A QUALITY CONTROL SYSTEM FOR THE MANUFACTURE OF SPRAY DRIED MILK POWDERS

A thesis presented in partial
fulfilment of the requirements for
the degree of Doctor of Philosophy
in Technology at Massey University

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February, 1981

To my wife, Jane

ABSTRACT

In the last decade the New Zealand dairy industry has greatly increased its spray drying capacity in response to the world market demand for spray dried milk products. Powder specifications are becoming increasingly complex and smaller quantities of each product are required as the number of different products grows. These factors have made it necessary to learn more about the way processing variables influence the product quality in order to improve product quality control.

A computer simulation model providing a complete description of the drier behaviour was developed from a series of experiments on a pilot scale spray drier. This took the form of regression equations relating the quality parameters of skim milk powder to the drier operating variables and the composition and physical properties of the skim milk. The model was then used in the development of a quality control system and also to simulate and evaluate a variety of commercial operating practices.

The characteristics of the spray drying process were investigated using the pilot plant evaporator and spray drier at the New Zealand Dairy Research Institute, which had been fully instrumented and interfaced to a process control computer. The drier studies confirmed the importance of low concentrate viscosity in the production of good quality milk powder. This could be achieved by keeping concentrate holding times to a minimum and by using high temperature, short time preheat treatments. The protein content of the skim milk was found to be the major determinant in the seasonal changes observed in concentrate viscosity, high protein contents giving high viscosities.

The study of the hydrodynamics of centrifugal pressure nozzle atomisers revealed that the nozzles used in milk powder drying fall into two distinct categories, each with characteristic behaviour in response to variations in fluid viscosity. The magnitude of the viscosity effect depends on the ratio of the swirl chamber and orifice diameters. The large capacity nozzles used in tall-form driers exhibit a marked decrease in pressure drop at constant flowrate as the viscosity of the concentrate fed to them is increased. This was found to play a very

important part in determining the overall behaviour of the drier. Five operating variables; the inlet air temperature and the concentrate total solids, feedrate, atomising pressure and temperature, proved to be necessary and sufficient to describe the drier performance and to predict the properties of the powder. Simulation studies of two outlet air temperature control strategies clearly demonstrated the superiority of inlet air temperature manipulation over that of concentrate feedrate, for driers employing large capacity nozzles.

The drier model was used in the selection, tuning and evaluation of a quality control system based on the SIMPLEX Evolutionary Operation scheme of Spendley et al. The process of spray drying milk powders presents several control problems. There are a number of quality parameters assessed by laboratory analysis, which means that feedback is multivariable, delayed and subject to error. Furthermore, the processing characteristics of milk change with time. A single measure of the powder quality was obtained from penalty functions based on economic considerations. After selection of the SIMPLEX step sizes with the help of the simulation model, a pilot plant trial of the scheme was conducted.

The Simplex evolutionary operation method was found to be a simple robust procedure which rapidly improved the product quality and maintained it in the face of disturbances typical of those likely to occur in commercial operation. The method provides two sets of plant conditions in advance, a feature which permits a substantial increase in the speed of attainment of optimum conditions for processes with setpoint response times similar to the time required to analyse the product quality. The Simplex method is therefore particularly suited to the manufacture of spray dried milk powders.

ACKNOWLEDGEMENTS

The author wishes to record his gratitude to all the technicians and technical officers who over the years performed several thousand often extremely tedious analyses of milk powder samples. The help furnished by the staff of the Analytical Chemistry Section of the NZDRI and the Auckland Regional Laboratory of the Dairy Division of the Ministry of Agriculture and Fisheries is also gratefully acknowledged.

Valuable advice and assistance on experimental design and statistical analysis was provided by R. H. Fletcher and I. R. Hughes, and all the members of the Milk Powders and Drying section provided advice, support and encouragement. In particular, the author wishes to thank Mr A. G. Baucke, who operated the pilot plant with great élan, for his many invaluable contributions to the experimental work. The help of Dr W. B. Sanderson, Dr D. J. Sandoz and Dr I. F. Boag as supervisors is also acknowledged with thanks.

The work described in this thesis was built on the foundation laid by the Milk Powder Control and Information Project, and thanks are due to all those who participated in that venture.

Finally, the author wishes to thank his employers, the NZDRI, who provided the research facilities, for their financial support.

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CHAPTER 1 - GENERAL INTRODUCTION

In the last decade the New Zealand dairy industry has greatly increased its spray drying capacity in response to the world market demand for spray dried milk products. Initially these products were skim milk and buttermilk powders. Wholemilk powders, formulated infant foods and stock foods and a range of caseinate, whey and lactalbumin powders have been added in more recent times. By 1979 there were 40 specifications for skim milk powders, 23 for wholemilk powders, 10 for other powders containing various levels of fat and 36 specifications for infant foods and beverages. Production of these powders totalled 263,000 tonnes during the 1978/79 dairying season (NZ Dairy Board, 1979). The increasing complexity of powder specifications and the smaller quantities of each product required as the number of different products grows, make severe demands on the quality control systems in the factories. Not only must more quality parameters be controlled, but each production run may last only a few weeks, so rapid achievement of acceptable quality is essential. This makes a detailed study of the influence of processing variables on the various quality parameters of milk powders timely.

A quality control system able to provide powder meeting specification at minimum production cost in a short time, and maintain the quality despite changes in the processing characteristics of the milk was developed using a computer simulation model of a pilot scale spray drier. The research facilities used were a legacy of a research project on the control of evaporators and spray driers initiated in 1973. The Milk Powder Control and Information Project, as it was known, involved the New Zealand Dairy Research Institute (NZDRI), the Physics and Engineering Laboratory of the DSIR, IBM (New Zealand) Ltd, Massey University and the New Zealand Dairy Board. By 1976 the pilot plant evaporator and spray drier at the NZDRI had been fully instrumented and interfaced to a process control computer which controlled the whole plant and recorded all the instrument readings.

Mathematical models of the relationships between the quality parameters of skim milk powder and the drier operating variables were obtained. Then a quality control system was developed which adjusted

these variables to ensure that the product met its specification at minimum cost. The thesis has been divided into two parts, reflecting these twin objectives.

The first objective was pursued by means of an extensive series of experiments on the computer controlled pilot scale spray drier. Response Surface Methodology was applied to obtain regression models for each of the powder properties of interest as functions of drier operating variables and the composition and physical properties of the skim milk. Additional equations linking some of the operating variables were also developed. The result was a computer simulation model providing a complete description of the drier behaviour and the means whereby a variety of commercial operating practices could be simulated and evaluated.

This model was then used in the selection, tuning and evaluation of a quality control scheme. The spray drying of milk powders is a process presenting several problems to any control system. There are a number of quality parameters, all of which must be assessed by laboratory analysis, which means that feedback is multivariable, delayed and subject to error. Furthermore, the processing characteristics of milk change with time. The SIMPLEX Evolutionary Operation scheme of Spendley et al. (1962) was chosen because of its proven ability to cope with the last three of these problems. The various aspects of the quality were reduced to a single quality descriptor by the use of penalty functions based on economic considerations. After selection of the SIMPLEX parameters with the help of an experimental program run on the simulation model, a pilot plant trial of the scheme was conducted.

PART I THE DRIER MODEL

CHAPTER 2 - INTRODUCTION

The objective of the drier studies was to develop a general description of the spray drying process relating the properties of the milk powder to the design and method of operation of a spray drier.

Spray driers may be placed into two categories according to their means of atomisation; centrifugal disk or pressure nozzle. This division of drier types may be extended by sub-dividing nozzle atomising driers into multiple nozzle and tall-form driers. The multiple nozzle driers have from 12 to 30 small capacity nozzles of the Spraying Systems SX series. Their drying chambers are either of the horizontal box configuration or conical. The horizontal driers have the air inlets and nozzles mounted in one end wall. The conical driers have multiple air inlets in a short cylindrical section above the cone. The nozzles are either fitted around the circumference or mounted centrally within the chamber. Tall-form driers, as their name suggests, are tall upright cylinders with height to diameter ratios in excess of 2.5 to 1. The drying air enters through one or more inlets in the roof and from one to four large capacity nozzles are arranged in the air inlets spraying vertically downwards. The most commonly used nozzles are Delavan SDX series, but nozzles made by Coulter, Morinaga and Spraying Systems are also used. The pilot scale drier used in this work is a tall-form, nozzle atomising type. The present study is strictly applicable only to this type of drier.

The radial spray pattern of the droplets dictates the configurations of disk atomising driers. They all have large diameters, and are cylindrical with flat or conical bottoms.

2.1 - Spray Drying in the New Zealand Dairy Industry

The Milk Powders and Drying Section of the NZDRI periodically surveys the equipment installed in New Zealand milk powder factories. The results of these surveys have been collated together with some new

information and the relevant material has been summarised to give an overall impression of the types of drier in use, and the ways in which they are operated.

The growth of spray drying in the New Zealand dairy industry is illustrated in Figure 2.1 which shows the total number of driers in each of these three categories year by year from 1940 to 1980. The three-fold increase in drier numbers between 1964 and 1968 is particularly noteworthy. The total processing capacity increased four and a half times over the same period, reflecting the larger size of the driers being installed. Disk atomising driers contributed most to this increase. The years 1972 to 1976 saw a similar rapid growth in drier numbers, with tall-form driers making up 12.3 % of the total capacity by 1976. Table 2-1 gives the inventory of spray driers at the start of the 1980/81 dairying season.

TABLE 2-1 Spray Drier Numbers and Capacities in Mid-1980

Drier Type	No.	% of Total	Capacity (tonne/h)	% of Total
Disk Atomising	35	53.8 %	73.3	58.5 %
Multiple Nozzle	22	33.9 %	31.2	24.9 %
Tall-Form	8	12.3 %	20.8	16.6 %
Total	65	100 %	125.3	100 %

There has been a steady trend towards the installation of larger driers over the 40 years of spray drying in the dairy industry. This is shown in Table 2-2 which gives the average capacity of each drier type installed in each decade from 1940 to 1980.

The way these driers are operated depends on the type of air heater, the atomisation system employed, and the provision of automatic control. As an example, the inlet air temperature is not generally used

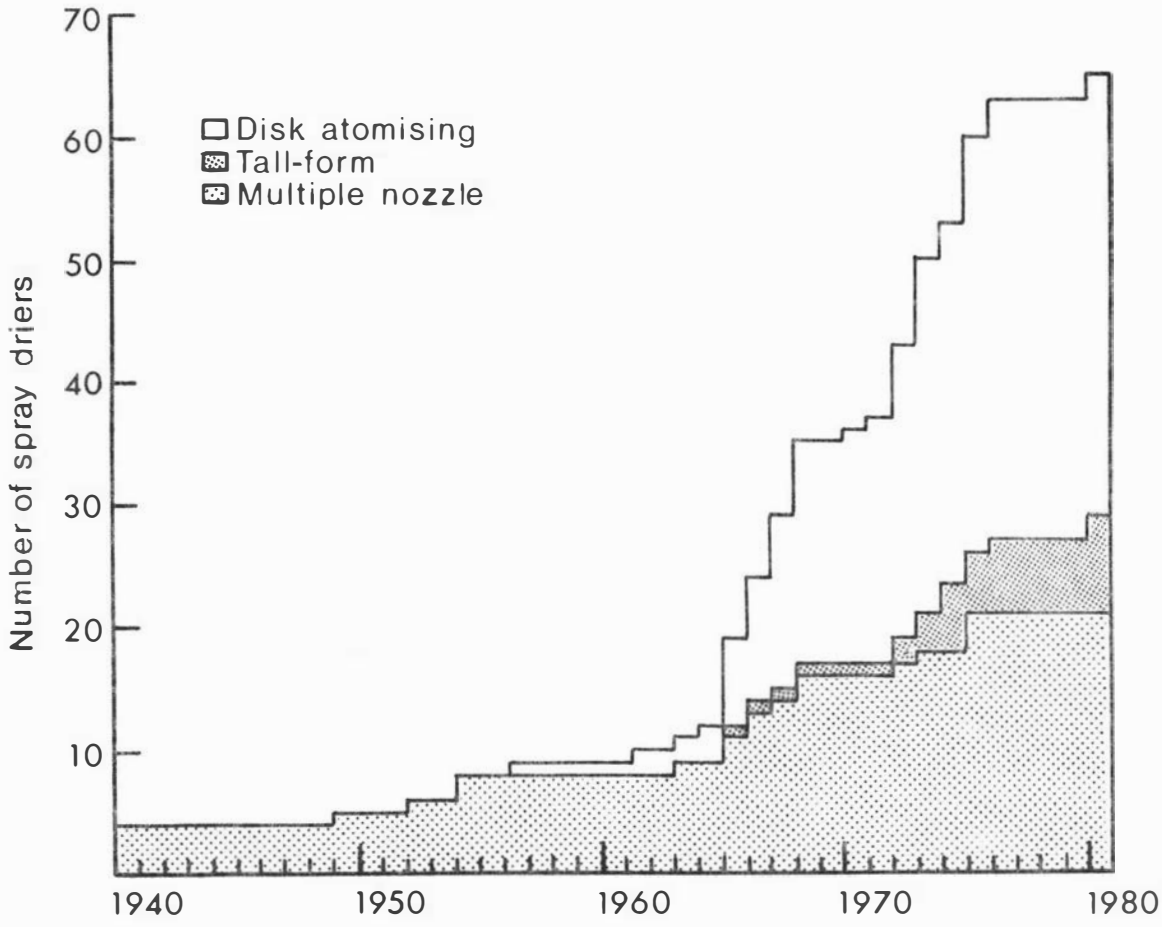


FIGURE 2.1 Numbers of Spray Driers installed in the New Zealand Dairy Industry, 1940 to 1980

TABLE 2-2 Average Spray Drier Capacities (tonne/h)

Decade	Disk Atomising	Multiple Nozzle	Tall-Form
1940-1950	-	0.90	-
1951-1960	1.02	0.83	-
1961-1970	1.83	1.25	0.95
1971-1980	2.45	2.37	2.83

to control the moisture of the powder because of the slow response times of indirect oil or gas fired air heaters, and the use of low pressure steam radiators which are normally run at their maximum attainable air temperature. These types of air heaters are the most common, as Table 2-3 shows. Automatic control systems fitted to disk atomising driers invariably control the outlet air temperature by varying the concentrate feedrate. The control systems fitted to nozzle atomising driers are set out in Table 2-4. Half of the multiple nozzle driers fitted with steam radiators have steam pressure regulators which are usually set to give the highest steam pressure possible, consistent with the fluctuations in the boiler supply pressure. Tall-form driers are capable of operating with much higher inlet air temperatures without product quality problems, and the air heaters chosen for them reflect this. Hot

TABLE 2-3 Air Heaters Fitted to Each Type of Spray Drier

Air Heater	Disk Atomising	Multiple Nozzle	Tall-Form
Indirect Oil or Gas Fired	16	1	1
Steam Radiator	17	20	2
Steam & Hot Oil Radiators	0	1	2
Hot Oil Radiator	0	0	2
Direct Gas Fired	2	0	1

TABLE 2-4 Nozzle Atomising Drier Operating Variables and their Control Systems

Operating Variables and Control Systems	Multiple Nozzle	Tall-Form
Inlet Air Temperature		
Manual, fixed temperature	11	1
Automatic, fixed temperature	11	2
Automatic, variable temperature	0	5
Drying Air Flowrate		
Manual, remote adjustment	0	1
Atomising Pressure		
Fixed speed pump, manual by-pass	22	4
Variable speed pump	0	4
Concentrate Temperature		
No provision to change temperature	5	2
Manual, fixed temperature	17	4
Automatic, fixed temperature	0	2

oil radiators with automatically controlled by-pass valves are used as the sole means of heating, or as booster heaters following a steam radiator in four tall-form driers.

These aspects of the design of spray driers place practical limitations on the way they can be operated without the installation of additional equipment. The choice of manipulated variables for drier control systems will depend on the speed of response to changes in each input variable. This will be considered further in Chapter 5, when the simulation model is assembled, and again in Chapter 8, when applying the evolutionary optimisation scheme.

2.2 - Literature Review

The literature on spray drying covers a wide range of topics including the physics of atomisation, spray-air mixing, heat transfer to and mass transfer from drying droplets, airflow patterns in driers and the physical properties of spray dried products. Annual reviews of the drying literature appeared in Industrial and Engineering Chemistry written by Friedman (1946-1951), Marshall (1953), Gluckert (1954,1955), Bagnoli (1956,1957) and McCormick (1959-1970). Comprehensive reviews have been written by Marshall (1952) and Masters (1968, 1972). Papers on the specific topics of spray drier control systems, centrifugal pressure nozzle hydrodynamics and the effects of drying conditions on powder properties have been selected from those reviewed for more detailed examination.

2.2.1 Spray Drier Moisture Control Systems

The design of control systems for spray driers has been studied by Hatfield (1971) who identified variations of the two basic arrangements for controlling the outlet air temperature; manipulation of the inlet air temperature and liquid feedrate. The inherent safety of the former system is discussed. Six case studies show the dependence of the optimum choice of system on the details of the product, and the means of atomisation, air heating and powder collection. Masters (1972) gives a similar treatment of the subject and describes four schemes for coupling an evaporator and spray drier. Both authors give considerable attention to safety systems.

An alternative approach developed by Shinskey (1968) for fluidised bed driers has been applied to a multiple nozzle spray drier by Myron, Shinskey and Baker (1973). A conventional control system adjusts the inlet air temperature to keep the outlet air temperature at setpoint. This setpoint is itself adjusted in response to changes in the inlet air wet and dry bulb temperatures by a second controller cascaded on to the first. The authors report a substantial reduction in moisture variation when their compensated control system was applied.

The advent of on-line infra-red moisture meters has made direct

control of product moisture possible. At least one such system has been installed on milk powder driers in the U.S.A. (Moisture Register Co., 1976).

A feature common to all these systems is that only one operating variable is manipulated in order to control the powder moisture. The choice of this variable, the design of the drier and the nature of the product will determine what other properties of the powder will be affected by the action taken to control the moisture. As an example, consider the effect of fluctuations in the total solids of the concentrate fed to a drier. Both the evaporative load and the degree of atomisation will be altered. If the feedrate is adjusted to restore the evaporative load, or the inlet air temperature is adjusted to change the energy input, the moisture may be returned to its target value, but the particle size, bulk density and solubility of the powder will have changed, as will the powder production rate. An alternative means of regulating the energy input, namely changing the drying air flowrate, has been investigated by Woodhams (1970), and found to have little effect on any of the powder properties except moisture. In general it will be necessary to manipulate several of the drier operating variables if control is to be exercised simultaneously over more than one of the powder quality variables.

2.2.2 Centrifugal Pressure Nozzle Hydrodynamics

The hydrodynamic behaviour of centrifugal pressure atomising nozzles has an important bearing on the performance of spray driers employing this means of atomisation. The size and geometry of the nozzles, and the viscosity and density of the fluid passed through them affect the relation between the flowrate through the nozzles and the pressure drop across them. This in turn affects the size distribution of the droplets in the spray and hence the surface area available for evaporation. These factors will also influence the droplet trajectories and the spray-air mixing.

The effect of nozzle orifice size and swirl velocity on the flow-pressure relationship for centrifugal pressure nozzles has been well researched by Marshall (1954), Hayashi (1962), Dombrowski and Munday

(1968) and Masters (1972) among others. The nozzle manufacturers provide data tables giving flowrate, pressure and spray angle information for their nozzles. The effect of fluid viscosity has received less attention, however. McIrvine (1953) found that the flowrate through centrifugal pressure nozzles operated at constant pressure increased with increasing viscosity. This increase continued until an air core could no longer form in the centre of the orifice. At higher viscosities still, atomisation became incomplete and the flowrate declined. This behaviour has also been reported for fuel atomisers by Giffen and Muraszew (1953) and Frazer, Eisenklam and Dombrowski (1957). Watanabe (1974) reported that for a wide range of sizes of Spraying Systems and Delavan nozzles, the flowrate at constant pressure declined with increasing fluid viscosity. Little quantitative information on the effect of viscosity is available in the literature, however. Some of the results of the present study have been reported by Bloore (1978).

2.2.3 The Influence of Process Variables on Powder Properties

The atomisation conditions are central to the spray drying process, and they play an important role in defining such powder properties as particle size, particle density and bulk density through their influence on droplet size. These powder properties also depend on the viscosity, temperature and concentration of the material being dried and on the inlet air temperature. Duffie and Marshall (1953), Tate and Marshall (1953) and Crosby and Marshall (1958) have examined these effects. The final particle size may be larger or smaller than the initial droplet size, depending on the material. For this reason only work done on milk powders has been selected for closer study.

The effects of atomising pressure and nozzle orifice size on the physical characteristics of wholemilk powder were investigated by Tracy, Hetrick and Krienke (1951) using Spraying Systems SX type nozzles. They found that for a constant nozzle size, the bulk density increased with increasing atomising pressure and decreased with increasing nozzle size at constant pressure. When the nozzle size was increased at constant feedrate, and the pressure was allowed to fall, the bulk density increased.

Amundson (1960) carried out a detailed study of the effects of concentrate properties and drier operating variables on skim milk powder. The effect of preheat treatment on concentrate viscosity was also investigated. The spray drier used in this work was a pilot scale tall-form unit fitted with Spraying Systems nozzles at the University of Wisconsin. A description of this drier, which was also used by Woodhams (1970) has been given by Amundson (1967). The variables Amundson studied, their levels and effects are given in Table 2-5. Each of these variables was studied independently, with the outlet air temperature being kept constant by varying the drying air flowrate.

TABLE 2-5 The Effects of Increasing Levels of the Processing Variables on Skim Milk Powder Properties Found by Amundson

Variable and Range	Solubility	Bulk Density	Particle Size	Covariates
Concentrate				
total solids (25 - 45 %)	increase	increase	none	viscosity, air flowrate
viscosity (148 - 1214 cp)	decrease	none	none	preheat temperature
temperature (16 - 71 C)	none	none	none	viscosity
Nozzle				
orifice size (0.79 - 1.61 mm)	increase	small decrease	small increase	feedrate air flowrate
core size (17, 20, 21)	none	none	small increase	feedrate air flowrate
Atomising pressure (6.9 - 20.7 MPa)	not reported	increase	decrease	feedrate air flowrate
Inlet air temperature (204 - 316 C)	decrease	decrease	increase	air flowrate

Hayashi (1962) made an extensive investigation of atomisation and spray drying mechanisms using skim milk concentrate. He found the particle density and the bulk density of the powder to be highly correlated. The bulk density was also found to increase with decreasing particle size.

Skim milk is preheated before evaporation to denature a proportion of the whey proteins. The amount of undenatured protein remaining is measured by the Whey Protein Nitrogen Index (WPNI). Both the temperature and time of preheating may be varied, and a regression model of WPNI as a function of the preheat temperature and time has been fitted by Baucke and Newstead (1972).

The formation of vacuoles in milk powder particles with a resultant decrease in particle density has been studied by Verhey (1971, 1972a, 1972b). When centrifugal pressure atomisation was employed, a small amount of air was incorporated into the droplets. The subsequent expansion of this air was dependent on the inlet air temperature.

The important quality parameters of spray dried milk powders and the factors affecting each of them have been summarised by Woodhams and Murray (1974).

A feature of much of the literature on spray drying in the dairy industry is the correlation of powder properties with the drier outlet air temperature. While this is a convenient way of expressing the results of experimental work, the outlet air temperature is itself dependent on the drier input variables. Any relationship observed between output variables will depend on which inputs are varied. One objective of the present study was to derive a comprehensive model of the powder properties as functions of the input variables, so that any of the various ways spray driers are operated may be simulated.

CHAPTER 3 - EXPERIMENTAL

3.1 - The Pilot Scale Equipment

The pilot plant used in this work comprises a Wiegand three effect falling-film evaporator and De Laval tall-form spray drier with a nominal processing capacity of 1800 l/h of skim milk to give 180 kg/h of powder. It is interfaced to an IBM System/7 process control computer which records up to 65 instrument readings and controls a total of ten variables on the evaporator and drier.

The evaporator is fed from a balance tank of 270 l capacity fitted with two float valves to maintain a constant head of milk or to admit water when the level falls below a lower limit. The evaporator is fitted with two sets of preheating equipment; two shell and tube heat exchangers for indirect preheating and a two stage direct steam injection unit. This latter system was used in all the experimental work described here. The preheated milk may be held for times ranging from one second to four minutes by means of a set of holding tubes before it enters the evaporator. A mixing condenser with a barometric leg and a two stage steam jet ejector vacuum maintenance system with an interstage mixing condenser is fitted to the evaporator. Between the evaporator and the drier are two balance tanks, one of which is used as a surge vessel. A schematic diagram of the evaporator is shown in Figure 3.1.

Concentrate from the evaporator is pumped by a centrifugal pump through a plate heat exchanger, a swept surface heat exchanger and then through a measurement train comprising a thermocouple, a viscometer, a sample cock, a volumetric flowmeter, and a densitometer before it is delivered to a high pressure pump. This pump is fitted with a variable speed gearbox and is rated to 34 MPa (5000 psi). The high pressure concentrate passes a pressure sensor and another thermocouple before arriving at a single centrifugal pressure nozzle atomiser. Figures 3.2 and 3.3 show photographs of the drier feed system and its associated instrumentation. The former shows the equipment as installed during the 1977/78 dairying season when only the plate heat exchanger was available and the latter photograph depicts the drier feed system as from mid 1978

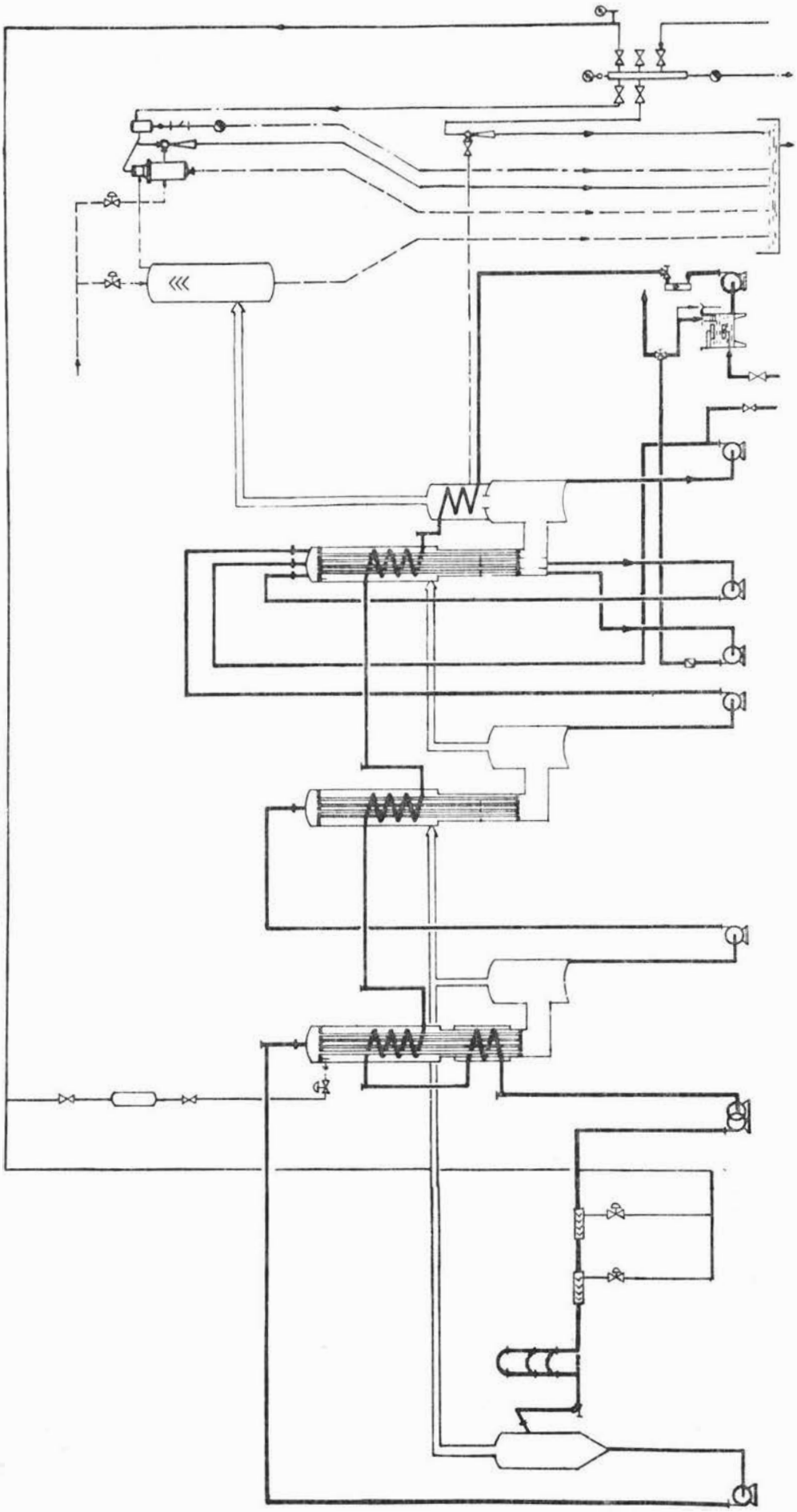


FIGURE 3.1 A Schematic diagram of the Wiegand evaporator

when the swept surface heat exchanger was installed. Details of the pumps and heat exchangers are given in Appendix I.

The drier has an inlet fan and an exhaust fan, each of which is fitted with motorised dampers to permit the airflow to the drier and the air pressure in the chamber to be independently regulated. The inlet air is heated by a direct fired natural gas burner. The drying chamber is an upright cylinder 2.13 m in diameter with a conical base. The overall height of the chamber is 9.15 m. A sketch of the general arrangement is shown in Figure 3.4.

Air enters the chamber through a throat placed centrally in the roof. The atomising nozzle is mounted centrally within the throat and usually projects about 80 mm into the chamber. The drier was supplied with two throat sections with diameters of 203 mm and 305 mm, although the larger throat is the one normally used. It is possible to change from one section to the other only when the drier is shut down and cool enough to work on. The height of the nozzle relative to the drier roof may be adjusted over a 170 mm range without interruption to the operation of the drier using the variable position nozzle holder illustrated in Figure 3.5. The nozzle can be changed only by removing the nozzle holder. In the course of the experimental work a technique was developed for changing the nozzle without shutting down the drier completely. The drier was fed with water until the feed line had been purged of concentrate and the air heater was turned to its low-fire position, giving an air inlet temperature of about 140 C. The flow of water was then stopped, the nozzle changed and powder production resumed before the outlet air temperature could rise to an unnacceptable level.

The drying chamber is fitted with a bustle for partial separation of the powder from the drying air. Several different ducting arrangements are possible, but only two were used in this work. These are illustrated in Figure 3.6. The first, which proved unsatisfactory for experimental work uses the bustle to remove the drying air to the two primary cyclones. The powder from these cyclones and the bottom of the cone is conveyed pneumatically to another cyclone, where it is bagged off. The primary cyclones and the cone blocked when the powder moisture

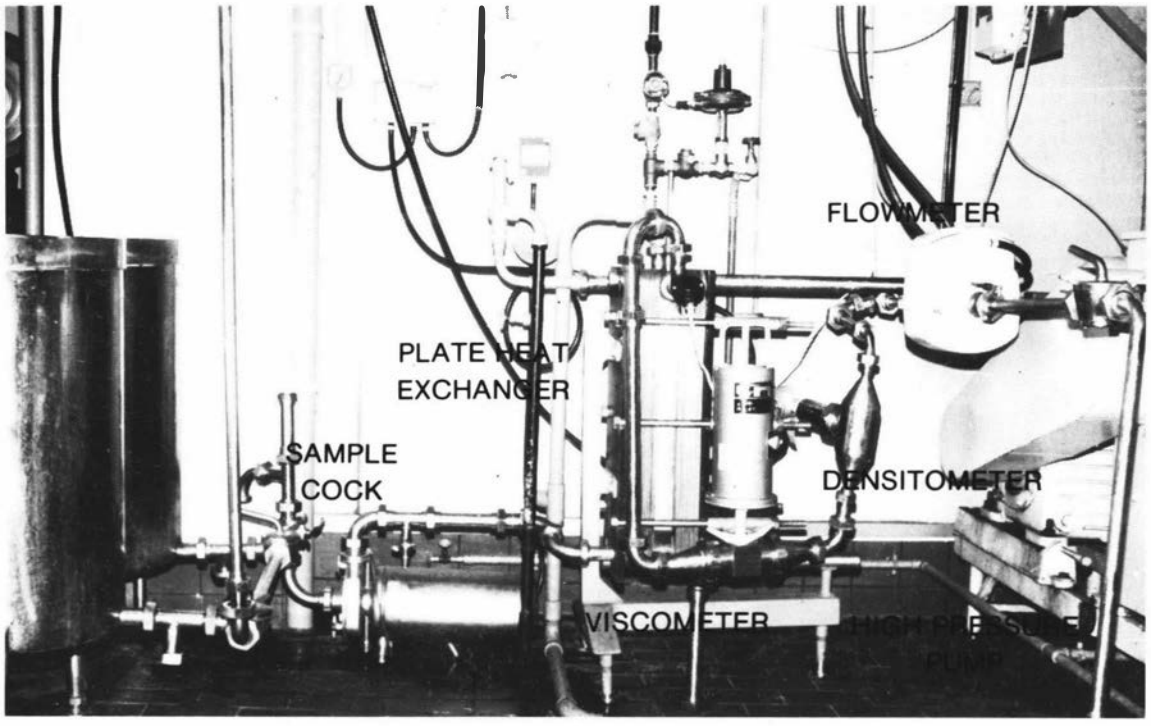


Figure 3.2 A photograph of the spray drier feed line showing the instruments as installed during the 1977/78 dairying season

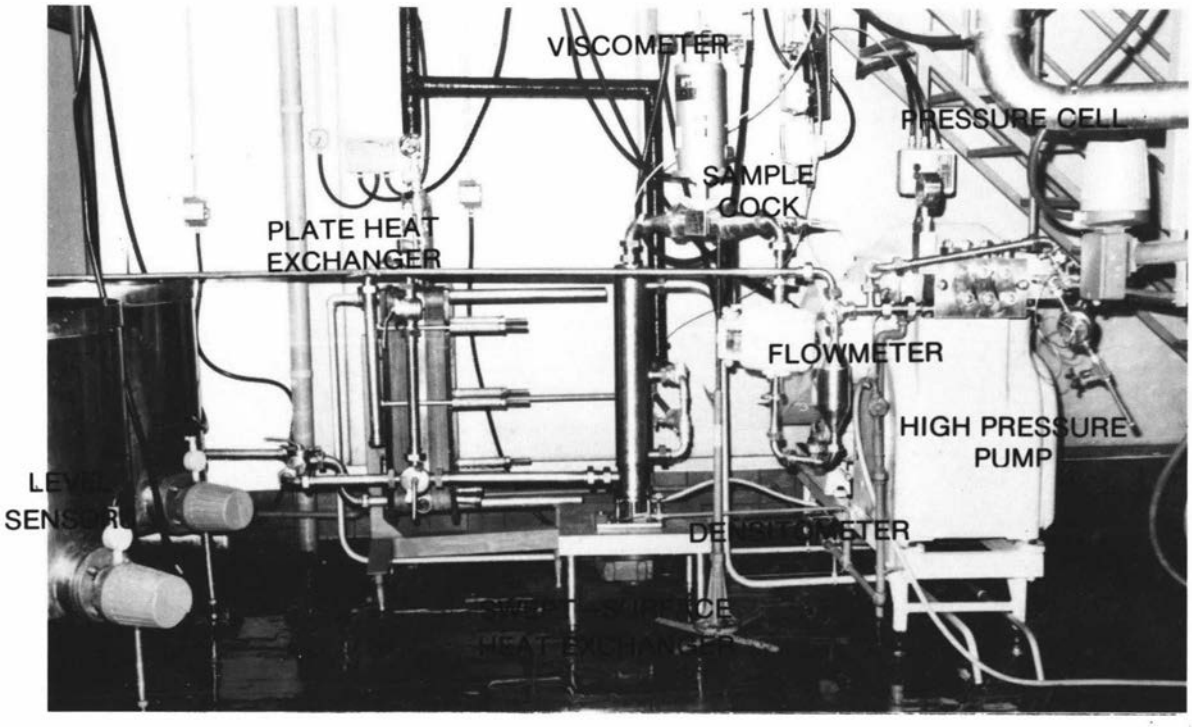
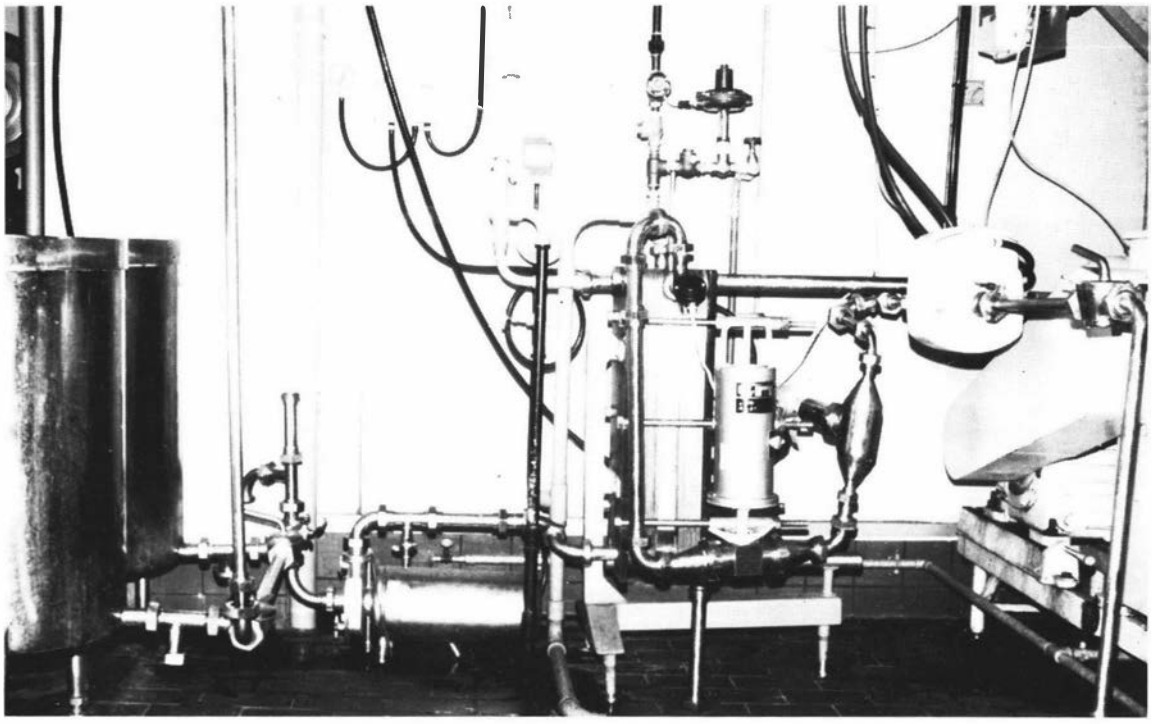


Figure 3.3 A photograph of the spray drier feed line showing the instruments as installed during the 1978/79 and 1979/80 dairying seasons



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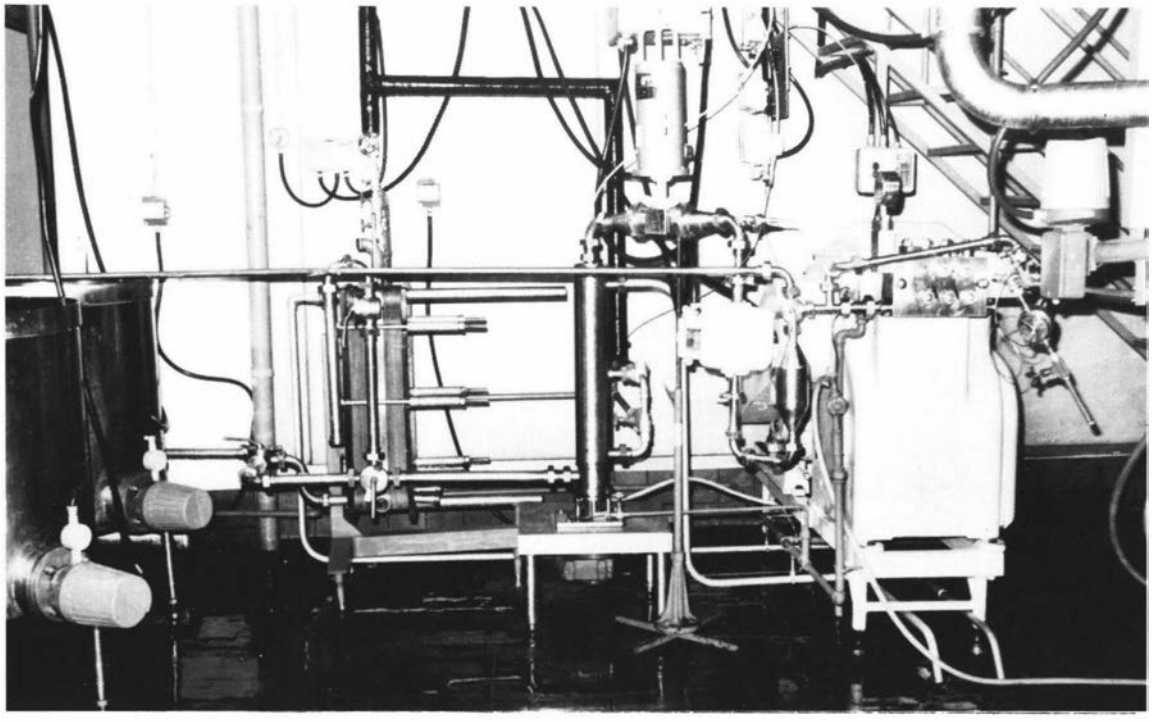

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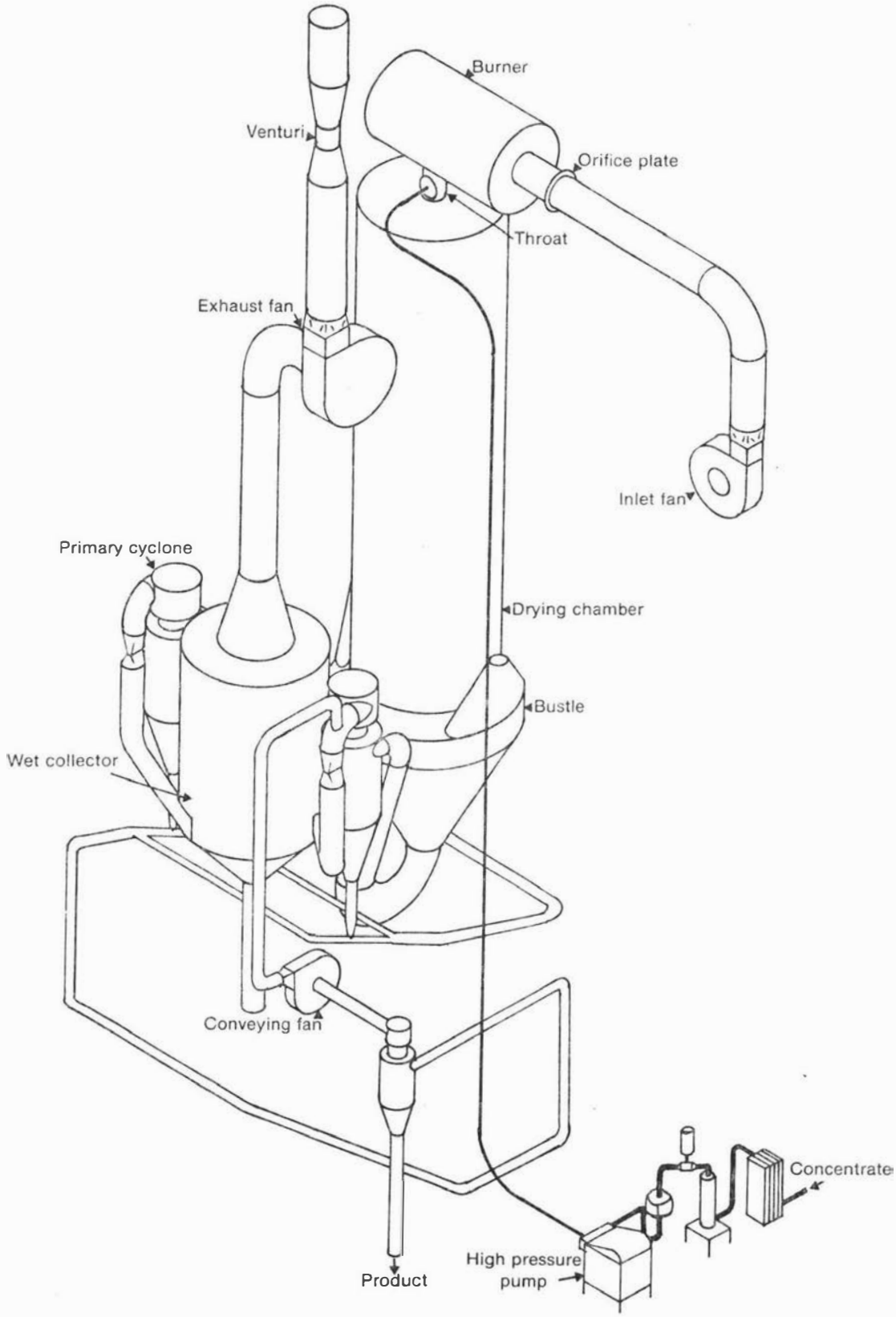


FIGURE 3.4 A sketch showing the general arrangement of the De Laval spray drier

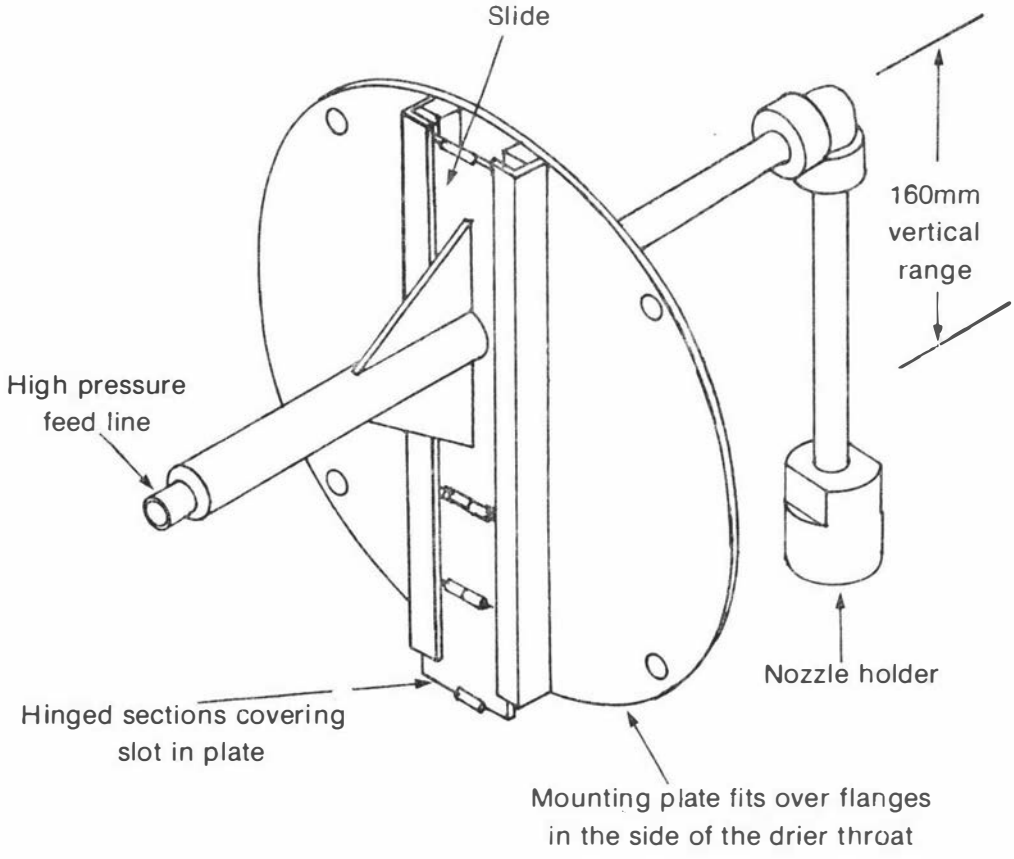
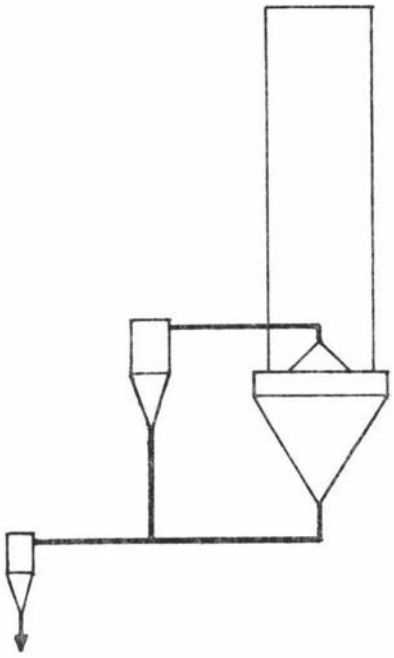
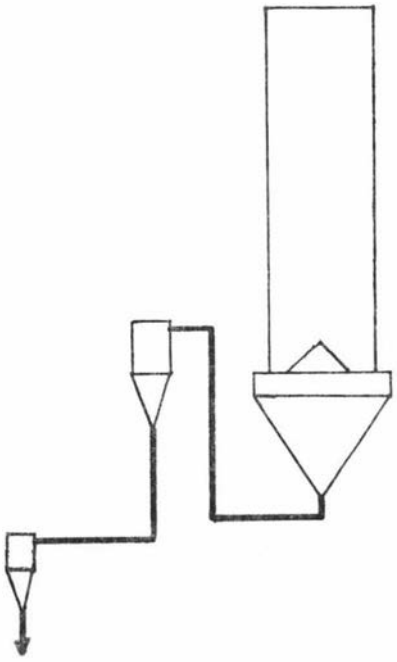


FIGURE 3.5 A sketch of the device constructed to allow the height of the nozzle to be varied



(a) Powder collected from the chamber and primary cyclones



(b) All powder collected from the primary cyclones

FIGURE 3.6 A diagram showing two of the drier ducting arrangements

exceeded 4 %. The second arrangement discharges all the powder with the air to the primary cyclones. The duct leading from the bottom of the cone is 460 mm in diameter and no blocking was experienced even at powder moistures in excess of 8 %. A pneumatic conveying line takes the powder from the primary cyclones to the cyclone at the bagging-off point. The exhaust air from all the cyclones passes to a venturi wet scrubber which removes any remaining powder as an anti-pollution measure. The exhaust fan discharges the air from the scrubber through an exhaust stack. The pneumatic conveying system uses filtered air which is cooled to about 3 C and then heated to about 16 C in a dehumidifier.

3.2 - Process Instruments and Actuators

Full details of the instruments and actuators fitted to the evaporator and drier are given by Marlow (1978). The description which follows covers only those instruments whose readings were used as experimental variables or covariates or as checks on abnormal conditions which might invalidate the results of a particular run. The names of the manufacturers, the model numbers and the calibrated ranges of these instruments are given in Appendix II. The valves and actuators on the pilot plant are fitted with either electro-pneumatic positioners or electro-pneumatic converters which accept a 4 to 20 mA current signal and transmit a 20 to 100 kPa (3 to 15 psi.) air signal.

The temperature of the skim milk leaving the evaporator feed balance tank is measured. Because the milk and water temperatures are usually different, this provides a warning when a supply tank runs dry and the float valve in the balance tank starts admitting water. The pre-heat temperature of the milk is measured and controlled. The temperature of the concentrate as it leaves the densitometer on the evaporator product line is measured so that total solids may be calculated from the density. All the temperatures are measured by copper-constantan thermocouples attached to a 50 channel scanning digital voltmeter. Melting ice is used to provide a reference temperature.

The flowrate of skim milk to the evaporator is measured with a magnetic flowmeter. The flowrate was kept constant at a figure which

ensured that the concentrate flowrate was always in excess of the drier feedrate. The skim milk flowrate determines the preheat holding time once a holding tube has been chosen. The flowrate of concentrate leaving the third effect is also measured with a magnetic flowmeter. This flowrate varies inversely with the total solids of the concentrate if the feed concentration and flowrate are fixed. These meters measure volumetric flowrate by sensing the voltage induced across a moving electrically conductive fluid by an imposed magnetic field. This voltage is converted to a 4 to 20 mA current signal.

A direct thrust actuator equipped with a positioner adjusts the variable speed hydraulic gearbox on the evaporator feed pump. The steam flow to each of the two preheaters and the steam flow to the first effect of the evaporator are regulated by control valves fitted with positioners. A control valve with a positioner is fitted to the water supply line to the main condenser and to the line to the inter-ejector condenser of the evaporator.

A densitometer is installed in the discharge line from the third effect of the evaporator. This instrument is used in controlling the concentration of the evaporated milk. The densitometers employ an element vibrated at its natural frequency, this frequency decreasing as the fluid density increases. Frequency to current converters are used to provide 4 to 20 mA current output signals.

The weight of liquid in each of the two balance tanks between the evaporator and drier is measured by liquid level transmitters. These are pressure transmitters fitted with stainless steel isolating diaphragms and are mounted in the tank wall at the bottom of each tank. When one of the tanks is used as a surge vessel, a check that the weight of liquid in the tank is increasing ensures that the evaporator is delivering more concentrate than the drier is taking. This enables the age of the concentrate at the time of drying to be established.

Great care was taken in installing the instruments in the drier feed line to keep the piping volume to a minimum so as to minimise the residence time of the concentrate between the heater and the nozzle. This was done because the viscosity of skim milk concentrate increases

with time, especially at temperatures above about 40 C. This phenomenon has been investigated by Buckingham (1978) for skim milk from the same source as that used in this work.

An in-line rotating bob viscometer measures the viscosity of fluids in the high pressure pump feed line. An electric motor with a three speed gearbox drives a stainless steel bob immersed in the flowing fluid through a magnetic coupling. The torque exerted by the motor in turning the bob at constant speed is measured by a variable resistor forming a voltage divider. A resistance to current converter provides a 4 to 20 mA current output. A second viscometer with a higher viscosity range is also available.

The temperature of the material leaving the swept-surface heat exchanger in the drier feed line is measured and may be controlled by a pneumatic valve installed in the cold water line to a steam-water mixer which supplies the heat exchanger. Prior to the installation of the swept-surface unit at the start of the 1978/79 dairying season the valve and steam-water mixer supplied the plate heat exchanger. At that time a pneumatic controller sent its output signal directly to the valve. Since mid 1978 an electro-pneumatic converter has been fitted to permit computer control of the heater.

A densitometer is mounted in the feed line to the high pressure pump where it is used in computing mass flowrates and in experiments to determine the relationship between concentrate total solids, density and temperature.

The flowrate of concentrate or other material to the high pressure pump is measured with a magnetic flowmeter. The variable speed hydraulic gearbox on the high pressure pump has a pneumatic actuator with a positioner to permit computer control of the flowrate.

The pressure of the fluid leaving the high pressure pump is measured with a pressure transmitter fitted with a stainless steel isolating diaphragm and a liquid filled extension tube. The siting of this instrument means that in normal operation the indicated pressure is the sum of the pressure drop across the 17 m high pressure line and that

across the nozzle itself. The temperature of the concentrate measured again about 2 m before the nozzle because the line is not insulated and appreciable heating or cooling may occur.

The flowrate of the drying air entering the burner is measured with an orifice plate and differential pressure transmitter. A constant drier airflow was required in most of the experimental work. The inlet and outlet fans on the the spray drier have sets of dampers adjusted by direct thrust pneumatic actuators fitted with positioners.

The absolute pressure of the drying air in the duct between the inlet fan and the burner is measured with a pressure transmitter. This measurement may be used with the differential pressure across the orifice plate in the duct and the air temperature at this point to calculate the mass flowrate of the air entering the drier. The pressure in the drying chamber relative to atmospheric is measured by a pressure transmitter mounted in the roof of the drier. This pressure was held constant throughout the experimental work. All the pressure, differential pressure and level transmitters are of the force balance type and give a 4 to 20 mA current output signal.

There is a control valve with a separate actuator and positioner on the gas supply line to the air heater. The flowrate of the natural gas to the burner is measured with an orifice plate and differential pressure transmitter. The gas consumption may be used in calculating the contribution of the water formed by combustion to the humidity of the drying air.

The temperature of the drier inlet air is measured before and after the burner. The former reading is used to correct the airflow measurement and the latter is the drier inlet air temperature. The conveying air temperature is measured after the chiller unit and again after the heating section of the dehumidifier to check that the dehumidifier is operating correctly. The temperature of the drier exhaust air is measured as it leaves the primary cyclones.

The absolute humidity of the air in the inlet duct to the burner is measured by a resistance bulb thermometer in a dewcell element. The

dewcell temperature is directly related to the dewpoint and hence to the absolute humidity of the air. A temperature transmitter provides a 4 to 20 mA current signal proportional to the dewcell temperature.

3.3 - Instrument Calibration

The pressure transmitters were all calibrated by the Physics and Engineering Laboratories of the DSIR when installed between 1974 and 1975. They were check calibrated by the Applied Mechanics Department of the same organisation during the winter of 1979. None of the instruments used in the experimental programme required adjustment.

The manufacturer's calibration line was used for both of the in-line viscometers. The electronic transmitters were adjusted to provide signals at the computer which were proportional to the instrument dial indicators.

The densitometers were calibrated using sucrose solutions of various concentrations. These solutions were deaerated and brought to 20 C. The density of each solution was determined with a 100 ml density bottle. The densitometers were then filled with the solution and their readings recorded. Usually deaerated water at a known temperature and sucrose solutions with five different densities covering the range indicated in Appendix II were used. Straight lines relating each instrument reading to the density were fitted by least squares regression and the resultant equations were entered into the process control computer. The densitometers were check calibrated at approximately three monthly intervals. The Barton densitometer in the feed line to the high pressure pump drifted during the experimental work, and required such frequent recalibration by early 1979 that the readings for January and February 1979 were between 50 and 60 kg/m^3 too high. It was not possible to calibrate this instrument immediately before these replicates of the main experiment. This was done just prior to the March 1979 runs, however. The Barton densitometer was replaced with the Dynatrol in December 1979. The Solartron densitometer has required only one very minor adjustment to compensate for drift over the six years it has been installed.

The magnetic flowmeter in the feed line to the high pressure pump was calibrated by running water through the meter and collecting it for a timed period in a milk can. This estimate of the flow was then compared with the average flowrate over the same period as logged by the computer. This was done at four flowrates and a calibration line relating the two sets of measurements was fitted by least squares. During the 1978/79 dairying season the flowmeter was found to be sensitive to the way in which the water was run through it, although this had not been observed at the start of the previous season. Three reproducible but different lines were obtained when tap water was run through the meter with the flowrate being adjusted by a valve before the meter, when the centrifugal pump was used with the flowrate being adjusted by a valve after the meter and when the centrifugal and high pressure pumps were used with the flowrate being set by the speed of the high pressure pump. Shortly after the end of the 1978/79 season the teflon lining of the flow tube became detached from the tube wall. Another flowmeter taken from the evaporator also failed and further work had to await the arrival of a replacement meter. The experimental implications of this aberrant behaviour are discussed in Chapter 4.

3.4 - Laboratory Analyses

The analyses performed during the course of the experimental work fall into three categories. The first is the measurement of concentrate total solids. This was one of the experimental variables. The second comprises the measurement of the powder moisture, Solubility Index and other quality parameters. The results of these analyses were used as response variables in the statistical analysis of the experimental results. The third category includes the composition of the milk solids, for example the protein content. These measurements were used as covariates in the statistical analysis of the experimental results. Details of the laboratory equipment used in those analyses performed largely by machine are given in Appendix II.

3.4.1 Total Solids Determination

The total solids of the concentrate and the skim milk prior to

evaporation were determined by the method of Mojonnier and Troy (1925). The results are expressed as the weight percentage of dry solids in the sample.

3.4.2 Powder Quality Analyses

These analyses were carried out by the staff of the Milk Powders and Drying Section of the NZDRI. The measurement of moisture content by the oven method, Whey Protein Nitrogen Index (WPNI) and Solubility Index was done in accordance with the methods set out in the Dairy Division publication "Standard Chemical Methods", (1979). The method for each measurement is briefly outlined here.

The moisture content of the powders made in the course of the experimental work was determined by measuring the loss of weight of a sample of powder oven dried for two and a half hours at 108 C. The result is expressed as the percentage of moisture in the moist sample. For the optimisation trial, the oven method was too slow , so an automatic Karl-Fischer titrator was used as described by Thomasow et al. (1972).

The Solubility Index (SI) test measures the volume of sediment remaining after centrifuging a reconstituted powder sample. A 10 g sample of powder is mixed with water in a standard mixer and allowed to stand for 15 minutes at 24 C before being centrifuged. The SI is the volume in ml of sediment and is therefore a measure of insolubility. Again, this method was too slow for the optimisation trial, and a faster version of the method, described in Appendix V was developed.

The bulk density of a powder is measured by filling a measuring cylinder with powder and determining the weight of powder. The cylinder is then mechanically tapped 10, 100 and 1000 times with the volume being recorded after each of these numbers of taps has elapsed. The bulk densities are calculated as the powder weight divided by its respective volumes and are expressed as g/ml. The initial density is known as the poured bulk density.

The Whey Protein Nitrogen Index (WPNI) is used to assess the

extent to which the whey proteins in the milk have been denatured by the preheat treatment given prior to evaporation. The test method is that of Sanderson (1970) and involves precipitating the caseins and denatured whey proteins by saturating the reconstituted skim milk with sodium chloride. The undenatured whey protein nitrogen content of the filtrate is then estimated by binding with amido black, centrifuging and reading the optical density of the supernatant at 615 nm in a spectrophotometer. Calibration is done using standard powders. The result is expressed in mg undenatured whey protein per g of powder.

The particle density of the powder has been measured in two ways. The volume of a weighed amount of powder is measured with an air pycnometer. Since air penetrates all of the interstices in the powder particles which communicate with the exterior surface, this density provides an estimate of the relative volume of closed vacuoles in the particles. The other density measurement is made with a glass density bottle of known volume and isopropyl alcohol of known density. The weight of a bottle containing a weighed amount of powder suspended in the alcohol enables the volume and hence the density of the powder to be calculated. This density is required by the Andreasen pipette size analysis method described below. In general the densities given by this method are lower than those measured with the air pycnometer, since the alcohol does not permeate the particles as readily as air.

The powders made in the course of the experimental work frequently had more than 50% by weight less than 45 μm. For this reason an Andreasen pipette liquid sedimentation apparatus was used for size analysis, using the method of British Standard 3406: part 2: 1968. This procedure gives the cumulative weight percentages of the powder less than each of five different particle sizes. From this particle size distribution the surface-volume mean diameter (D_{sv}) and the standard deviation of the particle size distribution (σ_g) may be calculated. An example of the calculation and a set of size distribution curves appear in Appendix V.

3.4.3 Compositional Analyses

All compositional analyses were performed by the staff of the

Analytical Chemistry Section of the NZDRI. Some of the mineral analyses were repeated in a single batch by the Auckland Regional Laboratory of the Ministry of Agriculture and Fisheries, Dairy Division. This was done to eliminate possible errors arising from changes in laboratory staff over the two years of the experimental work. A description of the method, or where available a reference to a published method, is given for each analysis. One sample of powder from each day's milk was submitted for analysis. The moisture content of the sample was determined before and after analysis, and the composition was expressed on a dry powder basis using the average of the two moisture contents.

The lactose content of the powders is determined using the copper reduction method of Lane and Eynon as modified by McDowall and Dolby (1935).

The protein content of the powders is obtained by multiplying the Total Nitrogen determined by the Kjeldahl method using an automatic analyser by 6.38 in accordance with British Standard 1741.

Non Protein Nitrogen and Non Casein Nitrogen determinations are made by preparing solutions according to the method of Rowland (1938) and then analysing them as for Total Nitrogen.

The ash content of a skim milk powder is determined by the method given in British Standard 1743, except that the furnace conditions are 550 C for 15 hours.

The calcium content of a milk powder is found by dissolving a sample of the powder in deionised water and then following the direct titration method of Pearce (1977). This is a complexometric method.

The levels of sodium and potassium in skim milk powder are determined by reconstituting the powder and precipitating the protein with trichloroacetic acid. Appropriate dilutions of the filtered supernatant are then analysed against standard solutions by flame emmission spectrophotometry.

The magnesium content of the powder is determined by flame absorption spectrophotometry of diluted filtrates from the sodium procedure, using the same equipment.

The inorganic phosphate content of skim milk powder is determined colourimetrically as the phosphomolybdate complex using the method of Watanabe and Olsen (1965).

The results of the calcium, sodium, potassium, magnesium and phosphate analyses are expressed as millimoles per kilogram of dry powder. The ash content is expressed as weight percent ash in the dry powder.

3.5 - Experimental Considerations

The variables whose effects were to be investigated were identified by considering heat and mass balances together with information obtained from the literature and from dairy companies operating spray driers. The variables fall naturally into three groups; drier design variables, plant operating variables and properties of the drying air, milk and milk concentrate. These variables are listed in Table 3-1.

The choices of designs for the experimental work were severely restricted by four considerations.

- A maximum volume of 8300 l of skim milk could be handled on any one day, giving a processing time of about four and a half hours.
- The time required to stabilise the plant at each new set of operating conditions.
- The accuracy with which each independent variable could be controlled.
- The need to cater for day to day variations in the composition and hence the processing characteristics of the milk.

TABLE 3-1. Variables of Interest in Describing the Behaviour of the Spray Drier

Design Variables:	G	Drying air flowrate
	D	Throat diameter
	N_p	Position of nozzle in throat
	N_o	Nozzle orifice diameter
	N_s	Nozzle swirl chamber
Operating Variables:	T_p	Milk preheat temperature
	t_p	Milk preheat holding time
	T	Inlet air temperature
	TS	Concentrate total solids
	F	Concentrate volumetric feedrate
	P	Atomising pressure
	T_c	Concentrate temperature
	t_c	Concentrate holding time
Material Properties:	H	Drying air humidity
	μ	Concentrate viscosity
	ρ	Concentrate density
	C	Milk composition - Lactose, Protein, Ash, Fat, Sodium, Magnesium, Potassium, Calcium, Phosphate

Table 3-2 gives the times required to change variable levels and the accuracy of control expressed as the 95 % confidence intervals about the setpoints. The precision of each measurement is given in the same way. The times required to accomplish a level change are sometimes additive, for example the concentrate temperature took about four minutes to change, but when this temperature was used to change the concentrate viscosity and hence the atomising pressure, final adjustment had to wait until the concentrate total solids had stabilised. The long time for the preheat temperature change is a reflection of the disturbance this change causes in the concentrate total solids. The drier inlet throat section could be changed twice and the preheat holding tubes once

TABLE 3-2 Times Required to Change Variable Levels and the Accuracy of their Control and Measurement

	Variable	Time (min)	95 % confidence intervals	
			Control	Measurement
G	Drying air flowrate (kg/min)	2	±0.8	±0.6
D	Throat diameter	40	-	-
N_p	Position of nozzle	1	-	-
N_o	Nozzle orifice diameter	10	-	-
N_s	Nozzle swirl chamber	10	-	-
T_p	Milk preheat temperature (C)	15	±0.4	±0.3
t_p	Milk preheat holding time	60	±0.4 %	±0.4 %
T	Inlet air temperature (C)	2	±0.7	±0.3
TS	Concentrate total solids (%)	15	±0.8	±0.2
F	Concentrate feedrate (l/h)	2	±5.5	±1.0
P	Atomising pressure (MPa)	4	±0.8	±0.1
T_c	Concentrate temperature (C)	4	±0.8	+0.3
μ	Concentrate viscosity (cp)	4	± 5 %	±0.8 cp

in any one day.

These constraints dictated that the effects of the variables in Table 3-1 be investigated in a series of experiments, each using a restricted subset of these variables. These subsets were chosen using the approach of Rudd and Watson (1968). These authors have formulated a systematic procedure for the analysis of the information flow structure of the equations which describe a process. This procedure is intended to assist in the design of processes, but may be applied to the analysis of an existing process whose design equations are unknown.

The procedure begins by considering the degrees of freedom of the system, i.e. the number of variables whose values may be chosen independently. If the process has M variables, and there are N independent equations linking them, then the number of degrees of freedom is M-N. When the process is running, there should be no remaining degrees of freedom because all the variables which may be freely chosen will have

been set at values such that the process is optimised in some way. The optimal choice of these values of the operating variables is the objective of the present study. The number of degrees of freedom for industrial spray driers varies from plant to plant depending on the design of the driers and their ancillary equipment. The implications of this are discussed in Chapter 8.

A matrix called the structural array is drawn up. The columns correspond to all the variables which enter into the design or operation of the process and the rows correspond to all the equations linking them. The matrix elements are all zero except where a variable enters an equation, when the matrix has the element 1. The structural array for the spray drier is shown in Table 3-3. The variable symbols are those of Table 3-1, with the addition of subscripts i for inlet and o for outlet, and the abbreviation of WPNI by W and the nozzle variables by N. Columns in which variables fixed by design or the environment appear are deleted (set to zero). The following algorithm is then applied.

1) Locate a column with only one non-zero element and delete the column and corresponding equation.

2) Repeat step 1 until all the equations have been eliminated.

TABLE 3-3 Structural Array for Spray Drying Variables

	VARIABLES																		
EQUATIONS	G	D	N	T_p	t_p	T	P	F	TS	μ	T_c	t_c	H_i	T_o	H_o	M	BD	SI	W
Mass balance	1	0	0	0	0	1	0	1	1	0	0	0	1	1	1	1	0	0	0
Heat balance	1	0	0	0	0	1	0	1	1	0	1	0	1	1	1	1	0	0	0
Nozzle equn	0	0	1	0	0	0	1	1	1	1	0	0	0	0	0	0	0	0	0
Viscosity fn	0	0	0	1	1	0	0	0	1	1	1	1	0	0	0	0	0	0	0
TS-density fn	0	0	0	0	0	0	0	0	1	0	1	0	0	0	0	0	0	0	0
WPNI equation	0	0	0	1	1	0	0	0	0	0	0	0	0	0	0	0	0	0	1
Moisture equn	1	1	1	0	0	1	1	0	1	1	0	0	1	0	0	1	0	0	0
BD equation	1	1	1	0	0	1	1	0	1	1	0	0	0	0	0	0	1	0	0
SI equation	1	1	1	0	0	1	1	0	1	1	0	0	0	0	0	0	0	1	0

The remaining variables are the recommended design variables, the choice of which gives the precedence order in which the equations are to be solved. Should this algorithm not eliminate all the equations, those remaining are solved simultaneously. The precedence order is the reverse of the order in which the equations were deleted from the matrix. A computer program to implement this algorithm was written so that the effects of fixing different variables could be readily determined. This analysis was helpful in deciding how to partition the variables into groups which could be investigated independently of one another without danger of overlooking interactions between variables in different groups. The following partitioning of variables was decided upon for the pilot plant study.

Group 1 $WPNI = f (T_p, T_p)$

Group 2 $TS = f (\rho, T_c)$

Group 3 $\mu = f (TS, T_c, C)$

Group 4 $\mu = f (TS, T_p, t_p, T_c, t_c)$

Group 5 $P = f (F, \mu, \rho)$

Group 6 $(M, SI, BD) = f (T, TS, F, P, T_c, C)$

Group 7 $(M, SI, BD) = f (D, N_p, N_s, N_o)$

Once the independent variables for each experiment had been decided upon, experimental designs could be chosen. The maximum and minimum levels for each variable were determined from previous operating experience and industrial practice. The accuracy of control then fixed the closest practicable spacing of the levels within the allowable range. Designs were sought which would give polynomial response surface models with first order interaction and squared terms. A high degree of orthogonality was felt to be desirable. In particular, designs were sought which could be run in orthogonal blocks, each block being carried out on a different day with a different milk supply.

In an extensive review of Response Surface Methodology, Hill and Hunter (1966) summarise the experimental designs available and give some examples of the successful use of the technique. They point out the benefits of canonical analysis in gaining insight into the nature of the response surface and in elucidating the underlying mechanisms.

It was also necessary to consider the run order for the experiments. The time taken to accomplish a change in the levels of different variables varied widely as Table 3-2 shows. Complete randomisation of the run order within each block was therefore impractical. The selection of good time order sequences when factor level changes are expensive or time consuming has not received much attention in the literature. Joiner and Campbell (1976) reviewed what work has been reported in this area, and have proposed a type of Monte Carlo technique to generate sets of run orderings which offer good protection against time order dependencies within the constraints placed on the number of level changes that are practical.

The method adopted for the experimental work on the spray drier was to fix the number of level changes permitted each day for the variable which took the longest time to change. Depending on the experiment, this was the concentrate total solids, the air inlet throat diameter or the preheat holding time. The run order for this variable was then chosen randomly. The other variable levels for each run were then picked by plotting them on a graph of variable level against run number so as to obtain a pattern which showed no apparent linear or quadratic time dependence. An example of the graph for one of the replicates of the main seasonal experiment is given in Figure 3.7.

3.6 - Experimental Designs and Methods

Seven experiments were conducted to obtain the regression models required to describe the characteristics of the milk concentrate and the drying process, and to confirm that the partitioning of the variables into subsets was correct. The experimental designs chosen were all full or fractional factorials. These designs have clearly separated variable levels and are reasonably tolerant of failure to attain the target values for the independent variables exactly. They meet the orthogonality and blocking criteria set out above. The design matrix for each experiment is given in Appendix III, along with the values of each level of the independent variables. The experimental data are tabulated in Appendix VI. No experimental work was undertaken to obtain the equations for variables in groups 1 and 2. The effect of preheat conditions on

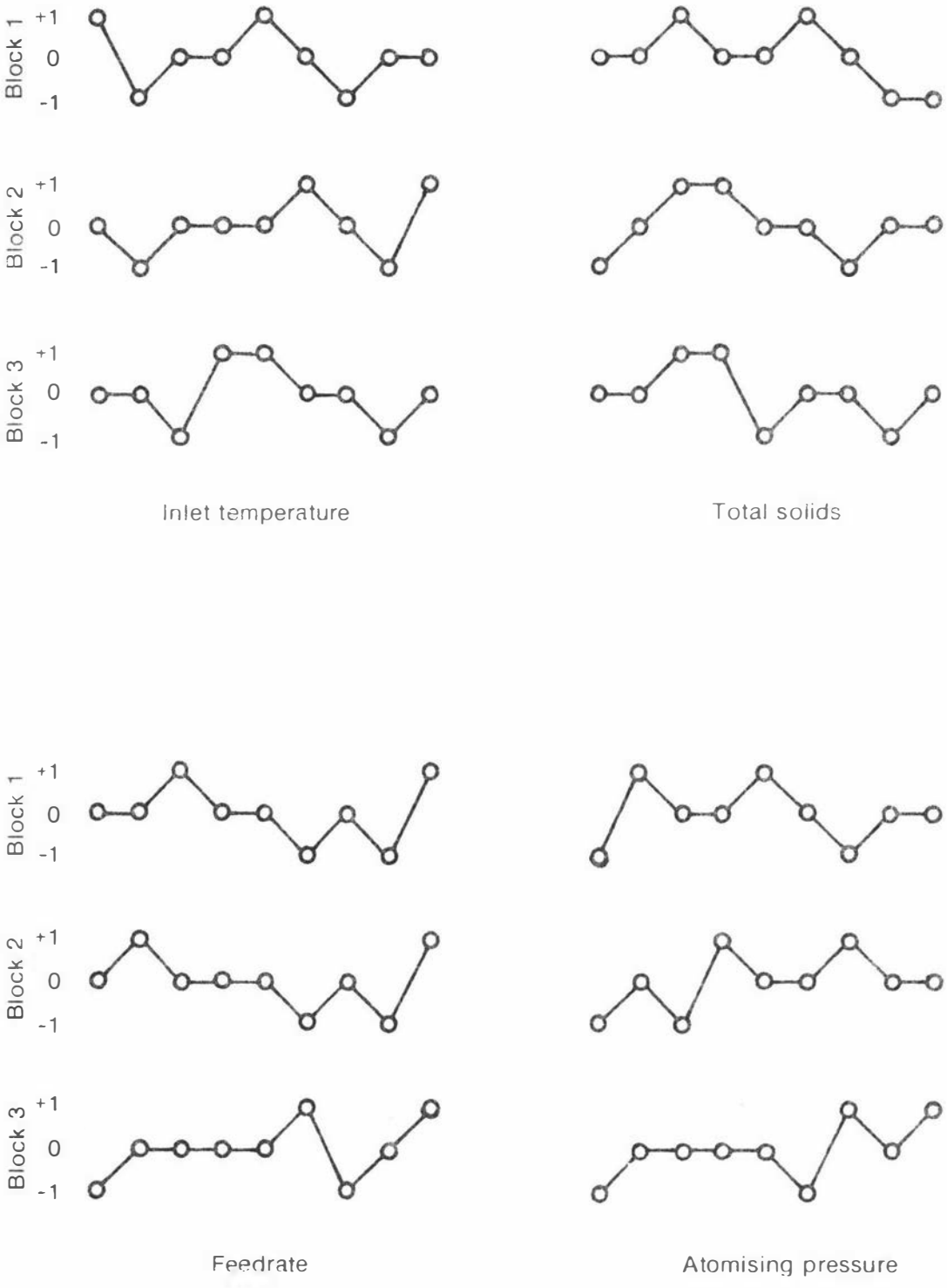


FIGURE 3.7 The run order for replicate 10 of the main seasonal experiment

WPNI had already been extensively investigated at the NZDRI by Baucke and Newstead (1972). A regression equation for skim milk concentrate total solids as a function of density and temperature was fitted to the data reported by Hall and Hedrick (1966). These two relationships are presented as part of the spray drier simulation model in Chapter 5.

The procedure for the experiments in which powder was made was as follows.

1. Change the evaporator final effect concentrate density setpoint.

2. Change the inlet air temperature setpoint.

3. Change the concentrate feedrate setpoint.

4. Begin adjusting the temperature of the concentrate after the heat exchanger in the drier feed line.

5. When the concentrate density has reached setpoint and a further 150 seconds have elapsed to allow the change to reach the heat exchanger, make a final adjustment to the concentrate temperature to change its viscosity and thereby bring the atomising pressure to its setpoint.

6. Withdraw a sample of concentrate from the line over a 30 second period, noting the computer recording interval corresponding to the start of sampling.

7. Start powder sampling 150 seconds (first season) or 60 seconds (second season) after the start of concentrate sampling.

8. Once the powder sample has been taken, repeat the procedure.

The instrument readings were logged at 8 second intervals and the readings were related to the same material as it flowed through the pipework to the nozzle. The instrumentation was re-arranged between the two seasons and this accounts for the different delays between concentrate and powder sampling during the two seasons.

There was a delay of six minutes between changing the third effect density setpoint and the arrival of the start of the density change at the swept surface heat exchanger. Time could therefore be saved by entering the next density setpoint into the computer while waiting for the concentrate temperature and atomising pressure to settle. If this did not happen before the total solids began to change the point was missed, and would have to be attempted later in the day. A program was run at the end of each day to calculate the means and standard deviations of five readings of each variable for each run. The intervals over which they were taken were determined automatically from the concentrate flowrate and the volume of the pipework between each instrument in the high pressure pump feed line.

The largest experiment was used to obtain models of the powder properties as functions of air inlet temperature, concentrate total solids, concentrate feedrate and atomising pressure. At the same time models of concentrate viscosity as a function of total solids, temperature and the milk composition were obtained. A 3^{4-1} factorial design due to Box and Behnken (1960) was used. This design has the useful property that it breaks down into three orthogonal blocks of nine runs. Each block took a day to perform, so that the estimates of the main effects and two-way interactions were independent of day to day variations in milk composition. The experiment was performed 11 times between December 1977 and March 1979. It was not possible to complete the fifth replicate because the milk quality deteriorated to the point where the evaporator fouled shortly after processing started on the third day. This replicate was omitted from the statistical analysis, but the data were kept as a check on the predictive accuracy of the models.

The viscosity readings were regressed against the concentrate total solids and temperature each day, and the collected readings from both seasons were regressed against these variables and the milk

composition.

A 2^4 factorial design in two orthogonal blocks was used to investigate the effect of preheat temperature and holding time on the viscosity of skim milk concentrate. The levels of preheat temperature and time were chosen so that the low temperature, long time combination would give approximately the same WPNI as the high temperature, short time one. Two levels of concentrate total solids and concentrate temperature were used. The concentrate left the final effect of the evaporator at 45 C and took 150 seconds to reach the outlet of the swept-surface heat exchanger, where its temperature and viscosity were measured. It then passed at constant flowrate through a length of lagged pipe sufficient to give a further 150 seconds holding time before its viscosity was measured with a second viscometer. The concentrate was then pumped through a Delavan SB 54 nozzle and disposed of without drying. The atomising pressure was recorded to serve as a third estimate of viscosity. Only one change in preheat holding time was possible on each day, so the run order for this variable was low, high on the first day and high, low on the second, to guard against any linear time trend within each day.

A simple mixed two and three level factorial design was carried out to determine the influence of the drier inlet throat diameter and the position of the nozzle relative to the roof of the drier on the drying performance. Because of the time it took to change the throat section the three nozzle positions were nested within the two throat diameters. The design was repeated on the same day in such a way as to confer immunity to any linear time trend.

A similar design was used to investigate the effects of varying the nozzle spray angle, the nozzle position and the atomising pressure. Two nozzles of similar capacity but different spray angle were used. At the time this experiment was conducted, the time required to change the nozzle meant that all the runs with the first nozzle had to be completed before the second was installed. The three nozzle positions and two atomising pressures were therefore nested within spray angle. This experiment was not replicated.

The effects of the choice of nozzle orifice size and swirl chamber for Delavan SDX series nozzles were studied at two concentrate flowrates with a replicated 2^3 factorial design. The atomising pressures were adjusted to low and high levels corresponding to the high and low flowrates. The rapid nozzle changing technique described earlier made it possible to randomise the run order.

Once it was possible to make several changes of nozzle on one day, a further experiment involving nozzles could be be conducted. This used a 2^3 factorial design to study Spraying Systems SX series nozzles with two orifice sizes at two inlet air temperatures and two concentrate viscosities. The objective was to determine the effect of concentrate viscosity on the properties of the powder independently of the effect viscosity has on the pressure-flowrate relationship of nozzles.

The pressure-flowrate relationships of a variety of nozzle atomisers were investigated using the pilot scale equipment. Most of this work was done with aqueous sugar solutions of approximately 75 % (w/w) sucrose content. The sugar was dissolved in hot water in one of the balance tanks between the evaporator and spray drier. The solution was circulated through the plate heat exchanger, the swept surface heat exchanger, the instruments in the high pressure pump feed line and back to the tank by the centrifugal pump, until a clear, bubble-free solution was obtained.

The nozzle under test was installed on a branch of the drier high pressure feed line within a metre of the outlet of the high pressure pump. The spray was confined by a length of plastic hose fitted over the nozzle. The gaps between the hexagonal nozzle holder and the hose were sealed with tape to reduce air entrainment in the solution. The solution was collected in a small vessel and pumped back into the balance tank. The volume of the solution was about 90 l, so that on average the holding time in the balance tank was 20 min. This allowed bubbles of air incorporated in the solution to escape.

The plate heat exchanger was supplied with chilled water at about 4 C when temperatures close to or below that of the cold water supply were required. The swept-surface heat exchanger was used for regulating

the fluid temperature and hence its viscosity. The flowrate and temperature of the test fluid were controlled by the computer which also recorded the measurements of temperature, viscosity, density, flowrate and atomising pressure every three seconds. The means and standard deviations of eleven successive intervals in each run were calculated and printed by a program run at the end of each day.

CHAPTER 4 - RESULTS

The results of the experimental work have been divided into four categories, each of which will be presented separately. The appropriate models for each aspect of the behaviour of the drier and the concentrate fed to it will be brought together in the next chapter to form a simulation model of the drying process.

4.1 - Nozzle Hydrodynamics

Preliminary experiments using the pilot plant on skim milk concentrate showed that some nozzles exhibited a reduction in pressure drop at constant flowrate as the viscosity of the fluid pumped through them was increased. This effect was very pronounced for Delavan SDX series nozzles with orifice diameters from 1.37 mm to 1.75 mm and swirl chambers SA, SB and SC over the range of viscosities normally encountered in spray dried milk powder manufacture. In contrast, Spraying Systems SX series nozzles exhibited little response to viscosity changes over a wide range of orifice and core sizes. The results of this work are given in Figure 4.1 in the form of graphs of flow number; the ratio of flowrate (l/h) to the square root of pressure (MPa), against viscosity (p).

4.1.1 Nozzle Characterisation

A further series of experiments was conducted to obtain mathematical models of the viscosity sensitivity of the flow characteristics of these nozzles. The relationship of viscosity sensitivity to nozzle geometry was also investigated. The fluid chosen for this work was an aqueous sugar solution of approximately 75% (w/w) sucrose content. The actual concentration varied slightly from run to run due to dilution on filling the pipework and concentration through evaporation when high temperatures were used. This material was chosen for its Newtonian flow properties since the shear rate at which the viscosity was measured was calculated at 356 s^{-1} whereas the shear rates present in the orifices of the nozzles may be in excess of 125 000 s^{-1}. Skim milk concentrate was not used because it exhibits shear-thinning behaviour (Sone and Fukada, 1962 and Verhey, 1972a). The nozzles were also tested on water at various temperatures.

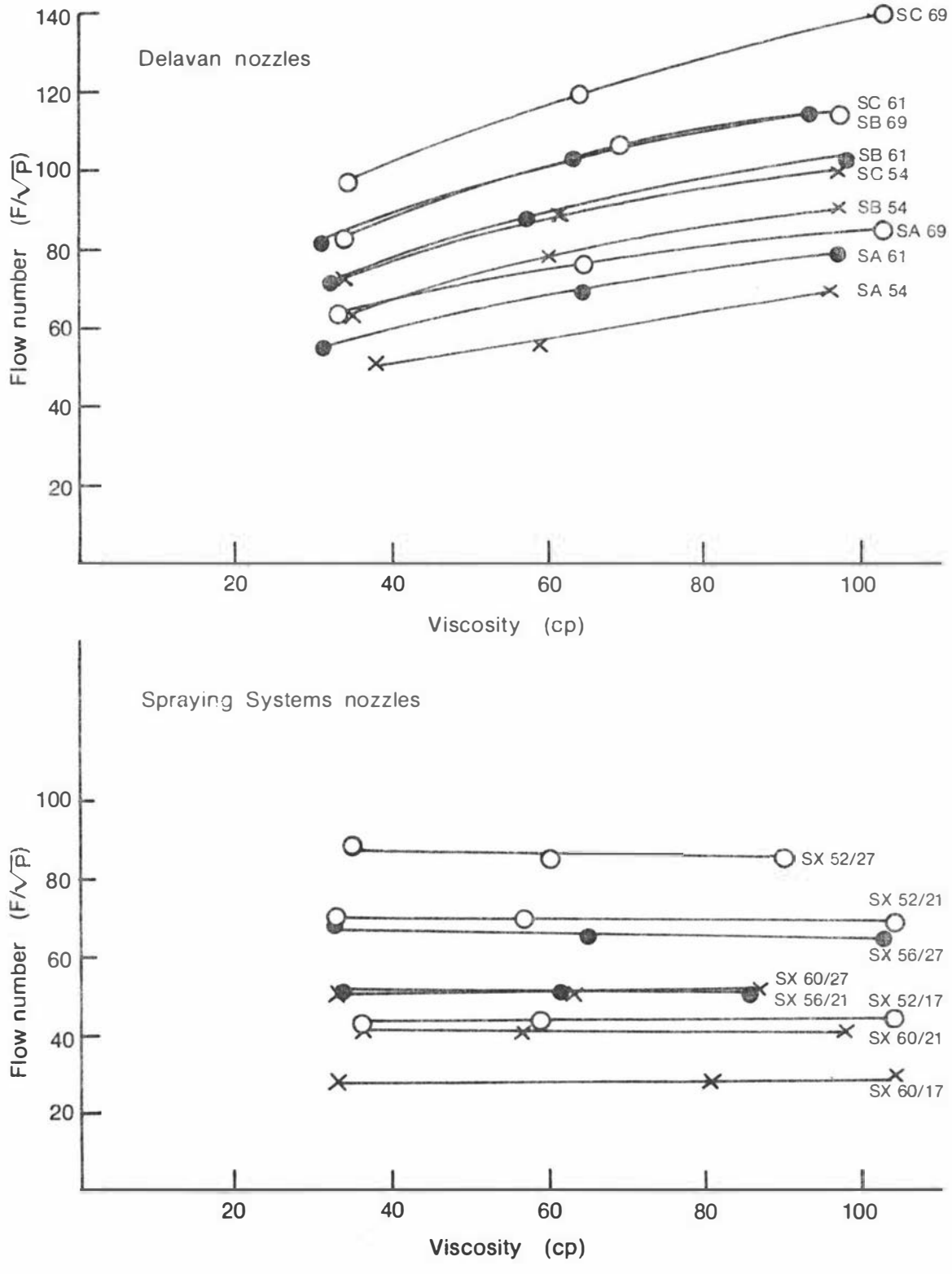


FIGURE 4.1 Graphs of flow number vs viscosity for some Delavan SDX series and Spraying Systems SX series nozzles

Four types of nozzle were chosen for this study; the Delavan SDX series and the Spraying Systems SX, SBC and Whirl Jet series. The nozzles are illustrated in Figure 4.2 and their dimensions are given in Table 4-1.

For the SX series nozzles, the cross-sectional area of the inlet ports is that measured normal to the axis of each slot. The tangential component of the liquid velocity will be one half that of the velocity in the slots themselves due to the angle at which the slots are set.

A single parameter was chosen to describe the geometry of the nozzles. This was R, the ratio of swirl chamber diameter d_m to the orifice diameter d_2. The value of d_m was taken to be twice the radius of the centre of the liquid inlet port or ports.

The data obtained from each nozzle were analysed in two stages. First, a model of the form $P = F^n \mu^m$ was fitted by multiple linear regression of ln P against ln F and ln μ, where P is the atomising pressure in MPa, F is the flowrate in l/h and u is the viscosity in poise. The coefficient n was then used to calculate the ratio P/F^n for each run. This ratio was then regressed against viscosity using the using an iterative numerical technique to give a model;

$$P / F^n = B_0 + B_1 \exp(B_2 \mu)$$

When multiplied through by F^n the resulting model was;

$$P = F^n (B_0 + B_1 \exp(B_2 \mu))$$

This is not a least squares fit, since n was estimated independently of B_0, B_1 and B_2. In order to assess the accuracy of the model, the predicted pressures were calculated for each run. The absolute value of the differences between the measured and predicted pressures were expressed as percentages of the measured pressure. The mean percentage was then taken to give a measure of the accuracy of the models.

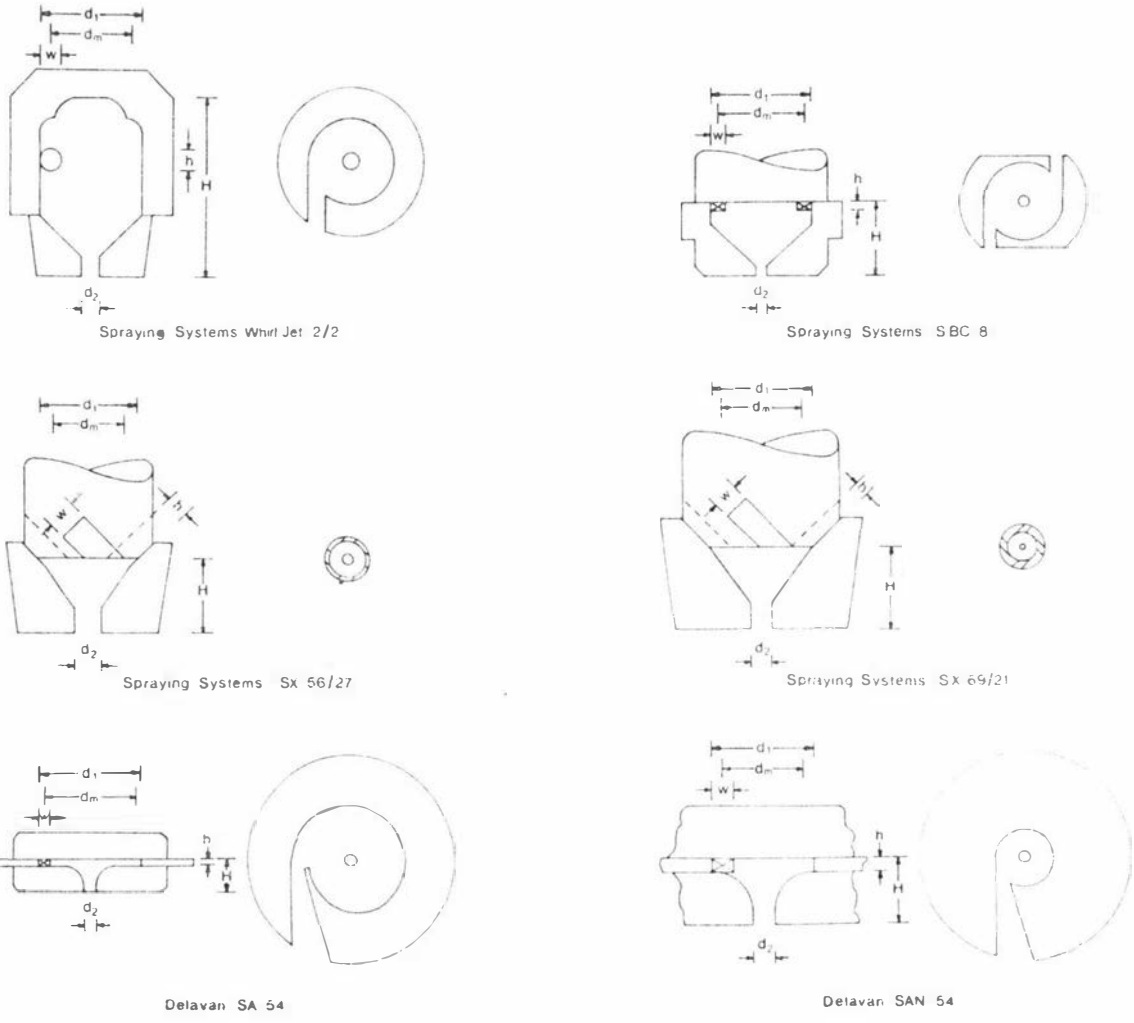


FIGURE 4.2 Drawings of the nozzles tested

TABLE 4-1 Dimensions of Atomiser Nozzles Tested

Nozzle	d mm	d mm	d mm	H mm	h mm	w mm	A mm
Spraying Systems							
Whirl Jet 2/2	11.113	9.938	1.981	19.304	2.440	2.440	4.676
SBC 8	9.525	8.001	1.194	6.604	0.762	1.524	2.323
SX 56/27	4.318	3.099	1.181	3.198	1.219	0.635	3.097
SX 69/21	3.556	2.286	0.742	2.794	0.889	0.508	1.806
Delavan							
SA 54	12.700	11.176	1.372	4.013	0.838	1.524	1.277
"SAN 54"	6.350	4.826	1.372	4.013	0.838	1.524	1.277
SB 54	12.700	11.176	1.372	4.547	1.372	1.524	2.091

Since the viscosity of the sugar solution was manipulated by varying its temperature, both the temperature and density of the solution were confounded with its viscosity. The temperature and density ranged from 16 to 73 C and 1290 to 1379 kg/m^3 respectively. The atomising pressure at constant flowrate is reported to be approximately proportional to the density of the fluid (Masters, 1972). The greatest percentage density difference measured during the evaluation of any one nozzle was 5.2 % with the average being 4.6 %. The density and temperature were not included in the analysis.

The models fitted to the experimental data are given below with the mean percentage error in using them to estimate the experimentally measured pressures.

Spraying Systems Whirl Jet 2/2

$$P = F^{1.9828}(1.8655 + 4.0923 \exp(-2.2483 \mu)) \times 10^{-5} \quad MPE = 3.2\ \%$$

Spraying Systems SBC 8

$$P = F^{2.2774}(2.2429 + 5.9144 \exp(-1.7212 \mu)) \times 10^{-5} \quad MPE = 6.4\ \%$$

Spraying Systems SX 56/27

$$P = F^{1.8901}(2.6957 + 1.9916 \exp(0.1182 \mu)) \times 10^{-4} \quad MPE = 2.2\ \%$$

or $$P = F^{1.8901}\ \mu^{0.0504} \times 10^{-4} \quad MPE = 2.9\ \%$$

Spraying Systems SX 69/21

$$P = F^{1.8154}(2.7796 + 1.3768 \exp(-0.2550 \mu)) \times 10^{-3} \quad MPE = 2.2\ \%$$

or $$P = F^{1.8154}\ \mu^{-0.0601} \times 10^{-3} \quad MPE = 2.1\ \%$$

Delavan SA 54

$$P = F^{2.1473} \left(6.9312 + 16.4761 \exp(-3.0694\,\mu) \right) \times 10^{-5} \qquad \text{MPE} = 4.2\ \%$$

Delavan SAN 54 (swirl chamber made by NZDRI)

$$P = F^{2.2830} \left(3.8367 + 9.3191 \exp(-1.9900\,\mu) \right) \times 10^{-5} \qquad \text{MPE} = 3.5\ \%$$

These models were all fitted to data obtained with the faulty magnetic flowmeter. A correction must be applied to the flow before they are used to calculate the pressure. The next section describes how this correction, which is given below, was obtained. If the correct flowrate is F_c then use

$$F = 1.081\,F_c + 12.22$$

in the equations above. These relationships are illustrated in Figure 4.3 as graphs of pressure against viscosity for various flowrates. The curves have been labelled with the correct flowrates.

The Delavan SB 54 nozzle used in the main seasonal experiment and a Spraying Systems SX 56/27 were tested with the replacement flowmeter. The equations below were fitted using the correct flowrate. The SB 54 equation was obtained by an iterative process varying the power n so as to minimise the mean percentage error.

Delavan SB 54

$$P = F^{2.345} \left(2.3679 + 5.30334 \exp(-3.4639\,\mu) \right) \times 10^{-5} \qquad \text{MPE} = 2.2\ \%$$

Spraying Systems SX 56/27

$$P = 1.3579\,F^{1.7086}\,\mu^{0.0514} \times 10^{-3} \qquad \text{MPE} = 1.4\ \%$$

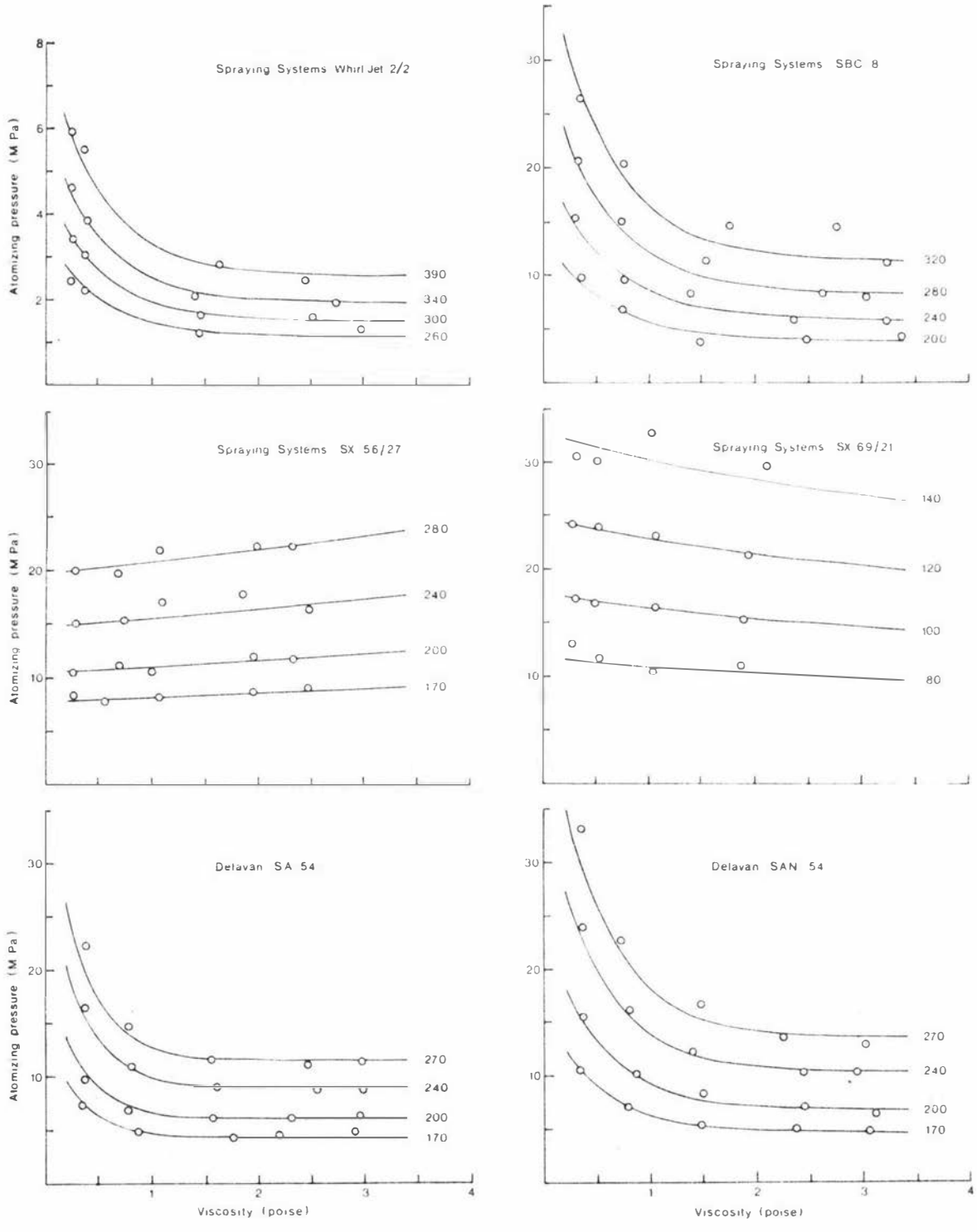


FIGURE 4.3 Graphs of pressure vs viscosity at various flowrates for a selection of pressure nozzles

The relationship for the SB 54 is plotted in Figure 4.4 as curves of constant flowrate on a pressure-viscosity graph.

The form of the viscosity dependence is that of an exponential decay. The three model paramaters may be manipulated to give an intercept at zero viscosity, an asymptote at infinite viscosity and a decay constant as follows:

$$\text{If } P / F^{n} = B_0 + B_1 \exp (B_2 \mu)$$

then when $\mu = 0$, $P / F^{n} = B_0 + B_1$

$\mu \rightarrow \infty$, $P / F^{n} \rightarrow B_0$

and $D = -1 / B_2$ is the decay constant

$M = (B_0 + B_1) / B_0$ is the ratio of the intercept to the asymptote and expresses the magnitude of the viscosity effect.

The results obtained using water as the test fluid were analysed separately because the density of the water under the test conditions was only 990 to 1010 kg/m^3 compared with the average density of 1330 kg/m^3 for the sugar solutions. On water, all the nozzles showed a dependence of pressure on flowrate raised to the 1.7 power except the Whirl Jet 2/2 for which the power was 1.6. The pressures recorded at a viscosity of 0.94 cp were 94 percent of those at 0.50 cp except for the Delavan SA 54 and SB 54 and the Spraying Systems SX 57/27 nozzles where the pressure fell to 89 percent of that at the lower viscosity. For all but the SX series nozzles, the pressures measured on water were higher than those obtained by extrapolating the curves obtained using sugar solutions, despite the considerably lower density of the water. The pressures measured on water lay very close to the extrapolated sugar values for the SX series nozzles.

The values of D and M derived from the models are given in Table 4-2, together with R, the ratio of swirl chamber diameter to orifice diameter for each nozzle. The terms D and M cannot be defined for the SX 56/27 nozzle as it did not exhibit a decline in pressure at increasing viscosities. Power law models have been given as alternatives for both Spraying Systems nozzles.

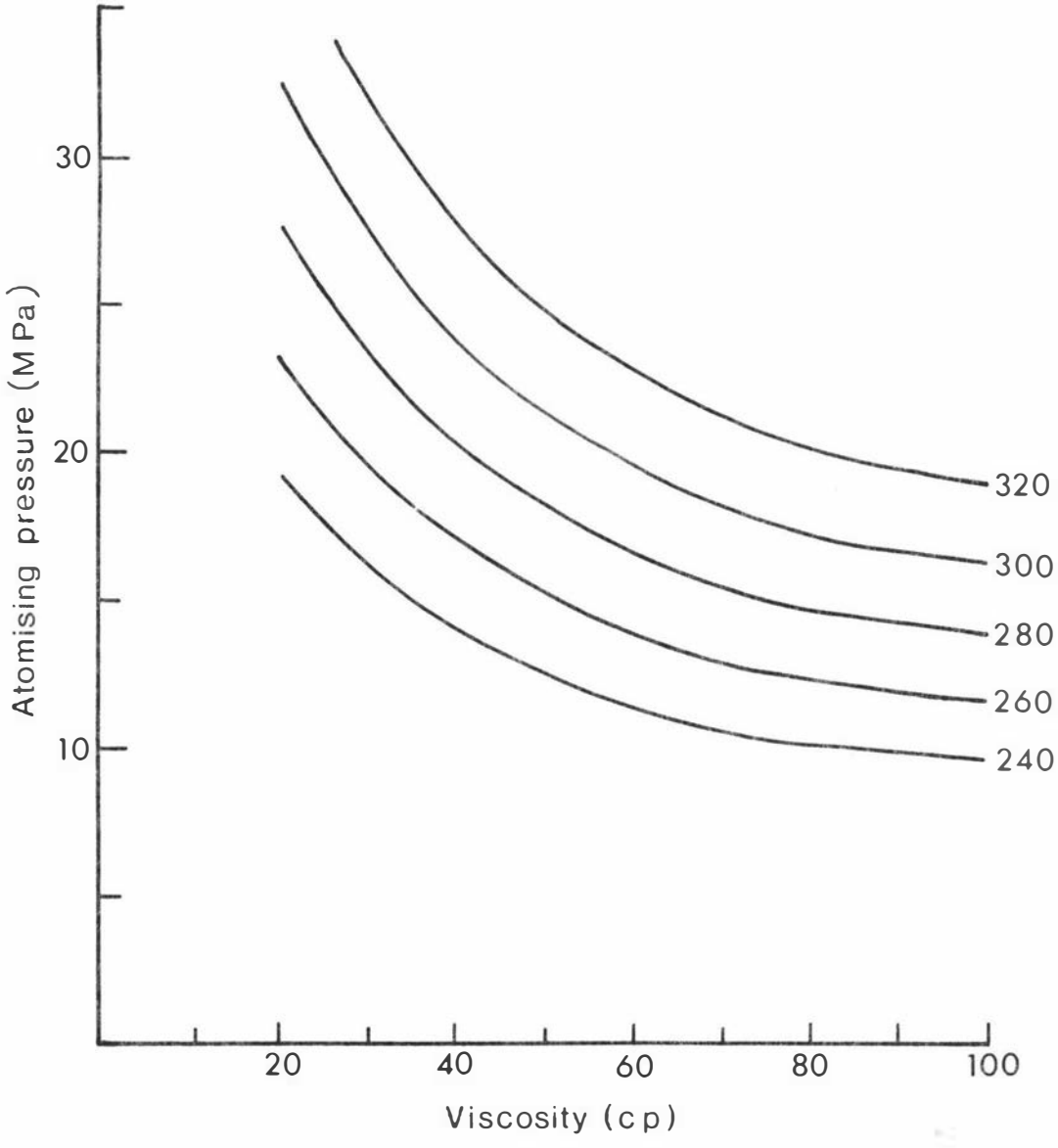


FIGURE 4.4 The effect of viscosity on the pressure-flowrate relationship for a Delevan SB 54 Nozzle

TABLE 4-2 The Effect of Nozzle Geometry on the Viscosity Sensitivity of the Nozzles

Nozzle	Diameter Ratio R	Decay Constant (poise)	Magnitude M
Spraying Systems			
Whirl Jet 2/2	5.02	0.44	3.19
SBC 8	6.70	0.58	3.64
SX 56/27	2.62	-	-
SX 69/21	3.08	3.92	1.50
Delavan			
SA 54	8.15	0.33	3.38
SAN 54	3.52	0.50	3.43
SB 54	8.15	0.29	3.24

4.1.2 Retrospective Recalibration of the Flowmeter

As described in Chapter 3, the magnetic flowmeter on the feed line to the high pressure pump developed a fault which meant that readings taken in the 1977/78 and 1978/79 dairying seasons were both in error by different amounts. The hydrodynamics of the Spraying Systems SX series nozzles make them suitable for use as flowmeters, since they are not greatly affected by viscosity changes. Only one suitable set of data was available for the 1977/78 season. This was obtained with an SX 56/27 nozzle on skim milk concentrate. The following equation was derived by using data from runs on the same nozzle with the replacement meter to predict the correct flowrates from the 1977/78 pressure measurements. The flowrates thus derived were then compared with the original measurements.

$$F(1980) = 0.7925\ F(1977/78) + 13.65$$

Two sets of sugar runs using an SX 56/27 and a water run using an SB 54 nozzle were available from the 1978/79 season. When corrections to the three flowrates used in the main seasonal experiment were made using each of these three sets of results, the flowrates agreed to within

$\pm$ 3 l/h. The mean values of the predicted flowrates weighted for the number of data points in each set were used to give the equation below.

$$F(1980) = 0.9250\ F(1978/79) - 11.30$$

These equations were used to correct all the flowrate measurements in the experiments carried out in each of the two seasons affected by the faulty meter. Confirmation that the corrections were of the right order was obtained by comparing the mean moisture contents and outlet air temperatures for the two seasons. The differences in both were consistent with the flowrates having been about 20 l/h higher during the second season, as the equations above predict.

4.2 Factors Affecting Concentrate Viscosity

The viscosity of concentrated skim milk is influenced by many factors. These include the composition of the milk, the preheat treatment it is given before evaporation, the total solids, temperature and holding time of the concentrate. Data on the effects of composition, total solids and concentrate temperature were obtained in the course of the main seasonal experiment. The effects of preheat treatment were investigated in a separate experiment.

4.2.1 Seasonal Changes in Viscosity

The following regression models were fitted to the data from each of the 30 days on which blocks of the main seasonal experiment were run.

$$\mu = 42.80 + 11.23\ TS - 1.16\ TS.T_c - 10.66\ T_c + 2.87\ T_c^2 + \text{block coefficients} \qquad r^2 = 0.9177$$

and

$$\ln \mu = 3.746 + 0.244\ TS + 0.003\ TS.T_c - 0.218\ T_c + 0.044\ T_c^2 + \text{block coefficients} \qquad r^2 = 0.9221$$

where TS = (concentrate total solids - 47.7) / 2

T_c = (concentrate temperature - 45.3) / 10

Taking the natural logarithm of the viscosity not only gave a slightly better fit, but also reduced the total solids - temperature interaction to a negligible value, so this was the model chosen for further development.

Adding the intercept to each block coefficient gives the viscosity at the mean total solids (47.7 %) and concentrate temperature (45.3 C) for each day. When these viscosities are plotted in the form of a bar chart, the rise in viscosity at the end of each season is clearly apparent. The viscosity and protein content are plotted in this way in Figure 4.5. The fat, ash, sodium, potassium, calcium, magnesium and phosphate levels in each day's milk are plotted in the same way along with the viscosities in Figure 4.6. There is a similarity between the pattern of viscosity and protein content. When the block terms were all replaced by the protein content, the following model was obtained.

$$\ln \mu = 3.765 + 0.207\ TS - 0.207\ T_C + 0.044\ T_C^2 + 0.075\ Prot$$

$$r^2 = 0.8618$$

where Prot = (protein content - 39.74)
and TS and T_C have their former meanings.

The addition of other compositional variables did not significantly improve the model. The root mean square of the residuals was taken as an estimate of the standard error of prediction near the centre of the region investigated. For the above model this was 9.4 % of the viscosity, compared with 7.0 % for the model including all the block terms. The standard error is a percentage as a consequence of taking the logarithm of the viscosity. This equation is plotted in Figure 4.7 for two protein contents. As the concentrate temperature increases, the viscosity passes through a minimum for any given total solids. At this point the viscosity increase with holding time begins to outweigh the reduction in viscosity due to the rising temperature.

4.2.2 The Effects of Preheat Treatment on Viscosity

The preheat holding time and temperature were found to have significant effects on the viscosity of the concentrate both individually

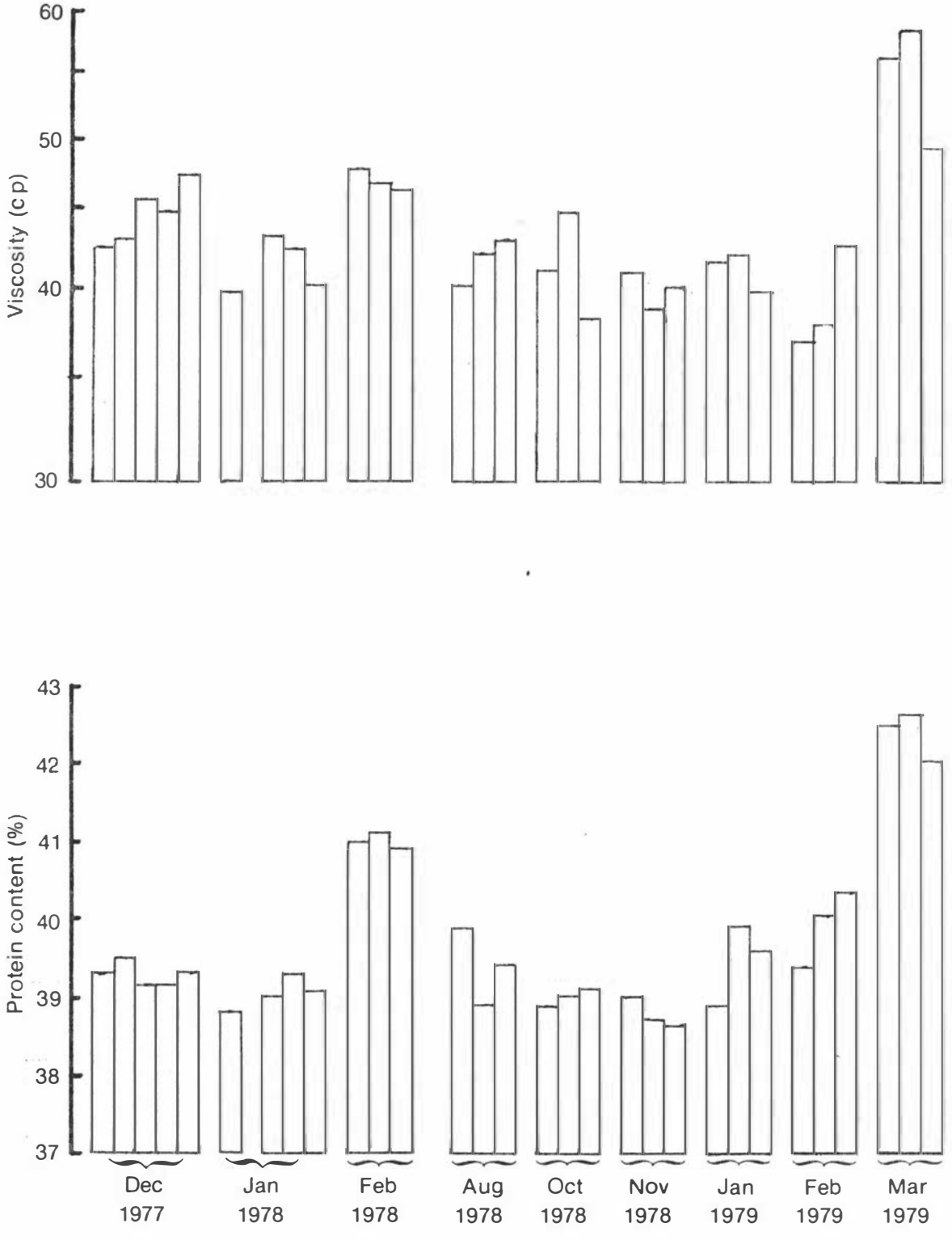


FIGURE 4.5 Bar charts of the concentrate viscosity and protein content over two dairying seasons

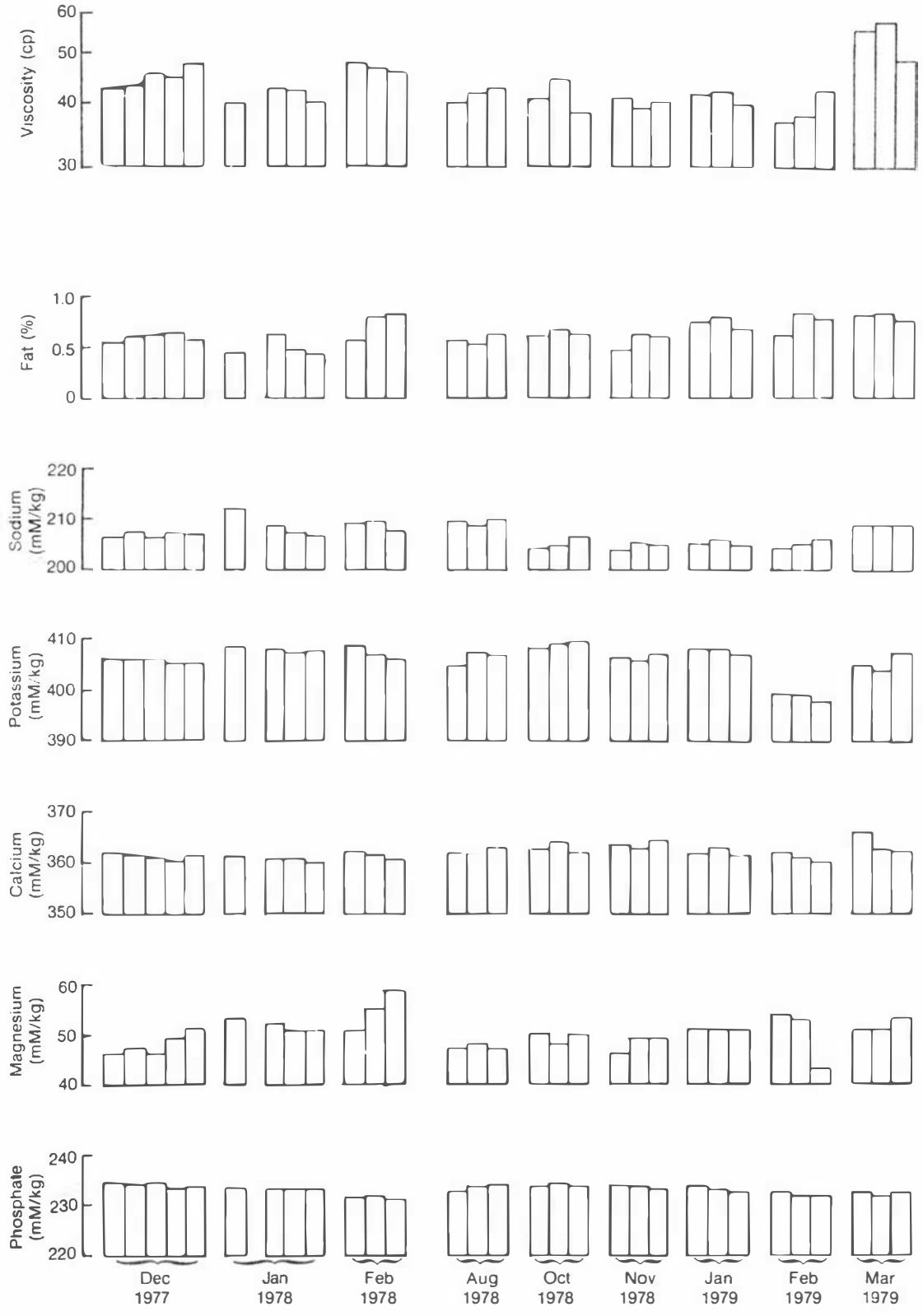


FIGURE 4.6 Bar charts of concentrate viscosity and the milk mineral content over the two seasons

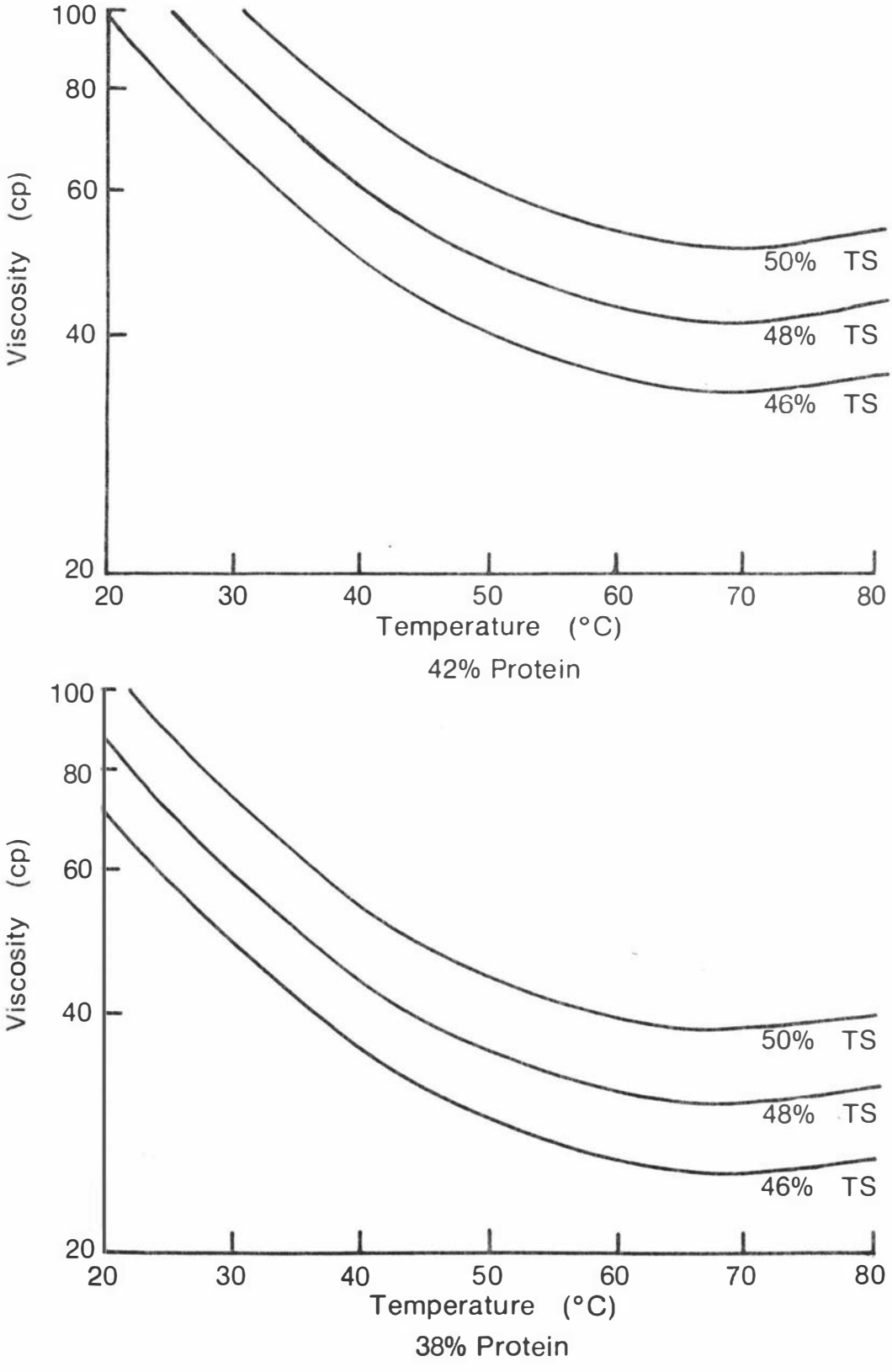


FIGURE 4.7 Graphs of concentrate viscosity against temperature for various total solids and protein contents

and in combination. The 2^4 factorial design employed also included the concentrate total solids and temperature as independent variables. A fifth variable was added by measuring the viscosity twice; immediately after the concentrate heater, 150 seconds after the concentrate left the densitometer on the discharge from the evaporator (μ_1), and again after a further 150 seconds holding in an insulated line (μ_2). The concentrate left the evaporator at 45 C and it was then maintained at this temperature or heated to 60 C. The following regression models were fitted to the data.

$$\ln \mu_1 = 3.911 + 0.066\ t_p + 0.188\ T_p + 0.202\ TS - 0.129\ T_c + 0.124\ t_p\ T_p + 0.075\ T_p\ TS$$

$$\ln \mu_2 = 4.684 + 0.205\ t_p + 0.259\ T_p + 0.176\ TS - 0.129\ T_c + 0.088\ t_p\ T_p + 0.115\ T_p\ T_c$$

where t_p = (preheat time - 65) / 55
T_p = (preheat temperature - 96.5) / 16.5
TS = (total solids - 48.25) / 0.85
T_c = (concentrate temperature - 52.5) / 7.5

Graphs of viscosity against preheat temperature for each of the two holding times are plotted in Figures 4.8 and 4.9. Each combination of levels of total solids and concentrate temperature is shown. In general, raising the preheat temperature increases the concentrate viscosity and this effect is greater at the longer preheat holding time.

Figures 4.8 and 4.9 may be compared with Figure 4.10 which shows WPNI plotted against preheat temperature over the same range of preheat conditions, plotted from the equation of Baucke and Newstead (1972) given below the graph. There is an obvious inverse relationship between the responses of viscosity and WPNI to preheat treatment. A specification requiring a low WPNI will inevitably give rise to a relatively high concentrate viscosity although the preheat temperature and holding time may still be chosen to minimise this viscosity.

The rate of increase in viscosity with concentrate holding time was investigated by taking the ratio of the viscosity after 300 seconds

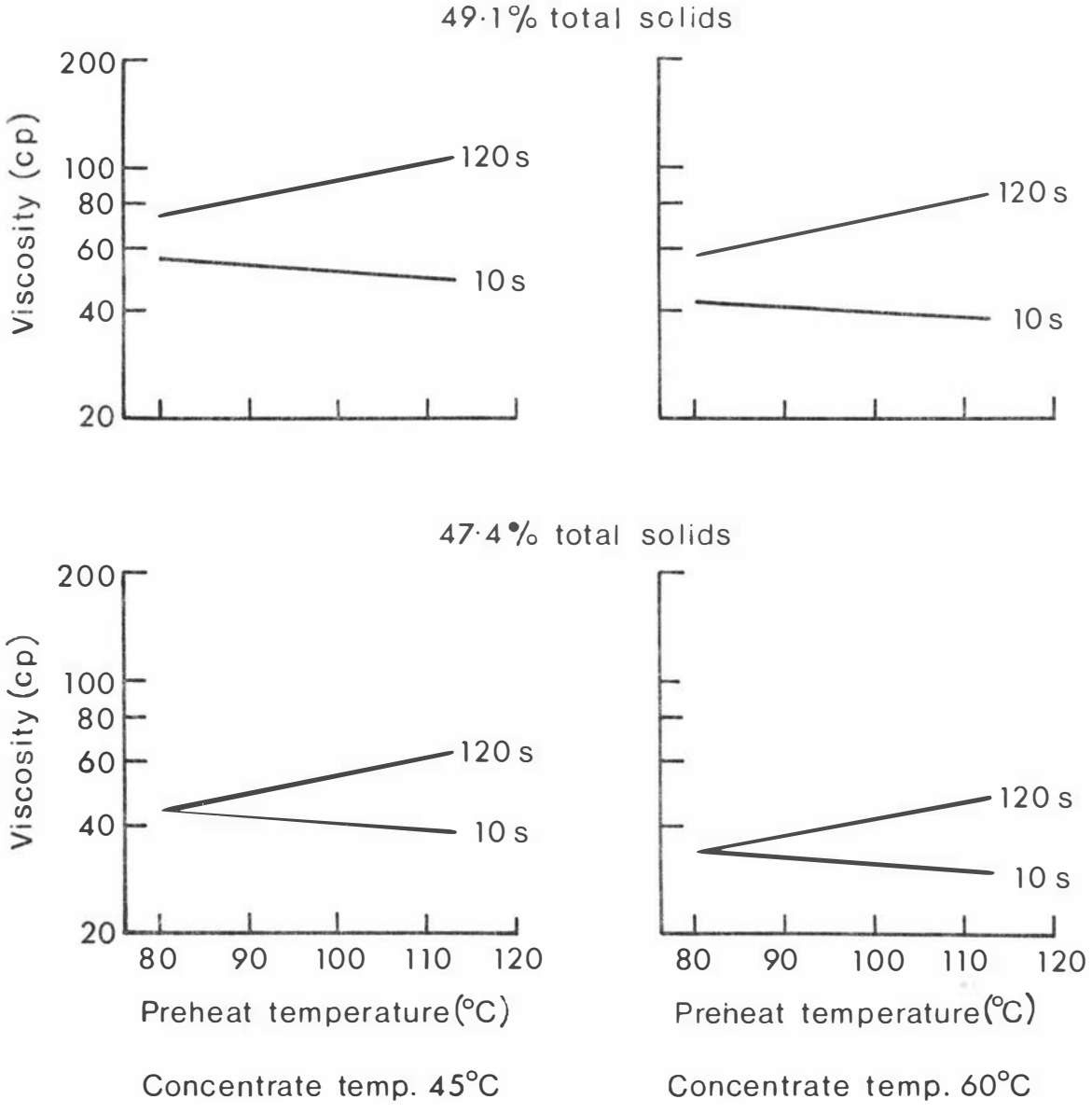


FIGURE 4.8 Graphs illustrating the effect of preheat conditions on the viscosity of concentrate measured immediately after the concentrate heater

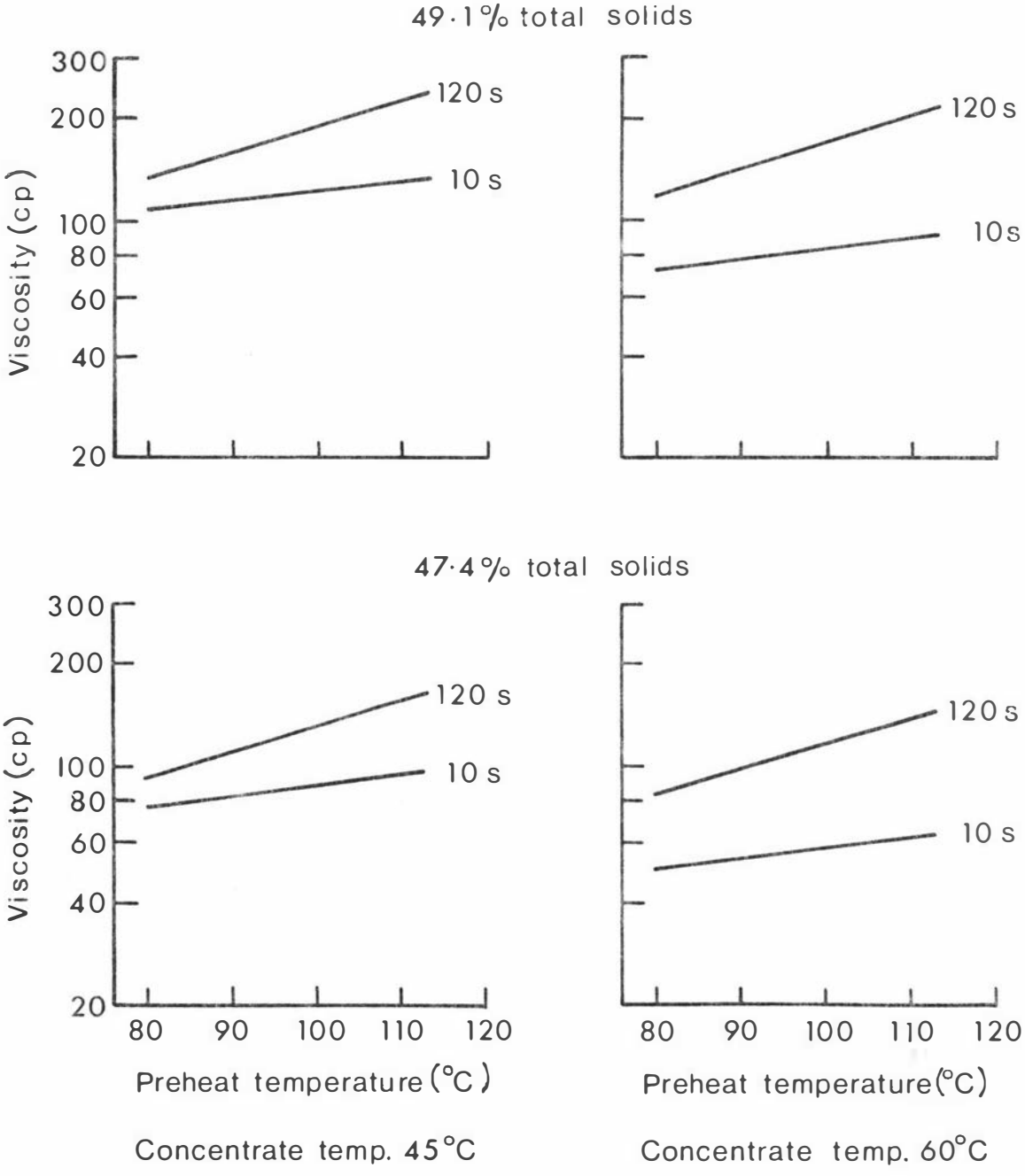
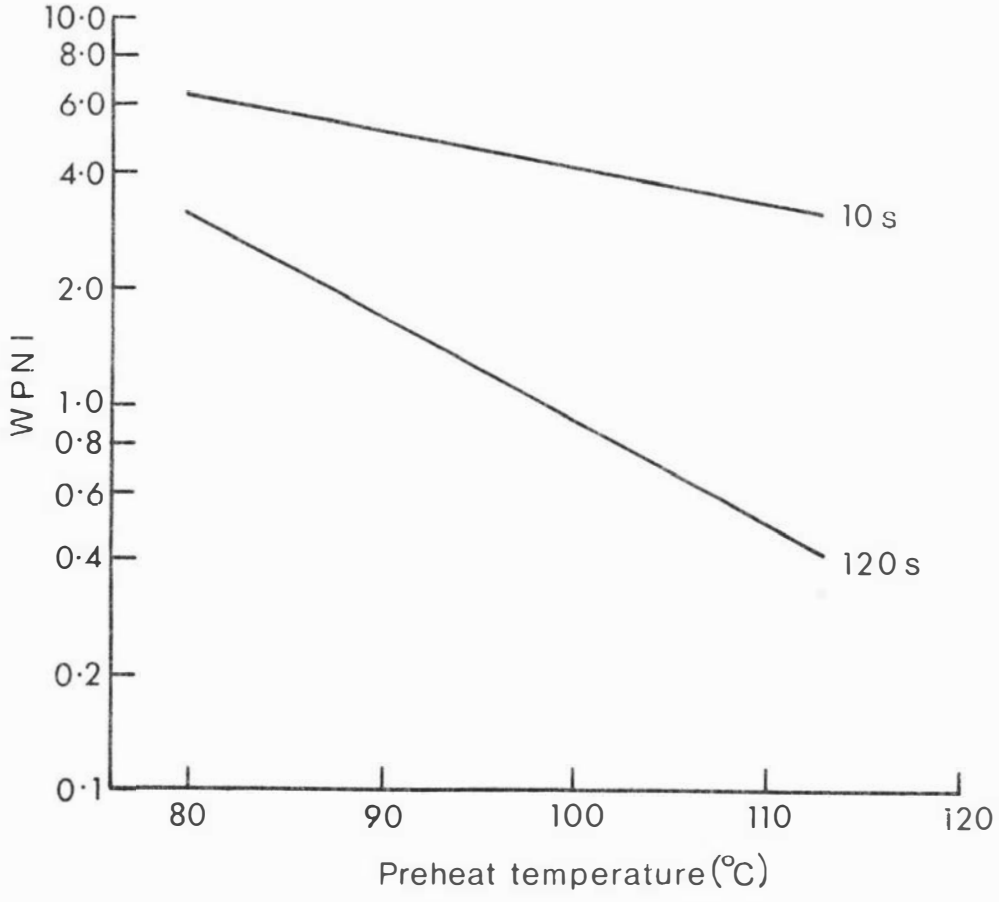


FIGURE 4.9 Graphs illustrating the effect of preheat conditions on the viscosity of concentrate measured 150 s after the concentrate heater



$$\text{Ln WPNI} = 12.56 - 1.902\sqrt{t_p}\,\text{Ln}\,T_p + 11.07\sqrt{t_p} - 1.782\,\text{Ln}\,T_p$$

Where t_p = holding time (s)

T_p = preheat temperature (K)

FIGURE 4.10 Graph illustrating the effect of preheat conditions on WPNI

holding at 45 C to the viscosity 150 seconds after the concentrate left the evaporator. Table 4-3 gives the mean values of this ratio for each level of preheat holding time and temperature, concentrate total solids and their combinations.

It is clear that the rate of viscosity increase for a given WPNI may be minimised by using a high temperature, short time (HTST) preheat treatment combination.

TABLE 4-3 The Effects of Preheat Conditions and Concentrate Total Solids on the Rate of Viscosity Increase at 45 C

Holding Time (s)		Temperature (C)		Total Solids (%)	
10	120	80	113	47.4	49.1
2.01	2.41	2.28	2.11	2.19	2.23

		Time (s)	
		10	120
Temp. (C)	80	2.04	2.52
	113	1.97	2.26

		Time (s)	
		10	120
TS (%)	47.4	2.10	2.34
	49.1	1.89	2.46

		Temp. (C)	
		80	113
TS (%)	47.4	2.20	2.18
	49.1	2.36	2.05

4.3 - The Effects of some Drier Design Variables

The drier design variables investigated were the diameter of the air inlet throat, the vertical position of the nozzle relative to the drier roof, and the nozzle swirl chamber and orifice sizes. Three separate experiments were conducted. The effects of all the variables on the powder bulk density measured at 0, 10, 100 and 1000 taps on the stamp volumeter were similar, with the value of the density increasing with the number of taps, so only the 100 taps bulk density has been reported in presenting the results of the factorial experiments.

4.3.1 Throat Diameter and Nozzle Position

The effects on the powder properties of changing the drier air inlet throat diameter and nozzle position were measured with the operating variables held constant. The concentrate total solids for the first nine runs averaged 49.6 %, but the average for the last three runs was only 47.4 %. Since replicate 11 of the main seasonal experiment had been run the previous week, it was possible to correct the responses for the 2.2 % drop in total solids using the reduced regression models for that replicate. Table 4-4 gives the original and corected responses.

TABLE 4-4 Corrected Responses for the Throat Diameter - Nozzle Position Experiment

Run No.		M (%)	SI (ml)	Bulk Densities 0	10	100	1000	ρ_p	D_{sv} (μm)	σ_g	T_o (C)
10	orig.	4.13	0.30	0.61	0.62	0.72	0.80	1.27	45	2.29	92.1
	corr.	3.72	0.40	0.62	0.63	0.74	0.82	1.30	39	2.29	95.1
11	orig.	4.41	0.20	0.60	0.62	0.72	0.80	1.30	52	2.17	92.3
	corr.	4.00	0.30	0.61	0.63	0.74	0.82	1.33	46	2.17	95.3
12	orig.	4.47	0.30	0.60	0.61	0.70	0.80	1.29	49	2.25	92.7
	corr.	4.06	0.40	0.61	0.62	0.72	0.82	1.32	43	2.25	95.7

An analysis of variance on the corrected data showed that the throat diameter, nozzle position and their interaction had no effect significant at the 10 % confidence level on any of the responses except bulk density. Table 4-5 gives the effects of these two variables on the 100 taps bulk density. The effect of the throat diameter was significant at the 0.1 % level, but the throat diameter-nozzle position interaction was not significant. It is evident that the bulk density increased when the throat diameter was reduced. Because the air flowrate through the drier was kept constant, the air inlet velocity increased by a factor of 2.26 when the smaller diameter throat was installed. This promotes more vigorous mixing of the spray and the air.

TABLE 4-5 The Effects of Throat Diameter and Nozzle Position on the Powder Bulk Density

	Throat Diameter		Nozzle Position		
	203 mm	305 mm	-80 mm	0	+80 mm
Bulk Density (g/ml) (100 taps)	0.72	0.64	0.68	0.68	0.67

4.3.2 Nozzle Orifice Size, Swirl Chamber and Drier Feedrate

A replicated 2^3 factorial experiment in orifice size, swirl chamber and concentrate flowrate was carried out. The concentrate viscosity was adjusted to keep the atomising pressure constant as the nozzles were changed. The pressure was allowed to increase with the flowrate, however. The air inlet temperature and concentrate total solids were kept constant at 210 C and 48.4 % respectively, values near the centre of the region explored by the main seasonal experiment. The mean responses at each level of the independent variables are given in Table 4-6, together with their significance levels. A significant interaction between the effects of nozzle orifice size and swirl chamber on both the bulk density and particle density of the powder was observed. This is illustrated in Table 4-7.

TABLE 4-6 The Effects of Nozzle Orifice Size and Swirl Chamber and Drier Feedrate

Responses and covariates	Orifice		Swirl Chamber		Feedrate	
	54	61	SA	SB	268	287
Moisture (%)	4.48	4.14	4.69	3.93	4.31	
Solubility Index (ml)	0.40		0.40		0.53	0.26
Bulk Density (100 taps, g/ml)	0.65	0.61	0.65	0.61	0.63	
Particle Density (g/ml)	1.24		1.24		1.24	
Particle Size D_{sv} (μm)	57	54	57	53	59	52
σ_g	2.20	2.04	2.29	1.95	2.22	2.02
Outlet Air Temperature (C)	90.4	92.0	89.8	92.6	92.6	89.8
Concentrate Viscosity (cp)	91.4	63.9	103.2	52.1	81.8	73.5
Concentrate Temperature (C)	27.0	36.2	22.9	40.3	30.8	32.4
Atomising Pressure (MPa)	21.5	20.9	21.6	20.8	19.2	23.2

TABLE 4-7 The Interaction Between Nozzle Orifice Size and Swirl Chamber

Bulk Density 100 taps (g/ml)		Orifice 54	Orifice 61
Swirl Chamber	SA	0.66	0.65
	SB	0.65	0.58

Particle Density (g/ml)		Orifice 54	Orifice 61
Swirl Chamber	SA	1.23	1.25
	SB	1.26	1.22

4.3.3 Inlet Temperature, Nozzle Orifice Size and Viscosity

A 2^3 experiment was carried out to investigate the magnitude of any interactions between the air inlet temperature, the nozzle orifice size and the concentrate viscosity. Spraying Systems SX series nozzles with a number 21 core and two different orifice sizes were used because at constant feedrate, the atomising pressure had been found to be almost independent of concentrate viscosity for these nozzles. The atomising

pressure was maintained at a constant 22 MPa, so the feedrate was confounded with the nozzle size, and to a much smaller extent, with the viscosity. The concentrate total solids was constant and the viscosity was varied by changing the concentrate temperature. Table 4-8 gives the mean values of the response variables and covariates at each level of the independent variables. The significant interactions are presented in Table 4-9.

TABLE 4-8 The Effects of Inlet Air Temperature, Nozzle Orifice Size and Concentrate Viscosity

Responses and Covariates	Inlet air Temperature (C)		Orifice size		Concentrate Viscosity (cp)	
	195	225	52	50	30	80
Moisture (%)	6.58	4.15	5.05	5.69	4.80	5.94
Solubility Index (ml)	0.31	1.48	0.90		0.86	0.93
Bulk Density (100 taps, g/ml)	0.76	0.60	0.66	0.70	0.66	0.70
Particle Density (g/ml)	1.33	1.28	1.31		1.31	
Particle Size D_{sv} (μm)	55	59	55	59	55	59
σ_g	2.39	2.15	2.45	2.29	2.19	2.35
Outlet Air Temperature (C)	82.8	97.7	91.1	89.4	91.1	89.3
Concentrate Feedrate (l/h)	299.0	299.6	292.2	306.4	301.3	297.3
Concentrate Temperature (C)	33.8	34.0	33.9	33.9	43.8	24.0

TABLE 4-9 Mean Values of the Responses for Various Combinations of the Independent Variables Showing the Interactions

Moisture (%)		Temperature 195	Temperature 225
Viscosity (cp)	30	5.90	3.70
	80	7.27	4.61

Moisture (%)		Orifice 52	Orifice 50
Viscosity (cp)	30	4.37	5.23
	80	5.73	6.15

TABLE 4-9 Continued

Bulk Density 100 taps (g/ml)		Temperature 195	225
Viscosity (cp)	30	0.74	0.57
	80	0.78	0.63

Bulk Density 100 taps (g/ml)		Orifice 52	50
Viscosity (cp)	30	0.63	0.68
	80	0.65	0.71

Particle Density (g/ml)		Temperature 195	225
Orifice	52	1.30	1.28
	50	1.36	1.28

Particle Density (g/ml)		Orifice 52	50
Viscosity (cp)	30	1.29	1.34
	80	1.29	1.29

Particle Size D_{sv} (μm)		Temperature 195	225
Orifice	52	51	59
	50	60	59

Particle Size σ_g		Orifice 52	50
Viscosity (cp)	30	2.07	2.32
	80	2.43	2.27

Outlet Air Temperature (C)		Orifice 52	50
Viscosity	30	92.7	89.6
	80	89.5	89.2

4.4 - The Effects of the Drier Operating Variables

Analysis of the results of the 3^{4-1} experiment to determine the effects of the drier operating variables was complicated by the presence of a covariate, the concentrate temperature. This temperature was varied to obtain the desired combinations of concentrate flowrate and atomising pressure. It was possible to do this because of the effect of temperature on the concentrate viscosity, and the effect of the viscosity on the pressure-flowrate relationship of the nozzle used. As has already been shown, however, the protein content of the milk also influences the concentrate viscosity, so the average concentrate temperature required varied from day to day.

The results were analysed in two stages. First, regression models for each response variable were fitted. These had coefficients for each of the independent variables their two-way interactions and squares, and for the concentrate temperature and its square. The second stage was to obtain an equation relating the concentrate temperature to the concentrate total solids, flowrate, atomising pressure and protein content. This equation was then used in calculating the responses at the average protein content for the two seasons.

Graphs illustrating the effects of the independent variables at the average milk protein content on each of the responses will now be given. The variables have been scaled so as to range from -1 to +1. The scaling equations are given below.

$$T = (T - 210) / 15 \quad C$$

$$TS = (TS - 47.7) / 2 \quad \%$$

$$F = (F - 279) / 20 \quad l/h$$

$$P = (P - 24) / 4 \quad MPa$$

$$T_C = (T_C - 45.3) / 10 \quad C$$

The coefficient of determination r^2 and the root mean square (rms) residual are given as indications of the accuracy of prediction using the models, assuming that the concentrate temperature is measured, and not calculated.

Each illustration shows contours of constant response variable on graphs of inlet air temperature against concentrate feedrate. These graphs are drawn for three total solids and three atomising pressures.

There are only four degrees of freedom in the system, however, so that in order to change the total solids without changing the feedrate or atomising pressure it is necessary to adjust the concentrate temperature to maintain a constant viscosity. The total solids effect is therefore confounded with that of concentrate temperature. Similarly, any change in atomising pressure at constant flowrate and total solids must be accompanied by a change in concentrate temperature.

4.4.1 Moisture

The equation for the powder moisture content is:

$$M = 3.498 - 0.839\ T + 0.095\ T^2 + 0.466\ F - 0.294\ P + 0.068\ T.TS - 0.105\ T.F + 0.092\ T.P - 0.142\ TS.F + 0.038\ F^2 - 0.132\ F.P - 0.398\ T_C + 0.050\ T_C^2$$

$r^2 = 0.9481$ rms residual = 0.21 % moisture

This relationship is illustrated in Figure 4.11. The effect of increasing the inlet temperature is to reduce the moisture content, and decreasing the concentrate feedrate has the same effect. Increasing the atomising pressure also reduces the moisture content of the powder. Increasing the total solids of the concentrate causes a small decrease in the moisture.

4.4.2 Solubility Index

The equation for the Solubility Index of the powder is not as satisfactory as those for most of the other powder properties. One of the reasons for this is that the mixer used to disperse the powder was found to be operating at only 80 % of the required number of revolutions per minute shortly after the start of the second season of the experimental work. Upon investigation it was evident that the tacho-

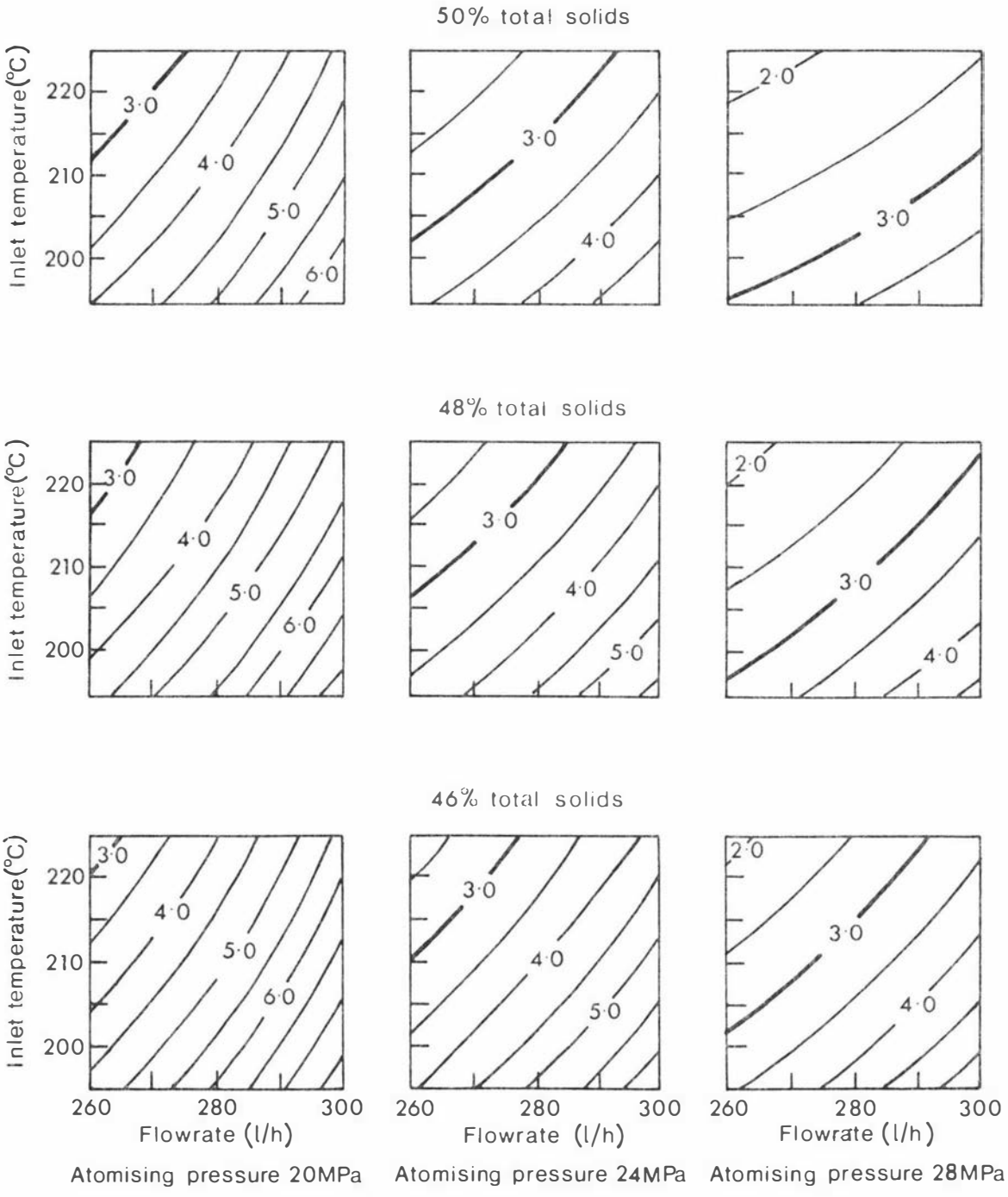


FIGURE 4.11 Moisture contours

meter had been progressively indicating higher over an unknown period. Tests conducted using a range of mixer speeds showed that the SI rose as the mixer speed was reduced. The effect of the fault in the tachometer was to make many of the SI values for the first season too high by an amount which could not be estimated with any confidence. No correction was therefore applied, and the figures were used as they came from the laboratory. The equation is given below and the relationship is illustrated in Figure 4.12.

$$SI = 0.1 \exp (1.064 + 1.289\ T + 0.299\ TS - 0.461\ F - 0.382\ P + 0.098\ P^2 - 0.095\ T_C)$$

$r^2 = 0.7316$ rms residual = 80 % of SI value

The inlet air temperature clearly has the dominant effect on the SI, with higher temperatures giving higher SI values. Higher concentrate flowrates and atomising pressures both reduce the SI, while increasing the concentrate total solids causes only a very small increase. An increase in total solids requires an increase in concentrate temperature if the viscosity is to be held constant. The above equation indicates that the effect of concentrate total solids and temperature are opposite in sign, giving rise to the small residual effect shown in Figure 4.12.

4.4.3 Bulk Density

The models for bulk density measured at 0, 10, 100 and 1000 taps on a stamp volumeter are given below. Only the model for 100 taps is illustrated in Figure 4.13, as the models all have similar forms, and the simulation model uses this density.

$$BD\ (0) = 0.512 - 0.085\ T + 0.003\ TS + 0.018\ F - 0.015\ T.TS + 0.017\ T.F - 0.006\ T.P + 0.032\ T^2 + 0.005\ P^2 - 0.036\ T_C + 0.005\ T_C^2$$

$r^2 = 0.9438$ rms residual = 0.017 g/ml

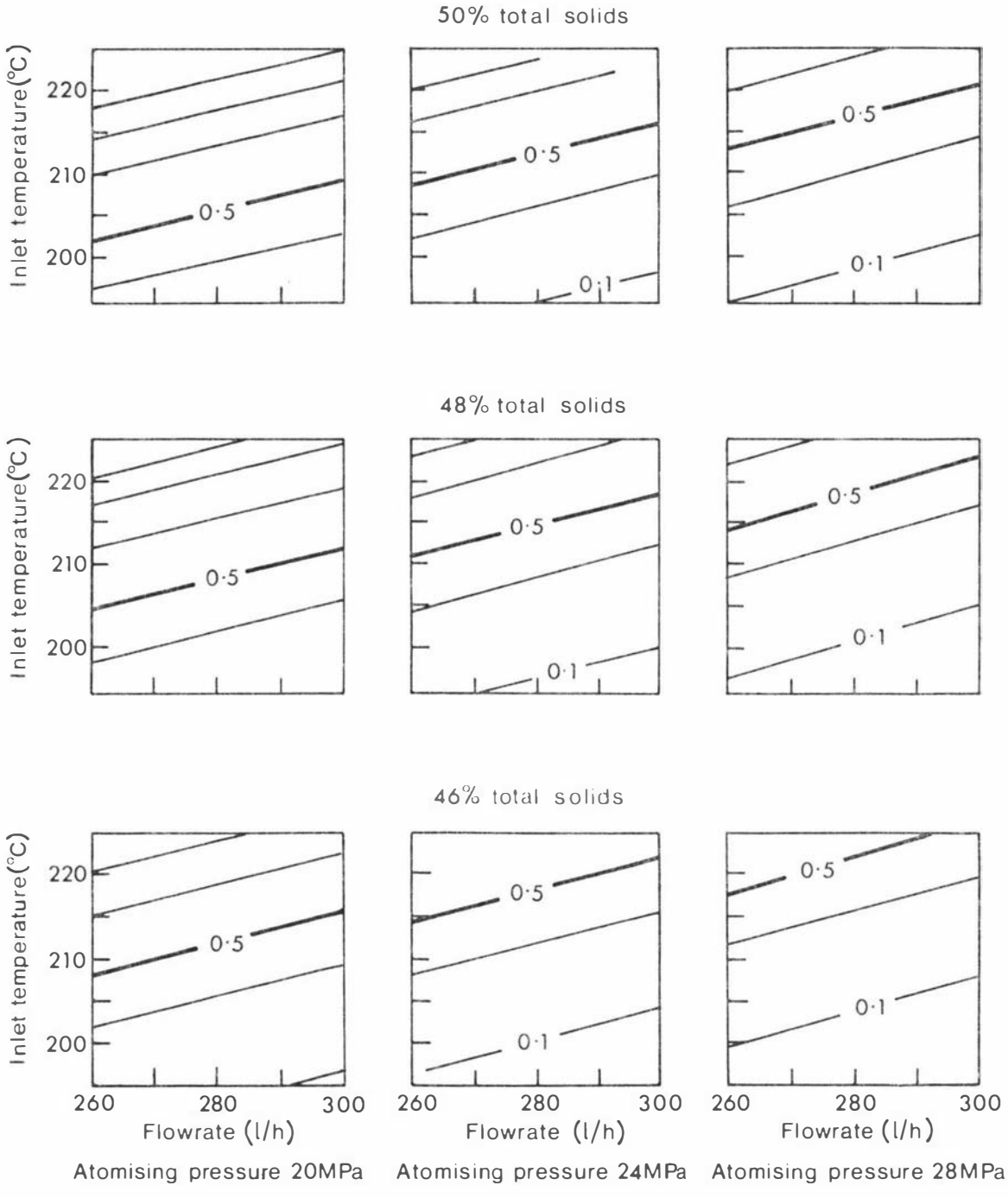


FIGURE 4.12 Solubility Index contours

$$BD(10) = 0.526 - 0.009\,T + 0.004\,TS + 0.019\,F + 0.006\,P - 0.016\,T.TS + 0.018\,T.F - 0.006\,T.P + 0.004\,TS.P - 0.004\,F.P - 0.015\,T^2 - 0.032\,T_C + 0.002\,T_C^2$$

$r^2 = 0.9503$ rms residual = 0.016 g/ml

$$BD(100) = 0.600 - 0.098\,T + 0.009\,TS + 0.019\,F + 0.005\,P - 0.018\,T.TS + 0.021\,T.F - 0.008\,T.P - 0.006\,F.P - 0.016\,T^2 - 0.041\,T_C + 0.004\,T_C^2$$

$r^2 = 0.9511$ rms residual = 0.019 g/ml

$$BD(1000) = 0.711 - 0.104\,T - 0.004\,TS + 0.013\,P - 0.020\,T.TS + 0.021\,T.F - 0.009\,T.P - 0.006\,F.P - 0.020\,T^2 + 0.007\,P^2 - 0.032\,T_C^2$$

$r^2 = 0.9515$ rms residual = 0.019 g/ml

The inlet air temperature has the greatest effect on the bulk density of the powder, with higher temperatures giving rise to lower bulk densities. The concentrate flowrate also has a strong infuence. High flowrates increase the bulk density. The effect of increasing the atomising pressure is generally to reduce the bulk density, but it also increases the effect of the inlet temperature and reduces that of the flowrate.

4.4.4 Particle Density

The following model was fitted to the particle density measured with an air pycnometer.

$$\rho_p = 1.227 - 0.066\,T + 0.005\,TS + 0.017\,F + 0.005\,P - 0.010\,T.TS + 0.018\,T.F - 0.031\,T.P - 0.006\,F^2 - 0.028\,T_C$$

$r^2 = 0.7939$ rms residual = 0.029 g/ml

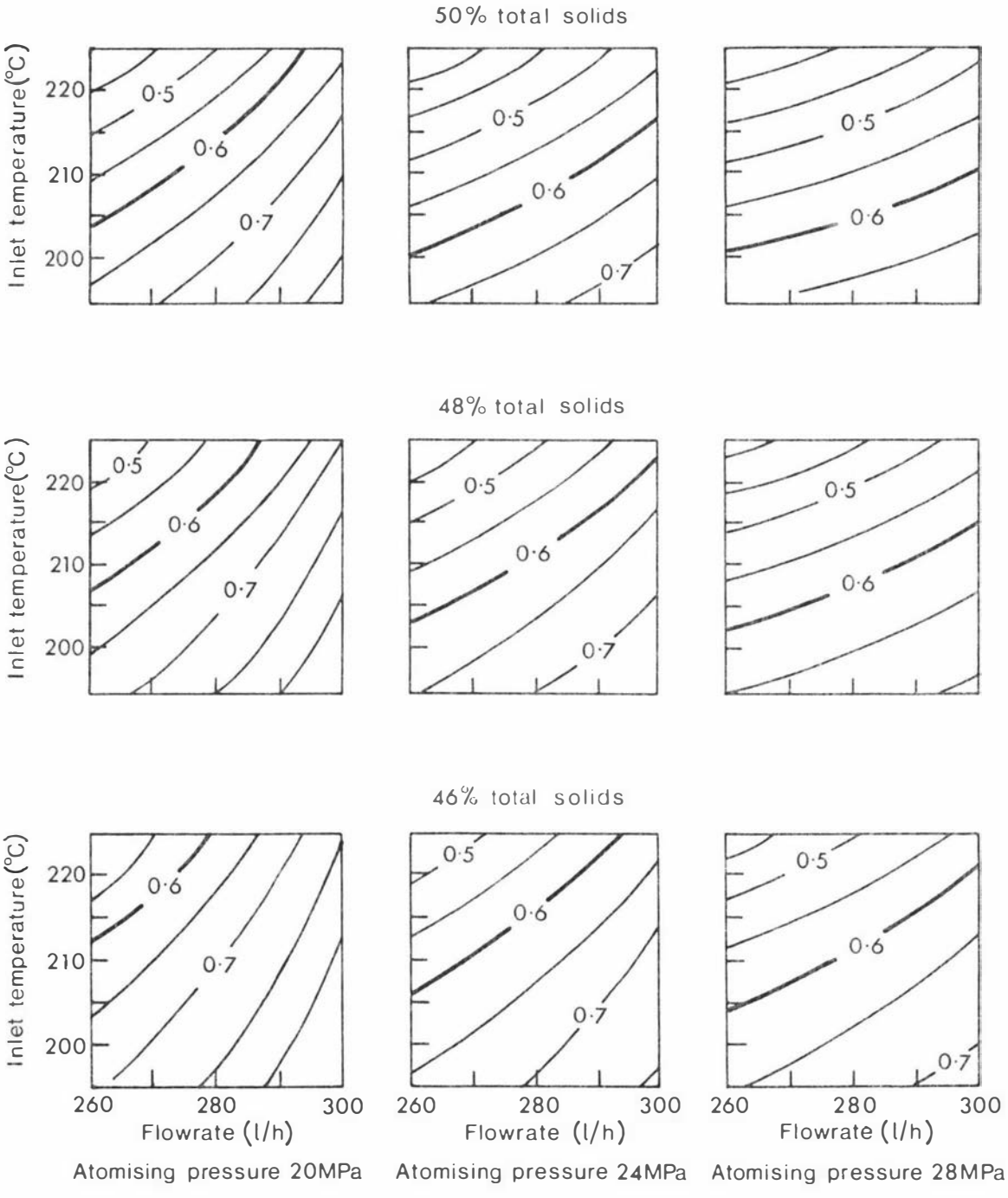


FIGURE 4.13 Bulk Density (100 taps) contours

This relationship is illustrated in Figure 4.14. There is an obvious similarity between the contours for particle density and those for the bulk density in Figure 4.13.

4.4.5 Particle Size Distribution

The results of the statistical analysis of the particle size distribution data were disappointing in that only low coefficients of determination were obtained when the data from all the replicates were analysed together. There were two response variables describing the particle size distribution, the surface-volume mean particle diameter D_{sv} and the standard deviation of the distribution, σ_g . Problems were experienced with the Andreasen pipette size analysis method. The results varied between technicians. At first, each block of the experiment was assigned to one technician only so that inter-technician variance could be accounted for, however this system broke down under the stress of large numbers of samples. The major source of error in measuring D_{sv} was the determination of the density of the powder in isopropyl alcohol. This did not affect the value of σ_g, however, as Appendix V shows.

The models for all ten replicates are given below.

$$D_{sv} = 42.7 + 6.6\ T + 2.0\ TS + 3.5\ T.TS - 4.0\ T.F + 3.8\ T^2 - 6.0\ P^2 + 2.0\ T_c$$

$r^2 = 0.4034$ rms residual = 8.6 μm

$$\sigma_g = 1.989 + 0.064\ TS + 0.110\ F - 0.199\ P + 0.057\ T.P - 0.050\ F.P - 0.046\ T_c$$

$r^2 = 0.4455$ rms residual = 0.21

The results graphed in Figure 4.15 are from replicate three of the experiment, all samples having been analysed by one technician. The equations fitted to these data are given below. The D_{sv} model is clearly an improvement, but that for σ_g is still not particularly satisfactory.

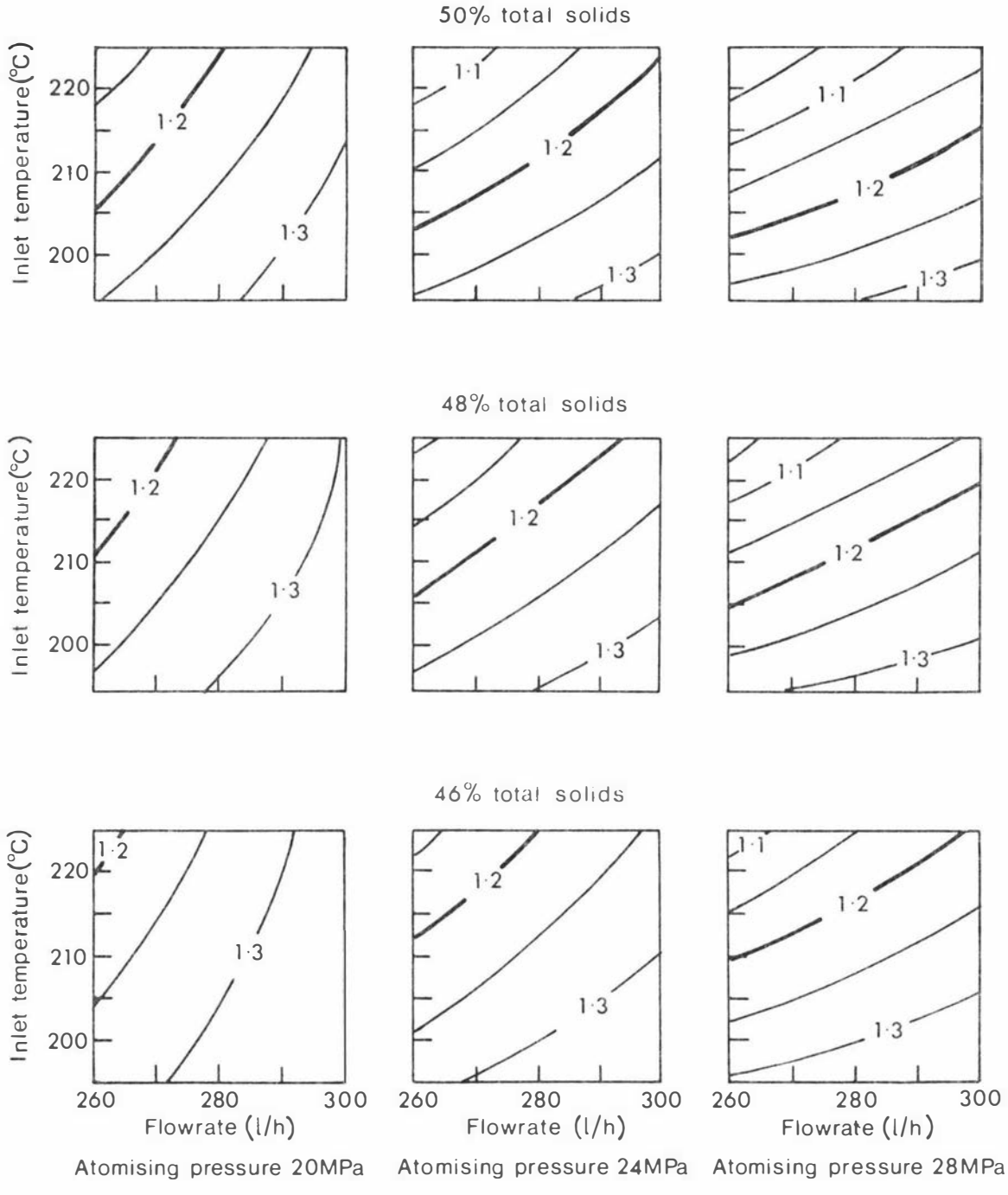


FIGURE 4.14 Particle density contours

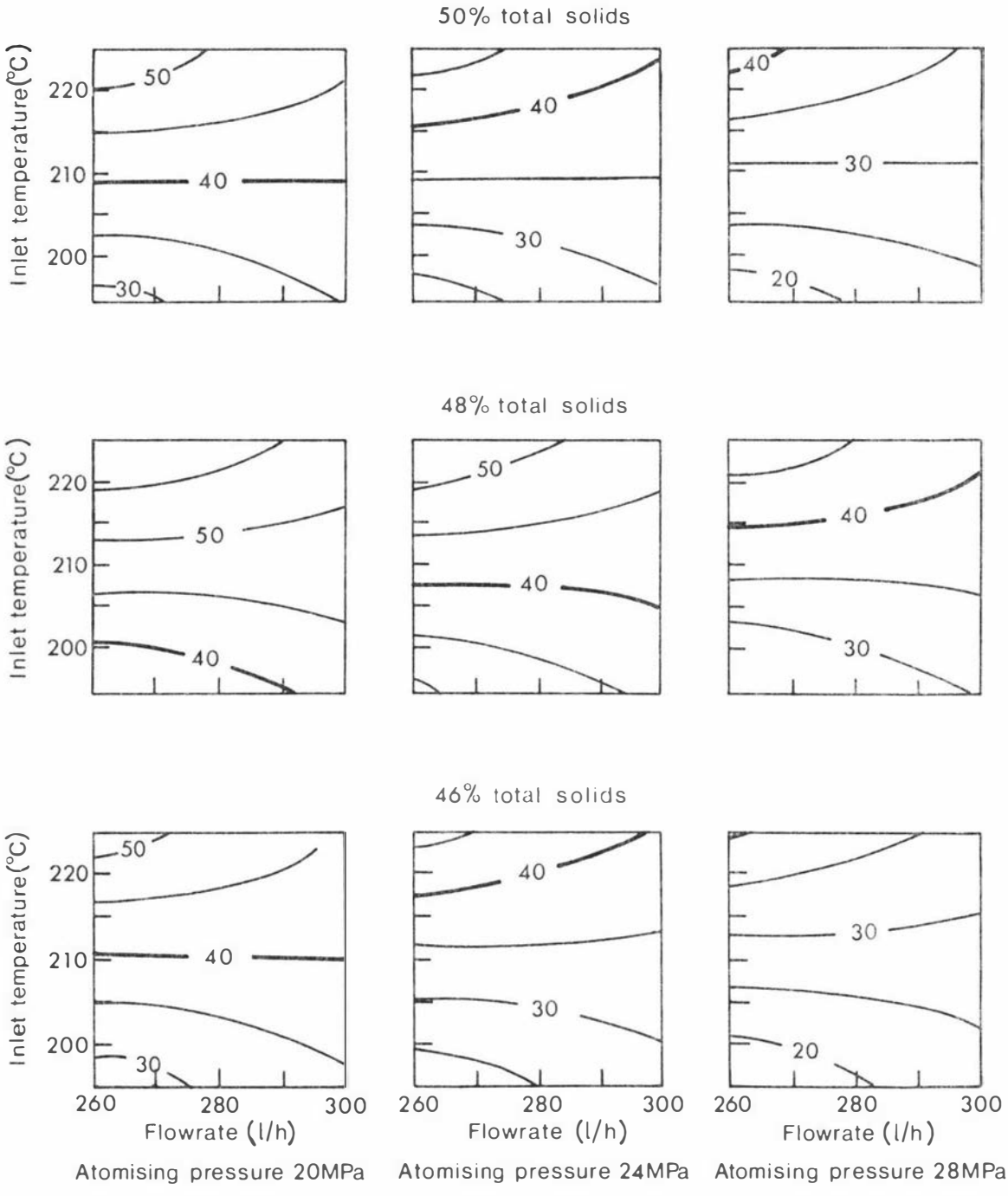


FIGURE 4.15 Mean particle size (D_{sv}) contours

$$D_{sv} = 41.5 + 8.846\ T + 2.680\ TS - 5.714\ P - 3.253\ T.F - 3.264\ TS.F + 8.738\ T^2 - 7.181\ TS^2$$

$r^2 = 0.9230$ rms residual = 2.6 μm

$$\sigma_g = 1.806 - 0.193\ P + 0.081\ P^2 + 0.157\ TS^2 + 0.090\ F$$

$r^2 = 0.6979$ rms residual = 0.11

The higher the inlet air temperature, the larger the mean particle size. The higher the atomising pressure, the smaller the size. The inlet temperature does not affect σ_g.

4.4.6 Outlet Air Temperature

The outlet air temperatures plotted in Figure 4.16 are generally similar in form to the moisture contours in Figure 4.11, except for the effect of atomising pressure. Increased atomising pressures give markedly lower powder moistures, yet give only slightly elevated outlet temperatures. This is a reflection of the smaller particle size, and hence increased surface area available for evaporation, caused by the higher pressure. The regression equation is given below.

$$T_o = 93.09 + 7.21\ T + 0.83\ TS - 2.54\ F - 0.82\ P + 0.34\ F.P - 0.25\ T^2 + 2.25\ T_c - 0.18\ T_c^2$$

$r^2 = 0.9584$ rms residual = 1.26 C

4.4.7 The Operating Envelope

Some idea of the relative ease of manufacturing powder meeting a given specification may be gained by overlaying two or more of the contour graphs just presented. For example, suppose that a specification calls for a moisture content not greater than 4.0 % and a Solubility Index not greater than 0.5 ml. Figure 4.17 shows the area in

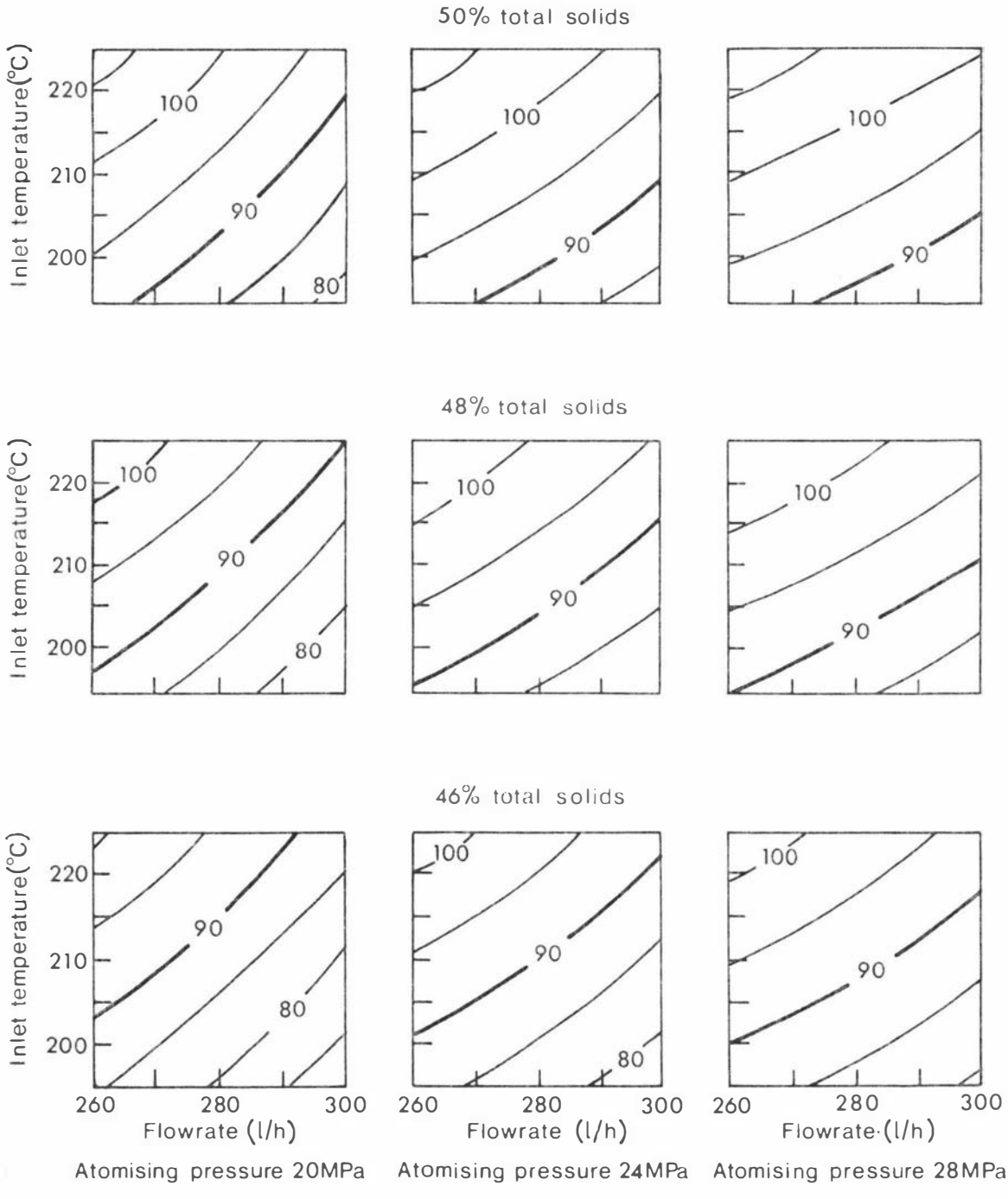


FIGURE 4.16 Outlet air temperature contours

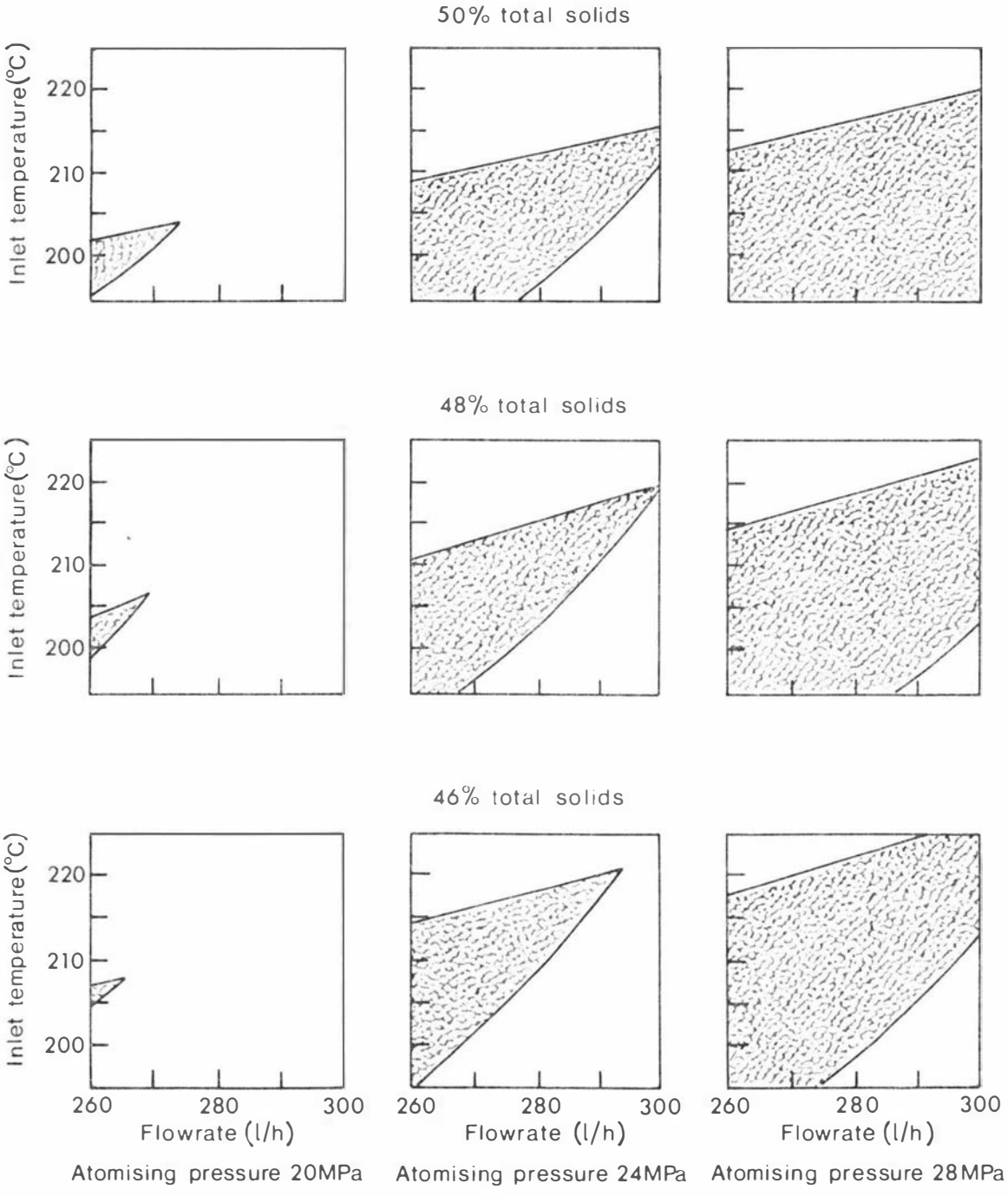


FIGURE 4.17 Graphs showing the area of drier operation satisfying the requirement that: M ⩽ 4.0 % and SI ⩽ 0.5 ml

the operating variable space which satisfies these requirements. There is obviously much more latitude in the selection of the operating variables at high atomising pressures.

CHAPTER 5 - THE DRIER MODEL

The regression equations describing the characteristics of the skim milk concentrate and the spray drier have been combined to form a computer simulation model of the drying process. This model may be used to simulate a wide variety of drier operating modes and incorporates provision for introducing changes in milk composition and preheat treatment. The following section examines the structure of this model while the second section describes the simulation program.

5.1 - The Model Structure

A structural array similar to that in Table 3-3 was used to determine the order in which the regression equations must be solved to give the unknown powder properties resulting from any given set of input variables. This order depends on which of the inputs are fixed, which are manipulated and which are dependent on the inputs already specified. Some of the possible options are given in Table 5-1. The variable symbols are those of Table 3-1. In both cases the following variables are assumed to have been fixed:

- milk composition
- preheat temperature
- preheat holding time
- drier air flowrate
- the number of nozzles
- the nozzle orifice size
- the nozzle swirl chamber or core

The options marked with an asterisk (*) are used in nozzle atomising spray driers. Regardless of which option is chosen, five operating variables must be specified before the product quality variables can be calculated. There are three different sets of dependent variables listed in Table 5-1, so the simulation model should have provision for the following calculations.

TABLE 5-1 Options for Operating Spray Driers

Option No.	Degrees of Freedom	Fixed Variables	Manipulated Variables	Dependent Variables
1 *	4	-	T, TS, F, P	μ, T_c
2	4	-	T, TS, F, T_c	μ, P
3	4	-	T, TS, P, T_c	μ, F
4 *	3	T_c	T, TS, F	μ, P
5 *	3	T_c	T, TS, P	μ, F
6	3	T	TS, P, T_c	μ, F
7	2	T, T_c	TS, F	μ, P
8 *	2	T, T_c	TS, P	μ, F

Viscosity sensitive nozzles

(1) μ from F, P and then T_c from μ, TS

(2) μ from TS, T_c and then P from μ, F

(3) μ from TS, T_c and then F from μ, P

Viscosity insensitive nozzles

(4) P from F

(5) F from P

The concentrate viscosity is an intermediate variable in the calculations and may be calculated from the concentrate total solids and temperature in the last two cases even though it is not required. This simplifies the programming, as then only the first three cases need be considered.

The simulation model also incorporates two outlet air temperature controllers. One manipulates the inlet air temperature and the other manipulates the concentrate feedrate or atomising pressure.

The structure of the simulation program is illustrated in Figure 5.1 by means of a flowchart.

5.2 - The Simulation Program

Each of the modules shown in Figure 5.1 will now be considered in detail. Some of the regression models given in Chapter 4 are used directly, but some further analysis was required before all the necessary equations could be derived.

5.2.1 The Pressure, Flowrate, Viscosity Relation

The viscosity of the concentrate at the nozzle is not the same as that measured with the Contraves viscometer as the concentrate leaves the heat exchanger in the feed line to the high pressure pump. The age and temperature of the concentrate are different, and the shear rate is 1000 times greater at the nozzle. Using the viscosity model in Chapter 4 to obtain the viscosity as a function of the concentrate total solids and temperature, and then calculating the pressure at a given flowrate from the nozzle equation gives very poor agreement with the experimental observations. Instead, a pseudo-viscosity was calculated from the pressure and flowrate readings for the two season's data as follows.

For the SB 54 nozzle

$$P = F^{2.345} \left(2.3679 + 5.30334 \exp\left(-0.03464\, \mu\right)\right) \times 10^{-4}$$

so

$$\mu = \frac{\ln\left(\left(10000\, P / F^{2.345} - 2.3679\right) / 5.30334\right)}{-0.03464}$$

for a sugar solution with an average density 1.103 times that of the concentrate. The pressures measured on concentrate were multiplied by 1.103 before being used in this equation, on the assumption that at constant volumetric flowrate, the pressure is directly proportional to the fluid density (Masters,1972 p. 170)

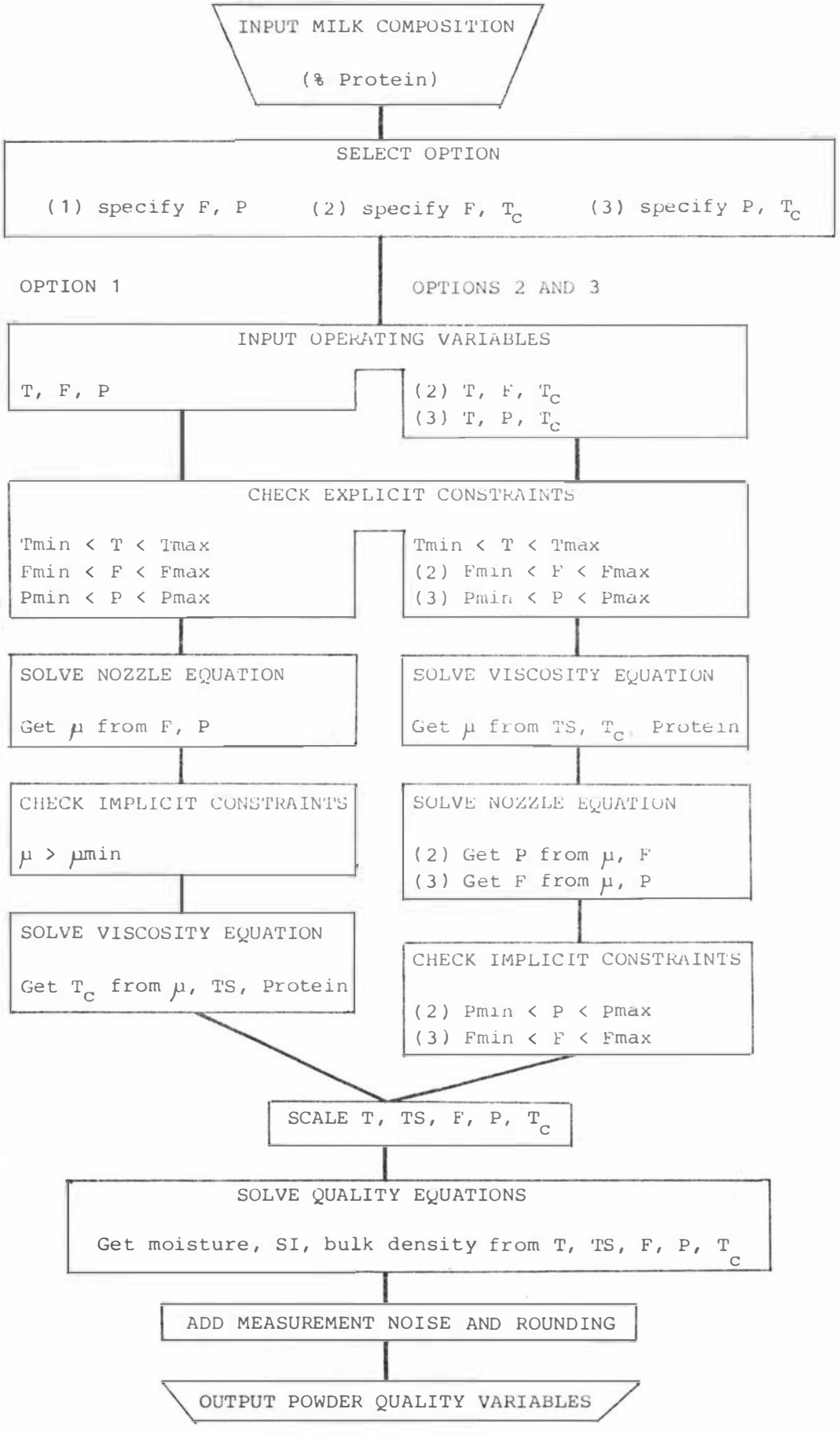


FIGURE 5.1 A Flowchart of the Drier Simulation Model

5.2.2 The Viscosity, Total Solids, Temperature Relation

The pseudo-viscosity calculated above was then regressed against the concentrate total solids, temperature and protein content to give:

$$\mu' = 18.46 + 7.40\ TS - 8.54\ T_c + 2.50\ T_c^2 - 0.82\ T_c\ .TS - 0.45\ Prot - 1.19\ TS.Prot - 1.64\ Prot^2 \quad cp$$

with $r^2 = 0.6867$ and root mean square residual = 5.1 cp.

where TS = (total solids - 47.7) / 2

T_c = (High pressure conc. temp. - 43.7) / 10

and Prot = Protein - 39.74

This model was significantly better than one in which the natural logarithm of the viscosity was used as the response variable, but the rms of the residuals was still 22 % of the mean pseudo-viscosity.

This pseudo-viscosity passes through a minimum as the temperature of the concentrate is raised. The temperature which minimises the viscosity is found by setting to zero the first derivative of the equation with respect to temperature, giving:

$$T_c\ (\min \mu') = (0.82\ TS + 8.54) / (2 \times 2.50)$$

The minimum viscosity may now be calculated, and used to check for an implicit constraint violation when option 1 of the simulation program is chosen. In this option the concentrate flowrate and atomising pressure are specified, so that the pseudo-viscosity may be calculated from the nozzle equation. Figure 5.2 gives the equations used in the simulation model for each of the three options.

5.2.3 The Powder Quality Equations

The equations presented in Chapter 4 are used to calculate the moisture, Solubility Index, bulk density and other quality variables. The throughput of milk solids is also calculated at this stage, using the following equation.

$$\rho_c = 989 + 0.0166\ TS^2 - (\ 0.0076\ T_c - 3.75\ )\ TS - (\ 0.064 + 0.0024\ T_c\ )\ T_c$$

where ρ_c = concentrate density (kg/m3)

TS = concentrate total solids (%)

T_c = concentrate temperature (C)

This is a regression equation fitted to the data of Hall and Hedrick (1966) with the intercept (989 kg/m^3) adjusted to give better agreement with data from New Zealand skim milk concentrates. The solids throughput is then:

$$G = F\ (\ \rho_c/1000\ )\ (\ TS/100\ )\quad kg/h$$ where F is in l/h.

This quantity is needed if the processing rates of the drier and evaporator are to be matched. At the evaporator final effect temperature of 44 C, the total solids-density equation is approximately:

$$TS = (\ \rho_c - 943.7\ )\ /\ 5$$

5.2.4 Measurement Noise and Rounding

Each of the quality variables has an error associated with it. This may be represented by the standard deviation of the laboratory analysis. The simulation program generates normally distributed random numbers with zero mean and unit standard deviation by summing 12 numbers taken from a uniform distribution with range 0 to 1, and subtracting 6. The central limit theorem ensures that 12 is a sufficient number of samples to give a good approximation to a normal distribution. The random noise is multiplied by the standard deviation of each test and is added to the quality variable. Every time the quality variables are evaluated, new noise values are calculated.

The final step is to round the values of moisture, Solubility Index and bulk density to the number of decimal places to which they are usually reported. As an example, this is done for moisture below.

SUBROUTINE TO SOLVE NOZZLE EQUATIONS

Initially set T_c = 43.7 C, so that when scaled T_c = 0

$$\mu_{cp} = 18.46 + 7.40\ TS - 8.54\ T_c + 2.50\ T_c^2 - 0.82\ T_c.TS - 0.45\ Prot - 1.19\ TS.Prot - 1.64\ Prot^2$$

OPTION 1 - Given F, P get μ' and hence Tc

$$\mu' = \ln ((10000\ P / F^{2.345} - 2.3679) / 5.30334) / (-0.03464)$$

$$T_c(\min \mu) = 0.2 (0.82\ TS + 8.54)$$

$$\min \mu = \mu - 8.54\ T_c(\min \mu) + 2.50\ T_c^2(\min \mu) - 0.82\ T_c(\min \mu)\ TS$$

If $\mu' < \min \mu$ then constraint violated, RETURN

Now solve quadratic in T_c

$$a = -0.03464$$
$$b = -8.54 + 0.82\ TS$$
$$c = 18.46 - \mu' + 7.40\ TS - 0.45\ Prot - 1.19\ TS.Prot + 1.64\ Prot^2$$

$$T = \frac{-a - \sqrt{b^2 - 4\ a\ c}}{2\ a}$$

RETURN

OPTION 2 - Given F, T_c get P

$$\mu = \mu - 8.54\ T_c + 2.50\ T_c^2 - 0.82\ T_c.TS$$

$$P = F^{2.345} (2.3679 + 5.30334 \exp (-0.03464\ \mu)) \times 10^{-5} / 1.103$$

RETURN

OPTION 3 - Given P, T_c get F

$$\mu = \mu - 8.54\ T_c + 2.50\ T_c^2 - 0.82\ T_c.TS$$

$$F = (1.103\ P / (2.3679 + 5.30334 \exp (-0.03464\ \mu)) \times 10^{-5})$$

RETURN

Where TS = (TS - 47.4) / 2
T_c = (T_c - 43.7) / 10
F = F (l/h)
P = P (MPa)

FIGURE 5.2 The Simulation Model Subroutine Solving the Equations Relating the Nozzle Pressure, Flowrate and Viscosity and the Concentrate Viscosity, Total Solids and Temperature.

Moisture = Integer value of (10 x Moisture + 0.5) / 10

The standard deviations of the analyses and the number of decimal places to which they are reported are given in Table 5-2. The standard deviation data are based on unpublished surveys of analytical methods conducted by the NZDRI.

TABLE 5-2 Standard Deviations and Reporting Precision for Laboratory Analyses

Analysis	Standard Deviation	Result Reported to Nearest:	
Moisture	0.15 % moisture	0.1 % moisture	
Solubility Index	10 % of reading	SI < 0.45,	0.05 ml
		0.5 ≤ SI ≤ 1.9,	0.1 ml
		SI ≥ 2.0,	0.2 ml
Bulk Density	0.005 g/ml	0.01 g/ml	

5.2.5 Outlet Temperature Control

Two outlet air temperature controllers may be used with the drier simulation model. In one the inlet air temperature is manipulated and in the other the concentrate flowrate or atomising pressure is manipulated. In practice, the latter type of controller adjusts the speed of the high pressure pump. The procedures for simulating these controllers are as follows.

- Inlet air temperature manipulated

The concentrate total solids, flowrate and temperature are fixed, so it is only necessary to solve the outlet temperature equation given in section 4.6 for the inlet air temperature.

- Concentrate flowrate or atomising pressure manipulated

For this controller configuration the inlet air temperature and the concentrate total solids and temperature are fixed. The concentrate viscosity is defined by the last two variables. The outlet air temperature equation given in section 4.6 and the pressure, flowrate, viscosity equation for the nozzle must be solved simultaneously to give both the concentrate flowrate and the atomising pressure.

The equations used in implementing the controllers are given in Figure 5.3. Newton's method is employed to solve the two nonlinear simultaneous equations involved in the second controller.

5.3 - Some Simulation Results

This section presents the results of simulation runs under four drier operating regimes used in the dairy industry. In each case the concentrate temperature is fixed at 43.7 C, a value close to that at which the concentrate leaves the final effect of an evaporator. The preheat treatment conditions are 110 C for 10 seconds, and the milk protein content is constant at 39.74 %. No measurement noise has been added to the response variables.

5.3.1 The Effects of Inlet Temperature, Total Solids and Flowrate at Fixed Concentrate Temperature

Figure 5.4 gives contours of constant response variable on graphs of inlet temperature vs flowrate for three levels of total solids. The relatively small effect of the flowrate on the moisture content may be ascribed to the counter effect of the increase in atomising pressure which accompanies an increase in flowrate. An increase in powder moisture with increasing total solids is apparent, reflecting the greater difficulty of drying the concentrate at higher viscosities and consequenty lower atomising pressures.

The Solubility Index contours are even steeper than in Figure 4.12 because the flowrate and pressure effects are mutually reinforcing.

OPTION 1 - Given TS, F, P, T_c and T_{out} get T by solving the quadratic equation as follows.

$$a = -0.25$$

$$b = 7.21$$

$$c = 93.09 - T_{out} + 0.83\ TS - 2.54\ F - 0.82\ P + 0.34\ F.P + 2.25\ T_c - 0.18\ T_c^2$$

$$T = \frac{-b - \sqrt{b^2 - 4\ a\ c}}{2\ a}$$

OPTION 2 - Given T, TS, T_c and T_{out} get F and P

First guess a flowrate F_1. Get the corresponding pressure P_1 from:

$$P = F^{2.345}\ (\ 2.3679 + 5.30334\ \exp(-0.03464\ \mu\ ))\ \times\ 10^{-4}$$

scale $P_1 = (\ P - 24\ )/4$ and substitute in:

$$F_1 = (\ T_{out} - 93.09 - 7.21\ T - 0.83\ TS + 0.82\ P + 0.25\ T^2 - 2.25\ T_c + 0.18\ T_c^2\ )\ /\ (\ 0.34\ P_1 - 2.54\ )$$

unscale $F = F_1 \times 20 + 279$ and subtract from the former value to form the difference D. Evaluate D at $F_1 - 1$ and $F_1 + 1$ so as to get the slope of a secant to the function. Use Newton's method to calculate the next F value.

$$F_k = F_{k-1} - 2\ D_{F_k}\ /\ (\ D_{F_{k+1}} - D_{F_{k-1}}\ )$$

Continue until successive F values differ by less than 0.05 l/h. This usually takes only three or four iterations.

FIGURE 5.3 The Simulation Model Subroutine Implementing the Outlet Temperature Controllers

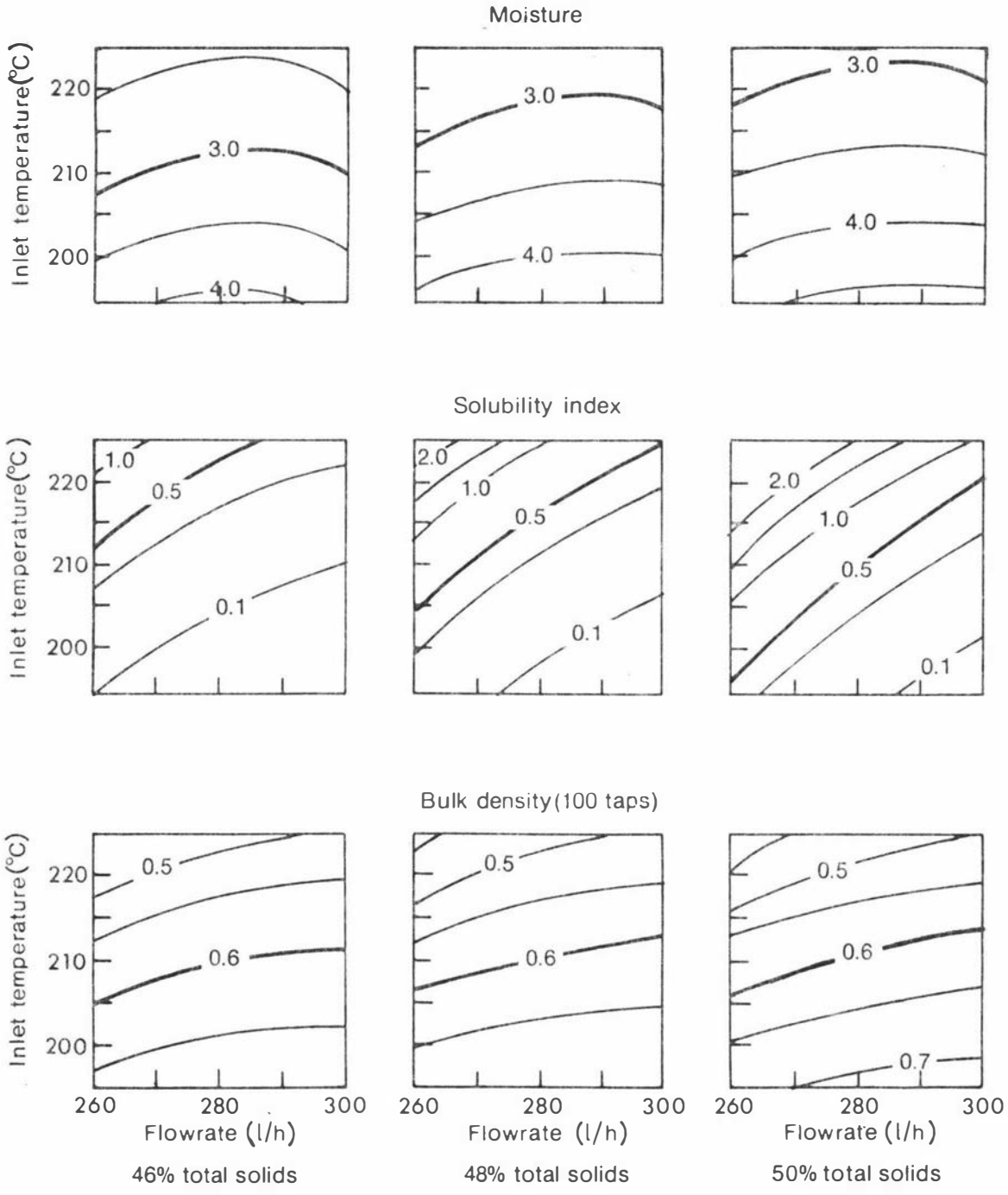


FIGURE 5.4 The Effects of Inlet Temperature, Total Solids and Flowrate at Fixed Concentrate Temperature

Increasing the concentrate total solids causes a greater increase in SI than is shown in Figure 4.12 because the atomising pressure is reduced by the higher viscosity of the concentrate. The bulk density is not strongly affected by changes in concentrate flowrate when the atomising pressure is allowed to change with the flowrate.

5.3.2 The Effects of Inlet Temperature, Total Solids and Atomising Pressure at Fixed Concentrate Temperature

Figure 5-5 gives contours of constant response variable on graphs of inlet air temperature vs atomising pressure for three different concentrate total solids. The overall picture is similar to Figure 5-4, the main difference being that the Solubility Index is not affected by changes in total solids. This is a reflection of the importance of high atomising pressures in reducing the SI of the powder. For a given flowrate the atomising pressure falls as the total solids increases, giving a greater SI as seen in Figure 5-4. For a given atomising pressure, however, the flowrate increases as the total solids increases, counteracting the effect on SI of the higher total solids.

5.3.3 A Comparison of Two Outlet Temperature Controllers

In comparing the two types of outlet air temperature controller, the primary concern must be how closely the powder moisture can be controlled by fixing the outlet temperature. It is therefore necessary to examine the effects of possible disturbances in the variables not being manipulated by the controller. The simulation model was used to generate graphs of moisture content against outlet temperature for each type of controller at three levels of concentrate total solids and either atomising pressure or inlet temperature, as appropriate. The temperature of the concentrate was fixed at 43.7 C. These graphs are presented in Figure 5.6.

When the inlet temperature is the manipulated variable the moisture falls 0.1 % for each 1 C rise in outlet temperature. The moisture increases 0.1 % when the atomising pressure is raised by about 1 MPa, so control of the pressure to within $\pm$0.5 MPa will be sufficient to avoid interference with the control of moisture. The effect of

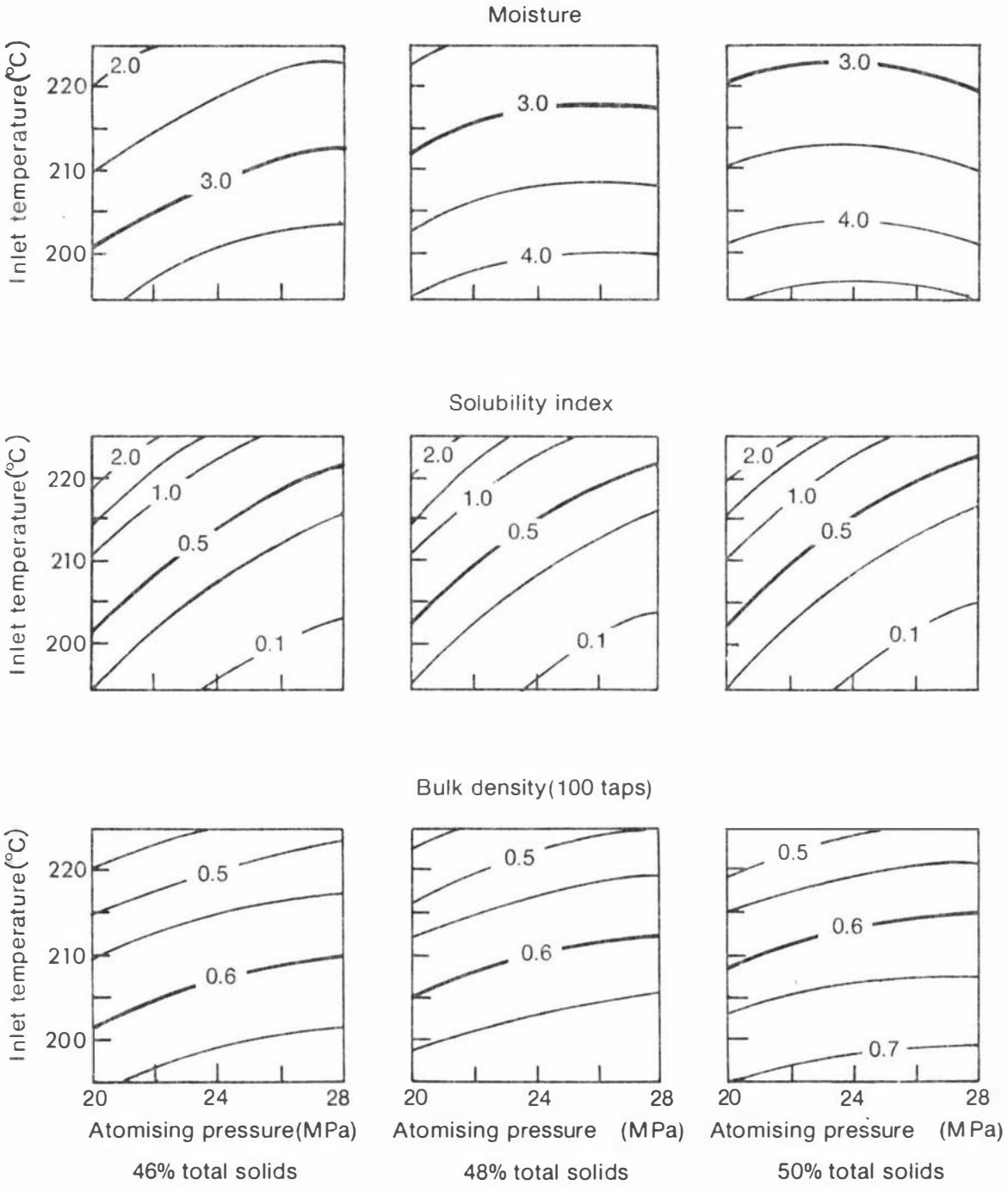


FIGURE 5.5 The Effects of Inlet Temperature, Total Solids and Atomising Pressure at Fixed Concentrate Temperature

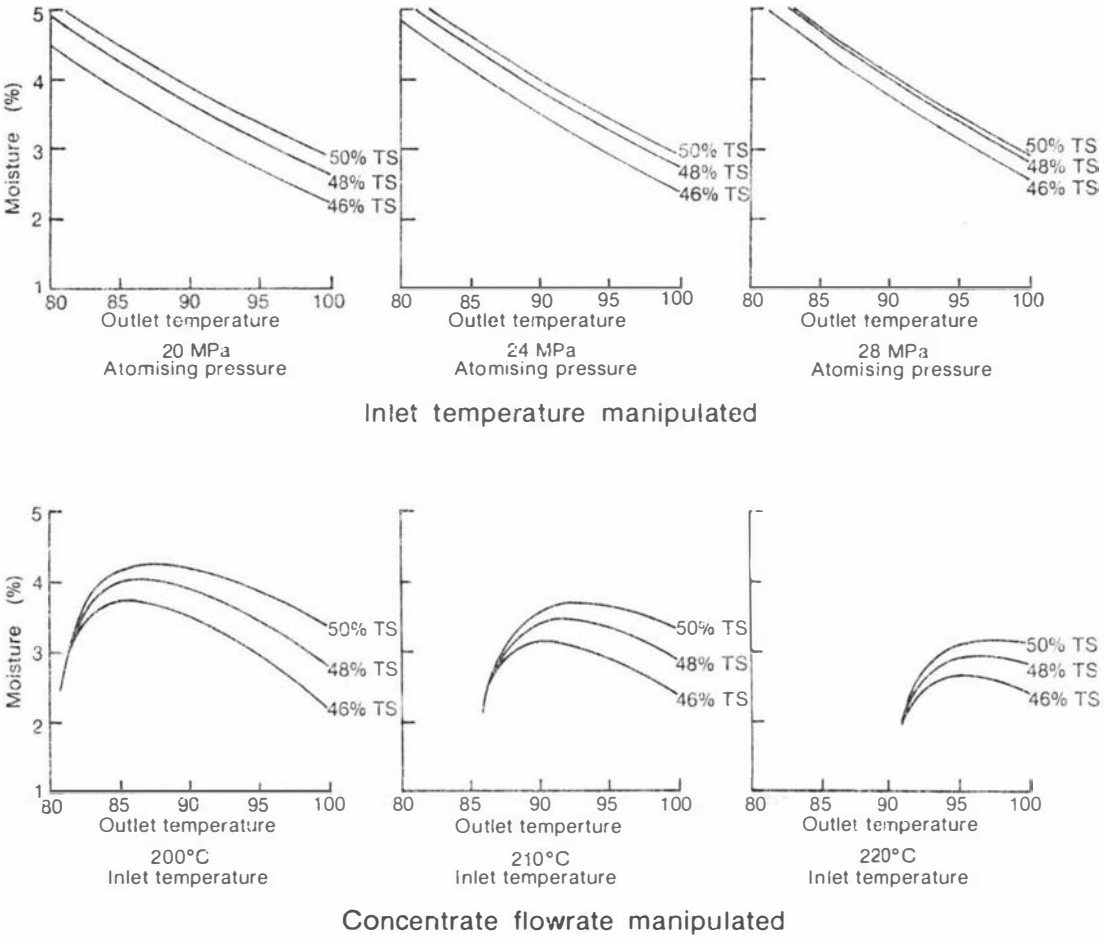


FIGURE 5.6 Graphs of moisture vs outlet temperature for two types of outlet temperature controller

fluctuating total solids decreases as the total solids increase. At about 48 % total solids, variations of $\pm$ 0.8 % solids will cause a 0.1 % moisture change.

When the speed of the high pressure pump is used to control the outlet temperature, the graphs of moisture against outlet temperature have a very different form. As the pump speed is increased, the feedrate and the atomising pressure both increase, but the pressure rises as the 2.345 power of the feedrate. Initially, at high outlet temperatures, the moisture increases as the feedrate increases. As the outlet temperature falls, the pressure effect starts to dominate, and the moisture levels off and then starts to decrease. Eventually a point is reached where no further reduction in the outlet temperature is possible. In practice the pressure limit of the pump would be reached before this point, however. The effect of this behaviour is to make the moisture much less responsive to outlet temperature changes than when the first type of controller is used. Total solids fluctuations, however, have a much greater effect on the powder moisture. The use of the pump speed as a manipulated variable is therefore inferior to the manipulation of the inlet temperature on two counts.

Figure 5.7 gives graphs of Solubility Index as a function of outlet temperature for both types of controller. In both cases the SI increases with increasing outlet temperature and with increasing concentrate total solids.

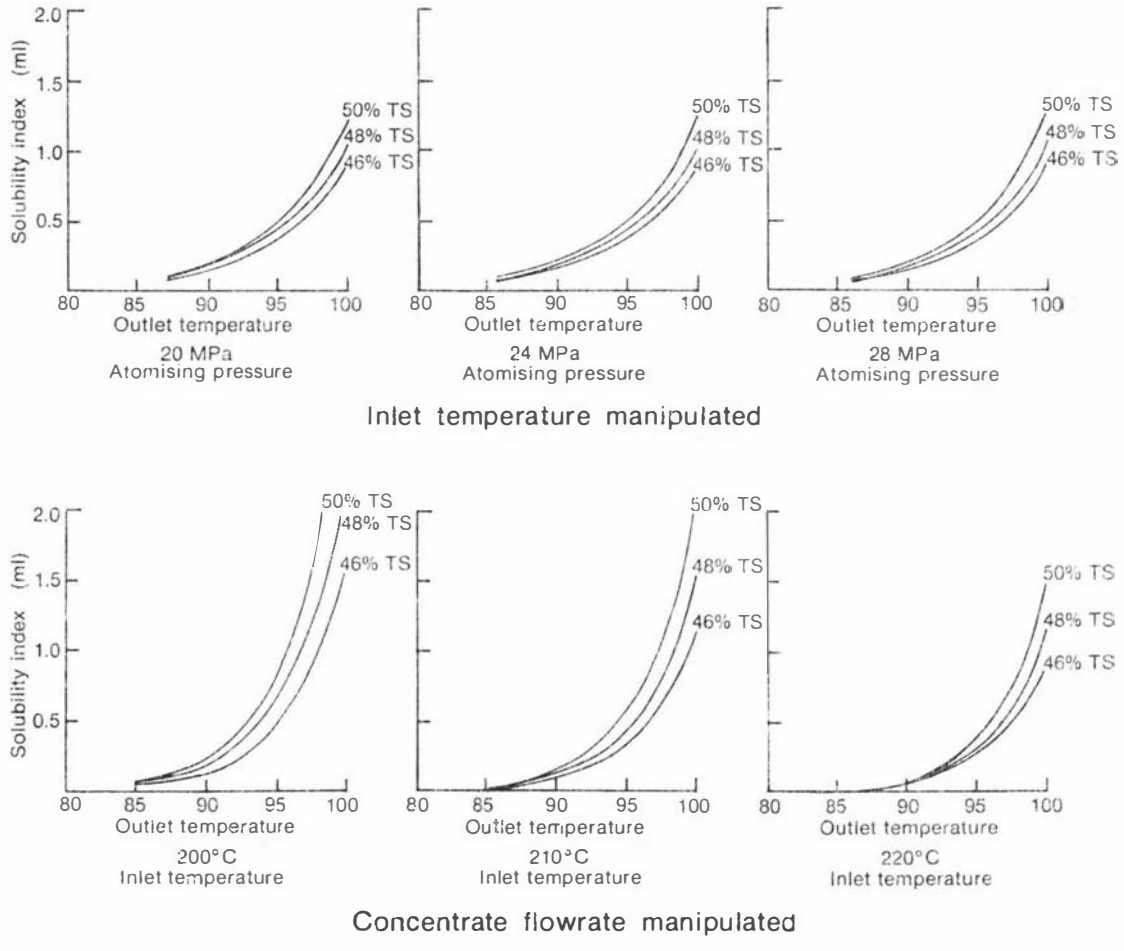


FIGURE 5.7 Graphs of SI vs outlet temperature for two types of outlet temperature controller

CHAPTER 6 - DISCUSSION

6.1 - Methodology

Empirical model building is an art which relies heavily on its practicioner's experience of the system to be modelled and an element of luck in choosing appropriate model forms. Systematic ways of formulating process models are, however, slowly being developed. The concept of degrees of freedom introduced to continuous processes by Morse (1951), has been useful in determining the number of independent variables which may be assigned values arbitrarily. Extensions of this approach to detailed investigation of the structure of the equations describing a process have concentrated on the design problem (Rudd and Watson, 1968). With the exception of the choice of nozzle, however, the spray drying process had already been designed and the desired outputs were known. What were required were the process inputs necessary to obtain the specified powder quality. Because of the impracticality of performing a single all-encompassing experiment in all the variables, some means of establishing the best subsets of process variables for the experimental work was required. Rudd and Watson's technique was easily adapted to this task, and proved successful.

6.2 - Atomisation

The experimental work on the nozzle hydrodynamics shows that the atomising nozzles in most widespread use in the New Zealand dairy industry fall into two distinct classes; those with little sensitivity to fluid viscosity changes, and those which show a marked reduction in pressure drop as the fluid viscosity is increased. This difference may be attributed to the ratio of the diameters of the swirl chamber and orifice. This ratio is large for nozzles which exhibit viscosity sensitivity and small for nozzle which do not. The ratio represents the acceleration undergone by the fluid in a free vortex if angular momentum is conserved as it spirals inward from the inlet port to the orifice, and is therefore a measure of the degree of shear present within the swirl chamber. The experimental findings conflict with those of Watanabe (1974), who found that for a wide range of Spraying Systems and Delavan

nozzles, flowrates at constant pressure were lower for milk concentrate than for water.

The reduction in pressure drop with increasing fluid viscosity when the feedrate is held constant has been explained by Dombrowski and Munday (1968) who reasoned that because tangential velocities in nozzle swirl chambers are generally higher than axial velocities, the tangential velocities will be more affected by fluid viscosity. Since a loss in tangential velocity reduces the diameter of the air core which occupies the centre of the swirl chamber, the cross sectional area of the liquid film issuing from the orifice increases, reducing the overall pressure drop over the nozzle. At high viscosities the tangential and axial velocities become comparable, the liquid film nearly fills the orifice, and the pressure drop begins to increase. Once the air core is eliminated completely, the pressure drop should rise linearly with increasing viscosity as the nozzle has become a simple orifice operating in laminar flow.

Most of the nozzles studied did not show a pressure drop increase until the viscosity exceeded 5 to 8 poise, which was well outside the range normally encountered in skim milk and whole milk drying. These products are usually atomised at viscosities between 0.2 and 1.0 poise. The lower limit is imposed by the need to concentrate the material as much as possible before drying since water removal in a spray drier requires approximately ten times the energy per kilogram required by a multiple effect evaporator (King, 1967). The upper limit is imposed by product quality considerations, particularly solubility requirements (King, Sanderson and Woodhams, 1974).

The viscosity of the concentrate affects the performance of spray driers in three ways. The nozzle flowrate-pressure relationship may be influenced as just described, depending on the choice of nozzle. The mean particle size increases as the viscosity increases as shown in Table 4-8, reducing the drying rate. Finally, the drying rate of a droplet of given size is reduced as the viscosity of the fluid increases (Marshall, 1954).

Large capacity nozzles show a significant decrease in pressure

drop with increasing fluid viscosity, so driers using them have all three effects acting in concert. If such a drier is run at constant atomising pressure and the concentrate viscosity increases, then the feedrate and therefore the evaporative load will increase. The droplet size will increase because of the greater flowrate, reducing the drying rate. The overall effect will be a large increase in the moisture content of the powder as a result of a small increase in concentrate viscosity due, for example, to variations in total solids. With the advent in the last decade of large tall-form driers employing a small number of large capacity nozzles, low concentrate viscosities and good viscosity control have become increasingly important in attaining optimum performance.

6.3 - Concentrate Viscosity

The relatively high standard errors of prediction of the regression equations for concentrate viscosity reflect the susceptibility of the viscosity measurements to error. The in-line viscometer was used near the lower limit of its range for most of the experimental work on skim milk concentrates. It was also subjected to considerable temperature variation during each day, and changing bearing friction and possible fouling of the surfaces of the measuring elements may have contributed to the error.

The viscosity of skim milk concentrate was found to be influenced by five principal factors; temperature, total solids, age, protein content and preheat treatment. A high temperature, short time preheat treatment gave a lower concentrate viscosity than a low temperature, long time treatment giving the same WPNI. It also gave a lower rate of viscosity increase with time. Since high temperature, short time preheat treatment combinations have gained widespread acceptance in New Zealand recent years, no further reduction in concentrate viscosity seems likely by altering preheat conditions.

More progress is possible by reducing the time concentrate is held before drying. Improved process control of evaporators and driers will permit the use of much smaller balance tank volumes, reducing the concentrate holding time without increasing the risk of running the tank

dry. Tighter control of concentrate total solids will improve product uniformity directly and through the effect the total solids has on viscosity. The introduction of concentrate temperature manipulation for viscosity control may also prove attractive.

The marked increase in concentrate viscosity towards the end of each dairying season is largely attributable to the protein content of the milk, which rises at these times due to the combined effects of the lactational cycle of the cows and the effect of late summer weather on pasture growth. None of the other compositional factors investigated had significant effects on the concentrate viscosity.

6.4 - Drier Design Features

Spray driers vary widely in their design, and individual driers are sometimes modified in the hope of increasing their throughput or of overcoming problems with high SI values or powder deposition on their interior surfaces. It was therefore decided to investigate the effects of some of the drier design variables which influence spray-air mixing, the heart of the spray drying process.

The position of the atomising nozzle relative to the drier roof had no effect over an 80 mm range above and below the normal postion. This indicates that the spray-air mixing was not affected by modest changes about the normal position.

Increasing the air inlet velocity by a factor of 2.26 by reducing the throat diameter increased the average bulk density of the powder from 0.64 to 0.72 g/ml, measured at 100 taps. This was accompanied by a very slight increase in the particle density. None of the other powder properties was significantly affected. This is surprising, as the more vigorous mixing of the spray with the drying air at the higher inlet velocity might be expected to promote more rapid drying with less heat damage. The lower inlet velocity was used in all the other experimental work, although the higher velocity is more representative of industrial tall- form driers.

The only other design variables investigated were those involved in the selection of atomising nozzles; the type of nozzle, and the swirl chamber and orifice sizes. The sensitivity of the atomising nozzles to viscosity changes plays such an important part in determining the overall behaviour of a spray drier that driers may be put into two distinct classes based on the type of nozzle installed.

When the swirl chamber and orifice sizes of Delavan SDX series nozzles were changed and the concentrate feedrate and atomising pressure were kept constant, the powder moisture content decreased with increasing swirl chamber and orifice size. This was accompanied by a decrease in particle size due to the considerably reduced concentrate viscosity needed to maintain the same pressure drop for the given flowrate. When the orifice size of a Spraying Systems SX series nozzle was increased at constant concentrate viscosity and atomising pressure, the powder moisture content and mean particle size increased, reflecting the increased flowrate through the larger orifice. This effect was observed by Amundson (1960), as shown in Table 2-5. A Delavan SB 54 nozzle was selected for all the other experimental work carried out to determine the effects of the operating variables on the product quality.

6.5 - Operating Variables

The full instrumentation and computer control of the pilot plant evaporator and spray drier used in this work has made it possible to examine the effects of all the operating variables simultaneously. Interactions between the variables could therefore be investigated. This represents an advance on the work of Amundson and others whose facilities constrained them to the investigation of one variable at a time. The result of the experimental work has been a complete description of the quality of skim milk powder in terms of the physical properties of the skim milk and the process operating variables. This is evidenced by the generally good fits of the regression equations. It has also been possible to separate the effect of concentrate total solids directly on the powder properties from its effect on the concentrate viscosity, and consequently on the nozzle hydrodynamics. The limited number of degrees of freedom in the system means that these

effects can be distinguished only by investigating several different combinations of independent variables, each with its own set of covariates. The emphasis throughout the work has been on obtaining input-output relations, with no attempt being made to correlate output variables such as moisture and outlet temperature.

The inlet air temperature determines the major energy input into the drier. Higher temperatures gave lower moistures, but also increased the heat damage to the product, as shown by raised SI values. Wright (1932) showed that heating partially dried powder adversely affects the solubility of the final product. Higher temperatures also increased 'ballooning' of powder particles, increasing the mean particle size and reducing the particle density and bulk density. These effects are similar to those found by Amundson given in Table 2-5.

The other operating variables, the total solids, temperature and feedrate of the concentrate, and the atomising pressure, are all inter-related. The concentrate temperature and total solids determine the viscosity, which in turn may affect the relationship between the feedrate and the atomising pressure. The choice of nozzle determines which combinations of three of the four variables may be varied independently, leaving the fourth variable as a covariate.

The drier behaviour that will be observed depends on the choice of covariate. This is illustrated by comparing the powder moisture contours in Figures 4.11, 5.4 and 5.5. In the first case, when the concentrate temperature was manipulated to give a specified concentrate feedrate and atomising pressure for each level of total solids, increasing the concentrate total solids reduced the moisture. When the temperature of the concentrate was fixed, and either the feedrate or atomising pressure was chosen as the covariate, the moisture content of the powder increased with increasing concentrate total solids. The conclusion to be drawn from this is that when the concentrate viscosity is allowed to increase with the total solids, and no additional energy is put into the system, drying is made more difficult. When the viscosity is kept approximately constant by heating the concentrate as the total solids rises, the reduced evaporative load and the additional thermal energy input combine to make drying easier.

Similar behaviour was exhibited by the Solubility Index, as a comparison of Figures 4.12, 5.5 and 5.4 shows. When the concentrate temperature was the covariate, as in Figure 4.12, increasing the concentrate total solids decreased the SI of the powder. When the concentrate temperature was fixed, and the feedrate was the covariate, the total solids had no effect on the SI. When the atomising pressure was chosen as the covariate, Figure 5.4 shows that increasing the concentrate total solids increased the SI of the powder.

The effect of the atomising pressure was amplified by the way the Delavan SB 54 nozzle reacted to viscosity changes. The pressure could be increased by increasing the feedrate or by reducing the concentrate viscosity, by the use of low total solids or high concentrate temperatures. In the former case, illustrated in Figure 5.5, the increased evaporative load outweighed the effect of finer atomisation, and the moisture content of the powder rose. The SI was reduced and the particle and bulk densities increased. In the latter case, the concentrate dried more readily and with less heat damage, to give a powder with a smaller mean particle size and lower particle and bulk densities. The Solubility Index improved as the atomising pressure was raised, in both cases. High atomising pressures are therefore desirable if low SI values are to be attained.

6.6 The Simulation Model

The drier simulation model was used to generate the graphs depicting the drier behaviour discussed in the last section. The model was also used to evaluate two outlet air temperature controllers, and to select the parameters of the optimisation scheme described in Part II.

A disappointing aspect of the model development was the lack of a clearly defined relationship between the concentrate viscosity measured with the Contraves viscometer and the pseudo-viscosity calculated from the feedrate and atomising pressure. Making allowance for the difference in concentrate temperature between the viscometer and the nozzle and the variation in residence time in the feed line as the flowrate changed did not significantly improve the fit. The pseudo-viscosities were generally

lower than those measured with the viscometer, so it is likely that some degree of shear thinning is taking place. Variations in the sensitivity of the viscosity to the shear rate, coupled with the measurement errors discussed in Section 6.2 probably account for the poor correlation between the two viscosities.

Steady-state models such as the one developed for the pilot scale drier are powerful tools in the investigation of process control strategies. The dynamics of each item of ancillary equipment such as the evaporator and the air heater will place constraints on the feasibility of some control loop configurations, but if there are several configurations which appear practical, only a study of the steady-state process input-output relationships will decide which is the best. In the case of the spray drier, the criterion for choosing the better of the two proposed strategies was that the influence of fluctuations in the concentrate total solids on the powder moisture content should be minimised.

The use of the simulation model in the quality control system development will be discussed in Part II.

PART II OPTIMISATION

CHAPTER 7 - INTRODUCTION

There are two main lines of attack on the problem of optimising product quality and production economics in a continuous process. One is to carry out a response surface experimental design to obtain a model of the plant behaviour which may then be optimised by any suitable function maximisation procedure to give the plant settings corresponding to the desired product quality. This approach has been well illustrated by Bacon (1970). The other is to optimise the plant by a form of Evolutionary Operation, a technique originated by Box (1957).

The evolutionary approach was adopted in this work because the raw material, milk, is a complex biological fluid. The composition and hence the processing characteristics of milk change with the lactational cycle of the cow, the condition of the pasture or other feed, and the weather. The age of the milk when processed and its storage conditions also affect the processing characteristics. This variablility in the raw material implies that the optimum processing conditions will change with time. The development of statistical models sufficiently comprehensive to allow for this variability would be prohibitively costly, particularly when the diversity of drier types in the New Zealand dairy industry is considered.

Evolutionary techniques present other difficulties, namely the selection of technique from the number that have been proposed, and the determination of the best step sizes and other parameters for the technique chosen, given that large scale industrial experimentation has been ruled out. The Simplex EVOP scheme was chosen after a detailed examination of the literature on the theory and applications of the various evolutionary schemes which have been proposed. The selection of the parameter values was made by a series of trials on the simulation model of the spray drier described in Part I. The fact that this model is specific to the pilot scale drier does not affect the transfer of the evolutionary scheme to commercial driers. The qualitative similarity of all nozzle atomising driers ensures that only minor changes in parameter values should be necessary.

7.1 - Evolutionary Optimisation Techniques

Evolutionary Operation (EVOP) is a systematic procedure for the optimisation of industrial processes. As originally propounded by Box (1957) the EVOP scheme involves the repeated application of a two level factorial experimental design centred on the current process operating conditions. The levels of the independent variables are chosen to be sufficiently close to the current conditions that no serious harm is done to the product quality. Analysis of variance is used to distinguish the effects of the variables, and hence the direction of greatest process improvement, from the experimental error or measurement "noise". The centre of the factorial design is then moved in this direction, and the cycle is repeated.

This basic scheme has been modified in a number of ways since its introduction. Lowe (1964) has summarised two such variants, Rotating Square Evolutionary Operation (ROVOP), and Random Evolutionary Operation (REVOP). ROVOP has the advantage that the differences between the levels of the independent variables increase and decrease during operation so that the initial choice does not greatly affect the optimising performance. Quadratic terms for the fitting of a response surface are also available. The experimental design and its analysis become very complicated when more than three factors are varied. REVOP uses randomly chosen combinations of variable settings and takes steps in the direction of improving response until no further improvement is found. A new combination of variables is then chosen at random and the cycle is repeated. Neither scheme has found many applications, according to surveys by Hunter and Kittrell (1966) and Hahn and Dershowitz (1974).

The Factorial EVOP scheme was originally intended to be carried out by a committee, however the procedure has been automated by Simmons (1976). In this version, the differences between the levels of the factors and the size of the steps taken in the direction of greatest quality improvement are varied. An added refinement is that linear and quadratic drifts are corrected for after every two replications of the factorial design. The result is a noise tolerant steepest ascent maximisation procedure suitable for use in conjunction with a computer

control system.

Another form of Evolutionary Operation, Simplex EVOP, has been developed by Spendley, Hext and Himsworth (1962). They considered how an EVOP procedure might be devised which would more rapidly approach and attain optimum conditions, and be automatic in operation. The basic design is the regular simplex in k dimensions, where k is the number of factors under investigation. Once the initial simplex has been completed, a new experimental point is immediately chosen. This point is that required to complete the adjacent simplex formed by replacing the point of the current simplex corresponding to the worst response by it by its reflection in the hyperplane of the remaining points. This defines a steepest ascent procedure in which the frequency and extent of the steps are rigorously defined. The advantages of the Simplex scheme over Factorial EVOP are that the arithmetic is extremely simple, the direction of advance is dependent solely on the ranking of the responses, and not on their numerical values, only the k+1 most recent responses are used, so that a moving optimum may be readily followed, and a new factor previously held constant may be introduced at any stage. Examples of the application of Simplex EVOP to industrial processes are given by Lowe (1964 and 1973), Kenworthy (1967) and Glass and Bruley (1973).

The Simplex method has been modified for use in error-free function minimisation by Nelder and Mead (1965). The use of regular simplices has been dropped in favour of adjusting the distance the discarded point is to be reflected, thereby expanding or contracting the simplex. By this means the simplices adapt themselves to the local landscape and finally contract on to the minimum. A stopping criterion is also provided. Olsson and Nelson (1975) provide examples of the use of this technique on six different functions. Deming and Morgan (1973) have used the original Simplex EVOP method in analytical chemistry and least-squares curve fitting. They also quote an example of the use of the modified form in experimental optimisation.

7.2 - The Choice of Optimisation Scheme

In a detailed comparison of maximum-seeking methods, Brooks (1959) found the steepest ascent method to give the closest approach to the maximum of a two factor function for a fixed number of trials. This applied for both 16 and 30 trials, and for response surfaces with and without error. The steepest ascent method employed a two factor factorial design for slope estimation. Brooks and Mickey (1961) subsequently proved that in the absence of error the optimum design for gradient determination has one more point than the number of factors. They suggest the simplex design as a suitable choice for this purpose. When experimental error is present, they conclude that replication is not advisable. Spendley et al. (1962) investigated the performance of their Simplex EVOP scheme on Brooks'response surfaces and found it to be only slightly less effective than the best steepest ascent procedure. From further simulation runs they found that in the presence of error, the rate of advance is inversely proportional to the error standard deviation, so that replication of observations is actually harmful. The efficiency of the Simplex EVOP technique was found to increase in direct proportion to the number of factors investigated. When the Simplex procedure is continued indefinitely rather than terminated at the maximum, the average fall in response was approximately proportional to the square root of the error standard deviation. Conversely, when the standard deviation was fixed, the average fall in response was directly proportional to the step size. Carpenter and Sweeny (1965) demonstrated that when the ratio of gradient to error is greater than 0.5 the most rapid progress is made without replication of observations.

On theoretical grounds then, the Simplex EVOP scheme appears to offer the most rapid possible improvement in response from a limited number of trials. Continued operation, which is a requirement if a moving optimum is to be tracked, does not cause any serious diminution of response. This has been confirmed in industrial applications reported by Lowe (1964 and 1974) and Kenworthy (1967). The Simplex EVOP procedure was therefore adopted for the empirical optimisation of the spray drying process.

CHAPTER 8 - THE SIMPLEX SCHEME

8.1 - The Simplex Algorithm

The description which follows has been abridged from that given by Spendley et al. (1962).

The basic design of the scheme is the regular simplex in k dimensions, where k is the number of factors under investigation. When k is 2, the experimental points lie on the vertices of an equilateral triangle. When k is 3, the points lie on the apices of a tetrahedron. In the drier optimisation work k was 3 or 4. Relative to a chosen origin a regular simplex of unit edge is conveniently specified by the (k+1) x k design matrix:

$$D = \begin{bmatrix} 0 & 0 & 0 & \cdots & 0 \\ p & q & q & \cdots & q \\ q & p & q & \cdots & q \\ \cdot & \cdot & \cdot & \cdots & q \\ q & q & q & \cdots & p \end{bmatrix}$$

where $p = ((k-1) + \sqrt{k+1}) / (k/2)$

and $q = (\sqrt{k+1} - 1) / (k/2)$

For k = 2, p = 0.9659 and q = 0.2588

k = 3, p = 0.9428 and q = 0.2357

k = 4, p = 0.9256 and q = 0.2185

The rows of the matrix give the k co-ordinates of each of the k+1 vertices of the simplex. D was chosen as the starting simplex for all the simulation work and for two of the experimental trials of the EVOP scheme.

In general, any simplex S with vertices V_1, V_2, ... V_{k+1} and centre C_o, may have a new simplex S_j constructed on any face. S_j will have k vertices in common with S_o, and one new vertex V_j^*, the mirror image of V_j in the common face. To find any one co-ordinate of V_j^* we take twice the average of the corresponding coordinates for the common

vertices $V_1, \ldots V_{j-1}, V_{j+1}, \ldots V_{k+1}$ and subtract the corresponding co-ordinate of V_j. In vector notation:

$$V_j^* = 2/k \left(V_1 + V_2 + \ldots + V_{j-1} + V_{j+1} + \ldots + V_{k+1} \right) - V_j$$

Suppose now that S_o is a simplex in the factor space and that the responses at the vertices have been estimated by experimental readings y_j. Then we move through the factor space in that direction $C_o \rightarrow C_p$ which is nearest to the direction of steepest ascent by applying these rules.

Rule 1 Ascertain the lowest reading y_p of $y_1 \ldots y_{k+1}$. Complete a new simplex S_p by excluding the point V_p corresponding to y_p and replacing it by V_p^* defined as above.

Rule 2 If a result has occurred in (k+1) successive simplices, and is not then discarded by application of Rule 1, do not move, but discard the result and replace it by a new observation at the same point.

Rule 3 If y_p is the lowest reading in S_o, and if the next observation made, y_p^*, is the lowest reading in the new simplex S_p, do not apply Rule 1 and return to S_o. Move out of S_p by rejecting the second lowest reading, which is also the second lowest in S_o.

A fourth rule has been added by Kenworthy (1967).

Rule 4 Where there are constraints applied to the system and the move suggested by the above rules would be into an area forbidden by these constraints, then the move must be made from the point with the least satisfactory response which allows the system to remain within the permitted region.

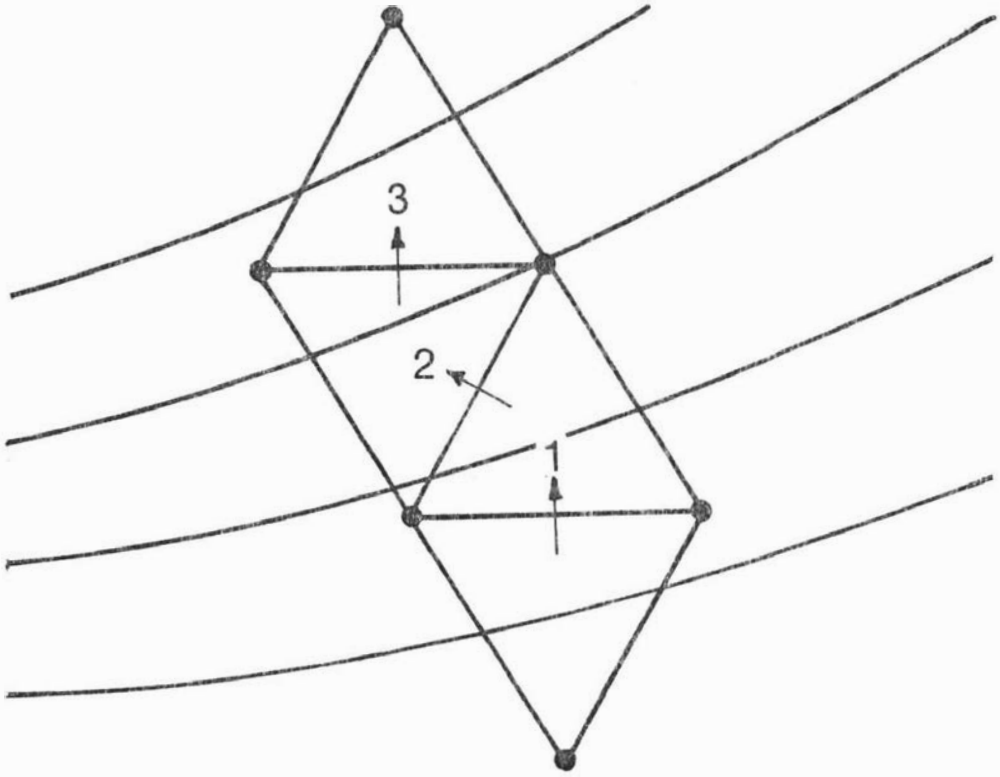
These rules have an implication which has not been mentioned in the literature, and was not appreciated until the experimental trial was begun. This is that unless a point is being repeated, as soon as each new response is measured the next two experimental points may be determined. The first point is obtained by application of Rule 1.

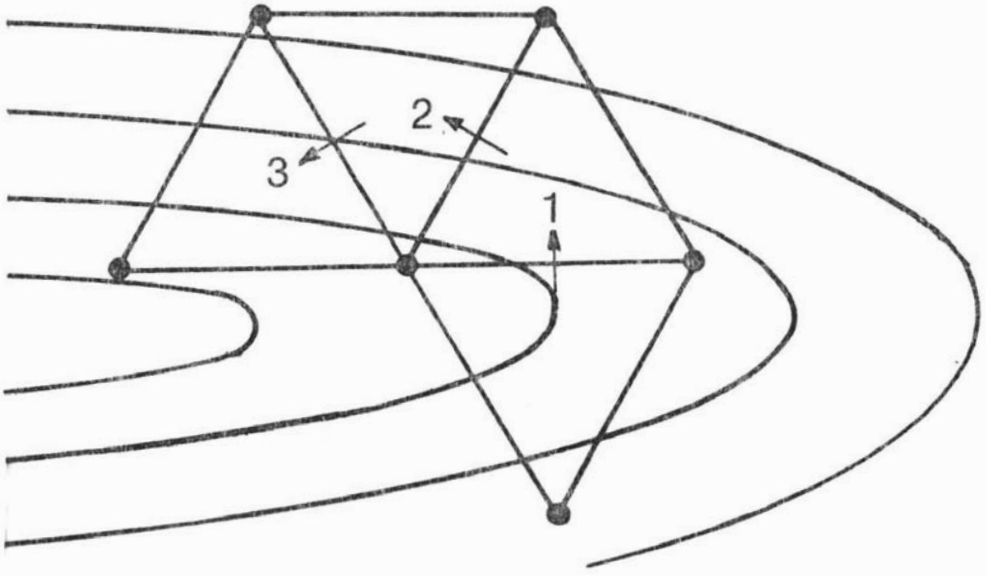
Suppose that the response at this point is better than that at the worst point, V_j, in the current simplex. The following move will be made by rejecting V_j from the new simplex, so the co-ordinates of this point may be calculated in advance. If, on the other hand, the new response is no better than that at V , then Rule 3 requires that V_j^*, which is now the second worst point, be reflected from the new simplex. In both cases then, the next two moves are the same. Only on the third move will account be taken of any failure to improve. This is illustrated in Figure 8.1.

This feature of the Simplex EVOP scheme is of great practical benefit when the time taken to measure the response is of the same order as that required to change the plant settings and settle the process at the new setpoints. This was the case in the experimental trial where the laboratory analyses of the powder moisture, Solubility Index and bulk density and the calculation of the response took about six minutes to complete. It took between 10 and 15 minutes to attain new setpoints on the evaporator and drier. The setpoints could be altered as soon as each powder sample had been taken, saving a considerable amount of time. This would also apply in the commercial implementation of the Simplex EVOP scheme, since the response times of commercial driers are at least twice as long as that of the pilot plant with its computer process control system, and the standard analytical methods which take at least 20 minutes to perform would probably be used.

The only time the calculation cannot be done in advance is when Rule 2 is invoked to repeat an over-age point. Under these circumstances the penalty for remaining at the same conditions for longer than usual is likely to be small, since only a very good point will survive long enough to be repeated. The full benefits of rapid movement are therefore available even when there are significant delays in determining the response.

The implementation of the Simplex EVOP scheme requires that the variables to be manipulated be chosen. The sizes of the steps to be made in each co-ordinate direction must be decided upon. The possibility of varying the sizes of the steps during operation must be considered. It will be necessary to establish the constraints, both explicit and





(b) First move does not improve the response

FIGURE 8.1 Three successive moves for the Simplex EVOP scheme

implicit, on the manipulated variables. Finally, the criterion for ranking the responses of the process to each set of plant conditions is of great importance.

Once these decisions have been made, a starting position and orientation in the variable space may be chosen and the evolutionary optimisation process may be initiated. These requirements are examined in the following sections.

8.2 - Choice of Manipulated Variables

The drier studies provide considerable guidance on the choice of the manipulated variables. Given that a nozzle has been selected and that the airflow through the drier and the inlet geometry are fixed, four degrees of freedom remain. These are reduced to three or two in many industrial driers. Nozzle atomising driers using from 12 to 30 Spraying Systems nozzles of small capacity have no means of independently varying the feedrate and atomising pressure, except by shutting off or turning on one or more nozzles. Many of these driers have steam coil air heaters which have no means of temperature control other than a pressure regulator. The air inlet temperature is therefore fixed. Few of the tall-form driers have concentrate heat exchangers, so although the nozzles they use show marked sensitivity to fluid viscosity, it is not possible to take advantage of this to provide an additional degree of freedom. Some of these driers have variable speed high pressure pumps, and control the concentrate feedrate, while the rest have pumps run at constant speed and use a bypass valve to regulate the atomising pressure.

These considerations mean that two, three and four variable evolutionary operation must be considered, with a range of possible choices of variable within each category. The possible combinations are listed in Table 8-1. The concentrate temperature, total solids, feedrate and atomising pressure are interrelated. The first two variables affect the concentrate viscosity and this in turn affects the last two variables. The curve of atomising pressure as a function of viscosity at constant feedrate given in Figure 4.4 and the graphs of viscosity against concen-

TABLE 8-1 Choices of Manipulated Variables

Two Variables:	Concentrate feedrate or atomising pressure
	Concentrate total solids or inlet air temperature
Three variables:	Concentrate feedrate or atomising pressure
	Concentrate total solids
	Air inlet temperature
Four Variables:	Any two of; concentrate feedrate
	concentrate temperature
	atomising pressure
	Concentrate total solids
	Air inlet temperature

trate temperature given in Figure 4.7 may be combined to show the effect of concentrate temperature on the atomising pressure. This has been done in Figure 8.2 for a Delavan SB 54 nozzle and a constant feedrate of 280 l/h. For concentrate temperatures below 55 C, the atomising pressure rises linearly with increasing temperature. Lower total solids give higher pressures because of the reduced concentrate viscosity.

8.3 Choice of Step Sizes

The correct choice of step size for each manipulated variable is important for the success of the Simplex scheme. Spendley et al. (1962) suggested that the steps be scaled so that the changes in response due to steps in each variable are of equal interest to the experimenter. This becomes complicated when there are several responses, each reacting differently to changes in the operating variables. The size of the steps must be large enough for the process control system to be able to achieve them dependably, and for the changes in response to be detected through the measurement noise. The steps should not be too large, however, or the optimum will never be approached sufficiently closely.

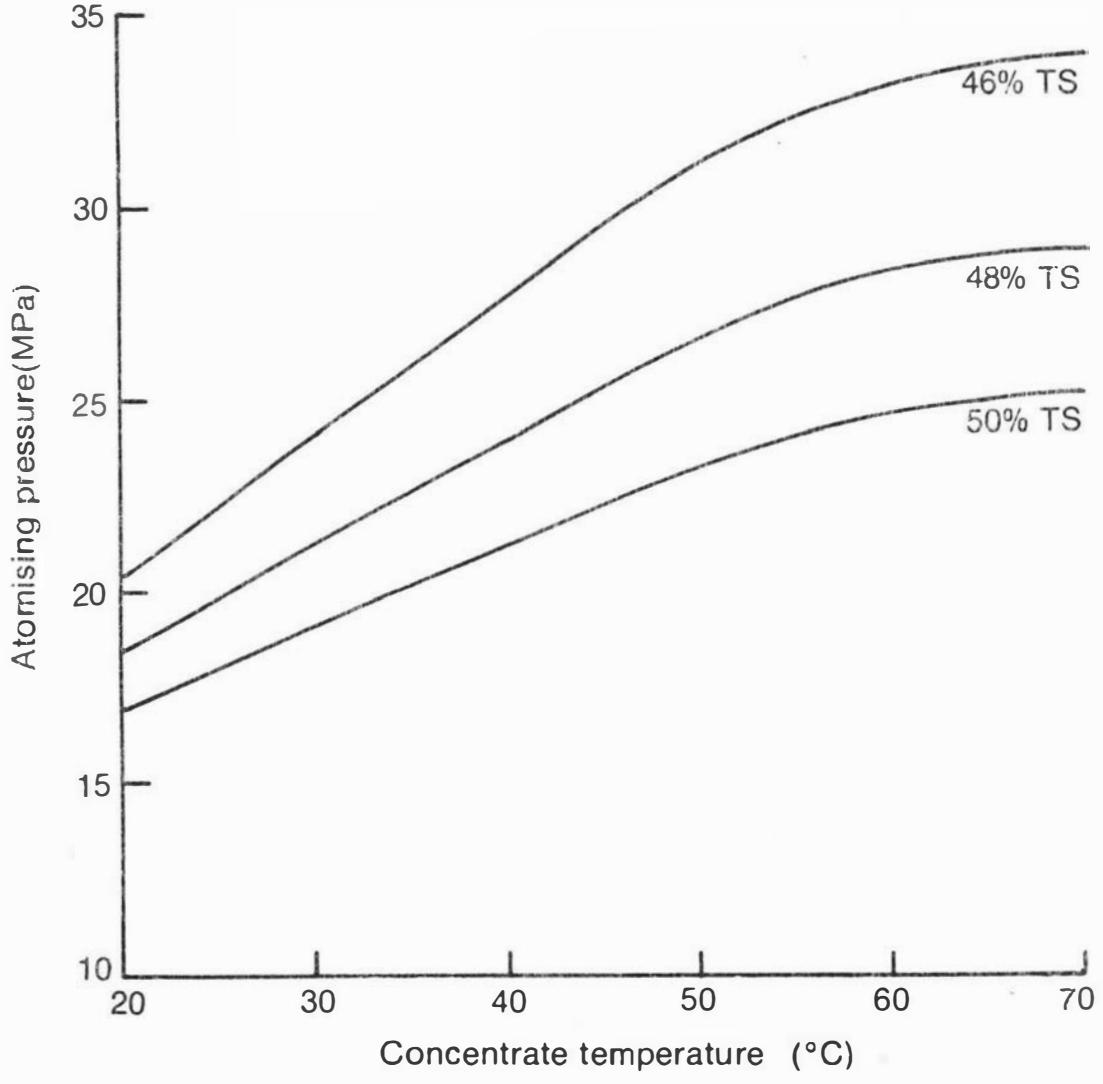


FIGURE 8.2 Graphs of atomising pressure against concentrate temperature at a constant feedrate of 280 l/h and three concentrate total solids

The step sizes for each operating variable were chosen by running a series of experiments on the simulation model. Two sizes for each step were arrived at by considering the change in each quality variable for a unit change in each operating variable in the light of the analytical standard deviations given in Table 3-2. A two level factorial design was then performed with these step sizes in the Simplex scheme. The improvement obtained after 30 points had been run was used as the response variable. Table 8-1 gives the preferred step sizes and the multiplying factor which is used to obtain the length of the edges of the simplex from the length of the step in each variable direction. These factors are the reciprocals of the p values in Section 8.1.

TABLE 8-2 Step Sizes for the Manipulated Variables

Number of Variables	Edge Factor	T (C)	TS (%)	F (l/h)	P (MPa)	T_c(C)
3	1.0607	4.0	1.5	4.0	-	-
3		4.0	1.5	-	1.3	-
4	1.0804	4.0	1.5	4.0	1.3	-
4		4.0	1.5	4.0	-	2.0
4		4.0	1.5	-	1.3	2.0

8.4 - Ranking the Responses

The optimisation of a multi-response system like the spray drier presents a problem in multiple criteria decision making. One approach to such a problem is to reduce the responses to common units, usually monetary, so that a single criterion, total cost, may be applied. This is seldom straightforward. Other approaches involve the more or less arbitrary selection of weightings for the relative importance of each response variable. This is even less straightforward. The Simplex

method requires only that the worst and second worst combinations of responses be identified, so there is no need for exact determination of the absolute desirability of all the response combinations. Rather than invest a lot of effort on this small, if important, aspect of the work, it was decided to assign monetary values to each of the response variables.

Four response variables were chosen. The powder moisture content and Solubility Index appear in the purchasing specifications of most powders, and so must be included. The bulk density was included because of the need to minimise storage and shipping costs by maximising the density, and because both upper and lower limits are set in the specifications of powders to be packaged in fixed volume containers such as packets and cans. Finally, it is necessary to match the processing rates of the evaporator and spray drier, so the throughput of milk solids was included as a response variable.

Penalty functions were developed for each response variable, based on the cost relative to that at a target value of the response. This provides a common basis for comparing the responses with each other. The targets, which are analogous to the setpoints of a controller, were chosen to maximise the financial return without running an undue risk of exceeding the specification limits. A cost penalty is incurred on deviation from the targets and the sum of the four penalties acts as the error signal in the quality control loop. The constituent penalty functions were set up to provide appropriate error feedback, and therefore do not necessarily reflect the true merit of the samples of powder. For example, a powder sample with the maximum permissable moisture content will incur a severe penalty because of the risk that it will be analysed as over the limit, even though the returns are greatest at this moisture content. For this reason a second function was developed to measure the true cost associated with each powder sample. This enables the performance of the quality control system to be assessed more realistically than by examination of the error function alone. These functions will now be considered individually.

8.4.1 Milk Solids Throughput

The throughput of milk solids is fixed by the evaporator feedrate and the skim milk total solids. The drier throughput cannot exceed that of the evaporator for long without the drier having to be switched over to water. Conversely, if the drier throughput is too low, concentrate will accumulate and the consequent age-thickening will cause problems. The following function expresses this.

$$P_G = W (T_G - G) \qquad \text{for } G \leq T_G$$
$$P_G = W (G - T_G)^2 \qquad \text{for } G > T_G$$

where

G = solids throughput kg/h

T_G = target for G

P_G = penalty for G

W = a weighting factor

This function is graphed for a target value of 160 kg/h and a weight of 0.05 in Figure 8.3.

8.4.2 Powder Moisture Content

Milk powder is sold by weight, so the higher the moisture content the higher the return for a given quantity of milk solids, always providing that the powder meets its specification. If the moisture content exceeds 4.0 % a lower price is paid and if the moisture is too high the product must be reprocessed before it may be sold. Taking a price of $ 525 per tonne for powder with a moisture content not greater than 4.0 % and $ 500 per tonne for powder with a higher moisture, the following penalty function expresses the cost in $/h relative to zero at the target moisture content T_M.

The weight of powder produced per hour at the target solids throughput T_G and moisture content T_M is given by:

$$H = T_G + (T_G T_M) / (100 - T_M)$$

The penalty function is then:

$$P_M = 0.00525\ H\ (T_M - M\ (100 - T_M) / (100 - M)) \qquad \text{for } M \leqslant T_M$$

$$P_M = 0.025\ H\ (M - T_M) / (4.1 - T_M) \qquad \text{for } T_M < M \leqslant 4.0\ \%$$

$$P_M = 0.025\ H \qquad \text{for } M > 4.0\ \%$$

For $T_M = 3.7\ \%$ and $T_G = 160$ kg/h, $H = 166.1$ kg/h and the function simplifies to:

$$P_M = 0.9\ (3.7 - M) \qquad \text{for } M \leqslant 3.7\ \%$$

$$P_M = 10.4\ (M - 3.7) \qquad \text{for } 3.7\ \% < M \leqslant 4.0\ \%$$

$$P_M = 4.15 \qquad \text{for } M > 4.0\ \%$$

The constant slope between the target and 4.1 % moisture reflects the increasing risk of exceeding the specification limit. The penalty function is graphed for the above target values in Figure 8.3.

8.4.3 Solubility Index

The Solubility Index scale may be divided into four ranges for payment purposes, with no penalty being incurred for SI values below the target and penalties increasing by stages as the SI increases.

$$P_{SI} = 0 \qquad \text{for } SI \leqslant T_{SI}$$

$$P_{SI} = 0.015\ H\ (SI - T_{SI}) / (0.6 - T_{SI}) \qquad \text{for } T_{SI} < SI \leqslant 0.5$$

$$P_{SI} = 0.015\ H \qquad \text{for } 0.5 < SI \leqslant 1.2$$

$$P_{SI} = 0.025\ H \qquad \text{for } 1.2 < SI \leqslant 2.0$$

This function is illustrated for the standard target values $T_{SI} = 0.3$ ml and $T_G = 160$ kg/h in Figure 8.3.

8.4.4 Bulk Density (100 taps)

An arbitrary decision was made to use the following penalty

function, on the assumption that a restricted range of bulk density was desirable, but that payment for the powder was not directly affected by the actual bulk density.

$$P_{BD} = 0 \qquad \text{for } |T_{BD} - BD| \leq 0.01 \text{ g/ml}$$

$$P_{BD} = 5\,(|T_{BD} - BD| - 0.01) \qquad \text{for } |T_{BD} - BD| > 0.01 \text{ g/ml}$$

In simulation this was found to provide sufficient incentive to move the bulk density to the target without interfering with the more important task of keeping the moisture and SI within specification. Figure 8.3 illustrates this function for the standard moisture and solids throughput targets.

8.4.5 The Aggregate Penalty Functions

The primary purpose of the penalty functions is to provide a means of ranking the samples of powder produced at each point in the simplex so that the plant settings may be moved away from undesirable values. This is achieved by adding the four penalties.

$$P_1 = P_G + P_M + P_{SI} + P_{BD}$$

It is also convenient to have a measure of the actual cost of each powder sample to gauge the effectiveness of the Simplex algorithm and the wisdom of the choice of the target values. Simply adding the penalties is inappropriate for two resaons. Powder which exceeds the specification limits in two respects is not doubly penalised by the payment formula, and the ramp functions between the targets and the specification limits are there to reflect risk and not the actual payment. A second penalty function was defined to measure the real merit of a powder sample. No account is taken of the solids throughput, since this is assumed to be determined by the evaporator in the long term, and there is no direct financial return from matching the capacities of the evaporator and drier. The bulk density penalty has been included because of the impact of the density on packaging and transport costs. The function is given below.

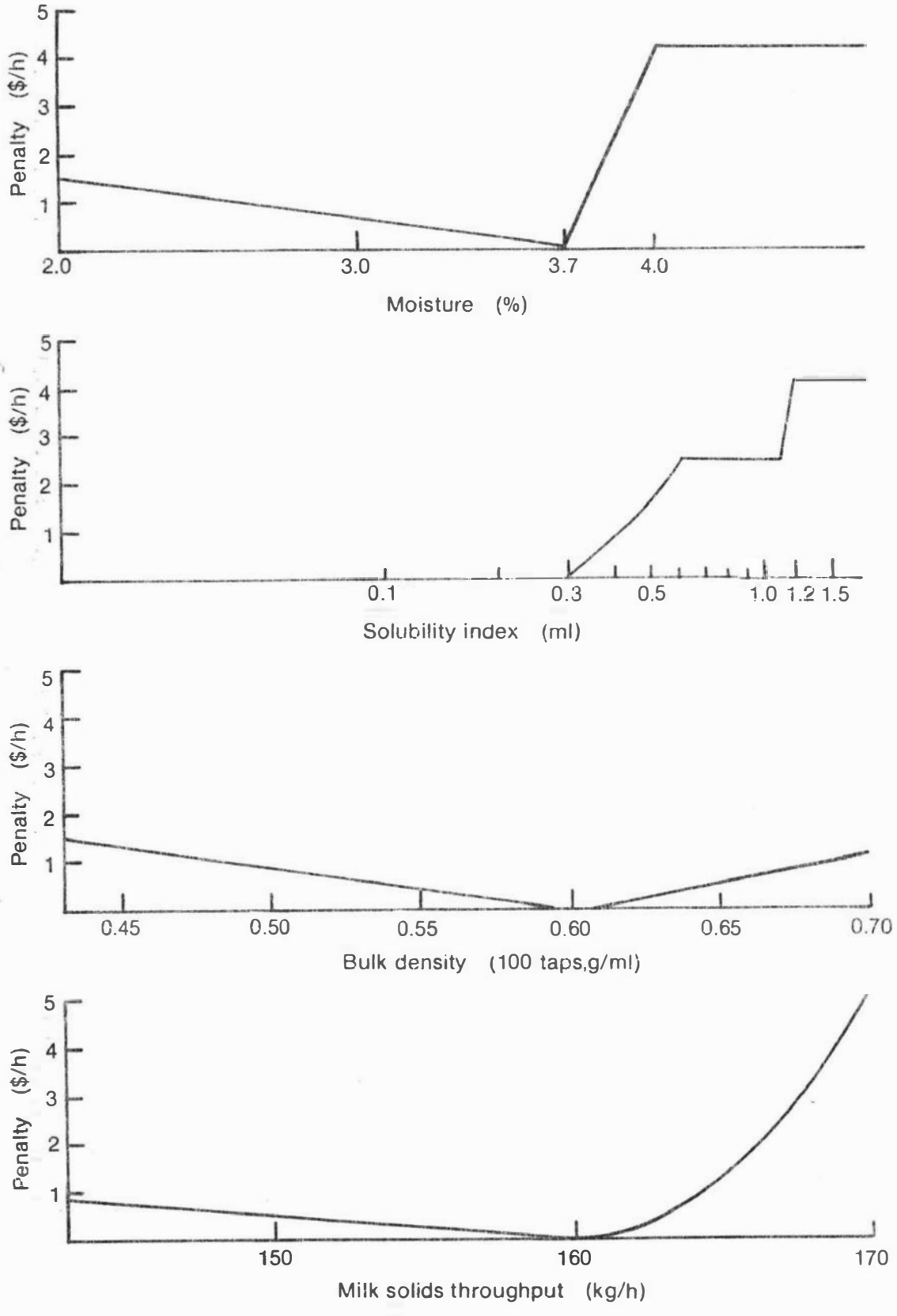


FIGURE 8.3 Graphs of penalty functions for moisture, Solubility Index, bulk density (100 taps) and milk solids throughput

$$P_2 = 0.00525\ H\ (4.0 - 96\ M/(100 - M)) + P_{BD}$$

for $M \leq 4.0$ % and $SI \leq 0.5$ ml

$$P_2 = 0.00510\ H\ (4.0 - 96\ M/(100 - M)) + 0.015\ H + P_{BD}$$

for $M \leq 4.0$ % and 0.5 ml $< SI \leq 1.2$ ml

$$P_2 = 0.00500\ H\ (4.0 - 96\ M/(100 - M)) + 0.025\ H + P_{BD}$$

for $M \leq 4.0$ % and 1.2 ml $< SI \leq 2.0$ ml

$$P_2 = 0.00500\ H\ (5.0 - 95\ M/(100 - M)) + 0.025\ H + P_{BD}$$

for 4.0 % $< M \leq 5.0$ % and $SI \leq 2.0$ ml

CHAPTER 9 - RESULTS AND DISCUSSION

The results of the pilot plant and simulation model trials of the Simplex EVOP scheme are presented in the form of bar charts. These show the deviation of moisture, Solubility Index, bulk density and milk solids throughput from their respective targets for each sample of powder. The penalty functions P_1 and P_2 are also graphed. The former is the error signal providing feedback to the quality control system, while the latter measures the true cost of each sample of product. Only 30 points are graphed because the acceptability of the Simplex scheme hinges on how rapidly the quality variables can be brought to the targets, and it is possible to assess this from 30 points. The long term behaviour of the control system has been assessed by integrating P_2 over 300 points. In each set of bar charts, the first k+1 points comprising the initial simplex are separated from the rest by a dashed line. Because these points are pre-determined, systematic improvement can only occur from point k+2 onwards. The specification limits of 4.0 % moisture and 0.5 ml SI are indicated by dotted lines.

9.1 Pilot Plant Trial Results

The Simplex evolutionary operation scheme described in Chapter 8 was evaluated in the course of four eight hour runs on the pilot plant. By using rapid methods of analysis, and by taking advantage of the fact that the Simplex scheme provides two sets of operating conditions in advance, the time between samples was reduced to about 25 minutes. This meant that allowing for start-up and process upsets, from 15 to 18 powder samples could be produced each day. A Delavan SB 54 nozzle was used in all the runs. The results are tabulated in Appendix VII.

On the first day three variables were manipulated; inlet air temperature, concentrate total solids and concentrate feedrate. The concentrate temperature was maintained at 44 C. The initial simplex straddled the mean values of the manipulated variables for the main seasonal experiment. The targets for moisture, SI, bulk density and milk solids throughput were 3.7 %, 0.3 ml, 0.60 g/ml and 160 kg/h respectively. Figure 9.1 shows the results of the 16 runs performed.

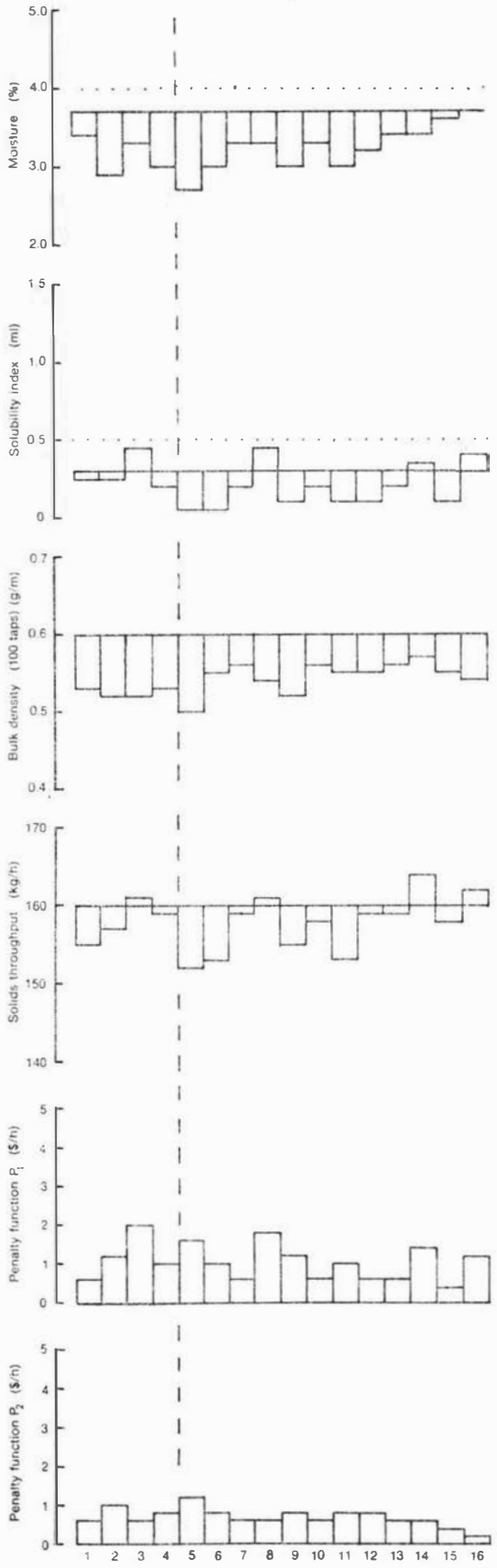


FIGURE 9.1 The results of the first day of the pilot plant trial of the Simplex scheme

The initial powder quality was within specification, with the moisture and bulk density both low. The moisture gradually rose towards its target while the SI and solids throughput were maintained at acceptable levels. Little improvement in bulk density was evident, reflecting the small penalty applied to deviations of this variable from its target. The second penalty function P_2 steadily improved. Even in only 12 moves the systematic improvement obtained without any violent change in the product quality is significant.

On the second day, four manipulated variables were used; inlet air temperature and the total solids, feedrate and temperature of the concentrate. Eighteen samples were obtained, representing 13 moves. Again, the initial simplex was close to the mean values of the variables in the main seasonal experiment, and the powder quality lay well inside the specification limits. The targets for all the quality variables were the same as for the first day. The results are graphed in Figure 9.2. On the fourth move the moisture content rose to 4.1 %, outside the limit. Instead of reflecting the second worst point in the current simplex, the second worst point in the previous simplex was reflected by mistake. The effects of this became apparent two moves later, when point five had to be repeated because it had reached the maximum age limit. Two further points required repetition during the day, slowing the rate of progress. Notwithstanding the incorrect application of Rule 3, the process was swiftly moved away from the specification limits. The results of this set of runs illustrate the behaviour of the scheme when the powder quality is nearly on target.

The third and fourth days were run together to investigate the effectiveness of the Simplex scheme in getting a product initially out of specification, into specification and to examine the effect of introducing disturbances, once good quality powder had been achieved. The four variables used on the second day were manipulated. The targets were also the same. The initial conditions were chosen to give low moistures and high Solubility Indices. The first 17 samples were produced from factory supply milk on the third day, and the remaining 15 were produced from town supply milk with a lower protein content on the fourth day. A further disturbance was introduced after the 27th sample by changing the preheat temperature from 100 C to 114 C. The preheat

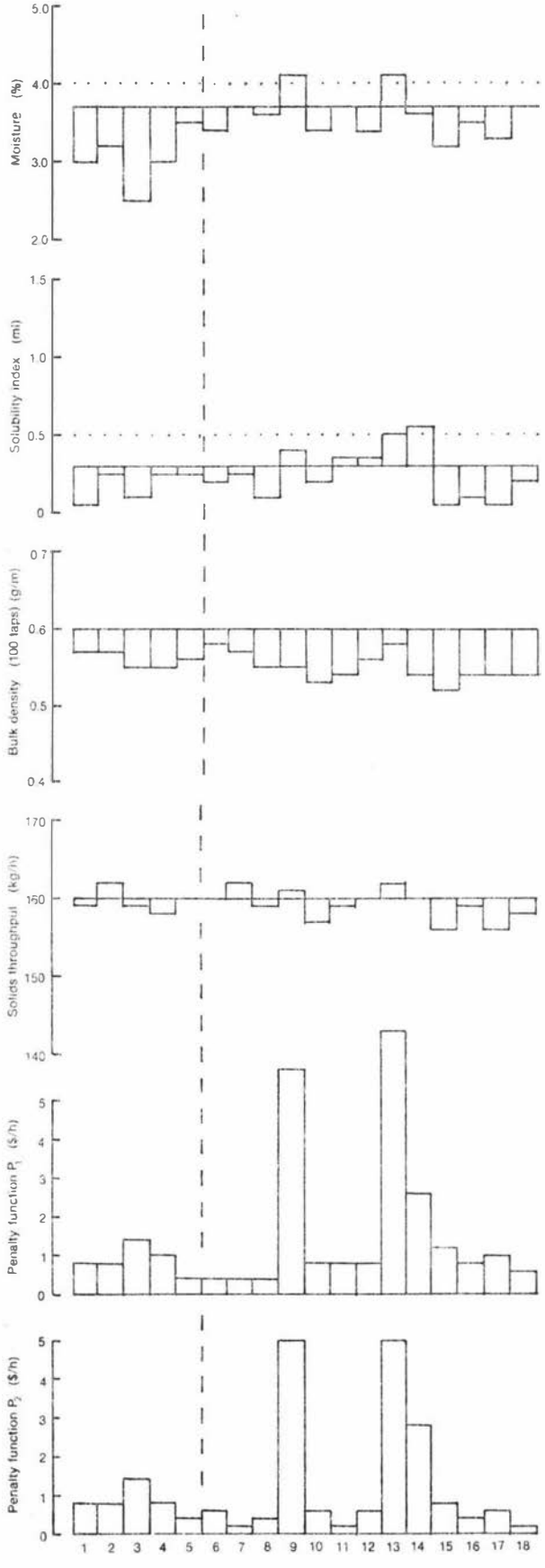


FIGURE 9.2 The results of the second day of the pilot plant trial of the Simplex scheme

holding time was 10 seconds for all the runs.

The results are graphed in Figure 9.3. Three moves were sufficient to bring the powder within specification. The moisture was subsequently kept very close to the target. There was a small rise at the point when the source of the milk was changed. The SI also exhibited a rise at this point, in this case taking the SI out of specification. The bulk density of the powder did not appear to be affected by the change, but gradually improved to the extent that the penalty associated with it was zero for 9 of the last 12 samples. The milk solids throughput varied much more than it did on the first two days, and was always below the target. The rapid response of the optimisation scheme to the disturbances, and its success in maintaining the quality of the powder close to the targets is apparent.

9.2 - Simulation Results

Commercial application of the Simplex EVOP scheme would not, in general, be preceeded by detailed process modelling. Indeed, the major benefit of evolutionary operation methods is their ability to optimise processes whose characteristics are largely unknown. This implies that the selection of the step sizes for the variables to be manipulated must be made on the basis of informed guess-work.

In order to examine the sensitivity of the Simplex scheme to the choice of step size, a replicated three level full factorial experiment in step size was run on the simulation model. The sizes were 0.67, 1.0 and 1.5 times those given in Table 8-2, representing a $\pm$ 50 % variation about the values chosen for the pilot plant trial. The response was the ratio of the average penalty P_2 over the final simplex to that over the initial simplex, after 25 moves. This ratio was then multiplied by 100 to give the final cost as a percentage of the initial cost. In all cases, the initial product quality was within specification, although the moisture and SI were some distance away from their targets. The mean percentage improvement over the two replicates was taken, to reduce the effect of the analytical error added to the model. Occasionally one of the points in an initial simplex gave out of specification product

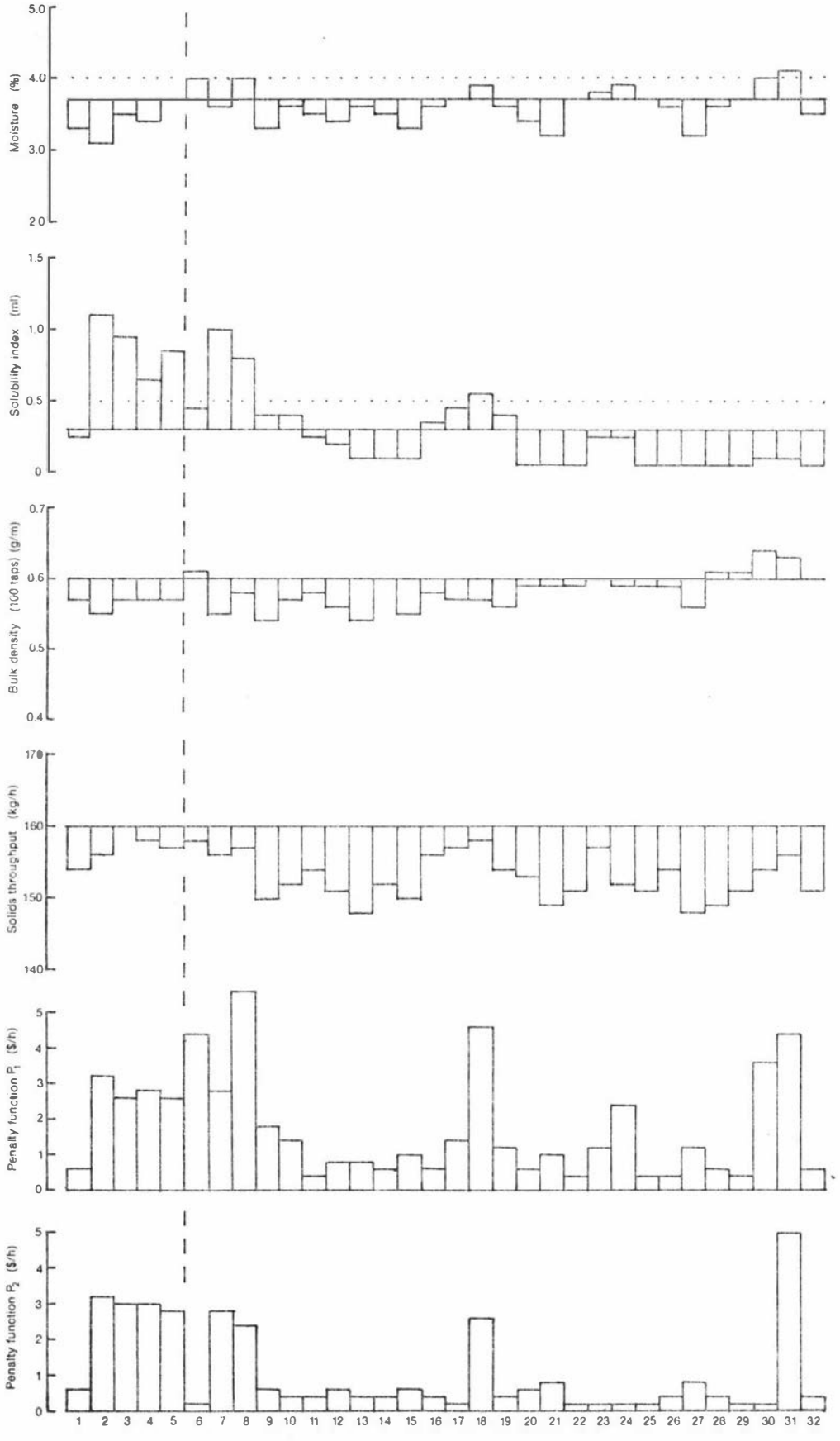


FIGURE 9.3 The results of the third and fourth days of the pilot plant trial of the Simplex scheme

quality, in which case that run was repeated. The experiment was carried out for two sets of manipulated variables, one with three and one with four independent variables. The results are summarised in Table 9-1.

The average percentages cannot be compared between the two options because the degree of improvement depended very much on how far from the targets the initial quality was. If the product quality is exactly on target, P_2 = 0.27 \$/h. The average of P_2 over the initial simplices was 0.61 \$/h for option one and 0.67 \$/h for option four, so the percentages expected if all the points in the final simplices were on target are 44 % and 40 % respectively.

TABLE 9-1 The Effect of Departures from the Optimal Step Sizes

Option 1	Final Average cost P_2 as a Percentage of the Initial Average P_2 after 25 moves								
Variable	Inlet Temperature (C)			Total Solids (%)			Feedrate (l/h)		
Step sizes	2.67	4.00	6.00	1.00	1.50	2.25	2.67	4.00	6.00
Percentage	57	51	82	66	54	70	83	48	59

Option 4	Final Average cost P_2 as a Percentage of the Initial Average P_2 after 25 moves					
Variable	Inlet Temperature (C)			Total Solids (%)		
Step sizes	2.67	4.00	6.00	1.00	1.50	2.25
Percentage	73	70	71	71	71	72
Variable	Feedrate (l/h)			Concentrate Temperature (C)		
Step Sizes	2.67	4.00	6.00	2.67	4.00	6.00
Percentage	68	76	70	68	72	74

Option one uses three manipulated variables, and the improvement over 25 moves is clearly best for the step sizes given in Table 8-2. Too large a step size for the inlet air temperature, or too small a step size for the concentrate feedrate gave particularly poor results. Otherwise, the improvement obtained did not vary greatly with changing step size, considering the effect measurement "noise" had on the response.

Four variables were manipulated in option four, and the effects of varying the step sizes were not significant, except for the small improvement in using a concentrate feedrate step larger or smaller than the 4 l/h chosen for the pilot plant trial.

These results are encouraging, since they demonstrate that the Simplex EVOP procedure is capable of improving the product quality even when the step sizes selected are not optimal. This is important if the technique is to win acceptance in a commercial environment in which accurate estimation of the best step sizes is difficult and modification of the sizes once the EVOP scheme has been introduced is impractical.

The effect of varying the target values was also examined using the simulation model. A three level factorial experiment in the moisture and SI target values was carried out for the same two options used above. The response variable was the sum of the penalty function P_2 over 300 points. In this way the long term benefits of high targets could be judged against the increased risk of exceeding the specification limits The bulk density target was 0.60 g/ml and the solids throughput target was 160 kg/h in both cases. Table 9-2 gives the sum of P_2 for each combination of target values. If all 300 points had been on target, the sum would be 81. The number of times the product exceeded specification is given in brackets after the integrated P_2 value.

The choice of target values had more effect on the performance of the optimisation scheme than the choice of step sizes. When the moisture target was too close to the specification limit of 4.0 %, the incidence of out of specification product and the integrated penalty function both increased significantly. The effect of varying the SI target was less pronounced, probably because the SI penalty contributes to P_2 only when the SI exceeds the target. The Sums of P_2 are considerably larger than

TABLE 9-2 The Effect of Changing the Moisture and SI Targets on the Sum of P_2 Over 300 Points and the Number of Times the Product Exceeded Specification

	Option 1 T, TS, F manipulated			Option 4 T, TS, F T_c manipulated		
SI Target	Moisture Target 3.6	 3.7	 3.8	Moisture Target 3.6	 3.7	 3.8
0.2	181 (5)	177 (6)	185 (11)	174 (1)	190 (9)	233 (19)
0.3	183 (6)	173 (6)	214 (20)	172 (2)	167 (6)	191 (13)
0.4	171 (3)	211 (13)	204 (18)	180 (4)	166 (8)	172 (11)

the expected long-term value of 81. The reason for this is that the step sizes were optimised for rapid improvement in product quality over 25 moves. When smaller steps were tried over 300 points, the sum of P approached 81, but the initial improvement was markedly slower.

The results show the dangers of selecting targets to close to the specification limits. When the targets were at or below the 3.7 % moisture and 0.3 ml SI chosen for the pilot plant trial, however, the performance of the scheme was not greatly affected by the exact choice of target levels. This was to be expected from the form of the penalty functions.

These two simulation model experiments have demonstrated the robust nature of the Simplex Evolutionary Operation method.

Finally, the last two days of the pilot plant trial were simulated without introducing changes in milk composition or preheat treatment. Figure 9.4 shows the results. The overall pattern of the bar charts is similar to that of Figure 9.3, with the moisture content remaining close to the target while the SI was rapidly brought under control. The bulk density rose by an amount similar to that seen in Figure 9.3, but the average level was 0.02 g/ml higher. The milk solids throughput behaved almost exactly as on the pilot plant trial.

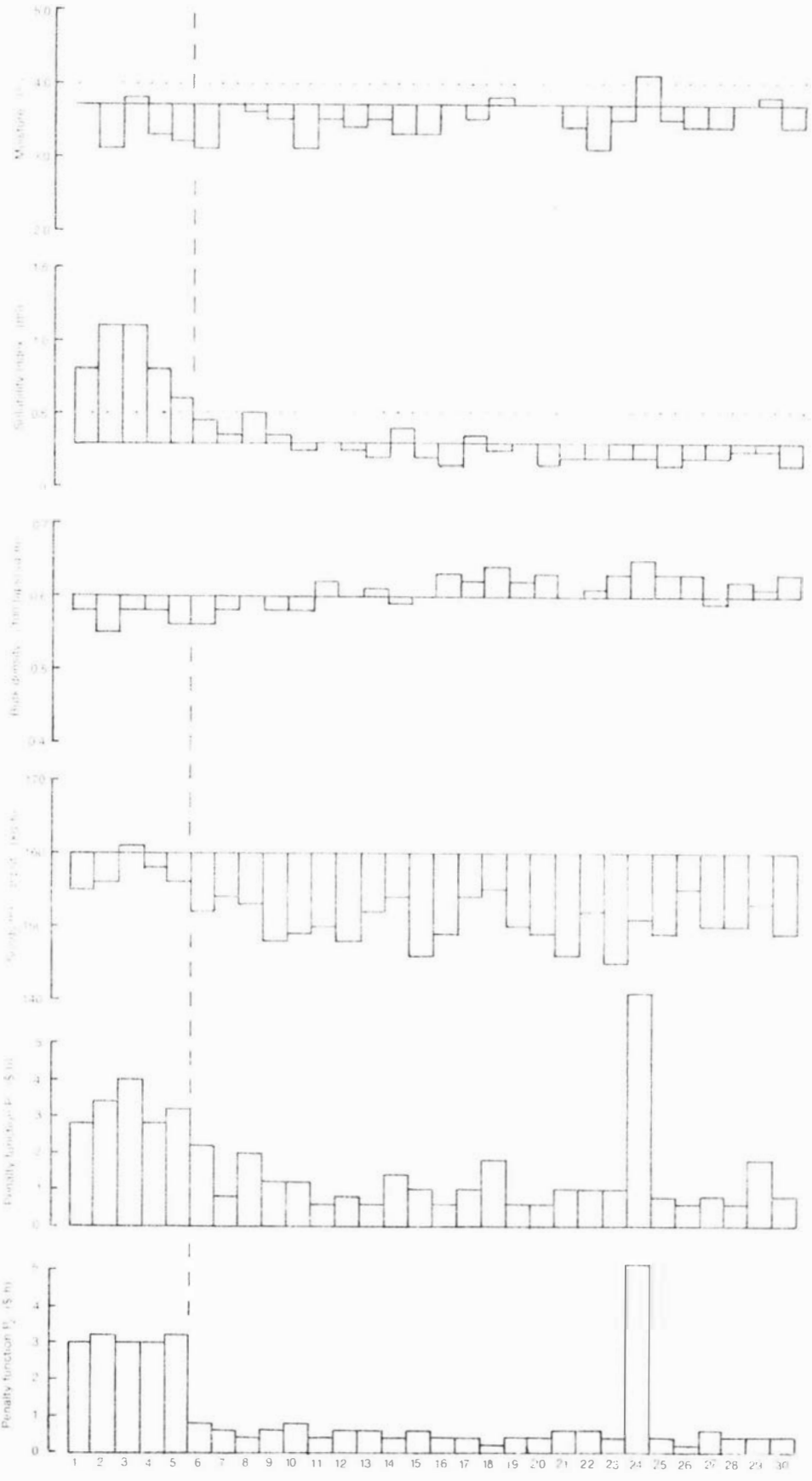


FIGURE 9.4 The results of a simulation model run with the same initial conditions as the third and fourth days of the pilot plant trial of the Simplex scheme

The close correspondence between the results obtained with the quality control system on the simulation model and on the pilot scale drier trial is evidence of the accuracy of the model. It also supports the application of conclusions about the sensitivity of the Simplex scheme's performance to step size and target value changes obtained from simulation studies to the actual process. Nozzle atomising spray driers of widely differing capacities show qualitatively similar behaviour, provided that they employ nozzles with similar viscosity sensitivity. This has been observed in many of the installations listed in Table 2-4. The Simplex evolutionary operation technique described here could therefore be expected to perform well on a full-size spray drier. It should be noted that the quality control system has been optimised for rapid initial improvement and response to disturbances. The step sizes are larger than desirable for use in very long runs under mid-season conditions, when the milk composition is nearly constant.

PART III CONCLUSIONS

CHAPTER 10 - CONCLUSIONS

A number of conclusions may be drawn from the work presented here concerning the effectiveness of the methods used to build the drier model, the insights gained from the model, the trial of the Simplex EVOP procedure and the application of this procedure to commercial driers.

The combination of Response Surface Methodology with the systems analysis technique of Rudd and Watson has proved to be a powerful technique for empirical model building. The use of a systematic approach to the selection of independent variables for the experimental programme guarranteed that a necessary and sufficient set of equations describing the spray drier could be obtained by performing the minimum number of experiments. Second order polynomial models in five operating variables were fitted to the experimental data. These variables, the inlet air temperature and the concentrate total solids, feedrate, atomising pressure and temperature, proved to be sufficient to describe the drier performance and to predict the properties of the powder.

The drier studies confirmed the importance of low concentrate viscosity in the production of good quality milk powder. This could be achieved by keeping concentrate holding times to a minimum and by using high temperature, short time preheat treatments. The protein content of the skim milk was found to be the major determinant in the seasonal changes observed in concentrate viscosity, high protein contents giving high viscosities.

The study of the hydrodynamics of centrifugal pressure nozzle atomisers has revealed that the nozzles used in spray dried milk powder manufacture fall into two distinct categories, each with characteristic behaviour in response to variations in fluid viscosity. The magnitude of the viscosity effect appears to be proportional to the ratio of the swirl chamber and orifice diameters. The large capacity nozzles used in tall-form driers exhibit a marked decrease in pressure drop at constant flowrate as the viscosity of the concentrate fed to them is increased.

The spray drier studies showed that the sensitivity of the nozzle to viscosity changes plays a very important part in determining the overall behaviour of the drier. Simulation studies of two outlet air temperature control strategies clearly demonstrated the superiority of inlet air temperature manipulation over that of concentrate feedrate, for driers employing large capacity nozzles.

The Simplex evolutionary operation method was found to be a simple robust procedure which rapidly improved the product quality and maintained it in the face of disturbances typical of those likely to occur in commercial operation. The close correspondence between the results of the pilot plant trial and the simulation runs demonstrated the accuracy of the drier model and the efficacy of the Simplex scheme.

The Simplex method provides two sets of plant conditions in advance. This feature permits a substantial increase in the speed of attainment of optimum conditions for processes with setpoint response times similar to the time required to analyse the product quality. The Simplex method is therefore particularly suitable for application to the manufacture of spray dried milk powders.

APPENDIX I - Pilot Plant Equipment

Plant Item	Manufacturer	Model
Three effect falling-film evaporator	Wiegand Apparatebau G.m.b.H., Karlsruhe, Germany	
Pilot tall-form spray drier	The De Laval Separator Co., Spray Dryer Division, River Falls, Wisconsin, U.S.A.	
Centrifugal pump	F. Stamp G.m.b.H., Hamburg, Germany	Fristam FP 722
Plate heat exchanger	A.P.V., Crawley, Sussex, U.K.	
Swept-surface heat exchanger	Crepaco Inc., Chicago, Illinois, U.S.A.	1 VT-422
Gear pump	Stainless Steel Pumps Ltd., Eastbourne, Sussex, U.K.	1/2 inch Handipump
High pressure pump	APV-Manton Gaulin Crawley, Sussex, U.K.	KL3-5PS
Variable speed gearbox for high pressure pump	Carter Gears Ltd, Bradford, Yorkshire, U.K.	AM 26
Centrifugal pressure nozzles	Delavan Manufacturing Company, West Des Moines, Iowa, U.S.A.	SDX Series
Centrifugal pressure nozzles	Spraying Systems Co., Bellwood, Illinois, U.S.A.	SX Series

APPENDIX II - Pilot Plant and Laboratory Instrumentation

Measurement and Calibrated range	Instrument Type	Manufacturer	Model
Drier air flowrate 0 to 248 kg/min	Differential pressure & orifice plate	Foxboro Company Ltd., La Salle, Quebec, Canada	E13 DL
Gas flowrate 0 to 34 Nm3/h	Differential pressure & orifice plate	Foxboro Company Ltd., La Salle, Quebec, Canada	E13 DL
Concentrate flowrate 0 to 800 l/h	Magnetic flowmeter	The Foxboro Co., Foxboro, Massachusetts, U.S.A.	2801-DTCC-SS with 696 A converter
Concentrate flowrate 0 to 1600 l/h	Magnetic flowmeter	The Foxboro Co., Foxboro, Massachusetts, U.S.A.	2801-DTCC-SS with E96 converter
Atomising pressure 0 to 34.5 MPa	Gauge pressure	Foxboro Company Ltd., La Salle, Quebec, Canada	E11 GH
Drier inlet duct pressure 61 to 114 kPa	Absolute pressure	Foxboro Company Ltd., La Salle, Quebec, Canada	E11AM
Drier chamber pressure -0.6 to +0.6 kPa	Gauge pressure	Foxboro Company Ltd., La Salle, Quebec, Canada	E17 DL
Ambient air absolute humidity 0.0026 to 0.1280 kg/kg dry air	Dew point measuring system	Foxboro Proprietary Ltd., Lilydale, Victoria, Australia	Dewcell 2711 AG with E94 transmitter
Concentrate density 950 to 1200 kg/m3	Twin-tube vibrating densitometer	The Solartron Electronic Group Ltd., Farnborough, Hampshire, U.K.	NT 1762 with LT 1761 converter
Concentrate density 950 to 1350 kg/m3	Vibrating vane densitometer	Barton ITT, City of Industry California, U.S.A.	662 with 653 converter
Concentrate density 950 to 1350 kg/m3	Vibrating U-tube densitometer	Automation Products Inc., Houston, Texas, U.S.A.	Dynatrol CL-10TY with EC-213-GA-10 converter
Concentrate viscosity 60 to 4000 cp	Rotating bob viscometer	Contraves AG, Zurich, Switzerland	DC20 STV-fc

Measurement and Calibrated range	Instrument Type	Manufacturer	Model
Concentrate viscosity 22 to 450 cp	Rotating bob viscometer	Contraves AG, Zurich, Switzerland	DC30 STV-fc
Powder Moisture	Automatic Karl-Fischer Titrator	Metrohm, Herisau Switzerland	E547/2
Bulk density	Stamp volumeter	J. Engelsmann A.G. Apparatebau, Ludwigshafen am Rhein, Germany	
Particle Density	Air pycnometer	Micromeritics Instrument Corp., Atlanta, Georgia, U.S.A.	1302
Nitrogen content	Automatic Kjeldahl Apparatus	A/S Foss Electric, Hillerod, Denmark	Kjelfoss

APPENDIX III - Experimental Design Matrices

Fractional 3 Level 4 Factor Factorial
(Box and Behnken, 1960)

X(1)	X(2)	X(3)	X(4)	
-1	-1	0	0	
+1	-1	0	0	
-1	+1	0	0	
+1	+1	0	0	
0	0	-1	-1	Block 1
0	0	+1	-1	
0	0	-1	+1	
0	0	+1	+1	
0	0	0	0	
-1	0	0	-1	
+1	0	0	-1	
-1	0	0	+1	
+1	0	0	+1	
0	-1	-1	0	Block 2
0	+1	-1	0	
0	-1	+1	0	
0	+1	+1	0	
0	0	0	0	
0	-1	0	-1	
0	+1	0	-1	
0	-1	0	+1	
0	+1	0	+1	
-1	0	-1	0	Block 3
+1	0	-1	0	
-1	0	+1	0	
+1	0	+1	0	
0	0	0	0	

This design was replicated eleven times throughout the 1977/78 and 1978/79 dairying seasons. The levels of the variables were as follows.

X(1) = Inlet Air Temperature (C)	195 (-1)	210 (0)	225 (+1)
X(2) = Concentrate total solids (%)	45.7 (-1)	47.7 (0)	49.7 (+1)
X(3) = Concentrate flowrate (l/h)	300 (-1)	320 (0)	340 (+1) *
X(4) = Atomising Pressure (MPa)	20 (-1)	24 (0)	28 (+1)

* Note these were the targets before adjustment of the flowmeter calibration figures.

For Replicates 1 to 5 the true levels were: 251.4 267.3 283.1 l/h
For Replicates 6 to 11 the true levels were: 266.2 285.5 303.2 l/h

Other conditions held constant:

Preheat temperature	110 C
Preheat holding time	10 s
Nozzle	Delavan SDX SB 54

Design Matrix For Experiment to Determine the Effect of
Preheat Treatment on Concentrate Viscosity (5, 6 March 1980)

2 Level 4 Factor Full Factorial

X(1)	X(2)	X(3)	X(4)	
-1	-1	-1	-1	
-1	+1	-1	-1	
-1	-1	+1	+1	
-1	+1	+1	+1	
+1	+1	+1	+1	Block 1
+1	-1	+1	-1	
+1	-1	-1	+1	
+1	+1	-1	-1	
+1	+1	-1	+1	
+1	+1	+1	-1	
+1	-1	-1	-1	
+1	-1	+1	+1	
-1	-1	-1	+1	Block 2
-1	+1	+1	-1	
-1	-1	+1	-1	
-1	+1	-1	+1	

X(1) = Preheat holding time (s) 10 (-1) or 120 (+1)
X(2) = Preheat temperature (C) 80 (-1) or 113 (+1)
X(3) = Concentrate total solids (%) 47.4 (-1) or 49.1 (+1)
X(4) = Concentrate temperature (C) 45 (-1) or 60 (+1)

A fifth variable was obtained by measuring the reponse (viscosity) in two locations in the concentrate line;

X(5) = Concentrate holding time (s) 150 (-1) or 300 (+1)

Other conditions held constant:

Concentrate flowrate 288 l/h

Design for Experiment to Determine the Effects of Drier Throat Diameter and Nozzle Position (29 March 1979)

Replicated Mixed 2 and 3 level Factorial

X(1)	X(2)	
-1	+1	
-1	-1	
-1	O	
+1	-1	Replicate 1
+1	O	
+1	+1	
---	---	
+1	+1	
+1	O	
+1	-1	
-1	-1	Replicate 2
-1	O	
-1	+1	

X(1) = Throat diameter (mm) 203 (-1) or 305 (+1)
X(2) = Nozzle position (mm) -80 (-1), 0 (O) or +80 (+1)

Other conditions held constant:

Preheat temperature	110 C
Preheat holding time	10 s
Air inlet temperature	210 C
Concentrate total solids	49.6 %
Concentrate flowrate	266 l/h
Atomising pressure	20 Mpa
Concentrate temperature	40 C
Concentrate viscosity	60 cp
Nozzle	Delavan SDX SB 54

Design Matrix For Experiment to Investigate the Effects of Changing Nozzle, Nozzle Position and Atomising Pressure (27 February 1978)

Mixed 2 Level 2 Factor and 3 Level 1 Factor Factorial

X(1)	X(2)	X(3)
-1	-1	-1
-1	-1	0
-1	-1	+1
-1	+1	-1
-1	+1	0
-1	+1	+1
+1	-1	-1
+1	-1	0
+1	-1	+1
+1	+1	-1
+1	+1	0
+1	+1	+1

X(1) = Nozzle SB 54 (-1) or SA 69 (+1)
X(2) = Atomising pressure (MPa) 15 (-1) or 25 (+1)
X(3) = Nozzle position (mm) -80 (-1), 0 (0) or +80 (+1)

Other conditions held constant:

Preheat temperature	110 C
Preheat holding time	10 s
Concentrate total solids	47.8 % and 49.0 %
Concentrate feedrate	267 l/h
Air Inlet Temperature	210.0 C and 201.3 C

Design Matrix for two Experiments Involving Nozzles

2 Level 3 Factor Full Factorial

X(1)	X(2)	X(3)
-1	-1	-1
-1	-1	+1
-1	+1	-1
-1	+1	+1
+1	-1	-1
+1	-1	+1
+1	+1	-1
+1	+1	+1

First Experiment (11 October 1978)

X(1) = Air inlet temperature (C)	195 (-1)	or	225 (+1)
X(2) = Nozzle orifice size	52 (-1)	or	50 (+1)
X(3) = Viscosity (cp)	30 (-1)	or	80 (+1)

Other conditions held constant:

Preheat temperature	110 C
Preheat holding time	10 s
Concentrate total solids	47.9 %
Atomising pressure	22 MPa

Second Experiment (Replicated 13, 14 December 1978)

X(1) = Swirl chamber	SA (-1)	or	SB (+1)
X(2) = Orifice size	54 (-1)	or	61 (+1)
X(3) = Concentrate flowrate (l/h)	268 (-1)	or	287 (+1)
(Atomising pressure (MPa))	19	or	23

Other conditions held constant:

Preheat temperature	110 C
Preheat holding time	10 s
Air inlet temperature	210 C
Concentrate total solids	48.4 %

APPENDIX IV Example of Statistical Model Building

The following is an example of the statistical model building procedure. The response variable is the powder moisture content multiplied by ten. The data from all 301 samples made in the main seasonal experiment are included. The independent variables have been scaled as follows.

Air inlet temperature	T = (T-210)/15
Concentrate total solids	TS = (TS-47.7)/2
Concentrate flowrate	F = (F-279)/20
Atomising pressure	P = (P-24)/4
High pressure conc. temp.	Thp = (Thp-43.7)/10

Multiple Correlation 0.9489

Analysis of Variance

Source	df	SS	MS	F		
TOTAL	300	25533.9000				
REGRESSION	16	24228.8000	1514.3000	329.5110	***	
X(1)	1	9093.6200	9093.6200	1978.7700	***	T
X(2)	1	1203.9300	1203.9300	261.9750	***	TS
X(3)	1	7193.3800	7193.3800	1565.2800	***	F
X(4)	1	4587.3400	4587.3400	998.2040	***	P
X(5)	1	45.3454	45.3454	9.8672	**	T.TS
X(6)	1	49.8848	49.8848	10.8549	**	T.F
X(7)	1	33.9743	33.9743	7.3928	**	T.P
X(8)	1	141.3240	141.3240	30.7522	***	TS.F
X(9)	1	23.9949	23.9949	5.2213	*	TS.P
X(10)	1	264.5370	264.5370	57.5632	***	F.P
X(11)	1	40.6354	40.6354	8.8423	**	T.T
X(12)	1	0.1383	0.1383	0.0301	ns	TS.TS
X(13)	1	12.9969	12.9969	2.8281	10%	F.F
X(14)	1	28.1082	28.1082	6.1163	*	P.P
X(15)	1	1459.4200	1459.4200	317.5700	***	Thp
X(16)	1	50.1541	50.1541	10.9135	**	Thp.Thp
RESIDUALS	284	1305.1500	4.5956			

Coefficients

	B(i)	Variance	T value		
B(0)	34.9316				
B(1)	-8.3700	0.0368	-43.6268	***	T
B(2)	-0.0763	0.0663	-0.2964	ns	TS
B(3)	4.7078	0.0595	19.3038	***	F
B(4)	-3.0074	0.0619	-12.0918	***	P
B(5)	0.6541	0.0866	2.2229	*	T.TS
B(6)	-1.0639	0.0820	-3.7159	***	T.F
B(7)	0.9146	0.0967	2.9409	**	T.P
B(8)	-1.5063	0.1080	-4.5837	***	TS.F
B(9)	0.4700	0.0988	1.4951	ns	TS.P
B(10)	-1.4635	0.0939	-4.7761	***	F.P
B(11)	0.9608	0.0651	3.7661	***	T.T
B(12)	-0.0237	0.0512	-0.1049	ns	TS.TS
B(13)	0.4179	0.0490	1.8885	10%	F.F
B(14)	0.3276	0.0451	1.5429	ns	P.P
B(15)	-3.8730	0.0519	-17.0075	***	Thp
B(16)	0.4396	0.0177	3.3035	**	Thp.Thp

Residuals

	Y	Yhat	Residual
Max	64.1000	63.3060	7.8247
Min	18.3000	19.5993	-7.1921

This full model including all product and squared terms may now be reduced using a forward selection algorithm to identify those terms which are not of sufficient significance to warrant inclusion in the final model.

Forward Selection

SS Total 25533.40000

Pass	Var Added	SSReg	RMS	R-Square	partial-F	
1	15	11282.6000	47.6613	0.4419	236.72500	***
2	1	20342.9000	17.4177	0.7967	520.17500	***
3	3	22279.3000	10.9566	0.8726	176.73200	***
4	4	23543.0000	6.7244	0.9220	187.92600	***
5	16	23842.2000	5.7329	0.9338	52.19330	***
6	11	23928.0000	5.4604	0.9371	15.72200	***
7	8	23992.9000	5.2576	0.9397	12.34070	***
8	10	24072.8000	5.0020	0.9428	15.97250	***
9	6	24136.3000	4.8010	0.9453	13.22260	***
10	7	24170.5000	4.6996	0.9466	7.27658	**
11	5	24195.0000	4.6312	0.9476	5.28587	*
12	13	24209.4000	4.5972	0.9481	3.13738	10%
13	9	24216.7000	4.5876	0.9484	1.60466	ns
14	14	24228.2000	4.5636	0.9489	2.50448	ns
15	2	24228.6000	4.5781	0.9489	0.09441	ns
16	12	24228.7000	4.5941	0.9489	0.01093	ns

The last four variables (TS.P, P.P, TS and TS.TS) are not significant at the 10% level and so will be deleted from the model. This is accomplished by moving the variables as shown below and running the regression program again.

Transformations

TCODE	Var No.	Old Var No.	Term
Y	13	17	
M	2	11	
M	9	13	
M	11	15	
M	12	16	

Multiple Regression Multiple Correlation 0.9481

Analysis of Variance

Source	df	SS	MS	F		
TOTAL	300	25533.9000				
REGRESSION	12	24209.5000	2017.4600	438.7050	***	
X(1)	1	9093.6200	9093.6200	1977.4500	***	T
X(2)	1	48.7274	48.7274	10.5960	**	T.T
X(3)	1	7363.0300	7363.0300	1601.1200	***	F
X(4)	1	3935.1500	3935.1500	855.7150	***	P
X(5)	1	30.1785	30.1785	6.5624	*	T.TS
X(6)	1	45.7940	45.7940	9.9581	**	T.F
X(7)	1	58.9506	58.9506	12.8190	***	TS.F
X(8)	1	82.4873	82.4873	17.9372	***	TS.P
X(9)	1	4.1237	4.1237	0.8967	ns	F.F
X(10)	1	398.2180	398.2180	86.5941	***	F.P
X(11)	1	3052.7800	3052.7800	663.8400	***	Thp
X(12)	1	96.4505	96.4505	20.9735	***	Thp.Thp
RESIDUALS	288	1324.4200	4.5987			

Coefficients

	B(i)	Variance	T value		
B(0)	34.9788				
B(1)	-8.3934	0.0367	-43.8175	***	T
B(2)	0.9509	0.0641	3.7560	***	T.T
B(3)	4.6560	0.0457	21.7891	***	F
B(4)	-2.9430	0.0444	-13.9609	***	P
B(5)	0.6760	0.0863	2.3004	*	T.TS
B(6)	-1.0537	0.0820	-3.6801	***	T.F
B(7)	0.9237	0.0962	2.9781	**	T.P
B(8)	-1.4207	0.0952	-4.6045	***	TS.F
B(9)	0.3830	0.0468	1.7710	10%	F.F
B(10)	-1.3166	0.0856	-4.4988	***	F.P
B(11)	-3.9818	0.0252	-25.0746	***	Thp
B(12)	0.5041	0.0121	4.5797	***	Thp.Thp

Residuals

	Y	Yhat	Residual
Max	64.1000	62.8542	8.5146
Min	18.3000	19.6052	-6.5817

This is the final model in its reduced form. This procedure has been used in fitting all the models derived from the experimental work.

APPENDIX V Methods for laboratory analyses

Size Analysis

The Andreasen pipette size analysis method involves sampling an intiially uniform suspension of powder particles in isopropyl alcohol after 2, 4, 8, 16, 32 and 64 minutes of sedimentation. The particle size represented by each sample is calculated by the formula:

$$d = \frac{175\sqrt{\mu h}}{t\,(\rho_p - \rho_f)}$$

where μ = viscosity of the isopropyl alcohol (poise)
h = height of alcohol column above pipette (cm)
t = sampling time (min)
ρ_p = density of powder (g/ml)
ρ_f = density of isopropyl alcohol (g/ml)

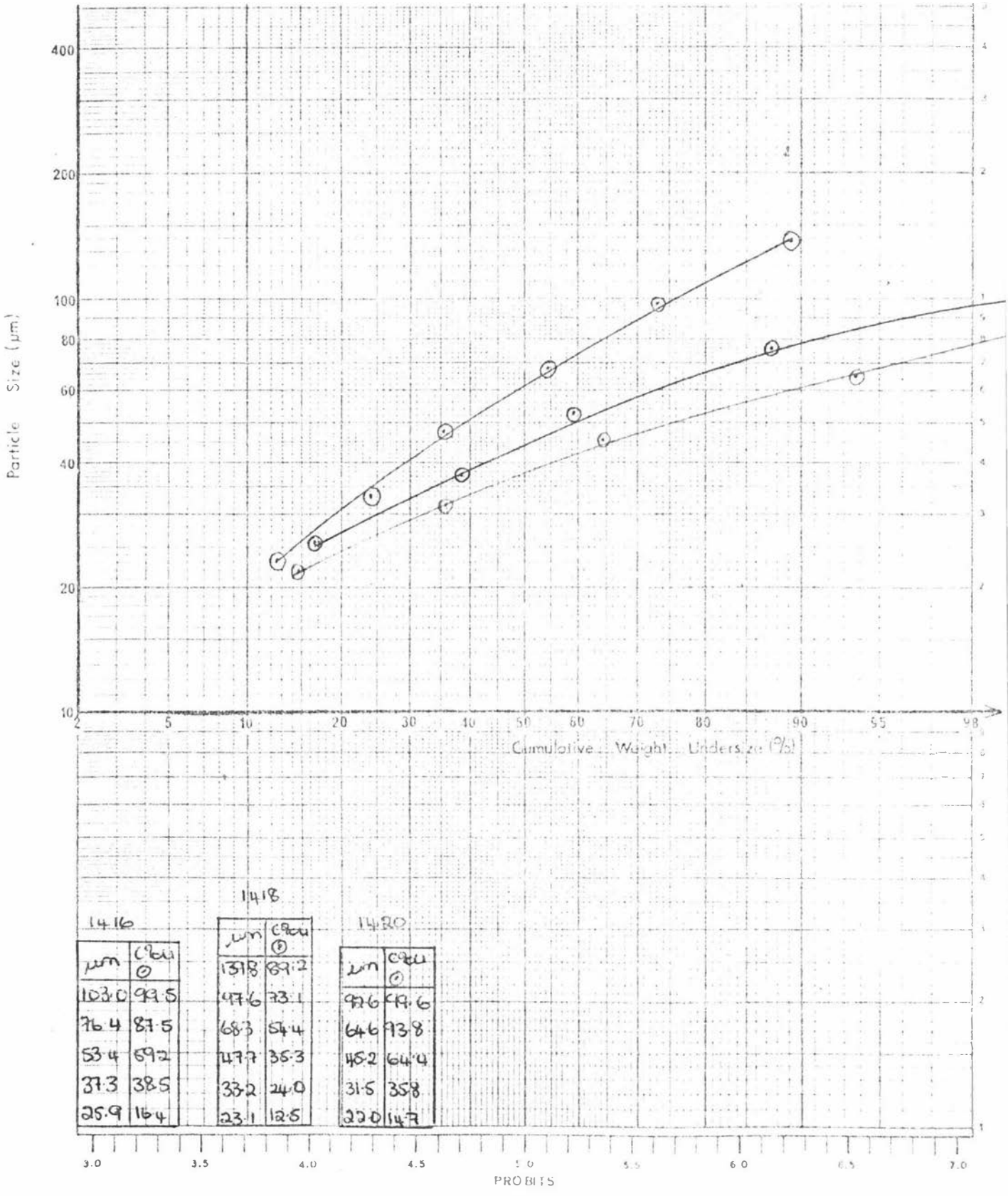
The calculation of the cumulative weight percentage of the powder under each size is then determined as follows.

$$C = 0.96\ w\ v\ /\ 600$$

Cumulative % undersize = weight extracted / C

where C = initial concentration
0.96 = correction factor for the powder moisture
w = sample weight (g)
v = pipette volume (ml)

The size is obviously very sensitive to small errors in the powder density determination, particularly when the powder density approaches that of the alcohol (0.7848 g/ml at 17 C). The results of the analysis are plotted on log-normal probability graph paper as shown overleaf. The surface-volume mean particle size and the standard deviation of the size distribution may then be calculated as follows. First, a least squares best fit straight line is drawn through the points. The 50 % (weight) size is denoted M and the standard deviation of the



1416

μm	C% ⊙
103·0	99·5
76·4	87·5
53·4	69·2
37·3	38·5
25·9	16·4

1418

μm	C% ⊙
131·8	89·2
97·6	73·1
68·3	54·4
47·7	36·3
33·2	24·0
23·1	12·5

1420

μm	C% ⊙
92·6	79·6
64·6	93·8
45·2	64·4
31·5	35·8
22·0	14·7

FIGURE A.1 A typical size distribution graph

distribution is:

$$\sigma_g = 84\ \%\ \text{size} / 50\ \%\ \text{size} = 50\ \%\ \text{size} / 16\ \%\ \text{size}$$

and the surface-volume mean diameter D_{sv} is:

$$D_{sv} = \ln M - 0.5 (\ln \sigma_g)$$

If the data are not well approximated by a staight line, the estimates of σ_g and D_{sv} will be in error, with σ_g being affected to the greater extent.

Rapid Solubility Index Method

For the optimisation trial a rapid version of the ADMI Solubility Index method was developed.

Step 1 - Mix 10 g of powder as for the standard method
Step 2 - Immediately transfer the liquid to an SI tube and centrifuge for 60 seconds at 5000 rpm
Step 3 - Read the tube without rinsing and spinning again
Step 4 - Multiply the reading by 2/3 and round to the nearest 0.05 ml

This method gave results which agreed well with the official method. The powders from the optimisation trial were subsequently analysed for SI and in 20 out of the 66 samples the values were identical. In a further 26 cases the rapid method gave values 0.05 ml higher. Agreement was best at low SI values.

APPENDIX VI - Data from Seasonal Experiment

Replicate 1 (7-Dec-77, 8-Dec-77, 13-Dec-77)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.l. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σ g	To (C)
1	224.0	48.9	266.3	18.32	35.8	35.8	59.9	1190.9	2.99	2.80	0.43	0.44	0.51	0.59	1.28	47	2.15	101.6
2	210.2	46.0	254.0	25.28	48.1	45.8	41.0	1168.4	2.73	0.40	0.49	0.50	0.57	0.70	1.22	28	1.62	96.3
3	209.7	46.0	282.8	24.66	34.5	34.9	46.3	1181.5	3.86	0.35	0.54	0.55	0.62	0.75	1.27	30	2.04	90.7
4	224.7	47.1	267.9	27.82	55.9	52.6	32.1	1174.5	2.05	2.00	0.38	0.39	0.43	0.55	1.08	41	2.08	103.0
5	210.0	47.4	266.8	24.69	49.8	47.5	36.7	1180.8	3.02	0.90	0.48	0.49	0.56	0.68	1.22	40	3.08	97.0
6	209.6	49.9	253.7	21.22	61.0	55.2	38.6	1183.2	2.91	1.40	0.46	0.47	0.54	0.65	1.15	38	2.23	99.9
7	209.8	48.8	282.9	25.02	48.4	46.8	47.8	1192.4	3.50	1.00	0.51	0.52	0.59	0.71	1.25	29	2.22	94.5
8	195.5	47.6	278.1	31.02	62.8	56.8	31.6	1177.0	3.52	0.05	0.57	0.58	0.67	0.80	1.31	23	1.63	87.2
9	224.7	47.3	266.4	21.30	38.4	38.1	50.3	1190.5	3.32	2.50	0.41	0.42	0.49	0.59	1.18	56	1.40	100.4
10	194.7	46.9	285.2	26.11	39.7	38.5	48.6	1188.3	4.56	0.05	0.59	0.60	0.68	0.81	1.33	33	1.82	84.1
11	195.2	47.7	282.9	21.85	36.2	36.1	57.1	1191.5	5.18	0.10	0.61	0.62	0.71	0.81	1.34	38	2.01	83.3
12	210.4	47.7	266.1	24.23	47.7	44.9	38.6	1184.2	3.18	0.40	0.50	0.51	0.58	0.69	1.16	80	1.73	94.4
13	223.7	48.2	252.3	23.44	57.5	52.3	32.5	1181.6	2.13	1.60	0.35	0.35	0.40	0.50	1.09	59	1.81	103.9
14	225.1	47.7	284.7	24.78	38.8	38.2	51.6	1190.1	3.04	1.80	0.44	0.45	0.51	0.62	1.40	42	2.12	98.7
15	210.5	45.5	269.7	30.51	47.1	45.8	33.0	1169.9	2.82	0.50	0.52	0.53	0.59	0.73	1.10	31	1.54	93.0
16	195.4	47.7	252.1	23.90	54.6	51.1	34.9	1179.0	3.40	0.10	0.56	0.58	0.65	0.77	1.26	35	1.81	90.2
17	209.7	45.9	270.2	19.72	27.1	30.6	62.4	1188.9	4.08	0.75	0.55	0.56	0.63	0.73	1.26	38	2.46	91.7
18	209.8	49.7	266.6	20.09	44.5	43.1	54.6	1193.8	3.55	1.60	0.50	0.52	0.59	0.68	1.22	37	1.88	95.8
19	209.3	49.3	268.0	24.09	58.2	51.9	42.7	1189.6	3.18	0.60	0.48	0.50	0.56	0.67	1.19	36	2.45	96.8
20	210.3	46.7	251.1	21.30	48.0	44.0	38.0	1183.9	3.08	1.20	0.46	0.47	0.54	0.64	1.16	47	1.98	98.3
21	225.2	46.0	266.2	24.12	40.5	38.6	42.3	1180.8	2.61	1.20	0.42	0.42	0.48	0.59	1.09	49	1.81	100.1
22	224.1	50.0	268.6	23.45	61.9	55.7	45.9	1185.4	2.64	2.60	0.37	0.38	0.43	0.52	1.14	67	2.04	103.4
23	194.8	49.2	267.7	23.27	61.7	55.4	45.8	1185.4	3.85	0.26	0.56	0.57	0.65	0.78	1.30	41	1.92	91.5
24	194.9	46.1	268.5	24.39	40.0	38.8	41.3	1181.7	4.47	0.05	0.58	0.59	0.68	0.79	1.31	42	1.74	85.5
25	210.0	47.6	252.0	23.26	60.2	54.7	34.2	1181.8	2.82	0.70	0.47	0.48	0.54	0.66	1.15	46	1.74	97.2
26	210.2	47.6	283.4	28.59	49.3	46.4	40.1	1187.5	3.35	0.40	0.51	0.52	0.59	0.73	1.23	36	1.88	92.1
27	209.7	46.9	267.9	24.55	47.3	44.8	39.9	1187.3	3.26	0.50	0.48	0.51	0.57	0.69	1.21	42	1.89	95.1
28	209.5	47.5	283.5	19.48	32.4	33.5	67.0	1198.4	4.42	0.70	0.53	0.56	0.64	0.74	1.23	48	2.09	91.5

Replicate 2 (14-Dec-77, 15-Dec-77, 9-Jan-78)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σ g	To (C)
29	195.3	49.5	268.8	21.04	59.9	54.0	45.4	1187.0	4.36	0.45	0.59	0.60	0.68	0.78	1.30	69	2.15	89.9
30	210.0	47.9	245.5	17.34	49.7	46.3	40.5	1188.3	3.05	1.60	0.48	0.50	0.57	0.66	1.19	76	2.38	98.2
31	209.8	47.4	267.5	23.53	52.9	49.5	36.1	1182.9	3.00	0.50	0.48	0.50	0.56	0.69	1.17	88	1.85	96.1
32	210.5	47.3	253.6	21.23	61.5	56.1	35.9	1178.1	2.73	1.90	0.43	0.44	0.50	0.62	1.12	55	1.76	99.4
33	210.0	47.5	289.6	29.46	54.4	53.3	36.7	1180.2	3.03	0.75	0.51	0.52	0.58	0.72	1.18	54	1.57	94.0
34	224.8	48.5	268.2	21.70	64.0	46.5	41.3	1184.2	2.39	3.80	0.35	0.35	0.41	0.49	1.12	76	2.34	105.8
35	194.7	45.3	267.4	25.98	50.5	49.2	29.5	1174.9	3.41	0.20	0.55	0.56	0.63	0.76	1.30	78	2.07	90.1
36	224.7	45.8	271.1	25.23	47.2	46.2	31.4	1178.5	2.22	2.00	0.37	0.38	0.43	0.54	1.09	59	1.74	102.8
37	209.8	47.5	283.3	19.56	37.6	38.1	54.1	1195.3	3.69	1.80	0.51	0.52	0.60	0.70	1.21	51	2.19	95.4
38	209.5	46.0	253.2	22.17	52.8	49.5	28.5	1166.5	2.49	0.60	0.45	0.46	0.52	0.63	1.11	44	1.62	99.1
39	209.5	48.4	284.4	23.78	44.6	55.5	55.5	1189.4	3.18	0.80	0.48	0.50	0.56	0.68	1.19	42	1.80	96.4
40	210.2	45.5	285.1	24.85	37.2	36.6	43.5	1182.7	3.56	0.20	0.51	0.52	0.59	0.73	1.26	35	1.79	91.0
41	225.6	47.0	267.6	19.01	40.8	38.2	48.0	1188.7	2.78	2.90	0.40	0.42	0.48	0.57	1.17	66	1.89	102.0
42	210.5	49.1	252.1	15.83	62.3	54.9	46.2	1186.4	2.83	3.40	0.45	0.46	0.53	0.62	1.19	65	1.93	102.6
43	209.1	47.1	268.8	23.82	58.1	53.7	34.1	1181.8	2.70	0.90	0.45	0.47	0.53	0.66	1.16	44	1.72	98.2
44	194.6	47.9	280.2	25.67	66.6	59.6	40.3	1175.2	3.66	0.05	0.57	0.58	0.65	0.79	1.26	33	1.65	89.1
45	226.0	47.1	267.6	24.09	65.6	59.9	35.4	1173.1	1.98	1.50	0.32	0.33	0.37	0.49	1.13	90	2.29	105.1
46	194.8	47.7	268.5	17.53	38.0	41.1	56.2	1197.8	4.13	0.50	0.56	0.57	0.66	0.77	1.27	45	2.15	92.0
47	209.9	45.2	269.5	21.33	28.6	30.3	46.9	1190.0	4.09	0.20	0.57	0.59	0.68	0.77	1.29	37	1.95	90.4
48	194.9	47.3	293.2	26.06	34.9	35.5	52.9	1194.2	5.28	0.05	0.62	0.64	0.73	0.82	1.32	38	1.99	81.2
49	224.5	46.9	284.3	24.71	35.4	35.7	50.4	1194.3	3.31	0.60	0.48	0.49	0.56	0.65	1.20	42	2.12	95.7
50	209.8	45.5	263.8	28.38	42.5	43.4	32.9	1181.7	2.79	0.05	0.53	0.54	0.61	0.75	1.26	47	1.81	92.8
51	210.4	47.8	268.0	24.05	40.8	41.1	42.9	1191.3	3.25	0.05	0.52	0.53	0.61	0.72	1.23	37	2.11	93.9
52	195.7	47.9	252.4	23.48	48.9	46.7	34.3	1186.6	3.58	0.15	0.57	0.58	0.66	0.78	1.27	35	1.71	90.0
53	225.5	47.1	253.7	24.69	48.4	47.2	34.0	1183.6	2.13	1.60	0.38	0.38	0.44	0.54	1.08	53	1.61	103.6
54	210.3	49.9	269.5	26.60	61.4	55.4	41.5	1192.9	2.79	0.30	0.48	0.50	0.57	0.68	1.19	40	1.94	97.5
55	209.6	49.3	277.6	21.26	36.2	35.6	62.3	1203.1	4.00	0.30	0.56	0.58	0.66	0.74	1.25	45	2.18	93.7

Replicate 3 (17-Jan-78, 18-Jan-78, 19-Jan-78)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
56	224.5	47.6	285.5	24.79	37.0	36.3	52.3	1192.8	3.55	1.20	0.47	0.48	0.55	0.65	1.19	55	1.76	94.4
57	210.5	49.0	271.0	19.56	40.2	39.3	58.1	1200.3	3.92	0.30	0.54	0.55	0.63	0.72	1.23	44	2.29	92.4
58	210.2	47.2	271.1	24.56	41.8	42.0	44.6	1189.1	3.53	0.40	0.51	0.52	0.61	0.71	1.21	42	1.74	91.7
59	209.3	45.4	266.9	21.62	29.4	31.2	51.0	1187.2	3.22	0.38	0.55	0.57	0.65	0.75	1.26	36	2.27	89.8
60	210.7	45.8	266.2	19.05	26.5	28.7	58.6	1191.5	4.28	1.20	0.57	0.59	0.67	0.79	1.27	36	2.38	89.6
61	210.2	46.0	269.3	29.89	44.8	42.9	33.2	1174.8	2.89	0.05	0.52	0.53	0.60	0.76	1.27	24	1.86	89.6
62	210.4	48.5	266.5	27.20	64.2	59.5	40.0	1186.9	2.61	0.40	0.49	0.49	0.56	0.69	1.26	36	1.77	95.3
63	225.6	47.2	253.7	24.29	49.1	47.9	38.0	1190.1	2.11	1.80	0.36	0.36	0.42	0.52	1.10	63	1.87	103.1
64	194.7	46.6	253.5	24.89	50.8	48.6	32.7	1187.9	3.32	0.05	0.55	0.57	0.65	0.78	1.28	32	1.83	88.7
65	195.1	47.0	281.4	25.46	37.0	37.6	49.8	1199.3	4.71	0.05	0.60	0.61	0.70	0.80	1.31	38	2.02	82.4
66	210.3	47.4	269.9	25.28	41.8	41.7	42.8	1194.2	3.42	0.35	0.51	0.53	0.60	0.72	1.25	42	1.76	91.2
67	210.4	45.8	252.4	23.53	42.8	42.8	33.9	1175.6	2.95	0.25	0.49	0.50	0.58	0.70	1.20	38	1.69	95.7
68	225.5	47.2	266.9	20.59	36.2	37.5	51.9	1190.8	2.93	2.00	0.42	0.43	0.50	0.60	1.17	65	1.94	101.7
69	209.6	45.3	282.8	25.15	29.3	32.1	49.0	1184.7	4.24	0.10	0.56	0.57	0.65	0.76	1.26	34	2.18	88.9
70	210.8	49.3	252.4	21.25	62.3	58.1	41.7	1186.4	2.90	1.20	0.44	0.45	0.51	0.61	1.16	48	1.99	101.0
71	224.9	47.5	263.1	27.48	51.6	51.6	35.1	1193.5	2.16	1.10	0.39	0.40	0.45	0.57	1.10	56	1.85	103.8
72	210.1	47.5	270.4	25.04	43.0	41.9	44.3	1193.5	3.41	0.30	0.50	0.51	0.59	0.71	1.24	38	2.01	94.3
73	195.3	48.1	277.9	20.78	32.5	34.7	64.1	1199.3	5.21	0.15	0.61	0.63	0.72	0.79	1.29	50	2.07	85.3
74	210.1	48.6	282.9	24.83	41.2	40.8	54.1	1196.8	3.92	0.30	0.53	0.55	0.63	0.73	1.25	37	1.91	92.2
75	195.5	47.9	267.6	29.49	60.8	55.6	33.7	1174.4	3.58	0.05	0.58	0.59	0.67	0.79	1.32	31	1.62	88.4
76	210.4	48.6	255.5	19.69	42.3	41.1	44.0	1191.9	3.38	1.40	0.50	0.51	0.59	0.68	1.18	47	1.99	96.0
77	210.1	48.0	285.2	20.67	30.2	32.7	62.9	1197.7	4.34	0.30	0.57	0.58	0.67	0.76	1.25	46	2.10	90.9
78	224.5	45.8	265.4	24.53	37.1	38.1	37.3	1181.4	2.49	1.00	0.43	0.44	0.49	0.61	1.17	49	1.72	100.1
79	209.6	47.9	269.4	24.79	44.3	43.0	40.6	1188.1	3.40	0.30	0.50	0.52	0.58	0.71	1.24	40	1.74	93.9
80	210.1	47.8	282.6	27.45	45.1	42.9	42.1	1191.3	3.42	0.20	0.53	0.54	0.61	0.74	1.23	36	1.60	91.6
81	195.7	48.9	267.2	25.05	54.9	51.8	43.3	1191.5	3.78	0.10	0.57	0.59	0.67	0.79	1.30	38	1.85	88.7
82	225.2	49.3	268.3	23.96	50.4	49.0	43.7	1192.5	2.48	1.70	0.39	0.39	0.46	0.55	1.15	63	1.84	103.5
83	194.9	45.8	269.0	24.86	32.9	35.0	43.5	1183.1	4.65	0.05	0.59	0.61	0.69	0.81	1.29	39	1.81	83.9
84	210.4	47.6	256.0	28.87	70.2	64.3	32.4	1176.1	2.34	0.20	0.47	0.48	0.55	0.67	1.14	39	1.58	98.9

Replicate 4 (21-Feb-78, 22-Feb-78, 23-Feb-78)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
85	209.2	49.2	252.0	20.16	54.6	51.5	42.7	1193.1	3.01	1.20	0.46	0.47	0.55	0.64	1.14	46	2.20	99.7
86	209.4	49.1	282.3	19.53	38.7	38.2	71.9	1204.1	4.28	0.30	0.55	0.56	0.65	0.73	-	25	2.97	92.5
87	225.4	46.5	265.6	24.44	46.1	45.3	38.9	1186.5	2.29	1.20	0.39	0.40	0.47	0.57	1.00	42	1.86	102.7
88	225.1	50.8	270.2	22.07	69.3	62.3	60.1	1195.0	2.25	2.30	0.35	0.35	0.41	0.49	1.08	64	2.17	106.8
89	195.1	50.0	270.3	22.55	70.5	63.0	59.8	1195.1	3.61	0.20	0.56	0.57	0.66	0.76	1.25	53	2.00	94.0
90	210.0	49.1	265.3	23.87	54.0	53.2	43.3	1196.0	2.88	0.30	0.50	0.51	0.59	0.70	1.20	50	1.66	97.4
91	210.2	49.5	283.1	27.07	49.3	49.9	49.3	1201.4	3.18	0.18	0.53	0.55	0.63	0.75	1.24	51	1.75	93.1
92	194.7	47.4	269.4	24.11	40.9	41.3	46.2	1198.5	4.19	0.10	0.60	0.61	0.70	0.80	1.30	47	1.84	85.9
93	210.2	49.1	250.1	23.02	80.7	69.9	56.5	1184.4	2.45	0.80	0.45	0.46	0.53	0.63	1.22	61	1.94	101.5
94	210.1	48.3	269.8	23.42	46.9	47.3	48.0	1215.6	3.17	0.60	0.50	0.51	0.60	0.68	1.19	66	1.84	95.9
95	210.4	48.5	282.5	29.12	52.6	50.0	45.4	1212.4	3.20	0.20	0.51	0.53	0.60	0.73	1.22	65	1.66	93.8
96	210.5	49.0	267.9	24.74	49.8	48.6	45.5	1214.7	2.69	0.60	0.49	0.50	0.58	0.66	1.13	59	1.99	95.7
97	195.6	48.9	250.6	24.24	69.2	62.8	38.1	1184.7	3.16	0.20	0.56	0.57	0.67	0.77	1.26	34	1.64	92.6
98	225.0	49.5	252.1	23.90	69.8	62.6	39.4	1186.5	2.07	0.80	0.33	0.34	0.39	0.48	1.03	91	1.91	105.4
99	225.5	49.4	286.8	23.49	39.2	40.9	67.7	1206.9	3.13	0.80	0.44	0.46	0.53	0.62	1.17	52	1.98	99.4
100	210.1	46.4	266.1	28.10	50.2	47.9	34.7	1190.2	2.89	0.10	0.50	0.52	0.59	0.72	1.22	33	1.53	94.4
101	210.0	49.8	267.6	20.28	47.9	46.0	59.6	1206.9	3.43	0.70	0.51	0.52	0.61	0.69	1.19	50	1.90	96.7
102	210.4	46.4	267.3	19.19	30.8	33.0	66.5	1202.9	4.17	0.20	0.57	0.58	0.66	0.73	1.23	49	2.07	92.7
103	209.1	49.9	267.5	25.94	73.0	65.7	57.3	1196.2	2.69	0.30	0.44	0.46	0.54	0.64	1.15	44	1.87	100.6
104	209.9	48.4	268.8	24.66	43.3	42.2	47.8	1216.0	3.35	0.20	0.50	0.51	0.60	0.69	1.21	41	1.77	94.4
105	194.9	47.4	281.4	24.57	35.5	37.3	60.3	1218.5	4.67	0.05	0.60	0.61	0.71	0.81	1.29	35	2.18	84.0
106	210.0	48.1	268.8	24.70	43.3	43.4	48.4	1213.7	3.38	0.18	0.49	0.51	0.58	0.69	1.18	36	1.94	93.9
107	210.8	47.7	281.2	23.52	36.9	36.3	58.0	1197.2	4.12	0.20	0.54	0.56	0.65	0.75	1.25	46	2.08	90.5
108	209.5	47.4	255.7	24.10	51.2	47.2	39.8	1187.8	3.04	0.15	0.48	0.49	0.56	0.68	1.19	40	1.73	95.7
109	195.1	48.8	266.4	20.80	43.3	41.6	56.0	1199.5	4.66	0.10	0.60	0.61	0.72	0.80	1.27	46	2.16	87.5
110	209.7	50.9	284.1	25.27	52.3	49.6	57.6	1203.3	3.54	0.25	0.51	0.52	0.61	0.70	1.22	47	1.94	93.6
111	224.9	48.9	266.5	27.29	61.1	56.9	39.3	1191.1	2.14	0.90	0.34	0.35	0.40	0.51	1.07	56	2.29	104.7
112	210.5	48.4	266.6	24.48	48.7	47.2	47.0	1197.3	3.14	0.25	0.50	0.51	0.60	0.69	1.19	45	1.66	96.6
113	194.9	49.7	265.1	28.58	70.2	64.7	37.3	1189.7	3.11	0.10	0.54	0.55	0.64	0.77	1.29	41	1.92	91.4
114	225.2	50.0	265.4	19.57	41.2	40.9	65.4	1207.2	2.88	0.80	0.42	0.43	0.51	0.59	1.17	64	2.22	103.0
115	225.5	49.9	258.3	22.52	73.4	66.2	60.1	1202.0	1.93	1.00	0.31	0.32	0.36	0.45	1.11	45	2.09	110.3

Replicate 6 (29-Aug-78, 30-Aug-78, 31-Aug-78)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
116	210.2	46.1	268.8	21.30	33.7	33.9	43.0	1193.2	3.96	0.50	0.55	0.57	0.65	0.75	1.22	26	2.38	92.7
117	210.4	49.3	304.6	24.98	35.4	36.2	54.8	1205.7	4.24	0.40	0.56	0.58	0.67	0.76	1.26	31	2.30	88.8
118	209.9	46.3	306.6	24.23	23.2	25.8	62.5	1202.4	5.15	0.15	0.59	0.60	0.69	0.79	1.29	36	2.35	84.1
119	225.4	47.8	285.1	19.78	26.8	28.1	68.3	1205.5	4.04	1.90	0.50	0.52	0.60	0.67	1.22	44	2.10	95.1
120	210.4	49.0	266.6	23.22	47.6	44.3	49.6	1194.0	3.26	1.20	0.50	0.51	0.59	0.70	1.21	38	1.82	95.3
121	210.4	48.1	288.5	22.83	31.9	34.0	60.6	1202.3	4.22	0.50	0.54	0.56	0.65	0.76	1.27	37	2.20	90.4
122	225.1	48.5	287.0	28.17	43.8	42.2	45.2	1195.3	2.72	1.60	0.44	0.46	0.52	0.64	1.16	38	1.95	98.3
123	194.5	47.2	283.9	20.08	27.5	29.2	65.0	1204.2	5.74	0.15	0.62	0.63	0.72	0.83	1.31	42	2.37	81.6
124	195.0	46.9	281.4	27.49	44.8	42.0	42.2	1193.8	4.36	0.10	0.58	0.60	0.68	0.81	1.31	28	1.86	83.6
125	210.2	47.2	284.3	23.57	34.7	35.7	52.7	1200.3	4.15	0.60	0.55	0.56	0.65	0.76	1.25	40	2.12	89.8
126	210.2	45.8	286.1	20.90	22.1	24.8	66.8	1195.3	4.87	0.45	0.60	0.62	0.70	0.79	1.30	40	2.41	87.2
127	195.2	46.4	300.5	22.62	24.0	25.6	66.9	1199.7	6.41	0.10	0.65	0.66	0.76	0.84	1.35	40	2.29	77.3
128	224.9	47.5	303.0	22.00	26.3	27.2	71.7	1203.7	4.40	1.30	0.56	0.57	0.65	0.73	1.25	47	2.28	89.9
129	210.1	48.3	287.1	19.56	30.8	31.2	71.3	1204.1	4.82	0.95	0.58	0.60	0.69	0.77	1.31	43	2.37	89.6
130	209.5	44.1	267.8	30.23	39.1	37.6	33.7	1184.4	3.13	0.15	0.50	0.51	0.57	0.75	1.28	25	1.76	89.3
131	209.8	47.1	283.1	24.26	34.7	35.6	53.7	1197.6	4.07	0.70	0.56	0.57	0.65	0.75	1.31	45	2.12	89.7
132	195.7	47.8	267.3	23.04	38.7	39.4	44.4	1190.8	4.38	0.14	0.59	0.60	0.68	0.79	1.32	35	1.98	85.6
133	225.1	47.0	265.2	23.96	42.6	40.6	41.8	1196.3	2.54	1.50	0.41	0.42	0.48	0.59	1.17	44	1.97	101.3
134	225.0	46.6	307.3	25.45	28.5	30.4	65.2	1201.2	3.83	0.75	0.51	0.53	0.61	0.71	1.25	45	2.13	93.3
135	209.8	48.4	280.0	27.17	47.6	44.8	42.7	1204.3	3.37	0.90	0.52	0.54	0.61	0.73	1.26	40	1.69	93.0
136	195.2	48.6	281.0	24.17	44.3	43.1	51.5	1202.0	4.45	0.12	0.59	0.60	0.69	0.80	1.29	29	2.17	85.6
137	209.3	48.0	269.5	20.47	34.6	35.1	58.0	1202.0	3.91	0.85	0.55	0.56	0.64	0.74	1.32	43	2.16	92.6
138	210.4	47.2	289.2	25.20	32.6	32.4	52.4	1201.6	4.18	0.28	0.56	0.57	0.66	0.77	1.29	28	1.97	88.2
139	210.6	46.5	264.3	27.64	48.1	45.6	34.9	1191.1	2.72	0.40	0.50	0.52	0.59	0.73	1.22	30	1.60	93.8
140	209.8	47.1	305.3	28.35	34.9	34.5	58.0	1202.2	4.39	0.10	0.59	0.60	0.69	0.79	1.30	37	1.87	86.0
141	210.0	46.6	302.6	22.11	20.9	24.4	92.1	1205.0	5.23	0.30	0.60	0.61	0.70	0.79	1.26	39	2.19	84.9
142	195.3	45.9	286.4	23.63	25.4	26.2	54.8	1198.8	5.75	0.05	0.64	0.65	0.74	0.82	1.32	38	1.85	78.8
143	226.0	44.0	284.1	25.04	26.0	26.7	50.2	1196.6	3.77	0.60	0.50	0.52	0.60	0.70	1.28	43	1.84	89.6
144	225.1	48.7	284.1	22.85	43.4	36.8	55.4	1209.5	3.20	2.01	0.45	0.47	0.54	0.63	1.33	55	1.83	97.4

Replicate 7 (3-Oct-78, 4-Oct-78, 5-Oct-78)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σ g	To (C)
145	209.8	46.4	265.4	21.54	32.1	33.6	49.0	1214.9	3.87	0.50	0.57	0.58	0.67	0.74	1.24	47	1.94	92.8
146	210.2	46.4	281.9	23.87	31.8	32.6	50.1	1205.3	4.08	0.35	0.56	0.57	0.65	0.76	1.25	52	1.83	89.6
147	194.7	48.2	283.5	24.94	40.9	39.2	47.1	1206.4	4.68	0.10	0.60	0.62	0.70	0.81	1.30	39	1.97	84.4
148	224.1	48.1	283.6	25.17	40.1	39.3	44.0	1206.9	3.22	1.20	0.45	0.48	0.54	0.64	1.22	46	1.78	94.9
149	225.8	44.8	284.5	24.83	24.0	27.5	51.5	1207.8	3.46	0.50	0.51	0.52	0.59	0.70	1.24	44	1.80	95.2
150	209.0	46.0	298.9	20.65	19.9	23.2	80.7	1212.8	5.70	0.20	0.61	0.62	0.72	0.80	1.29	59	1.97	85.4
151	209.3	46.6	261.3	24.64	46.0	41.7	39.7	1192.8	3.18	0.25	0.53	0.54	0.62	0.75	1.25	36	1.66	91.3
152	210.9	46.3	298.6	29.83	34.3	38.1	48.7	1204.1	3.79	0.15	0.55	0.56	0.64	0.76	1.27	39	1.74	89.0
153	195.3	44.6	283.9	22.85	20.9	26.8	59.8	1200.6	5.75	0.10	0.62	0.64	0.75	0.82	1.34	43	2.02	80.3
154	209.3	45.8	286.5	24.36	30.5	31.0	52.3	1204.5	4.50	0.30	0.57	0.58	0.66	0.77	1.31	43	1.84	88.2
155	224.5	46.8	288.8	20.14	27.1	28.3	56.8	1208.6	3.48	1.60	0.49	0.51	0.58	0.66	1.22	52	2.00	97.5
156	194.6	46.5	288.7	27.68	41.7	40.9	43.1	1199.6	4.12	0.05	0.57	0.58	0.66	0.80	1.29	35	1.63	84.1
157	210.5	46.6	286.8	24.40	34.2	34.3	51.9	1204.1	3.80	0.80	0.53	0.54	0.62	0.73	1.28	47	1.85	91.0
158	225.8	46.0	281.9	28.09	43.4	41.1	40.3	1196.3	2.34	1.40	0.44	0.46	0.52	0.64	1.17	37	1.67	98.4
159	210.0	47.8	267.6	23.56	48.7	46.0	42.3	1202.7	2.89	1.50	0.47	0.50	0.56	0.67	1.18	41	1.83	96.9
160	193.5	45.9	283.8	18.69	22.8	25.3	74.3	1208.9	6.34	0.10	0.61	0.63	0.73	0.82	1.30	51	1.92	81.3
161	210.1	43.9	263.0	24.02	36.5	35.3	41.8	1193.2	3.13	0.50	0.51	0.52	0.59	0.72	1.23	37	1.55	92.4
162	209.9	45.4	305.3	23.21	17.8	23.2	75.0	1203.7	5.26	0.20	0.59	0.60	0.69	0.79	1.30	39	1.90	84.0
163	210.3	46.0	280.6	24.38	34.0	33.1	50.8	1201.7	3.96	0.80	0.54	0.55	0.63	0.73	1.25	45	1.84	89.9
164	210.2	45.4	285.9	20.16	21.4	24.2	60.1	1202.3	4.89	0.60	0.58	0.59	0.69	0.78	1.30	43	2.14	87.9
165	194.7	47.2	305.5	24.70	31.2	31.2	53.1	1205.2	5.77	0.10	0.61	0.63	0.72	0.84	1.37	38	1.96	79.0
166	209.8	49.6	281.5	19.57	38.1	36.8	56.1	1210.0	4.26	1.30	0.54	0.56	0.65	0.73	1.26	48	2.07	92.4
167	210.5	49.2	288.2	28.07	51.0	49.7	38.1	1198.4	3.10	0.70	0.50	0.58	0.58	0.71	1.23	32	1.78	94.0
168	224.3	46.6	264.0	24.07	44.4	42.5	32.4	1192.8	2.33	1.60	0.38	0.39	0.44	0.56	1.12	46	1.82	104.8
169	209.4	45.4	284.7	29.02	37.8	38.9	35.3	1192.5	3.22	0.25	0.52	0.53	0.60	0.76	1.29	29	1.58	90.4
170	195.3	46.6	264.0	22.87	41.5	41.6	39.0	1196.0	4.03	0.10	0.57	0.58	0.66	0.78	1.29	33	1.85	87.4
171	225.6	46.5	304.2	24.50	27.1	29.8	58.4	1207.1	3.66	0.70	0.49	0.51	0.59	0.68	1.24	43	1.99	94.0
172	209.5	46.5	286.9	23.67	33.9	33.9	49.0	1204.4	4.07	0.60	0.55	0.56	0.64	0.75	1.28	39	1.93	90.4
173	195.0	47.5	266.2	23.48	44.4	40.8	37.6	1193.8	4.24	0.10	0.56	0.57	0.66	0.78	1.30	35	1.74	87.5

Replicate 8 (14-Nov-78, 15-Nov-78, 16-Nov-78)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
174	210.5	47.9	287.3	24.27	46.2	43.0	41.8	1210.6	3.82	0.50	0.51	0.52	0.60	0.69	1.28	43	2.05	91.2
175	216.6	48.0	284.0	27.56	59.6	53.6	32.9	1198.6	2.71	0.35	0.45	0.46	0.53	0.65	1.17	31	1.84	98.5
176	209.5	46.9	284.8	22.96	33.2	33.7	46.1	1210.4	4.47	0.30	0.55	0.57	0.65	0.74	1.26	50	2.30	89.2
177	209.8	46.4	269.9	24.68	46.5	45.1	35.3	1204.3	3.24	0.20	0.51	0.52	0.60	0.72	1.29	39	1.80	93.0
178	194.4	48.1	286.6	19.88	34.7	33.6	56.7	1220.5	6.08	0.10	0.63	0.64	0.75	0.81	1.27	55	2.45	82.4
179	224.3	47.8	286.6	20.07	33.9	33.0	57.1	1220.0	4.13	0.90	0.49	0.51	0.60	0.66	1.26	59	2.26	96.1
180	211.2	49.5	267.9	21.28	70.8	64.7	45.1	1212.6	2.97	1.00	0.44	0.45	0.53	0.61	1.21	57	2.07	100.2
181	210.2	49.7	305.3	24.27	43.0	42.6	53.7	1224.6	4.27	0.40	0.53	0.55	0.63	0.72	1.25	49	2.06	90.3
182	194.6	47.7	286.2	27.85	58.4	53.1	34.7	1211.5	4.04	0.05	0.57	0.58	0.67	0.79	1.31	34	1.64	84.6
183	225.4	48.2	281.6	27.91	61.4	56.3	32.8	1215.7	2.39	1.00	0.36	0.38	0.44	0.54	1.10	51	1.82	100.9
184	209.3	48.3	289.1	25.88	46.7	45.6	40.4	1220.7	3.57	0.30	0.50	0.51	0.59	0.69	1.24	43	1.90	92.8
185	195.2	50.5	287.8	23.68	54.9	49.5	45.6	1211.7	4.53	0.05	0.60	0.61	0.71	0.80	1.26	40	2.00	86.0
186	195.4	46.2	287.3	23.99	33.6	34.0	44.1	1207.5	5.18	0.05	0.61	0.63	0.73	0.81	1.30	43	1.95	81.2
187	224.1	46.7	286.0	23.79	37.0	35.9	43.8	1207.8	3.25	0.60	0.48	0.50	0.57	0.66	1.19	44	2.06	96.7
188	210.2	48.4	288.5	23.67	50.5	47.6	38.4	1208.6	3.49	0.25	0.51	0.53	0.60	0.71	1.23	41	1.87	94.8
189	225.3	48.9	285.1	23.52	60.0	54.9	37.6	1208.1	2.32	1.60	0.36	0.37	0.42	0.51	1.11	78	1.91	105.0
190	210.5	47.7	297.3	28.98	59.3	55.8	30.8	1201.5	2.88	0.18	0.51	0.53	0.59	0.73	1.21	34	1.57	95.0
191	210.1	48.5	266.8	22.55	70.1	64.5	30.6	1196.3	2.47	0.50	0.44	0.45	0.51	0.62	1.14	40	1.84	100.8
192	210.5	47.7	264.3	22.76	56.7	59.3	29.0	1200.0	2.51	0.55	0.44	0.45	0.51	0.62	1.13	44	1.82	100.1
193	210.2	48.2	266.9	19.98	50.0	48.1	36.4	1211.8	2.98	1.00	0.48	0.49	0.56	0.66	1.20	50	1.97	98.6
194	210.3	47.5	307.7	20.36	30.0	30.9	62.4	1223.0	5.03	0.35	0.60	0.61	0.70	0.78	1.30	47	2.55	88.9
195	210.7	48.0	284.0	23.80	50.1	47.6	36.1	1209.9	3.22	0.30	0.50	0.52	0.59	0.70	1.21	39	1.84	94.1
196	209.4	47.7	308.7	28.70	50.1	47.7	38.1	1209.2	3.37	0.05	0.55	0.56	0.63	0.76	1.31	36	1.71	91.5
197	224.7	47.5	307.6	24.54	34.9	35.2	53.3	1214.2	3.60	0.50	0.50	0.51	0.57	0.67	1.22	37	2.70	94.8
198	195.5	47.3	266.4	24.27	60.3	52.7	30.9	1196.9	3.43	0.05	0.56	0.57	0.64	0.76	1.23	37	1.73	88.5
199	210.1	49.3	281.4	23.49	65.9	60.0	39.1	1205.1	3.01	0.60	0.49	0.51	0.57	0.66	1.18	48	2.01	96.6
200	209.7	45.7	285.3	28.49	48.1	45.9	29.4	1199.6	2.98	0.05	0.51	0.53	0.59	0.73	1.24	36	1.54	90.7
201	209.7	45.8	288.7	19.43	23.6	26.4	58.5	1217.2	5.24	0.25	0.59	0.61	0.69	0.78	1.27	51	2.38	88.1
202	210.2	49.8	285.3	20.72	52.9	48.3	49.8	1220.4	3.39	0.80	0.52	0.53	0.61	0.70	1.25	49	1.91	94.9
203	209.8	47.8	285.8	24.47	48.2	45.8	38.0	1213.0	3.37	0.30	0.51	0.56	0.61	0.71	1.24	50	2.18	92.2
204	225.3	47.6	267.1	24.22	57.8	52.7	31.8	1208.0	2.03	1.20	0.34	0.35	0.39	0.49	1.09	118	2.07	104.0
205	194.9	48.2	300.8	22.99	33.6	34.6	54.0	1222.1	5.43	0.05	0.57	0.59	0.67	0.79	1.29	48	2.34	79.9
206	194.9	47.8	300.9	24.01	36.9	35.4	49.3	1221.6	5.39	0.05	0.62	0.64	0.73	0.83	1.31	48	1.95	79.6

Replicate 9 (8-Jan-79, 9-Jan-79, 10-Jan-79)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σ g	To (C)
207	210.3	49.8	284.9	27.93	56.7	51.5	41.8	1240.9	2.79	0.50	0.49	0.50	0.57	0.69	1.16	36	1.76	95.2
208	209.6	49.8	286.6	20.46	35.7	35.6	72.2	1259.5	3.99	0.70	0.54	0.55	0.63	0.72	1.22	50	2.26	92.4
209	225.5	48.4	268.1	24.03	44.2	42.9	44.0	1245.2	2.32	1.80	0.36	0.37	0.43	0.52	1.11	63	1.96	103.2
210	195.9	48.5	268.5	24.24	44.6	42.3	42.3	1244.1	3.89	0.10	0.55	0.57	0.66	0.78	1.26	38	1.93	88.2
211	195.3	48.1	306.4	24.52	30.2	31.3	70.8	1254.8	5.51	0.05	0.61	0.63	0.72	0.82	1.30	46	2.28	79.8
212	209.7	48.5	285.1	23.99	37.4	36.0	53.8	1250.5	3.98	0.30	0.53	0.55	0.64	0.74	1.27	48	2.11	90.4
213	209.8	46.4	287.5	28.14	34.1	35.0	47.0	1243.0	3.63	0.05	0.52	0.53	0.61	0.74	1.26	33	1.81	88.6
214	209.7	46.3	285.3	19.98	17.9	22.5	83.8	1252.4	5.03	0.20	0.59	0.61	0.69	0.78	1.26	44	2.60	87.1
215	225.6	47.6	302.0	23.94	29.0	29.6	71.3	1254.4	3.87	0.70	0.51	0.52	0.59	0.67	1.21	43	2.58	94.1
216	209.6	47.5	285.3	24.21	36.3	35.4	55.9	1251.2	3.97	0.25	0.54	0.56	0.63	0.74	1.24	35	2.20	90.8
217	209.8	50.3	285.0	20.35	36.0	35.4	73.8	1259.4	4.31	0.50	0.55	0.56	0.65	0.72	1.24	53	2.59	92.1
218	210.0	48.0	286.1	24.20	35.6	35.3	56.1	1251.4	3.90	0.30	0.54	0.55	0.63	0.74	1.24	54	1.65	91.0
219	209.8	48.4	305.1	20.37	21.8	24.7	100.4	1262.5	5.43	0.20	0.59	0.61	0.71	0.77	1.25	57	2.52	85.0
220	210.5	48.6	283.0	23.88	38.0	37.2	58.3	1252.0	3.81	0.30	0.57	0.58	0.66	0.76	1.28	49	2.00	90.0
221	225.0	46.5	284.3	24.41	29.6	30.5	55.6	1247.7	3.24	0.50	0.48	0.50	0.57	0.68	1.23	39	2.39	95.2
222	224.7	49.7	283.9	24.55	43.5	42.3	54.5	1254.0	2.70	1.00	0.43	0.44	0.51	0.61	1.19	51	2.64	99.7
223	195.3	49.6	285.3	24.23	44.3	43.0	55.3	1254.9	4.53	0.05	0.61	0.62	0.70	0.81	1.31	40	2.31	86.2
224	195.2	47.1	285.0	23.83	29.4	31.9	59.2	1249.7	5.41	0.05	0.62	0.63	0.72	0.83	1.32	43	2.16	81.1
225	209.6	48.8	268.4	20.17	36.7	36.6	60.1	1254.0	3.87	0.40	0.54	0.55	0.63	0.72	1.23	51	2.52	93.5
226	210.0	48.0	301.3	28.26	37.1	37.5	55.1	1250.9	3.74	0.05	0.55	0.56	0.64	0.76	1.26	36	2.60	89.4
227	210.7	48.5	265.9	28.36	59.3	55.1	34.4	1236.5	2.54	0.15	0.48	0.49	0.56	0.70	1.20	33	1.58	96.2
228	210.1	48.7	286.1	24.13	36.8	38.3	59.2	1253.7	3.90	0.25	0.55	0.56	0.64	0.74	1.25	51	2.26	91.5
229	210.0	48.1	284.8	23.73	35.3	38.5	59.2	1254.6	3.85	0.15	0.56	0.57	0.65	0.75	1.25	40	2.15	92.1
230	210.2	46.4	266.5	24.16	41.0	39.7	38.2	1235.2	2.96	0.10	0.51	0.53	0.61	0.71	1.20	38	1.76	92.7
231	195.3	48.1	285.0	28.25	50.2	47.5	39.6	1240.3	3.74	0.05	0.56	0.57	0.66	0.79	1.29	35	1.65	84.9
232	195.1	48.0	285.1	19.52	27.2	31.1	75.2	1254.9	5.23	0.05	0.62	0.64	0.73	0.80	1.27	46	2.47	82.8
233	223.9	48.4	286.6	20.01	30.8	31.2	68.0	1254.3	3.47	0.80	0.48	0.49	0.57	0.65	1.20	56	2.19	95.7
234	209.3	48.6	286.6	23.39	38.1	36.8	52.4	1250.9	3.70	0.25	0.53	0.54	0.63	0.75	1.26	69	2.04	91.2
235	210.8	46.6	308.1	24.16	21.6	25.7	73.7	1249.5	4.95	0.20	0.59	0.60	0.68	0.79	1.27	28	2.30	84.8
236	209.8	49.6	302.1	24.55	39.2	37.1	61.0	1252.6	4.06	0.25	0.55	0.57	0.65	0.74	1.24	55	2.22	89.3
237	209.5	50.2	265.8	23.60	58.3	52.9	40.7	1242.0	2.59	1.20	0.44	0.46	0.52	0.63	1.16	44	1.86	98.6
238	225.6	48.9	284.4	28.19	49.4	46.4	40.5	1241.4	2.23	1.30	0.39	0.40	0.45	0.56	1.10	49	1.71	101.2
239	210.6	48.8	285.7	23.87	37.6	37.7	55.2	1250.4	3.71	0.50	0.52	0.54	0.61	0.72	1.23	43	2.23	92.8
240	209.8	46.8	303.5	23.97	21.7	24.9	71.6	1249.9	4.94	0.15	0.60	0.61	0.69	0.78	1.27	48	2.14	85.0
241	210.1	49.0	284.4	23.87	38.4	37.5	54.0	1250.4	3.85	0.60	0.53	0.55	0.63	0.72	1.25	49	2.09	91.8

Replicate 10 (12-Feb-79, 13-Feb-79, 14-Feb-79)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
242	224.9	48.8	289.7	21.12	43.2	41.2	44.6	1259.5	2.80	1.20	0.41	0.43	0.50	0.57	1.17	63	2.14	100.8
243	209.6	49.6	303.7	24.37	46.7	45.5	47.9	1265.1	3.78	0.20	0.52	0.53	0.62	0.72	1.23	44	2.04	90.4
244	210.1	48.0	285.0	23.63	46.6	44.7	38.6	1256.3	3.27	0.20	0.50	0.51	0.59	0.69	1.25	39	1.93	92.4
245	224.7	48.0	285.3	28.47	67.8	61.9	27.9	1242.1	1.86	0.55	0.34	0.35	0.39	0.51	1.05	54	1.92	102.6
246	209.9	50.6	272.2	22.44	71.0	65.1	37.5	1255.7	2.44	0.40	0.43	0.44	0.51	0.60	1.19	47	1.90	99.9
247	195.2	48.7	284.0	20.46	38.1	39.0	51.0	1273.6	5.02	0.10	0.62	0.63	0.72	0.80	1.29	38	2.33	84.4
248	209.6	46.8	265.8	23.66	48.9	47.0	31.1	1259.3	2.65	0.10	0.47	0.48	0.55	0.67	1.19	38	1.68	94.2
249	209.8	46.6	303.8	24.08	31.7	31.9	51.0	1272.8	4.65	0.10	0.57	0.59	0.68	0.76	1.27	44	2.16	86.0
250	195.7	48.7	284.3	27.94	58.4	62.7	31.1	1251.3	3.59	0.05	0.54	0.56	0.64	0.77	1.28	33	1.53	86.7
251	210.1	47.8	284.2	19.59	37.1	35.8	48.4	1257.6	4.30	0.50	0.55	0.56	0.65	0.72	1.24	56	2.11	92.2
252	194.2	48.4	300.1	24.19	47.7	44.7	44.3	1257.8	4.81	0.05	0.61	0.63	0.72	0.80	1.29	44	1.93	83.1
253	209.7	51.1	283.8	19.94	51.9	47.5	49.2	1259.3	3.69	0.80	0.51	0.53	0.61	0.67	1.23	54	2.32	95.3
254	210.3	51.2	288.7	24.16	67.5	64.0	40.0	1245.8	2.86	0.30	0.45	0.46	0.53	0.63	1.19	48	1.95	97.8
255	209.7	48.7	286.1	24.12	51.9	49.6	38.4	1256.9	3.08	0.15	0.48	0.50	0.57	0.67	1.21	45	1.73	94.5
256	224.7	48.8	263.2	23.37	69.4	63.3	29.8	1258.3	1.83	1.00	0.29	0.30	0.34	0.43	1.04	-	-	107.7
257	210.3	46.4	284.1	28.17	52.3	51.9	29.8	1254.8	2.86	0.10	0.47	0.48	0.55	0.68	1.20	35	1.54	93.7
258	195.4	49.4	264.4	23.20	67.9	63.2	31.1	1258.4	3.14	0.05	0.51	0.53	0.61	0.72	1.23	39	1.84	92.1
259	225.7	49.5	304.4	23.70	42.0	41.5	51.4	1262.4	3.12	0.60	0.44	0.45	0.52	0.59	1.20	50	2.01	97.8
260	209.9	48.7	285.8	23.82	47.9	46.4	41.4	1262.8	3.41	0.20	0.50	0.52	0.58	0.67	1.22	44	1.89	93.7
261	209.4	51.6	283.6	25.62	77.4	68.4	54.6	1256.3	2.73	0.30	0.44	0.46	0.52	0.61	1.17	50	1.74	99.0
262	210.0	48.8	268.0	20.01	52.1	48.3	41.4	1260.9	3.35	0.70	0.48	0.50	0.57	0.65	1.21	54	1.82	97.0
263	209.8	49.4	284.7	24.19	57.0	51.6	38.4	1257.2	3.22	0.25	0.49	0.50	0.58	0.68	1.23	42	1.81	94.5
264	195.6	50.4	283.9	23.13	75.3	64.7	48.0	1262.1	4.05	0.10	0.58	0.59	0.68	0.76	1.27	38	1.81	90.1
265	224.7	49.8	283.2	24.14	73.8	66.6	44.5	1265.7	2.49	0.80	0.34	0.35	0.41	0.49	1.10	77	2.02	103.5
266	195.0	50.4	283.2	24.15	73.0	66.8	43.5	1269.4	3.41	0.10	0.55	0.56	0.64	0.75	1.27	39	1.88	93.6
267	209.7	48.5	302.3	19.75	33.9	35.4	69.3	1290.0	4.91	0.25	0.58	0.59	0.68	0.76	1.24	47	2.54	90.0
268	210.7	48.8	261.3	23.33	74.1	68.0	32.8	1268.4	2.52	0.30	0.40	0.41	0.47	0.58	1.01	48	1.78	100.4
269	195.2	46.4	283.0	23.76	41.8	42.8	40.4	1285.3	4.47	0.10	0.59	0.60	0.68	0.78	1.28	38	2.16	84.5
270	225.4	47.0	284.3	23.85	45.0	43.1	41.1	1286.2	2.91	0.50	0.42	0.43	0.49	0.57	1.22	52	2.04	97.5
271	209.7	48.3	303.0	27.74	51.0	49.5	42.3	1292.9	3.63	0.10	0.53	0.54	0.61	0.72	1.24	47	1.75	90.7
272	210.2	48.0	282.6	24.31	51.8	49.2	39.4	1293.6	3.32	0.25	0.50	0.51	0.58	0.68	1.20	58	1.88	94.0

Replicate 11 (19-Mar-79, 20-Mar-79, 21-Mar-79)

Run No.	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
273	211.3	48.8	286.8	25.62	67.6	60.8	48.5	1210.2	3.21	0.15	0.48	0.49	0.56	0.68	1.21	37	1.79	93.8
274	210.5	49.6	286.1	20.00	46.8	45.9	69.6	1222.8	4.52	0.35	0.52	0.54	0.62	0.73	1.22	42	2.53	91.2
275	209.9	46.0	288.3	19.95	29.9	30.7	69.4	1221.3	5.60	0.15	0.60	0.61	0.70	0.79	1.23	33	2.63	85.3
276	194.8	47.8	267.4	23.94	63.9	54.2	38.5	1216.1	4.18	0.05	0.56	0.57	0.65	0.79	1.26	24	1.88	85.2
277	209.7	45.9	285.1	27.59	49.1	46.6	40.3	1212.1	3.63	0.05	0.50	0.52	0.58	0.73	1.23	30	1.83	88.0
278	224.6	46.7	301.7	23.89	39.1	38.5	63.6	1217.5	4.06	0.30	0.50	0.51	0.58	0.68	1.22	29	2.13	92.8
279	224.4	47.2	266.7	23.98	59.7	55.6	41.5	1215.0	2.43	0.80	0.35	0.36	0.41	0.52	1.22	56	1.83	101.8
280	210.0	46.7	284.5	24.20	46.5	44.3	50.8	1217.0	4.18	0.10	0.52	0.53	0.62	0.72	1.23	24	2.13	89.4
281	195.8	46.6	302.3	24.24	38.7	37.9	61.0	1220.1	5.76	0.05	0.62	0.64	0.72	0.84	1.27	38	2.31	82.8
282	209.8	47.8	268.1	19.65	56.0	52.0	49.7	1186.2	3.53	0.70	0.49	0.50	0.57	0.68	1.16	47	2.21	96.1
283	225.3	46.1	283.2	24.19	50.4	48.6	42.0	1176.9	2.58	0.40	0.41	0.42	0.48	0.60	1.14	57	1.75	98.2
284	195.2	49.3	288.8	23.07	71.1	64.1	55.3	1181.7	4.10	0.10	0.58	0.59	0.67	0.80	1.27	44	2.15	88.0
285	225.0	49.8	289.5	22.67	70.8	64.3	61.3	1183.4	2.94	1.20	0.37	0.38	0.44	0.52	1.10	69	2.08	100.0
286	195.2	45.8	282.0	23.34	46.7	45.5	45.3	1183.0	4.69	0.05	0.57	0.58	0.67	0.81	1.28	39	2.04	83.1
287	209.5	47.8	267.4	22.38	66.1	63.3	41.1	1196.3	2.82	0.50	0.47	0.48	0.54	0.66	1.17	41	2.00	97.8
288	209.9	47.9	302.9	19.73	37.2	38.1	79.2	1210.2	4.97	0.25	0.57	0.58	0.67	0.77	1.25	47	2.46	87.6
289	209.7	47.8	303.3	28.17	61.2	56.5	45.3	1210.8	3.45	0.05	0.51	0.52	0.59	0.75	1.24	31	1.88	89.6
290	210.0	48.2	282.5	23.68	62.0	57.3	47.7	1212.0	3.16	0.10	0.49	0.50	0.57	0.69	1.19	40	1.94	94.4
291	209.1	48.2	285.0	23.97	59.9	56.9	48.3	1209.6	3.19	0.20	0.49	0.50	0.57	0.69	1.20	41	1.94	94.2
292	209.8	45.1	266.2	24.12	51.6	48.3	31.9	1175.9	2.94	1.00	0.48	0.49	0.56	0.70	1.26	30	1.95	95.0
293	209.7	45.6	303.5	24.21	33.1	33.8	51.3	1186.7	4.83	0.05	0.57	0.58	0.66	0.77	1.23	35	2.17	85.9
294	224.5	46.7	283.4	20.43	39.7	38.8	52.1	1191.6	3.46	0.80	0.45	0.47	0.53	0.62	1.22	43	2.93	98.5
295	225.4	47.7	286.7	28.31	62.2	58.2	33.3	1177.6	2.34	0.15	0.38	0.39	0.43	0.55	1.05	44	1.90	101.8
296	209.8	47.2	287.1	23.30	44.8	44.4	48.1	1190.7	3.63	0.05	0.53	0.54	0.61	0.72	1.21	45	1.99	91.9
297	210.1	48.8	265.7	22.19	70.9	63.2	43.4	1183.0	2.81	0.15	0.43	0.45	0.51	0.62	1.13	51	1.92	98.9
298	210.7	48.8	301.2	23.85	46.6	44.7	57.0	1196.4	4.15	0.10	0.53	0.54	0.62	0.73	1.21	41	2.51	90.8
299	194.8	47.1	283.3	19.58	37.2	36.3	57.8	1196.9	5.80	0.05	0.61	0.63	0.72	0.83	1.27	39	3.04	82.6
300	194.6	46.9	287.1	28.55	59.0	56.2	32.6	1179.9	3.70	0.05	0.57	0.58	0.65	0.80	1.33	29	1.85	84.6
301	209.6	46.4	286.7	23.99	44.2	44.5	46.1	1188.7	3.67	0.05	0.53	0.54	0.61	0.73	1.26	38	1.78	90.0

APPENDIX VI - Experimental Data

Daily mean values of variables uncorrelated with the independent variables

Date	Rep	Air Flow (kg/min)	Inlet Pres. (kPa)	Ambient Temp (C)	Conveying Air Temp (C)
7-Dec-77	1	96.9	104.20	37.9	3.8
8-Dec-77	1	97.1	103.83	38.1	3.5
13-Dec-77	1	96.9	103.70	36.7	2.6
14-Dec-77	2	97.1	103.66	37.1	3.3
15-Dec-77	2	97.0	103.45	36.8	3.5
9-Jan-78	2	97.0	104.40	41.2	3.8
17-Jan-78	3	96.9	105.77	38.9	4.2
18-Jan-78	3	97.0	105.67	41.4	4.5
19-Jan-78	3	97.1	105.08	38.8	4.4
21-Feb-78	4	97.0	105.05	42.0	4.2
22-Feb-78	4	97.0	105.04	40.4	4.7
23-Feb-78	4	97.1	104.88	40.8	5.4
29-Aug-78	6	97.0	104.36	37.3	3.7
30-Aug-78	6	97.0	104.95	36.1	3.9
31-Aug-78	6	97.0	104.75	34.5	4.1
3-Oct-78	7	97.1	105.17	38.9	3.2
4-Oct-78	7	97.0	104.18	37.8	2.0
5-Oct-78	7	96.9	103.12	36.3	3.7
14-Nov-78	8	97.0	103.62	34.2	2.0
15-Nov-78	8	97.0	103.44	35.2	2.8
16-Nov-78	8	97.0	104.72	34.7	1.0
8-Jan-79	9	97.0	104.10	39.3	3.7
9-Jan-79	9	97.0	104.76	44.4	3.0
10-Jan-79	9	97.0	104.84	41.4	2.2
12-Feb-79	10	97.0	104.50	38.9	3.1
13-Feb-79	10	97.0	104.22	40.6	2.2
14-Feb-79	10	97.0	103.71	40.2	3.1
19-Mar-79	11	97.0	104.20	40.9	2.7
20-Mar-79	11	97.0	103.96	42.9	3.7
21-Mar-79	11	97.0	103.71	43.1	3.5

APPENDIX VI - Milk Composition for Seasonal Experiment

Date	Rep	Protein (%)	Ca (mM/kg)	Mg (mM/kg)	Na (mM/kg)	K (mM/kg)	PO4 (mM/kg)	Fat (%)
7-Dec-77	1	39.3	361.9	46.0	206.2	406.1	234.2	0.56
8-Dec-77	1	39.5	361.4	47.0	207.3	405.9	234.0	0.61
13-Dec-77	1	39.1	361.2	46.0	206.1	406.0	234.3	0.62
14-Dec-77	2	39.1	360.4	49.0	207.3	405.2	233.5	0.63
16-Dec-77	2	39.3	361.5	51.0	207.1	405.2	233.8	0.58
9-Jan-78	2	38.8	361.4	53.0	212.3	408.2	233.3	0.46
17-Jan-78	3	39.0	360.9	52.0	208.7	408.0	233.4	0.64
18-Jan-78	3	39.3	360.9	51.0	207.3	407.7	233.3	0.59
19-Jan-78	3	39.1	360.1	51.0	206.6	407.8	233.4	0.54
21-Feb-78	4	41.0	362.1	51.0	209.3	408.8	231.4	0.69
22-Feb-78	4	41.1	361.6	55.0	209.4	406.7	231.7	0.81
23-Feb-78	4	40.9	361.3	59.0	207.6	406.0	231.3	0.82
29-Aug-78	6	39.9	362.0	47.0	209.3	404.8	232.7	0.57
30-Aug-78	6	38.9	362.1	48.0	209.0	407.6	233.3	0.54
31-Aug-78	6	39.4	362.5	47.0	209.7	407.0	233.7	0.63
3-Oct-78	7	38.9	362.3	50.0	204.0	408.1	233.4	0.62
4-Oct-78	7	39.0	364.1	48.0	204.8	408.7	234.0	0.66
5-Oct-78	7	39.1	362.1	50.0	206.2	409.2	233.8	0.63
14-Nov-78	8	39.0	363.7	46.0	203.9	405.9	233.8	0.49
15-Nov-78	8	38.7	363.1	49.0	204.9	405.7	233.6	0.63
16-Nov-78	8	38.6	364.4	49.0	204.7	407.1	233.4	0.62
8-Jan-79	9	38.9	361.6	51.0	205.0	407.8	233.0	0.77
9-Jan-79	9	39.9	362.8	51.0	205.9	407.9	233.5	0.80
10-Jan-79	9	39.6	361.3	51.0	205.1	406.7	232.9	0.69
12-Feb-79	10	39.4	361.8	54.0	204.6	399.5	232.7	0.62
13-Feb-79	10	40.0	361.0	53.0	204.9	399.3	231.6	0.83
14-Feb-79	10	40.3	360.7	43.0	206.2	397.8	231.5	0.79
19-Mar-79	11	42.5	366.0	49.0	208.9	405.1	232.3	0.92
20-Mar-79	11	42.6	362.5	49.0	208.9	404.0	231.8	0.93
21-Mar-79	11	42.0	362.1	53.0	208.6	407.3	232.2	0.78

APPENDIX VI - Data from Experiment to Determine the Effect of Preheat Treatment on Concentrate Viscosity

Runs 1 - 10 5 March 1980, Runs 11 - 22 6 March 1980

Run No.	t_p (s)	T_p (C)	TS (%)	T_c (C)	μ_1 (cp)	μ_2 (cp)	T_{hp} (C)	F (l/h)	P (MPa)	WPNI (mg)
1	10	80	48.5	45.1	49.3	98	39.7	289.5	18.72	7.9
2	10	113	48.0	45.1	49.5	101	39.6	288.6	16.33	4.0
3	10	80	49.4	59.8	55.1	-	52.5	286.4	19.45	8.1
4	10	113	49.7	59.7	76.0	-	52.6	288.6	17.13	3.9
5	120	113	48.8	59.7	73.3	237	52.1	288.1	13.07	0.7
6	120	80	49.6	44.8	61.5	139	39.4	289.5	14.53	5.8
7	120	80	47.0	59.7	31.2	50	50.7	287.7	21.57	5.8
8	120	113	47.2	45.0	67.3	139	40.4	289.5	13.67	1.4
9	120	113	47.6	59.1	53.1	147	48.8	287.7	15.05	0.8
10	120	113	49.0	44.9	101.8	249	40.4	289.8	10.33	0.7
11	120	80	46.8	44.9	37.4	97	40.0	289.2	19.08	5.8
12	120	80	47.3	59.6	28.7	95	50.4	288.1	21.98	5.4
13	120	80	49.2	59.8	35.2	77	51.5	287.9	19.53	5.6
14	120	80	48.7	45.6	45.5	126	42.9	289.6	17.47	5.7
15	10	80	46.4	60.4	32.1	36	52.4	287.8	23.30	7.2
16	10	113	48.7	45.0	59.2	98	39.8	289.9	13.96	3.4
17	10	113	48.6	60.3	49.6	116	51.4	287.8	16.26	3.3
18	10	80	49.4	60.4	38.6	77	51.8	287.1	19.84	7.5
19	10	80	49.0	45.1	52.5	113	40.3	290.3	17.24	7.8
20	10	113	47.7	60.3	35.3	89	51.6	288.0	19.79	4.4
21	10	113	47.0	44.4	45.3	104	42.1	289.6	17.69	6.4
22	10	80	47.8	45.1	47.6	96	39.9	289.3	16.92	4.7

APPENDIX VI - Data from Experiment to Determine the Effects of Air Inlet Throat Diameter and Nozzle Position
(29 March 1979)

Run No.	D	Np	T in (C)	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
1	-1	+1	210.1	50.5	265.6	19.94	39.9	39.1	59.8	1190.9	4.10	0.30	0.60	0.61	0.70	0.79	1.30	45	2.36	94.4
2	-1	-1	210.8	49.8	265.9	20.15	39.9	38.6	62.0	1190.3	3.75	0.30	0.59	0.61	0.70	0.79	1.27	60	2.18	93.8
3	-1	0	210.2	50.5	265.6	19.63	39.4	38.0	63.4	1191.2	3.81	0.30	0.59	0.60	0.70	0.79	1.26	46	2.24	93.4
4	+1	-1	209.3	48.2	264.4	20.35	43.1	40.3	55.0	1189.9	3.75	0.35	0.55	0.56	0.64	0.73	1.31	47	2.06	94.1
5	+1	0	210.2	49.2	263.7	19.73	39.2	38.6	57.7	1190.2	3.75	0.30	0.55	0.56	0.65	0.74	1.26	52	2.05	93.6
6	+1	+1	209.5	49.9	264.2	20.28	41.6	40.2	56.1	1188.6	3.59	0.25	0.54	0.55	0.63	0.73	1.29	47	2.02	94.0
7	+1	+1	211.4	49.3	264.4	19.17	38.4	38.7	62.3	1191.1	4.05	0.50	0.53	0.54	0.63	0.72	1.26	48	2.18	94.0
8	+1	0	209.8	48.6	265.9	20.13	40.9	40.1	57.5	1188.9	3.96	0.40	0.54	0.55	0.64	0.73	1.22	50	2.10	93.7
9	+1	-1	209.2	50.0	265.9	19.68	39.7	39.8	60.4	1190.3	4.03	0.40	0.55	0.57	0.65	0.74	1.25	45	2.23	93.3
10	-1	-1	209.6	47.7	267.2	20.34	40.7	39.8	64.4	1191.2	4.13	0.30	0.61	0.62	0.72	0.80	1.27	45	2.29	92.1
11	-1	0	209.7	47.4	265.6	19.68	38.0	38.2	64.2	1192.7	4.41	0.20	0.60	0.62	0.72	0.80	1.30	52	2.17	92.3
12	-1	+1	209.9	47.2	264.8	20.17	38.7	39.0	61.4	1190.5	4.47	0.30	0.60	0.61	0.70	0.80	1.29	49	2.25	92.7

Data from Experiment to Determine the Effects of Nozzle Orifice Size, Concentrate Viscosity and Air Inlet Temperature
(11 October 1978)

Run No.	T in (C)	No	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
1	195.6	50	47.8	300.3	22.15	20.8	23.5	77.5	1215.6	7.55	0.35	0.65	0.67	0.79	0.83	1.31	64	2.34	82.1
2	225.8	50	48.1	311.2	22.16	47.3	44.7	31.0	1199.9	4.12	1.40	0.50	0.52	0.60	0.66	1.28	60	2.14	97.1
3	223.9	52	48.0	295.1	21.94	21.1	23.5	78.6	1211.5	4.47	1.40	0.52	0.54	0.62	0.68	1.28	62	2.28	97.0
4	194.7	52	47.9	289.5	22.35	47.2	44.3	37.5	1199.9	5.46	0.30	0.63	0.64	0.72	0.80	1.29	50	2.13	85.1
5	195.3	52	47.7	293.1	22.27	20.4	24.7	84.0	1212.5	6.98	0.35	0.64	0.66	0.76	0.83	1.30	52	2.57	81.9
6	195.3	50	47.5	313.1	22.15	44.9	42.8	40.3	1201.2	6.34	0.25	0.65	0.67	0.76	0.83	1.40	55	2.50	82.0
7	225.5	50	48.1	300.9	22.30	21.7	24.4	82.9	1213.7	4.74	1.60	0.54	0.56	0.63	0.69	1.27	58	2.19	96.3
8	225.1	52	48.2	291.2	21.96	48.7	43.2	32.1	1197.4	3.28	1.50	0.44	0.47	0.54	0.61	1.28	56	2.00	100.2

APPENDIX VI - Data from Experiment to Determine the Effects of Nozzle Orifice Size, Swirl Chamber and Concentrate Flowrate
Runs 1 - 8 13 December 1978, Runs 9 - 16 14 December 1978

Run No.	T in (C)	Ns No	T.S. (%)	F (l/h)	P (MPa)	T lp (C)	T hp (C)	Visc. (cp)	Density (kg/m3)	M (%)	S.I. (ml)	Bulk Densities (g/ml) 0	10	100	1000	ρp (g/ml)	Dsv (μm)	σg	To (C)
1	209.7	SB 61	48.3	287.0	22.66	51.2	48.8	38.2	1223.7	3.56	0.30	0.49	0.50	0.58	0.69	1.25	44	2.00	92.7
2	210.1	SB 61	48.1	265.6	18.69	50.6	47.8	39.2	1225.6	3.68	0.80	0.48	0.49	0.57	0.66	1.15	60	1.98	96.1
3	209.8	SA 54	48.9	269.3	19.08	16.8	20.3	129.9	1245.5	5.04	0.70	0.55	0.56	0.66	0.70	1.22	65	3.35	91.2
4	209.7	SA 54	48.9	288.8	23.79	18.7	21.4	110.5	1241.0	4.93	0.20	0.57	0.59	0.67	0.74	1.24	62	2.16	88.0
5	209.6	SA 61	48.0	283.9	23.29	29.0	29.0	73.0	1237.1	4.62	0.35	0.54	0.55	0.63	0.72	1.28	55	2.10	89.5
6	209.6	SA 61	48.1	266.2	18.51	23.3	25.7	94.1	1239.7	4.33	0.60.	0.54	0.55	0.63	0.70	1.22	64	2.06	92.8
7	210.3	SB 54	48.0	287.5	22.90	35.4	35.9	60.4	1232.9	4.13	0.15	0.53	0.55	0.63	0.76	1.27	48	1.86	89.9
8	210.4	SB 54	49.0	269.2	18.40	34.7	35.3	65.1	1234.6	4.25	0.50	0.55	0.56	0.64	0.72	1.24	64	1.89	92.5
9	209.8	SB 54	47.4	266.1	19.18	29.9	30.9	71.5	1211.0	4.15	0.50	0.57	0.58	0.66	0.74	1.25	53	2.12	93.5
10	209.8	SB 54	48.0	282.4	22.42	29.3	31.3	69.2	1211.5	4.38	0.30	0.57	0.58	0.67	0.77	1.28	58	1.96	90.6
11	209.8	SA 61	47.4	266.3	18.72	17.3	21.4	105.5	1235.7	4.69	0.30	0.56	0.57	0.66	0.73	1.25	57	2.24	90.7
12	209.5	SA 61	47.3	281.6	22.85	21.5	24.4	87.8	1236.3	4.97	0.50	0.58	0.59	0.67	0.77	1.26	54	2.18	88.7
13	210.2	SA 54	47.7	283.4	24.57	16.8	20.7	114.7	1207.3	4.57	0.10	0.56	0.57	0.65	0.74	1.23	50	2.20	87.9
14	209.8	SA 54	47.6	266.8	21.79	16.5	20.0	110.0	1238.2	4.35	0.30	0.55	0.57	0.65	0.73	1.22	56	2.06	89.6
15	209.7	SB 61	48.4	264.6	18.98	50.2	44.6	39.2	1236.0	3.80	0.50	0.49	0.50	0.58	0.67	1.25	53	2.06	94.0
16	209.9	SB 61	47.5	283.4	23.30	49.8	48.0	34.0	1231.5	3.47	0.20	0.49	0.50	0.57	0.69	1.24	43	1.73	91.3

Results of Simplex Pilot Plant Trial 12 May 1980

Run No.	T (C)	TS (%)	F (l/h)	M (%)	S.I. (ml)	BD (g/ml)	G (kg/h)	P1	P2	P3	P4	Total P	Current Simplex	Worst Point	Action Taken
1	216.0	47.0	280.0	3.4	0.25	0.53	155.1	0.27	0.0	0.30	0.122	0.692	- - - -	-	
2	220.0	47.4	281.0	2.9	0.25	0.52	157.2	0.72	0.0	0.35	0.070	1.140	- - - -	-	
3	217.0	48.5	281.0	3.3	0.45	0.52	161.7	0.36	1.2	0.35	0.070	1.980	- - - -	-	
4	217.0	47.4	284.0	3.0	0.20	0.53	158.9	0.63	0.0	0.30	0.028	0.958	1 2 3 4	3	refl 3
5	218.3	46.0	282.3	2.7	0.05	0.50	152.4	0.90	0.0	0.45	0.189	1.539	1 2 5 4	5	refl 2
6	214.2	46.2	283.3	3.0	0.05	0.55	153.7	0.63	0.0	0.20	0.156	0.986	1 6 5 4	5	refl 5
7	213.2	47.7	282.5	3.3	0.20	0.56	159.3	0.36	0.0	0.15	0.015	0.525	1 6 7 4	6	refl 6
8	216.6	48.5	281.1	3.3	0.45	0.54	161.7	0.36	1.2	0.25	0.084	1.894	1 8 7 4	8	rept 1
9	216.0	47.0	280.0	3.0	0.15	0.52	155.1	0.63	0.0	0.35	0.122	1.102	9 8 7 4	8	refl 4
10	213.5	48.1	278.4	3.3	0.20	0.56	158.6	0.35	0.0	0.15	0.033	0.543	9 8 7 10	8	refl 8
11	211.9	46.7	279.5	3.0	0.15	0.55	153.7	0.63	0.0	0.20	0.157	0.987	9 11 7 10	9	rept 7
12	213.2	47.7	282.5	3.2	0.15	0.55	159.3	0.45	0.0	0.20	0.015	0.665	9 11 12 10	9	refl 9
13	209.7	48.0	280.2	3.4	0.20	0.56	159.2	0.27	0.0	0.15	0.015	0.580	13 11 12 10	11	refl 11
14	212.3	49.2	281.3	3.4	0.35	0.57	164.7	0.27	0.4	0.10	0.543	1.313	13 14 12 10	14	repl 10
15	213.5	48.1	278.4	3.6	0.10	0.55	158.6	0.09	0.0	0.20	0.033	0.323	13 14 12 15	14	refl 12
16	210.5	49.2	277.4	3.7	0.40	0.54	162.3	0.00	0.8	0.25	0.138	1.188	13 14 16 15	14	refl 14

Results of Simplex Pilot Plant Trial 13 May 1980

Run No.	T (C)	TS (%)	F (l/h)	T (C)	M (%)	S.I. (ml)	BD (g/ml)	G (kg/h)	P1	P2	P3	P4	Total P	Current Simplex	Worst Point	Action Taken
1	209.7	48.0	280.2	44.0	3.0	0.05	0.57	159.2	0.63	0.0	0.10	0.040	0.770	- - - - -	-	
2	210.5	49.2	277.4	44.0	3.2	0.25	0.57	162.4	0.45	0.0	0.10	0.288	0.838	- - - - -	-	
3	213.2	47.7	282.5	44.0	2.5	0.15	0.55	159.3	1.08	0.0	0.20	0.035	1.315	- - - - -	-	
4	213.5	48.1	278.4	44.0	3.0	0.25	0.55	158.6	0.63	0.0	0.20	0.070	0.900	- - - - -	-	
5	211.7	48.3	279.6	40.0	3.5	0.25	0.56	160.0	0.18	0.0	0.15	0.000	0.330	1 2 3 4 5	3	refl 3
6	209.5	49.1	275.3	42.0	3.4	0.20	0.58	160.7	0.27	0.0	0.05	0.025	0.345	1 2 6 4 5	4	refl 4
7	207.2	49.2	277.9	41.0	3.7	0.25	0.57	162.6	0.00	0.0	0.10	0.338	0.438	1 2 6 7 5	2	refl 2
8	208.6	48.1	279.1	39.5	3.6	0.15	0.55	159.0	0.09	0.0	0.20	0.050	0.340	1 8 6 7 5	1	refl 1
9	208.8	49.3	275.8	37.3	4.1	0.40	0.55	161.8	4.16	1.2	0.20	0.162	5.722	1 8 6 7 5	9	refl 7
10	212.6	47.6	279.2	41.8	3.4	0.20	0.53	157.0	0.27	0.0	0.30	0.150	0.720	1 8 6 10 5	1	refl 1
11	211.5	48.5	276.4	37.7	3.7	0.35	0.54	159.0	0.00	0.4	0.25	0.050	0.700	11 8 6 10 5	10	rept 5
12	211.7	48.3	279.6	40.0	3.4	0.35	0.56	160.0	0.27	0.4	0.15	0.000	0.820	11 8 6 10 12	12	refl 10
13	208.1	49.4	276.0	37.8	4.1	0.50	0.58	162.3	4.16	1.6	0.35	0.265	6.735	11 8 6 13 12	13	rept 6
14	209.5	49.1	275.3	42.0	3.6	0.55	0.54	160.7	0.09	2.0	0.55	0.025	2.665	11 8 14 10 12	13	refl 14
15	212.7	47.1	281.9	37.5	3.2	0.05	0.52	156.6	0.45	0.0	0.65	0.170	1.270	11 8 15 10 12	15	rept 8
16	208.6	48.1	279.1	39.5	3.5	0.15	0.54	159.0	0.18	0.0	0.55	0.050	0.783	11 16 15 10 12	15	refl 12
17	211.0	47.4	278.7	38.3	3.3	0.05	0.54	156.0	0.36	0.0	0.55	0.020	0.930	11 16 15 10 17	15	refl 15
18	209.2	48.6	274.8	41.2	3.7	0.20	0.54	158.6	0.00	0.0	0.55	0.070	0.620	11 16 18 10 17	17	

Results of Simplex Pilot Plant Trial 14, 15 May 1980

Run No.	T (C)	TS (%)	F (l/h)	T (C)	M (%)	S.I. (ml)	BD (g/ml)	G (kg/h)	P1	P2	P3	P4	Total P	Current Simplex	Worst Point	Action Taken
1	216.0	47.5	275.0	36.0	3.3	0.25	0.57	154.3	0.36	0.0	0.05	0.285	0.695	- - - - -	-	
2	220.0	47.9	276.0	36.0	3.1	1.10	0.55	156.4	0.54	2.4	0.15	0.180	3.270	- - - - -	-	
3	217.0	49.0	276.0	36.0	3.5	0.95	0.57	160.8	0.18	2.4	0.05	0.016	2.646	- - - - -	-	
4	217.0	47.9	279.0	36.0	3.4	0.65	0.57	158.0	0.27	2.4	0.05	0.100	2.820	- - - - -	-	
5	217.5	48.0	276.5	32.0	3.7	0.85	0.57	157.1	0.00	2.4	0.05	0.145	2.595	1 2 3 4 5	2	refl 2
6	213.8	48.3	277.3	34.0	4.0	0.45	0.61	158.8	3.12	1.2	0.05	0.060	4.430	1 2 3 4 5	6	refl 4
7	218.3	48.3	272.8	34.0	3.6	1.00	0.55	156.3	0.09	2.4	0.15	0.185	2.825	1 2 3 7 5	2	refl 2
8	214.4	48.5	274.2	33.0	4.0	0.80	0.58	157.8	3.12	2.4	0.00	0.110	5.630	1 2 3 7 5	8	refl 3
9	218.9	46.7	274.2	33.0	3.3	0.40	0.54	150.8	0.36	0.8	0.20	0.460	1.820	1 2 9 7 5	2	refl 2
10	215.4	47.3	273.3	31.5	3.6	0.40	0.57	152.7	0.09	0.8	0.05	0.365	1.305	1 10 9 7 5	7	rept 1
11	216.0	47.5	275.0	36.0	3.5	0.30	0.58	154.3	0.18	0.0	0.00	0.285	0.465	11 10 9 7 5	7	refl 7
12	215.6	46.6	276.7	32.3	3.4	0.20	0.56	151.7	0.27	0.0	0.10	0.415	0.785	11 10 9 12 5	5	refl 5
13	215.5	46.2	273.1	34.4	3.6	0.10	0.54	148.2	0.09	0.0	0.20	0.590	0.880	11 10 9 12 13	9	refl 9
14	212.4	47.0	274.9	34.1	3.5	0.15	0.60	152.3	0.18	0.0	0.00	0.385	0.565	11 10 14 12 13	10	refl 10
15	214.4	46.2	276.6	36.9	3.3	0.15	0.55	150.1	0.36	0.0	0.15	0.495	1.005	11 15 14 12 13	15	refl 13
16	213.8	47.5	278.5	35.3	3.6	0.35	0.58	156.2	0.09	0.4	0.00	0.190	0.680	11 15 14 12 16	15	refl 15
17	214.5	48.1	276.0	32.0	3.7	0.45	0.57	157.2	0.00	1.2	0.05	0.140	1.390	11 17 14 12 16	17	refl 12
18	212.8	48.4	275.5	36.4	3.9	0.60	0.57	158.1	2.08	2.4	0.05	0.095	4.625	11 17 14 18 16	18	rept 11
19	216.0	47.5	275.0	36.0	3.6	0.40	0.56	154.3	0.09	0.8	0.10	0.285	1.275	19 17 14 18 16	18	refl 17
20	212.9	47.1	276.0	38.9	3.4	0.05	0.59	153.3	0.27	0.0	0.00	0.335	0.605	19 20 14 18 16	18	refl 18
21	214.8	46.1	276.7	35.8	3.2	0.05	0.59	149.8	0.45	0.0	0.00	0.510	0.960	19 20 14 21 16	19	refl 19
22	210.9	46.4	278.1	36.1	3.7	0.05	0.59	151.7	0.00	0.0	0.00	0.415	0.415	22 20 14 21 16	21	refl 21
23	210.2	47.9	277.1	36.4	3.8	0.25	0.60	157.0	1.04	0.0	0.00	0.150	1.190	22 20 14 23 16	23	rept 14
24	212.4	47.0	274.9	34.1	3.9	0.25	0.59	152.3	2.08	0.0	0.00	0.385	2.465	22 20 24 23 16	24	refl 16
25	209.4	46.7	274.6	37.5	3.7	0.05	0.59	151.0	0.00	0.0	0.00	0.450	0.450	22 20 24 23 25	24	refl 24
26	209.3	47.0	278.0	40.4	3.6	0.05	0.59	154.0	0.09	0.0	0.00	0.300	0.390	22 20 26 23 25	23	refl 23
27	211.1	45.8	276.3	40.1	3.2	0.05	0.56	148.4	0.45	0.0	0.10	0.580	1.130	22 20 26 27 25	27	refl 20
28	207.6	45.8	277.5	38.2	3.6	0.05	0.61	149.1	0.09	0.0	0.05	0.545	0.685	22 28 26 27 25	27	rept 22
29	210.9	46.4	278.1	36.1	3.7	0.05	0.61	151.7	0.00	0.0	0.05	0.415	0.455	29 28 26 27 25	27	refl 27
30	207.5	47.2	277.8	36.0	4.0	0.15	0.64	154.7	3.12	0.0	0.20	0.265	3.585	29 28 26 30 25	30	refl 28
31	210.9	47.9	276.7	36.8	4.1	0.15	0.63	156.8	4.16	0.0	0.15	0.160	4.470	29 31 26 30 25	31	rept 25
32	209.4	46.7	274.6	37.5	3.5	0.05	0.60	151.0	0.18	0.0	0.00	0.450	0.630	29 31 26 30 32	31	

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