Blender Tax Credit/Excise Tax Exemption to Fuel Products Made from Ethanol

In March 1990, the Internal Revenue Service published its interpretation of the term "qualified mixture" as used in section 40 of the Internal Revenue Code. The IRS' interpretation of the phrase had the effect of making ethyl tertiary butyl ether (ETBE) eligible to receive the full value (then 60 cents per gallon, now 54 cents) of the ethanol Blender Tax Credit or Federal Excise Tax exemption when ETBE was blended to gasoline. The IRS reasoned that since ETBE was produced from ethanol (and butene) in a chemical combination reaction and since there was "no significant loss of energy content of the alcohol," ETBE was essentially equivalent to ethanol used as a gasoline blending stock. The decision, controversial at the time, was reaffirmed by the IRS in August 1995 and stands unmodified to this day.[24]

---

[22] Personal communication with James Caldwell, U.S. EPA, November 1, 2000.

[23] Ibid.

[24] *Federal Register*, Vol. 55, No. 47, p. 8946, March 9,1990.

Since EL is also made from ethanol, the question arises of whether EL might be eligible for some part or all of the ethanol tax credit. For an answer, ANTARES spoke with the National Biodiesel Board, since biodiesel can also be made using ethanol. To date, the question of whether biodiesel would qualify has not been put to the IRS by the NBB or by any biodiesel producer. Largely because methanol is considerably cheaper than ethanol, the U.S. biodiesel industry's product is soy methyl esters rather than ethyl esters. The matter is further complicated by the fact that the esterification reaction that produces biodiesel, unlike the ETBE formation reaction, also generates a by-product, namely glycerine. Some part of the feedstock alcohol and its energy content could presumably be considered to appear in the glycerine. This would play into the calculation of how much of the ethanol tax credit would be applicable to the biodiesel product.

Ethyl levulinate, which is also produced using ethanol, could conceivably be eligible for a portion of the ethanol blender tax credit. However, since the IRS hasn't ruled (or been asked to rule) on this matter, an EL producer would have to petition for a ruling. That process could be lengthy, but a favorable ruling could have a significant impact on the costs of producing EL. However, it is important ro realize that only EL destined for blending to a transportation fuel would qualify for the credit. *Fuels blended with ethanol or an ethanol derivative do not qualify for the credit if they are used for any application other than highway transportation.*

2.6.2 Chemicals

The diversity of the chemical products that can be derived from LA are such that a detailed assessment of all of the regulatory drivers for these industries is beyond the scope of this report. However, for products such as pesticides/herbicides that have the potential to be considered pollutants (as opposed to those that are chemical feedstocks for other products) some meaningful information can be gathered from recent government actions.

Early in November 2000, EPA is finalizing its action to ban the use of mixing zones that dilute toxic chemicals discharged into the Great Lakes system. This action prohibits new discharges of toxic chemicals into mixing zones and phases out the use of existing mixing zones in the Great Lakes over the next ten years. It has been found that toxic discharges into mixing zones (a mixing zone is an area where pollutants are mixed with cleaner receiving waters to dilute their concentration in the water) build up in the Great Lakes system and threaten human health, aquatic life and wildlife. This regulation will ban up to 700,000 toxic pounds annually of chemicals that are discharged into the lakes and that accumulate in fish and wildlife, including mercury, dioxin, PCBs and pesticides (including DDT, DDD, DDE).

Of more significance to the chemical industry is EPA pursuit of similar rules nationwide. Late in 1999, a spokesman for the Chemical Manufacturers Association (now the American Chemistry Council) stated that if extended nationally the cost of complying would run "tens of billions of dollars."

Other actions specifically targeted at pesticide/herbicide use include the recent banning of the common pesticide chlorpyrifos and heightened concern about child health risks associated with other chemical contaminants.

In June 2000, the EPA and the manufacturers of chlorpyrifos (an organophosphate) agreed to eliminate nearly all household applications of this insecticide. It is the most common insecticide exterminators use to kill cockroaches and termites and it is applied to many fruits and vegetables. Professionals will still be able to use the product, but exterminator companies have agreed to phase out its use where children could be exposed. EPA estimates that chlorpyrifos accounted for 800 unintentional poisonings in 1999 and says that it is highly persistent in the environment. Vice President Elin Miller of Dow Agrosciences, the primary supplier of this substance, says it is safe when used as directed, but admitted that 1996 Food Quality Protection Act "has fundamentally changed they way in which pesticides are regulated in the United States." It should be noted that EPA plans to review additional organophosphates by the end of the year and that the agency has already banned the use of methyl parathion and azinphos methyl.

The Clinton White House has implemented the Children's Health Initiative, which will involve toxicity testing of hundreds of chemicals to see if they are making children ill. Citing a change in illness patterns among children, this initiative is gaining popular and bi-partisan support. Representative John Porter of Illinois, Chairman of the Appropriations Subcommittee on Labor, Health & Human Services, Education and Related Agencies proclaimed in an opening hearing on the subject that protecting our children from environmental dangers is "one of the most important subjects of all." This may be beneficial to the extent that DALA is less toxic. This could be important to future marketing efforts.

With respect to any products that Biofine wishes to introduce into these markets, an additional item to consider is the time and expense of registering and labeling a pesticide for use. Some sources suggested that this process could take 8-10 years and may cost as much as $50 million. The Food Quality Protection Act (FQPA) and prior legislation that it supports also allow EPA to levy registration and maintenance fees on manufacturers to cover its costs under the legislation.

Through the FQPA, the 104th Congress enacted significant changes to the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA), governing U.S. sale and use of pesticide products, and the Federal Food, Drug, and Cosmetic Act (FFDCA), which limits pesticide residues on food. The vehicle of these changes was H.R. 1627, the "Food Quality Protection Act of 1996" (FQPA), enacted August 3, 1996, as Public Law 104-170. Under FIFRA, the new law will facilitate registrations and re-registrations of pesticides for special (so-called"minor") uses and authorize collection of maintenance fees to support pesticide re-registration.

Food safety provisions will establish a single standard of safety for pesticide residue on raw and processed foods; provide information through large food retail stores to consumers about the health risks of pesticide residues and how to avoid them; preempt state and local food safety laws if they are based on concentrations of pesticide residues below recently established Federal residue limits (called"tolerances"); and ensure that tolerances protect the health of infants and children.

2.6.3 Power

Policy makers have taken a serious interest in renewable power, especially at the state level. The restructuring bills of many states now include some provision to encourage the production of green energy through renewable portfolio standards (RPS), system/societal benefits charges (SBC), or both. For renewable energy advocates, the movement appears to be the ticket to rapid market penetration and widespread acceptance of their technologies.

Renewable Portfolio Standards (RPS)

Renewable portfolio standards are emerging as an important mechanism for states to encourage the development of renewable energy resources. RPS provisions usually dictate that a minimum amount of renewable electricity be included in the state's electricity mix. Although Federal legislation is also being considered, states are seizing the opportunity in their restructuring bills to move renewable technologies of all sorts to the forefront of public policy. Of course, there are some states that appear to be taking a more serious approach to promoting renewable energy than others. For example, most outside observers believe that, despite the apparent size of Maine's RPS provision (30% starting in 2000), it will do little to encourage new renewable energy use. About 45-50% of Maine's electricity is already supplied by qualified renewable energy sources. Exhibit 13, on the following page, highlights some of the more important aspects of the RPS provisions passed in states to date.

EXHIBIT 13: Renewable Portfolio Standards[25]

State	Renewables Standard Level	Status As of July 2000
Arizona	0.2% in 2001; 0.8% by 2004; 1.1% for 2007–2012 after review in 2004; 50% solar	RPS funded by SBC charges and utility and customer contributions; rulemaking later in 2000
Connecticut	Class I or II Technologies: 5.5% in 2000, 7% in 2009; Class I Technologies: 0.5% in 2000, 6% in 2009	Individual suppliers may petition for two-year delay; decision not to apply the RPS to default suppliers currently under appeal to state Superior Court
Maine	30% in 2000 and thereafter	RPS took effect in March 2000 and includes high-efficiency cogeneration systems
Massachusetts	1% new renewables in 2003, 4% in 2009, and increasing 1% per year	RPS not adopted for existing renewables; focus on creating tradable environmental certificates
Nevada	0.2% in 2001, 1% in 2009; 50% of standard must come from new solar	One utility may be exempted until 2005; two major utilities have sued to overturn restructuring law
New Jersey	Class I or II Technologies: 2.5%; Class I Technologies: 0.5% in 2001, 4% in 2012	Implementation regulations not yet determined, though draft regulations have been released
New Mexico	5% of energy to serve standard-offer customers	Standard depends on availability of New Mexico renewable resources; expected to start at lower than 5%
Pennsylvania	For PECO, West Penn, and PP&L, 20% of residential customers served by competitive default provider: 2% in 2001, increasing 0.5% per year; for GPU, 0.2% in 2001 for 20% of customers, increasing to 80% in 2004	Requirement imposed on service-territory basis; GPU's solicitation of default suppliers did not receive any bids; PECO Energy and PP&L auctions due later this year
Texas	New and existing renewables: 1280 MW by 2003, 2880 MW by 2009 (2000 MW must come from new renewable resources)	Regulations issued in December 1999; methane gas produced by landfill qualifies, with electric conversion factor; state ISO is working on creating credit-trading system.
Wisconsin	0.5% by 2001, increasing to 2.2% by 2011 (0.6% can come from pre-1998 facilities)	Draft regulations sent to the Wisconsin Legislature in March 2000

---

[25] Porter, Kevin, *Biomass Power and State Renewable Energy Policies Under Electric Industry Restructuring*, Bioenergy 2000, October 2000.

System/Societal Benefits Charges

While RPS provisions mandate renewable content within a given state's generation mix, SBC provisions usually provide funding to support the development of renewable energy and/or energy efficiency projects. The funds are created through surcharges levied on consumers and are intended to provide a public benefit. Exhibit 14 provides highlights on the SBC programs adopted so far.

EXHIBIT 14: System/Societal Benefits Charges[26]

State	Level of Support for Renewables	Status As of July 2000
California	$135 million per year for four years beginning in 1998	45% used to support existing renewables; 30% to support new renewables; 10% to support solar and other emerging renewables; 15% to support green power market
Connecticut	Approximately $14 million per year in 2000; $30 million per year in 2004 and thereafter	First investment made in green power aggregator
Delaware	$1.5 million per year for renewable energy and energy efficiency	Implementation efforts just getting under way
Illinois	$5 million per year for 10 years beginning in 1999; also a $250 million clean energy fund	Ongoing grant and rebate programs: funds to date have gone largely to PV and solar thermal systems
Massachusetts	Approximately $26 million per year from 1998 on	Litigation prevented fund disbursement, but favorable court decision will allow new funds to be released
Montana	Approximately $2 million per year, 1999–2003	Utilities receive credit against SBC allocation for expenses on covered programs; state administers remaining funds
New Jersey	$29–$35 million per year, 2000–2007	Board of Public Utilities considering two different fund administration proposals
New Mexico	$4 million per year beginning in 2001	Restructuring law contemplates a revisiting of financial support for renewables
New York	$15 million for three years beginning in 1999	Willow tree planting and co-firing with coal-fired plant
Oregon	$8.7 million annually for 10 years	Oregon PUC staff proposed draft rules in April 2000 for collecting SBC funds; separately, a task force is exploring program administration and implementation issues
Pennsylvania	$11million per year fund, including renewables, 1999–2005; Renewable Energy Pilot Fund raises $3.9 million per year, 2001–2002	Renewable Energy Pilot largely focused on solar; only one utility SBC fund in operation; merger settlement will add $20 million to PECO SBC fund
Rhode Island	Approximately $2 million per year 1998–2002	Has funded wind studies, landfill gas, and PV projects
Wisconsin	Approximately $3.6 million per year	Grant applications and requirements to be established

---

[26] Porter, Kevin, *Biomass Power and State Renewable Energy Policies Under Electric Industry Restructuring*, Bioenergy 2000, October 2000.

Biomass Eligibility under RPS and SBC Programs

Exhibit 15 provides an additional perspective on the eligibility of biomass under these programs. More than half the 17 states implementing an RPS and/or an SBC program have placed restrictions on the eligibility of biomass. The reasons include concerns about sustainability and emissions. These restrictions are even more problematic when one considers that some of the states haven't even clarified their meaning. Several, including Connecticut, are waiting for a claim from a sustainable biomass power supplier before clarifying their definitions.

EXHIBIT 15: Biomass Eligibility[27]

State	Eligibility of Biomass
Arizona	Biomass resource must be in-state
California	Biomass must be in-state, must not be utility-owned, and must be off the fixed-price portion of the utility power purchase contracts
Connecticut	For the RPS, a biomass generator is a Class I renewable if facility began operating after July 1998, and if the biomass fuel "is cultivated and harvested in a sustainable manner"; Class II renewable applies to existing MSW and biomass facilities that do not meet Class I definition; low-emission advanced biomass conversion technologies are the only biomass technology eligible for the SBC
Delaware	To be determined
Illinois	Dedicated crops for energy production and organic waste biomass (animal manure, crop residues, wastewater sludge); construction debris, waste wood, and office waste are not allowed
Maine	Biomass under 100 MW; high-efficiency co-generation of any size qualifies
Massachusetts	Existing biomass and MSW meeting certain air emission limits are eligible for existing RPS, should one be designed. Only low-emission, advanced biomass eligible for "new" tier of RPS; considering an NOx-based criteria to qualify advanced biomass; incremental generation from retrofitted biomass may qualify if emission threshold met; recommended that biomass co-firing be eligible as long as emissions threshold met (only the renewables output qualifies); considered "new" on case-by-case basis
Montana	Biomass eligible for SBC funds; Montana Power expressed preference for solar, wind, and geothermal in 1999 RFP; RFP in 2000 only for wind
Nevada	Biomass must be "naturally regenerated"; precise definition under negotiation in stakeholder group
New Jersey	Biomass qualifies for existing RPS tier; only biomass harvested in sustainable manner qualifies for new RPS tier
New Mexico	Renewables must be low or zero emissions; biomass limited to landfill gas, anaerobic digestion, or fuel cells
New York	Biomass eligible; focus has been on growing of willow trees for biomass co-firing
Pennsylvania	Biomass eligible for RPS and SBC
Oregon	Dedicated energy crops "available on a renewable basis," landfill methane, digester gas, and "low-emission nontoxic biomass based on solid organic fuels from wood, forest and field residues"
Rhode Island	Sustainably managed biomass
Texas	Biomass and biomass-based waste products qualify, including landfill gas; excludes waste products from inorganic sources; Biomass co-firing essentially not allowed—fossil co-firing limited to 2% of annual fuel input; existing fossil plants cannot be repowered to use renewable fuels
Wisconsin	RPS only applies to electric utilities; biomass and biomass co-firing eligible

---

[27] Porter, Kevin, *Biomass Power and State Renewable Energy Policies Under Electric Industry Restructuring*, Bioenergy 2000, October 2000.

The net benefit of RPS/SBC programs for biomass energy is difficult to assess. However, eligibility restrictions, vague definitions, and short time frames do not provide investors any assurance that state governments will be committed to long-term support of biomass power projects. Instead, it appears that good marketing and buy-in from motivated consumers will be the most likely mechanism for creating and sustaining new green power markets over the next 20 years.

3.0 MARKET DEVELOPMENT PLAN

3.1 General Market Development Planning

It is part of Biofine's underlying business strategy to attempt to build large-scale facilities. This strategy is premised on the need to profitably recover development costs and to capture economies of scale that will reduce production costs. In order to provide a stable platform for business growth, Biofine understands that at least one high-volume market must be established. To be successful at low initial market penetration rates, Biofine also recognizes that the larger this market, the better. Provided that start-up operations can break even supplying products to this market, added-value products such as high- value chemicals will provide the profit margins needed to make the projects succeed.

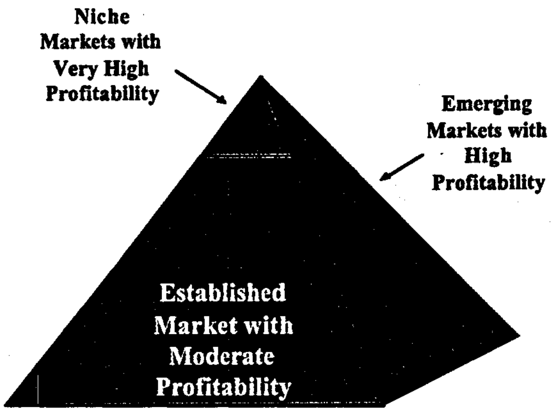


EXHIBIT 16: Value Added Pyramid

This is a sound business model and it has been employed by many industries to provide stable growth and returns. The key question is how to develop the high-volume markets that currently present themselves to Biofine (primarily energy markets) as a platform. Within these markets, Biofine's products have the advantage of being environmentally beneficial, but they are also more expensive than the incumbent products. This does not necessarily rule out market penetration, but it does suggest that Biofine will have to focus its efforts on identifying creative strategies to market its platform product and pursue as many incentives as possible.

To accomplish this, Biofine must first address any technical and regulatory issues that stand in the way of future project development due diligence. Clear fact sheets for each proposed product that include material safety data sheets, handling guidelines, fuel analyses, discussions of helpful incentives and regulations, and a marketing strategy are all needed. The remainder of this report provides ANTARES' suggestion on a few specific items that should be addressed.

3.2 Addressing Technical Issues: Recommendations/Action list

3.2.1 Transportation / Home Heating Fuel Applications

At a minimum, MTHF should be blended to regular gasoline (test fuels based on premium and mid-grade gasolines would also be desirable) at several levels (say 5, 10 and 15 percent by volume) and RON and MON determinations made on the blends. Blending octane behavior can vary with the concentration of the blending component in the base fuel and the composition of the base fuel (its olefin content, for example).

Summarizing, the following actions should be pursued:

- Conduct additional testing for MTHF that includes multiple blends and RON, MON determinations.
- Perform a detailed estimate of MTHF production costs that carefully consider the costs of producing hydrogen for the process.
- Conduct additional testing for ethyl levulinate to include:
    - Identification of economical additives that provide solution stability for EL/FO blends
    - Cetane number determination
    - Once a stable mixture is identified, Biofine should conduct other tests to accurately assess other fuel characteristics such as flashpoint. Tests made to date for this parameter were inconclusive
    - Short-term engine tests.
- For EL in home heating oil market, ANTARES recommends that Biofine commission diffusion burner tests.

These tests are all intended to provide information critical to certifying and complying with federal regulations and consensus specifications for using these fuels in specific markets.

3.2.2 Power Applications

As noted in the previous discussion, ANTARES does not expect any show-stopping technical issues when using EL blends in combustion turbines, provided the fuel can be completely characterized and that blending issues with fuel oil are addressed. It will be important to thoroughly characterize the fuel and obtain performance guarantees from the manufacturer of the proposed power plant. Specifically:

- Conduct laboratory testing and fuel analyses that include
    - an ultimate analysis
    - smoke point and other parameters specified by ASTM test protocols for combustion turbine fuels. These tests can be conducted relatively inexpensively and will pave the way for more expensive tests that will be required to certify turbine performance on the fuel.

- Work with a major turbine manufacturer to guarantee fuel blend performance using laboratory combustor tests or obtain hard performance guarantees.
- Perform a detailed heat/energy balance analysis with the specific intent of accurately modeling overall thermal/electric efficiency of a combined cycle plant using various EL/FO blends.

Biofine is also considering the use of process char for on-site heat/power. This seems to be a practical and economic way to convert this waste stream into an added-value product. However, the characteristics of the char are such that special handling systems may be required. ANTARES recommends that Biofine seek input from the manufacturers of industrial-size packaged biomass boilers to determine the most beneficial technology. ANTARES also suggests that Biofine conduct an ultimate analysis on the char. This will provide valuable information to the manufacturer and help in making final decisions about the appropriate technology.

3.3 Addressing Regulatory Issues: Recommendations/Action list

3.3.1 Fuel Applications

With Respect to the Clean Air Act

Obtaining a 211(f) waiver is of utmost importance if Biofine intends to market a gasoline replacement fuel. To obtain this waiver, Biofine must demonstrate that MTHF is "substantially similar" to fuels or additives used in certifying model year 1975 cars and light trucks. Proving this point will require a well-planned testing regime. Specifically, Biofine will need to:

- develop emissions data from vehicles operated on base gasoline and gasoline blended with MTHF,
- determine EPA's position on using MTHF as a cosolvent with ethanol and gasoline,
- obtain data on evaporative emissions,
- submit materials compatibility data,
- evaluate vehicle driveability on MTHF-blended fuel,
- make maximum use of existing data in 211(f) application.

With Respect to Registering the Fuel with the EPA

As noted earlier, registering MTHF as a fuel is a different process from obtaining the 211(f) waiver and extensive health effects testing can be required. The extent of this testing is determined in part by the amount of oxygen in the finished fuel. The threshold that triggers mandatory health effects testing is 1.5 percent oxygen in the finished fuel. ANTARES estimates that MTHF blends in excess of 11.3% on a volume basis, would have this impact on a regular gasoline.

Therefore ANTARES recommends the following:

- Pursue/support legislative action that changes or waives the CAA oxygen requirement (2%) for RFG when using a substantially renewable fuel.
- Provided that the first can be obtained, market MTHF as an oxygenate with blend volume targets below 11.3% on a volume basis to avoid extensive health testing.
- Begin registration process with EPA as soon as possible if Biofine wishes to pursue this market and begin outlining test protocol to meet other testing requirements.

With Respect to EPAct

One approach for Biofine to improve its market under EPAct would be to propose a light-duty fuel, which might substitute for E-85 in current Flexible Fuel Vehicles. This could be done as a new fuel (requiring a rulemaking), or perhaps simply as a fuel meeting the "biologically derived material" standard. Again, under either of these approaches, DOE might be the least of the barriers. Section 211(f) of the Clean Air Act requires EPA to approve use of any fuel not "substantially similar" to current petroleum fuels. A minimum of six months is required for EPA's ruling from the time the agency is petitioned for a 211(f) waiver. Under current programs, there is growing concerns about FFVs which are not using any alternative fuel, so any efforts here would only be as good as the commitment to make the fuel widely available at a competitive price with gasoline.

To move forward with its diesel replacement fuel, the simplest approach might be for Biofine to make the claim that its fuel is basically another form of biodiesel, and should be accounted for under the program in the same manner. Proposing a distinctly separate heavy-duty fuel would require a full rulemaking process. DOE is struggling with the question of how to address an alternative diesel fuel, Fischer-Tropsch diesel (FTD). DOE's difficulty goes back to EPAct's focus on vehicles, and not fuels. Since FTD could be used in any diesel vehicle, DOE is concerned that designating FTD an alternative fuel could make every diesel vehicle an AFV, a highly unpopular result with the alternative fuels industry. At this time, it is unclear how this situation will be resolved. It has been suggested that FTD be treated like biodiesel, but without the specific legislative authority, it is unclear that DOE could do this. Therefore, moving forward with a new Pencor alternative diesel fuel might be difficult, or at least complex.

Whichever approach is selected, there may be something different coming from DOE in the future. DOE recently held several workshops on a potential rule to cover Private and Local Government fleets. Such a rule would be limited to light-duty vehicles, but would expand the potential market for alternative fuels significantly. (Some projections have indicated that a Private and Local rule might require acquisitions of 400,000 AFVs a year.) DOE has proposed several options, including an AFV acquisition program similar to existing ones, a slightly more flexible AFV acquisition program which allows some credit based on the amount of alternative fuel used, and a program based solely on fuel use (a Replacement Fuel Program). At this point in time, if a rule or guidance is proposed for moving alternative fuels forward, it will likely include elements of either of the last two. Thus, there would be some emphasis on fuel use to meet requirements, or the requirements themselves might be based on fuel use. DOE is also considering whether to develop requirements for transit bus fleets. Transit buses currently account for 1% of the transportation petroleum use in the United States. A requirement for transit buses could allow a fuel provider to focus initially on a few large, centrally fueled fleets to generate a sizable demand for his early fuel production. A final decision on a Private and Local Rule will be left to the Bush Administration. However, these rules, at best, may be imposed by 2002 or 2003.

With Respect to the Ethanol Tax Credit

The size of the transportation fuel market for EL mandates that Biofine consider pursuing a private ruling from the IRS on the eligibility of the fuel to receive a pro rata share of the credit for EL. The fuel certainly meets the policy specifications that the credit was intended to encourage and the economics of the fuel could be substantially improved. ANTARES suggests that Biofine contact the IRS to investigate the feasibility of such an application and determine the related costs as soon as possible.

3.3.2 Power Applications

The analyses presented in this report suggest that Biofine's plans to generate and sell green electricity will require some green market or renewable energy policy support to be successful. ANTARES suggests that Biofine take pro-active steps to supporting legislation on a Federal level and in states that appear especially attractive for biomass power. Although many states have already passed RPS/SBC legislation, the terms of these incentives are often limited. For example, New York's SBC program is due to expire and efforts are underway by various renewable energy groups to renew it. Biofine should consider investing some time in supporting these efforts and representing its interests. However, ultimately ANTARES does not expect state or Federal renewable energy regulations to present the greatest challenge to selling green power. It will be far more important for Biofine to make a strong case to the environmental community that Biofine's proposed feedstocks and the resulting fuels meet their requirements for producing ***green*** energy.

3.3.3 Chemicals

The most obvious action item for Biofine in this sector is to pursue registration of DALA as an active ingredient in specific pesticide applications. The expense and lead time on such an application is such that gaining a thorough and early understanding of the legal requirements for this product will be crucial to developing the market or securing an industry partner. As a first step, ANTARES suggests that Biofine obtain a copy of the pesticide registration application and begin developing test protocols that will provide sufficient data to satisfy EPA's new health and safety requirements. As an adjunct, ANTARES suggests that Biofine secure the services of one of the many chemical industry consulting firms that specialize in health and toxicity testing. These companies are intimately familiar with international government regulations regarding new chemical applications and can develop test protocols, conduct testing, and assist Biofine make applications to the appropriate agencies. In lieu of this strategy, Biofine may also seek to secure an industry partner that is capable of carrying out these services independently or at least sharing the costs.

3.4 Market Opportunities/Priorities

The information collected in this report suggests the following:

1) Energy markets (electricity, heat, power) will be difficult to penetrate without significant incentives, additional testing, and cross subsidization from sales to high-value chemical markets. Recognizing that the costs used in this report for the chemical derivatives relied on large-scale plant economies, an interesting question arises. At what scale must biorefineries be developed to make LA derivative production costs low enough to compete on their own in the obvious, high-value chemical markets? ANTARES believes that resolving this questions as accurately as possible is key to successful and profitable deployment of Biofine's technologies for two reasons. 1) The energy markets are lower in value and in all probability products sold into this market might be marginally profitable if government incentives or special market conditions can be brought to bear. However, in the absence of such conditions, product sales into this market should be considered secondary and only be considered if economic modeling suggests a net benefit in lowering production costs for other LA derivatives that can be obtained. 2) There are several chemical markets which appear large enough to support the construction of dedicated Biofine biorefineries at moderate scales. Confirmation of the economics to support this statement should motivate Biofine to place a high priority on identifying chemical industry partners that find it cost-effective to use LA derivative products in their capacity addition plans.

2) In this report, Texas emerged as a state with considerable potential for siting a biorefinery. This may seem counter-intuitive since it is the heartland of America's petroleum industry, but consider the evidence:

   A) Texas has enacted legislation calling for a substantial increase in green electricity, leading the nation in expected additional green MWs needed.
   B) Texas leads the nation in the number of refineries and refining capacity. These facilities blend fuels to specification and provided that some of the technical issues with MTHF and EL blends are resolved, these products could be blended at these sites. If refiner interest could be stirred, this would probably be more economical than blending at the rack as is done with ethanol for technical reasons.
   C) Many of the relevant chemical industries, such as BDO, are concentrated in Texas.
   D) Texas also has a substantial biomass resource base.

Taken as a whole, these facts suggest that significant synergies may be obtained by locating a biorefinery in Texas. Strategically, gaining the interest of the chemical industry in LA derivative products may provide the credibility Biofine needs to also attract refiner interest.

3) The chemical market for BPA (for which Biofine hopes to substitute DPA), BDO, and THF are substantial. Although there is overlap between BDO and THF, these markets seem to offer both volume and price points that are attractive as first markets for a biorefinery. A more detailed mass flow analysis will be required to more precisely determine economic feasibility, but based on early projections, Biofine's production costs for these commodities are far below industry sales prices. The growth in these industries is motivating manufacturers to build new plants or expand capacity at existing ones. The list of key manufacturers in the U.S. is small and Biofine should consider these companies in their pursuit of strategic partners.

4.0 BUILDING THE FIRST LARGE-SCALE PLANT

4.1 Strategy

The wide range of products that can be manufactured through Biofine's biorefinery technologies is such that it provides a challenge to focus on downstream markets. Having considered the general market and trends for potential Biofine products, ANTARES, in consultation with Chemical Industry Services, Inc. (CIS) suggest that the Biofine proceeds with its large scale commercialization plans based on the following precepts:

- Biofine should seek to convert as much LA as possible into high value chemical products. Chemicals appear to have the best chance of providing high profit margins, but initially market penetration may be small and some marketing will be required to identify customers.
- Plan to convert the remaining LA produced from the first large-scale plant into EL. The EL produced will initially be used to power a combustion turbine to generate electricity *and* for the home heating oil market. In both cases Biofine must stress the importance of this fuel to provide "green peaking energy" to mitigate price spikes in the electricity and fuel oil markets. However, as markets for chemical products increase, a growing percentage of the plants should be transitioned away from the lower value energy markets.
- If possible, Biofine should seek to locate the first plant: 1) in area where home heating oil and electricity prices are a major concern; 2) there is coincident biomass resources from a reliable supply such as pulp and paper (P&P) mills, MSW, agricultural, or furniture manufacturing wastes; and 3) there is sufficient bulk transportation infrastructure to support the shipping and receipt of large quantities of LA and EL.

- LA from Biofines smaller plant at South Glens Falls should be used: 1) to sell into the existing LA market to the greatest extent possible; and 2) provide product samples to prospective customers.

4.2 Market Penetration Expectations (Initial Products)

4.2.1 DPA

As noted previously, the first large scale Biofine will rely heavily on EL energy products to provide the manufacturing economics necessary to produce low-cost LA. However, preliminary economic analyses suggest that it will be chemical products, even produced in small volumes, that will drive the plants profitability. Therefore, selecting the right chemical product to initially pursue will be critical to the success of the plant. Based on information received from CIS, ANTARES suggests that DPA be considered the top candidate for early production at Biofines first large-scale biorefinery. Biofine plans to promote DPA as a replacement for bisphenol A (BPA) in the production of polycarbonate resins and epoxy resins. DPA was used in this application but because of its cost and availability, it lost the market to BPA.

The 2 billion pound market for BPA continues to grow with the end-uses being low cost, high volume plastics like polyesters and polycarbonates. Biofine should be able to sell 300MM lbs. (a 15% penetration) to allow a BPA producer to test DPA in polymers and delay the need to build another BPA expansion. Targeted manufacturers should find $0.40 / lb. considerably below their cost of around $0.60 per pound, and the market penetration figure reflects the belief that this will provide enough incentive in a make/buy decision.

Work funded by Biofine shows that some interesting polymer properties can be obtained by adding DPA to certain BPA formulations. The work also shows that interesting properties can be obtained in the resins with low level addition of DPA as a polymer modifier which should allow for a premium price for DPA. Although additional research will be required, Biofine should be able to sell about 20MM pounds as an additive with a price of $0.60 a pound.

4.2.2 Formic Acid

The Biofine process produces one pound formic acid (FA) for every 2.5 pounds of LA in an aqueous solution. This material can be recovered as FA or as a formic acid salt. There is a U.S. market for approximately 60 million pounds of FA at approximately $0.45 per pound. Based on the industry experience of CIS, Inc., Biofine could move up to 50% of the market with a price of $0.20-$.22 per pound without being a disruptive factor in the market. In fact, current producers might purchase 30 million from Biofine to accomplish the dual objective of obtaining low cost FA and maintain control on new production.

4.2.3 Ethyl Levulinate

Solvent Market

The solvent market is huge with all companies looking for new solvents that give better performance. Conventional high volatile organic compounds (VOC) solvents that have undesirable attributes such as being flammable, toxic, irritating and environmentally undesirable. EL may be an excellent candidate for this purpose especially as a solvent in the food, electronic, low VOC, degreasing, printing ink, and other applications. At least one West Coast-based company is very bullish on LA esters and has an ongoing project that involves a process for EL synthesis from LA and EL application studies.

Biofine will be producing hundred's of millions of pounds of EL for use in fuel applications at a cost of well under $0.25/lb. Several LA esters are currently produced by small specialty chemical companies and sold into high value added markets such as flavor and fragrance uses. Prices are generally over $10.00/lb.

Our market forecast shows new applications for EL as a commercial solvent at 30 million pounds and a price of $0.50 for the early market volumes. This is at 30% of the current price for THF. Thirty million pounds is likely to be a conservative estimate of under 5% penetration. At this price level EL is also favorably priced against Ethyl Acetate and Dibutylether.

Electricity Market

Over 6,000 MW of required renewable capacity is contained in state legislation that has passed or is currently pending. Since this capacity is legislated, it represents a relatively firm bottom to the potential market that Biofine has to consider for green power. About 2,000 MW of the total is scheduled for deployment in the Northeast, New England, and Mid Atlantic. Excluding Massachusetts (legislation in this state is unlikely to spur new renewable capacity) approximately 1,300 MW of new capacity has been mandated in the same general region that is currently experiencing a home heating oil crisis. This coincidence with another related market for EL could be critical to the first plant's success. This region has the added attraction as being very well suited for biomass derived generation. Further, solar, wind, and geothermal resources are less prevalent in these areas than in other regions throughout the country. As a result, ANTARES considers the general area along the middle and upper east coast an excellent target area for deploying an EL based generation plant. The following discussions focus on this region.

Before considering the penetration that Biofine generated green power may make into the describe market, one must consider the price and acceptance that electricity derived from a fuel oil / ethyl levulinate blend is likely to receive. Serving the needs of legislative mandates, a good first target, will require that states agree to the type and manner in which the renewable power is produced. As discussed in section 2.3.1 *(Electricity Section)*, decoupling the "green portion" from the fossil portion of the electricity produced may offer one way to clearly demonstrate that green power is produced at plant using a blended fuel. Further, depending on the results of combustor tests, it may prove possible to use very high EL blends or even eliminate fuel oil from the mix in combustion turbine applications. Using 100% EL would alleviate concerns that environmental groups may raise with regards to cofiring EL with fuel oil. In either case, the value of this renewable power will be based on supply and demand. In states like New Jersey where it is unclear how renewable power mandates will be met, distribution companies may pay a premium for large-scale, baseload renewable power simply because they have no choice and are legally bound to provide this service. Based on the UCS report[28], and data collected from DOE, one might expect such a premium to be on the order of 2 cents/kWh for renewable power.

Obtaining a green premium from customers directly, outside of a legislative mandate may be tougher. In the absence of such government support, customers must be individually convinced of the value of this generation and their reaction to any product is likely to be specific to its characteristics. Here, the Green-e certification can help, but even with such certification, recent results suggest that market penetration is likely to be low. It may even be lower in states where a legislative mandates exist since customers may feel that their state governments are already doing there part for them. However, on the flip side, perception, not market forces may play a more important role in deciding the magnitude of the premium. Good marketing may be able to convey to customers that larger premiums are needed to provide sustainable, long-term renewable energy solutions.

ANTARES considered these factors carefully assessing the probable premium and potential market penetration that Biofine might expect from providing a green power product. Several scenarios are presented below. In presenting this table two general assumptions were made: 1) the Northeast because of its other energy demands relevant to the Biofine process is the target region; and 2) green premiums would be added to the average wholesale electricity costs for the current generation mix in the region; and RPS provide the impetus for creating a green power market.

EXHIBIT 17: Green Power Potential

General Assumptions

RPS Market for NE/New Engl/Mid Atlantic	1,300	MW
1999 Total Capacity for Region	99,650	MW
2005 Non-Renewable Capacity for Region	108,140	MW
2010 Non-Renewable Capacity for Region	125,250	MW
Wholesale Electricity Price Current Mix	3.0	cents/kWh

New Plant Assumptions

Electricity Plant Heat Rate	7,000	Btu/kWh
EL Heat Content	95,425	Btu/gal
New Capacity Availability	80%	

Notes

1) PJM and ISO NE average wholesale prices in 1999 were about 2-4 cents/kWh PJMs average jumped to as high as 9 c/kWh in July 1999
2) Non-renewable generation forecasts taken from EIA Annual Energy Outlook for Northeast / New England / MidAtlantic region
3) New non-renewable capacity calculated by subtracting 1999 non-renewable from forecasts of total non-renewable capacity in 2005 and 2010

	Electricity Prices (cents/kWh)			Total Market (MW)		Market Penetration		EL Demand (Kgal)	
Scenario Name	Wholesale	Premium	Total	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010
RPS Market Only (conservative)	3.0	2.0	5.0	650	1,300	2.5%	5.0%	8,354	33,415
RPS Market Only (optimistic)	3.0	4.0	7.0	650	1,300	4.0%	12.0%	13,366	80,197
RPS & 1% new non-renewable (conservative)	3.0	2.0	5.0	735	1,556	2.5%	5.0%	9,445	39,996
RPS & 1% new non-renewable (optimistic)	3.0	4.0	7.0	735	1,556	4.0%	12.0%	15,112	95,989

---

[28] UCS, *Clean Power Surge: Ranking the States*, April 2000.

The extreme cases shown in this table (RPS Market Only-conservative, RPS & 1% of non-renewable-optimistic) demonstrate that annual demand for EL in the electricity market could stretch over a large range. In the more pessimistic cases, approximately 40% of the LA output of just one 500 tpd biorefinery could meet early demand. By 2010, approximately 10 times this amount might be required if optimistic predictions hold true. However, it should be noted that even using optimistic green pricing premiums, green power is likely to provide very low margins. As stated earlier, it will be higher value chemical commodities that ensure biorefinery profitability.

Transportation Fuel

In the highway diesel fuel market, ethyl levulinate (EL) will compete directly with biodiesel as a diesel fuel replacement or blending stock. Over the past half-dozen years, biodiesel has reached an estimated production of from two million to four million gallons annually. A small portion of this is marketed as industrial solvent (fatty acid methyl esters, or FAME), but the majority is blended, usually at the 20 volume percent level, to diesel fuel. In reaching even this modest level of acceptance as a fuel, biodiesel has had several key advantages:

- The Energy Conservation Reauthorization Act of 1998 (P.L.105-388, 1998) specifically granted it status under the Energy Policy Act of 1992 (EPAct) as a replacement fuel for which fleets may receive credit toward the purchase of alternative fuel vehicles required by EPAct
- Strong identification in the eyes of government (all levels) and the public with regional and local agricultural interests (analogous to ethanol), and with the economic fortunes of farming communities
- Demonstrated technical benefits as a fuel, including cetane number improvement, compatibility with most in-service diesel engines, acceptance by engine manufacturers and standards organizations and measurable emissions improvements
- Completion of the Environmental Protection Agency's health effects protocols.

The vast majority of U.S. biodiesel is made from soybean oil (hence its strong association with farming interests) and methanol. EL, on the other hand, is made using ethanol, an agriculturally derived commodity that enjoys favorable tax treatment from the Federal Government and several state governments when it's used as a motor fuel. Since EL in essence "contains" at least some ethanol, legislation and/or an Internal Revenue Service ruling might be achievable which would grant it some or all of the ethanol tax credit. Both avenues should be pursued; in this market penetration estimate, we assume only that EL has achieved the same status as biodiesel with respect to the Energy Policy Act (first item, above). This should be an easy and persuasive argument to make to Congress, but the process is apt to be lengthy.

We further assume that:

- Biofine will commence marketing EL-diesel fuel blends with evidence in hand that they are compatible with most or all major engine manufacturers' products and meet applicable ASTM standards
- EL, as produced, is essentially sulfur-free (at least below 15 ppm) and that its combustion emissions are below those of conventional diesel fuel in at least some particulars
- Biofine has laid the groundwork by associating its product in target markets with other locally produced commodities (e.g., agricultural residues, ethanol) and by securing the interest and support of regional fleet operators for demonstration programs
- EL-diesel fuel blends will be made and marketed in or near regions where ethanol is produced to minimize the cost of the ethanol input to EL production.

Under these assumptions, a reasonable upper-limit estimate is that within two years of commencing EL production, a 500 ton-per-day Biofine plant could dispose of 10 percent of its output, or about 1.9 million gallons annually, to the transportation fuel market. This is approximately equal to the low estimate of biodiesel's annual production. Greater penetration of this market in the 2000 to 2005 time frame does not seem likely.

Beginning in mid-2006, road diesel fuel must meet stringent sulfur content standards under EPA rules promulgated in early 2001. According to a study conducted for the American Petroleum Institute (API) by Charles River Associates (CRA) and Baker and O'Brien, Inc., the price of diesel fuel may increase by as much as 14 cents per gallon. This can be expected to improve the economic competitiveness of EL (and biodiesel). CRA predicts EPA's rules will also produce a near- to medium-term shortage of diesel fuel of as much as 320,000 barrels per day. This is 12 percent of projected 2007 diesel fuel supply. The rules are expected to cause some refiners to reduce or end production and others to close refineries. API has said the national fuel distribution system cannot reliably supply diesel fuel at or below 15 ppm sulfur while also handling much higher-sulfur products such as jet fuel and heating oil. Early impacts of the diesel desulfurization rules, according to CRA, could include regional shortages, price spikes and spot outages of up to a week in remoter, less-populated areas.

The transition to ultra-low-sulfur diesel (ULSD) fuel may also create (or be blamed for) engine performance or maintenance problems, as during the 1993 introduction of lower-sulfur diesel fuels, CARB (California Air Resources Board) diesel fuel in particular. It seems to be widely accepted that ULSD fuels will need additives to assure adequate lubricity. EL shows some promise as a lubricity additive, and biodiesel is very effective in this role.

The general atmosphere of uncertainty that will prevail during the transition to ultra-low-sulfur diesel fuels (ULSD) could work to the advantage of EL as a diesel fuel blending stock. Where EL is available, it could help relieve supply shortfalls and moderate local or regional DF prices. If EL can be produced as an effectively zero-sulfur product, it could be blended to DF delivered with above-spec sulfur content to bring it back into specification. This would permit the EL-blended fuel to be sold as highway diesel and prevent its being downgraded to the off-highway market. These factors could be expected to work to the advantage of EL and EL-blended highway diesel fuel. It cannot be predicted how long the transitional period's price and supply upsets will persist. Nevertheless, it seems likely that for one to three years beyond the June 2006 ULSD introduction date there will be discernible effects, at least in regional fuel markets, of this landmark event. EL may thus have additional time to build market share beyond mid-2006. Exhibit 18, shows that Biofine, by capturing 0.125 percent of the expected diesel fuel shortage in 2007 (320,000 bbl/day), could add additional annual market volume of approximately 6 million gallons of EL annually.

Exhibit 18: EL Potential In Transportation Fuel Market

TRANSPORTATION MARKET ANALYSIS

General Assumptions

Diesel Heat Content	131,067 Btu/gal
EL Heat Content	95,425 Btu/gal
Price Conversion Factor	0.73

Notes

1) Assumes Diesel users will not be willing to pay any premium for EL on bulk market.
EL Prices set at parity with diesel prices on a $/MMBtu basis
2) Diesel shortage case assumes that existing market through 2005 maintained.
Also assumes that PBLP captures 0.125% of 320,000 bbl/day shortage

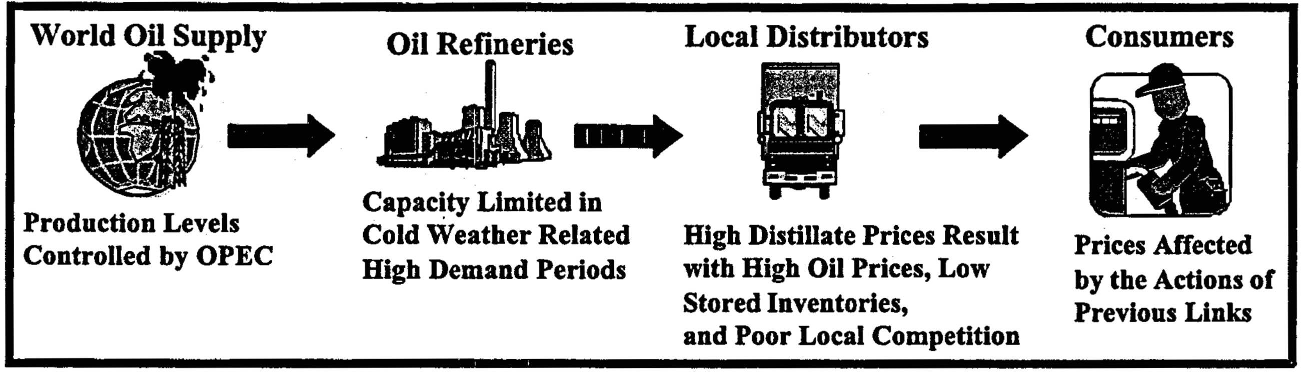
	Diesel Prices ($/gal)		EL Prices ($/gal)		Total Market (Bgal)		Market Penetration		EL Demand (Kgal)	
Scenario Name	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010
Business as Usual	$ 0.67	$ 0.68	$ 0.49	$ 0.50	32	32	0.006%	0.006%	1,900	1,900
Business as Usual + Diesel Shortage	$ 0.67	$ 0.87	$ 0.49	$ 0.63	32	32	0.006%	0.025%	1,900	8,032

The advent of ULSD fuels will reduce the technical advantages conferred by EL (and biodiesel). In the 2007 model year, the first highway diesel engines will appear that have been certified to EPA's Tier 2 emissions standards. The combination of ULSD and advanced emissions controls will reduce the incremental emissions improvements obtainable from either EL or biodiesel. Biodiesel may suffer somewhat less than EL in this environment, since biodiesel is a high-cetane blending stock and an oxygenate, both of which improve emissions. EL, also an oxygenate, reduces the cetane number of diesel fuel and thus offers smaller emissions benefits and no engine performance improvement. A second consideration is that the per-gallon heating value of diesel fuel will decline as a result of deep desulfurization. Blending stocks like EL and biodiesel, both lower-energy fuels than diesel fuel, will have greater effects on the heating value of ULSD blends than on blends with today's diesel fuel. Since vehicle fuel economy (miles per gallon) is directly related to fuel energy content, this may make them less attractive to vehicle operators.

Home Heating Oil Market

Exhibit 19, seen below, briefly summarizes the role of each economic contributor within the home heating oil industry

Exhibit 19: Economic Links of The Home Heating Oil Market



Many unpredictable outside influences combine to determine the price of crude oil and all oil derivatives. In the case of the home heating oil market, weather is the ultimate driver of demand. In a colder-than-average winter with other influences held constant, demand will be higher than normal - this will stimulate a price spike. The severity and duration of the cold snap, coupled with internal industry factors (e.g., low inventories), will determine the magnitude of the price spike. The oil industry begins with the world oil producers, then moves down to the refiners, the local distributors, and finally, the consumers. The final link in the chain, the consumer, is economically affected by how the previous three links prepare and deal with demand fluctuations. The following discussion provides an overview of the vital links that are key to understanding heating oil economics.

The world oil producers are the first and most important link in the overall oil industry picture because they set the stage for all oil product supply. Here the Organization of Petroleum Exporting Countries (OPEC) exerts sufficient market control to have an impact on oil prices. Generally, OPEC is guided by the basic rules of supply and demand to keep prices within an acceptable range. However, severe or mild weather conditions or changes in the global economy can alter the demand and cause price swings. OPEC attempts to accurately adjust production level up or down to compensate for any demand alterations incurred by the global environment. OPEC may respond to supply crisis for better or worse depending on the politics, economics, and the climate. For example, severe weather is difficult to anticipate and prices can go up or down dramatically. This was the case in 1998, a year marked by much milder than normal winter weather in the U.S. This, coupled with economic recession in Asia, led to a sharp decline in global oil demand. OPEC didn't anticipate either of these crises. The result was an oversupply and a sharp decline in crude prices.

Whenever the price of oil drops below the range of $22-$28 per barrel, one can anticipate OPEC will reduce production to raise prices. In March 1999, representatives of the major oil-producing nations met to map out a plan that would create higher prices. OPEC voted to cut production deeply for at least a year. OPEC initially cut crude oil production by 7.5%, or more that 2 million barrels a day. They further cut output by more than 4.3 million barrels per day. These cuts resulted in spiking the price of a barrel of crude oil from $10, in March 1999, to $28 in January 2000. Generally, these spikes continue until demand is abated. At this time, OPEC will continue normal levels of production, which will eventually level out prices.

Domestic refining capacity also plays a major role in the equation. The U.S. refining industry cannot be profitably operated to meet peaking seasonal demands for distillate and gasoline. These refineries are usually already running well over 90% capacity and if an extended period of cold weather arrives, the refining industry is unprepared to compensate for the sudden increase in demand. Even if favorably priced crude oil is abundant, their ability to increase output is limited. In addition, refineries are presently large, but few in number. This characteristic is more financially efficient for the refineries, but can leave consumers at the mercy of the supply crises, especially during severe cold weather episodes when transportation of heating oil can be delayed. Delays lead to buyers' bidding prices up further to attract product. In the future, the U.S. refining industry may be even less prepared to handle sudden increases in demand. Refining capacity is expected to remain unchanged, with all refining expansions being made outside the U.S. This will only increase prices to the consumer during periods of high demand due to the inevitable increase in shipping and distribution fees.

The increase in crude oil prices to the refiner are passed down to the local distributers. Storage shortages, especially during recent winters, have become a common problem. Storage is an expensive element of the oil industry. Due to recent mild winters, suppliers have purposely kept their inventories low to avoid extra regulatory and administrative costs. When a severe winter arrives, they are unprepared for the sudden jump in demand. During this time, the available heating oil is used faster than it can be replenished. Refineries normally cannot keep up with demand during cold periods, sending inventories down. Wholesale buyers become concerned that supplies are not adequate to cover short-term customer demand and bid up prices of available product. In the Northeast, for example, additional supplies usually must come from some distance away such as the Gulf Coast or Europe. It costs more to transport heating oil from these sources, and it also can take two or three weeks to arrive. During the time resupply is occurring, the supply of heating oil in the region drops even further. Buyer anxiety about finding product in the short term rises, and so do prices - sometimes sharply - until new supplies arrive.

Competitive differences between local distributors contribute to heating oil pricing as well. Consumers in remote or rural locations may face higher prices because there are fewer distribution competitors. Regional operating costs will also affect prices. These higher prices can be due to higher costs of transporting the product to remote locations.

In addition, the dealers' cost of doing business can vary substantially depending on the area of the country in which the dealer is located. Costs of doing business include wages and salaries, benefits, equipment, lease/rent, insurance, overhead and state and local fees.

According to studies conducted through the DOE, low inventories are likely to occur in approximately 1 of every 3 years[29]. So, in theory, a price spike of some degree will occur approximately once in every three years. The study used the history of heating degree days combined with the probabilities of low inventories and created a statistical model. During these price spikes, a moderately more expensive alternative fuel, such as EL, will have a greater opportunity to penetrate the home heating oil market. EL production that can be brought into a local heating oil supply pool on short notice during price spikes would offset any regional or local heating oil supply shortcomings with EL.

The previously mentioned crisis-forming-factors can be transformed into a lucrative opportunity for an EL manufacturer. Strategic location in the Northeast offers prompt, relatively inexpensive access to over three-quarters of the U.S. heating oil market. The northeastern United States is an ideal region for EL heating oil market penetration. Of the 7.7 million households in the U.S. that use heating oil, 5.3 million households, or roughly 69% reside in this region. In 1999, 4.9 billion gallons of heating oil were sold to residential consumers in the Northeast; this is 78% of total U.S. residential fuel oil sales.

The implementation of EL blends with heating oil may significantly reduce the pressure from local suppliers. Suppliers face stiff penalties in the competitive market for failing to correctly estimate the level of inventories that they will need. If inventories are too high, suppliers pay unnecessary storage costs, reducing profits. If inventories are too low, they lose customers to their competitors. Suppliers stock the expected winter demand to maximize profits. However, if the Northeast is struck with a severe winter weather condition, stocks are depleted rapidly. The unexpected addition in demand can be matched by EL/FO blends. This is the best alternative for the supplier. The decision allows the supplier to keep stocks at an appropriate level, which will allow the supplier to maintain credibility with customers. The supplier can maximize the situation even further by marketing the EL/FO blend as a "green product."

Working against the use of EL to level price spikes is the political desire to mitigate future price spikes. In July 2000, President Clinton issued an Executive Order that created a Northeast home heating oil reserve of 2 million barrels (84 million gallons). It is unclear how effective this reserve will be, but it will certainly buffer prices somewhat. In addition, while EL could make an excellent, renewable substitute for heating oil, there is the practical matter of the market's seasonality to consider. Home heating oil is primarily sold during the heating season. Producing EL year-round to serve this market would require large-scale storage facilities. Biofine will probably find this is unwise for the same reasons the existing industry keeps its inventories low. This suggests that, like a traditional refiner, Biofine may find its best

---

[29] U.S. Department of Energy, ***Report to Congress On The Feasibility of Establishing a Heating Oil Component to The Strategic Petroleum Reserve***, 1996.

interest lies in adopting a strategy of providing product to this market as it becomes economic to do so. At least initially, Biofine could arrange to provide EL to distributors at specific price points using heating oil market indicators as triggers. During these periods, EL production for steadier markets (transportation, solvent and green electricity) would be reduced to fulfill these contracts. A considerable amount of attention would necessarily be paid to interlinking all the EL markets in a way that maximizes profitability.

By this reasoning, ANTARES believes that the heating oil market does not itself provide an initial opportunity to expand overall EL production. Instead, this market may provide a premium on some portion of the EL being produced for other markets. The combination of a green premium and price spikes associated with heating oil shortages will allow EL to demand a premium above prices expected in the transportation and electricity sectors. Based on this presumption, Exhibit 20 provides an estimate of the additional annual revenue the heating oil market may offer for EL products.

Exhibit 20: EL Potential In Home Heating Oil Market

HOME HEATING OIL MARKET ANALYSIS - PEAK SEASON POTENTIAL

Notes

1) Price spike premiums based on 1999 vs 2000 heating oil prices
2) Green premium required to bring $0.90/gal EL into parity with heating oil @ $1.00/gal. Premium could be higher based on marketing acceptance.
3) Market penetration keeps demand at EL plants within production parameters of existing capacity forecast above. Actual ability for PBLP to meet market demand spikes will be dependent on supply contracts for non-seasonal EL capacity.

	Green Pricing Premium		Price Volatility Premium		Total Market (Bgal)		Market Penetration		Redirected EL (Kgal)	
Scenario Name	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010
Seasonal Price Spike (incremental analysis)	$ 0.04	$ 0.04	$ 0.30	$ 0.30	5	5	0.100%	0.200%	5,000	10,000

4.2.4 *Levulinic Acid*

The existing market for LA is small (under 1 million pounds per year) and supplied by a European manufacturer who requires long lead times. We believe that 25% of the customers would qualify and support a USA producer at a selling price of $2.00 per pound versus $4 to $5 per pound. The other 75% make such high-value-added derivatives from the LA that they would likely be indifferent to the savings. In addition to the existing market, CIS, Inc. experience suggests that approximately 10 million pounds at up to $0.50/lb may be sold to companies who would develop their own higher value-added uses for LA. Ten million pounds for new uses is thought to be a conservative number based on reports from researchers on the versatility of low-cost LA as a new feedstock.

4.3 Market Penetration Expectations (Longer-Term Product Deployment)

4.3.1 Tetrahydrofuran (THF)

THF has two markets. About 75% of the THF produced goes into the production of polytetramethylene ether glycol (PTMEG). The remainder is primarily sold into the solvents market. THF available at high volumes and low cost would be very attractive to PTMEG producers. These companies normally have to expand both THF and PTMEG capacity to meet new market demands, but high-volume THF from Biofine would eliminate significant capital cost.

A new THF plant is normally 20-25 million pounds. This capacity is just under 10% of the domestic market, which suggests that a large-scale Biofine plant might be designed to offset the need for at least one future plant. Current production costs suggest a PTMEG producer might consider purchasing THF from Biofine at $0.60/lb, a price that should be well below their production costs. Likewise, a penetration of 13% of the solvent market should be achievable because at $1.00 per pound it would allow a reliable supplier and a price considerably below the $1.50 solvent price - which is always tight.

4.3.2 Delta Amino Levulinic Acid (DALA)

Agricultural Markets

The agricultural marketplace is always alert for more efficient and environmentally friendly pesticides. The marketing plan is for Biofine to establish strategic partnership(s) with major agricultural chemical companies to develop herbicide and pesticide markets.

Our market forecast is based on the value of the U.S. pesticide market being $600 million at the producer level; 23% is herbicide; 73% is insecticide.

The Department of Agriculture has published statistics on the treated acreage and the pounds of herbicide used by fifteen Midwestern and mid-central states on corn and soybean crops in 1999. In corn, 68 million acres used 154 million pounds of herbicide. Although Atrazine was the most popular material, the market was very fragmented, with over forty chemical compounds used by growers.

In soybeans, total acreage was also 68 million, with 71 million pounds of herbicide used in that application. Glyphosate had the highest volume, but over thirty different chemical compounds were used.

Market penetration for DALA could be achieved by marketing the product through existing agricultural companies who would like to be on the cutting edge of pesticide control technologies. Our market estimate assumes a 10% penetration into corn and soybean crops with 1.5 pounds of DALA used per acre at a price of $2.00 per pound. One hundred thirty-nine million acres multiplied by 10% equals 14 million acres times 1.5 pound per acre times $2.00 per pound equals $42 million.

Human Medicinal Markets

Because of insufficient data and the immaturity of the research, ANTARES has not forecast a pharmaceutical market for DALA. However, as noted earlier, the potential for this substance to safely treat certain types of cancer could be substantial. This high-value and growing market could become an increasingly important part of Biofine's long-term marketing strategy.

4.4 Market Penetration Summary

Exhibit 21 provides a summary of the market discussion presented above. Since the timing of market penetration is difficult to predict, potential annual market volumes are presented for two distinct time frames: 1) 2000-2005; and 2) 2006-2010. For lack of a better model, linear extrapolation could be used to estimate year-by-year penetration. However, actual penetration will depend on specific market volumes needed by customers and practical considerations such as minimum downstream chemical plant capacity requirements.

EXHIBIT 21: Market Penetration Summary

BIOFINE MARKET ESTIMATES

Notes

1) Table assumes RPS & 1% non-renewable market penetration for electricity sales using EL

2) Diesel shortage case used in evaluating EL application in transportation sector

	PBLP Vol (Mlbs)		Market Price ($/lb)		Sales Revenue ($M)	
Product Name	In 2005	In 2010	In 2005	In 2010	In 2005	In 2010
Levulinic Acid	0	10	$ 2.00	$ 0.54	$ 0.5	$ 5.5
Formic Acid	30	30	$ 0.22	$ 0.22	$ 6.5	$ 6.5
Diphenolic Acid	300	320	$ 0.40	$ 0.41	$ 120.0	$ 132.0
Ethyl Levulinate	159	910	$ 0.14	$ 0.12	$ 22.8	$ 111.7
Tetrahydrafuran	-	40	$ -	$ 0.70	$ -	$ 28.0
DALA	-	23	$ -	$ 2.04	$ -	$ 46.0
Total	*489*	*1,333*			*$ 149.8*	*$ 329.7*

The results suggest that by 2010, Biofine biorefineries may be producing a mix of products worth over $300M in annual revenue. It is also apparent that reaching this goal will require focusing on the development of chemical markets. Approximately 2/3 of the market potential in 2010 is derived from sales in this sector. While the energy sector may provide a launch pad for the first new plant, it is the chemical sector that will add value to Biofine projects and spark large scale deployment.

5.0 CONCLUSIONS

The markets for products derived from LA are very diverse, covering major sectors of the global economy. While this is most certainly a strength for the technology and Biofine, it does challenge Biofine to focus its early efforts to move toward deploying a larger, second plant.

As a result of this effort, ANTARES has determined the following:

- Several chemicals appear to have market volume and price points that make exploring these markets and strategic partnerships with industry leaders (including international trading companies) a high priority.
- Profitable entry into the energy markets for LA derivatives will require substantial green market support and in some cases additional legislative action. Biofine should begin immediately to contact green energy marketing groups and discuss the potential for including heat/power generated using Biofine products in their offerings. However, these efforts should be subordinated to an increased effort to target the chemical industry as a customer of LA derivatives.
- Biofine should query the IRS on the requirements to obtain a private ruling for receiving the ethanol tax credit on a prorata basis for the manufacture of ethyl levulinate
- Biofine must place a high priority on additional fuel testing to meet government regulations for key markets and to determine ways to resolve key technical issues.
- Biofine must begin the process of identifying regulatory requirements for marketing a new pesticide/herbicide as soon as possible. Lead times and testing may already push deployment of such a product into late this decade.
- Cost curves detailing the relationship of biorefinery size to production costs of LA and derivative products must be developed to determine optimal plant sizing for given markets.
- A detailed heat and energy balance for a combined cycle plant operating on EL/FO blends should be performed using data collected from additional fuel testing.
- Biofine's deployment plans for a 500 tpd facility must identify energy markets that will value the "green" renewable nature of EL.
- The Northeast may offer price and market volume benefits that make this region especially attractive. The region is coincident with mandated renewable energy capacity, a substantial heating oil market that is forecasted to be in cyclical crisis, and a substantial supply of potential cellulosic resources.
- Texas and California may offer interesting siting opportunities for a biorefinery.
- By 2010, revenue from LA derivative products could reach over $300M/year

REFERENCES

Advanced Petroleum-Based Fuels-Diesel Emissions Control Program, *New Government/Industry Program Focuses on Optimizing Systems to Reduce Diesel Emissions*, September, 2000.

American Chemical Society, *Seeing Green*, April 2000.

Armesto, Charles and Gerald Ondrey, *A Renewed Boost for Ethanol*, Chemical Engineering, February 1999.

BASF Corporation, Product Information - Formic Acid, 1999-2000.

Biodiesel and Ongoing Research, Fact Sheet published by the National Renewable Energy Laboratory, May 2000.

Britannica.com Inc., *Plastic (Thermoplastic and Thermosetting Resins)*, 2000.

Canadian Corporate News, *Canadian Gas, Canfor Enter into MOU*, October, 2000.

Center for Resource Solutions, *Green-e Verification Results 1999*, September 2000.

Chem Expo, Chemical Market Reporter, 1996-1999.

Chemical Market Resources, Inc., *Thermoplastic Polyolefin Elastomers, Markets, Technologies & Trends, 1999-2004*, 2000.

Deutsch, David, *Fueling Up On Ethanol*, Chemical Engineering, October 2000.

E-Chemicals, Industry News, December 1999.

Energy Information Administration, *National Gas Monthly*, September 2000.

Energy Information Administration, *Fuel, Oil, Kerosene Sales 1999*, 1999.

Energy Information Administration, *Petroleum Marketing Monthly*, October 2000.

Energy Information Administration, *Petroleum Supply Annual*, vol.1, 1999.

Energy Information Administration, *Weekly Petroleum Status Reports*, 2000.

Executive Steering Group, *The Technology Roadmap for Plant/Crop-Based Renewable Resources 2020*, February 1999.

Federal Energy Regulatory Commission, *Monthly Report on Cost and Quality of Fuels for Electric Plants*, June 2000.
Gas Turbine World, *1999-2000 Handbook*, vol.20, 1999.

Graphic Media Publishings, Inc., Market Reports, September 2000.

International Energy Agency, *World Energy Outlook*, 1996 Edition, 1996.

Lambrides, Mark, *The Renewable Energy Policy Manual*, United States Export Council for Renewable Energy.

Langfang Triple Well Chemicals Co., LTD, Levulinic Acid Chemical Properties.

Mueller Associates, Inc., *Status of Alcohol Fuels Utilization Technology for Stationary Gas Turbines*, prepared for U.S. Department of Energy, HCP/M2098-01/UC-13, April, 1979.

National Safety Council, *Formic Acid Chemical Backgrounder*, 2000.

Oil & Gas Journal *Petroleum Refining Catalyst Demand, 1989-2003*, October 2000.

Pencor/Biofine, L.P., *Creating Tomorrow's New Chemicals, Solving Today's Waste Disposal Problems*, July 2000.

Polysort, Market Research Reports, 1999.

Porter, Kevin and Wiser, Ryan (National Renewable Energy Laboratory and Ernest Orlando Lawrence Berkeley National Laboratory), *Biomass Power and State Renewable Energy Policies Under Electric Industry Restructuring*, 2000.

Reilly, Christopher, *Crude Oil Pushes Prices UP, But for How Long?*, Cahners Business Information, 2000.

Reilly, Christopher, *Distributors Increase Service Focus*, Cahners Business Information, 2000.

Reilly, Christopher, *Feedstocks Confuse Price Outlook*, Cahners Business Information, 2000.

Reilly, Christopher, *Prices Will Stabilize, Then Rise in '99*, Cahners Business Information, 2000.
Reilly, Christopher, *The Very Best Suppliers Go the Extra Mile*, 2000.

Reilly, Christopher, *Strong Demand Will Raise Prices*, Cahners Business Information, 2000.

SCDC Research, *Canola New Products and Uses Research List*, 2000.

SRI Consulting, *Chemical Economics Handbook*, 1,4-Butanediol Product Review, 1997.

SRI Consulting, *Chemical Economics Handbook*, Formic Acid-United States Data Summary, 1993.

SRI Consulting, *Chemical Economics Handbook*, Public Reports, 2000.

The Columbia Encyclopedia, *Formic Acid*, Sixth Edition, 2000.

The Good Scents Company, Ethyl Levulinate Chemical Properties, October 2000.

The Oxygenated Fuels Association, *Representatives Oppose MTBE Legislation*, October 2000.

U.S. Department of Energy, *Bioenergy: Growing an Integrated Industry*, November, 1998.

U.S. Department of Energy, *Plant/Crop-Based Renewable Resources 2020*, January 1998.
Washington Week, *MTBE Phaseout*, October, 2000.

U.S. Department of Energy, *Report to Congress On The Feasibility of Establishing a Heating Oil Component to The Strategic Petroleum Reserve*, 1996.

World Fuels Today, *Gasoline, Crude Sell-Offs Freezes Oxygenate Buying*, October, 2000.

APPENDIX A

Material Safety Data Sheets
(1,4-Butanediol and 2-Methyltetrahydrofuran)

1,4-Butanediol

Technical Data Sheet

January 1997

Formula: . . . . $C_4H_{10}O_2$
Molecular Weight: **90.1**
Product Number: **598061**
CAS Registry Number: . . . **110-63-4**

$HO(CH_2)_4OH$

Product Specifications	Value	Test Method
Assay, % minimum	99.5	STI 8403
Water, wt. % maximum	0.05	STI 8413
Color, APHA maximum	10	STI 8415

Physical Properties	
Solidification point, °C	20
Boiling point, °C	230
Density @ 20°C, g/ml	1.017
Flash point, °C	134
Ignition temperature, °C	370

Description

1,4-Butanediol is a colorless and almost odorless liquid. This hygroscopic diol is soluble in water, alcohols, esters, ketones, glycol ethers and glycol ether acetates, immiscible in aliphatic and aromatic hydrocarbons and diethylether.

BD is the ethynylation product of acetylene and formaldehyde, a bifunctional, primary alcohol.

Safety

1,4-Butanediol is not a skin irritant, but may be irritating to mucous membranes or the respiratory tract.

Vapors may irritate the eyes. If severe, iritis, conjunctivitis or diffuse corneal opacity may result. Proper respiratory and protective eyewear are recommended.

Always refer to the Material Safety Data Sheet (MSDS) for detailed information on safety.

Applications

Intermediate used in the production of:

- polyether diols (Poly THF®)
- polyether diols (adipate, polycaprolactone)
- urethane polymers
  - TPU's
  - cast elastomer (noncellular)
  - microcellular elastomer (foam)
- polyester polymers
  - PBT
  - copolyester elastomers

It is also used as:

- plasticizer (e.g., in paper and cellulosics)
- carrier solvent in printing inks
- cleaning agent

For other applications, please refer to the Technical Information Bulletin.

Packaging

Available in 450 lb (net weight) non-returnable steel drums.

Also available in bulk quantities.

Storage & Handling

1,4-Butanediol has an almost unlimited shelf life in unopened, original containers if protected from heat. It is neither explosive nor spontaneously flammable in air. However, it is combustible.

Always refer to the Material Safety Data Sheet (MSDS) for detailed information on handling and disposal.

IMPORTANT: While the descriptions, designs, data and information contained herein are presented in good faith and believed to be accurate, it is provided for your guidance only. Because many factors may affect processing or application/use, we recommend that you make tests to determine the suitability of a product for your particular purpose prior to use. NO WARRANTIES OF ANY KIND, EITHER EXPRESS OR IMPLIED, INCLUDING WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, ARE MADE REGARDING PRODUCTS DESCRIBED OR DESIGNS, DATA OR INFORMATION SET FORTH, OR THAT THE PRODUCTS, DESIGNS, DATA OR INFORMATION MAY BE USED WITHOUT INFRINGING THE INTELLECTUAL PROPERTY RIGHTS OF OTHERS. IN NO CASE SHALL THE DESCRIPTIONS, INFORMATION, DATA OR DESIGNS PROVIDED BE CONSIDERED A PART OF OUR TERMS AND CONDITIONS OF SALE. Further, you expressly understand and agree that the descriptions, designs, data and information furnished by BASF hereunder are given gratis and BASF assumes no obligation or liability for the description, designs, data and information given or results obtained, all such being given and accepted at your risk.

Customer Service

EAST	1-800-426-8696
WEST	1-800-543-1740

BASF Corporation
Chemicals Division
3000 Continental Drive - North
Mount Olive, New Jersey 07828-1234

World Wide Web: http://www.basf.com

BASF

Chemical Intermediates

Material Safety Data Sheet

Page : 1
Original Date: 05/27/1992
Revision Date: 01/29/2000

BASF CORPORATION
3000 CONTINENTAL DRIVE NORTH

MOUNT OLIVE, NJ 07828
(973) 426-4671

EMERGENCY TELEPHONE: (800) 424-9300 CHEMTREC
(800) 832-HELP (BASF Hotline)
BOTH NUMBERS ARE AVAILABLE DAYS, NIGHTS, WEEKENDS, & HOLIDAYS.

SECTION 1 - PRODUCT INFORMATION

1,4 BUTANEDIOL
Product ID: NCI 598061
Common Chemical Name:
1,4-Butanediol
Synonyms:
1,4-Butylene Glycol
Molecular Formula:
HOCH(2)CH(2)CH(2)CH(2)OH
Chemical Family: Glycol
Molecular Wt.: 90.1

SECTION 2 - INGREDIENTS

Chemical Name:				CAS		Amount
Butanediol				110-63-4		100.0 %
MAK	MAK EXP. LIMIT	TWA	50	PPM		

SECTION 3 - PHYSICAL PROPERTIES

Color: Colorless
Form/Appearance: Oily Liquid
Odor: Nearly odorless
Odor Intensity: None

	Typical	Low/High	U.O.M.
Specific Gravity:	1.015		
pH:		7 - 8	SU

	Typical	Low/High	Deg.	@	Pressure
Boiling Pt:	229		C	1	ATMOSPHERES
Freezing Pt:	< 19		C	1	ATMOSPHERES

Decomp. Tmp: NOT AVAILABLE
Solubility in Water Description: Miscible
Vapor Pressure: < 1 MILLIBARS X 20 DEG. C XX
Vapor Density (Air = 1): 3.1

SECTION 4 - FIRE AND EXPLOSION DATA

	Typical	Low/High	Deg.	Method
Flash Point:	134		C	DIN 51 758

SECTION 4 - FIRE AND EXPLOSION DATA (cont)

	Typical	Low/High	Deg.	Method
Autoignition:	370		C	NONE SPECIFIED

Extinguishing Media:

Use water fog, foam or dry chemical extinguishing media.

Fire Fighting Procedures:

Firefighters should be equipped with self-contained breathing apparatus and turn out gear.

Unusual Hazards:

Explosion hazard is low when exposed to heat or flames. Can react with oxidizing materials.

SECTION 5 - HEALTH EFFECTS

Routes of entry for solids and liquids include eye and skin contact, ingestion and inhalation. Routes of entry for gases include inhalation and eye contact. Skin contact may be a route of entry for liquified gases.

Toxicology Test Data:

Rat, Oral LD50 - 1500 MG/KG
Moderately Toxic
Rat, Dermal LD50 - > 2000 MG/KG
Slightly Toxic
Rabbit, Primary Skin Irritation -
Nonirritating
Rabbit, Eye Irritation (unrinsed) -
Nonirritating
Rat, Inhalation Safety Screen, 4 hr - > 5.1 MG/L
Moderately Toxic
Sensitizing potential -
Not a sensitizer
Ames Salmonella Assay -
Negative (no mutagenic response)

Acute Overexposure Effects:

Contact with the eyes and skin may result in slight irritation. If ingested, 1,4-butanediol is a depressant. Symptoms of acute overexposure include deep narcosis, constriction of pupils, and loss of reflexes. Damage to the kidneys has also been reported after ingestion.

Chronic Overexposure Effects:

There are no known chronic effects associated with this material.

First Aid Procedures - Skin:

Wash affected areas with soap and water. Remove and launder contaminated clothing before reuse. If irritation develops, get medical attention.

First Aid Procedures - Eyes:

Immediately rinse eyes with running water for 15 minutes. If irritation develops, get medical attention.

First Aid Procedures - Ingestion:

If swallowed, dilute with water and immediately induce vomiting. Never give fluids or induce vomiting if the victim is unconscious or having convulsions. Get immediate medical attention.

SECTION 5 - HEALTH EFFECTS (cont)

First Aid Procedures - Inhalation:
Move to fresh air. Aid in breathing, if necessary, and get immediate medical attention.

First Aid Procedures - Notes to Physicians:
None known.

First Aid Procedures - Aggravated Medical Conditions:
No data is available which addresses medical conditions that are generally recognized as being aggravated by exposure to this product. Please refer to the effects of overexposure section for effects observed in animals.

First Aid Procedures - Special Precautions:
None

SECTION 6 - REACTIVITY DATA

Stability Data:
Stable

Incompatability:
Strong oxidizing agents.

Conditions/Hazards to Avoid:
Avoid exposure to temperatures >150 C.

Hazardous Decomposition/Polymerization:
HAZARDOUS DECOMPOSITION PRODUCTS: Unstable when heated, can form flammable THF at >150 C. Other decomposition products include CO,COX. Polymerization: Does not occur.

Corrosive Properties:
Not corrosive.

Oxidizer Properties:
Not an oxidizer

SECTION 7 - PERSONAL PROTECTION

Clothing:
Gloves, coveralls, apron, boots as necessary to minimize contact.

Eyes:
Chemical goggles; also wear a face shield if splashing hazard exists.

Respiration:
If vapors or mists are generated, wear a NIOSH/MSHA approved organic vapor/mist respirator or an air-supplied respirator as appropriate.

Ventilation:
Use local exhaust to control vapors/mists.

Explosion Proofing:
See Section 4 - Fire and Explosion Data.

Other Personal Protection Data:
Eyewash fountains and safety showers must be easily accessible. Shower after handling.

SECTION 8 - SPILL-LEAK/ENVIRONMENTAL

General:
Spills should be contained, solidified and placed in suitable containers for disposal in a licensed facility. This material is not regulated by RCRA or CERCLA ("Superfund"). Wear appropriate respiratory protection and protective clothing and provide adequate

ventilation during clean-up.

Waste Disposal:
Incinerate or bury in a licensed facility. Do not discharge into waterways or sewer systems without proper authority.

Container Disposal:
Dispose of in a licensed facility. Recommend crushing or other means to prevent unauthorized reuse.

Other Spill/Leak Procedures:
When product is properly discharged in low concentrations to biological waste water treatment plants it should not cause any disturbances in the degradation activity of the activated sludge.

Environmental Toxicity Test Data:
Golden Orfe, static 96 hr LC50 - > 10 MG/L
Insignificant Hazard
Elimination (OECD 301A) - > 90 PERCENT
Readily Biodegradable
Daphnia magna, 48 hr static EC50 - > 500 MG/L
Practically Nontoxic
Acute Aquatic Toxicity, 96 hr LC50 - > 500 MG/L
Practically Nontoxic
Bacterial Toxicity, EC10 (17 hr) - > 10,000 MG/L
TEST RATING NOT FOUND

SECTION 9 - STORAGE AND HANDLING

General:
Keep containers closed.

Other Storage and Handling Data:
Consult other sections of this MSDS for information on reactivity and flammability.

SECTION 10 - REGULATORY INFORMATION

TSCA Inventory Status
Listed on Inventory: YES

RCRA Haz. Waste No .:
CERCLA: NO Reportable Qty.: (If YES)
State Regulatory Information: (By Component) NJ/PA/MA RTK
CAS: 110-63-4 NO
NAME: Butanediol

Hazard Ratings:

	Health:	Fire:	Reactivity:	Special:
HMIS	1	1	0	NA
NFPA	1	1	0	NA

This product is hazardous or contains components which are hazardous according to the OSHA Hazard Communication Standard.

SECTION 11 - TRANSPORTATION INFORMATION

DOT Proper Shipping Name:
N/A

SECTION 11 - TRANSPORTATION INFORMATION (cont)

DOT Technical Name:
N/A
DOT Primary Hazard Class:
N/A
DOT Secondary Hazard Class:
N/A
DOT Label Required:
N/A
DOT Placard Required:
N/A
DOT Poison Constituent:
N/A
BASF Commodity Codes: NA NA UN/NA Code: NONE E/R Guide:
Bill of Lading Description:
NOT REGULATED BY THE DEPARTMENT OF TRANSPORTATION
CLASS: P. G. SHIPPING NAME:
IATA: NONE

IMO: NONE

TDG: NONE

"IMPORTANT: WHILE THE DESCRIPTIONS, DESIGNS, DATA AND INFORMATION CONTAINED HEREIN ARE PRESENTED IN GOOD FAITH AND BELIEVED TO BE ACCURATE, IT IS PROVIDED FOR YOUR GUIDANCE ONLY. BECAUSE MANY FACTORS MAY AFFECT PROCESSING OR APPLICATION/USE, WE RECOMMEND THAT YOU MAKE TESTS TO DETERMINE THE SUITABILITY OF A PRODUCT FOR YOUR PARTICULAR PURPOSE PRIOR TO USE. NO WARRANTIES OF ANY KIND, EITHER EXPRESSED OR IMPLIED, INCLUDING WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, ARE MADE REGARDING PRODUCTS DESCRIBED OR DESIGNS, DATA OR INFORMATION SET FORTH, OR THAT THE PRODUCTS, DESIGNS, DATA OR INFORMATION MAY BE USED WITHOUT INFRINGING THE INTELLECTUAL PROPERTY RIGHTS OF OTHERS. IN NO CASE SHALL THE DESCRIPTIONS, INFORMATION, DATA OR DESIGNS PROVIDED BE CONSIDERED A PART OF OUR TERMS AND CONDITIONS OF SALE. FURTHER, YOU EXPRESSLY UNDERSTAND AND AGREE THAT THE DESCRIPTIONS, DESIGNS, DATA, AND INFORMATION FURNISHED BY BASF HEREUNDER ARE GIVEN GRATIS AND BASF ASSUMES NO OBLIGATION OR LIABILITY FOR THE DESCRIPTION, DESIGNS, DATA AND INFORMATION GIVEN OR RESULTS OBTAINED, ALL SUCH BEING GIVEN AND ACCEPTED AT YOUR RISK".

END OF DATA SHEET

PENN
Specialty Chemicals, Inc.

P.O. Box 80035 • 3324 Chelsea Avenue
Memphis, TN 38108
Phone: 901-320-4000• Fax: 901-320-4005

SALES SPECIFICATION

QO® 2-Methyltetrahydrofuran

Specification Items	Sales Specification	Test Method
Methyltetrahydrofuran, wt% min	99.0	TA-02-1
Moisture, ppm max	300	TA-02-2
Antioxidant, BHT, ppm	150-400	TA-02-3

Appearance: Clear water white to pale yellow liquid.

Specification Number: SC-02
Record Number: 000
Effective Date: 5/31/1996

QO CHEMICALS®

SALES SPECIFICATION

Page No.: 1 of 1

PRODUCT: QO® 2-Methyltetrahydrofuran

CUSTOMER: ALL

SPECIFICATION NO.: SC-02 ***REVISION NUMBER:*** 3

EFFECTIVE DATE: January 1, 1993 ***SUPERSEDES:*** All Previous

REASON FOR CHANGE: To change format

NOTES: Certificates of Analysis (COA) will record all analyses marked "C".

QUANTITIES:

CAS No.: 96-47-9

EINECS No.: 2025074

Formula: $C_5H_{10}O$

Product Specifications

Specification Items	Sales Spec	Customer Requirement	Test Method
2-Methyltetrahydrofuran wt%, min.	98.0	C	TA-02-1
Water wt%, max.	0.2	C	TA-02-2
BHT ppm w/w	150-400	C	TA-02-3

C = Item must be on COA.

Claims regarding the quality of QO® as determined by the above referenced methods must be submitted to: QO Chemicals, Inc., P.O. Box 2500, West Lafayette, Indiana 47906, within 15 days after receipt of shipment.

Material Safety Data Sheet

May be used to comply with OSHA's Hazard Communication Standard, 29CFR 1910.1200. Standard must be consulted for specific requirements.

METHF
Quick Identifier
QO®2-METHYLTETRAHYDROFURAN
Common Name (used on label and list)

Section I

Manufacturer's Name and Address
Penn Specialty Chemicals, Inc.
3324 Chelsea Avenue
Memphis, TN 38106

Emergency Telephone Number
ChemTrec: 800-424-9300
HAZARD INFORMATION SERVICES:800-228-5635
(For Emergency Medical Information)

For Product Information and Other Calls:
Penn Specialty Chemicals, Inc.:901-320-4000
Effective: November 2, 1999
Supersedes: September 6, 1996

Section II-Hazardous Ingredients/ Identity Information

Hazardous Component(s) Chemical & Common Name(s)	OSHA PEL	ACGIH TLV	CAS NO.
2-Methyltetrahydrofuran; Furan, 2-methyltetrahydro	Not established	Not established	96-47-9

Section III-Physical/Chemical Characteristics

Boiling Point	Vapor Density(Air=1)	Specific Gravity (H_2O = 1)	Vapor Pressure (mm Hg)
172-176°F, 78-80°C	2.9	0.857	102 @ 68°F, 20°C

Solubility in Water
Appreciable (15 wt % approx.)

Reactivity in Water
None

Appearance and Color
Clear, colorless to yellow, mobile liquid.
Mildly irritating odor

Melting Point/Freezing Point
-213°F, -136°C

Section IV-Fire and Explosion Hazard Data

Flash Point	Method Used	Flammable Limits in Air, % Volume	LEL Lower	UEL Upper
12°F, -11°C	Setaflash		1.5	8.9

Auto-ignition temperature
518°F, 270°C

Extinguisher Media
Dry chemical, carbon dioxide, foam.
Water may spread fire and be ineffective because of low flash point.

Special Fire Fighting Procedures
Evacuate contaminated area.
Firefighters should have eye protection and wear self-contained breathing apparatus.
Fight fire from protected location or maximum possible location.
Use water spray to cool containers exposed to fire.

Unusual Fire and Explosion Hazards
Vapors are heavier than air and may travel to a source of ignition and flash back. Methyltetrahydrofuran is a dangerous fire and explosion hazard when exposed to ignition sources. See section V below.

Section V-Physical Hazards/Reactivity Data

Stability		Conditions to Avoid
	Unstable	
	Stable X	Normally stable. Do not distill to dryness; may contain peroxides, which if heated may become explosive.

Incompatibility (Materials to Avoid)
Oxidizers.

Hazardous Decomposition Products
Combustion produces carbon dioxide and carbon monoxide.

Hazardous Polymerization		Conditions to Avoid
	May Occur	
	Will Not Occur X	

Section VI - Health Hazards

Chemical Listed as Carcinogen or Potential Carcinogen	National Toxicology	IARC Monographs	OSHA Regulated
	Yes	Yes	Yes
	No X	No X	No X

Signs and Symptoms

Exposure

Mild odor.

Acute Overexposure

Strong odor, headache, nausea, dizziness. disorientation, drowsiness. Irritation of eyes and skin.

Chronic Overexposure

No data available.

Emergency and First Aid Procedures for Overexposure - Obtain prompt medical attention.

Inhalation

Move to fresh air. If necessary, restore and support breathing.

Eyes

Immediately flush with water for 15 minutes while lifting eyelids and rolling eyes.
Get immediate medical attention.

Skin

Wash promptly with soap and water.

Ingestion

Get medical help. If conscious and medical help not readily available, give water.

Section VII - Special Precautions and Spill/Leak Procedures

Precautions to be taken in Handling and Storage

Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage warehouse. room or cabinet. Always ground containers and piping when transferring.

Other Precautions

Store under nitrogen to reduce fire hazard and for product quality.
Avoid skin contact. Keep containers tightly closed. No smoking or eating in handling area.

Steps to be taken in case Material is Released or Spilled

Evacuate unprotected personnel. Shut off sources of ignition. Pick up spill on sand, earth or other noncombustible, absorbent material. Place in covered container for disposal.
Flush area with water to remove last traces.

Waste Disposal Methods (Consult Federal, State, and Local Regulations)

Dispose of as an ignitable waste in accord with regulations.

Section VIII - Special Protection and Control Measures

Respiratory Protection *(Specify Type)*

NIOSH approved organic vapor air purifying respirator.
Explosion proof ventilation and local exhaust required.

Eye Protection	Protective Gloves	Other Protective Clothing or Equipment
Chemical goggles and face shield.	Butyl or Viton rubber.	Eye fountain and safety shower.

Work Hygienic Practices

Avoid direct contact with 2-Methyltetrahydrofuran. Wash thoroughly after handling.

Transport Information:

DOT: Methyltetrahydrofuran, 3, UN2536, PG II.
Label: Flammable liquid.

APPENDIX B

Chemical Industry Services, Inc.
(Levulinic Acid Derivatives Report)

Chemical Industry Services, Inc.
2707 Stemwell Boulevard, Richmond, VA 23236
Tel: (804) 745-4565
Fax: (804) 745-6877
e-mail: rcbilski@rcn.com

Levulinic Acid Derivatives

Levulinic Acid (LA) can be produced cost effectively at very low costs, in large volumes and in high yields from renewable feedstocks by a new industrial process. The technology is being demonstrated and validated on a one ton/day scale at a facility in Glens Falls, NY. The biorefinery concept is to use a base load volume to produce materials for fuel applications, which is the lowest-value use, and to use a significant percentage of the LA for the production of much higher-value-added chemicals. This strategy is not unlike the concept used by the petroleum industry to manufacture a wide range of products from crude petroleum, including fuels and petrochemicals.

LA has been frequently discussed by researchers as a desirable building block (platform chemical) for the production of other chemicals. However, its availability (one million pounds/year) and high cost ($4.00 - $6.00/lb) simply did not make its use viable for most applications. LA is a versatile chemical in that its C5 linear configuration, and activity at the acid and ketone sites allow a wide range of chemical reactions.

The criteria for selecting the targeted chemical derivatives were:

- Large existing or potential markets
- Commercially viable synthesis from LA
- Known or technically viable processes
- High yields
- Marketable secondary or by-products
- Low production cost for multi-million pound volumes
- Site compatibility with LA plant
- High-value-added derivatives
- Domestic and foreign markets
- Commercial entry through existing suppliers
- Ease of market entry
- Growth markets
- High profit margins

Some of the LA derivatives that were identified that met most of the selection criteria are listed below:

- LA (itself)
- Methyl THF
- THF
- 1, 4 BDO
- DPA
- DALA
- Succinic Acid
- Ethyl Levulinate
- Acetoacrylic acid

- Angelicalactone
- Valerolactone
- Formic Acid (LA co-product)

Phase 1 (Near-term) targeted derivatives

Levulinic Acid (LA)

The current market for LA is one million pounds annually with a selling price of $4.00 - $6.00 per pound. Although LA is frequently identified by researchers as a desirable building block, its price and availability have not allowed for any high- volume commercial products. With prices of under $0.25 per pound, which is lower than many of the current petrochemical building blocks, LA should stimulate a lot of technical and commercial interest.

Our market forecast has 25% of the current users purchasing LA from PBLP to qualify a domestic supply at $2 per pound.

Additionally, we forecast 10MM pounds at up to $0.50/lb for sales to companies who would develop their own higher-value-added uses for LA.

Formic Acid (FA)

The Biofine process produces one pound of FA for every 2.5 pounds of LA in an aqueous solution. This material can be recovered as FA or as a formic acid salt. There is a USA market for 60 million pounds of FA at approximately $0.45/lb. and we assume that current producers would purchase 30 million from PBLP at $0.22/lb to keep the new production within their control.

Excess FA coming out of LA plants could be concentrated and used at the plant as fuel.

Diphenolic Acid (DPA)

PBCP plans to promote DPA as a replacement for bisphenol A (BPA) in the production of polycarbonate resins and epoxy resins. DPA was used in this application but because of its cost and availability lost the market to BPA.

The current capacity for BPA in the United States is 2 billion pounds with four producers. Research funded at Rensselaer Polytechnic Institute (RPI) shows that DPA can be a cost effective substitute for DPA in polycarbonate and polyester resins. Also, the work shows that interesting properties can be obtained in the resins with low-level addition of DPA as a polymer modifier. This should allow for a premium price for DPA.

Our market forecast shows a 15% penetration (300 million pounds) as a BPA substitute at $0.40/lb which we believe is $0.20 below BPA cost. We also show a 20 million-pound-market as a polymer additive in resins and coatings at $0.60/lb.

Ethyl levulinate (EL)

PBLP will be producing hundreds of millions of pounds of EL for fuel applications at a cost well under $0.25 per pound. Several LA esters are currently produced by small specialty chemical companies and sold into high-value- added markets such as flavor and fragrance. Prices are generally over $10.00/pound.

The chemical industry is looking for new, low-cost products to replace conventional high VOC solvents that are flammable, toxic, irritating and environmentally undesirable.

EL may be an excellent candidate as a solvent in the food, electronic, low-VOC, degreasing, printing ink, etc applications.

A West Coast company has an ongoing project that involves a process for EL synthesis from LA and EL application studies.

Our market forecast shows new applications for EL as a commercial solvent at 30 million pounds and a price of $0.50 for the early market volumes. This is at 30% of the current price for THF and NMP. It is also favorably priced against DMSO, ethyl acetate and dibutyl ether.

Phase 2 (longer term) targeted derivatives

Tetrahydrofuran (THF)

THF, which is a four-carbon cyclic ether, is produced in the USA by four or five manufacturers who use a variety of processes. The annual volume is 300 million pounds. About 75% is used to produce polytetramethylene ether glycol (PTMEG) and 25% as a specialty solvent. The PTMEG market continues to grow globally and it is expected additional THF will be required. The current solvent price for THF is $1.50 per pound, a price under upward pressure from the producers' internal use for PTMEG. The cost of THF is estimated at $0.80/lb.

THF available in high volumes and at low cost would be very attractive to PTMEG producers. They normally have to expand both THF and PTMEG capacity but high-volume THF from PBLP would eliminate a significant amount of capital cost.

Our marketing forecast is based on the premise that a new THF plant is normally 20–25 million pounds. PBLP would build a 40-million-pound plant with 30 million sold to a PTMEG producer at $0.60/lb and 10 million pounds sold into the solvent market at a stable price of $1.00 per pound.

Delta Amino Levulinic Acid (DALA)

The agricultural market place is always on the alert for more efficient and environmentally friendly pesticides. A new process for delta amino levulinic acid (DALA), a broad spectrum herbicide and insecticide made from LA, has been developed. Each step in the new process provides high yields (over 80%) and high purity (over 90%) providing a commercially viable process. DALA is highly effective and quickly biodegradable.

The marketing plan is for PBLP to establish strategic partnership(s) with major agricultural chemical companies to develop herbicide and pesticide markets.

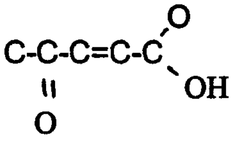
Our market forecast is based on the value of the USA pesticide market being $600,000,000 at the producer level; 23% is herbicide; 73% is insecticide.

The Department of Agriculture has published statistics on the number of acres and the pounds of herbicide used by fifteen (15) Mid-Western and mid-central states on corn and soybean crops in 1999. In corn, 68 million acres used 154 million pounds of herbicide. Although Atrazine was the most popular product, the market was very fragmented, with over forty (40) chemical compounds used by growers.

In soybeans 68 million acres was also planted, with 71 million pounds of herbicide used in that application. Glyphosate had the highest volume but over thirty (30) different chemical compounds were used.

Our market estimate assumes a 10% penetration into the corn and soybean crops with one-and-a-half pounds of DALA used per acre at a price of $2.00 per pound. 139 million acres X 10% equals 14 million acres times 1.5 pounds per acre times $2.00 per pound equals $42 million.

Aceto acrylic Acid (AAA)
In the production of delta amino levulinic acid (DALA), LBLP would produce one mole of AAA per mole of DALA.



$\mathrm{C{-}C({=}O){-}C{=}C{-}C({=}O)OH}$

This compound looks very similar to LA, except for the double bond between carbon 2 and 3, and may be an attractive platform chemical for AAA derivatives.

No work has been done on identifying high-value uses for AAA. However, it should not present a disposal problem since it is likely that AAA can be hydrogenated, a very common chemical procedure, into crude LA and returned to a LA production plant for purification into virgin LA

In our market forecast we would give it a value of $0.10 per pound.

Chemical Industry Services, Inc.
2707 Stemwell Boulevard, Richmond, VA 23236
Tel: (804) 745-4565
Fax: (804) 745-6877
e-mail: rcbilski@rcn.com

January 29, 2001

Rationale for Market Penetration Numbers
LA Chemical Derivatives

Overview:

In developing the market penetration percentages for the seven (7) targeted (chosen) derivatives, several concepts were considered and used in our work. The first was that the amount of LA needed to make the chemicals could be obtained by taking half the plant capacity of one or two PBLP plants, leaving the other half for use in the fuels markets. This would allow the plants to produce LA at a low unit cost and place half the volume in high-value chemicals.

The other major concept was to choose volumes that would allow our customers to use PBLP chemicals and avoid or delay expansions of their own plants by using the PBLP materials to grow their business.

Also, because of PBLP's low cost for LA and derivatives, we were able to price our products below our customer's costs, making it an easy make-or-buy decision.

Individual Products:

LA

Existing market is small (under 1 million pounds) and supplied by a European manufacturer who requires long lead times. We believe that 25% of the customers would qualify and support a U.S. producer at a selling price of $2.00 per pound versus $4 to $5 per pound. The other 75% make such high value derivatives from the LA that they would likely be indifferent to the savings.

Ten million pounds for new uses is a conservative number based on reports from researchers as to the versatility of low-cost LA as a new feedstock.

FA

The current market is 60 million pounds. PBLP co-produces FA with LA and needs to sell it, even at a low cost, to avoid using it as a fuel at the LA plant. We believe that PBLP can move up to 50% of the market at $0.20 per pound to the current producers and avoid being a disruptive factor in the market.

DPA

The 2-billion-pound market for BPA continues to grow. Since the principal end-uses are low-cost, high-volume plastics like polyesters and polycarbonates, PBLP should be able to sell 300MM lb. (a 15% penetration) to allow a BPA producer to test DPA in polymers and defer building another BPA expansion. They should find $0.40/lb. considerably below their cost of around $0.60 per pound.

Work funded by PBLP shows that some interesting polymer properties can be obtained by adding DPA to certain BPA formulations. This should allow PBLP to sell about 20MM pounds as an additive with price of $0.60 a pound.

EL as a Solvent

The solvent market is huge, with all companies looking for new solvents that give better performance, lower VOCs and biodegradability. We have not done a lot of work in this application but recently learned of a West Coast company who is very bullish on LA esters. Thirty million pounds is likely to be a conservative estimate of under 5% penetration.

THF

About 75% of the THF produced goes into the production of PTMEG. At a 10% penetration (30 million pounds) PBLP can offer THF at a price of $0.60 per pound. This should be below any producer's cost and allow them to avoid building a THF plant when they need to add PTMEG capacity.

The solvent market penetration of 13% is achievable because at $1.00 per pound it would allow a reliable supplier and a price considerably below the $1.50 solvent price – which is always tight.

DALA

The corn and soybean markets are served by over seventy (70) different compounds, many of which are under attack by the EPA for toxicity, environmental pollution and questionable effectiveness. A new herbicide compound, working by a unique mechanism, as DALA does in killing weeds, and rapidly biodegrading, should be an AG Conventions star attraction. The 10% market penetration would be achieved by marketing the product through existing AG companies who would like to be on the cutting edge with this technology.

Raymond J. Bilski

APPENDICES

Manager Run sheet

Date: 9-Mar
Senior operator: CM
Run ID#: 030998a

Element ID	Description	Design	Set points This run	Allow. rge	Notes
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	175	None	Remains set for run 3.0% H_2SO_4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.
	Ratio of #Solids # recycle acid.	0.15	0.08	0 - 0.1	80 #/hr KC, once at thermal equilibrium.
LIC-0104	Level control in T-101.	30"	30	15-35	
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to maintain FT-121.				
	FT-121	2.3 gpm	2.3	None.	Minimum allowable setting = 10%. 12 sec. Res. time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	None.	Controlled from Honeywell UDC controller, don't adjust manually.
PIC-0202	R-101 pressure control.	400 psi	380	None.	Controlled from Honeywell UDC controller, don't adjust manually.
LIC-0205	R-102 level control.	36"	42	None.	30 min. res. time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210	None.	
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	In MAN		
LIC-0215	Level control in HE-101.	15"	10	As required to maintain PIC-208	
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20		
TIC-0312	Hydrolysate temperature control.	100 F	100	None.	
LIC-0324	T-201 level control.	12"	-	On/off control	
FIC-0344	Cascade flow controller setting flow to C-301.	-			
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12	15-Jun	
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for the loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100	None.	
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	25	20 - 30	
TIC-0612	Recycle acid cooler.	100 F	100	None.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	18	10 - 25	
FIC-0630	MTHF flow control to C-301. Ratioed from FIC-0344. (gpm MTHF/gpm Hyd.)	Ratio	1.5	None.	
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	60#/gpm	40 - 60	
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, cascade set point from LIC-415.	-			
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	50	40 - 60	
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mmHg	150	150 - 200	

03-09-98 prelim

FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10	10 - 20	
TIC-0724	MTHF stripper bottom's temperature control, Provides cascaded set point to LIC-0720.	250 F	200	180 - 220	
FIC-0746	Steam flow controller to HE-510, cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	10	2 - 20	
FIC-0861	Crude LVAC flow control loop FIC-0861 Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	None.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	6.8	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/11/97
RUN DESIGNATION:	091197-1
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	10 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.05 gpm	
Total cellulose in	3.9 kg/hr	
Total potential LVAC in	2.8 kg/hr	
LVAC conc from recycle acid	3.19 g/L	
LVAC in from recycle acid	1.5	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.30 gpm 534 L/hr	
LVAC composition	6.76 g/L	
Total LVAC out	3.61 kg/hr	
Yield		
LVAC produced/fiber in	53.2%	
Crude LVAC composition	476 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/16/97
RUN DESIGNATION:	091597-2
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	25 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.20 gpm	
Total cellulose in	9.8 kg/hr	
Total potential LVAC in	7.0 kg/hr	
LVAC conc from recycle acid	1.17 g/L	
LVAC in from recycle acid	0.6	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.46 gpm 573 L/hr	
LVAC composition	8.3 g/L	
Total LVAC out	4.75 kg/hr	
Yield		
LVAC produced/fiber in	**42.4%**	
Crude LVAC composition	510 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/18/97
RUN DESIGNATION:	091597-3
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	20 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.35 gpm	
Total cellulose in	7.8 kg/hr	
Total potential LVAC in	5.6 kg/hr	
LVAC conc from recycle acid	0.31 g/L	
LVAC in from recycle acid	0.2	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.63 gpm	
	612 L/hr	
LVAC composition	7 g/L	
Total LVAC out	4.28 kg/hr	
Yield		
LVAC produced/fiber in	**52.5%**	
Crude LVAC composition	403 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/23/97
RUN DESIGNATION:	092297-1
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	20 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.30 gpm	
Total cellulose in	7.8 kg/hr	
Total potential LVAC in	5.6 kg/hr	
LVAC conc from recycle acid	0.72 g/L	
LVAC in from recycle acid	0.4	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.58 gpm	
	599 L/hr	
LVAC composition	6.22 g/L	
Total LVAC out	3.73 kg/hr	
Yield		
LVAC produced/fiber in	**42.6%**	
Crude LVAC composition	750 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/23/97
RUN DESIGNATION:	092297-2
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	20 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.30 gpm	
Total cellulose in	7.8 kg/hr	
Total potential LVAC in	5.6 kg/hr	
LVAC conc from recycle acid	1.23 g/L	
LVAC in from recycle acid	0.7	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.58 gpm 599 L/hr	
LVAC composition	7.82 g/L	
Total LVAC out	4.68 kg/hr	
Yield		
LVAC produced/fiber in	**51.3%**	
Crude LVAC composition	718 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/23/97
RUN DESIGNATION:	092297-3
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	20 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.30 gpm	
Total cellulose in	7.8 kg/hr	
Total potential LVAC in	5.6 kg/hr	
LVAC conc from recycle acid	0.45 g/L	
LVAC in from recycle acid	0.2	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.58 gpm 599 L/hr	
LVAC composition	5.55 g/L	
Total LVAC out	3.32 kg/hr	
Yield		
LVAC produced/fiber in	**39.3%**	
Crude LVAC composition	718 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/23/97
RUN DESIGNATION:	092297-4
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	20 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	2.35 gpm	
Total cellulose in	7.8 kg/hr	
Total potential LVAC in	5.6 kg/hr	
LVAC conc from recycle acid	0.87 g/L	
LVAC in from recycle acid	0.5	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.63 gpm	
	612 L/hr	
LVAC composition	5.49 g/L	
Total LVAC out	3.36 kg/hr	
Yield		
LVAC produced/fiber in	**36.8%**	
Crude LVAC composition	718 g/L	

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE:	9/30/97
DATE OF RUN:	9/23/97
RUN DESIGNATION:	092297-5
FEED TYPE AND SOURCE:	SWK pulp
COMPILED BY:	CS

Process flow	Composition/flow	Notes
Feed solids		
Feed rate	16 #/hr	
% solids	94.0%	
% cellulose (dry basis)	92.0%	
P-101 feed rate	1.85 gpm	
Total cellulose in	6.3 kg/hr	
Total potential LVAC in	4.5 kg/hr	
LVAC conc from recycle acid	1.49 g/L	
LVAC in from recycle acid	0.6	
Hydrolysate discharge to CF-201		
Total liquid flowrate	2.07 gpm 482 L/hr	
LVAC composition	6.6 g/L	
Total LVAC out	3.18 kg/hr	
Yield		
LVAC produced/fiber in	**40.5%**	
Crude LVAC composition	718 g/L	

LVAC PRODUCTION RUN SUMMARY

DATE: ~~5/11/97~~
DATE OF RUN: 02/02/98 ~~[illegible]~~ through 5/14/98
RUN DESIGNATION: 020298a
FEED TYPE AND SOURCE: 0

Bags Fed	Pounds	% Moisture	Dry Weight(lbs)	Cellulose Comp. (%)	Cellullose Weight(lbs)
-	2070.0	35.0%	1345.5	58.0%	780.4

	LVAC Drums	Production Gallons	Schedule (Pounds)	LVAC (g/L) (average)	#LVAC
Crude	0.0	Not determined	0.0	0.0	0.0
Refined	0.0	Not determined	0.0	0.0	0.0
Change in process inventory					
T-205		0.0	0.0	0.0	0.0
T-702		0.0	0.0	0.0	0.0
Process losses					
Condensate		0.0	0.0	0.0	0.0
Centrifuge tar	0.0	0.0	0.0	0.0	0.0
Acid stripper discharge		0.0	0.0	0.0	0.0
WFE tar		0.0	0.0	0.0	0.0

Totals	LVAC make (pounds)	0
Yield	lb LVAC/lb fiber	0.0%
	Acid stripper waste discharge (gpm)$_{\text{average}}$	-
	hours discharged	-
	Liquid Effluent (gallons)	-
	LP steam use (pounds)	20,000
	Power use kW.hrs	10,000

COMMENTS: (Significant non-routine activity, un-planned maintenance planned maintenance, etc.)

P-102 rotor was replaced due to wear.
FT-121 electrodes replaced.
Centrifuge feed tube broke due to CF-201 vibration and was replaced.
Replace bearings and reassemble C-301.
PI-0119 gasket leak repaired.

02-02-98 prelim

R-101, R-102, T-102, CF-201 mass balance

Date of Run: 02-Feb-98
Run ID: 020298a
Sample description 0 0:00

Condensate
To waste

Flowrate, gpm	1.1
Flowrate, #/hr	537.9
Glucose	0.0
Formic acid	2.4
LVAC	0.4
Xylose	0.0
Furfural	0.0

Recycle acid
From C-410

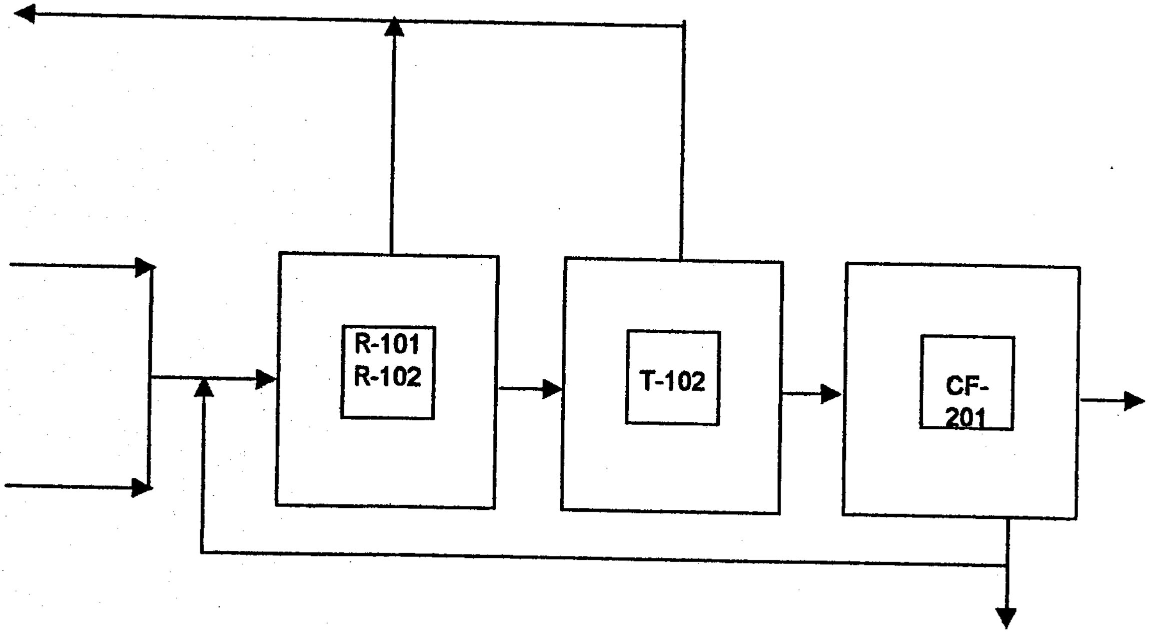
Flowrate, gpm	2.3
Flowrate, #/hr	1145.4
Glucose	0.8
Formic acid	2.9
LVAC	8.4
Xylose	0.0
Furfural	0.0

Steam to R-101

Flowrate, #/hr	538.2

Solids to T-101

Flowrate, #/hr	80
Cellulose	58%
Ash	8%
% solids	65%



Solid waste

Flowrate (#/hr)	41.5
Glucose	0.8
Formic acid	3.5
LVAC	12.5
Xylose	0.0
Furfural	0.0

To C-301

Flowrate, gpm	2.5
Flowrate, #/hr	1252.8
Glucose	1.1
Formic acid	4.9
LVAC	17.5
Xylose	0.0
Furfural	0.0

	R-101	R-102
Pressure (psig)	380	200
Temp. (F)	428	394
Residence time	12	30
	(sec.)	(min.)

Reactor yield 55.2% LVAC of stoichiometric
39.5% LVAC/Cellulose (wt%)

Manager Run sheet

Date: 2-Feb
Senior operator: CM
Run ID#: 020298a

Element ID	Description		Set points		Notes	
		Design	This run	Allow. rge		
PID 001	**Reactant Blending and Wood Feed**					
AIC-0102	Recycle acid a pH control	150 µS/cm	175	None	Remains set for run	3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.	
	Ratio of #Solids # recycle acid.	0.15	0.1	0 - 0.1	100 #/hr KC, once at thermal equilibrium.	
LIC-0104	Level control in T-101.	30"	30	15-35		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-				
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to maintain FT-121.					
	FT-121	2.3 gpm	2.3	None.	Minimum allowable setting = 10%.	12 sec. Res. time
PID-002	**Reaction**					
TIC-0201	R-101 temperature control.	428 F	428	None.	Controlled from Honeywell UDC controller, don't adjust manually.	
PIC-0202	R-101 pressure control.	400 psi	380	None.	Controlled from Honeywell UDC controller, don't adjust manually.	
LIC-0205	R-102 level control.	36"	42	None.		30 min. res. time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig	
	The controller receives a set point from TIC-0212.	210 psig	210	None.		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	In MAN			
LIC-0215	Level control in HE-101.	15"	10	As required to maintain PIC-208		
PID-003	**Solids Removal**					
LIC-0301	Level control in T-102.	30"	20			
TIC-0312	Hydrolysate temperature control.	100 F	100	None.		
LIC-0324	T-201 level control.	12"	-	On/off control		
FIC-0344	Cascade flow controller setting flow to C-301.	-				
PID-004	**Solvent Extraction**					
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12	15-Jun		
FIC-0702	Flow control the flow of MTHF to the solvent stripper.					
	The set point for ths loop is generated from LIC-0405.	-				
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100	None.		
PID-006	**Acid Stripper**					
LIC-0601	Level control in C-410.	20"	25	20 - 30		
TIC-0612	Recycle acid cooler.	100 F	100	None.		
LIC-0624	T-415 interface level control to FIC-0641.	18"	18	10 - 25		
FIC-0630	MTHF flow control to C-301. Ratioed from FIC-0344. (gpm MTHF/gpm Hyd.)	Ratio	1.5	None.		
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	60#/gpm	40 - 60		
	This loop is based on a set point from FIC-0641.					
FIC-0641	Reflux/feed flow controller to C-410, cascade set point from LIC-415.	-				
PID-007	**Crude LA MTHF Stripping**					
LIC-0703	Level control in C-510.	6"	50	40 - 60		
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mmHg	150	150 - 200		

FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10	10 - 20	
TIC-0724	MTHF stripper bottom's temperature control, Provides cascaded set point to LIC-0720.	250 F	200	180 - 220	
FIC-0746	Steam flow controller to HE-510, cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	10	2 - 20	
FIC-0861	Crude LVAC flow control loop FIC-0861 Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	None.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	6.8	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

LVAC PRODUCTION RUN SUMMARY

DATE: 2/16/98
DATE OF RUN: 2/9/98 through 2/12/98
RUN DESIGNATION: 020998ABCD
FEED TYPE AND SOURCE: Dry paper mill sludge ex-KC/HF
COMPILED BY: CS

Bags Fed	Pounds	% Moisture	Dry Weight(lbs)	Cellulose Comp. (%)	Cellullose Weight(lbs)
12	4,520	35.0%	2938	58.0%	1704

	LVAC Drums	Production Gallons	Schedule (Pounds)	LVAC (g/L) (average)	#LVAC
Crude	4	123	1,123	231	243
Refined	-	-	-	-	-
In process					
T-205		170	1,408	60	88
T-702		-	-	-	-
Process losses					
Centrifuge tar	5		2,511	60	90
Acid stripper discharge		3,600	29,880	11	338
WFE tar	-	-	-	-	-

Totals	LVAC make (pounds)	760
Yield	lb LVAC/lb cellulose	44.6%
Acid stripper waste discharge	(lb/hr)$_{average}$	598
	hours discharged	50
	Liquid Effluent (gallons)	3,600
	LP steam use (pounds)	26,900
	Power use KW.hrs	15,000

COMMENTS: (Significant non-routine activity, un-planned maintenance planned maintenance, etc.)

Excessive loss of LVAC through acid stripper due to solids in extractor, extractor maintenance performed after run.
Input calculations are estimated averages to be confirmed with lab analysis
Significant variations in feed composition noted through bags and between bags
Pumping aid added during run
Operating software to be changed to ensure consistent solvent flow to extraction

CONTINUOUS LVAC YIELD ACROSS R-101, R-102

DATE: 2/16/97
DATE OF RUN: 2/9/97
RUN DESIGNATION: 020997D
FEED TYPE AND SOURCE: KCBB
COMPILED BY: CS

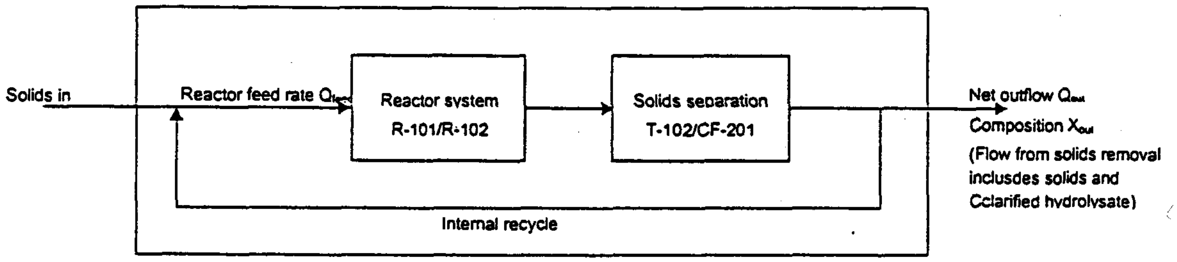
Process flow	Composition/flow	Notes
Feed solids		
Feed rate	80 #/hr	
% solids	65.0%	
% cellulose (dry basis)	58.0%	
P-101 feed rate	2.50 gpm	
Total cellulose in	13.7 kg/hr	
Total potential LVAC in	9.7 kg/hr	
LVAC conc from recycle acid	0.00 g/L	
LVAC in from recycle acid	0.0	
Hydrolysate discharge to C-301		
Total liquid flowrate	0.42 gpm 98 L/hr	
LVAC composition	59.3 g/L	
Total LVAC out	5.79 kg/hr	
Yield		
LVAC produced/fiber in	42%	
Crude LVAC composition	750 g/L	

Revised 04/22/98
w/Actual cellulose.

BioMetics, Inc.
Memorandum

TO: SWF
COPY: HG
DATE: March 4, 1998
FROM: Colin South
SUBJECT: Preliminary steady state reactor yield, plant run 02/09/98 - 02/13/98

Steve:
In addition ot the overall input/output mass balance reported on the standard plant reporting data sheet the LVAC yield across R-101/102 has been calculated using continuous steady state data.. The configuration of the reactor system is as shown:



The balance across the reactors in the current configuration is calculated from a continuous balance as follows:

Process flow	Calculation
Rate of cellulose in	Rate of solids addition (kg/hr) x %solids x % cellulose
Rate of potential LVAC in	Rate of cellulose in x stoichiometic constant (MW LVAC/ MW cellulose monomer)
Rate of LVAC out	Q_{out} (L/hr) x LVAC composition (g/L)
LVAC yield (% of cellulose feed)	LVAC out/rate of cellulose in
LVAC yield (% of stoichiometric)	LVAC out/rate of potential LVAC in

The latest run of 02/23 had the following steady state compositions:

Process flow	Composition/flow	Notes
Feed solids	Feed rate(kg/hr) = 80#/hr KC/BB % solids = 50% % cellulose (dry basis) = 80% Total cellulose in (kg/kr) = 14.5 kg/hr Total potential LVAC in (kg/hr) = 10.3	
Hydrolysate discharge to CF-201	Total liquid flowrate(L/hr) = 110.7 L/hr (0.48 gpm) LVAC composition (g/L)= 65 g/L Total LVAC out (kg/hr) = 7.2 kg/hr	Calculated from water and steam inflows. Steady state composition is to be confirmed.
Yields	LVAC out/fiber in = 49.6%	

CS March 4, 1998
G:\FILES\DOCS\096-DOE\RUNDATA\Run spreadsheets\98 - 02 (Feb)\reactor yield 0209d.wpd

Manager Run sheet

Date: 02/09/98
Senior operator: CM
Run ID#: 020998A.

02/09 15:00 - 02/09 -22:00

Element ID	Description	Set points			Notes
		Design	This run	Allow. range	
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	165	Do not deviate.	Remains set for run 3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.
	Ratio of #Solids # recycle acid.	0.15	80 #/hr KC		Set for each individual feed bag as per atached feed sheet
LIC-0104	Level control in T-101.	30"	In MAN		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.				
	FT-121	2.3 gpm	2.3	Do not deviate.	Minimum allowable setting = 10%. 12 sec. residence time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
PIC-0202	R-101 pressure control.	400 psi	390	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
LIC-0205	R-102 level control.	32"	42	Do not deviate.	30 min. residence time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	392	Do not deviate.	
LIC-0215	Level control in HE-101.	15"	15		
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20		
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.	
LIC-0324	T-201 level control.	12"	NA		
FIC-0344	Cascade flow controller setting flow to C-301.	-	Recycle to maintain T-101 level		
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12		
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for ths loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100		
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	25		
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	22		
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	1.0		
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm		
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-			
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	NA		
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mm Hg	200		
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10		
TIC-0724	MTHF stripper bottom's temperature control,				
	Provides cascaded set point to LIC-0720.	250 F	180		
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 - 20"		
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	Do not deviate.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

Manager Run sheet

Date: 02/09/98
Senior operator: CM
Run ID#: c20998 B

02/09 10:00 - 02/10 7:00

Element ID	Description	Set points			Notes	
		Design	This run	Allow. range		
PID 001	**Reactant Blending and Wood Feed**					
AIC-0102	Recycle acid a pH control	150 μS/cm	165	Do not deviate.	Remains set for run	3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.	
	Ratio of #Solids # recycle acid.	0.15	100 #/hr KC 20#/hr binder		Set for each individual feed bag as per atached feed sheet	
LIC-0104	Level control in T-101.	30"	In MAN			
WI-0105	Indication and totalization only (incl. w/ VP-101).	-				
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.					
	FT-121	2.3 gpm	2.3	Do not deviate.	Minimum allowable setting = 10%.	12 sec. residence time
PID-002	**Reaction**					
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.	
PIC-0202	R-101 pressure control.	400 psi	390	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.	
LIC-0205	R-102 level control.	32"	42	Do not deviate.		30 min. residence time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig	
	The controller receives a set point from TIC-0212.	210 psig	210			
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	392	Do not deviate.		
LIC-0215	Level control in HE-101.	15"	15			
PID-003	**Solids Removal**					
LIC-0301	Level control in T-102.	30"	20			
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.		
LIC-0324	T-201 level control.	12"	NA			
FIC-0344	Cascade flow controller setting flow to C-301.	-	Recycle to maintain T-101 level			
PID-004	**Solvent Extraction**					
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12			
FIC-0702	Flow control the flow of MTHF to the solvent stripper.					
	The set point for ths loop is generated from LIC-0405.	-				
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100			
PID-006	**Acid Stripper**					
LIC-0601	Level control in C-410.	20"	25			
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.		
LIC-0624	T-415 interface level control to FIC-0641.	18"	22			
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	1.0			
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm			
	This loop is based on a set point from FIC-0641.					
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-				
PID-007	**Crude LA MTHF Stripping**					
LIC-0703	Level control in C-510.	6"	NA			
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mm Hg	200			
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-				
LIC-0722	MTHF level control in T-510.	10"	10			
TIC-0724	MTHF stripper bottom's temperature control,					
	Provides cascaded set point to LIC-0720.	250 F	180			
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702	-	Computer controlled			
PID 008	**Finishing (Air stripping and wiped film evaporation)**					
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 - 20"			
FIC-0861	Crude LVAC flow control loop FIC-0861					
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled			
TIC-0862	Air stripper inlet air temperature	300 F	300	Do not deviate.		
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller	
UFD 071	**Collection & Waste Neutralization Systems**					
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5		
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0		
	Effluent tank at start of run (circle).	T-7132	T-7133			

Manager Run sheet

Date: c2/10/98
Senior operator: DQ.
Run ID#: 020998C

02/10 7:15 - 02/10 -23:00

Element ID	Description	Set points			Notes
		Design	This run	Allow. range	
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	165	Do not deviate.	Remains set for run 3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.
	Ratio of #Solids # recycle acid.	0.15	100 #/hr KC 40#/hr binder		Set for each individual feed bag as per atached feed sheet
LIC-0104	Level control in T-101.	30"	In MAN		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.				
	FT-121	2.3 gpm	2.3	Do not deviate.	Minimum allowable setting = 10%. 12 sec. residence time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
PIC-0202	R-101 pressure control.	400 psi	390	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
LIC-0205	R-102 level control.	32"	42	Do not deviate.	30 min. residence time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	392	Do not deviate.	
LIC-0215	Level control in HE-101.	15"	15		
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20		
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.	
LIC-0324	T-201 level control.	12"	NA		
FIC-0344	Cascade flow controller setting flow to C-301.	-	Recycle to maintain T-101 level		
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12		
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for ths loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100		
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	25		
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	22		
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	1.0		
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm		
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-			
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	NA		
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mm Hg	200		
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10		
TIC-0724	MTHF stripper bottom's temperature control,				
	Provides cascaded set point to LIC-0720.	250 F	180		
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 , 20"		
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	Do not deviate.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

Manager Run sheet

Date: 02/12/98
Senior operator: DQ.
Run ID#: 021298D

02/12 7:15 - 02/12 -23:00

Element ID	Description	Set points			Notes
		Design	This run	Allow. range	
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	165	Do not deviate.	Remains set for run 3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.
	Ratio of #Solids # recycle acid.	0.15	80 #/hr KC		Set for each individual feed bag as per atached feed sheet
LIC-0104	Level control in T-101.	30"	In MAN		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.				
	FT-121	2.3 gpm	2.3	Do not deviate.	Minimum allowable setting = 10%. 12 sec. residence time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
PIC-0202	R-101 pressure control.	400 psi	390	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
LIC-0205	R-102 level control.	32"	42	Do not deviate.	30 min. residence time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	392	Do not deviate.	
LIC-0215	Level control in HE-101.	15"	15		
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20		
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.	
LIC-0324	T-201 level control.	12"	NA		
FIC-0344	Cascade flow controller setting flow to C-301.	-	Recycle to maintain T-101 level		
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12		
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for ths loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100		
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	25		
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	22		
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	1.0		
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm		
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-			
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	NA		
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mm Hg	200		
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10		
TIC-0724	MTHF stripper bottom's temperature control,				
	Provides cascaded set point to LIC-0720.	250 F	180		
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 - 20"		
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	Do not deviate.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

PLANT WEEKLY OPERATIONAL SUMMARY

PERIOD START DATE: 2/23/98 **PERIOD END DATE:** 2/27/98

OVERALL INVENTORY CHANGES

ITEM	UNITS		No./QUANTITY
Feedstock (Sludge fiber)	Bags		7 1/2
	Pounds	("As-is")	3280
	% Moist'r	(Average)	50
	Dry Pounds		1640
Crude Product	Drums		3
	Gallons		69.2
	Pounds		591
	% LVAC	(Average)	425
Refined Product	Drums		-
	Gallons		
	Pounds		
	% LVAC	(Average)	
Waste Tar Quantity	Loads		-
	Tons	(Estim.)	
Liquid Effluent	Loads		2
	Gallons	(Estim.)	4500
Estimated L.P. Steam	Pounds		28550
Estimated Power	KWHrs		15000

COMMENTS: (Significant non-routine activity, un-planned maintenance planned maintenance, etc.)

Calculations above are impacted by non steady state operation due to acid stripper.

Input calculations are estimated averages to be confirmed with lab analysis

Significant variations in composition noted through bags and between bags

T-101 modified to take non spec. feedstock

    Installed feeding platform
    Feeding 20#/hr binder with feed
    Feedstock composition to be determined

Centrifuge cleared and bearing changed prior to run

Extractor cleared of solids blockage (probably from previously bypassing centrifuge)

Poor acid stripper performance (unable to process solvent stripper stripping steam)

    Added bed delimiter, removed 50% of packing
    Modified distribution plate to EDLON recommendations

WFE distillate pump gear changed following breakage

Solvent stripper vacuum poor (apparently from stripping steam)

Circulated crude LVAC through air stripper and WFE to remove excess water from solvent stripper

GC septa have limited life on acidified samples alternate sources/types being evaluated

Manager Run sheet

Date: 02/23/98
Senior operator: CM
Run ID#: 022398A.

Element ID	Description	Set points			Notes	
		Design	This run	Allow. range		
PID 001	**Reactant Blending and Wood Feed**					
AIC-0102	Recycle acid a pH control	150 µS/cm	165	Do not deviate.	Remains set for run	3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.	
	Ratio of #Solids # recycle acid.	0.15	80 #KC 20 # binder/ hr		Set for each individual feed bag as per atached feed sheet	
LIC-0104	Level control in T-101.	30"	In MAN try to maintain constant levels in T101 and T205 by bleeding ~0.6 gpm to back room			
WI-0105	Indication and totalization only (incl. w/ VP-101).	-				
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.					
	FT-121	2.3 gpm	2.3	Do not deviate.	Minimum allowable setting = 10%.	12 sec. residence time
PID-002	**Reaction**					
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.	
PIC-0202	R-101 pressure control.	400 psi	380	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.	
LIC-0205	R-102 level control.	32"	42	Do not deviate.		30 min. residence time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig	
	The controller receives a set point from TIC-0212.	210 psig	210			
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	392	Do not deviate.		
LIC-0215	Level control in HE-101.	15"	15			
PID-003	**Solids Removal**					
LIC-0301	Level control in T-102.	30"	20			
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.		
LIC-0324	T-201 level control.	12"	-			
FIC-0344	Cascade flow controller setting flow to C-301.	-				
PID-004	**Solvent Extraction**					
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12			
FIC-0702	Flow control the flow of MTHF to the solvent stripper.					
	The set point for ths loop is generated from LIC-0405.	-				
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100			
PID-006	**Acid Stripper**					
LIC-0601	Level control in C-410.	20"	20			
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.		
LIC-0624	T-415 interface level control to FIC-0641.	18"	18			
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	0.75			
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm			
	This loop is based on a set point from FIC-0641.					
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-				
PID-007	**Crude LA MTHF Stripping**					
LIC-0703	Level control in C-510.	6"	6			
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mm Hg	200			
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-				
LIC-0722	MTHF level control in T-510.	10"	10			
TIC-0724	MTHF stripper bottom's temperature control,					
	Provides cascaded set point to LIC-0720.	250 F	200			
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702	-	Computer controlled			
PID 008	**Finishing (Air stripping and wiped film evaporation)**					
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 - 20"			
FIC-0861	Crude LVAC flow control loop FIC-0861					
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled			
TIC-0862	Air stripper inlet air temperature	300 F	300	Do not deviate.		
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller	
UFD 071	**Collection & Waste Neutralization Systems**					
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5		
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0		
	Effluent tank at start of run (circle).	T-7132	T-7133			

Manager Run sheet

Date: 2/17/98
Senior operator: CS#1
Run ID#: START up Tues

Element ID	Description	Set points Design	This run	Allow. range	Notes
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	165	Do not deviate.	Remains set for run 3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.
	Ratio of #Solids # recycle acid.	0.15	100#KC/20# binder /hr		Set for each individual feed bag as per atached feed sheet
LIC-0104	Level control in T-101.	30"	In MAN		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.				
	FT-121	2.3 gpm	2.3	Do not deviate.	Minimum allowable setting = 10%. 12 sec. residence time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
PIC-0202	R-101 pressure control.	400 psi	390	Do not deviate.	Controlled from Honeywell UDC controllers, do not adjust controllers manually.
LIC-0205	R-102 level control.	32"	42	Do not deviate.	30 min. residence time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	392	Do not deviate.	
LIC-0215	Level control in HE-101.	15"	As required		
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20		
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.	
LIC-0324	T-201 level control.	12"	NA		
FIC-0344	Cascade flow controller setting flow to C-301.	-	Recycle to maintain T-101 level		
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12		
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for ths loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100		
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	25		
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	22		
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	1.0		
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm		
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-			
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	-		
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mm Hg	200		
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10		
TIC-0724	MTHF stripper bottom's temperature control,				
	Provides cascaded set point to LIC-0720.	250 F	190 set manually from FIC-720		
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702	-	Computer controlled		

PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 - 20"		
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	Do not deviate.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

LVAC PRODUCTION RUN SUMMARY

DATE: 3/10/97
DATE OF RUN: 3/2/98 through 3/6/98
RUN DESIGNATION:
FEED TYPE AND SOURCE: Dry paper mill sludge ex-KC/HF

Bags Fed	Pounds	% Moisture	Dry Weight(lbs)	Cellulose Comp. (%)	Cellullose Weight(lbs)
3	1190	32.5%	804	58.0%	466

	LVAC Drums	Production Gallons	Schedule (Pounds)	LVAC (g/L (average)	#LVAC
Crude	1.0	41	374	421	144
Refined	0.0	0	0	0	0
Change in process inventory					
T-205		0	0	0	0
T-702		0	0	0	0
Process losses					
Centrifuge tar	1.0	55	532	60	28
Acid stripper discharge		1536	12749	6	77
WFE tar	0.0	0	0	0	0

Totals	LVAC make (pounds)	248
Yield	lb LVAC/lb fiber	53.3%

Acid stripper waste discharge (gpm)$_{average}$	0.8
hours discharged	32
Liquid Effluent (gallons)	1,536
LP steam use (pounds)	10,500
Power use kW.hrs	5,500

COMMENTS: (Significant non-routine activity, un-planned maintenance planned maintenance, etc.)

Calculations above are impacted by periodic non steady state operation arising from repeated malfunction of the centrifuge.
Input calculations are estimated averages to be confirmed with lab analysis
Calculations above are preliminary and will be verified by 3/13/98
Significant variations in composition noted through bags and between bags
Centrifuge malfunction; unit sent out to be cleared and bearing changed
Solvent stripper vacuum poor (apparently from stripping steam)
Replaced packing on P101
WFE distillate pump replaced with more reliable reservoir system
Minor piping modifications around air stripper to improve reliability
Addition of control valve facilitating level control in T101 in conjuction with recycle mode of operation
Removal of packing from HE-510 and adjoining lines, reinsulated and proofed for vacuum leaks. Much improved vacuum performance.
Replaced seals on VP-513 - exhibited oring degradation.
Circulated crude LVAC through air stripper and WFE to remove excess water
Laboratory work focusing on increasing sample throughput rate and accuracy of analysis.

PIC-0710	Pressure control loop for the MTHF vacuum system.		Computer controlled		
FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-		Max steam flow 150 #/hr	
LIC-0722	MTHF level control in T-510.	10"	10	5 - 20	
TIC-0724	MTHF stripper bottom's temperature control,	250 F	200	180 - 220	
	Preheat steam ratio (#/hr per gpm)	200	200	150 - 200	
FIC-0746	Steam flow controller to HE-510, receives cacaded flow control from FIC-0702		Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	2 - 20"		
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	220	200 - 250	
	Chiller temperature setpoint	60F	75	55 - 80	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

Manager Run sheet

Date:	9-Mar
Senior operator:	CM
Run ID#:	030998a

Element ID	Description		Set points		Notes
		Design	This run	Allow. rge	
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	175	None	Remains set for run 3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.
	Ratio of #Solids # recycle acid.	0.15	0.08	0 - 0.1	80 #/hr KC, once at thermal equilibrium.
LIC-0104	Level control in T-101.	30"	30	15-35	
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to maintain FT-121.				
	FT-121	2.3 gpm	2.3	None.	Minimum allowable setting = 10%. 12 sec. Res. time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	None.	Controlled from Honeywell UDC controller, don't adjust manually.
PIC-0202	R-101 pressure control.	400 psi	380	None.	Controlled from Honeywell UDC controller, don't adjust manually.
LIC-0205	R-102 level control.	36"	42	None.	30 min. res. time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210	None.	
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	In MAN		
LIC-0215	Level control in HE-101.	15"	10	As required to maintain PIC-208	
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20		
TIC-0312	Hydrolysate temperature control.	100 F	100	None.	
LIC-0324	T-201 level control.	12"	-	On/off control	
FIC-0344	Cascade flow controller setting flow to C-301.	-			
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12	15-Jun	
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for ths loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100	None.	
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	25	20 - 30	
TIC-0612	Recycle acid cooler.	100 F	100	None.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	18	10 - 25	
FIC-0630	MTHF flow control to C-301. Ratioed from FIC-0344. (gpm MTHF/gpm Hyd.)	Ratio	1.5	None.	
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	60#/gpm	40 - 60	
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, cascade set point from LIC-415.	-			
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	50	40 - 60	
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mmHg	150	150 - 200	

FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10	10 - 20	
TIC-0724	MTHF stripper bottom's temperature control,				
	Provides cascaded set point to LIC-0720.	250 F	200	180 - 220	
FIC-0746	Steam flow controller to HE-510, cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	Finishing (Air stripping and wiped film evaporation)				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	10	2 - 20	
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	None.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	Collection & Waste Neutralization Systems				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	6.8	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

R-101, R-102, T-102, CF-201 mass balance

Date of Run:	9-Mar-98
Run ID:	030998a
Sample description:	0

Condensate
To waste

Flowrate, gpm	1.2
Flowrate, #/hr	596.4
Glucose (g/L)	0.0
Formic acid (g/L)	2.7
LVAC (g/L)	1.3
Xylose (g/L)	0.0
Furfural (g/L)	0.0

Recycle acid
From C-410

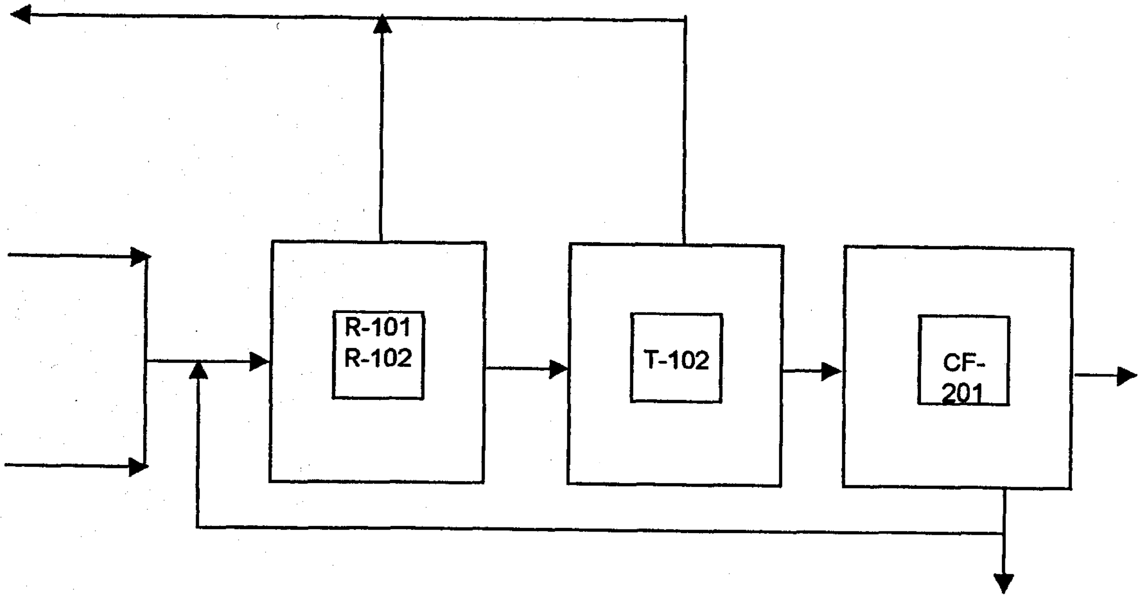
Flowrate, gpm	0.5
Flowrate, #/hr	259.0
Glucose (g/L)	0.5
Formic acid (g/L)	3.3
LVAC (g/L)	11.9
Xylose (g/L)	n.d.
Furfural (g/L)	n.d.

Steam to R-101

Flowrate, #/hr	596.7

Solids to T-101

Flowrate, #/hr	80
Cellulose	58%
Ash	8%
% solids	50%



Solid waste

Flowrate, #/hr	34.6
Glucose (g/L)	0.4
Formic acid (g/L)	5.1
LVAC (g/L)	38.9
Xylose (g/L)	0.0
Furfural (g/L)	0.0

To C-301

Flowrate, gpm	0.5
Flowrate, #/hr	259.0
Glucose (g/L)	0.4
Formic acid (g/L)	5.1
LVAC (g/L)	38.9
Xylose (g/L)	0.0
Furfural (g/L)	0.0

	R-101	R-102
Pressure (psig)	380	200
Temp. (F)	428	394
Residence time	13.3043 (sec.)	30 (min.)

Reactor yield	61.4%	LVAC of stoichiometric
	43.9%	LVAC/Cellulose (wt%)
	105.9%	Formic based on LVAC production

03-09-98 prelim-rev 1

C-301 mass balance

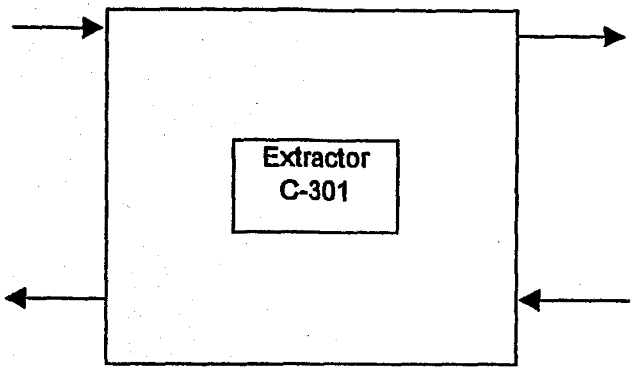
Date of Run: 09-Mar-98
Run ID: 030998a
Sample descriptior 030998a 0:00

Hydrolysate
From T-205

Flowrate, gpm	0.5
Glucose (g/L)	0.4
Formic acid (g/L)	5.1
LVAC (g/L)	38.9
Xylose (g/L)	0.0
Furfural (g/L)	0.0

MTHF with Levulinic Acid
To C-510

Flowrate, gpm	0.7
Glucose (g/L)	n.d.
Formic acid (g/L)	4.5
LVAC (g/L)	37.3
Xylose (g/L)	n.d.
Furfural (g/L)	n.d.



Recycle Acid to C-410

Flowrate, gpm	0.6
Glucose (g/L)	0.5
Formic acid (g/L)	3.3
LVAC (g/L)	11.9
Xylose (g/L)	n.d.
Furfural (g/L)	n.d.

Recycle MTHF

Flowrate, gpm	0.6
Glucose (g/L)	n.d.
Formic acid (g/L)	3.6
LVAC (g/L)	17.4
Xylose (g/L)	n.d.
Furfural (g/L)	n.d.

LVAC balance	96.50%
LVAC recovery to C-510	63.79%

Manager Run sheet

Date: 5/4/98
Senior operator: DQ
Run ID#: r050498

Element ID	Description	Set points			Notes
		Design	This run	Allow. range	
PID 001	**Reactant Blending and Wood Feed**				
AIC-0102	Recycle acid a pH control	150 µS/cm	165	Do not deviate.	Remains set for run 3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:			Introduce solids once reactor at operating conditions.	
	Ratio of #Solids # recycle acid.	0.15	-	Soft wood kraft @ 20#/hr	
LIC-0104	Level control in T-101.	30"	30		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-			
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to obtain desired FT-121.				
	FT-121	2.3 gpm	2.3	Do not deviate.	Min. setting = 30%. 12 sec. Res. time
PID-002	**Reaction**				
TIC-0201	R-101 temperature control.	428 F	428	Do not deviate.	Se manually at Honeywell UDC controller
PIC-0202	R-101 pressure control.	400 psi	380	Do not deviate.	Controlled from Honeywell UDC controller
LIC-0205	R-102 level control.	55"	65	Do not deviate.	39.4 min. Res. time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig
	The controller receives a set point from TIC-0212.	210 psig	210		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	-	Not active set MANUAL =0% output	
LIC-0215	Level control in HE-101.	15"	-	As required to maintain PT-208	
PID-003	**Solids Removal**				
LIC-0301	Level control in T-102.	30"	20	10 - 50	
TIC-0312	Hydrolysate temperature control.	100 F	100	Do not deviate.	
LIC-0324	T-201 level control.	12"	-	Automatic on/off control	
FIC-0344	Cascade flow controller setting flow to C-301.	-		Max 1 gpm	
PID-004	**Solvent Extraction**				
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12		
FIC-0702	Flow control the flow of MTHF to the solvent stripper.				
	The set point for the loop is generated from LIC-0405.	-			
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100	Not to exced 140	
PID-006	**Acid Stripper**				
LIC-0601	Level control in C-410.	20"	20	15 - 30	
TIC-0612	Recycle acid cooler.	100 F	100	Do not deviate.	
LIC-0624	T-415 interface level control to FIC-0641.	18"	18	12 - 25	
FIC-0630	MTHF flow control to C-301. Flow is ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	0.75	1.5	
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	40#/gpm	40	
	This loop is based on a set point from FIC-0641.				
FIC-0641	Reflux/feed flow controller to C-410, controller cascade set point from LIC-415.	-		Not to exceed 2.5 gpm	
PID-007	**Crude LA MTHF Stripping**				
LIC-0703	Level control in C-510.	6"	50		
	Solvent stripper pressure remote set point	150 mm Hg	200	150 - 250	

LVAC PRODUCTION RUN SUMMARY

DATE: 5/11/97

DATE OF RUN: 5/11/98 through 5/14/98

RUN DESIGNATION:

FEED TYPE AND SOURCE: Sorted and dried MSW

Bags Fed	Pounds	% Moisture	Dry Weight(lbs)	Cellulose Comp. (%)	Cellullose Weight(lbs)
-	0.0	0.0%	0.0	0.0%	0.0

	LVAC Drums	Production Gallons	Schedule (Pounds)	LVAC (g/L) (average)	#LVAC
Crude	0.0	0.0	0.0	0.0	0.0
Refined	0.0	0.0	0.0	0.0	0.0
Change in process inventory					
T-205		0.0	0.0	0.0	0.0
T-702		0.0	0.0	0.0	0.0
Process losses					
Condensate		1000.0	8300.0	0.0	0.0
Centrifuge tar	0.0	0.0	0.0	0.0	0.0
Acid stripper discharge		0.0	0.0	0.0	0.0
WFE tar	0.0	0.0	0.0	0.0	0.0

Totals	LVAC make (pounds)	0
Yield	lb LVAC/lb fiber	0.0%
	Acid stripper waste discharge (gpm)$_{average}$	-
	hours discharged	-
	Liquid Effluent (gallons)	1,000
	LP steam use (pounds)	2,000
	Power use kW.hrs	2,000

COMMENTS: (Significant non-routine activity, un-planned maintenance planned maintenance, etc.)

HP reactor skid

LIC-0215 pressure sensor diaphragms replaced with Zr diaphragms

Relief lines from R-102 and T-102 separated to prevent back pressure on T-102 in the event of R-102 disc failure.

WFE

Sightglass broken during cleaning. Replaced with spare glass.

Devolitizer cracked during operation. Replaced glass unit with stainless steel drum.

Vacuum pump not holding vacuum. Loses pressure from 10 - 100 mmHg over 1 hour operation. Oil to be replaced with higher viscosity.

WFE feed pump P-512 bushings replaced.

Solvent stripper

Solids deposition in base of Solvent stripper cleared.

MPS boiler

MPS boiler filling with water during process run. Notified EPIC for resolution.

Unable to run Solvent room Thursday due to boiler problems.

Manager Run sheet

Date: 11-May
Senior operator: CM
Run ID#: 051198a

Element ID	Description	Design	Set points This run	Allow. rge	Notes	
PID 001	**Reactant Blending and Wood Feed**					
AIC-0102	Recycle acid a pH control	150 µS/cm	100	None	Remains set for run	3.0% H2SO4
FIQC-0103	Solids flow controller for the solids feeding system, VP-101:				Introduce solids once reactor system approaches operating conditions.	
	Ratio of #Solids # recycle acid.	0.15			50 #/hr MSW, record at each addition	
LIC-0104	Level control in T-101.	30"	30	15-35		
WI-0105	Indication and totalization only (incl. w/ VP-101).	-				
SIC-0115	The speed control for P-101 to set slurry flowrate. Set % to maintain FT-121.					
	FT-121	2.3 gpm	2.3	None.	Minimum allowable setting = 30%.	12 sec. Res. time
PID-002	**Reaction**					
TIC-0201	R-101 temperature control.	428 F	428	None.	Controlled manually from Honeywell UDC controller	
PIC-0202	R-101 pressure control.	400 psi	380	None.	Controlled from Honeywell UDC controller, don't adjust manually.	
LIC-0205	R-102 level control.	32"	65	None.		30 min. res. time
PIC-0208	Cascade controller for a self-regulating loop that maintains R-102 pressure.				Bursting disc pressure = 250 psig	
	The controller receives a set point from TIC-0212.	210 psig	210	None.		
TIC-0212	R-102 temperature control loop, provides cascaded set point to PIC-208.	392 F	In MAN			
LIC-0215	Level control in HE-101.	15"	15	As required to maintain PIC-208		
PID-003	**Solids Removal**					
LIC-0301	Level control in T-102.	30"	20			
TIC-0312	Hydrolysate temperature control.	100 F	100	None.		
LIC-0324	T-201 level control.	12"	-	On/off control		
FIC-0344	Cascade flow controller setting flow to C-301.	-				
PID-004	**Solvent Extraction**					
LIC-0405	T-301 level control, provides cascade flow set point to FIC-0702.	12"	12			
FIC-0702	Flow control the flow of MTHF to the solvent stripper.					
	The set point for ths loop is generated from LIC-0405.	-				
TIC-0409	Temperature control for the recycle MTHF to C-301.	100 F	100	None.		
PID-006	**Acid Stripper**					
LIC-0601	Level control in C-410.	20"	25	20 - 30		
TIC-0612	Recycle acid cooler.	100 F	100	None.		
LIC-0624	T-415 interface level control to FIC-0641.	18"	18	10 - 25		
FIC-0630	MTHF flow control to C-301. Ratioed from FIC-0344. (gpm MTHF/gpm)	Ratio	1.5	None.		
FIC-0633	Flow control loop for Steam to HE-410.	Ratio	60#/gpm	40 - 60		
	This loop is based on a set point from FIC-0641.					
FIC-0641	Reflux/feed flow controller to C-410, cascade set point from LIC-415.	-				
PID-007	**Crude LA MTHF Stripping**					
LIC-0703	Level control in C-510.	6"	50	40 - 60		
PIC-0710	Pressure control loop for the MTHF vacuum system.	150 mmHg	150	150 - 200		

FIC-0720	Steam stripper controller, this flow is a slave to TIC-0724.	-			
LIC-0722	MTHF level control in T-510.	10"	10	10 - 20	
TIC-0724	MTHF stripper bottom's temperature control,				
	Provides cascaded set point to LIC-0720.	250 F	200	180 - 220	
FIC-0746	Steam flow controller to HE-510, cacaded flow control from FIC-0702	-	Computer controlled		
PID 008	**Finishing (Air stripping and wiped film evaporation)**				
LIC-0860	Level control on T-550, cascades flow set point to FIC-0861.	10"	10	2 - 20	
FIC-0861	Crude LVAC flow control loop FIC-0861				
	Receives cacade set point changes from LIC-0860.	0.15 gpm	Computer controlled		
TIC-0862	Air stripper inlet air temperature	300 F	300	None.	
	Chiller temperature setpoint	60F	60	55 - 70	Set in field at chiller
UFD 071	**Collection & Waste Neutralization Systems**				
AIC-7102	First stage pH neutralization.	6.5	6.5	5.5 - 7.5	
AIC-7103	Second stage pH neutralization.	7	7	7.0 - 8.0	
	Effluent tank at start of run (circle).	T-7132	T-7133		

August 30, 1998

BIOMETICS INC.

MUNICIPAL SOLID WASTE CONVERSION PROJECT

FINAL REPORT

CONTRACT No: 4204-ERTER-ER-96

Abstract: The Biofine demonstration plant at South Glens Falls was operated using a cellulose-rich feedstock derived from municipal waste under a program funded by New York State Energy Research and Development Authority. Plant equipment modifications and operational conditions were found to allow a substantial portion of the feedstock to be continuously processed over an extended period to produce levulinic acid. Process feedrates as high as 170 pounds per hour (4080 pounds per day) were achieved. Yields of levulinic acid were in the range 30 pounds of levulinic acid per 100 pounds of cellulose fed. The quality of the final product was high (95+%) being comparable to that obtained from paper sludge. Overall, the experiment was successful indicating that with certain equipment and operational modifications the use of MSW as a feedstock for the process is feasible.

1.0 Introduction and Background

The purpose of this experiment was to test the feasibility of using the cellulose-rich portion of municipal solid waste as a feedstock for the Biofine process. The Biofine demonstration plant at S. Glens Falls, NY is primarily designed to use papermill waste sludge as its feedstock. It employs the Biofine process technology to convert the cellulose fraction of the waste sludge to levulinic acid in high yield (1,2). A diagram of the Biofine process is provided on the next page.

The organic fraction of municipal solid waste (MSW) is the portion of raw municipal waste remaining after separation of the plastics, synthetic fibers, metals, inorganic aggregates and glass components. It is a cellulose-rich material containing between 45% and 70% cellulose. Unlike the other separated components, this material cannot usually be recycled and must be either incinerated or land-filled. Incineration or land-filling results in the degredation of this fraction to greenhouse gasses such as carbon dioxide and methane. It is estimated that in the U.S. alone degredation of municipal garbage in incinerators or landfills adds some 200 million tons per year of greenhouse gas to the atmosphere. In addition, it is estimated that the available energy from this organic fraction of MSW is around 2 Quads (or 2 quadrillion BTU) per year. An environmentally sound technology which would allow conversion of this resource to useful chemicals and fuels would be highly desirable if not essential to society's future well-being. If proven to be capable of processing MSW-derived feedstock, this technology has the potential to make an important contribution to society both in the U.S. and in New York State.

The overall conclusion of the project is that with certain operational and engineering modifications, the MSW derived fiber fraction is a desirable feedstock for the Biofine process.

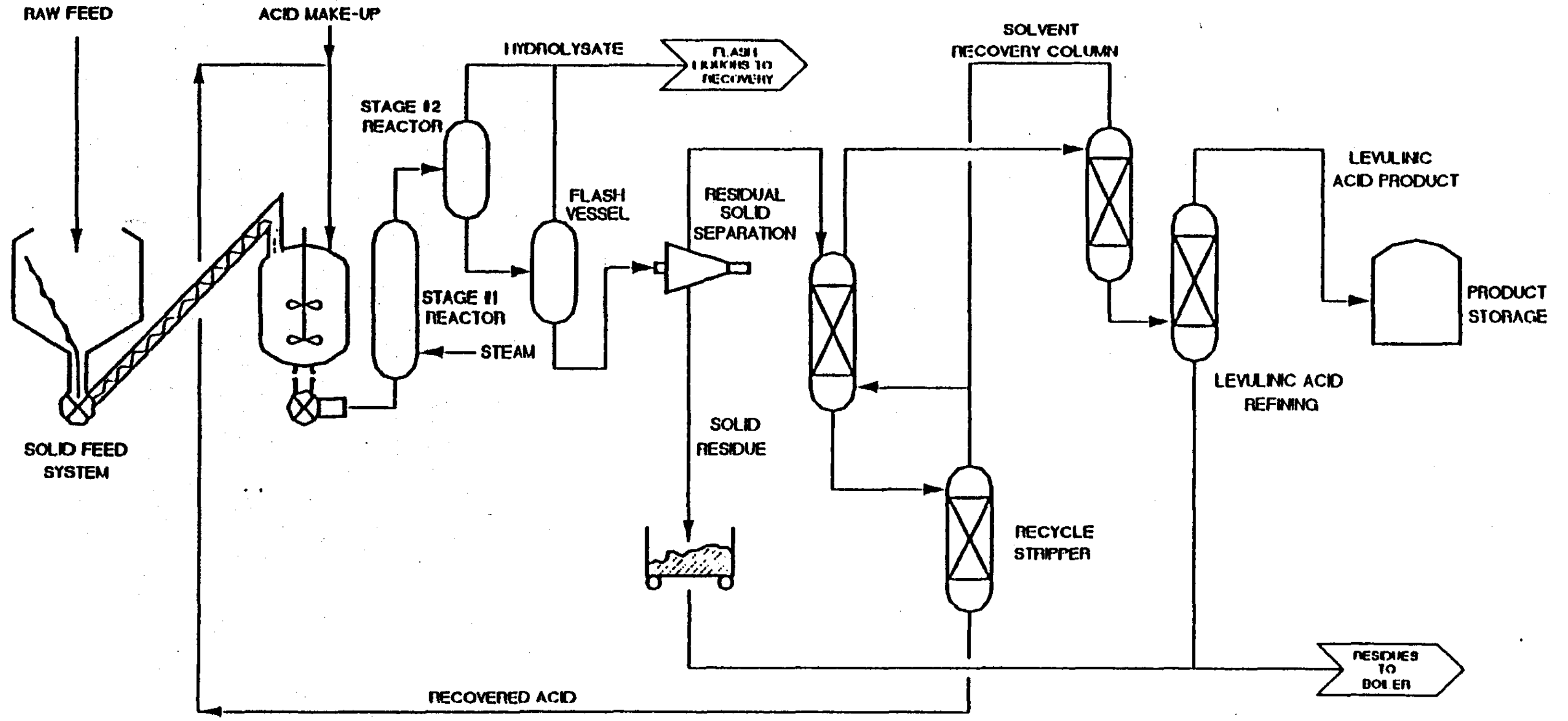
Attempts to operate the plant lasted over a period of four months. Initially, operations were unsuccessful due to the heterogeneous nature of the feedstock and due to the fraction of oversize (greater than two inch) particles present. Once the larger particles were removed, operations were perfected to the point where high rates could be fed to the process continuously for extended operations. It is evident from the results that MSW-derived fiber is a desirable feedstock for the Biofine process.

In the first part of the project, two loads of New York City (Bronx) residential MSW were positively sorted (i.e. the cellulose-rich material was taken out of the raw refuse). The cellulose-rich material was then dried to around 15% moisture, shredded and delivered to Biofine's plant. MSW arrived on plant on Feb 28th and was stored in stable condition for over two months prior to processing. The quantity of feedstock received was approximately 10 tons.

The analysis of the MSW-derived fiber fraction received was measured by BioMetics as follows:

- moisture content - 12%,
- ash - 16%, (titratable alkalinity 6%) (based on dry weight)
- average cellulose - 49%. (based on dry weight)

A copy of the previous report is included in Appendix 1 for reference.


RAW FEED
ACID MAKE-UP
HYDROLYSATE
FLASH LIQUIDS TO RECOVERY
SOLVENT RECOVERY COLUMN
STAGE #2 REACTOR
FLASH VESSEL
RESIDUAL SOLID SEPARATION
LEVULINIC ACID PRODUCT
STAGE #1 REACTOR
STEAM
PRODUCT STORAGE
LEVULINIC ACID REFINING
SOLID FEED SYSTEM
SOLID RESIDUE
RECYCLE STRIPPER
RESIDUES TO BOILER
RECOVERED ACID

BIOFINE

2.0 Summary of Results and Operational Experience

The first attempt to process the MSW-derived feedstock commenced in May 1998. Short periods of stable operation (up to one hour) were achieved but process stoppages were frequent preventing achievement of steady state operation. The main causes of stoppage were found to be oversize (greater than two inch) pieces of feedstock which had escaped separation during the preparation phase at other locations (ref report on Tasks 3 thru 7, dated March 1, 1998). These oversize particles consisted of non-hydrolysable materials such as metal and plastic pieces. Occasionally, large particles of non-hydrolysable wood were also found but it was concluded that this only occurred as a consequence of poor reactor control rather than a primary cause. Pieces of feedstock responsible for blockage were successfully pumped into the reactor system by the feed pump P-101, but would become lodged in the process control valves particularly the pressure control valve on the plug flow reactor, R-101. The maximum opening of this valve is approximately 0.75 inch. Additionally, inspection of the feed pump P-101 indicated that there was a high degree of wear occurring on the viton stator due to feeding of this material. Approximately 1 ton of feed material was put through the system during this initial attempt.

It was decided to carry out a re-sorting of the material to separate oversize pieces. The resorting was carried out manually by means of a two inch aperture mesh screen. The re-sorting operation lasted three weeks and resulted in the rejection of around 3 tons of feed material.

The plant was restarted on the MSW feed for a second operational campaign in July. Longer periods of 1 to 2 hours un-interrupted operation were achieved between blockages. During this second campaign blockages were confined to control valves downstream of the first stage reactor. The pressure control valve for R-101 remained free of blockage although pressure control and, consequently, reactor temperature were erratic. It was concluded from this that the larger particles removed during the re-sorting operation were the cause of earlier R-101 pressure control valve blockages. The nature of the particles causing blockage were mainly metal, glass and small rocks with some solid plastic pieces. No plastic film was found in the sample inspected.

Blockages during this second campaign were experienced primarily in the smaller control valves controlling pressure in R-102, level in HE-101, level in T-102 and level in T-201. These valves have maximum trim openings of 0.25 inch or less. In addition, fairly frequent blockages were found in the bottom outlet from T-102 (the system flash cooler). Approximately two tons of feedstock were used up during this campaign.

Based on the findings and experiences of this second campaign, several modifications were made to the plant to improve operations. These were as follows:

- The "variable aperture" ball in the pressure control valve on R-101 was replaced with a "full bore" ball.
- "Blowdown" drains and back-flush piping was installed around the control valves which were most frequently blocked.
- A strainer was installed at the base of T-102 to separate any large solids from

the bottom outlet.

- The controller for level control in HE-101 was tuned to give a high gain, high reset rate "Bang-Bang" operational characteristic to allow any lodged particles to be freed quickly whilst maintaining reasonable control.

The plant was restarted for the third campaign in early August 1998. Initial plant operation was continuous but erratic due to poor pressure control in R-102. (The burst disc on R-102 ruptured several times).

Analysis of plant operating data indicated strongly that periods of unsteady operation were correlated with initial loss of pressure in R-102 and sudden increases in condensate quantity from R-102. From this it was concluded that the feedstock material had a high tendency to foam, especially if allowed to boil due to sudden pressure reduction from saturated conditions. Small solid particles were being carried over into the condenser with the foam and were becoming lodged in the R-102 vent pressure control valve. This resulted in very large pressure losses as the valve opened and lodged particles were suddenly freed. Particles responsible for blockage of the small control valves were mainly small rocks, metal shavings, ceramics and solid plastics.

If the foaming tendency could be reduced then stable plant operation appeared possible. An anti-foam agent was identified which showed some promise in laboratory experiments. It was found during subsequent operation that this agent also had the additional benefit of assisting in the wetting, blending and pumping of the feedstock in the process.

Inspection of R-101 confirmed that a high degree of wear was continuing to occur on the viton stator. Pump performance had fallen to the point where solids had to be present in the feed for the pump to achieve the required reactor pressure. Although this only presented a problem on start-up where it is desirable to reach reactor conditions using water only, it was indicative of the high degree of pump wear.

Approximately one ton of feedstock was used during this third campaign.

The plant was restarted for the fourth campaign in late August. With addition of the anti-foam agent steady operation was achieved. R-102 pressure control became reasonably stable. Blockages of control valves and T-102 became of low enough frequency that operator intervention could maintain the plant in operation with a minor effect on operating conditions. Typical particulates found in the valve-clearance blow-down streams consisted of metal and small rocks and ceramics. Very few plastics were found and no plastic film was found.

Sustained plant feed-rates were achieved as high as 170 pounds per hour. A typical operational day's results is shown on the table in this section. Approximately 2700 liters of hydrolysate were obtained. The overall yield was measured at 0.3 pounds of levulinic acid per pound of cellulose. Hydrolysate was collected for processing and purification in the back-end of the process. Approximately one ton of feedstock material was used during this campaign.

It was decided to halt operations after demonstration of success due to fears concerning the wear on

P-101. At the time of cessation of the trial P-101 was barely capable of maintaining R-101 at process pressure. Discussions with the pump manufacturer, Robbins and Myers, (Allentown, PA) raised the possibility of replacing viton with a harder wearing elastomer, however schedule and cost precluded this at the time.

The hydrolysate material produced in the hydrolysis reaction was then processed through the extraction and purification steps. The hydrolysate processed well with good recovery (94%). No difference was detected between the performance of this part of the process on MSW-derived hydrolysate compared with paper sludge derived hydrolysate. The purity of the finished product was high (95+%). Again, no significant difference in final product purity was found for the product from MSW-derived feedstock when compared with product from paper sludge. The purified material will be shipped to Pacific Northwest National Laboratories (PNNL) for further testing.

Date. 8/26/98
Run ID: r082698a

LVAC pumped to drums as hydrolyzate

			triplicates of concentration			average*	levulinic	
drum id	gallons	liters	gm/l	gm/l	gm/l	gm/l	kg	lb
001	43.33	164.00	12.07	11.94	11.64	11.88	1.95	4.29
002	40.00	151.40	26.84	26.53	26.04	26.47	4.01	8.82
003	49.00	151.40	26.62	26.16	24.65	25.81	3.91	8.60
004	46.00	177.90	25.30	24.59	24.66	24.85	4.42	9.72
005	86.70	338.91	24.70	22.56	24.81	24.02	3.34	7.34
006	55.00	208.18	18.95	19.50	19.80	19.42	4.04	8.89
007	52.00	196.82	20.39	19.89	19.86	20.05	3.95	8.68
008	22.70	85.92	21.07	20.56	20.31	20.65	1.77	3.90
009	45.00	170.33	20.45	19.50	19.65	19.87	3.38	7.44
010	40.00	151.40	22.21	21.62	21.83	21.89	3.31	7.29
011	45.00	56.78	21.86	21.29	21.32	21.49	1.22	2.68
Totals	436.73	1653.02					35.30	77.65

*average concentration
during duration of run
21.35 gm/l

LVAC exiting via T201

			concentration	levulinic	
	gallons	liters	gm/l	kg	lb
startup	132.00	499.62	4.20	2.10	4.62
shutdown	297.00	1124.15	2.60	2.92	6.43
Totals	429.00	1623.77		5.02	11.05

Feed information

lbs refuse derived feed stock (rdf) fed	656.00 lbs
percent moisture	12.00 %
percent cellulose	49.00 % (dry weight basis)
lbs cellulose fed	282.87 lbs

Yield

Overall yield 0.34 lbs levulinic/lb cellulose

0 Conclusions

The project resulted in the successful sustained operation of the Biofine demonstration plant using MSW-derived cellulose as feedstock. The operational experience gained and plant modifications resulted in achieving continuous operation and steady state conditions. The technical lessons learned on this project will be key to allowing a large scale plant to be designed, constructed and operated to allow processing of the celluose-rich fiber fraction of MSW. Such large scale plants would be capable of processing 1000 to 2000 dry tons per day of MSW-derived fiber.

The main conclusions of the project are as follows:

- Although the plant was designed to operate on paper sludge it has been successfully operated on a more heterogeneous MSW-derived feedstock. All operational difficulties encountered can be mitigated by engineering or procedural modifications.

- The blockage problems initially encountered with operation on the MSW-derived feedstock were due primarily to the small scale of the plant equipment. Most of these problems would not be encountered on a large scale (500 to 1000 dry ton per day) plant due to the much larger control valve and pipe sizes. On a large plant, typical pipe size will be 3 to 6 inch and control valve trims will be 2 inch to 4 inch. On the Glens Falls plant typical pipe sizes are 0.5 to 1 inch and control valve size 0.25 to 0.75 inch.

- It was evident from inspection of the material causing the blockages that it was primarily metal and rock. Although some plastic material was found this was mainly large solid chunks. Plastic film did not appear to have any effect on operations. It is speculated that plastic materials are softened enough at the temperature of operation of the reactor to extrude through most of the control valves. In a larger MSW recycling operation metal, large plastic material and rocks would be more thoroughly removed. Commercially available technology exists to accomplish this.

- The plant modifications made to allow smooth operation can all be incorporated permanently into the demonstration plant if further operation on MSW-derived feedstock is warranted. These modifications can all be implemented on a large scale plant and can be automated to reduce the need for continual operator intervention.

- Certain critical points in the process were identified as places where non-hydrolysable solids collected. In a future plant provision would be made to purge these points in the process.

- The anti-foam agent used is readily available and can be used at low levels (200 to 400 ppm).

- The yield of levulinic acid obtained was 0.31 pounds per pound of cellulose. This is somewhat lower than the yield range of 0.5 to 0.6 pounds per pound obtained from paper sludge. This explained by several factors: Firstly, The relatively short duration of stable operation on MSW compared to the continuous days of operation obtained on paper sludge. This results in the low yield "start-up and shut-down" material having a large effect on the results; secondly, the reactor operating conditions were more variable using the more heterogeneous MSW when compared with the more homogeneous paper sludge feedstock. As noted previously, at the end of the operation P-101 was barely capable of maintaining pressure in R-101. This affects the reactor temperature which if variable results in decreased yield. The yield on the MSW-derived feedstock would be expected to approach that obtained from paper sludge in longer operational campaigns.

- In any future MSW-based operations at S. Glens Falls the feed pump P-101 would be modified to include a harder wearing elastomeric stator. In the large scale plant a different type of pump from the Moyno would be employed. This would be a Schwing or Putzmeister reciprocating pump. These pumps are not available at the small scale of operation of the demonstration plant. The remaining MSW at the S. Glens Falls plant will be used to carry out pump trials using these pumps.

- The hydrolysate containing the dilute levulinic acid exhibited good, predictable processing characteristics. It was extracted and purified identically to the hydrolysate produced from paper sludge. The recovery of levulinic acid was acceptable at 94% and the purity of the product was similar to that found for paper sludge feedstocks. (95+%)

4.0 BIBLIOGRAPHY

1. U.S. Patent 5,608,105 (3/4/97) - S.W. Fitzpatrick, - Assigned to Biofine Inc.
2. U.S. Patent 4,897,497 (1/30/90) - S.W. Fitzpatrick, - Assigned to Biofine Inc.