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**COMMERCIAL EXTRACTION OF SOYBEAN OIL
USING NON-INFLAMMABLE SOLVENTS**

by

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**A Thesis Submitted to the Graduate Faculty
for the Degree of**

DOCTOR OF PHILOSOPHY

Major Subject: Chemical Engineering

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1947

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INTRODUCTION

The production, processing, and distribution of food-stuffs have always been of primary importance to the human race. Of the three main classes of foods, proteins and fats are usually much less available than carbohydrates as shown by their relative prices.

Soybeans have been an important source of protein and oil for thousands of years. They must have been grown in the Orient long before the Chinese emperor, Shen Nung mentioned them in a materia medica, "Ben Tsao Gang Mu", in 2838 B.C. (29) (37). Soybean oil was first imported into North America about 1900 by manufacturers of soap and paint. Domestic production of soybeans increased slowly so that although the United States consumed large quantities of oil during World War I, practically all of it was imported. Because of the relatively poor quality of much of this oil, usage dropped markedly after 1920 (1). Beginning shortly after 1924, when 5,000,000 bushels of soybeans and 1,000,000 pounds of oil were produced, the industry expanded so rapidly that in 1944 production reached 190,000,000 bushels and 1,240,000,000 pounds of oil (2). In world production of soybeans today the United States and Manchuria lead all other countries,

with the United States far excelling in production of soybean oil. Illinois, Iowa, and Indiana in that order are the leading soybean producing states. The great development of the crop in the corn belt has been aided by the discovery of high yielding varieties, the ease with which the crop can be grown, its beneficial effect upon farm land, and its resistance to drought. These factors have enabled it in many cases to be a more profitable crop than oats in crop rotation. A major reason for the rapid increase in soybean production has been the great demand in America for fats and proteins, especially in times of war.

Since 1936, from 86 to 96 per cent of the soybean oil produced in the United States has been used in edible products, being consumed mainly as shortening, oleomargarine, and various cooking, frying, and salad oils. The essential role of fats in nutrition is well discussed by Bailey (9). As an edible oil, soybean oil has the desirable characteristics of low bleaching costs, whiter products, good consistency behavior, and good resistance to rancidity, but it is handicapped by poor flavor stability (1). A solution to the problem of flavor reversion, which is being studied intensively (8) (33) (34) (35) (50) (57), will greatly aid soybean oil in meeting future competition from palm, coconut, and other imported oils. The amount

of soybean oil used in paints and resins should increase as methods, such as that of the Kellogg Company (91), for producing faster drying fractions are introduced.

Over 95 per cent of the soybean meal is consumed as high protein livestock feed (2). Most of the remainder of the oil-free meal is used in the manufacture of water-proof glue for plywood and as an adhesive for wallpaper. Purified soybean protein is used in large amounts for sizing and coating paper, in the manufacture of water soluble paints, and in fire extinguisher mixtures. One of the most interesting uses of soybean protein is as a source of textile fibers, a process which is now in pilot plant production (27). Only a small percentage of the meal is used in plastics, although a considerable amount of research has been done to increase the use of meal for this purpose (71) (105).

Oil may be removed from soybeans either by squeezing it out by the application of pressure or by dissolving it out with solvents. Of the 11 pounds of oil in a bushel of soybeans, pressing methods remove 8.8 pounds while solvent extraction systems remove 10.6 pounds, or 20.5 per cent more. Since crude soybean oil sells for about five times as much as soybean meal, the advantage of solvent extraction (assuming equal processing costs) is obvious. A characteristic of soybeans greatly aiding the solvent

extraction of the oil is their ability to be rolled into thin, firm, easily extracted flakes.

Most solvent plants now in operation use hexane as the solvent. This solvent is so inflammable that elaborate safety precautions (24) (85) and skilled personnel are required to operate the plants; therefore they can be used only in large scale (50 tons a day or more) installations and even then at considerable risk. The fact that there is a great need for an efficient, small-scale solvent extraction system has long been realized. At first the use of hexane in small plants was considered (106) (107), but because of the inflammability of the solvent such plants were never widely adopted.

The Chemical Engineering Department at Iowa State College has been studying solvent extraction of soybeans for over 15 years. Trichloroethylene has been found to be the most suitable solvent from the standpoint of cost, non-corrosiveness, low solubility in water, low boiling point, and non-inflammability. In 1936 work supported by the du Pont Company was started at Iowa State College for the purpose of developing a small-scale plant using trichloroethylene as the solvent. A pilot plant was developed having a capacity of one ton of soybeans per (24 hour) day (63) (68). The flaked beans were carried through the extractor with a screw conveyor. Work on this

type of plant was taken over by the Detrex corporation, which now has a few small installations in Indiana and Ohio (2) (41) (42).

Efficient countercurrent extraction in an extractor using a screw conveyor is hindered by the fact that the conveyor will not run full of flakes causing part of the solvent to short circuit some of the flakes. Some of the solvent also short circuits between the screw and the casing. Another disadvantage of the screw conveyor is the large amount of fines obtained.

In the summer of 1941 work was started at Iowa State College on a pilot plant for extracting soybean oil with non-inflammable solvents using a Redler chain conveyor for carrying the soybean flakes through the solvent. The flakes almost completed a loop as they were carried without agitation through the solvent, up through a drainage section, and through a section of drier. The pilot plant operated very satisfactorily and furnished data for the design and construction of a commercial plant at Plainfield, Iowa, owned by J. Roach Sons, now processing 15 tons of soybeans a day. This plant is believed to be the most satisfactory small-scale soybean processing plant now available. Certain problems worked out in connection with this plant are presented in this thesis.

HISTORICAL

Processing Methods

Pressing methods.

The origin of the first oil presses is lost in antiquity. Decidedly primitive presses are still used in parts of the Orient today. In the United States soybeans at first were processed mainly in the South in cottonseed presses. The first mill to produce oil and meal from beans grown in America was a cottonseed oil mill, using hydraulic presses, at Elizabeth City, N. C., in 1915 (37). In the United States today only a small percentage of the beans are processed in hydraulic presses, where pressures of about 4,000 pounds per square inch give a cake containing 6 or 7 per cent oil.

Modern expeller plants handle more than 70 per cent of the beans processed in the United States. The expellers are continuous and remove more oil than hydraulic presses, using pressures up to ten tons per square inch (9) to produce a cake containing 4 to 5 per cent oil. Descriptions of the commonly used French and Anderson expellers are given by Markley and Goss, who also list

the manufacturers of other continuous presses (72).

Solvent extraction systems

The fact that oil can be dissolved from seeds has long been known. In 1788, the famous Swedish chemist, Ekeberg, graduating from the University of Upsala, presented a thesis on "Oils Extracted from Seeds" (115). The first patent for the extraction of oil from oil-bearing materials by the use of volatile solvents was issued in France in 1855 to Deiss, who used carbon bisulfide (24). Continuous solvent extraction using hydrocarbon solvents was pioneered in Germany, where the Hildebrandt and the Bollman, or Hansa-Mühle, extractors were developed. The trend in America is toward continuous solvent extraction of soybeans for reasons which have been repeatedly pointed out (14) (37) (77) (92) (106). This trend is shown by the following comparison of the per cent of total beans processed (51).

	Expeller	Solvent	Hydraulic
1936	68.5	13.2	18.5 (sic.)
1939	74.4	20.2	5.4
1947*	71.6	25.9	2.5

Among the most widely used systems in the United States are the Bonotto (17) (20) (23), the Allis-Chalmers

*Compiled from: American Soybean Association. Soybean blue book. Hudson, Iowa. 1947.

extractor, and the V. D. Anderson Company extractor (3), all of which are quite similar. Early installations included many Bollman and Hildebrandt extractors. Description of all these extractors and many others are given by Goss (51) (52), by Markley and Goss (72), and by Bailey (9). A bibliography on the solvent extraction of soybeans is given by Beckel (11). Among the patents dealing with soybean extraction not listed by Beckel are the following: (16) (19) (38) (39) (43) (45) (56) (67) (69) (82) (86) (97) (100) (109) (110) (116).

The Ford Motor Company has developed an extraction system designed for community use using hexane, but the only installations to date have been made by the Ford Motor Company (2) (61). The main reason for the lack of popularity of the Ford system is the fire and explosion hazard contributed by the solvent. With this fact in mind Kircher (63), McCracken (68), and others working at Iowa State College in 1937 and 1938 successfully adapted the screw-type conveyor used by Ford to a system using high density, non-inflammable trichloroethylene. McCracken and the Detrex Corporation continued this work and now have an installation at Danville, Indiana, with a capacity of 25 tons a day (2).

Solvents with a gravity greater than that of soybeans, 1.23, cannot be used in the column-type Bonotto extractor

because in that system the flakes fall through the solvent by gravity. The screw conveyor used by Detrex has certain disadvantages. It does not run completely full; so part of the solvent by-passes the flakes. Part of the solvent also passed between the screw and the casing. It also produces a solvent-oil solution containing a large amount of fine meal. Carrying the soybean flakes through the solvent by means of a Redler conveyor was tried at Iowa State College and found to work splendidly.

Solvents

Extraction solvents have been divided into three classes according to fire hazards: inflammable, such as hexane; semi-inflammable, such as ethylene dichloride; and non-inflammable, such as trichloroethylene (106). The literature is profuse with proposed solvents which have various advantages, such as separating into layers rich and layers poor in oil, extracting more oil, extracting a better grade of oil, extracting certain fractions of oil or separating into layers containing certain fractions of oil, giving a better quality of meal for certain purposes, cheapness, and non-inflammability.

Among the solvents proposed are liquid propane (58) (94), liquid butane (36), hexane, methyl alcohol and trichloroethylene (73) (78), ethyl alcohol and trichloroethylene (74) (78), ethyl alcohol and isopropanol (98)

(99), ethyl alcohol and carbon disulfide (79), ethyl alcohol (12), benzene, sulfur dioxide, carbon dioxide (93), furfural (46), carbon tetrachloride, methylene chloride, dichloropentanes (117), dichloroethane (89), trichloroethylene, and dichloroethylene. Beckel (11) has compiled other references to many of these solvents, while Measamer (73) gives a good evaluation of the most promising ones. Hexane is the most commonly used solvent today because of its low cost in spite of the fire and explosion hazard connected with its use. Trichloroethylene is the most promising of the chlorinated hydrocarbons. Semi-inflammable dichloroethylene and highly volatile, non-inflammable methylene chloride have desirable properties which would make them widely used if trouble caused by the inflammability of one and the volatility and solubility in water of the other could be overcome. Alcohol has long been considered but so far has been used where certain types of protein or extracts are desired and on beans of low moisture content (12). Measamer (73) had good results using a mixture of 25 per cent trichloroethylene and 75 per cent ethyl alcohol, but his work has not been followed up. Liquid propane is used in the new Kellogg process of oil refining, for which remarkable results are claimed (91).

Investigators have proven conclusively that properly

heated (98° C. for 15 minutes) soybean meal from which the oil has been extracted with hexane is fully as palatable and nutritious as expeller meal (54) (55) (75). When trichloroethylene was first introduced as a solvent, a few cases of cattle poisoning by the meal were encountered (103). However, recent work (113) has shown that properly heated meal extracted with trichloroethylene is nutritious and non-poisonous; so the early cases of poisoning must have been due to impure solvent or failing to heat the meal sufficiently in drying. Many hundreds of tons of trichloroethylene extracted meal, some of it not of the highest quality, have been fed during the course of recent work at Iowa State College and other places with no harmful results.

The toxicity of trichloroethylene to plant workers is discussed in detail by McCracken (68). While the solvent is toxic, it is not highly so, and when used with reasonable care should not endanger workers. A necessary requirement of any plant using expensive trichloroethylene (eight cents a pound) is that solvent losses from the system be kept to a minimum. The writer has seen men work in atmospheres containing large amounts of solvent, was once almost overcome by the vapor, and has seen one man completely unconscious from breathing hot vapor. In all cases temporarily harmful, but not severe, reactions followed.

Workers who may be allergic to trichloroethylene, or who develop a liking for the "dopey" feeling brought on by breathing too much solvent should be discharged. A gas mask for use in case of trouble should be available in any trichloroethylene plant.

Trichloroethylene is a better solvent than hexane (5) and extracts more coloring matter giving a darker colored oil. However, Sievers and McIntire have reported that trichloroethylene extracted oil refines to a lighter color (96).

Analytical Control Methods

Methods of procedure for testing soybeans and soybean products are readily available (80). The most important tests in plant operation are moisture and oil in the original beans, moisture and oil content of the meal, solvent content of the solvent-oil solution leaving the extractor and leaving the evaporator, and solvent content of the finished oil. In running the analyses for the oil content of the beans and meal the report of the soybean analysis committee for 1939-1940 (76) and the work of Krober and Collins on the effect of the humidity of the air on the oil extracted (64) should be consulted. The solvent content of solutions leaving the extractor and the

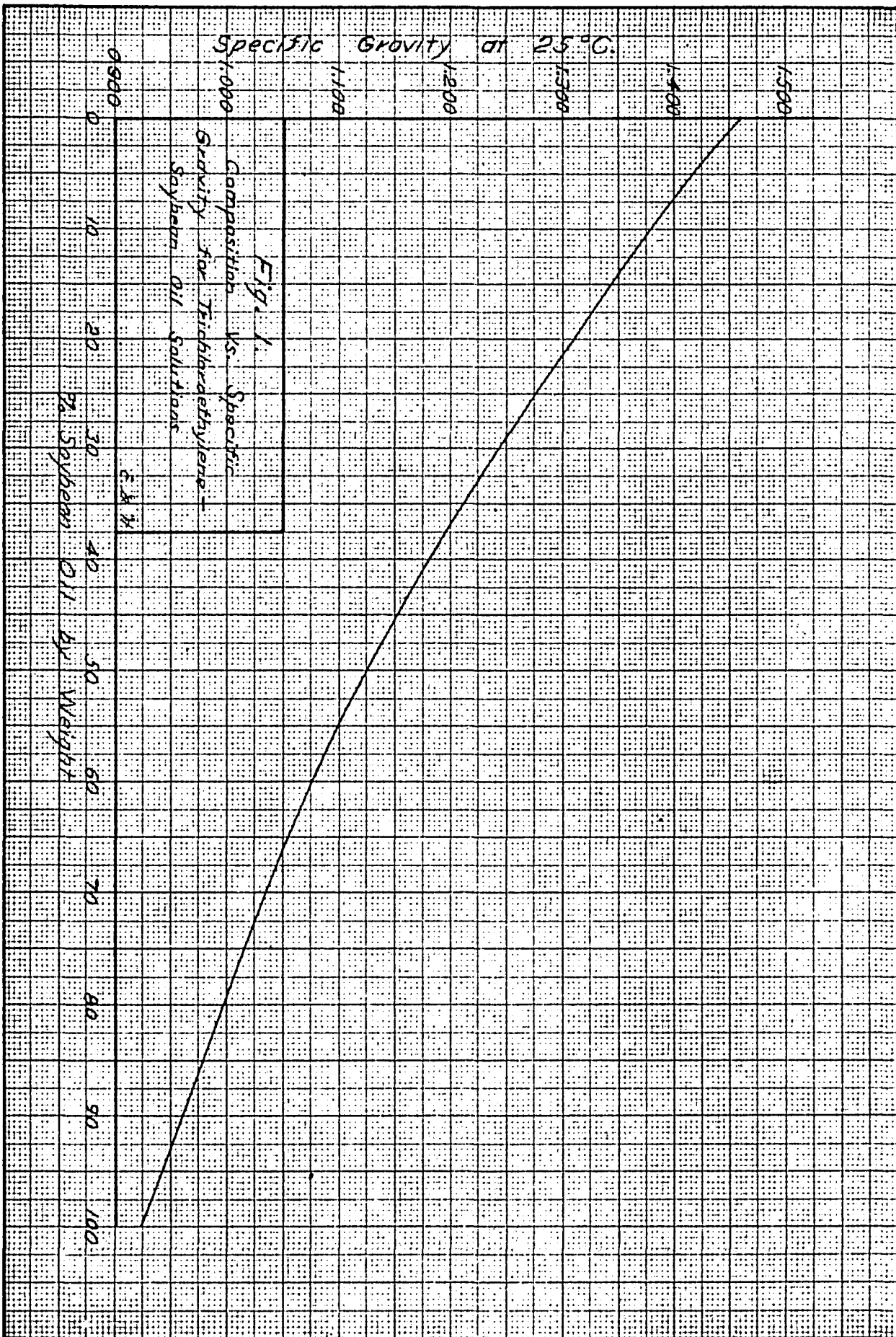
evaporator are determined with the aid of a Westphal balance and a specific gravity oil content curve. Such a curve is shown in Fig. 1. The most important test in ordinary plant operation is the test for solvent in the finished oil. The Fujiwara pyridine test has been adapted by Gilchrist for use in determining trichloroethylene in soybean oil (49). McCracken modified the test to make it more accurate, but less rapid, and also used it for the determination of solvent in the meal (68). When there is over 1 per cent solvent in the meal, and between 1 and 10 per cent solvent in the oil, steam distillation and recovery of the solvent is perhaps the most accurate method of analysis.

A test for adequate heat treatment of the meal is based upon the inactivation of the enzymes by proper heating (30).

Unit Operations in Oil Extraction Systems

Extraction

To extract oil from soybeans it is essential that the seed be ground or flaked, preferably the latter. Such reduction in size is done commercially by cracking the beans in rolls having Le Page cuts and rolling them into flakes. The flaking operation not only provides



less distance for diffusion but ruptures part of the oil cells. According to Osborn and Katz (84), soybeans rolled into flakes from 0.008 to 0.021 inches in thickness have from 70 to 90 per cent of their oil cells ruptured.

The calculation of the number of units in stepwise countercurrent extraction may be determined by the use of overall material balances and the stepwise solution of balances for each stage as shown by Badger and McCabe (7). The same authors work out an example of stepwise countercurrent extraction of oil from meal with benzene using the graphical method of Ravenscroft (90). Elgin has devised a graphical method for solving countercurrent leaching problems using an equilateral triangle to represent the three component systems (44). However, according to Armstrong and Kammermeyer, both the Ravenscroft and Elgin methods are more tedious and less rigorous than the graphical method they present (4). Recently, Ruth has announced a semigraphical method of solving countercurrent extraction problems which is claimed to be versatile, accurate, and relatively simple (95). In general the rate of extraction depends both upon the rate of diffusion through the solid itself and upon the rate of diffusion from the interface into the bulk of the solvent.

In an effort to obtain data on the extraction of soybean flakes Boucher, Brier, and Osburn extracted porous

plates saturated with soybean oil and found that the liquid film was negligible as compared to the resistance to diffusion within the solid (25). Later King, Katz, and Brier found the same to be true of the extraction of soybean flakes with trichloroethylene (62). They also found that the simple diffusion theory for uniform porous solids did not correlate the data for the soybean flakes. They present valuable curves and data for the prediction of extraction time under commercial conditions. Using certain assumptions Osburn and Katz (84) resolved the curves for extraction of soybean oil with trichloroethylene into two component parts. They believe that 70 to 90 per cent of the oil is extracted with a diffusion coefficient of 4×10^{-6} square feet per hour, while the remainder is extracted at a much lower rate of 5×10^{-7} square feet per hour. There was a tendency for the fraction extracted with the lower coefficient to be larger as flake thickness increased.

Measamer (73) found experimentally that the variables having the greatest effect on the rate of oil extraction were the flake thickness and extraction temperature, with the former having the greater effect. Agitation, concentration of oil in the solvent (up to 14.2 per cent), solvent-bean ratio, and fineness of the flakes did not greatly influence the rate of oil extraction. Moisture

affects the rate of extraction of oil in small scale laboratory apparatus (32) (64) (76), but little has been published concerning its effect upon large scale continuous operations.

Filtration

In all soybean oil extraction plants a certain amount of fine meal produced by breaking up of the flakes is entrained by the solvent-oil solution, or "miscella", and has to be removed somewhere in the process. Most plants perform a preliminary filtration at the miscella outlets by covering the outlets with screens or slotted plates. In addition, many plants run the miscella through settling chambers. Bilbe (13) gives data to show that the settling rates of meal from 1 to 100 microns in size is very slow in hexane-oil solutions. The V. D. Anderson Company extractor is surmounted by a settling chamber containing many vertical compartments in which fines are settled out by application of Stokes' law (3).

The Kennedy extractor consists of a series of chambers or sections through which the material to be extracted is conveyed by a series of perforated paddle wheels. Fines are reduced in the Kennedy extractor by allowing the miscella to run through additional sections beyond the flake inlet so the miscella can be filtered through the

perforated paddle wheels (52). Bonotto has patented methods of running the miscella through a conveyor section filled with fresh flakes (18) (21).

For complete removal of the fines some kind of filter is usually placed between the preliminary filter at the miscella outlets and the evaporator. This filter may be any of several enclosed types and usually is run under pressure. Sparkler and bag-type filters (26) as well as centrifugal filters are rather common. The Ford system filter consists of a chamber containing a circular drum made of perforated sheet metal with a canvas cloth covering it. McDonald (70) has patented a process in which a liquid which is immiscible with the miscella and has a different specific gravity than the miscella is added to it. This liquid is absorbable by the suspended fines, which are removed along with the liquid by decantation.

The reason for removing all fines from the miscella is to prevent them from fouling the evaporator and stripping column, especially the latter. Evaporators which will handle fines can be comparatively easily constructed. Attempts to produce stripping columns capable of handling fines have resulted in the sieve-plate column discussed by Bilbe (13) and the Detrex process sieve-plate column patented by Dinley (40). Stripping devices

capable of handling fines using filtros plates have been built at Iowa State College, but so far they have not been operated successfully (48) (59).

Little data on the amount of fines produced by commercial plants are reported in the literature. However, values of over 1 per cent of the weight of the beans are probably not uncommon. Cleaning filters is a difficult and labor consuming task in many plants especially when beans with over 12 per cent moisture are encountered.

Evaporation

One might expect that low boiling solvents such as hexane or trichloroethylene would boil out of non-volatile soybean oil at rather constant temperatures. Such is not the case for as the solution becomes richer in oil the boiling point rises higher and higher as shown in Fig. 2. Last traces of solvent can be removed by direct heat only by heating the oil to a very high temperature or by heating it for a long period of time. To prevent overheating of the oil the solvent is removed in at least two steps. Most of the solvent is removed by direct heat in a variety of ways with the remainder being removed by scrubbing with an inert gas, usually steam.

In the Ford extraction system the hexane-oil solution leaving the filters enters the bottom of a still jacket



through a closed coil which carries it up through the steam jacket and opens into the still body at the top. By the time the solution leaves the coil it has been preheated to such a temperature that most of the solvent vaporizes immediately (107).

An evaporator used in early work at Iowa State College consisted of 19 brass tubes ($3/4$ inch O.D., No. 20 B.W.G.) 5 feet long mounted in a shell of 5 inch standard iron pipe (63) (68). Using steam at 15-20 pounds per square inch gauge miscella was concentrated to 60 per cent oil, 40 per cent trichloroethylene at a rate of about 15 pounds of oil an hour. One disadvantage of this type of falling film evaporator was the difficulty experienced in getting the liquid to film properly down the tubes. When used with miscella containing fines, the tubes gradually became fouled.

Another method of performing the evaporation is simply to boil the hexane-oil solution in large tanks until it reaches a concentration of 50 to 55 per cent oil. Then it is concentrated to 90 to 95 per cent oil in a falling film type of evaporator.

The more modern plants use a process which was worked out independently in connection with trichloroethylene extraction at Iowa State College. Most of the solvent flashes out after passing through a forced or natural

circulation vertical long tube evaporator giving a solution containing over 90 per cent oil in the case of hexane miscella and over 80 per cent oil for trichloroethylene miscella. Descriptions of evaporators of this type are given in the experimental section of this thesis. Although their work did not include solvent-oil solutions, the studies made by Badger and others (15) (28) (104) on vertical long tube evaporators are of great value in understanding the behavior of such evaporators.

Stripping

The last traces of solvent have to be removed from soybean oil by allowing the solvent to diffuse into an inert gas. The inert gas most commonly used is dry steam because it can easily be separated from the solvent by condensation and decantation.

Information concerning the principles of stripping operations is readily available in chemical engineering literature. Stripping, like other diffusion operations, is more efficient when thin films of the liquid are brought into contact with the gas. Usually either bubble-cap columns, sieve-plate columns, or packed towers are used for this purpose. According to Walker, Lewis, and McAdams (114) bubble-cap columns give more efficient contact than packed towers. Bubble-cap columns are expensive and hard

to clean, especially if the column has to be dismantled before cleaning. Sieve-plate columns are less expensive but less flexible than bubble-cap columns. They are said to have the advantage of handling miscella containing fines. Packed columns are cheap, rather flexible, and have low back pressure and low liquid hold-up. Packed columns may be cleaned by dumping the packing, which is less of a job than dismantling bubble-cap or sieve-plate columns. The greatest disadvantage of the packed column is a tendency to channel causing a reduction in efficiency. Channeling may be reduced by using towers with a diameter at least eight times the diameter of the packing, by proper initial distribution, and by providing sufficient flow of liquid to keep the packing wet at all times (10). Perry (88) lists the characteristics of a number of types of tower packings.

Most hexane extraction plants use either bubble-cap or packed columns. The General Mills plant at Belmond, Iowa, uses a bubble-cap column while the plant at Redfield, Iowa, uses 1-inch Raschig rings and runs the column under a vacuum. The Detrex process has a sieve-plate column patented by Dinley (40), which is claimed to be capable of handling fines. Bonotto (22) has patented a stripping process in which a bank of tubes surrounded by a steam jacket is immersed in the miscella. Jets of

steam are blown up through the miscella atomizing it and carrying it through the tubes. The steam entrains and carries off the last traces of solvent.

The stripping column used by Kircher (63) and McCracken (68) at Iowa State College consisted of vertical brass tubes surrounded by a steam jacket. It was identical in construction to their evaporator described in the preceding section on evaporation. The column did not have a very large capacity, probably because of improper filming of the liquid down the tubes, and became fouled when miscella containing fines was stripped. The falling film type of stripping column gave a light colored oil and with modifications might be satisfactory.

Vapor pressure-composition-temperature curves for trichloroethylene-soybean oil solutions as well as mass transfer coefficients for diffusion of trichloroethylene from soybean oil into air are given by Measamer (73).

Drying and toasting

The removal of solvent from soybean meal is commonly done in jacketed tubular driers containing ribbon or paddle conveyors, or a combination of the two. The driers are usually made in sections placed one above the other, and the vapor may be taken off at the end of each section or pass through all the sections and be taken off

only from the upper section.

Sometimes steam is blown into the driers counter-currently to the meal to remove all traces of solvent and aid in toasting the meal. Often the meal is discharged from the driers into large cookers where the last traces of solvent are removed and the meal partially toasted.

Toasting soybean meal by heating it in the presence of moisture increases its palatability and nutritive value. Many consumers have become accustomed to dark colored expeller meal and are somewhat prejudiced against light colored extracted meal; therefore most processors strive to produce well browned meal. The most reliable work yet reported shows that light colored extracted meal produced by heating extracted meal at 98° C. for 15 minutes is fully as nutritious as the darker expeller meal (54) (55).

Kircher (63) gives a large amount of data on the drier and steamer (toaster) used in a pilot plant. The meal entering the drier contained 90 pounds of trichloroethylene and 17 pounds of water per 100 pounds of dry oil-free meal. Ninety per cent of the trichloroethylene and 50 per cent of the water were evaporated in the drier. An overall heat transfer coefficient of 3 or 4 B.t.u./hr./sq.ft./°F. based on the total heating area was realized.

Leaving the drier at 100° F., the meal entered a steamer which was also a jacketed tube containing a

ribbon conveyor. Here the meal was heated to 115-118° C. and steam was blown through it. The remaining 10 per cent of the solvent and 30 per cent more of the water were removed in the steamer. In the steamer the overall heat transfer coefficient was 1 B.t.u./hr./sq.ft./°F. Linear vapor velocity in the drier did not exceed 2 feet per second and in the steamer not more than 1 foot per second. Meal left the steamer through a barrel valve.

Aside from the proper capacity and vapor velocity two other problems are met in drying meal. Some fine meal dust is always entrained with the vapor and has to be removed to prevent fouling of the vapor lines and condenser. This meal is removed between the drier and condenser either with hot water sprays alone or with sprays following some kind of settling chamber. Most of the hot spray water is recirculated, but part is run to the drain to carry off the fine meal.

A second problem encountered in drying meal is condensation of water vapor in cool spots in the drier causing the meal to become sticky and lumpy or even clog the drier. This trouble is prevented by eliminating cool spots in those parts of the drier where water vapor is likely to be present, mainly in that part of the drier or steamer nearest the meal outlet.

EXPERIMENTAL AND DISCUSSION

Apparatus

Pilot plant apparatus

The method of procedure used in this work was to first work out the process as thoroughly as possible using pilot plant apparatus. Then the process was expanded to a commercial scale with the pilot plant being used whenever possible to work out problems met in commercial operation.

The pilot plant was erected in the Chemical Engineering Building in the summer of 1941. The general arrangement of the equipment is shown in Fig. 3. A rectangular chamber 4 by 5½ inches made in the form of a loop with an overall length of 42 feet was the heart of the system. The chamber housed a Redler chain conveyor, size 5, made by the Stephens-Adamson Company of Aurora, Illinois. The chamber at the top of the loop was enlarged to 6½ by 6½ inches for a length of 11 feet, where it was surrounded by a steam jacket.

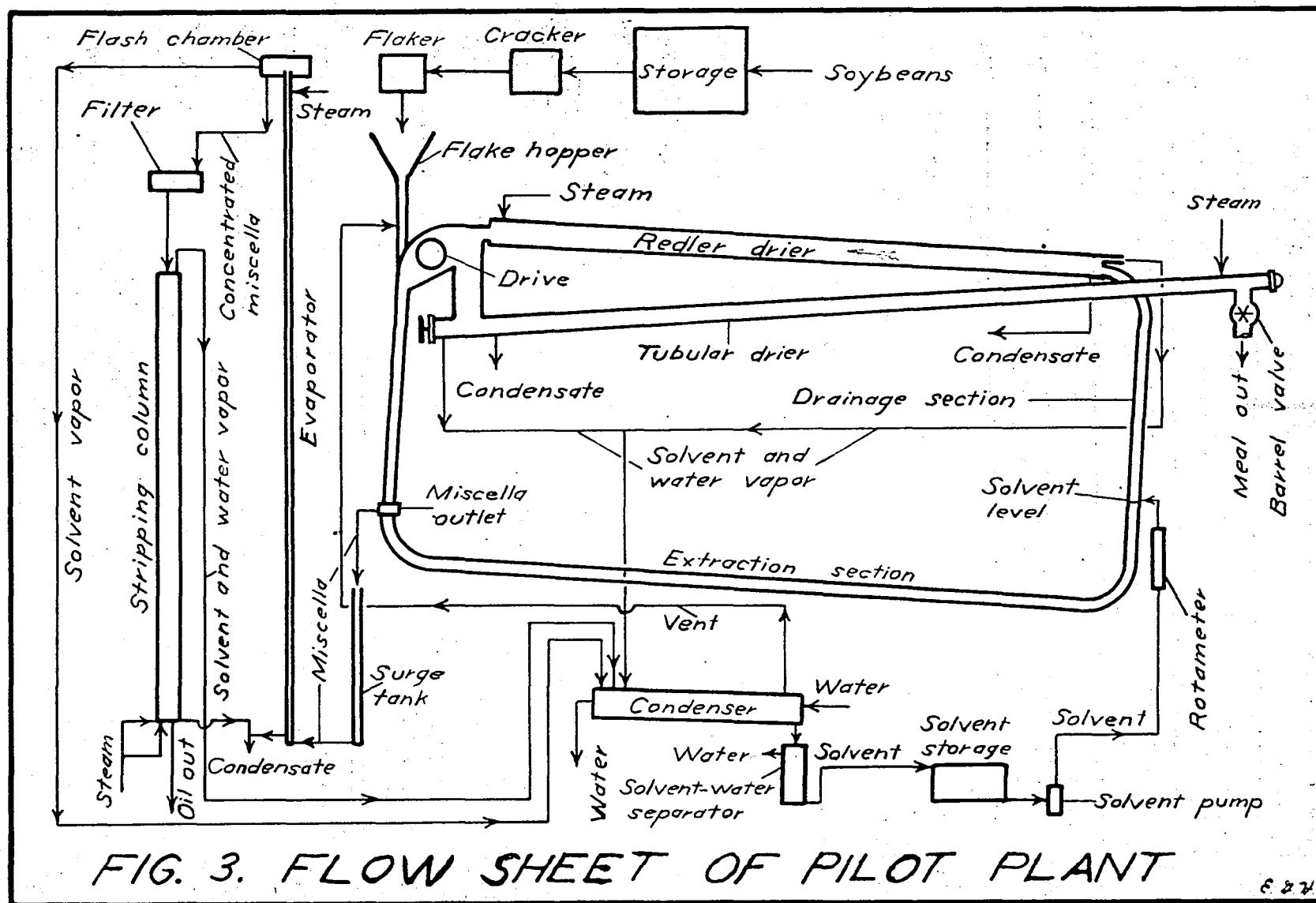
Soybeans were prepared for extraction by being heated to about 50° C., or steamed if they were very dry, and then cracked and rolled into flakes. The beans were

cracked into four to six pieces in a small coffee mill-type cracker made by the Enterprise Manufacturing Company of Philadelphia, Pennsylvania. A pair of 18-inch diameter rolls with 8-inch faces (National Oat Crusher, No. 10, Style A, manufactured by the Excel Manufacturing Company of Pottersville, N. Y.) was used for flaking the cracked beans. The coffee mill and flaking rolls were separated from the extractor so that all the flakes had to be dumped into the hopper by hand. Flakes usually averaged about 0.16 square inches in area and 0.010 inches in thickness.

Leaving the hopper the flakes were carried by the Redler conveyor down through the solvent in the extraction section, then upwards through a drainage section, and through the drier across the top of the loop. After leaving the Redler section, the extracted and partially dried meal fell into a 6-inch pipe steam jacketed to a length of 15 feet 8 inches and containing an open-flight screw conveyor. The conveying element in the drier was a 3/4-inch iron rod bent in the form of a spiral with 6-inch pitch. The dried meal left the system through a barrel valve. The pilot plant had a capacity of 1½ tons of beans a day.

Commercial plant apparatus

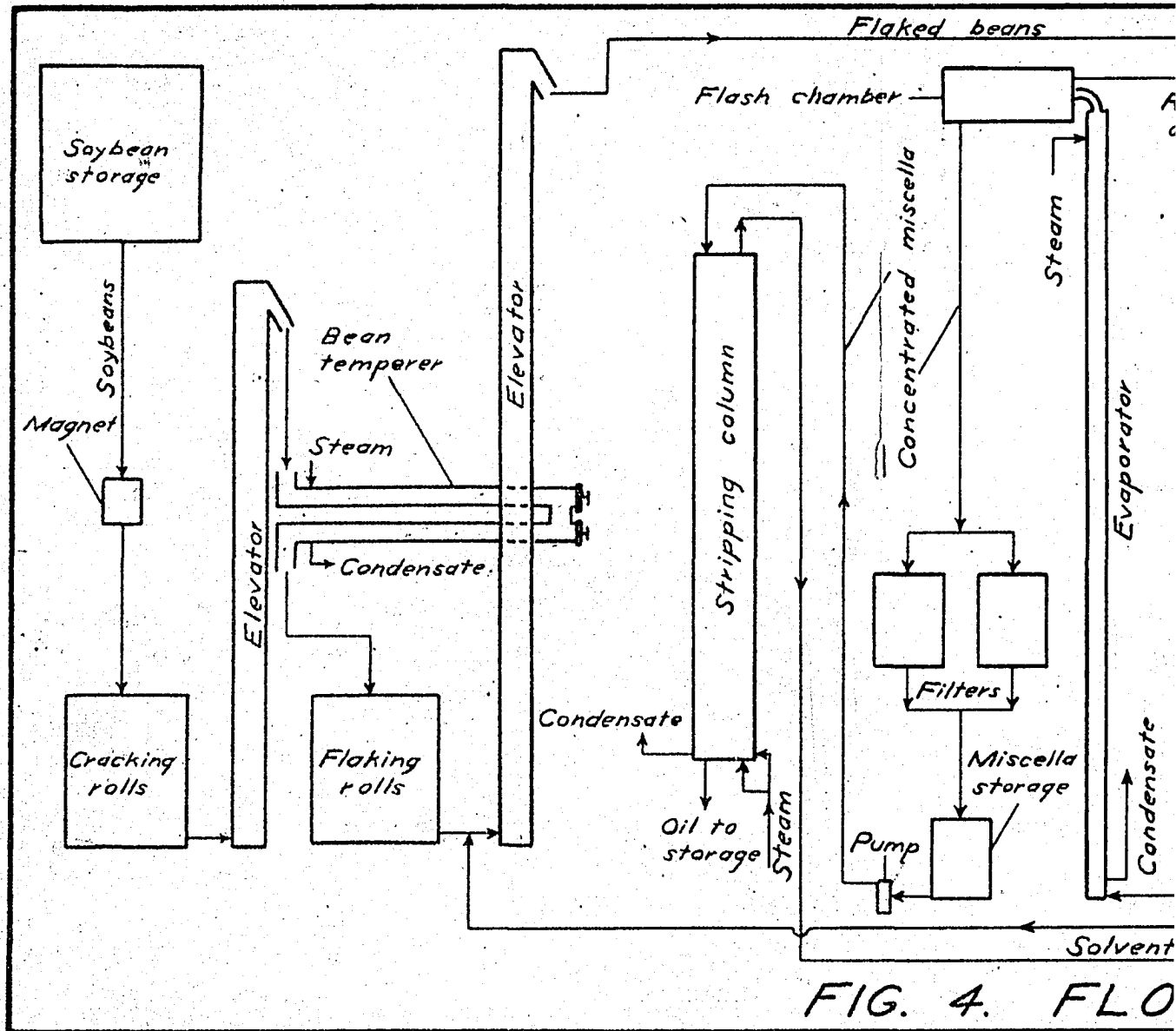
Beans in the commercial plant at Plainfield, Iowa,

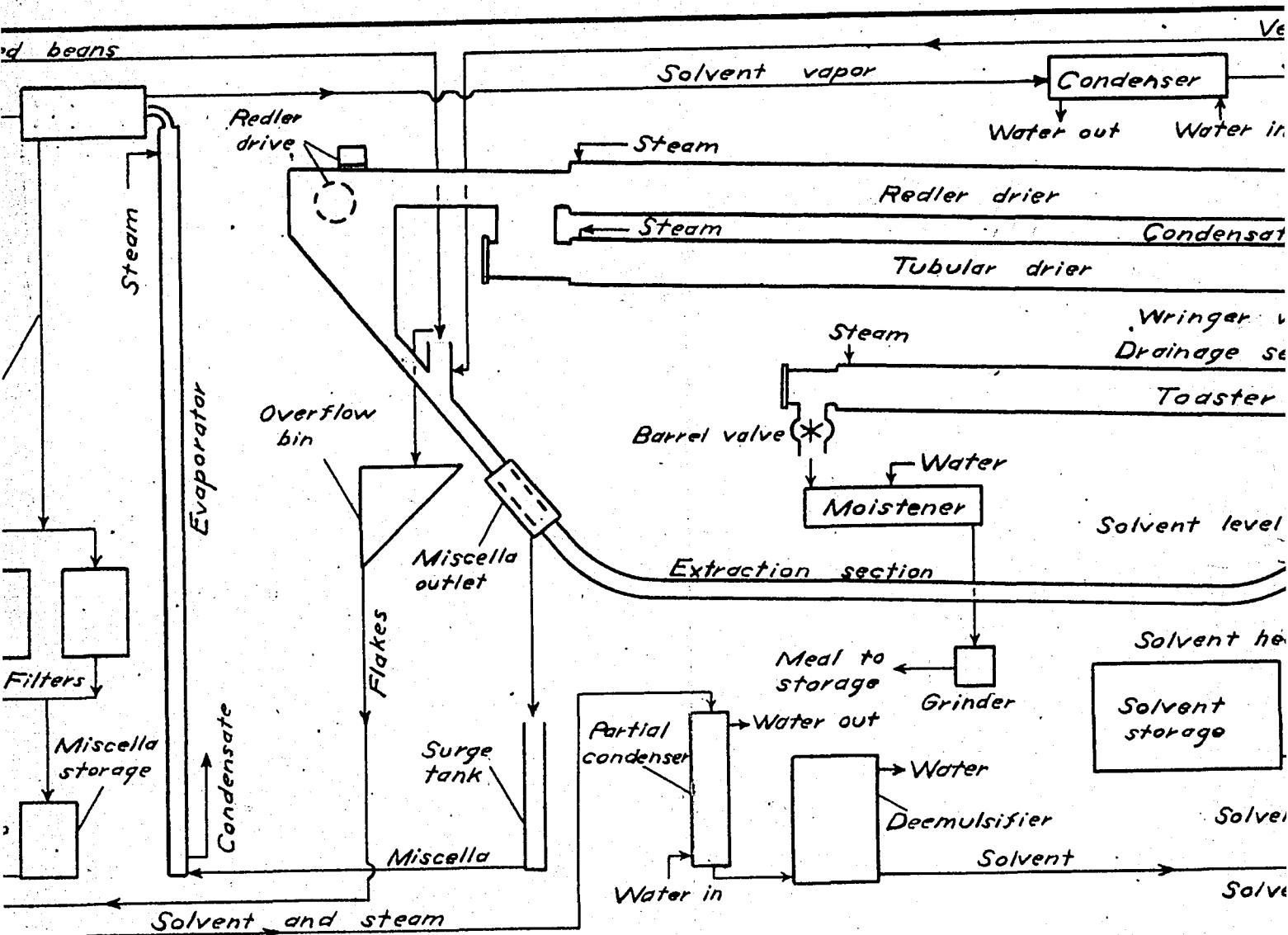


were cracked in a double set of rolls having Le Page cuts. After being cracked the beans were tempered to 150° F. by being passed through two long screw conveyors heated with steam at 90 pounds per square inch. Flaking was done by a pair of 24-inch diameter rolls with 20-inch faces, which were bought secondhand and remodeled. The rolls traveled at about 200 r.p.m. and were considerably oversize for the 15 ton plant. A flow sheet of the plant is given in Fig. 4. The flaking rolls are shown in Photograph 1.

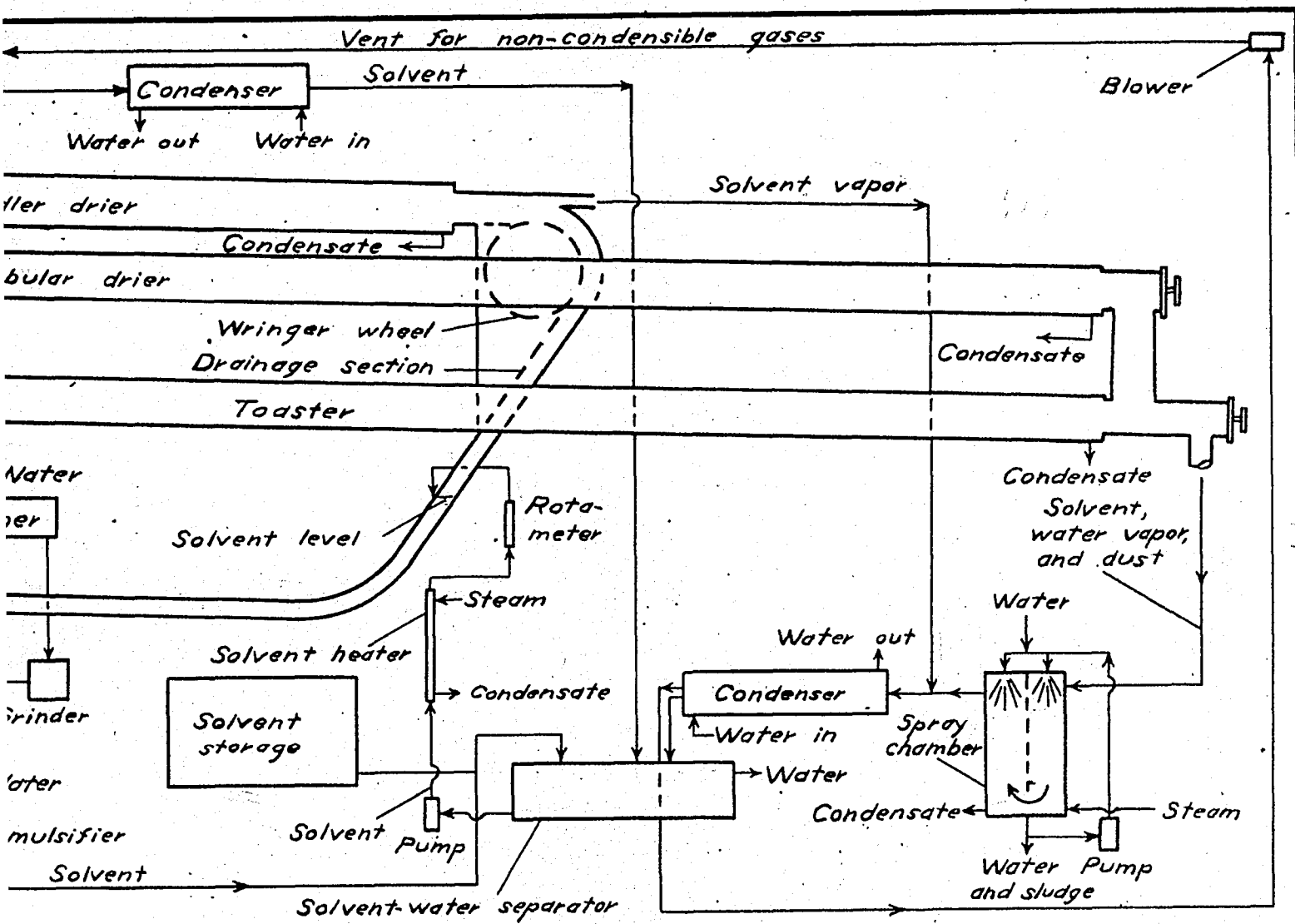
The size 11 Redler conveyor used in the Plainfield plant was housed in a casing 7 1/8 by 12 inches and had a developed length of 90 feet. The casing was expanded at the top to 12 by 12 inches where it was surrounded by a steam jacket. Leaving the Redler, the meal passed through more sections of drier having a diameter of 1 foot and an overall length of 98 feet. The conveying element in the driers was a ribbon 2 inches wide bent into a spiral with a 1-foot pitch. The second section of drier served as a toaster. The meal left the toaster through a barrel valve and fell into a screw conveyor with out-and-folded flights, where water was added to bring the moisture content of the meal up to 12.5 per cent. The meal was then ground and bagged.

Miscella left the extractor through 40-mesh screens.



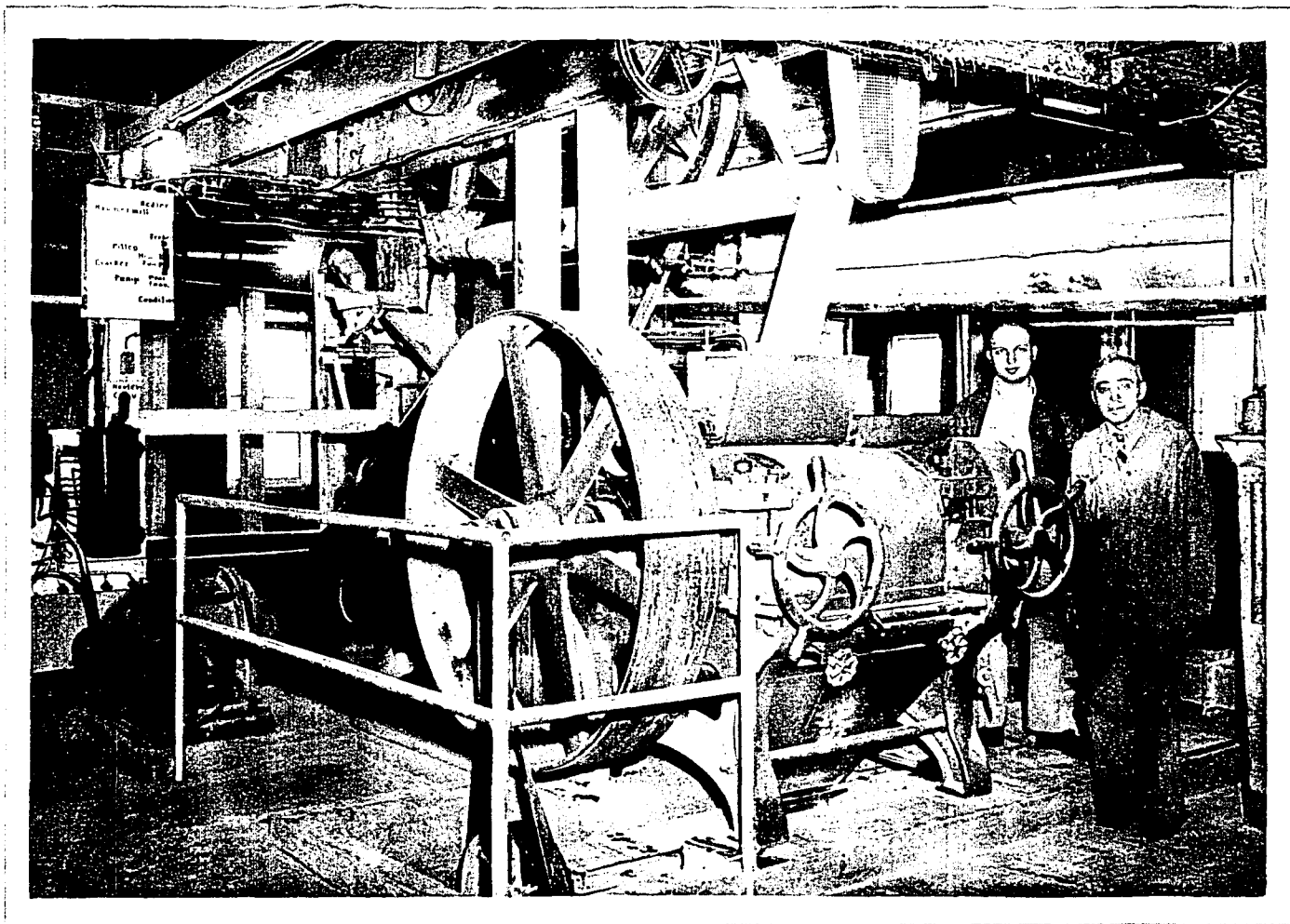


4. FLOW SHEET OF COMMERCIAL PL



COMMERCIAL PLANT

E. & N.



Photograph 1.

Flaking Rolls Used in Commercial Plant

Most of the solvent was evaporated in a climbing-film evaporator. The hot concentrated miscella leaving the evaporator was continuously filtered by gravity and stripped in a column packed with Berl saddles. The stripped oil was pumped directly to storage.

Analytical Procedures

Oil in soybeans

The standard method for determining the oil content of soybeans is to extract a two gram ground sample for two hours, regrind, and extract for three additional hours using petroleum ether as the solvent. In this work the standard method was used whenever an accurate determination of oil content was desired.

A rapid, accurate determination of oil content has long been sought by the vegetable oil industry. It is well known that in general the oil content of soybeans varies inversely with the protein content (83). Soybean oil has a specific gravity of 0.925, while the soybean itself has a gravity of 1.2 to 1.3. From these considerations it was thought that there might be some relationship between specific gravity and oil content, allowing, of course, for moisture content.

Samples of seven pure varieties of soybeans with

varying oil contents which had been grown under approximately the same conditions were obtained. These beans were analyzed using the standard procedures for oil and moisture. The specific gravity of the beans was found by weighing a 40 gram sample and measuring its displacement in water. The results are tabulated in Table I.

Table I
Relationship of Specific Gravity to Oil Content of Soybeans

Variety	Moisture %	Oil % (wet basis)	Oil % (dry basis)	Oil plus Moisture %	Specific gravity at 70°/70° F.
Ottowa-Mandarin	8.67	19.58	21.42	28.25	1.200
Lincoln	8.25	19.80	21.60	28.05	1.220
Dunfield	8.41	19.34	21.12	27.75	1.231
Illini	8.60	19.06	20.84	27.66	1.244
Habaro	8.45	18.23	19.90	26.68	1.210
Mukden	8.43	18.01	19.69	26.44	1.235
Mandell	6.91	19.14	20.57	26.05	1.215

While the results shown in Table I are not conclusive, they make it appear unlikely that there is enough relationship between the specific gravity and oil content of soybeans to serve as the basis of a test for commercial purposes.

A method of estimating the amount of oil that will be removed during extraction is given in the section on

extraction.

Oil in meal

The standard method of determining the oil content of extracted soybean meal is the same as that of determining oil in soybeans, which requires about eight hours of time. Measamer (73) discusses proposed faster methods and presents a quick hydrometer method which has certain advantages. The quick hydrometer method requires stirring a sample of meal with trichloroethylene and taking the specific gravity of the solution with very sensitive hydrometers in a constant temperature bath. This method was tried and found to be rather time consuming and to require considerable skill in manipulation of equipment.

The method finally adopted for use in plant control was simply to extract a five gram sample of the meal in a medium sized Soxhlet extractor with hexane for one hour. The solution was filtered, the solvent evaporated, and the oil weighed. For oil contents under 1 per cent this method, which requires two to three hours total time and about half an hour net working time, gives reasonably accurate results. Meal containing from 1 to 2 per cent oil should be extracted for two hours.

Solvent in oil

For oil contents up to 90 per cent the best method

of analysis for control purposes is to take the specific gravity of the solution with a Westphal balance and read the composition from a chart. Above 90 per cent oil the solution becomes so viscous that accurate specific gravity readings are hard to take. From 90 to 99 per cent oil the steam distillation method used by Measamer (73) is perhaps the best available.

In a new process, such as the extraction of soybean oil with trichloroethylene, it is essential that the first products reaching the market be of the highest quality possible in order not to give the process a bad name. Therefore, it is desirable that all traces of solvent be removed from the oil. Solvent contents of oil ranging from 1.0 to below 0.02 per cent solvent are best determined by the Fujiwara pyridine test as modified by Gilchrist (49). This test is fairly accurate, almost foolproof, and requires less than five minutes time. The procedure is as follows:

1. Add 1 ml. of pyridine to 1 $\frac{1}{2}$ ml. of 10 per cent sodium hydroxide in a test tube.
2. Heat to boiling while shaking.
3. Add two drops of soybean oil and shake sufficiently to mix the oil with the pyridine but not with the sodium hydroxide.
4. After one minute compare the depth of color in

the pyridine layer with a color standard.

Color standards are made by mixing cobaltous chloride and sodium dichromate solutions, slightly acidified with hydrochloric acid until the depth of color created by a known oil sample is matched.

The Fujiwara pyridine test has been applied to other chlorinated compounds (6) and is still being investigated in work which will be reported later.

Solvent in water

At 25° C. trichloroethylene is soluble in water to the extent of 0.11 grams per 100 grams of water. In normal operation water should not leave the plant containing more trichloroethylene than is required to saturate it. The same pyridine test that was used for detection of solvent in oil was used for determination of solvent in water. In this case a standard was made up by allowing water to stand over trichloroethylene until saturated. Then a test was made matching the color produced by two drops of this water with a standard made by dissolving cobaltous chloride in water.

Solvent in meal

When there is a large amount of solvent in the meal, the best method of analysis is to weigh a sample in a

tightly stoppered bottle and then heat it in an oven at 130° C. for two hours to determine the total volatile content. Weigh a separate sample and run a moisture determination using xylene. The difference is the percentage of trichloroethylene in the original sample.

Extracted meal should have had all the solvent removed long before it reaches the end of the driers. In case of faulty operation some solvent may be present in the meal. Faulty operation in the driers may be most accurately determined by running a moisture determination on the meal to see if it is being dried as much as usual. However, a test for small amounts of trichloroethylene in the meal is also valuable. A test for trichloroethylene in soybean meal previously used in soybean work at Iowa State College (68) is rather time consuming. The following test, which will detect trichloroethylene in the meal down to 0.05 per cent, was devised:

1. Weigh a tightly stoppered 125 ml. flask containing 40 ml. of Skellysolve B.
2. Rapidly add 10 grams of meal. Shake well.
3. Add 1 ml. of pyridine to $1\frac{1}{2}$ ml. of 10 per cent sodium hydroxide and heat to boiling.
4. Add four drops of the hexane from the flask containing the meal and shake to mix with the top layer.

5. Compare depth of color with color standard made of cobaltous chloride in water.

Color standards ranged from 0.5 to 0.05 per cent solvent. It was found that although solvent could not be tasted in the oil much below 0.5 per cent, it could be tasted in meal even below 0.05 per cent. Another good way to detect the presence of small amounts of solvent in the meal is to catch a sample of hot meal as it leaves the drier and allow it to cool in a tightly stoppered bottle. Upon opening the bottle traces of solvent may be detected by its odor.

Other tests

All other tests on the beans, meal, and oil were made according to standard procedures.

Extraction

Prediction of extraction behavior

The extraction plant operator uses flakes varying in thickness, size, moisture content, and oil content. To predict the behavior of flakes in the extraction section of the apparatus tests were devised, which, although rather crude, gave remarkably good results.

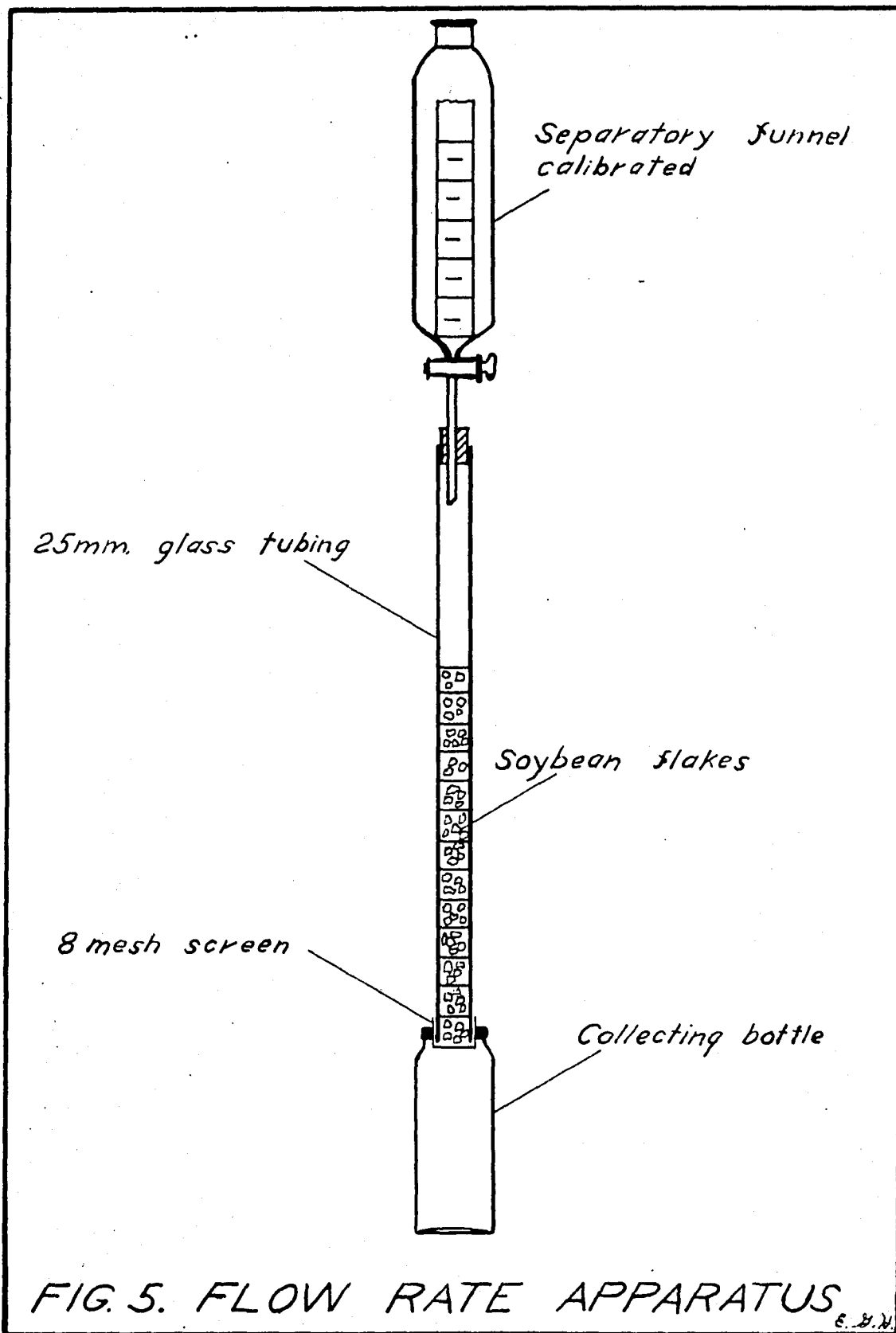
The rate of extraction was estimated by a Soxhlet

extraction of the flakes with hexane for one to two hours. The degree of extraction thus obtained was about the same as that obtained in continuous extractors using trichloroethylene with 15 to 30 minutes extraction time.

In an extractor using a Redler conveyor the material moves through the solvent slowly as a continuous mass with no agitation. The mass of material must be porous enough to allow the solvent to pass through a considerable length of it under very low pressure. Porosity of the material was estimated by use of the apparatus shown in Fig. 5. The 25 mm. tube was filled to the 1-foot mark with the material to be tested and shaken just enough to make the material settle somewhat. The separatory funnel was filled to the 500 ml. mark with trichloroethylene. Then solvent was allowed to run out of the separatory funnel at such a rate that the solvent level in the tube was always at the 1-foot mark. The time required for all the solvent to run out of the funnel was taken. Solvent ran through good soybean flakes in about 35 seconds. If much over 60 seconds were required, the material either would not be porous enough to be used in the extractor or would work only at reduced speed.

Method of feeding the extractor

When the extractor was first constructed, it was



intended that the material be fed into the extraction chamber at a point about two-thirds of the way from the solvent inlet end so that the other third could be used as an evaporator. Difficulty was experienced in getting the flakes to feed into the miscella and in properly evaporating solvent from the miscella; so this procedure was temporarily abandoned. The flakes were next fed in at the upper corner of the Redler loop falling around the sprocket wheel as shown in Fig. 3. The flakes fed in well from this position except for a tendency to bridge and not fill the chain properly when they were moist. The next method of feeding tried and the method used in the commercial plant (Fig. 4) was to feed the flakes into the open side of the chain. This method of feeding worked satisfactorily.

Factors affecting rate of extraction

The controlling factor in a system for extracting oil from soybeans is the length of time the solvent is in contact with the material to be extracted. In a system such as that developed at Iowa State College this time of contact is in turn limited by the length to which the extractor can be made and the speed with which the material can be conveyed without causing too much pumping of the

solvent by the flakes.

The pilot plant extractor length was 16 feet 9 inches and the chain speed 11 inches a minute. Solvent at the solvent inlet end seldom rose more than a foot higher than the miscella outlet. Flakes 0.010 inches in thickness were extracted to about 1 per cent oil when miscella averaged 20 per cent oil.

The Plainfield extraction length was about 30 feet. Chain speed varied from 11 to 16 inches per minute as capacity varied from 10 to 15 tons per day. Pumping varied from 1 foot with good flakes at 10 tons to 4 feet with poor flakes at 15 tons. Pumping in itself is not a bad feature as long as extraction is countercurrent unless the solvent level reaches the Redler drier.

Flake thickness exerts a decided influence upon the rate of extraction as shown by Messamers(73) and by King, Katz, and Brier (62). The commercial plant left 2 per cent oil in flakes 0.180 inches in thickness compared to 0.7 per cent oil in flakes 0.010 inches in thickness running at the same rate. Flakes from 0.0080 to 0.010 inches in thickness were found to be most satisfactory because they were thin enough for good extraction and yet would stand a moderate amount of handling without breaking up into fines. Tempering the cracked beans to about 150° F. before flaking aided greatly in producing flakes

with few fines and in making the flakes pliable enough to hold together until they reached the extractor. The flakes produced had an average area of 0.16 square inch.

Temperature has been found to have considerable effect upon the rate of extraction of oil from soybeans (73). For this reason the steam jacketed Redler drier section not only aided in the drying but aided in extraction by virtue of the fact that the Redler conveyor transferred its heat into the extraction part of the system. Tempering the beans aided extraction for the same reason. Since all solvent pumped into the Redler had to be evaporated eventually it was found to be good practice to heat the solvent to about 140° F. before passing it into the extractor. Heating the solvent also placed less of a load on the driers, especially on cold winter days. The extraction chamber was not insulated, but should have been to reduce heat losses.

The major portion of the extraction chamber was inclined to an angle of 1°30' to aid the tendency of the solution to rise as it became richer in oil and therefore lighter. Such a design reduces eddy currents and is believed to make the extractor the most perfectly counter-current extractor now available for soybeans. The optimum concentration of oil in the solvent leaving the extractor was around 20 per cent oil by weight. Running at 10 tons

of soybeans a day the Plainfield plant gave a meal containing 0.6 to 0.8 per cent oil and at 15 tons a day a meal averaging 0.7 to 0.9 per cent oil.

The effect of moisture upon the rate of extraction of soybean oil from both flakes and ground meal in the laboratory is somewhat controversial (64) (65) (73) (76). One is inclined to give most weight to the work of Krober and Collins (64) who say that when ground soybeans with moisture contents from 4.35 to 16.8 per cent are analyzed for oil content the amount of extractable material is not dependent upon the original moisture levels. However, their curves do indicate that moisture content exerts some influence upon the rate of extraction. Beans containing from 8 to 13 per cent moisture were extracted in the pilot plant and from 12 to 14 per cent moisture in the commercial plant. If there was any difference in the rate of extraction for flakes in these moisture ranges, it was slight.

Properties of oil extracted with trichloroethylene

The crude oil obtained from the Plainfield plant was light in color and had a good odor and taste. Oil averaging less than 0.02 per cent trichloroethylene was consistently obtained. Other properties of the oil are listed below.

Crude oil as shipped:

Free fatty acid	0.7
Moisture and volatile	0.24
Gardner break	0.43
Refining loss, %	4.2

Oil from which foots had settled:

Viscosity, centipoises, 100° F.	30.9
Specific gravity at 60°/60° F.	0.9243
Refractive index, 25° C.	1.4727
Lovibond color, 25 mm.	70 yellow
	5.2 red

Refined oil:

Lovibond color, 133 mm.	70 yellow
	11.5 red

Refined and bleached oil:

Lovibond color, 133 mm.	14 yellow
	1.4 red

Recommendations

a. The extractor cross section should be somewhat larger than the Plainfield extractor so that over 15 tons a day of soybeans can be processed without reducing the extraction time or causing undue pumping.

b. It might be possible to design the extractor in such a way that a miscella richer in oil could be obtained

without reducing the completeness of extraction.

c. The extraction chamber should be insulated to save heat and make extraction more efficient.

d. The effect of moisture upon the rate of extraction should be studied further.

e. The effect of operating conditions upon the quality of oil obtained from the commercial plant should be further studied in order to obtain the highest possible quality of oil.

Filtration

Pilot plant

Screens. In cracking and flaking soybeans and conveying them to the extractor a certain amount of breaking up into fine particles is unavoidable. Since it has been shown that agitation has little effect upon the rate of extraction of soybean oil from flakes (25) (62) (73), an extraction system such as that used in this work is highly desirable. Carrying the flakes through the solvent in a slowly moving mass not only prevents them from breaking into fines, but also provides a certain amount of filtering action for the fines that are produced and carried with the solvent. This fact has been recognized by at least one worker, who has patented a miscella filter

which consists of a slowly moving mass of soybean flakes carried by a Redler conveyor (21).

In order to keep as many fines as possible from leaving the extractor, the miscella was passed through screens placed on each side of the Redler casing. One hundred mesh screens were tried and found to be too fine. Sixty mesh screens operated satisfactorily at times but were not as foolproof as 40 mesh, which were finally used. Sixty mesh screens placed with the wire tilted 45° from horizontal operated somewhat better than when in the regular horizontal position.

The wiping action of the flakes passing the screens kept them from clogging with fines. Whenever the Redler chain ran partially empty because of difficulty in feeding, the screens tended to clog. The action of the screens in operation was rather interesting. All the miscella ran out the lower third or fourth of the screens. Flakes could be seen moving past the upper part of the screens gradually pushing fine particles of meal through the screens. If the miscella rose to such a height that the screens were completely covered, no more fine particles were pushed through and the miscella also practically stopped coming through. The screens would then have to be by-passed or the solvent cut off until the miscella level dropped to the lower part of the screens. Wet,

soggy flakes also tended to clog the screens.

Data on the fines produced in the pilot plant are given in Table 2. In runs 1 through 5, miscella containing 20 per cent oil was filtered. The cake averaged 20 per cent oil. In runs 6 through 8 concentrated miscella containing 80 per cent oil was filtered. The cake contained 30 to 40 per cent oil. In all runs the beans were flaked and allowed to dry to from 6 to 9 per cent moisture before being run through the plant.

Table 2
Fines Obtained From Pilot Plant

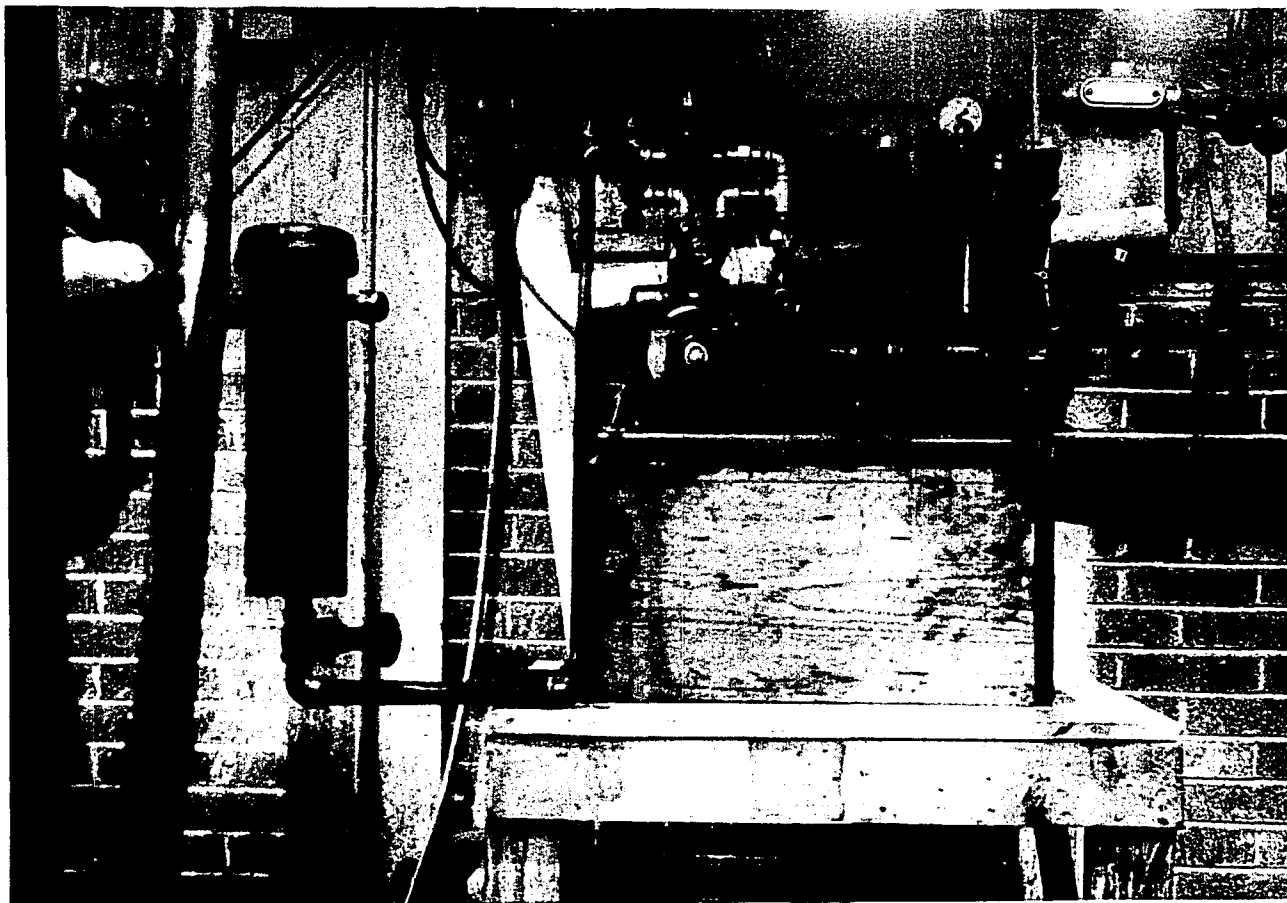
Run no.	Lb. fines (20% oil basis)	Lb. soy-beans	Fines as % of beans	Lb. oil	Fines as % of oil	Mesh screen
1	5.25	850	0.62	111	4.73	60
2	9.0	650	1.38	135	6.66	40
3	3.75	400	0.94	68	5.52	40
4	8.25	875	0.92	150	5.50	40
5	5.0	500	1.00	90	5.55	60
6	5.7	1792	0.32	300	1.90	40
7	9.6	2532	0.38	400	2.40	40
8	2.4	575	0.42	---	----	40

Filtration of dilute miscella. Attempts were made at first to eliminate the necessity for filtering the oil.

The falling film evaporator and stripping column used by Kircher (63) and McCracken (68) were used in preliminary work. However, the tubes became badly fouled with fines after a few runs and had to be cleaned.

Dilute miscella was filtered with a small Sparkler filter, which had three plates 8 inches in diameter and 1 inch deep. The filter was fed with a small Viking pump and worked very well at all times. It is shown in Photograph 2. The pressure remained well below 50 pounds per square inch until the filter was almost completely full. Circular sheets of paper formed the filter medium. Capacity of the filter was about 9 pounds of solvent-free fines. Before cleaning the filter was allowed to stand overnight. Then it was dismantled, the rather dry cake containing about 20 per cent oil was readily removed and the paper filtering medium discarded. No fouling of the evaporator or stripping column was observed when using filtered miscella.

Filtration of concentrated miscella. In the course of work on evaporation (described in the section on evaporation), it was found that connecting the rising film evaporator surge tank directly to the miscella outlet caused the miscella to surge back and forth through the screens and decreased the amount of fines leaving the extractor. The fines obtained readily passed through the evaporator and



Photograph 2.

Sparkler Filter Used in Pilot Plant

flash chamber, but they would not pass through the packed stripping column. Therefore, it was decided to investigate the possibility of filtering the concentrated miscella.

The following facts were considered:

Points in favor of filtering miscella before evaporation.

1. Lower viscosity, 1.37 centipoises for a 21 per cent solution at 37.8° C. compared to about 3.5 centipoises for an 80 per cent solution at 100° C. (60).
2. Less oil in the cake making the filter easier to clean and the cake easier to handle.
3. No possibility of fouling the evaporator with fines.

Points in favor of filtering concentrated (80 per cent oil) miscella.

1. With the evaporator surge tank connected to the miscella outlet fewer fines are obtained.
2. Only a third as large a volume of liquid has to be filtered.
3. The gravity of the concentrated miscella, 1.00, is lower than that of the fines, 1.23, so that part of the fines can settle out in the filter.
4. The vapor pressure of trichloroethylene at 30° C. in a solution containing 80 per cent oil is 25 mm.

of mercury compared to 100 mm. for a 20 per cent solution at the same temperature. Therefore, less solvent will be lost by evaporation.

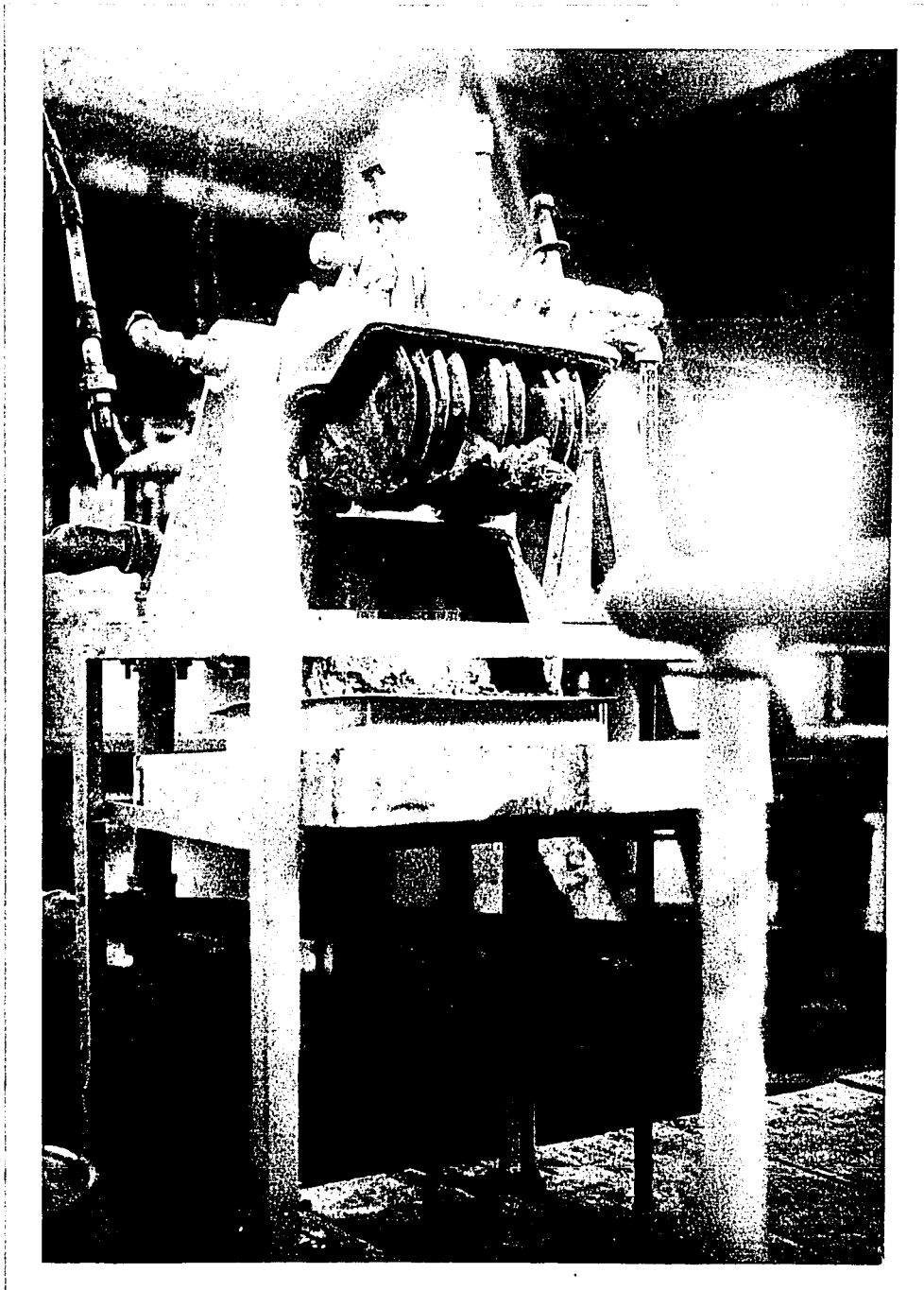
5. The evaporator flash tank was several feet higher than the top of the stripping column. The filter could be placed between the two using gravity feed, eliminating the need for a pump, and making the entire system in more perfect dynamic equilibrium.

Filtering concentrated miscella was tried with excellent results.

A bag filter was at first considered but was not adopted because it was thought that other types of filters would have greater capacity and be more easily cleaned. The small Sweetland filter shown in Photograph 3 was installed about 6 feet below the bottom of the flash chamber. A U-bend was placed in the filtrate outlet line to serve as a trap to prevent steam from the stripping column from wetting the filter cloth. During a run of 15 hours, 1792 pounds of beans were processed. At no time did the head of miscella exceed 3 feet above the top of the filter. After the run air was blown through the filter to aid in draining off the oil and the filter allowed to stand for 24 hours. When the filter was opened, it was

found that most of the fines had settled out filling the bottom fourth of the filter as shown in Photograph 3. About $5/6$ of the cloth was covered with fines in a layer from $1/8$ to $3/8$ of an inch thick. Evidently the filter would have run much longer before becoming full enough to reduce the rate of filtration. Six and a half pounds of fines containing 30 per cent oil were obtained. The cake had a density of 53 pounds per cubic foot. After the run the stripping column was opened and found to be clean. In view of the fact that for the last six hours of the run the good flakes were mixed with flakes containing considerable fines made from old, dry beans, the low yield of fines 0.32 per cent of the weight of the beans on the 20 per cent oil basis was considered remarkable.

The Sweetland filter was replaced by the small laboratory size Kelly filter shown in Photograph 4. The filter contained three leaves covered with filter canvas and had an inside diameter of 8 inches, a length of 19 inches and a total filtering area of 2.9 square feet. A run of 23 hours was made during which time the filter operated under a head of 2 to 3 feet of concentrated miscella. This time the filter was allowed to drain half a day and was then blown out with steam followed by air. Fines filled the lower fourth of the filter and covered all the



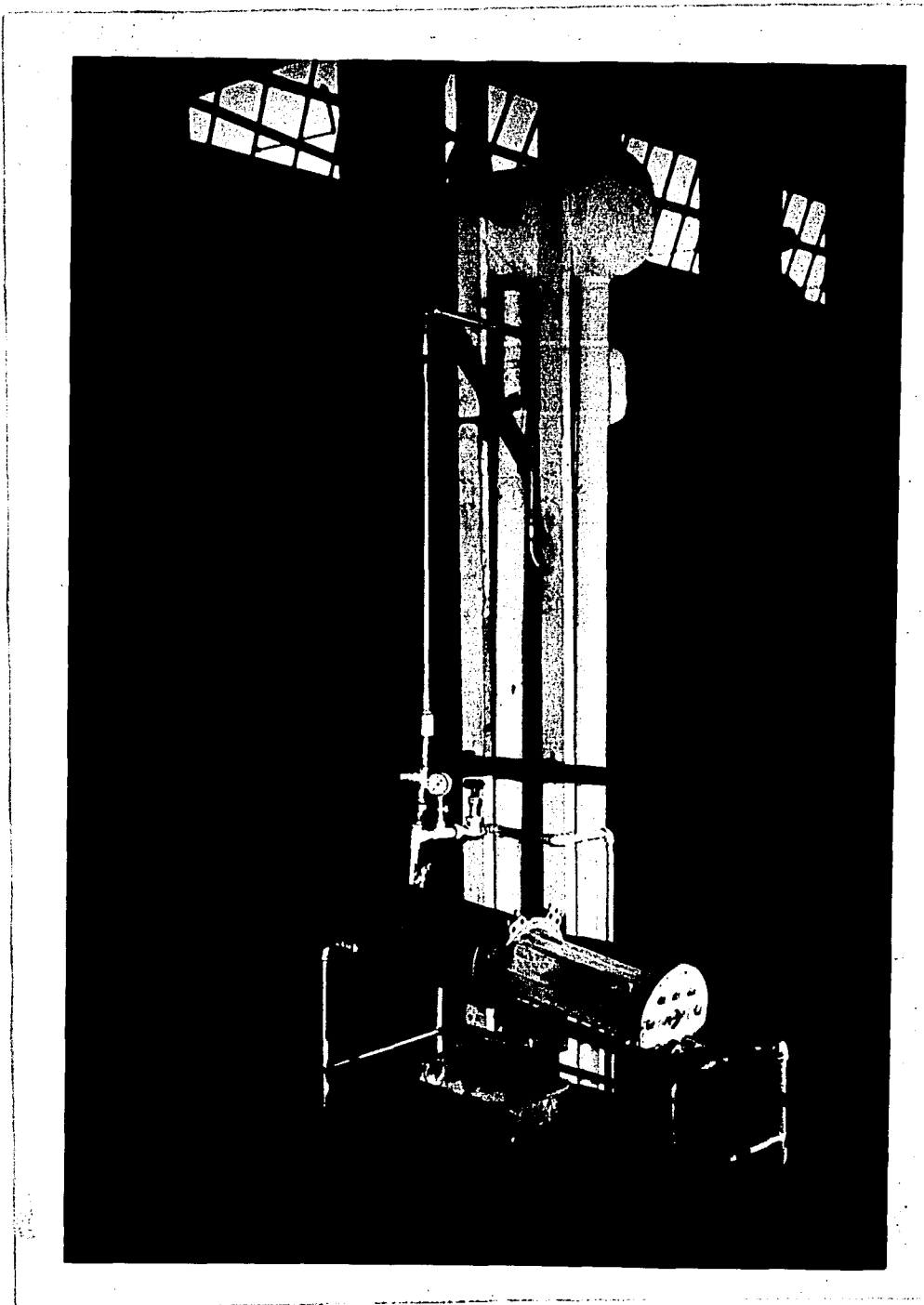
Photograph 3

Sweetland Filter Used in Pilot Plant

filter surface to a depth of $1/8$ of an inch. Steaming made the cake stick to the cloth so it was harder to remove than usual. Eleven pounds of fines containing 30 per cent oil were obtained from 2532 pounds of flakes, a yield of 0.38 per cent of the weight of the beans on the 20 per cent oil basis. Density of the cake was 59 pounds per cubic foot. The Kelly filter operated as well as the Sweetland and was more easily cleaned so it was used in all further work on the pilot plant. Between runs it was cleaned by removing the leaves and washing them in trichloroethylene.

Commercial plant filtration

For use with the Plainfield plant two homemade Kelly-type filters were constructed. The filtering medium consisted of six leaves, each leaf consisting of a canvas covered framework. The framework consisted of an upper pipe and a lower pipe ($1/2$ inch) with heavy wire netting stretched between them. Each of the pipes was fastened to and extended through a large plate which made up the front of the filter. The upper pipes were capped and the lower ones were fastened to a header for removing the miscella. A casing made of sheet metal 36 inches long, 24 inches wide, and 16 inches deep was suspended from rollers in such a way that it could be moved forward to



Photograph 4

View of Kelly Filter Used in Pilot Plant and
Top of Rising Film Evaporator

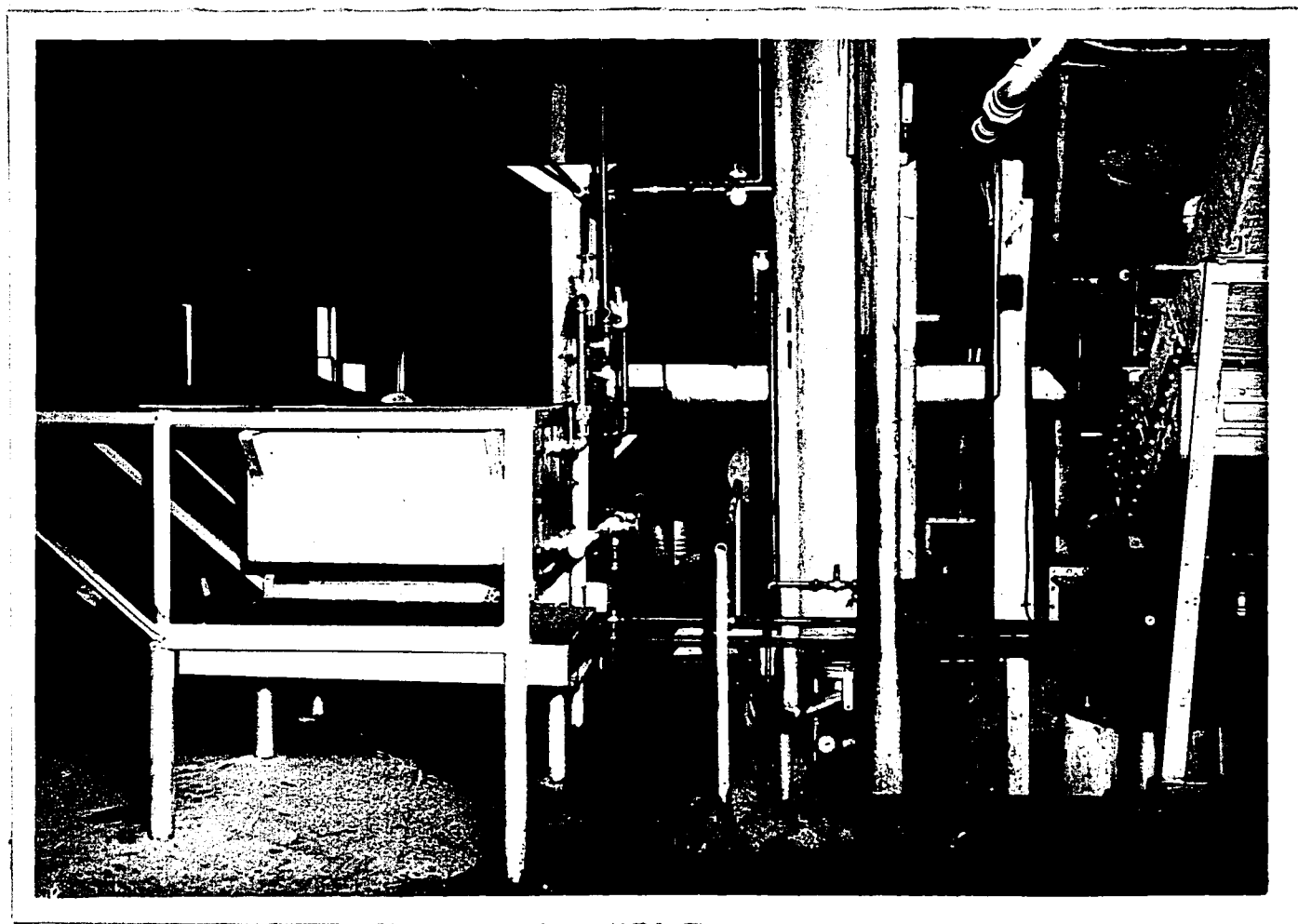
cover the six leaves and be fastened to the front plate. The filters are shown in Photograph 5.

Miscella to be filtered entered through a hole in the top center of the front plate. A vent was placed about 4 inches from the top but was later moved to the very top of the casing when it was discovered that solvent saturated air was being trapped between the vent and the top of the casing causing the top of the filter cloth to rot.

The filters were originally placed about 2 feet below the evaporator flash chamber so that the miscella could be filtered and run into the stripping column by gravity. Later the filters were moved to the first floor, about 10 feet below the flash chamber for the following reasons:

1. To give more pressure so the length of the filtering cycle could be increased;
2. Because the stripping column had been made taller and miscella had to be pumped into it anyway;
3. For ease and convenience in cleaning.

Canvas filter cloths were used at first and were cleaned after the cake was removed by pumping solvent back through the leaves until the filter was full. Later a special kind of "Soytex" filter cloth was used because it was said to have a longer life and to be more easily cleaned. One set of "Soytex" filter cloths lasted for six months of continuous operation and was removed only because



Photograph 5

View of Filters Used in Commercial Plant, Bottom of
Stripping Column, and Section of Extraction Chamber

improper venting caused them to rot at the top. Solvent was not pumped back through the leaves using the "Soytex" cloth. Instead the cake was scraped off and the cloths scrubbed with brushes dipped in trichloroethylene.

Ordinarily a filter would run for two to four days using 13 to 14 per cent moisture beans at 15 tons a day. The head on the filter gradually rose until it reached the flash chamber 10 feet above the top of the filters. With good flakes, more experience in operating the filters, and perhaps the use of a little auxiliary pressure one filter could probably be made to last for an entire week's run.

The low yield of fines obtained from the plant was most encouraging. The percentage of fines ranged from 0.08 to 0.03 per cent of the weight of the beans on the basis of 20 per cent oil in the fines, averaging perhaps 0.05 per cent. As in the pilot plant, most of the fines settled out in the bottom of the filter rather than remaining in suspension and being deposited on the leaves. Cake thickness varied from a quarter of an inch to a very thin smear depending upon the moisture content of the beans and the original condition of the cloth. Corn in the beans was also blamed at times for stopping up the filter. The cake contained about 40-50 per cent oil. Screen analysis data on the filter cake obtained from

the Plainfield plant are given in Table 3.

Table 3
Screen Analysis of Soybean Filter Cake

Mesh (Tyler std.)	% Retained	Cumulative % retained
35	0.32	0.32
48	6.27	6.59
60	9.28	15.87
80	15.23	31.10
100	11.45	42.55
150	15.34	57.89
200	19.93	77.82
270	20.24	98.06
Passing		
270	1.94	100.00

Disposal of fines

Two methods of disposal of the fines were tried. First the fines were mixed with flakes entering the extractor in the ratio of 1 pound of fines to 100 pounds of flakes. Of the fines which were added, 67 per cent passed out with the miscella and entered the filter. In another experiment fines were run through the flaking rolls along with the cracked beans in an attempt to press them into the flakes. The flakes were screened on a

40 mesh screen and found to contain 11.2 per cent fines compared to 4.9 per cent for ordinary flakes. The additional amount of fines screened was a large proportion of those added so this method was discarded. Feeding the fines back into the extractor with the flakes was not tried in the Plainfield plant.

Recommendations

- a. A means of continuously scraping the cake from the filter should be tried.
- b. Filtering dilute miscella should be further investigated.
- c. A type of preliminary filter other than 40 mesh screens on the miscella outlets to keep more fines from leaving the extractor might be desirable.
- d. The use of additional pressure on the filters might increase the length of filter cycles enough to be economical.
- e. Some method for economic disposal of the fines should be sought.
- f. Running the concentrated miscella through some kind of a settling chamber might increase the length of the filter cycle.

Evaporation

Falling film evaporator

The evaporator used at the beginning of this work was the same as that used and described by Kircher (63) and McCracken (68). It consisted of a shell of 5-inch standard iron pipe containing 19 brass tubes 5 feet long ($3/4$ inch O.D., No. 20 B.W.G.). The tubes were expanded into headers at the top and bottom of the shell. The tubes were carefully leveled across the top and the entire assembly was always kept perfectly vertical so the miscella would run down all the tubes uniformly. Steam pressure used in the shell was 30 to 40 pounds per square inch. Miscella entered at the top directly from the extractor, ran over an inverted cone, and ran down the tubes in thin films. Miscella at 30° C. containing 20 per cent oil could be concentrated to miscella at 110° C. containing 60 per cent oil at a rate of 25 pounds of oil an hour. Under these conditions the heat transfer coefficient based on the inside area of the tubes was 8.0 B.t.u./hr./sq.ft./ $^{\circ}$ F.

The stripping section used by Kircher and McCracken was identical with the evaporating section and was directly below it, the two being separated by a spool piece containing the vapor outlet. When it was found that the

stripping section was leaving solvent in the oil (see the following section on stripping), that section was used as an evaporator to supplement the upper section. Using the combined evaporators and steam at 40 pounds per square inch miscella could be concentrated to 80 per cent oil at a rate of 25 pounds of oil an hour. The combined evaporator did not darken the oil noticeably and might have been used had it not been replaced by a more efficient evaporator.

Rising film evaporators

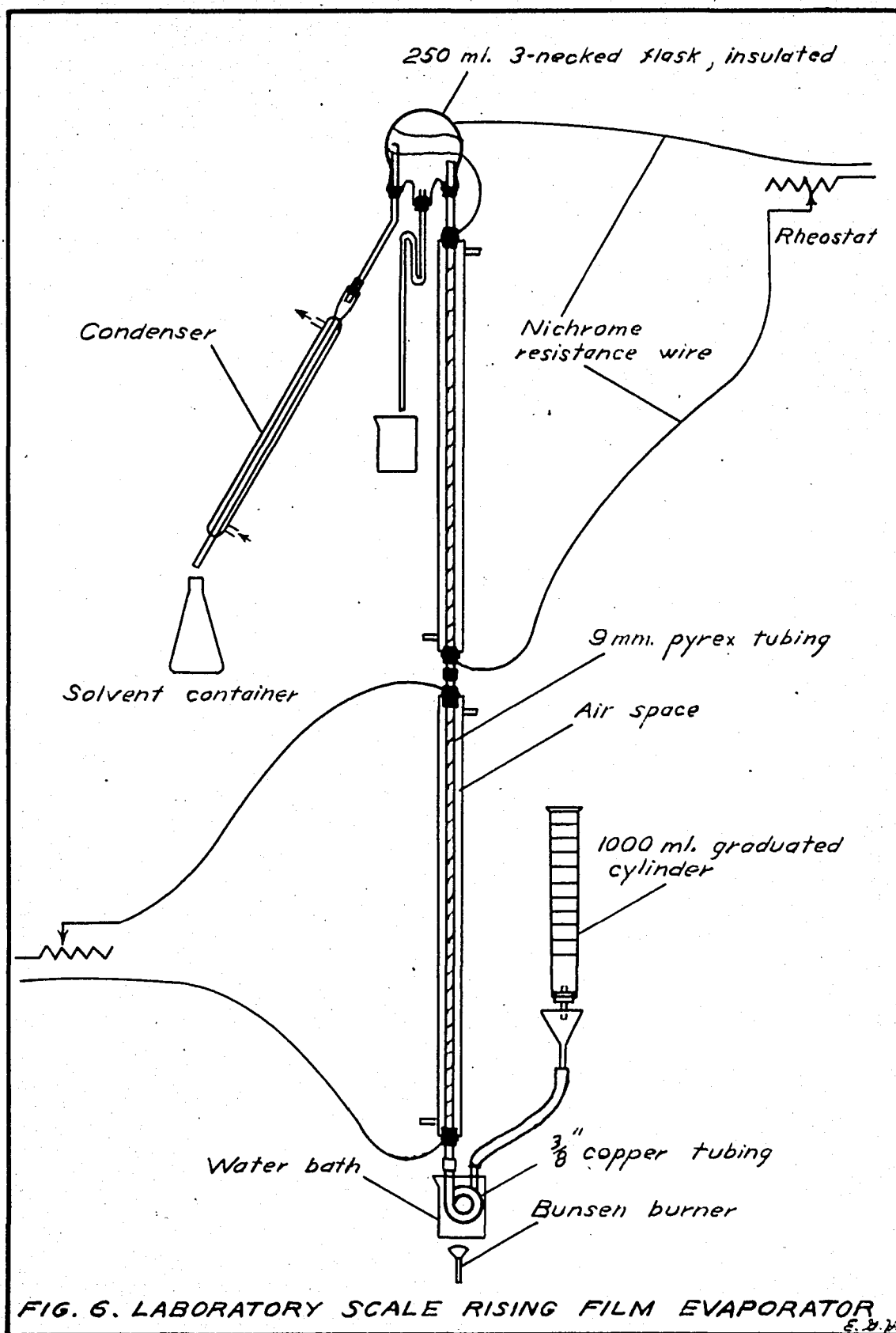
Laboratory scale apparatus. The rising film principle for evaporation of hexane from soybean oil-hexane solutions is being used in some of the more modern solvent plants. Research concerning the application of long tube rising film evaporators to trichloroethylene miscella was carried out at Iowa State College and has resulted in rather unique designs and methods of operation.

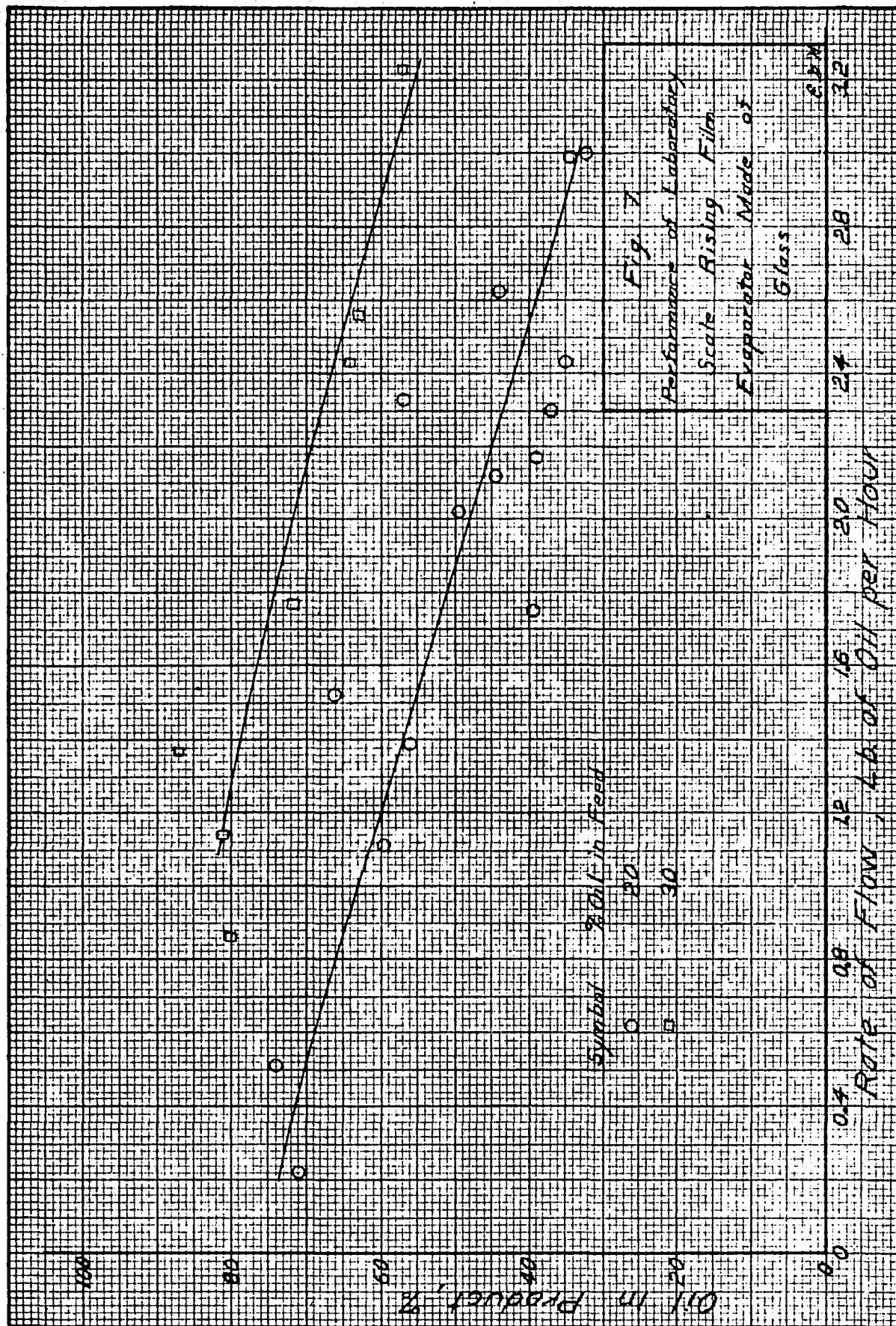
In connection with his senior research problem Havlik (53) in 1944 found that a trichloroethylene-soybean oil solution could be concentrated using the rising film principle even with rather crude apparatus. Havlik refined his apparatus to that shown in Fig. 6. The column was made of two glass Liebig condensers fastened together with a short piece of rubber tubing, which held the

glass-to-glass contact in place. Resistance wire was wound around the inner glass tube (9 mm. in diameter) for a length of 6 feet 8 inches. The feed was kept at a constant head by a chicken-feeder type of arrangement. Preheating the feed aided somewhat probably because of the low heat transfer coefficient through the glass. Data taken on this apparatus are plotted in Fig. 7. The rising film action could be seen in this apparatus and is described by Havlik as follows:

"As the liquid boiled in the tube, a foam of variable thickness persisted above the surface. Above this foamy area each bubble gradually spread entirely across the tube until it became shaped like a fairly thick circular disc. The existence of this disc was possible because of surface tension and because the pressure was slightly greater beneath it than it was on the upper side. Each disc was made up of a solution of oil and solvent, the solvent constantly evaporating from both the top and bottom surfaces of the disc. Evaporation also took place from the liquid film adhering to the sides of the tube. The formation of the vapor pushed the discs apart and since they originated at the bottom of the column, the discs were pushed upward. The velocity of each disc was affected not only by the vaporization of the solvent from the particular disc but also by the vaporization from every disc below it. Also, a slight additional expansion of vapor was caused by the decrease in pressure and increase in temperature as the disc moved up the tube. For these reasons the velocity of the disc was being constantly increased until it left the top of the tube."

In order to obtain better heat transfer the two vertical Liebig condensers shown in Fig. 6 were replaced by a single length of 3/8-inch pipe 8 feet long. The pipe was wrapped with a single thickness of asbestos paper,





then with resistance wire and then with three layers of asbestos paper. This apparatus had a greater capacity than that shown in Fig. 6 and operated more smoothly. Concentrated miscella came out of the top of the pipe in little gushes, which struck against the top of the flask. Data on this evaporator are given in Table 4.

Table 4

Performance of Laboratory Scale Rising Film Evaporator

Rate (lb. oil per hr.)	Oil in product %	Rate (lb. oil per hr.)	Oil in product %
1.91	60.9	1.63	63.5
1.81	52.0	1.63	71.0
1.79	56.7	1.53	82.5
1.73	69.0	1.46	73.5
1.73	59.0	1.11	76.0
1.73	56.0	1.10	84.0
1.73	56.0	1.05	84.4
1.73	56.0		

Forty mesh fines were placed in the feed in the ratio of 14 grams of fines to 1.1 liters of feed. The fines tended to stop up the U-bend leading from the flash bottle but left the 3/8-inch pipe perfectly clean.

Pilot plant scale rising film evaporators. The next evaporator constructed was that shown in Fig. 8. The evaporator consisted of a single, vertical, $\frac{1}{2}$ -inch pipe jacketed to a length of 12 feet with a $1\frac{1}{4}$ -inch pipe. The outer pipe was lagged with 85 per cent magnesia insulation.

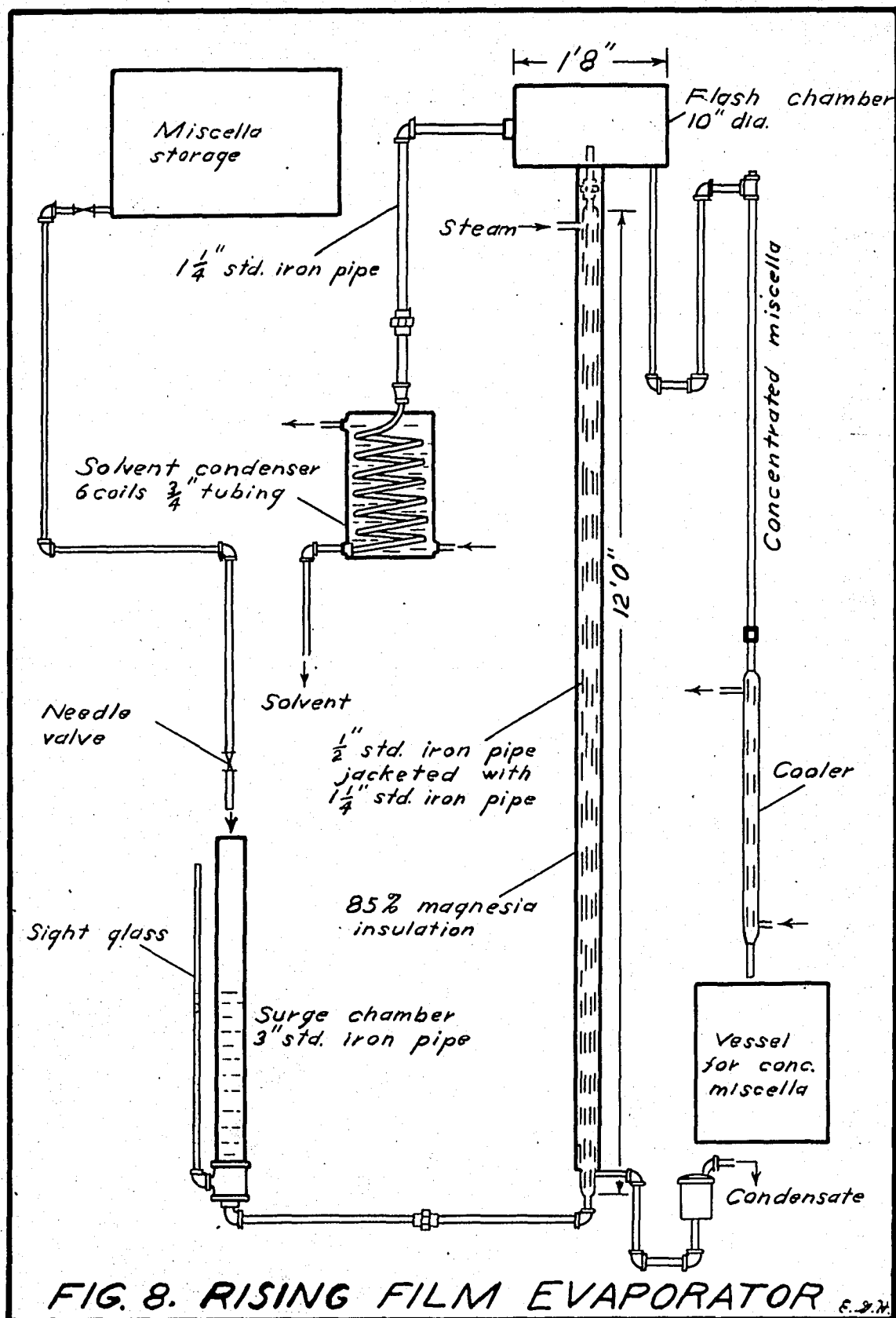
The top of the $\frac{1}{2}$ -inch pipe projected 3 inches into an insulated flash chamber.

At first the flash chamber consisted of a one gallon can and the feed tank of a ten gallon carboy. Later the can was replaced by a 20 inch by 10 inch diameter tank lying on its side and the ten gallon carboy by a 50 gallon tank. Miscella from the feed tank entered a 3-inch pipe 4 feet long, which served as a surge tank, and ran through a $\frac{1}{2}$ -inch pipe to the evaporator. The evaporator would adjust itself to different rates of feed by an increase or decrease in the head of liquid in the surge tank. For experimental purposes the head was held constant at certain levels by manual operation of a needle valve controlling the rate of flow into the surge tank. Data using the apparatus described are summarized in Table 5.

Table 5

Performance of Rising Film Evaporator
Made of $\frac{1}{2}$ -Inch Pipe 12 Feet Long

Rate (lb. oil per hr.)	% Oil in product	Head (inches)	'Length' 'of run' '(min.)'	Steam pressure (lb./sq. in.)	% Oil in feed
13.0	89	12.5	105	80	20
13.9	87	17.5	45	75	20
14.1	88	14.5	90	80	20
15.5	83	16.0	90	77	20
19.2	84	21.0	65	67	20
21.0	75	25.5	85	77	20



The data in Table 5 show that soybean oil miscella may be concentrated from 20 per cent to 80 per cent oil at a rate of 20 pounds per hour of oil using the apparatus described. The overall heat transfer coefficient based on the inside area was 54 B.t.u./hr./sq.ft./° F.

Concentrated miscella left the evaporator at 135° C. The action taking place within the evaporator could not be seen, but it was much more vigorous than that in the laboratory scale evaporators. Vapor and liquid struck the top of the flash chamber with great force every 2½ seconds. As the vapor and liquid spurted out at one end, the contents of the surge tank surged or "burped" back and forth.

In an attempt to increase the capacity the evaporator was replaced by another ¾-inch standard iron pipe jacketed to a length of 20 feet with 1½-inch pipe. The flash chamber and surge tank used on the evaporator just described were used. The data in Table 6 show results of runs made using the longer evaporator.

The data in Table 6 are somewhat erratic perhaps because the runs were not long enough to reduce the effect of starting and stopping conditions. Nevertheless, these data show that the capacity of the 20 foot evaporator was not much greater than that of the 12 foot evaporator

using $\frac{1}{8}$ -inch pipe.

Table 6

Performance of Rising Film Evaporator
Made of $\frac{1}{8}$ -Inch Pipe 20 Feet Long

Rate (lb. oil per hr.)	% Oil in product	Head (inches)	Length of run (min.)	Steam pressure (lb./sq.in.)	% Oil in feed
14.8	83	31	135	77	20
17.0	84	31	115	74	20
18.0	81	25	105	77	20
18.0	86.6	36	110	78	20
18.9	85.4	36	100	74	20
20.0	75	48	90	74	20
20.1	76	42	90	76	20
20.4	81.0	42	80	74	20
20.6	86.6	36	85	78	20
27.0	80.7	36	45	76	20

The 20 foot evaporator would concentrate miscella from 20 to 80 per cent oil at a rate of 23 pounds of oil per hour. The head required for a given rate of flow using the 20 foot pipe was higher than that required for a 12 foot pipe as shown in Tables 5 and 6. This difference was due mainly to the greater height to which the liquid and vapor had to be pushed. It was also likely that in a $\frac{1}{8}$ -inch pipe friction increased so rapidly with increase in rate that even very high heads would not greatly increase the capacity.

At this stage in the evaporator research an evaporator was connected to the pilot plant. This consisted of two

$\frac{1}{8}$ -inch pipes jacketed to a length of 20 feet and projecting 4 inches into the 20 inch by 10 inch diameter flash chamber shown in Fig. 8. Feed was introduced directly from the extractor into a 3-inch pipe 4 feet long, which served as a surge tank. Two $\frac{1}{8}$ -inch pipes 4 feet long fitted with gate valves ran from the surge tank into the evaporator tubes. Vapor was removed through a $1\frac{1}{2}$ -inch pipe leading from the side of the flash tank to the condenser and fitted with a baffle plate. At first a separate condenser was used for the evaporator, but later the vapor was run into the same condenser used by the rest of the system with no apparent change in behavior. Concentrated miscella ran out the bottom of the flash chamber through a U-bend trap to the filter. The upper part of apparatus is shown in Photograph 4. The evaporator was oversize for the pilot plant because it should have been capable of handling over 40 pounds of oil an hour, while the plant produced only 25. When running with the plant, the evaporator concentrated miscella to from 78 to 84 per cent oil.

The level of miscella in the surge tank varied from 12 to 36 inches above the bottom of the steam jackets, usually remaining at about 14 inches. Whether the tubes flashed together or separately was not determined, but

at any rate flashing occurred about every two seconds, with an accompanying surge of about 3 inches in the surge tank.

The behavior of the system at the miscella outlets was quite interesting. The upper three-fourths of the screens (described in the section on filtration) was covered with damp fines, which were not continually working through as usual. The miscella running out the lower fourth of the screens would run out, then temporarily reverse in direction, and then run out again due to pressure differences caused by the intermittent action of the evaporator. As air and vapor were pushed back out of the surge tank they entrained liquid which splashed upon the screens and kept them wet. Even when good flakes were mixed with flakes made from old, dry beans the percentage of fines was low. It seemed that the action described was the cause for the reduction of fines; however, this phase of the problem is still being investigated.

Since the capacity of the evaporator could not be greatly increased by making it taller, it was decided to try using a larger pipe. A 1-inch standard iron pipe jacketed to a length of 11 feet 3 inches was available and was used for the purpose. The apparatus was similar to that shown in Fig. 8. Feed entered a surge tank made of 3-inch pipe in which the head was kept as nearly constant

as possible. Miscella ran from the surge tank into the evaporator through a horizontal $\frac{1}{2}$ -inch pipe. The flash chamber was a can 22 inches by 12 inches in diameter lying on its side. Solvent vapor was carried by a 1-inch pipe to a $\frac{1}{2}$ -inch copper coil immersed in cooling water. Data for this evaporator are given in Table 7.

Table 7

Performance of Rising Film Evaporator
Made of 1-Inch Pipe

Rate (lb. oil per hr.)	% Oil in product	Head (inches)	Length of run (min.)	Steam pressure (lb./sq.in.)	% Oil in feed
30.0	82	16	108	73	20.0
32.7	79	16	60	30	20.0
34.5	84	16	85	68	22.0
39.7	78	12*	90	--	19.1
41.6	82	22	60	72	20.6
45.6	83	22	74	83	22.4
47.4	79	28	60	78	20.0
51.6	71	28	60	--	20.0
57.9	72	34	60	80	21.0
51.4	89	22	105	88	30.0
52.0	92	26	95	85	40.0

*Miscella preheated to 60° C.

When concentrating 40 pounds per hour of oil from a 20 per cent to an 80 per cent solution the overall heat transfer coefficient based on the inside area was 68 B.t.u./hr./sq.ft./° F. with the steam pressure at 75 pounds per square inch. Preheating the miscella gave no advantages which could not be obtained by raising the head somewhat.

Usually the surge tank was placed 4 feet from the evaporator, but this distance could be decreased to 14 inches without having vapor back up into the tank. The evaporator flashed every three seconds and the temperature in the flash chamber was about 135° C. Miscella containing 30 or 40 per cent oil produced surges of only about $\frac{1}{4}$ inch in the surge tank compared to 2 or 3 inches using 20 per cent miscella. The more concentrated miscella would stop going through the evaporator at all if the head dropped much below the values shown in Table 7.

The 1-inch pipe had roughly four times as large a cross sectional area as the $\frac{1}{2}$ -inch pipe but was handling only twice as much miscella. This was expected because the heating area was twice as great. However, the 1-inch pipe should handle four times as much miscella as the $\frac{1}{2}$ -inch pipe before the vapor velocity would become as great. Therefore, it was decided to leave the diameter at 1 inch and increase the height considerably for the commercial plant.

Commercial plant evaporation

For the commercial extraction system an evaporator made of six 1-inch pipes jacketed to a length of 17 feet and placed side-by-side was constructed. From the bottom of the jacketed pipes (tubes) six 1-inch horizontal pipes

4 feet long connected into the bottom of a surge tank. The surge tank was 4 feet high, 28 inches long, and 4 inches wide and was placed directly below the miscella outlets. From the top of the tubes six 1-inch long sweep elbows ran directly into the front of the flash chamber. The flash chamber was 18 inches high, 30 inches wide, and 4 feet long. A baffle arrangement was placed inside the flash chamber to reduce entrainment of liquid particles by the vapor. A 5-inch vapor line led from the front of the flash chamber just above the tube inlets. Concentrated miscella was withdrawn from the bottom of the flash chamber through a U-bend trap and ran directly into a filter.

When the evaporator was first tested it was observed that the tubes flashed separately. Even so, a pronounced surging effect took place in the surge tank. The miscella had a tendency to flow intermittently through the screens due to the ratchet drive on the extractor. When miscella was run through the screens with the extractor turned off, it was observed that surging affected the flow much less than in the pilot plant. This decrease in the amount of surging may have been due to the cushioning effect of the air and vapor in the large chambers enclosing the miscella outlets in the commercial plant. Whether, then, the surging effect produced by connecting the evaporator directly to the miscella outlets was in part responsible for the

extremely low percentage of fines obtained in the commercial plant was not determined conclusively.

With the plant processing from 8 to 10 tons of beans a day, all six evaporator tubes were used at first. When, after about three months, the degree of concentration fell from 83 to 75 per cent the tubes were opened and found to be covered inside with a layer of fines and hardened oil. This fouling was believed to have been caused by their running dry part of the time; so after they were cleaned three were cut out of the system. As an additional precaution, steam was cut off the tubes every time the plant had to be shut down. Three tubes had sufficient capacity to handle the plant at rates up to 12 tons of beans a day. They kept themselves well flushed and rarely had to be cleaned. Cleaning consisted of running an auger down through the pipes to remove the bulk of the deposited material and loosen the remainder.

When concentrating the oil from 12 tons of beans a day (4,200 pounds) in miscella containing 20 per cent oil to miscella containing 80 per cent oil, with 95 pounds per square inch of steam on the jackets, and miscella entering at 110° F. and leaving at 280° F., the evaporator had an overall heat transfer coefficient based on the inside area of 65 B.t.u./hr./sq.ft./° F.

Recommendations for further study

a. The optimum height, diameter, and steam pressure for operation of a rising film evaporator for soybean oil-trichloroethylene solutions should be further investigated.

b. More concentrated miscella may be prepared for the packed column by recirculating part of the miscella from the flash chamber back through the evaporator. If such practice would not deteriorate the quality of the oil, a saving in steam could be realized.

c. A cyclone separator type of flash chamber might give better results than a rectangular box.

d. The evaporators should be installed in such a manner that they could be drained and cleaned without shutting down the plant. They should be made of very smooth pipe for ease in cleaning.

e. The effect of the surging action upon the amount of fines produced in the plants should be studied further.

Stripping

Preliminary stripping

Early in this research the falling film stripping column used by Kircher (63) and McCracken (68) was used

in a large number of runs. The column worked well except for the fact that the tubes gradually became fouled with fines unless the miscella was filtered. Another trouble with the stripping section was that unless an excessive amount of steam was used in stripping, the oil contained from 2 to 4 per cent solvent when running at a rate of 25 pounds of oil an hour.

After the rising film evaporator was developed, the column was run in a different manner. Concentrated miscella from the evaporator containing 80 per cent oil was run into the upper section of the column. Steam was blown into the stripping section and both sections were steam heated. Oil samples taken during a run showed 0.8, 0.6, and 0.2 per cent solvent. The latter percentage was obtained only by turning on so much steam that part of the oil was entrained by the vapor.

Disk stripper

A stripping apparatus similar in construction to the Porcion type of evaporator was made in hopes that it would handle fines and strip the oil completely. The stripper consisted of a series of sheet metal disks partially (about $1/3$) submerged in miscella and enclosed in a tight metal box heated on the underside by Meker burners. Construction data were as follows:

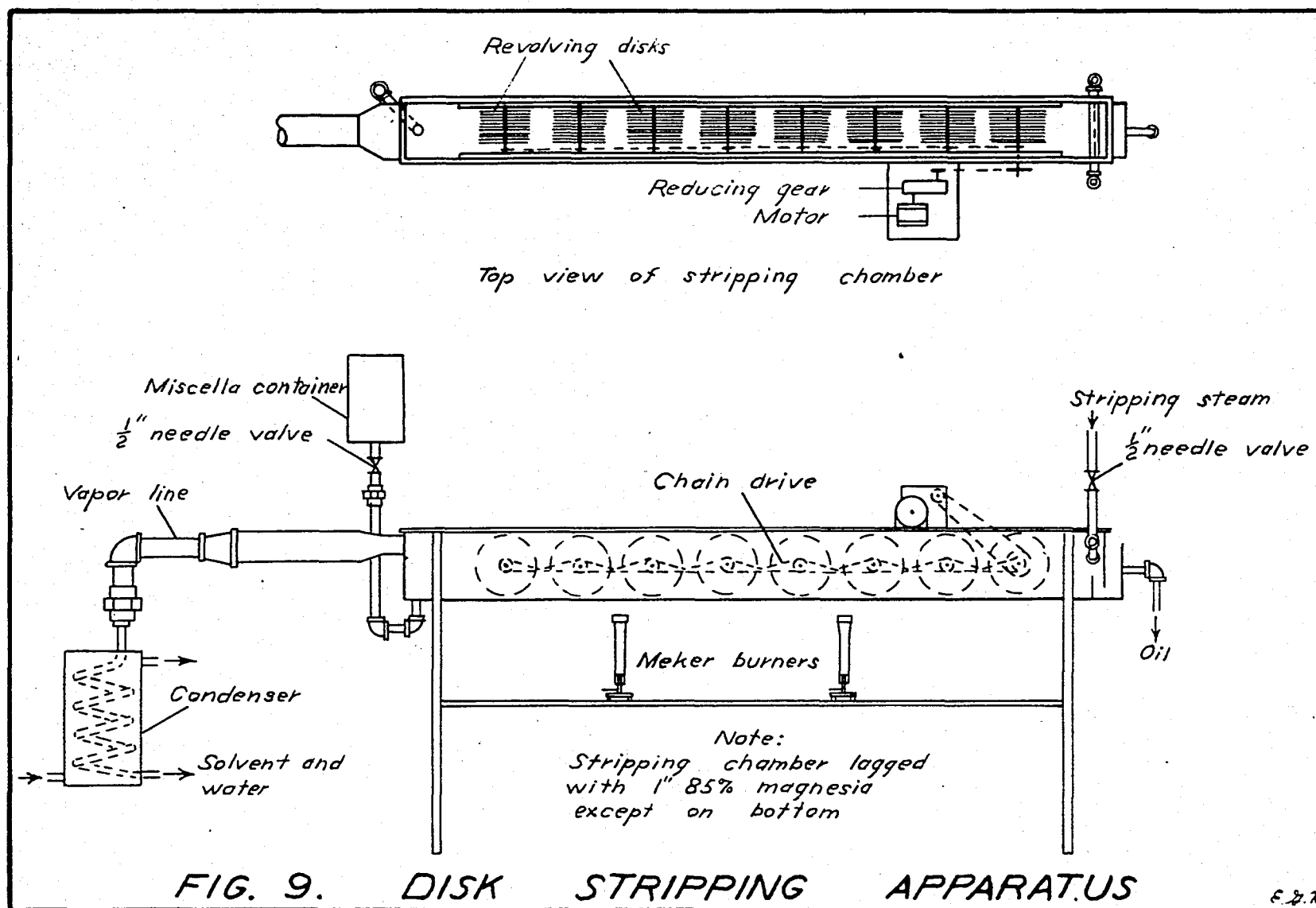
Metal box	6½ inches by 7½ inches by 6 feet
Diameter of disks	6 1/8 inches
Number of disks per shaft	15
Number of shafts	8
Size of motor	½ h.p.
Speed reducer	home made
Speed at reducer	32 r.p.m.

The stripper is shown in Fig. 9. Miscella fed to the stripper was made up to approximate that which could be obtained from the old stripping column consisting of brass tubes surrounded by a steam jacket. The feed was preheated to 110 to 130° C., and the entire apparatus except for the underside was lagged. During the first few runs heat was supplied by the Meker burners at such a rate that the temperature of the product ranged from 150 to 165° C. At such temperatures the solder holding the disks to the shaft melted. The data in Table 8 were taken after the disks had been securely bolted to the shaft. The temperature of the product then ranged from 120 to 120° C.

Table 8

Performance of Disk Stripping Apparatus

Feed rate (lb./hr.)	Tri. in product (%)	Steam rate (lb./hr.)	Speed at disks (rev./min.)	Tri. in feed (%)
20-25	0.4	----	25.6	2.0
27	0.4	14	64	2.0
28	0.25	14.4	40	2.0
30	0.1	12	40	2.0
31.8	0.3	12.1	40	2.0
33.6	0.45	14.5	25.6	2.0
25	0.4	10	25.6	6.0



The apparatus probably stripped the oil better at 40 revolutions per minute than at 64 because centrifugal force threw off much of the oil film on the disks. At rates of speed below 40 revolutions per minute the oil was exposed less frequently to the gaseous phase consisting mainly of stripping steam. The oil was darkened considerably by overheating and was not stripped completely in any run.

A strip of sheet metal 1 inch by 2 inches when dipped into oil at 120° C. removed an average of 0.22 grams of oil each time it was lifted out. By direct proportion a piece of sheet metal with an area of one square foot should remove 7.92 grams. A stripping apparatus made of 120 disks 6 inches in diameter has an area of 47.2 square feet. If 30 per cent of the area were submerged, 33.0 square feet would be exposed at any given time covered with 0.575 pounds of oil. With oil travelling at 25 pounds per hour through the apparatus each particle of oil could be exposed in a thin film for 1.38 minutes.

Stoneware stripping column

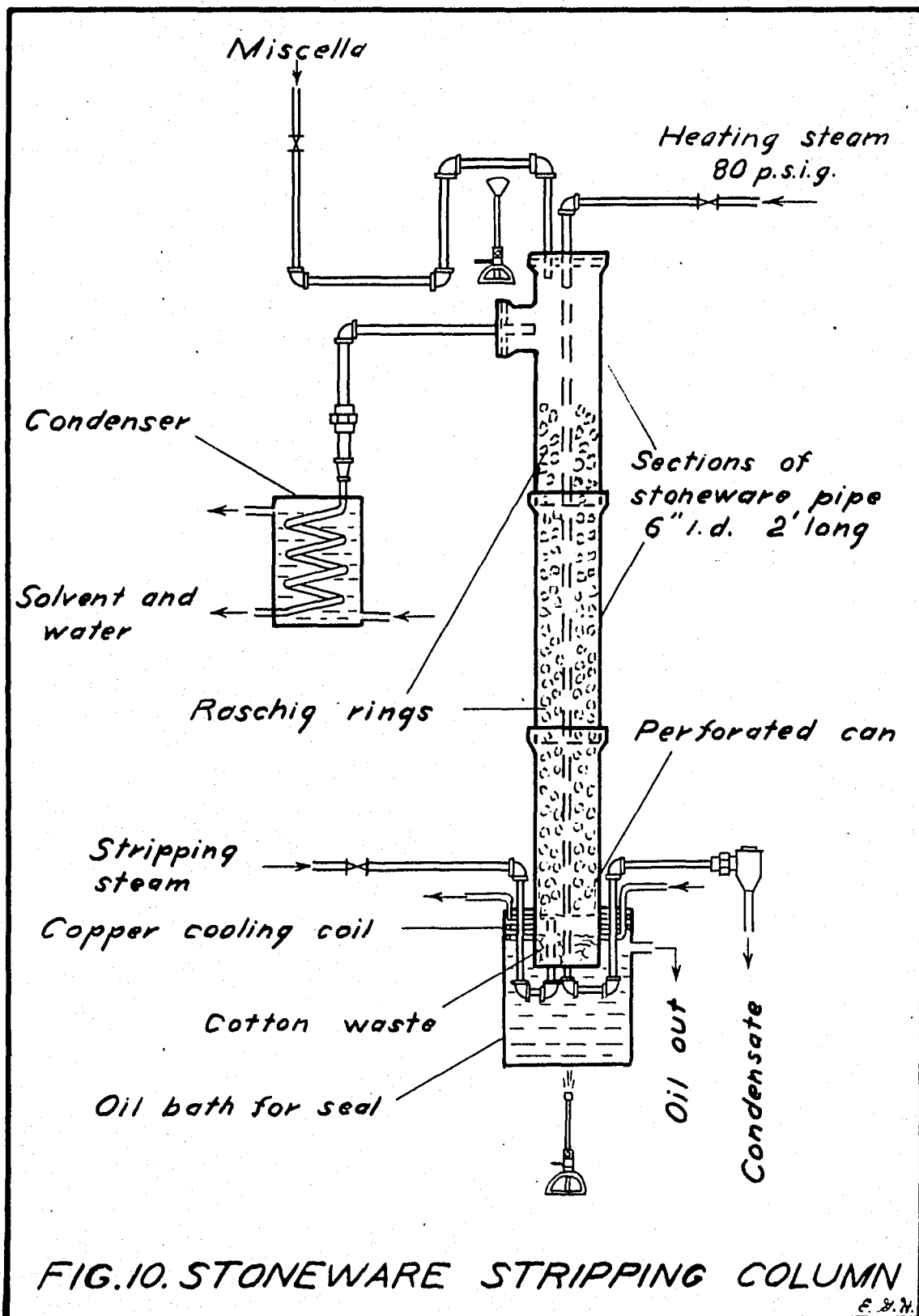
After it became apparent that no simple type of stripping apparatus capable of handling fines was immediately forthcoming, it was decided to compromise and develop a column which would strip filtered miscella. Steel was

rather scarce at the time; so the first column was made of stoneware.

Two straight sections of 6-inch stoneware sewer tile were surmounted by a section the same size with a side outlet. Pipe outlets in the upper end were made through wood plates sealed into place with a mixture of litharge and glycerin. Joints between tile were packed with oakum then covered with cement. At first the lower end was open but could be sealed by placing it in a bucket of oil. The apparatus is shown in Fig. 10.

The lower $2\frac{1}{2}$ feet of the column was packed with 1-inch Raschig rings while the upper 1 foot was packed with $\frac{3}{4}$ -inch rings deposited at random. A $\frac{3}{4}$ -inch pipe connected to a 90 pounds per square inch steam line ran vertically through the column to supply additional heat. Stripping steam was obtained by throttling 90 pound steam with globe valves. A burner was provided so that the miscella could be heated. The condenser was made of coils of $\frac{3}{8}$ -inch copper tubing immersed in a water bath.

During the first run it was found that the level of oil in the bucket could not be raised sufficiently high to keep steam from bubbling out. This difficulty was overcome by packing the lower end of the column with cotton waste, which was sufficiently porous to allow oil to run out but offered enough resistance to keep steam



from coming out. In spite of the heating pipe down the middle of the column, about eight pounds of steam an hour condensed and ran out with the oil. This mixture in contact with the hot pipes at the bottom had a tendency to foam. Therefore, three coils of copper tubing through which cooling water circulated were placed inside the top of the bucket. Except in extreme cases (when the bucket was heated strongly with a burner) the coils broke down the foam sufficiently to prevent it from overflowing.

After the first few runs one vertical crack appeared in each of the three sections of pipe. These were sealed somewhat with strips of wood over asbestos gaskets. However, steam still escaped through the cracks and around the wood disks in the top of the column. Attempts to seal these cracks were unsuccessful. Increasing the size of the vapor outlet would have helped by reducing pressure at the top of the column.

Feed was made up by adding trichloroethylene to oil and was of the strength that could easily be produced by the falling film evaporator and stripping column. In color and appearance the quality of the oil was satisfactory. However, it always contained a considerable amount of water. This water settled within a few hours leaving clear oil at the top. Despite the low heat transfer

coefficient of the tile, the outside of the column was quite hot during the runs. A certain amount of condensation might in some cases be useful for throwing out phosphatides and stripping last traces of solvent from the oil. This being the case, the worst defect of the stoneware stripping column was its tendency to crack.

As shown in Table 9, the column would strip 15 pounds per hour of oil containing 2 per cent trichloroethylene to 0.1 per cent or less solvent. It would strip 15 pounds per hour of oil containing 4 per cent solvent to 0.2 per cent or less. This performance was better than that of the disk stripper; so work on packed columns was continued.

Table 9

Performance of Stoneware Stripping Column

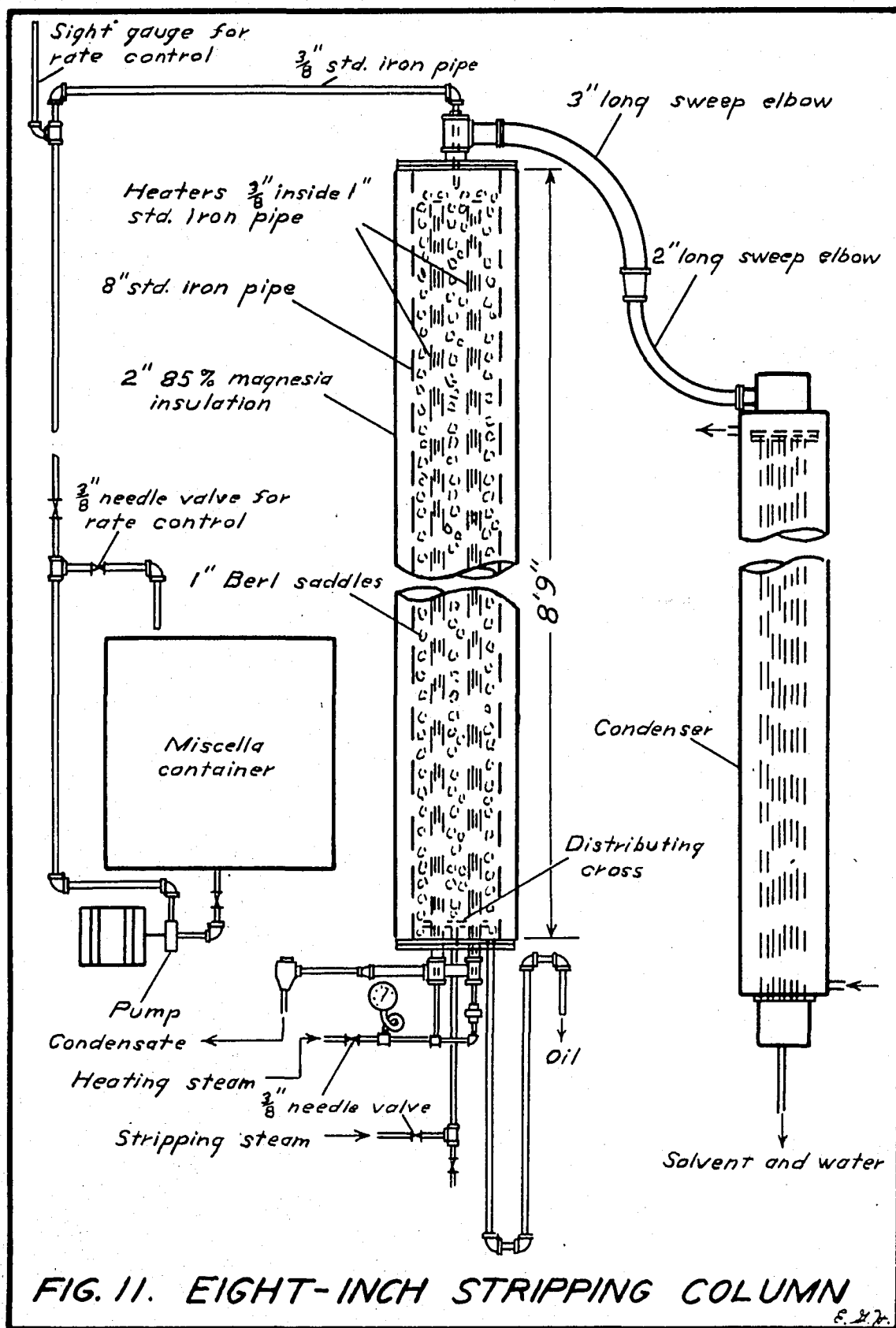
Feed rate (lb.oil/hr.)	Tri. in product (%)	Steam to condenser (lb./hr.)	Steam condensed in oil (lb./hr.)	Tri. in feed (%)
8.5	0.00	7	8	2
9	0.05	7.5	8	2
9.5	0.3	11	8	2
13	0.1	9	8	2
15	0.2	-	8	2
17	0.05	-	8	2
17	0.5	10	8	2
17	0.1	8	8	2
20	0.1	10	8	2
24	0.0	1	8	2
16	0.3	7	8	4.3
16	0.2	7	8	4.3
16	0.6	8	8	11

Packed columns operated above 100° C

Columns with ceramic packing. A stripping column was constructed of an 8-inch standard iron pipe 9 feet long. The column was flanged at each end and covered with 2 inches of 85 per cent magnesia insulation.

The column was to be operated above 100° C. so it was necessary to supply additional heat. This heat was supplied by a rather unique design which eliminated the necessity for expansion joints. Four 1-inch pipes placed in a 4½ inch diameter circle projected through the bottom plate and up to within 6 inches of the top of the column. The pipes were welded closed at the top and connected to tees at the bottom. Within these 1-inch pipes were placed four 3/8-inch pipes, which were open at the top and connected to a steam line at the bottom. Steam entered the 3/8-inch pipes, rose in them until it reached the top, descended the 1-inch pipes, and passed out the side outlets of the 1-inch tees as condensate.

Stripping steam entered the bottom of the column and was distributed through a perforated cross. Vapor left the column through a 3-inch tee at the top and passed through a long sweep elbow to a condenser. Feed was introduced through a 3/8-inch pipe projecting through the tee in the top. The column is shown in Fig. 11.



The column was first filled $3/4$ full of $3/4$ -inch Raschig rings and $1/4$ full of 1-inch rings because not enough of either were available to fill the column. Miscella was made up by adding trichloroethylene to oil obtained from the pilot plant and previously stripped. It was pumped into the top of the column at room temperature. Steam pressure on the pipes was usually 50 pounds per square inch. Data taken on several runs are summarized in Table 10. These data are rather erratic and are of value mainly as an indication of the amount of stripping steam required. Runs ranged from $2\frac{1}{2}$ to 4 hours in length. The oil rates given are those at which the oil was completely stripped. The capacity of the column using the Raschig rings was not determined accurately but was believed to be in the neighborhood of 100 pounds of oil per hour when using 60 pounds of stripping steam an hour.

Table 10

Column Packed with $3/4$ and 1-Inch Raschig Rings

Tri. in feed (%)	Feed rate (lb. oil/hr.)	Stripping steam (lb./hr.)	Lb. oil per lb. steam
2	50	19	2.63
4	70	21	3.33
10	50	27	1.85
12	60	21	2.86
14	50	21	2.38
16	40	27	1.48
20	44	27	1.63
25	38	21	1.81
30	45	28	1.60

In an attempt to increase the capacity of the packed column the 3/4 and 1-inch Raschig rings were replaced by Berl saddles. Table 11 shows results of runs made using the saddles. Although the saddles were not quite as efficient as the smaller rings, they had a higher capacity and caused less pressure drop through the column. When fouled with fines the column was boiled out with a 5 per cent caustic solution, and occasionally the packing was removed and washed with a stream of water. The Berl saddles were more easily cleaned than the Raschig rings.

Table 11

Column Packed with 1-Inch Berl Saddles

Tri. in feed (%)	Feed rate (lb. oil/hr.)	Stripping steam (lb./hr.)	Lb. oil per lb. steam	Length of run (hr.)	Steam pressure (lb./sq.in.)
5	120	66.5	1.80	3 $\frac{1}{8}$	40
10	120	63	1.90	3 $\frac{1}{6}$	40
15	111	63.5	1.75	3 $\frac{1}{6}$	35
20	116	63.5	1.83	3	40
20	120	62	1.93	2	55
20	100	66	1.52	4	35
30	97	63.5	1.53	2 $\frac{1}{8}$	40
18*	120	62	1.93	1 $\frac{1}{8}$	40

*Run in combination with extraction plant

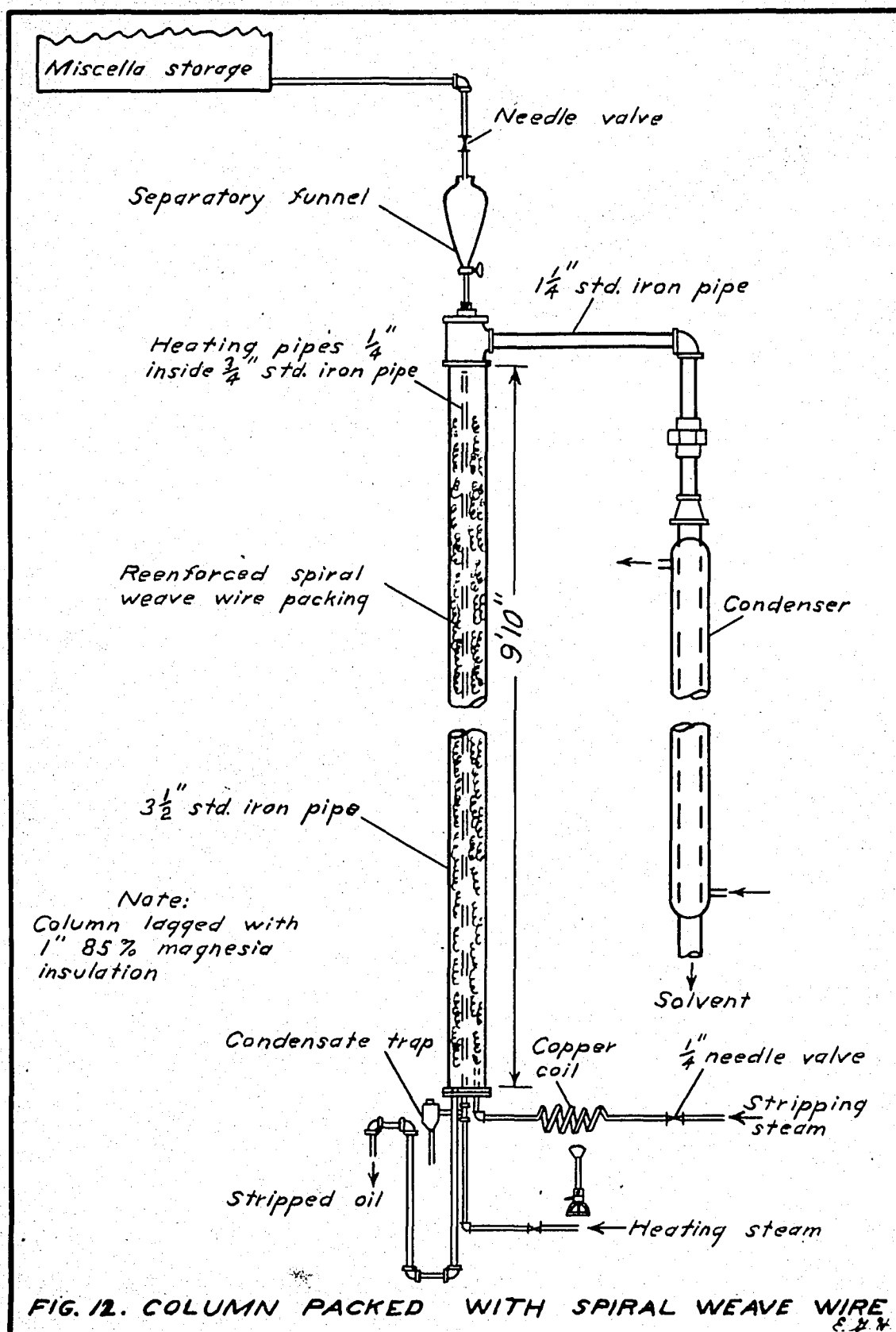
Spiral weave wire packing. Among the numerous types of materials investigated for possible use as tower packing are metal cloth and spiral weave wire.

Vilbrandt (112) investigated hose knitted copper

ribbons (34 gage copper) in a stoneware tower. He compared 1-inch Raschig rings with the wire cloth used alone and with a bubble-cap column using water and air for flow studies. He found that the knit packing alone gave slightly higher Murphree plate efficiencies than the bubble-cap alone and lower than the combination. Skow (101) tested spiral weave metallic cloth as a packing agent in rectification and found that the material was as good as any yet tested.

Spiral weave wire as a packing for soybean oil stripping columns was studied in a column constructed by Pavlik (87). The column consisted of a $3\frac{1}{2}$ -inch standard iron pipe 10 feet long containing a $3/4$ -inch heating pipe, which reached to within 8 inches of the top of the column. Vapor left the column through a $1\frac{1}{4}$ -inch pipe connected to the side outlet of a tee at the top of the column. Miscella was fed into the column by gravity. Stripping steam entered through a small pipe projecting 3 inches above the bottom plate. Oil left the column through a U-bend which served as a trap to keep steam from blowing out. The column was lagged with 85 per cent magnesia insulation. It is shown in Fig. 12.

One section of eight mesh reinforced spiral weave wire 9 feet wide was wound around the heating pipe to form a coil 9 feet long and the entire unit placed in the



3 $\frac{1}{8}$ -inch pipe.

Feed consisting of a solution containing 20 per cent trichloroethylene and 80 per cent soybean oil was fed into the column. Table 12 shows the performance of the column.

Heating steam at 80 pounds per square inch gauge was used in all the runs. The greatest trouble experienced with the apparatus was in preventing oil from being carried out the vapor line by the stripping steam. The oil obtained

Table 12

Column Packed with Spiral Weave Wire

Tri. in feed (%)	Feed rate (lb. oil per hr.)	Stripping steam (lb./hr.)	Lb. oil per lb. steam	Width of wire (ft.)	Feed Temp. ° C.
20	32	8	4.00	6 $\frac{1}{8}$	60
20	30	5.5	5.45	6 $\frac{1}{8}$	room
20	40	6.6	6.06	6	room
20	45	9.2	4.90	6	room
20	50	10.8	4.63	6	room

was darkened considerably by overheating. However, the spiral weave wire gave better efficiencies than either the mixture of 3/4 and 1-inch Raschig rings or 1 inch Berl saddles.

Device for breaking emulsions

Development of apparatus. Crude soybean oil contains phosphatides which are excellent emulsifying and foam

producing agents. These phosphatides are soluble in dry oil but insoluble in wet oil. They absorb moisture, swell up, and settle out as flocs. When oil containing these phosphatides is heated to 100° C. or higher in the presence of even a small amount of water, a large amount of very stable foam is produced as the water boils out.

Foaming in stripping operations may be reduced by several processes. One method is to use a gas such as air for stripping. However, this method is not practicable because of the difficulty of removing solvent vapor from the air. Another method is to use steam as the stripping medium and never heat the oil above 100° C. In this case the oil has to be separated from water and phosphatides. Usually the best method is to use dry stripping steam and keep all parts of the column above 100° C.

In the operation of stripping columns at Iowa State College it was found to be quite difficult to prevent all foaming and thus prevent any entrainment of oil by the steam. The presence of even a small amount of colloiddally dispersed oil in steam and solvent vapor caused troublesome emulsions to form on condensation. These emulsions contained solvent and were so stable and heavy that at times they even clogged the condenser. If they passed through the condenser, they could not be broken by letting

them stand for any reasonable length of time so they passed out the overflow carrying solvent from the solvent-water separator.

In experiments with emulsions it was found that they could be formed in test tubes by shaking a mixture of water and trichloroethylene or hexane with extracted soybean oil or with finely divided solids such as powdered charcoal, Fuller's earth, phenolphthalein, magnesium, manganese dioxide, or soybean meal. With soybean meal a mixture of $1/3$ water and $2/3$ trichloroethylene gave an emulsion in the water layer. A mixture of $2/3$ water and $1/3$ trichloroethylene gave a very heavy emulsion in the solvent layer.

The emulsions obtained from the stripping columns in this work were in the water layer and contained 2 to 10 per cent solvent. The most effective methods of breaking the emulsions were to add calcium chloride and agitate or to heat the emulsions until the solvent boiled off.

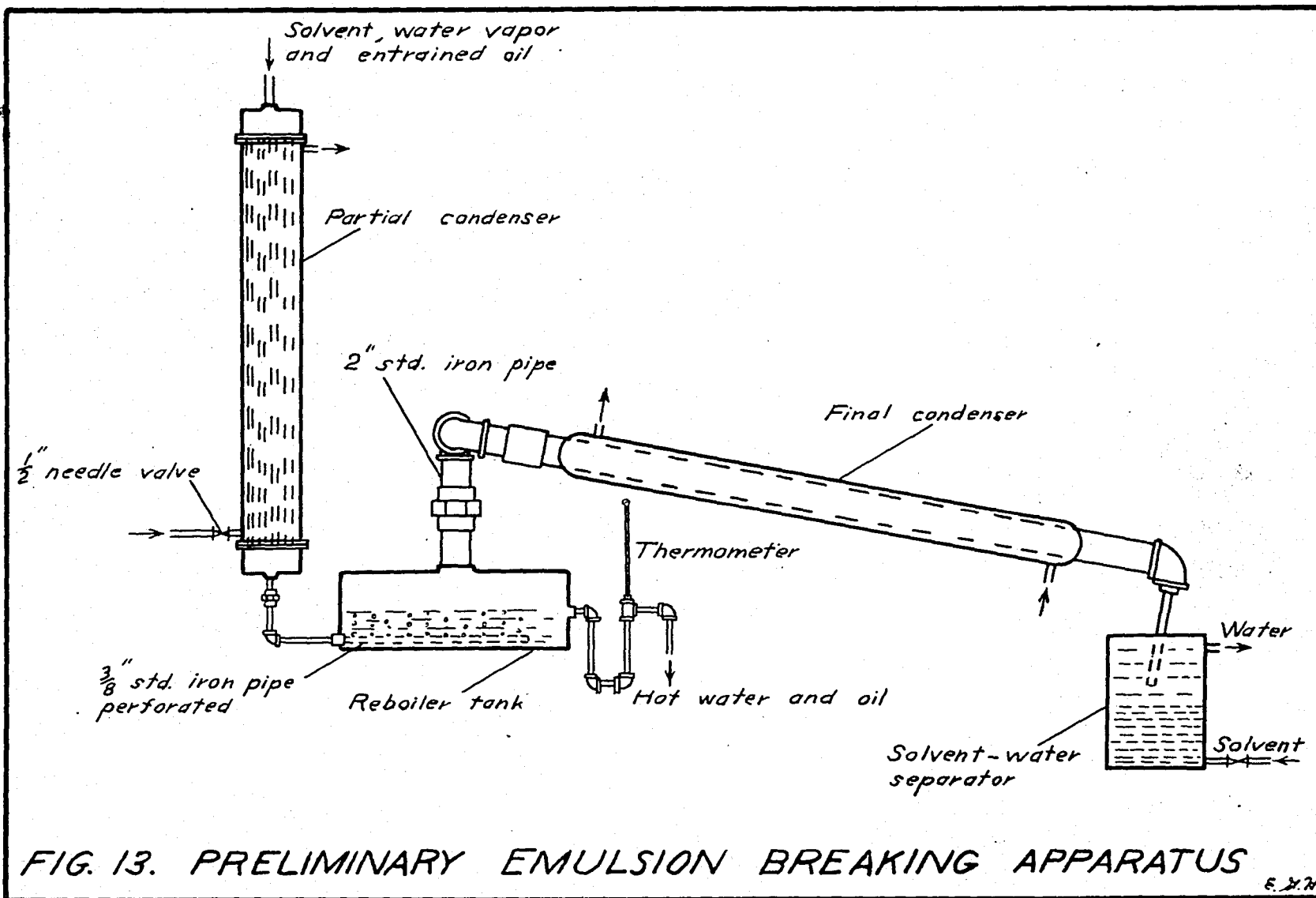
It was reasoned that if the heavy emulsions could be broken by heating the mixtures until the solvent boiled off, they could be eliminated by the reverse process; that is by condensing the bulk of the water before condensing the solvent and remaining water. Coming from the stripping column there were about four

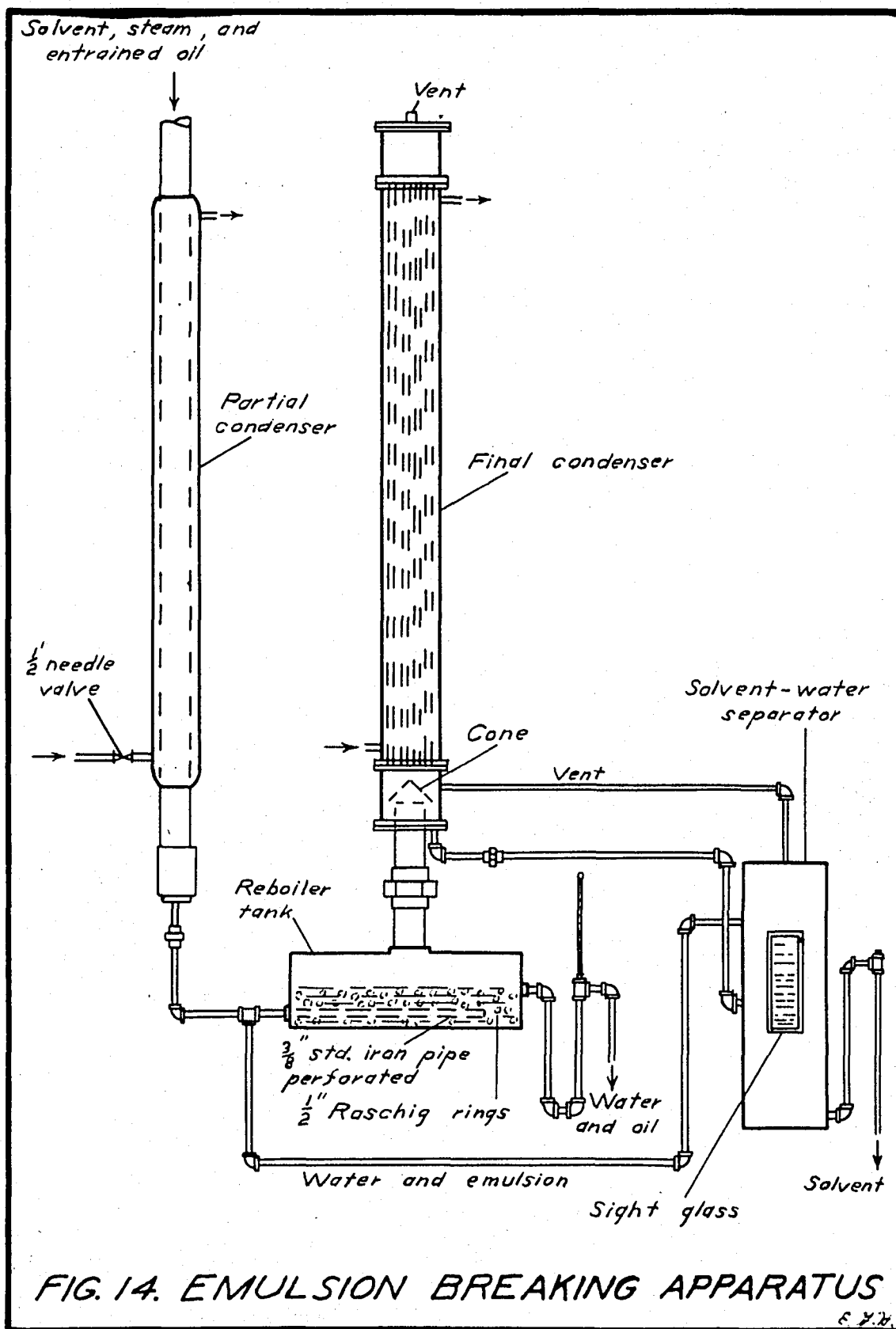
pounds of steam per pound of solvent. Since trichloroethylene and water steam distill in the ratio of 13.4 parts of trichloroethylene to one part of water, the bulk of the steam could be condensed easily without condensing any of the solvent.

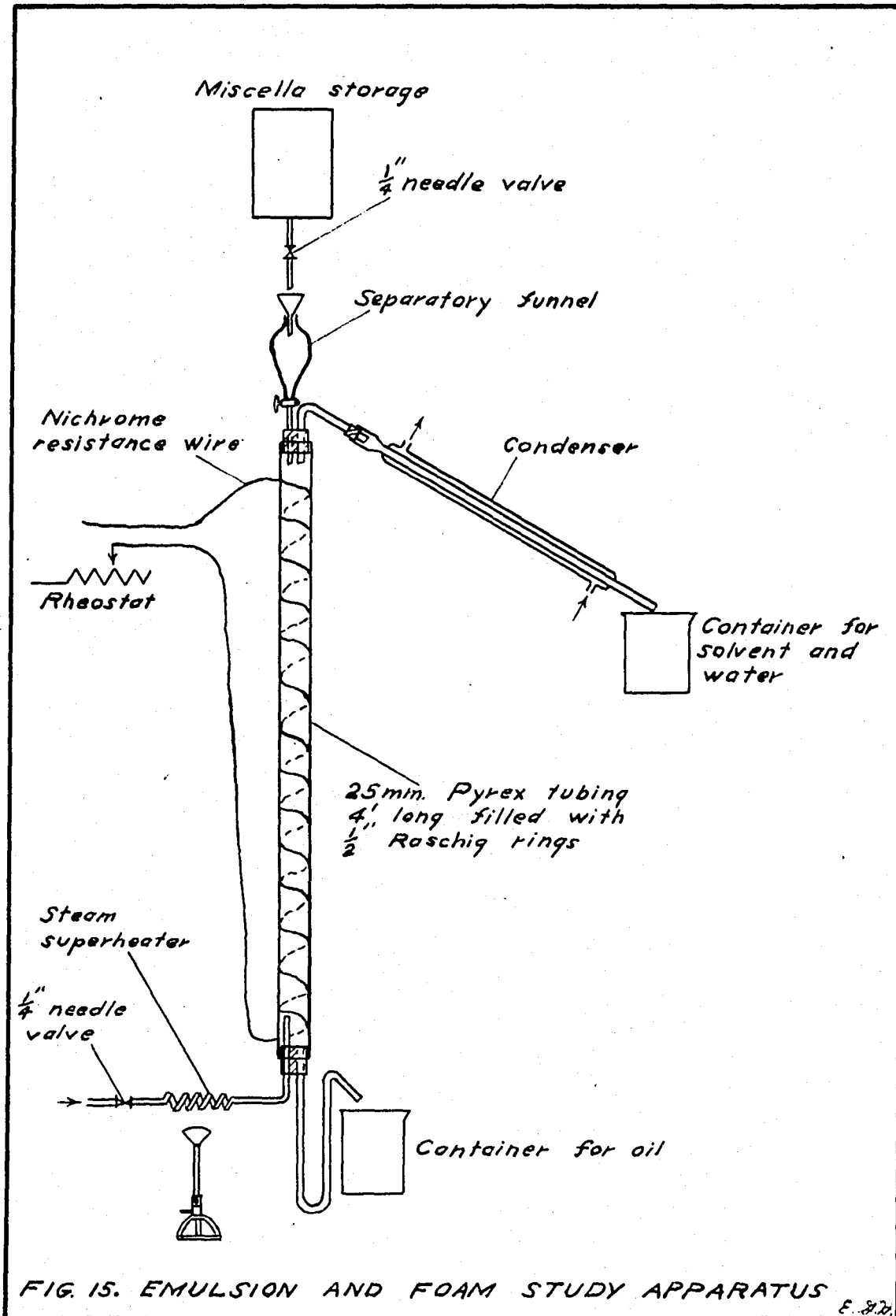
This procedure was tried using the apparatus shown in Fig. 13. From $3/4$ to $5/6$ of the water was condensed in the partial condenser. It was found that although the amount of emulsion produced was greatly reduced some was still present in that part of the water condensed in the final condenser.

Apparatus similar to that shown in Fig. 14 was constructed so that the emulsion formed in the final condenser could be run back into the tank where heat from the condensing steam revaporized all the solvent in the emulsion. The final condenser was placed vertically to eliminate any possibility of having the tubes clogged by emulsion. The hot water ($85-95^{\circ}$ C.) leaving the reboiler tank contained 0.1 per cent oil and even less trichloroethylene than is soluble in water at room temperature. Clean solvent passed out the bottom of the separator tank.

This type of deemulsifier was added to the commercial plant and found to work very well. The system put very little back pressure on the packed column and was found







to be very flexible and easily controlled.

Further studies on emulsion formation. In order to further study foam and emulsion formation the apparatus shown in Fig. 15 was constructed. The glass tubing was 1 and 13/16 inches in diameter and 4 feet long packed to within 4 inches of the top with 1/4-inch carbon Raschig rings. Both wet and dry steam were used for stripping. The only noticeable difference in their behavior was that wet steam stripping gave an oil containing a considerable amount of water.

When enough oil was run into the column to wet the rings and steam passed through it, a cloudy white emulsion was formed in the condenser beginning at column temperatures of 135° C. As the column temperature was raised the emulsion became thicker until at 170° C. the condenser was completely clogged by a thick white gel. This gel would break on prolonged standing or on heating or stirring to give a large amount of water with a tiny bit of oil-like material floating on top.

When oil was continuously run into the column, little emulsion was formed unless the oil was preheated to almost 170° C. Even then the amount of emulsion was less than when no oil was being added. The only effect of adding trichloroethylene to the oil was to make the gel somewhat thinner when appreciable amounts of tri-

chloroethylene were added.

Expeller oil from which most of the lecithin had been removed gave only a slight amount of emulsion even at 170° C. When commercial lecithin was added to the expeller oil, the emulsion was about the same as that obtained with freshly extracted oil.

From these studies it is evident that in addition to emulsion formed by entrainment of oil in steam discussed in the preceeding section emulsions may also be formed by steam distillation of a substance from soybean oil at temperatures above 135° C. The emulsion is formed in the condenser used for condensing the stripping steam and solvent. It seems likely that this substance is obtained from the phosphatide portion of the oil.

Stripping column behavior in commercial plant

The stripping column used in the commercial plant consisted of two range boilers with the ends cut off 14 inches in diameter and 5 feet long welded together lengthwise and filled with 1-inch Berl saddles. Heat was supplied by eight vertical 1-inch pipes placed on a 10 inch diameter circle inside the column. Inside the 1-inch pipes were 3/8-inch pipes through which the steam entered. The 1-inch pipes were connected to a condensate

header and the 3/8-inch pipes to a steam header at the bottom of the column. The column was lagged with 2 inches of 85 per cent magnesia insulation. After a few runs the height of the columns was increased to 14 feet, packed to 13 feet.

Stripping steam entered through slots cut in the underside of a cross at the bottom of the column. Steam and solvent vapor left the column through a 5-inch vapor line at the top of the column. Stripping steam at atmospheric pressure was dried by passing it through a pipe jacketed with high pressure steam.

Concentrated miscella containing 80 per cent oil was stripped completely of solvent in the column. Miscella at first was run directly into the column from the filters by gravity. This miscella had a temperature of about 110° C. Later the height of the column was extended to 14 feet and at the same time the filters were lowered. Miscella then ran from the filters into a 50 gallon tank from which it was pumped into the column. This miscella had cooled to about 90° C. before it reached the top of the column. When an attempt was made to run without reheating it to above 100° C., the entire top of the packing was filled with lecithin. Using reheated miscella no further trouble with phosphatides being deposited in the

column was experienced.

Heating steam was supplied to the 1-inch pipes at 30 to 40 pounds per square inch gauge. Oil left the column at 115 to 135° C. For completely removing all solvent from 150 to 180 pounds of oil per hour about one pound of steam per pound of oil was required.

Three types of distributors were tried. At first the miscella overflowed from a pan 8 inches in diameter. The pan was removed when it was suspected that it was not overflowing uniformly and was causing added entrainment of the oil. A five point distributor was next used but was abandoned because the holes sometimes became plugged. A center point feed was finally used because it was very reliable and consistent in its behavior even though it was realized that such a feed was not the most efficient.

Steam requirements in stripping operations

The amount of steam required to strip trichloroethylene from soybean oil can be calculated by assuming a vaporization efficiency. Typical feed to a column would consist of a solution containing 80 per cent oil at 110° C. The vapor pressure of trichloroethylene above this solution may be calculated by Henry's law (31)

$$p = 74X\left(\frac{t}{100}\right)^{3.4},$$

where

p is the partial pressure of trichloroethylene
in millimeters of mercury

t is the temperature in ° F.

X is the mol fraction of trichloroethylene
in the liquid.

Using this law the partial pressure of trichloroethylene would be 458 millimeters of mercury. Measamer (73) found experimentally that the vapor pressure of trichloroethylene above such a solution was 422 millimeters of mercury.

Using the value of 422 millimeters of mercury and a vaporization efficiency of 50 per cent, the pounds of steam per pound of trichloroethylene leaving the column would be

$$\frac{(549)(18)}{(211)(131)} = 0.357$$

Since four pounds of oil enter the column for every pound of trichloroethylene, the pounds of steam per pound of oil would be $0.357 \div 4 = 0.09$.

This amount of steam is far below the one pound of steam per pound of oil used in the commercial plant, the 0.5 to 0.6 pounds of steam used in the pilot plant column

packed with Berl saddles and the 0.125 to 0.16 pounds of steam per pound of oil obtained from the column packed with spiral weave wire. To strip the 11 pounds of oil obtained from a bushel of soybeans in the commercial plant costs only 0.5 cents. Nevertheless, savings should be made whenever possible without sacrificing good operating behavior.

Recommendations

a. The disk stripper could be changed in several ways to make it more efficient. Dividing the lower section into compartments would prevent short circuiting of the liquid. Using internally heated disks or using squeegees to remove the oil from the disks should increase the efficiency. Increased surface could be obtained by putting the disks closer together or making the stripper longer.

b. Columns capable of handling fines such as those containing porous plates should be further examined.

c. A smaller size of saddles, a different type of packing, or a taller column might increase stripping efficiency.

d. The best method of distributing feed into the column should be sought.

e. A steam jacket for heating should give better temperature control, give less local overheating of the

oil, and cause less channeling than the vertical steam pipes.

f. The possibility of running the column under a vacuum should be considered.

g. By properly controlling operating conditions of the stripping column it should be possible to obtain a better quality of oil. It also might be possible to prevent the formation of emulsions.

Drying and Toasting

Preliminary data

Drying rate studies were made using the apparatus shown in Fig. 16. A 250 ml. three-necked flask was fitted with a stirrer made of six pieces of piano wire projecting through a $\frac{1}{4}$ -inch shaft.

Flakes were prepared for drying tests by being extracted to an oil content of 1 per cent with trichloroethylene. The moisture content of the flakes was varied by allowing them to stand over water in a closed container or heating them in an oven at 50° C.

Flakes and solvent were added to the flask, the stirrer was started, and the oil bath raised until the flask was immersed. Immediately after immersion the temperature of the oil bath fell from 170° C. to 150-155° C.

The bath was heated at such a rate that the temperature rose 1° C. a minute until 165° C. was reached. Then the bath was held at that temperature. The oil bath was not stirred.

Solvent and water vapor leaving the flask were condensed and collected in a graduated cylinder. The amount of water held up in droplets on the walls of the condenser was estimated at different stages of the run and determined at the end of the run by being taken up in absorbent cotton and weighed.

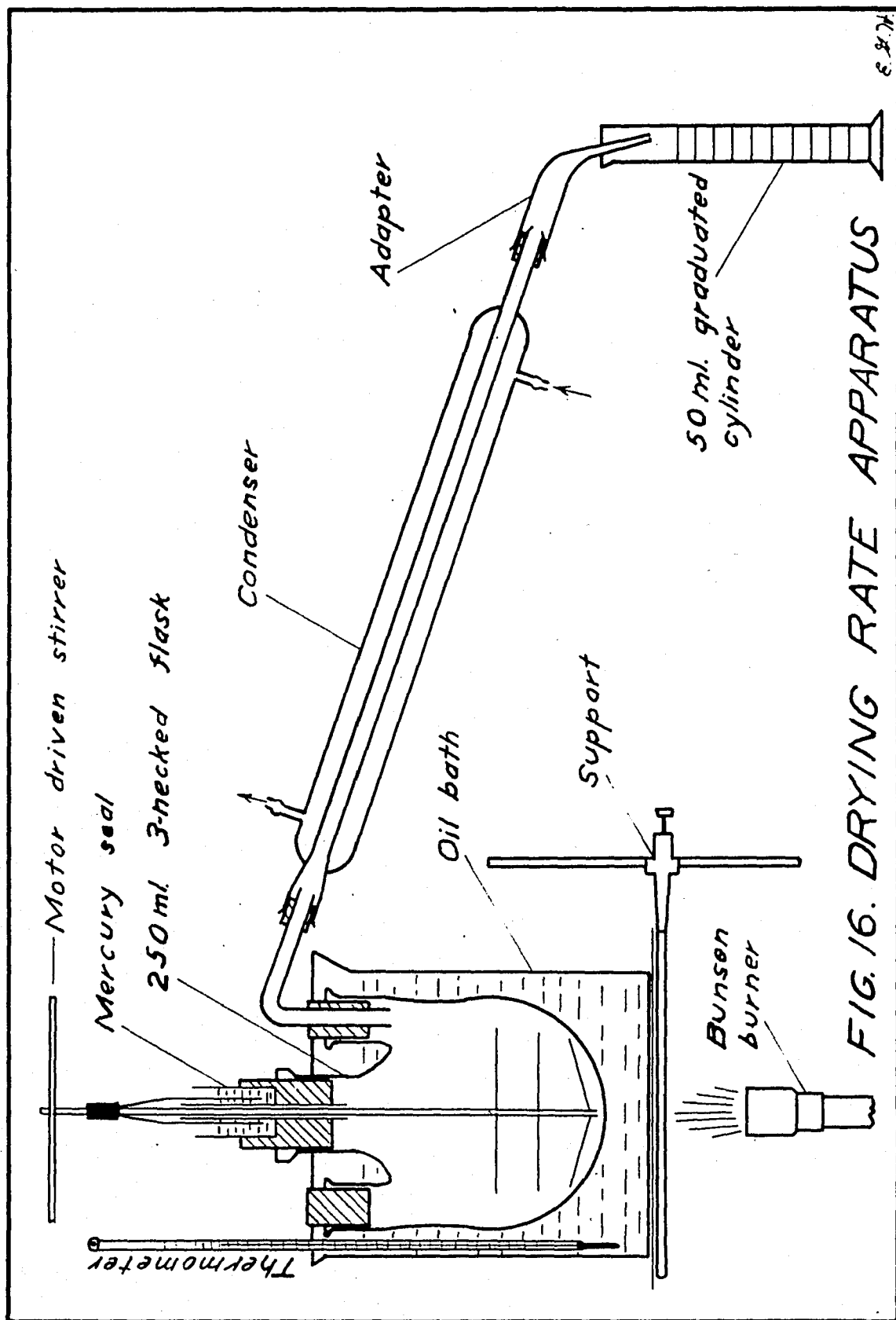
In Fig. 17 is shown the variation of meal temperature and solvent distilled with the length of time after heating started. The flask contained 51 grams (35 ml.) of solvent and 45 grams of 8.90 per cent moisture meal. The meal was not stirred. The slight hump in the curve just after the meal reached 80° C. was obtained in many runs. The meal may have been slightly superheated at the start or its thermal conductivity may have decreased after evaporation started. Most of the solvent was evaporated before the meal reached the boiling point of trichloroethylene, 87° C., and practically all of it evaporated before the meal reached 100° C.

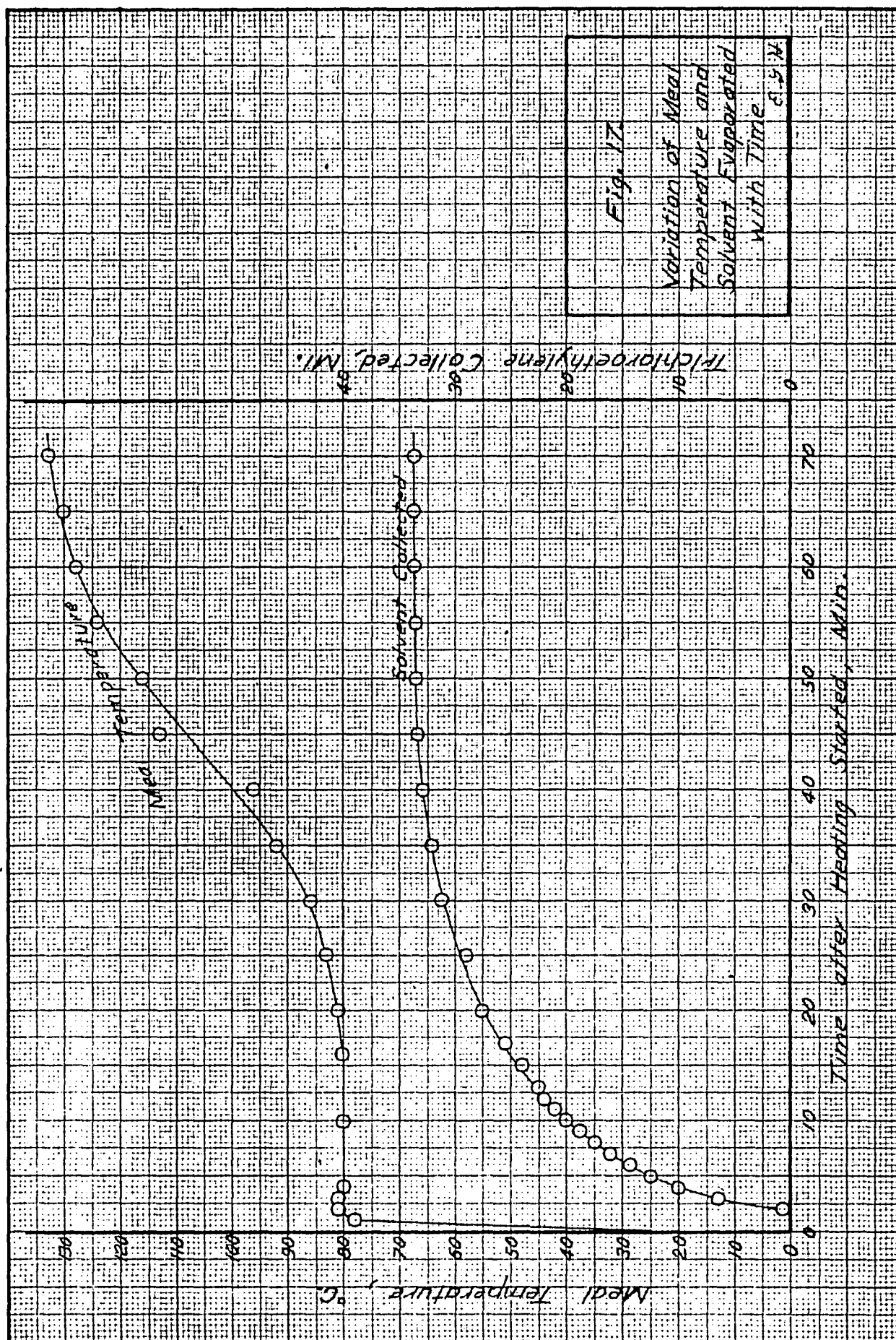
In the next experiment 50 grams of extracted soybean flakes containing different percentages of moisture were

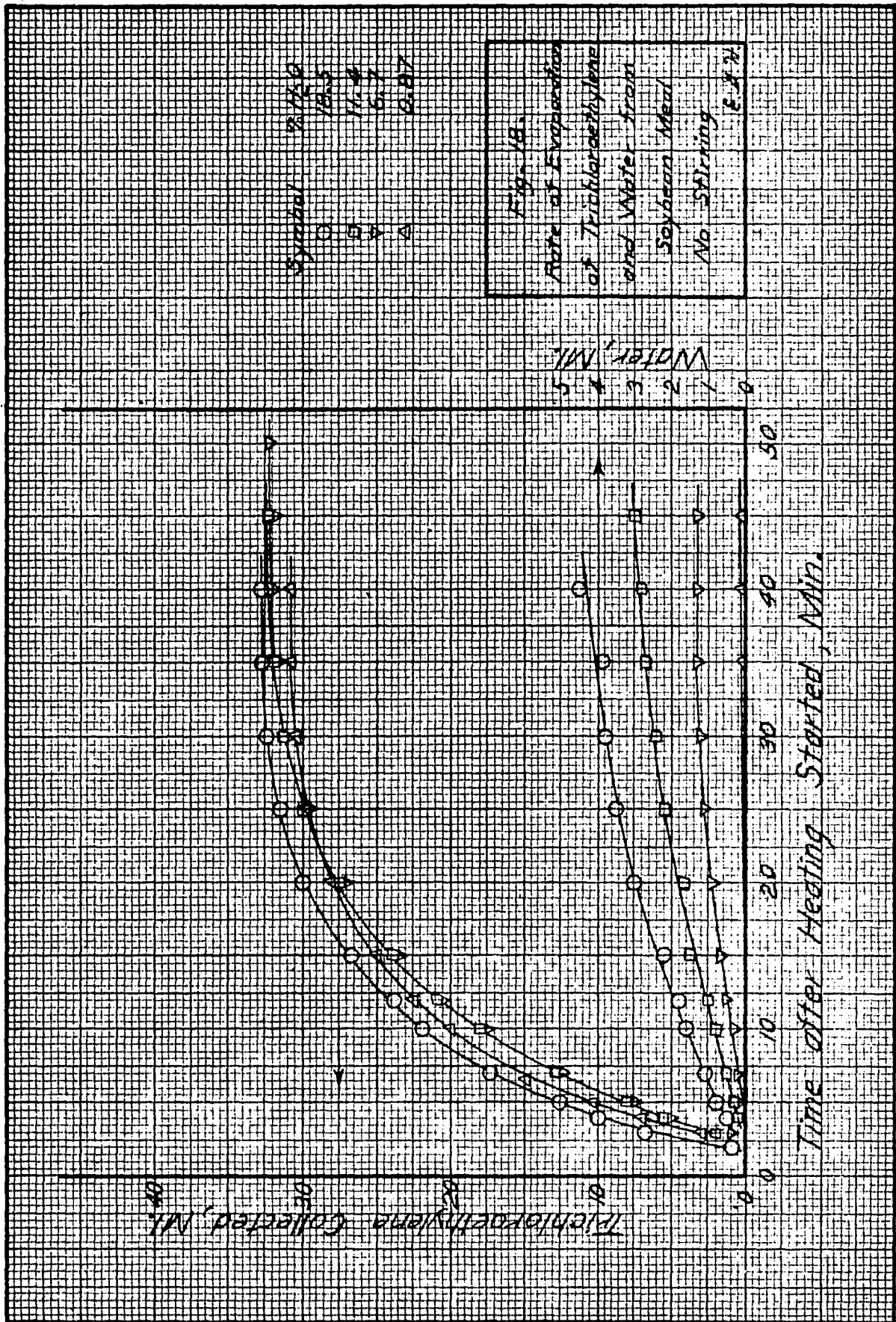
heated with 50 grams (34.2 ml.) of trichloroethylene without being stirred. The rate of evolution of trichloroethylene and water are shown in Fig. 18. Solvent was still being given off 35 minutes after heat was applied. Flakes containing 18.5 per cent moisture gave up trichloroethylene somewhat more rapidly than flakes with lower moisture contents. Such behavior might be explained by the fact that 50 grams of high moisture content flakes contained less solid matter through which the solvent had to diffuse than drier flakes. The thermal conductivity of the wetter flakes also should be higher. On the other hand one would expect that the greater amount of moisture being evaporated from the wetter flakes would be evaporated at the expense of the trichloroethylene.

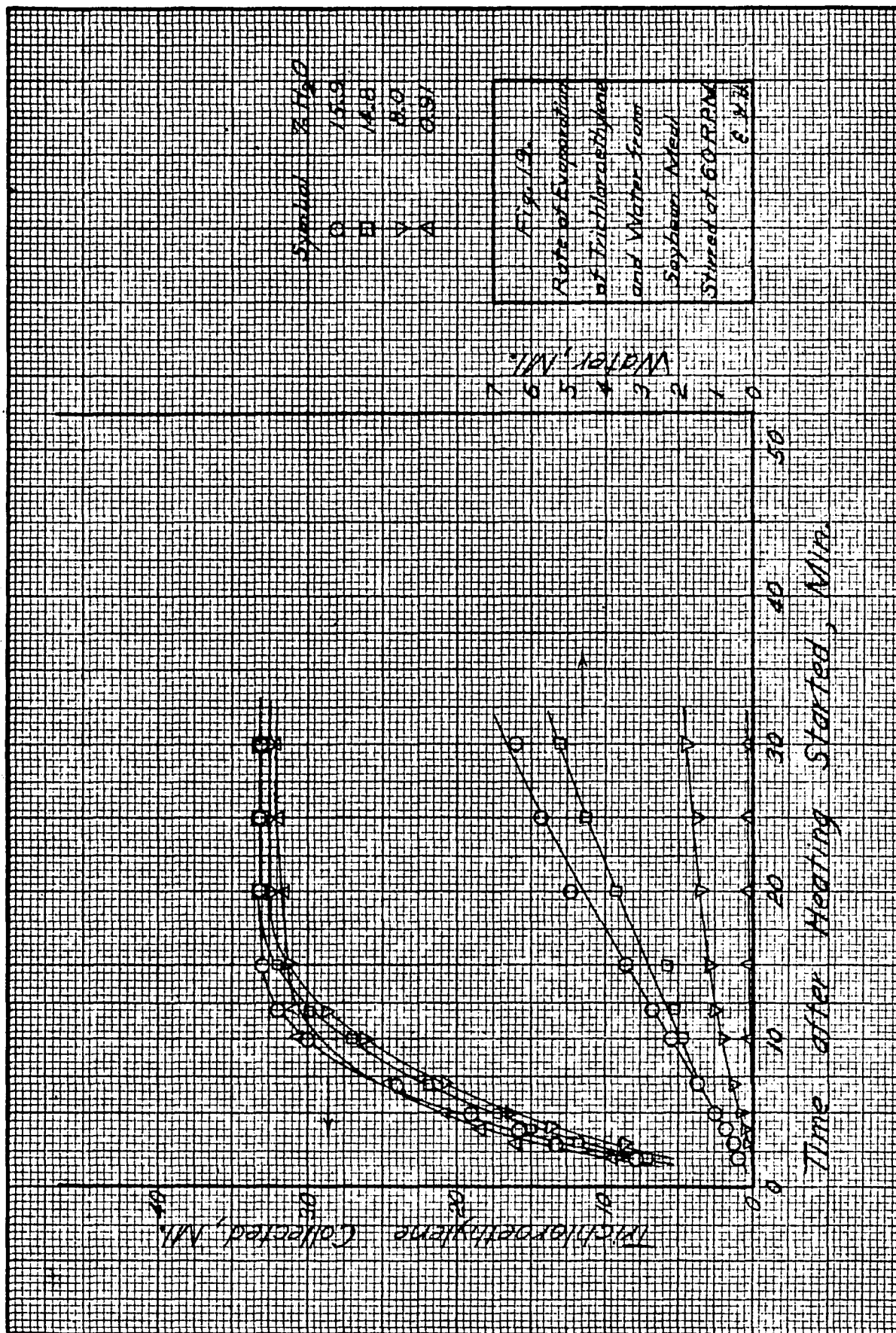
The above experiments were repeated with the stirrer revolving at 60 revolutions per minute. Flakes containing 0.91 to 15.9 per cent moisture were dried. There were no very marked differences in the rate of drying. Both Fig. 18 and Fig. 19 show that moisture is evolved at a rather rapid rate long after all solvent has evaporated.

Fifty grams (34.2 ml.) of soybean flakes were again heated with 50 grams of trichloroethylene with the stirrer operated at different rates of speed. The flakes had approximately the same moisture content in each case.









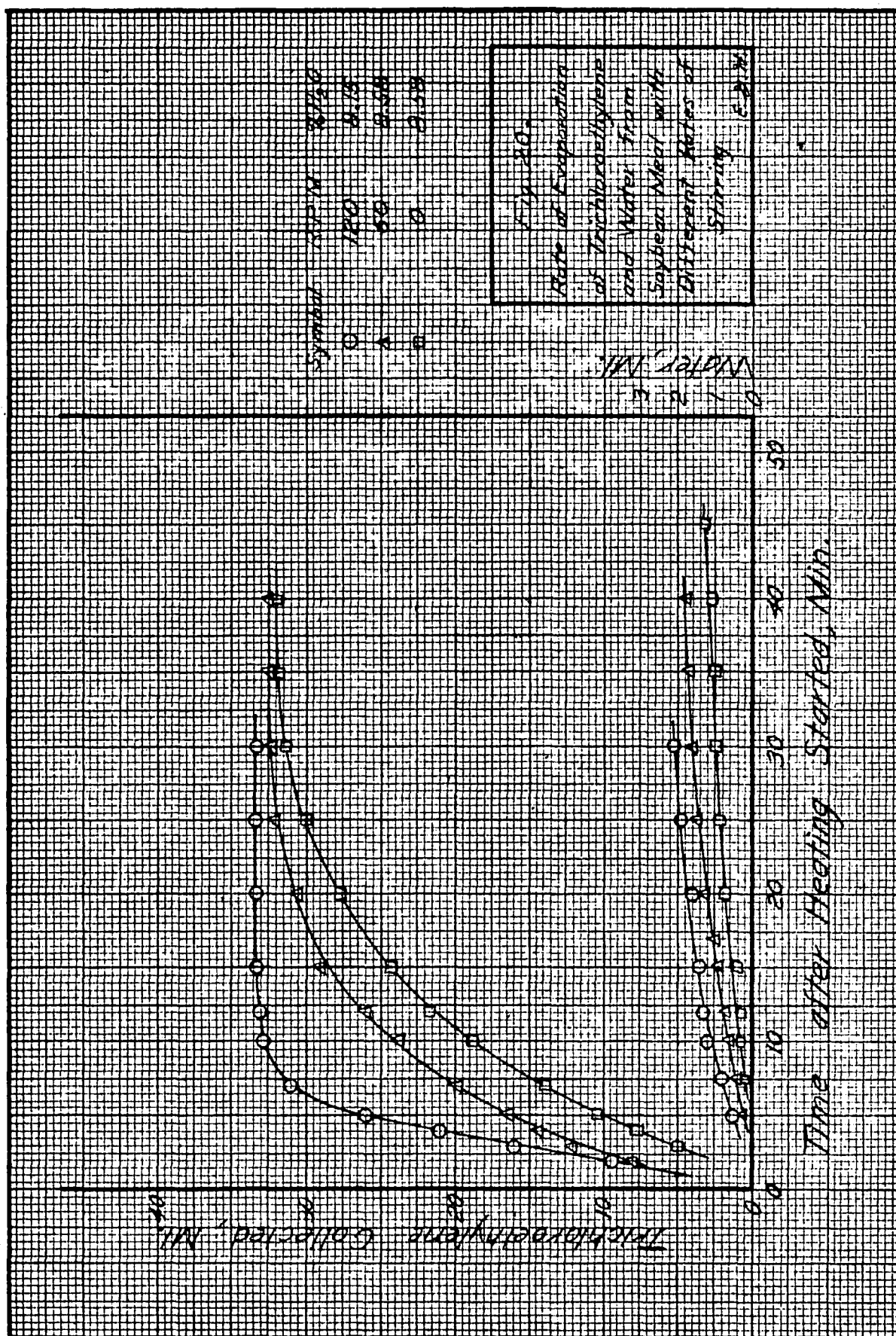


Fig. 20 shows the marked differences in the rates of removal of both trichloroethylene and water produced by stirring at different rates. The flakes which were stirred rapidly had a darker and more uniform color.

It was observed that the amount of browning, or toasting, of the meal varied directly with the original moisture content of the meal. Meal heated to the same temperature for the same length of time in both a flask and a moisture oven showed that the higher the original moisture content of the beans the darker the color of the heated meal.

A few experiments were made using infra-red radiation for toasting soybean meal. A 250-watt lamp placed 6 inches above the meal gave good toasting in five to ten minutes. The infra-red radiation had a tendency to burn the dark colored particles and leave the lighter colored particles untoasted. By agitating the meal properly while toasting and grinding it after toasting the amount of uneven browning might be reduced. Infra-red toasting would, of course, be more expensive than toasting with steam heat.

Pilot plant drying

The meal leaving the extraction section of the pilot plant passed through a drainage section about 4 feet in length and entered a drying section. The drying section

was 11 feet in length and $6\frac{1}{2}$ by $6\frac{1}{2}$ inches in cross section. Meal was carried through this part of the system by the Redler conveyor and was heated by steam at 60 pounds per square inch.

Each 100 pounds of dry, extracted meal entering the Redler drier carried with it 13.7 pounds of water and a maximum of 148 pounds of trichloroethylene. Leaving the drier with each 100 pounds of dry, extracted meal were 13 pounds of water and 87 pounds of trichloroethylene. Assuming that the material entered the drier at 25° C. and left at 80° C., the overall heat transfer coefficient, based on the total inside heating area, was 2.3 B.t.u./hr./sq.ft./ $^{\circ}$ F. when processing 125 pounds of beans per hour.

Partially dried meal from the Redler drier fell into a lower drier 6 inches in diameter jacketed to a length of 15 feet 8 inches with a steam jacket containing steam at 80 pounds per square inch gauge. The conveying element in the tubular drier was a ribbon conveyor. The ribbon was formed by a $\frac{3}{8}$ -inch rod bent into the form of a spiral with 6 inch pitch. Leaving the tubular drier were 5.3 pounds of water per 100 pounds of dry, extracted meal. The temperature of the meal leaving was about 110° C. The overall heat transfer coefficient of the tubular drier based on the total inside heating area

when processing 125 pounds per hour of soybean flakes was 6.3 B.t.u./hr./sq.ft./° F. Vapor velocity in the Redler drier was 0.40 feet per second and in the tubular drier 0.90 feet per second.

Vapor in both driers travelled countercurrently to the meal and was removed through 3-inch vapor lines. The driers were inclined slightly to take advantage of the difference in weight of trichloroethylene and water vapor. Water vapor, being lighter, would tend to rise toward the higher end and leaving solvent at the lower end. Therefore, the vapor outlet was placed at the lower end and the meal outlet at the higher end of the drier.

When processing beans at a rate of 125 pounds per hour, the drying capacity was barely sufficient to remove all the solvent from the meal. Steam blown into the outlet end of the tubular drier aided in removing solvent but caused some meal to stick to the barrel valve.

Commercial plant drying

Drying and toasting were performed in the commercial plant in one section of Redler drier and two sections of tubular drier, one of which served as a toaster. As in the pilot plant, the Redler drier was the upper part of the continuous loop travelled by the Redler chain. It had a cross-section 1 foot square and was 25 feet long

surrounded by a jacket containing steam at 90-100 pounds per square inch. The tubular drier and the toaster had an inside diameter of 1 foot and were also surrounded by a jacket containing steam at 90-100 pounds per square inch gauge. The conveying elements in the tubular drier and toaster were ribbon conveyors. The ribbons were made of steel 2 inches in width bent into the form of a spiral with 1 foot pitch. The Redler drier was jacketed to a length of 24 feet, the tubular drier to a length of 42 feet, and the toaster to 37 feet.

Vapor was removed as shown in Fig. 4. Vapor in the Redler drier travelled countercurrently to the meal and left through a 6-inch vapor line. Vapor was also removed at a point between the tubular drier and the toaster so that vapor in the drier moved concurrently with the meal and in the toaster countercurrently. The driers and the toaster were tilted in such a way that the lower end was the end toward which the vapor was flowing. Leaving the tubular drier and toaster the vapor passed through a spray chamber, where hot spray water removed meal dust, and through an 8-inch vapor line to the condenser.

Meal entering the Redler drier contained 1.1 to 1.3 pounds of trichloroethylene per pound of wet, extracted meal. Meal entering the tubular drier contained solvent and filled the drier to a higher level than dried meal;

on the average the drier and toaster were almost half full of meal when processing 15 tons of beans a day. The meal remained in the Redler drier for about 20 minutes, in the tubular drier for 18 minutes, and in the toaster 15 minutes.

When 15 tons of 13 per cent moisture beans a day were processed, meal containing 6 per cent moisture was obtained. At this rate about 10 per cent of the solvent and 5 per cent of the water evaporated were removed in the Redler drier. Eighty per cent of the solvent and 40 per cent of the water were evaporated in the tubular drier. About 10 per cent of the solvent and 55 per cent of the water were removed in the toaster. Heat transfer coefficients on such equipment vary considerably depending upon the water and solvent content of the meal and the amount of stirring. The Redler drier had an overall heat transfer coefficient based upon the total inside heating area of 3.5 B.t.u./hr./sq.ft./° F. The tubular drier had a coefficient of between 7 and 10 averaging about 8 B.t.u./hr./sq.ft./° F. The toaster had a coefficient of between 5 and 8, averaging about 6 B.t.u./hr./sq.ft./° F.

Assuming that 1.1 pounds of solvent per pound of meal were leaving the extractor and that all the solvent and half the water vapor was passing through the tubular drier, the maximum vapor velocity was 9 feet per second. Assuming that a maximum of 20 per cent of the solvent and

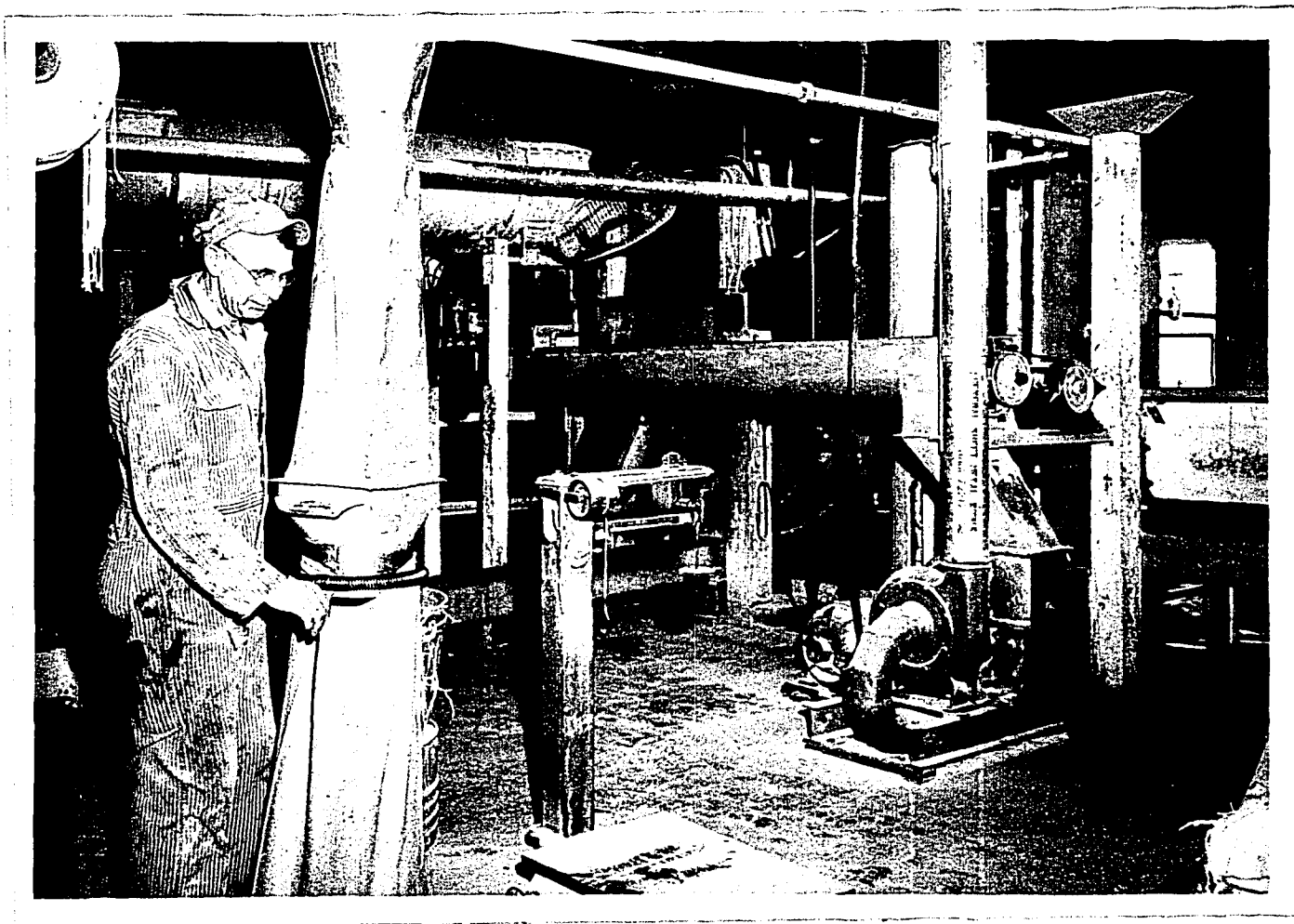
60 per cent of the water were being removed in the toaster, the vapor velocity in that unit was not over 4 feet a second when processing 15 tons of soybeans a day. The outlet end of the toaster, the meal moistener, and the meal grinder are shown in Photograph 6. Part of the extraction chamber is also shown.

Conveyor study apparatus

As shown in a previous section, stirring soybean meal while it is being heated greatly increases the rate of removal of both solvent and water. Agitation should not be violent enough to cause excessive dusting and should not be performed by devices which excessively restrict the flow of meal or vapor at any point.

A simple type of apparatus was used for studying the action of agitating conveyors. It consisted of a trough 1 foot wide and 6 feet long lined with sheet metal. The trough had a rounded bottom, high sides, and an open top. Running the length of the trough was a 2-inch shaft fitted with a crank at one end and with supports for ribbon conveyors.

The conveying elements were made of 26 gauge sheet metal. Such light construction made possible the testing of an almost endless variety of conveyors and of conveyors fitted with agitating devices. The amount of agitation



Photograph 6

Outlet End of Toaster, Meal Moistener, Meal
Grinder, and Section of Extraction Chamber

was estimated by adding dark colored material to the meal at one end and observing the rate at which it was mixed with the meal.

Closed-flight conveyors and ribbon-flight conveyors with a width of over 1 inch gave very little agitation. The agitator used in early work in the commercial plant consisted of steel bars placed horizontally between the flights of ribbon at intervals. Examination of this type of apparatus showed that although the mixing bars gave excellent agitation they restricted the movement of large lumps of meal causing the driers at times to become plugged. A mixing and conveying device similar to a cut-and-folded flight looked promising in the conveyor study apparatus. When this type of mixing conveyor was installed in the commercial plant, it gave excellent results. Other types of conveyors have given satisfactory results in the experimental apparatus but they have not yet been thoroughly investigated.

Recommendations

a. Operating behavior of the driers should be carefully examined in order to obtain a better design than that used at present.

b. Some means of providing agitation in the Redler drier would greatly increase the heat transfer in that section.

c. A better type of stirring conveyor than that now in use might be found.

d. The effect of blowing steam into the toasting section of the drier should be studied further.

e. After all the trichloroethylene has been removed from the meal, water vapor is still given off. It might be possible to restrict the movement of this water vapor in such a way that it would aid in toasting the meal.

f. Adding a brown dye to the water sprayed upon the meal before it is ground might be one way of satisfying the demand for a dark colored meal. A better way of solving this problem would be to educate the consumers to the fact that light colored extracted meal is as good as dark expeller meal.

Condensation

Operating behavior

The vaporized solvent present in an extraction plant has to be condensed and reused for further extraction. Complete recovery of the solvent is especially desirable in a plant using relatively expensive trichloroethylene. The Plainfield plant operating at 15 tons of beans a day had a solvent loss of 8 to 10 pounds a ton. This loss was within the limit set when the plant was designed and will

undoubtedly be reduced in the future. Barring leaks and faulty drier operation the main losses of solvent occur in some phase of the condensation operation. The solvent either leaves with condensed water, spray water, or with non-condensable gases vented from the condensers.

The condensers used in all of this work were those which were already on hand and did not necessarily represent the best design. Kircher (63) found that vapors consisting mainly of trichloroethylene gave an overall heat transfer coefficient of 120 B.t.u./hr./sq.ft./° F. when condensing inside vertical tubes with water on the outside. In checking this value trichloroethylene condensing inside a $\frac{3}{4}$ -inch copper coil immersed in water gave an overall heat transfer coefficient of 135 B.t.u./hr./sq.ft./° F. A mixture of half trichloroethylene and half steam condensing in a 1-inch horizontal copper tube with water on the outside had an overall heat transfer coefficient of 155 B.t.u./hr./sq.ft./° F. based on the inside area. In comparison the condenser used on the driers in the Plainfield plant had an overall heat transfer coefficient of 25 B.t.u./hr./sq.ft./° F. when condensing a mixture of ten parts trichloroethylene and one part water.

Tubular condensers may be operated with the vapor condensing either inside or outside the tubes. If the vapor contains material which is likely to be deposited and

foul the condensers, it is better to condense the vapor inside the tubes because the inside can be cleaned easily by removing the end plates and running rods or water through the tubes. Vapor in the commercial plant condensed inside the tubes.

In the pilot plant condensed solvent and water ran into a small solvent-water separator from which the solvent ran into a storage tank before reentering the extractor. Condensed solvent and water in the commercial plant ran into a rectangular tank capable of holding 1700 pounds of solvent, which served both as a solvent-water separator and as a supply tank. Non-condensable gases in both plants were vented from the condensers into the incoming flakes. A small fan was used in the commercial plant to aid in removing non-condensable gases, but its necessity has not yet been completely demonstrated.

Some corrosion of the iron took place in the condensers and in other parts of the system where water and trichloroethylene condensed together. Evidently, trichloroethylene, which is a good solvent for oils, removed the grease film protecting the iron allowing the moisture to wet it and increase the rate of oxidation. Those parts of system where there was no condensation were not corroded.

Recommendations

- a. Many phases of the condensation operation should

be studied including the dimensions of the condensers, recovery of heat from the vapors, the best method of venting non-condensable gases, and the best way to overcome the moderate corrosion produced in certain parts of the plant.

Extraction of Oil from Materials Other Than Soybeans

Extraction of milkweed seed oil

Preliminary data. Although the development of milkweed as a commercial crop has long been considered (47), such a development has never materialized. During World War II milkweed floss was badly needed for its insulating and buoyant properties. A nation-wide campaign was conducted in which school children collected milkweed pods. The pods were broken open and the seed and floss separated at processing centers. Large amounts of seed accumulated at these centers. The seed contained about 37.5 per cent protein, 23 per cent oil, 11.5 per cent fiber, and 4 per cent ash. Seed were sent from the Petosky, Michigan, processing plant to Iowa State College in order to determine whether they could be processed in the system developed for soybean oil extraction.

The milkweed seed as received contained 6.5 per cent

moisture. When these whole seeds were extracted for two hours with trichloroethylene, only 0.7 per cent oil was obtained because the solvent was unable to penetrate the seed coat. Finely ground seed yielded 19 per cent oil after a two hour Soxhlet extraction using trichloroethylene. After the seed had been steamed to a moisture content of 11 to 12 per cent and flaked, the yield of oil on a two hour extraction ranged from 21 to 24 per cent. Soxhlet extraction of the flaked seed produced 16.4 per cent oil in 15 minutes, 20.7 to 22 per cent in 30 minutes, and 21 to 23 per cent in 45 minutes. The extractor syphoned every three minutes. The oil contents are based on a moisture content of 10 per cent.

In order to be processed satisfactorily in the extraction system under consideration, the oleaginous material must form a bed which will permit ready passage of the solvent. Studies of the resistance to passage of the solvent were made using the apparatus previously described and shown in Fig. 5.

Solvent ran through the whole untreated seed quite rapidly, but it did not extract an appreciable quantity of oil. When whole seed with no treatment or treated by only being heated, were run between flaking rolls, the product contained a large amount of fines and did not allow rapid penetration of the solvent. Seed steamed to a moisture

content of 11 to 12 per cent made good flakes both with flaking rolls and with a roll-and-sleeve type flaker. Seed broken in a Wiley mill, a coffee-type mill, and a ball mill contained too many fines for satisfactory operation. The meat of the seed, which contains most of the oil, makes up a larger proportion of the fines than of the whole seed as shown by the data in Table 13.

Table 13

Oil Content of Milkweed Seed Fines

Mesh	Fines (% by weight)	Oil in fines (%)	Oil in portion retained (%)
16	20	28.6	18.8
40	9	32.5	22.0

The flakes used in the pilot plant averaged 0.010 inches in thickness. Whole flakes averaged 0.25 inches in width and 0.35 inches in length.

Operation of extractor. The extraction system used for extracting milkweed seed oil was modified somewhat from that shown in Fig. 3. The seed were not run through the cracker; instead they were steamed and flaked whole. The falling film evaporator described in the section on evaporation was used for concentrating the miscella leaving the extractor. Last traces of solvent were stripped in the 8 inch diameter column packed with Berl saddles

shown in Fig. 11. Between the extractor and the evaporator was the small Sparkler filter shown in Photograph 2, which was used for filtering miscella.

Since the author knows of no other work on the extraction of milkweed seed oil on a scale as large as that used at Iowa State College, the operating behavior of the extraction apparatus will be discussed in some detail. As might be expected, considerable difficulty was experienced during the first few runs. However, the system was eventually made to operate smoothly.

Run No. 1:

The milkweed seed were steamed and flaked and the flakes allowed to air dry overnight. The Redler chain carrying the flakes through the solvent travelled 11 inches a minute. Miscella ran directly from the extractor to the evaporator without passing through the filter. The run lasted five hours during which time a total of 557 pounds of flakes were extracted giving 70 pounds of oil, as overall yield of 12.6 per cent.

The tubes in the falling film evaporator were almost completely clogged with fines by the end of the run. Fine meal also fouled the packing in the stripping column. The vapor line from the tubular drier was almost completely plugged with fine meal and floss.

During most of the run miscella contained 25 to 27

per cent oil. Solvent was pumped up the drainage end of the extractor to some extent by the mass of moving material. Difficulty was experienced in removing last traces of solvent from the meal.

Run No. 2:

Milkweed seed were steamed, flaked, and allowed to air dry. The flakes were lighter than soybean flakes, a given volume weighing $3/5$ as much. Miscella, after leaving the extractor through a 40 mesh screen, was pumped through a small Sparkler filter. The filter would not handle the miscella fast enough to keep up with the plant. After the filter pressure had risen to 50 pounds per square inch, the extractor was shut down and the filter cleaned. Only $1\frac{1}{4}$ pounds of solvent-free fines were obtained. The cake thickness was about $\frac{1}{4}$ inch.

The run was continued with the extractor speed reduced to 5.6 inches a minute. Filter aid (Celite) was added to the miscella before it entered the filter. The filter worked somewhat better, but the pressure rose to 50 pounds per square inch, and miscella almost ceased running through the filter before the end of the run. Three and three-fourths pounds of fines and filter aid were obtained. Twenty-two pounds of oil were obtained from 160 pounds of seed. The low yield of oil, 13.7 per cent, was hardly surprising in view of the irregular

operation of the extractor. The run was discontinued after seven hours.

Run No. 3:

Milkweed seed were allowed to stand in large cans overnight after being steamed and flaked. The extractor was run at 11 inches a minute and the Sparkler filter was by-passed. At the beginning of the run solvent ran through the flakes satisfactorily. However, the miscella soon stopped running out the screens and solvent was pumped up the drainage end of the extractor. Apparently this trouble was caused by a dense slug of material because after a short time the solvent level fell in the drainage section and miscella again ran through the screens.

After about three hours of operation the same trouble was again encountered. This time the solvent level stayed high in the drainage section and miscella did not run through the screen. During this part of the run it was observed that the Redler conveyor, which should have been full of flakes going from the feed hopper into the extraction section, was running almost empty as it passed the screens on the miscella outlets. Under such conditions one would expect solvent to pass through the extractor very rapidly. Since the opposite was true, flakes or fines must have continuously accumulated in an

impervious cake at the lower end of the extractor. Solvent by this time had been in the extractor for so long that it had dissolved a large amount of oil causing its viscosity to rise aggravating the difficulty. By striking with a hammer one could tell that the liquid had penetrated only about half the length of the extractor. Flakes were used at a rate of 45 pounds an hour. The run was discontinued after four hours.

Run No. 4:

Flakes were from the same lot as those of Run No. 3. The filter was again by-passed. The chain speed was slowed to 5.6 inches a minute. Despite the slower speed the same trouble experienced in the previous run was encountered. Impervious slugs of solid matter could have been caused by an irregular rate of feeding, the presence of fines, or the presence of floss in the flakes. When one of the slugs was removed from the Redler drier and placed in a testing tube solvent was barely able to penetrate it. When an attempt was made to re-extract the meal the extractor pumped solvent worse than ever. An excessive amount of solvent was lost with the meal. Length of the run was three hours.

Run No. 5:

Whole milkweed seed with no treatment at all were run through the extractor. Solvent penetrated the seed

easily and at no time was there any sign of pumping or other difficulty. The seed looked almost the same leaving as they did on entering the extractor. The oil was not extracted. A miscella sample contained 2 per cent oil.

Run No. 6:

Before the seed were steamed and flaked for this run, the floss was removed by shaking on a three mesh screen. The Redler conveyor was run at 11 inches a minute. The filter between the miscella outlet and the evaporator was again by-passed. Solvent ran through the flakes satisfactorily for $2\frac{1}{2}$ hours. The rate of solvent entering was then slightly reduced. Pumping started at one end and the miscella level dropped in the other. This state of affairs continued throughout the rest of the run. The miscella did not rise to the height of the outlet even though the solvent feed was raised to a very high rate. The miscella did rise to the height of the outlet screens just before the end of the run after five gallons of solvent were poured into the system at a point about $\frac{1}{3}$ of the way down the extraction chamber. The length of the run was five hours. A sample of meal obtained during the first of the run contained 4.2 per cent oil.

Difficulty in making the flakes feed in satisfactorily seemed to be causing a large part of the trouble. At the beginning of the run the flakes were drier and fed in

faster (60 pounds per hour) and more uniformly than at the end (50 pounds per hour), when more trouble was experienced. Since a given volume of flakes is three-fifths as heavy as the same volume of soybean flakes, the milkweed flakes should enter at a rate of about 75 pounds per hour when running 11 inches a minute. It was decided to dry the flakes before making the next run.

Run No. 7:7:

The seed were steamed, flaked, and air dried on the floor for four days. The Redler conveyor travelled 11 inches a minute and the filter was not used. Solvent ran through the flakes freely with only slight pumping. The flakes were fed in at 70 to 80 pounds per hour and filled the chain most of the time. The increased rate of feed, brought about by drying the flakes, undoubtedly caused the extractor to work better. During the 3-1/3 hour run 34 pounds of oil were obtained from 240 pounds of flakes, a yield of 14.2 per cent. Meal contained 8 per cent oil.

The miscella contained about 20 per cent oil as compared to 7 per cent at the beginning of the previous run when better extraction was obtained. Poor extraction may have been caused by channeling, by running a miscella too rich in oil, or by insufficient extraction time.

Data obtained by Soxhlet extraction indicated that improper flaking did not cause such poor extraction.

The floss was screened from the seed used in this run before they were flaked. Floss left in the seed hinders extraction to a certain extent and greatly interferes with the flaking operation as performed in the pilot plant.

Run No. 8:

Milkweed seed were screened to remove floss, steamed, flaked, and spread on the floor to air dry for several days. The flakes then contained 6.9 per cent moisture. The Redler conveyor travelled 11 inches a minute and the filter was not used. During the entire length of the run the extractor operated quite satisfactorily. At only one time was there any excessive amount of pumping and that was just after the flakes bridged in the hopper causing part of the extractor to run empty. Some solvent was lost with the meal. It was calculated that each pound of milkweed meal carried almost twice as much solvent into the driers as a pound of soybean meal.

Steam pressure on both parts of the falling film evaporator was 40 pounds per square inch gauge. The concentrated miscella leaving the bottom of the evaporator contained 80 per cent oil. The evaporator was found to

be quite clean at the end of the run, even though 7 pounds of oily fines collected in the settling chamber at its base. Consequently, the fouling of the evaporator tubes encountered in Run No. 1 must have taken place at least in part before the milkweed seed miscella was run through them. The packed stripping column was somewhat fouled with fines. Seventy pounds of oil were obtained from 375 pounds of flakes, a yield of 18.65 per cent.

The rate at which solvent ran through the extractor was varied in that the miscella contained 10, 14, and 18 per cent oil. Evidently the extraction time will have to be lengthened if better extraction is to be obtained. The extraction time in this run was about 15 minutes.

The behavior of the extractor in the last two runs was most encouraging. Milkweed seed can undoubtedly be successfully extracted in the apparatus developed at Iowa State College. The problems of filtering the milkweed seed fines from the solvent-oil solution and of obtaining somewhat better extraction remain to be solved.

The milkweed seed oil had a green color probably because the pods were picked before they had opened. The oil had a free fatty acid content of 3.2 per cent and a refining loss of 14 per cent. Lovibond color readings for a one inch depth of the oil gave the following

results: crude oil, yellow 120, red 4.8; refined oil, yellow 132, red 3.5; refined and bleached, yellow 74, red 0.

The milkweed seed meal was high in protein; but had a bitter taste. A few tests on rats showed that the rats would not eat the meal unless they were almost starved. When they did eat the meal they gained weight. Cattle also did not like to eat the meal. It has been reported (81) that the seed from the common milkweed, Asclepias syrica, contain a poisonous glucoside similar in properties to a toxic glucoside found in the Asclepias galicoides. If some method of eliminating the bitter taste and the poisonous constituent (if present) in the meal could be found, milkweed would probably become a profitable crop, particularly for submarginal land.

Recommendations.

- (1) More complete extraction of the oil could be obtained by lengthening the extraction time.
- (2) By properly feeding the flakes into the extractor it should be possible to run the flakes without having to dry them. The main reason for drying the flakes was to get them to fall through the hopper and into the chain properly.
- (3) Complete removal of the floss, which is easily accomplished would facilitate processing the seed.

(4) The removal of milkweed seed fines from solvent-oil solutions should be further studied.

(5) A method of making milkweed seed meal palatable and non-poisonous should be sought.

Extraction of cottonseed oil

Preliminary data. Cottonseed meats contain about 36 per cent oil, 29 per cent protein, 18 per cent carbohydrates, 6 per cent fiber, 4 per cent ash, and 6 per cent water. Production of cottonseed oil in the United States during the period 1937-1942 averaged over 1,400,000,000 pounds annually (9). Usually the oil is removed by squeezing it out through the application of pressure. Only very recently has solvent extraction been applied to the winning of cottonseed oil (108). This lag in the application of solvent extraction to cottonseed has been due partially to the fact that the increase in oil obtained is only about 8 per cent and partially to difficulties met in the extraction process.

The cottonseed meats used in these studies were furnished by the Forrest City Cotton Oil Mill, a division of Armour and Company. The hulls had been removed before shipment, but the removal was not complete. The meats were stored in bags until ready for use.

The meats contained 6 per cent moisture and 35 per

cent oil (dry basis). The meats as received contained too many fines to permit solvent to flow through them at the necessary speed. When the meats were shaken on a ten mesh screen, 25 per cent passed and 75 per cent were retained. The portion passed contained 26 per cent oil (6 per cent moisture basis) and that retained contained 33.8 per cent oil. The portion retained would permit sufficiently rapid penetration of the solvent, but the solvent removed the oil very slowly. Only 11.2 per cent oil was removed by a half hour Soxhlet extraction of the whole meats and 14.2 per cent by a one hour extraction with trichloroethylene. Attempts to flake the meats with no pretreatment and attempts to flake meats which had been merely heated crumbled them and produced too many fines to permit rapid penetration of the solvent.

When the meats were steamed to a moisture content of 11 to 12 per cent they became quite pliable. Good flakes were prepared from these meats by running them through rolls ordinarily used to flake soybeans. However, the pressure on the rolls had to be greatly reduced to prevent crumbling the meats. Tests on the rate of flow of solvent through the flakes showed that the rate of flow increased as flake thickness increased as one would naturally expect. The rate of flow through dry flakes was much

higher than for moist flakes because the former are not very pliable and do not tend to settle into a compact impervious mass. Cottonseed flakes are not as strong as soybean flakes of the same thickness. Consequently, flaked cottonseed meats must be thicker to prevent them from breaking up into more fines and to permit solvent to flow through them at the same rate.

The data in Table 14 were obtained when flakes of different thicknesses were extracted with trichloroethylene in Soxhlet extractors.

Table 14

Flake Thickness and Oil Extracted from Cottonseed Flakes

Flake thickness			Moisture (%)	Oil (% dry basis)		
Max.	Min.	Ave.		15 min.	30 min.	45 min.
0.040	0.025	0.030	14.8	29.6	31.7	35.0
0.030	0.010	0.020	9.28	31.8	31.9	34.0
0.039	0.016	0.027	7.60	29.7	31.5	35.0

Flakes were to be in contact with the solvent in the extraction pilot plant for 15 to 30 minutes. Evidently if good extraction was to be obtained, the flakes should have an average thickness of less than 0.020 inches.

Meats which had been flaked and allowed to dry for 48 hours were extracted with both trichloroethylene and

hexane in Soxhlet extractors. As shown by Table 15 trichloroethylene extracts a greater percentage of oil.

Table 15

Extraction of Cottonseed Oil
with Hexane and Trichloroethylene

Solvent	Oil (% dry basis)	
	1 hr.	2 hr.
Hexane	31.6	31.9
Trichloroethylene	33.3	33.9

Pilot plant runs on cottonseed oil extraction.

The apparatus used in the extraction of cottonseed oil was exactly the same as that used for extraction of milkweed seed oil. The two section falling film evaporator described in the section on evaporation was used for evaporating solvent from the solvent-oil solution. Stripping was performed in the 8 inch diameter column packed with Berl saddles. The meats were not cracked before they were flaked and were dumped into the feed hopper by hand.

Run No. 1:

Cottonseed meats were steamed until pliable, flaked, and allowed to dry overnight on the floor. The flakes then contained 7.26 per cent moisture. They were rather thick, averaging 0.0258 inches in thickness. Five hundred

milliliters of solvent ran through fresh flakes in the rate testing tube shown in Fig. 5 in 84 seconds and through air dried flakes in 59 seconds.

The Sparkler filter was by-passed. The Redler conveyor was run at 5.6 inches a minute in order to allow more time for extraction (about 30 minutes). Solvent pumped to a height of 16 inches in the drainage section of the extractor but ran through the flakes satisfactorily at all times. In order to help obtain good extraction the solvent was run through the extractor quite rapidly. Some oil was entrained by the vapor and carried from the evaporator, through the condenser, to the solvent storage tank. This was first noticed when the trichloroethylene in the rotameter became colored. Entrainment was caused by a high rate of solvent flow, by fouling of the evaporator with fines, or both.

The length of the run was two hours and six minutes during which time 250 pounds of meats were run through the extractor. The overall yield of oil was 24 per cent. Meal samples contained 3.53 and 7.34 per cent oil. The former sample probably had been soaking in the solvent before the run started.

Run No. 2:

Cottonseed meats were steamed, flaked, and allowed to air dry for two days. The flakes contained 7.8 per

cent water after drying and averaged 0.022 inches in thickness.

The Redler conveyor was operated at 5.6 inches a minute. The filter was not used. Solvent rose to a height of 12 inches above the solvent inlet in the drainage section of the extractor. Some oil was again entrained and carried into the storage tank by the solvent. Although the amount of oil contained in the solvent was not very great (about 0.2 per cent) it did hinder complete extraction because one pound of solvent was carried into the driers with every pound of meal.

The run lasted two hours. Two hundred pounds of flakes were extracted giving 56 pounds of oil, an overall yield of 28 per cent. Meal samples contained 4.3, 4.7, and 4.6 per cent oil when determined by a two hour Soxhlet extraction with trichloroethylene.

The oil obtained was dark in color. It was decided to clean the evaporator tubes in order to help prevent entrainment and to run thinner flakes to obtain better extraction.

Run No. 3:

Meats were flaked and air dried for 24 hours. They then contained 6.9 per cent moisture. These flakes were thin (0.0169 inches) and contained more fines than any yet prepared.

The Redler conveyor was run at 5.6 inches a minute and the filter was not used. During most of the run the system operated quite satisfactorily. However, about 45 minutes before the end of the run the solvent became colored with entrained oil. The evaporator tubes were found to be fouled with solids when examined at the end of the run. This fouling of the tubes must have caused entrainment of oil by the vapor.

This run lasted two hours and forty-one minutes. A total of 250 pounds of flakes were extracted. Sixty-three pounds of oil were obtained, giving an overall yield of 25 per cent. The overall yield of oil did not check with the degree of extraction as found by analysis of the meal. Meal samples contained 2.7, 2.8, and 4.0 per cent oil when extracted with trichloroethylene. It was decided to make another run using flakes of the same thickness but with the miscella filter connected.

Run No. 4:

After being flaked the meats were allowed to air dry for 48 hours. The dried flakes contained 5.64 per cent moisture and averaged 0.0150 inches in thickness.

The conveyor speed was 5.6 inches a minute. At the beginning of the run the miscella was pumped through the Sparkler filter before going to the evaporator.

After one hour and forty-five minutes the pressure on the filter rose to 60 pounds per square inch, and miscella practically ceased running through. The run was stopped for 15 minutes while the filter was by-passed. A cake three-fourths of an inch in thickness had formed in the filter. The cake weighed $4\frac{1}{2}$ pounds and was collected from 158 pounds of flakes.

Solvent was run through the extractor at a rather high rate to obtain better extraction. The solvent began to pick up oil even before the filter was by-passed. This behavior was probably caused by irregular operation of the filter. No miscella would be pumped through until a considerable amount collected in the settling chamber just before the pump. Then all the miscella would be pumped through in a short time causing a great surge of vapor in the stripping column.

Trichloroethylene was pumped into the drainage section of the extractor to a height of 19 inches but ran through the flakes satisfactorily at all times. The run lasted $2\frac{1}{2}$ hours during which time 225 pounds of flakes were extracted. A total of 60 pounds of oil were obtained, giving an overall yield of 26.7 per cent. One hour Soxhlet extraction of the meal gave the following results:

Sample No.	Per cent of oil	
	Trichloroethylene	Hexane
1	2.7	1.87
2	3.2	
3	3.7	2.41
4	2.7	

Run No. 5:

Cottonseed meats were flaked and air dried for four days. As usual, the flakes turned brown on prolonged exposure to air. The flake thickness averaged 0.0184 inches. A rate test on the dry flakes showed that 500 milliliters of trichloroethylene would run through the flakes in the rate tube shown in Fig. 5 in 61 seconds. The dried flakes contained 5.47 per cent moisture.

The miscella was not filtered. The extractor conveyor was run at 5.6 inches a minute so that the extraction time was 30 minutes. Solvent was run through the extractor at a rather rapid rate giving a miscella containing 14 to 15 per cent oil. The solvent rose in the drainage section to a height of 16.5 inches above the solvent inlet.

The extraction system operated quite satisfactorily. A total of 575 pounds of flakes were extracted in six hours and five minutes, a rate of 96 pounds an hour. One hundred and sixty-five pounds of oil were obtained, a

yield of 28.7 per cent. Twelve pounds of oily fines were obtained from the settling chamber at the bottom of the evaporator.

Soxhlet extraction of the meal for two hours with hexane gave the following results:

Sample No.	Per cent oil
1	2.0
2	1.9
3	2.5
4	2.2
5	2.2

The drying section of the pilot plant was not sufficient to remove all traces of solvent from the cottonseed meal. However, the meal had a good taste, odor, and color and appeared to be of prime quality. Cottonseed meal has a poisonous constituent, gossypol, which has to be destroyed before the meal can be fed to certain kinds of animals. Since trichloroethylene is a solvent for gossypol, it would seem that an additional cooking process for the destruction of gossypol would not be needed.

A poor quality of oil was obtained. The main reason for the low quality was undoubtedly overheating in the evaporating and stripping processes. Using old meats and allowing the flakes to air dry for a long period before

use also lowered the quality of the oil.

Recommendations.

(1) A better quality of oil could be obtained from meats which are fresh and are extracted immediately after they are flaked. Further work should be done on extracting meats immediately after they are flaked.

(2) The key to the success of trichloroethylene as a solvent for cottonseed oil is in the recovery of a good grade of oil. Removal of the color cells from the miscella, removal of the fines, and stripping under vacuum should be studied.

(3) Trichloroethylene, unlike hexane, extracts gossypol from cottonseed meats. The effect of this material upon the oil should be determined. The meal should also be examined to see if it is free of poisonous gossypol.

Extraction of oatmeal oil

Preliminary data. Oats contain such a low percentage of oil that it is usually not profitable to extract it. However, for certain purposes such as the preparation of precooked cereal the oil should be removed so that it will not give the cereal a bad taste by turning rancid. A typical sample of oatmeal contains 7.3 per cent oil, 66.3 per cent carbohydrates, 16.7 per cent protein, and 1.3 per cent fiber (66).

Buckeye Brand, Quick Cooking, oatmeal was furnished by the Quaker Oats Company of Cedar Rapids, Iowa. The oatmeal contained 6.5 per cent oil and 8 per cent water. Five hundred milliliters of trichloroethylene ran through a bed of the oatmeal in the apparatus shown in Fig. 5 in 89 seconds indicating that the meal probably could be processed in the extraction apparatus. Drying the oatmeal did not change the rate of penetration of the solvent.

Pilot plant extraction. The system used for oatmeal oil extraction was that shown in Fig. 3 except that the oatmeal was dumped directly into the feed hopper with no pretreatment. Since the oatmeal offered more resistance to the flow of solvent than soybeans, the extractor was slowed to 5.6 inches a minute.

Running at 100 pounds of oatmeal an hour the meal pumped solvent to a height of 7 to 11 inches in the drainage section. Miscella contained 4 to 10 per cent oil and a large amount of fines. After a few hours operation the Kelly filter had to be by-passed because the rate of filtration had fallen to practically zero. With the filter by-passed the stripping column was fouled with fines and the stripped oil contained a large amount of fines. Filtering the miscella or oil was very difficult because the cake formed was almost impervious. Except

for the large amount of fines and the difficulty of removing them, the system worked very satisfactorily. Extraction of the meal with hexane for an hour in Soxhlet apparatus gave the following results:

Sample No.	% Oil
1	0.95
2	0.86
3	0.71
4	0.74
5	1.37
6	0.86

Recommendations.

- (1) Screening the fines from the oatmeal would greatly simplify the extraction process.
- (2) Further work should be done on the separation of oatmeal fines from miscella.

Extraction of corn oil

Preliminary data. Corn germs are obtained as a by-product of the corn-starch and grain alcohol industries. Most of the germs are separated from the rest of the kernel by the wet milling process and are pressed to remove the oil. Production of corn oil in the United States in 1941 was over 200,000,000 pounds (9) most of it

being used as a salad and cooking oil. The germs contain about 56 per cent oil, 20 per cent carbohydrates, 11.5 per cent protein, and 9 per cent fiber (111).

Wet process corn germs were supplied by the American Maize Products Company of Roby, Indiana. The germs as received contained 3.6 per cent moisture and 51 per cent oil. Although untreated whole germs permitted rapid flow of the solvent, oil was removed from them very slowly. A 30 minute Soxhlet extraction of whole germs with trichloroethylene yielded only 14 per cent oil. Flaking untreated germs and flaking germs which had been only heated produced too many fines to allow rapid penetration of solvent.

Corn germs steamed to moisture contents of 6 to 10 per cent could be rolled into good flakes. Thick flakes contained fewer fines and permitted more rapid flow of the solvent through the bed than did thin flakes but were less easily extracted as shown by the data in Table 16 on Soxhlet extraction with trichloroethylene.

Pilot plant runs. The arrangement of pilot plant apparatus used for extracting corn germs was that shown in Fig. 3. The germs were steamed to a moisture content of 6 to 10 per cent and run through the rolls without being cracked.

Table 16

Extraction of Corn Germs

Extraction time (min.)	Flake thickness (in.)	Oil extracted (%)	Oil removed by additional 2 hr. extraction (%)
60	0.012	50.5	1.6
60	0.0088	50.0	1.3
45	0.009	49.2	0.89
45	0.009	49.0	1.92
30	0.0116	43.6	4.7
30	0.0105	47.9	3.8

Run No. 1:

In the first run the extractor was run at 5.6 inches a minute. Flakes 0.015 inches in thickness were extracted at a rate of 44 pounds an hour. Miscella left the extractor containing 14 to 18 per cent oil. Meal samples contained 6.3, 5.9, and 5.5 per cent oil.

Run No. 2:

Flakes having the same thickness as in the previous run were next extracted with the conveyor running 3.3 inches per minute. Extraction time was, therefore, about 55 minutes. Miscella contained 10 per cent oil. Meal samples containing 3.7, 4.9, and 5.2 per cent oil were obtained.

Run No. 3:

The next step was to reduce the thickness of the flakes to 0.011 inches. With the conveyor running 3.3 inches per minute miscella containing 11 to 21 per cent oil and meal samples containing 3.3 per cent oil were obtained.

Run No. 4:

Since a considerable amount of oil was being left in the meal even with an extraction time of 55 minutes, it was thought that the mass of flakes might be shrinking as oil was extracted allowing solvent to by-pass the flakes. Examination of the extracted and partially dried flakes being carried in the Redler chain was inconclusive. Therefore, it was decided to re-extract flakes which had been previously extracted. Meal containing 6 per cent oil was extracted with the conveyor running 5.6 and 3.3 inches a minute. The corresponding rates of feed were 34 and 20 pounds of meal an hour. Meal samples contained 3.4 and 2.0 per cent oil indicating that short-circuiting of the solvent was not occurring.

Run No. 5:

Meal containing 3.5 per cent oil was extracted with the conveyor running 5.6 and 3.3 inches a minute. The solvent was preheated to 67° C. before entering the

extractor. Meal samples contained 1.1 and 0.98 per cent oil. Miscella contained 1 per cent oil.

Run No. 6:

In order to aid the removal of oil from the germs it was decided to use thin flakes and heated solvent. The extractor was run at 5.6 inches a minute giving an extraction time of 30 minutes. The solvent was heated to 67° C. About 45 pounds an hour of germs were extracted. Flakes 0.009 inches in thickness were extracted to 3.5 per cent oil and flakes 0.007 inches in thickness were extracted to 2.5 per cent oil. Miscella leaving the extractor contained 17 to 21.5 per cent oil and leaving the evaporator contained 85 per cent oil.

Aside from obtaining complete extraction very little difficulty was experienced in extracting corn germs. Only slight pumping of solvent in the drainage section occurred at any time. One to 2 per cent of the weight of the germs was obtained as fines on the dry, oil-free bases. The filter cake produced by these fines was not as porous as that made by soybean fines, but did not cause any particular difficulty in the filtering operation. The oil was free from solvent and appeared to be of good quality. With the extraction section modified so as to give hot extraction for at least 45 minutes the system

could undoubtedly be used to prepare a high quality of corn germ oil and meal.

Recommendations.

(1) The conditions under which one could best obtain very thin corn germ flakes should be determined.

(2) The effect of extraction time upon the amount of oil obtained from corn germs should be further studied.

(3) The extraction section of the apparatus should be modified so as to give a longer extraction time without sacrificing capacity or causing pumping. Perhaps a two step extraction would be feasible.

(4) Filtering corn germ fines and stripping corn germ oil should be studied to obtain data for equipment design.

CONCLUSIONS

Apparatus

1. Apparatus for the continuous countercurrent extraction of soybean oil with solvents such as trichloroethylene at a rate of 2.6 tons of oil a day has been designed and operated successfully.

Analytical Control Procedures

1. Rapid and simple methods for the determination of oil in soybean meal and for trichloroethylene in oil, water, and meal have been developed. Laboratory equipment for a small extraction plant should include a Westphal balance (or a series of hydrometers ranging from 0.9 to 1.4), a moisture oven, a Steinlite moisture tester, an analytical balance, a Soxhlet extraction apparatus, and various smaller pieces of equipment. Reagents for the Fujiwara pyridine test should also be on hand.

Extraction

1. To be extracted in apparatus similar to that de-

scribed the oleaginous material must form a bed sufficiently porous to permit rapid penetration by the solvent. Apparatus for the estimation of the rate of flow of solvent through various materials has been constructed.

2. Soybeans need only be dried sufficiently to keep in storage. Beans containing as high as 14 per cent moisture have been successfully extracted in the apparatus described.

3. Beans should be cracked and heated before they are flaked. Flakes should not be more than 0.010 inches in thickness.

4. Flakes should be fed into the extractor at a point above the miscella level.

5. The extraction chamber should be inclined in such a way that the miscella will rise as it becomes richer in oil. The solvent inlet should be several feet higher than the miscella outlet.

6. The optimum concentration of oil in the miscella is about 20 per cent. To obtain this concentration about 2 pounds of solvent per pound of soybeans must be fed into the extractor.

7. An extraction time of 20 minutes is sufficient for good extraction. Running at 15 tons of soybeans a day the commercial plant produced a meal containing 0.7 to

0.9 per cent oil.

8. Heating the solvent before it enters the extractor seems to be desirable because it gives better extraction, helps the driers in cold weather, and wastes very little heat.

9. Oil considerably better in quality than required by commercial standards can be consistently obtained.

Filtration

1. Because of the filtering action of the bed of soybean flakes carried by the Redler conveyor and of the screens on the miscella outlets only 0.05 per cent of the beans are obtained as fines.

2. The solvent-oil solution containing fines may be filtered either after it leaves the extractor containing 20 per cent oil or after it leaves the evaporator containing 80 per cent oil. If concentrated miscella is filtered over 50 per cent the fines may be removed by settling to the bottom of the filter.

3. The small amount of fines obtained makes disposal of the filter cake a minor problem.

4. Pressure filtration, except for gravity, is not necessary. Filters should be placed so that a head of at least 10 feet is available. Filters may be of light

construction and made inexpensively. Only two filter cleanings are required during a week by a plant processing 15 tons of 13 per cent moisture beans a day.

5. Moisture affects the rate of filtration. The higher the moisture content of the beans the lower the rate of filtration.

6. Miscella containing such a low percentage of fines as that obtained from the plant described can be concentrated in a rising film evaporator for several months before the evaporator tubes have to be cleaned. The fines will not pass through a packed column.

7. Filter cloth especially designed for soybean oil filtration, such as "Soytex" filter cloth, is recommended.

Evaporation

1. The recovery of soybean oil from solutions of the oil and trichloroethylene is best accomplished in two steps. The solution is first concentrated to about 80 per cent oil. Then the remaining trichloroethylene is removed by stripping with steam.

2. Solutions of trichloroethylene and soybean oil can be concentrated in either falling film or rising film evaporators. The latter have a higher capacity and are less likely to become fouled with fines.

3. One-inch pipes, 17 feet long, jacketed with steam at 70 to 90 pounds per square inch gauge are suitable for concentrating miscella. One such vertical pipe will concentrate a solution from 20 to 80 per cent oil at a rate of 50 pounds of oil an hour.
4. More highly concentrated solutions may be obtained by recirculating part of the concentrated miscella.
5. The evaporator should be constructed so that part of the tubes can be disconnected and cleaned without interfering with the operation of the rest of the evaporator.
6. The miscella surge tank should be connected directly to the miscella outlets so the surging action will be exerted through the screens.
7. The bottom of the evaporator should be at least four feet below the miscella outlets so gravity feed can be used.
8. The evaporator can be made of standard iron pipe. The surge tank and flash chamber can be made of light sheet iron.

Stripping

1. Steam is the most satisfactory medium for stripping trichloroethylene from soybean oil.
2. A number of different types of stripping apparatus

can be used. Packed columns are the most satisfactory for small plants.

3. Stripping columns can be operated below 100° C. if means of separating the oil, phosphatides, and water are provided.

4. Stripping at temperatures above 100° C. at atmospheric pressure is recommended. Solutions to be stripped must contain over 50 per cent oil, preferably over 80 per cent. The miscella must be above 100° C., the steam must be dry, and all condensation of moisture inside the column must be prevented.

5. Means for distributing the steam and miscella entering the column should be provided.

6. The column should not be overheated. Overheating increases entrainment by causing the miscella to boil and decreases the quality of the oil.

7. A stripping column 14 inches in diameter packed to a depth of 14 feet with 1-inch Berl saddles will strip all detectable traces of solvent from an 80 per cent solution of soybean oil in trichloroethylene at a rate of 200 pounds of oil an hour. About 180 pounds per hour of stripping steam are required.

8. Suitable dry stripping steam can be obtained by jacketing a low pressure steam line with high pressure

steam.

9. Heavy emulsions of oil and water, which hold solvent in suspension, may be broken by the emulsion breaking apparatus described. The oil and hot water pass to the drain and the solvent is recovered. Such heavy emulsions may be caused by entrainment of oil and subsequent condensation of the steam and solvent. They may be caused also by overheating the column while no fresh miscella is being stripped but while steam is passing through the column. Overheating may occur at the beginning or end of runs.

10. The stripping column can be cleared by boiling a dilute caustic solution in it.

11. Judging from the behavior of pilot plant scale stripping columns, the efficiency of the stripping column used in the commercial plant can be increased considerably.

Drying and Toasting

1. Part of the solvent can be removed from the soybean meal by steam jacketing a section of the Redler drier. Jacketed tubular driers containing ribbon conveyors are suitable for removing the last traces of solvent from the meal.

2. Some means of stirring the meal in the driers is essential. Stirring should not be violent enough to cause excessive dusting and the stirrers should be capable of handling lumps of meal.
3. When driers are made in several sections, one above the other, the drop between driers should be as small as possible to prevent undue dusting.
4. The vapor leaving the driers should be sprayed with hot water to remove dust. Most of the water can be re-circulated. For a small plant ordinary garden hose nozzles give a good spray and are easily cleaned.
5. About half the water removed from the soybeans leaves with the trichloroethylene. The remainder is removed after all the solvent has evaporated. Blowing steam into the end of the toaster is not necessary when beans of high moisture content are being processed because water vapor from the meal sweeps out solvent vapor.
6. The driers should be tilted so heavy solvent vapor will fall toward the vapor line.
7. Vapor in the toaster should flow countercurrently to the meal. In the driers it may flow in either direction. A barrel valve forms an effective vapor seal at the end of the toaster.
8. Vapor velocities should be kept low in the driers and

toaster to reduce dusting.

9. Cool spots where moisture can condense should be avoided in the driers and toaster. Moisture condensing in such regions causes the meal to become sticky and lumpy and causes the iron to corrode. When the driers are kept hot no noticeable corrosion occurs.

Condensation

1. The minimum number of condensers required is three--- two small ones for the emulsion breaking apparatus and one large one for the driers, toaster, and evaporator.
2. The main condenser should be placed below the level of the drier and toaster and above the solvent-water separator.
3. Non-condensable gases should be vented from the condensers into the incoming flakes. A small blower placed in the vent line aids in getting vapor to the condenser but probably is not essential.
4. Vapor should condense inside the tubes so they can be cleaned from time to time.
5. Steel construction is suitable for the condensers. Some corrosion of the steel occurs but it is not sufficient to justify more expensive construction materials.

Extraction of Oil from Materials Other Than Soybeans

Extraction of milkweed seed oil

- a. The major portion of the oil in milkweed seed cannot be extracted unless the seed coat is broken.
- b. The best way to prepare milkweed seed for extraction in the apparatus developed at Iowa State College is to steam the seed to a moisture content of 11 to 12 per cent, flake them, and allow them to dry somewhat. Complete removal of the floss is desirable but not essential.
- c. With an extraction time of 15 minutes meal containing 4 to 5 per cent oil is obtained. More complete extraction can be obtained by using a longer extraction time.
- d. Milkweed seed fines are harder to filter from the miscella than soybean fines.
- e. Good quality milkweed seed oil can be obtained by extraction with trichloroethylene.

Extraction of cottonseed oil

- a. Cottonseed meats should be steamed before they are flaked and should be flaked with reduced pressure on the rolls. Drying the flakes before extraction is

helpful.

b. Using the pilot plant apparatus described and an extraction time of 30 minutes cottonseed meats can be extracted to 2 per cent oil.

c. A good quality of meal can be obtained from the process. The method of recovery of a good quality of oil has not yet been worked out.

d. More fines are obtained from the processing of cottonseed meats than from soybean processing. The fines are somewhat harder to filter than soybean fines.

Extraction of oatmeal oil

a. The amount of oil in oatmeal can be reduced to considerably less than 1 per cent using the soybean pilot plant.

b. The elimination of the very hard to filter fines from oatmeal or a method of removing them from the miscella is essential to the success of a process of extracting them.

Extraction of corn oil

a. Corn germs can be extracted to less than 3 per cent oil using the pilot plant described and an extraction time of 30 minutes. With the alteration of the

extraction section of the plant to give a longer extraction time corn oil can be profitably extracted with trichloroethylene.

SUMMARY

There has long been a need for an efficient small-scale plant for the extraction of soybean and similar oils. For a number of years research toward the development of such a plant has been undertaken at Iowa State College.

After considerable preliminary work a pilot plant was erected using a Redler chain to convey the soybean flakes through a non-inflammable solvent, trichloroethylene. All the unit operations involved were studied carefully and the system made as simple and fool-proof as possible. With slight modifications the plant was found to be capable of extracting the oil from several types of oleaginous materials using trichloroethylene or similar solvents.

A commercial plant was erected at Plainfield, Iowa, in connection with a small elevator and feed mixing company. Experimentation was continued at this plant. The plant has been operating continuously for over a year and has a capacity of over 15 tons of soybeans a day. High quality oil and meal are obtained at very reasonable operating costs. It is expected that many similar plants will be constructed in the near future.

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