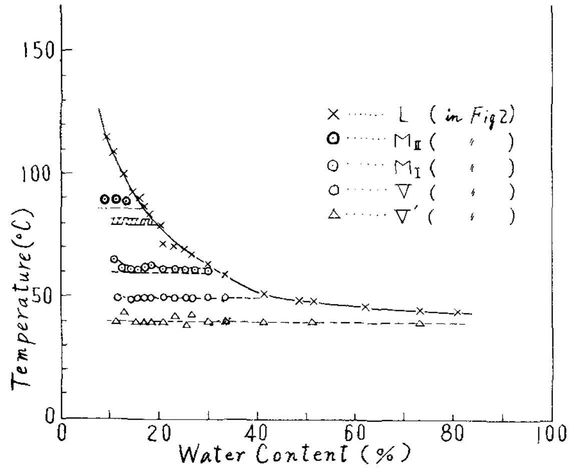

FIG. 5. Phase transition points for soaps of various moisture contents shown by electrical resistance values. (L, M_{II}, etc., refer to corresponding points in Figure 2.)

seem to indicate phase transitions at about 50° and 40°C., which were not found by McBain.

Thiessen and Stauff (5) have noted that there are phase changes near the melting point of the constituent fatty acids in a soap and have named this the "genotypic point." Since fatty acids in commercial soap have a melting point around 40°C., the horizontal line at this temperature in Figure 5 may be the "genotypic point."

Studies with single pure soaps (7) have shown that transition points are practically independent of water content. For the sodium soaps of lauric to stearic acids, conductivity decreased to a minimum between 80° and 90°C., after which it started to increase again. This effect was independent of the kind of fatty acid or water content. It appears that these effects may predominate at around M_{II} of Figure 2.

Summary

Electrical resistance values are reported for soaps of various moisture contents. Marked changes in resistance were found at 40–50°C. Below this temperature the logarithm of conductivity is a linear function of inverse temperature, and the slope of the curve is practically independent of water content. Above 40° resistance is markedly affected by the water content of the soap. For soaps containing 12–30% water, heating and cooling curves show definite maxima and minima in electrical resistance values. This effect is less obvious in soaps containing more than 30% water. The phase transition points estimated from resistance measurements are in good agreement with those deduced from other physical measurements.

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Flash Desolventizer Operation to Produce Soybean Protein Flakes[1]

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SOYBEAN PROTEIN FLAKES are useful for many industrial applications, for example, in preparing plywood or paper-coating adhesives. However flaked, hexane-extracted soybeans are not suitable unless precautions are taken to minimize denaturation because of heat treatment incurred in the desolventizing operation. Previous results obtained by Belter *et al.* with a flash desolventizer indicated that such a unit is well suited to hexane removal without extensive protein denaturation (2). However the effects of process variables were not studied; moreover aged soybeans with partially denatured protein were used. The present investigation was undertaken to establish desirable process conditions for flash desolventizing hexane-extracted flakes without significant protein denaturation. Soybeans from a current harvest were used.

The flash desolventizer process employs superheated vapors in direct contact with solvent-wet feed to provide the heat required for vaporization of solvent and to convey solids through the desolventizing tube to a cyclone separator. A major portion of these vapors is reheated and recycled, and the remainder is sent to a condenser. This desolventizing process combines certain features of a horizontal, vapor type of desolventizer (4) with those of a flash or pneumatic conveying drier.

The performance of a flash desolventizer is determined by several factors that control the heat transfer rate and consequently the rate at which entrained solvent is vaporized. One of the most important is the direct contact obtained between solids and superheated vapors whereby the flakes' extensive surface area is fully used for heat transfer. Turbulent vapor flow in the desolventizing tube and a high temperature differential at the solids inlet also increase the rate. As the solids are simultaneously dried and carried to the cyclone separator, the temperature differential progressively decreases. Meanwhile solvent and moisture evaporating from the solids moderate the latter's temperature rise. The high heat-transfer rate, combined with the thin flakes, permits rapid solvent

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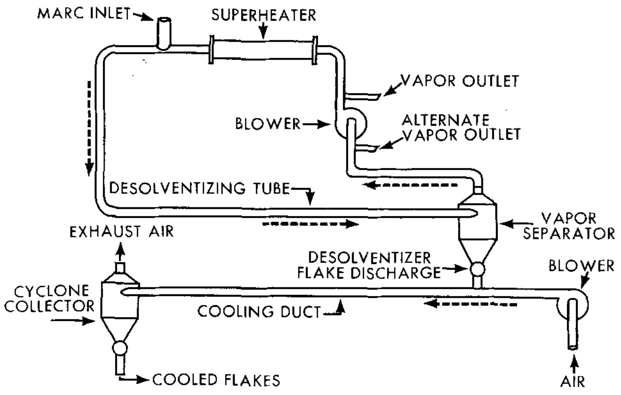
[2] This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, United States Department of Agriculture.

removal while subjecting the solids to elevated temperature for only a few seconds. By cooling immediately, damage to the heat-sensitive proteins is held to a minimum. In the flash desolventizing system a high degree of solvent removal is obtainable even though the solvent is vaporized into an atmosphere of its own vapor.

Materials and Equipment

Cleaned soybeans, No. 1 grade, Clark variety, of seed quality from the 1957 crop were processed one to three months after harvest. They were conditioned to 11% moisture, cracked, partially dehulled, heated under mild conditions, and flaked in pilot-plant preparation equipment. The flakes averaged 9.5% moisture and 0.009 in. thick with a standard deviation about 0.003 in. They were extracted with commercial hexane (b.p. 150–160°F.) in a continuous countercurrent extractor to a residual oil content of 1.3 to 0.4%, depending upon the solids feed rate. The extracted flakes were drained about 90 sec. on an inclined drag conveyor to 35–40% total volatiles, transported by a 4-in. screw conveyor, and discharged through a choke feed into the solids inlet of the flash desolventizer.

A flow diagram for the flash desolventizer is shown in Figure 1. The ductwork for the system consisted of 3-in. i.d. galvanized-steel tubing, which formed a closed circuit with a 250-cfm. turboblower, a single pass shell-and-tube heat exchanger, and a 20-in. diameter cyclone separator. A conventional 3-in. sheet metal tee was used as the solids inlet in the duct, and the distance to the cyclone was 24 ft. A 3-in. gate valve in the blower suction line controlled the recycle vapor flow. Vapors to the condenser were withdrawn on the discharge side of the blower. Desolventized solids, at temperatures of 160–230°F., were discharged from the hot cyclone separator through a rotary feeder valve directly into a cool air stream. This air stream provided rapid cooling to minimize heat-induced protein denaturation and simultaneously conveyed the product through 30 ft. of 4-in. o.d. duct to another cyclone separator. The installation's evaporative capacity was approximately 73 lb. of hexane and 3½ lb. of water per hour when operated with a recycle vapor temperature of 310°F. and a vapor velocity of about 66 ft./sec. The vapor transport lines, blower, and cyclone separator were wrapped with steam tracer copper tubing and covered with magnesia insulation.



FLOW DIAGRAM OF FLASH DESOLVENTIZER

Fig. 1. Flow diagram for flash desolventizer and cooling units.

Experimental Operation

Equilibrium test periods were either 1 or ½ hr. in length after steady-state conditions were reached, based upon recorded vapor temperatures at the cyclone inlet and outlet and the solids inlet. The equipment responded quickly to any changes, and often only 15–30 min. were required to reach equilibrium. A valve in the vapor take-off line was throttled to maintain essentially atmospheric pressure at the solids inlet as measured by a water manometer. This pressure fluctuated about ± 2 cm. A small stream of carbon dioxide gas was introduced into the side port of the solids inlet tee to minimize entry of superheated vapors into this port because, upon condensation, these vapors interfered with proper feeding of the solids, increased the desolventizer load, and induced additional protein denaturation because of the higher moisture level. With a suitable feeding device this gas stream is not needed. Steam at 120 p.s.i.g. was used in all tracer lines with the exception that none was used for the desolventizing duct, and the pressure in those wrapped around the hot cyclone separator was reduced to 15 p.s.i.g. to approach adiabatic conditions.

Vapor velocity measurements were made with a Pitot tube. To determine the recycle vapor composition, hexane and water were condensed from a sample of the hot vapors, and an Orsat analysis was then made of the noncondensable portion. A composite of samples taken hourly of the extractor feed was used for the moisture and Nitrogen Solubility Index (NSI) determinations. The decrease in NSI values in subsequent processing steps was used to measure the extent of protein denaturation. Spot samples for analysis were taken of the extracted, drained flakes, and of desolventized solids from just below the rotary valve and after the air cooler. The hot desolventized solids collected for NSI determinations were rapidly cooled on a metal tray.

Desolventized solids temperatures were measured below the rotary valve by a thermocouple in a cup arranged for external manipulation.

Total volatiles determinations, *i.e.*, for moisture and solvent (M&VM), were made by the A.O.C.S. Official Method BA-2-38. NSI values were obtained by a modification of the method of Smith and Circle (5) with the water-solids mixture agitated for 2 hrs. in a 25°C. constant temperature bath with flat-blade paddles driven at 125 r.p.m.

The residual hexane content was estimated by a spectrophotometric method, based upon absorption in the ultraviolet by benzene, which appeared as a contaminant in the hexane. The absorption determination was made on an iso-octane solution used to extract hexane and benzene from the solids. The method was calibrated against samples containing known amounts of hexane.

The effects of recycle vapor temperature, recycle vapor velocity, and solids feed rate were studied. Recycle vapor temperatures were varied between 225° and 310°F., vapor velocities of approximately 45, 55, and 66 ft./sec. at the solids inlet were used, and flaked soybeans were fed to the extractor at rates from 50 to 200 lb./hr. The feed of de-oiled, solvent-wet flakes to the desolventizer is calculated to be about 18% more than these rates. Moisture content of the extractor feed was maintained essentially constant, and the length of the desolventizing tube was not changed for these tests.

The lowest vapor velocity used was the minimum observed to prevent momentary deposition of solids at the solids inlet. The highest velocity was obtained with the 3-in. gate valve set wide open.

Results and Discussion

Total volatiles in the hot desolventized solids varied between 2.4 and 9.7%, depending upon processing conditions. Correlation between M&VM and solids temperature is shown in Figure 2. At low feed rates the solids were dried much more than necessary. As the feed rate was increased, the desolventizer work load and moisture and volatiles content of the product naturally increased. For the 200 lb./hr. rate the total of volatiles was about 9%, which is slightly higher than desired as will be shown later. For a given solids temperature, the expected range in M&VM is represented by the heavy, broken lines, parameters A and B. Within this range the volatile content is influenced by vapor velocity, vapor temperature, and solids feed rate. The three solid lines represent results obtained at a solids feed rate of 150 lb./hr. with vapor velocities of 66, 55, and 45 ft./sec. For a given recycle vapor temperature the greatest amount of evaporation naturally was obtained at the highest velocity. As the velocity was decreased to 55 and then to 45 ft./sec., the total volatiles increased an approximate 0.75% in each case. For each of the three solid lines, the point lying on parameter A represents a recycle vapor temperature of 305°F. Other points on the 66 ft./sec. line represent progressively decreasing vapor temperatures of 270, 250, and 230°F. In this case a 75°F. decrease in the recycle vapor temperature resulted in an approximate 2% increase in the total volatiles. A shorter temperature range was covered with the other velocities.

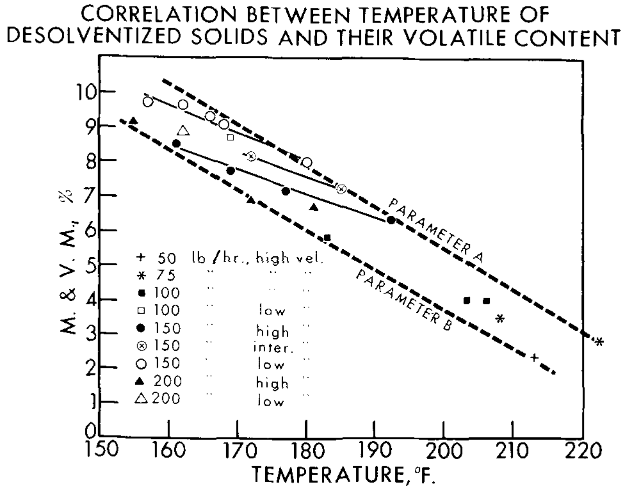


FIG. 2. Correlation between desolventized solids temperature and total volatiles content.

Of particular importance is the residual hexane content of the hot desolventized solids. The correlation with temperature is represented in Figure 3, and with M&VM in Figure 4. Optimum conditions for hexane removal were obtained by heating desolventized solids to about 170°F. and reducing their M&VM to about 7.5%. Further removal of volatiles resulted in little additional hexane removal. About one-half of the desolventized solids gave positive results in a so-called "pop" test, but the results did not correlate with the residual hexane analysis. No study was made of the means for removing the 0.3% or so of residual solvent.

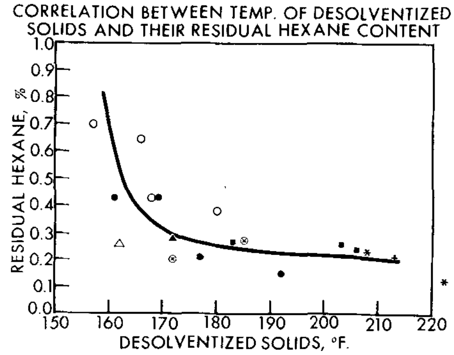


FIG. 3. Correlation between desolventized solids temperature and residual hexane content. (Legent for symbols same as Fig. 2.)

The influence of solids feed rate upon total volatiles of the desolventized solids can be seen in Figure 5. With the highest vapor velocity and an inlet vapor temperature of 300–310°F. the total content of volatiles gradually increased from 2.4 to 6.6% as the solids rate was increased from 50 to 200 lb./hr. A comparison of curves I, II, and III indicates that changing the vapor velocity about 20 ft./sec. has as much influence upon removal of volatile matter as a 75°F. change in the vapor temperature.

A correlation between inlet vapor temperature and the temperature differential between outlet vapors and desolventized solids is shown in Figure 6. While this correlation applies only to our installation since such a change as using a longer desolventizing tube undoubtedly would have decreased the temperature differential, correlations of this type can be used for design purposes. It may be noted that vapor velocity and solids feed rate had no influence on differential.

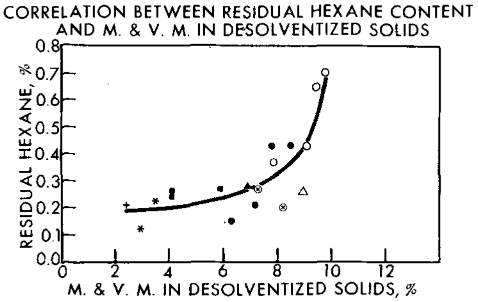


FIG. 4. Correlation between residual hexane content and moisture and volatile matter in desolventized solids. (Legend for symbols same as Fig. 2.)

Approximately two-thirds to three-fourths of the over-all temperature drop for the vapors occurred in the desolventizing duct and the remainder in the cyclone. Thus a major portion of the evaporation occurred in the duct, and much of it undoubtedly occurred near the solids inlet.

In any pneumatic conveying operation some attri-

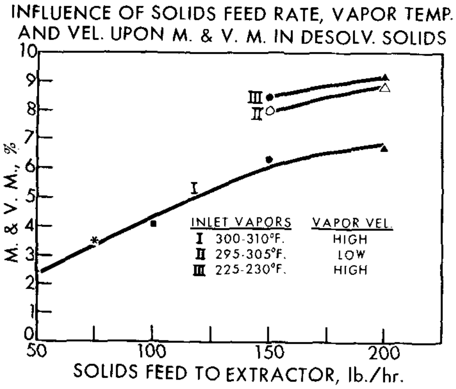


FIG. 5. Influence of solids feed rate, recycle vapor temperature, and velocity upon moisture and volatile matter in desolventized solids. (*Legend for symbols same as Fig. 2.*)

tion can be expected. Because soybean flakes are friable, some grinding naturally occurs. The extent to which the solids are reduced in size is shown by the sieve analysis in Table I. For this test the extractor was operated at a solids feed rate of 200 lb./hr., and the desolventizer was operated at the highest vapor velocity and an inlet vapor temperature of 265°F. The data indicate appreciable breakage of the flakes into particles that pass a 20-mesh sieve but only a small increase in material passing a 200-mesh sieve. This attrition causes no disadvantage when the soybean protein flakes are ground before being used, as is the normal case.

TABLE I
Sieve Analysis of Desolventizer Solids

U. S. standard sieve number	Cumulative % retained	
	Feed	Solids from hot cyclone
5	17.0	......
8	43.2	2.1
14	74.0	16.4
20	90.4	50.8
40	96.6	79.8
60	98.4	89.2
200	99.9	97.5
Pan	100.0	100.0

The average NSI value for spent flakes entering the desolventizer was 91.5%. Product NSI values for material desolventized at maximum flake temperatures between 160 and 190°F. (6 tests) and pneumatically cooled ranged between 87 and 92% and averaged 89.4% for an over-all average loss of about 2.1 NSI units. This decrease is about the same as that reported for commercial soybean processing operations wherein a horizontal, vapor type of desolventizer was employed (3). When high-solubility protein flakes are desired, this desolventizer is followed by a deodorizer operated under vacuum (1).

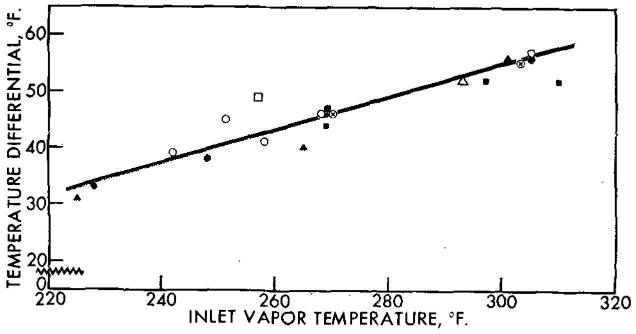


FIG. 6. Correlation of inlet vapor temperature with temperature differential between outlet vapors and desolventized solids. (*Legend for symbols same as Fig. 2.*)

Attempts were made to correlate the product NSI values with volatiles content and temperature of the flash-desolventized solids, inlet and outlet vapor temperatures, hexane/water ratio for the recycle vapors, and vapor velocity, but no consistent correlation could be obtained. Certain data indicate that the NSI value decreases roughly one percentage point for every 10°F. rise in the desolventized solids temperature between 160° and 190°F. There is also some indication that the NSI value decreases as water vapor content of the recycle vapors increases, but this effect was not fully established. Material sampled before the pneumatic cooler had NSI values averaging 0.7 unit higher than the product.

In all this work some air was drawn into the system through the rotary valve below the hot vapor separator although the vacuum in the cyclone seldom exceeded 20 cm. of water. The resultant concentration of recycle vapor noncondensables varied from 15% for the highest solids feed rate to 60% for the lowest. A limited number of tests were made to learn the effect of changing the system's pressure level to eliminate entrance of air. Both vapor and solids temperatures rose immediately, and the recycle vapor noncondensable content dropped to 1–3%. Temperature of the desolventized solids increased 15–18°F., and of the outlet vapors 10–15°F. A variation in solids feed rate had no apparent influence on this temperature rise. Operation under this pressure also increased total volatiles in the desolventized solids by approximately 1% and residual hexane content by 0.15–0.20%.

In these same tests the product NSI value decreased about seven rather than the expected two units. Part of this additional denaturation was attributed to the solids being heated to 205–220°F., which is rather high. When the solids temperature was lowered to 190°F. in one test, the NSI value showed a decrease of a little more than three units. The lower solids temperature undoubtedly was a contributing factor, and the presence of less steam in the recycle vapors possibly also minimized denaturation.

A few tests were made with one-year-old soybeans from the 1956 crop. These naturally were a little lower in their initial NSI value, but the actual NSI decrease was comparable to that obtained when using beans from the 1957 crop. The degree of solvent removal also was not affected by age of the beans.

Costs

Table II lists the equipment required for flash desolventizing and cooling operations in a solvent-extraction plant which processes 100 tons of soybeans daily into soybean protein flakes. For this estimate the following assumptions were made. Desolventizer feed contains 7.5% moisture and 35.0% hexane, and the product 7.2 and 0.3%, respectively; vapor velocity at solids inlet, 100 ft./sec., recycle vapors enter the desolventizing zone at 345°F. and leave the cyclone separator at 230°F. For every pound of volatiles- and oil-free solids, 4.55 lbs. of recycle vapors are used, a ratio that is in the range employed for some pneumatic conveying driers. For the cooling operation it was assumed that solids are cooled from 180° to

TABLE II

Estimated Investment Cost of Flash Desolventizer and Cooler

Equipment	Estimated delivered cost[a]
Desolventizer Unit—Total cost $15,000	
Superheater: steel tubes and shell, fixed tube sheet, 300-lb. working pressure shell, 1,330 sq. ft.	$ 5,500
Gas booster: 2,920 cfm. of hexane and steam vapors at 230°F., 1.0 p.s.i. differential, with 30 h.p. exp.-proof motor and two 10-in. expansion joints	3,300
Desuperheater-condenser: tube and shell, fixed tube sheet, 150-lb. working pressure shell, steel shell, copper tubes, 150 sq. ft.	1,000
Solids rotary feeder valves (2): 8-in., bronze construction, with 1 h.p. exp.-proof gear motors	2,000
Cyclone collector: low resistance-type, mild steel, gas-tight, to handle 3,700 cfm. of hexane-steam vapor	1,200
Dust filters (2): simple, intermittent duty cloth filter, gas-tight, for handling 780 cfm. of hexane-steam vapor, at 230°F.	700
Solids inlet: Venturi-type section with 10-in. vapor inlet and outlet, steel	300
Pipe and fittings: 100 ft. of 10-in. iron pipe for desolventizing duct and recycle vapor stream, necessary welding flanges, ells, and tees	1,000
Cooling Unit—Total cost $4,800	
Cyclone collector: low resistance-type, mild steel, to handle 3,000 cfm. of air	300
Solids rotary feeder valve: for use with above cyclone, 8-in. size, with ¾ h.p. exp.-proof gear motor	800
Dust filter: cloth-type, continuous, automatic, to handle 3,000 cfm. of air; exhaust fan and 7.5 h.p. exp.-proof motor and drive included	2,800
Pipe and fittings: 60 ft. of 14-in. pipe, including necessary welding ells and flanges, 15 ft. of 14-in. diam. sheet metal duct, and solids inlet	900
Desolventizing and cooling equipment delivered	19.800
Allowance for installation, 43% of delivered cost	8,500
Allowance for piping and electrical wiring, 36% of delivered cost	7,100
Allowance for instrumentation, 5% of installed cost of equipment	1,400
TOTAL cost of equipment	$36,800

[a] 1957 basis.

140°F. with air entering at 95° and leaving at 125°F., and an air velocity of 40 ft./sec.

The cost of steam, cooling water, and labor for a flash desolventizer should be essentially the same as for other types of oilseeds desolventizing equipment that employ steam as a source of indirect heat. The electrical power requirements may be a little more but will be balanced by lower maintenance costs for this simpler equipment with fewer moving parts. Approximately 1 gal. of hexane per ton of product is required as make-up solvent, over and above the the amount required to cover the usual solvent loss. This added quantity increases the product cost only slightly.

Summary and Conclusions

The flash desolventizing process removes hexane to produce high-quality, essentially undenatured, soybean protein flakes for industrial uses. The most practical level of solvent removal is achieved when solids are heated to 170–190°F. and dried to a moisture and volatiles content of 7–8%. The desolventized solids should then contain less than 0.5% residual hexane. In desolventizing extracted, partially dehulled, high-quality soybeans, the NSI value of the solids will be decreased by only about two or three percentage points. When two-month-old soybeans or those from the preceding crop year are desolventized, no difference is noted in either the degree of protein denaturation incurred or the residual hexane content of the product. Friable soybean flakes naturally are subject to some attrition during desolventizing, but this breakage is not a handicap for the type of product normally obtained.

An increase in recycle vapor velocity from approximately 45 to 66 ft./sec. had about as much effect on solvent removal as did an increase in recycle vapor temperature from 230 to 305°F. The vapor velocity undoubtedly can be increased from approximately 66 ft./sec., the maximum used in this work, to 80–100 ft./sec. which will permit either greater throughput or a reduction in size of the desolventizing duct. These benefits must be balanced against the cost of the additional power required. A recycle vapor temperature of 350°, and possibly 400° F., is considered more practical and should give a satisfactory product as long as solids are not discharged above about 190°F.

The installed cost of a flash desolventizer should be less than the cost of systems presently used to produce soybean protein flakes and operating costs should be in the same range, but the solvent loss may be a little higher.

Acknowledgment

The authors express their appreciation to J. R. Dilley for the mechanical design and layout of the cooling unit, to F. C. Wohlrabe for mechanical advice, and to A. K. Smith and E. H. Melvin for advice and assistance with analytical methods.

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[Received October 16, 1958]

Report of the Smalley Committee, 1958-1959

HEREWITH is presented the 41st annual report of the Smalley Committee. Again this season nine different types of samples were distributed by seven subcommittees. These included cottonseed, soybeans, peanuts, meals, vegetable oils, tallow and grease, glycerine, drying oils, and edible fats. In all, 4,367 samples were distributed to 494 collaborators, and about 15,000 results were tabulated. Table I shows the distribution and participation. There was a slight drop in participation of about 2%.

As of April 2, 1959, the Smalley account showed $7,431.66 as total receipts for the season, $6,482.54 as total expense, and $949.12 as the net. This will be reduced at least $100 by some charges yet to be paid. The contemplated purchase of a new mixer for preparation of the cottonseed samples will probably leave about $200 in the account for the next season. A detailed accounting has been given to the Governing Board of the Society. A final report has been sent to the collaborators, summarizing the work and listing the relative standings, which are based on the various grading systems.

It is fitting to express thanks to various subcom-