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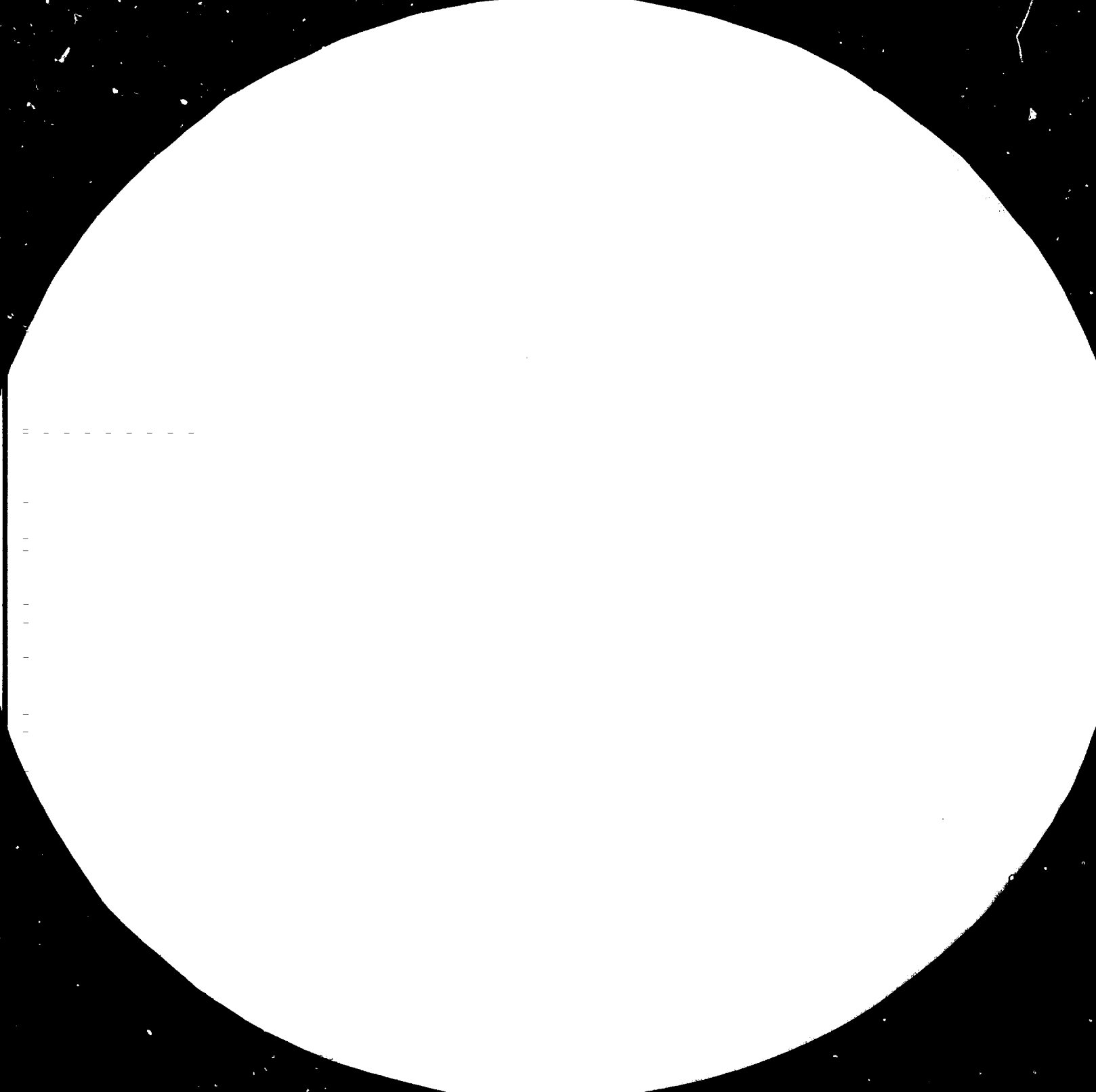
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DP/ID/SER.A/559  
9 January 1985  
ENGLISH

NATIONAL CANE SUGAR INDUSTRY RESEARCH CENTRE

DP/CPR/82/005

CHINA

14348

Technical report: Treatment of Effluent from an Alcohol Distillery and  
Utilization of Molasses\*

Prepared for the Government of China  
by the United Nations Industrial Development Organization  
acting as executing agency for the United Nations Development Programme

Based on the work of R. B. Brooks  
Expert in the Treatment of Effluent from an Alcohol Distillery  
and Utilization of Molasses

3126

United Nations Industrial Development Organization  
Vienna

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V.85-20359

TABLE OF CONTENTS

1.	INTRODUCTION	3
2.	STILLAGE DISPOSAL	5
	2.1 General	5
	2.2 Irrigation	8
	2.3 Biostil	10
	2.4 Disposal within the sugar factory	11
	2.5 Anaerobic Digestion	13
	2.6 Aerobic Treatment	17
	2.7 Incineration	18
	2.8 Growth of Protein	23
3.	FERMENTION OPTIONS	26
4.	IMMOBILIZATION	28
5.	CONCLUSIONS	31
6.	RECOMMENDATIONS	35

*Appendices.*

## 1. INTRODUCTION

This report describes the activities carried out by Dr. R.B. Brooks under the short term consultancy DP/CPR/82/005/11-02/31.7.C. This consultancy was to advise, lecture and present information on effluent treatment in alcohol distilleries and to discuss other related areas of technology where appropriate. The job description varied slightly during the lead up to the visit, both by UNIDO and the National Cane Sugar Industry Research Centre (NCIRC) but during the briefing session by Mr. Sissingh (UNDP) this was clarified in an explanation of the whole advisory programme between UNIDO and NCIRC. The task as described was to have a flexible approach to the lectures and seminars on the broad subject of distillery waste and fermentation, and to pass on as much written and reference material as possible. Two literature searches were carried out prior to arrival in China.

- . stillage analysis, treatment, usage and disposal;
- . immobilised enzymes and cells.

These literature reviews are included in Appendix 1 and 2. Copies of various other papers were handed over including material which has yet to be published as also were copies of the overhead projector slides, Appendix 3 (Figs. 1-62).

The programme as devised by NCIRC within the time constraint set by UNIDO ended on 27 October, Appendix 4 and included only one factory/distillery visit.

The NCIRC had been unable to find a visiting expert to give lectures on:

- . the Australian Cane Payment System
- . the Australian Sugar Industry Structure
- . other Asian Cane Payment Systems

The author volunteered to give this series of talks by extending the programme another eight days. This series of talks could be presented to UNIDO in a separate report. This amended programme, Appendix 5, allowed two more factory visits.

Apart from the main task of the expert, some observations were made about the sugar industry in China, factory operation, and the NCIRC. These have been included in Appendix 6.

## 2. STILLAGE DISPOSAL

There are a number of streams of waste water leaving a distillery particularly the fermenter and condenser cooling waters, the fermenter wash water, and the stillage. In China, effluent is only considered polluting i.e. it is only penalised financially, if the COD is greater than 100 mg/l - irrespective of the volume to be disposed of! Thus, with care, stillage is the only waste stream leaving a distillery which is considered polluting by the regulations of China. When a waste stream has a COD greater than 100 mg/l it is penalised financially on a daily basis only. The penalty is Y1000 per day for each day there is a discharge greater than 100 mg/l concentration with no regard for volume or total concentration. Of course, stillage is also considered polluting because it is dark brown whereas liquid condensate from the bottom of the rectifying column is often disposed of without penalty because it is like water in colour (but probably greater than 100 mg/l COD).

### 2.1 General

It is often said that there is no widespread agreement on the "best" disposal method for stillage. The reasons are obvious.

- . stillage characteristics vary widely depending on how and where it is produced.
- . the environmental considerations are different at different sites
- . the economics of disposal techniques vary widely.

It is unfortunate that most scientific papers neglect some of these factors in presenting case histories and solutions. Distillery still bottoms have many names, Fig. 1. Some of them give an indication of the country of origin or the type of feed material that may have been fermented. The molasses may have been pretreated, Fig 2, by acid addition (increasing stillage ash), sterilization (reducing by-product formation), stillage recycling (reducing total stillage volume but



marginally increasing ash). Nitrogen and phosphate nutrients may have been added to the molasses to improve fermentation (increasing stillage ash). The type of sugar cane and the way it is grown will affect the molasses analysis as will the degree of exhaustion of the molasses.

There are many different stillages, Fig 3, depending on the feed material to be fermented. Even if the crop is sugar cane, the actual distillery feed may be juice, syrup, molasses or pieces of cane. Even if the actual feed is cane molasses the product may be alcohol, citric acid, yeast, or some other product.

It can be clearly shown, Fig 4, that stillages are different and so solutions to disposal problems will be different. Stillage from a cane molasses distillery in Australia has six times the COD and 14 times the ash content of a cassava starch distillery in Brazil-yet these extra facts are often not reported. Molasses varies widely between beet and cane, cane molasses varies widely between countries and even within a country, and from year to year, Fig 6. Chinese cane molasses stillage seems to have a much higher nitrogen and potassium level than Brazil but a much lower lime and phosphate level, Fig. 7. This, as will be seen later can mean it will produce different results in irrigation, cattle feeding etc.

Cane sugar factories in different countries process different quality cane but also operate with different efficiencies, Fig.8,9, leaving the final molasses with different amounts of sugar, ash, non fermentables etc. This implies that factories within a country will have a variable analysis stillage to dispose of dependent on the geographical location of the factory in that country, the time of the year (cane maturity), the boiling system in the factory (which may change with mixed juice purity). This is particularly so in China where each factory usually ferments its own molasses within weeks of production compared to larger distilleries which collect molasses from various factories and may have 6-12 months storage capacity with the inherent smoothing out of differences.

A simple change to the distillery operation such as slopping back, Fig.10, can radically change the stillage analysis. It is also clear why distilleries operating with a cereal feed e.g. rice, sorghum, wheat or corn can recycle a large amount of stillage before the ash rises to the level at which it will begin to slow down fermentation, but cane molasses stillage can only be recycled a small amount or none at all. This can explain why some distilleries claim recycling of stillage is very useful in saving dilution water, reducing stillage volume and concentrating stillage without using evaporation, others claim they cannot recycle stillage as it slows down fermentation. As can be seen, Fig 10, a cane molasses of 12% ash needs a dilution of three times its volume of water to produce a substrate of the correct sugar concentration to ferment to alcohol. It will produce a stillage of four times the volume of molasses at 3% ash. However, with a cereal molasses distillery, the output of stillage can be reduced by a factor of 2.5 times using stillage recycle without unduly raising the ash in the fermenting medium.

If the stillage is recycled correctly, using a settling tank and flocculant, a precipitate can be induced, reducing the scaling in the distillery and reducing the ash in the fermentation medium, Fig. 11.

Stillage is no different to any other effluent, it must be disposed of by land, water or air. However the constituents in the stillage are changed, they must still eventually recycle back into the environment. The duty of an environmental engineer is to recycle the effluent in such a way that no permanent damage is done to any part of the ecosystem.

There are a very large number of treatment/disposal systems for stillage, Fig.13, most of which have some role to play in the correct situation. There is no uniquely best way, and often a combination of systems is better than just one method.

Stillage has two polluting factors, Fig. 14,

- . inorganic
- . organic

and nearly all of the treatment methods are concerned with reducing the organic material whereas many of the problems caused by disposing of stillage into the river or on land are because of the inorganic constituents. Remember, the stillage can have a salts concentration equal to sea water and this alone may cause pollution in a small fresh water river even if all of the organic material has been removed.

The organic matter in stillage can be analysed by the BOD<sub>5</sub>, COD, TOC, or PV, Fig. 15. The most appropriate for stillage analysis is the COD. The BOD<sub>5</sub> has a number of disadvantages,

- . it is difficult to reproduce accurately
- . it does not measure the total polluting load inflicted on the environment.
- . it only gives historic data as the analysis method takes five days (COD only 4 hrs).
- . incubation equipment is expensive.

## 2.2 Irrigation

Stillage contains valuable inorganic constituents, Fig. 16.

The proportion of N,P,K,Ca and Mg will vary depending on all the previously discussed factors. However, the "Manual of Cane Growing" N.J. King (1965) has the following quotation with regard to irrigation of stillage and molasses.

"It is unfortunate that no provision exists for the recovery of plant nutrients from the distillery residues which run to waste and are lost to the industry".

"74% of the potash entering the factory in the cane crop is eventually concentrated in the molasses. In addition, a quarter of the phosphoric acid in the cane is located in the molasses".

"Molasses is the most valuable byproduct from the farmers point of view".

He was a world recognised authority on cane agronomy and he considered that irrigation of molasses on to cane was good for it. Yet, it can be seen that molasses has a COD twelve times greater than stillage and an ash level four times greater, Fig. 15.

The best answer for stillage disposal would appear to be to irrigate it back on to the cane it originally came from. To do this, it can be seen, Fig. 17, that stillage in Australia may have to be irrigated at  $4.9 \text{ m}^3/\text{ha}/\text{yr}$ . There have been papers written on the problems caused by irrigation. However, a literature search, Fig. 17, shows that irrigation rates in these cases have been excessive. The very high irrigation rates used by Dubey in the U.K. were with a much lower ash level beet molasses stillage, again showing that a full understanding of each situation must precede using any successful disposal system.

Many countries use large central distilleries many hundreds of miles from cane growing areas. This may give economies of scale but removes the possibility of disposing of the stillage back on the cane fields. However, if it is concentrated to the same volume as the original molasses then it may be back loaded in the same tankers that carried the molasses. A small distillery, annexed to the sugar factory is in an ideal situation to recycle its stillage back to the cane fields. It is wise to irrigate fields after they have been ploughed out or immediately following ratoon harvesting.

The best way of classifying the hazardous nature of any irrigation water is shown, Fig. 19. Although the salinity hazard of stillage is high C3 or C4, the SAR is S1.

In 1981, John Warner was hosted by the Ministry of Light Industry to tour Guangdong Province and make recommendations for sugar cane agriculture. His major finding was that cane soils in China are deficient in potash. Stillage is very rich in potash.

### 2.3 Biostil

The Biostil process has caught the imagination of the technical people in China because it claims to use less equipment than a conventional system and it virtually eliminates stillage. This is not the best way of describing the Biostil process, particularly when discussing sugar cane molasses stillage, Fig. 20.

Instead of claiming virtual elimination of effluent it is more scientifically correct to say there is no elimination of inorganic material, no elimination of non fermentable organic material and perhaps a 10% reduction in BOD<sub>5</sub>. The stillage volume is lower because stillage is recycled, reducing the need for some dilution water. The concentration of all the components, both organic and inorganic, are much higher in the stillage from Biostil.

If a conventional system uses stillage recycle, particularly if it also uses the stillage from the rectifying column for molasses dilution, and an osmotolerant yeast is used as is used in the Biostil process, then stillage volume will also be reduced. Biostil will achieve a stillage with 30% dry solids while conventional will achieve perhaps 13-14% dry solids. If the disposal system is irrigation, anaerobic digestion, aerobic purification, pumping to river or sea, use as cattle feed or biomass production then there is no advantage in concentrating the stillage and thus no advantage in the Biostil process. In fact, there is a disadvantage as the energy demand for the conventional process is lower and equipment costs for the conventional process are lower than Biostil.

The process is shown, Fig. 21, with a few minor additions to the flowsheet normally published. Biostil will use a few percent less energy than conventional systems if the stillage is to be incinerated but equipment cost could be higher.

#### 2.4 Disposal within the Sugar Factory

Advantage can be taken of the situation where a distillery is located adjacent to the sugar factory, Fig. 22. The stillage can be poured on to the bagasse or coal going into the sugar mill boiler. This has been tried successfully on a trial basis in Australia, and in China where smaller non-suspension fired boilers are used it will be even more successful. With Chinese factories claiming very low final bagasse moistures, stillage which has been concentrated a little by recycling may be disposed of in this way. The point must be reiterated, that the solution to a stillage disposal problem may be to dispose of some one way and some another.

The stillage could be disposed of completely by spraying into a boiler chimney prior to the ash scrubbers in some countries that have high stack flue gas temperatures (500-600°C). China claim flue gas temperatures of only 150-200°C which would be too low. However, a method was discussed where they could use the flue gas to concentrate the stillage, using stillage to sluice ash from the boilers and scrubbing the flue gas at the same time.

Direct spraying of stillage into flue gas leaving the stack would increase the solids in the gas from 0.1 up to 2.0 grains per standard cubic foot which would not be tolerable under Australian air pollution regulations but may be quite acceptable in China.

Filter cake is usually used in China as a fish food in ponds adjacent to cane blocks. In Australia and many other countries it is a valuable cane soil additive. It can be seen, Fig. 23, that stillage and filter cake are similar in analysis and so stillage can be a good substitute for filter cake either in fish ponds or in the field.

There is potential for filter cake and stillage to be disposed of together and improve sugar production at the same time. We have found in Australia that a batch basket centrifuge of the type used for sugar can reduce the moisture content of filter cake from 74% down to 40%. This can give up to an extra 0.5% extra factory sugar production, Fig. 24, which would pay for the cost of the centrifuge. Stillage can then be added to the filter cake to increase its moisture level back to 74%. This combined mix can be belt conveyed, shovelled and transported by open truck without dripping stillage. These calculations and trials should be repeated by the Chinese to find out how their filter cake will centrifuge with their fugals. Again, it may be a partial solution which can be used with another method to dispose of all the stillage.

## 2.5 Anaerobic Digestion

There is a great deal of skill and experience of anaerobic digestors in China, particularly with regard to the domestic production of digester gas from animal/human waste. Biological purification of stillage whether aerobic, anaerobic or a combination of the two is only doing what would happen in nature but it is carried out more intensively and in such a way that any release to the outside environment is controlled so that nature is not unduly disturbed.

There are many ways of purifying stillage biologically, Fig. 25 and of course they all work technically. The task is to choose a method or methods which are economic in terms of resource use (man, money, materials). It is essential to realise that biological treatment only affects the organic material, the inorganic constituents in the stillage do not change and these are the one's that often cause most trouble. Not only that, but much of the soluble organic carbon in the stillage is converted to insoluble organic carbon (cell growth) which must still be separated and disposed of. This solid will amount to 10%-15% of the weight of the chemical oxygen demand removed. It must be disposed of on land.

Digester gas of 50% methane and 50% carbon dioxide has a high calorific value, about equal to ethanol and coal on a weight basis, Fig. 26. The yield of gas can be theoretically up to 830 litres/kg of starch or protein, Fig. 27. One cubic metre of biogas can run a 0.2 kW engine for 5.5 hrs.

However, a factory cannot be built solely dependent on biogas for power. It must have an alternative energy source because of the instability in the production of biogas and the need for a factory to keep operating. Thus, extra capital must always be spent on two alternative power sources, biogas and a conventional. This can often prove uneconomic.



Traditional digester systems operate on 30-40 day residence times. This would be uneconomic with the large volumes of stillage produced daily. If digestion is to be chosen for reduction of COD in the stillage then it is essential that a system is used which can fully complete the digestion in 1-2 days. There is no point experimenting with systems which are known to take 30-40 days. A small Chinese distillery producing 10 million litres per year of ethanol would require a traditional digester of 30,000 to 40,000m<sup>3</sup> capacity and would have to have the ability to be shut down for 240 days each year yet start up and run at the start of the next crushing season - a difficult task for a biological system. Only 1-2 day residence time systems are worth experimenting with because at least if the tests succeed technically the system stands some chance of succeeding economically.

The first of the modern systems which has been commercially operated at short residence time is the Upflow Anaerobic Sludge Blanket, Fig. 30. It can operate at loadings of 12 kg COD/m<sup>3</sup>/day reducing a 60,000 mg/l to 300-600 mg/l and produce 0.35m<sup>3</sup> methane per kg COD loaded. It is worth pointing out that this excellent achievement would under the present Chinese regulations still result in the distillery being fined the same penalty as if no COD reduction had taken place. A UASB digester flowsheet is shown, Fig. 31, together with a number of important references on results that have been achieved to date, Fig. 32.

A modern development which has proved of great interest to Chinese technical workers is the Tower digester (Upflow Flocculated Digester), Fig. 33. It is very similar to the UASB but it has never operated on full scale and never had a cane molasses stillage feed. Although it was called a tower digester, recent results have shown that on full scale, because it is scaled up on diameter not height, it will be a squat, fully mixed, tank Fig. 33. Its original design comes from the tower fermenter first made popular in the 1960's which has never created much demand. The main difference between the UASB and the UFFD,

Fig. 35, is that the UFFD uses flocculent to promote sludge blanket formation. Sulphide problems produced in pilot scale experiments were overcome by ferric chloride addition. Anaerobic digestors may be operated at 30-35°C (mesophilic) Fig. 37 or 50-55°C (thermophilic), Fig. 38. The results from a wide range of workers show that there is little difference in general performance between those two temperature regimes. All of the references given in Fig. 37 & 38 can be found in the very complete literature review given in Appendix 1. With stillage feed available at such a high temperature (100°C), and the high ambient temperatures of the tropics, there is the opportunity to operate at thermophilic temperatures. However, as the general evidence is that there is no real advantage, and certainly thermophilic operation is more temperamental, it would be advisable to concentrate on digestion in the mesophilic range.

The Chinese have been concentrating research on a two-stage process though no reasons were given. Perhaps a system of two mixed tanks in series would give better kinetics than one mixed tank, or they could optimise the hydrolytic step in the first tank and optimise the methanogens in the second step. However, once the pH in the first tank drops below 5.5 methanogen growth would stop totally and nothing would control the hydrolytic step so the pH would continue to drop until the hydrolytic bacteria were inhibited. A recycle would not improve matters as the methanogens in the second stage would not survive the low pH. It would be better to have a single mixed tank with a large recycle. One Chinese system had been set up using three tanks in series but loadings were low, removal rate very low and pH in the first tank very low. I suggested it would operate better if the three tanks were put in parallel and a recycle was used.

There are other high rate, intense systems such as anaerobic filters which are fully described in references given which could be researched.

Lagoon treatment of stillage is an attractive alternative in terms of capital and operating costs provided the land is available and inexpensive. However, land is not a cheap resource in China particularly near sugar/distillery factories.

Digestors cannot reduce the COD of stillage to below 100 mg/l so the financial penalty would still be exactly the same as if the stillage was discharged without treatment. The effluent would also still contain all the inorganics, so it would still have the same conductivity as seawater. A solid sludge, which would quickly start to smell, would be produced of about 5-10% of the volume of original stillage. This would have to be mixed into soil quickly before it encouraged fly breeding etc.

It is essential in China to have a second aerobic stage to purify the effluent from the anaerobic digestors to below 100 mg/l COD.

## 2.6 Aerobic Treatment

Aerobic biological treatment of waste is well documented. The higher intensity processes use more power and less area than the low rate processes, Fig. 38. Activated sludge systems Fig. 39, and trickling filters, Fig. 40 can both be used to produce effluents of COD less than 100 mg/l. It would be uneconomic to treat raw stillage because of the very large power demand but anaerobically digested stillage of 500-1000 mg/l COD could be purified to below 100 mg/l COD. It would still have a high colour, still have a high inorganic content and in China would also have a high ammonia (or nitrate/nitrite) content. Biological sludge production would be 0.3 - 0.6 kgs cells per kg COD removed and this dry weight of cells would be produced as a 2-4% dry weight gelatinous sludge. This sludge must be disposed of by digestion or by immediately mixing in with soil. Again, the point is made that organic carbonaceous material does not just disappear. It is often converted into another form which still has to be disposed of.

There is a process called the ANAMET process which has had one application on beet molasses stillage, Fig. 41. It is a combination anaerobic/aerobic system. It has an optional additional ammonia stripping system (Type 2) which helps the aerobic part of the process reduce the BOD<sub>5</sub> much lower than normally is the case.

## 2.7 Incineration

Stillage has been disposed of by incineration by a very few factories in the world, notable in Japan and Europe. The European experience was with sugar beet molasses stillage. Beet stillage has a very low melting point (approx. 650°C) and so furnace deashing is no problem as the ash runs out of the specially designed furnace bottom. This slagging (melting) characteristic caused a great deal of concern to sugar cane people when they considered disposing of concentrated sugar cane molasses stillage in boilers used for burning bagasse. However, we found from laboratory trials in 1974 that sugar cane derived ash had a much higher melting point (1300-1500°C) than sugar beet derived ash. Preliminary tests on burning the stillage in a suspension fire: bagasse boiler were satisfactory. It is known that the higher the silica ratio in the ash, the higher the melting point but in practice we did not find any effect of mixing bagasse with stillage before burning.

The possibility exists with incineration that the organic material in stillage will burn with sufficient heat release to provide enough energy to evaporate dilute stillage to a syrup concentrated enough to burn and have excess heat left over to help power the distillery. Again, only the organic pollutants are removed by incineration, the inorganics are still all present. There has been a good deal of talk about recovering crude potash, or just concentrated ash to use as a cheap fertiliser from the residual liquor after incineration. Of course, if the distillery is close to the cane fields then the fertiliser value can be obtained by irrigating the dilute stillage before incineration.

Autothermal conditions can exist with stillage having a total dry solids of as low as 35%. However, the flame temperature would be too low because of the water present, and unrecoverable latent heat would be lost from the stack that could be recovered by an extra stage in a multieffect evaporator, evaporating that 35% stillage to 60%.

Stillage varies widely in its elemental analysis, particularly with regard to the amount of K, Ca and Mg and these are the elements which affect the melting point of the ash the most. A Distillery adjacent to a sugar factory may choose to combine concentrated stillage with bagasse and burn the combination resulting in a different elemental mix. China, because of coal pricing policy and the need for wood fibre for paper pulp, burns little bagasse in sugar factory boilers but mainly coal. Again, this will result in a different ash and different melting characteristics. The advice given to China in this regard was to team up with the local University in their incineration studies. A local Professor of Physical Chemistry would be an invaluable team member in trials on melting characteristics of various inorganic components.

In January 1982, the first large scale modern sugar cane molasses stillage incinerator started up in Thailand. Engineers from the National Cane Sugar Research Institute had an extended visit of many months at that factory recently. I also visited the factory recently. Unfortunately, this factory does not analyse its molasses feed and its two major problems are scaling in evaporators and the high melting point of the stillage ash necessitating shut downs each shift to manually deash the furnaces. There are claims that this second problem will be overcome in any new design but it is unfortunate that lack of detailed data on the molasses being fermented makes it difficult to automatically transfer useful information to a different country, (different molasses = different stillage = different ash).

This distillery in Thailand is a large stand alone factory a long way from any sugar factory and the cane fields which sourced the cane/sugar/molasses. It is adjacent to a relatively small river which it could not discharge into without causing a major polluting nuisance. The situation is very different to any in China. Incineration was chosen as the only potential solution.

Burning the concentrated stillage raises 39 tph of steam of which 18 tph is needed for evaporating dilute stillage to produce the concentrate. The residual 21 tph is supplemented by a 19 tph fuel oil steam package boiler which together provides all the energy for the 180,000 litre/day distillery. This distillery actually ferments a mixture of molasses and sticky rice, again altering the stillage characteristics.

When burning the concentrated stillage as a supplementary fuel, the energy saving amounts to \$1.5-2 million each year but as the stillage disposal section cost \$25 million it certainly is not worthwhile economically. A second problem is that the condensate from the stillage evaporators has a pH of 3.3, a COD of 3,500 mg/l, and 84% of the total volume of the original stillage. Thus, although the claim is total removal of all polluting character from the stillage, the reality appears to be a reduction of 16% in volume, a removal of about 95% of the COD, a process which is not very economic, and still leaves all of the inorganic content to dispose of. However, for a large stand-alone distillery, it may be the only way. The Chinese engineers who spent a great deal of time at this incineration facility thought that all of the COD was getting in to the condensate because of entrainment of stillage in the water vapour. I believed some of it would have been because of volatile organic matter. This should be investigated further as under the Chinese legislation, a distillery discharging this condensate would be fined as much as if it was discharging untreated stillage.

A multinozzle burner, Fig. 43, is a good idea for burning waste such as concentrated stillage, as fuel oil, waste oil and other materials can be burned at the same time.

There are Chinese experiments going on at present on burning stillage in admixture with coal. This should work well as stillage ash would stay on the grate, fused with the coal and cause no fly ash problem. When burning a spray of stillage in a suspension fired bagasse boiler a large amount of fly ash is produced.

Under the incineration heading, the Zimpro (or Zimmerman) process should be discussed. This was a new idea to China. The Zimpro process is a wet air oxidation process which has been used for more than 45 years. It operates on the principal that aqueous suspensions or solutions of organic matter can react with the oxygen in air or pure oxygen at elevated temperature and pressure. This reaction is exothermic and because the "burning" takes place in the liquid phase, no latent heat is lost nor the heat of stack discharge gases. The system has been used particularly with sewage sludges and many difficult to oxidise organic molecules. Theoretically, once the waste has a COD greater than 1,050 mg/l, with normal heat losses, the reaction becomes autothermal. Certainly, with dilute stillage of COD greater than 20,000-30,000 mg/l the reaction will generate a great deal of extra energy which could be used to run a distillery. There is certainly more potential to raise energy by using the Zimpro process on dilute stillage at the 10-12% concentration it normally leaves the bottom of a conventional distillation tower than can be raised from incineration of a stillage concentrated to 60% solids. However, the Zimpro process is expensive in capital cost, with most of the equipment needing to be imported from overseas. The stainless steel reactor would need to be many cms. thick. The Zimpro section for a factory the size of the Thailand distillery (180,000 litres/day) would cost \$15-\$25 million. The Zimpro people have a great deal of laboratory experience on this type of stillage waste but no experience on full scale.

In a comparison of anaerobic digestion and incineration it is difficult to determine which process may be better, Fig. 44.

- neither process has an operating plant on full scale anywhere in the world which produces an effluent of less than 100 mg/l, but it is potentially possible with incineration.
- neither process treats the inorganic part of the stillage which can be a problem affecting discharge to small streams or on land, but incineration gives the possibility of further processing to concentrated crude potash fertiliser.



- e incineration will give twice the recoverable energy of anaerobic digestion by a more reliable method (biological processes are notoriously unstable)
- incineration will work at full efficiency immediately after start-up, whereas biological anaerobic digestion will take weeks and perhaps months to reach full efficiency. Chinese distilleries only operate for 6-8 months each year.
- digestion produces a secondary pollutant of 240-360 tpd of wet microbiological sludge which must be disposed of on land - it has good fertilising value but then so does the stillage before anaerobic digestion.

## 2.8 Growth of Protein

The Chinese scientists were very interested in the production of protein from stillage either by growing micro-organisms or by feeding animals.

### Animal feed

We have had experience feeding stillage to cattle as have a few other people in the world. The most important point to be made is that the amount that can be fed in an animal ration is dependent primarily on the potassium content of the stillage causing diahorrea and to some extent on the overall salts content. These two factors limit the amount of stillage which can be incorporated in an animal diet to below 5% with Australian stillage. The main 95% of the animal feed in a lot feeding situation must be made up of protein, fat, fibre, carbohydrate. There has been very limited reporting of "factors" in stillage which assist in an animals diet, probably dependent on whether the yeast is hydrolysed by leaving it in fermented molasses going to distillation, or whether it is removed for alternative disposal. Certainly in a lot feeding situation, feeding stillage at below 5% of the animals diet, the feed lot would generate infinitely more pollution than the untreated stillage.

### Biomass/Microbial Protein

It is important to realise that in growing protein, the stillage provides very little of the nitrogen, it only provides the carbon source. There are references to a number of workers that have grown, candida utilis, "yeasts", chlorella vulgaris, trichospora, "fungi" and "fodder yeasts" on stillage, Fig. 45. Generally, the stillage is first sterilised, then ammonium salts, urea, phosphates, yeast extract and other minor constituents are added. Molasses is also often added to improve cell growth sometimes by adding up to 50%, but this is not truly growing biomass on exhausted stillage.

It may not be possible to get the same results as other workers in this field. The biomass is growing on the organic constituents left in the stillage. The amount of sugars, fusel oils, organic acids and esters furfural, and glycerol etc. left in or produced by the original yeast fermentation will vary depending on the efficiency of fermentation, the degree of infection, the type of fermentation used, the staleness of sugar cane (polysaccharides).

The standard way of approaching this type of work is to try and grow all the "standard" type microbial growths on the waste - in this case, the stillage, and proceed with what seems to grow best. I advocated a different route with the Chinese technologists. The stillage should be analysed by HPLC, GLC and thin layer chromatography to find out what organic species are available as the major carbon sources. There will be carbon compounds which are non fermentable by the alcohol producing yeasts used in China, residual sugars which are not fermented during the standard fermentation and there will be all the byproducts of the EMP glycolytic pathway such as glycerol, fusel oils, acetaldehyde, acetic acid, lactic acid, butanediol etc.

Most yeasts can ferment glucose, galactose, maltose, melibiose, trehalose, but very few can ferment cellobiose. *Saccharomyces pombe* can use pentose (xylose, ribose) as can *Candida/Torula* under aerobic but not fermentative conditions. Some yeasts have high tolerance to salts, and the inorganic salts content will vary between countries. There may be some use in adding specific enzymes to breakdown the residual polysaccharides into fermentable sugars.

One major drawback is that all of the relatively easy to metabolise organic constituents will be used up, leaving difficult to biologically attack molecules, no nitrogen and no phosphate, Fig. 46. This partially biologically oxidised stillage will still contain a residual COD of 50% of the original.

There is little use of stillage as a raw material for biomass production and the reasons are clear. The economics for producing feed or food yeast are favourable in few places in the world. The stillage only provides carbon with a very small amount of nitrogen yet biomass is 46% protein, so the nitrogen to carbon ratio should be 15:100, i.e. some nitrogen source acceptable to the microbes should be added. Phosphate must also be added. The fermentation will have an optimum of 30-32°C and as fermentation is exothermic (3870 cal/gm yeast growth) the fermentation must be cooled. Cooling is no problem in Europe where surface waters are usually below 20°C but in the tropics it is a major problem. There is a large electricity demand as growing biomass is aerobic, using 400 volumes of air per volume of stillage. This air should be sterilised as should the stillage, to ensure only the chosen strain is the one growing. The centrifuged biomass will not be first quality as cane molasses stillage is full of ash and dark brown in colour, imparting high colour and flavour to the biomass.

The major problem is what to do with the waste from the biomass production system. It will still have 50% of the original organic strength, a higher salts content than originally and the "easiest" organics have been removed. If incineration is contemplated, only half the heat can be generated which means it is no longer thermally self sufficient, and only half the methane gas would be produced by anaerobic digestion.

3. FERMENTATION OPTIONS

The Chinese technologists were very interested in improving the fermentation section of the distillery, particularly to discuss ways of reducing the capital cost of the equipment by changing to other technology. This section discusses the value in investigating fermentation.

The total cost of equipment delivered to site of a normal cane molasses distillery is only 29% of the overall fully operational cost of the factory, Fig. 49. The fermentation section equipment would only be 25% of this i.e. 7% of the overall operational factory cost.

In a 10 year financial analysis of an ethanol distillery, the capital cost amounts to only 28% of the factory gate price of ethanol, Fig. 51. Thus, a 25% saving in the fermentation equipment cost will result in only a 1.5% saving in the overall distillery and only result in a 0.4% saving in the factory gate price of ethanol (say 0.2 cents in 50 cents). It is difficult to see how scientific papers can state that ethanol cost can be reduced by 25-50% by reducing the cost of "expensive" batch fermenters. It is worth noting, Fig. 52, that the same 0.4% saving in ethanol cost can be achieved with a 0.8% extra yield from the molasses say, producing 252 litres/tonne rather than 250 litres per tonne. While it is not easy to simply lift yield, it is certainly easy to lose yield

- by having a small amount of infection in a continuous fermentation which is perhaps not seen to be important enough to shut down the production line for
- by losing a small amount of yield by not fully completing the fermentation due to carry over in a stirred tank reactor.

The value in batch fermentation is that each yeast pitching can be fresh ensuring no loss of product through contamination and each fermentation can be left until fermentation really is complete.

4. IMMOBILISATION

There are two types of immobilisation, immobilised enzymes and immobilised cells (yeast or bacteria) - to produce ethanol.

Firstly, to consider the use of immobilised enzymes. Extracellular enzymes are generally stable while intracellular enzymes are unstable (or need closely defined conditions to operate well). The technique of immobilisation stabilises intracellular enzymes to allow them to be used outside of the cell. Intracellular enzymes are also expensive to manufacture because of their instability and the fact that cells must be ruptured to release them. Normally, enzymes are soluble and so are lost with the product. This is not acceptable with expensive enzymes. The concept of immobilising enzymes to hold them within a reactor is therefore a good one for unstable, expensive enzymes. The cost of such enzymes would be prohibitive for the production of such a cheap final product as ethanol, Fig. 53. There are twelve different enzymes which convert glucose to ethanol. Most of them have not been isolated on anything other than very small scale. They would all have different rates of reaction, different pH and other environmental optima.

Now let us consider immobilised yeast or bacterial cells for ethanol production. In a batch fermentation system, the cells are all held within the reactor (fermentor) so no immobilisation technique is required. The advantage of immobilised cells is in the operation of a continuous stirred tank reactor or a tubular plug flow reactor.

One benefit often quoted in the literature is that the cells are held by immobilisation thus removing the need for equipment to separate the new cell growth from the finished product i.e. no settling tank or centrifuge is required. This is not true as ethanol production is growth linked and an immobilised system would be operated in such a way that cells sloughed off the immobilised cell granules would just equal new cell growth.

These discarded cells would constitute most of the total cells produced during fermentation, and would still need to be separated. A second benefit often quoted in the literature is that the system can be operated at a high flow rate, one that would give wash out if the cells were freely suspended. However, at a high dilution rate, certainly productivity is higher but yield drops. As discussed earlier in the fermentation section, maximum yield is critical to the economics of ethanol production.

Certainly, one potential use of immobilised cells is in a tubular plug flow reactor system, Fig. 54. The fermentation tanks can be replaced by a simple tubular converter which will give very high conversion efficiency and by using a few columns in series (as with active carbon columns or ion exchange columns), the exhausted section can be replaced at regular intervals.

Most work in immobilised cells has been carried out with clean liquids, defined media, filtered solutions. Molasses is a very dirty syrup. Dilute molasses would soon foul up any packed column. Attempts to prefilter dilute molasses would result in having to discard fermentable sugar with the gums, waxes and other insoluble solids.

It has been said that fermenting more completely with such a system would have the dual benefit of producing more ethanol and at the same time reducing the COD of the effluent. Certainly, with pure glucose, an increase of substrate removal from 96% to 99% reduces the subsequent effluent by a factor of 4 i.e. from 4% down to 1%. However, with the large amount of non fermentable organic material in molasses, there is only a very small reduction of COD (less than 1,000 mg/l) for a similar improvement in fermentation efficiency.



There are many ways of immobilising cells using adsorption, entrapment and coupling systems, Fig. 55. Although there appears little possibility of practical application of such systems, it was of great interest to the Chinese technologists and so was discussed in detail, Fig. 57. A number of scientific papers of major importance in this area were handed over and a special literature search was completed, Appendix 2.

One area which is usually not discussed in scientific papers is the actual cost of producing the immobilised cells. We took one of the simpler gel immobilisation systems and designed a large scale production flow sheet, Fig. 59. A large number of assumptions have to be made as there is no published information on this topic. Assuming the gel beads have to be stored under sterile conditions in a moist condition with a limited life until immersed in a growth medium, we have estimated that the gel bead production unit of a 10 million litre per year distillery would have a capital cost of \$450,000 per year and running cost of \$55,000 per year, Fig. 60, and would still need the same yeast culture system. The fully erected batch fermentation system for such a distillery would be only \$1.8 million. Thus, any continuous system using immobilised gel beads would have to be less than \$1.3 million and in general, it is found that continuous systems are more expensive than batch systems.

The advantages and disadvantages of immobilisation are fully listed, Fig. 61. As economics finally dictate any decision, it would seem that immobilisation will not have any future in ethanol production.

5. CONCLUSIONS

The primary task of this project was not to reach conclusions but to pass on as many ideas as possible which have been established, tried or perhaps only talked about in other countries. The team at the NCIRC are all very dedicated applied researchers who although they have some pieces of very modern equipment are limited by budget restraints more so than they would be in an institute of comparable standing in many developed countries.

However, there were signs that some individual workers thought the answer to problems was simply to buy scientific equipment.

During discussions with the NCIRC it was apparent that their teams would be strengthened by using a more multi-disciplinary approach. Particularly, any work involving stillage should have agriculturalists involved in equal representation with technologists, as the ultimate site for disposal of either stillage, biologically purified stillage, inorganic salts in the stillage, or biological sludge grown during anaerobic or aerobic purification will be on land. Hopefully, this land will be used for growing cane, rice or some other important crop, and the valuable fertiliser constituents will not be lost.

The NCIRC should also combine with other learned Institutions such as the South China Institute of Technology. While resources of money, materials and men are in short supply in China there should be no duplication of work.

The question of why do research is often asked, and there are a number of equally valid answers, Fig. 62.

China particularly wants to improve productivity in sugar production with the long term goal of producing 10 million tonnes in 15 years time compared to 3.8 million tonnes now. Davy Agro analyses of factory data we have been given suggests that there are improvements to overall factory recovery that can be made using up-to-date Australian technology i.e. operating techniques (knowledge). This would certainly be economic but would only result in about 5% extra sugar recovery. While the percentage is small, a 5% improvement in just the larger factories is the equivalent of 2 extra sugar factories. This work should be left to others who already have the experience in this area.

The major advances in improved productivity will be in cane breeding, cane growing, cane harvesting and scheduling so that much larger crops of cane are grown per hectare, they are harvested at maturity and the better quality cane that is grown, is delivered to the factory in good condition. Paying for cane on quality rather than simply weight was the subject of a separate series of lectures and may be reported on at some future date.

Another research goal is to reduce costs of production. This can often best be done by actually working at the factory, rather than in the isolated atmosphere of a research institute. It was apparent that most of the NCIRC technologists had great appreciation of the practicalities of applied research as applied to sugar factories, but it would be worthwhile forming a review team including an economic manager which could assess the economic value in pursuing some research goals. What can be done and what should be done are not necessarily the same.

Often the only thought in research technologists heads is to educate themselves, they do the work because they would like to do it. This role can be taken by Universities but should not be the role of an industry dedicated Institute. At this stage in the development of the NCIRC, with allocation of resources spread thin, the luxury of research for its own sake should be left to peoples free time. Much of the work done at the Institute should be the investigation and application of other peoples findings. The Institute should see itself as a funnel to gather information from all over the world and to apply to the Chinese situation those developments which are most relevant. It should not blindly follow the conclusions of others. Conditions are different in different countries. General scientific reporting often excludes those things that are taken for granted i.e. an intimate knowledge of their own countries sugar industry, which of course is not known by other countries workers.

The Institute is in a better position than the individual sugar factory to sift the information from overseas but it is important always to acknowledge the work of others, to quote the original reference.

These thoughts influenced the lectures and answers to questions. The task was to try and pass on as much information and experience as possible in terms of photocopies of papers and references rather than give simple conclusions. In two weeks it is not possible to understand the Chinese situation.

Some things stand out however, with respect to stillage handling. The technologist will take the approach with stillage -

"How can we get more out of it?"

and will think of:

- energy (incineration, digestion).
- other by-products (animal feed, biomass).

A multidisciplinary team including agriculturalists would consider the wider question:

"Where is stillage of most use?"

and as:

- the major productivity increases are in the field,
- Chinese soils are deficient in potash,
- potash is a major component of stillage,
- the distilleries are all located in cane growing areas,
- there can be no cheaper disposal system than irrigating stillage on cane,
- even the water in the stillage is then of some value,
- no disposal system other than irrigation purifies the organic matter to below COD = 100 mg/l,
- no disposal system other than irrigation uses up the inorganic salts in the stillage.

the conclusion could be reached that disposal of stillage on to cane is the best solution to the problem. The major advances in productivity, to meet the target of 10 million tonnes sugar by 2000 will be in the field, including the installation of a payment for cane on quality system.

6. RECOMMENDATIONS

These suggested recommendations should not be read in isolation from the main report as is sometimes the situation with people wishing to obtain a quick review.

- 1) The pollution control regulations must be altered. At present, a fine is imposed on a daily basis for any volume of effluent which exceeds 100 mg/l COD. It is no simple matter to regulate on volume and concentration because for that to be done, these parameters must be regularly measured. Perhaps a system of penalties could be introduced based on the process and equipment installed to treat the waste, with occasional measurements made by one appointed officer from the Institute who's job is to travel around the industry taking snap samples.
- 2) A great deal more analyses of molasses and stillage must be made. There is little point in instituting a number of stillage disposal programmes before a few seasons of regular, careful analyses on both molasses and stillage. The range of individual inorganic and organic components should be known first to make comparisons with other workers overseas e.g. Acceptable irrigation rates will be directly proportional to total inorganic salts, incineration heat release will be proportional to COD yet problems with scaling of evaporators and deashing problems in the boiler will be dependent on the amount and type of inorganic salts etc.
- 3) Although a waste of a valuable resource, stillage can be acceptably disposed of in a river if the river has a sufficient flowrate, aeration rate, and width to accept it. If there is a situation where a factory is discharging to a river and believes it is doing no environmental damage because the river can adequately purify the load, then it is suggested that the

factory fund an independent postgraduate study of the problem - considerably cheaper than the pollution fine and a more constructive use of the money. It would make an excellent MSc thesis, provide money to the University, and provide extra funds to employ the postgraduate. It would be totally independent.

- 4) During discussions on the measurement of pollution using BOD, COD and TOC, it was apparent that the BOD<sub>5</sub> method being used relied on chance contamination to seed the dilute stillage with bacteria. This is normally not acceptable with any effluent and is particularly unsound practice with an effluent which has effectively been sterilised during production. As there is no way that the Institute can obtain a fresh daily sample of seed without running their own laboratory activated sludge effluent treatment pilot plant it is suggested they discontinue measuring BOD<sub>5</sub>. There is no point in measuring BOD<sub>5</sub> knowing it is the wrong method because it will still be reported and will then be accepted as correct. The COD is a more meaningful measurement of stillage polluting value even if the BOD<sub>5</sub> method is done in the correct manner.
- 5) In discussions with microbiologists, there was a great desire to study thermotolerant yeasts as "the fermentation temperature often rose above 35-40°C". At the three distilleries visited, they had no problem keeping the temperature below 35°C. It would be worth considering studying osmotolerant yeasts rather than thermotolerant ones. There is some evidence, that osmotolerant yeasts produce a smaller amount of glycerol i.e. a higher yield of alcohol, molasses fermentation speed is affected by the ash content of the molasses and this limits the degree to which stillage can be recycled. Recycling of stillage can help in many ways,
  - reduce distillery water use
  - improve exhaustion of sugars in stillage
  - reduce the volume of stillage to be disposed of.

- 6) It is worth trying to use stillage instead of water to scrub flue gas and as a long term project, to use the hot flue gas to evaporate stillage to a concentrate by direct contact. This was discussed in detail, but it is not done on full scale.
  
- 7) One of the most useful ways of disposing of stillage is by fugalling filter mud and then re-adding stillage to produce a cake of similar wettness to that prior to fugalling. This was suggested to the NCIRC, discussed in detail with them and received enthusiastic response. The author tried this method in 1974 on pilot scale with some success. The Chinese have promised to try it. The liquid centrifuged off contains valuable sugar. Its recovery can pay for the entire process and the stillage gets "free" transport to the cane fields - or perhaps in China to the fish ponds.



**Davy Agro**

**APPENDIX 1**

**STILLAGE LITERATURE REVIEW**

## STILLAGE LITERATURE REVIEW

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**Davy Agro**

**APPENDIX 2**

**IMMOBILIZED CELLS AND ENZYMES LITERATURE REVIEW**

## Davy Agro

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**Davy Agro**

**APPENDIX 3**

**OVERHEAD PROJECTOR SLIDES**

# DISTILLERY STILL BOTTOMS



1. STILLAGE
2. DUNDER
3. DUNDAR
4. VINASSE
5. SLOP
6. VINHOTO
7. C.M.S. - CONDENSED MOLASSES SOLUBLE
8. F.E.L.
9. C.P.B.
10. BORLANDA CONCENTRADA
11. DICKSCHLEMPPE
12. DICKLAUGE
13. D.M.S. - DRIED MOLASSES SOLUBLE



Day

# SUGAR CANE - MOLASSES - ETHANOL

1. WAS MOLASSES PRETREATED? e.g. STILLAGE RECYCLE  
H<sub>2</sub>SO<sub>4</sub> ADDITION  
STERILIZATION

2. WERE NUTRIENTS ADDED? e.g. NITROGEN  
PHOSPHATE

3. COUNTRY OF ORIGIN? e.g. IS CANE FERTILISED  
IS MOLASSES EXHAUSTED.

# MANY DIFFERENT TYPES OF STILLAGE

SUGAR BEET	JUICE	ENZYMES
GRAIN	SYRUP	AMINO ACIDS
RICE	MOLASSES	CITRIC ACID
SUGAR CANE	SOLID PHASE	BAKERS YEAST
		INDUSTRIAL ETHANOL
		DRINKING ETHANOL





# STILLAGE ANALYSIS

	SUGAR BEET	AUSTRALIA		BRAZIL		CHINA MOLASSES
		MOLASSES	CANE JUICE	MOLASSES	CASSAVA	
DRY MATTER %	5.5	11.9	2.2	10	2.5	8.3
COD (mg/l)	48,000	110,000	27,000		18,000	68,000
BOD <sub>5</sub> (mg/l)		65,000	18,000		12,000	36,400
NITROGEN %	0.36	0.23	0.06	0.75	0.25	0.14
TOTAL SUGARS %		1.33		1.3	0.68	1.5
GUMS %		3.33		1.0	0.6	
TOTAL ASH %		3.2	0.6	2.3	0.23	2.3
Na %		0.03	0.01			
K %		1.2	0.15			0.4
Ca %		0.28	0.05	0.65	0.009	
Mg %		0.17		0.32	0.008	
PTG %		0.04				
P %	0.01	0.007	0.008	0.02	0.02	0.04
Cl %		1.1		0.15	0.01	
SO <sub>4</sub> %	0.03	0.3				

\* Reducing Sugar by Fehling's method - furfural etc and other non-sugar organics



Day

CAUTION 小 心

YOUR STILLAGE MAY BE DIFFERENT

YOU MAY GET DIFFERENT RESULTS.<sup>5</sup>

# MOLASSES ANALYSIS.



Davy

	AUSTRALIA CANE	WORLD CANE	WORLD BEET	CUBA CANE	INDIA CANE	CHINA CANE
DRY MATTER	72-80	75	76	76	73	76
SUCROSE	30-39	30-40	48-52	33.9	41.8	34
REDUCING SUGARS	8.7-19.9	15-20	2-12	16.6		20
NON FERMENTABLE		2-4	.5-2			
GUMS	11	4	6-9			
ORGANIC ACIDS		3	6-8			
PROTEIN (N x 6.25)	3-6	2-3	6-8	3.6	7.5	
TOTAL ASH	7.5-12.5	8-18	7-14	5.4	11	12-15
Na	.02-.17	.01-.7	.3-1.3			
K	2.6-5.1	1.2-6.4	2.3-4.4			
Ca	.7-1.2	.03-.95	.08-1			
Mg	.16-.74					
P	.3-1.1					
S	.04-.08	.4-2	.02-.07		.25	
Cl	1.3-3.9	.7-3	.3-1.5			
PH	5.5-6.5	5.1-6.0	6.5-9			

D

## STILLAGE FROM VARIOUS FEEDS.

D  
a  
v  
y

BRAZIL	CANE MOLASSES	CANE JUICE	CASSAVA STARCH	CHINA
ORGANIC MATTER	63.4	19.5	21.8	
TOTAL NITROGEN	1.2	0.3	0.4	3.4
SO <sub>4</sub> g/litre	84	0.6	0.1	
CaO g/litre	3.6	0.7	0.1	2.2
P <sub>2</sub> O <sub>5</sub> g/litre	0.2	0.2	0.2	0.05
MgO g/litre	1.0	0.2	0.1	
K <sub>2</sub> O g/litre	7.8	1.2	1.1	11.1



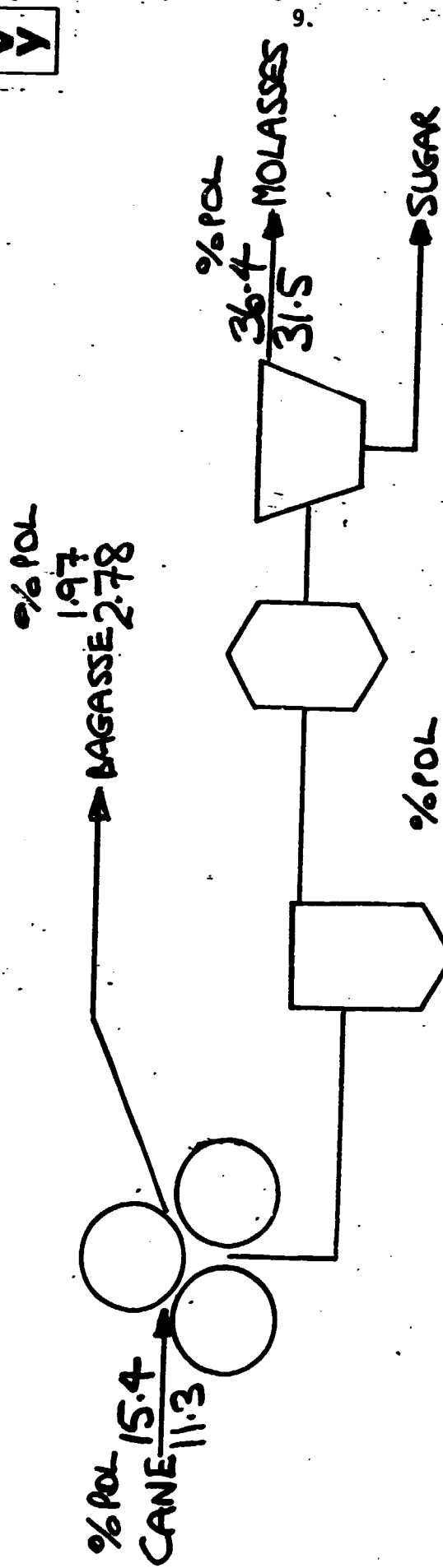
# SUGAR FACTORY DATA



Davies

	CHINA	AUSTRALIA	INDONESIA
MIXED JUICE BRIX	15.5	17.0	12.6
POL	12.6	15.0	9.48
PURITY	81	88	75
POL % CANE	12.5	15.4	11.3
FIBRE % CANE	11.0	14.2	15.5
SUGAR ON CANE	11.0	14.38	9.12
MOLASSES ON CANE	3.2	2.9	3.5
MOLASSES BRIX	92	87.3	92.4
POL	27	31.9	32.1
PURITY (APP)	29	36.5	34.7
PURITY (TRUS)	37	46.6	
SUCROSE	34	36.3	36.4
DRY SUBSTANCE	-	78	79.5
REDUCING SUGARS	20	11.9	17.0
ASH	-	14.18	16.0
R.S./ASH RATIO	-	0.84	1.06
FILTER CAKE ON CANE	3.0	5.55	4.04
POL	3.0	1.59	3.95
WATER	74	74.2	71.7
ASH	-	8.6	

# SUGAR FACTORY



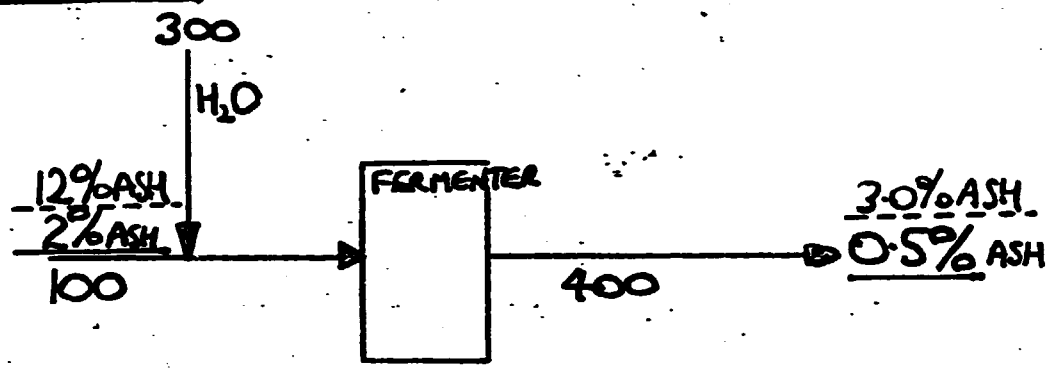
	AUSTRALIA	CHINA?	INDONESIA
<u>POL BALANCE</u>			
SUGAR	89.6	84.0	80.0
BAGASSE	3.7	7.0	8.1
MOLASSES	6.3	7.0	9.5
FILTER CAKE	0.6	0.6	1.0
UNDETERMINED	- 0.2	1.4	1.4



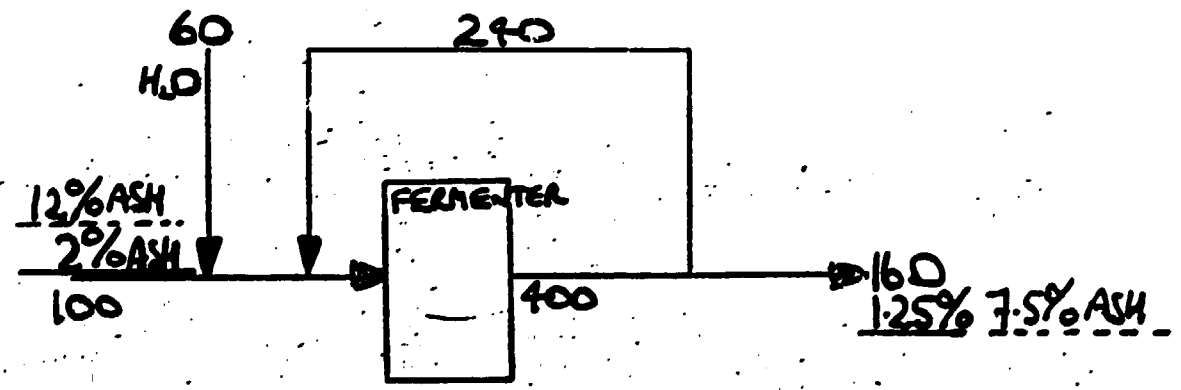
# SLOPPING BACK.

PER 100 OF MOLASSES: CEREAL MOLASSES CANE MOLASSES

## NIL RECYCLE



## 60% RECYCLE



CEREAL WITH RECYCLE OF 60% LESS THAN MOLASSES WITH NO RECYCLE.

# DISTILLERY MOLASSES MUD



WATER %	60
ASH %	26*
ORGANICS %	14
K %	4.3
POL %	1.3
BOD <sub>5</sub> mg/l	100,000
TONNES/DAY	27.4

\* MAINLY  $\text{CaSO}_4 \cdot \text{K}_2\text{SO}_4 \cdot \text{H}_2\text{O}$

DOUBLE SALT

# STILLAGE DISPOSAL



1. LAND

IRRIGATION ON SOIL

EVAPORATION

2. WATER

RIVER

OCEAN

UNDERGROUND

3. AIR

AS  $\text{CH}_4$  OR  $\text{CO}_2$

N AS  $\text{NH}_3$

# TREATMENT METHODS



1. IRRIGATION
2. MARINE DISPOSAL
3. MIX WITH FILTER CAKE
4. INCINERATION
  - 4.1. WITH BAGASSE
  - 4.2. SEPARATE FURNACE
  - 4.3. ZIMPRO
5. ANAEROBIC DIGESTION
  - 5.1. THERMOPHILIC
  - 5.2. MESOPHILIC
  - 5.3. UASB
  - 5.4. TOWER
  - 5.5. FLUIDISED BED
  - 5.6. FILTER
6. ANAEROBIC/AEROBIC
  - 6.1. ANAMET
  - 6.2. LAGOONS
7. AEROBIC
  - 7.1. BIOMASS
  - 7.2. ACTIVATED SLUDGE
8. ANIMAL FEED
9. PHYSICAL-CHEMICAL
  - 9.1. REVERSE OSMOSIS
  - 9.2. ELECTRODIALYSIS
  - 9.3. SEDIMENTATION.



STILLAGE HAS TWO POLLUTING FACTORS

1. ASH (INORGANICS, SALTS)
2. ORGANIC MATTER (BODs, COD)

ALL TREATMENT METHODS REMOVE ORGANIC MATTER

BUT DISPOSAL IN RIVER OR SOIL IS MAINLY A PROBLEM OF ASH

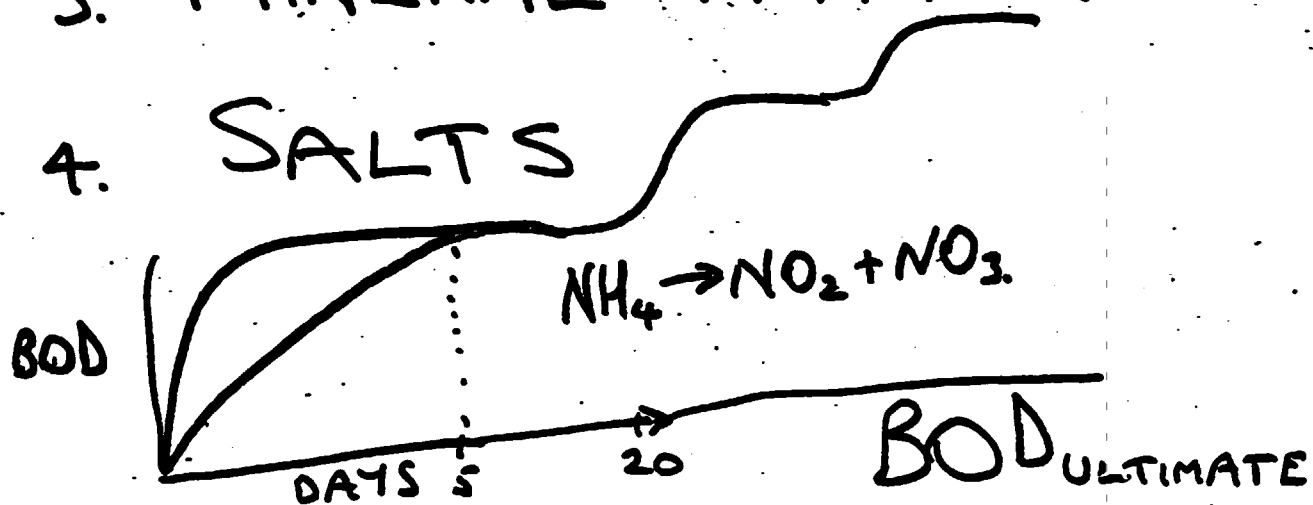


1. INORGANICS

2. ASH

3. MINERAL MATTER

4. SALTS



1. ORGANICS

2.  $\text{BOD}_5$  = BIOLOGICAL OXYGEN DEMAND

3.  $\text{COD}$  = CHEMICAL OXYGEN DEMAND

4.  $\text{TOC}$  = Total Organic Carbon  
not  $\text{CO}_3$ ,  $\text{CO}_4$

5. P.V = Permanganate Value.  
3min and 4hr.





# VALUE OF DUNDR

10 MILLION LITRES  $C_2H_5OH$  PER YEAR

\$

N	Aqua $NH_3$	80,000
P	Superphosphate	14,000
K	Potassium Chloride	400,000
Ca	Line	24,000
Mg	Magnesium Sulphate	500,000
		<u>\$1,018,000</u>



---

STILLAGE

<sup>COD</sup>  
50,000 mg/l

MOLASSES

600,000 mg/l



# IRRIGATION

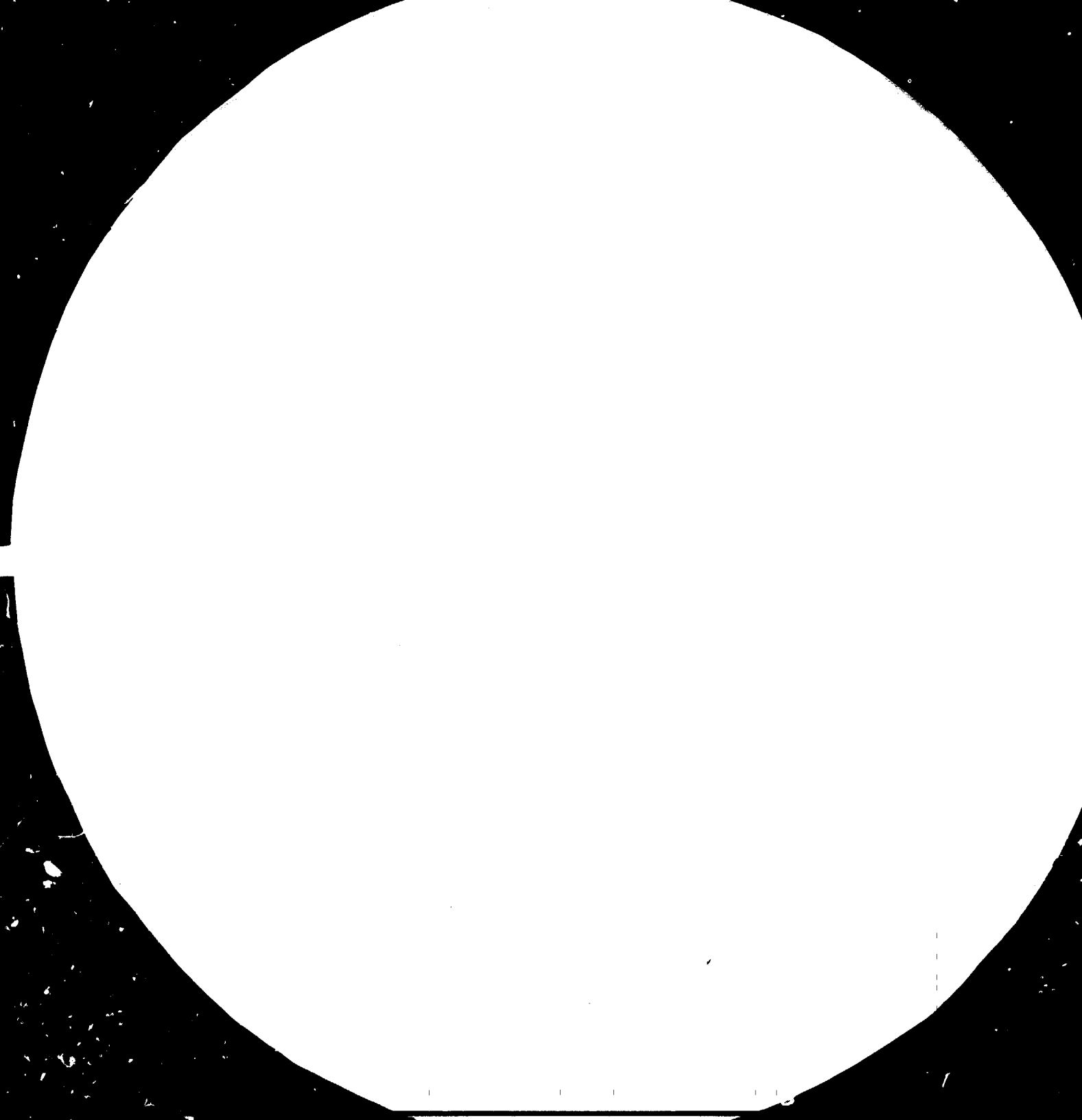
REF.	APPLICATION m <sup>3</sup> /ha	COUNTRY	
ALMEIDA	650-1000	BRAZIL	PROBLEMS
ROSS	93 /DAY		OK
DUBEY	1280 /YR	UK	OK
GUMARAES	382-419	BRAZIL	OK
COOPER	185	W. INDIES	OK
BEISKE	6.4-12.9	AUSTRALIA	OK
MONTEIRO	650-1000	BRAZIL	PROBLEMS
GLORIA	35-50	BRAZIL	OK
USHER	400	AUSTRALIA	OK

At 70 TC/ha

$$328 \text{ TE/hr} = 46 \text{ T C}_2\text{H}_5\text{OH/day} = 552 \text{ T STILLAGE/day}$$

$$\text{So, } 70 \text{ TC} = 4.9 \text{ T STILLAGE} = 4.9 \text{ m}^3 \text{ STILLAGE}$$

MUST IRRIGATE  $4.9 \text{ m}^3/\text{ha/yr}$  TO RETURN ALL  
DUNDR TO ALL CANE IT CAME FROM.





145

150

155

160

165

170

175

180

185

190

195

200

205

210

215

220

225

230

240

250

255

260

265

270

275

28

32

36

40

2.5

2.2

2.0

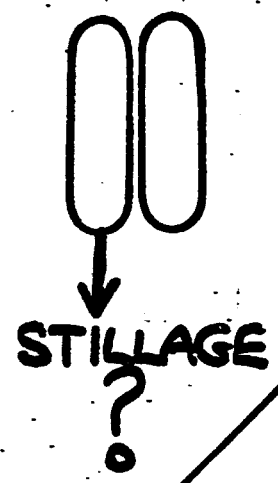
1.8

1.6

MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS  
STANDARD REFERENCE MATERIAL 1010a  
(ANSI and ISO TEST CHART No. 2)

18. CENTRAL DISTILLERY

Davy

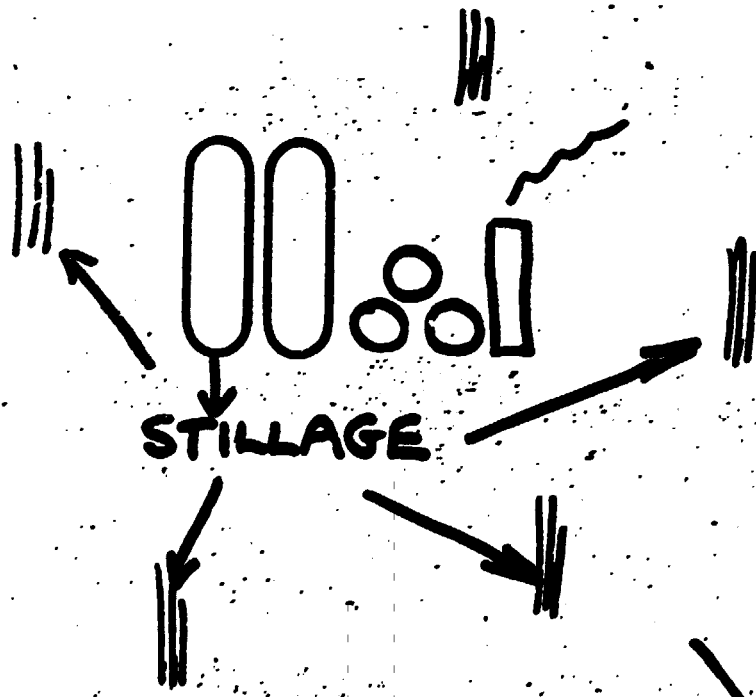


← 20 - 200 MILES →



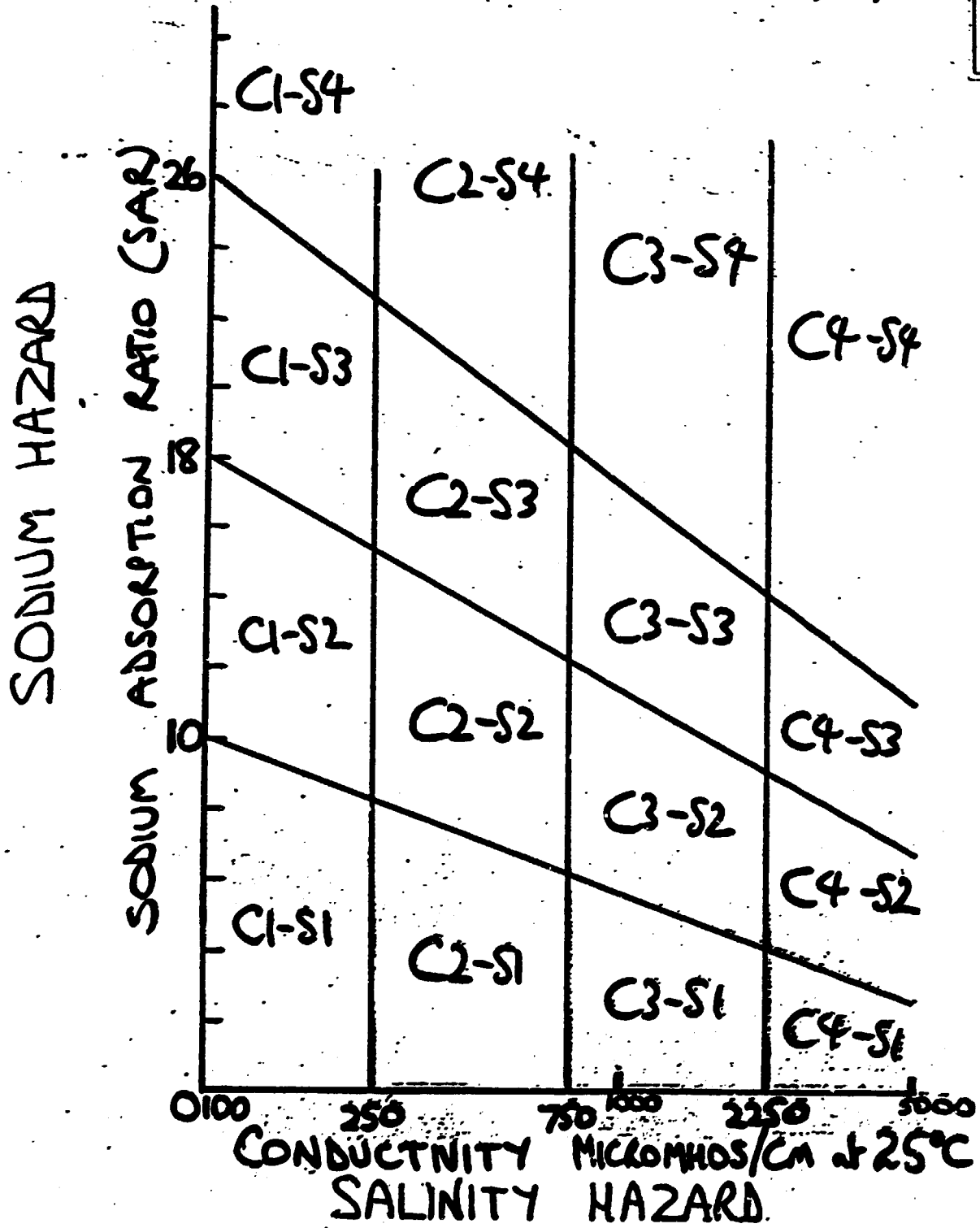
ANNEXED DISTILLERY

SOIL TYPE?  
RAINFALL?  
CLIMATE?



$$SAR \text{ is } \frac{Na^+}{\sqrt{(Ca^{++} + Mg^{++})/2}}$$

millequivalents/litre



CLASSIFICATION OF IRRIGATION WATER

BIOSTIL - FOR CANE MOLASSES STILLAGE

1. ELIMINATION OF EFFLUENT IS CLAIMED.

BUT NO ASH REMOVAL AND ONLY 10% BOD<sub>5</sub> LESS  
THE STILLAGE VOLUME IS REDUCED TO 3-5 LITRES/LITRE ETHANOL

2. LESS HEAT REQUIRED BY BIOSTIL IS CLAIMED.

I CALCULATE 48% LESS HEAT IF 60% SOLIDS IS REQUIRED  
AND MORE HEAT IF STILLAGE CONCENTRATION  
NOT NECESSARY.

3. IF 9%  $C_2H_5OH$  ← LIKE IN CHINA  
(RATHER THAN 7%) THEN CONVENTIONAL IS

19% LOWER HEAT THAN BIOSTIL FOR CANE MOLASSE

4. THEY CLAIM LOWER EQUIPMENT COST FOR BIOSTIL  
HOWEVER CONVENTIONAL IS NON PRESSURE VESSEL MILD STEEL  
WITH NO STIRRER AND NO METERING PUMPS  
BIOSTIL IS STAINLESS STEEL PRESSURE VESSEL, STEAM  
STERILISABLE, STIRRED, METERING PUMPS

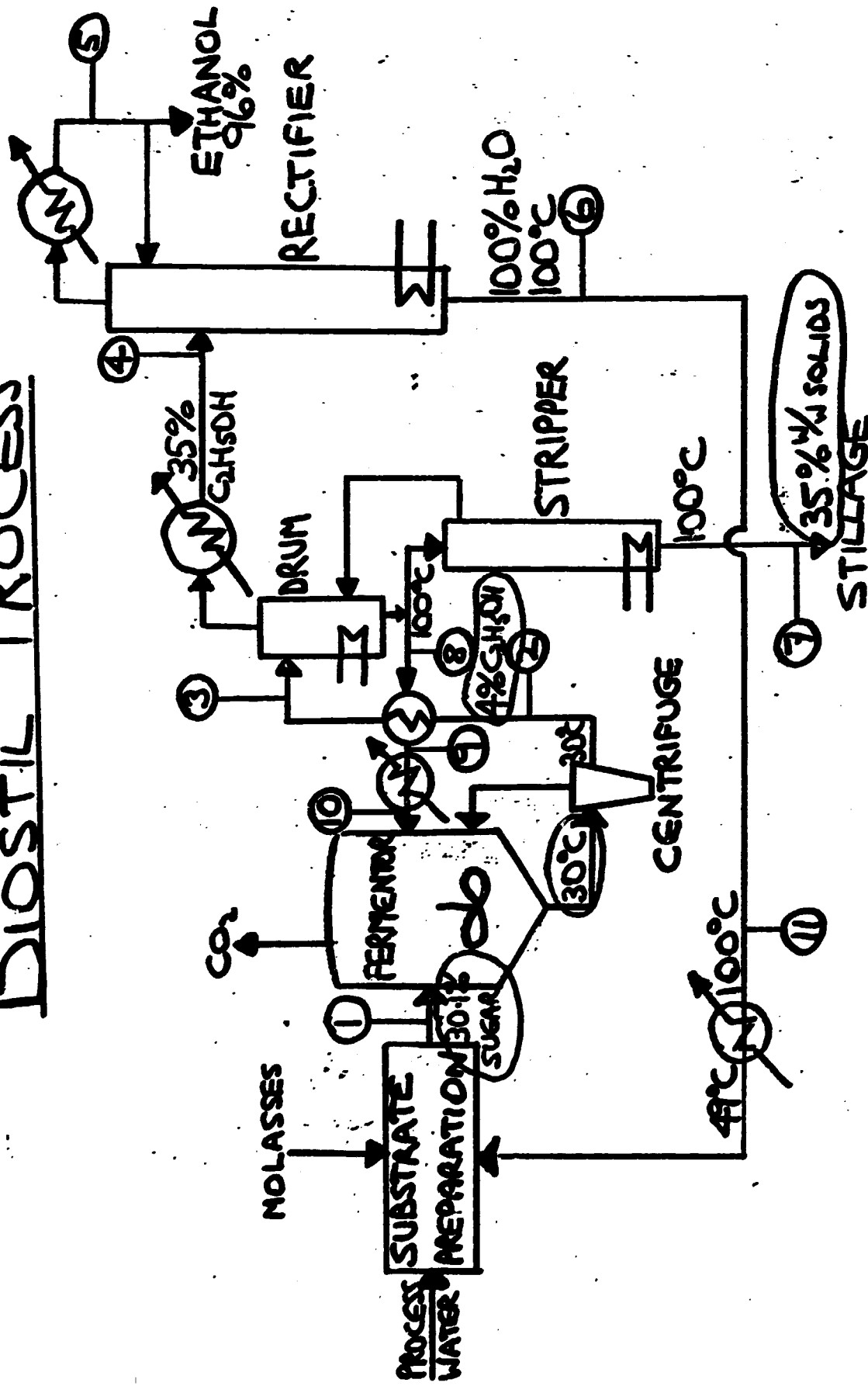
5. THEY ADMIT BIOSTIL NEEDS 25% LARGE CENTRIFUGE  
BUT A CONVENTIONAL FERMENTATION SYSTEM NEEDS  
NO CENTRIFUGE. PEOPLE CLAIM  $C_2H_5OH$  LOSS OCCURS  
DURING CENTRIFUGING.

6. I HAVE USED 35%  $w/w$  DS FOR STILLAGE IN MY  
CALCULATIONS. IN CORRESPONDENCE THEY CLAIM  
BIOSTIL WILL ONLY GIVE 30%  $w/w$  DS WITH THIS MOLASSES.

7. WITH OSMOTOLERANT YEAST CONVENTIONAL  
BATCH FERMENTATION COULD REACH HIGHER THAN  
12.5%  $w/w$  DS. A USEFUL RESEARCH PROJECT FOR CHINA.

8. CONVENTIONAL FACTORY GIVES 9-12 LITRES STILLAGE/LITRE ETHANOL  
DEPENDING ON STILLAGE RECYCLE.

# BIOSTIL PROCESS





## METHODS OF DISPOSAL IN SUGAR MILL

1. SPRAY ON BAGASSE GOING TO BOILER  
(OR COAL)
2. SPRAY INTO SUGAR MILL BOILER
3. SPRAY INTO CHIMNEY
  - a) Before dry collectors
  - b) After collectors + scrubber
4. MIX WITH FILTER CAKE  
(FEED TO FISH PONDS)
5. IRRIGATE ON CANE FIELDS.

---

<u>MILLAQUIN FACTORY 7900 TCD.</u>		
	BOD(TPD)	ASH(TPD)
SUGAR FACTORY	14.5	95.6*
DISTILLERY	18.9	12.1*

\* FILTER CAKE 37.9 TPD ; BAGASSE 57.7 TPD

\* 70-80% SOLUBLE.



# FILTER CAKE-A GOOD SOIL ADDITIVE

	CAKE	STILLAGE
H <sub>2</sub> O %	74	87.5
Ash %	8.6	3.2
N %	0.48	0.3-0.75
P %	0.31	0.02
K %	0.02	0.85*
Pol %	4	
BOD <sub>5</sub> mg/l	40,000	50,000
TONNES/DAY	440	380

\*CHINA CANE SOILS ARE  
DEFICIENT IN POTASH.

John WARNER, 1981 Tour of GUANDONG with MIN. of LIGHT INDUSTRY

# FILTER CAKE + STILLAGE



D  
a  
v  
y

FILTER CAKE: MOISTURE % 74  
 POL % 1.59  
 JUICE PURITY 85  
 JUICE POL % 2.15

SJM GIVES 90.5%

FUGAL TO 40% H<sub>2</sub>O = 4.8 TPD EXTRA SUGAR

(0.5% OF FACTORY PRODUCTION)

ADD STILLAGE AT 10%. CAN USE 66% OF STILLAGE

ADD STILLAGE AT 13.3% CAN USE ALL STILLAGE

N  
P  
K

FKTER CAKE

0.5%

0.3%

0.02%

FILTER CAKE + STILLAGE

0.5%

0.2%

0.49%

SLOPPING BACK MAY REACH THIS



BIOLOGICAL TREATMENT DO WHAT NATURE DOES, BUT MORE INTENSIVELY.

1. ANAEROBIC DIGESTION

THERMOPHILIC 55°C

MESOPHILIC 35°C

TALK OF SEWAGE.

2. ANAEROBIC FILTERS

3. LAGOONS

4. ACTIVATED SLUDGE

5. TRICKLING FILTERS.

AEROBIC 0.3-0.6 Kgs SLUDGE/Kg COD REMOVED

ANAEROBIC 0.1-0.15 kgs SLUDGE/Kg COD REMOVED.

WHAT TO DO WITH SLUDGE?

IF PUT ON LAND THEN WHY USE PROCESS

AS INORGANICS ARE IRRIGATION PROBLEM AND ASH UNCHANGED



DIGESTION - GAS YIELD

STARCH  $\rightarrow$  830 litres/kg of 50%  $\text{CH}_4$

PROTEIN  $\rightarrow$  830 litres/kg of 70%  $\text{CH}_4$

CARBON  $\rightarrow$  1970 litres/kg of 50%  $\text{CH}_4$   
IN ORGANIC MATERIAL

SLUDGE YIELD 0.1-0.2 kg/kg  $\text{BOD}_5$  REMOVED



Day



# DIGESTION - ENERGY FACTS

DIGESTOR GAS @ 50% CH<sub>4</sub> = 20,000 kJ/m<sup>3</sup>

At 26.7°C, to evaporate 1 kg H<sub>2</sub>O

$$= (100 - 26.7) \times 4.18 = 2233 = 2540 \text{ kJ/m}^3$$

$$1 \text{ m}^3 \text{ BIOGAS evaporates } \frac{20000}{2540} = 7.87 \text{ kg H}_2\text{O}$$

$$\text{Engine efficiency} = 20\%$$

$$1 \text{ m}^3 \text{ BIOGAS} = 20,000 \times 0.2 = 4,000 \text{ kJ}$$

$$\text{OR} = 1.13 \text{ kWh}$$

$$\text{OR } 0.2 \text{ kW ENGINE FOR } 5.5 \text{ hrs.}$$



# DIGESTORS

## 1. TRADITIONAL

Tanks, lagoons: 30 DAYS Residence Time  
5,000 - 10,000 mg/l BOD<sub>5</sub>

## 2. MODERN

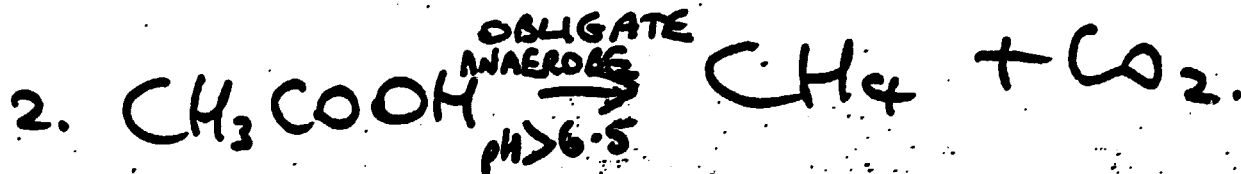
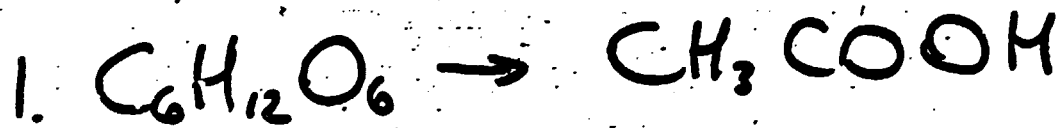
2.1. FILTER SYSTEMS

2.2. FLUIDISED BED SYSTEMS

2.3. SLUDGE BLANKET

2.4. TOWER

2.5. CHINESE TWO STAGE SYSTEM.





# UASB - DIGESTION



## UPFLOW ANAEROBIC SLUDGE BLANKET

LETTINGA, HOLLAND

FEED COD  $60,000 \text{ mg/l}$

LOADING  $12 \text{ kg COD/m}^3 \text{ d}^{-1}$

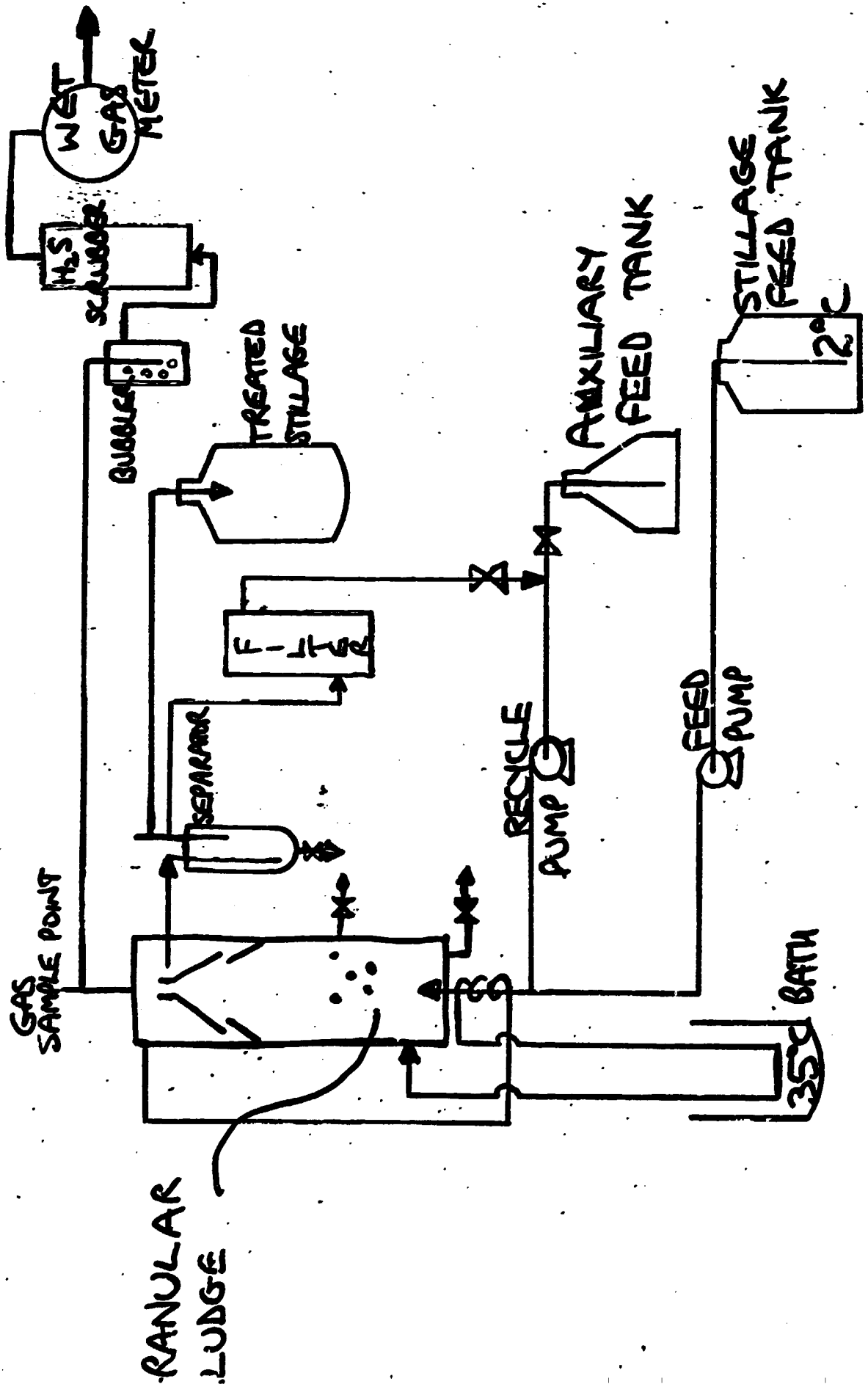
REDUCTION  $90-95\% (300-600 \text{ mg/l})$

METHANE  $0.35 \text{ m}^3 \text{ CH}_4 / \text{kg COD}$   
( $3 \text{ m}^3 \text{ CH}_4 / \text{m}^3 \text{ d}^{-1}$ )

HYDRAULIC LOADING  $4 \text{ m}^3 / \text{m}^3 \text{ d}^{-1}$

COD:N:P  $100:2:0.3$

# UASB-DIGESTER



DAVY

# UASB DIGESTION

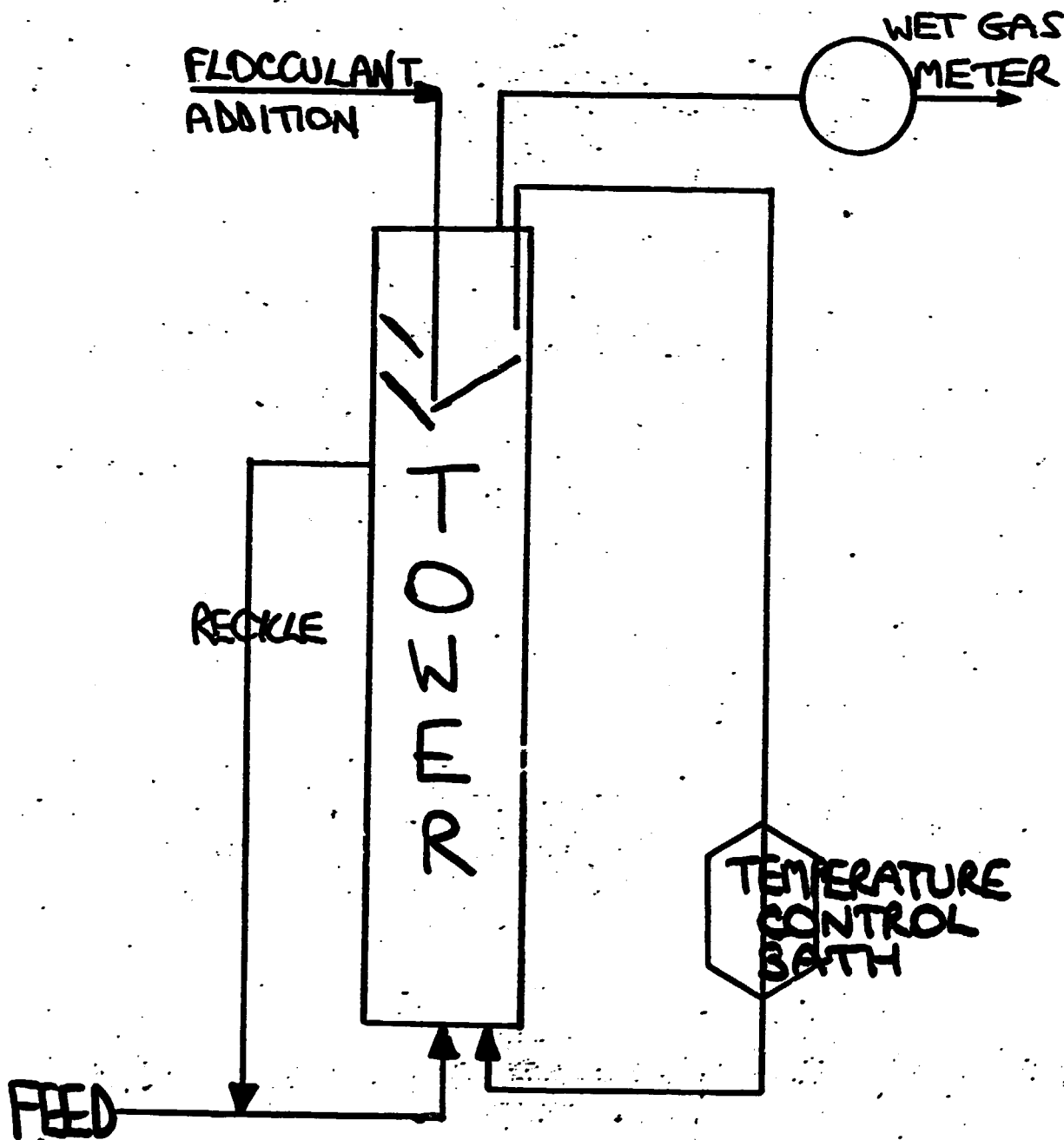


1. STANDER G.J. (1967) Proc. of 22nd Indust Waste Conf., p.892-907
2. HALBERT E.J. (1980) Proc. of 16th Conf. Inst of Brew (Australia) p219
3. RIBIERO C.C. (1981) Proc. Biochem. 16 (3) 8-13
4. RIERA F.S. (1982) Biotechnol. Lett. 4(2) 127-132
5. BRAUN R. (1982) Proc. Biochem. 17(4) 25-27
6. LETTINGA G. (1983) Wat. Sci. Tech. 15, 177-195
7. LETTINGA G. (1980) Biotechnol. Bioeng. 22(4) 699-734

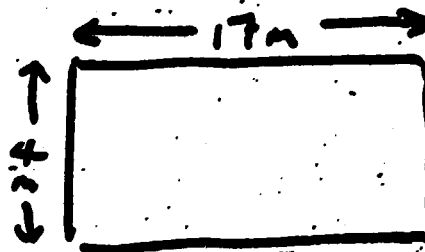


# TOWER DIGESTER - UFFD

## UpFlow Flocculated Digester



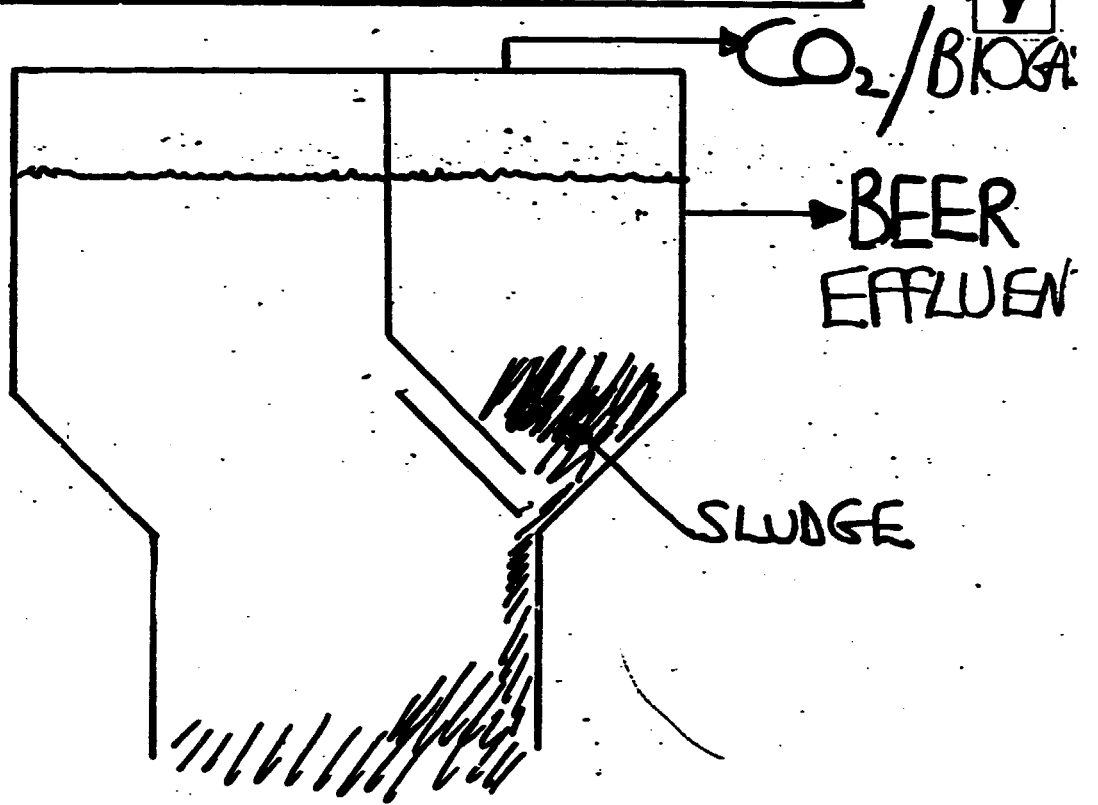
PILOT 0.01m<sup>3</sup>



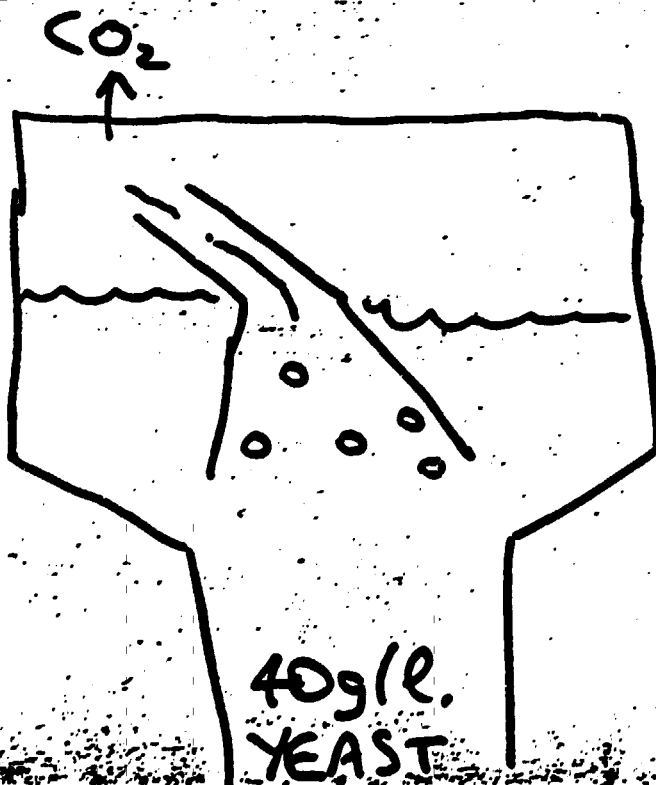
FULL SCALE (1000m<sup>3</sup>)



# TOWER (TOP DESIGN)



## APV Co. - BEER MAKING



# UFFD - DIGESTION.



FEED COD 5,000-10,000 mg/l

LOADING 25 kg COD/m<sup>3</sup>d<sup>-1</sup>  
(1-1.4 kg COD/kg V SS)

REDUCTION 95%

METHANE 0.32 m<sup>3</sup> CH<sub>4</sub>/kg COD

HYDRAULIC LOADING 2.5-5 m<sup>3</sup>/m<sup>3</sup>d<sup>-1</sup>

SLUDGE PRODUCED 0.1-0.15 kg/kg COD  
(7½% of STILLAGE AS 5% SLUDGE)

FLOCCULENT 40-60 mg/l ZETAG 88N  
ZETAG 76.

1500 mg/l S → 440 mg/l SULPHIDE FeS ppt. ∴ FeCl<sub>2</sub> add

NEVER CANE MOLASSES STILLAGE.



# ANAEROBIC DIGESTION (THERMOPHILIC)

REF.	INITIAL BOD mg/l	LOADING kg BOD/m <sup>3</sup> /d	HOLDING TIME (DAYS)	% BOD REMOVAL
SEN				
BASU		4-1	7-25	84-92
BASU		2-3.5	10	97-88
BORUFF	17,000	2.8	6.0	72 vs
BORUFF	17,000	8.5	2.0	58 vs
BUSWELL	15,000	2.4		99
DAVIDSON	700	0.7	1.0	93
DAVIDSON	700			85
STANDER		8.8		
STANDER		11.7		
JACKSON			10	60
ONO		6.4		96
SONADA	6:1 dilution	4.3		70
SONADA	3:1 dilution	15.3		70

## ANAEROBIC DIGESTION (MESOPHILIC)

D  
a  
v  
y

REF.	INITIAL BOD mg/l	LOADING kg BOD/m <sup>3</sup> /d	HOLDING TIME (DAYS)	% BOD REMOVAL
BIAGGI				87
HAURY	10,000			80
MERKEL	7,000		3.7	
PETTET		8.8 vs	3.75	55 TOC
RADHAKRISHNAN		3	10	80
DE		1.9	5	89
CILLIE		3.2	6.9	97.3
GHOSE			12	70
SEN		3.77		91.6
HAITT	100,000 <sup>COO</sup>	5.95	16.7	71.9
TANAKA		13.4 vs		
VITKOVSKAYA	15,000	1.84		95
BURNETT				30-50
KEENAN	22,620	2.83	8	35
SHEA	55,000	0.09	22.1	80
ROTH	55,000	9.86	5.5	80
SANDERS	12,320	1.23	10	98.8
CHUANG	4,500		10	85



# ESTIMATE OF EFFLUENT TREATMENT NEEDS.

BASED ON 58,000 LITRES PER DAY ETHANOL.\*

	AREA (HA.)	POWER (KW)	SLUDGE* PRODUCED	% BOD REMOVED	EFFLUENT mg/l.
ACTIVATED SLUDGE					
Shrs - 12hrs 1. HIGH RATE	2	400	250	70	60
1 DAY 2. CONVENTIONAL	4	10.00	270	90	20
2-3 DAYS 3. EXTENDED	10	20.00	170	95	15
AEROBIC LAGOON	80	2000	120	80	20
FACULTATIVE POND	500	0	50	70	?
ANAEROBIC LAGOON	800	0	50	80	?

\* THIS SLUDGE IS TONNES PER DAY WET WHICH MUST BE SPREAD ON LAND.

\* 60,000 mg/l BOD<sub>5</sub>; 80,000 mg/l COD IEMILLAQUIN SIZE



# ACTIVATED SLUDGE

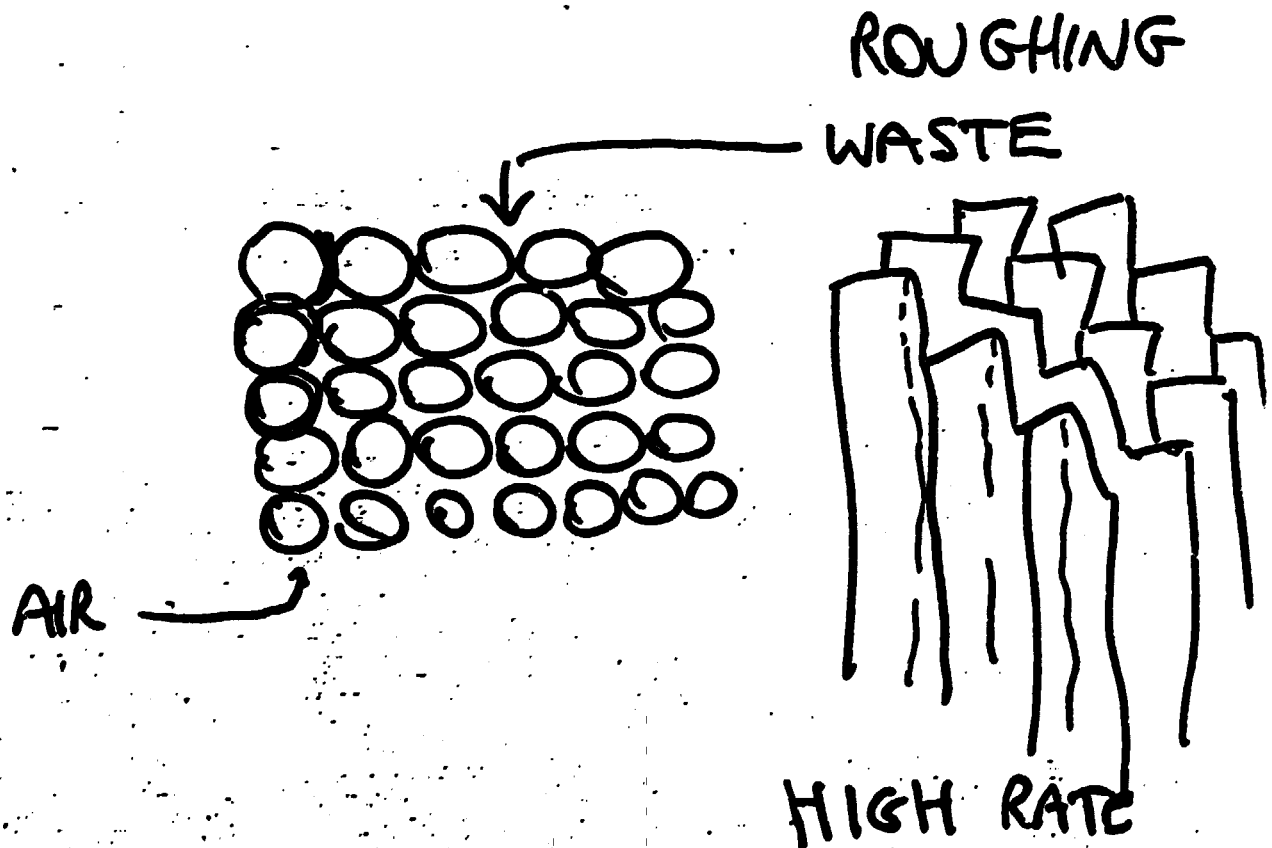


REF.	INITIAL BOD mg/l	LOADING kg BOD/m <sup>3</sup> /d	HOLDING TIME (DAYS)	% BOD REMOVAL
LONDONG	15400	0.97-6.21	18-51	82-65
	150			80
BURNETT	11,000	64 cod	4.5	33.3 cod
BIAGGI				28
BURKHEAD	266-564	0.29		91.5
ECKENFELDER		0.45		85-96
MATSUMOTO		0.15		85
WU	2350	2.45	24	93
	2350	6.75	8	} 96
		0.64	16	
BOLTON	3000	46		90

# TRICKLING FILTER

D  
a  
v  
y

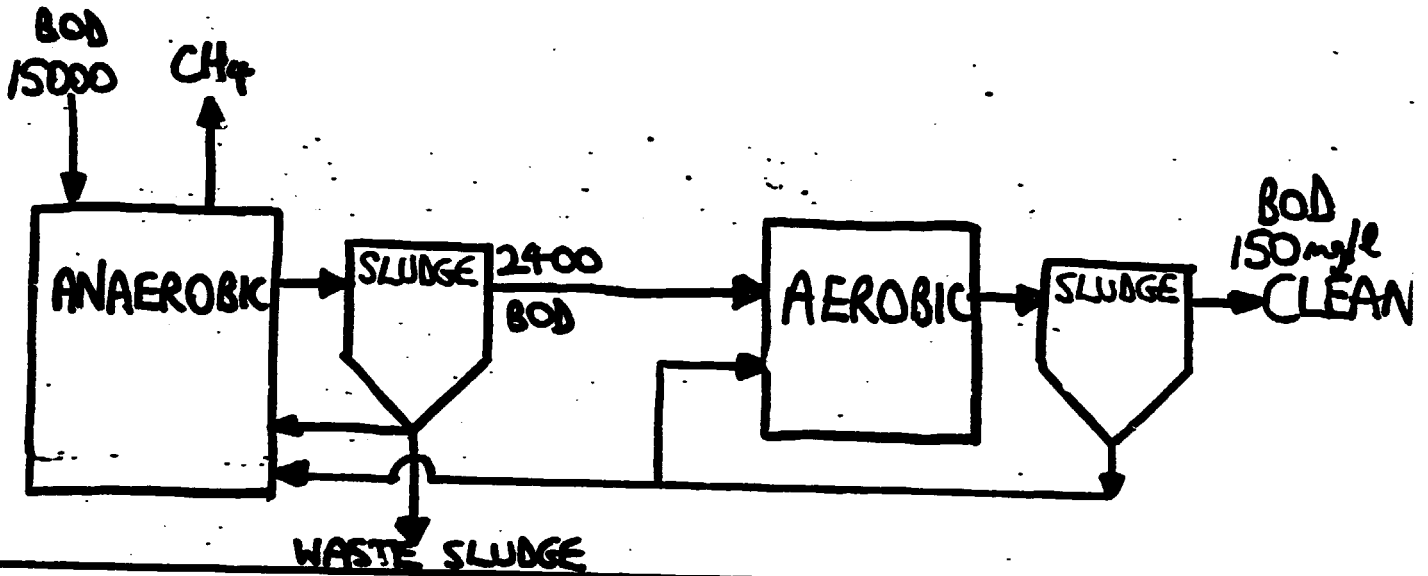
REF.	INITIAL BOD mg/l	LOADING kg BOD/m <sup>2</sup> /d	RECYCLE RATIO	% BOD REMOVAL
ROBERTS		15-0.66	3-11	33-77
DAVIDSON		0.45	4	93
GURNHAM				
ECKENFELDER				50
CHIFFERFIELD	1000			98
CAMPBELL		0.87-315		
CALLELY		1.7	3	66
BASU		0.23		
		7		



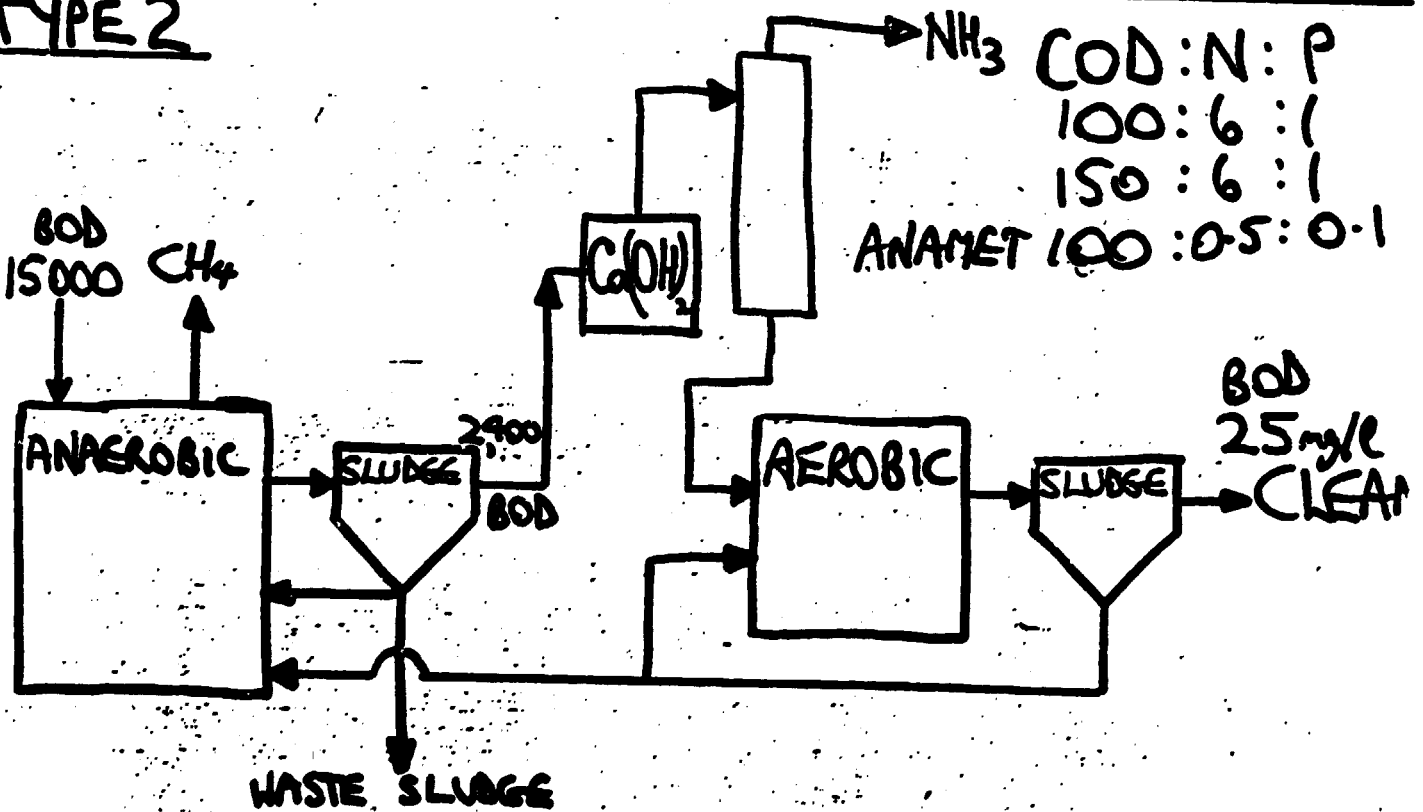


# ANAMET PROCESS

## TYPE 1



## TYPE 2



INCINERATION DOES NOT DESTROY ALL THE COD  
STILLAGE AT 10% W SOLIDS

CONCENTRATE AT 60% W SOLIDS.

CONDENSATE FROM EVAPORATORS IS  
ALMOST THE SAME VOLUME AS  
ORIGINAL STILLAGE.

10 PARTS SOLID 90 WATER.

10 PARTS SOLID 6 WATER = 60% SOLID

100 PARTS STILLAGE gives 84 CONDENSATE

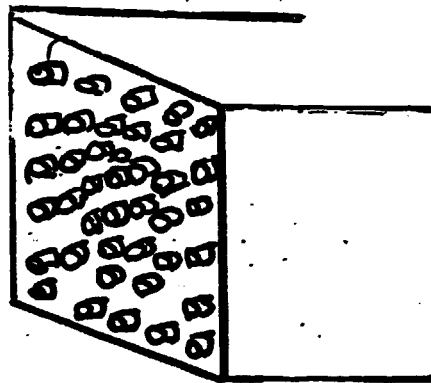
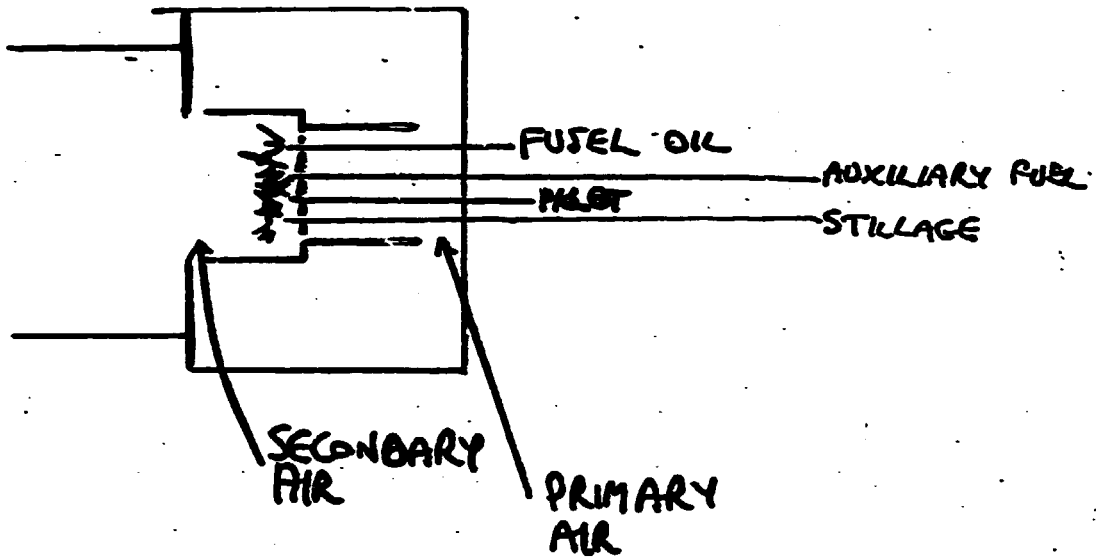
REDUCED THE VOLUME BY 16% CONDENSATE IS POLLUTING

pH = 3.3 COD = 3,500 mg/l





# MULTINOZZLE BURNER



# DIGESTION vs. INCINERATION.

FOR 180,000 LITRES PER DAY ETHANOL DISTILLERY.

D  
A  
V  
Y

## INCINERATION:

STEAM

396ph steam raised - 186ph for evaporation of dinder = 504 TPD

EFFLUENT IS ASH AND WATER (CLEAN) 000

AND \$12-15 MILLION IN CAPITAL \*  
AND THERE ARE A NUMBER IN OPERATION

## DIGESTION:

ASSUMING 80% EFFICIENCY GAS BOILER

-258 TPD

EFFLUENT IS 240-360 TPD WET SLUDGE AT 5% SOLIDS

AND 900 M<sup>3</sup>PD OF 10,000 mg/l CO<sub>2</sub> LIQUID.

AND \$12 MILLION IN CAPITAL.

AND DIGESTOR NEEDS POWER

AND THERE ARE NO FULL SCALE FACTORIES ON CANE MOLASSES STILLAGE

\* THE FACTORY IN THAILAND COST \$25 MILLION BUT IT WAS THE FIRST IN WORLD. A SECOND FACTORY WOULD ONLY BE \$12-15 MILLION

# BIOMASS FROM CANE MOLASSES STILLAGE

Davy

ORGANISM	REF.	BATCH TIME (hr)	YIELD kg/m <sup>3</sup>	% ORGANIC REMOVED
CANNA UTILIS	PAZ(1973)	9-12	18.75	
CANNA UTILIS	CHANG(73)	12	24	
YEASTS	IL'INA(69)		12-16	
FODDER YEAST	ISIK(77)		13.35	
CHLORELLA VULGARIS	OKUBA(67)	96		85
TRICHOSPORA FUNGI	IL'INA(69)		18-21	
	ROLZ(75)		11-19	56

45.

① STERILIZE

② ADD  $(NH_4)_2HPO_4$ ,  $CON_2H_4$ ,  $CaHPO_4$ ,  $(NH_4)_2SO_4$ , MOLASSES  
YEAST EXTRACT, ANTIFOAM, CHLORAMPHENICOL

③ 55-65 VOLUMES AIR PER VOLUME LIQUID PER HOUR.

IE. 400 VOLUMES AIR PER VOLUME STILLAGE

④ FERMENTATION AT 30-32°C COOLING WATER UP TO 37°C IN TROPICS  
SO MAYBE NEED REFRIGERATION.





# YEAST FROM STILLAGE

ACETIC ACID

GLYCEROL

FRUCTOSE

LACTIC ACID

SUCCINIC ACID

MALIC ACID

} USED

## LEAVING

DIFFICULT TO REMOVE

ORGANICS.

NO NITROGEN

NO PHOSPHATE

# BIOMASS FROM STILLAGE

## PROBLEMS.



1. ECONOMICS NOT FAVORABLE.
2. STILLAGE ONLY PROVIDES CARBON  
CELLS 4% PROTEIN IE. 7.4% NITROGEN  
AND 50% CARBON
3. MUST ADD NITROGEN  
UREA,  $(NH_4)_2SO_4$ ,  $(NH_4)_2HPO_4$   
 $CON_2H_4$
4. MUST ADD PHOSPHATE  
 $(Na)_3PO_4$ ,  $(NH_4)_2HPO_4$
5. FERMENTATION AT 30-32°C  
EXOTHERMIC SO MUST COOL 3870  $cm^3/gm$  YEAST GROW  
COOLING WATER IN EUROPE LESS THAN 20°C  
COOLING WATER IN TROPICS OFTEN 37°C  
SO PERHAPS REFRIGERATION.
6. LARGE ELECTRICITY DEMAND  
GROWING BIOMASS VERY AEROBIC.  
400 VOLUMES AIR PER VOLUME STILLAGE.

SEE PAGE 2.

# BIOMASS FROM STILLAGE.

## MORE PROBLEMS



### 7. MUST STERILISE STILLAGE

MUST BE CERTAIN ONLY PURE STRAIN OF FUNGI, YEAST, ALGAE GROWS OTHERWISE PERHAPS TOXIC PROBLEMS.

### 8. WHAT TO DO WITH WASTE?

a) STILL  $\frac{1}{2}$  ORIGINAL ORGANIC STRENGTH  
SAY 30,000 - 40,000 mg/l BOD<sub>5</sub>.

b) PROBABLY HIGHER SALTS THAN BEFORE

c) EASIEST ORGANICS NOW REMOVED

d) INCINERATION GIVES ONLY HALF HEAT  
I.E. NOW NOT SELF SUFFICIENT

e) DIGESTION MUCH HARDER, LESS THAN  $\frac{1}{2}$  CH<sub>4</sub>

### 9. CENTRIFUGED BIOMASS NOT CLEAN

AS CANE MOLASSES STILLAGE IS DARK BROWN AND FULL OF ASH, YEAST WILL HAVE STRONG TASTE AND COLOUR.



# CANE MOLASSES DISTILLERY

TOTAL DISTILLERY (OPERATIONAL) 100

COST OF EQUIPMENT\* (AT SITE) 29

FERMENTATION EQUIPMENT (AT SITE) 7

ALL TANKS, VESSELS, DISTILLATION COLUMNS, PUMPS, HEAT EXCHANGERS, VALVES, COMPRESSOR, BOILER, TURBINE. ERECTED.

NOT INCLUDING BUILDING, PIPING, INSTRUMENTS, ELECTRICAL



## COST BREAKDOWN OF ETHANOL

	%
CAPITAL COST	28
RUNNING COST*	72

\* INCLUDES LABOUR 6%, CHEMICALS 2%,  
MAINTENANCE 7%, MOLASSES 51%\*

\* Molasses @ \$72/tonne and 250 litres/tonne



## VALUE IN IMPROVING FERMENTATION

A 25% SAVING IN  
FERMENTATION EQUIPMENT COST

=

A 1.5% SAVING IN FACTORY

=

0.4% SAVING IN  $C_2H_5OH$  COST.

YET PEOPLE SAY THEY  
CAN REDUCE  $C_2H_5OH$  COST  
BY 25-50%, BY CHANGING  
FERMENTATION



## VALUE IN IMPROVING FERMENTATION

A 0.8% EXTRA YIELD  
OF  $C_2H_5OH$  FROM MOLASSES  
ALSO GIVES

A 0.4% SAVING IN  $C_2H_5OH$  COST.

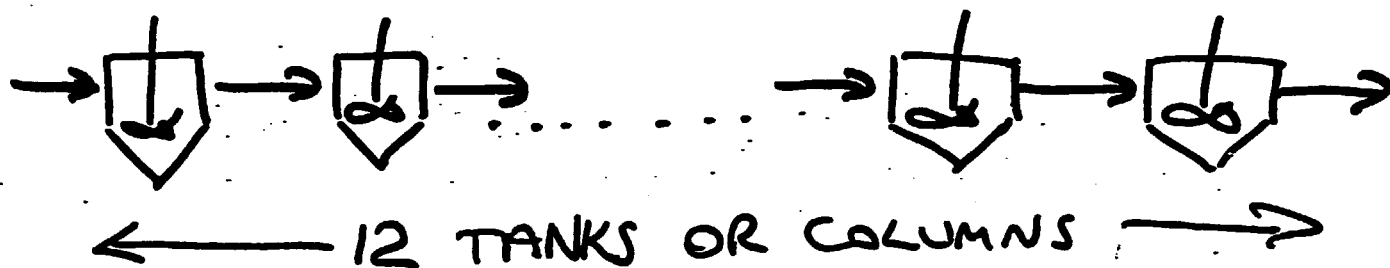
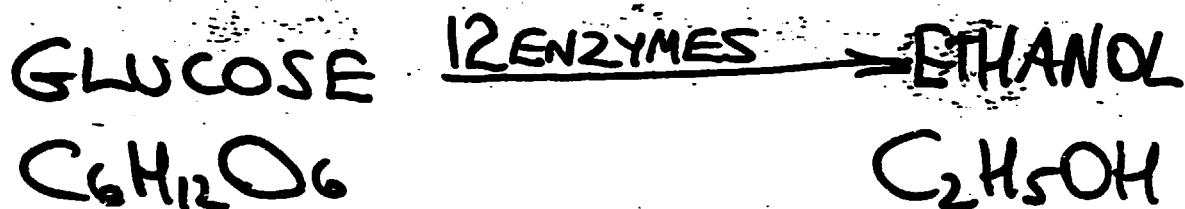
IE. 252 litres/tonne MOLASSES  
NOT

250 litres/tonne MOLASSES.



# IMMOBILISED ENZYMES FOR $C_2H_5OH$ .

12 DIFFERENT ENZYMES CONVERT



CHEAP PRODUCT.

NOT SENSIBLE.

IMMOBILISED ENZYMES NO USE  
 FOR  $C_2H_5OH$  FROM  $C_6H_{12}O_6$ .



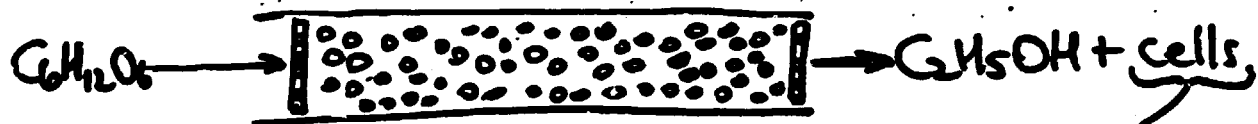
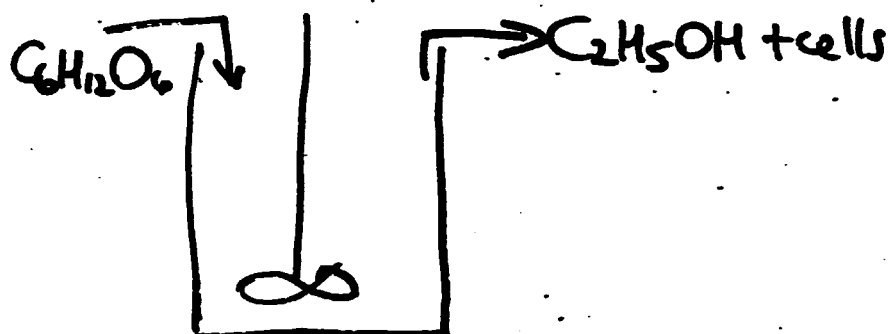
# IMMOBILISED CELLS.



## BATCH.



## CONTINUOUS.



BECAUSE YEAST GROWS.

# IMMOBILISATION METHODS.

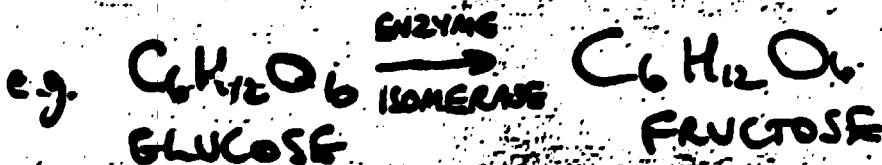
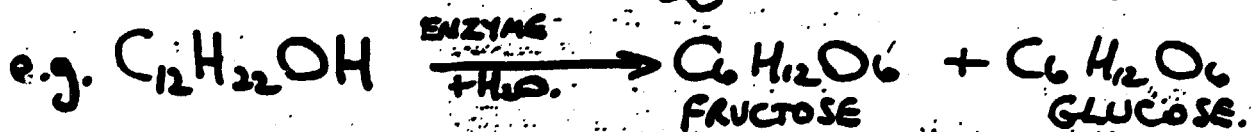
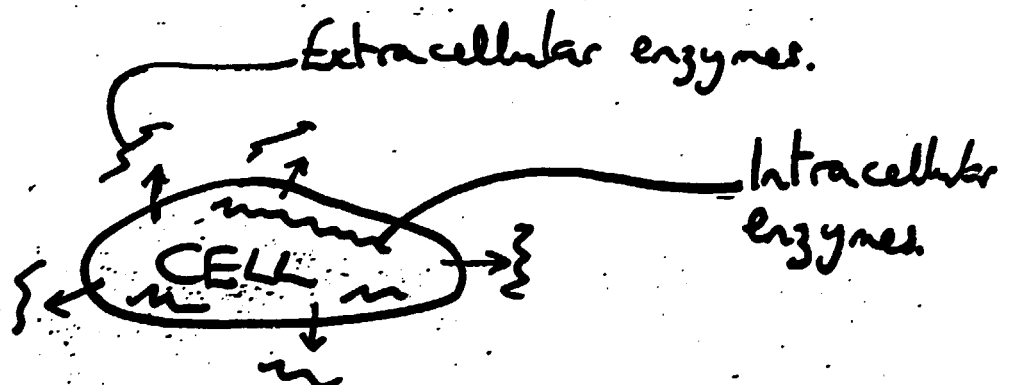
ADSORPTION	CELL	CELL WALL
		CHARGE
SURFACE		AGE
		PH
		SURFACE CHARGE
		SURFACE AREA
ENTRAPMENT		AGAR
		ALGINATE
		CARRAGEENAN
		CELLOPHANE
		ACRYLAMIDE
COUPLING		ISOCYANATE
		GLUTARALDEHYDE
		METAL HYDROXIDE
		AMINO SILAN

# IMMOBILISED ENZYMES + CELLS.

1. Extracellular enzymes stable
2. Intracellular enzymes unstable.

## IMMOBILISATION

- a) Stabilised intracellular enzymes.
- b) Confines them in reactor.
- c) Enzymes usually soluble and are lost with product unless held by immobilisation



## ENTRAPMENT

① 1 ml yeast culture + 50 ml of 4.5% CARRAGEENAN GEL soln @ 37°C

MIX.

② Add drop by drop to 2% KCl soln @ 20°C

MAKE 4 mm diameter drops.

③ Stir gently for 2 hrs

57  
① 5mls of 125g/l yeast added to 50mls of 2% Na alginate @ 37°C

② Extrude as drops into 0.05M CaCl<sub>2</sub> @ pH = 4.5 @ 4°C

(Syringe + 26 gauge needle gave 1.8 mm beads  
18 gauge needle gave 3 mm beads.)

③ Stir slowly for 12 hrs @ 4°C

④ Activate by growing 40 hrs @ 30°C in 10% glucose soln with added yeast extract and peptone



# IMMOBILISED CELLS.

BATCH FERMENTATION NO  
CELLS ALREADY CONTAINED IN FERMENTER.

CONTINUOUS FERMENTATION PERHAPS

BUT

WHAT ARE ADVANTAGES.

1. PLUG FLOW - BETTER YIELD
2. SMALL FERMENTER. - CHEAPER
3. NO NEED TO SEPARATE CELLS.

BUT.

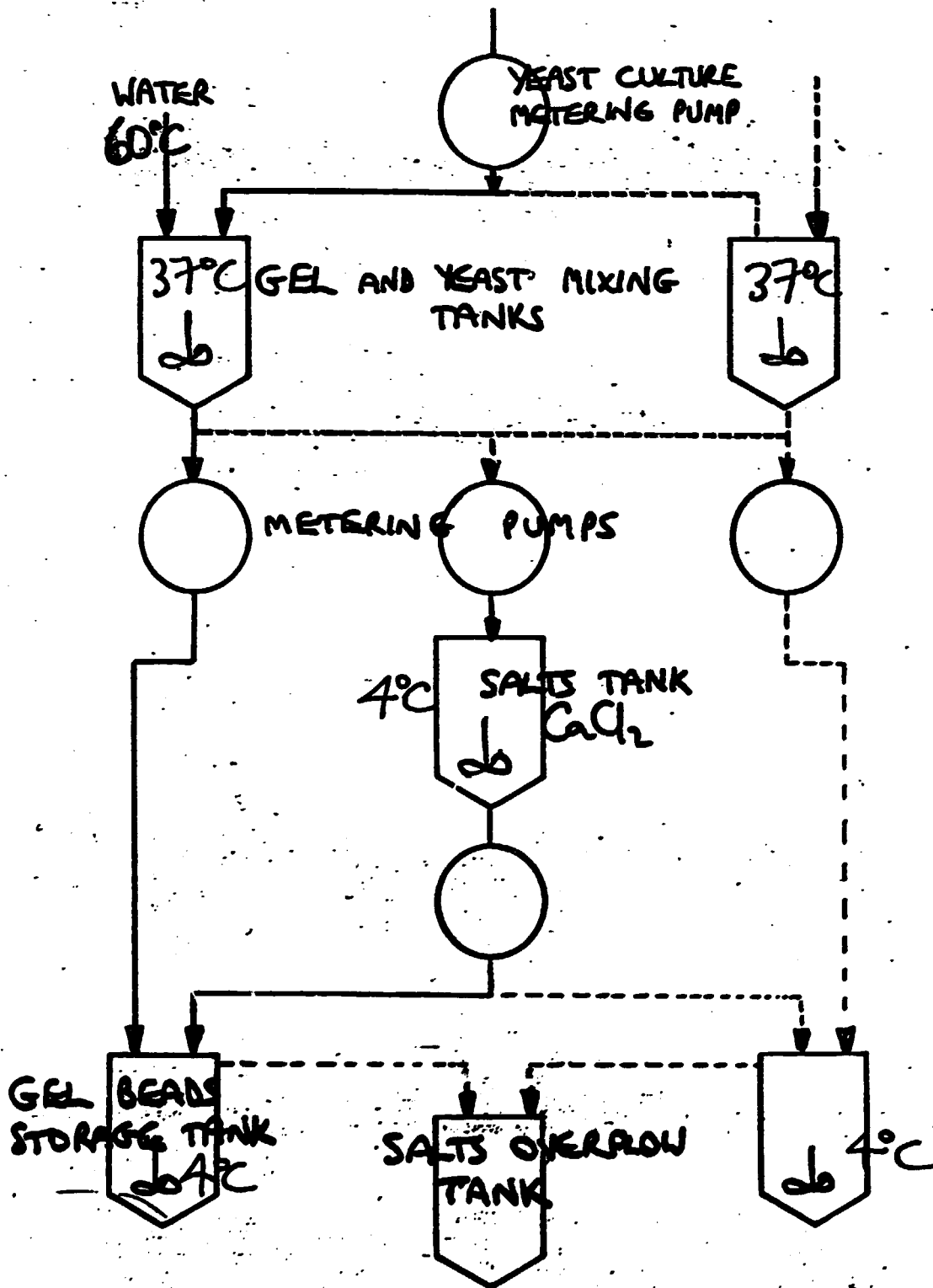
1. MOLASSES DIRTY, CLOG REACTOR (GUMS etc)  
BATCH FLEXIBILITY LOST
2. VERY COMPLICATED TUBULAR REACTOR - EXPENSIVE
3. C<sub>2</sub>H<sub>5</sub>OH PRODUCTION CELL GROWTH LINKED

$$Y_{x/s} = 0.05 = 5\%$$

SO STILL NEED TO SEPARATE CELLS



# IMMOBILISED CELLS - COST.





# IMMOBILISED CELLS. COST

## I. GEL BEADS WITH YEAST IN THEM

- WOULD DRY OUT IF NOT KEPT IN WATER
- HAVE NO GROWTH MEDIUM IN THEM
- MUST BE STORED STERILE.

SO MUST BE MADE AT DISTILLERY.

FOR  $10 \times 10^6$  LITRE PER YEAR DISTILLERY

CAPITAL COST                      \$ 450,000\*

RUNNING COST/YEAR              55,000

\* TOTAL FULLY ERECTED BATCH FERMENTATION SYSTEM  
ONLY = \$1.8 MILLION



# IMMOBILISE CELLS.

## ADVANTAGES.



1. INCREASE REACTION RATE AS MORE CELLS
2. HIGHER SPECIFIC PRODUCT YIELD.
3. OPERATE CONTINUOUSLY AT HIGH DILUTION WITH NO WASHOUT
4. ELIMINATES COSTLY FERMENTER DESIGN
5. EASIER CONTROL OF FERMENTATION
6. SUBSTRATE INHIBITION EFFECT REDUCED
7. ETHANOL INHIBITION EFFECT REDUCED

## DISADVANTAGES.

1. COST OF MANUFACTURE NOT COVERED BY LOW PRODUCT COST
2. NO INDUSTRIALISED SYSTEMS ARE IN OPERATION
3. GROWTH OF CELLS BLOCKS VOID VOLUME
4. GUMS + COLLOIDS BLOCK VOID VOLUME
5. ADDITIONAL BARRIER TO DIFFUSION.
6. DIFFICULT TO REMOVE WASTE GAS I.E.  $\text{CO}_2$ .
7.  $Y_{P/S}$  HIGH,  $Y_{X/S}$  LOW BUT % CONVERSION LOW - COST.
8. REACTOR MUST BE STERILISABLE.

# WHY DO RESEARCH?



## 1. TO IMPROVE PRODUCTIVITY

It is sound ECONOMICALLY to get more yield at greater internal cost if it does not affect trade balance.

## 2. TO REDUCE COSTS.

The cost of Research must be recovered by the cost reductions it suggests.

## 3. TO EDUCATE RESEARCHERS.

Unfortunately this is often the first thought in researchers' heads.

## 4. TO REPEAT another COUNTRIES WORK.

This often happens.

NEVER BE AFRAID TO REPEAT OTHERS WORK

BUT **ALWAYS** QUOTE THE ORIGINAL

APPENDIX 4

Itinerary for Dr.R.B. Brook's visit  
(11-02 Post)  
1984

	A.M.	P.M.
Oct. 15th Mon.	- Arrive in Guangzhou	
16th Tues.	- Session	Session
17th Wedn.	- Session	Session
18th Thurs.	- Session	Session
19th Fri.	- Discussion	Discussion
20th Sat.	- Discussion	Discussion
21st Sun.	- Free	Free
22nd Mon.	- Visit Shunde Mill	
23rd Tues.	- Visit Sugarcane Industry Research Institute.	
24th Wedn.	- Discussion and Consultation.	
25th Thurs.	- Discussion and Consultation.	
26th Fri.	- Discussion and Consultation.	Free
27th Sat.	- Back to Australia.	

APPENDIX 5

ITINERARY FOR DR. R. B. BROOKS'S VISIT  
(11-02 Post)  
1984

Oct. 15th Mon. - Arrive in Guangzhou  
8-12 A.M. 2-5 P.M.  
16th Tues. - Session Session  
17th Wedn. - Session Session  
18th Thurs. - Session Session  
19th Fri. - Discussion Discussion  
20th Sat. - Discussion Discussion  
21st Sun. - Free Free  
22nd Mon. - Visit Shunde Sugar Mill and Distillery -  
Consultation.  
23rd Tues. - Visit Sugarcane Industry Research Institute.  
8-12 A.M. 1-5 P.M.  
24th Wedn. - Discussion and Consultation.  
25th Thurs. - Discussion and Consultation.  
26th Fri. - Discussion and Consultation.  
27th Sat. - Visit Guangzhou Sugar Mill - Consultation.  
28th Sun. - Free  
29th Mon. - Visit Shitou Sugar Mill and Distillery -  
Consultation  
8-12 A.M. 1-5 P.M.  
30th Tues. - Discussion and Consultation  
31st Wedn. - Discussion and Consultation  
Nov. 1st Thurs. - Discussion and Consultation  
2nd Fri. - Discussion and Consultation  
3rd Sat. - Free  
4th Sun. - Back to Australia.

**Davy Agro**

**APPENDIX 6 OBSERVATIONS**

## **Davy Agro**

### SUGAR INDUSTRY OF CHINA

#### GENERAL

Using the definition of acceptance by most countries, China does not produce any refined sugar from cane or raw cane sugar, rather it is white sugar. As in most developing countries a non centrifugal brown sugar is also produced.

Most of the equipment is relatively old, or old style, with the largest factories those that had milling trains imported in the 1950's and 1960's. Since that time, Australia has produced a much more efficient 5 roll mill which together with feed chutes has vastly improved performance over a 3 roll mill.

Although no factories were running during the visit, the installed equipment would tend to suggest that cane preparation could be improved.

It is clear from many discussions with Institute and factory staff that there is a positive move to try and develop a cane payment system based on quality rather than weight. Lectures were given on cane payment systems based on quality which may be the subject of a separate report. The major advantage of a system of cane payment based on quality is that it gives the farmer and the factory real incentive to improve their performance and get paid for it.

China has one advantage over many of its developing country near neighbours, farmers are assigned to a particular factory and cane transport is handled either by the factory or by a transport company which handles all the cane for a factory. This gives China the opportunity to schedule cane deliveries in a way that few countries can e.g. Australia. Cane can thus be delivered freshly cut.

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There were discrepancies in the pol balances of the factories we were given some results for. This could be because of pol inflated by dextran in stale cane.

There is a real need for a better quality refined sugar for industrial users. This will enable China to produce goods for internal use and also for export which meet all the internationally accepted criteria. These goods would include fruit juice, soft drink, pharmaceutical use, canned fruits etc.

Fibre is not measured by analysis now in any of China's factories, it is calculated by weighing and analysing mixed juice and analysing bagasse, weighing imbibition water and assuming no loss of pol in the milling train. Although the bag method for fibre was time consuming, it is a backward step to abandon its use.

### NCIRC

The National Cane Sugar Research Institute lies 40 minutes S.E. of the city of Guangzhou. It was founded in 1957 as a combination of the Industrial and Agricultural Institutes founded earlier. There are eight departments,

- |                        |  |
|------------------------|--|
| <b>Industrial</b>      | 1. Analysis/Quality Control                |
|                        | 2. Automation/Instrumentation              |
|                        | 3. Pollution Control/Byproducts            |
|                        | 4. Sugar Technology                        |
| <b>Agricultural</b>    | 5. Cane Breeding                           |
|                        | 6. Agronomics                              |
|                        | 7. Plant Protection                        |
| <b>General</b>         | 8. Information/Library                     |
| <b>and three farms</b> | 1 Hainan Breeding Station (53 ha)          |
|                        | 2. Jianjiang for new varieties on red soil |
|                        | 3. Guanzhou (80 ha)                        |

There are 700 workers which includes 200 technologists and 100 administrative people. The top staff are very knowledgeable about sugar in other countries.

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Some of the major investigations made in recent years have been

- o Diffusers (first full scale factory in 1964)
- o Sulphitation tubular reactor (in 1964)
- o Shredder (in 1975)
- o In-line pH meter (1983)

Presently, they are researching:

- o a particle board from bagasse process (two factories in production).
- o Chinese rum (not a popular drink)
- o phosphatation/flotation for refined sugar
- o a turbidimeter
- o a white sugar reflectance meter
- o a five roll mill

These subjects are all reasonably well documented in the literature and so there should be little difficulty in transferring the information to Chinese context.

The Institute generates cash flow by making instruments in its Automation/Instrumentation department for the sugar industry. This, on the surface seems a good idea, in that it uses the facilities and technical resources to the full. However, the cash flow generated could be so useful in funding research at the Institute that it takes precedence over research and development. The in-line pH meters are guaranteed for life which seems like a good idea but of course the Institute does get funding and if an instrument became a loss making venture it would drain funds from the research budget.

It would seem that the first factory/major unit operation etc. is developed, designed, and commissioned by the NCIRC but future ones are then designed by the Guandong Design Institute. Both of these Institutes are National Institutes located near Guangzhou.



## Davy Agro

### DISTILLERIES

There is seldom any molasses clarification, pasteurisation or stillage recycle and the molasses is usually very fresh and often very warm. Molasses of this type is usually very "lively" and generates a great deal of foam during fermentation.

The standard batch fermentation is not universally used in China. Shitow distillery used a standard batch system, pitching a fresh yeast culture in each new fermentation, sterilising the dilution water (treated river water) with bleaching powder before using it to dilute molasses. They aim for 8% <sup>v</sup>/v ethanol in about 20 hrs fermentation. No bacteriacides are used during fermentation but antifoam is usually needed. They use a mixed yeast culture to give both fast fermentation and higher final ethanol concentration.

Many distilleries in China have converted to a continuous fermentation system using from 6-12 vessels in series. Usually, molasses is fed to the first two in the series and these are the only ones which need water cooling. Bacteriacides are added to control infection. (There are some remarks in the fermentation section of the main report which may be considered valid here).

There is a major worry in most distilleries that there is a large residual sugar content in stillage, causing the systems to lose yield. This resulted in many discussions and advice on how to confirm whether the substances being measured as residual reducing sugar were actually fermentable e.g. furfural will be detected by the reducing sugar method but is not fermentable. The HPLC will soon confirm whether the so called residual reducing sugar is fermentable.

Distillation systems are similar throughout China, similar to most overseas distilleries.

- o wash column (22 trays)
- o aldehyde column
- o rectification column (67 trays)

to make 95% industrial spirit from an 8% ethanol feed.

## **Davy Agro**

It is interesting that even today, columns are made out of cast iron which would be too expensive for most countries in the world. In China, stainless steel is more expensive than cast iron for such equipment. Hot stillage is very corrosive and again, cast iron pipe is an ideal way to handle it.