Operating conditions for optimum behavior of a continuous countercurrent, countergravity extraction plant

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INTRODUCTION

Seeds from plants of various kinds have always been used as a food. Prior to the discovery of America the standard European crops were wheat, oats, barley, millet and rye. Of these wheat was the staple used extensively by all European peoples. With the discovery of America came new kinds of food such as Indian corn or maize, potatoes, and squash.

Prior to this time there was growing in northern China, and particularly in the province of Manchuria, a plant known by the name of "Soja bean," the seed from which was the primary food in that part of the world. The only vegetable oil known to the people who inhabited this portion of the earth was that which was obtained from soybeans. Both the oil and the beans were prepared in many forms for human consumption. The plant, today known as the soybean, is now grown all over the world, and its seeds are becoming increasingly important.

Soybeans were first brought into this country in 1804 for use primarily as a stock food. In 1908 fluctuation in the price of linseed oil, caused by a wilt which infected the flax plant, brought about the investigation of soybean oil as a substitute. Since then the rapid rise in soybean production has been one of the most striking agricultural developments in the United States. The production in the United States was 3,000,000 bushels in 1920; 18,527,000 bushels in 1934; and 211,500,000 bushels in 1942 (59).

There are several reasons for this increase in the production of
soybeans. The plant being a legume adds nitrogen to the soil, thus making it a valuable plant in crop rotation. It leaves the soil loose and in very good condition, and it will grow on almost any kind of soil. The seed of the soybean plant, because of its high oil and protein content, has become the basis of one of the most rapidly growing industries in the United States. At present more than fifty mills, including a few cottonseed oil mills, are processing soybeans; more than fifty concerns are manufacturing soybean food products and soybean flour; and hundreds of factories are producing various industrial materials containing soybean products.

Soybean oil after being refined is an excellent edible product. This was its most important use in 1935. Over 50,000,000 pounds, or about fifty per cent of the total produced, were used for "compounds" and lard substitutes. The paint and varnish industries were the next major users, consuming a total of 13,000,000 pounds in 1935 (1). Soybean oil has not yet been developed to the point where it can completely replace linseed oil in paint. According to Herberer (27), it plays a definite role of its own in that it forms a more elastic and tougher paint film without affecting the drying time or strength.

The meal, or material left after the bean has been freed of oil, contains a high percentage of protein matter, making it valuable as a substitute for meat scraps in all kinds of stock and poultry foods. The plastic industry is now using soybean meal extensively without further refining (57). The protein is taken from the meal and put to many uses, including sizing of paper and gluing of plywood and other materials (45).
Soybeans contain 1.5 to 3.0 per cent of the phosphatide lecithin, making it an excellent source of this material. Lecithin is valuable as a food and as an emulsifying agent, thus making its recovery of commercial significance.

There are two general methods of separating the oil from the meal: the pressure extraction method and the solvent extraction method. In a comparison of the pressure extraction system and solvent extraction system Meyerweissflog (42) makes the statement that no pressure method used to date has succeeded in lowering the oil content of the bean below five per cent. Thus at least twenty-five per cent of the original oil is residual with the meal and is sold at approximately one sixth the price of the oil, a decided disadvantage from an economic standpoint. The solvent extraction system is capable of decreasing the oil content to five tenths of one per cent, and has the added advantage of lower power costs than the pressure system.

The solvent extraction system also has its disadvantages, the most outstanding of which is the explosiveness of the solvents which have been used. Two disastrous explosions occurred in 1935 in soybean oil extraction plants where petroleum solvents were used as an extraction medium. After only six hours of operation, a plant near Momence, Illinois, exploded, killing two persons and injuring two others (6). The Chicago plant of the Glidden Company was the second to explode, causing the death of eleven people and injuring 45 (6). The loss of property in these two explosions was also a serious item, amounting to millions of dollars. Another major loss was the effect upon the soybean industry. Progress
in this industry was naturally slowed down because of these disasters. Inexperienced operators in charge of the Momence plant may have been partly responsible for that accident, but in the Glidden plant all usual precautions had presumably been taken. Thus it appears that an explosive solvent is an ever present danger.

Some work has been done on the use of nonexplosive solvents, particularly chlorinated hydrocarbons, for soybean oil extraction. Carbon tetrachloride is a good solvent for the oil, but has the undesirable property of corroding metal (55). Ethylene dichloride is a good solvent. It causes no corrosion but is slightly flammable. Trichloroethylene is nonflammable, practically noncorrosive, and an excellent solvent. It, therefore, offers great possibilities in the field of solvent extraction. The operating conditions for the optimum behavior of a continuous, countercurrent, countergravity extraction plant using trichloroethylene as a solvent will be discussed in this paper.
Pressure Methods

According to Sweeney and Arnold (55), who give a detailed description of pressure methods of extraction, primitive oil presses to remove oil from soybeans have been in operation in Manchuria for centuries although in more recent years the hydraulic press has been utilized for this purpose. The principle is the same in all cases, involving first, the preparation of the seed by grinding and cooking and second, the forcing out of the oil from the ruptured oil cells by the application of pressure.

The hydraulic press method of removing the oil from the bean is very inefficient and costly and has been replaced in part by the expeller method. The expeller itself works on a principle similar to that of a meat grinder. The beans are subjected to the end thrust of a powerful screw or worm, mounted on a steel shaft which rotates within a slotted conical cage. Usually several worm flights are used, being so arranged as to give a gradually increasing pressure as the beans move toward the outlet. The oil is forced out through the slots in the cage, and the meal is forced through the conical end of the cylinder into a hopper.

As has been pointed out (42) neither the expeller nor the hydraulic press reduces the oil content of the meal below five per cent. This fact, coupled with the great labor expense of the hydraulic press method and the high power requirements of the expeller method, indicates that
perhaps all pressure methods will be superseded by solvent extraction plants.

Both Sweeney and Arnold (55) and Meyerweissflog (42) state that solvent extraction is a cheaper method of removing the oil from the bean than pressure methods. Both oil and meal produced by the extraction method are equal in quality to those produced by pressure methods.

The Solvent Extraction Systems

Volatile solvent extraction began in 1855 (9) when a French patent was granted to Deiss, for the use of carbon bisulfide in the removal of grease from bones and wool. The following year Deiss added additional claims to his patent covering extraction of oil from any oil bearing seed by use of the same solvent. The discovery of petroleum offered new solvents to the world, and in 1864, Irvine, Richardson, and Lund first used gasoline as a solvent for oils. Hydrocarbons have been used extensively since that time for the extraction of oil from oil bearing materials.

Solvent extraction systems have undergone considerable development since the time of Deiss and many devices of varying kinds have been used. The simplest of these devices is a covered kettle in which the material to be extracted is placed. It is covered with solvent and agitated for some time. The solvent oil mixture is then drained from the kettle and the solvent evaporated, leaving an oil residue. The meal is washed with fresh solvent, and when sufficiently free from oil, is partially freed
from its solvent by heating. Any solvent residual in the meal after heating is removed by steaming.

Several systems have been patterned after the simple one described above. Sweeney and Arnold (55) gave detailed descriptions of the stationary, large scale Soxhlet, and the rotary systems, which are modifications of the above method.

The stationary system consists of a large drum, suitable agitators, solvent inlet, and an outlet for the solvent oil mixture. The extractable material is placed in the drum and solvent circulated over it until the extraction is complete. The solvent is separated from the oil by distillation and from the meal by heating and steaming. The large scale Soxhlet system allows the solvent to vaporize, condense, flow through the extractable material and then into the vaporizing chamber in a manner similar to that in the laboratory Soxhlet extractor used for determination of oil or fat. In the rotary system, horizontal, steam jacketed rotating drums are used to secure agitation. The drums are charged periodically, through man holes, with beans and solvent, and then rotated to produce a mixing action. When equilibrium has been reached the oil solvent mixture is drained off and new solvent added. This process is repeated until the desired oil content in the meal is reached. The oil and solvent are then separated by distillation and the solvent removed from the meal by heating and steaming or heating in a vacuum.

Satow (51) used the rotary drum type of extractor satisfactorily on soybeans after designing a new steam- and vacuum-tight stuffing box. Previously this extractor had not been successful because the stuffing boxes had a tendency to leak.
Whiton (65), in 1926, suggested that the Soxhlet type extractor should be used for cotton seed oil extraction. He proposed that an extractor of not more than 5,000- or 6,000-pound capacity be used. He gave the following advantages for this type of extractor: no moving parts, no pumps or agitators, proper size of machine, operation independent of the other extractors, no expensive technical supervision, and in general the most practical type of extractor. He predicted that within the next ten years many cottonseed oil plants will be using this type of extractor.

Contrary to Whiton's prediction the increasing number of uses for vegetable oils, especially soybean oil, in the last decade has necessitated the adoption of more efficient methods of extraction. The continuous method has, therefore, come into existence and will probably displace all other methods, especially for large scale operation.

Richert (49) says that the pioneers in soybean oil extraction were the Hansa mills in Hamburg, Germany. They have continually increased their capacity until they now process as high as 1,200 tons per day, an amount equal to the entire 1934 crop of soybeans in the United States. It is obvious that a continuous system is necessary for capacities of this magnitude.

The extractors at the Hansa mills are of the continuous counter-current type and the method used is known as the Hansa Mills system. The system consists of a series of baskets fastened to an endless rotating chain in a large tank. The baskets always remain upright until they are tilted or tipped. The baskets filled with beans pass down through the
solvent, which is contained in one side of the tank, and up the other side where they are met by fresh solvent. The solvent oil mixture is removed from the system at the point where fresh beans enter. The meal is dumped from the baskets by means of a trip and is conveyed through a series of driers which remove the solvent. The Central Soya Company is operating an extractor of this type at Decatur, Indiana.

The Boehm system of extraction (30) consists of a set of vertical extractors so arranged in stages that one is above the other in a joint casing. The extractable material lies at the bottom on a sieve and the solvent is allowed to trickle through it. The stages are so arranged that each may dump its charge of beans into the one below and receive another charge from the one above. The beans enter the system at the top, pass down through and are removed at the bottom. The system is made countercurrent by an arrangement of pumps and pipe lines which pumps solvent, rich in oil, over fresh material, and fresh solvent over material nearly exhausted of oil. The solvent and oil are separated in the usual manner.

Flummerfelt (55) patented a process for solvent extraction. Flaked beans pass through a conveyor into a tank containing solvent. The meal is withdrawn from the other end of the tank by a screw conveyor through which fresh solvent is flowing, thereby making the system countercurrent. The meal is then passed through a drying chamber where the solvent is vaporized, and the oil solvent mixture is processed in the usual way.

The Ford system of extraction was described by Taylor (57). The extraction chamber consisted of a long pipe one foot in diameter. It
sloped on a ten degree angle from the horizontal, and was lower at the
feed end. The material movement was made positive by the use of a
screw conveyor. The solvent entered at a point approximately half way
between the two ends of the pipe, flowed countercurrent to the beans and
left at a point near the bean entrance. Only a part of the inclined tube
was used for extraction space; the remaining portion was steam jacketed
and used as a drier. The partially dried meal passed from the high end
of the extractor into a steamer where the final traces of solvent were
removed. Two of those units are in operation at Saline, Michigan, each
having a capacity of six tons per 24 hours. Two are in operation at
Milan, Michigan, having the same capacity, and two are in operation at
the River Rouge plant in Dearborn, Michigan, each having a capacity of
25 tons per 24 hours.

Righhouse (8) described another continuous countercurrent extraction
system, in which a vertical pipe, fitted with a screw conveyor, passed
down through a vertical tank. The extracted material left the system
through this screw. The tank was kept about half full of solvent at all
times. The beans were fed in with a second screw conveyor and allowed to
fall down through the solvent by gravity, then removed by the vertical
screw. The fresh solvent was fed in through the center pipe, making the
system countercurrent.

Meyerweissflog (12) states that the best known system in operation
in the United States was the Hildebrandt system. The Archer-Daniels-
Midland Company, of Minneapolis, Minnesota, has one unit in operation at
its Chicago plant and the Glidden Company of Cleveland, Ohio, has another
in operation at its Chicago plant. The Clinton Company of Clinton, Iowa, also has one in operation extracting oil from corn germs. In this system the flaked beans are fed in through one leg of a U-shaped cylinder by means of a perforated screw conveyor. Solvent is fed in through the other leg and flows countercurrent to the beans, leaving the extractor near the point of bean entrance. The meal is heated to remove the solvent, and the oil-solvent mixture is separated by means of distillation.

Extraction Solvents

The first solvent used in the extraction industry, carbon-hisulfide, was used extensively in the southern part of Europe (2) until development of petroleum gave rise to new solvents. Sweeney and Arnold (55) classify solvents used for extraction into three groups: flammable, semiflammable, and nonflammable.

Flammable solvents are petroleum ether, gasoline (both ordinary varieties and specially refined solvent gasolines), ether, benzol, carbon bisulfide, and acetone. Ethylene dichloride is an example of semiflammable solvents since it ignites only under conditions which are not usually met in practice. In this class there are also numerous mixtures of flammable solvents with nonflammable ones; the latter being added in such portions as to make the mixture not readily flammable. The nonflammable solvents are principally those containing chlorine, such as trichloroethylene, carbon tetrachloride, and trichloroethane.

A special fraction of gasoline is the most common solvent used today. Ordinary gasoline, used as motor fuel, is usually unsatisfactory for extraction purposes because of its wide boiling range, which makes it difficult to remove the solvent from the oil and meal.
The following comparison of the extraction powers of various solvents, based on carbon bisulfide having a value of 100, is given by Satow (51):

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Extraction Power</th>
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<tr>
<td>Carbon bisulfide</td>
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</tr>
<tr>
<td>Ether</td>
<td>93.61</td>
</tr>
<tr>
<td>Gasoline (B. p. - 75° C.)</td>
<td>81.28</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>63.82</td>
</tr>
</tbody>
</table>

He does not recommend the use of carbon bisulfide because of its flammability and high cost, but recommends gasoline because of its cheapness.

The value of various solvents in soybean oil extraction was studied by Webber (64). He stated that methyl, ethyl, iso-propyl, n-butyl, and amyl alcohols gave high yields of oil, were easily removed from the oil, and extracted the oil easily. Their disadvantages were that they are flammable and toxic in varying degrees. He also stated that they were expensive, especially those boiling at a temperature higher than ethyl alcohol, and that they tended to give a dark oil. Dichloroethylene and trichloroethylene were found to be good extracting solvents and were recommended because of their nonflammability and the good grade of oil which they produced.

Fergason (19) studied the use of trichloroethylene for the extraction of soybean oil and found it to be a very good solvent. It was nonflammable, extracted the oil more rapidly than any other solvent tried, did not extract extraneous matter, could be removed completely from the meal, and did not corrode steel.
Burd and Millor (7) experimented with a mixture of water and butanol as a solvent for soybean oil. They found that it would dissolve the oil when hot, and then upon cooling separate into two phases, one of which was rich in oil. They reported that a good grade of oil was obtained by this method. The mixture was not toxic and was not as flammable as gasoline.

Li, King and Lin (33) stated that oil and fat in the soybean can be extracted by chloroform, ether, carbon bisulfide, carbon tetrachloride, gasoline, benzene, or a mixture of gasoline and alcohol. The mixture is the best solvent.

Since trichloroethylene was used as a solvent in these laboratory studies a discussion of its history as a solvent will be given.

An article appeared in Chemical Age (3) discussing the properties of chlorinated hydrocarbons. The following quotation from the article concerns trichloroethylene:

Trichloroethylene is the most important member of the chlorinated hydrocarbon group. It is almost the ideal solvent and may therefore be taken here to illustrate the commercial uses of the whole series, which have been manufactured on a large scale since 1903. Trichloroethylene has been used successfully for oil extraction for 20 years, during which time its manufacture has been greatly improved.

A disadvantage which has been suggested by some writers and experimenters is that trichloroethylene is somewhat toxic. Stuber (54) published an article on this subject reviewing 284 cases of poisoning, 26 of which were fatal. Even though the author points out that light forms of poisoning may be quite common and will only rarely be reported, serious accidents are not frequent and all too often are the result of careless
handling and poor installation of machinery and inadequate ventilation. Furthermore, it should be emphasized that in many instances the poisoning resulted from handling a mixture of which trichloroethylene was only one constituent; and finally it should be pointed out that up to a few years ago the purity of the technical product was not of as high a standard as it is at present.

Since 1931 seven additional fatal accidents have been reported in the literature. Koch (33) reported a case of an individual who died from narcosis caused by gross negligence in handling the material. Pfreimtter (48) reported a similar case where a workman handled "E-Stoff," a material containing trichloroethylene, in a tank without adequate protection. Vallee and Leclercq (60) reported a fatal accident in which a workman inhaled trichloroethylene vapors from a paint for five hours on two consecutive days. He showed symptoms of "intoxication" on the evening of the second day and was later found dead by his neighbors. This patient, according to the authors, had been suffering previously from injuries of the liver, so that the autopsy findings which showed congestion and edema of the lungs, multiple small hemorrhages of the pleura, and fatty degeneration of the liver, cannot be credited exclusively to the effects of trichloroethylene. It also appears not to have been definitely established what volatile compounds other than trichloroethylene may have been in the paint. Three additional fatalities were reported by Holstein (20). Two of these patients died as a direct effect of narcosis, whereas the third, a woman, died from septicemia as a consequence of an infection of the burns caused by trichloroethylene.
Hansen (26) reported one fatal accident from the inhalation of trichloroethylene vapors, developed at elevated temperatures in a closed room. The pulmonary edema observed in this case was not very marked and trichloroethylene could be isolated from different organs. Since this patient was suffering from extensive tuberculosis, the question whether or not this was a contributing factor is open to discussion.

Matruchot (37) offered a list of materials and attempted to classify them as to relative toxicities. He gave trichloroethylene a rating of one and on it bases the toxicity of the other solvents, using that figure as the basic unit. The list follows:

<table>
<thead>
<tr>
<th>Material</th>
<th>Rating</th>
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<tr>
<td>Trichloroethylene</td>
<td>1.0</td>
</tr>
<tr>
<td>Perchloroethylene</td>
<td>1.4</td>
</tr>
<tr>
<td>Dichloroethylene</td>
<td>1.5</td>
</tr>
<tr>
<td>Dichloroethane</td>
<td>1.6</td>
</tr>
<tr>
<td>Benzene (pure)</td>
<td>2.5</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>2.6</td>
</tr>
<tr>
<td>Benzene (90%)</td>
<td>2.8</td>
</tr>
<tr>
<td>Chloroform</td>
<td>4.7</td>
</tr>
<tr>
<td>Trichlorethane</td>
<td>5.0</td>
</tr>
<tr>
<td>Tetrachlorethane</td>
<td>6.0</td>
</tr>
</tbody>
</table>

It is convenient to group the solvents, by consideration of their physiological action into classes of varying degrees of danger in a way similar to that in which the degree of flammability has been classified by flash point limits. Attempts have been made to attain this end from several points of view. A classification of this type should be studied
very carefully before placing too much confidence in its accuracy. One solvent may be an irritant, one a narcotic and yet another one causing functional injury to the nervous system. Other solvents might produce some combination of the above physiological actions. It is difficult to group the above solvents into a comparative classification, unless they are classified according to the type of action on the organism. In this event one product would have to be placed in several classes, and one would occupy different degrees of importance in each.

It appears, therefore, that the acute toxic action of trichloroethylene consists mainly in a depression of the central nervous system which may be of short duration followed rapidly by complete recovery but which may easily become fatal if the patient is not immediately removed from exposure. Eichert (18) reported two cases of acute medicinal trichloroethylene poisoning which showed all symptoms of an acute psychosis with rapid and complete recovery.

Carrieu (13), Nuck (44), Pfreimteter (48) and Holstein (29) have reported that trichloroethylene, if handled improperly, is liable to cause injury to the skin, resulting in irritation and even blister formation. Oettel (46) studied the irritation effect of dichloroethylene and trichloroethylene on the human skin. He found that after 5 minutes contact, both caused slight burning which reached its maximum after 10 minutes and after an additional 5 minutes anesthesia became manifest. Exposure for 5 hours caused small blisters on the edge of the exposed zone. It should always be remembered that trichloroethylene is an excellent fat solvent, and that it will remove the protective fat film
from the skin and render the latter susceptible to secondary skin infections.

Several nonfatal cases caused from handling trichloroethylene reported since 1929 are shown in Table 1.

Dhers (16) pointed out that poisonous symptoms are noticed after several hours of exposure, and that the vapor concentration determines whether the symptoms are mild or acute. He said that the vapors give a pleasant sensation and may establish a habit making workers reluctant to report the symptoms. Other effects of trichloroethylene poisoning have been reported such as: an abnormally tired feeling, headache, giddiness (47), and a fading of the pupils of the eyes (34).

According to Shaw (5) trichloroethylene is a narcotic similar to chloroform. There is no evidence that a man could be poisoned by taking small doses, but it could have an acute effect if taken in large amounts. In 1933 the Imperial Chemical Industries, Ltd., made tests and warned plant owners not to permit smoking. If air containing 0.1 of one per cent of trichloroethylene vapor was drawn through the lighted end of a cigarette, the temperature of the lighted end being 600° C., the air inhaled would contain three parts per million of phosgene gas. Shaw stated that he thinks twenty-five parts per million of phosgene is fatal. Rats showed no signs of poisoning when subjected to the vapors; in fact they gained weight.

Van Thomsche (61) stated that light and heat decompose trichloroethylene, producing phosgene, carbon monoxide, and hydrochloric acid, the reaction becoming violent in the presence of magnesium turnings, aluminum turnings, or alkalies. According to him the products of the
<table>
<thead>
<tr>
<th>Year</th>
<th>Author</th>
<th>Operation</th>
<th>No. of cases</th>
<th>Symptoms</th>
</tr>
</thead>
<tbody>
<tr>
<td>1933</td>
<td>Christensen (14)</td>
<td>Cleaning shoes.</td>
<td>1</td>
<td>Inhaled trichloroethylene vapors 1/2 hour. Pulmonary disturbances. Complete recovery after 5 hours.</td>
</tr>
<tr>
<td>1933</td>
<td>Roholm (50)</td>
<td>No details</td>
<td>4</td>
<td>No details given in abstract. Emphasizes effect on basal nerves.</td>
</tr>
<tr>
<td>1934</td>
<td>Persson (47)</td>
<td>Cleaning metal parts.</td>
<td>2</td>
<td>Dizziness, unsteady gait, paresthesia of feet and hands, headache; symptoms first relieved in fresh air, later became persistent. Recovery after 6 to 6 1/2 months.</td>
</tr>
<tr>
<td>1935</td>
<td>Kunz and Isenschmidt (34)</td>
<td>Diamond worker.</td>
<td>1</td>
<td>Exposure for 16 months. Retrobulbar neuritis, sensory disturbances in hands and arms, absence of patellar and achillea reflexes, slow recovery.</td>
</tr>
<tr>
<td>1935</td>
<td>Gilbert (22)</td>
<td>Workman exposed for several years.</td>
<td>1</td>
<td>After single gastric attack, severe abdominal pain, nausea, vomiting, dyspnea, retrosternal pain, cough, bronchitis and reduced sense of smell and taste and abolition of corneal reflexes. Complaints gradually disappeared.</td>
</tr>
</tbody>
</table>
reaction dissolve the lipoids of the nervous system giving an effect similar to that of chloroform but trichloroethylene itself is less toxic than benzene and saturated hydrocarbons. There is a difference in the narcotic and toxic doses but it can produce death.

Eichert (18) in discussion of trichloroethylene intoxication following large and prolonged doses, reported a failure to find any of the more serious symptoms attributed to industrial trichloroethylene poisoning. This tends to confirm the general belief that the toxic effects noted among industrial workers may be attributable to some contamination in the solvent. It cannot be overlooked, however, that inhalation of trichloroethylene has caused severe and fatal accidents recognizable by certain narcotic symptoms, irritation of the gastrointestinal tract, injury to the skin, and development of habituation. These effects may be minimized by careful use and careful handling of the product.

In case of poisoning by trichloroethylene, characterized by depression of the central nervous system or narcosis, the patient should be transferred to fresh air. Rest is of paramount importance. In cases where respiration has ceased, artificial respiration with oxygen containing 10 per cent carbon dioxide should be started immediately. If the patient is still breathing, the administration of pure oxygen is indicated. Irritation of the gastrointestinal tract should be treated symptomatically by giving a bland diet and demulcents, such as starchy soup.

As soon as there are any indications that a workman is developing a craving for exposure to trichloroethylene, he should be transferred to some other job.
Stockman (53) stated there have been several cases of cattle poisoning by extracted soybean meal. He said there was ample evidence that naphtha-extracted soybeans do not cause poisoning; therefore he concluded that the soybean meal which caused the death of the cattle must have been extracted with trichloroethylene. He admitted, however, that trichloroethylene was not poisonous when given to cattle in large doses (one to three ounces) over long periods of time. His conclusion was (a) that the products obtained by heating the trichloroethylene might have been poisonous, but this was doubtful, (b) that the trichloroethylene, when heated in contact with the soybeans, may have reacted with them producing a poisonous product, or (c) that the trichloroethylene was impure and contained poisonous materials.

No other literature was found mentioning the fact that cattle were killed by trichloroethylene extracted meal. It is the opinion that Stockman had no grounds for his statements, and it is seriously doubted that cattle were ever killed by feeding meal extracted with trichloroethylene. As far as is known, no meal extracted with trichloroethylene has been sold on the market.

Another disadvantage of trichloroethylene is its slightly corrosive properties. Sweeney and Arnold (55), however, state that this does not seem to be a prohibiting factor in its use as a solvent. Laboratory tests show that it would take over thirty years for it to appreciably affect an extractor.

S. S. Drozdov and N. B. Drozdov (17) stated that iron, lead, and copper were appreciably corroded by trichloroethylene and dichloroethane, the corrosion by the liquids being greater than by the vapors. The
solvent, when refluxed over these metals in a Soxhlet extractor, caused
greater corrosion than when allowed to stand upon them. The presence of
water increased the corrosion. In all cases the iron was corroded most
and the copper the least. Trichloroethylene was more corrosive than
dichloroethane.

A patent owned by the Canadian Industries, Ltd. (12), states that
when a small amount of phenolic substance is added to chlorinated hydro­
carbons and particularly to trichloroethylene, the product is stabilized
and the development of an objectionable amount of acidity is prevented.

Recovery of Solvent from Oil and Meal

In any solvent extraction process, the solvent must be recovered
from both the meal and the oil.

The removal of the solvent from the meal has always been a more or
less standard procedure, consisting of: first, heating the meal to
drive off all vapors possible, and second, steaming to remove any residual
solvent. The water and solvent vapors are condensed and separated (57).
A system is described by Sweeney and Arnold (55) in which steam is blown
in under reduced pressure to prevent condensation on the meal. Satow
(51) used a vacuum to remove the vapors.

In the removal of the solvent from the oil the first step consists
of a preliminary evaporation to remove most of the solvent. The second
step, the removal of the remaining solvent, must be carefully carried
out because the oil is injured if the temperature used is too high.
The Ford process (57) utilized a simple flash type still, a solvent con-
demser, and a cooling tank. The still was heated by a steam jacket. The solvent oil moisture was carried in a steam jacketed coil to the top of the still where it entered and was vaporized completely. Ferguson (19) found that steam blown through oil having a trace of solvent, will completely remove the latter. Satow (51) passed dry steam directly into the concentrated solvent oil mixture, and found that the resulting emulsion could be separated by centrifuging.

Preparation of Material to be Extracted

Taylor (67) gave the following description of the flaking process used in the Ford plant:

The flaker itself operates on the principle of a meat grinder, the body being a section of twelve inch pipe containing a revolving steel core on which is a half inch raised spiral. The beans are forced out through an adjustable slit at one end. It is by varying the thickness of the flakes, that the residual oil content of the meal is controlled within a range of from 0.5 to 0.6 per cent.

According to an article in Chemical Age (2), maximum efficiency is obtained when the material to be extracted is in the form of thin flakes, shavings or threads.

All other continuous countercurrent extraction systems which are operating today use beans prepared by first cracking into small pieces by special rolls, followed by flaking in a set of hardened rolls. These flaking rolls are usually thirty to thirty-six inches in diameter.

For the experimental work at Iowa State College, soybeans have been flaked on ordinary crushing rolls.
EXPERIMENTAL

Purpose

The object of this research was to place in operation and operate continuously a continuous, countercurrent, countergravity solvent extraction plant. The laboratory data preliminary to the design of a commercial extraction plant for soybeans have been given by McCracken (39), and the design of this plant, based on the laboratory data, was given by Kircher (32).

Materials Used

Trichloroethylene

(a) The trichloroethylene used in this work was furnished by the E. I. duPont deNemours Company. It was a commercial grade extraction solvent having the following properties:

1. Chemical formula \( \text{CHCl}_3 \)
2. Molecular weight 131.38
3. Boiling point at 760 mm. \( \circ \text{C.} \) \( \circ \text{F.} \) 86.70 188.10
4. Freezing point \( \circ \text{C.} \) \( \circ \text{F.} \) -88.0 -126.4
5. Latent heat of vaporization (at boiling point) \( \text{B.t.u.} / \text{lb.} \) -103.1
6. Specific heat at 20° C. \( \text{Cal.} / \text{gram} / ^\circ \text{C.} \) or \( \text{B.t.u.} / \text{lb.} / ^\circ \text{F.} \) 0.223
7. Specific gravity at 20°/4° C.  
   1.462

8. Pounds per gallon at 20° C.  
   12.20

9. Vapor density (at boiling point and 760 mm.)  
   (gram/liter) 4.45  
   (lb./cu. ft.) 0.276  
   (air = 1) 4.53

10. Solubility in water at 25° C. (gram per 100 gram)  
   0.17

11. Infractive index N^20  
    1.4777

12. Vapor pressure at 30° C. (mm. Hg)  
    94.0

13. Flammability  
    None

14. Boiling range, 760 mm., °C.  
    86 - 87.5

15. No cloud at  
    -10° C.

16. Maximum water per cent by weight  
    0.002

17. Maximum acid per cent by weight as HCl  
    None

18. Maximum residue per cent by weight  
    0.002

19. Maximum color  
    Saybolt #24

20. Free halogen  
    None

(b) Trichloroethylene may undergo the following reactions:

1. When heated with resorcinol and a small piece of caustic potash,  
   a red zone is formed around the latter.

2. When heated with aniline and an alcoholic solution of potassium  
   hydroxide, a faint isonitrile-like odor is given off.

3. With alcoholic potassium hydroxide a clear solution is im-  
   mediately formed (as distinguished from tetrachloroethanes).

4. When heated with concentrated caustic alkali, glycolic acid and  
   traces of the spontaneously combustible dichloroacetylene are  
   formed; at higher temperatures hexachlorobenzene is formed.
5. According to Bruning and Schnetka (11) it can be detected by mixing two cc. of the test material with one cc. of pyridine and underlying this mixture with one cc. of a fifty per cent solution of sodium hydroxide. In the presence of trichloroethylene a red violet color is formed. This is, however, not stable; methyl and ethyl alcohol and tissue extracts interfere with the color formation.

Soybeans

The soybeans used were purchased by the Engineering Experiment Station of Iowa State College from farmers residing in Story and adjoining counties. No attempt was made to classify these beans as to type.

Extraction plant

The extraction plant was designed and constructed in the laboratories of the Chemical Engineering Department of Iowa State College. Details of the design are given by Kircher (32).

Methods of Analysis

Oil in bean

Extraction in a Soxhlet apparatus using petroleum ether is a standard method for the determination of oil in oil bearing seeds. The time of extraction varies with the material extracted. McKinney, Carter and Jamison (40) recommend a two hour extraction of the undried sample of
ground beans with petroleum ether, after which the sample is reground and extracted again for two hours. Cox (15) recommends the process of double extraction, but does not mention the solvent used.

Because of its nonflammable nature methylene chloride rather than petroleum ether was used as the solvent for all laboratory analyses. The extractions were performed in a Soxhlet extractor, the solvent siphoning over every two and one half minutes. Each sample was allowed to remain in the extractor until eight cycles were completed. The solvent was then distilled from the receiving flask, the oil weighed, and the per cent oil calculated on the dry basis.

**Oil in final meal**

The above procedure was also used for the determination of the oil in the finished meal.

**Moisture in the beans**

The percentage of moisture was determined by placing a known quantity of flakes in a constant temperature oven and drying to constant weight at 110° C.

**Moisture in finished meal**

The same method was used for the determination of moisture in the meal as was used for the determination of moisture in the beans.

**Oil content of miscella**

Known amounts of soybean oil and trichloroethylene were mixed together.
The specific gravities of these various mixtures were determined using a Westphal balance and these values were plotted against the percentage of oil in the same (Curve No. 1). With this curve it is very simple to determine the oil content of a solvent-oil mixture by merely checking the gravity and reading the percentage of oil from the curve.

**Trichloroethylene in oil**

Advantage was taken of the reaction given by Bruning and Schmetka (11) for the development of a quantitative method for the determination of small amounts of trichloroethylene in the oil. This reaction is a color reaction resulting when pyridine, 50 per cent sodium hydroxide, and trichloroethylene are mixed and heated. The color standards were made as follows:

Samples of light yellow expressed soybean oil were prepared containing 0.002, 0.006, 0.010, and 0.020 per cent by weight of trichloroethylene. One half gram samples of the prepared mixture were then treated with pyridine and sodium hydroxide in the following manner: A 3-gram sample was added to a 20 x 150 mm. pyrex test tube which contained 2 cc. of distilled water. Two cc. of Baker's pyridine and 2 cc. of 50 per cent sodium hydroxide solution were then added. The test tube was stoppered and shaken very vigorously for 15 seconds. The mixture was then heated in a boiling water bath for one minute, shaken vigorously for one and one half minutes, and then heated again for 30 seconds. Two clear layers were formed, the upper oil and pyridine layer being a reddish brown. The mixture was allowed to stand for four minutes. A color standard
was then made to match this color by mixing water solutions of cobaltous chloride and potassium dichromate which had been made slightly acid with hydrochloric acid. After the colors of the four samples had been matched the tubes were corked and sealed with paraffin.

The sample which contained the unknown amount of trichloroethylene was diluted with nine parts of light yellow expressed soybean oil. One half gram of the mixture was then added to a 20 x 150 mm. pyrex test tube and treated in the same manner as the samples from which the standards were made. The mixture was allowed to stand for four minutes and the color was then compared with the color standards by placing the test tubes in a box having slots in the front and back, making it possible to see through the oil and pyridine layer. The box was then placed in the light with a piece of white paper held at a forty-five degree angle to act as a reflector and background. If the color obtained was not within the range of the color standards, the dilution of the original sample was adjusted accordingly and the test repeated. Daylight is preferable to electric light for comparing the colors.

If the sample developed, on completion of the test, a color which matched that designated as "Number 1 color standard for determining trichloroethylene in extracted oil," then the initial oil undiluted contained 0.02 per cent trichloroethylene. On this same basis the respective values of the four color standards follow:

No. 1 color standard, color developed if undiluted oil contains 0.02% Tri.
No. 2 color standard, color developed if undiluted oil contains 0.05% Tri.
No. 3 color standard, color developed if undiluted oil contains 0.10% Tri.
No. 4 color standard, color developed if undiluted oil contains 0.20% Tri.
An alternative method which was used occasionally to check the color method was the Volhard method. The sample was decomposed with sodium and alcohol. The alcohol was then distilled after a water addition, and pure nitric acid was added in excess. The chloride was then precipitated with silver nitrate, filtered, and washed thoroughly. The filtrate was then titrated with potassium thiocyanide using ferric ammonium alum as an indicator. The amount of silver nitrate used in precipitating the chloride was noted and the percentage trichloroethylene calculated.

**Trichloroethylene in finished meal**

Color standards were made as follows: Water was saturated with trichloroethylene at 25°C. by occasionally shaking, over a period of several days, a mixture of the two in a closed container. When the water was saturated it contained 0.08 per cent trichloroethylene. When one part of the trichloroethylene saturated water was diluted with 159, 63, 31, and 15 parts of pure water, two cc. aliquots of the diluted solutions contained 0.01, 0.025, 0.05, and 0.1 mg. of trichloroethylene, respectively. A two cc. aliquot was placed in a 20 x 150 mm. pyrex test tube. Two cc. of a fifty per cent sodium hydroxide solution and two cc. of Baker's pyridine were mixed with the sample. The test tube was stoppered and shaken vigorously for fifteen seconds. It was then placed in a boiling water bath and heated with gentle agitation for one half minute. It was then shaken for ten seconds and a color standard immediately prepared to match the color formed. The color standard consisted of a mixture of cobaltous chloride and copper sulfate solution.
acidified with hydrochloric acid. Although perfect matching of the colors was not obtained, they were close enough for the purpose for which they were intended. The test tubes were then corked and sealed with paraffin.

One gram of the meal to be tested was placed in a one and one half by two end one half inch cloth bag. The bag was tied and lowered into a one liter distillation flask. The flask was attached to a twenty inch condenser, the end of which was connected to a Kjeldahl bulb that extended below the surface of five cc. of water in a small Erlenmeyer receiver. Fifty cc. of water were then added to the flask and heated. About forty-two cc. of distillate were collected. The distillate was shaken and a two cc. aliquot taken. This aliquot was treated in the same manner as the samples from which the standards were prepared. The color obtained was immediately matched with the standards. The distillate was diluted with distilled water if the concentration of trichloroethylene was too high to be measured.

If the color matched that of the Number one standard, then the two cc. aliquot contained 0.025 mg. of trichloroethylene. Since the amount of trichloroethylene in the aliquot sample was known, a value could then be calculated for the percentage of trichloroethylene present in the original sample. The four color standards for the analysis have the following values:

No. 1 color developed if two cc. aliquot contains 0.025 mg. Tri.
No. 2 color developed if two cc. aliquot contains 0.050 mg. Tri.
No. 3 color developed if two cc. aliquot contains 0.100 mg. Tri.
No. 4 color developed if two cc. aliquot contains 0.200 mg. Tri.
Operation of Extraction Plant

General discussion

This thesis is concerned with the operation of a continuous countercurrent extraction plant the design of which was given by Kircher (32). All sketches which appear in the body of the thesis are schematic, and are not drawn to scale. The photographs show both the finished plant and the plant during construction. Their purpose is to help give the reader a mental picture of the components of the extraction system.

Figure 1 represents a schematic diagram of the system as it was set up for the preliminary runs with the exception of feed hopper 1, which was installed later. The material flow will be traced at this time to acquaint the reader with the continuous countercurrent system.

Soybean flakes were fed into the feed hopper 3 and fell by gravity into extraction tube 4. Extraction tube 4 was fitted with a solid flight screw conveyor having a diameter of six inches and a pitch of one. The screw conveyor carried the flakes down tube 4 to junction 5 where they were transferred to upright tube 6 which was fitted with a solid flight conveyor, having a diameter of six inches and a pitch of one half. The extracted meal was then discharged from upright tube 6 through tube 7 by gravity and into drier tube 8. Drier tube 8 was fitted with a ribbon type conveyor having a diameter of six inches and a pitch of one. Drier tube 8 was steam jacketed and was fitted with sight glasses in the center. The meal was then discharged from drier tube 8 into drier tube 9 which was identical to tube 8 except that the ribbon conveyor in
tube 9 was of the opposite hand. The meal was then discharged from drier tube 9 into steamer 10 which was fitted with a conveyor similar to that in drier tube 8. From steamer 10 the meal was discharged as finished meal through rotary valve 11. Tube 10 was called the steamer because live steam was injected into the inner tube through the injection cocks 23.

The solvent vapor and the water vapor which were released in the drier and steamer sections escaped through the vapor lines 12 and 16. Vapor line 16 was connected to both the steamer 10 and drier tube 9. These vapors were then condensed in condenser 13. The liquids, trichloroethylene and water, were separated, by difference in gravity and by virtue of their immiscibility, in the water separator 14. The trichloroethylene thus separated flowed continuously through solvent line 15 to upright tube 6 where it flowed down tube 6 and through tube 4 countercurrent to the movement of flakes. The solvent oil mixture flowed continuously from the extractor tube 4 through fine slots at 2. It then went to evaporator 17 and still 18. Steam was injected into the still through steam inlet 24 and pure oil flowed from the oil outlet 25. The solvent and water vapors from both the evaporator and the still escaped through vapor lines 21 and 22, respectively, and were condensed in condenser 13. The solvent and water were separated in the normal manner and the solvent returned to the system while the water was led to the sewer.

The body of the thesis is composed of reports of the preliminary runs and twenty-one runs in the one hundred series. The reports of the runs numbered below one hundred were given by McCracken (39). These runs were made on smaller scale laboratory equipment.
Temperature readings, pressure readings, solvent control observations, flake feed control observations, and routine inspection of all pieces of equipment were made every thirty minutes while the plant was in operation. During "solvent loss" runs detailed records of all solvent added to or removed from the system were kept. It seems unnecessary to record all of those data sheets which would add many pages to the bulk of the thesis, but instead a condensed table is given. Each figure in Table 2 is an average of normal readings taken each thirty minutes during operation. Only the normal readings were averaged, the abnormal ones are discussed under each individual run.

Preliminary runs

The extraction equipment, when completely assembled, was operated for several hours. During this period neither beans nor solvent were fed into the system. Bearings, conveyors, drives, and all moving parts were constantly checked for overheating or binding. After the plant had operated successfully for several hours, dry soybean flakes were continuously fed to the extractor at the rate of sixty pounds per hour. The system contained no solvent since it was desired to check the material movement of dry flakes through the extractor, meal driers, and meal steamer. The feed to the extractor was increased to one hundred pounds per hour and no indication of plugging occurred in either the extractor or the driers. The speed of the screw in the top drier was 3 r.p.m., and in the bottom drier and the steamer 3.7 r.p.m. After several hours of successful operation it seemed safe to fill the system with solvent.
Table 2

Average Data of All the Runs Made

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<th>$T_2$</th>
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<th>$T_5$</th>
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*Experimenting with flow of gas in driers*
Nomenclature of Columns in Table 2

\[ T_1 \] - Temperature of vapor leaving evaporator.
\[ T_2 \] - Temperature of trichloroethylene entering extractor.
\[ T_3 \] - Temperature of vapor leaving second drier.
\[ T_4 \] - Temperature of vapor leaving steamer.
\[ T_5 \] - Temperature of oil leaving still.
\[ T_6 \] - Temperature of vapor leaving still.
\[ T_7 \] - Temperature of miscella in settling tank.
\[ T_8 \] - Temperature of vapor leaving top drier.
\[ T_9 \] - Temperature of miscella leaving extractor.
\[ T_{10} \] - Temperature of water to the condenser.
\[ T_{11} \] - Temperature of vapors leaving center of top drier.
\[ T_{12} \] - Temperature of meal leaving steamer.
\[ P_1 \] - Pressure on condenser, inches of water.
\[ P_2 \] - Pressure on steamer, inches of water.
\[ P_3 \] - Pressure on top drier, inches of water.
\[ P_4 \] - Steam pressure on driers.
\[ P_5 \] - Steam pressure on evaporator and still.
Run No. 101

The system was filled with solvent and all the conveyors placed in motion. The flakes were fed to the system at a rate of approximately 100 pounds per hour. Solvent was added from time to time until the system was sufficiently filled. The condensate resulting from the evaporation of the solvent from the meal and miscella flowed continuously from the water separator to the extractor as shown in Figure 1. The flakes used in this run were prepared on the small rolls in the Chemical Engineering Building and contained an appreciable amount of very fine material some of which approached colloidal dimensions. It was observed that the miscella flowing from the extractor contained fine particles of meal in suspension. Part of these solids was removed by an 80 mesh screen which was placed in box 2, Figure 1. The finer solids passed through the screen and entered the distillation equipment with the miscella. During the early part of the run the vaporizer and still seemed to be working satisfactorily. Dry steam was injected into the bottom of still 18, Figure 1, and the oil flowing from the still seemed fairly free from solvent. In the early stages of the run no bumping or foaming of the oil was observed in sight glasses, 19 and 20, Figure 1. The miscella flow was quite constant, and only a small amount of solvent was added to the system after the miscella started flowing. The meal was discharged from the steamer dry and practically solvent free. As the run progressed it became apparent that the miscella line between the vaporizer and still was beginning to fill with the fines from the miscella. An accumulation of fine material in the bottom of the still began to cause
foaming and bumping of the oil. The foam was seen in the sight glass above the still and was also observed in the sight glass above the evaporator. The oil leaving the still was dark in color and contained suspended solids. After about six hours of operation the flow of the oil from the still became very erratic due to the solids which were accumulating. This accumulation could not be removed during operation since no provision had been made to drain the still bottom. Near the end of the run it became increasingly noticeable that the still bottom was filling rapidly. The bumping and foaming became continuous and it was observed that solids were actually thrown into the vapor line above the still. After about eight hours of operation it was necessary to shut down since the still was foaming continuously and was not stripping the solvent from the oil. When the still was opened, it was found that the steaming jet at the bottom was nearly plugged with solids, and that the inside of the filming tubes was coated with solids. The pipe line connecting the evaporator to the still was also found to be partly closed.

It seemed apparent that some method of removing the solids from the solvent oil mixture was necessary if this type of distillation equipment were to be used. Since the solvent oil mixture from the extractor was very high in solvent content, and had a high specific gravity, it seemed advisable to attempt a separation of the solids from the solvent oil mixture after the oil had been concentrated in the evaporator. A settling tank was placed between the evaporator and the still, Figure II. This tank was supplied with an external heating coil in an attempt to keep the temperature of the mixture approximately 100° C. as it entered
the still. The tank was equipped with a conical bottom and a quick opening valve so that the solids could be drained away from time to time.

During the shutdown the evaporator and still were opened and examined. It was found that only one tube in the evaporator and two tubes in the still had been working properly. This faulty operation was caused by the variation in height of the tubes above the tube sheet. The ends of all the tubes were filed and leveled so that they all protruded a uniform height above the header plate. The ends of the filming tubes were then rounded to sharp edge to facilitate the flow of liquid into the tubes. While the two distillation units were open at both top and bottom, they were checked by pouring liquid through them. It was found that almost every tube was filming properly after this alteration. The distributing device was changed in the top of both the evaporator and still so that a more uniform flow might be expected. The tops of the tubes which were immediately below the liquid inlet were closed so that the solvent oil mixture could not short circuit through these few tubes. The evaporator and still were adjusted so that the tube sheets were horizontal when the units were vertical, thereby insuring maximum efficiency of distribution of the miscella down the filming tubes.

Run No. 102

The chief difficulty encountered in this run was that the ribbon conveyor in the top drier was not carrying the wet meal forward from the point of its entry. The wet meal, which entered the top drier from the extractor by gravity, would accumulate on the inside of the ribbon, or
open flight, and rotate with the screw instead of moving forward in the normal manner. On several occasions, during the course of an hour, this condition was observed. When this phenomena occurred the pipe connecting the extractor to the driers would fill with meal making it necessary to relieve this condition from time to time. After observing the action of the conveyor where the wet meal entered the top drier, (inspection could be made through a porthole in the drier) it was decided that improved operation might be obtained by installing several feet of solid flight conveyor where the meal entered. The plant was shut down temporarily until a few feet of solid flight conveyor could be obtained.

The ribbon conveyor (Photograph I) was removed from the drier and a section about five feet long cut away. This section was removed from the entrance end of the drier. Solid flight was installed and the conveyor reassembled in the drier.

While the unit was shut down temporarily the drive on the rotary valve at the end of the steamer was changed to a separate motor and drive, including speed reducer and jackshaft. It had previously been operated by a chain drive from the main drive shaft (Photograph II). About fifteen inch centers were used between the sprockets on the speed reducer and the valve. A three-sixteenth inch brass shear pin was placed through the sprocket on the valve shaft. The aleomite fixture was extended so the valve could be easily lubricated. It was thought that a drive having shorter centers would make the rotary valve operate more smoothly.

A sprocket was broken on the drier drive mechanism (Photograph III) when the top drier filled with wet meal. This sprocket was replaced and the unit was ready to continue the run.
Photograph I
Top drier showing ribbon conveyor.
Photograph II
Rotary valve showing original chain drive.
Photograph III
Drier drive mechanism.
The run was continued after the above changes were made. The flakes used were prepared on the small diameter rolls in the Chemical Engineering Building and contained powdered material and fines. The settling tank appeared to remove a part of the solids carried by the miscella, but it was observed that the liquid entering and leaving the still also contained fine meal in suspension. The finished oil was dark in color, but the taste was good and it seemed to be well stripped of solvent. The solids in the oil probably helped to give it a dark color. On standing the solids settled readily, leaving a clear oil. The meal leaving the steamer through the rotary valve was dry, light in color, and practically free from solvent. The rotary valve operated without serious trouble. The vaporizer and still worked satisfactorily, and no foaming or bumping occurred. The settling tank was not cleaned or drained during this run, the solids being allowed to remain in the bottom of the tank. There seemed to be no accumulation of meal in either the top drier or the steamer, and the section of solid flight conveyor caused a positive movement of the wet meal away from the point of entry. The bottom drier, however, showed an accumulation of meal. The plant was shut down after twelve hours of continuous operation because the supply of flakes was exhausted. The capacity of the small rolls which were being used was less than the capacity of the extractor and unless a supply of flakes was made in advance a long run could not be made.

The average feed of flakes per hour to the extractor was seventy pounds, and the average oil content of the miscella was twenty-five per cent (see Table 2).
Before the next run a two foot section of solid flighting was installed in the conveyor of the second drier at the point where the meal was discharged from the first drier to the second. Evidence of accumulation was noticed during the last run and it was thought that the solid flight would facilitate the movement of the meal away from the point of entry. A twenty inch section of solid flighting was also placed in the conveyor of the steamer at the point where the meal entered from the bottom drier. No evidence of accumulation had been noticed here, but since it had been observed in the two driers this precaution was taken.

Run No. 103

This run was started for the purpose of checking the conveyor action and material movement in the driers and steamer after the changes which were made at the termination of Run No. 102. The equipment ran continuously for five hours before the conveyor in the bottom drier plugged. Abnormal tension was first noticed in the drive chain. The material movement in the driers was checked, through sight glasses located in the center (see Photograph IV). It was observed that partially dried meal was accumulating in the upright tube, through which the meal discharged from the first drier to the second, and was also accumulating in the top drier. The steamer seemed to be perfectly free from meal indicating that the plug must be in the second drier. An attempt was made to locate the plug and determine its exact position by inserting a rod through inspection holes which were conveniently spaced. It was thought that the meal might be broken loose without disassembling this section of the
Photograph IV
Upper drier showing sight glasses (right center).
drying mechanism. The plug could not be conveniently reached; therefore, the plant was shut down and allowed to cool for several hours. When cool the driving mechanism was removed and the screw conveyor pulled from the second drier. It was found that moist meal (meal wet with water) had accumulated in the section of solid flight and had become a rotating plug which had baked to a very hard mass. The meal at this point contained only a slight trace of solvent, indicating that it must have contained some water which had evaporated and helped to remove the solvent vapor. It could not be accurately determined just why the plug started. A lump of meal may have started the accumulation which grew into a solid plug, or meal wet with water may have started accumulating because of its sticky nature.

In an effort to prevent a recurrence of the trouble, the section of solid flighting on the conveyor was shortened to about one foot. Several horizontal bars which were welded to the conveyor flights parallel to the conveyor axis (see Photograph 1) were removed. It was thought that these bars might impede the forward movement of the meal and facilitate the forming of a plug. The bars were placed there originally in an effort to continuously remove the meal from the heated surface of the dryer and allow the new meal to come in contact with the heat transfer surface as explained by Kircher (53).

Run No. 104

This run was started immediately after the conveyor and drive were assembled and was continued for seventeen and one half hours. The termination of this run was caused by solids settling out of the oil.
and miscella and accumulating in the bottom of the still and vaporizer. The entrainment of solids in the miscella seems to be an inherent characteristic of a solvent extraction plant, and handling them in an efficient manner offers a serious problem. The settling tank which had been installed previously between the vaporizer and the stripping still only removed a part of the solids carried with the miscella. The settling tank was not cleaned after the two short runs preceding this one, and no doubt contained an appreciable amount of sludge at the beginning of the run.

The operation of the plant was never satisfactory during the run. The miscella flow was not constant in volume and on occasion reached a volume sufficient to flood the vaporizer, forcing a solvent oil mixture into the vapor line above the vaporizing unit. This unsteady flow was the result of two conditions. The flakes which were used in this run were very poor and contained many fines. This fine material caused partial plugging at times and allowed the level to rise in the upright extractor leg and fall in the slightly inclined extractor leg. When this partial plug or resistance to solvent flow was released, the solvent oil mixture rushed to the miscella outlet in the effort to seek a hydraulic level and consequently flooded the vaporizer. The second reason why the flow was unsteady was a direct result of the flooding. The condensed vapors flowed directly from the water separator to the extractor, therefore all the condensate, whether a small volume or a large volume per unit time, flowed directly back into the system from which it came without having the flow regulated by a pump or other metering device.
After seven and one half hours of operation a part of the sludge which had accumulated in the settling tank was removed. This appeared to improve the operation of both the evaporator and the still, which had been foaming and carrying entrained liquid and solids into the vapor lines above the evaporating units. It was assumed that the erratic operation of these two units was caused by the solid accumulation in the bottom of each unit as well as an accumulation of solids in the liquid lines. This accumulation caused the liquid level to build up in the bottom of the vaporizer and still. It was also apparent at times that an undue pressure was building up in the settling tank. This pressure was released from time to time by opening a vent on the tank, and by draining liquid from both the line entering the tank and the line leaving the tank.

Sludge was again removed from the tank after thirteen hours of operation, and after sixteen hours of operation. The last four hours of operation indicated that the bottom of the still was almost completely plugged. Foaming of the oil became more frequent and it could be observed in the sight glass above the stripping still. An attempt to remove some of the solids from the still was made by opening a valve at the bottom and allowing oil to flow out. This was done from time to time with very little improvement in operation. The oil obtained was dark in color and contained many solids in suspension. The meal from the steamer was free from solvent. Dry steam was flowing continuously into the bottom of the stripping still and into the discharge end of the steamer.
In summarizing the operation of the plant during this run it is seen that the major difficulties encountered were: (1) foaming in both the vaporizer and still, (2) pressure building up in the settling tank, (3) plugging of the settling tank, miscella lines, and still bottom.

At the end of the run the outlet line from the still was practically closed by sludge which had accumulated during the run. This in all probability caused the foaming and priming. The vapor lines were removed and found to contain an accumulation of soybean meal. The horizontal sections were almost completely closed especially the drier and steamer lines. This was an accumulation from the previous runs. The condenser tube sheet was also covered with fine meal. This accounted for the pressure in the settling tank, since the vent line was tapped into the vapor line connecting the still to the condenser. This pressure differential might also aid the frequent surging of the liquid in the extractor.

On examining the still it was found that the inside walls of the filming tubes were covered with a deposit of charred meal and perhaps polymerized oil. It is possible that much of the dark color of the finished oil arises from contact with meal under the temperature conditions existing in the still.

The conveyors in the steamer and driers operated continuously and freely throughout the run. They ran about one fourth full of meal and no indication of a plug was noticed. Preparation for the next run will be given under run No. 105.
Run No. 105

The equipment was completely cleaned after run No. 104 which terminated after seventeen and one half hours of operation. The evaporator, settling tank, still and all connecting lines were cleaned. It was found that the settling tank was nearly full of sludge on the side where the miscella entered. This material had not drained from the tank even though the plug valve at the bottom was opened periodically. The sludge had accumulated and settled in the form of a cone near the miscella inlet. The lines connecting the vaporizer and the still to the settling tank were also partially plugged with solids. The oil outlet pipe from the bottom of the still was nearly closed. The vapor lines from the driers and steamer to the condenser were blown with a blast of air, and were found to be partially plugged with fine meal which had been carried over by the vapors. Inspection of the head of the condenser showed a deposit of meal on the tube sheet but the tubes were not closed and were apparently clean. In preparing for Run No. 105 the miscella removed from the settling tank after Run No. 104 was filtered to remove the solids, and was then returned to the tank. The drier and steamer were partially emptied of meal, and checked thoroughly. Before the equipment was started air was blown through the steam jets of the steamer. A blast of air similar to this was used at the beginning of the previous runs and may have resulted in carrying the solids into the vapor lines and condenser. After the driers and steamer were up to temperature, dry steam was injected into the bottom of the steamer. The vapor outlet at the end of the top drier was closed leaving only
the outlet from the bottom drier and steamer open (Photograph V). This was an attempt to prevent the surging of pressure in the system. The plant was started and flakes fed at the rate of sixty pounds per hour. After operating for one hour, foaming and priming were observed in the sight glasses above the evaporator and stripping still. The flow of miscella was not uniform, and when a surge occurred the evaporator would flood causing the foaming and priming. After four hours of operation it was observed, during a routine inspection, that a plug had formed in the vapor line from the bottom drier. This plug was removed and a rush of vapors from the port hole followed carrying meal from the driers. It was apparent that excessive pressure was forming in the system. The vapor lines were again inspected and found to be almost completely closed with meal. The plant was shut down after eight hours of operation.

The equipment was thoroughly inspected and the following components of the plant fully cleaned: the condenser, both top and bottom; the vapor lines to the condenser; the driers and steamer; the settling tank; and the still. It was found that the top of the condenser was completely filled with meal and the whole cross-sectional area of the tubes closed.

The characteristics which were not conducive to continuous operation were as follows: The accumulation of meal in the vapor lines and condenser; excessive pressure in the system; and the accumulation of solids in the settling tank, extractor, and still.

The accumulation of meal in the vapor lines may have been caused by one of two operations, or a combination of the two. Before the equipment was started a blast of air was blown through the dry steam injection
Photograph V

Driers. The vapor line from steamer and lower drier are in the right foreground, the condenser at the left.
ports. At this time some meal may have been blown into the condenser, but it is unlikely that all the meal found in the condenser and vapor lines was carried in by the air. The second operation which might account for the accumulation was the method of putting the dry steam into the steamer. The inlot ports were located along the bottom of the steamer in a manner shown in Figure I and Photograph VI. The dry steam passed up through the meal as it was moved along the drier. The vapor velocity may have been great enough to keep the fine meal in suspension and carry it into the condenser.

The excessive pressure in the system was in all probability caused by the plugging of the vapor lines coupled with the fact that too much dry steam was allowed to enter the steamer. This high and uneven pressure would cause pulsations of the liquid level resulting in an uneven flow of miscella from the extractor. The abnormal pressure in the settling tank was also caused by the plugged vapor lines.

The accumulation of solids in the settling tank, connecting lines, still and evaporator resulted in very poor operation of these units. At times the solvent was not completely stripped from the oil. The installation of a filter between the extractor and evaporator was considered, but was not installed at this time.

The average temperature readings are recorded in Table 2. They are of the correct order of magnitude with two exceptions. $T_b$, which is the temperature of the vapor leaving the still, and $T_g$, which is the temperature of the vapor leaving the top drier are abnormally high, indicating excessive steam pressure.
Photograph VI
Steamer.
Run No. 106

The steam pressure on the driers and steamer was reduced to fifty pound gage, and the steam pressure on the still reduced to twenty-five pound gage. The air was completely removed from the steam jacket by allowing the steam air mixture to flow from petcocks which were provided for that purpose. These petcocks were allowed to remain slightly open so that any noncondensible gases which might become trapped in the jacket could escape. The vapor line from the meal steamer to the condenser was left closed. This gave the steam which was injected into the steamer a longer path before it reached a vapor line leading to the condenser. The partial pressure of solvent in the gaseous mixture in the second drier was not reduced and better stripping of solvent from the meal resulted.

Water manometers were placed on the steamer, top drier, and the condenser. The temperature of the miscella in the settling was allowed to reach 70° C. before the equipment was started. The expansion of the miscella in the tank was sufficient to cause the tank to overflow into the still before the still was hot, but no foaming resulted since live steam had not been turned into the unit.

The equipment was started and flakes were fed at the rate of about sixty pounds per hour. The flakes were quite thick, averaging about thirty-five or forty-five thousandths of an inch. Good extraction could not be expected in the theoretical extraction time of 35 minutes which was being allowed. Since material movement was the important factor at this particular time, no attempt was made to control the thickness of the flakes.
After thirty minutes of operation it was noticed that the pressure
in the top drier was rising and that the tension in the chain driving
the drier screws was increasing constantly. The plant was immediately
shut down. Just as the plant was shut down the water was blown from the
manometer, and the pin in the drier driving mechanism was sheared. The
steam was turned off and an investigation showed that the top drier was
completely frozen and could not be turned even when a thirty-six inch
pipe wrench was used. The meal which was left in the bottom drier and
steamer was removed from the system by rotating the screws to prevent
the meal from cooking and sticking to the side of the hot tube. During
the period of cleaning the driers, the rotary valve began to offer a
great resistance to turning, finally turning with such difficulty that
the pin in the driving sprocket sheared. The valve was dismantled and
thoroughly cleaned. Fine meal had worked between the surface of the core
face and the shell wall causing undue resistance to turning. After
cleaning, the valve was well lubricated and reassembled. This valve
was one which had been made in the shop of the Engineering Experiment
Station.

The top drier was opened after the equipment had completely cooled.
It was found that the open flighting of conveyor at the immediate end
of the solid flighting was completely filled with meal which formed a
hard solid core about a foot long and had to be removed with a hammer.
The meal had moved freely from the inlet to the drier until the open
flighting was encountered. Here it was apparently still wet with solvent
and perhaps some water. If the steam from the steamer came past the vapor
outlet, the water present in the meal may have resulted from the condensation of steam from the steamer or water vapor resulting from the moisture in the flaked beans at the cool spot near the meal inlet.

Some of the double flighting (flighting with long pitch, Photograph 1) was broken loose, and therefore was removed. This left only single flighting with a pitch of six inches for a distance of eighteen inches from the end of the solid flight. The drier was reassembled and the driving mechanism checked carefully. No apparent damage had resulted from the heavy load which it carried for a short time.

The temperature of the driers and steamer was varied by varying the steam pressure in the jacket from 50 to 75 pounds. Air and steam were injected into the steamer alternately at the various temperature levels in an attempt to determine the steam pressure which would raise the temperature of the effluent gas high enough to prevent the condensation of moisture on the meal. The extractor was not operated and no meal was fed to the driers. These experiments were conducted over a ten-hour period and it was decided that fifty pounds gage pressure would be continued in the drier. It was advisable to keep the temperature of the inside of the tube as low as possible in order to minimize the pyrolysis of the trichloroethylene vapors. The equipment was completely cleaned and made ready for the next run.

Run No. 107

The purpose of this run was twofold: (1) to check the operation of the top drier after the auxiliary flights had been removed, and (2) to
establish, if possible, the amount of steam which should be injected into the meal steamer to insure optimum stripping of the solvent from the meal.

The plant was operated for twenty hours with no evidence of plugging. The driers and steamer seemed to be filled to about one third capacity throughout the run with no indication of accumulation.

Immediately after the meal began to flow from the meal steamer through the rotary valve, the dry steam injector was opened and a very small amount of steam allowed to flow into the steamer. The water from the solvent-water separator was measured over a five-hour period and was found to be flowing at a rate of ten pounds per hour. The meal during this time contained slight traces of solvent.

The steam was increased slightly during the next five-hour period. The water rate from the solvent-water separator during this period was fifteen pounds per hour and the meal was free from solvent.

The steam was again increased to twenty pounds per hour. The meal was free from solvent but the increased vapor velocity seemed to keep more of the fine meal in suspension. It was decided to keep the rate of dry steam as near to fifteen pounds per hour as possible during the runs which were to follow.

The average rate of feed of the flaked beans to the extractor was seventy pounds per hour, varying from sixty to eighty pounds per hour.

The system seemed to be in good condition, and the material movement sufficiently positive to start a solvent loss run, which is recorded as Run No. 108. The average pressures and temperatures will be found in Table 2.
Run No. 106

The purpose of this run was to evaluate the solvent loss from the system during continuous operation. Since the original cost of trichloroethylene is much higher than that of other extraction solvents, it is evident that the solvent loss must be kept as low as possible. The plant was operated for seventeen and one half hours until equilibrium conditions were reached. At this time the "solvent loss" test run was started.

The value of all the variables such as: oil content of miscella, temperature, pressure, solvent-to-meal ratio, extraction time and drying time was noted. These variables were held as nearly constant as possible throughout the run. After the test run began, the weight of all the material entering and leaving the extractor was recorded, and extreme caution to avoid solvent losses was exercised. This procedure was followed over a test period of fifty-two hours. At the end of this run a correction was made for all solvent which was removed from the system during the test period, and which could not be legitimately termed "solvent loss." The remaining solvent, which was lost and unaccounted for, was considered to be the legitimate operating loss and was reported as per cent by weight of solids fed to the extractor. This method of evaluating the solvent loss was subject to the following errors: (1) It was impossible to maintain uniform operating conditions during the test period, and was almost impossible to establish identical equilibrium conditions at the beginning and the end of the test period. Any change in the amount of solvent contained in the system between the beginning and the end of a solvent evaluation run should be made up by solvent
addition, and the amount of the addition called the true solvent loss. This is only true when the terminal equilibrium conditions of the run are the same. (2) Because of the difficulty of matching terminal conditions in the system, during an evaluation run, the error in calculating the solvent loss becomes less as the run becomes longer. More meal is fed to the system as the run becomes longer and the error, whatever it may be, due to difference in terminal equilibrium conditions, becomes less per unit weight.

The plant was set up essentially the same as in Run No. 107 with three exceptions: (1) The miscella from the extractor passed through an eighty mesh screen before entering the evaporator (see Photograph VII). (2) The vapors were removed from the meal drying system through one vapor line only. All lines were closed except the one leading from the top drier (Figure I). (3) The settling tank was vented to the atmosphere through a one quarter inch pipe about ten feet long instead of venting it to the vapor line. The vent line was equipped with a plug cock so it could be opened or closed at will. Normally throughout the run no odor of solvent could be detected at the end of the vent pipe. Periodically the sludge was drained from the bottom of the settling tank and placed in a closed container so that the solvent contained in it which would be recoverable might be evaluated and subtracted from the total solvent loss figure. The miscella from the settling tank flowed by gravity into the stripping still. The oil level in the still bottom was maintained about six inches deep by a hydraulic leg. A bleeder valve was installed at the bottom of this leg which permitted the operator to drain the solids from the still bottom if necessary. The dry steam injected into the
Photograph VII
Miscella outlet with 80-mesh screen in frame at an angle.
stripper passed through the oil pool and flowed countercurrent to the solvent-oil mixture which was filming down the tubes. The same steam pressures were used as in the previous run (Table 2).

Operation of the meal driers and steamer was begun by using air as the sweeping vapor. After several hours of operation, however, the air was cut off and dry steam used to strip the final traces of solvent from the meal. Dry steam was used throughout the duration of the solvent-loss evaluation period.

The oil-solvent mixture from the absorption tower, where the solvent vapors in the noncondensible gases were absorbed, was added to the miscella from the extractor. A small container at the top of the absorber was kept full of soybean oil. A small stream of oil flowed from this container continuously into the absorption tower and from the tower to the evaporator where the mixture was reprocessed to recover the solvent from the oil.

Certain difficulties were encountered during the run. Solids collected in the miscella line between the evaporator and the settling tank and between the settling tank and the still. These lines were cleaned several times during the run. Priming and foaming occurred in the still. Solids also collected in the still bottom and in the oil line leaving the still. This accumulation of solids caused stopping of the run after fifty-two hours. At the end of the run the tubes in both the evaporator and still were cleaned removing a considerable amount of charred solids.

The operation of the rotary valve was the only mechanical trouble
which was encountered throughout the whole period. The rotating core of this valve had a tendency to stick and when this occurred the pin in the drive shaft sheared. It required an average of about fifteen minutes to service and reassemble the valve. During such periods the conveyors in the extractor, meal driers, and steamer were stopped, and the system opened. This occurred six times during the run and was a source of solvent loss.

During the evaluation period three thousand six hundred and forty pounds of soybeans were processed and sixty-four and one half pounds of solvent were added to the system in order to maintain the conditions existing at the beginning of the test period. The total weight of water recovered from the solvent water separator during the test was nine hundred and seventy pounds. Assuming that this water (temperature 25° C.) was saturated with solvent, a loss of one and sixty-five hundredths pounds of solvent was incurred. Nine pounds of solvent was accounted for in the sludge which was removed from the settling tank and was not returned to the system during the test period. This left fifty-three and eight tenths (53.8) pounds of solvent as an operating loss. The loss of solvent in the meal and the oil was negligible.

The following factors made the solvent loss unnecessarily high in this test run: (1) About one half hour was required to remove the sludge from the settling tank, and this operation was necessary about every four hours. During the time of removal, the sludge as well as the bottom of the settling tank was open to the atmosphere allowing solvent vapors to escape. (2) The rotary valve was removed from the system six times for a period of fifteen minutes each.
The following are data on the evaluation run:

- Weight flakes fed to extractor: 3,640 pounds
- Weight solvent lost: 53.8 pounds
- Thickness of flakes used: 1/16 in. - 1/8 in.
- Moisture content of flakes (wet basis): 10 - 14%
- Weight of flakes fed to extractor per hour: 70 pounds
- Weight per cent of oil in miscella from extractor: 12 - 30
- Weight per cent of oil in extracted meal (dry basis): 3.2 - 5
- Weight per cent moisture in extracted meal (wet basis): 3.0 - 8
- Per cent solvent loss: 1.48

The range in the variable is given above rather than a single or average value. Average values are given in Table 2.

At the end of the run samples of miscella were taken at different points down the extractor tube. The analysis of the samples showed the following oil contents:

- Miscella outlet: 31.5%
- 6 ft. from miscella outlet: 16.0
- 10 ft. from miscella outlet: 10.0
- 14 ft. from miscella outlet: 6.0
- 18 ft. from miscella outlet: 3.0
- 22 ft. from miscella outlet: 1.5
- 29 ft. from miscella outlet: 0.0
Run No. 109

In an attempt to overcome some of the difficulties which were apparent in Run No. 108 and previous runs, the following equipment changes were made:

1. The settling tank, which was a source of trouble at all times, was condemned as impractical. While a very large one, having a capacity great enough to allow the solvent oil mixture several hours settling time might have eliminated the solid accumulation in the stripping still, the large inventory of expensive solvent required was considered impractical. Filters were considered at this time, but were rejected because of the additional problem of extracting the oil from the filter cake and recovering the solvent which would be residual after the extraction. For this reason it was decided to make an effort to devise a distillation method which would handle the solids in the miscella. The jacketed kettles which formed the bottoms of the evaporator and stripper were removed. A piece of six inch pipe, one foot long, was flanged at both ends with standard six inch flanges. A half coupling was welded in the side of this pipe to serve as a vapor outlet. The evaporator was now placed on top of the stripping still and connected to it by the one foot length of pipe which will be called the spool piece (see Figure III and Photograph VIII). The vapors from both the evaporator and the stripping still left the distillation systems through the same outlet in the spool piece. The vapor outlet in the top of the evaporator was closed. The miscella entered the system, from the extractor, through the usual liquid seal, filled down the tubes of this unit and then dropped into the tubes.
Photograph VIII
Stripper.
of the stripping still. A cover was placed over the vapor outlet to
eliminate the entrainment of oil particles by the vapor leaving the
evaporator and still. The vapors which were released in the evaporator
fell downward toward the vapor outlet through which they were removed.
The remaining solvent-oil mixture was heated to about one hundred degrees
centigrade before dropping into the stripping still. The remaining
solvent vapor and the dry steam from the stripping still were forced out
to the condenser through the same outlet as the vapors from the evaporator.
The still was not absolutely vertical, but since the oil dropped into
the still from a position above the tubes this was not considered to be
a serious defect. The original jacketed still bottom was removed and a
jacketed section open at the bottom substituted for it. This left the
lower end of the still entirely open. A liquid seal was maintained by
allowing the end of the still to dip into a special bucket which was
filled with oil (Figure V and Photograph IX). The solids in the miscella
could now drop straight through both the evaporator and the still and
settle out in the oil pool at the bottom of the still. The settled
material could be removed from the bucket from time to time without
interfering with the operation of the distillation equipment or without
removing solvent from the system.

(2) The vapor lines from the second drier and the meal steamer were
removed and the outlets plugged. The vapor line from the top drier was
left open to carry the vapors from both driers and the meal steamer.

(3) The steam trap and condensate line were removed from the still
and evaporator as it was decided to regulate the jacket pressure by a
Photograph IX

Lower end of elevator
bleeder valve. The line through which the miscella flowed from the extractor to the preheater was changed to one half inch in order that the solids would be carried through by a liquid flowing at a high velocity.

This run (No. 109) was made to check the plant after the above changes were made. In particular it was desired to determine the effect of allowing the solids in the miscella to pass into the evaporator and still in their new arrangement.

After checking the plant completely all equipment was started. The flakes were fed at a rate of seventy pounds per hour, and an attempt was made to keep the oil content of the miscella about twenty per cent. Considerable trouble was experienced with the rotary valve, the core of which froze quite frequently causing the pin to shear. After six hours of operation it became apparent that the rotary valve could not be made to operate successfully. The plant was shut down and the valve removed. In its place was substituted a conical hopper from a cyclone separator (Figure IV). The meal from the steamer then fell continuously into the hopper, and was removed periodically from the bottom through a manual valve. It was hoped that a vapor seal could be maintained at the end of the steamer and at the same time the meal successfully removed from the system.

The plant was again started and operated for a period of twelve hours. Several interruptions occurred during this time. The solvent level in the upright leg of the extractor varied over a distance of several inches causing erratic flow of miscella from the extractor, and
a drive gear was broken. The main points of interest observed during this run were the following:

(1) The hopper attached to the end of the meal steamer did not form a seal for the solvent vapors. Vapors actually condensed in the hopper wetting the meal which was then discharged through the bottom of the hopper. Solvent vapors could be detected around the hopper by their odor and solvent could be detected in the meal. Sometimes the meal built up on the side of the hopper forming a bridge which was very difficult to remove. The operation of this method of removing the meal from the steamer was very unsatisfactory.

(2) Both air and dry steam were used to sweep the solvent vapors from the meal steamer but both were ineffective.

(3) There seemed to be no accumulation of solids in the evaporator and still. The oil was dark in color, but with few exceptions was free from solvent. Two or three times large amounts of miscella came from the extractor flooding the distillation equipment. When this occurred a trace of solvent could be detected in the oil.

At the end of this run the system was left full of flakes.

Run No. 110

No changes were made in the equipment after Run No. 109. The main items of interest in this run were to check the accumulation of solids in the still and evaporator, and to study the effect of feeding the solids, which were removed from the bottom of the still, back into the extractor with the flakes.
The equipment was operated for a period of seven hours. After the plant was operating smoothly, the solids removed from the bottom of the still were fed to the extractor at a rate equivalent to that at which they accumulated in the finished oil. It was found that a marked increase in the percentage of solids removed from the extractor by the miscella resulted (Table 3). This indicates that some difficulty would be experienced if solids recovered from the oil were continuously fed back with fresh flakes into the extractor.

A few simple filtrations were made using a Buchner funnel. It was found that the solids were quite difficult to filter from the oil, while the miscella was easily separated from the solids as it came from the extractor, indicating that the easiest filtration could be accomplished between the extractor and the evaporator.

After the run was completed the evaporator and still were opened for inspection. It was found that after twenty-five hours of operation these units were still open and no serious plugging of the tubes had occurred. However, a definite deposit of solid material was found on the inside of all the tubes. This material was charred in some places and attached very firmly to the tube walls.

The hopper, which was used in place of the rotary valve, was no more effective as a vapor seal than it was in Run No. 109. The oil from the still was dark in color but free from solvent.

Run No. 111

The plant assembly was exactly the same for this run as for Run No. 110.
Table 3

Effect of Adding Solids from Still Back to System

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Time taken</th>
<th>Operating conditions</th>
<th>% solids in miscella by volume</th>
<th>% solids in miscella by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3:30 PM</td>
<td>Normal</td>
<td>6</td>
<td>5.3</td>
</tr>
<tr>
<td>2</td>
<td>5:00</td>
<td>Normal</td>
<td>6</td>
<td>5.3</td>
</tr>
<tr>
<td>3</td>
<td>5:25-5:45</td>
<td>5 lb. solids added per hour</td>
<td>18</td>
<td>16.6</td>
</tr>
<tr>
<td>4</td>
<td>5:45-6:45</td>
<td>5 lb. solids added per hour</td>
<td>18.5</td>
<td>17.3</td>
</tr>
<tr>
<td>5</td>
<td>6:45-7:45</td>
<td>5 lb. solids added per hour</td>
<td>20.0</td>
<td>18.6</td>
</tr>
</tbody>
</table>
The stripping still was immediately below the evaporator and bolted to it. The cyclone separator hopper was used in place of a rotary valve to remove the meal from the system. The fact had been proved previously that this means of removing the meal from the drying system was inefficient, but no rotary valve, which would operate satisfactorily was available at the time.

The purpose of the run was to determine if there was a pressure difference in the system depending upon whether the solvent vapors were or were not absorbed from the noncondensible gases leaving the system through the absorption tower. The absorption tower consisted of a six foot length of three inch pipe, filled with raschig rings. All equipment was vented to it (see Photograph X).

Seventy pounds of flakes per hour were fed to the extractor. The solvent level was increased until the miscella strength was twenty percent oil. This called for ninety-one pounds of solvent per hour or a solvent-meal ratio of one and three tenths. The average readings for the run may be noted in Table 2.

When the run was started a continuous flow of absorbing oil was fed to the top of the absorption column. This flow was directed upon a spreader plate which allowed the oil to be equally distributed over the entire area of the tower packing. The plant was run continuously for two hours under these conditions and the pressure readings during this period were as follows:

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Condenser</td>
<td>0 in. of water</td>
</tr>
<tr>
<td>Drier</td>
<td>3/8 in. of water</td>
</tr>
<tr>
<td>Steamer</td>
<td>1/4 in. of water</td>
</tr>
</tbody>
</table>
Photograph X
General view. Condenser and absorption tower are in rear center.
The oil flow to the absorption tower was then stopped and the plant allowed to run under the same conditions as before except that the non-condensible gases were allowed to pass upward through the packed tower in which no absorbing medium was flowing. This operation was continued for a period of two hours. At the beginning of this period (for about fifteen minutes) the pressure in the system remained the same as in the previous two hour period. It then began to steadily rise until a pressure of two inches of water existed in the system. This seemed to be a maximum pressure since it went no higher during the next hour. The oil flow was started again and almost immediately the pressures dropped back to the same values which existed during the first two hours of operation. After one half hour the oil flow to the absorber was stopped again, and at the end of one hour the pressures were comparable to the pressures listed above under the same conditions. The equipment was shut down after five and one half hours of operation.

In order to keep the pressure in the system at a minimum it is apparent that the solvent vapors must be removed from the noncondensible gases continuously. It is important that the system operate at a pressure which approaches atmospheric. A positive pressure differential between the atmosphere and the interior of the extraction system increases the possibility of solvent leaks.

Run No. 112

This run was made to check the effect of passing saturated steam instead of superheated steam into the oil-solvent mixture at the bottom of the stripping still. After the equipment had been running for a few
minutes and the evaporator and still were up to temperature, saturated steam was passed into the bottom of the still. After about an hour the oil from the still became cloudy and seemed to contain water. The steam pressure on the jacket was raised to forty pounds. The oil cleared up, but foaming was observed in the sight glass which was installed in the vapor line. The pressure was gradually decreased to 25 pounds over a period of two hours. The foaming ceased in the sight glass and the emulsion again started flowing from the still. After six hours of operation it was concluded that superheated or dry steam should be used as a stripping medium.

Run No. 113

The following changes and additions in the extraction equipment were made before Run No. 113 was begun:

(1) It had been quite apparent in previous runs that the solids which were entrained in the miscella could not be handled satisfactorily in the tubular type of distillation equipment. It was decided to install two filters in parallel. These filters were constructed of eight inch pipe, thirty inches long. The filtering medium consisted of a round canvas bag. The miscella filtered by gravity through the bags and the clear miscella flowed to a miscella storage tank. From this tank it was pumped continuously to the distillation equipment at a predetermined rate. A by-pass was placed in the miscella line leading to the filter so that the unfiltered miscella could be directed to the distillation equipment if so desired (see Figure V and Photograph XI).
Photograph XI
Filters with miscella tank and pump underneath.
(2) The cyclone hopper which had proven unsatisfactory in previous runs was removed and a commercial rotary valve installed at the outlet end of the meal steamer. This valve was driven by a chain from the main drive shaft which ran parallel to the axis of the valve.

(3) Some difficulty had been encountered in previous runs in obtaining a uniform feed of flakes. This seemed to be related to the fact that the feed hopper was so located that the flakes entered the extractor at the liquid level. For this run the feed hopper was moved back to a point where the flakes were fed into the dry extractor tube behind the miscella outlet (see Photograph XII).

It was also hoped that the fresh flakes would filter some of the larger solids from the miscella, thus helping to keep them in the system.

(4) A calibrated rotameter was installed in the solvent line between the condenser and the extractor. All the solvent condensed flowed directly to the extractor, but the flowmeter indicated the amount of solvent that was flowing.

(5) In previous runs no provisions were made for continuously adding the solvent make-up to compensate for the solvent which was lost during operation. This make-up was added periodically as the oil content of the miscella increased above twenty per cent. A small proportioning pump (Hills-McConna) was installed for this run. The solvent make-up was added continuously during the run (Photograph XIII).

(6) A steam coil containing steam under pressure was wrapped around the short section of pipe connecting the meal steamer and the rotary valve in an effort to prevent condensation of moisture above the valve.
Photograph XII
New feed hopper.
Photograph XIII
General view with proportioning pump at left.
(7) The whole system was thoroughly cleaned and checked in preparation for this run. The distillation equipment and driers were in perfect condition.

An attempt was made to feed about eighty pounds of flakes to the extractor per hour, maintaining a solvent meal ratio of 1.3 and an oil content in the miscella of twenty per cent. After fifteen hours of operation a "solvent loss" evaluation run was started. An accurate material balance was recorded during this test period which lasted for fifty-five hours. The plant was operated for a total of seventy hours and no serious difficulty arose throughout the whole run. The filters handled the flow excellently, filtering all the solids from the miscella. There was plenty of time to allow the filter to drain, clean it, and insert a new bag while the second one was filtering. The pump was easily controlled, pumping a uniform amount to the distillation equipment at all times.

The rotary valve worked satisfactorily throughout the run. There was no tendency for the meal to accumulate above the valve and the core of the valve showed no signs of freezing as was the case with the first valve.

A more uniform feed of flakes was secured after the feed hopper was moved above the solvent level. The miscella leaving the extractor, however, carried as many solids as in previous runs. The flakes apparently do not act as a filter as they pass the miscella outlet.

The rotameter was of decided value in indicating the rate of flow and the change in rate from time to time. The flow was not always steady, but by adding solvent through the proportioning pump an average flow was
maintained which corresponded to a solvent meal ratio of approximately one and three tenths.

The steam coil, which was installed to heat the section of pipe above the rotary valve, worked very well. No evidence of condensation was apparent, and the meal did not have a tendency to bridge over or accumulate in that section. Data on this run are given in Table 4.

The following method was used to determine the solvent lost. The plant was operated until uniform conditions were established, then with the system in equilibrium the weights of all materials entering and leaving the system during a definite time interval were recorded. The particles which were carried out of the system with the miscella were removed in the filters and the weight of this material recorded. The amount of solvent thus removed from the system was determined, as this constituted an apparent but not a real loss of solvent. For the same reason a correction was made for the solvent held up in the filter and in the miscella storage tank at the end of the test period. The total weight of solvent removed from the system during the evaluation period, and accounted for, is given in Table 4. The difference between this figure and that given for the total amount of make-up solvent added during the test period was considered as solvent loss. This method of calculating solvent loss assumes that operating conditions at the beginning and at the end of the test period are identical. Since it was almost impossible to operate the pilot plant in such a way that perfectly uniform conditions are maintained during an extended time interval, the above method of evaluating solvent loss is subject to possible errors.
Table 4
Data for Run 113

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of hours of operation</td>
<td>70</td>
</tr>
<tr>
<td>Number of hours of operation during test period</td>
<td>55</td>
</tr>
<tr>
<td>Weight of soybeans processed during test period</td>
<td>4,270 lb</td>
</tr>
<tr>
<td>Weight of soybeans processed per hour</td>
<td>77.6 lb</td>
</tr>
<tr>
<td>Average moisture content of soybean flakes (% wet basis)</td>
<td>8.5</td>
</tr>
<tr>
<td>Average oil content of soybean flakes (% dry basis)</td>
<td>20.0</td>
</tr>
<tr>
<td>Weight of meal recovered during test period</td>
<td>3,220 lb</td>
</tr>
<tr>
<td>Average moisture content of meal (% wet basis)</td>
<td>2.7</td>
</tr>
<tr>
<td>Average oil content of meal (% dry basis)</td>
<td>2.6</td>
</tr>
<tr>
<td>Weight of soybean oil recovered during test period</td>
<td>700 lb</td>
</tr>
<tr>
<td>Weight of wet solids removed from filters during test period</td>
<td>142 lb</td>
</tr>
<tr>
<td>Weight of oil in solids removed from filters during test period</td>
<td>24.7 lb</td>
</tr>
<tr>
<td>Weight of dry fat free meal in solids removed from filters during test period</td>
<td>41.3 lb</td>
</tr>
<tr>
<td>Temperature of meal leaving steamer</td>
<td>113.0°C</td>
</tr>
<tr>
<td>Weight of solvent added to system during test period</td>
<td>137.0 lb</td>
</tr>
<tr>
<td>Weight of solvent removed from system during test period and accounted for</td>
<td>100.4 lb</td>
</tr>
<tr>
<td>Weight of solvent unaccounted for and considered as a loss from the system</td>
<td>36.6 lb</td>
</tr>
<tr>
<td>Solvent loss, based on per cent by weight of soybeans processed</td>
<td>0.86%</td>
</tr>
<tr>
<td>Electric power used during test period</td>
<td>27.4 k.w.h.</td>
</tr>
<tr>
<td>Steam consumption per hour</td>
<td>85 lb</td>
</tr>
<tr>
<td>Water from solvent water separator per hour</td>
<td>13.5 lb</td>
</tr>
</tbody>
</table>
Using one ton of soybeans as a basis, the following figures are calculated as a result of this run:

Time required to extract one ton of soybeans 25.7 hr.
Yield of oil (assuming 98% recovery) 352.0 lb.
Yield of soybean meal (assuming 5% moisture) 1,560.0 lb.
Solvent loss per ton of soybeans extracted 17.2 lb.
Solvent loss, per cent of soybeans extracted 0.86%
Steam required per ton of beans processed 2,200.0 lb.
Electric power required per ton of beans processed 13.0 k.w.h.
When the equipment was taken apart, after the run, it was found that the vapor line from the meal drier to the condenser was partially closed. The tube walls in the evaporator and still showed evidence of having accumulated a charred substance. This substance was not in abundance but was noticeable.

Run No. 114

The purpose of this run was to investigate the operation of the plant when the extractor screw was completely filled with flake, and to investigate the foaming and priming in the still.

The by-pass line from the miscella outlet on the extractor to the evaporator was changed to a two inch pipe. A shallow running trap just deep enough to form a seal at atmospheric pressure was placed in this line. The plant was started and the miscella fed directly to the evaporator without passing through the filter. The first few minutes of operation indicated that something was radically wrong. Excessive priming occurred in the still, building sufficient pressure to break the seal between the extractor and the evaporator, allowing vapors from the evaporator to come through the miscella line into the extractor. It was thought that the miscella flowing into the evaporator might be falling directly into the filming tubes and perhaps concentrating in two or three instead of filming down the entire tube bundle as it should.

The following changes were made in an attempt to smooth out the operation of the distillation equipment: (1) A metal cone was placed in the top of the evaporator so that the entering miscella could not fall
directly into the tubes, but would have to flow onto the tube sheet and then film down the tubes of the evaporator (Figure VI). (2) A deeper running trap was placed in the line carrying the miscella from the extractor to the vaporizer.

The plant was again started, and almost immediately the same trouble occurred as previously. The miscella flow from the extractor was checked and found to be constant. It was quite apparent, however, that the miscella flow to the evaporator was not constant. The oil leaving the still bottom was not completely stripped of solvent even though more than the usual amount of dry steam was injected into the steaming section of the still. The difficulty still seemed to be associated with an uneven flow of miscella into the evaporator, coupled with the fact that the miscella was falling from the tubes of the evaporator directly into the tubes of the stripping still, and probably was not filming down the tubes as was intended. In order to insure filming of the miscella in the stripping still, a cone, similar to that in the evaporator, was installed just above the filming tubes of the stripping still (Figure VI). The cone was placed in such a position that the liquid from the evaporator would have to fall upon it and be distributed to the tube sheet. It would then film down the tubes since the top of the tubes were all level. The cone was in such a position that it did not interfere with the vapor movement from the unit. Both the evaporator and the still were checked and made vertical. When vertical the top of the filming tubes in both units are level and in a horizontal plane. The two inch miscella line from the extractor to the evaporator was changed to a one and one half
inch line with a running trap about three inches deep. It was thought that the smaller line would tend to smooth out the flow since a smaller amount of liquid would be thrown into the evaporator when a pressure surge occurred.

The plant was again started and the same pressure changes coupled with uneven flow of miscella to the evaporator were evident in a very short time. The unit was again shut down and the one and one half inch miscella line changed to a three quarter inch line. The running trap was made about six inches deep. After this change was made the unit operated with no apparent pressure changes and the solvent was stripped from the oil leaving the bottom of the still.

The next objective was to determine the maximum amount of flakes which could be put through the system in a given time without causing undue resistance to the flow of solvent which flowed countercurrent to the movement of flakes. Any increase in the resistance to the flow of the solvent would result in a head of solvent in the upright leg of the extractor. Since capacity was to be increased the speed of the meal driers was changed from 4.6 r.p.m. to 5.7 r.p.m. By gradually increasing the feed to the extractor until the conveyor was completely full it was found that the maximum amount which could be fed was one hundred and five pounds per hour. The increase in the liquid head, in the upright leg of the extractor, required to maintain the flow of solvent through the system when the flights of the screw were completely filled with flakes, was about one foot. The rate of one hundred and five pounds per hour was maintained by allowing the feed hopper to remain about half
full of flakes at all times. The plant was operated at this maximum capacity for a period of fifteen hours. Both the oil and the meal obtained were satisfactory.

It was also observed that the amount of solids entrained in the miscella was less when the conveyor flights were completely filled with flakes. Whether this was a result of running the system full of flakes, or whether a type of flake containing fewer fines was used, was not determined. The oil was not as dark in color as the oil obtained in previous runs from unfiltered miscella. No difficulty arose from accumulation of solids in the evaporator and still, although an investigation showed evidence that an accumulation had started.

The valve in the vapor line which connected the meal steamer and the condenser was opened during the run. The pressure on the system was immediately increased by one inch of water. The valve was closed again and the pressure went back to normal. This procedure was repeated a number of times with the same results.

It is apparent that the miscella feed line to the evaporator, when miscella is fed by gravity, should be a three quarter or one half inch line. If there is a pressure surge, a much smaller volume of miscella is released into the evaporator when a small line is used. This smooths out the pressure differences and results in more uniform and efficient operating conditions.

The cones above the tube sheets in both the evaporator and still had no noticeable effect upon the operation of the distillation equipment. It is thought, however, that they aid proper filming of the mixture down the tubes thus making the unit more efficient.
The maximum amount of flakes which could be fed to the system, without a force feed, seemed to be one hundred and five pounds. This rate of feed resulted in a solvent head of one foot when a solvent meal ratio of one and three tenths was maintained.

Run No. 115

The purpose of this run was to recheck the operating conditions when the extractor was fed at a rate equivalent to one hundred and five pounds per hour, and to check operating conditions when the vapors were removed from the center of the top drier instead of at the end. It was thought that there might be a distinct advantage to this change in vapor removal since there would be countercurrent flow of water vapor from the end of the steamer, through the system to the point where the solvent began to evaporate.

A three inch vapor line was placed in the center of the top drier and extended to the condenser. A valve was placed in the line so that it might be opened and closed at will. A valve was also placed in the two inch vapor line which was located at the end of the top drier. This two inch line had served as a vapor outlet since the line from the steamer was closed (see Figure VII).

The solvent inlet line which allowed solvent to flow into the extractor was raised twenty-two inches. This change was made due to the rise in head when the extractor screw was filled with flakes.

As a result of observations made during the run it was decided that the center of the top drier was not a satisfactory location for a single vapor outlet handling all the vapors from the meal driers and steamer.
The observations made were as follows: When the plant was operating at full capacity, the two inch vapor line was closed and the three inch line, which had been installed in the middle of the top drier, was opened. The pressure in the system was immediately increased to five inches of water and remained at that level until the two inch line was opened. The pressure dropped to three tenths of one inch with both valves open. When the three inch line was closed leaving the two inch line open, the pressure remained at five tenths of one inch. It has been found on several occasions previously that when the vapor line from the steamer was left open, the pressure in the system was higher and the meal discharged usually contained solvent. On the basis of the above observations it seemed that the proper place for the vapor line, to insure optimum pressure conditions, was at the end of the top drier, or generally speaking, at the point in the drying system where the surface solvent had just been evaporated.

Using flakes varying from $1/32$ to $1/64$ of an inch, and allowing the flakes to feed automatically into the extractor from the hopper, the maximum capacity was found to be one hundred and five pounds per hour. For continuous operation, and in order to avoid raising the head in the upright leg of the extractor, it would probably be best to operate with a feed of one hundred pounds per hour or less.

The system was operated for a period of fifteen hours. See Table 2 for observed data.

Run No. 116

The purpose of this run was to determine the effect of feeding the
filter sludge back into the extractor at a rate considerably above the normal rate at which it was collected in the filter bags. In a previous run the sludge was fed back to the extractor at a rate equivalent to that at which it was leaving the unit, and it was found that the fine solids thus added were entrained in the miscella and carried from the system again. It was thought that if, instead of feeding the sludge to the system at a rate equivalent to its discharge in the miscella, a much larger amount was fed over a given period, some of the fines would be entrained with the flakes and carried through the extractor.

The fine meal was a source of trouble from the time the plant was started. In flaking the beans a certain amount of flour was produced, due in part to the use of small diameter flaking rolls and in part to the fact that the moisture content of the beans cannot be closely adjusted prior to flaking. As a result, in all the runs made on the pilot plant were used which contained a large amount of fines and flour. As a result of the method used for removing the miscella from the extractor, practically the only material which could be washed from the extractor was flour and very small particles of flake. The solids in the miscella either had to be filtered from the mixture before stripping the solvent from the oil, or provisions had to be made to periodically clean the evaporator and still if an unfiltered miscella was processed. In previous runs it was shown that the solids could be easily removed from the miscella by passing it through a bag filter. As the material collected in the filter contained an appreciable amount of oil and solvent some provision for recovery was necessary. The easiest way to handle this sludge, if
possible, would be to feed it back into the extractor where the oil would be extracted and the solvent recovered from the fine meal in the driers.

The diameter of the conveyor shaft was made larger where the flakes fed from the hopper. This was done to decrease the feed to about one hundred pounds per hour. The increase in diameter was accomplished by wrapping the shaft with narrow leather belting.

The vapor line from the meal steamer to the condenser was removed, and the connections closed. The vapors were removed through the two vapor lines leading from the top drier, Figure VII.

The filters were used during the run. The sludge, when taken from the filter bag, was blended with flakes and fed to the extractor at a rate several times greater than the rate at which they were collected in the filter bag.

The plant was operated at a rate of one hundred pounds of flakes per hour for eight hours, and the solids collected in the filter during this time were weighed. This gave a figure for the normal rate at which solids were being removed from the extractor by the miscella. At the end of the eight hours of operation a new filter was put on the line. During the next hour of operation thirty pounds of filter sludge blended with seventy pounds of flakes were fed to the extractor. The plant was then run for three hours at the normal rate of one hundred pounds of flakes per hour. At the end of this time the material collected in the filter was weighed. After correcting for the normal accumulation of solids in the filter it was found that out of the thirty pounds of sludge fed into
the extractor, twenty-eight pounds had washed out again and was found in
the filter. This result was very significant as it showed quite con-
clusively that nearly all the fines entering the extractor were carried
cut by the miscella. Any attempt to filter the miscella before it leaves
the extractor would merely complicate matters. If a self-cleaning filtering
device could be made to function at the miscella outlet, holding the
filtered sludge inside the system, a plug would soon form at the feed
end of the extractor. If the fine particles were cleaned from the filtering
medium they would become entrained in the miscella again, and would
be carried to the filtering device where the cycle would be repeated.
This would be a vicious cycle and each particle would play its part.
While this was going on more fines would be added with the fresh flakes
and the accumulation would increase continuously until a plug occurred.

The real solution to the problem of solid entrainment in
the miscella is to use the proper type of flaking rolls, and condition the
beans in such a way that fines are not produced during the flaking
operation.

Conditioning the beans properly would consist of a proper adjustment
of temperature and moisture. Even under the above conditions some fines
would be formed. It seems apparent that these fines should be allowed to
leave the system with the miscella. The miscella should then be filtered
in a suitable filter before passing to the evaporator and still.

Run No. 117

The equipment for this run was left the same as in Run No. 116.
with one exception. The dry steam inlet was placed in the end of the steamer allowing the steam to enter through a port in the blind flange, rather than through the ports in the bottom of the steamer. This change was made to prevent excess agitation, and prevent the fine flour from being suspended in the vapor and carried to the condenser.

The purpose of the run was to again evaluate solvent loss from the system when operated continuously. The oil content of the miscella was held as close to twenty per cent as possible. Flakes were fed at the rate of one hundred pounds per hour and a solvent meal ratio of approximately one and three tenths was maintained.

After the system was operated for three hours, dry steam was fed into the meal steamer through the new port in the bearing flange. Steam was metered to the stripping still immediately after the miscella flow had started. After the system had been running ten hours an increased pressure (about four inches of water) was observed. It was found that the oil flow to the absorbing column had stopped. This flow was started again and the pressure dropped to normal. This happened again after twelve hours of operation. After fifteen hours the drive pinion on the upright screw came loose requiring a shutdown of thirty minutes while repairs were made. This upset the equilibrium which had been attained, so it was decided to operate a few more hours before starting the solvent-loss evaluation.

After twenty hours of operation a plug occurred in the top of the upright extractor leg. This was the first time a plug had formed in this section of the system. The plug was cleared away and the unit was in operation again after fifteen minutes.
The solvent level seemed to be abnormally high in the upright leg of the extractor. Several times during the run flakes entered the solvent line to the extractor causing the flow to cease. The solvent then accumulated in the water separator and eventually overflowed through the water line. This would indicate that the liquid level was at least twenty-two inches above the normal or theoretical level. After thirty-nine hours of operation a plug occurred between the second drier and the steamer. Investigation showed the meal to be wet indicating condensation in this unjacketed section. The plug was removed and operation resumed.

After forty-two hours of operation the rotary valve plugged. Investigation here showed that the meal was wet from condensation. The valve regulating the steam flow to the coil, which was wound around the pipe section between the valve and the steamer, was plugged. The valve was opened and operations resumed.

Forty-eight hours after the run had started a plug of wet sticky material formed directly under the vapor line in the center of the top drier. Apparently condensate from the vapor line was running back and wetting the meal. The excess moisture was probably due to the fact that high moisture content flakes were being fed at that time.

After fifty hours a plug occurred in the same spot as before, under the vapor outlet in the top drier. The run was stopped at this time since poor operation was preventing a test run from starting.

Upon opening the top drier it was found that the meal was wet and that the flighting was bent. The bending probably occurred after the
plug had formed and resistance to rotation became great. The bent section was repaired and the conveyor placed in position. It was found that the vapor line at this point was almost closed with meal. The vapor line at the end of the top drier had accumulated some meal, but was open sufficiently to allow vapor passage without undue resistance.

All the flakes fed the unit during this run were screened over a fourteen mesh screen to remove as many fines as possible. This was to decrease the amount of sludge which would normally accumulate in the filter. Even after screening there was quite a volume of sludge to handle.

Notes on operation:

- Number of hours of operation: 53.0
- Weight of flakes fed: 5141.0
- Weight of flakes fed per hour: 97.0
- Sludge in filter per one hundred pounds of flakes: 3.0

See Table 2 for record of average pressure and temperatures.

Run No. 118

Before beginning Run No. 118 the following equipment changes were made: (1) The three inch vapor line in the middle of the top drier was removed and the opening plugged. The two inch vapor line at the end of the drier was removed and replaced with a three inch line, Figure VII. (2) In order to reduce the rate of flake feed to the extractor from one hundred pounds per hour to about eighty-five pounds per hour a second piece of belting was wrapped around the conveyor shaft where the
flakes entered the extractor from the hopper. (3) The conveyor in the top drier was removed and a section of the long, pitch flighting which had been bent in such a way as to easily impede progress of the meal was taken away.

As in the previous run, the flakes were screened over a fourteen mesh inclined screen. The screen was made to vibrate automatically by attaching an eccentric and an electric motor.

The equipment was started and the run began in the normal manner. The dry steam was bled into the steamer at about the same time flakes began to flow to the extractor. Dry steam was turned into the still as soon as the miscella pump was started.

The only difficulty experienced during this run was the periodic increase of solvent head in the upright leg of the extractor. The solvent level would rise above the sight glass from time to time and would allow flakes to enter the solvent inlet line. These flakes caused the line to plug which was a constant source of trouble. A number of times the line was removed and solvent lost within the sight glass or below it for a period of twelve hours. At this time the level went above the glass for a period of about two minutes, shown at point D, and two and one half hours later it went above for two more minutes, shown at E. The feed rate was returned to eighty-five pounds per hour at F and no more trouble was encountered during the remainder of the run.

The solvent loss test period was started after ten hours of operation, and continued for a period of forty-seven hours. The plant was operated for a period of thirteen hours after the solvent loss period was ended. The average normal operating data are recorded in Table 2.
Solvent loss data are given in Table 5.

Table 5
Solvent Loss Data -- Run 118

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of solvent added to system</td>
<td>179.62 lb.</td>
</tr>
<tr>
<td>Weight of solvent removed in filters</td>
<td>64.70 lb.</td>
</tr>
<tr>
<td>Weight of solvent removed in condensed steam</td>
<td>1.80 lb.</td>
</tr>
<tr>
<td>Weight of solvent drained from system</td>
<td>52.70 lb.</td>
</tr>
<tr>
<td>Estimated weight of tri. in oil 3915 lb. x 0.14% =</td>
<td>5.50 lb.</td>
</tr>
<tr>
<td>Estimated weight of tri. in meal 0.5% of 3039 lb. =</td>
<td>15.20</td>
</tr>
<tr>
<td>Total weight of solvent accounted for</td>
<td>139.50 lb.</td>
</tr>
<tr>
<td>Weight of solvent unaccounted for</td>
<td>39.72 lb.</td>
</tr>
<tr>
<td>Weight of flakes processed during test period</td>
<td>3,915 lb.</td>
</tr>
<tr>
<td>Per cent solvent loss</td>
<td>1.01%</td>
</tr>
<tr>
<td>Weight of flakes fed during entire run</td>
<td>58.10 lb.</td>
</tr>
<tr>
<td>Average rate of feed</td>
<td>83 lb.</td>
</tr>
<tr>
<td>Average per cent of solvent in oil</td>
<td>0.14</td>
</tr>
<tr>
<td>Average per cent of solvent in meal</td>
<td>0.50</td>
</tr>
<tr>
<td>Weight of extracted meal</td>
<td>3,039 lb.</td>
</tr>
<tr>
<td>Oil content of meal</td>
<td>2.0%</td>
</tr>
</tbody>
</table>
It was decided to plot the rise and fall of the solvent level in
the upright leg in an attempt to find out what was causing the varying
resistance to the flow of solvent through the system. The sight glass
in the extractor leg was eight inches long. The rise and fall in this
glass was plotted every five minutes beginning about twenty-four hours
after the run was started (see Curve No. 2). The method of feeding the
flakes to the extractor consisted of dumping fifty pounds of flakes into
the hopper and allowing the hopper to become empty before dumping another.
The time of dumping, and the time the hopper was empty again were plotted
directly above the rise and fall chart. It is interesting to note that
the peak or highest point in the solvent level rise occurs periodically
and in almost every instance at intervals equivalent to the time required
for fifty pounds of flakes to enter the extractor, reaching a peak when
the hopper is about one half empty. The peaks in the curve at points
one to nine inclusive are such high points. It would seem that these
elevations in solvent level were a function of the method of feeding.
About six hours after the start of operations (point A on the curve)
the flake level in the hopper was placed at about one third the height
when containing fifty pounds. The level immediately dropped and stayed
within the sight glass for about four and one half hours, when it went
above the glass for about ten minutes (point B). It again fell into the
glass and stayed at about four inches for over two hours, at which time
it went above the sight glass and remained for more than one hour. The
flake feed was then lowered to seventy-five pounds per hour (point C).
The level dropped and remained down, except for three short time inter­
vals, for the duration of the run.
Curve No. 2: Rise and Fall of Liquid Level in
The solvent loss figure of one and one hundredth per cent is higher than one would expect in a commercial plant of a higher capacity since it is very difficult to make an accurate solvent loss determination on a small capacity unit. The per cent loss will decrease as the capacity is increased since the loss is based on the weight of raw material fed to the extractor. It is not improbable that a figure of less than five tenths per cent would be attained in a plant having a capacity of twenty tons or more per day.

Run No. 119

The object of this run was to observe operation when corn germ's were fed to the system. Several tons of mechanically processed corn germ's, contaminated with fiber and starch as a result of the mechanical separation, were extracted.

There were two equipment changes made prior to this run. A solvent storage tank was installed. The solvent from the water separator was directed to this storage tank instead of directly to the upright leg of the extractor. A small gear pump, with a suitable by-pass, was installed to pump the solvent to the extractor at a uniform rate. A flowmeter was placed in the line in order that a uniform rate of flow could be maintained (see Figure VIII). The second change consisted of an additional space for steam distillation of the oil, (see Figure IX). The trough which was previously used as a seal at the bottom of the still was removed, and the original bottom replaced. The oil was brought out of the bottom through a leg into a second steaming section filled with raschig rings.
for a height of eighteen inches. The vapors were removed through the same vapor line as the vapors from the evaporator and the still.

The corn germ, starch, and fiber had previously been rolled in a roller mill into thin particles. Many of the particles were quite fine, however, about forty per cent passing a fourteen mesh sieve.

This material, as received, was fed to the extractor at a rate of seventy-five pounds per hour. After two and one half hours of operation the extractor leg was completely filled with solids and would no longer rotate. Solvent fed into the system at the opposite end would not flow through the tube filled with meal, but would only raise the solvent level in the upright tube. The unit was shut down and the extractor screw removed. The fine material had formed a solid plug for several feet down the extractor tube beginning at the liquid level. The whole system was cleaned and the screws replaced. The unit was placed in operation again to see if a duplication of the previous trouble would occur. After three hours of operation the same thing occurred for the second time.

The whole system was again cleaned by pulling the screws from the tubing. It seemed quite obvious that the fine starch and small particles in the raw material were causing the trouble. It was decided that the only way the plant could be operated with this material was to screen the fines out and only process the coarser particles. A fourteen mesh screen was used to make the separation, about forty per cent of the material passing through the screen.

The coarse material was then fed to the extractor. There was no trouble in material handling as long as the screened material was used.

The average normal operating conditions may be found in Table 2 with a separate summary in Table 6.
Table 6
Summary of Data on Extraction of Corn Germs

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duration of run (continuous)</td>
<td>47 hr.</td>
</tr>
<tr>
<td>Weight of material fed to extractor</td>
<td>3,525 lb.</td>
</tr>
<tr>
<td>Rate of feed to extractor</td>
<td>75 lb./hr.</td>
</tr>
<tr>
<td>Bulk density of material fed to extractor</td>
<td>17.15 lb./cu.ft.</td>
</tr>
<tr>
<td>Moisture in raw material (wet basis)</td>
<td>5.0%</td>
</tr>
<tr>
<td>Oil content of feed</td>
<td>19.85%</td>
</tr>
<tr>
<td>Weight of solvent hold up per pound of dry fat free meal</td>
<td>1.05 lb.</td>
</tr>
<tr>
<td>Average oil content of meal from extractor</td>
<td>1.00%</td>
</tr>
<tr>
<td>Weight of filter sludge</td>
<td>368 lb.</td>
</tr>
<tr>
<td>Oil content filter sludge</td>
<td>12.1%</td>
</tr>
<tr>
<td>Volatile matter in sludge</td>
<td>42.8%</td>
</tr>
<tr>
<td>Average oil content of miscella</td>
<td>15.0%</td>
</tr>
<tr>
<td>Solvent content of oil</td>
<td>0.1%</td>
</tr>
</tbody>
</table>
The oil from the still seemed quite dark in color. The refining loss was quite high, running as high as nineteen per cent on some samples. The free fatty acid value was also higher than normal.

The major portion of the solids which was entrained in the miscella and trapped in the filter was starch. A visual examination indicated about ninety-five per cent of the filter sludge to be starch. This seemed to be a very convenient way to recover the starch which is normally lost in a mechanical separation process and at the same time recover the oil from the corn germ. Several hundred pounds of this filter sludge was taken to the Leader Iron Works in Decatur, Illinois where a series of filtering tests were made. It was decided that an Oliver filter would work splendidly in conjunction with the extraction system when mechanically processed corn germs were extracted.

**Run No. 120**

The equipment arrangement was left the same for this run as for Run No. 119 (see Figures VIII and IX). The vapors were removed from the driers through the three inch line which was located at the end of the top drier. This seemed to be the optimum location for the vapor line. The preheater and still were in the same position as previously, and the additional still, which was added before No. 119, was left in the system. The filters were used, and the miscella which was pumped to the distillation equipment was free from solids. The solvent was metered to the extractor through a flow meter at a constant rate.

It seemed that the equipment arrangement, as outlined above, approached
that which would be used in a commercial extraction plant. The purpose of this run was to give this arrangement a thorough test. The previous solvent loss runs indicated that an extraction plant using trichloroethylene could be operated with a sufficiently low solvent loss to make the solvent and the system commercial.

The system ran continuously without a shutdown for one hundred and one hours. The solvent meal ratio was maintained a little higher than in previous runs in order that a lower oil content miscella would flow from the system. This was an attempt to change the gravity gradient in the system so that a lower oil content meal would be obtained. This goal was reached as the average oil content of the meal was one per cent. The liquid level in the upright leg of the extractor was higher than in previous runs, because of the increased solvent meal ratio, but it was consistent. The higher level resulted in a shorter drainage space for the meal and also resulted in the meal carrying more than eight tenths pound of solvent per pound of dry fat free meal to the drier.

Toward the end of the run the solvent-meal ratio was decreased and the oil content of the miscella allowed to rise to about twenty-two per cent. This was an opportunity to observe the effect on the oil content of the extracted meal. It was found that the oil content of the meal increased sharply as the oil content of the miscella increased. This indicates that the extraction length or extraction time is a little too short, since it might be advisable to operate with a twenty or twenty-five per cent miscella in order to decrease distillation costs.

At the end of the run the still bottom was removed and the whole
distillation unit inspected. The bottom contained some solids, which
were probably lost to the system while changing filters, and also a
small amount of a tarry substance. This may have been polymerized oil,
phosphatides or perhaps a soluble protein which had precipitated.

The vapor line was free from solids at its junction with the drier,
but the horizontal section was about half full of fines. There was only
a very small amount, an estimated one pound, in the condenser.

The pressure on the system was equivalent to one half inch of water
throughout the whole run. The average temperatures and pressures are
given in Table 2, with a summary of data in Table 7.

Run No. 121

It was decided to change the position of the condenser and increase
the size of the vapor line from the meal drier. The condenser was placed
horizontally in the same plane as the second drier (see Figure X) and
the vapor line leading to the top drier was made of five inch pipe. This
position of the condenser was designed to allow the system to operate at
atmospheric pressure or perhaps a slight vacuum, while the five inch
vapor line would keep the vapor velocities so low that no meal would be
carried to the condenser. The vapors were also condensed on the outside
of the tubes instead of on the inside as in all previous runs.

The plant was operated continuously for a period of twelve hours
with no trouble of any kind experienced throughout the run. The water
from the condenser leaving through the solvent-water separator was checked
continuously for solids and none were found. The solvent water interface
Table 7
Material Balance -- Run 120

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length of run</td>
<td>101 hr., 8 min.</td>
</tr>
<tr>
<td>Weight of flakes fed to extractor</td>
<td>8,550 lb.</td>
</tr>
<tr>
<td>Flakes fed to extractor per hour (average)</td>
<td>82.56 lb.</td>
</tr>
<tr>
<td>Moisture content of flakes (average) wet basis</td>
<td>8.5%</td>
</tr>
<tr>
<td>Oil content of flakes (average) dry basis</td>
<td>21.84%</td>
</tr>
<tr>
<td>Weight of meal from extractor</td>
<td>5,904 lb.</td>
</tr>
<tr>
<td>Moisture content of meal (average)</td>
<td>2%</td>
</tr>
<tr>
<td>Weight of meal from extractor (dry basis)</td>
<td>5,786 lb.</td>
</tr>
<tr>
<td>Oil content of meal from extractor (average) dry basis</td>
<td>1%</td>
</tr>
<tr>
<td>Weight of oil in extracted meal</td>
<td>57.86 lb.</td>
</tr>
<tr>
<td>Weight of oil from extractor (total)</td>
<td>1,563 lb.</td>
</tr>
<tr>
<td>Weight of sludge from filters (total)</td>
<td>394 lb.</td>
</tr>
<tr>
<td>Volatile in sludge</td>
<td>55.4%</td>
</tr>
<tr>
<td>Oil content of nonvolatile sludge</td>
<td>31.2%</td>
</tr>
<tr>
<td>Weight of dry solids in sludge</td>
<td>175.7 lb.</td>
</tr>
<tr>
<td>Weight of oil in sludge</td>
<td>54.8 lb.</td>
</tr>
<tr>
<td>Weight of dry fat free solids in sludge</td>
<td>120.9 lb.</td>
</tr>
<tr>
<td>Weight of dry fat free solids in sludge corrected to 815 per cent moisture (wet basis) and 21.84 per cent oil (dry basis)</td>
<td>155.33 lb.</td>
</tr>
<tr>
<td>Per cent of original feed reaching filter</td>
<td>1.86%</td>
</tr>
<tr>
<td>Condenser water used per hour</td>
<td>9.90 lb.</td>
</tr>
<tr>
<td>Temperature of condenser water</td>
<td>11° C. in, 30° C out</td>
</tr>
<tr>
<td>Total power consumed (extractor)</td>
<td>55 K.W.H.</td>
</tr>
<tr>
<td>Power per ton of beans processed</td>
<td>13.1 K.W.H.</td>
</tr>
<tr>
<td>Steam consumed per pound of flakes</td>
<td>1 lb.</td>
</tr>
</tbody>
</table>
was also free from meal indicating no meal was carried to the condenser. The pressure on the driers and steamer was zero inch of water, and the pressure on the condenser was one fourth inch of water vacuum.

This would indicate that the condenser should be placed in the same plane or below the drier. The vapor outlet should be of sufficient size that the vapor velocity is not increased as it enters the vapor line but it slightly decreased, allowing any entrained solids to settle back into the drier.

The following is a summary of the data collected. Average readings may be seen in Table 2.

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length of run</td>
<td>12 hr.</td>
</tr>
<tr>
<td>Weight of flakes fed to extractor</td>
<td>1,020 lb.</td>
</tr>
<tr>
<td>Weight of flakes fed per hour</td>
<td>85 lb.</td>
</tr>
<tr>
<td>Moisture content of flakes (wet basis)</td>
<td>10%</td>
</tr>
<tr>
<td>Oil content of flakes (dry basis)</td>
<td>21%</td>
</tr>
<tr>
<td>Oil content of miscella</td>
<td>15%</td>
</tr>
</tbody>
</table>
DISCUSSION

In constructing a continuous, countercurrent, countergravity extraction plant for the extraction of oil from the soybean, using trichloroethylene as a solvent, it is recommended that the equipment be arranged as in Run No. 121. The flow sheet shown in Figure XI is the recommended sequence of material flow.

The flakes enter the system through the feed hopper, Figure XI, and are moved through the extractor tube and upright tube by rotating solid flight screw conveyors. The extracted meal saturated with solvent falls by gravity from the upright screw into the horizontal drier. The size and number of these drier sections will depend upon the capacity of the plant in question. The last drier section is called the steamer because dry steam is passed into this section to sweep the solvent vapors from the system. The driers and steamer are fitted with ribbon flight conveyors, except at the sections of meal transfer. At these sections the ribbon flight is replaced with solid flight or at least a wide ribbon. This solid flight will be placed on the conveyor into which the meal transfers. The extracted and dried meal will be released from the steamer by means of a suitable rotary valve.

The vapors from the driers and steamer should flow through a vapor line without increasing their velocity to a rate high enough to carry entrained solids to a condenser. This condenser is independent of the condenser which condenses the vapors from the evaporator and still. The
condensed vapors are then separated in the water separator, and the solvent returned to solvent storage.

Fresh solvent from the solvent storage is pumped through a flowmeter to the upright leg of the extractor. The solvent flows through the extractor countercurrent to the movement of bean flakes and emerges as a solvent-oil mixture (miscella) near the feed hopper. It then flows to a raw miscella storage from which it is pumped through a flowmeter and suitable filters to a filtered miscella storage. From filtered miscella storage it is pumped through a flowmeter to the evaporator and still, from which the product pure oil emerges.

Steam is injected into the still at its bottom. The solvent and water vapor from the still and the solvent vapor from the evaporator are condensed in a condenser independent of the drier and steamer condenser. The condensed vapors are then separated in a water separator and the solvent returned to storage.

All of the equipment is vented through proper vent lines to an absorption tower where a suitable means of recovery of the solvent vapors from the noncondensible gases is employed. This may be an absorption system of some type or might be a refrigeration system.

All the heat transfer equipment should be lagged and a maximum of fifty pounds steam pressure carried on the driers and steamer, and a maximum of twenty-five pounds carried on the evaporator and still.

Photographs XIV to XX, inclusive, show additional views of the experimental equipment.
Photograph XIV
End view of driers and steamer.
Photograph IV
Miscella outlet box.
Photograph XVI
Junction of the two extractor tubes.
Photograph XVII
General view during construction.
Photograph XVIII
Driving mechanism of upright extractor screw.
Photograph XIX
General view.
Photograph XX
Double flight conveyor.
CONCLUSIONS

1. Trichloroethylene is an excellent solvent for soybean and corn oil extraction.

2. Trichloroethylene does not excessively corrode iron when used as a solvent in a closed extraction system extracting oil from the soybean.

3. Trichloroethylene is easily kept neutral or basic when used as a solvent in a closed extraction system extracting oil from the soybean.

4. It is possible to keep the solvent loss within economic commercial limits when trichloroethylene is used as a solvent for soybeans in a closed extraction system.

5. The extraction plant as set up in the last run is suitable for the extraction of the oil from soybeans when trichloroethylene is used as the solvent.

6. The soybeans should be made into flakes having a maximum thickness of fifteen thousandths of an inch for maximum extraction in minimum time.

7. The beans should be tempered by adjustment of moisture and temperature before flaking. This tempering makes the bean more plastic and reduces the fine flour to a minimum.

8. The miscella should be filtered after it leaves the extractor and before it enters the evaporator.
9. The filtered miscella should be pumped continuously and at a uniform rate to the evaporator.

10. The vapor lines from the evaporator and still should be large enough to keep the vapor velocity low. This prevents entrained oil from carrying to the condenser.

11. The live steam should be injected into the meal steamer at a point above the meal level in the steamer.

12. The vapor line from the meal driers to the condenser should be large enough to keep the vapor velocity low. This prevents fine meal from carrying to the condenser.

13. All heated or jacketed equipment, including vapor line, should be well lagged to prevent condensation of solvent and water in the system.

14. The vapor from the driers should exit to the condenser through one and only one vapor line. This line should be located at a point in the drying system where the surface solvent has just dried from the meal.

15. The solvent metered to the system should be pumped continuously and at a known rate. The conditions in the system must be kept constant for efficient operation.

16. The condenser, which condenses the vapors from the meal driers and steamer, should be kept at about the same level as the steamer or lower.

17. The analytical processes given are sufficient to properly control the plant when in operation.
SUMMARY

1. Trichloroethylene is a satisfactory solvent for the extraction of oil from the soybean.

2. The extraction system as arranged in Run No. 121 is entirely satisfactory for the use of trichloroethylene as a solvent in extracting the oil from the soybean if proper analytical control is practiced.

3. No other continuous, countercurrent, countercurrent extraction system known is suitable for the use of trichloroethylene as the extracting medium.

4. The flow sheet as shown in Figure XI and discussed under the chapter "Discussion" is recommended as one to follow in constructing an extraction plant for the extraction of the oil from the soybean using trichloroethylene as the solvent.
LITERATURE CITED


Statistics of the consumption and production of animal and vegetable fats and oils during 1935 are given.


Descriptions of the chlorine derivatives of Ethylene and Ethane and uses for these liquids are given. A brief history of extraction by volatile solvents is included. Historical mention of dry cleaning, degreasing, and textile scouring is given together with a discussion of the use of chlorinated hydrocarbons in these connections.


Discussion of the advantages of chlorinated hydrocarbons and especially trichloroethylene for use in oil extraction, dry cleaning, wool, degreasing and textile scouring. Precautions for their use are given.


A discussion of the decomposition of trichloroethylene and decomposition inhibition by stabilizers. Results of systematic tests on metals are given.


A discussion of the cause of the death of a worker using trichloroethylene for cleaning metal.


Descriptions of the explosions in two soybean oil plants. Conclusions as to the cause of the explosions and recommendations for explosion prevention are given.

A saturated solution of water in N-butyl alcohol was used to extract oil from soybeans. Beans were extracted hot. On cooling a separation into two phases was obtained, one rich in oil.


Bonotto describes the history of solvent extraction and gives recommendations for safety precautions in modern extraction plants.


There is a difference in the method of analyzing for moisture and oil contents of soybeans and cotton seeds.


Physiological effects caused by inhaling large quantities of trichloroethylene vapors. He also gives uses of trichloroethylene.
17. Drozdov, S. S., and Drozdov, N. S. The Corrosive Properties of Di-
1934.


Eichert reviews the literature of industrial poisoning by
trichloroethylene and notes that in addition to its narcotic action
this compound has been asserted to have an injurious action on two
cranial nerves, the optic and the sensory division of the trigeminal.
In spite of the wide clinical use of trichloroethylene for the
 treatment of facial neuralgia, Eichert can find only one report in
the literature of a toxic syndrome following prolonged and excessive
use of this narcotic as a therapeutic agent. He is able to add two
cases of his own, but in none of these three was there anesthesia
of the area innervated by the trigeminal nerve or evidence of injury
to the optic nerve. He therefore concludes that pure trichloro-
ethylene does not affect these nerves and that the industrial in-
toxication which has been said to result in sensory paralysis and
optic nerve atrophy must have been caused by a mixed poison, a
theory widely held in Germany ever since the researches of Kalinowsky
threw doubt on the causative role of trichloroethylene in Plessner's
famous cases.

19. Fergason, R. T. Trichloroethylene for Solvent Extraction of Soybean
Oil. Unpublished B.S. Thesis. Iowa State College Library, Ames,
Iowa. 1936.

Trichloroethylene was successfully used for the extraction of
soybean oil. It was discovered that a mixture of trichloroethylene
and ethyl alcohol would dissolve considerable quantities of soybean
oil while hot and would, on cooling, separate into two phases, one
rich in oil, the other lean in oil.

20. Gasq. Chloroacetylene Solvents. Toxicology of Trichloroethylene

Acute intoxication due to C₂HCl₃ manifests itself in man by
narcosis, loss of equilibrium and of sensitiveness. Post-mortem
examination shows a generalized congestion of all the organs.
Chronic intoxication produces nervous disturbances, paralysis of
the trigeminal and optic nerves and polyneuritis. The toxic dose
has not yet been definitely established, but it would seem that a
limit of 5 mg. per liter should be set as a maximum C₂HCl₃ con-
centration of industrial atmosphere.

Two workers used trichloroethylene in a very badly constructed degreasing plant. They fell ill with the usual symptoms of fresh trichloroethylene exposure and heat pains as well; the 59 year old man was affected on the first day of work though the younger worker was affected only after several months of work. The older man was disabled after five days and eventually died one and one half years later of sclerosis of the heart vessels. After one and one half years of work the younger man was disabled for several months with pains in the heart, but improved gradually, although after five years he still suffered a feeling of oppression on exertion.


The most dangerous and widely used toxic solvents, particularly tetra and pentachloroethanes, and di, tri, and perchlorethylene, are discussed.


These solvents as a class are toxic, capable of producing acute poisonings and occasionally blood changes. The lungs are the chief portal of entry into the body. Skin absorption is possible. Frequently, on long exposures, the chloride and neutral sulfur content of the urine increases as does the acidity. Danger from fire and explosions exists in many instances. Thermal decomposition produces hydrochloric acid and phosgene. The specific properties and action of carbon tetrachloride, trichloroethylene, dichloromethane, methyl bromide and methyl chloride are discussed in detail.


Poisons discussed are certain chlorinated hydrocarbons such as carbon tetrachloride, ethylene dichloride, trichloroethylene, and tetrachloroethane. Petroleum fumes and carbon bisulfide are also discussed.

In cleaning the oil tanks of a ship, trichloroethylene was used after being heated to 100°F. Two workers cleaning below the flood of the machine room fell unconscious from the vapors. One came to after five days, but the other, who was also tuberculous, could not be survived.


A brief history of the development of soybean oil. A review of the composition and modern uses of soybean oil is given.


A review of a number of cases of poisoning. The symptoms in most cases were headache, conjunctivitis, stomach pains and vomiting. The toxicity is greater than would be expected.


He explains the operation of the Rollman, Boehm, and Ford systems of extraction.


Descriptions of the phosphatides associated with the soybeans. Several methods now used for the separation of the phosphatides, i.e., the Tcherdnyzov, Rollman and Rewald methods are given.


The design of a continuous countercurrent extraction plant using trichloroethylene as the solvent is given.


Continuous inhalation of trichloroethylene vapor caused a fading of the pupil in the eye. Abnormal conditions were noticed in other parts of the body.


The author considers Lehman's list of comparative toxicities of chlorinated hydrocarbons misleading. The list follows:

| Perchloroethylene | 1 | The author prefers the following list:
| Trichloroethylene | 2 |
| Dichloroethane | 2.6 |
| Benzene (90%) | 5.0 |
| Trichloroethane | 5.5 |
| Dichloroethylene | 9.0 |
| Carbon tetrachloride | 16.4 |
| Chloroform | 24.6 |


The incidence of poisoning by solvents in industry in England during the period 1926-37 is presented and discussed. Statistics are given for C\textsubscript{2}H\textsubscript{2}, C\textsubscript{6}H\textsubscript{6}, tetrachloroethane, naphtha, gasoline, trichloroethylene, ethylene dichloride, CCl\textsubscript{4}, ethylene chlorohydrin and p-dioxane. A general discussion and brief review of the literature of toxic solvents is included.

A discussion of the data leading to the design of a commercial extraction plant.


The discussion of a system for the extraction of oil from the soybean. The solvent used was a mixture of trichloroethylene and alcohol. After the solvent oil mixture cooled, it separated into two layers, one rich in oil and the other lean in oil.


History of soybean oil processes. Description of extraction systems and advantages of continuous solvent extraction.


Hazards due to EtOH, MeCO, ClC₂H₂Cl, CCl₄, tri and perchloroethylene, and cyclohexanol are discussed.


A discussion of the protein of the soybean and its uses in the industries.


49. Richert, T. G. Oils, Their Production and Consumption. Oil and Soap, 12:149-152. 1935.

Review of world's production of vegetable oils. A description of the extraction plant at the Hansa Mills, Hamburg, Germany.


Procedures and results of various researches on the soybean.


The usual symptoms are intoxication, weakness and tiredness after work. Most of the cases are very mild.


The procedure, discussion, and results of feeding tests on cattle using extracted soybean meal.


Economic discussion of soybeans, detailed description of the uses of soybeans, and various methods of processing with costs are given.


A description of the Ford process of extracting oil from the soybean. Also tells how to convert the meal into plastics.


All solvents other than water are regarded as toxic to some degree. Transient and cumulative poisoning are discussed briefly. The aromatic hydrocarbons and the chlorinated hydrocarbons are the most toxic of ordinary solvents. Safety precautions are outlined.


Pathological symptoms and disorders due to poisoning by various solvents are reviewed.


Discussion of the use of solvent extraction in the vegetable oil industry. He also makes recommendations for extraction units.

ACKNOWLEDGMENTS

The author wishes to take this opportunity to express his appreciation to Dr. O. R. Sweeney for helpful suggestions in carrying out the work connected with this problem.

The work was carried out by a research group, and much credit is given to Mr. C. E. Kircher for his helpful suggestions and assistance.

The research was made possible by E. I. duPont deNemours Company. Dr. A. A. Levine, Dr. C. W. Tucker, and Mr. A. G. Hamilton, of the R & R Division of the company, contributed much of their valuable time and gave many helpful suggestions.
VITA

I was born June 17, 1908 to Ewing Wilburn McCracken and Arizona Elizabeth Fisk McCracken at Green Forest, Arkansas. I attended elementary schools at Osage, Arkansas, Denver, Arkansas, Red Brush, Iowa, Nortonville, Iowa, and Liberty, Iowa. I was graduated from the high school in Osceola, Iowa in 1927. I attended the Missouri School of Mines and Metallurgy, University of Missouri, Rolla, Missouri, and was graduated in Ceramic Engineering in 1931. I specialized in Refractories under Dr. M. E. Holmes and Professor C. Dodd. I later received the Master of Science Degree in Chemical Engineering from Iowa State College in 1937. Dr. C. R. Sweeney was in charge of the work. I then continued work in the same department leading to a degree of Doctor of Philosophy.