Invert Sugar from Sugar Cane Molasses

- A Pilot Plant Study

by

Hendrik Nicolaas Petrus Stolz

Thesis submitted in partial fulfillment
of the requirements for the Degree

of

Master of Science in Engineering
(Chemical Engineering)

in the Department of Process Engineering
at the University of Stellenbosch

Supervised by
Prof. J.H. Knoetze

STELLENBOSCH
April 2005

Declaration

I, the undersigned, hereby declare that the work contained in this thesis is my own original work and that I have not previously in its entirety or in part submitted it at any university for a degree.

Signed in Malelane on this 23rd day of November 2004.

HNP Stolz
13545353-2000

Synopsis

An investigation was done into the recovery of invert sugar from sugar cane molasses. A pilot plant was designed and constructed to evaluate the clarification and separation of molasses to produce invert sugar syrup. The aim of the pilot plant was to prove the process and deliver data so as to facilitate the design and prove the financial viability of a commercial plant. The pilot plant had to process 300 kg/day of molasses.

The clarification of molasses by centrifugal separation, a known desludging process, did not produce a product of acceptable quality which could be used in a chromatographic separator. The results were disappointing. The product obtained was also not suitable for dead end pressure filtration. The turbidity remaining after the centrifugal separation also did not respond to a second flocculation process.

Conventional settling clarification was investigated. Seven factors that could influence the consolidation and settling of suspended solids in molasses were identified, namely: the age of the diluted molasses, the temperature of the flocculated mixture, the variations across various batches of flocculant, the effect of reaction time of the phosphoric acid, the optimum flocculant dosing concentration, the optimum concentration of the molasses solution and the effect of increasing the acid dosage.

The optimum conditions to clarify molasses through settling were found to be: fresh molasses, at 28 Brix and 60℃, allowing 10 min intervals between acidification with 3,75 g (as 100%) phosphoric acid/ kg dry material (assumed equal to Brix) and neutralisation with 5 g (as 100%) caustic/kg dry material (assumed equal to Brix), flocculation with any batch of flocculant 6195, dosed as a 1000 ppm solution. Commercial equipment was evaluated. The pilot plant E-cat clarifier was operated at 300 l/h and a thick sludge formed. The overflow was clear and it could be filtered. The molasses obtained was suitable for chromatographic separation.

The recovery of sugars from molasses sludge has economic merit. From the evaluation of centrifugal separation and gravity separation it is clear that gravity separation again is the best method. The sweet-water obtained is consistent within the clarity requirement of 10 NTU/Brix and can be used to dilute raw molasses in the upstream processing step. The clarification process that was developed is patented. [Bekker, Stolz *et.al.* (2001)]

A sugar recovery of 93.9 mass % at a purity of 99.7 mass % from molasses, was achieved using a simulated moving bed, ion exclusion, pilot plant. The operating conditions for this performance were: feed flow at 14 l/h and at a temperature above 60 ℃; water flow at 63 l/h and at a temperature above 65 ℃; extract flow at 21 l/h; raffinate flow at 56 l/h; loop flow at 78 l/h and step time at 1326 seconds. This relates to the following bed volumes of the various separation zones: Bed Volume Zone 1 = 0.694; Bed Volume Zone 2 = 0.591; Bed Volume Zone 3 = 0.661; Bed Volume Zone 4 = 0.383. There is a trade-off between purity and recovery and a reduction in water usage.

A preliminary environmental impact assessment and conceptual mass balance were done. The proposed plant integrates well into the existing Komati Mill of TSB and does not pose any significant environmental threat. The plant requires certain services from the mill. The mass balance investigated the water and steam consumption of the plant. Process integration was done so as to obtain the optimum utility consumption. The utility consumption of the plant does not exceed the capacity available at the mill. A small boiler is however required to produce steam during the annual mill maintenance period.

Various techniques were used in a cost estimation for the invert sugar plant. The internal rate of return (IRR) is 42% for a fixed capital investment of R94,270,000.00. The net return rate (NRR) for the project is 4%/year, the net present value (NPV) - discounted at a 30% cost-of-capital is R41,782,000.00. The net payout time (NPT) is 5.207 years. The project fulfils the financial requirements set by TSB.

It is now possible and viable to desugarize cane molasses.

Sinopsis

‘n Ondersoek na die herwinning van invert suiker uit melasse is gedoen. Vir die doel is 'n proefaanleg ontwerp en gebou om die suiweringproses te ondersoek en te ontwikkel. Die proefaanleg moes die tegnologie van die skeidingsproses bewys en data genereer, wat gebruik kan word om 'n kommersiële aanleg te ontwerp en ook om die lewensvatbaarheid van die proses te toets. Die aanleg moes 300-kg melasse per dag kon hanteer.

Oorspronklik is gefokus op bekende ontslykingsmetodes met behulp van ‘n sentrifuge. Die resultate was egter teleurstellend en die kwaliteit van die melasse was swak. Die produk was nie geskik vir filtrasie of chromatografiese skeiding nie. ‘n Moontlike verbetering deur sekondêre flokkulasie is getoets maar die ontslyking was nie moontlik nie. Konvensionele flokkulasie en sedimentasie is ondersoek. Sewe faktore wat die vorming, konsolidasie en skeiding van gesuspendeerde soliede deeltjies in melasse beïnvloed is geïdentifiseer, naamlik: die ouderdom van die melasse oplossing, temperatuur, variasies in verskillende lotte van die flokkulant, reaksietyd van fosforsuur in die mengsel, vlak van flokkulant toediening, konsentrasie van die melasse oplossing en die vlak van fosforsuur dosis.

Die optimum kondisies vir skeiding is: Vars melasse by 28 Brix en 60°C, laat 10 min reaksietyd toe vir die fosforsuur wat toegedien word as 'n 3,75 g (as 100%) fosforsuur per kg droë materiaal (aanvaar gelyk aan Brix) en geneutraliseer met 5 g (as 100%) bytsoda/kg droë materiaal (aanvaar gelyk aan Brix), gebruik 20 dpm van enige lot van flokkulant 6195, verskaf deur Montan Chemicals, wat toegedien word uit ‘n 1000 dpm oplossing.

Kommersiële toerusting is daarna getoets. Die loodsaanleg verdikker (van die E-Cat tipe) is gevoer teen 300l/h en het ‘n dik slyk gevorm met ‘n helder bo-loop wat wel gefiltreer kon word. Hierdie proses het ‘n produk gelewer wat voldoen aan die kriteria vir chromatografiese skeiding. Die herwinning van suikers uit melasseslyk hou ekonomiese voordeel in. Uit die evaluasie van sentrifugale skeiding en gravitasie skeiding blyk dit weereens dat gravitasie skeiding die beste metode is om melasseslyk te ontsuiker. Die soetwater is telkens binne die helderheidstandaard van 10 NTU/Brix en kan gebruik word as verdunningswater om die rou melasse vroeër in die proses op te los. Die skeidingsproses wat hier ontwikkel is, is gepatenteer. [Bekker, Stolz *et.al.* (2001)]

Ontslykte melasse is ontsuiker met 'n Gesimuleerde Bewegende Bed Ioon Eksklusie loodsaanleg. Die suiker herwinning was 93.9 massa % teen 'n 99.7 massa % suiwerheid. Die bedryfsparameters vir die aanleg was: voertempo 14l/h en warmer as 60 ℃; water vloei by 63 l/h en warmer as 65 ℃; ekstrak vloei teen 21 l/h; raffinaat vloei teen 56 l/h; lusvloei teen 78 l/h en 'n staptyd van 1326 sekondes. Die resulterende bed-volumes vir die verskillende skeiding zones is: Bed Volume Zone 1 = 0.694; Bed Volume Zone 2 = 0.591; Bed Volume Zone 3 = 0.661; Bed Volume Zone 4 = 0.383. Daar is ‘n wisselwerking tussen herwinning en produksuiwerheid asook vir die optimering van die water gebruik.

Die voorlopige omgewingsimpak en konsep massabalans is gedoen. Die voorgenome aanleg skakel ook goed in by die bestaande Komati Meul van TSB en hou geen noemenswaardige gevaar vir die omgewing in nie. Die aanleg benodig sekere dienste vanaf die bestaande meule. Die massabalans het in beginsel die water en stoomintegrasie ook ondersoek om die aanleg so ekonomies moonlik te bedryf. Dienste wat deur die proses benodig word kan voorsien word deur die dienste wat tans beskikbaar is by die meule. Stoom in die afseisoen moet egter deur ‘n aparte ketel gelewer word.

Verskeie tegnieke is gebruik om die finansieële lewensvatbaarheid van die projek te toets. 'n Omsetkoers ('IRR') van 42% op die kapitale belegging van R94,270,000.00 kan bereik word. Die winsgewendheid van die aanleg word bepaal deur die netto omsetkoers, wat bereken is as 4%/jaar. Die netto huidge waarde van die aanleg, verdiskonteer teen 30% is R41,782,000.00. Die aanleg se verdiskonteerde terugbetaaltyd is 5.207 jaar. Die finansiële lewensvatbaarheid van die projek voldoen aan TSB se vereistes.

Dit is dus nou moontlik en lewensvatbaar om melasse van riet-suiker fabrieke te ontsuiker.

Table of Contents

LIST OF FIGURES

LIST OF TABLES

Acknowledgements

The author would like to acknowledge the following persons and institutions for their effort in making this project a success.

- My wife Carmen, for her love, understanding and support during the research and during the progress of this thesis.
- Transvaal Sugar Ltd. for their commitment to technology development and for their permission to use details of the project in this thesis.
- Industrial Development Corporation of South Africa for the active interest in this project and the equipment provided. Without their financial support this project would not have been studied in as much detail.
- Prof. Hansie Knoetze for his patience and guidance during this project. This thesis would not have been possible without his probing questions and endless support.
- Baker Hughes for allowing TSB to use the E-Cat for an extended period of time.
- A special word of thanks to all the guys who got their hands dirty on the pilot plant with me: Coligny Naude, Andrew van der Heever, Richard Gomes, Bruce Harman, Brian de Beer, Matt van Heerden, Dewald Olwage, Martin van Gass and Kurt Merckel.
- Isabel Bekker for the never-ending analyses on the HPLC in the laboratory and Dr. Piet Bekker for his ideas, support and guidance through this project.
- Mr. Glynn Jones, for his sympathetic ear, patience and advice while proofreading this manuscript to correct the spelling and grammar and Germie Pienaar for having to decypher my handwriting and all the typing.
- Applexion in general, but special thanks goes to: Marc-Andre Theoleyre, Yvan Bathany, Pascal Galman, Stanislas Baudoin, Xavier Lancrenon, Daniel Herve and Tony Randall for the supply of the SMB unit, their valuable advice, always mailing me numerous articles from their library if I could not find it locally and their ongoing support of and belief in this project.
- My Lord and Saviour for all the blessings He bestowed on me and for the opportunity to study and learn about His wonderful creation.

Nomenclature

BV = Bed Volume
BV1 = Bed Volume of Separation for Zone 1
BV2 = Bed Volume of Separation for Zone 2
BV3 = Bed Volume of Separation for Zone 3
BV4 = Bed Volume of Separation for Zone 4

C = Concentration of adsorbed component remaining in solution
C = Corrosion allowance
ColourLoad_{BV} = Accumulated Colour trapped by the resin per bed volume
Cv = Critical value of separation
DS_{refr} = Dry substance (solids) content of the sample, refractometric, % (g/100g)
E = Joint Factor
F_x = Flow rate of component X
h_X = Enthalpy of liquid (of stream X)
H_X = Enthalpy of steam/vapour (of stream X)
InletBrix = Refractometer Brix of solution entering a decolourising column
InletColour = ICUMSA Colour of solution entering a decolourising column
K = Affinity Coefficient of the resin or equilibrium coefficient
= (Concentration in resin) / (Concentration in solution)
K = Freundlich Constant or the specific capacity
K_M = Michaelis constant
M = Manufacturing Tolerance
OD_F = Optical density (at 420 nm) of the filtered sample
OD_U = Optical density (at 420 nm) of the unfiltered sample
OutletBrix = Refractometer Brix of solution leaving a decolourising column
OutletColour = ICUMSA Colour of solution leaving a decolourising column
P = Pressure
q = Evaporation Rate
Q_E = Extract flow out of the system [Volume/h]
Q_F = Feed (molasses) flow into the system [Volume/h]
Q_L = Loop flow in the system [Volume/h]
Q_W = Water flow into the system [Volume/h]
R = Dry substance measured in the diluted solution, %(g/100g)
RV = Resin Volume of one column of the SMB [Volume]
S = Allowable Stress (= 145000 kPa for 304 S/S)
S = Weight of sample (g)
[S] = Substrate concentration in the Michaelis-Menten Equation
StepTime = Time to complete one step on the SMB [seconds]

t = Wall thickness of a vessel

V_{max} = Maximum velocity of the reaction for a specified enzyme concentration

W = Weight of added water (g)

Y = Coefficient that incorporates ductility of the material (=0.4)

$\frac{x}{m}$ = Amount of Adsorbed component per volume of resin.

$\frac{1}{n}$ = Freundlich Exponent or the measure of the energy of the sorption reaction

ΔBV = the amount of bed volumes between the inlet and the outlet samples.

ε = Void volume of the resin (approx. 0.38)

λ = Heat of evaporation

ℓ = Length of cuvette in cm (usually 1 cm)

μ = Viscosity

υ_l = Kinematic liquid viscosity

ρ = Density

Glossary

Brix	Total dissolved solids in a solution. (The analytical method is discussed in Chapter 3, and it is the Same as DS_{refr}.)
Clarifier	A device in which suspended solids are removed by settling to the bottom to yield a clear liquid overflow.
Desugarisation	A process that is well established in the beet sugar industry to recover the last sucrose (and sometimes glucose and fructose) from the molasses. Until now, there was no viable process to achieve the same in the cane sugar industry.
International Unit (IU):	One international unit (IU) of activity is defined as the amount of enzyme needed to hydrolyse 1 µmole of sucrose to invert sugar per minute at pH=4.5 and 55ºC.
Invertase	A commercial enzyme used to invert sucrose to glucose and fructose. Yeasts of the *Saccharomyces* species usually express this enzyme.
Inversion	The chemical reaction whereby sucrose is reduced to its monomer sugars namely glucose and fructose. The name in indicative of the effect of the reaction on plane polarised light.
Ion Exclusion	A phenomenon occurring on ion-exchange resin where molecules without a charge diffuse into and out of the resin beads, but charged molecules are repelled (or excluded from entering) thereby causing a separation between charged and un-charged molecules in a liquid flowing through the resin.
Molasses	The thick viscous brown to black liquid remaining after sucrose has been removed from sugarcane or sugar beet juice.
NTU	Nephelometric Turbidity Unit is a measure of turbidity commonly used in the water treatment industry.
NTU/Brix	An adaptation of the NTU turbidity method to relate the turbidity of a solution containing sugars to the dissolved solids (Brix) in the solution.
Simulated Moving Bed (SMB) Chromatography	A continuous method for the industrial separation of components using the principle of chromatography. In a SMB the resin is stationary in the columns and the separation is moved through the system by altering the liquid inlet and outlet positions around the system. It is contrary to a true moving bed where the resin bed moves relative to the stationary liquid inlet and outlet positions.

Chapter 1
Introduction

Sugars are the raw materials for numerous chemical and food products. Sugars, in crystalline or liquid form, can be utilised as they are in the bakery, confectionery, sweets and cold drink industries. Sugar can also be converted into other products. Sucrose, also known as table sugar, consists of two other sugar molecules: glucose and fructose. A mixture of glucose and fructose (usually derived from sucrose) is called invert sugar. Glucose and fructose are monosaccharide hexoses (six carbon) sugars. Invert sugar, in particular, forms the basis of a whole industry according to Paturau (1989). Direct chemical conversion or fermentation accounts for most of the products manufactured from invert sugars. Typical fermentation products are ethanol, acetic acid, lactic acid, citric acid, and lysine. Typical chemical conversion products are sorbitol & mannitol, obtained from the hydrogenation of glucose and fructose respectively. Vitamin C, or ascorbic acid, is a product obtained by both fermentation and chemical conversion.

Extensive studies have been done on the manufacture of a variety of compounds from sugars. A single search at the US Patents Office listed more than 34 000 patents where the words “sugar” and “alcohol” appears.

Upgrading the by-products of the traditional cane-sugar industry holds an economic advantage. Paturau (1989) tabulates the upgrading of by-products and base it on the saleable value of the end product obtained with fermentation or chemical conversion. A few products and their respective value in ZAR/metric ton[♣] that may be obtained from invert sugars in molasses, and discussed by Paturau (1989), are: Acetic acid (70%) - ZAR4000, Ethanol (absolute) - ZAR5000, Lactic acid (80%)- ZAR7000 and Lysine - ZAR22000.

Cane molasses is also known as "blackstrap" molasses, C-molasses or simply molasses. C-Molasses is the dark brown, thick, concentrated mother liquor remaining after all the economically viable recoverable sugar has been crystallised from the cane juice. About 10% of the sugar (sucrose) entering a sugar factory ends up in molasses

---

[♣] Personal Communication: June 2004 – Protea Industrial Chemicals as non-recoverable sugar. Molasses however also contains significant proportions of valuable glucose and fructose, or invert sugar, as well as dissolved inorganic constituents such as potassium, sodium, calcium, magnesium, phosphates and nitrates, which originate from the fertiliser applied in the cane fields and which are conveyed with the cane to the factory. C-Molasses also contains insoluble, suspended materials, mostly of an organic nature, which consist of plant pigments, gums, waxes and factory formed substances. C-Molasses compositions vary throughout the season and even from cane field to cane field. Soffiantini *et.al.* (1971) compared the molasses composition of four of the Natal factories. Walford (1996) reported on the composition of cane juice. MacGillvray and Matic (1970) also discussed the composition of South African molasses. The Sugar Milling Research Institute (SMRI) publishes a weekly report of the molasses composition of all the mills in the South African industry. An annual review of the milling season is published in the Proceedings of the South African Sugar Technologists Association. (Refer to Lionnet (2001) as a typical example.)

A typical composition of C-molasses is presented in Table 1.1 below.

Table 1.1: Composition of Cane Molasses (Results from the 1998/1999 campaign, determined by analysis in the TSB & SMRI laboratories)

Molasses Composition	
Component	**Mass%**
Dry Solids	80.45
Ash (on dry solids)	19.01
Total Sugar (on dry solids)	56.2
Sucrose (on dry solids)	36.9
Glucose (on dry solids)	8.1
Fructose (on dry solids)	11.2
Other Properties	
Suspended Solids on volume	10 %
Suspended Solids on Dry Basis	5.2 %
Ca^{++} in molasses	7050 ppm
SG of molasses	1.45

In September 2004, the C-molasses price was R210,00 per metric tonne. The local sugar price at the same time was R2800,00 per metric tonne. Molasses still contains on average about 45% sugars, of which 28 % (wet mass) typically is sucrose and the rest, a mixture of glucose and fructose. Total molasses sugars can therefore be priced at R467,00 per metric tonne - one of the cheapest available sources of sugar.

The Komati mill produces between 79 000 and 87 400 metric tonnes of molasses per season. This molasses is sold mainly to cattle feed producers or to the fermentation industry. A small amount is also exported. This amount of molasses has the potential to produce up to 35 000 metric tonnes of invert sugar per year. Assuming the market price for invert sugar is the same as the price of white sugar, it would mean an additional income of around R79 million rand per year.

Extracting and utilising the sugar in cane molasses has always proved to be a difficult task that could not be economically justified by the cane sugar industry. The result is that most of the molasses is either used for cattle feed or in the fermentation industry to manufacture yeast or alcohol. On the other hand, the beet-sugar industry is fortunate to have a "clean" molasses that can be treated to recover this last amount of sugar. Being able to recover these sugars from cane molasses could provide a rather cheap source of carbohydrates. Utilising these sugars to produce food and chemical compounds are a logical value-adding step.

In the beet sugar industry chromatography is extensively used to desugarise beet molasses. This separation is based on the principle of ion exclusion where the molecules having an ionic charge (typically salts) are repelled by the charge on the separating resin and are effectively “washed” out of the column in the eluent. The functionality of the resin has an influence on the separation. To protect the resin from blockage by suspended solids the specification for molasses entering an ion exclusion unit is rather strict. The turbidity (as an indication of the amount of suspended solids in the liquid) must be below 10 NTU/Brix, the ICUMSA turbidity should be below 3000, the molasses must have less than 0,1 per cent sludge and the Calcium content must be below 2000 parts per million to prevent ion exchange with the resin.

The main objective of the study was to develop and to define a process to produce a liquid product, starting from Komati Mill's C-molasses, from which invert sugar could be separated, decolourised and concentrated. The resulting sugar stream must have a colour and composition acceptable to the liquid sugar market. To achieve this goal the following objectives were set:

1. Identify and determine the variables required to remove the suspended solids from a C-molasses solution.

2. Determine the variables to achieve efficient separation of salts and sugars using chromatography.
3. Achieve adequate decolourisation & deashing
4. Ensure the process can be commercialised by:
    a) Evaluating scale-up characteristics and identify risks for reliable scale-up.
    b) Determining a mass & energy balance for a commercial plant.
    c) Investigating the environmental implications of the plant
    d) Proving the economic feasibility of the project measured against the financial criteria set by TSB.

To develop and qualify the process, a pilot plant was designed and built according to descriptions of desugarisation processes obtained from literature.

Trials were initially done on B-molasses, an intermediate product of the mill process. The motivation for this was that B-molasses is much cleaner than C-molasses and it contains more sugar. The use of a cleaner feedstock with less concentrated impurities and higher sugars content should make the development process easier and quicker. This was found not to be the case.

C-molasses is freely available and at a lower cost than B-molasses. The clarification and separation of C-molasses turned out to be more reliable than that of B-molasses. Significantly more development work needs to be done to qualify the B-molasses option.

The sugar stream from C-molasses eluting from the chromatographic columns had a dissolved solids content of about 30° Brix, which consisted of more than 98% invert sugar. After de-ashing and decolourisation with ion exchange resins, and evaporation to 80° Brix a suitable product was produced.

The product obtained from the process was a mixture of glucose and fructose. The components can be further separated in another chromatography step if pure fractions are required.

The logical development of this thesis is presented in Table 1.2.

Table 1.2: A layout of the logical development of the thesis.

A review of the present “state of the art” as reported in the literature.
Laboratory Methods and Analytical procedures used throughout the study.
Pilot Plant Design Criteria & Design.
The evolution, results and discussion of the method devised to clarify molasses.
The recovery of product from the effluent sludge to increase the plant efficiency and overall invert sugar yield.
The methods investigated to achieve complete inversion of the sucrose to invert sugars.
Chromatographic Separation of the “clean” invert syrup on a simulated moving bed system.
Methods investigated to remove the ash and colour from the invert sugar stream leaving the chromatography plant.
Finally the evaluation of the environmental impact, conceptual design and financial viability of a proposed commercial plant producing invert sugar from molasses.

In September 2004 the local market price for "liquid sugar" type products was around R4000/mt. The separated glucose and fructose could also be crystallised and sold in dry powder form.

Chapter 2

Literature study, theory, concepts and principles

2.1 The origin of molasses (A little sugar technology)

The process description that follows is mostly applicable to the Komati Mill. Very detailed and informative texts are available on the topic of sugar technology and the reader is referred to the textbook by Chen and Chou (1993). A detailed and condensed description is also found in the short course offered by the Sugar Milling Research Institute (SMRI) (Pillay).

Sugar cane enters the mill from the fields and is sliced. The sliced cane then goes through a hammer mill where the cane is shredded.

The juice from the shredded cane is extracted in a counter-flow diffuser with hot water. The moist fibre leaving the diffuser is dewatered in one or more dewatering mills. The pressed juice is pumped back to the diffuser. The dewatered fibre, or bagasse, exiting the mills is used as boiler fuel to generate steam and electricity.

The juice leaving the diffuser is heated and flashed to release entrapped air. Lime is added to the juice to neutralise the plant acids and to assist in the clarification stage. An anionic flocculant is then added, before the mixture enters a clarifier. In the clarifier sand, fibre and other suspended solids settle out. The overflow is a clear liquid that is heated and evaporated in a multiple effect evaporator to produce syrup. The syrup is concentrated to supersaturation, and seeded with finely ground sugar crystals to initiate the crystallisation process. The resulting massecuite contains about 55% crystals. A batch operated basket centrifuge is used to separate the crystals from the mother liquor.

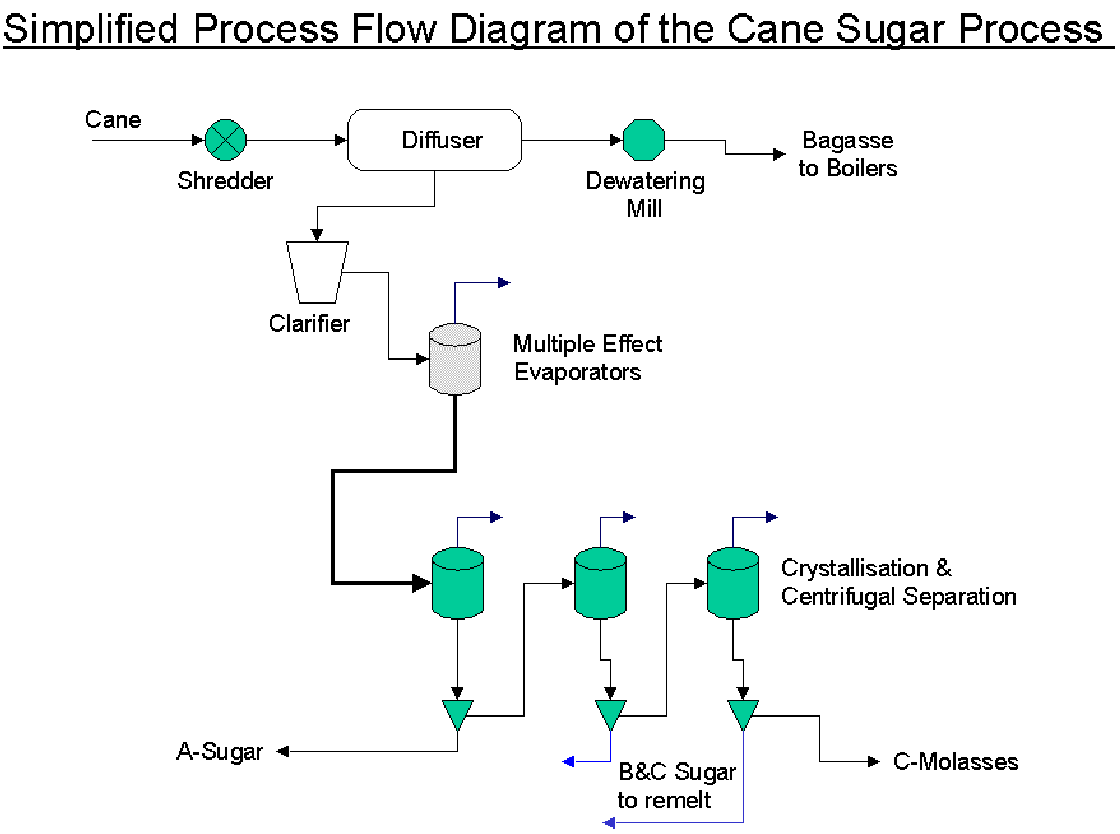


Figure 2.1: A Simplified PFD of the cane sugar manufacturing process.

The sugar crystal at this stage is called A-sugar. It is the common brown household sugar familiar to all. The mother liquor (or A molasses) still has a significant amount of sugar in solution. It is pumped back to the crystallisation pans where the process repeats itself twice producing a B-sugar and B-molasses and C-sugar and C-molasses respectively. B- and C-sugars are dark and rich in molasses, which is entrapped in the lamella during crystallisation. These sugars are melted with clarified juice and returned to the syrup to be recrystallised.

Continuous centrifuges are used to separate both the B-sugar from the B-molasses and the C-sugar from the C-molasses. The crystal breakage that occurs when the crystal leaves the basket and hits the sidewall of the centrifuge aids in dissolving the crystals.

Unprecipitated lime, colour bodies and a variety of products formed during the boiling process end up in the C-molasses. It is not feasible to recover the sucrose remaining in the C-molasses with another crystallisation step. The sucrose content is low, the total molasses volume is small, and the concentrated salts, invert sugar and impurities make this molasses very viscous. A typical problem occurs in the sugar industry when low quality cane with a high invert sugar concentration is milled causing the formation of elongated crystals that blinds the centrifuge screens. The C-molasses leaves the mill as a by-product and is typically sold to the animal feed industry or to the fermentation industry.

Very little work has been done worldwide on the desugarisation of cane molasses. Desugarisation of beet molasses is however, well known.

2.2 Literature on the desugarisation of cane molasses

Pynnonen (1996) reviewed the history and status of molasses desugarization in the beet-sugar industry. He also discussed the important technical and economic differences between cane and beet molasses with regard to recovery via ion exclusion chromatography and highlighted the hurdles to cane molasses sugar recovery.

Table 2.1: The differences between cane and beet molasses. [Pynnonen (1996)]

	Beet Molasses	**Cane Molasses**	**TSB Cane Molasses**
% Suspended solids	Trace	3%	
% Solids in liquid phase	80	80	80
% Sucrose on Dry Solids	60	44	37
% Invert on Dry Solids	<1%	15	20
Molasses hardness (as ppm Calcium)	3500	18000	8000

For purposes of comparison, average values for the composition of TSB cane molasses are also shown.

Pynnonen (1996) identified three technical challenges with regard to the desugarization of cane molasses compared to beet molasses. The first is the presence of both sucrose and invert sugar, their separation and the recovery of sucrose via the conventional sugar manufacturing process.

In the envisaged TSB process all the sucrose is to be inverted to glucose and fructose and recovered as invert sugar only. This eliminates one hurdle and has an important advantage. During inversion (hydrolysis) of sucrose one molecule of water is added to every molecule of sucrose. The result is an increase in mass of about 5%. Economically this is a quite significant gain on the large quantities of molasses involved.

Pynnonen's second hurdle is the higher viscosity and higher suspended solids content of cane molasses: 1-6% vs. the 0.5% of beet molasses. Without efficient solids removal, the chromatographic separation process breaks down. The filtration of cane molasses presents many more difficulties because of waxes, colloids, gums and polysaccharides that are not present at such high levels in beet molasses. He speculated that a two-stage removal process might be required, the first being a rough stage to take out the larger, more easily removable suspended solids by either a settling tank or a high g-force centrifuge. The second stage would be similar to the fine beet molasses desugarisation filtration. This fine filtration process could use membranes ("cross flow" filtration) or pressure filters.

Pynnonen's last hurdle is effective and efficient softening. Chromatographic separation of sugars from salts does not occur if the ion-exchange resin used as chromatographic packing is in the calcium or magnesium forms. The resin must be in the same ionic form as the salts that are present in the molasses (potassium or sodium). An efficient softening system is required because the chromatographic separating resin prefers the high valence metal ions (typical ions like calcium, magnesium and iron). These ions will accumulate on the resin if the incoming molasses has not been adequately softened. Various levels of hardness have been stated. Pynnonen recommended a maximum hardness of 100-ppm divalent ions, expressed as weight equivalent of calcium ion relative to the dry solids in molasses.

Schoenrock *et.al.* (1976) described a system used in a beet sugar factory where ion exchange was used. They employed a novel regeneration scheme to save on operating expense by utilising effluent flows and chemical recovery. In the envisaged TSB process, the raffinate, which is rich in potassium, will be used to regenerate the softener column.

Paananen, (1996) from Finnsugar, stated that they do not employ thin juice softening in their beet sugar factories and operate their beet sugar molasses desugarization plants typically at levels of 300-500ppm calcium with good results. Henscheid, *et.al.* (1991) described the experiences with the softening system installed at the Twin Falls plant of Amalgamated Sugar. In this system, the regeneration of the softener is achieved by using chromatographic raffinate. The process yields a product with less than 0.006g CaO/100g dry solids.

Paananen (1996) had also desugarised 3000 tonnes of cane molasses in 1980 at Finnsugar's Kantvik plant. They used a phosphate clarification process in which flocculant was also used. They removed the phosphate precipitate by centrifugal separation and thereby reduced the calcium level by 30-40%.

Schneider (1978) extensively studied the desugarization of cane molasses in Germany in the 1970's. He concluded that divalent ions should be reduced to about 0.2% CaO on dry substance. He worked with 28-35 Brix molasses solutions and used phosphoric acid at levels up to 1000ppm on dry solids to reduce the calcium and magnesium content to levels low enough for chromatographic separation. Centrifugation and filtration was employed to obtain the required clarity. Schneider also considered it necessary to treat molasses with phosphate only when the total divalent ionic content exceeded 47% of the total ionic content of the molasses.

Saska and Lancrenon (1994) suggested that the calcium and magnesium level of clarified molasses should be no more than 2000 ppm/Brix and the turbidity not more than 10NTU/Brix. They succeeded in attaining the required turbidity by addition of a flocculant to a 40-Brix molasses solution followed by filtration on a filter press with filter-aid. What is interesting in their approach was the three-product outlet from the separator allowing sucrose recovery as an alternative to the normal invert/ash separation. They also compiled breakthrough curves for the softening step. In an earlier work, Saska and co-workers (1992) achieved the required clarity of the molasses by phosphatation (pH=3,8) followed by neutralisation with caustic (pH=7,4) and finally filtration.

Bernhardt (2000) and co-workers at the SMRI in Durban published a report on the production of high-test molasses. They reported work done on treating cane molasses to achieve a suitable feed material for chromatography. They preferred a centrifugal separation using sulphuric acid to treat the molasses. The same authors published a paper in 1999 concerning a process to produce invert sugar from cane molasses. This paper was a precursor to the final report mentioned above. They reported a product after ion exclusion of three Brix, with 62% (of dry solids) as invert and a pH of 2.4.

Hongisto (1977, Part 2) in his discussion of the Finnsugar desugarisation process mentioned that a calcium content lower than 0.2% would allow the resin to operate at between 500 – 2000 cycles before regeneration in the chromatography unit. Riffer (1976) filed a patent describing the process developed by California and Hawaiian Sugar Company to desugarise Hawaiian molasses. He suggested a pH of around 4,0 was needed to give the best performance. He reported that the extract from the two batch columns was collected in fractions and decolourised using a resin in the base (OH^-) form together with granular activated carbon. A 90% colour removal was achieved. They were able to completely remove the cations on Amberlite IR-252 resin in the hydrogen form.

Sugar y Azucar (1994) reported that more than 90% of the sugar lost in beet molasses could be recovered by ion exclusion. They offered a comparison between cane and beet and discussed a number of processes in use in the United States.

Based on these descriptions a conceptual flow diagram was compiled and a pilot plant was constructed.

Chapter 3

Materials and Methods of Analysis

3.1 Introduction

To ensure uniformity in the analysis of sugar products the international sugar community has created the International Commission for the Uniformity of Measurement in Sugar Analysis (ICUMSA). ICUMSA has established, and is constantly updating, a series of laboratory methods that is used worldwide by the industry. In this study all the typical sugar analyses are done using the official ICUMSA methods. This chapter serves as a background to these analytical methods, as well as some other methods not covered by ICUMSA.

3.2 Official Refractometric method for Dry Substance (Solids Content) By Indirect Methods (ICUMSA 1970 & 1974)

The refractive index method is used for the determination of the amount of dissolved substances (refractometric solids) in solutions containing mainly sucrose. For sugar products containing crystals it is necessary to dilute the sample. The refractometric values of solutions of sugar products can be used as a more or less accurate measure of their dry substance (solids) content since the non-sugars present in these products have a similar influence as that of sucrose on the refractive index. Measurements on the solutions are generally carried out with sugar refractometers, which are graduated in % sucrose (g/100g) according to the ICUMSA tables of refractive indices at temperatures of 20.0°C or (for use under tropical conditions) at 27.0℃.

3.2.1. APPARATUS

Sugar refractometer or Zeiss refractometer

Thermostat controlled at 20.0±0.1°C or, for tropical conditions, at 27,0±0.1°C.

Plastic rod, e.g. perspex or polypropylene

3.2.2 PROCEDURE

Samples containing undissolved sugar crystals are diluted with a known amount of water (generally 1:1 by weight). Samples containing suspended solids are filtered or centrifuged. The temperature of the sample is adjusted approximately to the temperature of the instrument. Temperature controlled water (20.0 or 27.0°C) is circulated through the refractometer prism jacket with the prisms closed for a sufficient length of time to ensure that equilibrium has been reached. The refractometer prism is opened, and a drop of sample is applied to the fixed prism face by means of the plastic rod. The sample is quickly extended as a line along the prism without actually touching the prism face with the rod and taking care to avoid the formation of air bubbles. Sufficient time must be allowed for the samples to reach the temperature of the prism. The refractometer reading is recorded.

If the reading cannot be made at either of the temperatures specified above, the circulating water is adjusted to some other suitable fixed temperature. After the reading is made it is converted to the value at 20.0 or 27.0°C using the ICUMSA International Temperature Correction Table (1974). The prisms are cleaned with water at room temperature and soft tissue; hot water should not be used for this purpose. For especially high accuracy, the use of a dipping refractometer is recommended. With these instruments several exchangeable prisms cover a wide range of refractive index.

Standardisation is carried out at least daily by adjusting the instruments to a reading of 1.33299 for the refractive index of distilled water at 20.0°C or 1.33228 at 27.0°C corresponding to zero solids content. If the instrument is not equipped with a sucrose scale, the readings are converted to sucrose content with the International Scale of Refractive Indices. The dry substances of diluted samples are calculated as: (ICUMSA, 1970 & 1974)

$$DS_{\text{refs}} = R\left[\frac{S+W}{S}\right]$$

Where DS_{refr} = Dry substance (solids) content of original sample, refractometric, % (g/100g),
R = Dry substance measured in the diluted solution, %(g/100g),
S = Weight of sample, g, and
W = Weight of added water, g

3.3 **Official method for Conductivity ash** (ICUMSA, 1970 & 1974)

The specific conductivity of a sugar solution of known concentration is determined. It is assumed that the conductivity has its own significance and the equivalent ash is calculated by the application of a constant factor. Two concentrations may be used, i.e. 28g/100g for white sugar and other products of very low conductivity and 5g/100g for all other products. The factors used for transforming measured conductivity into ash are purely conventional and are applicable only to sugar solutions.

3.3.2. APPARATUS

Sugar ash bridge or null balance bridge
Volumetric flasks, class A, capacity 100, 500 and 1000cm^3
Pipettes, class A, 10cm^3

3.3.3. REAGENTS

- Purified water: for preparation of all solutions (sugar and potassium chloride) distilled or de-ionised water with a conductively of less than 2µS/cm must be used.
- Potassium chloride, 0.01N: potassium chloride (745.5mg), preferably after heating to 500°C to dehydrate it, is weighed out, dissolved in water in a 1000cm3 volumetric flask and made up to the mark
- Potassium chloride, 0.0025N: this solution is used for the measurement of high conductivities: 250cm3 of potassium chloride solution, 0.01N is diluted and made up to the mark in a

1000cm^3 volumetric flask. It has a conductivity of 328µS/cm at 20°C.

- Potassium chloride, 0.0002N: this solution is used for the measurement of low conductivities: 10cm^3 of potassium chloride solution, 0.01N is diluted and made up to the mark in a 500cm3 volumetric flask. It has a conductivity of 26.6 ± 0.3µS/cm at 20°C after deduction of the specific conductivity of the water used.

3.3.4 PROCEDURE

A solution is prepared by dissolving 5g of the sample in water in a 100cm^3 volumetric flask at 20°C. In the event of the conductivity exceeding 500 µS/cm, or the solids content of the solution being less than 5g, white sugar of low ash content must be added in such a way as to maintain the total solids concentration at 5g/100cm^3.

After thorough mixing, the solution is transferred into the measuring cell and the conductivity measured at 20 ± 0.2°C. The measurement is checked by means of the appropriate reference solution.

3.3.5 SUGAR ASH BRIDGE

This comprises a balanced bridge circuit with null point indicator and shall comply with the following specification:

- Frequency: 50 to 2000 Hz
- Range: 0.001 to 0.1% ash for white sugar and 0.01 to 0.9% ash for raw sugar
- Accuracy of built-in standards: ± 1% or better
- Accuracy of measurement: ± 3% or better but, for low ash (conductivity) values, not less than 0.001% (0.5±µS/cm)
- Indication: visual
- Scale units: ohms or Siemens (=S) or ash units
- Temperature of solution: the standard temperature of measurement shall be 20°C.

- Temperature compensation: the bridge shall be provided with a temperature compensating mechanism. Temperature compensation shall be in accordance with the current ICUMSA recommendation given for conductivity ash measurements. They are:
  - At a concentration of $50 mg/cm^3$, 2.3%/°C with a maximum of ± 5°C.
  - At a concentration of 28g/100g, 2.6%/°C with a maximum of ± 5°C.

3.3.6 NULL BALANCE BRIDGE

This comprises a null valance with low voltage valve or semiconductor amplifier or direct-reading conductivity bridge, and shall comply with the following specification:

- Frequency: 50 to 2000 Hz
- Range: 0 to 500µS/cm
- Electrode voltage: 0.2 to 10V
- Accuracy of built-in standards: ± 1% or better
- Accuracy of measurement: ± 3% or better but for low ash (conductivity) values, not less than 0.5µS/cm
- Indication: visual
- Scale unit: ohms or siemens (=S)
- Electrodes: with fixed distance
- Cell construction: of glass or synthetic material
- Temperature measurement: means for measuring the temperature of the solution to be provided
- Cell constant: within the range 0.2 to 3 cm^{-1}

3.4 Jar Test for Optimum Coagulant Dosage

In order to determine if a flocculant is capable of coagulating and settling sludge, a rapid method has been developed and the adaptation of the method is reported here.

3.4.1 APPARATUS

6 Glass containers of uniform size and shape, each of 1L capacity.
Six pointed stirring device for simultaneous mixing (known as a flocculator)

3.4.2 METHOD

Determine the turbidity, pH and alkalinity of the molasses solution.
Fill each of the jars to the 1L mark with the solution.
One jar is to be used as a control.
Dose the five remaining jars with different amounts of the coagulant(s).
Mix the water by rapid and vigorous stirring for 1 minute, ensuring complete dispersion of the chemicals.
Continue mixing, slowly and gently for 15 to 20 minutes, to keep all the solids in suspension and aid the formation of flocks.
. Allow the mixture to settle for 30 minutes, or until clarification has occurred.
Remove portions of the clarified molasses and test for the residual turbidity.

3.5 Laboratory Methods for the Measurement of Turbidity

The conventional method used to measure turbidity in the sugar industry is the ICUMSA method. This method was developed by the industry and is managed by the International Commission for the Uniformity of Measurement in Sugar Analysis. The method is based on the difference in light absorption (optical density) between an unfiltered sample and a sample filtered through a 0.45 μm membrane paper. The method requires the samples to be at $pH=7 \pm 0.02$ to avoid colour effects brought about by pH which may influence the measurement. The measurement is done using light at 420 nm. The turbidity can be calculated by substituting the values in the following equation: (ICUMSA Methods)[℘]

[℘] Reported in the Applexion Laboratory Manual.

$$Turbidity = \frac{1000(OD_U - OD_F)\ell}{\left(\left[\frac{(200+Brix)Brix}{54000}\right]+1\right)\left(\frac{Brix}{100}\right)}$$

Where: OD_U = Optical density of the unfiltered sample,

OD_F = Optical density of the filtered sample,

ℓ = Length of cuvette in cm (usually 1 cm)

Brix = Brix of the sample being measured.

The common method to express turbidity in industry is the NTU - Nephelometry Turbidity Units. This method measures reflected light scattered by suspended particles at an angle of 90° to the incident light source. It is also possible to measure the absorption of light through a cuvette and to relate the finding to a turbidity value. Commercial measuring instruments are available that directly reports turbidity in NTU. Formazin is used as a reference in these methods. Further official methods can be obtained from Clarke (1995).

Godshall (1997) reported that there is no consensus on the way to measure turbidity in the sugar industry. She states that Nephelometry has been recommended as a way to obtain a true measure of turbidity in sugar solutions.

Chapter 4

Pilot Plant Design Criteria, Design Calculations and Final Construction

4.1 Introduction

The life cycle of a development project usually involves the following stages: concept identification and gathering of background information, laboratory or small scale testing, pilot scale testing, commercialisation and decommissioning. At the end of each stage the results are evaluated and used to test if the project fulfils the goals defined by the concept. A pilot plant can be defined in one of two ways: it is a plant used to confirm laboratory observations and generate design data for the commercial plant or it is a plant used to demonstrate a concept or process. The two definitions are closely related to each other. Typically a new successful concept that has progressed to the pilot plant stage will achieve both goals. It will demonstrate the feasibility and practicality of the concept while providing the necessary information to enable the design of the commercial plant. The latter type of pilot plant is typically used as a training facility for operators on the commercial plant. Another application is to test the influence of process modifications before implementation on the commercial plant. A pilot plant can begin as a test unit and continue to operate after the construction of the commercial plant.

For the clarification of molasses to produce invert sugar after chromatographic separation, a pilot plant had to be constructed on such a scale that the limitations of bench scale evaluation could be overcome. The pilot plant had to be able to use commercial equipment to achieve the required product quality.

The original process concept was based on the methods investigated and recommended by Applexion[♣]. Consideration was also given to the work reported in literature. From these sources, the initial process flow diagram was constructed and it formed the basis for the design of the pilot plant.

4.2 Process Description

The sequence of process steps used to produce the invert sugar feedstock is presented in Figure 4.1.

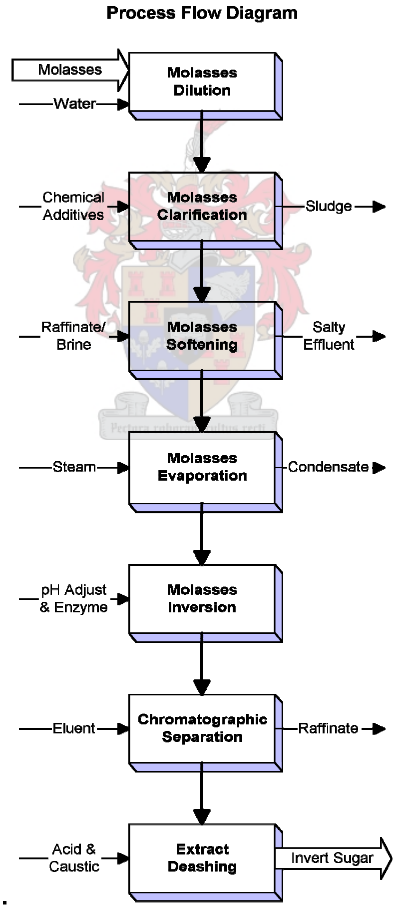


Figure 4.1: Simplified flow diagram for the production of Liquid Invert Sugar from sugar cane molasses

---

[♣] Applexion is a process engineering company based in France.

Molasses is collected from either the factory molasses pumps or from the molasses tank and carted to the pilot plant. The molasses is then pumped into the dilution tank. The molasses is diluted to the appropriate concentration by the addition of water, usually exhaust steam condensate from the factory. Chemicals, H_3PO_4 and NaOH, are added as required. The molasses is then allowed to react and pumped to a centrifuge. The centrifuge has an internal pump that would pump the mother liquor into the filter feed tank.

Filtration was initially done using a Gasquet Pressure Leaf filter of 5 m^2 installed in the skid framework. A press filter later replaced it. Filtration was done using Dicalite filter-aid to precoat the filter. A finer grade was used as body feed in the molasses. Filtered molasses was tested for clarity and, when suitable, it was softened in an ion exchange column filled with strong cationic resin, Rohm & Haas Amberlite 252, in the sodium (Na^+) form. The softener was regenerated with raffinate and after regeneration the column reached equilibrium in the potassium (K^+) and sodium (Na^+) form. The reaction on a cation exchange resin can be presented as follows:

$$2\text{Resin}^- \text{-}K^+ + Ca^{2+} \rightarrow \text{Resin}_2{}^- \text{-}Ca^{2+} + 2K^+$$

The soft molasses was then evaporated to 65 Brix and inverted. Inversion is the “breakdown” conversion of sucrose (a disaccharide) into glucose and fructose (monosaccharides). Inversion can be done either by enzyme treatment or by acid catalysis. In the initial bench scale experiments done by Applexion, the acid catalysis method was not recommended due to the high level of acid required to allow full inversion due to the high buffer-effect of the molasses. Syrup pH was adjusted with hydrochloric acid to the recommended level, if required. Inversion was allowed to take place at 65℃ in either one of the inversion reactors. The inverted syrup was tested for clarity and if suitable was pumped to the chromatography feed tank.

The chromatographic separation plant consisted of eight columns, each holding a resin volume of 70 litres. The resin used was supplied by Applexion, but later augmented with Rohm & Haas Amberlite 1320K. The molasses was eluted with water and two fractions leave the plant: extract and raffinate. The extract is the sugar fraction and the raffinate contained the salt fraction. Raffinate was collected and evaporated to 35 Brix and used to regenerate the softener column. The extract was collected and de-ashed through a train of cation and anion columns. There were two cation columns and two anion columns. The first cation and anion column performed the deashing duty, while the second series performed the work of a polisher. Amberlite 252 from Rohm & Haas was used in both cation columns, while the anion columns were filled with Rohm & Haas Amberlite IRA92. The extract was then decolourised and evaporated to yield a liquid sugar product. The properties of the resins used in this investigation are included in Appendix A.

4.3 Design Criteria

The pilot plant was designed based on the requirements of a possible downstream glycol production plant. The glycol plant required 21 kg/h of feed material at about 30 Brix, when operating at full capacity. This translates to 151-kg dry material per day. The molasses clarification plant was therefore specified to produce the daily requirement of invert sugar syrup required for the glycol plant. It is proposed to eventually operate the molasses desugarisation plant and the glycol plant in series.

The chromatographic plant is schematically represented in Figure 4.2. It was operated at a molasses flow rate of 14 l/h. Water, to act as eluent, was fed at 63 l/h. This produced an extract stream of 21 l/h at about 30 Brix and a raffinate stream of 56 l/h at about 9 Brix.

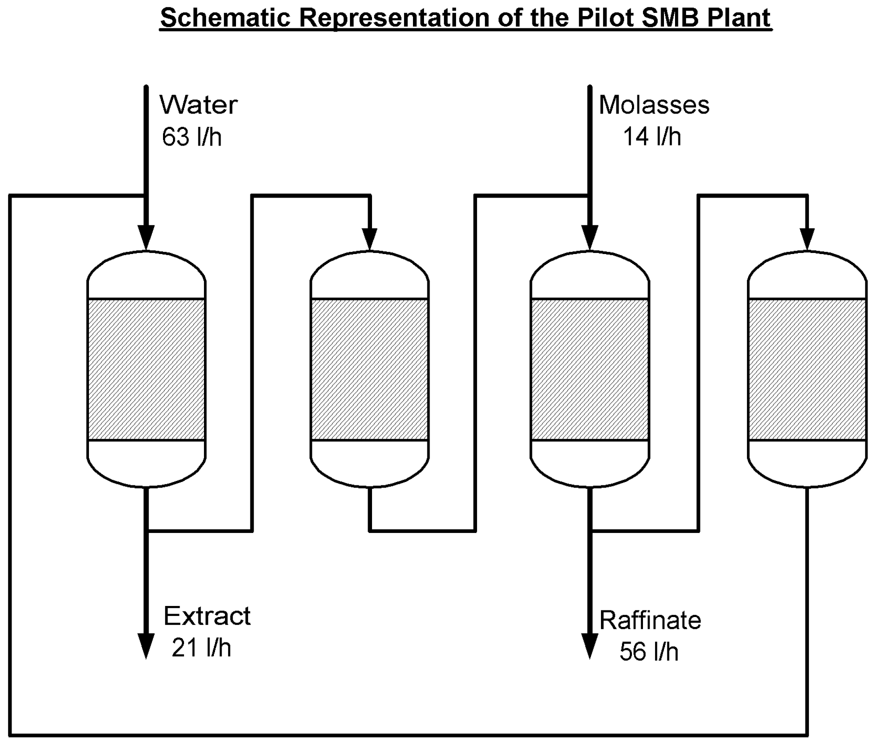


Figure 4.2: A Schematic diagram of the SMB system used in the study

The chromatography plant delivered 151 kg dry material per day in the extract and 74 kg dry material per day in the raffinate from 225 kg per day in the feed molasses.

The pilot plant also had to conform to a few other requirements.

1. The pilot plant was to be constructed as economically as possible.
2. The pilot plant had to be flexible in design and operation i.e. modular in design so that it could easily be modified to accommodate changes in operation and in

the process. It also had to be able to connect to third party test rigs in order to allow proofing of the process on commercially available equipment.

3. The pilot plant had to comply with the relevant safety regulations. All pressure vessels had to be designed and constructed according to the relevant standards, inspected and certified.
4. The pilot plant equipment had to operate on 220/380VAC to match the electricity requirement of the chromatography plant, rather than at 525V, which is the norm for the sugar factory.

4.4 Physical Properties of Components

Molasses has the following composition:

Table 4.1: Composition of cane molasses from the Komati Mill (Results from the 1998/1999 campaign, determined by analysis in the TSB & SMRI laboratories)

Molasses Composition	
Component	**Mass %**
Dry Solids	80.45
Ash (on dry solids)	19.01
Total Sugar	46.2
Sucrose (on dry solids)	36.9
Glucose	8.1
Fructose	11.2
Other Properties	
Suspended Solids on volume	10 %
Suspended Solids on Dry Basis	5 %
Ca^{++} in molasses	7050 ppm
SG of molasses	1.45

The following relationships and values are used for molasses:

Densities of sugar solutions are calculated with the formula[♠]

$$\rho = \left[\frac{(200 + Brix) \cdot Brix}{54000} \right] + 1 . \qquad (1)$$

This gives good results if compared to literature.

Viscosity of various sugar factory products are referenced in literature and represented in the graph displayed in Figure 4.3.

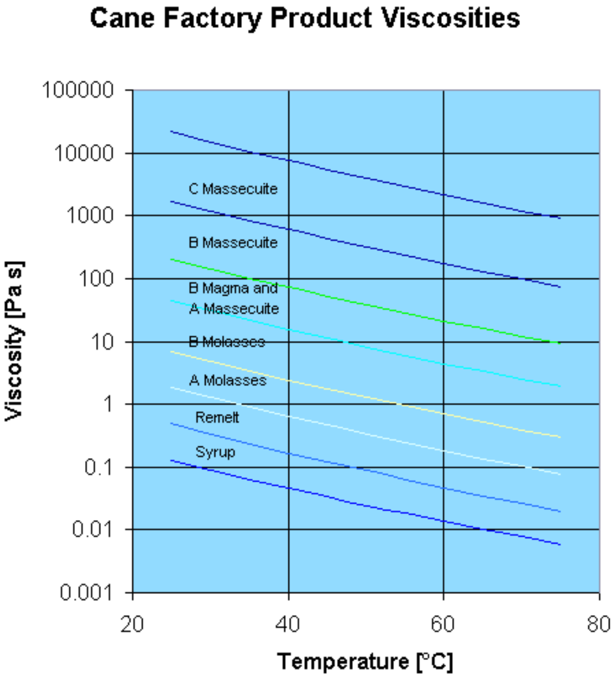


Figure 4.3: Viscosity of selected cane factory products

(Reported by Rouillard & Love and obtained from http://www.sugartech.co.za/viscosity.html)

Table 4.2 Viscosity of molasses (Agrimol Feed Molasses Datasheet)

Temp	**0℃**	**10℃**	**20℃**	**30℃**
Viscosity (cP)	22 000	5000-15000	1900-4900	500-2700

The viscosity of C Molasses is between that of B molasses and A Massecuite. For the design it was assumed that C Molasses contains 30% sucrose. The viscosity (in cP) of a pure solution containing 30% sucrose is listed in Table 4.2. Viscosities for invert

---

♠ Personal correspondence: Y Bathany, Applexion & Applexion NAP Process Book (February 1995) sugar solutions are given in the graph below: ("Corrected RDS" refers to Refractive Dry Substance with a correction for the difference between sucrose and invert readings.)

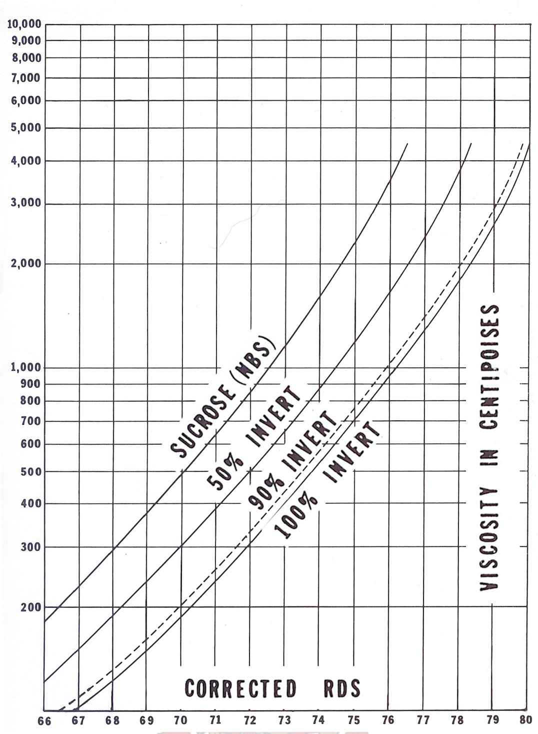


Figure 4.4: The effect of concentration on viscosity at 20℃ for varying proportions of invert as reported by California and Hawaiian Sugar Co. (1970) in Handbook of Sugars.

4.5 Design Calculations

The basic and some detailed calculations, used to determine the size of the pilot plant, are included in Appendix B: Design Calculations. The Appendix also lists the utilities required by the plant.

In Figure 4.5, below, the mass balance that was calculated for the plant is graphically represented. This figure shows the flows on a “per hour” basis spread over a 24-hour day.

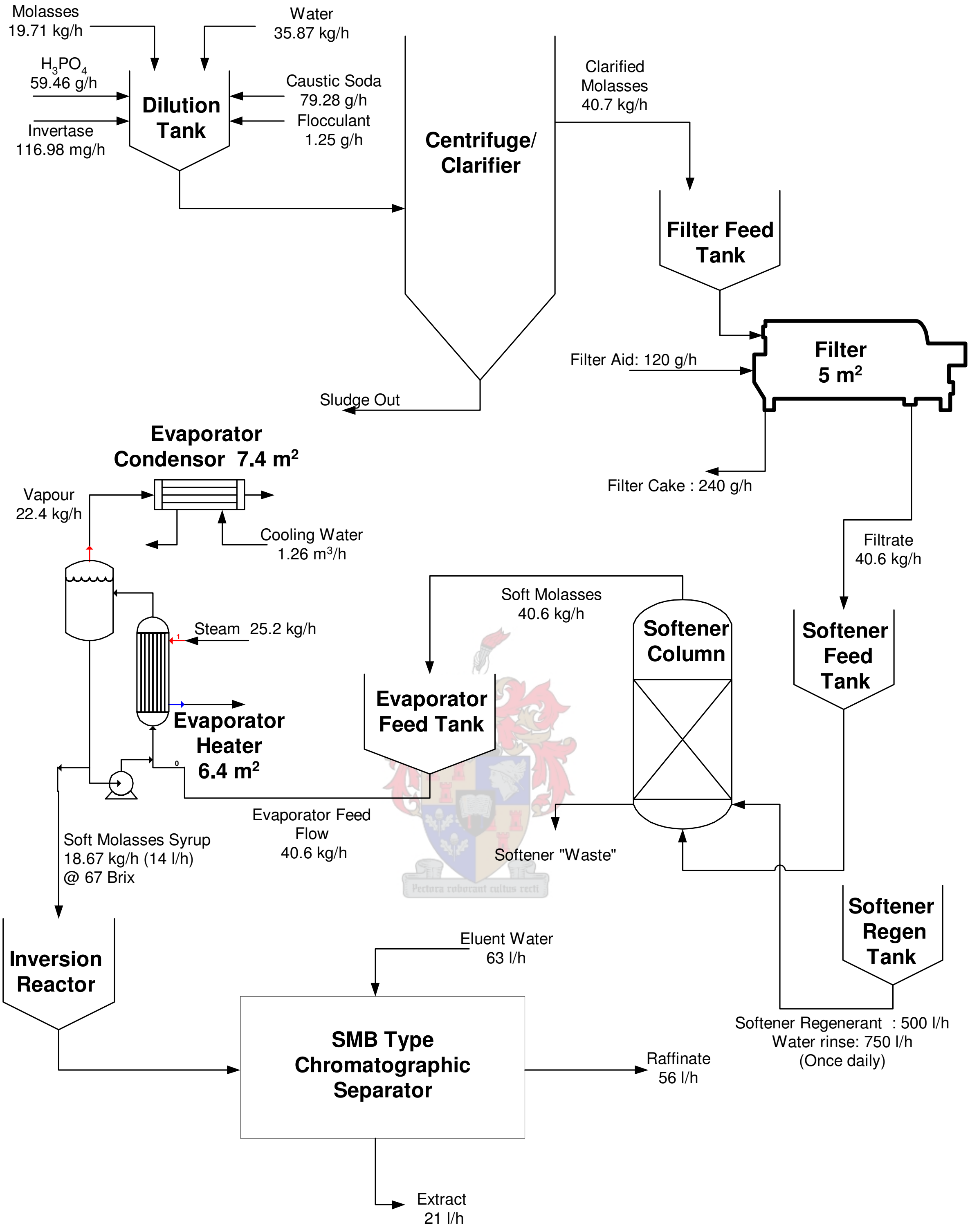


Figure 4.5: The calculated mass balance for the proposed pilot plant.

The detailed structural, steelwork and mechanical designs were done by a mechanical engineer.

4.6 Construction Materials

Traditionally sugar cane juice is pumped in iron pipes. The various mills allow for corrosion by replacing certain pipe sections during the off-season every year. Corrosion and the subsequent leaching of metallic ions into the product stream are unacceptable for the invert sugar process. This coupled with the need to produce a product with no microbiological contamination necessitated the need to use an inert construction material. The cost of the material was also considered in the selection of the material. Stainless steels are commonly used in the fruit juice and dairy industries, and in numerous other applications in the food industry. Typical grades of stainless steel in use are types 304/304L or 316/316L.

The material specification for the pilot plant process vessels and piping was for Stainless Steel Type 316L. The addition of Molybdenum to Type 304 improves the corrosion resistance in reducing conditions, in particular to solutions containing chlorides. Type 316L is also 1.3 times more resistant to corrosion than type 304. The 316L is a low carbon version of type 316 (<0.03 %C) used to prevent carbide precipitation during welding. Type 316 is more expensive than 304 when buying large quantities. On a commercial unit a trade off has to be made between the use of 304L or 316L. In the case of the pilot plant, all the valves, fittings piping etc. were available in 316L at almost the same cost as those made from 304.

Mild steel was used for the structure. Galvanised pipe was specified for the supply of instrument air, cooling water and domestic water and to handle the effluent return to the factory. Schedule 40, mild steel steam pipe, was specified for the steam and condensate supplies and for the pilot plant heating circuit. Schedule 40 pipe is the standard pipe used in the sugar industry for low-pressure steam and hot water pipelines.

4.7 Ancillary Equipment

The pilot plant has ancillary equipment installed. A Mono pump, with a capacity of 500 l/h, is used to pump the molasses from drums into the plant for treatment. The same pump is also used to pump sludge from the centrifuge to the effluent collection tank. A disk bowl centrifuge was installed on the plant for the separation of sludge from molasses. To ensure a constant feed to the centrifuge, as well as ensuring a low shear in the flocculated molasses, a mono pump with a variable speed drive was selected. This allowed molasses to be pumped into the centrifuge at rates between 0 to 1000 litres per hour. All the other process pumps were Salmson stainless steel four stage centrifugal pumps each with a capacity of 1000 litres per hour.

A hot water tank and high capacity KSB centrifugal pump ($7m^3/h$) was installed outside the building to provide backup dilution water and heating to the process vessels.

Process vessels were fitted with an internal coil, rolled from 6 meter of stainless tubing to provide a heating surface of 0.98 m^2. Vessels were also fitted with paddle or propeller type stirrers. Two tanks, used as inversion reactors, were fitted with Yokogawa pH-probes.

A variable area flow meter (rotameter) was used to measure flow into the centrifuge. Magnetic flow meters were used to measure flow into the ion exchange columns. Flow control on these was achieved with the use of actuated ball valves. Actuated ball valves were used to control condensate flow through the heating coils. To prevent water hammer caused by a valve closing quickly, the release of air from the valves was throttled to reduce the closing speed of the valve.

4.8 Plant Layout

Due to the high cost of automation it was decided that the pilot plant would be operated manually with the exception of temperature control in the vessels, and flow control through the ion exchange columns, both of which were automated. It was also felt that automation of the pilot plant could adversely influence the ease of adjusting the plant to variations in operating procedure, if the controllers had to be reprogrammed and field instruments had to be relocated.

The proximity of the main condensate storage tank of the factory to the pilot plant dictated the use of hot condensate as the heating medium for the vessels. This was cheaper to install, operate and maintain than the alternative medium of steam. The use of steam also requires specialised welding and equipment. Steam piping must undergo a third party inspection and pressure test before being commissioned. Leaks in a steam pipe could harm operators in the immediate vicinity of the leak. Furthermore it is also known that invert sugar is inclined to degrade and forms highly coloured caramels very easily and quickly when exposed to high temperatures, as would have been the case if steam was used as heating medium in the tubes. As the process temperatures would seldom exceed 70℃, and condensate can be safely pumped and used at this temperature, the danger of damage to the invert sugars was therefore reduced. A condensate leak, although dangerous, does not have as much danger as a steam leak, and moreover, condensate was also required as dilution water.

The large variations in molasses composition at different sugar factories make pilot plant evaluation so as to generate design data at each factory a prerequisite activity for full-scale design. Transportability of the pilot plant thus became a design requirement. This was accomplished by building the pilot plant in two skids. Each of the skids measured 3-meters wide, 6-meters long and 3-meters high. Tanks were suspended on a supporting level, 1.5-meters above the floor. All the process vessels, piping and equipment was installed within the outer framework. Only the utility piping protruded from the skids. Inter connecting piping, between the two skids were installed with a coupling (either flange or dairy type union) that ended flush on the structure. Piping for utilities ran through the centre of each skid from where it branched to the relevant vessels.

Each skid had it's own air manifold, electrical distribution and circuit breaker box, and instrumentation box. Below, in Figure 4.6, is a diagrammatic representation of the plan layout of the major pieces of equipment on the pilot plant. Pumps, piping and instrumentation are not shown.

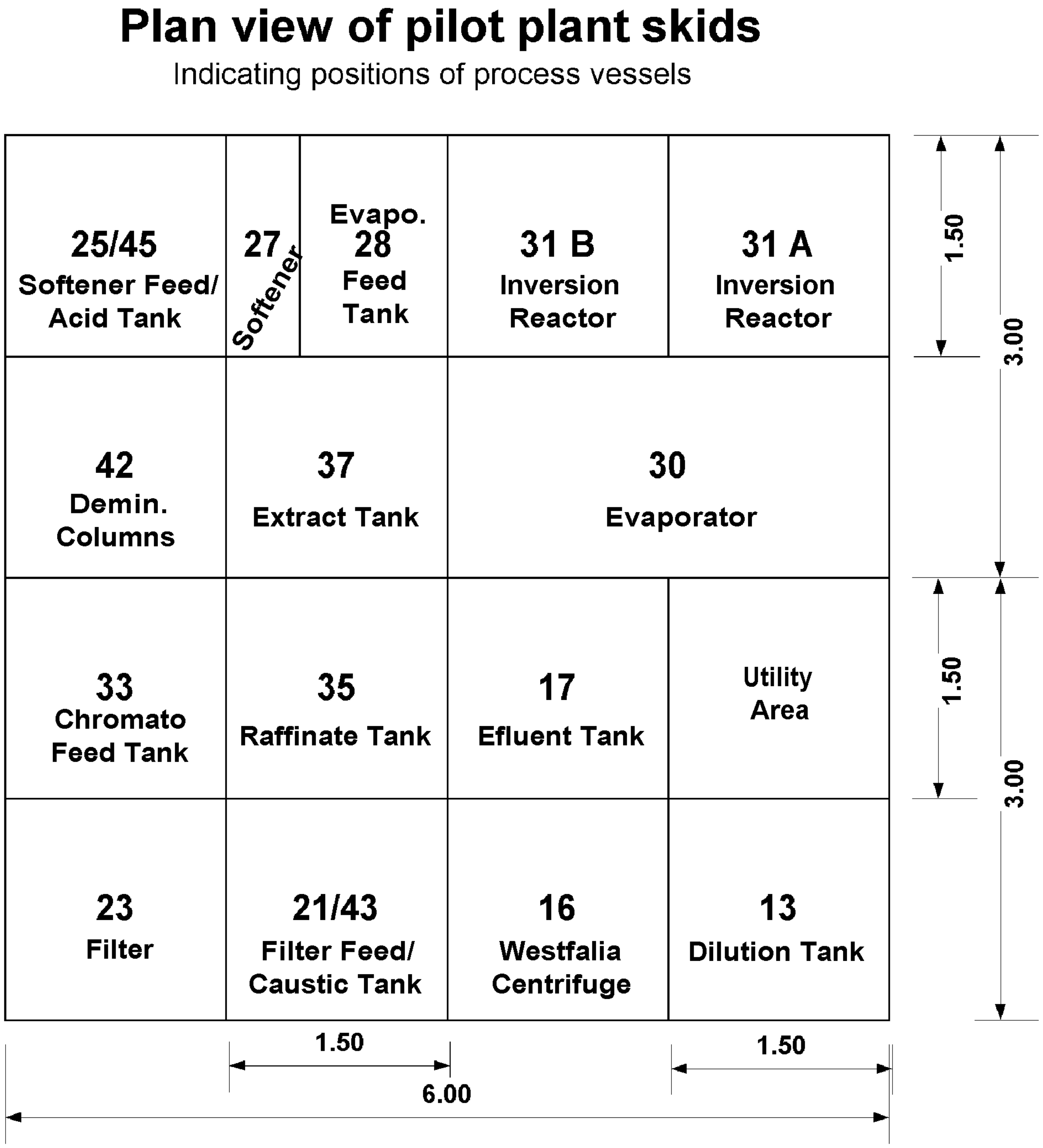


Figure 4.6: Diagram indicating the relative position of the major process items on the pilot plant.

The numbers in the diagram refer to the position of each vessel or processing unit. They were derived from the identification numbers allocated to the various equipment items during the procurement phase.

4.9 Final Installation & Commissioning

The pilot plant was to be constructed as economically as possible. This need resulted in a search and procurement of suitable second hand equipment. Some tanks and vessels were salvaged from previous pilot plants.

A forced circulation type evaporator was sourced and installed. The heat exchanger had a mild steel shell with stainless steel tubes and a heat transfer area of 6m^2. The condenser was made of stainless steel with a heat transfer area of 10m^2. The vapour liquid separator consisted of a vessel, 780-mm in diameter with a conical section on top and at the bottom. The separator was fitted with a 100-mm diameter tangential inlet and a 150-mm diameter vapour outlet. This installed unit is compared to the designed specification in Table 4.3.

The heat exchanger was slightly smaller than the one specified, but this would result in a slightly slower evaporation rate, should the assumption about the steam temperature be correct. The extra heat transfer area in the condenser would aid cooling of the condensate so as to prevent cavitation in the liquid ring vacuum pump. It also allowed a degree of safety should the cooling water flow rate decrease or the cooling water temperature increase. The inlet area of the separator was larger than specified, which would result in a lower flow rate of the two-phase flow entering the separator. The outlet pipe area is smaller than specified, which would result in a higher vapour velocity, which may cause entrainment if the velocity exceeded 4.2 m/s. However the selection of the pump and the larger inlet area provided an inlet velocity of below 3m/s. Finally the wall thickness was adequate to handle the expected vacuum.

Table 4.3: Comparison between designed evaporator equipment and available equipment installed.

Description	**Design Specification**	**Actually Installed**
Heat Exchanger:		
Heat Transfer Area	6.4 m^2	6.0 m^2
Condenser:		
Heat Transfer Area	7.4 m^2	10.0 m^2
Separator:		
Wall thickness	4.0 mm	4.5 mm
Inlet Area	0.025 m^2	0.031 m^2
Outlet Area	0.442 m^2	0.071 m^2

Stainless steel tubing with a nominal diameter of 40-mm was installed as the main process pipe, as this was immediately available. The pipe was connected to the pumps using standard Table D[♠], stainless steel flanges. All fittings were dairy type fittings. Butterfly valves were used in all instances except on the automated valves where ball valves were used. Pipe sections were connected either by dairy type unions or by Table D flanges. Flanges were "slip-on" type and welded to the piping. All welds were "pickled and passivated" after completion so as to prevent corrosion.

Mild steel was used for the structural support. Electrical cabling was supported on galvanised cable racks. Instrumentation cabling was installed inside galvanised

---

[♠] The dimensions and ratings for Table D flanges are available from suppliers, like Macsteel Trading (Pty) Ltd.

conduit pipe. Instrument air was distributed to the various instruments from a central manifold, on each side of the plant, using 6 mm plastic tubing.

The photographs below shows the pilot plant as installed.

Figure 4.7: Skid 1, with the dilution tank in the back, and only a corner of the filter showing.

Figure 4.8: The vapour-liquid separator and condenser.

Figure 4.9: The chromatography feed tank with utility piping visible in the front. The deashing columns are at the back.

Figure 4.10: The Chromatographic Separation Pilot Plant

Figure 4.11: Skid 2 with the inversion reactors in front and the utility supply piping can be seen above the personnel.

4.10 Conclusion

The pilot plant was successfully operated for the duration of the pilot plant testing. During this time only minor modifications were made. Most notably was the replacement of the pressure leaf filter with a press filter. The pilot plant was later extended to include a clarifier and some more decolourising and deashing columns.

The following chapters report on the design data that was gathered during the course of the project and how this was compiled into a feasibility study.

Chapter 5
Clarification of Molasses

5.1 Introduction

This clarification step is probably the most important step in the process, for without efficient solids removal the chromatographic desugarisation process fails. A pre-requisite for a successful chromatographic separation is a very low level of suspended solids in solution (<10 NTU/Brix) in the feed material. Suspended solids tend to block the flow path through the resin thereby hampering the separation performance of the chromatography process.

Various authors (Bernhardt (1998); Chen *et.al.* (1965); Davis *et.al*. (1962) and Saska (1993)) claim successful clarification of molasses by chemical treatment with phosphoric acid/caustic soda, the addition of sodium carbonate or sulphuric acid and the removal of the insoluble salts and adsorbed organic suspended solids by centrifugation. In most cases the resulting mother liquor also required a filtration step, aided by the addition of filter-aides in order to achieve the desired clarity.

Centrifugal clarification of molasses after treatment with sulphuric acid to convert calcium salts into insoluble gypsum was also the subject of a project by the Sugar Milling Research Institute (SMRI) to produce a feedstock for fermentation (Bernhardt, 1998). The centrifugal clarification method was investigated on both B-molasses and C-molasses obtained from the TSB Komati Mill. Based on indications from literature (Saska and Lancrenon, 1994), the use of polymeric flocculants to enhance clarification was also investigated. These ionically charged particles coagulate the suspended particles in the molasses and thereby change their characteristics to such an extent that they become bigger and yield more easily to the centrifugal force in the separator.

Ideally, the centrifuged mother liquor should be clear enough to be handled directly in a packed bed resin column. If this could not be achieved, the second best option will be a product of such a quality that a simple dead-end filtration step with the use of filter-aid should be able to yield the desired quality of molasses.

5.2 Pilot Plant Equipment and Operation

A condensed flow diagram of the process is presented below in Fig 5.1. This chapter covers the various attempts to optimise the first block in the diagram – Dilution and Clarification.

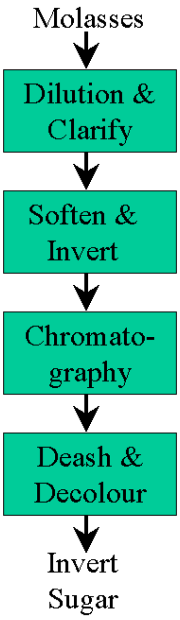


Fig 5.1 Condensed process flow diagram of proposed molasses desugarisation Process

The pilot plant at the Komati mill was equipped with a Westfalia Disk Bowl Centrifuge, which is pictured in Figure 5.2.

Figure 5.2: The Westfalia Disk Bowl Separator seen from the operator side. The feed and product pipework is visible at the back and the water pipe used to open and close the bowl is visible in the front. Sludge discharge is behind the unit on the right hand side.

The machine has an operating capacity of 4000 l/h at a backpressure of 4 bar. Normal operating flow for the molasses was between 600 l/h and 1200 l/h. The bowl is equipped with 47 conical disks. The machine builds a sludge layer on the inside of the bowl which then has to be released through the discharge ports into the housing and out through the discharge pipe. The bowl opens the discharge ports if water is pumped into a cavity at the bottom of the bowl. Due to the centripetal force, the water is forced into a channel between the two components of the bowl. The water pressure in the channel then lifts the top section, thereby opening the bowl's discharge ports. At the same time a release port is opened for the water and if the water pressure is not sustained the bowl will close as the water leaves the channel.

A cycle on the unit implies that the unit is partially desludged twice and completely desludged once. Partial desludging means that the bowl was opened for a short while, usually three to five seconds, while the unit is running at full feed rate. A complete desludging requires the feed flow to be stopped before the bowl is opened to discharge all the remaining sludge that has collected in the bowl. A time interval of five minutes was used between each step, resulting in a cycle time of 15 minutes. Factory "bearing cooling water" (BCW) was filtered through a 100-micron self-cleaning stainless steel Tekleen filter before being used to open the bowl. A safety filter (Y line strainer) of 100 microns was also installed directly before the centrifuge. This prevented plugging of the water ports in the bowl with bagasse or foreign matter.

Molasses was diluted and chemically treated in a 1000 litre stainless steel tank fitted with a Stallion propeller-type stirrer, with a diameter of about 300 mm and running at 1400 rpm, and a heating coil. Heating of the solution was done with warm factory condensate recycling through the pilot plant. Condensate was also used to dilute the molasses and to wash the centrifuge after each trial.

The molasses feed for a chromatography plant must have a turbidity of less than 10 NTU/Brix and a sludge content below 0.1 %. This is to prevent the pores on the resin from blocking and thereby reducing the available surface area and path length required for the liquid to achieve the desired separation. The following section describes the trials that were conducted on the centrifuge and the results obtained.

5.4 B Molasses Clarification

The clarification of B molasses was investigated first. The reason for this decision was that B molasses contains less concentrated impurities than final or C-molasses. The sugar content of B molasses is also higher than that of C molasses. A process that works well on B molasses could well be extrapolated and optimised for use on C molasses.

The detailed experimental descriptions and results obtained for the work done on B-molasses are presented in Appendix C.

The centrifugal clarification of B-molasses did not yield a product that was suitable for use in an ion exclusion process. However centrifugal clarification could be employed as a first step to clarify molasses, followed by a suitable downstream polishing process, which still had to be developed and installed. It was also evident that polishing by pressure filtration was not a suitable step, as the clarification in the centrifuge did not remove those particles that cause blinding of the filter media.

5.5 C-Molasses Clarification

Between the centrifugal studies on B-Molasses and the C-molasses trials, a laboratory study was conducted to evaluate the settling behaviour of sludge in phosphatated and flocculated B- and C-molasses. A few variables were investigated and it became evident through these experiments that the concentration of the feed solution is of vital importance to the successful clarification of the molasses - lower concentrations tend to produce a better quality product with the formation of large flocs. The flocculant concentration was also identified as an important variable. Higher concentrations of flocculant did not necessarily aid better coagulation of sludge, and in some instances, high flocculant dosages turned the molasses mixture into a slimy liquid.

Following these observations, the trials on C-molasses were conducted at much lower concentrations than those done earlier on the B-molasses. The first series of trials were aimed at studying the clarification ability of various flocculants **without** the addition of chemicals. A further series of trials investigated the clarification efficiency of a combination of flocculants and chemicals.

The detailed experimental descriptions and results obtained from the investigation of the clarification of C-molasses are presented in Appendix D. In these trials different types of flocculants and the combined effect of phosphatation and flocculation was investigated.

The combination of chemicals and flocculant did improve the clarification efficiency of molasses. The flocculant 6195 proved to be the most effective. (It also delivered the best performance in laboratory jug tests.) The results were also repeated in a further trial. Molasses treated during the last two trials was clear enough to be filtered - although it started to blind the filter towards the end of the run.

Overall, the centrifugal separation of molasses did not yield acceptable results. It is believed that the turbulence inside the centrifuge disintegrates the floc-particles, and once broken, they did not separate under centrifugal force as a sludge. It would seem that the density differential between disintegrated flocculant particles and molasses is too small to allow for adequate removal of the sludge by the centripetal force as it passes through the centrifuge. Centrifugation, with or without flocculant and/or chemical treatment, did not completely remove those particles that cause the blinding of the filter.

It was however evident that the flocculated suspended solids of chemically treated molasses tended to settle in the feed tank. This led to a series of investigations into the settling clarification of C-molasses.

5.6 Factors Affecting the Settling Clarification of Molasses

Based on the settling observed in the tanks during the trials with the centrifuge, an extensive series of laboratory experiments were conducted to identify a flocculant (or flocculants) capable of consolidating the suspended solids in molasses and causing it to settle out as a sludge from the molasses mixture. A large number of flocculants were tested but only a few could produce the desired sedimentation or come close to the desired result. The flocculant selected for this series of experiments was a cationic acrylate type flocculant from Montan Chemicals; product number 6195, this flocculant performed the best in the centrifugal separation trials.

Seifert Hazen and Sawyer (1990) published a general discussion of the settling regimes observed during clarification. Shelly (1997) quoted Richard Cordile (p66) as saying: ”Determining the ideal blend for site-specific conditions is really more an art than a science”.

Simpson (1996) reported that the measured zeta potential must be zero for optimum settling. He described four functions for a good flocculant: firstly it must accelerate the settling rate, secondly it must reduce the mud volume, the third criterion is that it must produce a clean overflow and finally this overflow must be easy to filter.

A number of factors affecting the efficiency of the clarification process were identified and investigated. These included the effects of temperature, age of molasses, variation in batches of flocculant, timing of chemical addition, concentration of flocculant, amount of conditioning chemicals used and concentration of molasses solids in solution before flocculation. These factors were studied on the pilot plant and were found to play an important role in the final process leading to the design and operation of a commercial plant.

5.7 Pilot Plant Equipment

For the purpose of the pilot plant experiments the amount of dry material was assumed to be equal to the Brix of the solution so as to simplify the operation of the pilot plant. The Brix determined by refractometer is very close to the real dissolved solids. It should be noted that the inaccuracy brought about by the assumption affected only the amount of chemicals added.

Settling trials were conducted in the pilot plant at the Komati mill. Figure 5.3 is a photograph of part of the plant. The tank in the background was the tank most often used for the dilution.

Standard 1000 litre stainless steel tanks were used as “clarifiers”. These tanks have a diameter of approximately 1 meter and are equipped with Stallion & Lightning propeller-type stirrers, with a diameter of about 300 mm and running at 1400 rpm, to facilitate mixing. Temperature is measured with a resistance temperature detector (RTD) probe in the bottom part of the tank. An internal heating coil, which is made from 6 meter rolled stainless steel tube and which is connected to the hot water circuit of the plant provides the heating surface required. Although this type of vessel was known not to be the best suited for the clarification, it proved to be quite capable of handling the process. The internal coil was a drawback because sludge settled on the piping. This probably resulted in some sludge being transferred during the siphoning stage. This configuration was chosen because it was easy to modify the available tanks, and moreover, tanks with an external heating jacket were too expensive.

Figure 5.3: A view of part of the molasses pre-treatment pilot plant. The tank at the back is the dilution and centrifuge feed tank. The centrifuge is the white and shiny object in the centre. The tank in the foreground is the filter feed tank. The orange enclosure houses the electrical panel.

Molasses was collected either from the factory molasses pumps or from the molasses tank. The collected molasses was slightly diluted in a drum to allow easier pumping into the plant. Final dilution took place inside the pilot plant tank. Phosphoric acid was added as an 80% solution at a ratio of 3.75 g H_3PO_4 (as 100%)/kg Brix. Caustic was added as a 50% solution at a ratio of 5 g (as 100%)/kg Brix. Flocculant was prepared in a 50-litre plastic conical bottom tank. Air was bubbled through the mixture to allow gentle, but thorough, low shear agitation and dissolving of the flocculant powder in the water.

The flocculant solution was added to the molasses mixture and the tank was stirred for about 30 seconds to allow proper dispersion of flocculant throughout the tank. The amount of flocculant added was 20 ppm. To determine the settling rate, a sample of the mixture was taken in a 1000 cc measuring cylinder and allowed to settle. Settling was considered complete when the volume of the sedimented sludge at the bottom of the flask remained constant. Initially the settling usually took about one hour. Once the sludge was judged to be settled, the clear liquid fraction was siphoned from the sludge into the next available tank for further processing and analysis.

In order to generate the data below a method of experimental design, reported by Murphy (1977) was used. It was, however, not always practical to rigorously follow the sequence prescribed by the statistical experimental design.

5.8 Factors affecting the clarification of molasses - Results and Discussion

5.8.1. Age of the diluted molasses

Trials were conducted in parallel to determine whether the age of diluted molasses was of any consequence to the clarification efficiency of the flocculant. Two tanks of molasses were prepared. The one tank was left overnight (usually to allow enough time for the inversion reaction to complete) while the other was treated immediately. The settling rates of the sludge were measured, as well as product clarity.

Typical settling and clarity values for old molasses and for freshly prepared molasses are presented in Table 5.1. All these trials were conducted at the same temperature, concentration and flocculant dose to ensure comparable results.

Table 5.1: Settling and clarity of "old" and fresh molasses

Experiment Number	**Settling Rate (Time to settle floc)**	**Recovered Volume of Clear Liquor**	**Clarity of Clear Liquor** (NTU/Brix)	**Averages for the variables reported**
Old Molasses				**Clarity:** Mean = 13.87
27	N/d	45 %	14.56	
28	N/d	39 %	40.11	
34	60 min	40 %	3.09	
35	60 min	40 %	3.04	**Volume:** Mean = 34.8
36	60 min	27 %	19.77	
37	60 min	32 %	13.69	
39	90 min	17 %	5.09	**Settling Time:** Mean = 64.3
40	60 min	31 %	19.37	
41	60 min	42.5 %	6.14	
Fresh Molasses				**Clarity:** Mean = 9.66
23	30 min	88 %	19.1	
38	N/d	80 %	16.53	
42	10 min	90 %	5.2	**Volume:** Mean = 83.13
43	15 min	85 %	10.63	
44	15 min	80 %	2.78	
45	22 min	85 %	9.09	**Settling Time:** Mean = 20.6
47	22 min	80 %	7.5	
48	30 min	77 %	6.48	

For comparison, the settling and clarity values for "old" molasses are presented in graphically in Figure 5.4 and for "fresh" molasses, in Figure 5.5.

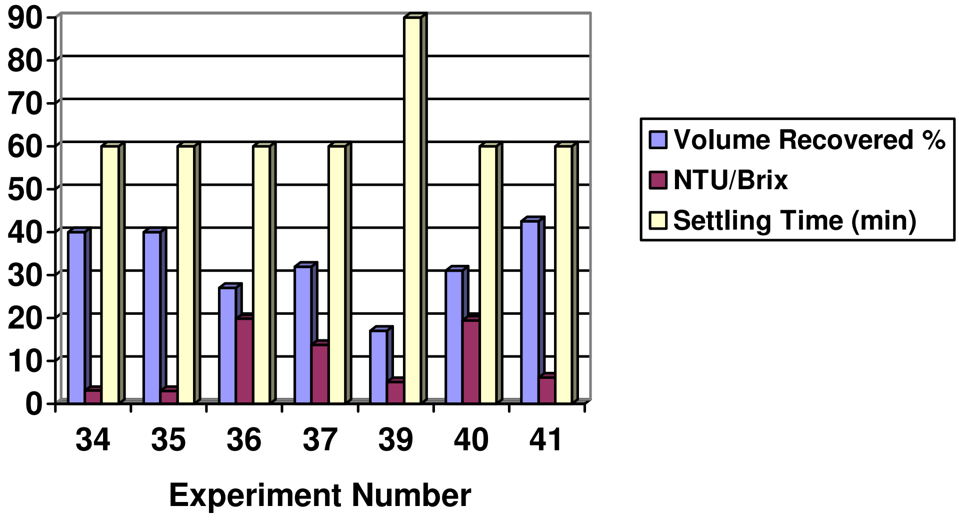


Figure 5.4: Clarification Behaviour of "Old" Molasses

Comparison of the bar charts in Figures 5.4 and 5.5 clearly reveals that fresh molasses yield more products and settles faster than older molasses.

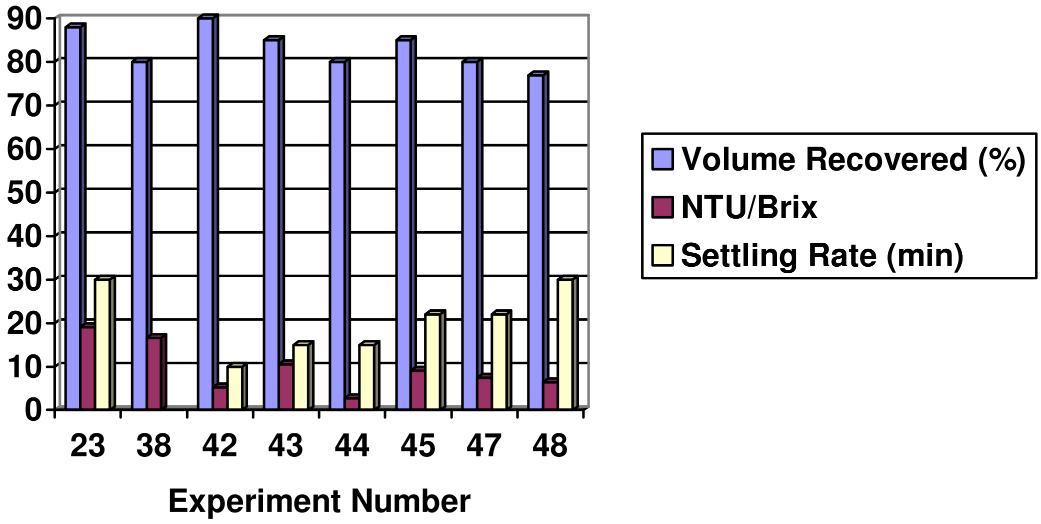


Figure 5.5: Clarification Behaviour of Fresh Molasses

Very good clarity was obtained on the older molasses, but the recovery of clear liquid and the settling time was worse than that of the fresh molasses. If the clarification process could be enhanced, it might be possible to use any age of molasses in the process and obtain acceptable clarity. It was therefore also recommended that a series of trials be executed to determine whether the inversion process has any influence on the clarification process or not. These trails were to be conducted as part of the evaluation of the commercial clarifier.

5.8.2 Effect of temperature on recovery and turbidity

The effect of solution temperature on the settling of sludge was evaluated by varying the bulk solution temperature for consecutive trials. The bulk molasses solution in this case was previously inverted. The clarity and settling data were compared in Table 5.2. From this preliminary trial a temperature of between 55℃ and 60℃ was used in consecutive experiments. The results for experiment 44, from Table 5.1, are included to allow for comparison with fresh molasses. All other parameters remained the same for these trials.

Table 5.2: Settling and clarity at various temperatures

Experiment Number	Temperature Of Mixture	NTU/Brix	Recovered Volume
27	60℃	14.5	45 %
28	50℃	40.1	39 %
44 (Fresh)	60℃	2.78	80 %

A graphic presentation of the results is shown as Figure 5.6.

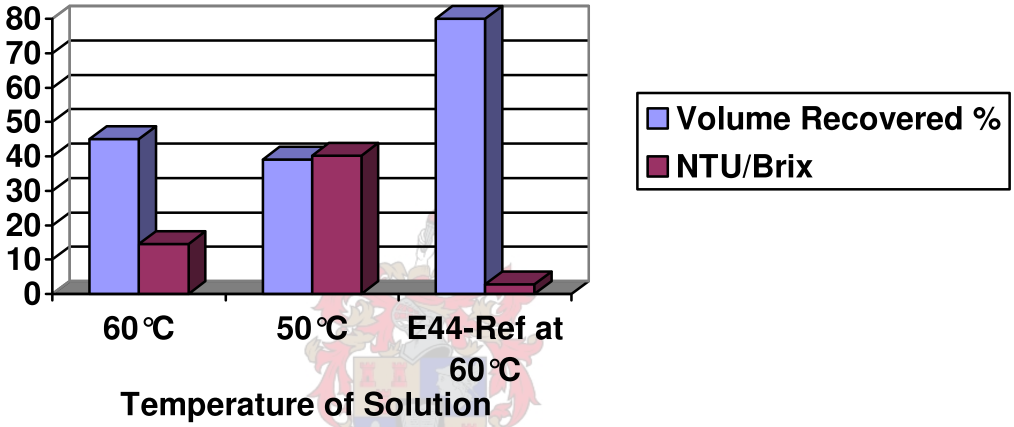


Figure 5.6: Effect of Temperature on Clarification

The higher temperature seemed to yield a clearer product. The lower viscosity at the higher temperature could possibly be the reason, as fine particles would tend to settle faster at lower viscosity. It was felt that more comparative trials were needed to confirm that the higher temperature always yields a clearer overflow and a better-compacted sludge. This effect will be investigated on the commercial clarifier.

5.8.3 Repeatability of flocculation across various batches of flocculants

Trials were conducted to determine whether different batches of the same flocculant produced the same results. Samples of a number of production batches were obtained from the supplier and these were evaluated during a series of plant experiments. All these trials were done using fresh molasses solution at around 20 Brix and the flocculant was dosed to a final concentration of 20ppm in all cases. The results are listed in Table 5.3. Note that the clarity measurements were made with a Turbidiquant turbidimeter. Note that the values are all, with one exception, below ten.

Table 5.3: Various batches of flocculant and the clarification results.

Experiment Number	Flocculant Batch	Clarity of Clear Liquor NTU/Brix	Statistical analysis of the various batches
46	H2484	3.8	Mean: 6.1
47	H2484	7.5	St. Dev: 1.6
48	H2484	6.4	Hypothesis test: z = -0.23
49	H2484	6.7	True if: -1.64 ≤ z ≤ 1.64
51	UCR4333	5.1	Mean: 6.0
52	UCR4333	6.6	St. Dev: 0.7
53	UCR4333	6.6	Hypothesis test: z = -0.25
54	UCR4333	5.7	True if: -1.89 ≤ z ≤ 1.89
57	UCR4458	8.2	Mean: 9.4
58	UCR4458	10.2	St. Dev: 1.1
59	UCR4458	9.8	Hypothesis test: z = 0.74
			True if: -1.37 ≤ z ≤ 1.37
		Population Average Clarity	7.0
		Population Standard Deviation	1.9

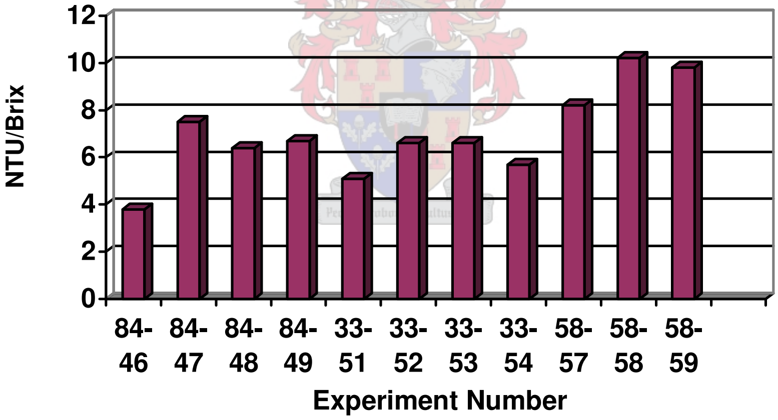


Figure 5.7: Clarification Behaviour of Different Flocculant Batches
(The prefix to the experiment number is the last two digits of the batch number)

The results are presented graphically in Figure 5.7. A hypothesis test on the mean clarity for the three batches, using a 90% significance level, proved that the various batches of flocculant did not affect the product or the settling of the sludge significantly. The experiments 58-58 and 58-59 yielded slightly longer settling times.

5.8.4. The effect of time between additions of various chemical additives

Trials were conducted to determine if the time period between additions of chemicals had an effect on the clarification behaviour of the molasses. Inversion, using enzymes, requires 8 - 12 hours to complete. The pH adjustment was done with phosphoric acid. Caustic soda and flocculant were added after 8 - 12 hours. In other trials chemicals were added after much shorter time intervals. The effects are detailed in Table 5.4. These results are represented in the graph in Figure 5.8.

Table 5.4: Variations in the time between the addition of H_3PO_4 and NaOH.

Experiment Number	Time between Dilution and H_3PO_4	Time between H_3PO_4 and NaOH	NTU/Brix	Settling Time	Statistical Means calculated for
42	10 min	8-12 hrs	5.2	10 min	Clarity: 6.19 NTU/Brix Settling: 16.67 minutes
43	10 min	8-12 hrs	10.6	20 min	
44	10 min	8-12 hrs	2.78	20 min	
45	10 min	10 min	9.09	20 min	Clarity: 7.69 NTU/Brix Settling: 21.67 min
47	10 min	10 min	7.5	15 min	
48	10 min	10 min	6.48	30 min	

The results indicate that the reaction time, of the phosphoric acid, did not seem to influence the clarification of the molasses. This was an advantage since the required plant capacity to allow enough residence time to complete the phosphotation and

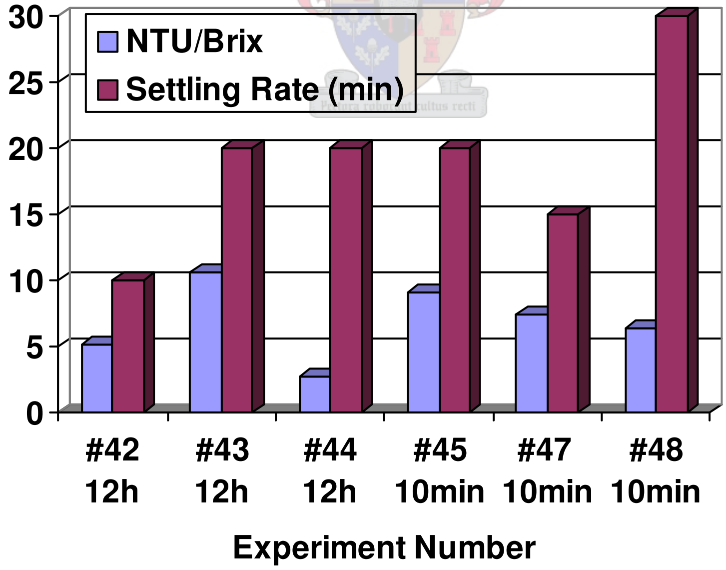


Figure 5.8: The effect of acid-reaction time on clarificatio

neutralisation reactions could be drastically reduced. This aspect was not investigated further in the evaluation of the commercial clarifier since the reaction is not necessaraily critical. The molasses was allowed to invert overnight, in the presence of phosphoric acid. Caustic can be added to the clarifier feed by a dosing pump and would still neutralise the molasses solution in the pipe leading to the clarifier.

5.8.5 The optimum flocculant concentration

Flocculant 6195 is a cationic flocculant, meaning it will bond to negatively charged partickles in the solution. It has a moderately strong charge and has a polyacrylamide matrix. The concentration of the flocculant solution that is dosed to the molasses was investigated. Usually a solution containing 1000-ppm flocculant in water was prepared. This solution was dosed to the diluted molasses mixture to allow a final concentration of 20 ppm flocculant in the bulk liquid. The concentration was varied to investigate the effect it would have on the clarification. Both experiments (24 & 25) were done on old molasses using the same feed batch. The outcome of these trials is listed in Table 5.5. A comparative trial is experiment 23 using fresh molasses and a 1000-ppm solution dosed as 20-ppm to the mixture. A graphical presentation is provided in Figure 5.9.

Table 5.5: Various concentrations of flocculant and the clarification of molasses

Experiment Number	Concentration of flocculant solution	Volume of Recovered Liquor	Settling Rate (Time to reach stable volume)
23 (Fresh)	1000 ppm	90 %	30 min
24	800 ppm	34 %	15 min
25	1000 ppm	54 %	40 min

The optimum concentration for the flocculant solution that was dosed to the bulk liquid was 1000 ppm. This concentration allows for proper floc formation and proper settling. A series of laboratory experiments were done to confirm this. The results[ℵ]

[ℵ] TSB Internal Report: Evaluation of flocculants for the clarification of molasses – A Aspelund & B Bjarnarsson.

are described in another paper. Further optimisation trials have to be done to verify the observations above.

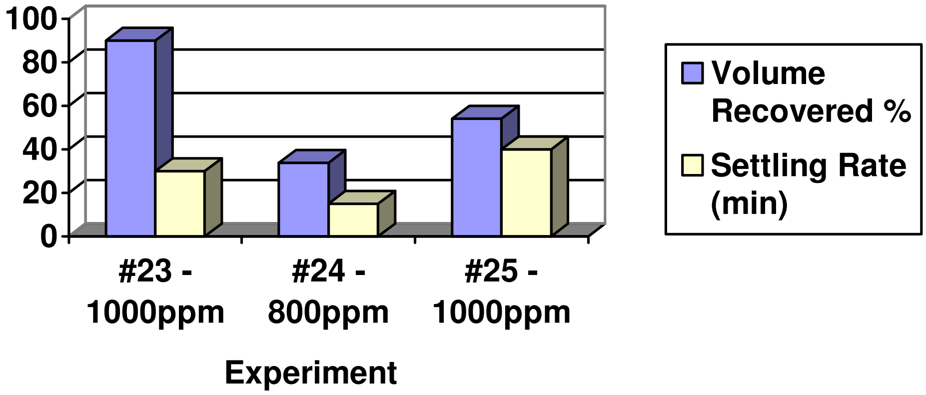


Figure 5.9: Effect of flocculant dosing concentration on clarification

5.8.6. The optimum concentration for the molasses solution

There are process descriptions in the literature detailing the clarification of molasses at about 50 Brix (Schneider, 1978). Trials by the author and co-workers indicated that a lower concentration enhances the clarification efficiency of the flocculant. This is part of the subject of a filed patent (Bekker PI, Stolz HNP, *et.al.* 2001). The upper limit of the concentration where the flocculant still gave good performance, was determined. Various concentrations and the clarification results are reported in Table 5.6. All these were treated in the same way and dosed with the same amount and concentration of flocculant. This data is graphically represented in Figure 5.10. It is evident that at a molasses concentration above 30 Brix the settling rate of the flocculated sludge is unacceptably long, although clarity is acceptable. The optimum concentration of the molasses was found to be 28 Brix. Concentrations between 25 Brix and 28 Brix provided good settling rates of the floc and clear product.

Table 5.6: Concentration of the molasses solution and its effect on clarification.

Experiment Number	Concentration of molasses	NTU/Brix	Volume of Recovered Liquor	Settling
46	32.4	3.79	20 %	60 min
47	28.7	7.5	80 %	15 min
48	33.2	6.48	78 %	50 min
49	27.1	6.73	89 %	15 min
50	30.3	N/d	80 %	40 min
60	24.7	26.0	74 %	5 min
61	26.5	7.90	91 %	10 min
62	28.9	7.04	91 %	10 min

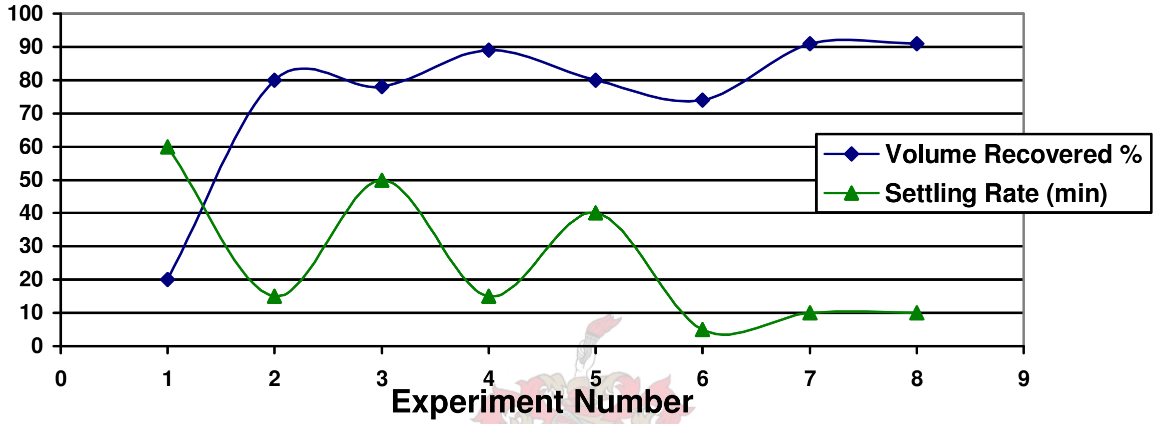


Figure 5.10: Effect of Molasses Concentration on Clarification

This phenomenon is attributed to the smaller differential in density between the flocculated sludge and the molasses at higher concentration. The density of molasses at 25 Brix is 1.104 kg/l and at 35 Brix it is 1.152 kg/l. The driving force for settling is larger at the lower molasses concentration. The clarity for these trials were all acceptable, although there was some contamination in the final product of trial 60, caused by sludge that was accidentally siphoned. The sludge, once exposed to shear stress, does not coagulate anymore and causes a fine haze to appear in the liquid.

5.8.7. The optimum chemical dosing.

The dosage ratio of H_3PO_4 was accidentally changed during the course of the project. The specified dosage for H_3PO_4 is 3,00 kg Acid (as 100%)/Mt Dry Solids. This converts to 3,00 x (100/80) = 3.75 kg H_3PO_4 (as 80%)/Mt Dry Solids. The H_3PO_4

dosage was accidentally changed to a value of, 3,75 kg H_3PO_4 (as 100%)/Mt Dry Solids. This led to the trend listed in Table 5.7.

Table 5.7: The variation of acid dosage to the settling performance of sludge.

Experiment Number	Dosage of H_3PO_4 (as 100%) (kg/t DM)	NTU/Brix	Volume of Recovered Liquid	Settling Rate	Statistical Means for Clarity and Volume
22	3.0	22.99	30 %	N/d	Clarity: 20.98 Volume: 68%
23	3.0	19.12	90 %	60 min	
26	3.0	20.83	84 %	25 min	
42	3.75	5.2	70 %	10 min	Clarity: 6.19 Volume: 76.6
43	3.75	10.6	80 %	10 min	
44	3.75	2.78	80 %	10 min	

This data can be represented graphically as seen in Figure 5.11.

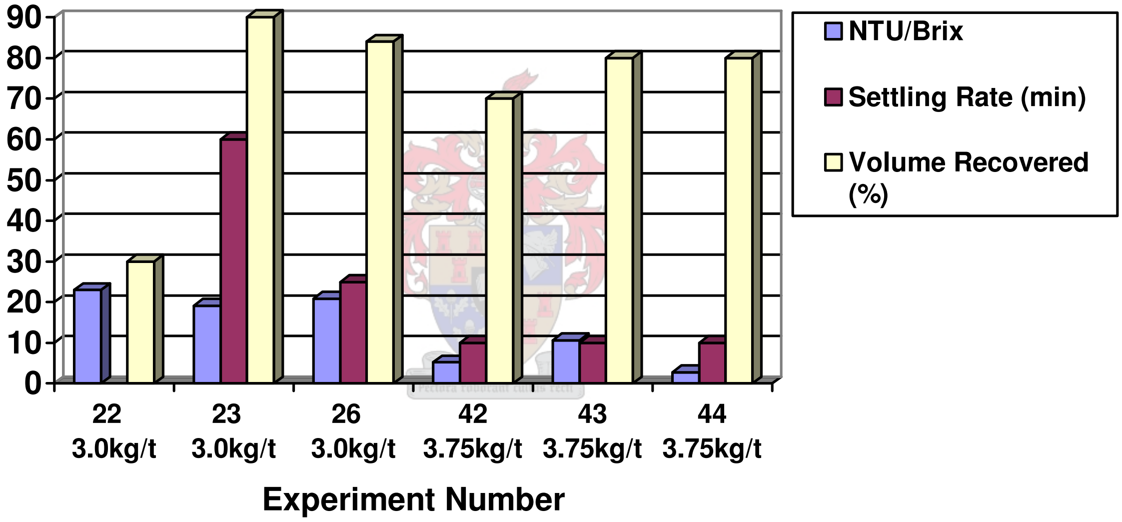


Figure 5.11: Effect of Increased Acid Dosage to the settling of sludge

As can be seen, the higher H_3PO_4 dosage led to acceptable clarification in a relatively short time. Future experiments to confirm the trend and to determine the optimum settling performance, product quality and acid requirement, were done during the optimisation of the commercial clarifier. A higher acid dosage would need more caustic to adjust the pH. This would have financial implications and a trade-off must be found. This was followed up during the investigation and optimisation of the commercial clarifier.

5.9 Discussion & Implications

Freshly diluted molasses was easier to clarify than older, stored molasses after flocculation. The sludge volume of fresh molasses was far less than the sludge volume of molasses that had been diluted for some time. Temperature played a significant role in the settling of the sludge - the optimum being between 55°C and 60°C. Higher temperatures seemed to prevent the formation of large floc particles and reduced the settling rate of the floc that had formed. Lower temperatures inhibited the settling of the floc particles.

The performance of the flocculant (polyelectrolyte) was consistent over a series of different flocculant batches. The clarification action was consistent over a range of molasses conditions. Chemical addition could occur within a reasonably short time. There were no significant or noticeable differences in the clarification behaviour between batches that had received chemicals in 10-minute intervals and batches that were treated after 9 to 10 hours with caustic soda and flocculant.

The concentration of flocculant solution used to dose the molasses solution appears important. The optimum concentration was 1000 ppm. A lower limit was 900 ppm and 1100 ppm, as the maximum limit, appeared acceptable. Dosing with concentrations lower than 900 ppm resulted in delayed floc formation and poor settling. Dosing with concentrations above 1100-ppm resulted in the flocculant not mixing properly with the solution before settling resulting in a slimy solution. The optimum amount of flocculant to dose was 20 ppm (mass of flocculant to volume of molasses solution). Present indications are that molasses with a lower sludge content did not require as much flocculant as molasses with a higher sludge content

The maximum concentration of solution before flocculation was 30 Brix. At concentrations higher than 30 Brix the settling of the flocculant was slow and incomplete. The optimum concentration was 28 Brix. This provided proper settling and the clarity was similar to that of lower brix solutions. Phosphoric acid dosed as 3,75 g (as 100%)/ kg Dry material yielded better settling performance than those tests done with a lower dosage of acid. Reaching this point it was clear that setting in a clarifier held a significant advantage to using a centrifuge.

Tests to evaluate the clarification on commercial equipment were now required. During these tests, attention would be given to the uncertainties listed in the above discussion.

5.10 Pilot Plant Evaluation of a Commercial Clarifier

Clarification and thickening is a well-known process used extensively in the water treatment industry. Juice clarification is used in the sugar industry the world over (Chen and Chou (1993)). The clarification unit operation as such is therefore not a novelty for the sugar industry. Because of the wide rage of commercial equipment available it was difficult to make a choice about a specific system suitable for use in molasses desugarisation off hand. Some investigation had to be done. The equipment had to comply with the following requirements:

1. High efficiency to separate sludge and liquid with a small density difference.
2. Low shear to prevent flocculant clusters from disintegrating.
3. Simple in construction, operation and low in maintenance.

The principles of design and control of clarifiers and thickeners were reported by Seifert (1987). He presented a list of criteria for selecting a clarifier for a specific purpose. Investigation of various commercially available products led to the detailed evaluation of the E-Cat^{TM} clarifier distributed by EIMCO. A pilot unit was obtained and tested under process conditions at the pilot plant at Komati Mill.

Evaluation was started based on the operating conditions determined during the settling trials on molasses. The process was optimised to determine the optimum operating conditions for the process. Special attention was to be given to clarify the uncertainties identified during the previous trials.

5.11 E-CAT ™ operating principle

Energy, in the form of turbulence, must be applied to a solution treated with flocculant to assist the formation of flocs by causing particles to collide. Collided particles, in the absence of destructive shear forces, will remain joined and will grow in size, resulting in an increase in density and settling velocity until the agglomerated particles separate by gravity from the suspending liquid. A diagram of a typical E-Cat^{TM} system is shown in Figure 5.12.

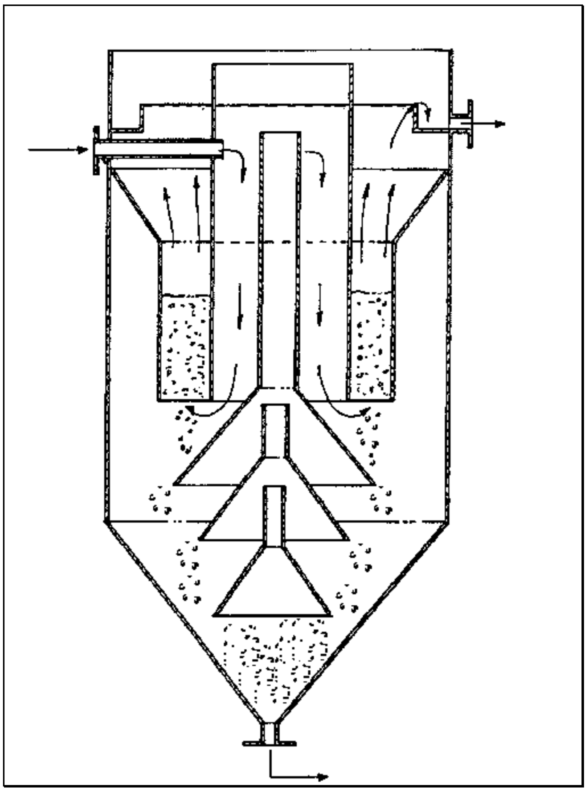


Figure 5.12: Drawing indicating the flow of clear liquid and agglomerated solids in an E-CAT ™ clarifier. Also shown are the relative positions of the clarifying cylinders and the dewatering cones. [Batson, RG (1998)]

The feed solution is pumped into a centre feed well. Flocculant is added to the feed either inline or directly in the feed well. Agglomeration starts taking place in the feed section of the clarifier. Product flows from the feed section into the second section of the clarifier, the hindered zone. The only way for liquid to leave the clarifier is through one of the clarification cylinders protruding from the zone. The liquid accelerates through the cylinders, causing turbulence and improving the potential of collisions between particles. A separation layer is then formed. Should particles exit the cylinder, the sudden loss of kinetic energy will cause it to settle again.

Figure 5.13 shows the turbulence caused by the reduction in area through a clarifying cylinder:

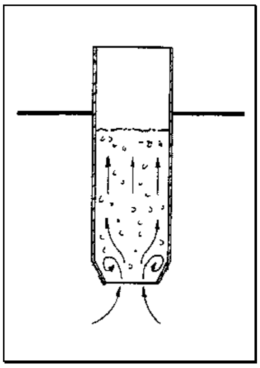


Figure 5.13: Turbulence in a clarifying cylinder [Batson, RG (1998)]

Heavy consolidated floc settles from the bottom of the cylinders through the hindered zone down to the compaction zone. The floc, settling through the bottom part of the hindered zone and the top of the compaction zone, might hit a dewatering cone during their downward motion. Floc and agglomerated solids slide from the sides of the cone. The dewatering cone also channels liquid, expelled by the compaction of the flocs to the centre of the off-take pipe. This pipe releases its contents in the feed well and aids mixing of the incoming feed product. Figure 5.14 shows the principle of operation of a dewatering cone.

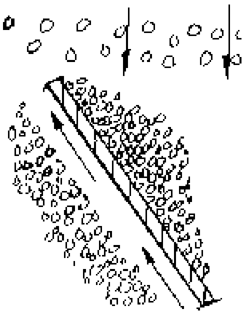


Figure 5.14: Settling on a sloped surface. [Batson, RG (1994)]

The mixing occurring in the E-Cat^{TM} is of low shear so that minimal floc breakage occurs.

5.12 E-CAT ™ installation at Komati Pilot Plant

Baker Hughes Company supplied a 300-mm nominal diameter pilot clarifier. The unit was erected outside the pilot plant building at the Komati Mill. Molasses was diluted and prepared by treatment with phosphoric acid and caustic in a tank in the pilot plant building from where it was pumped to the clarifier. Flocculant was initially pumped into the feed zone of the E-Cat^{TM}, but changed at a later stage so as to allow a longer mixing time between the liquids. Overflow was piped to a receiving tank in the pilot plant building.

The feed entered the clarifier through the side, below the product overflow. The pilot unit was only equipped with one clarifying cylinder, located in the centre of the unit. There were three dewatering cones installed in the lower part of the hindered zone and the compaction zone. The liquid collected by the cones was returned to the top of the unit directly into the feed zone.

Flow-rate into the unit was 300 l/h and temperature was maintained at 65℃. Optimisation of the clarification and pre-treatment process was done. A series of trials were conducted to determine the operating efficiency of the unit.

5.13 Trials, Results and Discussion

Diluted molasses, pre-treated with phosphoric acid and caustic soda solution was continuously pumped into the clarifier. A flocculant solution was injected into the molasses feed line and thoroughly mixed using a mixing loop, just before it entered the clarifier.

Various trials were conducted to determine the optimum operating parameters of the clarifier. It was noted that substantial cost savings could be achieved should the clarifier be able to facilitate efficient agglomeration and settling of the particles in order to process the molasses at the lowest possible chemical dosage.

Figure 5.15: The E-CAT ™ clarifier installed at the pilot plant

The precipitation of calcium-phosphate salts occurs at pH above neutral and an investigation into the decalcification of molasses in the clarifier, rather than on a softener was also done.

The trials were conducted at a 300-litre/hour flow-rate, which allowed for an hour retention time in the clarifier. The operating temperature was set at 65°C throughout the trials. In some cases this temperature could not be achieved and in the results the reduced efficiency of the process at lower temperatures can be seen. The results obtained during these trials are listed in Table 5.8 below.

Table 5.8 ***Molasses Clarification on the E-CAT™ Clarifier***

Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			% NTU Removed
		Brix	pH	NTU/Brix	Brix	pH	NTU/Brix	
A Series	***Ave***	***23.28***	***5.80***	***100.72***	***16.24***	***5.99***	***37.06***	***63%***
3.75kg H_3PO_4/tDS	**St Dev**	***1.78***	***0.24***	***7.48***	***1.87***	***0.12***	***9.95***	***11%***
Notes: 5kg NaOH/tDS (added as 50% lye); 300 l/h; 65°C; 20 ppm 6195								
B Series	***Ave***	***25.19***	***5.85***	***101.6***	***21.48***	***6.56***	***29.99***	***70%***
3.75kg H_3PO_4/tDS	**St Dev**	***0.77***	***0.22***	***7.55***	***3.08***	***0.55***	***5.08***	***6%***

Table 5.8 ***Molasses Clarification on the E-CAT™ Clarifier***

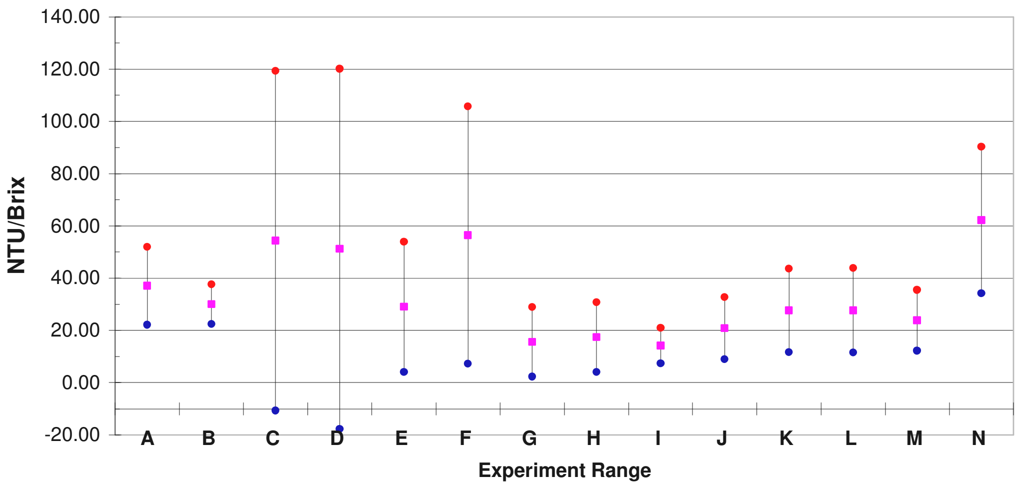
Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			
		Brix	pH	NTU/Brix	Brix	pH	NTU/Brix	% NTU Removed
Notes: 5kg NaOH/tDS (added as 50% lye); 300 l/h; 65°C; 20 ppm 6195								
C Series	***Ave***	***23.07***	***6.24***	***121.0***	***21.27***	***6.70***	***54.30***	***54%***
3.5kg H_3PO_4/tDS	**St Dev**	***1.44***	***0.69***	***20.22***	***2.46***	***0.46***	***43.38***	***39%***
Notes: Adj. pH to 7.0 with 50% NaOH lye; 20 ppm 6195; 300 l/h; 65°C								
D Series	***Ave***	***24.77***	***6.18***	***122.6***	***22.22***	***7.33***	***51.17***	***56%***
1% H_3PO_4 on DS	**St Dev**	***1.33***	***0.27***	***30.55***	***3.06***	***2.20***	***46.00***	***42%***
Notes: Adj. pH to 7.4 with 50% NaOH lye; 20 ppm 6195; 300 l/h; 65°C								
E Series	***Ave***	***24.10***	***5.93***	***119.3***	***20.10***	***6.76***	***29.01***	***74%***
1% H_3PO_4 on DS	**St Dev**	***3.01***	***0.19***	***14.19***	***4.88***	***0.83***	***16.63***	***16%***
Notes: Adj. pH to 7.4 with 50% NaOH lye; 20 ppm 6195; 300 l/h; 65°C								
F Series	***Ave***	***25.64***	***6.27***	***112.5***	***21.79***	***5.85***	***56.43***	***46%***
1% H_3PO_4 on DS	**St Dev**	***1.54***	***0.86***	***23.66***	***2.91***	***1.53***	***32.82***	***37%***
Notes: Adj. pH to 7.4 with 50% NaOH lye; 20 ppm 6195; 300 l/h; 65°C								
G Series	***Ave***	***22.49***	***5.98***	***99.1***	***20.69***	***5.95***	***15.57***	***84%***
1000 ppm H_3PO_4	**St Dev**	***0.52***	***0.35***	***16.02***	***0.96***	***1.48***	***8.93***	***11%***
Notes: Adj. pH to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h; 65°C; deaerate molasses; inline floc mixer								
H Series	***Ave***	***22.21***	***5.73***	***95.6***	***19.79***	***5.82***	***17.39***	***82%***
1000 ppm H_3PO_4	**St Dev**	***1.18***	***0.29***	***6.04***	***2.26***	***1.41***	***8.90***	***10%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65°C								
I Series	***Ave***	***22.33***	***5.40***	***127.5***	***21.82***	***6.12***	***14.20***	***89%***
750 ppm H_3PO_4	**St Dev**	***0.34***	***0.14***	***13.86***	***0.59***	***1.79***	***4.54***	***4%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65°C								
J Series	***Ave***	***22.46***	***5.52***	***134.4***	***21.43***	***5.83***	***20.80***	***84%***
500 ppm H_3PO_4	**St Dev**	***0.49***	***0.08***	***17.84***	***0.85***	***0.25***	***7.96***	***6%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65°C								
K Series	***Ave***	***22.34***	***5.28***	***126.9***	***21.30***	***5.79***	***27.63***	***78%***
250 ppm H_3PO_4	**St Dev**	***0.65***	***0.21***	***14.05***	***1.02***	***0.35***	***10.61***	***10%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65°C								
L Series	***Ave***	***22.21***	***5.38***	***108.5***	***20.83***	***5.23***	***27.66***	***74%***
0 ppm H_3PO_4	**St Dev**	***1.24***	***0.18***	***13.48***	***1.69***	***0.28***	***10.77***	***11%***
Notes: No pH Adjustments - 20 ppm 6195 on molasses at 22 Brix; 300l/h at 65°C								
M Series	***Ave***	***22.18***	***5.44***	***118.3***	***21.84***	***7.08***	***23.82***	***80%***
0 ppm H_3PO_4	**St Dev**	***1.19***	***0.05***	***13.49***	***1.01***	***1.84***	***7.80***	***7%***
Notes: pH > 7 with NaOH – 20ppm 6195								
N Series	***Ave***	***22.77***	***5.36***	***109.9***	***22.41***	***6.68***	***62.21***	***43%***
0 ppm H_3PO_4	**St Dev**	***0.98***	***0.12***	***10.53***	***0.94***	***0.39***	***18.68***	***16%***
Notes: pH > 7 with NaOH – 20ppm Magnafloc LT27								

The results from the table above are graphically represented in Figures 5.16 and 5.17, which show, overflow clarity and clarification efficiency. The marker in the centre of each line is the average value obtained for that series of tests while the other points indicate the range as follows:

High = Average + 1,5 (Standard Deviation)

Low = Average – 1,5 (Standard Deviation)

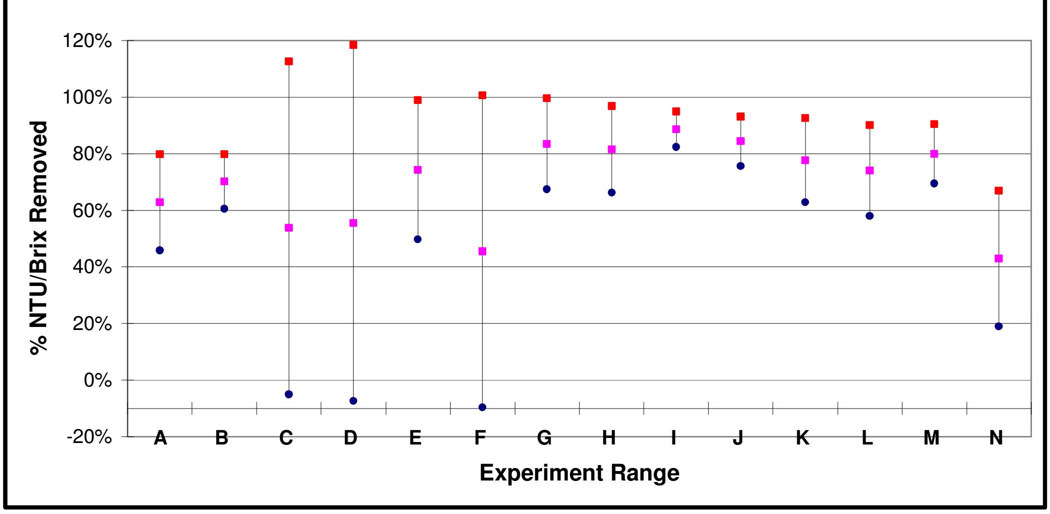
Figure 5.16: Overflow Clarity



Most of the large deviations can be attributed to the effect of temperature. In the existing pilot set-up it is very difficult to maintain a constant temperature in the clarifier while adjusting other parameters. In an attempt to remove calcium (as a precipitated phosphate salt) from the molasses the pH of Trials C, D, E and F were raised to 7.0 or higher. The clarification was not efficient under these conditions. A repeat during series M, with no phosphoric acid added did however, show an adequate performance. The addition of anionic flocculant, during trial N, did not improve the clarification of the molasses.

Series A-F were all pre-treated with phosphoric acid. Quantities were varied from 3.75g/kg DS to 10g/kg DS. In series A and B caustic soda (5g/kg DS) as 50% lye was added before the addition of flocculant (20ppm) and clarification. Turbidity removal on average varied between 44 and 78%. In general a too wide scatter in turbidity removal was obtained. The reason for increased turbidity in a few cases was also traced to microbial activity occurring in the clarifier when not in continuous operation. At start-up the clarifier is normally filled with water. Pre-treated and flocculated molasses flowing into the clarifier then displaces the water.

Figure 5.17: **Clarification Efficiency**



Later in the trials boiling hot water was used to fill and rinse the clarifier in an attempt to lower the microbial fermentation and raise the temperature of the clarifier at start-up.

The lower Brix values in the overflow column of Table 5.8 are due to dilution brought about by the displacement process. The amount of phosphoric acid added was then reduced to 3g/kg DS and enough caustic lye was added to obtain an overflow pH of about 7 (series C). The average overflow turbidity remained similar to those of series A and B but the spread in turbidities became much wider. For the experiments in series D, E and F phosphoric acid addition was increased to 10g/kg DS and sufficient lye was added to adjust the pH to 7.4. It can be seen (Figures 5.16 and 5.17) that the modified pre-treatment did not effect the average turbidity to any large extent but the spreads in turbidity remaining and turbidity removed remained unacceptably high.

Despite the overall unacceptability of the results obtained, it was encouraging that in every series (C.D.E.F) a number of experiments did yield batches of molasses that were clarified to below the goal of 10 NTU/Brix. It was not possible to determine the reason(s) for this phenomenon scientifically by statistical means due to the variation allowed in the initial concentrations and the pre-treatments that were used. Also of note was the tremendous buffering capacity of molasses. Despite the fact that the pH was adjusted to 7.4 in the last three experiments before clarification, the average overflow pH of the clarified molasses was well below 7, two to three hours later.

From scatter diagrams of Turbidity vs. Brix and Turbidity vs. pH it was concluded that the best results could be obtained by maintaining the solids concentration at 22±1 Brix and aiming for an overflow pH of 5.5 - 6. The addition of more phosphoric acid and caustic soda to increase the amount of phosphates formed at a higher pH had a detrimental rather than a beneficial effect. It was therefore decided to reduce the addition of phosphoric acid to 1000ppm and add just enough caustic soda lye to bring the pH to about 6.5. Dilution was also avoided in the clarifier, by displacement of molasses with molasses and not water with molasses.

Some other improvements were also introduced. It was noticed that the sludge recovered from the bottom of the clarifier had a sponge like appearance as if air was mixed into it. Using the conditions specified above, series "G" was conducted by first de-aerating the pre-treated molasses using the vacuum capability of the pilot evaporator. The detailed results are presented in Table 5.9.

Table 5.9 - Deaerated Molasses

Experiment Number		**Feed (diluted molasses)**			**Overflow (clarified molasses)**			
		Brix	**pH**	**NTU/Brix**	**Brix**	**pH**	**NTU/Brix**	**% NTU Removed**
G Series	***Ave***	***22.49***	***5.98***	***99.1***	***20.69***	***5.95***	***15.57***	***84%***
1000 ppm H_3PO_4	**St Dev**	***0.52***	***0.35***	***16.02***	***0.96***	***1.67***	***8.93***	***11%***
Notes: Adj. pH to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h; 65°C; deaerate molasses; inline floc mixer								
G2		22.97	5.86	125.1	21.24	6.30	11.8	**90.6%**
G3		22.80	5.86	89.4	21.15	5.22	18.9	**78.8%**
G4		22.80	5.86	89.4	21.58	5.27	29.6	**66.9%**
G5		21.80	6.60	104.2	19.20	6.75	7.2	**93.0%**
G6		22.07	5.71	87.5	20.30	6.21	10.3	**88.2%**

The sludge resulting from the de-aerated feed became much less spongy in appearance. Simultaneously, the average turbidities of the overflows decreased to less than 20 NTU/Brix while the variation in turbidity was much less. After the necessary modifications to avoid the capture of air in the clarifier feed and tightening up on the flow control to the clarifier and the flocculant dosing and mixing operations, series H were produced at a rate of about 3 batches in 24 hours.

Table 5.10 - Deaerated Molasses

Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			
		Brix	pH	NTU/Brix	Brix	pH	NTU/Brix	% NTU Removed
H Series	***Ave***	***22.21***	***5.73***	***95.6***	***19.79***	***5.82***	***17.39***	***82%***
1000 ppm H_3PO_4	**St Dev**	***1.18***	***0.29***	***6.04***	***2.26***	***0.71***	***8.90***	***10%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65°C								
H2		21.30	5.74	97.7	20.34	6.54	9.5	**90.3%**
H3		22.74	5.95	90.0	21.11	6.29	11.0	**87.8%**
H4		20.39	6.10	95.2	18.80	4.72	31.1	**67.4%**
H5		22.90	5.29	85.5	19.20	5.20	28.8	**66.4%**
H7		21.40	5.94	103.2	15.45	6.59	15.5	**85.0%**
H8		23.32	5.53	99.4	21.40	5.91	10.4	**89.6%**
H9		23.44	5.56	98.5	22.20	5.47	15.5	**84.2%**

Series H, reported in Table 5.10, confirmed the reproducibility and consistency of the turbidity removal process that evolved. Average turbidity removal and the standard deviation of turbidities were similar to that of series G.

The effect of phosphoric acid addition was again investigated in series I, J, K and L. Addition of phosphoric acid was reduced from 1000ppm/DS (G, H) to 750-ppm (I), 500ppm(J), 250ppm (K) and 0 ppm (L). The effects of this reduction can be seen in the detailed results of the experiments presented in Table 5.11.

Table 5.11 - Effect of Phosphoric Acid on Clarification

Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			
		Brix	pH	NTU/ Brix	Brix	pH	NTU/ Brix	% NTU Removed
I Series	***Ave***	***22.33***	***5.40***	***127.5***	***21.82***	***6.12***	***14.20***	***89%***
750 ppm H_3PO_4	**St Dev**	***0.34***	***0.14***	***13.86***	***0.59***	***1.79***	***4.54***	***4%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65°C								
I2		22.73	5.42	107.5	21.12	6.56	9.8	**90.9%**
I3		22.90	5.06	112.2	23.00	6.52	23.0	**79.5%**
I5		22.44	5.56	126.2	21.80	5.47	19.5	**84.6%**
I7		22.20	5.41	155.2	21.20	6.05	12.2	**92.1%**
I8		22.10	5.43	128.4	21.32	5.95	10.9	**91.5%**
I9		21.88	5.47	124.1	22.04	5.88	10.9	**91.2%**
I10		22.16	5.47	124.2	21.70	6.04	11.1	**91.0%**
I11		22.50	5.40	136.3	22.27	6.34	14.2	**89.6%**
I12		22.04	5.38	133.2	21.96	6.23	16.2	**87.9%**
J Series	***Ave***	***22.46***	***5.52***	***134.4***	***21.43***	***5.83***	***20.80***	***84%***
500 ppm H_3PO_4	**St Dev**	***0.49***	***0.08***	***17.84***	***0.85***	***0.25***	***7.96***	***6%***

Table 5.11 - Effect of Phosphoric Acid on Clarification

Experiment Number		**Feed (diluted molasses)**			**Overflow (clarified molasses)**			
		Brix	**pH**	**NTU/ Brix**	**Brix**	**pH**	**NTU/ Brix**	**% NTU**
								Removed
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65℃								
J4		23.10	5.53	176.9	21.91	5.51	12.8	**92.8%**
J5		22.10	5.60	122.4	21.71	5.99	12.6	**89.7%**
J6		22.15	5.43	148.3	20.96	6.06	18.4	**87.6%**
J7		23.12	5.52	122.1	21.20	5.41	24.4	**80.0%**
J8		22.90	5.49	142.2	21.92	5.82	20.6	**85.5%**
J9		22.30	5.38	120.9	21.65	6.04	17.3	**85.7%**
J10		22.10	5.46	158.3	21.36	6.01	26.4	**83.3%**
J11		22.66	5.46	126.4	21.60	5.68	14.2	**88.8%**
J12		21.32	5.54	125.0	18.90	5.63	32.0	**74.4%**
J13		22.80	5.47	122.9	21.81	5.55	17.5	**85.8%**
J14		22.30	5.60	124.6	22.52	6.08	19.3	**84.6%**
J15		22.93	5.57	110.8	22.16	6.11	17.6	**84.2%**
J16		22.33	5.56	143.9	21.20	6.14	41.3	**71.3%**
J17		22.30	5.69	136.9	21.10	5.59	16.8	**87.7%**
K Series	***Ave***	***22.34***	***5.28***	***126.9***	***21.30***	***5.79***	***27.63***	***78%***
250 ppm H_3PO_4	**St Dev**	***0.65***	***0.21***	***14.05***	***1.02***	***0.35***	***10.61***	***10%***
Notes: pH adjusted to 6.5 with NaOH (50%) lye; 20 ppm 6195; 300 l/h at 65℃								
K2		24.12	5.22	134.6	21.40	5.46	16.4	**87.8%**
K3		21.92	5.35	116.8	21.20	6.11	21.3	**81.8%**
K4		21.80	5.46	132.9	21.13	5.64	17.3	**87.0%**
K5		22.20	5.01	126.7	21.96	5.14	34.1	**73.1%**
K6		22.96	4.90	119.2	19.70	5.25	26.1	**78.1%**
K7		22.00	4.96	122.4	21.20	5.30	18.2	**85.1%**
K8		22.20	5.06	160.2	21.40	5.63	29.5	**81.6%**
K9		21.90	5.38	111.0	21.80	5.54	26.0	**76.6%**
K11		22.44	5.28	132.9	21.10	6.27	22.3	**83.2%**
K12		22.80	5.38	111.7	21.60	5.78	21.5	**80.7%**
K13		22.60	5.37	124.1	24.01	5.72	35.2	**71.6%**
K15		22.72	5.40	149.3	20.76	6.24	18.5	**87.6%**
K16		22.08	5.36	122.1	20.10	6.40	33.0	**73.0%**
K17		21.32	5.57	130.0	20.10	6.52	39.0	**70.0%**
K18		22.10	5.55	109.3	22.01	5.87	55.9	**48.9%**
L Series	***Ave***	***22.21***	***5.38***	***108.5***	***20.83***	***5.23***	***27.66***	***74%***
0 ppm H_3PO_4	**St Dev**	***1.24***	***0.18***	***13.48***	***1.69***	***0.28***	***10.77***	***11%***
Notes: No pH Adjustments - 20 ppm 6195 on molasses at 22 Brix; 300l/h at 65℃								
L2		22.12	5.37	106.2	20.50	5.21	23.0	**78.4%**
L3		22.10	5.34	109.4	21.20	5.31	32.9	**69.9%**
L4		22.10	5.40	108.1	15.96	5.41	23.2	**78.6%**
L6		21.52	5.51	108.0	21.52	5.46	23.2	**78.5%**
L7		19.60	5.57	116.1	18.90	5.52	26.7	**77.0%**
L8		22.70	5.65	100.7	21.30	5.33	19.3	**80.9%**
L9		22.60	5.57	140.7	21.74	5.48	23.8	**83.1%**
L10		21.62	5.59	130.9	20.90	5.35	18.1	**86.2%**
L12		19.80	5.37	113.9	18.80	5.23	26.8	**76.4%**
L13		23.20	5.44	99.7	21.50	5.04	29.8	**70.1%**

Table 5.11 - Effect of Phosphoric Acid on Clarification

Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			
		Brix	pH	NTU/ Brix	Brix	pH	NTU/ Brix	% NTU Removed
L14		22.00	5.48	91.9	22.31	6.14	24.6	**73.3%**
L15		22.18	5.11	93.3	21.56	6.25	28.0	**70.0%**
L16		23.20	5.07	90.3	21.50	0.00	17.3	**80.9%**
L17		24.44	5.22	108.4	20.61	6.00	56.9	**47.5%**
L18		23.69	5.20	101.4	22.60	6.07	48.7	**52.0%**
L19		22.53	5.16	116.6	22.42	5.85	20.4	**82.5%**

It was found that the required clarity could still be obtained at 50% of the original phosphoric acid level. In continuous mode with better-controlled conditions, it might be possible to get the required clarity with no chemical pre-treatment at all. This finding has significant economic implications because the chemicals used in the pre-treatment are the single most expensive component in the invert recovery process.

In the M and N series no phosphoric acid was added but the natural pH of the molasses was adjusted to more than seven with 50% caustic soda solution before flocculation and clarification. When using the cationic 6195 flocculant, the turbidity remaining was below 24 NTU/Brix and average removal was about 83%.

Table 5.12 - Series M (Cationic flocculant) and N (Anionic flocculant)

Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			
		Brix	pH	NTU/Brix	Brix	pH	NTU/Brix	% NTU Removed
M Series	***Ave***	***22.18***	***5.44***	***118.3***	***21.84***	***7.08***	***23.82***	***80%***
0 ppm H_3PO_4	**St Dev**	***1.19***	***0.05***	***13.49***	***1.01***	***1.84***	***7.80***	***7%***
Notes: pH > 7 with NaOH - 20ppm 6195								
M2		23.40	5.50	107.9	21.85	7.24	34.2	**68.3%**
M3		21.33	5.48	140.3	20.95	7.24	29.9	**78.7%**
M4		20.56	5.41	121.2	20.90	7.16	16.0	**86.8%**
M5		22.70	5.45	114.1	22.10	7.10	20.3	**82.2%**
M6		22.90	5.37	107.9	23.38	6.68	18.7	**82.7%**
N Series	***Ave***	***22.77***	***5.36***	***109.9***	***22.41***	***6.68***	***62.21***	***43%***
0 ppm H_3PO_4	**St Dev**	***0.98***	***0.12***	***10.53***	***0.94***	***0.39***	***18.68***	***16%***
Notes: pH > 7 with NaOH - 20ppm Magnafloc LT27								
N1		23.35	5.33	110.6	23.30	6.83	66.7	**39.7%**
N2		23.37	5.36	102.6	23.29	7.21	69.1	**32.7%**
N3		22.86	5.29	123.1	22.10	7.28	93.9	**23.7%**
N4		23.21	5.34	114.3	22.20	6.74	82.9	**27.5%**
N5		21.10	5.16	113.9	22.37	6.49	33.2	**70.8%**

Table 5.12 - Series M (Cationic flocculant) and N (Anionic flocculant)

Experiment Number		Feed (diluted molasses)			Overflow (clarified molasses)			
		Brix	pH	NTU/Brix	Brix	pH	NTU/Brix	% NTU Removed
N6		23.40	5.31	124.0	23.11	6.61	49.5	**60.1%**
N7		23.83	5.48	97.7	22.48	6.27	47.2	**51.7%**
N8		22.60	5.54	93.3	20.20	6.57	63.4	**32.0%**
N9		21.20	5.47	109.8	22.60	6.08	54.2	**50.6%**

With Magnafloc, a well-known sugar industry anionic flocculant in series N the average turbidity increased to more than 60 NTU/Brix and average turbidity removal dropped to about 40%. This confirmed the selectivity of the particles to a cationic flocculant.

Good results were obtained between the G series of trials and the M series. The concentration of the molasses solution was kept close to 22.5 Brix for the duration of these trials. The result proved that the process could run without the addition of phosphoric acid as shown in trials L and M. It is felt that an economic and reliable way to operate the plant would be to add between 500 and 750-ppm phosphoric acid to the diluted molasses, and then adjusting the pH to between 6,3 and 6,7 with the addition of caustic soda lye. It is also felt that, based on the criteria set by Simpson (1996), it is obvious that flocculant 6195 is the flocculant of choice. Furthermore the selection of the E-CAT ™ as the correct design of clarifier, met the criteria set by Seifert (1987).

5.14 Conclusion

The E-Cat™ clarifier met all the requirements for molasses clarification. It is a low shear clarifier that prevents soft flocculated clusters from breaking up. There is no turbulence inside the settling chamber that can break up floc-clusters. It has a high separation efficiency and provides a good separation zone in which the solids can settle. The compaction zone offers good dewatering of the sludge leaving a thick jelly-like sludge. A suitable desweetening process for the sludge will enhance product recovery. The operating conditions for the unit have been determined and are reported below in Table 5.13. The separation efficiency of the E-CAT ™ is such that the consumption of chemicals during the pre-treatment phase can be reduced.

Table 5.13: Operating Conditions for molasses clarification on an E-Cat

Variable / Condition	Value	Reference Experiments
Feed flow of molasses (Expressed as a flux relative to the surface area of the clarifier)	4244 l/m^2h	Series A-M
H_3PO_4 addition	500 – 750 ppm	Series I & J
Feed Brix	22 – 28 Brix	Series A-M
Feed Temperature	Not below 65°C	Series A-M
Feed pH	6,5 to 7,0	Series I & J
Flocculant Type	Montan 6195	Series A-M
Flocculant make-up solution	1000 ppm	
Flocculant concentration in clarifier feed solution.	20 ppm	Series A-M

The equipment has no moveable parts, implying negligible maintenance costs. Commercial E-CAT ™ systems have a smaller footprint and need a smaller unit capacity to produce the same clarification efficiency as normal clarifiers. For the commercial plant, an E- CAT ™ system will be recommended since all the problems facing the commercial clarification of molasses have been addressed on the pilot plant.

As stated above, the recovery of sugars from the sludge should be investigated further as this will definitely enhance product recovery. Investigations into this aspect are discussed in the next chapter, Chapter 6.

Chapter 6

Sludge Desweetening during Molasses Clarification

6.1 Introduction

Recovery of 90 % clarified molasses liquor implies that 10 % of the sugars will be lost in the liquid remaining with the settled sludge. It is of economic importance to recover as much as possible of the invert sugar from the sludge. It was felt that washing the sludge and recycling the wash water containing the invert sugar (sweet-water) to the molasses dilution operation might be able to do this.

A pre-requisite is that the recovered sweet-water must be clean enough to be used through the rest of the process. Apart from the recovery of invert sugars, another advantage of the dilution of molasses with sweet-water was considered to be the possibility of conducting the pre-treatment process at a higher total solids concentration, and thereby saving on energy and plant capacity. The optimum concentration of molasses for clarification was investigated and is reported earlier, in Chapter 5.

It was therefore felt that a cost-effective method had to be established to reliably recover most of the remaining sugars from the sludge, and two methods were evaluated. Ideally, the process to be adopted must produce a relatively low turbidity in the sweet-water, give a high recovery of sugars, and it has to be time and cost efficient.

6.2. Investigation of sludge desweetening.

Fresh C-Molasses was diluted to 20 - 25 Brix solutions. Phosphoric acid was added at a ratio of 3.75 g/kg Brix. Depending on other trials in progress this mixture was left agitating at 60℃ for a few minutes or for up to 8-10 hours. After this time, the mixture was neutralised with NaOH added in the ratio 5.0 g/kg Brix. The flocculant was dosed at a 20-ppm level. Settling rate was measured and logged. After the settling of the sludge had stabilised, the clear liquid was siphoned from the sludge as described previously. For all these trials flocculant 6195 was used.

The sludge remaining in the tank was then diluted with water, quickly stirred and allowed to settle. Two methods to recover sweet-water were compared. In the first investigation a disk bowl centrifuge was used to separate the sludge and sweet-water. This process involved feeding the sludge mixture with a Mono pump at a rate of 600 l/h into the Disk Bowl Centrifuge. The centrifuge was the same used in the clarification trials discussed in Chapter 5. The bowl was partially cleaned every five minutes and completely cleaned after every 15 minutes. The Mono pump was used to reduce shear and turbulence, thereby minimising disintegration of coagulated sludge particles.

In the second method, sweet water was recovered by siphoning the clean liquid from the sediment sludge layer in the tank. The same method used to dilute the sludge as above was used but the mixture was left to settle by gravity. During some trials the settling rate were measured using a 1000 cc measuring flask. The results are listed in the results and discussion section below.

Stirring caused the flocculated sludge to disintegrate. A low shear mixer would be required to dilute the sludge on an industrial scale. To coagulate the fine flocculated particles, it was sometimes necessary to add a small amount of so-called floc-builder. Floc-builder is a flocculant of opposite polarity to the flocculant in present use. The floc-builder used in the trials is an anionic flocculant from Floccotan, referenced to as K109.

The sweet-water with clarity of less than 10 NTU/Brix was used as part of the dilution water for subsequent molasses dilution. This resulted in it being possible to operate the clarification process at higher concentration.

6.3. Results and Discussion of the two recovery processes

6.3.1. Centrifugal separation of molasses sludge and sweet-water

The results of the separation trial are listed in Table 6.1. This consisted of a single trial on the Westfalia disk-bowl centrifuge, which was operated at one third of its capacity (i.e. 1300 l/h) with sludge being removed at the normal five minute intervals.

Table 6.1: Results of centrifugal separation of sweet-water and sludge.

Trial Number	Siphoned Juice Brix	Siphoned Juice Clarity NTU/Brix	Sweet-water Brix	Sweet-water Clarity NTU/Brix
99-22	22.18	22.99	16.1	467.7

The resulting sweet-water from the above trial was not considered clear enough for further trials. It was evident from visual observation of the recovered liquor that the floc particles had broken apart in the centrifuge and therefore ended up in the fluid stream. The feed product met the specification set by the manufacturer regarding sludge loading; the machine was thus not overloaded. The use of such a low flowrate should, in theory, favour the separation of heavy sludge particles from the mother liquor. A jug test (as described in Chapter 3) on the sludge mixture fed to the centrifuge showed that the suspended sludge particles coagulated and settled well. This was however not evident in the mother liquor obtained after the centrifuge.

The results obtained during the above observation and jug test prompted the further investigation into the settling behaviour of the diluted sludge, and the separation test on the centrifuge was subsequently abandoned.

6.3.2. Settling clarification of molasses sludge and sweet-water

From the earlier success with settling a decision was made to evaluate the settling performance of the sludge in a separate secondary clarifier. The E-Cat commercial clarifier was not suitable for these tests due to its large size. For these trials the clear molasses was siphoned from the settled sludge blanket in the tank. Water was added to the sludge remaining after the siphoning of the clear molasses was complete. This water was gently mixed with the sludge blanket and the mixture was allowed to settle. The clear liquid above the new sludge blanket was siphoned and the results of these separation trials are listed below in Table 6.2. To some of the trials a so-called “floc-builder” was added.

The settling time for the sludge was longer than the time required for settling molasses - as was reported previously in Chapter 5. This could possibly be attributed to some floc particles breaking up whilst stirring. Even at a slow settling rate the equipment and operating cost of a settling clarifier would still be considerably less than that of other methods of treatment.

The sludge did appear to settle more or less consistently after one hour. A turbidity of below 20 NTU/Brix is acceptable to add as part of the water to dilute the molasses and this was achieved in most of the trails.

Table 6.2: Results of the siphoning separation of sweet-water and sludge. (F/B indicates the use of floc-builder)

Trial Number	Siphoned Juice Brix	Siphoned Juice Clarity NTU/Brix	Sweet-water Brix	Sweet-water Clarity NTU/Brix	Settling Rate Min.
99-24	19.5	0.31	18.7	0.0	N/d
99-27 (F/B)	17.1	14.56	9.4	9.57	N/d
99-28	18.0	40.11	11.4	5.44	N/d
99-33	21.1	12.32	8.1	2.59	5
99-34	22.3	3.09	15.3	0.0	60
99-35	19.4	3.04	10.8	0.0	60
99-36	25.9	19.77	10.8	11.35	55
99-37	20.6	13.69	14.0	0.0	60
99-38 (F/B)	22.2	5.09	11.4	6.32	55
99-40	19.1	19.37	10.0	32.2	55
99-41	18.9	6.14	11.4	52.98	60
99-43	22.3	10.63	7.38	20.6	60
99-45	26.2	9.09	5.1	18.5	53
99-46	31.4	3.79	7.4	2.94	35
99-49	26.6	6.73	9.6	0	60

For some of the trials the liquid after settling was so clear that no reliable reading could be obtained on the Turbiquant, and these were indicated with a zero value in the table above. In some trials the settling was complete in less than 60 minutes and one trial, number 99-33, had an exceptional good clarity and settled very quickly. This result could not be repeated in subsequent trials and will therefore not be considered in the final design. The reason for the higher values of trials 99-40 and 9-41 could not be determined. Trial 99-40 had settled completely and the sweet water was siphoned after 55 minutes. The sludge blanket of trial 99-41 was stable after 60 minutes and no additional settling was observed. In both these trials the “clear” liquid was visibly turbid. A most likely reason for this could be that the sludge was excessively damaged during mixing and that the flocculated particles did not conglomerate again.

The use of a belt press filter can be investigated to act as a final step to de-water the sludge. However, further dilution will only cause a lot of low concentration waters and a sludge that is still wet.

The above results indicate that a settling type recovery system can be used successfully to recover sugars from the sludge.

6.4. Conclusion

The settling clarification of diluted molasses sludge proved to be the more feasible method compared to centrifugation. It produced a clear sweet-water, which could be used as part of the water for the dilution of molasses.

By applying this method one can maintain a higher solids concentration in the molasses solution phase during the clarification stage, which in turn produces an acceptable clarity upstream in the process.

A desweetening clarifier could be installed close to the mud outlet of the primary clarifier. Adequate turbulence must be induced during the dilution to allow proper diffusion of sugars from the sludge, but with a low shear to prevent breakage of the flocculant. In this way it is felt that a molasses treatment process has been developed to give a product suitable for separation on a simulated moving bed (SMB) system, at an acceptable recovery and efficiency.

An important operation that has not received any attention is the inversion of the sucrose to glucose and fructose. This investigation will be discussed in the following chapter.

Chapter 7

Inversion of Sugar in Molasses

7.1. Introduction

Commercialisation of the molasses desugarisation process will involve commercial inversion of sucrose. Sucrose, commonly known as table sugar, is a disaccharide composed of an alpha-D-glucose molecule and a beta-D-fructose molecule linked by an alpha-1,4-glycosidic bond. When this bond is broken in a hydrolysis reaction, an equimolar mixture of glucose and fructose is generated. This mixture of monosaccharides is called invert sugar, which is derived from the fact that sucrose rotates the plane of polarised light to the right i.e., dextrorotatory, +66.5º, whereas the hydrolysis products rotate the plane of polarised light to the left i.e., laevorotatory, -20º for the mixture (+52.5º for D(+)-glucose and -92º for D(-)-fructose). Other common disaccharides are maltose and lactose.

Sucrose can be hydrolysed in the presence of an enzyme called invertase or sucrase. The inversion reaction of sucrose to fructose and glucose can be represented as follows:

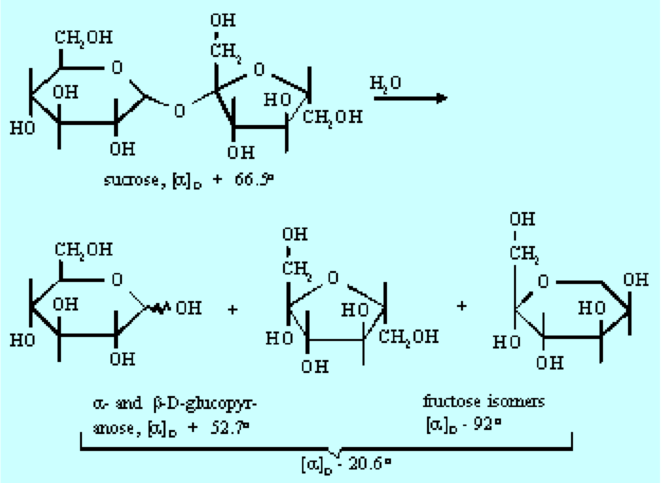


Figure 7.1: The inversion of Sucrose

This reaction is of interest historically, in that Michaelis and Menten in 1913 made the first satisfactory mathematical analysis of the effect of substrate on the reaction velocity of this reaction. In order to determine the parameters of the Michaelis-Menten Equation, (eq. 7.1) one needs to study the inversion at a number of substrate (sucrose) concentrations:

$$\text{Rate} = V_{max}[S]/(K_M+[S]) \qquad (7.1)$$

The rate is the measured change in the number of moles of sucrose divided by the time interval of the measurement, V_{max} is the maximum velocity of the reaction for a specified enzyme concentration, [S] is the substrate concentration, and K_M is the Michaelis constant that corresponds to the dissociation equilibrium constant for the enzyme substrate complex.

Equation 7.1 may be rearranged to give:

$$1/\text{Rate} = 1/V_{max} + K_M/(V_{max}[S]) \qquad (7.2)$$

Thus a plot of 1/rate vs. 1/[S] should give a straight line with a slope of K_M/V_{max} and an intercept of $1/V_{max}$ on the y-axis and an intercept of $1/K_M$ on the x-axis. This was not studied in detail during the pilot plant evaluation since adequate data is available for the process from the enzyme and yeast suppliers. Some kinetics however were reported and is described below.

The aim of the work on the pilot plant was to establish a condition with acceptable inversion (preferably as close to 100% as possible) and good economic viability – using the least amount of invertase possible. Some further optimisation can be done and the kinetic parameters can be verified in due course.

Sucrose can also be inverted with acids. Wilhelmy (Germany) studied the rate of inversion of sucrose (hydrolysis into D-(+)-glucose and D-(-)-fructose in the presence of an acid) in 1850 and found it to be proportional to the concentrations of both the sugar and the acid.

A quick inversion with acid will require a low pH requiring a higher use of acid and later, base to neutralize the mixture. Molasses has a high buffer capacity and to achieve this very low pH requires a large amount of acid. Bernhardt (2000) and co-workers at the SMRI investigated the use of sulphuric acid in order to precipitate calcium and magnesium from the molasses and simultaneously invert the molasses. The application of this method and the precipitation of these salts were discussed previously in Chapter 5. It was also found that the sulphuric acid dehydrates the sugars in the molasses leaving a very dark, almost black solution to be neutralised.

Neutralisation with caustic soda was found to be violent. Another disadvantage is that acid inversion also increases the salt content that has to be removed during chromatography. It was therefore decided to abandon the investigation into acid inversion and only investigate the other alternative – enzymes.

During the trials at the pilot plant two methods of inversion were investigated:

- Inversion with pure invertase enzyme, and
- Inversion with high invertase yeast.

Since the optimum pH for enzyme activity is around pH = 4,5 to pH = 5,5 the inversion step was “moved” from the initial sequence to after the phosphatation step, before clarification. The advantage of moving the inversion forward is threefold:

- The molasses is already slightly acidic due to the phosphoric acid treatment to assist clarification and precipitate calcium.
- There is no need for an additional neutralisation of the inverted molasses, and lastly,
- Suspended cell material or enzyme can be removed with the sludge eliminating the need for a further filtration step.

7.2. Invertase

The official name for invertase is beta-fructofuranosidase (EC3.2.1.26), which implies that the reaction catalysed by this enzyme is the hydrolysis of the terminal nonreducing beta-fructofuranoside residues in beta-fructofuranosides. Note that alpha-D-glucosidase, which splits off a terminal glucose unit, can also catalyse this reaction. Sucrose can be hydrolysed relatively easily; the reaction proceeds in an acidic environment without the aid of invertase.

Invertase is mainly used in the food (confectionery) industry where fructose is preferred over sucrose because it is sweeter and does not crystallise as easily. Fructose also enhances the “mouth feel” of food products. However, the use of invertase is rather limited because another enzyme, glucose isomerase, can be used to convert glucose to fructose at a lower cost. This is enzyme process is used extensively in the American High Fructose Corn Syrup industry. For health and taste reasons, its use in the food industry requires that invertase be highly purified. A wide range of micro-organisms produces invertase and can, thus, utilise sucrose as a nutrient. Commercially, invertase is biosynthesised chiefly by yeast strains of *Saccharomyces cerevisiae* or *Saccharomyces carlsbergensis*. Even within the same yeast culture, invertase exists in more than one form. For example, the intracellular invertase has a molecular weight of 135,000 Daltons, whereas the extra cellular variety has a molecular weight of 270,000 Daltons.

Contrary to most other enzymes, invertase exhibits relatively high activity over a broad range of pH (3.5-5.5), with the optimum pH near 4.5. The enzyme activity reaches a maximum at about 55ºC. The Michaelis-Menten values of various enzymes vary widely, but for most enzymes Km is constant between 2 millimole and 5 millimole. The Michaelis-Menten value for the free enzyme is typically around 30 millimole. This can be used as an approximation for calculations if known values are not available.

Although the mechanism of inhibition of invertase by heavy metal ions seems to differ, there is no question that invertase is strongly inhibited by these metal ions. For example, Ag^+ ions attach to the histidine side chains of the invertase molecule and render it inactive. Therefore, a softening stage may be an advantage prior to inversion.

7.3. Gist Brocades Invertase (Maxinvert)

Maxinvert, the trade name for the invertase enzyme from the Gist Brocades Company, is provided either as a liquid or in a crystalline form. The crystalline form of the enzyme was preferred for the pilot research project. The enzyme is stable and the product is more “robust” than the liquid. The enzyme in the powder form can survive the typical conditions that are experienced in the pilot plant.

The inversion results from two enzyme trials done in the laboratory at Malelane are presented in the charts below. The enzyme added was measured in International Units (IU or SU). One international unit of activity is defined as the amount of enzyme needed to hydrolyse 1 µmole of sucrose to invert sugar per minute at pH=4.5 and at 55ºC.

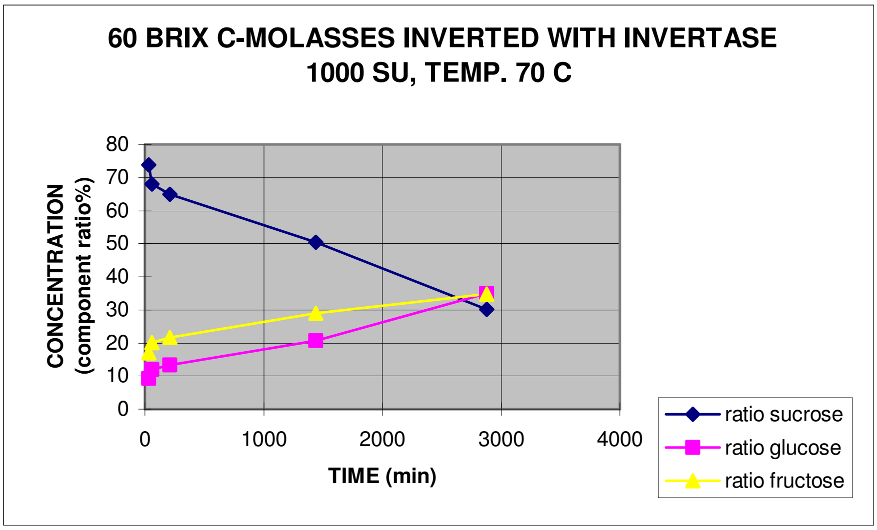


Figure 7.2: 60 Brix C-Molasses Inverted With Invertase: 1000 SU

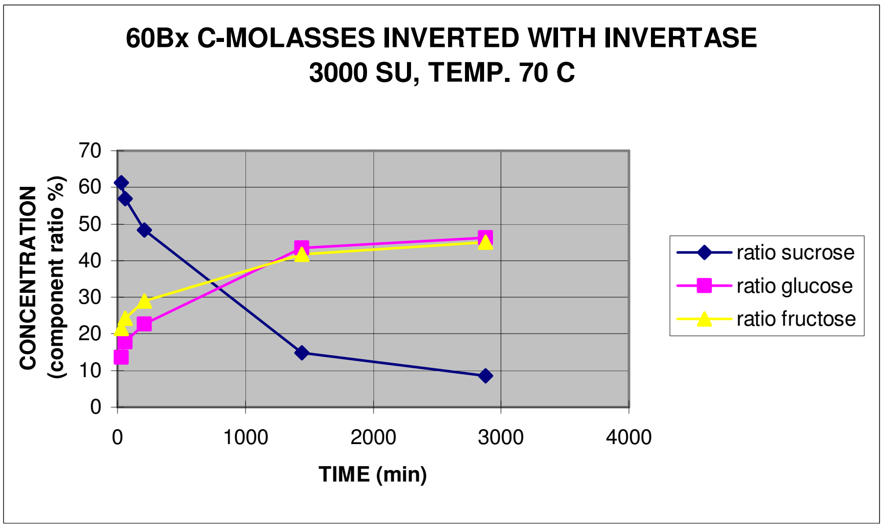
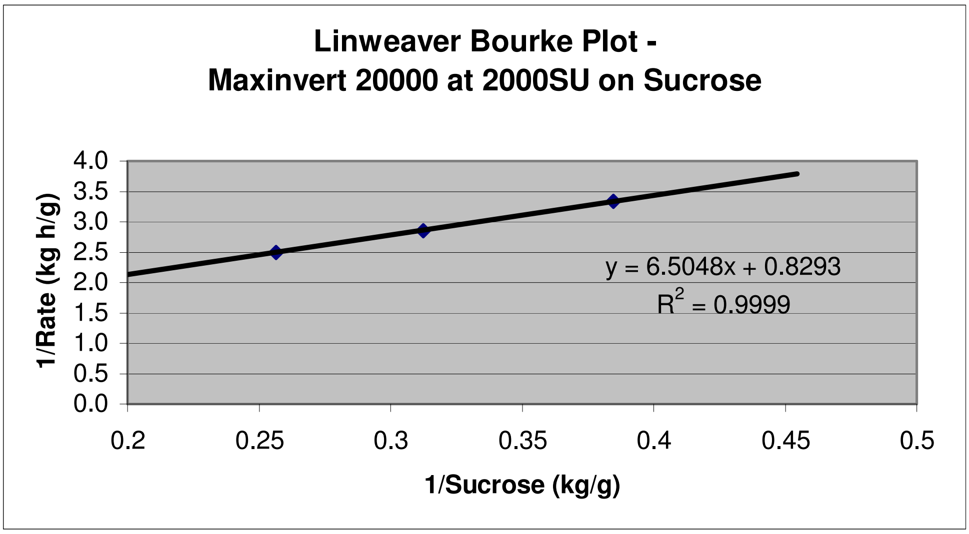


Figure 7.3: 60 Brix C-Molasses Inverted With Invertase: 3000 SU

The following Linweaver Bourke Plot was made from data supplied by Gist Brocades for their Maxinvert 20000 on sucrose.



1/vmax =	0.8293	vmax =	1.205836
Km/vmax =	6.5048	Km =	7.843724

Figure 7.4: Linweaver Bourke Plot - Maxinvert 20000 at 2000SU on Sucrose

Using the results obtained in the laboratory and the Gist Brocades data, the inversion of sucrose in the molasses on the pilot plant was standardized to the following conditions:

Dry substance: 50%
pH: 4,5 – 5,0
Temperature: 60°C
Dosage of enzyme: 2000 SU = 0,1 g enzyme / kg sucrose.

For a C-molasses with the typical analysis:

Brix	84.64
Sucrose Purity	35.05

590 litres of molasses is diluted with water to 1000 litres to yield a 50 Brix solution, which contains 236 kg sucrose and, for inversion based on the data above, requires 23,6 g of enzyme. The enzyme is added as a powder to the molasses. On the pilot plant the molasses was inverted completely after 12 hours.

7.4 High Invertase Yeast

High invertase yeast (HIY) is a special strain of yeast, typically from the genus *Saccharomyces*, known for its ability to produce high concentrations of invertase enzyme. The yeast can be used for inversion of sucrose without first incurring the high cost of recovery of the enzyme. To prevent the yeast from fermenting the sugar or molasses syrup, it is essential to maintain a high sugar concentration and slightly elevated temperature. The high sugar concentration upsets the osmotic pressure of the yeast causing the cell to burst (autolyse). Autolysis of the cell sets invertase contained in the yeast cell free. This further enhances the invertase activity of the HIY.

This is a relatively inexpensive method to obtain and use invertase for inversion. In an application where HIY is intended, care must be taken to ensure that the cellular material that is added does not have a detrimental effect on the process.

During the reporting period, HIY was never used on already clarified molasses. The HIY was always added to the tanks prior to clarification, as described in the discussion for the free enzyme.

The molasses solution in the inversion reactor under investigation was prepared as follows:

Desired Brix: 50-55
Desired Temperature: 55 ± 3°C
Desired pH 4,5 – 6,5
Recommended Concentration HIY: 2,6-kg slash/(t Sucrose)

Using the same base data as in the Maxinvert example above, 590 litres of Komati C-molasses was diluted to 50 Brix and has 236 kg of sucrose in solution. To this 614 g slash was added. The yeast was made into slurry in about 2-3 litres of water (<50°C) before adding the slurry to the molasses. The inversion results from these yeast trials are presented in the tables and charts below.

Table 7.1: Inversion Trial 1 with 620g Yeast in 1 m^3 of molasses

INVERSION 1

TANK 31A YEAST: 620 g

RATIO % 1m^3 of Molasses 50 Brix

SAMPLE	FEED						
HOURS	0	1.5	3	4.5	6	8.00	12
SUCROSE	68.84	44.22	35.76	8.72	7.53	2.32	1.51
GLUCOSE	9.26	23.13	27.60	42.21	41.79	42.74	44.97
FRUCTOSE	12.76	26.03	29.26	44.55	44.67	46.08	49.27
UNKNOWNS	9.14	6.63	7.38	4.52	6.02	8.86	4.24
TOTAL	100	100	100	100	100	100	100

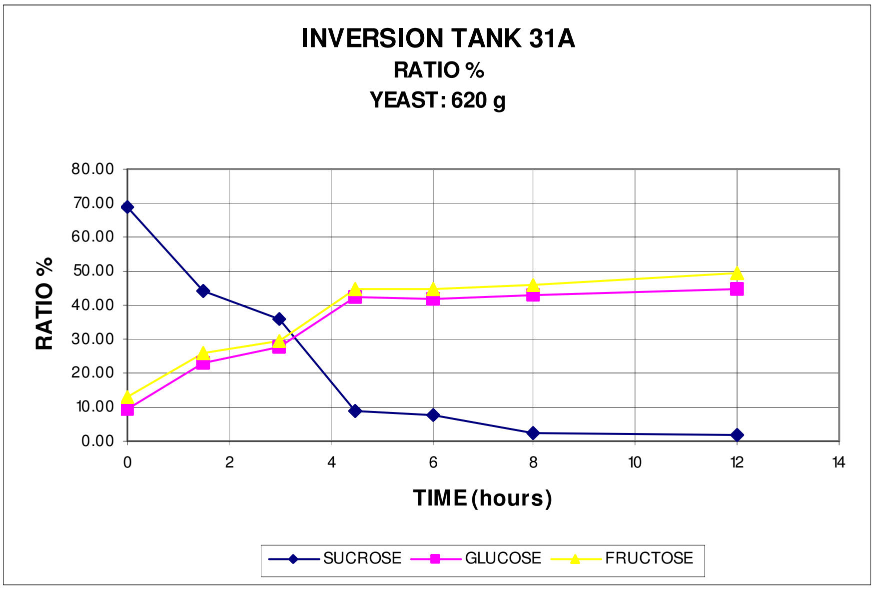


Figure 7.5: Inversion with 620 g yeast

Table 7.2: Inversion Trial 2 with 920g Yeast in 1 m^3 of molasses

INVERSION 2

TANK 31B YEAST: 920 g

RATIO % 1m^3 of Molasses 50 Brix

SAMPLE	FEED					
HOURS	0	1.5	3	4.5	6	12
SUCROSE	68.84	19.30	14.91	1.10	1.22	2.94
GLUCOSE	9.26	35.07	38.79	44.77	45.79	44.45
FRUCTOSE	12.76	37.68	40.92	48.75	49.38	48.95
UNKNOWNS	9.14	7.95	5.38	5.37	3.61	3.65
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00

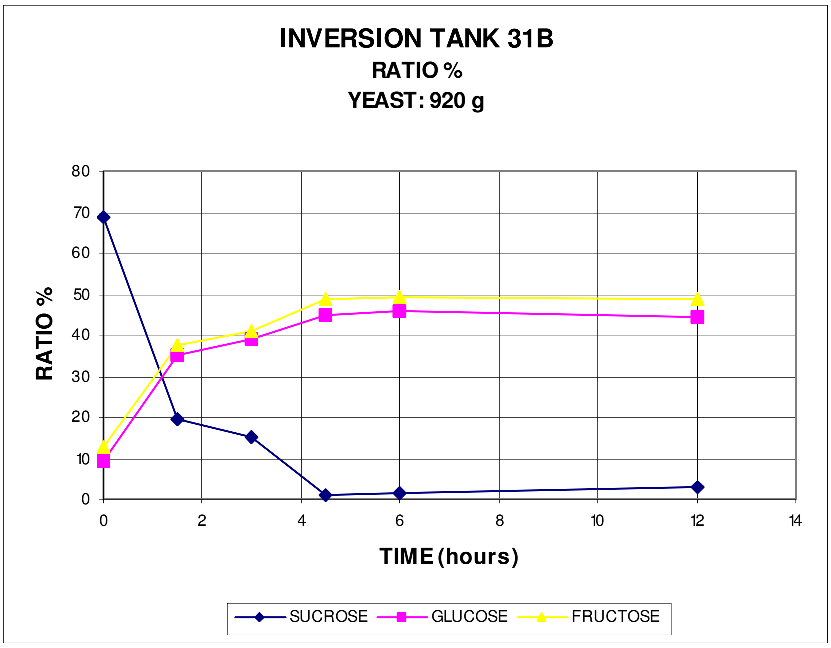


Figure 7.6: Inversion with 920 g yeast

The above results show that suitable inversion is possible in 12 hours by using HIY at a concentration of 2,6 kg/t sucrose.

As expected, the results of the second trial show that a higher concentration of invertase results in a quicker inversion. Using the results of the first experiment the following kinetic data was calculated.

Table 7.3: Application of Yeast to Sucrose Inversion in Molasses

INVERSION 1

TANK 31A YEAST: 620 g

Michaelis Menten Kinetics

Time (t)	0	1.5	3	6	12
Dt		1.5	1.5	3	6
Sucrose ppm (s)	2457.304	1630.338	1316.724	311.274	64.079
Glucose ppm	330.510	852.772	1015.980	1727.965	1904.993
Fructose ppm	455.649	959.663	1077.248	1846.844	2086.880
Sucrose inverted ppm (Ds)	0.000	826.966	313.614	1005.450	247.195
Rate (Ds/Dt)		551.31067	209.076	335.15	41.199167
1/Rate		1.8139E-03	4.7829E-03	2.9837E-03	2.4272E-02
1/Sucrose		6.1337E-04	7.5946E-04	3.2126E-03	1.5606E-02

Now: Plot 1/rate (on y-axis) vs. 1/sucrose (on x-axis)

1/Rate		1.8139E-03	4.7829E-03	4.2855E-02	1.5989E-01
1/Sucrose		6.1337E-04	7.5946E-04	3.2126E-03	1.5606E-02

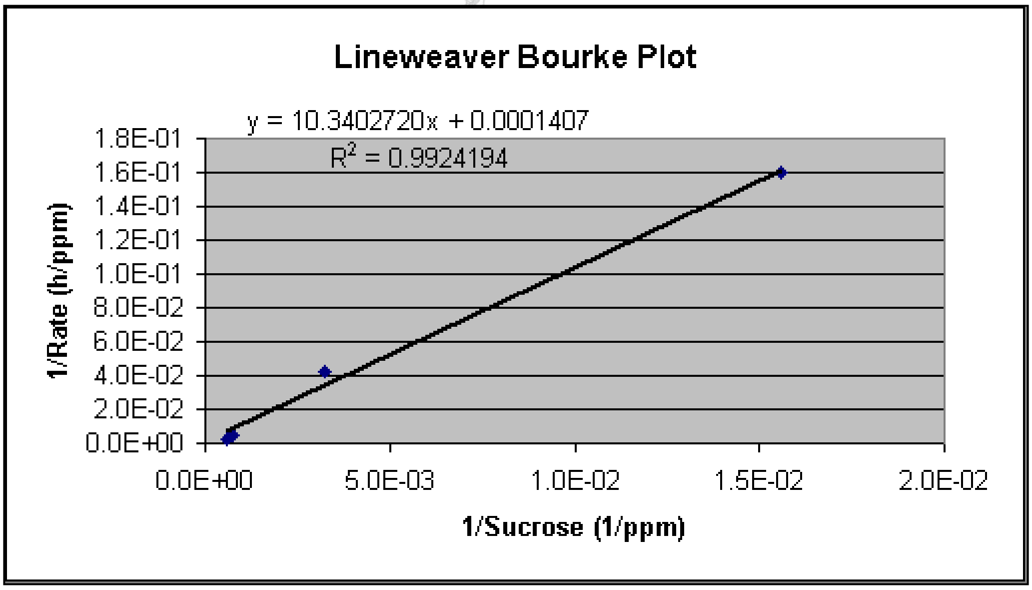


Figure 7.7: Lineweaver Bourke Plot of HIY kinetics on molasses

From the plot: 1/vmax = 0.0001407 Therefore vmax = 7108.331 ppm/h

Km/Vmax = 10.3400 and Km= 73500.142

These trials were the only trials where the inversion rate was studied against time. The priority for the process lay in establishing suitable products to be separated on ion-exclusion chromatography. For all the further trails on molasses, only a final sample was analysed to verify that all the sucrose had been fully inverted.

7.5 Conclusion

Limited work was done on determining the kinetics of the inversion of sucrose in molasses. The inversion step now occurs before neutralisation of the phosphatation step, before clarification, resulting in a process saving. High Invertase Yeast produces an equivalent product to that obtained by using pure invertase enzyme, resulting in a significant cost saving.

The Simulated Moving Bed (SMB)-system that is responsible for the separation of salts and invert sugar will now be discussed.

Chapter 8

Chromatographic Separation of Clarified Molasses

8.1. Introduction

Chromatography refers to a very wide spectrum of technologies that are used in the food, pharmaceutical and chemical process industries to separate chemical compounds. In all chromatography processes, separation is accomplished by manipulating the varying affinities of the compounds to be separated, to the stationary phase. A chromatographic separation process, making use of the ion exclusion principle, was adopted for the separation of sugars from salts in the desugarisation of molasses.

A pilot plant study was conducted to investigate the separation of sugar cane molasses. In order to specify the operating conditions of a commercial plant the following objectives were pursued:

a) No dry solids in the basic loop - this would ensure that there would be no build-up of any component in the system and all the solids would be leaving the unit in the extract and raffinate streams.

b) Optimal invert sugar recovery - the optimum recovery of invert sugar in the extract would enhance the process economics. Sugars in the raffinate are lost to the effluent treatment system or sold with the raffinate as liquid fertiliser.

c) Reach the desired purity in the extract - in order to reduce the downstream treatment operating and capital costs, a certain purity in the extract had to be achieved.

d) Optimise the water consumption - water is used as the eluent through the process. Optimum water use would result in a higher concentration of products, which implies reduced steam consumption in the downstream evaporation and would also result in lower operating cost for the separation unit.

Charton and Nicoud (1995) presented a design procedure for a simulated moving bed system based on Langmuir isotherms. Peacock (1996) described the process of desugarisation of refinery molasses on a resin in the sodium form in a batch column.

Hongisto (1977, Part 1) described the Donnan membrane effect, which drives the ion exclusion separation. He also identified separation variables and operating parameters for a beet molasses separator. In the paper he discussed the use of the "critical velocity" parameter to avoid a "fingering and tailing" phenomena in the separation system. Schoenrock *et.al.* (1983) described a slightly modified simulated moving bed system that they used. Kearney (2002) described chromatography as a mature and reliable technology. He states that more than 90% of the beet molasses produced in the United States is treated in some manner via chromatography.

In work by Saska and co workers (1992) using DOW resin in the potassium form they proposed a method to integrate the pulse data obtained from a laboratory test to determine mean and standard deviation variables of the separating performance of the system. Paillat and Cotillon (2000) published a review of the application of chromatography in the sugar industry. They discussed the requirements and various systems in practice today such as: simulated moving bed (SMB), multistage SMB, sequential simulated moving bed (SSMB) and sequential multiprofile systems.

The ion exclusion chromatography of molasses was, in essence, a separation of non-ionic and ionised components, such as sugars and the ash components respectively. In the present case all the sucrose had been inverted to glucose and fructose. These two monosaccharides have similar molecular weights and both have a neutral ionic charge. For separating purposes they can be regarded as one component - invert sugar.

The ion exclusion resin used as a stationary phase is a cationic resin in the monovalent potassium form. To explain the process, one needs to recall that similar charges repel each other and opposite charges attract each other. When the molasses solution flows through the resin bed, the cations in the solution are repelled by the cations on the resin. The cations are *excluded* from the resin body and are eluted first from the column. Large molecules such as colour bodies, high molecular weight gums and polysaccharides are too big to enter the pores of the resin and are likewise "excluded" with the ash components. The small neutral sugar molecules however, move through the interspatial channels between the resin beads and are diffused into and out of the interstitial pores on the resin beads. This retardation effect results in the sugars being eluted much slower than the cations and anions from the resin.

Successful operation of a chromatography plant relies on the “critical value of separation” (Cv). This is the volume of eluent needed for elution of the component from the resin. Critical values are related to the affinity coefficient and can be calculated for the various components by the following relationship[§]:

$$Cv = \varepsilon + (1-\varepsilon)\,K \qquad (8.1)$$

Where

ε = Void volume of the resin (approx. 0.38)

K = Affinity Coefficient of the resin or equilibrium coefficient

= (Concentration in resin) / (Concentration in solution)

The operating conditions for a plant are typically based on setting the various separation zone flows in relation to the critical value.

8.2. Pilot Plant Installation

The pilot plant consists of eight columns, connected in series and filled with 75 litres of resin each. Six manifolds are used to connect the columns. They are for Molasses, Water, 'Column to Column', Extract, Raffinate and Loop. Various resin manufacturers produce resins dedicated to chromatography. The performances of the resins vary, mainly due to the functionality of the resin. The performance also differs between different products of the same functionality. The separation of invert sugar and salt has been well defined by various technology suppliers, mainly from experience in the American corn milling and international beet sugar industries.

A pulse test is a laboratory batch separation test done to determine the separation performance of a resin. The results of such a test proved Amberlite 1320K from Rohm & Haas to be the most effective resin for the separation of invert sugar from salt in the present case. Based on this result, it was decided to use this resin in the pilot plant and evaluate its performance on a larger scale.

[§] Personal correspondence: Marc-Andre Theoleyre R&D Manager Applexion; Nov 2000

Part of the product specification for a typical chromatography resin is listed in Table 8.1 below:

Table 8.1: Characteristics of a typical Chromatography Resin. (Rohm and Haas Amberlite 1320K, product Data Sheet)

Polymer Matrix Structure	Styrene-divinylbenzene
Functional Groups	Sulphonates
Physical Appearance	Spheres
Whole bead count	>98%
Max Operating Temperature	120℃
Bulk Density	Approx. 870 g/L
Mean size	300 – 340 µm
Uniformity Coefficient	<1.15

The inputs into the plant were eluent water and molasses. The outlets were for extract and raffinate. The water, molasses, loop, extract and raffinate flows were measured using Micro Motion mass flowmeters. Feed streams and the loop flow were pumped with Tuthill gear pumps –fitted with Altivar motor drives. Pneumatic ball valves were used to channel the flow into the correct manifold. The columns were operated in down-flow. Jumo PID controllers were used to control flowrates and the raffinate backpressure. A Siemens PLC controlled the manifold valves, changing them at the appropriate step time, and also provided the run signals to the pump motor controllers.

A detailed PID is attached in Appendix D. A simplified block diagram is presented in Figure 8.1 to explain the principle of operation.

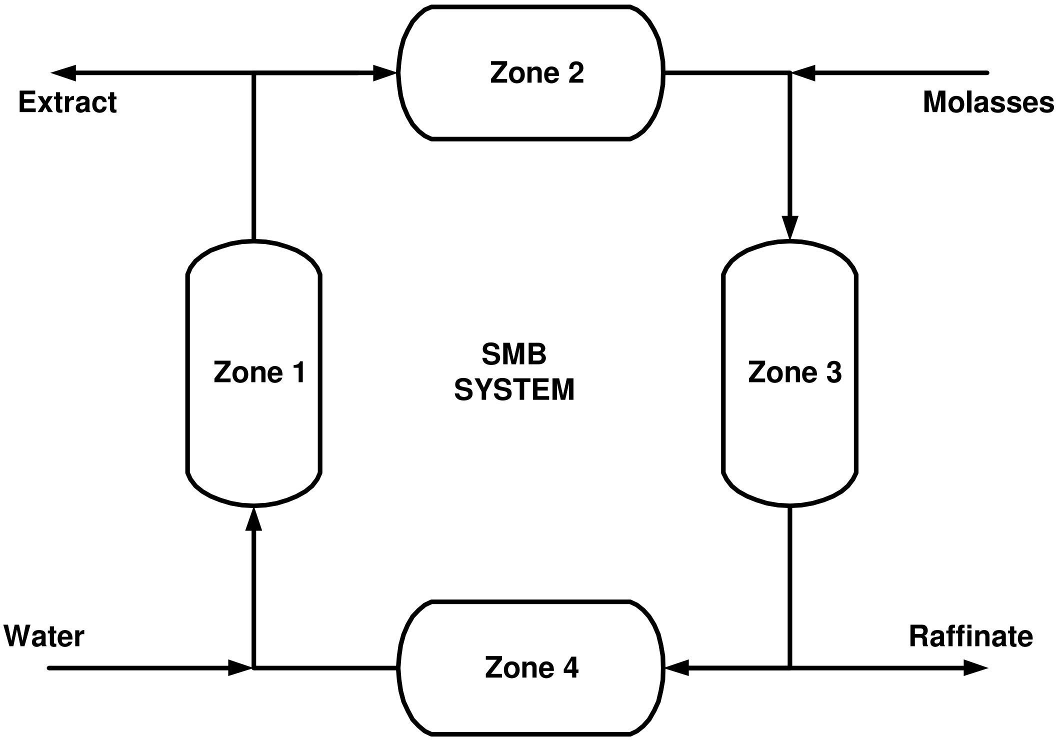


Figure 8.1: A schematic representation of the separation zones in a simulated moving bed system.

The eight columns were divided into four virtual separation zones. Zone 1 was the region between the water inlet to the extract outlet (2 columns). Zone 2 stretched from the extract outlet to the feed (molasses) inlet (2 columns). Zone 3 started at the feed inlet and ended at the raffinate outlet (3 columns). The fourth zone completed the loop from the raffinate outlet to the water inlet (1 column). This configuration is often referred to as 2/2/3/1. Other configurations were possible and the selection of the configuration depended on the separation profile required. Reprogramming the PLC on the unit could change this configuration. Table 8.2 lists the purpose of each of the different zones.

Table 8.2: The purpose of each of the zones in the SMB**:**

	Zone 1	**Zone 2**	**Zone 3**	**Zone 4**
Salts	Elution	Elution	Elution	Retention
Sugars	Elution	Retention	Retention	Retention

The reader is referred to the publications of Stolz and Bekker (2001) and Paillat and Cotillon (2000) for a further discussion on the separation zones and the interrelationship between separation variables.

Based on the separation experiments in France before the commissioning of the pilot plant, Applexion recommended the following flowrates:

Molasses Feed:	14 l/h
Water:	63 l/h
Extract (Sugar):	21 l/h
Raffinate (Salts):	56 l/h
Loop:	78 l/h
Step time:	1326 seconds.

The bed volumes (BV's) for each separation zone can be calculated[♥] from the flow rates, step time and resin volume as follows:

$$BV1 = \frac{(Q_W + Q_L) x StepTime}{3600 x RV} \tag{8.2}$$

$$BV2 = \frac{(Q_W + Q_L - Q_E) x StepTime}{3600 x RV} \tag{8.3}$$

$$BV3 = \frac{(Q_W + Q_L - Q_E + Q_F) x StepTime}{3600 x RV} \tag{8.4}$$

$$BV4 = \frac{(Q_L) x StepTime}{3600 x RV} \tag{8.5}$$

Where: Q_W = Water flow into the system [Volume/h],
Q_L = Loop flow in the system [Volume/h],
Q_E = Extract flow out of the system [Volume/h],
Q_F = Feed (molasses) flow into the system [Volume/h],
StepTime = Time to complete one step [seconds],
RV = Resin Volume of one column [Volume].

The value of the calculated separation bed volume is dimensionless and can be used directly for scaling purposes. Two systems with similar resin, operating on the same feed material and having the same bed volumes of separation will produce a similar product, irrespective of the physical size of the columns.

[♥] Heckly, S. (1998); Chromatographic Separation Unit Handbook, 97.10.3108.PR09.Rev.0

The range of bed volumes (BV's) for each zone specified from laboratory tests in France by Applexion and those achieved on the pilot plant using the Applexion setpoints are reported in Table 8.3.

Table 8.3 Comparison of specified and achieved bed volumes.

Setpoints	Specified	Achieved
BV1	> 0.6900	0.692
BV2	= 0.5875	0.580
BV3	= 0.6564	0.648
BV4	< 0.3830	0.383

The pilot plant was commissioned with the above setpoint. For the initial trials the plant was operating at half its capacity. The initial objectives of the trials were to reproduce the separation performance of the initial pilot plant work in France. Thereafter a series of adjustments were to be made to optimise the performance of the unit to meet all four of the objectives stated above. The optimum operating point would then be the initial setpoint for the commercial plant.

Figure 8.2: The Chromatography Pilot Plant. The control panel is visible in front and between the columns some of the manifold piping is visible.

During the course of these initial trials (The A-series) a lot of effort was put into the centrifugal clarification of molasses. From these trials only a few succeeded in producing a molasses clear enough to be used in the chromatography plant. In order to operate as long as possible with the available molasses, the plant was operated at lower flowrates than that originally specified. The step time was also adjusted to allow the bed volumes of each zone to remain valid and within the range specified.

8.3. Results of the A, B and C-series of trials

The averages of the results from the A- and B-series of trials are listed in Table 8.4. In order to obtain a better sugar recovery the set point on the extract in the Series A trials was adjusted. This was referenced as the B-series of trials. The effect can be seen as a change in BV values.

Table 8.4: The results from the A-series of trials with the original setpoint and the B-series with the adjusted setpoint.

	Original Setpoints - A Series -	**Adjusted Setpoints - B Series -**
BV1	0.692	0.692
BV2	0.589	0.580
BV3	0.658	0.648
BV4	0.383	0.383
Recovery		
Salt/Extract %	0.050	0.033
Tot Invert/Extract %	89.12	85.67
Salt/Raffinate %	99.95	99.97
Tot Invert/Raffinate %	10.88	14.33
Extract		
Brix	16.2	19.5
Salts %	1.13	0.53
Sucrose %	0.00	0.49
Glucose %	12.83	27.88
Fructose %	86.02	71.23
Tot Invert %	98.85	99.11
Conductivity µS/cm	1056	1043
Raffinate		
Brix	6.9	10.0
Salts %	72.11	52.58
Sucrose %	4.07	14.19
Glucose %	20.84	33.22
Fructose %	4.27	0.0
Tot Invert %	25.11	33.22
Conductivity µS/cm	18915	22990
Loop		
Brix	0.52	0.65
Salts %	71.41	68.61
Sucrose %	0.00	0.00
Glucose %	8.53	11.62
Fructose %	19.97	19.77

	Original Setpoints - A Series -	**Adjusted Setpoints - B Series -**
Tot Invert %	28.50	31.39
Conductivity µS/cm	863.7	947.0
Feed		
Brix	60.8	60.8
Salts %	21.25	21.25
Sucrose %	6.46	6.46
Glucose %	35.38	35.38
Fructose %	36.89	36.89
Tot Invert %	72.27	72.27
Conductivity µS/cm	9763	9763

The results of the change can be seen in the above table and are immediately evident by the higher brix of the extract stream. This was caused by an increase in the glucose concentration in the extract. The shift in the cut-off of the curve is also evident in the fact that no fructose is detected in the raffinate - this indicates that the sugar is better retained by the system while the salt is eluted from the system.

The data for the adjusted setpoint is for one cycle only. The unit approaches equilibrium after three full cycles (slightly longer than eight hours) after the setpoint change, when running in full production. This results in a time delay of more than sixteen hours to achieve equilibrium when running at half the production capacity. A problem that was encountered at such a low production rate was a loss of temperature throughout the unit. This had a large effect on the feed pumps since the increase in viscosity at the lower temperatures caused the feed pressures to increase due to the larger pressure drop across the columns.

The feed to the pilot plant for the trials during the C-series were inverted clarified and softened C-molasses at 60°Brix. The preparation method is described in an earlier chapter. The average of the analyses of the feed product is reflected in Table 8.5.

Table 8.5: Average analysis of feed molasses for the C-series.

Brix	°	58.7
Density	kg/l	1.282
Salts	%DS	23.65
Sucrose	%DS	2.56
Glucose	%DS	35.31
Fructose	%DS	38.47
Total invert	%DS	73.79
Colour	ICUMSA	181,866
Turbidity	NTU/Brix	12.07
Conductivity	µS/cm	9,304

Typical operating conditions on the pilot plant were:

Inlet Temperature of water:	65 °C to 70 °C
Inlet Temperature of molasses:	55 °C to 60 °C
Inlet Pressure of water:	3.2 bar to 3.6 bar
Inlet Pressure of molasses:	1.5 bar to 1.7 bar
Extract Temperature:	50 °C to 60 °C
Raffinate Temperature:	55 °C to 65 °C

Table 8.6: Results obtained from the C-series of trails.

	Average values of the results obtained from trials conducted during the C-series	**Average values of the results obtained by adjusting the BV setpoint by increasing the extract flowrate**	**Average values of the results obtained by maintaining the BV setpoint at the Applexion specification**
BV1	0.697	0.700	0.694
BV2	0.592	0.592	0.591
BV3	0.661	0.661	0.661
BV4	0.383	0.383	0.383
Recovery			
Salt/Extract %	2.29	3.2	1.0
Tot Invert/Extract %	92.45	91.5	93.9
Salt/Raffinate %	97.71	96.8	99.0
Tot Invert/Raffinate %	7.55	8.5	6.1
Extract			
Brix	26.32	24.7	28.7
Salts %	0.48	0.7	0.2
Sucrose %	0.89	1.4	0.1
Glucose %	44.90	46.1	43.1
Fructose %	53.70	51.8	56.6
Tot Invert %	98.59	97.8	99.7
Conductivity µS/cm	1083	1195	915
Raffinate			
Brix	10.4	10.7	10.0
Salts %	69.73	68.9	71.0
Sucrose %	11.83	12.1	11.4
Glucose %	16.64	17.3	15.7
Fructose %	1.80	1.7	1.9
Tot Invert %	18.44	19.0	17.6
Conductivity µS/cm	16453	16977	15662
Loop			
Brix	0.87	0.9	0.8
Salts %	61.45	61.1%	61.9%
Sucrose %	8.80	9.1%	8.3%
Glucose %	11.99	11.0%	13.4%
Fructose %	17.75	18.7%	16.4%
Tot Invert %	0.30	0.3%	0.3%
Conductivity µS/cm	1002	1070	901

The trials conducted in the C-series disproved the assumption made during the A and B series trails, that an adjustment was necessary to improve the recovery and the sugar yield. The performance of the pilot plant matched that of the small test unit used by Applexion on C-molasses. The C-series trials were conducted at full production capacity of the pilot plant.

The effect of temperature was clearly demonstrated at one stage when the feed tank was drained to below the heating coils. The lowered temperature of the feed resulted in a significant increase of the inlet pressure of both the water and the molasses. After the level of molasses was restored and the temperature normalised, the pressure of both pumps decreased to normal operating pressures. A similar effect was witnessed when the water temperature decreased. This can be attributed to the viscosity increase in the molasses, as can be seen in Figure 4.3.

The pilot plant performance met three of the objectives set out earlier namely:

a) No dry solids was present in the basic loop
b) The Invert Sugar recovery was optimised
c) The desired purity in the Extract was reached.

Reaching 93.9 % recovery of sugars at 99.7 % purity was very close to the optimum. However, the trade-off between increasing the recovery at the cost of reducing the purity had to be investigated. To do this, the values of BV2 and BV3 needed to be adjusted while BV1 and BV4 had to remain constant. A drop in BV2 and BV3 should result in a higher recovery with lower purity, while an increase in BV2 and BV3 should result in an even purer product with lower recovery. Having a pure product has the advantage that it saves money on the downstream deashing of the extract stream. The raffinate stream is to be re-used in the process to regenerate the softening column, and some sugar could be recovered from the resin during the wash cycle as a sweet-water. The effect of the remaining sugar in the regenerant effluent and its downstream utilisation should be investigated.

It was felt that further trials with a higher feed concentration were needed to optimise the water consumption of the unit, which at this stage was 6.084-kg water/kg solids. These trials were referred to as the D-Series trials.

8.4. Results: The D-series of trials

Before these trials, considerable effort was placed on finalising the pre-treatment so that the work on decolourisation could commence. A large quantity of inverted molasses syrup was prepared. The extract from the chromatography was directly passed into the decolourising columns as described in Chapter 9.

Operation started with the same operating parameters as were used in the A-C trails. The performance of the system was adequate for the first few production cycles. It then became evident that the extract brix was very low (2 Brix and lower). A study was conducted to try and determine the cause of this observation. All the “missing” solids seemed to appear in the raffinate. Effectively the unit was only diluting the feed, with an excess water stream and a dilute feed stream leaving the system. Two faulty valves were identified and repaired. These valves caused the streams to short circuit and not flow through the columns in the preferred manner. However the problem persisted. Samples were taken to determine outlet brix every 15 minutes in an attempt to see whether the sampling interval used previously was not coinciding with a column coming online at the same time, or just before the sample was taken. That would result in low solids content due to the displacement of water from the column during the initial time period. The results from this trial showed a consistent trend in the extract – clearly the low concentration was not due entirely to the cycle interval of the unit. The results obtained from the continuous monitoring of the Brix of the extract are depicted graphically in Figure 8.3.

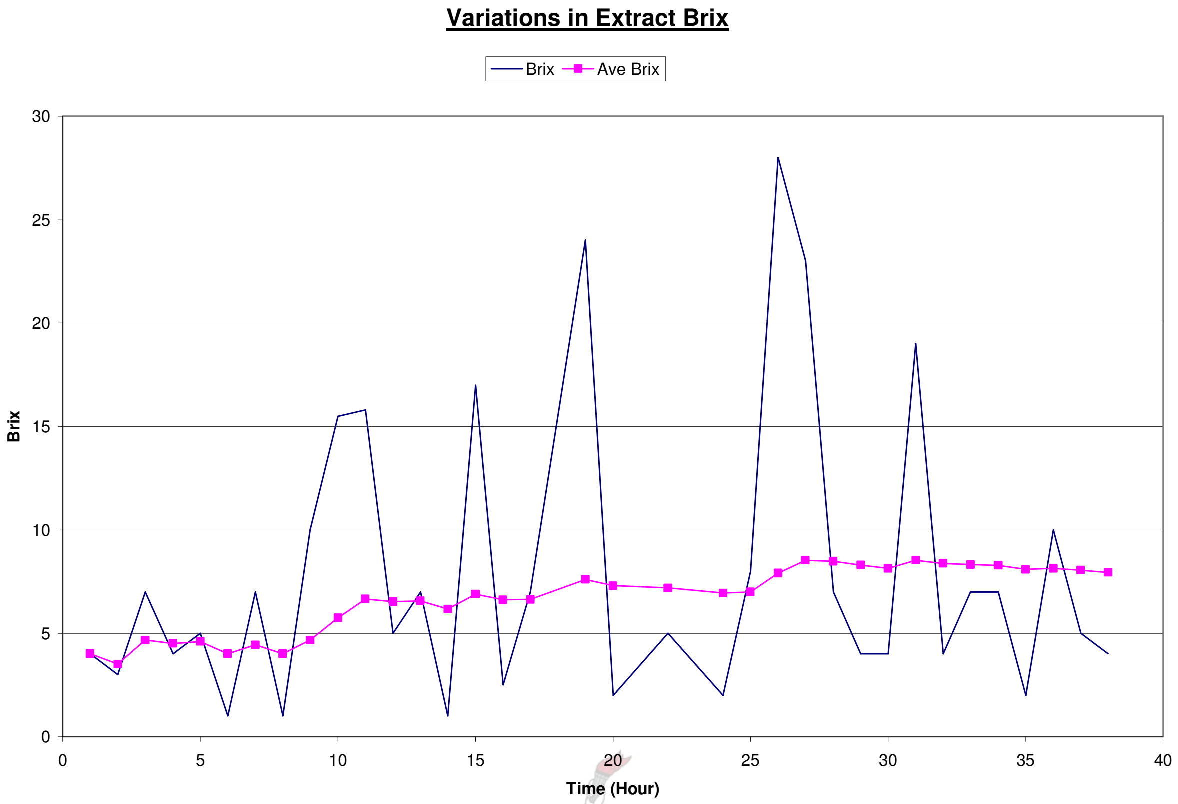


Figure 8.3: Variations in the extract Brix as observed during the start of series D.

The column profile over a step was then determined. This is an indication of the separation occurring inside the SMB system.

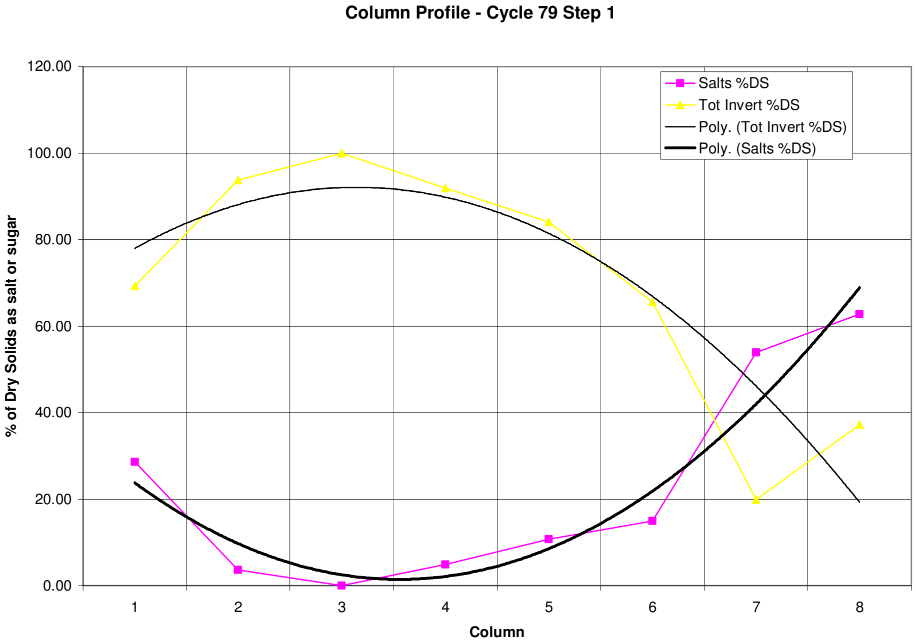


Fig 8.4: The concentration profile over the SMB during Cycle 79, step 1.

The results obtained from Cycle 79 Step 1 are summarised in Table 8.7, while the dry matter profile is illustrated in the graph in Figure 8.4. The flows into and out of the SMB during Step 1 are shown in Figure 8.5.

Table 8.7: Column Profile for Cycle 79 – Step # 1

Date		24 Nov 2000							
Time		06:17							
Cycle #		79	Step # 1	Time of Sample: 670 s					
Setpoints									
Feed	l/h	14							
Water	l/h	61.3	BV1 =	0.672					
Extract	l/h	21	BV2 =	0.567					
Raffinate	l/h	54.3	BV3 =	0.637					
Loop	l/h	73.2	BV4 =	0.365					
Step time	sec	1348							
Column #		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Brix	%	0	0	31	34	28	20	7	1
Density		1.000	1.000	1.133	1.147	1.118	1.081	1.027	1.004
Colour	ICUMSA			34175	121876	204215	42611	267305	257921
Turb	NTU/Brix			1.60	0.60	0.45	2.53	0.83	10.90
Cond	milliS/cm	6.21	1.38	1.277	1.309	1.303	1.443	96.8	6.82
PH		5.08	5.34	5.12	4.95	5.14	5.18	4.67	5.36
Salts	**%DS**	**28.56**	**3.66**	**0.00**	**4.88**	**10.73**	**14.98**	**53.93**	**62.77**
Sucrose	%DS	3.12	2.60	0.00	3.21	5.20	16.05	26.19	0.00
Glucose	%DS	30.66	17.72	16.96	45.62	51.33	38.91	18.22	13.14
Fructose	%DS	38.59	76.02	83.04	46.29	32.74	26.68	1.66	24.09
Tot Invert	***%DS***	***69.25***	***93.75***	***100.00***	***91.91***	***84.07***	***65.60***	***19.88***	***37.23***

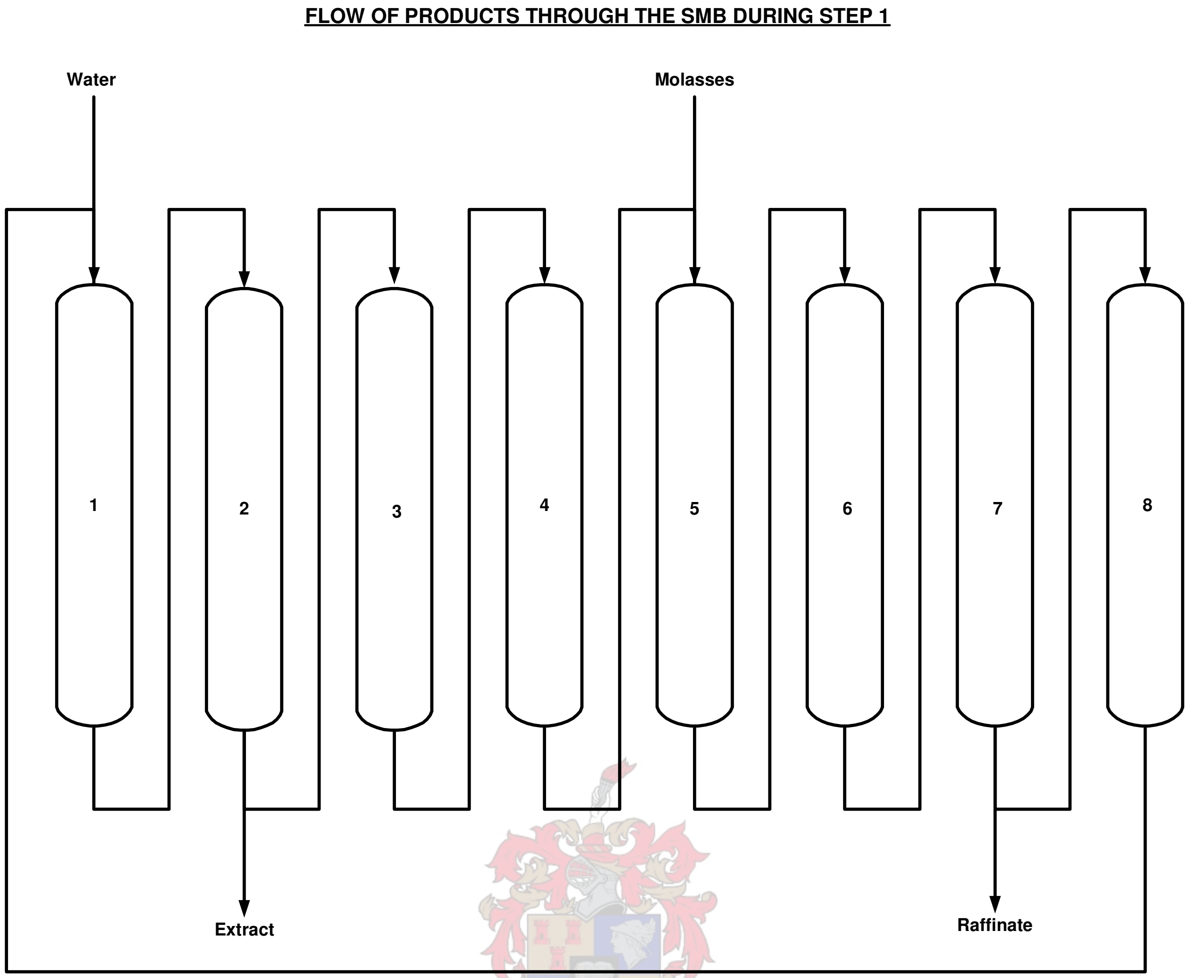


Figure 8.5: A Schematic representation of the flows into and out of the SMB during Step 1 of each cycle.

The separation of invert in the system seems to be very good with the invert having a very high purity (low salts content) on column 2. However the salt fraction has a large amount of invert. Raffinate is leaving the system on column 7 and according to the column profile; there is a significant amount of sugar in the raffinate. This is typical of a too high eluent flow and unstable values for BV2 and BV3.

Sucrose can be used as an indicator of separation efficiency as it is more excluded than invert sugar and less retained than the salt. It elutes between the two components. By looking at the sugar peak, one can get a picture of the efficiency of separation. Further examination of the pilot plant showed that the mass balance across the system balanced, and that the plant was performing in the proposed operating band to optimise the separation. The results however, indicated that this specific condition was not beneficial to the separation of molasses on the SMB.

Adjustments were then made to the flow rates resulting in a change in the Bed Volumes of separation (BV1 to BV4). The results are reported in Table 8.8 and Figure 8.6.

Table 8.8: Adjusted flow (and BV) to increase extract concentration

Date		27 Nov 2000							
Time									
Cycle #		98	Step # 1	Time of Sample: 874 s					
Column #		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Setpoints									
Feed	l/h	9.4							
Water	l/h	40.9	BV1 = 0.653						
Extract	l/h	14	BV2 = 0.550						
Raffinate	l/h	36.2	BV3 = 0.619						
Loop	l/h	47.3	BV4 = 0.350						
Step time	sec	2000							
Column		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Brix	%	0	0	31	34	28	20	7	1
Density		1.000	1.000	1.133	1.147	1.118	1.081	1.027	1.004
Colour	ICUMSA			34175	121876	204215	42611	267305	257921
Turb	NTU/Brix			1.60	0.60	0.45	2.53	0.83	10.90
Cond	milliS/cm	6.21	1.38	1.277	1.309	1.303	1.443	96.8	6.82
PH		5.08	5.34	5.12	4.95	5.14	5.18	4.67	5.36
Salts	%DS	***11.73***	***18.86***	***25.59***	***45.30***	***18.30***	***13.39***	***1.46***	***0.95***
Sucrose	%DS	10.45	14.01	19.03	0.00	2.61	6.32	0.00	2.09
Glucose	%DS	40.29	48.58	38.57	36.77	47.94	43.82	36.83	48.57
Fructose	%DS	37.52	18.55	16.81	17.94	31.16	36.47	61.71	48.38
Total Invert	***%DS***	***77.82***	***67.13***	***55.38***	***54.70***	***79.10***	***80.29***	***98.54***	***96.95***

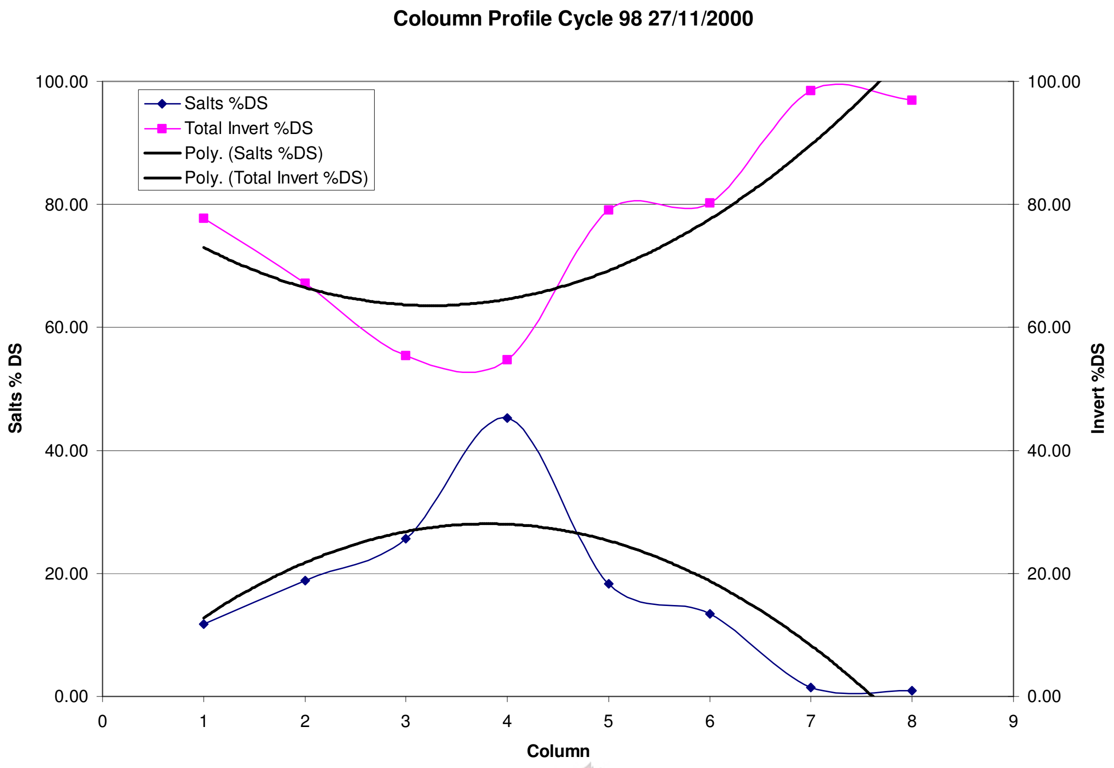


Fig 8.6: Column Profile after changing BV's cycle 98, Step#1

The adjustments made to the various BV values did improve the separation of the unit. The performance of the unit thereafter remained satisfactory for the duration of the trials. The performance of the unit with these new setpoints is summarised in Table 8.9.

Table 8.9: Summary of the results obtained during the D-series of trails.

	Values of the results obtained from cycles 98 to 100	**Values of the results obtained from cycles 101 to 103**
BV1	0.653	0.653
BV2	0.550	0.550
BV3	0.619	0.619
BV4	0.350	0.350
Recovery		
Salt/Extract %	1.37	0.20
Tot Invert/Extract %	99.04	97.70
Salt/Raffinate %	98.73	99.81
Tot Invert/Raffinate %	0.96	2.30
Extract		
Brix	29	20.6
Salts %	0.26	0.06
Sucrose %	0.00	0.17
Glucose %	39.82	28.10
Fructose %	59.92	71.68

	Values of the results obtained from cycles 98 to 100	**Values of the results obtained from cycles 101 to 103**
Tot Invert %	99.74	99.77
Conductivity mS/cm	11.4	6.96
Raffinate		
Brix	9	10.6
Salts %	64.44	60.39
Sucrose %	32.19	34.86
Glucose %	0.00	0.00
Fructose %	3.37	4.75
Tot Invert %	3.37	4.75
Conductivity mS/cm	160.4	186
Feed		
Brix	64	64
Salts %	15.70	16.77
Sucrose %	11.89	11.23
Glucose %	35.09	33.04
Fructose %	37.32	38.96
Tot Invert %	72.41	72.00
Conductivity mS/cm	71.8	42.3
Loop		
Brix	0.5	0.5
Salts %	1.52	42.33
Sucrose %	0.00	17.71
Glucose %	52.65	6.57
Fructose %	45.83	33.39
Tot Invert %	98.48	39.97
Conductivity mS/cm	1.61	1.91

The cause for the disturbance was, and still is not clearly known especially since the unit had been operated under these conditions before. The molasses entering the unit was softened with a resin in the sodium form. It is postulated that the chromatography resin acted as an ion exchange resin and exchanged potassium for sodium thereby changing the affinity of the resin. The intention was to regenerate the softener with potassium rich raffinate. However, a potassium salt was substituted until such time that sufficient raffinate was available to be used as regenerant. The potassium rich molasses could also have been a contributing factor to the excellent separation achieved in these trials.

Ageing of the resin is also a possibility contributing to the disturbance. It is common for an ion exchange resin to have a high initial efficiency, which later levels out to a plateau, and which represents the real commercial efficiency expected for the lifetime of the resin. The resin used in the chromatography pilot plant was new resin. It is theoretically possible that the resin reached the plateau phase of operation only during the D-series trials. It is common practice in industrial applications to monitor the profile of the system at regular intervals so as to prevent such separation phase transition from occurring. During this series of trials is was noted that some of the pneumatic actuators on the manifold valves were “sticky” and sometimes failed to open or close the valve completely, even though the feedback indicator showed that the valve had taken the required action. These valves were found on the bottom manifolds housing the extract, raffinate and loop pipes. Eventually some of the air solenoid valves on the actuators had to be replaced and it solved the problem. It is therefore strongly suspected that the real cause for the disturbance was a valve that did not close properly and allowed liquid to bypass the resin system and flow into the raffinate manifold. There are no flow indicators on the piping linking the columns, so it is possible for this phenomenon to occur without any indication.

8.5. Conclusion

The separation of sugars from molasses was achieved using a simulated moving bed pilot plant. A sugar recovery of 93.9 % at a purity of 99.7 % from molasses, having a purity of 78.9 %, was achieved. The operating conditions for this performance was:

Feed: 14 l/h at a temperature > 60 ℃
Water: 63 l/h at a temperature > 65 ℃
Extract: 21 l/h
Raffinate: 56 l/h
Loop: 78 l/h
Step time: 1326 seconds.

This relates to the following separation zone bed volumes:

BV1 = 0.694
BV2 = 0.591
BV3 = 0.661
BV4 = 0.383

Maintaining the same bed volumes but at a lower productivity or capacity of the unit did not produce acceptable performance mostly because of heat loss. The recovery of sugar was 89.1 % at a purity of 98.9 %. The temperature of the feed and the system determine the operating pressure. A higher temperature resulted in lower operating pressures. This has an impact on the operation of the pumps in a commercial plant.

With this set of operating parameters the ratio of eluent water to feed was 4.5.
The investigation to eliminate the phase transition during the D-series of trials resulted in the following operating conditions of the unit:
Feed: 14 l/h at a temperature > 60 ℃
Water: 60.9 l/h at a temperature > 65 ℃
Extract: 20.9 l/h
Raffinate: 53.9 l/h
Loop: 70.4 l/h
Step time: 1343 seconds.

This relates to the following bed volumes:
BV1 = 0.653
BV2 = 0.550
BV3 = 0.619
BV4 = 0.350.

The ratio of water to feed was reduced to 4,35 and the invert recovery in the extract was 98.37% at a product purity of 99.63%. This set of operating conditions was superior to that obtained in the earlier trials due to the higher invert recovery. The separation efficiency of the unit was not affected by a lower productivity using these bed volume setpoints. No pressure problems were experienced partly because of an improved clarification process. The operating temperature range was the same as the earlier trials.

It is important to periodically monitor the profile of the separation through the system. Ageing of resin, slight changes in functionality and fouling can influence the separation efficiency of the system. In a commercial plant this pro-active setpoint adjustment will prevent a phase transition. Having finally produced a good quality extract, the way was opened for the evaluation of a suitable decolourising process for the extract. This investigation will now be described.

Chapter 9

Decolourisation of Chromatographic Extract

9.1. Introduction

In the market, a variety of liquid sugar products are sold. They vary mainly in composition and colour. To compete in the market a process is required to decolourise the chromatographic extract.

A lot of work has been done to determine and identify the colourants and colour producing molecules present in sugar factory products. Farber and Carpenter (year unknown) listed a number of components identified by them. Most notable is chlorogenic acid - a compound that has a yellowish colour and is very difficult to remove from sugar solutions. Lancrenon and Herve (1988) reviewed the use of ion exchange resins in the sugar industry and discussed amongst others the state of decolourisation with resin.

The aim of the trials was to produce a “colourless” invert sugar solution with no ash. The invert sugar exiting the chromatography plant as extract was pumped through a series of ion exchange columns. The columns were filled with cationic, anionic and adsorbent resin. An attempt was made to determine the capacity of the various ion exchange resins. The configuration of the sequence of resin columns was also investigated to determine an optimum arrangement for the industrial plant.

9.2. Resin Properties

The following resins were evaluated: Amberlite 252; Amberlite IRA92; Amberlite IRA96; Amberlite IRA958 & Optipore SD-2. Their basic properties are shown in Table 9.1 below.

Table 9.1: Resins that were evaluated in the study

Resin	Supplier	Type	Matrix	Functional Group
Amberlite 252	Rohm and Haas	Strong Acid Cation (SAC)	Macroporous Styrene DVB Copolymer	$-SO_3^-$
Amberlite IRA92	Rohm and Haas	Weak Base Anion (WBA)	Macroporous Styrene DVB Copolymer	Secondary Amine $-NR_2$
Amberlite IRA96	Rohm and Haas	Weak Base Anion (WBA)	Macroporous Styrene DVB Copolymer	Tertiary Amine
Amberlite IRA958	Rohm and Haas	Strong Base Anion (SBA)	Macroreticular Crosslinked Acrylic	Quaternary Amine
Optipore SD-2	Dow	Adsorbent (ADS)	Macroporous Styrene DVB Copolymer	Tertiary Amine

Detail datasheets of the resins are included in Appendix A.

9.3. Experimental Design

The columns were made from 100 mm stainless steel pipe and operated down-flow at a flow-rate of 22 l/h. Since each column was filled with 7 litres of resin this results in:

$$\text{Flow rate (BV/h)} = \frac{\text{Flow (l/h)}}{\text{Bed Volume (l)}} = \frac{22}{7} = 3.14\text{BV/h}$$

The flow-rate was varied to evaluate the resin at lower operating flows. The flow-rates under investigation varied from 14 l/h (2 BV/h) to 22 l/h (3.14 BV/h).

Samples of the product were taken and analysed after every two-bed volumes (i.e. after every 14 litres of solution) to determine the breakthrough. Other samples after every four bed volumes were taken for production or control runs. The following tests were done on each sample: Brix, pH, Conductivity and ICUMSA Colour. Methods for these analyses are described in Chapter 3.

Operation of the one train would cease when a specified “breakthrough” was achieved on the columns. All the columns on the saturated train would then be regenerated while the train in stand-by would handle production. The breakthrough curves for each column in the train were compiled and the capacity and colour loads were determined. The total amount of colour entering the column can be considered as the ICUMSA colour multiplied by the solids content. This brings the dimensionless ICUMSA Colour in relation to the solids content of the liquid.

ColourValue = (Icumsa Colour)*(Brix/100)

To calculate the total colour on the resin, one needs to integrate the colour curve, or determine the area under the curve. A simplification of this is to consider small intervals and to assume that the area under the curve is rectangular for that incremental interval. This is not entirely true, but gives a good approximation of the total colour contained in the extract. It can be expressed as:

AccumulatedColour = $\sum$ (ColourValue * IncrementalFlow)

In the case of the decolourisation, the incremental flow can be considered as the difference in “Bed Volume” of the liquid flowing through the column. The colour in the decolourised extract leaving the columns can be calculated in a similar manner.

To obtain a colour load per unit of resin volume, the difference between the inlet and the outlet indicates the colour remaining on the resin. Dividing this “retained” colour by the resin volume gives a value representing the colour load per unit volume of resin. This colour load has dimensionless units. The colour load was thus calculated with the following formula as derived above:

$$\text{Colour Load}_{\text{BV}} = \frac{\left(\Delta\text{BV}\cdot\frac{\text{Inlet Brix}}{100}\cdot\text{InletColour}\right)-\left(\Delta\text{BV}\cdot\frac{\text{Outlet Brix}}{100}\cdot\text{OutletColour}\right)}{\text{Resin Volume}}$$

Where:

ΔBV = the amount of bed volumes between the inlet and the outlet samples.
Inlet Brix = Refractometer Brix of solution entering column
Outlet Brix = Refractometer Brix of solution leaving column
Inlet Colour = ICUMSA Colour of solution entering column
Outlet Colour = ICUMSA Colour of solution leaving column.

The capacity of the resin was determined by calculating the cumulative colour load over the column. The amount of Bed Volumes passing through the column before a specified cut-off value was reached gave an indication of the efficiency of the resin.

Also, an attempt was made to describe the adsorption behaviour of the colour bodies on the resin in the last of the decolourising columns with Freundlich isotherms.

9.4. Plant Layout

Twelve columns made of 316L Stainless Steel pipe (100 mm nominal diameter), were arranged in two trains of six columns each. Each column was filled with a layer of filter sand at the bottom followed by the required amount of resin. The arrangement of the two trains and the resin in the individual columns are listed in Table 9.2.

Table 9.2: Arrangement & resin fill of the columns on the Pilot Plant.

Resin	Train 1	Train 2
Amberlite 252 (SAC)	Column 1 with 7 litres	Column 1 with 7 litres
Amberlite IRA92 (WBA)	Column 2 with 7 litres	Column 2 with 7 litres
Amberlite 252 (SAC) **or** Amberlite IRA958 (SBA)	Column 3 with 7 litres	Column 3 with 7 litres
Optipore SD-2 (ADS) **or** Amberlite IRA96 (WBA)	Column 4 with 7 litres	Column 4 with 7 litres
Amberlite IRA96 (WBA) **or** Optipore SD-2 (ADS) **or** Doulite XAD761	Column 5 with 7 litres	Column 5 with 7 litres
Optipore SD-2 (ADS) **or** Doulite XAD761	Column 6 with 7 litres	Column 6 with 7 litres

The decolourising trains were connected to the extract outlet port on the ion exclusion plant. One train was in operation while the other was in either regeneration or standby.

Figure 9.1 shows the operating arrangement:

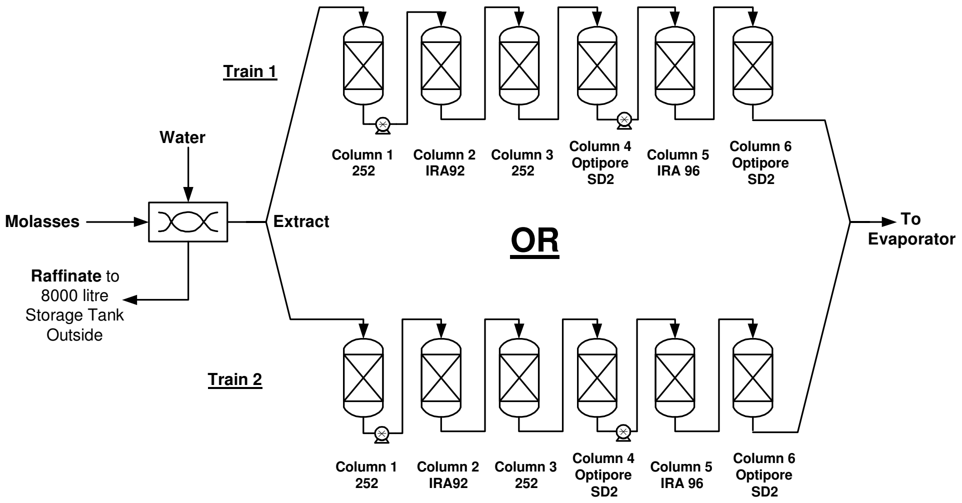


Figure 9.1: The operating arrangement of the decolourising columns on the pilot plant.

Temperature loss was overcome with the installation of a coiled heat exchanger between columns three and four. The heat exchanger had two coils in a jacket flushed with hot water.

Regeneration of the columns was in counter-flow to the operation: i.e. up-flow. Table 9.3 details the regeneration conditions of the various resins.

Table 9.3: Regeneration conditions of the resins evaluated

	Amberlite 252	**Amberlite IRA 92**	**Amberlite IRA 96**	**Amberlite IRA 958**	**Optipore SD-2**
Water Rinse	2 BV at 2 BV/h	2 to 5 BV at 2 to 5 BV/h	2 to 5 BV at 2 to 5 BV/h	2.5 BV at 2.5 BV/h	2 BV at 2 BV/h
Regenerant	HCl; 1 N soln.	NaOH; 1 N soln.	NaOH; 1N soln.	KCl; 10% soln.	NaOH; 1 N soln.
Regenerant Quantity and Flow	2 BV at 2 BV/h	2 BV at 2 BV/h	2 BV at 2 BV/h	2 BV at 2 BV/h	1 BV at 1 BV/h
Slow Rinse	2 BV at 2 BV/h	2 BV at 2 BV/h	2 BV at 2 BV/h	2 BV at 2 BV/h	2 BV at 2 BV/h
Fast Rinse	5 BV at 5 BV/h	5 BV at 5 BV/h	5 BV at 5 BV/h	5 BV at 5 BV/h	5 BV at 5 BV/h

Sufficient stock solutions of the regenerants were made up to ensure quick and efficient regeneration. Solutions were made with demineralised water. Demineralised water was also used to rinse the columns. Potassium rich effluent from the IRA958 regeneration was returned to the raffinate tank for softener regeneration.

Desweetening, regeneration and rinsing lasted between 4 hours to 4 ½ hours after which the columns were returned to stand-by.

9.5. Breakthrough Characteristics

The results that were obtained from the experiments are presented below. The columns were connected directly to the outlet of the SMB unit, as indicated in Figure 9.1 earlier. This however, led to “flutter” of the results because of the change in concentration of the extract throughout the step. However, the general trends are visible and where required, a trend line was fitted over the variable under investigation so as to smooth out the actual curve.

Ideally, the trials should have been conducted using a homogeneous feed that was a composite of the extract produced on the SMB. Due to a variety of reasons this was not possible. A suitable pump was not available and the project budget did not allow for the purchase of such a pump. Secondly, and more importantly, was the danger of fermentation, as there were no sterile storage facilities available close to the pilot plant. It was decided not to add preservative to the extract as this might influence the decolourisation and disqualify the product in the market. Furthermore, the existing arrangement allowed the resin to operate under typical plant conditions, rather than laboratory conditions. Even with the flutter, conductivity and colour breakthrough were clearly seen as shown in the results that follow.

9.5.1 Train 1

The results of the experiments on Train 1 at 2.3 BV/h are reported in Table 9.4.

Table 9.4: The results of the experiment on Train 1 at 2.3 BV/h flow.

Train 1- *Run at 2.3 BV/h*	***Ave Brix***	***Ave pH***	***Ave Cond mS/cm***	***Ave Col***	***Colour Load***	***BV's to Break-through***	***% Colour Removed***
Extract	28.3	5.0	4.30	36459			
Column 1 – Amberlite 252 H	21.4	2.6	2.6	29903	12448	Cond.: 57.5 Col: Cont.	18.0
Column 2 – Amberlite IRA92	21.1	3.8	0.4	19704	17223	Cond.: 50.6 Col: Cont.	34.1
Column 3 – Amberlite 252 H	20.7	3.1	0.6	14997	8076	Cond.: None Col: None	23.9
Column 4 – Optipore SD2	21.0	3.6	0.6	8170	11391	Cond.: N/A Col: 43.7	45.5
Column 5 – Amberlite IRA96	20.7	4.8	0.0	4035	5876	Cond.: None Col: 46	50.6
Column 6 – Optipore SD2	20.3	5.3	0.1	1472	3287	Cond.: N/A Col: 32.2	63.5

The graphical representation of the performance data of each column in the train, during this trial, is given in Figures 9.2 to Figure 9.7. The bed volume for each column was based on the bed volumes that had passed through the first column.

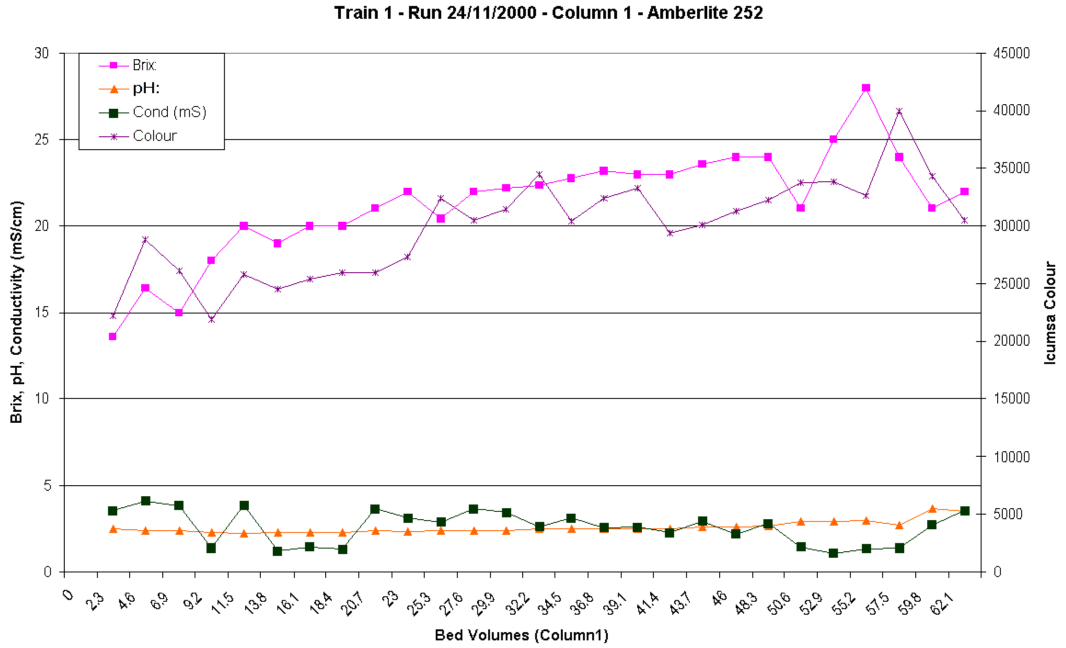


Figure 9.2: The graphical representation of the breakthrough of Column 1

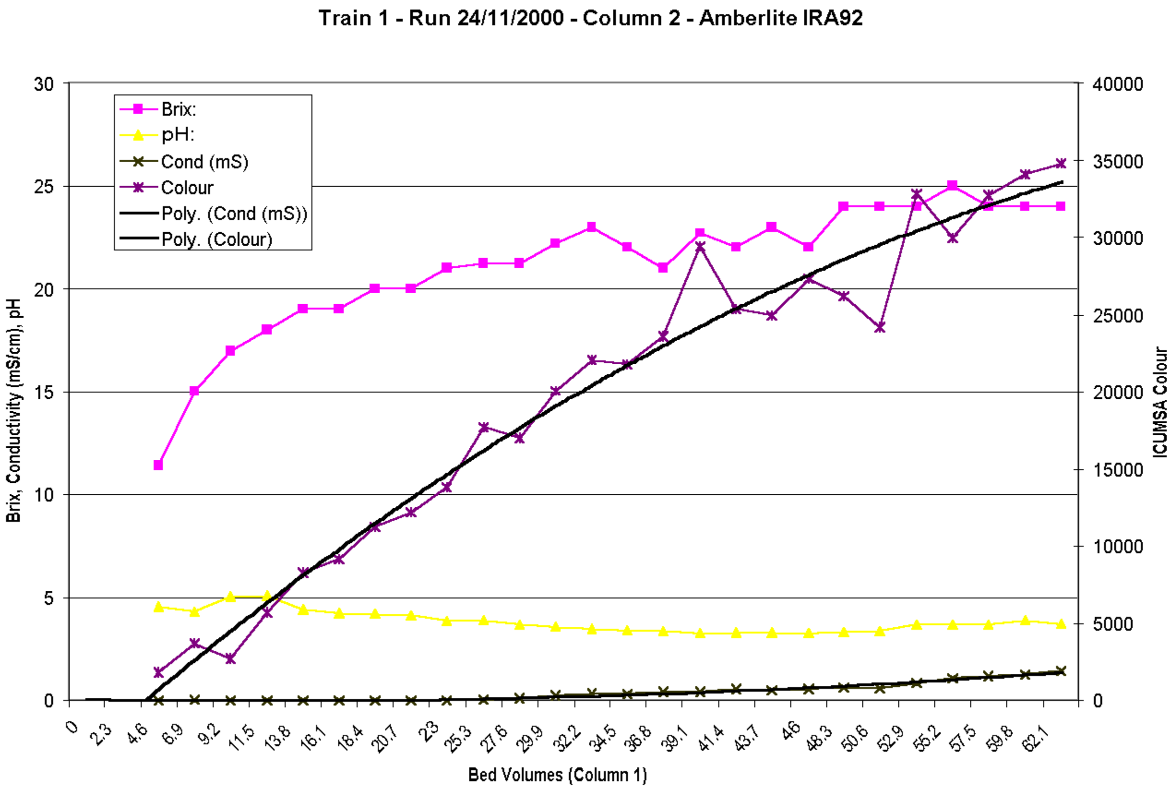


Figure 9.3: The graphical representation of the breakthrough of Column 2.

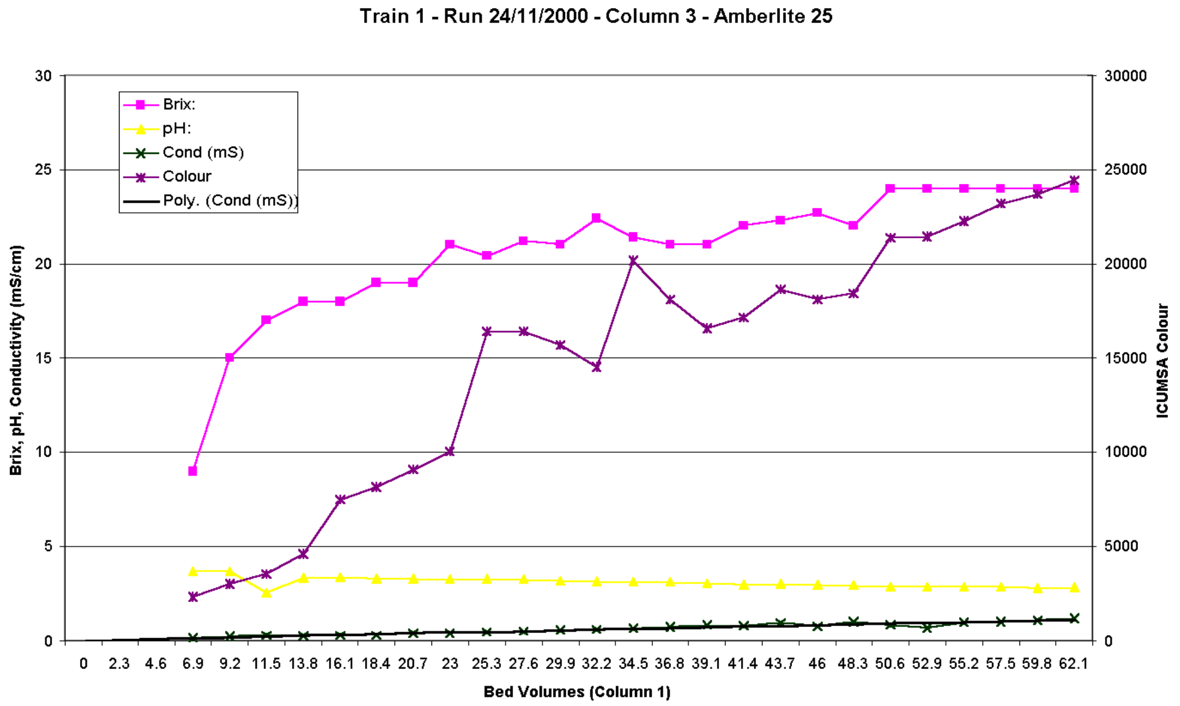


Figure 9.4: The graphical representation of the breakthrough of Column 3**.**

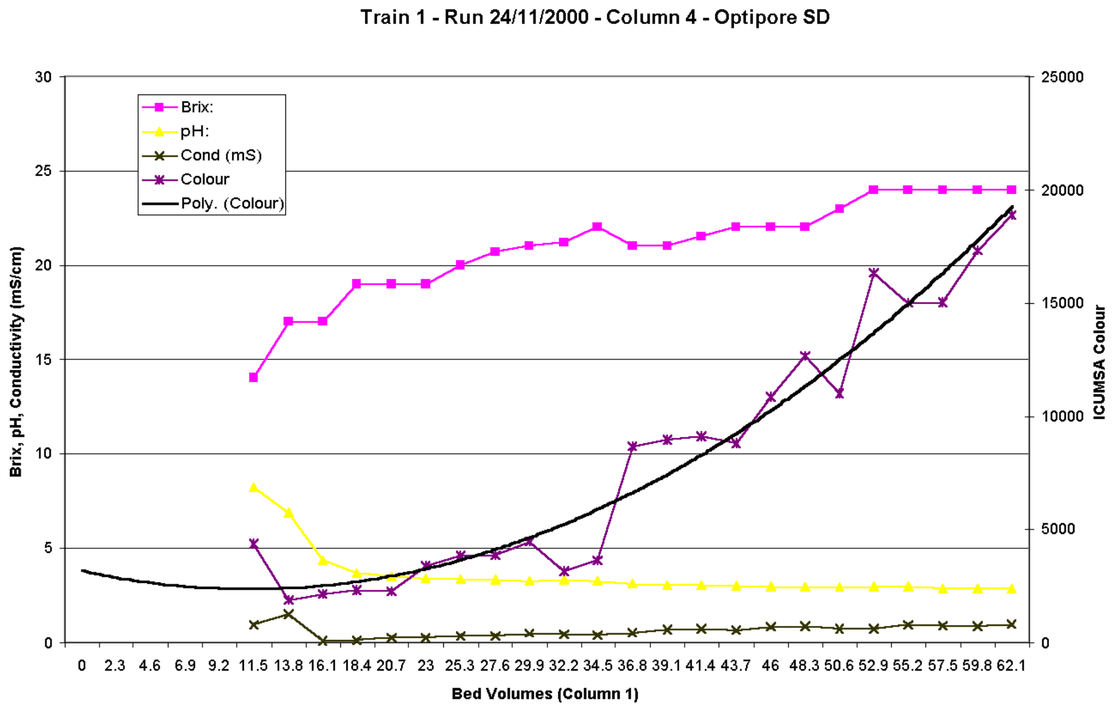


Figure 9.5: The graphical representation of the breakthrough of Column 4

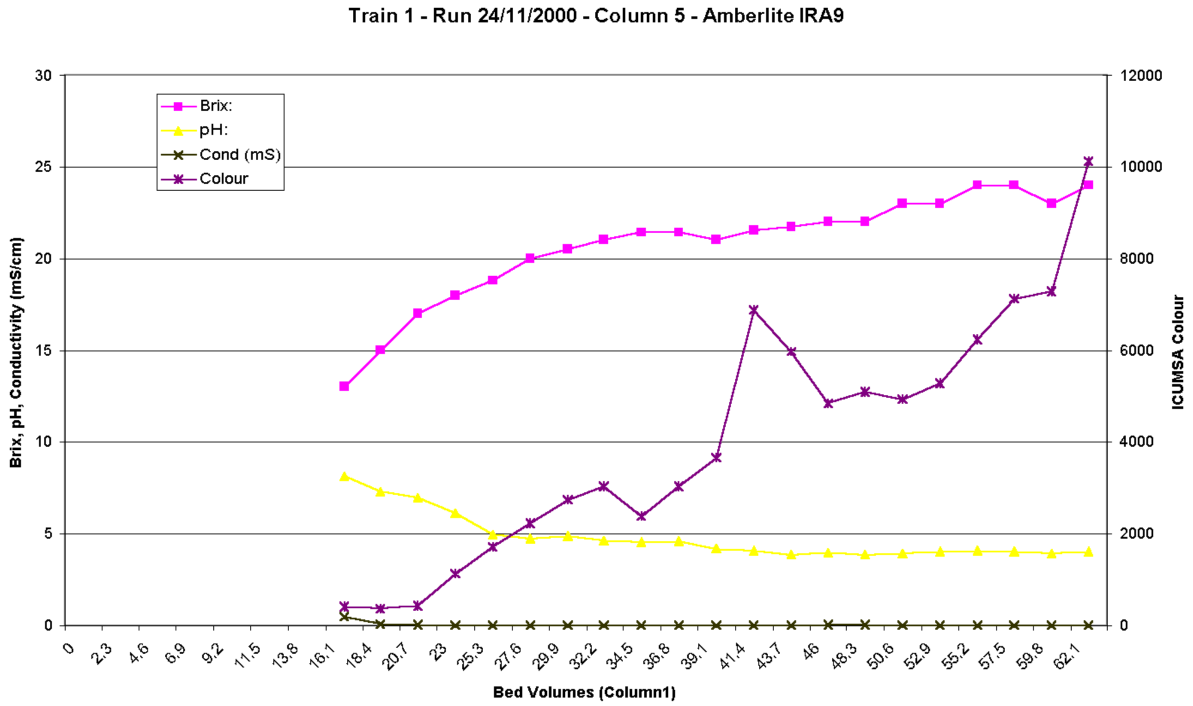


Figure 9.6: The graphical representation of the breakthrough of Column 5

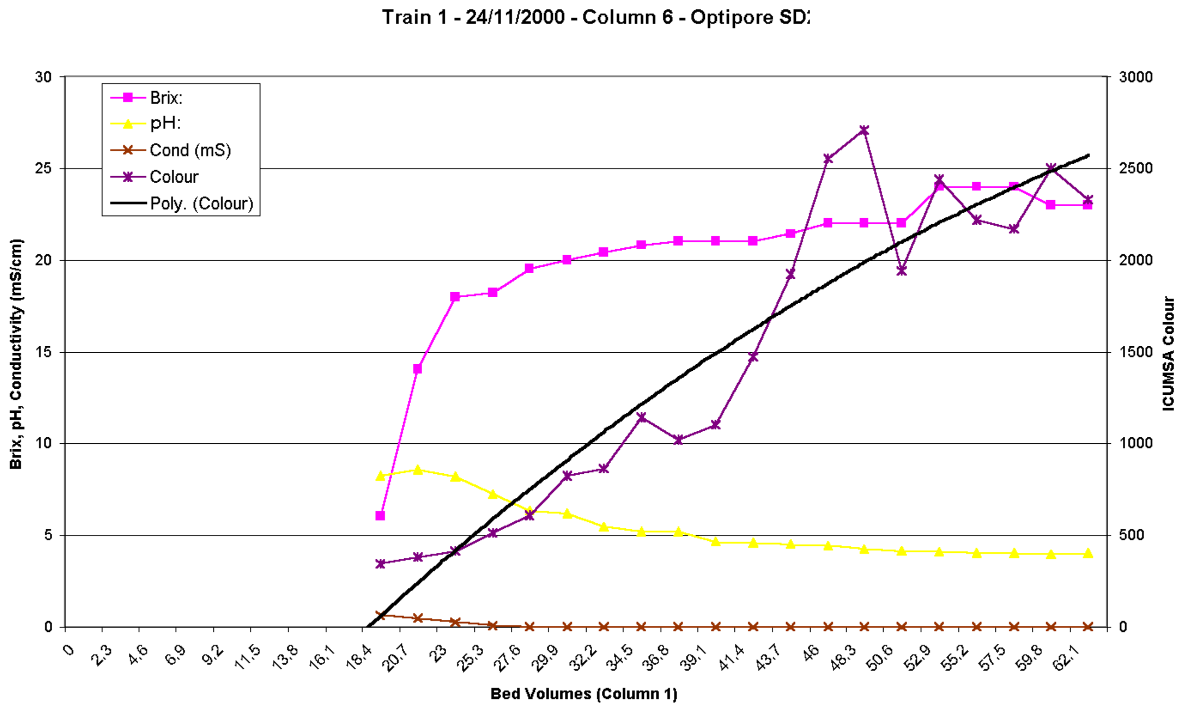


Figure 9.7: The graphical representation of the breakthrough of Column 6.

It is obvious that the columns effectively remove colour and conductivity. The final colour of the liquid exiting the train during this run was 1472 ICUMSA.

A second trail was done with a lower flow at 14 l/h (2 BV/h). The results are reported in Table 9.5 below.

Table 9.5: The results of the second experiment on Train 1 at 2 BV/h.

Train 1 *Run at 2 BV/h*	***Ave Brix***	***Ave pH***	***Ave Cond mS/cm***	***Ave Col***	***Colour Load***	***BV's to Break-through***	***% Colour Removed***
Extract	25.5	5.2	8.80	37149			
Column 1 - Amberlite 252 H	23.0	4.6	5.6	35954	2656	Cond.: Cont. Col: Cont.	3.2
Column 2 - Amberlite IRA92	21.9	4.1	4.4	30291	5167	Cond.: Cont. Col: 33.4	15.8
Column 3 - Amberlite 252 H	20.4	2.6	3.2	29709	6159	Cond.: Cont. Col: N/A	1.9
Column 4 - Optipore SD2	22.6	2.9	4.5	24350	12864	Cond.: N/A Col: Cont.	18.0
Column 5 - Amberlite IRA96	19.3	3.3	1.1	24042	13700	Cond.: Cont. Col: 12	1.3
Column 6 - Optipore SD2	19.8	3.9	0.8	5437	11575	Cond.: N/A Col: 12	77.4

The decolourising efficiency was not as effective as was hoped after the increase in residence time of the extract in the columns. The graphical representations of the individual column performances are shown in Figures 9.8 to 9.13.

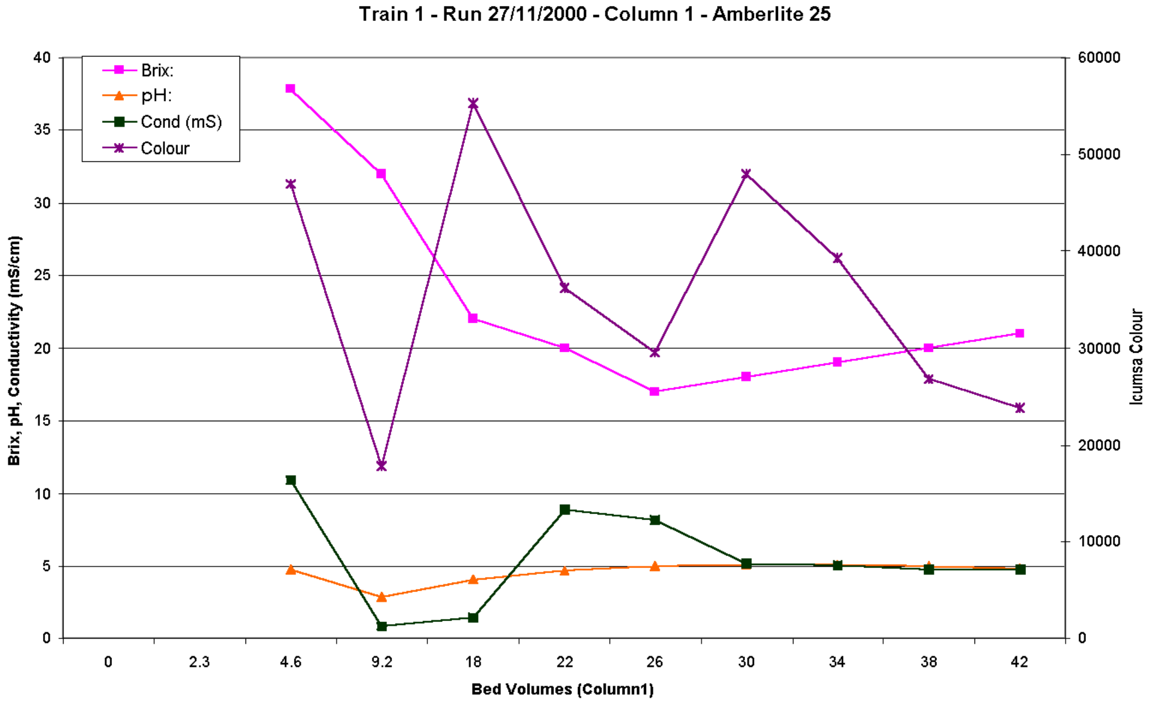


Figure 9.8: The breakthrough curve of Column 1 operating at 2BV/h.

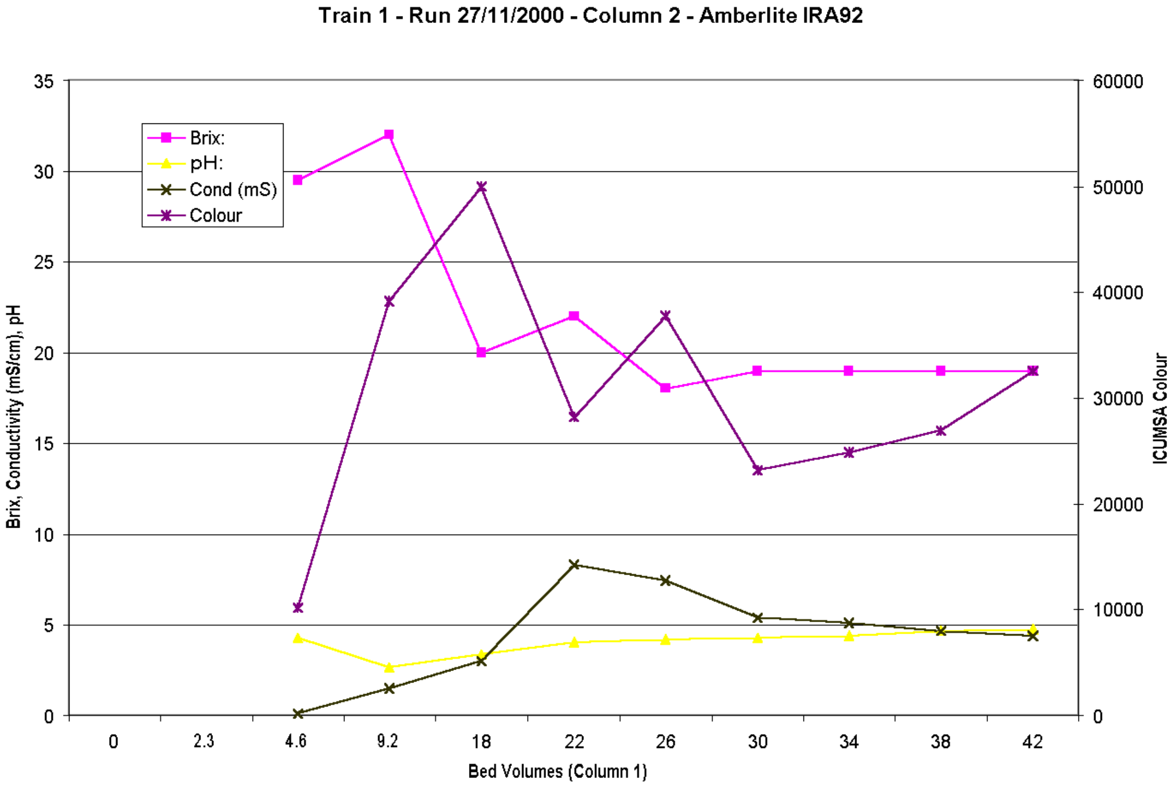


Figure 9.9: The breakthrough curve of Column 2 operating at 2BV/h.

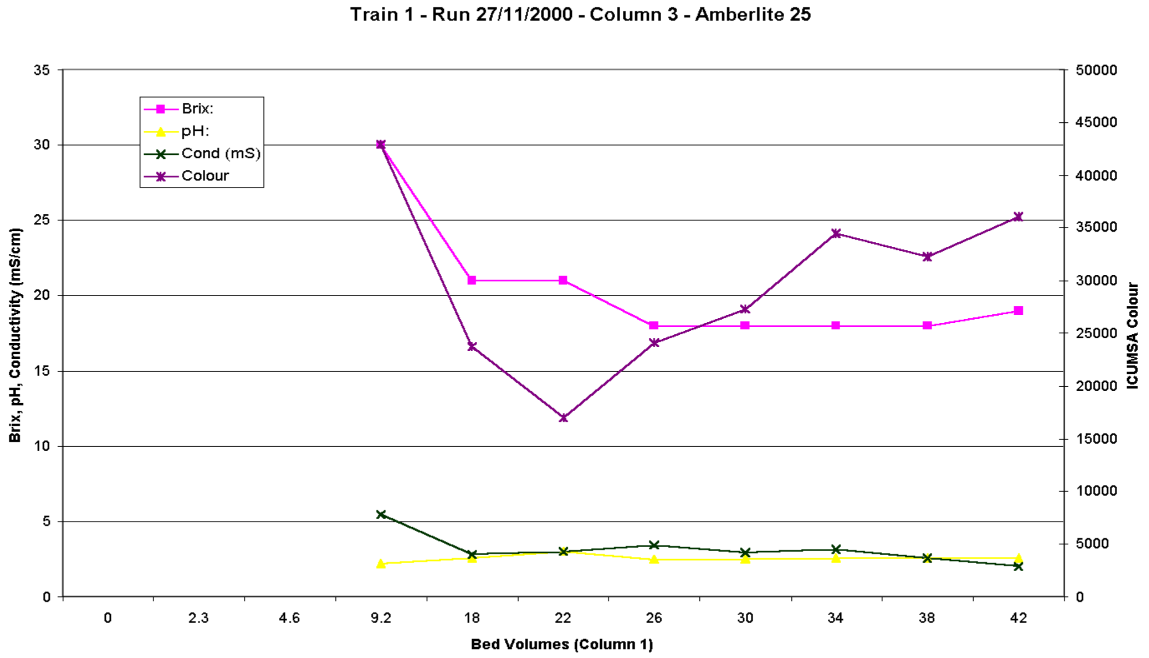


Figure 9.10: The breakthrough curve of Column 3 operating at 2BV/h.

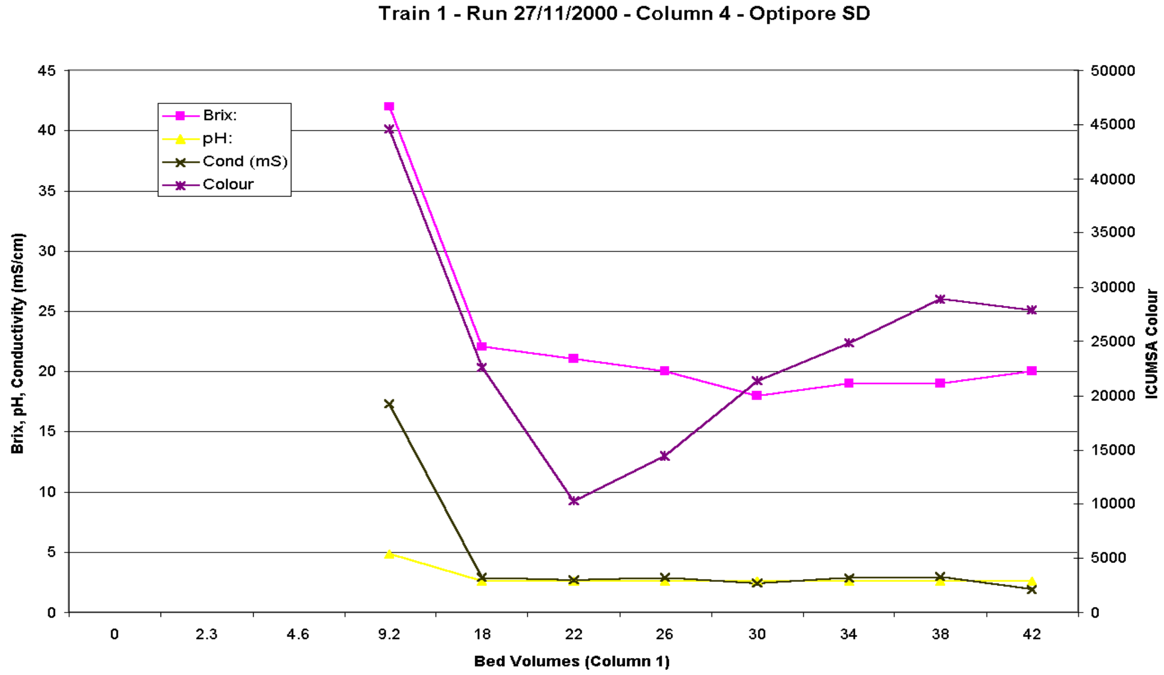


Figure 9.11: The breakthrough curve of Column 4 operating at 2BV/h.

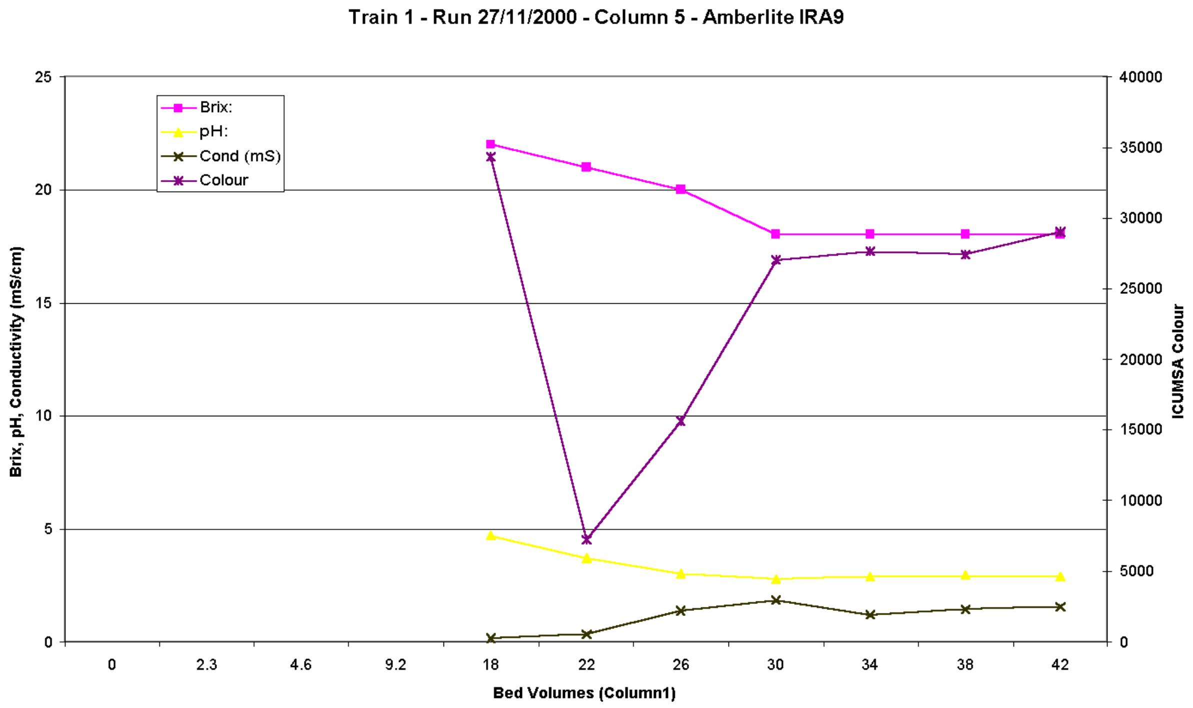


Figure 9.12: The breakthrough curve of Column 5 operating at 2BV/h.

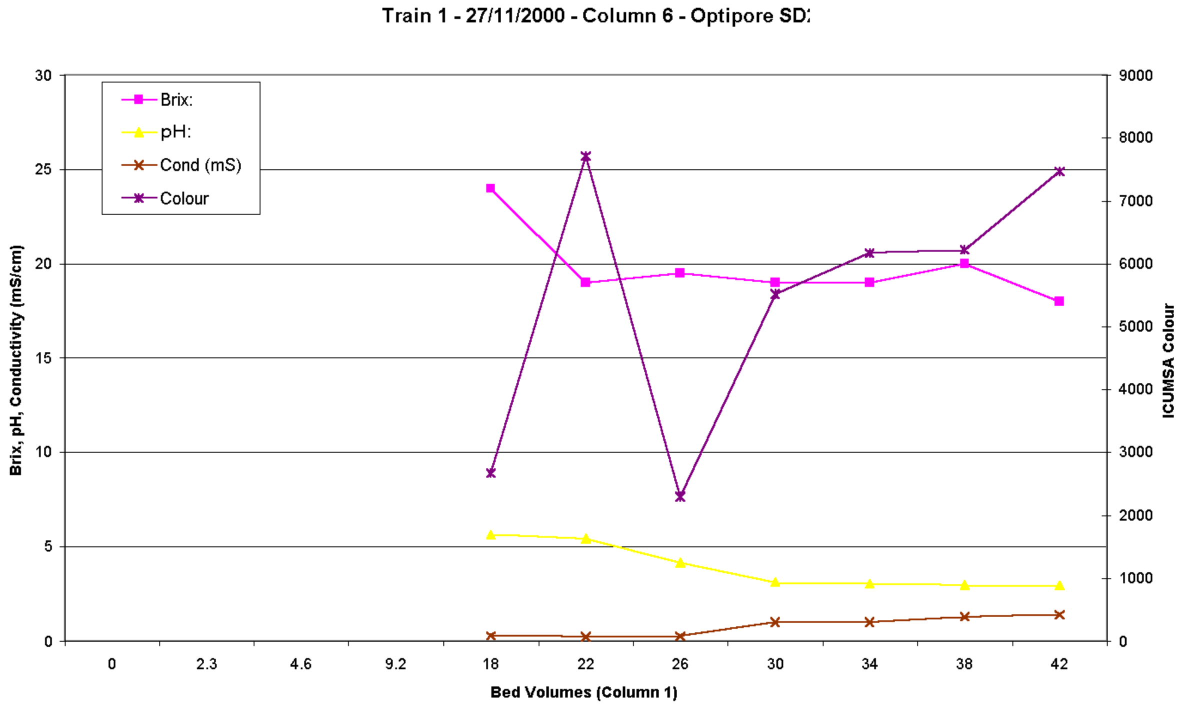


Fig 9.13: The breakthrough curve of Column 6 operating at 2BV/h.

It is noticeable during this second series of trials that the last Optipore column was responsible for the bulk of the decolourisation that was achieved. A possible cause for this was the inefficient regeneration of the resin in the primary columns. The irregular behaviour of the conductivity curve confirms this assumption.

9.5.2 Train 2

The results of the first experiment on Train 2 are reported in Table 9.6 and the breakthrough curves are presented in Figures 9.14 to 9.19.

Table 9.6: The results of the test on Train 2 operating at 2,3 BV/h.

Train 2 *Run at 2.3 BV/h*	***Ave Brix***	***Ave pH***	***Ave Cond mS/cm***	***Ave Col***	***Colour Load***	***BV's to Break-through***	***% Colour Removed***
Extract	35.3	4.78	4.956	30682			
Column 1 – Amberlite 252 H	24.7	2.63	2.760	34182	15980	Cond.: 55.2 Col: Cont.	-11.41
Column 2 – Amberlite IRA92	23.6	3.64	0.800	25086	16306	Cond.: 50.6 Col: Cont.	26.61
Column 3 – Amberlite 252 H	23.1	2.99	0.740	19283	11695	Cond.: None Col: Cont.	23.13
Column 4 – Optipore SD2	22.7	3.52	0.744	11983	13164	Cond.: N/A Col: Cont.	37.86
Column 5 – Amberlite IRA96	21.9	4.75	0.124	4976	11427	Cond.: None Col: 43.7	58.47
Column 6 – Optipore SD2	21.6	5.47	0.164	1510	5316	Cond.: N/A Col: 36.8	69.65

It can be seen that the last three columns again performed well in the removal of colour from the extract. The final colour obtained was comparable to that obtained with Train 1 under similar conditions. The de-ashing was also complete. The column capacity, measured as Bed Volumes to breakthrough was also comparable to that on Train 1.

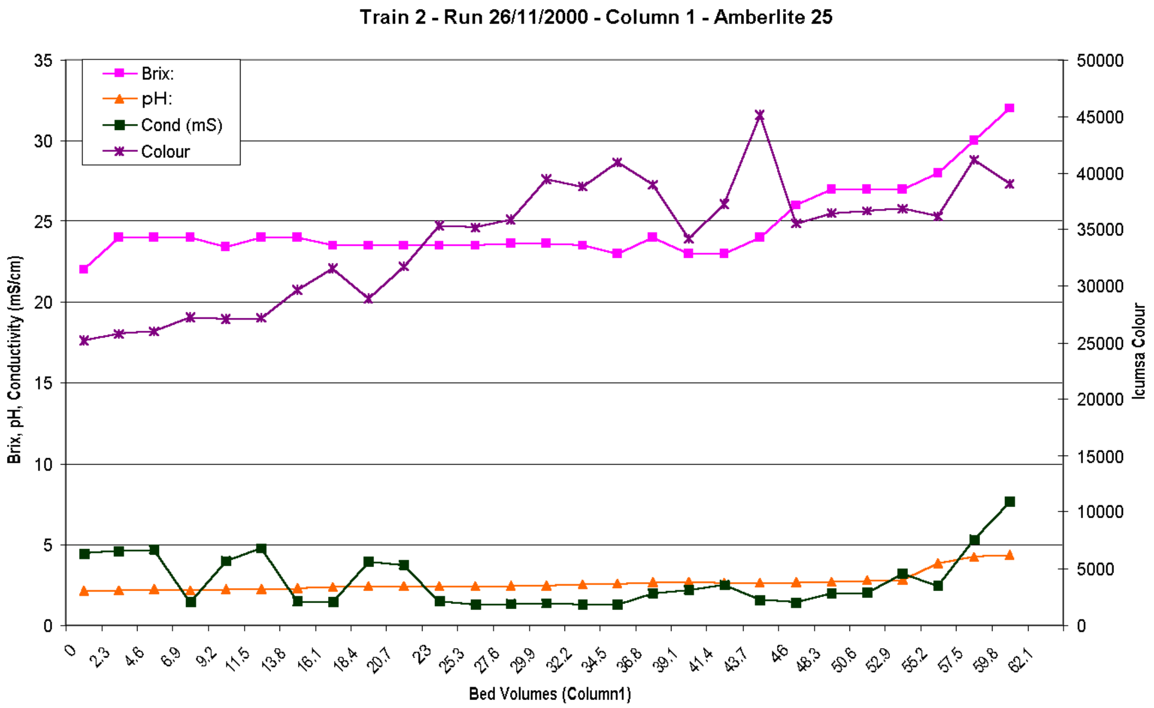


Figure 9.14: The breakthrough curves of Column 1 at 2,3 BV/h.

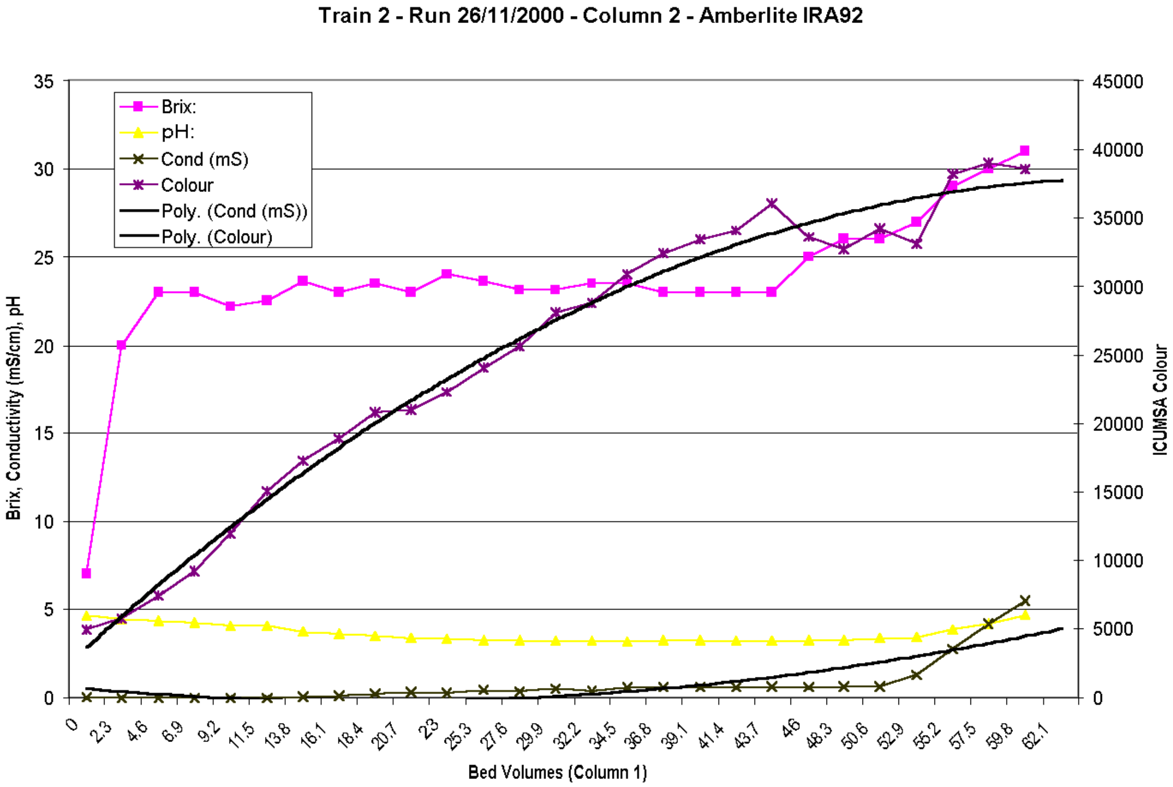


Figure 9.15: The breakthrough curves of Column 2 at 2,3 BV/h.

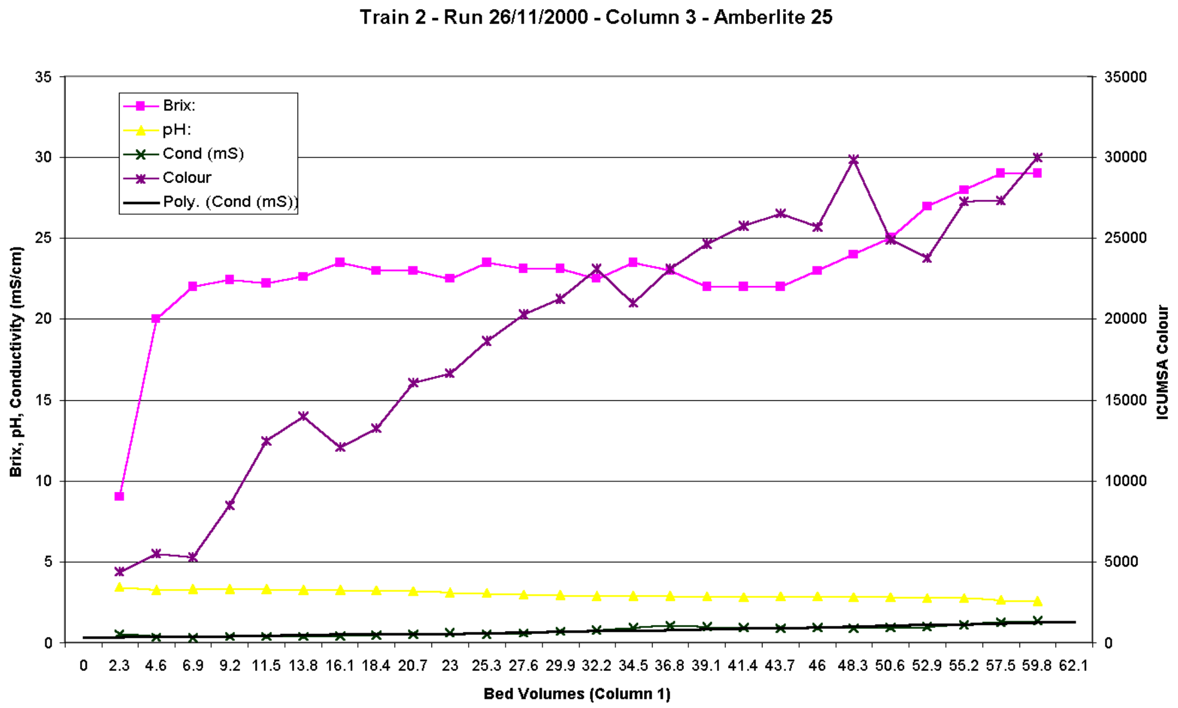


Figure 9.16: The breakthrough curves of Column 3 at 2,3 BV/h.

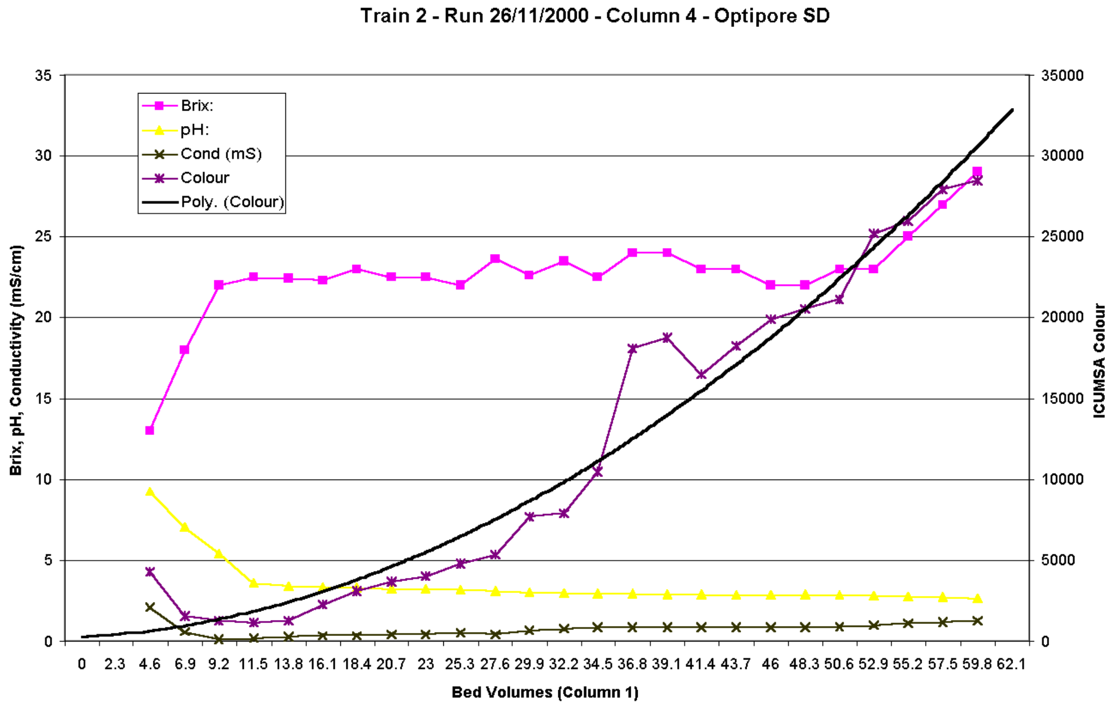


Figure 9.17: The breakthrough curves of Column 4 at 2,3 BV/h.

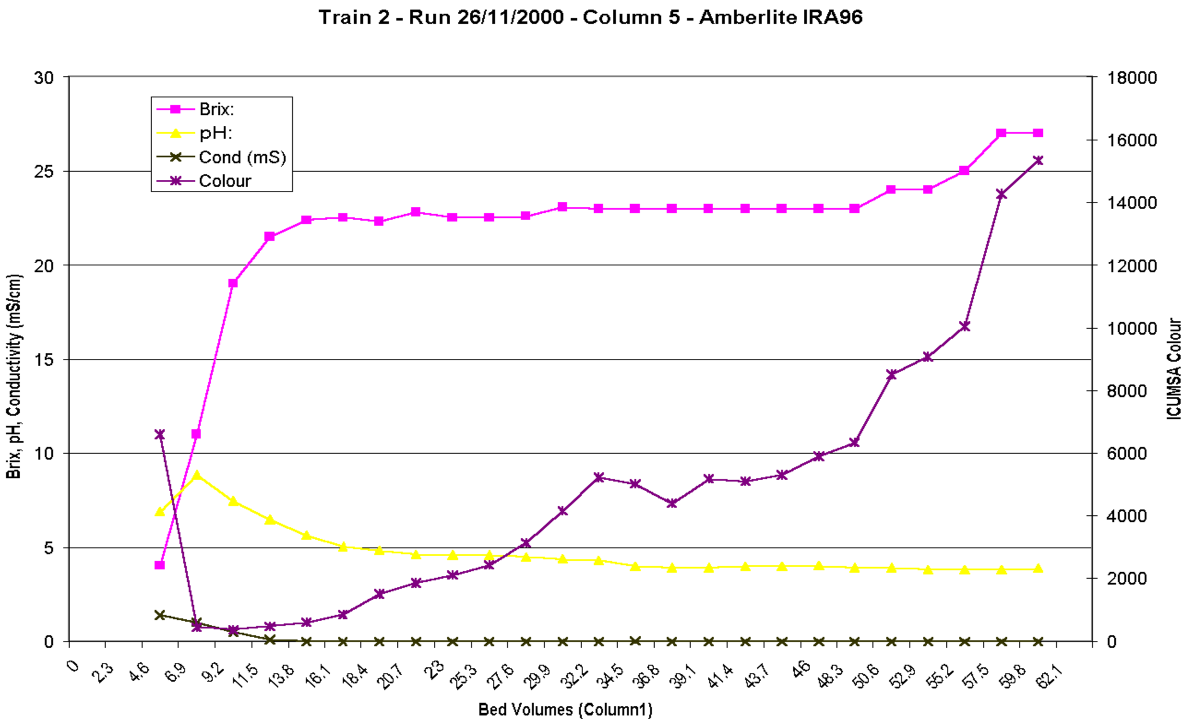


Figure 9.18: The breakthrough curves of Column 5 at 2,3 BV/h.

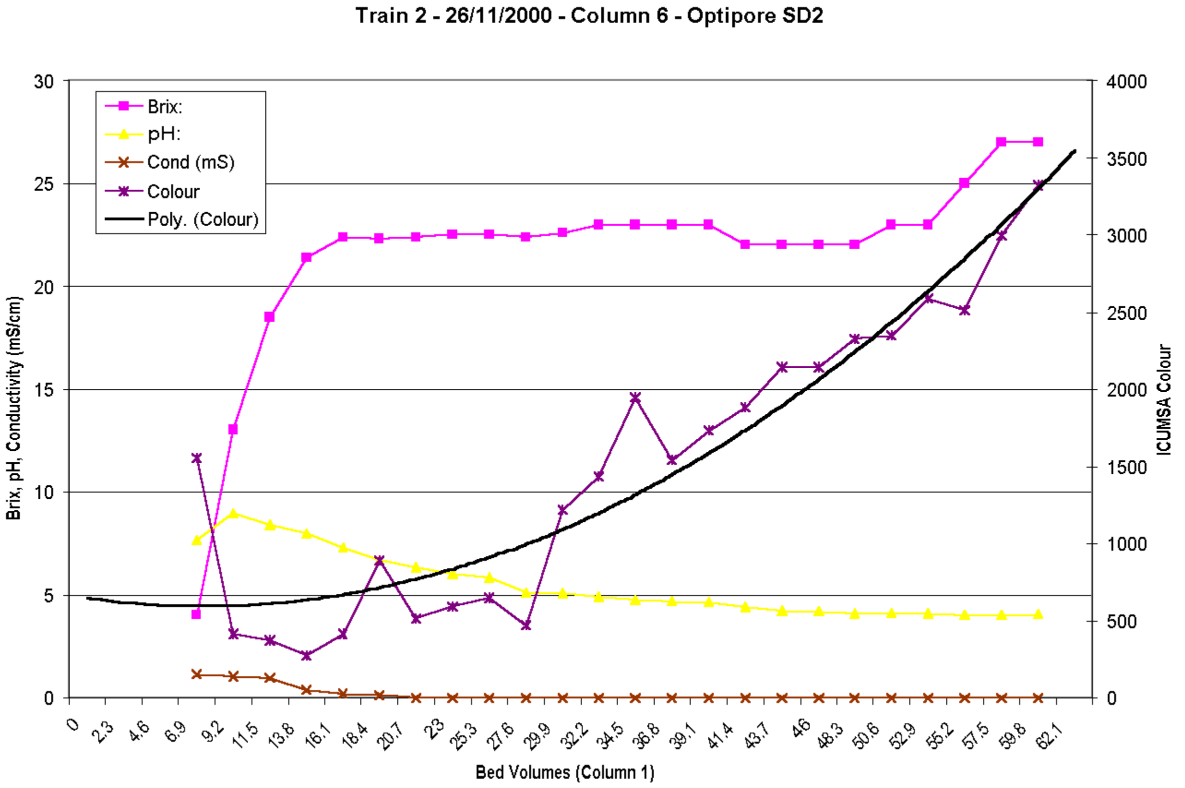


Figure 9.19: The breakthrough curves of Column 6 at 2,3 BV/h.

The flow rate was then reduced to 2 BV/h to see what the column performance would be, given that the similar test on Train 1 was very disappointing at the lower flow rates. The results of this second test are detailed in Table 9.7.

Table 9.7: The results of the second test on Train 2 with a reduced flow of 2.0 BV/h.

Train 2 *Run at 2.0 BV/h*	***Ave Brix***	***Ave pH***	***Ave Cond mS/cm***	***Ave Col***	***Colour Load***	***BV's to Break-through***	***% Colour Removed***
Extract	21.3	4.37	5.337	39027			
Column 1 – Amberlite 252 H	22.9	2.97	3.925	28859	-5140	Cond.: 42 Col: 34	26.05
Column 2 – Amberlite IRA92	21.9	3.71	4.804	22746	2843	Cond.: 42 Col: 34	21.18
Column 3 – Amberlite 252 H	20.4	3.21	0.787	16048	2793	Cond.: 30 Col: 38	29.44
Column 4 – Optipore SD2	19.4	3.67	0.776	10713	2705	Cond.: N/A Col: 38	33.25
Column 5 – Amberlite IRA96	21.4	4.75	0.222	5029	2947	Cond.: None Col: 28	53.05
Column 6 – Optipore SD2	20.1	5.85	0.263	1857	2222	Cond.: N/A Col: 28	63.07

The breakthrough curves are presented in Figures 9.20 to 9.25.

Figure 9.20: The breakthrough curves of Column 1 at the reduced flow of 2.0 BV/h.

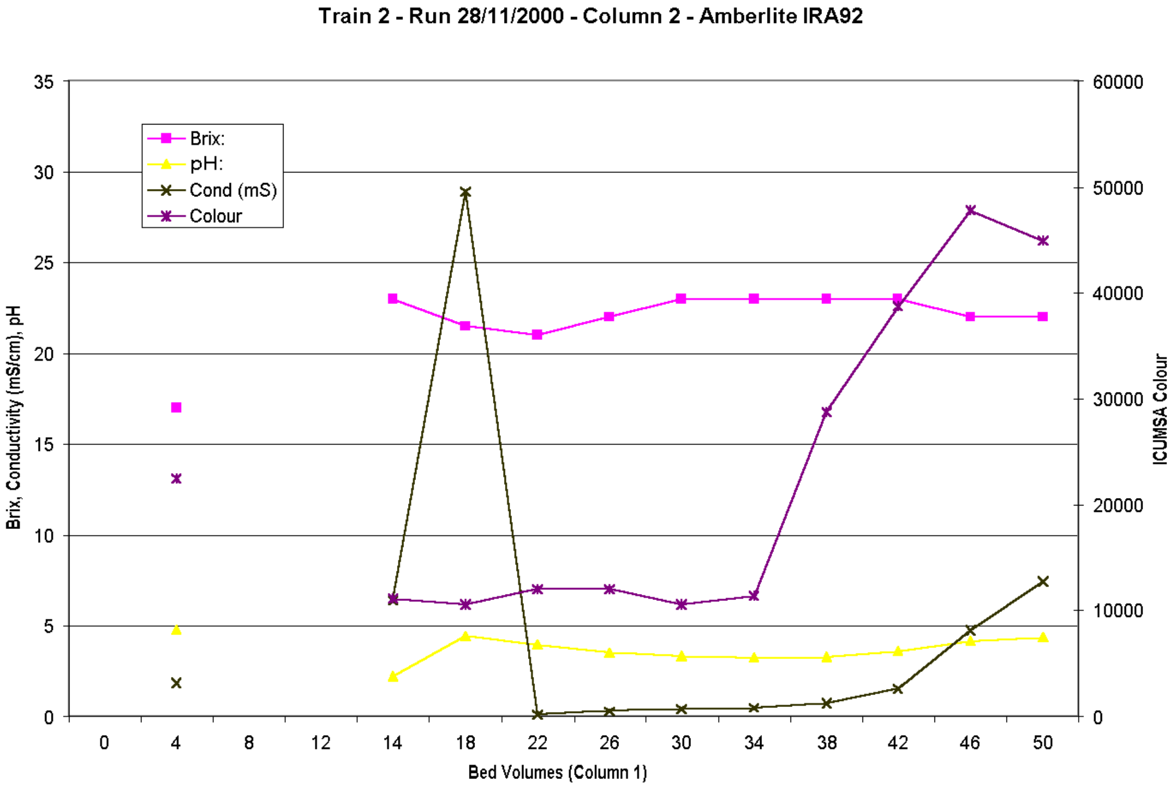


Figure 9.21: The breakthrough curves of Column 2 at the reduced flow of 2.0 BV/h.

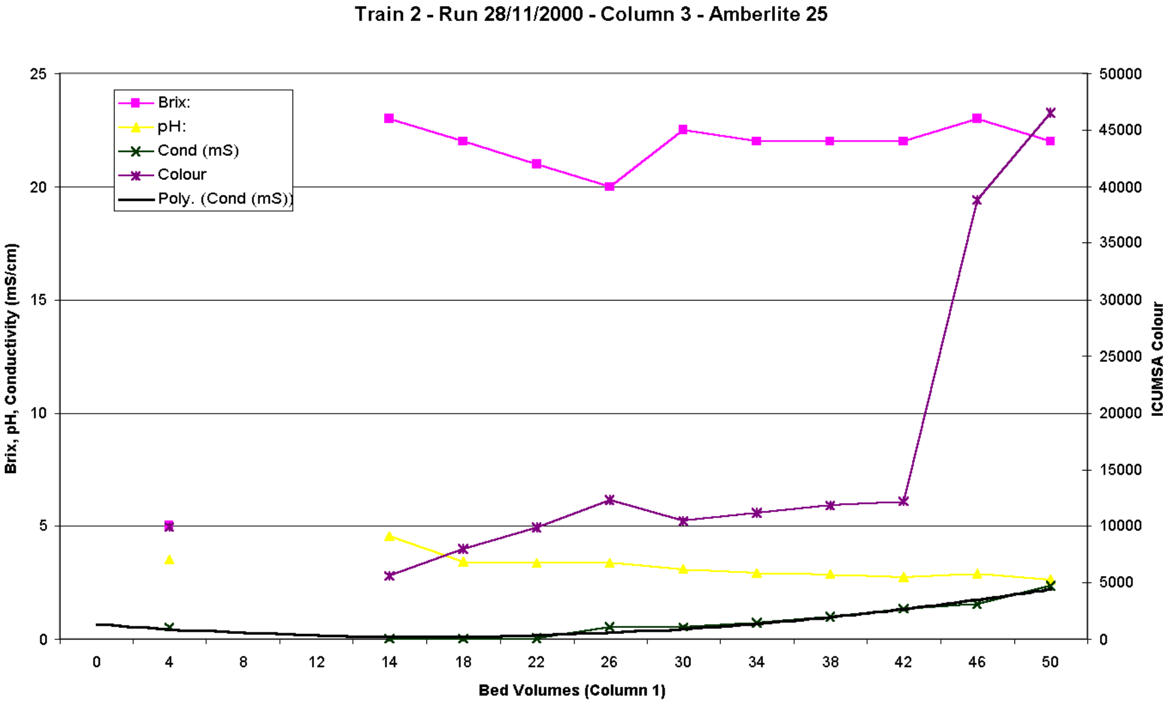


Figure 9.22: The breakthrough curves of Column 3 at the reduced flow of 2.0 BV/h.

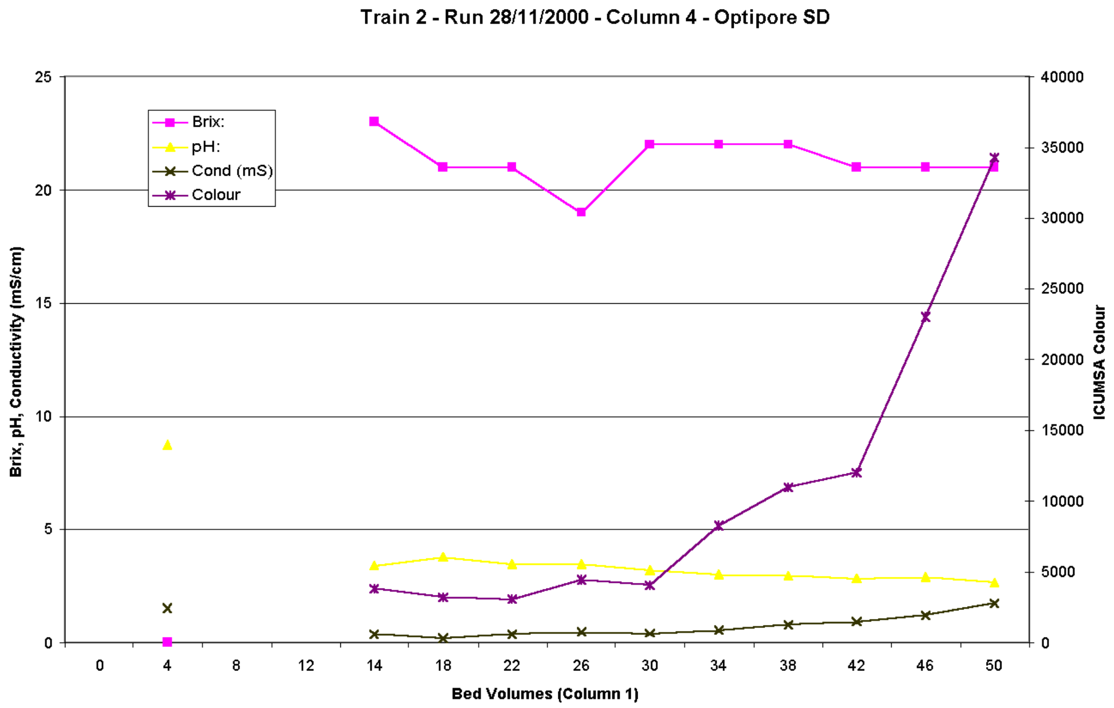


Figure 9.23: The breakthrough curves of Column 4 at the reduced flow of 2.0 BV/h.

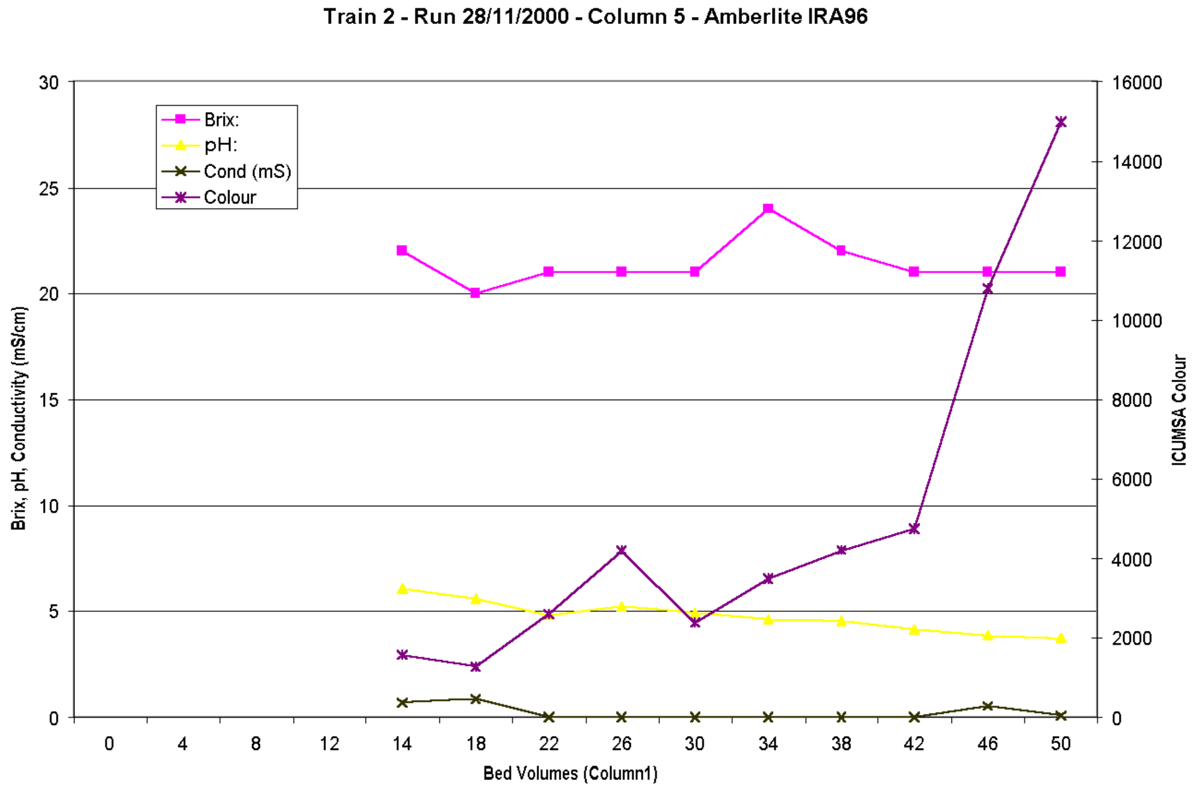


Figure 9.24: The breakthrough curves of Column 5 at the reduced flow of 2.0 BV/h.

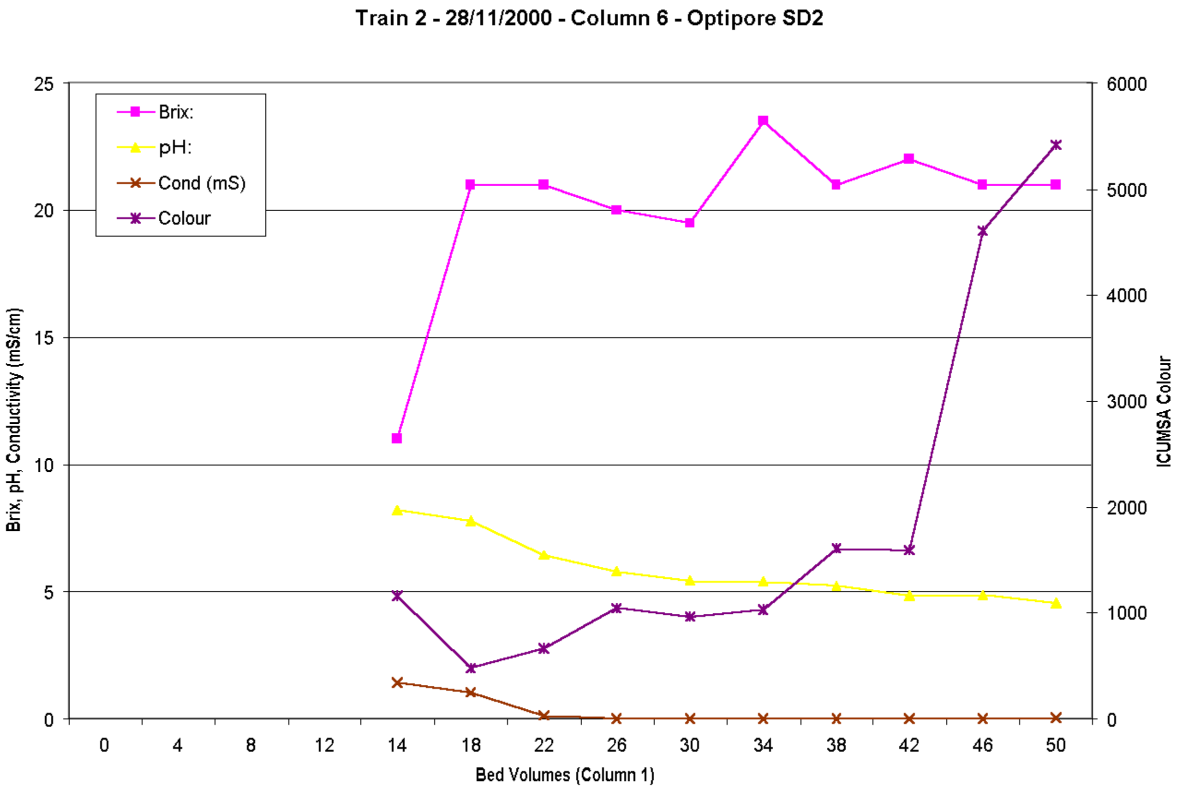


Figure 9.25: The breakthrough curves of Column 6 at the reduced flow of 2.0 BV/h.

The outlet colour was of acceptable quality and comparable to that of the runs at the higher flow rate. What was disturbing was the column breakthrough, which occurred earlier than the breakthrough when the columns were operated at the higher flowrate.

A possible explanation for this phenomenon lies in the flow configuration. For an ion exchange column that operates in up-flow, the whole resin bed is fluidised and is compacted at the top of the column. The resin is then exhausted in typical plug–flow “bands” spread throughout the resin (provided the liquid distribution is adequate). If the flow is not sufficient to completely fluidise the resin bed, mixing can occur causing early breakthrough. It was therefore suggested that the flow on the columns be changed to operate and regenerate in down flow direction only.

9.6. Derived Isotherms

Adsorption of molecules from liquid to solid is best described by the Freundlich isotherm. The Freundlich Isotherm assumes a single layer adsorption on the surface of the adsorbent. The following equation is derived from the equation presented by Chakrabarty (1991) and Coulson *et.al.* (1991):

$$\frac{x}{m} = K\,C^{1/n} \qquad (9.1)$$

Where: $\frac{x}{m}$: Amount of Adsorbed component per volume of resin.

K : Freundlich Constant or the specific capacity

C : Concentration of adsorbed component remaining in solution.

$\frac{1}{n}$: Freundlich Exponent or the measure of the energy of the sorption reaction.

By taking logarithms to the base 10 on either side of equation (9.1) and with rearrangement, the linear form of the isotherm can be derived:

$$\mathrm{Log}(x/m) = \mathrm{Log}(K) + (1/n)\,\mathrm{Log}(C) \qquad (9.2)$$

The values for K and 1/n can now be derived, where Log (K) is the intercept of the isotherm and 1/n is the slope.

The following Freundlich Isotherms were derived, using equation (9.2) for the final decolourising columns (Columns 4-6):

Table 9.8: The Freundlich Isotherms for the decolourising columns.

Train	***Date***	***Inter-val (BV)***	***Column 4 Optipore SD-2***		***Column 5 Amberlite IRA96***		***Column 6 Optipore SD-2***	
			1/n	**K**	**1/n**	**K**	**1/n**	**K**
1	24/11/2000	2	1.1776	0.2885	1.1010	1.3518	2.0898	6.0548E-04
1	27/11/2000	4	1.6716	1.9948E-04			0.8549	8.3811
2	26/11/2000	2	0.5691	102.2351	1.8292	1.2221E-03	1.9587	1.7799E-03
2	28/11/2000	4	0.5691	22.8981	1.0834	0.2430	0.9359	1.4612
Applex	July 2000	2	1.1592	116.8423	0.5104	7069.6840		

The isotherms agree with the experimental results more or less over the restricted concentration ranges, but they remain empirical. Being empirical, however, does not mean they cannot be used, for if the parameters of a reasonably reliable isotherm are known, then reasonably reliable results can be obtained for the extent of the surface coverage under various conditions. It is important to note that the flow-rate through the resin, and the temperature of the solution as well as the resin column, must be kept constant throughout the test. Maintaining flow-rate throughout the column was easily achieved with the advanced instrumentation on the chromatography plant. Temperature was controlled as best as possible, but variations did occur and may possibly explain some of the inconsistencies in the obtained results. It was also difficult to keep the extract composition constant and the range of values seen is typical of an industrial application.

9.7 Conclusion

Decolourisation of chromatographic extract through a series of ion exchange resins produced a product with an average colour of 1500 ICUMSA. This is a significant drop in colour, the colour of the liquid invert now being equivalent to industrial brown sugar. The colour characteristics of the molasses call for a very tedious decolourisation stage, which is expensive. If the market would accept a “darker” coloured product, the process could be simplified resulting in significant cost savings. The product is also completely ash free.

Furthermore continuous ion exchange on either a carousel or in a simulated moving bed (SMB) may possibly reduce the operating cost however at the higher capital cost of the more sophisticated equipment.

At this point it was felt that the development of the technology and the optimisation of the process had been completed. It was now possible to assess the feasibility of the process, on a commercial scale.

Chapter 10

Issues Regarding the Proposed Commercial Plant: Environmental Study, Conceptual Design and Financial Viability

10.1 Environmental impact Assessment

Environmental impact management should play a more significant role in all spheres of society. As determined in Schedule 4 of the Constitution of South Africa, 1996, the environment is a concurrent function of the relevant national and provincial departments. For the national and provincial environmental departments, a major role is to set specific regulatory norms and standards for impact management and to ensure that individuals and organisations meet these.

In order to provide for thorough and uniform control of the environmental impact of development projects, draft legislation for a compulsory environmental impact assessment (EIA) was published for comment on 4 March 1994 and an amended draft on 1 November 1997.

The Department of Environmental Affairs and Tourism published the Draft Guideline in September 1997 to coincide with the publication of the List of Activities and Regulations for EIAs in the Government Gazette of 5 September 1997. A Guideline Document has been published and is available on request from the various environmental departments to provide the various role players with a uniform basis for implementing the regulations.

The promulgation of legislation for a compulsory EIA may be regarded as a very significant step in formalising environmental impact assessment in South Africa and is in line with similar developments internationally. In this manner, the integration of environmental impact management with process development activities will be facilitated. This should lead to more responsible and environmentally sensitive process development.

The guidelines and the act call for specialised consultants to assist and partake in the setting up of an environmental management system. This chapter is a small part of the final EIA necessary for the molasses desugarisation plant. The author undertook the scoping of the EIA with the aid of the Guideline Document.

10.2 Project Phases of the EIA

For the purposes of assessing the various impacts, the project has to be divided into four phases from which impacting activities can be identified:

- Pre-construction phase

  This includes all activities on and off site up to the start of construction. This does not include the transport of materials, but does not include the initial site preparation.

- Construction phase

  All activities on and off site, including the transport of materials.

- Operational phase

  All activities, including operation and maintenance of structures.

- Decommissioning phase

  Any activity related to the physical dismantling of the structures.

The activities arising from each phase should be included in an assessment table. This is to facilitate the identification of those activities, which require certain management actions to mitigate the environmental impacts arising from them.

10. 3 Impact Assessment of the Proposed Invert Sugar Plant.

It is beyond the scope of this thesis to describe the full EIA for the plant. This chapter will only deal with specific aspects of the report, and is a short screening exercise done by the author. According to the Act, the EIA (or scoping) should be done by an independent specialist.

10.3.1 The Environment Concerned.

The proposed plant would be erected on or next to the premises of the existing Komati sugar mill. (See the map in Figure 10.1.)

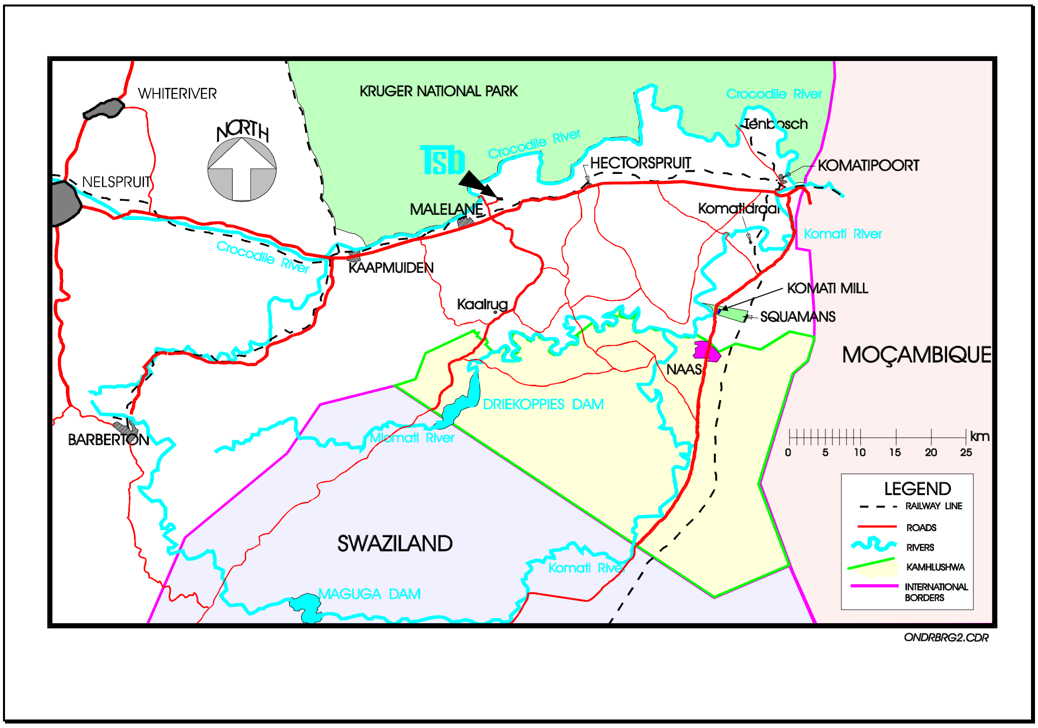


Figure 10.1 A map of the Onderberg region of Mpumalanga showing the two TSB mills and the larger towns in the area.

The mill is located in an area 32 kilometres south of Komatipoort, en route to the Swaziland border in the Mpumalanga Lowveld. It is about eight kilometres west of the border with Mozambique. The Komati Sugar Mill was erected on a farm and is surrounded with sugar cane farmland. The area is also extensively farmed with bananas and vegetables.

The closest residential area to the factory is the town of Naas, formerly part of the homeland of Kangwane. It is located five kilometres south of the factory. The area lies within the Komati River catchment area and sources most of its water from this river. The Komati River is a tributary of the Crocodile River and the two rivers join at the South Africa-Mozambique border. In Mozambique the river is known as the Inkomati River. There are numerous unspoilt wilderness areas in the region and there is a thriving tourist industry. It is also the last habitat of one of the most endangered species of cycads in the country.

The Invert Sugar plant would not extend the existing agricultural farmland. The final expansion of land for farming was accomplished with the completion of phase two of the Komati Mill Project. The plant would also not be erected outside the already industrial zoned area of the existing mill.

10.3.2 The Activity to be undertaken.

The Invert Sugar Plant would utilise between 76000 – 85000 mt of molasses from the Komati Mill per year to produce liquid or crystalline sugars. Installed capacity will be 87 400 mt/a.

The plant would require electricity and water, both of which would be supplied by the existing Komati Mill.

10.3.3 The Impacts Identified

10.3.3.1 Air Pollution

No significant increase in air pollution is foreseen due to the fact that the electricity and steam for the plant would be supplied from the existing factory. No additional boilers are needed. The existing factory is energy efficient and is presently producing excess electricity, which is channelled into the Escom national grid. It is anticipated that the new plant would absorb part of this excess capacity. The plant will require a bagasse boiler for the off-season, generating excess “green energy” that can be supplied to the national grid.

The process produces no fumes other than water vapour and this constitutes no threat to air pollution.

10.3.3.2 Water Pollution

The water balance of the process indicates a process water requirement of 13,5 mt/h to the plant. The existing Komati factory is operating at a positive water balance and no additional water from the Komati River would be needed to operate the plant.

Liquid effluent from the plant consists of a salt solution. This salt solution would be concentrated and the water recovered. The remaining solution is rich in, amongst others, potassium and is suitable to be used as a liquid fertiliser on the surrounding farmland.

The plant will be situated more than one kilometre inland from the bank of the nearest river. Spillage of chemicals or products within the perimeter of the plant would not lead to immediate, if any, contamination of the water source.

Uncontained spillage of flocculant in the water source would not result in significant long-term effects since it would coagulate organic material in the water and settle to the bottom. The flocculant is biodegradable and poses no threat to the environment.

Uncontained spillage of acids would be neutralised by adding dilute caustic soda solution to the water. The resulting neutral water would contain elevated amounts of sodium salts. The salt dissolves easily and significant dilution is available to prevent any lasting environmental impact. Spillage of caustic soda would similarly be treated with hydrochloric acid.

Uncontained spillage of invert sugar would lead to a momentary increase in the Chemical Oxygen Demand (COD) and the Biological Oxygen Demand (BOD) of the water sources. Glucose and fructose are the basic monosaccharides used in the metabolism of organisms. A release of these monosaccharides would cause a momentary increase in micro-organisms in the environment, typically bacteria and fungi, to ferment these sugars. Sufficient dilution is available to dissipate the compounds quickly in the water system. The river has a healthy aquatic microbe population that would absorb the spillage without causing a significant shock to the river system.

The existing effluent plant at the Komati Mill would treat sewage and storm water effluents.

10.3.3.3 Soil Pollution

Solid waste produced by the factory can be divided into two parts: Domestic Waste and Expelled Process Solids.

The plant's contribution to the Domestic Waste of the Komati Mill would be minor. The plant would have a minor increase, if any, on the amount of personnel employed at the mill. The existing Waste Disposal infrastructure at the Komati Mill and the expanded dumpsite would not be taxed by inclusion of the Invert Plant.

Expelled Process Solids are rich in plant organic and inorganic material and makes excellent compost. In the event of filter powder being used this would be added to the solids. Farmers from the area collect the cane factory's filter mud for fertilisation of their cane fields. The solids generated by the Invert Plant could be added to the filter mud without extensive plant modifications and with no detrimental effect on the environment. Excess could be taken to the factory's dumpsite.

10.3.3.4 Light Pollution

The new plant will be an addition to the existing plant. There are no nature conservancies, or tourist attractions in the immediate vicinity that could be influenced by the addition of artificial light. The present mill is close to a town and there is no effect due to the lighting presently being used. In all, only a new flood mast would be added to the skyline. It is therefore felt that the additional light emitted from the expansion would not cause any significant increase to the luminous intensity of the light already being emitted from the present plant.

10.3.3.5 Visual Appearance

The architecture of the existing Komati Mill is pleasing to the eye. The new plant would fit in with the existing structure and architecture. No part of the plant would be higher than the structure already existing. The skyline of the present plant would therefore not be affected.

10.3.4 Determination of significance

The significance of each type of pollution is indicated in the table below.

Table 10.1 The significance of each type of pollution due to an Invert Plant.

	Low	**Medium**	**High**
Water Pollution		X	
Air Pollution	X		
Soil Pollution	X		
Light Pollution	X		
Visual Appearance	X		

The only source of pollution that could have an effect on the environment is water pollution.

10.4.5 Mitigation

It is clear from the identified impacts and the probabilities study that the measures taken to protect the environment are adequate. The mitigation of environmental hazards was part of the original design. Therefore no further mitigation steps are required to enhance the environmental acceptability of the plant. However proper training and environmental awareness of personnel in all aspects related to the process will be essential. Consultation with all interested and affected parties will be an ongoing process.

10.4 Conceptual Design of a Commercial Invert Sugar Plant

To enable a reliable cost estimation, a conceptual design of a plant was required. This design included the mass and energy balances to provide a basis for the sizing of equipment. Optimisation regarding water and energy usage also formed part of such a design. Important information relating to the environmental impact assessment was extracted from this conceptual design.

For the purpose of the feasibility study detailed later in the chapter, the conceptual design of the Invert Sugar Plant related to a design undertaken during the first part of the project. The final flow diagram and economics changed in the latter part of the project. The new plant design would use a clarifier to settle suspended solids and would use a filter as a polishing process. Molasses would then pass through a membrane filter before softening. The decolourisation section also changed. The molasses carification process was patented [Bekker PI, Stolz HNP, *et.al*. (2001)] and the new plant was designed according to the new process. Further refinements regarding the process and information pertaining to the design of the new plant is proprietary information and is in the process of international licensing, but the reader can discover much of the detail in the relevant chapters of this thesis.

Water and energy are scarce and expensive commodities. Proper integration of these into the process would reduce capital and operating cost and could yield a plant that has less impact on the environment.

10.5 Initial Mass Balance

The mass balance was derived from an Excel calculation workbook and some SUGARS^{TM} simulation calculations. SUGARS^{TM} simulations are described in several papers (Stolz and Weiss (1997); Alvarez *et.al.* (2000))

The mass balance presented here still uses a centrifuge as primary clarifier. The initial process economics were based on this model. The input and output flows obtained from the balance and associated costs for the intended invert sugar plant are shown in Table 10.2.

Table 10.2: The flow and associated cost of the streams entering and leaving the plant.

Molasses Pre-treatment										
Unit Operation / Operating Step	**Flowrate**		**Flowrate**		**Based on DS**		**Cost per Unit**		**Cost/t DS**	**Unit**
Component	**IN**	**Units**	**OUT**	**Units**	**Value**	**Unit**	**Value (R)**	**Unit**		**Sub-total**
Dilution										**192.92**
Molasses	13.7	t/h			1.2	t/tDS	155.00	t	192.67	
Water	5.6	t/h			0.5	t/tDS	0.50	t	0.25	
pH Adjustment & Inversion										**21.49**
H3PO4 (100%)	33.0	kg/h			3.0	kg/tDS	4.90	kg	14.70	
Enzyme (HIY)	10.7	kg/h			0.97	kg/tDS	7.00	g	6.79	
Dilution										**1.75**
Water	38.6	t/h			3.5	t/tDS	0.50	t	1.75	
Chemical Addition										**14.70**
NaOH	55.1	kg/h			5.0	kg/tDS	2.94	kg	14.70	
Flocculant	1.3	kg/h			0.1	kg/tDS	0.03	kg	0.00	
Centrifugal Separation										**0.02**
Wash Water	0.5	t/h			44.2	kg/tDS	0.0005	kg	0.02	
Sludge			3.7	t/h	0.3	t/tDS		t	0.00	
Filtration										**77.79**
Wash Water	8.7	t/h	8.7	t/h	0.8	t/tDS	0.50	t	0.39	
Filter Powder	231.6	kg/h			21.0	kg/tDS	3.68	kg	77.40	
Filter Cake			514.8	kg/h	46.7	kg/tDS		kg	0.00	
Softening										**1.94**
Regen Chemicals	3.2	t/h			0.29	t/tDS		t	0.00	
Spent Regen Chemicals			3.2	t/h	0.29	t/tDS		t	0.00	
Electrical Power - Pretreatment	305.1	kWh			27.7	kWh/t DS	0.07	kWh	1.94	
Concentration										**32.95**
Steam	13.4	t/h			1.2	t/tDS	27.00	t	32.95	
Vapour			38.6	t/h	3.5	t/tDS		t	0.00	
Condensate			13.4	t/h	1.2	t/tDS		t	0.00	
Chromatography										**7.48**
Water	37.7	t/h			3.4	t/tDS	0.55	t	1.88	
Raffinate			33.9	t/h	3.1	t/tDS		t	0.00	
Resin Make-up	3.1	litre/h			0.28	litre/t DS	15.75	litre	4.41	

Molasses Pre-treatment

Unit Operation / Operating Step	**Flowrate**		**Flowrate**		**Based on DS**		**Cost per Unit**		**Cost/t DS**	**Unit**
Component	**IN**	**Units**	**OUT**	**Units**	**Value**	**Unit**	**Value (R)**	**Unit**		**Sub-total**
Electrical Power	154.2	kWh			14.0	kWh/t DS	0.07	kWh	0.98	
NaOH (100%)	0.55	kg/h			0.05	kg/t DS	2.94	kg	0.15	
HCl (100%)	0.55	kg/h			0.05	kg/t DS	1.29	kg	0.06	
Demineralisation and Decolourisation										**42.04**
HCl (30%)	0.2	t/h			18.4	kg/tDS	1.18	kg	21.69	
NaOH	0.1	t/h			5.8	kg/tDS	2.94	kg	17.10	
Water	10.8	t/h			1.0	t/tDS		t	0.00	
Combined effluents			8.8	t/h	0.8	t/tDS		t	0.00	
Resin Make-up	1.4	litre/h			0.13	litre/t DS	25.00	litre	3.25	
Electrical Power										
Concentration										**15.48**
Steam	6.2	t/h			0.6	t/tDS	27.00	t	15.29	
Vapour			13.4	t/h	1.2	t/tDS		t	0.00	
Condensate			6.2	t/h	0.6	t/tDS		t	0.00	
Electrical Power	28.6	kWh			2.6	kWh/t DS	0.07	kWh	0.18	
Raffinate Concentration										**12.15**
Steam	4.8	t/h			0.4	t/tDS	27.00	t	11.70	
Vapour			16.9	t/h	1.5	t/tDS		t	0.00	
Condensate			4.8	t/h	0.4	t/tDS		t	0.00	
Electrical Power	71.7	kWh			6.5	kWh/t DS	0.07	kWh	0.46	
							Total Cost R/tDS			**420.72**

The mass flow through the proposed plant is graphically depicted in the Process Flow Diagram presented in Figures 10.2-10.4.

Molasses Pretreatment

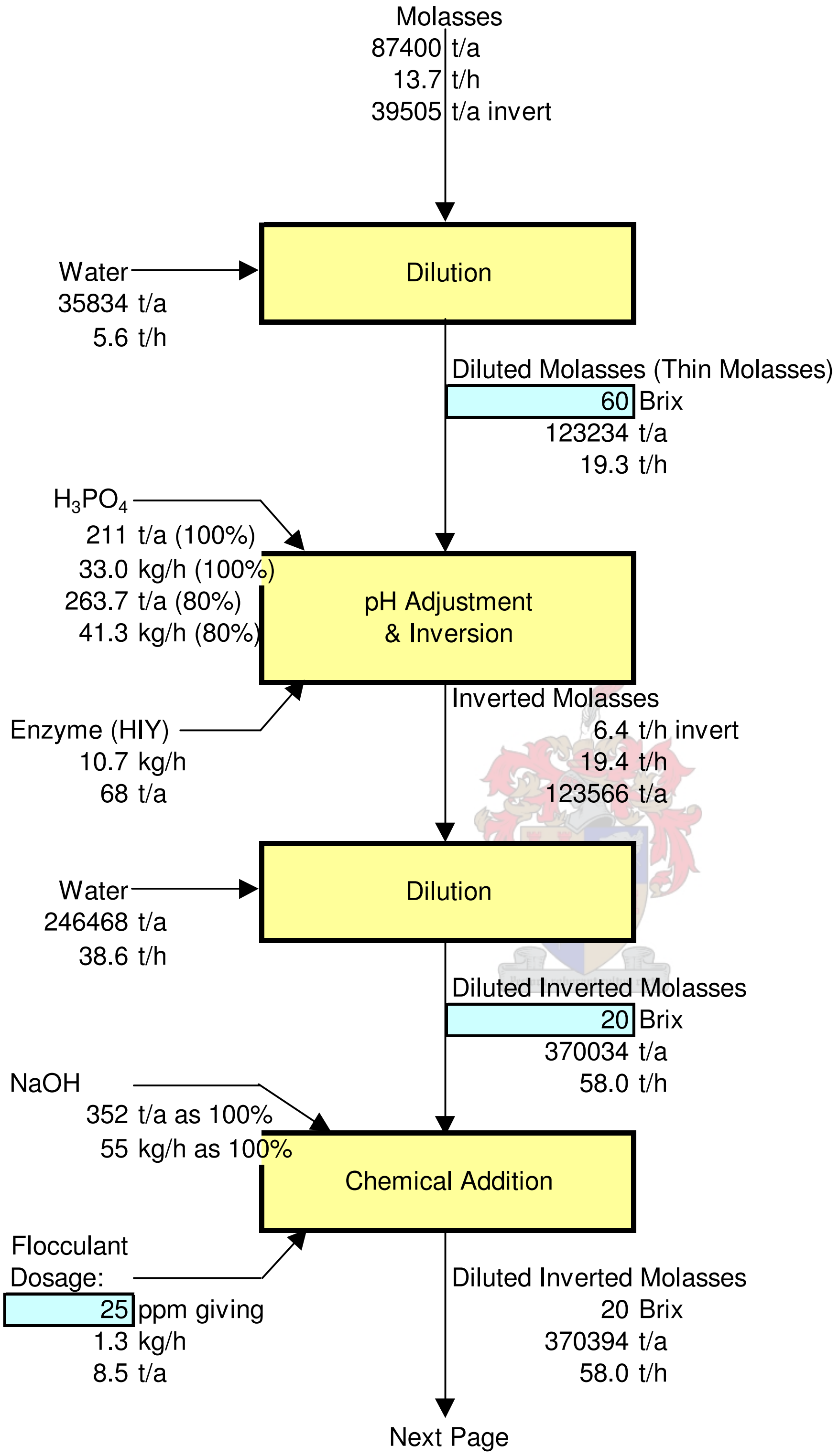


Fig 10.2: The Mass Balance of the proposed Invert sugar plant (Part 1)

Molasses Pretreatment

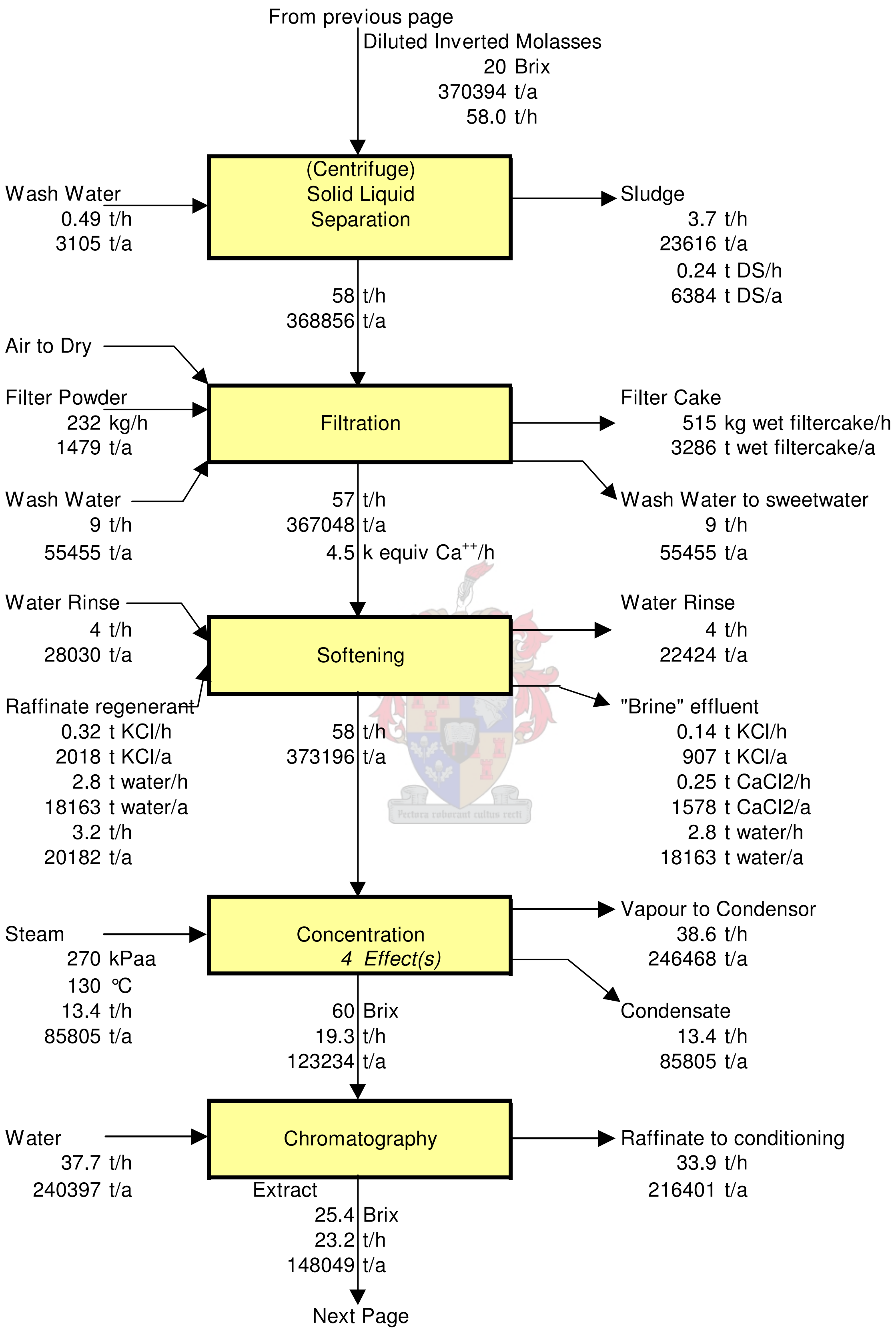


Fig 10.3: The Mass Balance of the proposed Invert sugar plant (Part 2)

Molasses Pretreatment

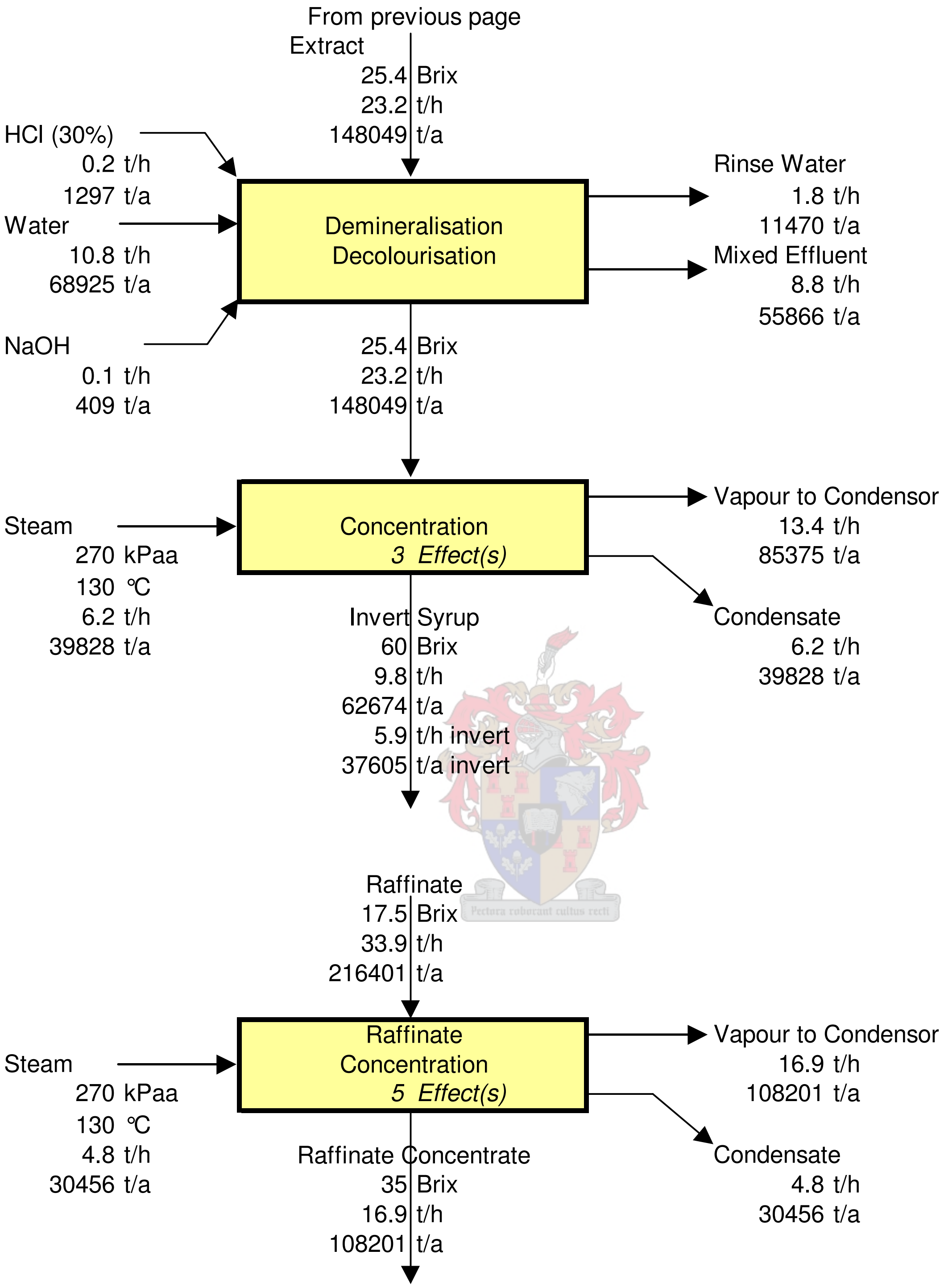


Fig 10.4: The Mass Balance of the proposed Invert sugar plant (Part 3)

10.6 Water and Energy Integration

Water is of prime importance in the South African context, as the country does not have an unlimited supply available. It is of more importance for the Invert Sugar plant since an industrial quota is not available in the proposed area of construction. The area is also environmentally sensitive. Even though the proposed plant is located in a subtropical rainfall area, the main water source that could be utilised for industrial applications is river water from the Komati River. Pinch techniques were used to determine theoretical minimum requirements for the plant. The water distribution around the factory is listed in Table 10.3 below:

Table 10.3: The results of the water "pinch" and the water distribution around the plant.

Molasses Pretreatment

WATER RETICULATION – Phase 1

Water Consumers	t/h	Water Producers	t/h
Dilution 1	5.61	Dilution 1	0
Dilution 2	38.61	Dilution 2	0
Centrifuge	0.49	Centrifuge	0
Filter Wash	8.69	Filter Wash	8.69
Softening Rinse	0.88	Softening Rinse	0.88
Concentration	0	Concentration Vapour	38.61
Chromatography	37.66	Chromatography	0
Demin/Decolour	1.80	Demin/Decolour	1.80
Concentration Extract	0	Concentration Extract	13.37
Concentration Raffinate	0	Concentration Raffinate	16.95
	93.7		80.29
Nett Consumption:	**13.4**	**t/h Costing R 6.70/h**	

The distribution of water through the plant is indicated in the block flow diagram in Figure 10.5 below.

The theoretical minimum energy requirement for the plant was calculated as: 8.98 MW or the equivalent of 14.87 t/h steam at 270 kPaa and 130°C. The procedure used to determine this was similar to that used for water. The practical application of the "pinch" result fails to supply enough heat to the plant to maintain adequate heating of the process without elaborate piping and heat exchanger networks. The optimum energy "pinch" in this case could not be implemented. It is proposed to maintain an adequate steam supply to each of the evaporation stations individually. Energy from a raw-sugar factory is competitively priced and is usually available in excess quantities.

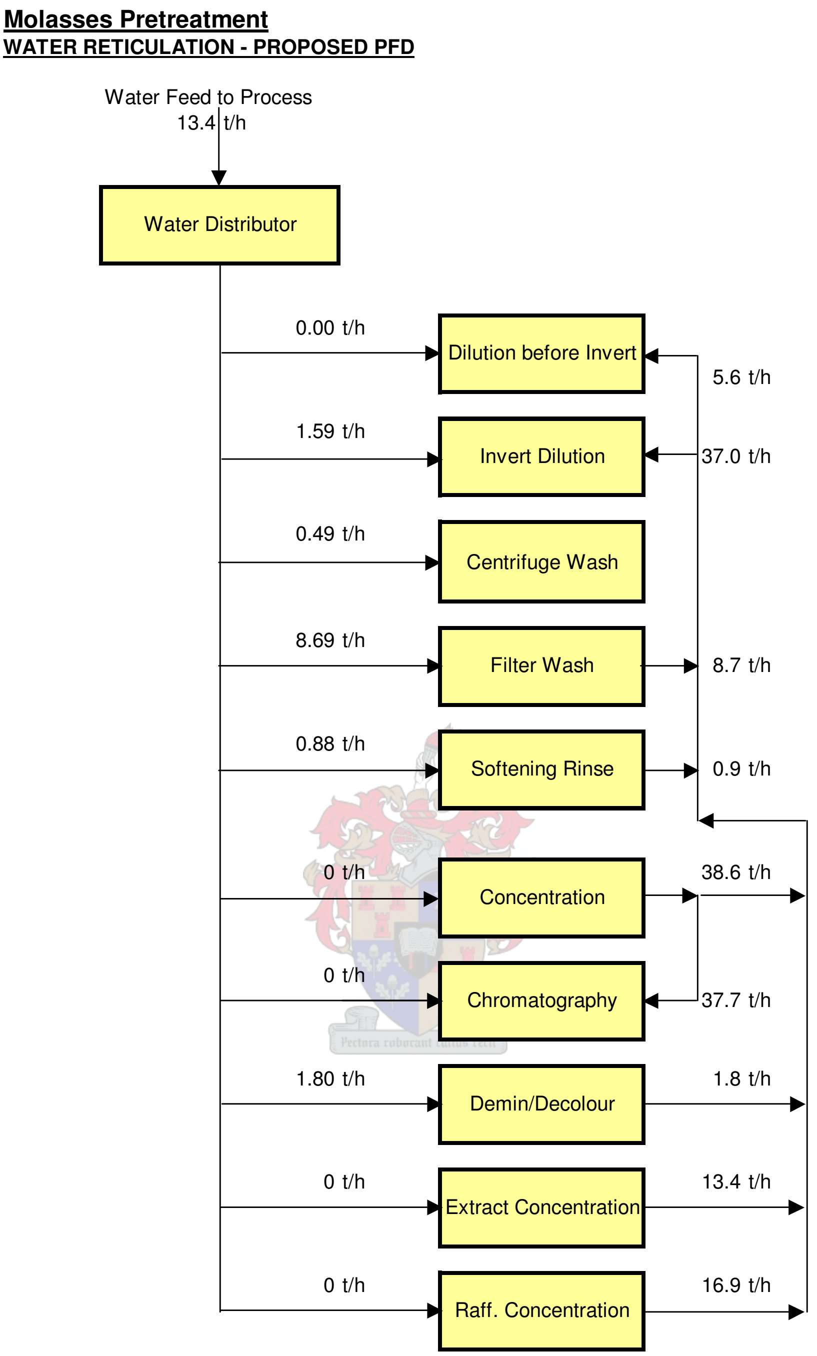


Figure 10.5: The process water reticulation around the plant.

Komati Mill generates its own electricity from the combustion of the cane bagasse. The mill is a producer of electricity and exports to the Escom grid. Excess low-pressure steam is condensed in a "waste heat evaporator". In this evaporator, raw river water is evaporated to condense the extra steam and to return the "waste heat" condensate to the boiler feed water tank. Utilizing this energy source to add value to the existing process is a logical and viable approach.

10.7 The Mass Balance including the E-Cat™

The following mass balance was complied so as to accommodate the E-Cat™ clarifier into the plant design. A condensed flow sheet listing the daily flows is shown in Figure 10.6.

The detailed flow diagram used in the “SUGARS™” simulation of the plant is attached in Appendix F. With the capacity and flow data available, the next step in the feasibility study could be addressed – the process economics.

10.8 Financial Viability of a Commercial Invert Sugar Plant

It usually takes a long time from the conception of an idea to the final erection and commissioning of a production plant. One of the first steps in the project is to evaluate the profitability of the process. In the private sector, plants are built to make a profit. The risks and capital requirements are high. It is therefore of utmost importance that all the design and financial calculations, from the very beginning, are as accurate as possible. Ward (1989) and IDC Technologies (2000) reported on a variety of techniques to assist in the determination of profitability.

One should always bear in mind that factors in industry and the market may change such as: a change in demand, changes in technology to produce the same product, and cheaper substitutions for the product. Forecasting is therefore a hazardous task. Ulrich (1992) proposed a different approach to minimise the guesswork normally associated with costing a new plant.

As the project progresses, it is important to correlate the reality with the original predictions. It is better to halt a project in time, than to build a plant that cannot make a profit.

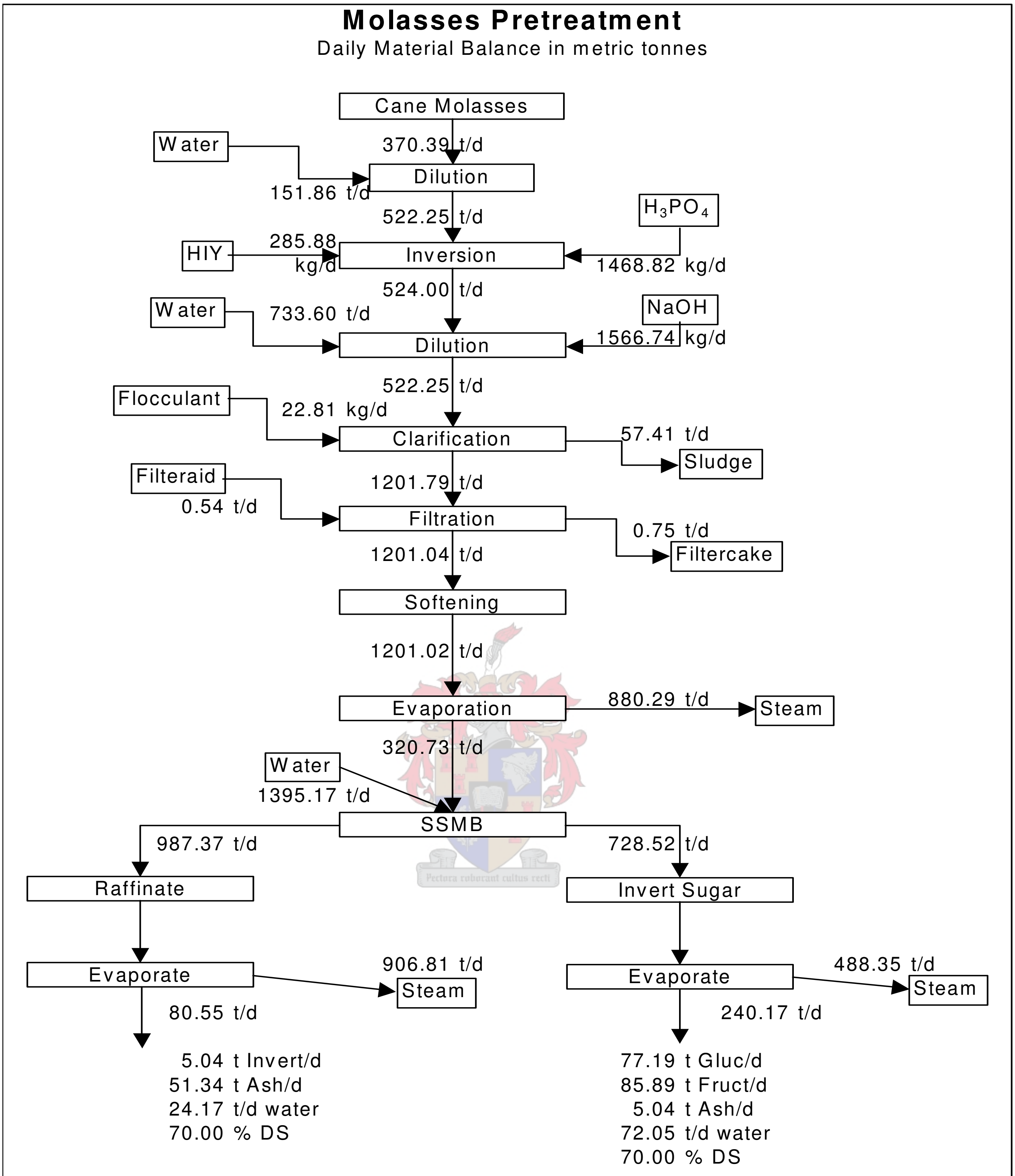


Figure 10.6: Condensed process flow diagram showing the key figures

10.9 Cost Estimation for the Invert Syrup Plant

The plant is intended to be an expansion of the Komati Mill (brown-field development). Infrastructure and land is therefore available and not included in the estimations. The cost estimation for the invert plant was made in early 2000 and was adjusted to a

2004 value. Expansion to the Komati Mill has just been completed and this project was a reliable source of cost data. The mill has been in operation since 1994 and most of the operating costs are well established.

10.9.1 Estimated Capital Cost

Applexion supplied cost data[1] on the chromatographic separation unit for a 100,000-tpa plant (converted from French Franc to USD). This estimate uses a Rand-Dollar exchange rate of R6.30/US$. The estimate included the cost for centrifuges to clarify the molasses; the use of a clarifier was still being investigated at the time. The centrifuges were priced at US$1,000,000.00[2]. An Eimco E-Cat Clarifier was priced at R1,800,000.00[3] for a 10m-diameter unit. There are therefore considerable savings to be made by using a clarifier rather than centrifuges

The estimated capital cost for the plant is presented in Table 10.4. This estimate is related to the Guthrie method, but the ratios used to calculate the additional items were also based on the figures for the Komati Mill Expansion in 1999. These represented existing South African conditions for a factory expansion.

The estimate should be regarded as provisional with an accuracy of -10% and +20%.

10.9.2 Estimate of Operating Cost

Due to the simple nature of the process, no additional personnel will be required and the Komati Mill would operate and maintain the plant. Purchases, despatch and financial services will also be integrated into the existing Komati Mill structure.

Protea Industrial Chemicals, NCP Chloorkop, Chemserve, Montan Chemicals and Rohm & Haas provided the prices for bulk chemicals delivered to the Komati Mill. Utilities such as electricity, water, sludge disposal etc. were extracted from actual mill figures as explained previously.

It is assumed that the technology supplier will supply the first batch of resin - this is included in the capital cost estimate. Resin replacement is provided for in the

---

[1] Applexion: personal correspondence

[2] α-Laval and GEA Westfalia: personal correspondence

[3] Baker Hughes: personal correspondence

operating cost estimate. Table 10.5 below lists the operating requirements and the process costs.

Table 10.4: The Estimated capital cost of the invert sugar plant.

Estimated capital cost

	Molasses pre-treatment	**US$'000**	
1	Dilution, heating	45	
2	Centrifugation machines	1,000	
3	Softeners & resins 2 columns	337	
	Molasses Inversion		
4	Inversion tanks & stirrers 8 tanks	293	
5	Molasses conditioning	35	
	Chromatographic separator		
6	Train A with 4 columns & 275 m3 resins	3,450	
7	Molasses % elution water degassing	inc.	
8	Resins service tank & pumps	inc.	
	Glucose-fructose demin. & decolor.		
9	IX plant 6 columns & 56 m3 resins	871	
10	Regeneration equipment	inc.	
	Glucose-fructose evaporator		
11	3 effects & thermo plate evaporator	378	
	Raffinate evaporator		
12	5 effects & thermo tubular evaporator	970	
	Sub -Total Equipment	7,379	
	Additional budget estimate		
13	Instrumentation & valves	664	9.0%
14	Piping	738	10.0%
15	Civil Works	517	7.0%
16	Electrical plant	812	11.0%
17	Building	369	5.0%
18	Insulation & painting	295	4.0%
19	Insurance	37	0.5%
20	Contingency	1,476	20.0%
21	Inspection	30	
22	Intellectual property	800	
23	Project Engineering	1,847	15.0%
	Total estimated capital cost	***14,963***	USD
		94,270	R'000
	Depreciation rate	10.0%	

The price for electricity was the internal purchase price from the factory's power station. Low-pressure steam would be used for heating and evaporation. The cost of steam is estimated at 80% of the generation cost of steam using coal as fuel, and was determined as R22.00/t.

Table 10.5: Operating requirements and costs for the invert sugar plant.

Element	Unit	R/Unit	Units / tDS	R / tDS
Molasses	ton	165.00	1.24	204.6
Molasses pre - treatment & Inversion				**84.83**
H_3PO_4(100%)	kg	4.90	3.00	14.7
NaOH(100%)	kg	2.94	5.00	14.7
Electricity	kWh	0.07	23.70	1.659
Water soft	m^3	0.55	0.75	0.4125
Steam	ton	22.00	1.00	22
Resin Softening Cation	litre	15.50	0.08	1.24
Enzyme	kg	7.00	0.97	6.79
Flocculant	g	0.03	105.00	2.73
Filter aid	kg	3.68	5.23	19.2464
Electricity	kWh	0.07	4.00	0.28
Sludge disposal	ton	2.50	0.43	1.07592
Chromatographic separation				**10.13**
Water elution	m^3	0.55	3.20	1.76
Electricity	kWh	0.07	14.00	0.98
Steam	ton	22.00	0.04	0.88
Resin Exclusion	litre	22.00	0.28	6.16
Water Backwash	m^3	0.55	0.01	0.0055
NaOH(100%)	kg	2.94	0.05	0.147
HCl(100%)	kg	3.92	0.05	0.196
Demineralisation				**69.20**
HCl(100%)	kg	3.92	9.40	36.848
NaOH(100%)	kg	2.94	9.90	29.106
Water soft	m^3	0.55	1.70	0.935
Resin Softening Anion	litre	20.00	0.07	1.3
Resin Softening Cation	litre	15.50	0.07	1.0075
Sugar concentration				**1.28**
Steam	ton	22.00	0.05	1.1
Electricity	kWh	0.07	2.60	0.182
Raffinate concentration				**5.30**
Steam	ton	22.00	0.22	4.84
Electricity	kWh	0.07	6.51	0.4557
Total Process materials				**375.34**

10.9.3 Estimate of Working Capital

The working capital was estimated according to two commonly used methods:

Method 1: The Working Capital, estimated as 30% of yearly sales (Financial Year 1), is R17.82 million or R660/ton Dry Invert Sugar.

Method 2: The Working Capital, estimated as 15% of capital, is R14.14 million or R523.7/ton Dry Invert Sugar.

For the cost estimation, the higher value will be used since there will be no production during Financial Year 0.

10.10 Profitability

Profitability is a measure of the total income of the project compared to the capital and cost outlay. It can also be described as *cash flow analysis*. It provides an answer to the question: What does the investment cost and how much cash will it generate each year? This question can also be restated as: What is the current investment and what are the future benefits?

It is important to note that cash flow analysis indicates cash flows, **not** profits. Accounting profits, as reported in the income statement, are a short-term measurement of an investment. Cash flow analysis (profitability) is a technique used to evaluate the project over the full life of the project.

There are broadly four categories of techniques used to calculate profitability.
Category I: Payout Time. The criterion here is time, such as the time needed to recover the investment.
Category II: Return on Investment: This uses rate of return as a criteria, but does not recognise the timing of cash flows.
Category III: Present Value: This recognises the timing of cash flows, but expresses the profitability as a surplus (or deficiency) net present value compared to that at some referenced rate of return.
Category IV: Discounted Cash Flow Rate of Return: The rate of return is used, but the timing of cash flows is also accounted for.

10.11 Cash Flow Analysis for the Invert Syrup Plant

The commodity prices were escalated with the CPI (Consumers Price Index) to provide a base for the calculation of the cash flow. The detailed cash flow analysis, financial statements and process costs are included in Appendix E.

10.11.1 Return on Original Investment

For a Capital Investment of R94,270,000.00 and a working capital of R17,820,000.00 the total investment equals R112,090,000.00. The profits from this estimate are listed in Table 10.6.

Table 10.6: The yearly profits.

	FY 1	FY 2	FY 3	FY 4	FY 5	FY 6	FY 7	FY 8	FY 9	FY 10
R'000	12,418	21,769	30,347	42,820	48,687	49,379	56,197	63,528	71,418	79,916

The average yearly profit is:

$$\frac{(12418+21769+30347+42820+48687+49379+56197+63528+71418+79916)}{10}=47{,}647.9$$

Thus: $ROI = \dfrac{47{,}647{,}900}{112{,}090{,}000} = 42.5\%$ (Return on investment)

And the payout time is <u>2.352 years</u>.

10.11.2 Net Present Value (NPV)

The following values for NPV were obtained by varying the discount rate:

Table 10.7: NPV obtained vs. the discount rate for the invert plant.

Rate	NPV (R'000)
10%	230,109
20%	105,694
30%	41,782
40%	5,412
50%	-17,101
60%	-32,020

The relationship between NPV and discount rate for the invert plant is shown in Figure 10.7

It was clear that the IRR lies between 40% and 50% - where the curve intersects the axis and NPV=0. The IRR was found by inspection to be around 43%. Microsoft Excel's IRR-function solved the IRR value (as is reported in the cash flow in the appendix) as 42%.

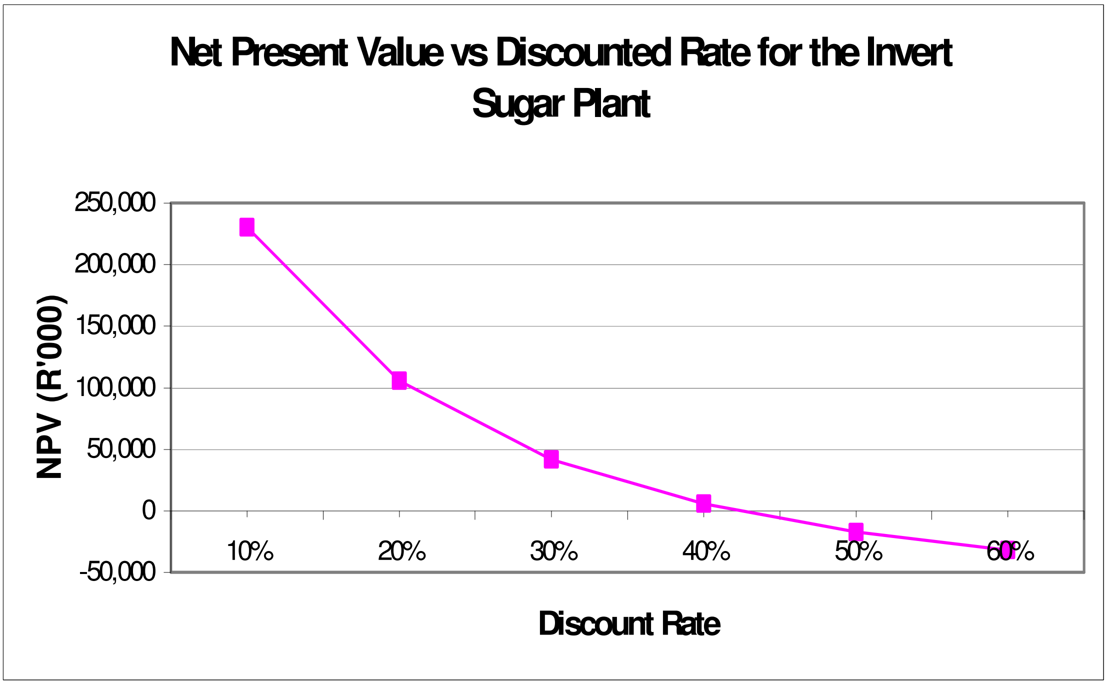


Figure 10.7: NPV vs. Discount rate for the invert plant.

10.11.3 Net Return Rate (NRR)

The NRR for the project was calculated at a 30% discount rate. The value of 30% is the hurdle rate required internally for achieving project feasibility. The $NPV_{30\%,0}$=R41,782,000.00.

The project life spans Financial year 0 to 11 = 11years. This resulted in:

$$NRR = \frac{41782}{(94270)(11)} = 4.0\% / yr$$

The Net Payout Time (NPT) can be interpolated from the $NPV_{30\%,0}$ yearly data.

The NPV becomes positive during financial year 6. Interpolation reveals that the NPV reaches zero after 5 years, 2 months and 18 days.

To summarize: the $NPV_{30\%,0}$=R41,782,000.00 is the profit criteria, the NRR = 4.0%/year is a profitability criteria and the normalised cash generation rate was the NPT of 5 years and 76 days.

Table 10.8: Yearly data of $NPV_{30\%,0}$.

Year	NPV at 30%, based on 2000 (R'000)
FY 0	-94,270
FY 1	-76,341
FY 2	-52,173
FY 3	-29,187
FY 4	-17,259
FY 5	-2,301
FY 6	8,814
FY 7	18,030
FY 8	25,682
FY 9	32,044
FY 10	37,341
FY 11	41,782

10.12 Sensitivity Analysis for the Invert Syrup Plant

In the course of evaluating a project a large number of estimates have to be made. It becomes necessary to recognise how sensitive the final estimate is to the individual component estimates. It is easier to predict expenses than either sales or profits. Fairly accurate estimations on capital and processing costs can be made. Errors in these estimates have a smaller effect than errors in sales price, sales volume and cost of raw materials. It is therefore worthwhile to set up tables and plot curves to show the effect of variations on profitability. This procedure is called sensitivity analysis and is an important tool in cost analysis. The purpose was to determine to which factors the profitability of the project was the most sensitive. It was carried out to observe the effect of departures from expected values.

The sensitivity analysis for the invert sugar plant was done by varying the following figures:

Selling Price: -9% and +9%
Molasses Price: +21%
Capital: -10% and +20%

The sensitivity analysis is included in Appendix F. An extract from the results are listed in Table 10.9.

Table 10.9: Results of the sensitivity analysis.

SENSITIVITY ANALYSIS		**PROJECT IRR %**	**NRR %/year**	**NPV (30%) R'000**	**NPT Years**
Base Case		42	4.0	41,782	5.207
Selling price	-9%	37	2.2	22,767	6.686
	9%	47	5.9	60,798	4.385
Molasses Price	21%	40	3.2	33,226	5.790
Capex	20%	35	1.8	22,928	7.108
	-10%	46	5.5	51,209	4.524

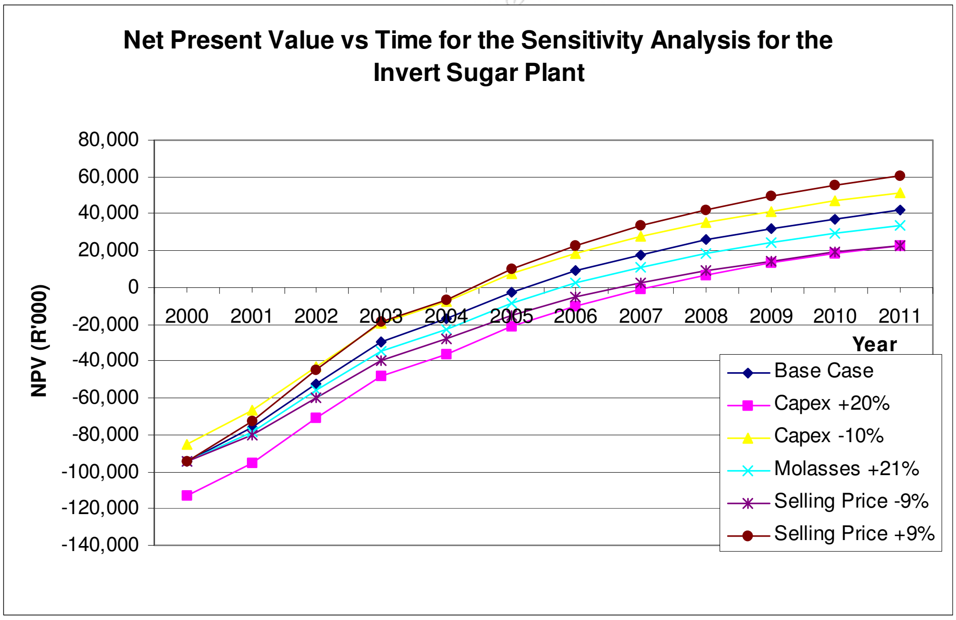


Figure 10.8: NPV vs. Time – The results of the sensitivity analysis.

The chart in Figure 10.8 displays the $NPV_{30\%,\ 0}$ against time with the variations that were investigated in the sensitivity analysis. It is clear that a higher selling price and a lower capital outlay would result in a more profitable project. The factor that had the most influence on the profitability is the 20% increase on capital. The price increase on molasses did not affect the profitability significantly. This allowed a large margin of safety should molasses have to be obtained from other sources.

It is important to note that a small change in IRR could have a large effect on the profitability of a project as determined with the NRR. It is also evident that the profitability of the project is much more sensitive to changes in selling price of invert than to capital outlay or feedstock cost. Invert syrup from molasses will compete with sugar in the local industrial market and if sugar was sold to local industry for less than about R1800/t, the viability of invert production from molasses would be threatened.

10.12 Conclusion

Cost estimation and the prediction of the profitability of a project are the most important components in the feasibility study of any project. Numerous techniques exist which can be used to determine the financial viability of a project.

A cost estimate for the invert sugar plant resulted in an internal rate of return (IRR) of 42% for a fixed capital investment of R94,270,000.00. The net return rate (NRR) for the project was 4%/year, the net present value (NPV) - discounted at a 30% cost-of-capital was R41,782,000.00. The net payout time (NPT) was 5.207 years.

The sensitivity analysis revealed that the price of molasses did not influence the project profitability to any degree. The profitability was much more sensitive to changes in the selling price of invert and a large increase in capital outlay.

The project met the financial feasibility criteria set by TSB: the IRR exceeded 25% and the plant was profitable at a cost-of-capital of 30%. The return on investment (ROI) payout time was 2.35 years. The discounted payout time was 5.027 years.

Chapter 11
Conclusion

A study on a pilot plant was conducted to try and establish a process, which would allow the desugarisation of cane molasses using ion exclusion chromatography. Previous work on this subject has had limited success in establishing a reliable and cost effective method to consistently produce sludge free molasses suitable for a desugarisation system. To this end, a pilot plant was designed and constructed to evaluate the clarification and separation of molasses so as to produce invert sugar syrup. The aim of the pilot plant was to prove the process and to provide data, which would facilitate the design and evaluate the financial viability of a commercial plant. The pilot plant had to process 300 kg/day of molasses.

Centrifugal clarification of molasses did not prove to be effective in clarifying the molasses and giving a product suitable for ion exchange or chromatography. Chemical treatment was able to improve the quality of the product, but the results were still not acceptable. The characteristics of this product were such that dead end pressure filtration became difficult. There were indications that a suitable product could be obtained by the gravitational settling of the molasses sludge, and this observation was investigated further in detail.

In this study, seven factors that could possibly affect the consolidation and settling of suspended solids in molasses were investigated. These included the age of the diluted molasses, the temperature of the flocculated mixture, the variations across various batches of flocculant, the effect of the reaction time of the phosphoric acid, the optimum flocculant dosing concentration, the optimum concentration of the molasses solution and the effect of increasing the acid dosage. The optimum conditions to clarify molasses were found to be: fresh molasses, at 28 Brix and 60℃, allowing 10 min intervals between acidification with 3,75 g (as 100%) phosphoric acid/ kg dry material (assumed equal to Brix) and neutralisation with 5 g (as 100%) caustic/kg dry material (assumed equal to Brix), floc with any batch of flocculant 6195, dosed as a 1000 ppm solution. The turbidity of the molasses solutions were effectively reduced by more than 80 per cent to yield a value of less than 10 NTU/Brix, which is suitable for chromatography. Part of the calcium was also successfully removed in this first clarification stage, while the rest was removed later by ion exchange on a strong base resin.

The concentration of the flocculant stock solution was found to be critical for achieving a proper settling of the molasses. The flocculant solution must be added to the molasses at a concentration of 0.1% and the concentration in the clarifier feed should be 20 ppm. The age of the molasses solution initially appeared to affect the clarification efficiency. Older molasses did not clarify as efficiently as fresh molasses, but once the process on the commercial E-Cat™ clarifier had been optimised, the age of the molasses did not appear to be a critical variable. Inversion of the sucrose in the molasses with high invertase yeast before clarification on the E-Cat, removed the need for additional processing steps. The yeast was then successfully removed from the molasses during the clarification stage. The optimum operating conditions for the E-Cat™ clarifier were determined and are presented in Table 11.1.

Table 11.1: Operating Conditions for Molasses Clarification on an E-Cat™

Feed flow of inverted molasses (Expressed as a flux relative to the surface area of the clarifier)	4244 l/m^{2}h
H_3PO_4 addition	500 – 750 ppm
Feed Brix	22 – 28 Brix
Feed Temperature	Not below 65^{o}C
Feed pH	6,5 to 7,0
Flocculant Type	Montan 6195
Flocculant make-up solution	1000 ppm
Flocculant concentration in clarifier feed solution.	20 ppm

The recovery of sugars from the molasses sludge has economic merit. From the evaluation of centrifugal and gravity separation, it is clear that gravity separation is the best method. The sweet-water obtained is consistent within the clarity requirement of 10 NTU/Brix and can be used to dilute raw molasses in the upstream processing step.

The pilot plant process development program proved that molasses can be successfully be clarified to allow its use in a chromatography plant. The clarification process reported in this thesis and the ensuing intellectual properly are now protected by a South African patent [Bekker, PI, Stolz HNP *et.al.* (2001)]. This patent relates to the clarification of cane molasses and the preparation of the molasses syrup for desugarisation using ion exclusion chromatography. The molasses treatment process requires less chemical treatment and uses simpler equipment than any other process previously suggested. This process yields a molasses product suitable for desugarisation using chromatography. The simplicity and efficiency of the new clarification process increase the financial viability of the overall desugarisation process to such an extent that is now possible to commercialise the process.

Molasses that was free of sludge and suspended solids, was prepared using the methods described in this thesis. This was concentrated to a syrup for separation on a chromatography pilot plant. A sugar recovery from the molasses of 93.9 mass % at a purity of 99.7 mass % was achieved on a simulated moving bed, ion exclusion pilot plant. The operating conditions (described as Separation Bed Volumes) for this performance were:

BV1 = 0.694; BV2 = 0.591; BV3 = 0.661; BV4 = 0.383.

These separation variables are all that is required to allow commercial scale-up of the simulated moving bed from the pilot plant design. The trade-off between purity and recovery and a reduction in water usage can be investigated further. Being able to reliably and continuously operate the chromatography plant is further proof that the clarification process is indeed reliable and that the molasses that was prepared with this method was suitable for industrial separation.

The liquid sugar fraction, after deashing and decolourising contains a negligible amount of salt and very little colour. The product is comparable to industrial brown sugar and will compete for market share in the liquid sugar market.

Various techniques were used in a cost estimate and profitability study for the invert sugar plant. This resulted in an internal rate of return (IRR) of 42% for a fixed capital investment of R94,270,000.00. The net return rate (NRR) for the project is 4%/year, the net present value (NPV) - discounted at a 30% cost-of-capital, is R41,782,000.00. The net payout time (NPT) is 5.207 years. The molasses desugarisation process met the financial requirements set by TSB to warrant further detailed investigations.

This project developed the first commercially viable method to consistently desugarise sugar cane molasses. The process described in this thesis and its further refinements are presently the subject of an international technology licensing investigation. None of the methods reported in the literature can reliably achieve these results on a cost effective basis. It is now possible to produce liquid invert syrup, a high value food product, from a very cheap source of sugar, resulting in significant income for the company.

References

Alvarez, JF, Baez-Smith, CY and Weiss LW (2001) Modelling the new technology raw sugar factory/refinery using SUGARS^{TM} for Windows$^{®}$. *Int Sug J,* 103(1231). Obtained from: http://www.sugarsonline.com/SugarsPapers/New Tech-2001.pdf

Applexion Laboratory Manual – (1996)

Bekker, PI, Stolz HNP *et.al.* (2001) A method of clarifying molasses, SA Patent 20015995, 20 July 2001.

Bernhardt, HW (1998). Centrifugal clarification of molasses. *Proc S Afr Sug Technol Ass* 72:278-284

Bernhardt, HW, Davis, SB, Peacock, SD and Walford, SN (2000). The production of high test molasses (HTM) from final molasses. Sugar Milling Research Institute, Report no. 168, January 2000.

Batson, RG (1994). Process for treating a suspension of solid particles in a carrier liquid. European Patent 0 585 103 A2, 2 March 1994.

Batson, RG (1998). Separation of a suspension into its component parts, US Patent 5,800,715; September 1, 1998.

Chen, JCP and Chou, CC (1993). Cane Sugar Handbook, 12th Edition, John Wiley and Sons, New York. 1090pp

Chen, JCP and Proskowetz, F (1965). Further investigations of the centrifugation of B-molasses. *Proc Int Soc Sug Cane Tecnol* XXII:1677-1683

Chakrabarty, D.K. (1991). Adsorption and Catalysis by Solids, Wiley Eastern Ltd.

Charton, F and Nicoud, RM (1995). Complete design of a simulated moving bed. *J. Chromatogr. A* 702 (1995) 97-112

Clarke, MA (Chapter Editor) (1995). Chapter 44 – Sugars and Sugar Products in Official Methods of Analysis, 1995, 16th Edition Association of Official Analytical Chemists (AOAC).

Coulson JM, Richardson JF *et.al.* (1991). Coulson & Richardson's Chemical Engineering, Volume 2, Particle Technology and Separation Processes, 1991, 4th Edition, Pergamon Press.

Davis, CW and Clarke, WB (1962). Removal of sludge from B-molasses by centrifugal separation. *Proc Int Soc Sug Cane Tecnol* XXII:856-865

Davis, SB, Peacock, SD and Walford, SN (1997). Ion exclusion chromatography: molasses pretreatment and high test molasses (HTM) production. *Proc S Afr Sug Technol Ass* 71:146-152

De Whalley, H.C.S. (Ed.) (1964), 'ICUMSA Methods of Sugar Analysis' Elsevier, Amsterdam.

Farber, L. and Carpenter, F.G. (unknown). Identification of Sugar Colorants. Conference unknown. p145-153

Godshall, MA (1997). Symposium on Sugar Color Part 2. Color Analysis, *Sugar Industry Technologists* 1997, p211-231

Green, DW ed. (1984). Perry's Chemical Engineers' Handbook, 6th Edition, 1984, McGraw Hill.

Heckly, S. (1998); Chromatographic Separation Unit Handbook, Applexion 97.10.3108.PR09.Rev.0.

Henscheid,T, Valasquez, L and Meacham, D (1991). Five years' experience with weak cation softening on thin juice, *Sugar y Azucar*, February 1991, 43-48.

Himmelblau, DM (1982). Basic Principles and Calculations in Chemical Engineering, 4th Edition, 1982, Prentice-Hall.

Hongisto, HJ (1977). Chromatographic separation of sugar solutions: the Finnsugar molasses desugarisation process Part I, *Int Sug J,* 79:100-104.

Hongisto, HJ (1977). Chromatographic separation of sugar solutions: the Finnsugar molasses desugarisation process Part II, *Int Sug J,* 79:131-134.

Honig, P (1963). Principles of Sugar Technology, Volume III. Elsevier Publishing Company, New York.

Hugot, E (1972). Handbook of Cane Sugar Engineering, translated by GH Jenkins, 2nd Edition, 1972, Elsevier Press, New York.

ICUMSA, Report of the Proceedings 15th Session (1970): Subj.11 Rec. 3,105

ICUMSA, Report of the Proceeding 16th Session (1974): Subj.2, Rec. 1.22

ICUMSA, Report of the Proceedings 16th session (1974): Subj. 10, rec. 3,126

ICUMSA, Report of the Proceeding 16th Session (1974): Subj.11 Rec. 1,2,3,& 7,142

ICUMSA, Report of the Proceedings 16th Session (1974): Subj. 12, Rec. 1, 156

ICUMSA, Report of the Proceedings 16th Session (1974): Subj.16,

ICUMSA, Report of the Proceeding 17th Session (1978): Subj.11, Rec.7

IDC Technologies (2000). Techniques for the Ranking of Investment Proposals, Http://www.idc-online.com/assets/files/fm10.pdf

Kearney, M (1990). Simulated Moving Bed technology applied to the chromatographic recovery of sucrose from sucrose syrups. *Proc. Sugar Process. Research*, pp291-304

Kearney, M (1990). The use of infinite series for optimising placement and operation of chromatographic separators. *Proc. Sugar Process. Research*, pp168-179

Kearney, M (2002). Chromatographic applications in the cane sugar industry. *Int Sug J,* 104(1241): 194-203

Lancrenon, X. and Herve, D. (1988). Recent trends in the use of ion exchange in the sugar industry. *Sugar Technology Reviews*, 14:207-274

Lionnet, GRE (2001). Seventy-sixth annual review of the milling season in South Africa (2000-2001). *Proc S Afr Sug Technol Ass* 75:6-27

MacGillvray, AW and Matic, M (1970). Composition of South African Final Molasses. *Proc S Afr Sug Technol Ass* 44:81-87

Meade, GP and Chen, JCP (1977). Cane Sugar Handbook, Tenth Edition, 1977, Wiley Interscience, John Wiley & Sons, New York.

Michaelis, L and Menten, ML (1913). *Biochem. Z.*, 49, 333

Murphy, T.D. (1977). Design and analysis of industrial experiments. *Chemical Eng* June 6, 1977: 168-182

Paananen, H.A (1996). Trends in the chromatographic separation of molasses. *Proceedings of the Workshop on Separation Processes in the Sugar Industry,* Sugar Processing Research Institute, April 18, 1996, New Oleans.

Paillat D and Cotillon M (2000). Different industrial applications of continuous chromatography both in the sugar industry and for the prtoduction of by-products. *Zuckerindustrie* 125:47-51

Paturau, J.M. (1989). By-products of the cane sugar industry, an introduction to their industrial utilization. 3rd Edition, 1989, Elsevier Sugar Series, Volume 11. Elsevier, Amsterdam.

Peacock, SD. (1996). Ion exclusion desugarisation of refinery Jet 4. *Proc S Afr Sug Technol Ass* 70:171-176

Pillay, J.V. (unknown). Essential Sugar Technology, Short course presented by the Sugar Milling Research Institute, Durban.

Riffer, R. (1976). Process for the production of colourless sugar syrup from cane molasses. US Patent 4,046,590. September 8, 1976.

Powell, TE (1985). A review of recent developments in project evaluation. *Chemical Eng.* Nov 1985:187-194

Proskowetz, F and Chen, JCP (1962). Centrifugation of B-molasses and its clarification effect. *Proc Int Soc Sug Cane Tecnol* XXI: 866-869

Pynnonnen, B. (1996). A look at sucrose recovery via chromatographic separation. *Proceedings of the Workshop on Separation Processes in the Sugar Industry,* Sugar Processing Research Institute, April 18, 1996, New Oleans.

Rearick, DE and Kearney, M (1996). Removal of nonsugars from cane molasses by ion-exclusion chromatography. *Proc Int Sug Cane Technol* XXII: 167-171

Rohm and Haas Co. (1996) Amberlite 252RFH Technical Specification Sheet.

Rohm and Haas Co. (1996). Amberlite IRA92 Technical Specification Sheet

Rohm and Haas Co. (1996). Amberlite IR120 Technical Specification Sheet.

Saska, M, Wu, MD, Clarke, SJ and Iqbal, K (1992). Continuous Separation of Sugarcane Molasses with a Simulated Moving Bed Adsorber. Adsoption Equilibria, Kinetics and Application. *Sep Sci and Tech*, 1992, 27(13):1711-1732.

Saska, M (1993). An AudubonSugar Institute-Applexion process for desugarization of cane molasses. *Proc Sugar Ind Techno*l. pp 110-131

Saska, M and Lancrenon, X. (1994). Applications of continuous chromatographic separation in the sugar industry. III. Desugarization of cane molasses. *Int. Sugar Jnl.* 1994 96:403-410

Seifert, JA (1987). Selecting Thickeners and Clarifiers. *Chemical Eng.* October 12, 1987, 94(14):11-

Seifert Hazen, JA and Sawyer, PC (1990). Getting the most out of Thickeners and Clarifiers. *Chemical Eng.* August 1990, 97(8):80-

Schenck, FW (2000). High fructose syrups – a review. *Int Sug J,* 102:285-288

Schneider, HG (1978). Ion exclusion in cane sugar refining. *Proc Sugar Ind Technol.* Paper 415: 110-131

Schoenrock, KWR, Gupta, A and Rounds, HG (1976). Process for the purification of impure sugar juice. US Patent 3,982,956. September 28, 1976.

Schoenrock, KWR, Kearney, D and Rearick, E (1983). Method and apparatus for the sorption and separation of dissolved constituents. US Patent 4,412,866. November 1, 1983.

Shelly, S.A. (1997). The battle cry for coagulants and flocculants: charge! *Chemical Eng.* June 1997, 104(6): 63-64,66

Simpson, R. (1996). The chemistry of clarification. *Proc S Afr Sug Technol Ass* 70:267-271

Sinnot, RK ed. (1993). Coulson & Richardson's Chemical Engineering, Volume 6, Design, 1993, Pergamon Press.

Soffiantini, VA, Jennings, RP and Wilkes, R (1971). Seasonal variations in molasses composition. *Proc S Afr Sug Technol Ass* 45:99-106

Stolz, HNP and Bekker, PI (2001). Enhancement of sugar production Part 2: Chromatographic Separation of sugarcane molasses. *Proc S Afr Sug Technol Ass* 75

Stolz, HNP and Weiss LW (1997). Simulation of the Malelane and Komati Mills using SUGARS^{TM} Simulation software. *Proc S Afr Sug Technol Ass* 71:184-188

Sugar y Azucar (1994). Ion exclusion technology and beet molasses desugaring in the United States, *Sugar y Azucar*, 89 (2):16-34

Thompson, MC (1993). Progress report on the centrifugal clarification of molasses. SMRI Technical Report 1653, 14 May: 27pp

Ulrich, GD (1992). Combine capital and fuel cost to eliminate guesswork for new plants or additions. *Chemical Eng.* February 1992, 99(2):110-

Walford, SN (1996). Composition of Cane Juice. *Proc S Afr Sug Technol Ass* 70:265-266

Ward, TJ (1989). Estimate Profitability using Net Return Rate. *Chemical Eng.* March 1989, 96(3): 151

Western Sugar Company (1991). State of the art desugarization facility. *Sugar y Azucar*, 86 (6): 16

APPENDIX A

PROPERTIES OF ION EXCHANGE RESINS USED IN THE PILOT PLANT

AMBERLITE® 252 Na

Industrial Grade Strong Acid Cation Exchanger

PRODUCT DATA SHEET

AMBERLITE 252 Na is a macroporous cation exchange resin based on sulphonated cross-linked polystyrene. It has a moderate degree of crosslinking resulting in good regeneration efficiency. It is very resistant to osmotic shock and to mechanical attrition. AMBERLITE 252 Na has a reduced amount f ng it to be used for the treatment of highly concentrated solutions. AMBERLITE 252 Na u f e in a variety of demanding applications such as condensate treatment, decalcification t gar juices and treatment of oxidising solutions.

PROPERTIES

Matrix	ylbenzene copolymer
Functional groups	3
form	Light grey beads
Ionic form as shipped	
T a xchange capacity [1]	≥ 1.8 eq/L (Na⁺ form)
Moisture holding cap t [1]	7 54 % (Na⁺ form)
Shipping weight	810 g/L
Specific gravity	1.20 to 1.24 (Na⁺ form)
Particle size	
Uniform t c f	≤ 1.8
Harmonic mean size	590 - 840 µm
Fine co te t [1]	< 0.300 mm : 1.0 % max
Coarse beads	> 1 8 5

[1] *Contractual value*

Test methods are available on request.

SUGGESTED OPERATING CONDITIONS

Mi im b de th	700 mm		
Service flow rate	5 to 40 BV*/h (ug) 5 o 70 BV/h or 5 to 120 m/h (water)		
Regenerant	NaCl	HCl	H_2SO_4
Flow ra e (B /h	2 to 8	4 to 6	4 to 12
Concentration (%)	10	4 to	to 5
L vel (g/L)	80 to 400	45 to 150	50 to 2
imum contact time	30 minutes		
Slow rinse	2 BV at eneration flow rate		
Fast rinse	2 to 4 BV at service flow rate		

** 1 BV (Volume) = 1 m^3 solution per m^3 resin*

HYDRAULIC CHARACTERISTICS

AMBERLITE 252 Na gives pre s e drop of about 12 kPa/m bed depth per 10 m/h at 15°C A b k sh fl te f 5 /h gives a bed expansion of about 65 % at 15°C in water.

Pressure drop data a li a t s r o the service run with a clear water and a correctly classified bed.

These data ar v id fo w e t nt and have to be corrected according to the solution to be treated.

LIMITS OF USE

Rohm and a ma f t e i esins for food processing and o b water applications. As governmental e la ons vary from country to country, it is recommended t t potential users seek advice from their Amberlite representative in order to determine the best res n c oice and optimum operating conditions.

l ur roducts are produced in ISO 9002 certified manufact ing facilities

and Haas/Ion Exchange Resins - Philadelphia, PA - Tel. (800) RH AMBER - Fax: (215) 537-4157
Rohm and Haas/Ion Ex ge e ns - 75579 Paris Cedex 12 - Tel. (33) 1 40 02 50 00 Fax : 1 43 45 28 19

WEB SITE: http:// ww rohmha s.com/ ionexchange

ROHM AND HAAS

AMBERLITE is a trademark of Rohm and Haas Company, Philadelphia, U.S.A.

Ion exchange resins and polymeric adsorbents, as produced, contain by-products resulting from the manufacturing process. The user must determine the extent to which organic by-products must be removed for any particular use and establish techniques to assure that the appropriate level of purity is achieved for that use. The user must ensure compliance with all prudent safety standards and regulatory requirements governing the application. Except where specifically otherwise stated, Rohm and Haas Company does not recommend its ion exchange resins or polymeric adsorbents, as supplied, as being suitable or appropriately pure for any particular use. Consult your Rohm and Haas technical representative for further information. Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact. Nitric acid and other strong oxidising agents can cause explosive type reactions when mixed with Ion Exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidising agent such as nitric acid is contemplated. Before using strong oxidising agents in contact with Ion Exchange Resins, consult sources knowledgeable in the handling of these materials.

AMBERLITE® CR1320 K

Chromatographic Separation Resin

For Beet Sugar Recovery and Polydextrose Purification

PRODUCT DATA SHEET

AMBERLITE CR1320 K is a uniform particle size, chromatographic separation resin specifically designed for the recovery of sucrose and betaine from beet sugar molasses. It is also used for the purification of dextrose and polydextroses made from starches. The jetting process from which AMBERLITE CR1320K is made results in an extremely uniform product in both its particle size and microporosity. This uniformity provides the user with very sharp and consistent separations.

AMBERLITE CR1320 K is widely used in the recovery of sucrose and betaine from beet molasses in all commonly used SMB chromatographic systems. AMBERLITE CR1320 is also available in the Ca form for separation of glucose and fructose and the purification of other specialty sugars.

PROPERTIES

Matrix	Styrene divinylbenzene copolymer
Functional groups	Sulfonates
Physical form	Light translucent beads
Ionic form as shipped	K^+
Total exchange capacity [1]	≥ 1.50 eq.L (H^+ form)
Moisture holding capacity [1]	58 to 64 % (H^+ form)
Shipping weight	850 g/L
Harmonic mean size [1]	0.310 - 0.350 mm
Uniformity coefficient [1]	≤ 1.15
Fines content [1]	> 0.212 mm : ≥ 99%
Maximum reversible swelling	$Na^+ \rightarrow H^+$: 5 to 10 % $K^+ \rightarrow H^+$: 12 to 18 %
Maximum operating temperature	120°C
Chemical resistance	Insoluble in dilute solutions of acids or bases and common solvents

[1] *Contractual value*

Test methods are available on request.

FOOD PROCESSING

As governmental regulations vary from country to country, it is recommended that potential users of resins for food processing applications contact their Rohm and Haas representative to assess the best choice of resin and optimum operating conditions.

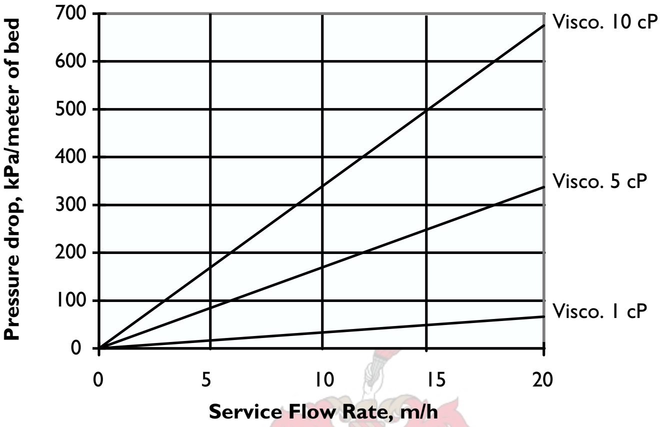
HYDRAULIC CHARACTERISTICS

Figure 1 shows the pressure drop data for AMBERLITE CR1320 K, as a function of service flow rate and viscosity of the solution to be treated.

Conversion Factor:

- 1 kPa/m equals 0.0442 psi/ft

Figure 1: Pressure Drop (at 24°C)



All our products are produced in ISO 9002 certified manufacturing facilities.

Pectora roborant cultus recti

Rohm and Haas/Ion Exchange Resins - Philadelphia, PA - Tel. (800) RH AMBER - Fax: (215) 409-4534
Rohm and Haas/Ion Exchange Resins - 75579 Paris Cedex 12 - Tel. (33) 1 40 02 50 00 - Fax : 1 43 45 28 19

WEB SITE: http://www.rohmhaas.com/ionexchange

AMBERLITE is a trademark of Rohm and Haas Company, Philadelphia, U.S.A.

Ion exchange resins and polymeric adsorbents, as produced, contain by-products resulting from the manufacturing process. The user must determine the extent to which organic by-products must be removed for any particular use and establish techniques to assure that the appropriate level of purity is achieved for that use. The user must ensure compliance with all prudent safety standards and regulatory requirements governing the application. Except where specifically otherwise stated, Rohm and Haas Company does not recommend its ion exchange resins or polymeric adsorbents, as supplied, as being suitable or appropriately pure for any particular use. Consult your Rohm and Haas technical representative for further information. Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact. Nitric acid and other strong oxidising agents can cause explosive type reactions when mixed with Ion Exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidising agent such as nitric acid is contemplated. Before using strong oxidising agents in contact with Ion Exchange Resins, consult sources knowledgeable in the handling of these materials.

Rohm and Haas Company makes no warranties either expressed or implied as to the accuracy of appropriateness of this data and expressly excludes any liability upon Rohm and Haas arising out of its use. We recommend that the prospective users determine for themselves the suitability of Rohm and Haas materials and suggestions for any use prior to their adoption. Suggestions for uses of our products of the inclusion of descriptive material from patents and the citation of specific patents in this publication should not be understood as recommending the use of our products in violation of any patent or as permission or license to use any patents of the Rohm and Haas Company. Material Safety Data Sheets outlining the hazards and handling methods for our products are available on request.

©2001 Rohm and Haas Company

AMBERLITE® IRA92RF

Industrial Grade Weak Base Anion Exchanger

PRODUCT DATA SHEET

AMBERLITE IRA92RF is a high capacity polystyrene, weak base anion exchanger. This resin is highly efficient for the uptake of strong acids (e.g. HCl, H_2SO_4) when foll s g d cation exchanger in the H form. Its macroporous structure ensures excellent adsorption and desorption of organic matter. It has an outstanding mechanic and o st l , making it suitable for the treatment of solution with high ionic concentrati R A as a reduced amount of fines allowing to minimize the pressure drop.

PROPERTIES

Matrix	Macroporous polystyrene
Functional groups	NR_2 : 90 %
Physical form	Ivory-coloured beads
I o as ship	Free Base (FB)
Total exchange capacity [1]	≥ 1. e)
holding capacity [1]	40 to 50 % (FB form)
Shipping weight	
Specific gravity	1.035 0 r
e size	
Harmonic mean size	600 to 800 µm
Uniformity coeffi	≤ 1.5
Fines content [1]	< 0.300 mm : 0.2 % max
Maxim m e ble s	FB → Cl^- : 25 %
Chemical resistance	Insoluble in dilute solutions of acids or bases and common solvents

Test methods avail ble p r

SUGGESTED OPERATING CONDITIONS

ting temperature limit	90°C (FB form)		
Service flow rate	0 BV*/h		
Regenerants	NaOH	NH_3	Na_2CO_3
Level (g/L)	40 to 80	40 to 80	60 to 130
Concentration (%)	2 to 6	2 to 3	5 to 8
Flow rate (BV/h)	2 to 8	2 to 8	2 to 8
Minimum cont	30 minutes		
Slow rinse	2 BV at regeneration flow rate		
Fast rinse	4 to 8 BV at service flow rate		

** 1 BV (Bed Volume)= 1 m 1 n per m^3 resin*

APPLICATIONS

The high total capacity of AMBERLITE IRA92RF makes it particularly suitable for e o l of strong anions from solutions with l y i s v ids ; its regeneration eff c nc is close to the theoretical output. A high operating capacity is obtained from AMBERLITE IRA92R u er nditions where a high TDS water is tre e a m e t sp i c low rate. The combined adso p on f e cy h i l sta l of AMBERLITE IRA92RF make it the product of choice for demineralisation g c . co t o its outstanding characteristics AMBERLITE IRA92RF is used in the followi sp ci pl o s

cidification of formol,
- Purification o alcaloids,
- Demineralisation of g at e l to u s
- over m from cooling circu
- y r er n electroplating workshop.

FOOD PROCESSING

Rohm and H as t e cial resins for fo d roc ss ng an d nking wa er applications. As governmental r gul i s ar r country to countr r o en a p tial users contact their Duolite repr s n t e s s the best choice of resin and optimum operating condi on .

A r ro c a e produced in I 90 2 e if d anufacturing facilities.

Pectora roborant cultus recti

Rohm and Haas/Ion Exchange Resins Ph l elp ia P - el. (800) RH AMBER F x: (21) 37 415
Ro m and Haas/Ion Exchange Resins - 7 7 Pa e x Te . (33) 1 40 02 0 : 5 2 1

WEB SITE: http:// www.rohmhaas.com/ io ch

AMBERLITE is a trademark of Rohm and Haas Company, Philadelphia, U.S.A.
Ion exchange resins and polymeric adsorbents, as produced, contain by-products resulting from the manufacturing process. The user must determine the extent to which organic by-products must be removed for any particular use and establish techniques to assure that the appropriate level of purity is achieved for that use. The user must ensure compliance with all prudent safety standards and regulatory requirements governing the application. Except where specifically otherwise stated, Rohm and Haas Company does not recommend its ion exchange resins or polymeric adsorbents, as supplied, as being suitable or appropriately pure for any particular use. Consult your Rohm and Haas technical representative for further information. Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact. Nitric acid and other strong oxidising agents can cause explosive type reactions when mixed with Ion Exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidising agent such as nitric acid is contemplated. Before using strong oxidising agents in contact with Ion Exchange Resins, consult sources knowledgeable in the handling of these materials.

Rohm and Haas Company makes no warranties either expressed or implied as to the accuracy of appropriateness of this data and expressly excludes any liability upon Rohm and Haas arising out of its use. We recommend that the prospective users determine for themselves the suitability of Rohm and Haas materials and suggestions for any use prior to their adoption. Suggestions for uses of our products of the inclusion of descriptive material from patents and the citation of specific patents in this publication should not be understood as recommending the use of our products in violation of any patent or as permission or license to use any patents of the Rohm and Haas Company. Material Safety Data Sheets outlining the hazards and handling methods for our products are available on request.

©2000 Rohm and Haas Company

Product Information

DOWEX OPTIPORE SD-2 Adsorbent

DOWEX OPTIPORE SD-2 Polymeric Adsorbent for Sucrose and Other Sweetener Applications

DOWEX* OPTIPORE* SD-2 adsorbent is an adsorbent with a high specific surface area and high porosity which shows exceptional mechanical, thermal and chemical stability. It is specially designed for decolorization as well as taste and odor removal in sweetener applications and complies with the U.S. Food, Drug and Cosmetic Act as amended under Food Additive Regulation 21 CFR 173.25.

This adsorbent has similar pore size distribution and adsorption properties to activated carbon so that it can be used as a direct replacement for carbon in many sweeteners applications. DOWEX OPTIPORE SD-2 has the additional advantage of containing macropores which improve the bulk movement of solutions in and out of the bead. The resulting improvement in kinetics leads to faster production flow rates and smaller beds.

The adsorbent is lightly functionalized with weak base groups to provide a hydrophilic character giving the adsorbent good wettability and compatibility with acid and base regenerants. Regeneration requires only dilute caustic, dilute acid, and hot water.

More detailed information on the use of DOWEX OPTIPORE SD-2 adsorbent in your particular application can be obtained from your Dow technical contact or from your Dow sales representative.

Performance Specifications

Product	Type	Matrix	Functional Group
DOWEX OPTIPORE SD-2 Adsorbent	Adsorbent	Macroporous styrene divinylbenzene copolymer	Tertiary amine
General Properties			
Dry Weight Capacity		0.8 meq/g	
Typical Surface Area		800 m^2/g	
Average Pore Diameter		50 Angstrom	
Water Content (%)		50-62	
Particle Size: >1.19 mm		≤ 2%	
<0.297 mm (total)		≤ 3%	
<0.210 mm		0.3% max.	
<0.105 mm		0.0% max.	
Swelling, max.		5%	
Sphericity, min.		90%	
Density		1.04 g/ml	
Shipping weight, approx.		670 g/l (42 lb/ft^3)	
Typical Operating Condition Ranges (dependent on application)			
Operating Temperature		50°-85°C	
Operating pH		3-8.5	
Service / Regeneration Flow Rates		2-6 BV/hr	
Regenerant Type		NaOH, HCl or H_2SO_4	

*Trademark of The Dow Chemical Company

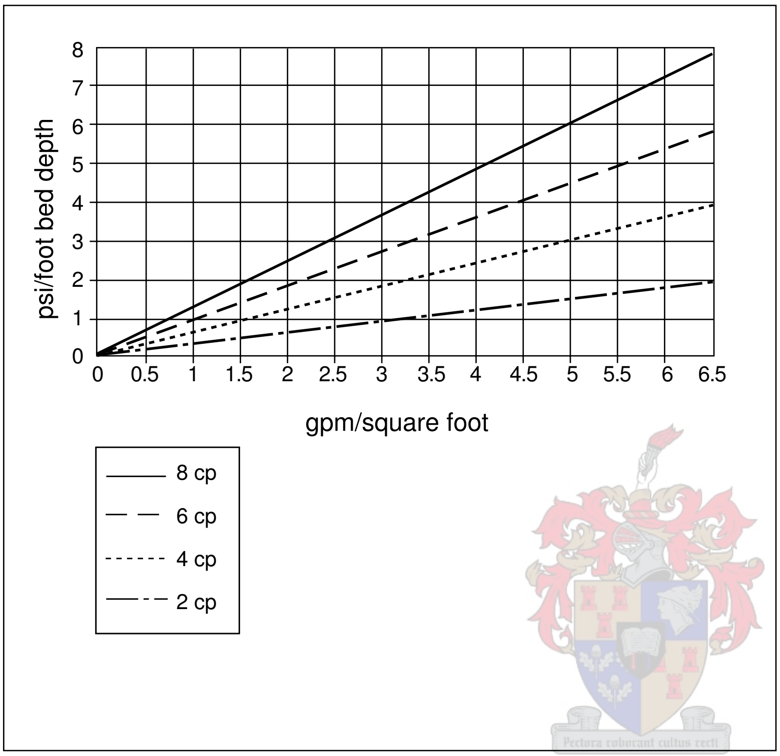
DOWEX
OPTIPORE ADSORBENTS

For more information about
DOWEX OPTIPORE adsorbents,
call Dow Liquid Separations Businesses:

North America	1-800-447-4369
Latin America	(+55) 11-5188-9345
Europe	(+31) 20-691-6268
Japan	(+81) 3-5460-2100
Australia	(+61) 2-9776-3226

http://www.dow.com/liquidseps

Figure 1. Pressure Drop for DOWEX OPTIPORE SD-2 Adsorbent



[11] **Patent number:** 4,950,332
[45] **Date of patent:** Aug, 21, 1990

[57] **Abstract**
A process for decolorizing an aqueous sugar solution by contacting the solution with an absorbent resin, wherein the adsorbent resin is a macroporous copolymer being post-crosslinked in a swollen state in the presence of a Friedel-Crafts catalyst and functionalized with hydrophilic groups prior to contact with aqueous sugar solution.

[75] **Inventors:**
Richard T. Springfield,
H. Robert Gotlz, Seth I. Norman,
Upen J. Bharwada, Robert L.
LaBrie, all of Midland, Mich.

[73] Assignee:
The Dow Chemical Company
Midland, Mich.

Warning: Oxidizing agents such as nitric acid attack organic ion exchange resins under certain conditions. This could lead to anything from slight resin degradation to a violent exothermic reaction (explosion). Before using strong oxidizing agents, consult sources knowledgeable in handling such materials.

Published July 1998.

Form No. 177-01666-798QRP
CH 171-385-E-798

ROHM AND HAAS

ION EXCHANGE RESINS

PRODUCT DATA SHEET

AMBERLITE IRA958 Cl

Industrial Grade Strong Base Anion Exchange Resin

AMBERLITE IRA958 Cl is a macroreticular strongly basic anion exchange resin exhibiting quaternary ammonium functionality in a crosslinked acrylic polymer matrix. The high porosity of its macroreticular structure allows more complete removal of large organic molecules and provides excellent resistance to physical breakdown by attrition and osmotic shock. The acrylic composition of the matrix contributes to excellent desorption of organics during regeneration. AMBERLITE IRA958 Cl is particularly useful as an organic scavenger for the adsorption of natural organic matter from surface water. Placed ahead of an ion exchange deionisation system, AMBERLITE IRA958 Cl helps prevent organic fouling of the working anion exchange resins of the plant. AMBERLITE IRA958 Cl is also used in sugar juice decolourisation to remove colour bodies from cane sugar syrups and in purification of various chemical and pharmaceutical products.
AMBERLITE IRA958 Cl complies with the FDA 21 CFR 173.25 regulation : all that is required at the time of commissioning is to perform a full regeneration cycle followed by a rinse with at least 10 bedvolumes of water.

PROPERTIES	
Matrix	Crosslinked acrylic macroreticular structure
Functional groups	Quaternary ammonium
Physical form	White opaque beads
Ionic form as shipped	Chloride
Total exchange capacity [1]	≥ 0.8 eq/L (Cl^- form)
Moisture holding capacity [1]	66 to 72 % (Cl^- form)
Specific gravity	1.05 to 1.08 (Cl^- form)
Shipping weight	720 g/L
Particle size	
Uniformity coefficient	≤ 1.8
Harmonic mean size	630 to 850 µm
Fine contents [1]	< 0.355 mm : 1.0 % max
Coarse beads	> 1.180 mm : 5.0 % max
[1] Contractual value	
Test methods are available on request.	

SUGGESTED OPERATING CONDITIONS	(Scavenger)
Maximum operating temperature	80℃ (Cl^-)
Minimum bed depth	600 mm
Service flow rate	8 to 40 BV*/h
Regenerant	NaOH 2 % + NaCl 10 %
Flow rate (BV/h)	2 to 6 2 to 6
Level (g/L)	6 to 40 160 to 300
Minimum contact time	30 minutes
Slow rinse	5 to 10 BV
Fast rinse	Same as regenerant for first bed displacement, then same as service flow rate
** 1 BV (Bed Volume) = 1 m^3 solution per m^3 resin*	

SUGGESTED OPERATING CONDITIONS (Sugar)

Service	2 to 4 BV*/h
Sweetening off	2.5 BV/h at 60°C with softened or demineralised water
Backwash	7 to 8 m/h at 60°C with softened or demineralised water
Regenerant	NaCl
Level	150 to 250 g/L
Concentration	10 % at 60°C
Rinse requirement	5 to 10 BV
Sweetening on	2.5 BV/h dilute syrup

** 1 BV (Bed Volume) = 1 m^3 solution per m^3 resin*

PERFORMANCE (Scavenger)

Based on previous experience in industrial installations and laboratory studies, an average capacity for organics of 10 to 40 g (as $KMnO_4$) per litre of resin can be expected.

This value should be considered as an approximate indication and it is recommended that some column tests be undertaken on site in order to determine the operating capacity of AMBERLITE IRA958 Cl for a particular water.

HYDRAULIC CHARACTERISTICS (Water treatment)

AMBERLITE IRA958 Cl gives a pressure drop of about 13 kPa/m bed depth per 10 m/h at 15°C.
A backwash flow rate of 6 m/h gives a bed expansion of about 65 % at 15°C.
Pressure drop data are valid at the start of the service run with a clear water and a correctly classified bed.

LIMITS OF USE

Rohm and Haas manufactures special resins for food processing and potable water applications. As governmental regulations vary from country to country, it is recommended that potential users of resins for food processing applications contact their Rohm and Haas representative to assess the best choice of resin and optimum operating conditions.

In Europe, all our products are produced in ISO 9002 certified manufacturing facilities.

CAUTION

Ion exchange resins and polymeric adsorbents, as produced, contain by-products resulting from the manufacturing process. The user must determine the extent to which organic by-products must be removed for any particular use and establish techniques to assure that the appropriate level of purity is achieved for that use. The user must ensure compliance with all prudent safety standards and regulatory requirements governing the application. Except where specifically otherwise stated, Rohm and Haas Company does not recommend its ion exchange resins or polymeric adsorbents, as supplied, as being suitable or appropriately pure for any particular use. Consult your Rohm and Haas technical representative for further information. Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact. Nitric acid and other strong oxidising agents can cause explosive type reactions when mixed with Ion Exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidising agent such as nitric acid is contemplated. Before using strong oxidising agents in contact with Ion Exchange Resins, consult sources knowledgeable in the handling of these materials.

Rohm and Haas Company makes no warranties either expressed or implied as to the accuracy of appropriateness of this data and expressly excludes any liability upon Rohm and Haas arising out of its use. We recommend that the prospective users determine for themselves the suitability of Rohm and Haas materials and suggestions for any use prior to their adoption. Suggestions for uses of our products of the inclusion of descriptive material from patents and the citation of specific patents in this publication should not be understood as recommending the use of our products in violation of any patent or as permission or license to use any patents of the Rohm and Haas Company. Material Safety Data Sheets outlining the hazards and handling methods for our products are available on request.

AMBERLITE is a trademark of Rohm and Haas Company, Philadelphia, U.S.A.

ROHM AND HAAS ION EXCHANGE RESINS

PRODUCT DATA SHEET

AMBERLITE IRA96SB

Industrial Grade Weak Base Anion Exchanger

AMBERLITE IRA96SB is a macroreticular weak base anion exchange resin. Its very stable structure and limited reversible swelling make it very resistant to osmotic shock. The high degree of porosity of this resin provides efficient adsorption of large organic molecules and their desorption during regeneration, thus allowing excellent protection against organic fouling. AMBERLITE IRA96SB is intended primarily for the removal of strong acids from water following a strongly acidic cation exchange resin, and it provides excellent protection against organic fouling for the strong base anion exchange resin placed in the same vessel. The particle size distribution of AMBERLITE IRA96SB has been specifically selected to give optimum performance in stratified bed applications combined with AMBERJET 4400 Cl or AMBERLITE IRA458RF Cl.

PROPERTIES	
Matrix	Styrene divinylbenzene copolymer
Functional groups	Tertiary amine
Physical form	Opaque spherical beads
Ionic form as shipped	Free base (FB)
Total exchange capacity [1]	≥ 1.25 eq/L (FB form)
Moisture holding capacity [1]	57 to 63 % (FB form)
Specific gravity	1.040 to 1.060 (FB form)
Shipping weight	670 g/L
Particle size	
Uniformity coefficient	≤ 1.60
Harmonic mean size	440 - 590 µm
Fine contents [1]	< 0.300 mm : 1.0 % max
Coarse beads	> 0.710 mm : 5.0 % max
Maximum reversible swelling	$\text{FB} \rightarrow \text{Cl}^-$: 15 %

[1] *Contractual value*

Test methods are available on request.

SUGGESTED OPERATING CONDITIONS	
Maximum operating temperature	100°C
Minimum bed depth	700 mm
Service flow rate	5 to 40 BV*/h
Regenerant	NaOH
Flow rate	2 to 8 BV/h
Concentration	2 to 4 %
Level	120 % of ionic load
Minimum contact time	30 minutes
Slow rinse	2 BV at regeneration flow rate
Fast rinse	4 to 8 BV at service flow rate

* *1 BV (Bed Volume) = 1 m^3 solution per m^3 resin*

PERFORMANCE

Operating capacity
The operating capacity of AMBERLITE IRA96SB, when used to deionise water, depends on a number of factors:
Ionic load,
CO_2 content,
SO_4/FMA ratio,
Water temperature.
The Engineering data sheet EDS 0255 A provides information to calculate the operating capacity of AMBERLITE IRA96SB used in water treatment.

Organic matter
Thanks to its high porosity, AMBERLITE IRA96SB can adsorb reversibly organic molecules from solution. It is therefore very useful to protect strongly basic resins from irreversible fouling.

Physical stability
The tough, durable structure of AMBERLITE IRA96SB associated with the limited reversible volume change in service offers excellent resistance to attrition from osmotic or physical stress. In addition, the resin has outstanding resistance to oxidation.

HYDRAULIC CHARACTERISTICS

AMBERLITE IRA96SB gives a pressure drop of about 25 kPa/m bed depth per 10 m/h at 15°C.
A backwash flow rate of 2.5 m/h gives a bed expansion of about 70 % at 15°C.

LIMITS OF USE

AMBERLITE IRA96SB is suitable for industrial uses. For all other specific applications such as pharmaceutical, food processing or potable water applications, it is recommended that all potential users seek advice from Rohm and Haas in order to determine the best resin choice and optimum operating conditions.

In Europe, all our products are produced in ISO 9002 certified manufacturing facilities.

CAUTION

Ion exchange resins and polymeric adsorbents, as produced, contain by-products resulting from the manufacturing process. The user must determine the extent to which organic by-products must be removed for any particular use and establish techniques to assure that the appropriate level of purity is achieved for that use. The user must ensure compliance with all prudent safety standards and regulatory requirements governing the application. Except where specifically otherwise stated, Rohm and Haas Company does not recommend its ion exchange resins or polymeric adsorbents, as supplied, as being suitable or appropriately pure for any particular use. Consult your Rohm and Haas technical representative for further information. Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact. Nitric acid and other strong oxidising agents can cause explosive type reactions when mixed with Ion Exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidising agent such as nitric acid is contemplated. Before using strong oxidising agents in contact with Ion Exchange Resins, consult sources knowledgeable in the handling of these materials.

APPENDIX B

Design Calculations For The Pilot Plant

B.1 Mass Balance for continuous operation of the pilot plant:

B.1.1 Chromatography

Chromatographic Feed flow rate is specified as 14 l/h at 67 Brix.

The density of the feed is thus:[i]

$$\rho = \left[\frac{(200 + Brix)\cdot Brix}{54000}\right] + 1 = 1.33 \text{ kg/l} \tag{1}$$

Q_{feed} = 18.64 kg/h

Assume that for the clean syrup: Brix = Dry Material

then: 12.49 kg Dry Material/h

Mass balance across chromatographic separation:[ii]

Total Balance:

$$Q_{feed} + Q_{water} = Q_{Raffinate} + Q_{Extract} \tag{2}$$

Sugar Balance:

$$Q_{feed}\,X_{feed} = Q_{Raffinate}\,X_{Raffinate} + Q_{Extract}\,X_{Extract} \tag{3}$$

Salt Balance:

$$Q_{feed}\,Y_{feed} = Q_{Raffinate}\,Y_{Raffinate} + Q_{Ekstrak}\,Y_{Extract} \tag{4}$$

According to operating instructions from Applexion –

Q_{water} = 63 kg/h, $F_{Extract}$ = 21 l/h at 30 Brix and $F_{Raffinate}$ = 56 l/h at 9 Brix.

This converts to: $Q_{Extract}$ = 23.7 kg/h; $Q_{Raffinate}$ = 58.2 kg/h

From an analysis of clarified, softened inverted molasses the following results were obtained if one lumps the sugar components in the "sugars" fraction and all the other components in the "salts" fraction:

Table B.1: Sugar and salts fraction

Salts as % of Dry Material	26.21
Sugars as % of Dry Material	73.79

In the 12.49 kg Dry Material/h feed solution, to the chromatography plant, there is:

0.7379 * 12.49 = <u>9.22 kg Sugars/h</u>,

and <u>3.27 kg Salts/h</u> ⇒

Under the assumption that the remaining sucrose after inversion is negligible, the lumped Sugars consists of 48% Glucose and 52% Fructose.

The chromatography plant is thus fed with <u>4.43 kg/h Glucose</u> and <u>4.79 kg/h Fructose.</u>

B1.2 Inversion

The following reaction occurs during inversion of sucrose:

$$\text{Sucrose} + \text{Water} \xrightarrow{[enzyme]} \text{Glucose} + \text{Fructose} \qquad (5)$$

$$\text{MM: } 342.31 + 18.06 \qquad 180.16 + 180.16$$

1 kg Sucrose = 2.92 mole, 2.92 mole glucose/fructose weighs 526.32 g.
1000 g Sucrose produces 1052.64g invert sugar.
Mass increase of <u>5.26 %</u>

Molasses sugar composition indicates the following ratio: 65.67 % Sucrose, 14.41 % Glucose and 19.93% Fructose.

Mass balance across inversion step, where x is the unknown amount of lumped sugar in the feed, reveals:

$$(1.0526)(0.6567)\,x + 0.1441x + 0.1993x = 1.035x = 9.22 \qquad (6)$$

thus:

$$x = 8.911 \text{ kg dry sugars/h}$$

The sugars before inversion are: 1.284 kg Glucose/h
1.776 kg Fructose/h and
5.849 kg Sucrose/h.

The enzyme required to achieve inversion in 24 hours is 20-mg/kg sucrose or 116.98mg/h.

The salt does not take part in the inversion. The salt entering the reactor is 3.27 kg/h.

Thus: 12.18-kg Dry material/h enters the reactor at 65 Brix. The total flow to the reactor is then 18.18 kg/h.

B1.3 Evaporation

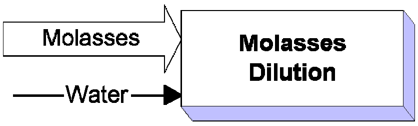
Molasses is clarified at 30 Brix. A solids balance over the evaporator results in 40.6 kg/h of diluted molasses to be concentrated to 65 Brix.

The composition of the stream into the evaporator is:
8.911 kg Sugars/h
3.27 kg Salts/h and
28.42 kg Water/h of which
22.42 kg/h has to be evaporated.

B1.4 Softening

Analysis shows that clarified molasses on average contains about 2000 ppm Ca^{2+} and 2000 ppm Mg^{2+}. This is equivalent to: 99.8 meq/l Ca^{2+} and 164.53 meq/l Mg^{2+}.

At a flow rate of 40.6 kg/h at 30 Brix, we treat 45.79 l/h of solution. Complete softening of the molasses means that 4.5696 eq Ca/h and 7.5335 eq Mg/h had to be removed and replaced by Sodium or Potassium ions.



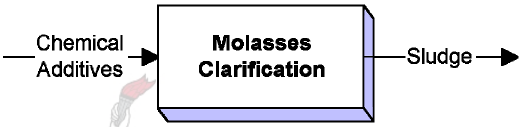
B1.5 Molasses dilution

Molasses dry solids consist of 56.2% sugars. The mass of solids in the raw molasses solution is:

8.911/0.562 = 15.856 kg dry solids/h.

Molasses has 80.45% dry solids, thus: 15.856/0.8045 = 19.71 kg molasses/h is required to feed the plant.

To achieve a 30 Brix solution, the addition of 35.87 kg/h of water is required.



B1.6 Clarification/Filtration

Clarification of molasses removes suspended solids from the molasses. It is also removing some calcium and magnesium salts from the solution. It is required to remove all the suspended solids before the molasses can be softened. Therefore 3.675 kg/h of molasses dry solids will be removed in the clarification/filtration stage.

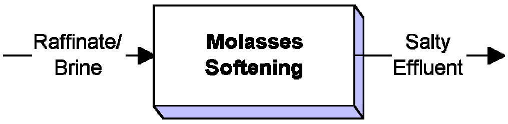
Chemical additives to the molasses are 3.75g phosphoric acid (100%)/ kg dry solids and 5 g caustic soda (100%)/ kg dry solids. Flocculant is dosed as 20 ppm to the molasses solution.

Phosphoric acid required: 3.75*15.856 = 59.46 g H_3PO_4 (100%)/h
Caustic soda required: 5*15.856 = 79.28 g NaOH (100%)/h
Flocculant required: 20*55.58*1.128 = 1.254 g/h

Dicalite 431 will be used as body feed and Dicalite 4251 will be used to precoat the filter.

A 5m²-leaf filter coated with a 2mm layer of precoat would require 0.01m^3 filteraid. The wet cake density of Dicalite 4251 is 162 kg/m^3. Thus 1.62-kg filteraid would be required to coat the filter. The bodyfeed should not exceed the chamber volume of the filter. At a dosage of 2 g/l of molasses a total of 111.16 g/h of Dicalite 431 is required. This will occupy a volume of 427 ml/h and a total volume of 10.26 l/day.

B.2 Equipment design



B2.1 Ion exchange - Softening

Rohm & Haas specify the capacity of Amberlite 252Na[iii] to be 1.8 eq/L, assuming a 75% efficiency of the resin the capacity will be 1.35 eq/L. To treat the molasses one would require 8.963 L/h of resin to be saturated or 215-L resin per 24hr day.

Added a 14% safety factor - the resin requirement would be 250 L/day.

Allow 25% freeboard for expansion of resin during regeneration. Thus the resulting column capacity is 275 litres.

A 400-mm nominal bore stainless steel pipe was used to construct the vessel. The vessel would be 2540 mm high. Maximum pressure in the vessel would be 600 kPa.

Wall thickness (t) of the vessel could be calculated with the following formula[iv]:

$$t = M\left[\frac{Pd_o}{2(SE + PY)} + C\right] \tag{7}$$

Where: P = Maximum operating pressure (gauge)

M = Manufacturing Tolerance (=1.125 assuming a 12.5% tolerance)

E = Joint Factor (= 0.65 for a single butt weld joint)

S = Allowable Stress (= 145000 kPa for 304 S/S)

Y = Coefficient that incorporates ductility of the material (=0.4)

C = Corrosion allowance (4mm)

The pressure due to the weight of the resin and the filled column was added to the maximum operating pressure. The bulk density of resin is 0.81 kg/L and assumes the column is filled to capacity, then:

Pressure due to resin weight[v] $= 0.00981\rho\Delta Z$

$= 0.00981*0.810*2.54 = 0.020$ kPa

Assume column completely filled with molasses at 30 brix:

Pressure due to liquid = $0.00981(1.128)*2.54 = 0.028$ kPa

Total pressure due to internals: $P_I = 0.048$kPa

Total pressure in column = <u>600.048 kPa</u>

$$t = 1.125\left[\frac{600.048*406}{2(145000*0.65+600.048*0.4)}+3\right] = 4.825mm \qquad (7)$$

Use a <u>5-mm</u> wall thickness.

The column will have bolted flanged ends. Two flanges are welded to the ends of the column body. Onto these a distributor plate is bolted, followed by a short pipe section acting as a distribution void space and the open end is closed with a blank flange in which the inlet/outlet stud is welded. The flanges are <u>15 mm thick</u>, made from stainless steel, <u>Outside diameter = 609mm, inside diameter = 406mm</u>. Blank flanges were used as distributor plates. These were drilled and fitted with distribution nozzles. Five nozzles were fitted to each plate. The nozzles were arranged in a pentagon and each hole diameter was 30 mm.

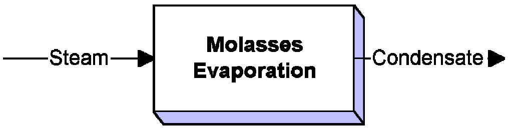
Regeneration with salt: Until equilibrium is reached on the chromatography plant, and until the raffinate could be recycled to regenerate the resin. The resin had to be regenerated with salt. Rohm & Haas suggests the regeneration conditions in Table B2.1:

Table B.2: Regeneration conditions from Rohm & Haas

	NaCl
Flow Rate (BV/h)	2 to 8
Concentration (%)	10
Level (g/L)	80 to 400
Minimum contact time	30 min
Slow rinse	2 BV at regeneration flow rate
Fast rinse	2 to 4 BV at service flow rate

For 250 litres of resin, 50 kg salt will result in a 200g/L level. The amount of water needed to get the right concentration is 450 litres. A flow rate of 2 BV/h would result in a flow of 500l/h. This would satisfy the requirement for minimum contact time. Rinse water requirement will be 500 litre of water, at 500 l/h, for the slow rinse.

The cycle would consist of: operation, de-sweetening of the resin with 250 litres of water at 1000 l/h, regeneration with 500 litre of a 10% salt solution at 500 l/h flow rate followed by a slow rinse with 500 litres water at 500 l/h.



B2.2 Evaporation

From the mass balance it follows that 22.42-kg water/h has to be evaporated. The same evaporator would be used for both the concentration of molasses before inversion and the concentration of chromatography extract. An evaporator would therefore be required to handle the total daily flow at once (batch evaporation).

B2.2.1 Heat Exchanger Specification

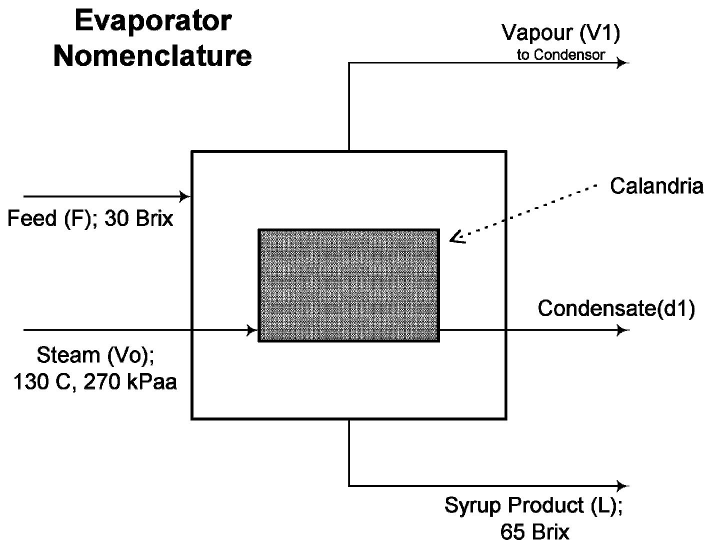


Figure B.1: Evaporator Nomenclature

The following would apply:

Water to be evaporated: = 22.42kg/h * 24 h = 538.5 kg.

Assume that complete evaporation would occur in one hour.

Balances[ii&vi]:

Mass: $V_o + F = L_1 + V_1 + D_1$ (8)

Solids: $30*F = 65*L_1$ (9)

Energy: $V_oH_o + Fh_f = V_1H_1 + L_1h_{L1} + D_1h_{D1}$ (10)

Heat Transfer: $V_oH_o - D_1h_{D1} = V_o \lambda$ (11)

Evaporation Rate: $q = CA\Delta T = 538.5$ kg/h (12)

And[vii] $C = 0.001*(100-B)*(T-54)$ kg Vapour/(h°Cm²) (13)

Where B = average Brix in the evaporator and

T = steam temperature

Thus

$$C = 0.001 \times (100 - (\frac{(65+30)}{2})) \times (130-54) = 3.99 \text{ kg Vapour/(h°Cm}^2\text{)}$$

and ΔT = mean overall temperature difference

= (130 - 65) = 65°C

From the evaporation rate formula follows:

$A = q/(C \times \Delta T) =$ <u>2.08 m²</u>.

From the linear regression of the Dühring line for sucrose solutions the following is derived [vii]:

Boiling Point Elevation (BPE) = 2*Brix/(100-Brix) (14)

For this case: BPE = 3.71℃

Thus V_1 is 3.71℃ overheated and V_1=61.3℃ saturated.

We find: H_o = 2721 kJ/kg

h_f = 223 kJ/kg

H_1 = 2619.7 kJ/kg

h_{L1} = 274.7 kJ/kg

h_{D1} = 546.4 kJ/kg

λ = 2174.6 kJ/kg

Solving the balances results in: $\underline{L_1 = 461.54\ \text{kg}}$

$\underline{F = 1000\ \text{kg}}$

$\underline{V_o = 604.4\ \text{kg}}$

$\underline{D_1 = 604.4\ \text{kg}}$

The calculation for area above is derived for cane juice solutions. The fouling characteristics of molasses are much worse than that of sugar cane juice. The pilot plant steam supply is from the factory's waste heat evaporator. The length of the supply pipe, as well as atmospheric conditions aids in cooling down the steam to the pilot plant. Combining both factors in the assumption that the steam arrives at the pilot plant at 100℃ the following result from the evaporation rate formula:

C = 0.001x(100-47.5)x(100-54)=2.415 kg Vapour/(h℃m²)

And then: A = q/(C x ΔT) = 538.5/(2.415*(100-65)) = $\underline{6.37\ \text{m}^2}$

An evaporator with a heating surface of 6.4 m² should be able to achieve the desired evaporation rate.

B2.2.2 Vapour Liquid Separator

A cyclone will be used as a Vapour-Liquid Separator. The diagram in Figure B2.2.1 indicates the measurements for a high efficiency cyclone:

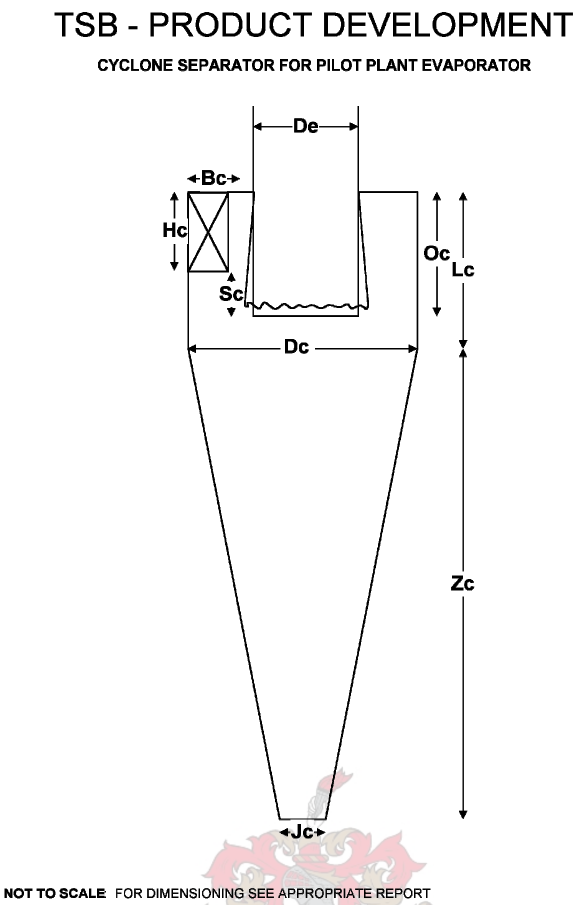


Figure B.2: Diagram detailing the key dimensions of a cyclone [v].

From the design it follows that for a Dc = 500 mm, De = 375 mm, Bc = 100 mm, Hc = 250 mm, Oc = 438 mm, Lc = 750 mm, Zc = 1250 mm, Jc = 188 mm, Sc= 63 mm and a total height = 2000 mm.

Volume of cone = 124 litres.

To separate droplets larger than 10 μm the inlet velocity can be derived from[iv]:

$$D_p^2 = \frac{9 \times \mu \times B_c}{\pi \times N_{TC} \times V_c \times (\rho_s - \rho)} \qquad (15)$$

where: μ = Viscosity (poise) = 3.06 x 10^{-4} poise

π = 3.14159

ρ_s= particle density = 1.3 g/cc

ρ = fluid density = 0.1612 g/cc

B_c = Inlet width = 10.0 cm

D_P = droplet diameter = 0.001 cm (= 10 μm)

N_{TC} = Number of turns made by gas stream

= Length of cyclone/width of inlet = 20

V_c = Inlet gas velocity

This results in V_c = 385.3 cm/s = <u>3.85 m/s</u>

Volumetric flow = V_c x Area = 346 m3/h

In a cyclone entrainment occurs if [iv]:

$$V_c = 6.516 - 0.2865 \ln Re = 6.516 - 0.2865 \ln (4Q/(h_i \upsilon_l)) \quad (16)$$

Where V_c = Cyclone Inlet gas velocity

Re = Reynolds number of liquid on the cyclone wall

Q = Volumetric liquid flow rate

h_i = Inlet height of cyclone

υ_l = kinematic liquid viscosity

In this case Re = 2400204, resulting in <u>V_c = 8.26 m/s</u>

The design is safe and no entrainment would occur.

The pressure drop over the cyclone will be assumed negligible since the unit operates under vacuum.

The wall thickness of the cyclone, under vacuum can be tested with the formula [vi]:

$$P_c = 2.2\, E(t/D_o)^3 \quad (17)$$

Where P_c = Critical pressure to cause buckling = 101.325 kPa

E = Young's modulus = 210 000 MPa

t = Wall thickness in mm

D_o = External diameter = 2t + 500 mm

t = 3.052 mm

say t = 4 mm.

B.2.2.3 Condenser

The evaporator was to be fitted with a condenser to prevent condensation in the vacuum pump. The condensate leaves the condenser, mixes with cooling water and seals the vanes on the vacuum pump. To prevent cavitation in the vacuum pump the condensate should approach the cooling water temperature. For design the ambient temperature at the plant was considered to be 35°C. The cooling water available at the Komati Mill is at 30°C and this will be used for the cooling water requirement of the pilot plant.

For water vapour at 0.25 bar abs. the steam tables yields:

Enthalpy of vapour = 2618 kJ/kg

Enthalpy of water = 272 kJ/kg

Latent heat = 2346 kJ/kg

For 540 kg/h of vapour this results in a condenser duty of 352 kW to condensate the vapour.

Assume condensate will leave the condenser at 45°C and the cooling water temperature will rise with 10°C, from 30°C to 40°C.

For the condenser the overall heat transfer coefficient is determined as 1700 $w/m^2 {}^\circ C$

The log mean temperature difference for the condensation section is [viii] :

$$\Delta T_{lm} = \frac{(t_2 - t_1)}{\ln\left[\frac{T_{sat} - t_1}{T_{sat} - t_2}\right]} \qquad (18)$$

where T_{sat} = Saturation temperature of water = 65°C

t_1 = cooling medium inlet temperature = 30°C

t_2 = cooling medium outlet = 40°C

thus

ΔT_{lm} = 29.7 °C

Thus the area required for condensation is: A = 6.97 m².
For the cooling section:

$$\Delta T_{lm} = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln\left[\dfrac{T_1 - t_2}{T_2 - t_1}\right]} \qquad (19)$$

where T_1 = inlet temperature of condensate = 65°C
T_2 = outlet temperature of condensate = 45°C
t_1 = cooling medium inlet temperature = 30°C
t_2 = cooling medium outlet = 40°C

thus

ΔT_{lm} = 19.6 °C

This results in an area required for cooling: A = 0.38 m².

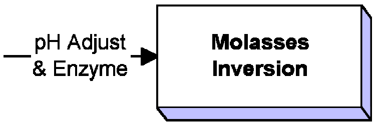
The total area for the condenser is thus 7.35 m².

The condensing section would require a cooling water flow of 8.4 kg/s (30.3 m^3/h) and the cooling section would require 0.30 kg/s (1.08 m^3/h).

The cooling utility is to be piped from and the hot utility returned back to the factory's bearing cooling water (BCW) circuit passing overhead of the pilot plant. The pumps at the cooling tower delivers water at a pressure of 600-kPag. This water is reticulated through the factory and the hot water is collected in a tank on the factory's fifth floor from where it gravitates back to the cooling towers. The pilot plant cooling water circuit was designed to provide for adequate flow when needed to the condenser. From design principles the following pipe specification was obtained:
Equivalent length of pipe = 52 m
Friction Pressure drop = 137.8 kPa
Linear velocity of water in pipe = 4.22 m/s (within range $1 \leq u \leq 5$ m/s)

Pipe: Outside Diameter = 60.3 mm
Inside Diameter = 51.3 mm
Wall thickness = 4.5 mm

Thus a 50-mm nominal diameter pipe would be sufficient to provide cooling water to the condenser.



B.2.3 Inversion

The inversion of sugar to glucose and fructose is a well-known process in the sweetener industry. Either acid or enzyme can accomplish inversion. It was found that the buffer capacity of molasses causes an excessively large quantity of mineral acid to bring the pH to about pH=2 for hydrolysis. Apart from cost of acid and caustic to neutralise the molasses, the additional salt also had to be removed on the chromatography.

The following enzyme reaction takes place, in acidic conditions (pH range 4.5 - 5.5):

$$\text{Sucrose} + \text{Water} \xrightarrow{[\mathit{Invertase}]} \text{Glucose} + \text{Fructose} \qquad (5)$$

$$\text{MM: } 342.3051 + 18.0157 \qquad 180.1604 + 180.1604$$

$$C_{12}H_{22}O_{11} + H_2O \xrightarrow{[\mathit{Invertase}]} C_6H_{12}O_6 + C_6H_{12}O_6$$

A suitable enzyme from Gist Brocades known as "Maxinvert 200,000" was decided upon. The time to reach 99% inversion of a sucrose solution with various dosages of enzyme is listed below.

Table B.3: 99% Inversion of a sucrose solution with dosages of enzyme

Dosage g/kg Sucrose	**Time (h) to reach 99% conversion**
0.05	> 24
0.10	12
0.15	8
0.20	6

The enzyme dosage can be converted to the common unit used by Gist Brocades, the SU where: 1000 SU = 0.05g enzyme/ kg dry sucrose. It follows that enzyme dosed at 0.1g enzyme/kg sucrose (2000 SU) is both economical and practical. The temperature for inversion was set at 60℃ and the molasses was acidified to pH between 4.5 and 5.5 using hydrochloric acid.

Gist Brocades lists the following conversions at the conditions above:

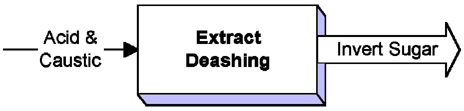
Table B.4: Enzyme inversion of sucrose by Gist Brocades.

Time (h)	Sucrose g/kg	Glucose g/kg	Fructose g/kg	Conversion %
0	495.0	<0.1	<0.1	0
2	148.0	144	174	70.1
4	51.0	199	230	89.7
6	23.2	216	248	95.3
8	12.0	231	269	97.6
12	4.7	241	263	99.0
14	3.2	246	266	99.4
16	3.9	243	267	99.2
18	2.6	239	263	99.5
20	2.4	241	266	99.5
24	2.2	242	270	99.6

According to the results obtained in the Gist Brocades experiments it is clear that 14 hours (or longer) incubation time should be sufficient to, almost, completely invert the sucrose in a sugar solution. Because molasses, even after softening, is not a pure sucrose solution, and the fact that the concentrations between the Gist Brocades experiments and the proposed operation of the pilot plant differ, it was decided that for contingency the inversion should be allowed to continue for 20-24 hours. The inversion was to be confirmed by analysis of sucrose, glucose and fructose on an HPLC. Should precipitation occur, or clarity diminishes during the inversion step the product would be filtered before chromatographic separation.

Because of this delay it was decided to operate the pilot plant initially in a batch configuration, while in the final stages of inversion, a new batch would be diluted and processed upstream.

B2.4 De-ashing of Chromatography Extract



The following results were obtained by Applexion during September 1997 and used as the basis for the design of the chromatography unit.

Extract Brix: 30.0
Conductivity of extract: 485 µS
Purity of extract: 97.7% (99.4% when a purity correction is taken into account following a check test)

For the deashing Rohm & Haas Amberlite 252RFH were used as the cation resin and Amberlite IRA92 as the anion resin. Amberlite 252RFH is a macroporous resin based on sulphonated crosslinked polystyrene. The resin was developed for use in packed and floating beds and is very resistant to osmotic shock and mechanical attrition. Amberlite IRA92 is a high capacity macroporous polystyrene weak base resin. It is highly effective in the uptake of strong acids when used downstream from a strong acid cation exchanger in the H-form. It is also highly effective in the adsorption and desorption of organic colouring matter. This resin also shows excellent osmotic and mechanical stability. The detail datasheets of all the resins discussed in this study are included in Appendix A.

The design was made using an extract purity of 97.7% and assuming the remaining 2.3% is ash.

No detailed ash analysis was available and the following assumptions were made:

- Ash consists only of sodium (M=22.9898) and potassium (MM=39.098) cations when the softener is in equilibrium after regeneration with raffinate. Initial conditions would only feed sodium cations to the chromatographic separator. Therefore assume only sodium cations will be present in the extract. (Potassium will be present due to ion exchange occurring on the resin.)

- All the ash components will be simple salts in the form Na-Xx. Thus there will be one anion for very one sodium cation.
- At high sugar purities, Brix and actual solid content are equal.

From the mass balance and the Applexion data it follows that one would treat 1500 litres/day of extract. This results in 11.39-kg ash/day that has to be removed on the resin.

This is equivalent to 247.6 mole sodium ions in solution, or 0.165 eq/l.

The listed capacity for 252RFH is[ix] 1.7 eq/L, resulting in 145.7 Litre of resin at full efficiency.
The listed capacity for IRA92 is[x] 1.6 eq/L, resulting in 154.8 Litre of resin at full efficiency.

To ensure complete ion exchange in non-ideal conditions and to compensate for variations in resin selectivity for certain ionic species, the anionic resin was assumed to be 60% effective and the cationic resin 70% effective. It resulted in a total resin requirement of 247.6 Litres of both cation and anion resin.

The column will be similar to the one specified for softening. The distributor plates differ only in nozzle distribution, since a different nozzle will be used. Nozzles on the deashing columns have a lower pressure drop across the distributor plate than the ones in the softener. They were selected to reduce the total pressure drop over the four columns. For the deashing columns seven nozzles per plate are specified, arranged in a hexagon, with a nozzle in the centre. Nozzle holes for these nozzles are 35 mm in diameter.

Regeneration of the resins would be done with hydrochloric acid for the Amberlite 252RFH and with caustic soda for the Amberlite IRA92. Rohm & Haas lists the following conditions for regeneration:

Table B.5: Conditions for regeneration listed by Rohm & Haas

	252 RFH	**IRA92**
Regenerant	**HCl**	**NaOH**
Flow Rate (BV/h)	4 to 6	2 to 8
Concentration (%)	4 to 10	2 to 6
Level (g/L)	45 to 100	40 to 80
Minimum contact time	30 min	30 min
Slow rinse	2 BV at regeneration flow rate	2 BV at regeneration flow rate
Fast rinse	2 to 4 BV at service flow rate	4 to 8 BV at service flow rate

For 250 litres of resin, 15 kg of pure regenerant will be at the 60 g/L level. Thus 15 kg of caustic soda in 285-kg water will result in a 5% solution. This must be pumped through the anion column at a maximum flow rate of 500 kg/h. The regeneration must be followed with a slow rinse of 500 litres of water, flowing at 500 l/h.

For the cation resin, 15 kg of pure hydrochloric acid results from 50 kg 30% solution. This 50-kg of commercial acid must be diluted in 250-kg water to give a 5% solution. The acid solution should be pumped at 1000 l/h to meet the required flow rate. The contact time would then be 20 minutes, which would not be sufficient to allow proper regeneration. The required contact time would be met using 500 litres of regeneration solution. This would consist of 84-kg of commercial hydrochloric acid diluted in 416-kg water. The regeneration must then be followed with a slow rinse using 500 litres of water, flowing at 1000 l/h.

B.3. Utilities Supply

The pilot plant required the following utilities from the factory:

Instrument Air: Operation of the pneumatic actuators on the control valves
Applexion pilot plant - 8 Nm^3/h - maximum

Pre-treatment plant - 3 Nm^3/h - maximum

Cooling Water: Condenser cooling, vacuum pump lubrication and the discharge of the centrifuge sludge - 32 m^3/h at maximum consumption

Domestic Water: Washing of tanks and plant - 1000 l/day

Condensate: Pure water to dilute molasses and act as a heating medium - 1000 l/day

Steam: Evaporator heating and heating of condensate to maintain plant temperature. - 1500 kg/day

Electricity: A 160 A, 550 V supply is fed into the pilot plant substation and transformers to provide the required power for the plant:

Installed Power - 54 kW consisting of:
Chromatography - 6 kW
Pre-treatment - 45 kW
Office & Plant Lighting - 3 kW

Effluent generated by the pilot plant is collected in a tank and pumped into the factory's effluent removal circuit.

References for Appendix B

[i] Applexion Laboratory Manual – used during a related project in 1996

[ii] Himmelblau, DM (1982) Basic Principles and Calculations in Chemical Engineering, 4th Edittion, 1982, Prentice-Hall.

[iii] Rohm and Haas Co. (1996) Amberlite IR120 Technical Specification Sheet.

[iv] Green, DW (1984) ed. Perry's Chemical Engineers' Handbook, 6th Edition, 1984, McGraw Hill

[v] Sinnot, RK (1993) ed. Coulson & Richardson's Chemical Engineering, Volume 6, Design, 1993, Pergamon Press.

[vi] Coulson JM, Richardson JF *et.al.* (1991) Coulson & Richardson's Chemical Engineering, Volume 2, Particle Technology and Separation Processes, 1991, 4th Edition, Pergamon Press.

[vii] Hugot, E (1972). Handbook of Cane Sugar Engineering, translated by GH Jenkins, 2nd Edition, 1972, Elsevier Press, New York.

[viii] Honig, P (1963). Principles of Sugar Technology, Volume III. Elsevier Publishing Company, New York.

[ix] Rohm and Haas Co. (1996) Amberlite 252RFH Technical Specification Sheet.

[x] Rohm and Haas Co. (1996) Amberlite IRA92 Technical Specification Sheet

APPENDIX C

Results from the Centrifugal Separation of B-Molasses

- Trial Descriptions and Results

C.1: Trials with phosphoric acid/caustic soda.

Two initial runs on the machine were done to familiarise the operators with the operating procedure and to develop a standard for the separation obtainable without any additives. The trials were conducted with approximately 30 Brix solutions at 65℃ and a flow rate of 450 l/h and 900 l/h respectively. The results are listed in Table C1 below:

Table C.1: Trials with phosphoric acid and caustic soda

Trial	Brix	ICUMSA Turbidity Feed (Turb/Brix)	ICUMSA Turbidity Mother Liquor (Turb/Brix)	Turbidity Removed
BM01	26.6	47899 (1801)	26640 (1001)	44%
BM02	36.1	46060 (1276)	33057 (915)	28%

Applexion had experience with the clarification of cane molasses and in a report mentioned the use of phosphoric acid to assist in the removal of suspended solids from molasses. As a first attempt to evaluate the separation performance of the centrifugal separation, the mother liquor of BM01 was treated further by adding 0.5 % H_3PO_4 (as 100%) on Brix, and allowing the mixture to react at 65℃ for one hour before being neutralised with NaOH. This solution was then centrifuged at 1200 l/h and yielded the following result:

Table C.2: Further treatment of trial BM01

Trial	Brix	ICUMSA Turbidity Feed (Turb/Brix)	ICUMSA Turbidity Mother Liquor (Turb/Brix)	Turbidity Removed
BM03	26.4	24874 (942)	19121 (724)	23%

The total reduction in turbidity for the molasses used in BM01 and BM03 was 60%. Diluted molasses have a pH around 5.5 ± 0.4, after neutralisation the pH is 7.0 ± 0.4

To reduce the amount of water that needs to be evaporated at a later stage a solution with a higher concentration was prepared. This was centrifuged at 600l/h to yield the first mother liquor. Phosphoric acid was added to the mother liquor at a ratio of 0.5% (as 100%) on Brix. The mixture was allowed to stir at 65℃ for one hour before neutralisation with caustic soda. This mixture was centrifuged to yield the second mother liquor.

Table C.3: High concentration, double centrifuged molasses

Trial BM04	**Brix**	**ICUMSA Turbidity (Turb/Brix)**	**Turbidity Removed**
Feed	34.6	52153 (1507)	
Mother Liq. 1	34.4	28830 (838)	44.7%
Mother Liq. 2	32.0	20269 (633)	29.7%

As was observed during the BM01/03 trial, the first clarification step reduced the turbidity by 44%. The second clarification, after phosphitation, produced a further drop of 25% in the turbidity. This resulted in a total turbidity removal of 58%.

C2. Effect of H_3PO_4 addition

A trial was done where the amount of phosphoric acid was altered on the same feed solution. A 30 Brix solution of B molasses was made up and 0.5% phosphoric acid on Brix was added, and then left for one hour before being neutralised and half of it centrifuged, to give mother liquor 1. To the remainder another 0.5% phosphoric acid was added, left for one hour, neutralised and centrifuged to give mother liquor 2. Both the mother liquors were filtered to give filtrate 1 and filtrate 2 respectively.

Table C.4: The effect of phosphoric acid on clarification

Trial BM05	Brix	ICUMSA Turbidity	Turbidity/Brix (% removed from feed)
Feed	27.3	48521	1777
Mother Liq. 1	26.9	19433	722 (59%)
Filtrate 1	18.05	18023	998 (44%)
Mother Liq. 2	26.4	32385	1226 (31%)
Filtrate 2	16.91	12003	709 (60%)

This experiment was repeated at a slightly higher Brix. A similar trend to the trial above was found.

Table C.5: Clarification at higher Brix

Trial BM06	Brix	ICUMSA Turbidity	Turbidity/Brix (% removed from feed)
Feed	32.45	44264	1366
Mother Liq. 1	30.9	27652	894 (35%)
Filtrate 1	19.3	14984	776 (43%)
Mother Liq. 2	31.1	32452	1043 (24%)
Filtrate 2	17.2	16523	960 (30%)

A great deal of dilution occurred during these trials. The sludge-discharging step on the centrifuge also added water to the process stream. The filter is coated with Dicalite made into slurry with water. The filter chamber has to stay wet and molasses displaces the water in the filter chamber and in the precoat. The large filter chamber caused a significant dilution of the molasses. The filter blocked after treating 500 litres of molasses. It had to be cleaned, and a new precoat cake had to be built to complete the filtration of the mother liquor. The addition of a second amount of phosphoric acid did not have an advantage. The resultant overall turbidity removal was actually less than that of the molasses only receiving half of the phosphoric acid dosage.

Even with a reduction in turbidity of 60%, the molasses is not yet clean enough for use on resin.

C3. Trials with Carbonation

Finnsugar described a process where Na_2CO_3 was used to treat molasses. A trial was done using Na_2CO_3 with the intention of precipitating $CaCO_3$ in the place of phosphates. The pH of the molasses was adjusted to nine by the addition of Na_2CO_3 to the batch. After six hours at 65 ℃ the batch was centrifuged to remove precipitate. Eleven kilograms of powdered Na_2CO_3 were required to treat 500 litres of molasses. The batch could not be processed completely because the precipitated $CaC0_3$ plugged the outlet ports of the centrifuge bowl. The following results were obtained from the processed part of the batch.

Table C.6: Carbonation of B-Molasses

Trial BM08	Brix	ICUMSA Turbidity	Turbidity/Brix (% removed from feed)
Feed	28.38	36446	1284
Mother Liq. 1	20.6	24396	1184 (8%)

Another trial was conducted with Na_2CO_3. The desludging rate on the centrifuge was increased. A cycle now consisted of a partial desludges every two minutes and a full desludge after two partial cleans. This did not block the centrifuge ports but did not produce an acceptable product either. A routine inspection revealed that a large amount of sludge was stuck inside the sludge collection dome. Upon dismantling inspection revealed that the disks were severely plugged with sludge. The bowl including the disk stack required extensive cleaning. An attempt was made to filter the mother liquor but it plugged the filter before any significant amount of filtrate was produced.

A Spectroquant became available and turbidity was also determined in NTU to compare the results to that of other workers in the field. The results for the second carbonatation trial are listed in the following table.

Table C.7: Carbonatation trial of B-molasses

Trial BM10	Brix	ICUMSA Turbidity	NTU/Brix (Spectroquant)	Turbidity/Brix (% removed from feed)
Feed	30.12	57984	701	1925
Mother Liq. 1	26.35	24330	512	923 (52%)

It was concluded that clarification, using carbonation was not any better than that using phosphoric acid/caustic soda. Plugging of the ports of the centrifuge and also the pressure leaf filter appear to be insurmountable obstacles to overcome before carbonation can be qualified as a process.

C4. Trials with Flocculants

An extensive test program to identify suitable polymeric flocculants that would assist in the removal of suspended solids from the molasses, were launched.

Flocculant trials were conducted with samples received from Aquatan, an agent for NCP flocculants. Flocculants were dosed at 200ppm on the molasses mixture without the addition of any chemicals. Flocculants were prepared by allowing the polymer to hydrate fully in a 1000-ppm solution with water. The flocculants used in the trials were all cationicly charged. They were: C498 for trial BM11, C494 for trial BM12, and a 50/50 mixture of C498 and C496 for trial BM13 and C496 for trial BM13 and C448 for trial BM14. After flocculation the molasses was centrifuged three times yielding the results reported in Table C.8.

The feed analysis for trial BM12 is missing. It is evident that addition of flocculants to the mixture did improve the clarification of the molasses. The clarity of the molasses was not yet suitable for chromatography. Further turbidity removal by filtration of the solutions proved marginally successful.

Table C.8: Clarification with flocculants

Trial #	Brix Feed	ICUMSA Turbidity Feed (Turb/Brix)	NTU/ Brix Feed	Brix Mother Liquor 3	ICUMSA Turbidity Mother Liquor (Turb/Brix)	NTU/ Brix Mother Liquor	% ICUMSA Turb. Removed
BM11	28.7	71317 (2485)	672	26.2	6363 (243)	N/d	90%
BM12	N/d	N/d	N/d	32.7	38749 (1185)	349	N/d
BM13	33.6	37560 (1118)	455	33.9	20528 (606)	204	46%
BM14	37.6	47281 (1257)	904	41.3	11068 (268)	218	79%

The filter had to be desweetened, cleaned and precoated after every 500 litres processed. Some filter-aid did leak through at stages causing unexpected increases in turbidity.

C5. Trials with Flocculants on Previously Clarified Molasses

A trial was conducted to see whether the clarification of molasses could be improved with the addition of flocculants to an already clarified mother liquor. The original flocculant used was C448 (NCP - Aquatan), a cationic polyacrylamide, dosed at 200 ppm on the molasses. After centrifugation the molasses was inverted and dosed with 200 ppm of Magnafloc LT27 (Allied Colloids and used in the sugar mill's juice clarification step) an anionic polyacrylamide flocculant. The rationale being that the anionic flocculant would bind with the remainder of the cationic flocculant that passed through the centrifuge. This should form large flocculant clusters that would aid the clarification of the mixture. Anionic flocculant that is not acting on the cationic remnants should attract and remove particles repelled by the cationic flocculant. The following results were obtained:

Table C.9: Reworking the previously clarified molasses.

Trial BM15	Brix	ICUMSA Turbidity (Turb/Brix)	NTU/Brix (Spectroquant)	% Turb Removed (ICUMSA)
Feed	37.56	37397 (995)	334	
Mother Liq. 1	40.7	13436 (330)	226	66
Mother Liq. 2	37.9	9600 (253)	302	23

The solution did not form any floc particles that were visible. Separation was also not that significant, as it did not produce a clear enough product for downstream processing.

C6. Molasses Treatment with Sulphuric Acid

The SMRI was investigating the clarification of molasses by the addition of H_2SO_4. In this trail H_2SO_4 was added to the molasses to drop the pH to a value of two. A total of 15 litres of H_2SO_4 (98%) was needed to achieve the desired pH in a 1000 litre molasses solution. After a reaction time of 45 minutes at 95 °C, 200 ppm DPC56C was added to the molasses.

Table C.10: B-molasses with sulphuric acid

Trial BM16	Brix	ICUMSA Turbidity (Turb/Brix)	NTU/Brix (Spectroquant)	% Turbidity Removed (ICUMSA)
Feed	36.4	83981 (2307)	110	
Mother Liq. 1	33.32	26433 (793)	105	66%
Mother Liq. 2	34.3	13302 (388)	102	83%

Referring to the trend in ICUMSA values is evident that 83% turbidity was removed. The NTU/Brix results do not support this observation. The sulphuric acid caused the brown molasses mixture to turn black due to the dehydration and charring of the organic compounds specifically sugars. The solution had a charred smell. It was concluded that concentrated sulphuric acid does not offer a suitable pre-treatment option for clarification. The use of H_2SO_4 was not further investigated because of the large volume of acid required to achieve the desired pH and the fact that the acid dehydrates the sugars.

C7. Trials with Chemical additives and Flocculants

Based on literature indications the feasibility of combining the phosphoric acid clarification with that of the flocculant was investigated. Molasses was diluted to about 30 Brix, 100-ppm flocculant DPC56C was added followed by 9 g/l

H_3PO_4 (as 100%). It was allowed to react for one hour before 9 g/l NaOH(s) was added. A further 100-ppm DPC56C was added to allow agglomeration of the formed particles. It was centrifuged at 1200 l/h to remove the sludge.

Table C.11: B-molasses clarification with additives and flocculants

Trial #	**Brix Feed**	**ICUMSA Turbidity Feed (Turb/Brix)**	**NTU/ Brix Feed**	**Brix Mother Liquor**	**ICUMSA Turbidity Mother Liquor (Turb/Brix)**	**NTU/ Brix Mother Liquor**	**% ICUMSA Turb. Removed**
BM17	31.7	70461 (2223)	514	29.78	1977 (66)	57	97 %
BM18	36.9	88978 (2411)	637	38.1	3951 (104)	21	96 %

The great extent of turbidity removal (97 & 96%) indicated that clarification could be improved by a combination of phosphatation and flocculation. It was also evident that the floc that formed tended to settle rather than stay in suspension. Investigations into the treatment of C-molasses got under way to evaluate the process on the feedstock that would ultimately be used in an invert sugar plant.

APPENDIX D:
Centrifugal Clarification Of C-Molasses
– Trial Descriptions and Results

D.1. Trials with flocculant only

For control purposes two trials were done without the addition of flocculant. This served as a basis against which the degree of clarification could be measured.

The flocculants that were investigated are MAGNAFLOC LT27 (Allied Colloids), DPC56C (NCP - Aquatan), DPC4D9B (NCP - Aquatan) and ACC4110 (Cetco, USA). These were dosed as a 1000-ppm solution to the molasses according to the schedule presented in Table D.1.

Table D.1: Centrifugal Separation of C-Molasses with various flocculants.

Trial #	Flocculant Used	Dosage of flocculant to molasses (ppm)	Flocculant Charge
99-01	Magnafloc LT27	15	Anionic
99-02	DPC56C	15	Cationic
99-03a	DPC4D9B	15	Cationic
99-03b	DPC4D9B	10	Cationic
99-04a	DPC4D9B	10	Cationic
99-05a	DPC4D9B	10	Cationic
99-06	DPC4D9B	15	Cationic
99-07a	Magnafloc LT27	15	Anionic
99-08a	Magnafloc LT27	15	Anionic
99-08b	DPC4D9B	15	Cationic
99-08c	ACC4110/DPC56C (50/50)	15	Cationic
99-08d	DPC56C	15	Cationic
99-09a	Magnafloc LT27	15	Anionic
99-09b	DPC4D9B	15	Cationic
99-09c	DPC56C	15	Cationic
99-10a	Magnafloc LT27	15	Anionic
99-10b	DPC4D9B	15	Cationic
99-10c	DPC56C	15	Cationic
99-20	None	N/A.	
99-21	None	N/A.	

The molasses was centrifuged at 600-l/h throughput. The centrifugal pump of the previous experiments was replaced with a mono pump for the rest of the trials. It was believed that the turbulence caused by the impeller of the pump caused floc clusters to disintegrate. The results are reported in Table D.2.

Table D.2: Centrifugal Separation of C-Molasses with various flocculants using a Mono-pump.

Trial # (Floc-culant used)	**Brix Feed**	**ICUMSA Turbidity Feed (Turb/Brix)**	**NTU/ Brix Feed**	**Brix Mother Liquor**	**ICUMSA Turbidity Mother Liquor (Turb/Brix)**	**NTU/ Brix Mother Liquor**	**% NTU/Brix Change**
99-01 (LT27)	18.75	60786 (3242)	192	19.15	23483 (1226)	36.55	81 %
99-02 (56C)	20.5	62953 (3071)	219.51	20.03	28749 (1435)	39.94	82 %
99-03a (4D9B)	17.25	73064 (4236)	208.7	16.0	15778 (986)	37.5	82 %
99-03b (4D9B)	17.25	73064 (4236)	208.7	17.25	40868 (2369)	34.78	83 %
99-04a (4D9B)	19.48	48275 (2478)	169.4	19.0	26297 (1384)	39.32	77 %
99-05a (4D9B)	19.6	86255 (4401)	212.04	20.0	31184 (1559)	67.75	68 %
99-06 (4D9B)	22.7	58850 (2593)	242.29	21.3	38007 (1784)	98.59	59 %
99-07a (LT27)	13.3	81595 (6135)	548.87	12.75	29279 (2296)	31.45	94 %
99-08a (LT27)	15.96	90626 (5678)	463.66	17.64	55318 (3136)	18.42	96 %
99-08b (4D9B)	18.63	101113 (5427)	305.96	17.8	63116 (3546)	162.92	47 %
99-08c (MIX)	14.83	76373 (5150)	289.21	12.5	17224 (1378)	102.4	65 %
99-08d (56C)	18.53	92161 (4974)	339.99	17.45	7026 (403)	57.31	83 %
99-09a (LT27)	13.41	108996 (8128)	253.54	12.82	33111 (2583)	117	54 %
99-09b (4D9B)	14.42	79404 (5507)	340.71	12.96	42681 (3293)	72.3	79 %
99-09c (56C)	13.9	62758 (4515)	262.95	13.55	37511 (2768)	228.78	13 %
99-10a (LT27)	13.4	96178 (7177)	265.15	12.38	64662 (5223)	172.37	35 %
99-10b (4D9B)	13.4	96178 (7177)	265.15	12.32	48525 (3939)	39.61	85 %
99-10c (56C)	14.0	86544 (6182)	474.79	14.35	38481 (2682)	112.61	76 %
99-20 (NONE)	18.1	88333 (4880)	469.06	17.25	76131 (4413)	394.09	16 %
99-21 (NONE)	17.4	63370 (3642)	798.85	17.42	71780 (4121)	280.6	65 %

The findings from the laboratory experiments are also evident in the table above: Trial 99-03a and 99-06 differ only in concentration and the clarification was much more efficient during Trial 99-03a with a lower concentration. The clarification behaviour of each type of flocculant is graphically represented below in Figures D.1 to D.3.

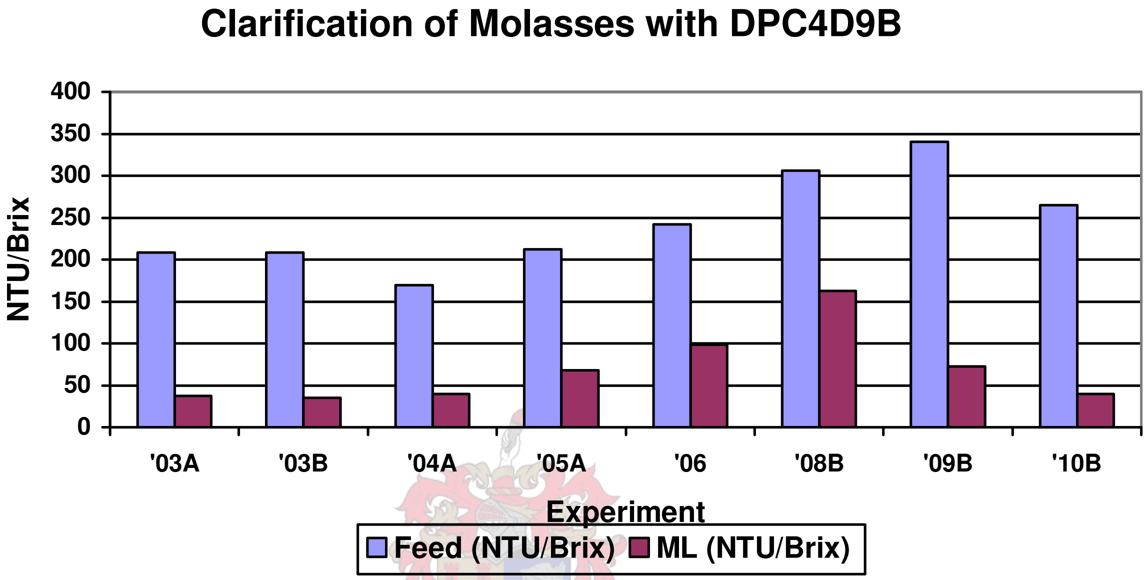


Figure D.1: C Molasses clarified with DPC4D9B

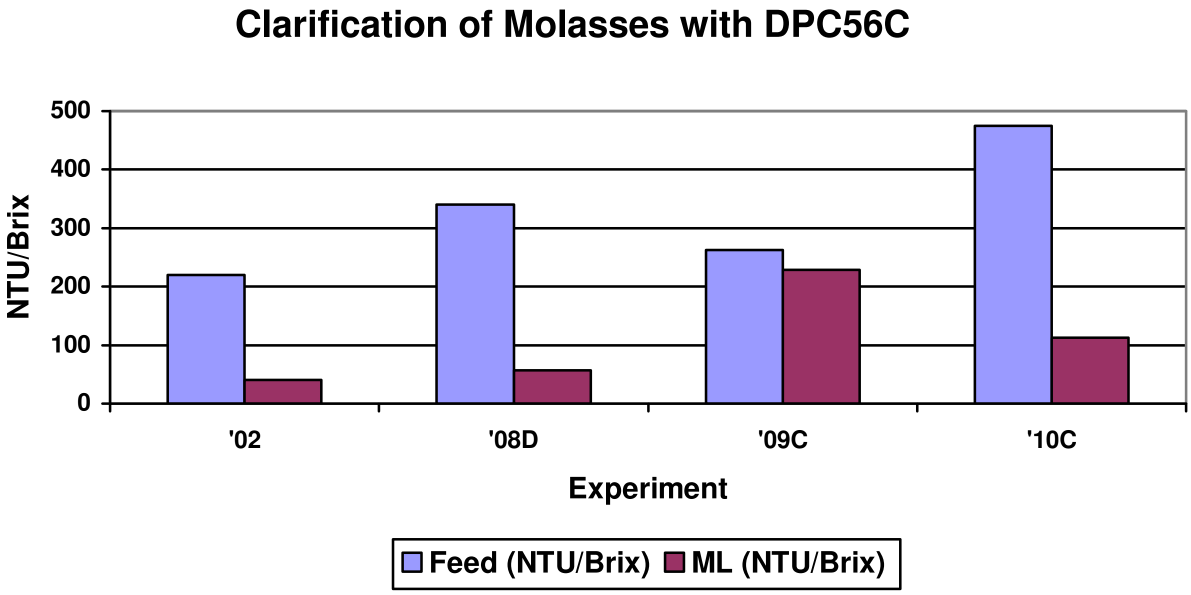


Figure D.2: C Molasses clarified with DPC4D9B

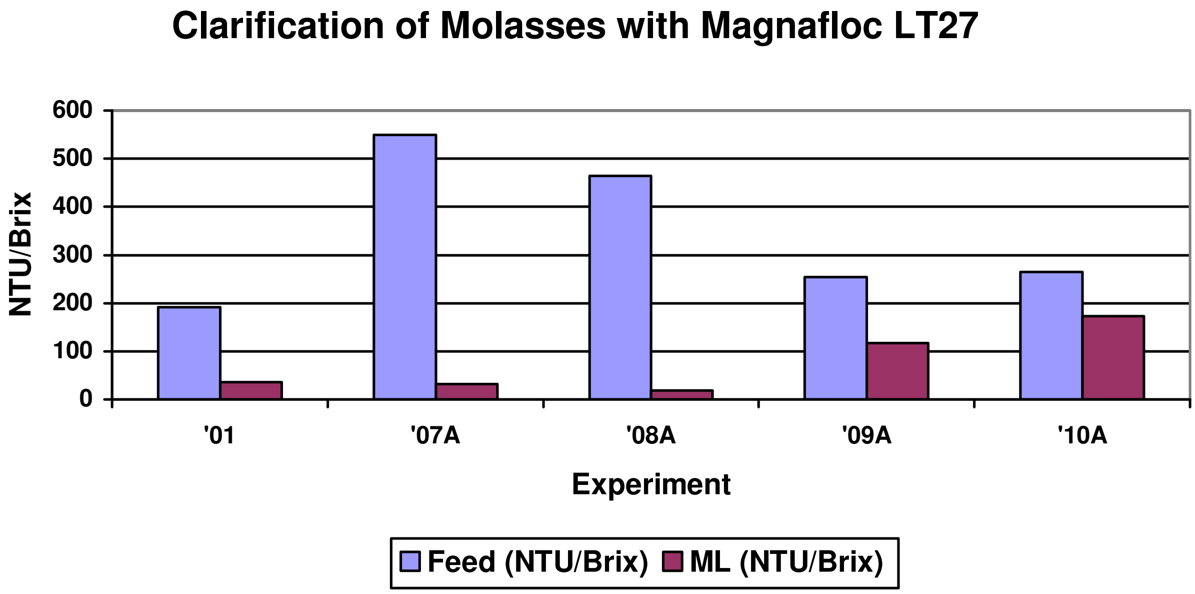


Figure D.3: C Molasses clarified with Magnafloc LT27

It is evident from the results that, even at low concentration, flocculants alone are not able to produce a product with suitable clarity. The same trend was obtained during these trials as the the trend obtained during the experiments on B Molasses. During trials 99-07 to 99-10 the process was operated at low concentration and at ambient temperature. Magnafloc produced excellent results in the first two trials. The clarification could however not be repeated in trial 99-09a and 99-10a. The results were not very consistent over the trials.

What is notable is that the cationic flocculants gave a much more consistent clarification result than the anionic flocculant. This means the turbidity forming compounds are mostly negatively charged, or better attracted to the positive charge of the flocculant. The combination of chemical addition and flocculation was further investigated with C-molasses.

D.2. Trials with phosphatation and flocculation

Failing to achieve a suitable product by using flocculants alone a series of trials were launched during which the effects of phosphatation were investigated. These trials involved the addition of phosphoric acid (usually 3g H_3PO_4 (as 100%) / kg Brix) to the molasses, allowing 30 to 60 minutes to mix and neutralising the solution with caustic soda lye (the usual amount of caustic needed is 5g NaOH(s)/kg Brix). Flocculant was then added to the molasses mixture as a 1000-ppm solution and the batch was centrifuged. The centrifuge was operated at a throughput of 600 l/h.

The trials were conducted with the following flocculants DPC4D9B (NCP - Aquatan), Talosep A3XL (Tyte & Lyle - Enzymes SA), DDPC56C (NCP - Aquatan), Magnafloc LT27 (Allied Colloids), ACC4110 (Cetco, USA), 4195 (Protea Industrial Chemicals), 6197 (Protea Industrial Chemicals) and 6195 (Protea Industrial Chemicals).

The following table presents a summary of the trials conducted**:**

Table D.3: Description of the trials with Phosphatation

Trial #	H_3PO_4 Added	NaOH Added	Flocculant Used	Flocculant Dosage to Molasses (ppm)
99-04b	To pH=4,5	To Neutral	DPC4D9B	10
99-05b	To pH=4,5	To Neutral	DPC4D9B	10
99-11a	3g/kg Brix	5g/kg Brix	Talosep A3XL	12
99-11b	3g/kg Brix	5g/kg Brix	DPC56C	12
99-11c	3g/kg Brix	5g/kg Brix	DPC4D9B	12
99-11d	3g/kg Brix	5g/kg Brix	Magnafloc LT27	12
99-13	3g/kg Brix	5g/kg Brix	No Floc	N/a
99-14	3g/kg Brix	5g/kg Brix	No Floc	N/a
99-15	3g/kg Brix	5g/kg Brix	ACC4110	20
99-16	3g/kg Brix	To Neutral	ACC4110	10
99-17	3g/kg Brix	To Neutral	4195	20
99-18a	3g/kg Brix	To Neutral	6197	20
99-18b	3g/kg Brix	To Neutral	6195	20
99-19	3g/kg Brix	To Neutral	6195	20

Two trials were done with only chemical treatment and no flocculant. The following results were obtained from the trials listed above:

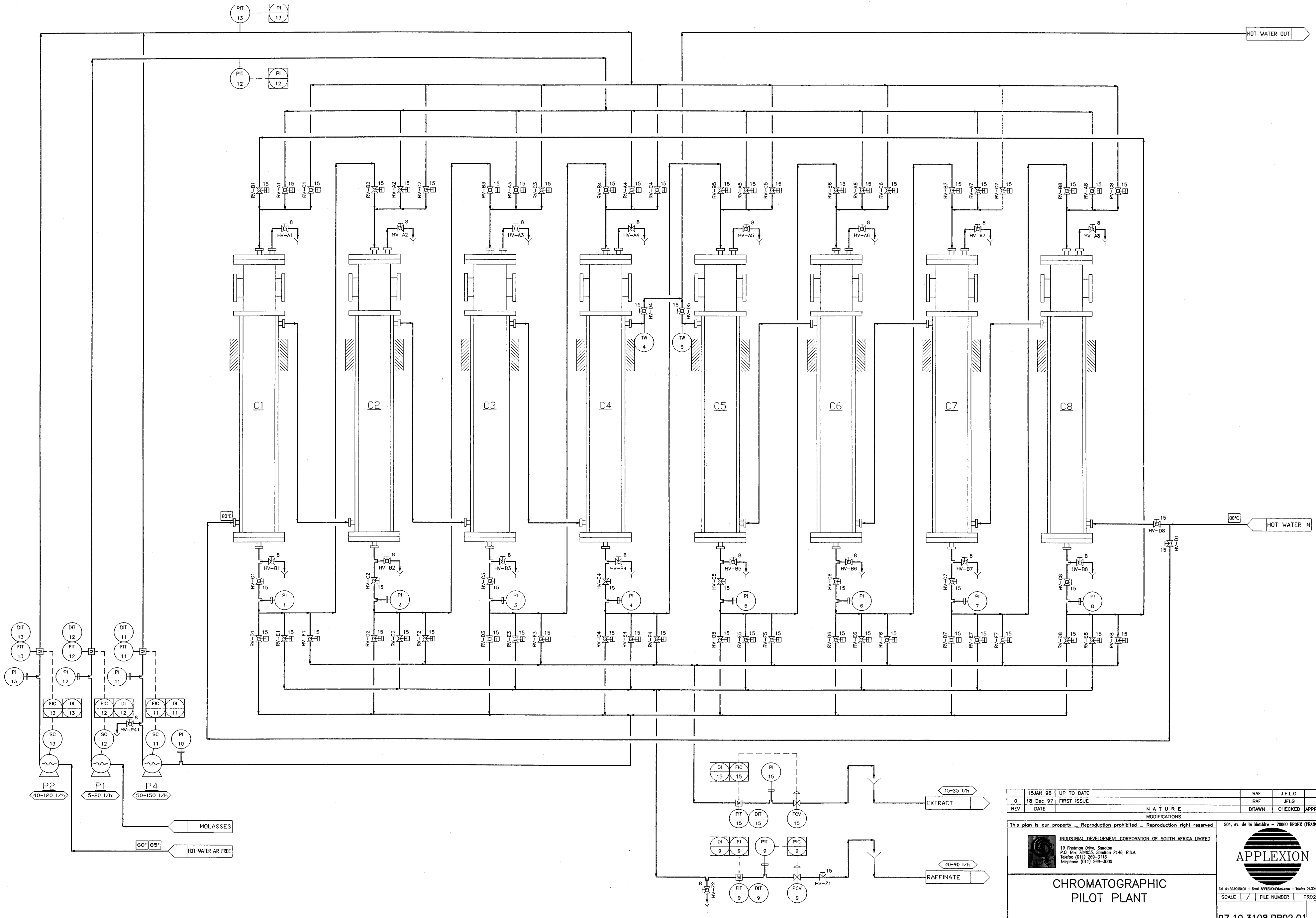
Table D.4: Results of the trials with Phosphatation

Trial #	Brix Feed	ICUMSA Turbidity Feed (Turb/Brix)	NTU/ Brix Feed	Brix Mother Liquor	ICUMSA Turbidity Mother Liquor (Turb/Brix)	NTU/ Brix Mother Liquor	% NTU/Brix Change
99-04b	19.48	48275 (2478)	169.4	17.0	34712 (2042)	129.4	24%
99-05b	19.6	86255 (4401)	212.0	19.0	20874 (1099)	45.8	78%
99-11a	13.8	23398 (1696)	173.9	13.7	42475 (3100)	123.6	29%
99-11b	17.0	91050 (5356)	194.1	19.5	24102 (1236)	71.9	63%
99-11c	14.6	58634 (4016)	219.1	14.3	16460 (1151)	99.3	55%
99-11d	19.0	72019 (3790)	284.2	20.9		98.7	65%
99-13	19.1	78454 (4108)	257.0	17.6	35474 (2016)	142.0	45%
99-14	14.0	18643 (1332)	242.9	14.2	30202 (2127)	91.9	62%
99-15	26.7	67512 (2529)	289.9	24.55	121572 (4952)	110.6	62%
99-16	21.2	123017 (5803)	409.9	14.1	19848 (1408)	58.3	86%
99-17	18.6	77083 (4144)	359.4	16.6	52979 (3192)	250.2	30%
99-18a	19.4	19824 (1022)	227.0	17.0	17222 (1013)	47.61	79%
99-18b	19.4	19824 (1022)	227.0	17.2	11964 (696)	***20.0***	91%
99-19	17.8		296.9	17.1		***29.31***	90%

Except for the last two trials the overall performance of the centrifugal process did not yield a product suitable for further processing.

APPENDIX E

PID OF THE APPLEXION SMB PILOT PLANT



1	15JAN 98	UP TO DATE	RAF	J.F.L.G.	
0	18 Dec 97	FIRST ISSUE	RAF	JFLG	
REV	DATE	NATURE	DRAWN	CHECKED	APPROVED

MODIFICATIONS

INDUSTRIAL DEVELOPMENT CORPORATION OF SOUTH AFRICA LIMITED
19 Fredman Drive, Sandton
P.O. Box 784055, Sandton 2146, R.S.A.
Telefax (011) 269-3116
Telephone (011) 269-3000

264, av. de la Mauldre - 78680 EPONE (FRANCE)
APPLEXION
Tel. 01.30.90.50.00 - Email APPLEXIONF@aol.com - Telefax 01.30.91.05.31

CHROMATOGRAPHIC
PILOT PLANT

PID

SCALE / FILE NUMBER PR020101

97.10.3108.PR02.01 1

Appendix F

Financial Statements prepared for the Invert Sugar Plant

KEY FIGURES OF INTEREST

Financials

Year	**2004**	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**
R'000	FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10
Turnover	59,400	72,633	87,991	110,214	120,023	130,826	142,600	155,434	169,423	184,671	201,291
Nett Assets	96,228	89,337	82,854	77,686	70,139	62,783	55,613	48,645	41,900	35,395	38,581
IBIT	25,266	33,952	44,018	58,748	64,816	71,497	78,781	86,719	95,372	104,804	124,512
%IBIT on Turnover	43%	47%	50%	53%	54%	55%	55%	56%	56%	57%	62%
%IBIT on Nett Assets	26%	38%	53%	76%	92%	114%	142%	178%	228%	296%	323%
PAT	12,418	21,769	30,347	42,820	48,687	49,379	56,197	63,528	71,418	79,916	97,384
%PAT on Nett Assets	13%	24%	37%	55%	69%	79%	101%	131%	170%	226%	252%

Break - even

Invert Sugars ('000t DS)	10.263	9.881	9.483	9.101	8.686	8.300	7.947	7.622	7.325	7.052	4.018
Selling price/t	1,264	1,248	1,256	1,262	1,354	1,455	1,565	1,685	1,816	1,958	1,883

SENSITIVITY ANALYSIS

		PROJECT IRR
		Normal
Base Case		42
Selling price	-9%	37
	9%	47
Molasses Price	21%	40
Capex	20%	35
	-10%	46

Year	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
R'000	FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Cash Flow												
Generated from operation	0	34,693	43,379	53,445	68,175	74,242	80,924	88,207	96,146	104,799	114,231	124,512
IBIT	0	25,266	33,952	44,018	58,748	64,816	71,497	78,781	86,719	95,372	104,804	124,512
Add Depreciation	0	9,427	9,427	9,427	9,427	9,427	9,427	9,427	9,427	9,427	9,427	0
Taxation paid	0	0	0	0	29,848	16,826	25,203	28,125	31,266	34,648	38,290	41,736
Interest paid	0	11,566	6,894	4,705	1,617	-698	-3,084	-5,541	-8,075	-10,693	-13,402	-14,608
Invested in operation	94,270	11,385	2,536	2,944	4,259	1,880	2,070	2,257	2,460	2,681	2,923	3,186
In Fixed Assets	94,270	0	0	0	0	0	0	0	0	0	0	0
In Current Assets	0	11,385	2,536	2,944	4,259	1,880	2,070	2,257	2,460	2,681	2,923	3,186
Dividends paid	0	0	0	0	0	0	0	0	0	0	0	0
Nett	-94,270	11,742	33,949	45,796	32,450	56,234	56,735	63,367	70,495	78,163	86,421	94,199
Funded by:												
Equity loan	0	0	0	0	0	0	0	0	0	0	0	0
Short term loan	-94,270	11,742	33,949	45,796	32,450	56,234	56,735	63,367	70,495	78,163	86,421	94,199
Total funding	-94,270	11,742	33,949	45,796	32,450	56,234	56,735	63,367	70,495	78,163	86,421	94,199
	0	0	0	0	0	0	0	0	0	0	0	0
Internal Rate of Return												
Investment :												
Invested in Fixed Assets	-94,270											
Invested in Working Capital	0	-11,385	-2,536	-2,944	-4,259	-1,880	-2,070	-2,257	-2,460	-2,681	-2,923	-3,186
Cash Generation												
Cash IBIT	0	34,693	43,379	53,445	68,175	74,242	80,924	88,207	96,146	104,799	114,231	124,512
Tax	0	0	0	0	-29,848	-16,826	-25,203	-28,125	-31,266	-34,648	-38,290	-41,736
Cash available to Investors	-94,270	23,308	40,843	50,502	34,067	55,537	53,651	57,826	62,420	67,470	73,019	79,590
IRR on investment	**42%**											

Year	**2004**	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
R'000	FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Income Statement												
Invert Sugars ('000t DS)	0.000	27.000	31.000	35.000	40.781	40.781	40.781	40.781	40.781	40.781	40.781	40.781
Sales	0	59,400	72,633	87,991	110,214	120,023	130,826	142,600	155,434	169,423	184,671	201,291
Process materials	0	18,639	22,791	27,611	34,584	37,662	41,052	44,746	48,774	53,163	57,948	63,163
Packaging materials	0	3	3	4	5	5	6	6	7	7	8	8
Margin	0	40,758	49,838	60,376	75,626	82,356	89,768	97,847	106,654	116,253	126,715	138,120
Employment cost		2,102	2,239	2,402	2,582	2,812	3,065	3,341	3,642	3,969	4,327	4,716
Maintenance		3,963	4,221	4,529	4,868	5,302	5,779	6,299	6,866	7,484	8,157	8,892
Depreciation	0	9,427	9,427	9,427	9,427	9,427	9,427	9,427	9,427	9,427	9,427	0
IBIT	0	25,266	33,952	44,018	58,748	64,816	71,497	78,781	86,719	95,372	104,804	124,512
Interest		11,566	6,894	4,705	1,617	-698	-3,084	-5,541	-8,075	-10,693	-13,402	-14,608
PBT	0	13,700	27,058	39,313	57,131	65,513	74,581	84,322	94,794	106,065	118,206	139,120
Tax	0	1,282	5,289	8,966	14,311	16,826	25,203	28,125	31,266	34,648	38,290	41,736
PAT	0	12,418	21,769	30,347	42,820	48,687	49,379	56,197	63,528	71,418	79,916	97,384
Dividends												
Retained	0	12,418	21,769	30,347	42,820	48,687	49,379	56,197	63,528	71,418	79,916	97,384

Year	**2004**	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
R'000	FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Balance sheet												
Inventory	0	5,107	6,245	7,565	9,476	10,319	11,248	12,260	13,364	14,566	15,877	17,306
Raw materials	0	2,043	2,498	3,026	3,790	4,127	4,499	4,904	5,345	5,826	6,350	6,922
Finished Stock	0	3,064	3,747	4,539	5,686	6,192	6,749	7,357	8,019	8,740	9,527	10,384
Debtors	0	7,323	8,955	10,848	13,588	14,797	16,129	17,581	19,163	20,888	22,768	24,817
Creditors	0	1,045	1,278	1,548	1,940	2,112	2,302	2,509	2,735	2,982	3,250	3,542
Nett Current Assets	0	11,385	13,921	16,865	21,124	23,004	25,075	27,332	29,791	32,473	35,395	38,581
Fixed Assets	94,270	84,843	75,416	65,989	56,562	47,135	37,708	28,281	18,854	9,427	0	0
Cost value	94,270	94,270	94,270	94,270	94,270	94,270	94,270	94,270	94,270	94,270	94,270	94,270
Accumulated Depreciation	0	9,427	18,854	28,281	37,708	47,135	56,562	65,989	75,416	84,843	94,270	94,270
Capital Employed	94,270	96,228	89,337	82,854	77,686	70,139	62,783	55,613	48,645	41,900	35,395	38,581
Equity	0	12,418	34,187	64,534	107,354	156,041	205,420	261,617	325,145	396,563	476,479	573,863
Equity loan	0	0	0	0	0	0	0	0	0	0	0	0
Retained Earnings	0	12,418	34,187	64,534	107,354	156,041	205,420	261,617	325,145	396,563	476,479	573,863
Loans	94,270	82,528	48,579	2,783	-29,668	-85,902	-142,637	-206,005	-276,500	-354,663	-441,084	-535,283
Deferred Tax	0	1,282	6,571	15,537	0	0	0	0	0	0	0	0

Process Cost

			2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Element	**Unit**	**R/Unit**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**	**R / tDS**
Molasse														
	ton	165.00	204.6	215.035	229.012	245.73	264.159	287.67	313.56	341.78	372.541	406.069	442.615	482.451
Molasse pre - treatment & Inversion			**84.83**	**89.16**	**94.96**	**101.89**	**109.53**	**119.28**	**130.01**	**141.71**	**154.47**	**168.37**	**183.52**	**200.04**
H^3PO^4(100%)	kg	4.90	14.7	15.4497	16.4539	17.6551	18.9792	20.6683	22.5285	24.5561	26.7661	29.1751	31.8008	34.6629
NaOH(100%)	kg	2.94	14.7	15.4497	16.4539	17.6551	18.9792	20.6683	22.5285	24.5561	26.7661	29.1751	31.8008	34.6629
Electricity	kWh	0.07	1.659	1.74361	1.85694	1.9925	2.14194	2.33257	2.5425	2.77133	3.02075	3.29261	3.58895	3.91195
Water soft	m^3	0.55	0.4125	0.43354	0.46172	0.49542	0.53258	0.57998	0.63218	0.68907	0.75109	0.81869	0.89237	0.97268
Steam	ton	22.00	22	23.122	24.6249	26.4225	28.4042	30.9322	33.7161	36.7506	40.0581	43.6634	47.5931	51.8764
Resin Softening Cation	litre	15.50	1.24	1.30324	1.38795	1.48927	1.60097	1.74345	1.90036	2.0714	2.25782	2.46103	2.68252	2.92394
Enzyme	kg	7.00	6.79	7.13629	7.60015	8.15496	8.76658	9.54681	10.406	11.3426	12.3634	13.4761	14.6889	16.011
Flocculant	g	0.03	2.73	2.86923	3.05573	3.2788	3.52471	3.83841	4.18386	4.56041	4.97085	5.41822	5.90587	6.43739
Filter aid	kg	3.68	19.2464	20.228	21.5428	23.1154	24.8491	27.0606	29.4961	32.1507	35.0443	38.1983	41.6361	45.3834
Electricity	kWh	0.07	0.28	0.29428	0.31341	0.33629	0.36151	0.39368	0.42911	0.46773	0.50983	0.55572	0.60573	0.66025
Sludge diposal	ton	2.50	1.07592	1.1308	1.2043	1.29221	1.38913	1.51276	1.64891	1.79731	1.95907	2.13538	2.32757	2.53705
Chromatographic separation			**10.13**	**10.65**	**11.34**	**12.16**	**13.08**	**14.24**	**15.52**	**16.92**	**18.44**	**20.10**	**21.91**	**23.88**
Water elution	m^3	0.55	1.76	1.84976	1.96999	2.1138	2.27234	2.47458	2.69729	2.94005	3.20465	3.49307	3.80744	4.15011
Electricity	kWh	0.07	0.98	1.02998	1.09693	1.177	1.26528	1.37789	1.5019	1.63707	1.78441	1.945	2.12005	2.31086
Steam	ton	22.00	0.88	0.92488	0.985	1.0569	1.13617	1.23729	1.34864	1.47002	1.60232	1.74653	1.90372	2.07506
Resin Exclusion	litre	22.00	6.16	6.47416	6.89498	7.39831	7.95319	8.66102	9.44051	10.2902	11.2163	12.2257	13.3261	14.5254
Water Backwash	m^3	0.55	0.0055	0.00578	0.00616	0.00661	0.0071	0.00773	0.00843	0.00919	0.01001	0.01092	0.0119	0.01297
NaOH(100%)	kg	2.94	0.147	0.1545	0.16454	0.17655	0.18979	0.20668	0.22528	0.24556	0.26766	0.29175	0.31801	0.34663
HCl(100%)	kg	3.92	0.196	0.206	0.21939	0.2354	0.25306	0.27558	0.30038	0.32741	0.35688	0.389	0.42401	0.46217

Process Cost			**2004**	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Demineralisation			**69.20**	**72.73**	**77.45**	**83.11**	**89.34**	**97.29**	**106.05**	**115.59**	**125.99**	**137.33**	**149.69**	**163.17**
HCl(100%)	kg	3.92	36.848	38.7272	41.2445	44.2554	47.5745	51.8087	56.4714	61.5539	67.0937	73.1321	79.714	86.8883
NaOH(100%)	kg	2.94	29.106	30.5904	32.5788	34.957	37.5788	40.9233	44.6064	48.621	52.9969	57.7666	62.9656	68.6325
Water soft	m^3	0.55	0.935	0.98269	1.04656	1.12296	1.20718	1.31462	1.43294	1.5619	1.70247	1.85569	2.0227	2.20475
Resin Softening Anion	litre	20.00	1.3	1.3663	1.45511	1.56133	1.67843	1.82781	1.99232	2.17162	2.36707	2.58011	2.81232	3.06543
Resin Softening Cation	litre	15.50	1.0075	1.05888	1.12771	1.21003	1.30079	1.41656	1.54404	1.68301	1.83448	1.99958	2.17955	2.3757
Sugar concentration			**1.28**	**1.35**	**1.43**	**1.54**	**1.66**	**1.80**	**1.96**	**2.14**	**2.33**	**2.54**	**2.77**	**3.02**
Steam	ton	22.00	1.1	1.1561	1.23125	1.32113	1.42021	1.54661	1.68581	1.83753	2.00291	2.18317	2.37965	2.59382
Electricity	kWh	0.07	0.182	0.19128	0.20372	0.21859	0.23498	0.25589	0.27892	0.30403	0.33139	0.36121	0.39372	0.42916
Raffinate concentration			**5.30**	**5.57**	**5.93**	**6.36**	**6.84**	**7.45**	**8.12**	**8.85**	**9.64**	**10.51**	**11.46**	**12.49**
Steam	ton	22.00	4.84	5.08684	5.41748	5.81296	6.24893	6.80509	7.41755	8.08513	8.81279	9.60594	10.4705	11.4128
Electricity	kWh	0.07	0.4557	0.47894	0.51007	0.54731	0.58836	0.64072	0.69838	0.76124	0.82975	0.90443	0.98583	1.07455
Total Process materials			**375.34**	**394.48**	**420.12**	**450.79**	**484.60**	**527.73**	**575.22**	**626.99**	**683.42**	**744.93**	**811.97**	**885.05**
Packaging materials														
Packaging - Drums	R/a		0	2,838	3,022	4,324	4,648	5,062	5,517	6,014	6,555	7,145	7,788	8,489
Invert produced	tDS		0	27,000	31,000	35,000	40,781	40,781	40,781	40,781	40,781	40,781	40,781	40,781
	l/a		0	41,538	47,692	53,846	62,740	62,740	62,740	62,740	62,740	62,740	62,740	62,740
Invert Sales	l/m		0	3,462	3,974	4,487	5,228	5,228	5,228	5,228	5,228	5,228	5,228	5,228
Drum capacity	l		1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000
Turn around	weeks		6	6	6	6	6	6	6	6	6	6	6	6
Drums required			0	6	6	8	8	8	8	8	8	8	8	8

Sensitivity Analyses

IRR	**42%**											
	2004	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
	FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
CPI												
Input		5.10%	6.50%	7.30%	7.50%	8.90%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%
Base		5.10%	6.50%	7.30%	7.50%	8.90%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%
Assume		9.00%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%	9.00%
Increase		3.90%	2.50%	1.70%	1.50%	0.10%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
Change in IRR	-2%											

Invert sugar Price increase												
Input		0.0%	6.5%	7.3%	7.5%	8.9%	9.0%	9.0%	9.0%	9.0%	9.0%	9.0%
Base		0.0%	6.5%	7.3%	7.5%	8.9%	9.0%	9.0%	9.0%	9.0%	9.0%	9.0%
Assume		0.0%	4.5%	5.3%	5.5%	6.9%	7.0%	7.0%	7.0%	7.0%	7.0%	7.0%
Increase		0.00%	-2.00%	-2.00%	-2.00%	-2.00%	-2.00%	-2.00%	-2.00%	-2.00%	-2.00%	-2.00%
Change in IRR	-3%											

Selling price (R/tDS)		**Molasse** Purchase Price (R/t)	**Capital investment** R/US$
Input	2200	165	6.30
Base	2200	165	6.30
Assume	2000	200	7.00
Increase	-9%	21%	11%
Change in IRR	-5%	-2%	-4%

SENSITIVITY ANALYSIS

Net Present Value		**Year**	**2004**	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
R'000			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Base Case		**30%**	**Discounting Rate**											
Investment :														
Invested in Fixed Assets			**-94,270**											
Invested in Working Capital			0	-11,385	-2,536	-2,944	-4,259	-1,880	-2,070	-2,257	-2,460	-2,681	-2,923	-3,186
Cash Generation														
Cash IBIT			0	34,693	43,379	53,445	68,175	74,242	80,924	88,207	96,146	104,799	114,231	124,512
Tax			0	0	0	0	-29,848	-16,826	-25,203	-28,125	-31,266	-34,648	-38,290	-41,736
Cash available to Investors			-94,270	23,308	40,843	50,502	34,067	55,537	53,651	57,826	62,420	67,470	73,019	79,590
		Year	0	1	2	3	4	5	6	7	8	9	10	11
NPV	Rate	30%												
	Individual NPV's		-94,270	17,929	24,167	22,987	11,928	14,958	11,115	9,216	7,652	6,362	5,297	4,441
	NPV =	*41,782*	-94,270	-76,341	-52,173	-29,187	-17,259	-2,301	8,814	18,030	25,682	32,044	37,341	41,782
	IRR=	*42%*	**NRR =**	*4.0*	**NPT =**	5.207	years							
Capex +20%			2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
Investment :			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Invested in Fixed Assets			**-113124**											
Invested in Working Capital			0	-11,385	-2,536	-2,944	-4,259	-1,880	-2,070	-2,257	-2,460	-2,681	-2,923	-3,186
Cash Generation														
Cash IBIT			0	34,693	43,379	53,445	68,175	74,242	80,924	88,207	96,146	104,799	114,231	124,512
Tax			0	0	0	0	-29,848	-16,826	-25,203	-28,125	-31,266	-34,648	-38,290	-41,736
Cash available to Investors			-113,124	23,308	40,843	50,502	34,067	55,537	53,651	57,826	62,420	67,470	73,019	79,590

SENSITIVITY ANALYSIS

		Year	0	1	2	3	4	5	6	7	8	9	10	11
NPV	Rate	30%												
	Individual NPV's		-113,124	17,929	24,167	22,987	11,928	14,958	11,115	9,216	7,652	6,362	5,297	4,441
	NPV =	*22,928*	-113,124	-95,194	-71,027	-48,040	-36,113	-21,155	-10,040	-824	6,828	13,190	18,487	22,928
	IRR=	*36%*	**NRR =** *1.8*		**NPT =**	7.108	years							

Capex -10%			**2004**	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
Investment :			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Invested in Fixed Assets			**-84843**											
Invested in Working Capital			0	-11,385	-2,536	-2,944	-4,259	-1,880	-2,070	-2,257	-2,460	-2,681	-2,923	-3,186
Cash Generation														
Cash IBIT			0	34,693	43,379	53,445	68,175	74,242	80,924	88,207	96,146	104,799	114,231	124,512
Tax			0	0	0	0	-29,848	-16,826	-25,203	-28,125	-31,266	-34,648	-38,290	-41,736
Cash available to Investors			-84,843	23,308	40,843	50,502	34,067	55,537	53,651	57,826	62,420	67,470	73,019	79,590
		Year	0	1	2	3	4	5	6	7	8	9	10	11
NPV	Rate	30%												
	Individual NPV's		-84,843	17,929	24,167	22,987	11,928	14,958	11,115	9,216	7,652	6,362	5,297	4,441
	NPV =	*51,209*	-84,843	-66,914	-42,746	-19,760	-7,832	7,126	18,241	27,457	35,109	41,471	46,768	51,209
	IRR=	*46%*	**NRR =** *5.5*		**NPT =**	4.524	years							

Molasses +21%	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
Investment :	FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Invested in Fixed Assets	**-94270**											
Invested in Working Capital	0	-11,970	-2,666	-3,095	-4,478	-1,977	-2,177	-2,373	-2,586	-2,819	-3,073	-3,349

SENSITIVITY ANALYSIS

Cash Generation														
Cash IBIT			0	32,559	40,770	50,285	64,216	69,931	76,225	83,085	90,563	98,714	107,598	117,282
Tax			0	0	0	0	-25,632	-15,252	-23,487	-26,255	-29,229	-32,427	-35,869	-39,097
Cash available to Investors			-94,270	20,589	38,104	47,190	34,106	52,702	50,561	54,457	58,748	63,468	68,656	74,836
		Year	0	1	2	3	4	5	6	7	8	9	10	11
NPV	Rate	30%												
	Individual NPV's		-94,270	15,838	22,547	21,479	11,941	14,194	10,475	8,679	7,202	5,985	4,980	4,176
	NPV =	*33,226*	-94,270	-78,432	-55,885	-34,406	-22,464	-8,270	2,205	10,883	18,085	24,070	29,050	33,226
	IRR=	*40%*	**NRR =**	*3.2*	**NPT =**	5.790	years							

Selling Price -9%			2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
Investment :			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Invested in Fixed Assets			**-94270**											
Invested in Working Capital			0	-10,726	-2,389	-2,773	-4,013	-1,771	-1,951	-2,126	-2,317	-2,526	-2,753	-3,001
Cash Generation														
Cash IBIT			0	29,347	36,842	45,526	58,256	63,440	69,150	75,374	82,157	89,551	97,611	106,396
Tax			0	0	0	0	-19,799	-13,102	-21,144	-23,701	-26,444	-29,391	-32,561	-35,491
Cash available to Investors			-94,270	18,621	34,453	42,753	34,444	48,567	46,055	49,547	53,396	57,634	62,297	67,904
		Year	0	1	2	3	4	5	6	7	8	9	10	11
NPV	Rate	30%												
	Individual NPV's		-94,270	14,324	20,386	19,460	12,060	13,081	9,542	7,896	6,546	5,435	4,519	3,789
	NPV =	*22,767*	-94,270	-79,946	-59,559	-40,100	-28,040	-14,959	-5,418	2,478	9,024	14,459	18,978	22,767
	IRR=	*37%*	**NRR =**	*2.2*	**NPT =**	6.686	years							

SENSITIVITY ANALYSIS

Selling Price +9%			2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
Investment :			FY0	FY1	FY2	FY3	FY4	FY5	FY6	FY7	FY8	FY9	FY10	FY11
Invested in Fixed Assets			**-94270**											
Invested in Working Capital			0	-12,044	-2,683	-3,114	-4,506	-1,989	-2,190	-2,387	-2,602	-2,836	-3,092	-3,370
Cash Generation														
Cash IBIT			0	40,039	49,916	61,365	78,094	85,045	92,699	101,041	110,135	120,047	130,852	142,628
Tax			0	0	0	0	-39,898	-20,549	-29,261	-32,549	-36,089	-39,904	-44,019	-47,981
Cash available to Investors			-94,270	27,995	47,233	58,251	33,690	62,507	61,248	66,105	71,444	77,307	83,741	91,277
		Year	0	1	2	3	4	5	6	7	8	9	10	11
NPV	Rate	30%												
	Individual NPV's		-94,270	21,535	27,949	26,514	11,796	16,835	12,689	10,535	8,758	7,290	6,074	5,093
	NPV =	*60,798*	-94,270	-72,735	-44,787	-18,273	-6,477	10,358	23,047	33,582	42,340	49,630	55,705	60,798
	IRR=	*47%*	**NRR =**	*5.9*	**NPT =**	4.385years								

Estimated capital cost

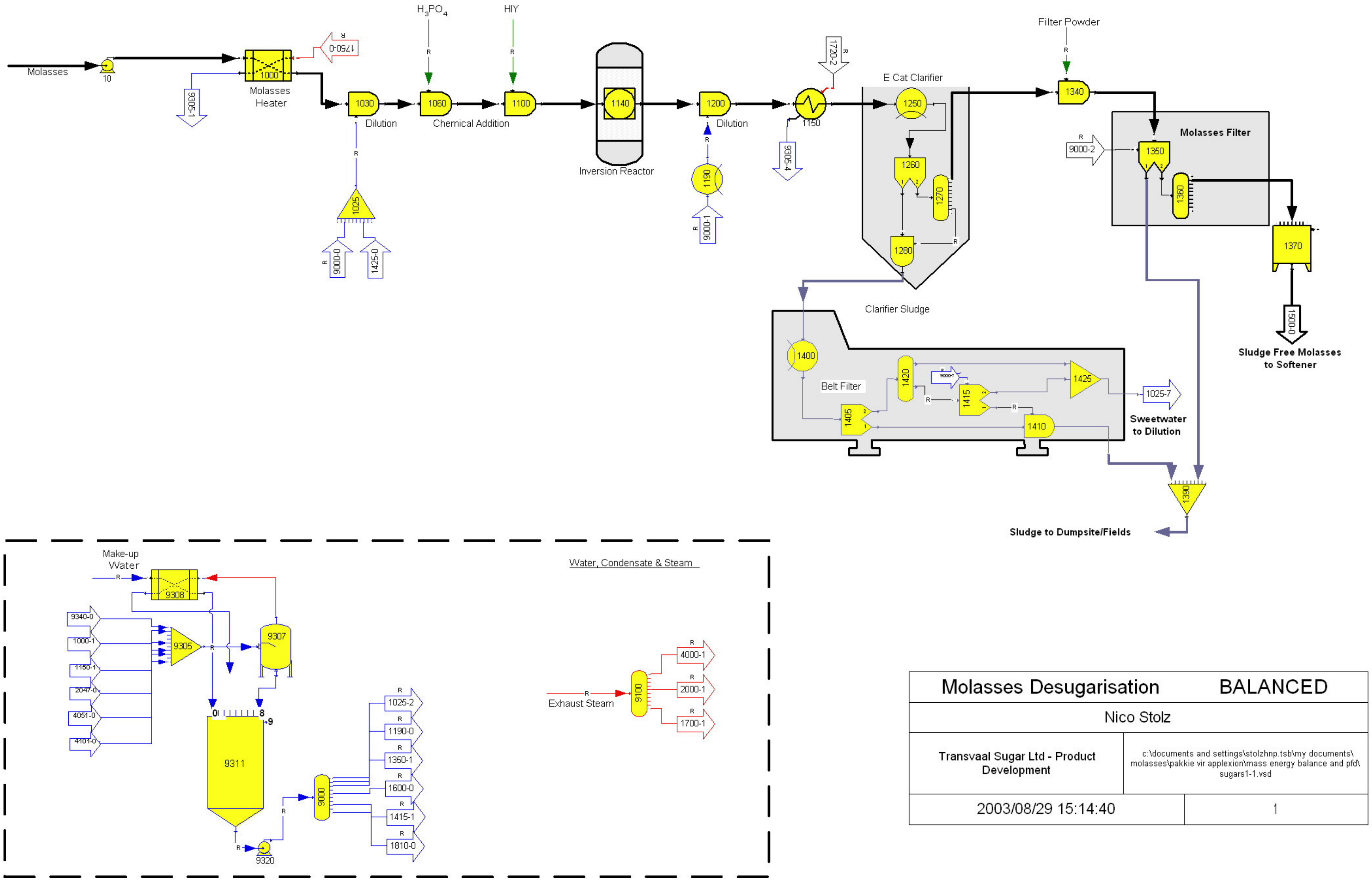
	Molasses pre-treatment		US$'000	
1	Dilution, heating		45	
2	Centrifugation	machines	1,000	
3	Softeners & resins	2 columns	337	
	Molasses Inversion			
4	Inversion tanks & stirrers	8 tanks	293	
5	Molasses conditioning		35	
	Chromatographic separator			
6	Train A with 4 columns & 275 m3 resins		3,450	
7	Molasses % elution water degassing		inc	
8	Resins service tank & pumps		inc	
	Glucose-fructose demine-decolor			
9	Ix plant 6 columns & 56 m3 resins		871	
10	Regeneration equipment		inc	
	Glucose-fructose evaporator			
11	3 effects & thermp plate evaporator		378	
	Raffinate evaporator			
12	5 effects & thermo tubular evaporator		970	
	Sub -Total Equipment		7,379	
	Additional budget estimate			
13	Instrumentation & valves		664	9.0%
14	Piping		738	10.0%
15	Civils		517	7.0%
16	Electrical plant		812	11.0%
17	Building		369	5.0%
18	Insulation & painting		295	4.0%
19	Insurance		37	0.5%
20	Contingency		1,476	20.0%
21	Inspection		30	
22	Intellectual property		800	
23	Project Engineering		1,847	15.0%
	Total estimated capital cost		14,963	USD
			94,270	R'000
	Depreciation rate		10.0%	

Appendix G

SUGARS Simulation of the proposed Invert Sugar Plant

TSB Molasses Desugarisation Process

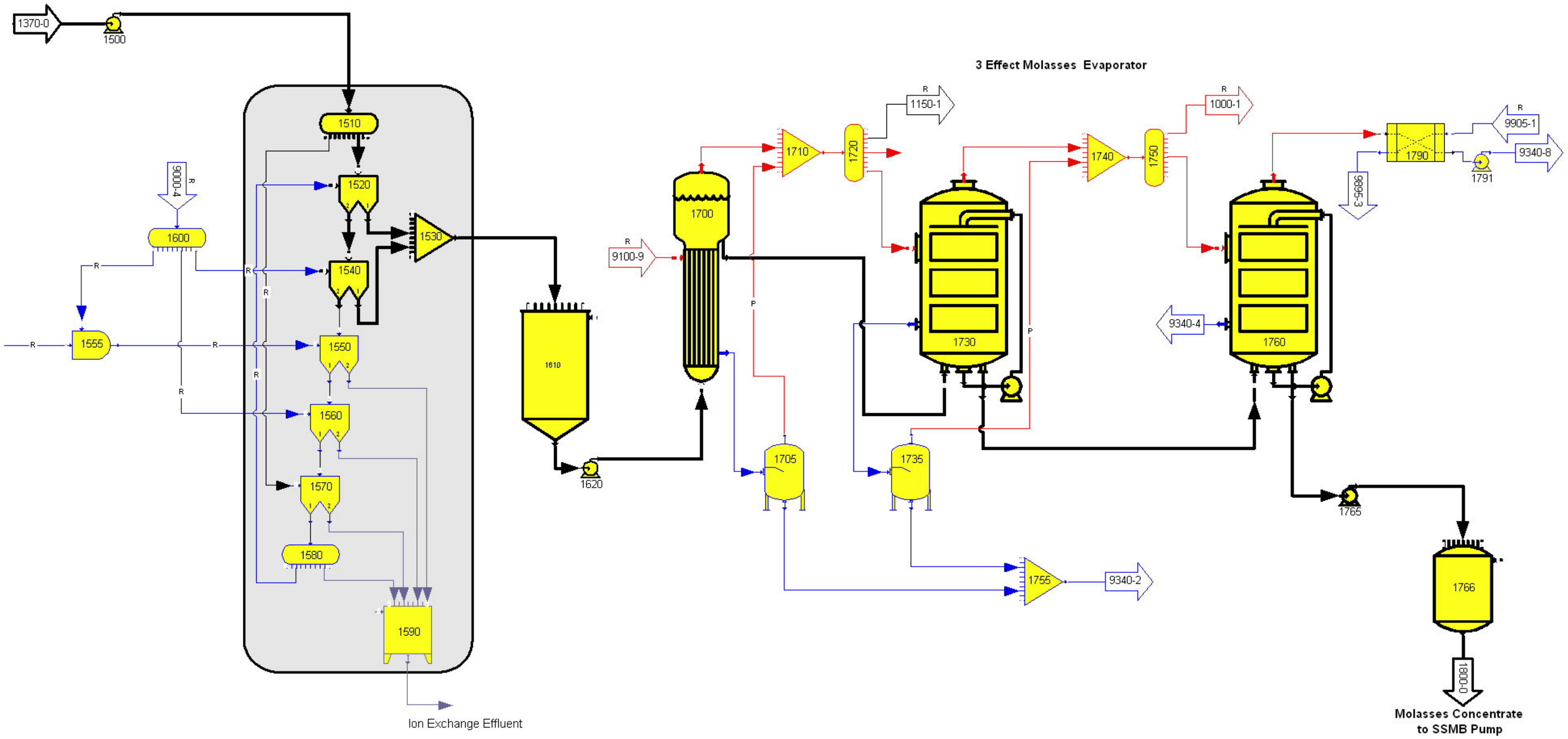
Part 1 - Molasses Clarification

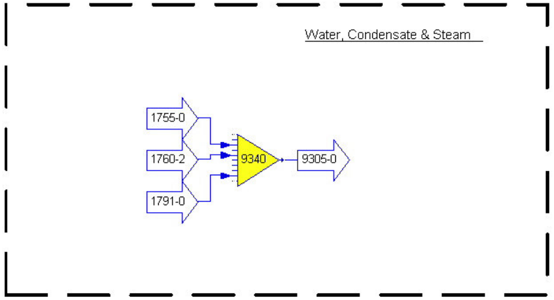


Molasses Desugarisation	BALANCED
Nico Stolz	
Transvaal Sugar Ltd - Product Development	c:\documents and settings\stolzhnp.tsb\my documents\molasses\pakkie vir applexion\mass energy balance and pfd\sugars1-1.vsd
2003/08/29 15:14:40	1

TSB Molasses Desugarisation Process

Part 2 - Molasses Softening & Evaporation

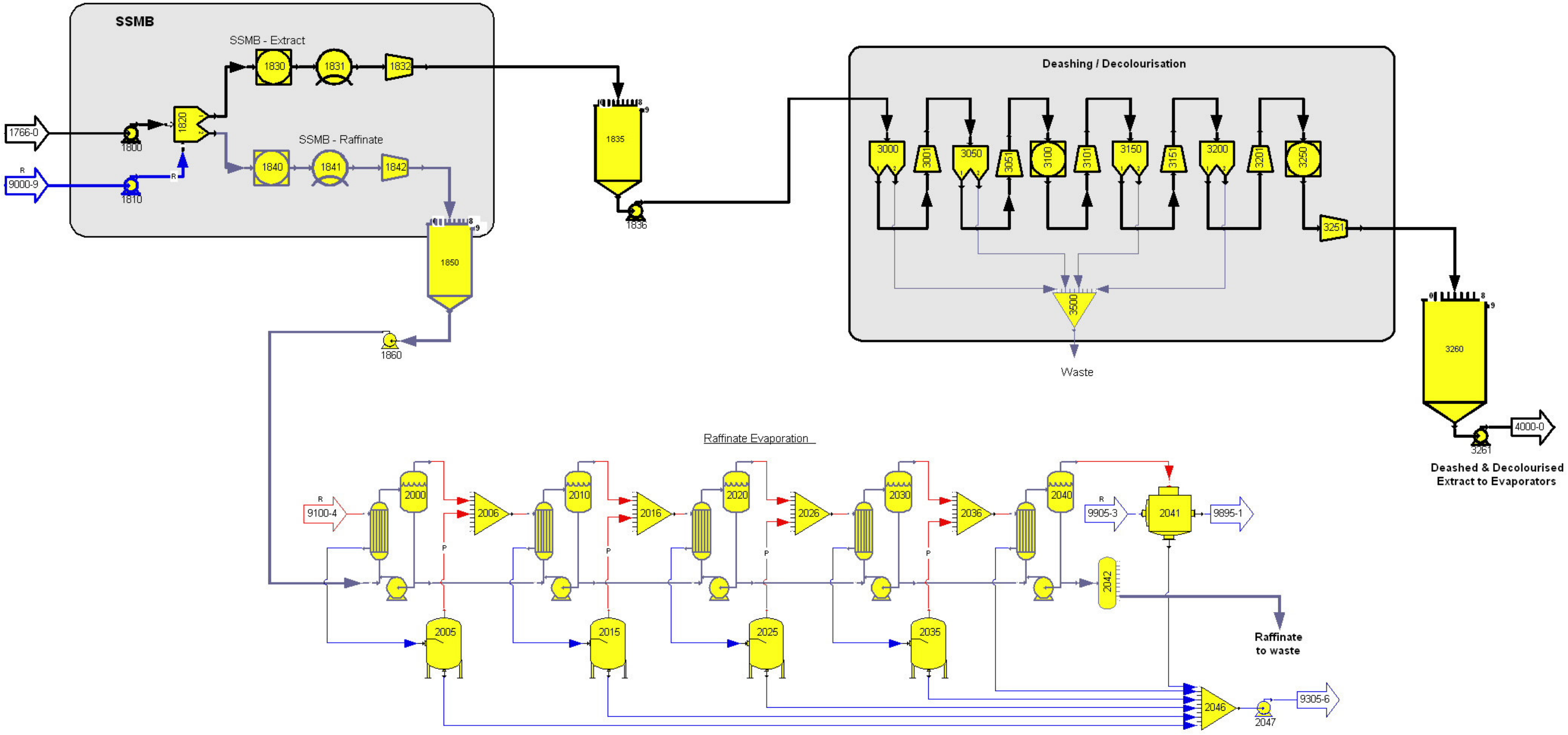




Molasses Desugarisation	BALANCED
Nico Stolz	
Transvaal Sugar Ltd - Product Development	c:\documents and settings\stolzhnp.tsb\my documents\ molasses\pakkie vir applexion\mass energy balance and pfd\ sugars1-1.vsd
2003/08/29 15:06:34	2

TSB Molasses Desugarisation Process

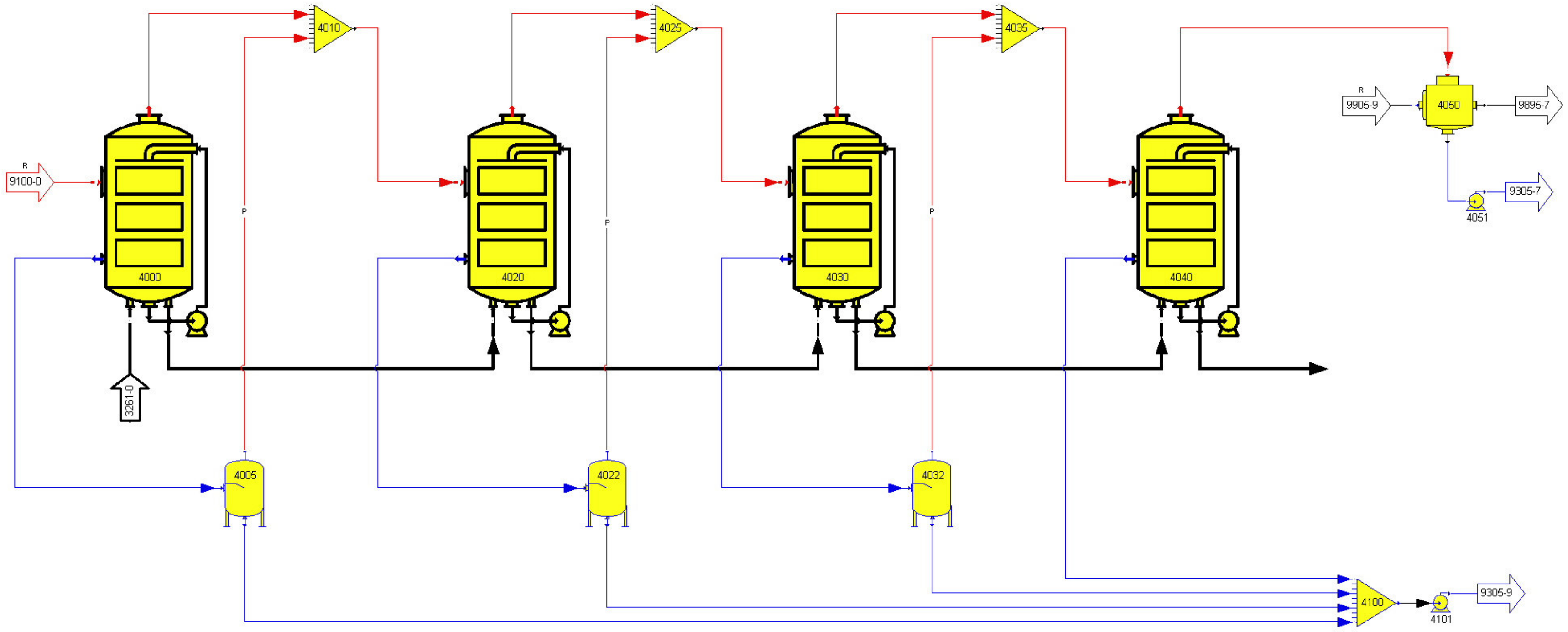
Part 3 - Molasses SSMB, Deash, Decolour & Raffinate Evaporation



Molasses Desugarisation	BALANCED
Nico Stolz	
Transvaal Sugar Ltd - Product Development	c:\documents and settings\stolzhnp.tsb\my documents\molasses\pakkie vir applexion\mass energy balance and pfd\sugars1-1.vsd
2003/08/29 15:06:34	3

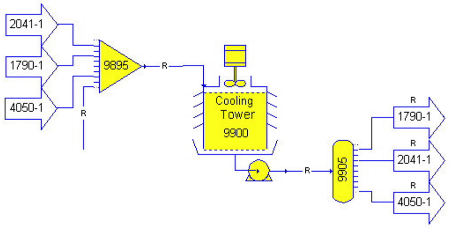
TSB Molasses Desugarisation Process

Part 4 - Extract Evaporation



Molasses Desugarisation	BALANCED
Nico Stolz	
Transvaal Sugar Ltd - Product Development	c:\documents and settings\stolzhnp.tsb\my documents\molasses\pakkie vir applexion\mass energy balance and pfd\sugars1-1.vsd
2003/08/29 15:06:34	4

TSB Molasses Desugarisation Process
Part 5 - Komati Mill Utilities



Cooling Towers - CCW Supply and Return

Molasses Desugarisation	BALANCED
Nico Stolz	
Transvaal Sugar Ltd - Product Development	c:\documents and settings\stolzhnp.tsb\my documents\molasses\pakkie vir applexion\mass energy balance and pfd\sugars1-1.vsd
2003/08/29 15:14:40	5