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THE UNIVERSITY OF ALBERTA

ON THE EFFICIENT USE OF STEAM IN
THE DEODORIZATION OF MILK AND CREAM

by



TERRY W. RACZUK

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH
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DEPARTMENT OF FOOD SCIENCE

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THE UNIVERSITY OF ALBERTA
FACULTY OF GRADUATE STUDIES AND RESEARCH

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research, for acceptance, a thesis entitled ON THE EFFICIENT USE OF STEAM IN THE DEODORIZATION OF MILK AND CREAM submitted by T. W. RACZUK in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

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ABSTRACT

In the dairy industry there are important physical and economic limitations to the amount of steam available for the deodorization of milk and cream. The present work is a study of a number of factors which affect the efficiency of the use of steam in the deodorization process.

Vapour liquid equilibrium coefficients have been determined using a dynamic equilibrium still for diacetyl in water and in skim milk at atmospheric and sub-atmospheric pressures. This compound was used as a reference taint compound in deodorization in a modified high speed laboratory type bowl centrifuge. The concentration of diacetyl in the deodorized liquid was calculated for equilibrium between the vapour, aqueous and fat phases in the outlet streams, and the closeness of approach to equilibrium was expressed as an approach ratio, i.e. the ratio of actual outlet stream concentration minus the equilibrium outlet concentration to inlet concentration minus the equilibrium outlet concentration.

The effects of liquid flow rate, vapour flow rate, speed of centrifuge and initial concentration of taint compound in the liquid feed on the approach ratio were examined. The results for eight tests of various combinations of liquid (water, skim milk, milk and cream), input with and without spray, and thickness of fluid layer on the inside of the

centrifuge bowl are reported. In general the approach ratios and residence times showed that the product of mass transfer coefficient and surface area ($k_L a$) increased strongly with increasing liquid flow rate and decreased with increasing fat content. With the thin liquid layer, higher speeds gave greater total mass transfer, as did spray injection. Vapour flow rate did not have a significant effect on the approach ratio. The effect of concentration of diacetyl in the liquid feed on the approach ratio was quite significant in some tests but no reasons were apparent.

An analysis of various systems of interconnecting equilibrium deodorizing stages has been done. There are

1. a system in which a fat depleted or fat enriched stream is recycled (with or without deodorization) and mixed with the liquid feed stream;
2. a stepwise flash system in which steam is injected into and condensed by the liquid feed and is then flashed off in a series of stages with decreasing pressure;
3. a crossflow system in which the cream passes through a number of stages and is contacted in each by fresh steam;
4. a countercurrent system in which steam and cream flow in opposite directions through a series of stages.

Taint reduction ratios have been calculated for systems 2., 3., and 4. for three compounds for which adequate equilibrium data is available, i.e. diacetyl, acetoin and

benzyl mercaptan.

It was found that recycling (system 1. above) did not increase the steam economy of the process. The steam economies in the multistage stepwise flash and crossflow systems were similar and better than those obtainable with a single stage system. Use of five or more stages did not greatly improve the taint reduction ratio over that obtainable with four stages.

Countercurrent systems were more efficient in the use of steam than either of the other multistage systems (2. and 3. above). An increase in the steam flow to a countercurrent system gave a greater increase in the taint reduction than did the same increase in steam flow to either of the other multistage systems.

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I. INTRODUCTION

A. OFF-FLAVORS IN MILK

Flavor is a most important quality factor influencing customer selection and consumption of milk and milk products. Off-flavors in milk can be produced by the cow under disturbed conditions of metabolism, (e.g. ketosis); formed by bacteria after the production of the milk; absorbed from the atmosphere via the lungs of the cow; or derived from the feed of the animal. This latter source of off-flavor can be the normal feed available at the particular time and/or the weeds present and incorporated with the feed.

Off-flavors caused by weeds and feed are the most frequently encountered (up to 75% of total flavor defects) and the main attempts by the dairy industry to eliminate these defects in the 200 years since they were first observed has been to prevent the cow from eating the material causing the off-flavors. However, preventative measures can only be taken if alternate supplies of food are available for the cow, particularly in the short period of time directly before milking.

Because it is not always possible to prevent feed, weed and the other types of off-flavor from occurring, the industry has used various techniques for the removal of the off-flavor. This study deals with some of the

engineering aspects of odor removal, in particular with the efficient use of steam in deodorizing systems.

II. REVIEW OF THE LITERATURE

A. MILK AND CREAM AS HETEROGENEOUS SYSTEMS

Kostygov (1966) describes milk as a polyphase, polydispersed heterogeneous liquid. Webb and Johnson (1965, p. 407) describe milk as a discontinuous oil or fat phase suspended in the milk plasma. The discontinuous phase which represents about 3 - 4% by weight of the milk as produced, is in the form of tiny globules ranging in size from 0.1 to 10 microns with an average diameter of about 2 - 3 microns.

The size of the fat globule can be decreased by homogenization. An excellent study on the influence of homogenization on the size distribution of milk fat globules is given by Walstra (1966). The numerous methods for determining the number and size distribution include microscopy, photo micrography, spectroturbidity and electronic counting methods. A good discussion and comparison of these methods has been given by Walstra (1969).

The dispersed phase has a considerable interfacial area. Surface active agents (e.g. proteins) are also abundantly present and they tend to collect at the interface to form complex membranes which have a profound effect on the behaviour of the fat droplets.

King (1955) postulated that the membrane structure was a layer of phospholipid with other less polar lipids

and molecules of high melting triglycerides extending from the fat globule core in concentric layers according to polarity. The outmost layer was depicted as being composed of phospholipids which in turn were closely associated with an adsorbed layer of protein. The nature of the compounds in the interface has been the subject of intense study and the knowledge to 1962 has been summarized by Brunner (1965). More recent reviews have been by Koyama (1967) [in Japanese] and Prentice (1969). The general consensus is that the membrane on the milk fat globule consists essentially of two discrete layers, i.e. the inner layer which is firmly bound to the globule and is a true biological membrane consisting of globular protein and an outer layer consisting mainly of phospholipids. A layer of high melting triglycerides is situated just inside the inner globular protein membrane with the lower melting triglycerides forming the bulk of the interior of the milk fat globule. The outer layers particularly have a strong influence on the stability of the milk emulsion.

The other major constituents of milk are water (86 - 88%), lactose (ca. 4.5%), casein (2.5%), lactalbumin and lactoglobulin (0.4 - 0.7%), minerals, vitamins and enzymes. The lactose and protein compounds in the aqueous phase give viscosities and densities greater than those of water. The viscosity of skim milk is 1.5 to 2 times that of water and the density of skim milk is 3.2 to 3.5% greater than that of water at the same temperature.

B. NORMAL MILK FLAVOR

Milk can be described as a bland food substance imparting a smooth rich feeling in the mouth, combined with a slightly sweet or salty sensation. The flavor should never be bitter or sour nor should off-odors be present.

Flavor has been described as a blend of sensations combining feel, temperature and pungency or blandness, as well as smell and taste (Moncrief, 1967, p. 650). To produce a taste sensation, a substance must be in solution in order to diffuse into the receptor sites in the taste buds. To have an odor, the substance must be volatile. The odor is detected by the olfactory nerves in the nasal passages. The number of discernable odors is large and very difficult to describe except by comparison. The tactile or "feel" characteristics also contribute to flavor.

It should be noted that the extremely bland nature of milk makes it particularly susceptible to off-flavors caused by minute quantities of abnormal substances.

C. OFF-FLAVORS AND ODORS

i. Types of Off-flavors

a. Feed and Weed Flavors

Substances in the material ingested by the cow can cause off-flavors in the milk. The first mention in

the literature that off-flavors in milk could stem from the diet of the cow was by Bradley (1757). The relationship of the off-flavor to the feed or weeds simply resulted from the observation that the smell from the milk resembled that of the weed or feed.

Since 1757, there have been many studies on the effects of various feeds on the flavor of milk. A comprehensive review on off-flavors in milk to 1953 is given by Strobel et al.

The importance of feed and weed flavors in milk has been shown by several surveys. Dahlberg et al. (1953) in a study sponsored by the National Research Council (U.S.) covering a milk supply of eight widely separated cities found that of 169 samples, 127 (or 75%) were criticized for having a feed flavor. It was shown by Downs et al. (1954) that more than 44% of the milk samples used in Collegiate Students Dairy Products Judging Contests since World War II contained defects listed as feed or weed either alone or in combination with other defects. The Food Industry Manual (1958, p. 440-41) shows that in 6758 samples of defective milk, feed flavors were rated 2305 times (i.e. 36% of total defects).

b. Off-flavors Caused by Metabolic Disorders

Metabolic disorders can also cause severe off-

flavor problems, especially ketosis, which is fairly common in late winter and early spring and particularly after calving (Jenness and Patton, 1959, p. 391).

c. Miscellaneous Off-flavors

Other types of flavor defect which have been detected in raw, untreated milk are "cowy", rancid, stale, salty, flat, sweet and bitter (Food Industry Manual, 1958, p. 440-41). Some of these defects may be due to bacterial spoilage and enzymic and/or non-enzymic chemical reactions taking place after the milk has been produced. Other off-flavors which occur have sources which have not yet been identified.

ii. Prevention or Minimization of Off-flavors

a. Feed and Weed Off-flavors

Often it is not possible to eliminate the tainting substance from the feed because of lack of alternate feed supplies. Elimination of feed and weed off-flavors in the milk is a problem because of the ease with which the tainting substances can be transferred from the feed to the milk.

The feed and weed flavors can be transferred to the milk via the cow's respiratory system and via the digestive tract. That transfer to the milk is by far the quickest via the respiratory tract was shown by Babcock

in 1925, subsequent work by MacDonald and Crawford (1927) and recently by the U.S. Department of Agriculture (Dougherty, 1970). Garlic flavors were detected in milk in as little as 1 minute after inhalation. Longer times were necessary if the inhalation factor was eliminated, and transfer was only from the digestive tract to the blood and thence to the milk. Jenness and Patton (1959, p. 389) state that garlic flavor can be detected in milk in as little as 20 minutes after subjecting the cow to garlic vapours. Peterson and Brereton (1942) also show that some materials were transmitted to the mammary gland via the respiratory system. In any case, odors belched from the rumen are inhaled into the lungs and hence both pathways to the milk contribute to odor transfer.

Blood provides a deodorizing function if the flavor concentration is higher in the milk than in the blood. Hence, a preventative measure is to not feed odor causing substances for several hours before milking, thus minimizing the concentration of odors. The time interval between eating and milking is an important factor influencing the intensity of feed flavor. This is the principle underlying withholding of feeds for several hours before milking. If this withholding period is too long, however, production suffers. These controlled feeding and/or milking recommendations have resulted from

many studies of feed and weed off-flavor, e.g. Trout et al. (1940), Babcock (1923, 1924, 1925, 1927), Gamble and Kelley (1922), Trout and Taylor (1935). The recommendations and the associated pasture management practices have not always been accepted by the producer (Morgan and Pereira, 1962). Hence, feed and weed off-flavors have remained an important problem.

Some compounds accumulate in the cow's body tissue and subsequently transfer to the milk. In such cases, controlled feeding has little or no effect. The best example of this is the study by Lindquist and Donaldson (1948) where mothball-like flavor persisted for three weeks after the feed was discontinued.

Absorption of odors from the atmosphere after milking is another source of off-flavors. King (1897) demonstrated that corn silage odors entered the milk by absorption from the air after milking but this absorption was not as rapid as that through the respiratory system of the cow before milking. Then Harding et al. (1900) and Marshall (1902) suggested that absorption could play quite an important role in off-flavor development. It was not until the studies of Brueckner (1939), Trout and McMillan (1943) and Babcock (1951) that it was shown that while absorption could and did occur, direct absorption is only a serious problem under abnormal conditions. This is because a relatively small exposed surface area is available for

absorption after milk production.

b. Off-flavors Caused by Metabolic Disorders

When off-flavors due to metabolic disorders are encountered current practice involves identification of the animal or animals concerned and their segregation from the milk herd for the duration of the disease.

c. Miscellaneous Off-flavors

Off-flavors, the sources of which have not been identified, cannot readily be prevented. However, good production practices will reduce the frequency and severity of off-flavors due to bacterial spoilage and/or chemical reaction between production and consumption or further processing.

iii. Flavor Removal Methods

Because it has been shown that off-flavors in milk cannot be completely eliminated before production, efforts have been directed to methods and equipment for deodorization.

Several systems for flavor and odor removal have been used. These are as follows:

a. Aeration

The first deodorization method mentioned in the literature was aeration. Russell (1897) mentioned that

aeration, i.e. "bringing the milk more or less completely in contact with air", would allow an opportunity for the escape of the volatile substances. He further stressed that air free from taint and dust be used. Pasteurization of milk was just becoming popular at the time and Russell noted that heating drove off dissolved gases and that even such persistent odors as garlic could be greatly diminished in this way. The second mention of the beneficial effects of aeration is by Marshall (1902). Ayers and Johnson (1914) showed that garlic taint in milk could be removed by aeration and that increasing the temperature from 145°F to 160°F improved the removal but this improvement was at the expense of the development of a cooked flavor. A similar observation was made by Gray and Eaton in 1917.

Haglund (1917) succeeded in removing garlic taint from cream by letting the cream run over an open cooler at 85 - 90°C three to four times without allowing it to cool. A repetition of the experiment in 1936 by Platon and Olsoon was not as successful.

Babcock (1923, 1924) in his studies on the effects of succulent feed flavors on milk suggests the use of aeration to remove or reduce the off-flavor. In 1928 he recommended that the milk be properly aerated in order to decrease the intensity of feed and barn taints. Similar studies by Gamble and Kelly (1922) on sweet clover silage and Weaver et al.

(1935) on alfalfa hay drew the same conclusion that aeration removed some of the off-flavor.

It is significant that the above investigators recommend controlled feeding practices as well as aeration to minimize or remove odors. Beck et al. (1938) found that aeration did not noticeably improve the flavor of milk from wheat or rye pasture and that the time of removal from the pasture had a greater influence on flavor and odor than any other factor.

MacCurdy and Trout (1940) studied the removal of corn and alfalfa silage flavor from milk. They found that vacuum holder pasteurization and forced aeration holder pasteurization using an air injector was superior to un-aerated or aerated holder pasteurization using stirring only. All these processes were superior to flash pasteurization in removing corn silage flavor from milk.

McDowall (1953a) stated that aeration (usually in an open surface type cooler) is reasonably effective in removal of gaseous and other relatively volatile taints but it is of little use for the removal of relatively high boiling taints. Bergman and Joost (1956) using the "System Hoekstra" aeration found that a triple treatment improved the garlic off-flavor but found it ineffective against fodder flavors.

Dunkley (1965) has concluded that the value of

aerators or surface coolers as a means of reducing feed flavors is often overrated. He cites an experiment at the University of California where alfalfa flavored milk cooled over a surface cooler was compared with the same milk cooled in a container. Experienced judges had difficulty in consistently detecting the sample with supposedly less flavor.

Hence, it is concluded that although aeration may cause some reduction in the flavor intensity of some feeds, it cannot be depended on as an effective method of flavor improvement.

b. Washing

A deodorizing method that can be applied to cream only is the use of water or untainted skim milk for washing. Water, however, tends to give the cream, and subsequently the butter, a "washed" flavor (McDowall, 1955a). Further, at a time when off-flavors in cream are a problem, it is difficult to obtain untainted skim milk in the quantities required for washing. In any case, the additional storage and mixing facilities required are expensive and hence undesirable.

MacDonald and Crawford (1929) in studying the bitter flavor of bitterweed in cream found the causative substance to be more aqueous than fat soluble. They showed that washing was effective in removing this bitter substance

from cream.

Another suggestion has been to use mineral oil or liquid paraffin as an extracting agent. It was found partially effective for removal of onion or garlic flavors by MacDonald and Crawford (1927) and was again tried by McDowall (1955a). This method has not been pursued because it is difficult to remove the oil or paraffin completely. The studies of MacDonald and Crawford (1927), MacDonald and Glaser (1929) and McDowall (1955a) would therefore indicate that removal of water soluble materials is best accomplished by aqueous washing, while fat soluble materials are more easily removed by washing with paraffin or oil.

c. Vacuum Treatment

One method of off-flavor removal has been to subject milk in a vat pasteurizer to a vacuum of about 10 inches of mercury (Hall, 1959). MacCurdy and Trout (1940) studied the effects of vacuum treatment and aeration (by stirring and air sparging) on the removal of corn and alfalfa silage flavor. They found that vacuum treatment gave a significant removal of corn silage flavor and lower concentrations of alfalfa flavor. Further studies using commercial apparatus showed that vacuum treatment alone was effective in reducing off-flavors (Hedrick and Trout, 1959).

There have been many innovations in commercial

vacuum equipment. A number of the more commonly used innovations are discussed by Roahen and Mitten (1956), McDowall (1953b), and Hunziker (1940, Chapter 12). Many of the innovations are described only in patents, and hence the quantitative effects of the various innovations are not well documented. In the period since the mid-fifties there has been relatively little emphasis on innovations and modifications to existing equipment. In 1955, Smith and Mitten reported that vacuum treatment was effective in removing off-flavors. Roahen and Mitten (1956) stated that steam and vacuum were better than vacuum alone; Hedrick and Trout in 1959 came to the same conclusion. Graves et al. (1962) included steam injection as a variable and concluded that it did not have any significant effect except for the removal of garlic flavors.

Cotner et al. (1960), using bench scale laboratory apparatus, showed that vacuum treatment improved the flavor of milk tainted with silage, alfalfa and rye and that steam injection was definitely useful for its removal. The effect of vacuum treatment generally decreases as the vacuum is increased (McDowall, 1953b, p. 334). This appears to be due to the lowering of temperature by evaporation and the consequent lowering of the vapour pressure of the odor compounds which reduces the tendency for the gases to escape.

d. Steam Distillation

Steam distillation is the process of desorption or stripping in which the inert vapour phase is steam. Other inert gases such as nitrogen, carbon dioxide or air, can be used as the inert vapour phase, but the pressures required are greater than the pressure of steam at the same temperature. Steam is the most convenient vapour carrier because it facilitates control of temperature, is readily available in the quantities and purity required and is readily condensed and removed from the equipment. Further, it has been found that direct steam heating eliminates the necessity for and problems of heat exchangers for pasteurization. Dilution of the product occurs, but this problem is minimized by subjecting the milk at the end of the process to a decreased pressure which causes vaporization of the water, a lowering of temperature and also some further removal of odors. An excellent discussion of this problem has been presented by Hallstrom (1966).

Much of the work on the development of equipment using the principle of steam distillation was done in the period 1920 to 1950. The only major development since 1950 appears to be the rotary deodorizer developed by the APV Co. Ltd., England. Other workers in the period since 1950 have tended to concentrate on modifications and minor improve-

ments to the major types of equipment then in widespread use.

Typical of the types of equipment developed are: the Jensen Super-Deodorizer, the Rogers High Temperature Cream Pasteurizer, the Sealtest Process and the Vacreator.

The Jensen Super-Deodorizer consists of a cylindrical type vacuum chamber with the lower half being water jacketed and the upper part containing a receiving pan on which the cream is subjected to a vacuum. The cream from the receiving pan is directed to a steam injector device and the mixture is released onto a distributing cone in the lower vacuum chamber.

In the Rogers High Temperature Cream Pasteurizer, the steam and cream are mixed at high temperature in a pasteurizing tube and the cream is then discharged into the first vacuum chamber (11 - 15 inches of mercury) which is equipped with a special diffusion head for atomization of the cream into a fine mist. The higher vacuum (26 inches of mercury) in the second chamber draws the cream through a dispersion jet that atomizes it a second time.

The Sealtest Process uses a Venturi or other type of mixer to mix the cream and steam. The contact time is controlled by varying the length of the line carrying the steam and cream to the vacuum pan. Entrance of the cream into the vacuum pan (26 inches of mercury) is through an

adjustable distributor that atomizes it into a fine mist. The pan is jacketed and heated to 165°F to prevent condensation of the odor-laden vapours.

The Vacreator Vacuum Pasteurizer was designed to pasteurize, deodorize, and cool in one unit. Ejector condensers create the vacua and provide the means for removing the vapours and gases. The pasteurizing section, maintained at a vacuum of 6 - 11 inches of mercury is designed to disperse cream through the steam in order to heat it to 190 - 200°F and thereby pasteurize the product. The uptake pipe from the pasteurizer into the first section of the deodorizer directs the fluids tangentially around the walls of the intermediate chamber. Separation of the phases occurs and the vapours are expelled while the cream flows down the walls of the chamber. The cream passes through a float valve and an uptake pipe to enter the final vacuum chamber tangentially. The cream separates from the vapour which is produced by the final vacuum of about 28 inches of mercury, and flows down the wall of the chamber. Conditions of operation can be adjusted to have the raw cream entering at a temperature a few degrees higher than that of the cream leaving the machine. This compensates for heat losses from the equipment and ensures that the fat content of the product is equal to that of the feed.

A number of workers have experimented with modifications to the Vacreator and similar pieces of equipment with many of the modifications involving direct injection of steam into the cream and flashing of the somewhat diluted cream into an evacuated vessel. Typical of these modifications are those of Roberts et al. (1940) who injected steam directly into the milk at pressures greater than atmospheric and flashed the liquid into an evacuated vessel; Rogers and Pont (1965) who adapted Roberts' steam injection technique to a preheating system for a Vacreator, with temperatures as high as 280°F; and Major (1966) who developed a special nozzle for contacting steam at 115 psig with cream before flashing into a chamber at 2 psig. Rogers and Pont reported that the off-flavors were less pronounced when their preheating system was employed.

It is evident that the trend has been toward the use of higher pressures and temperatures, and larger pressure drops through the flashing nozzle. Although it has not been stated explicitly in the literature, the principal advantage of the use of higher temperatures is that a greater quantity of vapour is produced from a given quantity of liquid than is produced from the same quantity of liquid at lower temperatures. The ratio of the concentration of the taint compound in the steam to that in the cream is independent of the temperature before

flashing, because the equilibrium between the vapour and liquid is established in the vacuum chamber. The greater quantity of steam produced by flashing from a higher temperature does however effect a greater removal of the taint compound.

The Vacreator has been used as a two stage flash deodorizer, with the partially deodorized liquid from the first stage being further deodorized by flashing into the second unit, which operates at a lower pressure than the first. The advantages of two step over single step flashing have not been clearly stated and systems with more than two stages do not even appear to have been considered.

Several workers (Hunziker, 1940, p. 311; Jordan, 1959) have stated that "for maximum efficiency prompt removal from the cream of the volatilized substances is indispensable (i.e. volatilized substances)" and that "delayed expulsion causes their reabsorption by the cream ...". That this is not so has been implied by Scott (1954a) who discusses the rate of transfer of taint compound from the fat globules to the aqueous phase. He shows that up to 80 - 100 seconds may be required for 90% approach to equilibrium between a fat globule 10 microns in diameter and the surrounding aqueous phase. Scott's calculations are based on a larger than average fat globule and hence the 90% approach time will be less than 80 - 100 seconds, as the rate

of molecular diffusion from a spherical particle is inversely proportional to the square of the diameter. These observations emphasize the necessity of allowing sufficient time of contact between the steam and cream to allow equilibrium to be closely approached.

The use of countercurrent steam and cream flows does not appear to have been seriously considered until the early 1950's. Dummett in 1953 applied for a patent on the use of countercurrent flows in cream deodorization. The patent which was published four years later (Dummett, 1957) described the use of spray chambers for contacting the steam with the milk or cream. He recommended that a countercurrent flow pattern be used within the chambers, but that cocurrent or cross flow patterns could also be used. His main claim is that the cream and steam should flow through the several stages in countercurrent fashion. A countercurrent deodorizer consisting of a number of dished discs rotating on a vertical shaft at 1500 rpm inside a vessel under vacuum has been developed by the APV Co. Ltd., Great Britain. The cream, which is fed to the top of the column, is flung against the wall by each disc. It is then directed from the wall towards the center of the next lower disc. Low pressure steam enters at the bottom of the apparatus and flows countercurrent to the descending stream of cream.

Scott (1954**b**) also considered the use of multiple contact and various flow patterns for deodorization, including "steam stitching" which is in effect a counter-current arrangement. His experiments showed that steam economies were obtained by the use of countercurrent flow systems.

Prior to the early 1950's, all evaluation of the effectiveness of taint removal by various processes was done subjectively, because instrumental or chemical methods were not readily available for precise measurement of the concentrations of the odor-causing compounds in the parts per million ranges in which they occurred. In any case, many of the natural odor-causing compounds had not then been identified.

In order to obtain quantitative data, McDowall in 1953 suggested that naturally occurring taint compounds be added to milk for deodorization studies. His reference substances included diacetyl, acetoin, skatole, indole, benzyl mercaptan, mesityl oxide and some α -diketones. His results have been reported in a series of articles, viz., (McDowall, 1955**a**, **b**, 1956**a**, 1957, 1959**a**, **b**, **c**, **d**, **e**, 1964, 1965). These studies include data obtained using both a Vacreator and a laboratory still.

Scott, working in conjunction with McDowall, studied the characteristics of the Vacreator using McDowall's

vapour-liquid equilibria data for diacetyl and acetoin. Scott's series of papers (1954a, b, 1955, 1956a, b and 1957) provide the first quantitative data on the removal of taint compounds from milk and cream in commercial equipment. Undoubtedly, further quantitative studies have been done, but data from these do not appear to have been reported in the literature. This is very surprising in view of the fact that taints in milk and cream continue to be a serious problem in the industry.

e. Chemical Neutralization of Off-Flavors

Another approach has been to use various chemicals to mask or neutralize the off-flavor in milk and cream. These include such chemicals as alkalies, chlorine compounds, hydrogen peroxide and Listerine (Hunziker, 1940, p. 319, and McDowall, 1953b, p. 718). McDowall (1965) reported that hypochlorites neutralized the taints of landcress in milk. Chlorinated phosphates will also neutralize taints in milk and cream, and in fact, it is known to the author of the present work that these compounds have been used in some parts of Canada, although these practices are of doubtful legality.

The use of chemical additives in milk products is prohibited by law by the majority of dairy producing countries throughout the world with the exceptions including alkalies for neutralizing acids, salt for flavor and coloring agents for appearance. It is doubtful that odor

neutralization compounds will be permitted in the near future in spite of the fact that chemical treatment may prove to be the method of taint reduction.

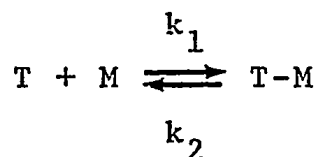
D. MASS TRANSFER IN DISPERSED THREE PHASE SYSTEMS

The basic process in deodorization of milk and cream is to transfer the odor compounds from the aqueous and fat phases to the gas phase. Since the fat globules are dispersed and completely surrounded by the aqueous phase, it is apparent that the most difficult molecules of odor compounds to remove are those in the fat phase.

Scott (1954a) has suggested that odor compounds can be transferred directly from the dispersed phase to the gas phase, by-passing the aqueous phase. There is some evidence that direct transfer may occur in other dispersed three phase systems (Bartholomew et al., 1950; Hixson and Gaden, 1950), but these are systems in which a significant quantity of the dispersed phase is at the interface between the continuous liquid and gas phases (e.g. mineral flotation systems and fermentations involving extensive mycelial networks). Studies on the growth of individual microbial cells with liquid media (e.g. Walden, 1970) indicate that, in general, direct mass transfer between the dispersed and the gas phases is not important. Hence, it is assumed for the purposes of this

study that direct fat to vapour phase mass transfer is negligible compared with transfer via the aqueous phase, because milk and cream corresponds more closely with the microbial systems than with the mineral flotation or mycelial fermentation systems.

It is not unlikely that the taint compounds interact chemically with the milk components, and equilibria between free and bound taint compounds are established, as follows:



where T, M and T-M represent the taint compound, the milk component and the taint-milk component complex, respectively, and k_1 and k_2 are the rate constants for the forward and reverse reactions. If the value of k_2 is small, then the rate of release of taint (T) from the complex (T-M) may be a controlling factor in the rate of removal of taint from the milk. However, no clear evidence has been presented that this is a controlling factor in the rate of deodorization.

i. Multiple Resistances to Mass Transfer

Once the taint compound has been released from any complexes which may occur (either in fat or aqueous phases, or at interfaces), it meets several mass transfer

resistances before it is completely removed from the milk. Taint initially in the fat phase meets resistances in transfer to the aqueous phase. Transfer of taint from the aqueous phase meets further resistance before it is removed completely from the liquid phases. These resistances are as follows:

1. the liquid film resistance in the fat globule;
2. the liquid-liquid interfacial resistance;
3. the liquid-film resistance around the fat globule;
4. the liquid path resistance due to poor bulk mixing;
5. the liquid film resistance between the bulk of the continuous phase and the vapour-liquid interface;
6. the liquid-gas interfacial resistance;
7. the gas film resistance.

ii. Relative Importance of Mass Transfer Resistances

The relative importance of these resistances is difficult to evaluate. However, it is known that in gas-liquid contacting systems, without chemical reaction, the gas phase resistance to mass transfer of slightly soluble gases is very much less than the liquid phase resistance (Sideman, 1966).

Surface active agents, which have the property of migrating to and collecting at the interfaces are also known to affect the rate of mass transfer, e.g. by adding

an additional interfacial resistance (resistances 2 and 6 above) (e.g. Hawke and Alexander, 1960) and/or by dampening the interfacial turbulence (resistances 1, 3 and 5) (Lewis, 1954; Davies, 1963). In contrast to Lewis' study, Blokker (1957) found that surface active agents gave a negligible contribution to the total transfer resistance even when present in strong films.

Davies (1963) found that protein concentrations as low as 0.0005% reduced the circulation in droplets of oil up to 5 mm in diameter. The milk fat globules are much smaller than 5 mm and the protein concentrations are greater than 0.0005%, and therefore it is extremely unlikely that there is any circulation inside the fat globule. This observation implies that mass transfer out of the fat globule can only be by molecular diffusion. Since mass transfer by molecular diffusion is slower than by convection, the resistance to transfer of odor compounds to the surface of the fat globule (resistance 1 above) must be at least as significant as the resistance in the liquid film on the outside of the fat globule, i.e. resistance 3.

The nature of the interface between the fat globule and the aqueous phase has been studied, with most of the studies being done on the compounds from unheated unhomogenized milk. Homogenization decreases the size of the fat globule, and hence increases the total surface

area as well as changing the nature of the protein membrane.

The effects of homogenization were demonstrated by Tobias and Serf (1959). They recovered protein membrane from raw and homogenized milk and found that the amount of protein recovered was inversely proportional to the average fat globule radius, which was as expected from theoretical considerations. They also showed (using electrophoresis) that the protein membrane on the fat globule in the homogenized milk was different from that in the raw milk.

Hawke and Alexander (1960) showed that the nature of the compounds present in the interface can markedly affect the rates of mass transfer through that interface. It is therefore expected that the overall rate of mass transfer through the interfacial membrane will be a function of both the total surface area and the type of protein at the interface.

The effect of type of protein on overall rates of transfer of mass from the fat globule to the aqueous phase in milk do not appear to have been studied. There are indications, however, in the work of Major et al. (1962) that the resistance to mass transfer due to the membrane is important, as they found in experiments on water washing of butter oil, that a taint free product was only obtained when the emulsion was completely broken down.

The effect of size of globule has been studied in model oil-water systems by Rozen and Bezzubova (1967). They showed that the rates do increase but not in proportion to surface area. The relative insensitivity of mass transfer rate to change in drop diameter may be because the diameters of the particles are of the same order of magnitude as the thickness of the interfacial concentration boundary layers.

The thickness of the interfacial concentration boundary layer can only be reduced by increasing the relative velocity between the fat globule and the continuous fluid phase. This relative velocity (and hence the mass transfer coefficient k_L) can be increased by subjecting the emulsion to an increased gravitational field. The effect of the gravitational field g on k_L can be deduced from the equations of Calderbank and Moo-Young (1961) and of Gal-or and Hoelscher (1966).

Calderbank and Moo-Young's equation is

$$k_L = 0.31 \left| \frac{|\rho_c - \rho_d| \mu_c g}{\rho_c^2} \right|^{1/3} \left| \frac{D \rho_c}{\mu_c} \right|^{2/3}$$

where k_L = average mass transfer coefficient for the
 continuous phase Lt^{-1}
 ρ = density ML^{-3}

μ = viscosity	$ML^{-1}t^{-1}$
g = acceleration	Lt^{-2}
D = diffusion coefficient	L^2t^{-1}

Subscripts c and d refer to the continuous and dispersed phases, respectively, and the terms M , L and t refer to the dimensions mass, length and time, respectively.

Gal-or and Hoelscher's equation is

$$k_L = 0.371 \left| \frac{D|\rho_d - \rho_c|g}{2\mu_c + 3\mu_d} \right|^{1/2} \sqrt{a_{32}}$$

where a_{32} is the surface mean radius L

Densities, viscosities and diffusivities are fixed by the material and the operating temperature. If the size of the fat globule is important as is indicated by Gal-or and Hoelscher's equation, then the total mass transfer can be increased by decreasing the average size of the fat globules. The overall mass transfer rate is inversely proportional to the square root of the surface mean radius (a_{32}) and hence only a limited increase in the overall mass transfer rate can be achieved with the changes in globule size that are possible with standard homogenization techniques.

The only remaining variable is the acceleration due to gravity (g). If the material is subjected to mass transfer in a suitably designed centrifuge, the gravita-

tional acceleration term in the equations can be replaced by the centrifugal acceleration which can be several orders of magnitude greater than the acceleration due to gravity.

It should be noted that the diffusion coefficient (D) which is a factor in both Calderbank and Moo-Young's and Gal-or and Hoelscher's equations is decreased by the presence of a dispersed phase. This has been shown by Houghton (1966) who considered a model based on the diffusivity of solute in the continuous phase being composed of contributions from molecular and Brownian motion of the solute and the globules respectively. He shows that compounds very soluble in the dispersed phase must exhibit an effective diffusivity which is less than the diffusivity one would expect if the disperse phase was not present or if the odor compound were preferentially soluble in the continuous phase. The effect of the second liquid phase on the diffusion coefficient (D), and hence on the mass transfer coefficient k_L does not appear to have been considered in the design of steam stripping equipment.

E. DESIGN OF MASS TRANSFER SYSTEMS FOR REMOVAL OF ODORS FROM DISPERSED THREE PHASE SYSTEMS

i. Design Criteria

Scott (1954a) has suggested that the deodorization treatment should be such that it minimizes "the least vola-

tile of the objectionable taints". The degree of "minimization" of the "least volatile of the objectionable taints" depends on the type of equipment and the amount of steam available, and on the initial concentration and properties of the taint compound. Because the final concentration of the taint compound may or may not be below the threshold concentration, the author of the present work suggests that the criterion for design and operation of a deodorizing system should be that the concentrations of all objectionable odors be reduced below their respective threshold concentrations. The threshold concentrations which are acceptable will depend on the use of the deodorized product; for example, the acceptable odor concentration in deodorized cream used as such may be different from that when the cream is used for butter production.

ii. Types of Deodorizing Elements

Various methods of mixing and separating the vapour and liquid are available. Four types of elements, differing in the internal flow patterns of the steam and liquid, are as follows:

a. Totally Mixed Elements

The enriched and depleted phases within the element are completely mixed and equilibrium may or may not be achieved before the two streams are separated. An example is flash equilibrium processes where steam is injected into

and condensed in the liquid and then flashed into a vacuum chamber.

b. Crossflow Elements

The liquid and vapour phases flow at right angles, as in a bubble cap tray, or as in a spray chamber where the steam passes horizontally through a vertical spray of liquid droplets. The concentration of taint in the steam leaving the element may be in equilibrium with the liquid it contacted last, but the average concentrations in the streams leaving the element will not be equal to those in the streams leaving a totally mixed or cocurrent element in which equilibrium is achieved.

c. Cocurrent Flow Elements

The liquid and vapour phases move through the deodorizing element in the same direction, and an equilibrium distribution of odor compound between the outgoing streams will be obtained if sufficient contact time is provided. Cocurrent flow elements have been used in some of the earlier Vacreactors (Scott, 1956a).

d. Countercurrent Flow Elements

The two streams pass through the element in opposite directions, and a concentration gradient is established between the two streams. The ratio of concentration

of odor compound in the outgoing vapour to that in the outgoing liquid may be much greater than the corresponding ratio for an equilibrium mixed or cocurrent element, and is limited only by the area available for mass transfer, the mass transfer resistances and by diffusion and back mixing in the two streams within the element.

The newer Vacreators and the APV cream treatment units use countercurrent flow patterns within the deodorizing elements, but it is likely that back mixing and recirculation has an important effect, especially in the Vacreator, and the element may in effect be nothing more than a totally mixed contacting element.

iii. Arrangements of Deodorizing Elements

There are several ways of combining deodorizing elements in order to effect a greater removal of odor compound than is possible in a single element. These are as follows:

a. Recirculation with Stripping of the Recycled Stream

The milk or cream leaving the deodorizing element can be separated into fat rich and fat depleted streams, and part of one of these streams can be treated in another deodorizing element and then mixed with the incoming untreated liquid stream.

b. Stepwise Flash Systems

Steam is injected into the liquid under high pressure, and the liquid is then passed through several chambers with successively decreasing pressures. Some vapour and odor compound is removed in each stage, and the pressure in the final chamber is such that no dilution of the milk or cream with the condensed steam has taken place.

c. Crossflow Systems

The liquid is passed through a series of contact elements and is contacted with steam in each element. This requires that the steam flow to the system is split to feed the several elements, and the overall effect of the arrangement is such that the liquid flows across the steam flow.

d. Countercurrent Systems

The liquid and the vapour streams flow in opposite directions through a series of contact elements, with some transfer of odor compound from the liquid to the vapour occurring in each stage.

iv. Design Constraints

The properties of milk and cream impose a number of limitations on the type of deodorizing element that can be used, and on the way in which these elements can

be interconnected in any taint removal system. The factors which impose constraints on design and operation of deodorizers for milk and cream are as follows:

a. Potential for Bacterial Spoilage

Milk and cream are extremely good substrates for the growth and multiplication of a wide range of micro-organisms, both pathogenic and non-pathogenic. If Stagnant zones are not avoided in all parts of the equipment where temperatures may permit or encourage bacterial growth, a build-up of micro-organisms will occur during the processing. Further, the construction and finish of all equipment must be such that it can be completely cleaned and sanitized. Design limitations imposed by the potential for bacterial spoilage are summarized in various codes for construction and practice (e.g. 3-A Accepted Practices (1958)).

b. Heat Sensitivity

The application of heating in excess of that required for pasteurization or sterilization and inactivation of enzymes can result in deleterious effects. The nutritive value of the milk may be reduced by destruction of some of the essential amino acids and vitamins, and caramelization may occur and "cooked" flavors may be developed. Hence the industry has endeavored to keep residence times as short as possible wherever

elevated temperatures are used.

Milk and cream proteins are easily denatured by heat and hence hot zones or hot spots where burn on can occur must be avoided.

c. Tendency to Foam

Many of the proteins and phospholipids in milk are extremely surface active, and hence the possibility of foam formation must be considered in the design of deodorizing equipment. This tendency to foam imposes severe constraints on systems where steam is passed through the liquid phase as small bubbles, and can also be a limitation in systems where a jet of liquid impinges on a solid surface.

The potential for bacterial spoilage, the heat sensitivity and/or the foaming tendency of milk and cream make the use of a wide range of vapour-liquid contacting systems impossible. Packed towers are precluded because of cleaning and sanitizing problems, and bubble cap and sieve plate columns cannot be used because of the tendency to form foams. Cleaning and sanitizing are additional problems in bubble cap and sieve plate columns, and the residence times may introduce problems of overheating when these columns are used at higher pressures and temperatures. These limitations do not preclude the use of wetted wall columns as has been shown by Scott (1956a). The reason why wetted wall columns have not found wide applicability

is probably that the liquid film resistance is too high to permit efficient removal of the taint in columns of reasonable size.

These design constraints do not, however, apply to systems using sprays of liquid, and therefore sprays have been far more widely studied and used than any other system. Systems in which the liquid is sprayed directly into a stream of steam, and in which the steam is condensed by injecting it into the liquid flow upstream from the spray nozzle have been used.

v. Design Data

A great deal of work has been done on the identification of the compounds or combinations of compounds responsible for various taints, but in spite of this there is still much uncertainty as to the identity of many of the compounds involved. Once the compounds have been identified, certain properties of these compounds in milk and cream must be determined to enable rational design of deodorizing equipment to be done. Among the data required are the following:

a. Initial and Threshold Concentrations of Taint Compounds

For the most efficient use of steam the concentration of a taint compound should not be reduced much below its threshold concentration, which may depend on the end

use of the product. It is also necessary to know the initial concentration of the compound in the tainted product.

For the present work, the most satisfactory definition of threshold concentration is that of Moncrieff (1967, p. 143), who states that threshold concentration is "the minimum concentration of a particular substance which just arouses sensation." Threshold concentration data for a range of volatile substances in milk and/or cream are reported by Langler and Day, 1964; Forss et al., 1967; and by Siek et al., 1969.

It appears, however, that no studies have been done to determine the likely or possible concentrations of taint compounds in naturally tainted material. Possible likely initial concentrations can be guessed at for the compounds used as reference substances by McDowall (1955a, 1956b) and by Scott (1954a, b; 1956a, b), if it is assumed that the input concentrations that they used were typical of naturally tainted material. Difficulties in measurement of the very low concentrations of their reference compounds (about 10 ppm for diacetyl and acetoin) may, however, have made it necessary for them to use initial concentrations in their tests which were greater than those in the naturally tainted materials, and hence the concentrations of taints which they used can only be regarded as upper limits.

Taint threshold concentration data for selected

compounds are included in Table 1.

b. Vapour Liquid Equilibria Data

Vapour liquid equilibria data are required for design of taint removal equipment, regardless of whether equilibrium is achieved in a particular stage or not. The most important data are those of McDowall (1955a, 1956b, 1957, 1959a, 1959b, 1964, 1965) and Walker (1965) who have determined vapour liquid equilibria data for a number of compounds in water, lactose solutions, skim milk and cream at pressures close to 1 atmosphere. They showed that the concentration of the taint compound in the vapour is a linear function of the concentration in the liquid for low concentrations in the liquid (i.e. less than 20 ppm). Some compounds (e.g. diacetyl, benzyl mercaptan) gave non linear vapour liquid equilibrium relationships at higher concentrations. There does not appear to be any data available for the sub atmospheric pressures encountered in the Vacreator and other types of deodorizing equipment.

In the linear range the vapour liquid equilibrium relationships can be expressed in the form

$$y = kp \quad (II.1)$$

where y is the concentration of taint compound in the vapour, p is the concentration in the continuous phase (i.e. aqueous phase in milk and cream) and k is the vapour liquid equi-

librium coefficient. Values of k for selected compounds are shown in Table 1.

c. Fat Phase Aqueous Phase Distribution Data

Most odor compounds tend to be much more soluble in fats and oils than in water and aqueous solutions (Moncrieff, 1967, p. 601), (MacDonald and Crawford, 1927), (McDowall, 1955a), whereas substances which tend to be more soluble in the aqueous phase than in the fat phase tend to be more readily detected by their taste than by their smell (McDonald and Glaser, 1929), (Trout and McMillan, 1943) and (Dunkley, 1968).

The equilibrium relationships for distribution of taint compound between the fat and the aqueous phases can be expressed in the form

$$f = Dp \quad (\text{II.2})$$

where f and p are the concentrations of the taint compound in the fat phase and the aqueous (or plasma) phase, respectively, and D is the fat phase aqueous phase distribution coefficient. Values of D for several tainting substances have been determined and reported by McDowall (1959c), and Walker (1965). These appear to be the only fat phase aqueous phase distribution data available for tainting substances in milk and cream. Values of D for selected compounds are included in Table 1.

Table 1

THRESHOLD CONCENTRATION AND EQUILIBRIA DATA FOR SELECTED COMPOUNDS

Compound	Odor Thresholds		Equilibria Data ¹			
	In solution µg/gm	In air µg/L	(vapour/liquid) water	(vapour/liquid) skim milk	(vapour/liquid) 30% cream	(liquid/liquid) fat/aqueous phase
acetoin			1.29	1.29		0.21 ^a at 100°F 0.19 at 180°F
benzyl mercaptan	0.001 ²		105	73 ³	1.30	82 ^b at 109°F 101 at 136°F 126 at 165°F 160 at 212°F
diacetyl	0.05 ⁴		39.3	28.4	26-28	0.47 ^a at 100°F 0.74 at 180°F 1.33 at 212°F
indole			2.4		0.31	
skatole		0.33 ⁵	4.7		0.14	

¹ McDowall (1959b, c, 1964, 1965)

² Forss (1951)

³ Walker (1965)

⁴ Jenness and Patton (1959, p. 372)

⁵ Viessmann (1965)

^a For acetoin and diacetyl, McDowall used butter fat and 4.5% lactose solution.

^b For benzyl mercaptan, Walker used butter and water.

Some workers (e.g. McDowall, 1957, and Scott, 1954a) have expressed the vapour liquid equilibrium data in terms of the overall concentration in the liquid, i.e.

$$y = k'x \quad (\text{II.3})$$

where x is the concentration of taint compound in the milk or cream, and k' is the vapour total liquid equilibrium coefficient. The vapour total liquid equilibrium coefficient is a function of the vapour aqueous phase equilibrium coefficient k , the fat content (F/L) and the fat phase aqueous phase distribution coefficient, as is shown below.

The taint mass balance equation for mass L of milk or cream with masses P and F of aqueous phase and fat phase, respectively, is

$$Lx = Pp + Ff \quad (\text{II.4})$$

Elimination of f from equations (II.2) and (II.4) gives the relationship

$$x = (1 + (F/L)(D - 1))p \quad (\text{II.5})$$

The ratio of the vapour total liquid equilibrium coefficient (k') to the vapour aqueous phase equilibrium coefficient (k) i.e. k'/k , is equal to $(y/x)/(y/p)$ (from equations (II.1) and (II.3)). That is

$$\frac{k'}{k} = \frac{p}{x}$$

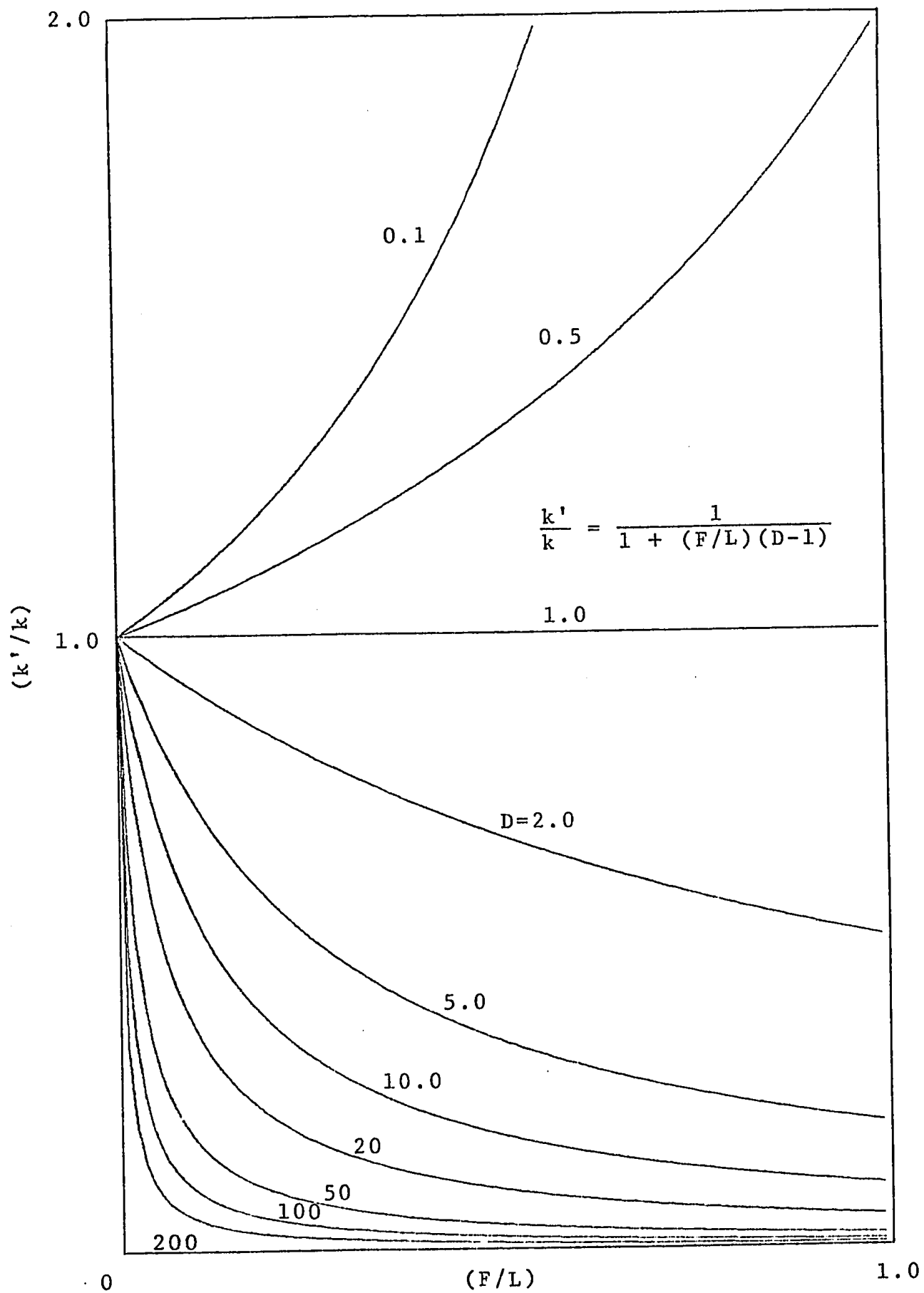
$$= \frac{1}{1 + (F/L)(D - 1)}$$

The ratio k'/k is plotted in Figure 1 as a function of fat content (F/L) for various values of the fat phase aqueous phase distribution coefficient (D) . This shows clearly the very marked effect of fat content on the vapour total liquid equilibrium coefficient in milk and cream $(F/L < 0.30)$ for odor compounds which are fairly fat soluble (say, $D > 20$), and emphasizes the desirability of expressing the vapour concentration in terms of the vapour aqueous phase equilibrium coefficient (k) and the taint concentration in the aqueous phase (p) . This is especially important in systems such as the Vacreator and other devices where concentration and/or dilution of the milk or cream takes place as it passes through the equipment.

d. Mass Transfer Resistance Data

If equilibrium between the vapour and liquid streams leaving any stage is not closely approached then data on transfer of mass from one phase to another are required. Very little attention has been given to mass transfer rates in milk and cream systems in terms of mass transfer resistances and mass transfer coefficients.

Scott (1954a) has indicated that mass transfer



from the interior to the surface of a fat globule may be an important factor in determining the overall rate of approach to equilibrium, when he states that a "90% reduction of taint would take place in 80 - 100 sec. provided that the globule surface was kept at zero concentration." This figure assumes that the diffusivity of the taint compound in the fat is 1×10^{-5} ft²/hr, and that the fat globule size is 10 μ in diameter.

The transfer of taint through the boundary layer around small particles has been studied in a number of real and model systems, including dissolution of various solids in various aqueous and non aqueous solvents, transfer of a solute between the two phases in kerosene-water emulsions (summarized by Calderbank and Moo-Young, 1961), transfer of oxygen and other dissolved gases to and from red blood cells (e.g. Hershey et al., 1967), and transfer of oxygen and other dissolved substrates to micro-organisms. Mass transfer data from these systems will probably be applicable to mass transfer between the liquid phases in milk and cream in deodorizing systems which use similar methods of contacting the liquid and vapour phases. It is unfortunate that most of the data listed above are for falling films or stirred vessels, and hence the applicability to milk and cream deodorization in sprays will be somewhat limited. There do not appear to be any data on mass transfer

between the liquid phases in sprays of emulsions in a vapour phase.

Transfer of volatile materials and dissolved gas from a continuous phase to a gas phase is very well documented in the chemical engineering literature, for sparged vessels, falling films and for sprays. The only data for milk and cream are those of Scott (1956a), who reported stripping characteristics of cream in spray columns (large drops formed by liquid falling under gravity through 1/8 in. diameter holes) and in a wetted wall column. He presented his data as heights of transfer units based on the individual liquid and gas films ($(HTU)_l$ and $(HTU)_g$, respectively), with overall height of a transfer unit (based on the liquid film resistance, i.e. $(HTU)_L$) being given by

$$(HTU)_L = (HTU)_l + (L/mV)(HTU)_g$$

where L and V are the liquid and vapour flow rates.

The term m is the vapour total liquid equilibrium coefficient, i.e. k' of the present study.

III. THE PRESENT STUDY

In this investigation, two approaches have been made to the problem of increasing the economy of use of steam in the deodorization of milk and cream. The first is a study of some factors affecting the rate of mass transfer in a single stage deodorizer, and the second is an analysis of the steam economies of a number of systems involving several interconnected deodorizing stages.

The study of mass transfer rates was made because it appears that there is a significant resistance to the transfer of mass from the fat globule to the continuous phase. The factor which was thought to be the most likely to decrease this resistance was the gravitational field, and therefore continuous bowl type centrifuge was set up as a deodorizer, and a series of experiments was done using water, skim milk, whole milk and cream.

A reference substance was used in all tests and measurements of initial and final concentration of the taint compound were made for all runs. Diacetyl was chosen as the reference compound because it is one of the few taint compounds for which both vapour liquid equilibrium coefficients and fat phase aqueous phase distribution coefficients were available. Vapour liquid equilibrium data were available for atmospheric pressure only, and it was therefore necessary to perform experiments (using

a modified Hala still) to determine the vapour liquid equilibrium coefficients at sub atmospheric pressures.

An analysis of steam economies of systems involving recycling of a fat enriched stream was made because of the statement by McDowall (1958) that "the most efficient removal of taint is obtained with a high fat content cream." Included in this analysis was consideration of a system in which a fat depleted (i.e. skim milk) stream was deodorized and then used to "wash" the incoming cream or milk.

The analysis of steam economies was then extended to three other systems of interconnected deodorizing stages, i.e. step flash, crossflow and countercurrent systems. In order to simplify these analyses it was assumed that deodorizing stages were available (or could be designed) so that equilibrium between the outgoing streams was closely approached for all combinations of taint, steam and milk or cream.

IV. EXPERIMENTAL WORK

A. OUTLINE OF EXPERIMENTS

The first series of experiments involved the determination of vapour liquid equilibria in diacetyl milk and diacetyl cream systems. The operation of the Hala still was checked with the ethanol water system and the values obtained checked against literature values. The diacetyl water system was first studied and the data obtained at atmospheric pressure was compared to the data given by McDowall (1955a). Data for the diacetyl water system at reduced pressures was then obtained.

The above procedures were then repeated for the diacetyl skim milk and the diacetyl cream systems.

Difficulties in maintaining equilibrium conditions between the aqueous and fat phases prevented useful results from being obtained in the tests on the diacetyl cream system.

The colorimetric method for determination of diacetyl concentration used for the vapour liquid equilibria studies was not sensitive enough to be used for the diacetyl concentration determinations at the concentrations proposed for the mass transfer studies and hence a gas chromatograph was set up for these analyses.

The mass transfer studies were carried out using a Sharples laboratory centrifuge which had been modified

to allow introduction of two streams (i.e. liquid and vapour phases) and separate collection of the outlet vapour and deodorized liquid product. The factors which were studied were centrifugal acceleration, liquid and vapour flow rates, initial diacetyl concentration, solute and fat contents of the liquid streams (i.e. water, skim milk, whole milk and cream), and thickness of the fluid layer on the inside of the centrifuge bowl. The effects of injection of the liquid stream into the bowl with and without spraying were also studied.

B. MATERIALS

i. Water, Milk, Cream and Steam

The water used for the tests on diacetyl water systems was the distilled water available in the laboratory.

Normal commercial grades of milk and cream were obtained from a local dairy and used in the tests.

The steam for all tests was produced from the laboratory supply of distilled water.

ii. Diacetyl

Anhydrous sodium sulfate was added to the diacetyl as received from Nutritional Biochemicals Corporation, Cleveland, Ohio, U.S.A. The diacetyl was then vacuum distilled, with the top fraction being discarded. The fraction having a steady boiling point was collected. The

residue had a peculiar odor, different from the characteristic butter smell.

The collected fraction had a yellow-greenish color, characteristic odor, refractive index of $n_D^{18} = 1.3968$ and the boiling point of 88.5°C corrected to 760 mm pressure.

C. METHODS OF ANALYSIS

i. Milk and Cream

The only analyses done on milk and cream were fat tests. The analysis was done by the Roese - Gottlieb or solvent extraction method, (McDowall, 1953b, p. 114, Chapter 38), testing fat in milk and milk products.

ii. Diacetyl

Most of the methods of diacetyl analysis are modifications of the original Tschugaeff reaction as first used by van Niel (1927). They depend on the formation of dimethylglyoxime from diacetyl and hydroxylamine hydrochloride and subsequent development of a pink color with ferrous sulfate.

These methods when applied to milk and cream require a stripping and concentrating step and Barnicoat (1935) proposed that steam distillation be used for this initial step. Prill and Hammer (1938) improved this method by reducing sample size. A method using nitrogen purge through a 10 - 20 ml sample instead of steam has been devised by Owades and Jakovac (1963) for beer and adapted to dairy

products by Pack et al. (1964).

Absorbance methods for diacetyl analysis have been directed to analysis of reaction products rather than diacetyl itself. The absorbance of diacetyl in the ultra violet region of the spectrum is only moderate (International Critical Tables, 1929). Speck (1948) proposed that the purple color produced by diacetyl and chromotropic acid in the presence of concentrated sulfuric acid and Englis et al. (1953) proposed that dimethylglyoxime, the reaction product of diacetyl and hydroxylamine hydrochloride, which has a strong absorption at 226m μ be used as the reaction compound.

Polarographic determinations of diacetyl in aqueous solution have been carried out by Shikata and Tachi (1930) and Adkins and Cox (1938). The application to diacetyl determination in buttermilk has been described by Ferren et al. (1967). The advantage is that a prior extraction is not required, and the analysis is then simple and fast. The method was not successfully applied to milk and cream. The difficulty appeared to be due to either the protein or fat interfering with the dropping electrode. The use of some extraction method prior to the polarographic analysis was not tried since the inherent simplicity of the method was then lost.

Doelle (1967) showed that diacetyl could be detected using a hydrogen flame ionization detector.

Shilman and Schaper (1966) demonstrated that the response of the electron capture detector to diacetyl is about twelve hundred times more than to a hydrogen flame ionization detector. Then Scanlan and Lindsay (1968) using a gas entrainment, on column, trapping procedure for flavor volatiles designed by Morgan and Day (1965) and a pulsed electron capture detector were able to quantitate diacetyl in the parts per billion range in aqueous solution.

In this study, all diacetyl solutions were standardized using the method of Pack et al. (1964). The method of Pack et al. (1964) was also used for the diacetyl determinations in the vapour liquid equilibrium studies.

Polarographic methods (Ferren et al. (1967) were used initially in the vapour-liquid equilibrium study but could not be used for fat containing materials. Alternate analysis methods for diacetyl were then devised using gas chromatography and modifications of the trapping procedure of Morgan and Day (1965). A Varian Aerograph Model 200 gas chromatograph with a concentric electron capture detector was tried. The first run was successful but it was found that the water which eluted immediately after the diacetyl inactivated the tritium source for several hours. Scanlan (1968) had similar problems, but apparently these were not as severe with the pulsed electron capture detector, as with the detector used in this study. The electron

capture detector was therefore abandoned in favor of the less sensitive hydrogen flame ionization detector which is essentially insensitive to water.

Morgan and Day's trapping procedure involved disconnecting the packed column in the gas chromatograph, inserting the packed trap loop in front of the column and reconnecting. Use of a six way valve¹ attached to the injection port on the exterior of the instrument (Figure 2) made it possible to simplify the injection procedure considerably. The two possible arrangements are sampling position and injection position.

In the sampling position, the carrier gas passes through the valve and into the column. A dry ice, cellulose mixture is placed under the sample loop so that several inches of the loop are in the cold mixture. Helium purge gas carrying the volatiles enters the sample loop, as shown in Figure 2. The volatiles condense on the packing and the helium gas is allowed to escape into the inverted collecting burette (Figure 3).

When the purge time is completed, movement of the valve stem redirects the flow as shown in the injection position in Figure 2. The helium carrier gas then flows through the loop and into the column. The dry ice bath is quickly removed and replaced with a hot water bath in

¹ Varian Aerograph, Heated Type Gas Sampling Valve - Six Port Catalogue No. 57-000036-00

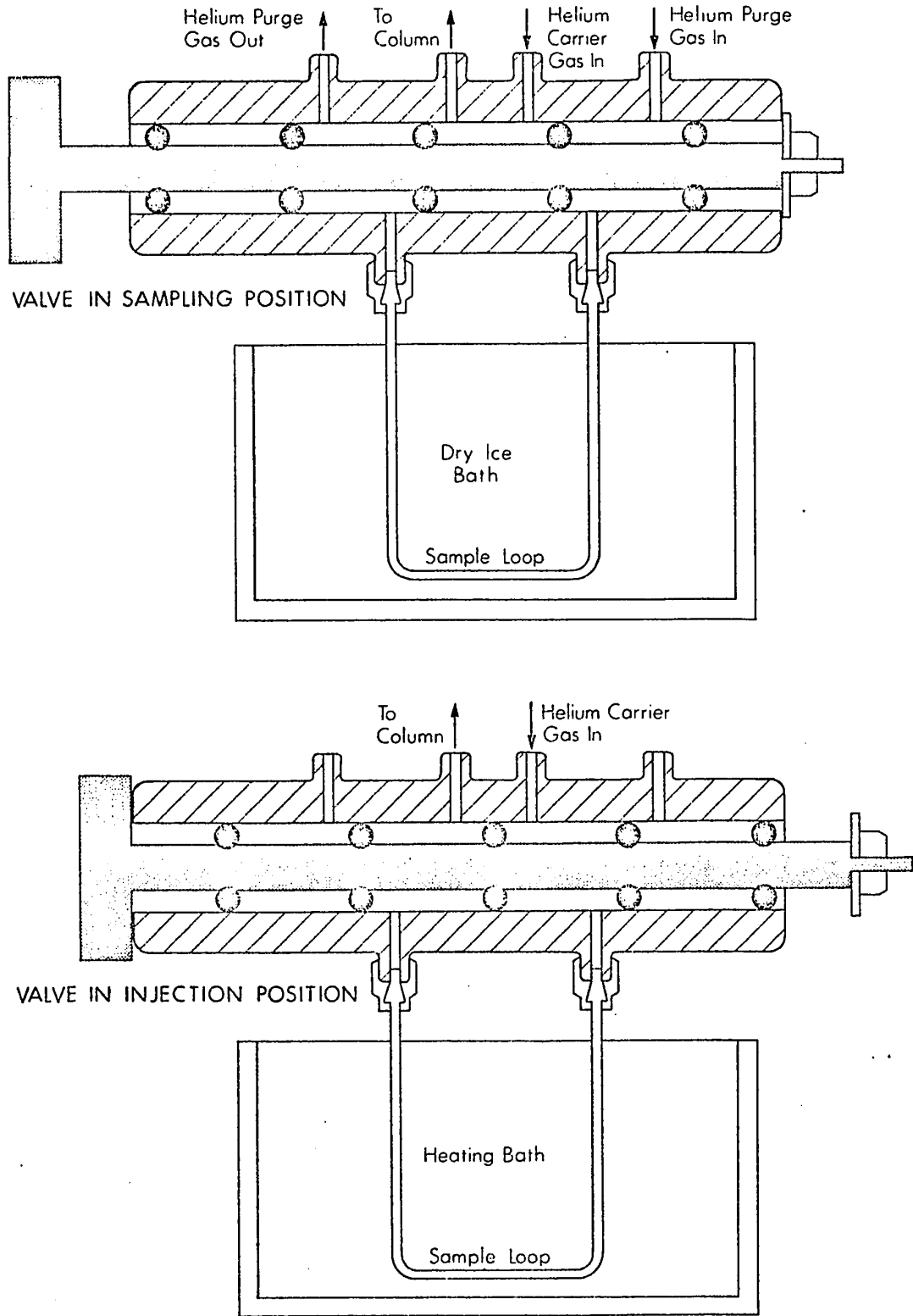


Figure 2

SIX WAY VALVE FOR VOLATILE ENTRAPMENT

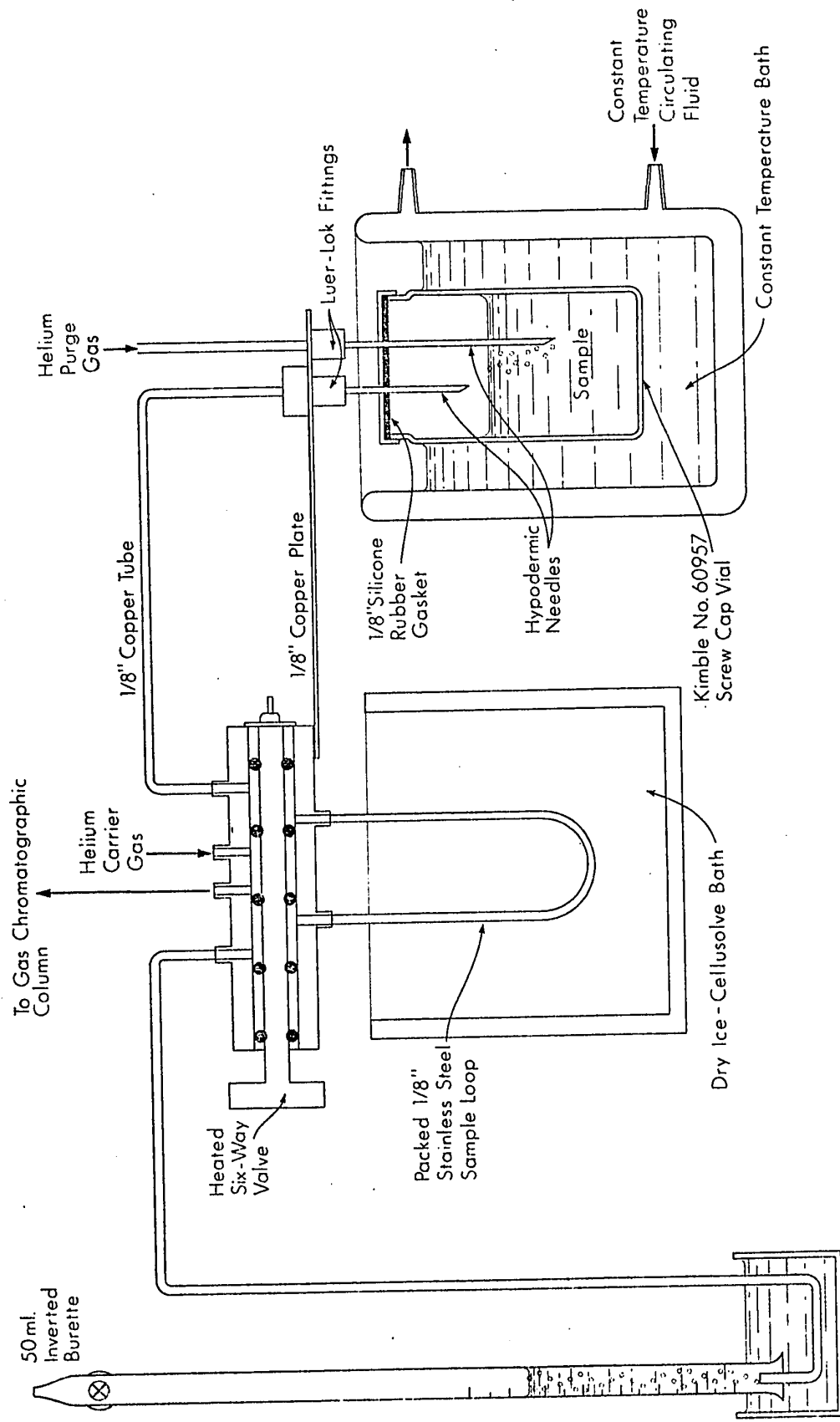


Figure 3

GAS CHROMATOGRAPHIC SAMPLE TRAPPING SYSTEM

order to volatilize the trapped compounds. The change of the valve from sampling position to injection position is very rapid and causes a negligible momentary change in the detector response.

An electric heater imbedded in the sampling valve prevents the gases from condensing in the valve. The non-packed volume in the valve is kept to a minimum by using short connections and capillary holes.

A copper plate was clamped onto the valve (Figure 3). Conveniently close to the end of the plate were soldered two Luer-Lok fittings spaced $7/16$ inch apart. From one fitting a minimum length of $1/8$ inch copper tube was connected to the helium purge gas inlet of the six way valve. The other Luer-Lok fitting was connected to a helium cylinder with $1/8$ inch copper tubing. Heat conducted along the copper plate ensured that gases would not condense in the copper tubing. Gaskets cut from $1/8$ inch silicone rubber with No. 3 and No. 1 cork borers were placed into the Luer-Lok fittings to provide a leak proof seat against which a $1/2$ inch and $1\ 1/2$ inch hypodermic needle would sit.

Screw cap vials (Kimble No. 60957, Size No. 1) were modified by drilling two $1/8$ inch holes, $7/16$ inch apart in the cap and lining the cap with a $1/8$ inch thick silicone gasket. The holes would accept the two hypodermic

needles as shown in Figure 3.

The vial was kept at a constant temperature of 36°F for at least one-half hour before purging and also during the purge by connecting a glass sleeve to a circulating pump in the constant temperature bath. A similar arrangement was used to heat the sample loop to around 70 - 75°C during the injection period.

Morgan and Day (1965) suggested that the aqueous solution be saturated with anhydrous sodium sulfate to decrease the vapour pressure of the water and hence increase the height of the diacetyl peak as shown in Figure 4, and that time be used to determine the volume of purge gas. However, it was found that salt precipitated just inside the submerged needle. Because this restricted and occasionally blocked the gas flow completely, the use of sodium sulfate was discontinued.

To insure the same amount of purge gas for each test, a 50ml burette was inverted into a beaker, as shown in Figure 3, and the purge was stopped when 50ml of gas was collected. The collection of the gas started at the moment the 1/2 inch needle penetrated the silicone gasket in the glass vial and with some practice, the vial could be removed exactly at the 50ml point. The six way valve was then moved into the injection position, and the sample loop heated as described above.

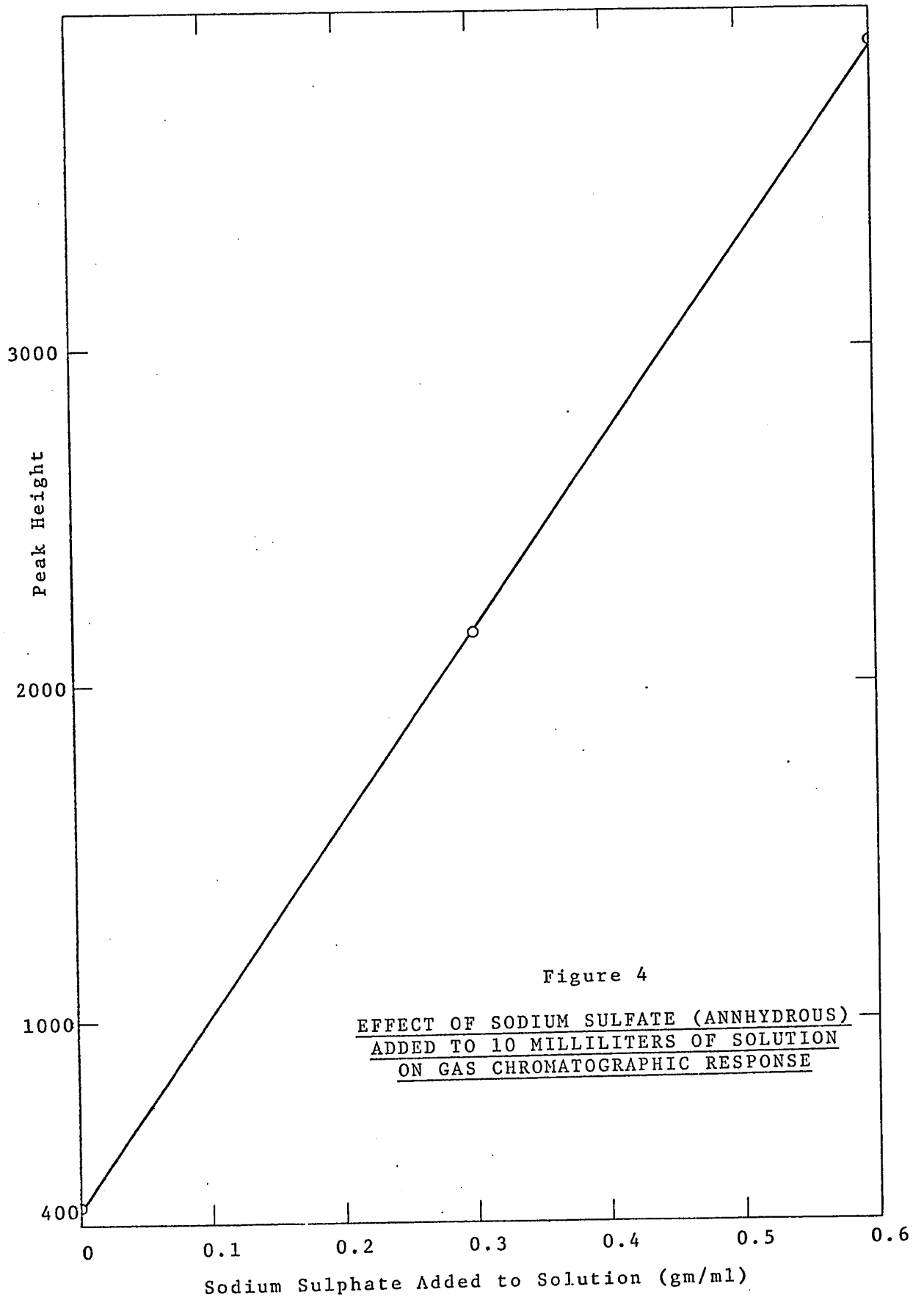


Figure 4

EFFECT OF SODIUM SULFATE (ANNHYDROUS)
ADDED TO 10 MILLILITERS OF SOLUTION
ON GAS CHROMATOGRAPHIC RESPONSE

The typical response recorded is shown in Figure 5. It has been calculated (Appendix B) that 50ml of purge gas transfers approximately 4×10^{-9} g of diacetyl from a 1 ppm solution to the trap. As expected, increasing purge volume increases the diacetyl peak height as is shown in Figure 6.

Normally, the peak height would be proportional to the concentration of diacetyl. This was found to be the case except for very low concentrations. Suppression of the response of the detector to diacetyl was found to be due to some of the diacetyl being adsorbed onto the glass vial containing the sample.

Several milligrams of tetradecanol was added to suppress foaming in the vial during the purge. This addition of antifoam did not influence the response of the detector to diacetyl.

D. VAPOUR-LIQUID EQUILIBRIUM

Because milk and cream are heat labile materials conventional static equilibrium stills using recirculation are not suitable for vapour-liquid equilibrium determination. A very simple instrument using dynamic (i.e. single pass) methods for equilibrium determinations has been designed by Hala et al. (1967). The Hala still design was chosen since it fulfilled many of the requirements:

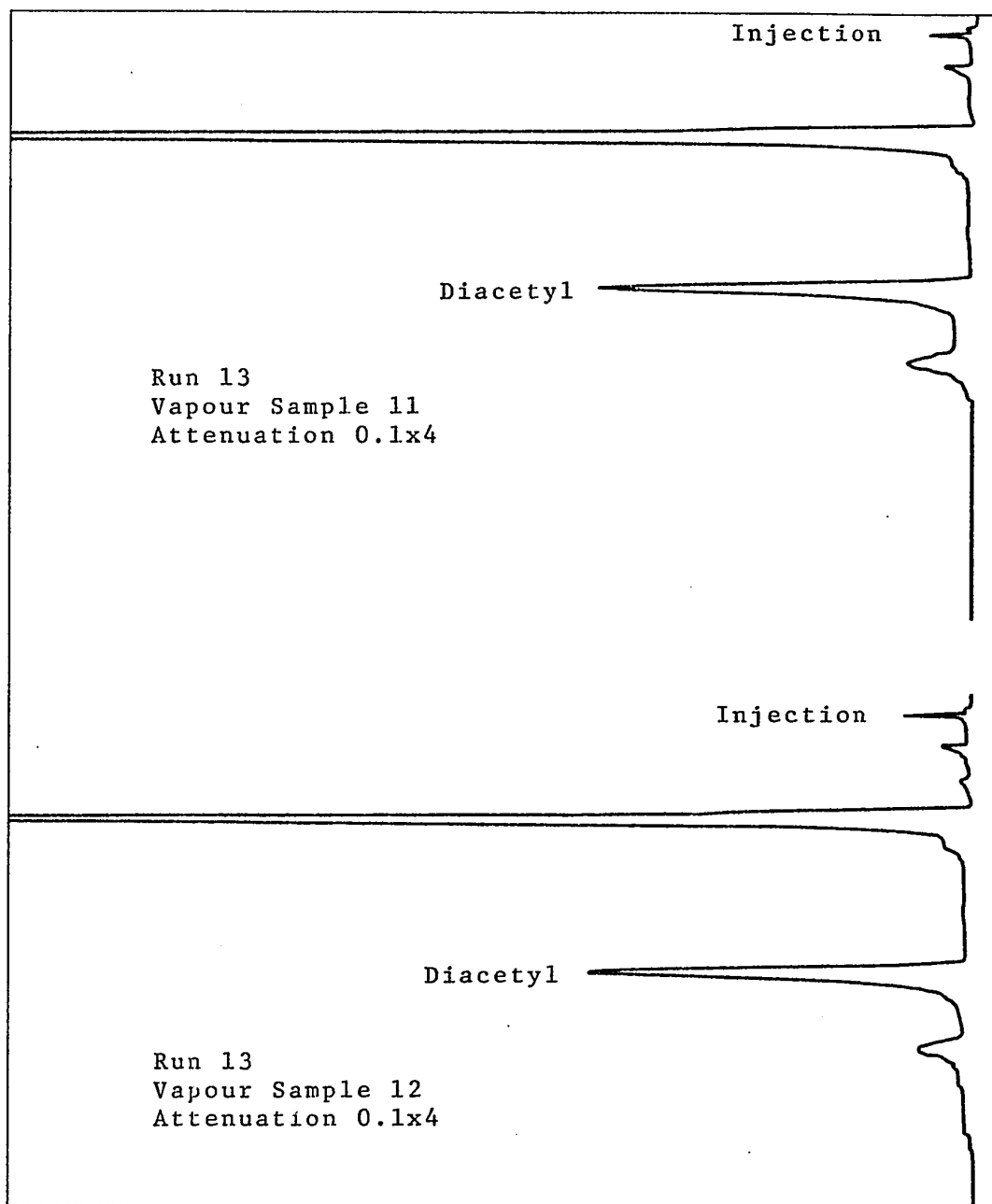


Figure 5

TYPICAL CHROMATOGRAMS FOR DIACETYL

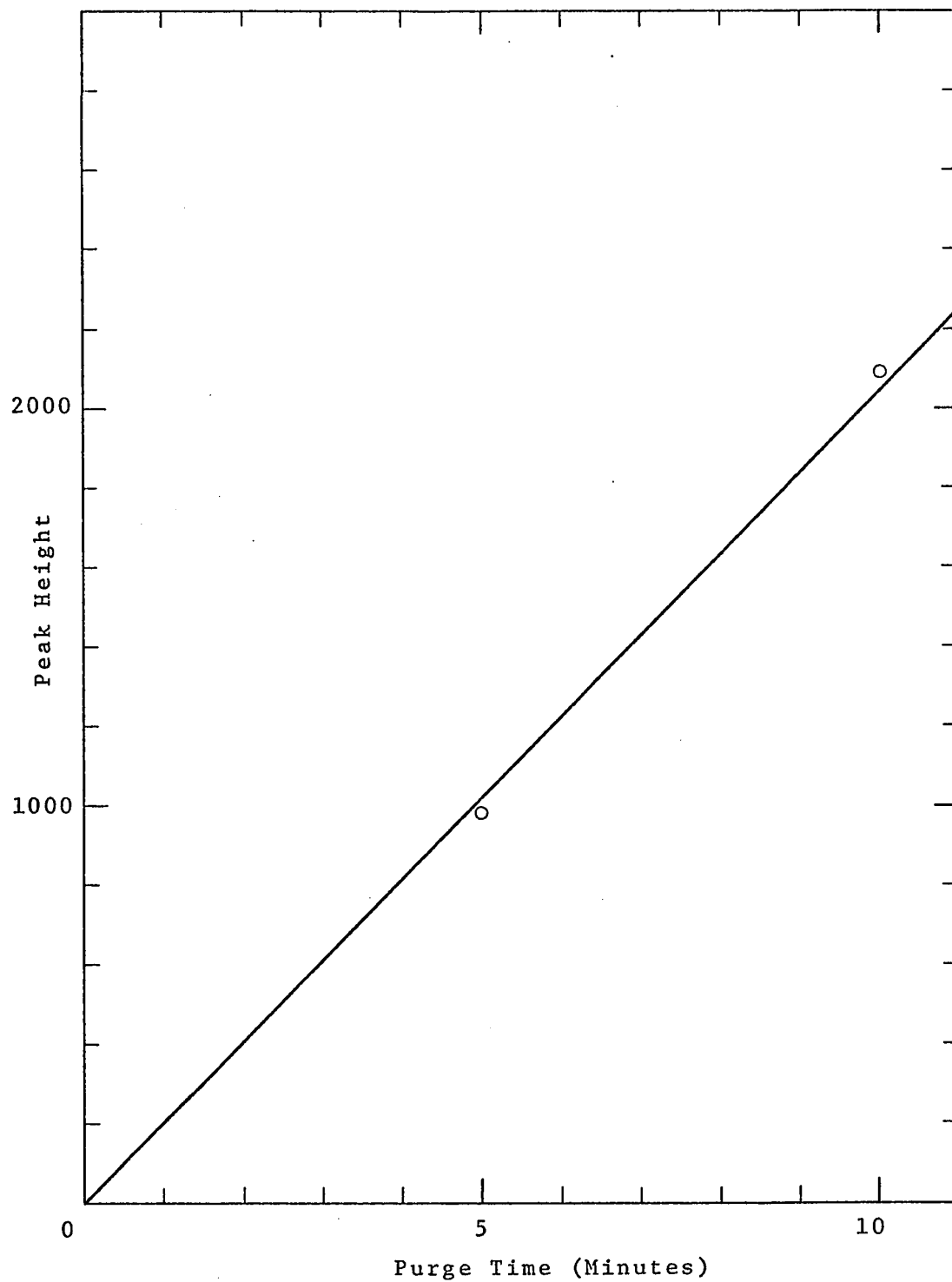


Figure 6

EFFECT OF PURGE TIME (VOLUME)
ON GAS CHROMATOGRAPHIC RESPONSE

1. It has a very short contact time at high temperature to minimize heat effects on the milk or cream.
2. It is dynamic in principle and hence does not require any recirculation system.
3. Pressure can be readily adjusted and controlled, especially at pressures below atmospheric.

Further, it is much simpler to operate than most other equilibrium stills. Detailed plans were obtained from Hala and these were used for the construction of the dynamic still, as shown in Figure 7.

The starting liquid was introduced into the supply vessel. The float mechanism immediately established a constant level in the vapourizing tube where the preheated liquid was brought to boiling by a steam heated jacket. The mixture of vapour and liquid then spurts on the thermometer in the equilibrium chamber and drains to the droplet separator. A ground glass, short stem thermometer was used instead of the thermometer well shown in Hala's work.

The equilibrium vapour is separated from the liquid in the equilibrium chamber and after condensation drains into the collecting flask. The rotating sampler permits a sample to be taken without interfering with or interrupting the established steady operation.

Large diameter vacuum hose was used to interconnect the vapour and liquid sampling receivers. A

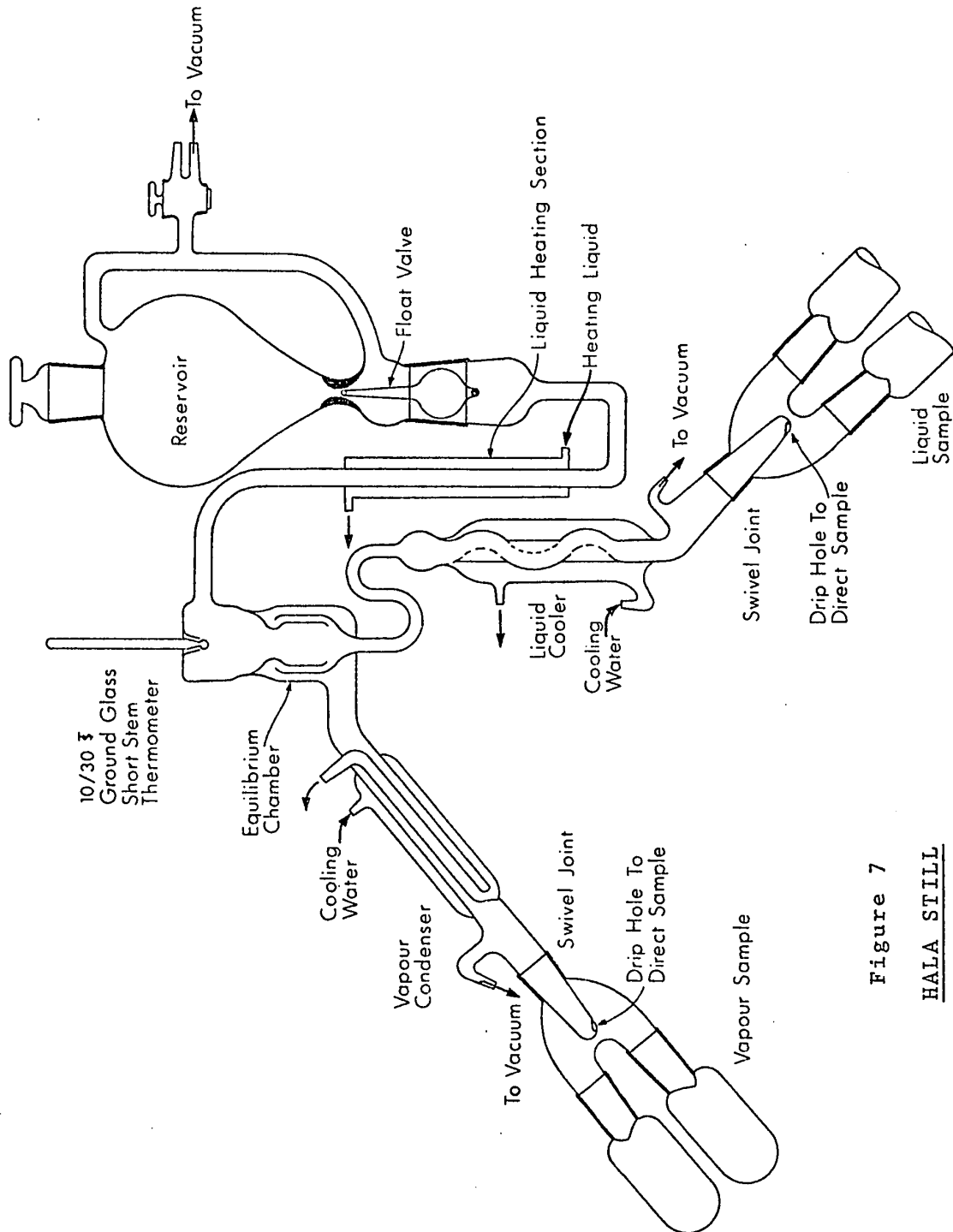


Figure 7

HALA STILL

pressure differential would cause the syphon to empty and thus spoil the test.

Foaming of the sample in the equilibrium chamber was a serious problem. Although an antifoam agent could be added to the milk, it was felt that this could alter the properties of the fluid. Instead various methods of injection into the equilibrium chamber were used as an alternate solution. The first runs were conducted with the vapour-liquid mixture impinging directly onto the thermometer bulb in the equilibrium chamber. The foaming problems were overcome by having the fluid enter the equilibrium chamber tangentially. Cleanliness of the glassware also seemed to affect the operation of the still and after each test the interior was washed with detergent and rinsed out with distilled water. It is of interest to note that McDowall (1955**b**) had similar problems in his equilibrium still. His paper showing alternate disengagement vessels was received after the above trials and it was found that he too had solved foaming problems with a tangential entry to the equilibrium chamber.

The pressure in the still was accurately and easily controlled with a capstan type manostat. A mercury manometer connected to the vacuum line was used to determine the actual pressure in the equilibrium still.

E. LIQUID-LIQUID EQUILIBRIUM

Equilibrium data for a three phase system requires, according to Gibb's phase rule, analysis of the phases and specification of one intensive variable other than composition (e.g. temperature or pressure) in order to completely describe the system. Overall liquid-vapour separation occurs in the Hala still but additionally, liquid-liquid separation is also required. This liquid-liquid separation should occur at the equilibrium conditions of overall liquid-vapour separation, otherwise the equilibrium state is upset.

A separation of the two liquid phases as a second step was readily achieved in a Sorvall Superspeed, Model RC-2B centrifuge (Ivan Sorvall Inc., Norwalk, Conn., U.S.A.). It was hoped that data at atmospheric pressure could be obtained using this additional step. However, satisfactory recovery of the two phases was possible only at temperatures below that of the vapour liquid separation.

As mentioned above, this change in temperature meant that equilibrium conditions were different and the desired data could therefore not be obtained in this manner. It was therefore necessary in the remainder of this study to use published fat phase aqueous phase distribution coefficients (e.g. that of McDowall, 1959c).

F. THE CENTRIFUGAL EQUILIBRIUM CELL AND CENTRIFUGAL DEODORIZER

A need to separate the two liquid phases in the equilibrium chamber and thus not upset the original established equilibrium gives rise to the possibility of using a centrifuge tube as an equilibrium cell. It was first envisaged that a centrifuge permitting the entrance of two separate fluid streams (milk and steam) and the exit of three separated fluid streams (fat phase, aqueous phase and skim) could be constructed. In this way separation of the two fluid streams could be achieved without upsetting the established equilibrium condition. If in addition a probe could be positioned inside the rotor permitting withdrawal of sample of the three phases anywhere along the tube then longitudinal changes of concentration could be used for indication of attainment of equilibrium along the rotor within the centrifuge.

It has not been possible in this study to get complete three phase equilibrium data from a centrifugal equilibrium cell because the available Sharples laboratory centrifuge could not be modified to allow the collection of the three phases separately.

However, it was possible to modify the centrifuge so that it could be used as a deodorizer, with the major change being a modification of the injection nozzle to allow separate injection of the vapour and liquid streams directly

into the rotating bowl, as shown in Figures 8 and 9. The liquid phase flows into the bowl through the inner tube, and steam through the annulus. The straight arrangement of tubes (Figure 9(a)) produced a fine spray, and the curved tube (Figure 9(b)) allowed the liquid to be directed on to the inner wall of the bowl in an unbroken stream. The latter arrangement was used to eliminate any effect of deodorization in the injection spray. It was also necessary to remove a small vaned impeller (normally used to impart angular momentum to the incoming liquid) so that the modified injection ports could be inserted.

Deodorization experiments were done using two thicknesses of fluid layer in the centrifuge. The thicker of the two layers was obtained by using the unmodified centrifuge bowl. The densities of the fat and aqueous phases of milk and cream are such that no fat overflowed the inner dam, and hence both liquid phases flowed through the underflow port and out over the outer dam, as shown in Figure 10. The vapour flowed out through the overflow port.

The thinner liquid layer was obtained by drilling three 1/8 inch diameter holes near the top of the centrifuge bowl as shown in Figure 11. The liquid flowed out through these holes quite readily, and could be collected in the lower collecting bowl as in the thick film experiments. The original underflow outlet was not closed off, and hence steam could exit from the equipment through both the overflow

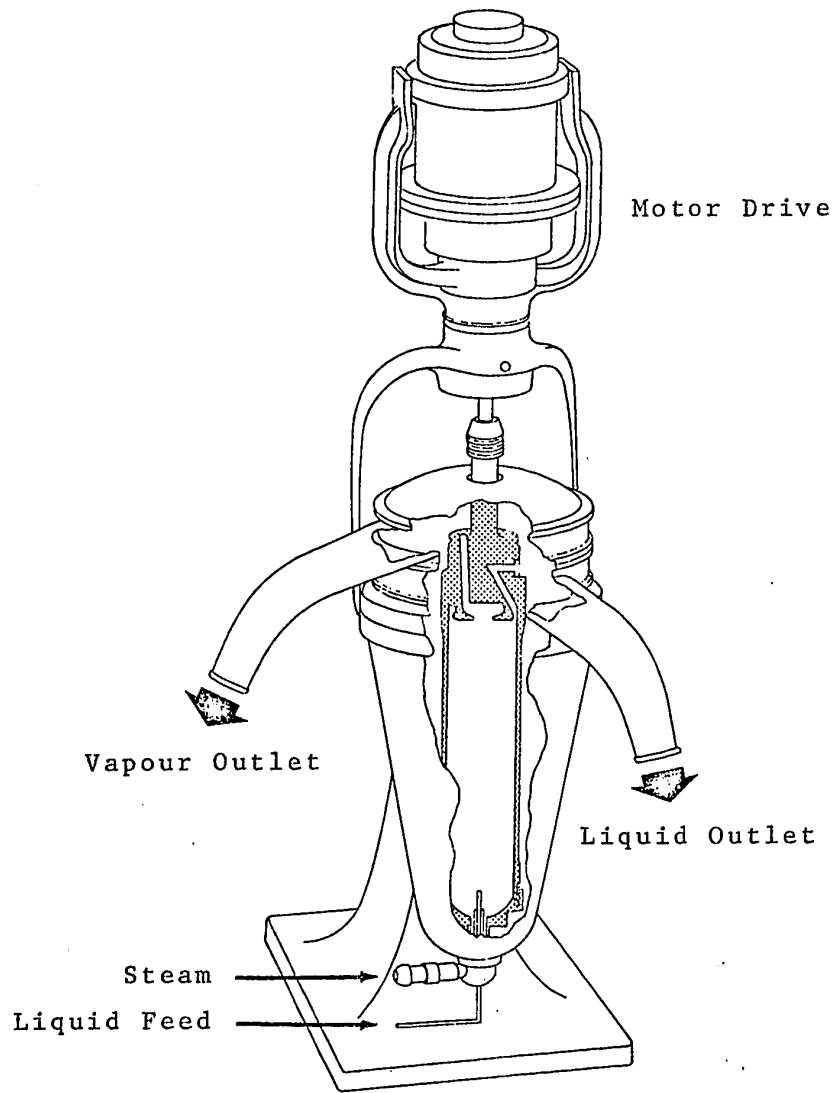
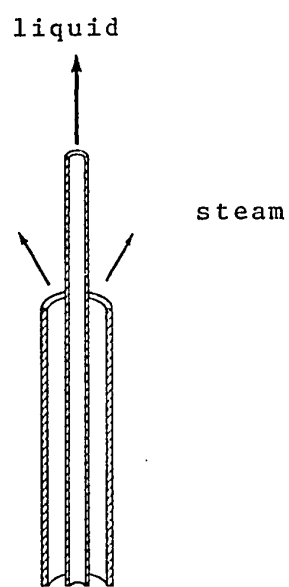
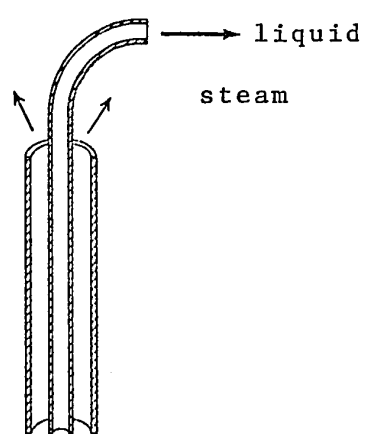


Figure 8

MODIFIED SHARPLES LABORATORY CENTRIFUGE



a



b

Figure 9

DETAILS OF INJECTION PORTS

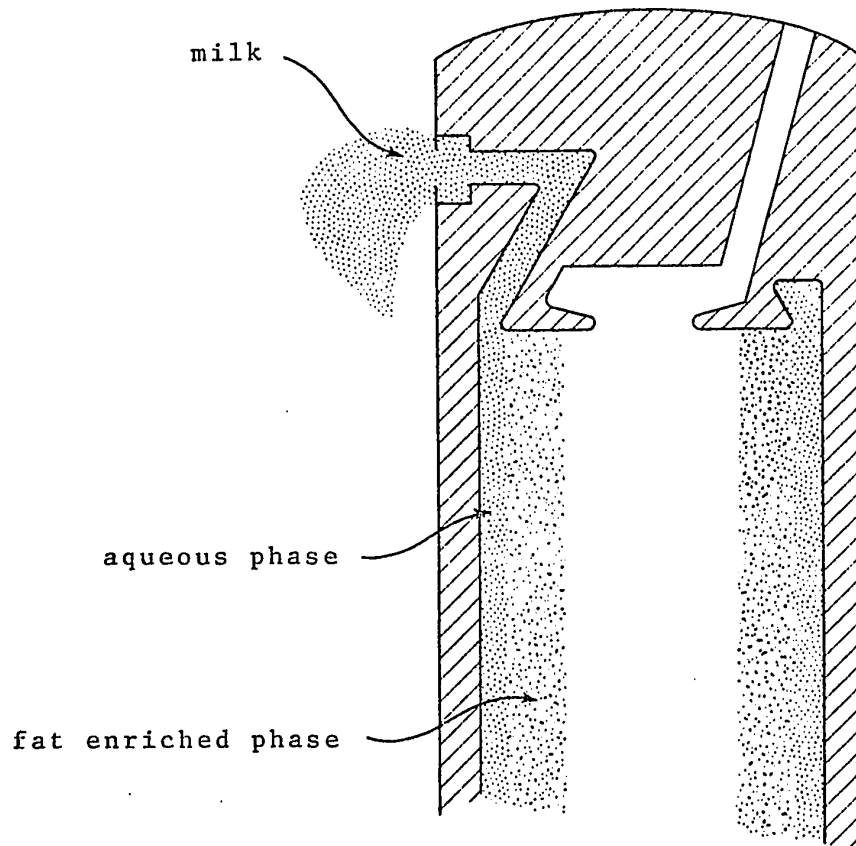


Figure 10

DETAIL OF EXIT PORTS

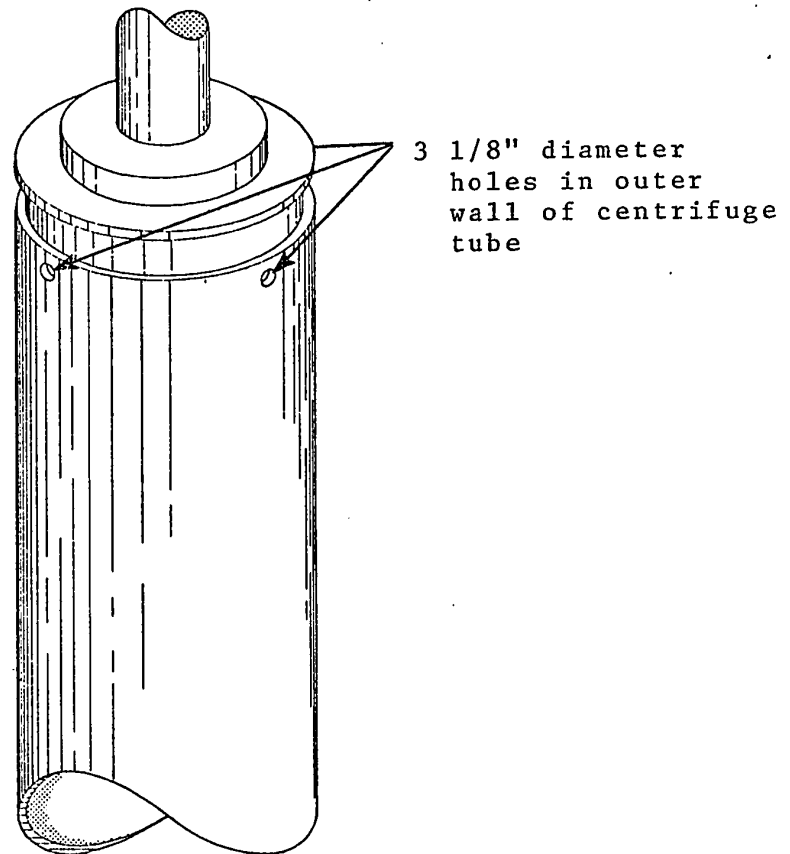


Figure 11

MODIFICATION OF CENTRIFUGE BOWL
FOR THIN FILM EXPERIMENTS

and underflow ports, as well as through the three 1/8 inch holes.

Measurements of the amount of liquid holdup in thick fluid layer experiments ($V_h = 300$ ml) gives a fluid depth on the inside of the bowl of approximately 1.0 cm. It was not possible, however, to measure the holdup in the thin film experiments. If it is assumed that the thickness of the liquid film at the outlet holes is zero, then minimum holdup volumes ($V_{h,min}$) can be calculated as follows:

The equation for the free surface of a liquid in a vertical cylindrical container of radius R and height L rotating about its axis, with an angular velocity ω is given by

$$Z - Z_0 = \omega^2 r^2 / 2g$$

where r is the radius of the free surface at height $(Z - Z_0)$ above the free surface of the liquid at the axis, and g is the acceleration due to gravity. In the experimental centrifuge the angular velocities were such that the point $Z = Z_0$ was below the centrifuge, and the value of Z_0 was obtained by putting $r = R$ at $Z = L$; i.e.

$$Z_0 = L - \omega^2 R^2 / 2g$$

The holdup volume (V_h) is then given by

$$V_{h,min} = \pi R^2 L - \int_0^L \pi r^2 dz$$

where r^2 is a function of Z ; i.e.

$$r^2 = R^2 + (Z - L) 2g/\omega^2$$

On integration this gives

$$V_{h,\min} = \pi g L^2 / \omega^2$$

For speeds of 6000 and 24,000 RPM and height = 9.0 inches, the holdup volumes are 4.10 ml and 0.25 ml, respectively. It should be noted that these are minimum holdup volumes and will only be obtained in the case of zero liquid flow. The liquid cannot spill over the lip of the outlet holes with zero thickness (i.e. with finite velocity) and hence the actual thickness of the film will be increased by the depth of liquid at the lip. There will also be entrance effects and effects of fluid friction as the liquid flows up the wall which will tend to make the actual holdup volumes somewhat greater than the minimum (or zero flow) holdup volumes.

Residence times based on the measured or calculated holdup volumes have been calculated for the two liquid flow rates used in the experiments. These are shown in Table 2.

The liquid and vapour feed systems and the sample collecting systems for outlet streams are shown in Figure 12.

Table 2

MINIMUM RESIDENCE TIMES

Experiment	Residence Times (Seconds)	
	Liquid Flow rate = 68 ml/min	Liquid Flow rate = 460 ml/min
Thick film, both speeds	258	38
Thin film, 6000 RPM	4.8	0.7
Thin film, 24,000 RPM	0.3	0.05

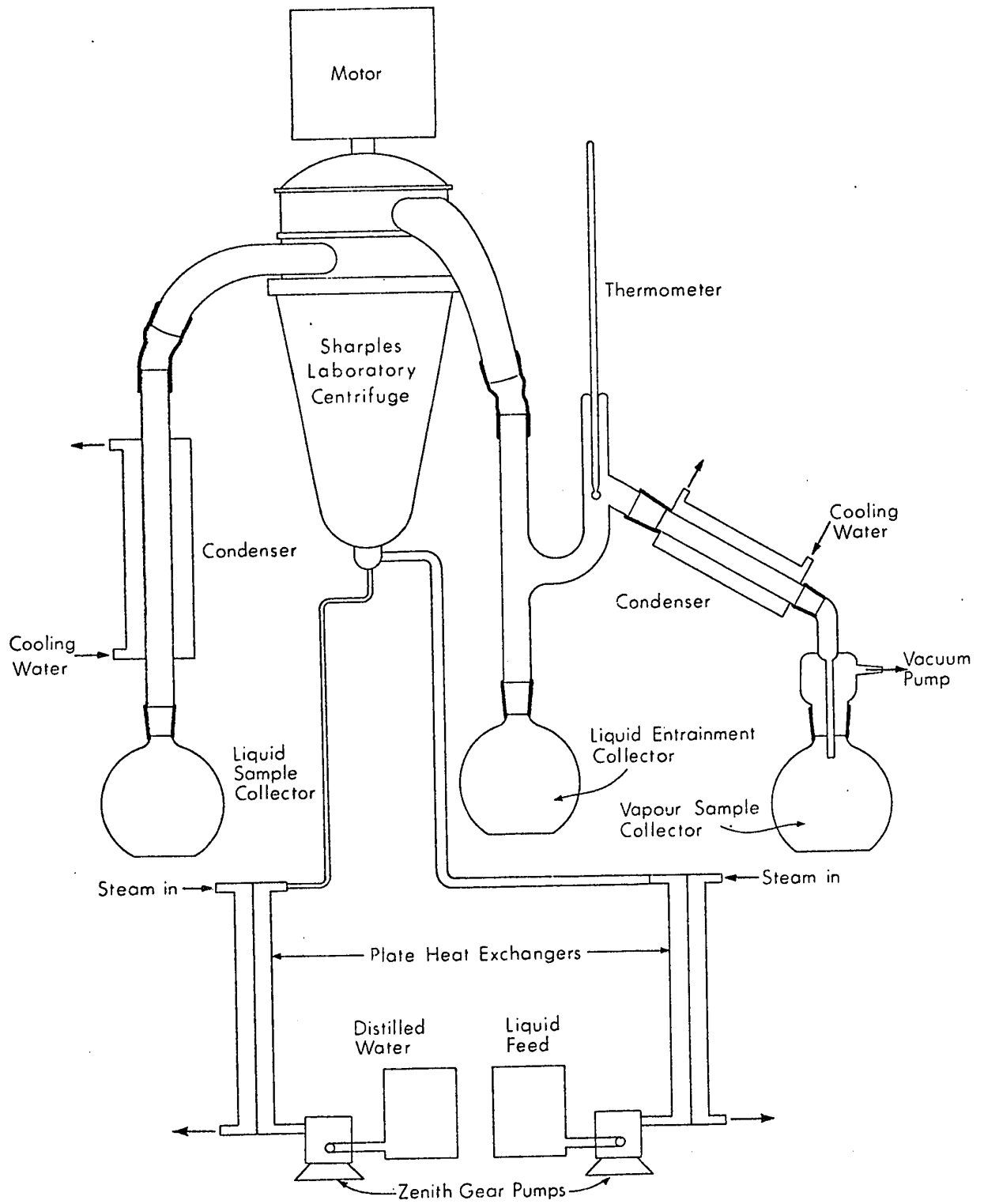


Figure 12

FEED AND OUTLET SYSTEMS OF THE CENTRIFUGAL DEODORIZER

A stainless steel gear pump¹ was used to feed a steady uniform flow of the liquid stream through a steam heated stainless steel heat exchanger to the injection port of the centrifuge. Culinary steam for the deodorizer was generated by pumping distilled water at a steady uniform rate through another steam heated stainless steel plate heat exchanger.

The deodorized liquid stream flowed from the lower collecting bowl of the centrifuge to a Liebig condenser which served to cool the liquid before collection of samples for analysis.

The vapour from the centrifuge was drawn through a "Y" tube fitted with a flask to collect any liquid entrained with the vapour, and thence to a Liebig condenser cooled with ice water. A thermometer was inserted into the vapour line at the upper end of the condenser, and samples of condensed vapour were collected in a flask fitted to the lower end of the condenser. The line from the vacuum pump (which was used to reduce loss of vapour) was connected to the fitting at the lower end of the vapour stream condenser.

Aliquots of about 25 ml were taken from the collected liquid and condensed vapour, and stored at approximately 5°C in test tubes with screw caps until required for analysis.

¹ Zenith Products Co., 432 Cherry Street, West Newton, Mass., U.S.A.

The speed of the centrifuge was adjusted with the aid of a strobe light¹.

¹ Strobotac, type 1538-A; General Radio Company, Concord, Mass., U.S.A.

V. RESULTS AND DISCUSSION

A. VAPOUR LIQUID EQUILIBRIUM EXPERIMENTS

The operation of the Hala vapour liquid equilibrium still was checked with the ethanol water system. The ethanol and water concentrations were determined with a pycnometer and the resulting data are presented in Figure 13. The data published in Perry (1963) p. 13-5, is also presented for comparison. Two data which were obtained with the liquid impinging on the thermometer at the higher ethanol concentration did not fall close to the curve of the published data. It was found that modification of the inlet to the equilibrium chamber gave values which were consistent with the published data. This inlet was used for all subsequent determinations of vapour liquid equilibrium constants.

Vapour liquid equilibrium data were obtained for diacetyl in water at three pressures, and for diacetyl in skim milk at two pressures. The data for water and for skim milk are presented in Figures 14 and 15, respectively, and the vapour liquid equilibrium constants calculated from the slopes of the curves are presented in Table 3, and are shown graphically (along with the data of McDowall, 1955a) in Figure 16. It is seen that the vapour liquid equilibrium constants for diacetyl in water and for diacetyl in skim milk are linear functions of pressure, and that the data obtained in the present work and the data of McDowall (1955a)

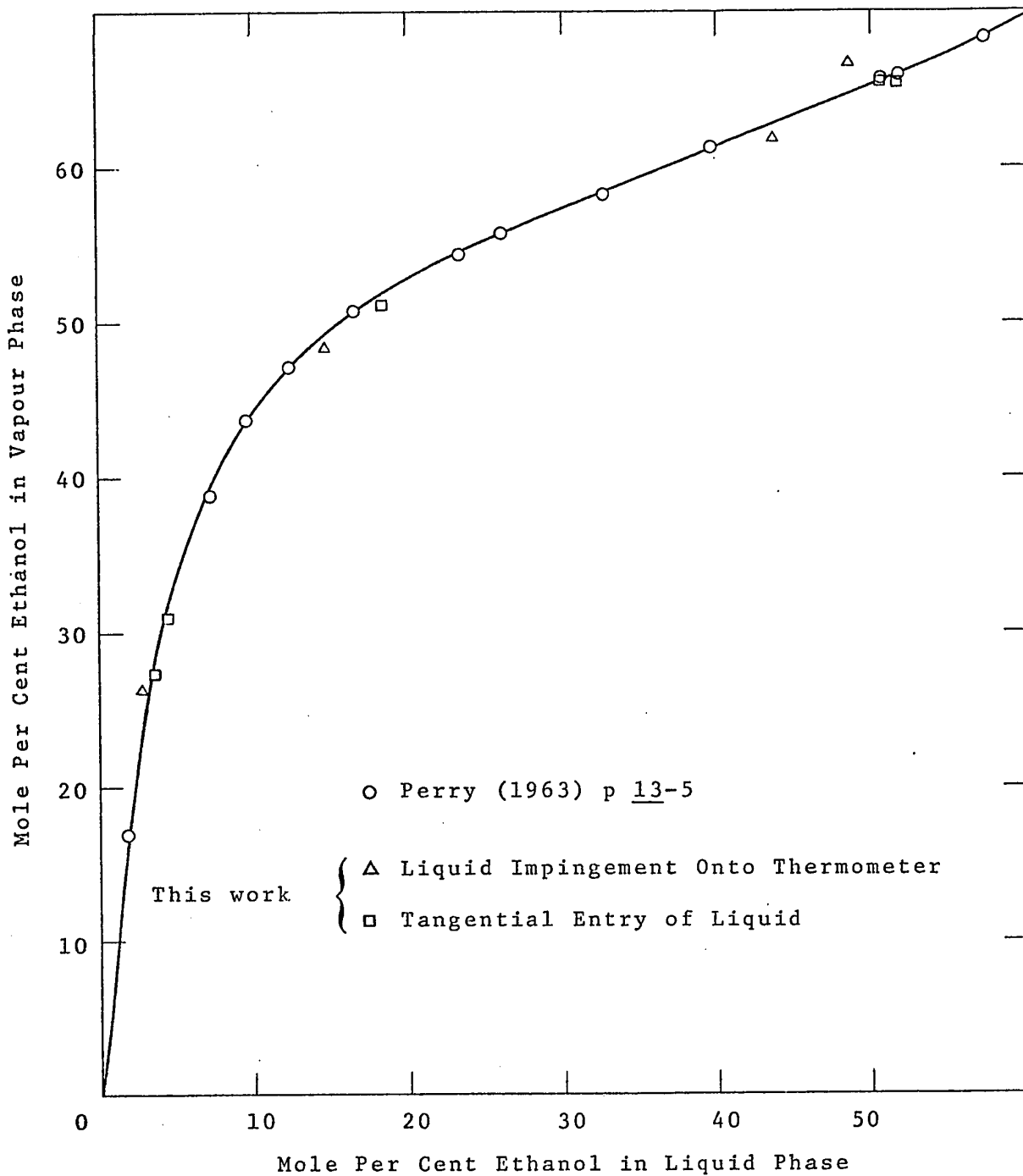


Figure 13

VAPOUR LIQUID EQUILIBRIUM DATA FOR ETHANOL WATER
AT ATMOSPHERIC PRESSURE

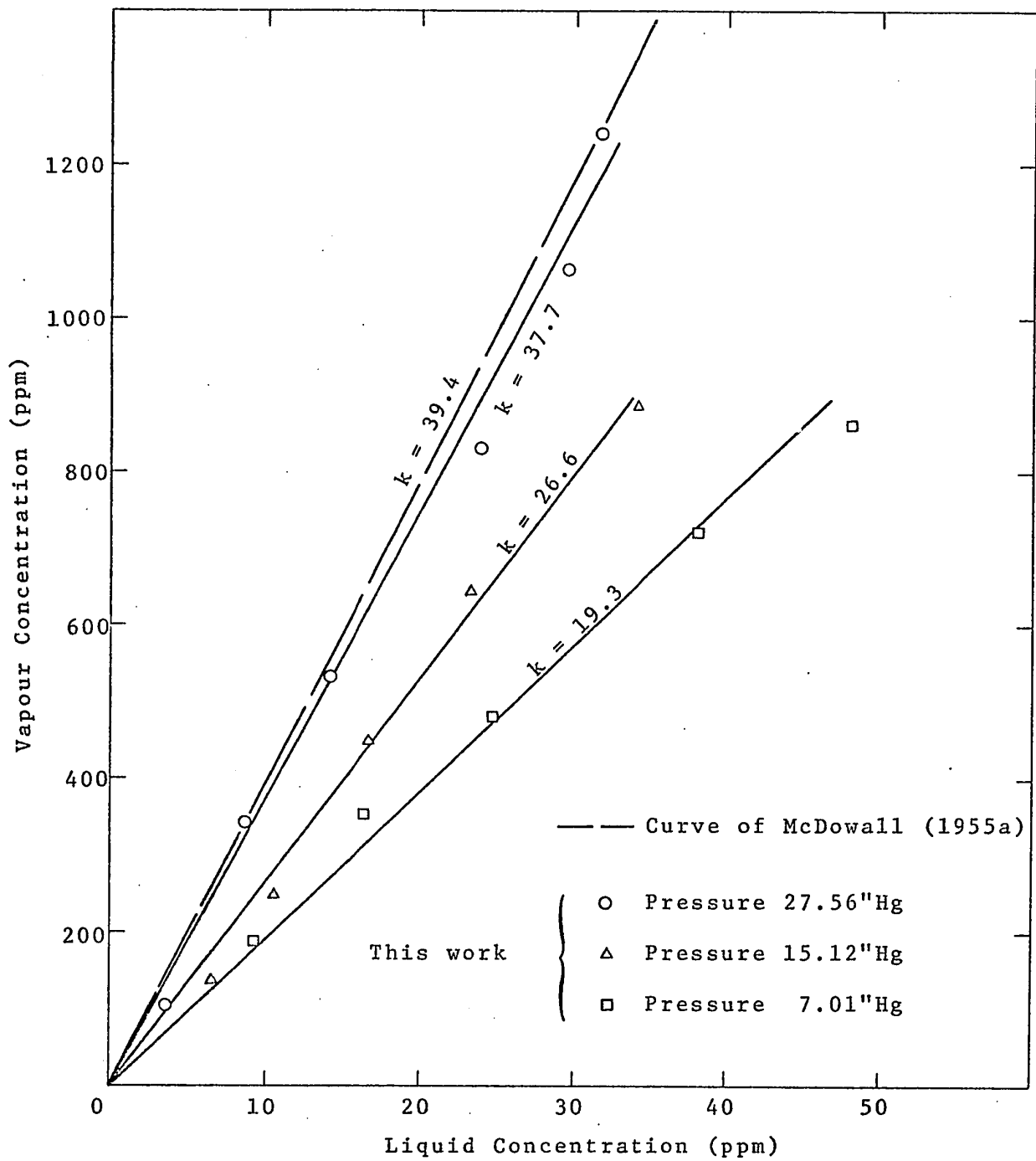


Figure 14

VAPOUR LIQUID EQUILIBRIUM RELATIONSHIPS FOR
DIACETYL WATER

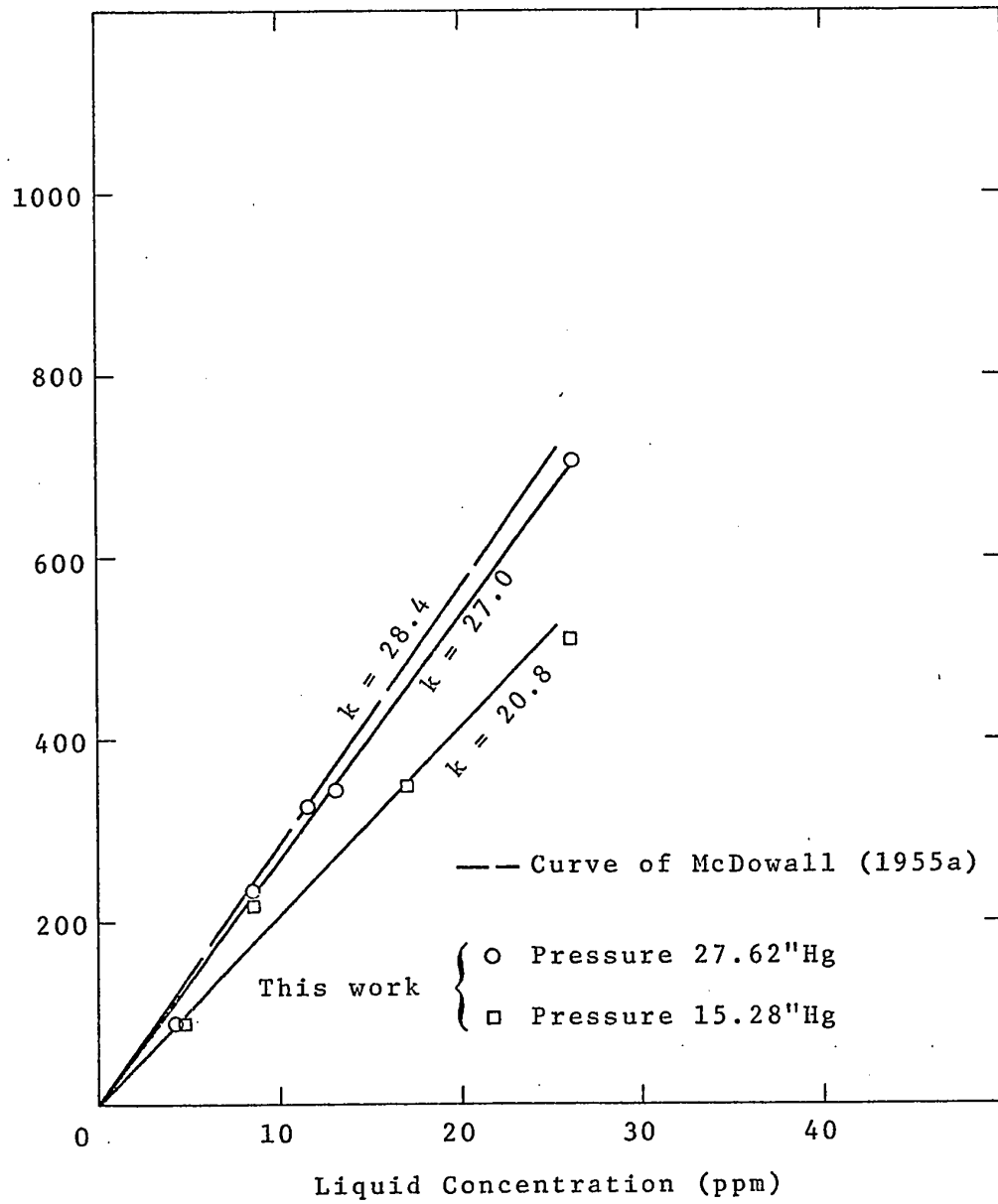


Figure 15

VAPOUR LIQUID EQUILIBRIUM RELATIONSHIPS FOR
DIACETYL SKIM MILK

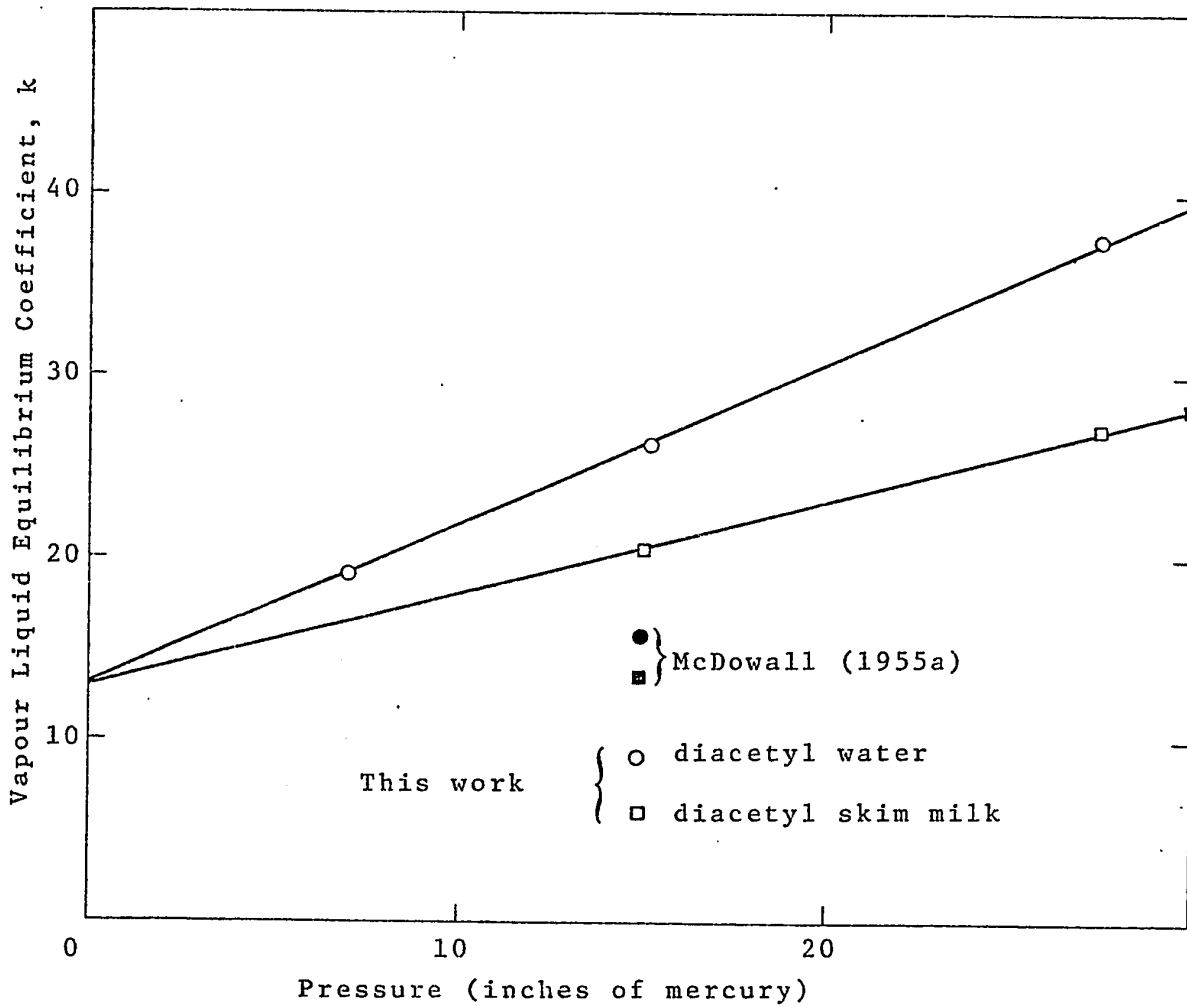


Figure 16

EFFECT OF PRESSURE ON
VAPOUR LIQUID EQUILIBRIUM CONSTANTS
FOR DIACETYL IN WATER AND IN SKIM MILK

Table 3

VAPOUR LIQUID EQUILIBRIUM COEFFICIENTS FOR
DIACETYL WATER AND DIACETYL SKIM MILK
SYSTEMS AT REDUCED PRESSURES

System	Pressure (in. Hg)	Vapour Liquid Equilibrium Coefficient
diacetyl water	27.56	37.7
	15.12	26.6
	7.01	19.3
diacetyl skim milk	27.62	27.0
	15.28	20.8

are consistent. It is of interest to note that the lines for the diacetyl water and for the diacetyl skim milk systems intersect at zero pressure. It is not known whether this phenomenon is peculiar to diacetyl or not. If it can be applied to other taint water and taint skim milk systems then it could be very useful in determining the effect of pressure on the vapour liquid equilibrium coefficient for skim milk, when data for the taint in skim milk at one pressure only are available along with data for the taint in water at several pressures.

B. CENTRIFUGAL DEODORIZATION EXPERIMENTS

i. Results

a. Raw Data

The types of liquid being deodorized, the types of injection port and the holdup volumes used in the eight tests reported (i.e. tests 12 - 19) are summarized in Table 4. Two initial diacetyl concentrations, ranging from 0.45 to 2.7 ppm and from 3.9 to 24.6 for the low and high concentrations, respectively), two steam flow rates (50 and 100 ml of water per minute to the steam generator), two liquid flow rates (68 and 460 ml/min) and three speeds (0, 6000 and 24,000 RPM) were used in each test. The outcoming vapour was condensed and collected, and the liquid and condensed vapour flow rates were both measured. Samples of the two exit streams were taken and diacetyl concentrations were determined. The results for tests 12 to 19 are shown in Tables 5 to 12, respectively. It should be noted that the inlet steam and outlet vapour flow rates are expressed in terms of flow rates of water to the steam generator and of condensate, respectively.

b. Calculation of Approach Ratios and Replacement of Anomalous Values

During the experiments it was found that significant quantities of diacetyl were being lost from the system. It appeared that the main source of error in the diacetyl

Table 4

COMBINATIONS OF TYPE OF LIQUID, TYPE OF INJECTION PORT
AND HOLD UP VOLUME USED IN CENTRIFUGAL
DEODORIZATION EXPERIMENTS

<u>Test No.</u>	<u>Type of Liquid</u>	<u>Type of Liquid Injection</u>	<u>Hold Up Volume (ml)</u>
12	water	spray	300
13	skim milk	spray	300
14	homo. milk	spray	300
15	cream	spray	300
16	water	no spray	300
17	water	no spray	<10
18	skim milk	no spray	<10
19	unhomog. milk	no spray	<10

Table 6

TEST NO. 13 - SKIM MILK - DIACETYL

Speed RPM	Liquid Concentration In ppm	Steam Flow In ml/min	Liquid Flow In ml/min	Vapour Flow Out ml/min	Vapour Concentration Out ppm	Liquid Flow Out ml/min	Liquid Concentration Out ppm
0	2.1	50	68	37	2.1	77	0.4
6000	2.1	50	68	35	2.8	74	0.3
24000	2.1	50	68	31	2.3	72	0.2
0	2.1	50	460	35	8.9	500	0.7
6000	2.1	50	460	35	7.5	430	0.5
24000	2.1	50	460	33	8.7	505	0.7
0	2.1	100	68	40	2.6	118	0.3
6000	2.1	100	68	39	2.6	125	0.7
24000	2.1	100	68	35	2.5	122	0.4
0	2.1	100	460	38	7.5	530	0.6
6000	2.1	100	460	45	7	480	0.7
24000	2.1	100	460	38	8.5	550	0.7
0	9.6	50	68	39	14	69	1
6000	9.6	50	68	46	18	62	1.4
24000	9.6	50	68	34	12.2	83	1.6
0	9.6	50	460	46	95	450	2.8
6000	9.6	50	460	46	76	480	2.8
24000	9.6	50	460	42	80	480	3.2
0	9.6	100	68	44	12.2	114	1
6000	9.6	100	68	40	9.6	110	0.8
24000	9.6	100	68	39	12.5	130	1.5
0	9.6	100	460	42	87	500	4.2
6000	9.6	100	460	42	77	470	2.8
24000	9.6	100	460	39	85	526	3.4

Table 7

TEST NO. 14 - HOMOGENIZED MILK - DIACETYL

Speed RPM	Liquid Concentration In ppm	Steam Flow In ml/min	Liquid Flow In ml/min	Vapour Flow Out ml/min	Vapour Concentration Out ppm	Liquid Flow Out ml/min	Liquid Concentration Out ppm
0	2.6	50	68	37	4	87	1.4
6000	2.6	50	68	36	4.1	75	1.1
24000	2.6	50	68	34	4	75	1.4
0	2.6	50	460	36	14.5	480	1.2
6000	2.6	50	460	35	15.5	475	1.2
24000	2.6	50	460	35	17.1	480	1.2
0	2.6	100	68	42	4	118	0.4
6000	2.6	100	68	40	4	105	0.5
24000	2.6	100	68	39	3.7	125	0.5
0	2.6	100	460	40	13.5	495	1.7
6000	2.6	100	460	45	11.8	450	0.9
24000	2.6	100	460	40	14	500	1
0	13.5	50	68	40	20.6	85	1.6
6000	13.5	50	68	48	23	79	2.5
24000	13.5	50	68	40	20.6	86	3
0	13.5	50	460	45	124	500	4
6000	13.5	50	460	45	88	450	4.5
24000	13.5	50	460	45	110	480	4.9
0	13.5	100	68	45	18.5	112	1.7
6000	13.5	100	68	40	17.5	120	1.6
24000	13.5	100	68	40	17.8	115	2
0	13.5	100	460	40	76	500	4.5
6000	13.5	100	460	45	115	480	4.5
24000	13.5	100	460	40	97	500	4.1

Table 8
 TEST NO. 15 - 10% CREAM - DIACETYL

Speed RPM	Liquid Concentration In ppm	Steam Flow In ml/min	Liquid Flow In ml/min	Vapour Flow Out ml/min	Vapour Concentration Out ppm	Liquid Flow Out ml/min	Liquid Concentration Out ppm
0	1.6	50	68	35	1.9	82	0.2
6000	1.6	50	68	34	2	85	0.4
24000	1.6	50	68	34	1.9	88	0.2
0	1.6	50	460	35	4.5	490	1
6000	1.6	50	460	38	4.6	415	0.6
24000	1.6	50	460	35	5	475	0.6
0	1.6	100	68	36	2	114	0.3
6000	1.6	100	68	38	1.6	102	0.3
24000	1.6	100	68	38	1.4	111	0.2
0	1.6	100	460	41	3.9	530	0.6
6000	1.6	100	460	46	3.7	430	0.4
24000	1.6	100	460	43	4	475	0.5
0	3.9	50	68	38	4.5	86	0.6
6000	3.9	50	68	41	5.8	109	0.8
24000	3.9	50	68	38	4.9	89	0.8
0	3.9	50	460	42	16.7	490	1.4
6000	3.9	50	460	44	25.7	420	1.6
24000	3.9	50	460	41	21.5	480	1.8
0	3.9	100	68	40	4.6	130	0.6
6000	3.9	100	68	39	5.4	105	0.5
24000	3.9	100	68	36	5.8	130	0.6
0	3.9	100	460	38	16.5	515	1.5
6000	3.9	100	460	40	14	450	1.5
24000	3.9	100	460	38	18	460	1.8

Table 9

TEST NO. 16 - WATER - DIACETYL

Speed	Liquid Concentration In	Steam Flow In	Liquid Flow In	Vapour Flow Out	Vapour Concentration Out	Liquid Flow Out	Liquid Concentration Out
RPM	ppm	ml/min	ml/min	ml/min	ppm	ml/min	ppm
0	1.1	50	68	40	2.2	76	0.4
6000	1.1	50	68	39	2.3	75	0.2
24000	1.1	50	68	34	2	78	0.2
0	1.1	50	460	37	5.6	485	0.9
6000	1.1	50	460	39	5.3	470	0.6
24000	1.1	50	460	37	4.8	485	0.8
0	1.1	100	68	65	1.7	82	0.2
6000	1.1	100	68	60	1.2	80	0.2
24000	1.1	100	68	64	1.2	84	0.2
0	1.1	100	460	65	7	480	0.5
6000	1.1	100	460	63	2.4	485	0.3
24000	1.1	100	460	66	3.4	500	0.3
0	9.5	50	68	38.5	13	80	0.7
6000	9.5	50	68	39	21	75	0.3
24000	9.5	50	68	33	20	77	0.3
0	9.5	50	460	40	41	500	3.3
6000	9.5	50	460	43	29	485	3.7
24000	9.5	50	460	38	32	485	3.7
0	9.5	100	68	66	10.5	71	0.9
6000	9.5	100	68	60	6.6	72	1.5
24000	9.5	100	68	62.6	7.3	76	1.5
0	9.5	100	460	66	50	510	2.7
6000	9.5	100	460	45	20	485	2.7
24000	9.5	100	460	46	9.7	470	2.9

Table 10

TEST NO. 17 - WATER - DIACETYL

Speed RPM	Liquid Concentration In ppm	Steam Flow In ml/min	Liquid Flow In ml/min	Vapour Flow Out ml/min	Vapour Concentration Out ppm	Liquid Flow Out ml/min	Liquid Concentration Out ppm
0	0.45	50	68	36	0.4	78	0.09
6000	0.45	50	68	38	0.78	74	0.05
24000	0.45	50	68	35	0.75	78	0.09
0	0.45	50	460	37	2.65	500	0.2
6000	0.45	50	460	37	2.76	500	0.2
24000	0.45	50	460	38	2.76	500	0.2
0	0.45	100	68	71	0.2	89	0.06
6000	0.45	100	68	72	0.25	89	0.09
24000	0.45	100	68	70	0.2	88	0.05
0	0.45	100	460	67	1.15	500	0.15
6000	0.45	100	460	68	0.85	500	0.13
24000	0.45	100	460	69	1.1	500	0.1
0	4.3	50	68	42	5.44	82	0.95
6000	4.3	50	68	39	7.22	84	0.55
24000	4.3	50	68	37	7.22	84	0.6
0	4.3	50	460	39	24.8	500	2
6000	4.3	50	460	38	28.3	500	2.3
24000	4.3	50	460	39	34	500	2.2
0	4.3	100	68	75	2.25	88	0.75
6000	4.3	100	68	73	0.7	88	0.33
24000	4.3	100	68	71	1.15	90	0.38
0	4.3	100	460	74	10.9	500	1.85
6000	4.3	100	460	73	11.9	500	1.7
24000	4.3	100	460	72	12.4	500	1.35

Table 11

TEST NO. 18 - SKIM MILK - DIACETYL

Speed RPM	Liquid Concentration In ppm	Steam Flow In ml/min	Liquid Flow In ml/min	Vapour Flow Out ml/min	Vapour Concentration Out ppm	Liquid Flow Out ml/min	Liquid Concentration Out ppm
0	2.7	50	68	40	5	87	0.8
6000	2.7	50	68	40	4.5	87	0.5
24000	2.7	50	68	40	4.8	87	0.3
0	2.7	50	460	40	5.7	500	1.6
6000	2.7	50	460	40	28.5	500	1.7
24000	2.7	50	460	40	45	500	1
0	2.7	100	68	71	2.7	85	0.5
6000	2.7	100	68	71	2.8	85	0.3
24000	2.7	100	68	71	2.1	85	0.2
0	2.7	100	460	71	17.5	500	1
6000	2.7	100	460	71	17.7	500	1.2
24000	2.7	100	460	71	25.5	500	0.7
0	24.6	50	68	40	14.2	85	10.1
6000	24.6	50	68	40	26	85	3
24000	24.6	50	68	40	28	85	2.7
0	24.6	50	460	40	330	500	13.6
6000	24.6	50	460	40	390	500	12.2
24000	24.6	50	460	40	420	500	11.7
0	24.6	100	68	71	19.5	85	3
6000	24.6	100	68	71	25	85	2.3
24000	24.6	100	68	71	26.5	85	2.5
0	24.6	100	460	71	120	500	10.5
6000	24.6	100	460	71	140	500	12.5
24000	24.6	100	460	71	180	500	9

Table 12

TEST NO. 19 - 3% RAW MILK - DIACETYL

Speed	Liquid Concentration In	Steam Flow In	Liquid Flow In	Vapour Flow Out	Vapour Concentration Out	Liquid Flow Out	Liquid Concentration Out
RPM	ppm	ml/min	ml/min	ml/min	ppm	ml/min	ppm
0	2.5	50	68	40	5.7	87	0.8
6000	2.5	50	68	38	7.8	87	0.6
24000	2.5	50	68	38	7.6	87	0.5
0	2.5	50	460	41	4.8	490	2.4
6000	2.5	50	460	41	5.2	490	1.6
24000	2.5	50	460	41	5.2	490	1.7
0	2.5	100	68	71	3.3	92	0.3
6000	2.5	100	68	71	3.1	92	0.5
24000	2.5	100	68	71	4.8	92	0.4
0	2.5	100	460	70	17.5	515	1.5
6000	2.5	100	460	70	20.1	515	1.5
24000	2.5	100	460	70	17.5	515	1
0	24.5	50	68	40	22.2	92	8
6000	24.5	50	68	40	35	92	6.2
24000	24.5	50	68	40	36.1	92	4
0	24.5	50	460	40	325	500	10.7
6000	24.5	50	460	40	280	500	11.4
24000	24.5	50	460	40	355	500	13
0	24.5	100	68	71	36.5	92	2.9
6000	24.5	100	68	71	15	92	5.7
24000	24.5	100	68	71	8.5	92	2.5
0	24.5	100	460	71	175	515	10.3
6000	24.5	100	460	71	140	515	8.5
24000	24.5	100	460	71	140	515	9

mass balances were in the vapour flow rates and concentrations. In spite of several modifications to equipment and techniques it was found that it was not possible to reduce all mass balance errors to less than 10% of the incoming diacetyl. The vapour flow rates may have been in error because there was no guarantee with the equipment used that all the vapour was drawn through the condenser, even when a vacuum pump was used. Further, some air was of necessity drawn through the condenser and this would effect some stripping of diacetyl from the cooled condensate, and in fact diacetyl odor was detected at the exhaust of the vacuum pump in some of the tests.

The effectiveness of the centrifugal deodorizer has been expressed as an approach ratio R which is given by the equation

$$R = \frac{x_2 - x_{2e}}{x_1 - x_{2e}} \quad (V.1)$$

where x_1 and x_2 are the concentrations of diacetyl in the inlet and outlet streams, respectively, and x_{2e} is the concentration of diacetyl in the liquid stream leaving a single stage deodorizer in which equilibrium between the vapour, aqueous and fat phases is achieved.

The equilibrium outlet concentration x_{2e} is calculated using the equation

$$x_{2e} = \frac{L_1 x_1 (1 + (F_2/L_2)(D - 1))}{L_2 (1 + (F_2/L_2)(D - 1)) + Vk} \quad (V.2)$$

where F , L and V are mass flow rates of fat, liquid and vapour, respectively, x is the diacetyl concentration in the liquid and subscripts 1 and 2 refer to the inlet and outlet liquid flows.

In the early calculations the vapour flow rate was calculated from the overall mass balance equation, i.e.

$$V = L_1 + S - L_2 \quad (V.3)$$

but the effect of the scatter in the observed values of L_2 was to give a scatter in the vapour flow as calculated by difference above. This then gave a large scatter in the values of x_{2e} because the product Vk is the largest term in the denominator of equation V.2 above. The error introduced in this way was such that 28 of the 192 values of the approach ratio calculated from the experimental data were less than 0, which implies that equilibrium has been approached and passed in a single mixing stage.

In the analyses of variance, these ratios were considered to be "incorrect" and hence they were treated as missing data and replaced with expected values based on the other data in that particular analysis. In any

case, if the approach ratio R is less than 0, it is impossible to calculate the logarithm of the approach ratio, which is proportional to $(-k_L A.t)$ the product of the mass transfer coefficient, the total area of contact and the time of contact in a simple mixing stage, and replacement of the values of $\log R$ in any analysis of variance on $\log R$ is mandatory.

Because the number of data to be treated as incorrect was large (i.e. 28 of 192), alternative methods of reducing the number of "incorrect" values was sought. The only reasonable possibility was to assume that no condensation of input steam occurred, i.e. $V = S$ and $L_2 = L_1$. With many of the data, the vapour flow calculated from $V = S$ is greater than that calculated by difference from equation V.3, and hence the equilibrium outlet liquid diacetyl concentration x_{2e} as calculated with $V = S$ is less than that calculated with equation V.3. The recalculated approach ratios on the new basis would then tend to be greater than those based on equation V.3, but it was felt the significance of the different variables and the general trends could be more clearly evaluated if the number of "incorrect" ratios could be reduced.

When the approach ratios were recalculated using the assumption of no condensation, it was found that only 4 of the 192 values of the approach ratio were less than 0, and hence all calculations and analyses of variance have

been done on this basis, i.e. using the equation

$$x_{2e} = \frac{L_1 x_1 (1 + (F_1/L_1)(D - 1))}{L_1 (1 + (F_1/L_1)(D - 1)) + S_k} \quad (\text{V.4})$$

The approach ratios as calculated for the eight tests are shown in Table 13.

All calculations were done by an IBM 360/67 computer operating under MTS (Michigan Terminal System). The APL program used for calculating the approach ratios from the raw data of Tables 5 to 12 is presented in Appendix C. The raw data (flow rates, concentrations, etc.) were stored in the user's individual library, and were copied into the active workspace as required. Additional data (i.e. equilibrium coefficients, fat contents and densities of the liquid streams, as shown in Table 14, were entered through the terminal. The approach ratios were compared to zero and all negative (i.e. "incorrect") values were replaced with zeroes.

The calculation of the expected values of the approach ratio to replace the "incorrect" values is based on the model for analysis of variance, i.e.

$$R_{IJ\dots N} = \bar{R} + \Delta R_I + \Delta R_J + \dots + \Delta R_N + E \quad (\text{V.5})$$

Table 13
APPROACH RATIOS FOR CENTRIFUGAL DEODORIZER

Factor	Test 12	Test 13	Test 14	Test 15	Test 16	Test 17	Test 18	Test 19
C ₁ V ₁ L ₁ R ₁	0.0689	0.1502	0.5153	0.0806	0.3416	0.1723	0.2613	0.2859
C ₁ V ₁ L ₁ R ₂	0.0689	0.1002	0.3942	0.2120	0.1535	0.0804	0.1447	0.2019
C ₁ V ₁ L ₁ R ₃	0.1723	0.0502	0.5153	0.0806	0.1535	0.1723	0.0669	0.1599
C ₁ V ₁ L ₂ R ₁	0.3213	0.1067	0.2771	0.4955	0.7756	0.3144	0.4541	0.9463
C ₁ V ₁ L ₂ R ₂	0.1978	-0.0210	0.2771	0.1591	0.4391	0.3144	0.5037	0.5167
C ₁ V ₁ L ₂ R ₃	0.1361	0.1067	0.2771	0.1591	0.6634	0.3144	0.1563	0.5704
C ₁ V ₂ L ₁ R ₁	0.0844	0.1216	0.1327	0.1669	0.1677	0.1183	0.1649	0.0980
C ₁ V ₂ L ₁ R ₂	0.0844	0.3168	0.1721	0.1669	0.1677	0.1862	0.0890	0.1800
C ₁ V ₂ L ₁ R ₃	0.0844	0.1704	0.1721	0.1028	0.1677	0.0957	0.0511	0.1390
C ₁ V ₂ L ₂ R ₁	0.3856	0.1650	0.5949	0.2674	0.3907	0.2553	0.2640	0.5319
C ₁ V ₂ L ₂ R ₂	-0.0053	0.2207	0.2348	0.1210	0.1876	0.2057	0.3506	0.5319
C ₁ V ₂ L ₂ R ₃	0.1064	0.2207	0.2798	0.1942	0.1876	0.1312	0.1341	0.2978
C ₂ V ₁ L ₁ R ₁	0.1396	0.0596	0.0743	0.1109	0.0416	0.1940	0.3813	0.2928
C ₂ V ₁ L ₁ R ₂	0.0852	0.1033	0.1443	0.1648	-0.0019	0.0977	0.0783	0.2156
C ₂ V ₁ L ₁ R ₃	0.0961	0.1252	0.1832	0.1648	-0.0019	0.1098	0.0655	0.1213
C ₂ V ₁ L ₂ R ₁	0.1037	0.0508	0.0553	0.1375	0.1946	0.3399	0.4008	0.2438
C ₂ V ₁ L ₂ R ₂	0.0387	0.0508	0.1050	0.2065	0.2466	0.4260	0.3246	0.2822
C ₂ V ₁ L ₂ R ₃	0.0777	0.1067	0.1448	0.2755	0.2466	0.3973	0.2973	0.3698
C ₂ V ₂ L ₁ R ₁	0.0791	0.0819	0.1040	0.1324	0.0791	0.1601	0.1001	0.0963
C ₂ V ₂ L ₁ R ₂	0.0898	0.0606	0.0964	0.1061	0.1433	0.0608	0.0710	0.2134
C ₂ V ₂ L ₁ R ₃	0.0909	0.1353	0.1268	0.1324	0.1433	0.0726	0.0793	0.0796
C ₂ V ₂ L ₂ R ₁	0.2710	0.3425	0.2198	0.2788	0.2004	0.3635	0.3300	0.3217
C ₂ V ₂ L ₂ R ₂	0.1652	0.1720	0.2198	0.2788	0.2004	0.3246	0.4250	0.2357
C ₂ V ₂ L ₂ R ₃	0.1652	0.2450	0.1851	0.3689	0.2240	0.2337	0.2587	0.2596

Table 14

EQUILIBRIUM COEFFICIENTS, FAT CONTENTS
AND FLUID DENSITIES FOR TESTS 12-19

Test No.	Vapour Aqueous	Fat Phase	Fat Content	Fluid Density
	Phase Equilibrium Coefficient	Aqueous Phase Distribution Coefficient		
	K	D	F	RH
12	39.3	1.33	0	1.00
13	28.4	1.33	0	1.037
14	28.4	1.33	0.030	1.035
15	28.4	1.33	0.10	1.024
16	39.3	1.33	0	1.00
17	39.3	1.33	0	1.00
18	28.4	1.33	0	1.037
19	28.4	1.33	0.030	1.035

where $R_{IJ\dots N}$ is the observed value of the approach ratio for level I of factor A, level J of factor B, etc., \bar{R} is the overall mean approach ratio, and ΔR_I , ΔR_J , etc. are the differences from the overall mean due to level I of factor A, level J of factor B, etc., respectively, and E is the error term.

The differences due to the particular levels of the various effects (i.e. ΔR_I , ΔR_J , etc.) are given by equations of the form

$$\begin{aligned} \Delta R_I &= \bar{R}_I - \bar{R} \\ &= \frac{1}{n_I} \sum_{\substack{\text{all} \\ j\dots n}} R_{Ij\dots n} - \frac{1}{n_T} \sum_{\substack{\text{all} \\ ij\dots n}} R_{ij\dots n} \end{aligned} \quad (\text{V.6})$$

where \bar{R}_I is the average value of the approach ratio for level I of factor A and n_I is the number of "correct" values of the approach ratio at level I. The term \bar{R} is the overall mean approach ratio, and n_T is the total number of "correct" values of the approach ratio. The summations in equation V.6 above can be expressed in the general form (all j...n) and (all ij...n) only if all values of R to be recalculated have been replaced by zeroes. If the values to be recalculated are not replaced by zeroes before the calculation is done, then the "incorrect" values

must be excluded from the summations.

The expected value of the approach ratio is then given by

$$\left(R_{IJ\dots N} \right)_{\text{expected}} = \bar{R} + \Delta R_I + \Delta R_J + \dots \Delta R_N$$

and because there is no contribution from experimental error, (i.e. E of equation V.5), the number of degrees of freedom of the error sum of squares in the analysis of variance is reduced by 1 for each value which has been replaced by a "corrected" value.

An APL program (named REPLACE) was prepared to calculate the expected values, and is presented in Appendix D. The matrix Z which is to be corrected could be entered from the terminal or copied from the user's individual library and the shape of the array (i.e. numbers of levels of the various factors), the coordinates of the points to be recalculated and the number of these points was entered through the terminal before calling the program. The complete array with expected values replacing the "incorrect" values of the original array was then printed out.

It should be noted that average approach ratios for various combinations of factors were calculated from the "correct" data, i.e. the "expected" values were not included in the averages. These averages were calculated

using the sum reduction function (+/) over the desired coordinates of the array of data in which the "incorrect" data had been replaced by zeroes. The number of terms in each average was computed using the sum reduction function on an array of 0's and 1's which represented the "incorrect" and "correct" data, respectively. The APL statement for calculation and print out of the averages is

```
□←M←(+/[A](+/[B](+/[C](J1))))÷(+/[A](+/[B](+/[C](J1≠0))))
```

where J1 is the array of data, M is the matrix of averages and the values of A, B and C depend on the coordinates over which the averaging is done.

ii. Analysis of Results

a. Analysis of Variance Calculations

All analyses of variance were done using the ANOVA APL library program which presented the results of the analysis as a matrix T of four columns, viz. identification of factors, degrees of freedom, sums of squares and mean squares. The function definition of the ANOVA program as called from the library was changed in the workspace from

```
V T←ANOVA D;DIM;N;REPS;K;R;CT;V;I;S;
```

to

V ANOVA D;DIM;N;REPS;K;R;CT;V;I;S;

to make T a global variable. This makes the matrix T accessible after the ANOVA program is executed, and enables the operator to refer to individual columns within the matrix T, and to operate on these without having to re-enter all the required degrees of freedom, sums of squares, etc.

Before the matrix T from the ANOVA program was printed out, the variance ratios based on the highest order interaction were calculated by an APL program 'PRINT' (Appendix E). An extra column was added to the left of matrix T from the ANOVA program, numbering the lines consecutively from the top of the matrix for easy reference, and the variance ratios were printed in a column to the left of the matrix T of the ANOVA results. An APL program (POOL, as presented in Appendix F) was used to pool the sums of squares and degrees of freedom for the factors whose effects were not significant. The POOL program calculated a better estimate of the error mean square and the new estimates of the variance ratio for the factors which appeared to be significant, and presented the data as a table of 4 columns (viz., factor number and factor identification (columns 1 and 2, respectively, of the table produced by the PRINT program), the new estimate of the

variance ratio based on the pooled error mean square, and the degrees of freedom). The identification numbers of the factors which were not to be pooled, and the number of points which had been corrected were entered through the terminal when called for by the program. The pooled error sum of squares, the pooled error degrees of freedom (adjusted for the number of points replaced by expected values) and the new estimate of the error mean square were also printed out by the POOL program.

Probability levels have only been considered to be significant when they occurred with greater frequency than would be expected by chance in any given series of analyses. For example, if two factors in a series of analyses of variance gave probability levels of 0.02 and 0.01 (i.e. 1 chance in 50 and 1 chance in 100, respectively, that the effects of the factors are due to experimental error alone). then the 0.01 probability level was considered to be not significant if the total number of mean squares in the series of analyses of variance was greater than 150 (i.e. 50 plus 100). If, on the other hand, 6 factors had given probability levels of 0.01 in a series of analyses with much fewer than 600 mean squares, then the probability level of 0.01 would have been considered to be significant.

Three sets of analyses of variance have been done. The first analysis is for the eight tests taken together to determine the significances of the overall effects

of test T, speed of rotation R, liquid flow rate L, vapour flow rate V and initial concentration of diacetyl C. In the second set the tests are analysed individually, and in the third set the tests are analysed in various grouping to determine the significances of the effects of type of fluid F, type of liquid and steam entrance port E, and of the hold up volume. The results of these three sets of analyses of variance are presented and discussed in section ii.b., ii.c. and ii.d., and the important effects are summarized in section iii.

b. Overall Effects

Four data were replaced by expected values of the approach ratio for the analysis of variance of the overall set of approach ratios for the eight tests. The values of expected approach ratio R used to replace the "incorrect" values of R for $C_1V_2L_2R_2T_1$, $C_1V_1L_2R_2T_2$, $C_2V_1L_1R_2T_5$ and $C_2V_1L_1R_3T_5$ are 0.2080, 0.2486, 0.1626 and 0.1475, respectively.

The numbers of degrees of freedom, sums of squares, the mean squares and variance ratios and probability levels for the significant factors are shown in Table 15.

The vapour flow rate does not have a significant effect on the approach ratio although it does appear in a weak interaction with initial diacetyl concentration. This lack of significance indicates that the controlling resis-

Table 15

SUMMARY OF OVERALL ANALYSIS OF VARIANCE FOR ALL TESTS

Identifica- tion of Factor	Degrees of Freedom	Sum of Squares	Mean Square	Variance Ratio	Probabil- ity Level
T	7	0.4649	0.0664	5.34	<0.001
R	2	0.0977	0.0488	3.92	0.025
L	1	0.8418	0.8418	67.65	<<0.001
C	1	0.1811	0.1811	14.55	<0.001
CxV	1	0.0755	0.0755	6.07	0.02
Error	175	2.178	0.01245		
Total	187	3.839			

tance to mass transfer is not in the vapour phase. This is as expected, because there are more resistances to mass transfer from two phase liquid systems (such as milk and cream) to a vapour than there are in systems in which a single liquid is contacted with a vapour. In any case, the liquid film resistance is usually much greater than the resistance to mass transfer in the vapour film.

Table 15 shows that speed of rotation R is significant at the 0.025 level, which is only slightly less significant than the concentration by vapour flow interaction which has a probability level of 0.02. It is expected that effects with these probabilities would arise by chance from experimental error approximately once in every 40 or 50 mean squares. In this analysis of variance there are nearly 30 insignificant mean squares, and it is doubtful whether either or both of these factors are in fact significant.

This very low significance of the effect of speed on the approach ratio was unexpected, in view of the fact that the flow conditions for the zero speed runs were very different from the flows at 6000 and 24,000 RPM. It was expected that the zero speed run would enable the effect of the spray alone to be determined, but the small effect of speed on the approach ratio does not allow this to be done with any precision.

The average approach ratios at each of the three speeds are plotted in Figure 17 for the eight tests. With the exception of test 13 for which the points are closely grouped, the approach ratios for zero speed are all greater than the 6000 and 24,000 RPM values, which indicates the centrifuge is increasing the amount of deodorization. The 6000 RPM values of the approach ratio are approximately equal to, or are less than, the 24,000 RPM values for tests 12 to 16 in which the hold up volume was large (i.e. about 390 ml), whereas the 6000 RPM approach ratios for tests 17, 18 and 19 (in which the hold up volume was less than 10 ml) are greater than the approach ratios for the 24,000 RPM runs. The effect of speed is more significant when tests 17, 18 and 19 are analysed as a group, and the possible reasons for this are discussed in section d.

The effect of concentration of diacetyl in the liquid feed to the centrifuge on the approach ratio is quite significant, with a probability level of less than 0.001. The average approach ratios for each combination of liquid flow rate and concentration are plotted for each test in Figure 18. In several combinations the approach ratios for the two concentrations are almost equal, but where there is an appreciable difference, the low concentration tended to give greater values of the approach ratio than did the higher concentration for any particular liquid flow rate.

AVERAGE APPROACH RATIO FOR FIGURE 17

Speed		Test Number							
		12	13	14	15	16	17	18	19
0	(○)	0.182	0.135	0.247	0.209	0.274	0.240	0.295	0.352
6000	(□)	0.104	0.146	0.206	0.177	0.220	0.212	0.248	0.297
24000	(△)	0.116	0.145	0.236	0.184	0.255	0.191	0.139	0.250

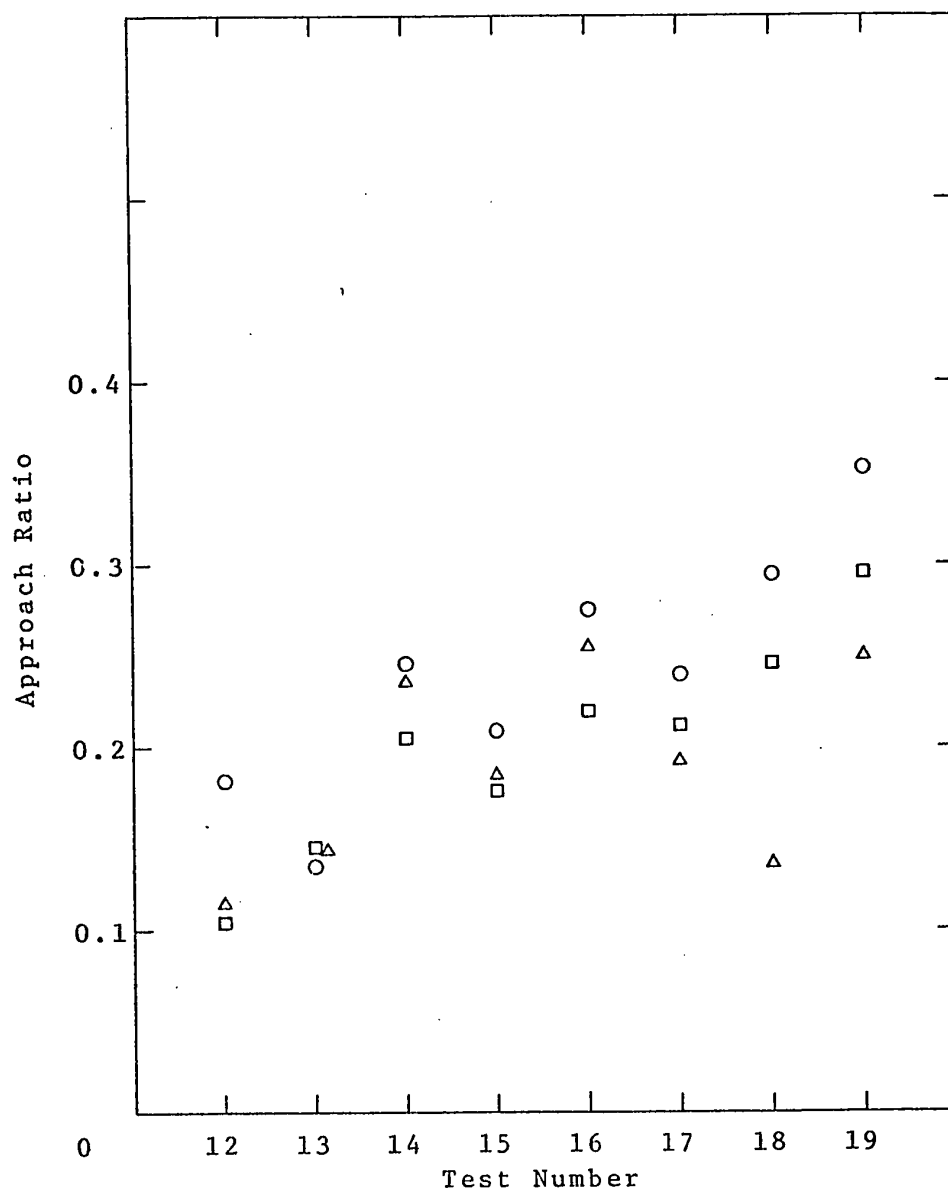


Figure 17

AVERAGE APPROACH RATIOS
AT THREE SPEEDS FOR TESTS 12-19

AVERAGE APPROACH RATIOS FOR FIGURE 18

Factor	Test Number							
	12	13	14	15	16	17	18	19
L ₁ C ₁ (Δ)	0.094	0.152	0.317	0.135	0.192	0.138	0.130	0.177
L ₁ C ₂ (▲)	0.097	0.094	0.122	0.135	0.102	0.116	0.129	0.170
L ₂ C ₁ (□)	0.229	0.164	0.323	0.233	0.441	0.256	0.310	0.566
L ₂ C ₂ (■)	0.137	0.161	0.155	0.258	0.219	0.348	0.339	0.285

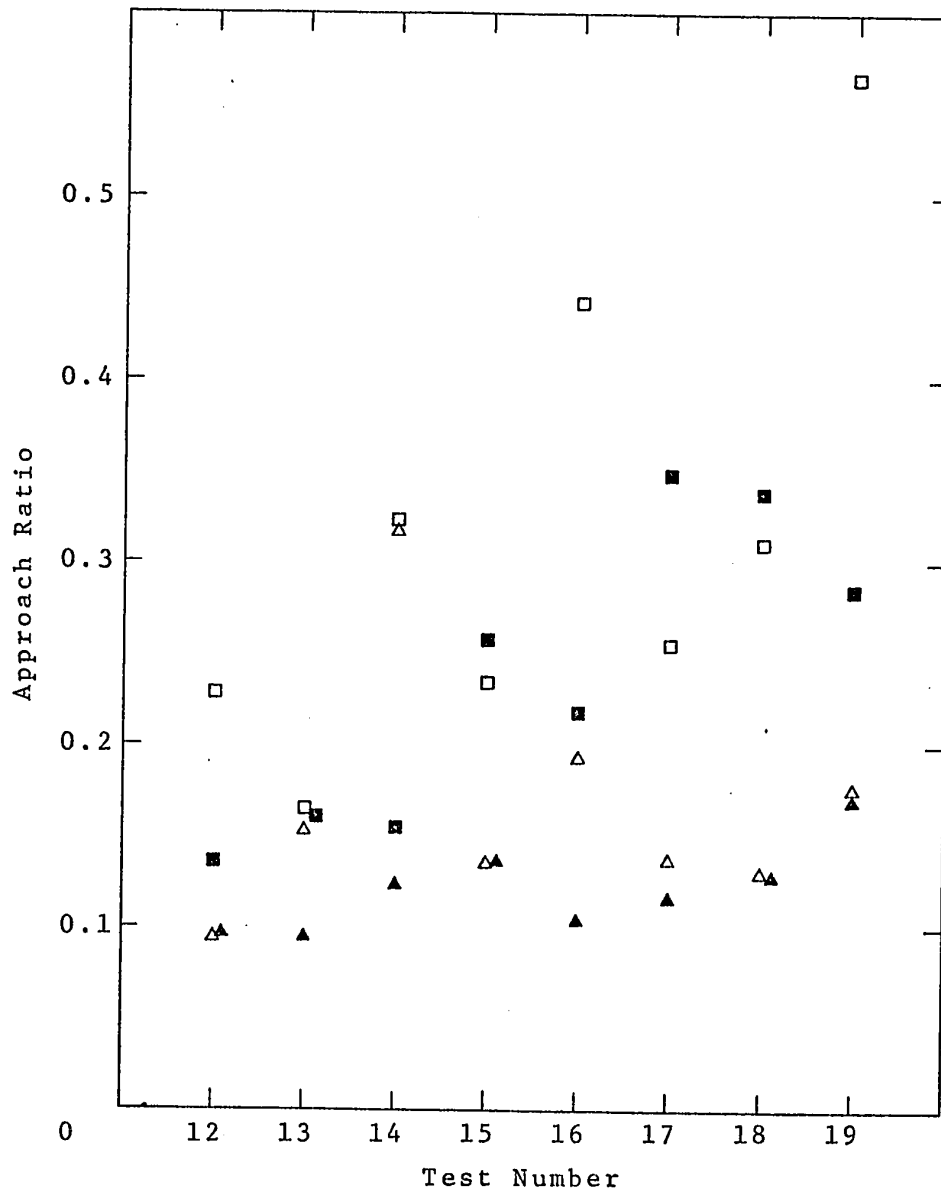


Figure 18

AVERAGE APPROACH RATIOS FOR TWO CONCENTRATIONS
AND TWO LIQUID FLOW RATES FOR TESTS 12-19

It was first thought that this trend was due to uncertainties in the measurement of the diacetyl concentration in the liquid product stream. It is possible that a small systematic error could be introduced at very low concentrations, where the standard curve of diacetyl concentration versus gas chromatograph peak height was not linear. However, examination of the diacetyl concentrations in the liquid product stream in the tables of raw data (Tables 5 to 12 for tests 12 to 19, respectively), showed that the tests in which the effect of concentration is most significant are those tests in which the outlet diacetyl concentrations are relatively high, and hence it is unlikely that this is the reason for the observed effect of diacetyl concentration in the feed liquid. Further examination of the raw data did not lead to any alternative explanation for the quite unexpected effect of concentration.

Liquid flow rate had a much more significant effect on approach ratio than did any other factor (including diacetyl concentration in the feed). The probability level for this effect could not be determined precisely because the variance ratio was much greater than that expected at the 0.001 probability level. Figure 19 (average approach ratios for the two liquid flow rates for test 12 - 19) shows that for all tests the lower liquid flow gave a lower approach ratio than the higher liquid flow (i.e.

AVERAGE APPROACH RATIOS FOR FIGURE 19

Factor	Test Number							
	12	13	14	15	16	17	18	19
L ₁ (Δ)	0.095	0.123	0.219	0.135	0.156	0.127	0.129	0.174
L ₂ (\square)	0.181	0.170	0.239	0.245	0.330	0.302	0.325	0.426

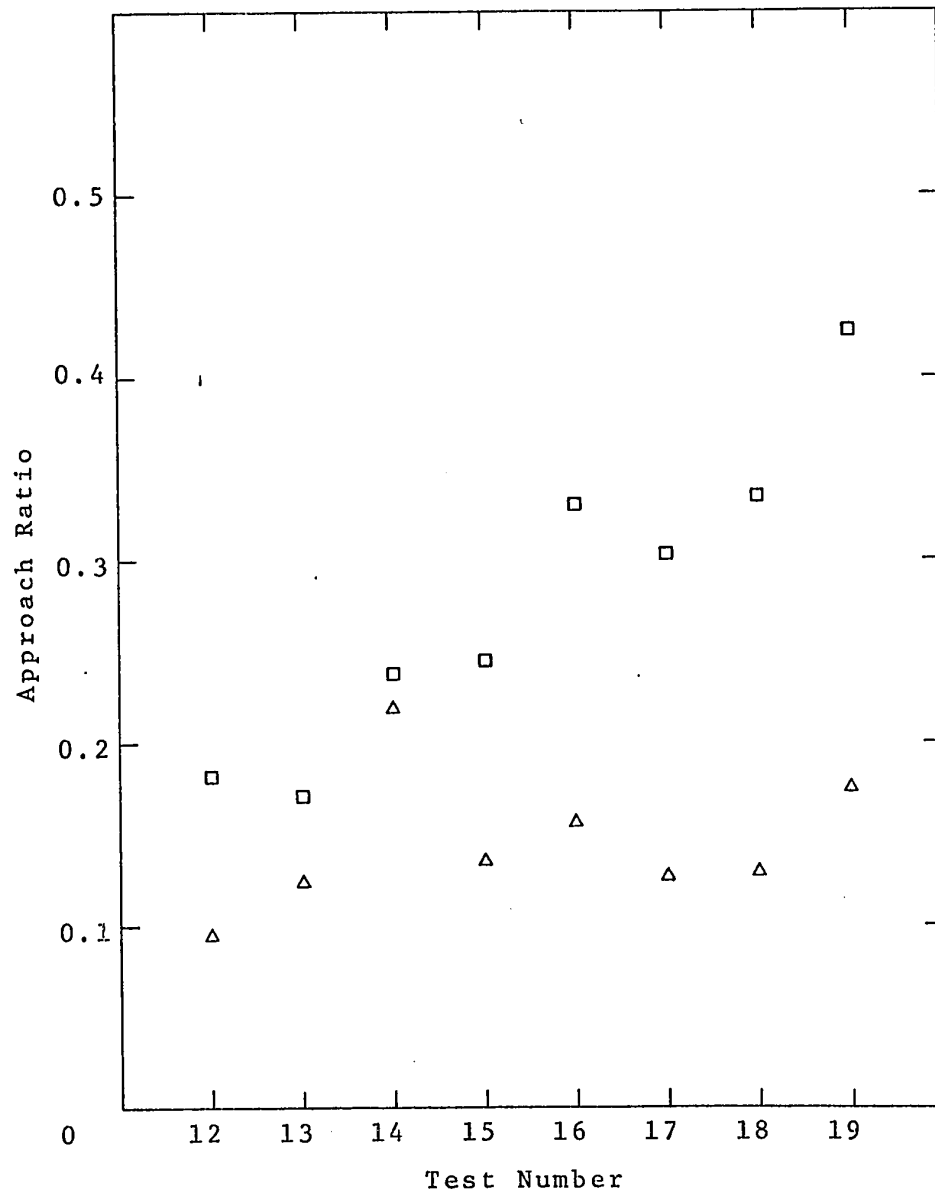


Figure 19

AVERAGE APPROACH RATIOS.
FOR THE TWO LIQUID FLOW RATES FOR TESTS 12-19

the lower the liquid flow rate the more closely equilibrium is approached). This is as expected because the lower liquid flow rates give longer residence times in the centrifugal deodorizer, and is further evidence that there is significant transfer of odor to the vapour from the liquid phase on the wall of the centrifuge bowl. The effect of liquid flow rate is much greater in tests 16 - 19 than it is in tests 12 - 15, and the reasons for this are discussed in section d. below.

The significant effect of test on the approach ratio (probability level less than 0.001) was expected, as there are several factors (viz., type of fluid being deodorized, type of injection port and hold up volume), which were varied between the tests. Test did not appear as a factor in any significant interaction, and therefore the effects of all other factors are approximately the same for all the individual tests.

Examination of sets of approach ratios averaged over various combinations of factors indicated that some factors could be more significant in some individual tests, even though they were not significant enough to show up in the analysis of variance of the overall data. Analyses of variance have therefore been done for the individual tests (with new "expected" values calculated where necessary) and the results of these analyses are presented and dis-

cussed in section c. below.

c. Individual Tests

Expected values to replace the "incorrect data" of tests 12, 13 and 16 were calculated for each individual test. The values of approach ratio for $C_1V_2L_2R_2$ of test 12, $C_1V_1L_2R_2$ of test 13 and $C_2V_1L_1R_2$ and $C_2V_1L_1R_3$ of test 16 are 0.1788, 0.1323, 0.1211 and 0.1565, respectively. The results of the analysis of variance on each individual test are shown in Table 16.

It is seen that there are 5 factors which are significant at the 0.025 probability level. Factors with this probability level would be expected to arise by chance once in 40 mean squares, and as there are 128 (i.e. 8×16) mean squares in this series of analyses, it cannot be said with any certainty that any of these five factors is significant.

Concentration occurs as a significant factor in only two tests, i.e. test 14 with a probability level of less than 0.001 and test 16 with a probability level of 0.005. Figure 18, in which average approach ratios for the combinations of concentration and liquid flow rates are plotted against test number, shows that tests 14 and 16 are the only tests in which the average approach ratios for C_1 for both L_1 and L_2 are appreciably greater than the corresponding ratios for C_2 . The reason for this effect in these

Table 16

SUMMARY OF ANALYSES OF VARIANCE ON INDIVIDUAL TESTS

Test No. (Fluid) (Entrance) (Hold Up)	Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Variance Ratio	Probability
<u>12</u> Water Spray Large	L	1	0.0420	0.0420	8.33	0.01
	Error	19	0.0957	0.00504	-	-
<u>13</u> Skim Milk Spray Large	V	1	0.0513	0.0513	14.30	0.001
	Error	19	0.0682	0.00359	-	-
<u>14</u> Homo Milk Spray Large	C	1	0.1987	0.1987	23.99	<0.001
	LxV	1	0.1094	0.1094	13.21	0.001
	Error	21	0.1739	0.0083	-	-
<u>15</u> Cream Spray Large	L	1	0.0727	0.0727	10.98	0.005
	Error	22	0.1457	0.00662	-	-
<u>16</u> Water Stream Large	L	1	0.1872	0.1872	16.33	0.001
	V	1	0.0677	0.0677	5.90	0.025
	C	1	0.1347	0.1347	11.75	0.005
	LxV	1	0.0482	0.0482	4.21	0.025
	VxC	1	0.0642	0.0642	5.60	0.025
	Error	16	0.1835	0.01147	-	-
<u>17</u> Water Stream Small	L	1	0.1838	0.1838	48.87	<<0.001
	V	1	0.0219	0.0219	5.83	0.025
	Error	21	0.0790	0.00376	-	-
<u>18</u> Skim Milk Stream Small	L	1	0.2293	0.2293	20.48	<0.001
	Error	22	0.2463	0.01120	-	-
<u>19</u> Unhomo Milk Stream Small	L	1	0.3811	0.3811	19.92	<0.001
	C	1	0.0622	0.0622	6.50	0.025
	Error	21	0.4017	0.0191	-	-

two runs is not apparent, as pointed out in section B.ii.b., page 114, but it is clear that the effect of concentration is not a general effect for all tests. Further work will need to be done to see whether the effect of concentration on approach ratio is real or not.

The vapour flow rate V occurs as a significant factor in test 13 (probability level 0.001) and as an interaction term $L \times V$ in test 14 (probability level 0.001). The average approach ratios for test 14 (0.304, 0.189, 0.134 and 0.289 for L_1V_1 , L_2V_1 , L_1V_2 and L_2V_2 , respectively) show that at the low vapour flow rate the approach ratio decreases with increasing flow rate, and vice versa at the high vapour flow rate. The average approach ratios for L_1 and L_2 are approximately equal (0.219 and 0.239, respectively) and hence the effect of $L \times V$ is significant even while liquid flow rate itself has no significant effect. As with the effects of concentration in tests 14 and 16, examination of the raw data gives no indication as to the exact cause of this effect.

In test 13 the average approach ratios for V_1 and V_2 are 0.092 and 0.188, respectively, which indicates that equilibrium is more closely approached at the vapour flow rate than it is at the higher vapour flow rate. This is opposite to that which would be expected if the resistance to the mass transfer in the vapour phase is the rate con-

trolling factor. Table 17 shows that in all other tests the average approach ratio for lower vapour flow rate is approximately equal to or greater than the average approach ratio for the higher vapour flow rate, even though the effect of vapour flow rate was not significant as such in any other test. Again, examination of the raw data does not indicate why vapour flow rate has the effect it does.

Table 16 shows that the liquid flow rate occurs as a significant factor in more tests than any other single factor (i.e. in all tests except 13 and 14) and where it does occur it is the most significant effect in the particular test. The average approach ratios for the two liquid flows have been presented in Figure 19 (page 115), and in all cases the lower liquid flow rate gives the closest approach to equilibrium (i.e. for all tests the approach ratios for the lower liquid flow rates are less than those for the higher liquid flow rates.)

d. Effects of Type of Fluid, Type of Entrance and Hold Up Volume

(1) Tests with High Hold Up and Spray Injection

Water, skim milk, milk (3% fat) and cream (10% fat) were used in tests 12, 13, 14 and 15, respectively. The layer of fluid on the wall was approximately 1 cm thick (hold up volume = 300 ml) and the liquid was injected in the form of a spray using the nozzle shown in Figure 9(a), page 71. The "incorrect" values of approach ratio for

Table 17

AVERAGE APPROACH RATIOS FOR THE TWO VAPOUR FLOW RATES FOR TESTS 12-19

Factor	Test Number								
	12	13	14	15	16	17	18	19	
V ₁	0.1255	0.09186	0.2469	0.1873	0.3256	0.2444	0.2612	0.3505	
V ₂	0.1459	0.1877	0.2115	0.1931	0.1883	0.184	0.1931	0.2487	

points $C_1V_2L_2R_2$ of test 12 and $C_1V_1L_2R_2$ of test 13 were replaced by 0.1958 and 0.1814, respectively. The results of the analysis of variance are shown in Table 18.

The effect of type of fluid (denoted by F in Table 18) has a probability level of 0.001, and the interaction F x C has a probability level of less than 0.001. The average approach ratios for C_1 and C_2 are shown for tests 12 - 15 in Figure 20. At the higher initial concentration (C_2) the effect of fat is to increase the approach ratio, i.e. when a second phase is present, equilibrium is not as closely approached. At the lower initial concentration (C_1) the average approach ratio for test 14 is appreciably greater than any other approach ratio in this set. The reason for this is not apparent from examination of the raw data, but it should be noted that test 14 was unusual in that it is the only run in which concentration occurs with a probability level less than 0.001. It appears that the high value of the average approach ratio for C_1 for test 14 is the source of the interaction effect F x C.

Two other interactions appear in the analysis of variance summarized in Table 18, viz. L x V and V x C, with probability levels of 0.001 and 0.01, respectively. The average approach ratios are shown in Figure 21 and 22 for combinations of L and V, and of V and C, respectively. The effects of V and of C were not expected to be significant,

Table 18

SUMMARY OF ANALYSIS OF VARIANCE ON TESTS
WITH HIGH HOLD UP VOLUME AND
SPRAY INJECTION OF LIQUID

Identifica- tion of Factor	Degrees of Freedom	Sum of Squares	Mean Square	Variance Ratio	Probabil- ity Level
F	3	0.1331	0.0444	9.99	0.001
L	1	0.0986	0.0986	22.20	<0.001
C	1	0.0885	0.0885	19.93	0.001
LxV	1	0.0760	0.0760	4.78	0.001
FxC	3	0.1277	0.0426	17.12	<0.001
VxC	1	0.0295	0.0295	9.59	0.01
Error	83	0.59615	0.007183	6.65	
Total	93	1.1495			

AVERAGE APPROACH RATIOS FOR FIGURE 20

Factor	Test Number			
	12	13	14	15
C_1 (O)	0.156	0.157	0.320	0.184
C_2 (Δ)	0.117	0.128	0.138	0.197

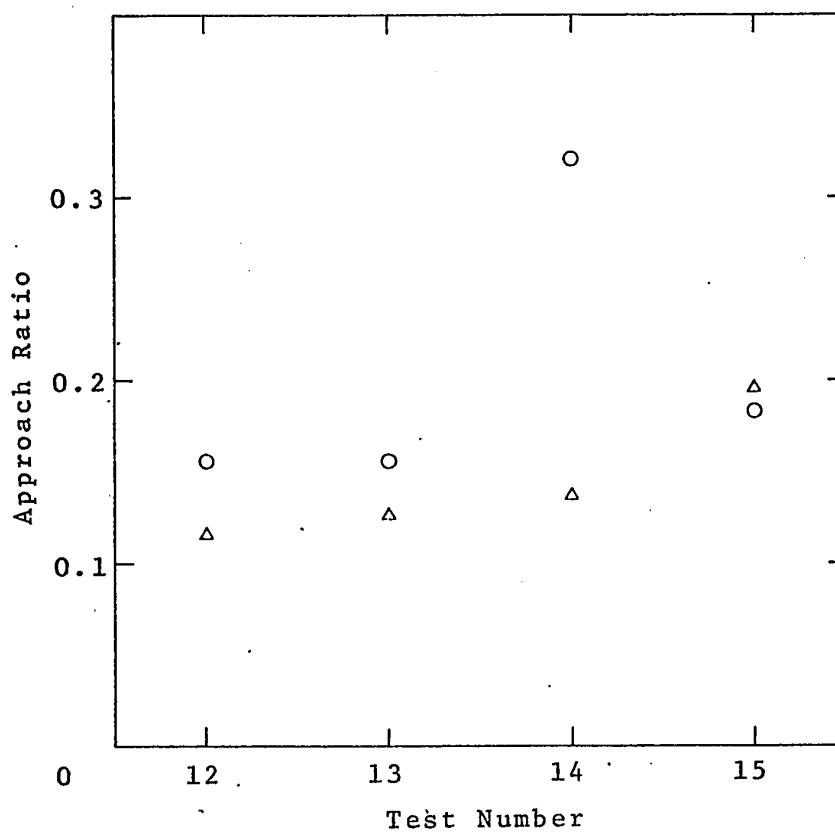


Figure 20

AVERAGE APPROACH RATIOS FOR THE TWO CONCENTRATIONS
FOR TESTS 12-15

either alone or in interactions. It is not clear from Figure 21 and 22 or from the raw data why these effects are significant, and in any case, further tests need to be done to evaluate the magnitudes of these effects.

The effect of liquid flow rate is very significant (probability level less than 0.001). The average approach ratios for the two liquid flows are shown for the eight tests (12 - 19) in Figure 19 (page 115), and it is seen that the lower liquid flow rates for tests 12 - 15 give smaller approach ratios, i.e. equilibrium is more closely approached at the lower liquid flow rates.

Even though the effect of speed of rotation R is not significant, the values of the average approach ratios for 6000 RPM are all less than those for 24,000 RPM for all tests with thick layers of liquid and spray injection (i.e. tests 12 - 15), as shown in Figure 17 (page 112). (It should be noted that the order is reversed for all thin layer tests (i.e. tests 17 - 19)). In tests 14 and 15, in which two liquid phases are present in the thick layer, the fat (i.e. light phase) will form a layer adjacent to the vapour phase, but the fat globules will probably not coalesce because of the very stable nature of the protein membrane around each fat globule. The liquid droplets formed by the spray nozzle will settle on to the top of the fat layer, and the fat globules will remain at or near the

AVERAGE APPROACH RATIOS FOR FIGURE 21

Factor	Test Number	
	L ₁	L ₂
V ₁ (O)	0.161	0.168
V ₂ (Δ)	0.125	0.248

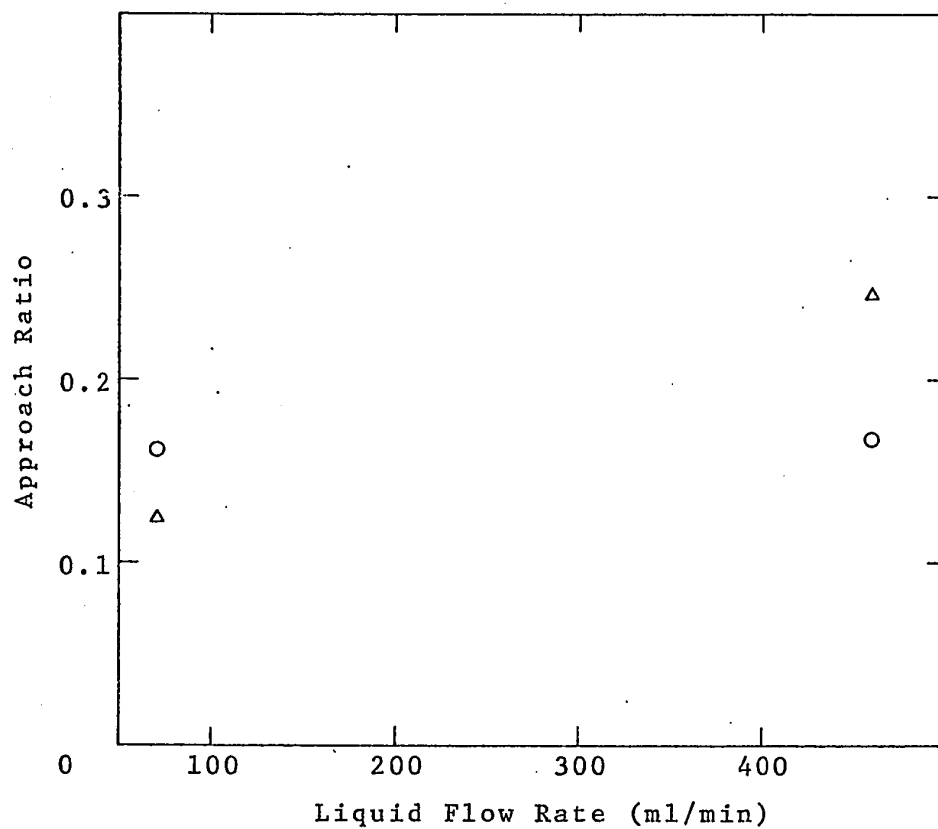


Figure 21

AVERAGE APPROACH RATIO
FOR BOTH LIQUID FLOWS AND VAPOUR FLOWS
FOR TESTS 12-15

AVERAGE APPROACH RATIOS FOR FIGURE 22

Factor	Vapour Flow Rates	
	50 ml/min	100 ml/min
C ₁ (Δ)	0.214	0.199
C ₂ (□)	0.117	0.173

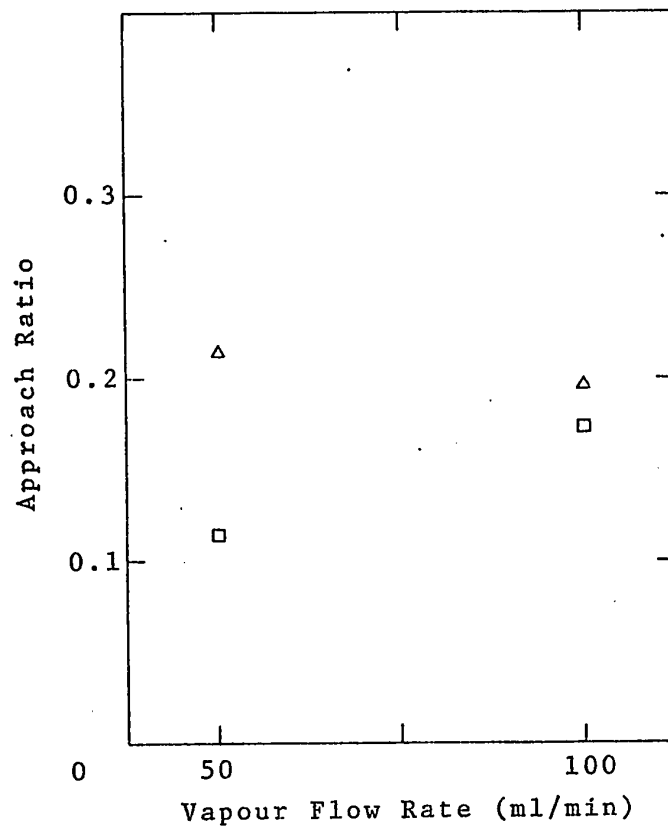


Figure 22

AVERAGE APPROACH RATIOS FOR BOTH VAPOUR
FLOW RATES AND BOTH CONCENTRATIONS
FOR TESTS 12-15

surface, while the aqueous phase will flow through the "packed bed" of fat globules to the underlying aqueous layer. Because the residence time of the droplets in the spray is much less than the 80 - 100 seconds calculated by Scott (1954a) for a 90% approach to equilibrium between the fat and the aqueous phases (page 46 of the present work), the taint which is removed in the spray will be stripped mostly from the aqueous phase. A concentration driving force for mass transfer between the fat globule and the aqueous phase will therefore exist when the droplet settles onto the liquid surface on the wall of the centrifuge bowl, and transfer of taint from the fat globules in the "packed bed" to the aqueous phase will take place as the aqueous phase flows through the "packed bed" of fat globules. The taint compound is moved further away from the steam in the centrifuge bowl, i.e. it is carried to the underlying aqueous layer. The velocity of flow of the aqueous phase through the "packed bed" of fat globules will be greater (and the time for mass transfer will therefore be less) at 24,000 RPM than it is at 6000 RPM.

In most mass transfer systems the mass transfer coefficient k_L is proportional to some power m of velocity, where m is less than 1, and hence the total mass transferred from the fat globules in the "packed bed" to the aqueous layer as it flows through the "packed bed"

will be less at 24,000 RPM than it is at 6000 RPM. This means that the concentration of taint compound in the underlying aqueous phase is greater at the lower speed than it is at the higher speed, and hence any stripping in the spray as the liquid is ejected from the centrifuge to the collecting bowl will be greater for 6000 RPM than it is for 24,000 RPM, as observed for tests 14 and 15.

(2) Tests with Low Hold Up and Liquid Injection Without Spray

Water, skim milk, and milk (3% fat) were used in tests 17, 18 and 19, respectively. There were no "incorrect" values of approach ratio to replace in this set of tests. The results of the analysis of variance of the low hold up (i.e. thin liquid layer) tests are shown in Table 19.

Concentration C does not appear alone as a significant factor although it does occur in two unexpectedly significant interactions $F \times C$ and $V \times L \times C$ (probability levels 0.001). Average approach ratios for combinations of L and C are shown in Figure 23. Concentration does not appear to have much effect at the low liquid flow rate (L_1), but has a different effect for each liquid at the high liquid flow rate (L_2) and hence the effect of $F \times L \times C$ is significant. Figure 24 shows average approach ratios for the two concentrations for the three tests 17, 18 and 19. The approach ratios for the low concentrations are

Table 19

SUMMARY OF ANALYSIS OF VARIANCE FOR THE TESTS
WITH LOW HOLD UP VOLUME AND
LIQUID INJECTION WITHOUT SPRAY

Identifica- tion of Factor	Degrees of Freedom	Sum of Squares	Mean Square	Variance Ratio	Probabil- ity Level
F	2	0.1018	0.0509	7.38	0.001
R	2	0.1269	0.0635	9.20	0.001
L	1	0.7751	0.7751	112.38	<<0.001
V	1	0.1061	0.1061	15.38	0.001
FxC	2	0.1150	0.0575	8.33	0.001
FxLxC	2	0.1237	0.0618	8.97	0.001
Error	61	0.4207	0.006896		
Total	71	1.7692			

AVERAGE APPROACH RATIOS FOR FIGURE 23

131.

Factor	Test Number		
	17	18	19
C ₁ L ₁ (△)	0.138	0.130	0.177
C ₁ L ₂ (□)	0.256	0.311	0.566
C ₂ L ₁ (▲)	0.116	0.129	0.170
C ₂ L ₂ (■)	0.348	0.339	0.286

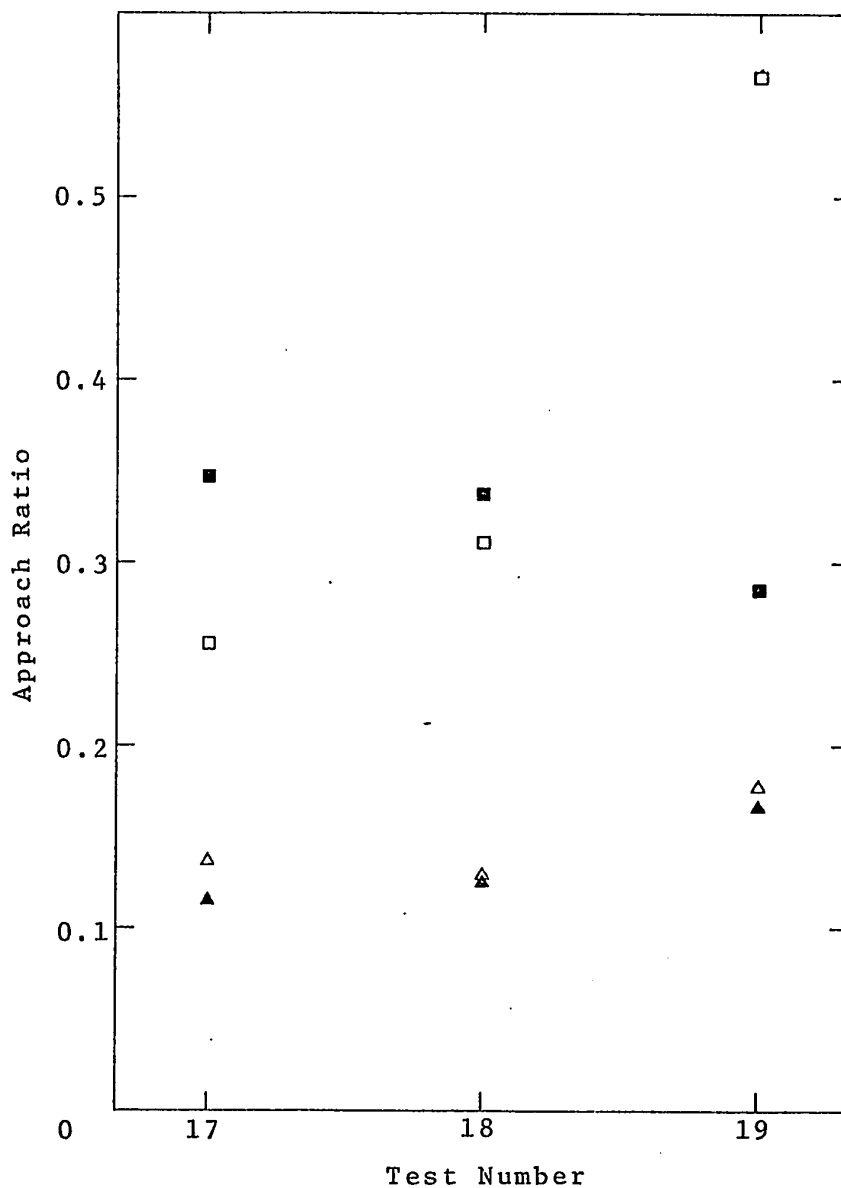


Figure 23

AVERAGE APPROACH RATIOS
FOR TWO CONCENTRATIONS AND
TWO LIQUID FLOW RATES FOR TESTS 17-19

AVERAGE APPROACH RATIOS FOR FIGURE 24

Factor	Test Number		
	17	18	19
C ₁ (□)	0.197	0.220	0.372
C ₂ (△)	0.232	0.234	0.228

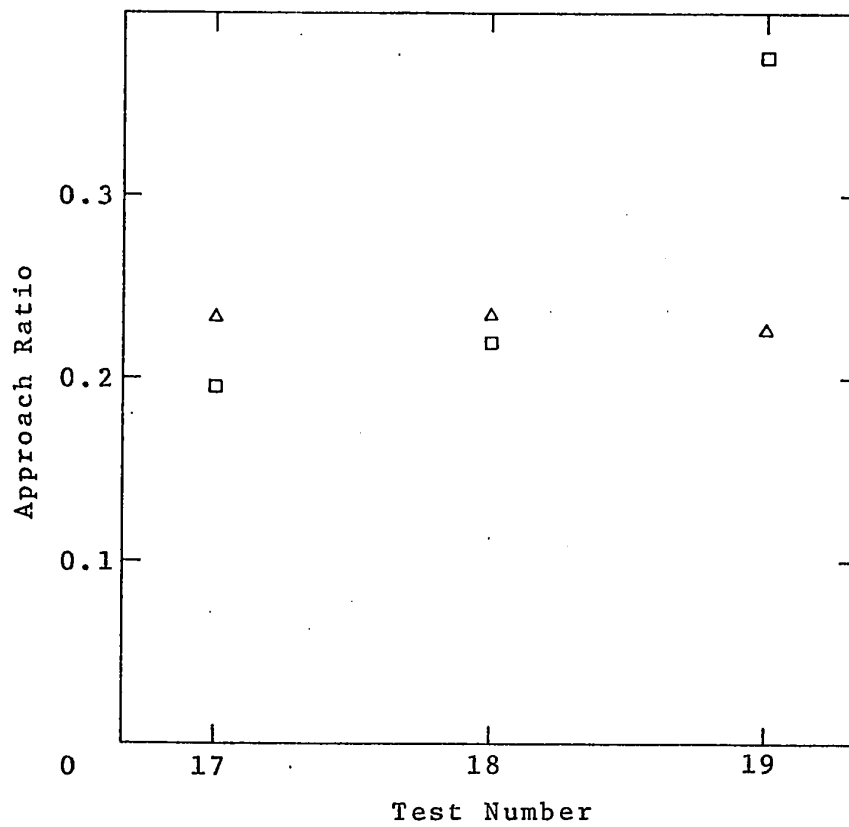


Figure 24

AVERAGE APPROACH RATIOS FOR BOTH
CONCENTRATIONS FOR TESTS 17-19

approximately the same for all three tests, but at the high concentrations, the approach ratio depends on the type of liquid being deodorized, and hence the interaction $F \times C$ is significant. Further work will be needed to determine the magnitudes of these interactive effects, if in fact they are real.

The effects of type of fluid F , speed of rotation R and vapour flow rate V are all significant with probability levels of 0.001, and the effect of liquid flow rate L is very significant, with a probability level of much less than 0.001.

The effect of vapour flow rate on the approach ratio for the three thin film tests is shown in Figure 25. It is seen that equilibrium is more closely approached with higher vapour flow rate V_2 than it is with the lower vapour flow rate V_1 . Higher speeds also give a closer approach to equilibrium than do the lower speeds, as is shown for tests 17 - 19 in Figure 17, page 112. Figure 19, page 115 shows the effect of liquid flow rate on approach ratio, and for all three tests 17, 18 and 19, equilibrium is most closely approached at lower liquid flow rate.

The approach ratios for 17 (water) and 18 (skim milk) are approximately equal (0.214 and 0.227) and for test 19 (3% milk) is 0.300, which indicates that equilibrium is not as closely approached when a fat phase is

AVERAGE APPROACH RATIOS FOR FIGURE 25

Vapour Flow Rate (ml/min)		Test Number		
		17	18	19
50	(Δ)	0.244	0.261	0.351
100	(\square)	0.184	0.193	0.249

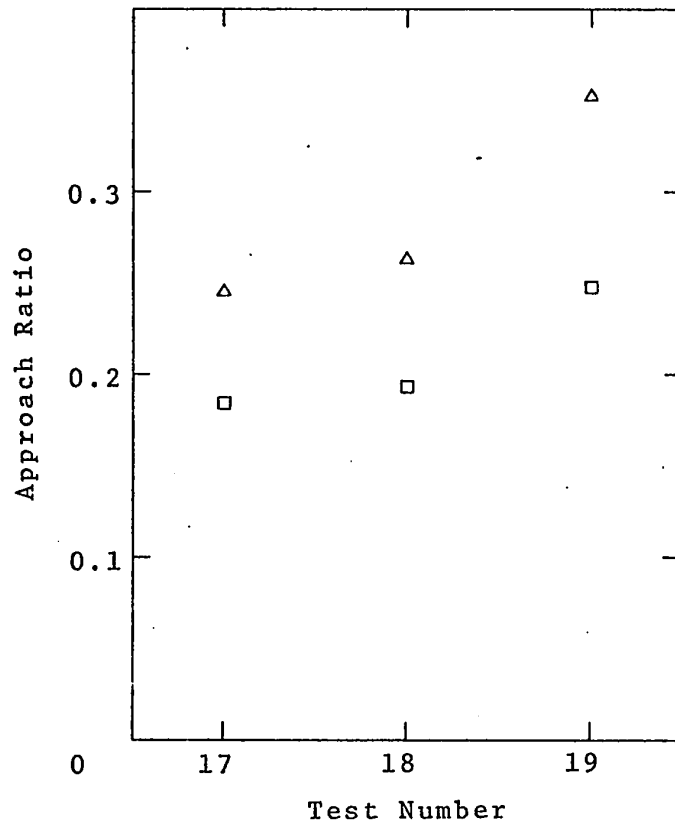


Figure 25

AVERAGE APPROACH RATIOS
FOR BOTH VAPOUR FLOW RATES
FOR TESTS 17-19

present.

The Reynolds number for fluid flow in a thin film is $Re = 4\Gamma/\mu$ (Perry 1963, p. 5-55), where Γ is the mass flow rate per unit width of film and μ is the viscosity of the liquid. The film Reynolds numbers based on fluid flow up the wall of centrifuge bowl are approximately 100 and 700 for the low and high liquid flow rates, respectively. The flow would therefore appear to be in the wavy laminar flow regime.

The thickness of the fluid layer has not been determined, but calculations based on the minimum hold up volumes (page 75) indicate that the fluid layers on the wall are much less than 1 mm thick, even at 6000 RPM. The velocity gradient in the thin fluid layer is increased by a decrease in thickness, and the velocity gradient may be sufficient to impart a significant spin to the fat globules with a resulting reduction in the resistance to mass transfer through the boundary layer surrounding the fat globule.

The Reynolds numbers calculated above are based on the flow up the wall, with no account taken of entrance effects. The fluid entering the centrifuge in a steady coherent stream through the nozzle shown in Figure 9(b), p. 71, must be accelerated to the angular velocity of the bowl, and hence there will be a tangential velocity with

respect to the bowl. It is also likely that the thickness of the layer will be increased near the entrance, and this increase of thickness along with the tangential component of velocity could well give film Reynolds numbers in the turbulent flow regime, i.e. Reynolds numbers greater than 1000 to 2000.

Turbulence in the liquid film will increase the overall mass transfer rates by decreasing the resistance to mass transfer in the liquid boundary layer at the liquid vapour interface. It is expected that the turbulence will be greater with the higher liquid flow rate, and greater mass transfer rates would partially offset the effect of reduced residence time at higher liquid flow rate.

In the case when a second (i.e. fat) phase is present, the turbulence will tend to prevent the globules from being spun out of the aqueous phase. The rate of mass transfer from the fat globules to the surrounding aqueous phase, will therefore be increased as per the equations of Calderbank and Moo-Young (1961), and of Gal-or and Hoelscher (1966), and this will lead to an increase in the overall mass transfer rate. The overall mass transfer rate for a two phase system will still be somewhat less than that for a single phase liquid.

The residence time decreases as the speed of the bowl increases (as shown in Table 2, page 76). Entrance

effects, however, may increase the residence times and reduce the effect of speed on residence time so that the effect of speed on mass transfer more than offsets the effect of residence time on total mass transfer.

The vapour entering the centrifuge bowl is directed up the axis at high velocity and recirculates down the surface of the liquid layer on the wall. It must also acquire an angular velocity by momentum transfer from the spinning liquid layer. The higher vapour flows will give lower vapour phase mass transfer resistance, because of the greater vertical recirculation velocities and because of the greater rate of transfer of momentum at the vapour liquid interface, and this could lead to greater overall mass transfer rates with the higher flow rates if the mass transfer resistance in the liquid phase have been reduced sufficiently by the turbulence and other effects.

This analysis of the mass transfer processes in the thin film centrifugal deodorizer is consistent with the observed effects of type of fluid, speed of rotation and liquid and vapour flow rates. It is clear, however, that the data of the present work does not permit the relative significance of all of the interrelated factors to be determined and further work will be required to do this and to determine the magnitudes of these effects.

(3) High Hold Up Tests with Water, with and without Spray

In tests 12 and 16 water was deodorized in the centrifuge using the large hold up volume. The liquid injection system in test 12 was the spray nozzle shown in Figure 9a, page 71, while the injection of the liquid in test 16 was through the "no spray" nozzle, shown in Figure 9b. An analysis of variance was done on these two tests to determine the effect of spray, and the results of this analysis are shown in Table 20.

Liquid flow rate again appears as the most significant effect (probability level 0.001), and the unexplained effect of concentration also appears, with a probability level of 0.005.

The effect of test (T in Table 20) is significant at the probability level 0.005 which indicates that spray has a significant effect. The average approach ratios for 12 and 16 are 0.135 and 0.251, respectively, which shows that equilibrium is more closely approached when spray is used than when it is not. This is as expected because the surface area for mass transfer in the spray is very much greater than that of the surface of the liquid vapour interface on the wall of the centrifuge.

(4) Tests Using Water Without Spray, with High and Low Hold Up Volume

The inlet nozzle used in tests 16 and 17 was the

Table 20

SUMMARY OF ANALYSIS OF VARIANCE ON TESTS 12 AND 16

Identifica- tion of Factor	Degrees of Freedom	Sum of Squares	Mean Square	Variance Ratio	Probabil- ity Level
T	1	0.1283	0.1283	9.78	0.005
L	1	0.2062	0.2062	15.72	0.001
C	1	0.1110	0.1110	8.46	0.005
Error	41	0.5376	0.01311		
Total	45	0.9830			

no spray nozzle shown in Figure 9b, page 71, and the water was deodorized in a thick layer (i.e. high hold up volume) in test 16, and in a thin layer (i.e. low hold up volume) in test 17. Comparison of these two tests allows the effect of hold up volume to be evaluated when spray is not used.

The results of the analysis of variance of tests 16 and 17 taken together are shown in Table 21. Again the effect of liquid flow is most significant (probability level much less than 0.001), and the effects of vapour V and of the interaction of test and concentration TxC are both significant at the 0.005 probability level. The interaction TxC probably arises from the unexplained effect of concentration in test 16 (Table 16, page 118), and the factor V arises because the approach ratios for the low vapour flow rate V_1 in both tests are greater than those for the higher vapour flow rate V_2 . The effect of test T is not significant on its own, which shows that the overall amount of mass transfer is nearly the same for thick and thin films, as is shown by the overall average approach ratios (0.251 for test 16 and 0.214 for test 17). The residence times in test 17 are an order of magnitude less than those for test 16. Hence the overall mass transfer coefficient for the thin layer of test 17 must be about one order of magnitude greater than that for the thick layer of test 16, as the areas of the vapour liquid interfaces

Table 21

SUMMARY OF ANALYSIS OF VARIANCE ON TESTS 16 AND 17

Identifica- tion of Factor	Degrees of Freedom	Sum of Squares	Mean Square	Variance Ratio	Probabil- ity Level
L	1	0.3608	0.3608	35.78	<<0.001
V	1	0.0882	0.0882	8.75	0.005
TxC	1	0.0971	0.0971	9.63	0.005
Error	42	0.4235	0.01008		
Total	45	0.9697			

are of the same order of magnitude (220 cm² for test 16 and 360 cm² for test 17).

The main mass transfer resistance in the liquid phase is in the boundary layer at the vapour liquid interface. This comparison of thick and thin layer tests with a single phase liquid feed therefore shows that the conditions in the liquid boundary layer are very strongly affected by the thickness of the layer. No data from the present study are available which will allow a similar comparison for a two phase liquid system (i.e. comparison of thick and thin of milk and/or cream without spray injection). In view of the large apparent increase in liquid boundary mass transfer coefficient, this is an important area in which further work could be done, so that the efficiency with which steam is used in deodorization can be increased.

VI. ANALYSIS OF RECYCLING AND MULTISTAGE SYSTEMS

Often it is not possible to get satisfactory deodorization in a single stage deodorizer with a reasonable amount of steam, and hence various arrangements of single stages have been tried. In cases where the taints are at very high initial concentrations or are particularly difficult to remove, the cream or milk has been passed through the same equipment several times, with an increase in the total amount of steam used.

Some of the systems of multiple stages do not appear to be very rationally designed from the point of efficient use of steam. One example is the system described by Scott (1957) in which a tandem Vacreator (two counter-current stages) is used with another Vacreator with separate steam supply to "pretreat" the feed liquid before deodorization in the tandem Vacreator.

Further, McDowall (1958) states that "more efficient" use of steam is obtained by deodorizing a fluid of high fat content than one of low fat content, and hence a system in which a fat enriched stream is recycled and mixed with the feed liquid has been analyzed.

MacDonald (1927) and MacDowall (1955a) have suggested that the cream can be washed with taint free skim milk to remove taint from the fat phase. This can

be useful if a sufficient quantity of taint free skim milk is available. If sufficient quantities of taint free skim milk are not available (as is often the case when taints are a problem) then tainted skim milk can be deodorized and used to "wash" the cream. This system is included in the analysis of recycling system mentioned above.

As discussed in the Review of the Literature (Section II.E. in pages 34 and 35), there are three methods of combining deodorizing stages, viz. stepwise flash, cross flow and countercurrent. An analysis of these systems is presented in section B. below.

In the analyses of the recycling systems and of the multistage systems, several assumptions have been made, as follows:

1. Deodorizing stages are available, or can be designed so that equilibrium between the exit streams can be closely approached for any combination of steam flow, fat content and type of taint.

2. The steam to any stage is dry and saturated, and the liquid feed is at the same temperature as the steam.

3. There are no heat losses from the equipment, i.e. there is no condensation of steam and subsequent dilution of the liquid stream in any contact stage.

4. The heat of vaporization of the taint compounds is negligible because the taints are present in very small

concentrations.

5. The temperature of the cream and milk leaving the system is the same as that at which the feed enter, i.e. there is no net dilution or concentration of the product.

6. The equilibrium coefficients k and D as defined by equations II.1 (page 40) and II.2 (page 41), respectively, are independent of temperature.

A. RECYCLING SYSTEMS

The flow diagram for the generalized recycling system is presented as Figure 26. In Figure 26 (and in the analysis that follows), "steam" refers to the vapour phase introduced to a deodorizing stage (usually free of taint) and "vapour" refers to the tainted vapour phase from any deodorizing stage.

The milk or cream is fed to the feed stream deodorizer, and passes from this to a centrifuge. One stream from the centrifuge is taken off as product stream (stream 4), while the other (stream 5) is recycled. It may be deodorized in the recycle stream deodorizer. Stream 10 (the output from the recycle stream deodorizer) can be completely recycled (i.e. stream 11 is zero) or it can be split into two streams, one of which is recycled and mixed with the liquid feed, with the other stream being taken off

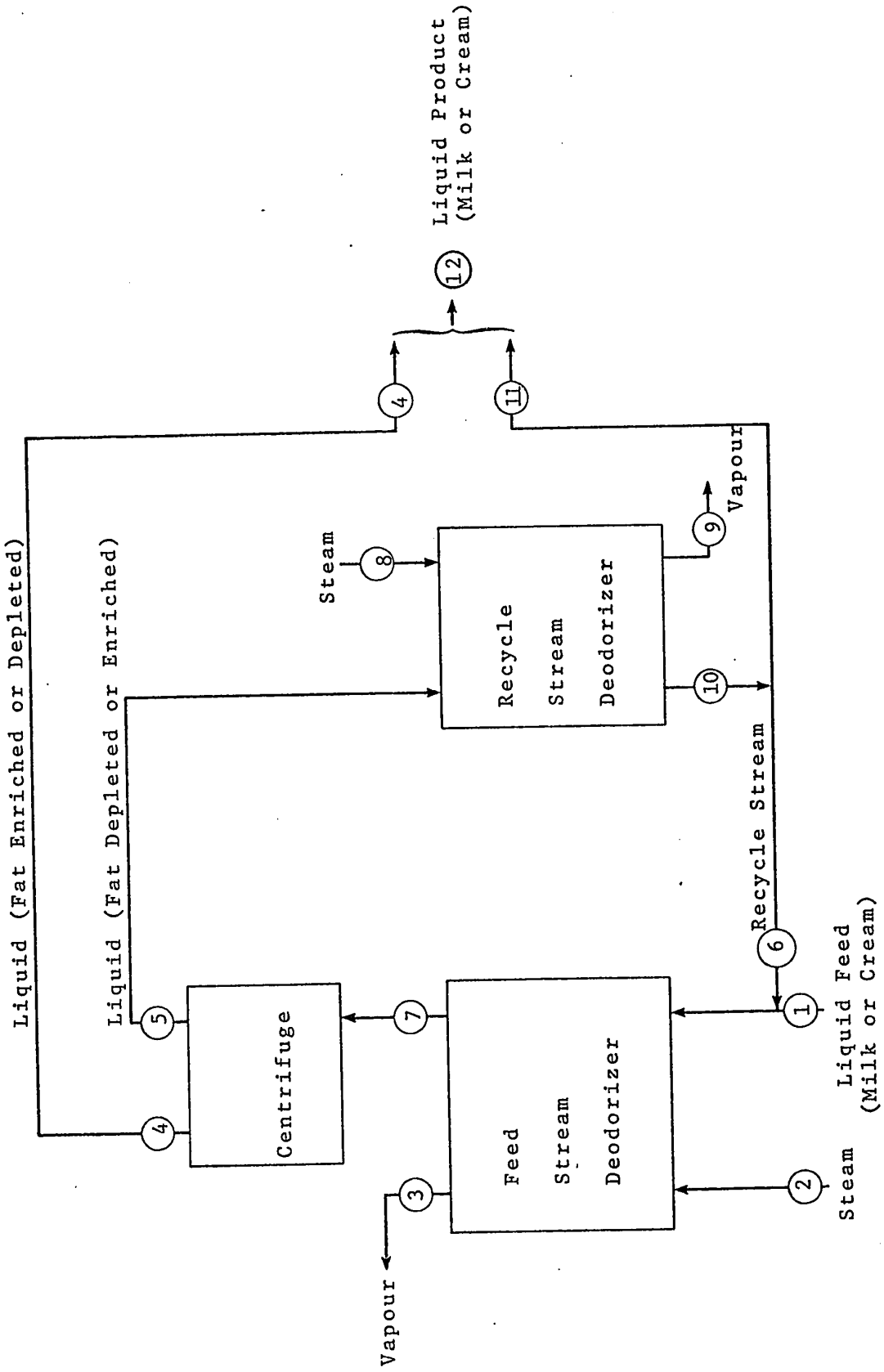


Figure 26

FLOW DIAGRAM FOR RECYCLING SYSTEMS

as part of the total product stream (i.e. stream 12).

The material balances for the taint compound for the whole system, the feed stream deodorizer and the centrifuge, and the recycle stream deodorizer are, respectively,

$$L_1x_1 + S_2s_2 + S_8s_8 = L_{12}x_{12} + V_3y_3 + V_9y_9 \quad (\text{VI.1})$$

$$L_1x_1 + S_2s_2 + L_6x_6 = V_3y_3 + L_4x_4 + L_5x_5 \quad (\text{VI.2})$$

and

$$L_5x_5 + S_8s_8 = L_{10}x_{10} + V_9y_9 \quad (\text{VI.3})$$

where L, S and V refer to mass flow rates, and x, s and y refer to the taint concentrations of the liquid, steam and vapour streams, respectively. The numerical subscripts identify the various streams (as shown in Figure 26).

An equation for the taint reduction ratio (x_{12}/x_1) of the general recycling system can be obtained from equation VI.1, if y_3 and y_9 can be expressed in terms of the relevant mass flow rates and the input taint concentrations x_1 , S_2 and S_i .

Equation II.1 (page 40) can be used to express the vapour concentrations y_3 and y_9 in terms of the vapour phase aqueous phase equilibrium coefficient k and the concentrations of taint in the aqueous phases of the

streams leaving the respective deodorizing stages; i.e.

$$y_3 = kp_7 \quad (\text{VI.4})$$

and

$$y_9 = kp_{10} \quad (\text{VI.5})$$

where p_n is the concentration of taint in the aqueous phase of stream n . The concentration of taint in each phase in streams 4 and 5 are equal to those in stream 7, because no mass transfer between the phases takes place in the centrifuge, i.e

$$p_4 = p_5 = p_7 \quad (\text{VI.6})$$

and

$$f_4 = f_5 = f_7 \quad (\text{VI.7})$$

where f_n is the concentration of taint in the fat phase of stream n .

The simple splitting of stream 10 into streams 6 and 11 gives

$$p_6 = p_{10} = p_{11} \quad (\text{VI.8})$$

and

$$f_6 = f_{10} = f_{11} \quad (\text{VI.9})$$

The taint concentration x_n in stream n is given by

$$L_n x_n = F_n f_n + P_n p_n \quad (\text{VI.10})$$

where F_n and P_n are the mass flow rates of fat phase and aqueous phase in stream n . Substitution of equation II.2, page 41, in equation VI.8, leads to the equation

$$x_n = A_n p_n$$

where

$$A_n = 1 + (F_n/L_n)(D-1) \quad (\text{VI.11})$$

Material balances around the recycle stream deodorizer and the splitting of stream 10 give

$$F_5 = F_{10} \quad (\text{VI.12})$$

$$L_5 = L_{10} \quad (\text{VI.13})$$

$$F_5/L_5 = F_6/L_6 = F_{10}/F_{10} = F_{11}/L_{11} \quad (\text{VI.14})$$

$$A_5 = A_6 = A_{10} = A_{11} \quad (\text{VI.15})$$

Substitution of equations VI.4, VI.6 and VI.11 into

equation VI.2, and equations VI.5, VI.8 and VI.11 into equation VI.3, leads to

$$L_1 x_1 + S_2 s_2 + L_6 A_6 p_6 = (V_3 k + L_4 A_4 + L_5 A_5) p_5 \quad (\text{VI.16})$$

and

$$L_5 A_5 p_5 + S_8 s_8 = (L_{10} A_{10} + V_9 k) p_6 \quad (\text{VI.17})$$

respectively.

Solving equations VI.16 and VI.17 for p_5 and p_6 gives the equations

$$p_5 = \frac{(L_5 A_5 + V_9 k)(L_1 x_1 + S_2 s_2)}{\Delta} \quad (\text{VI.18})$$

$$+ \frac{((L_5 A_5 + V_9 k)(V_3 k + L_6 A_6 + L_1 A_1) - \Delta) S_8 s_8}{L_5 A_5 \Delta}$$

and

$$p_6 = \frac{L_5 A_5 (L_1 x_1 + S_2 s_2) + (V_3 k + L_6 A_6 + L_1 A_1) S_8 s_8}{\Delta} \quad (\text{VI.19})$$

where

$$\Delta = (V_3 k + L_1 A_1)(L_5 A_5 + V_9 k) + L_6 A_6 V_9 k \quad (\text{VI.20})$$

Substitution of equations VI.4, VI.5, VI.6, VI.8 and VI.11 into equation VI.1 gives

$$L_1 x_1 + S_2 s_2 + S_8 s_8 = L_{12} x_{12} + V_3 k p_5 + V_9 k p_6$$

and then substitution of VI.15, VI.18 and VI.19 into this equation leads to the equation for the taint reduction ratio, i.e.

$$\frac{x_{12}}{x_1} = \frac{(L_5 A_5 (L_1 A_1 - V_9 k) + V_9 k (L_1 A_1 + L_6 A_6)) (L_1 x_1 + S_2 s_2)}{\Delta L_1 x_1} \quad (\text{VI.21})$$

$$+ \frac{(L_5 A_5 (V_3 k + L_1 A_1) - L_6 A_6 V_3 k) S_8 s_8}{\Delta L_1 x_1}$$

Normally the stripping steam will be free of taint (i.e. $S_2 = S_8 = 0$) and the taint reduction ratio x_{12}/x_1 is given by

$$\frac{x_{12}}{x_1} = \frac{L_1 A_1 L_5 A_5 + L_1 A_1 V_9 k - L_5 A_5 V_9 k + L_6 A_6 V_9 k}{L_1 A_1 L_5 A_5 + L_1 A_1 V_9 k + L_5 A_5 V_3 k + L_6 A_6 V_9 k + V_3 k V_9 k} \quad (\text{VI.22})$$

In a recycling system the quantities which can be varied are L_5 , L_6 , A_5 , A_6 , V_3 and V_9 . The values of these operating variables which give the greatest taint reduction (i.e. smallest value of x_{12}/x_1) can be determined from a consideration of equation VI.21, as follows.

The recycle flow rate L_6 which gives the optimum taint reduction ratio determined by differentiating equation

VI.22 with respect to L_6 , i.e.

$$\frac{d}{dL_6} \left(\frac{x_{12}}{x_1} \right) = \frac{A_5 V_9 k (L_5 A_5 V_3 k + L_5 A_5 V_9 k + V_3 k V_9 k)}{\Delta^2} \quad (\text{VI.23})$$

where Δ is defined by equation VI.20 . Equation VI.23 shows that $d(x_{12}/x_1)/dL_6$ is greater than or equal to zero for all values of the operating variables, and hence the optimum value of the recycle flow rate L_6 will be when L_6 has its minimum value, i.e. when $L_6 = 0$.

The fact that the minimum value of the taint reduction ratio occurs when the recycle stream flow rate L_6 is zero shows that McDowall's observation (1958) that the "most efficient removal of taint is obtained with a high fat content cream" cannot be applied to systems in which a fat enriched stream is recycled to increase the fat content in the deodorizing stage. It further shows that if skim milk has to be stripped of taint before it is used to "wash" milk or cream, then the total amount of steam used is equal to or greater than that for the same overall reduction of taint in an equivalent non recycling system.

The optimum values of the remaining operating variables (i.e. L_5 , A_5 , V_3 and V_9) can be obtained in a similar way to the optimum value of L_6 .

The optimum value of L_5 can be obtained by putting

$L_6 = 0$ in equation VI.22 and differentiating with respect to L_5 . This gives

$$\frac{d}{dL_5} \left(\frac{x_{12}}{x_1} \right) = \frac{-A_5(L_1 A_1 V_9^2 k^2 + V_3 k V_9^2 k^2)}{\Delta^2}$$

where Δ is defined by equation VI.20 with $L_6 = 0$.

The minimum value of the taint reduction ratio occurs when L_5 has its maximum value, that is when $L_5 = L_1$ (and $L_4 = 0$), because $d(x_{12}/x_1)/dL_6$ is less than or equal to zero for all values of the operating variables.

When $L_6 = 0$, $A_5 = A_1$ because the fat contents (F_1/L_1 and F_5/L_5 of streams 5 and 1, respectively) are equal.

The equation for taint reduction then gives

$$\frac{x_{12}}{x_1} = \frac{L_1^2 A_1^2}{(L_1 A_1 + V_3 k)(L_1 A_1 + V_9 k)} \quad (\text{VI.24})$$

and the system is a simple cross flow system with two stages.

The optimum value of vapour flows V_3 and V_9 is determined by putting $V_9 = S_T - V_3$ in equation VI.24, where S_T is the total steam flow. The resulting equation is then differentiated with respect to V_3 to give

$$\frac{d}{dV_3} \left(\frac{x_{12}}{x_1} \right) = \frac{-L_1^2 A_1^2 k^2 (S_T - 2V_3)}{(L_1^2 A_1^2 + L_1 A_1 S_T k + S_T k V_9 k - V_9^2 k^2)^2}$$

The minimum value of x_{12}/x_1 occurs when $S_T = 2V_3$ i.e. when $V_3 = V_9 = S_T/2$, provided, of course, that the values of k and D are not temperature dependent.

B. MULTISTAGE DEODORIZING SYSTEMS

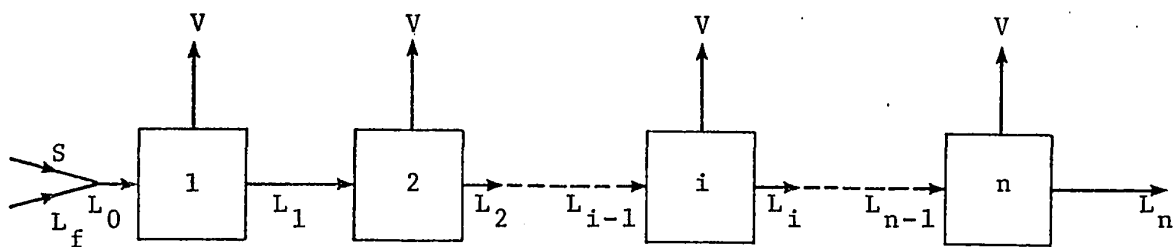
i. Development of the Equations

The flow diagrams for stepwise flash, crossflow and countercurrent systems of deodorizing stages are shown in Figure 27. The general equations for n stages of each of the three systems are developed as follows.

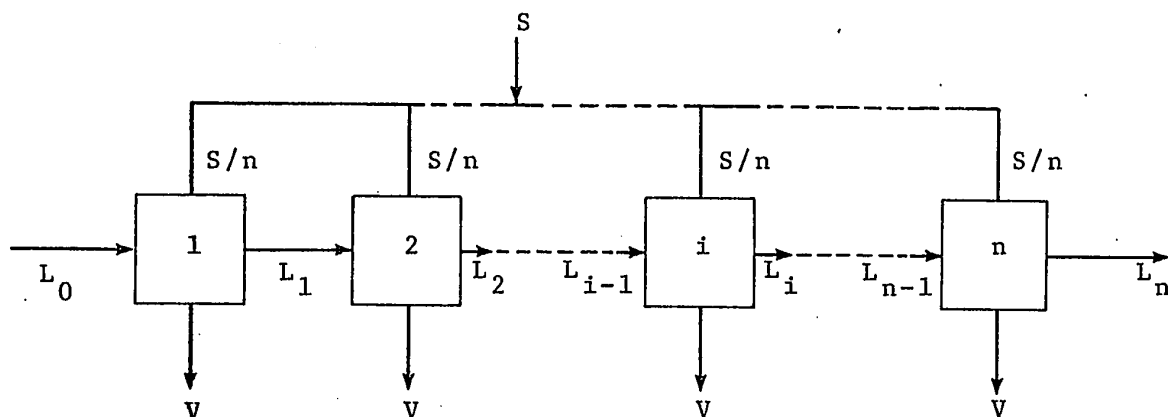
a. Stepwise Flash System

In the stepwise flash system (Figure 27a), the steam is injected into the liquid and condensed under high pressure, and the vapour is flashed off as the liquid flows through a series of stages of decreasing pressure. In this analysis it is assumed the pressures in the various stages are controlled so that equal amounts of vapour are removed from each stage, i.e. $V = s/n$.

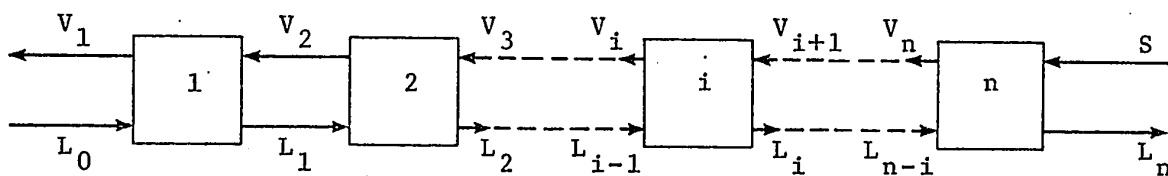
For the i^{th} stage the material balances and equilibrium data give



a. Stepwise Flash System



b. Crossflow System



c. Countercurrent System

Figure 27

MULTISTAGE DEODORIZING SYSTEMS

$$\frac{L_i x_i}{L_{i-1} x_{i-1}} = \frac{L_i A_i}{(S/n)k + L_i A_i} \quad (\text{VI.25})$$

The fat flow rates through all stages are equal,
i.e.

$$F_0 = F_1 = F_2 = \dots = F_i = \dots = F_n = F_f$$

where subscript f refers to the feed stream.

The term A_i is therefore given by

$$A_i = 1 + (F_f/L_i)(D-1)$$

whence

$$L_i A_i = L_i + F_f(D-1) \quad (\text{VI.26})$$

The liquid flow from the i^{th} stage L_i is given by overall material balance over stage $(i+1)$ to n , i.e.

$$\begin{aligned} L_i &= L_n + (n-i)(S/n) \\ &= L_f + (n-i)(S/n) \end{aligned} \quad (\text{VI.27})$$

because $L_f = L_n$.

Equations VI.26 and VI.27 give that

$$\begin{aligned}
 L_i A_i &= L_f + F_f(D-1) + (n-i)(S/n) \\
 &= L_f A_f + (n-i)(S/n)
 \end{aligned}$$

and hence VI.25 becomes

$$\frac{L_i x_i}{L_{i-1} x_{i-1}} = \frac{nL_f A_f + (n-i)S}{Sk + nL_f A_f + (n-i)S}$$

The taint reduction ratio x_n/x_f is equal to $L_n x_n/L_f x_f$ because $L_n = L_f$, and the ratio $L_n x_n/L_f x_f$ is equal to $L_n x_n/L_0 x_0$ because the mass flow of taint to the steam injection nozzle $L_f x_f$ is equal to the mass flow of taint to stage 1 ($L_0 x_0$). The ratio $L_n x_n/L_0 x_0$ is given by the product

$$\frac{L_1 x_1}{L_0 x_0} \cdot \frac{L_2 x_2}{L_1 x_1} \cdot \dots \cdot \frac{L_n x_n}{L_{n-1} x_{n-1}}$$

and hence the taint reduction ratio x_n/x_f for stepwise flash deodorization is given by the equation

$$\frac{x_n}{x_f} = \prod_{i=1}^n \left[\frac{nL_f A_f + (n-i)S}{Sk + nL_f A_f + (n-i)S} \right] \quad (\text{VI.28})$$

b. Crossflow System

In the crossflow system shown in Figure 27b, the steam is divided equally between the n stages.

For the i^{th} stage the material balances and the equilibrium equations give that

$$\frac{x_i}{x_{i-1}} = \frac{nL_i A_i}{nL_i A_i + Sk}$$

Because $L_0 = L_i = L_1 = L_2 = \dots = L_n$, and $A_0 = A_1 = A_2 = \dots = A_n$, the overall taint reduction ratio x_n/x_0 for crossflow deodorization is given by

$$\frac{x_n}{x_0} = \left[\frac{nL_0 A_0}{nL_0 A_0 + Sk} \right]^n \quad (\text{VI.29})$$

c. Countercurrent System

The mass balances and equilibrium equations for all stages of a countercurrent deodorizing system, as shown in Figure 27c, except the n^{th} give

$$n_{i-1} = (1+B)x_i - Bx_{i+1} \quad (\text{XI.30})$$

where

$$B = Sk/L_0 A_0$$

For stage n

$$x_{n-1} = (1+B)x_n$$

and for stage $n-1$ equation VI.30 gives

$$x_{n-2} = (1+B)x_{n-1} - Bx_n$$

which simplifies to

$$x_{n-2} = (1 + B + B^2)x_n$$

Similarly, application of equation VI.30 over all n stages leads to the equation

$$x_1 = (1 + B + B^2 + \dots + B^{n-1})x_n,$$

and the general equation for the taint reduction ratio x_n/x_0 for countercurrent flow is

$$\begin{aligned} \frac{x_n}{x_0} &= \frac{1}{\sum_{i=0}^{n-1} (B^i)} \\ &= \frac{1}{\sum_{i=0}^{n-1} \left(\frac{Sk}{L_0 A_0} \right)^i} \end{aligned} \quad (\text{VI.31})$$

ii. Taint Reduction Ratios for Diacetyl, Acetoin and Benzyl Mercaptan for Multistage Deodorizing Systems

Taint reduction ratios for a range of steam flows and fat contents have been calculated for stepwise flash, crossflow and countercurrent systems of 1, 2, ... , 10 stages for diacetyl, the reference compound used in the experimental section of this study. Similar sets of taint reduction ratios have also been calculated for acetoin (which is much more soluble in the aqueous phase than diacetyl) and for benzyl mercaptan (which is much more soluble in the fat phase than diacetyl). The values of the equilibrium coefficients k and D (as defined by equations II.1, page 40, and II.2, page 41, respectively) are 28.4 and 1.33 for diacetyl, 1.29 and 0.21 for acetoin, and 73 and 160 for benzyl mercaptan, respectively. No attempt has been made in these calculations to take account of any variation of k and D with temperature which may occur, as these will depend on the way in which any particular system is operated.

The steam flow rates have been expressed as ratios of steam to feed flow rates. The steam flow ratios range from 0.1 to 1.0 lb of steam per lb of milk or cream. Fat contents of 2%, 3%, 4%, 5%, 10%, 20% and 30% have been used to cover the range of fat contents commonly occurring in milk and cream deodorization.

The FORTRAN IV program used to calculate the

taint reduction ratios is presented in Appendix G.

The taint reduction ratios for diacetyl for the 10 steam flows for stepwise flash, crossflow and counter-current systems of up to 10 stages are shown in Tables 22 to 28 for the fat contents of 2%, 3%, 4%, 5%, 10%, 20% and 30%, respectively. The corresponding taint reduction ratios for acetoin and benzyl mercaptan are presented in Tables 29 to 35, and 36 to 42, respectively.

a. Comparison of Multistage Deodorizing Systems

Comparison of the corresponding data for the stepwise flash, crossflow and countercurrent systems in Tables 22 to 42 shows that the countercurrent system gives the greatest taint reduction for all systems of two or more stages. These effects are also shown in Figure 28 to 30, and Figures 31 to 33 in which the taint reduction ratios are plotted against vapour flow rate and number of stages, respectively, for the three compounds and fat contents of 3% and 30%.

The tables and the graphs also show that the crossflow system is only slightly better than the stepwise flash system. The greatest difference between these two systems occurs with acetoin. This is shown particularly clearly in Figure 29. The larger difference occurs because D for acetoin is much less than D for diacetyl and benzyl

TABLE 29

TAINT CONCENTRATION REDUCTION RATIOS FOR ACETICIN

K = 1.29 ; D = 0.21

FAT CONTENT OF FLUID = 2.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.884	0.792	0.718	0.656	0.604	0.560	0.522	0.488	0.459	0.433
	2	0.883	0.790	0.714	0.651	0.597	0.552	0.512	0.478	0.448	0.421
	3	0.883	0.789	0.712	0.649	0.595	0.548	0.509	0.474	0.443	0.416
	4	0.883	0.789	0.712	0.647	0.593	0.547	0.507	0.472	0.441	0.414
	5	0.883	0.789	0.711	0.647	0.592	0.546	0.505	0.470	0.439	0.412
	6	0.883	0.788	0.711	0.646	0.592	0.545	0.504	0.469	0.438	0.411
	7	0.883	0.788	0.711	0.646	0.591	0.544	0.504	0.469	0.437	0.410
	8	0.883	0.788	0.711	0.646	0.591	0.544	0.503	0.468	0.437	0.409
	9	0.883	0.788	0.711	0.646	0.591	0.544	0.503	0.468	0.436	0.409
	10	0.883	0.788	0.710	0.645	0.590	0.543	0.503	0.467	0.436	0.408
CROSS FLOW	1	0.884	0.792	0.718	0.656	0.604	0.560	0.522	0.488	0.459	0.433
	2	0.881	0.782	0.698	0.628	0.567	0.515	0.470	0.430	0.396	0.365
	3	0.880	0.778	0.691	0.617	0.553	0.497	0.449	0.407	0.370	0.337
	4	0.879	0.776	0.687	0.611	0.545	0.488	0.438	0.394	0.356	0.322
	5	0.879	0.775	0.685	0.607	0.540	0.482	0.431	0.386	0.347	0.312
	6	0.878	0.774	0.683	0.605	0.537	0.478	0.426	0.380	0.341	0.306
	7	0.878	0.773	0.682	0.603	0.534	0.475	0.422	0.376	0.336	0.301
	8	0.878	0.773	0.681	0.602	0.533	0.472	0.420	0.373	0.333	0.297
	9	0.878	0.772	0.681	0.601	0.531	0.471	0.417	0.371	0.330	0.294
	10	0.878	0.772	0.680	0.600	0.530	0.469	0.416	0.369	0.328	0.292
COUNTER CURRENT	1	0.884	0.792	0.718	0.656	0.604	0.560	0.522	0.488	0.459	0.433
	2	0.871	0.751	0.646	0.556	0.480	0.416	0.362	0.318	0.280	0.248
	3	0.869	0.741	0.622	0.515	0.423	0.346	0.283	0.233	0.192	0.159
	4	0.869	0.739	0.613	0.495	0.392	0.305	0.236	0.181	0.140	0.108
	5	0.865	0.738	0.609	0.486	0.374	0.280	0.204	0.148	0.106	0.076
	6	0.865	0.738	0.608	0.481	0.364	0.262	0.182	0.123	0.082	0.055
	7	0.869	0.738	0.607	0.478	0.357	0.250	0.166	0.105	0.065	0.040
	8	0.869	0.738	0.607	0.477	0.353	0.241	0.153	0.091	0.052	0.030
	9	0.869	0.738	0.607	0.476	0.350	0.235	0.143	0.080	0.043	0.022
	10	0.869	0.738	0.607	0.476	0.348	0.230	0.135	0.071	0.035	0.017

TABLE 30

TAINI CONCENTRATION REDUCTION RATIOS FOR ACETIC ACID

K = 1.29 ; D = 0.21

FAT CONTENT OF FLUID = 3.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.883	0.791	0.716	0.654	0.602	0.558	0.520	0.486	0.457	0.431
	2	0.883	0.789	0.712	0.649	0.595	0.550	0.510	0.476	0.446	0.419
	3	0.882	0.788	0.711	0.647	0.593	0.546	0.506	0.472	0.441	0.414
	4	0.882	0.788	0.710	0.646	0.591	0.545	0.504	0.469	0.439	0.412
	5	0.882	0.787	0.710	0.645	0.590	0.544	0.503	0.468	0.437	0.410
	6	0.882	0.787	0.709	0.644	0.590	0.543	0.502	0.467	0.436	0.409
	7	0.882	0.787	0.709	0.644	0.589	0.542	0.502	0.466	0.435	0.408
	8	0.882	0.787	0.709	0.644	0.589	0.542	0.501	0.466	0.435	0.407
	9	0.882	0.787	0.709	0.644	0.589	0.542	0.501	0.465	0.434	0.407
	10	0.882	0.787	0.709	0.644	0.588	0.541	0.501	0.465	0.434	0.406
CROSS FLOW	1	0.883	0.791	0.716	0.654	0.602	0.558	0.520	0.486	0.457	0.431
	2	0.880	0.780	0.697	0.626	0.565	0.513	0.468	0.428	0.393	0.363
	3	0.879	0.776	0.689	0.615	0.550	0.495	0.447	0.404	0.367	0.335
	4	0.878	0.774	0.685	0.609	0.543	0.485	0.435	0.391	0.353	0.319
	5	0.878	0.773	0.683	0.605	0.538	0.479	0.428	0.383	0.344	0.310
	6	0.877	0.772	0.681	0.603	0.534	0.475	0.423	0.378	0.338	0.303
	7	0.877	0.772	0.680	0.601	0.532	0.472	0.419	0.374	0.333	0.298
	8	0.877	0.771	0.679	0.599	0.530	0.470	0.417	0.371	0.330	0.294
	9	0.877	0.771	0.678	0.598	0.529	0.468	0.415	0.368	0.327	0.291
	10	0.877	0.770	0.678	0.597	0.527	0.466	0.413	0.366	0.325	0.289
COUNTER CURRENT	1	0.883	0.791	0.716	0.654	0.602	0.558	0.520	0.486	0.457	0.431
	2	0.870	0.750	0.644	0.553	0.477	0.413	0.360	0.315	0.278	0.246
	3	0.868	0.739	0.619	0.511	0.419	0.343	0.280	0.230	0.189	0.157
	4	0.868	0.737	0.610	0.492	0.386	0.302	0.232	0.178	0.137	0.106
	5	0.868	0.736	0.606	0.482	0.370	0.276	0.201	0.144	0.103	0.074
	6	0.868	0.736	0.605	0.477	0.359	0.258	0.178	0.120	0.080	0.053
	7	0.868	0.736	0.604	0.474	0.352	0.246	0.162	0.102	0.063	0.039
	8	0.868	0.736	0.604	0.473	0.348	0.236	0.149	0.088	0.050	0.028
	9	0.868	0.736	0.604	0.472	0.345	0.230	0.139	0.077	0.041	0.021
	10	0.868	0.736	0.604	0.472	0.343	0.225	0.130	0.068	0.033	0.016

TABLE 31

TAINC CONCENTRATION REDUCTION RATIOS FOR ACETONIN

K = 1.29 ; D = 0.21
 FAT CONIENT OF FLUID = 4.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FICH RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.882	0.790	0.714	0.652	0.600	0.556	0.517	0.484	0.455	0.429
	2	0.882	0.787	0.711	0.647	0.593	0.547	0.508	0.474	0.444	0.417
	3	0.881	0.787	0.709	0.645	0.590	0.544	0.504	0.470	0.439	0.412
	4	0.881	0.786	0.708	0.644	0.589	0.542	0.502	0.467	0.437	0.409
	5	0.881	0.786	0.708	0.643	0.588	0.541	0.501	0.466	0.435	0.408
	6	0.881	0.786	0.708	0.643	0.588	0.541	0.500	0.465	0.434	0.407
	7	0.881	0.786	0.707	0.642	0.587	0.540	0.500	0.464	0.433	0.406
	8	0.881	0.786	0.707	0.642	0.587	0.540	0.499	0.464	0.433	0.405
	9	0.881	0.785	0.707	0.642	0.587	0.539	0.499	0.463	0.432	0.405
	10	0.881	0.785	0.707	0.642	0.586	0.539	0.498	0.463	0.432	0.404
CROSS FLOW	1	0.882	0.790	0.714	0.652	0.600	0.556	0.517	0.484	0.455	0.429
	2	0.879	0.779	0.695	0.624	0.563	0.510	0.465	0.426	0.391	0.360
	3	0.878	0.775	0.687	0.612	0.548	0.492	0.444	0.402	0.365	0.332
	4	0.877	0.773	0.683	0.606	0.540	0.483	0.432	0.389	0.350	0.317
	5	0.877	0.771	0.681	0.603	0.535	0.476	0.425	0.381	0.341	0.307
	6	0.877	0.771	0.679	0.600	0.532	0.472	0.420	0.375	0.335	0.300
	7	0.876	0.770	0.678	0.598	0.529	0.469	0.417	0.371	0.331	0.295
	8	0.876	0.769	0.677	0.597	0.527	0.467	0.414	0.368	0.327	0.292
	9	0.876	0.769	0.676	0.596	0.526	0.465	0.412	0.365	0.324	0.289
	10	0.876	0.769	0.676	0.595	0.525	0.464	0.410	0.363	0.322	0.286
COUNTER CURRENT	1	0.882	0.790	0.714	0.652	0.600	0.556	0.517	0.484	0.455	0.429
	2	0.865	0.748	0.641	0.550	0.474	0.410	0.357	0.312	0.275	0.244
	3	0.867	0.737	0.616	0.508	0.416	0.339	0.277	0.227	0.187	0.155
	4	0.867	0.735	0.607	0.488	0.384	0.298	0.229	0.175	0.135	0.104
	5	0.867	0.734	0.603	0.478	0.366	0.272	0.197	0.141	0.101	0.072
	6	0.867	0.734	0.601	0.473	0.355	0.254	0.174	0.117	0.078	0.052
	7	0.867	0.734	0.601	0.470	0.347	0.241	0.158	0.099	0.061	0.037
	8	0.867	0.734	0.601	0.469	0.343	0.232	0.145	0.085	0.048	0.027
	9	0.867	0.734	0.600	0.468	0.340	0.225	0.134	0.074	0.039	0.020
	10	0.867	0.734	0.600	0.468	0.338	0.219	0.126	0.065	0.031	0.015

TABLE 32

TAINT CONCENTRATION REDUCTION RATIOS FOR ACETOIN

K = 1.29 ; D = 0.21
 FAT CONTENT OF FLUID = 5.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.882	0.788	0.713	0.651	0.598	0.554	0.515	0.482	0.453	0.427
	2	0.881	0.786	0.709	0.645	0.591	0.545	0.506	0.472	0.442	0.415
	3	0.881	0.785	0.707	0.643	0.588	0.542	0.502	0.467	0.437	0.410
	4	0.880	0.785	0.707	0.642	0.587	0.540	0.500	0.465	0.435	0.407
	5	0.880	0.785	0.706	0.641	0.586	0.539	0.499	0.464	0.433	0.406
	6	0.880	0.784	0.706	0.641	0.586	0.539	0.498	0.463	0.432	0.405
	7	0.880	0.784	0.706	0.640	0.585	0.538	0.497	0.462	0.431	0.404
	8	0.880	0.784	0.705	0.640	0.585	0.538	0.497	0.462	0.431	0.403
	9	0.880	0.784	0.705	0.640	0.584	0.537	0.497	0.461	0.430	0.403
	10	0.880	0.784	0.705	0.640	0.584	0.537	0.496	0.461	0.430	0.402
CROSS FLOW	1	0.882	0.788	0.713	0.651	0.598	0.554	0.515	0.482	0.453	0.427
	2	0.878	0.777	0.693	0.621	0.560	0.508	0.463	0.423	0.388	0.358
	3	0.877	0.773	0.685	0.610	0.546	0.490	0.441	0.399	0.362	0.330
	4	0.876	0.771	0.681	0.604	0.538	0.480	0.430	0.386	0.348	0.314
	5	0.876	0.770	0.679	0.600	0.533	0.474	0.423	0.378	0.339	0.304
	6	0.876	0.769	0.677	0.598	0.529	0.469	0.418	0.372	0.332	0.298
	7	0.875	0.768	0.676	0.596	0.527	0.466	0.414	0.368	0.328	0.293
	8	0.875	0.768	0.675	0.595	0.525	0.464	0.411	0.365	0.324	0.289
	9	0.875	0.767	0.674	0.593	0.523	0.462	0.409	0.362	0.322	0.286
	10	0.875	0.767	0.674	0.593	0.522	0.461	0.407	0.360	0.319	0.284
COUNTER CURRENT	1	0.882	0.788	0.713	0.651	0.598	0.554	0.515	0.482	0.453	0.427
	2	0.866	0.746	0.639	0.548	0.471	0.407	0.354	0.310	0.272	0.241
	3	0.866	0.735	0.613	0.505	0.412	0.336	0.274	0.224	0.184	0.152
	4	0.866	0.732	0.603	0.484	0.380	0.294	0.225	0.172	0.132	0.102
	5	0.866	0.732	0.600	0.474	0.362	0.267	0.193	0.138	0.099	0.070
	6	0.866	0.731	0.598	0.469	0.350	0.249	0.171	0.114	0.075	0.050
	7	0.866	0.731	0.598	0.466	0.338	0.236	0.154	0.096	0.059	0.036
	8	0.866	0.731	0.597	0.465	0.338	0.227	0.140	0.082	0.046	0.026
	9	0.866	0.731	0.597	0.464	0.335	0.220	0.130	0.071	0.037	0.019
	10	0.866	0.731	0.597	0.463	0.333	0.214	0.121	0.062	0.030	0.014

TABLE 33

TAINT CONCENTRATION REDUCTION RATIOS FOR ACETOIN

K = 1.29 ; D = 0.21
 FAT CONTENT OF FLUID = 10.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.877	0.781	0.704	0.641	0.588	0.543	0.505	0.472	0.442	0.417
	2	0.876	0.779	0.700	0.635	0.581	0.535	0.495	0.461	0.431	0.405
	3	0.876	0.778	0.698	0.633	0.578	0.531	0.491	0.457	0.426	0.399
	4	0.876	0.777	0.698	0.632	0.576	0.529	0.489	0.454	0.424	0.397
	5	0.876	0.777	0.697	0.631	0.575	0.528	0.488	0.453	0.422	0.395
	6	0.876	0.777	0.697	0.630	0.575	0.528	0.487	0.452	0.421	0.394
	7	0.876	0.777	0.697	0.630	0.574	0.527	0.486	0.451	0.420	0.393
	8	0.876	0.777	0.696	0.630	0.574	0.527	0.486	0.450	0.420	0.392
	9	0.876	0.777	0.696	0.630	0.574	0.526	0.485	0.450	0.419	0.392
	10	0.876	0.777	0.696	0.629	0.574	0.526	0.485	0.450	0.419	0.391
CROSS FLOW	1	0.877	0.781	0.704	0.641	0.588	0.543	0.505	0.472	0.442	0.417
	2	0.873	0.769	0.683	0.610	0.549	0.496	0.450	0.411	0.376	0.346
	3	0.872	0.765	0.675	0.598	0.533	0.477	0.428	0.386	0.349	0.317
	4	0.871	0.763	0.671	0.592	0.524	0.466	0.416	0.372	0.334	0.301
	5	0.871	0.761	0.668	0.588	0.519	0.460	0.408	0.364	0.325	0.291
	6	0.871	0.760	0.666	0.585	0.516	0.455	0.403	0.358	0.318	0.284
	7	0.871	0.760	0.665	0.583	0.513	0.452	0.399	0.354	0.314	0.279
	8	0.870	0.759	0.664	0.582	0.511	0.450	0.397	0.350	0.310	0.275
	9	0.870	0.759	0.663	0.581	0.509	0.448	0.394	0.346	0.307	0.272
	10	0.870	0.759	0.663	0.580	0.508	0.446	0.392	0.346	0.305	0.270
COUNTER CURRENT	1	0.877	0.781	0.704	0.641	0.588	0.543	0.505	0.472	0.442	0.417
	2	0.862	0.736	0.626	0.534	0.456	0.393	0.340	0.296	0.260	0.229
	3	0.860	0.724	0.598	0.488	0.395	0.318	0.257	0.209	0.171	0.141
	4	0.860	0.721	0.588	0.465	0.360	0.275	0.208	0.157	0.119	0.091
	5	0.860	0.720	0.583	0.454	0.340	0.246	0.175	0.123	0.086	0.061
	6	0.860	0.720	0.581	0.447	0.327	0.227	0.151	0.099	0.064	0.042
	7	0.860	0.720	0.580	0.444	0.318	0.212	0.134	0.081	0.048	0.029
	8	0.860	0.720	0.580	0.442	0.312	0.202	0.120	0.068	0.037	0.020
	9	0.860	0.720	0.580	0.441	0.308	0.194	0.109	0.057	0.029	0.014
	10	0.860	0.720	0.580	0.440	0.306	0.187	0.100	0.048	0.022	0.010

TABLE 34

TAINT CONCENTRATION REDUCTION RATIOS FOR
ACETOIN

$K = 1.29 ; D = 0.21$

FAT CONCENT OF FLUID = 20.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.867	0.765	0.685	0.620	0.566	0.521	0.483	0.449	0.420	0.395
	2	0.866	0.763	0.680	0.614	0.558	0.512	0.472	0.438	0.408	0.382
	3	0.866	0.762	0.679	0.611	0.555	0.508	0.468	0.433	0.403	0.377
	4	0.866	0.761	0.678	0.610	0.553	0.506	0.466	0.431	0.401	0.374
	5	0.866	0.761	0.677	0.609	0.552	0.505	0.464	0.429	0.399	0.372
	6	0.866	0.761	0.677	0.608	0.552	0.504	0.463	0.428	0.398	0.371
	7	0.866	0.761	0.677	0.608	0.551	0.503	0.463	0.427	0.397	0.370
	8	0.865	0.760	0.676	0.608	0.551	0.503	0.462	0.427	0.396	0.369
	9	0.865	0.760	0.676	0.608	0.551	0.503	0.462	0.426	0.396	0.369
	10	0.865	0.760	0.676	0.607	0.550	0.502	0.461	0.426	0.395	0.368
CROSS FLOW	1	0.867	0.765	0.685	0.620	0.566	0.521	0.483	0.449	0.420	0.395
	2	0.863	0.752	0.661	0.586	0.523	0.469	0.424	0.384	0.350	0.321
	3	0.861	0.747	0.652	0.573	0.505	0.448	0.400	0.358	0.322	0.290
	4	0.860	0.744	0.647	0.565	0.496	0.437	0.387	0.343	0.306	0.273
	5	0.860	0.743	0.644	0.561	0.490	0.430	0.378	0.334	0.296	0.263
	6	0.860	0.742	0.642	0.558	0.486	0.425	0.373	0.328	0.289	0.256
	7	0.859	0.741	0.641	0.556	0.483	0.422	0.369	0.323	0.284	0.250
	8	0.859	0.740	0.640	0.554	0.481	0.419	0.366	0.320	0.280	0.246
	9	0.859	0.740	0.639	0.553	0.479	0.417	0.363	0.317	0.277	0.243
	10	0.859	0.739	0.638	0.552	0.478	0.415	0.361	0.315	0.275	0.240
COUNTER CURRENT	1	0.867	0.765	0.685	0.620	0.566	0.521	0.483	0.449	0.420	0.395
	2	0.850	0.714	0.598	0.503	0.425	0.362	0.310	0.268	0.234	0.205
	3	0.847	0.700	0.566	0.451	0.357	0.282	0.224	0.180	0.145	0.118
	4	0.847	0.695	0.552	0.424	0.318	0.235	0.173	0.128	0.095	0.072
	5	0.847	0.694	0.546	0.409	0.293	0.204	0.139	0.094	0.065	0.045
	6	0.847	0.694	0.543	0.400	0.277	0.181	0.115	0.072	0.045	0.028
	7	0.847	0.694	0.541	0.395	0.265	0.165	0.097	0.055	0.031	0.018
	8	0.847	0.694	0.541	0.392	0.257	0.152	0.083	0.043	0.022	0.012
	9	0.847	0.694	0.541	0.390	0.251	0.142	0.072	0.034	0.016	0.008
	10	0.847	0.694	0.540	0.389	0.247	0.134	0.063	0.027	0.011	0.005

TABLE 35

TAINT CONCENTRATION REDUCTION RATIOS FOR ACETOIN

K = 1.29 ; D = 0.21

FAT CONTENT OF FLUID = 30.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FICR RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.855	0.747	0.663	0.557	0.542	0.496	0.458	0.425	0.397	0.372
	2	0.854	0.744	0.658	0.589	0.533	0.486	0.447	0.413	0.384	0.359
	3	0.854	0.743	0.656	0.587	0.530	0.482	0.442	0.408	0.379	0.353
	4	0.854	0.742	0.655	0.585	0.528	0.480	0.440	0.406	0.376	0.350
	5	0.854	0.742	0.655	0.584	0.527	0.479	0.438	0.404	0.374	0.348
	6	0.854	0.742	0.654	0.584	0.526	0.478	0.437	0.403	0.373	0.347
	7	0.853	0.742	0.654	0.583	0.525	0.477	0.437	0.402	0.372	0.346
	8	0.853	0.742	0.654	0.583	0.525	0.477	0.436	0.401	0.371	0.345
	9	0.853	0.741	0.653	0.583	0.525	0.476	0.436	0.401	0.371	0.344
	10	0.853	0.741	0.653	0.582	0.524	0.476	0.435	0.400	0.370	0.344
CROSS FLOW	1	0.855	0.747	0.663	0.597	0.542	0.496	0.458	0.425	0.397	0.372
	2	0.850	0.732	0.636	0.558	0.494	0.440	0.395	0.356	0.323	0.294
	3	0.848	0.726	0.626	0.543	0.475	0.417	0.369	0.327	0.292	0.262
	4	0.847	0.723	0.620	0.535	0.464	0.405	0.355	0.312	0.275	0.244
	5	0.847	0.721	0.617	0.530	0.458	0.397	0.346	0.302	0.265	0.233
	6	0.846	0.720	0.615	0.527	0.453	0.392	0.340	0.295	0.258	0.225
	7	0.846	0.719	0.613	0.524	0.450	0.388	0.335	0.290	0.252	0.220
	8	0.846	0.718	0.612	0.522	0.448	0.385	0.332	0.287	0.248	0.216
	9	0.846	0.718	0.611	0.521	0.446	0.382	0.329	0.284	0.245	0.212
	10	0.846	0.717	0.610	0.520	0.444	0.381	0.327	0.281	0.243	0.210
COUNTER CURRENT	1	0.855	0.747	0.663	0.597	0.542	0.496	0.458	0.425	0.397	0.372
	2	0.835	0.688	0.567	0.469	0.391	0.329	0.279	0.239	0.207	0.180
	3	0.832	0.671	0.528	0.409	0.316	0.245	0.191	0.150	0.120	0.096
	4	0.831	0.665	0.510	0.377	0.272	0.194	0.139	0.100	0.073	0.054
	5	0.831	0.663	0.501	0.358	0.244	0.161	0.105	0.069	0.046	0.031
	6	0.831	0.662	0.497	0.346	0.224	0.137	0.081	0.048	0.029	0.018
	7	0.831	0.662	0.495	0.339	0.209	0.119	0.064	0.035	0.019	0.011
	8	0.831	0.662	0.494	0.334	0.198	0.105	0.052	0.025	0.012	0.006
	9	0.831	0.662	0.493	0.330	0.190	0.094	0.042	0.018	0.008	0.004
	10	0.831	0.662	0.493	0.328	0.184	0.085	0.034	0.013	0.005	0.002

TABLE 41

TAINT CONCENTRATION REDUCTION RATIOS FOR
BENZYL MERCAPTAN

K = 73.00 ; D = 160.00

FAT CONTENT OF FLUID = 20.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.818	0.692	0.600	0.529	0.473	0.428	0.391	0.360	0.333	0.310
	2	0.810	0.669	0.563	0.480	0.414	0.361	0.317	0.281	0.251	0.226
	3	0.807	0.661	0.548	0.460	0.390	0.333	0.287	0.249	0.218	0.192
	4	0.805	0.656	0.540	0.449	0.376	0.318	0.271	0.232	0.200	0.173
	5	0.805	0.653	0.535	0.442	0.368	0.309	0.260	0.221	0.188	0.162
	6	0.804	0.652	0.532	0.438	0.362	0.302	0.253	0.213	0.181	0.154
	7	0.803	0.650	0.530	0.434	0.358	0.297	0.248	0.208	0.175	0.148
	8	0.803	0.649	0.528	0.432	0.355	0.293	0.244	0.203	0.171	0.144
	9	0.803	0.648	0.526	0.430	0.353	0.291	0.241	0.200	0.167	0.140
	10	0.803	0.648	0.525	0.428	0.351	0.288	0.238	0.197	0.164	0.137
CROSS FLOW	1	0.818	0.692	0.600	0.529	0.473	0.428	0.391	0.360	0.333	0.310
	2	0.810	0.669	0.562	0.479	0.413	0.360	0.316	0.280	0.250	0.224
	3	0.807	0.660	0.547	0.459	0.388	0.331	0.285	0.247	0.216	0.189
	4	0.805	0.656	0.539	0.448	0.375	0.316	0.268	0.229	0.197	0.170
	5	0.804	0.653	0.534	0.441	0.366	0.306	0.258	0.218	0.186	0.159
	6	0.804	0.651	0.531	0.436	0.360	0.299	0.250	0.210	0.178	0.151
	7	0.803	0.650	0.528	0.433	0.356	0.295	0.245	0.205	0.172	0.145
	8	0.803	0.648	0.527	0.430	0.353	0.291	0.241	0.200	0.167	0.140
	9	0.803	0.648	0.525	0.428	0.350	0.288	0.238	0.197	0.164	0.137
	10	0.802	0.647	0.524	0.426	0.348	0.286	0.235	0.194	0.161	0.134
COUNTER CURRENT	1	0.818	0.652	0.600	0.529	0.473	0.428	0.391	0.360	0.333	0.310
	2	0.786	0.609	0.473	0.373	0.298	0.243	0.201	0.168	0.143	0.122
	3	0.779	0.578	0.415	0.295	0.211	0.154	0.114	0.086	0.066	0.052
	4	0.778	0.565	0.383	0.249	0.160	0.103	0.068	0.046	0.032	0.023
	5	0.778	0.559	0.365	0.219	0.125	0.072	0.042	0.025	0.016	0.010
	6	0.777	0.557	0.353	0.157	0.101	0.051	0.026	0.014	0.008	0.005
	7	0.777	0.556	0.346	0.181	0.083	0.037	0.017	0.008	0.004	0.002
	8	0.777	0.555	0.341	0.169	0.070	0.027	0.011	0.004	0.001	0.001
	9	0.777	0.555	0.338	0.160	0.059	0.020	0.007	0.002	0.001	0.000
	10	0.777	0.555	0.336	0.152	0.050	0.015	0.004	0.001	0.000	0.000

TABLE 42

TAINT CONCENTRATION REDUCTION RATIOS FOR
BENZYL MERCAPTAN

K = 73.00 ; D = 160.00
FAT CONTENT OF FLUID = 30.0 PERCENT

TYPE OF PROCESS	NUMBER OF STAGES	STEAM FLOW RATIO									
		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
STEP FLASH	1	0.870	0.769	0.690	0.625	0.572	0.526	0.488	0.455	0.426	0.400
	2	0.865	0.756	0.667	0.592	0.530	0.477	0.431	0.392	0.358	0.328
	3	0.864	0.752	0.658	0.580	0.513	0.457	0.408	0.366	0.330	0.299
	4	0.863	0.749	0.654	0.573	0.504	0.446	0.396	0.352	0.315	0.282
	5	0.863	0.748	0.651	0.569	0.499	0.439	0.388	0.343	0.305	0.272
	6	0.862	0.747	0.649	0.566	0.495	0.434	0.382	0.337	0.299	0.265
	7	0.862	0.746	0.647	0.564	0.492	0.431	0.378	0.333	0.294	0.260
	8	0.862	0.745	0.646	0.562	0.490	0.428	0.375	0.330	0.290	0.256
	9	0.862	0.745	0.646	0.561	0.488	0.426	0.373	0.327	0.287	0.253
	10	0.862	0.745	0.645	0.560	0.487	0.425	0.371	0.325	0.285	0.250
CROSS FLOW	1	0.870	0.769	0.690	0.625	0.572	0.526	0.488	0.455	0.426	0.400
	2	0.865	0.756	0.667	0.592	0.529	0.476	0.430	0.391	0.357	0.327
	3	0.864	0.751	0.658	0.579	0.512	0.455	0.407	0.365	0.328	0.296
	4	0.863	0.749	0.653	0.572	0.503	0.444	0.394	0.350	0.313	0.280
	5	0.863	0.747	0.650	0.568	0.497	0.437	0.386	0.341	0.303	0.270
	6	0.862	0.746	0.648	0.565	0.493	0.433	0.380	0.335	0.296	0.262
	7	0.862	0.746	0.647	0.563	0.491	0.429	0.376	0.331	0.291	0.257
	8	0.862	0.745	0.646	0.561	0.488	0.426	0.373	0.327	0.287	0.253
	9	0.862	0.745	0.645	0.560	0.487	0.424	0.371	0.324	0.284	0.250
	10	0.862	0.744	0.644	0.559	0.485	0.423	0.369	0.322	0.282	0.247
COUNTER CURRENT	1	0.870	0.769	0.690	0.625	0.572	0.526	0.488	0.455	0.426	0.400
	2	0.853	0.720	0.605	0.510	0.433	0.369	0.317	0.275	0.240	0.211
	3	0.851	0.706	0.574	0.460	0.366	0.291	0.232	0.187	0.151	0.123
	4	0.850	0.702	0.561	0.434	0.328	0.244	0.181	0.135	0.101	0.076
	5	0.850	0.701	0.555	0.420	0.304	0.214	0.147	0.101	0.069	0.048
	6	0.850	0.700	0.552	0.412	0.289	0.192	0.123	0.078	0.049	0.031
	7	0.850	0.700	0.551	0.407	0.278	0.176	0.105	0.061	0.035	0.020
	8	0.850	0.700	0.551	0.404	0.271	0.164	0.091	0.048	0.025	0.013
	9	0.850	0.700	0.550	0.403	0.265	0.154	0.080	0.039	0.018	0.009
	10	0.850	0.700	0.550	0.402	0.261	0.146	0.071	0.031	0.013	0.006

mercaptan. This can also be deduced from a consideration of the equations for stepwise flash and crossflow systems, i.e. equations VI.28 and VI.29, respectively. These equations can be rewritten as

$$\frac{x_n}{x_0} = \prod_{i=1}^n \left[\frac{1}{1 + \frac{Sk}{nL_0A_0 + (n-i)S}} \right] \quad (\text{VI.32})$$

for the stepwise flash system, and

$$\frac{x_n}{x_0} = \prod_{i=1}^n \left[\frac{1}{1 + \frac{Sk}{nL_0A_0}} \right] \quad (\text{VI.33})$$

for the crossflow system. Comparison of corresponding terms of these equations shows that the taint reduction for crossflow system is greater than that for the stepwise flash system, and that the difference between the two systems increases as A_0 decreases (i.e. as D becomes small). The difference is also increased when the number of stages n and the steam flow rate S are increased.

b. Effect of Number of Stages

Figures 31 to 33 and Tables 22 to 42 show that for crossflow and stepwise flash systems the effect of additional stages beyond 3 or 4 is quite small. With the

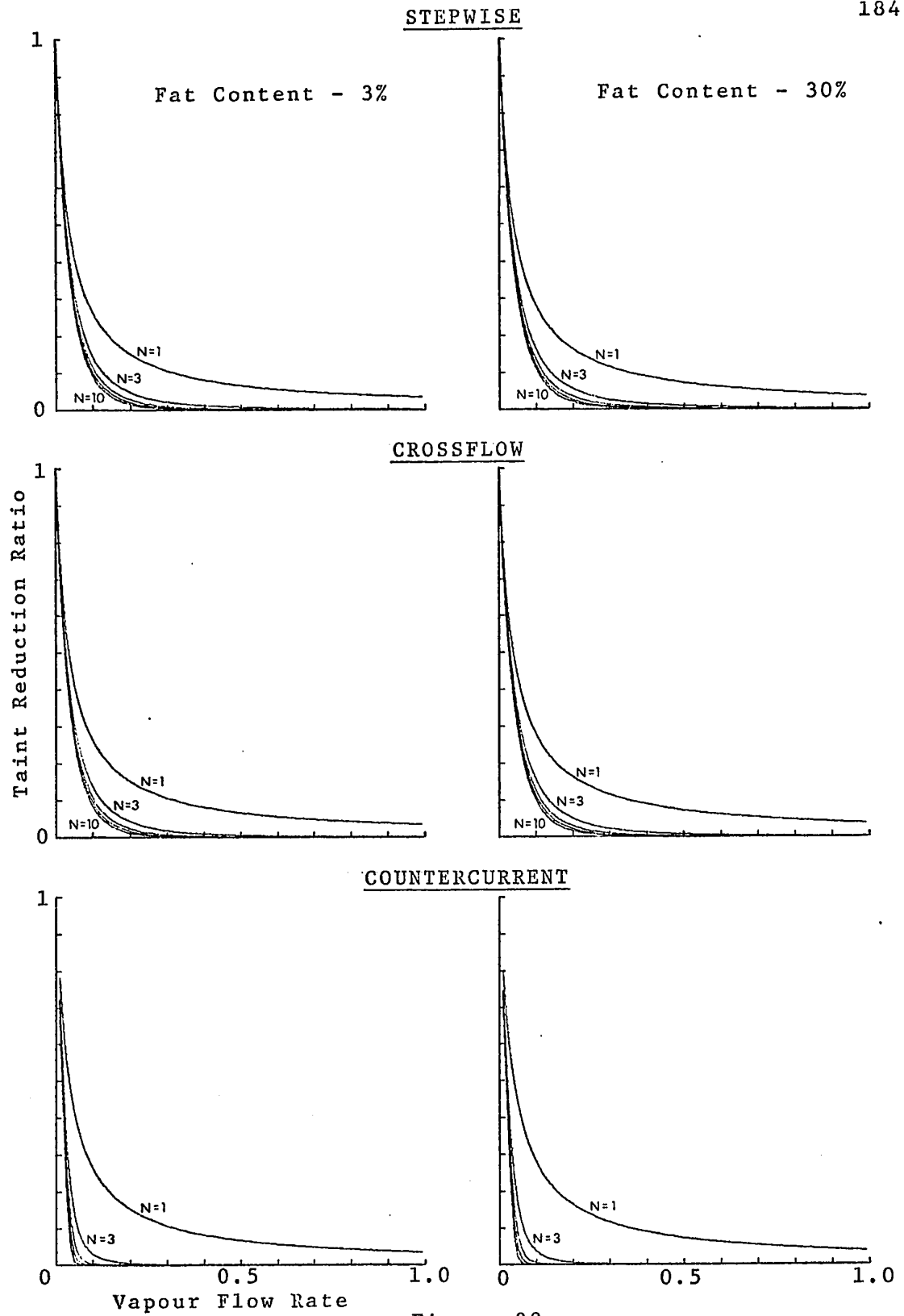
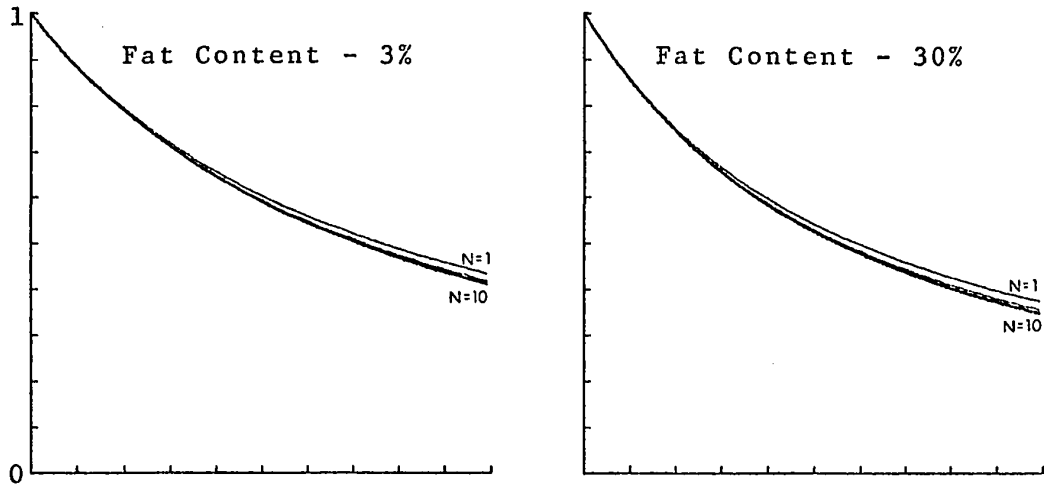
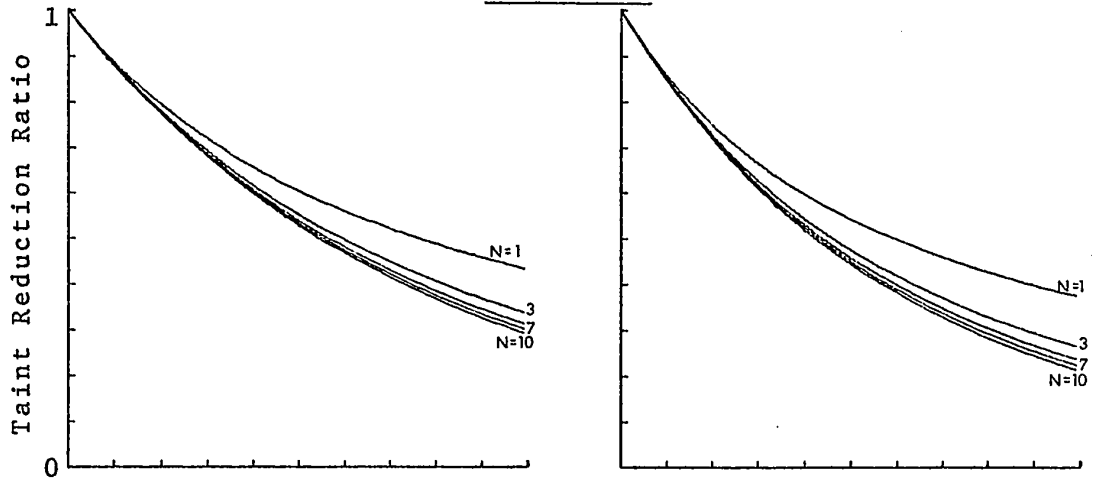


Figure 28

EFFECT OF VAPOUR FLOW RATE ON TAIN T REDUCTION
RATIOS FOR DIACETYL IN
MULTISTAGE DEODORIZING SYSTEMS



CROSSFLOW



COUNTERCURRENT

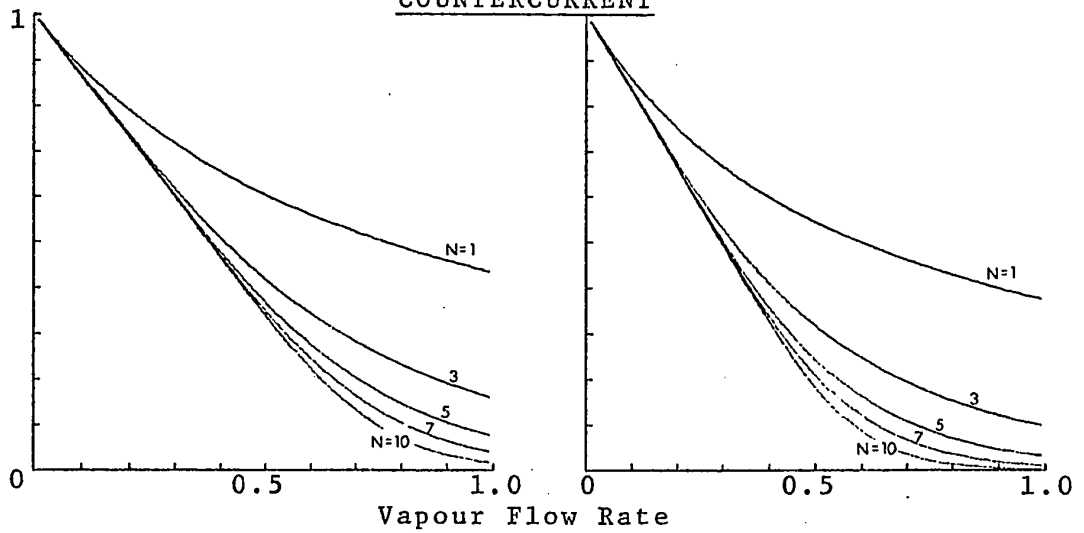
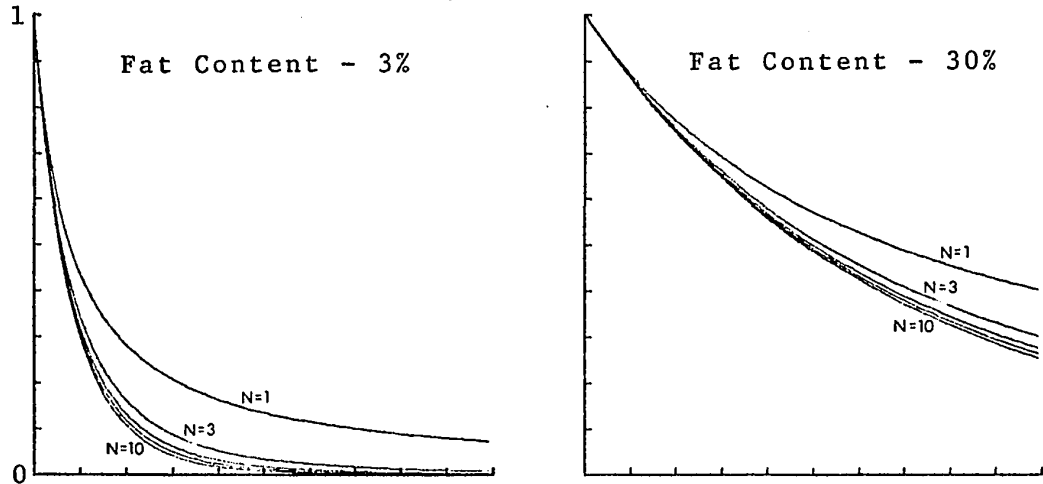


Figure 29

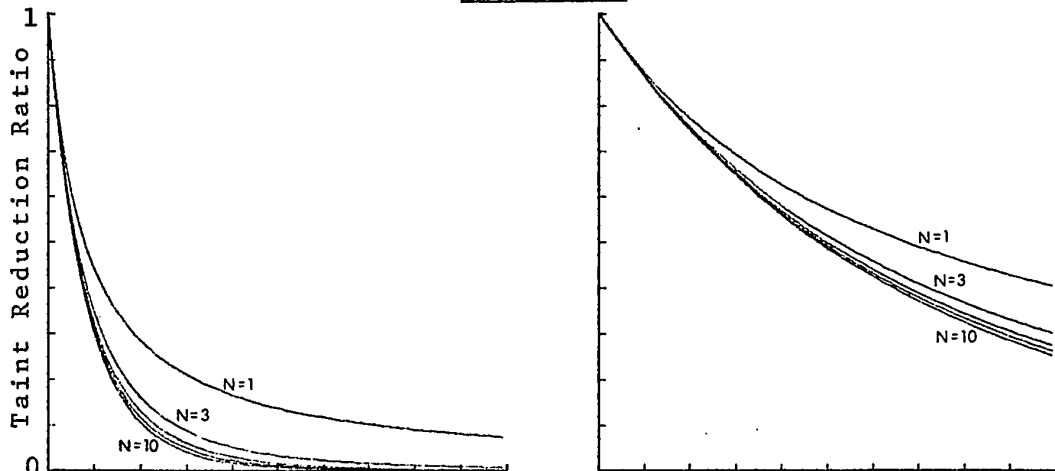
EFFECT OF VAPOUR FLOW RATE ON TAINT REDUCTION RATIOS FOR ACETOIN IN MULTISTAGE DEODORIZING SYSTEMS

STEPWISE

186.



CROSSFLOW



COUNTERCURRENT

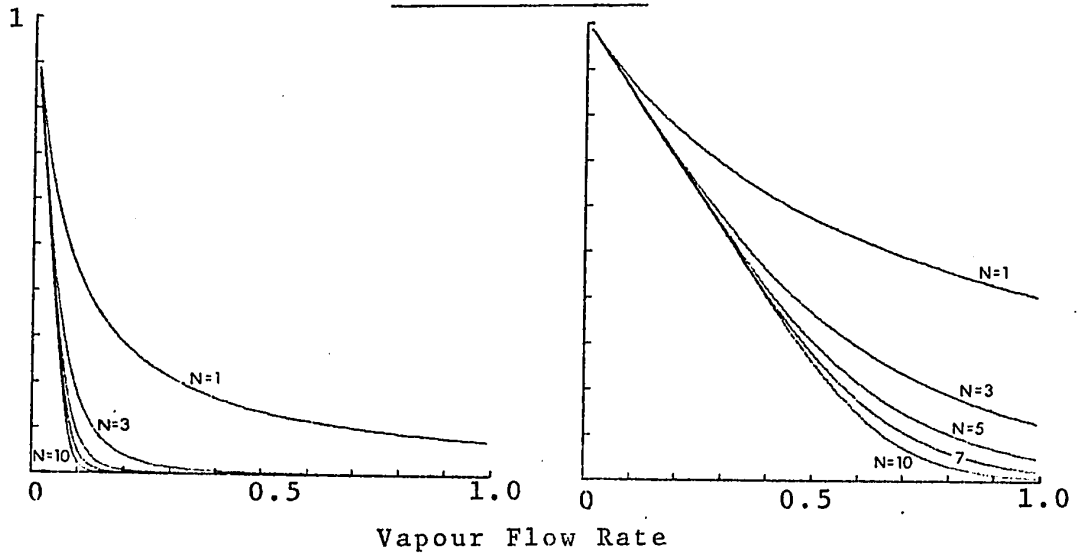


Figure 30

EFFECT OF VAPOUR FLOW RATE ON TAINT REDUCTION RATIOS FOR BENZYL MERCAPTAN IN MULTISTAGE DEODORIZING SYSTEMS

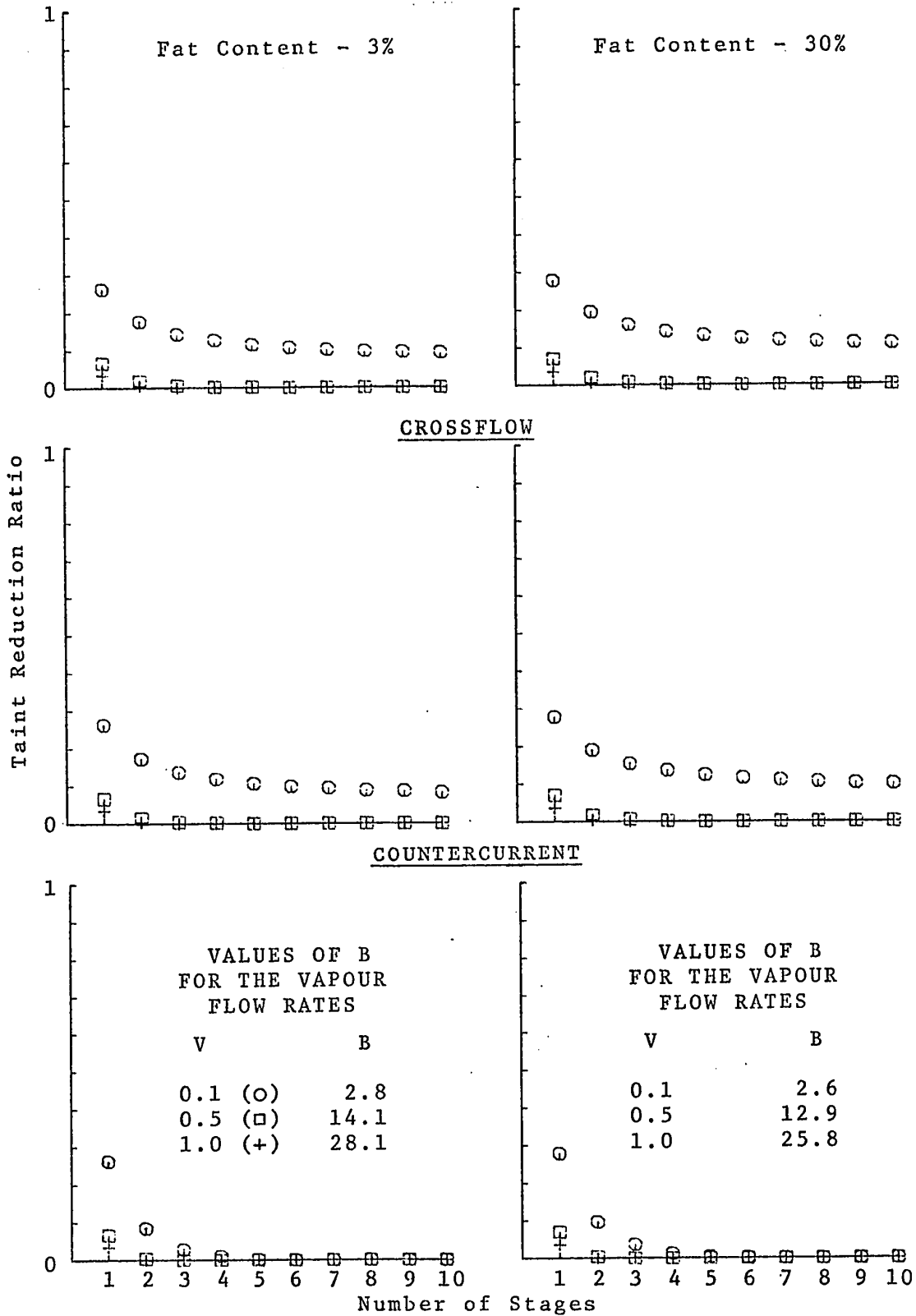


Figure 31

TAINT REDUCTION RATIOS FOR DIACETYL
FOR 1, 2, . . . , 10 STAGES
IN MULTISTAGE DEODORIZING SYSTEMS

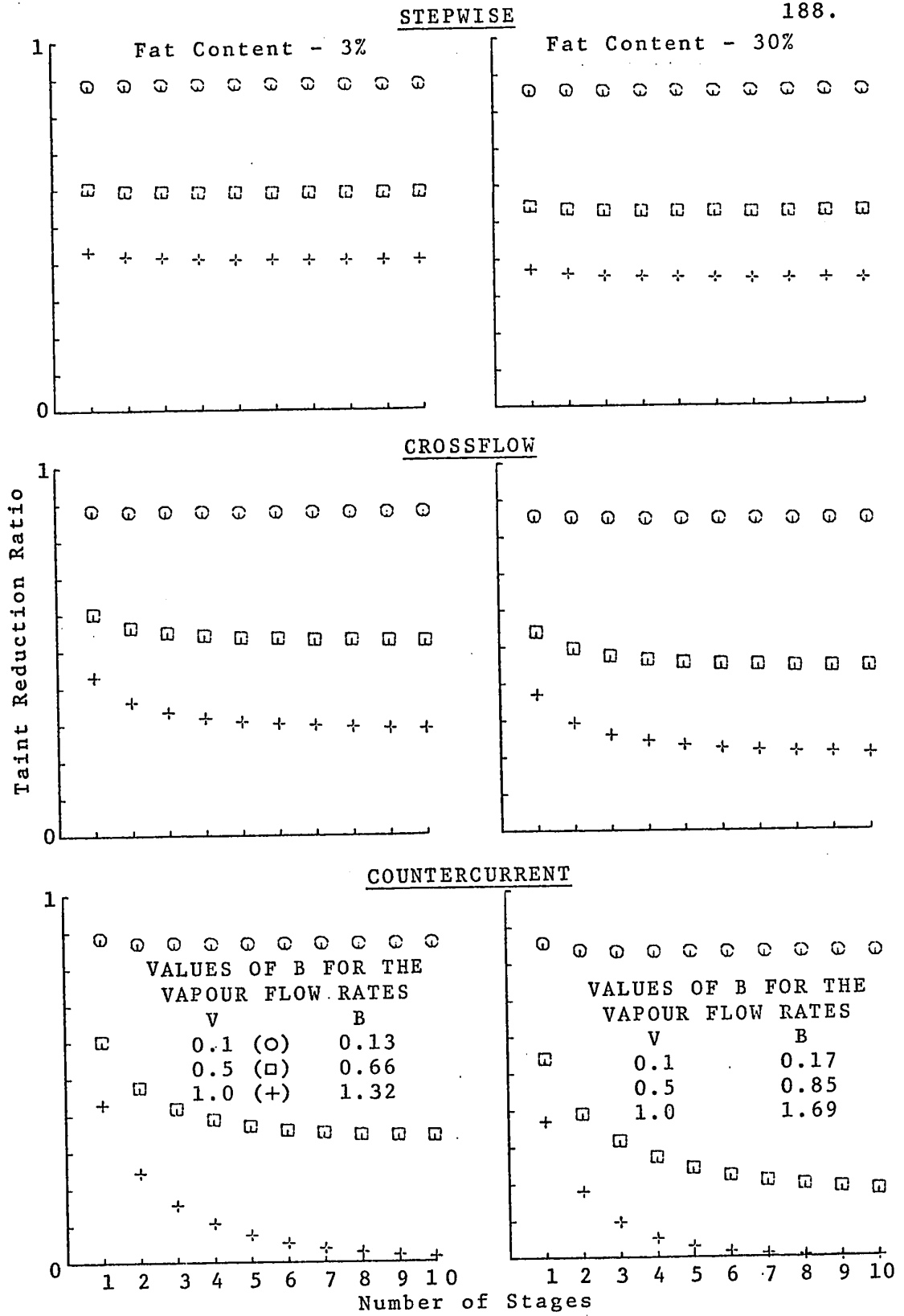


Figure 32

TAIN T REDUCTION RATIOS FOR ACETOIN
FOR 1, 2, ..., 10 STAGES
IN MULTISTAGE DEODORIZING SYSTEMS

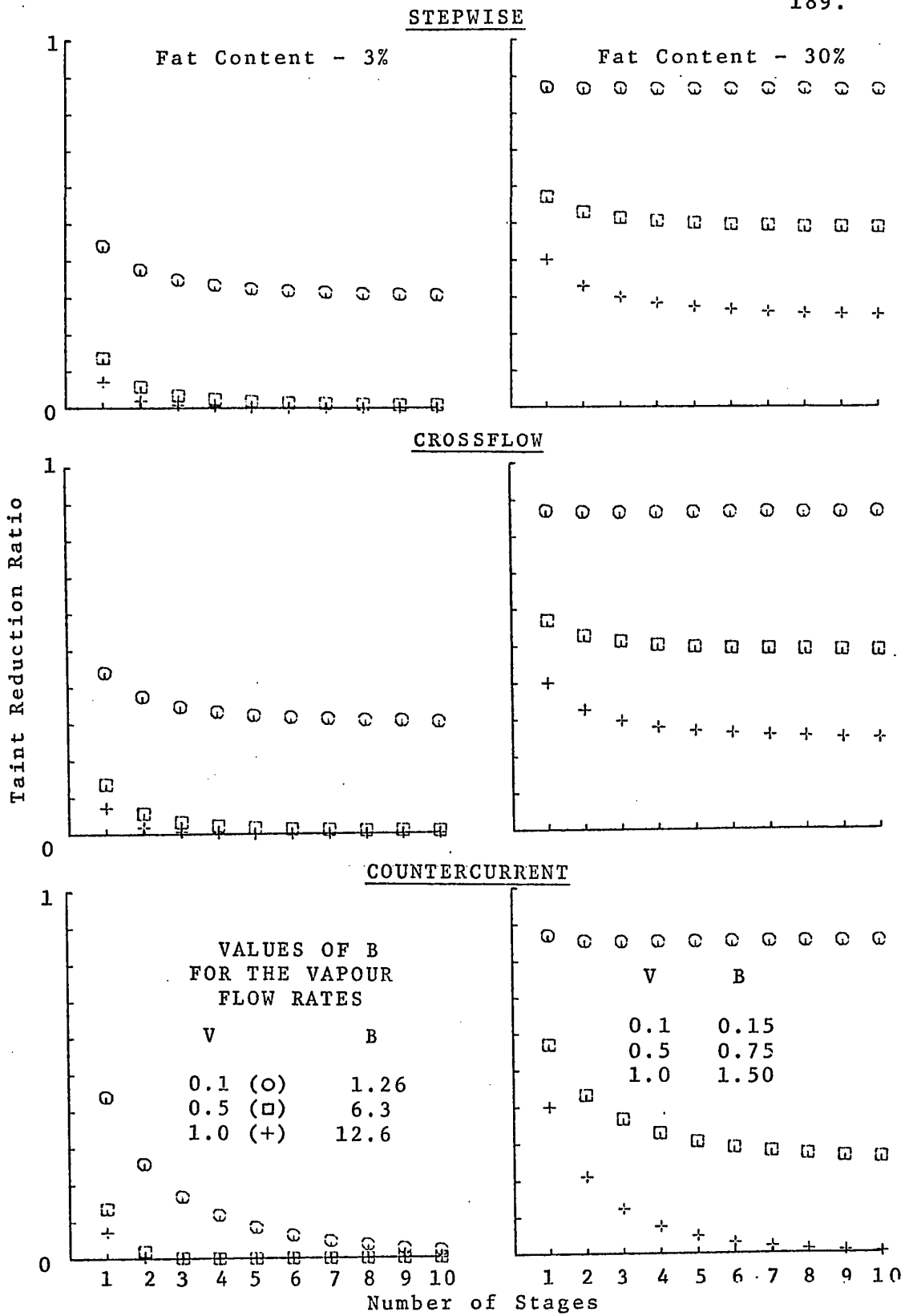


Figure 33

TAINT REDUCTION RATIOS FOR BENZYL MERCAPTAN
FOR 1, 2, . . . , 10 STAGES
IN MULTISTAGE DEODORIZING SYSTEMS

countercurrent system, however, the effect of a 4th, 5th, etc. stage sometimes gives a further significant reduction in the taint concentration ratio. The criterion for the usefulness of increasing the number of stages beyond 3 or 4 is determined from a consideration of equation VI.31. The series in the denominator is convergent for an infinite number of stages if $B(= Sk/L_0 A_0)$ is less than one, and hence there are operating conditions when even an infinite number of stages will not reduce the taint concentration below a certain level. When a stage $(n+1)$ is added to a finite system of n stages, then a significant increase in the taint reduction will only be obtained if B^{n+1} is not very much smaller than $1 + B + B^2 + \dots + B^n$.

If B is greater than one, this criterion always applies, and if B is much smaller than one, the criterion only applies when the number of stages is small.

In the special case where $B = 1$, the effect of the addition of an extra stage to a countercurrent system can be calculated from a simple relationship derived from equation VI.31, i.e.

$$\frac{x_n}{x_0} = \frac{1}{n + 1}$$

where n is the number of stages.

Values of B for the three steam flow ratios plotted in Figures 31 to 33 are shown on the graphs of taint reduction ratio vs number of (countercurrent) stages in the figures.

c. Effect of Steam Flow Rate

Figures 28 to 30 show the effect of ratio of steam flow rate to liquid flow rate on the taint reduction ratio for diacetyl, acetoin and benzyl mercaptan, respectively, in multistage deodorizers. In all cases increase of steam flow ratio increases the taint reduction, but the curves for the countercurrent systems are different in form from those for the stepwise flash and the crossflow systems.

From the point of view of steam economy, the important quantity is $dS/d(x_n/x_0)$. This quantity is a measure of the increase in steam flow rate required for a unit decrease in the taint reduction ratio.

Figures 28 to 30 show that the curves of taint reduction ratio vs steam flow rate for the stepwise flash and crossflow systems are very similar in form. Equation VI.33 for crossflow deodorization can be rearranged to give

$$S = \frac{nL_0A_0}{k} \left[\left(\frac{x_n}{x_0} \right)^{-1/n} - 1 \right]$$

which leads to

$$\begin{aligned} \frac{dS}{d(x_n/x_0)} &= \frac{-L_0 A_0}{k} \left[\frac{x_n}{x_0} \right]^{-(1+1/n)} \\ &\doteq \frac{-L_0 A_0}{k} \left[\frac{x_n}{x_0} \right]^{-1} \end{aligned} \quad (\text{VI.34})$$

when n is large.

At relatively low steam flow rates the taint reduction ratio for several countercurrent stages will be given by the linear portion of the curve. The equation of this section of the curve is given by equation VI.31 when $n = \infty$,

$$\begin{aligned} \text{i.e.} \quad \frac{x_n}{x_0} &= \frac{1}{\sum_{i=0}^{\infty} (B^i)} \\ &= 1 - B \quad \text{when } B \text{ is less than } 1. \end{aligned}$$

$$\text{i.e.} \quad S = \frac{L_0 A_0}{k} \left[1 - \frac{x_n}{x_0} \right]$$

This gives

$$\frac{dS}{d(x_n/x_0)} = \frac{-L_0 A_0}{k} \quad (\text{VI.35})$$

Comparison of equations VI.34 and VI.35 shows that $dS/d(x_n/x_0)$ for the crossflow system (and hence for the

stepwise flash system) is greater than that for a counter-current system, because (x_n/x_0) is always less than 1 . Hence, when an increase in the taint reduction (i.e. a decrease in the taint reduction ratio) is required, the increase in steam flow in the countercurrent system will be less than that for the other two systems.

VII. CONCLUSIONS AND RECOMMENDATIONS

A. VAPOUR LIQUID AND LIQUID LIQUID EQUILIBRIA DATA

The present study shows that the vapour liquid equilibrium coefficient is a linear function of the pressure at which stripping takes place for diacetyl in water and in skim milk. Corresponding data for other taint compounds are not available, and hence further vapour liquid equilibrium studies are required.

Further experiments may also be required to determine the effect of temperature on the distribution of some taint compounds between the two liquid phases in milk and cream.

B. CENTRIFUGAL DEODORIZATION

The experimental work shows that significant deodorization takes place in the centrifugal deodorizer even though equilibrium was not approached very closely with most of the flow rates and speeds used in this study.

It appears that the resistance to mass transfer in the vapour phase does not make a significant contribution to the overall resistance to mass transfer, whereas the factors which are likely to affect the several resistances to mass transfer in the liquid phase, i.e. liquid flow rate and speed of rotation, were significant. The mass transfer coefficient appeared to be greater for the higher

liquid flow rates than for the lower liquid flow rates. The effect of speed did not appear to be significant with large hold up volumes but was quite significant with the low hold up volumes. It appears that an increase in speed for the low hold up volume experiments changes the flow conditions in some way so as to increase the overall mass transfer coefficient. Further work is needed to determine the mechanism and the magnitude of the effect of speed on deodorization in thin films. The results of the experiments indicate that the effect of speed on deodorization may in part be due to the effect of centrifugal acceleration on the thickness of the aqueous boundary layer around the fat globules, as well as to the greater turbulence at higher speeds.

The experiments also show that the area of the vapour liquid interface is significant, because in general the tests in which the liquid was injected in the form of a spray gave a closer approach to equilibrium than the experiments in which the liquid was directed onto the wall with no spray.

The concentration of diacetyl in the feed liquid appeared to have a significant effect on the approach ratio in some of the tests. The reason for this effect is not apparent from the present study. Further work will need to be done to determine whether the effect of concentration

and a small number of other unexplained effects are real.

The author recommends that further studies of deodorization of systems with two liquid phases be done. The equipment should be vapour tight, and the vapour condensing system should be improved over that used in the present system so that adequate mass balances may be obtained. Steam jacketing may be desirable to minimize condensation. Improved methods of determination of taint concentration in the outlet streams and/or higher taint concentrations in the feed stream are desirable. The study should be extended to include higher fat contents, and compounds which have a higher fat phase aqueous phase distribution coefficient than that of diacetyl.

C. MULTISTAGE DEODORIZING SYSTEMS

Analysis of the recycling system has shown that recycling of either the skim milk or the fat rich phase does not decrease the steam requirements for deodorizing if the fat content of the product is the same as that of the feed stream.

The taint reduction obtained in the stepwise flash system has been shown to be almost the same as that obtained in the crossflow system with the crossflow system giving lower taint reduction ratios than the stepwise flash system for compounds which are more soluble in the aqueous phase

than in the fat phase, e.g. acetoin. Multistage stepwise and crossflow systems give significantly greater taint reductions than single stages but there is little merit in using more than three to five stages with either system.

Greater taint reduction is obtained in a countercurrent system than either the crossflow or stepwise flow system for a given steam flow rate and a number of stages. Further, when an odor problem occurs, an increase in the steam flow rate will have a greater effect on the taint reduction ratio in the countercurrent deodorizing system than in either the crossflow or stepwise systems, under normal operating conditions.

If equilibrium is not achieved, as assumed for the above conclusions, the number of stages required for a given taint reduction ratio will increase. It is expected, however, that the systems will be similar in general characteristics to systems in which equilibrium is achieved.

The author recommends that future studies in milk and cream deodorizing may best be devoted to designing a countercurrent deodorizing system which will be suitable for milk and cream. It should be possible to design a centrifugal deodorizer in which the steam and liquid flows are such that the system is equivalent to several countercurrent stages. Advantages of a system of this type would be that

1. residence times would be short and high temperatures could be used if required;

2. interstage pumps for the liquid stream would not be required as they are with countercurrent connection of simple mixing stages, such as Vacreactors;

3. heat losses would be smaller than with most conventional deodorizers and hence condensation of steam would be less;

4. good contact between the steam and liquid could be obtained without formation of foam;

5. construction would be of a simple form so that the equipment could be easily cleaned and sterilized.

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IX. NOMENCLATURE

- a_{32} = surface mean radius (L).
- A = total area of contact (L^2).
Also defined by $A_n = 1 + (F_n/L_n)(D-1)$ in equation VI.11 .
- B = SK/L_0A_0 (p. 158).
- C = denotes concentration in the analysis of variance.
- D = diffusion coefficient, (L^2t^{-1}).
= f/p = fat phase aqueous phase distribution coefficient as defined by equation II.2 .
- E = error term defined by equation V.5 .
- f = concentration of taint compound in fat phase.
- F = mass flow rate of fat phase, (Mt^{-1}).
Used to denote "fluid" in the analysis of variance (p. 129).
- g = acceleration due to gravity, (Lt^{-2}).
- k = y/p = vapour phase aqueous phase equilibrium coefficient as defined by equation II.1 .
- k_L = average mass transfer coefficient for the continuous phase, (Lt^{-1}).
- k' = y/x = vapour total liquid equilibrium coefficient.
- k_1 = reaction rate constant (p. 25).
- k_2 = reaction rate constant (p. 25).
- L = mass flow rate of total liquid phase, (Mt^{-1}).
It is used to denote the dimension, length and the effect of liquid flow rate in the analysis of variance. It is also the length of the centrifuge bowl (p. 74), (L).
- m = k' = vapour total liquid equilibrium coefficient (p. 47).
- M = denotes the dimension, mass.
It is also used to represent a milk component (p. 25).

- p = concentrations of the taint compound in the aqueous phase.
- P = mass flow rate of the aqueous phase, (Mt^{-1}).
- r = radius, (L).
- R = approach ratio defined by equation V.1 .
It also denotes the effect of speed of rotation in the analysis of variance.
- Re = $4r/\mu$ = Reynolds number.
- \bar{R} = overall mean approach ratio (equation V.5).
- RPM = revolutions per minute.
- s = concentration of the taint compound in the inlet steam.
- S = mass flow rate of steam, (Mt^{-1}).
- t = time, (t).
- T = represents a taint compound (p. 25).
It is a matrix in the ANOVA program (p. 105).
It also denotes the effect of test in the analysis of variance.
- T-M = represents a taint milk complex.
- V = mass flow rate of vapour, (Mt^{-1}).
It also denotes the effect of vapour flow rate in the analysis of variance.
- V_h = holdup volume (p. 74), (L^3).
- x = concentration of the taint compound in the overall liquid phase.
- x_{2e} = concentration of a taint in the liquid stream leaving a single stage deodorizer in which equilibrium between phases is achieved (equation V.2).
- y = concentration of the taint compound in the vapour phase.
- Z = a matrix of data used in APL computation (p. 104 and Appendix D).

= height of the free surface in the centrifuge bowl
(p. 74), (L).

OTHER SYMBOLS

Σ = summation operator

Π = product operator

Γ = mass flow rate per unit width of film, ($Mt^{-1}L^{-1}$)

Δ = $(V_3K + L_1A_1)(L_5A_5 + V_9K) + L_6A_6V_9K$ (equation VI.20)

μ = viscosity, ($Mt^{-1}L^{-1}$).
It can also be 10^{-6} meters, (L).

ρ = density, ($M L^{-3}$)

ω = angular velocity

X. APPENDIX

APPENDIX A

Physical Properties of Diacetyl¹

Technical name	2, 3 - Butanedione
Molecular weight	86.09
Chemical formula	$C_4H_6O_2$
Soluble in about four parts of water	
Carrier of aroma of butter, vinegar, coffee and other food products	

Vapour Pressure of Diacetyl²

Temperature °C	Vapour Pressure mm Hg
20.92	35.5
26.45	43.4
35.53	82.9
49.58	164.2
61.58	280.3
73.64	402.6
80.05	530.5
81.79	597.5
87.79	764.2

¹ Merck Index, pg. 317

² McDowall (1955a)

APPENDIX B

CALCULATION OF THE AMOUNT OF DIACETYL COLLECTED IN THE COLD TRAP

Assume that the starting liquid concentration is 1 ppm and that the ideal gas law and Raoults law apply.

1 ppm diacetyl solution is approximately 0.295×10^{-6} Molar
in diacetyl.

The vapour pressure at 36°C is 83 mm Hg (Appendix A).

Therefore, the partial pressure of diacetyl is

$$\begin{aligned} (0.295 \times 10^{-6}) 83 &= 17.4 \times 10^{-6} \text{ mm Hg (Raoults Law)} \\ &= 2.28 \times 10^{-8} \text{ atmospheres} \end{aligned}$$

Since 5 cc of gas are passed through the cold trap, using the ideal gas law, the weight of diacetyl in the gas is

$$\frac{MPV}{RT} = \frac{86.09(2.28 \times 10^{-8}) 50}{82.06(309)} \approx 4 \times 10^{-9} \text{ grams}$$

The amount of diacetyl in the vial (10 cc of solution) is 10×10^{-6} grams. Therefore, the amount removed in the 50 cc purge is less than 1% of the available diacetyl.

APPENDIX C

APPROACH RATIO PROGRAM

```
V CALC M
[1] V←10
[2] I←0
[3] I←I+1
[4] A←1+F×(D-1)
[5] X2E←(M[I;3]×RH×M[I;1]×A)÷((M[I;3]×A)+M[I;2]×K)
[6] AR←(M[I;7]-X2E)÷(M[I;1]-X2E)
[7] →10×1AR≤0
[8] LR←10⊗AR
[9] →12
[10] LR←0
[11] AR←0
[12] V←V,X2E,AR,LR
[13] →3×1I<24
V
```

The approach ratio for every run within each test is calculated by this program. The raw data was entered from the terminal or copied from the user's library, as were the numerical values for K, D, F and RH, (the vapour phase aqueous phase equilibrium coefficient, the liquid liquid distribution coefficient, the fat content and the density of the liquid phase, respectively). The approach ratio program first enters an empty vector and initializes and increments I. The value of A is calculated and used in the calculation of the outlet liquid concentration. Line 6 calculates the approach ratio and line 7 directs the empty vector to the sequence of operations to step 10, if $AR \leq 0$ or computes the logarithm of the approach ratio if $AR > 0$.

If AR is ≤ 0 , lines 10 and 11 set the logarithm AR and AR equal to zero respectively. The calculated values of $X2E$, AR and LR form the first three terms in vector V if $I=1$ or are concatenated to the V from the previous calculation if $I>1$. Line 13 branches back to line 3 if $I<24$ or ends calculations if $I=24$.

APPENDIX D

REPLACE PROGRAM

```
∇ REPLACE Z;NN;CV;P;R;Q;A1;A2;A3;A4;A5;A6
[1] CV←(F)ρ0
[2] R←(NE,ρF)ρ0
[3] P←0
[4] P←P+1
[5] Q←0
[6] Q←Q+1
[7] NN←0
[8] NN←NN+1
[9] X←(NE,6)ρX
[10] R[P;Q]←((R[P;Q])+(X[NN;Q]=X[P;Q]))
[11] →8×1NN<NE
[12] →6×1Q<ρF
[13] →4×1P<NE
[14] P←0
[15] P←P+1
[16] ND←×/F
[17] Z←(F)ρZ
[18] A1←(+/,Z[(X[P;1]);;;;])÷((ND F[1])-R[P;1])
[19] A2←(+/,Z[;(X[P;2]);;;;])÷((ND F[2])-R[P;2])
[20] A3←(+/,Z[;;(X[P;3]);;;])÷((ND F[3])-R[P;3])
[21] A4←(+/,Z[;;;(X[P;4]);;])÷((ND F[4])-R[P;4])
[22] A5←(+/,Z[;;;;(X[P;5]);])÷((ND F[5])-R[P;5])
[23] A6←(+/,Z[;;;;;(X[P;6]);])÷((ND F[6])-R[P;6])
[24] CV[(X[P;1]);(X[P;2]);(X[P;3]);(X[P;4]);(X[P;5]);
(X[P;6])]←(A1+A2+A3+A4+A5+A6)-4×(+/,Z)÷ND-NE
[25] →15×1P<NE
[26] □←Z+CV
∇
```

The replace program calculates the expected value of the approach ratio. An array of the approach ratios with zeroes in place of the "incorrect" values is entered from the terminal or copied from the user's library. Also entered are the number of error terms NE, the dimension of the array F, and the coordinates of the "incorrect" values X.

The first eight lines enter empty arrays CV and Q and initialize and increment P, Q and NN. X, which was entered as a string, is restructured in line 9. Line 10 calculates the value of R for each P and Q. The next three lines loop the calculations so that all the error terms are calculated. Lines 14 and 15 initialize and increment P so that the next lines are looped for the number of error terms.

The number of terms in the array is calculated by travelling F in line 16. The array of approach ratios is restructured in line 17 and then lines 18 - 23 calculate the differences from the overall mean due to each level. The corrected value is calculated for each error term and the corrections are added to the array of approach ratios with zeroes in place of the "incorrect" values. The corrected matrix is then printed out in line 26.

APPENDIX E

PRINT PROGRAM

```
∇ PRINT Z
[1] T2← 0 1 1 1 1 0 \Z
[2] T2[;1]←1(ρZ[;1])
[3] T2[;6]←Z[;4] Z[((ρZ[;1])-1);4]
[4] T2
∇
```

The print program requires as its input the T matrix from the ANOVA calculations. The T matrix which normally has four columns (identification, degrees of freedom, sum of squares and mean square) is expanded by two columns in the first line. The second line simply numbers the lines and this becomes column one. The variance ratio is calculated in line 3 and this becomes column 6. The matrix is then printed out.

APPENDIX F

POOL PROGRAM

```

V POOL T;M;MM;N;Q
[1] 'PRINT P'
[2] P←□
[3] 'PRINT NO OF ERRORS'
[4] NE←□
[5] M← $\iota(\rho T[;1])$ 
[6] MM←(M∈P)
[7] NM← $\sim(MM)$ 
[8] Q←M×( $\sim M$ ∈P)
[9] Q←(NM)/Q
[10] SSS←(+/[1] T[Q;3])-T[ρM;3]
[11] SDF←(+/[1] T[Q;2])-NE+T[ρM;2]
[12] MS←SSS÷SDF
[13] VAR←((ρT[;1]),4)ρ0
[14] VAR[;1]← $\iota(\rho T[;1])$ 
[15] VAR[;2]←T[;1]
[16] VAR[P;3]←(T[P;4]÷MS)
[17] VAR[;4]←T[;2]
[18] □←VAR←MM/VAR
[19] 'ERROR SUM OF SQUARES IS ';SSS
[20] 'ERROR MEAN SQUARE DF IS ';SDF
[21] 'ERROR MEAN SQUARE IS ';MS
V
```

The pool program is used to pool the sums of squares and degrees of freedom for the factors whose effects were not significant. The matrix T from the ANOVA program is required for this calculation. The first four lines ask for the row numbers of the significant factors and the number of error terms, i.e. the number of corrected approach ratios to be entered from the terminal. Line 5 calculates the number of rows in matrix T and generates an index of this number of rows. Line 6 inserts 1 for the significant

factors and 0 for the non-significant factors. Line 7 inserts 0 for the significant factors and 1 for the non-significant factors. The non-significant factors are identified in line 8 and compressed in line 9 to remove the zeroes. The sum of squares, sum of the degrees of freedom and the mean square are calculated in the next three lines.

Line 13 enters a blank array and the following four lines enter the row numbers, identification variance ratio and degrees of freedom into the printed array, VAR.

Quotes and the numerical values of error sum of squares, error mean square degrees of freedom and error mean square are printed by the last three lines.

APPENDIX G


```

C      TAINT REDUCTION RATIO PROGRAM
C
C      THIS PROGRAM CALCULATES THE TAINT REDUCTION RATIOS FOR STEP
C      FLASH, CROSS FLOW AND COUNTER CURRENT PROCESSES OF 1,2,...,N STAGES,
C      FOR A RANGE OF STEAM FLOWS AND FAT CONTENTS. IT IS ASSUMED THAT AN
C      EQUILIBRIUM DISTRIBUTION OF TAINT COMPOUND BETWEEN THE FAT PHASE,
C      THE AQUEOUS PHASE AND THE VAPOUR PHASE IS ACHIEVED IN EACH STAGE.
C      THE DATA REQUIRED FOR EACH COMPOUND ARE K (THE VAPOUR PHASE AQUEOUS
C      PHASE EQUILIBRIUM COEFFICIENT) AND D (THE FAT PHASE AQUEOUS PHASE
C      DISTRIBUTION COEFFICIENT).
C
C      INITIALIZE, DIMENSION AND DEFINE VARIABLES.
C
C      REAL K
C      DIMENSION F(10),S(10),QI(10,10)
C      REAL*8 BLANK,STEP,FLASH,CROSS,FLOW,COUNT,CURR,TYPE,CCMP(3)
C      READ(5,908)BLANK,STEP,FLASH,CROSS,FLCM,CCUNTR,CURR
C      908 FORMAT(7A7)
C
C      READ NUMBER OF STAGES, AND THE FAT CONTENTS AND STEAM FLOW RATIOS.
C
C      READ(5,909)NMAX,NFAT,NSSTEAM
C      909 FORMAT(3I2)
C      READ(5,907)(F(I),I=1,NFAT)
C      READ(5,907)(S(J),J=1,NSSTEAM)
C      907 FORMAT(10F4.2)
C
C      READ DATA CARD FOR COMPOUND(M) (I.E. NAME OF COMPOUND, K, D)
C
C      200 READ(5,910,END=999)COMP,K,D
C      910 FORMAT(3A8,2F6.2)
C
C      CALCULATE RA FOR COMPOUND(M), FAT(I).
C
C      DO 451 I=1,NFAT
C      RA=1+F(I)*(D-1)
C      PCFAT=100*F(I)
C
C      WRITE TITLE AND COLUMN HEADINGS FOR TABLE OF TAINT REDUCTION
C      RATIOS FOR COMPOUND(M), FAT(I).
C
C      WRITE(6,911)CCMP
C      911 FORMAT(1,'T1',T41,'TAINT CONCENTRATION REDUCTION RATIOS FOR',T51,3A8)
C      WRITE(6,906)K,D,PCFAT
C      906 FORMAT(T51,'K = ',F6.2,' ; D = ',F6.2/T45,'FAT CONTENT OF FLUID =
C      K ',F4.1,' PERCENT')
C      WRITE(6,912)
C      912 FORMAT(/T10,8(' '),T20,9(' '),T31,59(' '))
C      WRITE(6,913)
C      913 FORMAT(T10,'TYPE OF NUMBER',T52,'STEAM FLOW RATIO',T31,59(' '
C      K'))
C      WRITE(6,914)(S(J),J=1,10)
C      914 FORMAT(T10,'PROCESS OF STAGES',T29,10F6.1/' ',T10,8(' '),T20,9('
C      K_'),T31,10('----- '))

```

```

0026 C
0027 C CALCULATE TAINI REDUCTION RATIOS FOR ALL STEAM FLOW RATIOS, FOR
0028 C STEP FLASH PROCESSES OF 1,2,.....NMAX STAGES
0029 C
0030 C
0031 DO 250 J=1,10
0032 DO 250 N=1,NMAX
      V=S(J)/N
      CIJN=I
      DO 249 L=1,N
249      CIJN=CIJN*(RA+(N-L)*V)/(V*K*RA+(N-L)*V)
250      OI(J,N)=OIJN
C
C DETERMINE POSITION OF *STEP FLASH* IN FIRST COLUMN AND WRITE
C THE TAINI REDUCTION RATIOS FOR THE STEP FLASH PROCESSES.
C
C DO 251 N=1,NMAX
      TYPE=BLANK
      ITYPE=2*NMAX-4*N
      IF(ITYPE.LT. 3.AND.ITYPE.GT.-1)ITYPE=STEP
      IF(ITYPE.LT.-1.AND.ITYPE.GT.-5)ITYPE=FLASH
251      WRITE (6,916)ITYPE,N,(OI(J,N),J=1,10)
252      WRITE(6,917)
253      FORMAT(//)
C
C CALCULATE TAINI REDUCTION RATIOS FOR ALL STEAM FLOW RATIOS, FOR
C CROSS FLOW PROCESSES OF 1,2,.....NMAX STAGES.
C
C DO 350 J=1,10
0042 DO 350 N=1,NMAX
0043 G=RA/(RA+S(J)*K/N)
0044 OIJN=I
0045 DO 349 L=1,N
0046 CIJN=CIJN*G
0047 OI(J,N)=OIJN
0048 C
C DETERMINE POSITION OF *CROSS FLOW* IN FIRST COLUMN AND WRITE
C THE TAINI REDUCTION RATIOS FOR THE CROSS FLOW PROCESSES.
C
C DO 351 N=1,NMAX
      TYPE=BLANK
      ITYPE=2*NMAX-4*N
      IF(ITYPE.LT. 3.AND.ITYPE.GT.-1)ITYPE=CROSS
      IF(ITYPE.LT.-1.AND.ITYPE.GT.-5)ITYPE=FLOW
351      WRITE (6,916)ITYPE,N,(OI(J,N),J=1,10)
352      WRITE(6,917)
C
C CALCULATE TAINI REDUCTION RATIOS FOR ALL STEAM FLOW RATIOS, FOR
C COUNTER CURRENT PROCESSES OF 1,2,.....NMAX STAGES
C
C DO 450 J=1,10
0056 B=S(J)*K/RA
0057 SUMB=I
0058 DO 450 N=1,NMAX
0059 SUMB=1+SUMB*B
0060

```

```
0061      450 OI(J,N)=1/SUMB  
C      DETERMINE POSITION OF COUNTER CURRENT IN FIRST COLUMN AND WRITE  
C      THE TAINT REDUCTION RATIOS FOR THE COUNTER CURRENT PROCESSES.  
C  
0062      DO 451 N=1,NMAX  
0063      TYPE=BLANK  
0064      ITYPE=2*NMAX-4*N  
0065      IF(ITYPE.LT. 3.AND.ITYPE.GT.-1)ITYPE=COUNTR  
0066      IF(ITYPE.LT.-1.AND.ITYPE.GT.-5)ITYPE=CURR  
0067      451 WRITE (6,916)ITYPE,N,(OI(J,N),J=1,10)  
C  
C      READ THE DATA CARD FOR THE NEXT COMPOUND.  
C  
0068      GO TO 200  
0069      999 STOP  
C  
C      THE FIRST DATA CARD (FORMAT(7A7)) READS  
C      (      STEP      FLASH      CROSS      FLOW      COUNTERCURRENT)  
C  
0070      END
```

TOTAL MEMORY REQUIREMENTS 000CF4 BYTES
22:59.30 2.965 RC=0