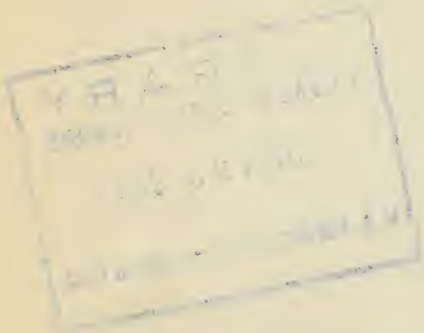
Report of the Chief of the Bureau of Agricultural and Industrial Chemistry Agricultural Research Administration

1947

UNITED STATES DEPARTMENT OF AGRICULTURE



REPORT OF THE CHIEF OF THE BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY, AGRICULTURAL RESEARCH ADMINISTRATION, 1947

UNITED STATES DEPARTMENT OF AGRICULTURE,
Washington, D. C., September 19, 1947.

Dr. W. V. LAMBERT,
Agricultural Research Administrator.

DEAR DR. LAMBERT: I present herewith the report of the Bureau of Agricultural and Industrial Chemistry for the fiscal year ended June 30, 1947.

Sincerely,

LOUIS B. HOWARD, *Chief.*

CONTENTS

INTRODUCTION

Research in the Bureau of Agricultural and Industrial Chemistry during the fiscal year 1947 continued mainly along the lines followed in 1946. The principal objective of the work was to expand the industrial utilization of agricultural products and byproducts for food, feed, and nonfood purposes.

Brief statements are given in this report on some of the important results of research during the past year. More detailed information on some of these and other research activities of the Bureau may be found in the 290 publications issued and in the specifications of the 27 public-service patents granted to employees. A list of publications and patents is available in mimeographed form.

Three outstanding research accomplishments of the Bureau, two of which were incident to World War II, received official recognition by the scientific world this year.

In November 1946 the Northern Regional Laboratory was the recipient of one of the four awards of the Albert and Mary Lasker Foundation for research in medicine and public health administration made annually to individuals or groups. The award, compris-

ing a silver statuette of Winged Victory (symbolic of achievement), was accompanied by the following citation:

Though volumes have been written regarding penicillin as a boon to mankind and though wide recognition has been given to its discoverers, the American Public Health Association here cites the Northern Regional Laboratory for its work in translating the pilot-plant work done by others into mass production.

On March 20, 1947, Dr. George W. Irving, Jr., head of the Biologically Active Compounds Division which cooperates with the Bureau of Plant Industry, Soils, and Agricultural Engineering in fundamental research on chemical reactions in plants, received the 1946 Washington Academy of Science award for outstanding achievement in the field of the physical sciences in recognition of his service in biochemistry and especially for his work on antibiotics in tomato plants.

On May 7, 1947, Dr. Henry Stevens and Dr. E. J. Coulson of the Allergens Research Division were presented with Certificates of Appreciation from the Medical Department of the United States Army, signed by the Surgeon General for the Secretary of War, for nearly 3 years of secret and very urgent work on allergic reactions resulting from the use of certain types of vaccines, which was undertaken by the Department of Agriculture at the request of the Medical Department of the Army.

There were more than the usual number of personnel changes in key positions of the Bureau's organization during the past fiscal year. Dr. T. L. Swenson, formerly Director of the Western Regional Laboratory, resigned from his position as Special Assistant to the Chief to accept a position with a private research organization. Dr. Lee T. Smith, formerly head of the Carbohydrate Division of the Eastern Regional Laboratory, Dr. A. F. Langlykke, formerly head of the Fermentation Division of the Northern Regional Laboratory, and I. E. Knapp, formerly head of the Naval Stores Research Division, resigned to accept positions in private industry. As in previous instances, the value of the experience gained in various phases of the Bureau's research work and in the direct supervision of such work was recognized by a private research organization and three industrial firms that could offer these men more attractive compensation than they could expect to receive in their Government positions. Dr. H. D. Lightbody, formerly head of the Biochemical Division of the Western Regional Laboratory, was transferred at his request to the Quartermaster Corps of the War Department.

Vacancies in key positions were filled by promotions from other positions within the Bureau's organization. Dr. Michael J. Copley was appointed Director of the Western Regional Laboratory. Dr. B. A. Brice succeeded Dr. Copley as head of the Analytical and Physical Chemistry Division of the Eastern Regional Laboratory. Dr. Charles H. Fisher became head of the Carbohydrate Division of the Eastern Regional Laboratory, and E. L. Patton, formerly a chemical engineer in the Naval Stores Station, became head of the Naval Stores Research Division. At the end of the year the positions as head of the Fermentation Division of the Northern Regional Laboratory and head of the Biochemical Division of the Western Regional Laboratory were still vacant.

Dr. Charles A. Browne, formerly Chief of the Bureau of Chemistry, and a collaborator in this Bureau since his retirement from the position of Supervisor of Chemical Research in the Bureau of Agricultural Chemistry and Engineering in 1940, died on February 3, 1947.

The administrative officers of the Bureau and heads of its research laboratories and divisions, as of June 30, 1947, are listed at the end of this Report.

CONTINUED RESEARCH ON COTTON NEEDED TO MEET INCREASING COMPETITION

During the past year the Southern Regional Research Laboratory has brought to a conclusion several technological surveys on cotton as its part in a broad program of research on postwar agricultural and economic problems of the Cotton Belt, conducted cooperatively by several Government agencies and other organizations and sponsored by the House Committee on Agriculture. This program, the most comprehensive effort yet made to bring together information relating to the problems of cotton and southern agriculture, has supplied data of value in the guidance of research to increase the utilization of cotton and in connection with cotton merchandising activities and national cotton policy.

The Southern Regional Laboratory participated in 3 of the 10 projects of the program. These required the accumulation of data on: (1) Cotton goods production and distribution, techniques, costs, and margins; (2) the competitive position of cotton by major end-use markets; and (3) production of synthetic fibers and paper. Specifically, studies were made on: The possibilities of reducing costs and improving quality in the chemical finishing of cotton goods; cotton's competitive position for use in bags, tires, cordage, and twine; and rayon's position as a competitor of cotton in various uses. In addition, the Southern Laboratory helped to prepare over-all summaries of these 3 projects, combining a report of its findings with those of the other agencies involved.

The survey on cotton's competitive position for use in bags showed that consumption of cotton for bags climbed to 820,000 bales in 1943, owing largely to increase in wartime packaging requirements accompanied by curtailment of imports of burlap from India. With burlap once more available and the use of paper increasing, the consumption of cotton for bags dropped to 440,000 bales in 1946. Although cotton will face continued competition from burlap and growing competition from paper for use in bags, bags are expected to represent the largest single end use of cotton in 1950, with an estimated consumption of 575,000 bales under highly prosperous conditions and 536,000 bales under conditions of moderate prosperity. These estimates are on the basis of an assumed price of 25 cents per pound for cotton.

The integrated reports of the cooperative program showed: (1) That manufacturing processes alone account for 49 percent, and wholesalers' and retailers' margins alone average about 40 percent of the consumer's dollar paid for cotton clothing and household products; (2) that a total of 7.7 million bales of cotton will be consumed in the United States in 1950 if cotton is priced at 25 cents per pound and business conditions are moderately good; and (3) that cotton has lost important end-use markets during the last 15 years to paper and

rayon and faces the prospect of intensified competition from these materials as well as from other natural and synthetic fibers.

A poll of technical opinion indicated that, on the basis of present trends, by 1955 rayon will have reached parity with cotton in quality, with cotton's advantages in some properties offset by rayon's advantages in others. It was concluded that if consumption of cotton is to be maintained at or near present levels, cotton must have a competitive price and be supported by a more intensified merchandising program and continued research on adapting cotton to specific uses.

COTTON FABRICS TO BE MADE MORE WEATHER- AND ROT-RESISTANT

In connection with research at the Southern Regional Research Laboratory on methods of preserving cotton fabrics for industrial and farm uses, the results of 5 years' exposure tests with treated and untreated cotton fabrics have been assembled and interpreted. From the relationship found to exist between breaking strength and cuprammonium fluidity, it was possible to establish patterns of weathering degradation that will be useful in the formulation of protective finishes for cotton in out-of-door uses. Chemical compounds and mixtures that seem promising as fungicides, water repellents, and utraviolet-screening materials are being tested as finishes.

The degrading action of sunlight on cotton fabrics, uncomplicated by the other destructive factors always present in natural weathering, has been studied by means of the rotating solar exposure cabinet, mentioned in the report for 1946, which excludes rain, wind, dust, and micro-organisms and controls the temperature and humidity of the samples. Exposure of treated and untreated samples behind six glass windows, each one having a different color and therefore transmitting a different fraction of sunlight, makes it possible to determine the effects of light from various parts of the solar spectrum. The rotation of the cabinet so as to follow the apparent motion of the sun keeps the samples and their glass covers perpendicular to the sun's rays and accelerates the degrading effects. To have a basis for comparison, samples are also exposed outside the cabinet to total weather during the same period of time. Data have been collected on the radiant energy incident upon the samples and on the chemical and physical evidence of deterioration in portions of identical fabrics exposed to the energy from five different spectral regions as well as from total solar radiation.

It appeared that radiant energy from some parts of the sun's spectrum was more damaging than that from other spectral regions, and there was some evidence to substantiate the theory that rays of certain wave lengths from different regions in the spectrum may interact in such a manner as to reduce the net degrading effect of their combined incident energy. It was concluded that sunlight accounts for practically all of the degrading effects on cotton fabrics caused by weather exposure in the absence of pronounced microbiological attack or of damage by corrosive fumes.

The carbon arc as an artificial light source has been employed in experiments to develop techniques for rapid evaluation of treatments designed to improve the weather resistance of cotton fabrics. A num-

ber of modifications have been applied to a commercial carbon-arc "weathering" machine to make accelerated tests more comparable with natural conditions. It is hoped to establish enough correlation to permit interpretation of the results of accelerated tests in terms of the degradation suffered by cotton fabrics during exposure to weather—at least to the extent of permitting evaluation of the relative merits of various finishes.

In continued pilot-plant research on the partial acetylation of cotton goods as the best means known for protecting against rot and mildew, additional lots of experimental cloth were made and sent to various organizations for service tests in different end uses. Partially acetylated cotton has proved to be the best material yet found for bags subject to rotting in domestic water-softening systems. The chief interest in the treated cotton at present comes from the fishing and plastic industries. Service tests of acetylated seine yarn and fish nets have given favorable results, and one manufacturer has requested the acetylation of a large quantity of gill netting with the necessary twine and cord. The good resistance to heat and electricity which acetylated cotton possesses makes it suitable for use in the electrical insulation and lamination fields. Work is in progress on controlling the numerous variables of the process so as to obtain a more uniform product.

PROGRESS MADE IN DEVELOPMENT OF SELF-SEALING COTTON FABRICS

Research on the improvement of cotton fabrics that resist the passage of water through swelling of the constituent fibers when wet was continued by the Southern Regional Research Laboratory in cooperation with the Office of the Quartermaster General and other agencies. The granting of a public service patent on a process for increasing the water resistance of cotton fabrics by addition of a swellable material to the yarn prior to weaving was previously reported. During the past year different cottons were studied with regard to swelling properties in order to determine if any type of cotton might be more suitable than others for the manufacture of self-sealing fabrics suitable for outer garments, tents, and tarpaulins.

It was demonstrated that cottons differ in their ability to close the minute spaces that exist in tighly woven fabrics. A tightly woven fabric made from a thin-walled (immature) cotton is more resistant to penetration by water than a similar fabric made from a thick-walled (mature) cotton. Thus, cotton that would be considered inferior according to the usual standards might prove to be superior for this use. This advantage promises a more profitable market for the late-season cottons.

Conventional cotton finishing processes have a tendency to diminish the swelling capacity of the cotton in piece goods and, for this reason, to lower the closing capacity of the finished fabrics, in addition to having adverse mechanical effects. Therefore attempts are being made to reduce finishing to a minimum as well as to adapt more satisfactorily the essential finishing operations to the requirements of the self-sealing-by-swelling type of cotton fabrics.

DYE METHOD USED FOR SELECTING COTTONS OF DESIRED MATURITY

Chemists at the Southern Regional Research Laboratory have devised a technique of differential dyeing for determining certain important qualities of cotton. It distinguishes between thin-walled and thick-walled cotton by utilizing the different dyeing characteristics of these two types of fibers. The test is easily made in a laboratory or dye house. A sample of cotton is dyed in a bath containing a mixture of two specially selected dyes, one a direct red and the other a direct green. On removal from the dye bath, thick-walled (mature) fibers will be red, and thin-walled (immature) fibers will be green.

Good qualitative correlation between proportion of mature fibers and color has been observed in samples containing from 78 to only 22 percent of mature fibers. When fully perfected, the new dyeing method should facilitate the estimation of average maturity by the total color effect. The degree of maturity of cotton influences its properties, and this technique, which permits recognition of immature fibers in admixture with mature fibers, promises a number of applications to the study of cotton at all stages from raw fiber to goods in service.

This new research tool can be used to follow the development of fibers during growth as an aid in selecting cotton of better quality. It is already serving in the selection of fibers, according to their properties, for use in fundamental research. Its application has solved serious mill difficulties in both manufacturing and dyeing; and the method is sufficiently promising that several mill organizations are trying it as an aid in the selection of cotton for manufacturing.

IMPROVED DEVICE DEVELOPED FOR STRETCHING COTTON TIRE CORD

In further efforts to develop an improved type of cotton tire cord the Southern Regional Research Laboratory studied means for increasing uniformity, strength, heat resistance, and fatigue resistance without loss of essential elastic properties of the cord.

An improved stretching device was developed for cotton tire cord. It can be used in connection with existing commercial winding machinery and operated at a high production rate. A comparison of cords stretched on this new device with similar cords stretched on other types of commercial stretchers showed that it produces a much more uniform cord with respect to elongation and strength properties. One of the chief features of this device is that it permits the application of a constant tension with variable speed. Two tire-cord manufacturers have indicated a desire to build similar units. An application for a public service patent is pending.

Road tests are still in progress on three groups of 9.00–20, S6, 10–ply cotton-cord tires made with (1) a commercial regular production cotton cord, (2) cord produced from Wilds 13 cotton according to the regular commercial process, and (3) cord produced from Wilds 13 cotton according to the dual-stretching process developed at the Southern Regional Laboratory. A similar set of rayon-cord tires is being run along with the cotton tires to obtain comparable data. On indoor wheel tests run by the National Bureau of Standards, regular commercial cotton-cord tires gave the lowest mileage, the rayon-cord

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tires gave the highest, and the two groups of experimental cotton-cord tires gave intermediate mileages.

The ply-building machine constructed on the basis of information obtained from a tire-manufacturing company was improved in several respects. The application of, and compression of the cord into, the skim-stock has been appreciably speeded up. A number of difficulties were encountered at first in adjusting the machine and in working out a satisfactory operating technique, but these have been overcome, and the machine is now ready for regular experimental use. Plans and drawings of this improved machine were furnished upon request to a number of tire manufacturers.

Extended research on the chemical degradation of cellulose of cotton tire cord under the influence of heat, as indicated by cuprammonium fluidity measurements and strength, showed that the rubber in which tire cord is embedded exerts a protective action on both cotton and rayon cords against degradation, presumably owing to a reduction in the amount of atmospheric oxidation. Dipping the cords in rubber latex to improve their adhesion did not provide any protection, and calendering the fabric coated with skim-stock provided only a little protection.

The effects of moisture and temperature on stress relaxation of stretched cotton and 2200/2 rayon cords suggested that fiber breakage occurs by a mechanism involving slippage of the cellulose molecules rather than the rupture of primary valences.

COLOR OF COTTONSEED PRODUCTS DEPENDS ON PIGMENT CONTENT AND ON CONDITIONS OF STORAGE AND PROCESSING

The nature and control of color in cottonseed meal and oil received further attention at the Southern Regional Research Laboratory because color is an important factor in determining the suitability of cottonseed products for different uses. The two major pigments of cottonseed kernels, gossypol and gossypurpurin, were found to occur only in distinct glands and to constitute essentially all the pigments of these glands. The pale-yellow pigments occurring outside the glands are similar to gossypol in color but appear to be quite different chemically.

In an investigation of the relation between conditions of processing cottonseed and the pigmentation of the expressed oil, it was found that oils produced from seed precooked in steam-jacketed pans without addition of moisture were more highly colored than the corresponding oils produced from wet-cooked seed, and that the principal pigments in oils produced by the two methods are entirely different.

Conditions of storing cottonseed and cottonseed oil were also found to influence the degree of pigmentation in the oil. Criteria were established for judging which oils can be stored safely and which should be refined immediately. The rate of increase of color that can only be removed by bleaching was greatest in oils obtained from stored seed cooked in steam-jacketed pans without addition of moisture and least in oils from fresh seed cooked similarly, but with addition of moisture. The increase of such color during storage of either the seed or crude oil is dependent on the amount of pigment material originally present in the seed and on the temperature during storage.

Color can be minimized by maintaining low temperature during storage of the seed and by storage of the seed rather than the crude oils.

REMOVAL OF PIGMENT GLANDS MAKES COTTONSEED MEAL A BETTER FEED

A more nutritious feed is the verdict of the laboratories that have tested a new type of cottonseed meal produced by a fractionation method described briefly in last year's report. This meal, obtained in process-development research at the Southern Regional Laboratory, is essentially free of pigment glands, which distinguishes it from cottonseed meals obtained by the usual solvent or compression methods. The pigment glands, which comprise 3 to 4 percent of the meats, are recovered as a separate fraction. Thus, cottonseed is separated into three parts—oil, pigment glands, and substantially gland-free meal—instead of just oil and meal, which are the products obtained by present commercial methods. In the process-development research during the past year, about 800 pounds of the gland-free meal and 55 pounds of pigment glands were obtained. This limited supply of meal and glands was shared with 11 industrial, university, State, and Federal laboratories for cooperative research on the nutritional and pharmacological properties of these products.

Cottonseed meal obtained by the present commercial methods is of limited usefulness as a source of protein in poultry and swine feeds, because it has a toxic effect when fed too freely. But with the new type of cottonseed meal as the only source of vegetable protein in the diet, chicks made excellent growth, and after 6 weeks they were in excellent physical condition and weighed above the average, according to reports from one cooperating laboratory.

In another laboratory feeding experiments, starting with 2-week-old chicks, were carried on for 4 weeks. The chicks showed the same superior growth by being fed with substantially gland-free cottonseed meal as the sole source of protein without adding an animal-growth factor to the diet. The hatchability of eggs laid by hens fed this meal as the sole source of protein and without addition of the animal-growth factor was also superior. Two other laboratories reported that during storage of eggs from hens fed the new type of cottonseed meal the whites did not turn pink and very few yolks became discolored. These results are particularly significant to the poultry industry, because the growth factor for chicks has previously been found only in fish solubles, high-grade meat scraps, and other feeds that have been in short supply, and also because the inclusion of ordinary cottonseed meal in the diet of laying hens causes undesirable egg-yolk discoloration.

Limitations on the use of cottonseed meal in feed have often been attributed to the presence of gossypol, but recent experiments have shown that gossypol is not the principal factor. Chemical and pharmacological research is being continued to determine the nature of the physiologically active constituent of pigment glands whose presence in cottonseed meal lowers the nutritive value.

The scale of operations in the chemical engineering research on development of the gland-separation process for cottonseed has not only produced sufficient pigment glands and substantially gland-free

meal for nutritional and pharmacological investigations, but has also provided valuable engineering data for practical application of the process. Further development in a pilot plant is underway to determine the feasibility of the process for commercial adoption.

BETTER PEANUT MEAL FOR INDUSTRIAL USES OBTAINED BY SOLVENT EXTRACTION

The object of research at the Southern Regional Research Laboratory on the solvent extraction of peanuts is to produce a peanut meal of low oil and solvent content without damage to the quality of the protein so as to get a better raw material for the production of protein fibers and adhesives. During the past year the chief interest was in the production of enough peanut meal of low fat content to provide the protein needed for research in the Laboratory and by cooperating industrial concerns.

Experiments were made with both batch and continuous processes of solvent extraction. A portable, batch, single-cell, solvent-extraction unit, with auxiliary equipment for oil and solvent recovery, was used with commercial hexane as the solvent and yielded a meal containing as little as 0.5 percent residual oil. This batch extractor had a holding capacity of 120 pounds of peanut flakes. A continuous solvent-extraction unit, constructed for use with either peanut or cottonseed flakes was also used with commercial hexane as solvent. It comprised a two-stage screw-type extractor, meal dryer and cooler, miscella filters, and auxiliary oil and solvent recovery equipment.

This unit processed up to 150 pounds of flaked peanut kernels per hour, and the oil and solvent-recovery equipment could be operated simultaneously with the extraction and drying equipment. About 38 batches of flaked peanut kernels and partially defatted meal, totaling nearly 3,700 pounds, were extracted in the portable, batch, solvent-extraction unit, and nearly 9,000 pounds of flaked peanut kernels were processed in the continuous solvent-extraction unit.

In the experiments with both processes for extracting peanuts there were three problems to be solved in addition to the attainment of efficiency in the extraction equipment. These were: (1) Prevention of a reddish color in the meal from the pigment of the seed skins; (2) preparation of physically stable flakes that would not crumble or powder during extraction or solvent removal; and (3) complete recovery of solvent from the extracted meal without damage to the protein.

To meet the first of these problems the Southern Regional Laboratory developed a modified blanching process in which mild heat and an air blast were used for removing the skins from the shelled peanuts before they were flaked for extraction. The process did not adversely affect the protein. Formerly the problem was met by chemical treatment of the shelled peanuts to remove the pigments from the skins, but this left the meal diluted with the skins.

In attempts to solve the second problem, it was found that a fairly resistant flake can be obtained by adjusting the moisture content of the shelled peanuts and then heating before flaking. An increased flake thickness gave more resistance to mechanical strain at all moisture contents.

The third problem, that of complete solvent recovery without damage to the protein, was still under investigation, and additional continuous drying units were being procured for that purpose.

It was originally planned at the Southern Regional Laboratory to develop a solvent-extraction process and equipment especially for cottonseed; but, because of the marked industrial interest in peanut protein for fiber production, all of the earlier runs were made on peanuts. Experiments on cottonseed extraction were started near the end of the year. Although none of the experimental runs was wholly satisfactory, usable quantities of meal and oil of satisfactory quality were produced, and the tests yielded valuable engineering information. It appears that the material-handling problems in solvent extraction of peanuts are more serious than those in cottonseed extraction, and their solution may require considerably more time for research and engineering development.

INDUSTRIAL INTEREST SHOWN IN PEANUT PROTEIN

During the year sizable quantities of peanut materials—kernels, flaked kernels, skins, meals, and extracted protein—were prepared and furnished to cooperating industrial firms that are interested in the use of the protein as a raw material in manufacturing processes. There is a fast-growing interest in the production of peanut-protein fiber, for apparel fabrics. At least three companies have formulated plans to engage in one or more of the operations required to obtain synthetic fiber from shelled peanuts. These operations include removal of the coloring matter in the skins prior to processing, or removal of the skins, processing by solvent extraction to obtain a meal of low oil content, separation of a purified protein from the meal, and spinning of synthetic fibers from the protein by wet processes similar to those employed in the viscose-rayon industry.

Research in the Southern Regional Laboratory points to the possibility of using peanut protein in the manufacture of industrial products other than fibers. For example, a process has been developed for modifying peanut protein to make it suitable for use in paper-coating compositions. The modified protein can be incorporated with paper-coating minerals by means of a special formula. A number of producers and consumers to whom samples of the protein and copies of the coating formula were sent have returned favorable reports. The preliminary tests have indicated that peanut protein has adhesive properties superior to some proteins now employed in paper-coating mixtures. A cold water paint utilizing the adhesive property of peanut protein has been produced experimentally in small quantities. This paint did not have a disagreeable odor and performed well in laboratory tests. The mixed paint had good flowing, brushing, and spreading qualities. It set to touch in 2 hours and dried within 6 hours at room temperature.

A survey was made of the status in the textile industry of various types of synthetic fibers made from proteins, with particular reference to the possible development of fiber from peanut protein and the problems of its manufacture and production. A comparison of the potentialities of various proteins for fiber production showed that the future relative position of the protein-base fibers, now at a stage

of development comparable to that of rayon 30 years ago, depends as much upon economic as upon technical considerations. The major technical problem is to obtain a meal satisfactory for the industrial extraction of protein, and the principal economic consideration is to have adequate sources of raw material. Casein is the present exclusive commercal source, but it is likely that soybean and peanut proteins will also soon be in the field.

Proteins available for the manufacture of fibers have this feature in common—they are obtainable only as byproducts, and the potential supply of any one of them is thus largely limited by demand for the principal product. The cake and meal byproducts of oilseed processing have protein contents ranging from 35 to about 60 percent, the higher percentage being for the peanut products. During the past few years, the average production of soybean cake and meal has been about 3½ million tons, of cottonseed cake and meal slightly less than 2 million tons, and of linseed cake and meal about 800,000 tons, the average total production from these three sources being about 6¼ million tons. The annual production of peanut cake and meal, obtained as byproducts from the crushing of about 200,000 tons of peanuts annually for oil, has averaged about 100,000 tons. With additional sources of industrial protein becoming commercially available, their relative consumption for the production of artificial fibers will depend more and more upon price as well as on suitability and technological developments.

SOYBEAN PROTEIN USED AS ADHESIVE FOR SHOTGUN-SHELL CASINGS

For the last 10 years chemists in this Bureau have studied the preparation and utilization of soybean protein and have developed much new and important information on this subject. Recent industrial interest in soybean protein is partly a result of past research efforts which have contributed substantially to the increased industrial utilization of soybean protein. One phase of current studies on soybeans at the Northern Regional Research Laboratory relates to the adhesive properties of soybean protein.

In 1946 a large firm manufacturing shotgun shells was considering new sources of adhesive for their casings because the price of casein, their usual source, was high, and the supply limited. After many experiments the company concluded that most synthetic materials were unsuited to their operations and product. It then turned to natural materials. Since soybean protein was known to be similar in many respects to casein and was the subject of research at the Northern Regional Laboratory, this Bureau was appealed to for assistance. As a result, the Laboratory conducted a number of experiments and finally found several formulations of soybean-protein adhesive that might be suitable. In cooperation with the Laboratory, this company conducted full-scale plant trials and made numerous experimental shells for testing. As the result of shooting tests these shells were reported to be equal or superior to those previously manufactured. Actual commercial use of soybean protein for shotgun-shell casings was initiated early in 1947.

CASEIN BRISTLES NOW MADE BY CONTINUOUS METHOD

Recent annual reports of this Bureau have described the progressive improvement of procedures for making casein bristles. Thus far, however, all processes have involved some interruption between the start and finish of the production operations. The development of a completely continuous method has now been accomplished, so that the filaments automatically move from the points of extrusion to the final collecting reel.

The continuous process involves the extrusion of filaments into water at 40° C. containing clay to prevent sticking, hardening the filaments with quinone at 45° C. on a thread-advancing reel for 18 minutes, and then washing and drying the filaments while they are advancing. The equipment required to effect these operations is simple, and the amount of labor required in the production of the filaments is small. The ease of operating the equipment, as well as the yield and quality of the bristle produced, has justified the effort spent in developing the continuous method. The weight of usable bristle obtained is practically the same as the weight of casein used. The tensile strength of the bristle produced by the continuous method is the same as that of the bristle made by the batch method, but the bristle made by the continuous method is more uniform and flexible. One commercial firm has erected a factory for applying this process and has been making pilot-plant runs on the production of bristles and paint brushes. If the brushes prove to be satisfactory in service, the company will put them on the market as soon as all the manufacturing kinks have been ironed out.

Besides developing a continuous method for making casein bristles the Eastern Regional Laboratory has reinvestigated the whole question of bristle quality and uniformity, as well as the problem of brush fabrication. It is of interest that an unused specimen brush constructed in 1942 shows no deterioration in the casein bristle. Moreover, brushes made from the current output of casein bristle are of better quality than the early brushes, owing mainly to greater strength and uniformity of the fiber. Considerable attention has been given to the different bristle-imbedding agents such as styrene-polyester, Norelac, and other commercial synthetic resins. The properties desired in the setting material are rapid setting at low temperature, inertness toward the bristle, and resistance to paint oils and thinners. Data on processes and products are available to any company seriously interested in the manufacture of casein bristles.

DEFINITE ADVANCES MADE IN CASEIN FIBER

Casein textile fiber has been on the market in the United States for about five years; it is now established as a commercial fiber, and no longer is regarded as a laboratory curiosity or pilot-plant possibility. Its success shows the tremendous future in store for this man-made protein fiber if its present weaknesses in tensile strength and water resistance can be overcome.

For several years the Eastern Regional Research Laboratory has been working intensively on this problem, and considerable success has been achieved. It has produced, on a laboratory scale, a casein fiber

that is almost twice as strong, both wet and dry, as the commercial fiber. Furthermore, the Eastern Regional Laboratory investigated various procedures for their effectiveness in making the fiber resistant to the boiling dye bath and made some progress in obtaining this property together with higher strength. These results, which came from a careful, thorough study of each of the many steps involved in the process, as well as the basic information obtained in the investigation should be of great value to the textile industry.

Another significant development was the successful spinning of the improved casein fiber into a silk-like yarn composed of continuous filaments. Such a yarn would appear to have a large potential field of use in knitted goods—underwear, socks, and the like—and in sheer woven fabrics. Silk was formerly used for such products, but in recent years silk has been almost entirely replaced by rayon and nylon.

Because of the importance of water absorption by casein fibers, a fundamental study of the causes was undertaken. Work done during the year showed that one-fourth of the water taken up is held by the amino groups of casein, and that the absorbing effect of these groups can be altered or even completely eliminated by suitable chemical modification. Study of the other absorbing centers in the casein molecule was under way at the end of the year.

TWO FRACTIONS OF CASEIN DIFFER IN AMINO ACID CONTENTS

In the annual report for 1945 it was announced that the Eastern Regional Research Laboratory had succeeded in isolating for the first time two fractions, alpha-casein and beta-casein, from the industrially important protein casein. The isolation of these two fractions provided an unusual opportunity for obtaining useful information about this widely utilized protein by determining the composition of its components. Extensive amino acid analyses, including chemical, enzymatic, and micro-biological techniques of assay, were made on casein and on its isolated fractions. The data obtained thus far indicate that lysine, arginine, tyrosine, tryptophane, and cystine are present in considerably smaller amounts in beta-casein than in the alpha fraction; that serine and methionine, on the other hand, are present in higher content in the beta component; and that histidine and threonine occur in equal concentration in both components.

The alpha-casein and beta-casein used in the analyses were carefully fractionated in the course of their preparation, so neither fraction contained any of the other. Different preparations of the same fraction might be expected, therefore, to contain reasonably constant proportions of constituent amino acids. Concordant values, which provide evidence for this constancy of amino acid composition in the individual casein fractions, were obtained by analyses of two distinct sets of preparations for three amino acids, as well as for amino nitrogen and phosphorus.

At the end of the year further analyses were in progress, and it was anticipated that when the complete amino acid compositions of alpha-casein and beta-casein had been elucidated, exploitation of the useful properties of these proteins, as well as those of unfractionated casein, would be facilitated.

PROGRESS MADE IN SEPARATION OF MILK-WHEY PROTEINS

Milk whey contains a complex mixture of proteins that are similar to blood-serum proteins and may eventually be found to be similarly useful for special medical uses.

In order to purify the protein components of whey and to determine their properties, large-scale fractionations were made by several methods at the Eastern Regional Research Laboratory. These fractionations resulted in the separation of several of the protein components in relatively pure form. The gamma globulin of normal whey was separated and shown to be different from the immune gamma globulin of first milk (colostrum). A crystalline protein with properties differing from those of the previously described crystalline beta lactoglobulin was isolated. By electrophoresis (migration of particles in an electric field) this new protein was shown to have two components, while by the same means beta lactoglobulin was shown to have three components. The separation of the components of these crystalline proteins will be of general importance, since this protein is used as a standard protein by protein chemists.

STRAW IS POSSIBLE SOURCE OF NEEDED STRUCTURAL MATERIALS

The insulating-board industry is rapidly expanding its manufacturing facilities above its rated capacity of almost 1 billion square feet per year. The products replace scarce lumber and steel for such uses as sheathing, plaster base, interior finish, roof insulation and imitation brick siding in dwellings and other buildings. Although wood is used as a raw material by many manufacturers of insulating board, the industry was largely pioneered by products made from sugarcane bagasse, which is now used to the extent of about 200,000 tons per year.

Bagasse has proved to be an outstanding raw material. It is possible to produce longer and tougher fibers from bagasse than can be produced by processing wood. These bagasse fibers, properly used, produce insulating board having an impact strength two or three times that of similar board made from wood fibers. High impact strength is associated with ruggedness, an important property in a building material. Straw and cornstalks also have been used industrially on a small scale for producing structural insulating materials.

Very little technical information has been published on the manufacture of insulating board. Because of current housing shortages and lack of sufficient building materials, it is desirable to have such information available to persons contemplating such manufacture.

A study undertaken some time ago by the Northern Regional Research Laboratory to determine the fundamental relationships of fiber size and kind to properties of finished board now shows that wheat straw is equal, or in some ways superior, to sugarcane bagasse for producing insulating boards of exceptional merit. From this study very practical information has been obtained for the manufacture of a line of fiber-board products having a wide range of densities and strengths. The Laboratory is prepared to give manufacturing information to those able to undertake the manufacturing operations. Serious consideration is being given by industry to the use of wheat straw for this purpose.

765067—47——3

At present the strawboard industry uses about 800,000 tons of straw annually. Last year about 70 million tons of wheat straw were produced, of which about 3 million tons were baled and not more than 15 million tons could be accounted for as having been hauled for commercial or farm use. A considerable amount was burned, and the remainder was left in the fields to be used as a mulch or was plowed into the soil.

The wire-bound-box industry uses about 1.5 billion square feet of low-grade wood veneer annually, but the cost of making this veneer has increased rapidly, and its producers are seeking more profitable markets. Therefore the Northern Regional Laboratory cooperated with the wire-bound-box industry during the past fiscal year in studying the possibility of making a satisfactory board from wheat straw. A conclusion has not been reached as yet, but the industry offered encouragement and showed a willingness to change the box design to aid in solving the problem. The progress made thus far is largely due to the information developed in the fundamental fiber studies. The consumption of straw by this industry would be about 1 million tons per year if straw board suitable for its product can be produced cheaply.

PLANT FOR SYNTHETIC LIQUID FUELS INVESTIGATIONS NOW IN OPERATION

The semiworks plant of the Synthetic Liquid Fuels Project, on the grounds of the Northern Regional Research Laboratory, began operations in November 1946. The work under this project is a contribution by the Department of Agriculture to the broad national program for studies on the production of synthetic liquid fuels from nonpetroleum sources, authorized by the Seventy-eighth Congress under Public Law 290, April 5, 1944. It is planned under this project to study the production of sugars from agricultural residues on a semiworks scale, so that complete cost and design data for a full-scale plant may be developed. Cooperative studies on the fermentation of these sugars to liquid fuels and on the evaluation of the experimental fuels are being conducted by the Northern Regional Laboratory.

The process under investigation by the Synthetic Liquid Fuels Project has two steps. In the first step the pentosans of agricultural residues are hydrolyzed to pentoses with dilute sulfuric acid; in the second step the cellulose in the residue is hydrolyzed to dextrose by a concentrated sulfuric acid method. About 60 runs were made in the semiworks plant, and each run involved the use of about 5,000 pounds of corncobs. The hydrolysis data from the plant indicated that about 94 percent of the pentosans in corncobs can be hydrolyzed to conversion products, 87 percent being converted to pentose sugars and 7 percent to furfural, with the simultaneous hydrolysis of only 1 to 2 percent of the cellulose.

After the first step of the process was developed to give satisfactory yields of products, studies on the operation of the equipment in the second step of the process were begun. It was planned to operate the whole plant as a unit after June 30, 1947.

The fermentologists of the Northern Regional Laboratory were successful in their attempts to ferment the pentose sugar solutions produced in the semiworks plant to butanol, acetone, and ethyl alcohol,

which are useful in blended liquid fuels. They developed fermentation techniques that give laboratory yields of such products almost equivalent to commercial yields by the fermentation of molasses. It was planned to expand this work into a series of pilot-plant-scale fermentations in which 8,000 gallons of pentose sugar solution per week would be used.

Instead of being fermented, the pentose sugars may be converted by chemical treatment to furfural, which in turn may be converted to various chemicals having fuel value. Toward the end of the year a small pilot-plant unit for studies on the production of furfural was assembled in the semiworks plant. Sufficient data are not yet available to warrant significant conclusions.

Chemists in the Northern Regional Laboratory converted 1-gallon lots of furfural to products having fuel value. These products, together with butanol, acetone, and ethyl alcohol, are being evaluated as motor fuels by the motor-fuels-testing section of the Laboratory.

The coordinated liquid fuels program of the Synthetic Liquid Fuels Project and the Northern Regional Laboratory is expected to permit a complete evaluation of the production of synthetic liquid fuels from agricultural residues.

FURTHER APPLICATION MADE OF BUREAU'S PROCESS FOR CLEANING PINE GUM

During the past fiscal year five new plants, in addition to those previously reported, undertook application of the Bureau's gum-cleaning process as a means of insuring clear rosin from a uniformly cleaned turpentine gum. The staff of the Naval Stores Station at Olustee, Fla., cooperated with commercial firms in the installation and initial operation of these new plants and in training inexperienced operators. Advice was given to turpentine operators by Station representatives on timber selection, cup materials, and improved methods for hanging turpentine cups. Advice was also given to processors on plant operations and to many gum farmers on woods work.

The advantages of the Bureau's gum-cleaning process include separation of the gum from chips, straw, and finer solid material by pressure filtration and subsequent recovery of all gum from the waste material by washing with steam and turpentine. A final removal of the water-soluble impurities from the turpentine-diluted gum is effected by washing with water. Thus the rosin obtained is thoroughly clean and brilliant.

The iron stain that gets into the gum from rusty turpentine cups and discolors the resulting rosin may be removed by the proper use of oxalic acid in the washing process, as described in a public-service patent previously issued to a member of the Naval Stores Research Division. When the iron stain is dissolved and removed as oxalates, a clean and iron-free gum is obtained from which a paler and clearer rosin can be made. The quality of the rosin can be raised as much as four grades, depending upon the age and original grade of the gum.

Hundreds of gum farmers and naval stores producers who formerly distilled their turpentine gum with the primitive fire still are now selling the gum to improved central gum-cleaning and distilling plants. This has resulted in increased income to the gum farmers, and likewise is advantageous to the processor. Of the total 31 central process-

ing plants now operating in the naval stores industry, 20 are using the Bureau's gum-cleaning process.

ROSIN AND TURPENTINE MADE ECONOMICALLY BY NEW CONTINUOUS PROCESS FOR DISTILLING PINE GUM

Successful demonstrations of the continuous flash-distillation process for turpentine gum, developed by engineers of the Naval Stores Station, at Olustee, Fla., were made December 3 and 4, 1946, at the nearby plant of a cooperating naval stores producer at Lake City. More than 100 visitors attended, including processors of pine gum and manufacturers of steam distilled wood turpentine, chemists, chemical engineers, foresters, gum farmers, and pulpwood operators. Because of its adaptability, the flash-distillation process, in conjunction with the Bureau's gum-cleaning process, is useful for obtaining a dry rosin, a rosin containing any desired percentage of turpentine, a cleaned gum of high turpentine dilution, or a cleaned gum of normal turpentine content.

Applications of this process are not confined to the naval stores industry. It may be applied, for example, in the manufacture of ester gum (rosin glyceride) and similar products whose quality depends upon the effective removal of solvents having low boiling points. The throughput of the present continuous flash still, having a column 8 inches in diameter, is greater than that of the regular fire still, but does not equal that of the batch-type steam still; therefore a larger continuous unit is under construction.

The economic advantages of the flash-distillation process were especially noted by gum processors and engineers. The fact that one man can do the work formerly done by the stiller and a laborer engaged in pouring or ladling the rosin is an accomplishment in labor saving. The simplicity of operation, ease of control, and saving in first cost of the continuous still are remarkable. The object of further work on the continuous flash-distillation process is to design a still column that is equally saving in steam consumption but of sufficiently large diameter (possibly 12 inches) to insure a throughput greater than that of the present batch steam still.

GUMS FROM LONGLEAF AND SLASH PINES DIFFER IN VISCOSITY

In order to aid engineers in designing equipment for cleaning, handling, and processing pine oleoresins, further data were obtained on the viscosities of longleaf and slash pine gums diluted with turpentine in the temperature range of 20° to 80° C. and at turpentine concentrations of about 20, 25, 30, and 40 percents by weight. There were distinct differences in the viscosities of gums from the two species of pine that serve as sources of turpentine and rosin in this country. This fact should be of interest to consumers of gum thus, imitation Burgundy pitch, and other special naval stores products.

NEW CHEMICALS AND RUBBER DERIVED FROM TERPENE HYDROCARBONS

Further studies were made on the recently discovered addition reaction between beta-pinene (the less abundant of the two principal terpene hydrocarbons in gum spirits of turpentine) and highly halo-

genated organic compounds (like carbon tetrachloride) to form new chemical products. More halogenated compounds were found to undergo this reaction with beta-pinene, and more catalysts of the peroxide type were found to be effective. Specific uses have not yet been developed for any of the addition products.

Studies on the emulsion copolymerization of styrene with isoprene, obtained from terpenes by decomposition with heat, were concluded. Certain rubberlike products, obtained in good yield, had excellent physical properties after being compounded according to a standard tire-tread formula and vulcanized; the tensile strength ran as high as 3,770 pounds per square inch, and the elongation at the break up to 700 percent.

RESIN-ACID COMPOUND FOUND USEFUL IN MAKING SYNTHETIC RUBBERS

In studying the utility of some of the compounds of resin acids from pine gum as emulsifying agents, or as extenders of other emulsifying agents, for the preparation of synthetic rubber, the addition compound of levopimaric acid and maleic anhydride was made, and its sodium salt was tested. This product alone was not sufficiently soaplike to serve as an emulsifying agent; but when a small amount of it (0.2 to 1 percent on weight of monomers used) was added to the fatty acid soaps commonly used for this purpose, elastomers were formed that showed considerable improvement in physical properties over controls prepared without this resin-acid derivative. The addition of 0.2 percent of this product (based on weight of monomers) to the fatty acid soap used in the emulsion polymerization of GR–S rubber increased the tensile strength of the vulcanized rubber from 2,760 to 3,800 pounds per square inch, and gave elongations at the break up to 600 percent.

PROGRESS MADE IN DEVELOPMENT OF NEW DOMESTIC TANNIN SUPPLIES

From investigations at the Eastern Regional Research Laboratory further progress was made in the development of new tannin supplies which may help to increase domestic production and reduce the dependence of the United States upon foreign tannins. The materials to which particular attention has been directed include: canaigre roots, sumac leaves, scrub oak bark, and pecan shells. Canaigre and sumac can be grown as field crops, and pecan shells are a byproduct of a nut crop that is harvested annually.

Canaigre roots.—Additional experimental plantings of canaigre were made at Lubbock and Winter Haven, Tex., and at State College, N. Mex., in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering. Larger field plantings are being planned in order to obtain roots in quantity for the production of tanning extract for use in semicommercial tanning tests.

Information was secured on the rate of root growth and changes in tannin, sugar, and starch contents and purity of extractives during growth. From a plot planted in September, 100 hills were harvested each month beginning in February and ending in September. Quite rapid increases in root growth and tannin content began in April.

Calculated yields of roots in tons per acre increased from 0.8 ton in February to 13.0 tons in September. The maximum tannin content—22.4 percent obtained by water extraction—was found in samples collected in August. Total sugars decreased from 25 percent in February to 11 percent in July and then increased gradually. Starch, on the other hand, increased from 19.7 percent in February to 35.8 percent in May and then decreased slowly. The results of these periodic harvest studies indicate that, from the standpoint of root yields, tannin content, and purity of extractives, the best time for harvesting canaigre roots is in August.

Among the bacterial cultures that have been used successfully for the fermentation of sugars in canaigre liquors in the presence of tannin, seven were identified as *Aerobacter aerogenes*, and two as *A. cloacae*. Aeration of liquors during fermentation increased the rate of sugar loss and purity increase very markedly. Studies of the fermentation of sugars in canaigre liquors by means of yeasts are in progress. Growth of nine known cultures of yeasts was completely inhibited by 0.5 percent of powdered canaigre root in the broth medium. One culture, an *Endomycopsis* species isolated from partially extracted canaigre by using added nutrients, heavy inoculation, and aeration, reduced the sugar contents and raised liquor purities to above 60 in 12 hours, but about 10 percent of the total tannin was destroyed. The difficulties encountered in the fermentation of canaigre liquors by the more commonly used bacteria were explained by the finding of an inhibitory substance in canaigre root. This substance has not been identified thus far.

Pilot-scale studies for adapting the successful laboratory extraction procedures for canaigre to larger-scale processing were started. Dewatering of canaigre slurries was satisfactorily accomplished by screening and pressing instead of centrifuging. Wet pulping of moistened shredded roots by means of a machine that produces a cutting and shearing action was found to be more satisfactory than pebble-milling.

Methods for the analytical extraction of canaigre roots for the determination of tannin were improved. More complete and concordant tannin extraction values were obtained by using a 50-percent-by-volume mixture of acetone and water as the solvent.

Sumac leaves.—Research on the development of domestic sumac as a source of tannin was continued in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering. Ninety samples of sumac collected in 1946 from plants grown at Beltsville, Md., were analyzed for tannin. The tannin content of 32 of these samples, which were obtained from root plantings of *Rhus copallina* of high tannin content, ranged from 34.0 to 41.6 percent on the moisture-free basis. The results indicate the suitability of these strains for use as propagation stock.

The effect of temperature on the extraction of sumac tannin was systematically studied with three domestic sumacs—*Rhus copallina*, *R. glabra*, and *R. typhina*—and with Sicilian sumac, *R. coriaria*. Extractions were made at 2° C. and 10° intervals from 10° to 100° C. Soluble extractives and tannin increased as extraction temperatures were raised, except for an interval between 30° and 60° where they decreased. The greatest amount of tannin was extracted at 100° C.

These data are important in connection with the commercial leaching of sumac for the manufacture of sumac extract.

To study the tanning qualities of sumac, quantities of *Rhus glabra* and *R. typhina* leaves were dried under the following conditions: (1) in air as a control; (2) at 135° C. (275° F.) with forced draft; (3) in air after being treated with sulfur dioxide; and (4) in air after being held for a time in a moist condition to induce partial spoilage. These several lots of sumac are being used in tanning tests to determine the effect of the various drying conditions on sumac quality.

Physical tests made on leathers commercially tanned with leaves of *Rhus copallina*, *R. glabra*, *R. typhina*, and with the Sicilian sumac, *R. coriaria*, indicated that there are no differences between these various tannages as regards surface smoothness, shrinkage temperature, and real and apparent density, but that leather tanned with *R. copallina* is not as stiff and is more compressible than leathers tanned with the other three species of sumac.

Scrub oak bark.—In cooperation with the Florida Engineering and Industrial Experiment Station, further progress was made in the study of scrub oak bark as a potential source of tannin. Due to inability to secure the needed field equipment for chipping the logs and separating the bark from the wood, it was not possible to get the 200 tons of bark required for the commercial preparation of tanning extract for use in making heavy leather tanning tests. Considerable interest has been shown in the possible utilization of scrub oak trees—the bark for making tanning extract and the wood for the production of wood pulp. Interested parties include tanning extract manufacturers, paper manufacturers, producers of paper pulp, and tanners.

Further data are being secured on the tannin content of scrub oak barks. Previous studies showed that the bark from moderately large *Quercus laevis* trees averages slightly above 10 percent in tannin content. Since trees of small diameter predominate, and it is proposed to use the bark from these and from the larger branches, a study of the tannin content of bark from small trees and branches was begun. Fifty-six representative samples of bark were collected from *Quercus laevis* and *Quercus cinerea* trees ranging from 1 to 6 inches in diameter. The analyses of these samples were completed, and a preliminary examination of the results indicated that the quality of bark obtained from limbs and the trunks of small trees is fully equal to that of bark from large trees.

Pecan shells.—Pecan shells that accumulate at pecan shelling centers are being used to a limited extent as a source of tannin. Preliminary studies of shells from six improved varieties of pecans showed that the tannin is located principally in the soft shell liner, very little being in the hard outer shell. The proportion of liner material ranges from 28.5 to 34.5 percent of the whole-shell weight, and from 13.2 to 15.9 percent of the whole-nut weight. On the moisture-free basis, the tannin content of the liner ranges from 25.6 to 47.8 percent, while that of the hard outer shell is only 0.4 to 1.1 percent. More detailed studies of tannin recovery from this material are in progress. Domestic production of pecans reached a peak of 140,165,000 pounds in 1944.

FIBRILLAR STRUCTURE OF HIDE SUBSTANCE REVEALED BY ELECTRON MICROSCOPE

A beginning has been made in determining the structure of collagen fibrils—the principal submicroscopic constituents of skins—and the manner in which the fibrils are altered during the curing and tanning of hides and skins.

Examination of collagen from cowhide, rat-tail tendon, ligamentum nuchae, and sharkskin by means of the electron microscope has revealed that normal collagen dispersed in water has a fibrillar structure. The fibrils have characteristic cross bands recurring at 660 Å, Angstrom units. At present it is possible to examine solid material only after it has been thoroughly dried, so the normal shape of fibrils in fresh skin cannot be determined directly. There is evidence that the fibrils have serrate edges and that some have the form of hollow tubes, while others appear as flat, twisted ribbons or flattened tubes.

Sharkskin fibrils showed little cross banding. They resembled abnormally swollen collagen, indicating that sharkskin collagen is more highly hydrated than most types of collagen. Vegetable tanned sharkskin fibrils showed the normal form and striations.

Collagen, when dispersed in nonaqueous media, was broken into fragments showing few fibrils. Leather also showed incomplete fibrillation.

INDUSTRIAL PROCESSING OF SWEETPOTATOES AWAITS LOWER PRODUCTION COSTS

In continued cooperation with the United States Sugar Corp., technical studies were conducted during the 1946-47 operations of the sweetpotato starch factory at Clewiston, Fla., to evaluate quantitatively the performance of the individual equipment units new in application to sweetpotato starch extraction and purification. It was clear that, while a few changes in equipment might be desirable for maximum efficiency, the design and equipment of the plant were in general good. Recoveries of finished starch practically as high as were ever previously obtained under optimum conditions in pilot-plant or commercial-scale operations were obtained. The basic flow-sheet, as developed in the Bureau's research over a period of years and essentially carried over to the new plant, was demonstrated to be sound.

Unfortunately, suspension of further starch-factory operations was necessary pending solution of some agricultural problems that are of essentially the same nature as those encountered in the starch enterprise at Laurel, Miss. During the time the Mississippi plant was in operation the cost of growing sweetpotatoes exceeded the price that the factory could afford to pay for raw material, and Government subsidies were necessary to maintain production of raw material. In the Florida undertaking the results of extensive experimental plantings had encouraged the prospect that, with the long growing and harvesting seasons and other favorable conditions in the vicinity, sufficiently heavy yields would be assured to reduce crop production costs to less than $10 per ton, even with relatively high costs per acre. Such did not prove to be the case when production was scaled up to the volume required to sustain the factory.

The consensus of representative groups of agricultural workers meeting at the Southern Regional Laboratory in March 1947 was that, with the practices and equipment so far available, the costs of sweetpotato production would continue higher than is compatible with utilization of the crop for industrial raw material. It was recognized that the principal factors contributing to these high costs were: (1) Inadequate control of disease and insect pests; (2) inefficient procedures for producing and setting plants; and (3) lack of effective mechanization in harvesting and handling operations.

The sweetpotato crop is of high potential importance in the adjustment of southern farm economy to pending conditions. The extended outlets which research has developed for sweetpotatoes in the manufacture of starch and byproducts and the production of stock feed afford a potential outlet for at least double the acreage of the present crop. Research now in progress offers promise of some limited increase in the cost allowable for sweetpotatoes as industrial raw material by decreasing the costs of processing and increasing the value of the products and byproducts. If the industrial exploitation of sweetpotatoes is to be made profitable, the cost of producing the crop must be lowered. This reduction can be effected, but it will require intensive and concerted horticultural, plant pathological, and agricultural engineering research over a number of years.

During the 1946–47 season dehydration of sweetpotatoes to produce a high-quality carbohydrate stock feed attained considerable proportions. In Louisiana alone 25,000 tons of such feed are estimated to have been produced from 3,000,000 bushels of sweetpotatoes. This development owes its origin to pioneer investigations conducted by the Bureau several years ago in connection with the sweetpotato-starch enterprise at Laurel, Miss.

SIMPLIFIED SWEETPOTATO-STARCH PROCESS IN PROSPECT

With the processes so far employed, efficient manufacture of sweetpotato starch has appeared to require large-volume operation, and the trend has been in that direction. But during the past year intensive efforts were directed toward development of an improved and simplified sweetpotato-starch process that would be adaptable to a small, compact plant of low overhead and labor costs. Such a plant could be set up in a small community and operated by a group of cooperative sweetpotato growers to better advantage than the plants that have operated so far. The results of extensive experiments in the pilot plant at New Orleans make very tangible the prospect for a process that employs several stages of batch or continuous centrifuging, fine screening, and bleaching with a materially simplified flow sheet. Improved continuous centrifugals being developed by several manufacturers should permit making the process continuous throughout.

CRUDE PROTEIN OF SWEETPOTATOES RECOVERED FOR FEEDING TESTS

In continued research and development work on the recovery of feed protein from the waste "fruit water" of sweetpotato-starch manufacture, the pilot plant in the laboratory at New Orleans set up

765067—47——4

for the continuous flocculation and concentration of the crude protein was increased to a capacity of 200 gallons or more of fruit water per hour, with practically automatic control of the operations. The conditions for much more effective dewatering of the crude protein coagulate by filtration were established.

From full-scale runs in conjunction with the starch pilot plant, sufficient crude protein was recovered and returned to the byproduct pulp to produce several hundred pounds of protein-enriched feed for evaluation in preliminary feeding trials by the Florida Agricultural Experiment Station. Investigation of the chemical constitution of the protein thus recovered has indicated that it should be of good nutrient value. The recovery step has been carried far enough to indicate its technological feasibility for factory-scale exploitation, and, when further data have been acquired on translating the final dewatering step to commercial filtration equipment, the economic feasibility of the whole process can be appraised.

USE OF ALLYL STARCH FOR VARNISH ADVANCED BY NEW RESEARCH RESULTS

Previous reports told about the preparation by the Eastern Regional Research Laboratory of a new starch compound, allyl starch, which dissolves in certain organic solvents to yield a lacquer or spirit varnish that polymerizes after drying and forms a very hard, glossy, insoluble, and heat-resistant coating. As reported earlier, allyl starch (in which two of the three hydroxyl groups in each glucose unit of starch are replaced by allyl groups) is made from starch, allyl chloride, and caustic soda, all of which are available commercially at low cost.

Most of the research on allyl starch during the past fiscal year was directed toward the development of improved methods for making allyl starch and improved formulas for preparing allyl starch lacquers or varnishes. The first-mentioned work was particularly urgent because of the requests from industrial firms for help in installing efficient manufacturing methods. After the manufacturing variables were studied individually, optimum operating conditions—requiring the minimum quantity of allyl chloride—were found.

In connection with the second objective, it was necessary to learn which of the commercial solvents, thinners, and plasticizing resins are compatible with allyl starch. It was found that allyl starch is versatile, in that many solvents, resins, and driers can be used with it to prepare superior varnishes. The efficient and economical methods and improved formulas that resulted from these investigations are described in a technical publication issued for the benefit of the many organizations interested in either the manufacture or utilization of allyl starch.

The newly published information on manufacturing procedures and varnish formulations has stimulated interest in allyl starch and has facilitated its production and utilization on a larger scale. One industrial firm has obtained a license to manufacture allyl starch under patents assigned to the Secretary of Agriculture, and at least three companies are making allyl starch on a pilot-plant scale. The pilot-plant production should provide adequate quantities of allyl

starch for the extensive utilization studies now being conducted in several research laboratories.

NEW PLASTICIZERS MADE FROM LACTIC ACID

Softening or plasticizing oils are mixed with many resins and elastomers to increase their flexibility and toughness and to facilitate fabrication operations prior to manufacture of the finished articles. These softeners or plasticizers, which are important components of plastics and elastomers, frequently comprise as much as 30 to 45 percent of the finished composition. In fact, the modern plastics industry is dependent upon plasticizers, and probably as much as 200,000,000 pounds of plasticizers—some commanding prices as high as $1 per pound—will be required in 1947.

Because of the importance of plasticizers and the potential outlet for agricultural materials represented by them, some lactic acid derivatives having the properties required in plasticizers were prepared and studied in the Eastern Regional Research Laboratory. Particular attention was given to the production, by economical methods, of products suitable for plasticizing vinyl resins and cellulose derivatives, including cellulose acetate and ethyl cellulose. Most of the lactic acid derivatives included in the investigation were made by treating lactic esters with dibasic acid chlorides, including diglycol bis-chloroformate, or by preparing and acylating the polylactates of various alcohols and glycols. These new plasticizers are not costly, because the yields are high, and the reagents used to prepare most of them are available commercially at low cost.

Certain important properties, such as volatility, compatibility with typical commercial resins, solubility in water, stability in boiling water, and viscosity, were determined for the experimental plasticizers, and after the plasticizers were mixed with a vinyl chloride resin tensile strength, elongation, and flexibility were determined for the resulting composition. The promising results obtained thus far in the evaluation studies indicate that lactic acid can be converted into various derivatives that are suitable for plasticizing several commercial resins. Since some of the lactic acid derivatives are about as effective as the currently preferred commercial plasticizers, several industrial firms have expressed an interest in them and are conducting evaluation and marketing studies.

ACONITATE PRODUCED COMMERCIALLY FROM SUGARCANE MOLASSES

During the last cane-grinding season, the process for recovery of calcium aconitate, developed through the pilot-plant stage by the Agricultural Chemical Research Division, was successfully operated at full plant capacity by the largest sugar mill in Louisiana. Three carloads of high-grade aconitate was produced, and this byproduct of sugar manufacture was marketed at a profit, since the indicated demand is larger than the total potential production from Louisiana molasses. The procedure and conditions for treating second or "B" molasses that had been worked out in the pilot-plant experiments were confirmed in operation of the large commercial plant.

The process, which is continuous, effectively precipitates and recovers aconitate in an operating cycle of about one and one-half hours,

returning the "B" molasses to the sugar mill at the concentration required for further sugar crystallization. The only difficulty encountered was a lack of mechanical efficiency in the continuous centrifuges for removing the crystallized product from the viscous molasses. Work in cooperation with manufacturers of the centrifuges has since indicated that the machines can be brought up to highly efficient performance. When this is done, it will be possible to produce around 500,000 pounds of high-grade aconitate per year at this sugar mill without reducing the sugar content of the molasses being treated.

An incidental advantage of operating this process in a sugar mill is the possibility it affords for improving the quality of the final molasses. It is planned to install an additional continuous centrifuge of a type that is suitable for removing impurities precipitated with the aconitate in treating the "B" molasses; by this means subsequent sugar production will be facilitated, and a premium grade of final molasses entirely free of sediment will be obtained.

Several other sugar mills in Louisiana and Puerto Rico are interested in the possibilities of this process, and numerous samples of "B" molasses have been analyzed for aconitic acid at the New Orleans laboratory for interested parties. The present process can be profitably operated at any mill where the average aconitic acid content is 3½ percent or more of the total solids in the molasses. Research is continuing at New Orleans on the conditions necessary for more complete precipitation of suitable salts of aconitic acid in an effort to increase recoveries and make the process applicable to molasses of lower aconitic acid content.

SUGAR LOSSES REDUCED BY FORCED VENTILATION IN STORAGE OF SUGAR BEETS

About one-third of the sugar beets produced annually are processed after storage in open piles for 30 to 120 days. During such storage considerable loss of sugar occurs, but this is partly preventable. In recent years experimental work has been carried on by the Agricultural Chemical Research Division in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering to learn what factors are responsible for the loss of sugar from beets in piles and to find practical means for improving commercial storage of beets along the lines indicated by the experimental results.

Following a successful large-scale experiment in 1945–46, which made use of forced ventilation, the industry made extensive plant-scale trials of this storage procedure. During the past year 10 comparisons were made by beet-sugar manufacturers between ordinary piles of beets and piles provided with ventilating channels into which night air was blown. In nine cases the sugar recoveries indicated that forced ventilation prevented one-third to two-thirds of the sugar loss shown by the control piles. The results of the tenth experiment were useless because of severe freezing damage, but the experiment demonstrated the need for development of protection against freezing for storage piles that are to be held through the winter.

From basic studies it has been learned that the best storage temperature is about 35° F. Addition of carbon dioxide to the storage atmosphere reduces the sugar loss, but not as effectively as cooling. Beets dried to the extent of losing 10 percent in weight before storage

are very susceptible to spoilage. High-topped beets lose sugar more rapidly from respiration, but are more resistant than low-topped beets to spoilage organisms.

SACCHARIC ACID MADE FROM DEXTROSE

Saccharic acid is of interest both as industrial raw material and as a food acid. Because it is structurally related to adipic acid, one of the basic materials in the production of nylon polymers, saccharic acid may have utility in the production of synthetic resins, plastics, and fibers. However, it is expected that its major use will be in food products. In confections, carbonated beverages, baking powers, and pharmaceuticals, saccharic acid is expected to find uses similar to those of citric and tartaric acids.

A systematic investigation by the Northern Regional Research Laboratory on the oxidation of dextrose with nitric acid resulted in an improved process for the preparation of saccharic acid in nearly 50-percent yield. In addition, a procedure was established for the conversion of the reaction byproducts to oxalic acid in 80-percent yield. The laboratory studies on the conversion of dextrose sugar to saccharic acid were completed, and, on the basis of the laboratory results, a pilot-plant unit was constructed and is ready for operation to obtain engineering and cost data.

Because saccharic acid had not been available previously, its suitability as a food acid was not established. It was necessary, therefore, to have biological assays made of its effects on animals. The Pharmacology Laboratory of the Bureau made such tests with rats. No ill effects on the health or growth of rats were observed when saccharic acid was added to the diet at both low and high levels and fed for short periods. Post-mortem examinations of test animals will be made to determine whether any chronic disorders occurred after long periods of ingestion. If the evidence shows the absence of chronic toxicity effects, then saccharic acid will be considered biologically suitable for all normal food uses of organic acids.

GRAIN PROCESSING INDUSTRIES AIDED BY FUNDAMENTAL RESEARCH

Industrial processing of grains is based upon the physical and chemical properties of the kernel constituents. The separation, in pure form, of the major constituents—starch, protein, and oil—is also dependent upon the structural forms within the kernel. Information on kernel structure and properties is of importance in the wet-milling, dry-milling, baking, cereal manufacturing, and fermentation industries.

The first fundamental studies on grain at the Northern Regional Research Laboratory were made on corn with particular attention to the steeping operation which softens the corn and allows the separation of the starch, germ, and protein fractions. Microscopic and chemical examinations showed that sulfurous acid in the steep water exerts a specific action not equalled by other mineral or organic acids.

Thin sections of corn kernels were steeped under various combinations of sulfurous acid concentration of the steep and duration of

steeping. It was demonstrated microscopically that when corn kernels are subjected to a sulfurous acid steep, considerable break-down and dispersion of the protein is effected. The higher the sulfurous acid content of the steep and the longer the steeping period, the greater was the amount of break-down.

Immediately following release of results from the study of the corn kernel before and during steeping, much interest was shown by research and production workers in industries processing other grains. This emphasized the need for similar studies on other grains.

Barley was chosen as the second grain for fundamental study. The techniques developed during the work on corn were modified for application to this grain. Barley starch granules, like those of corn, were found to be embedded in a proteinaceous matrix within the endosperm cells. In barley, however, both large and small starch granules occur within the same cell throughout the storage tissue. Less protein was found in the tissue at the base of the crease of the barley kernel than in other portions of the endosperm.

These studies have provided a foundation for research on the improvement of corn steeping. The results obtained in the Laboratory have been of immediate practical value, since they led to the formulation of special procedures for the wet-milling of soft, moldy, and heat-damaged corn.

HIGHER OIL CONTENT MAY BECOME GOAL IN CORN BREEDING

Oil is the most valuable constituent of corn, when considered on a unit-weight basis. For nonfeed purposes, a pound of corn oil has always been worth much more than a pound of corn protein or starch. When included in feed derived from corn, the oil furnishes more energy per unit weight than does any other constituent. However, the extensive plant-breeding program that has resulted in the hybrid corn now grown almost exclusively in the Corn Belt has never considered increased oil content as a goal, but instead has sought and achieved increased yield, better drought and disease resistance, and other agronomic improvements. Since a higher oil content of corn may be considered important, plant breeders need information based on chemical analyses in order to guide them in attaining such a goal.

The Northern Regional Research Laboratory has found that an increase in total oil content of corn kernels is reflected proportionately in higher oil content of corn germs, which alone are used as the source of oil in the corn-products refining industry. From analytical data on hundreds of samples, supplied by the Bureau of Plant Industry, Soils, and Agricultural Engineering, the Laboratory has been able to identify a group of pure inbred lines which tends toward increased oil content in the double cross hybrid and, correspondingly, another group of inbred lines which leads to very low oil content in the double cross. Hybrid corn-seed producers have expressed great interest in these data, and when sufficient additional inbred lines are classified, high oil content may be added to the other desirable characteristics now regarded as essential for good varieties of hybrid corn.

VITAMIN CONTENT OF GRAIN SORGHUMS CAN BE INCREASED BY CROSS-BREEDING

Studies on the vitamin content of selected grain sorghums made by the Northern Regional Research Laboratory indicated that nicotinic acid levels might be influenced markedly by the particular varieties employed in producing hybrids. To examine this possibility more closely, 339 samples from seed heads of different plants of a Westland-Cody cross supplied by the Kansas Agricultural Experiment Station were analyzed for nicotinic acid (niacin). Cody normally contains about 71 micrograms of nicotinic acid per gram, and Westland about 42 micrograms per gram. Of the progeny analyzed, only 3 percent contained nicotinic acid in a concentration equal to or lower than that found in the Westland parent, whereas 33 percent contained the vitamin in concentrations equal to or greater than that found in the Cody parent. Three samples contained more than 100 micrograms of nicotinic acid per gram. Nicotinic acid synthesis apparently represents a dominant character, and it seems probable that nutritionally more desirable sorghums can be developed by selective cross-breeding. Such vitamin-rich sorghums would likewise have a greater byproduct value when used as raw materials for alcohol production.

Similar studies, but on a more limited scale, have been made on other cereal grains, including oats, rye, barley, wheat, and corn. As yet no evidence has been obtained which would indicate that marked differences in vitamin content represent a varietal character in the first four of these crops. Some evidence, however, is at hand which indicates that the nicotinic acid content of corn is genetically controlled, and that nutritionally improved varieties of this grain can also be developed through hybridization.

WET MILLING OF GRAIN SORGHUM IS PROSPECTIVE NEW INDUSTRY

Grain sorghum is becoming an important crop throughout the Dust Bowl area. The plant is drought-resistant, and varieties that can be harvested with the combine have been developed. Several industrial firms are interested in the possibility of processing the grain for the recovery of starch and related products, and one is building a plant at Corpus Christi, Tex., capable of wet milling 20,000 bushels of grain sorghum per day.

In most varieties of grain sorghum, the hull contains plant pigments which, during steeping operations, permeate the kernel and impart color to the starch. Thus the starches produced from such grains by the wet-milling procedure as used for corn are tinted with shades of yellow, red, or purple, depending upon the proportions of various pigments in the particular lot of grain. The Northern Regional Research Laboratory has found that pearling of grain sorghum to remove the hull prior to wet milling makes it possible to produce "white" starch having a protein content no greater than that of cornstarch. The pearling operation increases the cost of processing, but the extra cost may be offset, at least in part, by extracting a "carnauba-like" wax which the Kansas State Agricultural Experiment Station has found to exist in the hulls. The uses to which this wax may be put and its market value remain to be determined.

When grain sorghum typical of that grown in the Southwest is pearled, 83 percent of the grain is recovered as dehulled sorghum and 17 percent is removed as sorghum "bran." The wax has been extracted from the bran experimentally, and it has been estimated that about one-half pound of wax can be obtained from the bran that would be removed from 100 pounds of whole grain. The pearled grain responds much better to the wet-milling operations than does the whole grain. A germ fraction of extra-high purity is obtained from the pearled grain, and the amount of coarse fiber in the grain is greatly reduced. The yield of starch from a pound of pearled grain is greater than that from a pound of whole grain, and the starch is whiter and contains less protein.

Farmers are inclined to harvest grain sorghum while its moisture content is rather high in order to avoid losses from shattering. Therefore it would be necessary to dry the grain artificially before putting it in storage, and experience with corn has shown that artificial drying makes wet milling of the grain more difficult. In cooperation with one of the large wet-milling firms, experiments are in progress to determine optimum drying conditions.

Although development work is still in progress on the wet milling of grain sorghum, it appears that, through the cooperative efforts of industry, the Kansas State Agricultural Experiment Station, and the Northern Regional Laboratory, a foundation has been laid for an entirely new industry.

STUDIES MADE ON DRYING OF CULL AND SURPLUS WHITE POTATOES FOR FEED

Because of the Government's policy of removing surplus potatoes from the food market to assist growers, and hence the need for a profitable nonfood use for surplus potatoes, a survey was made of the results thus far obtained in large-scale commercial ventures in dehydration of potatoes for feed in this country. It showed that of the various processes used up to that time one seemed to be outstanding. This process consists of washing potatoes, pulping them in a hammermill equipped with special hammers, adding about 0.8 percent lime, dewatering in a continuous press and drying in a direct-heat rotary dryer. Cost estimates were prepared on this and other processes that have been tried on a large scale. They showed that the above-mentioned process offers certain economies, but has attendant risks. With a factory costing about $42,000 and having an annual capacity (125-day operating season) of 1,700 tons of dried potatoes containing 12 percent moisture, the cost of producing the product, not including cost of potatoes, would be about $26 per ton.

Because the drying of disintegrated potatoes by direct heat is hazardous, pilot-plant experiments were carried out at the Eastern Regional Research Laboratory to develop an equally cheap but safe method. This is based upon washing the potatoes and distintegrating them in a hammermill equipped with blunt hammers and a screen having ⅛-inch openings. At high moisture content the product sticks very badly to any dryer except one having temperatures over 700° F. Such a direct-heat dryer is hazardous because of the tendency for starch to form an explosive mixture with air.

It was found that if the pulped potatoes are mixed with enough dried potatoes to reduce the average moisture content to not more than 43 percent, sticking is overcome, and drying can be done safely in a rotary steam-tube dryer. This is the cheapest safe method of drying potatoes for feed that has been devised thus far. The processing cost of the product (not including cost of potatoes) is estimated to be about $23 per ton. The process eliminates all stream contamination, and the product should contain all the starch, proteins, minerals, and other nutrients that were in the raw potatoes. A factory processing 75 tons of potatoes a day for 125 days would produce about 2,100 tons of feed containing 12 percent moisture.

Correlation of available data on the feeding value of dried potatoes for cattle, sheep, and hogs showed that, in general, they can be considered to have a value of about nine-tenths that of No. 2 yellow corn. On this basis, and at an estimated cost of $23 per ton for processing the potatoes, the net income from sale of dried potatoes for feed in competition with No. 2 yellow corn at $2 a bushel would be enough to permit the payment of about $8.75 per ton for raw potatoes delivered at the factory and to give a return of 10 percent on the investment. By this process about 4½ tons of potatoes are required for producing a ton of dried product containing 12-percent moisture, whereas about 5½ tons are required by the process that requires pressing the potatoes before drying and discarding the entrained solids in the press liquor.

Pilot-plant experiments were made to determine the feasibility of hydrolyzing the starch in ground potatoes to sugars by means of mineral acid and then neutralizing and concentrating to produce a molasses-like stock feed. It was found that in order to carry the process out in a reasonably short time with comparatively simple equipment such high concentrations of acid were required for the hydrolysis that the finished product contained more than 10 percent of sodium chloride. It was also bitter and lacking in sweetness. Should laboratory experiments indicate the feasibility of pressure hydrolysis at lower acid concentrations, pilot-plant evaluation of such a procedure will be undertaken.

During the past year experiments were made also on the drum drying of partially dewatered potatoes and grinding into flour. They met with some success, and a potato flour of good appearance was prepared from washed but unpeeled potatoes. A sample of this flour was submitted to a representative of the baking industry for evaluation as an ingredient in bread.

CAROTENE IN LEAF MEALS FULLY UTILIZED BY CHICKS AS SOURCE OF VITAMIN A

It has been known for many years that carotene, one of the yellow pigments existing in many plants, and especially in green leaves, carrot roots, and yellow corn, is the precursor of vitamin A. There is conflicting evidence, however, as to the degree to which various animals can utilize carotene as a source of vitamin A. Chick-feeding mashes, for example, are commonly fortified with fish-liver oils as a source of vitamin A, regardless of the fact that some ingredients of the mash contribute considerable carotene. In the attempt of the Eastern Regional Research Laboratory to establish uses for vegetable-leaf

wastes high in carotene, this question was posed: Can chicks get *all* their vitamin A requirements from carotene?

In a series of cooperative feeding trials at the Delaware Agricultural Experiment Station, this question was answered in the affirmative. To one set of mashes esters of vitamin A (in fish-liver oil) were added at three levels—below normal, normal, and above normal. Other sets of similar mashes contained no vitamin A, but instead the calculated equivalents of carotene, and the carotene was in three different forms—leaf meal, extract of the meal, and carotene distilled from the extract. At the end of 9- to 12-week feeding periods it appeared that all of the carotene preparations were just as efficient as vitamin A esters in sustaining chick growth. Since the chick can utilize carotene to meet its vitamine A requirements, a valuable outlet for vegetable-leaf wastes is indicated.

Carotene concentrates, in the form of vegetable-leaf meals or extracts of such meals, have an advantage over the vitamin A esters in fish-liver oils for use in premixed feeds, because there is less chemical change with attendant loss of vitamin units when the carotene preparations are used.

PROGRESS MADE IN PRODUCING FEED YEAST FROM CITRUS AND SWEETPOTATO WASTE LIQUORS

Encouraging results previously obtained in the preliminary operation of a feed-yeast pilot plant, set up in a cooperating citrus cannery at Orlando, Fla., led to a thorough study of the equipment and process during the past year. The Bureau's Citrus Products Laboratory and its Southern Regional Research Laboratory conducted experimental runs on citrus press liquor after which the canning company operated the plant to produce a substantial quantity of yeast for commercial evaluation. Replacement of some improvised mechanical units, used during the preliminary operations of the previous season, with more efficient equipment of proper design made possible smooth, continuous operation and adequate control of the process. The new equipment included an air compressor and a pasteurizer of larger capacities, devices for automatically metering the nutrient solution, a continuous centrifuge especially designed for yeast concentration, and a larger drum drier. Accurate data obtained on yeast yields, on consumption of nutrients, and on reduction of biological oxygen demand of the effluent permitted estimates of possible profit from sale of the product and from reduced cost of waste disposal. The engineering data obtained on citrus press liquor are directly applicable to the production of feed yeast from sweetpotato processing waste water.

In these production tests it was established that ammonium sulfate was a cheaper and better source of nitrogen than the ammonium chloride previously used. It was shown also that the culture of torula or feed yeast could be kept free of contamination for several days without pasteurizing the raw press-liquor feed, provided vigorous growth was maintained in the fermenters. In treating about 140 gallons per hour of straight citrus press liquor containing 5 to 6 percent sugars, yields of dry yeast reached 36 percent based on total weight of sugars present. By dilution of the citrus press liquor with an equal volume of water the yield was increased to about 50 percent.

In a small laboratory fermenter yields up to 60 percent were obtained when one part of press liquor was diluted with three parts of water.

These results are in agreement with those obtained in earlier experiments on "fruit water" from sweetpotato starch processing, which contained only 0.75 to 1 percent total sugars. Accurate data on changes occurring in each of the three fermenters connected in series indicated that the third fermenter could be dispensed with, and that it might even be possible to operate continuously through a single fermentation chamber. Further research is needed to confirm these results and to determine the best conditions for growing feed yeast on the liquid wastes from both citrus and sweetpotato processing operations.

WASTES FROM FRUIT AND VEGETABLE PROCESSING UTILIZED

Fruit wastes.—Additional practical knowledge has been obtained by the Western Regional Research Laboratory on the problem of converting the large tonnage of fruit-cannery peel, cores, trimmings, discards, and other waste into useful products. In cooperation with a cannery at Olympia, Washington, and in commercial-type equipment, pear waste was used as a nutrient for growing feed yeast. The many operating problems were successfully solved, so far as the fermentation and yeast-recovery processes are concerned. One major obstacle, however, must be removed before this process will be ready for use. Thus far no commercially available equipment has been found to be entirely satisfactory for separating the pear pulp from the juice, which is a necessary step prior to the fermentation. Progress was made toward devising suitable equipment, and work on this problem will be continued.

Analysis of the yeast produced indicated that it should be a valuable poultry- and stock-feed ingredient because of its high protein and vitamin contents. In order to test this possibility in a practical way, poultry feeding trials are being conducted by Washington State College. Preliminary reports indicated a favorable outcome for the feeding trials.

The cannery waste from pears alone amounts to about 96,000 tons annually, which if converted into yeast would yield 3,000 tons of a concentrated feed having an estimated value of $600,000. In addition to the advantage of using a waste material to create a valuable and in fact badly needed feedstuff, a process of this type helps to solve the increasingly troublesome problem of stream pollution. Many canneries and food-freezing plants are confronted with the necessity of spending large amounts of money for waste disposal. Hence a way to recover values from these wastes would result in a worth-while net gain even though it paid only the cost of the process.

Vegetable wastes.—Previous reports mentioned another use for an extensive processing waste. At the Western Regional Laboratory it was found that a concentrated juice prepared from waste asparagus butts made an excellent medium for growing a number of useful microorganisms. A process for the production of juice concentrate on a large scale was developed and tested to a point that demonstrated its commercial feasibility. Among the organisms grown on asparagus juice was one called *Bacillus subtilis* which was discovered to yield a new antibiotic that was named subtilin.

During the past year further work on subtilin resulted in improved methods for its commercial production. Better recovery and purification were achieved, and important information was obtained on the nutritional requirements of *Bacillus subtilis*. This made it possible to supplement or "fertilize" any juice concentrates that were deficient in nutrients essential for *Bacillus subtilis*.

Additional clinical results thus far obtained by cooperating medical institutions continue to indicate that subtilin may be useful in treating tuberculosis and amoebic dysentery. If this proves to be the case, subtilin will become important in the large family of antibiotics which is now distinguished by such members as penicillin and streptomycin. Recent publications by the Bureau on methods for the production and purification of subtilin, together with the reports on its possible uses, have aroused considerable interest. Plans are being made by certain drug manufacturers to undertake supplying the subtilin that will be needed for more extensive testing by the medical profession.

Recent experiments at the Western Regional Laboratory showed that subtilin, or some other substance produced with it, has marked antifungal properties. This preliminary finding, if fully confirmed, may lead to additional uses of commercial importance.

Concentrated juice from asparagus waste was previously found to be an excellent medium for *Bacillus brevis*, which produces the complex substance tyrothricin from which the antibiotics tyrocidin and gramicidin are obtained. Gramicidin was more promising for medicinal use, but upon being tested by the University of California proved to be too toxic. A chemical modification of gramicidin was prepared at the Western Regional Laboratory, and this proved to be relatively nontoxic. Recent reports of collaborative tests indicated that it may be very useful against surface infections and that it does not produce sensitization to subsequent applications. This finding is important because it means that gramicidin can be used instead of penicillin for surface infections, thus reserving the full effectiveness of penicillin for systemic use when it may save life.

BETTER PRODUCTION METHOD AND NEW USES DEVELOPED FOR PECTIN PRODUCTS

Preliminary experiments in producing low-methoxyl pectin from waste citrus peel by a new process were made during the 1946 Valencia orange season. These experiments were on a semicommercial scale in cooperation with a large food-processing company in southern California. The method used is the one developed at the Western Regional Research Laboratory which was mentioned in last year's report. Much valuable information was obtained regarding the proper design and operation of equipment for the commercial application of this new process. The results were so encouraging that the cooperating company desired to continue the large-scale tests for another season and planned to invest considerable money in the needed additional equipment. A preliminary estimate of the cost of producing low-methoxyl pectin by this new process indicated that it is economically sound. It was discovered that dried citrus peel can be used successfully in this process. The use of dried peel is of considerable importance because it

permits year-round operation with the resulting advantage of more efficient production with regard to both labor and invested capital.

One of the promising new uses for low-methoxyl pectin being investigated at the Western Regional Laboratory is as a sausage casing or protective film for certain other foods. The process consists of dipping the sausage, cheese, dried fruit or other product in a warm solution of a calcium salt of low-methoxyl pectin. The adhering liquid gels on cooling and is then dried rapidly. The resulting film gives good protection and is attractive in appearance. A special advantage is that upon boiling it dissolves. If the food is fried or is eaten without cooking, the film may be consumed with the food, since it is tender and edible. By use of this dip-coating method, sausage can be marketed in the form of patties or any other desired form.

Another potential use for pectin is for spinning calcium pectinate fibers. These fibers have only fair strength, and although there is no indication at present that they would be suitable for weaving into fabrics, their use as a "scaffolding" in producing special open-weave fabrics is a distinct possibility, since the scaffolding threads could be easily removed when desired by washing the finished cloth in a solution containing a chemical that decomposes calcium pectinate.

VITAMIN C EXTRACTED FROM HULLS OF "ENGLISH" WALNUTS

Following the discovery by Russian scientists that green immature walnuts contain large amounts of vitamin C (ascorbic acid), the Western Regional Research Laboratory determined the quantity of vitamin C in the varieties of Persian or so-called English walnuts grown commercially in California. It was found that immature walnuts contain as much as 25 to 30 percent (on the dry-weight basis) and that the hulls removed from ripe nuts retain from 3 to 5 percent of vitamin C. Around 20,000 to 30,000 tons of suitable hulls are available each year for processing, and at present these constitute a disposal problem for the walnut growers. This quantity of hulls contains about 125,000 pounds of recoverable ascorbic acid, which at current market prices is worth nearly $1,500,000. In 1945 the total production of (synthetic) ascorbic acid in the United States amounted to 1,306,813 pounds. Ascorbic acid has been used extensively in medicinal preparations and recently came into use as an antioxidant to protect the color of frozen and canned fruits and vegetables.

A tentative procedure for recovering ascorbic acid from walnut hulls was developed at the Western Regional Laboratory. Briefly, it includes extraction of the vitamin with hot dilate sulfur dioxide solution, adsorption on ion-exchange resin, elution from the resin with acid, concentration, and crystallization in pure form.

In cooperation with the California Walnut Growers Association, the recovery procedure was tried on a fairly large scale at Lynden, Calif., during the last harvest season. It was demonstrated that continuous extraction equipment can be operated smoothly and efficiently. Additional cooperative work with the Association was planned for the 1947 season. From this work it was expected to obtain more complete cost and operational data which would make possible an economic evaluation of walnut hulls as a commercial source of ascorbic acid.

RUTIN FROM BUCKWHEAT PLANTS NOW SOLD BY DRUGGISTS ON DOCTORS' PRESCRIPTIONS

Further evidence is accumulating that rutin, the flavonol glucoside now being extracted from the buckwheat plant, is effective in reducing increased capillary fragility and permeability that sometimes accompany high blood pressure. In September 1946 rutin appeared on the market in tablet form for use in filling doctors' prescriptions. A number of drug manufacturers extracted rutin from buckwheat on a commercial scale during 1946, and several companies are specializing in the production of pure rutin for sale to wholesale drug firms for resale to tablet manufacturers. About 500 tons of buckwheat leaf meal was produced during 1946 by five manufacturers of alfalfa meal. It was expected that a greater quantity and better quality of buckwheat leaf meal would be produced during the 1947 season.

The rutin investigations of the Eastern Regional Research Laboratory are directed toward improving methods for preparing buckwheat leaf meal, extracting rutin from leaf meal and green plants, purifying rutin, and analyzing plant materials, rutin preparations, and biological fluids. Considerable effort has been devoted to cooperation with industry in establishing commercial production of buckwheat leaf meal and purified rutin and the encouragement of clinical and pharmacological investigations through conferences and by furnishing purified rutin for such investigations. Studies on the physiological effects of rutin are under way in the Bureau's Pharmacology Laboratory.

Preliminary experiments in cooperation with the Pennsylvania Agricultural Experiment Station and the Bureau of Plant Industry, Soils, and Agricultural Engineering indicated that Tartarian buckwheat (*Fagopyrum tataricum*) is superior to the Japanese variety (*F. esculentum*) both in percentage of rutin in the plants and in total yield of rutin per acre. In addition, more time is available for harvesting, since the rutin content does not begin to diminish as early as in the Japanese variety. Another advantage of the Tartarian variety is that it is more frost-resistant. Inquiries received from farmers and manufacturers indicated that a considerable acreage of Tartarian buckwheat would be grown during 1947 for rutin production.

During the past year the Laboratory staff advised numerous commercial driers of alfalfa on how their equipment could be adapted to the drying of buckwheat plants. In consequence, several companies were planning to dry buckwheat plants in such quantity that an adequate supply of leaf meal should be available for increased rutin production during the next year. additional data were obtained in the pilot plant on suitable conditions for buckwheat drying, and an improved method was developed for eliminating the stems from the more valuable leaf fraction.

Improvements were made in the three processes developed at the Eastern Regional Laboratory for extraction of rutin from buckwheat. These processes are: (1) Extraction of the green plant with alcohol; (2) extraction of buckwheat leaf meal with 70-percent alcohol; and (3) extraction of buckwheat leaf meal with hot water. Denatured alcohol, methyl alcohol, or isopropyl alcohol can be used in the alcohol-extraction processes. Removal of fats extracted by alcohol is now accomplished by a straining process instead of by extraction with benzene. Comparable yields of rutin were obtained by alcohol extrac-

tion and water extraction of the meal. Methods of refining crude rutin were improved, making possible a reduction in the number of crystallizations in both processes.

Information on improvements in processes and analytical methods was passed on to rutin producers and will be made more generally available by inclusion in a comprehensive publication on rutin that is in process of compilation.

Considerable interest in the use of rutin was aroused in medical circles. Many requests were received for rutin tablets and pure crystallized rutin for use in clinics, hospitals, research laboratories, and the private practice of physicians. Rutin was produced in the Eastern Regional Laboratory on a pilot-plant scale to furnish material for the extended clinical and pharmacological studies. Physicians at the University of Pennsylvania Medical School have demonstrated that rutin is effective in reducing increased capillary fragility and increased cutaneous lymphatic flow sometimes associated with hypertension, and that rutin reduces the severity of damage produced in rats by irradiation with X-rays. Publications have appeared indicating that rutin is effective in the treatment of diabetic retinitis, purpura, and hereditary telangiectasia. Progress has been made in the Bureau's Pharmacology Laboratory and in the University of Pennsylvania Medical School on the development of biological assay methods for evaluation of rutin. Other clinical and pharmacological investigations are in progress, and it is expected that a number of publications on rutin will appear shortly.

PURE TOMATIN ISOLATED IN CRYSTAL FORM

It was reported last year that extracts from tomato leaves possess antibiotic activity against some of the fungi and bacteria that cause diseases in plants and animals, and that this activity is due to a recently discovered substance which has been named tomatin. Because of the potential importance of tomatin in the control of fungus diseases in plants, animals, and humans, vigorous efforts were made during the past year to isolate and characterize this antibiotic agent in order to ascertain its value in direct, uncomplicated experiments.

Tomatin has been isolated in pure, crystalline form, and sufficient evidence has been accumulated to lead to its tentative identification. Laboratory tests in glassware showed that crystalline tomatin is very effective in preventing the growth of some fungi that cause disease in humans and certain plants, but that it is practically without effect against bacteria. This is in marked contrast to the crude tomatin preparations, which had been tested previously in the same way and found to prevent the growth of both fungi and bacteria. This difference suggests that unidentified substances besides tomatin may be present in the tomato leaf, and that these may contribute to the more general antibiotic effectiveness of the crude tomato-leaf extracts. Nevertheless, it is believed on the basis of the evidence in hand that the crystalline substance isolated (tomatin) is responsible for the major part of the antibiotic activity of tomato-leaf extracts, even though other antibiotic agents may be present.

Crystalline tomatin has been supplied to bacteriologists and clinicians of Duke University School of Medicine, at Durham, N. C., who are cooperating with this Bureau by investigating the toxicity and

clinical potentialities of tomatin for the control of fungus infections in man. On the outcome of the tests at Duke University will depend the immediate medical uses of tomatin. If these tests prove that tomatin is a safe and effective drug, the medical profession will be provided for the first time with an agent for controlling the serious fungus infections. However, even if these tests prove that tomatin is of little or no value as a therapeutic agent, the fact that tomatin has been crystallized in pure form still represents a long step forward in the investigation of the role played by this antibiotic agent in the normal disease defense of wilt-resistant tomato plants. With crystalline tomatin available, the problem of wilt resistance in tomatoes is now being prosecuted with renewed vigor.

Since the technique for the isolation and crystallization of one of the antibiotic agents in higher plants has been established, it is possible that the agents in cabbage, pepper, potato, and sweetpotato plants previously reported as having tomatinlike antibotic activity may now be isolated and characterized. These agents may have different and perhaps more desirable antibiotic properties than tomatin to recommend them for therapeutic use, and it may be found that they, too, play important roles in the normal disease defense of the plants that produce them.

INSECTICIDAL VALUE OF NICOTINE INCREASED BY ADDITION OF PHTHALONITRILE

Among the ways in which the Eastern Regional Research Laboratory has sought to improve the usefulness of nicotine as an insecticide has been to search for an activator or synergist. Synergism has been aptly defined as the property that makes two plus two equal five. In other words, the effectiveness of the mixture is greater than the sum of the effects when the materials are used separately. An inexpensive synergist would not only stretch the supply of nicotine, but would lower the cost per treatment.

Since synergists for pyrethrins are known, it seemed reasonable that some could be found for nicotine. In the search for them the procedure was to subject a mixture of nicotine and another compound to a preliminary "screening test" against several different insects. Any compound which "stayed on the screen" was presumed to be a possible synergist, and was later used with nicotine in quantitative tests designed to produce dosage-mortality curves. The latter were analyzed by a mathematical method developed in the Bureau of Entomology and Plant Quarantine. Several hundred compounds have been screened to date. They were picked largely at random, since as yet there is no basis for selection. Of those tested, 15 showed possible synergism, and the effects of 2 of them, phthalonitrile and pentachloroanisole, have been analyzed quantitatively.

Under the conditions used, mixtures of phthalonitrile and nicotine compounds showed marked synergism against the armyworm, definite synergism against the pea aphid, diamondback moth, and California oakworm, doubtful synergism against the green dockbeetle and celery leaf tier, and no synergism against the pomace fly and greenhouse thrip. Synergism was shown by the mixtures of phthalonitrile with three forms of nicotine—nicotine sulfate, nicotine bentonite, and cuprous nicotine cyanide—when they were used either as stomach poisons

or as fumigants. In many cases the toxicity of the nicotine was doubled or trebled. Mixtures of nicotine sulfate and pentachloranisole showed no synergism against the diamondback moth and armyworm.

Although phthalonitrile may not prove to be a feasible adjunct to be used with nicotine, its effect proves that nicotine synergists exist. The search for others can continue with increased confidence.

PATH OF SYNTHETIC GROWTH REGULATOR IN GROWING PLANTS INDICATED BY RADIOACTIVE IODINE IN MOLECULES

During the past year, formal application for radioactive iodine was submitted to and approved by the War Department, and the Bureau received two lots of radioiodine from the Clinton Laboratories, Oak Ridge, Tenn., for use in synthesizing "labelled" plant-growth regulators whose paths can be followed in growing plants by radioactivity measurements. Equipment for the measurement of radioactivity was designed, purchased, and installed, and the first phases of continuing investigations in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering on the mechanism of the action of plant-growth regulating substances in growing plants were completed.

The first objective was to determine whether or not plant-growth regulators of the type represented by 2,4–D (dichlorophenoxyacetic acid) are absorbed and translocated by representative dicotyledonous and monocotyledonous plants (beans and barley, respectively) and, if translocation occurs, to measure the amounts of the regulator that accumulate in various parts of the treated plant. It was found that a plant-growth regulator of the 2,4–D type is absorbed by the leaves of both bean and barley plants, is translocated predominantly to the terminal bud of bean plants where greatest inhibition in growth occurs, and is translocated predominantly to the second leaf of barley plants, where growth inhibition is inappreciable.

Thus for the first time it was possible to demonstrate directly that a plant-growth regulator of the 2,4–D type actually is absorbed and translocated by the growing plant. It was surprising that this growth regulator was absorbed both by dicotyledonous plants (where growth was inhibited) and by monocotyledonous plants (where no effect upon growth was shown).

Further work showed that the apparent incongruity between the growth-inhibiting effects of plant-growth regulators of the 2,4–D type in dicotyledonous plants and their failure to produce significant inhibition of growth in monocotyledonous plants is probably due to a difference in the way the regulator reacts with the plant constituents in the two cases. The investigation is being continued to determine the nature of the reactions. It is believed that after the basic nature of the inhibition mechanism is understood, it will be possible to develop growth regulators that are more specific and perhaps more potent in producing, on a practical scale, any desired degree of growth inhibition in growing plants.

GROWTH INHIBITORS ISOLATED FROM LIMA BEANS AND EGG WHITE

Certain protein substances are known to contain ingredients that interfere with their complete digestion by animals when such sub-

stances are fed in the natural or raw condition. These ingredients are called growth inhibitors. Obviously, it is necessary to destroy or counteract the effects of such growth inhibitors in food or feed materials by proper conditions of processing in order to utilize them efficiently. Moreover, these growth inhibitors may have antibiotic value or be useful for other purposes. Therefore the Western Regional Research Laboratory undertook to isolate some growth inhibitors from food products and to learn something about their chemical structure and properties, how stable they are under various processing conditions, and for what purposes they can be used. In order to guide the efforts to concentrate and isolate the growth inhibitors, biological assays comprising controlled feeding tests with white rats had to be used in the absence of sufficient knowledge of their chemical and physical properties on which to base analytical procedures.

Some evidence had been obtained in earlier investigations, by Government and other research workers studying proteins and nutrition problems, that certain unidentified ingredients of unheated lima bean and soybean proteins prevented the digestive action of trypsin, one of the proteolytic enzymes in the digestive tracts of animals. Therefore the growth inhibitors in these substances were called antitrypsins.

During the past year the Western Regional Laboratory extracted from lima beans and purified a substance that prevents the action of trypsin in laboratory tests. This substance is apparently a protein, and its ability to prevent tryptic digestion can be destroyed by heat. Although the purified substance is more active than the crude preparation in preventing tryptic digestion in glassware, it is only one-third as active as the crude preparation in preventing the growth of rats. The explanation of this unexpected difference in activity has not been found thus far. It may be due to a change in the protein that makes it somewhat susceptible to attack by other digestive enzymes, or it may indicate the presence of more than one growth inhibitor in the crude preparation.

The presence of a tryptic digestion inhibitor in raw egg white proteins has been known for some time as a result of investigations of Department of Agriculture scientists. Because of recent interest in antitrypsins as antibiotics and antigrowth factors, the study of egg white antitrypsin was reopened at the Western Regional Laboratory. By laboratory tests in glassware antitrypsin activity was equal in thin and thick egg white but was nearly absent in egg yolk. The activity was found to be concentrated in the ovomucoid fraction of the egg white, and further attempt to fractionate it was unsuccessful.

These results and other available data indicate that ovomucoid and the antitrypsin are identical, and that one molecule of ovomucoid combines with one molecule of trypsin to effect the inhibition. Heat denatured ovomucoid has no antitryptic activity.

Egg white antitrypsin, however, does not inhibit growth of animals. If the antitryptic factor of lima beans is identical with the growth inhibiting factor, then the mode of action of the two antitrypsins must be quite different. Further work to elucidate this question is in progress, and the possible uses of antitryptic factors as protective agents against micro-organisms are being investigated.

ALLERGENS RESEARCH EXTENDED TO FLAXSEED

The same principles of chemical fractionation that were first developed for isolation of the "1A" allergenic fraction from cottonseed were applied to flaxseed. A corresponding fraction was obtained and was designated as FS–1A. It was similar in chemical composition to the principal allergenic fraction of cottonseed in that both are composed of protein, identified as natural proteose, in combination with complex carbohydrate. The yield of the allergenic fraction FS–1A was only 0.07 percent of the defatted flaxseed. This was substantially smaller than the yields of corresponding fractions from cottonseed and castor-bean (1.1 percent of CS–1A from cottonseed and 1.7 percent of CB–1A from castor-bean). It is presumed, however, that gummy components of the flaxseed hulls, which interfered with the fractionation, accounted for the low yield of the 1A fraction from the flaxseed.

The flaxseed fraction FS–1A possessed specific allergenic activity, but it was less potent than the corresponding fractions from cottonseed and castor bean. This is shown by a comparison of figures for the smallest amounts, expressed in millimicrograms, required to induce a positive allergic response which are as follows: Flaxseed 1A, 10.0; cottonseed 1A, 1.0; castor-bean 1A, 0.1. The flaxseed allergen may be encountered in the whole or ground flaxseed and in the products made from the meal from which the oil has been expressed.

New products of cottonseed became available for examination this year as the result of the process developed at the Southern Regional Research Laboratory for solvent extraction and separation of the pigment glands of cottonseed. The defatted and depigmented cottonseed meal was fractionated to obtain the 1A component. This fraction contained a higher proportion of nitrogen and was lighter in color than the corresponding fraction from the same lot of seed defatted without extracting the pigment glands. Tests on the relative allergenic activity of the CS–1A fraction, the pigment glands, and the defatted cottonseed indicated that the pigment glands may contain an allergenic component different from CS–1A.

Last year's report directed attention to the usefulness of the anaphylactic reaction in correlating the chemical constitution of proteins with their capacity to induce an allergic response in the human or animal. The anaphylactic reaction of the guinea pig has been most used as a reliable and particularly sensitive test for qualitative identification of antigenic proteins. Study of the quantitative relations of anaphylaxis were continued during the past year with the object of determining the effect of the route of administration of the sensitizing dose of protein on the degree of anaphylatic sensitization established.

Three routes for administering the sensitizing dose of protein are in common use: (1) Subcutaneous (2) intraabdominal and (3) intravenous. Opinions of investigators differed with respect to the importance of discriminating among these methods, but no experimental data had been recorded to show whether, or to what degree, the anaphylactic reaction may be influenced by the route of injection of the sensitizing protein. Experiments completed during the past year showed that, for quantitative use of anaphylaxis, the route chosen for injection of the sensitizing dose has a significant influence on the

degree of sensitiveness established. The order of increasing effectiveness for these routes of administration are subcutaneous, intravenous, and intraabdominal.

Additional data will be accumulated to determine the precise quantitative relationship of these methods. Available evidence indicates that for establishing a uniform degree of sensitiveness to egg albumin, the minimal dose by the subcutaneous route is more than double the dose required when the protein is injected by the intraabdominal route. Thus, the route of administration must be considered when comparing the capacity of proteins to induce anaphylactic reactions.

ALLERGENS OF JOHNIN AND TUBERCULIN STUDIED TO INCREASE ACCURACY OF DISEASE DIAGNOSIS IN LIVESTOCK

First results are reported this year on a new field of research on the immunochemistry of allergens. Plans for this work were developed from preliminary experiments which were conducted last year in collaboration with the Bureau of Animal Industry. These experiments showed that johnin and tuberculin, the bacterial products used for differential diagnosis of Johne's disease and tuberculosis of cattle contained allergens that induce anaphylaxis in guinea pigs. This observation opened the way for a new approach to the solution of a fundamental problem encountered in distinguishing Johne's disease from tuberculosis at an early stage of infection.

In the latent or early stages of these diseases, either johnin or tuberculin occasionally produces a false positive skin reaction, leading to an uncertain or incorrect diagnosis. The false positive skin reactions are attributed to allergens common to both johnin and tuberculin. The presence of these common allergens may obscure the specific skin reaction which is usually induced by allergens that are characteristic of johnin and tuberculin, respectively. Accordingly, the fractionation of johnin and tuberculin in conjunction with immunologic study of the allergens of each product was started this year as a separate line of research.

JOHNIN AND TUBERCULIN SEPARATED INTO ANTIGENIC COMPONENTS

The separation of antigenic components of johnin and tuberculin was accomplished by fractionation. Preliminary experiments were confined to tuberculin, for the most part, because this material is produced in greater volume and at lower cost than johnin.

During the past year about 50 preparations of tuberculin and johnin were made and assayed biologically. The different procedures employed for fractionation of johnin and tuberculin included precipitation with graded concentrations of alcohol, dialysis, and electrophoresis. Neither fractionation with alcohol nor electrophoretic fractionation accomplished any significant increase in potency or specificity of the antigens of tuberculin. A favorable concentration of the antigens of tuberculin was obtained by a combination of dialysis and precipitation with alcohol. A fraction obtained by this procedure, representing about one-fifth of the nitrogen of the regular tuberculin, showed a fourfold increase in antigenic potency when compared with the regular tuberculin from which it was derived. There was no significant improvement in specificity, but this fraction was

superior to the regular tuberculin for inducing uniform anaphylactic sensitization in guinea pigs.

JOHNIN AND TUBERCULIN STUDIED WITH REGARD TO IMMUNOLOGIC PROPERTIES

In addition to the immunologic assay of products resulting from fractionation of johnin and tuberculin, several exploratory experiments were conducted to supply a foundation for planning future studies. The immediate object was to determine whether the anaphylactic response of the guinea pig could be used in place of the usual skin test on large animals to detect and characterize antigenic components of johnin and tuberculin. Bacterial products, such as these, are generally poor antigens for inducing anaphylaxis. Moreover, anaphylactic antigens are likely to be weakened by the prolonged heating employed in the preparation of regular johnin and regular tuberculin. The sensitizing effects of weak anaphylactogens can sometimes be increased if the rate of absorption after administration of the dose can be retarded in order to prolong the reaction to the sensitizing components. Formation of a precipitate with alum was tested as a method for thus increasing the sensitizing activity of johnin and tuberculin.

Injection of guinea pigs with regular tuberculin and regular johnin heated with alum induced anaphylactic sensitivity to these products. The sensitivity developed was not of the high degree desired, and the anaphylactic responses observed in subsequent tests were somewhat irregular. The johnin was the weaker of the two in anaphylactic activity. A substantial improvement in antigenic activity was gained with modified preparations of johnin and tuberculin. These were made without heating above room temperature in the final stage of concentration. When the unheated tuberculin and the unheated johnin were administered with alum precipitate the animals developed a higher and more uniform degree of anaphylactic sensitivity, sufficient to permit further studies for comparing the antigenic components of johnin and tuberculin by anaphylactic tests.

Guinea pigs sensitized with johnin showed an anaphylactic response to subsequent tests with johnin, and they also reacted to human tuberculin and avian tuberculin. Results of cross-reactions in paired tests with these preparations indicated that each of them contained some antigenic components that were common to all. This finding corresponds with and serves to explain the fact that nonspecific positive skin tests are observed when tuberculin and johnin are used to detect and differentiate infections of tuberculosis and Johne's disease in large animals.

Apparently, a common antigen may obscure or interfere with specific antigens upon which the correct diagnostic skin reaction depends. However, promising results were obtained from additional anaphylactic tests which showed that tuberculin and johnin contain antigenic components that are distinguishably different and characteristic of each product. Such specific antigenic components, and only antigens of this character, are desired in the ideal preparation used to detect and differentiate infections of closely related organisms. The practical attainment of this ideal type of diagnostic agent depends first upon identification of the specific antigen and second upon separation and preservation of these specific agents.

POLYMERIZABLE ESTERS MADE FROM OLEIC ACID

Although long-chain fatty compounds are useful as plasticizers and modifiers for some synthetic resins and elastomers, their low compatibility and tendency to exude have limited their use. If the fatty compound were an integral part of the polymer molecule, however, these disadvantages might be obviated. One method of accomplishing this would be to attach to the fatty molecule a group that would react readily with commercially useful unsaturated monomers, such as vinyl acetate and styrene, thus permitting chemical combination of the fatty compound with these monomers by copolymerization. The major difficulty in the practical achievement of this objective in the past has been the unavailability of sufficiently pure fatty compounds containing the necessary functional groups.

As an approach to the solution of this problem, the vinyl, chloroallyl and other similar unsaturated-alcohol esters of pure oleic acid have been prepared, and it has been determined that they copolymerize readily with a reactive olefinic monomer, such as vinyl acetate, thus incorporating the long chain as a structural unit of the polymer molecule. Unmodified polyvinyl acetate is hard and glass-like, but the copolymers of vinyl acetate with the unsaturated esters of oleic acid range in physical properties from viscous liquids, soluble in organic solvents, to insoluble rubber-like gels, depending upon the nature and proportions of the starting materials.

KEEPING QUALITY OF HOME-RENDERED LARD IMPROVED

Lard is a high-energy food material that is almost completely digestible and contains substances desirable for good nutrition. Strong or rancid lard is not only unpalatable, but some of its food value has been lost. Since rancid lard cannot be renovated easily, farmers and other householders who store it for home use frequently suffer considerable loss through rancidity.

Workers at the Eastern Regional Research Laboratory have found that the addition of 2 to 3 pounds of hydrogenated vegetable-oil shortening to 50 pounds of lard at the time of rendering results in greatly improved keeping quality. This inexpensive and simple method owes its effectiveness to the fact that hydrogenated vegetable-oil shortenings contain an abundance of natural antioxidants (tocopherols).

The preferred procedure is to add the hydrogenated vegetable-oil shortening to the rendered lard in the kettle just before settling and to separate the cracklings in the lard press. Another procedure is to add the shortening to the melted lard in the storage container. If this is done, however, careful stirring until the vegetable shortening is entirely melted is necessary to assure thorough mixing with the lard.

No known treatment will keep lard permanently fresh. Addition of hydrogenated vegetable-oil shortening only helps to keep lard fresh for a longer time. Hence care in following the best possible practices in rendering the fat are still important.

LARD PRESERVATIVE MADE FROM GALLIC ACID

Lauryl gallate, prepared from gallic acid by the new method of direct synthesis described in last year's report, continues to show promise as a preservative for lard and other fats. Further studies

confirmed the earlier laboratory tests which indicated that this compound has excellent antioxidant properties. They also showed that the protective action against rancidity is carried over into baked goods of the pastry type to a large extent, but that the carry-over into bakery products made with bicarbonate of soda (such as soda crackers) is not good.

Studies were undertaken by the Bureau's Pharmacology Laboratory to determine the toxicity of lauryl gallate. Present indications are that this compound is not toxic in amounts many times greater than those proposed for use as antioxidants in fats.

Now that a direct method of synthesis is available, there is considerable interest in the preparation, sale, and use of lauryl gallate as an antioxidant. Not the least important quality of this substance is its solubility in fats which leads to ease of incorporation with fat and to improved action over a wide range of storage conditions.

ANALYSIS OF OILS AND OILSEEDS TO BE MORE ACCURATE

When oils are used for food, their quantitative composition is probably not so important as is the presence or absence of particular substances, since even minute quantities of certain constituents may adversely affect the flavor or stability of the oil. However, for use of oils in paints and varnishes or as raw materials for chemical processing, it is extremely important to know their exact composition, particularly the proportions of the unsaturated acids—linolenic, linoleic, and oleic. The determination of these constituents has always been difficult, and the accepted method of analyzing oils containing all three of these unsaturated acids has required the determination of iodine and thiocyanogen numbers as well as the proportion of saturated fatty acids.

The Northern Regional Research Laboratory during the past year accumulated evidence that the empirical constants in general use for determining thiocyanogen numbers are not correct when applied to the analysis of oils. These constants were originally derived from analyses of methyl esters of the unsaturated acids and are generally used in official methods. Careful laboratory studies showed that the theoretical ratio is obtained between iodine numbers determined on an oil and on the mixed fatty acids prepared from it, but that this theoretical ratio does not hold for thiocyanogen numbers determined on the same two materials. Improvements were made in a newer, faster physical method for measuring linolenic ond linoleic acids in oil which involves alkali isomerization of the oil, followed by spectrophotometric examination. Since better solvents and conditions for isomerization have been discovered, this physical method may ultimately replace the thiocyanogen procedures.

RAPID ANALYTICAL METHODS DEVELOPED FOR TUNG OIL MILLS

The proper sampling of tung nuts delivered to the mills by trucks and rapid determination of their oil content have received attention from the Agricultural Chemical Research Division. As competition from foreign tung oil increases, accurate information on oil content will be required as a basis for economy in purchasing raw material and effective control of oil extraction to get maximum yields. Chem-

ists of the Tung Oil Laboratories are serving as members of the committee on analytical methods of the American Tung Oil Association and are conducting both research and referee work on improvement and standardization of the methods used. Procedures have been devised for properly withdrawing from a truckload of tung nuts a 50–100 pound sample and for taking from it a 100-nut representative subsample.

Rapid methods of determining oil and moisture in such a subsample have been devised and are being tried by referees to minimize labor and delay in obtaining information required for processing the various lots of nuts. These include a method based simply upon an estimate of the percentage by weight of kernels in the sample and an assumed oil content of kernels, since oil content has been found to be fairly constant for any given area throughout each season; also a method based upon determination of the refractive index of the solution obtained when a certain weight of a particular solvent is allowed to come to equilibrium with a definite weight of the finely ground sample of tung nuts. In the longer extraction procedure intended for more accurate oil determinations, drying of the subsample is essential, even if double extraction is employed.

FRUIT-ESSENCE PROCESS WIDELY ADOPTED BY INDUSTRY

The process developed at the Eastern Regional Research Laboratory for recovering in essence form the volatile flavoring constituents of apple and other fruit juices has been adopted by 25 industrial firms. Eighteen of these are producing essence for their own use rather than for sale. They incorporate it with their cooked fruit products to enhance their flavor, because the essence contributes the "top note" normally lost during cooking. Seven companies are producing apple essence and certain other fruit essences for sale. Thirty-six other companies are attempting to obtain the necessary equipment for fruit-essence recovery.

Although the Eastern Regional Laboratory has done some exploratory work on recovering essence from the juices of fruits other than apples, sufficient work has not been done on any of these fruits, except Concord grapes, to permit making specific recommendations for operating procedures. In order to determine the optimum conditions for essence recovery from some of the more important fruit juices, a portable essence-recovery unit has been constructed. This has a capacity of 10 gallons per hour of apple juice, as compared with 50 gallons per hour in our pilot-plant unit. The small unit can be taken to the source of supply of the fruit juice and, without consuming too large quantities of the fruits, will yield engineering data from which specific operating procedures can be recommended.

Several attempts have been made to recover apple essence from parings and cores. These accumulate in large quantities at apple processing plants and are frequently used for the production of vinegar. If the juice from them could be made to yield a satisfactory essence, the juice stripped of essence could still go into vinegar manufacture. The results so far have been discouraging. Wherever cores were used, with or without parings, the essence had a strong benzaldehyde flavor characteristic of apple seeds. Such essence can be used only in small quantities in a blend with good essence from whole

apples. Nor has a good essence been obtained from apple skins alone. This, however, may be attributable to flavor alteration while the skins are being transported to the laboratory. The portable unit will make it possible to establish by tests at apple-processing plants whether or not a satisfactory essence can be obtained from apple parings.

The identification and quantitative determination of the individual constituents of apple essence were undertaken by the Eastern Regional Laboratory. Although some constituents are still unknown, the following ones have been identified: Acetaldehyde; *n*-hexaldehyde; methanol; ethanol; *n*-butanol; *n*-hexanol; (probably) *n*-propanol; ethyl esters of formic, acetic, and propionic acids; and acetone.

SOFT VARIETIES OF APPLES CAN BE FIRMED FOR PIES

The highly flavored summer varieties of apples are favored for home pie making, but they are avoided by commercial bakers, because the slices disintegrate—"mush up"—too much. The same fault applies in some areas to such fall varieties as McIntosh and Delicious. The Massachusetts Agricultural Experiment Station published a method for firming McIntosh apple slices by means of calcium salt, which reacts with the pectin to form a stiff gel. The Eastern Regional Research Laboratory followed this up and developed several procedures for firming soft apple slices in general.

The procedures for firming fresh, canned, or frozen apple slices require only slight modifications in the usual methods of preparation. The apples are first peeled, cored, and sliced. If a delay is necessary at any point between peeling and subsequent steps in processing, the apples should be held in a weak salt brine (0.1 to 1.0 percent of salt by weight) to prevent browning.

The recommended procedure for firming *fresh slices* is to dip them in a dilute solution of calcium chloride. The concentration of calcium salt may vary from 0.1 percent to 1.0 percent, and the dipping time may vary from a few minutes to 1 hour. In most cases a 2- to 10-minute dip in 1.0-percent calcium chloride solution is satisfactory. With very soft apples it is often desirable to allow slices to stand several hours after dipping so that the calcium chloride solution may diffuse into the center of the slices.

Canned slices which have been firmed by a calcium chloride dip show case-hardening (nonuniform firming) at first, but this condition disappears after about 2 months' storage. Apparently the calcium salt migrates very slowly to the interior of the slice. If the slices have been deaerated prior to canning, they may be firmed by adding the calcium solution to the can, 1 or 2 ounces of a 1-percent calcium chloride solution being sufficient for a No. 10 can. Some plants use a combination evacuation and blanching process, which has proved to be an economical method of pretreating the slices to eliminate air and fill them with water.

Slices for freezing are blanched with steam for a sufficient time (usually 1½ to 2 minutes) to prevent browning, then dipped in a cold water bath containing 0.5 to 1.0 percent of calcium chloride. The calcium chloride cooling solution may be applied also as a spray. The time of contact may vary from 2 to 20 minutes and should be long enough to remove the excess heat from the slices. In commercial-scale operations it is advisable to cool and recirculate the calcium

chloride solution. Instead of treating apple slices with calcium salt before freezing, calcium chloride may be added to the dilute sulfurous acid (or sodium sulfite) solution used to prevent browning after thawing of the frozen slices.

Since 14 varieties of apples have been successfully firmed with calcium salt there is good reason to believe that all varieties can be firmed in this way.

ADVANCES MADE IN CANNING AND FREEZING PROCESSES IN NORTHWEST

Improvement in the color of commercially canned freestone peaches was made possible as a result of processing methods developed by the Fruit and Vegetable Products Laboratory at Pullman, Wash. These methods provide for the removal of gases entrapped within the fruit tissue and reduce to a minimum discoloration by oxidation during processing.

The possibility of reducing the cost of canning and freezing freestone peaches, without sacrifice in quality, was demonstrated by studies completed this year in cooperation with the Washington State Agricultural Experiment Station. No advantage to flavor of either the canned or frozen product was found when fruit was permitted to become soft-ripe on the tree, provided that fruit less mature at harvest was permitted to soften in storage before processing.

Flavor diminished noticeably only when the peaches were harvested so immature that 8 or more days in storage were required to ripen them after harvest. Lower quality and yield resulted when the peaches were permitted to soften on the tree because many were bruised during harvesting. When peaches were harvested at very immature stage low yields were obtained because they had not attained their maximum size. The best flavor in both canned and frozen peaches was obtained by ripening the fruit at moderate temperatures (70° to 80° F.); high temperatures, especially under conditions of high humidity, resulted in poor flavor. The nutritive value of peaches harvested at a firm-ripe stage and permitted to soften in storage was found to be equal to that of tree-ripened fruit.

A 2-year study on the canning and freezing technology of Idaho-grown vegetables was completed in cooperation with the Idaho Agricultural Experiment Station. Results of these studies demonstrated the suitability of the Palouse area for the production of canning and freezing peas on a limited-season basis. Further studies are under way to determine practical limits of the commercial processing season for this crop. The practicability of producing sweet corn for processing in the Lewiston (Idaho) area was shown. Golden Cross Bantam was found to be the variety best adapted to the area from the standpoint of both yield and quality of the canned and frozen product. Seneca Chief, a new variety, was considered very promising as a processing variety, while Seneca Golden was found suitable as an early cropping variety.

At present irrigation-grown tomatoes of the Pacific Northwest are considered unsuitable for the manufacture of high-quality canned tomatoes. In studies completed this year horticultural practices and processing methods required for the production of Grade A, whole, solid-pack tomatoes were established for tomatoes grown in the Lewiston area.

FROZEN FOODS IMPROVED IN QUALITY

The Western Regional Research Laboratory gave attention to several problems that are important to the rapidly growing frozen-food industry and supplied much information on the technology of freezing preservation of foods to visitors and correspondents.

At present there is no uniformity in the practice of blanching (scalding) sweet corn to inactivate enzymes before freezing. Frozen sweet corn is increasing rapidly in favor and in the volume of production. Work in cooperation with the Washington and Utah Agricultural Experiment Stations showed that blanching of sweet corn on the cob prior to cutting off the kernels results in a higher yield and more palatable product. The yield of cut corn is 20 percent greater, and the sugar content about 5 percent more, for corn blanched on the cob as compared to that cut prior to blanching.

The control of oxidative browning in frozen fruits constitutes one of the industry's most urgent and ever present problems. Therefore the process, described in last year's report, of using a dipping bath containing sodium sulfite, salt, and ascorbic acid was further investigated and adapted to various fruits. It is interesting to note that ascorbic acid, which may be obtained from waste hulls of "English" walnuts (as described in another section of this report), is useful as an antioxidant in the freezing preservation of foods.

A satisfactory and accurate analytical method for determining the susceptibility of different peach varieties and lots to oxidative browning was developed. By using this test it is possible to select varieties of fruit that are best suited to freezing preservation and in this manner to obtain products of improved quality.

When frozen fruits are defrosted an undesirably large amount of juice drains from the pieces. In commercial pie making this results either in pies that are too juicy or in a loss from discarded juice. Ways of processing and defrosting have been discovered that can aid in diminishing this difficulty. However the problem cannot be considered as completely solved, and more work must be done on it.

Last year's report described a jelly-like, full-flavored, frozen fruit spread. Storage of this product at different temperatures has yielded information on its keeping qualities as regards texture and flavor that will be useful when the sugar situation and the greater availability of fruit make it possible to run full-scale commercial tests to determine consumer acceptance and actual cost.

Preliminary experiments have shown that frozen vegetables may be safely packed in hermetically sealed containers insofar as the possibility of *Cl. botulinum* poison development is concerned. If this finding is adequately confirmed, it may revolutionize the packaging procedures for frozen foods. The advantage would be lower labor costs from mechanization of the packing procedure and greater ease of handling the packaged products.

DEHYDROFROZEN PEAS AND APPLES PROVE TO BE OF EXCELLENT QUALITY

Work was continued at the Western Regional Research Laboratory on the dehydrofreezing process for fruits and vegetables first announced in last year's report. Dehydrofreezing, as was then

explained, includes partial dehydration to remove a large part of the moisture followed by quick freezing, the product being kept in freezing storage until needed for use. Reduction in weight and volume of the food before freezing it deceases the storage, transportation, and handling costs. The question of best procedure for each step of the process and for different kinds of fruits and vegetables has been systematically attacked. Thus far, the greatest attention has been given to peas and apples. The maximum weight reduction that can be achieved during the preliminary dehydration without impairing the quality, especially the texture, of the product varies with different commodities. For peas and apples the limit appears to be about 50 percent, based on the weight of prepared raw material.

The culinary quality of dehydrofrozen peas is comparable to that of freshly shelled peas and equal to that of peas subjected to the conventional method of freezing preservation. This was shown by results of preference tests made with a trained taste panel. The early results from storage tests still in progress indicated that dehydrofrozen and frozen peas are about equally stable as regards color, flavor, and texture. The data were obtained on comparable samples of dehydrofrozen and frozen peas taken from storage at −10° F. after 6 months and again after 12 months.

Excellent pies were prepared from dehydrofrozen apples. The texture and flavor of the fruit in the pies were essentially equal to those in pies prepared from the same lot of apples while fresh, and there was no suggestion of "dried-apple flavor." Preliminary experiments indicated that when the original apples lack tartness, the final product can be improved in flavor by incorporating a small amount of citric acid in the water used for rehydration. In this manner it may be possible to utilize surplus apples of varieties grown principally for the fresh-fruit trade, such as Delicious, that are usually regarded as unsuitable for pies. The rehydration is relatively simple. Cold water in the required amount is poured over the frozen pieces, and the mixture is brought to the boiling point, after which it is permitted to stand for 25 minutes. The water is drained off and may be used for preparing syrup for use in the pie.

The experimental results demonstrated the feasibility of dehydrofreezing, as it does not impair quality nor prevent utilization of the product. It remains to accumulate data for the design of the dehydrofreezing equipment lay-out needed for pilot-scale work. Such work should provide facts upon which to base cost estimates on the commercial production of dehydrofrozen products. Extensive accomplishments during the war years at the Western Regional Research Laboratory on the dehydration of fruits and vegetables provide a comprehensive background of knowledge and experience for development work on dehydrofreezing.

EXCLUSION OF ORANGES AFFECTED WITH SOFT ROT NECESSARY TO AVOID COLIFORM BACTERIA IN FROZEN ORANGE JUICE

The production of frozen orange juice offers an important outlet for good sound oranges that cannot be sold on the fresh-fruit market because of overripeness, poor color, odd size or shape, or overabundance. Rapid expansion of the frozen-food industry has left unsolved a number of problems relating to such foods. One of these is the sanitary

significance of the micro-organisms in orange juice that is frozen without preliminary sterilization. Therefore the Western Regional Research Laboratory has studied the relationship of the kinds and numbers of micro-organisms in frozen orange juice to the sanitation measures taken in the processing plant.

Particular attention was given to the coliform group of bacteria, which generally occur in the product along with yeasts, molds, and other common microforms. The number of coliform organisms regularly found, as well as certain of their cultural characteristics, made it evident to earlier investigators that these organisms could not all have their origin in filth and consequently would have a sanitary significance differing substantially from that of coliforms observed in water supplies. This point, however, had not been proved, and, in any event, there existed no recognized procedure for examining orange juice to differentiate among the various types of coliforms.

The studies carried on at the Western Regional Laboratory indicated that most of the coliforms in commercial frozen orange juice differ characteristically from fecal coli, and accordingly are not to be regarded as of intestinal origin. A series of tests was devised which is tentatively regarded as satisfactory for adequate differentiation. It was shown that an orange spoiled by the condition known as soft rot may be the source of many of the nonfecal coli, and that these bacteria, although relatively inactive at the acidity of orange juice, nevertheless can grow and multiply if they are permitted to accumulate at various points along the processing line. From such points, of course, they find their way into the juice in large numbers.

These findings emphasize the critical importance of careful selection and washing of fruit for processing, as well as the necessity for scrupulous sanitation in the plant, if bacteria counts are to be kept at a low level. Further work will be done to learn the numbers and kinds of micro-organisms to be expected in frozen orange juice when prepared under various conditions and how to make routine examinations of the product. In the latter connection, studies are under way to determine the loss of viability by the various micro-organisms during freezing storage.

BETTER WESTERN CITRUS PRODUCTS FORESHADOWED

Further work was done by the Fruit and Vegetable Chemistry Laboratory at Los Angeles, Calif., on the preparation of standardized juice from western grapefruit by adjusting the sugar-acid ratio with cane sugar and sodium bicarbonate to make a product of uniform taste during the entire canning season. Samples from two seasons' packs of untreated and treated juices were submitted to the taste panel at the Western Regional Research Laboratory for consumer preference tests. The results of these tests clearly indicated a preference for the juices that had been modified by adding both sugar and bicarbonate. During the 1946–47 grapefruit season, one of the commercial processing plants standardized the sugar content of its juice pack by adding sugar to maintain a uniform reading on the Brix hydrometer. Other companies plan to put up experimental packs of juices adjusted with both sugar and bicarbonate.

Work on the quick-freezing of western citrus products was continued and expanded. The quick freezing of orange purees yielded

results that may have commercial application. During the winter orange season, Arizona "Sweets," Arizona Valencias, and California Navels were tested for suitability in making frozen purees. After several months' storage at 0° F., the purees were made into orange sherbets by one of the local ice-cream companies, and the sherbets were evaluated on the basis of flavor. The Navel orange sherbet was judged to be the best; the Valencia, next; and the "Sweet," the least desirable. Since Navel oranges are ordinarily not used for processing, the preparation of a frozen Navel orange puree may open a new field and provide a greater utilization of this fruit. The frozen puree could be prepared during a season of the year when other fruits are not available for freezing.

Studies were made on the keeping qualities of fresh, unpasteurized, chilled orange juice for distribution in and around Los Angeles and to more distant points by air express and fast truck service. Results indicated that, when fruit of good quality is used, the juice will remain good for 7 days or longer if it is thoroughly deaerated, chilled to temperatures around 32–38° F.. filled into containers without any headspace, and kept at 32–38° F. until consumed. No losses of ascorbic acid were noted in the samples. One of the largest producers of chilled, fresh orange juice in Southern California is now using this method of processing, and a local air-express company is planning to transport this type of product as far as Chicago.

Great difficulty has been experienced in packing canned grapefruit segments in Arizona or California because the segments are so tender that they become soft and mushy on heating. Several lots of grapefruit segments were heat-processed with varying amounts of calcium chloride being added to act as a firming agent by forming calcium pectate. On the basis of drained weights and eating tests, the calcium treatment had effectively firmed the segments and prevented them from becoming mushy when heated.

Frozen packs of grapefruit and Navel orange segments in equal parts were successfully prepared in the Laboratory during the past season. The raw segments were packed in grapefruit juice, in grapefruit-juice syrup, or in water syrup and quickly frozen. After 3- and 6-month storage periods at 0° F., the samples were tasted and found to be of excellent flavor, color, and texture, with no noticeable bitterness in the Navel orange sections. The segments packed in the grapefruit juice and in grapefruit syrup were far superior to those packed in the water syrup. This product has an excellent appeal to the eye because of the brilliant orange color of the Navel orange segments and the golden yellow color of the grapefruit segments.

Work was continued under a cooperative research project inaugurated by the Grapefruit Program Committee of the Arizona Grapefruit Advisory Board on the isolation and identification of the constituents of grapefruit juice. Thirty tons of desert grapefruit grown in the Imperial Valley of California were processed into juice, and the juice was concentrated to 50 percent of its original volume under vacuum. All volatile constituents were collected by low-temperature condensation and will be purified and identified. Twenty-four hundred gallons of freshly canned juice packed in a commercial plant will be similarly concentrated, and the constituents will be

identified to determine what changes were brought about by the processing. Another 2,400 gallons of commercially processed juice will be stored for 1 year and then concentrated and analyzed to determine the effects of storage on the flavoring constituents.

MORE LEARNED ABOUT MINOR CONSTITUENTS OF ORANGES

Continued progress was made at the New Orleans laboratory of the Agricultural Chemical Research Division on the separation of the lipid or fatlike fraction of orange juice and isolation from this fraction of substances that may contribute to the deterioration of orange juice during canning and storage. Promising results were obtained by electrodialyzing orange juice; that is, by passing a direct electric current through the juice and flowing water separated from each other by a membrane of transparent cellulose. Under the electrical potential the cells and cell fragments suspended in the juice collect on the membrane, which they cannot penetrate, and may be scraped off.

Examination of such material under the microscope, after staining it with suitable dye, showed that young cells from the tips of the juice sacs contained spherical plastids, while in the older cells the envelopes of these plastids were generally open, and mats of pigment and oily particles had exuded. Acetone caused the spherical plastids to swell and open with release of the pigments. By use of acetone as the solvent, the material collected by electrodialysis was separated into two fractions—a soluble colored fraction containing two carotenoid pigments and an insoluble waxy fraction of which the envelopes of intact plastids are composed. These fractions are being further separated into pure substances for the purpose of determining their chemical structures.

In a study on minor orange constituents soluble in organic solvents, a considerable quantity of a pure crystalline compound was obtained from the wall tissue of juice sacs of oranges. The sac-wall material was first screened from the juice and then extracted with hot methyl alcohol. It is of interest that as much as 40 to 50 milligrams of this pure compound, melting at 256° C., could be obtained from a single orange. While its structure has not yet been determined, this compound has phenolic properties and contains only carbon, hydrogen, and oxygen. The methyl-alcohol extract also contains a phosphatide and the phellonic acid previously identified as occurring in the deposits of cork-like material found adjacent to juice-sac walls.

NEW PHOSPHORUS-TRANSFERRING ENZYME FOUND IN CITRUS FRUITS

Growers of Navel oranges in California are at a disadvantage with respect to sales to processors owing to a tendency of the juice to become bitter in a short time after being squeezed out. Therefore, further work was done by the Enzyme Research Laboratory on the chemical changes that produce this bitter taste.

Several bitter substances are known to exist in citrus fruits, two new ones having been discovered recently by this Bureau; but the chief cause of the bitter taste in Navel oranges appears to be limonin, a substance that becomes bitter when exposed to weak acid. When the juice is squeezed out, some of this substance is extracted from the peel

and gets into the juice, which is acid. The acid of the juice gradually changes the substance to its bitter form.

Evidently the quantity of limonin in oranges decreases with ripeness, for the juice of very ripe Navel oranges develops little or no bitterness, whereas the juice of immature fruit becomes very bitter. Similarly, the juice of unripe Valencia oranges may at times develop the bitter taste. Since the degree of ripeness seemed to be an important factor in avoiding the formation of bitter substances, studies of the enzyme actions that produce ripening have been carried on.

Navel oranges artificially ripened by subjecting them to ethylene were found to have little or no bitter principle left thereafter. Unfortunately the treated oranges, though no longer bitter, developed a musty flavor that made them of poor quality. But the inverse relation between degree of ripeness and bitter principle seems to be established.

In further efforts to understand the phenomena of ripening in citrus fruits, the behavior of the phosphate in the fruit was studied. An enzyme capable of causing the migration of phosphate from one substance to another has been found and this reaction is being studied for its possible connection with the formation of sugar in the fruit. The reaction in question has not previously been observed and recorded, and its possible role in animal and human metabolism is now the subject of considerable discussion among biochemists.

The enzyme, or catalyst, that causes phosphate transfer is readily injured by heat. By measuring what is left of it after heating, an estimate of the extent of the heat treatment may be made. It is believed that this test may prove to be a practical means for controlling some factory processes and may also help in determining the treatment to which unknown samples have been subjected.

TOO MUCH SALT CAUSES LOSSES IN CUCUMBER PICKLING

Formation of hollow pickles, or "bloaters," is still one of the main causes for loss in the processing of cucumbers. Previous investigations by this Bureau showed that when the brine fermentation is accompanied by vigorous gas evolution the percentage of bloaters is usually high. In its continued attack on this problem the food fermentation section of the Agricultural Chemical Research Division, in cooperation with the North Carolina Agricultural Experiment Station at Raleigh, obtained cultures of yeasts from commercial cucumber fermentations and isolated and classified over three hundred strains of yeast from them. Some of these yeasts can ferment cucumber sugars and cause rapid evolution of gas in brines of relatively high salt concentrations. It is obvious that they contribute to excessive formation of gas and resultant bloaters in some commercial brining operations. They are especially likely to contribute to serious losses if salt concentrations greater than 15 percent are employed, because the higher salt concentrations interfere with lactic acid fermentation but favor gaseous yeast fermentation.

MORE PROGRESS MADE TOWARD NUTRITIONAL BALANCE IN CANDIES

The Agricultural Chemical Research Division, in cooperation with the National Confectioners' Association, has been studying the possibilities of adding protein to the types of candy which, as now produced,

are practically all carbohydrate. Significant progress was made during the past year, because more suitable grades of highly refined protein became available from manufacturers of soybean products. Experiments were made with soybean protein that contained 90 percent or more of pure protein and was tasteless and of excellent color.

It was possible to produce pulled hard candy, which ordinarily is an all-sugar candy, containing up to 5 percent of this protein. The candy was of excellent flavor and satisfactory texture, and could be economically marketed. Such products would help to overcome the objections of many people to the cheaper grades of hard candy by providing both protein and carbohydrate in a ratio that is considered nutritionally adequate. The improved, refined, protein products now available also make it possible to readily increase the protein contents of cast creams, nougat, and other types of soft candy to as much as 7 or 8 percent, which is fully adequate for nutritional balance.

A new type of cast marshmallow for chocolate dipping, in which fruit purees are used to impart attractive color and full, natural, fruit flavor, was developed during the year. Concentrated fruit purees are now available commercially, and candies of this type are being adopted for use in high-grade assortments. Chocolate-coated marshmallows containing raspberry puree were outstanding in flavor, and were rated excellent by all of the candy manufacturers to whom samples were submitted.

Experiments were undertaken for the purpose of replacing highly refined, but indigestible, mineral oil, commonly used as a dressing on candy-working slabs, with an equally serviceable, but digestible, product. Numerous vegetable oils were tested, but none of those on which complete data have been obtained thus far meets the severe requirements of a satisfactory slab dressing. The chief faults of vegetable oils are that they develop a rancidity that remains on hard candies and lack stability when exposed to the high temperature at which hard-candy batches are worked on the slab. The search is being extended to special synthetic glycerides and other classes of substances that seem to have suitable physical properties in order to determine if any available substance is free from the oxidizing tendency characteristic of vegetable oils and responsible for their lack of utility for this purpose.

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