9–2 Environmental Protection Countermeasures for a Citric Acid Plant

9–2–1 Kinds of Wastes Discharged from a Citric Acid Plant

Shown in Figure 9–1 are the kinds of wastes and the amounts discharged from a plant producing citric acid. The kinds of wastes, and the corresponding quantities, properties, and countermeasures are shown in Table 9–4.

Table 9–4 Kinds, Quantities and Properties of Wastes from a Citric Acid Plant

Kinds of Waste	Amount Discharged	Properties	Countermeasures
<< Air Pollution >>			
Boiler exhaust gas	3,668–6,927 Nm^3/h	Dust Concentration: 17.1g/Nm^3	Dust collector
<< Water Pollution >>			
Waste water	135 m^3/d	pH : 6–9 BOD : 10,000 mg/ℓ COD : 16,000 mg/ℓ SS :200–300 mg/ℓ	Biologically treated & discharged to a sewer
Floor drains and cleaning water	100 m^3/d	pH : 6–9 BOD : 500 mg/ℓ SS : 20 mg/ℓ	Discharged to a sewer
Waste water from the cooling tower	900 m^3/d	pH : 6–9 BOD : 15 mg/ℓ SS : 10 mg/ℓ	265m^3/day discharged to the lagoon
Domestic waste water from plant employees	0.2 m^3/d/head	pH : 6–9 BOD : 200 mg/ℓ SS : 200 mg/ℓ	Discharged to a sewer
Domestic waste water from company residences	0.2 m^3/d/head	pH : 6–9 BOD : 200 mg/ℓ SS : 200 mg/ℓ	Discharged to a sewer
<< Waste >>			
Waste mycelium	9 t/d	Water cont. : 80%	Compost
Gypsum	17 t/d	Water cont. : 25%	Marketable by-product
Waste carbon	513 kg/d	Water cont. : 65%	Incineration
Coal ash	2.0–3.2 t/d	Water cont. : 20%	Marketable by-product
Excess sludge	45 m^3/d	Water cont. : 99%	Marketable as fertilizer
Dust removed from boiler exhaust gas	1.2–2.3	Water cont. : 5%	Marketable by-product
Incineration ash	7–2 kg/d	Water cont. : 5%	Marketable by-product

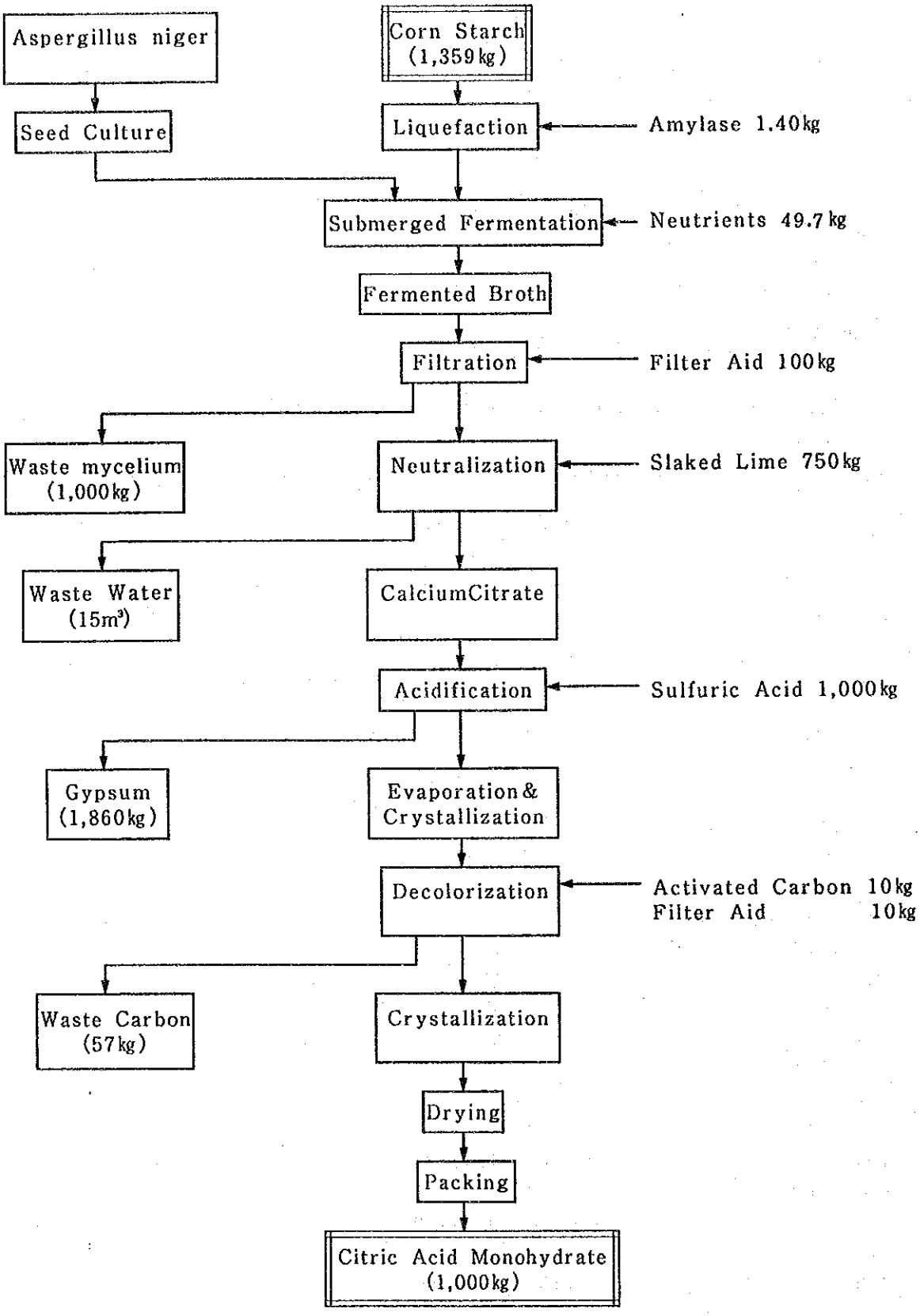


Figure 9–1 Kinds of Wastes from Citric Acid Plant

9–2–2 Atmospheric Pollution Control Countermeasures

In designing atmospheric pollution control countermeasures, it is necessary to analyse the quantity and quality of pollutants and to introduce protection measures which satisfy the relevant laws and regulations.

The proposed citric acid plant has a boiler for producing the steam used for crystallizing and drying the product. Since there is a shortage of petroleum products in Zimbabwe, a coal fired boiler will be used. Therefore, removal of fly ash and soot from the boiler flue gasses will be important. Specification of the coal fired boiler is assumed to be as follows:

- Combustion system : stoker type
- Evaporation rate : average 4 tons per hour, maximum 8 tons per hour
- Coal consumption : average 450 kg per hour, maximum 850 kg per hour

Domestic coal from Wankie will be used as boiler fuel. Typical analysis of Wankie coal is shown in Table 9–5.

Table 9–5 Coal Specifications in Zimbabwe

	Dry	Washed	For Cooking
1. Typical Coal Specification, %			
Inherent moisture	1.0 – 1.5	1.0 – 1.5	1.0 – 2.0
Ash	12.5 – 15.0	11.0 – 14.0	9.0 – 11.0
Volatile matter	22.0 – 25.0	23.0 – 26.0	25.5 – 27.5
Fixed carbon	61.0 – 62.0	61.0 – 62.0	62.0 – 63.0
Calorific value, MJ/kg	28.0 – 29.0	29.0 – 31.0	30.0 – 32.0
Sulphur	2.5 – 2.8	2.0 – 2.5	1.25– 1.55
Phosphorous			
2. Maximum Moisture, % (as Despatched)	7	7	7
3. Ash Fusion Temperature, °C	1,250	1,270	1,270

4. Typical Ash Analysis, %					
Silica	38.2	Alumina	33.2	Iron Oxide	10.8
Calcium Oxide	11.4	Sulphate	2.2	Alkalies	2.0
Magnesium Oxide	0.4	Titanium Oxide	1.7	Phosphate	0.1

In designing countermeasures for air pollution, it is necessary to thoroughly study the laws and regulations. As stated in the paragraph concerning the environmental protection act, there are no regulations for dust emitted from a boiler. However, when a plant is constructed, it is necessary to submit an application to register the process with the Ministry of Health and to have detailed discussions to get a construction permission. Judging from interviews conducted in Harare, during the field survey, regarding air pollution control measures for existing boilers and in view of the anticipated quality and quantity of dust in the exhaust gas, it is considered that permission for construction will be granted if the dust removal ratio exceeds 90% and the height of the chimney is 20 meters.

In order to study dust control, the characteristics of the dust were reviewed. The size distribution of particles resulting from burning domestic coal is shown in Table 9–6. 5.15% of the particles are less than 2.5 μm and 20% exceed 52.5 μm.

Table 9–6 Dust Particle Distribution

Type of Dust		:	Ash
Density (kg/m^3)		:	2,300
Bulk density (kg/m^3)		:	1,200
Particle size (μm)			Distribution (%)
less than	2.5		5.15
	5.0		11.49
	10.0		10.93
	15.0		9.94
	20.0		8.86
	25.0		7.79
	30.0		6.79
	35.0		5.88
	40.0		5.06
	45.0		4.34
	50.0		3.71
over	52.5		20.06
	Total		100.00

A cyclone removes dust efficiently at low cost and cyclones are widely used in Zimbabwe. The design conditions and specification of a suitable cyclone are shown in Table 9–7 and 9–8, respectively.

Table 9–7 Design Conditions for the Cyclone

Type of gas	Flue gas
Flow rate, Nm^3/h	3,668–6,927
Gas temperature, °C	190
Absolute pressure, mmH2O	10,330
Kind of dust	Ash
Concentration of dust, g/Nm^3	17.1
Density of dust, kg/m^3	2,300

Table 9–8 Specification of the Cyclone

Type of cyclone	Multi cyclone
Number of unit-cyclones	9
Unit-cyclone capacity, m^3/min	21.8
Pressure drop, mm H2O	105
kPa	1.03
Cut-off size of particles, μm	2.7
Cyclone dimensions	
Diameter of cyclones, mm	616
Height, mm	1,848
Material of cyclone	Carbon steel
Material thickness, mm	3.2
Weight, kg	112.6
Housing weight, kg	2,685
Dust bunker	
Type	Hopper
Dimension, mm	2,148 × 2,148
Height, mm	1,787
Volume, m^3	3.32
Period of dust discharge, hrs	3
Height of discharge point, mm	1,000
Thickness of bunker material, mm	4.5
Weight of bunker, kg	1,007
Cyclone support weight, kg	730
Total height, mm	6,135
Total weight, kg	5,435

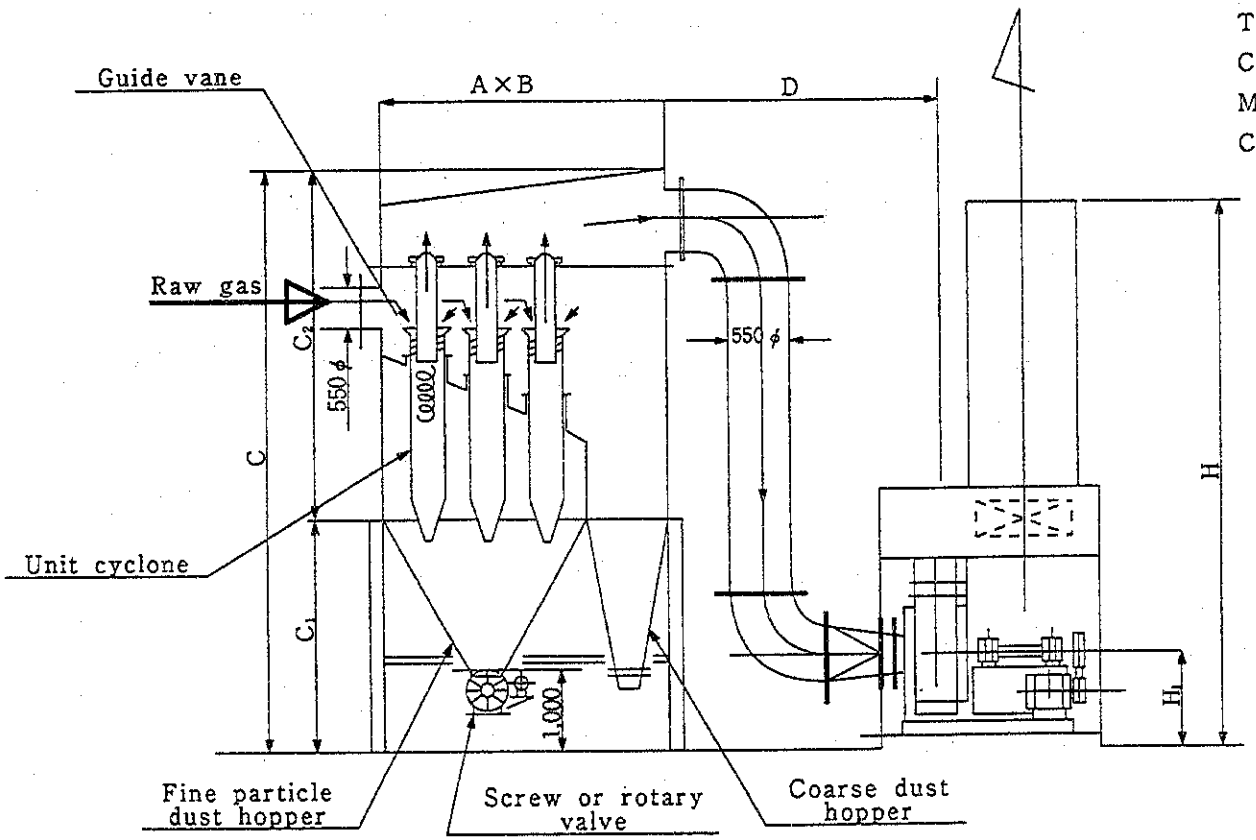
The cyclone characteristics are described below. Partial collection efficiencies and total collection efficiencies for particles to be removed by the specified cyclone are shown in Table 9–9. According to this table, the collection efficiency for dust particles exceeding 10 μm is over 99% and the overall total collection efficiency is 91.34%. Highly efficient removal of particulates can be expected.

Table 9–9 Cyclone Characteristics

Particle size (μm)		Partial collection efficiency (%)	Dust distribution (%)	Total collection efficiency (%)
2.5	less		5.15	
5.0		74.47	11.49	8.58
10.0		94.64	10.93	10.37
15.0		98.97	9.94	9.86
20.0		99.81	8.86	8.86
25.0		99.97	7.79	7.81
30.0		99.99	6.79	6.81
35.0		100.00	5.88	5.89
40.0		100.00	5.06	5.07
45.0		100.00	4.34	4.35
50.0		100.00	3.71	3.71
52.5	over	100.00	20.06	20.03
Total			100.00	91.34

The construction of the cyclone is shown in Figure 9–2. Exhaust gas from the boiler enters the multi-cyclone and dust is removed by the cyclones. Then the gas is exhausted to atmosphere through the chimney.

The space required for the dust collector is shown in Figure 9–3.



Specifications of Dust collector

Type : Multi-cyclone

Treatment gas quantity : 6,927m³/h

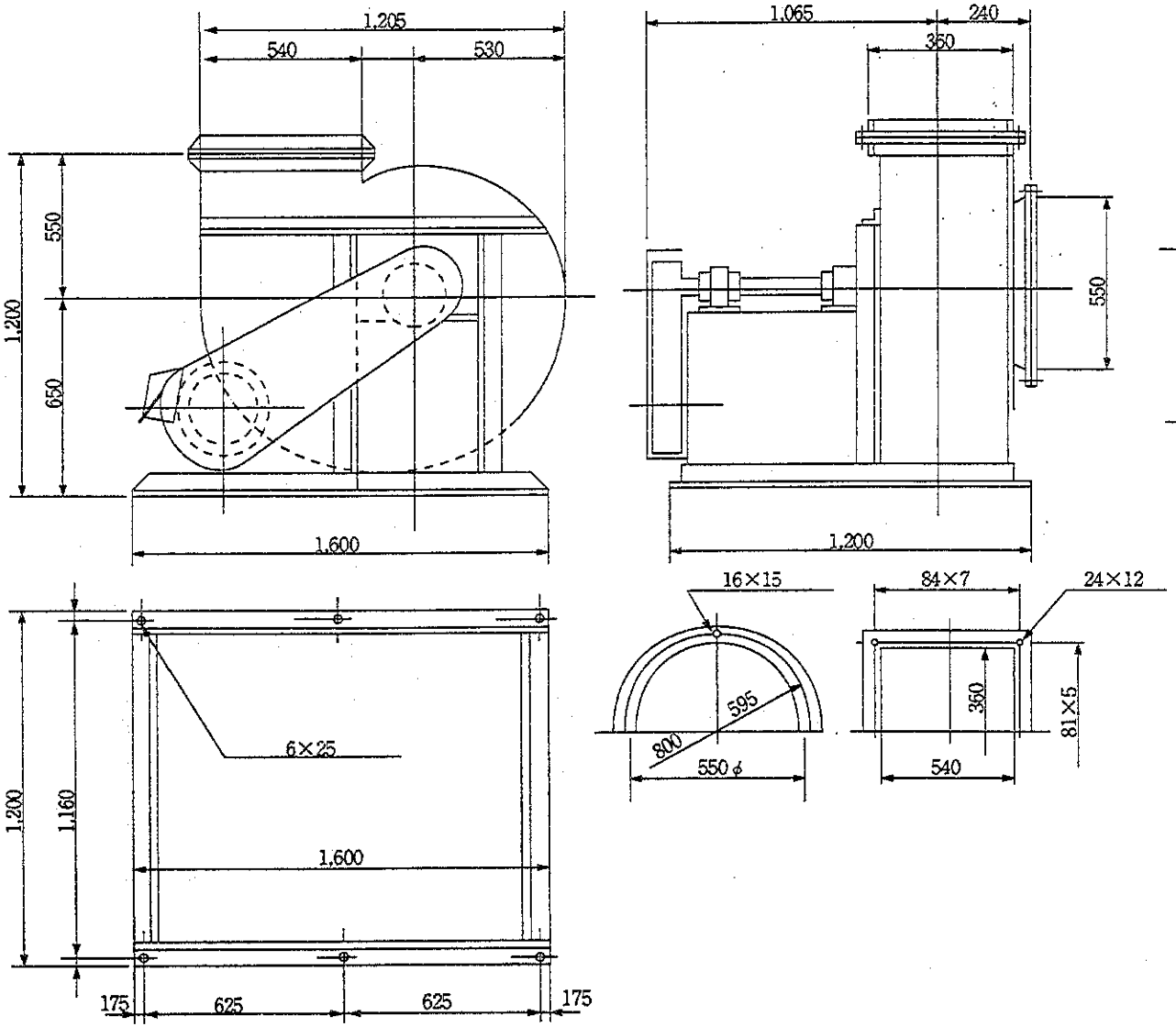
Collector pressure loss : 105mmH₂O

Motor output : 18.5kW

Collector dimensions

Symbol	Dimension
C	6,135
C_1	3,587
C_2	2,548
A×B	2,148×2,648
D	2,250
H	20,000
H_1	800

Figure 9–2–1 Structure Figure of Cyclone (1): Multicyclone



Exhaust fan	Airflow	200m³/mix
	Air pressure	300mmH₂O
	Gas	200℃ air
	r.p.m	1,950 r.p.m
	Drive system	Belt drive
Motor	Output	18.5kw
	Pole No.	4 P
	Voltage	3φ200V60Hz
	r.p.m	1,750 r.p.m

Figure 9–2–2 Structure Figure of Cyclone (2): Exhaust Fan

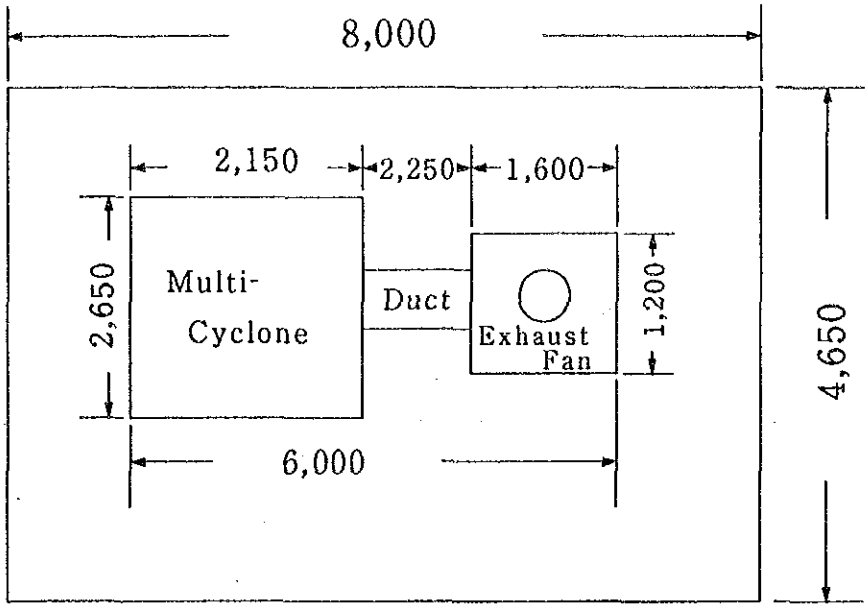


Figure 9–3 Installation Area

9–2–3 Water Pollution Control

(1) Pre-study items

(a) Regulations

Waste water from the citric acid plant will be discharged to the sewerage system of Harare city after treatment. Such waste water has a high BOD (Biochemical Oxygen Demand) and COD (Chemical Oxygen Demand) and a high SS (Suspended Solid) concentration, but contains no toxic substances. Harare city has its own regulations for industrial effluent water discharged into the city sewerage system, but there are no specific values for BOD and COD (there is only a lenient SS limit). Hence if the plant is only required to follow these regulations, no specific precautions for water pollution are necessary.

The treated sewage flows into Lake McILwaine through a river. This lake is polluted by domestic waste water, industrial waste water and agricultural chemicals. Due to eutrophication of the lake, water hyacinth grows extensively and covers a wide area of the lake, causing trouble with water intake and passage of boats. The lake is one of the sources of water for Harare city. In order to reduce the burden on the lake, it is necessary to treat waste water before it is discharged into the city sewerage system.

(b) Objectives for removal of pollutants

Based on Harare city regulations (Table 9–2, 9–3) and referring to Japanese regulations for pretreatment prior to discharge to a sewerage system which are shown in Table 9–10, the following requirements can be drawn up for this project.

– pH 6~9 (Harare city)

– BOD < 600 mg/liter (Japan)

– SS < 60 mg/liter (Japan)

Table 9–10 Regulations for an Industrial Pretreatment Facility in Japan

pH	5.0–9.0
BOD (mg/liter)	less than 600
SS (mg/liter)	less than 60
n-Hex. (mg/liter)	
Mineral oil	less than 5
Vegetable oils and fats	less than 30

(2) Basic plan for waste water treatment

Based on the above requirements, the waste water from the citric acid production process and other waste water should be treated as follows:

(a) Waste water from fermentation process containing a high concentration of BOD should be treated by a biological process and discharged to the Harare city sewers.

(b) Floor drains and cleaning waste water can be discharged to a sewer without treatment, because the BOD and SS concentrations are lower than the values in the regulations.

(c) The waste water from the cooling towers can be discharged to the lagoon tank for use as dilution water.

(d) Domestic waste water from factory employees and company residences can be discharged to a sewer without treatment.

(e) Blow-down water from the boiler and water used to wash ash can be discharged to a sewer without treatment.

(f) Excess sludge can be used as an agricultural fertilizer on maize farms.

(3) Conceptual design of the waste water treatment plant

In the conceptual design of a system, it is necessary, as the first step, to study the quality and quantity of raw waste water. The characteristics of waste water from a citric acid plant are that the values of BOD and COD are high but the SS concentration is low. The raw waste water does not contain hazardous materials. The composition of raw waste water is shown in Table 9–11.

Table 9–11 Characteristics of Raw Waste Water

pH	6.0–9.0
BOD (mg/liter)	10,000
COD (mg/liter)	16,000
SS (mg/liter)	200–300
Water quantity (m^3/day)	135

Volume of raw waste water to be treated is as follows:

- Maximum daily rate 400 m^3/day
- Operation time 24 h/day
- BOD quantity to be treated 1,354 kg/day

Target water quality after treatment is shown in Table 9–12.

Table 9–12 Quality of Treated Waste Water

pH	6.0–9.0
BOD (mg/liter)	less than 600
SS (mg/liter)	less than 60
Water quantity (m^3/day)	400

Since the BOD concentration is very high at 10,000 mg/liter, the raw waste water should be diluted by water discharged from the cooling tower (265 m^3/day) to give a BOD of 3,385 mg/liter before it is sent to the treatment plant.

The treatment process is as follows:

- Primary treatment : Screening process, to remove particles larger than 0.5 mm
- Secondary treatment : Batch type lagoon process
  BOD volumetric loading of 0.3 kg/m^3•day

There are 3 kinds of biological treatment processes namely; activated sludge process, aerobic digestion process and lagoon process. The improved batch type lagoon process has been adopted for the treatment of waste water for this project. The model of this lagoon process is shown in Figure 9–4.

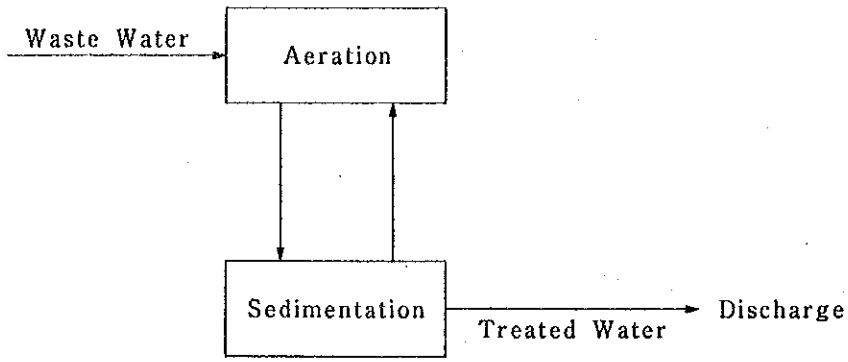


Figure 9–4 Model of Batch Type Lagoon Process

In this process, the waste water is treated in a single activated sludge tank.

Inflow of raw waste water, the aeration process, sedimentation process and discharge of treated water are continuously repeated in this tank.

The special features of this process are as follows:

- The structure is simple and the construction cost is lower than that of other processes.
- The treatment efficiency is higher than for the ordinary lagoon process.
- It is possible to save aeration power by an automatic control system which responds to fluctuations of the inflow. Also a stable performance can be achieved.
- It is possible to mix sludge completely in the lagoon tank by vertical and horizontal aeration. The power required for these mixing operations is lower than that required for the standard activated sludge process.
- Nitration and denitrification can be achieved by varying the aeration by an automatic control method.

This batch type lagoon process has a high efficiencies.

The operating schedule is shown in Figure 9–5. The flow sheet of the lagoon treatment system is shown in Figure 9–6.

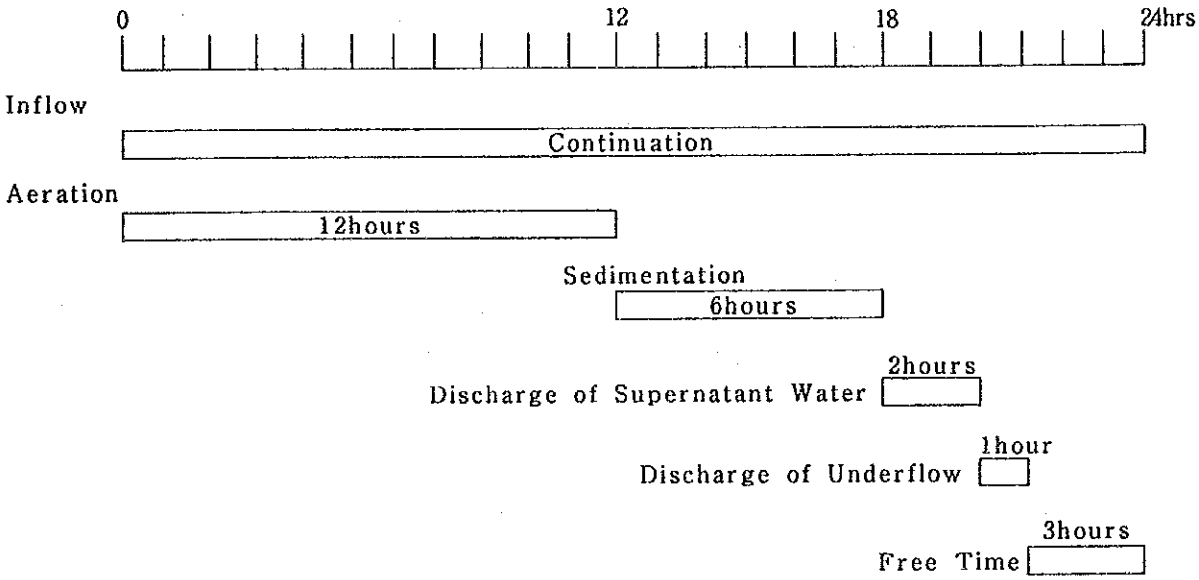


Figure 9–5 Operating Schedule of a Lagoon Tank

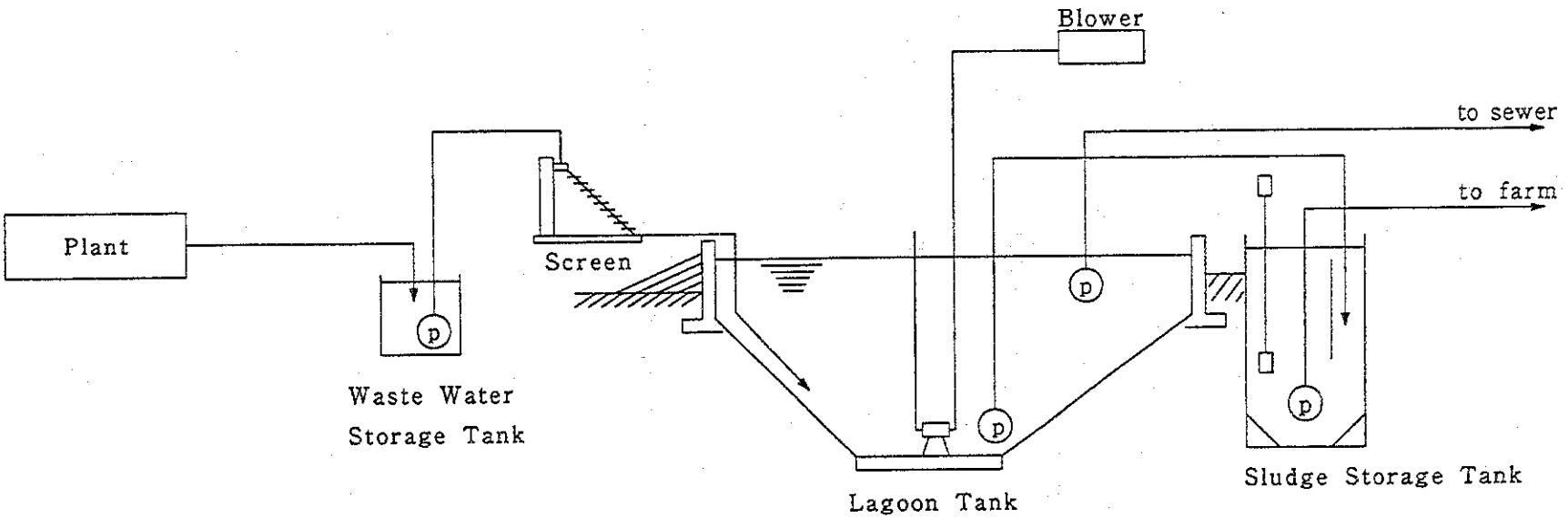
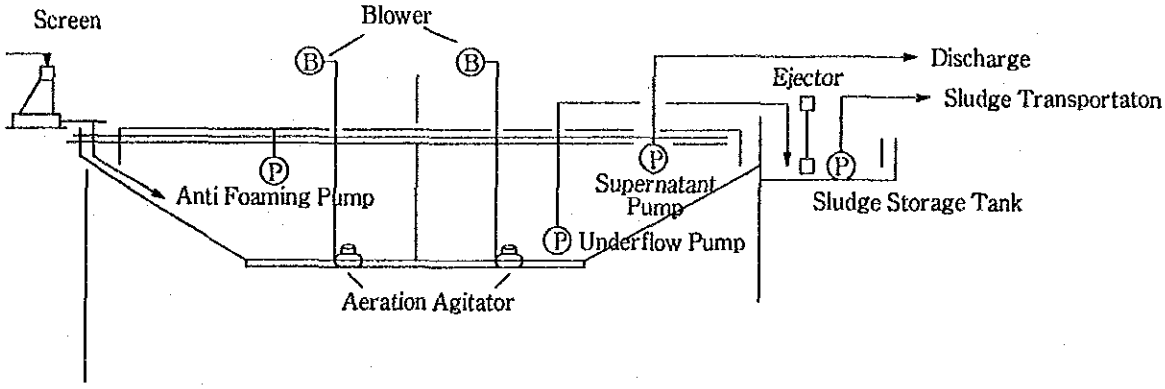


Figure 9–6 Flow Sheet of Batch Type Lagoon System

The cross section and principal dimensions of a lagoon tank are shown in Figure 9–7.

For reference, the general appearance of a typical lagoon with a volume of 4,000 m^3, a diameter of 32 m and depth of 9 m is shown in Picture 9–1.



Supernatant Depth	32 cm	
Undeflow Time	1 h	54 m^3/60 min = 900 ℓ /min
Discharge Time	2 h	400 m^3/120 min = 3.33 m^3/min

Dimensions of Lagoon Tank

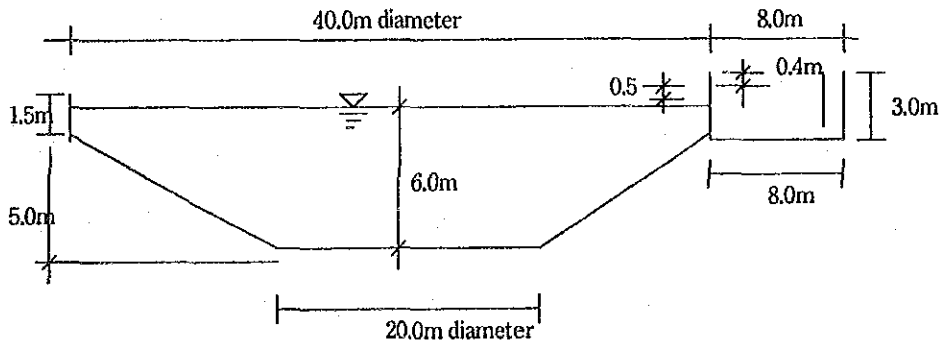
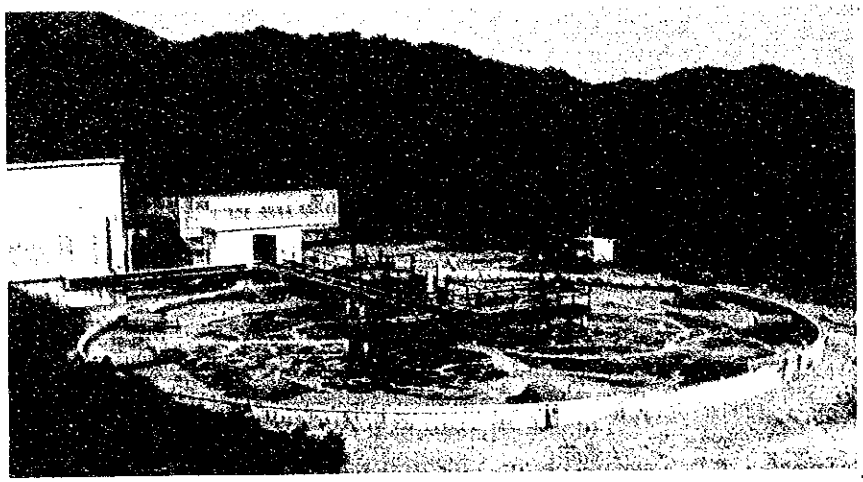


Figure 9–7 Cross Section and Principal Dimensions of a Lagoon Tank



Lagoon Capacity	:	4,000 m^3
Dia. of Lagoon Tank	:	32 m
Depth of Lagoon Tank	:	9 m

Picture 9–1 Appearance of Batch Type Lagoon (Actual Example)

(4) Specifications of the facilities

(a) Water Tank

The water tank will be as follows:

– Structure : Reinforced concrete structure covered with asphalt.

Control bridge to be an I beam steel structure.

– Finishing : Inside to have a water proof lining over the concrete structure.

Outside unfinished concrete.

The specifications of the various kinds of water tanks are given in Table 9–13.

Table 9–13 List of Waste Water Tanks

Name	Quantity	Specification
Lagoon tank	1	Effective capacity: 4,919 m^3
		Dimensions
		Diameter : 40 m
		Bottom Diameter : 20 m
		Retaining wall height : 1.5m
		Depth of conical section : 5 m
		Water Depth : 6 m
		Construction
		Retaining wall : Reinforced concrete
		Inclined surface : Asphalt layer (4 mm)
Sludge storage tank	1	Effective capacity : 160 m^3
		Dimensions : 8 m × 8 m × 3 m
		(effective depth 2.5 m)
Waste water storage tank	1	Effective capacity : 25 m^3
		Dimensions : 3 m × 3 m × 3.5 m
		(effective depth 2.8 m)

Sludge quantity is assumed to be about 40% of BOD removal and the quantity will be as follows:

400 m^3/d × (3,385 mg/liter–600 mg/liter) × 0.4

= 400 m^3/d × (3,385 kg/m^3–0.600 kg/m^3) × 0.4

= 445.6 kg/day

If the under flow concentration is assumed to be 10,000 mg/liter (10 kg/m^3), the amount of sludge discharged will be as follows:

445.6 kg/day ÷ 10 kg/m^3 = 45 m^3/day

The quantity of sludge to be stored is equal to 3-days production, thus 45 m^3/day × 3 day = 135 m^3.

(b) Equipment

Kinds and specifications of equipment required for the waste water treatment facility are shown in Table 9–14.

Table 9–14 Equipment List for the Waste Water Treatment Facility

Name	Quantity	Specification
Fine screen	1	Type: Vertical screen, 25 m^3/h, Spacing of screen 0.5 mm Stainless steel
Aeration agitator	4	15 kW Open portion 2,900 mm Size: ϕ 2,120 mm, H 2,390 mm 3,000 kg/each, 14 Nm^3/h
Blower	4	15 kW Size: 1,350 mm (L) × 920 mm (W) × 1,650 mm (H) 776 kg/each, 14 Nm^3/h
Anti foaming pump	2	2.2 kW, 200 V Head 10 m, 0.6 m^3/min, 34 kg
Underflow pump	1	5.5 kW, 200 V, Head 15 m 1.0 m^3/min, 55.5 kg
Supernatant pump	2	5.5 kW, 200 V, Head 15 m 1.0 m^3/min, 55.5 kg
Ejector for sludge agitation	4	2.2 kW, 200 V, 45 $Nm^3/h.m$, 3 mAq 63 m^3/h, 75 kg
Waste water pump	1	5.5 kW, 200 V, H 15 m, 2.5 m^3/min
Sludge transportation pump	2 (spare 1)	Depends on transportation distance
Automatic panel	1	Main & sub switches Over-current relay Over-voltage relay Time switch Instrument inverters: 15 kW × 8 Instrumentation: DO, ORP Recorder: 6 points
Safety fence	150 m	H 0.9 m, around the perimeter of the lagoon tank

(5) Required space

The area required for a batch type lagoon system is indicated in Figure 9–8.

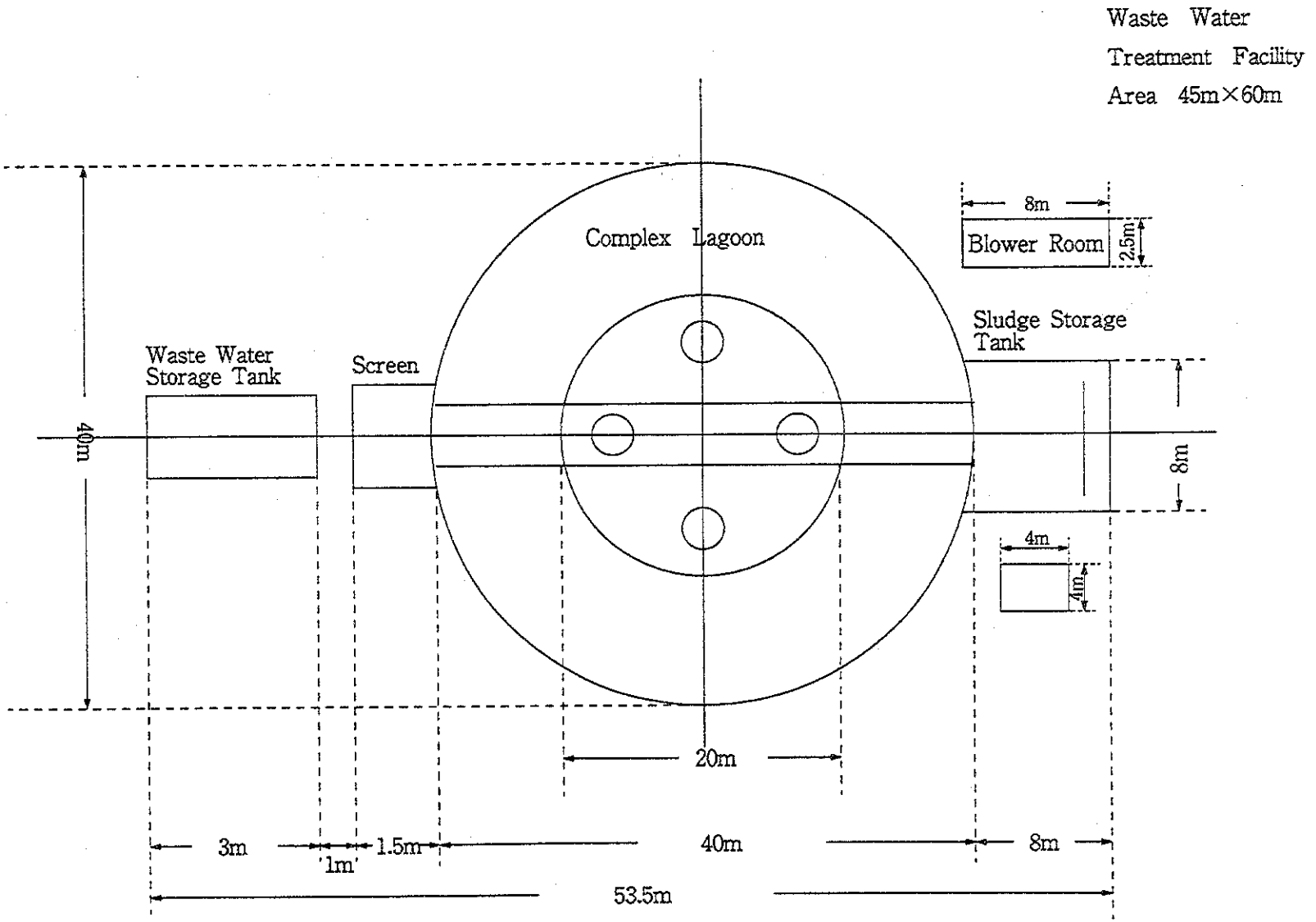


Figure 9–8 Installation Area of Batch Type Lagoon

(6) Utilities

Utilities required for operating a lagoon type waste water treatment plant are as follows:

(a) Electric power

– Incoming power	:	6,000 V AC, 50 Hz, 3 phase
– For motors	:	200 V AC, 50 Hz, 3 phase
– For Instrument & control	:	200 V AC, 50 Hz, single phase
– For Lighting	:	200 V AC, 50 Hz, 3 phase

(b) Water: Industrial water

(7) Treatment of other waste water

All other waste water can be discharged to the sewerage system of Harare city without treatment, because the BOD values are all lower than those specified in the regulations.

This waste water consists of the following:

– Floor drains and cleaning water

– Boiler blowdown water

– Domestic waste water from factory employees

– Domestic waste water from employees' housing

– Waste water from cooling boiler ash

9–2–4 Treatment of Wastes

(1) Basic policy for treatment of wastes

The following wastes are generated by the citric acid plant (please refer to Table 9-4).

– Waste mycelium	:	9 tons per day
– Gypsum	:	17 tons per day
– Waste activated carbon	:	513 kg per day
– Coal ash	:	2.0–3.2 tons per day
– Excess sludge	:	45 m^3 per day
– Boiler flue dust	:	1.2–2.3 tons per day
– Ash from Incinerator	:	7–72 kg per day

As a basic policy, wastes will be recycled whenever possible. Since the amount of discharged wastes is large, an incinerator will be provided for wastes which cannot be recycled.

(2) Recycling of wastes

(a) Waste mycelium

The amount of waste mycelium to be discharged is about 9 tons per day. The water content is as high as 80%. After reducing the water content to 30% or so, the waste mycelium can be used agriculturally as a compost.

(b) Gypsum

The waste which is discharged in the largest quantity is gypsum and the daily amount is about 17 tons. It is a useful by-product and can be used for cement, plaster board, plaster calcined gypsum and for agriculture.

(c) Coal ash

The quantity of coal ash discharged is about 2 to 3.2 tons per day. The coal ash can be recycled for use in making cement, as a fertilizer, and for the strength of foundations.

(d) Boiler flue dust

The amount discharged from the flue dust collector is about 1.2 to 2.3 tons per day. This dust will be reused as a foundation strengthening agent.

(e) Incinerator ash

The preferred use of waste activated sludge is as a fertilizer. If it cannot all be used on local farms, the excess waste activated sludge is to be incinerated. About 3.5 kilograms of incinerated ash is produced by burning the waste activated carbon. If the waste mycelium is incinerated together with the waste activated carbon, the amount of ash discharged will be about 72 kilograms per day. The ash can be used in the same way as coal ash.

(f) Excess sludge

The excess sludge is a well digested sludge and does not smell badly. This sludge is pumped up from the lagoon tank by a submersible pump and can be used as an agricultural fertilizer. The contents of the excess percolate into the ground when it is spread on the surface.

Picture 9–2 shows an example of sludge being spread on a peanut firm.

(a) Sprinkling of sludge

(b) Sprinkling state

(c) Cracks on sludge surface after several hours

Picture 9–2 Sprinkling of Sludge on Peanut Farm (Actual Example)

(3) Incinerator

(a) Specification of the incinerator

The incinerator is designed to burn about 10 tons per day of waste.

– Incinerator capacity : 300 kg/hr 2 sets

– Operation hours : 16 hrs/day

– Operator : 6 men/day

A drawing of the incinerator and its space requirements are shown in Figure 9–9.

The specification of the incinerator is given in Table 9–15.

Table 9–15 Incinerator Specification

Incinerator	Dimensions	: 1,605 mm (H) × 2,240 mm (W) × 1,320 m (L)
	Hopper	: 400 mm × 500 mm
	Stoker surface	: 0.74 m^2
	Primary capacity	: 0.73 m^3
	Total capacity	: 1.13 m^3
	Weight	: 4,500 kg
Chimney	Height	: 8,565 mm
	Outside diameter	: 420 mm
Secondary burner	Output	: 0.25 kW
	Fuel oil	: 4–19 liter/hr
Combustion system	Fire grate, Forced heating system, and Secondary combustion system	
Dust collector	Gravity type	

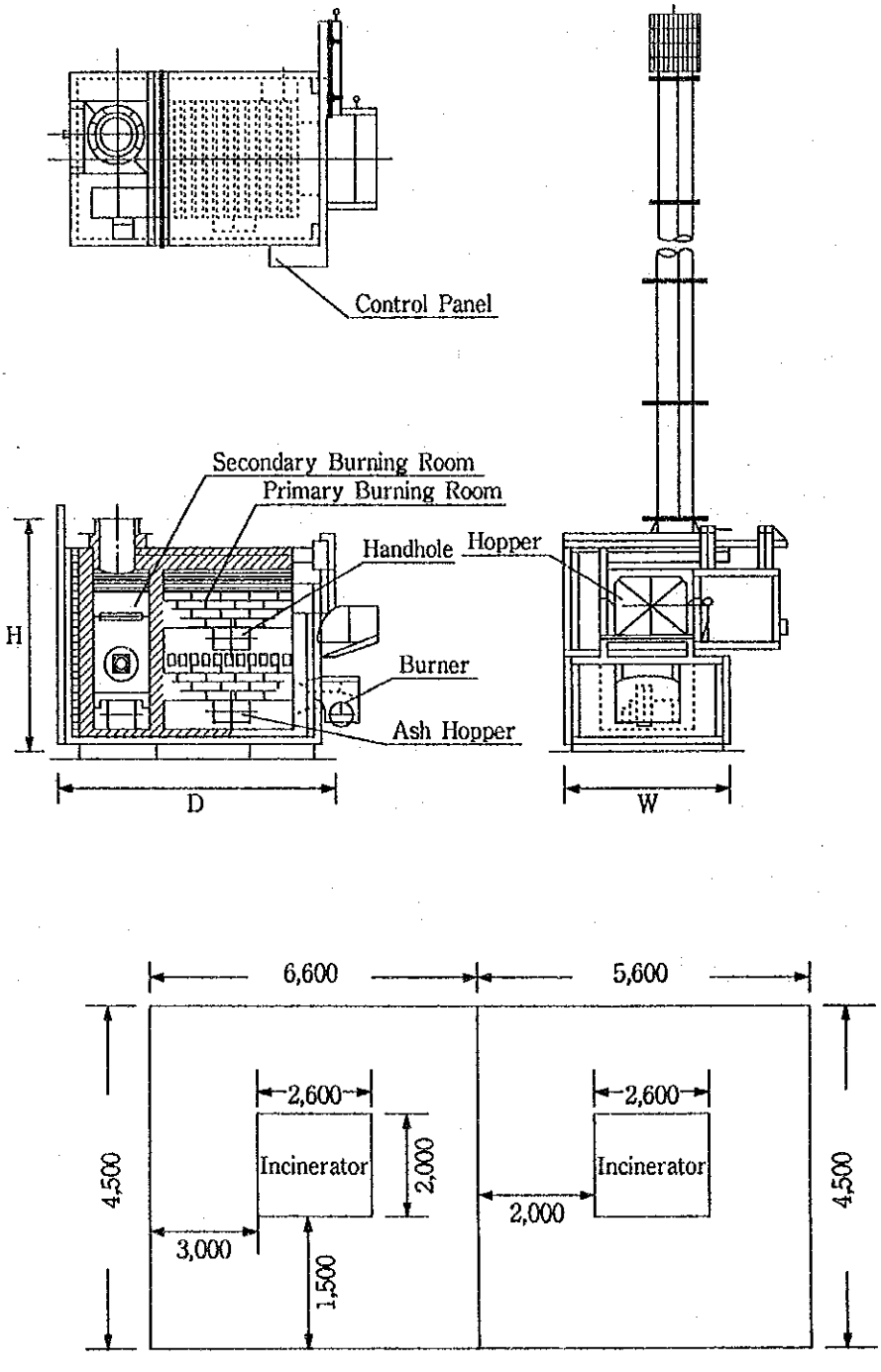


Figure 9–9 Incinerator and Installation Area

Chapter 10 Citric Acid Fermentation Tests

Chapter 10 Citric Acid Fermentation Tests

10-1 Objectives and Methods of the Fermentation Tests

As described in Chapter 4 "Citric Acid and its Manufacturing Technology", citric acid is manufactured by the fermentation method using carbohydrates as raw material. The fermentation processes utilize microorganisms and the combination of fungi, fermentation conditions, and raw materials affects the results of fermentation immensely. All commercialized technologies have been developed by conducting a series of tests to select and improve fungi which are suitable for use with inexpensive and abundant raw materials and by research and development of fermentation conditions appropriate to a specific fungus and raw materials. Appropriate combinations of raw materials and fermentation processes can be derived from experiences to some extent. However, there are a lot of unknowns in fermentation. Therefore, if an existing technology is to be applied to a specific raw material, fermentation tests are inevitably required.

Combinations of raw materials available in Zimbabwe at present or in the near future and technologies commercialized in Japan are as follows:

(1) The submerged culture fermentation process using cornstarch

(2) The solid culture fermentation process using sweet potato starch extraction residues and cassava starch extraction residues

In order to investigate the possibility of commercialization, fermentation tests were conducted by two companies who are actually producing citric acid using the above mentioned raw materials and fermentation processes. Also sugar products obtainable at reasonable prices in Zimbabwe such as crude sugar, concentrated cane juice and cane molasses can be used as the raw materials for citric acid production, although the applicable process is still in the laboratory stage. In order to investigate the possibility of citric acid production using sugar products, fermentation tests were conducted by Waseda University which is developing the above mentioned process.

10-2 Fermentation Tests using Cornstarch

10-2-1 Basic Policy

In this study, the compatibility of fermentation technology (fungi and fermentation conditions) and cornstarch manufactured in Zimbabwe (two brand names of cornstarch; STARCON and STARTEX-45) was reviewed. The test procedure was as follows:

(1) The raw materials were analyzed chemically in order to understand the characteristics of Zimbabwean cornstarch.

(2) By using fungi and fermentation conditions currently used industrially, flask fermentation tests were carried out and the compatibility of raw materials and the fermentation process was reviewed on a preliminary basis.

(3) In view of the results of (1) and (2) above, fermentation conditions were formulated.

(4) Flask fermentation tests were conducted under conditions considered suitable for Zimbabwean cornstarch using the same fungi and the tests were judged to see if the raw materials and fermentation conditions were appropriate.

(5) If the above tests showed that Zimbabwean cornstarch was suitable for the existing fermentation process, a large scale fermentation test using a 30 liter jar fermenter was conducted.

10-2-2 Analysis of Sample Raw Materials

(1) Analysis items and analysis method

Analysis items and methods were as follows:

(a) Outside appearance

The external appearance of the dried cornstarch such as particle size, color and existence of any foreign material was observed.

(b) Water suspension

25 grams of the specimen were mixed in a 100 m ℓ beaker with 75 m ℓ of distilled water which had been cooled down to 25°C after boiling. During mixing and agitation, the degree of cornstarch suspension and the liquid quality were observed.

(c) Electrical conductivity

The conductivity of the liquid suspension contained in the beaker was measured by a conductivity meter.

(d) pH value

After measuring conductivity, the contents of the beaker were tested with a pH meter to measure hydrogen ion concentration.

(e) Supernatant liquid

After measuring the pH value the material was allowed to stand for a certain period of time and the turbidity of the supernatant liquid was observed and compared with the turbidity of a standard specimen.

(f) Water content

5 grams of sample material were heated for 30 minutes at a temperature of 105°C and the reduction in the water content was measured by an infrared moisture meter.

(g) Sugar content

Cornstarch is converted to glucose by hydrolysis and the amount was measured by Somogyi's variation method. The method is as follows:

0.5 grams of sample material is accurately measured and transferred to a 100 m ℓ flask. 50 m ℓ of distilled water and 5 m ℓ of 25 percent hydrochloric acid are added to the flask. A cooling coil is mounted in the flask. The flask is heated for 2.5 hours in a boiling water bath, so that the sample material is completely hydrolyzed. After cooling, the sample material is neutralized by 6N sodium hydroxide using phenolpthalein as the indicator and the contents are transferred to a 250 m ℓ measuring flask and diluted accurately to 250 m ℓ with distilled water. 10 m ℓ pipet full of the diluted sample solution is taken and poured into a 100 m ℓ flask. 20 m ℓ of solution "A", described below, is added, heated and boiled on a hot plate to the extent of slight boiling for a period of exactly three minutes and then the solution is cooled immediately. After cooling 10 m ℓ each of solution "B" and solution "C" described below are added and mixed, and titrated with 0.07N sodium thiosulfate solution, using starch as the indicator. A blank test is carried out using the same procedure.

Solution "A": Solution of 225 grams of sodium triphosphate, 90 grams of Rochelle salt (sodium potassium tartrate), 30 grams of copper sulfate and 3.5 grams of potassium iodate, in sufficient distilled water to make 1 liter.

Solution "B": Solution of 90 grams of potassium oxalate and 40 grams of potassium iodide, in sufficient distilled water to make 1 liter.

Solution "C": 2N sulfuric acid

The glucose value of the sample material is calculated by the following formula:

Amount of glucose in 100 gram of sample material

$$= S \times (V_2 - V_1) \times 250 \div 10 \div W \times 100$$

where,

S = amount (g) of glucose equivalent to 1 m ℓ of 0.07N sodium thiosulfate solution (2.03)

V_1 = titrated amount (m ℓ) of 0.07N sodium thiosulfate for the sample material

V_2 = titrated amount (m ℓ) of 0.07N sodium thiosulfate for the blank test

W = amount (g) of sample cornstarch material used for the test

(h) Starch value

The starch value was calculated by multiplying the amount (%) of glucose measured in (g) above by 0.9.

(i) Protein

The percentage of nitrogen was obtained by the Kjeldahl method and protein was calculated by multiplying the nitrogen content by 6.25. The Kjeldahl method is as follows:

4 grams of sample material is accurately weighed and placed in a Kjeldahl decomposition flask, to which is added 10 grams of a decomposition accelerating agent (mixture of potassium sulfate and copper sulfate in a ratio of 9:1). Then 25 m ℓ of concentrated sulfuric acid and 5 m ℓ of 35 percent hydrogen peroxide solution are added and are decomposed carefully by heating. The decomposed solution is transferred to a Kjeldahl distillation unit and the total nitrogen content is obtained by a distillation analysis.

(j) Lipid

The amount of lipid was measured by the Soxhlet ether extraction method. The Soxhlet ether extraction method is as follows:

10 grams of the sample is accurately weighed and placed in a cylindrical paper filter (2.2 cm diameter and 9 cm height). On top of this are placed several layers containing a small amount of absorbent cotton under slight pressure. The cylindrical paper filter containing the sample material is dried at a temperature between 95 and 100°C for a period of 2 hours and is then placed in the Soxhlet lipid extraction unit, connected to a lipid measuring bin. This bin has been previously dried at 95 to 100°C and then weighed after being cooled in the desiccator.

Lipid in the sample material is extracted for 16 hours by ether. After extraction, the cylindrical filter is removed and the lipid measuring bin is placed in a dryer heated to 95 to 100°C in order to evaporate the ether. After cooling in the desiccator, the extracted lipid is weighed. The amount of lipid in the sample material is calculated by the following formula:

Lipid (%) = $(W_1 - W_2) \div W \times 100$

where,

W_1 : weight (g) of lipid measuring bin and extracted material

W_2 : weight (g) of lipid measuring bin

W : weight (g) of sample material used for the analysis

(k) Ash

The sample material was heated at 550 to 600°C in an electric furnace and the remaining ash was weighed.

(l) Metals

10 grams of the specimen was decomposed and dissolved in Aqua regia (a mixture of hydrochloric acid and nitric acid in the ratio of 1:3) and the sample material was decomposed completely by heating in nitric acid and sulfuric acid. Then, after adding diluted nitric acid, the solution was boiled and cooled. The metal contents were then measured.

(2) Results of the analyses

Two kinds of Zimbabwean cornstarch and a standard cornstarch made in Japan were analyzed by the analysis method described above and the results are shown in Table 10–1.

Table 10–1 Analysis Results of Cornstarch

	Japanese	STARCON	STARTEX–45
Color & Form	Light-yellow Powder	White Power	White Powder
Water Suspension	Normal	Normal	Normal
Supernatant Liquid	Normal	Normal	Normal
Water Content, %	12.9	4.8	14.8
Sugar Content, %	95.0	99.7	86.4
Starch Value, %	85.5	89.7	77.8
Protein, %	0.28	0.58	0.52
Lipid, %	0.050	0.063	0.088
Electrical Conductivity, μS/cm	100	218	2,028
Ash, %	0.03	0.16	0.83
pH	4.70	5.80	5.39
Copper, ppm	3.6	1.0	< 1.0
Manganese, ppm	< 1.0	< 1.0	2.0
Iron, ppm	3.0	9.5	39.0
Zinc, ppm	< 1.0	< 1.0	< 1.0
Magnesium, ppm	24	47	23

The results of the analysis showed that Zimbabwean cornstarch contains more protein and exceeds the 0.4 percent standard value for protein specified by the citric acid producing company who conducted this fermentation test. This excess protein means that it is considered slightly inferior as for citric acid production. Also the high electrical conductivity means that the cornstarch contains some electrolytes as impurities and this will present some problems.

10–2–3 Flask Test (First Round)

A fermentation test using an Erlenmeyer flask is widely used as a basic research method for selecting fermentation conditions. In this study, fermentation tests using flasks were carried out as preliminary tests prior to conducting jar fermentation tests. An outline of the flask test is described below. The conditions of the fermentation test are the same as those adopted for a commercial plant.

(1) Seed culture

50 grams of cornstarch was liquefied by an enzyme (0.05 grams of amylase in 10,000 units/grams). Nitrogen compound, phosphate, potassium compound and magnesium compound were added to the liquefied solution as inorganic nutrients together with organic nutrients and the solution was made up to a 1 liter volume by adding water. 200 m ℓ of the seed culture medium (liquefied cornstarch solution with nutrients) was poured into a 500 m ℓ Erlenmeyer flask which was sealed with a fermentation bung and then sterilized in an autoclave at 121°C for 20 minutes. After cooling, one platinum spatula full of spores of the fungus Aspergillus niger variation C-32 was transplanted into the flask under biochemically sterilized conditions and cultured for 45 hours at 35°C using a shaker rotating at 220 rpm.

(2) Main culture

The liquefaction enzyme (0.15 grams) was added to a water suspension of the sample material, which contains a defined amount (to be described later) of cornstarch. The liquefied solution was provided with nutrients and diluted with water to 900 m ℓ in a measuring cylinder. 90 m ℓ of the solution of liquefied cornstarch and nutrients were poured into each of ten 500 m ℓ Erlenmeyer flasks plugged with a bung, which were then sterilized in an autoclave at 121°C for 20 minutes. After subsequent cooling, 10 m ℓ of the seed fungi, obtained from the above mentioned seed culture process, was transplanted into each of the flasks under biochemically sterilized conditions. Fermentation was carried out at 35°C using a rotary shaker at 250 rpm. For a period of 6 to 8 days, each flask of the cultured medium was taken out once every 24 hours for observation and analysis of the fermentation process.

In this test, in order to calculate the yield of citric acid in a simplified manner, the total sugar content (total of sugar introduced from the main culture and sugar transferred from the seed) was fixed at 150 grams/liter. Using STARCON as an example, the method of calculating the amount of cornstarch to be used for the main culture is explained below.

(a) If the amount of sample material used in the main culture is A grams, then the amount of glucose produced from this cornstarch by decomposition by enzymes will be A×0.997 grams. This glucose solution, after being diluted to 900 m ℓ, is poured into 10 Erlenmeyer flasks so each will contain 90 m ℓ. Therefore, the amount of glucose in each Erlenmeyer flask is A × 0.997 × 10% = A × 0.0997 grams.

(b) Similarly in the seed culture process, 50 grams of cornstarch is diluted to one liter after liquefaction and 10 m ℓ of this is added to each Erlenmeyer flask for the main culture. Therefore, the amount of glucose added into each Erlenmeyer flask is 50×0.997 ÷ 100 ≒ 0.05 grams.

(c) From (a) and (b) above, the amount of glucose added to the Erlenmeyer flask is (A × 0.0997 + 0.05) grams and the volume of the solution in the Erlenmeyer flask is 90 + 10 = 100 m ℓ.

(d) Therefore, the concentration of glucose in the Erlenmeyer flask is:
(A × 0.0997 + 0.5) grams/100 m ℓ = (A × 0.997 + 5) grams/liter

(e) Since the concentration in (d) above is to be 150 grams/liter, the following equation can be solved for A which gives the required amount of cornstarch of 145.4 grams.
A × 0.997 + 5 = 150

(3) Results of flask test

Each Erlenmeyer flask was taken out every 24 hours and the contents were analyzed. The items analyzed and the methods used are as follows:

(a) pH : Measured by a glass electrode.

(b) Citric acid : 1 m ℓ of filtered culture liquid was titrated with 0.15N sodium hydroxide.

(c) Residual sugar : Sugar content of the filtered culture liquid was measured by Somogy's variation method.

(d) Fungi : The entire contents of each flask were filtered. The solid matter on the filter was washed with pure water, and then dried at 105°C. The solid matter was weighed and defined as fungi.

The results of flask test (1) are summarized in Table 10–2. The yields of citric acid were as follows:

– STARCON

Yield for total sugar : 115.5 gr/ℓ ÷ 150 gr/ℓ = 77.00%

Yield for consumed sugar : 115.5 gr/ℓ ÷ (150 – 12.8) gr/ℓ = 84.18%

– STARTEX-45

Yield for total sugar : 99.2 gr/ℓ ÷ 150 gr/ℓ = 66.13%

Yield for consumed sugar : 99.2 gr/ℓ ÷ (150 – 12.2) gr/ℓ = 71.99%

Table 10–2 Results of Flask Test (1)

Fermentation Period, Hours	pH	Citric Acid (CAM) (g/ℓ)	Residual Sugar (g/ℓ)	Fungi (g/ℓ)
<< Sample Name: STARCON >>				
0	3.35	--	--	--
24	2.70	5.2	131.2	11.4
48	2.27	21.5	89.5	14.4
72	2.00	49.0	65.0	16.3
96	1.85	70.6	47.5	16.4
120	1.74	93.2	30.3	16.3
144	1.72	108.5	17.9	16.1
168	1.69	115.5	12.8	16.1
<< Sample Name: STARTEX-45 >>				
0	3.36	--	--	--
24	2.65	4.0	134.9	11.2
48	2.28	17.1	112.2	14.5
72	2.02	35.9	85.0	16.9
96	1.80	56.2	62.5	19.0
120	1.70	75.0	45.6	19.8
144	1.70	88.6	23.1	19.7
168	1.68	99.2	12.2	19.3

10–2–4 Assessment of the Flask Test

Production yields of citric acid from the cornstarch produced in Zimbabwe showed inferior results compared to those obtained from cornstarch produced in Japan. Especially in the case of STARTEX-45, there was excess growth of fungi and the production of citric acid was reduced. However, it was concluded that both cornstarch can be used for citric acid production, by changing fermentation conditions.

10–2–5 Flask Test (Second Round)

Based on the results of the first flask fermentation tests, a second round of flask tests was carried out in which some of the fermentation conditions were changed with the aim of controlling the growth of fungi. The results are shown in Table 10–3.

Table 10–3 Results of Flask Test (2)

Fermentation Period, Hours	pH	Citric Acid (CAM) (g/ℓ)	Residual Sugar (g/ℓ)	Fungi (g/ℓ)
<< Sample Name: STARCON >>				
0	3.36	--	--	--
24	2.75	5.5	127.0	12.0
48	2.27	22.5	82.5	15.2
72	1.99	48.6	60.3	15.8
96	1.85	75.8	42.2	15.5
120	1.80	97.2	27.0	15.4
144	1.75	110.0	18.2	15.5
168	1.70	117.6	13.6	15.5
<< Sample Name: STARTEX-45 >>				
0	3.35	--	--	--
24	2.67	4.5	130.0	11.3
48	2.26	17.5	100.5	13.2
72	1.98	36.5	76.6	14.2
96	1.90	61.0	54.0	14.7
120	1.72	81.7	35.1	15.5
144	1.65	101.2	21.2	15.6
168	1.65	113.5	15.7	15.9

Yields of citric acid were as follows. Considerable improvements could be observed compared with the test results of the first round.

– STARCON

Yield for total sugar : 117.6 gr/ℓ ÷ 150 gr/ℓ = 78.40%

Yield for consumed sugar : 117.6 gr/ℓ ÷ (150 – 13.6) gr/ℓ = 86.22%

– STARTEX-45

Yield for total sugar : 113.5 gr/ℓ ÷ 150 gr/ℓ = 75.67%

Yield for consumed sugar : 113.5 gr/ℓ ÷ (150 – 15.7)gr/ℓ = 84.51%

10–2–6 Jar Fermentation Test

From the results of the flask tests, it was judged that Zimbabwean cornstarch could be used for citric acid production, therefore confirmation tests were carried out on a larger scale using Jar Fermenters (Figure 10–1). The jar fermenters used for the test were vessels of 28 cm diameter and 48 cm in height with an inner volume of 30 liters, equipped with a 6 bladed turbine type bottom impeller of 14 cm diameter, and a similar type top impeller with 4 blades. There was also an agitator shaft baffle consisting of 2 plates with a diameter impeller and a supercharger for aeration. The temperature, pH value, agitation speed, and aeration air volume of the jar could be controlled automatically or manually. The culture solution was charged into the jar with water, mixed by the agitator and sterilized with steam injected through the jacket and supercharger. The following is a description of the jar fermentation tests:

(1) Seed culture

Seed culture fermentation, prior to jar fermentation, was carried out in two steps. Cornstarch used for seed culture was Japanese cornstarch, in order to standardize fermentation conditions.

(a) First step

50 grams of sample material was liquefied with the enzyme (α-amylase), nutrients were added and the solution was diluted with water to make 1 liter. 200 mℓ of seed culture liquid was poured into a 500 mℓ Erlenmeyer flask and sealed with a bung, after it was sterilized in an autoclave at 121°C for 20 minutes. After cooling, spores of the C-32 variety of Aspergillus niger fungi were transplanted into the sterilized culture liquid by a platinum spatula (a medical spoon the size of an earpick) under biochemically sterilized conditions and culture was carried out for 45 hours at 35°C using a rotary shaker at 220 rpm.

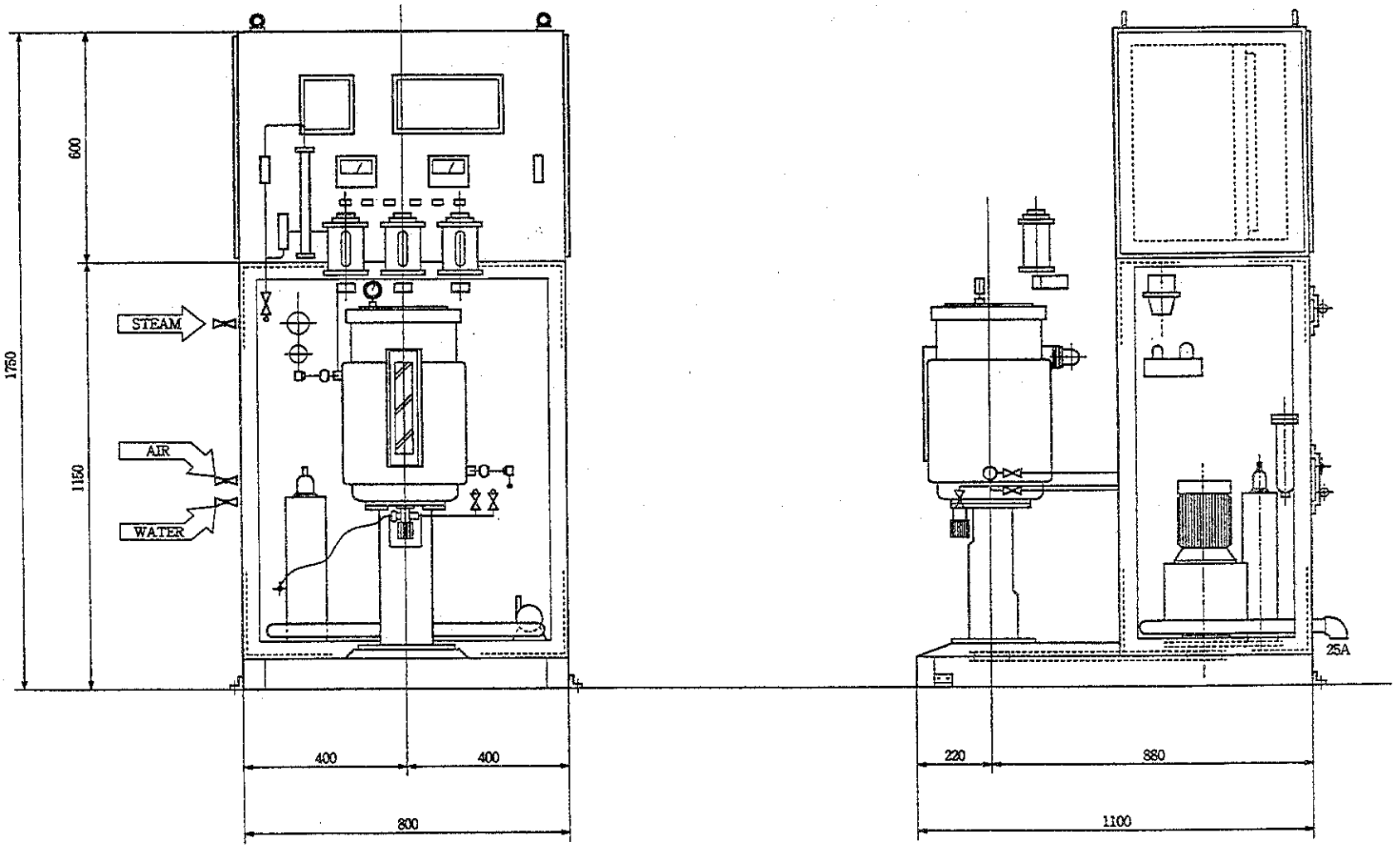


Figure 10–1 General View of a Jar Fementer

(b) Second step

180 m ℓ of liquefied cornstarch solution of the same concentration (50 gr/ ℓ) as that of the first step culture liquid was poured into each of ten 500 m ℓ Erlenmeyer flasks and sterilized. After cooling, 20 m ℓ of the first step seed culture was transplanted into each Erlenmeyer flask under biochemically sterilized conditions and subsequently fermented at 35°C for 24 hours using a rotary shaker at 220 rpm.

(2) Main culture

The required amount (to be described later) of cornstarch was liquefied and poured into the jar fermenter. Nutrients were added, and the contents were sterilized by injecting steam. After cooling, the culture solution from the ten 500 m ℓ Erlenmeyer flasks, totaling 2 liters, and 100 grams of cornstarch were poured into the jar. After seed plantation, the solution was made up to 20 liters by adding sterilized water. Fermentation was carried out at 35°C with aeration at 0.5 vvm (10 liters per minute).

The amount of cornstarch was fixed at 150 grams/liter as glucose. The required amount of cornstarch was calculated by the following formula using STARCON as an example:

Amount of cornstarch = 150 gr/ ℓ × 20 ℓ ÷ 0.997 = 3,009 gr

(3) Results of the jar test

150 m ℓ of fermentation liquid was taken out every 24 hours. The first 50 m ℓ was discorded and the next 100 m ℓ was used as a sample for the measurements. The test items and methods of analysis were the same as for the flask test. The results of the jar fermentation test are summarized in Table 10–4.

Table 10–4 Results of the Jar Tests

Fermentation Period, Hours	pH	Citric Acid (CAM) (g/ℓ)	Residual Sugar (g/ℓ)	Fungi (g/ℓ)
<< Sample Name: STARCON >>				
0	3.32	--	--	--
24	1.95	18.0	--	7.4
48	1.88	52.6	89.0	8.7
72	1.80	83.5	68.9	9.8
96	1.69	108.4	49.7	10.5
120	1.66	124.9	34.2	11.3
144	1.66	135.0	25.5	11.4
168	1.66	137.9	14.5	11.5
<< Sample Name: STARTEX-45 >>				
0	3.27	--	--	--
24	2.01	24.3	--	11.0
48	1.85	69.1	80.4	11.3
72	1.78	91.0	68.0	11.9
96	1.69	116.0	39.3	12.2
120	1.68	131.2	24.0	13.3
144	1.68	136.2	18.8	13.4
168	1.67	136.8	18.2	13.0
<< Sample Name: Japanese Corn Starch >>				
0	3.22	--	--	--
24	1.99	23.1	--	6.9
48	1.86	55.7	77.9	8.5
72	1.79	84.4	58.4	9.2
96	1.70	109.1	38.3	9.8
120	1.65	125.0	22.6	10.1
144	1.66	135.2	13.9	11.0
168	1.68	139.0	11.3	10.8

The yields of citric acid are as follows:

(a) STARCON

Culture medium : Measured volume of remaining culture medium at the end of fermentation plus the volume of extracted samples (8 × 150 mℓ) ..... 18.4 ℓ

Input of glucose : Input glucose + glucose from seed

= 150 gr/ℓ × 20 ℓ + 100 gr × 0.95%

= 3,095 gr

Yield for total glucose : 137.9 gr/ℓ × 18.4 ℓ ÷ 3,095 gr

= 81.98%

Yield for consumed glucose: (137.9 gr/ℓ × 18.4 ℓ) ÷ (3,095 – 14.5 gr/ℓ × 18.4 ℓ)

= 89.71%

(b) STARTEX-45

Culture medium : Measured volume of remaining culture medium at the end of fermentation plus the volume of extracted samples (8 × 150 mℓ) ..... 18.2 ℓ

Input of glucose : Input glucose + glucose from seed
= 150 gr/ℓ × 20 ℓ + 100 gr × 0.95%
= 3,095 gr

Yield for total glucose : 136.8 gr/ℓ × 18.2 ℓ ÷ 3,095 gr = 80.45%

Yield for consumed glucose: (136.8 gr/ℓ × 18.2 ℓ) ÷ (3,095 gr – 18.2 gr/ℓ × 18.2 ℓ)
= 90.09%

(c) Japanese made cornstarch

Culture liquid : Measured volume of remaining culture liquid at the end of fermentation plus the volume of extracted samples (8 × 150 mℓ) ..... 18.7 ℓ

Input of glucose : Input glucose + glucose from seed
= 150 gr/ℓ × 20 ℓ + 100 gr × 0.95%
= 3,095 gr

Yield for total glucose : 139.0 gr/ℓ × 18.7 ℓ ÷ 3,095 gr = 83.98%

Yield for consumed glucose: (139.0 gr/ℓ × 18.7 ℓ) ÷ (3,095 gr – 11.3 gr/ℓ × 18.7 ℓ)
= 90.14%

10–2–7 Results and Assessment

As a result of the flask fermentation tests and jar fermentation tests, it was positively confirmed that Zimbabwean cornstarch could be used for citric acid production. Zimbabwean cornstarch contains more protein, ash, and metals such as iron, than Japanese cornstarch, but it was found that these impurities would not be critically detrimental to the fermentation process.

10–3 Fermentation Tests using Sweet Potato and Cassava

10–3–1 Basic Policy

The main raw material for the commercial production of citric acid in Japan by the solid culture fermentation process is sweet potato starch extraction residues (residues remaining after producing starch from sweet potatoes). The study team tried to secure sweet potato starch waste similar to that produced in Japan. The team considered importing residues after starch extraction in Zimbabwe or importing raw sweet potatoes and making starch extraction residues in Japan. However, there is no production of sweet potato starch in Zimbabwe and there is no local organization which makes starch waste from sweet potatoes. Therefore, it was impossible to secure the sweet potato starch extraction residues in Zimbabwe. It was also judged that the import of raw sweet potatoes into Japan would be extremely difficult due to quarantine regulations. Therefore, as the next best measure, dried chips of sweet potatoes and cassavas were imported and starch extraction residues were made from them in Japan. Considering the amount of sweet potatoes and cassavas obtained in Zimbabwe, namely, 20 kg of dried sweet potato chips and 3 kgs of dried cassava chips, the following tests were carried out:

(1) Analysis of the dried sweet potato/cassava chips
(2) Preparation of starch extraction residues for fermentation tests, using dried sweet potato/cassava chips
(3) Analysis of the starch extraction residues prepared in this way
(4) Fermentation tests using the sweet potato starch extraction residues
(5) Fermentation tests using the cassava extraction residues
(6) Fermentation tests using the whole sweet potato
(7) Fermentation tests using the whole cassava

10–3–2 Analysis of Sample Material

(1) Items analyzed and methods used

The dried sweet potato and cassava chips imported from Zimbabwe were analyzed. Items analyzed and methods used are explained below.

(a) Water content

Accurately weighed sample material was dried in a constant temperature dryer at 100 to 110°C for three hours. After cooling, the sample was accurately weighed. The difference in weight before and after drying is the water content.

(b) Glucose

Sample material was hydrolyzed by hydrochloric acid and the glucose was measured by the Somogy variation method (refer to section 10–2).

(c) Starch value

The starch value could be obtained by multiplying the glucose value by 0.9.

(d) Fiber

A two grams sample of the material was weighed accurately, and then boiled in 0.255N sulfuric acid solution for 30 minutes. After washing with warm water, it was boiled in 0.313N sodium hydroxide for 30 minutes. The sample material, after being decomposed by the alkali, was poured into a Gooch crucible. After washing, it was dried at 110°C, then cooled and weighed accurately. The sample material was incinerated in an electric furnace at 450 to 550°C and, after cooling, was accurately weighed again. The difference of weight before and after incineration is defined as the fiber content.

(e) Protein

The nitrogen content was measured by the Kjeldahl method (refer to section 10–2). The protein value was obtained from the nitrogen content by multiplying by 6.25.

(f) Lipid

The amount of lipid was measured by the Soxhlet ether extraction method (refer to section 10–2).

(g) Ash

After incineration of sample material in an electric furnace at 550 to 600°C, the weight of the remaining matter was the ash content.

(2) Results of the analyses

The results of the analyses are shown in Table 10–5. Standard analysis data for dried Japanese sweet potato chips is shown for reference.

Table 10–5 Analysis Results of Dried Sweet Potato and Dried Cassava

(Unit: %)

	Sweet Potato (Zimbabwean)		Sweet Potato (Japanese)		Cassava (Zimbabwean)	
Water Content	9.80		14.70		11.09	
Glucose	75.21	(83.38)	81.10	(95.08)	76.12	(85.61)
Starch Value	67.69	(75.04)	72.99	(85.57)	68.51	(77.06)
Fiber	2.33	(2.58)	1.98	(2.32)	2.79	(3.14)
Protein	4.97	(5.51)	3.96	(4.64)	1.66	(1.87)
Lipid	0.88	(0.98)	0.74	(0.87)	0.83	(0.93)
Ash	3.36	(3.73)	2.23	(2.73)	4.00	(4.59)

Note: Figures in parentheses are dry basis.

10–3–3 Preparation of Starch Extraction Residues

Starch extraction residues were prepared from sweet potatoes and cassavas imported from Zimbabwe. The preparation process for starch extraction residues was as follows:

(1) Coarse crushing of raw material (sweet potatoes and cassavas)

(2) Water soaking (mixing, settling and discharging of supernatant liquid: these operations were repeated six times)

(3) Crushing by a mixer and adding water

(4) Filtration

(5) Separation of starch

The residues from the dried chips were as follows:

– Sweet potato : 21.0% (dry basis)

– Cassava : 19.1 % (dry basis)

10–3–4 Analysis of Starch Extraction Residues

The results of the analysis of starch extraction residues prepared for fermentation tests are shown in Table 10–6. Standard analysis data for Japanese starch extraction residues are also shown for reference. The analysis items and methods used are identical to those for dried chips. Since the standard analysis value of Japanese starch extraction residues is for a dry basis (water content about 18%), the analysis of starch extraction residues prepared for the fermentation tests was conducted on a dry basis.

Table 10–6 Analysis Results of Starch Extraction Residues from Sweet Potato & Cassava

(Unit: %)

	Sweet Potato (Zimbabwean)		Sweet Potato (Japanese)		Cassava (Zimbabwean)	
Water Content	15.00		18.10		13.59	
Glucose	64.45	(75.82)	53.14	(64.88)	61.18	(70.80)
Starch Value	58.00	(68.24)	47.83	(58.40)	55.06	(63.72)
Fiber	8.47	(9.96)	12.40	(15.14)	11.40	(13.19)
Protein	2.75	(3.24)	1.40	(1.71)	0.79	(0.91)
Lipid	0.54	(0.64)	0.80	(0.98)	0.47	(0.54)
Ash	1.73	(2.04)	1.80	(2.20)	2.45	(2.84)

Note: Figures in parentheses are dry basis.

10–3–5 Fermentation Tests using Sweet Potato Starch Extraction Residues

The fermentation tests using sweet potato starch extraction residues were conducted in the following sequence:

- Petri dish test
- Beaker test (500 m ℓ)
- Tests using trays which are used for the commercial production of citric acid

(1) Fermentation test using a Petri dish

(a) Test method

Nutrients (rice bran at the rate of 2 to 6% of the starch extraction residue by weight) were added to 15 grams of starch extraction residue (water content: 70.80%, de-watered total glucose: 75.82%) and this formed a solid culture fermentation medium. Petri dishes with a diameter of 8.7 cm and a depth of 1.8 cm were evenly filled with the fermentation medium. Then the filled petri dishes were placed in a pressurized autoclave (1.2 kg/cm^{2}g) and sterilized for 30 minutes. After cooling, separately fermented fungi were transplanted on to the sterilized fermentation medium which was then fermented at 30°C. After 48 hours and after 168 hours from the start of the test, a Petri dish was removed and the citric acid content was analyzed. The analysis method was as follows:

The contents of the Petri dish were removed and put in a 300 m ℓ Erlenmeyer flask, which was heated after adding 250 m ℓ of water. As soon as it had boiled, heating was stopped and it was then cooled in running water. 25 m ℓ of supernatant liquid was taken and titrated with 0.1N sodium hydroxide to measure the total acid content. This acid content represents the

amount of citric acid. The amount of glucose contained in the 15 grams of starch extraction residue is 15 gr × (100 – 70.80)% × 75.82% = 3.32 grams.

(b) Results of the test

The results of the fermentation test in Petri dishes are summarized in Table 10–7. Yields for glucose are calculated from the amount of citric acid produced divided by the glucose content in the starch extraction residue.

Table 10–7 Results of Fermentation Tests using Sweet Potato Residues in Petri Dishes

Sample No.	1	2	3	4	5	6	7	8
Nutrient	RB 2%	RB 3%	RB 4%	RB 5%	RB 6%	WB 3%	WB 4%	WB 5%
<< Fermentation Period; 48 Hours >>								
CAM (g)	0.92	1.09	1.22	1.37	1.29	1.08	1.29	1.32
Yield (%)	27.7	32.8	36.7	41.3	38.9	32.5	38.9	39.7
<< Fermentation Period; 168 Hours >>								
CAM (g)	2.68	2.53	2.44	2.23	2.03	2.48	2.39	2.19
Yield (%)	80.7	76.2	73.5	67.2	61.1	74.7	72.0	66.0

Note: RB and WB represent Rice Bran and Wheat Bran.

(2) Fermentation test using 500 m ℓ beakers

Good results were obtained from the fermentation tests using Petri dishes, and so fermentation tests with increased amounts of the sample material were conducted using 500 m ℓ beakers (diameter 9 cm, height 12 cm).

(a) Test method

The test method using beakers was fundamentally the same as that applied for the Petri dish test. The procedure differed in the following ways:

1) The amount of sample material used for the fermentation test was increased to 105 grams.
2) In order to provide appropriate aeration characteristics, 5 grams of bagasse were added.

(b) Test results

The results of the fermentation tests using 500 m ℓ beakers are summarized in Table 10–8.

Table 10–8 Results of Fermentation Tests using Sweet Potato Residues in 500 m ℓ Beakers

Sample No.	Nutrient	Yield (%)	
		Fermentation 48 Hours	Fermentation 168 Hours
1	Rice Bran 1%	21.0	78.9
2	Rice Bran 2%	26.9	79.3
3	Rice Bran 3%	32.1	76.1
4	Rice Bran 4%	31.5	70.4
5	Rice Bran 5%	36.4	62.2
6	Wheat Bran 2%	24.8	75.5
7	Wheat Bran 3%	27.0	73.4
8	Wheat Bran 4%	30.8	67.7

(3) Fermentation tests using tray pots

Good results were also obtained from the fermentation test with 500 m ℓ beakers, and so a fermentation test using a commercial production tray was carried out. The tray was placed in a fermentation room of a factory (Kyushu Chemical Co. Ltd.). The size of the tray was approximately 50 cm long × 35 cm wide × 12 cm deep.

(a) Test method

The fermentation method used for commercial solid culture production was applied during this test. Because of the limited amount of available raw material, one fifth of the tray was separated by a partition plate and filled with a solid culture fermentation medium composed of a mixture of 2,000 grams of starch extraction residues and 100 grams of bagasse.

(b) Test results

After 168 hours in a fermentation room, the yields of citric acid as a percentage of glucose were as follows:

– When 1% of rice bran was added as nutrient : 80.2%

– When 2% of rice bran was added as nutrient : 71.4%

For reference, the average yield of citric acid achieved commercially in Japan is approximately 85%.

(4) Summary

In the case of the Petri dish test, the yield for glucose was 80.7% and in the case of the 500 m ℓ beaker test, the yield for glucose was 79.3%. In the case of the fermentation test using a tray placed in an actual plant fermentation room, the yield obtained for glucose was 80.2%. These test results did not show much difference from the figures obtained in commercial operations in Japan. Sweet potatoes produced in Zimbabwe can be used as a raw material for citric acid production using the solid culture fermentation process. Regarding nutrients, tests were conducted using wheat bran in addition to the tests with rice bran. Although citric acid production is possible using wheat bran as a nutrient, the yield of citric acid was about 10% less than when rice bran was used.

10–3–6 Fermentation Tests using Cassava Starch Extraction Residues

The fermentation test using cassava starch extraction residues was carried out with Petri dishes. In view of the limited amount of available cassava, tests using 500 m ℓ beakers and trays were not performed. Nutrients (rice bran and wheat bran) and bagasse (0 to 1 g) were added to 15 grams of cassava starch extraction residues and fermentation tests were carried out in a similar way as was done for sweet potatoes. The results of the fermentation test are shown in Table 10–9.

Table 10–9 Results of Fermentation Tests using Cassava Residues in Petri Dishes

Sample No.	Nutrient	Bagasse	Yield (%)	
			Fermentation 48 Hours	Fermentation 168 Hours
1	Rice Bran 1%	--	10.1	62.3
2	Rice Bran 2%	--	16.0	75.7
3	Rice Bran 3%	--	19.4	85.4
4	Rice Bran 4%	--	26.9	81.0
5	Rice Bran 5%	--	32.1	72.8
6	Rice Bran 1%	1 g	--	80.6
7	Rice Bran 2%	1 g	--	84.0
8	Rice Bran 3%	1 g	--	87.7
9	Rice Bran 4%	1 g	--	81.7
10	Rice Bran 5%	1 g	--	80.2
11	Wheat Bran 1%	--	9.0	66.0
12	Wheat Barn 2%	--	15.7	75.7
13	Wheat Barn 3%	--	19.8	77.6
14	Wheat Bran 4%	--	26.5	76.5
15	Wheat Bran 5%	--	33.2	67.9

Because the amount of cassava obtained in Zimbabwe was small, tests using beakers and trays were not conducted. However, good results were obtained from the Petri dish tests and it is judged that citric acid production from cassava starch extraction residues is possible on an industrial scale. Fermentation tests were also carried out using a solid culture medium to which bagasse had been added. The reason for adding bagasse was that, judging from the condition of the cassava starch extraction residues, better fermentation results might be obtained if bagasse was used as a carrier. The results of the test were good, far better than originally anticipated.

10–3–7 Fermentation Tests using Whole Sweet Potatoes

The reason that Japanese citric acid producers use sweet potato extraction residues is that the price is low and it is suitable as an industrial raw material. The use of whole sweet potatoes is not profitable because they are expensive. Considering that the situation in Zimbabwe may be different, fermentation tests were made using crushed chips of whole sweet potatoes.

(1) Fermentation tests using Petri dishes

The solid culture fermentation medium was prepared by mixing 5 grams of crushed whole sweet potato chips, 1 to 2 grams of bagasse and 0 to 1.5% of rice bran as a nutrient. Fermentation tests were carried out using a test method similar to the one used for starch extraction residues. The results of the fermentation tests are shown in Table 10–10.

Table 10–10 Results of Fermentation Tests using Whole Sweet Potatoes in Petri Dishes

Sample No.	Rice Bran	Bagasse	Yield (%)		
			48 Hours	96 Hours	168 Hours
1	0.0%	1.0 g	41.2	73.8	58.8
2	0.5%	1.0 g	40.4	74.1	54.5
3	1.0%	1.0 g	48.9	73.8	51.3
4	1.5%	1.0 g	45.5	71.4	50.2
5	0.0%	1.5 g	48.9	78.1	61.0
6	0.5%	1.5 g	50.0	78.9	62.6
7	1.0%	1.5 g	51.6	79.7	55.9
8	1.5%	1.5 g	52.1	76.7	55.1
9	0.0%	2.0 g	52.4	80.5	60.2
10	0.5%	2.0 g	51.9	81.0	61.7
11	1.0%	2.0 g	54.9	80.7	60.2
12	1.5%	2.0 g	54.9	77.2	56.4

(2) Fermentation test by 500 mℓ beakers

The solid culture fermentation medium was prepared by mixing 50 grams of crushed whole sweet potato chips, 15 to 20 grams of bagasse and 0 to 1% of rice bran as a nutrient. Fermentation tests were carried out using a test method similar to the one used for Petri dish tests. The results of the fermentation tests are shown in Table 10–11.

Table 10–11 Results of Fermentation Tests using Whole Sweet Potatoes in 500 mℓ Beakers

Sample No.	Rice Bran	Bagasse	Yield (%)	
			48 Hours	96 Hours
1	0.0%	15 g	13.3	75.4
2	1.0%	15 g	20.0	75.3
3	0.0%	20 g	14.1	77.5
4	1.0%	20 g	17.7	76.6

(3) Summary

When whole sweet potatoes were used, the excess nitrogen content generated much heat. For industrial production using whole sweet potatoes, fermentation conditions and the plant and equipment should be carefully reviewed. However, fermentation is faster and a better yield is obtained. It would be possible to use whole sweet potatoes for citric acid production.

10–3–8 Fermentation Tests using Whole Cassavas

(1) Fermentation tests using Petri dishes

A solid culture fermentation medium was prepared by mixing 5 grams of crushed whole cassava chips, 1 to 2 grams of bagasse and 1 to 3.5% of rice bran as a nutrient. Fermentation tests identical to those for whole sweet potatoes were carried out. The results of fermentation tests are shown in Table 10–12.

Table 10–12 Results of Fermentation Tests using Whole Cassava in Petri Dishes

Sample No.	Rice Bran	Bagasse	Yield (%)		
			48 Hours	120 Hours	168 Hours
1	1.0%	1.0 g	27.2	75.9	67.2
2	2.0%	1.0 g	35.4	77.0	65.9
3	2.5%	1.0 g	35.2	76.5	63.2
4	3.0%	1.0 g	40.2	76.2	64.8
5	3.5%	1.0 g	42.6	73.0	62.2
6	1.0%	1.5 g	43.4	81.0	70.3
7	2.0%	1.5 g	43.4	81.7	68.0
8	2.5%	1.5 g	45.2	82.3	67.5
9	3.0%	1.5 g	47.3	80.9	67.5
10	3.5%	1.5 g	48.1	79.8	57.4
11	1.0%	2.0 g	50.3	84.9	71.7
12	2.0%	2.0 g	50.5	85.4	69.3
13	2.5%	2.0 g	52.6	83.6	64.3
14	3.0%	2.0 g	52.1	83.0	64.0
15	3.5%	2.0 g	21.0	82.5	64.3

(2) Fermentation tests using 500 m ℓ beakers

A solid culture fermentation medium was prepared by mixing 50 grams of crushed whole cassava chips, 15 to 20 grams of bagasse and 1 to 2% of rice bran. Fermentation tests were carried out using a similar test method to that used for the Petri dish tests. The results of the fermentation tests are shown in Table 10–13.

Table 10–13 Results of Fermentation Tests using Whole Cassavas in 500 m ℓ Beakers

Sample No.	Rice Bran	Bagasse	Yield (%)	
			48 Hours	120 Hours
1	1.0%	15 g	16.9	79.1
2	2.0%	15 g	19.2	80.4
3	1.0%	20 g	17.3	81.1
4	2.0%	20 g	22.6	82.5

(3) Summary

When whole cassava was used, good results were obtained from the Petri dish tests and in the case of the beaker tests, good results were obtained with yields of citric acid against glucose exceeding 80%. Nitrogen content in cassavas is lower than that of sweet potatoes, but the fermentation was very active and a considerable amount of heat generation was observed. Therefore, if fermentation is done on an industrial scale, it will be necessary to study fermentation conditions and the design of equipment for a factory carefully. The use of whole cassavas is promising.

10–3–9 Conclusions

Good results were obtained from fermentation tests conducted using starch extraction residues, which were prepared from sweet potatoes and cassavas. In the case of cassavas, the limited amount of sample material prevented us from conducting a tray scale fermentation test in an actual fermentation room. However, judging from the test results, it is expected that good results would also have been obtained if a tray scale test had been conducted. It was confirmed that good results were obtained for fermentation tests of crushed whole sweet potato and cassava chips, if suitable carriers were used. Due to excess nutrients as a result of the nitrogen content and other constituents, a large heat generation was observed. Therefore, in the case of large scale production it would be necessary to study fermentation conditions in order to suppress the heat generation.

From the series of tests, it is concluded that citric acid production using sweet potatoes and cassavas is technically possible with the solid culture fermentation process.

Wheat bran can be used as a nutrient for solid culture fermentation as a substitute for rice bran, although the yield achieved is somewhat inferior.

10–4 Fermentation Tests using Sugar Related Materials

10–4–1 Fundamental Policy

The possibility of producing citric acid by using cheaply available sugar related materials in Zimbabwe was examined. The semi-solid culture process which has used experimentally is a method of promoting the fermentation of a carbohydrate which has been spread on a suitable carrier. This method has been studied at a laboratory, but there is no commercial plant that uses this method.

10–4–2 Carrier and Raw Materials

Fermentation experiments were carried out using the following carrier and raw materials collected during the field survey:

(1) Carrier: Bagasse

(2) Sugar related raw materials:

- Crude sugar
- Affination syrup obtained in the process of refining crude sugar
- Condensed sugarcane syrup
- Process molasses which is obtained from the process of refining crude sugar
- "B" Molasses, mother liquor after extracting crude sugar twice by crystallization

10–4–3 Analysis of Raw Materials

Analysis of the above 5 kinds of sugar related materials was performed by the high speed liquid chromatograph method. The results of the analysis are shown in Table 10–14.

Table 10–14 Results of Raw Material Analysis

(Unit: %)

	Oligosaccharide	Sucrose	Glucose	Fructose	Total
Raw Sugar	0.0	91.5	5.3	2.8	99.6
Affination Syrup	2.3	1.5	26.3	25.0	55.3
Condensed Sugarcane Syrup	6.7	52.5	4.8	4.0	68.0
Process Molasses	11.8	32.1	4.6	4.7	53.2
"B" Molasses	23.4	35.0	4.5	7.1	70.0

10–4–4 Fermentation Tests

Dried bagasse (3.9 grams) was placed in a 9 cm diameter Petri dish as the carrier and 15 mℓ of fermentation medium was spread over the carrier. Then the dish and its contents were sterilized with steam. After cooling, fungi (Aspergillus niger yang No. 2) were transplanted and fermentation was performed under static conditions at 30°C. Included in the fermentation medium were nutrients consisting of 30 mg of ammonium nitrate, 150 mg of potassium dihydrogenphosphate, 3.75 mg of magnesium sulfate, 0.21 mg of manganese sulfate, and 0.3 mg of ferric chloride. After 3 days and 5 days, the amount of citric acid produced was measured and the yield for glucose calculated. Conditions of the fermentation medium and results of the fermentation tests are summarized in Table 10–15.

Table 10–15 Results of Fermentation Tests using the Semi-solid Culture Process

Test No.	Culture Medium		Fermentation Results			
	Raw Material	Total Sugar as Glucose	CAM (g)		CAM Yield (%)	
			3 days	5 days	3 days	5 days
1	Raw Sugar	2.10 g (140g/ℓ)	1.305	1.508	62.1	71.8
2	Raw Sugar	2.25 g (150g/ℓ)	1.328	1.643	59.0	73.0
3	Raw Sugar	2.40 g (160g/ℓ)	1.373	1.935	57.2	80.6
4	Raw Sugar	2.70 g (180g/ℓ)	1.485	2.073	55.0	76.7
5	Affination Syrup	2.10 g	0.195	0.458	16.8	39.5
6	Condensed Sugarcane Juice	1.34 g	0.486	0.567	53.4	62.3
7	Process Molasses	2.40 g	0.486	0.953	38.0	74.5
8	"B" Molasses	3.00 g	0.525	trace	25.0	trace

The maximum yield of the citric acid production using the raw sugar as the raw material was 80.6% and it was proved that the citric acid production by the semi-solid culture process is technically possible. The results of the fermentation test using the condensed sugarcane juice and the process molasses as the raw material were also comparatively good.

Chapter 11 Project Scheme

Chapter 11 Project Scheme

11–1 Plant Capacity

This feasibility study establishes a target territory for the citric acid market in Zimbabwe and other Southern African countries. This inevitably is based on a number of uncertain factors concerned with marketing and sales which may affect the introduction of a largely export oriented citric acid plant (i.e. a plant with a capacity of more than 4,000 tons/year). These include:

(1) The markets in the countries north of the Sahara are close to the major world source of citric acid, which is Europe, and the markets in the Middle East and further east are the territories of the South-East Asian and Chinese producers. So very tough sales competition can be expected from these major producers, except in Southern Africa.

(2) Currently, there is equilibrium in the world's supply and demand for citric acid.

It is estimated that the domestic market for citric acid in Zimbabwe in 1996, when the subject plant is going to start commercial operation, will be at 900 tons per year. Demand for citric acid in the Republic of South Africa in 1996 is forecast at 4,700 tons per year, and the demand for citric acid in other neighboring countries is estimated to be 600 tons per year in 1996, thus the total demand for citric acid in the subject market will be 6,200 tons per year. The potential sales expected for the subject plant will be 3,300 tons per year, which corresponds to 53% of the total area demand of 6,200 tons per year as described in Chapter 5.

From the viewpoint of economics, the production capacity of a citric acid plant should preferably be as large as possible. On the other hand, after constructing a large scale plant, if the product is not sold as planned and the plant is obliged to operate at reduced capacity, the financial burden of the project will be increased. This kind of situation should be avoided by all means. The plant capacity is, therefore, set at 3,000 tons per year, as citric acid monohydrate, which is somewhat on the conservative side of anticipated sales for this project.

11–2 Selection of Raw Materials

As the raw material for citric acid production by the process of fermentation by microorganisms, the following materials have been considered for this project:

(1) Sweet potato residues

(2) Cassava residues

(3) Cornstarch

(4) Sugar cane molasses

(5) Sugar intermediate products such as concentrated cane juice and crude sugar

Of these five kinds of candidate raw materials, citric acid production using sweet potato or cassava residues uses the solid culture fermentation method. The initial investment for the solid culture method is lower than that of the submerged culture method and it is a labor intensive operation. Thus the solid culture method can be recommended for application in Zimbabwe. The study team gave first priority to studying the possibility of securing sweet potato and cassava residues which are most suitable for the solid culture process.

However, sweet potato and cassava residues do not exist in Zimbabwe as described in detail in Chapter 7 "Raw Materials", and only a small amount of sweet potatoes and cassava are harvested by farmers using rather primitive cultivation methods.

Therefore, sweet potatoes and cassava have been deleted from the list of candidate raw materials for this project. All available sugarcane molasses are used as the raw material for producing ethanol for blending into gasoline for automobiles and a shortage of molasses is filled by importation from Zambia. A citric acid plant using crude sugar or concentrated cane juice as a raw material has not been developed yet and it will require a tremendous amount of time and money to develop one. The cost of crude sugar and concentrated cane juice is higher than that of molasses and it will be difficult to find an entrepreneur to commercialize the process. For the reasons mentioned above, sugarcane molasses, crude sugar and concentrated cane juice have been deleted from the list of candidate raw materials.

In the course of discussing the selection of raw materials, cornstarch remains as the practical raw material for this project.

The production of maize in Zimbabwe is approximately one million tons per year, although it has declined slightly in recent years. The harvest of maize per unit area is high in Zimbabwe and maize production is competitive internationally. The amount of maize needed to produce the cornstarch required for this project is about 6,000 tons per year and there will be no problem in securing this amount of maize. The annual production of cornstarch in Zimbabwe is 12,000 tons and currently there is no surplus capacity to supply the 4,100 tons of cornstarch required for the production of 3,000 tons of citric acid per year, but it would be possible to expand the production of cornstarch.

The price of cornstarch in Zimbabwe is high at present, but there is some room to improve the present price.

The reasons for selecting the raw material are described in detail in Chapter 7.

Table 11-1 summarizes several factors used as judgement criteria in the selection process and their current situation in Zimbabwe.

Table 11-1 Selection of a Raw Material

Material	Process	Availability	Industrial Technology	Material Cost	Adoption for the Project
Sweet potato residues	○ Solid culture	× No	○ Exists	N.A	× Reject
Cassava residues	○ Solid culture	× No	○ Exists	N.A	× Reject
Cornstarch	○ Submerged culture	○ Good	○ Exists	△*	○ Adopt
Sugarcane molasses	○ Solid culture	× Import	× No	○ Reasonable	× Reject
Sugarcane intermediate products	○ Solid culture	○ Good	× No	× Expensive	× Reject

Remark : * Expensive, but possibility of price reduction exists.
Symbol : ○ ..... Acceptable
× ..... Rejected
△ ..... Not ideal but acceptable

As is clear from the above table, cornstarch remains as the only one raw material for this project from the five candidates. Thus, cornstarch has been established as the raw material for this project.

11-3 Comparison of Processes for Citric Acid Production

An outline of the citric acid production process will be discussed in the following section from the viewpoint of applicability for this project.

11-3-1 Solid Culture Method

Fungi which produce citric acid breed under aerobic conditions. In the solid culture process, it is necessary to prepare fermentation beds which have good aeration capability so that fungi can maintain constant contact with air. Therefore, it is necessary that the nature of the raw material in itself provides good aeration.

Sweet potato and cassava residues contain a considerable amount of fiber in the raw material and these fibers form a porous bed. The fiber containing raw materials are mixed with nutrients, filled in trays after sterilizing, and planted with fungi which produce citric acid as they multiply. These trays are placed on shelves in fermentation rooms for fermentation. The construction cost of a citric acid plant using the solid fermentation method is less expensive than one using the submerged culture process. Also, in the case of solid fermentation, there are a number of operations which need manpower such as filling fermentation beds in trays, transportation of trays into fermentation rooms and placing trays onto shelves in the rooms, removal of trays after fermentation, washing of trays after use and so on. Thus this process would create more employment opportunities which meets one of the objectives set forth for this project.

Fermentation tests using the starch extraction residues, processed from Zimbabwe's sweet potatoes and cassavas, and Japanese rice bran showed yields against total sugar of about 80% for the sweet potato starch residues and about 85% for the cassava starch residues.

Thus, citric acid production by the solid culture fermentation process using either material is technically possible.

However, it was found during the field survey that the raw materials for this process, namely, sweet potato and cassava residues, and rice bran as a nutrient were not available in Zimbabwe, and adoption of the solid fermentation process for this project was abandoned. Among solid culture methods, there are other processes to produce citric acid by fermentation which use carriers to provide the porous beds for aeration, with carbohydrate raw material mixed into the carriers. One possible combination which could be considered would be to use bagasse as a carrier in combination with crude sugar, sugarcane

syrup or molasses. Some experiments have been conducted in a university laboratory and it was reported that citric acid was indeed produced by this solid culture fermentation process. However, there is no commercial plant using this process. Therefore, it cannot be used for this project.

11-3-2 Submerged Culture Method

The submerged culture process produces citric acid in fermentation tanks made of stainless steel. The tank is filled with liquid raw material and fungi are added. Sterilized compressed air is introduced into the tank which is agitated to maintain constant contact with the air for fermentation. The equipment of the submerged culture process is that of a modern chemical plant, consisting of raw material feed tanks, fermentation tanks, pumps, air compressors, heat exchangers, filters, valves and interconnecting pipework. Therefore, the investment cost of the plant for the submerged culture method is expensive compared with that of a solid culture process plant. A considerable amount of electricity will be consumed because motors are necessary to drive air compressors and agitators.

Various kinds of raw materials can be used in the submerged culture method, such as cornstarch, beet molasses, cane molasses, and sweet potatoes. A number of plants are in operation around the world using this process. Each plant owner develops proprietary fungi for his own use.

Operating plants using the submerged culture method are listed below according to the raw materials used.

- Cornstarch : Iwata Chemical Co., Ltd, Cargill, Pfizer, and Miles
- Beet molasses : Sturge
- Cane molasses : Bayer
- Sweet Potato : China (Vogelbusch)

Maize, the raw material for cornstarch, is harvested in large quantities in Zimbabwe, and Iwata Chemical Co., Ltd. is producing citric acid in Japan by the submerged fermentation process, using cornstarch.

Fermentation tests using two kinds of cornstarch made in Zimbabwe showed a yield of about 81% of total sugar, or a yield of about 90% of consumed sugar, which are almost the same as the yields for commercial production in Japan. Therefore, it is concluded that citric acid production by the submerged fermentation process using cornstarch is very promissing.

In Zimbabwe, the cost of electricity is also low. For these reasons, it is recommended that the submerged fermentation process should be adopted for this project.

11–4 Environmental Protection

This project is to produce citric acid through fermentation by microbiological reactions using cornstarch as the raw material. As neither toxic materials nor heavy metals are used, the proposed plant will have almost no effect on the environment.

The citric acid plant using the submerged fermentation process will create organic waste water from the separation process and waste gas from the steam boiler, which will require certain environmental protection measures. For the waste water treatment, an improved lagoon type process is to be installed, and for waste gas treatment, cyclones are to be furnished. Other wastes, such as mycelium and gypsum, will be used as much as possible as by-products.

11–5 Plant Site

As cornstarch has been selected as the raw material for citric acid production in this project, a maize collecting area, cornstarch producing area or adjacent areas will be preferable as a location for the plant site.

The following districts were visited by the study team during the field survey.

- Norton City : has large silos for storage of maize and is about 40 km south west from the center of Harare City.
- Chitungwiza City : about 20 km south from Harare City and one of its industrial suburbs.
- Mukuvisi area : about 10 km east from the center of Harare city.

As a result of the field survey, an estate next to the Zimbabwe Phosphate Industries site in the Mukuvisi district is considered best suited to be the site for the citric acid plant. A railway branch line covers to this district and can be used for the transportation of raw materials and citric acid. High voltage electricity lines run nearby, industrial water pipe lines and municipal sewage lines are already installed and all of these factors are considered as favorable for the selection of a plant site. Thus, this Mukuvisi district has been selected as the plant site for citric acid production.

11–6 Summary of the Project Scheme

The scheme for this project is summarized as follows:

- Plant capacity : 3,000 tons per year as citric acid monohydrate, complying with BP standards
- Raw material : Cornstarch made in Zimbabwe
- Process : Submerged culture method
- Plant site : Mukuvisi district, about 10 km east from the center of Harare city

The conceptual design, plant construction cost estimates, operating plan, financial analysis and so on will be provided for the abovementioned project scheme.

Chapter 12 Conceptual Design of the Plant

Chapter 12 Conceptual Design of the Plant

Described in this chapter is the conceptual design of a plant which produces 3,000 tons per year of high quality citric acid monohydrate by the submerged culture fermentation process using Zimbabwean cornstarch. Also referred to are basic technical matters relating to raw materials, utilities, and the fermentation, separation and refining processes required for the plant.

12–1 Outline of the Plant Construction Area

The site for the citric acid plant has been selected in an area adjacent to the Zimphos Company, located in Mukuvisi district, in the south eastern part of the city of Harare, as already described in Chapter 8. An outline of the plant construction area is described below.

(1) Location

Mukuvisi district is located about 10 km east from the center of the city of Harare and not far from Food & Industrial which is producing cornstarch. This district is well served by railway and road and is convenient for the transportation of basic raw materials, additional materials, fuel (coal) and products. The district is located close to a residential area, so it will be convenient for employees to travel to work. The city of Harare is in a region which produces maize, the raw material of cornstarch, and in the center of the citric acid consuming area.

(2) Climate

The climate of the city of Harare is shown below and will not present any problem for the construction and operation of a citric acid plant.

- Altitude : 1,472 meters
- Barometric pressure : 856 mb
- Temperature annual average : 18.1 °C
  maximum recorded : 35.4 °C (December, 1960)
  minimum recorded : −1.4 °C (June, 1968)
- Average wind velocity : 4 meters/second

- Annual precipitation : 820 mm
- Humidity (annual average) : 61 %
- Dry season : April to October
- Wet season : November to March

(3) Soil conditions

Soil in the Mukuvisi district is composed of coarse soil (gravel and coarse sand) and is firm. Zimbabwe is not in a seismic zone and has not experienced any earthquakes.

(4) Other aspects

In a suburb of Harare, there is the University of Zimbabwe which has a biochemistry department. Technical support on fermentation matters can be expected from the University.

12–2 Assumptions for the Plant Design

The conceptual design of the plant is based on the submerged culture fermentation technology used in a commercial plant in Japan for which cornstarch is the raw material. The project will use Zimbabwean cornstarch as the main raw material and so, the conceptual design of the plant was carried out based on the results of the fermentation tests mentioned in Chapter 10. Since this project will be the first full scale industrial application in Zimbabwe of aerobic fermentation technology using a fungus, the design is based on batch operation and a philosophy of easy operation, maintenance and administration. The plant is designed for production only and will not have a research and development organization. The following are the major assumptions used for the conceptual design of the plant.

(1) Plant capacity

The plant will produce 3,000 tons per year of citric acid monohydrate by operating 24 hours per day and 333 days per year. The inventory of raw materials and products is as follows:

- Domestic raw materials : 5 days supply
- Imported materials : 2 to 4 weeks supply
- Product : 4 weeks production

(2) Fungus

Aspergillus niger fungus which is suitable for submerged culture fermentation using cornstarch.

(3) Raw material

The raw material to be used is STARCON which gave the better fermentation test results of the two Zimbabwe cornstarches (brand names: STARCON and STARTEX–45). STARCON contains more impurities such as protein than Japanese cornstarch but it contains more starch (refer to Table 12–1). The material balance and consumption of raw materials and utilities in the conceptual design of the plant is based on 99.7 percent sugar content and 89.7 percent of starch as shown in Table 12–1.

Table 12–1 Analysis Results of Cornstarch

	STARCON	Japanese
Water Content, %	4.8	12.9
Sugar Content, %	99.7	95.0
Starch Value, %	89.7	85.5
Protein, %	0.58	0.28
Lipid, %	0.063	0.050
Electrical Conductivity, μS/cm	218	100
Ash, %	0.16	0.03

Among other materials required for producing citric acid, sulfuric acid and some nutrients produced in Zimbabwe can be used, but other chemicals and subsidiary materials which must meet required specifications will have to be imported from abroad. Major raw materials used for the citric acid production and the method of packaging are as follows:

(a) Fermentation process

- Cornstarch : Container bag
- Enzyme amylase : Can
- Ammonium nitrate : Bag
- Potassium phosphate : Bag

(b) Separation process

- Slaked lime : Container bag
- Sulfuric acid : Tank truck
- Activated carbon : Bag
- Filter aid : Bag

(4) Product

The quality of citric acid produced by this plant should satisfy British Pharmacopoeia specifications shown in Table 12-2. The product is to be sold after packing in 50 kg bags made of 4 layers of paper and 1 layer of polyethylene sheet.

Table 12-2 Specification of Citric Acid Monohydrate in UK (British Pharmacopeia)

Items	Specifications
Characteristics	Colorless crystals or a white, crystalline powder; efflorescent
Solubility	Soluble in less than 1 part of water and in 1.5 parts of ethanol (96%); sparingly soluble in ether
Clarity and Color of Aqueous solution	Within the limit
Barium (Ba)	Within the limit
Calcium (Ca)	Not more than 200 ppm
Heavy metals (Pb)	Not more than 10 ppm
Iron (Fe)	Not more than 50 ppm
Chloride (Cl)	Not more than 50 ppm
Oxalate ($C_2H_2O_4$)	Not more than 350 ppm
Sulphate (SO_4)	Not more than 150 ppm
Readily carbonisable substances	Within the limit
Sulphated Ash	Not more than 0.1%
Water (including crystallization water)	7.5 ~ 9.0%
Content	99.5 ~ 101.1%

(5) Electric power

Electricity is supplied by ZESA (Zimbabwean Electricity Supply Authority) through its electricity supply network. The city of Harare, the capital of Zimbabwe, is supplied by 3 electric power lines, in addition to having one power station in the city. Therefore, the electricity supply is very good. In view of the reliable electric supply, the plant does not have to have its own electric generator.

(6) Feed water and waste water

The plant is designed to use public utility water for the production process. Cooling water is to be based on a primary cooling water temperature of 22°C and a secondary cooling water temperature of 28°C. In order to conserve water, it is to be recycled through a cooling tower. Waste water is to be discharged to the public sewage system after treating to satisfy environmental regulations.

(7) Instrumentation and control

Automatic control is to be adopted for important processes, otherwise the plant will be operated manually as far as possible.

12–3 Outline of the Production Process of Citric Acid

12–3–1 Outline of the Fermentation Process

For citric acid fermentation, a pure strain of citric acid generating fungus is cultured in a laboratory and is then planted and cultured in three different sizes of fermenters in sequence. In this way citric acid accumulates in the liquid culture medium in the main fermenters on an industrial scale. In the fermentation process, the important operations are sterilization of raw materials by steam and prevention of contamination by various fungi during the fermentation process. A characteristic of this process is that heat is generated as fermentation progresses and a considerable amount of water is required for cooling. Details of the fermentation process are described below.

(1) Liquefaction of cornstarch

28,540 kg of cornstarch, the amount required for fermenting one batch of citric acid, is introduced into the make-up tank and a 30 percent starch milk solution is prepared by adding water. The enzyme amylase is added to the starch milk solution to promote liquefaction, and then steam is directly introduced through a jet cooker and the milky solution is heated to 90°C. It is then transferred to the reaction tank where the starch is liquefied by the action of the enzyme at high temperature.

(2) Flask seed culture

A milky solution is prepared by mixing 50 grams of cornstarch, 0.05 grams of the enzyme amylase for liquefaction and 800 m ℓ of water. After the starch is liquefied at a temperature of 90°C, nutrients are added and water is added to bring the volume of the solution to 1 liter. 330 m ℓ is poured into each of three 2 liter Erlenmyer flasks and sterilized in an autoclave at 121°C for 20 minutes and then cooled to ambient temperature. Aspergillus niger is planted in this medium and culture proceeds for 45 hours at 35°C using a rotary shaker.

(3) First seed fermentation

48 kgs of a solution of liquefied cornstarch and nutrients are introduced to the First Seed Tank and fermentation medium is prepared by adding water to give a volume of 680 liters. After being sterilized for 20 minutes at 121°C by injecting steam, the medium is cooled. After sterilization, the volume of medium in the First Seed Tank is increased to 800 liters. The seed in the three Erlenmyer flasks mentioned in (2) above is planted in the sterilized medium in the tank and the medium is fermented to breed seed fungus under the following conditions:

- Medium volume before planting the seed : 800 ℓ
- Seed volume : 990 m ℓ
- Fermentation temperature : 35 °C
- Aeration flow : 240 ℓ /minute
- Agitation : 160 rpm
- Fermentation period : 36 hours

(4) Second seed fermentation

6,743 kgs of a solution of liquefied cornstarch and nutrients are introduced into the Second Seed Tank and a fermentation medium is prepared by adding water to give a volume of 9.4 k ℓ. After being sterilized for 20 minutes at 121°C by injecting steam, the medium is cooled. After sterilization, the volume of medium in the Second Seed Tank is increased to 11.2 k ℓ. The seed in the First Seed Tank mentioned in (3) above is planted in the sterilized medium in the tank and the medium is fermented to breed seed fungus under the following conditions:

- Medium volume before planting seed : 11.2 k ℓ
- Seed volume : 800 ℓ
- Fermentation temperature : 35 °C
- Aeration flow : 3.6 m^3/minute
- Agitation : 100 rpm
- Fermentation period : 36 hours

(5) Main fermentation

Hot water and nutrients are introduced into the Medium Make-up Tank to give a volume of 10 k ℓ. The nutrient solution in the Medium Make-up Tank, the liquefied cornstarch solution in the Reaction Tank and the hot water are sterilized in a continuous sterilizer in this sequence at a flow-rate of 10 k ℓ / hour at 121°C for 5 minutes, and after cooling to 35°C, these solutions and the water are introduced into the Main Fermenter which has been sterilized beforehand. The flow of sterilized water into the main fermenter is stopped when the volume of the sterilized fermentation medium in the main fermenter reaches 168 k ℓ. After completing the series of operations described above, the seed produced in the Second Seed Tank is planted in the medium in the main fermenter and fermentation takes place under the following conditions:

- Medium volume before planting the seed : 168 k ℓ
- Seed volume : 12 k ℓ
- Fermentation temperature : 35 °C
- Aeration flow : 54 m^3/minute
- Agitation : 100 rpm
- Fermentation period : 160 hours

(6) Tank operating cycle

In this plant there are 3 Main Fermenter units, one First Seed Tank and one Second Seed Tank. The Main Fermenters are operated on an operating cycle which produces 3 batches every 7 days. Both the First and Second Seed Tanks are operated at intervals of 48 hours or 72 hours to suit the operating cycle of the Main Fermenters.

12–3–2 Outline of the Separation Process

The basic principle of the citric acid separation process is that citric acid is first crystallized and separated as calcium citrate which has low solubility. Then the separated calcium citrate is decomposed by sulfuric acid to give a citric acid solution which is concentrated to obtain citric acid crystals. In the industrial process, mycelium separation, decolorization, drying and so on are also necessary. Details of the separation process are explained below.

(1) Transfer of the fermented broth

After fermentation is completed, the whole contents of the Main Fermenter are transferred to the Broth Tank

(2) Filtration of the mycelium

Filter aid is coated onto the mycelium filter and the mycelium is separated from the broth by vacuum filtration. The small amount of mycelium remaining in the filtrate is completely removed in a filter press and transparent filtrate is obtained. The separated mycelium is discharged as a waste product.

(3) Crystallization of calcium citrate (neutralization)

About 14 k ℓ of the filtrate solution and about 0.6 k ℓ of secondary mother liquor are introduced into the Neutralization Tank and heated to 50°C by steam. A slurry of slaked lime which has been previously adjusted to 30 percent concentration is fed to the Neutralization Tank and mixed until a pH of 5.0 is reached. In the neutralization process, free citric acid is converted to calcium citrate which crystallizes due to its low solubility. The neutralization process is operated at the rate of six lots a day.

(4) Separation of calcium citrate

The slurry containing crystallized calcium citrate is transferred to a Nutche type vacuum filter, where crystals are separated form the solution. The crystals separated in the filter are washed by a water to remove solution adhering to the crystals. The separated calcium citrate is transferred to the acidification process. Filtrate and washing water are transferred to the waste water treatment process as waste water.

(5) Acidification of calcium citrate

Calcium citrate is charged into the Acidification Tank and mixed with diluted filtrate obtained from a subsequent process and adjusted to give a calcium citrate slurry with a concentration of approximately 24%. Then, by adding the chemically equivalent amount of sulfuric acid, the calcium citrate is converted to a slurry consisting of free citric acid and crystallized calcium sulfate.

(6) Separation of free citric acid and calcium sulfate

Decomposed slurry is transferred to a bucket type centrifuge and free citric acid is separated from the calcium sulfate. The separated calcium sulfate is washed with water. A small amount of citric acid remains in the diluted filtrate, so in order to recover this citric acid, the diluted filtrate is recycled to the acidification process mentioned in (5) above. The small amount of calcium sulfate crystals remaining in the citric acid solution is removed completely by a filter press and clear citric acid solution is obtained. The separated calcium sulfate is discharged as the by-product gypsum.

(7) First evaporation of citric acid

The citric acid solution obtained as described in (6) above is then mixed with recycled first mother liquor, second stage crude citric acid crystals and the final mother liquor. The resulting solution is then continuously evaporated under vacuum until the citric acid solution becomes 1,150 g/ℓ (as monohydrate).

(8) First crystallization of citric acid

The concentrated solution mentioned in (7) above is transferred to the First Crystallizer and cooled down while agitating. When the temperature of the solution becomes 35°C, seed crystals of citric acid monohydrate are added to initiate crystallization. The solution is further cooled below 20°C, and crystals of citric acid monohydrate are produced and separated.

(9) Separation of the first crude citric acid monohydrate

The slurry of citric acid monohydrate crystals is sent to a bucket type centrifuge where the first crude citric acid monohydrate and the first mother liquor are separated. The separated first crude citric acid monohydrate is sent to the decolorization process. The first mother liquor is stored in the First Mother Liquor Tank, and then sent back to the first evaporation process and second crystallization process where remaining citric acid is recovered.

(10) Recovery of second citric acid crystals

40% of the first mother liquor obtained from a process (9) mentioned above is evaporated under vacuum until the concentration of citric acid reaches 1,200 grams per liter as monohydrate, and is then transferred to the second crystallizer. In the second crystallizer, the concentrated solution is cooled while agitating. When the solution temperature reaches 35°C, a small amount of seed crystals is added to crystallize the citric acid. The solution is further cooled below 20°C and citric acid monohydrate is crystallized and separated. The slurry of citric acid monohydrate crystals is sent to a bucket type centrifuge where the second crude citric acid crystals and the second mother liquor are separated. The separated second crude citric acid crystals are sent back to the first evaporation process. The second mother liquor is stored in the Second Mother Liquor Tank, and then sent back to the calcium citrate crystallization (neutralization) process, where citric acid is recovered.

(11) Decolorization of citric acid solution

The crude crystals of citric acid obtained as described in (9) above and the final mother liquor are charged into the Decolorization Tank and the solution is adjusted to give a citric acid concentration of 1,150 grams per liter as monohydrate by adding diluted filtrate obtained in the subsequent process. The solution is heated to 80°C and the crystals are desolved. Activated carbon is then added to decolorize the solution which takes 30 minutes.

(12) Filtration of citric acid

The carbon filter is coated with filter aid and the decolored citric acid solution is filtered at a high temperature. The filtrate is then filtered by a polishing filter, and sent to the Final Crystallizer. The filtered activated carbon is washed by water using a volume equal to 10% of the decolored solution. Diluted filtrate from the washing operation is sent back to the decolorization process to prevent loss of citric acid. The separated activated carbon is discharged as Waste Carbon.

(13) Final crystallization of citric acid

The decolored filtrate is cooled while agitating. When the solution temperature reaches 35°C, a small amount of seed crystals is added to crystallize the citric acid monohydrate. The solution is further cooled below 20°C and crystals of citric acid are formed and separated.

(14) Separation of final crystals of citric acid

The slurry of crystallized citric acid monohydrate is sent to a bucket type centrifuge, where the final citric acid crystals and the final mother liquor are separated. The separated citric acid crystals are sent to the drying operation. The final mother liquor is stored in the Final Mother Liquor Tank, from where it is sent to the decolorization process and first evaporation process for recovery of citric acid.

(15) Drying of citric acid

Since the separated citric acid monohydrate contains about 3% of free water, it must be dried. The citric acid crystals are sent continuously by conveyor to a rotary type dryer unit where they are dried by hot air at about 60°C for removing free water. The dried citric acid is sent to the Storage Hopper by an air lift.

(16) Packaging of citric acid

Dried citric acid is packed in 50 kg bags after off-size citric acid has been removed by the Sifter.

12–3–3 Flow Sheet

Shown in Figure 12–1 is a simplified Block Flow Diagram of the total process of citric acid production. Figure 12–1 shows the consumption of raw materials and subsidiary materials as well as the amounts of waste liquid and waste solid discharged, during the production of 1,000 kg of citric acid monohydrate. Figure 12–2 shows the material balance for the entire process. Figure 12–2 shows the balance for a single batch of the fermentation process based on 3 batch operations every 7 days. As for the separation process, the figures indicate the number of days required and the product obtained by fermentation of 3 batches every 7 days. Figure 12–3 is the process flow sheet. Equipment numbers and names are shown for major equipment and equipment numbers only are shown for auxiliary equipment. *Equipment numbers and names refer to items on the "Equipment List" in Chapter 13.*

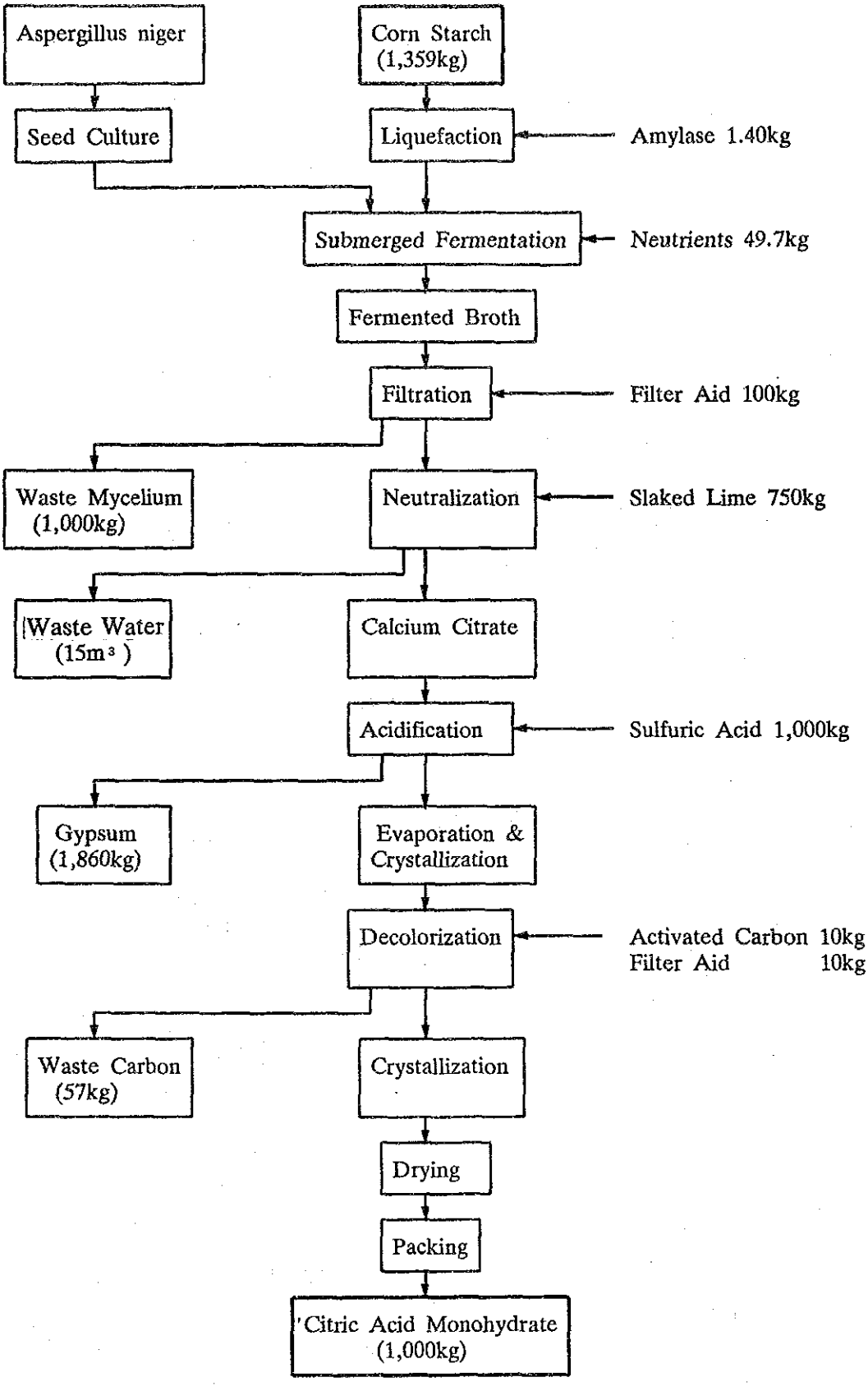
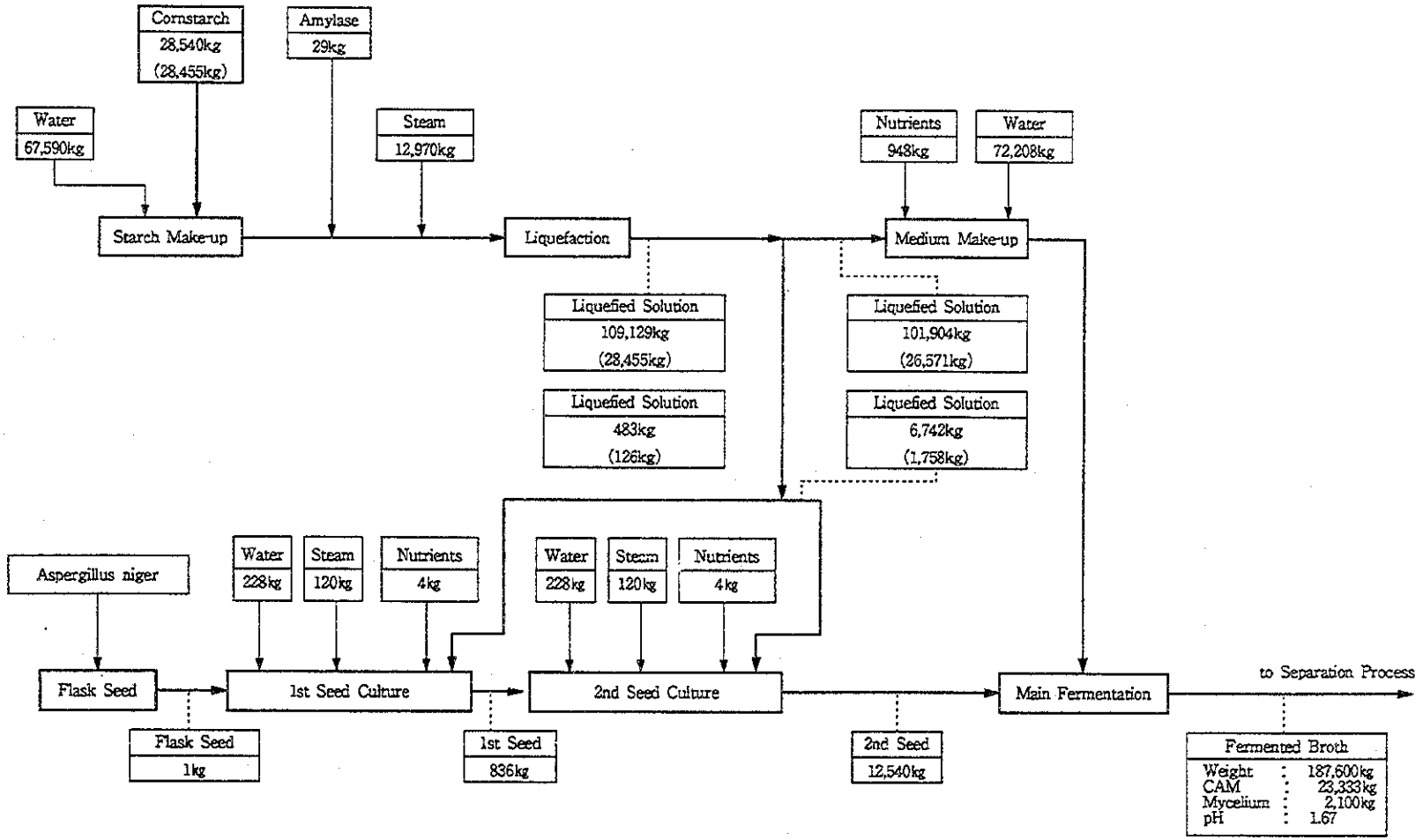


Figure 12-1 Process Block Diagram



Note : Figures in parenthesis are glucose amount.

Figure 12–2–1 Material Balance (per Batch : 3 Batch/7 Days), Fermantation Process

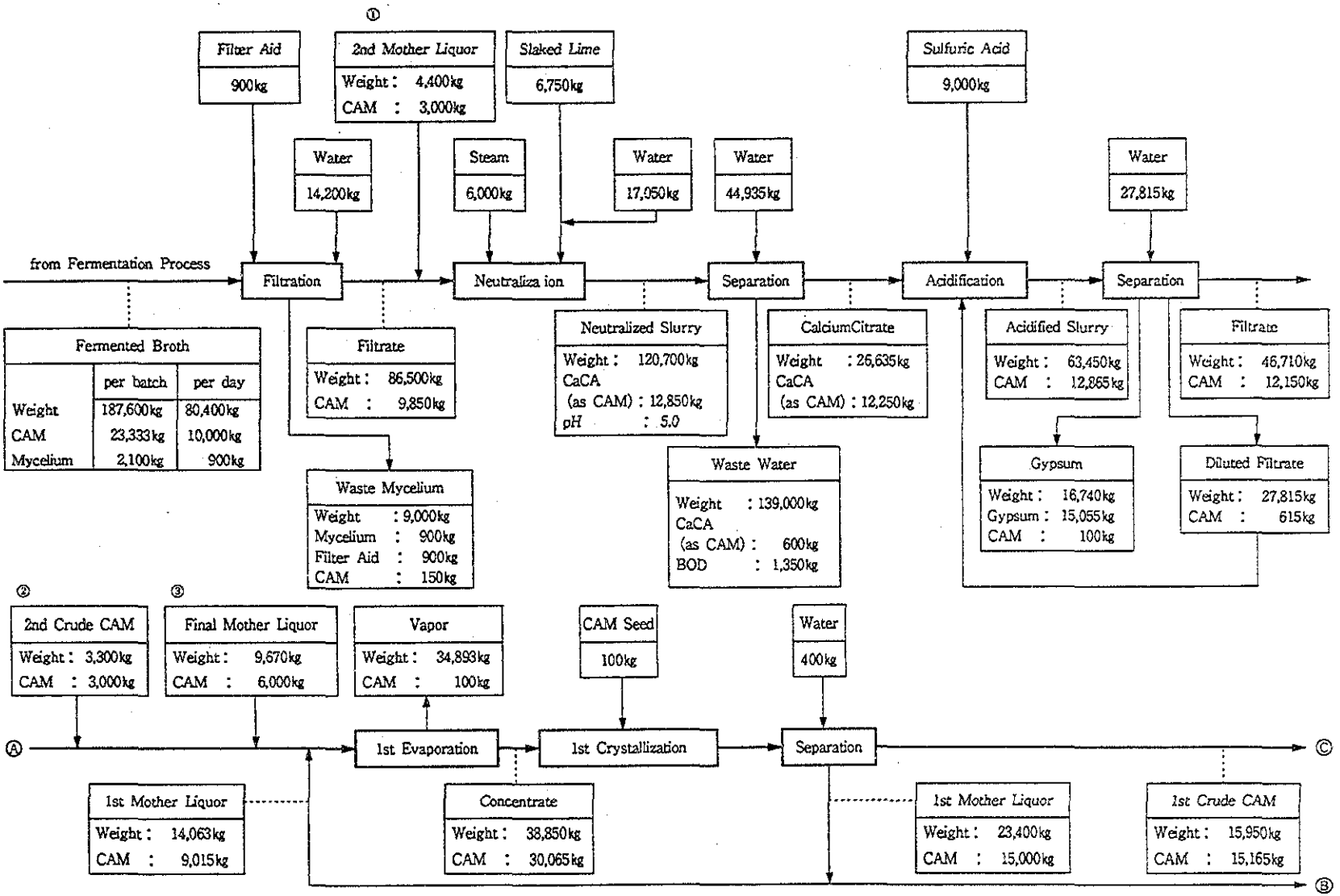


Figure 12–2–2 Material Balance (per Days), Separation Process (1/2)

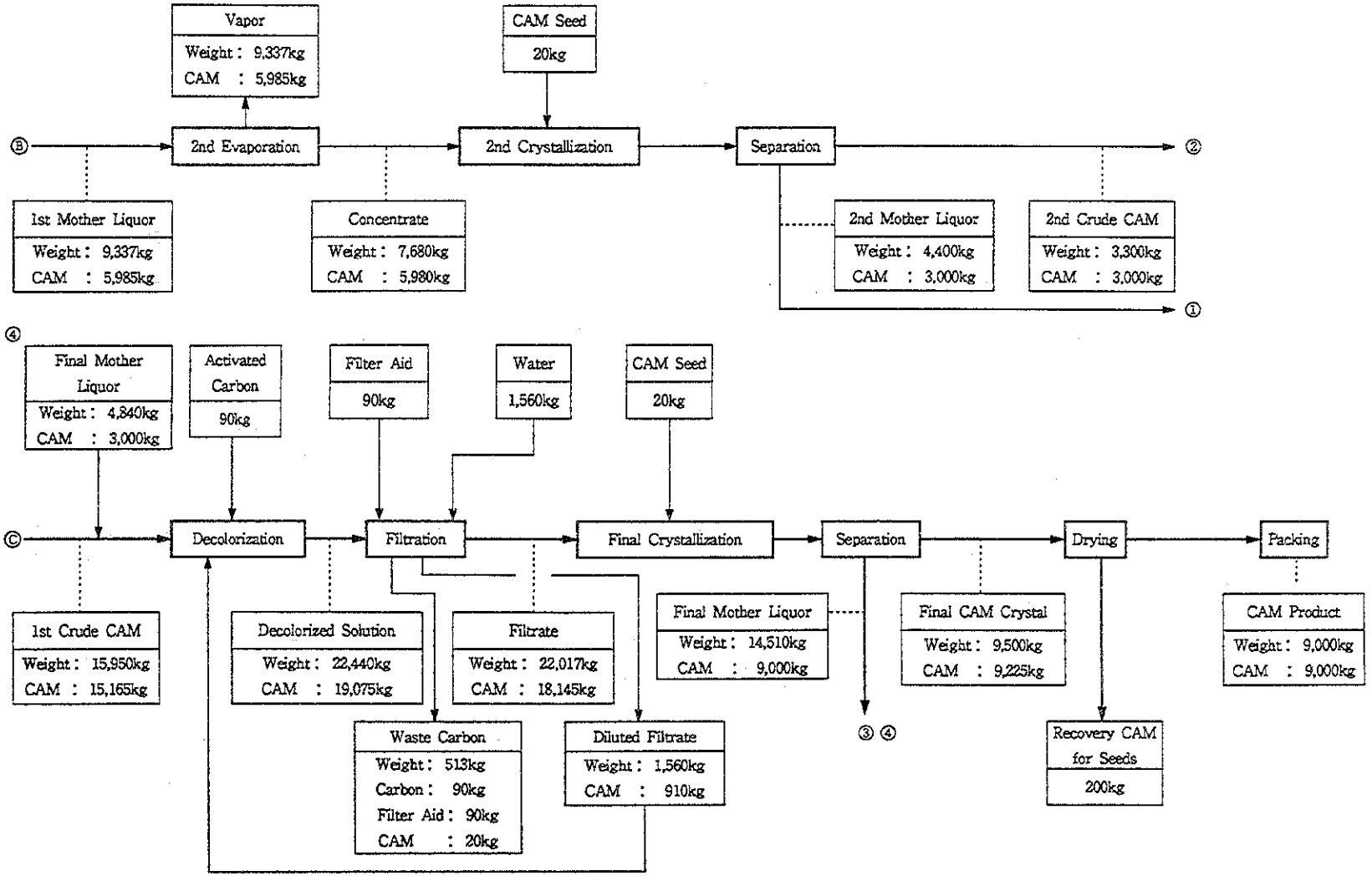


Figure 12–2–3 Material Balance (per Days), Separation Process (2/2)

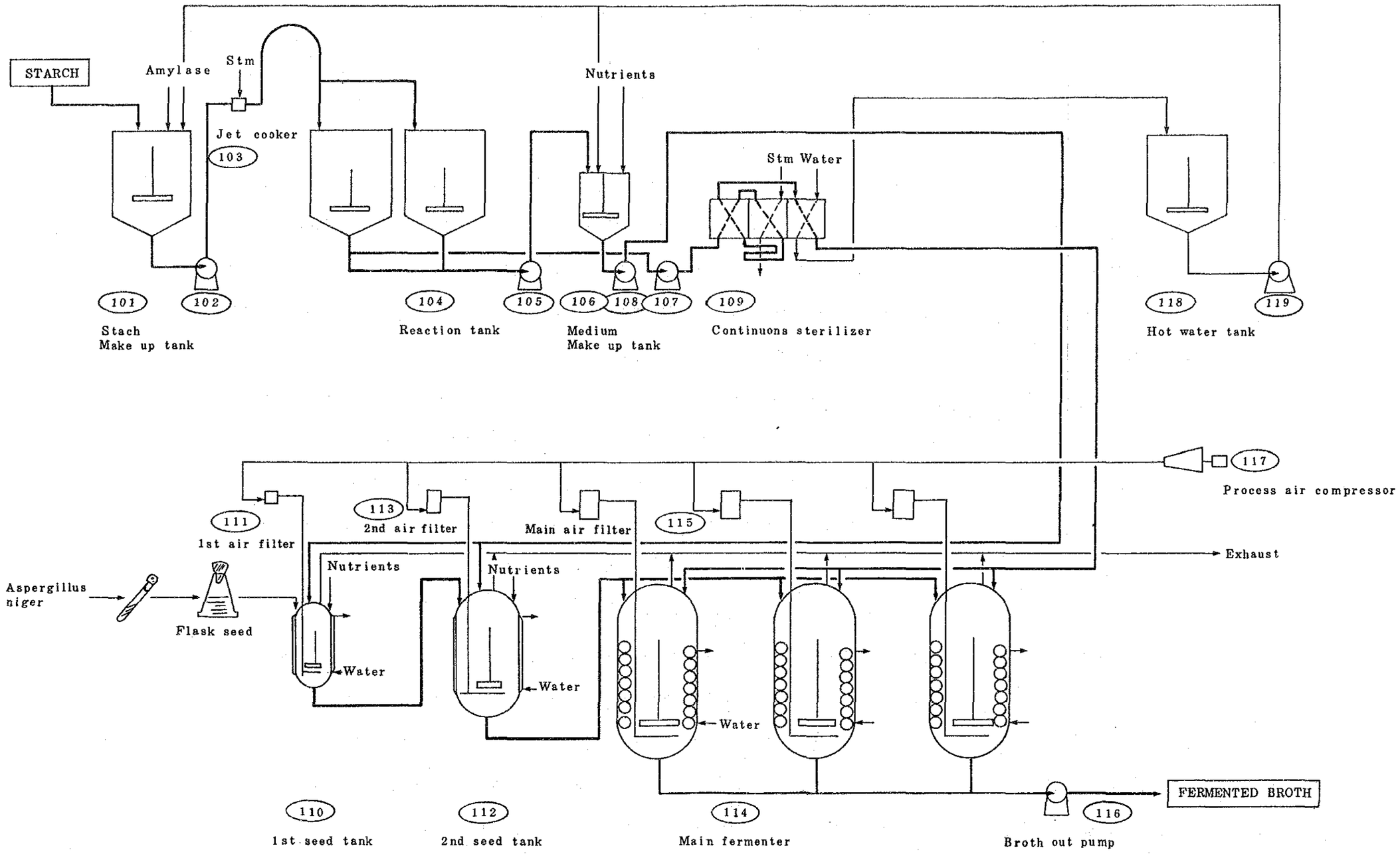


Figure 12–3–1 Process Flow Sheet (Fermentation Process)

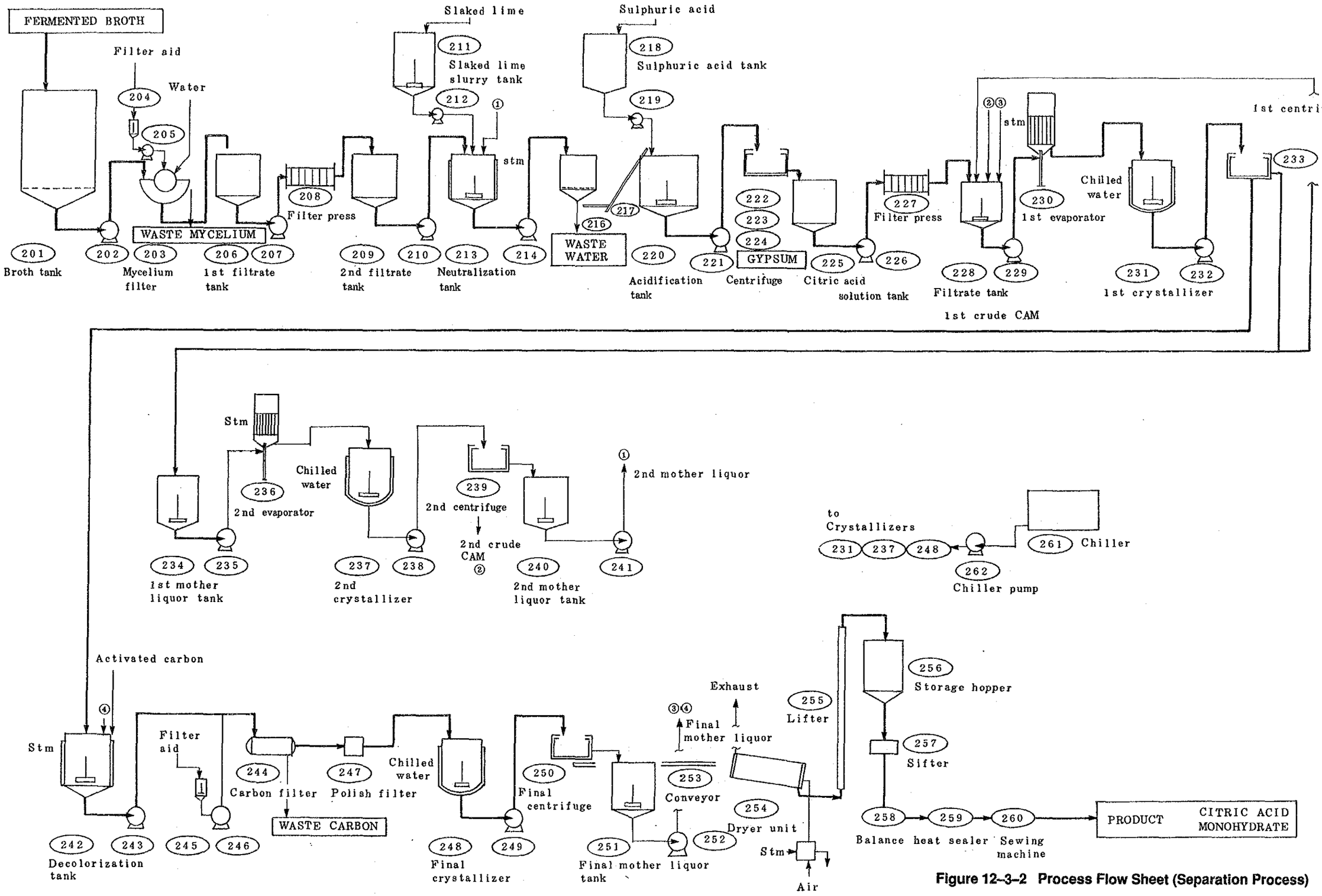


Figure 12–3–2 Process Flow Sheet (Separation Process)

12–3–4 Raw Materials

(1) Cornstarch

As described in "Assumptions for the Plant Design" in section 12–2, STARCON made in Zimbabwe will be used as the raw material.

(2) Sulfuric acid

Concentrated sulfuric acid made by Zimphos will be used. Specification of the Zimphos sulfuric acid is for a concentration of 98% or more and the impurities must be less than 5 ppm of heavy metal as Pb, less than 2 ppm as As_2O_3, less than 10 ppm of Cu, less than 30 ppm of Fe, and less than 1 ppm of Cd.

(3) Slaked lime

It is desirable that the slaked lime used for this process should contain the smallest amounts possible of Mg, Fe and SiO_2 as impurities. In particular, a high Mg content in the slaked lime reduces the recovery rate of citric acid. Therefore slaked lime containing a low Mg content is required. Slaked lime made in Zimbabwe contains a large amount of impurities as mentioned in Chapter 7 and cannot be used for this project. This feasibility study is based on slaked lime produced in Zambia (Ndola Lime containing 95% $Ca(OH)_2$, 0.8% MgO, 0.07% Fe and 0.39% SiO_2). It is desirable to use better quality slaked lime.

(4) Activated carbon

The activated carbon used for declorizing citric acid must not contain impurities soluble in citric acid. In Zimbabwe, bone black is produced for the sugar refining industry, but the bone black contains a lot of ash and cannot be used as it does not meet this requirement. Activated carbon which has high decolorization characteristics and a low impurity content should be selected for the actual operation of the plant. As an example of an activated carbon suitable for citric acid production, the specification of an activated carbon made in the Republic of South Africa (trade name Kopcarb P7–45A) is shown in Table 12–3.

Table 12–3 Quality of Activated Carbon

Color & Form	Black Powder
Water Content, %	1 ~ 2
Ash, %	2 ~ 3
Iodine Number, mg/g	more than 700
Methylene Blue Number, mg/g	more than 120
Phenol Number AWWA	not more than 2.5
Particle Size (smaller than 45 μ), %	more than 80
Bulk Density, g/ℓ	320 ~ 370

(5) Filter aid

Appropriate filter aid is to be imported. As an example, the specification of Japanese filter aid is shown in Table 12–4.

Table 12–4 Quality of Filter Aid

Color & Form	White or Light–yellow Powder
pH	6.5 ~ 7.5
Water Content, %	less than 3
Water Solubility, %	less than 0.5
Bulk Density (non–dense), mℓ/g	more than 5
(dense) , mℓ/g	more than 3.3

Chemical Composition, %		Particle Size Distribution, %	
SiO_2	90	~ 50 μ	10
Al_2O_3	7	50 ~ 40 μ	4
Fe_2O_3	1	40 ~ 30 μ	6
CaO	0.9	30 ~ 20 μ	14
MgO	0.8	20 ~ 10 μ	41
		10 ~ 5 μ	20
		5 μ ~	5

(6) Enzyme amylase

Since amylase for liquefaction of cornstarch is not produced in Zimbabwe, it is to be imported.

(7) Nutrients

As nutrients for fermentation, ammonium nitrate, potassium diphosphate and several other minor nutrients (zinc, copper, magnesium, etc.) are required. Ammonium nitrate made in Zimbabwe is to be used and all others will be imported.

12–3–5 Unit Consumption of Raw Materials

The material balance is calculated using a 82% yield for fermentation and a 90% yield for the separation process, which gives an overall yield of 73.8%. The fermentation yield of 82% for citric acid from cornstarch (glucose) is based on the fermentation test results. The yield of the separation process is based on data from an actual commercial plant in Japan. Unit consumption of raw materials is shown in Table 12–5.

Table 12–5 Unit Consumption of Raw Materials

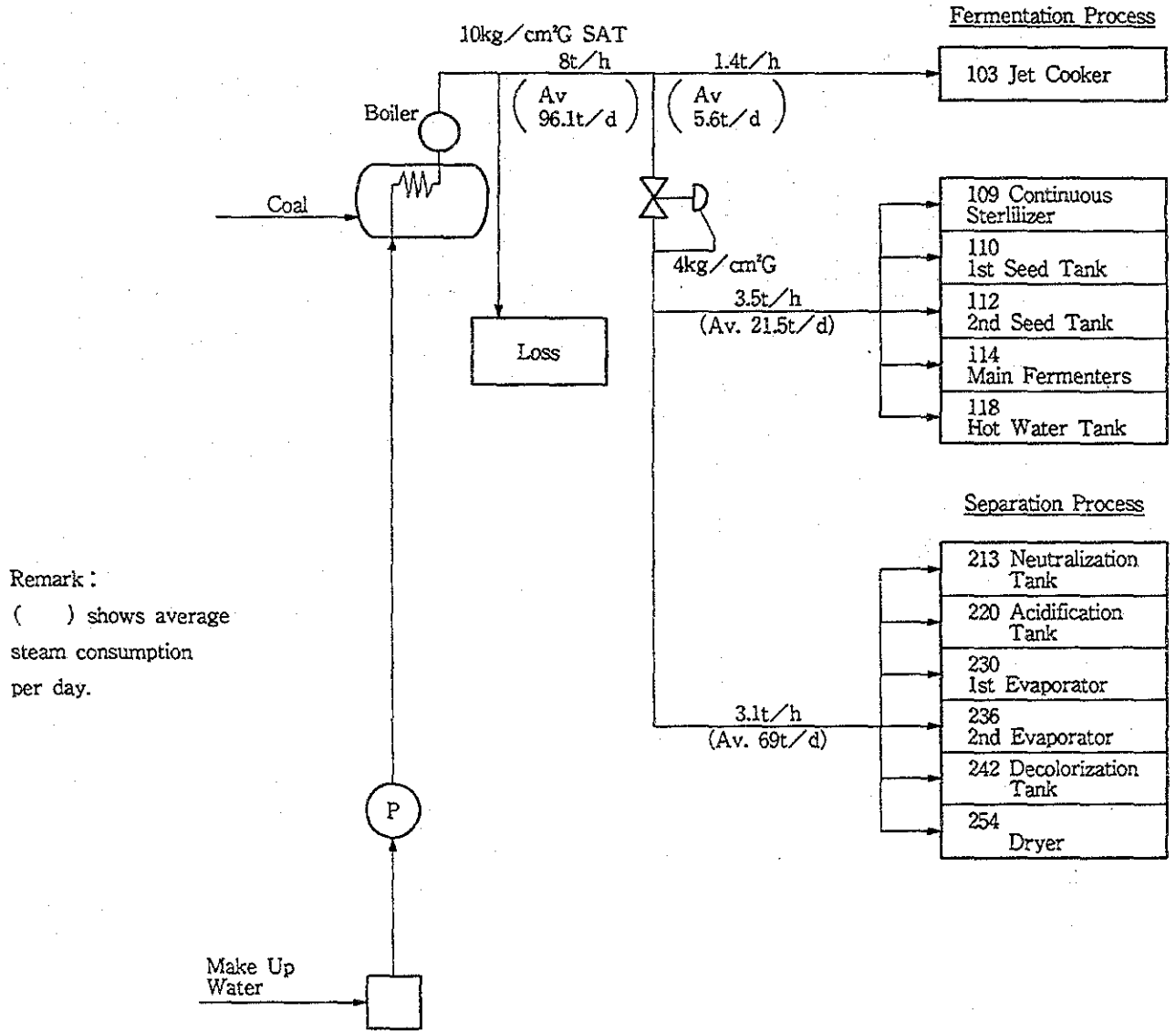
Raw Material	Unit Consumption (kgs per ton of CAM)
<<Fermentation Process>>	
Cornstarch	1,359
Ammonium Nitrate	29
Potassium Phosphate	17
Other Nutrients	3.7
<<Separation Process>>	
Slaked Lime	750
Sulfuric Acid	1,000
Activated Carbon	10
Filter Aid	110

12–3–6 Utilities

(1) Steam

Steam at 10 kg/cm^{2}G pressure is generated by a coal fired boiler and fed to the plant both as high pressure steam and also as low pressure steam after reducing the pressure. The high pressure steam is used only in the unit for liquefaction of cornstarch and all other units use low pressure steam. The steam balance is shown in Figure 12–4. Specification of the steam is as follows:

- High pressure steam : pressure : 10 kg/cm^{2}G
  temperature : Above 183°C
  (saturation temperature)
- Low pressure steam : pressure : 4 kg/cm^{2}G
  temperature : Above 151°C
  (saturation temperature)



Remark :
( ) shows average steam consumption per day.

Figure 12–4 Maximum Steam Consumption

Wankie coal is used as fuel for the boiler and washed Wankie coal has the following specification:

- Inherent water : 1.0 ~ 1.5%
- Ash : 11.0 ~ 14.0%
- Volatile matter : 23.0 ~ 26.0%
- Fixed carbon : 61.0 ~ 62.0%
- Calorific value : 29.0 ~ 31.0 MJ/kg (6,928 ~ 7,406 kcal/kg)
- Sulfur : 2.0 ~ 2.5%
- Phosphoric acid salt : 0.025 ~ 0.05%
- Melting point of ash : 1,270°C

(2) Water

Public utility water from McILwaine lake in the suburb of Harare will be supplied to the plant. The temperature of the water is relatively low throughout year. Water is a precious resource and is expected to be used effectively, so water will be recycled through a cooling tower. The results of the field survey show that water temperature can be maintained below 22°C after passing through the cooling tower even in the summer season. Based on this, the water balance will be as shown in Figure 12–5. The water pressure at the plant is designed to be 3 kg/cm²G. The quality of the municipal water in Harare is shown in Table 12–6.

Table 12–6 Quality of Municipal Water in Harare

pH	7.8	Calcium (Ca), mg/ℓ	55
Total Dissolved Solid, mg/ℓ	200	Magnesium (MgO), mg/ℓ	40
Total Alkali ($CaCO_3$), mg/ℓ	55	Sulfate (SO_4), mg/ℓ	55
Total Hardness ($CaCO_3$), mg/ℓ	85.0	Chloride (Cl), mg/ℓ	40
Free Ammonia (N), mg/ℓ	0.03	Fluoride (F), mg/ℓ	0.65
Magnesium (Mg), mg/ℓ	0.05	Phosphorous (P), mg/ℓ	0.01
Manganese (Mn), mg/ℓ	0.1	Silica (SiO_2), mg/ℓ	2
Copper (Cu), mg/ℓ	0.03	Iron (Fe), mg/ℓ	0.01
Aluminium (Al), mg/ℓ	0.05	Conductivity, μmho/cm	250

Source : Zimphos

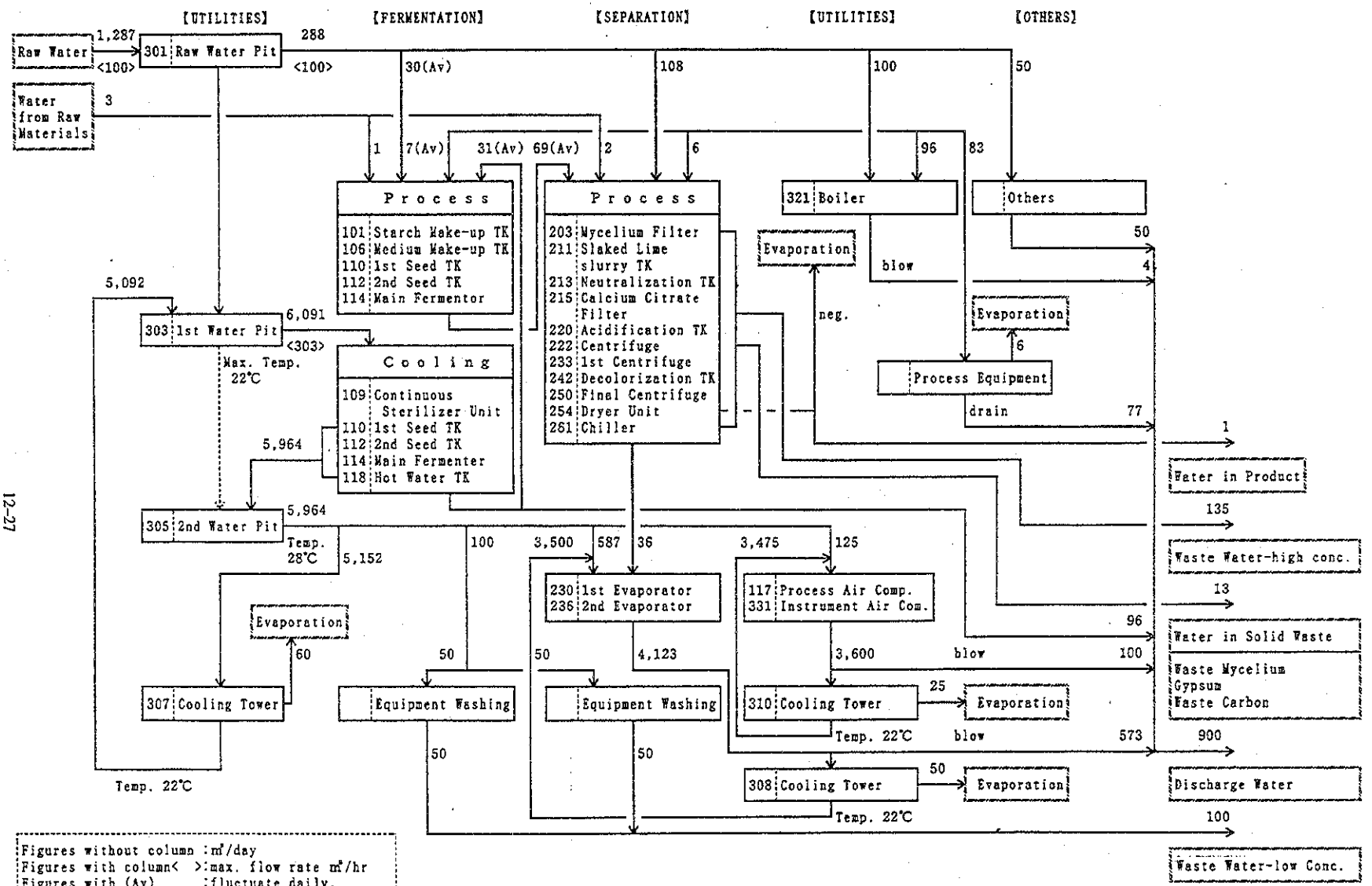
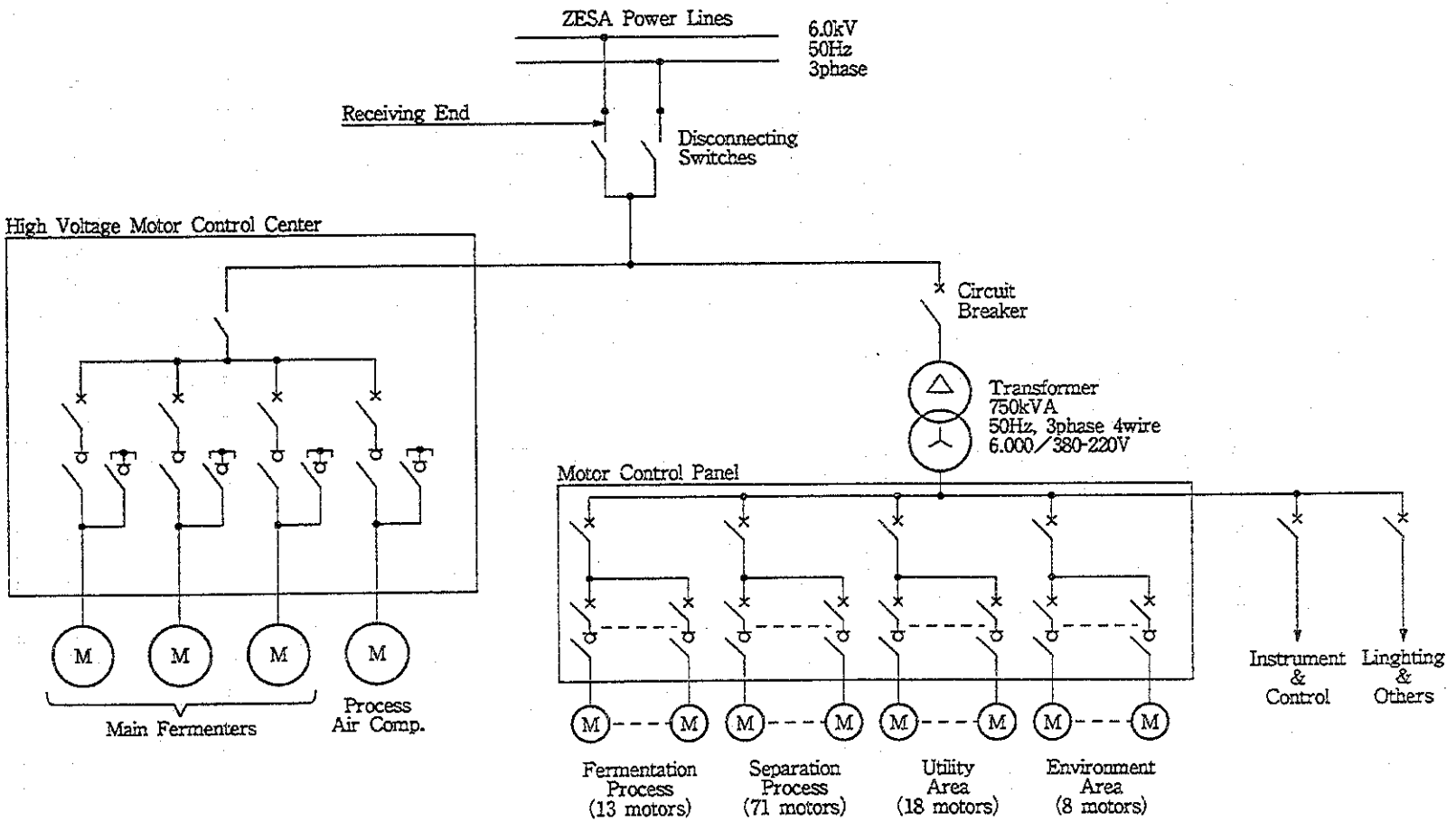


Figure 12–5 Schematic Diagram of Water Requirement

(3) Electricity

Powerful agitators and a large air compressor are required for the citric acid fermentation process. If power fails even for a short period of time the fungus in the fermentation process will change its nature. Therefore a reliable electricity supply is required. In the separation process, citric acid slurry must be kept in an agitated condition from the point of view of both steady production of citric acid and better maintenance of equipment. Again a stable supply of electricity is very important. Conceptual electrical schematic diagram is shown in Figure 12–6. Specification of the electricity supply is as follows:

- Incoming : 6,000V AC, 50 Hz, 3 phases
- Agitators for main fermenters and process air compressor : 6,000V AC, 50 Hz, 3 phases
- Other driving motors : 380V AC, 50 Hz, 3 phases
- Lighting : 200V AC, 50 Hz, 1 phase
- Instrument & Control : 200V AC, 50 Hz, 1 phase



	Fermentation Process	Separation Process	Utility	Environment	Instrument/ Lighting	(Total)
Estimated Installed Capacity	1,430 kW	300 kW	390 kW	160 kW	50 kW	2,300 kW
Estimated Demand	1,000 kW	115 kW	300 kW	80 kW	10 kW	1,500 kW

Figure 12–6 Electrical Schematic Diagram (Conceptual)

(4) Utility consumption

The utility requirements for producing one ton of citric acid monohydrate are coal: 1.2 tons, water: 145 m^3, electricity: 4,000 kWh.

12–3–7 Waste Water and Waste Materials

Shown in Table 12–7 are the kinds and quantities of waste water and waste materials discharged from citric acid production processes. Waste water and waste materials are also shown in Figures 12–1 and 12–2 and should be referred to. The waste water and waste materials are treated by the methods described in Chapter 9 "Environmental Protection".

Table 12–7 Waste Water and Solids from the Citric Acid Plant

	Volume		Remarks	
	per ton CAM	per year		
Waste Water, m^3				
High Concentration	15	45,000	BOD	10,000 mg/ℓ
Low Concentration	11.1	33,300	BOD	500 mg/ℓ
Waste Solids, tons				
Waste Mycelium	1.0	3,000		
Gypsum	1.86	5,580		
Waste Carbon	0.057	171		
Discharge Water, m^3	100	300,000	BOD less than 15 mg/ℓ	

12–4 Plot Plan

12–4–1 Plant Area

The space required for the plant is 150 meters × 210 meters and it is separated into areas for the administration office, production and utility facilities and environmental protection facilities. The layout is based on the following consideration:

(1) The office is located near the guard house so that outside people cannot easily get access to the production facilities and environmental protection facilities.

(2) For good plant security only one entrance is provided. The width of the entrance should be 15 meters for easy passage of raw materials and final product, people and vehicles.

(3) Production facilities and environmental facilities are located close to the office and the environmental protection facilities are separated according to their functions. The plant plot plan which takes all of the above into consideration is shown in Figure 12–7.

12–4–2 Plot Plan

The required areas and open spaces for the production processes and other facilities are given below:

(1)	Total required area for the plant	:	27,250 m^2 (excluding welfare facilities)
(2)	Production processes		
	– Fermentation process	:	1,325 m^2
			(including raw material storage area of 294 m^2)
	– Separation process	:	2,550 m^2
			(including product storage area of 294 m^2)
	– Boiler	:	646 m^2
(3)	Environmental protection facilities	:	3,456 m^2
(4)	Other facilities		
	– Maintenance building	:	361 m^2
	– Office	:	456 m^2
	– Guard house	:	20 m^2
	– Water facilities	:	1,075 m^2
	– Electrical substation	:	361 m^2
(5)	Parking lot	:	600 m^2
(6)	Roads	:	9,600 m^2
(7)	Open space, greenbelt	:	6,800 m^2

12–4–3 Equipment Layout

The main citric acid production processes are the fermentation process and the separation process. The equipment layouts for both processes are shown in Figure 12–8. Equipment numbers in Figure 12–8 correspond to items in the Equipment List described in Chapter 13.

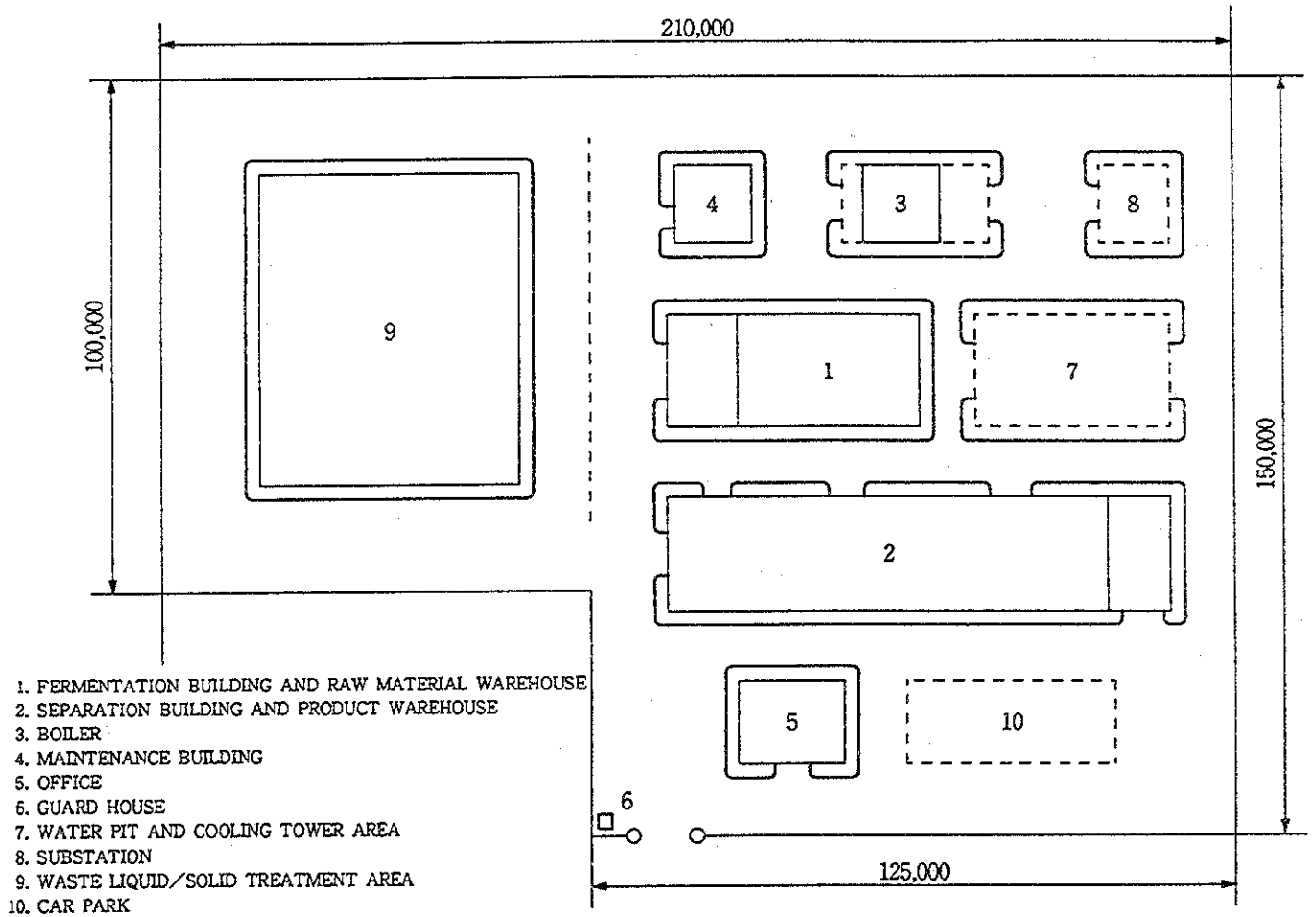
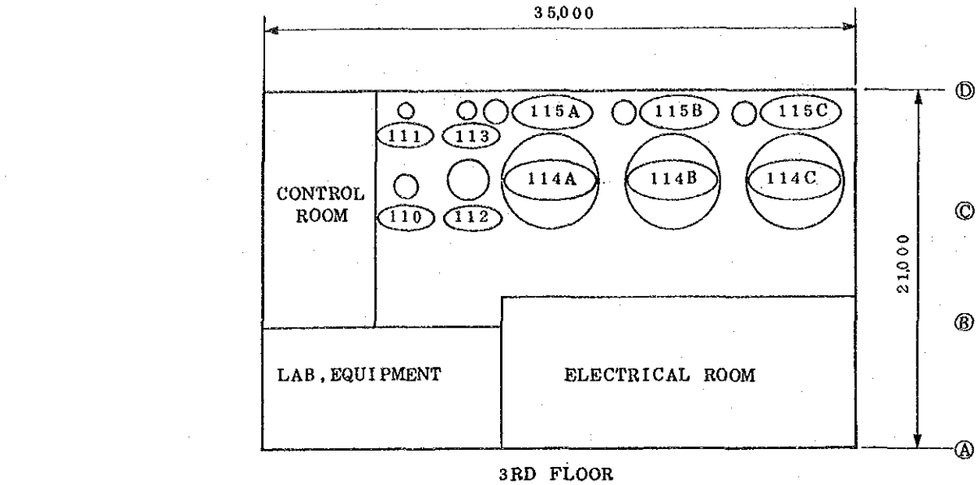
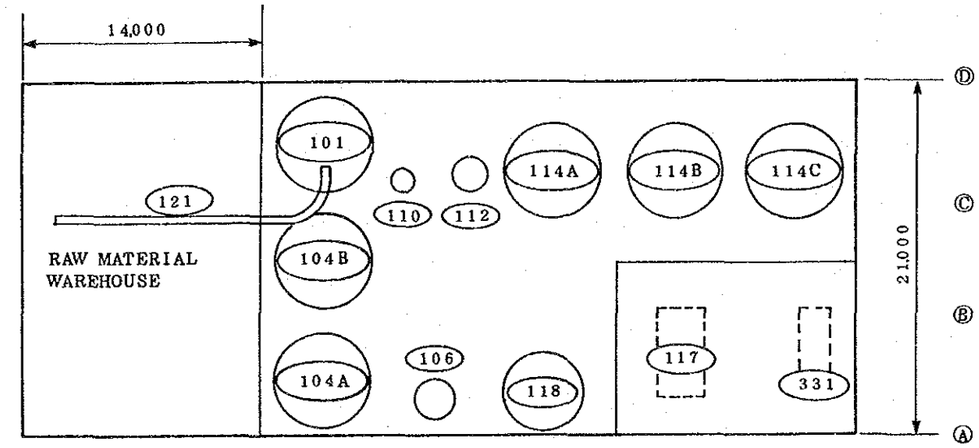


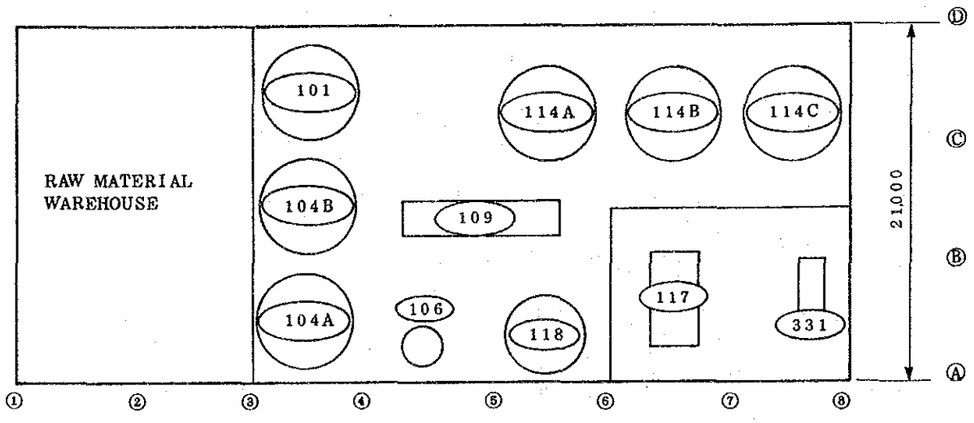
Figure 12–7 General Plot Plan



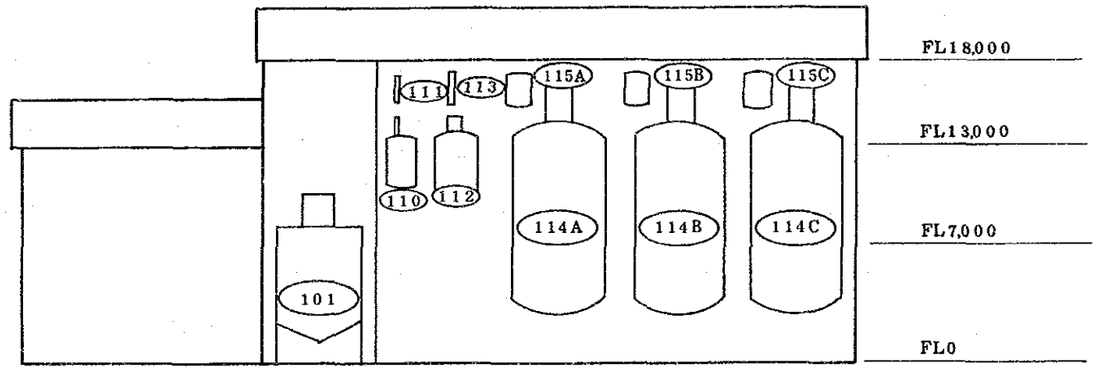
3RD FLOOR



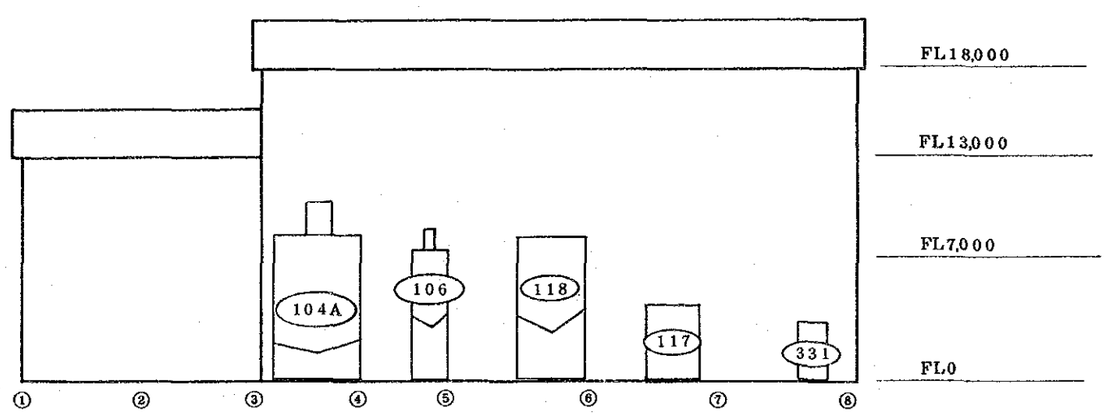
2ND FLOOR



1ST FLOOR

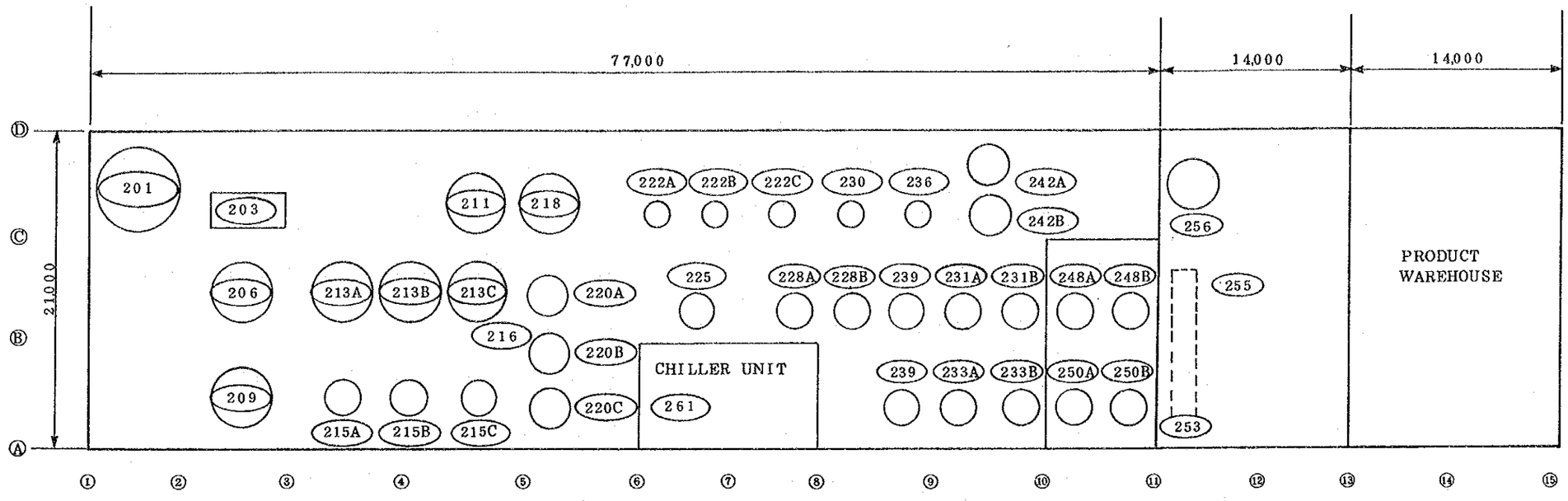


SECTION C



SECTION A

Figure 12–8–1 Equipment Layout, Fermentation Process



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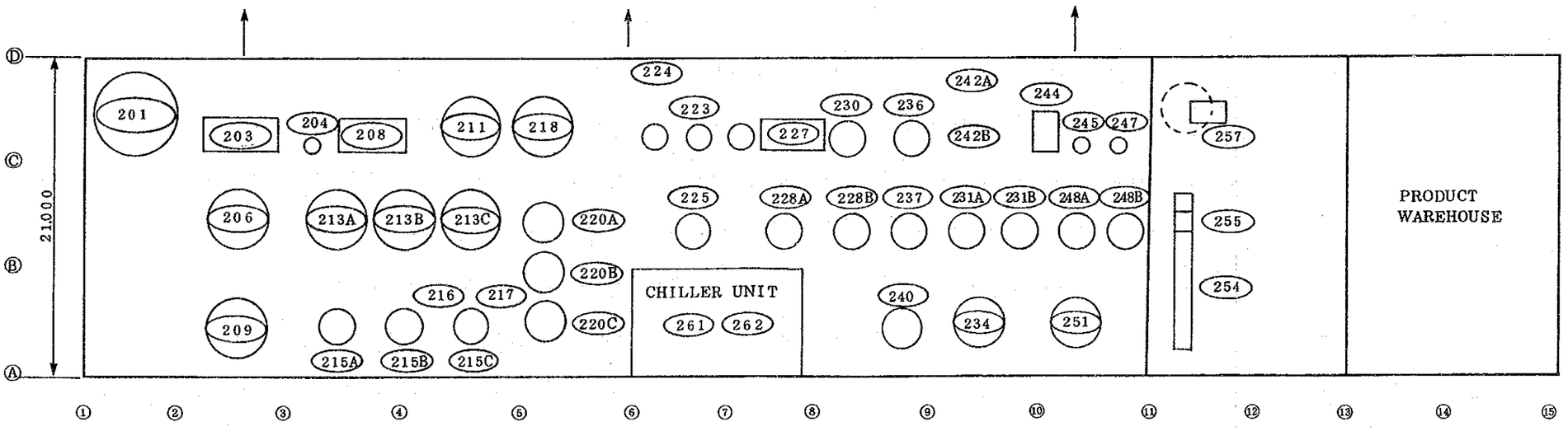
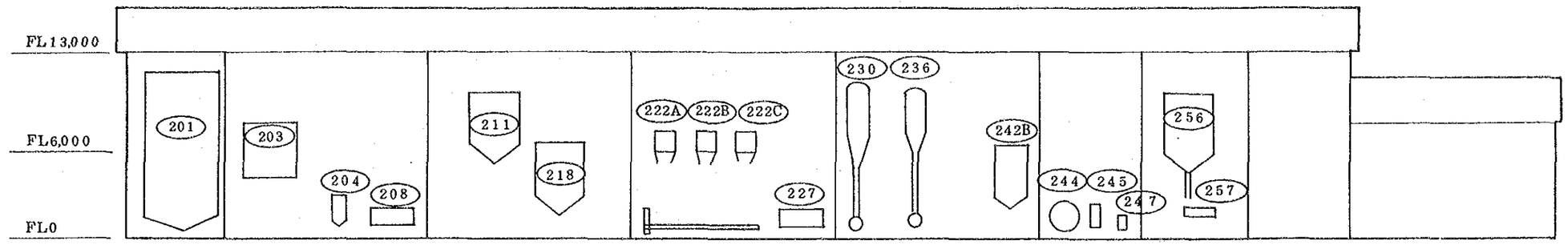


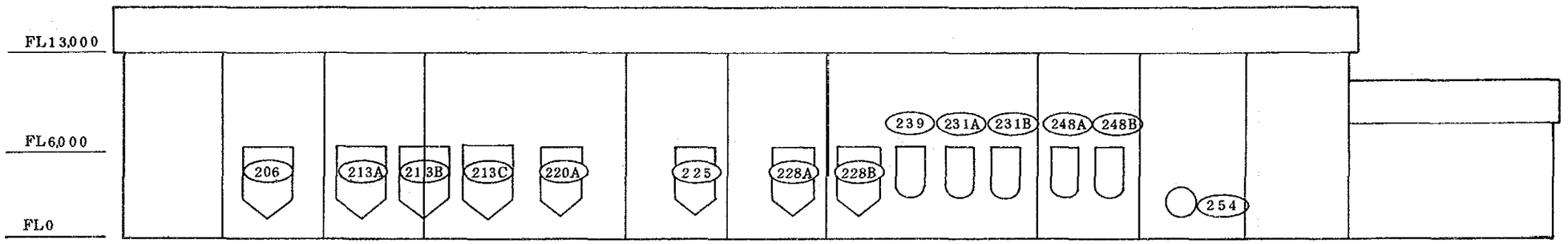
1ST FLOOR

WASTE LIQUID

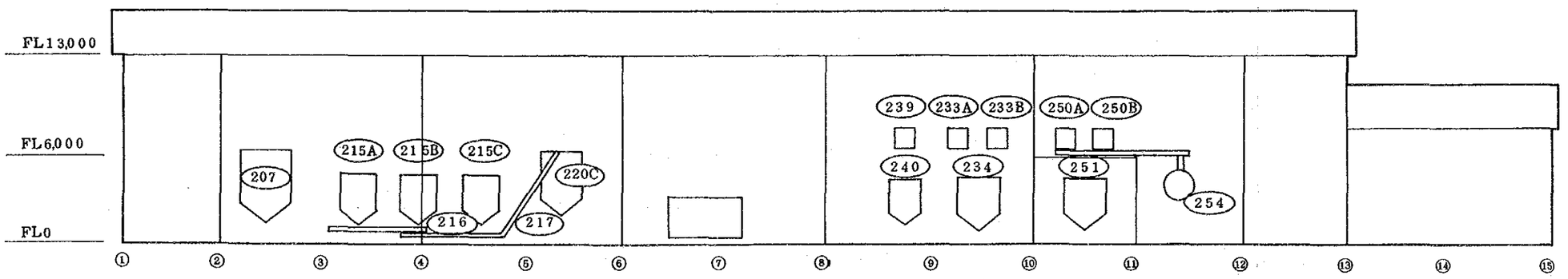
Figure 12–8–2 Equipment Layout, Separation Process (1/2)



SECTION C



SECTION B



SECTION A

Figure 12–8–3 Equipment Layout, Separation Process (2/2)

12–5 Plant Facilities

Citric acid production facilities are divided into 4 areas according to the process. The areas are as follows:

(1) Fermentation process facilities : area 100

(2) Separation process facilities : area 200

(3) Utility facilities : area 300

(4) Environmental protection facilities : area 400

Besides the above facilities, there is a laboratory. The laboratory equipment ranges from test tubes and flasks required for testing the fungi used for citric acid fermentation to equipment needed to analyze raw materials and products of the production processes.

12–5–1 Summary of Facilities

(1) Fermentation process facilities

This facility includes equipment required for the liquefaction of cornstarch and the equipment required for fermentation of citric acid consisting of 3 kinds of fermenters with aeration devices and mechanical agitators. Fermenters of 3 different sizes are used in sequence starting from pure fungi cultured in a laboratory. The fermenters require devices to sterilize the fermentation medium under biochemically sterilize conditions, and an air filter to remove dust and fungi from the feed air. The citric acid fermentation process generates heat, and therefore cooling equipment is designed into the fermenters.

(2) Separation process equipment

In the separation process, the following kinds of equipment are required to produce pure citric acid: fungus filtration, crystallization, separation, evaporation, dissolution, decolorization, filtration, drying, transferring and packaging units. The separation process via calcium citrate is unique amongst refining processes in the chemical industry. However, most of the equipment used in these processes are the same as these used for refining similar chemical products.

12–5–2 Outline Drawings for Major Equipment

This plant uses some special equipment and outline drawings are provided for the following items:

(1) Continuous Sterilization Unit

(2) First Seed Tank

(3) Second Seed Tank

(4) Main Fermenter

(5) First Air Filter

(6) Second Air Filter

(7) Main Air Filter

(8) Broth Tank

(9) Mycelium Filter

(10) Filter Press

(11) Carbon Filter

(12) Polishing Filter

(13) Calcium Citrate Filter

(14) Centrifuge

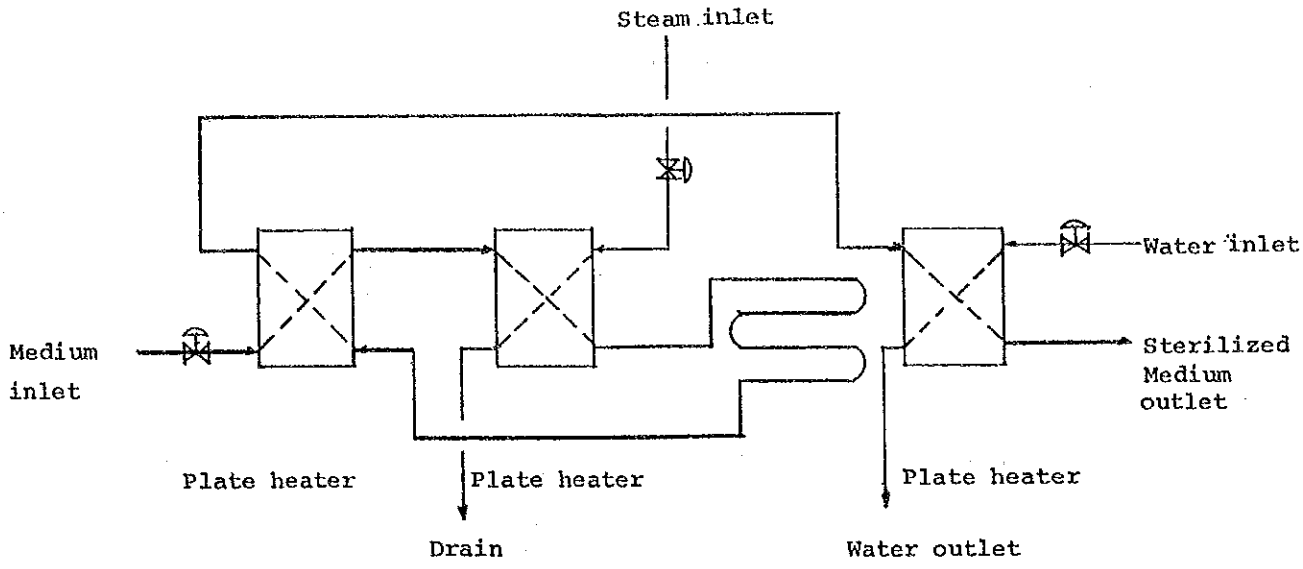
(15) Evaporator

(16) Crystallizer

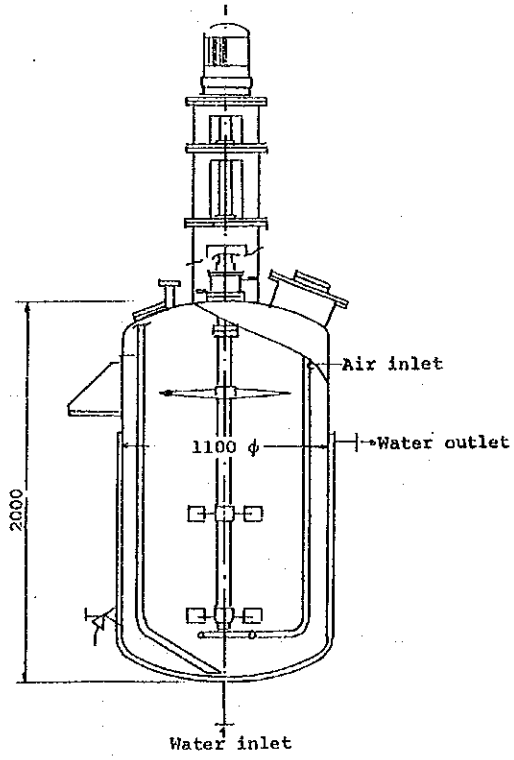
(17) Mother Liquor Tank

(18) Dryer Unit

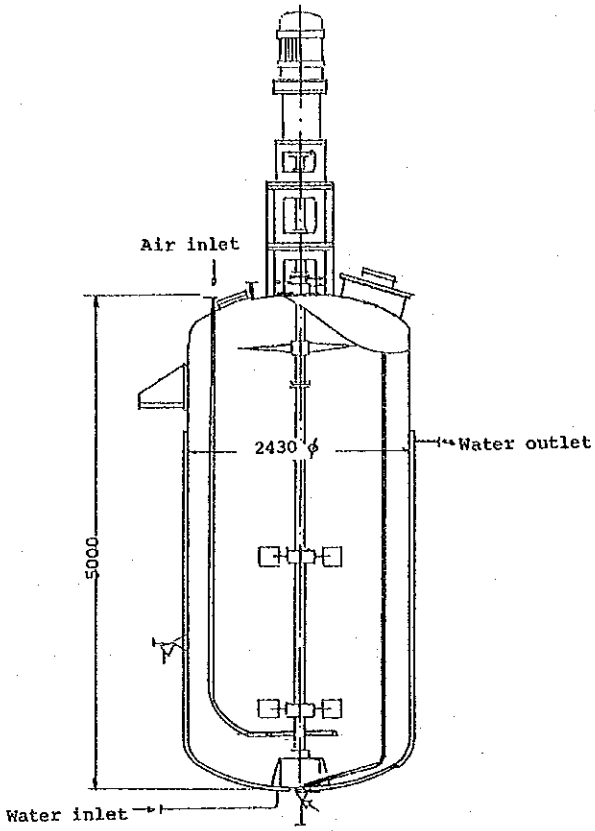
(19) Sifter



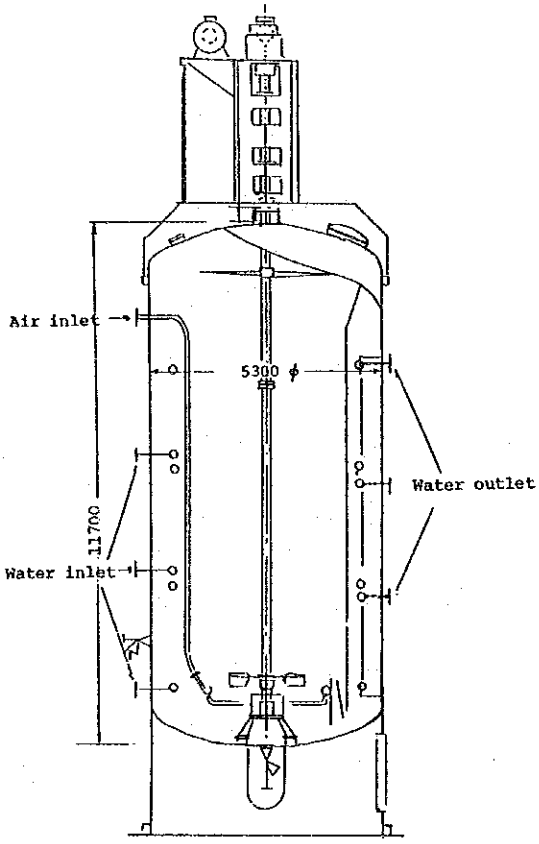
CONTINUOUS STERILIZER UNIT (EQUIP. NO. 109)



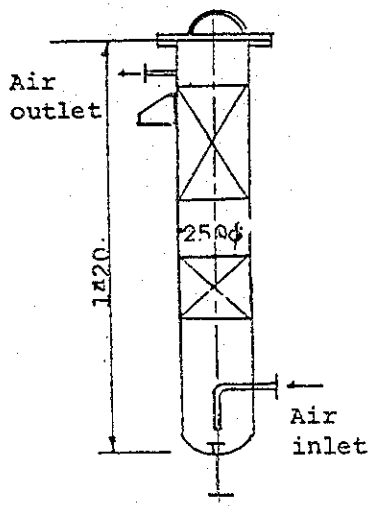
1ST SEED TANK (EQUIP. NO. 110)



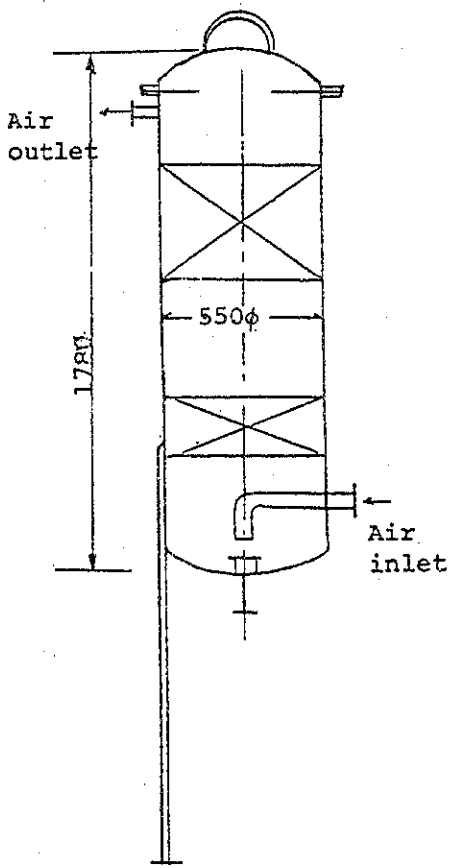
2ND SEED TANK (EQUIP. NO. 112)



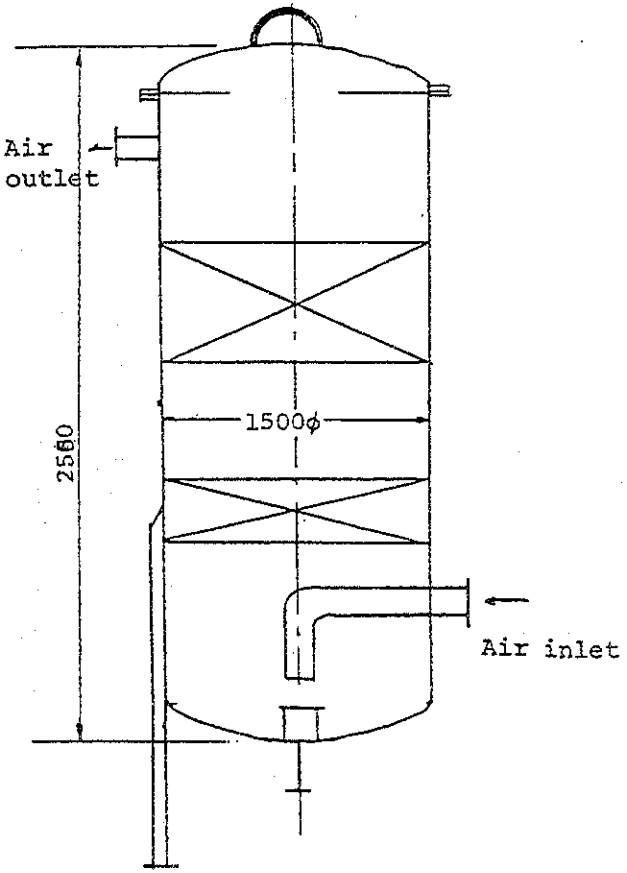
MAIN FERMENTER (EQUIP. NO. 114)



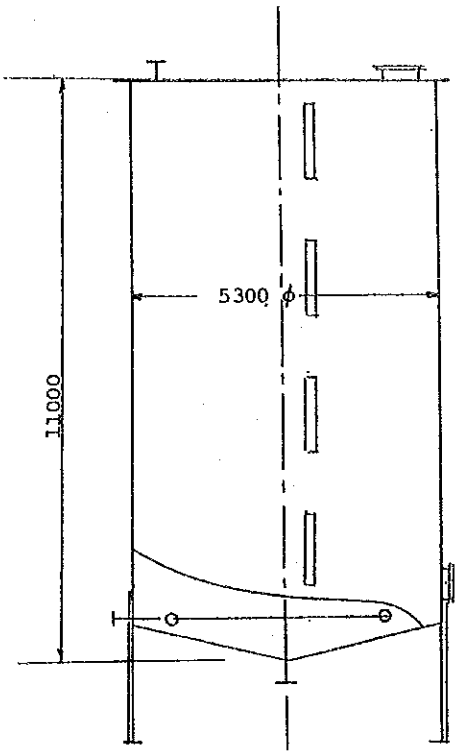
1ST AIR FILTER (EQUIP. NO. 111)



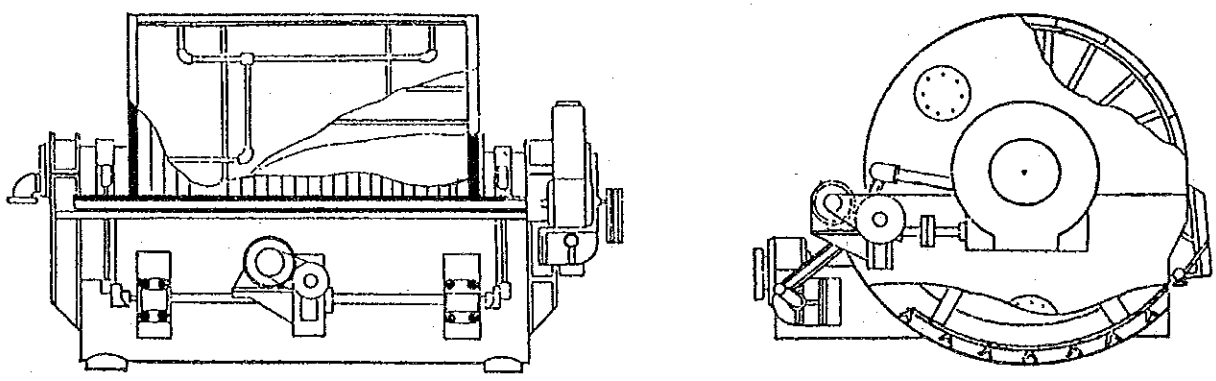
2ND AIR FILTER (EQUIP. NO. 113)



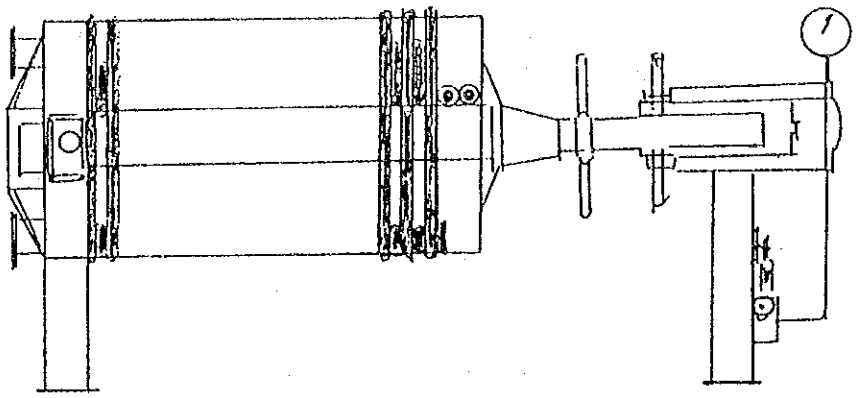
MAIN AIR FILTER (EQUIP. NO. 115)



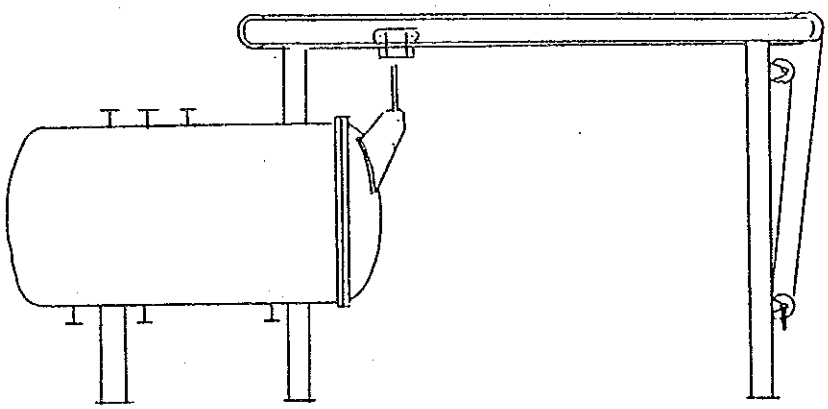
BROTH TANK (EQUIP. NO. 201)



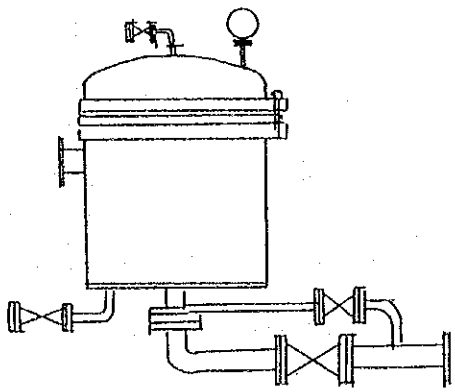
MYCELIUM FILTER (EQUIP. NO. 203)



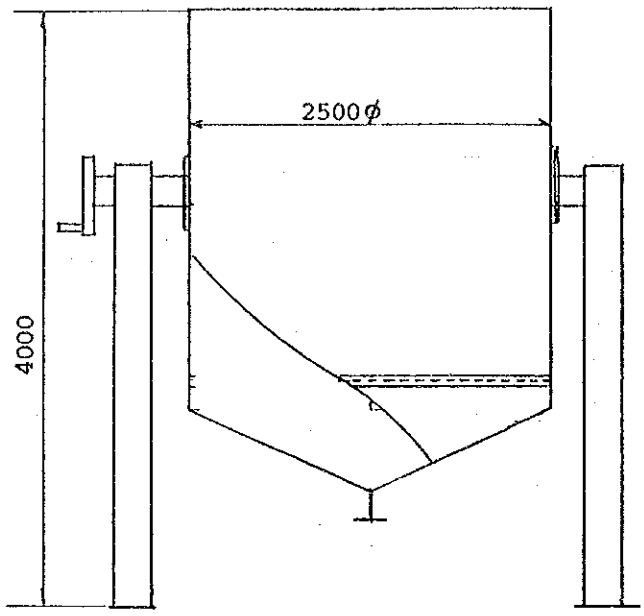
FILTER PRESS (EQUIP. NO. 208, 227)



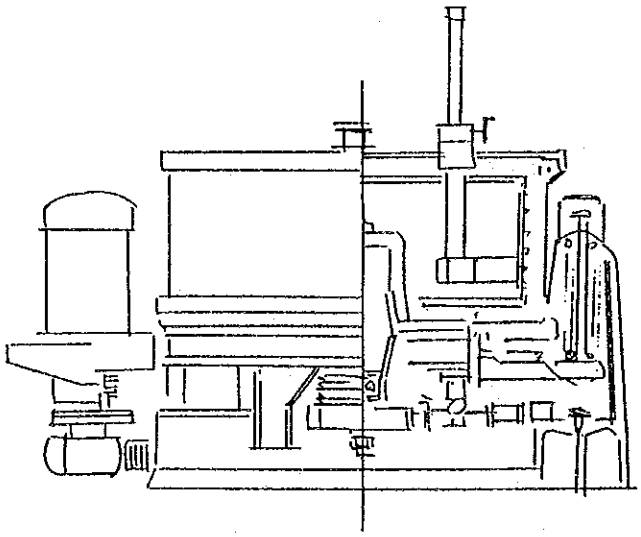
CARBON FILTER (EQUIP. NO. 244)



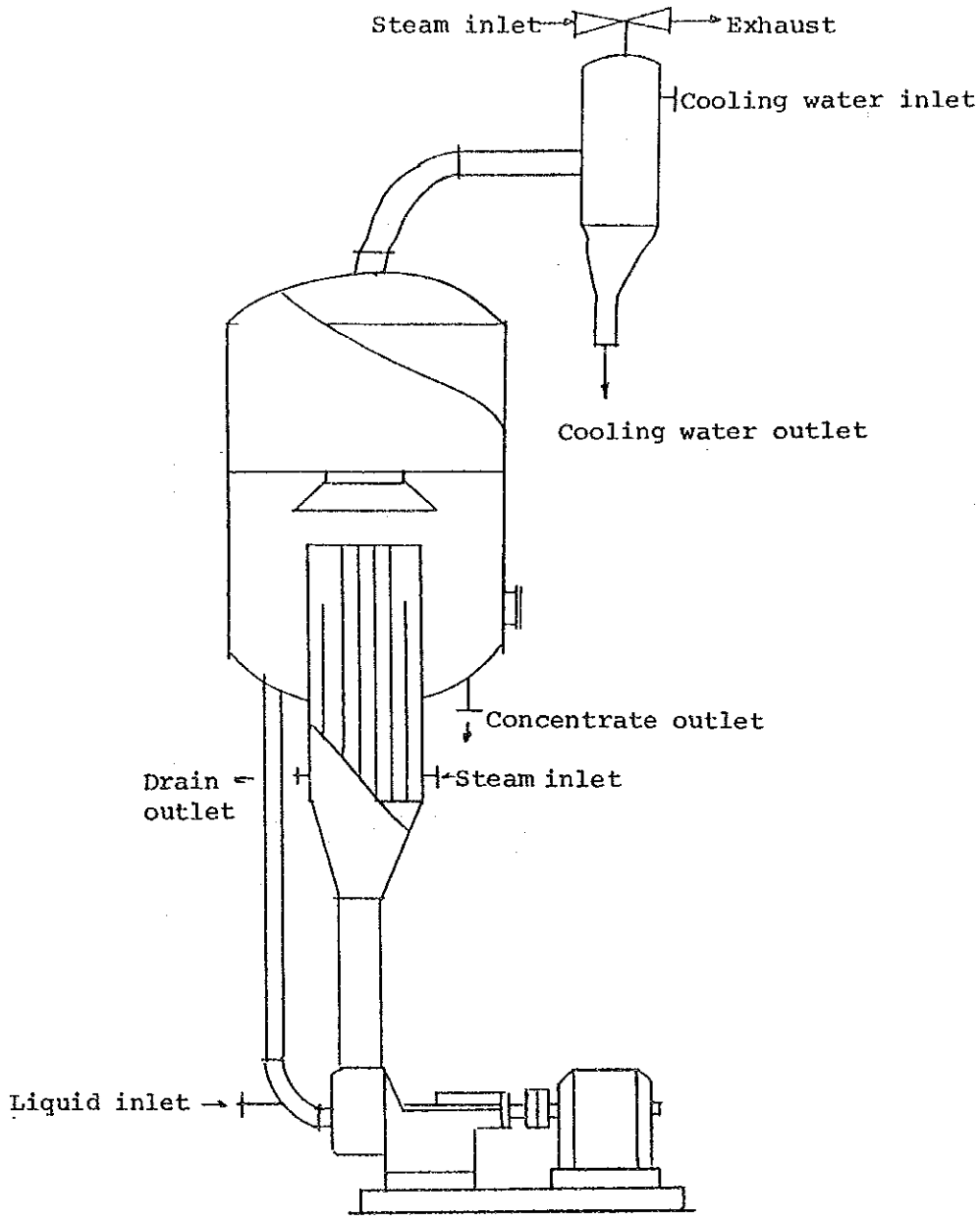
POLISH FILTER (EQUIP. NO. 247)



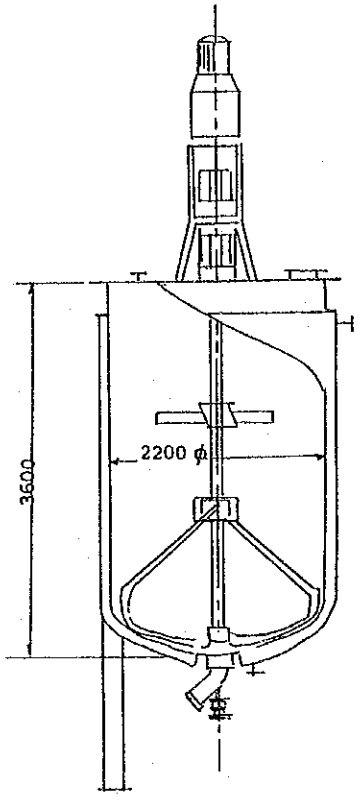
CALCIUM CITRATE FILTER (EQUIP. NO. 215)



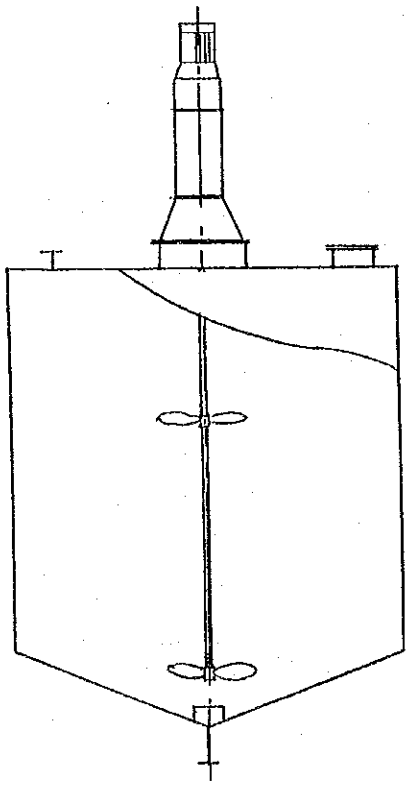
CENTRIFUGE (EQUIP. NOS. 222, 233, 239, 250)



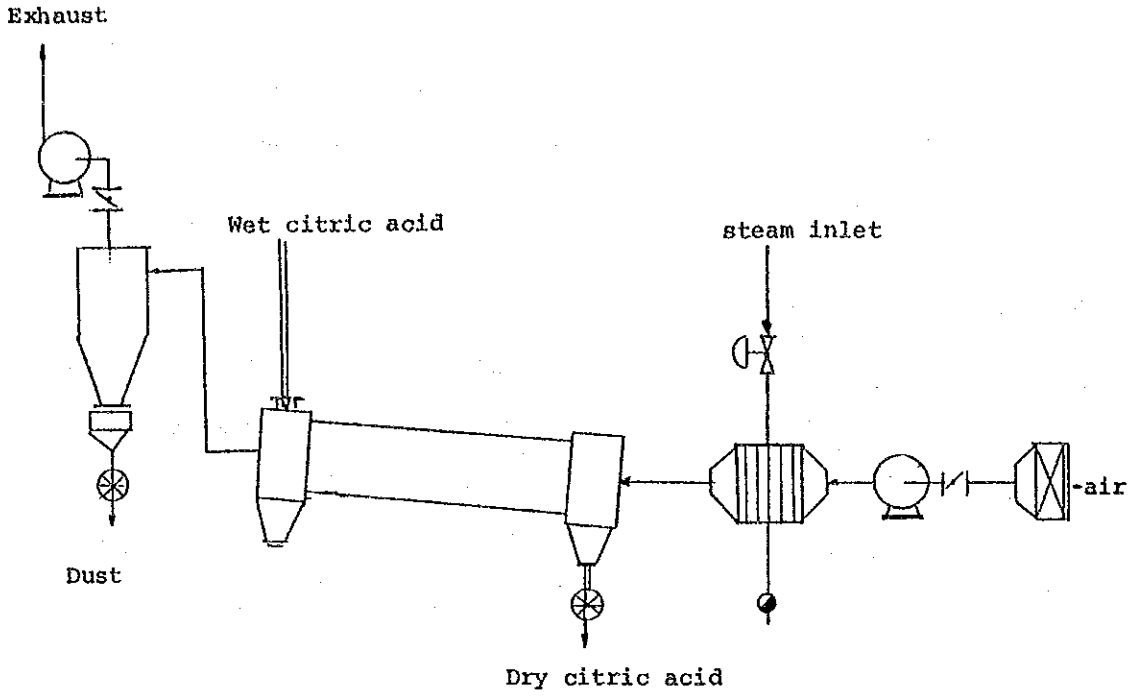
EVAPORATOR (EQUIP. NOS. 230, 236)



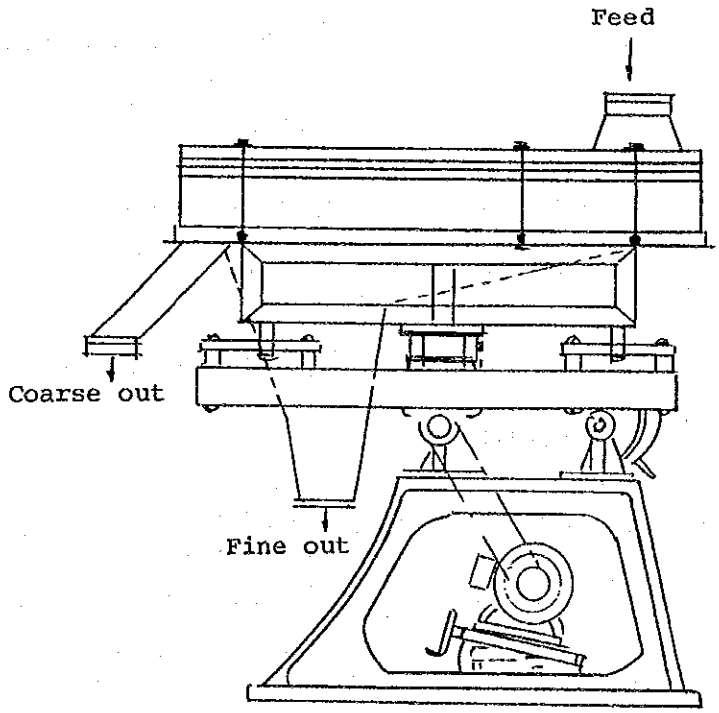
CRYSTALLIZER (EQUIP. NOS. 231, 237, 248)



MOTHER LIQUOR TANK (EQUIP. NOS. 234, 240, 251)



DRYER UNIT (EQUIP. NO. 254)



SIFTER (EQUIP. NO. 257)

12–6 Outline of Buildings and Structures

Buildings for the fermentation process and separation process are designed with a column span of 7 meters by 7 meters. Descriptions of the buildings and civil structures are shown below:

(1) Fermentation process and raw material warehouse

Structure	:	3 story reinforced concrete building	
Finish	:	– Walls	Brick
		– Floors	First floor is reinforced concrete.
			Second and third floors are checker plate.
		– Roof	Deck plate
Area	:	– Construction area	$21 \times 49 = 1{,}029\ m^2$
		– Floor area	$2{,}499\ m^2$

(2) Separation process and product warehouse

Structure	:	2 story reinforced concrete building	
Finish	:	– Walls	Brick
		– Floors	First floor is reinforced concrete.
			Second and third floors are checker plate.
		– Roof	Deck plate
Area	:	– Construction area	$21 \times 98 = 2{,}058\ m^2$
		– Floor area	$3{,}822\ m^2$

(3) Boiler house

Structure	:	Reinforced concrete	
Finish	:	– Walls	Brick
		– Floor	Reinforced concrete
		– Roof	Deck plate
Area	:	– Construction area	$15 \times 15 = 225\ m^2$

(4) Maintenance building

Structure	:	Single story reinforced concrete building	
Finish	:	– Walls	Brick
		– Floor	Reinforced concrete
		– Roof	Deck plate
Area	:	– Construction area	$15 \times 15 = 225$ m^2

(5) Office

Structure	:	Single story reinforced concrete building	
Finish	:	– Walls	Brick
		– Floor	Reinforced concrete covered with PVC tiles
		– Roof	Deck plate
		– Ceiling	Plywood
Area	:	– Construction area	$20 \times 15 = 300$ m^2

(6) Guard house

Structure	:	Single story reinforced concrete building	
Finish	:	– Walls	Brick
		– Floor	Reinforced concrete
		– Roof	Deck plate
Area	:	– Construction area	$4 \times 5 = 20$ m^2

(7) Pipe rack

Structure	:	Structural steel/concrete foundation
Rack height	:	5 meters
Rack width	:	1 meter
Total length	:	About 500 meters

(8) Road

Road width	:	8 meters
Finish	:	Gravel 150 mm thick, two layers and 30 mm of pre-mixture
Total length	:	About 1,200 meters

(9) Drainage

Structure	:	Concrete duct buried underground
Duct size	:	300 ~ 500 mm
Total length	:	About 1,200 meters

(10) Fence

Fence	:	Steel mesh
Fence height	:	2 meters
Total length	:	720 meters