AN EXPERIMENTAL PINEAPPLE JUICE CONCENTRATE PLANT INCORPORATING FLAVOUR RECOVERY

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SUMMARY

In order to investigate the concentration of Queensland-produced pineapple juice an experimental plant incorporating an ester recovery section has been designed and constructed. Pulp in the fresh juice is reduced to less than 1 per cent. by screening and centrifuging and then the juice is fed into a turbulent thin film evaporator which strips off the esters during concentration under vacuum. Esters and water are condensed together in a refrigerated surface condenser. The distillate is partially vaporized and then fractionated at atmospheric pressure to produce ester concentrate of 250-fold which is reincorporated with the concentrated juice (50–60° Brix). A detailed description of construction and operation of the plant is given.

I. INTRODUCTION

The production of single-strength canned and bottled fruit juices in Australia amounts to approximately 3½ million gallons annually, of which about 3 million gallons are pineapple juice. The freight and container costs involved in exporting pineapple juice from Queensland to other Australian States and overseas is relatively high when it is considered that the single-strength juice contains over 80 per cent. of water. In 1958 a project was initiated to investigate concentration as a means of reducing these costs in order to assist Queensland's major horticultural industry.

In Queensland, pineapple juice is expressed from the shell (comprising the skin, the flesh up to 1-in. thick, which is removed during the peeling operation), the core and the trimmings from the cylinders of flesh. Since this method differed from that used in Hawaii, where skins are not pressed, and this juice may have contained undesirable constituents extracted from the skin (Seale 1953), it was considered essential to install a small experimental juice concentration plant to undertake this work.

Although the production of frozen concentrated juices, particularly orange juice, has become a well-established industry in many countries, frozen concentrated pineapple juice is manufactured only in Hawaii.

Anon (1952), Smyser (1952), Jefferson and Lloyd (1952), Seale (1953), Tressler and Joslyn (1954) and Kefford (1954) reported that it was necessary to recover and reincorporate the esters† volatilized during the concentration of pineapple juice. The Hawaiian plant was described as a triple-effect evaporator through which the juice was passed twice. During the first pass 25 per cent. of the juice was evaporated, while in the second stage it was concentrated to about 66° Brix. Practically all esters were recovered from the calandrias of the second and third effect in the first pass.

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† "Ester" in this paper refers to volatile flavouring constituents, including esters, ketones, aldehydes, alcohols, etc.

In order to simplify production of juice concentrates on a laboratory scale, it was decided to use a flash evaporator similar to that described by Dimick and Makower (1951) as the first effect to strip off the esters with 20–25 per cent. of the water, and a single-pass turbulent thin-film exaporator for the second effect. This equipment, which was described briefly by Leverington (1962), has been exhaustively tested and modifications made until satisfactory operation has been attained.

II. INVESTIGATIONS LEADING TO MODIFICATIONS

Since Haagen-Smit *et al.* (1954) and Connell (1964) had reported that ethyl acetate was generally present in much higher concentrations than all other esters in pineapple, preliminary experiments with the pilot plant were carried out with an aqueous solution of ethyl acetate before proceeding with freshly expressed pineapple juice. The effectiveness of stripping methods was judged by the amount of esters recovered when determined as ethyl acetate by a modification of the method of Thompson (1950). Variables investigated with regard to ester stripping included feed rate, steam pressure, degree of vaporization, vacuum, cooling brine temperature, heating surface and diameter of vaporizing tubes. Ester recoveries of 60–70 per cent. could be obtained on runs of up to 2 hr. However, the fine stainless-steel preheating tubes (0·078 in. I.D.) readily fouled up, which is in contrast to the work reported by Walker and Patterson (1955), who used similar sized tubes. It appeared therefore that the recommendation made by Claffey *et al.* (1958), that a velocity of 20 ft/sec for pectinaceous juices through the heat exchanger tubes was necessary, may also apply to pineapple juice, which is very low in pectin. As the plant was designed to process 4-5 Imp. gal of juice per hour, it was obvious that to obtain this high velocity the diameter of the preheating tubes would have been smaller than practical limits. It was therefore decided that the juice would be evaporated to the required concentration in one pass of the turbulent thin-film evaporator and all the distillate fractionated. This modified technique necessitated the installation of a pasteurizer operating at 165°F. This reduction in temperature of pasteurization reduced the likelihood of adverse heating effects on the organoleptic qualities of the juice.

Since experiments had shown that there was a loss of aroma-bearing constituents in the column bottoms and that the maximum obtainable fold of the essence was rather limited, the height of the fractionating column was extended so that the number of theoretical plates would approach the 22 plates used commercially (Tressler and Joslyn 1954). Due to the difficulties experienced with reflux heating by either electrical means or heat exchangers and the small advantages gained in using this principle in a plant of this size, its use was discontinued.

The operation of the fractionating column under vacuum and the maintenance of the system at equilibrium proved very difficult. This was due to considerable pressure drop through the column as well as intermittent flooding of the scrubber and column caused by small fluctuations in vacuum. Although Smyser (1952), Seale (1953), Tressler and Joslyn (1954) and Wood (1961)

described vacuum fractionation plant for pineapple esters, the necessity for low-temperature fractionation was not indicated. Hugo (1959) described an experimental plant operated entirely under vacuum which he used for ester recovery and the concentration of pineapple juice, but in subsequent private communications he has described the difficulties experienced in recovering these esters fractionated under vacuum.

Since Milleville and Eskew (1944), Philips *et al.* (1951), Eskew *et al.* (1951*a*, 1951*b*, 1951*c*), Dimick and Simone (1952), Eskew *et al.* (1952), Walker *et al.* (1954), Walker and Patterson (1955), Eisenhardt *et al.* (1958), Claffey *et al.* (1958) and Eskew *et al.* (1959) have reported the successful fractionation at atmospheric pressure of a number of fruit juices, including apple, peach, pear, berry and grape, it was decided to investigate the feasibility of fractionating pineapple esters under the same conditions. The fractionating system was therefore converted to atmospheric pressure. Results to date have been quite satisfactory.

III. OUTLINE OF PROCESS

Photographs of the plant are shown in Figures 1 and 2 and a flow diagram of the process is illustrated in Figure 3. The commercially expressed juice is pasteurized at 165°F, cooled, screened, chilled to 35°F, centrifuged to less than 1 per cent. insoluble solids, homogenized at 2,500 lb/in.^{2} and then evaporated under vacuum in a single pass to approximately 60° Brix. During evaporation, esters are distilled off and condensed with the water. This distillate is then fractionated at atmospheric pressure and the esters removed from the system at a concentration of approximately 250-fold. These are blended with the concentrated juice, which is then canned and quick-frozen.

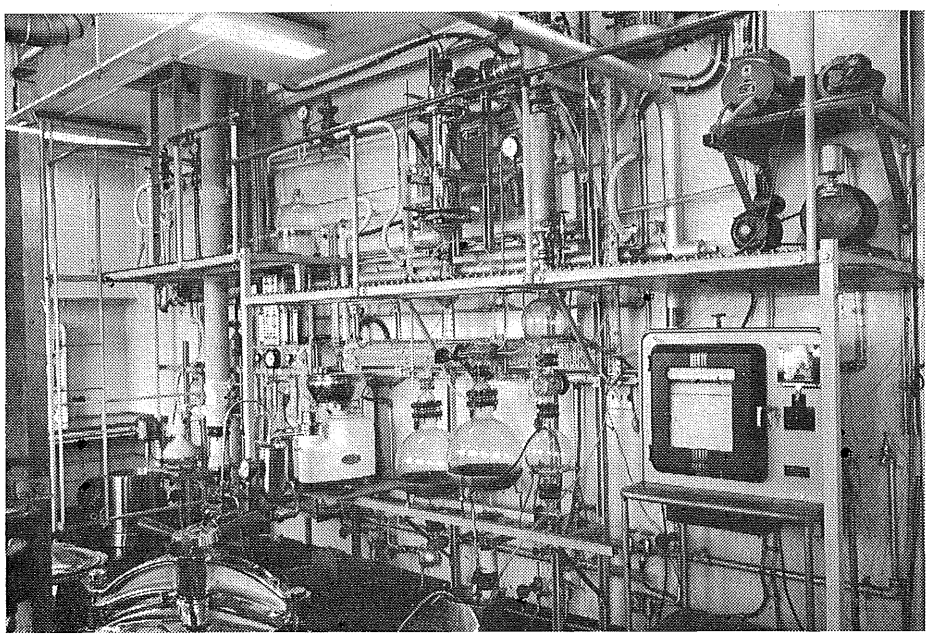

Fig. 1.—General view of pineapple juice concentration plant.

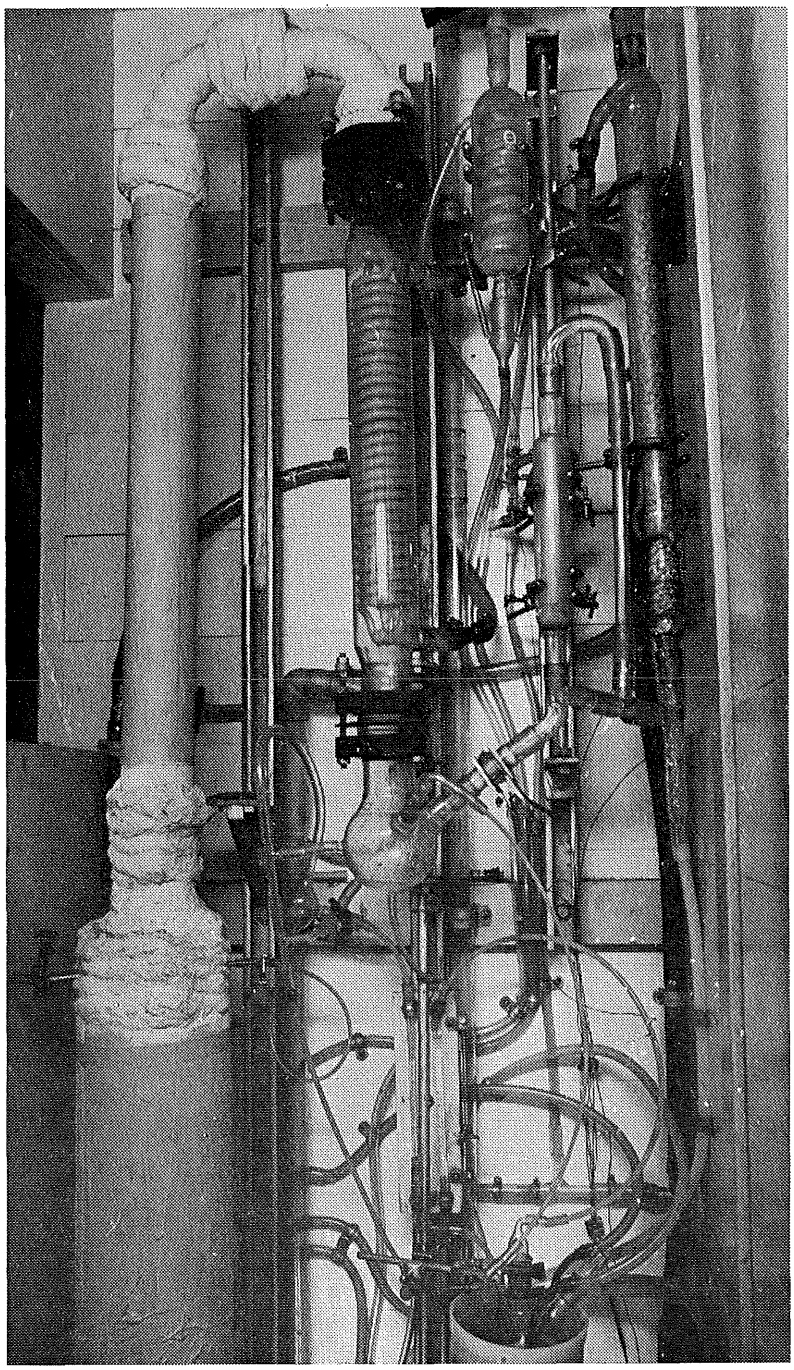

Fig. 2.—Upper section fractionating system showing fractionating column, essence condenser, reflux splitter, vent cooler, scrubber, scrubber liquid cooler and essence receiver.

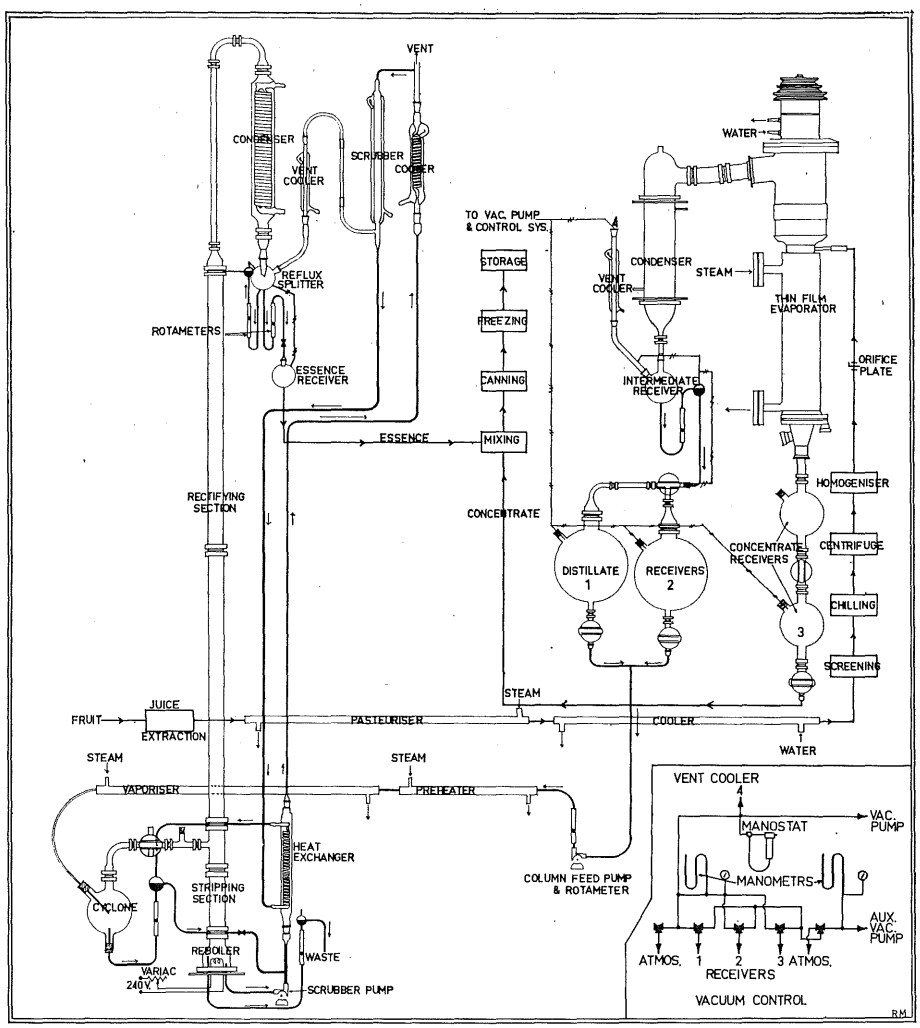

Fig. 3.—Flow diagram of pineapple juice concentration plant.

IV. CONSTRUCTION AND METHOD OF OPERATION

Juice Preparation.—Freshly expressed pineapple juice containing 20 per cent. pulp is drawn from a nearby cannery and pasteurized promptly at 165°F to prevent fermentation during processing. Continuously agitated juice is fed by a Mono* pump at 20 Imp. gal/hr through an 8-ft shell and tube heat exchanger consisting of a ½-in. O.D. 20-gauge stainless-steel† tube in which is centrally

* Mention of trade name or company in this paper does not imply recommendation or endorsement by the Queensland Department of Primary Industries over those not mentioned.

† Stainless-steel in this paper refers to 18/8 Mo grade.

placed a core of $\frac{5}{16}$-in. stainless-steel rod to ensure rapid flow over the hot surface. Steam at 25 lb/in.^{2} raises the temperature of the juice from 70°F to 165°F and then it is cooled to about 100°F in a similar 11-ft shell and tube heat exchanger. All connections are made with nylon pipe and compression fittings.

The cooled juice is then passed through a vibrating screen (shown in Figure 4) to remove about 80 per cent. of the pulp and some of the precipitated protein. Vibration of the screen is obtained by a variable speed cam (750–1450 r.p.m.), the stroke of which can be adjusted between $\frac{1}{32}$ and $\frac{6}{32}$ in. The upper screen is 20 mesh and the lower 42. Careful adjustment of the slope of the screen is essential to ensure efficient separation and to prevent excessive loss of juice with the discharged pulp. The maximum capacity of the unit is about 40 gal per hr when fed with juice containing 20 per cent. of pulp.

The screened juice runs into a refrigerated tank, where it is cooled rapidly to approximately 35°F to minimize microbial activity and ester losses. Due to the variation in total soluble solids of pineapples as pointed out by Leverington (1962), it is necessary to adjust the sugar concentration to 13° Brix by the addition of cane sugar to produce a standard product. Agitation of the chilled juice and suspended pulp is essential to ensure that the centrifuge is fed with homogeneous product.

The juice is pumped to a De Laval laboratory centrifuge, which not only removes fine pulp and particles of foreign matter but also most of the precipitated protein. Unfortunately, during this stage a considerable amount of froth is produced which is difficult to eliminate even with the addition of various antifoam agents. Provided foam does not accumulate in the juice feed no difficulties are experienced.

Approximately 1 per cent. protein is precipitated after centrifuging. To prevent this settling in the concentrate, thereby detracting from the appearance, the juice is homogenized at 2500 lb/in.^{2} in a Manton Gaulin 2-stage homogenizer.

Juice Concentration.—Although the juice was originally pumped into the evaporator using a positive delivery pump with by-pass for flow control, intermittent fluctuations in flow caused evaporation difficulties. A steadier flow has been obtained by drawing the juice via a rotameter into the evaporator by vacuum through a stainless-steel 0·04-in. capillary or alternatively through a $\frac{3}{32}$-in. needle valve.

The original evaporator described briefly by Leverington (1962) did not meet specification requirements and was replaced by a Luwa Model 020 turbulent thin-film evaporator. This unit, which has a 1·4-sq. ft. heating surface and a 2000 r.p.m. 4-bladed rotor with $\frac{1}{32}$-in. clearance, is shown in Figure 1. A modified $\frac{1}{2}$-in. pilot-operated reduction valve has been used to provide steam at $\frac{1}{2}$–15 lb/in.^{2} in conjunction with a $\frac{1}{2}$-in. ball-float steam trap.

The separation head of the evaporator has proved effective in preventing entrainment. To prevent any possible distortion of the shell during operation, which would in turn cause the rotor blades to scrape the wall, flexible metal bellows-type hoses are used for steam connections. Due to vibration of the evaporator, flexible connections in the form of Tygon tubing between the glass condenser and concentrate receivers have been essential. The lower carbon

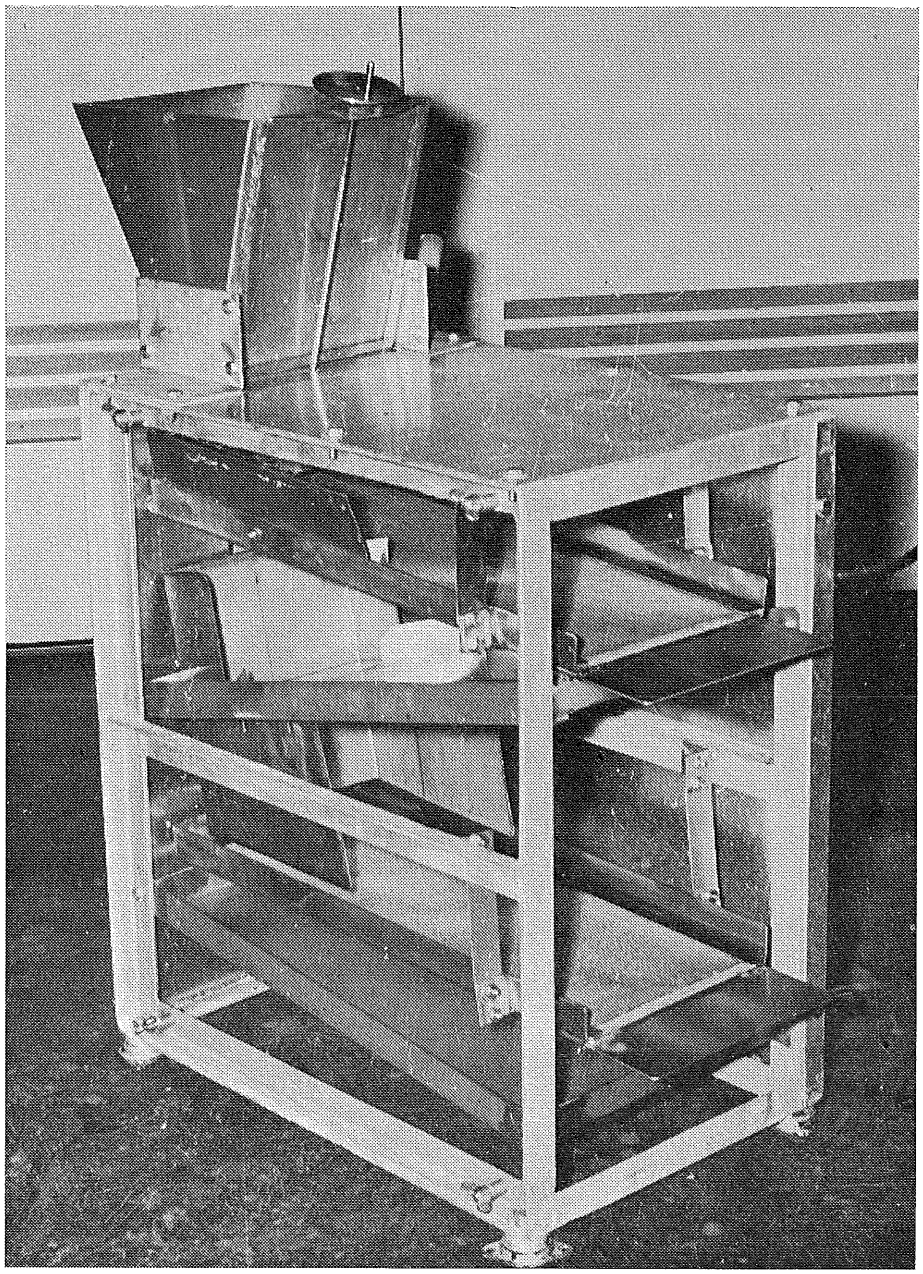

Fig. 4.—Vibrating screen.

bearing, which is lubricated by water metered in at 2 ml/min, has had to be frequently replaced, as small chips of carbon tend to break off and contaminate the products. An experimental Teflon bearing is now under trial. The concentrate receivers consist of two 5-l Q.V.F. flasks connected in series by means of a 1½-in. stopcock. This permits the lower flask to be discharged while the plant

is in operation. Since refrigeration is not provided for these flasks, there is a tendency for the concentrate to boil in these receivers on a hot day if the plant is operated at pressures below 25 mm Hg.

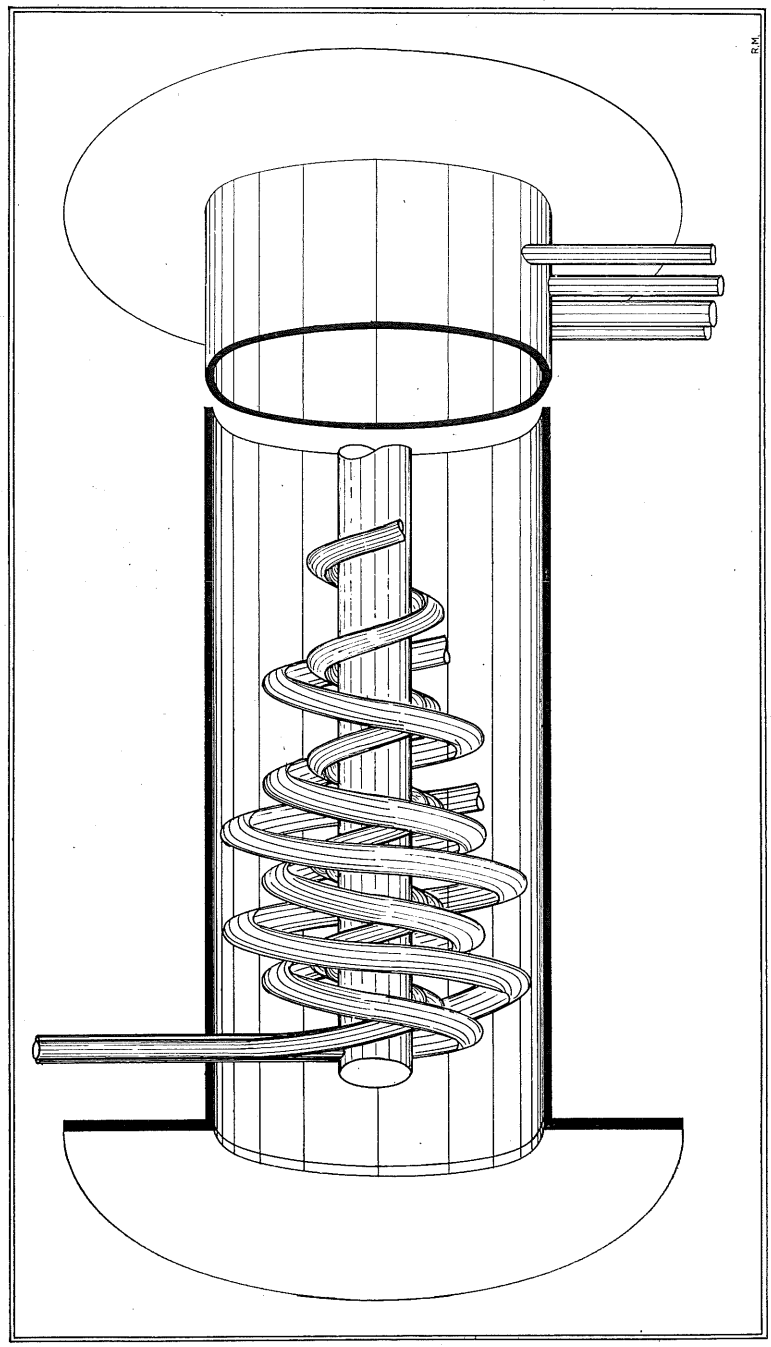

Fig. 5.—Main condenser.

The vapour outlet of the evaporator is connected to the stainless-steel condenser by means of a 3-in. line in which is fitted the bulb of a distant-reading thermometer. In order to reduce pressure drop in the condenser to a minimum, it was designed so that it had a free cross-sectional area equivalent to a 3-in. pipe. The cooling surface, comprising two ½-in. coils, one ⅜-in. coil and a 1¼-in. tube, has a total surface area of 9 sq. ft., which has proved adequate when using 20 per cent. glycol brine at 32°F. The brine is distributed in parallel to each cooling tube by means of a manifold, which is provided with screw clip valves to ensure that the temperature rise in each coil is constant. The design is shown in Figure 5. The intermediate condensate receiver consists of a modified 1-l flask to which is attached a 1¼-in. side arm and a 2 sq. ft. glass surface condenser which acts as a vent cooler. Every precaution is taken to ensure that water vapour is not visibly being condensed on this vent cooler and therefore that loss of esters to the vacuum is at an extreme minimum. The condensate receiver is normally half full, so that any slight variation in condensate rate due to hold up of condensed water on the coils does not cause fluctuations in the rotameter readings. A tube from the bottom of the intermediate condensate receiver is connected via a rotameter to a 1-in. 2-way T stopcock which permits distillate at 50°F to be directed to either of the two 20-l distillate receivers, which have ⅝-in. draincocks. To avoid loss of esters, vacuum is not applied directly on these flasks during filling, but a constant pressure is maintained by means of an 8-mm balancing tube which is looped from just ahead of the 2-way stopcock to the head of siphon breaker and in turn to the vent cooler line. This siphon breaker is fitted above the rotameter to ensure a steady flow and therefore accurate readings.

Ester Fractionation.—The ester-containing distillates, which are stored overnight in stainless-steel drums at 35°F, are pumped via a rotameter into the preheater, which consists of a 5-ft length of 0·078-in. I.D. stainless steel tubing encased in a steam jacket. The heated fluid then discharges into the vaporizer, which is a steam-jacketed 5-ft length of ½-in. O.D. stainless steel tubing. Each heat exchanger has its own pilot-operated reducing valve which can be regulated between ½ lb/in.^{2} and 15 lb/in.^{2} depending upon the degree of vaporization required. Normal feed rate is 240 c.c. per min, of which approximately 30 per cent. is vaporized. Liquid/vapour separation occurs as the mixture enters the 5-l cyclone separator, the vapour passing over into the vapour feed plate of the fractionating column. A 2-way T stopcock has been fitted between the column and the cyclone to enable samples of vapour to be withdrawn if required. The stripped water discharging from the cyclone passes through a rotameter and is fed into the bottom feed plate of the fractionating column in order to distill off any esters which may have been retained. To balance pressures in this part of the system, the head of the siphon breaker on top of the rotameter is connected to a feed plate on top of the stripping section of the column. The cyclone (which is heavily insulated above the liquid level) normally contains about 1½ l of water.

The fractionating column has been constructed of 3-in. Q.V.F. glass pipe sections and packed with ¼-in. glass raschig rings, the free volume being 74 per cent. The stripping and rectifying sections contain 18 in. and 100 in. of packing

respectively. The reboiler consists of a 6 in. x 3 in. pipe reducer with a ¼-in. stainless-steel base plate in which there is fitted two ¼-in. liquid discharge tubes and a 1800W 240V nichrome element wound on a ceramic bobbin. This element is mounted on ⅛-in. stainless-steel conductors which are insulated with nylon bushes as they pass to the junction box on the lower side of the reboiler plate. The heat output of this element is controlled by a 10A Variac, which is in the active line of the power supply to prevent any heating effects due to conductivity of the water. Column bottoms are discharged by an overflow system and flow rates are indicated in a rotameter. A series of experiments have been conducted at various reboiler settings and 110V has been found to be the maximum to ensure that the column does not flood when the system is fed with water at the rate of 240 ml/min, of which 30 per cent. is vaporized. The vapour feed plate consists of a 1-in. pipe discharging into the column. All liquid feed plates are constructed of ½-in. stainless-steel rings through which pass ¼-in. lines, the discharge point of which is exactly in the centre of the column and facing downwards. Three such plates are fitted, viz. one at the bottom of the stripping section to receive the cyclone liquid discharge, one at the vapour feed level where scrubber liquid is discharged back into the column, and the third at the top above the rectifying section to feed reflux liquid back into the system. The whole of the fractionating column and the 1-in. vapour pipe leading to the condenser is covered with 1½-in. magnesia insulation or tightly bound 1-in. asbestos rope. A 1-in. dia. inspection hole is cut in the insulation at the bottom of the rectifying section to observe any column flooding.

The glass coil condenser has a surface area of 3½ sq. ft. and a free cross-sectional area of 0·8 sq. in. The condensed liquid containing the esters is received in a reflux splitter, which is a modified 500-ml flask with take-off tubes for reflux and ester. To equalize pressures in this section of the plant, one side arm is connected to a siphon breaker on the reflux line and also to the essence receiver. Another arm is connected to a 650-sq. cm. glass condenser which acts as a vent cooler. The essence (ester concentrate) take-off is controlled by throttling the line, the balance of the distillate passing back as reflux. Both reflux and essence flow rates are indicated by rotameters. The scrubbing tower consists of a 350-sq. cm. Liebig condenser packed with ¼-in. ceramic berl saddles up through which vent gases pass counter-current to the scrubbing liquid. For scrubbing purposes, column bottoms are drawn from the reboiler and pumped through one side of a heat exchanger and then through a refrigerated cooler before being fed to the top of the refrigerated vent gas scrubber at 40 c.c./min. The liquid then passes through the other side of the heat exchanger and is discharged into the second column feed plate mentioned above. This arrangement results in water being fed into the scrubber at 35°F and it is discharged back into the column at about 120°F. To ensure that cavitation does not occur in the centrifugal scrubber pump, most of the boiling liquid it handles is bypassed back into the lower feed plate. The fractionating system is virtually closed, the only openings to atmosphere being through the liquid lock on the column discharge and the top of the scrubber.

In addition to dial-type and mercury thermometers placed at strategic points throughout the plant, 25 copper constantan thermocouples are fixed in thermometer pockets and on liquid lines. The thermocouples are connected to an electronic recorder which can be seen in Figure 1. All condenser refrigerant inlet and outlet lines have short sections of metal tube to which the thermocouple is bound with tape and asbestos rope.

Ancillary Equipment.—The evaporator, along with all the other associated equipment, is mounted on galvanized Unistrut scaffolding which is bolted to the brick wall as well as being supported by the catwalk which forms an integral part of the framework. This structural material was selected because of the infinite adjustment available in a channel of this type. The decking of the catwalk is galvanized heavy-gauge expanded metal welded to the framing. Small pieces of equipment (such as condensers) are fixed with laboratory retort clamps which are attached to vertical ½-in. aluminium alloy rods bolted to the main framework. Pipework, which is polyvinyl chloride, polythene, nylon, glass and butyrate, is generally mounted on galvanized cable tray and given distinctive coding for identification.

As shown in Figure 3, there are two independent vacuum systems in use. The main system, which maintains the reduced pressure in the evaporator and receivers, utilizes a 5 c.f.m. water-cooled oil-type gas ballast pump. A close check on air leaks is imperative to eliminate the risk of esters being carried over in the non-condensables (Tressler and Joslyn 1954). To control vacuum at the desired level, a Cartesian manostat was installed in series in the vacuum line as recommended by Walker and Patterson (1951), but it was soon found that a laboratory manostat was entirely inadequate due to the limited orifice size. The exhaust orifice of the manostat has therefore been modified in a manner recommended by Spadaro, Vix, and Gastrock (1946). It is connected in such a way that control is by means of a bleed into the vacuum line close to but just after the vent cooler of the evaporator. A mercury manometer with a scale of ± 90 mm is connected into the vacuum line.

The auxiliary vacuum system operated by a 1 c.f.m. oil-type vacuum pump with its associated manometer is installed to evacuate receivers after their contents have been discharged. These receivers are connected over to the main system when the auxiliary manometer reading corresponds with the main manometer reading. The sizing of vacuum lines is of extreme importance and to minimize pressure drops clear rigid 1¼-in. O.D. butyrate pipe is used for all the main lines.

The primary refrigeration system consists of a 5-ton sealed unit compressor, a 7½-ton evaporative cooler and two shell and tube heat exchangers through which passes the secondary refrigerant, which is an aqueous 20 per cent. ethylene glycol solution. This brine is circulated through condensers, vent coolers and the scrubbing system by a centrifugal pump at approximately 250 Imp. gal/hr. Adequate safeguards, including a control thermostat, safety thermostat as well as a differential pressure switch to prevent freezing of the glycol in the heat exchanger, have been installed. To avoid fluctuations in brine temperature as the compressor cuts in and cuts out, a 6 Imp. gal. header tank has been installed

and fitted with a 2kW element and thermostat so that the refrigeration capacity can be balanced against the heat load. In this way temperature of the brine can be controlled to ± 1°F.

V. NATURE OF DATA OBTAINED

It has not been practicable in a paper of this type to present all the data obtained during the development of this plant, but typical operating conditions now in use are as follows:—

Evaporation

Juice feed; 4–5 Imp. gal/hr; Brix 13°; temperature 50°F; pulp >1 per cent.
Evaporator jacket temperature: 212–240°F.
Product vapour—temperature: 70–140°F; pressure 20–150 mm Hg.
Distillate rate: 240–400 c.c./min.
Concentrate Brix: 50–70°.

Fractionation

Column feed: 250 c.c./min; 27 p.p.m. ester.
Preheater steam pressure: 5½ lb/in.^{2}; vaporizer steam pressure: 6 lb/in.^{2}.
Degree of vaporization: 37 per cent.; Reboiler voltage: 110.
Reflux ratio: 36:1; Essence fold: 250.
Recovery of essence based on column feed: 82 per cent.
Reflux rate: 51 c.c./min; Ester rate: 1·3 c.c./min.
Cyclone discharge rate 158 c.c./min; Reboiler discharge rate: 249 c.c./min.
Scrubber flow rate: 16 c.c./min; Ester in scrubber liquid: 9 p.p.m.

A suitable pilot plant having been developed, experimental work has been commenced to determine the suitability of Queensland-produced pineapple juice for concentration. Factors to be investigated include pasteurization temperature, enzyme destruction, protein precipitation, evaporation temperature, ester variation between and within season, importance of ester losses, desirability of cut-back juice, optimum ester level for summer and winter juices, feasibility of blending summer and winter esters, storage properties of concentrates and pasteurized concentrates.

VI. ACKNOWLEDGEMENTS

The authors wish to acknowledge the assistance of Messrs. N. Herriman and G. Harrison of the Public Works Department and Mr. P. C. Brookes of the University of Queensland for assistance in design, the Pineapple Sectional Group Committee of the Committee of Direction of Fruit Marketing for making the initial supplies of juice available, and the contributions of various members of the staff of this Laboratory, particularly Mr. R. G. Prince for assisting in construction and operation and Mr. C. J. Bagley for construction and modification.

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(Received for publication September 30, 1963)